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presented by

Robert Alan Gentner

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of the requirements for the

Doctoral degree in Chemistry

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UNDERSTANDING THE NATURE OF 3,6-BIS(BIS(3,5-DI-TERT-BUTYL-4-METHOXYPHENYL)METHYLENE)-1,2,4,5-TETRAOXOCYCLOHEXANE AND ITS RADICAL ANION AND DIANION

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By

Robert Alan Gentner

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ABSTRACT

UNDERSTANDING THE NATURE OF 3,6-BIS(BIS(3,5-DI-TERT-BUTYL-4-METHOXYPHENYL)METHYLENE)-1,2,4,5-TETRAOXOCYCLOHEXANE AND ITS RADICAL ANION AND DIANION

By

Robert Alan Gentner

In this work, the organic building block **1E** (3,6-Bis(bis(3,5-di-*tert*-butyl-4-methoxyphenyl)methylene)-1,2,4,5-tetraoxocyclohexane) and its monoradical monoanion $\mathbf{1E^{-\bullet}}$ and diradical dianion $\mathbf{1E^{2-\bullet\bullet}}$ were synthesized and studied for their potential to serve as paramagnetic organic linkers between multivalent metal centers in magnetic materials. The organic unit **1E** is easily reduced to $\mathbf{1E^{-\bullet}}$ and $\mathbf{1E^{2-\bullet\bullet}}$ through contact with an alkali metal mirror. The reduction can be followed by UV/Vis/NIR, which is presented herein, and by EPR spectroscopy.

According to X-ray diffraction techniques, one-dimensional paramagnetic chains are produced when the monoradical monoanion of $\mathbf{1E^{-\bullet}}$ comes into contact with a sodium metal mirror. The crystalline material of $\mathbf{(1E^{-\bullet})_2{\cdot}(Na^+)_2{\cdot}(C_4H_{10}O_2)_8}$ was analyzed by EPR and SQUID magnetometry and found to have a weak intermolecular spin-spin coupling of $2J = -0.2$ K. The closely related potassium(I) crystal structure was found to have a weak intermolecular spin-spin coupling of ~0K.

Further reduction of $\mathbf{1E^{-\bullet}}$ with an alkali metal mirror generates $\mathbf{1E^{2-\bullet\bullet}}$. This diradical dianion was designed to have a ground state triplet and facilitate ferromagnetic or ferrimagnetic coupling with multivalent metal centers through

intramolecular and intermolecular spin-spin interactions. However, EPR studies suggest that it behaves as a diradical with isolated unpaired electrons residing on the terminal diarylmethylene positions and interacting weakly. Failure of this diradical dianion and its salts to crystallize led to the exploration of the tetrone precursors **2A-E** (2,5-dihydroxy-1,4-benzoquinone derivatives).

Multi-dentate organic bases and various metal bases, which possess the ability to coordinate through hydrogen bonding or ionic bonding, were used to determine if the dimensionality of the proposed organic building blocks of **1** could be increased from one-dimensional chains into two-dimensional honeycomb sheets or three-dimensional diamond-like networks. Crystal structures of all dimensionalities were generated and are presented herein but none displayed the intended structural motifs. However, the diamagnetic crystals revealed potential design flaws within the tetrone framework. First, the twisting of the diarylmethyne positions suggested the diarylmethylene positions within the tetrone system may undergo the same phenomenon and cause the unpaired electrons to isolate themselves via this twisting. Second, the bulky *t*-butyl substituents effectively block a potentially useful mode of coordination between the aryl π-system and the metal cation center. Finally, they also cause large void spaces to be created between the linear chains which may prohibit the formation of honeycomb sheets or diamond-like networks.

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The work presented herein is dedicated to the Living God and His only begotten Son, my Lord, Jesus Christ, whose infinite wisdom and power created the universe and all that it contains.

"The fool says in his heart, 'There is no God.'"
But, "everyone who calls on the name of the Lord will be saved."
-Psalm 14:1-
-Romans 10:13-

Jesus said, "I am the way and the truth and the life.
No one comes to the Father except through me."
-John 14:6-

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Throughout the years of this study my life changed dramatically. I met my loving wife Jennifer, who gave birth to our two children: Morgan Rain (who turned three the day after I defended) and Noah Thomas, on the 4th of September in the year 2004. They have remained faithful and an enormous encouragement in the most trying of times. Their unconditional love for me remains unwavering-may God bless them for their dedication. The hardest thing

I had to do during the last three years was to look at my wife and daughter in their eyes and tell them that I could not spend time with them that day. I ask God never to allow that time to come again until my passing. May I return that dedication to them many times over.

I would like to extend the warmest thanks to my parents, Dennis and May, for dedicating their lives to raising me. Also, thank you to my younger brother, Michael, and my sister, Chris, for being there through the years and for their love. I cannot thank my family enough, especially my grandparents Betty and Elmer (who passed away during my undergraduate years) and the two grandparents who I hardly knew, Austin and Gracie Whitaker.

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INTRODUCTION

Magnets have long fascinated scientists because of their innate ability to exert an unseen attractive or repulsive force on objects without making physical contact. Traditional magnets are composed solely of inorganic materials in which the unpaired electrons reside in *d* or *f* orbitals.[1] However, the incorporation of organic components into traditional magnets promises rational control over their architectural and magnetic properties.[2] One of the strategies used in the construction of molecule-based organic magnetic materials is a hybrid 'metal-radical' approach[3] in which organic spin carriers are used in conjunction with diamagnetic or paramagnetic metal cations to achieve desired structural motifs and/or targeted magnetic properties. The main advantage of using an organic spin carrier over other systems utilizing diamagnetic organic components is direct intermolecular spin-spin contact, which promises robust magnetic interactions.

The inclusion of organic components into traditional magnets may give rise to properties not previously attributed to traditional magnets, including optical transparency, chirality, solubility, tunability, etc. In principle, one of the main advantages of molecular magnets over traditional magnets is synthetic flexibility. Syntheses can be carried out at or below room temperature, in various solvents, and the organic unit can be drastically altered, through the plethora of organic synthetic methodologies, to yield desired electronic or physical properties and characteristics. However, due to the intrinsically high reactivity of organic radicals, the number of paramagnetic ligands capable of forming stable

complexes with metal cations is limited.[4] Moreover, magnetism is a cooperative phenomenon and in order to exhibit spontaneous magnetization, organic magnetic assemblies must have the ability to propagate interspin ligand-cation interactions through extended solid state networks. Therefore, the organic component should be designed to bridge multiple metal centers. This requirement further reduces the number of useful organic building blocks to the few that possess well-defined multidentate coordinating abilities. Nitronyl-nitroxides,[5] radical anions of TCNE (tetracyanoethylene) and TCNQ (tetracyano-*p*-quinodimethane),[6] and *m*-xylylene coupled nitroxides[7,8] are representative examples of mono- and polyradicals that have been extensively investigated for the purpose of preparing molecular magnets. However, despite forming solids with cooperative magnetic behaviors, they also were found to have their limitations. In the case of nitronyl-nitroxides, they were found to lack geometrically selective coordinating abilities and the complexing metal cations required counteranions (usually diamagnetic), which tend to interrupt magnetic communication. For radical anions of TCNE and TCNQ, the coordination sites were found to be poorly defined and these species are furthermore prone to covalent dimerization. Finally, the *m*-xylylene coupled nitroxide[9] suffers from two main flaws: weak intramolecular ferromagnetic couplings and excessive flexibility of their molecular framework. For the systems mentioned, in most cases, ferrimagnetic interactions are naturally favored over the more desirable ferromagnetic interactions.

Previously in our group,[10] tetrones of **1** (see Figure 1) were designed and

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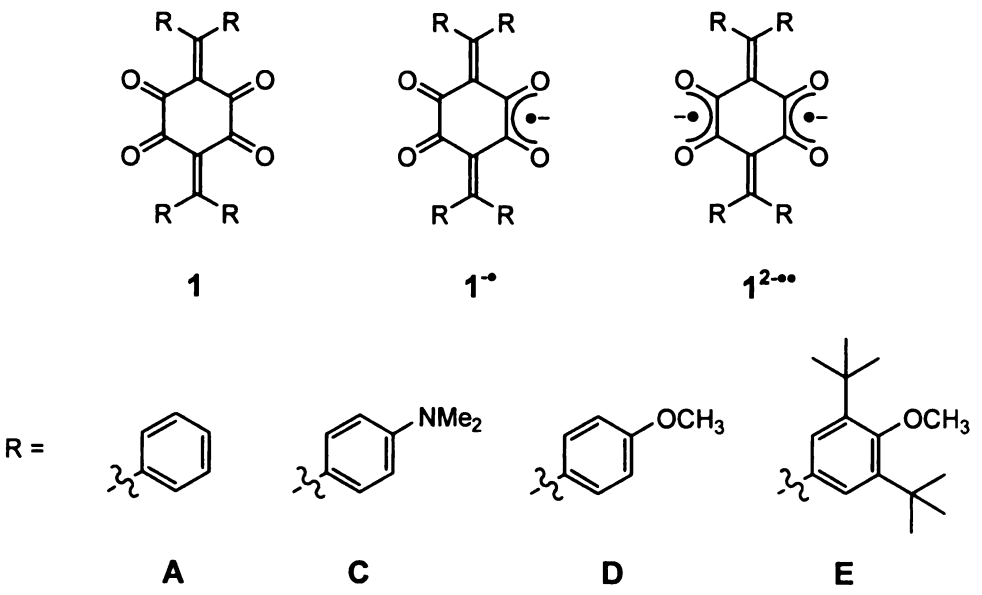

Figure 1. Tetrones that have been synthesized and characterized.

studied with *ab-initio* calculations (these will be discussed in greater detail in Chapter 1), synthesized, and characterized through various spectroscopic techniques. Radical anions of **1** have the potential to self-assemble into extended magnetic multi-dimensional networks with well-defined cation-ligand geometric and magnetic interactions. Such motifs, which are formed by the isostructural diamagnetic dihydroxybenzoquinone dianions (see Figure 2), include one-dimensional molecular chains,[11] two-dimensional honeycomb sheets[12] and even three-dimensional diamond-like networks.[13] In theory, the metal cations would be chelated by the paramagnetic monoradical monoanions of **1** instead of the existing diamagnetic organic unit(s). Coordination contacts in semiquinone/dihydroxybenzoquinone complexes can transfer strong magnetic couplings through predictable exchange mechanisms[4d,14] between the

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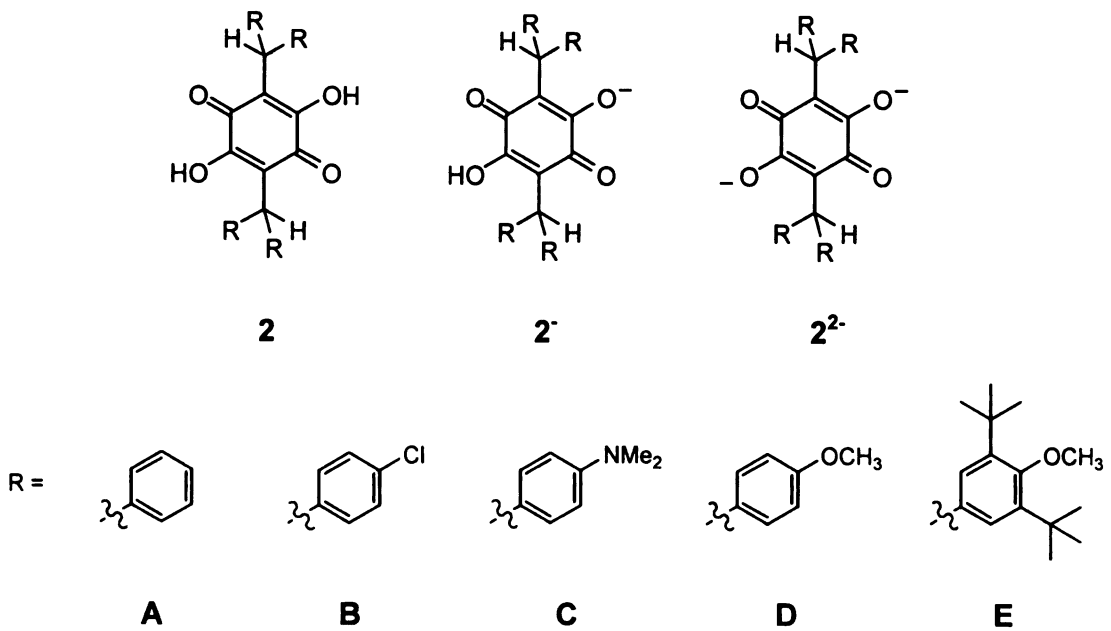


Figure 2. 2,5-dihydroxy-1,4-benzoquinone derivatives.

paramagnetic metal center and ligands. Monoradical monanions and diradical dianions of **1** may also be combined with diamagnetic metal cations in order to achieve materials with targeted properties. Second, the HOMO-LUMO energy gap of $\mathbf{1}^{2-\bullet\bullet}$ may be tuned by substituent variation on the methylene carbons, and/or two, by varying the metal cation, which is coordinated to the semidione sites. *Ab initio* MCSCF calculations on $\mathbf{1}^{2-\bullet\bullet}$ have found that electron-donating substituents for -R and coordination by highly charged metal cations can shrink the energy gap between these dianions' frontier orbitals, making them nearly degenerate. This degeneracy, coupled with the disjoint character of the frontier orbitals, would lead to a preference for the complexed diradical dianions to possess a ground state triplet. This high-spin preference should make it

theoretically possible to use the paramagnet $\mathbf{1}^{-\bullet}$, or the magnetically tunable $\mathbf{1}^{2-\bullet\bullet}$, as spin-bearing organic building blocks in the formation of molecular magnetic materials depending on the preferred strategy.

The goal of the present research was to extend the project beyond the theoretical realm and develop practical molecular magnetic materials with applications that exhibited useful characteristics, including ferromagnetism at or near room temperature or the ability to "fine-tune" the electronic state(s) of the organic-based magnetic material. The initial attempt was to crystallize monoradical monoanions and diradical dianions of tetrone **1** with various metal centers into extended multi-dimensional frameworks and then characterize their physical, electronic and magnetic properties through the use of X-ray crystallography, SQUID magnetometry, EPR (Electron Paramagnetic Resonance) spectroscopy and other various spectroscopic and chemical techniques. This research has been partially successful but failed in its overall goal to construct the targeted networks and produce molecular magnets with the desired properties and applications.

During the course of this study many difficulties plagued the research, as will be discussed in later chapters. As a result the more stable, disubstituted, 2,5-dihydroxy-1,4-benzoquinones of **2**, were used instead of the various tetrones of **1**. The dihydroxybenzoquinones of **2**, which are isostructural analogs to the tetrones, crystallized with metal and organic bases via ionic bonding and classical hydrogen bonding interactions, respectively. The crystal structures of **2**, although magnetically uninteresting, formed zero-dimensional inorganic/organic

salts, chains, sheets, and a three-dimensional network, revealing important information about the problems that might be occurring within the tetrone system. Ultimately, the main value of the present research work is the large structural database that has been built up for species of the basic framework of compounds **1** and **2**.

CHAPTER 1

BACKGROUND

1.1: Rational Design for $1^{2-\bullet\bullet}$

One common approach to the design and construction of organic building blocks for use in molecular magnets has been to synthesize derivatives of diradicals trimethylenemethane (**TMM**) and *m*-benzoquinodimethane (**MBQDM** or ***m*-xylylene**),[15] which are shown in Figure 3. There are several reasons for this: First, both **TMM** and **MBQDM** have been well characterized and have a substantial preference for a triplet ground state. **TMM** is ~15 kcal/mole more

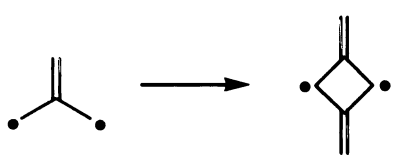
TMM **MBQDM**

Figure 3. **TMM** and **MBQDM**.

stable in the triplet state[16] than in the singlet state while **MBQDM** prefers the triplet state to the singlet state by ~10 kcal/mole[17]. With such a strong preference for the triplet state, organic building blocks that incorporate either of these two molecular fragments should serve as high-spin carriers. Second, the singlet-triplet energy splittings (ΔE_{ST}) are large, ~0.5 eV, so it should be possible to introduce the structural modifications needed to enable metal ion coordination

without switching the nature of the ground spin state,[15f] because both **TMM** and **MBQDM** lack the structural features required to coordinate to metal centers.

In making the first structural modification one could imagine transforming **TMM** into the diradical dimethylenecyclobutane-1,3-diyl (**DMCB**). Figure 4, which is shown below, shows the transformation of **TMM** into **DMCB**. **DMCB** is another well characterized[18] diradical that has a substantial preference for the triplet state. Its triplet preference, as calculated by *ab initio* methods, ranges from ~17.7 kcal/mole to ~20.2 kcal/mole depending on the level of theory and the basis set used. Like **TMM** and **MBQDM**, **DMCB** lacks the structural features



TMM **DMCB**

Figure 4. **DMCB** a derivative of **TMM**.

required to coordinate to metal centers, however, it does offer a stronger triplet preference and the opportunity to replace the vinyl hydrogen atoms with other substituents to either increase or decrease its triplet preference; fine tuning the energy levels is a desirable property of molecular magnets.[10] By replacing the sp^2 hybridized radical carbons of a non-Kekulé hydrocarbon (a hydrocarbon for which no closed shell structure can be drawn despite its organization by possessing an even number of electrons) with another spin-bearing unit, coordination to a metal center becomes possible. This methodology has yielded

a plethora of molecules and anions that possess a triplet ground state.[19]

Previous work done within our group,[10] replaced the sp^2 hybridized methyne units, CH•, with the semidione radical anion fragment $C_2O_2^{-\bullet}$. This drastic structural modification had been chosen because of its electrostatic character and on the calculated closeness between the one-electron energies of the semidione fragment and CH• SOMOs, as well as their congruent frontier orbital topologies.[10] Figure 5, which is shown below, graphically depicts the similarity between the SOMOs of **DMCB** and the proposed organic building block of $\mathbf{1}^{2-\bullet\bullet}$. According to Hückel calculations, despite the structural modification made to **DMCB**, the newly obtained dianion $\mathbf{1}^{2-\bullet\bullet}$ has frontier orbitals **A** and **B**,

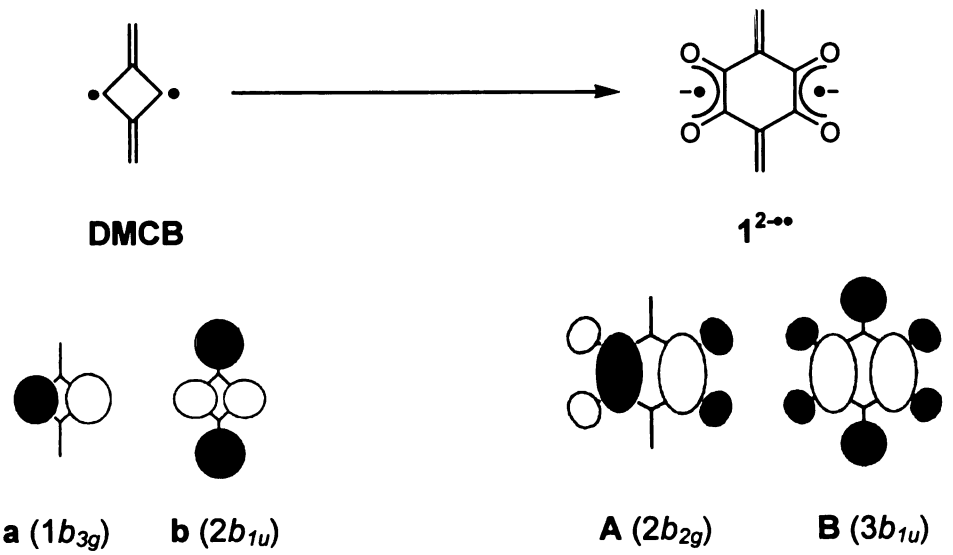


Figure 5. The transformation of **DMCB** into $\mathbf{1}^{2-\bullet\bullet}$.

which are similar to the frontier orbitals **a** and **b** in **DMCB**.[10] If $\mathbf{1}^{2-\bullet\bullet}$ is stable and the orbitals **A** and **B** are degenerate or near degenerate (see section 1.3) $\mathbf{1}^{2-\bullet\bullet}$

should possess a triplet ground state, provided that Jahn-Teller distortions are negligible or zero.

1.2: Self-assembly of $\mathbf{1}^{2-\bullet\bullet}$ and $\mathbf{2}^{2-}$

The dianion of 2,5-dihydroxy-1,4-benzoquinone, $\mathbf{2}^{2-}$, a closed shell structural analogue to $\mathbf{1}^{2-\bullet\bullet}$, has been reported to self-assemble with various trivalent metals, M^{3+}; where M=yttrium (Y), lanthanum (La), cerium (Ce), gadolinium (Gd), ytterbium (Yb), and lutetium (Lu), into two-dimensional honeycomb sheets similar to the one shown in Figure 6.[12] Despite the variation in the radii of the lanthanide ions the networks are isostructural. However, the topologies of the networks are unlike that of graphite, they are shown to undulate in a chair-like conformation similar to that of cyclohexane. Similar structures are also observed when chloranilic acid, a derivative of 2,5-dihydroxy-1,4-benzoquinone, is allowed to react with trivalent metal ions such as scandium (Sc), yttrium (Y), lanthanum (La), praseodymium (Pr), neodymium (Nd), gadolinium (Gd), terbium (Tb), ytterbium (Yb) and lutetium (Lu). In contrast, many of these polymeric networks show considerable architectural deviation producing an array of motifs.[12b]

If derivatives of $\mathbf{1}^{2-\bullet\bullet}$ were exchanged for $\mathbf{2}^{2-}$ then ferromagnetically coupled honeycomb networks might become obtainable, provided that $\mathbf{1}^{2-\bullet\bullet}$ were coordinated to a suitable trivalent metal cation. The coordinating π magnetic orbitals, between the chelating radical ligand and the metal cation, *must* not be aligned parallel, otherwise the unpaired electrons will adopt an antiferromagnetic

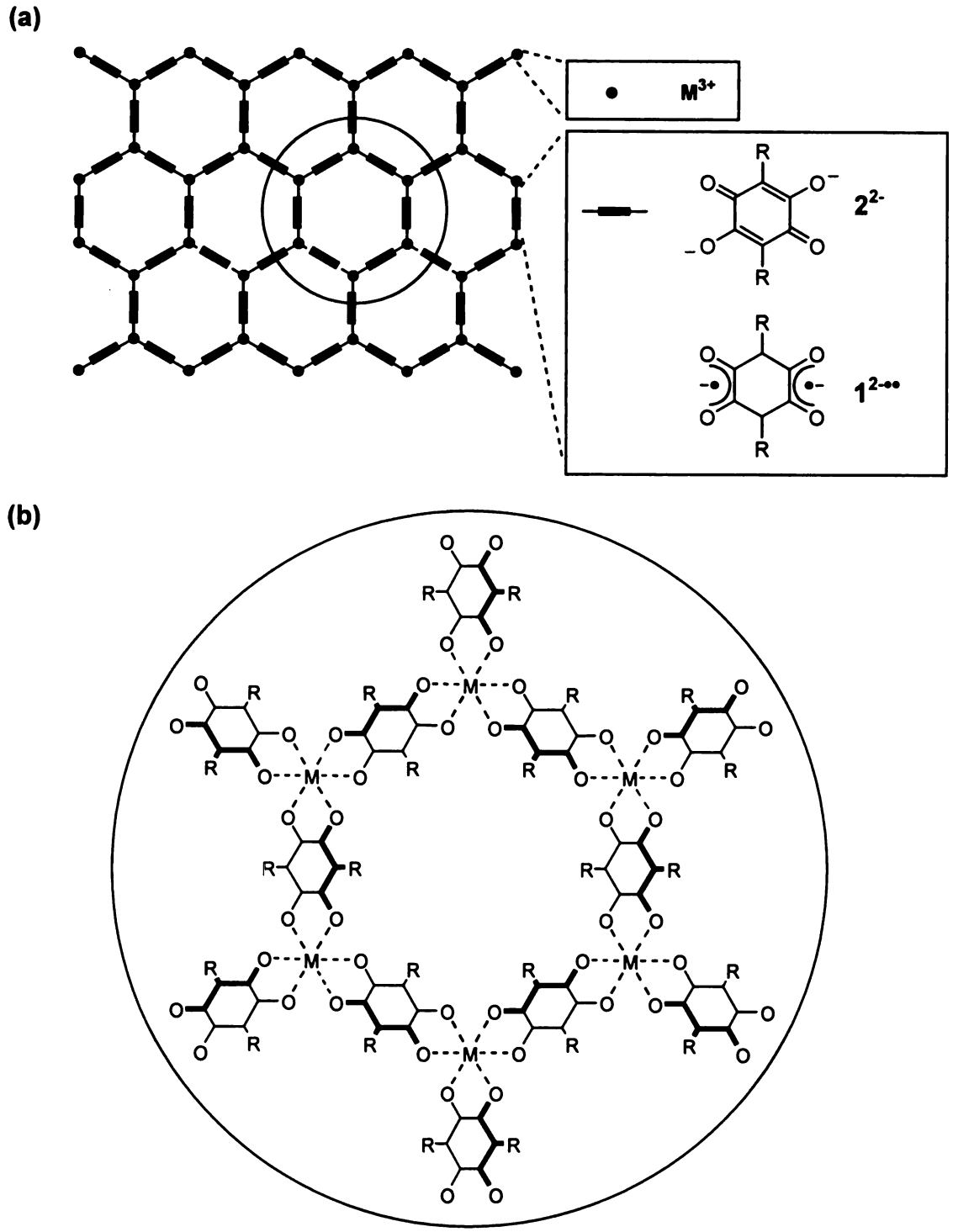


Figure 6. (a) A two-dimensional honeycomb sheet. (b) A fragment of the network showing the coordination of a trivalent metal cation, M^{3+}, and $\mathbf{2}^{2-}$ or $\mathbf{1}^{2-\bullet\bullet}$.

arrangement-a cancellation of their magnetic moments.[20] The paramagnetic semidione unit in the organic building block may give rise to ferromagnetic interactions when linked via the symmetrical octahedral coordination sphere of the trivalent metal cation-this should ensure high spin coupling. This has been demonstrated by the quartet ground states observed for the analogous gallium and aluminum complexes of *o*-semiquinones prepared by Pierpont *et al.* (see Figure 7). The *J* coupling between ligands in these two complexes are equal to 6.2 cm^{-1} (17.7 cal/mol) and 8.6 cm^{-1} (24.6 cal/mol) respectively.[20] However, antiferromagnetic or simple paramagnetic interactions may arise if the metal or its oxidation state is poorly chosen; in the case of Co^{3+} an antiferromagnetic interaction arises among the 3,6-di-*tert*-butylsemiquinone ligands with a *J* coupling equal to –39.1 cm^{-1} (-111.7 cal/mol).[21]

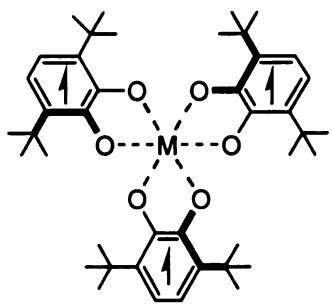


Figure 7. A tris(3,6-di-*tert*-butylsemiquinone)M complex, where M = Ga^{3+} and Al^{3+}, that exhibits a quartet ground state.

The two-dimensional honeycomb network is not just limited to molecules similar in structure to dianion $\mathbf{1}^{2-\bullet\bullet}$ but can be extended to molecules analogous

to the monoanion $1^{-\bullet}$ as well. If different metal cations or like-metal cations of different valences (+1 and +2 or +2 and +3, with counter ions in the latter case) were incorporated this would balance the charge distribution and possibly give rise to materials with an array of magnetic behaviors. This strategy of a mixed-metal system has been used to prepare honeycomb sheets consisting of oxalate (ox) anions with various transition metal ions [22] and materials exhibiting ferromagnetic behavior.[22a,b,d] It is known that a mixed-metal system can give rise to ferromagnetic interactions between the mixed-metal ions provided the magnetic orbitals are strictly orthogonal to each other.[23] For particular pairs of mixed-metal ions such as Chromium(III)-Copper(II), Chromium(III)-Nickel(II), Copper(II)-Vanadium(IV) oxide, low-spin Iron(III)-Copper(II), and Ruthenium(III)-Copper(II), a ferromagnetic spin-exchange has been experimentally confirmed by the magnetic investigations of their mixed-metal complexes.[24] In fact, within the series $\{[N(n\text{-}C_4H_9)_4][M^{2+}Cr^{3+}(ox)_3]\}_n$, M^{2+}=Mn, Fe, Co, Ni, Cu, which has been shown to be architecturally two-dimensional,[22c] ferromagnetic phase transitions have been determined at temperatures below 15 K.[22a] However, compounds of the series $\{[N(n\text{-}C_4H_9)_4][M^{2+}Fe^{3+}(ox)_3]\}_n$, where M^{2+}=Fe, Ni, are shown to behave as ferrimagnets at temperatures below 43 K and 28 K, respectively,[25] while the complex $\{[N(n\text{-}C_4H_9)_4][Mn^{2+}Fe^{3+}(ox)_3]\}_n$ reveals an antiferromagnetic transition around 50 K.[26] Therefore substituting $1^{-\bullet}$, a paramagnetic coupling ligand, in the place of the diamagnetic oxalate ligand should strengthen the interaction between transition metal centers and increase the T_c value.

Although the honeycomb sheet is a desirable target it may not produce a

ferromagnetic material with a high T_c value, because magnetic ordering through space is very weak at best. Often, materials that rely on through space spin-coupling exhibit antiferromagnetism or simple paramagnetism, though several attempts have been reported to be successfully accomplished, nitronyl nitroxides being one of them.[27] To avoid this possibility, a three-dimensional diamond-like structure that links each and every component to another should give rise to a material with the desired magnetic properties, see Figure 8, provided a suitable tetravalent metal cation is incorporated into the crystalline lattice.

In diamond each carbon is sp^3 hybridized and linked to four other sp^3 hybridized carbon atoms generating an extended three-dimensional network, adamantane is a subunit of diamond and is shown in Figure 8a. Substituting a tetravalent transition metal or lanthanide metal, M^{4+}, for each carbon atom, and replacing each carbon-carbon single bond with $\mathbf{1}^{2-\bullet\bullet}$ would potentially generate a material that exhibits ferromagnetic ordering at a relatively high T_c value. Replacing each carbon-carbon single bond with $\mathbf{2}^{2-}$ may also produce a material that exhibits ferromagnetic ordering but with a smaller T_c value than in the previous case; this is simply due to the large decrease in the number of unpaired electron spins. To date there are only a few diamond-like networks which incorporate 2,5-dihydroxy-1,4-benzoquinone or its derivatives.[12b,13] Two such structures were made from the reaction of $Y(NO_3)_3 \cdot 6H_2O$ with chloranilic acid in a methanol-chloroform-triethylamine solution and $Th(NO_3)_4 \cdot 4H_2O$ with chloranilic acid in a chloroform solution. Even these two crystals show structural distortions from the ideal geometries which are found in diamond.

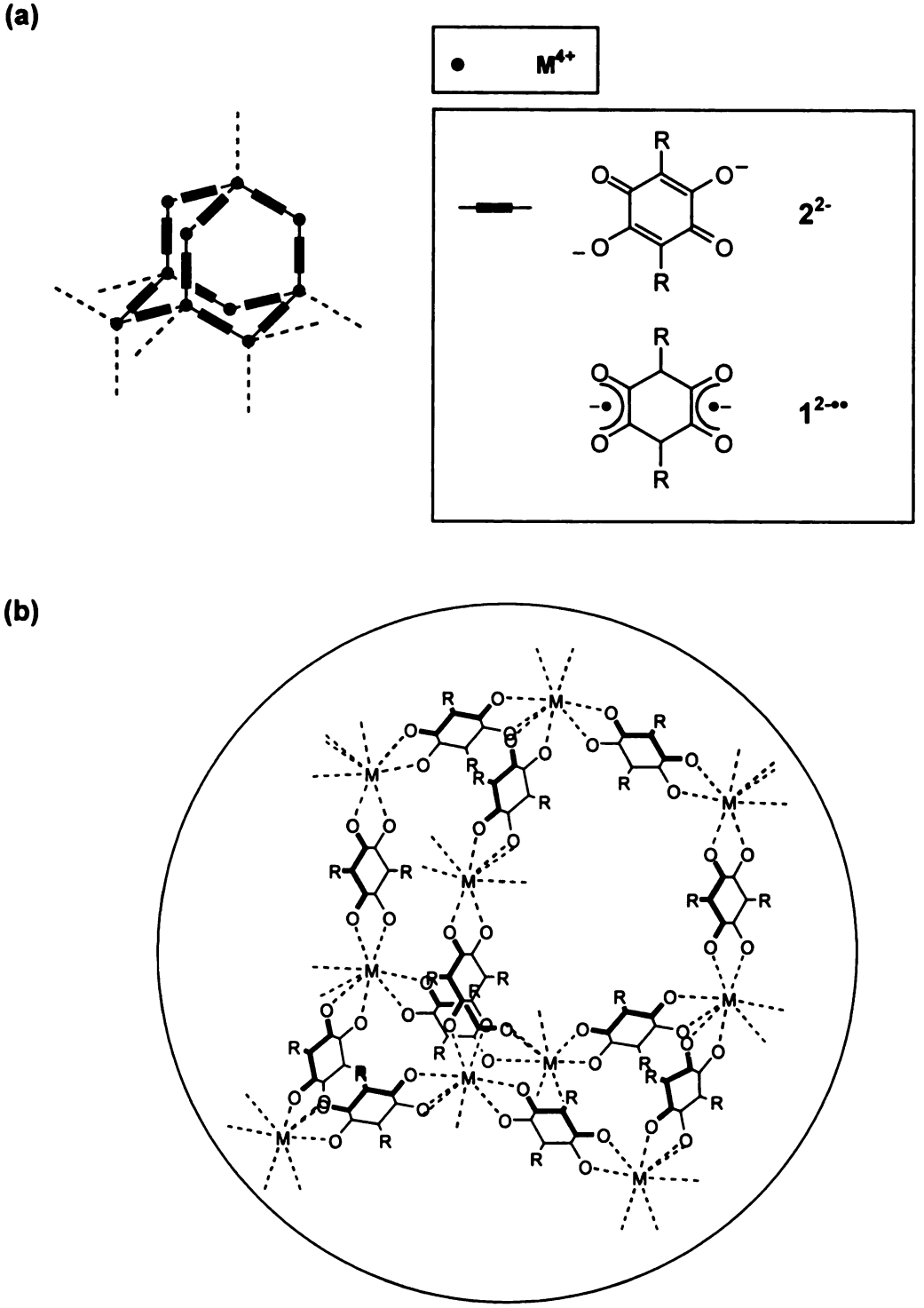


Figure 8. (a) Adamantane, a fragment of diamond. (b) A three-dimensional diamond network created by the coordination of a tetravalent transition metal cation, M^{4+}, and $\mathbf{2}^{2-}$ or $\mathbf{1}^{2-\bullet\bullet}$.

1.3: Theoretical Studies of $\mathbf{1}^{2-\bullet\bullet}$ as a Potential Building Block

As mentioned earlier, structurally modifying **DMCB** to the semiquinone diradical dianion does not significantly change the frontier orbitals and they retain the desired near degeneracy. However, unlike **DMCB** the semiquinone $\mathbf{1}^{2-\bullet\bullet}$ can be subjected to further structural modifications and the energies of the frontier orbitals of both **A** and **B** can be "fine-tuned" to optimize its preference for the triplet state. The organic spin carrier, $\mathbf{1}^{2-\bullet\bullet}$, can be structurally modified in two ways; first, the vinyl hydrogen atoms that are located on the terminal olefins can be replaced with either electron-withdrawing groups or with electron-donating groups. This should influence the energy level of the frontier orbital **B** more so than **A** because unlike **B**, orbital **A** has zero electron density on the terminal methylene carbons (see Figure 5). Since the energy of orbital **B** lies lower than that of **A**, according to Hückel and *ab-initio* calculations,[10] substituting electron-donating groups in the place of the hydrogen atoms should raise the energy level of orbital **B** and shrink the **A-B** gap.

Second, the semidione radical fragment can be coordinated to various metal centers that can vary in both size and charge. This should influence the frontier orbital of **A** more than **B**. Orbital **B** will be influenced significantly less than **A** because a large percentage of the electron density of orbital **B** lies on the methylene carbons where the metal cation would not be bound. The electrostatic interactions that arise between the charged metal cation and the semidione unit should thus lower the energy of orbital **A** more than of **B**, shrinking the **A-B** gap. When metal cations with increasing valencies (and decreasing atomic radii) are

used, the effective nuclear charge increases on the metal center creating a greater attraction for the electrons on the semidione unit thus lowering the overall energy of orbital **A**.

Several derivatives of $\mathbf{1}^{2-\bullet\bullet}$ were designed (see Figure 9) and analyzed by *ab-initio* CAS-MCSCF calculations (Complete Active Space Multi-Configurational Self-Consisted Field) with the focus being on the correlation between structural modifications and the magnitude and sign of $\Delta E_{(S-T)}$ (the singlet-triplet energy difference).[10] The species that were investigated were run at several levels of

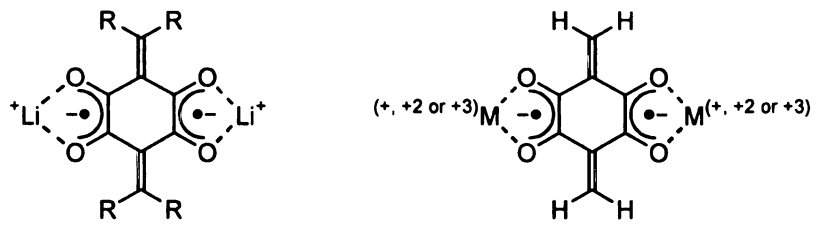


Figure 9. Derivatives of $\mathbf{1}^{2-\bullet\bullet}$ that were structurally modified through its substituents (**a**) or its coordinated metal center (**b**).

theory but only the results from the CAS(14,12) level of theory (fourteen electrons in twelve orbitals) with the 6-31+G* basis set are presented herein. In the first set of calculations the substituent R, where R = F (a weak π donor), H, OH (a strong π donor), was varied while the bound metal cation, Li^+, remained constant. In the second set of calculations the substituent was held constant-

hydrogen atoms, while the metal cation, M, and its charge changed, where M = Li^+, Be^{2+}, and Al^{3+}. In the latter cases additional fluoride ions were coordinated to the metal to balance the overall charge of the complex. Thus, the bound inorganic fragments were Li^+, BeF^+ and AlF_2^+, with the latter two in planar and tetrahedral geometries, respectively.

The initial predictions were correct; the stronger π donor raised the energy of orbital **B** and that species preferred the triplet state to the singlet state by ~4.5 kcal/mol. Similarly, when the semidione radical fragment was coordinated to the Al^{3+} cation the energy of orbital **A** decreased and that species preferred the triplet state to the singlet state by ~4.3 kcal/mol.[10] No calculations were carried out in which the species contained the hydroxyl unit as the substituent and the Al^{3+} as the metal cation. However, it has been predicted that the most optimized system would generate a preference for the triplet state to the singlet state of ~4 to 5 kcal/mol.[10] This is far less than the triplet preference of both **TMM** and **DMCB** which have preferences of ~15 kcal/mole and ~19 kcal/mol respectively. Despite the smaller preference for the triplet state, the theoretical calculations, suggest that $\mathbf{1}^{2-\bullet\bullet}$ could serve as an effective paramagnetic organic linker and, if it could be assembled into networks of appropriate topology, could generate the intended multidimensional molecular materials with tunable magnetic and electronic properties.

1.4: Synthesis of 1 as a Precursor to $\mathbf{1}^{2-\bullet\bullet}$

It is highly unlikely that **1** or $\mathbf{1}^{2-\bullet\bullet}$ would exist with vinyl hydrogen atoms or

hydroxyl groups as substituents because of potential rearrangements or reactions, i.e. keto-enol tautomerization or hydrogen atom abstraction. Therefore the aryl ring was chosen for the substituent R for several reasons. As in the structurally related triarylmethyl radical systems, the aryl substituents bring two assets: they offer steric protection against radical reactions such as dimerization or hydrogen atom abstraction; and the π conjugation allows perturbation of the energy of the frontier orbital **B** via relay of π donor/acceptors' effects on their rings. This turned out to be both an advantage and a disadvantage for the tetrone system, as will be discussed in further detail in Chapter 2. Several neutral tetrones of **1** containing various electron-donating substituents were successfully synthesized and characterized[28] by ^{1}H and ^{13}C NMR (Nuclear Magnetic Resonance), FT-IR (Fourier Transform Infrared Spectroscopy), UV/VIS/NIR (Ultraviolet/Visible/Near Infrared Spectroscopy), MS (Mass Spectroscopy), elemental analysis and X-ray crystallography. Figure 10 summarizes the synthesis of the neutral tetrones of **1**.

Disubstituted dihydroxyquinones, **2**, both chemical precursors and potential diamagnetic structured analogs of the desired tetrones, **1**, were each prepared by refluxing 2,5-dihydroxy-1,4-benzoquinone (DHQ) in a polar protic solvent (typically acetic acid-AcOH) with the appropriate diaryl methanol and a trace amount of sulfuric acid. Purification of **2** yielded the newly formed derivatives of DHQ. All these compounds, except **2B**, underwent oxidation with two equivalents of 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ) in refluxing benzene or toluene to generate a mixture of the corresponding tetrone and trione

(**2** is oxidized only once), followed by purification with an appropriate solvent or solvent mixture. Apparently, the deactivating nature of the *p*-chloro substituted aryl ring inhibited the reaction so that only a trace amount of the corresponding tetrone was formed, and that could not be easily separated from the reaction mixture. The complete synthetic details for **2A-E**, **1A**, **1C**, **1D** and **1E** are given in Chapter 2.

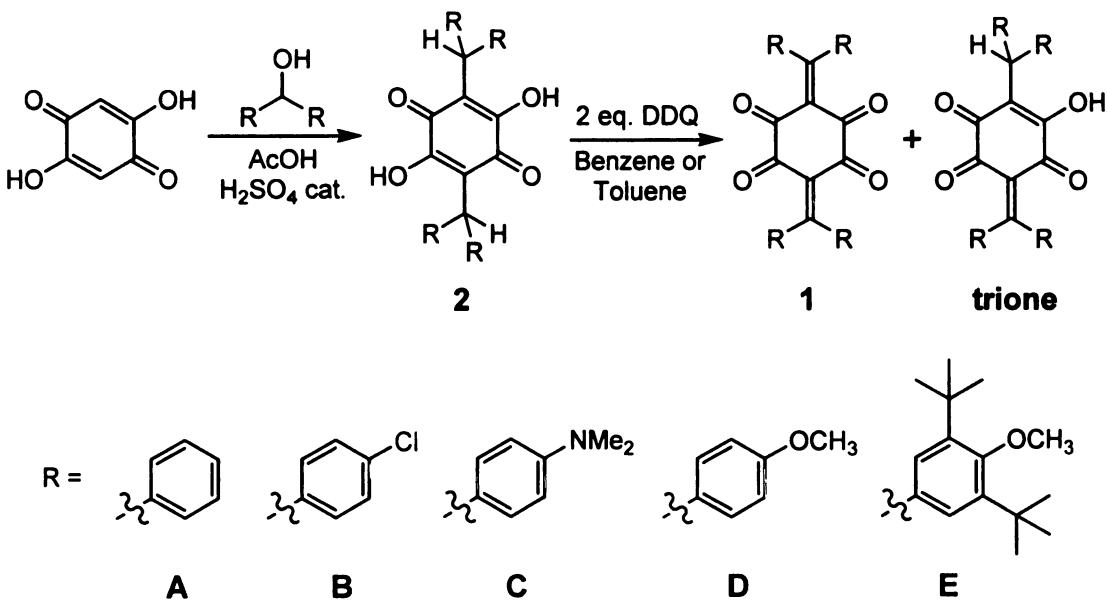


Figure 10. Synthetic scheme and structures of the dihydroxyquinones and tetrones (except **1B**) that have been prepared and characterized.

1.5: Electrochemistry of **1**

The electrochemistry of the tetrones was explored through the use of cyclic voltammetry (CV). Figure 11 presents the voltammogram of **1E** in

tetrahydrofuran (THF) as a typical example; the other tetrones produced similar voltammograms. Each tetrone, **1A-E** (except **1B**), exhibited two reduction waves $E_1°$ and $E_2°$, with corresponding oxidation waves, indicating reversibility (later confirmed by a UV/VIS experiment discussed in Chapter 2.5). The values of the reduction potentials for the tetrones, versus $E°=0.46$ V for the ferrocene/ferrocinium couple, are presented in Table 1.

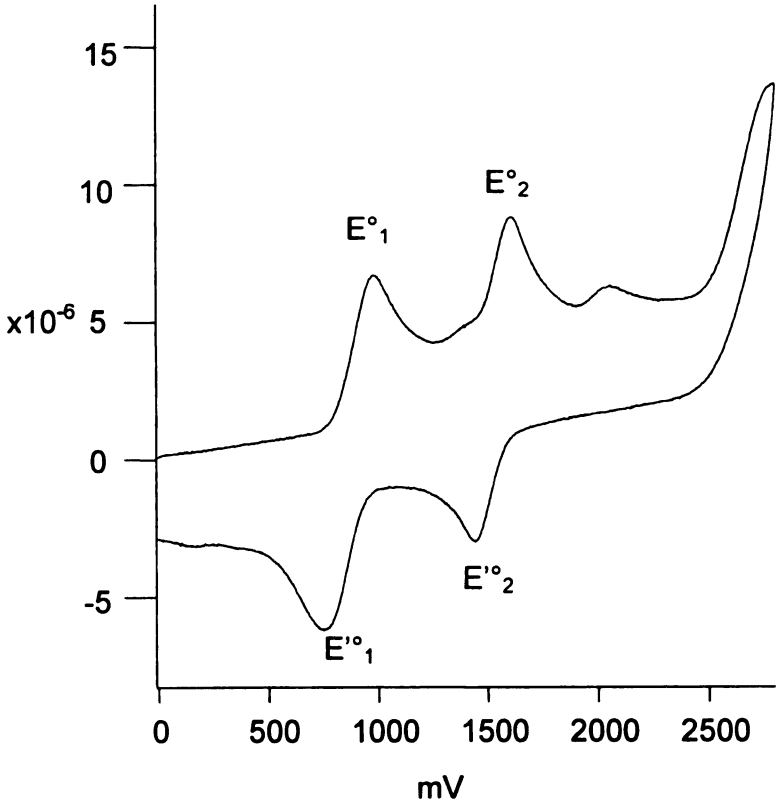


Figure 11. Typical cyclic voltammogram (CV) of the tetrones in tetrahydrofuran (THF) or *N*,*N*-dimethylformamide (DMF). The cyclic voltammogram shown is of **1E** in tetrahydrofuran.

Table 1. Reduction potentials from cyclic voltammetry of 1 mM solutions of tetrones **1A**, **1C**, **1D**, and **1E** with 0.1 M of (n-butyl)$_4$N(PF_6) as supporting electrolyte.

	$E_1°$	$E_2°$	$\Delta E_{1,2}°$
THF			
1A	-0.39	-1.10	0.71
1E	-0.57	-1.22	0.65
DMF			
1A	-0.28	-0.91	0.63
1D	-0.39	-0.96	0.57
1C	-0.78	-1.27	0.49

The monoanion monoradicals, $\mathbf{1}^{-\bullet}$, were generated by a one-electron reduction of **1** to yield the first wave, $E_1°$, in either tetrahydrofuran (THF) or *N*,*N*-dimethylformamide (DMF); due to solubility reasons. As predicted by theoretical calculations, there is a strong correlation between the reduction potentials and the aryl ring substituents' electron-donating abilities; stronger donors increase the reduction potential of the tetrones. The reduction potential of **1A** is lower in *N*,*N*-dimethylformamide (DMF) than in tetrahydrofuran (THF) as expected; presumably the relative solvation of the anion versus neutral **1A** is stronger in the more polar DMF.[28] The oxidizing abilities of **1A** and its derivatives are comparable to *p*- and *o*-benzoquinones measured under similar conditions (0.43 V and -0.47 V for *p*-benzoquinone in DMF[29] and 3,5-di-*tert*-butyl-*o*-quinone in THF[30] respectively.

The dianion diradicals, $\mathbf{1}^{2-\bullet\bullet}$, were generated by another one-electron

reduction to yield the second wave, $E_2°$. They were assigned as such because the values of the peak current, i_p, of $E_2°$ are close to those for $E_1°$. The decreasing separation between the first and second reduction waves for each tetrone suggests the two frontier orbitals become closer in energy across this series. This observation correlates with ROHF triplet calculations on model compounds of $\mathbf{1A}^{2-}$ that predict a decrease in the difference between the one-electron energies of the singly occupied frontier orbitals with increasing substituent donor abilities.[10] The electrochemistry of the tetrones suggests that the diradical dianion has an open-shell framework and provides support for the ability to "fine-tune" the organic spin carrier **1**.

The relative ease of reduction of uncoordinated **1A** and $\mathbf{1A}^{-\bullet}$ bodes well for preparation of complexes of monoradical monoanions and diradical dianions by electron transfer from metal centers to the neutral tetrone. It also ensures that like semiquinone and catecholate anions in their vast complexes. These systems will be compatible with a variety of transition or lanthanide metal cations.

1.6: UV/VIS/NIR Studies of $\mathbf{1}^{-\bullet}$ and $\mathbf{1}^{2-\bullet\bullet}$

The tetrones and their corresponding anions and dianions were subjected to UV/VIS/NIR spectroscopy to obtain further evidence regarding the energy gap between the two frontier orbitals **A** and **B**, and their responses to substituent variation. Acetonitrile solutions of the neutral tetrones of **1** were characterized with UV/VIS spectroscopy within the range of 200 nm to 1000 nm, while their reduction with *in-situ* prepared K^+C222K^- in tetrahydrofuran was monitored by

VIS/NIR (visible and near-infrared) spectroscopy in the 400-2000 nm range. Such systems, like the tetrones, which possess conjugated electron-donor and electron acceptor moieties often exhibit low energy charge-transfer transitions, which are currently investigated in the fields of nonlinear optics,[31] molecular electronics[32] and artificial photosynthetic models.[33] Each neutral tetrone shows both intense visible and ultraviolet absorption bands (see Table 2).[28] A bathochromic shift takes place in the visible region and is attributed to the increase in the electron-donating ability of the substituent in the order of –H (**1A**), –OMe (**1D**), $-NMe_2$ (**1C**). These values, compared with those observed for *p*-substituted diphenylmethyl carbocations (λ_{max} = 440 nm (–H), 507 nm (–OMe), 610 nm ($-NMe_2$)),[34] are in good agreement. The extinction coefficient of **1C** was found to be ~10^5 giving it an intense blue hue while the other tetrones had weaker absorptions and appear to be reddish-purple in appearance.

The VIS/NIR spectra of the monoradical monoanions and diradical dianions of the neutral tetrones were prepared by washing a solution composed of the tetrone, cryptand c(2.2.2)-4,17,3,16,21,24-hexaoxa-1,10-diazabicyclo-[8.8.8]hexacosane and tetrahydrofuran over a potassium metal mirror (the cryptand combines with the potassium metal to first form the complex $[K@c(2.2.2)]^+K^-$).[35] One purpose for the introduction of the cryptand is that it encapsulates the potassium metal preventing the interaction of the potassium metal cation with the reduced tetrone thus simplifying the UV/VIS or VIS/NIR spectrum. Monoradical monoanions of $\mathbf{1A}^{-\bullet}$, $\mathbf{1D}^{-\bullet}$, and $\mathbf{1E}^{-\bullet}$ have a wine-red to purple appearance to the eye, while that of $\mathbf{1C}^{-\bullet}$ appears aqua-green. In addition

to the visible transitions, all monoradical monoanions were found to possess a broad absorption band in the near infrared region. Like the visible bands of the neutral tetrones, these transitions' energies decrease with increasing electron-donating power of the aryl substituents; peak maxima fall at 1000, 1100, 1150 and 1500 nm for **1A**$^{-\bullet}$, **1D**$^{-\bullet}$, **1E**$^{-\bullet}$, and **1C**$^{-\bullet}$, respectively.[28] The correlation of the transition energy with the molecular structure suggests that the electron is excited from an orbital conjugated to the phenyl rings into another one that is relatively less affected by substitution. This correlation and the low energy of the transition would be in good agreement with a SOMO to LUMO electron excitation. In summary, electronic spectroscopic results, supported by electrochemistry data (*vide supra*), further indicates that the frontier orbital gap can be tuned by substituent variation at the para sites on the phenyl rings.

The diradical dianions' main absorptions in the VIS/NIR spectra are centered around 800 nm and are found to be much less affected by the phenyl rings' substituents, therefore all of the diradical dianions appear emerald-green. To confirm that the original tetrone framework was preserved through the two-electron reduction, a conproportionation reaction was setup whereby a solution of the diradical dianion was prepared by reduction of **1E** with two equivalents of cesium metal in tetrahydrofuran, then this solution was combined with an additional equivalent of **1E** and the optical spectrum was recorded. The spectrum was identical to that of the directly prepared monoradical monoanion, **1E**$^{-\bullet}$, but with an absorption intensity that was twice the original, demonstrating the reversibility of the electron addition and the robustness of the underlying

structure. More details concerning the reversibility of the tetrone system and the conproportionation reaction are discussed in Sections 2.4 and 2.5.

Table 2. Electronic Absorption Maxima of Tetrones **1A**, **1C**, **1D**, and **1E** and their monoradical monoanions, $\mathbf{1}^{-\bullet}$, and diradical dianions, $\mathbf{1}^{2-\bullet\bullet}$.

Tetrone	λ_{max} (nm)		
	Neutral (MeCN)	Monoanion (THF)	Dianion (THF)
1A	354	430 525 1000	485 800
1D	498 580 (shoulder)	530 600 (shoulder) 1100	745
1E	480	525 610 (shoulder) 1150	770
1C	602 674	425 635 1500	770

1.7: EPR/ENDOR Studies of $\mathbf{1}^{-\bullet}$ and $\mathbf{1}^{2-\bullet\bullet}$

Monoanions and dianions of **1** were analyzed by EPR/ENDOR to confirm their structure, examine their stability and determine the nature of the diradical dianions' preference for a ground state singlet versus a ground state triplet. Both EPR (Electron Paramagnetic Resonance) and ENDOR (Electron Nuclear Double Resonance) have been used extensively to map an electron's delocalization over the entire molecule and probe the strength of the spin-spin interactions of

unpaired electrons in polyradical systems.[36] Samples of the monoradical monoanions and diradical dianions for spectroscopic studies were prepared by the reduction of the tetrones, dissolved or suspended in tetrahydrofuran, with alkali metal mirrors. The 'free anions' were prepared by incorporating the macrobicyclic cryptand[2.2.2] (C222), which has a high binding affinity for potassium and would ensure that free ions would be observed spectroscopically by eliminating intermolecular spin-spin interactions and the formation of aggregates. Each of the species, $\mathbf{1}^{-\bullet}$ and $\mathbf{1}^{2-\bullet\bullet}$, generated strong EPR signals with g-values around 2.0030,[28] which is characteristic for organic radicals. Monoanion solutions prepared for EPR/ENDOR analysis were discovered to be stable at room temperature without noticeable decomposition for extended periods, under anaerobic conditions with scrupulously dried solvents, except for $\mathbf{1A}^{-\bullet}$ which has a half-life of ~1 hr. However, all monoradical monoanions were stable, indefinitely, at temperatures below -20°C.[28] Each of the species examined generated an ENDOR spectrum confirming the monoradical character of the system and its delocalization throughout the entire molecules π conjugated system. The ^{1}H hyperfine coupling constants and their relative signs were determined and are presented later (Chapter 2). Results from the analysis of the monoradicals were promising until the nature of the dianions ground state was explored.

The diradical dianions failed to show any signals that would ordinarily be characteristic of a triplet diradical, such as a broad triplet signal or a half-field transition peak. The ^{1}H hyperfine coupling constants were determined to be

identical to those of the monoanion except that the magnitudes of the measured values were twice as large with only half as many protons.[28] The results from the EPR and ENDOR experiments were puzzling, in the case of the diradical dianion, and were thought to arise from aggregates in the tetrahydrofuran solution or the decomposition of the diradical dianion itself; both of which turned out to be untrue. Experiments by Dr. Vanessa McCaffrey and myself were carried out to determine the true nature of the dianion and why the dianion generated the EPR and ENDOR spectra that were observed (see Chapter 2). A more detailed discussion of all the EPR and ENDOR results is presented there as well.

1.8: X-ray Crystallographic Studies of **1**

Single crystals of the neutral tetrones, **1** and **1C**, were grown from hot solutions of acetonitrile (MeCN) and dimethylsulfoxide (DMSO), respectively, and were found to be in agreement with the infrared (IR) and nuclear magnetic resonance (NMR) spectroscopic data.[28] Crystals of the neutral tetrones **1D** and **1E** were unattainable except for the serendipitous rearrangement of **1E** in a solution of tetrahydrofuran (discussed in Chapter 2). Molecules of **1A** crystallized into π stacks with an intermolecular separation equal to 4.61 Å, while those of **1C** crystallized as blocks of **1C**•$(DMSO)_3$ and were found to be arranged into sheets with relatively short intermolecular contacts (3.256 Å and 3.260 Å) between two of their carbonyl oxygen atoms and nitrogen atoms of two neighboring tetrone molecules. The tetrone **1C** may pack differently than that of **1A** because of its tendency to exist as a Zwitterion, whereby there is a transfer of charge between

the electron-donating amine nitrogen atoms and the electron-accepting oxygen atoms in the carbonyl moieties. The crystallographic results also revealed that the "core framework" (3,6-dimethylene-1,2,4,5-tetraoxocyclohexane ring), of **1A** and **1C**, had puckered into a chair-like conformation and had severe distortions with OCCO dihedral angles ranging from 20.2° to 26.9°[28] and the phenyl rings twisted out-of-plane in a manner resembling a propeller. The puckering of the tetrone core and the twisting of the phenyl rings most likely arise from electrostatic interactions between the electron densities localized on the exocyclic carbon-oxygen double bonds. The flexibility of the core framework and the twisting of the phenyl rings, which can be seen in two of the neutral tetrones, may have undesirable effects, because Jahn-Teller distortions may lower the preference for the diradical dianion from having a ground state triplet to one that prefers a ground state singlet.

1.9: Conclusions

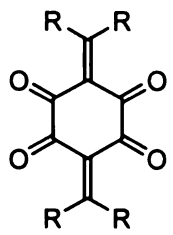
Theoretical and spectroscopic chemical studies of **1** suggest the potential for $\mathbf{1}^{-\bullet}$ and $\mathbf{1}^{2-\bullet\bullet}$ to serve as "tunable" organic spin-carrying units that would self-assemble into multi-dimensional networks giving rise to magnetic materials with desirable properties. The lack of a triplet EPR signal for $\mathbf{1}^{2-\bullet\bullet}$ is not desirable but not surprising either since theoretical calculations suggest that small multiply charged metal centers must be used in order to achieve the maximum preference for a ground state triplet. Tetrones of **1** can easily be reduced, in a reversible reaction, with alkali metal mirrors to afford both the monoradical

monoanion, $\mathbf{1}^{-\bullet}$, and the diradical dianion, $\mathbf{1}^{2-\bullet\bullet}$. If solutions of the monoradical monoanion or diradical dianion generated crystals suitable for X-ray analysis, this information coupled with that from EPR spectroscopy and SQUID magnetometry experiments could provide insight into the spin-spin interactions within our own system and expand our understanding of magnetism as a general natural phenomenon.

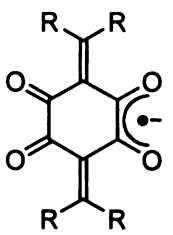
CHAPTER 2

SUCCESSES AND DISAPPOINTMENTS

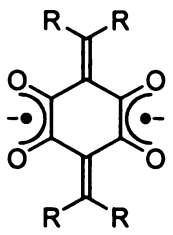
2.1: Introduction



1

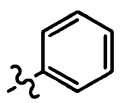


$1^{-\bullet}$

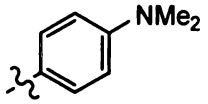


$1^{2-\bullet\bullet}$

R =



A



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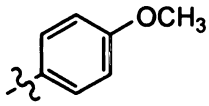

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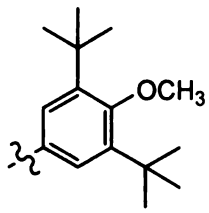


E

Tetrones of **1** were synthesized and studied for their potential to serve as paramagnetic organic linkers in organometallic magnetic materials. Of the four tetrones synthesized only **1E** possessed the potential to expand beyond the theoretical and chemical studies and into potential applications. **1A** could not be used because of its tendency to decompose or form dimers through a radical reaction at the *para*- position of the phenyl rings like in Gomberg's original triphenylmethyl radical work.[37] In addition to **1A**, **1C** and **1D** could not be used due to their insolubility in suitable polar aprotic solvents. The most likely reason

for the insolubility of **1C** are the four tertiary dimethylamino nitrogen atoms, which form resonance structures with the phenyl rings producing a species that is highly ionic in character and too polar for aprotic organic solvents to dissolve appreciably.[38] There is strong evidence for this in the crystal structure of **1C** where the dimethylamino nitrogen atoms are nearly planar and the nitrogen atoms have close contacts with neighboring tetrone oxygen atoms in the core, which more than likely bear the negative charge from an appropriately drawn resonance structure. No crystal structure of the neutral tetrone **1D** was obtained and it too lacked the ability to dissolve appreciably in organic solvents for reasons similar to **1C**.

1E was synthesized (see Figure 12 in section 2.2) to address the solubility and aggregation problems observed in **1A**, **1C**, and **1D**. The bulky *t*-butyl groups were added for several reasons; they have been known to protect atoms carrying unpaired electrons from reacting,[39] by interfering with aggregation that could occur through intermolecular π-π stacking between phenyl rings from different tetrone molecules. The *t*-butyl groups would also increase the molecules' overall solubility in organic solvents and provide a way to further influence the energy of orbital **B** by raising the electron donating ability of the phenyl ring into the methylene carbon site. Despite the enhancements made by the addition of the *t*-butyl groups to the tetrone system they may also turn out to have been one of the major sources for the failure of tetrone **1E** in forming extended networks. This will be discussed further in later chapters.

2.2: Synthesis of 1E

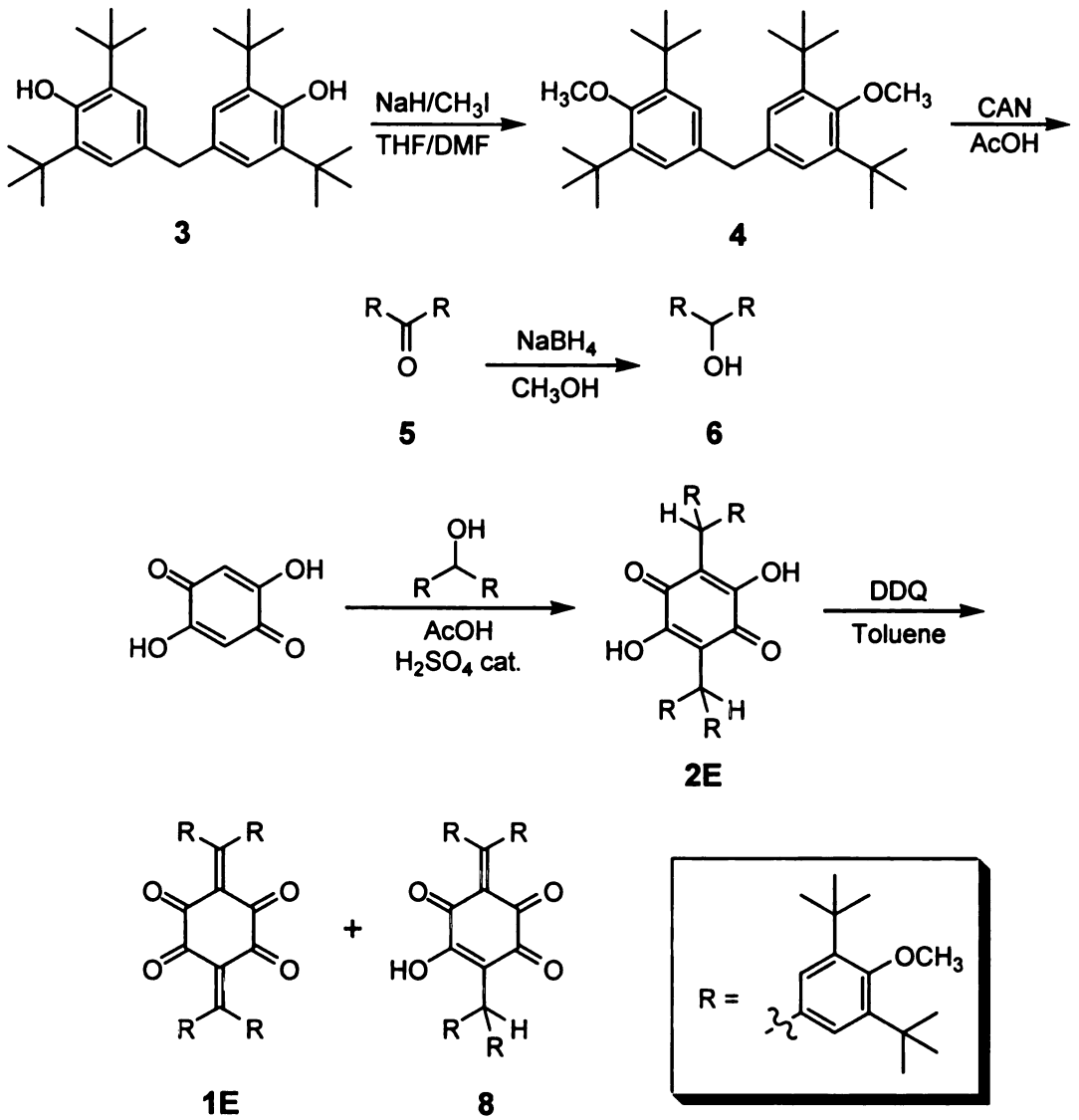


Figure 12. Synthesis of tetrone **1E**.

The synthesis of **1E** follows the synthetic scheme outlined in Figure 12 and began with the commercially available reagent, **3**, in a scalable five-step

procedure that utilized some of the most fundamental organic synthetic transformations. Compound **3** (*3,5,3',5'*-tetra-*tert*-butyl-*4,4'*-dihydroxydiphenylmethane) generated the Williamson ether product **4** (*3,5,3',5'*-tetra-*tert*-butyl-*4,4'*-dimethoxydiphenylmethane) upon treatment with a sodium hydride and methyl iodide mixture in a solution that consisted of tetrahydrofuran (THF) and *N,N*-dimethylformamide (DMF) in approximately one hour by a known procedure that methylated sterically hindered phenols.[40]

The benzylic position in the bis methylated product **4** was then oxidized in glacial acetic acid with ceric (IV) ammonium nitrate ($Ce(NH_4)_2(NO_3)_6$) otherwise known as CAN to form the benzophenone **5** (*3,5,3',5'*-tetra-*tert*-butyl-*4,4'*-dimethoxydiphenylmethanone).[41] Following this oxidative step was a standard borohydride reduction which converted the carbonyl **5** into the corresponding alcohol **6** (*3,5,3',5'*-tetra-*tert*-butyl-*4,4'*-dimethoxydiphenylmethanol). The bisphenylmethanol **6** then underwent an acid catalyzed double condensation reaction with 2,5-dihydroxy-1,4-benzoquinone (DHQ) in glacial acetic acid to generate **2E** (3,6-bis(bis(3,5-di-*tert*-butyl-4-methoxyphenyl)methyl)-2,5-dihydroxy-1,4-benzoquinone).

The last synthetic step proved to be the most challenging and provided the lowest yield among all of the synthetic steps due to purification difficulties. The final synthetic transformation was the conversion of **2E** into the tetrone **1E** (3,6-bis(bis(3,5-di-*tert*-butyl-4-methoxyphenyl)methylene)-1,2,4,5-tetraoxocyclohexane) via a double oxidation with 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ) in dry toluene under a nitrogen atmosphere. Although two

equivalents or a slight excess of 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ) was used **1E** was accompanied by product **8** and several others which were not isolated or identified. A weak and broad peak centered near 3450 cm^{-1} in the IR indicated the presence of an –OH group, most likely do to the trione **8** or unreacted starting material **2E**. It has been found that it is absolutely necessary for the impurities to be removed for they tend to interfere with the reduction of **1E** to $\mathbf{1E}^{-\bullet}$ and $\mathbf{1E}^{2-\bullet\bullet}$. Removal of the impurities as to obtain pure **1E** can be accomplished through subsequent recrystallizations with a toluene / *n*-octane mixture. The difficulty in the purification of **1E** might arise from a charge transfer complex that may form between **1E** and 2,3-dichloro-5,6-dicyano-1,4-dihydroxybenzene or the similarities in polarities among the species produced. The subsequent recrystallizations are certainly the cause for the low yields observed.

After the synthesis of **1E** has been completed the final product must be handled with some care as to avoid decomposition. The microcrystalline precipitate **1E** has been found to be stable in air for an extended period of time, approximately one year, provided that it was kept dry. However, slow decomposition does occur with oxygen. The complete decomposition of **1E** (see section 2.3) occurs within one month if left dissolved in an organic solvent such as tetrahydrofuran (THF) that has been exposed to air. The decomposition is dramatically slowed (by a few months) if an organic solvent such as dichloromethane (CH_2Cl_2), chloroform ($CHCl_3$) or toluene was used. This was discovered while attempting to grow crystals of **1E** suitable for X-ray analysis

(despite repeated attempts and variations in methodology crystals of **1E** still remains elusive). The decomposition of **1E** was a disappointment but not unexpected due to the electron deficiency of the cyclohexane core ring. One of the desired properties that any organic building block should posses is the ability to resist chemical reactivity with the atmosphere otherwise their potential applications are significantly reduced.

Experimental Section

All melting points were determined on a Thomas-Hoover melting point apparatus and are uncorrected. The ^{1}H NMR spectra and ^{13}C spectra were obtained at 300 MHz and 75.5 MHz respectively on a Varian GEMINI 300 spectrometer. The ^{1}H NMR signals were referenced to the residual proton resonances in the deuterated solvents: $CDCl_3$ (s, δ7.24) and $Si(CH_3)_4$ (s, δ0.00). The ^{13}C signals were referenced to that of the deuterated solvent: $CDCl_3$ (t, δ77.0). Infrared analyses were performed with an ATI Mattson Genesis Series FT-IR on KBr pellets containing 5% of the desired material. Elemental Analyses were performed with a Perkin Elmer 2400 Series II CHNS/O Analyzer. Samples submitted for mass analysis were introduced into a VG Trio 1 mass spectrometer by a direct probe inlet.

The reactions were carried out under a nitrogen atmosphere or with a drying tube. The solvents tetrahydrofuran and toluene were scrupulously dried by refluxing them with benzophenone and a sodium / potassium amalgam. Octane was dried by either the method mentioned above or by refluxing it with

calcium hydride (CaH_2). The remaining solvents and reagents were used as supplied (Aldrich or Fluka).

(3,5,3',5'-tetra-*tert*-butyl-4,4'-dimethoxydiphenylmethane)[40] 4:

Approximately 4.25 g (1.00 mmol) of **3** (424.667 g/mole) was added to a vigorously stirred solution comprised of 50.0 mL of tetrahydrofuran (THF), 5.0 mL of *N*,*N*-dimethylformamide (DMF) and 5.0 mL of methyl iodide (CH_3I), ρ = 2.279 g/mL, in a 150 mL roundbottom flask. To this solution 0.5 g (2.10 mmol) of sodium hydride (NaH), which had been washed with dry toluene, was slowly added to the reaction mixture as to avoid the rapid evolution of hydrogen gas. When frothing ceased a condenser and a drying tube filled with calcium hydroxide, $Ca(OH)_2$, was fixed to the roundbottom and the solution allowed to reflux for 24 hours. The solvents were then evaporated under vacuum.

The crude yellow product was then extracted with 100 mL of diethyl ether (Et_2O) and 50 mL of deionized water to remove the sodium iodide (NaI). The organic layer was then washed with three portions of deionized water, dried over magnesium sulfate ($MgSO_4$), filtered, and then evaporated to dryness leaving a tacky yellow residue. The yellow residue was recrystallized from absolute ethanol (EtOH) to give **4** (3.85 g, 0.85 mmol) in 85% yield as a fluffy white powder: melting point range = 147°C – 148°C (lit.[42] m.p. 158°C – 159°C); ^{1}H NMR δ($CDCl_3$ w/$Si(CH_3)_4$ (TMS), 300 MHz): δ1.40 (s, 36 H), δ3.68 (s, 6 H), δ3.87 (s, 2 H), δ7.04 (s, 4 H); FTIR: KBr, ν = 3067.68 cm^{-1}, 2959.34 cm^{-1}, 2914.60 cm^{-1}, and 2869.25 cm^{-1} (C–H), ν = 1218.64 cm^{-1} and 1007.05 cm^{-1} (C–

O–C). For the complete ^{1}H, ^{13}C and IR spectra see Appendices A1, A2 and B1 respectively.

(3,5,3',5'-tetra-*tert*-butyl-4,4'-dimethoxydiphenylmethanone)[41] 5:

A mixture of 35.0 g (7.73 mmol) of **4** (452.72 g/mol) and 170.0 g (31 mmol) $Ce(NH_4)_2(NO_3)_6$ (CAN) in 800 mL glacial acetic acid (CH_3CO_2H) was vigorously stirred and heated to reflux. After 5 hours, the mixture was then poured onto ice and then extracted with three 200 mL portions of diethyl ether (Et_2O). The combined ether extracts were then neutralized with several washings of a saturated sodium bicarbonate ($NaHCO_3$) solution, dried with magnesium sulfate ($MgSO_4$), filtered, and then evaporated, leaving behind a viscous orange residue. Recrystallization of the orange tar from methanol (CH_3OH) gave **5** (27.1 g, 5.81 mmol) in 75% yield as pale yellow crystals: melting point range = 158.5°C – 159.5°C (lit. mps. 165°C – 167°C[41a] and 165°C[43]); ^{1}H NMR $\delta(CDCl_3$, 300 MHz): δ1.43 (s, 36 H), δ3.73 (s, 6 H), δ7.72 (s, 4 H); FT-IR: KBr, ν = 1645.8 cm^{-1}(C=O); *m/z* 466 (M^+); Anal. Calcd for $C_{31}H_{46}O_3$: C, 79.78%; H, 9.93%. Found: C, 79.72%; H, 10.02%. For the complete ^{1}H, ^{13}C and IR spectra see Appendices A3, A4 and B2 respectively.

(3,5,3',5'-tetra-*tert*-butyl-4,4'-dimethoxydiphenylmethanol)[28] 6:

A stirred suspension comprised of 50 mL of methanol (CH_3OH) and 0.900 g (0.19 mmol) of **5** (466.70 g/mol) was added to a chilled Erlenmeyer flask. The reaction mixture began to froth after the slow addition of 1.122 g (2.97 mmol) of

sodium borohydride ($NaBH_4$). The mixture was then allowed to warm to ambient temperature, after vigorous frothing had ceased, producing a homogeneous solution. The solution was then refluxed for 1 hour at which it was then poured onto 250 mL of ice water. The resultant creamy white solution was extracted with three 150 mL portions of dichloromethane (CH_2Cl_2) yielding a lime green solution. The extracts were combined, dried with magnesium sulfate ($MgSO_4$) and filtered. The solvent was removed and the precipitate was recrystallized from a small volume of hexanes leaving **6** (0.562 g, 0.12 mmol) in 62% yield as white crystals: melting point range = 119.0°C – 120.0°C; ^1H NMR $\delta(CDCl_3$ w/$Si(CH_3)_4$ (TMS), 300 MHz): δ1.40 (s, 36 H), δ2.15 (d, 3.3 Hz, 1 H), δ3.67 (s, 6 H), δ5.73 (d, *J*=3.3 Hz, 1 H), δ7.23 (s, 4 H); FT-IR: KBr, ν = 3573.47 cm^{-1} (strong and sharp, –OH); *m/z* 468 (M^+); Anal. Calcd for $C_{31}H_{48}O_3$: C, 79.44%; H, 10.32%. Found: C, 79.60%; H, 10.34%. For the complete ^1H, ^{13}C and IR spectra see Appendices A5, A6 and B3 respectively.

(3,6-bis(bis(3,5-di-*tert*-butyl-4-methoxyphenyl)methyl)-2,5-dihydroxy-1,4-benzoquinone)[28] 2E:

A mixture of 1.4 g (1.0 mmol) of 2,5-dihydroxy-1,4-benzoquinone (DHQ) and 9.37 g (2.0 mmol) of **6** (468.72 g/mol) in 100 mL of glacial acetic acid (CH_3CO_2H), containing a few drops of concentrated sulfuric acid (H_2SO_4), was stirred continuously at reflux. After 1 hour, 50 mL of deionized water was then added to the solution and allowed to return to ambient temperature. The precipitate was then filtered and washed with several small portions of water

followed by cold methanol (CH_3OH). The crude product can be recrystallized from a variety of polar protic solvents including glacial acetic acid and/or an alcohol-water mixture producing **2E** (9.47 g, 0.91 mmol) in 91% yield as orange-yellow crystals: melting point range = 239.5°C – 240.5°C; ^1H NMR $\delta(CDCl_3$, 300 MHz): δ1.33 (s, 72 H), δ3.65 (s, 12 H), δ5.48 (s, 2 H), δ7.08 (s, 8 H), δ7.92 (s, 2 H); FT-IR: KBr, ν = 3311.19 cm^{-1} (moderate and broad, –OH), ν = 1697.06 cm^{-1} (moderate and sharp, C=O); Anal. Calcd for $C_{68}H_{96}O_8$: C, 78.42%; H, 9.29%. Found: C, 77.89%; H, 9.32%. For the complete ^1H, ^{13}C and IR spectra see Appendices A7, A8 and B4 respectively. Yellow crystals of **2E** suitable for X-ray analysis were grown by slow evaporation of an acetone solution from a loosely capped 5.0 mL vial. The structure is shown in Figure 13.

(3,6-bis(bis(3,5-di-*tert*-butyl-4-methoxyphenyl)methylene)-1,2,4,5-tetraoxocyclohexane) 1E:

A mixture of 4.6 g (0.44 mmol) of **2E** (1041.50 g/mol) and 2.0 g (0.88 mmol) of 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ) in 50 mL of dry toluene was stirred under a nitrogen atmosphere. After 6 hours of reflux the mixture was hot filtered, to remove 2,3-dichloro-5,6-dicyano-1,4-dihydroxybenzene, then reduced leaving behind a dark red almost black tarry mass. The remaining tarry mass was triturated with two 100 mL portions of hexanes and decanted. The remaining precipitate was recrystallized from a toluene-*n*-octane mixture producing **1E** (2.75 g, 0.27 mmol) in 60% yield as dark brick red microcrystalline aggregates of various sizes and colors. i.e. the larger

the aggregate the darker the shade of red and the smaller the aggregate the lighter the shade of red: melting point ≥ 300°C (dec.); ^{1}H NMR δ($CDCl_3$, 300 MHz): δ1.35 (s, 72 H), δ3.71 (s, 12 H), δ7.16 (s, 8 H); FT-IR: KBr, ν = 1652.70 cm^{-1} (moderate and sharp, C=O); Anal. Calcd for $C_{68}H_{92}O_8$: C, 78.72%; H, 8.94%. Found: C, 77.73%; H, 9.02%. For the complete ^{1}H, ^{13}C and IR spectra see Appendices A9, A10 and B5 respectively.

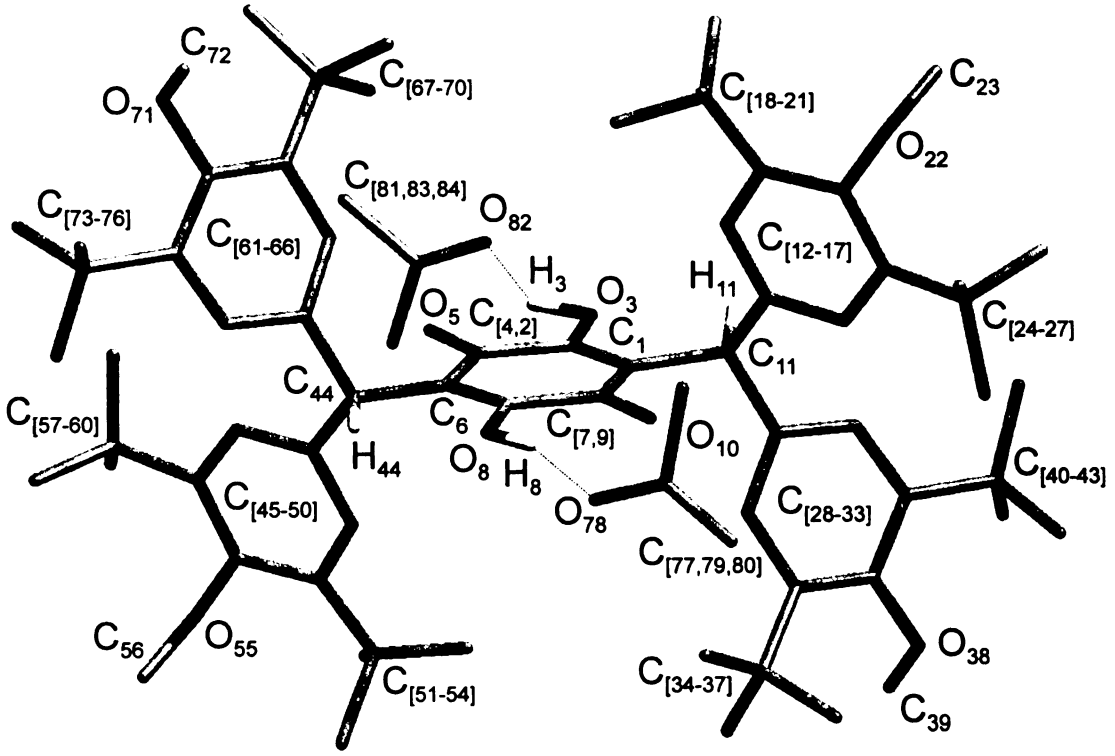


Figure 13. The crystal structure of **2E**. All non-essential hydrogen atoms have been removed for clarity.

The following synthetic compounds were first devised and characterized by Andrzej Misiolek and then repeated by Dr. Vanessa McCaffrey and myself.

(3,6-bis(bisphenyl)methyl)-2,5-dihydroxy-1,4-benzoquinone)[28] 2A:

A mixture of 2.0 g (14.3 mmol) of 2,5-dihydroxy-1,4-benzoquinone (DHQ), 5.5 g (29.9 mmol) of diphenylmethanol and 0.5 mL of concentrated H_2SO_4 were refluxed for one hour in 20 mL of glacial acetic acid and then left at room temperature to cool. The crystalline mass obtained was crushed with a spatula and vacuum-filtered through a glass frit. The collected yellow crystals were washed with a small amount of acetic acid and then thoroughly washed with water. The crystals were dried overnight at 90°C in air giving 6.5 g (96.5%) of orange powder. The purity of the resulting **2A** proved to be sufficient for the next step. It could, however, be recrystallized from 20 mL of acetic acid giving 6 g (89% yield) of the product: melting point range = 190°C – 192°C; ^{1}H NMR $\delta(CDCl_3$, 300 MHz): δ5.69 (s, 1 H), δ7.2-7.3 (m, 10 H), δ7.95 (s, 1 H); ^{13}C NMR $\delta(CDCl_3$, 75.5 MHz): δ=45.6, 117.3, 126.8, 128.4, 129.0, 140.6 (broadening caused by rapid proton shifts made it difficult to detect signals of C=O and C-OH carbons).

(3,6-bis(bisphenyl)methylene)-1,2,4,5-tetraoxocyclohexane)[28] 1A:

A mixture of 10.0 g (21.2 mmol) of **2A**, 10.0 g (44.1 mmol) of 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ) and 70 mL of dry benzene were gently refluxed with stirring for 24 hours under a nitrogen atmosphere. The solvent was

evaporated and the solid washed with 200 mL of diethyl ether (Et_2O) and air dried. The resulting red powder was dissolved in 200 mL of $CHCl_3$ and filtered. The separated solid, reduced DDQ, was washed with more $CHCl_3$ and combined chloroform solutions were concentrated to about 25 mL. When 300 mL of diethyl ether (Et_2O) was poured into the concentrate, after a while, red crystals of crude **1A** separated. The product was purified by recrystallization from hot acetonitrile, giving 2-10% of the product: melting point range = 288 °C – 290°C (dec.); ^1H NMR δ($CDCl_3$, 300 MHz): δ7.2-7.6 (m, 20 H); ^{13}C NMR δ($CDCl_3$, 75.5 MHz): δ=128.1, 132.0, 132.5, 140.0, 180.1, 186.6; Anal. Calcd for $C_{32}H_{20}O_4$: C, 82.04%; H, 4.30%. Found: C, 81.04%; H, 4.55%.

(3,6-bis(bis(4-methoxyphenyl)methyl)-2,5-dihydroxy-1,4-benzoquinone)[28] 2D:

To the well stirred refluxing solution of 2.0 g (14.3 mmol) of 2,5-dihydroxy-1,4-benzoquinone (DHQ) in 100 mL of glacial acetic acid containing a few drops of concentrated H_2SO_4 was added 7.0 g (28.7 mmol) of bis(4-methoxyphenyl)methanol. The solution first became red and after a while a yellow product precipitated. The suspension obtained was refluxed for an additional 15 minutes, then cooled to room temperature and filtered. The collected powder was washed first with acetic acid, then water, and dried giving 8.0 g (13.5 mmol) 94.4% of crude product which was recrystallized from dioxane: melting point range = 263°C – 264°C; ^1H NMR δ(DMSO-d_6, 300 MHz): δ3.71 (s, 6 H), δ5.46 (s, 1 H), δ6.82 (d, J=8.7 Hz, 4H), δ7.07 (d, J=8.7 Hz, 4H), δ11.0

(broad, 2H); ^{13}C NMR δ(DMSO-d_6, 75.5 MHz): δ=43.4, 55.0, 113.5, 118.0, 129.6, 133.9, 157.5.

(3,6-bis(bis(4-methoxyphenyl)methylene)-1,2,4,5-tetraoxocyclohexane)[28]

1D:

A mixture of 1.3 g (2.2 mmol) of **2D** (592.65 g/mole) and 1.0 g (4.4 mmol) of 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ) were refluxed for 3 hours in 50 mL of toluene. The solvent was evaporated and the solid extracted in a soxhlet extractor with 100 mL of methylene chloride (CH_2Cl_2). The resulting dark purple solution of tetrone was diluted with 100 mL of diethyl ether (Et_2O), left aside for approximately 1 hour and then filtered giving 0.9 g (70% yield) of the product: melting point ≥ 300°C (dec.); ^1H NMR δ($CDCl_3$, 300 MHz): δ3.87 (s, 3 H), δ6.88 (d, *J*=10 Hz, 2 H), δ7.28 (d, *J*=10 Hz, 2 H); Anal. Calcd for $C_{36}H_{28}O_8$: C, 73.46%; H, 4.79%. Found: C, 72.52%; H, 5.06%.

(3,6-bis(bis(4-dimethylaminophenyl)methyl)-2,5-dihydroxy-1,4-benzoquinone)[28] 2C:

A mixture of 2.0 g (14.3 mmol) of 2,5-dihydroxy-1,4-benzoquinone (DHQ) and 7.8 g (28.9 mmol) of 4,4'-bis(dimethylamino)benzhydrol (270.37 g/mol) was refluxed with stirring in 100 mL of absolute ethanol (EtOH) for 15 minutes. The solution was cooled and the purple product of spectroscopic purity (8.7 g, 13.5 mmol, 94.3%) filtered off, washed with ethanol and dried: melting point range = 169°C – 170°C (dec.); ^1H NMR δ($CDCl_3$, 300 MHz): δ2.90 (s, 12 H), δ4.0-4.5

(broad, 1 H), δ5.49 (s, 1 H), δ6.64 (d, *J*=9 Hz, 4 H), δ7.10 d, *J*=9 Hz, 4 H); ^{13}C NMR δ($CDCl_3$, 75.5 MHz): δ=40.7, 43.8, 112.5, 117.7, 129.6, 129.8, 148.9, 168.4.

(3,6-bis(bis(4-dimethylaminophenyl)methylene)-1,2,4,5-tetraoxocyclohexane)[28] 1C:

To a well stirred solution of 1.0 g (1.55 mmol) of **2C** dissolved in 100 mL of methylene chloride (CH_2Cl_2) was added slowly 0.7 g (3.1 mmol) of 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ) dissolved in 100 mL of chloroform ($CHCl_3$). During addition, the solution turned dark blue. It was left stirring for an additional 20 minutes and the solvent was then removed by evaporation. Separation of the product from reduced DDQ (DDQ-H_2) was achieved on a glass frit by slow dissolution of the protonated product with concentrated HCl until the acid was no longer yellow. The resulting acidic solution was neutralized first with NaOH and then with sodium bicarbonate until the solution reached pH 7-8. The precipitated blue suspension of product was suction filtered, washed with water, dried, and recrystallized from hot dimethyl sulfoxide (DMSO) giving 0.9 g (67% yield) as **1C**•($(CH_3)_2SO)_3$: melting point ≥ 300°C (dec.); ^1H NMR δ(DMSO-d_6, 300 MHz): δ3.13 (s, 12 H), δ6.76 (d, *J*=9 Hz, 4 H), δ7.20 (d, *J*=9 Hz, 4 H); Anal. Calcd for $C_{40}H_{40}N_4O_4$: C, 63.1%; H, 6.7%; N, 6.4%. Found: C, 62.43%; H, 6.94%; N, 6.61%.

2.3: Instability of **1E**

Designing organic building blocks from the ground up can be difficult. One characteristic that all organic building blocks should possess is their ability to resist unwanted chemical reactions such as decomposition. Over the course of this study, crystals of **1E** suitable for X-ray analysis were unobtainable despite repeated attempts at varying solvents, solvent mixtures, temperature, diffusion rate and crystal growing methodology. However, after a few mgs of **1E** was dissolved in the following solvents, tetrahydrofuran, toluene, dichloromethane, and chloroform, the solutions underwent dramatic color changes when allowed to stand in the presence of air over a given period. Initially, both the tetrahydrofuran and toluene solutions of **1E** had a dark red appearance while both dichloromethane and chloroform solutions of **1E** had a deep violet appearance. The violet color of the solutions, which contained chlorinated solvents, might be attributed to a solvation effect in which the electrons from the chlorine atoms interact with the π system within **1E** causing an absorption shift. A portion of each of the solutions was placed in NMR tubes and *n*-octane was injected from a syringe, so as not to disturb the bottom layer and to fill the remaining volume. Each of the NMR tubes were capped and allowed to remain undisturbed in a dark environment so single crystals suitable for structural determination by X-ray anylsis might be obtained.

After one month the tetrahydrofuran solution was completely yellow and two small lemon-yellow crystals adhered to the glass wall of the NMR tube, this will be discussed in further detail later in this section. The toluene solution was

still red but of a lighter shade. This was expected due to the change in concentration. The toluene / n-octane mixture was allowed to stand for another month and upon inspection the solution had further discolored to a yellow-orange solution with a microscopic amount of a yellow-orange precipitate unsuitable for X-ray analysis. The dichloromethane / *n*-octane and chloroform / *n*-octane solutions were completely diffused and were a lighter shade of purple. These solutions were allowed to stand for additional months and showed signs of degradation but no precipitate could be seen.

The two lemon-yellow crystals that precipitated from the tetrahydrofuran / *n*-octane solution were extracted in a glove bag containing a continuous flow of nitrogen. Of the two crystals investigated only one measuring 0.18 mm x 0.18 mm x 0.30 mm was suitable for X-ray analysis and a portion of its final structure is represented in Figures 14 and 15. Structural analysis[44] revealed that the cyclohexane core of **1E** had been compromised and rearranged into two *cis*-fused γ-lactone rings, **7**, disrupting a portion of the original π framework. In addition, there were two tetrahydrofuran rings in close contact, 2.87 Å, with the moieties labeled X. X was not well defined and its electron density, along with the tetrahydrofuran rings, showed large distortions according to the thermal parameters calculated by the X-ray software.[44] Two functional groups were allowed to represent X; $-CH_3$ and –OH but neither of them produced satisfactory results-the system failed to converge on a solution without the use of geometrical restraints (bond lengths and angles). When those systems failed, the bridgehead $-O_1X$ was replaced altogether with two additional functional groups: –CHO and

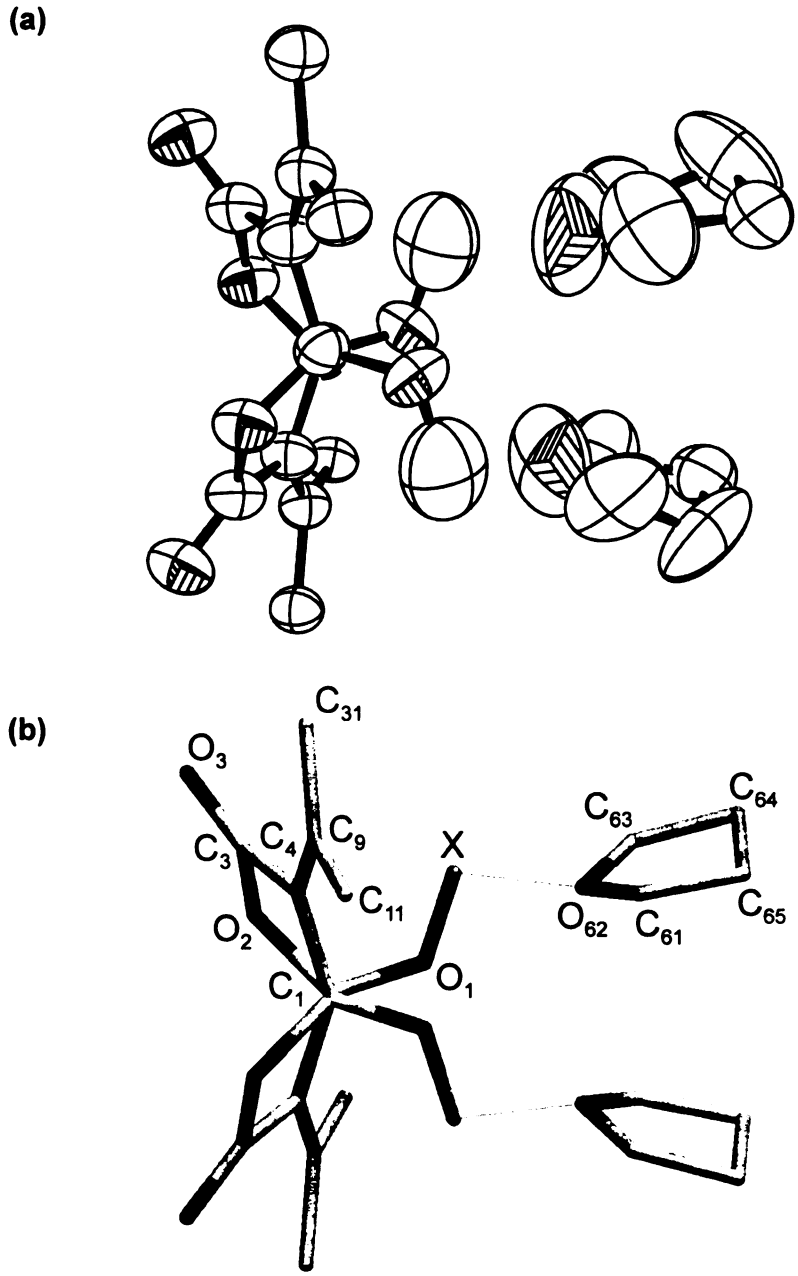


Figure 14. Rearranged tetrone core of **1E**, **7**, viewed down the a-axis.

(a) ORTEP drawing (50% probability).[45] (b) A stick representation.[46]

Hydrogen atoms and aryl rings have been removed for clarity.

(a)

(b)

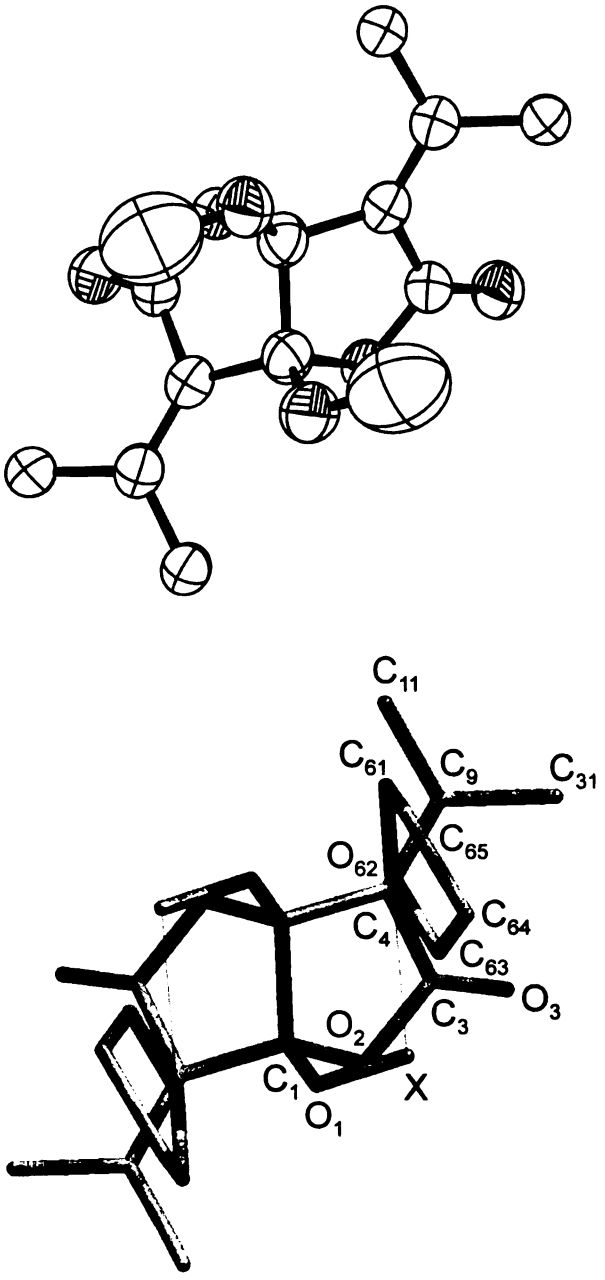


Figure 15. Rearranged tetrone core of **1E**, **7**, viewed down the b-axis. (a) ORTEP drawing (50% probability). (b) A stick representation. Hydrogen atoms, aryl rings and tetrahydrofuran rings (in the ORTEP plot only) have been removed for clarity.

Table 3. Selected bond lengths of the rearranged core of **1E**.

Atoms	Length, Å	Atoms	Length, Å
C_1-C_1'	1.578(6)	C_3-O_2	1.385(4)
C_1-O_1	1.374(4)	C_3-O_3	1.200(4)
C_1-O_2	1.428(4)	C_3-C_4	1.474(5)
C_1-C_4'	1.491(4)	C_9-C_4	1.359(4)
		C_9-C_{11}	1.472(4)
O_1-X	1.274(8)	C_9-C_{31}	1.479(4)
X-O_{62}	2.868*		

*Calculated bond distance.

Table 4. Selected bond angles of the rearranged core of **1E**.

Atoms	Angle, °	Atoms	Angle, °
C_4'-C_1-C_1'	103.3(3)	C_3-O_2-C_1	111.3(2)
O_1-C_1-C_1'	113.5(2)	C_4-C_3-O_2	109.3(3)
O_2-C_1-C_1'	102.5(2)	C_1'-C_4-C_3	105.7(3)
O_1-C_1-C_4'	114.1(3)		
O_1-C_1-O_2	109.7(3)	O_2-C_3-O_3	118.8(3)
O_2-C_1-C_4'	113.0(3)	C_4-C_3-O_3	131.4(3)
C_3-C_4-C_9	125.0(3)	C_{11}-C_9-C_4	122.2(3)
C_1'-C_4-C_9	129.2(3)	C_{31}-C_9-C_{11}	117.8(3)
		C_4-C_9-C_{31}	120.0(3)
C_1-O_1-X	113.9(4)		

Table 5. Average bond lengths.

Atoms	Length, Å	Atoms	Length, Å
C-C	1.54	C=C	1.34
C-O	1.43	C=O	1.23
O-O	1.48		
C-H	1.10	O-H	0.96

$-CH_2OH$. Neither of these two systems produced satisfactory results and failed in a similar manner as previously mentioned. Of the systems tested the final structure was obtained by allowing X to be represented by a $-CH_3$ group because it represented the crystallographic data the most accurately. However, this was not a logical choice considering the close contact between X and the oxygen atom contained within the tetrahydrofuran ring (see Tables 3, 4 and 5). If a hydrogen atom represented X, a classical hydrogen bond would exist and account for the close contact between it and the tetrahydrofuran ring. Despite the ambiguity in the crystal structure the rearrangement of the core is undeniable and can be accounted for, assuming that a hydrogen atom represents X.

Ethers including tetrahydrofuran[47] are well known for their tendency to form peroxides when distilled from their inhibitors and exposed to air for a period of time. As mentioned previously, the tetrahydrofuran/*n*-octane solution containing **1E** was exposed to the atmosphere. A mechanism that accounts for the oxidation and rearrangement of **1E** into **7** is represented in Figure 16. After the oxidation of the tetrahydrofuran ring into its corresponding peroxide, the peroxide attacks the electron deficient carbonyl inserting an oxygen atom (after rearrangement) via steps similar to a Baeyer-Villiger oxidation.[48] The acid catalyzed rearrangement was made possible by the small impurity of **8** or from some other unknown species. The corresponding anhydride would then exist in equilibrium with at least two other species. Water would then add in one of two places: First, water could attack the carbonyl of the anhydride, cleaving the seven-member ring and then rearrange itself into **7** by following tradition carbonyl

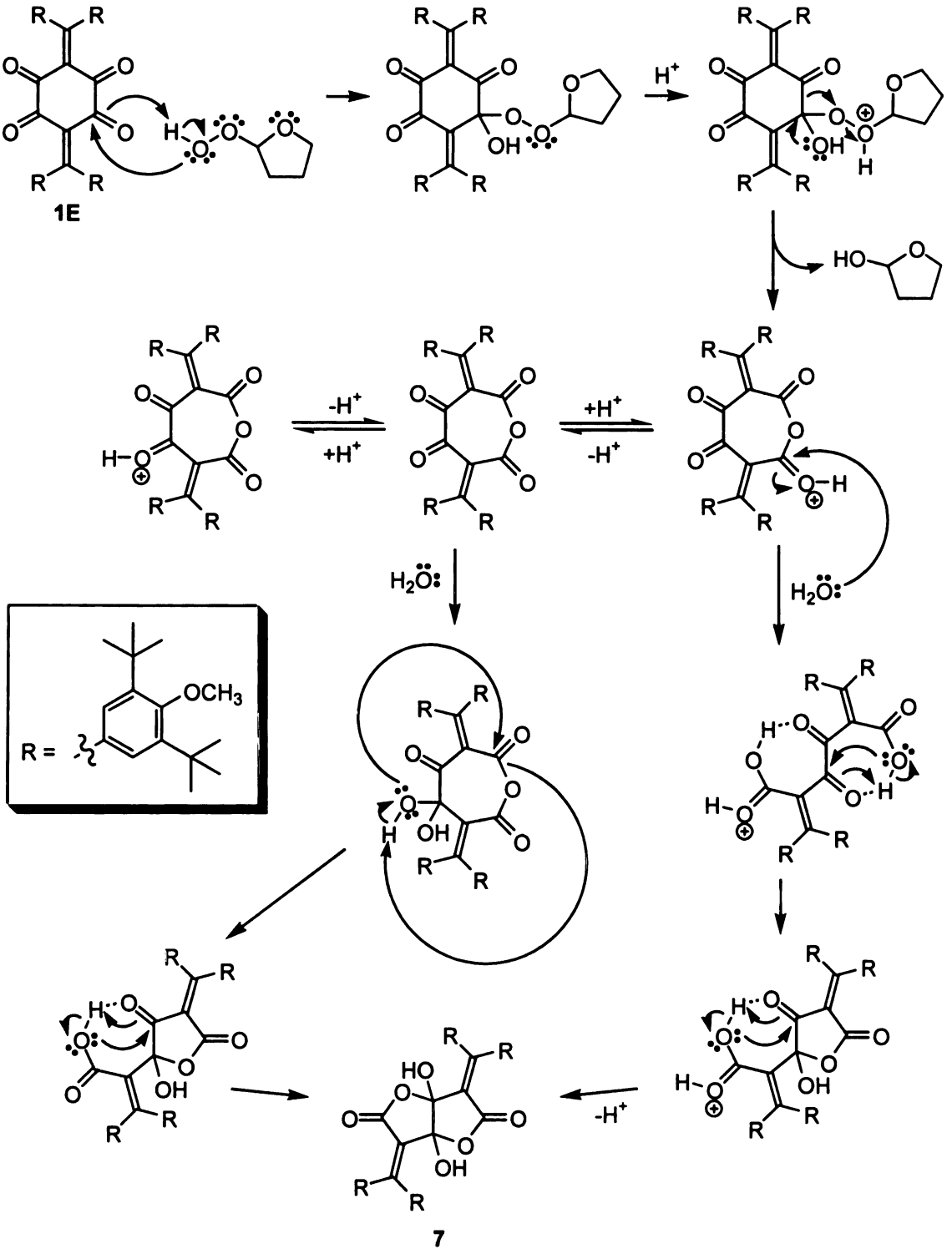


Figure 16. Rearrangement of **1E** into **7**.

chemistry. Second, water could produce an acetal by attacking one of the two carbonyls not contained within the anhydride functional group, and then rearrange itself to produce **7**.

The mechanism previously mentioned accounts for the oxidation and rearrangement of the core, the two bridgehead oxygen atoms, and for the close contact between X and O_{62} of the tetrahydrofuran ring. However, the electron density of X, which suggests an atom heavier than hydrogen, still remains a mystery. One explanation might be that one of the bridgehead hydroxyl groups was oxidized to form a peroxide. This coupled with the symmetry operators of the orthorhombic crystal system (P_{bcn} space group) would give the illusion that both positions of X must be a heavy atom when indeed only one should be. If this is true then explaining the close contact between O_{62} and the oxygen contained within the peroxide functional group becomes difficult if not impossible.

It was determined that oxygen gas was responsible for the decomposition of **1E** through the preparation of several tubes containing various concentrations of **1E** in a tetrahydrofuran/*n*-octane solution, which were then placed in numerous locations and subjected to a variety of conditions. The solutions were placed in environments that offered different degrees of lighting or no light, an atmosphere of nitrogen (provided by a continuous flow into a dry bag) or an atmosphere containing oxygen gas. One tube was prepared under vacuum and sealed and subjected to all of the conditions mentioned above. All solutions prepared changed from an initial color of red to a bright lemon yellow color except for the solution that was sealed in a tube under vacuum. The decomposition of the

organic building block via oxygen gas demonstrates the great lengths that must be taken to insure the survival of **1E** if it is to be used as a paramagnetic spin carrier in the design of organic magnetic materials.

2.4: Synthesis of $\mathbf{1E}^{-\bullet}$ and $\mathbf{1E}^{2-\bullet\bullet}$

In previous work,[28] it was reported that tetrones of **1** could be reduced under an inert atmosphere and monitored visually with the naked eye or more accurately, via UV/Vis/NIR spectroscopy, with various alkali metal mirrors to obtain $\mathbf{1}^{-\bullet}$ and $\mathbf{1}^{2-\bullet\bullet}$ in quantitative yields. A specialized glass Schlenk apparatus depicted in Figure 18, nicknamed 'T^2-cell', consisted of two 25.0 mL roundbottom chambers fitted with 10/20 glass stoppers, a 1.0 mm thick quartz UV/Vis/NIR optical cell, and an interchangeable EPR / NMR tube, for electron paramagnetic resonance studies, nuclear magnetic studies or crystal growth, connected via a Swagelock device, was used in the preparation of all air-sensitive paramagnetic species. Each of the chambers within the T^2-cell is separated with a Teflon stopcock that can withstand high vacuum. Figure 17 schematically shows the reactions that were performed in the T^2-cell.

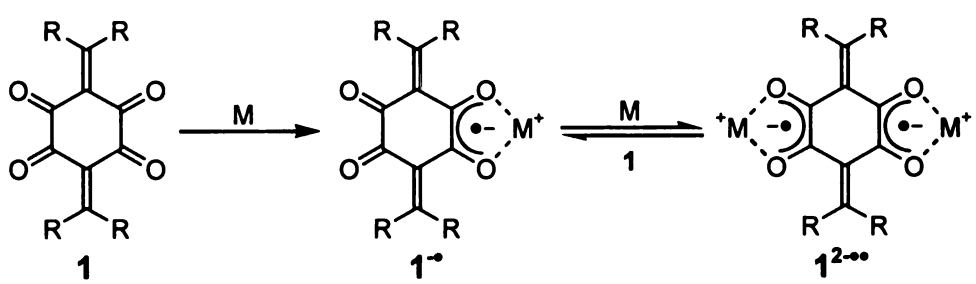


Figure 17. Alkali metal reduction of **1** to $\mathbf{1}^{-\bullet}$ and $\mathbf{1}^{2-\bullet\bullet}$.

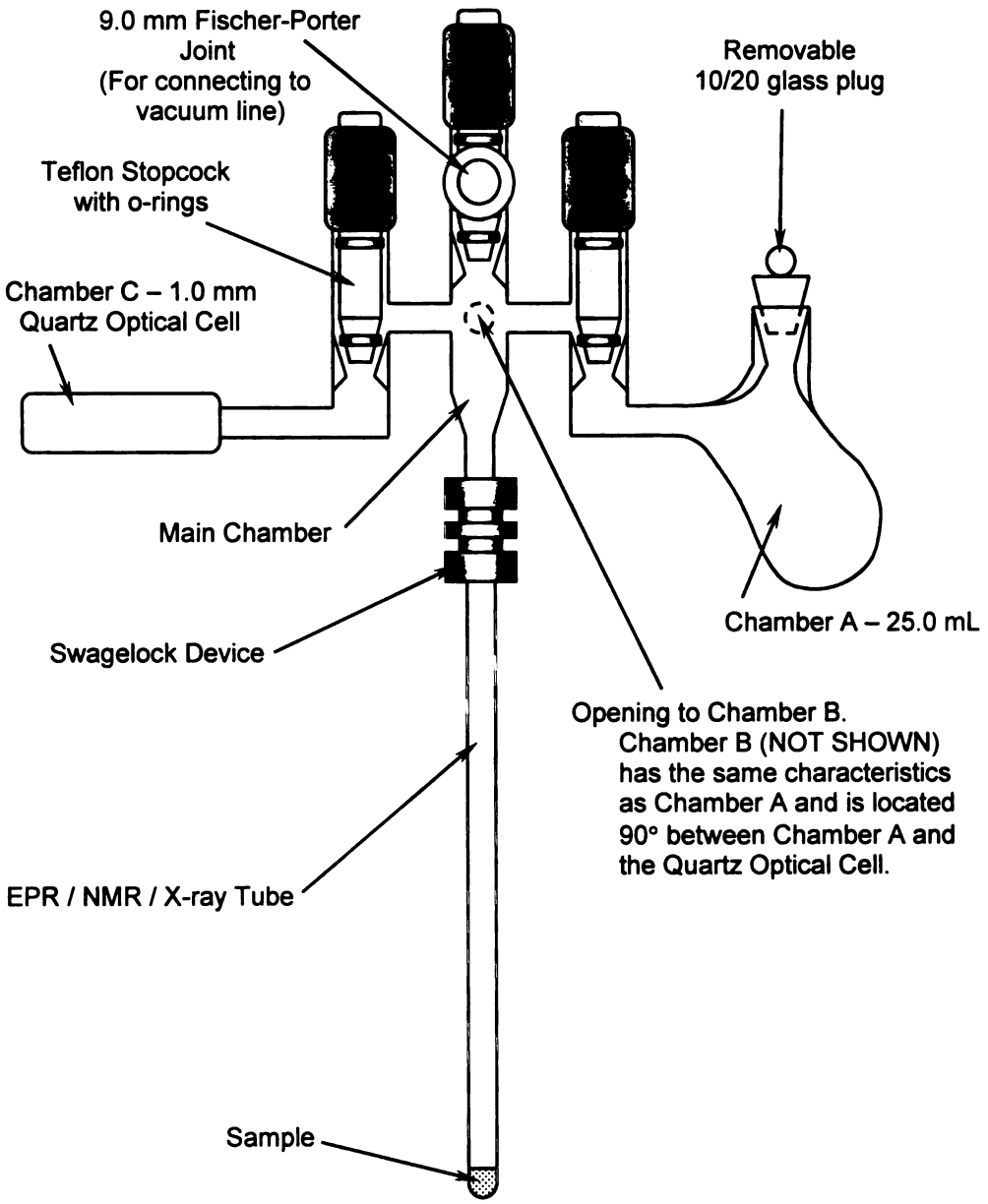


Figure 18. T^2-cell used in the preparation and analyses of $\mathbf{1E^{-\bullet}}$ and $\mathbf{1E^{2-\bullet\bullet}}$.

Synthesis of $1E^{-\bullet}$ and $1E^{2-\bullet\bullet}$:

In a typical experiment, between 5 and 15 mg (≤1 mg for EPR studies) of **1E** (1037.47 g/mol) was added to one of the two 25.0 mL chambers (or the tube) and the 10/20 glass plug was greased with Apiezon high vacuum grease, placed in the 10/20 joint and the Teflon stopcock was closed to isolate the chamber. The T^2-cell was connected to a vacuum line consisting of a low vacuum capable of reaching 20 mtorr and a high vacuum line capable of lowering pressures to 10^{-6} torr. The pressure inside the entire T^2-cell was reduced to 30 mtorr then to 5×10^{-6} torr at which the Teflon stopcock (main valve to allow air into the entire apparatus) was closed. The T^2-cell was placed in a dry box filled with an inert atmosphere of helium. The main valve (Teflon stopcock) was opened slowly as to allow the system to return to atmospheric pressure. The glass plug, 10/20, was removed from another chamber and an excess of alkali metal was placed into the chamber with the tip of a glass pipet and the glass plug returned. The T^2-cell was returned to the vacuum line and the inert atmosphere was evacuated to 5×10^{-6} torr.

The alkali metal mirror was created by sublimation under high vacuum with a gentle passing of a flame and with the Teflon stopcock to that chamber closed; if the valve was left open, the alkali metal tended to sublime outside this chamber, an undesired complication. After the mirror was formed an appropriate aprotic polar solvent like tetrahydrofuran was condensed with liquid nitrogen into the chamber containing **1E**; the vacuum bottle that was the source of the solvent was weighed before and after the transfer to determine the mass of the solvent

used (typically 4.0 g – 5.0 g if tetrahydrofuran is used). The tetrahydrofuran and **1E** mixture was allowed to return to ambient temperature and swirled to form a homogeneous solution.

The reduction of **1E** to $\mathbf{1E}^{-\bullet}$ and $\mathbf{1E}^{2-\bullet\bullet}$ was readily but controllably accomplished at ambient temperatures by washing a small portion of the entire solution over the metal mirror and/or washing the apparatus with cold ethanol that had been chilled with solid carbon dioxide (dry ice). As the reduction proceeded the solution's appearance took on a majestic purple hue; this was attributed to the monoradical monoanion $\mathbf{1E}^{-\bullet}$. Further reduction of the parent compound caused a second color shift to an intense aqua blue-green with an interim color of navy blue. The aqua blue-green color was attributed to the diradical dianion $\mathbf{1E}^{2-\bullet\bullet}$. The parent compound **1E** could be reduced beyond the diradical dianion, but the product's structural identity was never determined as other spectroscopic studies pointed to irreversible decomposition at this point. The 'trianion' or 'tetraanion' solution had an overall reddish-orange appearance and generates an EPR spectrum similar to the monoradical monoanion. The reductions could be monitored visually or with the quartz optical cell that was attached to the apparatus.

The radical species of $\mathbf{1E}^{-\bullet}$ and $\mathbf{1E}^{2-\bullet\bullet}$ at ambient temperatures inside the T^2-cell were found to show noticeable decomposition after approximately twenty-four hours. In contrast, no decomposition was ever observed for solutions of $\mathbf{1E}^{-\bullet}$ and $\mathbf{1E}^{2-\bullet\bullet}$ under vacuum in sealed glass tubes at ambient temperatures. The decomposition of the solution in the T^2-cell must have occurred via the slow

diffusion of oxygen gas between the o-ring and the glass wall. When solutions of $\mathbf{1E}^{-\bullet}$ and $\mathbf{1E}^{2-\bullet\bullet}$ were exposed to oxygen gas the resulting solution has a bright lemon yellow appearance; the same as that for the rearranged tetrone mentioned previously.

2.5: Visible / Near Infrared Spectroscopic Studies of $\mathbf{1E}^{-\bullet}$ and $\mathbf{1E}^{2-\bullet\bullet}$

As mentioned in the previous section, the reduction of **1E** to $\mathbf{1E}^{-\bullet}$ and $\mathbf{1E}^{2-\bullet\bullet}$ was spectrophotometrically observed by vis-NIR in the range of 400 nm – 2000 nm with a 1.0 mm thick quartz optical cell. The progress of the reduction was monitored by a Guided Wave Model 260 fiber-optic spectrophotometer and analyzed with Igor Pro 4.03 software.

The tube (main chamber) and chamber A of the T^2-cell were filled with approximately 5.0 mg of **1E** and chamber B with an excess of cesium metal according to the procedure mentioned in section 2.4. The entire cell was evacuated of its atmosphere and the appropriate valves were closed to isolate the chambers from each other and avoid cross-contamination. Approximately 4.0 – 5.0 g of freshly dried and degassed tetrahydrofuran was distilled into the optical chamber and the tube (main chamber) containing **1E**. A reference scan was taken and then this tetrahydrofuran was released from the optical cell and added to the chamber containing a homogeneous mixture of **1E** and tetrahydrofuran while under anaerobic conditions. There was some concern over the reversibility and stability of the diradical dianion so two equivalent quantities were used in this experiment. The neutral tetrone solution was continuously

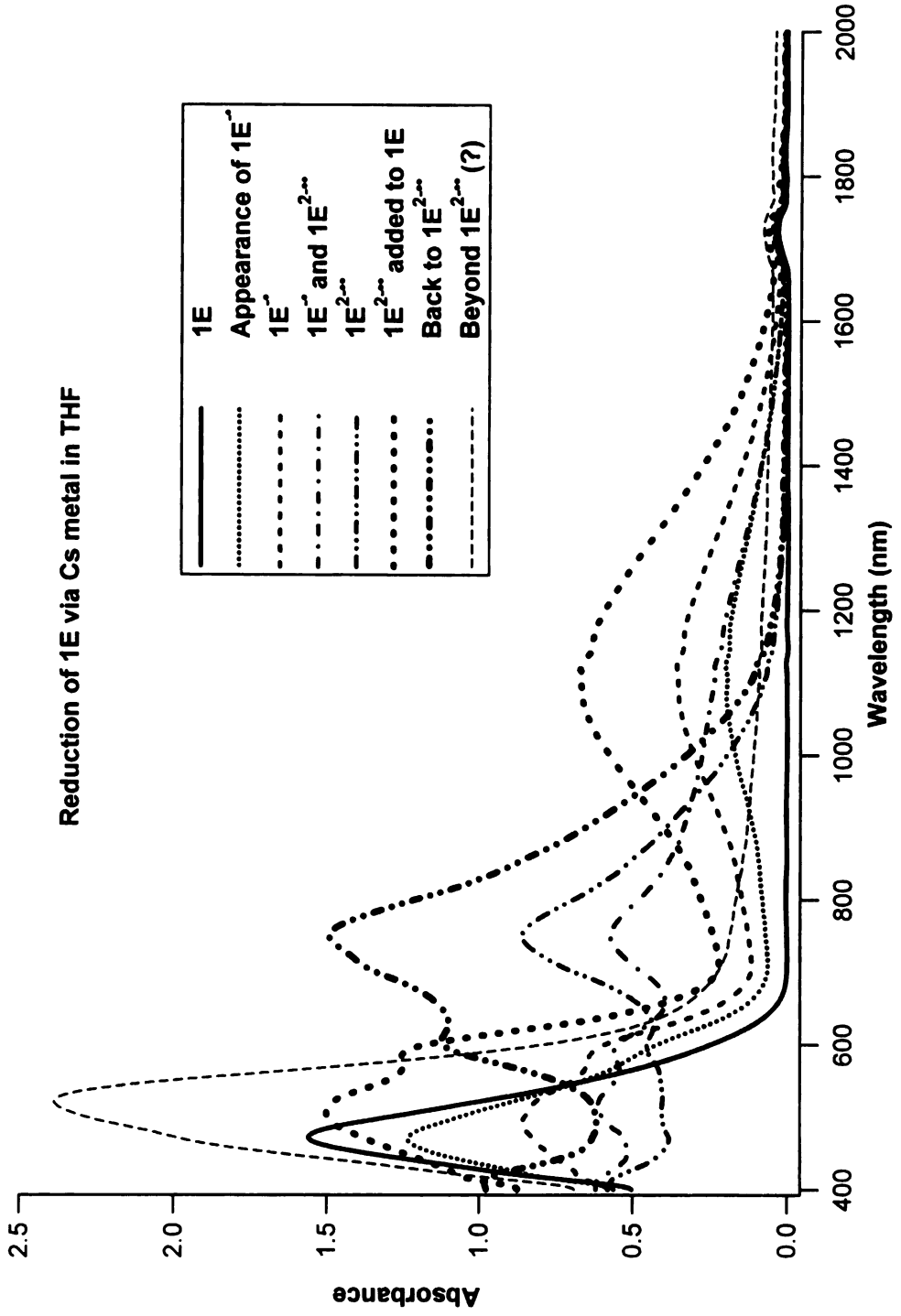


Figure 19. The Visible / Near-IR spectrum of the reduction of **1E** with cesium metal in tetrahydrofuran.

reduced and analyzed at small intervals to afford pure monoradical monoanion **1E**$^{-\bullet}$ with further reduction following to form **1E**$^{2-\bullet\bullet}$. See Figure 19 on page 59 for the Visible / Near-IR spectrum.

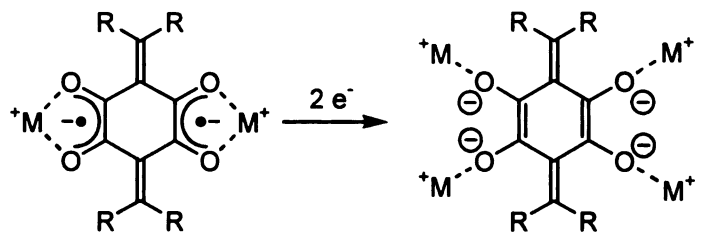
In tetrahydrofuran, the neutral tetrone **1E** was found to have a broad single absorption band with a λ_{max} of 472 nm. This is in good agreement with a λ_{max} of 480 nm obtained in acetonitrile.[28] Upon reducing the neutral tetrone further, a small absorption band appeared around 590 nm to 600 nm and a broad absorption band at 1120 nm. This absorption band spans nearly 1000 nm and began around 700 nm and proceeded to 1700 nm. The cause for this broadening was not fully understood until X-ray analysis and EPR experiments confirmed the nature of the monoradical monoanion and diradical dianion, discussed later in this chapter. Continued reduction produced a spectrum of the monoradical monoanion **1E**$^{-\bullet}$ with the following absorptions: λ_{max} = 494 nm, 600 nm shoulder that drops rapidly above 610 nm, and a broad absorption centered at 1120 nm. The diradical dianion **1E**$^{2-\bullet\bullet}$, generated by further contact with the cesium metal mirror, showed one main absorbance at 750 nm with another small peak located at 600 nm which may be due to residual **1E**$^{-\bullet}$. Extinction coefficients could not be accurately calculated for the species investigated because of undissolved starting material and formation of the diradical dianion produced a microcrystalline product, which precipitated from solution, some of which adhered to the glass wall as a thin film. This may be one reason why all attempts at growing crystals of **1E**$^{2-\bullet\bullet}$ have failed.

After the neutral tetrone was reduced to the diradical dianion it was

necessary to determine if the reaction was reversible. The solution was therefore combined with the neutral tetrone that had not undergone any previous reaction(s). Upon contact with the diradical dianion solution, the neutral tetrone immediately changed the color of the solution from an intense aqua blue-green back to a majestic purple indicative of the monoradical monoanion and the majority of the microcrystalline precipitate that had previously coated the inside of the glass apparatus had dissolved back into the tetrahydrofuran solution. This solution's absorption spectrum exactly matched the previous spectrum of $\mathbf{1E}^{-\bullet}$, but with an absorption intensity almost twice that of the original monoradical monoanion spectrum. This was expected considering the amount of the starting material had doubled while the volume had remained constant. Also, the transfer of one electron from the diradical dianion to the neutral tetrone to produce the monoradical monoanion was expected and would achieve the greatest distance between unpaired electrons; thus removing the intramolecular electron-electron interactions within the diradical species. Since the spectrum was identical it was concluded that the reaction was reversible and that decomposition does not occur. The instrument switching between the silicon detector and the lead sulfide detector caused the small inconsistent dip that was observed around 1125 nm.

The diradical dianion was found again by washing the solution over the cesium metal mirror. This produced a spectrum identical to the one before (for $\mathbf{1E}^{2-\bullet\bullet}$) except the intensity was found to be twice that of the original. Therefore one could conclude the reactions are reversible and there is an equilibrium that exists between the chemical species **1E**, $\mathbf{1E}^{-\bullet}$ and $\mathbf{1E}^{2-\bullet\bullet}$. Further reduction of

$1E^{2-\bullet\bullet}$ produced a species whose absorption intensity was greater than that of both $1E^{-\bullet}$ and $1E^{2-\bullet\bullet}$. Its λ_{max} was positioned at 520 nm and the spectrum "resembled" that of the neutral tetrone. This species is still unidentified but a possible structure is shown below in Figure 20.



$1E^{2-\bullet\bullet}$ $1E^{4-}$

Figure 20. Possible structure for the Visible / Near-IR signal λ_{max} = 520 nm.

The electronic spectroscopic studies that were conducted have shown the stability of $1E^{-\bullet}$ and $1E^{2-\bullet\bullet}$ at ambient temperatures under anaerobic conditions. This is an advantage for the system that has been devised within our group. If room temperature magnetic materials are to exist then they must possess organic building blocks that are stable at ambient temperatures. However, their reactivity in an ordinary atmosphere is a less desirable trait. Exposure of a solution of $1E^{-\bullet}$ or $1E^{2-\bullet\bullet}$ to oxygen gas causes an immediate degradation reaction to occur. The tetrone **1E** has also shown that the reaction pathway to generate $1E^{-\bullet}$ and $1E^{2-\bullet\bullet}$ is reversible and may be utilized as a possible means of electron transport such as that found in photosynthetic processes.

2.6: Crystal Structure of $(\mathbf{1E}^{\bullet-})_2\bullet(Na^+)_2\bullet(C_4H_{10}O_2)_8$

Solutions of $\mathbf{1E}^{\bullet-}$ and $\mathbf{1E}^{2-\bullet\bullet}$ were prepared by a method similar to that described in section 2.4 for the direct probing of their physical structure. All alkali metal cation-radical anion salt solutions of $\mathbf{1E}^{\bullet-}$ were prepared, however, only two metal cation solutions, sodium and potassium, produced crystals suitable for X-ray analysis. The sodium salt was produced from the reduction of **1E** in a solution of glyme (1,2-dimethoxyethane) while the potassium salt was produced from the reduction of **1E** in a solution of tetrahydrofuran; the potassium crystal was grown by Andrzej Misiolek. In the former case *n*-hexane was condensed onto a frozen solution of glyme and acted as the nonpolar co-solvent while in the latter case *n*-pentane acted as the nonpolar co-solvent. The tubes were then sealed under vacuum and allowed to slowly return to room temperature to avoid mixing by turbidity, which is often generated by a thermal gradient. Over a period of several weeks, the slow diffusion of *n*-hexane into glyme under anaerobic conditions caused the formation of small deep purple brick-like triclinic (P_1) crystals of $(\mathbf{1E}^{\bullet-})_2\bullet(Na^+)_2\bullet(C_4H_{10}O_2)_8$ to precipitate from the solution. The crystal structure of the sodium salt is shown in Figures 21 through 23.

The crystal structure of $(\mathbf{1E}^{\bullet-})_2\bullet(Na^+)_2\bullet(C_4H_{10}O_2)_8$ was found to contain two unique sodium atoms, two unique molecules of $\mathbf{1E}^{\bullet-}$ and eight molecules of glyme; four of which were coordinated to two sodium atoms while the others remained disordered and located in the void spaces between linear chains. In the one-dimensional structures, the anion of $\mathbf{1E}^{\bullet-}$ displayed the hoped-for chelating and bridging modes of coordination and it would be expected that the

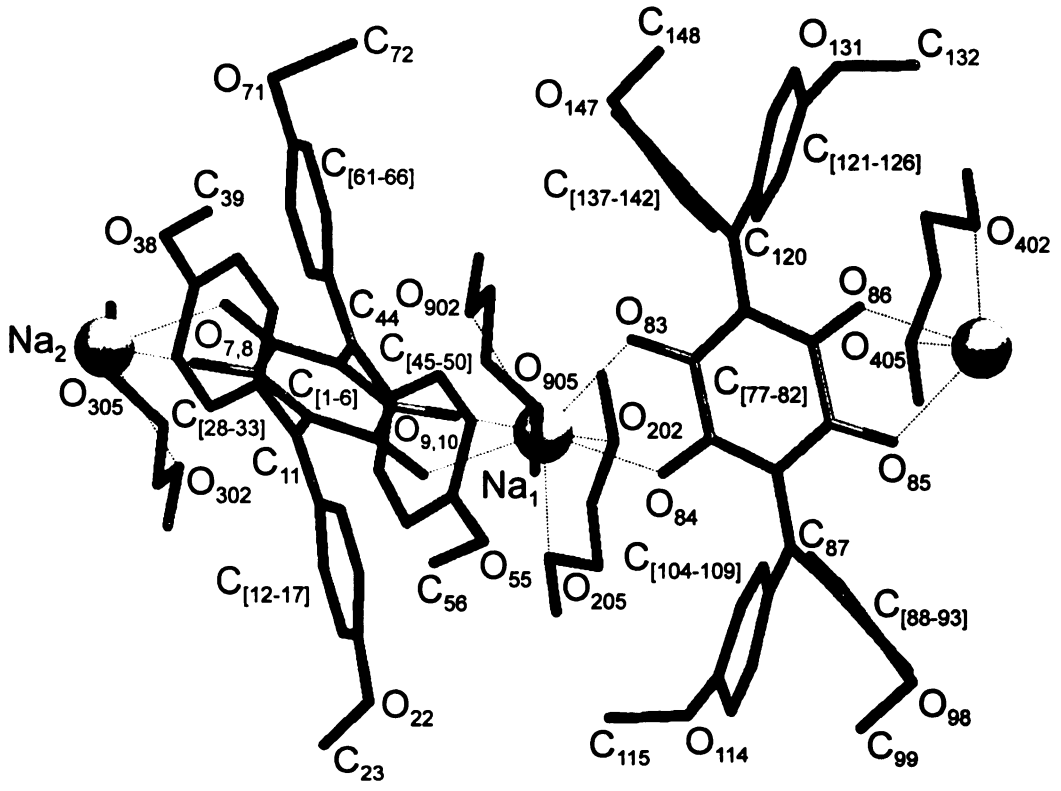


Figure 21. View of $(1E^{-})_2 \cdot (Na^{+})_2 \cdot (C_4H_{10}O_2)_8$ perpendicular to the bc plane. Disordered and uncoordinated solvent molecules, *t*-butyl groups and hydrogen atoms have been omitted for clarity.

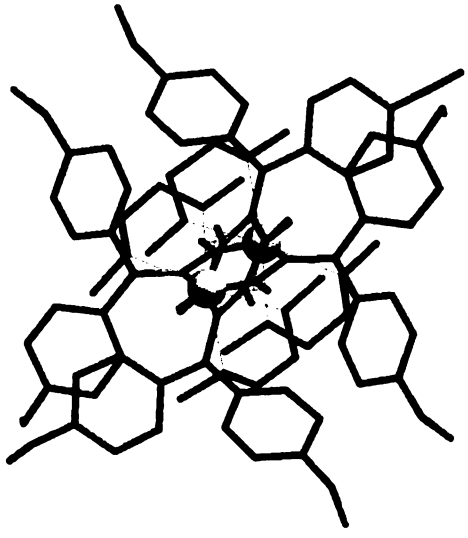


Figure 22. View of $(1E^{-})_2 \cdot (Na^{+})_2 \cdot (C_4H_{10}O_2)_8$ down the b-axis. Disordered and uncoordinated solvent molecules, *t*-butyl groups and hydrogen atoms have been omitted for clarity.

(a)

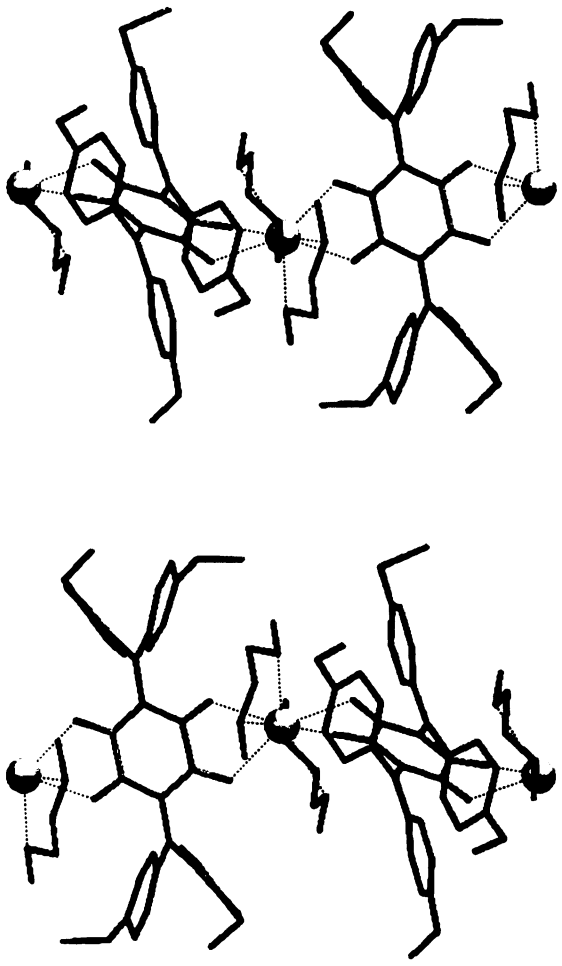
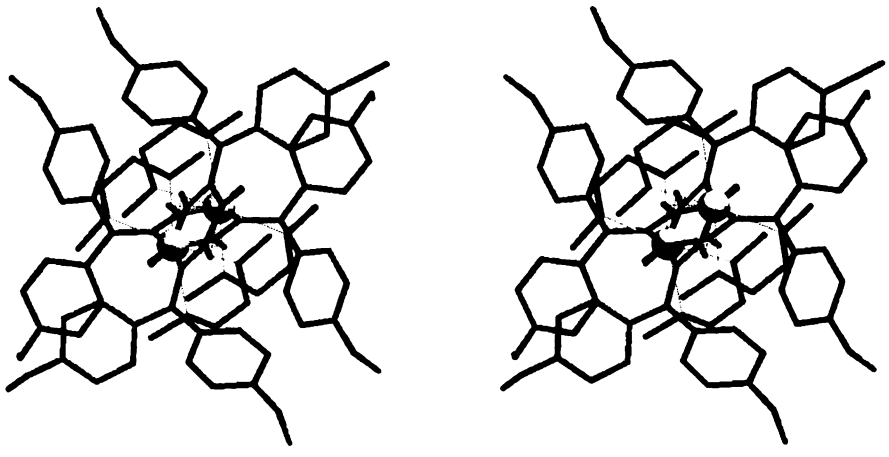


Figure 23. Packing of $(1E^{-\bullet})_2 \bullet (Na^+)_2 \bullet (C_4H_{10}O_2)_8$ perpendicular to the bc-plane (a) and down the b-axis (b). Disordered and uncoordinated solvent molecules, *t*-butyl groups and hydrogen atoms have been omitted for clarity.

(b)



other tetrone radical anions would adopt a similar mode of coordination.

The core of the radical anion $\mathbf{1E^{-\bullet}}$ was found to be virtually flat with each carbonyl bent out of plane creating torsional angles that were between (-14.01° to 14.44°). Selected bond lengths of the core for one of the tetrones are shown below in Table 6. All crystallographic data for $\mathbf{(1E^{-\bullet})_2 \bullet (Na^+)_2 \bullet (C_4H_{10}O_2)_8}$ can be found in Appendix E. The carbon-carbon bond lengths from one oxygen atom through the core to the other oxygen atom were found to be nearly identical, which suggests the electron density is delocalized through the periphery of the core whereas the carbon-carbon bond that connects adjacent carbonyls displayed a bond length more characteristic of a carbon-carbon single bond and that conjugation may not exist, in a circular fashion, around the entire core like that found in benzene.

Table 6. Selected bond lengths and distances for $\mathbf{1E^{-\bullet}}$.

Atoms	Length, Å	Atoms	Length, Å
C_1-C_2	1.478(8)	C_1-O_7	1.219(7)
C_2-C_3	1.486(9)	C_3-O_{10}	1.207(7)
C_3-C_4	1.549(8)	C_4-O_9	1.258(7)
C_4-C_5	1.453(9)	C_6-O_8	1.263(8)
C_5-C_6	1.452(9)		
C_6-C_1	1.529(9)	Na_1-Na_2	8.408
		Na_1-Na_1	16.315
		Na_2-Na_2	16.315

The highly substituted and bulky phenyl rings located at the methylene positions, indicated as such: C_{11}, C_{44}, C_{87}, C_{120}, adopted a propeller-like rotation

that causes a discontinuity of conjugation from the phenyl rings into the core and its adjoining oxygen atoms. The propeller-like arrangement of the phenyl rings was found to be rotated 30° to 40° from the plane of the core. The bond angles around C_{44} were found to exist in the range of 118.63° to 122.58° and the bond lengths ranged from 1.442 Å to 1.477 Å, which suggests that C_{44} is sp^2 hybridized and has a bond order that is between a carbon-carbon single bond and that of a carbon-carbon double bond. This was not fully understood until an EPR experiment (section 2.8) revealed that the methylene position(s) of the previously mentioned carbon atoms, act like an electron sink and the unpaired electron(s) reside on these methylene position(s). This was disturbing for it was predicted that the unpaired electron would reside on the more electronegative oxygen atoms delocalizing itself through the core thus communicating with the electrons on the metal center or through the metal center to the unpaired electron residing on the next coordinated tetrone. Apparently the larger electron sink of the methylene positions is far superior to the electronegativity of the oxygen atoms.

Each sodium atom was coordinated to eight oxygen atoms and adopted a distorted square antiprism geometry. This distortion rotated the tetrone core planes to an angle of 37.6°. They have been found to exist in a zigzag arrangement, with bond distances of those listed in Table 6. Looking down the b-axis of the unit cell (Figures 22 and 23b) the zigzag nature of the sodium atoms became apparent and the distance between sodium chains was found to be approximately 17.05 Å. This has been attributed to the large bulky *t*-butyl groups

located on the phenyl rings. Each linear chain is held together by ionic bonding or dipole-dipole interactions (in the case of the coordinated solvents), however, the chains are held together by the weakest intermolecular interactions, induced dipole-induced dipole interactions or van der Waals interactions. These weak interactions, undoubtedly have contributed to the difficultly in crystallizing other radical anion salts and dianion salts.

The crystallization of $(1E^{\bullet-})_2 \bullet (Na^+)_2 \bullet (C_4H_{10}O_2)_8$ and of the potassium salt demonstrates the ability of the tetrones to bind metal cations to form one-dimensional structures. A similar structural motif could be envisioned if the charge on the cation was increased to two and the diradical dianion were used. If the charge on the metal cation were increased other multi-dimensional structural motifs might be created. However, all attempts to date to generate crystalline materials of $1E^{2-\bullet\bullet}$ with alkali metals have failed to produce anything other than a fine precipitate unsuitable for crystallographic analysis. It was noted during UV/Vis/NIR that upon formation of the diradical dianion a thin-film precipitate could be found adhering to the glass walls, which may be caused from the formation of a highly charged complex and the weak intermolecular interactions between growing chains. This occurs even more so for the other more polar tetrones.

2.7: EPR and Magnetic Susceptibility of $(1E^{\bullet-})_2 \bullet (Na^+)_2 \bullet (C_4H_{10}O_2)_8$

Approximately 6.0 mg of crystalline $(1E^{\bullet-})_2 \bullet (Na^+)_2 \bullet (C_4H_{10}O_2)_8$ was prepared for EPR (electron paramagnetic resonance) analysis and magnetic susceptibility

determination by a method similar to that described in section 2.4. However, after the generation of the monoradical monoanion the solvent was evacuated and a thin film of purple needles crystallized on the glass wall. The tube was then sealed under vacuum and placed in a dry box containing an inert atmosphere of helium gas. The tube was cut and the crystals harvested and placed into their appropriate receptacles (a quartz tube for EPR analysis and a polyethylene bag for magnetic susceptibility determination). The spectrum of the EPR is in Figure 24 while the magnetic susceptibility data is graphically represented in Figure 25.

The EPR of crystalline $(\mathbf{1E}^{\bullet-})_2\bullet(Na^+)_2\bullet(C_4H_{10}O_2)_8$ was recorded at 273 K on a Bruker ESP300E spectrometer with the following conditions: modulation frequency 100 kHz, modulation amplitude 0.101 G, power 0.5 mW, gain 10^4 and a microwave frequency 9.47930 GHz. For the crystalline sodium salt $(\mathbf{1E}^{\bullet-})_2\bullet(Na^+)_2\bullet(C_4H_{10}O_2)_8$ a single peak centered at 3381.04 G with ΔH_{pp} = 2.4 G and a g-value of 2.0032 can be observed. The g-value and signal are characteristic for an organic molecule with one unpaired electron and further demonstrate the ability of the tetrone, **1E**, to be a viable organic paramagnetic building block for the production of molecular magnetic materials.

The magnetic susceptibility of $(\mathbf{1E}^{\bullet-})_2\bullet(Na^+)_2\bullet(C_4H_{10}O_2)_8$ was recorded over the range 2-300 K at a 1000 G field strength using a DC SQUID (Superconducting QUantum Interference Device) magnetometer. The crystalline material was found to be paramagnetic with weak spin-spin interactions. This was expected considering the physical characteristics of the crystalline material.

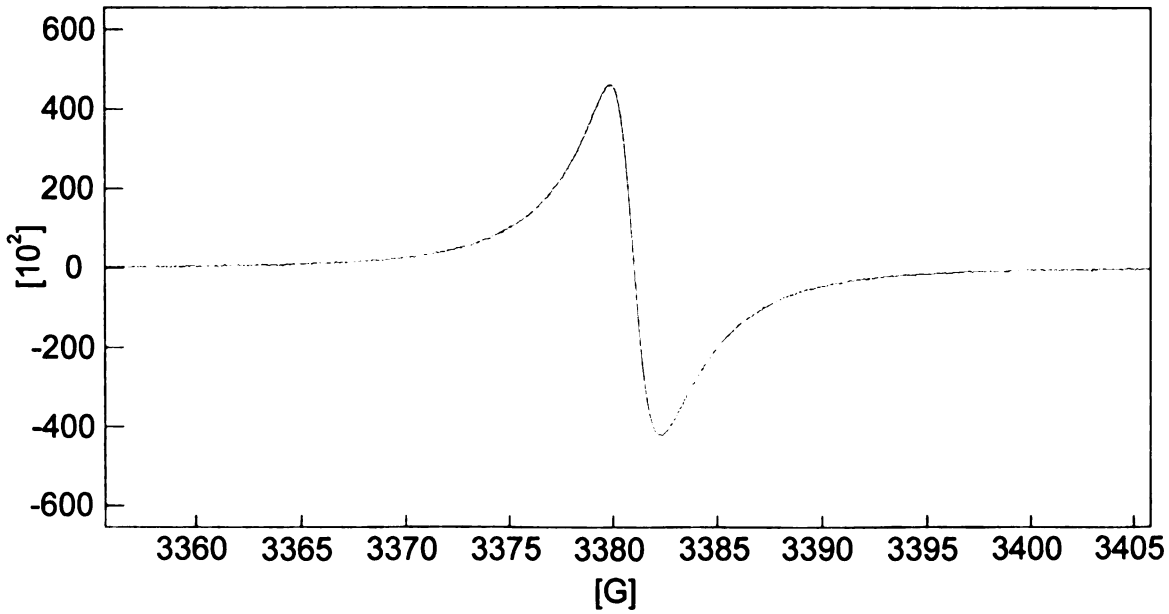


Figure 24. EPR of $(1E^{\cdot-})_2 \bullet (Na^+)_2 \bullet (C_4H_{10}O_2)_8$ recorded at 273 K.

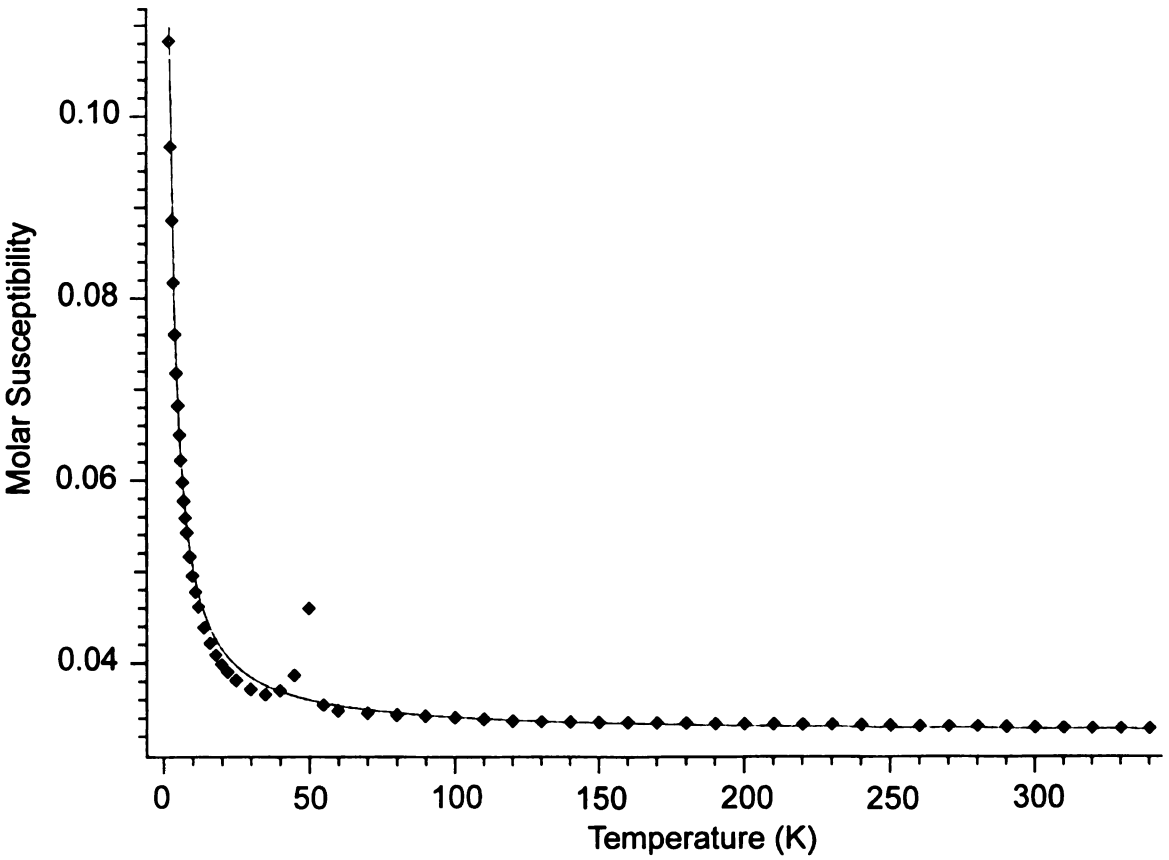


Figure 25. Molar susceptibility vs. Temperature for $(1E^{\cdot-})_2 \bullet (Na^+)_2 \bullet (C_4H_{10}O_2)_8$.

The unpaired electron which resides mostly on the methylene carbon (determined by other EPR experiments and discussed in the next section) is located ~7.8 Å away from the methylene site of the neighboring tetrone while the distance between the coordinated polymeric chains is ~17.0 Å. These distances are too great for magnetic ordering to take place even at the coldest of temperatures. The data was fit to the Curie-Weiss equation ($\chi = C / (T + \theta)$), this yielded a Weiss constant of θ = -0.6 K for the crystalline material of $(1E^{\cdot-})_2 \cdot (Na^+)_2 \cdot (C_4H_{10}O_2)_8$ while a Weiss constant of $|\theta| < 0.1$ K for the analogous potassium salt was observed. Around 50 K there is a deviation from the best fit Curie-Weiss equation, this has been attributed to the phase change of molecular oxygen.

Though very weak, the couplings vary based on cation size. In the sodium salt, where distances between metal-separated carbonyl oxygen atoms are in the range 4.02 Å to 4.52 Å, the magnetic coupling is slightly stronger than in the potassium salt with analogous distances in the range 5.16 Å to 5.17 Å. These coupling constants are extremely small and would fail to generate room temperature molecular magnets exhibiting bulk ferromagnetic behavior. However, diradical dianion complexes with transition metals should show stronger couplings due to a number of factors; a larger unpaired spin density on the tetrone unit that would give rise to intramolecular spin-spin interactions, stronger ionic bonding as a result of a larger Z_{eff} (effective nuclear charge) from a multivalent metal center and intermolecular spin-spin interactions between the unpaired electrons on the tetrone unit and the unpaired electrons residing in the

d or *f* orbitals of the transition metal. All of these factors combined should enhance the overall magnetic moment of the diradical dianion system.

2.8: The True Nature of $\mathbf{1E^{-\bullet}}$ and $\mathbf{1E^{2-\bullet\bullet}}$

Monoradical monoanions and diradical dianions of **1** were prepared and analyzed by EPR (Electron Paramagnetic Resonance), ENDOR (Electron Nuclear Double Resonance) and general triple ENDOR (nuclear-nuclear-electron resonance) spectroscopies[49] in order to confirm their structure, determine the nature of the diradical dianions' preference for a ground state singlet versus a ground state triplet, and determine the ^1H hyperfine splitting constants a_n and their relative signs, respectively. In a typical experiment, the tetrone (ca. 1 mg) and a small excess (ca. 1:2.2) of cryptand[2.2.2] were placed directly into a quartz EPR tube and the tube was then connected to the main chamber via a Swagelock device in a cell similar to the one shown in section 2.4 Figure 18. The alkali metal was placed in chamber A or B under anaerobic conditions (helium atmosphere) in a dry box. The helium atmosphere, contained within the apparatus, was evacuated to a pressure $\leq 10^{-5}$ torr on a high-vacuum line. The alkali metal mirror was formed from the gentle heating of a gas flame. After the freshly sublimed alkali metal mirror cooled to room temperature an appropriate amount of tetrahydrofuran, ~1 to 2 mL was then distilled into the EPR tube. The solution was then washed over the alkali metal mirror in a series of steps and the progress of reduction was monitored within the optical cell with a Guided Wave Model 260 fiber-optic spectrophotometer. In some cases, dilution of the solution

in the optical cell was necessary to keep the spectra on scale, so no extinction coefficients are reported. A small amount of solution was transferred to the quartz EPR tube, the tube was sealed off below the Swagelock fitting, and the sample was held in liquid nitrogen until it could be analyzed by EPR and ENDOR. The spectra were recorded on a Bruker (9.5 GHz) ESP300E spectrometer equipped with a DICE ENDOR unit and the temperature was controlled with an Oxford Instruments 9000 liquid helium cryostat. The g-values were measured by determining the field and microwave frequency with a Bruker ERO35M NMR gaussmeter and EIP model 25B frequency counter. Typical conditions for EPR were: modulation frequency 100 kHz, modulation amplitude 0.1 gauss, power 0.1mW, gain 10^5 and those for ENDOR were: modulation frequency 12.5 kHz, microwave power 20-32 mW, radio frequency power 200-300 W, modulation depth 100 kHz, and gain 10^5.

All monoradical monoanions, **1A**, **1C**, **1D**, and **1E**, in solution showed g-values characteristic for organic radicals (Table 7). The ^{1}H hyperfine splitting constants a_n and their relative signs were determined by Andrzej Misiolek and Andrew Ichimura which are presented in Table 7 also. As a representative example, the EPR and ENDOR spectra of **1A**$^{-\bullet}$ is displayed in Figure 26. Experimental EPR spectra of the radical anions were simulated by assigning values a_1 to the eight *ortho* protons, a_2 to the eight *meta* protons of the four phenyl groups and for **1A**$^{-\bullet}$, a_3 for the four *para* protons. As predicted by general triple ENDOR spectroscopy[49] for alternant hydrocarbons, a_1 and a_2 are opposite in sign. In general, simulations of high temperature (T>243 K) solution EPR

spectra based on ENDOR data show that the unpaired electron spin is delocalized over the entire molecule on the EPR time-scale. Observation of the ^{1}H hyperfine splitting values indicates there is a small, but negligible, increase in the splitting values as the substituents increase in electron-donating ability. However, despite this the spin density is not significantly altered by the phenyl rings' substituents. In the absence of cryptand[2.2.2], hyperfine splitting to the alkali metal nucleus can be observed when **1E** is reduced by Lithium metal (0.48 MHz) or Cesium metal (0.5 MHz).

Table 7. ^{1}H hyperfine splitting constants and isotropic g-values for monoradical monoanions and diradical dianions of tetrones **1A**, **1C**, **1D**, and **1E**, prepared by reduction in THF with stoichiometric quantities of potassium and cryptand[2.2.2].

Radical anion	a_1[a] (ortho)	a_2[a] (meta)	a_3[a] (para)	g-value
1A$^{-\bullet}$	±3.23	∓1.43	±3.23	2.0035
1D$^{-\bullet}$	±3.24	∓1.31	±0.36[b]	2.0032
1C$^{-\bullet}$	±3.26	∓1.08	-	2.0030
1E$^{-\bullet}$	±3.39	-	-	2.0031
1A$^{2-\bullet\bullet}$	±7.85	∓3.23	±7.85	2.0028
1E$^{2-\bullet\bullet}$	8.27	-	-	2.0028

a) In MHz; 2.80 MHz = 1 Gauss

b) *p*-methoxy protons

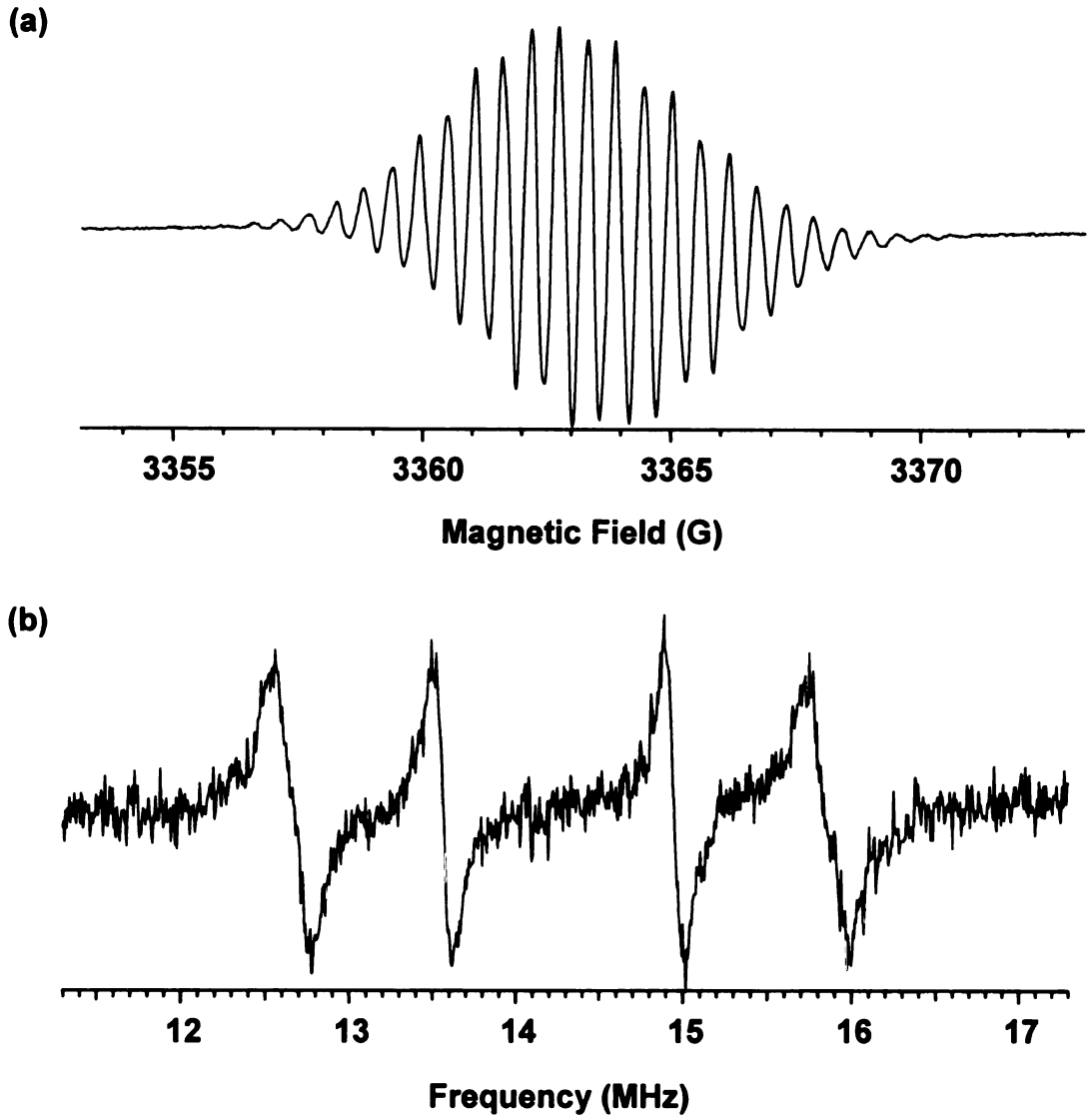


Figure 26. (a) EPR and (b) ENDOR of K•**1A**$^{-\bullet}$•C222 in tetrahydrofuran recorded at 246 K.

The diradical dianion of **1A** was prepared in several ways. The solution EPR spectrum shown in Figure 27 was obtained from the products of a reaction between **1A** with two equivalents of potassium metal and 15-crown-5 in tetrahydrofuran. The ^{1}H hyperfine coupling constants were found to be identical to the monoradical monoanion based on ENDOR measurements and simulation

of the solution EPR spectra. However, the magnitude of the measured values was found to be twice that of the monoradical monoanion which corresponds to exactly half the number of protons as shown by the spectral simulation. The

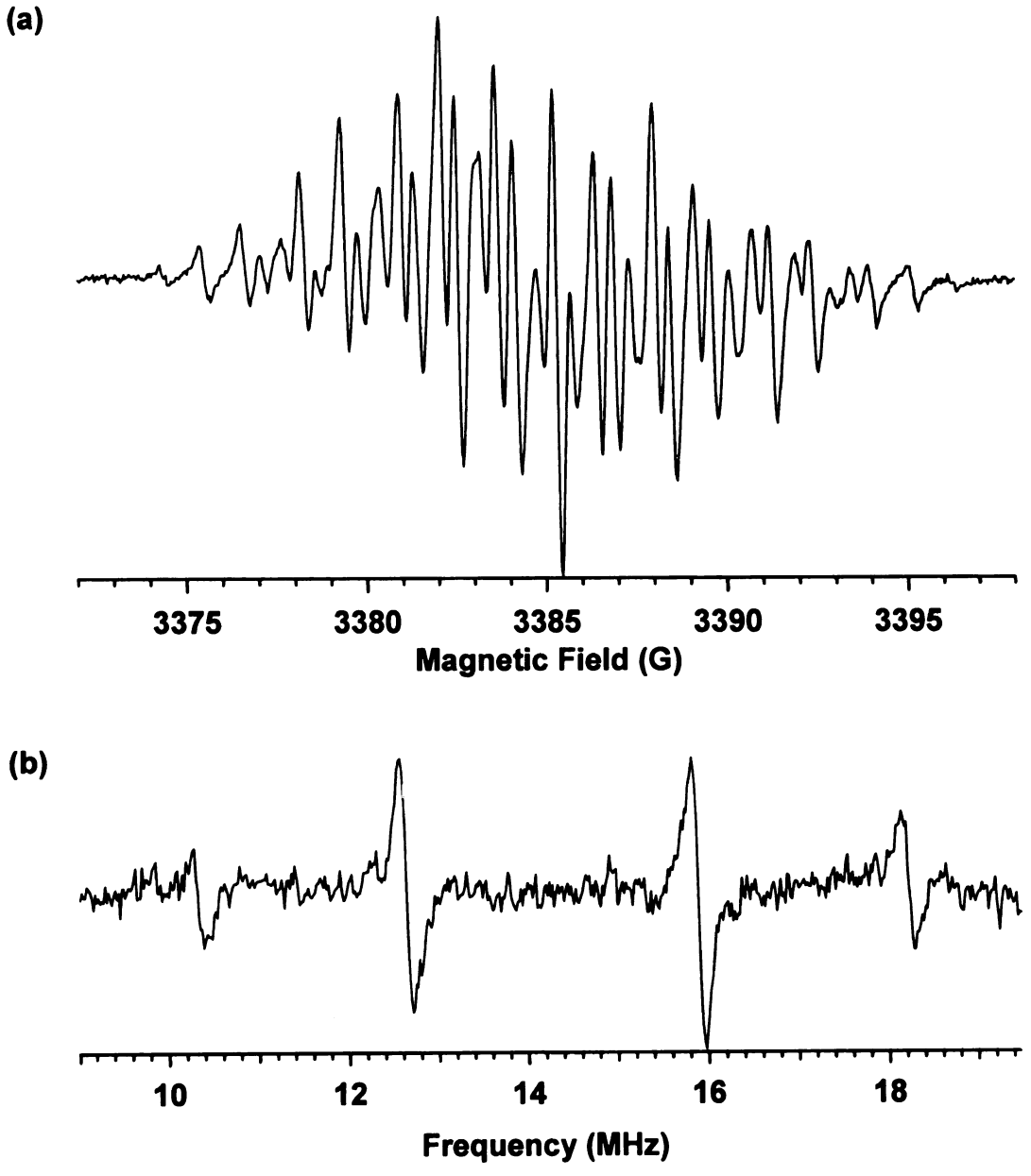
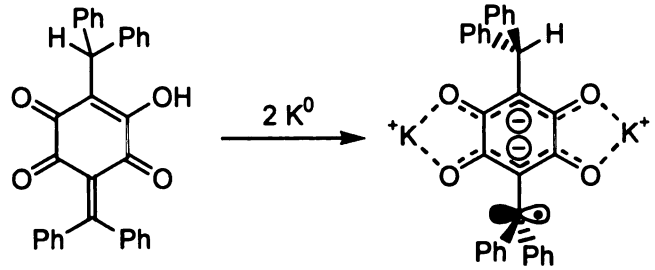


Figure 27. (a) EPR and (b) ENDOR spectra of $\mathbf{1A}^{2-\bullet\bullet}(K\bullet[15\text{-crown-}5]_2)_2$ in tetrahydrofuran recorded at 223 K.

diradical dianion apparently behaves as a biradical in which each electron spin is localized to one of the diphenylmethyl termini. The same phenomenon can be observed in the reduction of **1E** to $\mathbf{1E}^{-\bullet}$ and $\mathbf{1E}^{2-\bullet\bullet}$ with lithium, potassium and cesium metal. These spectra are shown in Figures 28 through 30, respectively.

To confirm the biradical behavior, that each unpaired electron was localized to one of the diphenylmethyl termini and that interpretation of the EPR spectra were correct, compound **8**, an intermediate and a minor product in the synthesis of tetrone **1**, was reacted with two equivalents of potassium to yield the monoradical dianion $\mathbf{8}^{2-\bullet}$, which is shown below. Both compounds $\mathbf{8}^{2-\bullet}$ and $\mathbf{1A}^{2-\bullet\bullet}$



8 $\qquad$ $\mathbf{8}^{2-\bullet}$

produced nearly identical EPR spectra (see Figure 31); spectra recorded by Dr. Vanessa McCaffrey. The spectral pattern and hyperfine coupling constants observed[28] indicate spin localization on the methylene site. Evidently, torsion about the C-C bonds connecting the two diarylmethyl subunits to the central $C_6O_4^{2-}$ ring effectively isolates the unpaired spins and is the reason for the absence of a triplet signal for the diradical dianions. The X-ray crystal structures of **1A**, **1C**, and the two monoradical monoanion salts of **1E** show torsional angles

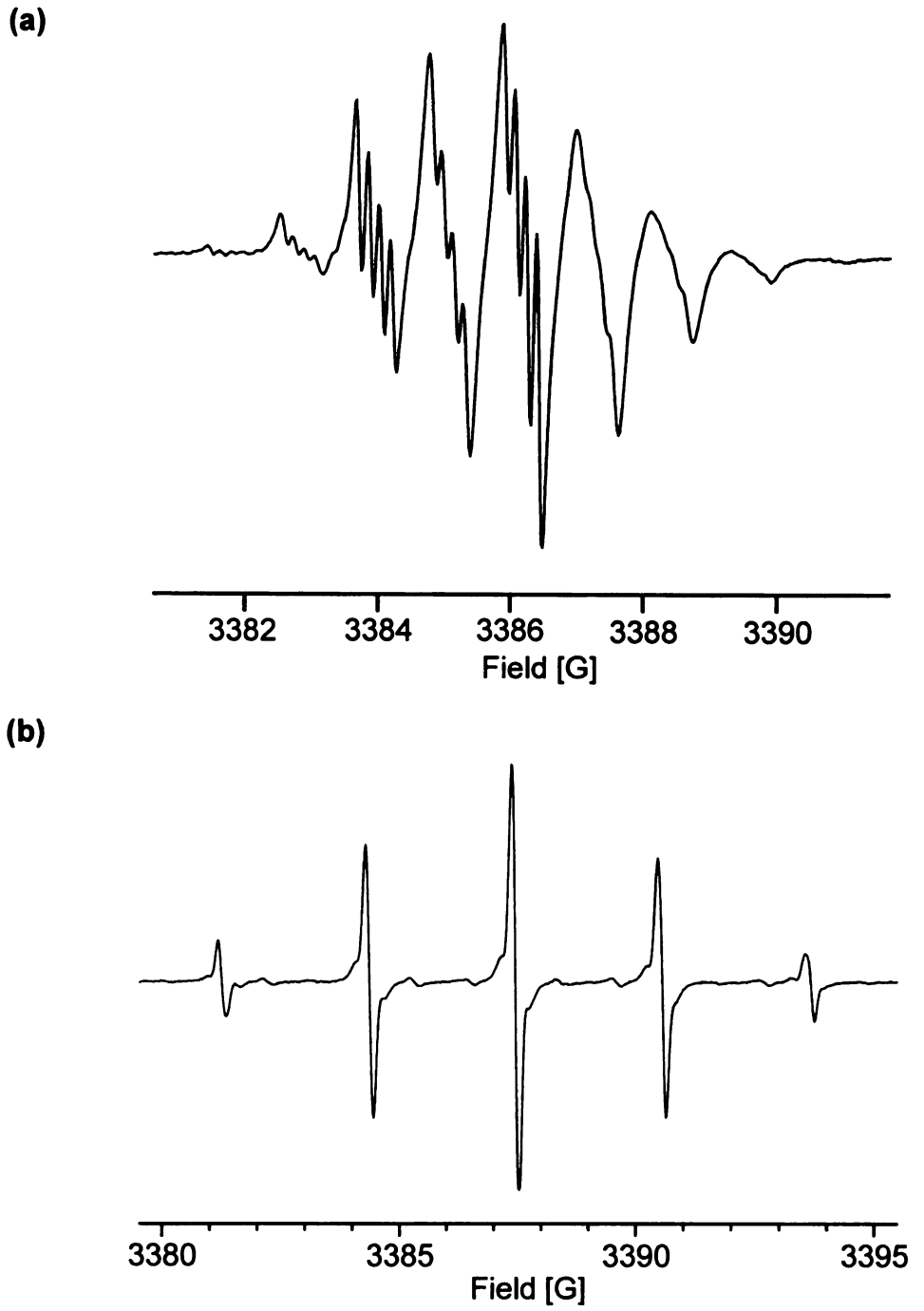


Figure 28. EPR spectrum of (a) $Li^{+}\bullet 1E^{-\bullet}$ (9-line spectrum) and (b) $(Li^{+})_2\bullet 1E^{2-\bullet\bullet}$ (5-line spectrum) in tetrahydrofuran (THF) at 293.0 K.

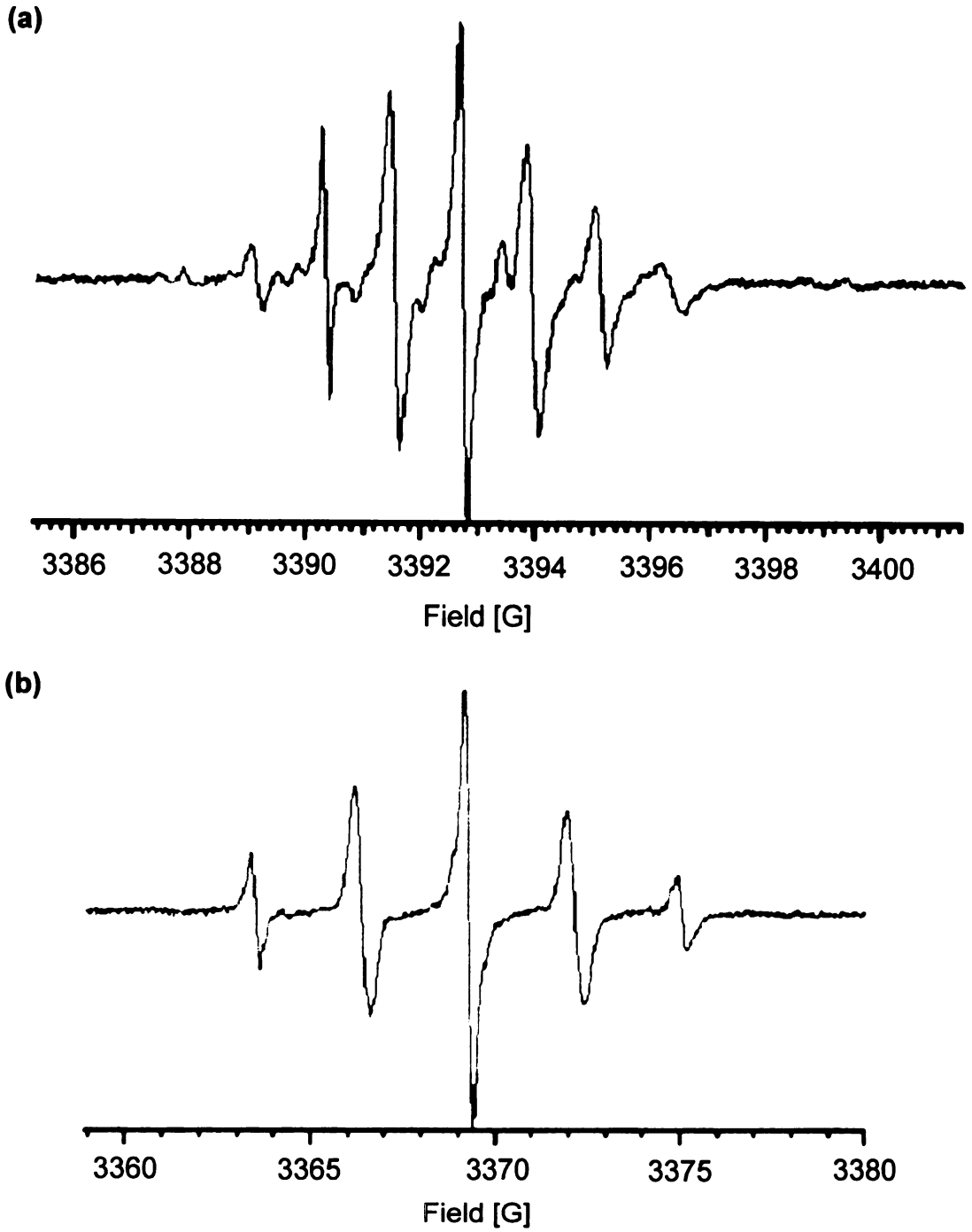


Figure 29. EPR spectrum of (a) $K^{+}\bullet 1E^{-\bullet}$ (9-line spectrum) and (b) $(K^{+})_2\bullet 1E^{2-\bullet\bullet}$ (5-line spectrum) in tetrahydrofuran (THF) at 298.0 K and 290.0 K, respectively.

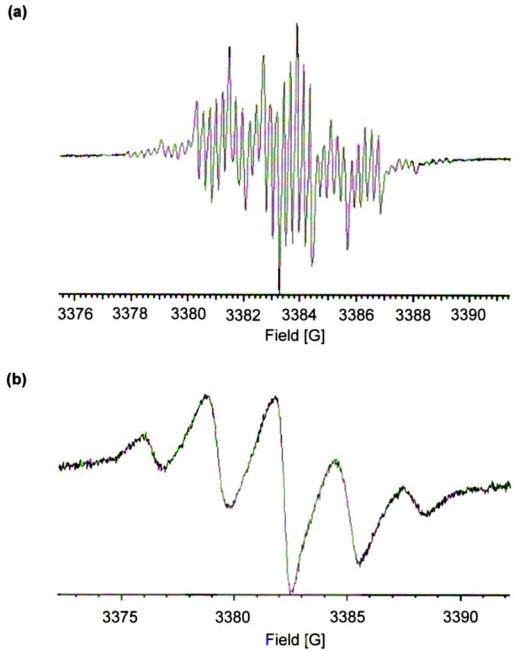


Figure 30. EPR spectrum of (a) $Cs^{+}\bullet 1E^{-\bullet}$ (9-line spectrum) and (b) $(Cs^{+})_2\bullet 1E^{2-\bullet\bullet}$ (5-line spectrum) in 2-methyltetrahydrofuran (2-MTHF) at 290.0 K and 294.0 K, respectively.

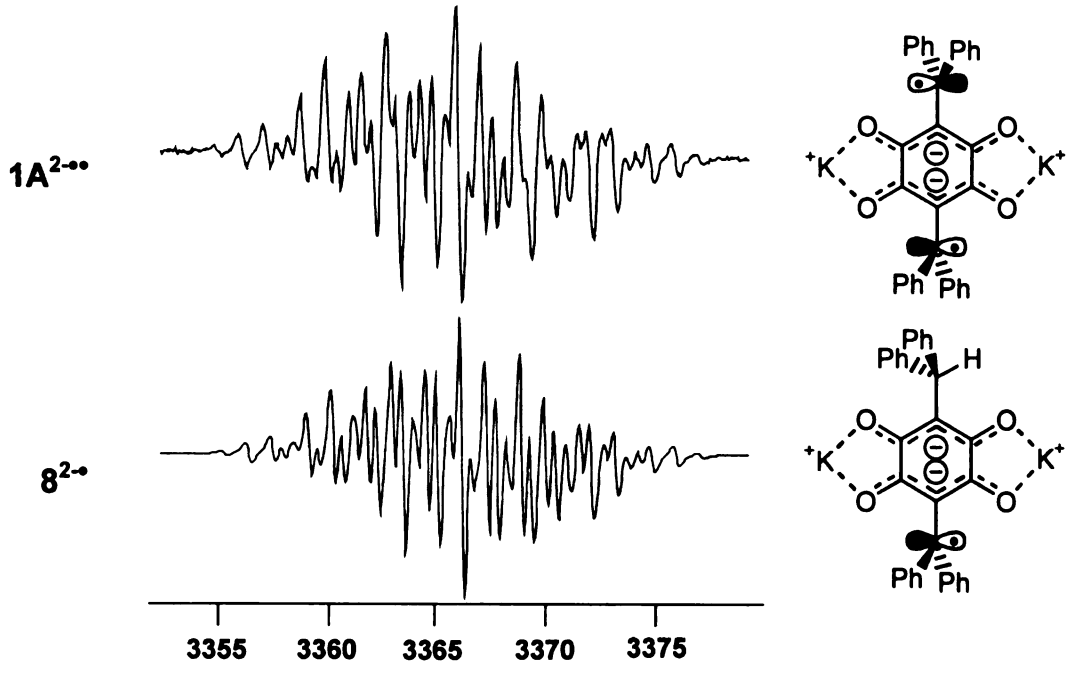


Figure 31. EPR spectra of $\mathbf{8}^{2-\bullet}$ and $\mathbf{1A}^{2-\bullet\bullet}$ at 280 K.

of 30-40 degrees about these bonds (*vide infra*). While anions derived from hydrocarbons tend to have their charge and spin correlated, this connection breaks down when heteroatoms are involved.[50] The pinacol coupling[51] and the similarities in spin densities[52] between benzophenone ketyls $Ar_2CO^{-\bullet}$ and the corresponding triarylmethyl radicals $Ar_3C^{\bullet}$ are, in part, a consequence of this effect. Similarly, charge localization at oxygen sites in the dianions $\mathbf{1}^{2-\bullet\bullet}$ due to strong association with the metal cations may displace spin density to the diarylmethylene subunits. Since, there is less delocalization than the monoanions, the dianions may be expected to have lower barriers to diarylmethyl twisting, consistent with the apparent electron localization seen here. This

hypothesis is strengthened by the absence of hyperfine coupling to the metal cations lithium(I) and cesium(I) in the solution EPR spectra of the diradical dianions. The only evidence of minor contact density at cesium(I) is a broader linewidth relative to the $(Li^+)_2 \bullet 1E^{2-\bullet\bullet}$ complex.

In order to determine the ground electronic states of the tetrones, the complexes $(Li^+)_2 \bullet 1E^{2-\bullet\bullet}$ and $(Cs^+)_2 \bullet 1E^{2-\bullet\bullet}$ were synthesized and then analyzed by temperature dependant EPR spectroscopy (see Appendix D for all temperature dependent EPR spectra). For a visual comparison, the EPR spectra of the complexes $(Li^+)_2 \bullet 1E^{2-\bullet\bullet}$ and $(Cs^+)_2 \bullet 1E^{2-\bullet\bullet}$ in a frozen matrix of tetrahydrofuran at 90 K are overlaid in Figure 32. The $(Cs^+)_2 \bullet 1E^{2-\bullet\bullet}$ complex was found to have a central transition at ~3374 G with a peak-to-peak linewidth of about 10 Gauss, while that of the $(Li^+)_2 \bullet 1E^{2-\bullet\bullet}$ complex exhibits a similar central transition and what appears to be a triplet signal with very small zero field splitting (ZFS) accompanied by a narrow monoradical signal; most likely due to the incomplete conversion of the monoradical monoanion into the diradical dianion. Another interesting feature of the temperature dependant EPR spectra is all EPR signals, for both the monoradical monoanions and diradical dianions with both the lithium(I) cation and cesium(I) cation, increase as the temperature is raised and reaches a maximum at 110 K for the lithium adduct and 87.5 K for the cesium adduct. Since, the monoradical and the diradical signals have a similar behavior, they must be caused by a common factor. ^1H hyperfine splitting to the lithium(I) cation was ruled out due to the negligible contact density in the diradical dianion solution, however, isotropic hyperfine coupling to four equivalent ortho hydrogen

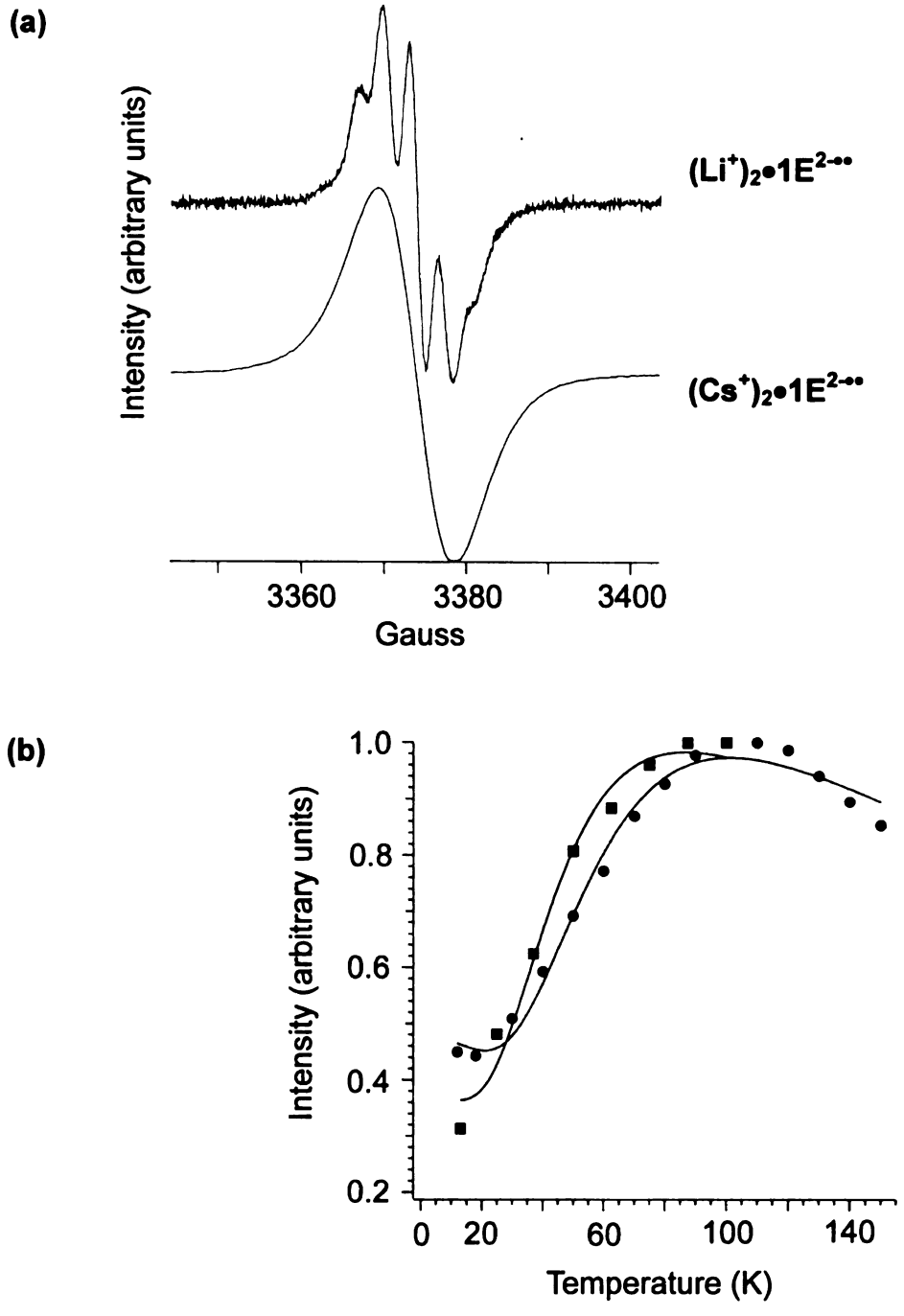


Figure 32. (a) EPR spectra of $(Li^+)_2 \bullet 1E^{2-\bullet\bullet}$ and $(Cs^+)_2 \bullet 1E^{2-\bullet\bullet}$ in a frozen matrix of tetrahydrofuran at 90 K and 75 K, respectively (b) Temperature dependence of the EPR signal intensity of the thermally populated triplet states of $(Li^+)_2 \bullet 1E^{2-\bullet\bullet}$ (circles) and $(Cs^+)_2 \bullet 1E^{2-\bullet\bullet}$ (squares).

atoms of the triarylmethyl fragments accounts for the lineshape and the temperature dependant behavior. Weak electron spin dipolar interactions, which give rise to zero field splitting, were found in the diradical dianion and the linewidth places an upper limit on $|D| = 5x10^{-4}$ cm^{-1}, where the D value reflects the magnetic interaction between the spins of the unpaired electrons and is proportional to r^{-3}, where r is the average distance between unpaired spins. The cesium adduct, $(Cs^+)_2 \bullet 1E^{2-\bullet\bullet}$, displayed weak sidebands (Appendix D), with a separation of ~120 G, which are caused by intermolecular dimers formed through ion-pair contacts. This was found while conducting experiments on the parent tetrone, $1A^{2-\bullet\bullet}$, with various alkali metals and with and without cryptand[2.2.2]. This will be discussed later in this section. A fit of the normalized EPR intensity versus temperature, between 4 K and 150 K, with a Bleaney-Bowers expression plus a Curie-law correction revealed the dianion diradical's possess singlet ground electronic states with small exchange parameters of -97(14) cm^{-1} and -124(6) cm^{-1} for $(Cs^+)_2 \bullet 1E^{2-\bullet\bullet}$ and $(Li^+)_2 \bullet 1E^{2-\bullet\bullet}$ adducts, respectively.

Solution EPR studies of the parent compound, $1A^{2-\bullet\bullet}$, produced different spectra depending on whether contact ion-pairs were allowed to form. When **1A** was reduced with two equivalents of potassium metal and 15-crown-5 in tetrahydrofuran (THF) to form the complex $(K^+)_2 \bullet 1A^{2-\bullet\bullet}$, a distinct triplet spectrum with $|D|=0.0065$ cm^{-1} and a faint but characteristic half-field transition was observed. The |D|-values were dependent on the metal and the reducing agents used, but were found to be smaller than 0.0065 cm^{-1}. However, the EPR spectrum of the frozen solution only consisted of a single line, with $\Delta H_{pp}=10$ G,

when contact ion-pair formation was prevented by using potassium metal and cryptand[2.2.2] in the preparation of $\mathbf{1A}^{2-\bullet\bullet}$. The temperature dependence of this signal indicated that $\mathbf{1A}^{2-\bullet\bullet}$ has a ground state singlet. Due to the presence of a monoradical signal with Curie-law behavior that offset the increase in the integrated intensities, the exchange parameter could not be reliably extracted from the data. It was now apparent that intermolecular ion-pair contacts in all of the tetrone complexes were responsible for the triplet signals observed and that these contacts were formed to a much greater extent in $\mathbf{1A}^{2-\bullet\bullet}$ than in $(\mathbf{Cs}^{+})_2\bullet\mathbf{1E}^{2-\bullet\bullet}$; most likely due to the eight t-butyl and the four methoxy groups sterically protecting the aromatic rings.

Theoretical calculations[10] predicted that the singlet-triplet gap of the diradical dianion could be tuned by perturbing the frontier molecular orbitals, **A** and **B**, with suitable cations and substituents when the geometry is constrained to be planar. However, crystallographic analysis of **1A** and **1C** revealed that even in their neutral unreduced form exist in chair-like conformations. Although no crystal data exists for **1D** and **1E**, there is no reason to believe that they would differ from this trend. Separation of the charge and spin as a result of bond torsion, which has lead to weak electron exchange and spin dipolar coupling in $(\mathbf{M}^{+})_2\bullet\mathbf{1E}^{2-\bullet\bullet}$ (M^+ is an alkali metal cation) in the diradical dianions revealed a powerful weakness within the present tetrone system and should be corrected in future work. The phenyl rings should be replaced with other non-aromatic groups (but with various electron-donating substituents to prevent the loss of tunability) in order to drive the unpaired electrons into the core framework, which would

create stronger magnetic interactions between the unpaired spins on the organic unit and the metal center. One of the major downfalls in the design of organometallic magnetic systems is providing the unpaired electrons an effective and efficient pathway via which they may communicate with each other intramolecularly and intermolecularly to generate materials with targeted magnetic properties.

2.9: Conclusions

Spectroscopic and solution studies of the tetrones, **1A**, **1C**, **1D**, and **1E**, support the near degeneracy of the frontier orbitals as well as the tunability of their energy gap by methylene site substitution. Previous theoretical calculations[10] suggest that diamagnetic or paramagnetic metal cations of greater charge, and smaller ionic radii, may tune the organic spin-carrier to be a ground state triplet. Currently, our diradical dianions possess a ground state singlet unless contact ion-pair formation is allowed, and even then the magnetic spin interactions are weak at best.

X-ray diffraction studies of the compound $(\mathbf{1E^{\bullet-}})_2\bullet(\mathbf{Na^+})_2\bullet(\mathbf{C_4H_{10}O_2})_8$ and its analogous potassium salt demonstrated the ability of the tetrone framework to coordinate into polymeric chains, through complexation with metal cations, while serving as an electron carrier. However, crystallization of the diradical dianion with metal cations of greater charge remains elusive. The SQUID data and EPR spectrum of $(\mathbf{1E^{\bullet-}})_2\bullet(\mathbf{Na^+})_2\bullet(\mathbf{C_4H_{10}O_2})_8$ were not serendipitous, considering there is an interchain distance of ~17.00 Å; taken together they suggest the organic unit

bears the spin density of one unpaired electron and has magnetic properties resembling those of a simple paramagnet. The Visible / near-IR confirmed, through a conproportionation reaction, the stability and reversibility between $\mathbf{1E}^{-\bullet}$ and $\mathbf{1E}^{2-\bullet\bullet}$, but only under anaerobic conditions. However, all of the species **1E**, $\mathbf{1E}^{-\bullet}$ and $\mathbf{1E}^{2-\bullet\bullet}$ experience decomposition at various rates upon contact with oxygen gas and should be handled with the utmost care; air sensitivity is another undesirable property of organic spin-carriers.

The solubility of $\mathbf{1E}^{2-\bullet\bullet}$ poses a real challenge; the eight *t*-butyl groups were added to increase its solubility in organic media, but X-ray quality crystals of its salts remain elusive. The ideal solvent mix must dissolve the neutral tetrone, tolerate the reduction chemistry, and compete with the strong coulombic attractions between cations and the doubly charged anion effectively enough to allow slow crystal growth rather than rapid precipitation of disordered materials.

CHAPTER 3

ORGANIC COMPLEXES AND NETWORKS OF 2

3.1: Introduction

The tetrone **1E** proved to be a viable organic building block capable of being reduced to $\mathbf{1E}^{-\bullet}$ by a one electron transfer from an alkali metal mirror under anaerobic conditions to generate the paramagnetic metal-organic crystalline salt $(\mathbf{1E}^{-\bullet})_2\bullet(Na^+)_2\bullet(C_4H_{10}O_2)_8$. X-ray analysis of this material revealed that it existed in coordinated polymeric chains, like the analogous potassium salt studied by Andrzej Misiolek. Both salts exhibited a similar paramagnetic behavior but with the potassium salt having weaker spin-spin interactions. It was theorized that higher dimensionalities would improve the electron-electron interactions and could be achieved if multiply charged cations, those typically found on alkaline earth metals and/or transition metals, were substituted for the alkali metals or by replacing the monoradical monoanion with the diradical dianion, $\mathbf{1E}^{2-\bullet\bullet}$. However, repeated attempts at incorporating the dianion or monoanion into a crystalline solid with a multiply charged metal center failed despite the various conditions and solvents that were used, except for the two crystals previously mentioned.

As a stepwise approach to the problem, we decided to study **2A**-**2E**, the structurally analogous dihydroxyquinone precursors of **1A**-**1E**, to ascertain whether the dimensionality of the tetrones could be increased beyond linear structures and into honeycomb sheets or diamond-like networks. For this study,

organic bases for deprotonation of the two acidic sites, which have pK_a values[26] of 2.95 and 4.87, and/or compounds capable of forming hydrogen bonds, were used to explore the coordination of the dihydroxyquinones in hopes that they would provide some insight into potential crystallization strategies for the tetrones and their paramagnetic anions or provide answers explaining the difficulties of crystallization found for the tetrones.

3.2: Hydrogen Bonded Complexes

Each of the dihydroxyquinones **2A-E** were dissolved in various polar protic and aprotic solvents and mixed with a weak organic base, triethylamine, capable of coordinating, through hydrogen bonding, to the acidic protons, forming a salt. Approximately 2 - 4 mg of **2** was placed into a 5.0 mL scintillation vial. To it was added 2 - 3 mL of one of the following solvents: methanol, ethanol, isopropanol, acetone, tetrahydrofuran, ethyl acetate, dimethylsulfoxide, diethyl ether, *N*,*N*-dimethylformamide, dichloromethane, chloroform, or toluene. Triethylamine (5 to 10 drops from a disposable pipet) was then added in excess to the vial, which caused the yellow solution to undergo an immediate color change to a shade of red, varying in darkness. The vial was then capped, shaken vigorously and allowed to stand for 24 hours at room temperature in order for an equilibrium to be established. After 24 hours, if the vial contained crystals they were extracted and taken for X-ray analysis and if it did not a syringe was used to remove 1.0 mL of the solution, avoiding any small particulate matter, and the solution was placed in an NMR tube. To the NMR tube was added *n*-octane to

fill the remaining volume. The NMR tube was then capped and set aside undisturbed for crystal growth by diffusion.

The tubes were checked continuously over a period of weeks and even months for signs of crystal growth. NMR tubes containing crystals were examined under a microscope for crystals suitable for X-ray analysis. Each of the dihydroxyquinones **2A-E** crystallized with triethylamine (except **2C**) to produce the hydrogen-bonded complexes shown in Figures 33 through 36. Selected bond distances and torsion angles are listed in Tables 8 and 9 respectively while all crystallographic data for each organic salt is in Appendix E.

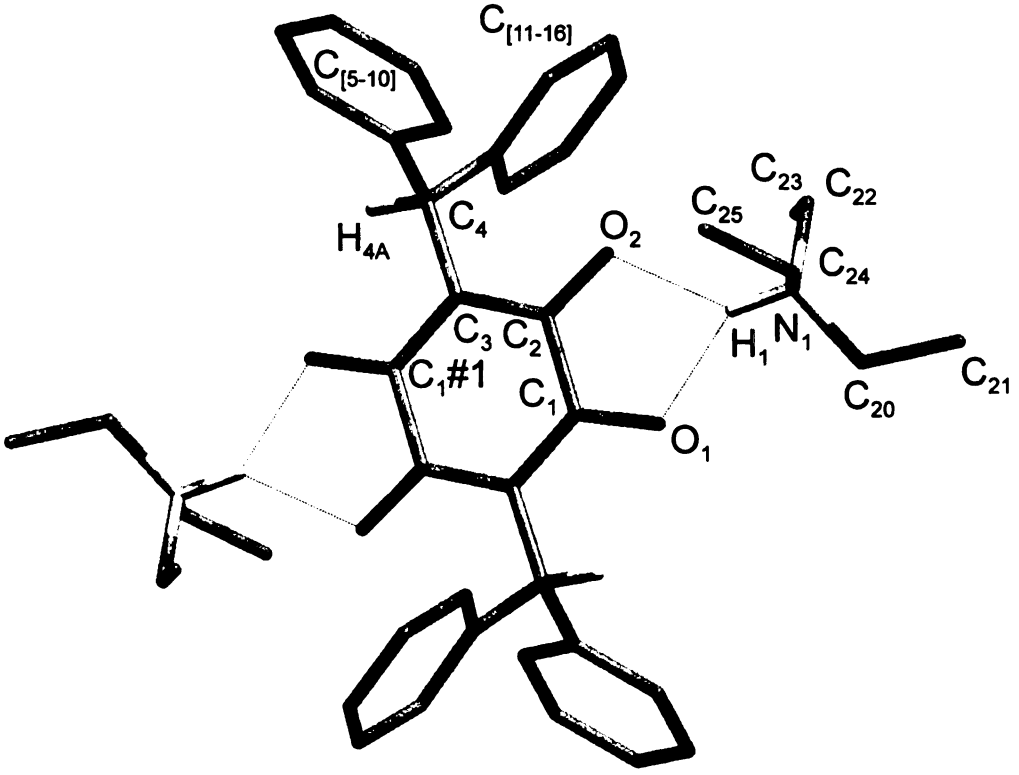


Figure 33. View of **2A•($C_6H_{15}N$)$_2$** down the b-axis. All non-essential hydrogen atoms have been omitted for clarity.

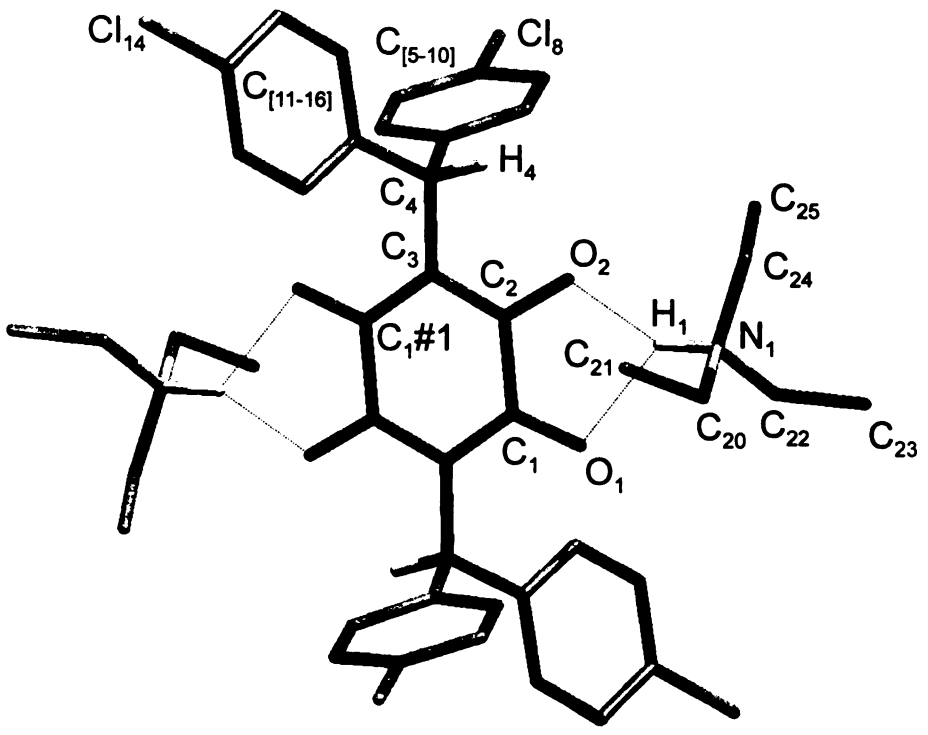


Figure 34. View of **2B•($C_6H_{15}N$)$_2$•(C_3H_6O)$_2$**. All non-essential hydrogen atoms and acetone solvent molecules have been omitted for clarity.

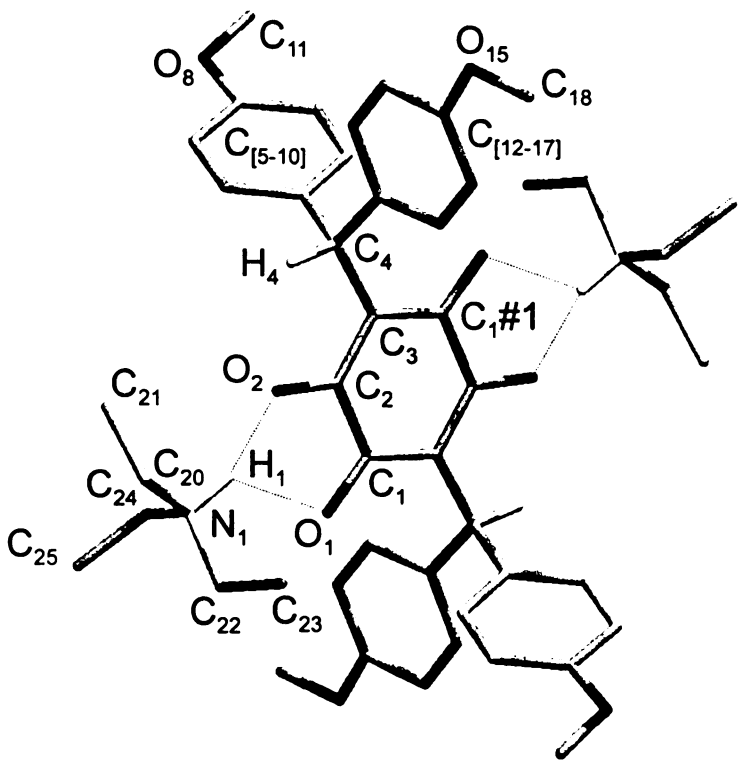


Figure 35. View of **2D•($C_6H_{15}N$)$_2$**. All non-essential hydrogen atoms have been omitted for clarity.

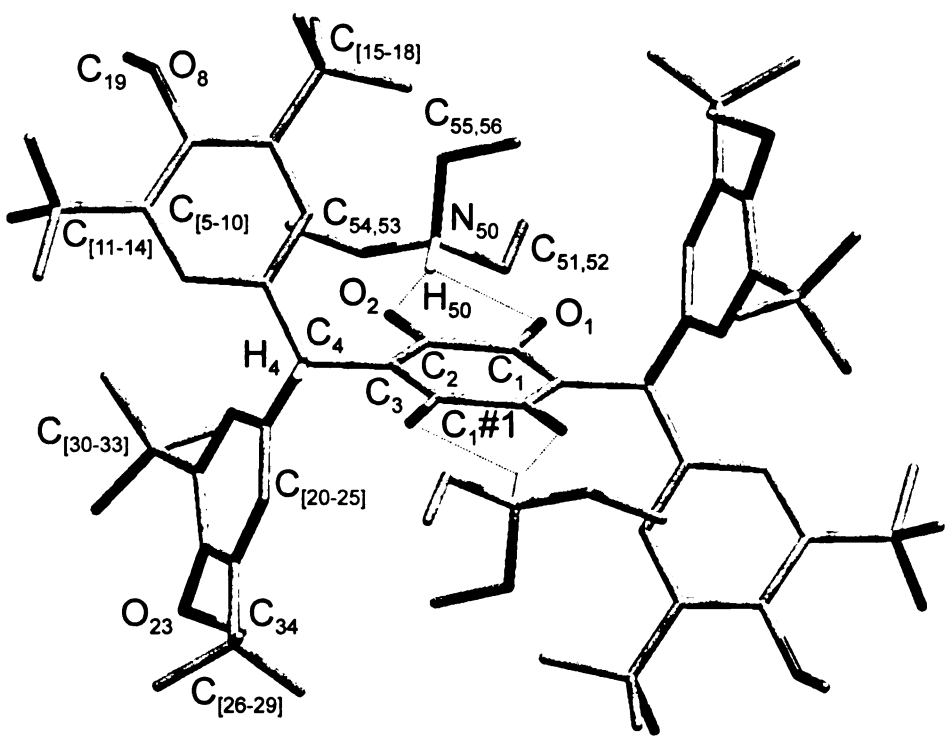


Figure 36. View of **2E•($C_6H_{15}N$)$_2$**. All non-essential hydrogen atoms have been omitted for clarity.

Table 8. Selected bond lengths for **2A-E**.

	Molecule			
Atoms	**2A**	**2B**	**2D**	**2E**
O_1-H_1	1.956*	2.14(2)	2.340(14)	2.47(2)**
O_2-H_1	2.029*	1.84(2)	1.793(15)	1.80(3)**
C_1-O_1	1.264(2)	1.246(2)	1.2504(12)	1.251(2)
C_2-O_2	1.256(2)	1.261(2)	1.2671(12)	1.268(2)
C_1-C_2	1.539(3)	1.540(3)	1.5459(14)	1.540(2)
C_2-C_3	1.408(2)	1.400(3)	1.3986(13)	1.394(2)
C_3-C_1#1	1.404(2)	1.413(3)	1.4148(14)	1.420(2)
C_3-C_4	1.522(3)	1.524(3)	1.5214(14)	1.520(2)

Bond lengths are in Å. The values marked (*) were calculated. The values marked (**) represent the distance to H_{50} instead of H_1. Atoms marked (#1)-symmetry operator used to generate equivalent atoms.

Table 9. Selected torsion angles for **2A-E**.

	Molecule			
Atoms	**2A**	**2B**	**2D**	**2E**
C_1#1-C_3-C_4-H_{4A}	19.46			
C_2-C_3-C_4-H_4		9.10	6.53	
C_1#1-C_3-C_4-H_4				10.16

Bond angles are in °. Atoms marked (#1)-symmetry operator used to generate equivalent atoms.

Figures 33 through 36 show the relative ease at which **2A-E** was deprotonated by triethylamine to form dark red organic crystalline complexes. The hydrogen, which is now located on the nitrogen, forms strong bifurcated

hydrogen bonds to each of the oxygens' lone-pair electrons with bond distances ranging from 1.793(15) Å to 2.47(2) Å. Further evidence for the deprotonation of **2A-E** is the similar carbon-carbon bond lengths that can be observed across the periphery of the core. The bond lengths observed for C_2-C_3 and C_3-C_1#1 (C_1#1 is the symmetry equivalent of C_1) are within the range of 1.394(2) Å to 1.420(2) Å and essentially identical meaning the electron density is evenly distributed from one oxygen atom to another across the core while the C_1-C_2 bond length of ~1.54 Å suggests there is virtually no conjugation between neighboring carbonyls. This was also observed for the tetrone crystal.

The second interesting feature of these crystals is the C_4 position where the methyne hydrogen resides. The methyne hydrogen atoms, H_4 or H_{4A}, are nearly coplanar to the core of the molecule. This places the phenyl rings in positions where they would feel the fewest steric and electronic interactions. The bond rotation that takes place along the C_3-C_4 bond in the dihydroxyquinone complexes is similar to the twisting found when **1E** is reduced to the monoradical monoanion by a sodium metal mirror and crystallized to generate the organometallic salt $(\mathbf{1E^{\bullet-}})_2{\bullet}(\mathbf{Na^+})_2{\bullet}(\mathbf{C_4H_{10}O_2})_8$. If the methyne hydrogen were to be replaced with an unpaired electron or a lone-pair of electrons it would essentially have no communication with the core or the metal cation that would be coordinated to the core. For communication or delocalization to take place the *p* orbital that the unpaired electron resides in must be parallel (maximizes orbital overlap) to the adjacent *p* orbital so orbital overlap can occur. Without orbital overlap the unpaired electron is essentially isolated from the core but not

from the phenyl rings as is observable by several spectroscopic techniques.

Although ammonium salts of **2** are magnetically uninteresting they are showing their structural relevancy to the tetrones of **1**. Triethylamine has successfully generated zero-dimensional molecular organic salts via the deprotonation of **2**. Therefore, if the monodentate ligand of triethylamine were to be replaced with a bidentate organic linker then linear chains similar to the paramagnetic salt of $(\mathbf{1E}^{-})_2 \cdot (\mathbf{Na}^{+})_2 \cdot (C_4H_{10}O_2)_8$ might be possible.

3.3: One-dimensional Networks

One-dimensional structures become possible if the organic base used contains more than one coordination site. Triethylamine was replaced with tetramethylethylenediamine (tmeda) a bidentate organic molecule that is capable of donating a lone pair of electrons from each of two nitrogen atoms. Solutions of **2A-E** were prepared in the same way as that mentioned in section 3.2 except that tetramethylethylenediamine was used instead of triethylamine. Most solutions turned red, which is indicative of the dianion of **2**, but failed to crystallize. Precipitation of a thin film or suspension was almost immediate upon the addition of tetramethylethylenediamine. Only **2E** was successfully crystallized from both acetone and ethyl acetate with tetramethylethylenediamine. The structures are virtually identical but only one of them is presented (see Figures 37 and 38). However, all crystallographic data for both crystals has been summarized in Appendix E.

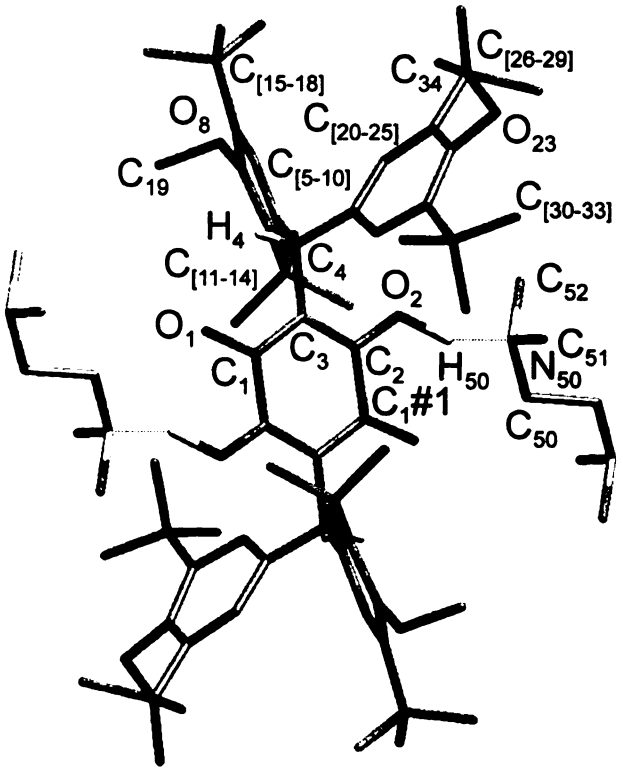


Figure 37. View of **2E•$C_6H_{16}N_2$** (from acetone) and **2E•$C_6H_{16}N_2$** (from ethyl acetate). All non-essential hydrogen atoms have been omitted for clarity.

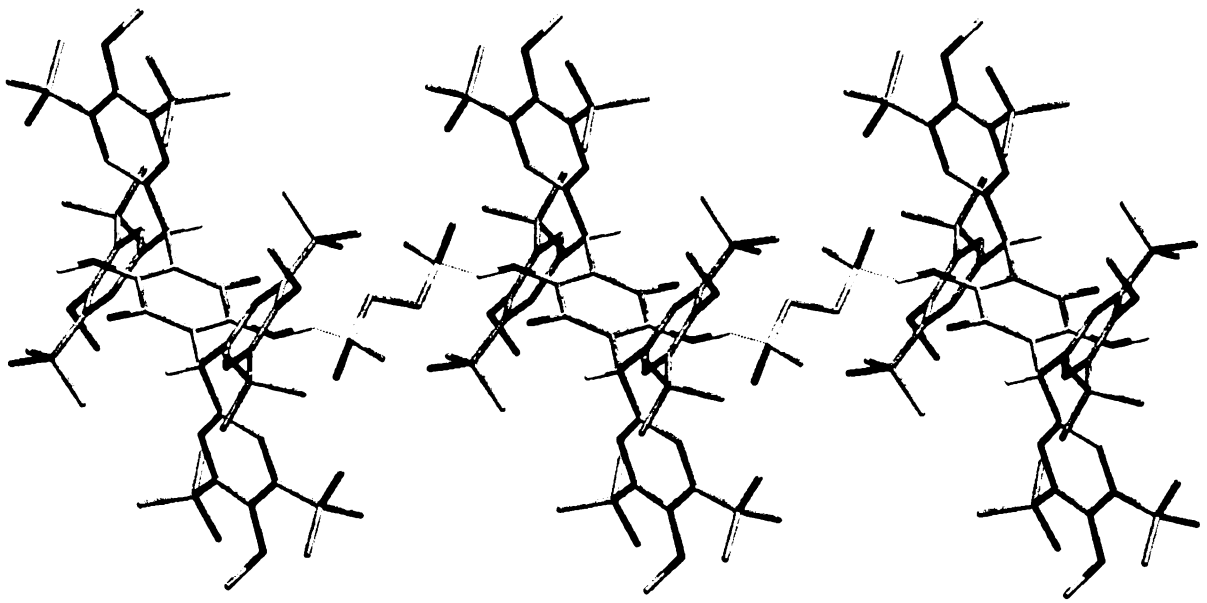


Figure 38. One-dimensional chain of **2E•$C_6H_{16}N_2$** (from acetone) or **2E•$C_6H_{16}N_2$** (from ethyl acetate) down the c-axis. All non-essential hydrogen atoms have been omitted for clarity.

As expected, tetramethylethylenediamine functions as a bidentate organic linker and forms linear chains through hydrogen bonding. The crystal structure reveals once again that the methyne hydrogen prefers a conformation that is nearly coplanar to that of the core minimizing the interactions that the phenyl rings may have with each other and the core framework. Selected bond lengths and torsion angles can be found in Tables 10 and 11 respectively. However, tetramethylethylenediamine does not completely remove the acidic proton from the diyhdroxyquinone core, which can be seen by comparing the carbon-carbon and carbon-oxygen bond lengths to each other.

Table 10. Selected bond lengths for **2E•$C_6H_{16}N_2$** (from acetone) and **2E•$C_6H_{16}N_2$** (from ethyl acetate).

	Molecule 2E•$C_6H_{16}N_2$	
Atoms	(from acetone)	(from ethyl acetate)
N_{50}-H_{50}	1.37(3)	1.36(3)
O_2-H_{50}	1.26(3)	1.26(3)
C_1-O_1	1.2318(19)	1.2342(17)
C_2-O_2	1.299(2)	1.2990(17)
C_1-C_3	1.437(2)	1.4355(19)
C_2-C_3	1.367(2)	1.3704(19)
C_1-C_2#1	1.527(2)	1.5284(18)
C_3-C_4	1.515(2)	1.5188(18)

Bond lengths are in Å. Atoms marked (#1)-symmetry operator used to generate equivalent atoms.

Table 11. Selected torsion angles for **2E**.

Atoms	Molecule 2E•$C_6H_{16}N_2$ (from acetone)	(from ethyl acetate)
C_1-C_3-C_4-H_4	6.16	8.25

Bond angles are in degrees (°).

From the crystal the bond lengths of C_1-C_3 and C_2-O_2 have more single bond character while those of C_2-C_3 and C_1-O_1 have more double bond character; this is consistent with the Lewis structure (shown in Chapter 2). This is not found in the crystal structures where triethylamine was used. However, they deviate slightly from the typical bond lengths ordinarily observed (see Table 5 in Chapter 2). A complete analysis of the crystallographic data revealed the electron density of the acidic proton (H_{50}) as diffuse and unevenly located between O_2 and N_{50} but the hydrogen atom remains in the vicinity and occupies the space closer to the oxygen atom for more time than in the presence of the nitrogen atom; this can be seen by the elongation of the O-H bond and the short N-H coordinated hydrogen bond. Also, the equilibrium process of proton transfer is what causes the deviations in the bond lengths and is the reason for the crystal to have a yellow-orange appearance. Solutions of the neutral compounds **2A-E** have a yellow appearance and change to red upon deprotonation with a base.

3.4: Two-dimensional Networks

Continuing with the same logic as before it might be feasible to construct

two-dimensional networks using organic bases capable of coordinating three ligands of **2**. Two-dimensional networks can exist in a variety of structural motifs, some of which are shown in Figure 39. Among them are the desired honeycomb sheets, and for this tris chelating organic molecules such as these were chosen: diethanolamine, triethanolamine, diethylenetriamine, tris(2-aminoethyl)amine, melamine, etc. Several of them formed two-dimensional networks, none of which formed the desired honeycomb motif, one formed a three-dimensional lattice (that is discussed in the next section) while many others (not mentioned) formed nothing except a thin film or a cloudy solution.

Tris(2-aminoethyl)amine has three terminal primary amines and one central tertiary amine each capable of deprotonation, forming hydrogen bonds or both. Due to insufficient space it would not be reasonable for tris(2-aminoethyl)amine to coordinate four molecules of **2**. However, it was expected that each terminal amine would deprotonate or hydrogen bond to one dihydroxyquinone site and coordinate three molecules of **2** in a propeller-like arrangement to form structures geometrically analogous to those found in Figures 6 and 7 in Chapter 1.

Examination of the crystal structure (see Figure 40) reveals only two deprotonations occur instead of the three deprotonations initially predicted, which may be a result of the build-up of coulombic charges on the tris(2-aminoethyl)amine molecule. It was also expected that each molecule of tris(2-aminoethyl)amine would coordinate three molecules of **2** to generate the desired honeycomb network. However, it was found that one terminal amine

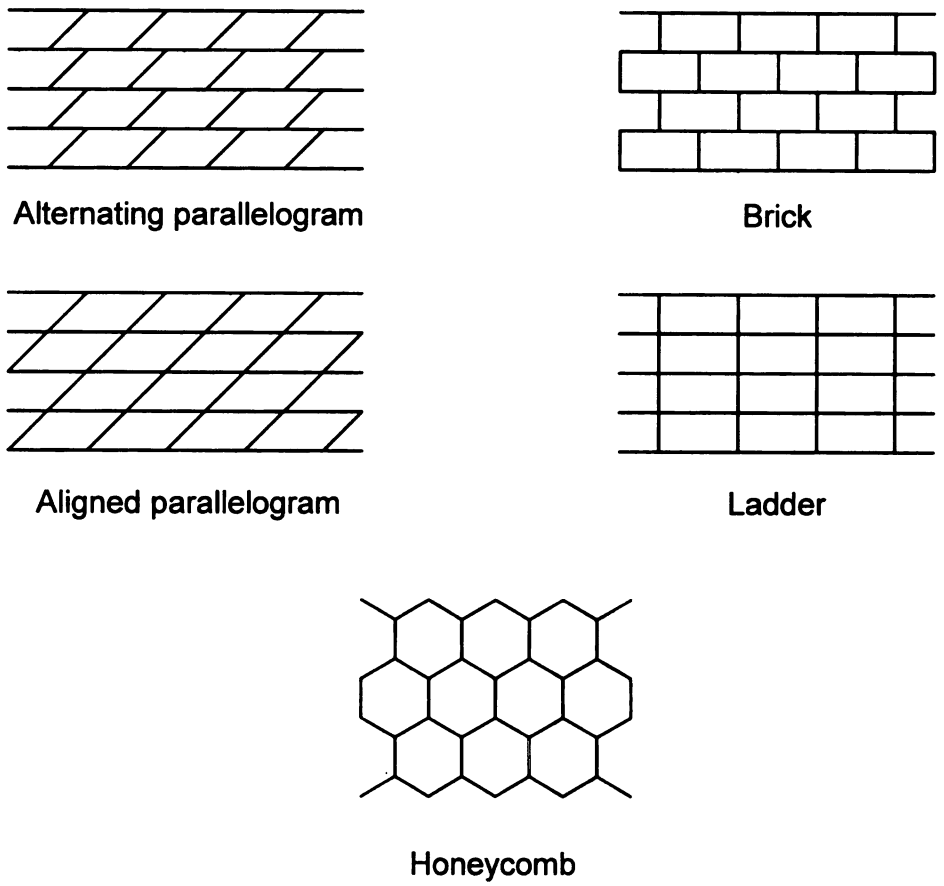


Figure 39. A few two-dimensional structural motifs.

forms hydrogen bonds, with distances ranging from 1.88(2) Å to 2.49(2) Å, to four oxygen atoms in two molecules of **2A** while the other proton is shared between the other two remaining ends of the tetraamine which is also coordinated to two oxygen atoms in two molecules of **2A** with hydrogen bond distances in the range of 1.83 Å to 2.21(3) Å. Sharing of the proton between N_{45A} and N_{43} created a more compact structure, which removed any void spaces that may have been created and ultimately led to a network other than the desired honeycomb motif.

Instead the crystal packing of **2A•$C_6H_{18}N_4$•C_2H_6O** (see Figure 41) formed two-dimensional aligned parallelogram sheets (see Figure 39) separated by the phenyl rings with the shortest distance being 5.027 Å.

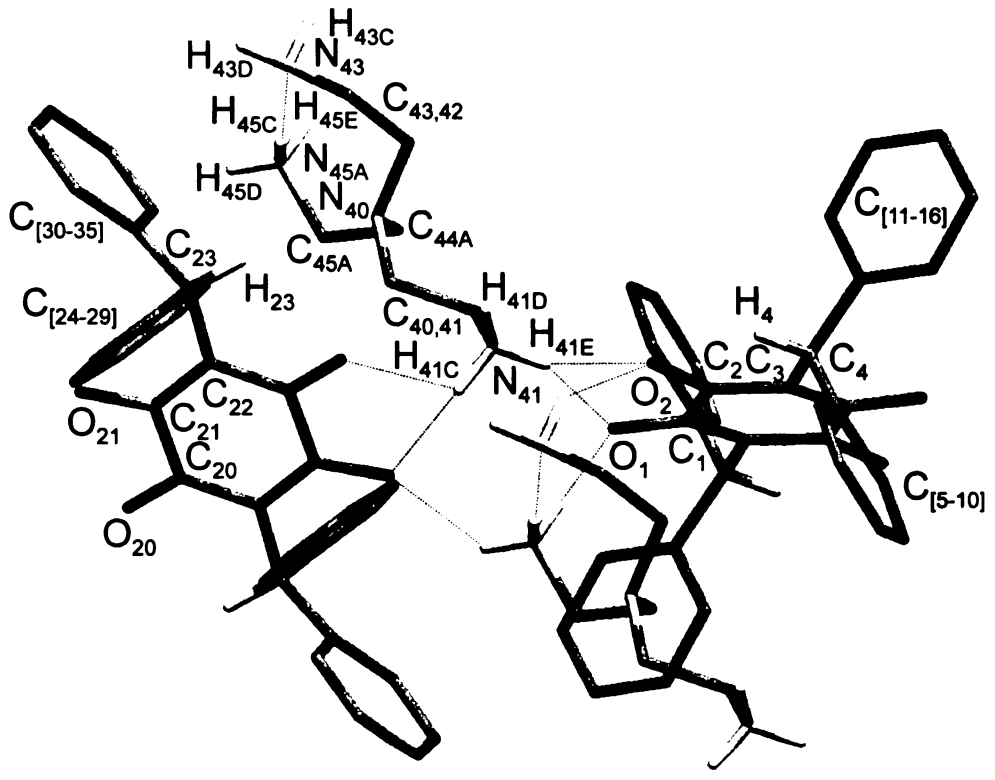


Figure 40. View of **2A•$C_6H_{18}N_4$•C_2H_6O**. All non-essential hydrogen atoms and ethanol solvent molecules have been omitted for clarity. There is disorder in the arm containing N_{40} through N_{45A} and only the major conformation has been shown.

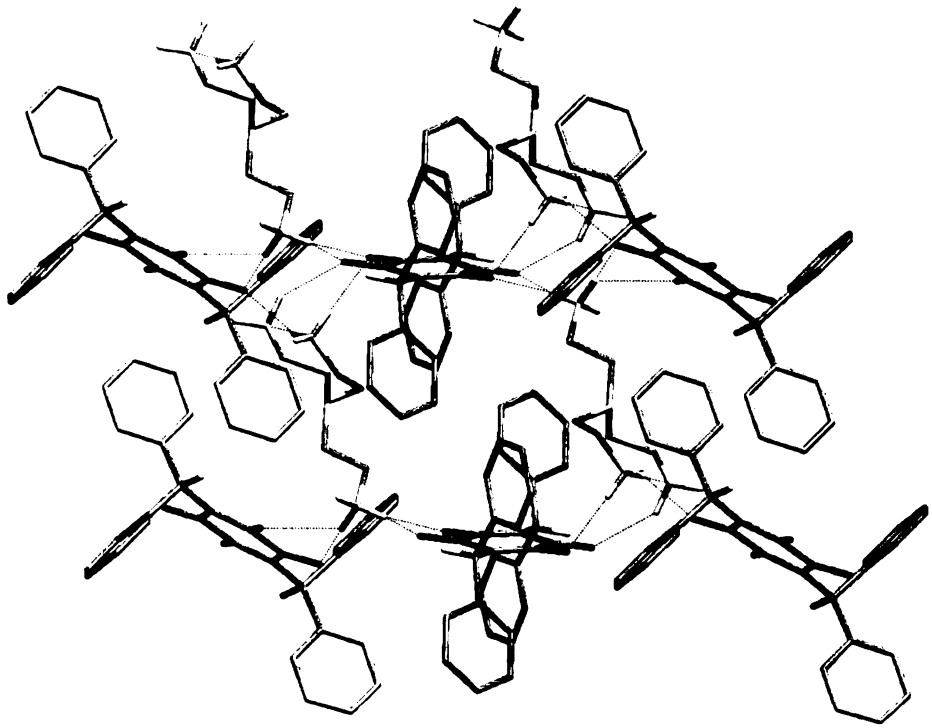


Figure 41. Packing of **2A•$C_6H_{18}N_4$•C_2H_6O** down the b-axis. All non-essential hydrogen atoms and ethanol solvent molecules have been omitted for clarity.

The flexibility of the aliphatic chains in tris(2-aminoethyl)amine allowed for various geometrical arrangements and in this case produced a structure other than the one intended. To remove the flexibility and reduce the number of conformations possible the molecule tris(2-aminoethyl)amine was replaced by the more compact and rigid molecule of melamine, a compound consisting of three primary amines all connected to an aromatic core with alternating carbon and nitrogen atoms. Melamine is larger than a metal center but could coordinate three molecules of **2** into a propeller-like arrangement revealing if there is

sufficient space for three molecules of $\mathbf{1}^{2-\bullet\bullet}$ to coordinate around a metal center. Of the five molecules, **2A-E**, only **2A** crystallized with melamine from an isopropanol solution to generate the two-dimensional structure observed in Figure 44. The unique molecules that comprise the unit cell are shown in Figure 43.

Crystallographic analysis reveals that the dihydroxyquinone core of **2A** exists as the monoanion with one acidic proton removed by N_{53} on melamine. Examination of the cores' bond lengths provides evidence for the existence of the monoanion instead of the neutral or dianion (see Figure 42) that has been observed in crystals previously mentioned. The carbon-oxygen bond lengths of C_1-O_1 and C_5-O_5 are identical and the bond lengths of C_1-C_6 and C_6-C_5 are nearly identical suggesting the negative charge is delocalized across these four bonds (generating bond orders larger than one but less than two) while there is a distinct difference between the carbon-oxygen bond lengths of C_2-O_2 and C_4-O_4 and carbon-carbon bond lengths of C_2-C_3 and C_3-C_4. All crystallographic data has been summarized in Appendix E.

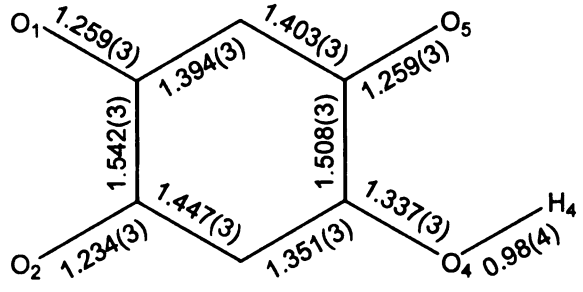


Figure 42. Selected bond lengths of the monoanion core in **2A•$C_3N_6H_6$•C_3H_8O**.

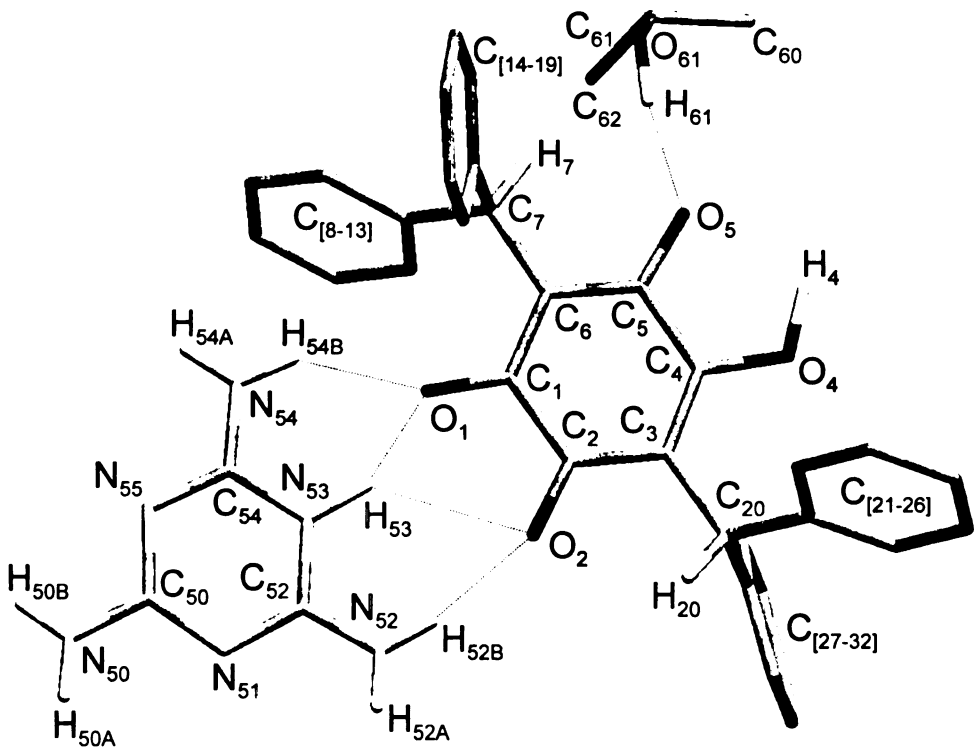


Figure 43. View of **2A•$C_3N_6H_6$•C_3H_8O** down the c-axis. All non-essential hydrogen atoms have been omitted for clarity.

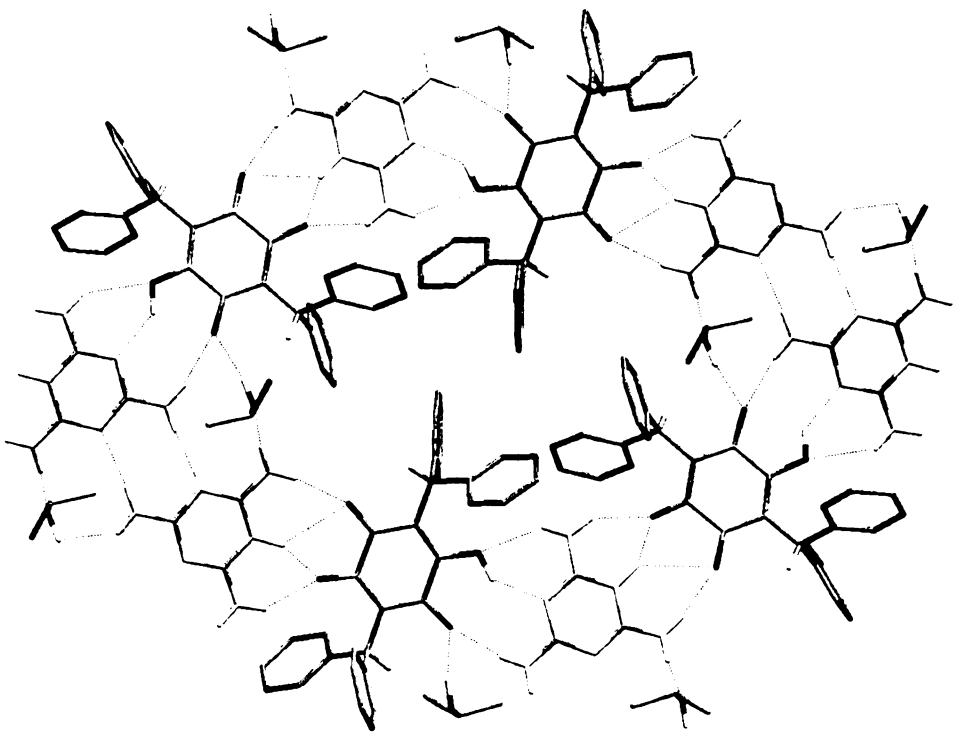


Figure 44. Packing of **2A•$C_3N_6H_6$•C_3H_8O** down the c-axis. All non-essential hydrogen atoms have been omitted for clarity.

The organic base melamine removed one dihydroxyquinone proton with one of the nitrogen atoms located within the aromatic ring. This is expected and observed in this crystal structure. Each of the three amino nitrogen atoms contributes significant electron density to the core through their corresponding resonance contributor. The bond angles around each of these nitrogen atoms suggests they are sp^2 hybridized giving each nitrogen atom a formal charge of positive one. This reduces their basicity in comparison with any of the three nitrogen atoms within the ring of the core. The nitrogen atoms contained within the ring are sp^2 hybridized as well, but each of these nitrogen atoms carries a lone-pair of electrons in an sp^2 orbital that is orthogonal to the circuitous π cloud that exists above and below the plane of the ring. It is obvious at this point that once the first proton has been added to melamine a second proton addition is unlikely. Coulombic repulsions between similar charges are too great for the addition of a second proton.

Melamine forms strong hydrogen bonds (listed in Appendix E) with two molecules of **2A**, one molecule of isopropanol and another molecule of melamine to generate two-dimensional sheets with a separation of 8.233 Å (the closest contact between melamine molecules). The phenyl rings and solvent molecules of isopropanol, which are coordinated through hydrogen bonds to both melamine and **2A**, fill the regions of space between two-dimensional sheets.

Unfortunately two-dimensional honeycomb sheets were not formed but again they are the least likely to form since they must create large void spaces that can be thermodynamically and/or entropically unfavorable. Hydrogen

bonding has been an effective pathway by which multidimensional networks of **2** have been constructed. It was hypothesized that the replacement of the terminal amino groups in molecules of diethylenetriamine and tris(2-aminoethyl)amine with terminal alcohol functionalities may generate different two-dimensional or three-dimensional structures (and hopefully the desired honeycomb array or diamond-like network). However, like their structurally relevant cousins the amines, they too fall prey to the flexibility of their skeletal structure and adopt the most compact geometry possible creating two-dimensional sheets. Figures 45 through 47 display the crystal structure of **2A** with diethanolamine and Figures 48 and 49 display the crystal structure of **2A** with triethanolamine. All crystallographic data for both crystal structures are summarized in Appendix E.

In both hydrogen-bonded organic crystalline complexes diethanolamine and triethanolamine generated the dianion of **2A** and the ammonium cation by deprotonation. There are multiple hydrogen bonds in each system creating a tightly compacted structure producing two-dimensional sheets with each layer separated by a distance of 3.986 Å and 4.041 Å (closest contact between carbon atoms in the phenyl rings) respectively. The core of **2A**, where the two negative charges reside and are delocalized across the periphery of the core, is fully deprotonated based on measured atomic distances between the nuclei (see Table 12). Further evidence that the core of **2A** is doubly deprotonated is the tightening of some of the hydrogen bonds between each of the oxygen atoms of **2A** and the terminal alcohol groups of diethanolamine or **2A** and the terminal alcohol groups of triethanolamine. The hydrogen bonds are listed in Table 13.

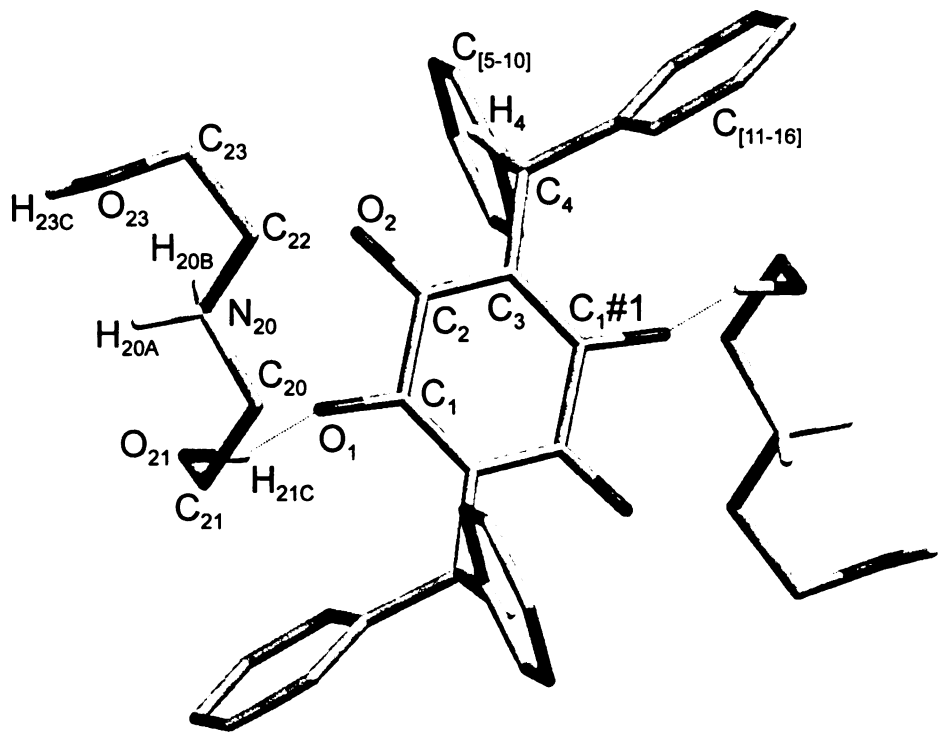


Figure 45. View of **2A•($C_4H_{11}NO_2$)$_2$**. All non-essential hydrogen atoms have been omitted for clarity.

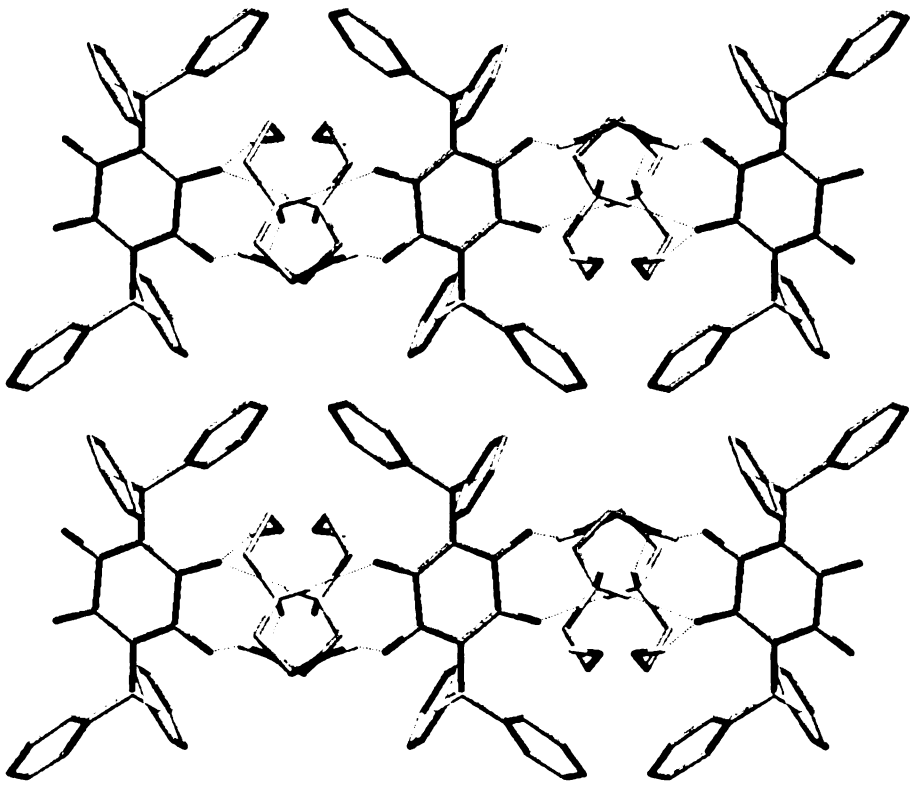


Figure 46. Packing of **2A•$(C_4H_{11}NO_2)_2$** down the c-axis. All non-essential hydrogen atoms have been omitted for clarity.

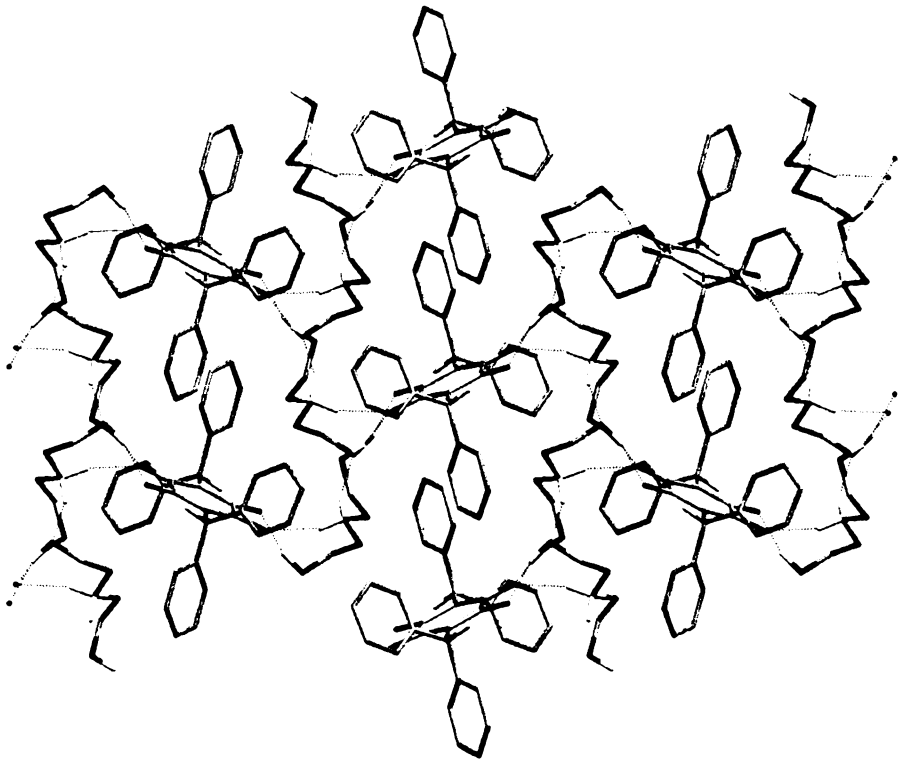


Figure 47. Packing of **2A•$(C_4H_{11}NO_2)_2$** down the a-axis. All non-essential hydrogen atoms have been omitted for clarity.

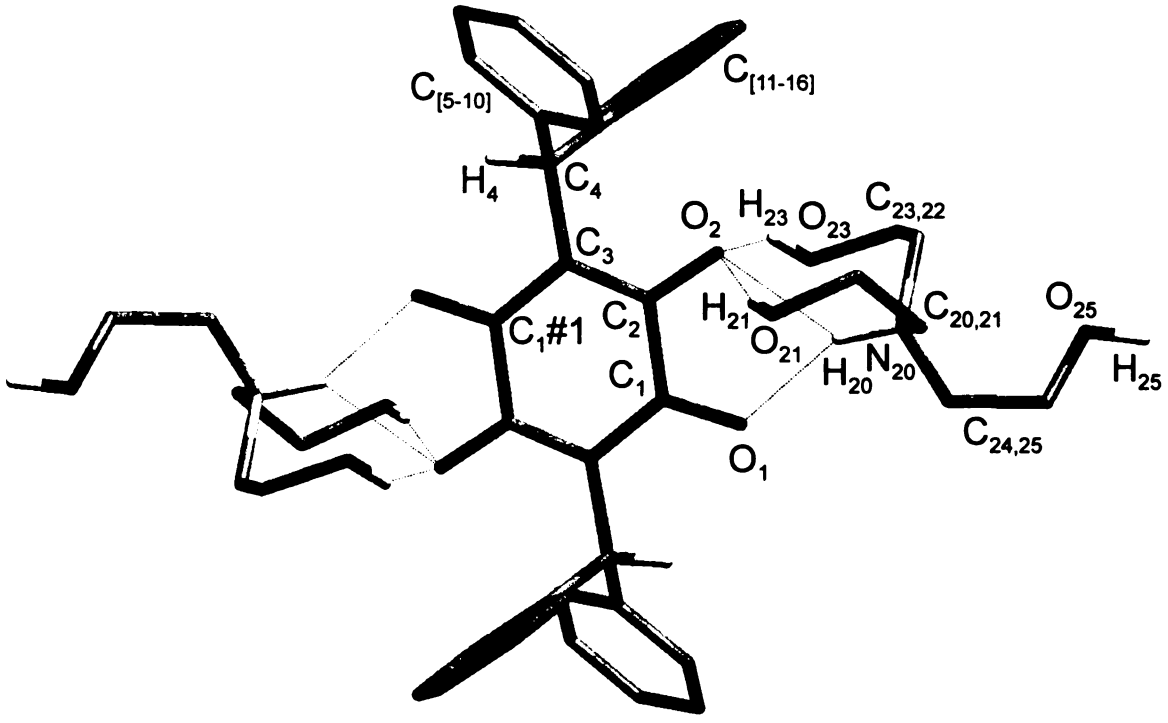


Figure 48. View of **2A•($C_6H_{15}NO_3$)$_2$**. All non-essential hydrogen atoms have been omitted for clarity.

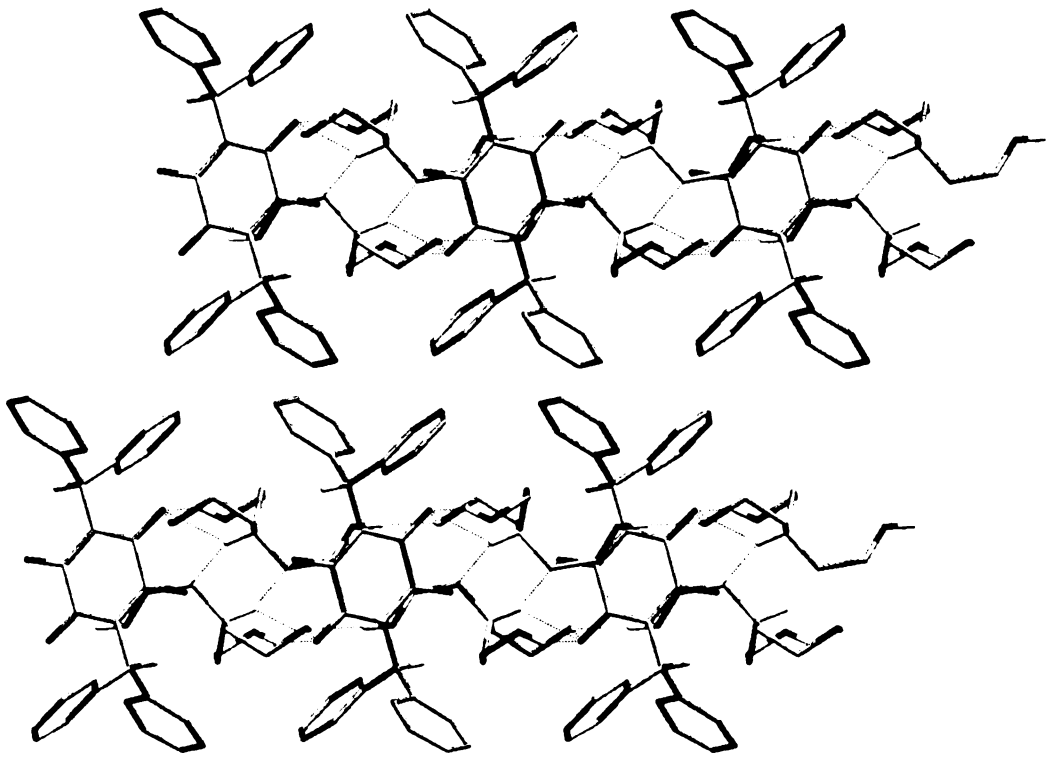


Figure 49. Packing of **2A•$(C_6H_{15}NO_3)_2$**. All non-essential hydrogen atoms have been omitted for clarity.

Table 12. Selected bond lengths for the core of **2A**•$(C_4H_{11}NO_2)_2$ and **2A**•$(C_6H_{15}NO_3)_2$.

	Molecule 2A	
Atoms	$(C_4H_{11}NO_2)_2$	$(C_6H_{15}NO_3)_2$
C_1-O_1	1.2318(19)	1.2342(17)
C_2-O_2	1.299(2)	1.2990(17)
C_1-C_2	1.437(2)	1.4355(19)
C_2-C_3	1.367(2)	1.3704(19)
C_3-C_1#1	1.527(2)	1.5284(18)

Bond lengths are in Å. Atoms marked (#1)-symmetry operator used to generate equivalent atoms.

Table 13. Selected hydrogen bond lengths for **2A**•$(C_4H_{11}NO_2)_2$ and **2A**•$(C_6H_{15}NO_3)_2$.

	Molecule 2A	
Atoms	$(C_4H_{11}NO_2)_2$	$(C_6H_{15}NO_3)_2$
O_1-H_{21C}	1.76(2)	
O_2-H_{23C}	1.76(3)	
O_1-H_{20}		1.966(15)
O_2-H_{21}		1.94(3)
O_2-H_{23}		2.00(2)

Hydrogen bond lengths are in Å.

Once again, in these structures the methyne hydrogen adopts a conformation in which it is nearly coplanar to the core of **2A**. The methyne hydrogen atoms are rotated out-of-plane, above and below the core, and are as

follows; 17.17° in the crystalline complex which incorporated diethanolamine and 18.62° in the crystalline complex which incorporated triethanolamine. In each of the crystal structures both methyne hydrogen atoms have adopted a conformation in which they point in opposite directions. This is not fully understood because free rotation about the C_3-C_4 bond would allow compounds of **2** to crystalline with the methyne hydrogen atoms pointed in opposite, identical or completely random directions. However, one crystal containing **2B** and diethylenetriamine generated a three-dimensional crystalline material in which the methyne hydrogen atoms were pointed in the same direction. That crystal is presented in the next section.

3.5: Three-dimensional Networks

Three-dimensional networks might be expected to form with an organic linker that could coordinate four molecules of **2**. However, a three-dimensional network was produced when compound **2B** crystallized as the dianion from a solution of ethanol and diethylenetriamine to afford the three-dimensional network represented in Figures 51 and 52. The unique molecules of **2B**•$(C_4H_{13}N_3)_2$ are presented in Figure 50. The three-dimensional structure was not expected to form considering both molecules diethylenetriamine and **2B** are bidentate (the central nitrogen in diethylenetriamine would be sterically unavailable if the two terminal amino groups were to be coordinated to **2B**). However, the terminal amino group of more than one molecule of diethylenetriamine coordinates to the two neighboring oxygen atoms on **2B**

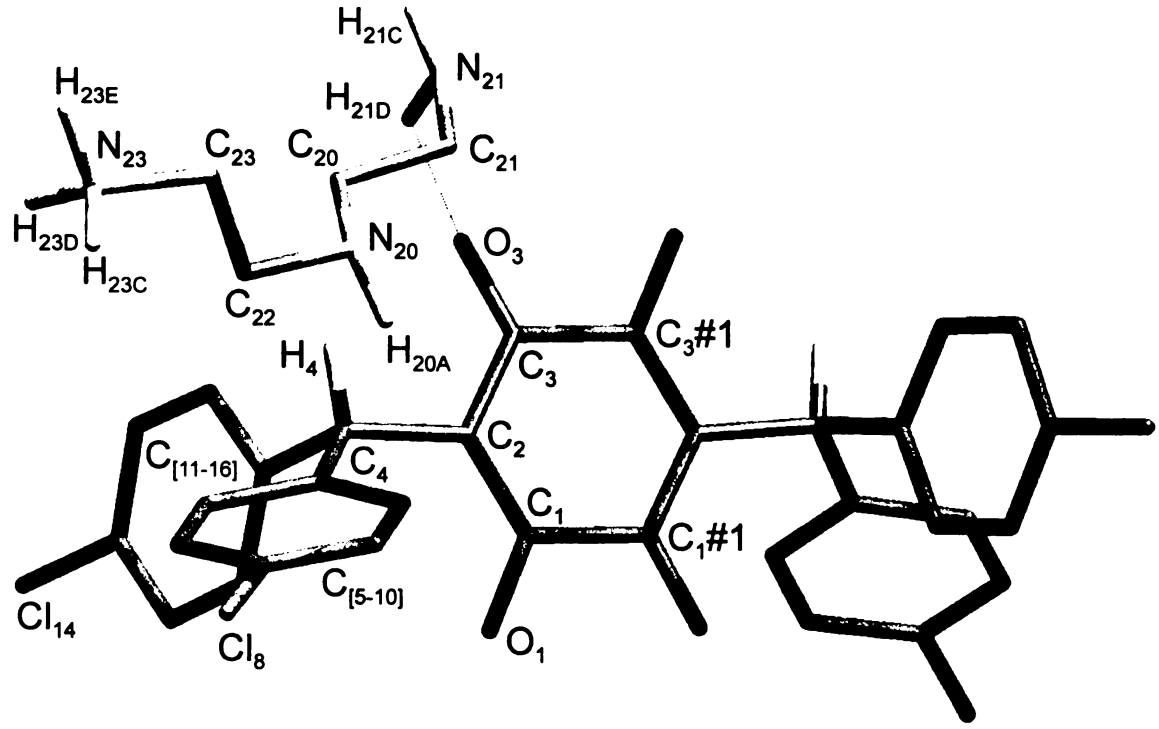


Figure 50. View of **2B•$(C_4H_{13}N_3)_2$**. All non-essential hydrogen atoms have been omitted for clarity and only one molecule of diethylenetriamine has been shown.

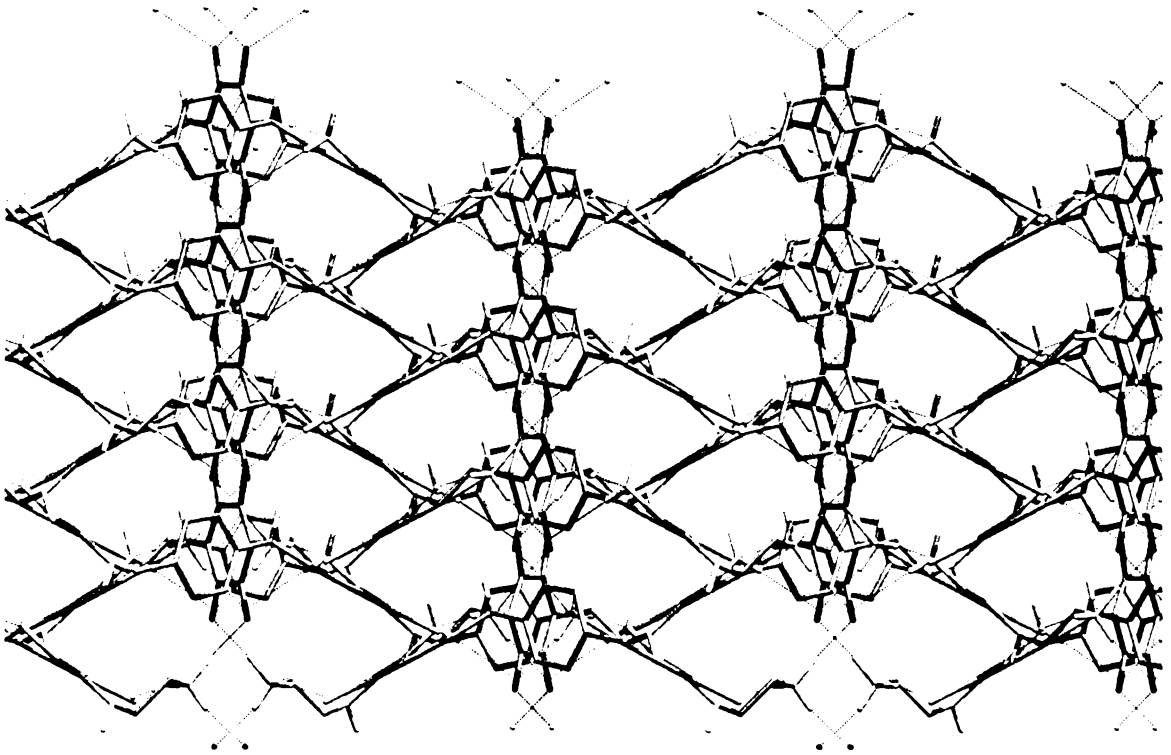


Figure 51. Packing of **2B•$(C_4H_{13}N_3)_2$** down the a-axis. All non-essential hydrogen atoms have been omitted for clarity.

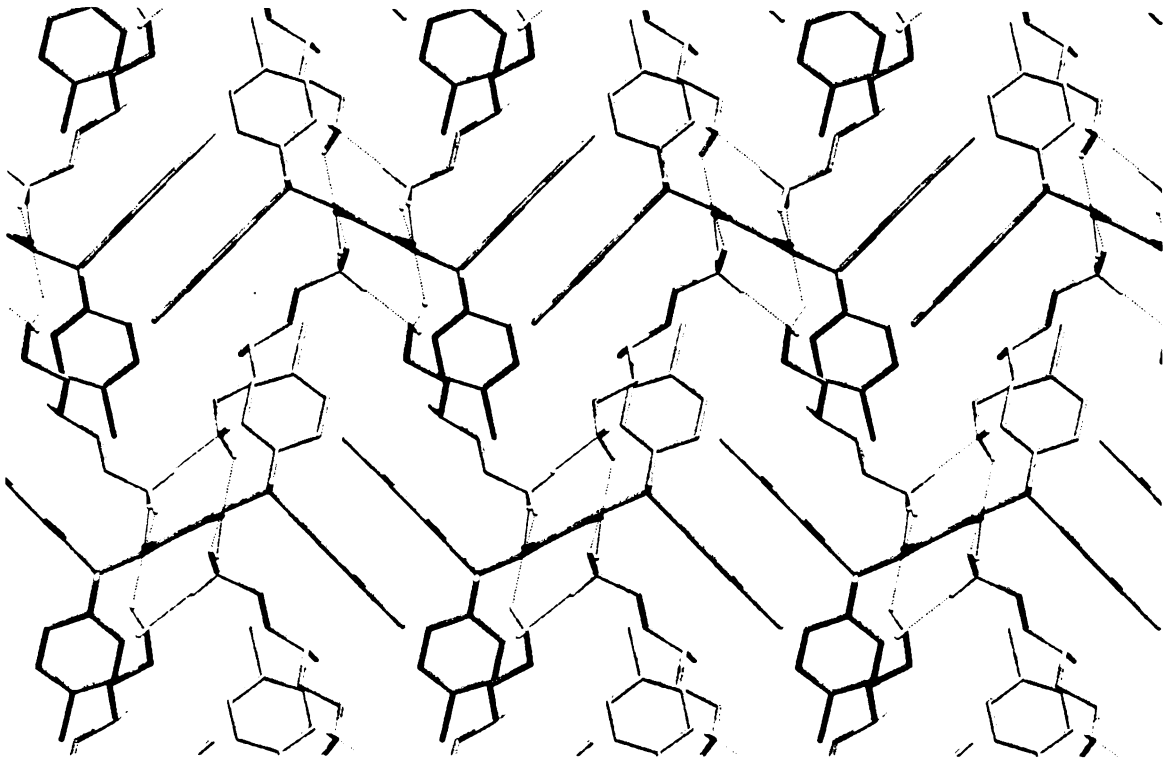


Figure 52. Packing of **2B•$(C_4H_{13}N_3)_2$** down the c-axis. All non-essential hydrogen atoms have been omitted for clarity.

through a series of hydrogen bonds. The crystal structure of **2B•$(C_4H_{13}N_3)_2$** has bond lengths and hydrogen bond distances (1.72(5) Å for the shortest to 2.40(6) Å for the longest) similar to the other dianion crystals in this chapter. Selected bond lengths for **2B•$(C_4H_{13}N_3)_2$** are in Table 14.

Table 14. Selected bond lengths for the core of **2B•$(C_4H_{13}N_3)_2$**.

	2B•$(C_4H_{13}N_3)_2$
Atoms	**Bond length, Å**
C_1-O_1	1.253(4)
C_3-O_3	1.259(4)
C_1-C_2	1.403(5)
C_2-C_3	1.401(5)
C_1-C_1#1	1.559(7)
C_3-C_3#1	1.545(7)

Bond lengths are in Å. Atoms marked (#1)-symmetry operator used to generate equivalent atoms.

Unique among the crystals studied herein, the crystal of **2B•$(C_4H_{13}N_3)_2$** has the methyne hydrogen atoms pointed in the same rather than the opposite direction. It is uncertain if the orientation of the methyne hydrogen atoms has any link to the crystallization of the three-dimensional network in **2B•$(C_4H_{13}N_3)_2$**. A more detailed investigation of this phenomenon would be necessary in order to determine if this is the case.

3.6: Conclusions

Multi-coordinate organic molecules capable of hydrogen bonding were chosen to explore whether the dihydroxyquinone derivatives **2A-E** would self-assemble into multi-dimensional networks. They were a success to a degree; however, they led to networks that were not easily predictable, a desired property of crystal engineering, (except in the case for triethylamine) and failed to form either the desired two-dimensional honeycomb network or the three-dimensional diamond-like network. However, several two-dimensional structures, mainly parallelogram sheets, and one three-dimensional network were observed.

The question "can compounds of **2A-E**, and their structural analogs **1A-E**, produce the desired honeycomb networks?" still remains a mystery. Organic multi-dentate linkers such as tris(2-aminoethyl)amine and triethanolamine were used to determine if there was sufficient space for three molecules of **2** to coordinate around a focal point and generate a two-dimensional honeycomb structure. If this structure was obtainable then there was the possibility the multi-dentate organic linker could be replaced with either a diamagnetic or paramagnetic metal cation. In the crystalline materials that were produced, the two-dimensional layers were compact, avoiding any likelihood that void spaces would be created. Both tris(2-aminoethyl)amine and triethanolamine coordinated to **2A** and adopted a compact conformation removing the void spaces that would be necessary to form the two-dimensional honeycomb structure. To eliminate the flexibility in the molecules, tris(2-aminoethyl)amine and triethanolamine, melamine was used. However, it also failed to crystallize in its intended manner.

Instead it dimerized with another molecule of melamine and created two-dimensional hydrogen bonded sheets separated by the phenyl ring substituents as well.

The first complexes of **2** to crystallize and be subjected to X-ray analysis were those that formed hydrogen bond interactions with triethylamine, a weak organic base. In each crystal it was found the methyne hydrogen atoms adopted conformations that were nearly coplanar to the dihydroxyquinone core and the phenyl rings existed in a propeller-like arrangement. These conformational characteristics suggest the tetrone system might have inherent flaws.

The flaws in the tetrone system do not arise within the framework of the core but in the substituents at the methylene position (the methyne position in the dihydroxyquinone case). It is clear that twisting occurs for both the double bond and the phenyl rings because of steric and/or electrostatic interactions. A consequence of this twisting is the partial deconjugation of the π system, which affects the energy level of orbital **B**, produces the EPR spectra reported in Chapter 2, and ultimately results in weak intramolecular electron-electron interactions (diradical dianion). This finding has fostered ideas for new ligands (see Chapter 5) in which the group located at the methylene position restricts the unpaired electron or electrons to the central semiquinone core.

Another finding that arose from the study of **2** and its hydrogen-bonded complexes was the process by which crystal formation takes place. In the formation of the hydrogen bonded complexes moderately strong dipole-dipole interactions are responsible for crystal growth. At ambient temperatures there is

an association/dissociation mechanism that takes place where molecules of **2** and the solvent continually interact (through hydrogen bonding) and have the ability to rearrange themselves upon coordination with other molecules in the crystal(s) that is/are precipitating. The best crystals (suitable for X-ray analysis) are grown through the slow and uniform deposition of the desired compound(s).

In the crystal of $(1E^{\bullet-})_2 \bullet (Na^+)_2 \bullet (C_4H_{10}O_2)_8$ strong ionic interactions (ignoring the coordinated solvent molecules) lead to the formation of linear chains but all attempts at producing crystals of the diradical dianion have failed. Even the generation of the dianion causes the slow precipitation of the dianion complex from solution (in the case of all other tetrones the monoradical monoanion species can be observed to precipitate from solution). The precipitation of the microcrystalline aggregate can be observed under a microscope or with a low-powered flashlight, suggesting a need for a stronger solvent or solvent pair. So far, no solvent has been found that can hold the diradical dianion in solution long enough for an association/dissociation mechanism to take place (like that found in the hydrogen bond system) and generate crystals suitable for X-ray anaylsis.

Longer aliphatic alkyl chains on the phenyl rings may improve the solubility of the monoradical monoanion and diradical dianion (better interactions with the surrounding solvent) but if such a crystal were generated the distances between the chains or sheets would be even greater, which would undoubtedly lead to lower electron-electron spin interactions, i.e. weaker electron-electron couplings, even than those observed for the sodium crystal of $(1E^{\bullet-})_2 \bullet (Na^+)_2 \bullet (C_4H_{10}O_2)_8$. The present system is unfortunate; increase the

solubility of the organic paramagnetic building block (adding bulkier and larger aliphatic groups to the phenyl rings) and the electron-electron interactions become weaker *or* if the solubility of the paramagnetic organic building block is decreased (by removing the bulky aliphatic *t*-butyl substituents) the electron-electron interactions may increase but crystals suitable for X-ray analysis become more unlikely.

CHAPTER 4

ORGANIC-METAL COMPLEXES AND NETWORKS OF 2

4.1: Introduction

Dihydroxyquinone and its derivatives have been known to crystallize with metal cations of various charges to produce organic-metal salts coordinated into networks of multiple dimensionalities.[11-13,53] Compounds of **2** are similar in structure to dihydroxyquinone and are expected to coordinate in a similar manner. Molecules of **2A-E** were subjected to alkali metal bases of various charges to determine whether they could generate multi-dimensional crystalline materials in hopes they would provide details for the failure of the diradical dianion of **1E** to crystallize into ordered networks or provide insight into how the diradical dianion of **1E** may crystallize. The coordination of compounds **2A-E** to transition metals of various charges would be a significant advancement and allow for the study of metal-to-metal spin-spin interactions. After complexation of the semiquinone species (deprotonated form of dihydroxyquinone) it may be feasible to carry out the chemical transformation of $\mathbf{2}^{2-}$ into $\mathbf{1}^{-\bullet}$ or $\mathbf{1}^{2-\bullet\bullet}$.

In this study, multi-dimensional crystalline networks and an organic-metal complex were formed through the coordination of **2A** or **2D** with alkali metal cations or the mercury(II) cation, they are presented herein. Solutions containing **2B** or **2E** (solutions of **2C** were not tested because it is virtually insoluble in organic solvents) and the previously mentioned cations failed to yield crystals

suitable for X-ray analysis. Transition metal acetoacetonates were also used and found to precipitate rapidly with compounds of **2** and $\mathbf{2^{2-}}$. Analysis of the precipitate by powder X-ray diffraction revealed the precipitate to be polymer-like with coordinated organic and metal cation units. The rapid coordination of **2** or $\mathbf{2^{2-}}$ to the transition metal cation without competition from the acetoacetonate counter ion and an insufficient amount of solvent interactions with the newly formed complex caused the immediate precipitation of the coordinated polymer. The physical properties of these easily created organic-metal polymers were not studied but should be vigorously pursued in future work.

Crystals containing the lithium(I) cation ($\mathbf{2A^{2-}\bullet(Li^{+})_2\bullet(C_4H_8O_2)_3}$), the potassium(I) cation ($\mathbf{2A^{2-}\bullet(K^{+})_2\bullet(C_6H_{12}O_2)_4}$) and the cesium(I) cation ($\mathbf{2D^{2-}\bullet(Cs^{+})_2\bullet(CH_4O)_4}$) were grown in the following manner. A few mgs of **2A** or **2D** were placed into a 5.0 mL scintillation vial. To the vial was added 2 to 3 mL of the following solvents: ethyl acetate for lithium, acetone for potassium and methanol for cesium. A few crystals (or one pellet in the case of potassium) of an appropriate alkali metal hydroxide ($LiOH\bullet H_2O$, KOH) or metal carbonate (Cs_2CO_3) were added, the vial capped and then shaken vigorously. Upon addition of the alkali metal base the solutions appearance changed from yellow to a deep red wine. The solutions remained capped and were allowed to stand for 24 hours.

Upon inspection of the vials red brick-like crystals were found to have precipitated from the solution containing potassium hydroxide and acetone while those of lithium and cesium did not have any noticeable crystal growth. 1.0 mL

of the lithium solution and 2.0 mL of the cesium solution were extracted with separate syringes and the solutions were placed into NMR tubes. The cesium solution was placed in an area free of vibrations and the solvent was allowed to evaporate while *n*-octane was slowly dispensed (through a syringe) into the lithium solution to fill the remaining volume of the NMR tube. This NMR tube was then capped and placed into an environment suitable for crystal growth. After several weeks crystals suitable for X-ray analysis developed in each NMR tube.

Crystals containing the sodium(I) cation ($\mathbf{2D^{2-}\bullet(Na^{+})_2\bullet(C_4H_8O)_4}$) and mercury(II) cation ($\mathbf{2A^{2-}\bullet([HgC_6H_5]^{+})_2\bullet(C_4H_8O)}$) were grown by a procedure similar to the one used for lithium. The source of the sodium(I) cation was sodium borohydride ($NaBH_4$) while that of the mercury(II) cation was phenylmercuric acetate (C_6H_5HgOAc where Ac = acetate). Both of these crystals were grown with a procedure that allows for the slow diffusion of *n*-octane into tetrahydrofuran. Also, in both crystalline materials, which were analyzed by X-ray diffraction, some of the tetrahydrofuran solvent molecules were disordered and therefore only the major contributor is shown.

4.2: Organic-Mercury(II) Complex

Phenylmercuric acetate precipitated with **2A** to generate the zero-dimensional purple crystalline material of $\mathbf{2A^{2-}\bullet([HgC_6H_5]^{+})_2\bullet(C_4H_8O)}$. See Figures 53 and 54. Each mercury(II) cation is chemically bound to a semiquinone coordination site on **2A**, a phenyl ring, which acts like a 'capping' agent and prevents an increase in the overall dimensionality and is coordinated

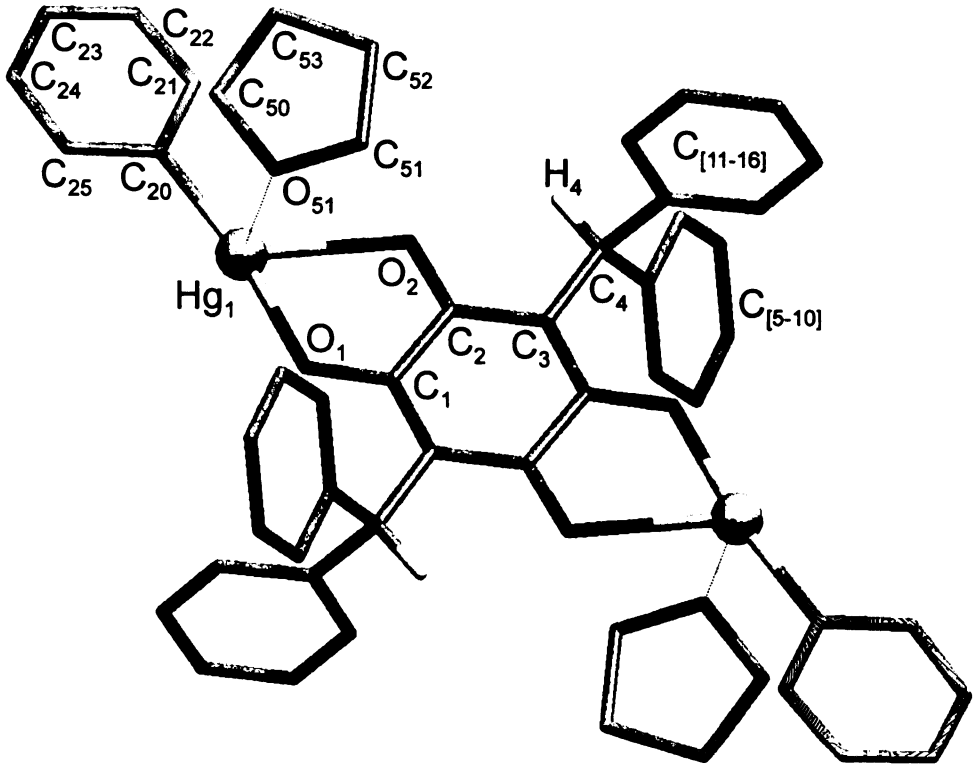


Figure 53. View of $2A^{2-} \bullet ([HgC_6H_5]^+)_2 \bullet (C_4H_8O)$. All non-essential hydrogen atoms have been omitted for clarity.

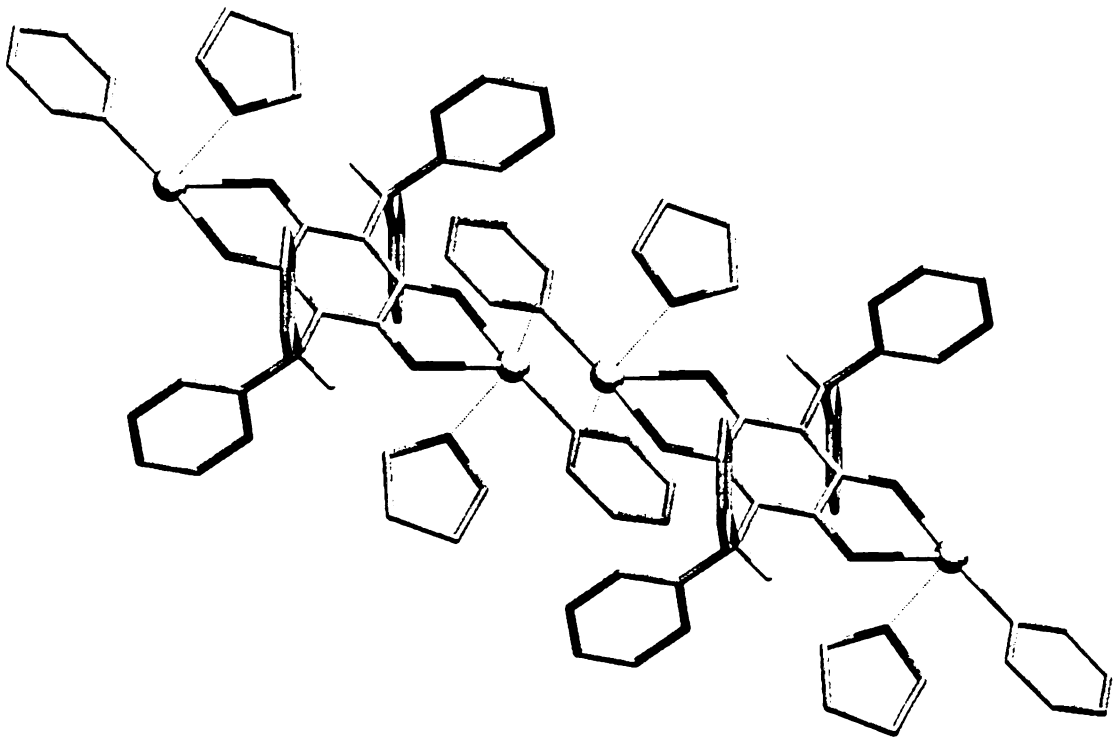


Figure 54. Linkage of $2A^{2-}\bullet([HgC_6H_5]^+)_2\bullet(C_4H_8O)$. All non-essential hydrogen atoms have been omitted for clarity.

to a disordered tetrahydrofuran molecule. The core of the dianion of **2A** shows an uneven distribution of electron density across the periphery of the core. This can be seen in the bond lengths that were determined by X-ray analysis (see Table 15). Examination of the carbon-oxygen bond lengths indicated the C_2-O_2 bond has more double bond character than the C_1-O_1 bond. This is in agreement with the contacts observed between the mercury(II) cation and each of the oxygen atoms. The shorter carbon-oxygen bond length (more double bond character) has the longer (weaker) mercury(II)-oxygen bond distance while the

Table 15. Selected bond lengths for $\mathbf{2A^{2-}\bullet([HgC_6H_5]^+)_2\bullet(C_4H_8O)}$.

Atoms	Bond length, Å
C_1-O_1	1.282(9)
C_2-O_2	1.246(9)
C_1-C_2	1.536(11)
C_2-C_3	1.429(11)
C_3-C_1#1	1.376(11)
C_3-C_4	1.519(11)
Hg_1-O_1	2.091(6)
Hg_1-O_2	2.500(6)
Hg_1-C_{20}	2.035(8)
Hg_1-O_{51}	2.897*
Hg_1-C_{20}	3.689*
Hg_1-C_{21}	3.202*
Hg_1-C_{22}	3.673*

Bond lengths are in Å. Bond distances marked (*) were calculated from close contacts between Hg_1 and the phenyl ring of another molecule.

longer carbon-oxygen bond (more anionic single bond character) has the shorter (stronger) mercury(II)-oxygen bond distance. This also can be seen by the offset of the mercury atom towards O_1 verses O_2. The mercury(II) cation also displays close contacts (2.897 Å) with one disordered tetrahydrofuran molecule and the phenyl ring of another complex directly below it (see Table 15). If these long interactions are counted, the mercury(II) cation would have a coordination number of seven.

The highly disordered tetrahydrofuran was found to be associated with two mercury(II) cations, only one of which is shown in Figure 54. The complex may form a two-dimensional crystalline material if two considerations are made; first, one-dimensional chains are formed via the close contacts of the mercury(II) cation and the phenyl ring of another complex, and two, the chains, which are layered in a crosshatch-like pattern are connected via the tetrahydrofuran molecule, which associates itself between individual linear structures. The rapid association of the oxygen atom in tetrahydrofuran with two mercury(II) cations (one in each linear structure) may be responsible for its disorder. Thermal agitation of the crystalline material may also be a factor in the disorder of the tetrahydrofuran molecules. No improvement was obtained upon repeated crystallizations, which produced similarly disordered crystals.

The crystallization of the mercury(II) cation with **2A** was the first in our group and provided hope that the original building blocks of **1** or its precursors **2** could serve as viable spin-carriers linking multiply charged transition metals. The complex of $\mathbf{2A^{2-}\cdot([HgC_6H_5]^+)_2\cdot(C_4H_8O)}$ may serve as an indirect pathway to

generate the structurally analogous diradical dianion complex. Higher multi-dimensional networks might be obtainable through the replacement of the capped mercury(II) cation via a metal-metal exchange mechanism or through coordination of the phenyl rings' aromatic π electrons with neutral transition metals like those found in the complexes of ferrocene and cobaltocene or a combination of these.

4.3: One-dimensional Networks

Lithium hydroxide monohydrate precipitated with **2A** from the slow diffusion of *n*-octane into ethyl acetate which generated the red crystalline material of $\mathbf{2A^{2-}}\bullet(Li^{+})_2\bullet(C_4H_8O_2)_3$ (see Figures 55 and 56). Two lithium(I) cations, which have different coordination geometries, were found to exist in the crystalline material. The first lithium(I) cation, Li_1, is coordinated to four oxygen atoms from two molecules of $\mathbf{2A^{2-}}$ with nearly identical bond distances (see Table 16) and one solvent molecule of ethyl acetate in a five-coordinate distorted square pyramid. The second lithium(I) cation, Li_2, is coordinated to two oxygen atoms from two molecules of $\mathbf{2A^{2-}}$ (see Table 17) and two oxygen atoms from two molecules of ethyl acetate in a four-coordinate pseudo-tetrahedral arrangement. The two lithium(I) cations have a separation of 2.726(5) Å and cause the linear chain to adopt a zigzag conformation. The linear chains, found in $\mathbf{2A^{2-}}\bullet(Li^{+})_2\bullet(C_4H_8O_2)_3$, were found to be separated by a distance of 3.776 Å (the shortest carbon-carbon contact between phenyl rings) while coordinated ethyl acetate solvent molecules were found to fill the void spaces. The crystal

packing of $2A^{2-} \bullet (Li^{+})_2 \bullet (C_4H_8O_2)_3$ can be seen in Figure 56.

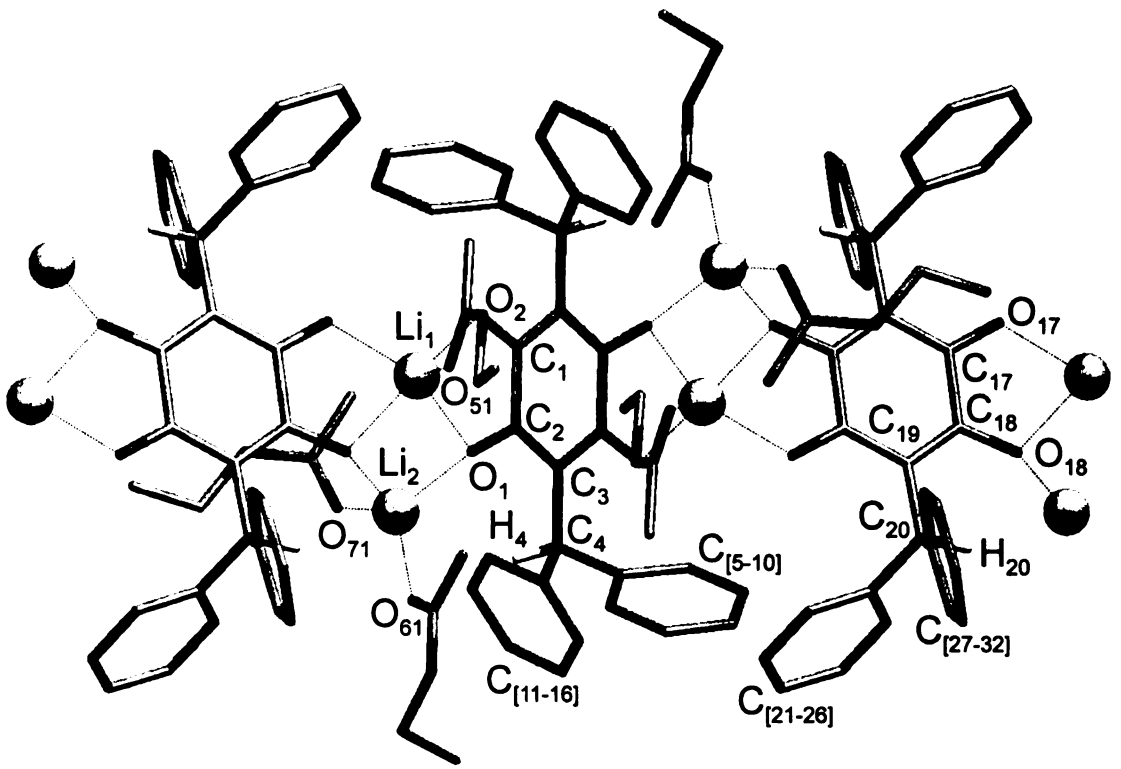


Figure 55. View of $2A^{2-} \bullet (Li^{+})_2 \bullet (C_4H_8O_2)_3$ down the a-axis. All non-essential hydrogen atoms have been omitted for clarity.

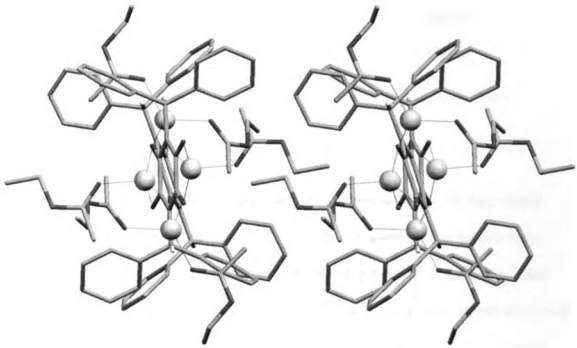


Figure 56. Packing of $2A^{2-}\bullet(Li^{+})_2\bullet(C_4H_8O_2)_3$. All non-essential hydrogen atoms have been omitted for clarity.

Table 16. Selected bond distances for $2A^{2-}\bullet(Li^{+})_2\bullet(C_4H_8O_2)_3$.

	$Li_{\#}^{+}$	
Atom	**1**	**2**
O_1	1.980(3)	
O_2	2.053(4)	1.881(4)
O_{17}	2.019(4)	
O_{18}	2.048(3)	1.903(4)
O_{51}	1.995(4)	
O_{61}		1.953(4)
O_{71}		1.965(4)
Li_1		2.726(5)
Li_2	2.726(5)	

Bond distances are in Å.

Each molecule of $2A^{2-}$ (designated as 1 and 2 in Table 16) has nearly identical carbon-oxygen and carbon-carbon bond distances around the core C_6O_4 fragments, implying full delocalization of the anionic charges (see Table 17). Even distribution of the core electrons produces an overall formal charge of -0.333 on each oxygen atom, which generates nearly identical lithium-oxygen bond distances, ~2.00 Å. However, this is different than in the crystal where the mercury(II) cation was found to be complexed to $2A^{2-}$. The difference must arise in the charge of the cation. The mercury(II) cation prefers an oxygen atom with a greater negative formal charge than the lithium(I) cation. This preference causes the uneven distribution of electrons within the core of $2A^{2-}$.

The shifting of electrons within the core framework, which can be observed between these two crystal structures, provides support for the theoretical calculations (mentioned in the Introduction) that more highly charged

Table 17. Selected bond lengths for $2A^{2-}\bullet(Li^{+})_2\bullet(C_4H_8O_2)_3$.

Atom(s)	$2A^{2-}$ 1	2
C_1-O_1 or C_{17}-O_{17}	1.251(2)	1.247(2)
C_2-O_2 or C_{18}-O_{18}	1.277(2)	1.275(2)
C_1-C_2 or C_{17}-C_{18}	1.545(3)	1.544(3)
C_2-C_3 or C_{18}-C_{19}	1.385(3)	1.387(3)
C_3-C_1#1 or C_{19}-C_{17}#2	1.416(3)	1.416(3)
C_3-C_4 or C_{19}-C_{20}	1.527(3)	1.526(3)

Bond lengths are in Å.

metal cations would bind more strongly to $\mathbf{1}^{-\bullet}$ and $\mathbf{1}^{2-\bullet\bullet}$ (or $\mathbf{2}^{-}$ and $\mathbf{2}^{2-}$) giving rise to stronger coupling interactions between electrons residing on the organic ligand and the electrons on the metal center. This is a promising aspect for this system, but the organic building blocks studied to date allow localization of the electrons on the methyne positions where they remain delocalized into the phenyl rings.

The red crystalline material of $2A^{2-}\bullet(K^{+})_2\bullet(C_6H_{12}O_2)_4$ (see Figures 57 and 58) grew overnight in a capped 5.0 mL scintillation vial containing 3.0 mL of acetone, a few mgs of **2A** and one potassium hydroxide pellet. The square column shaped crystal was then separated from the potassium hydroxide pellet surface with a microscale razor under a microscope and then analyzed by using X-ray diffraction. X-ray analysis determined the crystal to be consisted of linear chains which incorporated the potassium(I) cation and the aldol condensation product of acetone.

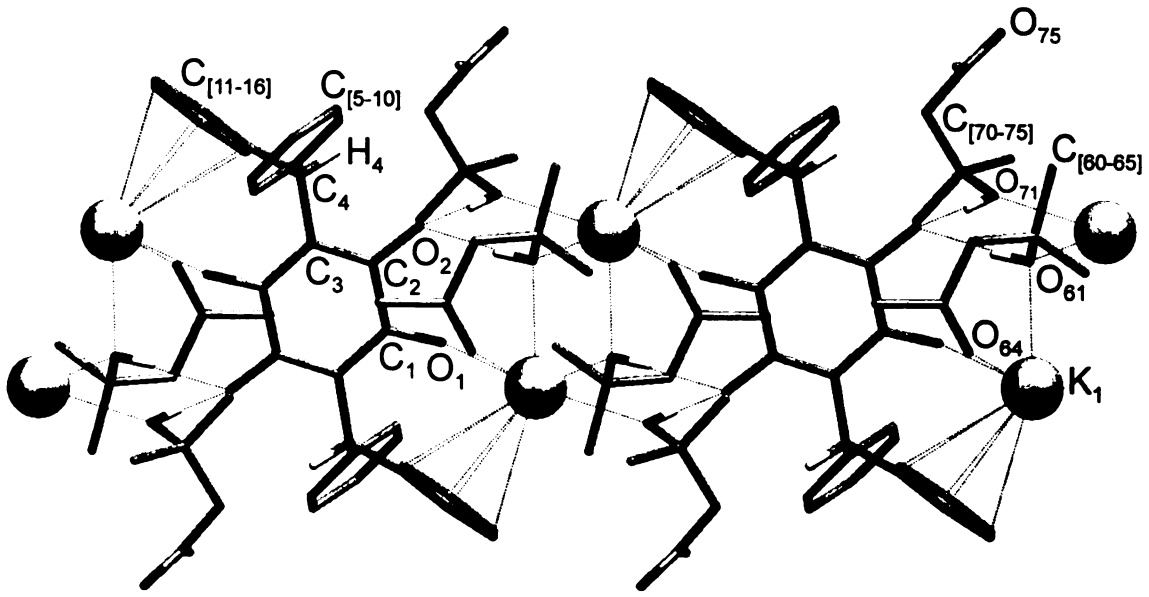


Figure 57. View of $2A^{2-} \bullet (K^+)_2 \bullet (C_6H_{12}O_2)_4$ down the a-axis. All non-essential hydrogen atoms have been omitted for clarity.

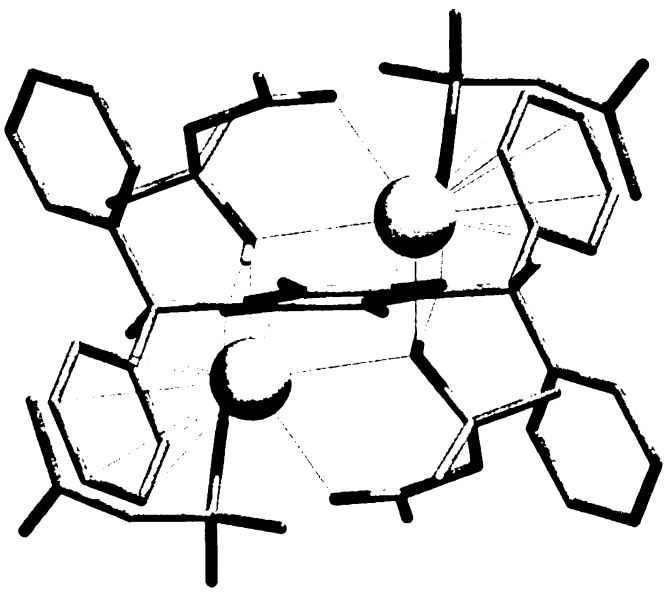


Figure 58. View of $2A^{2-} \bullet (K^+)_2 \bullet (C_6H_{12}O_2)_4$ down the b-axis. All non-essential hydrogen atoms have been omitted for clarity.

The potassium(I) cation was found to be coordinated to five oxygen atoms and the aromatic π system, comprised of carbons $C_{[11-16]}$ (see Table 18). The potassium(I) cation does not adopt a conformation that would place it directly between that of O_1 and O_2 like that found in the lithium crystal of $2A^{2-}\bullet(Li^{+})_2\bullet(C_4H_8O_2)_3$. Instead it is shifted towards O_1 removing itself from O_2 completely. This shift is caused by a couple of factors. The first is the competition between the electron density found within the aromatic π system and the electron density contained on the two oxygen atoms of the core. The second is the repulsive forces that arise with the interaction of one potassium(I) cation with its symmetrical equivalent. The 'push' (repulsive forces) of the cation interactions and the 'pull' (attractive forces) of the aromatic π system cause the potassium(I) cation to adopt the arrangement, shown in Figures 57 and 58, with aromatic π system-potassium(I) cation distances of 3.3 Å to 3.5 Å (typical π-π interactions between aromatic systems is 3.4 Å).

The cation-π interactions exhibited within the crystal are not unusual and have been found to be an important noncovalent binding force[54] in a variety of systems; such as in the gas phase,[55] in aqueous media using synthetic receptors,[56] and in a number of protein systems.[54,57,58] In order to provide a complete and accurate description of the cation-π interaction one would have to include a number of intermolecular forces, such as charge-quadrupole, charge-dipole, charge-induced dipole, charge transfer, dispersion forces, and, in some cases, a hydrophobic component.[54] However, to a first approximation the major component of a cation-π interaction is electrostatic, which involves the interaction

of a cation with a permanent quadrupole moment of an aromatic[59]-this quadrupole interaction has been known to control solid-state architecture[59b,60] and may account for the geometrical arrangements found within the crystals presented in this chapter.

Table 18. Selected bond distances for $2A^{2-}\bullet(K^{+})_2\bullet(C_6H_{12}O_2)_4$.

Atom	K_1^+
O_1	2.6172(13)
O_{61}	2.7578(12)
O_{61}#1	2.8377(14)
O_{64}	2.8087(14)
O_{71}	2.6732(13)
C_{11}#2	3.5020(17)
C_{12}#2	3.3932(19)
C_{13}#2	3.302(2)
C_{14}#2	3.3149(19)
C_{15}#2	3.3951(18)
C_{16}#2	3.4845(18)
K_1^+#1	4.1398(18)

Bond distances are in Å.

A clear indication that the cation-π interaction is mainly electrostatic in nature is the binding energies that have been found between the cation and benzene. The binding energy for the lithium(I)-benzene complex is 38.3 kcal/mole,[55b] for the sodium(I)-benzene complex it is 28.0 kcal/mole,[61] for the potassium(I)-benzene complex it is 19.2 kcal/mole,[55a] and for that of the rubidium(I)-benzene complex it is 15.8 kcal/mole[57] (*ab initio* with 6-31G** basis

set). If polarizability, dispersion forces, or charge-transfer effects were dominant then the rubidium(I) cation would be expected to have the higher binding energy but as expected for an electrostatic model the cation-π interaction increases with decreasing ionic radii. Another interesting finding is the binding energy between the potassium(I) cation and water is 17.9 kcal/mol[55a] (gas phase) and when complexes of the potassium(I) cation were prepared, which incorporated both benzene and water, the water molecules were the first to be removed by collisional activation.[62] This suggests the cation-π interaction would compete with the solvation of a cation in an aqueous medium.

It is now realized that the twisting and rotation of chemical bonds in order to fold a protein into its final three-dimensional structure is a delicate balance between many weak interactions: hydrogen bonding, salt bridges, the hydrophobic effect, and cation-π interactions. It has been clearly demonstrated that when a cationic sidechain (Lys or Arg) is near an aromatic sidechain (Phe, Tyr, or Trp), the geometry is biased toward one that would experience a favorable cation-π interaction.[63] In fact, over one-fourth of all structures in the data bank that incorporate Trp as an aromatic sidechain experience an energetically significant cation-π interaction.[63] The ammonium cation binding energy to benzene is 19.3 kcal/mole[55d] (this is comparable to the potassium(I) cation), the methylammonium cation binding energy to benzene is 18.8 kcal/mol[55d] and the tetramethylammonium cation binding energy to benzene is 9.4 kcal/mole[55e] (the binding energies indicated were determined experimentally in the gas phase).

It was mentioned earlier that the gas phase studies indicate the binding energies to be as follows: $Li^+ > Na^+ > K^+ > Rb^+$, however, in water a dramatic reordering is observed, and the affinity of a cation-π site follows the order $K^+ > Rb^+ >> Na^+ > Li^+$. This reordering may take place in other solvents as well and this may be a reason why the potassium(I) cation is coordinated to the π-system whereas in the crystal that incorporated the lithium(I) cation no observable cation-π interactions can be observed.

The potassium(I) cation also coordinates to four oxygen atoms in three molecules of 4-hydroxy-4-methyl-2-pentanone (aldol condensation product of acetone) and one oxygen atom of $\mathbf{2A^{2-}}$ generating a coordination sphere of eleven. The interesting feature is the selective coordination of the Aldol product versus that of acetone. Three of the four (ignoring O_1 on $\mathbf{2A^{2-}}$) oxygen atoms, which are coordinated to the potassium(I) cation utilize their electron density from the oxygen atom contained within the alcohol functional group instead of the electron density found on the oxygen atoms in the carbonyls. In addition to this, the protons located on these three oxygen atoms are coordinated to O_2 of $\mathbf{2A^{2-}}$ through hydrogen bonding interactions. The hydrogen bonding that takes place at O_2 may be a reason for the shifting of the potassium(I) cation also.

Hydrogen bonding interactions are stronger than dipole-dipole interactions and may be a reason that the aldol product was selectively removed from the acetone solution. Replacement of the aldol product with acetone would remove the hydrogen bonding interactions reducing the overall number and strength of interactions within this particular system. As mentioned before, reducing the

number of interactions (of the desired species) with the surrounding medium causes premature precipitation of the complex; therefore it is best to incorporate as many interactions (with sufficient strength) into the system as possible. The competition between the aldol product and acetone increases the complex's solubility in its surrounding medium allowing ordered crystalline networks to form. Unfortunately this strategy cannot be implemented with the radical anions of **1** because hydrogen atom abstraction would take place by the highly reactive unpaired electron, thus reducing the organic paramagnetic building block to an ordinary diamagnetic building block.

The core of $\mathbf{2A^{2-}}$ displays an uneven distribution of electron density similar to the core which incorporated the mercury(II) cation. However, in the crystal that incorporated the potassium(I) cation the metal ion is preferentially bound to the carbon-oxygen bond (C_1-O_1) that exhibits more double bond character (see Table 19) while the aldol product coordinates, through hydrogen bonding, to the carbon-oxygen bond that exhibits more anionic single bond character (C_2-O_2).

Table 19. Selected bond lengths for $\mathbf{2A^{2-}\bullet(K^{+})_2\bullet(C_6H_{12}O_2)_4}$.

Atoms	$2A^{2-}$
C_1-O_1	1.2451(16)
C_2-O_2	1.2780(16)
C_1-C_2	1.5419(19)
C_2-C_3	1.3904(18)
C_3-C_1#2	1.4192(19)
C_3-C_4	1.5291(19)

Bond lengths are in Å.

This may be explained by the large amount of electron density already donated into the potassium(I) cation via complexation with the aromatic π system of the phenyl ring and the lone-pair of electrons on several sp^3 hybridized oxygen atoms and one sp^2 hybridized oxygen atom. The distribution of electron density within the core is heavily influenced by the metal cation charge and its coordinating position within the crystal structure because both of the crystals incorporating the cations of potassium(I) or mercury(II) have an uneven distribution of electron density while the crystal incorporating the lithium(I) cation is nearly symmetrical.

The red crystalline material $\mathbf{2D^{2-}\bullet(Na^{+})_2\bullet(C_4H_8O)_4}$ was grown through the slow diffusion of a two-fold excess of *n*-octane into a 1.5 mL tetrahydrofuran solution containing a few mgs of **2D** and a few mgs of sodium borohydride ($NaBH_4$) over a period of several weeks. The crystal was analyzed by X-ray diffraction and is presented in Figures 59 and 60. The crystalline material incorporated two unique (unique to the unit cell) molecules of tetrahydrofuran; one coordinated to the sodium(I) cation and one uncoordinated but trapped between the linear chains.

The sodium(I) cations, unlike the potassium(I) cations in their crystals, lie in the same plane as the cores of $\mathbf{2D^{2-}}$ (compare Figure 58 with 60), enabling close contacts between the sodium(I) cation and three of the aromatic π system carbon atoms ($C_{5,9,10}$) with three longer contacts with $C_{6,7,8}$ (see Table 20). This configuration is most likely the result of the smaller atomic radius of sodium(I) cation (116 pm) in comparison to the larger atomic radius of the potassium(I)

cation (152 pm) and weaker sodium(I) cation-π interactions than potassium(I) cation-π interactions. The larger atomic radius coupled with the repulsive forces that exist between the positively charged metal cations forces the potassium(I) cations to be pushed out-of-plane (relative to the plane of the core) further than the sodium(I) cations. However, the potassium(I) cation crystallized with the dianion of **2A** while the sodium(I) cation crystallized with the dianion of **2D** so drawing a comparison between the two crystals may not be feasible.

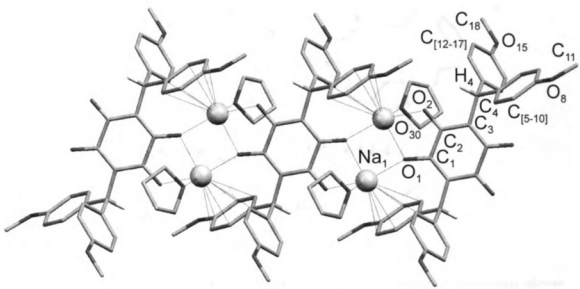


Figure 59. View of $2D^{2-}\cdot(Na^{+})_2\cdot(C_4H_8O)_4$. All non-essential hydrogen atoms and solvent molecules have been omitted for clarity.

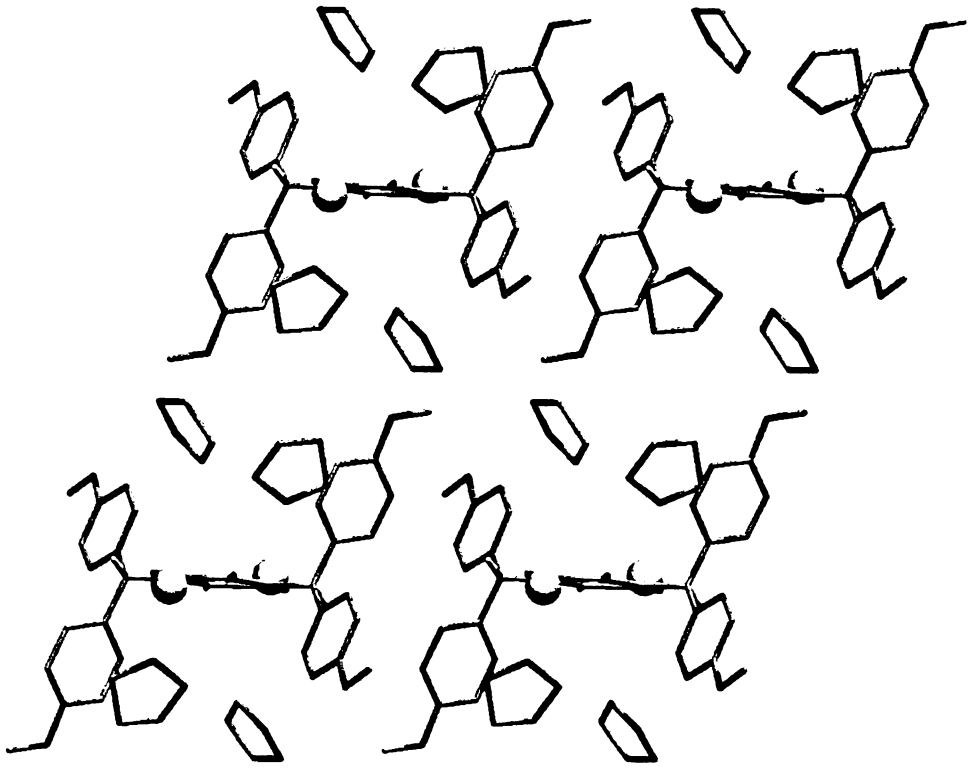


Figure 60. Packing of $2D^{2-} \bullet (Na^{+})_2 \bullet (C_4H_8O)_4$. All non-essential hydrogen atoms and coordination lines have been omitted for clarity.

Table 20. Selected bond distances for $2D^{2-}\bullet(Na^{+})_2\bullet(C_4H_8O)_4$.

Atom	Na_1^+	Na_1^+#1
O_1		2.289(2)
O_1#1	2.289(2)	
O_2	2.252(2)	2.351(2)
O_2#1	2.351(2)	
O_{30}	2.346(3)	
C_5	3.054(3)	
C_6	3.363*	
C_7	3.476*	
C_8	3.299*	
C_9	2.971(3)	
C_{10}	2.822(3)	
Na_1^+		3.189(2)
Na_1^+#1	3.189(2)	

Bond distances are in Å. Bond distances marked (*) were calculated from the close contacts found in the crystal structure.

It is unclear why the sodium(I) cation does not coordinate (equally between the six carbon atoms) with the *p*-methoxy substituted phenyl ring considering that this phenyl ring is more electron rich than the phenyl ring without substitution. One possible explanation is that the *p*-methoxy group is in resonance with the phenyl ring, weakening its Lewis basicity. Evidence of this can be seen in the near 120° bond angles (implying sp^2 hybridization) observed for carbon atoms $C_{8,15}$ and oxygen atoms $O_{8,15}$ (see Table 21) and the shorter carbon-oxygen bond lengths between the phenyl ring and the methoxy oxygen atom (see Table 22).

Table 21. Selected bond angles for $2D^{2-}\bullet(Na^{+})_2\bullet(C_4H_8O)_4$.

Atoms	Bond Angle, °
C_{14}-C_{15}-C_{16}	119.26
C_{14}-C_{15}-O_{15}	115.14
C_{16}-C_{15}-O_{15}	125.61
C_{15}-O_{15}-C_{18}	116.25
C_7-C_8-C_9	118.97
C_7-C_8-O_8	115.35
C_9-C_8-O_8	125.68
C_8-C_8-O_{11}	116.92

Bond angles are in ° and were calculated from the crystal structure. Exact values are found in the Appendix B.

Table 22. Selected bond lengths for $2D^{2-}\bullet(Na^{+})_2\bullet(C_4H_8O)_4$.

Atoms	$2D^{2-}$
C_1-O_1	1.252(3)
C_2-O_2	1.280(3)
C_1-C_2	1.533(4)
C_2-C_3	1.392(4)
C_3-C_1#2	1.424(3)
C_3-C_4	1.523(4)
C_{15}-O_{15}	1.372(4)
C_{18}-O_{15}	1.425(5)
C_8-O_8	1.368(3)
C_{11}-O_8	1.437(4)

Bond lengths are in Å.

Observation of the bond lengths within the core of $\mathbf{2D^{2-}}$ shows an uneven distribution of electron density. The carbon-oxygen bond length of C_2-O_2 is longer than the bond length of C_1-O_1. This indicates that the C_2-O_2 bond has more single bond character or is more anionic than the C_1-O_1 bond. The lengthening of the C_2-O_2 bond can be attributed to the coordination of two sodium(I) metal cations to O_2 whereas there is only one sodium(I) metal cation coordinated to O_1.

4.4: Two-dimensional Networks

The red crystalline material of $\mathbf{2D^{2-}\bullet(Cs^{+})_2\bullet(CH_4O)_4}$ was grown by the slow evaporation of a methanol solution containing 5.0 mg of **2D** and a few mgs of cesium carbonate (Cs_2CO_3) over a period of several weeks. The methanol solution was prepared in a 5.0 mL scintillation vial containing the above compounds. The vial (capped) was left to stand in ambient temperatures for 24 hours. Approximately 2.5 mL of the methanol solution was extracted and placed into an NMR tube. The NMR tube was then lightly capped and placed in a suitable environment for crystal growth. After several weeks the crystals were extracted and then analyzed by X-ray diffraction.

Crystallographic analysis revealed the formation of two-dimensional sheets (see Figures 61 and 62). This structure was not predicted since the other three alkali metal cation complexes produced one-dimensional motifs. The combination of the cesium(I) cation and the *p*-methoxy substituent on the phenyl rings produced sheets versus linear chains.

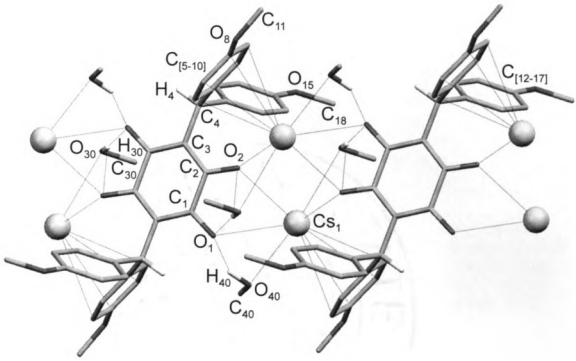


Figure 61. View of $2D^{2-}\bullet(Cs^{+})_2\bullet(CH_4O)_4$. All non-essential hydrogen atoms have been omitted for clarity.

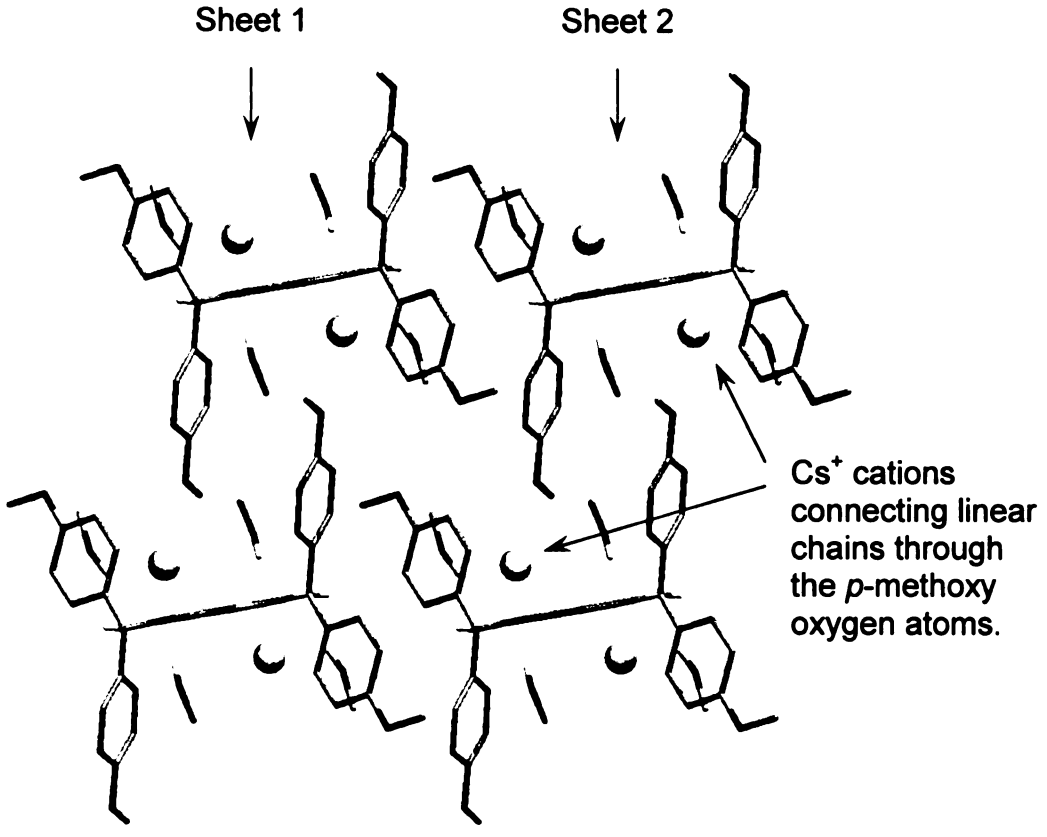


Figure 62. Packing of $2D^{2-}\bullet(Cs^+)_2\bullet(CH_4O)_4$ down the a-axis. All non-essential hydrogen atoms and coordination lines have been omitted for clarity. The linear chains are oriented perpendicular to the plane of the page.

The cesium(I) cations adopt a conformation similar to the potassium(I) cations in which they are located above and below the plane of the $\mathbf{2D^{2-}}$ core. Each cesium(I) cation is complexed to the aromatic π system (see Table 23) with cesium(I)-carbon contacts between 3.560(3) Å and 3.732(2) Å. These values are

Table 23. Selected bond distances for $\mathbf{2D^{2-}\bullet(Cs^{+})_2\bullet(CH_4O)_4}$.

Atom	Cs_1^+
O_1	3.1699(17)
O_2	3.039(2)
O_2#1	3.0268(14)
O_{15}#2	3.2054(16)
O_{30}	3.2092(19)
O_{40}	3.1091(19)
C_5#1	3.715(2)
C_6#1	3.598(2)
C_7#1	3.560(3)
C_8#1	3.607(2)
C_9#1	3.683(2)
C_{10}#1	3.732(2)

Bond distances are in Å.

slightly higher than those found in typical aromatic π bonding (3.4 Å) but are acceptable considering the atomic radius of the cesium(I) cation is 181 pm. The larger atomic radius of the cesium(I) cation (compared to the sodium(I) cation and the potassium(I) cation) allows the cesium(I) cation to coordinate to the oxygen atom, contained within the *p*-methoxy group, of a neighboring chain, linking the linear chains into sheets (see Figure 62). This is not observed in the

crystal containing the sodium(I) cation because the sodium(I) cation is nearly planar with the $\mathbf{2D^{2-}}$ core, unlike that of the cesium(I) cation (compare Figures 60 and 62), and the crystal containing the potassium(I) cation does not have the *p*-methoxy substituent to coordinate to.

The *p*-methoxy substituent adopts a conformation coplanar to the aromatic ring, as in the crystal containing the sodium(I) cation (see Figure 63 and Table 24). This is attributed to the various resonance structures that exist. Although there is a formal charge of positive one for the *p*-methoxy oxygen atom, the remaining lone pair coordinates to the cesium(I) cation, with a distance of 3.205 Å, linking the linear chains together (Figure 62).

Table 24. Selected bond angles for $\mathbf{2D^{2-}\bullet(Cs^{+})_2\bullet(CH_4O)_4}$.

Atoms	Bond Angle, °
C_{14}-C_{15}-C_{16}	119.35
C_{14}-C_{15}-O_{15}	116.27
C_{16}-C_{15}-O_{15}	124.37
C_{15}-O_{15}-C_{18}	116.64
C_7-C_8-C_9	119.71
C_7-C_8-O_8	115.76
C_9-C_8-O_8	124.53
C_8-C_8-O_{11}	116.48

Bond angles are in ° and were calculated from the crystal structure. Exact values are found in the Appendix B.

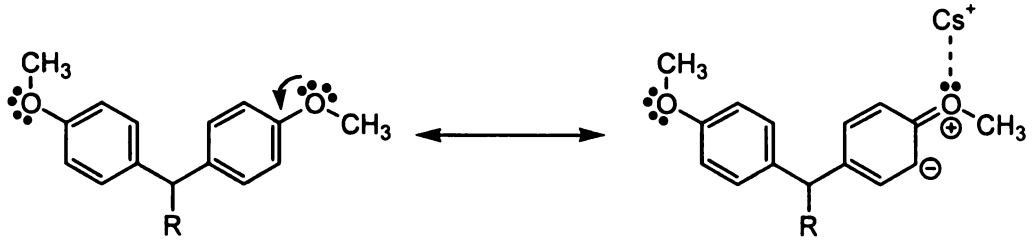


Figure 63. Resonance of the *p*-methoxy substituent and its coordination with the Cs^+ cation. Only one resonance structure has been shown.

The cesium(I) cation is coordinated to both O_1 and O_2 of the **2D**$^{2-}$ core and two methanol molecules while the methanol molecules are hydrogen bonded to both O_1 and O_2. This generates a core in which the electron density is delocalized equally across the periphery of the core, giving rise to nearly identical

Table 25. Selected bond lengths for **2D**$^{2-}$•$(Cs^+)_2$•$(CH_4O)_4$.

Atoms	2D^{2-}
C_1-O_1	1.269(2)
C_2-O_2	1.251(2)
C_1-C_2	1.550(2)
C_2-C_3	1.412(2)
C_3-C_1#3	1.398(2)
C_3-C_4	1.524(2)
C_{15}-O_{15}	1.377(2)
C_{18}-O_{15}	1.426(3)
C_8-O_8	1.373(2)
C_{11}-O_8	1.421(3)
Cs_1-Cs_1	4.306

Bond lengths are in Å.

carbon-carbon bond lengths and carbon-oxygen bond lengths (see Table 25). However, a small difference can be observed and is attributed to the coordination of the two methanol molecules through hydrogen bonding.

4.5: Conclusions

In designing organic building blocks for the purpose of constructing magnetic materials the organic building block should coordinate in a predictable manner. Metal hydroxides of lithium(I), sodium(I) and potassium(I) were used to deprotonate the organic ligands, **2A** or **2D**. Complexation of the metal(I) cation with the appropriate dianion, $\mathbf{2A^{2-}}$ or $\mathbf{2D^{2-}}$, produced X-ray quality crystals of $\mathbf{2A^{2-}\bullet(Li^{+})_2\bullet(C_4H_8O_2)_3}$, $\mathbf{2D^{2-}\bullet(Na^{+})_2\bullet(C_4H_8O)_4}$, and $\mathbf{2A^{2-}\bullet(K^{+})_2\bullet(C_6H_{12}O_2)_4}$ respectively. Each of these crystalline materials demonstrates the ability of the metal(I) cation to coordinate to $\mathbf{2A^{2-}}$ or $\mathbf{2D^{2-}}$ and generate one-dimensional chains. While these cations complexed with $\mathbf{2A^{2-}}$ or $\mathbf{2D^{2-}}$ and generated linear chains the cesium(I) metal cation complexed with $\mathbf{2D^{2-}}$ to generate a two-dimensional sheet-not predicted given the pattern of the first three alkali metals. However, careful manipulation of the substituents on the phenyl rings may lead to other two-dimensional and possibly three-dimensional crystalline materials. Attempts were made to grow crystals of **2D** with each alkali metal cation to draw concrete comparisons between crystal structures but only those crystals, previously presented, were suitable for X-ray analysis.

Although the alkali metal cations crystallized to generate chains there were slight deviations within each of them. This is expected considering that two

of the alkali metals crystallized with $\mathbf{2A^{2-}}$ and two crystallized with $\mathbf{2D^{2-}}$. As the radius of the alkali metal cation increased the general trend was for the alkali metal to increase its coordinating distance to the carbon-oxygen bonds of the core and move out-of-plane from the core adopting a position that was closer to the aromatic π system of the phenyl ring. The location of the alkali metal, relative to the core of $\mathbf{2A^{2-}}$ or $\mathbf{2D^{2-}}$, determined how the electron density was dispersed throughout the periphery of the organic building block. Considerable deviations from typical carbon-oxygen bond lengths can be seen within the core; the same for the carbon-carbon bonds. If this phenomenon exists for the diamagnetic building blocks of **2** then it is reasonable to expect that the same will hold true for the diradical dianions of **1**.

In each of the crystalline materials it was found that hydrogen bonding and cation-π interactions are responsible for directing the architectural outcome; this has been observed in other crystalline materials[59b,60] and in biological systems[54,56-58] as previously mentioned. The diradical dianion of **1E** may be unable to form a well-ordered crystalline solid because of its type of interactions with its environment; typically van der Waals forces.

The crystal structures presented in this chapter also show the conformation adopted by the methyne hydrogen atoms. In each case, the methyne hydrogen atoms orient themselves, through carbon-carbon bond rotation, to a position that is close to co-planar to the core of $\mathbf{2A^{2-}}$ or $\mathbf{2D^{2-}}$ in order to reduce the interactions of the phenyl rings with the core and increase the number of interactions with the alkali metal cations. However, no crystals were

obtained with a metal cation coordinated to $\mathbf{2E^{2-}}$. One reason for this may be the *t*-butyl groups themselves. The *t*-butyl groups were used as substituents on the phenyl rings to increase the solubility of the building block in organic solvents, but in doing so they sterically hinder a strong binding force, cation-π interactions, which can be seen among the other crystal structures presented in this chapter. If this is true for $\mathbf{2E^{2-}}$, then these interactions would not exist for the diradical dianion $\mathbf{1E^{2-\bullet\bullet}}$.

The mercury(II) cation-$\mathbf{2A^{2-}}$ complex was the first look at how a transition metal dication interacted and coordinated to the organic building blocks proposed in this project. It was assumed that any cation of any charge would coordinate to both of the oxygen atoms symmetrically, thus distributing the core's electron density equivalently across the periphery of the core. However, in all structures that incorporated a metal cation there is a preference for the metal cation to bind more strongly to one oxygen atom over another oxygen atom causing the distribution of the electron density to be shifted. The diradical dianions of $\mathbf{1^{2-\bullet\bullet}}$ are expected to be structurally equivalent to their precursors $\mathbf{2A^{2-}}$ and therefore to exhibit similar modes of coordination. This unsymmetrical mode of coordination must be taken into account and new theoretical calculations (previous theoretical calculations[10] had the metal cation bound symmetrically to the two oxygen atoms) must be carried out in order to observe what effects, if any, there are.

Aromatic π interactions with metal cations appear to be a common theme among the crystals present in this chapter and even exist within the molecular

complex of $2A^{2-}\bullet([HgC_6H_5]^+)_2\bullet(C_4H_8O)$. However, instead of the phenyl rings of the organic building block coordinating to the mercury(II) cation the phenyl ring directly bound to the mercury(II) cation, which is the most electron rich of the two, interacts with the mercury(II) cation of another molecular complex. This interaction extends the dimensionality of the complex from zero to one. If the organic unit of $2A^{2-}$ were replaced with $1A^{2-\bullet\bullet}$ it would be interesting to study the intramolecular electron-electron interactions and see if there exists intermolecular electron-electron communication from one organic unit to another via the π bridge linking the mercury(II) metal centers.

CHAPTER 5

CONCLUSIONS

The organic compound **1**, and its derivatives, were exclusively designed to carry multiple unpaired electrons and be utilized in the self-assembly of multi-dimensional molecular magnetic materials exhibiting ferromagnetic and ferrimagnetic behavior. The organic tetrone unit met some of the goals that were set forth prior to the beginning of this research. However, it failed to meet many others; i.e. $\mathbf{1}^{2-\bullet\bullet}$ having a preference for a triplet ground state. Despite the failure of the diradical dianions of $\mathbf{1}^{2-\bullet\bullet}$ to possess ground state triplets, structural modifications can be made to **1** which may increase the probability that the newly designed building blocks will have the hoped-for high-spin ground state. This will be discussed later in this chapter.

Compound **1**, and its substituted derivatives, was proven to be synthetically accessible, in relatively high purity but with modest yields, and found to be stable under dry conditions over an extended period of time, years. However, **1E** was found to decompose if left dissolved in organic solvents and exposed to the atmosphere. Each tetrone was found to be easily reduced to the monoradical monoanion, $\mathbf{1}^{-\bullet}$, and diradical dianion, $\mathbf{1}^{2-\bullet\bullet}$, upon contact with an alkali metal mirror. Spectroscopic solution studies of the anions and dianions supported the near degeneracy of the frontier orbitals, **A** and **B**, as well as the tunability of their gap by methylene site substitution; this was predicted by

theoretical calculations conducted prior to this research. Spectroscopic studies also showed the molecular framework, of the proposed organic building blocks, to be stable while undergoing oxidation-reduction reactions. Despite these successful accomplishments, the proposed diradical organic spin carriers were found to behave as biradicals in which each electron localized itself on a methylene site, due to phenyl ring twisting, which produced near zero intramolecular spin-spin coupling, according to the results obtained from EPR spectroscopy. This was unfortunate and design changes to the substituents on the methylene positions should prevent this from occurring in future work.

Crystallographic analysis using X-ray diffraction techniques coupled with SQUID magnetometry have shown that the monoradical monoanion of $\mathbf{1E^{-\bullet}}$ self-assembled with the potassium(I) and sodium(I) cations, to form $\mathbf{(1E^{-\bullet})_2 \bullet (K^+)_2 \bullet (C_4H_8O)_4}$ and $\mathbf{(1E^{-\bullet})_2 \bullet (Na^+)_2 \bullet (C_4H_{10}O_2)_8}$, respectively, which resulted in molecular chains. The linear chains were found to have magnetic properties which resemble those of a simple paramagnet with weak intermolecular spin-spin couplings of $2J$ = -0.2 K and ~0 K, respectively. The strength of the spin-spin coupling varies based on cation size and provides further proof that the properties of the molecular material can be 'fine-tuned' based on substituents or metal cations or both. Replacement of the alkali metal cations, sodium(I) and potassium(I), with more highly charged diamagnetic and paramagnetic transition metal or lanthanide metal cations should provide stronger electron-electron coupling and materials with higher dimensionalities.

As mentioned previously, structural modifications of the current tetrone

system are required in order to prevent the isolation of the two unpaired electrons on the methylene termini; this may give rise to stronger intramolecular electron-electron spin interactions and intermolecular spin interactions with the coordinating metal cation. Examples of those structures are presented below in Figure 64. The adamantylidene substituted compound offers both steric

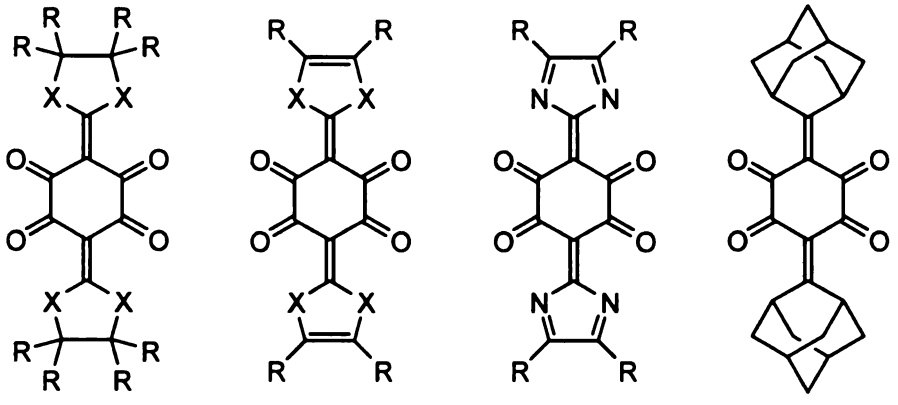


Figure 64. New organic building blocks that may offer better intramolecular and intermolecular spin-spin interactions.

protection and spin localization for the radical sites while the others provide a direct linkage of the methylene site to an electron-donating group thereby allowing the organic unit to maintain its tunability. However, these compounds may face the same danger inherent in the current structures. Before reduction takes place within the new compounds they are conformationally locked into position through the carbon-carbon double bond that exists between the core and

the methylene site. After reduction occurs twisting about the carbon-carbon may take place, which has already been shown to occur in when phenyl rings are the substituents. To date these compounds have not yet been synthesized or studied with theoretical calculations. If theoretical studies reveal these compounds to possess a ground state singlet then their syntheses would become moot.

Failure of other alkali metal cations to crystallize with $\mathbf{1E}^{-\bullet}$ and $\mathbf{1E}^{2-\bullet\bullet}$ led to the study of the tetrones' precursor and its dianion **2** and $\mathbf{2}^{2-}$, respectively. Compounds of **2** were subjected to weak cyclic and acyclic organic bases to determine whether **2** (structurally analogous to **1**) could form crystalline materials that exhibited higher dimensionalities. The weak organic bases crystallized with **2A-E** and generated zero-, one-, two- and three-dimensional organic complexes and lattices. In each case the methyne hydrogen atom adopted a conformation planar to the core but opposite in direction to each other, except in one case. It is not understood why the two methyne hydrogen atoms in the crystal structure of $\mathbf{2B}\bullet(C_4H_{13}N_3)_2$ adopted this conformation, however it was the only crystalline material in which a three-dimensional network was generated. Further crystallographic studies would be necessary in order to determine if the cis-oid conformation is energy-based or if there is another driving force for this arrangement.

In all of the two-dimensional networks produced from the interaction of **2** with a weak organic base the desired two-dimensional honeycomb sheets were not generated; instead more compact arrangements arose, apparently driven by

the need to eliminate void spaces. The acyclic tridentate organic linkers, triethanolamine and tris(2-aminoethyl)amine, adopted closed compact conformations instead of an open "Y"-like conformation. These two organic bases were used to determine if they would coordinate three molecules of **2** in a propeller-like arrangement and generate two-dimensional honeycomb networks. The more rigid base, melamine, was tried to avoid complications due to cation flexibility. Even with the use of melamine honeycomb structures failed to form; this may be a result of the large organic building blocks being used (not enough available space) or the two-dimensional honeycomb structure is entropically or thermodynamically unfavorable. Further studies would need to be done in order to draw a concrete conclusion.

Compounds of **2** were also subjected to various metal containing bases to determine if the dianions were capable of generating multi-dimensional crystalline networks. Many interesting findings came from this study. In each of the crystalline materials the phenyl rings coordinate to the metal cation except for the crystal containing the well solvated lithium(I) cation. Cation-π interactions are not uncommon and are found to be a strong driving force for crystallization in other systems, including protein folding.[62] These interactions between the aromatic π system and the metal cation may provide the needed pathway for these large organic units to form extended networks. Obtaining crystals containing the organic species **2B** and **2E** was unsuccessful and is likely due to the bulky *t*-butyl groups and the chlorine atoms, which sterically protect the phenyl rings' aromatic π system from coordinating to the metal cation. However,

the *p*-methoxy group did not hinder aromatic π complexation with the sodium(I) cation or with the cesium(I) cation. In the latter case the *p*-methoxy substituent coordinated to the cesium(I) cation of another chain linking the chains into two-dimensional sheets. The *p*-methoxy group may not impede π complexation with the metal cation because it increases the electron density of the phenyl ring and is coplanar to the core as a result of the various resonance structures that can be drawn. Aromatic π complexation also took place in the crystal containing the mercury(II) cation. The phenyl rings directly attached to the mercury(II) cation partially overlap with the mercury(II) cation of another complex creating chains.

In each of the organic-metal coordinated crystals the geometric location of the metal cation relative to the oxygen atoms of the core plays an integral role in the distribution of electron density within the core. This may have an impact on spin-spin coupling from the organic unit to the metal cation. Recent calculations show a lowering of symmetry occurs when the coordinating cation is lithium(I) or trialkyltin(I), Li^+ or R_3Sn^+, respectively.

The building blocks of **2** were subjected to other various metal containing bases but were not included within this work. Several divalent and trivalent transition metal cations, which are complexed with acetoacetonate, show promising results for generating coordinated organic-metal hybrid materials and should follow this work. The neutral compounds of **2** and their dianions $\mathbf{2A^{2-}}$ react almost instantaneously with transition metal acetoacetonate complexes. The material generated is a fine film which is unsuitable for X-ray analysis, however, these materials could be analyzed by EPR spectroscopy, SQUID

magnetometry and other spectroscopic methods. This may enhance our understanding of magnetic behavior.

After many failed attempts at producing single crystals the materials were analyzed by X-ray powder diffraction techniques. The spectra that were observed displayed no sharp peaks but instead displayed two very broad humps, which is indicative of a coordinated polymer with little likelihood of crystallizing into ordered materials. A possible explanation for the generation of the amorphous material instead of ordered crystalline materials may be as follows: When the building block of $\mathbf{1E}^{2-\bullet\bullet}$ or $\mathbf{2A}^{2-}$ complexes with a transition metal center there is a lack of an association/dissociation type mechanism that is needed to produce metal centers with ordered chiralities. For planar honeycomb networks, the chirality of each metal center *must* alternate back and forth from lambda-delta-lambda (or visa versa) and so on (other networks may be generated from other ordered patterns that can be imagined).[22e] Coordinated polymeric materials in which the metal centers have random chiralities are highly unlikely to self-assemble into ordered multi-dimensional structural motifs. However, molecular complexes with interesting properties may be produced if stoichiometric amounts of the paramagnetic diradical dianion $\mathbf{1}^{2-\bullet\bullet}$ or the diamagnetic dianion $\mathbf{2A}^{2-}$ are complexed with the metal acetoacetonates, where the acetoacetonates act as capping ligands. This would allow for EPR and magnetic susceptibility studies to be carried out provided that one of the species, organic unit or metal center, is a spin-bearing entity.

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UNDERSTANDING THE NATURE OF 3,6-BIS(BIS(3,5-DI-TERT-BUTYL-4-METHOXYPHENYL)METHYLENE)-1,2,4,5-TETRAOXOCYCLOHEXANE AND ITS RADICAL ANION AND DIANION

VOLUME II

By

Robert Alan Gentner

A DISSERTATION

Submitted to
Michigan State University
in partial fulfillment of the requirements
for the degree of

DOCTOR OF PHILOSOPHY

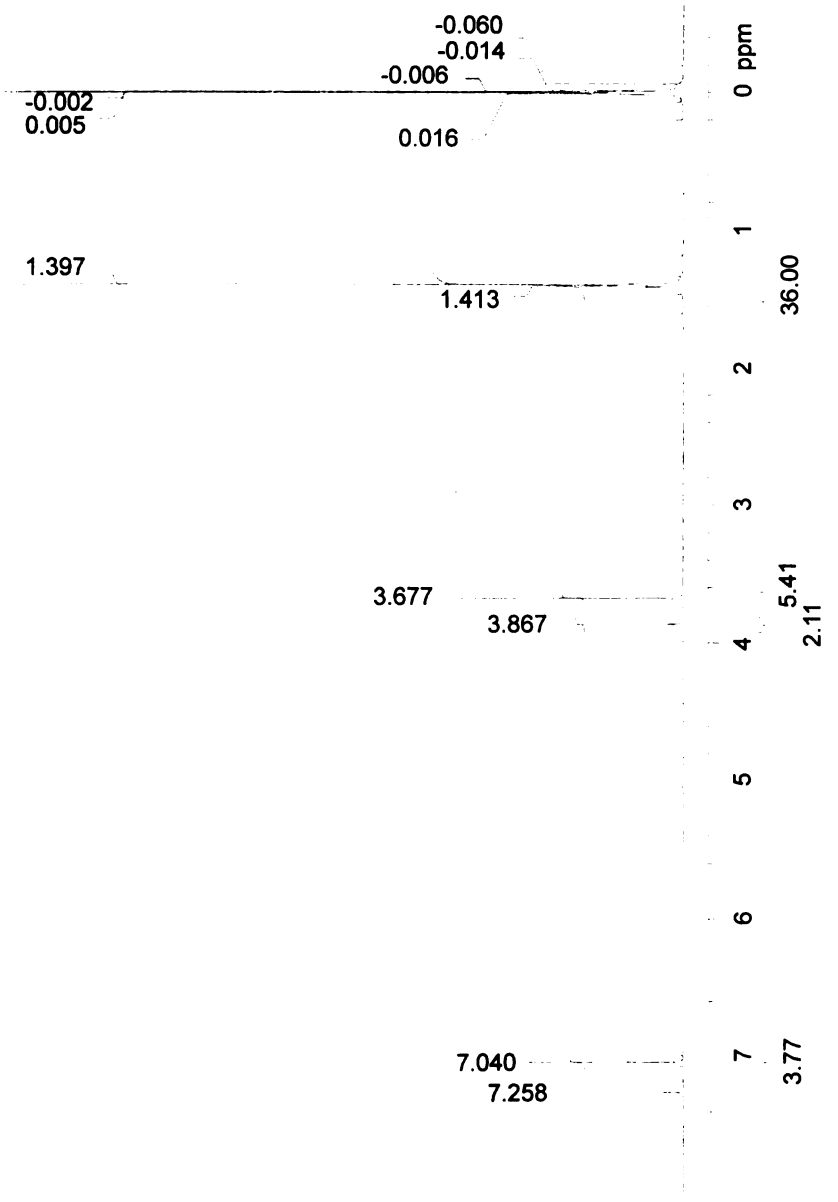
Department of Chemistry

2005

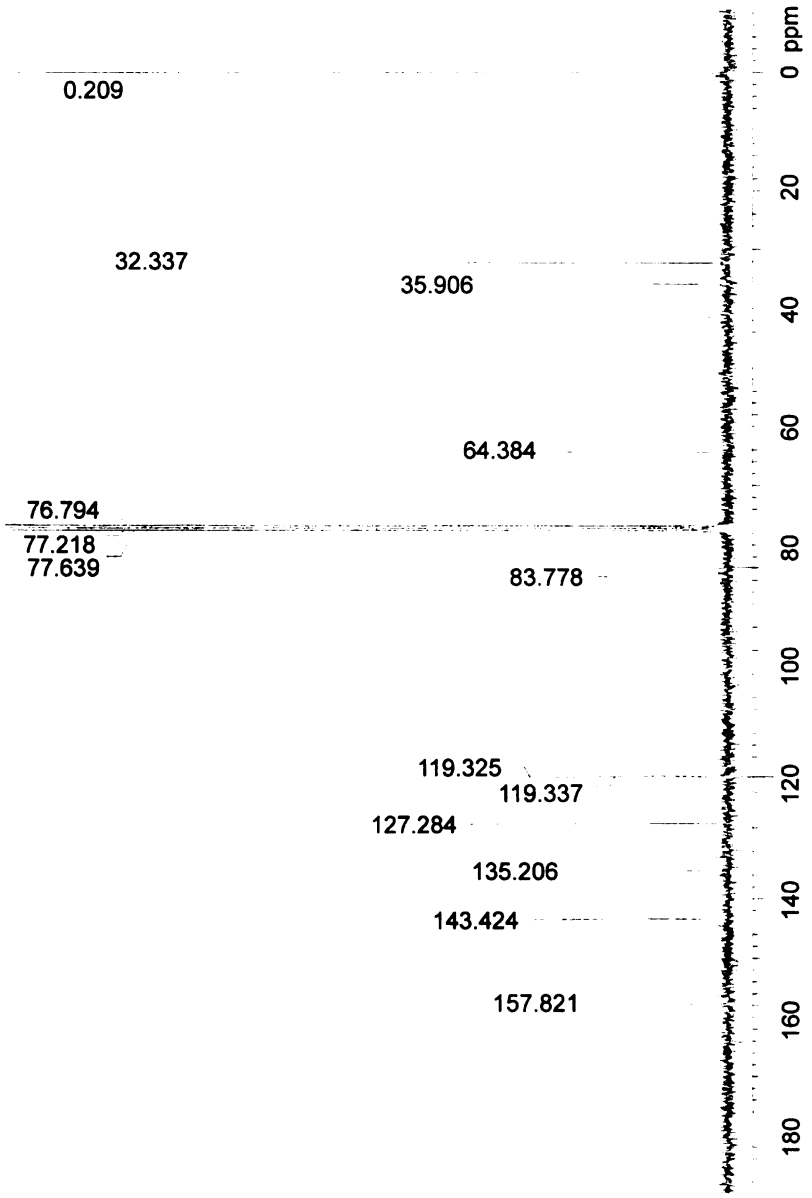
APPENDICES

APPENDIX A

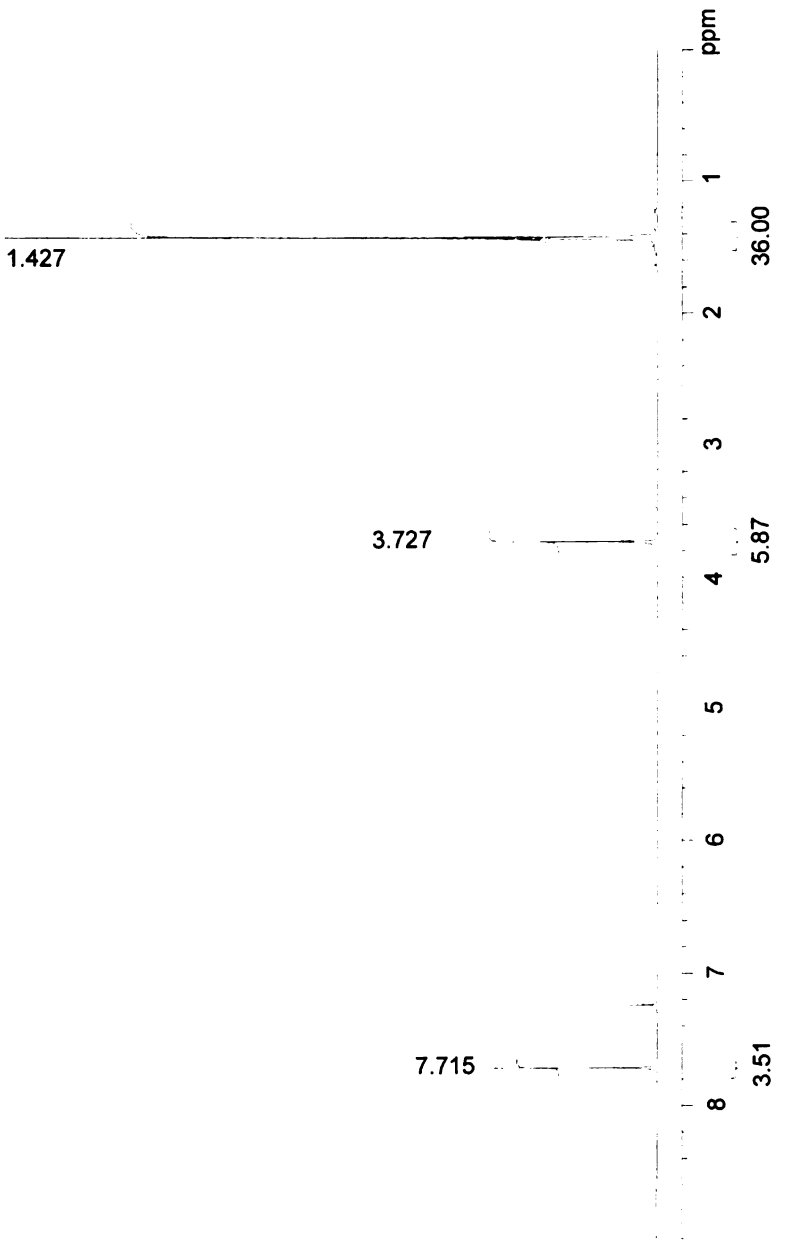
^{1}H and ^{13}C NMR



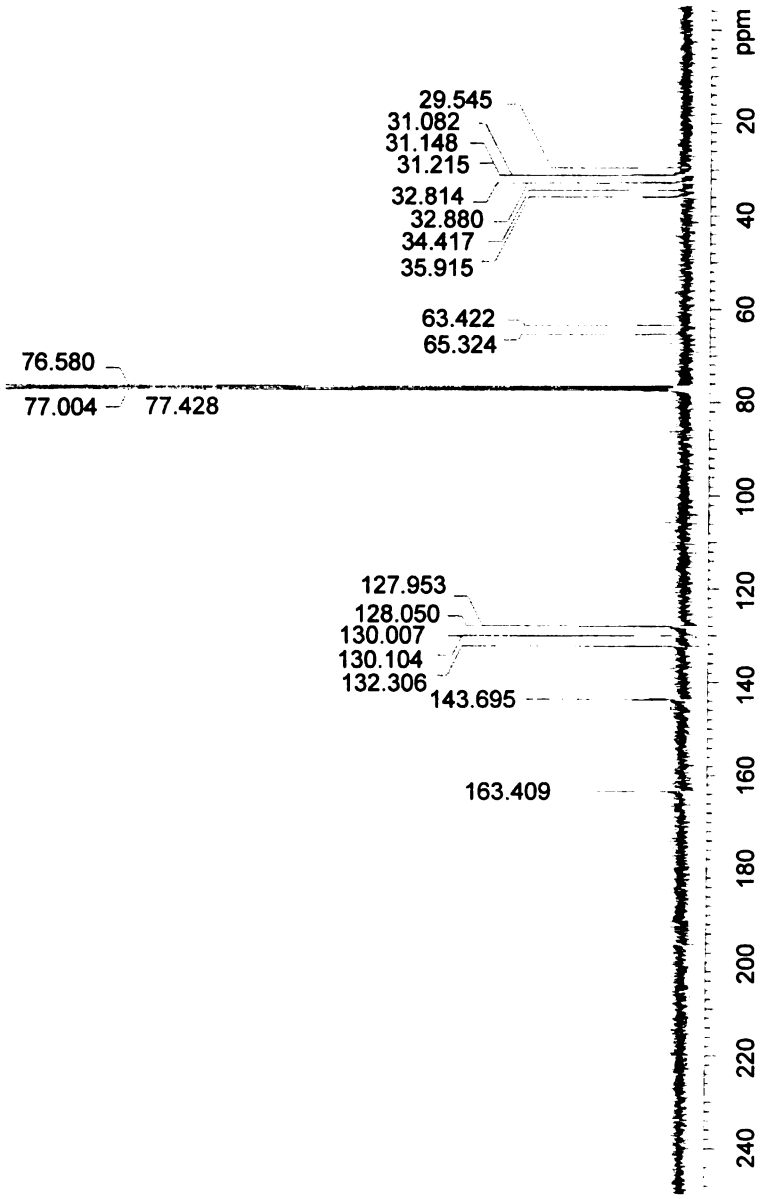
A1. ^{1}H NMR (300 MHz) of **4** (*3,5,3',5'*-tetra-*tert*-butyl-*4,4'*-dimethoxydiphenylmethane) in $CDCl_3$ (δ 7.24) with TMS at 20°C.



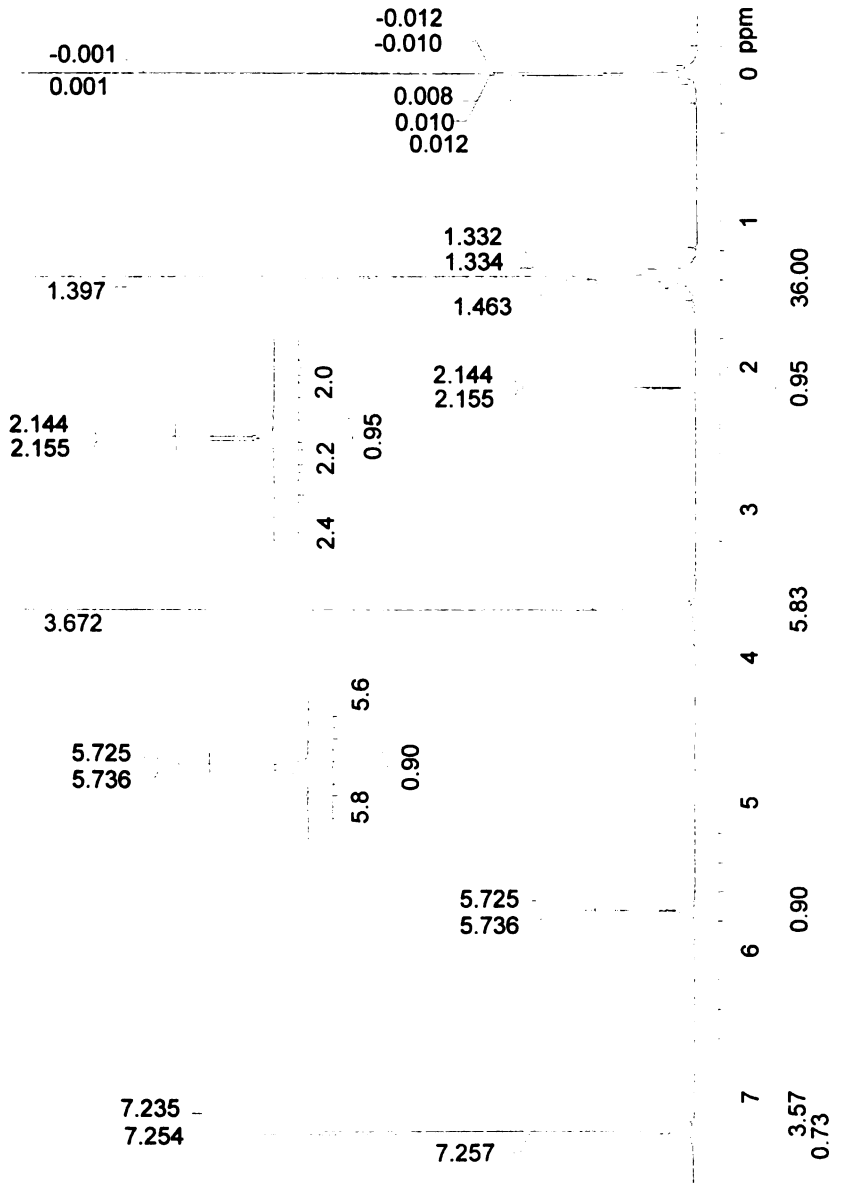
A2. ^{13}C NMR (75.5 MHz) of **4** (*3,5,3',5'*-tetra-*tert*-butyl-*4,4'*-dimethoxydiphenylmethane) in $CDCl_3$ (δ 77.0) with TMS at 20°C.



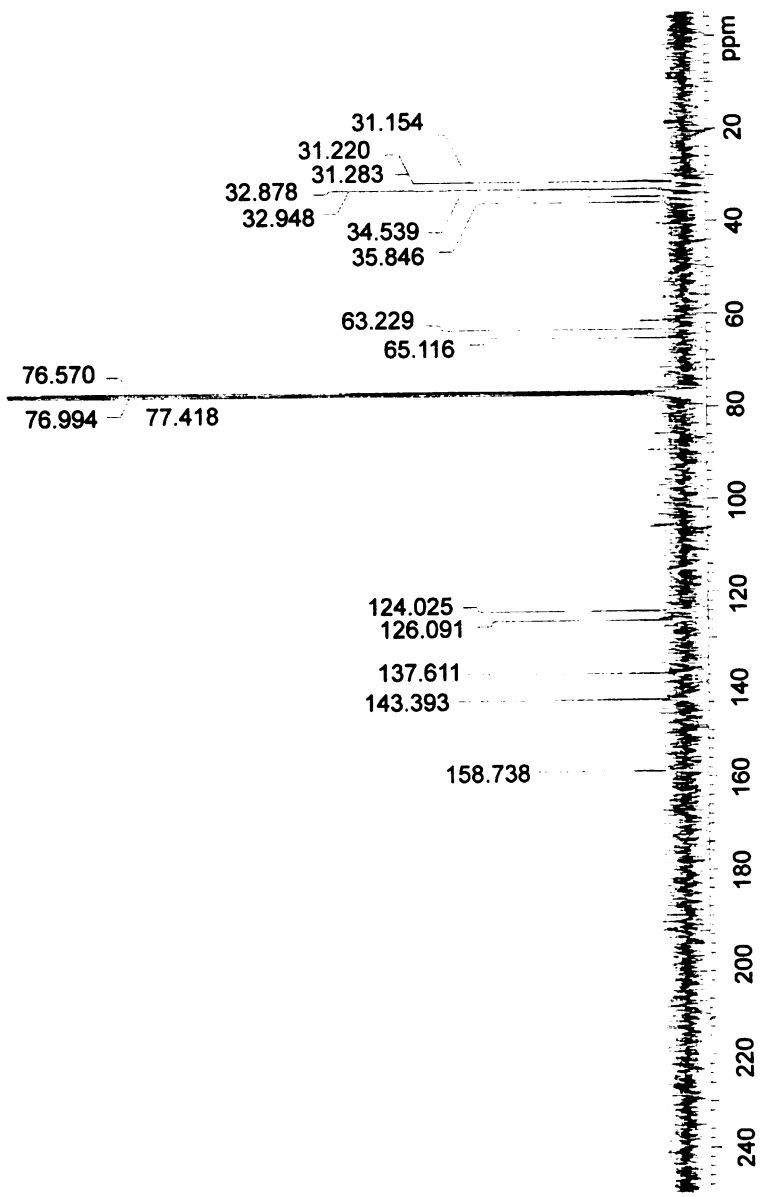
A3. ^{1}H NMR (300 MHz) of **5** (*3,5,3',5'*-tetra-*tert*-butyl-*4,4'*-dimethoxydiphenylmethanone) in $CDCl_3$ (δ 7.24) at 20°C.



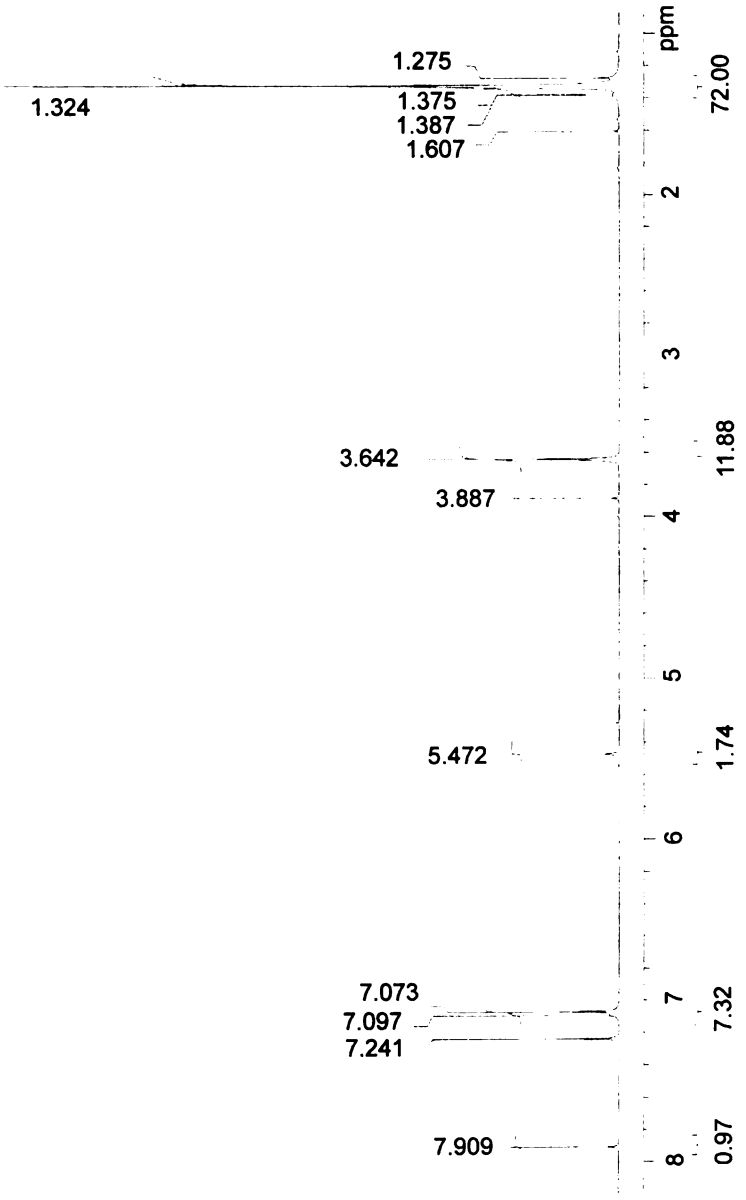
A4. ^{13}C NMR (75.5 MHz) of **5** (*3,5,3',5'*-tetra-*tert*-butyl-*4,4'*-dimethoxydiphenylmethanone) in $CDCl_3$ (δ 77.0) at 20°C.



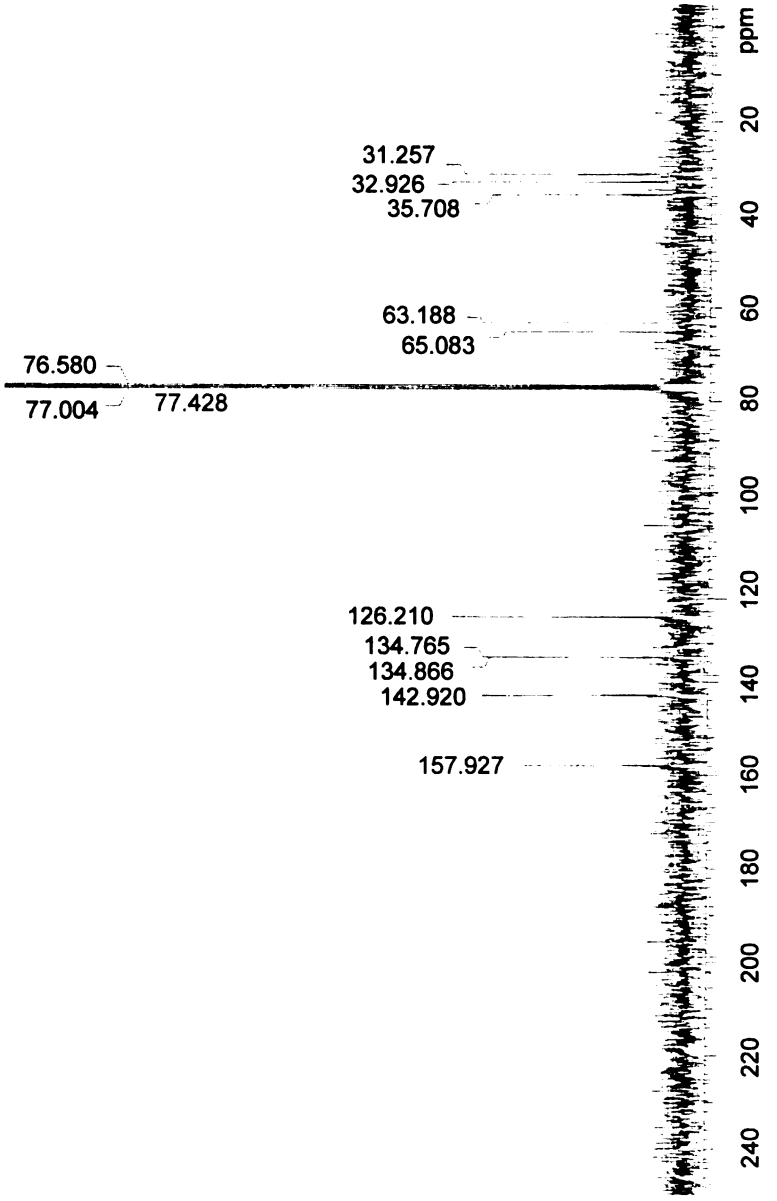
A5. ^{1}H NMR (300 MHz) of **6** (*3,5,3',5'*-tetra-*tert*-butyl-*4,4'*-dimethoxydiphenylmethanol) in $CDCl_3$ (δ 7.24) with TMS at 20°C.



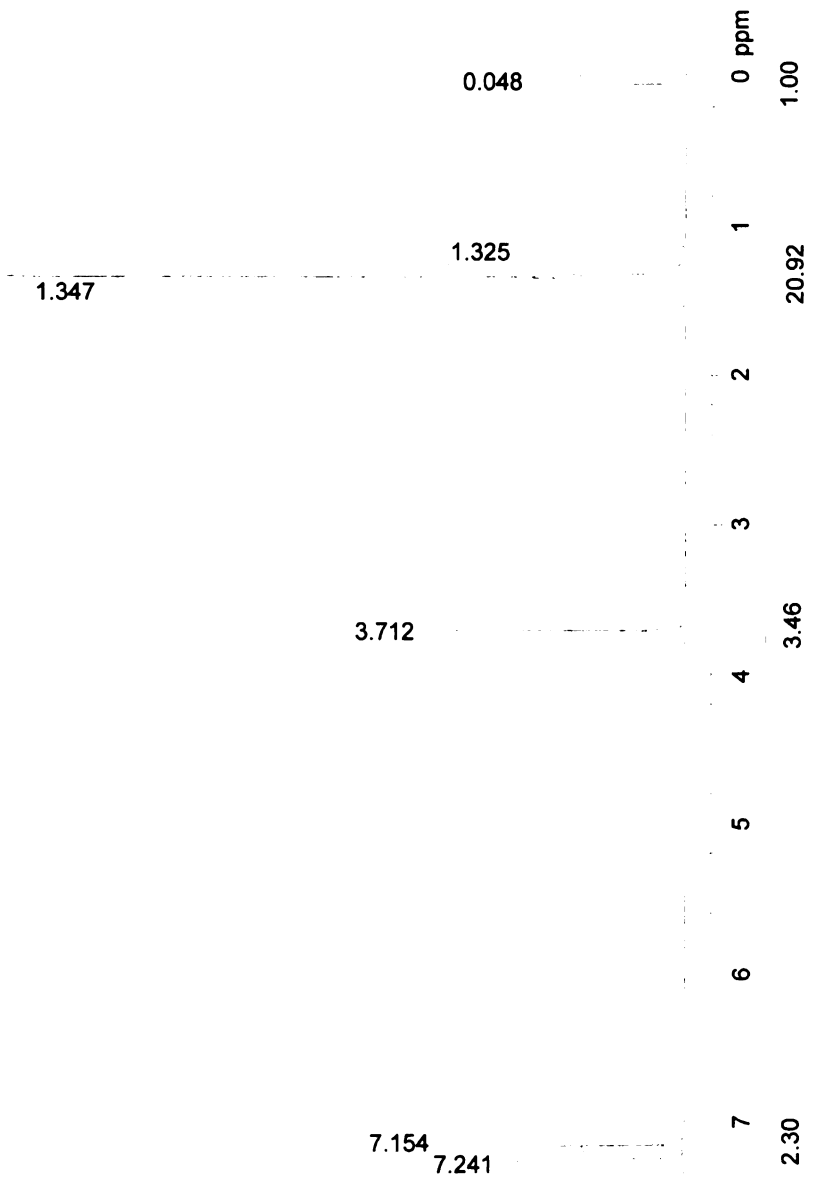
A6. ^{13}C NMR (75.5 MHz) of **6** (*3,5,3',5'*-tetra-*tert*-butyl-*4,4'*-dimethoxydiphenylmethanol) in $CDCl_3$ (δ 77.0) at 20°C.



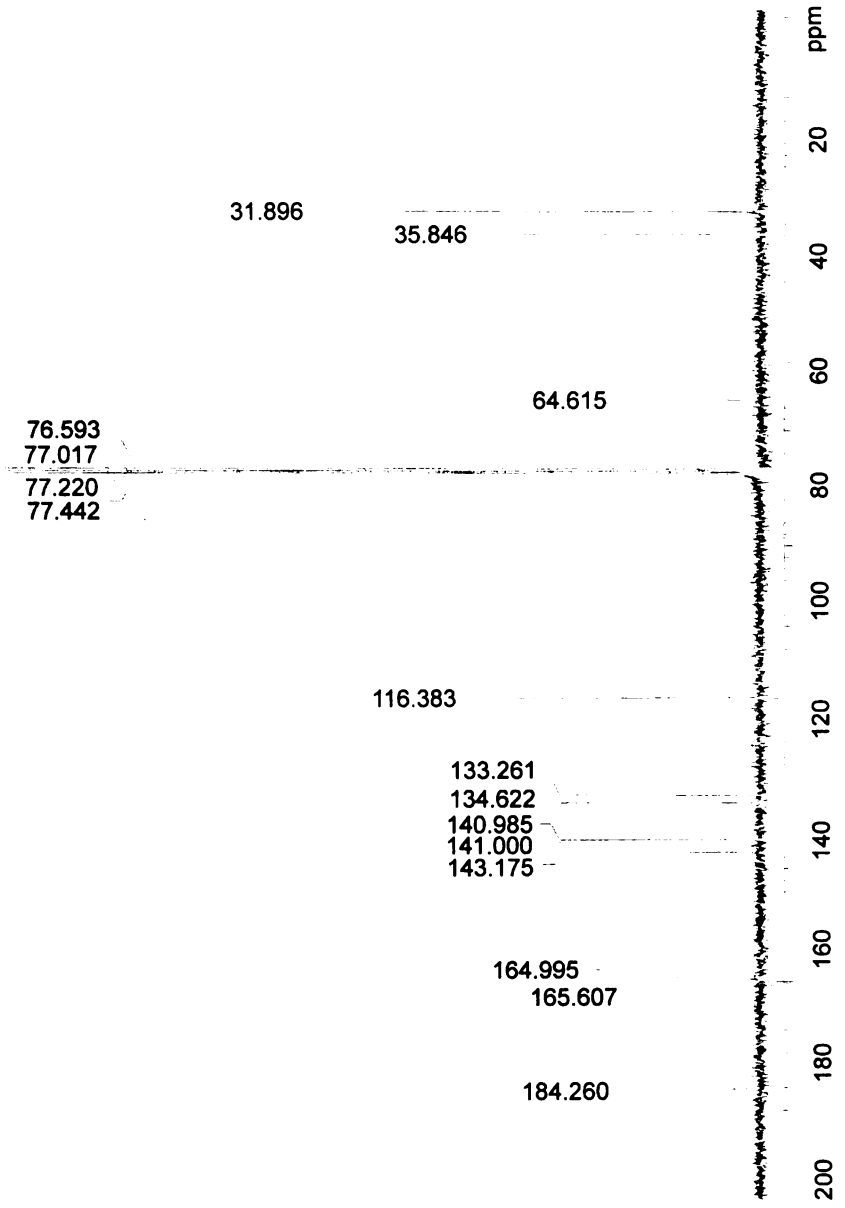
A7. ^{1}H NMR (300 MHz) of **2E** (*3,6*-bis(bis(*3,5*-di-*tert*-butyl-*4*-methoxyphenyl)methyl)-*2,5*-dihydroxy-*1,4*-benzoquinone) in $CDCl_3$ (δ 7.24) at 20°C.



A8. ^{13}C NMR (75.5 MHz) of **2E** (*3,6*-bis(bis(*3,5*-di-*tert*-butyl-*4*-methoxyphenyl)methyl)-*2,5*-dihydroxy-*1,4*-benzoquinone) in $CDCl_3$ (δ 77.0) at 20°C.



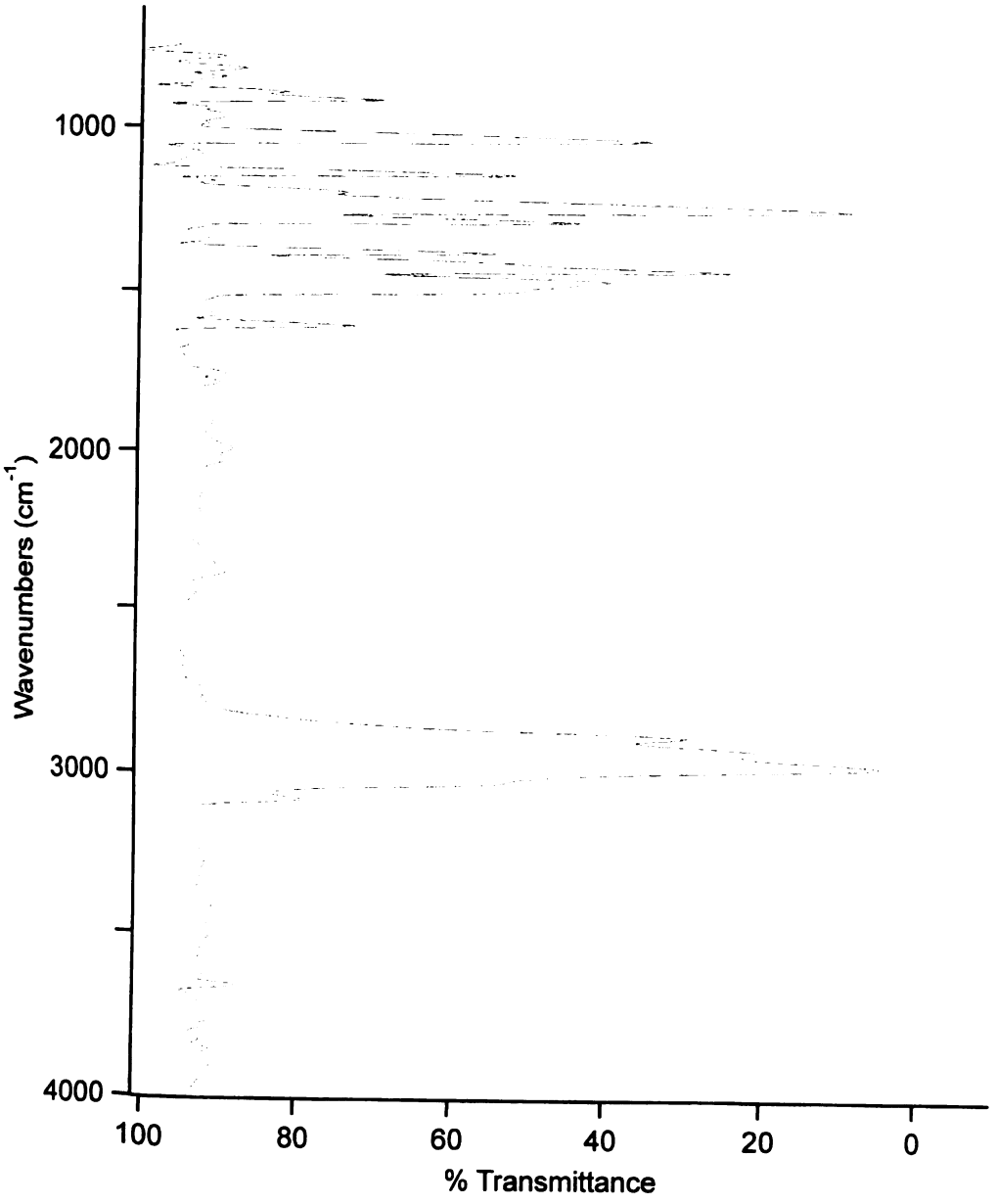
A9. ^{1}H NMR (300 MHz) of **1E** (*3,6*-bis(bis(*3,5*-di-*tert*-butyl-*4*-methoxyphenyl)methylene)-*1,2,4,5*-tetraoxocyclohexane) in $CDCl_3$ (δ 7.24) at 20°C.



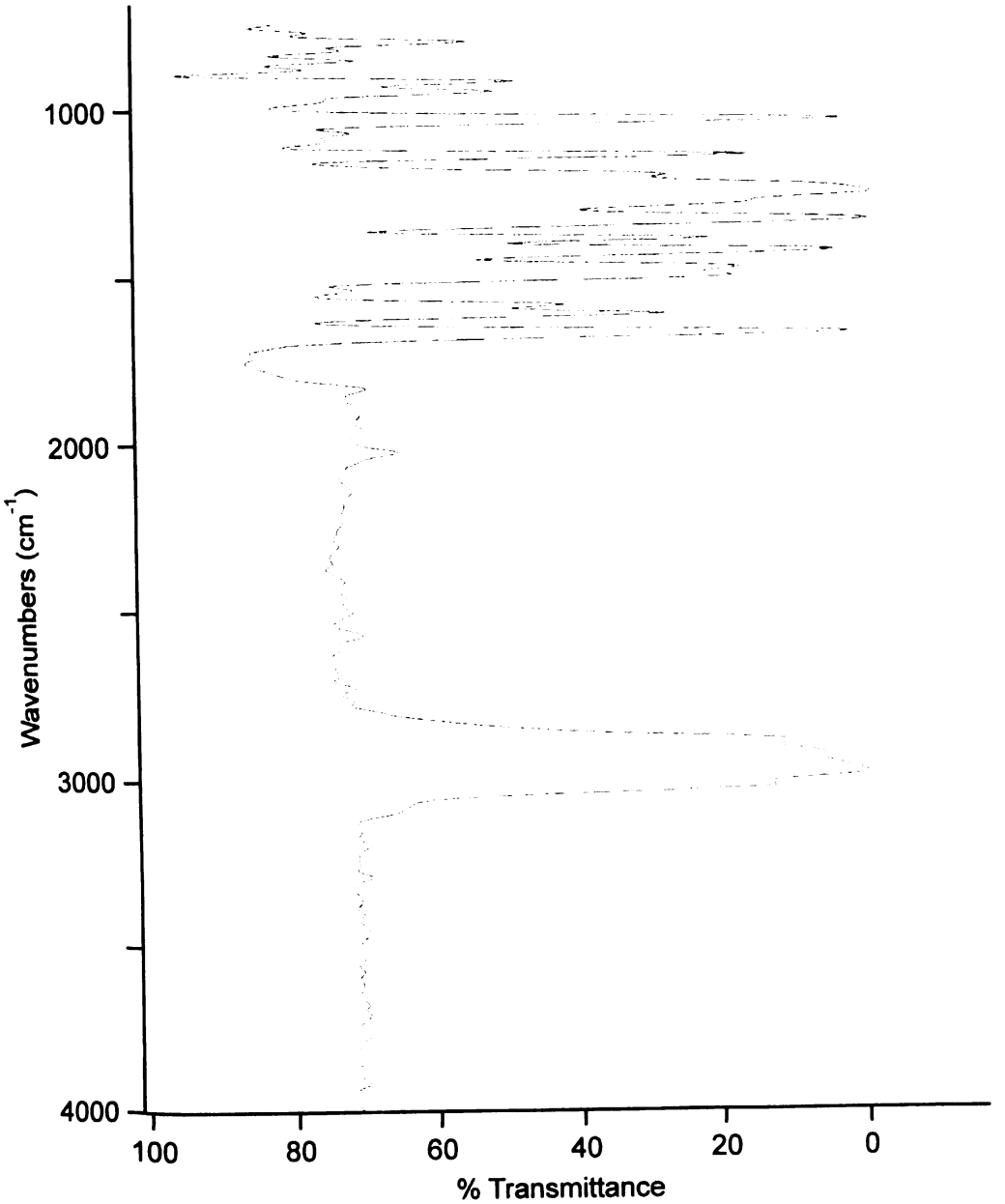
A10. ^{13}C NMR (75.5 MHz) of **1E** (*3,6*-bis(bis(*3,5*-di-*tert*-butyl-*4*-methoxyphenyl)methylene)-*1,2,4,5*-tetraoxocyclohexane) in $CDCl_3$ (δ 77.0) at 20°C.

APPENDIX B

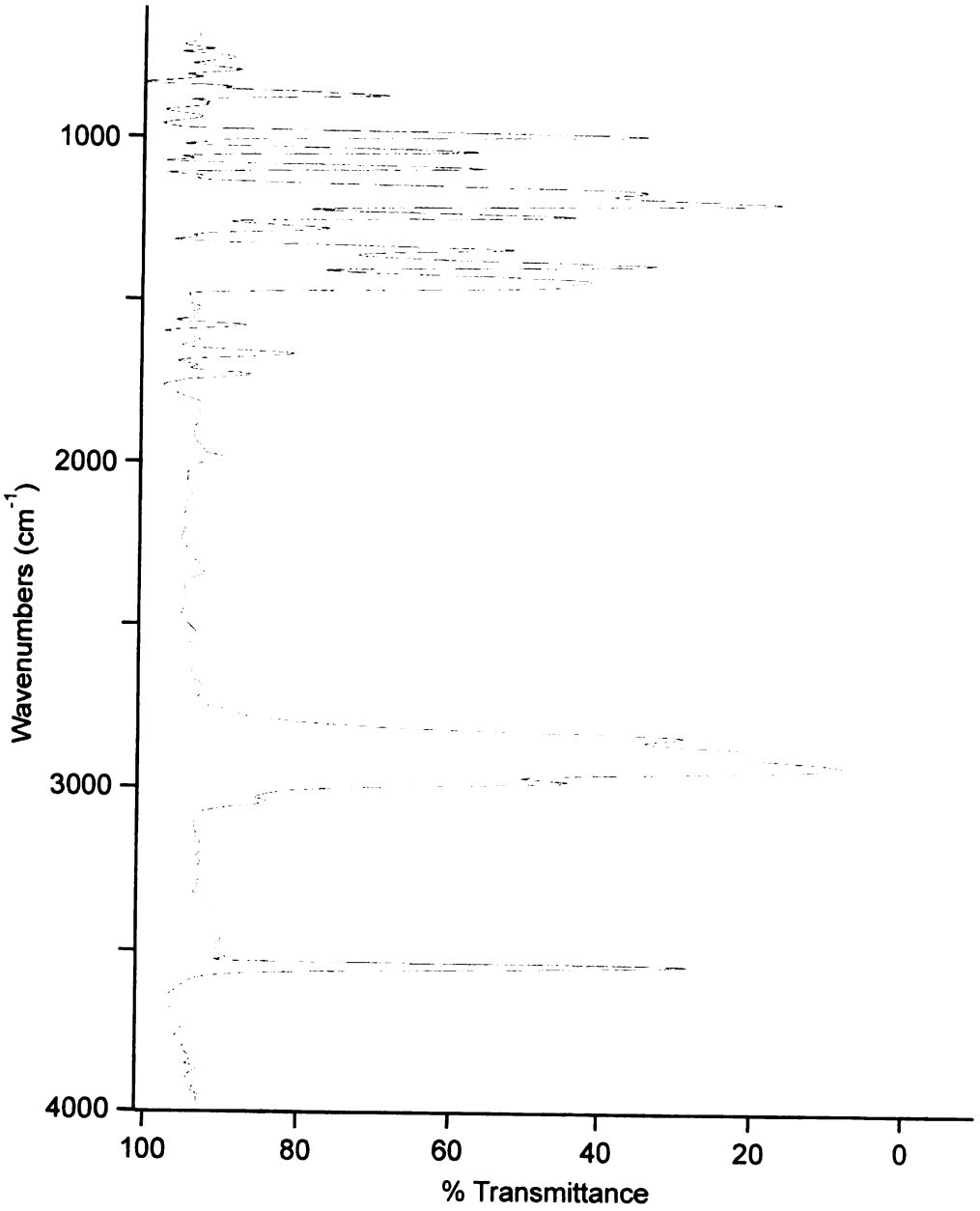
Infrared Spectra



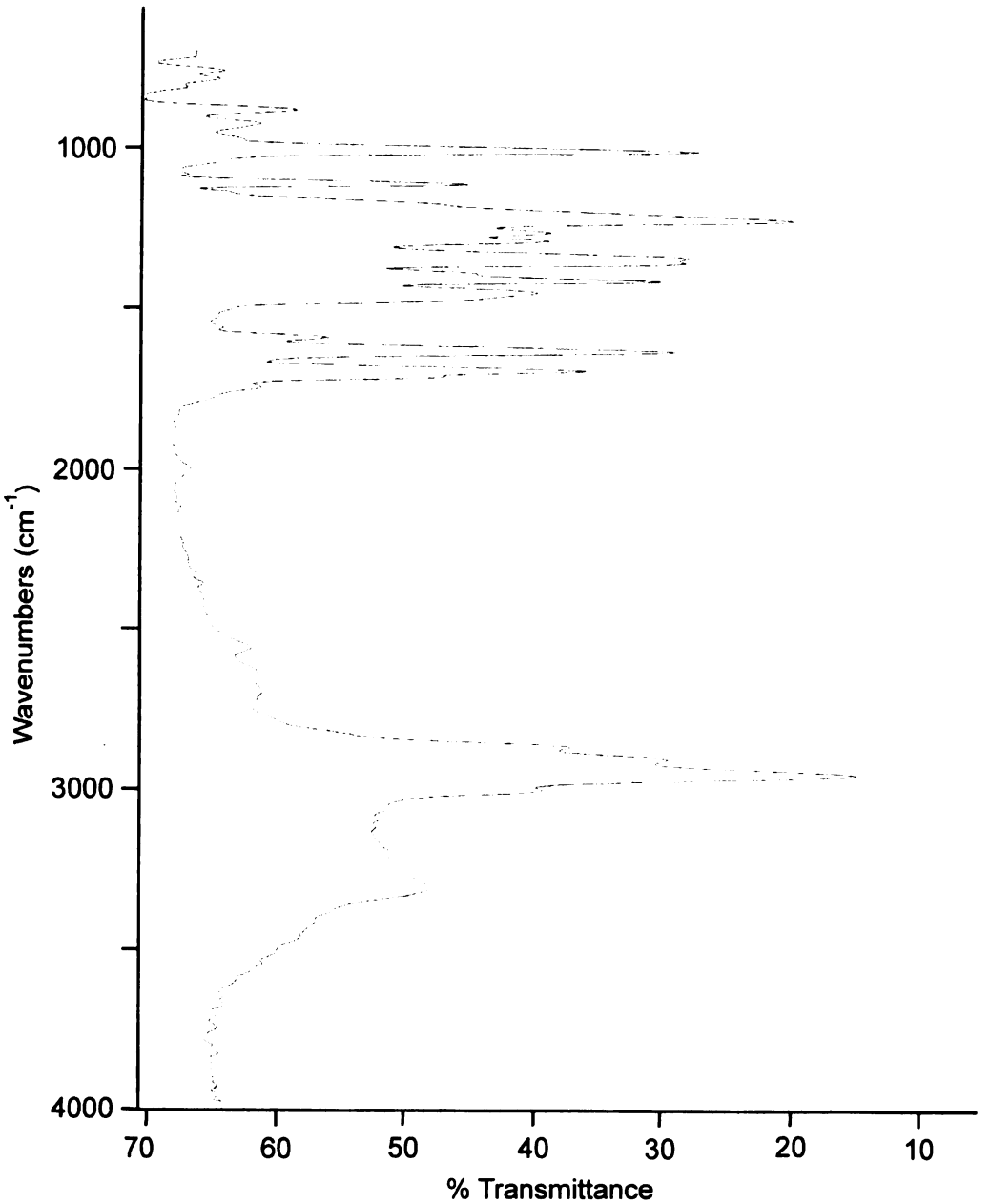
B1. FT-IR spectrum of **4** (*3,5,3',5'*-tetra-*tert*-butyl-*4,4'*-dimethoxydiphenylmethane) in neat KBr at 20°C.



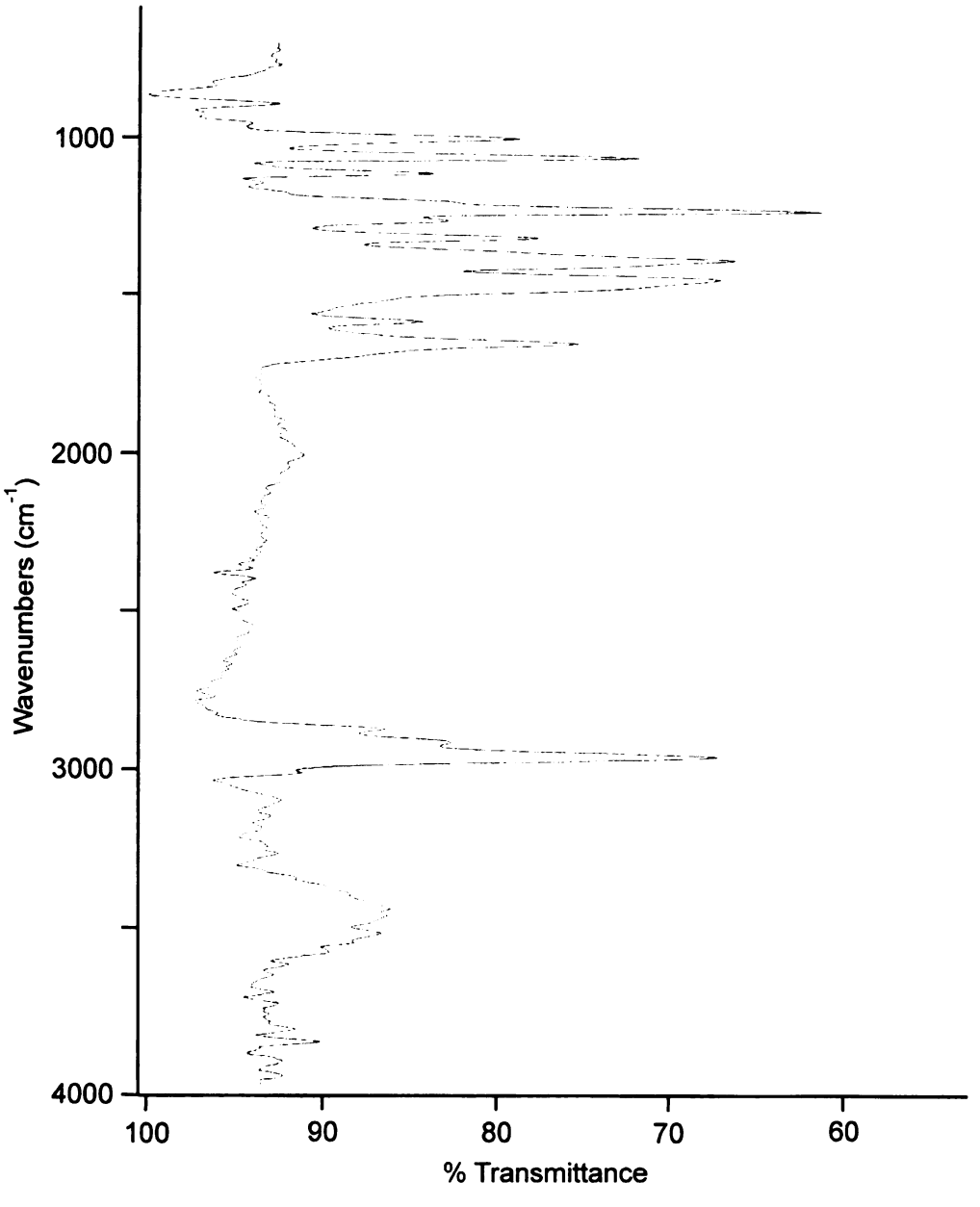
B2. FT-IR spectrum of **5** (*3,5,3',5'*-tetra-*tert*-butyl-*4,4'*-dimethoxydiphenylmethanone) in neat KBr at 20°C.



B3. FT-IR spectrum of **6** (*3,5,3',5'*-tetra-*tert*-butyl-*4,4'*-dimethoxydiphenylmethanol) in neat KBr at 20°C.



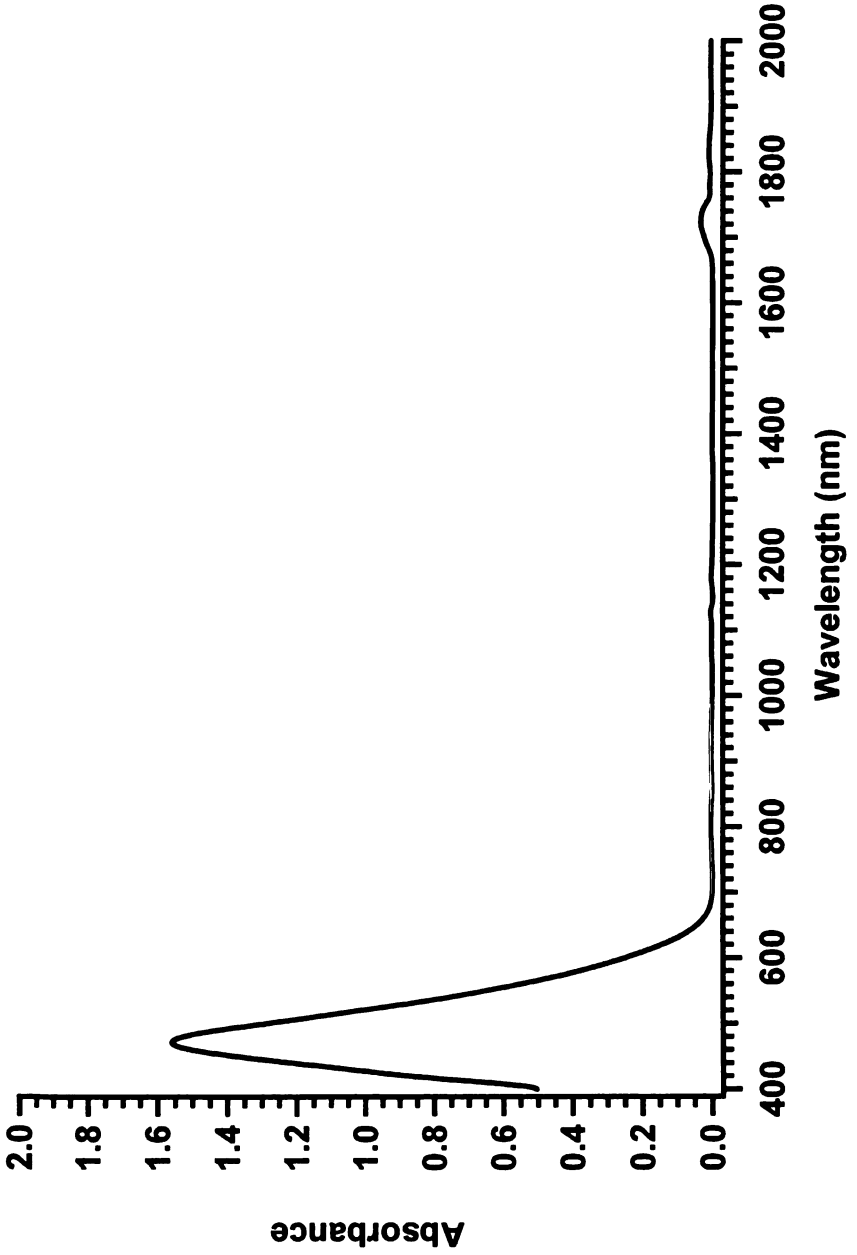
B4. FT-IR spectrum of **2E** (*3,6*-bis(bis(*3,5*-di-*tert*-butyl-*4*-methoxyphenyl)methyl)-*2,5*-dihydroxy-*1,4*-benzoquinone) in neat KBr at 20°C.



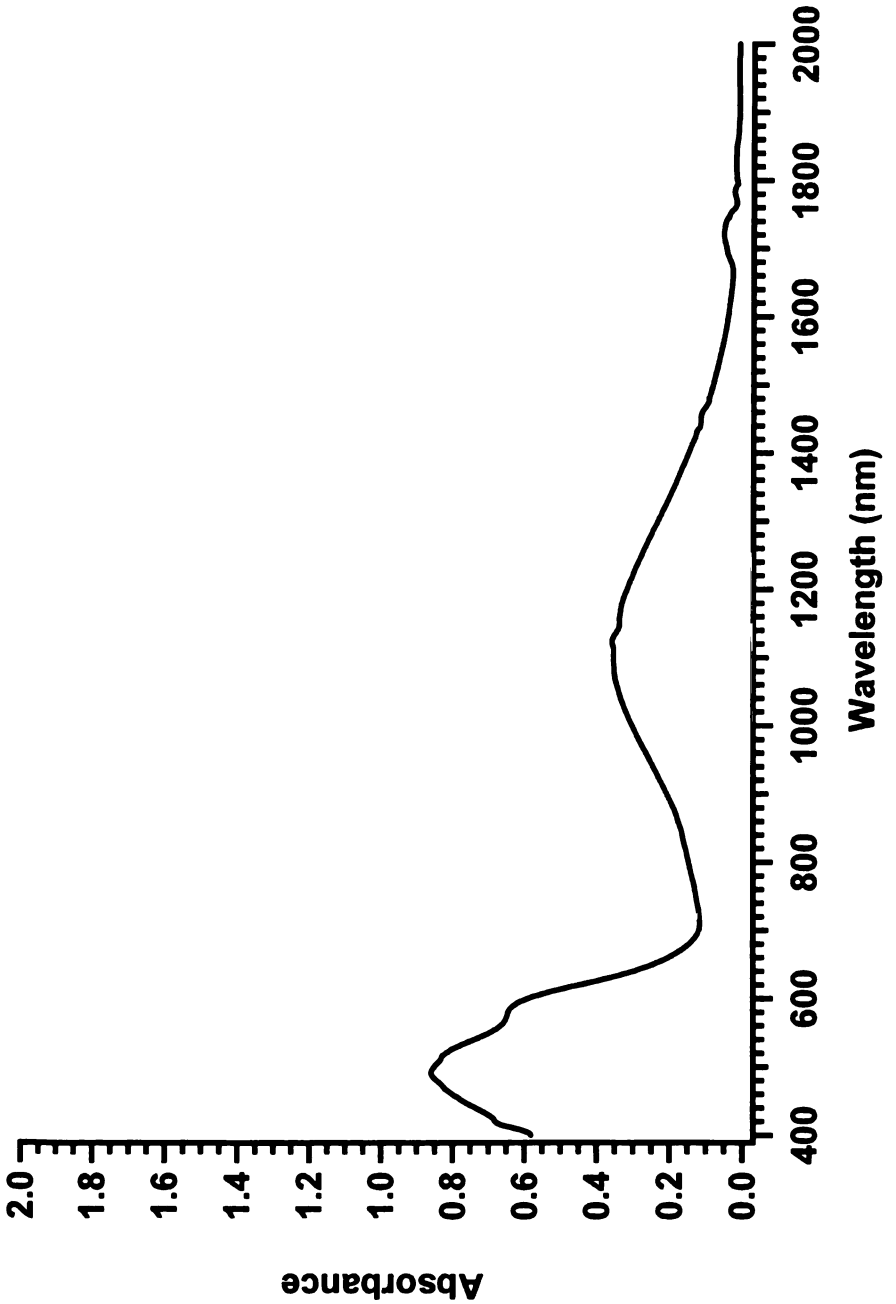
B5. FT-IR spectrum of **1E** (*3*,*6*-bis(bis(*3*,*5*-di-*tert*-butyl-*4*-methoxyphenyl)methylene)-*1*,*2*,*4*,*5*-tetraoxocyclohexane) in neat KBr at 20°C.

APPENDIX C

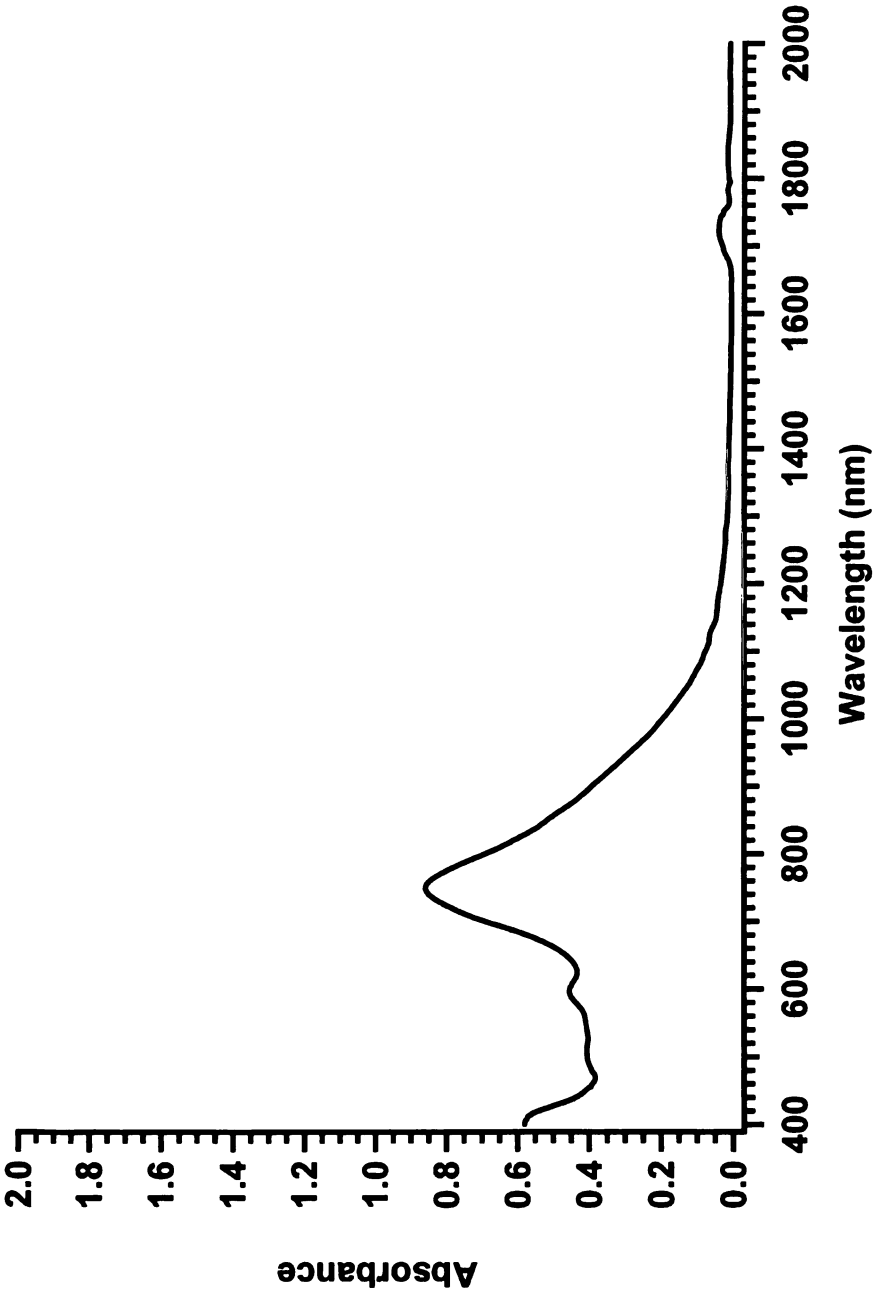
Visible/Near Infrared Spectra



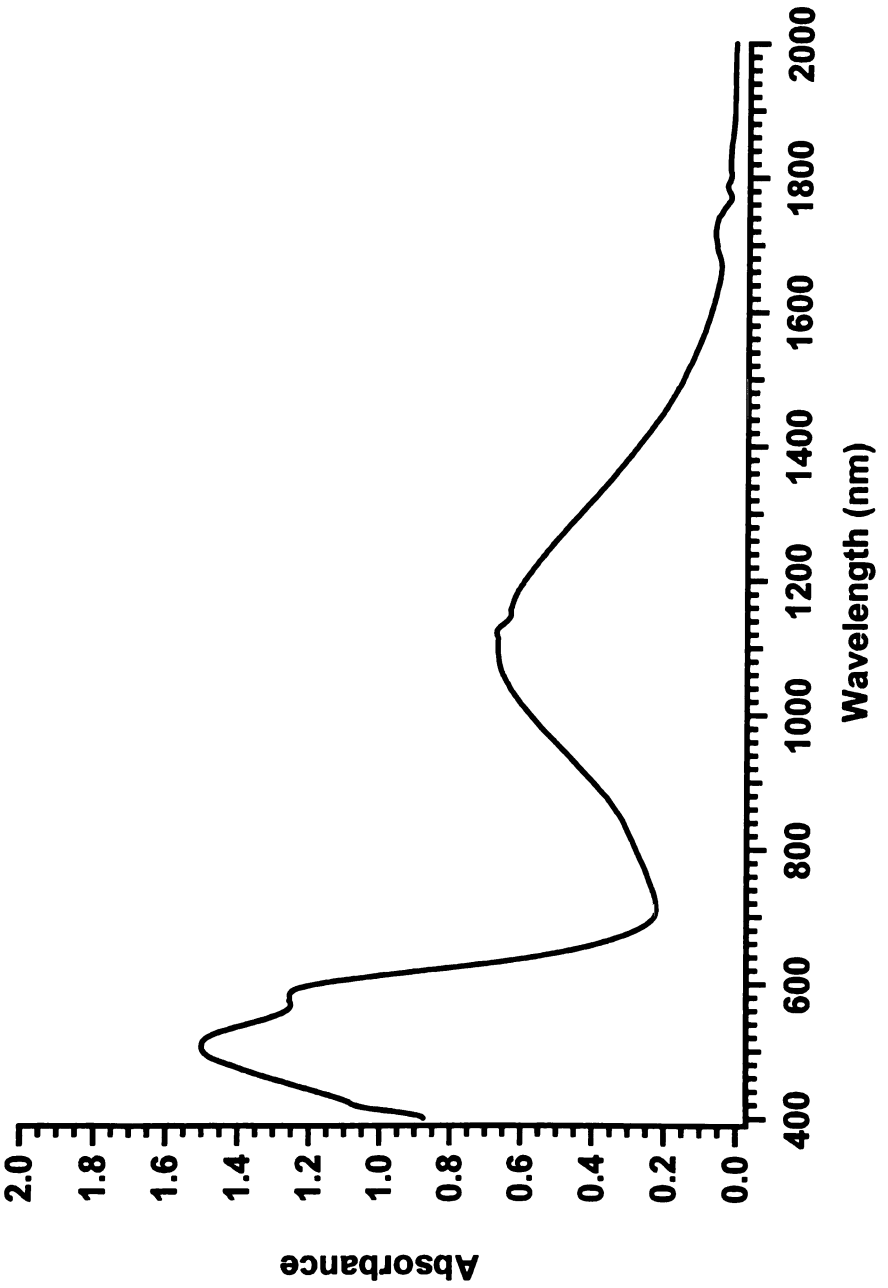
C1. Vis-NIR spectrum of **1E** (*3*,*6*-bis(bis(*3*,*5*-di-*tert*-butyl-*4*-methoxyphenyl)methylene)-*1*,*2*,*4*,*5*-tetraoxocyclohexane) in tetrahydrofuran at 20°C.



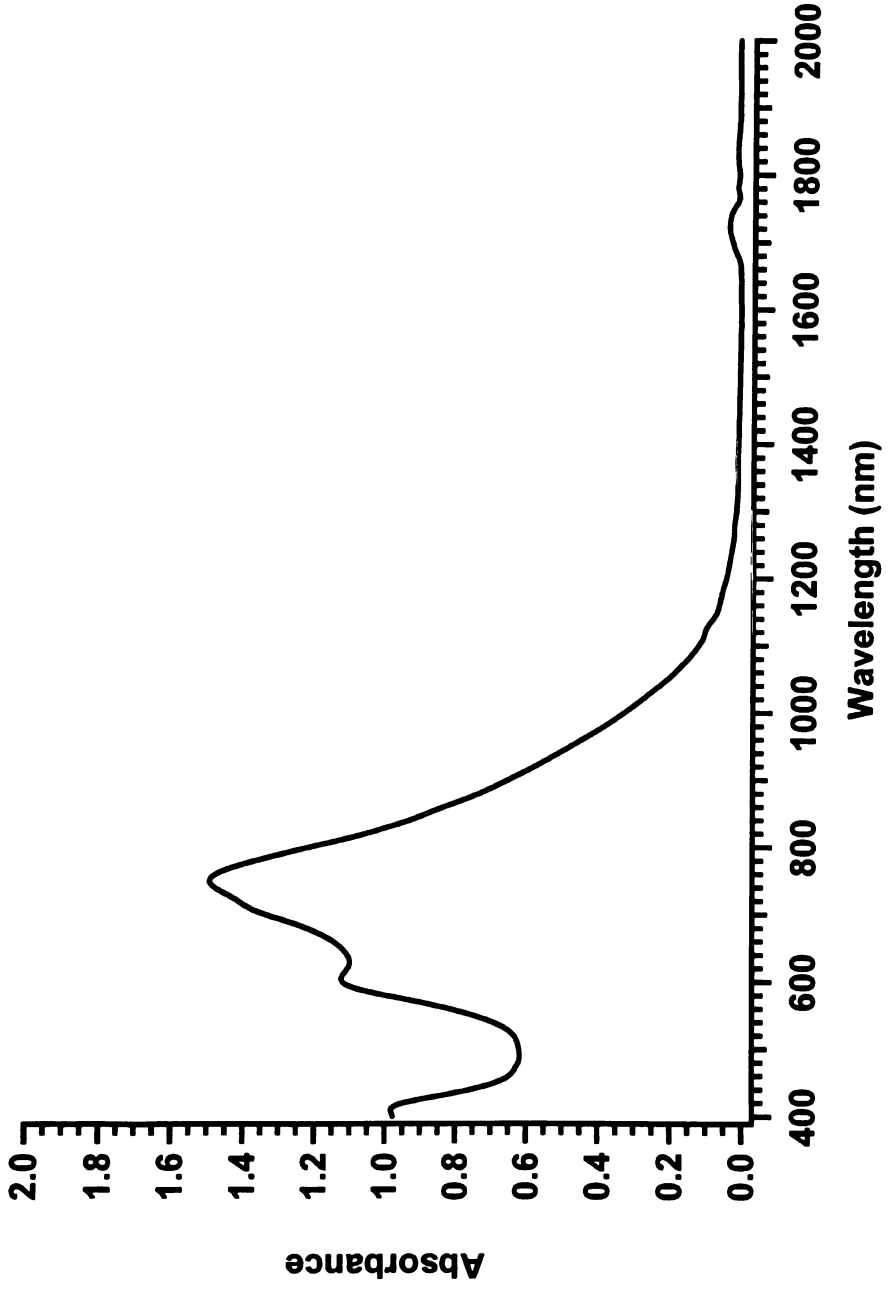
C2. Vis-NIR spectrum of $Cs^{+}•1E^{-•}$ (*3,6*-bis(bis(*3,5*-di-*tert*-butyl-*4*-methoxyphenyl)methylene)-*1,2,4,5*-tetraoxocyclohexane) monoradical monoanion in tetrahydrofuran at 20°C.



C3. Vis-NIR spectrum of $(Cs^{+})_2 \bullet 1E^{2-\bullet\bullet}$ (*3,6*-bis(bis(*3,5*-di-*tert*-butyl-*4*-methoxyphenyl)methylene)-*1,2,4,5*-tetraoxocyclohexane) diradical dianion in tetrahydrofuran at 20°C.

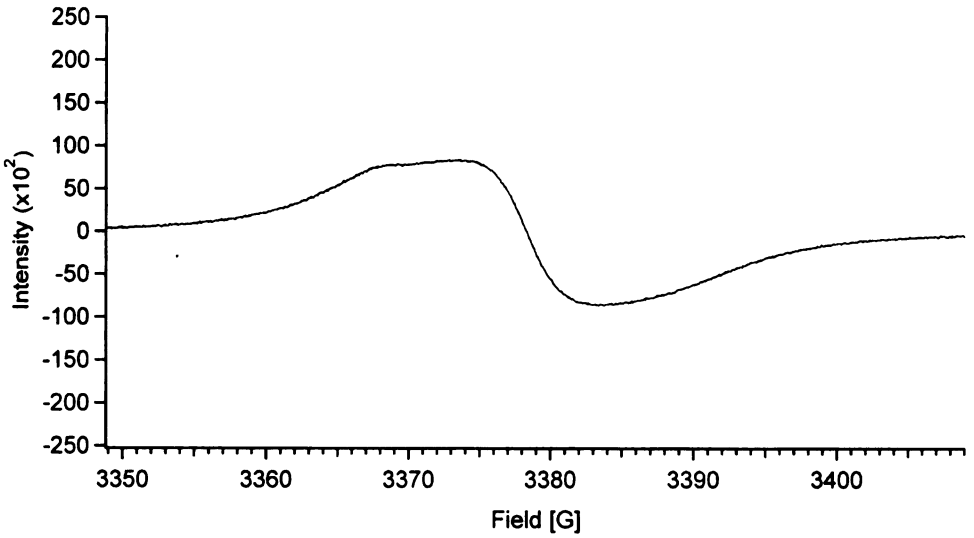


C4. Vis-NIR spectrum of the conproportionation reaction between $(Cs^{+})_2 \bullet 1E^{2-\bullet\bullet}$ and **1E** to generate $Cs^{+} \bullet 1E^{-\bullet}$ in tetrahydrofuran at 20°C.

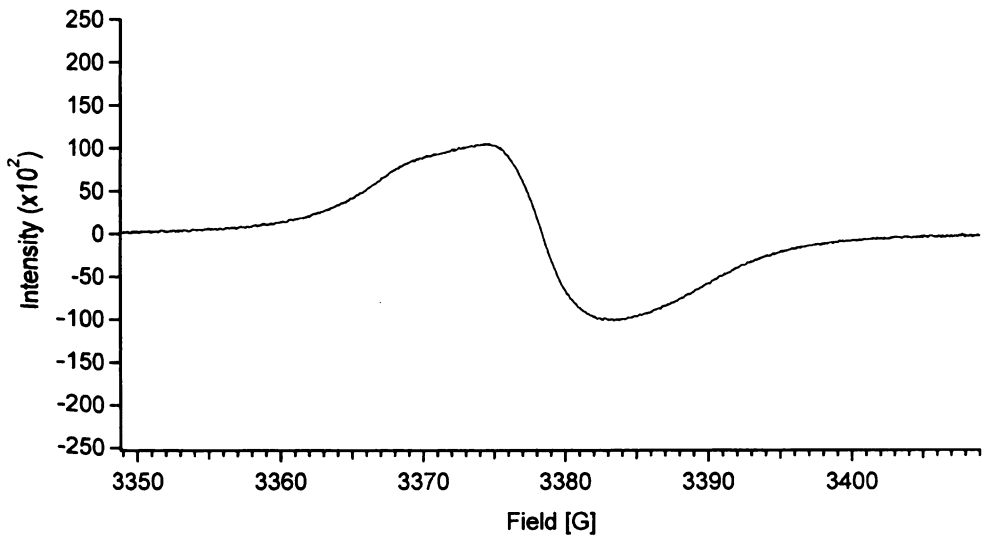


C5. Vis-NIR spectrum of $(Cs^{+})_2 \bullet 1E^{2-\bullet\bullet}$ (3,6-bis(bis(3,5-di-*tert*-butyl-4-methoxyphenyl)methylene)-1,2,4,5-tetraoxocyclohexane) diradical dianion in tetrahydrofuran at 20°C.

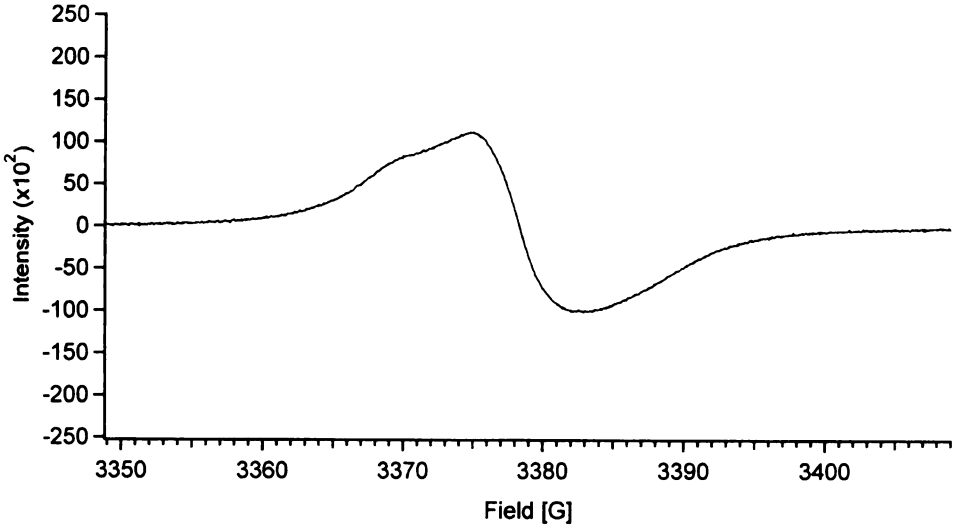
APPENDIX D



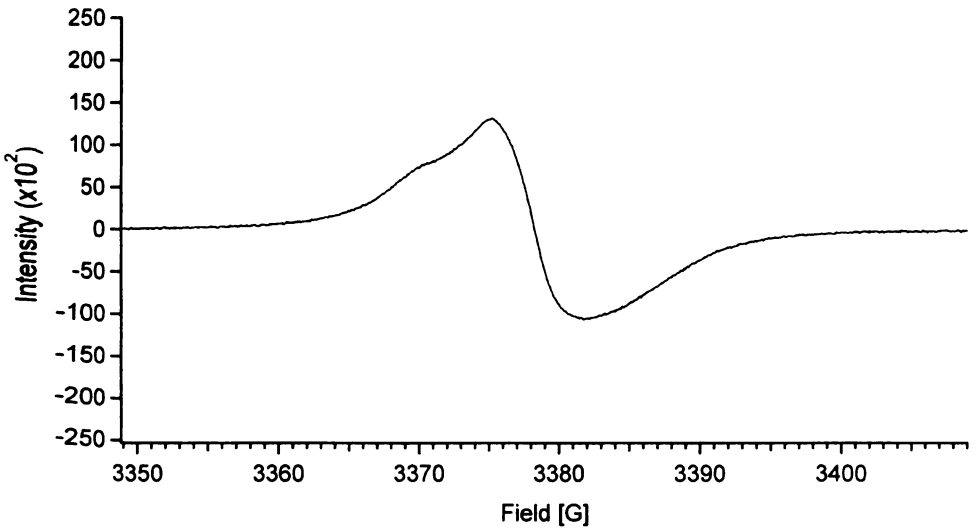
D1. EPR spectrum of $Li^+ \bullet 1E^{-\bullet}$ in tetrahydrofuran at 4.2 K.



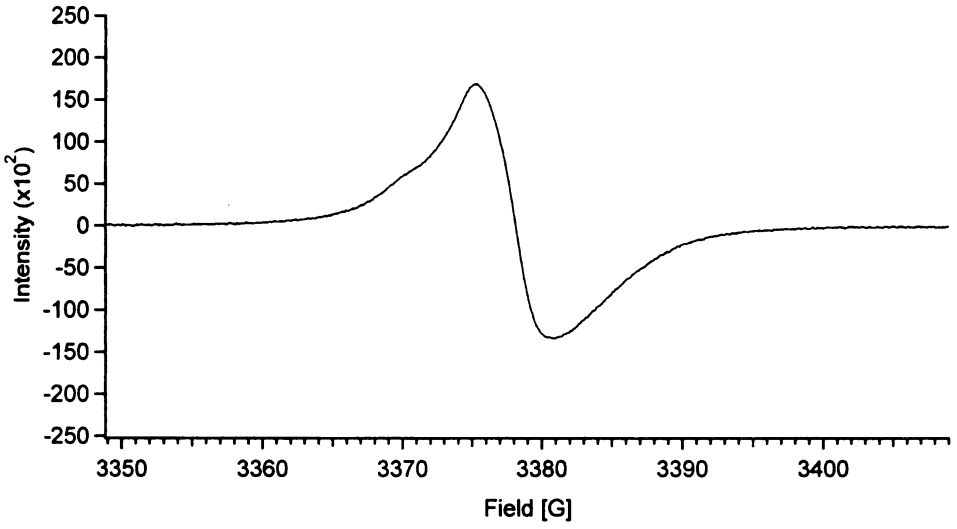
D2. EPR spectrum of $Li^+ \bullet 1E^{-\bullet}$ in tetrahydrofuran at 8.0 K.



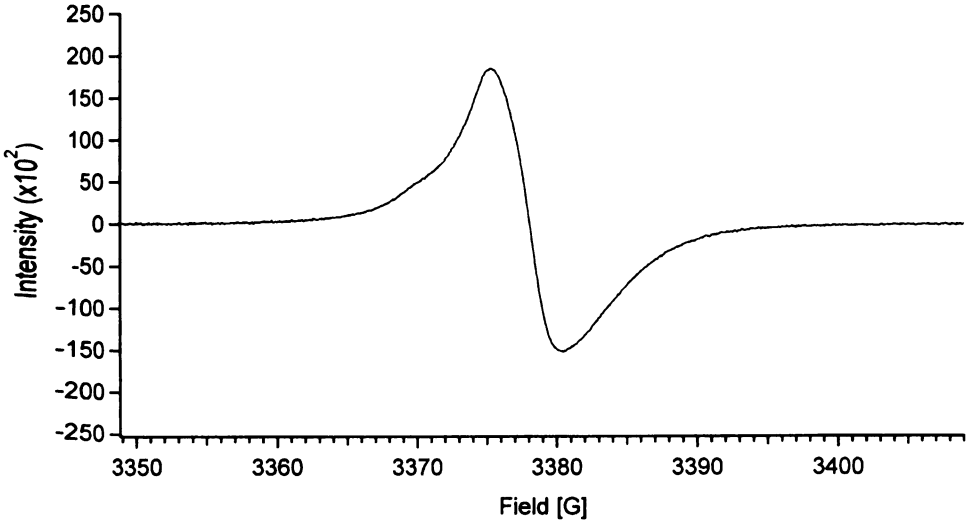
D3. EPR spectrum of $Li^+ \bullet 1E^{-\bullet}$ in tetrahydrofuran at 12.0 K.



D4. EPR spectrum of $Li^+ \bullet 1E^{-\bullet}$ in tetrahydrofuran at 18.0 K.



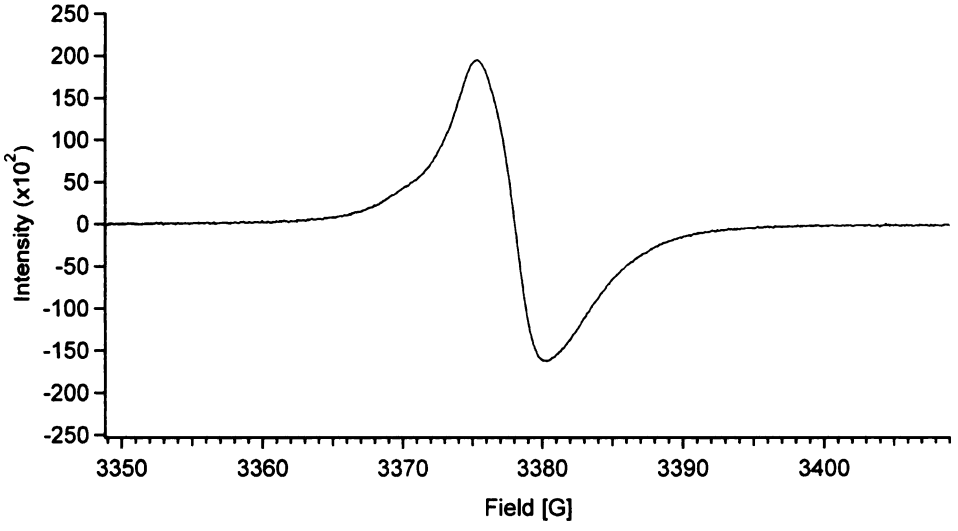
D5. EPR spectrum of $Li^+ \bullet 1E^{-\bullet}$ in tetrahydrofuran at 30.0 K.



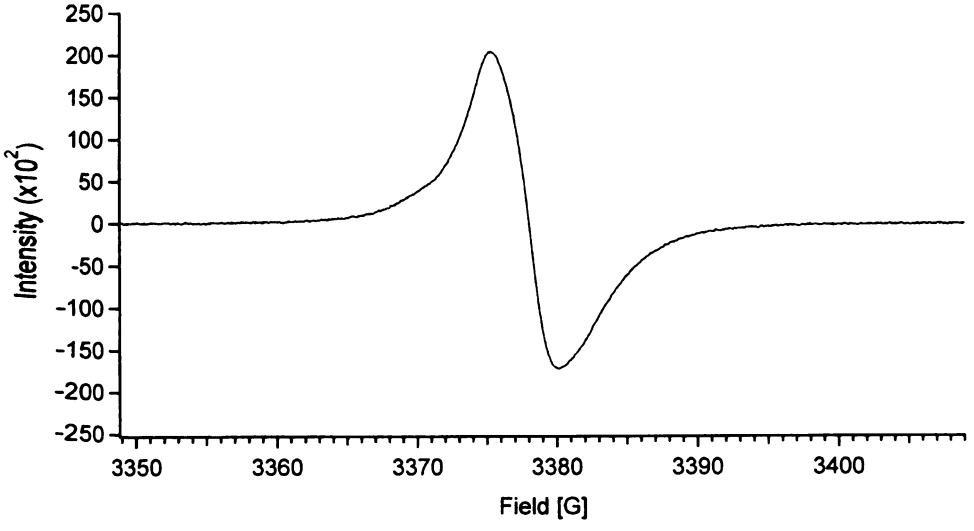
D6. EPR spectrum of $Li^+ \bullet 1E^{-\bullet}$ in tetrahydrofuran at 40.0 K.

Intensity (x10⁵)
3350
EPR s

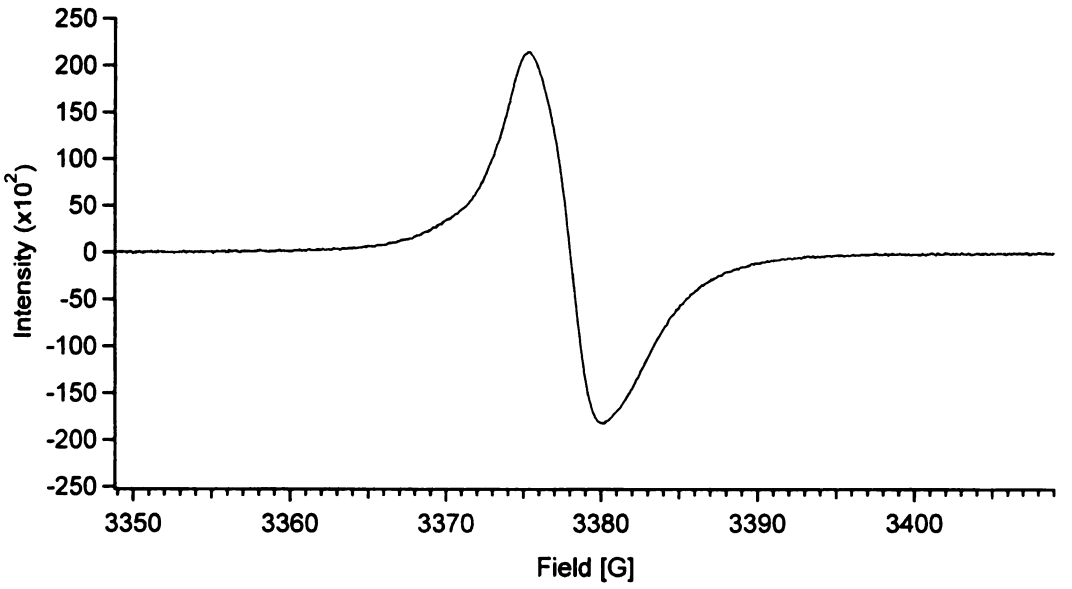
Intensity (x10⁵)
3350
EPR s



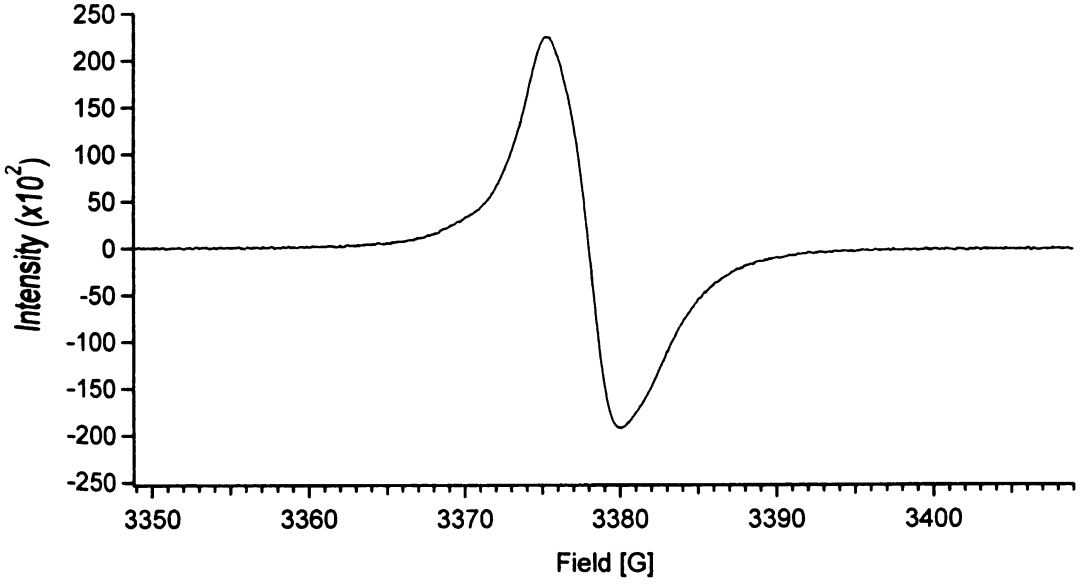
D7. EPR spectrum of Li^{+}•**1**$E^{-\bullet}$ in tetrahydrofuran at 50.0 K.



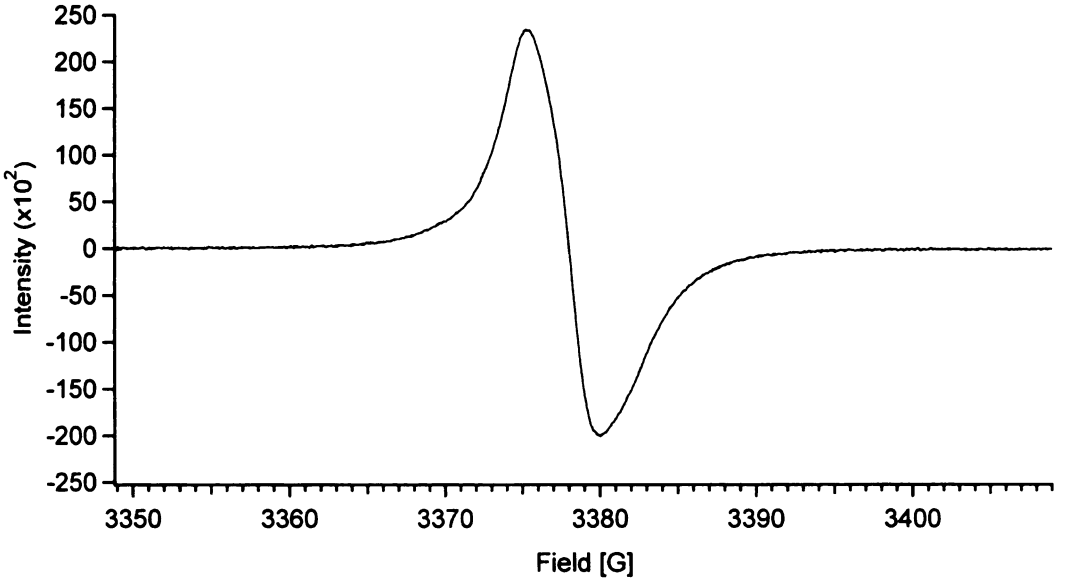
D8. EPR spectrum of Li^{+}•**1**$E^{-\bullet}$ in tetrahydrofuran at 60.0 K.



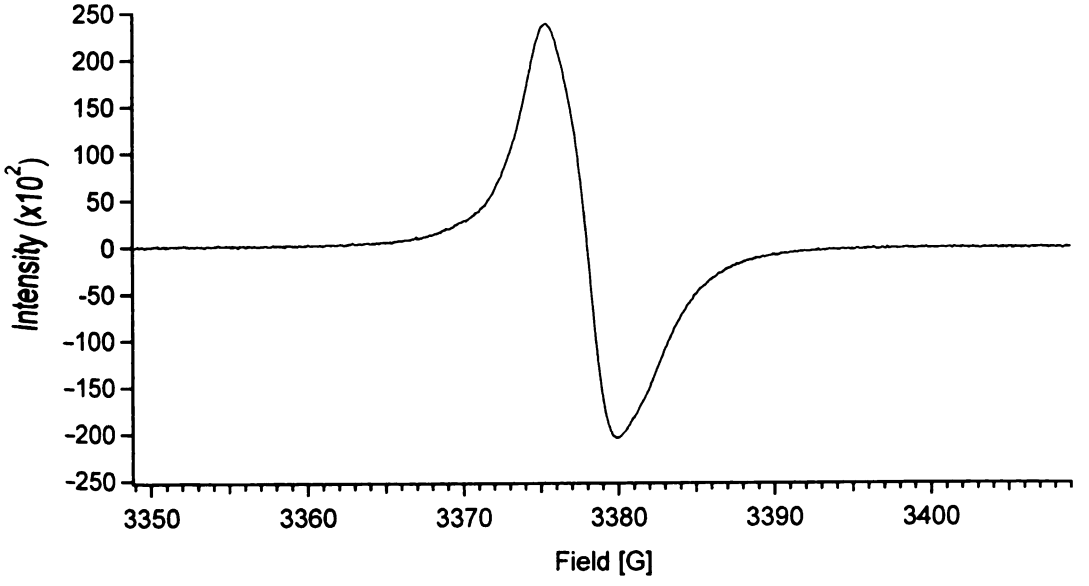
D9. EPR spectrum of $Li^+ \bullet 1E^{-\bullet}$ in tetrahydrofuran at 70.0 K.



D10. EPR spectrum of $Li^+ \bullet 1E^{-\bullet}$ in tetrahydrofuran at 80.0 K.



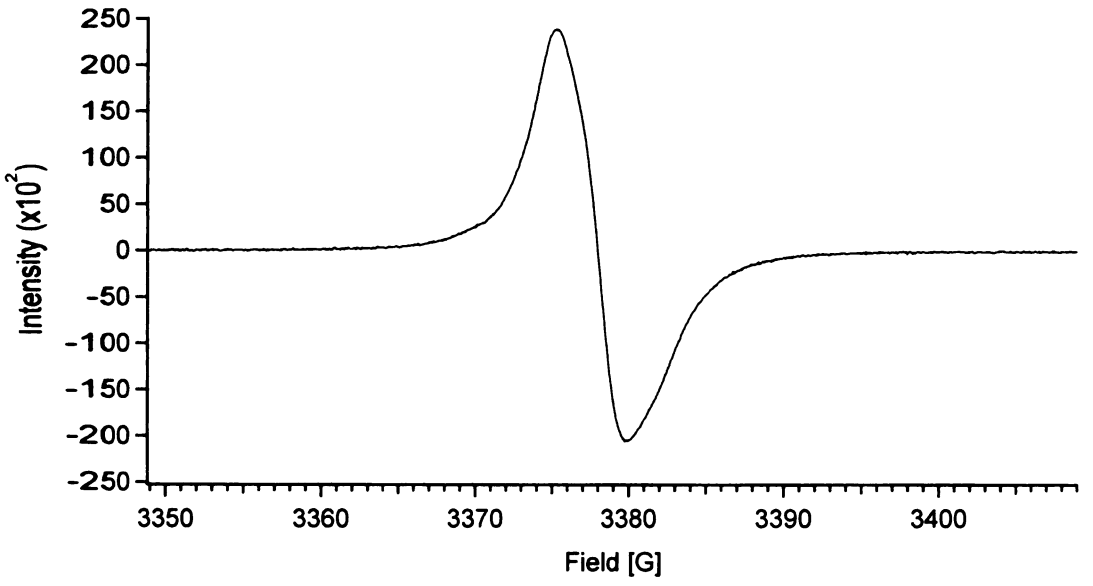
D11. EPR spectrum of $Li^{+}•1E^{-•}$ in tetrahydrofuran at 90.0 K.



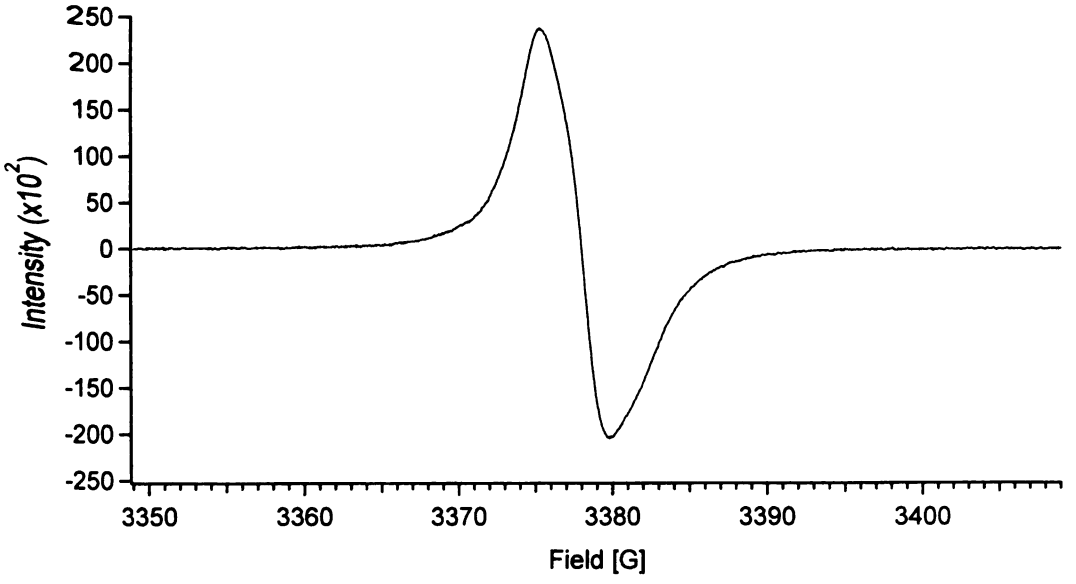
D12. EPR spectrum of $Li^{+}•1E^{-•}$ in tetrahydrofuran at 100.0 K.

23 EPR

24 EP



D13. EPR spectrum of $Li^{+}•1E^{-•}$ in tetrahydrofuran at 110.0 K.



D14. EPR spectrum of $Li^{+}•1E^{-•}$ in tetrahydrofuran at 120.0 K.

Intensity (x10²)

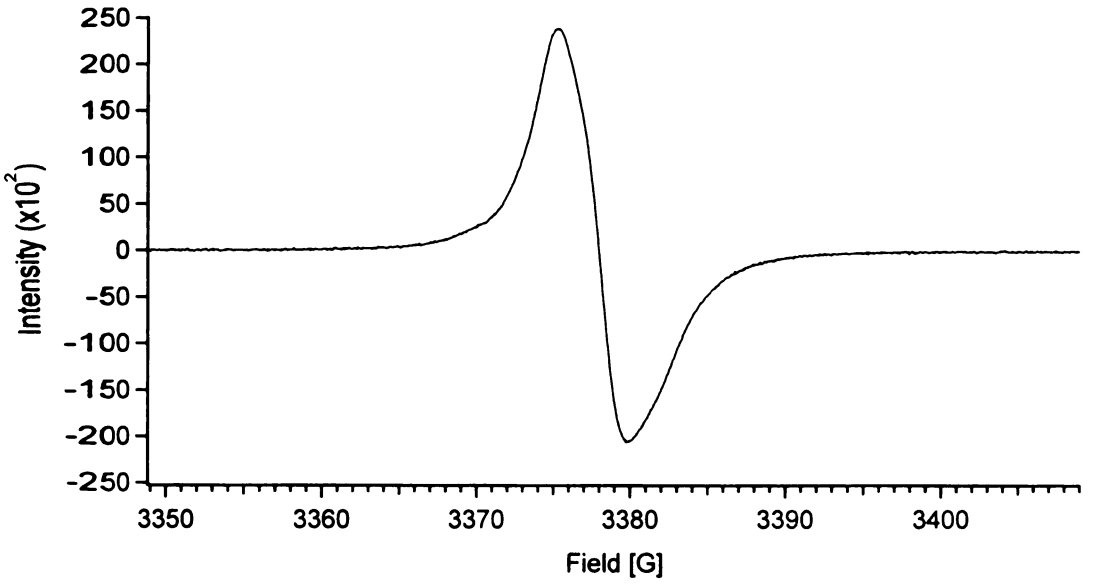
3350

3(3 EPR s

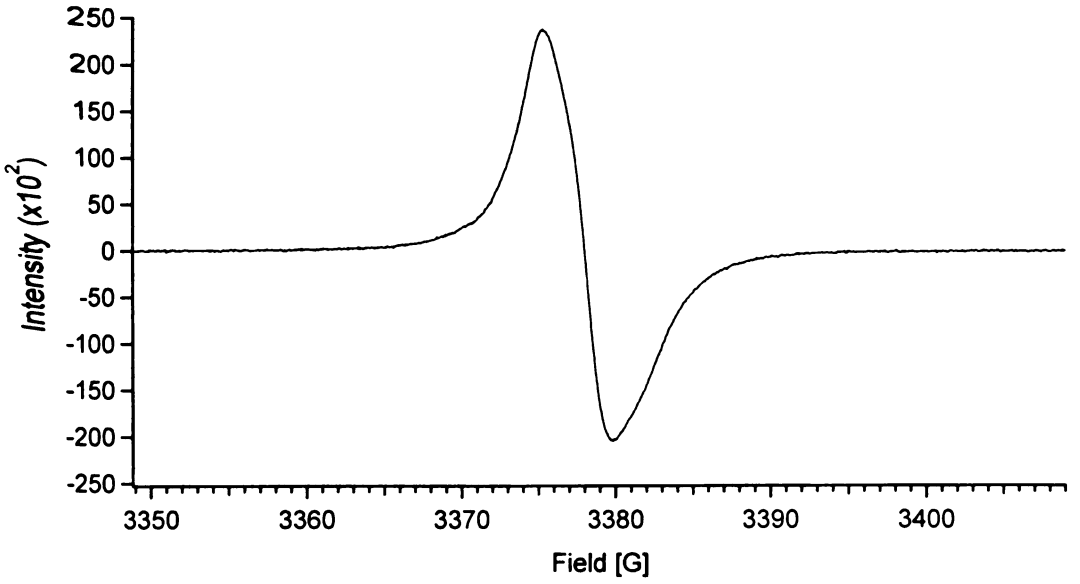
Intensity (x10²)

3351

D'4 EPR s



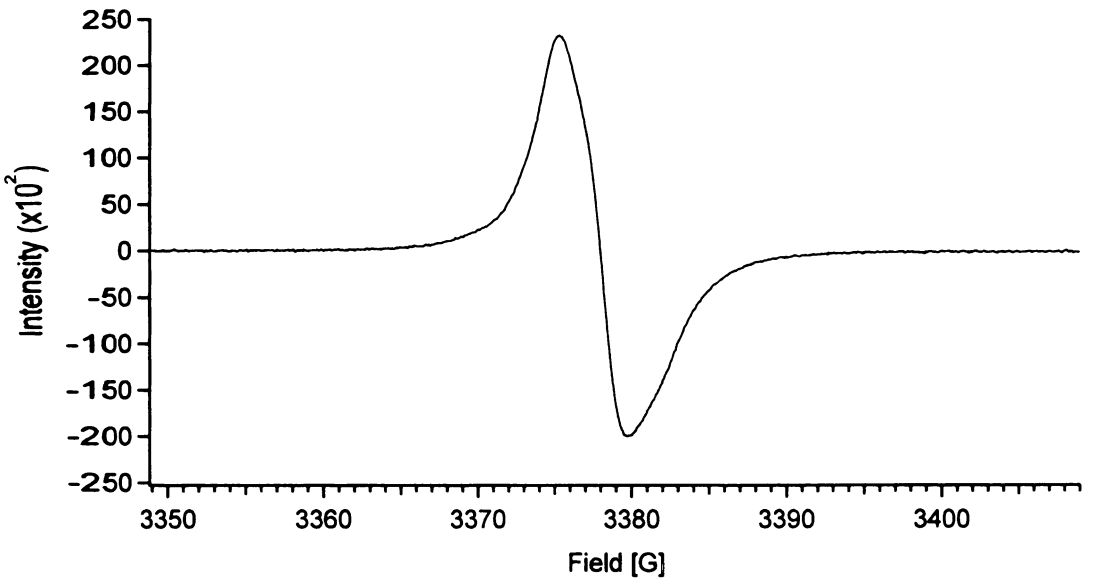
D13. EPR spectrum of $Li^+ \bullet 1E^{-\bullet}$ in tetrahydrofuran at 110.0 K.



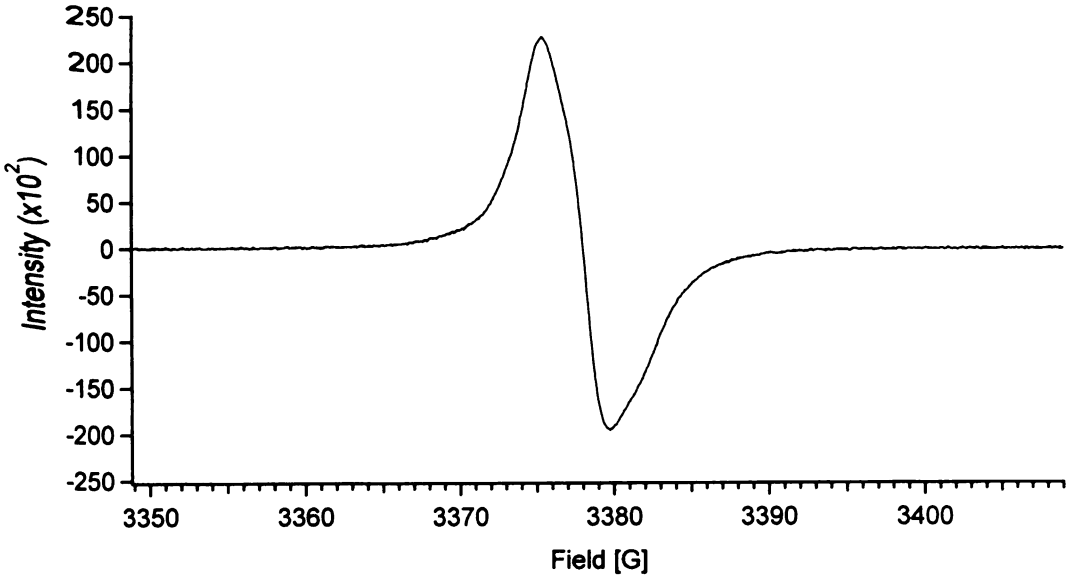
D14. EPR spectrum of $Li^+ \bullet 1E^{-\bullet}$ in tetrahydrofuran at 120.0 K.

D15. EPR s

D16. EPR



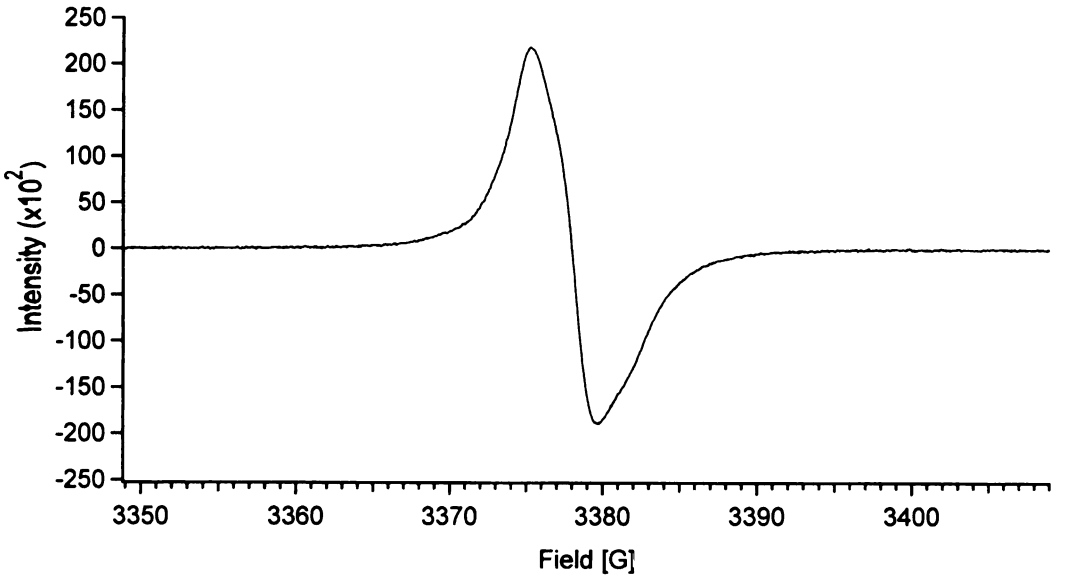
D15. EPR spectrum of $Li^{+}\bullet 1E^{-\bullet}$ in tetrahydrofuran at 130.0 K.



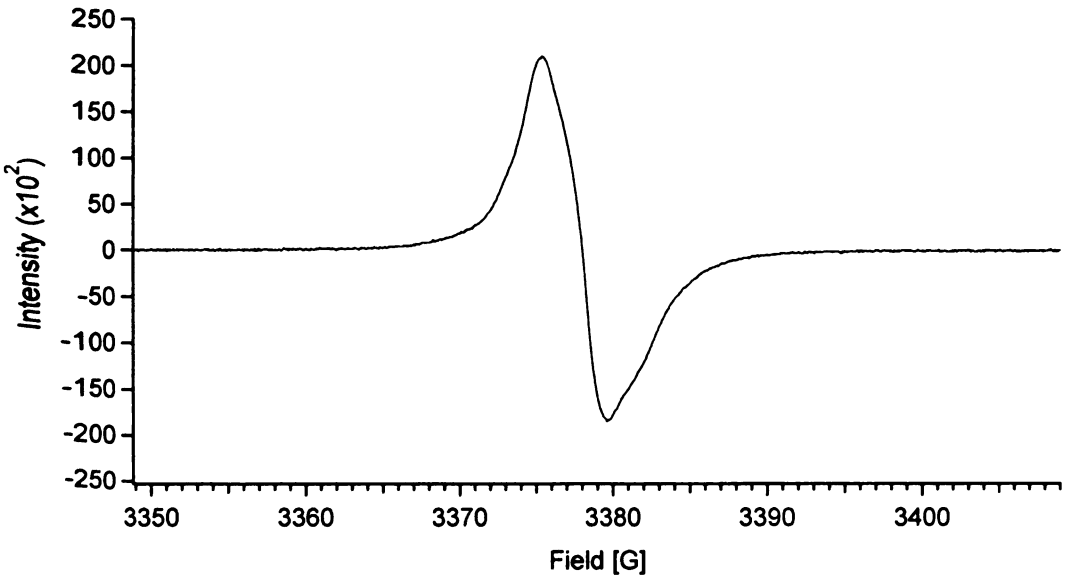
D16. EPR spectrum of $Li^{+}\bullet 1E^{-\bullet}$ in tetrahydrofuran at 140.0 K.

EPR s

EPR



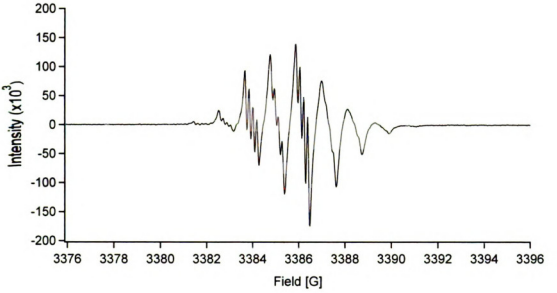
D17. EPR spectrum of $Li^{+}•1E^{-•}$ in tetrahydrofuran at 150.0 K.



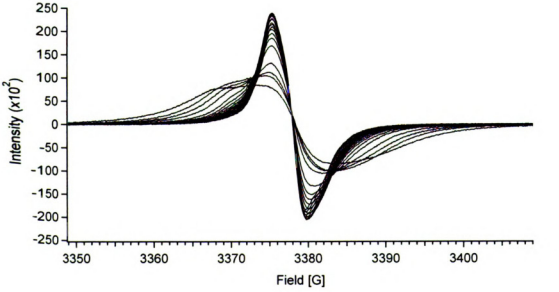
D18. EPR spectrum of $Li^{+}•1E^{-•}$ in tetrahydrofuran at 160.0 K.

Intensity (x10³)
EPR

Intensity (x10³)
EPR

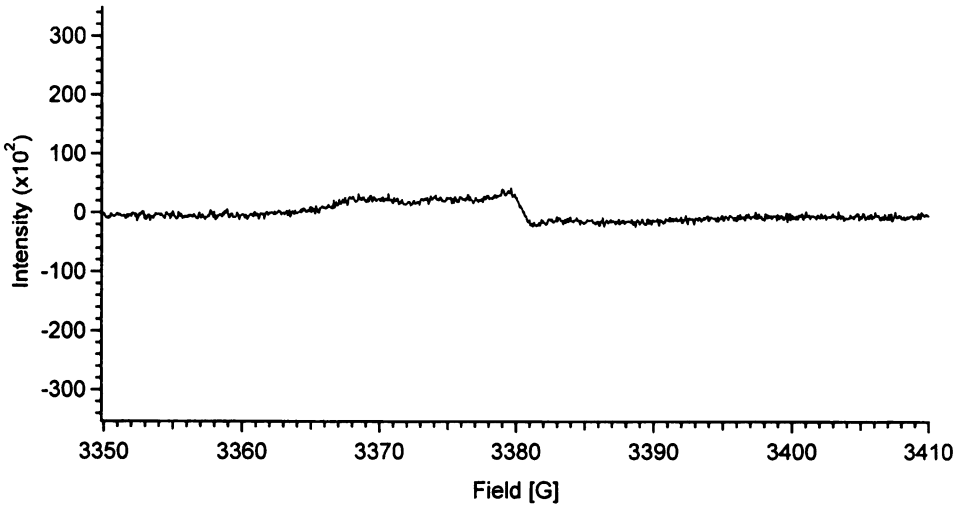


D19. EPR spectrum of $Li^+ \bullet 1E^{-\bullet}$ in tetrahydrofuran at 293.0 K.

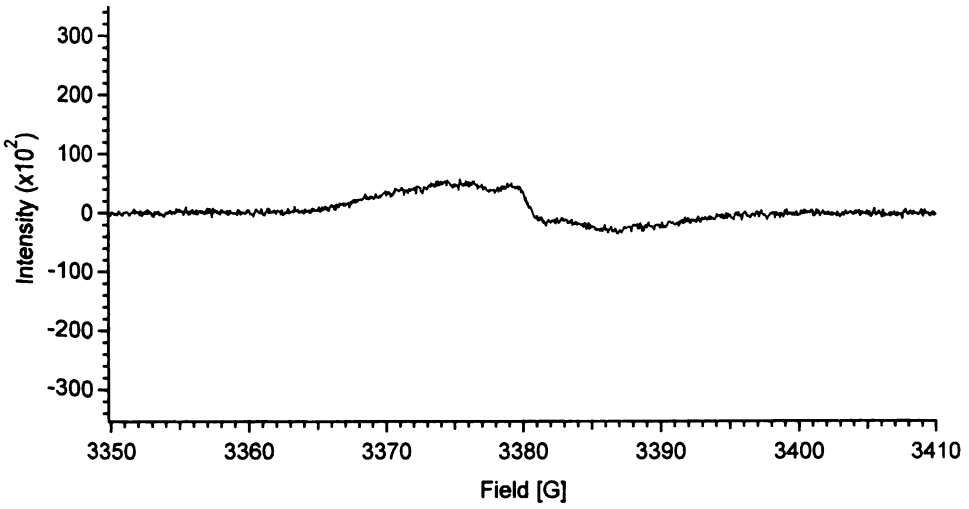


D20. EPR spectra of $Li^+ \bullet 1E^{-\bullet}$ in tetrahydrofuran at 4.2 K – 160.0 K.

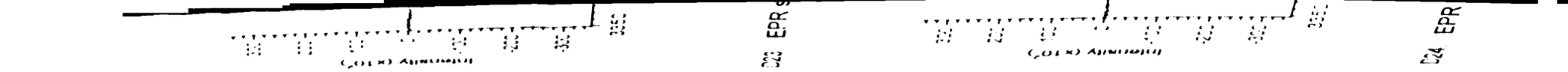
Intensity (x10²)
EPR s
Intensity (x10²)
EPR

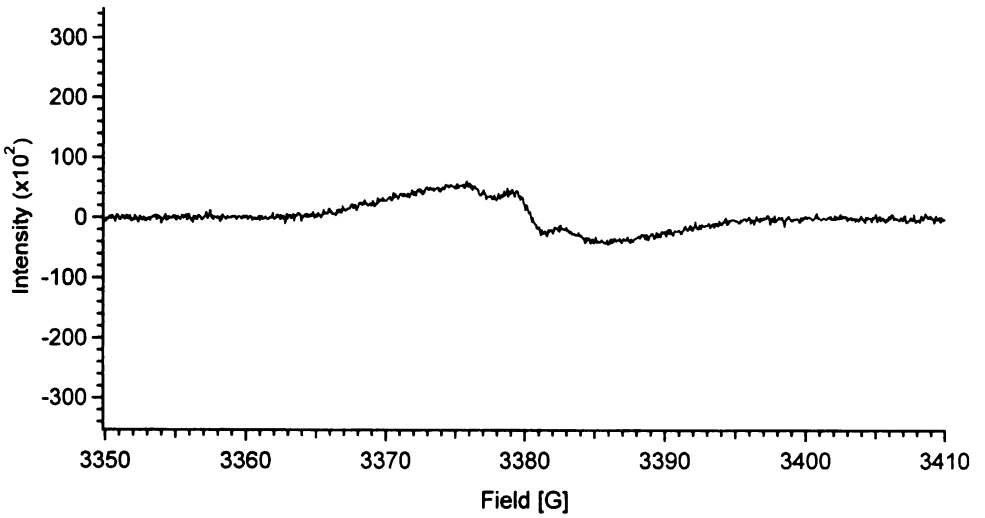


D21. EPR spectrum of $(Li^+)_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 4.1 K.

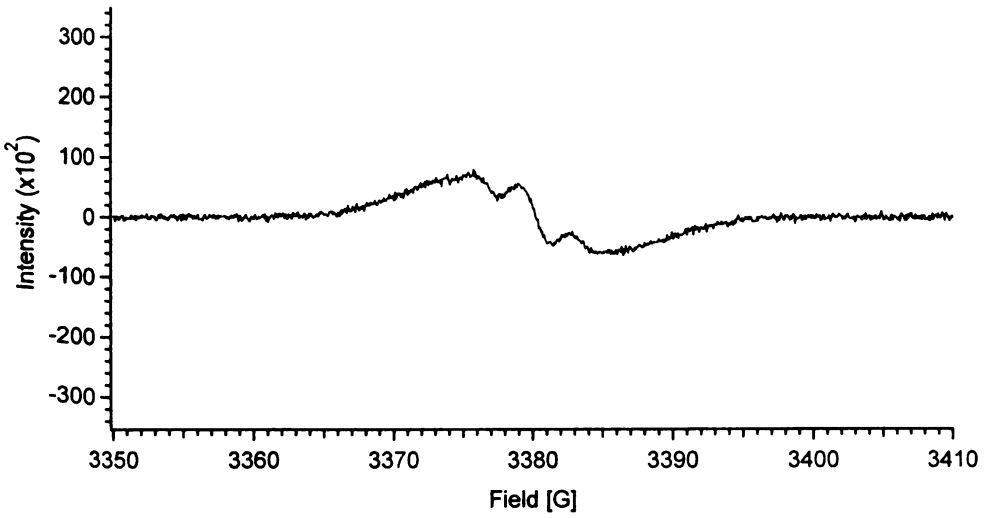


D22. EPR spectrum of $(Li^+)_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 13.0 K.


EPR
EPR



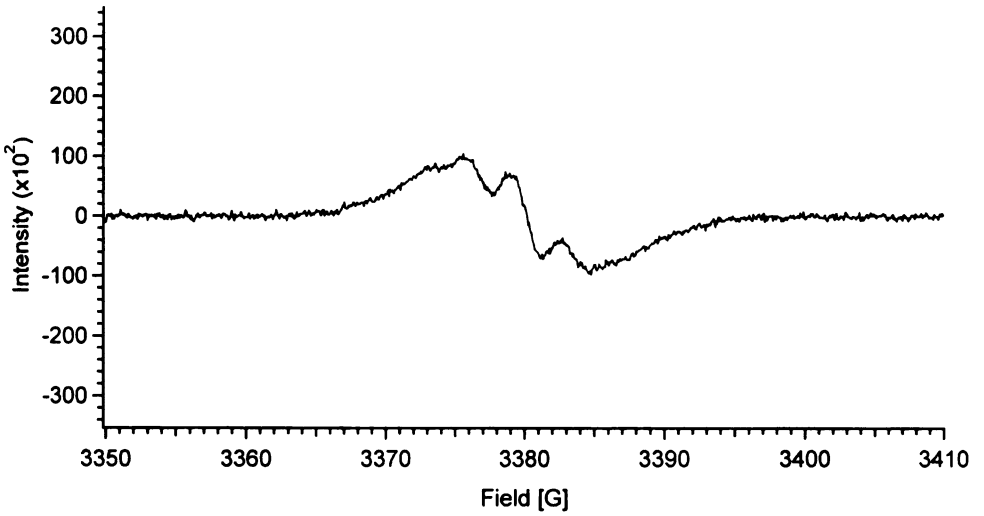
D23. EPR spectrum of $(Li^+)_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 20.0 K.



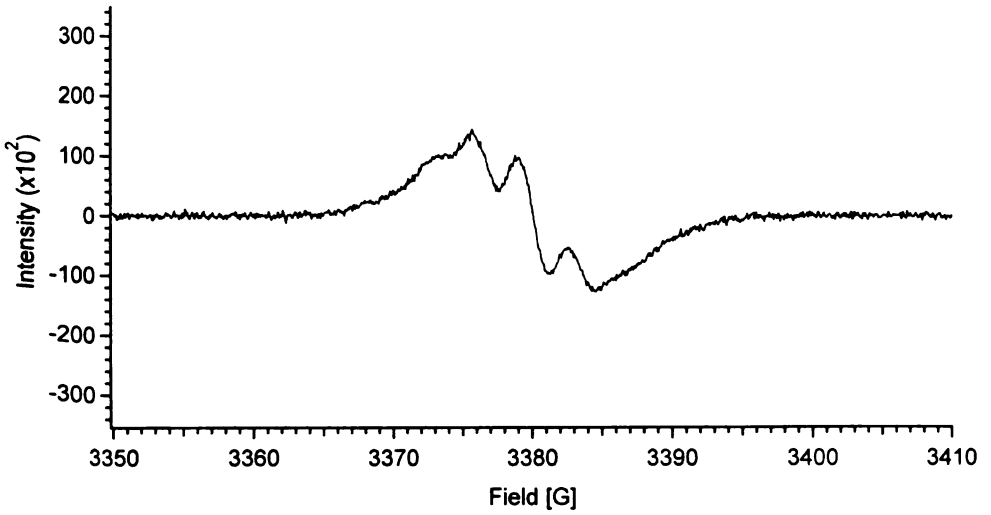
D24. EPR spectrum of $(Li^+)_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 30.0 K.

Intensity (x10³)
EPR

Intensity (x10³)
EPR



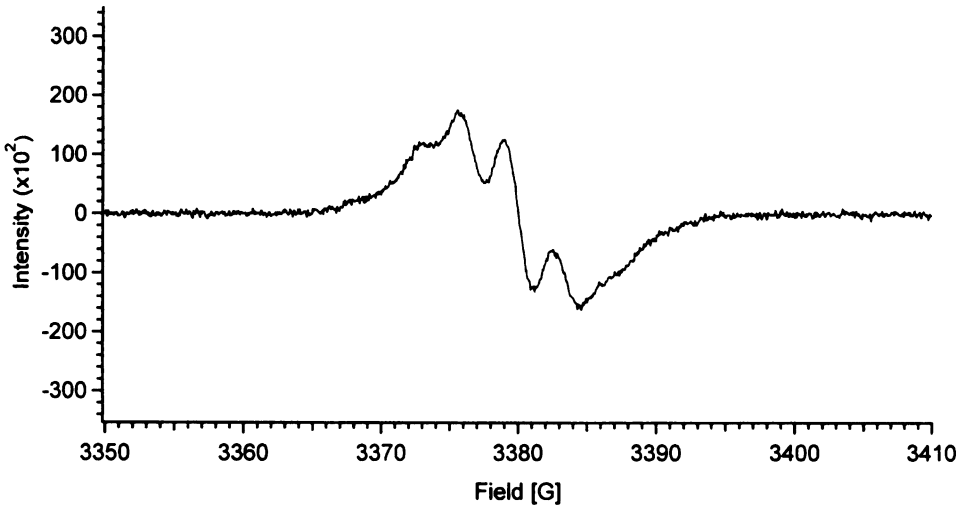
D25. EPR spectrum of $(Li^+)_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 40.0 K.



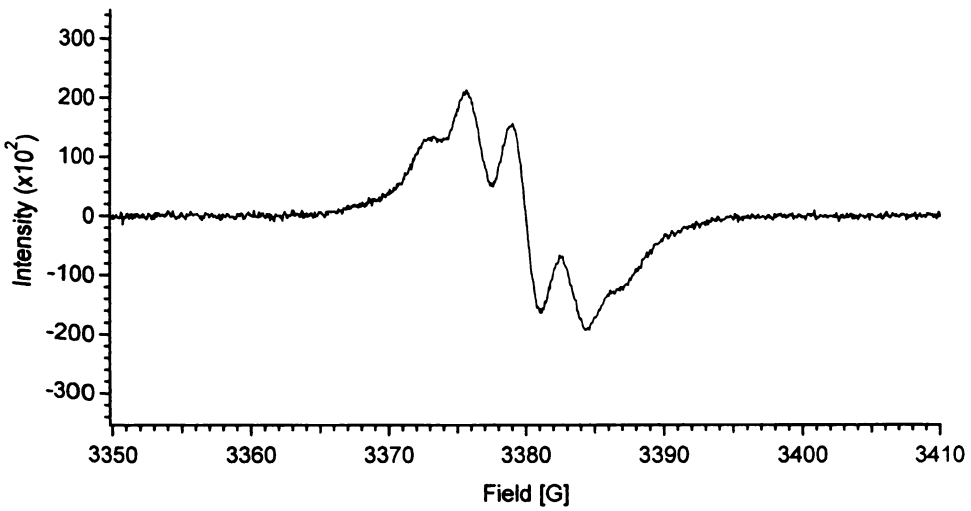
D26. EPR spectrum of $(Li^+)_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 50.0 K.

Intensity (x10²)
EPR

Intensity (x10²)
EPR

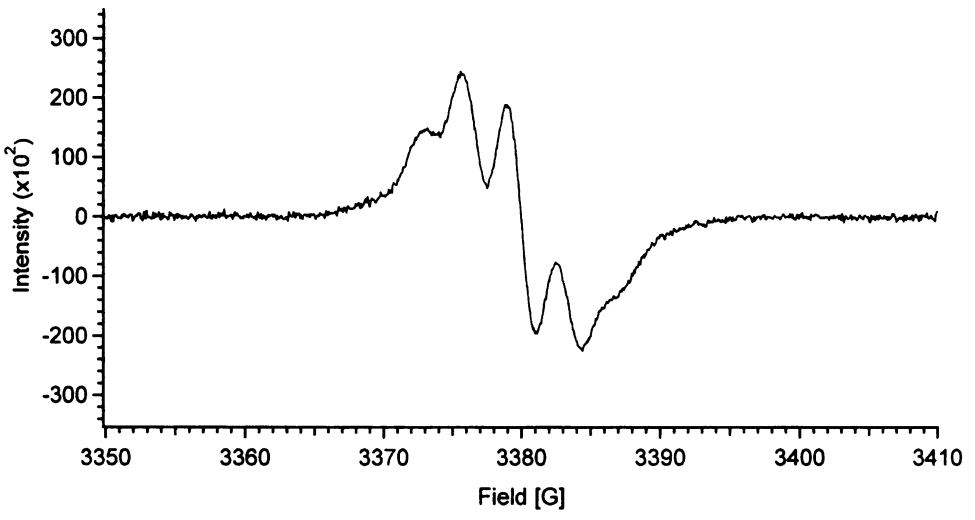


D27. EPR spectrum of $(Li^+)_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 60.0 K.

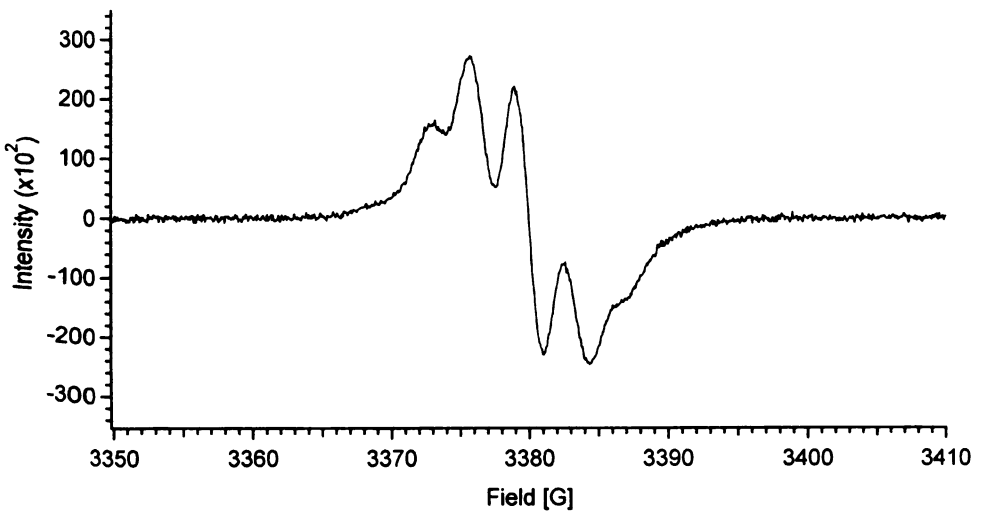


D28. EPR spectrum of $(Li^+)_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 70.0 K.

Intensity (x10⁵)
EPR
Intensity (x10⁵)
EPR



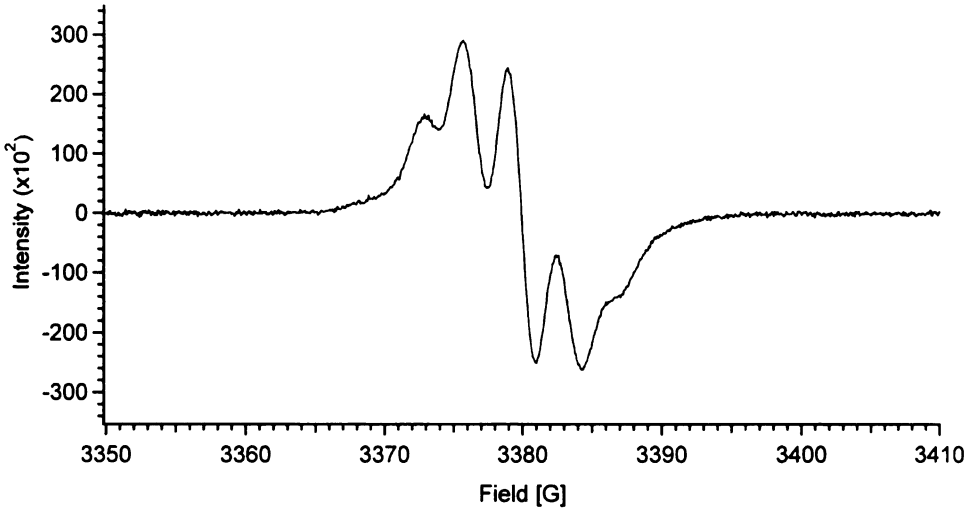
D29. EPR spectrum of $(Li^+)_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 80.0 K.



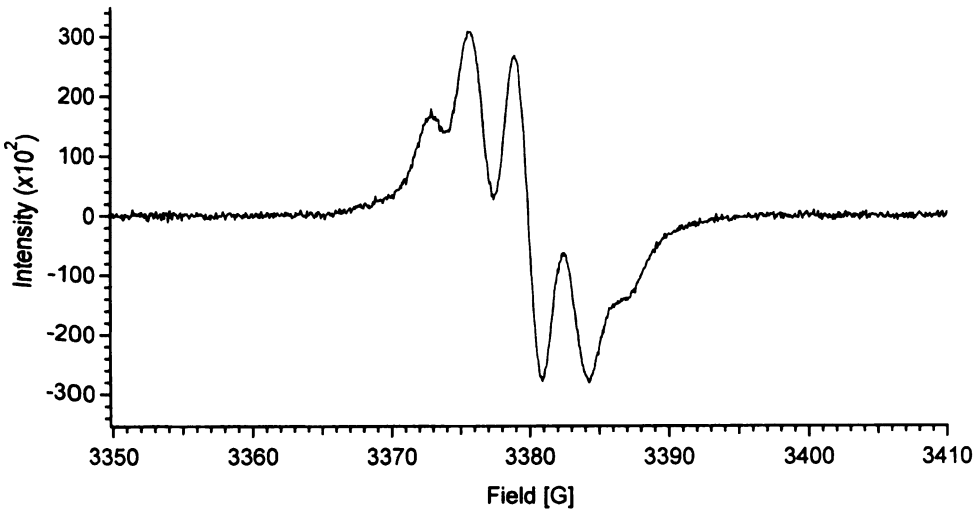
D30. EPR spectrum of $(Li^+)_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 90.0 K.

Intensity (x10⁴)
EPR

Intensity (x10⁴)
EPR



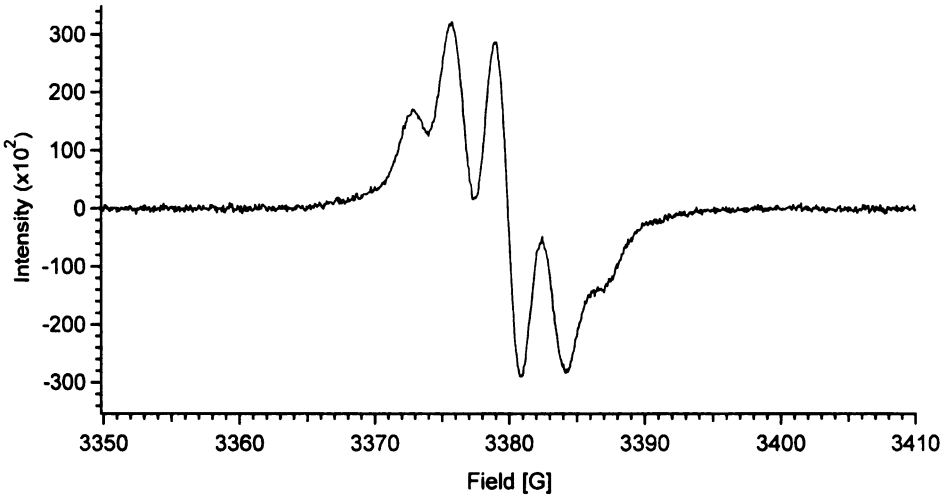
D31. EPR spectrum of $(Li^+)_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 100.0 K.



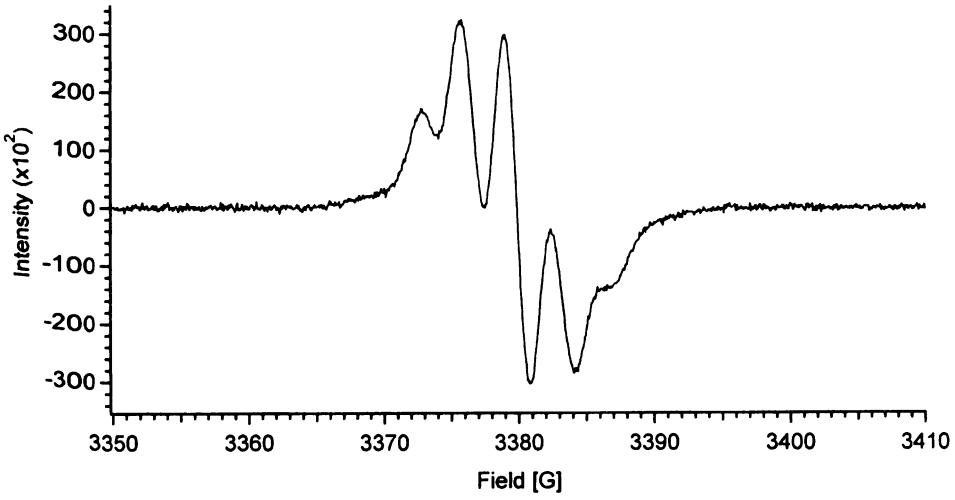
D32. EPR spectrum of $(Li^+)_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 110.0 K.

(a) EPR

(b) EPR



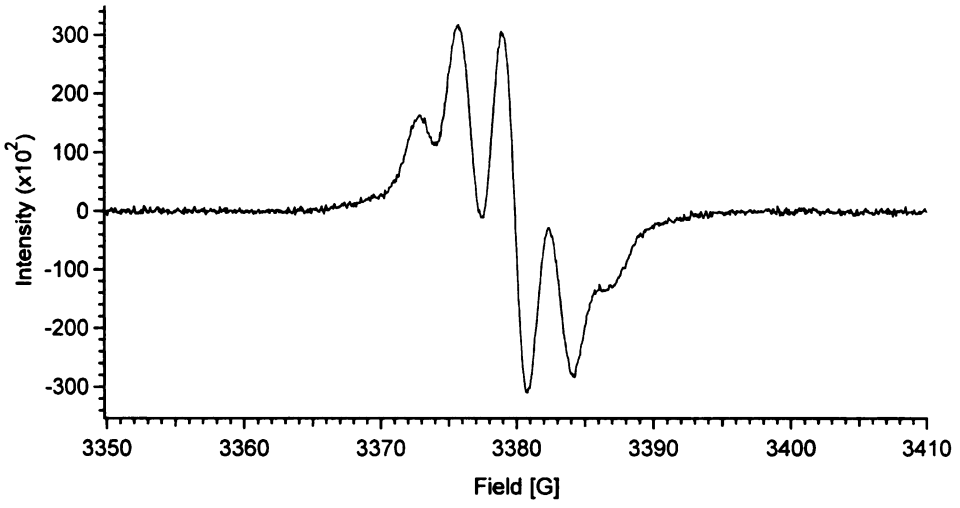
D33. EPR spectrum of $(Li^+)_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 120.0 K.



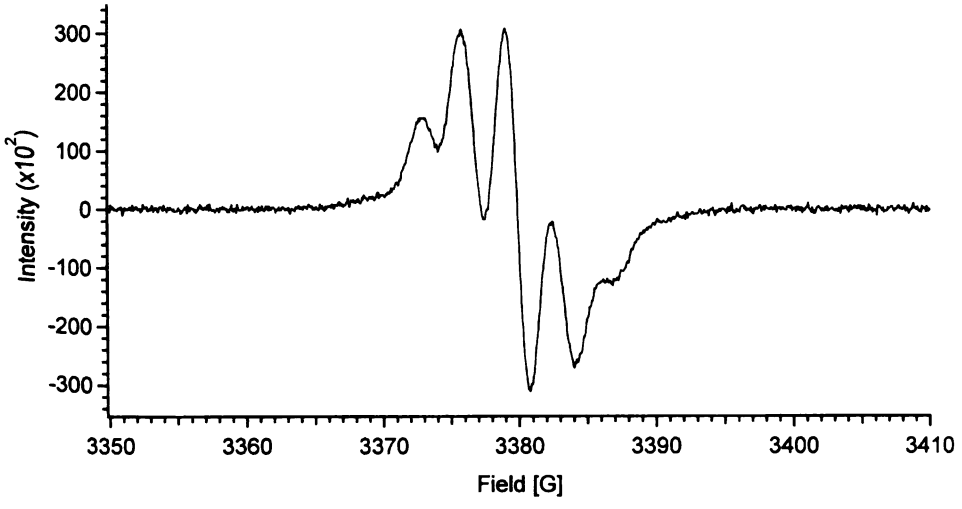
D34. EPR spectrum of $(Li^+)_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 130.0 K.

Intensity (x10²)
335
EPR

Intensity (x10²)
335
EPR



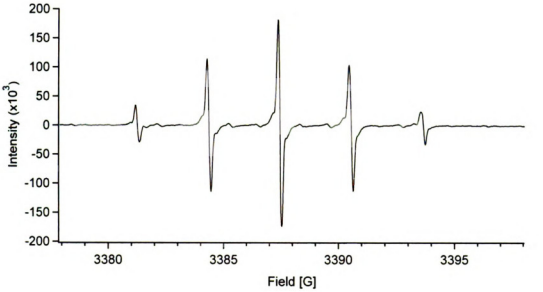
D35. EPR spectrum of $(Li^+)_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 140.0 K.



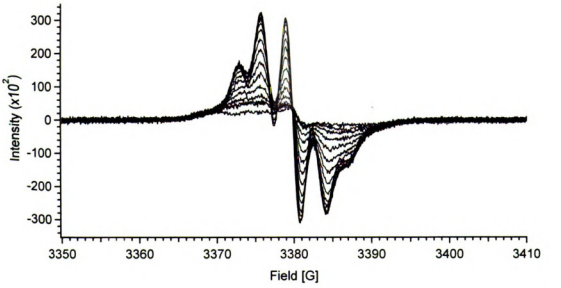
D36. EPR spectrum of $(Li^+)_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 150.0 K.

Intensity (x10⁴)
EPR

Intensity (x10⁴)
EPR

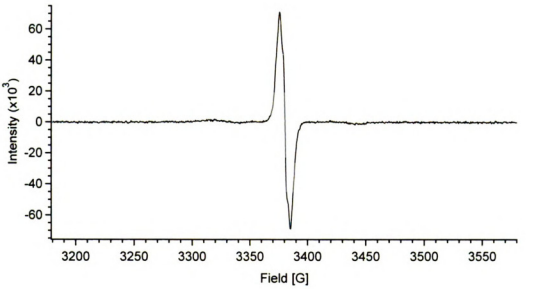


D37. EPR spectrum of $(Li^+)_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 293.0 K.

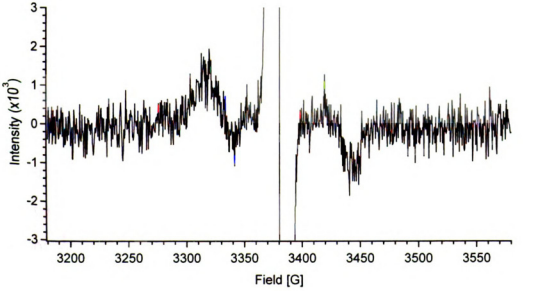


D38. EPR spectra of $(Li^+)_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran 4.1 K – 150.0 K.

Intensity (x10³)
EP
Intensity (x10³)
EP

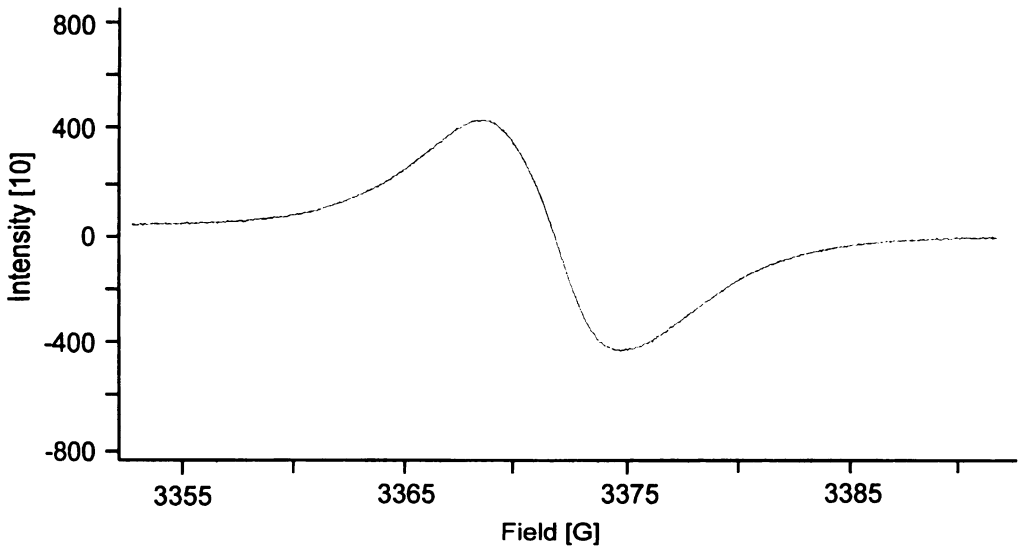


D39. EPR spectrum of $(Li^+)_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 100.0 K.

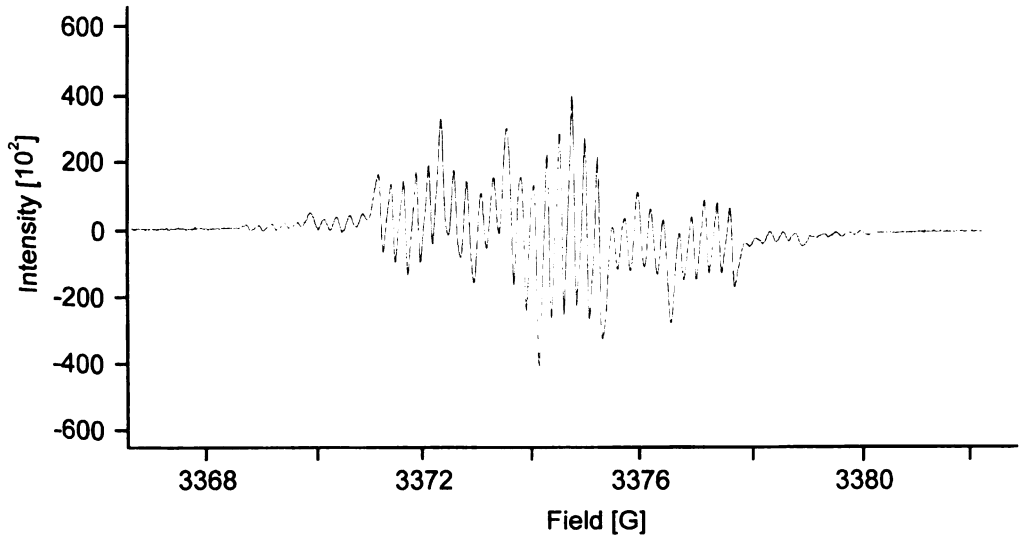


D40. EPR spectrum of $(Li^+)_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 100.0 K.

0
-400
-800
Left Amygdala
241. EP
600
400
200
0
-200
-400
-600
Left Amygdala
242. EP



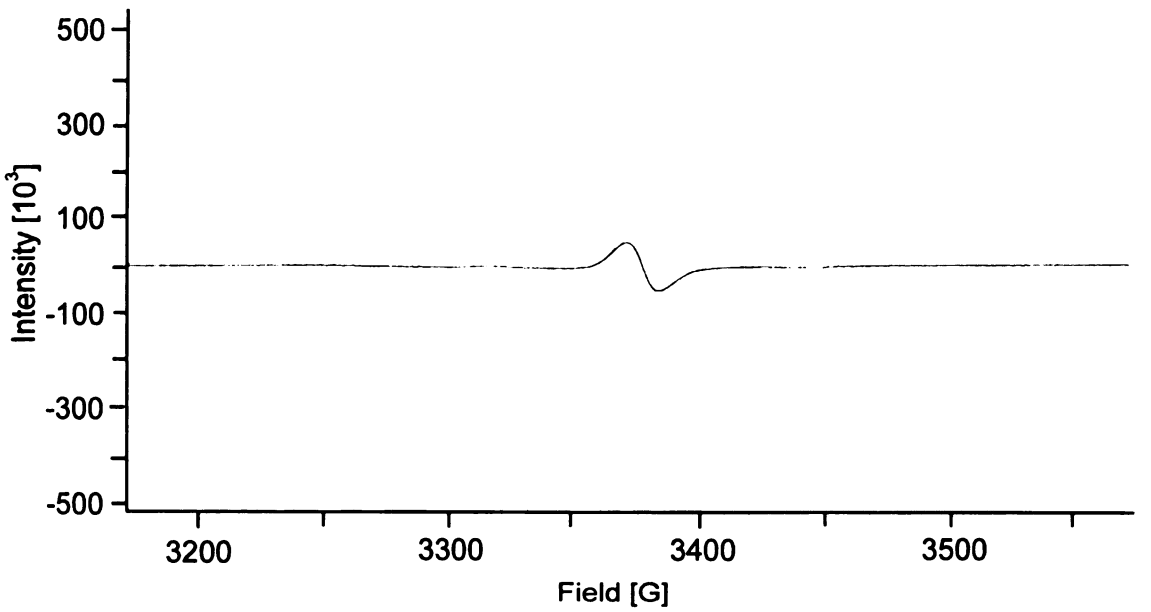
D41. EPR spectrum of $Cs^{+}•1E^{-•}$ in tetrahydrofuran at 5.0 K.



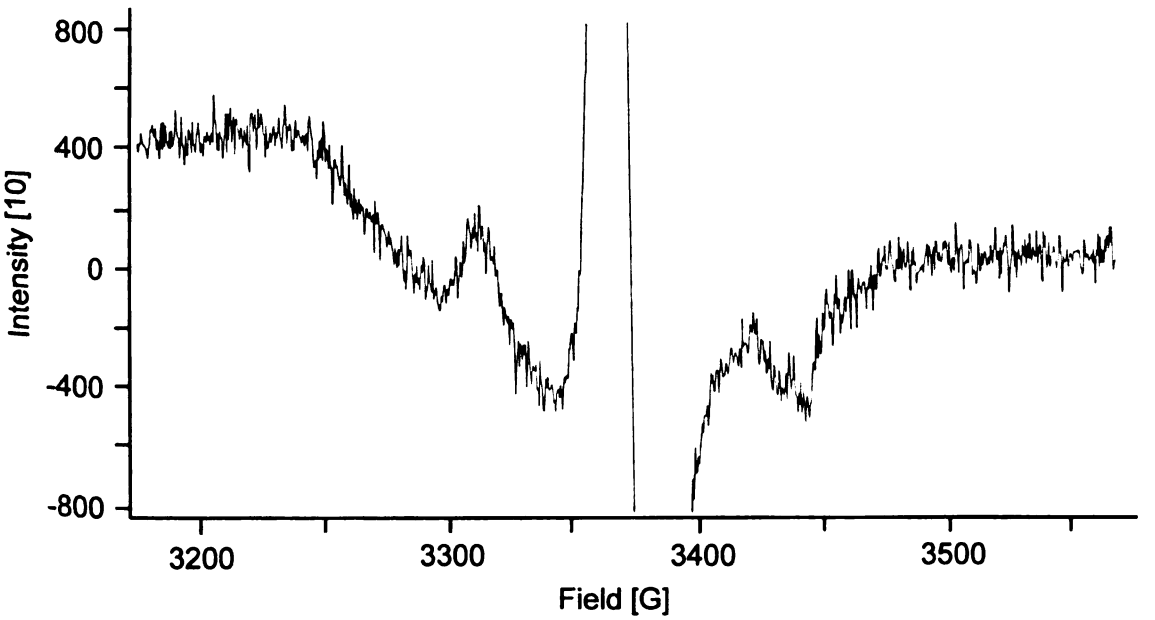
D42. EPR spectrum of $Cs^{+}•1E^{-•}$ in 2-methyltetrahydrofuran at 290.0 K.

Longitude (deg)
-50
-55
-60
-65
343 EP

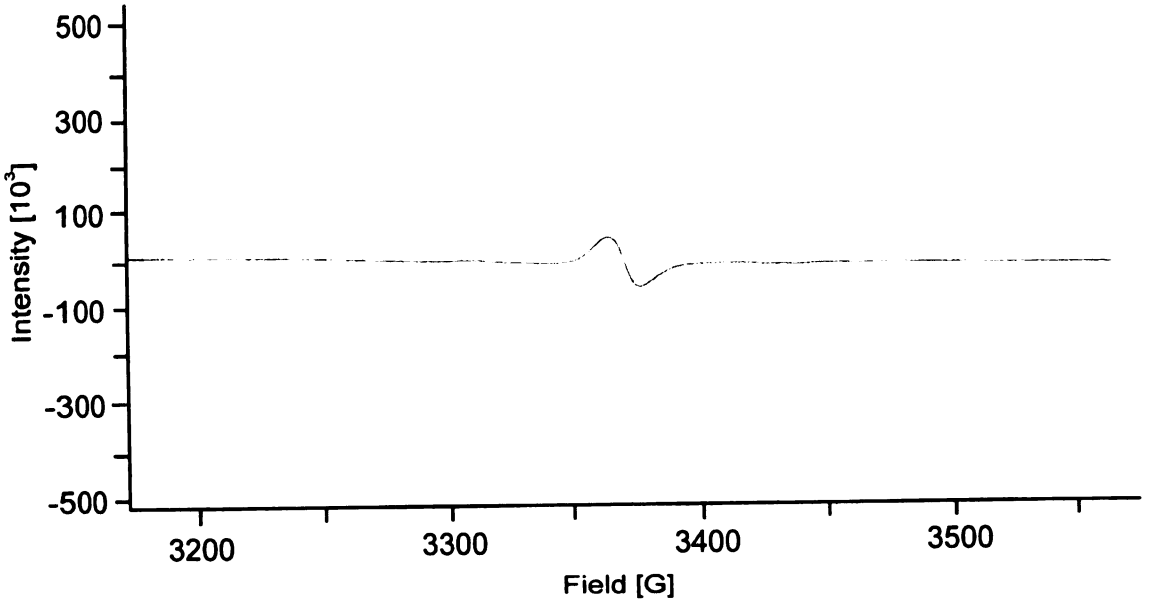
Longitude (deg)
-30
-35
-40
-45
344 EP



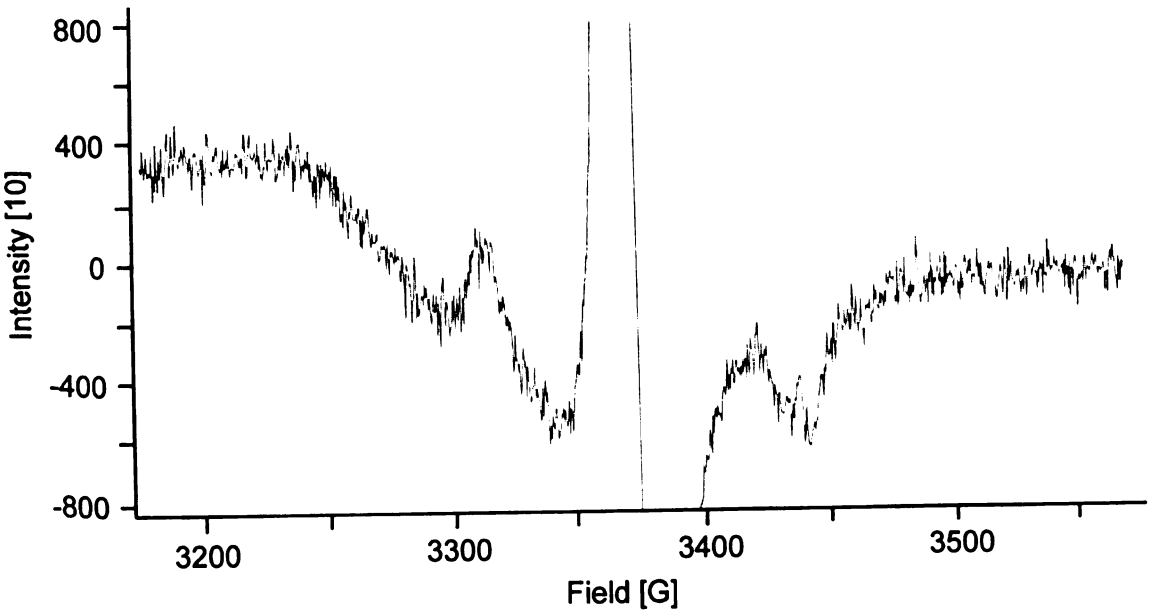
D43. EPR spectrum of $(Cs^{+})_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 4.7 K.



D44. EPR spectrum of $(Cs^{+})_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 4.7 K expanded.



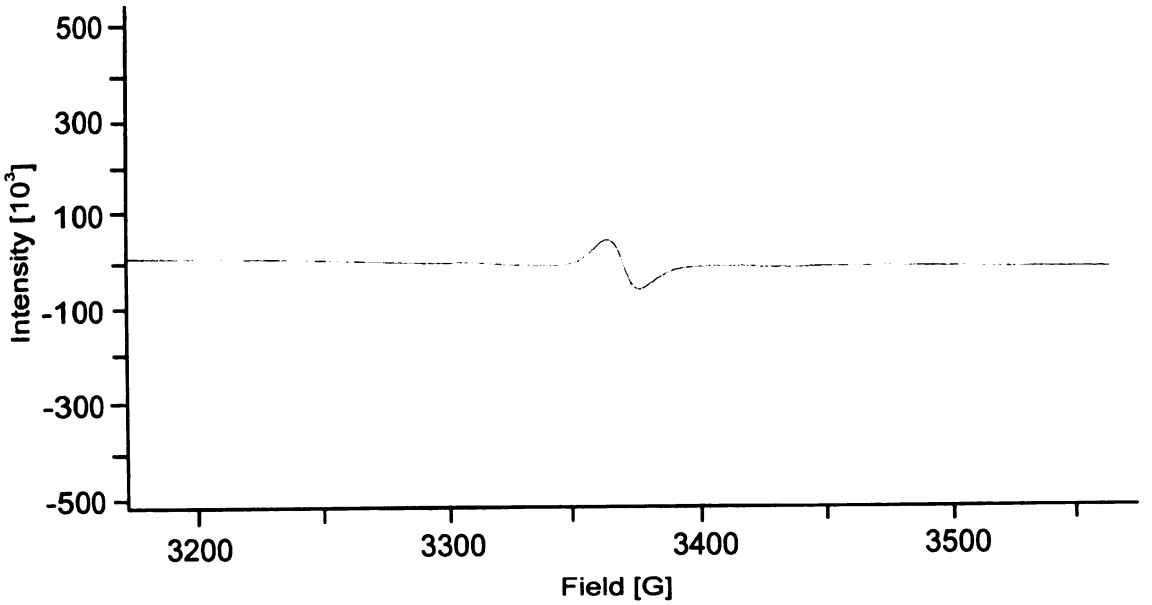
D45. EPR spectrum of $(Cs^{+})_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 5.3 K.



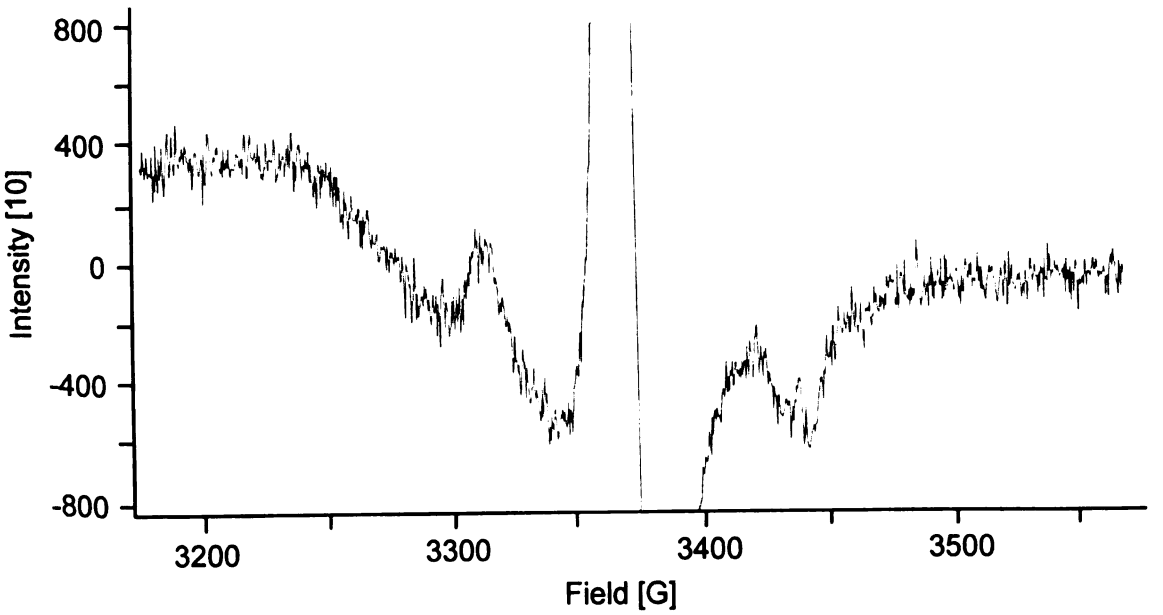
D46. EPR spectrum of $(Cs^{+})_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 5.3 K expanded.

Intensity (cps)
32
245 EP

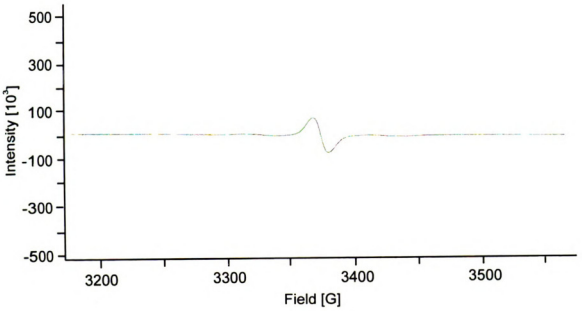
Intensity (cps)
246 EP



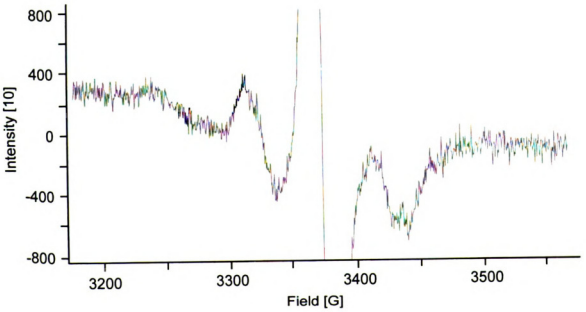
D45. EPR spectrum of $(Cs^{+})_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 5.3 K.



D46. EPR spectrum of $(Cs^{+})_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 5.3 K expanded.



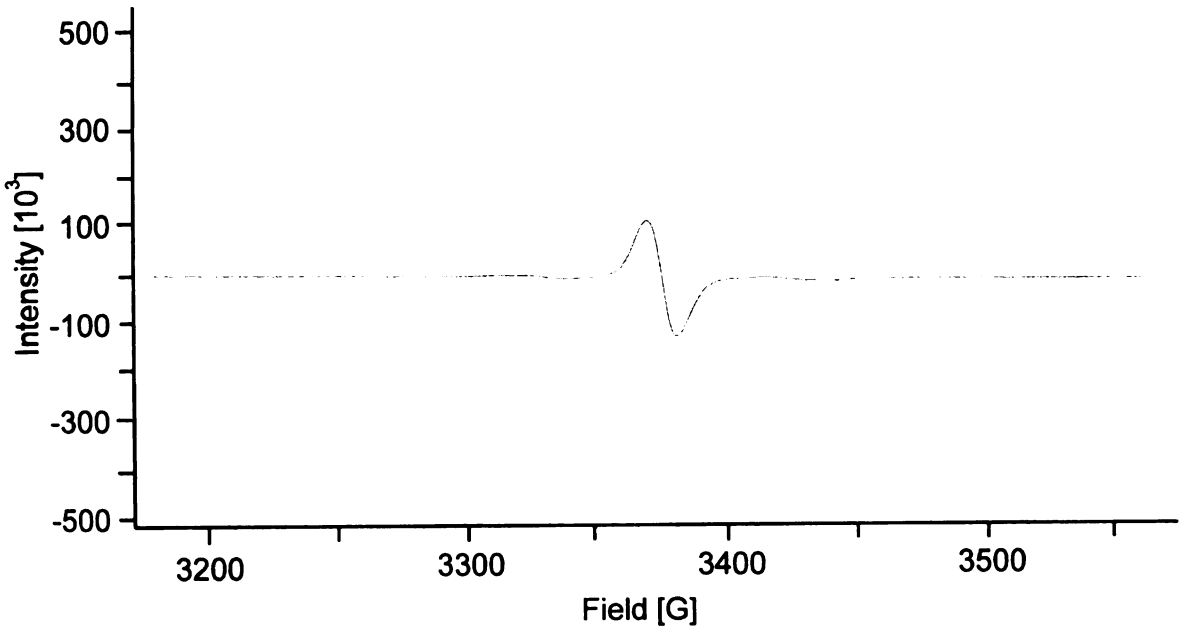
D47. EPR spectrum of $(Cs^{+})_2 \bullet 1E^{2-\bullet}$ in tetrahydrofuran at 13.0 K.



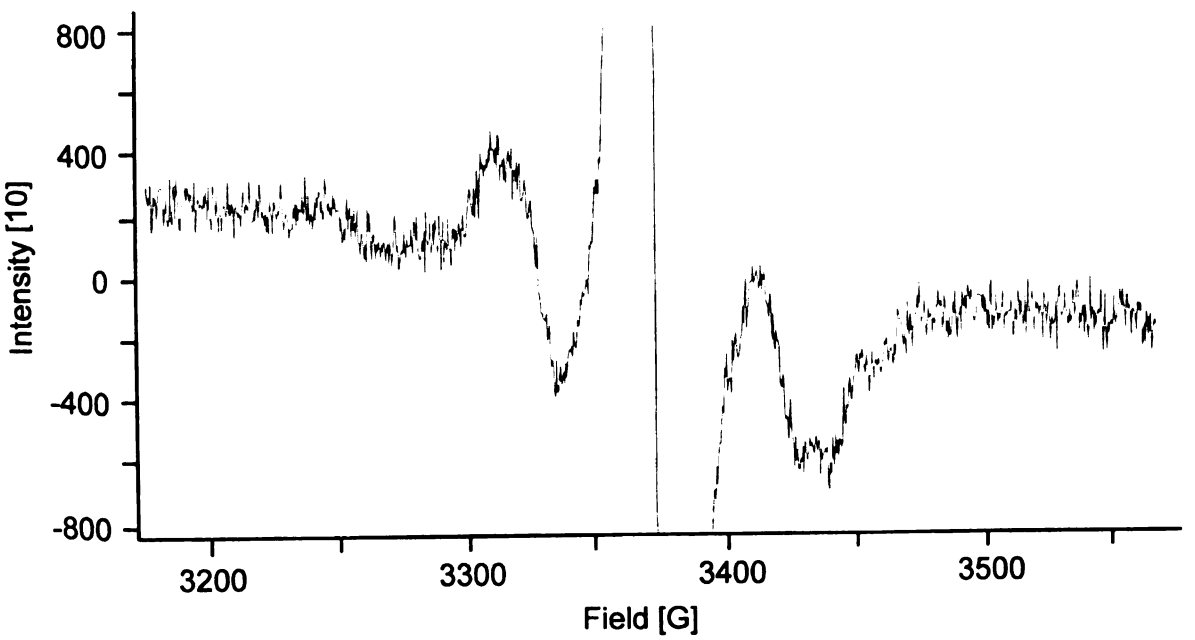
D48. EPR spectrum of $(Cs^{+})_2 \bullet 1E^{2-\bullet}$ in tetrahydrofuran at 13.0 K expanded.

Total Aluminum

Total Aluminum

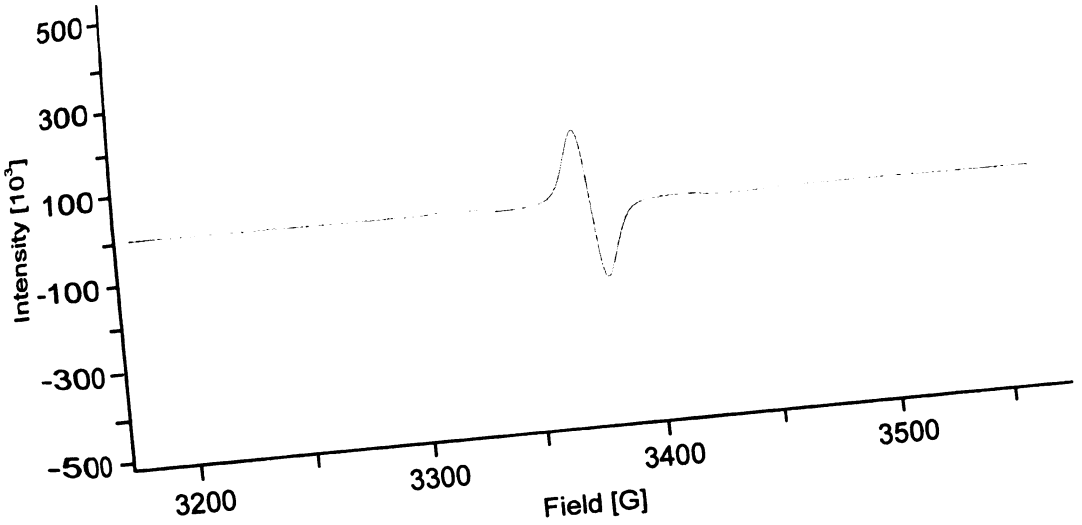


D49. EPR spectrum of $(Cs^{+})_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 25.0 K.

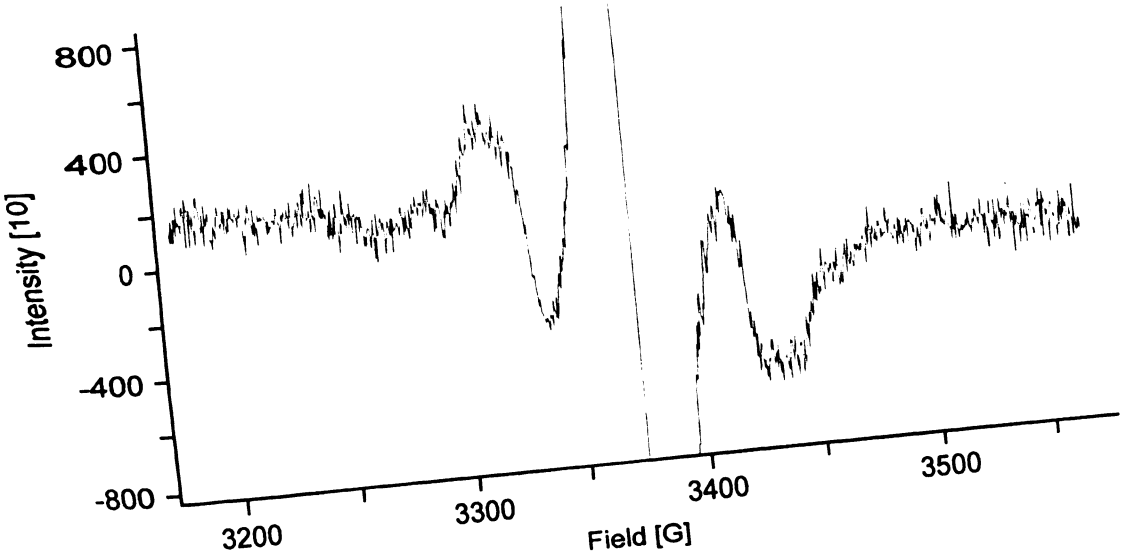


D50. EPR spectrum of $(Cs^{+})_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 25.0 K expanded.

Intensity [a.u.]
Intensity [a.u.]

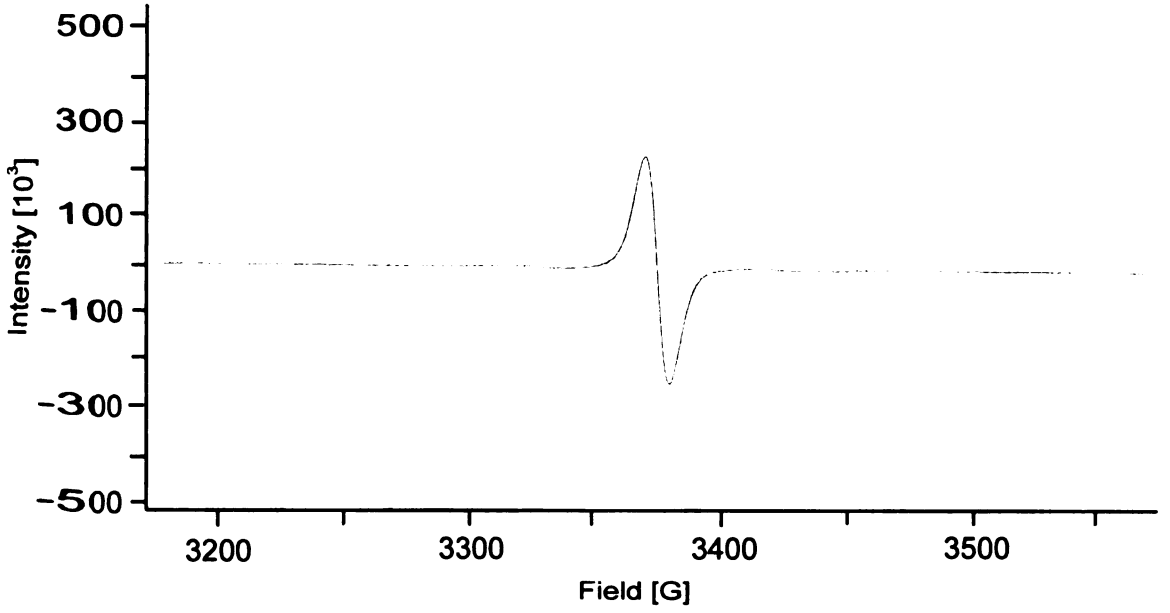


D51. EPR spectrum of $(Cs^{+})_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 37.0 K.

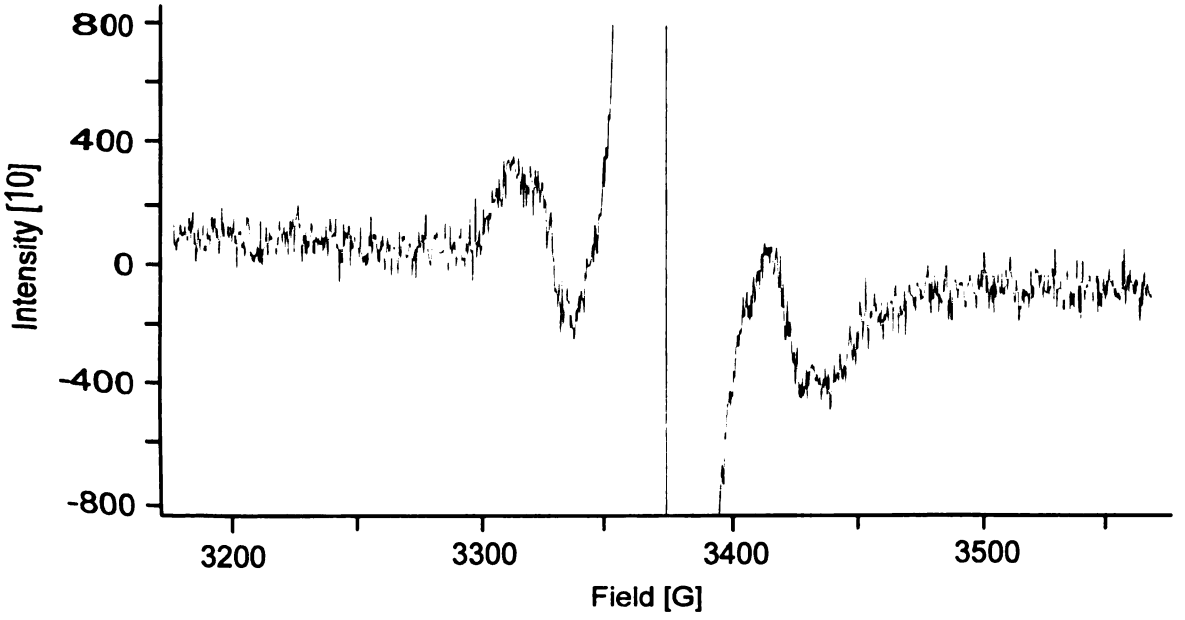


D52. EPR spectrum of $(Cs^{+})_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 37.0 K expanded.

Intensity [101]
Intensity [101]

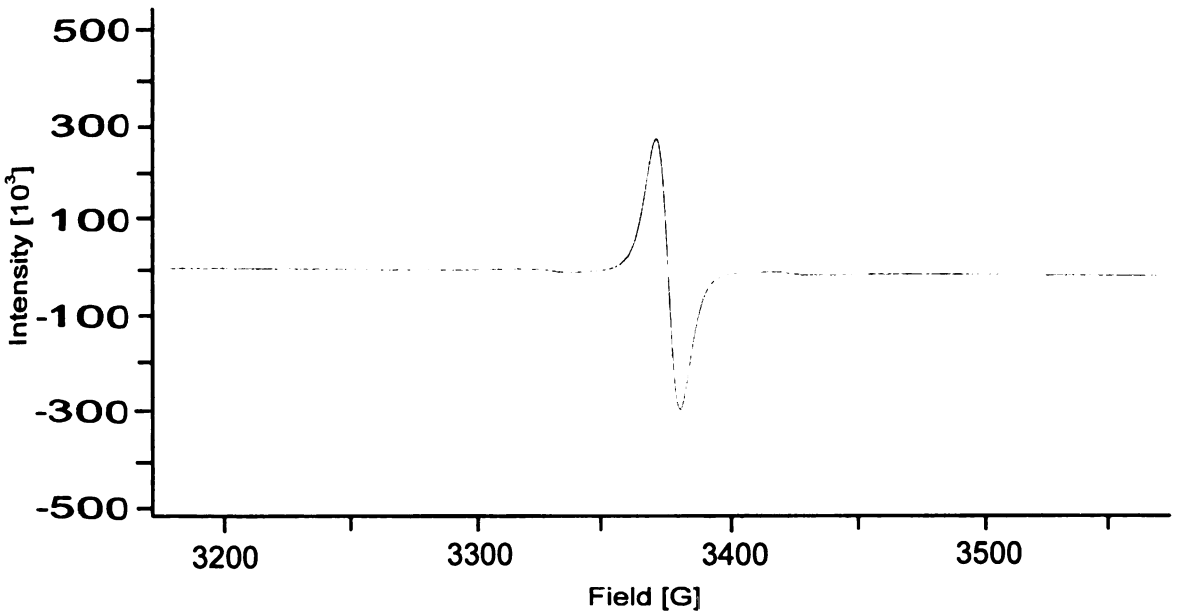


D53. EPR spectrum of $(Cs^+)_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 50.0 K.

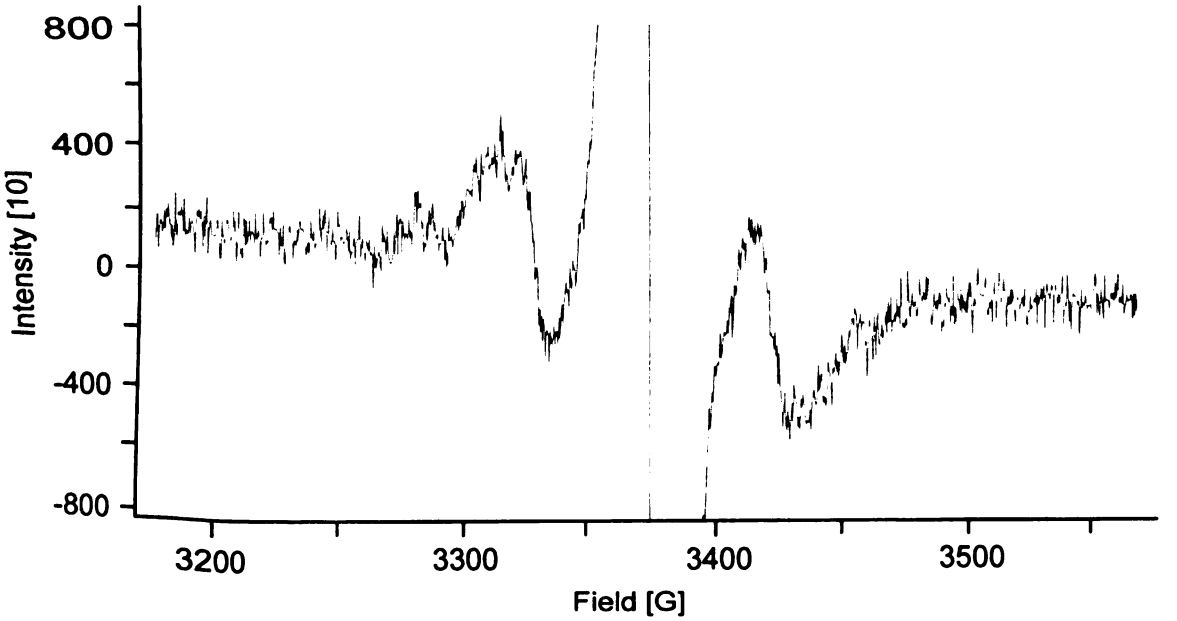


D54. EPR spectrum of $(Cs^+)_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 50.0 K expanded.

500
300
100
-100
-300
-500
Intensity [110]
55
E
300
100
0
-100
-300
Intensity [110]
55
E



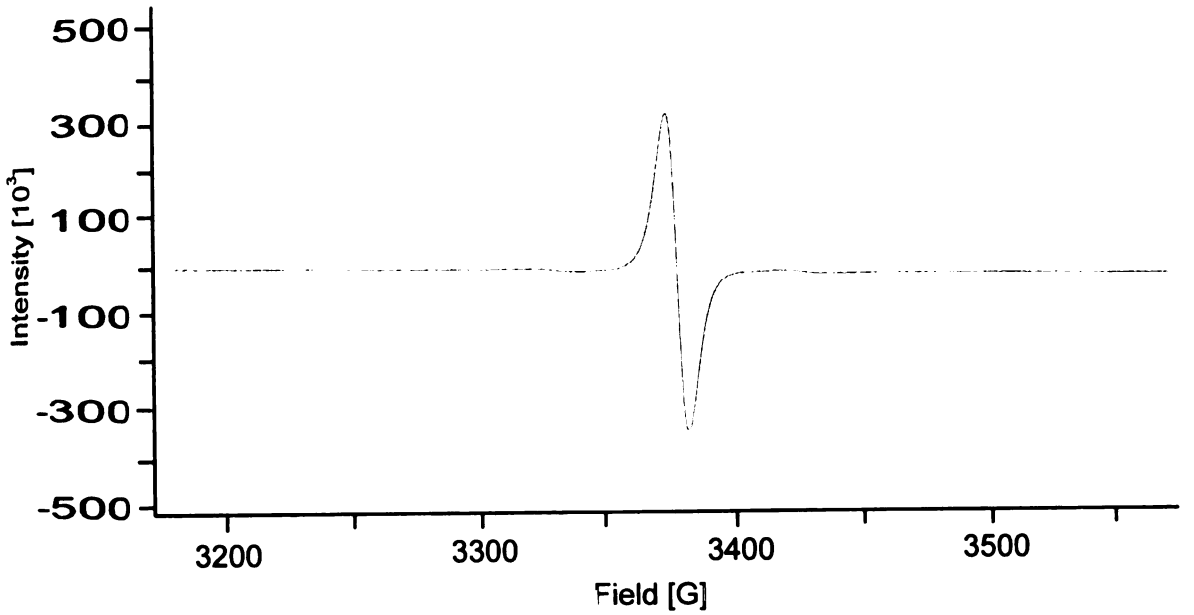
D55. EPR spectrum of $(Cs^+)_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 62.5 K.



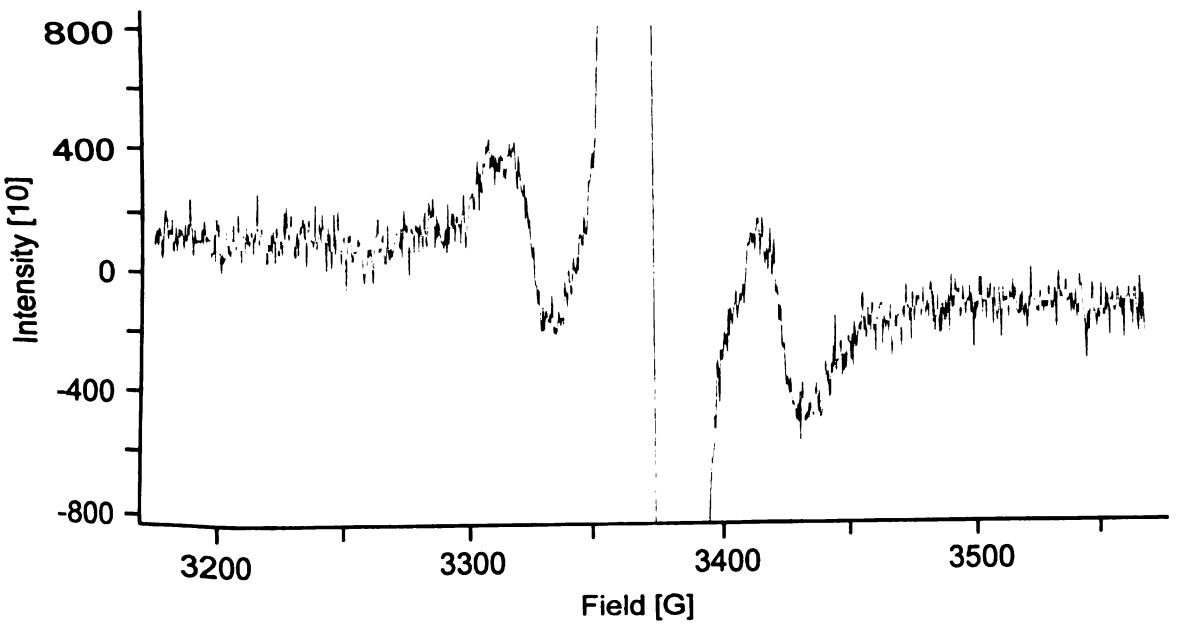
D56. EPR spectrum of $(Cs^+)_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 62.5 K expanded.

Aluminium
Foil (10)

Aluminium
Foil (10)

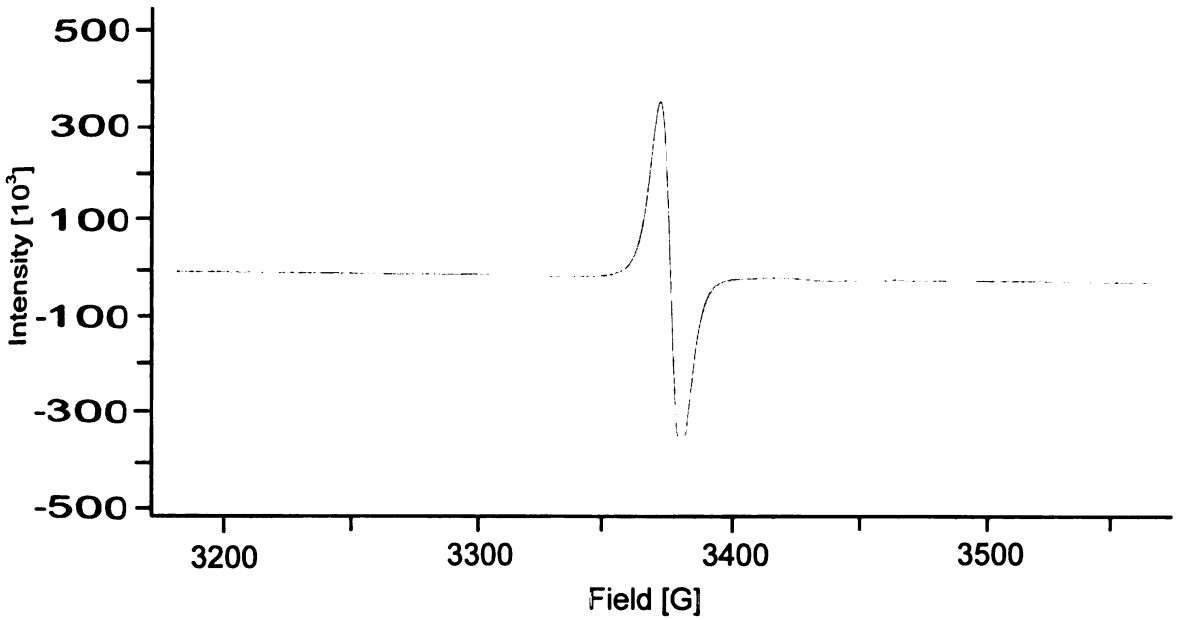


D57. EPR spectrum of $(Cs^{+})_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 75.0 K.

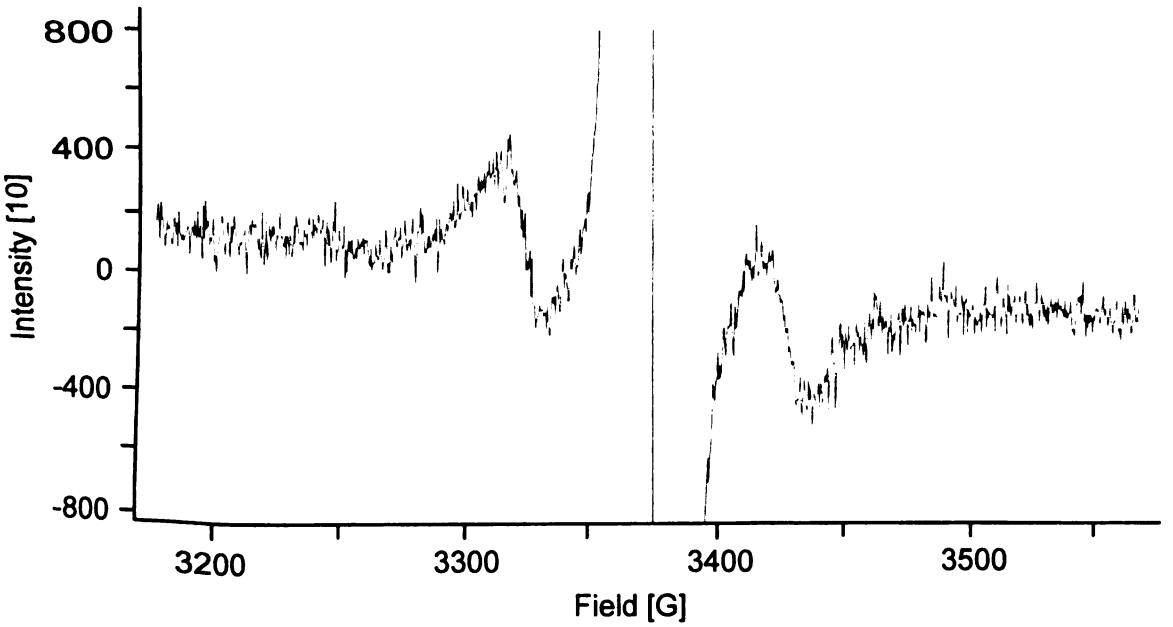


D58. EPR spectrum of $(Cs^{+})_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 75.0 K expanded.

Intensity (a.u.)
EPF
Intensity (a.u.)
EPP



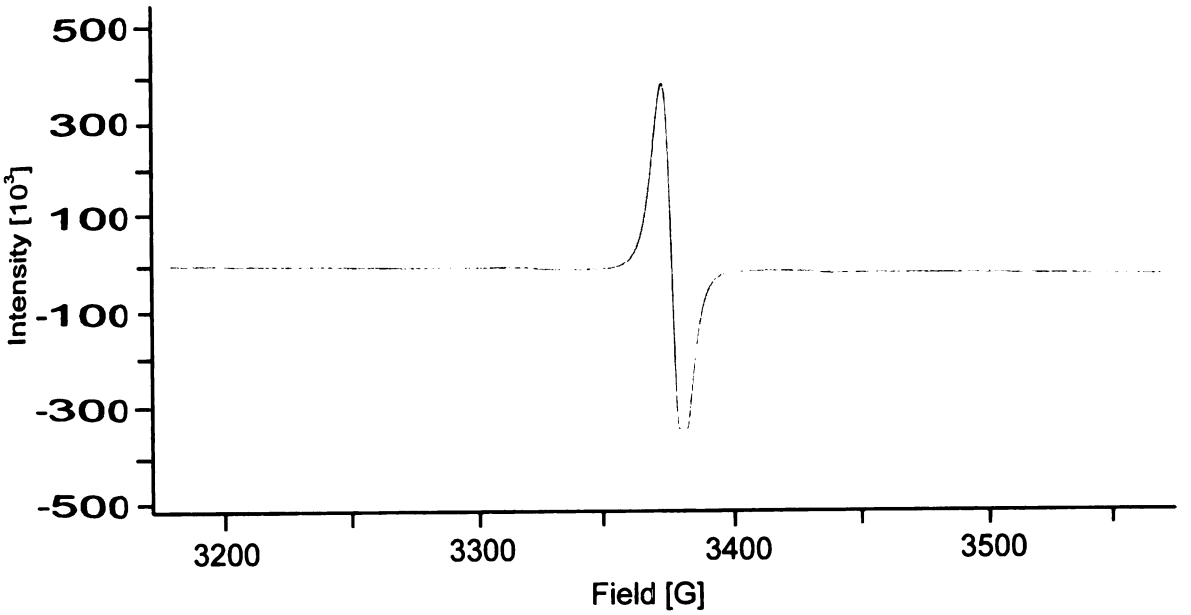
D59. EPR spectrum of $(Cs^{+})_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 87.5 K.



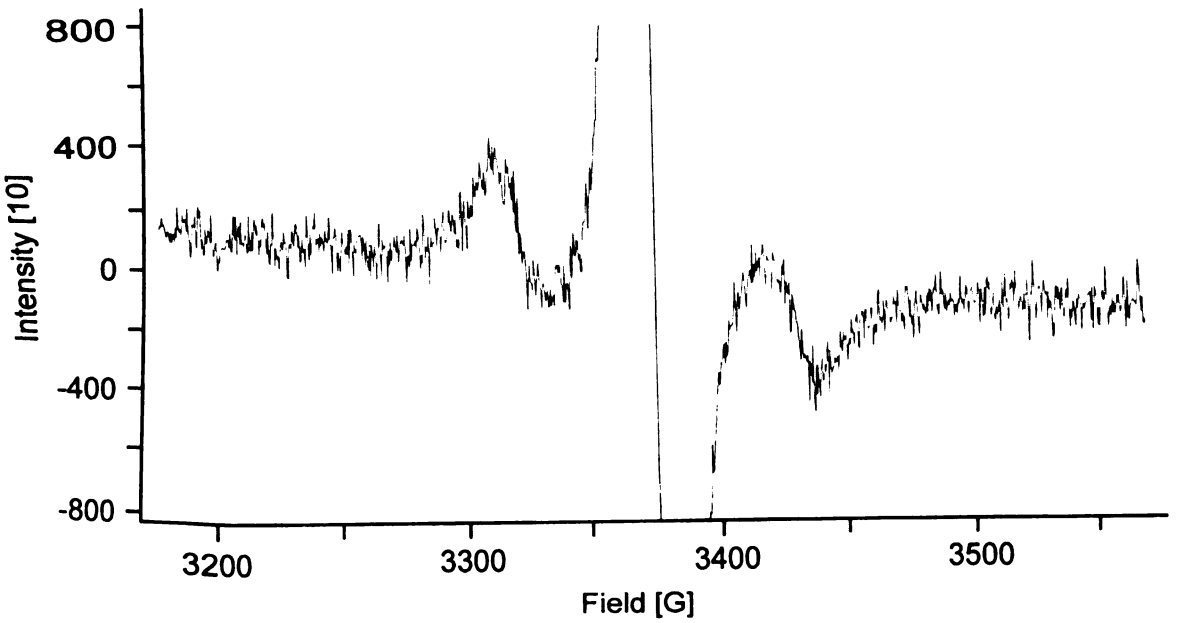
D60. EPR spectrum of $(Cs^{+})_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 87.5 K expanded.

Intensity [101]
362 EP

Intensity [101]
362 EP

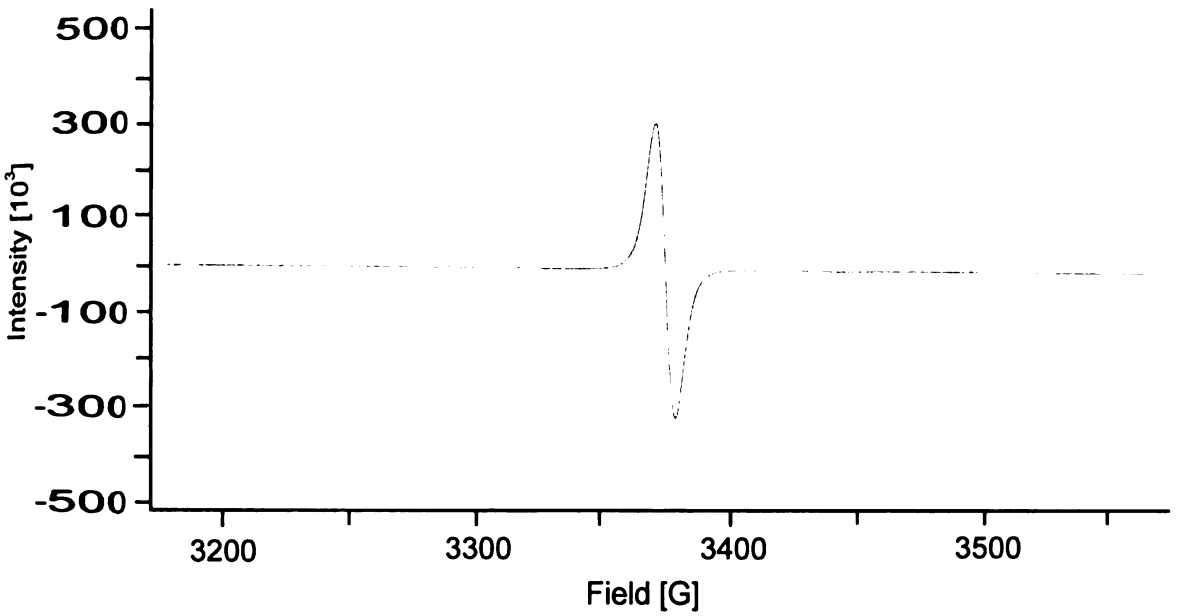


D61. EPR spectrum of $(Cs^{+})_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 100.0 K.

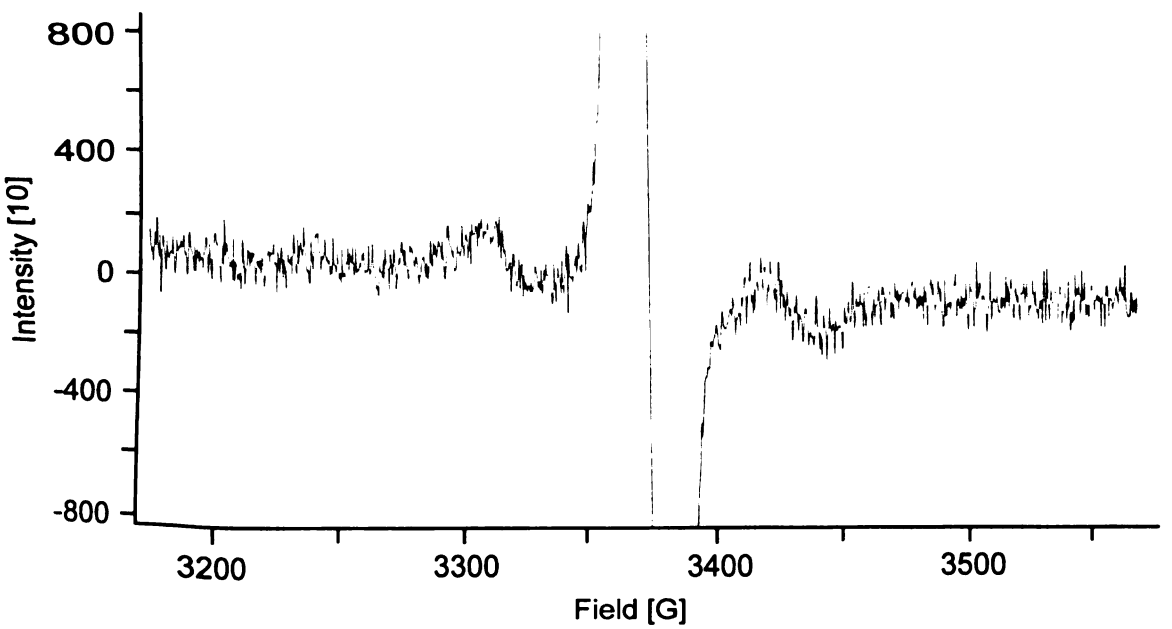


D62. EPR spectrum of $(Cs^{+})_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 100.0 K expanded.

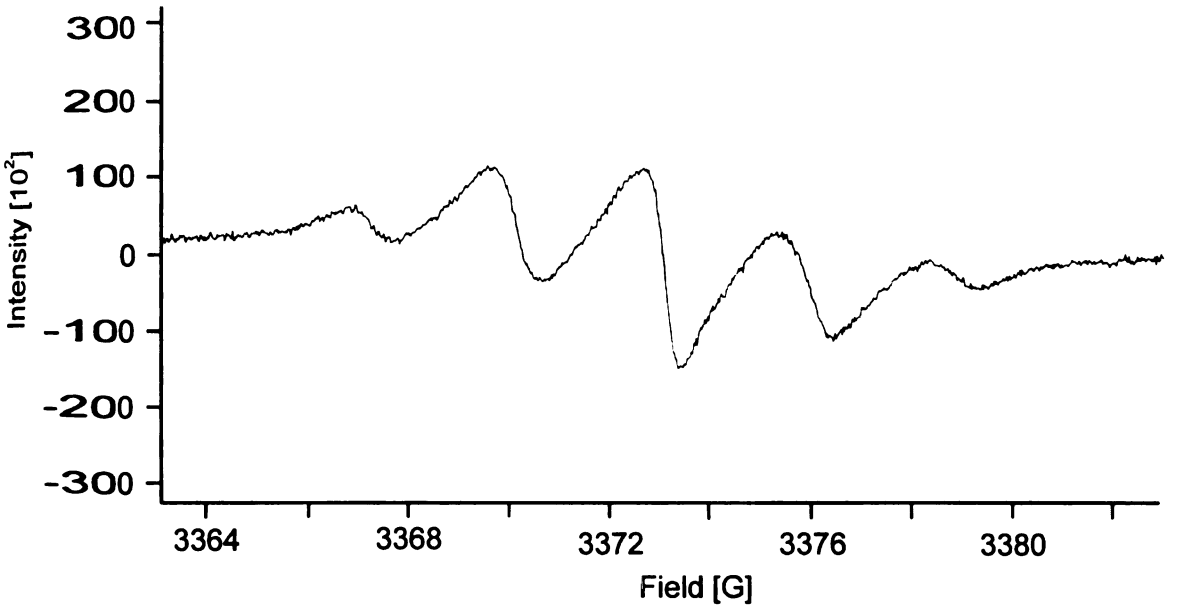
Azimuth
Azimuth



D63. EPR spectrum of $(Cs^{+})_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 120.0 K.



D64. EPR spectrum of $(Cs^{+})_2 \bullet 1E^{2-\bullet\bullet}$ in tetrahydrofuran at 120.0 K expanded.



D65. EPR spectrum of $(Cs^{+})_2 \bullet 1E^{2-\bullet}$ in tetrahydrofuran at 294.0 K.

Table E1

Identifica	
Empirica	
Formula	
Tempera	
Wavelen	
Crystal s	
Space gr	
Unit cel	
Volume	
Z	
Density	
Absorpti	
F(000)	
Crystal S	
Color	
Theta ra	
Index ra	
Reflecto	
Refinem	
Data / R	
Goodnes	
Final R i	
R indices	
Extinctio	
Largest	

APPENDIX E

Crystallographic Data

Table E1. Crystal and structure refinement data for **7**.

Identification code	**7**
Empirical formula	$C_{74}H_{116}O_{11}$
Formula weight	1181.73 g/mole
Temperature	173(2) K
Wavelength	0.71073 Å
Crystal system	Orthorhombic
Space group	P_{bcn}
Unit cell dimensions	a = 18.4052(37) Å
	b = 15.0498(30) Å
	c = 26.2313(52) Å
	α = 90.000°
	β = 90.000°
	γ = 90.000°
Volume	7265.93 $Å^3$
Z	4
Density (calculated)	1.137 Mg/m^3
Absorption coefficient	0.075 mm^{-1}
F(000)	2712
Crystal Size	0.18 mm x 0.18 mm x 0.30 mm
Color	lemon yellow
Theta range for data collection	1.55° to 28.44°
Index ranges	$-24 \le h \le 24$, $-20 \le k \le 20$, $-35 \le l \le 34$
Reflections collected / Unique	84282 / 9008 [R_{int} = 0.0998]
Refinement method	Full-matrix-least-squares on F^2
Data / Restraints / Parameters	9008 / 0 / 453
Goodness-of-fit on F^2	1.415
Final R indices [$I>2\sigma(I)$]	R_1 = 0.0986, wR_2 = 0.2575
R indices (all data)	R_1 = 0.2141, wR_2 = 0.2966
Extinction Coefficient	0.001225
Largest diff. peak and hole	0.412 and –0.607 e/A^3

Table E2. Atomic coordinates and equivalent isotropic displacement parameters ($Å^2$) for **7**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U_{eq}
C1	0.04282(17)	0.1791(2)	0.24867(12)	0.0522(8)
O1	0.07174(13)	0.25923(16)	0.23393(10)	0.0642(7)
O2	0.05723(12)	0.11364(15)	0.21058(8)	0.0562(6)
C3	-0.00118(17)	0.1041(2)	0.17774(12)	0.0531(8)
O3	0.00504(13)	0.05629(16)	0.14139(9)	0.0672(7)
C4	-0.06481(18)	0.1504(2)	0.19914(12)	0.0515(8)
C9	-0.12817(17)	0.1650(2)	0.17391(11)	0.0511(8)
C11	-0.19530(17)	0.1911(2)	0.20050(12)	0.0505(8)
C12	-0.21079(17)	0.1586(2)	0.24879(12)	0.0517(8)
C13	-0.27111(18)	0.1849(2)	0.27679(12)	0.0537(9)
C14	-0.31633(18)	0.2490(2)	0.25371(12)	0.0539(8)
O14	-0.37538(12)	0.28241(17)	0.28032(9)	0.0666(7)
C15	-0.30720(18)	0.2771(2)	0.20275(13)	0.0548(9)
C16	-0.24584(18)	0.2470(2)	0.17812(12)	0.0530(8)
C17	-0.2824(2)	0.1435(2)	0.33040(13)	0.0638(10)
C18	-0.2476(3)	0.0515(3)	0.33193(16)	0.0838(13)
C19	-0.2442(3)	0.2006(3)	0.37045(15)	0.0937(14)
C20	-0.3626(2)	0.1287(4)	0.34292(18)	0.0912(14)
C21	-0.36168(19)	0.3378(2)	0.17493(14)	0.0655(10)
C22	-0.3593(4)	0.4311(3)	0.1973(3)	0.129(2)
C23	-0.4376(2)	0.2976(4)	0.1764(2)	0.117(2)
C24	-0.3427(3)	0.3482(4)	0.11936(16)	0.1027(16)
C25	-0.3604(3)	0.3538(3)	0.31481(19)	0.0891(13)
C31	-0.13061(17)	0.1577(2)	0.11770(12)	0.0509(8)
C32	-0.18592(18)	0.1132(2)	0.09259(12)	0.0550(9)
C33	-0.18720(18)	0.1036(2)	0.03980(12)	0.0549(9)
C34	-0.12823(19)	0.1411(2)	0.01256(12)	0.0591(9)
O34	-0.12451(14)	0.12358(19)	-0.03899(8)	0.0746(8)
C35	-0.07727(18)	0.1963(2)	0.03598(13)	0.0598(9)
C36	-0.07873(18)	0.2007(2)	0.08850(13)	0.0582(9)
C37	-0.2521(2)	0.0590(3)	0.01316(13)	0.0669(10)
C38	-0.3144(2)	0.0431(3)	0.05034(16)	0.0834(13)
C39	-0.2811(3)	0.1183(4)	-0.02995(17)	0.0917(14)
C40	-0.2306(3)	-0.0326(3)	-0.00833(19)	0.0896(13)
C41	-0.0198(2)	0.2540(3)	0.00800(15)	0.0730(11)
C42	-0.0387(3)	0.2756(5)	-0.04697(19)	0.122(2)
C43	0.0537(3)	0.2068(4)	0.0102(3)	0.1197(19)
C44	-0.0090(4)	0.3411(4)	0.0362(2)	0.121(2)
C45	-0.0733(3)	0.0557(3)	-0.05319(17)	0.0908(14)

Table E2. continued...

C51	0.0516(4)	0.2845(5)	0.1898(3)	0.153(2)
C61	-0.3624(5)	-0.0567(6)	0.2000(4)	0.225(5)
O62	-0.4251(5)	-0.1050(4)	0.1971(3)	0.202(3)
C63	-0.4790(6)	-0.0629(8)	0.1743(3)	0.201(5)
C64	-0.4523(5)	0.0267(7)	0.1607(4)	0.194(4)
C65	-0.3811(4)	0.0315(5)	0.1903(2)	0.135(2)

Table E3. Bond Lengths (Å) and angles (°) for **7**.

Atoms	Length	Angle
C1-O1	1.373(4)	
C1-O2	1.428(4)	
C1-C4'	1.492(4)	
C1-C1'	1.578(6)	
O1-C51	1.274(8)	
O2-C3	1.385(4)	
C3-O3	1.200(4)	
C3-C4	1.474(5)	
C4-C9	1.359(4)	
C4-C1'	1.492(4)	
C9-C11	1.472(4)	
C9-C31	1.479(4)	
C11-C16	1.385(4)	
C11-C12	1.388(4)	
C12-C13	1.389(4)	
C12-H12A	0.9300	
C13-C14	1.411(5)	
C13-C17	1.552(5)	
C14-O14	1.386(4)	
C14-C15	1.412(5)	
O14-C25	1.431(5)	
C15-C16	1.377(4)	
C15-C21	1.540(5)	
C16-H16A	0.9300	
C17-C18	1.526(5)	
C17-C19	1.528(5)	
C17-C20	1.528(5)	
C18-H18A	0.9600	
C18-H18B	0.9600	
C18-H18C	0.9600	
C19-H19A	0.9600	
C19-H19B	0.9600	
C19-H19C	0.9600	
C20-H20A	0.9600	
C20-H20B	0.9600	
C20-H20C	0.9600	
C21-C24	1.507(5)	
C21-C23	1.522(6)	
C21-C22	1.523(6)	
C22-H22A	0.9600	
C22-H22B	0.9600	

Table E3. continued...

C22-H22C	0.9600
C23-H23A	0.9600
C23-H23B	0.9600
C23-H23C	0.9600
C24-H24A	0.9600
C24-H24B	0.9600
C24-H24C	0.9600
C25-H25A	0.9600
C25-H25B	0.9600
C25-H25C	0.9600
C31-C32	1.385(4)
C31-C36	1.385(4)
C32-C33	1.392(4)
C32-H32A	0.9300
C33-C34	1.417(5)
C33-C37	1.539(5)
C34-O34	1.379(4)
C34-C35	1.396(5)
O34-C45	1.439(5)
C35-C36	1.379(5)
C35-C41	1.552(5)
C36-H36A	0.9300
C37-C38	1.524(5)
C37-C40	1.541(5)
C37-C39	1.536(5)
C38-H38A	0.9600
C38-H38B	0.9600
C38-H38C	0.9600
C42-H42C	0.9600
C43-H43A	0.9600
C43-H43B	0.9600
C43-H43C	0.9600
C44-H44A	0.9600
C44-H44B	0.9600
C44-H44C	0.9600
C45-H45A	0.9600
C45-H45B	0.9600
C45-H45C	0.9600
C51-H51A	0.9600
C51-H51B	0.9600
C51-H51C	0.9600
C61-O62	1.366(9)
C61-C65	1.395(9)
C61-H61A	0.9700

Table E3. continued...

C61-H61B	0.9700
O62-C63	1.321(9)
C63-C64	1.477(11)
C63-H63A	0.9700
C63-H63B	0.9700
C64-C65	1.523(9)
C64-H64A	0.9700
C64-H64B	0.9700
C65-H65A	0.9700
C65-H65B	0.9700

O1-C1-O2	109.7(3)
O1-C1-C4'	114.1(3)
O2-C1-C4'	113.0(3)
O1-C1-C1'	113.56(19)
O2-C1-C1'	102.5(2)
C4'-C1-C1'	103.3(3)
C51-O1-C1	113.9(4)
C3-O2-C1	111.3(2)
O3-C3-O2	118.8(3)
O3-C3-C4	131.4(3)
O2-C3-C4	109.3(3)
C9-C4-C3	125.0(3)
C9-C4-C1'	129.2(3)
C3-C4-C1'	105.7(3)
C4-C9-C11	122.2(3)
C4-C9-C31	120.0(3)
C16-C11-C12	117.6(3)
C16-C11-C9	121.7(3)
C12-C11-C9	120.7(3)
C13-C12-C11	123.1(3)
C13-C12-H12A	118.4
C11-C12-H12A	118.4
C12-C13-C14	116.1(3)
C12-C13-C17	118.1(3)
C14-C13-C17	125.7(3)
O14-C14-C13	119.6(3)
O14-C14-C15	117.5(3)
C13-C14-C15	122.7(3)
C14-O14-C25	116.1(3)
C16-C15-C14	116.3(3)
C16-C15-C21	120.4(3)
C14-C15-C21	123.2(3)
C15-C16-C11	123.5(3)

Table E3. continued...

C15-C16-H16A	118.3
C11-C16-H16A	118.3
C18-C17-C19	107.4(4)
C18-C17-C20	105.5(4)
C19-C17-C20	112.2(4)
C18-C17-C13	109.4(3)
C19-C17-C13	109.6(3)
C20-C17-C13	112.5(3)
C17-C18-H18A	109.5
C17-C18-H18B	109.5
H18A-C18-H18B	109.5
C17-C18-H18C	109.5
H18A-C18-H18C	109.5
H18B-C18-H18C	109.5
C17-C19-H19A	109.5
C17-C19-H19B	109.5
H19A-C19-H19B	109.5
C17-C19-H19C	109.5
H19A-C19-H19C	109.5
H19B-C19-H19C	109.5
C17-C20-H20A	109.5
C17-C20-H20B	109.5
H20A-C20-H20B	109.5
C17-C20-H20C	109.5
H20A-C20-H20C	109.5
H20B-C20-H20C	109.5
C24-C21-C23	106.2(4)
C24-C21-C22	105.7(4)
C23-C21-C22	112.5(5)
C24-C21-C15	111.6(3)
C23-C21-C15	110.5(3)
C22-C21-C15	110.2(3)
C21-C22-H22A	109.5
C21-C22-H22B	109.5
H22A-C22-H22B	109.5
C21-C22-H22C	109.5
H22A-C22-H22C	109.5
H22B-C22-H22C	109.5
C21-C23-H23A	109.5
C21-C23-H23B	109.5
H23A-C23-H23B	109.5
C21-C23-H23C	109.5
H23A-C23-H23C	109.5
H23B-C23-H23C	109.5

Table E3. continued...

C21-C24-H24A	109.5
C21-C24-H24B	109.5
H24A-C24-H24B	109.5
C21-C24-H24C	109.5
H24A-C24-H24C	109.5
H24B-C24-H24C	109.5
O14-C25-H25A	109.5
O14-C25-H25B	109.5
H25A-C25-H25B	109.5
O14-C25-H25C	109.5
H25A-C25-H25C	109.5
H25B-C25-H25C	109.5
C32-C31-C36	118.0(3)
C32-C31-C9	122.2(3)
C36-C31-C9	119.7(3)
C31-C32-C33	122.4(3)
C31-C32-H32A	118.8
C33-C32-H32A	118.8
C32-C33-C34	116.6(3)
C32-C33-C37	120.7(3)
C34-C33-C37	122.7(3)
O34-C34-C35	120.8(3)
O34-C34-C33	117.1(3)
C35-C34-C33	122.0(3)
C34-O34-C45	115.0(3)
C36-C35-C34	117.0(3)
C36-C35-C41	117.3(3)
C34-C35-C41	125.6(3)
C35-C36-C31	122.9(3)
C35-C36-H36A	118.5
C31-C36-H36A	118.5
C38-C37-C40	106.7(4)
C38-C37-C33	111.2(3)
C40-C37-C33	110.9(3)
C38-C37-C39	107.5(3)
C40-C37-C39	109.9(3)
C33-C37-C39	110.5(3)
C37-C38-H38A	109.5
C37-C38-H38B	109.5
H38A-C38-H38B	109.5
C37-C38-H38C	109.5
H38A-C38-H38C	109.5
H38B-C38-H38C	109.5
C37-C39-H39A	109.5

Table E3. continued...

C37-C39-H39B	109.5
H39A-C39-H39B	109.5
C37-C39-H39C	109.5
H39A-C39-H39C	109.5
H39B-C39-H39C	109.5
C37-C40-H40A	109.5
C37-C40-H40B	109.5
H40A-C40-H40B	109.5
C37-C40-H40C	109.5
H40A-C40-H40C	109.5
H40B-C40-H40C	109.5
C42-C41-C44	107.9(5)
C42-C41-C43	109.7(5)
C44-C41-C43	105.5(5)
C42-C41-C35	114.4(4)
C44-C41-C35	110.0(3)
C43-C41-C35	109.0(3)
C41-C42-H42A	109.5
C41-C42-H42B	109.5
H42A-C42-H42B	109.5
C41-C42-H42C	109.5
H42A-C42-H42C	109.5
H42B-C42-H42C	109.5
C41-C43-H43A	109.5
C41-C43-H43B	109.5
H43A-C43-H43B	109.5
C41-C43-H43C	109.5
H43A-C43-H43C	109.5
H43B-C43-H43C	109.5
C41-C44-H44A	109.5
C41-C44-H44B	109.5
H44A-C44-H44B	109.5
C41-C44-H44C	109.5
H44A-C44-H44C	109.5
H44B-C44-H44C	109.5
O34-C45-H45A	109.5
O34-C45-H45B	109.5
H45A-C45-H45B	109.5
O34-C45-H45C	109.5
H45A-C45-H45C	109.5
H45B-C45-H45C	109.5
O1-C51-H51A	109.5
O1-C51-H51B	109.5
H51A-C51-H51B	109.5

Table E3. continued...

O1-C51-H51C	109.5
H51A-C51-H51C	109.5
H51B-C51-H51C	109.5
O62-C61-C65	106.7(7)
O62-C61-H61A	110.6
C65-C61-H61A	110.5
O62-C61-H61B	110.3
C65-C61-H61B	110.2
H61A-C61-H61B	108.6
C63-O62-C61	113.9(7)
O62-C63-C64	107.3(7)
O62-C63-H63A	110.2
C64-C63-H63A	110.2
O62-C63-H63B	110.3
C64-C63-H63B	110.3
H63A-C63-H63B	108.5
C63-C64-C65	102.0(6)
C63-C64-H64A	111.4
C65-C64-H64A	111.5
C63-C64-H64B	111.3
C65-C64-H64B	111.3
H64A-C64-H64B	109.2
C61-C65-C64	105.1(7)
C61-C65-H65A	110.7
C64-C65-H65A	110.7
C61-C65-H65B	110.9
C64-C65-H65B	110.7
H65A-C65-H65B	108.8

Symmetry transformation used to generate equivalent atoms:
(‘): -x, y, 1/2 - z

Table E4. Anisotropic displacement parameters ($Å^2$) for **7**. The anisotropic displacement factor exponent takes the form:
$-2\pi^2 [h^2a^{*2}U_{11}+...+2hka^*b^*U_{12}]$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0546(19)	0.0542(19)	0.0477(19)	0.0045(16)	0.0001(16)	-0.0074(16)
O1	0.0615(16)	0.0723(17)	0.0588(15)	0.0212(13)	-0.0017(13)	-0.0040(12)
O2	0.0520(13)	0.0685(15)	0.0479(13)	-0.0054(11)	0.0009(11)	0.0054(11)
C3	0.051(2)	0.065(2)	0.0434(19)	0.0001(17)	0.0007(16)	-0.0037(17)
O3	0.0663(16)	0.0806(17)	0.0548(15)	-0.0154(13)	0.0003(12)	0.0074(13)
C4	0.050(2)	0.060(2)	0.0447(19)	-0.0023(15)	0.0062(16)	0.0012(16)
C9	0.053(2)	0.055(2)	0.0451(19)	0.0008(15)	0.0034(16)	-0.0008(15)
C11	0.0461(19)	0.061(2)	0.0443(19)	-0.0031(15)	0.0012(15)	-0.0011(16)
C12	0.0507(19)	0.058(2)	0.0466(19)	-0.0025(16)	0.0004(16)	-0.0004(16)
C13	0.053(2)	0.064(2)	0.0435(19)	-0.0052(16)	0.0059(16)	-0.0044(17)
C14	0.049(2)	0.061(2)	0.051(2)	-0.0077(16)	0.0117(16)	-0.0016(16)
O14	0.0565(15)	0.0766(17)	0.0666(16)	-0.0110(13)	0.0094(12)	0.0052(12)
C15	0.049(2)	0.058(2)	0.057(2)	-0.0046(16)	-0.0011(17)	0.0020(16)
C16	0.051(2)	0.063(2)	0.0446(19)	0.0030(16)	0.0026(16)	-0.0059(17)
C17	0.068(2)	0.073(3)	0.050(2)	0.0008(17)	0.0043(18)	0.0015(19)
C18	0.101(4)	0.085(3)	0.065(3)	0.021(2)	0.022(3)	0.011(3)
C19	0.126(5)	0.098(4)	0.057(3)	-0.002(2)	-0.008(3)	-0.015(3)
C20	0.084(3)	0.111(4)	0.079(3)	0.010(3)	0.032(2)	-0.003(3)
C21	0.060(2)	0.068(2)	0.068(2)	0.0056(18)	-0.0028(18)	0.0112(18)
C22	0.162(6)	0.072(3)	0.153(5)	-0.007(3)	-0.057(4)	0.049(4)
C23	0.055(3)	0.184(6)	0.111(4)	0.061(4)	-0.015(3)	-0.003(3)
C24	0.096(4)	0.138(5)	0.074(3)	0.019(3)	-0.012(3)	0.041(3)
C25	0.084(3)	0.092(3)	0.092(3)	-0.033(3)	0.009(3)	0.013(3)
C31	0.0476(19)	0.061(2)	0.0445(18)	-0.0005(15)	0.0005(15)	0.0053(16)
C32	0.0485(19)	0.069(2)	0.048(2)	0.0030(16)	0.0030(16)	-0.0004(17)
C33	0.055(2)	0.065(2)	0.0445(19)	-0.0003(15)	-0.0013(16)	0.0060(17)
C34	0.057(2)	0.076(2)	0.044(2)	-0.0045(17)	0.0019(17)	0.0143(19)
O34	0.0775(18)	0.102(2)	0.0439(14)	-0.0043(13)	0.0038(12)	0.0138(15)
C35	0.054(2)	0.075(2)	0.051(2)	0.0111(17)	0.0049(17)	0.0011(18)
C36	0.049(2)	0.077(2)	0.049(2)	-0.0015(17)	-0.0005(17)	-0.0030(18)
C37	0.067(2)	0.084(3)	0.049(2)	-0.0081(19)	-0.0071(19)	-0.002(2)
C38	0.067(3)	0.111(4)	0.072(3)	-0.008(3)	-0.010(2)	-0.020(3)
C39	0.070(3)	0.125(4)	0.080(3)	0.010(3)	-0.023(3)	0.001(3)
C40	0.092(3)	0.089(3)	0.088(3)	-0.018(3)	-0.008(3)	-0.011(3)
C41	0.070(3)	0.089(3)	0.060(2)	0.007(2)	0.011(2)	-0.007(2)
C42	0.120(5)	0.165(6)	0.080(4)	0.048(4)	0.003(3)	-0.042(5)
C43	0.075(3)	0.128(5)	0.156(6)	0.002(4)	0.045(3)	-0.018(3)
C44	0.156(6)	0.095(4)	0.112(5)	0.012(3)	0.054(4)	-0.034(4)
C45	0.093(4)	0.107(4)	0.072(3)	-0.021(3)	0.017(3)	0.019(3)

Table E4. continued...

C51	0.142(6)	0.139(6)	0.178(7)	0.004(5)	0.016(5)	-0.029(4)
C61	0.144(7)	0.129(7)	0.401(15)	0.084(8)	0.022(8)	0.015(6)
O62	0.212(7)	0.132(5)	0.262(7)	-0.010(4)	0.023(6)	-0.041(5)
C63	0.193(9)	0.244(12)	0.165(7)	0.054(7)	-0.076(7)	-0.086(9)
C64	0.159(7)	0.209(9)	0.214(9)	0.106(7)	0.038(7)	0.063(7)
C65	0.166(7)	0.129(5)	0.108(4)	-0.014(4)	0.013(4)	-0.039(5)

Table E5. Hydrogen coordinates and isotropic displacement parameters ($Å^2$) for **7**.

Atom	x	y	z	U_{eq}
H12A	-0.1793	0.1172	0.2631	0.056(9)
H16A	-0.2379	0.2652	0.1447	0.036(7)
H18A	-0.1969	0.0564	0.3240	0.094(14)
H18B	-0.2708	0.0137	0.3073	0.113(17)
H18C	-0.2533	0.0265	0.3654	0.082(12)
H19A	-0.1941	0.2079	0.3612	0.14(2)
H19B	-0.2472	0.1719	0.4031	0.126(17)
H19C	-0.2673	0.2577	0.3722	0.13(2)
H20A	-0.3844	0.0926	0.3169	0.117(18)
H20B	-0.3870	0.1850	0.3445	0.13(2)
H20C	-0.3667	0.0992	0.3753	0.109(15)
H22A	-0.3712	0.4287	0.2329	0.53(15)
H22B	-0.3937	0.4681	0.1799	0.128(18)
H22C	-0.3114	0.4553	0.1932	0.24(5)
H23A	-0.4523	0.2894	0.2112	0.108(16)
H23B	-0.4372	0.2412	0.1592	2.0(8)
H23C	-0.4710	0.3369	0.1596	0.123(17)
H24A	-0.2947	0.3727	0.1163	0.16(3)
H24B	-0.3770	0.3873	0.1034	0.090(13)
H24C	-0.3442	0.2911	0.1030	0.098(16)
H25A	-0.4047	0.3720	0.3312	0.25(4)
H25B	-0.3403	0.4030	0.2963	0.33(6)
H25C	-0.3263	0.3343	0.3401	0.30(5)
H32A	-0.2236	0.0888	0.1117	0.047(8)
H36A	-0.0433	0.2340	0.1050	0.055(9)
H38A	-0.3293	0.0987	0.0649	0.074(12)
H38B	-0.2986	0.0038	0.0769	0.092(14)
H38C	-0.3546	0.0168	0.0326	0.100(14)
H39A	-0.2941	0.1754	-0.0164	0.13(2)
H39B	-0.3231	0.0911	-0.0450	0.089(13)
H39C	-0.2441	0.1257	-0.0554	0.14(2)
H40A	-0.1917	-0.0255	-0.0324	0.16(3)
H40B	-0.2717	-0.0589	-0.0250	0.097(14)
H40C	-0.2149	-0.0704	0.0190	0.14(2)
H42A	0.0023	0.3033	-0.0631	0.15(2)
H42B	-0.0795	0.3153	-0.0478	0.17(3)
H42C	-0.0507	0.2218	-0.0647	0.13(2)
H43A	0.0895	0.2420	-0.0073	0.101(14)
H43B	0.0499	0.1498	-0.0060	0.091(15)
H43C	0.0680	0.1991	0.0451	0.24(5)

Table E5. continued...

H44A	0.0268	0.3762	0.0187	0.097(14)
H44B	0.0072	0.3292	0.0703	0.14(2)
H44C	-0.0541	0.3730	0.0373	0.16(3)
H45A	-0.0744	0.0476	-0.0895	0.113(16)
H45B	-0.0860	0.0009	-0.0367	0.115(18)
H45C	-0.0254	0.0733	-0.0429	0.18(3)
H51A	0.0739	0.3405	0.1819	0.230
H51B	-0.0003	0.2909	0.1892	0.230
H51C	0.0661	0.2410	0.1650	0.230
H61A	-0.3276	-0.0777	0.1750	0.270
H61B	-0.3410	-0.0622	0.2337	0.270
H63A	-0.5205	-0.0584	0.1969	0.241
H63B	-0.4937	-0.0947	0.1438	0.241
H64A	-0.4859	0.0725	0.1717	0.233
H64B	-0.4442	0.0321	0.1243	0.233
H65A	-0.3438	0.0605	0.1702	0.161
H65B	-0.3875	0.0640	0.2219	0.161

Table E6. Crystal and structure refinement data for $(1E^{-})_2 \cdot (Na^{+})_2 \cdot (C_4H_{10}O_2)_8$.

Identification code	$(1E^{-})_2 \cdot (Na^{+})_2 \cdot (C_4H_{10}O_2)_8$
Molecular formula	$C_{168}H_{264}O_{32}Na_2$
Formula weight	2841.9045 g/mole
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group	P_1
Unit cell dimensions	a = 16.1411(4) Å b = 16.3146(2) Å c = 18.8482(4) Å α = 64.4540(10)° β = 72.4140(10)° γ = 88.4470(10)°
Volume	4238.05(15) $Å^3$
Z	1
Density (recalculated)	1.114 g/cm^3
Absorption coefficient	0.085 mm^{-1}
F(000)	1617
Crystal Size	? mm x ? mm x ? mm
Color	purple
Theta range for data collection	1.39° to 28.17°
Index ranges	$-20 \le h \le 21$, $-21 \le k \le 21$, $-24 \le l \le 24$
Reflections collected / Unique	50463 / 37328 [R_{int} = 0.0243]
Refinement method	Full-matrix-least-squares on F^2
Data / Restraints / Parameters	37328 / 3 / 1699
Goodness-of-fit on F^2	0.932
Final R indices [$I>2\sigma(I)$]	R_1 = 0.0728, wR_2 = 0.2082
R indices (all data)	R_1 = 0.1332, wR_2 = 0.2604
Largest diff. peak and hole	0.826 and –1.287 $e/Å^3$

Table

Table E7. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for $(1E^-)_2 \cdot (Na^+)_2 \cdot (C_4H_{10}O_2)_8$. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U_{eq}
Na(1)	7495(2)	9183(2)	30(2)	24(1)
C(1)	7476(4)	6220(4)	-613(4)	19(1)
Na(2)	8340(2)	4746(2)	8845(2)	27(1)
C(2)	6986(4)	6526(4)	12(4)	20(1)
C(3)	7436(4)	7231(4)	114(4)	20(1)
C(4)	8350(4)	7700(4)	-520(4)	20(1)
C(5)	8833(4)	7392(4)	-1129(4)	20(1)
C(6)	8386(4)	6694(4)	-1211(4)	20(1)
O(7)	7199(3)	5545(3)	-633(3)	25(1)
O(8)	8726(3)	6403(3)	-1743(3)	26(1)
O(9)	8616(3)	8401(3)	-493(3)	28(1)
O(10)	7104(3)	7523(3)	610(3)	25(1)
C(11)	6114(4)	6174(4)	474(4)	22(1)
C(12)	5713(4)	6010(4)	1350(4)	21(1)
C(13)	4816(4)	6108(4)	1687(4)	25(2)
C(14)	4428(4)	5928(5)	2494(5)	31(2)
C(15)	4951(5)	5639(6)	3016(4)	44(2)
C(16)	5869(5)	5596(4)	2709(4)	27(2)
C(17)	6199(4)	5775(4)	1896(4)	24(2)
C(18)	6476(5)	5398(5)	3258(4)	30(2)
C(19)	6423(6)	4411(6)	3816(6)	58(3)
C(20)	7459(5)	5696(7)	2669(5)	52(2)
C(21)	6296(6)	5961(6)	3748(5)	52(2)
O(22)	4559(4)	5353(6)	3865(3)	89(3)
C(23)	4280(7)	4569(9)	4420(6)	101(4)
C(24)	3446(5)	6081(6)	2817(5)	37(2)
C(25)	3135(6)	6577(9)	2077(7)	95(4)
C(26)	3377(6)	6746(6)	3210(5)	61(2)
C(27)	2870(6)	5204(7)	3411(6)	68(2)
C(28)	5501(4)	5936(5)	111(4)	26(2)
C(29)	4877(4)	5194(5)	554(4)	26(2)
C(30)	4268(4)	5007(5)	193(5)	29(2)
C(31)	4357(4)	5617(5)	-625(4)	26(2)
C(32)	5044(5)	6331(5)	-1121(4)	28(2)
C(33)	5605(4)	6532(4)	-743(4)	23(1)
C(34)	3641(5)	4134(5)	705(5)	35(2)
C(35)	2667(5)	4327(6)	913(6)	51(2)
C(36)	3772(5)	3518(5)	237(5)	41(2)
C(37)	3776(6)	3507(5)	1554(5)	44(2)

Table E7. continued...

O(38)	3753(3)	5457(4)	-970(3)	41(1)
C(39)	3055(6)	5995(7)	-1025(6)	56(2)
C(40)	5233(4)	6983(5)	-2052(4)	34(2)
C(41)	4807(6)	7861(5)	-2207(5)	44(2)
C(42)	6261(6)	7283(7)	-2471(5)	54(2)
C(43)	5039(6)	6477(6)	-2550(5)	52(2)
C(44)	9736(4)	7755(4)	-1609(4)	20(1)
C(45)	10303(4)	7969(4)	-1211(4)	18(1)
C(46)	10257(4)	7438(5)	-406(4)	25(2)
C(47)	10796(4)	7540(5)	7(4)	25(2)
C(48)	11475(4)	8293(5)	-498(5)	32(2)
C(49)	11524(4)	8925(4)	-1328(4)	24(1)
C(50)	10970(4)	8735(4)	-1659(4)	24(1)
C(51)	10554(5)	6925(5)	971(4)	34(2)
C(52)	9588(5)	6637(7)	1364(5)	49(2)
C(53)	10843(6)	7385(6)	1405(5)	51(2)
C(54)	11035(6)	6055(7)	1102(6)	67(3)
O(55)	12089(3)	8482(4)	-189(3)	38(1)
C(56)	12783(5)	7884(6)	-169(5)	57(2)
C(57)	12224(4)	9816(5)	-1844(4)	30(2)
C(58)	12107(5)	10384(6)	-1364(6)	47(2)
C(59)	13154(5)	9584(5)	-2052(5)	41(2)
C(60)	12051(6)	10395(5)	-2662(6)	51(2)
C(61)	10118(4)	7897(4)	-2469(4)	22(1)
C(62)	9606(4)	8150(4)	-2999(4)	23(1)
C(63)	9974(4)	8338(5)	-3849(4)	28(2)
C(64)	10887(4)	8314(5)	-4154(4)	31(2)
C(65)	11409(4)	8008(5)	-3636(4)	31(2)
C(66)	10992(5)	7826(5)	-2773(4)	26(2)
C(67)	9338(5)	8491(5)	-4354(4)	37(2)
C(68)	9524(6)	7902(7)	-4841(5)	58(3)
C(69)	8396(5)	8185(6)	-3806(5)	41(2)
C(70)	9427(7)	9544(6)	-4996(6)	73(3)
O(71)	11270(4)	8484(5)	-4990(3)	61(2)
C(72)	11578(8)	9620(11)	-5465(8)	203(11)
C(73)	12366(5)	7826(7)	-3889(5)	46(2)
C(74)	12730(5)	7337(6)	-3170(5)	47(2)
C(75)	12976(6)	8751(8)	-4417(9)	126(6)
C(76)	12496(8)	7282(12)	-4386(9)	124(6)
C(77)	7459(4)	11063(4)	99(4)	17(1)
C(78)	7791(4)	11682(4)	349(4)	16(1)
C(79)	8217(4)	12602(4)	-298(3)	14(1)
C(80)	8400(4)	12865(4)	-1243(4)	20(1)
C(81)	8032(4)	12226(4)	-1468(4)	19(1)

Table E7. continued...

C(82)	7640(4)	11323(4)	-811(4)	22(2)
O(83)	7456(3)	10703(3)	-975(3)	26(1)
O(84)	7064(3)	10280(3)	604(3)	28(1)
O(85)	8382(3)	13225(3)	-142(3)	24(1)
O(86)	8777(3)	13629(3)	-1735(3)	24(1)
C(87)	7777(4)	11422(4)	1188(4)	19(1)
C(88)	8536(4)	11754(4)	1329(4)	18(1)
C(89)	8465(5)	12010(4)	1951(4)	27(2)
C(90)	9200(4)	12259(4)	2095(4)	24(1)
C(91)	10031(5)	12241(5)	1583(4)	28(2)
C(92)	10142(4)	12072(5)	885(4)	26(2)
C(93)	9382(4)	11795(5)	802(4)	25(2)
C(94)	9091(5)	12594(5)	2776(5)	33(2)
C(95)	9363(6)	11881(7)	3505(4)	51(2)
C(96)	8123(6)	12672(7)	3175(6)	57(3)
C(97)	9605(6)	13564(6)	2410(6)	49(2)
O(98)	10769(3)	12439(4)	1750(3)	41(1)
C(99)	11172(6)	11656(8)	2129(6)	64(3)
C(100)	11010(5)	12234(5)	181(5)	34(2)
C(101)	11681(6)	12999(7)	-2(6)	61(3)
C(102)	10860(6)	12529(7)	-667(5)	63(3)
C(103)	11462(8)	11320(8)	346(8)	88(4)
C(104)	7032(4)	10836(5)	1907(4)	23(1)
C(105)	7148(4)	10263(4)	2671(4)	22(1)
C(106)	6456(5)	9686(5)	3389(4)	31(2)
C(107)	5641(4)	9623(5)	3260(4)	26(2)
C(108)	5453(4)	10252(5)	2539(4)	24(2)
C(109)	6161(4)	10808(4)	1889(4)	23(1)
C(110)	6615(5)	9162(6)	4209(5)	40(2)
C(111)	5851(6)	8769(7)	4999(5)	66(3)
C(112)	7219(8)	8372(7)	4193(7)	73(3)
C(113)	7205(6)	9822(6)	4337(5)	50(2)
O(114)	4958(3)	8974(3)	3895(3)	36(1)
C(115)	5053(6)	8082(5)	3890(5)	55(3)
C(116)	4513(4)	10363(5)	2497(4)	32(2)
C(117)	4018(6)	9459(7)	2674(6)	54(2)
C(118)	4529(5)	11045(6)	1621(5)	57(2)
C(119)	3985(5)	10717(6)	3106(4)	41(2)
C(120)	8058(4)	12499(4)	-2293(4)	19(1)
C(121)	8788(4)	13117(4)	-3029(4)	20(1)
C(122)	8678(5)	13673(5)	-3795(4)	28(2)
C(123)	9369(5)	14255(4)	-4488(4)	27(2)
C(124)	10196(5)	14281(5)	-4395(4)	32(2)
C(125)	10345(4)	13656(5)	-3653(4)	29(2)

Table E7. continued...

C(126)	9643(4)	13062(4)	-2981(4)	23(1)
C(127)	11300(5)	13569(6)	-3620(5)	39(2)
C(128)	11824(5)	14458(7)	-3827(6)	61(3)
C(129)	11312(5)	12822(8)	-2775(5)	78(4)
C(130)	11802(5)	13254(6)	-4305(6)	51(2)
O(131)	10875(3)	14954(4)	-5009(3)	42(1)
C(132)	10811(6)	15831(6)	-5002(6)	59(3)
C(133)	9129(5)	14765(5)	-5327(4)	38(2)
C(134)	8615(6)	14047(6)	-5393(5)	54(2)
C(135)	8649(5)	15554(6)	-5302(5)	56(3)
C(136)	9998(7)	15115(7)	-6123(5)	63(3)
C(137)	6454(4)	12122(4)	-1897(4)	20(1)
C(138)	5682(4)	11836(4)	-1973(4)	24(1)
C(139)	5788(4)	11670(5)	-2675(4)	26(2)
C(140)	6620(5)	11647(4)	-3197(4)	26(2)
C(141)	7360(4)	11933(4)	-3080(4)	21(1)
C(142)	7286(5)	12194(4)	-2430(4)	23(1)
C(143)	4799(4)	11712(6)	-1280(5)	38(2)
C(144)	4990(5)	11446(8)	-474(5)	62(3)
C(145)	4411(6)	12601(7)	-1491(6)	63(3)
C(146)	4179(6)	10911(8)	-1148(6)	69(3)
O(147)	5057(3)	11474(4)	-2855(3)	37(1)
C(148)	4591(6)	12215(6)	-3247(6)	56(2)
C(149)	6742(5)	11320(5)	-3889(4)	29(2)
C(150)	7701(4)	11212(5)	-4256(4)	28(2)
C(151)	6429(6)	11996(5)	-4597(5)	44(2)
C(152)	6208(5)	10381(5)	-3512(4)	39(2)
C(201)	9534(5)	10425(6)	-1221(5)	52(2)
O(202)	9007(4)	10136(4)	-334(4)	43(1)
C(203)	9438(6)	9599(6)	207(6)	46(2)
C(204)	8817(7)	9254(7)	1083(7)	69(3)
O(205)	8107(4)	8645(3)	1206(3)	41(1)
C(206)	7493(8)	8359(7)	1983(5)	76(3)
C(301)	8141(6)	4475(5)	851(5)	44(2)
O(302)	8765(4)	4992(4)	26(3)	44(1)
C(303)	9635(6)	4765(6)	-51(5)	44(2)
C(304)	10172(5)	5231(6)	-869(6)	46(2)
O(305)	9939(3)	4861(4)	-1352(3)	43(1)
C(306)	10418(6)	5303(7)	-2241(5)	64(3)
C(401)	8421(7)	15543(6)	-3090(6)	64(3)
O(402)	7701(4)	15252(4)	-2294(4)	50(2)
C(403)	7029(5)	14660(5)	-2194(5)	40(2)
C(404)	6395(6)	14377(6)	-1350(6)	53(2)
O(405)	6816(3)	13779(3)	-754(3)	36(1)

Table E7. continued...

C(406)	6293(4)	13482(5)	46(5)	44(2)
C(501)	-398(9)	9569(10)	2794(9)	116(4)
O(502)	391(3)	9452(3)	2881(3)	55(1)
C(503)	530(11)	8888(12)	2437(11)	160(6)
C(505)	2138(9)	9095(9)	2246(8)	202(4)
C(504)	1455(10)	8594(10)	2728(9)	136(5)
C(506)	2546(8)	9214(9)	1354(8)	117(4)
C(601)	6216(7)	4231(8)	6195(7)	84(3)
O(602)	5286(17)	4494(19)	6069(16)	371(12)
C(603)	5125(6)	5199(7)	6201(6)	84(2)
C(604)	4105(12)	5300(13)	6415(11)	152(6)
O(605)	3601(5)	4419(5)	6902(5)	102(2)
C(606)	3118(8)	4443(9)	7641(8)	108(4)
C(701)	5951(6)	3223(7)	2272(6)	68(2)
O(702)	5259(7)	2828(8)	3017(7)	149(4)
C(703)	5239(7)	2105(8)	3757(7)	101(3)
C(704)	4365(11)	1989(12)	4706(11)	169(6)
C(705)	4143(8)	996(8)	5280(7)	200(4)
C(706)	4917(8)	877(8)	4863(8)	127(3)
C(801)	9797(8)	842(8)	6591(8)	93(4)
O(802)	10596(7)	1047(7)	5839(6)	131(3)
C(803)	10205(5)	1851(5)	5392(5)	74(2)
C(804)	11021(5)	2398(5)	4470(5)	78(2)
O(805)	11428(7)	1990(8)	4286(7)	153(4)
C(806)	12207(5)	2369(5)	3584(5)	93(2)
C(901)	7666(6)	9412(6)	-1929(5)	47(2)
O(902)	7067(3)	8936(3)	-1146(3)	33(1)
C(903)	6219(5)	9191(5)	-1136(5)	43(2)
C(904)	5600(5)	8673(5)	-175(5)	41(2)
O(905)	5884(3)	9043(3)	274(3)	39(1)
C(906)	5388(6)	8612(7)	1095(7)	63(3)

Table E8. Bond Lengths (Å) and angles (°) for $(1E^{-})_2 \bullet (Na^{+})_2 \bullet (C_4H_{10}O_2)_8$.

Atoms	Length	Angle
Na(1)-O(9)	2.399(5)	
Na(1)-O(83)	2.398(5)	
Na(1)-O(84)	2.452(5)	
Na(1)-O(10)	2.459(5)	
Na(1)-O(905)	2.498(6)	
Na(1)-O(205)	2.503(6)	
Na(1)-O(202)	2.668(6)	
Na(1)-O(902)	2.694(5)	
Na(1)-C(3)	3.122(7)	
Na(1)-C(77)	3.124(6)	
C(1)-O(7)	1.219(7)	
C(1)-C(2)	1.478(8)	
C(1)-C(6)	1.529(9)	
Na(2)-O(85)#1	2.407(5)	
Na(2)-O(7)#2	2.437(5)	
Na(2)-O(8)#2	2.451(5)	
Na(2)-O(402)#1	2.474(6)	
Na(2)-O(305)#2	2.494(6)	
Na(2)-O(86)#1	2.489(5)	
Na(2)-O(405)#1	2.679(5)	
Na(2)-O(302)#2	2.699(6)	
C(2)-C(11)	1.393(9)	
C(2)-C(3)	1.486(9)	
C(3)-O(10)	1.207(7)	
C(3)-C(4)	1.549(8)	
C(4)-O(9)	1.258(7)	
C(4)-C(5)	1.453(9)	
C(5)-C(6)	1.452(9)	
C(5)-C(44)	1.442(8)	
C(6)-O(8)	1.263(8)	
O(7)-Na(2)#3	2.437(5)	
O(8)-Na(2)#3	2.452(5)	
C(11)-C(12)	1.481(9)	
C(11)-C(28)	1.502(9)	
C(12)-C(17)	1.397(9)	
C(12)-C(13)	1.430(9)	
C(13)-C(14)	1.357(10)	
C(14)-C(15)	1.408(11)	
C(14)-C(24)	1.573(9)	
C(15)-O(22)	1.393(8)	
C(15)-C(16)	1.431(10)	

Table E8. continued...

C(16)-C(17)	1.360(9)
C(16)-C(18)	1.563(10)
C(18)-C(19)	1.483(10)
C(18)-C(21)	1.528(11)
C(18)-C(20)	1.580(11)
O(22)-C(23)	1.234(13)
C(24)-C(27)	1.492(12)
C(24)-C(25)	1.510(12)
C(24)-C(26)	1.542(12)
C(28)-C(29)	1.365(9)
C(28)-C(33)	1.436(9)
C(29)-C(30)	1.457(9)
C(30)-C(31)	1.392(10)
C(30)-C(34)	1.512(10)
C(31)-C(32)	1.402(10)
C(31)-O(38)	1.409(8)
C(32)-C(33)	1.430(9)
C(32)-C(40)	1.545(10)
C(34)-C(37)	1.563(11)
C(34)-C(35)	1.561(10)
C(34)-C(36)	1.572(11)
O(38)-C(39)	1.410(10)
C(40)-C(41)	1.522(10)
C(40)-C(42)	1.592(11)
C(40)-C(43)	1.588(11)
C(44)-C(61)	1.463(9)
C(44)-C(45)	1.477(9)
C(45)-C(46)	1.363(9)
C(45)-C(50)	1.430(8)
C(46)-C(47)	1.385(9)
C(47)-C(48)	1.433(10)
C(47)-C(51)	1.575(9)
C(48)-O(55)	1.392(8)
C(48)-C(49)	1.433(9)
C(49)-C(50)	1.344(9)
C(49)-C(57)	1.595(9)
C(51)-C(53)	1.493(11)
C(51)-C(52)	1.498(11)
C(51)-C(54)	1.559(12)
O(55)-C(56)	1.464(9)
C(57)-C(59)	1.514(10)
C(57)-C(58)	1.525(11)
C(57)-C(60)	1.527(11)
C(61)-C(66)	1.375(9)

Table E8. continued...

C(61)-C(62)	1.407(9)
C(62)-C(63)	1.422(9)
C(63)-C(64)	1.417(9)
C(63)-C(67)	1.549(10)
C(64)-O(71)	1.409(9)
C(64)-C(65)	1.402(10)
C(65)-C(66)	1.455(9)
C(65)-C(73)	1.534(10)
C(67)-C(69)	1.508(10)
C(67)-C(68)	1.560(12)
C(67)-C(70)	1.597(11)
O(71)-C(72)	1.683(17)
C(73)-C(76)	1.515(15)
C(73)-C(74)	1.530(11)
C(73)-C(75)	1.558(13)
C(77)-O(84)	1.254(7)
C(77)-C(78)	1.457(8)
C(77)-C(82)	1.512(9)
C(78)-C(87)	1.442(8)
C(78)-C(79)	1.478(8)
C(79)-O(85)	1.228(7)
C(79)-C(80)	1.572(8)
C(80)-O(86)	1.219(7)
C(80)-C(81)	1.479(9)
C(81)-C(120)	1.408(9)
C(81)-C(82)	1.451(9)
C(82)-O(83)	1.245(8)
O(85)-Na(2)#4	2.407(5)
O(86)-Na(2)#4	2.489(5)
C(87)-C(104)	1.469(8)
C(87)-C(88)	1.490(9)
C(88)-C(89)	1.379(9)
C(88)-C(93)	1.409(8)
C(89)-C(90)	1.401(9)
C(90)-C(91)	1.405(9)
C(90)-C(94)	1.561(10)
C(91)-O(98)	1.399(8)
C(91)-C(92)	1.418(10)
C(92)-C(93)	1.393(9)
C(92)-C(100)	1.545(10)
C(94)-C(96)	1.545(12)
C(94)-C(95)	1.546(10)
C(94)-C(97)	1.566(11)
O(98)-C(99)	1.419(11)

Table E8. continued...

C(100)-C(101)	1.524(11)
C(100)-C(102)	1.552(11)
C(100)-C(103)	1.591(13)
C(104)-C(105)	1.406(9)
C(104)-C(109)	1.418(9)
C(105)-C(106)	1.418(9)
C(106)-C(107)	1.422(10)
C(106)-C(110)	1.508(10)
C(107)-O(114)	1.394(7)
C(107)-C(108)	1.422(9)
C(108)-C(109)	1.371(9)
C(108)-C(116)	1.546(9)
C(110)-C(111)	1.503(11)
C(110)-C(113)	1.595(12)
C(110)-C(112)	1.600(12)
O(114)-C(115)	1.463(10)
C(116)-C(117)	1.550(11)
C(116)-C(119)	1.527(11)
C(116)-C(118)	1.534(10)
C(120)-C(121)	1.482(8)
C(120)-C(142)	1.486(9)
C(121)-C(122)	1.399(9)
C(121)-C(126)	1.407(9)
C(122)-C(123)	1.400(9)
C(123)-C(124)	1.400(10)
C(123)-C(133)	1.600(9)
C(124)-O(131)	1.396(8)
C(124)-C(125)	1.419(10)
C(125)-C(126)	1.401(9)
C(125)-C(127)	1.563(10)
C(127)-C(129)	1.538(11)
C(127)-C(128)	1.534(12)
C(127)-C(130)	1.582(11)
O(131)-C(132)	1.436(10)
C(133)-C(135)	1.496(12)
C(133)-C(134)	1.523(12)
C(133)-C(136)	1.605(12)
C(137)-C(142)	1.385(9)
C(137)-C(138)	1.409(8)
C(138)-C(139)	1.422(9)
C(138)-C(143)	1.563(9)
C(139)-O(147)	1.404(8)
C(139)-C(140)	1.415(10)
C(140)-C(141)	1.403(8)

Table E8. continued...

C(140)-C(149)	1.570(9)
C(141)-C(142)	1.434(9)
C(143)-C(145)	1.503(12)
C(143)-C(144)	1.517(11)
C(143)-C(146)	1.552(12)
O(147)-C(148)	1.445(9)
C(149)-C(150)	1.533(9)
C(149)-C(151)	1.536(10)
C(149)-C(152)	1.536(10)
C(201)-O(202)	1.491(9)
O(202)-C(203)	1.384(9)
C(203)-C(204)	1.509(14)
C(204)-O(205)	1.435(11)
O(205)-C(206)	1.379(10)
C(301)-O(302)	1.453(9)
O(302)-C(303)	1.424(9)
O(302)-Na(2)#3	2.699(6)
C(303)-C(304)	1.387(12)
C(304)-O(305)	1.424(9)
O(305)-C(306)	1.460(9)
O(305)-Na(2)#3	2.494(6)
C(401)-O(402)	1.480(11)
O(402)-C(403)	1.386(9)
O(402)-Na(2)#4	2.474(6)
C(403)-C(404)	1.482(12)
C(404)-O(405)	1.468(10)
O(405)-C(406)	1.359(9)
O(405)-Na(2)#4	2.678(5)
C(501)-O(502)	1.331(14)
C(501)-C(503)	1.97(2)
O(502)-C(503)	1.462(18)
C(503)-C(504)	1.73(2)
C(505)-C(504)	1.230(17)
C(505)-C(506)	1.535(16)
C(601)-O(602)	1.61(3)
O(602)-C(603)	1.28(3)
C(603)-C(604)	1.59(2)
C(604)-O(605)	1.431(19)
O(605)-C(606)	1.392(13)
C(701)-O(702)	1.395(13)
O(702)-C(703)	1.376(14)
C(703)-C(704)	1.85(2)
C(704)-C(705)	1.486(19)
C(704)-C(706)	1.94(2)

Table E8. continued...

C(705)-C(706)	1.319(14)
C(801)-O(802)	1.516(15)
O(802)-C(803)	1.467(12)
C(803)-C(804)	1.701(10)
C(804)-O(805)	1.016(11)
O(805)-C(806)	1.429(13)
C(901)-O(902)	1.380(9)
O(902)-C(903)	1.417(10)
C(903)-C(904)	1.620(11)
C(904)-O(905)	1.408(10)
O(905)-C(906)	1.369(11)

O(9)-Na(1)-O(83)	113.74(18)
O(9)-Na(1)-O(84)	149.23(19)
O(83)-Na(1)-O(84)	65.32(16)
O(9)-Na(1)-O(10)	65.36(16)
O(83)-Na(1)-O(10)	149.18(19)
O(84)-Na(1)-O(10)	132.33(17)
O(9)-Na(1)-O(905)	130.9(2)
O(83)-Na(1)-O(905)	83.23(19)
O(84)-Na(1)-O(905)	79.87(18)
O(10)-Na(1)-O(905)	77.19(17)
O(9)-Na(1)-O(205)	83.13(19)
O(83)-Na(1)-O(205)	130.38(19)
O(84)-Na(1)-O(205)	77.11(18)
O(10)-Na(1)-O(205)	80.42(17)
O(905)-Na(1)-O(205)	121.7(2)
O(9)-Na(1)-O(202)	74.45(17)
O(83)-Na(1)-O(202)	73.87(17)
O(84)-Na(1)-O(202)	76.07(17)
O(10)-Na(1)-O(202)	130.17(19)
O(905)-Na(1)-O(202)	152.00(19)
O(205)-Na(1)-O(202)	66.27(19)
O(9)-Na(1)-O(902)	73.49(17)
O(83)-Na(1)-O(902)	75.75(16)
O(84)-Na(1)-O(902)	130.94(19)
O(10)-Na(1)-O(902)	74.70(16)
O(905)-Na(1)-O(902)	66.55(18)
O(205)-Na(1)-O(902)	151.18(18)
O(202)-Na(1)-O(902)	121.02(19)
O(9)-Na(1)-C(3)	48.50(16)
O(83)-Na(1)-C(3)	135.86(18)
O(84)-Na(1)-C(3)	153.27(17)
O(10)-Na(1)-C(3)	20.97(15)

Table E8. continued...

O(905)-Na(1)-C(3)	86.45(17)
O(205)-Na(1)-C(3)	91.09(17)
O(202)-Na(1)-C(3)	121.25(18)
O(902)-Na(1)-C(3)	60.83(16)
O(9)-Na(1)-C(77)	134.79(17)
O(83)-Na(1)-C(77)	47.93(15)
O(84)-Na(1)-C(77)	22.05(14)
O(10)-Na(1)-C(77)	154.32(18)
O(905)-Na(1)-C(77)	91.61(18)
O(205)-Na(1)-C(77)	86.28(17)
O(202)-Na(1)-C(77)	61.10(16)
O(902)-Na(1)-C(77)	122.19(17)
C(3)-Na(1)-C(77)	175.28(18)
O(7)-C(1)-C(2)	121.4(6)
O(7)-C(1)-C(6)	117.5(5)
C(2)-C(1)-C(6)	120.9(5)
O(85)#1-Na(2)-O(7)#2	113.93(18)
O(85)#1-Na(2)-O(8)#2	149.29(19)
O(7)#2-Na(2)-O(8)#2	65.31(16)
O(85)#1-Na(2)-O(402)#1	129.7(2)
O(7)#2-Na(2)-O(402)#1	82.18(19)
O(8)#2-Na(2)-O(402)#1	80.97(19)
O(85)#1-Na(2)-O(305)#2	81.75(18)
O(7)#2-Na(2)-O(305)#2	130.56(19)
O(8)#2-Na(2)-O(305)#2	78.13(18)
O(402)#1-Na(2)-O(305)#2	124.8(2)
O(85)#1-Na(2)-O(86)#1	65.44(16)
O(7)#2-Na(2)-O(86)#1	149.02(19)
O(8)#2-Na(2)-O(86)#1	132.18(18)
O(402)#1-Na(2)-O(86)#1	77.19(19)
O(305)#2-Na(2)-O(86)#1	80.41(18)
O(85)#1-Na(2)-O(405)#1	73.39(16)
O(7)#2-Na(2)-O(405)#1	73.86(16)
O(8)#2-Na(2)-O(405)#1	130.21(18)
O(402)#1-Na(2)-O(405)#1	65.97(19)
O(305)#2-Na(2)-O(405)#1	151.46(19)
O(86)#1-Na(2)-O(405)#1	76.70(17)
O(85)#1-Na(2)-O(302)#2	75.36(17)
O(7)#2-Na(2)-O(302)#2	73.80(17)
O(8)#2-Na(2)-O(302)#2	75.23(17)
O(402)#1-Na(2)-O(302)#2	151.5(2)
O(305)#2-Na(2)-O(302)#2	65.23(18)
O(86)#1-Na(2)-O(302)#2	130.86(19)
O(405)#1-Na(2)-O(302)#2	119.66(19)

Table E8. continued...

C(11)-C(2)-C(1)	120.1(5)
C(11)-C(2)-C(3)	121.3(5)
C(1)-C(2)-C(3)	118.5(5)
O(10)-C(3)-C(2)	124.3(5)
O(10)-C(3)-C(4)	117.5(6)
C(2)-C(3)-C(4)	117.9(5)
O(10)-C(3)-Na(1)	46.8(3)
C(2)-C(3)-Na(1)	147.7(4)
C(4)-C(3)-Na(1)	76.9(3)
O(9)-C(4)-C(5)	122.9(5)
O(9)-C(4)-C(3)	114.1(6)
C(5)-C(4)-C(3)	122.9(5)
C(6)-C(5)-C(4)	117.8(5)
C(6)-C(5)-C(44)	121.9(6)
C(4)-C(5)-C(44)	120.2(6)
O(8)-C(6)-C(5)	123.4(6)
O(8)-C(6)-C(1)	115.4(5)
C(5)-C(6)-C(1)	121.1(5)
C(1)-O(7)-Na(2)#3	114.0(4)
C(6)-O(8)-Na(2)#3	111.0(4)
C(4)-O(9)-Na(1)	115.6(4)
C(3)-O(10)-Na(1)	112.2(4)
C(2)-C(11)-C(12)	123.6(6)
C(2)-C(11)-C(28)	121.8(6)
C(12)-C(11)-C(28)	114.6(5)
C(17)-C(12)-C(13)	115.4(6)
C(17)-C(12)-C(11)	121.9(6)
C(13)-C(12)-C(11)	122.7(6)
C(14)-C(13)-C(12)	123.7(6)
C(13)-C(14)-C(15)	117.3(6)
C(13)-C(14)-C(24)	121.0(6)
C(15)-C(14)-C(24)	121.7(6)
O(22)-C(15)-C(14)	119.2(7)
O(22)-C(15)-C(16)	118.5(7)
C(14)-C(15)-C(16)	122.2(6)
C(17)-C(16)-C(15)	116.2(6)
C(17)-C(16)-C(18)	121.1(6)
C(15)-C(16)-C(18)	122.6(6)
C(16)-C(17)-C(12)	124.9(6)
C(19)-C(18)-C(21)	111.1(7)
C(19)-C(18)-C(16)	112.9(6)
C(21)-C(18)-C(16)	111.2(6)
C(19)-C(18)-C(20)	105.6(7)
C(21)-C(18)-C(20)	107.1(6)

Table E8. continued...

C(16)-C(18)-C(20)	108.6(5)
C(23)-O(22)-C(15)	128.6(10)
C(27)-C(24)-C(25)	110.3(8)
C(27)-C(24)-C(26)	110.9(7)
C(25)-C(24)-C(26)	104.4(8)
C(27)-C(24)-C(14)	112.3(7)
C(25)-C(24)-C(14)	108.4(6)
C(26)-C(24)-C(14)	110.3(7)
C(29)-C(28)-C(33)	120.7(6)
C(29)-C(28)-C(11)	122.9(6)
C(33)-C(28)-C(11)	116.4(6)
C(28)-C(29)-C(30)	121.4(6)
C(31)-C(30)-C(29)	116.8(6)
C(31)-C(30)-C(34)	125.0(6)
C(29)-C(30)-C(34)	117.9(7)
C(30)-C(31)-C(32)	123.0(6)
C(30)-C(31)-O(38)	117.2(6)
C(32)-C(31)-O(38)	119.7(6)
C(31)-C(32)-C(33)	118.8(6)
C(31)-C(32)-C(40)	126.1(6)
C(33)-C(32)-C(40)	115.0(6)
C(32)-C(33)-C(28)	118.6(6)
C(30)-C(34)-C(37)	114.1(6)
C(30)-C(34)-C(35)	112.0(6)
C(37)-C(34)-C(35)	106.2(7)
C(30)-C(34)-C(36)	110.8(6)
C(37)-C(34)-C(36)	104.4(6)
C(35)-C(34)-C(36)	109.0(6)
C(39)-O(38)-C(31)	118.2(6)
C(41)-C(40)-C(32)	113.5(6)
C(41)-C(40)-C(42)	106.4(6)
C(32)-C(40)-C(42)	108.4(6)
C(41)-C(40)-C(43)	113.0(6)
C(32)-C(40)-C(43)	112.3(6)
C(42)-C(40)-C(43)	102.3(7)
C(5)-C(44)-C(61)	122.6(6)
C(5)-C(44)-C(45)	118.8(6)
C(61)-C(44)-C(45)	118.6(5)
C(46)-C(45)-C(50)	115.1(6)
C(46)-C(45)-C(44)	123.0(5)
C(50)-C(45)-C(44)	121.9(6)
C(45)-C(46)-C(47)	127.6(6)
C(46)-C(47)-C(48)	113.8(6)
C(46)-C(47)-C(51)	119.2(6)

Table E8. continued...

C(48)-C(47)-C(51)	126.6(6)
O(55)-C(48)-C(47)	121.5(7)
O(55)-C(48)-C(49)	116.5(6)
C(47)-C(48)-C(49)	121.8(6)
C(50)-C(49)-C(48)	117.9(6)
C(50)-C(49)-C(57)	121.2(6)
C(48)-C(49)-C(57)	120.9(6)
C(49)-C(50)-C(45)	123.4(6)
C(53)-C(51)-C(52)	108.8(7)
C(53)-C(51)-C(54)	107.5(7)
C(52)-C(51)-C(54)	108.9(7)
C(53)-C(51)-C(47)	113.2(6)
C(52)-C(51)-C(47)	110.8(6)
C(54)-C(51)-C(47)	107.6(6)
C(48)-O(55)-C(56)	112.6(6)
C(59)-C(57)-C(58)	110.9(6)
C(59)-C(57)-C(60)	107.7(6)
C(58)-C(57)-C(60)	107.8(6)
C(59)-C(57)-C(49)	112.2(6)
C(58)-C(57)-C(49)	109.6(6)
C(60)-C(57)-C(49)	108.5(6)
C(66)-C(61)-C(62)	119.6(6)
C(66)-C(61)-C(44)	120.1(6)
C(62)-C(61)-C(44)	120.3(6)
C(61)-C(62)-C(63)	121.3(6)
C(64)-C(63)-C(62)	117.6(6)
C(64)-C(63)-C(67)	124.9(6)
C(62)-C(63)-C(67)	117.4(6)
O(71)-C(64)-C(65)	117.8(6)
O(71)-C(64)-C(63)	118.9(6)
C(65)-C(64)-C(63)	122.7(6)
C(64)-C(65)-C(66)	116.5(6)
C(64)-C(65)-C(73)	126.1(6)
C(66)-C(65)-C(73)	117.4(6)
C(61)-C(66)-C(65)	121.9(6)
C(69)-C(67)-C(68)	104.9(7)
C(69)-C(67)-C(63)	112.4(6)
C(68)-C(67)-C(63)	110.6(7)
C(69)-C(67)-C(70)	108.7(7)
C(68)-C(67)-C(70)	109.1(7)
C(63)-C(67)-C(70)	110.9(7)
C(64)-O(71)-C(72)	102.2(8)
C(76)-C(73)-C(65)	110.9(8)
C(76)-C(73)-C(74)	108.4(8)

Table E8. continued...

C(65)-C(73)-C(74)	115.2(6)
C(76)-C(73)-C(75)	109.5(10)
C(65)-C(73)-C(75)	109.9(8)
C(74)-C(73)-C(75)	102.6(8)
O(84)-C(77)-C(78)	123.1(5)
O(84)-C(77)-C(82)	116.5(5)
C(78)-C(77)-C(82)	120.2(5)
O(84)-C(77)-Na(1)	47.2(3)
C(78)-C(77)-Na(1)	148.1(4)
C(82)-C(77)-Na(1)	76.3(3)
C(87)-C(78)-C(77)	123.3(5)
C(87)-C(78)-C(79)	118.2(5)
C(77)-C(78)-C(79)	118.4(5)
O(85)-C(79)-C(78)	123.3(5)
O(85)-C(79)-C(80)	115.8(5)
C(78)-C(79)-C(80)	120.6(5)
O(86)-C(80)-C(81)	124.7(6)
O(86)-C(80)-C(79)	116.1(6)
C(81)-C(80)-C(79)	118.8(5)
C(120)-C(81)-C(82)	121.9(6)
C(120)-C(81)-C(80)	120.4(5)
C(82)-C(81)-C(80)	117.8(5)
O(83)-C(82)-C(81)	121.0(6)
O(83)-C(82)-C(77)	115.8(5)
C(81)-C(82)-C(77)	123.1(6)
C(82)-O(83)-Na(1)	114.9(4)
C(77)-O(84)-Na(1)	110.7(4)
C(79)-O(85)-Na(2)#4	115.6(4)
C(80)-O(86)-Na(2)#4	111.1(4)
C(78)-C(87)-C(104)	121.4(6)
C(78)-C(87)-C(88)	119.3(5)
C(104)-C(87)-C(88)	119.2(5)
C(89)-C(88)-C(93)	117.7(6)
C(89)-C(88)-C(87)	124.3(6)
C(93)-C(88)-C(87)	118.0(6)
C(88)-C(89)-C(90)	122.3(6)
C(89)-C(90)-C(91)	117.9(6)
C(89)-C(90)-C(94)	120.6(6)
C(91)-C(90)-C(94)	121.4(6)
O(98)-C(91)-C(90)	118.5(6)
O(98)-C(91)-C(92)	119.4(6)
C(90)-C(91)-C(92)	122.0(6)
C(93)-C(92)-C(91)	116.2(6)
C(93)-C(92)-C(100)	117.5(6)

Table E8. continued...

C(91)-C(92)-C(100)	126.2(6)
C(92)-C(93)-C(88)	123.4(6)
C(90)-C(94)-C(96)	111.6(6)
C(90)-C(94)-C(95)	110.3(6)
C(96)-C(94)-C(95)	104.5(7)
C(90)-C(94)-C(97)	111.8(6)
C(96)-C(94)-C(97)	106.6(7)
C(95)-C(94)-C(97)	111.7(7)
C(91)-O(98)-C(99)	113.7(6)
C(101)-C(100)-C(92)	115.8(6)
C(101)-C(100)-C(102)	103.0(7)
C(92)-C(100)-C(102)	111.7(6)
C(101)-C(100)-C(103)	108.9(8)
C(92)-C(100)-C(103)	111.3(7)
C(102)-C(100)-C(103)	105.4(8)
C(105)-C(104)-C(109)	115.6(6)
C(105)-C(104)-C(87)	120.8(6)
C(109)-C(104)-C(87)	123.6(6)
C(104)-C(105)-C(106)	123.7(6)
C(107)-C(106)-C(105)	115.1(6)
C(107)-C(106)-C(110)	124.3(6)
C(105)-C(106)-C(110)	120.6(6)
O(114)-C(107)-C(106)	119.4(6)
O(114)-C(107)-C(108)	117.2(6)
C(106)-C(107)-C(108)	123.1(6)
C(109)-C(108)-C(107)	115.6(6)
C(109)-C(108)-C(116)	121.2(6)
C(107)-C(108)-C(116)	123.1(6)
C(108)-C(109)-C(104)	125.3(6)
C(106)-C(110)-C(111)	119.6(7)
C(106)-C(110)-C(113)	108.8(6)
C(111)-C(110)-C(113)	104.4(7)
C(106)-C(110)-C(112)	111.3(7)
C(111)-C(110)-C(112)	108.6(7)
C(113)-C(110)-C(112)	102.7(7)
C(107)-O(114)-C(115)	110.3(5)
C(117)-C(116)-C(119)	109.1(6)
C(117)-C(116)-C(108)	112.5(7)
C(119)-C(116)-C(108)	110.9(6)
C(117)-C(116)-C(118)	105.3(7)
C(119)-C(116)-C(118)	108.1(7)
C(108)-C(116)-C(118)	110.7(5)
C(81)-C(120)-C(121)	123.7(6)
C(81)-C(120)-C(142)	117.9(5)

Table E8. continued...

C(121)-C(120)-C(142)	118.4(5)
C(122)-C(121)-C(126)	118.4(6)
C(122)-C(121)-C(120)	122.8(6)
C(126)-C(121)-C(120)	118.4(5)
C(123)-C(122)-C(121)	122.6(7)
C(122)-C(123)-C(124)	117.8(6)
C(122)-C(123)-C(133)	114.9(6)
C(124)-C(123)-C(133)	127.1(6)
O(131)-C(124)-C(123)	120.8(6)
O(131)-C(124)-C(125)	118.6(7)
C(123)-C(124)-C(125)	120.6(6)
C(124)-C(125)-C(126)	119.7(6)
C(124)-C(125)-C(127)	119.8(6)
C(126)-C(125)-C(127)	120.2(6)
C(121)-C(126)-C(125)	120.0(6)
C(129)-C(127)-C(128)	109.6(8)
C(129)-C(127)-C(125)	111.1(6)
C(128)-C(127)-C(125)	115.4(7)
C(129)-C(127)-C(130)	107.6(8)
C(128)-C(127)-C(130)	106.4(6)
C(125)-C(127)-C(130)	106.3(6)
C(124)-O(131)-C(132)	114.0(6)
C(135)-C(133)-C(134)	114.7(7)
C(135)-C(133)-C(123)	108.7(6)
C(134)-C(133)-C(123)	106.8(6)
C(135)-C(133)-C(136)	110.4(6)
C(134)-C(133)-C(136)	105.4(7)
C(123)-C(133)-C(136)	110.7(6)
C(142)-C(137)-C(138)	124.0(6)
C(139)-C(138)-C(137)	116.1(6)
C(139)-C(138)-C(143)	126.0(6)
C(137)-C(138)-C(143)	117.9(6)
O(147)-C(139)-C(138)	120.6(6)
O(147)-C(139)-C(140)	116.8(6)
C(138)-C(139)-C(140)	122.5(6)
C(141)-C(140)-C(139)	117.5(6)
C(141)-C(140)-C(149)	119.5(6)
C(139)-C(140)-C(149)	122.9(6)
C(140)-C(141)-C(142)	121.8(6)
C(137)-C(142)-C(141)	117.4(6)
C(137)-C(142)-C(120)	119.7(6)
C(141)-C(142)-C(120)	122.8(6)
C(145)-C(143)-C(144)	108.1(7)
C(145)-C(143)-C(138)	110.1(7)

Table E8. continued...

C(144)-C(143)-C(138)	108.1(5)
C(145)-C(143)-C(146)	113.6(7)
C(144)-C(143)-C(146)	107.3(8)
C(138)-C(143)-C(146)	109.6(6)
C(139)-O(147)-C(148)	119.0(6)
C(150)-C(149)-C(151)	108.6(6)
C(150)-C(149)-C(152)	107.3(6)
C(151)-C(149)-C(152)	108.5(6)
C(150)-C(149)-C(140)	111.5(6)
C(151)-C(149)-C(140)	111.3(6)
C(152)-C(149)-C(140)	109.5(5)
C(203)-O(202)-C(201)	112.4(7)
C(203)-O(202)-Na(1)	108.3(5)
C(201)-O(202)-Na(1)	109.9(5)
O(202)-C(203)-C(204)	108.5(7)
O(205)-C(204)-C(203)	109.7(7)
C(206)-O(205)-C(204)	111.6(8)
C(206)-O(205)-Na(1)	115.1(6)
C(204)-O(205)-Na(1)	114.4(5)
C(303)-O(302)-C(301)	114.0(6)
C(303)-O(302)-Na(2)#3	105.6(5)
C(301)-O(302)-Na(2)#3	112.6(5)
C(304)-C(303)-O(302)	110.3(6)
C(303)-C(304)-O(305)	108.8(6)
C(304)-O(305)-C(306)	116.0(6)
C(304)-O(305)-Na(2)#3	113.8(5)
C(306)-O(305)-Na(2)#3	108.6(5)
C(403)-O(402)-C(401)	113.7(6)
C(403)-O(402)-Na(2)#4	116.4(5)
C(401)-O(402)-Na(2)#4	108.6(5)
O(402)-C(403)-C(404)	107.4(6)
O(405)-C(404)-C(403)	107.3(7)
C(406)-O(405)-C(404)	112.4(6)
C(406)-O(405)-Na(2)#4	115.2(5)
C(404)-O(405)-Na(2)#4	104.9(4)
O(502)-C(501)-C(503)	47.9(7)
C(501)-O(502)-C(503)	89.6(10)
O(502)-C(503)-C(504)	87.6(10)
O(502)-C(503)-C(501)	42.5(6)
C(504)-C(503)-C(501)	129.3(12)
C(504)-C(505)-C(506)	122.8(14)
C(505)-C(504)-C(503)	115.9(13)
C(603)-O(602)-C(601)	107(2)
O(602)-C(603)-C(604)	110.8(16)

Table E8. continued...

O(605)-C(604)-C(603)	110.4(12)
C(606)-O(605)-C(604)	103.9(10)
C(703)-O(702)-C(701)	128.9(10)
O(702)-C(703)-C(704)	117.5(10)
C(705)-C(704)-C(703)	107.3(12)
C(705)-C(704)-C(706)	42.7(7)
C(703)-C(704)-C(706)	67.4(8)
C(706)-C(705)-C(704)	87.5(11)
C(705)-C(706)-C(704)	49.8(8)
C(803)-O(802)-C(801)	86.8(8)
O(802)-C(803)-C(804)	102.1(6)
O(805)-C(804)-C(803)	115.7(9)
C(804)-O(805)-C(806)	121.3(11)
C(901)-O(902)-C(903)	110.6(6)
C(901)-O(902)-Na(1)	112.8(5)
C(903)-O(902)-Na(1)	109.2(4)
O(902)-C(903)-C(904)	106.1(6)
O(905)-C(904)-C(903)	107.8(6)
C(906)-O(905)-C(904)	109.8(6)
C(906)-O(905)-Na(1)	114.0(5)
C(904)-O(905)-Na(1)	116.1(4)

Symmetry transformations used to generate equivalent atoms:

#1 x, y-1, z+1
#2 x, y, z+1
#3 x, y, z-1
#4 x, y+1, z-1

Table E9. Anisotropic displacement parameters ($Å^2 \times 10^3$) for $(1E^-)_2 \cdot (Na^+)_2 \cdot (C_4H_{10}O_2)_8$. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+...+2hka^*b^*U_{12}]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Na(1)	26(1)	19(1)	23(1)	-8(1)	-5(1)	-2(1)
C(1)	16(3)	25(3)	21(3)	-14(3)	-8(2)	11(3)
Na(2)	34(2)	22(1)	27(1)	-14(1)	-10(1)	4(1)
C(2)	28(3)	19(3)	15(3)	-7(3)	-11(3)	-2(3)
C(3)	13(3)	27(3)	14(3)	-6(3)	-4(2)	-1(3)
C(4)	27(3)	13(3)	16(3)	-1(2)	-9(3)	-10(2)
C(5)	13(3)	21(3)	20(3)	-5(3)	-1(2)	4(3)
C(6)	30(3)	12(3)	17(3)	-6(3)	-9(3)	3(3)
O(7)	23(2)	26(2)	30(3)	-17(2)	-5(2)	-5(2)
O(8)	29(3)	22(2)	20(2)	-7(2)	-2(2)	-4(2)
O(9)	24(2)	28(3)	35(3)	-19(2)	-3(2)	0(2)
O(10)	27(3)	28(3)	24(2)	-18(2)	0(2)	-4(2)
C(11)	27(3)	19(3)	22(3)	-12(3)	-7(3)	-1(3)
C(12)	26(3)	13(3)	19(3)	-3(3)	-6(3)	-1(3)
C(13)	13(3)	25(3)	31(4)	-9(3)	-4(3)	-2(3)
C(14)	20(3)	40(4)	30(4)	-13(3)	-7(3)	8(3)
C(15)	38(4)	63(5)	14(3)	-11(3)	8(3)	-4(4)
C(16)	31(4)	20(3)	21(3)	-2(3)	-8(3)	3(3)
C(17)	20(3)	24(3)	21(3)	-7(3)	-3(3)	-7(3)
C(18)	39(4)	31(4)	18(3)	-6(3)	-14(3)	7(3)
C(19)	69(6)	39(5)	65(6)	-4(4)	-49(5)	2(4)
C(20)	39(4)	92(7)	41(5)	-33(5)	-28(4)	10(4)
C(21)	51(5)	57(6)	46(5)	-21(4)	-17(4)	-3(4)
O(22)	41(4)	177(8)	8(3)	-14(4)	7(2)	9(4)
C(23)	60(5)	140(10)	39(5)	3(6)	6(4)	0(6)
C(24)	19(3)	54(5)	33(4)	-19(4)	-4(3)	12(3)
C(25)	41(5)	165(11)	62(7)	-40(7)	-16(5)	61(6)
C(26)	64(5)	76(5)	53(5)	-39(4)	-18(4)	15(4)
C(27)	42(5)	78(6)	58(5)	-21(5)	3(4)	6(4)
C(28)	19(3)	38(4)	27(4)	-18(3)	-11(3)	7(3)
C(29)	21(3)	34(4)	26(3)	-13(3)	-14(3)	0(3)
C(30)	14(3)	41(4)	40(4)	-25(4)	-9(3)	5(3)
C(31)	25(4)	30(4)	35(4)	-22(3)	-15(3)	1(3)
C(32)	36(4)	32(4)	23(3)	-14(3)	-18(3)	14(3)
C(33)	22(3)	21(3)	26(3)	-9(3)	-9(3)	-2(3)
C(34)	27(4)	39(4)	45(4)	-23(4)	-12(3)	3(3)
C(35)	31(4)	37(5)	78(7)	-28(5)	-3(4)	1(4)
C(36)	46(4)	34(4)	52(5)	-26(4)	-14(4)	-10(3)
C(37)	46(5)	39(5)	34(4)	-8(4)	-6(4)	-14(4)

Table E9. continued...

O(38)	31(3)	58(4)	42(3)	-23(3)	-21(2)	-4(3)
C(39)	43(5)	79(6)	74(6)	-48(5)	-34(4)	9(4)
C(40)	20(3)	51(5)	34(4)	-21(4)	-7(3)	-1(3)
C(41)	57(5)	29(4)	31(4)	0(3)	-16(4)	8(4)
C(42)	47(5)	61(6)	30(4)	-7(4)	-2(4)	-5(5)
C(43)	68(6)	67(5)	32(4)	-29(4)	-21(4)	-5(4)
C(44)	17(3)	13(3)	20(3)	0(3)	-6(3)	3(2)
C(45)	17(3)	16(3)	21(3)	-8(3)	-2(2)	-3(2)
C(46)	22(3)	27(4)	23(3)	-10(3)	-5(3)	1(3)
C(47)	20(3)	26(3)	27(4)	-11(3)	-6(3)	0(3)
C(48)	21(3)	44(4)	34(4)	-19(4)	-12(3)	7(3)
C(49)	23(3)	23(3)	23(3)	-8(3)	-5(3)	-4(3)
C(50)	26(3)	20(3)	17(3)	-6(3)	1(3)	-1(3)
C(51)	50(4)	35(4)	16(3)	-3(3)	-20(3)	10(3)
C(52)	34(4)	82(6)	18(4)	-11(4)	-9(3)	-6(4)
C(53)	60(5)	54(5)	35(4)	-14(4)	-20(4)	7(4)
C(54)	63(6)	78(7)	62(6)	-25(6)	-33(5)	28(5)
O(55)	35(3)	47(3)	46(3)	-24(3)	-24(2)	4(2)
C(56)	26(4)	66(5)	54(5)	4(4)	-26(3)	5(3)
C(57)	22(3)	27(4)	36(4)	-10(3)	-6(3)	-12(3)
C(58)	24(3)	49(5)	74(6)	-31(4)	-17(4)	5(3)
C(59)	24(4)	48(5)	41(4)	-15(4)	-5(3)	-13(3)
C(60)	44(5)	31(4)	62(6)	-2(4)	-25(4)	-17(4)
C(61)	17(3)	27(4)	16(3)	-8(3)	0(3)	-4(3)
C(62)	20(3)	23(3)	24(3)	-9(3)	-7(3)	9(3)
C(63)	26(4)	40(4)	16(3)	-10(3)	-7(3)	2(3)
C(64)	26(3)	40(4)	19(3)	-8(3)	-6(3)	6(3)
C(65)	23(3)	36(4)	18(3)	-6(3)	7(3)	-8(3)
C(66)	30(4)	31(4)	14(3)	-9(3)	-7(3)	2(3)
C(67)	34(4)	45(5)	27(4)	-10(4)	-11(3)	0(3)
C(68)	57(6)	78(6)	41(5)	-40(5)	0(4)	-2(5)
C(69)	27(4)	61(5)	33(4)	-20(4)	-8(3)	8(3)
C(70)	78(7)	49(6)	60(6)	13(5)	-35(5)	13(5)
O(71)	40(3)	106(5)	28(3)	-28(3)	-5(2)	5(3)
C(72)	63(7)	204(15)	103(9)	134(10)	-7(6)	-28(8)
C(73)	27(4)	69(6)	33(4)	-22(4)	2(3)	2(4)
C(74)	32(4)	70(5)	42(4)	-28(4)	-14(3)	18(4)
C(75)	17(4)	88(7)	170(13)	14(8)	1(5)	-24(4)
C(76)	77(8)	246(17)	138(11)	-156(12)	-53(8)	96(9)
C(77)	16(3)	11(3)	18(3)	-3(2)	-4(2)	-5(2)
C(78)	16(3)	19(3)	17(3)	-10(3)	-5(2)	2(2)
C(79)	12(3)	21(3)	9(3)	-8(2)	-2(2)	0(2)
C(80)	19(3)	29(4)	15(3)	-12(3)	-5(2)	10(3)
C(81)	20(3)	20(3)	15(3)	-5(3)	-6(3)	0(3)

Table E9. continued...

C(82)	21(3)	17(3)	31(4)	-11(3)	-12(3)	2(3)
O(83)	45(3)	23(2)	17(2)	-10(2)	-16(2)	2(2)
O(84)	31(3)	26(2)	20(2)	-10(2)	2(2)	-10(2)
O(85)	24(2)	22(2)	26(2)	-11(2)	-6(2)	-5(2)
O(86)	37(3)	17(2)	15(2)	-5(2)	-5(2)	-5(2)
C(87)	23(3)	23(3)	13(3)	-12(3)	-3(2)	5(3)
C(88)	14(3)	22(3)	16(3)	-5(3)	-6(2)	0(2)
C(89)	38(4)	27(4)	14(3)	-10(3)	-5(3)	0(3)
C(90)	23(3)	27(3)	18(3)	-7(3)	-5(3)	2(3)
C(91)	36(4)	29(4)	22(3)	-7(3)	-17(3)	-3(3)
C(92)	18(3)	36(4)	27(4)	-19(3)	-7(3)	11(3)
C(93)	27(3)	32(4)	18(3)	-14(3)	-4(3)	6(3)
C(94)	43(5)	32(4)	31(4)	-18(3)	-15(4)	1(4)
C(95)	59(5)	80(6)	20(4)	-22(4)	-23(4)	8(5)
C(96)	70(6)	75(6)	48(5)	-47(5)	-20(5)	9(5)
C(97)	59(6)	49(5)	52(5)	-28(4)	-29(4)	4(4)
O(98)	34(3)	60(3)	50(3)	-35(3)	-25(3)	5(3)
C(99)	53(5)	126(8)	57(5)	-64(6)	-44(4)	43(5)
C(100)	28(4)	44(4)	41(4)	-25(4)	-14(3)	5(3)
C(101)	43(5)	97(7)	46(5)	-42(5)	-1(4)	-23(5)
C(102)	46(5)	109(8)	40(5)	-43(5)	-5(4)	-25(5)
C(103)	78(8)	92(9)	96(10)	-54(8)	-13(7)	39(7)
C(104)	25(3)	28(4)	13(3)	-10(3)	-2(3)	-1(3)
C(105)	15(3)	26(3)	20(3)	-3(3)	-9(3)	0(3)
C(106)	36(4)	32(4)	24(4)	-11(3)	-10(3)	-5(3)
C(107)	23(3)	27(3)	16(3)	-5(3)	2(3)	-4(3)
C(108)	24(3)	33(4)	18(3)	-15(3)	-3(3)	-7(3)
C(109)	27(3)	28(3)	17(3)	-10(3)	-10(3)	5(3)
C(110)	43(4)	42(4)	24(4)	-8(3)	-8(3)	-2(3)
C(111)	64(6)	85(7)	18(4)	-1(4)	-5(4)	-1(5)
C(112)	119(9)	53(5)	72(7)	-28(5)	-65(6)	36(6)
C(113)	67(6)	46(5)	29(4)	-4(3)	-21(4)	-5(4)
O(114)	39(3)	26(3)	24(3)	0(2)	0(2)	-10(2)
C(115)	67(6)	32(5)	42(5)	-5(4)	-1(4)	-27(4)
C(116)	20(3)	48(5)	20(3)	-11(3)	-2(3)	-1(3)
C(117)	44(5)	70(6)	57(6)	-42(5)	-6(4)	-8(5)
C(118)	31(4)	81(6)	43(5)	-5(4)	-23(4)	-7(4)
C(119)	33(4)	54(5)	28(3)	-11(3)	-9(3)	0(3)
C(120)	16(3)	13(3)	27(3)	-5(3)	-9(3)	2(2)
C(121)	23(3)	17(3)	18(3)	-7(3)	-5(3)	-3(3)
C(122)	39(4)	27(4)	21(4)	-12(3)	-9(3)	0(3)
C(123)	36(4)	24(3)	9(3)	-1(3)	-4(3)	6(3)
C(124)	41(4)	31(4)	17(3)	-9(3)	-2(3)	-8(3)
C(125)	25(3)	36(4)	23(3)	-12(3)	-5(3)	5(3)

Table E9. continued...

C(126)	23(3)	25(3)	15(3)	-6(3)	-1(2)	-6(3)
C(127)	22(4)	61(5)	32(4)	-21(4)	-5(3)	-7(4)
C(128)	32(5)	91(7)	75(7)	-51(6)	-17(5)	-3(5)
C(129)	23(4)	142(10)	26(4)	-3(5)	-2(3)	15(5)
C(130)	31(4)	59(5)	66(6)	-38(5)	-6(4)	16(4)
O(131)	36(3)	48(3)	24(3)	-12(2)	9(2)	-14(3)
C(132)	40(4)	43(5)	70(6)	-14(5)	-3(4)	-1(4)
C(133)	45(4)	41(4)	15(3)	2(3)	-16(3)	5(3)
C(134)	66(5)	79(6)	26(4)	-21(4)	-30(4)	6(5)
C(135)	50(4)	61(6)	26(4)	9(4)	-11(3)	12(4)
C(136)	74(7)	71(7)	19(4)	1(4)	-10(4)	-8(5)
C(137)	25(3)	23(3)	19(3)	-10(3)	-13(3)	-2(3)
C(138)	27(3)	24(3)	21(3)	-8(3)	-11(3)	-4(3)
C(139)	21(3)	34(4)	30(4)	-16(3)	-14(3)	2(3)
C(140)	43(4)	21(3)	23(4)	-11(3)	-17(3)	-2(3)
C(141)	16(3)	25(3)	21(3)	-7(3)	-9(2)	3(3)
C(142)	36(4)	18(3)	17(3)	-10(3)	-11(3)	0(3)
C(143)	13(3)	74(6)	34(4)	-33(4)	-1(3)	-1(3)
C(144)	20(3)	126(9)	28(4)	-32(5)	9(3)	-6(4)
C(145)	42(5)	92(7)	69(6)	-52(6)	-17(4)	38(5)
C(146)	37(5)	104(8)	59(6)	-46(6)	10(4)	-31(5)
O(147)	32(3)	49(3)	39(3)	-21(3)	-21(2)	-1(2)
C(148)	43(4)	59(5)	73(6)	-25(5)	-37(4)	8(4)
C(149)	39(4)	34(4)	14(3)	-9(3)	-10(3)	-3(3)
C(150)	33(4)	36(3)	15(3)	-14(3)	-4(3)	0(3)
C(151)	62(5)	41(4)	31(4)	-15(3)	-19(4)	0(4)
C(152)	61(5)	31(4)	27(4)	-19(3)	-7(3)	-9(3)
C(201)	46(5)	48(5)	40(4)	-15(4)	13(3)	-18(4)
O(202)	42(3)	35(3)	57(4)	-23(3)	-18(3)	13(3)
C(203)	47(5)	43(4)	68(6)	-32(4)	-36(5)	23(4)
C(204)	102(8)	58(6)	90(8)	-41(6)	-76(7)	26(6)
O(205)	69(4)	21(2)	36(3)	-10(2)	-26(3)	6(3)
C(206)	129(9)	48(5)	19(4)	-3(4)	-2(5)	9(6)
C(301)	65(6)	35(4)	25(4)	-13(3)	-4(4)	1(4)
O(302)	54(3)	38(3)	35(3)	-8(3)	-22(3)	18(3)
C(303)	51(5)	46(5)	49(5)	-21(4)	-35(4)	13(4)
C(304)	40(4)	42(5)	73(6)	-29(4)	-34(4)	2(4)
O(305)	40(3)	42(3)	48(3)	-28(3)	-3(3)	-1(3)
C(306)	51(5)	76(6)	46(5)	-34(5)	24(4)	-23(5)
C(401)	105(7)	42(5)	48(5)	-14(4)	-38(5)	7(5)
O(402)	77(4)	47(3)	40(3)	-26(3)	-28(3)	10(3)
C(403)	56(5)	38(4)	51(5)	-30(4)	-34(4)	20(4)
C(404)	45(5)	47(5)	94(7)	-41(5)	-43(5)	12(4)
O(405)	22(2)	40(3)	46(3)	-18(3)	-11(2)	9(2)

Table E9. continued...

C(406)	20(3)	33(4)	72(5)	-25(4)	-6(3)	14(3)
C(901)	68(6)	42(5)	25(4)	-7(4)	-19(4)	22(4)
O(902)	44(3)	27(3)	27(3)	-12(2)	-11(2)	5(2)
C(903)	51(5)	32(4)	47(4)	-15(3)	-22(4)	2(4)
C(904)	30(4)	34(4)	54(5)	-20(4)	-7(3)	4(3)
O(905)	35(3)	34(3)	47(3)	-19(3)	-8(3)	-2(2)
C(906)	38(5)	74(6)	81(7)	-49(6)	-5(5)	11(4)

Table E10. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for $(1E^-)_2 \cdot (Na^+)_2 \cdot (C_4H_{10}O_2)_8$.

Atom	x	y	z	U_{eq}
H(13)	4481	6306	1334	30
H(17)	6792	5737	1688	28
H(19A)	5834	4188	4186	87
H(19B)	6812	4327	4133	87
H(19C)	6588	4079	3490	87
H(20A)	7533	6333	2288	78
H(20B)	7602	5340	2364	78
H(20C)	7839	5595	2997	78
H(21A)	5692	5826	4089	77
H(21B)	6421	6601	3369	77
H(21C)	6662	5810	4095	77
H(23A)	4022	4590	4942	151
H(23B)	4754	4202	4451	151
H(23C)	3846	4308	4299	151
H(25A)	3545	7105	1672	142
H(25B)	2572	6765	2252	142
H(25C)	3091	6175	1835	142
H(26A)	3785	7286	2834	92
H(26B)	3511	6454	3720	92
H(26C)	2793	6913	3322	92
H(27A)	2275	5328	3577	102
H(27B)	3056	4911	3891	102
H(27C)	2908	4810	3147	102
H(29)	4840	4799	1098	31
H(33)	6029	7039	-1044	28
H(35A)	2290	3758	1223	77
H(35B)	2540	4681	406	77
H(35C)	2570	4663	1237	77
H(36A)	3372	2965	573	62
H(36B)	4362	3368	133	62
H(36C)	3661	3844	-281	62
H(37A)	3373	2958	1834	66
H(37B)	3673	3826	1892	66
H(37C)	4365	3353	1458	66
H(39A)	2693	5810	-1265	85
H(39B)	3284	6628	-1368	85
H(39C)	2712	5912	-480	85
H(41A)	4185	7713	-1941	65
H(41B)	4932	8226	-2793	65
H(41C)	5036	8198	-1983	65

Table E10. continued...

H(42A)	6557	6752	-2418	80
H(42B)	6457	7581	-2198	80
H(42C)	6388	7698	-3048	80
H(43A)	4434	6227	-2320	78
H(43B)	5400	5992	-2508	78
H(43C)	5165	6906	-3124	78
H(46)	9812	6952	-99	30
H(50)	11022	9122	-2209	29
H(52A)	9392	6355	1079	73
H(52B)	9290	7164	1327	73
H(52C)	9463	6207	1937	73
H(53A)	11468	7530	1187	76
H(53B)	10673	6983	1989	76
H(53C)	10575	7937	1318	76
H(54A)	10901	5771	790	100
H(54B)	10844	5631	1681	100
H(54C)	11655	6225	915	100
H(56A)	13183	8032	57	85
H(56B)	13092	7970	-723	85
H(56C)	12530	7258	171	85
H(58A)	11507	10500	-1217	71
H(58B)	12479	10955	-1704	71
H(58C)	12260	10055	-869	71
H(59A)	13203	9193	-2321	61
H(59B)	13301	9274	-1551	61
H(59C)	13548	10136	-2417	61
H(60A)	11453	10520	-2548	76
H(60B)	12160	10069	-2993	76
H(60C)	12432	10962	-2957	76
H(62)	9015	8196	-2790	28
H(66)	11325	7657	-2418	31
H(68A)	9138	8022	-5166	86
H(68B)	9427	7265	-4456	86
H(68C)	10120	8057	-5202	86
H(69A)	8243	8499	-3464	62
H(69B)	8321	7537	-3459	62
H(69C)	8024	8323	-4146	62
H(70A)	9345	9913	-4703	110
H(70B)	8990	9631	-5265	110
H(70C)	9998	9720	-5405	110
H(72A)	11840	9819	-6052	305
H(72B)	11996	9757	-5249	305
H(72C)	11075	9932	-5366	305
H(74A)	12653	7673	-2849	70

Table E10. continued...

H(74B)	13342	7299	-3387	70
H(74C)	12424	6731	-2824	70
H(75A)	12913	9083	-4093	188
H(75B)	12817	9106	-4905	188
H(75C)	13573	8632	-4577	188
H(76A)	12305	7599	-4860	186
H(76B)	12162	6689	-4043	186
H(76C)	13106	7211	-4568	186
H(89)	7911	12018	2286	32
H(93)	9436	11630	377	30
H(95A)	9304	12101	3913	76
H(95B)	9960	11786	3301	76
H(95C)	8992	11314	3752	76
H(96A)	8078	12871	3595	85
H(96B)	7783	12085	3423	85
H(96C)	7905	13107	2757	85
H(97A)	9537	13739	2847	73
H(97B)	9378	14001	1999	73
H(97C)	10214	13547	2158	73
H(99A)	11652	11834	2255	96
H(99B)	11386	11385	1757	96
H(99C)	10752	11220	2633	96
H(10A)	11815	12881	493	91
H(10B)	11444	13572	-188	91
H(10C)	12205	13026	-427	91
H(10A)	11412	12625	-1089	94
H(10B)	10594	13087	-807	94
H(10C)	10482	12058	-630	94
H(10D)	11558	11091	874	132
H(10E)	12012	11447	-85	132
H(10F)	11088	10870	350	132
H(105)	7710	10263	2708	26
H(109)	6062	11198	1397	28
H(11A)	6060	8469	5464	99
H(11B)	5476	8335	4983	99
H(11C)	5528	9253	5055	99
H(11D)	7332	8070	4712	109
H(11E)	7762	8635	3749	109
H(11F)	6924	7935	4108	109
H(11G)	7322	9491	4852	76
H(11H)	6901	10332	4349	76
H(11I)	7748	10040	3886	76
H(11J)	4564	7649	4313	82
H(11K)	5585	7876	3996	82

Table E10. continued...

H(11L)	5072	8136	3357	82
H(11M)	4345	9236	2290	81
H(11N)	3451	9569	2612	81
H(11O)	3952	9012	3231	81
H(11P)	4864	10839	1226	86
H(11Q)	4791	11636	1489	86
H(11R)	3941	11087	1601	86
H(11S)	3940	10283	3662	62
H(11T)	3410	10797	3059	62
H(11U)	4274	11292	2981	62
H(122)	8125	13655	-3847	34
H(126)	9741	12631	-2504	27
H(12A)	11838	14915	-4365	91
H(12B)	12411	14347	-3826	91
H(12C)	11552	14669	-3418	91
H(12D)	11018	12255	-2664	117
H(12E)	11019	12999	-2346	117
H(12F)	11907	12746	-2786	117
H(13A)	11518	12676	-4181	76
H(13B)	12396	13192	-4309	76
H(13C)	11796	13703	-4839	76
H(13D)	11284	16264	-5451	88
H(13E)	10843	15778	-4484	88
H(13F)	10263	16035	-5066	88
H(13G)	8964	13561	-5396	81
H(13H)	8471	14322	-5899	81
H(13I)	8087	13804	-4927	81
H(13J)	8533	15888	-5816	85
H(13K)	9000	15951	-5220	85
H(13L)	8106	15330	-4852	85
H(13M)	10311	14605	-6120	95
H(13N)	10363	15556	-6106	95
H(13O)	9838	15392	-6620	95
H(137)	6401	12270	-1463	25
H(141)	7913	11953	-3434	26
H(14A)	5424	11896	-554	94
H(14B)	5205	10860	-309	94
H(14C)	4463	11414	-50	94
H(14D)	4830	13070	-1579	94
H(14E)	3894	12532	-1041	94
H(14F)	4260	12769	-1987	94
H(14G)	4470	10370	-1032	103
H(14H)	4026	11068	-1641	103
H(14I)	3658	10800	-689	103

Table E10. continued...

H(14J)	4096	11975	-3309	83
H(14K)	4974	12635	-3783	83
H(14L)	4395	12530	-2908	83
H(15A)	8057	11784	-4480	42
H(15B)	7756	11033	-4689	42
H(15C)	7890	10752	-3828	42
H(15D)	5832	12079	-4374	66
H(15E)	6471	11757	-4990	66
H(15F)	6789	12573	-4869	66
H(15G)	5606	10418	-3252	58
H(15H)	6433	9937	-3104	58
H(15I)	6250	10200	-3942	58
H(20D)	9233	10837	-1573	79
H(20E)	9615	9896	-1324	79
H(20F)	10094	10728	-1337	79
H(20G)	9951	9957	139	55
H(20H)	9626	9088	91	55
H(20I)	9127	8935	1474	83
H(20J)	8591	9766	1181	83
H(20K)	7020	7968	2039	114
H(20L)	7272	8882	2051	114
H(20M)	7760	8029	2400	114
H(30A)	7575	4676	862	67
H(30B)	8329	4572	1249	67
H(30C)	8108	3836	986	67
H(30A)	9859	4925	302	53
H(30B)	9636	4111	130	53
H(30C)	10780	5167	-901	56
H(30D)	10101	5877	-1081	56
H(30E)	10225	4995	-2507	96
H(30F)	11033	5275	-2328	96
H(30G)	10310	5932	-2474	96
H(40A)	8864	15947	-3119	96
H(40B)	8196	15856	-3543	96
H(40C)	8668	15015	-3126	96
H(40A)	7258	14129	-2253	48
H(40B)	6745	14964	-2610	48
H(40C)	6243	14910	-1257	64
H(40D)	5864	14052	-1288	64
H(40E)	6609	13131	413	65
H(40F)	5785	13106	144	65
H(40G)	6115	13999	147	65
H(50A)	-643	10016	2975	174
H(50B)	-772	8999	3124	174

Table E10. continued...

H(50C)	-348	9778	2221	174
H(50D)	3052	9663	1073	176
H(50E)	2126	9412	1064	176
H(50F)	2715	8640	1366	176
H(60D)	6372	3722	6075	126
H(40B)	8196	15856	-3543	96
H(40C)	8668	15015	-3126	96
H(40A)	7258	14129	-2253	48
H(40B)	6745	14964	-2610	48
H(40C)	6243	14910	-1257	64
H(40D)	5864	14052	-1288	64
H(40E)	6609	13131	413	65
H(40F)	5785	13106	144	65
H(40G)	6115	13999	147	65
H(50A)	-643	10016	2975	174
H(50B)	-772	8999	3124	174
H(50C)	-348	9778	2221	174
H(50D)	3052	9663	1073	176
H(50E)	2126	9412	1064	176
H(50F)	2715	8640	1366	176
H(60D)	6372	3722	6075	126
H(60E)	6176	4068	6760	126
H(60F)	6655	4746	5827	126
H(60K)	2781	3859	8021	162
H(60L)	2731	4906	7530	162
H(60M)	3509	4581	7881	162
H(90A)	8240	9251	-1919	71
H(90B)	7662	10057	-2098	71
H(90C)	7511	9259	-2315	71
H(90D)	6007	9002	-1479	51
H(90E)	6226	9848	-1343	51
H(90F)	4994	8763	-133	49
H(90G)	5645	8021	47	49
H(90H)	5594	8856	1401	94
H(90I)	5435	7968	1309	94
H(90J)	4787	8710	1152	94

Table E11. Crystal and structure refinement data for **2A•($C_4H_{11}NO_2$)$_2$**.

Identification code	**2A•($C_4H_{11}NO_2$)$_2$**
Empirical formula	$C_{40}H_{46}N_2O_8$
Formula weight	682.79 g/mole
Temperature	173(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	$P2_1/c$
Unit cell dimensions	a = 11.003(2) Å b = 18.114(4) Å c = 8.6118(17) Å α = 90.00° β = 91.23(3)° γ = 90.00°
Volume	1716.1(6) Å^3
Z	2
Density (calculated)	1.321 Mg/m^3
Absorption coefficient	0.092 mm^{-1}
F(000)	728
Crystal Size	0.54 mm x 0.28 mm x 0.14 mm
Color	wine red/purple
Theta range for data collection	1.85° to 28.29°
Index ranges	$-14 \le h \le 14$, $-19 \le k \le 23$, $-11 \le l \le 8$
Reflections collected / Unique	10837 / 4046 [R_{int} = 0.0438]
Refinement method	Full-matrix-least-squares on F^2
Data / Restraints / Parameters	4046 / 0 / 319
Goodness-of-fit on F^2	0.999
Final R indices [$I>2\sigma(I)$]	R_1 = 0.0436, wR_2 = 0.0903
R indices (all data)	R_1 = 0.0875, wR_2 = 0.1023
Extinction Coefficient	0.0091(15)
Largest diff. peak and hole	0.260 and -0.188 e/A^3

Table E12. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for **2A•$(C_4H_{11}NO_2)_2$**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U_{eq}
C(1)	5477(1)	649(1)	9261(2)	25(1)
O(1)	5745(1)	1259(1)	8639(1)	33(1)
C(2)	4137(1)	581(1)	9753(2)	23(1)
O(2)	3471(1)	1126(1)	9441(1)	32(1)
C(3)	3723(1)	-61(1)	10500(2)	25(1)
C(4)	2405(1)	-73(1)	11002(2)	27(1)
C(5)	2256(1)	144(1)	12698(2)	28(1)
C(6)	3099(2)	-40(1)	13860(2)	33(1)
C(7)	2918(2)	159(1)	15398(2)	39(1)
C(8)	1891(2)	546(1)	15798(2)	44(1)
C(9)	1058(2)	739(1)	14657(2)	47(1)
C(10)	1235(2)	543(1)	13120(2)	38(1)
C(11)	1697(1)	-772(1)	10548(2)	27(1)
C(12)	890(2)	-1110(1)	11535(2)	38(1)
C(13)	191(2)	-1710(1)	11045(2)	42(1)
C(14)	298(2)	-1985(1)	9573(2)	40(1)
C(15)	1103(2)	-1658(1)	8575(2)	41(1)
C(16)	1789(2)	-1058(1)	9055(2)	35(1)
N(20)	4839(1)	2174(1)	4127(2)	31(1)
C(20)	5940(2)	1693(1)	4212(2)	37(1)
C(21)	6947(2)	2046(1)	5149(2)	38(1)
O(21)	6573(1)	2230(1)	6665(1)	37(1)
C(22)	3882(2)	1852(1)	3069(2)	38(1)
C(23)	2920(2)	2406(1)	2663(2)	38(1)
O(23)	3428(1)	3013(1)	1860(1)	38(1)

Table E13. Bond lengths (Å) and angles (°) for **2A•$(C_4H_{11}NO_2)_2$**.

Atoms	Length	Angle
C(1)-O(1)	1.2658(17)	
C(1)-C(3)#1	1.395(2)	
C(1)-C(2)	1.548(2)	
C(2)-O(2)	1.2560(17)	
C(2)-C(3)	1.408(2)	
C(3)-C(1)#1	1.395(2)	
C(3)-C(4)	1.522(2)	
C(4)-C(5)	1.525(2)	
C(4)-C(11)	1.532(2)	
C(4)-H(4)	0.996(16)	
C(5)-C(6)	1.390(2)	
C(5)-C(10)	1.391(2)	
C(6)-C(7)	1.391(2)	
C(6)-H(6)	0.987(18)	
C(7)-C(8)	1.380(3)	
C(7)-H(7)	0.969(19)	
C(8)-C(9)	1.375(3)	
C(8)-H(8)	0.975(19)	
C(9)-C(10)	1.388(3)	
C(9)-H(9)	0.94(2)	
C(10)-H(10)	0.945(18)	
C(11)-C(12)	1.385(2)	
C(11)-C(16)	1.392(2)	
C(12)-C(13)	1.393(3)	
C(12)-H(12)	0.976(19)	
C(13)-C(14)	1.369(3)	
C(13)-H(13)	0.96(2)	
C(14)-C(15)	1.381(3)	
C(14)-H(14)	0.94(2)	
C(15)-C(16)	1.381(3)	
C(15)-H(15)	0.958(19)	
C(16)-H(16)	0.960(18)	
N(20)-C(20)	1.493(2)	
N(20)-C(22)	1.496(2)	
N(20)-H(20A)	0.97(2)	
N(20)-H(20B)	0.97(2)	
C(20)-C(21)	1.499(3)	
C(20)-H(20C)	0.984(18)	
C(20)-H(20D)	1.000(18)	
C(21)-O(21)	1.417(2)	
C(21)-H(21A)	0.962(18)	

Table E13. continued...

C(21)-H(21B)	1.023(17)
O(21)-H(21C)	0.94(2)
C(22)-C(23)	1.495(3)
C(22)-H(22A)	0.98(2)
C(22)-H(22B)	1.015(17)
C(23)-O(23)	1.420(2)
C(23)-H(23A)	1.031(18)
C(23)-H(23B)	0.979(18)
O(23)-H(23C)	1.00(3)

O(1)-C(1)-C(3)#1	125.26(13)
O(1)-C(1)-C(2)	114.72(13)
C(3)#1-C(1)-C(2)	120.01(13)
O(2)-C(2)-C(3)	123.62(13)
O(2)-C(2)-C(1)	115.66(13)
C(3)-C(2)-C(1)	120.71(13)
C(1)#1-C(3)-C(2)	119.23(13)
C(1)#1-C(3)-C(4)	123.31(13)
C(2)-C(3)-C(4)	117.46(13)
C(3)-C(4)-C(5)	112.99(13)
C(3)-C(4)-C(11)	114.93(13)
C(5)-C(4)-C(11)	113.11(12)
C(3)-C(4)-H(4)	104.1(9)
C(5)-C(4)-H(4)	105.0(9)
C(11)-C(4)-H(4)	105.5(9)
C(6)-C(5)-C(10)	117.80(16)
C(6)-C(5)-C(4)	122.99(14)
C(10)-C(5)-C(4)	119.21(16)
C(5)-C(6)-C(7)	121.06(16)
C(5)-C(6)-H(6)	121.1(10)
C(7)-C(6)-H(6)	117.9(10)
C(8)-C(7)-C(6)	120.33(19)
C(8)-C(7)-H(7)	119.8(11)
C(6)-C(7)-H(7)	119.9(11)
C(9)-C(8)-C(7)	119.16(18)
C(9)-C(8)-H(8)	121.3(11)
C(7)-C(8)-H(8)	119.5(11)
C(8)-C(9)-C(10)	120.77(18)
C(8)-C(9)-H(9)	122.4(12)
C(10)-C(9)-H(9)	116.8(12)
C(9)-C(10)-C(5)	120.88(19)
C(9)-C(10)-H(10)	121.8(11)
C(5)-C(10)-H(10)	117.2(11)
C(12)-C(11)-C(16)	117.53(16)

Table E13. continued...

C(12)-C(11)-C(4)	122.52(15)
C(16)-C(11)-C(4)	119.79(14)
C(11)-C(12)-C(13)	121.12(17)
C(11)-C(12)-H(12)	119.1(11)
C(13)-C(12)-H(12)	119.8(11)
C(14)-C(13)-C(12)	120.43(17)
C(14)-C(13)-H(13)	119.1(12)
C(12)-C(13)-H(13)	120.4(12)
C(13)-C(14)-C(15)	119.28(18)
C(13)-C(14)-H(14)	122.0(11)
C(15)-C(14)-H(14)	118.7(11)
C(16)-C(15)-C(14)	120.35(18)
C(16)-C(15)-H(15)	119.5(12)
C(14)-C(15)-H(15)	120.2(12)
C(15)-C(16)-C(11)	121.29(16)
C(15)-C(16)-H(16)	120.3(11)
C(11)-C(16)-H(16)	118.4(11)
C(20)-N(20)-C(22)	111.19(14)
C(20)-N(20)-H(20A)	107.8(11)
C(22)-N(20)-H(20A)	109.8(11)
C(20)-N(20)-H(20B)	112.6(11)
C(22)-N(20)-H(20B)	107.1(11)
H(20A)-N(20)-H(20B)	108.3(16)
N(20)-C(20)-C(21)	111.49(15)
N(20)-C(20)-H(20C)	105.3(10)
C(21)-C(20)-H(20C)	112.2(10)
N(20)-C(20)-H(20D)	105.5(9)
C(21)-C(20)-H(20D)	112.0(10)
H(20C)-C(20)-H(20D)	109.9(15)
O(21)-C(21)-C(20)	111.79(14)
O(21)-C(21)-H(21A)	105.4(10)
C(20)-C(21)-H(21A)	108.7(10)
O(21)-C(21)-H(21B)	111.0(9)
C(20)-C(21)-H(21B)	108.4(9)
H(21A)-C(21)-H(21B)	111.5(13)
C(21)-O(21)-H(21C)	107.9(14)
C(23)-C(22)-N(20)	111.46(15)
C(23)-C(22)-H(22A)	111.8(11)
N(20)-C(22)-H(22A)	106.8(11)
C(23)-C(22)-H(22B)	109.0(10)
N(20)-C(22)-H(22B)	106.3(10)
H(22A)-C(22)-H(22B)	111.3(15)
O(23)-C(23)-C(22)	110.46(14)
O(23)-C(23)-H(23A)	109.5(10)

Table E13. continued...

C(22)-C(23)-H(23A)	110.3(10)
O(23)-C(23)-H(23B)	109.8(10)
C(22)-C(23)-H(23B)	109.1(10)
H(23A)-C(23)-H(23B)	107.7(14)
C(23)-O(23)-H(23C)	106.0(14)

Symmetry transformations used to generate equivalent atoms:
#1 -x+1, -y, -z+2

Table E14. Anisotropic displacement parameters ($Å^2 \times 10^3$) for **2A•($C_4H_{11}NO_2$)$_2$**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+...+2hka^*b^*U_{12}]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(1)	27(1)	25(1)	23(1)	2(1)	6(1)	-2(1)
O(1)	31(1)	25(1)	42(1)	10(1)	12(1)	1(1)
C(2)	26(1)	24(1)	21(1)	-1(1)	4(1)	1(1)
O(2)	29(1)	27(1)	41(1)	9(1)	9(1)	5(1)
C(3)	24(1)	26(1)	24(1)	2(1)	6(1)	0(1)
C(4)	24(1)	26(1)	31(1)	5(1)	7(1)	2(1)
C(5)	27(1)	23(1)	36(1)	0(1)	12(1)	-5(1)
C(6)	37(1)	27(1)	36(1)	2(1)	11(1)	2(1)
C(7)	51(1)	31(1)	35(1)	-2(1)	6(1)	-4(1)
C(8)	56(1)	38(1)	41(1)	-10(1)	20(1)	-9(1)
C(9)	39(1)	46(1)	56(1)	-15(1)	19(1)	1(1)
C(10)	29(1)	41(1)	46(1)	-8(1)	9(1)	-1(1)
C(11)	22(1)	26(1)	32(1)	5(1)	4(1)	3(1)
C(12)	37(1)	44(1)	34(1)	0(1)	8(1)	-11(1)
C(13)	37(1)	44(1)	46(1)	6(1)	5(1)	-14(1)
C(14)	33(1)	34(1)	51(1)	-2(1)	-8(1)	-4(1)
C(15)	38(1)	44(1)	40(1)	-8(1)	0(1)	3(1)
C(16)	30(1)	38(1)	36(1)	1(1)	8(1)	0(1)
N(20)	34(1)	28(1)	30(1)	2(1)	-1(1)	-4(1)
C(20)	41(1)	33(1)	37(1)	4(1)	3(1)	6(1)
C(21)	36(1)	44(1)	33(1)	8(1)	7(1)	3(1)
O(21)	45(1)	34(1)	33(1)	5(1)	8(1)	-5(1)
C(22)	46(1)	29(1)	38(1)	-1(1)	-9(1)	-7(1)
C(23)	32(1)	40(1)	42(1)	-2(1)	-4(1)	-6(1)
O(23)	49(1)	31(1)	34(1)	-1(1)	-2(1)	5(1)

Table E15. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for **2A•$(C_4H_{11}NO_2)_2$**.

Atom	x	y	z	U_{eq}
H(4)	2015(13)	334(9)	10395(17)	27(4)
H(6)	3852(16)	-310(10)	13620(20)	44(5)
H(7)	3520(17)	31(10)	16190(20)	46(5)
H(8)	1767(16)	674(10)	16880(20)	46(5)
H(9)	353(18)	1008(11)	14870(20)	54(6)
H(10)	658(16)	656(10)	12330(20)	42(5)
H(12)	826(16)	-927(10)	12600(20)	51(5)
H(13)	-375(18)	-1935(11)	11730(20)	56(6)
H(14)	-137(17)	-2400(11)	9230(20)	50(5)
H(15)	1188(17)	-1844(11)	7540(20)	55(6)
H(16)	2325(16)	-819(10)	8350(20)	42(5)
H(20A)	5086(17)	2653(12)	3740(20)	56(6)
H(20B)	4473(17)	2243(10)	5130(20)	53(6)
H(20C)	6166(16)	1610(10)	3130(20)	47(5)
H(20D)	5664(15)	1219(10)	4684(19)	39(5)
H(21A)	7154(14)	2507(10)	4670(19)	35(5)
H(21B)	7671(15)	1692(10)	5187(18)	36(4)
H(21C)	6470(20)	1790(14)	7220(30)	79(8)
H(22A)	3549(17)	1423(11)	3600(20)	53(5)
H(22B)	4310(15)	1700(10)	2090(20)	40(5)
H(23A)	2511(16)	2592(10)	3650(20)	41(5)
H(23B)	2294(16)	2170(10)	2010(20)	41(5)
H(23C)	3620(20)	3397(14)	2670(30)	89(8)

Table E16. Torsion angles, ° for **2A•$(C_4H_{11}NO_2)_2$**.

Atoms	Angle
O(1) C(1) C(2) O(2)	1.88(19)
C(3)#1 C(1) C(2) O(2)	-177.44(13)
O(1) C(1) C(2) C(3)	-178.17(14)
C(3)#1 C(1) C(2) C(3)	2.5(2)
O(2) C(2) C(3) C(1)#1	177.46(14)
C(1) C(2) C(3) C(1)#1	-2.5(2)
O(2) C(2) C(3) C(4)	-2.3(2)
C(1) C(2) C(3) C(4)	177.71(13)
C(1)#1 C(3) C(4) C(5)	84.07(19)
C(2) C(3) C(4) C(5)	-96.15(16)
C(1)#1 C(3) C(4) C(11)	-47.8(2)
C(2) C(3) C(4) C(11)	131.99(15)
C(3) C(4) C(5) C(6)	-35.4(2)
C(11) C(4) C(5) C(6)	97.40(17)
C(3) C(4) C(5) C(10)	144.72(15)
C(11) C(4) C(5) C(10)	-82.53(18)
C(10) C(5) C(6) C(7)	1.0(2)
C(4) C(5) C(6) C(7)	-178.97(15)
C(5) C(6) C(7) C(8)	-0.1(3)
C(6) C(7) C(8) C(9)	-0.6(3)
C(7) C(8) C(9) C(10)	0.5(3)
C(8) C(9) C(10) C(5)	0.4(3)
C(6) C(5) C(10) C(9)	-1.1(2)
C(4) C(5) C(10) C(9)	178.85(16)
C(3) C(4) C(11) C(12)	139.48(16)
C(5) C(4) C(11) C(12)	7.7(2)
C(3) C(4) C(11) C(16)	-45.2(2)
C(5) C(4) C(11) C(16)	-176.97(14)
C(16) C(11) C(12) C(13)	-0.5(3)
C(4) C(11) C(12) C(13)	174.99(16)
C(11) C(12) C(13) C(14)	0.7(3)
C(12) C(13) C(14) C(15)	-0.3(3)
C(13) C(14) C(15) C(16)	-0.2(3)
C(14) C(15) C(16) C(11)	0.5(3)
C(12) C(11) C(16) C(15)	-0.1(3)
C(4) C(11) C(16) C(15)	-175.68(16)
C(22) N(20) C(20) C(21)	174.59(15)
N(20) C(20) C(21) O(21)	56.3(2)
C(20) N(20) C(22) C(23)	-165.74(15)
N(20) C(22) C(23) O(23)	61.8(2)

Table E16. continued...

Symmetry transformations used to generate equivalent atoms:
#1 -x+1, -y, -z+2

Table E17. Hydrogen bonds, lengths (Å) and angles (°) for **2A•($C_4H_{11}NO_2$)$_2$**.

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(20)-H(20A)...O(1)#2	0.97(2)	2.10(2)	3.0405(19)	162.3(16)
N(20)-H(20A)...O(21)#2	0.97(2)	2.460(19)	3.078(2)	121.4(15)
N(20)-H(20B)...O(23)#3	0.97(2)	1.96(2)	2.867(2)	155.6(16)
O(21)-H(21C)...O(1)	0.94(2)	1.76(2)	2.6230(16)	152(2)
O(23)-H(23C)...O(2)#2	1.00(3)	1.76(3)	2.7151(17)	157(2)
O(23)-H(23C)...O(1)#2	1.00(3)	2.55(2)	3.2275(18)	124.8(17)

Symmetry transformations used to generate equivalent atoms:
#2 x, -y+1/2, z-1/2
#3 x, -y+1/2, z+1/2

Table E

Identifica	
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Density	
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Crystal S	
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Goodnes	
Final R in	
R indices	
Extinctio	
Largest d	

Table E18. Crystal and structure refinement data for $2A^{2-}\bullet([HgC_6H_5]^+)_2\bullet(C_4H_8O)$.

Identification code	$2A^{2-}\bullet([HgC_6H_5]^+)_2\bullet(C_4H_8O)$
Empirical formula	$C_{24}H_{20}HgO_{2.50}$
Formula weight	548.99 g/mole
Temperature	173(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	C2/c
Unit cell dimensions	a = 18.474(4) Å b = 10.966(2) Å c = 21.211(4) Å α = 90.00° β = 113.81(3)° γ = 90.00°
Volume	3931.1(14) $Å^3$
Z	8
Density (calculated)	1.855 Mg/m^3
Absorption coefficient	7.849 mm^{-1}
F(000)	2112
Crystal Size	0.46 mm x 0.24 mm x 0.06 mm
Color	purple
Theta range for data collection	2.10° to 28.31°
Index ranges	$-24 \le h \le 24$, $-14 \le k \le 14$, $-28 \le l \le 28$
Reflections collected / Unique	23162 / 4784 [R_{int} = 0.0544]
Refinement method	Full-matrix-least-squares on F^2
Data / Restraints / Parameters	4784 / 128 / 310
Goodness-of-fit on F^2	1.052
Final R indices [$I>2\sigma(I)$]	R_1 = 0.0330, wR_2 = 0.0700
R indices (all data)	R_1 = 0.0489, wR_2 = 0.0733
Extinction Coefficient	0.00012(5)
Largest diff. peak and hole	2.200 and -1.032 e/A^3

Table E19. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3), and occupancies for $2A^{2-}\bullet([HgC_6H_5]^+)_2\bullet(C_4H_8O)$. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U_{eq}	Occ.
Hg(1)	728(1)	161(1)	4415(1)	33(1)	1
O(1)	1939(4)	-181(5)	4750(3)	30(1)	1
C(1)	2236(5)	-1256(7)	4870(4)	25(2)	1
O(2)	957(3)	-2087(5)	4570(3)	33(1)	1
C(2)	1675(5)	-2334(7)	4791(4)	24(2)	1
C(3)	1968(5)	-3552(7)	4948(4)	24(2)	1
C(4)	1384(5)	-4579(7)	4857(5)	26(2)	1
C(5)	1467(5)	-5235(7)	5518(5)	28(2)	1
C(6)	2110(6)	-5089(8)	6144(5)	35(2)	1
C(7)	2139(6)	-5683(9)	6731(5)	40(2)	1
C(8)	1520(6)	-6405(9)	6709(6)	45(2)	1
C(9)	870(6)	-6549(9)	6088(6)	42(2)	1
C(10)	847(5)	-5972(8)	5502(5)	33(2)	1
C(11)	1363(5)	-5445(7)	4284(4)	28(2)	1
C(12)	1651(6)	-6622(8)	4402(5)	36(2)	1
C(13)	1638(6)	-7363(9)	3861(5)	43(2)	1
C(14)	1331(6)	-6928(10)	3200(6)	50(3)	1
C(15)	1039(7)	-5772(11)	3071(6)	52(3)	1
C(16)	1047(6)	-5022(8)	3607(5)	40(2)	1
C(20)	-404(5)	791(9)	4052(5)	36(2)	1
C(21)	-1020(7)	31(11)	3996(6)	50(3)	1
C(22)	-1790(7)	505(17)	3740(7)	69(4)	1
C(23)	-1931(9)	1692(19)	3557(7)	88(6)	1
C(24)	-1337(9)	2427(14)	3599(6)	68(4)	1
C(25)	-567(7)	2021(10)	3841(5)	47(3)	1
C(50)	15(19)	306(17)	2570(20)	66(11)	0.50
O(51)	733(10)	-195(14)	3067(9)	60(5)	0.50
C(52)	787(12)	-1388(17)	2821(12)	60(9)	0.50
C(53)	-82(13)	-1890(20)	2450(20)	80(20)	0.50
C(54)	-592(11)	-724(19)	2305(15)	78(8)	0.50

Table E20. Bond lengths (Å) and angles (°) for $2A^{2-}\cdot([HgC_6H_5]^+)_2\cdot(C_4H_8O)$.

Atoms	Length	Angle
Hg(1)-C(20)	2.035(8)	
Hg(1)-O(1)	2.091(6)	
Hg(1)-O(2)	2.500(6)	
O(1)-C(1)	1.282(9)	
C(1)-C(3)#1	1.376(11)	
C(1)-C(2)	1.536(11)	
O(2)-C(2)	1.246(9)	
C(2)-C(3)	1.429(11)	
C(3)-C(1)#1	1.376(11)	
C(3)-C(4)	1.519(11)	
C(4)-C(5)	1.527(12)	
C(4)-C(11)	1.530(12)	
C(4)-H(4)	0.98(8)	
C(5)-C(6)	1.388(13)	
C(5)-C(10)	1.391(11)	
C(6)-C(7)	1.388(14)	
C(6)-H(6)	0.9300	
C(7)-C(8)	1.375(13)	
C(7)-H(7)	0.9300	
C(8)-C(9)	1.387(14)	
C(8)-H(8)	0.9300	
C(9)-C(10)	1.381(13)	
C(9)-H(9)	0.9300	
C(10)-H(10)	0.9300	
C(11)-C(12)	1.379(12)	
C(11)-C(16)	1.394(13)	
C(12)-C(13)	1.399(13)	
C(12)-H(12)	0.9300	
C(13)-C(14)	1.369(15)	
C(13)-H(13)	0.9300	
C(14)-C(15)	1.361(16)	
C(14)-H(14)	0.9300	
C(15)-C(16)	1.399(15)	
C(15)-H(15)	0.9300	
C(16)-H(16)	0.9300	
C(20)-C(21)	1.376(15)	
C(20)-C(25)	1.415(14)	
C(21)-C(22)	1.401(17)	
C(21)-H(21)	0.9300	
C(22)-C(23)	1.35(2)	
C(22)-H(22)	0.9300	

Table E20. continued...

C(23)-C(24)	1.34(2)
C(23)-H(23)	0.9300
C(24)-C(25)	1.377(15)
C(24)-H(24)	0.9300
C(25)-H(25)	0.9300
C(50)-C(50)#2	0.28(10)
C(50)-O(51)	1.43(3)
C(50)-C(54)#2	1.50(4)
C(50)-C(54)	1.529(13)
C(50)-O(51)#2	1.59(4)
C(50)-H(50A)	0.989(16)
C(50)-H(50B)	0.974(17)
O(51)-C(54)#2	0.93(3)
O(51)-C(52)	1.43(2)
O(51)-C(50)#2	1.59(4)
C(52)-C(54)#2	0.81(2)
C(52)-H(52A)	1.006(17)
C(52)-H(52B)	1.033(14)
C(54)-C(52)#2	0.81(2)
C(54)-O(51)#2	0.93(3)
C(54)-C(50)#2	1.50(4)
C(54)-C(54)#2	2.00(4)
C(54)-H(54A)	1.031(16)
C(54)-H(54B)	0.974(14)

C(20)-Hg(1)-O(1)	170.0(3)
C(20)-Hg(1)-O(2)	118.6(3)
O(1)-Hg(1)-O(2)	71.2(2)
C(1)-O(1)-Hg(1)	123.1(5)
O(1)-C(1)-C(3)#1	121.3(7)
O(1)-C(1)-C(2)	118.1(7)
C(3)#1-C(1)-C(2)	120.6(7)
C(2)-O(2)-Hg(1)	111.2(5)
O(2)-C(2)-C(3)	122.5(7)
O(2)-C(2)-C(1)	116.3(7)
C(3)-C(2)-C(1)	121.2(7)
C(1)#1-C(3)-C(2)	118.0(7)
C(1)#1-C(3)-C(4)	123.1(7)
C(2)-C(3)-C(4)	118.7(7)
C(3)-C(4)-C(5)	115.5(7)
C(3)-C(4)-C(11)	110.4(7)
C(5)-C(4)-C(11)	113.5(7)
C(3)-C(4)-H(4)	105(5)
C(5)-C(4)-H(4)	110(4)

Table E20. continued...

C(11)-C(4)-H(4)	101(5)
C(6)-C(5)-C(10)	118.0(8)
C(6)-C(5)-C(4)	123.6(8)
C(10)-C(5)-C(4)	118.3(8)
C(7)-C(6)-C(5)	120.8(9)
C(7)-C(6)-H(6)	119.7
C(5)-C(6)-H(6)	119.5
C(8)-C(7)-C(6)	120.6(10)
C(8)-C(7)-H(7)	119.6
C(6)-C(7)-H(7)	119.7
C(7)-C(8)-C(9)	119.2(10)
C(7)-C(8)-H(8)	120.4
C(9)-C(8)-H(8)	120.4
C(10)-C(9)-C(8)	120.1(9)
C(10)-C(9)-H(9)	120.0
C(8)-C(9)-H(9)	119.9
C(9)-C(10)-C(5)	121.3(9)
C(9)-C(10)-H(10)	119.5
C(5)-C(10)-H(10)	119.3
C(12)-C(11)-C(16)	117.9(8)
C(12)-C(11)-C(4)	123.6(8)
C(16)-C(11)-C(4)	118.5(8)
C(11)-C(12)-C(13)	121.2(9)
C(11)-C(12)-H(12)	119.4
C(13)-C(12)-H(12)	119.4
C(14)-C(13)-C(12)	120.0(10)
C(14)-C(13)-H(13)	119.9
C(12)-C(13)-H(13)	120.1
C(15)-C(14)-C(13)	119.8(10)
C(15)-C(14)-H(14)	120.0
C(13)-C(14)-H(14)	120.2
C(14)-C(15)-C(16)	120.7(11)
C(14)-C(15)-H(15)	119.7
C(16)-C(15)-H(15)	119.6
C(11)-C(16)-C(15)	120.3(9)
C(11)-C(16)-H(16)	119.8
C(15)-C(16)-H(16)	119.9
C(21)-C(20)-C(25)	119.2(9)
C(21)-C(20)-Hg(1)	120.9(8)
C(25)-C(20)-Hg(1)	119.9(8)
C(20)-C(21)-C(22)	118.9(12)
C(20)-C(21)-H(21)	120.4
C(22)-C(21)-H(21)	120.7
C(23)-C(22)-C(21)	121.0(14)

Table E20. continued...

C(23)-C(22)-H(22)	120.0
C(21)-C(22)-H(22)	119.0
C(24)-C(23)-C(22)	120.3(12)
C(24)-C(23)-H(23)	120.4
C(22)-C(23)-H(23)	119.4
C(23)-C(24)-C(25)	121.8(14)
C(23)-C(24)-H(24)	119.2
C(25)-C(24)-H(24)	119.0
C(24)-C(25)-C(20)	118.8(12)
C(24)-C(25)-H(25)	120.8
C(20)-C(25)-H(25)	120.3
C(50)#2-C(50)-O(51)	120(10)
C(50)#2-C(50)-C(54)#2	90(10)
O(51)-C(50)-C(54)#2	36.8(13)
C(50)#2-C(50)-C(54)	79(9)
O(51)-C(50)-C(54)	108.1(15)
C(54)#2-C(50)-C(54)	82.6(16)
C(50)#2-C(50)-O(51)#2	51(10)
O(51)-C(50)-O(51)#2	136(2)
C(54)#2-C(50)-O(51)#2	102(2)
C(54)-C(50)-O(51)#2	34.6(12)
C(50)#2-C(50)-H(50A)	40(4)
O(51)-C(50)-H(50A)	121(4)
C(54)#2-C(50)-H(50A)	112(4)
C(54)-C(50)-H(50A)	114.4(17)
O(51)#2-C(50)-H(50A)	80(2)
C(50)#2-C(50)-H(50B)	147(7)
O(51)-C(50)-H(50B)	85(4)
C(54)#2-C(50)-H(50B)	120(4)
C(54)-C(50)-H(50B)	114.5(16)
O(51)#2-C(50)-H(50B)	125(3)
H(50A)-C(50)-H(50B)	110(2)
C(54)#2-O(51)-C(52)	32.1(19)
C(54)#2-O(51)-C(50)	76(2)
C(52)-O(51)-C(50)	105(2)
C(54)#2-O(51)-C(50)#2	69(2)
C(52)-O(51)-C(50)#2	99.8(17)
C(50)-O(51)-C(50)#2	9(4)
C(54)#2-C(52)-O(51)	38(2)
C(54)#2-C(52)-H(52A)	100(5)
O(51)-C(52)-H(52A)	117(4)
C(54)#2-C(52)-H(52B)	131(5)
O(51)-C(52)-H(52B)	94(4)
H(52A)-C(52)-H(52B)	112(2)

Table E20. continued...

C(52)#2-C(54)-O(51)#2	110(4)
C(52)#2-C(54)-C(50)#2	158(4)
O(51)#2-C(54)-C(50)#2	67.4(19)
C(52)#2-C(54)-C(50)	162(3)
O(51)#2-C(54)-C(50)	76(3)
C(50)#2-C(54)-C(50)	11(4)
C(52)#2-C(54)-C(54)#2	114(3)
O(51)#2-C(54)-C(54)#2	104(2)
C(50)#2-C(54)-C(54)#2	49.3(11)
C(50)-C(54)-C(54)#2	48.1(14)
C(52)#2-C(54)-H(54A)	77(3)
O(51)#2-C(54)-H(54A)	33(3)
C(50)#2-C(54)-H(54A)	100(3)
C(50)-C(54)-H(54A)	109.4(13)
C(54)#2-C(54)-H(54A)	124(2)
C(52)#2-C(54)-H(54B)	81(4)
O(51)#2-C(54)-H(54B)	115(3)
C(50)#2-C(54)-H(54B)	121(4)
C(50)-C(54)-H(54B)	112.3(14)
C(54)#2-C(54)-H(54B)	130(3)
H(54A)-C(54)-H(54B)	106(2)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1/2, -y-1/2, -z+1

#2 -x, y, -z+1/2

Table E21. Anisotropic displacement parameters ($Å^2 \times 10^3$) for $2A^{2-} \cdot ([HgC_6H_5]^+)_2 \cdot (C_4H_8O)$. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+...+2hka^*b^*U_{12}]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Hg(1)	27(1)	38(1)	33(1)	1(1)	9(1)	9(1)
O(1)	23(3)	27(3)	38(4)	0(2)	9(3)	3(2)
C(1)	21(4)	32(4)	20(5)	-2(3)	7(3)	-2(3)
O(2)	22(3)	30(3)	41(4)	2(3)	7(3)	2(2)
C(2)	24(4)	30(4)	19(4)	-2(3)	8(4)	-2(3)
C(3)	20(4)	31(4)	18(4)	-2(3)	4(3)	-1(3)
C(4)	21(4)	28(4)	26(5)	2(3)	5(4)	0(3)
C(5)	28(4)	29(4)	30(5)	-3(3)	14(4)	1(3)
C(6)	29(5)	40(5)	35(5)	-2(4)	13(4)	-4(4)
C(7)	38(6)	47(5)	28(6)	5(4)	6(5)	2(4)
C(8)	46(6)	53(6)	38(7)	13(5)	20(5)	2(4)
C(9)	31(5)	49(6)	48(7)	8(5)	18(5)	-5(4)
C(10)	24(5)	35(5)	37(6)	-2(4)	9(4)	-2(3)
C(11)	20(4)	34(4)	24(5)	-3(3)	4(4)	-7(3)
C(12)	40(5)	34(5)	32(6)	-1(4)	12(5)	0(4)
C(13)	51(6)	39(5)	40(6)	-8(4)	18(5)	-4(4)
C(14)	47(7)	64(7)	38(7)	-20(5)	17(5)	-10(5)
C(15)	51(7)	72(8)	23(6)	0(5)	6(5)	-12(6)
C(16)	41(6)	40(6)	31(6)	0(4)	6(4)	-1(4)
C(20)	27(5)	50(6)	28(5)	-6(4)	8(4)	12(4)
C(21)	38(6)	74(8)	36(6)	-14(5)	14(5)	-1(5)
C(22)	32(6)	135(12)	37(7)	-29(8)	11(5)	-2(8)
C(23)	45(8)	175(18)	29(8)	-19(9)	-1(6)	56(10)
C(24)	70(10)	83(10)	37(7)	0(6)	9(7)	53(8)
C(25)	49(7)	55(6)	34(6)	0(5)	13(5)	21(5)
C(50)	100(20)	63(14)	35(16)	-4(18)	24(14)	-30(20)
O(51)	53(10)	64(10)	52(11)	-20(8)	8(8)	5(7)
C(52)	110(30)	24(10)	63(18)	-10(10)	52(19)	7(13)
C(53)	80(20)	60(20)	70(50)	-40(30)	-10(20)	46(18)

Table E22. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3), and occupancies for $2A^{2-}\bullet([HgC_6H_5]^+)_2\bullet(C_4H_8O)$.

Atom	x	y	z	U_{eq}	Occ.
H(4)	860(50)	-4200(70)	4640(40)	20(20)	1
H(6)	2528	-4588	6170	40(30)	1
H(7)	2581	-5592	7144	50(30)	1
H(8)	1538	-6791	7105	50(30)	1
H(9)	448	-7036	6067	50(30)	1
H(10)	409	-6079	5089	30(20)	1
H(12)	1857	-6928	4849	60(40)	1
H(13)	1837	-8153	3950	40(30)	1
H(14)	1323	-7420	2840	50(30)	1
H(15)	831	-5478	2621	130(70)	1
H(16)	841	-4237	3511	20(20)	1
H(21)	-926	-783	4126	40(30)	1
H(22)	-2210	-5	3695	230(140)	1
H(23)	-2444	1996	3401	130(60)	1
H(24)	-1447	3236	3460	70(50)	1
H(25)	-161	2547	3866	40(30)	1
H(50A)	20(40)	870(40)	2210(40)	79	0.50
H(50B)	-10(40)	760(50)	2960(30)	79	0.50
H(52A)	1100(20)	-1480(70)	2530(30)	72	0.50
H(52B)	1080(30)	-1750(60)	3307(17)	72	0.50
H(53A)	-170(20)	-2400(40)	2010(40)	97	0.50
H(53B)	-196(18)	-2480(30)	2730(40)	97	0.50
H(54A)	-940(20)	-580(30)	1790(20)	93	0.50
H(54B)	-950(30)	-740(40)	2540(30)	93	0.50

Table E23. Torsion angles (°) for $2A^{2-}\bullet([HgC_6H_5]^+)_2\bullet(C_4H_8O)$.

Atoms	Angle
C(20) Hg(1) O(1) C(1)	-166.8(16)
O(2) Hg(1) O(1) C(1)	0.3(6)
Hg(1) O(1) C(1) C(3)#1	176.3(6)
Hg(1) O(1) C(1) C(2)	-2.4(10)
C(20) Hg(1) O(2) C(2)	179.6(6)
O(1) Hg(1) O(2) C(2)	2.1(6)
Hg(1) O(2) C(2) C(3)	177.7(6)
Hg(1) O(2) C(2) C(1)	-3.8(9)
O(1) C(1) C(2) O(2)	4.4(12)
C(3)#1 C(1) C(2) O(2)	-174.3(8)
O(1) C(1) C(2) C(3)	-177.0(8)
C(3)#1 C(1) C(2) C(3)	4.3(13)
O(2) C(2) C(3) C(1)#1	174.3(8)
C(1) C(2) C(3) C(1)#1	-4.1(13)
O(2) C(2) C(3) C(4)	-1.4(12)
C(1) C(2) C(3) C(4)	-179.9(7)
C(1)#1 C(3) C(4) C(5)	69.5(11)
C(2) C(3) C(4) C(5)	-115.0(8)
C(1)#1 C(3) C(4) C(11)	-60.9(11)
C(2) C(3) C(4) C(11)	114.6(8)
C(3) C(4) C(5) C(6)	-11.9(12)
C(11) C(4) C(5) C(6)	117.0(9)
C(3) C(4) C(5) C(10)	164.8(7)
C(11) C(4) C(5) C(10)	-66.3(10)
C(10) C(5) C(6) C(7)	1.3(13)
C(4) C(5) C(6) C(7)	178.0(8)
C(5) C(6) C(7) C(8)	-1.6(15)
C(6) C(7) C(8) C(9)	1.0(15)
C(7) C(8) C(9) C(10)	0.0(15)
C(8) C(9) C(10) C(5)	-0.3(14)
C(6) C(5) C(10) C(9)	-0.3(12)
C(4) C(5) C(10) C(9)	-177.2(8)
C(3) C(4) C(11) C(12)	110.7(9)
C(5) C(4) C(11) C(12)	-20.7(11)
C(3) C(4) C(11) C(16)	-68.4(10)
C(5) C(4) C(11) C(16)	160.1(8)
C(16) C(11) C(12) C(13)	1.0(14)
C(4) C(11) C(12) C(13)	-178.2(8)
C(11) C(12) C(13) C(14)	-0.4(15)
C(12) C(13) C(14) C(15)	0.0(16)
C(13) C(14) C(15) C(16)	-0.1(17)

Table E23. continued...

C(12) C(11) C(16) C(15)	-1.1(14)
C(4) C(11) C(16) C(15)	178.1(9)
C(14) C(15) C(16) C(11)	0.6(16)
O(1) Hg(1) C(20) C(21)	176.3(14)
O(2) Hg(1) C(20) C(21)	10.3(9)
O(1) Hg(1) C(20) C(25)	-3(2)
O(2) Hg(1) C(20) C(25)	-168.6(7)
C(25) C(20) C(21) C(22)	-0.8(16)
Hg(1) C(20) C(21) C(22)	-179.7(8)
C(20) C(21) C(22) C(23)	-0.8(18)
C(21) C(22) C(23) C(24)	2(2)
C(22) C(23) C(24) C(25)	-2(2)
C(23) C(24) C(25) C(20)	-0.1(18)
C(21) C(20) C(25) C(24)	1.3(15)
Hg(1) C(20) C(25) C(24)	-179.8(8)
C(50)#2 C(50) O(51) C(54)#2	-39(8)
C(54) C(50) O(51) C(54)#2	49(3)
O(51)#2 C(50) O(51) C(54)#2	24(4)
C(50)#2 C(50) O(51) C(52)	-52(8)
C(54)#2 C(50) O(51) C(52)	-13(2)
C(54) C(50) O(51) C(52)	36(4)
O(51)#2 C(50) O(51) C(52)	11(5)
C(54)#2 C(50) O(51) C(50)#2	39(8)
C(54) C(50) O(51) C(50)#2	88(6)
O(51)#2 C(50) O(51) C(50)#2	63(5)
C(50) O(51) C(52) C(54)#2	24(4)
C(50)#2 O(51) C(52) C(54)#2	17(4)
C(50)#2 C(50) C(54) C(52)#2	83(15)
O(51) C(50) C(54) C(52)#2	-36(13)
C(54)#2 C(50) C(54) C(52)#2	-9(11)
O(51)#2 C(50) C(54) C(52)#2	114(13)
C(50)#2 C(50) C(54) O(51)#2	-31(10)
O(51) C(50) C(54) O(51)#2	-150(4)
C(54)#2 C(50) C(54) O(51)#2	-123(4)
O(51) C(50) C(54) C(50)#2	-119(13)
C(54)#2 C(50) C(54) C(50)#2	-92(12)
O(51)#2 C(50) C(54) C(50)#2	31(10)
C(50)#2 C(50) C(54) C(54)#2	92(12)
O(51) C(50) C(54) C(54)#2	-27(2)
O(51)#2 C(50) C(54) C(54)#2	123(4)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1/2, -y-1/2, -z+1

#2 -x, y, -z+1/2

Table 5

Identi	
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Table E24. Crystal and structure refinement data for $2A^{2-}\bullet(K^{+})_2\bullet(C_6H_{12}O_2)_4$.

Identification code	$2A^{2-}\bullet(K^{+})_2\bullet(C_6H_{12}O_2)_4$
Empirical formula	$C_{56}H_{70}K_2O_{12}$
Formula weight	1013.32 g/mole
Temperature	173(2) K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group	$P^{-}1$
Unit cell dimensions	a = 10.619(2) Å b = 10.756(2) Å c = 11.890(2) Å α = 88.29(3)° β = 73.30(3)° γ = 89.20(3)°
Volume	1300.2(5) $Å^3$
Z	1
Density (calculated)	1.294 Mg/m^3
Absorption coefficient	0.244 mm^{-1}
F(000)	540
Crystal Size	0.52 mm x 0.28 mm x 0.20 mm
Color	wine red/purple
Theta range for data collection	1.79° to 28.35°
Index ranges	$-13 \le h \le 13$, $-14 \le k \le 14$, $-15 \le l \le 15$
Reflections collected / Unique	15756 / 6146 [R_{int} = 0.0300]
Refinement method	Full-matrix-least-squares on F^2
Data / Restraints / Parameters	6146 / 0 / 457
Goodness-of-fit on F^2	1.026
Final R indices [$I>2\sigma(I)$]	R_1 = 0.0355, wR_2 = 0.0750
R indices (all data)	R_1 = 0.0616, wR_2 = 0.0818
Extinction Coefficient	0.0054(12)
Largest diff. peak and hole	0.311 and -0.192 e/A^3

Table E25. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for $2A^{2-}\bullet(K^+)_2\bullet(C_6H_{12}O_2)_4$. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U_{eq}
K(1)	8688(1)	4217(1)	1481(1)	25(1)
O(1)	10226(1)	2338(1)	637(1)	24(1)
C(1)	10094(1)	1234(1)	406(1)	18(1)
O(2)	10206(1)	1940(1)	-1550(1)	22(1)
C(2)	10124(1)	982(1)	-871(1)	18(1)
C(3)	10068(1)	-228(1)	-1234(1)	18(1)
C(4)	10158(1)	-423(1)	-2524(1)	19(1)
C(5)	8824(1)	-476(1)	-2758(1)	20(1)
C(6)	7883(2)	-1366(1)	-2208(1)	27(1)
C(7)	6683(2)	-1403(2)	-2446(2)	32(1)
C(8)	6387(2)	-542(2)	-3228(2)	33(1)
C(9)	7303(2)	344(2)	-3774(2)	33(1)
C(10)	8513(2)	371(1)	-3542(1)	27(1)
C(11)	11098(1)	-1473(1)	-3077(1)	20(1)
C(12)	12365(1)	-1504(1)	-2959(1)	24(1)
C(13)	13268(2)	-2397(1)	-3506(1)	28(1)
C(14)	12933(2)	-3283(1)	-4185(1)	28(1)
C(15)	11679(2)	-3272(1)	-4306(1)	29(1)
C(16)	10771(2)	-2375(1)	-3760(1)	24(1)
C(60)	7079(2)	5345(2)	-707(2)	34(1)
O(61)	9070(1)	4224(1)	-914(1)	25(1)
C(61)	7971(1)	4334(1)	-1393(1)	25(1)
C(62)	8470(2)	4738(2)	-2682(2)	39(1)
C(63)	7281(2)	3080(1)	-1299(1)	27(1)
O(64)	6745(1)	2940(1)	805(1)	44(1)
C(64)	6820(2)	2438(1)	-112(1)	28(1)
C(65)	6448(2)	1095(2)	-117(2)	43(1)
C(70)	5429(2)	6109(2)	2868(2)	33(1)
O(71)	7704(1)	6463(1)	2164(1)	28(1)
C(71)	6477(1)	7108(1)	2606(1)	26(1)
C(72)	6287(2)	8041(2)	1681(2)	35(1)
C(73)	6476(2)	7800(1)	3728(1)	29(1)
C(74)	6950(2)	6992(1)	4586(1)	28(1)
O(75)	6236(1)	6240(1)	5243(1)	40(1)
C(75)	8337(2)	7164(2)	4604(2)	37(1)

Table E26. Bond lengths (Å) and angles (°) for $2A^{2-}\bullet(K^{+})_2\bullet(C_6H_{12}O_2)_4$.

Atoms	Length	Angle
K(1)-O(1)	2.6172(13)	
K(1)-O(71)	2.6732(13)	
K(1)-O(61)	2.7578(12)	
K(1)-O(64)	2.8087(14)	
K(1)-O(61)#1	2.8377(14)	
K(1)-C(13)#2	3.302(2)	
K(1)-C(14)#2	3.3149(19)	
K(1)-C(12)#2	3.3932(19)	
K(1)-C(15)#2	3.3951(18)	
K(1)-C(16)#2	3.4845(18)	
K(1)-C(11)#2	3.5020(17)	
K(1)-K(1)#1	4.1398(18)	
K(1)-H(61)	3.013(17)	
K(1)-H(71)	3.06(2)	
O(1)-C(1)	1.2451(16)	
C(1)-C(3)#2	1.4192(19)	
C(1)-C(2)	1.5419(19)	
O(2)-C(2)	1.2780(16)	
C(2)-C(3)	1.3904(18)	
C(3)-C(1)#2	1.4192(19)	
C(3)-C(4)	1.5291(19)	
C(4)-C(5)	1.521(2)	
C(4)-C(11)	1.530(2)	
C(4)-H(4)	0.968(14)	
C(5)-C(10)	1.389(2)	
C(5)-C(6)	1.399(2)	
C(6)-C(7)	1.385(2)	
C(6)-H(6)	0.982(17)	
C(7)-C(8)	1.388(2)	
C(7)-H(7)	0.953(19)	
C(8)-C(9)	1.380(2)	
C(8)-H(8)	0.947(16)	
C(9)-C(10)	1.390(2)	
C(9)-H(9)	0.966(18)	
C(10)-H(10)	0.961(16)	
C(11)-C(12)	1.392(2)	
C(11)-C(16)	1.392(2)	
C(11)-K(1)#2	3.5020(17)	
C(12)-C(13)	1.385(2)	
C(12)-K(1)#2	3.3932(19)	
C(12)-H(12)	0.949(16)	

Table E26. continued...

C(13)-C(14)	1.382(2)
C(13)-K(1)#2	3.302(2)
C(13)-H(13)	0.937(17)
C(14)-C(15)	1.379(2)
C(14)-K(1)#2	3.3148(19)
C(14)-H(14)	0.938(16)
C(15)-C(16)	1.391(2)
C(15)-K(1)#2	3.3950(18)
C(15)-H(15)	0.973(17)
C(16)-K(1)#2	3.4845(18)
C(16)-H(16)	0.933(16)
C(60)-C(61)	1.524(2)
C(60)-H(60A)	0.96(2)
C(60)-H(60B)	1.008(19)
C(60)-H(60C)	0.969(19)
O(61)-C(61)	1.4409(18)
O(61)-K(1)#1	2.8377(14)
O(61)-H(61)	0.842(18)
C(61)-C(62)	1.523(2)
C(61)-C(63)	1.530(2)
C(62)-H(62A)	0.941(19)
C(62)-H(62B)	1.007(18)
C(62)-H(62C)	0.990(18)
C(63)-C(64)	1.505(2)
C(63)-H(63A)	0.961(17)
C(63)-H(63B)	0.960(18)
O(64)-C(64)	1.2125(19)
C(64)-C(65)	1.504(2)
C(65)-H(65A)	0.93(3)
C(65)-H(65B)	0.98(3)
C(65)-H(65C)	0.92(3)
C(70)-C(71)	1.517(2)
C(70)-H(70A)	1.01(2)
C(70)-H(70B)	0.967(19)
C(70)-H(70C)	0.959(19)
O(71)-C(71)	1.4345(18)
O(71)-H(71)	0.87(2)
C(71)-C(72)	1.522(2)
C(71)-C(73)	1.547(2)
C(72)-H(72A)	0.978(18)
C(72)-H(72B)	0.977(19)
C(72)-H(72C)	0.970(19)
C(73)-C(74)	1.510(2)
C(73)-H(73A)	0.996(17)

Table E26. continued...

C(73)-H(73B)	1.004(16)
C(74)-O(75)	1.2154(19)
C(74)-C(75)	1.493(2)
C(75)-H(75A)	1.03(2)
C(75)-H(75B)	0.95(2)
C(75)-H(75C)	0.95(2)

O(1)-K(1)-O(71)	165.07(4)
O(1)-K(1)-O(61)	72.15(4)
O(71)-K(1)-O(61)	105.42(4)
O(1)-K(1)-O(64)	85.73(4)
O(71)-K(1)-O(64)	107.00(4)
O(61)-K(1)-O(64)	66.07(4)
O(1)-K(1)-O(61)#1	88.97(4)
O(71)-K(1)-O(61)#1	76.11(4)
O(61)-K(1)-O(61)#1	84.58(4)
O(64)-K(1)-O(61)#1	150.40(4)
O(1)-K(1)-C(13)#2	91.29(4)
O(71)-K(1)-C(13)#2	101.29(5)
O(61)-K(1)-C(13)#2	127.76(4)
O(64)-K(1)-C(13)#2	63.49(4)
O(61)#1-K(1)-C(13)#2	145.85(4)
O(1)-K(1)-C(14)#2	104.27(5)
O(71)-K(1)-C(14)#2	84.96(5)
O(61)-K(1)-C(14)#2	151.04(4)
O(64)-K(1)-C(14)#2	85.11(5)
O(61)#1-K(1)-C(14)#2	124.36(4)
C(13)#2-K(1)-C(14)#2	24.10(4)
O(1)-K(1)-C(12)#2	67.46(4)
O(71)-K(1)-C(12)#2	124.85(4)
O(61)-K(1)-C(12)#2	116.19(4)
O(64)-K(1)-C(12)#2	63.69(4)
O(61)#1-K(1)-C(12)#2	139.27(4)
C(13)#2-K(1)-C(12)#2	23.83(4)
C(14)#2-K(1)-C(12)#2	41.98(4)
O(1)-K(1)-C(15)#2	92.89(4)
O(71)-K(1)-C(15)#2	91.28(5)
O(61)-K(1)-C(15)#2	162.68(4)
O(64)-K(1)-C(15)#2	105.04(5)
O(61)#1-K(1)-C(15)#2	104.30(5)
C(13)#2-K(1)-C(15)#2	41.58(4)
C(14)#2-K(1)-C(15)#2	23.68(4)
C(12)#2-K(1)-C(15)#2	47.92(4)
O(1)-K(1)-C(16)#2	69.68(4)

Table E26. continued...

O(71)-K(1)-C(16)#2	113.22(4)
O(61)-K(1)-C(16)#2	141.23(4)
O(64)-K(1)-C(16)#2	104.16(4)
O(61)#1-K(1)-C(16)#2	101.18(4)
C(13)#2-K(1)-C(16)#2	47.78(4)
C(14)#2-K(1)-C(16)#2	41.31(4)
C(12)#2-K(1)-C(16)#2	40.48(4)
C(15)#2-K(1)-C(16)#2	23.28(4)
O(1)-K(1)-C(11)#2	55.74(4)
O(71)-K(1)-C(11)#2	131.70(4)
O(61)-K(1)-C(11)#2	121.63(4)
O(64)-K(1)-C(11)#2	84.41(4)
O(61)#1-K(1)-C(11)#2	116.10(4)
C(13)#2-K(1)-C(11)#2	41.51(4)
C(14)#2-K(1)-C(11)#2	48.58(4)
C(12)#2-K(1)-C(11)#2	23.22(3)
C(15)#2-K(1)-C(11)#2	41.12(4)
C(16)#2-K(1)-C(11)#2	22.98(3)
O(1)-K(1)-K(1)#1	77.49(4)
O(71)-K(1)-K(1)#1	90.72(4)
O(61)-K(1)-K(1)#1	43.03(3)
O(64)-K(1)-K(1)#1	109.02(3)
O(61)#1-K(1)-K(1)#1	41.55(3)
C(13)#2-K(1)-K(1)#1	167.24(3)
C(14)#2-K(1)-K(1)#1	165.88(3)
C(12)#2-K(1)-K(1)#1	144.43(3)
C(15)#2-K(1)-K(1)#1	143.64(3)
C(16)#2-K(1)-K(1)#1	130.72(3)
C(11)#2-K(1)-K(1)#1	130.61(4)
O(1)-K(1)-H(61)	56.7(4)
O(71)-K(1)-H(61)	121.4(4)
O(61)-K(1)-H(61)	16.0(3)
O(64)-K(1)-H(61)	64.0(3)
O(61)#1-K(1)-H(61)	88.9(3)
C(13)#2-K(1)-H(61)	119.3(3)
C(14)#2-K(1)-H(61)	143.3(3)
C(12)#2-K(1)-H(61)	103.3(3)
C(15)#2-K(1)-H(61)	147.0(3)
C(16)#2-K(1)-H(61)	125.3(4)
C(11)#2-K(1)-H(61)	106.0(3)
K(1)#1-K(1)-H(61)	49.2(3)
O(1)-K(1)-H(71)	149.4(4)
O(71)-K(1)-H(71)	15.7(4)
O(61)-K(1)-H(71)	100.2(4)

Table E26. continued...

O(64)-K(1)-H(71)	119.0(4)
O(61)#1-K(1)-H(71)	60.6(4)
C(13)#2-K(1)-H(71)	115.2(4)
C(14)#2-K(1)-H(71)	96.1(4)
C(12)#2-K(1)-H(71)	137.8(4)
C(15)#2-K(1)-H(71)	97.1(4)
C(16)#2-K(1)-H(71)	116.1(4)
C(11)#2-K(1)-H(71)	138.0(4)
K(1)#1-K(1)-H(71)	77.3(4)
H(61)-K(1)-H(71)	115.5(5)
C(1)-O(1)-K(1)	137.09(9)
O(1)-C(1)-C(3)#2	124.11(12)
O(1)-C(1)-C(2)	116.24(12)
C(3)#2-C(1)-C(2)	119.64(11)
O(2)-C(2)-C(3)	123.43(12)
O(2)-C(2)-C(1)	115.98(11)
C(3)-C(2)-C(1)	120.59(12)
C(2)-C(3)-C(1)#2	119.66(12)
C(2)-C(3)-C(4)	118.18(12)
C(1)#2-C(3)-C(4)	122.15(11)
C(5)-C(4)-C(3)	113.53(12)
C(5)-C(4)-C(11)	114.02(11)
C(3)-C(4)-C(11)	113.50(11)
C(5)-C(4)-H(4)	104.6(8)
C(3)-C(4)-H(4)	106.6(8)
C(11)-C(4)-H(4)	103.3(8)
C(10)-C(5)-C(6)	117.93(14)
C(10)-C(5)-C(4)	120.05(13)
C(6)-C(5)-C(4)	122.02(13)
C(7)-C(6)-C(5)	120.87(15)
C(7)-C(6)-H(6)	121.2(10)
C(5)-C(6)-H(6)	117.9(10)
C(6)-C(7)-C(8)	120.29(16)
C(6)-C(7)-H(7)	121.4(11)
C(8)-C(7)-H(7)	118.3(11)
C(9)-C(8)-C(7)	119.53(16)
C(9)-C(8)-H(8)	119.7(9)
C(7)-C(8)-H(8)	120.7(9)
C(8)-C(9)-C(10)	120.07(16)
C(8)-C(9)-H(9)	120.8(11)
C(10)-C(9)-H(9)	119.2(11)
C(5)-C(10)-C(9)	121.32(15)
C(5)-C(10)-H(10)	119.1(9)
C(9)-C(10)-H(10)	119.5(9)

Table E26. continued...

C(12)-C(11)-C(16)	117.60(13)
C(12)-C(11)-C(4)	119.52(12)
C(16)-C(11)-C(4)	122.78(13)
C(12)-C(11)-K(1)#2	74.00(9)
C(16)-C(11)-K(1)#2	77.80(8)
C(4)-C(11)-K(1)#2	121.03(8)
C(13)-C(12)-C(11)	121.15(14)
C(13)-C(12)-K(1)#2	74.40(9)
C(11)-C(12)-K(1)#2	82.78(9)
C(13)-C(12)-H(12)	120.9(10)
C(11)-C(12)-H(12)	117.9(10)
K(1)#2-C(12)-H(12)	112.0(10)
C(14)-C(13)-C(12)	120.65(15)
C(14)-C(13)-K(1)#2	78.46(9)
C(12)-C(13)-K(1)#2	81.77(9)
C(14)-C(13)-H(13)	119.1(10)
C(12)-C(13)-H(13)	120.2(10)
K(1)#2-C(13)-H(13)	108.7(10)
C(15)-C(14)-C(13)	119.03(14)
C(15)-C(14)-K(1)#2	81.42(9)
C(13)-C(14)-K(1)#2	77.44(9)
C(15)-C(14)-H(14)	120.2(10)
C(13)-C(14)-H(14)	120.8(10)
K(1)#2-C(14)-H(14)	110.5(9)
C(14)-C(15)-C(16)	120.41(14)
C(14)-C(15)-K(1)#2	74.90(9)
C(16)-C(15)-K(1)#2	81.97(9)
C(14)-C(15)-H(15)	121.6(10)
C(16)-C(15)-H(15)	118.0(10)
K(1)#2-C(15)-H(15)	114.3(9)
C(15)-C(16)-C(11)	121.17(15)
C(15)-C(16)-K(1)#2	74.75(9)
C(11)-C(16)-K(1)#2	79.22(8)
C(15)-C(16)-H(16)	119.9(10)
C(11)-C(16)-H(16)	119.0(10)
K(1)#2-C(16)-H(16)	117.6(10)
C(61)-C(60)-H(60A)	108.5(12)
C(61)-C(60)-H(60B)	112.4(10)
H(60A)-C(60)-H(60B)	108.7(16)
C(61)-C(60)-H(60C)	110.1(11)
H(60A)-C(60)-H(60C)	109.4(16)
H(60B)-C(60)-H(60C)	107.7(15)
C(61)-O(61)-K(1)	120.67(8)
C(61)-O(61)-K(1)#1	126.20(8)

Table E26. continued...

K(1)-O(61)-K(1)#1	95.42(4)
C(61)-O(61)-H(61)	107.1(12)
K(1)-O(61)-H(61)	99.5(12)
K(1)#1-O(61)-H(61)	104.0(12)
O(61)-C(61)-C(62)	108.98(13)
O(61)-C(61)-C(60)	106.31(13)
C(62)-C(61)-C(60)	109.54(14)
O(61)-C(61)-C(63)	110.14(12)
C(62)-C(61)-C(63)	108.93(14)
C(60)-C(61)-C(63)	112.87(14)
C(61)-C(62)-H(62A)	110.8(11)
C(61)-C(62)-H(62B)	110.9(10)
H(62A)-C(62)-H(62B)	109.5(15)
C(61)-C(62)-H(62C)	109.5(10)
H(62A)-C(62)-H(62C)	109.0(15)
H(62B)-C(62)-H(62C)	107.0(14)
C(64)-C(63)-C(61)	118.29(13)
C(64)-C(63)-H(63A)	105.1(10)
C(61)-C(63)-H(63A)	109.7(10)
C(64)-C(63)-H(63B)	108.2(10)
C(61)-C(63)-H(63B)	107.1(10)
H(63A)-C(63)-H(63B)	108.1(14)
C(64)-O(64)-K(1)	129.17(11)
O(64)-C(64)-C(65)	120.57(16)
O(64)-C(64)-C(63)	123.91(14)
C(65)-C(64)-C(63)	115.52(15)
C(64)-C(65)-H(65A)	113.2(14)
C(64)-C(65)-H(65B)	110.2(15)
H(65A)-C(65)-H(65B)	102(2)
C(64)-C(65)-H(65C)	114.6(16)
H(65A)-C(65)-H(65C)	107(2)
H(65B)-C(65)-H(65C)	108(2)
C(71)-C(70)-H(70A)	110.4(10)
C(71)-C(70)-H(70B)	109.5(10)
H(70A)-C(70)-H(70B)	108.7(15)
C(71)-C(70)-H(70C)	112.1(11)
H(70A)-C(70)-H(70C)	109.9(15)
H(70B)-C(70)-H(70C)	106.2(15)
C(71)-O(71)-K(1)	141.50(9)
C(71)-O(71)-H(71)	109.1(13)
K(1)-O(71)-H(71)	108.3(13)
O(71)-C(71)-C(70)	105.55(12)
O(71)-C(71)-C(72)	109.67(13)
C(70)-C(71)-C(72)	111.10(14)

Table E26. continued...

O(71)-C(71)-C(73)	109.61(12)
C(70)-C(71)-C(73)	111.41(13)
C(72)-C(71)-C(73)	109.43(13)
C(71)-C(72)-H(72A)	112.6(10)
C(71)-C(72)-H(72B)	112.4(11)
H(72A)-C(72)-H(72B)	104.4(15)
C(71)-C(72)-H(72C)	111.8(11)
H(72A)-C(72)-H(72C)	106.1(14)
H(72B)-C(72)-H(72C)	109.1(15)
C(74)-C(73)-C(71)	112.64(12)
C(74)-C(73)-H(73A)	109.6(10)
C(71)-C(73)-H(73A)	108.5(10)
C(74)-C(73)-H(73B)	108.5(9)
C(71)-C(73)-H(73B)	109.8(9)
H(73A)-C(73)-H(73B)	107.7(13)
O(75)-C(74)-C(75)	121.17(16)
O(75)-C(74)-C(73)	121.77(15)
C(75)-C(74)-C(73)	117.06(15)
C(74)-C(75)-H(75A)	111.7(11)
C(74)-C(75)-H(75B)	110.7(13)
H(75A)-C(75)-H(75B)	109.1(16)
C(74)-C(75)-H(75C)	110.8(12)
H(75A)-C(75)-H(75C)	112.1(16)
H(75B)-C(75)-H(75C)	102.1(17)

Symmetry transformations used to generate equivalent atoms:

#1 -x+2, -y+1, -z

#2 -x+2, -y, -z

Table E27. Anisotropic displacement parameters ($Å^2$ x 10^3) for $2A^{2-}•(K^+)_2•(C_6H_{12}O_2)_4$. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+...+2hka^*b^*U_{12}]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
K(1)	30(1)	20(1)	26(1)	-3(1)	-8(1)	0(1)
O(1)	34(1)	15(1)	24(1)	-3(1)	-10(1)	0(1)
C(1)	17(1)	18(1)	20(1)	-3(1)	-5(1)	2(1)
O(2)	30(1)	16(1)	20(1)	2(1)	-8(1)	0(1)
C(2)	14(1)	18(1)	19(1)	1(1)	-4(1)	0(1)
C(3)	19(1)	19(1)	17(1)	-2(1)	-6(1)	1(1)
C(4)	23(1)	15(1)	19(1)	2(1)	-6(1)	-2(1)
C(5)	23(1)	19(1)	19(1)	-4(1)	-6(1)	3(1)
C(6)	29(1)	25(1)	27(1)	3(1)	-9(1)	-2(1)
C(7)	28(1)	30(1)	36(1)	-2(1)	-8(1)	-4(1)
C(8)	25(1)	36(1)	42(1)	-8(1)	-15(1)	3(1)
C(9)	35(1)	32(1)	37(1)	4(1)	-18(1)	4(1)
C(10)	29(1)	26(1)	26(1)	3(1)	-10(1)	-3(1)
C(11)	24(1)	19(1)	16(1)	2(1)	-4(1)	0(1)
C(12)	25(1)	23(1)	23(1)	-2(1)	-7(1)	-1(1)
C(13)	21(1)	32(1)	28(1)	3(1)	-4(1)	1(1)
C(14)	34(1)	25(1)	22(1)	0(1)	0(1)	7(1)
C(15)	40(1)	25(1)	22(1)	-6(1)	-8(1)	2(1)
C(16)	28(1)	25(1)	21(1)	-3(1)	-10(1)	1(1)
C(60)	33(1)	26(1)	42(1)	1(1)	-10(1)	6(1)
O(61)	25(1)	20(1)	32(1)	-3(1)	-11(1)	2(1)
C(61)	26(1)	25(1)	25(1)	2(1)	-9(1)	0(1)
C(62)	41(1)	44(1)	30(1)	8(1)	-9(1)	-6(1)
C(63)	28(1)	28(1)	29(1)	-1(1)	-13(1)	-2(1)
O(64)	61(1)	43(1)	31(1)	7(1)	-18(1)	-20(1)
C(64)	23(1)	28(1)	36(1)	3(1)	-14(1)	-3(1)
C(65)	43(1)	27(1)	56(1)	5(1)	-12(1)	-8(1)
C(70)	28(1)	38(1)	33(1)	0(1)	-9(1)	-4(1)
O(71)	23(1)	21(1)	36(1)	-1(1)	-4(1)	1(1)
C(71)	24(1)	26(1)	29(1)	-1(1)	-7(1)	3(1)
C(72)	36(1)	34(1)	34(1)	1(1)	-10(1)	9(1)
C(73)	30(1)	24(1)	33(1)	-3(1)	-8(1)	4(1)
C(74)	29(1)	28(1)	26(1)	-6(1)	-7(1)	0(1)
O(75)	41(1)	43(1)	37(1)	7(1)	-10(1)	-10(1)
C(75)	34(1)	44(1)	35(1)	1(1)	-13(1)	-2(1)

Table E28. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for $2A^{2-}•(K^+)_2•(C_6H_{12}O_2)_4$.

Atom	x	y	z	U_{eq}
H(4)	10571(13)	312(13)	-2956(12)	16(4)
H(6)	8110(16)	-1974(15)	-1663(15)	35(5)
H(7)	6039(18)	-2011(17)	-2081(16)	47(5)
H(8)	5575(16)	-571(14)	-3406(13)	25(4)
H(9)	7109(17)	962(17)	-4308(16)	43(5)
H(10)	9156(16)	974(15)	-3946(14)	29(4)
H(12)	12582(15)	-911(15)	-2471(14)	32(4)
H(13)	14110(17)	-2421(15)	-3401(14)	33(5)
H(14)	13542(15)	-3892(14)	-4552(14)	27(4)
H(15)	11404(16)	-3880(15)	-4777(14)	33(4)
H(16)	9928(16)	-2369(14)	-3852(14)	29(4)
H(60A)	7597(19)	6073(19)	-727(17)	55(6)
H(60B)	6657(18)	5085(17)	138(17)	47(5)
H(60C)	6382(19)	5542(16)	-1060(16)	47(5)
H(61)	9386(17)	3504(17)	-1057(15)	37(5)
H(62A)	8920(18)	5498(18)	-2768(16)	43(5)
H(62B)	7723(17)	4821(15)	-3044(15)	37(5)
H(62C)	9075(17)	4095(16)	-3122(15)	37(5)
H(63A)	6507(17)	3176(15)	-1559(14)	34(4)
H(63B)	7877(17)	2531(16)	-1822(15)	38(5)
H(65A)	5950(20)	790(20)	610(20)	78(8)
H(65B)	7240(30)	560(20)	-280(20)	95(9)
H(65C)	6000(30)	920(20)	-650(20)	93(9)
H(70A)	5446(18)	5663(17)	2127(17)	50(5)
H(70B)	4572(19)	6488(16)	3177(16)	44(5)
H(70C)	5529(18)	5520(17)	3458(17)	45(5)
H(71)	8330(20)	7007(18)	1922(17)	51(6)
H(72A)	6283(16)	7648(16)	951(16)	39(5)
H(72B)	7000(19)	8645(17)	1442(16)	47(5)
H(72C)	5456(19)	8483(16)	1947(16)	43(5)
H(73A)	7050(16)	8544(16)	3494(15)	36(5)
H(73B)	5563(16)	8100(14)	4133(14)	31(4)
H(75A)	8628(18)	6474(18)	5098(17)	53(6)
H(75B)	8440(20)	7950(20)	4911(19)	66(7)
H(75C)	8910(20)	7228(18)	3832(19)	54(6)

Table E29. Torsion angles (°) for $2A^{2-}\bullet(K^{+})_2\bullet(C_6H_{12}O_2)_4$.

Atoms	Angle
O(71) K(1) O(1) C(1)	-169.79(14)
O(61) K(1) O(1) C(1)	-86.89(13)
O(64) K(1) O(1) C(1)	-20.67(13)
O(61)#1 K(1) O(1) C(1)	-171.53(13)
C(13)#2 K(1) O(1) C(1)	42.63(13)
C(14)#2 K(1) O(1) C(1)	63.17(13)
C(12)#2 K(1) O(1) C(1)	42.58(13)
C(15)#2 K(1) O(1) C(1)	84.21(13)
C(16)#2 K(1) O(1) C(1)	86.15(13)
C(11)#2 K(1) O(1) C(1)	65.47(13)
K(1)#1 K(1) O(1) C(1)	-131.23(13)
K(1) O(1) C(1) C(3)#2	-86.06(17)
K(1) O(1) C(1) C(2)	94.37(14)
O(1) C(1) C(2) O(2)	-3.80(18)
C(3)#2 C(1) C(2) O(2)	176.60(12)
O(1) C(1) C(2) C(3)	175.87(12)
C(3)#2 C(1) C(2) C(3)	-3.7(2)
O(2) C(2) C(3) C(1)#2	-176.63(12)
C(1) C(2) C(3) C(1)#2	3.7(2)
O(2) C(2) C(3) C(4)	2.1(2)
C(1) C(2) C(3) C(4)	-177.57(12)
C(2) C(3) C(4) C(5)	-93.22(15)
C(1)#2 C(3) C(4) C(5)	85.46(16)
C(2) C(3) C(4) C(11)	134.45(13)
C(1)#2 C(3) C(4) C(11)	-46.87(17)
C(3) C(4) C(5) C(10)	121.11(14)
C(11) C(4) C(5) C(10)	-106.81(15)
C(3) C(4) C(5) C(6)	-59.26(17)
C(11) C(4) C(5) C(6)	72.82(17)
C(10) C(5) C(6) C(7)	0.4(2)
C(4) C(5) C(6) C(7)	-179.23(13)
C(5) C(6) C(7) C(8)	-0.9(2)
C(6) C(7) C(8) C(9)	0.6(2)
C(7) C(8) C(9) C(10)	0.2(2)
C(6) C(5) C(10) C(9)	0.3(2)
C(4) C(5) C(10) C(9)	179.97(13)
C(8) C(9) C(10) C(5)	-0.6(2)
C(5) C(4) C(11) C(12)	177.90(12)
C(3) C(4) C(11) C(12)	-50.00(17)
C(5) C(4) C(11) C(16)	1.63(19)
C(3) C(4) C(11) C(16)	133.73(14)

Table E29. continued...

C(5) C(4) C(11) K(1)#2	-93.76(12)
C(3) C(4) C(11) K(1)#2	38.33(14)
C(16) C(11) C(12) C(13)	0.3(2)
C(4) C(11) C(12) C(13)	-176.17(13)
K(1)#2 C(11) C(12) C(13)	66.85(13)
C(16) C(11) C(12) K(1)#2	-66.55(12)
C(4) C(11) C(12) K(1)#2	116.99(11)
C(11) C(12) C(13) C(14)	0.0(2)
K(1)#2 C(12) C(13) C(14)	71.30(14)
C(11) C(12) C(13) K(1)#2	-71.27(13)
C(12) C(13) C(14) C(15)	-0.5(2)
K(1)#2 C(13) C(14) C(15)	72.64(14)
C(12) C(13) C(14) K(1)#2	-73.09(14)
C(13) C(14) C(15) C(16)	0.6(2)
K(1)#2 C(14) C(15) C(16)	70.97(14)
C(13) C(14) C(15) K(1)#2	-70.41(13)
C(14) C(15) C(16) C(11)	-0.2(2)
K(1)#2 C(15) C(16) C(11)	66.95(13)
C(14) C(15) C(16) K(1)#2	-67.18(14)
C(12) C(11) C(16) C(15)	-0.2(2)
C(4) C(11) C(16) C(15)	176.14(13)
K(1)#2 C(11) C(16) C(15)	-64.65(14)
C(12) C(11) C(16) K(1)#2	64.45(12)
C(4) C(11) C(16) K(1)#2	-119.21(12)
O(1) K(1) O(61) C(61)	131.09(10)
O(71) K(1) O(61) C(61)	-64.29(10)
O(64) K(1) O(61) C(61)	37.80(9)
O(61)#1 K(1) O(61) C(61)	-138.25(11)
C(13)#2 K(1) O(61) C(61)	53.77(11)
C(14)#2 K(1) O(61) C(61)	43.78(13)
C(12)#2 K(1) O(61) C(61)	78.47(10)
C(15)#2 K(1) O(61) C(61)	99.82(15)
C(16)#2 K(1) O(61) C(61)	120.63(10)
C(11)#2 K(1) O(61) C(61)	104.32(10)
K(1)#1 K(1) O(61) C(61)	-138.25(11)
O(1) K(1) O(61) K(1)#1	-90.66(4)
O(71) K(1) O(61) K(1)#1	73.96(4)
O(64) K(1) O(61) K(1)#1	176.05(5)
O(61)#1 K(1) O(61) K(1)#1	-0.001(1)
C(13)#2 K(1) O(61) K(1)#1	-167.98(4)
C(14)#2 K(1) O(61) K(1)#1	-177.97(6)
C(12)#2 K(1) O(61) K(1)#1	-143.28(4)
C(15)#2 K(1) O(61) K(1)#1	-121.93(12)
C(16)#2 K(1) O(61) K(1)#1	-101.12(6)

Table E29. continued...

C(11)#2 K(1) O(61) K(1)#1	-117.42(4)
K(1) O(61) C(61) C(62)	161.49(10)
K(1)#1 O(61) C(61) C(62)	36.72(16)
K(1) O(61) C(61) C(60)	43.51(14)
K(1)#1 O(61) C(61) C(60)	-81.26(13)
K(1) O(61) C(61) C(63)	-79.08(13)
K(1)#1 O(61) C(61) C(63)	156.15(9)
O(61) C(61) C(63) C(64)	54.76(18)
C(62) C(61) C(63) C(64)	174.22(14)
C(60) C(61) C(63) C(64)	-63.88(19)
O(1) K(1) O(64) C(64)	-46.14(14)
O(71) K(1) O(64) C(64)	125.91(14)
O(61) K(1) O(64) C(64)	26.21(13)
O(61)#1 K(1) O(64) C(64)	34.18(18)
C(13)#2 K(1) O(64) C(64)	-139.72(15)
C(14)#2 K(1) O(64) C(64)	-150.89(14)
C(12)#2 K(1) O(64) C(64)	-113.07(14)
C(15)#2 K(1) O(64) C(64)	-137.99(13)
C(16)#2 K(1) O(64) C(64)	-113.94(14)
C(11)#2 K(1) O(64) C(64)	-102.10(14)
K(1)#1 K(1) O(64) C(64)	29.06(14)
K(1) O(64) C(64) C(65)	125.29(15)
K(1) O(64) C(64) C(63)	-54.6(2)
C(61) C(63) C(64) O(64)	13.8(2)
C(61) C(63) C(64) C(65)	-166.09(15)
O(1) K(1) O(71) C(71)	171.51(13)
O(61) K(1) O(71) C(71)	93.04(14)
O(64) K(1) O(71) C(71)	23.87(15)
O(61)#1 K(1) O(71) C(71)	173.30(14)
C(13)#2 K(1) O(71) C(71)	-41.62(15)
C(14)#2 K(1) O(71) C(71)	-59.44(14)
C(12)#2 K(1) O(71) C(71)	-45.53(15)
C(15)#2 K(1) O(71) C(71)	-82.29(15)
C(16)#2 K(1) O(71) C(71)	-90.31(15)
C(11)#2 K(1) O(71) C(71)	-73.95(15)
K(1)#1 K(1) O(71) C(71)	134.02(14)
K(1) O(71) C(71) C(70)	8.1(2)
K(1) O(71) C(71) C(72)	-111.64(15)
K(1) O(71) C(71) C(73)	128.20(13)
O(71) C(71) C(73) C(74)	-47.19(17)
C(70) C(71) C(73) C(74)	69.25(18)
C(72) C(71) C(73) C(74)	-167.49(14)
C(71) C(73) C(74) O(75)	-80.06(19)
C(71) C(73) C(74) C(75)	100.56(17)

Table E29. continued...

Symmetry transformations used to generate equivalent atoms:

#1 -x+2, -y+1, -z

#2 -x+2, -y, -z

Table E30. Crystal and structure refinement data for $2A^{2-}\bullet(Li^{+})_2\bullet(C_4H_8O_2)_3$.

Identification code	$2A^{2-}\bullet(Li^{+})_2\bullet(C_4H_8O_2)_3$
Empirical formula	$C_{44}H_{46}Li_2O_{10}$
Formula weight	748.69 g/mole
Temperature	173(2) K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group	$P^{-}1$
Unit cell dimensions	a = 12.082(2) Å b = 13.722(3) Å c = 14.830(3) Å α = 109.66(3)° β = 101.04(3)° γ = 109.56(3)°
Volume	2049.2(7) $Å^3$
Z	2
Density (calculated)	1.213 Mg/m^3
Absorption coefficient	0.084 mm^{-1}
F(000)	792
Crystal Size	0.52 mm x 0.10 mm x 0.08 mm
Color	red
Theta range for data collection	1.55° to 28.25°
Index ranges	$-15 \le h \le 15$, $-17 \le k \le 7$, $-17 \le l \le 19$
Reflections collected / Unique	13166 / 9232 [R_{int} = 0.0251]
Refinement method	Full-matrix-least-squares on F^2
Data / Restraints / Parameters	9232 / 0 / 613
Goodness-of-fit on F^2	0.973
Final R indices [$I>2\sigma(I)$]	R_1 = 0.0526, wR_2 = 0.1142
R indices (all data)	R_1 = 0.1160, wR_2 = 0.1356
Extinction Coefficient	0.0057(10)
Largest diff. peak and hole	0.330 and -0.257 e/A^3

Table E31. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for $2A^{2-}\bullet(Li^+)_2\bullet(C_4H_8O_2)_3$. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U_{eq}
Li(1)	1850(3)	7776(3)	4812(2)	25(1)
O(1)	2730(1)	8278(1)	3938(1)	28(1)
C(1)	3810(2)	9075(2)	4384(1)	23(1)
Li(2)	3176(3)	8422(3)	6787(3)	32(1)
O(2)	3451(1)	9136(1)	5915(1)	26(1)
C(2)	4243(2)	9616(2)	5561(1)	22(1)
C(3)	5404(2)	10534(2)	6137(1)	24(1)
C(4)	5819(2)	11094(2)	7299(2)	28(1)
C(5)	5711(2)	12227(2)	7704(2)	32(1)
C(6)	6441(2)	13193(2)	7607(2)	40(1)
C(7)	6286(3)	14192(2)	7961(2)	53(1)
C(8)	5405(3)	14252(3)	8428(2)	62(1)
C(9)	4689(3)	13312(3)	8540(2)	67(1)
C(10)	4845(2)	12313(2)	8182(2)	47(1)
C(11)	7061(2)	11126(2)	7817(2)	27(1)
C(12)	8003(2)	12081(2)	8668(2)	33(1)
C(13)	9098(2)	12053(2)	9130(2)	38(1)
C(14)	9280(2)	11073(2)	8756(2)	42(1)
C(15)	8346(2)	10110(2)	7920(2)	42(1)
C(16)	7255(2)	10139(2)	7463(2)	35(1)
O(17)	1223(1)	6063(1)	4033(1)	32(1)
C(17)	623(2)	5504(2)	4430(2)	25(1)
O(18)	1548(1)	7255(1)	5921(1)	30(1)
C(18)	784(2)	6199(2)	5548(1)	24(1)
C(19)	154(2)	5692(2)	6078(1)	24(1)
C(20)	322(2)	6433(2)	7170(2)	26(1)
C(21)	853(2)	6064(2)	7950(2)	28(1)
C(22)	2140(2)	6518(2)	8407(2)	43(1)
C(23)	2665(3)	6189(3)	9102(2)	56(1)
C(24)	1915(3)	5417(3)	9365(2)	56(1)
C(25)	627(3)	4968(2)	8939(2)	47(1)
C(26)	103(2)	5296(2)	8236(2)	34(1)
C(27)	-809(2)	6648(2)	7297(2)	29(1)
C(28)	-716(2)	7400(2)	8250(2)	38(1)
C(29)	-1709(2)	7644(2)	8397(2)	47(1)
C(30)	-2823(3)	7138(2)	7596(2)	49(1)
C(31)	-2926(3)	6401(2)	6657(2)	52(1)
C(32)	-1933(2)	6154(2)	6508(2)	40(1)
C(50)	-231(2)	7624(2)	2884(2)	53(1)

Table E31. continued...

O(51)	418(1)	8196(1)	4685(1)	40(1)
C(51)	-302(2)	8074(2)	3919(2)	33(1)
O(52)	-1273(1)	8336(1)	3932(1)	38(1)
C(52)	-1453(2)	8739(2)	4911(2)	41(1)
C(53)	-2618(3)	8890(3)	4716(2)	60(1)
C(60)	1439(2)	9732(2)	7426(2)	57(1)
O(61)	3101(1)	9314(1)	8096(1)	37(1)
C(61)	2336(2)	9686(2)	8232(2)	38(1)
O(62)	2220(2)	10102(2)	9142(1)	56(1)
C(62)	3055(3)	10075(3)	9991(2)	64(1)
C(63)	4262(3)	11106(3)	10454(2)	84(1)
C(70)	4419(2)	6938(2)	5107(2)	48(1)
O(71)	4151(2)	7524(2)	6741(1)	51(1)
C(71)	4173(2)	6795(2)	6010(2)	37(1)
O(72)	3956(2)	5760(1)	5927(1)	49(1)
C(72)	3613(4)	5495(3)	6733(2)	87(1)
C(73)	2919(4)	4354(3)	6424(3)	123(2)

Table E32. Bond lengths (Å) and angles (°) for $2A^{2-}\bullet(Li^{+})_2\bullet(C_4H_8O_2)_3$.

Atoms	Length	Angle
Li(1)-O(1)	1.980(3)	
Li(1)-O(51)	1.995(4)	
Li(1)-O(17)	2.019(4)	
Li(1)-O(18)	2.048(3)	
Li(1)-O(2)	2.053(4)	
Li(1)-Li(2)	2.726(5)	
Li(1)-C(17)	2.762(4)	
Li(1)-C(1)	2.774(4)	
O(1)-C(1)	1.251(2)	
C(1)-C(3)#1	1.416(3)	
C(1)-C(2)	1.545(3)	
Li(2)-O(2)	1.881(4)	
Li(2)-O(18)	1.903(4)	
Li(2)-O(61)	1.953(4)	
Li(2)-O(71)	1.965(4)	
O(2)-C(2)	1.277(2)	
C(2)-C(3)	1.385(3)	
C(3)-C(1)#1	1.416(3)	
C(3)-C(4)	1.527(3)	
C(4)-C(5)	1.526(3)	
C(4)-C(11)	1.529(3)	
C(4)-H(4)	0.98(2)	
C(5)-C(10)	1.387(3)	
C(5)-C(6)	1.396(3)	
C(6)-C(7)	1.383(3)	
C(6)-H(6)	0.97(2)	
C(7)-C(8)	1.387(4)	
C(7)-H(7)	1.01(3)	
C(8)-C(9)	1.374(5)	
C(8)-H(8)	0.99(3)	
C(9)-C(10)	1.384(4)	
C(9)-H(9)	0.97(3)	
C(10)-H(10)	0.99(2)	
C(11)-C(12)	1.394(3)	
C(11)-C(16)	1.395(3)	
C(12)-C(13)	1.388(3)	
C(12)-H(12)	1.02(2)	
C(13)-C(14)	1.380(3)	
C(13)-H(13)	0.99(2)	
C(14)-C(15)	1.386(4)	
C(14)-H(14)	0.98(2)	

Table E32. continued...

C(15)-C(16)	1.382(3)
C(15)-H(15)	0.96(3)
C(16)-H(16)	0.98(2)
O(17)-C(17)	1.247(2)
C(17)-C(19)#2	1.416(3)
C(17)-C(18)	1.544(3)
O(18)-C(18)	1.275(2)
C(18)-C(19)	1.387(3)
C(19)-C(17)#2	1.416(3)
C(19)-C(20)	1.526(3)
C(20)-C(27)	1.523(3)
C(20)-C(21)	1.527(3)
C(20)-H(20)	0.99(2)
C(21)-C(26)	1.384(3)
C(21)-C(22)	1.389(3)
C(22)-C(23)	1.387(4)
C(22)-H(22)	0.97(3)
C(23)-C(24)	1.365(4)
C(23)-H(23)	0.97(3)
C(24)-C(25)	1.385(4)
C(24)-H(24)	0.99(3)
C(25)-C(26)	1.395(3)
C(25)-H(25)	0.89(3)
C(26)-H(26)	0.93(2)
C(27)-C(32)	1.383(3)
C(27)-C(28)	1.399(3)
C(28)-C(29)	1.385(3)
C(28)-H(28)	0.98(2)
C(29)-C(30)	1.382(4)
C(29)-H(29)	0.96(3)
C(30)-C(31)	1.371(4)
C(30)-H(30)	0.96(2)
C(31)-C(32)	1.388(3)
C(31)-H(31)	0.99(3)
C(32)-H(32)	0.93(2)
C(50)-C(51)	1.481(3)
C(50)-H(50A)	0.9600
C(50)-H(50B)	0.9600
C(50)-H(50C)	0.9600
O(51)-C(51)	1.212(3)
C(51)-O(52)	1.339(2)
O(52)-C(52)	1.457(3)
C(52)-C(53)	1.479(4)
C(52)-H(52A)	1.00(2)

Table E32. continued...

C(52)-H(52B)	1.00(3)
C(53)-H(53A)	0.9600
C(53)-H(53B)	0.9600
C(53)-H(53C)	0.9600
C(60)-C(61)	1.486(3)
C(60)-H(60A)	0.9600
C(60)-H(60B)	0.9600
C(60)-H(60C)	0.9600
O(61)-C(61)	1.214(3)
C(61)-O(62)	1.331(3)
O(62)-C(62)	1.474(4)
C(62)-C(63)	1.477(4)
C(62)-H(62A)	1.11(3)
C(62)-H(62B)	0.94(3)
C(63)-H(63A)	0.9600
C(63)-H(63B)	0.9600
C(63)-H(63C)	0.9600
C(70)-C(71)	1.486(3)
C(70)-H(70A)	0.9600
C(70)-H(70B)	0.9600
C(70)-H(70C)	0.9600
O(71)-C(71)	1.214(3)
C(71)-O(72)	1.313(3)
O(72)-C(72)	1.453(3)
C(72)-C(73)	1.363(4)
C(72)-H(72A)	0.9700
C(72)-H(72B)	0.9700
C(73)-H(73A)	0.9600
C(73)-H(73B)	0.9600
C(73)-H(73C)	0.9600

O(1)-Li(1)-O(51)	106.13(17)
O(1)-Li(1)-O(17)	99.00(15)
O(51)-Li(1)-O(17)	109.97(16)
O(1)-Li(1)-O(18)	159.23(19)
O(51)-Li(1)-O(18)	93.73(14)
O(17)-Li(1)-O(18)	79.32(13)
O(1)-Li(1)-O(2)	80.21(13)
O(51)-Li(1)-O(2)	110.83(16)
O(17)-Li(1)-O(2)	137.64(18)
O(18)-Li(1)-O(2)	87.18(13)
O(1)-Li(1)-Li(2)	119.52(16)
O(51)-Li(1)-Li(2)	112.60(16)
O(17)-Li(1)-Li(2)	108.63(17)

Table E32. continued...

O(18)-Li(1)-Li(2)	44.20(10)
O(2)-Li(1)-Li(2)	43.59(10)
O(1)-Li(1)-C(17)	123.47(15)
O(51)-Li(1)-C(17)	100.18(13)
O(17)-Li(1)-C(17)	24.47(7)
O(18)-Li(1)-C(17)	56.59(10)
O(2)-Li(1)-C(17)	133.76(16)
Li(2)-Li(1)-C(17)	93.38(14)
O(1)-Li(1)-C(1)	23.79(7)
O(51)-Li(1)-C(1)	114.12(15)
O(17)-Li(1)-C(1)	114.38(14)
O(18)-Li(1)-C(1)	139.61(15)
O(2)-Li(1)-C(1)	56.50(9)
Li(2)-Li(1)-C(1)	96.43(13)
C(17)-Li(1)-C(1)	136.95(14)
C(1)-O(1)-Li(1)	116.51(15)
O(1)-C(1)-C(3)#1	123.21(17)
O(1)-C(1)-C(2)	115.22(17)
C(3)#1-C(1)-C(2)	121.58(17)
O(1)-C(1)-Li(1)	39.70(11)
C(3)#1-C(1)-Li(1)	162.47(15)
C(2)-C(1)-Li(1)	75.66(13)
O(2)-Li(2)-O(18)	96.71(17)
O(2)-Li(2)-O(61)	117.6(2)
O(18)-Li(2)-O(61)	109.39(18)
O(2)-Li(2)-O(71)	108.84(18)
O(18)-Li(2)-O(71)	101.41(18)
O(61)-Li(2)-O(71)	119.22(19)
O(2)-Li(2)-Li(1)	48.80(11)
O(18)-Li(2)-Li(1)	48.64(11)
O(61)-Li(2)-Li(1)	133.54(19)
O(71)-Li(2)-Li(1)	106.23(16)
C(2)-O(2)-Li(2)	145.77(17)
C(2)-O(2)-Li(1)	113.81(15)
Li(2)-O(2)-Li(1)	87.60(15)
O(2)-C(2)-C(3)	125.57(17)
O(2)-C(2)-C(1)	114.03(16)
C(3)-C(2)-C(1)	120.40(17)
C(2)-C(3)-C(1)#1	117.98(17)
C(2)-C(3)-C(4)	120.51(17)
C(1)#1-C(3)-C(4)	121.51(17)
C(5)-C(4)-C(3)	111.78(18)
C(5)-C(4)-C(11)	115.65(17)
C(3)-C(4)-C(11)	113.25(17)

Table E32. continued...

C(5)-C(4)-H(4)	106.3(11)
C(3)-C(4)-H(4)	105.6(12)
C(11)-C(4)-H(4)	103.1(12)
C(10)-C(5)-C(6)	117.6(2)
C(10)-C(5)-C(4)	119.4(2)
C(6)-C(5)-C(4)	123.0(2)
C(7)-C(6)-C(5)	120.9(3)
C(7)-C(6)-H(6)	121.1(14)
C(5)-C(6)-H(6)	117.8(14)
C(6)-C(7)-C(8)	120.3(3)
C(6)-C(7)-H(7)	121.1(16)
C(8)-C(7)-H(7)	118.6(15)
C(9)-C(8)-C(7)	119.4(3)
C(9)-C(8)-H(8)	119.9(16)
C(7)-C(8)-H(8)	120.6(16)
C(8)-C(9)-C(10)	120.1(3)
C(8)-C(9)-H(9)	119.8(17)
C(10)-C(9)-H(9)	120.1(17)
C(9)-C(10)-C(5)	121.7(3)
C(9)-C(10)-H(10)	122.3(14)
C(5)-C(10)-H(10)	116.0(13)
C(12)-C(11)-C(16)	117.3(2)
C(12)-C(11)-C(4)	123.35(19)
C(16)-C(11)-C(4)	119.25(19)
C(13)-C(12)-C(11)	121.1(2)
C(13)-C(12)-H(12)	120.0(14)
C(11)-C(12)-H(12)	118.8(13)
C(14)-C(13)-C(12)	120.6(2)
C(14)-C(13)-H(13)	122.5(14)
C(12)-C(13)-H(13)	116.8(14)
C(13)-C(14)-C(15)	119.2(2)
C(13)-C(14)-H(14)	123.1(13)
C(15)-C(14)-H(14)	117.7(13)
C(16)-C(15)-C(14)	120.1(2)
C(16)-C(15)-H(15)	119.8(15)
C(14)-C(15)-H(15)	120.1(15)
C(15)-C(16)-C(11)	121.7(2)
C(15)-C(16)-H(16)	120.3(14)
C(11)-C(16)-H(16)	118.0(14)
C(17)-O(17)-Li(1)	113.40(15)
O(17)-C(17)-C(19)#2	124.08(18)
O(17)-C(17)-C(18)	115.56(17)
C(19)#2-C(17)-C(18)	120.35(17)
O(17)-C(17)-Li(1)	42.12(11)

Table E32. continued...

C(19)#2-C(17)-Li(1)	162.01(15)
C(18)-C(17)-Li(1)	74.87(13)
C(18)-O(18)-Li(2)	147.11(18)
C(18)-O(18)-Li(1)	112.10(15)
Li(2)-O(18)-Li(1)	87.16(15)
O(18)-C(18)-C(19)	124.50(17)
O(18)-C(18)-C(17)	113.72(17)
C(19)-C(18)-C(17)	121.78(17)
C(18)-C(19)-C(17)#2	117.84(17)
C(18)-C(19)-C(20)	119.25(17)
C(17)#2-C(19)-C(20)	122.91(17)
C(27)-C(20)-C(19)	114.47(17)
C(27)-C(20)-C(21)	112.91(16)
C(19)-C(20)-C(21)	113.85(17)
C(27)-C(20)-H(20)	105.8(11)
C(19)-C(20)-H(20)	102.6(12)
C(21)-C(20)-H(20)	105.9(12)
C(26)-C(21)-C(22)	117.8(2)
C(26)-C(21)-C(20)	122.81(19)
C(22)-C(21)-C(20)	119.3(2)
C(23)-C(22)-C(21)	121.4(3)
C(23)-C(22)-H(22)	121.5(16)
C(21)-C(22)-H(22)	117.0(16)
C(24)-C(23)-C(22)	120.2(3)
C(24)-C(23)-H(23)	123.7(16)
C(22)-C(23)-H(23)	116.0(16)
C(23)-C(24)-C(25)	119.8(3)
C(23)-C(24)-H(24)	122.5(15)
C(25)-C(24)-H(24)	117.6(15)
C(24)-C(25)-C(26)	119.9(3)
C(24)-C(25)-H(25)	124.4(17)
C(26)-C(25)-H(25)	115.7(17)
C(21)-C(26)-C(25)	120.9(3)
C(21)-C(26)-H(26)	120.6(15)
C(25)-C(26)-H(26)	118.5(15)
C(32)-C(27)-C(28)	117.4(2)
C(32)-C(27)-C(20)	123.48(19)
C(28)-C(27)-C(20)	119.15(19)
C(29)-C(28)-C(27)	121.3(2)
C(29)-C(28)-H(28)	123.4(13)
C(27)-C(28)-H(28)	115.3(13)
C(30)-C(29)-C(28)	120.2(2)
C(30)-C(29)-H(29)	118.5(15)
C(28)-C(29)-H(29)	121.1(15)

Table E32. continued...

C(31)-C(30)-C(29)	119.1(2)
C(31)-C(30)-H(30)	121.5(15)
C(29)-C(30)-H(30)	119.4(15)
C(30)-C(31)-C(32)	120.8(3)
C(30)-C(31)-H(31)	118.9(15)
C(32)-C(31)-H(31)	119.9(15)
C(27)-C(32)-C(31)	121.2(2)
C(27)-C(32)-H(32)	119.1(14)
C(31)-C(32)-H(32)	119.6(14)
C(51)-C(50)-H(50A)	109.5
C(51)-C(50)-H(50B)	109.5
H(50A)-C(50)-H(50B)	109.5
C(51)-C(50)-H(50C)	109.5
H(50A)-C(50)-H(50C)	109.5
H(50B)-C(50)-H(50C)	109.5
C(51)-O(51)-Li(1)	128.17(17)
O(51)-C(51)-O(52)	122.1(2)
O(51)-C(51)-C(50)	125.3(2)
O(52)-C(51)-C(50)	112.5(2)
C(51)-O(52)-C(52)	116.56(18)
O(52)-C(52)-C(53)	106.4(2)
O(52)-C(52)-H(52A)	107.0(13)
C(53)-C(52)-H(52A)	114.4(13)
O(52)-C(52)-H(52B)	107.6(14)
C(53)-C(52)-H(52B)	111.2(13)
H(52A)-C(52)-H(52B)	109.9(19)
C(52)-C(53)-H(53A)	109.5
C(52)-C(53)-H(53B)	109.5
H(53A)-C(53)-H(53B)	109.5
C(52)-C(53)-H(53C)	109.5
H(53A)-C(53)-H(53C)	109.5
H(53B)-C(53)-H(53C)	109.5
C(61)-C(60)-H(60A)	109.5
C(61)-C(60)-H(60B)	109.5
H(60A)-C(60)-H(60B)	109.5
C(61)-C(60)-H(60C)	109.5
H(60A)-C(60)-H(60C)	109.5
H(60B)-C(60)-H(60C)	109.5
C(61)-O(61)-Li(2)	126.92(19)
O(61)-C(61)-O(62)	122.4(2)
O(61)-C(61)-C(60)	124.8(2)
O(62)-C(61)-C(60)	112.8(2)
C(61)-O(62)-C(62)	117.0(2)
O(62)-C(62)-C(63)	110.1(3)

Table E32. continued...

O(62)-C(62)-H(62A)	108.8(17)
C(63)-C(62)-H(62A)	112.1(16)
O(62)-C(62)-H(62B)	106.9(18)
C(63)-C(62)-H(62B)	109.5(18)
H(62A)-C(62)-H(62B)	109(3)
C(62)-C(63)-H(63A)	109.5
C(62)-C(63)-H(63B)	109.5
H(63A)-C(63)-H(63B)	109.5
C(62)-C(63)-H(63C)	109.5
H(63A)-C(63)-H(63C)	109.5
H(63B)-C(63)-H(63C)	109.5
C(71)-C(70)-H(70A)	109.5
C(71)-C(70)-H(70B)	109.5
H(70A)-C(70)-H(70B)	109.5
C(71)-C(70)-H(70C)	109.5
H(70A)-C(70)-H(70C)	109.5
H(70B)-C(70)-H(70C)	109.5
C(71)-O(71)-Li(2)	129.44(19)
O(71)-C(71)-O(72)	122.6(2)
O(71)-C(71)-C(70)	125.6(2)
O(72)-C(71)-C(70)	111.8(2)
C(71)-O(72)-C(72)	116.6(2)
C(73)-C(72)-O(72)	113.1(3)
C(73)-C(72)-H(72A)	109.0
O(72)-C(72)-H(72A)	108.9
C(73)-C(72)-H(72B)	109.0
O(72)-C(72)-H(72B)	109.0
H(72A)-C(72)-H(72B)	107.8
C(72)-C(73)-H(73A)	109.5
C(72)-C(73)-H(73B)	109.5
H(73A)-C(73)-H(73B)	109.5
C(72)-C(73)-H(73C)	109.5
H(73A)-C(73)-H(73C)	109.5
H(73B)-C(73)-H(73C)	109.5

Symmetry transformations used to generate equivalent atoms:

#1 -x+1, -y+2, -z+1

#2 -x, -y+1, -z+1

Table E33. Anisotropic displacement parameters ($Å^2 \times 10^3$) for $2A^{2-} \cdot (Li^+)_2 \cdot (C_4H_8O_2)_3$. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+...+2hka^*b^*U_{12}]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Li(1)	24(2)	24(2)	21(2)	11(1)	8(1)	5(1)
O(1)	25(1)	28(1)	23(1)	10(1)	7(1)	3(1)
C(1)	24(1)	23(1)	22(1)	11(1)	8(1)	9(1)
Li(2)	32(2)	32(2)	27(2)	14(2)	12(2)	8(2)
O(2)	25(1)	29(1)	23(1)	13(1)	10(1)	6(1)
C(2)	24(1)	24(1)	22(1)	14(1)	10(1)	11(1)
C(3)	25(1)	26(1)	19(1)	10(1)	8(1)	9(1)
C(4)	27(1)	28(1)	20(1)	10(1)	9(1)	3(1)
C(5)	34(1)	35(1)	18(1)	7(1)	4(1)	13(1)
C(6)	52(2)	34(1)	30(1)	11(1)	14(1)	18(1)
C(7)	75(2)	42(2)	39(2)	15(1)	10(1)	29(2)
C(8)	80(2)	60(2)	50(2)	14(2)	12(2)	50(2)
C(9)	61(2)	79(2)	65(2)	18(2)	27(2)	45(2)
C(10)	41(1)	52(2)	43(2)	12(1)	17(1)	21(1)
C(11)	31(1)	28(1)	22(1)	13(1)	10(1)	8(1)
C(12)	38(1)	31(1)	24(1)	11(1)	8(1)	10(1)
C(13)	38(1)	41(2)	25(1)	14(1)	4(1)	10(1)
C(14)	40(1)	52(2)	37(1)	26(1)	9(1)	21(1)
C(15)	53(2)	39(2)	42(1)	22(1)	17(1)	25(1)
C(16)	42(1)	29(1)	27(1)	10(1)	8(1)	11(1)
O(17)	36(1)	28(1)	27(1)	14(1)	15(1)	5(1)
C(17)	23(1)	27(1)	24(1)	14(1)	8(1)	7(1)
O(18)	34(1)	22(1)	24(1)	9(1)	10(1)	0(1)
C(18)	21(1)	25(1)	22(1)	10(1)	5(1)	7(1)
C(19)	25(1)	24(1)	19(1)	9(1)	8(1)	6(1)
C(20)	29(1)	23(1)	21(1)	9(1)	7(1)	5(1)
C(21)	35(1)	28(1)	19(1)	7(1)	11(1)	14(1)
C(22)	39(1)	55(2)	31(1)	16(1)	10(1)	20(1)
C(23)	51(2)	75(2)	40(2)	20(2)	8(1)	35(2)
C(24)	86(2)	62(2)	26(1)	15(1)	9(1)	50(2)
C(25)	79(2)	38(2)	27(1)	15(1)	19(1)	27(2)
C(26)	45(2)	31(1)	25(1)	13(1)	14(1)	13(1)
C(27)	36(1)	23(1)	27(1)	13(1)	11(1)	10(1)
C(28)	44(1)	40(1)	28(1)	13(1)	11(1)	19(1)
C(29)	60(2)	51(2)	38(2)	14(1)	20(1)	35(1)
C(30)	50(2)	55(2)	53(2)	22(1)	21(1)	34(1)
C(31)	44(2)	53(2)	49(2)	10(1)	7(1)	27(1)
C(32)	44(1)	39(1)	30(1)	8(1)	8(1)	20(1)
C(50)	54(2)	72(2)	36(1)	18(1)	18(1)	34(2)

Table E33. continued...

O(51)	36(1)	51(1)	34(1)	18(1)	12(1)	21(1)
C(51)	31(1)	31(1)	36(1)	13(1)	13(1)	12(1)
O(52)	37(1)	46(1)	36(1)	18(1)	15(1)	23(1)
C(52)	44(2)	41(2)	42(1)	16(1)	21(1)	19(1)
C(53)	61(2)	82(2)	70(2)	42(2)	41(2)	50(2)
C(60)	56(2)	68(2)	45(2)	18(1)	10(1)	33(2)
O(61)	40(1)	37(1)	28(1)	10(1)	14(1)	13(1)
C(61)	38(1)	35(1)	29(1)	8(1)	12(1)	9(1)
O(62)	55(1)	72(1)	34(1)	13(1)	21(1)	29(1)
C(62)	73(2)	86(2)	34(2)	24(2)	28(2)	31(2)
C(63)	74(2)	95(3)	48(2)	11(2)	6(2)	26(2)
C(70)	57(2)	51(2)	47(2)	26(1)	22(1)	28(1)
O(71)	62(1)	56(1)	38(1)	13(1)	12(1)	37(1)
C(71)	26(1)	39(1)	40(1)	15(1)	5(1)	14(1)
O(72)	64(1)	36(1)	41(1)	17(1)	20(1)	13(1)
C(72)	125(3)	59(2)	53(2)	28(2)	39(2)	4(2)
C(73)	160(4)	64(2)	75(3)	36(2)	12(3)	-21(2)

Table E34. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for $2A^{2-}•(Li^+)_2•(C_4H_8O_2)_3$.

Atom	x	y	z	U_{eq}
H(4)	5217(18)	10572(17)	7472(15)	26(5)
H(6)	7000(20)	13118(19)	7229(18)	42(7)
H(7)	6790(20)	14880(20)	7870(20)	68(9)
H(8)	5310(20)	14980(20)	8700(20)	61(8)
H(9)	4050(30)	13340(20)	8850(20)	72(9)
H(10)	4370(20)	11630(20)	8274(18)	46(7)
H(12)	7860(20)	12800(20)	8964(18)	54(7)
H(13)	9750(20)	12780(20)	9705(19)	47(7)
H(14)	10030(20)	11014(19)	9070(18)	45(7)
H(15)	8460(20)	9420(20)	7654(19)	56(8)
H(16)	6580(20)	9450(20)	6894(18)	45(7)
H(20)	985(19)	7189(18)	7318(15)	30(6)
H(22)	2650(20)	7110(20)	8250(20)	61(8)
H(23)	3570(20)	6580(20)	9410(20)	59(8)
H(24)	2250(20)	5140(20)	9830(20)	59(8)
H(25)	80(20)	4450(20)	9068(19)	52(8)
H(26)	-760(20)	4992(19)	7965(18)	43(7)
H(28)	100(20)	7749(18)	8788(17)	38(6)
H(29)	-1620(20)	8210(20)	9040(20)	56(7)
H(30)	-3520(20)	7290(20)	7715(18)	52(7)
H(31)	-3750(20)	5970(20)	6110(20)	65(8)
H(32)	-2002(19)	5689(19)	5860(18)	39(6)
H(50A)	385	7328	2891	80
H(50B)	-1	8232	2674	80
H(50C)	-1031	7022	2415	80
H(52A)	-690(20)	9460(20)	5387(17)	41(6)
H(52B)	-1540(20)	8130(20)	5162(18)	50(7)
H(53A)	-2753	9196	5348	75(9)
H(53B)	-3311	8162	4265	87(11)
H(53C)	-2547	9411	4407	116(14)
H(60A)	1565	9430	6789	86
H(60B)	1572	10514	7608	86
H(60C)	599	9283	7361	86
H(62A)	3170(30)	9270(30)	9700(20)	96(11)
H(62B)	2660(30)	10070(20)	10480(20)	74(9)
H(63A)	4796	11084	11010	126
H(63B)	4121	11780	10701	126
H(63C)	4655	11123	9950	126
H(70A)	4569	7702	5193	72
H(70B)	3707	6393	4505	72

Table E34. continued...

H(70C)	5140	6811	5040	72
H(72A)	4369	5762	7294	104
H(72B)	3141	5909	6982	104
H(73A)	2700	4233	6979	185
H(73B)	3397	3942	6210	185
H(73C)	2172	4082	5865	185

Table E35. Torsion angles (°) for $2A^{2-} \bullet (Li^{+})_2 \bullet (C_4H_8O_2)_3$.

Atoms	Angle
O(51) Li(1) O(1) C(1)	113.48(18)
O(17) Li(1) O(1) C(1)	-132.57(16)
O(18) Li(1) O(1) C(1)	-49.0(6)
O(2) Li(1) O(1) C(1)	4.47(19)
Li(2) Li(1) O(1) C(1)	-15.1(3)
C(17) Li(1) O(1) C(1)	-132.20(18)
Li(1) O(1) C(1) C(3)#1	174.69(18)
Li(1) O(1) C(1) C(2)	-5.3(2)
O(51) Li(1) C(1) O(1)	-74.9(2)
O(17) Li(1) C(1) O(1)	52.99(18)
O(18) Li(1) C(1) O(1)	155.6(3)
O(2) Li(1) C(1) O(1)	-174.7(2)
Li(2) Li(1) C(1) O(1)	166.8(2)
C(17) Li(1) C(1) O(1)	64.8(2)
O(1) Li(1) C(1) C(3)#1	-14.9(5)
O(51) Li(1) C(1) C(3)#1	-89.8(5)
O(17) Li(1) C(1) C(3)#1	38.1(6)
O(18) Li(1) C(1) C(3)#1	140.7(5)
O(2) Li(1) C(1) C(3)#1	170.4(5)
Li(2) Li(1) C(1) C(3)#1	151.9(5)
C(17) Li(1) C(1) C(3)#1	49.9(6)
O(1) Li(1) C(1) C(2)	175.1(2)
O(51) Li(1) C(1) C(2)	100.21(17)
O(17) Li(1) C(1) C(2)	-131.93(18)
O(18) Li(1) C(1) C(2)	-29.3(2)
O(2) Li(1) C(1) C(2)	0.37(11)
Li(2) Li(1) C(1) C(2)	-18.09(14)
C(17) Li(1) C(1) C(2)	-120.1(2)
O(1) Li(1) Li(2) O(2)	28.59(17)
O(51) Li(1) Li(2) O(2)	-96.97(18)
O(17) Li(1) Li(2) O(2)	140.96(19)
O(18) Li(1) Li(2) O(2)	-167.9(2)
C(17) Li(1) Li(2) O(2)	160.53(16)
C(1) Li(1) Li(2) O(2)	22.52(12)
O(1) Li(1) Li(2) O(18)	-163.5(2)
O(51) Li(1) Li(2) O(18)	70.91(17)
O(17) Li(1) Li(2) O(18)	-51.15(15)
O(2) Li(1) Li(2) O(18)	167.9(2)
C(17) Li(1) Li(2) O(18)	-31.59(11)
C(1) Li(1) Li(2) O(18)	-169.59(17)
O(1) Li(1) Li(2) O(61)	119.6(2)

Table E35. continued...

O(51) Li(1) Li(2) O(61)	-6.0(3)
O(17) Li(1) Li(2) O(61)	-128.1(2)
O(18) Li(1) Li(2) O(61)	-76.9(2)
O(2) Li(1) Li(2) O(61)	91.0(3)
C(17) Li(1) Li(2) O(61)	-108.5(2)
C(1) Li(1) Li(2) O(61)	113.5(2)
O(1) Li(1) Li(2) O(71)	-72.5(2)
O(51) Li(1) Li(2) O(71)	161.95(16)
O(17) Li(1) Li(2) O(71)	39.9(2)
O(18) Li(1) Li(2) O(71)	91.04(18)
O(2) Li(1) Li(2) O(71)	-101.07(18)
C(17) Li(1) Li(2) O(71)	59.45(16)
C(1) Li(1) Li(2) O(71)	-78.55(17)
O(18) Li(2) O(2) C(2)	-140.2(2)
O(61) Li(2) O(2) C(2)	103.8(3)
O(71) Li(2) O(2) C(2)	-35.7(4)
Li(1) Li(2) O(2) C(2)	-131.0(3)
O(18) Li(2) O(2) Li(1)	-9.12(16)
O(61) Li(2) O(2) Li(1)	-125.1(2)
O(71) Li(2) O(2) Li(1)	95.39(18)
O(1) Li(1) O(2) C(2)	-2.63(18)
O(51) Li(1) O(2) C(2)	-106.28(18)
O(17) Li(1) O(2) C(2)	90.0(3)
O(18) Li(1) O(2) C(2)	160.80(14)
Li(2) Li(1) O(2) C(2)	152.37(19)
C(17) Li(1) O(2) C(2)	124.9(2)
C(1) Li(1) O(2) C(2)	-0.47(13)
O(1) Li(1) O(2) Li(2)	-155.00(15)
O(51) Li(1) O(2) Li(2)	101.34(18)
O(17) Li(1) O(2) Li(2)	-62.3(3)
O(18) Li(1) O(2) Li(2)	8.42(15)
C(17) Li(1) O(2) Li(2)	-27.4(2)
C(1) Li(1) O(2) Li(2)	-152.84(14)
Li(2) O(2) C(2) C(3)	-54.1(4)
Li(1) O(2) C(2) C(3)	-178.69(19)
Li(2) O(2) C(2) C(1)	125.3(3)
Li(1) O(2) C(2) C(1)	0.8(2)
O(1) C(1) C(2) O(2)	2.9(2)
C(3)#1 C(1) C(2) O(2)	-177.03(18)
Li(1) C(1) C(2) O(2)	-0.54(15)
O(1) C(1) C(2) C(3)	-177.58(18)
C(3)#1 C(1) C(2) C(3)	2.5(3)
Li(1) C(1) C(2) C(3)	178.95(19)
O(2) C(2) C(3) C(1)#1	177.05(18)

Table E35. continued...

C(1) C(2) C(3) C(1)#1	-2.4(3)
O(2) C(2) C(3) C(4)	-2.4(3)
C(1) C(2) C(3) C(4)	178.13(17)
C(2) C(3) C(4) C(5)	-102.8(2)
C(1)#1 C(3) C(4) C(5)	77.8(2)
C(2) C(3) C(4) C(11)	124.5(2)
C(1)#1 C(3) C(4) C(11)	-55.0(3)
C(3) C(4) C(5) C(10)	113.3(2)
C(11) C(4) C(5) C(10)	-115.1(2)
C(3) C(4) C(5) C(6)	-65.9(3)
C(11) C(4) C(5) C(6)	65.6(3)
C(10) C(5) C(6) C(7)	-1.1(3)
C(4) C(5) C(6) C(7)	178.2(2)
C(5) C(6) C(7) C(8)	0.6(4)
C(6) C(7) C(8) C(9)	0.2(4)
C(7) C(8) C(9) C(10)	-0.4(4)
C(8) C(9) C(10) C(5)	-0.2(4)
C(6) C(5) C(10) C(9)	0.9(4)
C(4) C(5) C(10) C(9)	-178.4(2)
C(5) C(4) C(11) C(12)	6.1(3)
C(3) C(4) C(11) C(12)	137.0(2)
C(5) C(4) C(11) C(16)	-177.21(19)
C(3) C(4) C(11) C(16)	-46.3(3)
C(16) C(11) C(12) C(13)	1.1(3)
C(4) C(11) C(12) C(13)	177.9(2)
C(11) C(12) C(13) C(14)	-0.1(3)
C(12) C(13) C(14) C(15)	-0.8(4)
C(13) C(14) C(15) C(16)	0.7(4)
C(14) C(15) C(16) C(11)	0.4(4)
C(12) C(11) C(16) C(15)	-1.3(3)
C(4) C(11) C(16) C(15)	-178.2(2)
O(1) Li(1) O(17) C(17)	179.24(15)
O(51) Li(1) O(17) C(17)	-69.9(2)
O(18) Li(1) O(17) C(17)	20.25(17)
O(2) Li(1) O(17) C(17)	93.9(3)
Li(2) Li(1) O(17) C(17)	53.8(2)
C(1) Li(1) O(17) C(17)	160.20(15)
Li(1) O(17) C(17) C(19)#2	164.56(18)
Li(1) O(17) C(17) C(18)	-16.2(2)
O(1) Li(1) C(17) O(17)	-0.90(18)
O(51) Li(1) C(17) O(17)	116.3(2)
O(18) Li(1) C(17) O(17)	-156.0(2)
O(2) Li(1) C(17) O(17)	-111.5(3)
Li(2) Li(1) C(17) O(17)	-130.0(2)

Table E35. continued...

C(1) Li(1) C(17) O(17)	-26.9(2)
O(1) Li(1) C(17) C(19)#2	-46.5(6)
O(51) Li(1) C(17) C(19)#2	70.7(5)
O(17) Li(1) C(17) C(19)#2	-45.6(5)
O(18) Li(1) C(17) C(19)#2	158.5(5)
O(2) Li(1) C(17) C(19)#2	-157.0(4)
Li(2) Li(1) C(17) C(19)#2	-175.6(5)
C(1) Li(1) C(17) C(19)#2	-72.4(6)
O(1) Li(1) C(17) C(18)	164.0(2)
O(51) Li(1) C(17) C(18)	-78.80(15)
O(17) Li(1) C(17) C(18)	164.9(2)
O(18) Li(1) C(17) C(18)	8.95(10)
O(2) Li(1) C(17) C(18)	53.5(2)
Li(2) Li(1) C(17) C(18)	34.89(14)
C(1) Li(1) C(17) C(18)	138.0(2)
O(2) Li(2) O(18) C(18)	137.3(3)
O(61) Li(2) O(18) C(18)	-100.4(3)
O(71) Li(2) O(18) C(18)	26.4(4)
Li(1) Li(2) O(18) C(18)	128.1(3)
O(2) Li(2) O(18) Li(1)	9.15(16)
O(61) Li(2) O(18) Li(1)	131.5(2)
O(71) Li(2) O(18) Li(1)	-101.67(17)
O(1) Li(1) O(18) C(18)	-108.4(5)
O(51) Li(1) O(18) C(18)	88.42(17)
O(17) Li(1) O(18) C(18)	-21.21(17)
O(2) Li(1) O(18) C(18)	-160.87(14)
Li(2) Li(1) O(18) C(18)	-152.54(19)
C(17) Li(1) O(18) C(18)	-11.32(13)
C(1) Li(1) O(18) C(18)	-136.5(2)
O(1) Li(1) O(18) Li(2)	44.1(5)
O(51) Li(1) O(18) Li(2)	-119.04(16)
O(17) Li(1) O(18) Li(2)	131.32(15)
O(2) Li(1) O(18) Li(2)	-8.33(15)
C(17) Li(1) O(18) Li(2)	141.22(14)
C(1) Li(1) O(18) Li(2)	16.1(3)
Li(2) O(18) C(18) C(19)	76.0(4)
Li(1) O(18) C(18) C(19)	-162.06(19)
Li(2) O(18) C(18) C(17)	-103.3(3)
Li(1) O(18) C(18) C(17)	18.7(2)
O(17) C(17) C(18) O(18)	-2.0(2)
C(19)#2 C(17) C(18) O(18)	177.28(17)
Li(1) C(17) C(18) O(18)	-13.17(15)
O(17) C(17) C(18) C(19)	178.70(18)
C(19)#2 C(17) C(18) C(19)	-2.0(3)

Table E35. continued...

Li(1) C(17) C(18) C(19)	167.5(2)
O(18) C(18) C(19) C(17)#2	-177.25(18)
C(17) C(18) C(19) C(17)#2	2.0(3)
O(18) C(18) C(19) C(20)	2.7(3)
C(17) C(18) C(19) C(20)	-178.13(18)
C(18) C(19) C(20) C(27)	107.8(2)
C(17)#2 C(19) C(20) C(27)	-72.3(2)
C(18) C(19) C(20) C(21)	-120.2(2)
C(17)#2 C(19) C(20) C(21)	59.7(3)
C(27) C(20) C(21) C(26)	41.3(3)
C(19) C(20) C(21) C(26)	-91.5(2)
C(27) C(20) C(21) C(22)	-138.2(2)
C(19) C(20) C(21) C(22)	89.1(2)
C(26) C(21) C(22) C(23)	2.0(3)
C(20) C(21) C(22) C(23)	-178.6(2)
C(21) C(22) C(23) C(24)	-1.0(4)
C(22) C(23) C(24) C(25)	-0.4(4)
C(23) C(24) C(25) C(26)	0.8(4)
C(22) C(21) C(26) C(25)	-1.6(3)
C(20) C(21) C(26) C(25)	178.96(19)
C(24) C(25) C(26) C(21)	0.2(3)
C(19) C(20) C(27) C(32)	4.3(3)
C(21) C(20) C(27) C(32)	-128.2(2)
C(19) C(20) C(27) C(28)	-174.29(19)
C(21) C(20) C(27) C(28)	53.3(3)
C(32) C(27) C(28) C(29)	-0.3(3)
C(20) C(27) C(28) C(29)	178.4(2)
C(27) C(28) C(29) C(30)	0.3(4)
C(28) C(29) C(30) C(31)	-0.4(4)
C(29) C(30) C(31) C(32)	0.5(4)
C(28) C(27) C(32) C(31)	0.3(4)
C(20) C(27) C(32) C(31)	-178.3(2)
C(30) C(31) C(32) C(27)	-0.4(4)
O(1) Li(1) O(51) C(51)	43.7(3)
O(17) Li(1) O(51) C(51)	-62.5(3)
O(18) Li(1) O(51) C(51)	-142.47(19)
O(2) Li(1) O(51) C(51)	129.1(2)
Li(2) Li(1) O(51) C(51)	176.21(19)
C(17) Li(1) O(51) C(51)	-85.8(2)
C(1) Li(1) O(51) C(51)	67.6(2)
Li(1) O(51) C(51) O(52)	176.16(18)
Li(1) O(51) C(51) C(50)	-3.4(4)
O(51) C(51) O(52) C(52)	-2.2(3)
C(50) C(51) O(52) C(52)	177.4(2)

Table E35. continued...

C(51) O(52) C(52) C(53)	-176.1(2)
O(2) Li(2) O(61) C(61)	63.3(3)
O(18) Li(2) O(61) C(61)	-45.6(3)
O(71) Li(2) O(61) C(61)	-161.5(2)
Li(1) Li(2) O(61) C(61)	5.2(4)
Li(2) O(61) C(61) O(62)	167.5(2)
Li(2) O(61) C(61) C(60)	-12.5(4)
O(61) C(61) O(62) C(62)	-1.1(3)
C(60) C(61) O(62) C(62)	178.9(2)
C(61) O(62) C(62) C(63)	85.7(3)
O(2) Li(2) O(71) C(71)	-53.2(3)
O(18) Li(2) O(71) C(71)	48.0(3)
O(61) Li(2) O(71) C(71)	168.1(2)
Li(1) Li(2) O(71) C(71)	-1.9(3)
Li(2) O(71) C(71) O(72)	-125.0(2)
Li(2) O(71) C(71) C(70)	54.0(3)
O(71) C(71) O(72) C(72)	3.5(3)
C(70) C(71) O(72) C(72)	-175.6(2)
C(71) O(72) C(72) C(73)	154.2(3)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1, -y+2, -z+1

#2 -x, -y+1, -z+1

Table E36. Crystal and structure refinement data for **2A•$C_3N_6H_6$•C_3H_8O**.

Identification code	**2A•$C_3N_6H_6$•C_3H_8O**
Empirical formula	$C_{38}H_{38}N_6O_5$
Formula weight	658.74 g/mole
Temperature	173(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	$P2_1/c$
Unit cell dimensions	a = 12.694(3) Å b = 22.648(5) Å c = 13.560(3) Å α = 90.00° β = 117.86(3)° γ = 90.00°
Volume	3446.3(12) $Å^3$
Z	4
Density (calculated)	1.270 Mg/m^3
Absorption coefficient	0.086 mm^{-1}
F(000)	1392
Crystal Size	0.78 mm x 0.18 mm x 0.14 mm
Color	wine red
Theta range for data collection	1.80° to 28.32°
Index ranges	$-16 \le h \le 16$, $-29 \le k \le 29$, $-17 \le l \le 18$
Reflections collected / Unique	41277 / 8362 [R_{int} = 0.1507]
Refinement method	Full-matrix-least-squares on F^2
Data / Restraints / Parameters	8362 / 0 / 595
Goodness-of-fit on F^2	0.978
Final R indices [$I>2\sigma(I)$]	R_1 = 0.0651, wR_2 = 0.0988
R indices (all data)	R_1 = 0.2007, wR_2 = 0.1312
Extinction Coefficient	0.0028(4)
Largest diff. peak and hole	0.229 and -0.213 $e/Å^3$

Table E37. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for **2A•$C_3N_6H_6$•C_3H_8O**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U_{eq}
O(1)	-1120(1)	7691(1)	2358(2)	34(1)
C(1)	-28(2)	7601(1)	2988(2)	28(1)
O(2)	289(1)	8633(1)	2922(2)	37(1)
C(2)	776(2)	8152(1)	3283(2)	26(1)
C(3)	2055(2)	8095(1)	3938(2)	24(1)
O(4)	3638(1)	7460(1)	4986(1)	32(1)
C(4)	2475(2)	7549(1)	4322(2)	25(1)
O(5)	2263(1)	6534(1)	4484(1)	34(1)
C(5)	1705(2)	7005(1)	4067(2)	26(1)
C(6)	468(2)	7049(1)	3401(2)	26(1)
C(7)	-290(2)	6497(1)	3112(2)	29(1)
C(8)	-1399(2)	6566(1)	3266(2)	30(1)
C(9)	-2475(2)	6283(1)	2594(2)	33(1)
C(10)	-3449(3)	6338(1)	2793(3)	38(1)
C(11)	-3380(3)	6676(1)	3658(3)	43(1)
C(12)	-2320(3)	6954(1)	4346(3)	46(1)
C(13)	-1339(3)	6897(1)	4165(2)	39(1)
C(14)	-511(2)	6231(1)	1998(2)	35(1)
C(15)	-560(3)	5620(1)	1870(3)	53(1)
C(16)	-764(3)	5362(2)	868(4)	72(1)
C(17)	-894(3)	5712(2)	-9(4)	74(1)
C(18)	-831(3)	6316(2)	98(3)	63(1)
C(19)	-656(3)	6572(2)	1090(3)	45(1)
C(20)	2832(2)	8636(1)	4137(2)	28(1)
C(21)	3920(2)	8507(1)	3960(2)	28(1)
C(22)	3761(3)	8187(1)	3027(2)	39(1)
C(23)	4714(3)	8063(1)	2826(3)	48(1)
C(24)	5838(3)	8262(1)	3544(3)	46(1)
C(25)	6002(3)	8588(1)	4457(3)	44(1)
C(26)	5056(2)	8704(1)	4672(3)	36(1)
C(27)	3114(2)	8953(1)	5221(2)	31(1)
C(28)	3445(3)	9545(1)	5342(3)	43(1)
C(29)	3699(3)	9852(2)	6310(3)	55(1)
C(30)	3612(3)	9577(2)	7167(3)	60(1)
C(31)	3296(3)	8988(2)	7073(3)	61(1)
C(32)	3052(3)	8681(1)	6105(3)	49(1)
N(50)	-5830(2)	9307(1)	-124(2)	34(1)
C(50)	-4781(2)	9048(1)	378(2)	24(1)
N(51)	-3804(2)	9387(1)	652(2)	26(1)

Table E37. continued...

N(52)	-1752(2)	9389(1)	1512(2)	35(1)
C(52)	-2764(2)	9110(1)	1217(2)	26(1)
N(53)	-2705(2)	8531(1)	1520(2)	25(1)
N(54)	-3636(2)	7637(1)	1320(2)	32(1)
C(54)	-3732(2)	8212(1)	1119(2)	23(1)
N(55)	-4791(2)	8460(1)	555(2)	25(1)
C(60)	3127(3)	5267(2)	6730(3)	55(1)
O(61)	1685(2)	5363(1)	4770(2)	36(1)
C(61)	1836(3)	5240(1)	5871(2)	42(1)
C(62)	1057(3)	5647(2)	6132(4)	58(1)

Table E38. Bond lengths (Å) and angles (°) for **2A•$C_3N_6H_6$•C_3H_8O**.

Atoms	Length	Angle
O(1)-C(1)	1.259(3)	
C(1)-C(6)	1.394(3)	
C(1)-C(2)	1.542(3)	
O(2)-C(2)	1.234(3)	
C(2)-C(3)	1.447(3)	
C(3)-C(4)	1.351(3)	
C(3)-C(20)	1.516(3)	
O(4)-C(4)	1.337(3)	
O(4)-H(4)	0.98(4)	
C(4)-C(5)	1.508(3)	
O(5)-C(5)	1.259(3)	
C(5)-C(6)	1.403(3)	
C(6)-C(7)	1.512(3)	
C(7)-C(8)	1.526(3)	
C(7)-C(14)	1.526(4)	
C(7)-H(7)	0.99(2)	
C(8)-C(9)	1.394(3)	
C(8)-C(13)	1.402(4)	
C(9)-C(10)	1.389(4)	
C(9)-H(9)	0.97(2)	
C(10)-C(11)	1.370(4)	
C(10)-H(10)	0.98(2)	
C(11)-C(12)	1.379(4)	
C(11)-H(11)	1.03(3)	
C(12)-C(13)	1.384(4)	
C(12)-H(12)	1.03(3)	
C(13)-H(13)	0.97(3)	
C(14)-C(19)	1.391(4)	
C(14)-C(15)	1.393(4)	
C(15)-C(16)	1.387(5)	
C(15)-H(15)	1.01(3)	
C(16)-C(17)	1.373(6)	
C(16)-H(16)	0.95(3)	
C(17)-C(18)	1.375(5)	
C(17)-H(17)	0.99(3)	
C(18)-C(19)	1.383(4)	
C(18)-H(18)	1.01(3)	
C(19)-H(19)	0.97(2)	
C(20)-C(27)	1.522(4)	
C(20)-C(21)	1.535(3)	
C(20)-H(20)	0.99(2)	

Table E38. continued...

C(21)-C(26)	1.382(3)
C(21)-C(22)	1.389(4)
C(22)-C(23)	1.386(4)
C(22)-H(22)	0.99(2)
C(23)-C(24)	1.374(4)
C(23)-H(23)	0.95(3)
C(24)-C(25)	1.371(4)
C(24)-H(24)	1.02(3)
C(25)-C(26)	1.388(4)
C(25)-H(25)	0.94(3)
C(26)-H(26)	0.96(3)
C(27)-C(32)	1.382(4)
C(27)-C(28)	1.390(4)
C(28)-C(29)	1.384(4)
C(28)-H(28)	0.98(2)
C(29)-C(30)	1.368(5)
C(29)-H(29)	1.04(3)
C(30)-C(31)	1.382(4)
C(30)-H(30)	1.00(3)
C(31)-C(32)	1.385(4)
C(31)-H(31)	1.04(3)
C(32)-H(32)	1.02(3)
N(50)-C(50)	1.317(3)
N(50)-H(50A)	0.87(3)
N(50)-H(50B)	0.89(3)
C(50)-N(51)	1.354(3)
C(50)-N(55)	1.355(3)
N(51)-C(52)	1.333(3)
N(52)-C(52)	1.315(3)
N(52)-H(52A)	0.93(3)
N(52)-H(52B)	0.90(3)
C(52)-N(53)	1.366(3)
N(53)-C(54)	1.362(3)
N(53)-H(53)	1.01(3)
N(54)-C(54)	1.325(3)
N(54)-H(54A)	0.88(2)
N(54)-H(54B)	0.89(3)
C(54)-N(55)	1.321(3)
C(60)-C(61)	1.504(4)
C(60)-H(60A)	0.99(3)
C(60)-H(60B)	1.07(4)
C(60)-H(60C)	1.07(3)
O(61)-C(61)	1.441(3)
O(61)-H(61)	0.98(3)

Table E38. continued...

C(61)-C(62)	1.509(4)
C(61)-H(61A)	1.02(3)
C(62)-H(62A)	1.00(3)
C(62)-H(62B)	1.08(3)
C(62)-H(62C)	1.03(3)

O(1)-C(1)-C(6)	124.4(2)
O(1)-C(1)-C(2)	115.5(2)
C(6)-C(1)-C(2)	120.1(2)
O(2)-C(2)-C(3)	122.2(2)
O(2)-C(2)-C(1)	117.6(2)
C(3)-C(2)-C(1)	120.2(2)
C(4)-C(3)-C(2)	116.5(2)
C(4)-C(3)-C(20)	124.3(2)
C(2)-C(3)-C(20)	119.1(2)
C(4)-O(4)-H(4)	110(2)
O(4)-C(4)-C(3)	120.7(2)
O(4)-C(4)-C(5)	115.1(2)
C(3)-C(4)-C(5)	124.2(2)
O(5)-C(5)-C(6)	125.0(2)
O(5)-C(5)-C(4)	114.9(2)
C(6)-C(5)-C(4)	120.1(2)
C(1)-C(6)-C(5)	118.8(2)
C(1)-C(6)-C(7)	121.6(2)
C(5)-C(6)-C(7)	119.6(2)
C(6)-C(7)-C(8)	113.4(2)
C(6)-C(7)-C(14)	112.6(2)
C(8)-C(7)-C(14)	114.9(2)
C(6)-C(7)-H(7)	107.6(12)
C(8)-C(7)-H(7)	104.6(12)
C(14)-C(7)-H(7)	102.5(12)
C(9)-C(8)-C(13)	116.9(3)
C(9)-C(8)-C(7)	123.3(2)
C(13)-C(8)-C(7)	119.6(2)
C(10)-C(9)-C(8)	121.4(3)
C(10)-C(9)-H(9)	122.0(13)
C(8)-C(9)-H(9)	116.5(13)
C(11)-C(10)-C(9)	120.6(3)
C(11)-C(10)-H(10)	120.6(14)
C(9)-C(10)-H(10)	118.8(14)
C(10)-C(11)-C(12)	119.1(3)
C(10)-C(11)-H(11)	120.0(16)
C(12)-C(11)-H(11)	120.7(16)
C(11)-C(12)-C(13)	120.8(3)

Table E38. continued...

C(11)-C(12)-H(12)	118.8(16)
C(13)-C(12)-H(12)	120.4(16)
C(12)-C(13)-C(8)	121.1(3)
C(12)-C(13)-H(13)	120.1(16)
C(8)-C(13)-H(13)	118.8(16)
C(19)-C(14)-C(15)	117.6(3)
C(19)-C(14)-C(7)	122.9(2)
C(15)-C(14)-C(7)	119.5(3)
C(16)-C(15)-C(14)	121.1(4)
C(16)-C(15)-H(15)	123.1(16)
C(14)-C(15)-H(15)	115.8(16)
C(17)-C(16)-C(15)	119.9(4)
C(17)-C(16)-H(16)	127(2)
C(15)-C(16)-H(16)	113(2)
C(16)-C(17)-C(18)	120.4(4)
C(16)-C(17)-H(17)	120.0(19)
C(18)-C(17)-H(17)	119.4(19)
C(17)-C(18)-C(19)	119.6(4)
C(17)-C(18)-H(18)	119(2)
C(19)-C(18)-H(18)	121(2)
C(18)-C(19)-C(14)	121.5(3)
C(18)-C(19)-H(19)	120.3(16)
C(14)-C(19)-H(19)	117.9(16)
C(3)-C(20)-C(27)	113.1(2)
C(3)-C(20)-C(21)	112.1(2)
C(27)-C(20)-C(21)	114.7(2)
C(3)-C(20)-H(20)	103.8(13)
C(27)-C(20)-H(20)	106.5(13)
C(21)-C(20)-H(20)	105.4(13)
C(26)-C(21)-C(22)	117.7(3)
C(26)-C(21)-C(20)	123.4(2)
C(22)-C(21)-C(20)	118.9(2)
C(23)-C(22)-C(21)	121.0(3)
C(23)-C(22)-H(22)	119.7(14)
C(21)-C(22)-H(22)	119.2(14)
C(24)-C(23)-C(22)	120.5(3)
C(24)-C(23)-H(23)	121.9(17)
C(22)-C(23)-H(23)	117.6(17)
C(25)-C(24)-C(23)	119.1(3)
C(25)-C(24)-H(24)	120.8(17)
C(23)-C(24)-H(24)	120.1(17)
C(24)-C(25)-C(26)	120.7(3)
C(24)-C(25)-H(25)	121.6(19)
C(26)-C(25)-H(25)	117.7(19)

Table E38. continued...

C(21)-C(26)-C(25)	121.0(3)
C(21)-C(26)-H(26)	118.2(16)
C(25)-C(26)-H(26)	120.9(16)
C(32)-C(27)-C(28)	117.6(3)
C(32)-C(27)-C(20)	123.0(2)
C(28)-C(27)-C(20)	119.3(3)
C(29)-C(28)-C(27)	121.3(3)
C(29)-C(28)-H(28)	121.1(15)
C(27)-C(28)-H(28)	117.6(15)
C(30)-C(29)-C(28)	120.1(3)
C(30)-C(29)-H(29)	124.5(17)
C(28)-C(29)-H(29)	115.4(17)
C(29)-C(30)-C(31)	119.8(3)
C(29)-C(30)-H(30)	121.2(17)
C(31)-C(30)-H(30)	119.0(17)
C(30)-C(31)-C(32)	119.8(4)
C(30)-C(31)-H(31)	122.0(18)
C(32)-C(31)-H(31)	118.1(18)
C(27)-C(32)-C(31)	121.3(3)
C(27)-C(32)-H(32)	119.2(16)
C(31)-C(32)-H(32)	119.4(16)
C(50)-N(50)-H(50A)	122.0(18)
C(50)-N(50)-H(50B)	118.3(16)
H(50A)-N(50)-H(50B)	119(2)
N(50)-C(50)-N(51)	117.7(2)
N(50)-C(50)-N(55)	116.0(2)
N(51)-C(50)-N(55)	126.3(2)
C(52)-N(51)-C(50)	115.2(2)
C(52)-N(52)-H(52A)	117.2(17)
C(52)-N(52)-H(52B)	120.3(16)
H(52A)-N(52)-H(52B)	121(2)
N(52)-C(52)-N(51)	120.9(2)
N(52)-C(52)-N(53)	117.4(2)
N(51)-C(52)-N(53)	121.7(2)
C(54)-N(53)-C(52)	118.7(2)
C(54)-N(53)-H(53)	118.0(15)
C(52)-N(53)-H(53)	122.6(15)
C(54)-N(54)-H(54A)	117.0(16)
C(54)-N(54)-H(54B)	118.2(17)
H(54A)-N(54)-H(54B)	124(2)
N(55)-C(54)-N(54)	120.6(2)
N(55)-C(54)-N(53)	122.1(2)
N(54)-C(54)-N(53)	117.4(2)
C(54)-N(55)-C(50)	115.4(2)

Table E38. continued...

C(61)-C(60)-H(60A)	109.9(17)
C(61)-C(60)-H(60B)	108.9(18)
H(60A)-C(60)-H(60B)	111(2)
C(61)-C(60)-H(60C)	107.7(15)
H(60A)-C(60)-H(60C)	110(2)
H(60B)-C(60)-H(60C)	109(2)
C(61)-O(61)-H(61)	111(2)
O(61)-C(61)-C(60)	111.3(3)
O(61)-C(61)-C(62)	110.0(3)
C(60)-C(61)-C(62)	112.9(3)
O(61)-C(61)-H(61A)	102.3(14)
C(60)-C(61)-H(61A)	109.1(14)
C(62)-C(61)-H(61A)	110.8(14)
C(61)-C(62)-H(62A)	110.9(17)
C(61)-C(62)-H(62B)	104.4(16)
H(62A)-C(62)-H(62B)	104(2)
C(61)-C(62)-H(62C)	109.7(17)
H(62A)-C(62)-H(62C)	114(2)
H(62B)-C(62)-H(62C)	113(2)

Table E39. Anisotropic displacement parameters ($Å^2 \times 10^3$) for **2A•$C_3N_6H_6$•C_3H_8O**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+...+2hka^*b^*U_{12}]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O(1)	20(1)	28(1)	47(1)	7(1)	9(1)	3(1)
C(1)	21(2)	29(2)	32(2)	4(1)	11(1)	2(1)
O(2)	27(1)	26(1)	53(1)	11(1)	14(1)	7(1)
C(2)	27(2)	24(1)	28(2)	4(1)	14(1)	6(1)
C(3)	21(1)	21(1)	29(2)	0(1)	10(1)	2(1)
O(4)	21(1)	23(1)	41(1)	1(1)	6(1)	1(1)
C(4)	17(1)	30(2)	26(2)	-1(1)	7(1)	2(1)
O(5)	22(1)	23(1)	48(1)	6(1)	8(1)	4(1)
C(5)	24(2)	20(1)	34(2)	3(1)	11(1)	3(1)
C(6)	18(1)	22(1)	35(2)	3(1)	9(1)	2(1)
C(7)	18(1)	21(1)	39(2)	5(1)	6(1)	1(1)
C(8)	27(2)	23(1)	33(2)	7(1)	9(1)	-1(1)
C(9)	29(2)	33(2)	37(2)	-2(1)	14(2)	-1(1)
C(10)	27(2)	42(2)	42(2)	4(2)	15(2)	-3(1)
C(11)	41(2)	48(2)	46(2)	6(2)	26(2)	-3(2)
C(12)	52(2)	51(2)	44(2)	-5(2)	30(2)	-10(2)
C(13)	37(2)	40(2)	38(2)	-1(2)	16(2)	-10(2)
C(14)	18(1)	32(2)	52(2)	-7(2)	13(1)	1(1)
C(15)	37(2)	42(2)	74(3)	-13(2)	22(2)	-1(2)
C(16)	50(2)	56(3)	107(4)	-38(3)	33(2)	-7(2)
C(17)	45(2)	103(4)	75(3)	-46(3)	29(2)	-1(2)
C(18)	47(2)	84(3)	61(3)	-25(2)	28(2)	-5(2)
C(19)	34(2)	50(2)	51(2)	-9(2)	19(2)	-2(2)
C(20)	24(2)	22(1)	34(2)	2(1)	10(1)	0(1)
C(21)	30(2)	24(1)	30(2)	6(1)	12(1)	3(1)
C(22)	31(2)	46(2)	39(2)	-4(2)	15(2)	-7(2)
C(23)	52(2)	55(2)	42(2)	-9(2)	27(2)	-6(2)
C(24)	39(2)	57(2)	50(2)	7(2)	28(2)	4(2)
C(25)	28(2)	59(2)	47(2)	2(2)	18(2)	-4(2)
C(26)	28(2)	38(2)	37(2)	0(2)	13(2)	-2(1)
C(27)	24(2)	27(2)	39(2)	-2(1)	12(1)	-1(1)
C(28)	50(2)	29(2)	44(2)	-1(2)	18(2)	0(1)
C(29)	60(2)	34(2)	59(2)	-9(2)	18(2)	1(2)
C(30)	67(2)	51(2)	63(3)	-23(2)	31(2)	-5(2)
C(31)	83(3)	59(2)	54(2)	-20(2)	44(2)	-20(2)
C(32)	63(2)	38(2)	53(2)	-10(2)	34(2)	-11(2)
N(50)	19(1)	19(1)	57(2)	4(1)	13(1)	0(1)
C(50)	22(2)	22(1)	27(2)	1(1)	11(1)	5(1)
N(51)	19(1)	20(1)	35(1)	2(1)	9(1)	1(1)

Table E39. continued...

N(52)	17(1)	25(1)	54(2)	9(1)	9(1)	-1(1)
C(52)	26(2)	20(1)	30(2)	1(1)	12(1)	2(1)
N(53)	20(1)	19(1)	35(1)	3(1)	11(1)	0(1)
N(54)	21(1)	20(1)	50(2)	4(1)	11(1)	0(1)
C(54)	26(2)	18(1)	27(1)	2(1)	14(1)	1(1)
N(55)	21(1)	19(1)	32(1)	2(1)	10(1)	1(1)
C(60)	56(2)	52(2)	50(2)	3(2)	18(2)	5(2)
O(61)	41(1)	26(1)	41(1)	-2(1)	19(1)	-1(1)
C(61)	51(2)	36(2)	38(2)	-1(1)	19(2)	-11(2)
C(62)	45(2)	80(3)	54(2)	-15(2)	26(2)	-12(2)

Table E40. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for **2A•$C_3N_6H_6$•C_3H_8O**.

Atom	x	y	z	U_{eq}
H(4)	3830(30)	7038(16)	5020(30)	111(14)
H(7)	186(19)	6186(9)	3652(18)	23(6)
H(9)	-2522(18)	6069(9)	1957(18)	14(6)
H(10)	-4200(20)	6139(10)	2290(20)	34(7)
H(11)	-4120(20)	6742(12)	3760(20)	61(9)
H(12)	-2270(20)	7207(12)	5000(20)	63(9)
H(13)	-590(20)	7090(11)	4660(20)	51(9)
H(15)	-450(20)	5384(12)	2540(20)	48(9)
H(16)	-800(30)	4942(16)	880(30)	81(12)
H(17)	-960(30)	5530(14)	-700(30)	77(11)
H(18)	-960(30)	6567(15)	-560(30)	85(13)
H(19)	-540(20)	6995(12)	1200(20)	46(8)
H(20)	2330(20)	8907(10)	3524(19)	31(7)
H(22)	2960(20)	8037(10)	2510(20)	38(7)
H(23)	4560(20)	7831(12)	2190(20)	52(9)
H(24)	6540(30)	8166(12)	3400(20)	69(10)
H(25)	6760(30)	8735(13)	4960(30)	67(10)
H(26)	5170(20)	8927(11)	5320(20)	50(9)
H(28)	3510(20)	9734(11)	4720(20)	39(8)
H(29)	3910(30)	10296(14)	6310(20)	80(10)
H(30)	3770(30)	9796(13)	7870(30)	72(10)
H(31)	3220(30)	8763(14)	7710(30)	88(12)
H(32)	2810(20)	8249(13)	6040(20)	64(9)
H(50A)	-5900(20)	9689(13)	-230(20)	51(9)
H(50B)	-6470(20)	9091(11)	-280(20)	38(8)
H(52A)	-1790(20)	9756(13)	1190(20)	57(9)
H(52B)	-1060(20)	9199(11)	1890(20)	39(8)
H(53)	-1930(20)	8314(11)	1930(20)	54(8)
H(54A)	-4300(20)	7428(11)	1006(19)	31(8)
H(54B)	-2910(20)	7484(11)	1690(20)	46(9)
H(60A)	3600(30)	4992(13)	6520(20)	70(11)
H(60B)	3200(30)	5155(14)	7530(30)	97(13)
H(60C)	3420(20)	5712(13)	6760(20)	66(10)
H(61)	1790(30)	5786(16)	4680(30)	97(13)
H(61A)	1550(20)	4814(12)	5800(20)	46(8)
H(62A)	220(30)	5647(12)	5510(30)	67(10)
H(62B)	1380(30)	6082(15)	6110(20)	72(10)
H(62C)	1130(30)	5540(13)	6900(30)	71(10)

Table E41. Torsion angles (°) for **2A•$C_3N_6H_6$•C_3H_8O**.

Atoms	Angle
O(1) C(1) C(2) O(2)	2.8(3)
C(6) C(1) C(2) O(2)	-178.0(2)
O(1) C(1) C(2) C(3)	-175.7(2)
C(6) C(1) C(2) C(3)	3.5(3)
O(2) C(2) C(3) C(4)	177.4(2)
C(1) C(2) C(3) C(4)	-4.1(3)
O(2) C(2) C(3) C(20)	-3.4(4)
C(1) C(2) C(3) C(20)	175.1(2)
C(2) C(3) C(4) O(4)	-176.3(2)
C(20) C(3) C(4) O(4)	4.6(4)
C(2) C(3) C(4) C(5)	2.9(4)
C(20) C(3) C(4) C(5)	-176.3(2)
O(4) C(4) C(5) O(5)	-2.1(3)
C(3) C(4) C(5) O(5)	178.8(2)
O(4) C(4) C(5) C(6)	178.4(2)
C(3) C(4) C(5) C(6)	-0.8(4)
O(1) C(1) C(6) C(5)	177.8(2)
C(2) C(1) C(6) C(5)	-1.3(4)
O(1) C(1) C(6) C(7)	0.3(4)
C(2) C(1) C(6) C(7)	-178.8(2)
O(5) C(5) C(6) C(1)	-179.6(2)
C(4) C(5) C(6) C(1)	-0.1(4)
O(5) C(5) C(6) C(7)	-2.0(4)
C(4) C(5) C(6) C(7)	177.5(2)
C(1) C(6) C(7) C(8)	-48.8(3)
C(5) C(6) C(7) C(8)	133.7(2)
C(1) C(6) C(7) C(14)	83.8(3)
C(5) C(6) C(7) C(14)	-93.7(3)
C(6) C(7) C(8) C(9)	146.7(2)
C(14) C(7) C(8) C(9)	15.2(4)
C(6) C(7) C(8) C(13)	-37.4(3)
C(14) C(7) C(8) C(13)	-168.9(2)
C(13) C(8) C(9) C(10)	1.2(4)
C(7) C(8) C(9) C(10)	177.2(2)
C(8) C(9) C(10) C(11)	0.5(4)
C(9) C(10) C(11) C(12)	-1.3(4)
C(10) C(11) C(12) C(13)	0.4(4)
C(11) C(12) C(13) C(8)	1.3(4)
C(9) C(8) C(13) C(12)	-2.1(4)
C(7) C(8) C(13) C(12)	-178.3(3)
C(6) C(7) C(14) C(19)	-35.2(3)

Table E41. continued...

C(8) C(7) C(14) C(19)	96.6(3)
C(6) C(7) C(14) C(15)	144.0(2)
C(8) C(7) C(14) C(15)	-84.1(3)
C(19) C(14) C(15) C(16)	-0.9(4)
C(7) C(14) C(15) C(16)	179.8(3)
C(14) C(15) C(16) C(17)	1.4(5)
C(15) C(16) C(17) C(18)	-0.4(6)
C(16) C(17) C(18) C(19)	-1.1(5)
C(17) C(18) C(19) C(14)	1.6(5)
C(15) C(14) C(19) C(18)	-0.6(4)
C(7) C(14) C(19) C(18)	178.7(3)
C(4) C(3) C(20) C(27)	-87.4(3)
C(2) C(3) C(20) C(27)	93.5(3)
C(4) C(3) C(20) C(21)	44.2(3)
C(2) C(3) C(20) C(21)	-134.9(2)
C(3) C(20) C(21) C(26)	-136.2(3)
C(27) C(20) C(21) C(26)	-5.4(4)
C(3) C(20) C(21) C(22)	45.3(3)
C(27) C(20) C(21) C(22)	176.1(2)
C(26) C(21) C(22) C(23)	0.9(4)
C(20) C(21) C(22) C(23)	179.4(3)
C(21) C(22) C(23) C(24)	-0.8(5)
C(22) C(23) C(24) C(25)	-0.4(5)
C(23) C(24) C(25) C(26)	1.5(5)
C(22) C(21) C(26) C(25)	0.2(4)
C(20) C(21) C(26) C(25)	-178.3(2)
C(24) C(25) C(26) C(21)	-1.4(4)
C(3) C(20) C(27) C(32)	21.8(4)
C(21) C(20) C(27) C(32)	-108.5(3)
C(3) C(20) C(27) C(28)	-157.9(2)
C(21) C(20) C(27) C(28)	71.8(3)
C(32) C(27) C(28) C(29)	-0.3(4)
C(20) C(27) C(28) C(29)	179.5(3)
C(27) C(28) C(29) C(30)	-0.9(5)
C(28) C(29) C(30) C(31)	1.4(5)
C(29) C(30) C(31) C(32)	-0.9(5)
C(28) C(27) C(32) C(31)	0.8(4)
C(20) C(27) C(32) C(31)	-178.9(3)
C(30) C(31) C(32) C(27)	-0.2(5)
N(50) C(50) N(51) C(52)	176.2(2)
N(55) C(50) N(51) C(52)	-4.9(4)
C(50) N(51) C(52) N(52)	178.6(2)
C(50) N(51) C(52) N(53)	-1.9(3)
N(52) C(52) N(53) C(54)	-172.5(2)

Table E41. continued...

N(51) C(52) N(53) C(54)	8.0(4)
C(52) N(53) C(54) N(55)	-7.9(4)
C(52) N(53) C(54) N(54)	172.4(2)
N(54) C(54) N(55) C(50)	-178.6(2)
N(53) C(54) N(55) C(50)	1.7(3)
N(50) C(50) N(55) C(54)	-176.0(2)
N(51) C(50) N(55) C(54)	5.0(4)

Table E42. Hydrogen bonds, lengths (Å) and angles (°) for **2A•$C_3N_6H_6$•C_3H_8O**.

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(4)-H(4)...N(55)#1	0.98(4)	1.92(4)	2.734(3)	139(3)
N(50)-H(50A)...N(51)#2	0.87(3)	2.16(3)	3.027(3)	174(3)
N(50)-H(50B)...O(5)#3	0.89(3)	2.05(3)	2.928(3)	169(2)
N(52)-H(52A)...O(61)#4	0.93(3)	1.93(3)	2.833(3)	161(2)
N(52)-H(52B)...O(2)	0.90(3)	2.07(3)	2.944(3)	165(2)
N(53)-H(53)...O(1)	1.01(3)	1.68(3)	2.612(3)	152(2)
N(54)-H(54A)...O(4)#3	0.88(2)	2.34(2)	3.073(3)	141(2)
N(54)-H(54B)...O(1)	0.89(3)	2.07(3)	2.827(3)	143(2)
O(61)-H(61)...O(5)	0.98(3)	1.86(4)	2.827(2)	168(3)

Symmetry transformations used to generate equivalent atoms:

#1 x+1, -y+3/2, z+1/2
#2 -x-1, -y+2, -z
#3 x-1, -y+3/2, z-1/2
#4 -x, y+1/2, -z+1/2

Table E43. Crystal and structure refinement data for **2A•$(C_6H_{15}N)_2$**.

Identification code	**2A•$(C_6H_{15}N)_2$**
Empirical formula	$C_{44}H_{54}N_2O_4$
Formula weight	674.89 g/mole
Temperature	173(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	$P2_1/c$
Unit cell dimensions	a = 12.483(3) Å b = 9.853(2) Å c = 16.487(3) Å α = 90.00° β = 108.58(3)° γ = 90.00°
Volume	1922.1(7) $Å^3$
Z	2
Density (calculated)	1.166 Mg/m^3
Absorption coefficient	0.074 mm^{-1}
F(000)	728
Crystal Size	0.46 mm x 0.44 mm x 0.22 mm
Color	wine red
Theta range for data collection	2.44° to 28.29°
Index ranges	$-16 \le h \le 16$, $-11 \le k \le 12$, $-18 \le l \le 21$
Reflections collected / Unique	11960 / 4523 [R_{int} = 0.0583]
Refinement method	Full-matrix-least-squares on F^2
Data / Restraints / Parameters	4523 / 0 / 335
Goodness-of-fit on F^2	0.872
Final R indices [$I>2\sigma(I)$]	R_1 = 0.0552, wR_2 = 0.1279
R indices (all data)	R_1 = 0.1177, wR_2 = 0.1489
Extinction Coefficient	0.0052(19)
Largest diff. peak and hole	0.486 and -0.200 e/A^3

Table E44. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for **2A•$(C_6H_{15}N)_2$**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U_{eq}	Occ.
C(1)	10278(2)	4539(2)	872(1)	31(1)	1
O(1)	10402(1)	4155(2)	1627(1)	48(1)	1
N(1)	8739(2)	3921(2)	2396(1)	46(1)	1
C(2)	9062(2)	4889(2)	320(1)	30(1)	1
O(2)	8318(1)	4717(1)	674(1)	40(1)	1
C(3)	8835(2)	5360(2)	-524(1)	28(1)	1
C(4)	7656(2)	5783(2)	-1078(1)	29(1)	1
C(5)	6900(1)	4624(2)	-1535(1)	30(1)	1
C(6)	6314(2)	4741(2)	-2403(1)	38(1)	1
C(7)	5597(2)	3724(2)	-2846(2)	50(1)	1
C(8)	5471(2)	2557(3)	-2431(2)	51(1)	1
C(9)	6054(2)	2401(2)	-1567(2)	44(1)	1
C(10)	6768(2)	3430(2)	-1121(1)	36(1)	1
C(11)	7085(2)	6742(2)	-606(1)	34(1)	1
C(12)	6164(2)	6392(2)	-358(1)	44(1)	1
C(13)	5710(2)	7330(3)	81(2)	57(1)	1
C(14)	6147(2)	8597(3)	260(2)	60(1)	1
C(15)	7051(2)	8971(3)	-6(2)	61(1)	1
C(16)	7507(2)	8044(2)	-435(2)	48(1)	1
C(20)	9655(2)	4236(3)	3215(2)	62(1)	1
C(21)	9393(3)	3787(4)	4010(2)	69(1)	1
C(22)	7684(2)	4709(3)	2271(2)	62(1)	1
C(23)	7830(5)	6226(3)	2204(3)	88(1)	1
C(24)	8545(3)	2398(3)	2347(2)	58(1)	1
C(25)	8024(3)	1883(3)	1462(2)	66(1)	1

Table E45. Bond lengths (Å) and angles (°) for **2A•($C_6H_{15}N$)$_2$**.

Atoms	Length	Angle
C(1)-O(1)	1.264(2)	
C(1)-C(3)#1	1.404(2)	
C(1)-C(2)	1.539(3)	
N(1)-C(22)	1.485(3)	
N(1)-C(20)	1.497(3)	
N(1)-C(24)	1.518(3)	
N(1)-H(1)	0.98(3)	
C(2)-O(2)	1.256(2)	
C(2)-C(3)	1.408(2)	
C(3)-C(1)#1	1.404(2)	
C(3)-C(4)	1.522(3)	
C(4)-C(5)	1.519(3)	
C(4)-C(11)	1.536(3)	
C(4)-H(4A)	0.988(19)	
C(5)-C(6)	1.389(3)	
C(5)-C(10)	1.396(3)	
C(6)-C(7)	1.386(3)	
C(6)-H(6A)	1.03(2)	
C(7)-C(8)	1.373(3)	
C(7)-H(7A)	0.92(3)	
C(8)-C(9)	1.386(3)	
C(8)-H(8A)	0.98(2)	
C(9)-C(10)	1.395(3)	
C(9)-H(9A)	0.99(2)	
C(10)-H(10A)	0.967(19)	
C(11)-C(16)	1.381(3)	
C(11)-C(12)	1.382(3)	
C(12)-C(13)	1.400(3)	
C(12)-H(12A)	1.02(2)	
C(13)-C(14)	1.356(4)	
C(13)-H(13A)	0.98(3)	
C(14)-C(15)	1.384(4)	
C(14)-H(14A)	0.98(2)	
C(15)-C(16)	1.384(3)	
C(15)-H(15A)	0.96(3)	
C(16)-H(16A)	0.98(2)	
C(20)-C(21)	1.514(4)	
C(20)-H(20A)	1.04(3)	
C(20)-H(20B)	0.98(3)	
C(21)-H(21A)	0.98(4)	
C(21)-H(21B)	0.99(3)	

Table E45. continued...

C(21)-H(21C)	1.02(3)
C(22)-C(23)	1.514(4)
C(22)-H(22A)	1.03(3)
C(22)-H(22B)	1.05(3)
C(23)-H(23A)	0.92(4)
C(23)-H(23B)	0.98(3)
C(23)-H(23C)	1.04(4)
C(24)-C(25)	1.486(4)
C(24)-H(24A)	1.11(3)
C(24)-H(24B)	1.02(3)
C(25)-H(25A)	1.04(3)
C(25)-H(25B)	0.99(3)
C(25)-H(25C)	1.07(4)

O(1)-C(1)-C(3)#1	123.94(17)
O(1)-C(1)-C(2)	115.97(15)
C(3)#1-C(1)-C(2)	120.09(16)
C(22)-N(1)-C(20)	113.9(2)
C(22)-N(1)-C(24)	113.0(2)
C(20)-N(1)-C(24)	108.4(2)
C(22)-N(1)-H(1)	103.7(15)
C(20)-N(1)-H(1)	106.7(15)
C(24)-N(1)-H(1)	110.8(15)
O(2)-C(2)-C(3)	123.93(17)
O(2)-C(2)-C(1)	115.57(15)
C(3)-C(2)-C(1)	120.50(15)
C(1)#1-C(3)-C(2)	119.37(17)
C(1)#1-C(3)-C(4)	118.28(16)
C(2)-C(3)-C(4)	122.35(16)
C(5)-C(4)-C(3)	114.88(15)
C(5)-C(4)-C(11)	113.48(15)
C(3)-C(4)-C(11)	112.35(15)
C(5)-C(4)-H(4A)	106.2(11)
C(3)-C(4)-H(4A)	105.3(10)
C(11)-C(4)- H(4A)	103.4(10)
C(6)-C(5)-C(10)	117.89(18)
C(6)-C(5)-C(4)	119.25(17)
C(10)-C(5)-C(4)	122.86(18)
C(7)-C(6)-C(5)	121.5(2)
C(7)-C(6)-H(6A)	121.4(12)
C(5)-C(6)-H(6A)	117.0(12)
C(8)-C(7)-C(6)	120.0(2)
C(8)-C(7)-H(7A)	117.2(15)
C(6)-C(7)-H(7A)	122.8(15)

Table E45. continued...

C(7)-C(8)-C(9)	120.0(2)
C(7)-C(8)-H(8A)	119.6(14)
C(9)-C(8)-H(8A)	120.5(14)
C(8)-C(9)-C(10)	120.0(2)
C(8)-C(9)-H(9A)	119.5(12)
C(10)-C(9)-H(9A)	120.5(12)
C(9)-C(10)-C(5)	120.7(2)
C(9)-C(10)-H(10A)	121.0(10)
C(5)-C(10)-H(10A)	118.3(10)
C(16)-C(11)-C(12)	118.07(19)
C(16)-C(11)-C(4)	117.81(18)
C(12)-C(11)-C(4)	124.10(18)
C(11)-C(12)-C(13)	120.0(2)
C(11)-C(12)-H(12A)	118.8(12)
C(13)-C(12)-H(12A)	121.2(12)
C(14)-C(13)-C(12)	121.3(2)
C(14)-C(13)-H(13A)	121.8(14)
C(12)-C(13)-H(13A)	116.9(15)
C(13)-C(14)-C(15)	119.2(2)
C(13)-C(14)-H(14A)	124.2(14)
C(15)-C(14)-H(14A)	116.5(14)
C(14)-C(15)-C(16)	119.7(3)
C(14)-C(15)-H(15A)	122.6(15)
C(16)-C(15)-H(15A)	117.7(15)
C(11)-C(16)-C(15)	121.7(2)
C(11)-C(16)-H(16A)	118.6(13)
C(15)-C(16)-H(16A)	119.7(13)
N(1)-C(20)-C(21)	114.0(2)
N(1)-C(20)-H(20A)	102.3(15)
C(21)-C(20)-H(20A)	110.4(15)
N(1)-C(20)-H(20B)	102.8(15)
C(21)-C(20)-H(20B)	108.5(16)
H(20A)-C(20)-H(20B)	119(2)
C(20)-C(21)-H(21A)	108(2)
C(20)-C(21)-H(21B)	108.6(17)
H(21A)-C(21)-H(21B)	109(3)
C(20)-C(21)-H(21C)	110.0(16)
H(21A)-C(21)-H(21C)	105(3)
H(21B)-C(21)-H(21C)	116(2)
N(1)-C(22)-C(23)	113.9(3)
N(1)-C(22)-H(22A)	107.1(14)
C(23)-C(22)-H(22A)	109.8(13)
N(1)-C(22)-H(22B)	105.2(15)
C(23)-C(22)-H(22B)	111.9(15)

Table E45. continued...

H(22A)-C(22)-H(22B)	109(2)
C(22)-C(23)-H(23A)	105(2)
C(22)-C(23)-H(23B)	111.3(17)
H(23A)-C(23)-H(23B)	109(3)
C(22)-C(23)-H(23C)	106.7(17)
H(23A)-C(23)-H(23C)	112(3)
H(23B)-C(23)-H(23C)	112(3)
N(1)-C(24)-C(25)	113.7(2)
C(25)-C(24)-H(24A)	106.4(15)
N(1)-C(24)-H(24A)	108.9(14)
C(25)-C(24)-H(24B)	108.5(14)
N(1)-C(24)-H(24B)	105.7(13)
H(24A)-C(24)-H(24B)	114(2)
C(24)-C(25)-H(25A)	108.7(14)
C(24)-C(25)-H(25B)	107.4(16)
H(25A)-C(25)-H(25B)	106(2)
C(24)-C(25)-H(25C)	107.8(19)
H(25A)-C(25)-H(25C)	110(2)
H(25B)-C(25)-H(25C)	116(2)

Symmetry transformations used to generate equivalent atoms:
#1 -x+2, -y+1, -z

Table E46. Anisotropic displacement parameters ($Å^2$ x 10^3) for **2A•$(C_6H_{15}N)_2$**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+...+2hka^*b^*U_{12}]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(1)	32(1)	39(1)	26(1)	1(1)	12(1)	-1(1)
O(1)	35(1)	84(1)	28(1)	16(1)	14(1)	4(1)
N(1)	58(1)	54(1)	39(1)	-6(1)	31(1)	-11(1)
C(2)	30(1)	36(1)	27(1)	0(1)	14(1)	-2(1)
O(2)	30(1)	62(1)	33(1)	10(1)	17(1)	3(1)
C(3)	27(1)	34(1)	26(1)	1(1)	11(1)	-1(1)
C(4)	29(1)	37(1)	24(1)	2(1)	11(1)	1(1)
C(5)	24(1)	38(1)	32(1)	-3(1)	14(1)	3(1)
C(6)	33(1)	46(1)	34(1)	-2(1)	8(1)	5(1)
C(7)	41(1)	63(2)	38(1)	-7(1)	2(1)	-1(1)
C(8)	42(1)	57(2)	51(2)	-16(1)	11(1)	-14(1)
C(9)	45(1)	44(1)	50(2)	-4(1)	25(1)	-7(1)
C(10)	34(1)	45(1)	31(1)	-2(1)	14(1)	-1(1)
C(11)	32(1)	42(1)	24(1)	1(1)	6(1)	7(1)
C(12)	47(1)	50(1)	43(1)	0(1)	24(1)	9(1)
C(13)	61(2)	68(2)	50(2)	4(1)	30(1)	23(1)
C(14)	76(2)	64(2)	38(1)	-7(1)	15(1)	32(2)
C(15)	71(2)	53(2)	53(2)	-18(1)	10(1)	5(1)
C(16)	50(1)	50(1)	45(1)	-10(1)	14(1)	-1(1)
C(20)	60(2)	79(2)	53(2)	-15(1)	27(1)	-13(2)
C(21)	74(2)	93(3)	40(2)	-8(1)	19(2)	6(2)
C(22)	73(2)	75(2)	45(2)	4(1)	30(2)	5(2)
C(23)	143(4)	70(2)	60(2)	-4(2)	44(3)	22(2)
C(24)	79(2)	56(2)	49(2)	-3(1)	36(2)	-15(1)
C(25)	91(2)	58(2)	61(2)	-14(1)	40(2)	-13(2)

Table E47. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for **2A•$(C_6H_{15}N)_2$**.

Atom	x	y	z	U_{eq}	Occ.
H(1)	9010(20)	4230(30)	1929(19)	81(8)	1
H(4A)	7773(15)	6364(17)	-1531(13)	31(5)	1
H(6A)	6428(16)	5620(20)	-2702(14)	45(6)	1
H(7A)	5170(20)	3810(20)	-3412(17)	65(7)	1
H(8A)	4980(20)	1830(20)	-2752(16)	61(7)	1
H(9A)	5978(17)	1540(20)	-1276(13)	43(6)	1
H(10A)	7210(15)	3318(16)	-526(13)	26(5)	1
H(12A)	5817(17)	5450(20)	-511(14)	46(6)	1
H(13A)	5060(20)	7030(20)	250(16)	66(7)	1
H(14A)	5832(19)	9300(20)	537(16)	63(7)	1
H(15A)	7390(20)	9860(30)	106(17)	71(8)	1
H(16A)	8140(20)	8310(20)	-631(15)	56(7)	1
H(20A)	9700(20)	5290(30)	3195(17)	76(8)	1
H(20B)	10290(20)	3700(30)	3167(17)	71(8)	1
H(21A)	10020(30)	4090(40)	4510(30)	126(13)	1
H(21B)	9350(20)	2790(30)	4011(18)	89(10)	1
H(21C)	8690(20)	4290(30)	4052(18)	85(9)	1
H(22A)	7410(20)	4500(20)	2784(18)	69(7)	1
H(22B)	7100(20)	4310(30)	1712(19)	84(9)	1
H(23A)	7120(30)	6580(40)	2070(30)	129(16)	1
H(23B)	8130(20)	6450(30)	1740(20)	92(10)	1
H(23C)	8360(30)	6550(30)	2800(20)	104(11)	1
H(24A)	9380(20)	1880(30)	2607(18)	89(9)	1
H(24B)	8002(19)	2210(20)	2687(16)	64(7)	1
H(25A)	7920(20)	840(30)	1487(16)	73(8)	1
H(25B)	8580(20)	2020(30)	1151(18)	78(8)	1
H(25C)	7220(30)	2370(40)	1200(20)	124(13)	1

Table E48. Crystal and structure refinement data for **2A•$C_6H_{18}N_4$•C_2H_6O**.

Identification code	**2A•$C_6H_{18}N_4$•C_2H_6O**
Empirical formula	$C_{40}H_{48}N_4O_5$
Formula weight	664.82 g/mole
Temperature	173(2) K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group	$P^{-}1$
Unit cell dimensions	a = 9.0999(18) Å b = 13.382(3) Å c = 16.800(3) Å α = 108.83(3)° β = 96.46(3)° γ = 109.49(3)°
Volume	1768.8(6) $Å^3$
Z	2
Density (calculated)	1.248 Mg/m^3
Absorption coefficient	0.083 mm^{-1}
F(000)	712
Crystal Size	0.90 mm x 0.74 mm x 0.26 mm
Color	wine red
Theta range for data collection	1.74° to 28.34°
Index ranges	$-11 \le h \le 11$, $-17 \le k \le 13$, $-21 \le l \le 19$
Reflections collected / Unique	10967 / 7951 [R_{int} = 0.0129]
Refinement method	Full-matrix-least-squares on F^2
Data / Restraints / Parameters	7951 / 0 / 639
Goodness-of-fit on F^2	1.051
Final R indices [$I>2\sigma(I)$]	R_1 = 0.0469, wR_2 = 0.1294
R indices (all data)	R_1 = 0.0636, wR_2 = 0.1383
Extinction Coefficient	0.004(2)
Largest diff. peak and hole	0.520 and -0.330 e/A^3

Table E49. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for **2A•$C_6H_{18}N_4$•C_2H_6O**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U_{eq}	Occ.
C(1)	9221(2)	5406(1)	4460(1)	30(1)	1
O(1)	8574(1)	5667(1)	3896(1)	43(1)	1
C(2)	9139(2)	4166(1)	4138(1)	25(1)	1
O(2)	8251(1)	3512(1)	3394(1)	31(1)	1
C(3)	10018(2)	3838(1)	4678(1)	28(1)	1
C(4)	9891(2)	2605(1)	4350(1)	27(1)	1
C(5)	11428(2)	2462(1)	4165(1)	29(1)	1
C(6)	12969(2)	3279(2)	4623(1)	41(1)	1
C(7)	14325(2)	3139(2)	4372(1)	48(1)	1
C(8)	14164(2)	2189(2)	3669(1)	45(1)	1
C(9)	12642(2)	1358(2)	3218(1)	42(1)	1
C(10)	11293(2)	1495(1)	3468(1)	34(1)	1
C(11)	9097(2)	1980(1)	4904(1)	28(1)	1
C(12)	9768(2)	1420(1)	5297(1)	37(1)	1
C(13)	8920(2)	804(2)	5744(1)	46(1)	1
C(14)	7394(2)	742(2)	5798(1)	46(1)	1
C(15)	6723(2)	1317(2)	5421(1)	44(1)	1
C(16)	7568(2)	1935(1)	4984(1)	36(1)	1
C(20)	4951(2)	4022(1)	188(1)	27(1)	1
O(20)	5088(1)	3230(1)	387(1)	36(1)	1
C(21)	6304(2)	5226(1)	693(1)	27(1)	1
O(21)	7298(1)	5300(1)	1326(1)	35(1)	1
C(22)	6354(2)	6132(1)	449(1)	26(1)	1
C(23)	7686(2)	7330(1)	887(1)	30(1)	1
C(24)	7336(2)	8058(1)	1692(1)	38(1)	1
C(25)	7134(2)	7722(2)	2390(1)	53(1)	1
C(26)	6741(2)	8363(3)	3102(2)	73(1)	1
C(27)	6555(2)	9351(3)	3130(2)	82(1)	1
C(28)	6758(3)	9701(2)	2457(2)	79(1)	1
C(29)	7149(2)	9053(2)	1726(2)	57(1)	1
C(30)	9380(2)	7336(1)	1012(1)	28(1)	1
C(31)	10597(2)	8089(1)	1753(1)	35(1)	1
C(32)	12163(2)	8146(2)	1804(1)	43(1)	1
C(33)	12540(2)	7451(2)	1122(1)	40(1)	1
C(34)	11330(2)	6683(2)	383(1)	40(1)	1
C(35)	9771(2)	6633(2)	328(1)	37(1)	1
N(40)	2327(2)	4498(1)	2332(1)	36(1)	1
C(40)	3740(2)	4445(2)	2029(1)	38(1)	1
C(41)	5134(2)	4621(2)	2734(1)	45(1)	1

Table E49. continued...

N(41)	6169(2)	4051(2)	2351(1)	40(1)	1
C(42)	1449(2)	3478(2)	2493(1)	51(1)	1
C(43)	118(2)	2597(2)	1713(2)	54(1)	1
N(43)	-1149(2)	3018(1)	1586(1)	47(1)	1
C(44A)	2524(8)	5514(7)	3045(5)	40(1)	0.561(6)
C(45A)	1746(6)	6164(4)	2696(3)	42(1)	0.561(6)
N(45A)	9(3)	5489(2)	2374(2)	36(1)	0.561(6)
C(44B)	2617(12)	5742(9)	2861(6)	42(2)	0.439(6)
C(45B)	1047(7)	5917(5)	2771(3)	38(1)	0.439(6)
N(45B)	234(3)	5432(3)	1842(3)	34(1)	0.439(6)
C(50)	3709(4)	522(3)	875(2)	94(1)	1
C(51)	2012(4)	253(4)	630(2)	120(1)	1
O(50A)	4826(10)	1634(11)	1094(10)	70(3)	0.55(6)
O(50B)	4370(50)	1782(17)	1210(10)	107(7)	0.45(6)

Table E50. Bond lengths (Å) and angles (°) for **2A•$C_6H_{18}N_4$•C_2H_6O**.

Atoms	Length	Angle
C(1)-O(1)	1.2617(17)	
C(1)-C(3)#1	1.400(2)	
C(1)-C(2)	1.5426(19)	
C(2)-O(2)	1.2559(18)	
C(2)-C(3)	1.4078(18)	
C(3)-C(1)#1	1.400(2)	
C(3)-C(4)	1.5203(19)	
C(4)-C(5)	1.5251(19)	
C(4)-C(11)	1.528(2)	
C(4)-H(4)	0.981(16)	
C(5)-C(6)	1.394(2)	
C(5)-C(10)	1.395(2)	
C(6)-C(7)	1.399(2)	
C(6)-H(6)	0.97(2)	
C(7)-C(8)	1.378(3)	
C(7)-H(7)	0.98(2)	
C(8)-C(9)	1.386(3)	
C(8)-H(8)	1.01(2)	
C(9)-C(10)	1.391(2)	
C(9)-H(9)	0.96(2)	
C(10)-H(10)	0.932(19)	
C(11)-C(12)	1.381(2)	
C(11)-C(16)	1.396(2)	
C(12)-C(13)	1.394(2)	
C(12)-H(12)	0.93(2)	
C(13)-C(14)	1.377(3)	
C(13)-H(13)	0.95(2)	
C(14)-C(15)	1.382(3)	
C(14)-H(14)	0.96(2)	
C(15)-C(16)	1.382(2)	
C(15)-H(15)	0.99(2)	
C(16)-H(16)	0.997(19)	
C(20)-O(20)	1.2483(17)	
C(20)-C(22)#2	1.4159(19)	
C(20)-C(21)	1.546(2)	
C(21)-O(21)	1.2705(17)	
C(21)-C(22)	1.388(2)	
C(22)-C(20)#2	1.4159(19)	
C(22)-C(23)	1.524(2)	
C(23)-C(24)	1.527(2)	
C(23)-C(30)	1.5288(19)	

Table E50. continued...

C(23)-H(23)	1.000(18)
C(24)-C(29)	1.382(3)
C(24)-C(25)	1.395(3)
C(25)-C(26)	1.391(3)
C(25)-H(25)	0.93(2)
C(26)-C(27)	1.374(4)
C(26)-H(26)	1.01(3)
C(27)-C(28)	1.365(5)
C(27)-H(27)	0.97(3)
C(28)-C(29)	1.415(3)
C(28)-H(28)	0.89(3)
C(29)-H(29)	0.98(2)
C(30)-C(31)	1.388(2)
C(30)-C(35)	1.397(2)
C(31)-C(32)	1.393(2)
C(31)-H(31)	0.98(2)
C(32)-C(33)	1.382(3)
C(32)-H(32)	0.96(2)
C(33)-C(34)	1.388(3)
C(33)-H(33)	0.994(19)
C(34)-C(35)	1.389(2)
C(34)-H(34)	0.96(2)
C(35)-H(35)	0.977(18)
N(40)-C(44A)	1.432(9)
N(40)-C(40)	1.4500(19)
N(40)-C(42)	1.461(2)
N(40)-C(44B)	1.525(11)
C(40)-C(41)	1.533(2)
C(40)-H(40A)	1.00(2)
C(40)-H(40B)	1.03(2)
C(41)-N(41)	1.477(2)
C(41)-H(41A)	1.04(2)
C(41)-H(41B)	1.01(2)
N(41)-H(41C)	0.96(2)
N(41)-H(41D)	0.92(3)
N(41)-H(41E)	0.93(2)
C(42)-C(43)	1.506(3)
C(42)-H(42A)	0.98(2)
C(42)-H(42B)	1.01(3)
C(43)-N(43)	1.465(2)
C(43)-H(43A)	0.95(3)
C(43)-H(43B)	0.98(3)
N(43)-H(43C)	0.98(3)
N(43)-H(43D)	0.98(3)

Table E50. continued...

C(44A)-C(45A)	1.500(8)
C(44A)-H(44A)	0.9700
C(44A)-H(44B)	0.9700
C(45A)-N(45A)	1.465(6)
C(45A)-H(45A)	0.9700
C(45A)-H(45B)	0.9700
N(45A)-H(45C)	0.8900
N(45A)-H(45D)	0.8900
N(45A)-H(45E)	0.8900
C(44B)-C(45B)	1.523(11)
C(44B)-H(44C)	0.9700
C(44B)-H(44D)	0.9700
C(45B)-N(45B)	1.473(6)
C(45B)-H(45F)	0.9700
C(45B)-H(45G)	0.9700
N(45B)-H(45H)	0.8900
N(45B)-H(45I)	0.8900
N(45B)-H(45J)	0.8900
C(50)-O(50A)	1.391(12)
C(50)-C(51)	1.440(5)
C(50)-O(50B)	1.47(2)
C(50)-H(50A)	0.9700
C(50)-H(50B)	0.9700
C(50)-H(50C)	0.9700
C(50)-H(50D)	0.9700
C(51)-H(51A)	0.9600
C(51)-H(51B)	0.9600
C(51)-H(51C)	0.9600
O(50A)-H(50)	0.88(4)
O(50B)-H(50)	0.97(4)

O(1)-C(1)-C(3)#1	124.13(13)
O(1)-C(1)-C(2)	115.26(13)
C(3)#1-C(1)-C(2)	120.60(12)
O(2)-C(2)-C(3)	124.77(13)
O(2)-C(2)-C(1)	115.57(12)
C(3)-C(2)-C(1)	119.66(12)
C(1)#1-C(3)-C(2)	119.19(12)
C(1)#1-C(3)-C(4)	121.55(12)
C(2)-C(3)-C(4)	118.55(12)
C(3)-C(4)-C(5)	114.73(12)
C(3)-C(4)-C(11)	111.08(12)
C(5)-C(4)-C(11)	115.26(11)
C(3)-C(4)-H(4)	105.2(9)

Table E50. continued...

C(5)-C(4)-H(4)	104.4(9)
C(11)-C(4)-H(4)	104.8(9)
C(6)-C(5)-C(10)	117.78(14)
C(6)-C(5)-C(4)	123.66(14)
C(10)-C(5)-C(4)	118.45(13)
C(5)-C(6)-C(7)	120.68(17)
C(5)-C(6)-H(6)	119.7(12)
C(7)-C(6)-H(6)	119.6(12)
C(8)-C(7)-C(6)	120.63(17)
C(8)-C(7)-H(7)	118.0(13)
C(6)-C(7)-H(7)	121.4(13)
C(7)-C(8)-C(9)	119.42(17)
C(7)-C(8)-H(8)	117.8(11)
C(9)-C(8)-H(8)	122.8(11)
C(8)-C(9)-C(10)	119.99(17)
C(8)-C(9)-H(9)	122.1(12)
C(10)-C(9)-H(9)	117.9(12)
C(9)-C(10)-C(5)	121.47(16)
C(9)-C(10)-H(10)	120.2(11)
C(5)-C(10)-H(10)	118.3(11)
C(12)-C(11)-C(16)	118.23(14)
C(12)-C(11)-C(4)	124.12(13)
C(16)-C(11)-C(4)	117.59(13)
C(11)-C(12)-C(13)	120.63(16)
C(11)-C(12)-H(12)	120.6(12)
C(13)-C(12)-H(12)	118.8(12)
C(14)-C(13)-C(12)	120.51(17)
C(14)-C(13)-H(13)	122.6(13)
C(12)-C(13)-H(13)	116.8(13)
C(13)-C(14)-C(15)	119.40(16)
C(13)-C(14)-H(14)	123.7(13)
C(15)-C(14)-H(14)	116.9(13)
C(14)-C(15)-C(16)	120.13(16)
C(14)-C(15)-H(15)	120.2(13)
C(16)-C(15)-H(15)	119.6(13)
C(15)-C(16)-C(11)	121.06(15)
C(15)-C(16)-H(16)	122.2(11)
C(11)-C(16)-H(16)	116.8(11)
O(20)-C(20)-C(22)#2	123.75(13)
O(20)-C(20)-C(21)	115.93(12)
C(22)#2-C(20)-C(21)	120.32(13)
O(21)-C(21)-C(22)	124.90(13)
O(21)-C(21)-C(20)	115.13(13)
C(22)-C(21)-C(20)	119.96(11)

Table E50. continued...

C(21)-C(22)-C(20)#2	119.22(13)
C(21)-C(22)-C(23)	123.20(12)
C(20)#2-C(22)-C(23)	117.38(13)
C(22)-C(23)-C(24)	111.63(12)
C(22)-C(23)-C(30)	113.82(12)
C(24)-C(23)-C(30)	115.23(12)
C(22)-C(23)-H(23)	104.7(10)
C(24)-C(23)-H(23)	104.6(10)
C(30)-C(23)-H(23)	105.7(10)
C(29)-C(24)-C(25)	118.34(18)
C(29)-C(24)-C(23)	119.96(17)
C(25)-C(24)-C(23)	121.65(16)
C(26)-C(25)-C(24)	121.1(2)
C(26)-C(25)-H(25)	119.2(16)
C(24)-C(25)-H(25)	119.7(15)
C(27)-C(26)-C(25)	120.1(3)
C(27)-C(26)-H(26)	120.5(17)
C(25)-C(26)-H(26)	119.4(17)
C(28)-C(27)-C(26)	119.8(2)
C(28)-C(27)-H(27)	115.4(17)
C(26)-C(27)-H(27)	124.7(17)
C(27)-C(28)-C(29)	120.7(3)
C(27)-C(28)-H(28)	124.1(18)
C(29)-C(28)-H(28)	115.3(18)
C(24)-C(29)-C(28)	119.9(3)
C(24)-C(29)-H(29)	119.4(15)
C(28)-C(29)-H(29)	120.6(15)
C(31)-C(30)-C(35)	118.04(13)
C(31)-C(30)-C(23)	122.56(13)
C(35)-C(30)-C(23)	119.14(13)
C(30)-C(31)-C(32)	120.67(15)
C(30)-C(31)-H(31)	121.8(11)
C(32)-C(31)-H(31)	117.5(11)
C(33)-C(32)-C(31)	120.85(15)
C(33)-C(32)-H(32)	119.9(13)
C(31)-C(32)-H(32)	119.2(13)
C(32)-C(33)-C(34)	119.04(14)
C(32)-C(33)-H(33)	119.1(11)
C(34)-C(33)-H(33)	121.8(11)
C(33)-C(34)-C(35)	120.13(15)
C(33)-C(34)-H(34)	119.0(11)
C(35)-C(34)-H(34)	120.9(11)
C(34)-C(35)-C(30)	121.26(15)
C(34)-C(35)-H(35)	120.0(10)

Table E50. continued...

C(30)-C(35)-H(35)	118.7(10)
C(44A)-N(40)-C(40)	117.8(3)
C(44A)-N(40)-C(42)	110.1(3)
C(40)-N(40)-C(42)	112.30(14)
C(44A)-N(40)-C(44B)	19.0(3)
C(40)-N(40)-C(44B)	110.1(4)
C(42)-N(40)-C(44B)	127.7(3)
N(40)-C(40)-C(41)	114.07(13)
N(40)-C(40)-H(40A)	109.4(11)
C(41)-C(40)-H(40A)	109.1(11)
N(40)-C(40)-H(40B)	108.8(11)
C(41)-C(40)-H(40B)	107.9(11)
H(40A)-C(40)-H(40B)	107.3(15)
N(41)-C(41)-C(40)	110.96(15)
N(41)-C(41)-H(41A)	109.1(12)
C(40)-C(41)-H(41A)	108.2(12)
N(41)-C(41)-H(41B)	109.6(12)
C(40)-C(41)-H(41B)	109.5(12)
H(41A)-C(41)-H(41B)	109.4(17)
C(41)-N(41)-H(41C)	109.7(12)
C(41)-N(41)-H(41D)	110.2(15)
H(41C)-N(41)-H(41D)	105.6(19)
C(41)-N(41)-H(41E)	105.8(14)
H(41C)-N(41)-H(41E)	111.3(18)
H(41D)-N(41)-H(41E)	114(2)
N(40)-C(42)-C(43)	111.35(15)
N(40)-C(42)-H(42A)	106.5(13)
C(43)-C(42)-H(42A)	111.6(13)
N(40)-C(42)-H(42B)	109.3(15)
C(43)-C(42)-H(42B)	112.9(16)
H(42A)-C(42)-H(42B)	104.9(19)
N(43)-C(43)-C(42)	109.70(18)
N(43)-C(43)-H(43A)	104.9(15)
C(42)-C(43)-H(43A)	108.6(15)
N(43)-C(43)-H(43B)	111.5(14)
C(42)-C(43)-H(43B)	110.1(15)
H(43A)-C(43)-H(43B)	112(2)
C(43)-N(43)-H(43C)	107.6(15)
C(43)-N(43)-H(43D)	111.3(14)
H(43C)-N(43)-H(43D)	104(2)
N(40)-C(44A)-C(45A)	107.6(5)
N(40)-C(44A)-H(44A)	110.2
C(45A)-C(44A)-H(44A)	110.2
N(40)-C(44A)-H(44B)	110.2

Table E50. continued...

C(45A)-C(44A)-H(44B)	110.2
H(44A)-C(44A)-H(44B)	108.5
N(45A)-C(45A)-C(44A)	109.4(4)
N(45A)-C(45A)-H(45A)	109.8
C(44A)-C(45A)-H(45A)	109.8
N(45A)-C(45A)-H(45B)	109.8
C(44A)-C(45A)-H(45B)	109.8
H(45A)-C(45A)-H(45B)	108.2
C(45A)-N(45A)-H(45C)	109.5
C(45A)-N(45A)-H(45D)	109.5
H(45C)-N(45A)-H(45D)	109.5
C(45A)-N(45A)-H(45E)	109.5
H(45C)-N(45A)-H(45E)	109.5
H(45D)-N(45A)-H(45E)	109.5
C(45B)-C(44B)-N(40)	110.5(7)
C(45B)-C(44B)-H(44C)	109.6
N(40)-C(44B)-H(44C)	109.6
C(45B)-C(44B)-H(44D)	109.6
N(40)-C(44B)-H(44D)	109.6
H(44C)-C(44B)-H(44D)	108.1
N(45B)-C(45B)-C(44B)	108.5(5)
N(45B)-C(45B)-H(45F)	110.0
C(44B)-C(45B)-H(45F)	110.0
N(45B)-C(45B)-H(45G)	110.0
C(44B)-C(45B)-H(45G)	110.0
H(45F)-C(45B)-H(45G)	108.4
C(45B)-N(45B)-H(45H)	109.5
C(45B)-N(45B)-H(45I)	109.5
H(45H)-N(45B)-H(45I)	109.5
C(45B)-N(45B)-H(45J)	109.5
H(45H)-N(45B)-H(45J)	109.5
H(45I)-N(45B)-H(45J)	109.5
O(50A)-C(50)-C(51)	120.5(4)
O(50A)-C(50)-O(50B)	21.9(18)
C(51)-C(50)-O(50B)	101(2)
O(50A)-C(50)-H(50A)	107.2
C(51)-C(50)-H(50A)	107.2
O(50B)-C(50)-H(50A)	107.0
O(50A)-C(50)-H(50B)	107.2
C(51)-C(50)-H(50B)	107.2
O(50B)-C(50)-H(50B)	126.2
H(50A)-C(50)-H(50B)	106.8
O(50A)-C(50)-H(50C)	93.3
C(51)-C(50)-H(50C)	111.6

Table E50. continued...

O(50B)-C(50)-H(50C)	111.5
H(50A)-C(50)-H(50C)	117.1
H(50B)-C(50)-H(50C)	14.8
O(50A)-C(50)-H(50D)	109.0
C(51)-C(50)-H(50D)	111.6
O(50B)-C(50)-H(50D)	111.5
H(50A)-C(50)-H(50D)	7.7
H(50B)-C(50)-H(50D)	99.2
H(50C)-C(50)-H(50D)	109.4
C(50)-C(51)-H(51A)	109.5
C(50)-C(51)-H(51B)	109.5
H(51A)-C(51)-H(51B)	109.5
C(50)-C(51)-H(51C)	109.5
H(51A)-C(51)-H(51C)	109.5
H(51B)-C(51)-H(51C)	109.5
C(50)-O(50A)-H(50)	109(2)
C(50)-O(50B)-H(50)	98(3)

Symmetry transformations used to generate equivalent atoms:

#1 -x+2, -y+1, -z+1

#2 -x+1, -y+1, -z

Table E51. Anisotropic displacement parameters ($Å^2 \times 10^3$) for **2A•$C_6H_{18}N_4$•C_2H_6O**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+...+2hka^*b^*U_{12}]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(1)	28(1)	29(1)	31(1)	12(1)	-1(1)	12(1)
O(1)	53(1)	35(1)	35(1)	10(1)	-14(1)	21(1)
C(2)	23(1)	29(1)	25(1)	12(1)	4(1)	11(1)
O(2)	32(1)	33(1)	25(1)	8(1)	-1(1)	15(1)
C(3)	27(1)	26(1)	29(1)	10(1)	2(1)	12(1)
C(4)	28(1)	28(1)	26(1)	10(1)	1(1)	12(1)
C(5)	31(1)	32(1)	31(1)	18(1)	7(1)	16(1)
C(6)	34(1)	44(1)	42(1)	14(1)	3(1)	17(1)
C(7)	31(1)	59(1)	55(1)	25(1)	4(1)	17(1)
C(8)	41(1)	63(1)	52(1)	32(1)	18(1)	31(1)
C(9)	49(1)	48(1)	42(1)	21(1)	18(1)	28(1)
C(10)	36(1)	34(1)	37(1)	18(1)	10(1)	16(1)
C(11)	31(1)	24(1)	26(1)	7(1)	4(1)	11(1)
C(12)	41(1)	38(1)	37(1)	18(1)	10(1)	20(1)
C(13)	65(1)	43(1)	41(1)	24(1)	17(1)	26(1)
C(14)	59(1)	39(1)	37(1)	14(1)	21(1)	12(1)
C(15)	38(1)	44(1)	41(1)	9(1)	15(1)	12(1)
C(16)	34(1)	37(1)	36(1)	11(1)	8(1)	15(1)
C(20)	24(1)	39(1)	20(1)	13(1)	8(1)	14(1)
O(20)	35(1)	43(1)	33(1)	18(1)	5(1)	17(1)
C(21)	19(1)	42(1)	19(1)	12(1)	4(1)	11(1)
O(21)	23(1)	52(1)	28(1)	21(1)	-1(1)	10(1)
C(22)	20(1)	36(1)	20(1)	9(1)	4(1)	10(1)
C(23)	23(1)	35(1)	27(1)	9(1)	4(1)	10(1)
C(24)	18(1)	44(1)	35(1)	-2(1)	1(1)	9(1)
C(25)	38(1)	69(1)	36(1)	4(1)	12(1)	16(1)
C(26)	40(1)	96(2)	44(1)	-10(1)	14(1)	12(1)
C(27)	31(1)	86(2)	68(2)	-31(1)	10(1)	9(1)
C(28)	37(1)	54(1)	103(2)	-21(1)	2(1)	20(1)
C(29)	33(1)	50(1)	66(1)	-3(1)	0(1)	17(1)
C(30)	22(1)	34(1)	27(1)	13(1)	7(1)	9(1)
C(31)	25(1)	41(1)	31(1)	8(1)	6(1)	9(1)
C(32)	24(1)	52(1)	37(1)	8(1)	2(1)	8(1)
C(33)	24(1)	54(1)	42(1)	20(1)	12(1)	14(1)
C(34)	33(1)	52(1)	36(1)	14(1)	15(1)	19(1)
C(35)	29(1)	46(1)	28(1)	8(1)	5(1)	12(1)
N(40)	27(1)	50(1)	36(1)	17(1)	11(1)	17(1)
C(40)	26(1)	64(1)	34(1)	27(1)	10(1)	19(1)
C(41)	30(1)	76(1)	34(1)	27(1)	9(1)	23(1)

Table E51. continued...

N(41)	25(1)	66(1)	37(1)	31(1)	6(1)	19(1)
C(42)	36(1)	81(1)	56(1)	48(1)	19(1)	24(1)
C(43)	44(1)	48(1)	78(2)	33(1)	18(1)	18(1)
N(43)	34(1)	57(1)	45(1)	20(1)	6(1)	13(1)
C(44A)	23(2)	47(3)	43(3)	13(2)	-3(2)	13(2)
C(45A)	34(2)	38(2)	46(2)	13(2)	2(2)	11(2)
N(45A)	36(2)	47(2)	30(2)	14(1)	4(1)	24(1)
C(44B)	42(3)	48(4)	36(4)	15(2)	4(2)	20(3)
C(45B)	25(3)	47(3)	36(2)	10(2)	3(2)	16(2)
N(45B)	30(2)	45(2)	33(2)	20(1)	9(1)	19(1)
C(50)	98(2)	90(2)	127(3)	69(2)	51(2)	43(2)
C(51)	103(3)	153(3)	105(3)	79(3)	14(2)	28(2)
O(50A)	60(4)	66(3)	81(5)	47(4)	-3(2)	12(2)
O(50B)	157(15)	65(5)	58(4)	35(3)	5(7)	-8(7)

Table E52. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for **2A•$C_6H_{18}N_4$•C_2H_6O**.

Atom	x	y	z	U_{eq}	Occ.
H(4)	9122(19)	2222(13)	3780(10)	25(4)	1
H(6)	13100(20)	3961(18)	5108(13)	46(5)	1
H(7)	15420(30)	3709(19)	4690(14)	60(6)	1
H(8)	15170(20)	2142(16)	3498(12)	47(5)	1
H(9)	12480(20)	697(17)	2714(13)	45(5)	1
H(10)	10270(20)	933(16)	3171(12)	42(5)	1
H(12)	10780(30)	1424(18)	5252(13)	50(5)	1
H(13)	9460(30)	471(19)	6027(14)	55(6)	1
H(14)	6760(30)	323(19)	6093(14)	62(6)	1
H(15)	5620(30)	1275(19)	5452(14)	63(6)	1
H(16)	7110(20)	2354(16)	4700(12)	42(5)	1
H(23)	7610(20)	7706(15)	466(11)	35(4)	1
H(25)	7260(30)	7060(20)	2381(16)	69(7)	1
H(26)	6590(30)	8090(20)	3600(20)	93(9)	1
H(27)	6320(30)	9860(30)	3620(20)	96(9)	1
H(28)	6680(30)	10340(20)	2442(17)	79(8)	1
H(29)	7310(30)	9310(20)	1244(16)	68(7)	1
H(31)	10390(20)	8591(17)	2257(13)	44(5)	1
H(32)	12990(30)	8688(19)	2321(15)	60(6)	1
H(33)	13670(20)	7507(16)	1178(12)	45(5)	1
H(34)	11590(20)	6198(16)	-88(13)	43(5)	1
H(35)	8920(20)	6105(16)	-199(12)	38(4)	1
H(40A)	4120(20)	5032(17)	1772(13)	46(5)	1
H(40B)	3430(20)	3655(17)	1545(13)	46(5)	1
H(41A)	5810(30)	5500(20)	3051(14)	58(6)	1
H(41B)	4690(20)	4299(17)	3163(13)	52(5)	1
H(41C)	6580(20)	4349(17)	1939(13)	50(5)	1
H(41D)	5570(30)	3280(20)	2041(16)	68(7)	1
H(41E)	6990(30)	4211(19)	2812(15)	60(6)	1
H(42A)	1030(30)	3739(18)	2997(15)	59(6)	1
H(42B)	2230(30)	3170(20)	2687(17)	84(8)	1
H(43A)	530(30)	2530(20)	1207(16)	70(7)	1
H(43B)	-300(30)	1860(20)	1783(16)	75(7)	1
H(43C)	-1810(30)	2890(20)	1993(17)	79(8)	1
H(43D)	-1890(30)	2560(20)	1013(17)	74(7)	1
H(44A)	3658	5982	3311	62(11)	0.561(6)
H(44B)	2020	5310	3481	45(9)	0.561(6)
H(45A)	1954	6897	3150	61(11)	0.561(6)
H(45B)	2195	6311	2228	44(9)	0.561(6)
H(45C)	-172	4765	2055	51(10)	0.561(6)

Table E52. continued...

H(45D)	-419	5782	2051	190(40)	0.561(6)
H(45E)	-436	5511	2822	48(10)	0.561(6)
H(44C)	3076	5934	3467	40(12)	0.439(6)
H(44D)	3379	6249	2660	32(10)	0.439(6)
H(45F)	364	5537	3077	52(13)	0.439(6)
H(45G)	1264	6730	3019	24(9)	0.439(6)
H(45H)	-121	4669	1641	67(16)	0.439(6)
H(45I)	925	5697	1551	43(11)	0.439(6)
H(45J)	-593	5636	1772	34(11)	0.439(6)
H(50)	4610(40)	1880(30)	690(20)	95(10)	1
H(50A)	3893	309	1366	113	0.55(6)
H(50B)	3962	21	399	113	0.55(6)
H(50C)	4101	204	379	113	0.45(6)
H(50D)	3961	246	1319	113	0.45(6)
H(51A)	1423	-558	455	180	1
H(51B)	1791	478	157	180	1
H(51C)	1691	661	1116	180	1

Table E53. Torsion angles (°) for **2A•$C_6H_{18}N_4$•C_2H_6O**.

Atoms	Angle
O(1) C(1) C(2) O(2)	-9.60(19)
C(3)#1 C(1) C(2) O(2)	170.84(13)
O(1) C(1) C(2) C(3)	170.96(13)
C(3)#1 C(1) C(2) C(3)	-8.6(2)
O(2) C(2) C(3) C(1)#1	-170.91(13)
C(1) C(2) C(3) C(1)#1	8.5(2)
O(2) C(2) C(3) C(4)	-0.5(2)
C(1) C(2) C(3) C(4)	178.93(12)
C(1)#1 C(3) C(4) C(5)	-77.71(17)
C(2) C(3) C(4) C(5)	112.07(14)
C(1)#1 C(3) C(4) C(11)	55.21(17)
C(2) C(3) C(4) C(11)	-115.01(14)
C(3) C(4) C(5) C(6)	31.67(19)
C(11) C(4) C(5) C(6)	-99.26(17)
C(3) C(4) C(5) C(10)	-144.48(13)
C(11) C(4) C(5) C(10)	84.59(16)
C(10) C(5) C(6) C(7)	1.7(2)
C(4) C(5) C(6) C(7)	-174.51(15)
C(5) C(6) C(7) C(8)	-0.3(3)
C(6) C(7) C(8) C(9)	-1.0(3)
C(7) C(8) C(9) C(10)	0.9(3)
C(8) C(9) C(10) C(5)	0.6(2)
C(6) C(5) C(10) C(9)	-1.8(2)
C(4) C(5) C(10) C(9)	174.55(13)
C(3) C(4) C(11) C(12)	-125.95(15)
C(5) C(4) C(11) C(12)	6.7(2)
C(3) C(4) C(11) C(16)	56.84(17)
C(5) C(4) C(11) C(16)	-170.51(13)
C(16) C(11) C(12) C(13)	1.5(2)
C(4) C(11) C(12) C(13)	-175.71(15)
C(11) C(12) C(13) C(14)	0.2(3)
C(12) C(13) C(14) C(15)	-1.5(3)
C(13) C(14) C(15) C(16)	1.0(3)
C(14) C(15) C(16) C(11)	0.8(3)
C(12) C(11) C(16) C(15)	-2.0(2)
C(4) C(11) C(16) C(15)	175.37(14)
O(20) C(20) C(21) O(21)	-8.28(17)
C(22)#2 C(20) C(21) O(21)	171.03(12)
O(20) C(20) C(21) C(22)	172.53(12)
C(22)#2 C(20) C(21) C(22)	-8.2(2)
O(21) C(21) C(22) C(20)#2	-171.03(13)

Table E53. continued...

C(20) C(21) C(22) C(20)#2	8.1(2)
O(21) C(21) C(22) C(23)	3.6(2)
C(20) C(21) C(22) C(23)	-177.29(12)
C(21) C(22) C(23) C(24)	-85.65(16)
C(20)#2 C(22) C(23) C(24)	89.07(15)
C(21) C(22) C(23) C(30)	46.90(18)
C(20)#2 C(22) C(23) C(30)	-138.38(13)
C(22) C(23) C(24) C(29)	-118.00(16)
C(30) C(23) C(24) C(29)	110.16(16)
C(22) C(23) C(24) C(25)	59.42(18)
C(30) C(23) C(24) C(25)	-72.41(19)
C(29) C(24) C(25) C(26)	0.5(3)
C(23) C(24) C(25) C(26)	-176.94(15)
C(24) C(25) C(26) C(27)	-0.5(3)
C(25) C(26) C(27) C(28)	0.0(3)
C(26) C(27) C(28) C(29)	0.4(3)
C(25) C(24) C(29) C(28)	-0.1(2)
C(23) C(24) C(29) C(28)	177.36(15)
C(27) C(28) C(29) C(24)	-0.3(3)
C(22) C(23) C(30) C(31)	-141.10(14)
C(24) C(23) C(30) C(31)	-10.3(2)
C(22) C(23) C(30) C(35)	44.80(18)
C(24) C(23) C(30) C(35)	175.59(14)
C(35) C(30) C(31) C(32)	0.7(2)
C(23) C(30) C(31) C(32)	-173.43(15)
C(30) C(31) C(32) C(33)	-0.4(3)
C(31) C(32) C(33) C(34)	-0.5(3)
C(32) C(33) C(34) C(35)	1.1(3)
C(33) C(34) C(35) C(30)	-0.8(3)
C(31) C(30) C(35) C(34)	-0.1(2)
C(23) C(30) C(35) C(34)	174.25(15)
C(44A) N(40) C(40) C(41)	56.2(3)
C(42) N(40) C(40) C(41)	-73.4(2)
C(44B) N(40) C(40) C(41)	75.1(4)
N(40) C(40) C(41) N(41)	153.69(16)
C(44A) N(40) C(42) C(43)	132.7(3)
C(40) N(40) C(42) C(43)	-93.97(18)
C(44B) N(40) C(42) C(43)	124.3(5)
N(40) C(42) C(43) N(43)	-69.1(2)
C(40) N(40) C(44A) C(45A)	108.5(4)
C(42) N(40) C(44A) C(45A)	-121.0(4)
C(44B) N(40) C(44A) C(45A)	38.4(17)
N(40) C(44A) C(45A) N(45A)	65.3(6)
C(44A) N(40) C(44B) C(45B)	-93(2)

Table E53. continued...

C(40) N(40) C(44B) C(45B)	149.3(4)
C(42) N(40) C(44B) C(45B)	-68.3(6)
N(40) C(44B) C(45B) N(45B)	-49.4(7)

Symmetry transformations used to generate equivalent atoms:

#1 -x+2, -y+1, -z+1

#2 -x+1, -y+1, -z

Table E54. Hydrogen bonds, lengths (Å) and angles (°) for **2A•$C_6H_{18}N_4$•C_2H_6O**.

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(41)-H(41C)...O(21)	0.96(2)	1.88(2)	2.8162(19)	163.7(18)
N(41)-H(41C)...O(20)	0.96(2)	2.49(2)	3.046(2)	116.7(15)
N(41)-H(41D)...O(50B)	0.92(3)	1.89(3)	2.792(16)	168(2)
N(41)-H(41D)...O(50A)	0.92(3)	2.08(3)	2.964(15)	160(2)
N(41)-H(41E)...O(2)	0.93(2)	2.05(2)	2.8549(18)	144.6(19)
N(41)-H(41E)...O(1)	0.93(2)	2.11(2)	2.854(3)	136.9(19)
N(43)-H(43C)...O(2)#3	0.98(3)	2.21(3)	3.045(2)	142(2)
N(45A)-H(45C)...N(43)	0.89	2.03	2.886(3)	159.8
N(45A)-H(45D)...O(21)#3	0.89	2.06	2.752(3)	133.7
N(45A)-H(45E)...O(1)#3	0.89	2.09	2.978(4)	176.6
N(45B)-H(45H)...N(43)	0.89	2.06	2.910(4)	160.0
N(45B)-H(45J)...O(21)#3	0.89	1.83	2.643(3)	151.8

Symmetry transformations used to generate equivalent atoms:
#3 x-1, y, z

Table E55. Crystal and structure refinement data for **2A•$(C_6H_{15}NO_3)_2$**.

Identification code	**2A•$(C_6H_{15}NO_3)_2$**
Empirical formula	$C_{44}H_{54}N_4O_{10}$
Formula weight	770.89 g/mole
Temperature	173(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	$P2_1/c$
Unit cell dimensions	a = 12.525(3) Å b = 10.053(2) Å c = 16.359(3) Å α = 90.00° β = 108.90(3)° γ = 90.00°
Volume	1948.8(7) $Å^3$
Z	2
Density (calculated)	1.314 Mg/m^3
Absorption coefficient	0.093 mm^{-1}
F(000)	824
Crystal Size	0.86 mm x 0.74 mm x 0.16 mm
Color	wine red
Theta range for data collection	1.72° to 28.36°
Index ranges	$-16 \le h \le 16$, $-13 \le k \le 12$, $-21 \le l \le 21$
Reflections collected / Unique	22758 / 4741 [R_{int} = 0.0213]
Refinement method	Full-matrix-least-squares on F^2
Data / Restraints / Parameters	4741 / 0 / 362
Goodness-of-fit on F^2	1.037
Final R indices [I>2σ(I)]	R_1 = 0.0361, wR_2 = 0.0909
R indices (all data)	R_1 = 0.0483, wR_2 = 0.0972
Extinction Coefficient	0.0050(11)
Largest diff. peak and hole	0.313 and -0.180 e/A^3

Table E56. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for **2A•$(C_6H_{15}NO_3)_2$**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U_{eq}	Occ.
O(1)	10418(1)	3966(1)	1576(1)	33(1)	1
C(1)	10284(1)	4423(1)	841(1)	20(1)	1
O(2)	8318(1)	4460(1)	650(1)	25(1)	1
C(2)	9058(1)	4776(1)	302(1)	20(1)	1
C(3)	8818(1)	5379(1)	-501(1)	20(1)	1
C(4)	7637(1)	5825(1)	-1044(1)	20(1)	1
C(5)	6881(1)	4718(1)	-1552(1)	22(1)	1
C(6)	6277(1)	4939(1)	-2422(1)	31(1)	1
C(7)	5558(1)	3972(2)	-2913(1)	40(1)	1
C(8)	5433(1)	2771(2)	-2549(1)	38(1)	1
C(9)	6035(1)	2526(1)	-1690(1)	32(1)	1
C(10)	6756(1)	3494(1)	-1195(1)	26(1)	1
C(11)	7055(1)	6684(1)	-537(1)	22(1)	1
C(12)	7326(1)	8030(1)	-435(1)	28(1)	1
C(13)	6823(1)	8866(1)	10(1)	34(1)	1
C(14)	6033(1)	8376(1)	357(1)	36(1)	1
C(15)	5750(1)	7045(2)	257(1)	39(1)	1
C(16)	6255(1)	6207(1)	-187(1)	33(1)	1
N(20)	8854(1)	3657(1)	2491(1)	25(1)	1
C(20)	8620(1)	2180(1)	2406(1)	31(1)	1
C(21)	7985(1)	1700(1)	1504(1)	36(1)	1
O(21)	8632(1)	1758(1)	939(1)	43(1)	1
C(22)	7792(1)	4453(1)	2368(1)	30(1)	1
C(23)	7976(1)	5941(1)	2429(1)	35(1)	1
O(23)	8555(1)	6427(1)	1877(1)	38(1)	1
C(24)	9790(1)	3948(1)	3324(1)	29(1)	1
C(25)	9583(1)	3486(1)	4139(1)	32(1)	1
O(25)	8721(1)	4253(1)	4303(1)	36(1)	1

Table E57. Bond lengths (Å) and angles (°) for **2A•($C_6H_{15}NO_3$)$_2$**.

Atoms	Length	Angle
O(1)-C(1)	1.2471(13)	
C(1)-C(3)#1	1.4216(14)	
C(1)-C(2)	1.5441(15)	
O(2)-C(2)	1.2738(13)	
C(2)-C(3)	1.3889(14)	
C(3)-C(1)#1	1.4215(14)	
C(3)-C(4)	1.5250(15)	
C(4)-C(5)	1.5223(15)	
C(4)-C(11)	1.5348(15)	
C(4)-H(4)	0.989(13)	
C(5)-C(10)	1.3929(17)	
C(5)-C(6)	1.3956(16)	
C(6)-C(7)	1.3899(18)	
C(6)-H(6)	0.974(15)	
C(7)-C(8)	1.378(2)	
C(7)-H(7)	0.962(18)	
C(8)-C(9)	1.385(2)	
C(8)-H(8)	0.965(18)	
C(9)-C(10)	1.3934(17)	
C(9)-H(9)	0.977(16)	
C(10)-H(10)	0.960(15)	
C(11)-C(16)	1.3909(16)	
C(11)-C(12)	1.3915(16)	
C(12)-C(13)	1.3905(18)	
C(12)-H(12)	0.948(15)	
C(13)-C(14)	1.382(2)	
C(13)-H(13)	0.952(17)	
C(14)-C(15)	1.381(2)	
C(14)-H(14)	0.950(18)	
C(15)-C(16)	1.3911(18)	
C(15)-H(15)	0.956(18)	
C(16)-H(16)	0.988(16)	
N(20)-C(22)	1.5093(16)	
N(20)-C(24)	1.5110(16)	
N(20)-C(20)	1.5118(15)	
N(20)-H(20)	0.971(15)	
C(20)-C(21)	1.5111(18)	
C(20)-H(20A)	0.991(16)	
C(20)-H(20B)	0.990(16)	
C(21)-O(21)	1.4151(16)	
C(21)-H(21A)	0.989(17)	

Table E57. continued...

C(21)-H(21B)	1.002(17)
O(21)-H(21)	0.83(3)
C(22)-C(23)	1.5117(19)
C(22)-H(22A)	0.962(15)
C(22)-H(22B)	0.997(15)
C(23)-O(23)	1.4157(16)
C(23)-H(23A)	0.991(17)
C(23)-H(23B)	0.996(15)
O(23)-H(23)	0.88(2)
C(24)-C(25)	1.5108(17)
C(24)-H(24A)	0.966(15)
C(24)-H(24B)	0.959(15)
C(25)-O(25)	1.4210(16)
C(25)-H(25A)	0.980(16)
C(25)-H(25B)	0.992(16)
O(25)-H(25)	0.91(2)

O(1)-C(1)-C(3)#1	123.52(10)
O(1)-C(1)-C(2)	115.70(9)
C(3)#1-C(1)-C(2)	120.79(9)
O(2)-C(2)-C(3)	124.43(10)
O(2)-C(2)-C(1)	114.86(9)
C(3)-C(2)-C(1)	120.71(9)
C(2)-C(3)-C(1)#1	118.36(9)
C(2)-C(3)-C(4)	123.42(9)
C(1)#1-C(3)-C(4)	118.21(9)
C(5)-C(4)-C(3)	114.66(9)
C(5)-C(4)-C(11)	112.73(8)
C(3)-C(4)-C(11)	113.08(8)
C(5)-C(4)-H(4)	105.6(7)
C(3)-C(4)-H(4)	104.4(7)
C(11)-C(4)-H(4)	105.2(7)
C(10)-C(5)-C(6)	118.10(11)
C(10)-C(5)-C(4)	123.46(10)
C(6)-C(5)-C(4)	118.45(11)
C(7)-C(6)-C(5)	120.91(12)
C(7)-C(6)-H(6)	120.4(9)
C(5)-C(6)-H(6)	118.7(9)
C(8)-C(7)-C(6)	120.37(12)
C(8)-C(7)-H(7)	120.8(10)
C(6)-C(7)-H(7)	118.8(10)
C(7)-C(8)-C(9)	119.58(12)
C(7)-C(8)-H(8)	121.3(10)
C(9)-C(8)-H(8)	119.1(10)

Table E57. continued...

C(8)-C(9)-C(10)	120.20(13)
C(8)-C(9)-H(9)	120.8(9)
C(10)-C(9)-H(9)	119.0(9)
C(5)-C(10)-C(9)	120.83(11)
C(5)-C(10)-H(10)	119.5(9)
C(9)-C(10)-H(10)	119.6(9)
C(16)-C(11)-C(12)	117.84(11)
C(16)-C(11)-C(4)	124.03(10)
C(12)-C(11)-C(4)	118.11(10)
C(13)-C(12)-C(11)	121.07(12)
C(13)-C(12)-H(12)	120.1(9)
C(11)-C(12)-H(12)	118.8(9)
C(14)-C(13)-C(12)	120.42(12)
C(14)-C(13)-H(13)	120.2(10)
C(12)-C(13)-H(13)	119.4(10)
C(15)-C(14)-C(13)	119.17(12)
C(15)-C(14)-H(14)	120.7(11)
C(13)-C(14)-H(14)	120.1(11)
C(14)-C(15)-C(16)	120.44(13)
C(14)-C(15)-H(15)	119.7(11)
C(16)-C(15)-H(15)	119.8(11)
C(11)-C(16)-C(15)	121.06(12)
C(11)-C(16)-H(16)	121.1(9)
C(15)-C(16)-H(16)	117.8(9)
C(22)-N(20)-C(24)	114.20(9)
C(22)-N(20)-C(20)	111.85(10)
C(24)-N(20)-C(20)	110.10(9)
C(22)-N(20)-H(20)	106.7(8)
C(24)-N(20)-H(20)	105.1(8)
C(20)-N(20)-H(20)	108.5(9)
C(21)-C(20)-N(20)	115.32(11)
C(21)-C(20)-H(20A)	108.3(9)
N(20)-C(20)-H(20A)	106.0(9)
C(21)-C(20)-H(20B)	108.7(9)
N(20)-C(20)-H(20B)	106.1(9)
H(20A)-C(20)-H(20B)	112.6(12)
O(21)-C(21)-C(20)	113.13(11)
O(21)-C(21)-H(21A)	104.8(10)
C(20)-C(21)-H(21A)	107.1(9)
O(21)-C(21)-H(21B)	112.0(10)
C(20)-C(21)-H(21B)	111.4(10)
H(21A)-C(21)-H(21B)	107.9(14)
C(21)-O(21)-H(21)	98.6(16)
N(20)-C(22)-C(23)	113.99(10)

Table E57. continued...

N(20)-C(22)-H(22A)	108.6(9)
C(23)-C(22)-H(22A)	108.8(9)
N(20)-C(22)-H(22B)	106.0(9)
C(23)-C(22)-H(22B)	110.3(9)
H(22A)-C(22)-H(22B)	109.1(12)
O(23)-C(23)-C(22)	113.46(11)
O(23)-C(23)-H(23A)	111.4(10)
C(22)-C(23)-H(23A)	106.9(10)
O(23)-C(23)-H(23B)	105.0(8)
C(22)-C(23)-H(23B)	112.4(9)
H(23A)-C(23)-H(23B)	107.8(13)
C(23)-O(23)-H(23)	108.8(14)
C(25)-C(24)-N(20)	115.63(10)
C(25)-C(24)-H(24A)	109.4(9)
N(20)-C(24)-H(24A)	107.0(8)
C(25)-C(24)-H(24B)	108.5(8)
N(20)-C(24)-H(24B)	106.3(8)
H(24A)-C(24)-H(24B)	109.9(12)
O(25)-C(25)-C(24)	110.91(10)
O(25)-C(25)-H(25A)	110.4(9)
C(24)-C(25)-H(25A)	110.6(9)
O(25)-C(25)-H(25B)	110.9(9)
C(24)-C(25)-H(25B)	104.9(9)
H(25A)-C(25)-H(25B)	109.0(12)
C(25)-O(25)-H(25)	106.7(12)

Symmetry transformations used to generate equivalent atoms:
#1 -x+2, -y+1, -z

Table E58. Anisotropic displacement parameters ($Å^2 \times 10^3$) for **2A•($C_6H_{15}NO_3$)$_2$**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+...+2hka^*b^*U_{12}]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O(1)	25(1)	53(1)	23(1)	14(1)	11(1)	4(1)
C(1)	21(1)	22(1)	18(1)	1(1)	7(1)	-1(1)
O(2)	21(1)	35(1)	23(1)	4(1)	11(1)	-1(1)
C(2)	20(1)	21(1)	19(1)	-2(1)	9(1)	-2(1)
C(3)	18(1)	22(1)	19(1)	0(1)	6(1)	-1(1)
C(4)	19(1)	24(1)	18(1)	1(1)	7(1)	0(1)
C(5)	17(1)	28(1)	21(1)	-4(1)	7(1)	2(1)
C(6)	28(1)	35(1)	25(1)	-1(1)	4(1)	3(1)
C(7)	33(1)	52(1)	26(1)	-6(1)	-2(1)	0(1)
C(8)	30(1)	45(1)	37(1)	-16(1)	8(1)	-10(1)
C(9)	31(1)	32(1)	37(1)	-7(1)	17(1)	-7(1)
C(10)	25(1)	31(1)	24(1)	-3(1)	10(1)	-2(1)
C(11)	20(1)	26(1)	19(1)	-1(1)	4(1)	2(1)
C(12)	27(1)	29(1)	25(1)	-2(1)	6(1)	-3(1)
C(13)	41(1)	28(1)	31(1)	-6(1)	6(1)	1(1)
C(14)	38(1)	40(1)	32(1)	-9(1)	12(1)	8(1)
C(15)	37(1)	43(1)	47(1)	-8(1)	26(1)	-1(1)
C(16)	33(1)	30(1)	42(1)	-6(1)	21(1)	-4(1)
N(20)	30(1)	27(1)	22(1)	-2(1)	14(1)	-5(1)
C(20)	44(1)	28(1)	27(1)	-3(1)	19(1)	-9(1)
C(21)	42(1)	36(1)	33(1)	-10(1)	16(1)	-8(1)
O(21)	76(1)	32(1)	31(1)	-6(1)	31(1)	-14(1)
C(22)	30(1)	38(1)	25(1)	2(1)	13(1)	-1(1)
C(23)	44(1)	36(1)	28(1)	0(1)	16(1)	5(1)
O(23)	52(1)	35(1)	31(1)	-2(1)	16(1)	-9(1)
C(24)	28(1)	32(1)	28(1)	-5(1)	10(1)	-3(1)
C(25)	39(1)	31(1)	24(1)	-1(1)	7(1)	5(1)
O(25)	47(1)	38(1)	26(1)	4(1)	18(1)	9(1)

Table E59. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for **2A•$(C_6H_{15}NO_3)_2$**.

Atom	x	y	z	U_{eq}	Occ.
H(4)	7763(10)	6426(12)	-1482(8)	19(3)	1
H(6)	6381(12)	5780(15)	-2683(9)	36(4)	1
H(7)	5158(15)	4155(17)	-3511(12)	53(5)	1
H(8)	4948(14)	2085(18)	-2885(11)	51(5)	1
H(9)	5973(13)	1672(16)	-1424(10)	40(4)	1
H(10)	7159(12)	3322(14)	-597(10)	33(4)	1
H(12)	7860(13)	8373(15)	-680(10)	37(4)	1
H(13)	7013(13)	9787(17)	61(10)	45(4)	1
H(14)	5676(14)	8956(18)	648(11)	50(5)	1
H(15)	5196(15)	6700(17)	488(11)	52(5)	1
H(16)	6027(13)	5261(16)	-245(10)	41(4)	1
H(20)	9150(12)	3928(14)	2036(9)	32(4)	1
H(20A)	8159(13)	1981(15)	2783(10)	40(4)	1
H(20B)	9366(13)	1737(15)	2605(10)	36(4)	1
H(21A)	7829(14)	743(17)	1548(10)	45(4)	1
H(21B)	7243(15)	2169(17)	1261(11)	48(5)	1
H(21)	8630(20)	2580(30)	870(15)	88(8)	1
H(22A)	7465(12)	4184(14)	2798(9)	32(4)	1
H(22B)	7268(13)	4209(15)	1785(10)	38(4)	1
H(23A)	7223(15)	6358(17)	2298(11)	49(4)	1
H(23B)	8443(12)	6228(15)	3020(10)	34(4)	1
H(23)	8273(18)	6050(20)	1369(15)	77(7)	1
H(24A)	9904(12)	4899(15)	3350(9)	32(4)	1
H(24B)	10450(12)	3507(14)	3284(9)	29(3)	1
H(25A)	9386(12)	2540(16)	4100(9)	34(4)	1
H(25B)	10316(14)	3613(16)	4604(11)	46(4)	1
H(25)	8678(15)	3975(19)	4823(13)	61(5)	1

Table E60. Torsion angles (°) for **2A•($C_6H_{15}NO_3$)$_2$**.

Atoms	Angle
O(1) C(1) C(2) O(2)	-5.16(14)
C(3)#1 C(1) C(2) O(2)	174.91(10)
O(1) C(1) C(2) C(3)	175.43(10)
C(3)#1 C(1) C(2) C(3)	-4.50(17)
O(2) C(2) C(3) C(1)#1	-174.96(10)
C(1) C(2) C(3) C(1)#1	4.40(16)
O(2) C(2) C(3) C(4)	4.26(17)
C(1) C(2) C(3) C(4)	-176.38(9)
C(2) C(3) C(4) C(5)	-80.41(13)
C(1)#1 C(3) C(4) C(5)	98.81(11)
C(2) C(3) C(4) C(11)	50.72(14)
C(1)#1 C(3) C(4) C(11)	-130.06(10)
C(3) C(4) C(5) C(10)	47.27(14)
C(11) C(4) C(5) C(10)	-84.03(13)
C(3) C(4) C(5) C(6)	-132.95(11)
C(11) C(4) C(5) C(6)	95.75(12)
C(10) C(5) C(6) C(7)	0.99(17)
C(4) C(5) C(6) C(7)	-178.81(11)
C(5) C(6) C(7) C(8)	-0.3(2)
C(6) C(7) C(8) C(9)	-0.4(2)
C(7) C(8) C(9) C(10)	0.5(2)
C(6) C(5) C(10) C(9)	-0.93(16)
C(4) C(5) C(10) C(9)	178.85(10)
C(8) C(9) C(10) C(5)	0.21(18)
C(5) C(4) C(11) C(16)	32.14(15)
C(3) C(4) C(11) C(16)	-99.94(12)
C(5) C(4) C(11) C(12)	-146.54(10)
C(3) C(4) C(11) C(12)	81.37(12)
C(16) C(11) C(12) C(13)	0.68(17)
C(4) C(11) C(12) C(13)	179.45(10)
C(11) C(12) C(13) C(14)	-0.45(18)
C(12) C(13) C(14) C(15)	0.1(2)
C(13) C(14) C(15) C(16)	0.0(2)
C(12) C(11) C(16) C(15)	-0.56(18)
C(4) C(11) C(16) C(15)	-179.25(12)
C(14) C(15) C(16) C(11)	0.2(2)
C(22) N(20) C(20) C(21)	70.26(13)
C(24) N(20) C(20) C(21)	-161.64(11)
N(20) C(20) C(21) O(21)	70.00(15)
C(24) N(20) C(22) C(23)	55.21(13)
C(20) N(20) C(22) C(23)	-178.91(10)

Table E60. continued...

N(20) C(22) C(23) O(23)	52.93(15)
C(22) N(20) C(24) C(25)	69.21(14)
C(20) N(20) C(24) C(25)	-57.58(13)
N(20) C(24) C(25) O(25)	-69.62(14)

Symmetry transformations used to generate equivalent atoms:
#1 -x+2, -y+1, -z

Table E61. Hydrogen bonds, lengths (Å) and angles (°) for **2A•$(C_6H_{15}NO_3)_2$**.

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(20)-H(20)...O(1)	0.971(15)	1.966(15)	2.8405(14)	148.7(12)
N(20)-H(20)...O(2)	0.971(15)	2.233(14)	2.9768(14)	132.6(11)
O(21)-H(21)...O(2)	0.83(3)	1.94(3)	2.7630(14)	169(2)
O(21)-H(21)...O(1)	0.83(3)	2.57(2)	3.0827(15)	121(2)
O(23)-H(23)...O(2)	0.88(2)	2.00(2)	2.7644(14)	146(2)
O(25)-H(25)...O(21)#2	0.91(2)	1.99(2)	2.8988(14)	175.7(18)

Symmetry transformations used to generate equivalent atoms:
#2 x, -y+1/2, z+1/2

Table E62. Crystal and structure refinement data for **2B•$(C_6H_{15}N)_2$•$(C_3H_6O)_2$**.

Identification code	**2B•$(C_6H_{15}N)_2$•$(C_3H_6O)_2$**
Empirical formula	$C_{50}H_{62}Cl_4N_2O_6$
Formula weight	928.82 g/mole
Temperature	173(2) K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group	$P^{-}1$
Unit cell dimensions	a = 9.755(2) Å b = 11.204(2) Å c = 11.844(2) Å α = 76.30(3)° β = 89.02(3)° γ = 76.19(3)°
Volume	1220.2(4) $Å^3$
Z	1
Density (calculated)	1.264 Mg/m^3
Absorption coefficient	0.292 mm^{-1}
F(000)	492
Crystal Size	0.28 mm x 0.14 mm x 0.08 mm
Color	orange
Theta range for data collection	1.93° to 28.38°
Index ranges	$-12 \le h \le 13$, $-14 \le k \le 14$, $-6 \le l \le 15$
Reflections collected / Unique	7860 / 5494 [R_{int} = 0.0229]
Refinement method	Full-matrix-least-squares on F^2
Data / Restraints / Parameters	5494 / 0 / 405
Goodness-of-fit on F^2	0.929
Final R indices [$I>2\sigma(I)$]	R_1 = 0.0465, wR_2 = 0.0969
R indices (all data)	R_1 = 0.0943, wR_2 = 0.1111
Extinction Coefficient	0.0026(14)
Largest diff. peak and hole	0.338 and -0.245 e/A^3

Table E63. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for **2B•$(C_6H_{15}N)_2$•$(C_3H_6O)_2$**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U_{eq}	Occ.
C(1)	5354(2)	15979(2)	461(2)	21(1)	1
O(1)	5796(1)	16735(1)	894(1)	29(1)	1
N(1)	11498(2)	13866(2)	-2053(2)	29(1)	1
C(2)	6377(2)	14678(2)	546(2)	22(1)	1
O(2)	7547(1)	14513(1)	1071(1)	32(1)	1
C(3)	6001(2)	13757(2)	81(2)	21(1)	1
C(4)	7047(2)	12473(2)	250(2)	22(1)	1
C(5)	6584(2)	11486(2)	1198(2)	22(1)	1
C(6)	5546(2)	10882(2)	996(2)	28(1)	1
C(7)	5079(2)	10054(2)	1886(2)	31(1)	1
C(8)	5657(2)	9814(2)	3000(2)	30(1)	1
Cl(8)	5028(1)	8801(1)	4131(1)	45(1)	1
C(9)	6708(2)	10367(2)	3230(2)	32(1)	1
C(10)	7168(2)	11192(2)	2323(2)	28(1)	1
C(11)	7467(2)	12039(2)	-865(2)	23(1)	1
C(12)	8272(2)	10824(2)	-802(2)	32(1)	1
C(13)	8764(2)	10426(2)	-1792(2)	39(1)	1
C(14)	8441(2)	11262(2)	-2861(2)	35(1)	1
Cl(14)	9030(1)	10777(1)	-4117(1)	56(1)	1
C(15)	7662(2)	12473(2)	-2954(2)	35(1)	1
C(16)	7178(2)	12854(2)	-1957(2)	30(1)	1
C(20)	11205(3)	12783(2)	-1147(2)	36(1)	1
C(21)	10879(3)	13114(3)	6(2)	49(1)	1
C(22)	12259(3)	13413(3)	-3046(2)	37(1)	1
C(23)	11379(3)	12870(3)	-3738(3)	49(1)	1
C(24)	10197(3)	14910(2)	-2428(2)	40(1)	1
C(25)	10499(4)	16029(3)	-3297(3)	63(1)	1
C(30)	6040(3)	6084(2)	6388(2)	41(1)	1
O(30)	6946(2)	5534(2)	5856(2)	53(1)	1
C(31)	6376(3)	6785(3)	7224(3)	53(1)	1
C(32)	4522(3)	6078(3)	6251(3)	54(1)	1

Table E64. Bond lengths (Å) and angles (°) for **2B•$(C_6H_{15}N)_2$•$(C_3H_6O)_2$**.

Atoms	Length	Angle
C(1)-O(1)	1.246(2)	
C(1)-C(3)#1	1.413(3)	
C(1)-C(2)	1.540(3)	
N(1)-C(24)	1.497(3)	
N(1)-C(20)	1.498(3)	
N(1)-C(22)	1.505(3)	
N(1)-H(1)	0.97(2)	
C(2)-O(2)	1.261(2)	
C(2)-C(3)	1.400(3)	
C(3)-C(1)#1	1.413(3)	
C(3)-C(4)	1.524(3)	
C(4)-C(5)	1.523(3)	
C(4)-C(11)	1.529(3)	
C(4)-H(4)	0.991(18)	
C(5)-C(10)	1.393(3)	
C(5)-C(6)	1.397(3)	
C(6)-C(7)	1.378(3)	
C(6)-H(6)	0.93(2)	
C(7)-C(8)	1.384(3)	
C(7)-H(7)	0.92(2)	
C(8)-C(9)	1.379(3)	
C(8)-Cl(8)	1.744(2)	
C(9)-C(10)	1.386(3)	
C(9)-H(9)	0.96(2)	
C(10)-H(10)	0.98(2)	
C(11)-C(12)	1.385(3)	
C(11)-C(16)	1.385(3)	
C(12)-C(13)	1.389(3)	
C(12)-H(12)	0.89(2)	
C(13)-C(14)	1.376(3)	
C(13)-H(13)	0.97(2)	
C(14)-C(15)	1.367(3)	
C(14)-Cl(14)	1.745(2)	
C(15)-C(16)	1.387(3)	
C(15)-H(15)	0.92(2)	
C(16)-H(16)	0.96(2)	
C(20)-C(21)	1.507(4)	
C(20)-H(20A)	0.98(2)	
C(20)-H(20B)	0.95(2)	
C(21)-H(21A)	0.97(3)	
C(21)-H(21B)	1.00(3)	

Table E64. continued...

C(21)-H(21C)	1.01(3)
C(22)-C(23)	1.513(3)
C(22)-H(22A)	0.99(2)
C(22)-H(22B)	0.94(2)
C(23)-H(23A)	1.02(3)
C(23)-H(23B)	0.95(3)
C(23)-H(23C)	0.95(3)
C(24)-C(25)	1.507(4)
C(24)-H(24A)	1.03(2)
C(24)-H(24B)	0.96(2)
C(25)-H(25A)	1.02(3)
C(25)-H(25B)	1.01(4)
C(25)-H(25C)	0.99(3)
C(30)-O(30)	1.209(3)
C(30)-C(31)	1.486(4)
C(30)-C(32)	1.495(4)
C(31)-H(31A)	0.95(4)
C(31)-H(31B)	0.94(5)
C(31)-H(31C)	0.95(4)
C(32)-H(32A)	1.00(3)
C(32)-H(32B)	0.97(3)
C(32)-H(32C)	0.91(3)

O(1)-C(1)-C(3)#1	124.38(18)
O(1)-C(1)-C(2)	116.21(17)
C(3)#1-C(1)-C(2)	119.40(16)
C(24)-N(1)-C(20)	112.03(18)
C(24)-N(1)-C(22)	113.69(19)
C(20)-N(1)-C(22)	111.04(18)
C(24)-N(1)-H(1)	107.4(14)
C(20)-N(1)-H(1)	103.3(14)
C(22)-N(1)-H(1)	108.8(14)
O(2)-C(2)-C(3)	123.65(17)
O(2)-C(2)-C(1)	115.07(16)
C(3)-C(2)-C(1)	121.28(16)
C(2)-C(3)-C(1)#1	119.31(17)
C(2)-C(3)-C(4)	117.99(17)
C(1)#1-C(3)-C(4)	122.59(16)
C(5)-C(4)-C(3)	110.78(16)
C(5)-C(4)-C(11)	114.08(15)
C(3)-C(4)-C(11)	115.38(17)
C(5)-C(4)-H(4)	105.4(11)
C(3)-C(4)-H(4)	103.4(10)
C(11)-C(4)-H(4)	106.6(11)

Table E64. continued...

C(10)-C(5)-C(6)	117.75(19)
C(10)-C(5)-C(4)	119.70(17)
C(6)-C(5)-C(4)	122.51(18)
C(7)-C(6)-C(5)	121.4(2)
C(7)-C(6)-H(6)	120.5(13)
C(5)-C(6)-H(6)	118.0(13)
C(6)-C(7)-C(8)	119.2(2)
C(6)-C(7)-H(7)	119.5(13)
C(8)-C(7)-H(7)	121.2(13)
C(9)-C(8)-C(7)	121.2(2)
C(9)-C(8)-Cl(8)	119.68(18)
C(7)-C(8)-Cl(8)	119.08(17)
C(8)-C(9)-C(10)	118.7(2)
C(8)-C(9)-H(9)	119.6(13)
C(10)-C(9)-H(9)	121.7(13)
C(9)-C(10)-C(5)	121.7(2)
C(9)-C(10)-H(10)	118.0(13)
C(5)-C(10)-H(10)	120.2(12)
C(12)-C(11)-C(16)	117.4(2)
C(12)-C(11)-C(4)	120.04(19)
C(16)-C(11)-C(4)	122.31(17)
C(11)-C(12)-C(13)	121.7(2)
C(11)-C(12)-H(12)	119.4(15)
C(13)-C(12)-H(12)	118.9(15)
C(14)-C(13)-C(12)	119.0(2)
C(14)-C(13)-H(13)	116.0(15)
C(12)-C(13)-H(13)	125.0(15)
C(15)-C(14)-C(13)	120.9(2)
C(15)-C(14)-Cl(14)	119.4(2)
C(13)-C(14)-Cl(14)	119.74(18)
C(14)-C(15)-C(16)	119.4(2)
C(14)-C(15)-H(15)	119.6(15)
C(16)-C(15)-H(15)	121.0(15)
C(11)-C(16)-C(15)	121.6(2)
C(11)-C(16)-H(16)	117.0(12)
C(15)-C(16)-H(16)	121.3(12)
N(1)-C(20)-C(21)	112.5(2)
N(1)-C(20)-H(20A)	104.1(14)
C(21)-C(20)-H(20A)	110.4(14)
N(1)-C(20)-H(20B)	107.7(13)
C(21)-C(20)-H(20B)	112.1(13)
H(20A)-C(20)-H(20B)	109.7(18)
C(20)-C(21)-H(21A)	109.8(15)
C(20)-C(21)-H(21B)	114.1(16)

Table E64. continued...

H(21A)-C(21)-H(21B)	106(2)
C(20)-C(21)-H(21C)	110.1(17)
H(21A)-C(21)-H(21C)	106(2)
H(21B)-C(21)-H(21C)	111(2)
N(1)-C(22)-C(23)	113.5(2)
N(1)-C(22)-H(22A)	105.1(14)
C(23)-C(22)-H(22A)	110.5(13)
N(1)-C(22)-H(22B)	105.5(13)
C(23)-C(22)-H(22B)	111.6(13)
H(22A)-C(22)-H(22B)	110.3(19)
C(22)-C(23)-H(23A)	107.0(14)
C(22)-C(23)-H(23B)	109.9(16)
H(23A)-C(23)-H(23B)	101(2)
C(22)-C(23)-H(23C)	110.8(16)
H(23A)-C(23)-H(23C)	113(2)
H(23B)-C(23)-H(23C)	115(2)
N(1)-C(24)-C(25)	112.3(2)
N(1)-C(24)-H(24A)	108.5(13)
C(25)-C(24)-H(24A)	111.8(13)
N(1)-C(24)-H(24B)	107.9(13)
C(25)-C(24)-H(24B)	111.7(14)
H(24A)-C(24)-H(24B)	104.3(18)
C(24)-C(25)-H(25A)	108.0(18)
C(24)-C(25)-H(25B)	115(2)
H(25A)-C(25)-H(25B)	107(3)
C(24)-C(25)-H(25C)	111.8(17)
H(25A)-C(25)-H(25C)	112(2)
H(25B)-C(25)-H(25C)	104(3)
O(30)-C(30)-C(31)	122.0(3)
O(30)-C(30)-C(32)	121.6(3)
C(31)-C(30)-C(32)	116.4(3)
C(30)-C(31)-H(31A)	115(2)
C(30)-C(31)-H(31B)	111(2)
H(31A)-C(31)-H(31B)	112(3)
C(30)-C(31)-H(31C)	110(2)
H(31A)-C(31)-H(31C)	109(3)
H(31B)-C(31)-H(31C)	98(3)
C(30)-C(32)-H(32A)	110.0(17)
C(30)-C(32)-H(32B)	113.8(18)
H(32A)-C(32)-H(32B)	112(3)
C(30)-C(32)-H(32C)	108.1(19)
H(32A)-C(32)-H(32C)	105(2)
H(32B)-C(32)-H(32C)	107(3)

Table E64. continued...

Symmetry transformations used to generate equivalent atoms:
#1 -x+1,-y+3,-z

Table E65. Anisotropic displacement parameters ($Å^2 \times 10^3$) for **2B•($C_6H_{15}N$)$_2$•(C_3H_6O)$_2$**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+...+2hka^*b^*U_{12}]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(1)	22(1)	25(1)	19(1)	-6(1)	4(1)	-8(1)
O(1)	26(1)	27(1)	36(1)	-13(1)	-5(1)	-5(1)
N(1)	25(1)	36(1)	31(1)	-10(1)	-2(1)	-12(1)
C(2)	20(1)	24(1)	20(1)	-3(1)	1(1)	-5(1)
O(2)	22(1)	30(1)	44(1)	-14(1)	-10(1)	-2(1)
C(3)	20(1)	21(1)	20(1)	-4(1)	2(1)	-4(1)
C(4)	19(1)	21(1)	26(1)	-6(1)	-1(1)	-4(1)
C(5)	19(1)	22(1)	26(1)	-8(1)	2(1)	-2(1)
C(6)	30(1)	30(1)	24(1)	-7(1)	0(1)	-8(1)
C(7)	32(1)	34(1)	33(1)	-11(1)	4(1)	-15(1)
C(8)	34(1)	27(1)	29(1)	-6(1)	9(1)	-9(1)
Cl(8)	54(1)	46(1)	34(1)	-1(1)	13(1)	-22(1)
C(9)	36(1)	36(1)	22(1)	-6(1)	-1(1)	-7(1)
C(10)	27(1)	29(1)	28(1)	-8(1)	-1(1)	-7(1)
C(11)	17(1)	24(1)	30(1)	-10(1)	4(1)	-6(1)
C(12)	32(1)	26(1)	36(1)	-6(1)	7(1)	-3(1)
C(13)	38(1)	30(1)	51(2)	-18(1)	15(1)	-4(1)
C(14)	29(1)	48(1)	37(1)	-26(1)	12(1)	-13(1)
Cl(14)	53(1)	79(1)	50(1)	-44(1)	21(1)	-17(1)
C(15)	34(1)	44(1)	27(1)	-10(1)	5(1)	-7(1)
C(16)	30(1)	28(1)	28(1)	-7(1)	5(1)	-2(1)
C(20)	35(1)	36(1)	42(1)	-10(1)	4(1)	-16(1)
C(21)	59(2)	50(2)	41(2)	-9(1)	18(1)	-21(2)
C(22)	33(1)	52(2)	31(1)	-16(1)	4(1)	-16(1)
C(23)	46(2)	68(2)	43(2)	-28(2)	-2(1)	-17(2)
C(24)	31(1)	42(1)	49(2)	-14(1)	-10(1)	-6(1)
C(25)	55(2)	53(2)	70(2)	6(2)	-27(2)	-11(2)
C(30)	50(2)	33(1)	30(1)	4(1)	9(1)	-4(1)
O(30)	59(1)	47(1)	44(1)	-9(1)	18(1)	2(1)
C(31)	43(2)	62(2)	54(2)	-23(2)	6(2)	-4(2)
C(32)	56(2)	61(2)	48(2)	-10(2)	11(2)	-19(2)

Table E66. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for **2B•$(C_6H_{15}N)_2$•$(C_3H_6O)_2$**.

Atom	x	y	z	U_{eq}	Occ.
H(1)	12130(30)	14190(20)	-1640(20)	50(7)	1
H(4)	7910(20)	12609(17)	577(16)	19(5)	1
H(6)	5140(20)	11089(19)	250(19)	27(6)	1
H(7)	4420(20)	9640(20)	1723(18)	30(6)	1
H(9)	7120(20)	10160(20)	4010(20)	32(6)	1
H(10)	7890(20)	11602(19)	2501(18)	30(6)	1
H(12)	8480(20)	10280(20)	-110(20)	35(6)	1
H(13)	9350(20)	9600(20)	-1790(20)	45(7)	1
H(15)	7470(20)	13020(20)	-3680(20)	40(7)	1
H(16)	6650(20)	13700(20)	-1993(17)	25(5)	1
H(20A)	12080(30)	12120(20)	-1080(20)	42(7)	1
H(20B)	10460(20)	12520(20)	-1443(19)	33(6)	1
H(21A)	10850(30)	12360(30)	600(20)	60(8)	1
H(21B)	9940(30)	13720(30)	10(20)	62(8)	1
H(21C)	11660(30)	13450(30)	270(30)	75(9)	1
H(22A)	13110(30)	12760(20)	-2680(20)	42(7)	1
H(22B)	12520(20)	14120(20)	-3510(20)	35(6)	1
H(23A)	11960(30)	12670(20)	-4430(20)	54(7)	1
H(23B)	10600(30)	13520(30)	-4120(20)	61(9)	1
H(23C)	11130(30)	12150(30)	-3260(20)	55(8)	1
H(24A)	9750(20)	15160(20)	-1700(20)	41(6)	1
H(24B)	9520(20)	14570(20)	-2730(20)	39(6)	1
H(25A)	9590(30)	16720(30)	-3460(30)	83(10)	1
H(25B)	10820(40)	15860(40)	-4070(40)	124(16)	1
H(25C)	11280(30)	16330(30)	-3010(30)	73(9)	1
H(31A)	7350(40)	6720(30)	7340(30)	92(11)	1
H(31B)	5930(40)	6580(40)	7930(40)	130(16)	1
H(31C)	5890(40)	7660(40)	6990(30)	110(13)	1
H(32A)	4410(30)	5560(30)	5690(30)	74(9)	1
H(32B)	3890(30)	6920(30)	6050(30)	81(10)	1
H(32C)	4250(30)	5670(30)	6950(30)	77(10)	1

Table E67. Torsion angles (°) for **2B•$(C_6H_{15}N)_2$•$(C_3H_6O)_2$**.

Atoms	Angle
O(1) C(1) C(2) O(2)	1.4(2)
C(3)#1 C(1) C(2) O(2)	-178.17(17)
O(1) C(1) C(2) C(3)	-179.38(17)
C(3)#1 C(1) C(2) C(3)	1.0(3)
O(2) C(2) C(3) C(1)#1	178.10(18)
C(1) C(2) C(3) C(1)#1	-1.0(3)
O(2) C(2) C(3) C(4)	1.9(3)
C(1) C(2) C(3) C(4)	-177.27(17)
C(2) C(3) C(4) C(5)	103.4(2)
C(1)#1 C(3) C(4) C(5)	-72.7(2)
C(2) C(3) C(4) C(11)	-125.05(19)
C(1)#1 C(3) C(4) C(11)	58.8(2)
C(3) C(4) C(5) C(10)	-97.6(2)
C(11) C(4) C(5) C(10)	130.18(19)
C(3) C(4) C(5) C(6)	80.1(2)
C(11) C(4) C(5) C(6)	-52.1(3)
C(10) C(5) C(6) C(7)	2.1(3)
C(4) C(5) C(6) C(7)	-175.68(19)
C(5) C(6) C(7) C(8)	-0.3(3)
C(6) C(7) C(8) C(9)	-1.3(3)
C(6) C(7) C(8) Cl(8)	178.16(16)
C(7) C(8) C(9) C(10)	1.0(3)
Cl(8) C(8) C(9) C(10)	-178.45(16)
C(8) C(9) C(10) C(5)	0.9(3)
C(6) C(5) C(10) C(9)	-2.4(3)
C(4) C(5) C(10) C(9)	175.45(19)
C(5) C(4) C(11) C(12)	-42.1(3)
C(3) C(4) C(11) C(12)	-172.06(17)
C(5) C(4) C(11) C(16)	143.77(19)
C(3) C(4) C(11) C(16)	13.8(3)
C(16) C(11) C(12) C(13)	-0.7(3)
C(4) C(11) C(12) C(13)	-175.1(2)
C(11) C(12) C(13) C(14)	0.0(3)
C(12) C(13) C(14) C(15)	0.8(3)
C(12) C(13) C(14) Cl(14)	-179.25(17)
C(13) C(14) C(15) C(16)	-0.8(3)
Cl(14) C(14) C(15) C(16)	179.18(17)
C(12) C(11) C(16) C(15)	0.6(3)
C(4) C(11) C(16) C(15)	174.8(2)
C(14) C(15) C(16) C(11)	0.1(3)
C(24) N(1) C(20) C(21)	-69.9(3)

Table E67. continued...

C(22) N(1) C(20) C(21)	161.8(2)
C(24) N(1) C(22) C(23)	-61.3(3)
C(20) N(1) C(22) C(23)	66.1(3)
C(20) N(1) C(24) C(25)	177.2(2)
C(22) N(1) C(24) C(25)	-55.9(3)

Symmetry transformations used to generate equivalent atoms:
#1 -x+1, -y+3, -z

Table E68. Hydrogen bonds, lengths (Å) and angles (°) for **2B•$(C_6H_{15}N)_2$•$(C_3H_6O)_2$**.

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1)...O(2)#2	0.97(2)	1.84(2)	2.713(2)	149(2)
N(1)-H(1)...O(1)#2	0.97(2)	2.14(2)	2.853(2)	129.2(19)

Symmetry transformations used to generate equivalent atoms:
#2 -x+2, -y+3, -z

Table E69. Crystal and structure refinement data for **2B•$(C_4H_{13}N_3)_2$**.

Identification code	**2B•$(C_4H_{13}N_3)_2$**
Empirical formula	$C_{40}H_{46}Cl_4N_6O_4$
Formula weight	816.63 g/mole
Temperature	173(2) K
Wavelength	0.71073 Å
Crystal system	Orthorhombic
Space group	Fdd2
Unit cell dimensions	a = 24.048(5) Å b = 38.267(8) Å c = 8.7813(18) Å α = 90.000° β = 90.000° γ = 90.000°
Volume	8081(3) $Å^3$
Z	8
Density (calculated)	1.342 Mg/m^3
Absorption coefficient	0.341 mm^{-1}
F(000)	3424
Crystal Size	0.70 mm x 0.20 mm x 0.10 mm
Color	red
Theta range for data collection	2.00° to 28.33°
Index ranges	$-30 \le h \le 31$, $-44 \le k \le 49$, $-11 \le l \le 10$
Reflections collected / Unique	12792 / 4376 [R_{int} = 0.1330]
Refinement method	Full-matrix-least-squares on F^2
Data / Restraints / Parameters	4376 / 3 / 337
Goodness-of-fit on F^2	0.994
Final R indices [$I>2\sigma(I)$]	R_1 = 0.0580, wR_2 = 0.1222
R indices (all data)	R_1 = 0.1053, wR_2 = 0.1385
Absolute structure parameter	0.44(9)
Extinction Coefficient	0.00015(7)
Largest diff. peak and hole	0.372 and -0.374 e/A^3

Table E70. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for **2B•($C_4H_{13}N_3$)$_2$**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U_{eq}	Occ.
C(1)	2789(2)	2408(1)	5918(4)	21(1)	1
O(1)	2999(1)	2354(1)	7202(3)	26(1)	1
C(2)	3043(1)	2315(1)	4536(5)	21(1)	1
C(3)	2789(2)	2412(1)	3164(4)	22(1)	1
O(3)	3011(1)	2372(1)	1877(3)	31(1)	1
C(4)	3624(2)	2160(1)	4447(5)	22(1)	1
C(5)	3726(2)	1826(1)	5336(5)	22(1)	1
C(6)	4271(2)	1710(1)	5593(5)	28(1)	1
C(7)	4383(2)	1394(1)	6312(5)	30(1)	1
C(8)	3945(2)	1189(1)	6797(5)	27(1)	1
Cl(8)	4092(1)	787(1)	7662(1)	44(1)	1
C(9)	3403(2)	1291(1)	6572(5)	33(1)	1
C(10)	3295(2)	1611(1)	5840(5)	28(1)	1
C(11)	4065(2)	2441(1)	4736(4)	23(1)	1
C(12)	4216(2)	2540(1)	6194(5)	28(1)	1
C(13)	4615(2)	2797(1)	6444(6)	35(1)	1
C(14)	4862(2)	2950(1)	5207(6)	38(1)	1
Cl(14)	5382(1)	3267(1)	5512(2)	65(1)	1
C(15)	4726(2)	2860(1)	3734(6)	41(1)	1
C(16)	4322(2)	2603(1)	3515(6)	32(1)	1
N(20)	3380(2)	967(1)	2735(5)	38(1)	1
C(20)	3463(2)	1248(1)	1626(6)	41(1)	1
C(21)	2925(2)	1421(1)	1164(6)	42(1)	1
N(21)	2984(2)	1702(1)	9(5)	44(1)	1
C(22)	3893(2)	776(1)	3078(6)	38(1)	1
C(23)	3990(2)	502(1)	1885(6)	36(1)	1
N(23)	4550(1)	339(1)	2040(5)	28(1)	1

Table E71. Bond lengths (Å) and angles (°) for **2B•($C_4H_{13}N_3$)$_2$**.

Atoms	Length	Angle
C(1)-O(1)	1.253(4)	
C(1)-C(2)	1.403(5)	
C(1)-C(1)#1	1.559(7)	
C(2)-C(3)	1.401(5)	
C(2)-C(4)	1.520(5)	
C(3)-O(3)	1.259(4)	
C(3)-C(3)#1	1.545(7)	
C(4)-C(5)	1.519(5)	
C(4)-C(11)	1.530(5)	
C(4)-H(4)	0.97(4)	
C(5)-C(10)	1.394(5)	
C(5)-C(6)	1.402(5)	
C(6)-C(7)	1.390(6)	
C(6)-H(6)	1.05(4)	
C(7)-C(8)	1.380(6)	
C(7)-H(7)	1.00(4)	
C(8)-C(9)	1.375(6)	
C(8)-Cl(8)	1.753(4)	
C(9)-C(10)	1.407(5)	
C(9)-H(9)	0.83(5)	
C(10)-H(10)	0.98(4)	
C(11)-C(12)	1.384(6)	
C(11)-C(16)	1.386(6)	
C(12)-C(13)	1.390(6)	
C(12)-H(12)	0.87(4)	
C(13)-C(14)	1.371(7)	
C(13)-H(13)	0.91(4)	
C(14)-C(15)	1.378(7)	
C(14)-Cl(14)	1.762(4)	
C(15)-C(16)	1.394(6)	
C(15)-H(15)	0.92(5)	
C(16)-H(16)	1.00(4)	
N(20)-C(20)	1.464(6)	
N(20)-C(22)	1.466(6)	
N(20)-H(20A)	0.95(5)	
C(20)-C(21)	1.509(6)	
C(20)-H(20B)	1.15(6)	
C(20)-H(20C)	0.98(5)	
C(21)-N(21)	1.484(6)	
C(21)-H(21A)	1.09(8)	
C(21)-H(21B)	1.10(6)	

Table E71. continued...

N(21)-H(21C)	0.82(4)
N(21)-H(21D)	0.91(6)
C(22)-C(23)	1.499(6)
C(22)-H(22A)	0.87(5)
C(22)-H(22B)	1.13(4)
C(23)-N(23)	1.491(5)
C(23)-H(23A)	1.09(6)
C(23)-H(23B)	0.97(4)
N(23)-H(23C)	0.90(5)
N(23)-H(23D)	0.99(5)
N(23)-H(23E)	1.10(5)

O(1)-C(1)-C(2)	124.1(3)
O(1)-C(1)-C(1)#1	115.7(2)
C(2)-C(1)-C(1)#1	120.2(2)
C(3)-C(2)-C(1)	119.2(3)
C(3)-C(2)-C(4)	117.3(3)
C(1)-C(2)-C(4)	122.9(3)
O(3)-C(3)-C(2)	123.7(3)
O(3)-C(3)-C(3)#1	115.8(2)
C(2)-C(3)-C(3)#1	120.5(2)
C(5)-C(4)-C(2)	116.8(3)
C(5)-C(4)-C(11)	113.2(3)
C(2)-C(4)-C(11)	110.8(3)
C(5)-C(4)-H(4)	107.4(19)
C(2)-C(4)-H(4)	102.0(19)
C(11)-C(4)-H(4)	105.4(19)
C(10)-C(5)-C(6)	117.2(3)
C(10)-C(5)-C(4)	122.7(3)
C(6)-C(5)-C(4)	120.0(3)
C(7)-C(6)-C(5)	122.0(3)
C(7)-C(6)-H(6)	120(2)
C(5)-C(6)-H(6)	118(2)
C(8)-C(7)-C(6)	119.0(4)
C(8)-C(7)-H(7)	121(2)
C(6)-C(7)-H(7)	120(2)
C(9)-C(8)-C(7)	121.3(4)
C(9)-C(8)-Cl(8)	120.1(3)
C(7)-C(8)-Cl(8)	118.6(3)
C(8)-C(9)-C(10)	119.0(4)
C(8)-C(9)-H(9)	127(3)
C(10)-C(9)-H(9)	114(3)
C(5)-C(10)-C(9)	121.4(4)
C(5)-C(10)-H(10)	125(2)

Table E71. continued...

C(9)-C(10)-H(10)	114(2)
C(12)-C(11)-C(16)	118.4(4)
C(12)-C(11)-C(4)	121.9(4)
C(16)-C(11)-C(4)	119.7(4)
C(11)-C(12)-C(13)	121.5(4)
C(11)-C(12)-H(12)	119(2)
C(13)-C(12)-H(12)	119(2)
C(14)-C(13)-C(12)	118.5(5)
C(14)-C(13)-H(13)	124(3)
C(12)-C(13)-H(13)	118(3)
C(13)-C(14)-C(15)	122.2(4)
C(13)-C(14)-Cl(14)	118.9(4)
C(15)-C(14)-Cl(14)	119.0(4)
C(14)-C(15)-C(16)	118.2(5)
C(14)-C(15)-H(15)	118(3)
C(16)-C(15)-H(15)	123(3)
C(11)-C(16)-C(15)	121.3(5)
C(11)-C(16)-H(16)	118(2)
C(15)-C(16)-H(16)	120(2)
C(20)-N(20)-C(22)	112.9(4)
C(20)-N(20)-H(20A)	115(3)
C(22)-N(20)-H(20A)	104(3)
N(20)-C(20)-C(21)	112.7(4)
N(20)-C(20)-H(20B)	100(3)
C(21)-C(20)-H(20B)	117(3)
N(20)-C(20)-H(20C)	106(3)
C(21)-C(20)-H(20C)	111(3)
H(20B)-C(20)-H(20C)	109(4)
N(21)-C(21)-C(20)	114.9(4)
N(21)-C(21)-H(21A)	98(4)
C(20)-C(21)-H(21A)	112(4)
N(21)-C(21)-H(21B)	105(3)
C(20)-C(21)-H(21B)	110(3)
H(21A)-C(21)-H(21B)	115(5)
C(21)-N(21)-H(21C)	119(4)
C(21)-N(21)-H(21D)	107(4)
H(21C)-N(21)-H(21D)	115(5)
N(20)-C(22)-C(23)	109.6(4)
N(20)-C(22)-H(22A)	113(3)
C(23)-C(22)-H(22A)	110(3)
N(20)-C(22)-H(22B)	110(2)
C(23)-C(22)-H(22B)	109(2)
H(22A)-C(22)-H(22B)	106(4)
N(23)-C(23)-C(22)	111.6(4)

Table E71. continued...

N(23)-C(23)-H(23A)	106(3)
C(22)-C(23)-H(23A)	114(3)
N(23)-C(23)-H(23B)	107(2)
C(22)-C(23)-H(23B)	117(2)
H(23A)-C(23)-H(23B)	100(4)
C(23)-N(23)-H(23C)	109(3)
C(23)-N(23)-H(23D)	116(3)
H(23C)-N(23)-H(23D)	109(4)
C(23)-N(23)-H(23E)	111(3)
H(23C)-N(23)-H(23E)	106(4)
H(23D)-N(23)-H(23E)	105(5)

Symmetry transformations used to generate equivalent atoms:
#1 -x+1/2, -y+1/2, z

Table E72. Anisotropic displacement parameters ($Å^2$ x 10^3) for **2B•($C_4H_{13}N_3$)$_2$**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+...+2hka^*b^*U_{12}]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(1)	26(2)	16(2)	20(2)	-2(2)	-1(2)	-5(1)
O(1)	33(2)	28(1)	18(2)	-1(1)	-4(1)	4(1)
C(2)	23(2)	16(2)	24(2)	-2(2)	-3(2)	1(1)
C(3)	31(2)	15(2)	21(2)	-3(2)	4(2)	-4(2)
O(3)	36(2)	32(2)	25(2)	3(1)	5(1)	8(1)
C(4)	23(2)	20(2)	21(2)	-1(2)	4(2)	3(1)
C(5)	25(2)	19(2)	22(2)	-3(2)	1(2)	3(1)
C(6)	28(2)	23(2)	33(2)	2(2)	5(2)	-1(2)
C(7)	27(2)	25(2)	37(2)	3(2)	-4(2)	5(2)
C(8)	38(2)	14(2)	30(2)	2(2)	-3(2)	6(2)
Cl(8)	51(1)	25(1)	55(1)	13(1)	-4(1)	3(1)
C(9)	33(3)	21(2)	46(3)	2(2)	2(2)	-3(2)
C(10)	24(2)	20(2)	41(3)	0(2)	0(2)	3(2)
C(11)	21(2)	18(2)	29(2)	2(2)	2(2)	5(1)
C(12)	25(2)	26(2)	33(2)	5(2)	3(2)	2(2)
C(13)	32(2)	27(2)	45(3)	-4(2)	-6(2)	1(2)
C(14)	24(2)	23(2)	66(3)	2(2)	-1(2)	-5(2)
Cl(14)	39(1)	45(1)	110(1)	1(1)	-6(1)	-22(1)
C(15)	34(3)	31(2)	57(3)	9(2)	15(2)	-4(2)
C(16)	31(2)	30(2)	34(3)	2(2)	6(2)	0(2)
N(20)	35(2)	39(2)	41(2)	1(2)	6(2)	9(2)
C(20)	32(2)	36(2)	55(3)	4(2)	7(2)	2(2)
C(21)	42(3)	34(2)	50(3)	10(2)	6(2)	2(2)
N(21)	55(3)	31(2)	44(3)	-1(2)	6(2)	6(2)
C(22)	44(3)	41(3)	29(3)	-5(2)	-2(2)	10(2)
C(23)	49(3)	30(2)	30(3)	-6(2)	-9(2)	9(2)
N(23)	34(2)	26(2)	25(2)	1(2)	-2(2)	6(2)

Table E73. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for **2B•$(C_4H_{13}N_3)_2$**.

Atom	x	y	z	U_{eq}	Occ.
H(4)	3655(13)	2103(8)	3370(40)	6(8)	1
H(6)	4599(16)	1884(11)	5350(50)	32(11)	1
H(7)	4775(16)	1323(9)	6510(40)	20(10)	1
H(9)	3120(20)	1191(13)	6900(60)	49(15)	1
H(10)	2899(17)	1661(10)	5720(40)	28(11)	1
H(12)	4055(13)	2441(9)	6970(40)	7(8)	1
H(13)	4709(16)	2846(10)	7420(50)	22(11)	1
H(15)	4894(19)	2980(12)	2950(60)	42(13)	1
H(16)	4187(15)	2548(10)	2470(50)	19(10)	1
H(20A)	3250(20)	1041(13)	3700(60)	51(15)	1
H(20B)	3700(20)	1098(15)	690(80)	86(19)	1
H(20C)	3720(20)	1415(14)	2110(70)	60(15)	1
H(21A)	2660(30)	1250(20)	520(90)	120(20)	1
H(21B)	2730(20)	1546(15)	2150(60)	70(16)	1
H(21C)	3090(20)	1650(13)	-850(50)	50(17)	1
H(21D)	3170(20)	1880(17)	460(70)	70(20)	1
H(22A)	3893(18)	680(12)	3970(50)	35(13)	1
H(22B)	4257(17)	962(12)	3080(50)	36(12)	1
H(23A)	3690(20)	287(15)	1920(70)	72(17)	1
H(23B)	3948(16)	573(10)	830(50)	30(11)	1
H(23C)	4555(16)	204(11)	2880(50)	27(11)	1
H(23D)	4870(20)	501(14)	2070(60)	62(16)	1
H(23E)	4640(30)	162(17)	1080(70)	110(20)	1

Table E74. Torsion angles (°) for **2B•$(C_4H_{13}N_3)_2$**.

Atoms	Angle
O(1) C(1) C(2) C(3)	-175.1(3)
C(1)#1 C(1) C(2) C(3)	4.5(5)
O(1) C(1) C(2) C(4)	-4.3(5)
C(1)#1 C(1) C(2) C(4)	175.3(4)
C(1) C(2) C(3) O(3)	172.7(3)
C(4) C(2) C(3) O(3)	1.4(5)
C(1) C(2) C(3) C(3)#1	-5.8(6)
C(4) C(2) C(3) C(3)#1	-177.2(4)
C(3) C(2) C(4) C(5)	-131.8(4)
C(1) C(2) C(4) C(5)	57.2(5)
C(3) C(2) C(4) C(11)	96.6(4)
C(1) C(2) C(4) C(11)	-74.4(4)
C(2) C(4) C(5) C(10)	18.5(6)
C(11) C(4) C(5) C(10)	149.0(4)
C(2) C(4) C(5) C(6)	-165.7(4)
C(11) C(4) C(5) C(6)	-35.3(5)
C(10) C(5) C(6) C(7)	0.2(6)
C(4) C(5) C(6) C(7)	-175.7(4)
C(5) C(6) C(7) C(8)	-0.4(6)
C(6) C(7) C(8) C(9)	0.5(6)
C(6) C(7) C(8) Cl(8)	178.5(3)
C(7) C(8) C(9) C(10)	-0.4(6)
Cl(8) C(8) C(9) C(10)	-178.3(3)
C(6) C(5) C(10) C(9)	-0.1(6)
C(4) C(5) C(10) C(9)	175.7(4)
C(8) C(9) C(10) C(5)	0.2(6)
C(5) C(4) C(11) C(12)	-50.7(5)
C(2) C(4) C(11) C(12)	82.7(4)
C(5) C(4) C(11) C(16)	129.6(4)
C(2) C(4) C(11) C(16)	-96.9(4)
C(16) C(11) C(12) C(13)	0.0(6)
C(4) C(11) C(12) C(13)	-179.6(3)
C(11) C(12) C(13) C(14)	-0.4(6)
C(12) C(13) C(14) C(15)	0.5(7)
C(12) C(13) C(14) Cl(14)	-178.2(3)
C(13) C(14) C(15) C(16)	0.0(7)
Cl(14) C(14) C(15) C(16)	178.6(3)
C(12) C(11) C(16) C(15)	0.4(6)
C(4) C(11) C(16) C(15)	-180.0(4)
C(14) C(15) C(16) C(11)	-0.4(6)
C(22) N(20) C(20) C(21)	174.5(4)

Table E74. continued...

N(20) C(20) C(21) N(21)	-178.4(4)
C(20) N(20) C(22) C(23)	-83.1(5)
N(20) C(22) C(23) N(23)	170.8(4)

Symmetry transformations used to generate equivalent atoms:
#1 -x+1/2, -y+1/2, z

Table E75. Hydrogen bonds, lengths (Å) and angles(°) **2B•($C_4H_{13}N_3$)$_2$**.

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(21)-H(21D)...O(3)	0.91(6)	2.29(7)	3.043(6)	140(5)
N(23)-H(23C)...O(3)#2	0.90(5)	1.83(5)	2.725(5)	169(4)
N(23)-H(23D)...N(21)#3	0.99(5)	1.90(5)	2.879(5)	170(5)
N(23)-H(23E)...O(1)#4	1.10(5)	1.72(5)	2.770(4)	158(5)
N(23)-H(23E)...O(1)#5	1.10(5)	2.40(6)	3.157(5)	124(4)

Symmetry transformations used to generate equivalent atoms:

#2 -x+3/4, y-1/4, z+1/4
#3 x+1/4, -y+1/4, z+1/4
#4 -x+3/4, y-1/4, z-3/4
#5 x+1/4, -y+1/4, z-3/4

Table E76. Crystal and structure refinement data for **2D•($C_6H_{15}N$)$_2$**.

Identification code	**2D•($C_6H_{15}N$)$_2$**
Empirical formula	$C_{48}H_{62}N_2O_8$
Formula weight	795.00 g/mole
Temperature	173(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	$P2_1/n$
Unit cell dimensions	a = 9.842(2) Å b = 19.698(4) Å c = 11.844(2) Å α = 90.00° β = 104.62(3)° γ = 90.00°
Volume	2221.8(8) Å^3
Z	2
Density (calculated)	1.188 Mg/m^3
Absorption coefficient	0.080 mm^{-1}
F(000)	856
Crystal Size	0.96 mm x 0.84 mm x 0.70 mm
Color	wine red
Theta range for data collection	2.06° to 28.36°
Index ranges	$-13 \le h \le 12$, $-26 \le k \le 22$, $-15 \le l \le 8$
Reflections collected / Unique	14006 / 5291 [R_{int} = 0.0176]
Refinement method	Full-matrix-least-squares on F^2
Data / Restraints / Parameters	5291 / 0 / 387
Goodness-of-fit on F^2	1.049
Final R indices [$I>2\sigma(I)$]	R_1 = 0.0361, wR_2 = 0.0941
R indices (all data)	R_1 = 0.0470, wR_2 = 0.0991
Extinction Coefficient	0.0074(13)
Largest diff. peak and hole	0.235 and -0.167 e/A^3

Table E77. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for **2D•($C_6H_{15}N$)$_2$**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U_{eq}	Occ.
C(1)	336(1)	298(1)	3994(1)	25(1)	1
O(1)	754(1)	596(1)	3212(1)	32(1)	1
N(1)	3633(1)	1064(1)	3560(1)	35(1)	1
C(2)	1428(1)	227(1)	5181(1)	25(1)	1
O(2)	2655(1)	431(1)	5203(1)	32(1)	1
C(3)	1041(1)	-43(1)	6148(1)	25(1)	1
C(4)	2112(1)	-27(1)	7325(1)	26(1)	1
C(5)	1763(1)	504(1)	8152(1)	26(1)	1
C(6)	638(1)	436(1)	8655(1)	32(1)	1
C(7)	358(1)	918(1)	9428(1)	33(1)	1
C(8)	1229(1)	1479(1)	9718(1)	29(1)	1
O(8)	1067(1)	1976(1)	10490(1)	37(1)	1
C(9)	2363(1)	1558(1)	9232(1)	33(1)	1
C(10)	2615(1)	1074(1)	8454(1)	31(1)	1
C(11)	-81(1)	1900(1)	11019(1)	42(1)	1
C(12)	2536(1)	-707(1)	7935(1)	28(1)	1
C(13)	3335(1)	-709(1)	9096(1)	35(1)	1
C(14)	3876(1)	-1303(1)	9654(1)	40(1)	1
C(15)	3656(1)	-1917(1)	9065(1)	36(1)	1
O(15)	4269(1)	-2471(1)	9698(1)	51(1)	1
C(16)	2849(1)	-1934(1)	7920(1)	34(1)	1
C(17)	2295(1)	-1330(1)	7375(1)	31(1)	1
C(18)	4134(2)	-3110(1)	9118(2)	53(1)	1
C(20)	5161(1)	959(1)	4149(1)	49(1)	1
C(21)	5606(2)	1237(1)	5370(2)	57(1)	1
C(22)	3305(2)	816(1)	2323(1)	44(1)	1
C(23)	3411(2)	55(1)	2236(2)	55(1)	1
C(24)	3112(1)	1773(1)	3626(1)	41(1)	1
C(25)	3774(2)	2296(1)	2993(1)	54(1)	1

Table E78. Bond lengths (Å) and angles (°) for **2D•($C_6H_{15}N$)$_2$**.

Atoms	Length	Angle
C(1)-O(1)	1.2504(12)	
C(1)-C(3)#1	1.4149(14)	
C(1)-C(2)	1.5459(14)	
N(1)-C(24)	1.4967(16)	
N(1)-C(22)	1.5005(17)	
N(1)-C(20)	1.5036(16)	
N(1)-H(1)	0.928(15)	
C(2)-O(2)	1.2671(12)	
C(2)-C(3)	1.3986(13)	
C(3)-C(1)#1	1.4148(14)	
C(3)-C(4)	1.5214(14)	
C(4)-C(12)	1.5278(15)	
C(4)-C(5)	1.5303(14)	
C(4)-H(4)	1.002(12)	
C(5)-C(6)	1.3908(15)	
C(5)-C(10)	1.3928(15)	
C(6)-C(7)	1.3934(15)	
C(6)-H(6)	0.978(14)	
C(7)-C(8)	1.3878(15)	
C(7)-H(7)	0.961(14)	
C(8)-O(8)	1.3764(13)	
C(8)-C(9)	1.3878(16)	
O(8)-C(11)	1.4314(15)	
C(9)-C(10)	1.3902(16)	
C(9)-H(9)	0.990(14)	
C(10)-H(10)	1.002(14)	
C(11)-H(11A)	1.006(16)	
C(11)-H(11B)	1.015(15)	
C(11)-H(11C)	1.000(15)	
C(12)-C(17)	1.3869(15)	
C(12)-C(13)	1.4004(15)	
C(13)-C(14)	1.3818(17)	
C(13)-H(13)	0.948(14)	
C(14)-C(15)	1.3860(17)	
C(14)-H(14)	0.970(15)	
C(15)-O(15)	1.3745(14)	
C(15)-C(16)	1.3885(17)	
O(15)-C(18)	1.4248(17)	
C(16)-C(17)	1.3974(16)	
C(16)-H(16)	0.974(14)	
C(17)-H(17)	0.969(13)	

Table E78. continued...

C(18)-H(18A)	0.96(2)
C(18)-H(18B)	1.000(19)
C(18)-H(18C)	1.002(17)
C(20)-C(21)	1.504(2)
C(20)-H(20A)	0.970(18)
C(20)-H(20B)	1.075(16)
C(21)-H(21A)	0.98(2)
C(21)-H(21B)	1.027(18)
C(21)-H(21C)	1.01(2)
C(22)-C(23)	1.508(2)
C(22)-H(22A)	0.965(16)
C(22)-H(22B)	0.980(15)
C(23)-H(23A)	1.02(2)
C(23)-H(23B)	0.989(17)
C(23)-H(23C)	0.96(2)
C(24)-C(25)	1.5151(19)
C(24)-H(24A)	1.008(14)
C(24)-H(24B)	0.967(14)
C(25)-H(25A)	0.971(19)
C(25)-H(25B)	1.007(18)
C(25)-H(25C)	1.02(2)

O(1)-C(1)-C(3)#1	124.31(9)
O(1)-C(1)-C(2)	115.65(8)
C(3)#1-C(1)-C(2)	120.03(8)
C(24)-N(1)-C(22)	111.29(10)
C(24)-N(1)-C(20)	114.78(11)
C(22)-N(1)-C(20)	110.61(10)
C(24)-N(1)-H(1)	104.8(9)
C(22)-N(1)-H(1)	109.7(9)
C(20)-N(1)-H(1)	105.2(9)
O(2)-C(2)-C(3)	123.98(9)
O(2)-C(2)-C(1)	115.46(8)
C(3)-C(2)-C(1)	120.56(8)
C(2)-C(3)-C(1)#1	119.23(9)
C(2)-C(3)-C(4)	118.26(9)
C(1)#1-C(3)-C(4)	122.48(9)
C(3)-C(4)-C(12)	117.37(8)
C(3)-C(4)-C(5)	112.59(8)
C(12)-C(4)-C(5)	112.01(8)
C(3)-C(4)-H(4)	104.0(7)
C(12)-C(4)-H(4)	104.3(7)
C(5)-C(4)-H(4)	105.0(7)
C(6)-C(5)-C(10)	117.40(10)

Table E78. continued...

C(6)-C(5)-C(4)	122.68(9)
C(10)-C(5)-C(4)	119.90(9)
C(5)-C(6)-C(7)	121.80(10)
C(5)-C(6)-H(6)	119.2(8)
C(7)-C(6)-H(6)	119.0(8)
C(8)-C(7)-C(6)	119.51(10)
C(8)-C(7)-H(7)	121.1(8)
C(6)-C(7)-H(7)	119.4(8)
O(8)-C(8)-C(9)	115.85(10)
O(8)-C(8)-C(7)	124.28(10)
C(9)-C(8)-C(7)	119.87(10)
C(8)-O(8)-C(11)	116.94(9)
C(8)-C(9)-C(10)	119.64(10)
C(8)-C(9)-H(9)	117.9(8)
C(10)-C(9)-H(9)	122.4(8)
C(9)-C(10)-C(5)	121.77(10)
C(9)-C(10)-H(10)	118.9(8)
C(5)-C(10)-H(10)	119.3(8)
O(8)-C(11)-H(11A)	104.5(9)
O(8)-C(11)-H(11B)	110.5(8)
H(11A)-C(11)-H(11B)	110.7(12)
O(8)-C(11)-H(11C)	111.2(8)
H(11A)-C(11)-H(11C)	110.0(12)
H(11B)-C(11)-H(11C)	109.9(12)
C(17)-C(12)-C(13)	116.89(10)
C(17)-C(12)-C(4)	123.71(9)
C(13)-C(12)-C(4)	119.09(9)
C(14)-C(13)-C(12)	121.71(11)
C(14)-C(13)-H(13)	119.4(8)
C(12)-C(13)-H(13)	118.9(8)
C(13)-C(14)-C(15)	120.37(11)
C(13)-C(14)-H(14)	120.5(8)
C(15)-C(14)-H(14)	119.1(8)
O(15)-C(15)-C(14)	115.31(10)
O(15)-C(15)-C(16)	125.31(11)
C(14)-C(15)-C(16)	119.38(10)
C(15)-O(15)-C(18)	117.66(11)
C(15)-C(16)-C(17)	119.40(11)
C(15)-C(16)-H(16)	120.7(8)
C(17)-C(16)-H(16)	119.9(8)
C(12)-C(17)-C(16)	122.21(10)
C(12)-C(17)-H(17)	120.1(7)
C(16)-C(17)-H(17)	117.7(7)
O(15)-C(18)-H(18A)	102.9(11)

Table E78. continued...

O(15)-C(18)-H(18B)	108.7(10)
H(18A)-C(18)-H(18B)	116.1(15)
O(15)-C(18)-H(18C)	109.4(9)
H(18A)-C(18)-H(18C)	110.0(15)
H(18B)-C(18)-H(18C)	109.5(14)
N(1)-C(20)-C(21)	114.24(12)
N(1)-C(20)-H(20A)	105.7(10)
C(21)-C(20)-H(20A)	111.7(10)
N(1)-C(20)-H(20B)	105.3(9)
C(21)-C(20)-H(20B)	109.6(9)
H(20A)-C(20)-H(20B)	110.1(13)
C(20)-C(21)-H(21A)	108.2(11)
C(20)-C(21)-H(21B)	113.2(10)
H(21A)-C(21)-H(21B)	107.6(14)
C(20)-C(21)-H(21C)	114.9(12)
H(21A)-C(21)-H(21C)	108.0(17)
H(21B)-C(21)-H(21C)	104.6(15)
N(1)-C(22)-C(23)	112.88(11)
N(1)-C(22)-H(22A)	106.2(9)
C(23)-C(22)-H(22A)	112.0(9)
N(1)-C(22)-H(22B)	104.9(8)
C(23)-C(22)-H(22B)	110.2(9)
H(22A)-C(22)-H(22B)	110.3(12)
C(22)-C(23)-H(23A)	108.2(11)
C(22)-C(23)-H(23B)	109.6(10)
H(23A)-C(23)-H(23B)	108.1(14)
C(22)-C(23)-H(23C)	111.7(12)
H(23A)-C(23)-H(23C)	109.4(16)
H(23B)-C(23)-H(23C)	109.7(15)
N(1)-C(24)-C(25)	114.40(12)
N(1)-C(24)-H(24A)	106.2(8)
C(25)-C(24)-H(24A)	111.1(8)
N(1)-C(24)-H(24B)	105.2(8)
C(25)-C(24)-H(24B)	111.8(8)
H(24A)-C(24)-H(24B)	107.7(11)
C(24)-C(25)-H(25A)	106.5(11)
C(24)-C(25)-H(25B)	111.0(10)
H(25A)-C(25)-H(25B)	106.1(14)
C(24)-C(25)-H(25C)	112.9(10)
H(25A)-C(25)-H(25C)	108.9(15)
H(25B)-C(25)-H(25C)	111.0(14)

Symmetry transformations used to generate equivalent atoms:
#1 -x, -y, -z+1

Table E79. Anisotropic displacement parameters ($Å^2$ x 10^3) for **2D•($C_6H_{15}N)_2$**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+...+2hka^*b^*U_{12}]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(1)	28(1)	28(1)	21(1)	-1(1)	8(1)	0(1)
O(1)	31(1)	42(1)	23(1)	4(1)	9(1)	-6(1)
N(1)	32(1)	40(1)	35(1)	9(1)	15(1)	1(1)
C(2)	25(1)	27(1)	23(1)	-2(1)	7(1)	-1(1)
O(2)	25(1)	44(1)	27(1)	4(1)	8(1)	-5(1)
C(3)	26(1)	30(1)	21(1)	0(1)	6(1)	0(1)
C(4)	24(1)	34(1)	21(1)	0(1)	6(1)	-2(1)
C(5)	27(1)	31(1)	20(1)	2(1)	4(1)	0(1)
C(6)	32(1)	33(1)	31(1)	-3(1)	10(1)	-6(1)
C(7)	31(1)	37(1)	33(1)	-3(1)	12(1)	-3(1)
C(8)	33(1)	29(1)	24(1)	1(1)	6(1)	3(1)
O(8)	42(1)	34(1)	36(1)	-6(1)	15(1)	-1(1)
C(9)	35(1)	34(1)	31(1)	-2(1)	8(1)	-7(1)
C(10)	29(1)	39(1)	27(1)	-1(1)	9(1)	-6(1)
C(11)	45(1)	45(1)	42(1)	-6(1)	20(1)	2(1)
C(12)	24(1)	36(1)	24(1)	0(1)	7(1)	1(1)
C(13)	36(1)	37(1)	28(1)	-4(1)	1(1)	2(1)
C(14)	40(1)	44(1)	30(1)	1(1)	-3(1)	3(1)
C(15)	34(1)	37(1)	37(1)	5(1)	6(1)	3(1)
O(15)	59(1)	37(1)	48(1)	7(1)	-2(1)	4(1)
C(16)	35(1)	35(1)	34(1)	-3(1)	10(1)	-2(1)
C(17)	29(1)	38(1)	24(1)	-2(1)	7(1)	-1(1)
C(18)	63(1)	34(1)	59(1)	9(1)	10(1)	2(1)
C(20)	32(1)	63(1)	54(1)	19(1)	15(1)	4(1)
C(21)	44(1)	57(1)	62(1)	12(1)	-2(1)	-7(1)
C(22)	50(1)	54(1)	35(1)	7(1)	22(1)	2(1)
C(23)	59(1)	58(1)	55(1)	-6(1)	24(1)	7(1)
C(24)	39(1)	38(1)	44(1)	5(1)	9(1)	-1(1)
C(25)	62(1)	44(1)	51(1)	12(1)	4(1)	-13(1)

Table E80. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for **2D•$(C_6H_{15}N)_2$**.

Atom	x	y	z	U_{eq}	Occ.
H(1)	3150(15)	804(7)	3985(13)	45(4)	1
H(4)	2991(12)	141(6)	7138(10)	25(3)	1
H(6)	22(15)	41(7)	8459(11)	40(3)	1
H(7)	-432(15)	854(7)	9756(12)	41(3)	1
H(9)	2971(14)	1960(7)	9463(12)	40(3)	1
H(10)	3432(14)	1139(7)	8103(11)	38(3)	1
H(11A)	-4(17)	2303(8)	11553(14)	56(4)	1
H(11B)	13(15)	1460(8)	11480(13)	47(4)	1
H(11C)	-1001(16)	1910(7)	10420(13)	46(4)	1
H(13)	3518(14)	-292(7)	9503(12)	37(3)	1
H(14)	4417(15)	-1295(7)	10461(13)	46(4)	1
H(16)	2662(14)	-2362(7)	7496(12)	40(3)	1
H(17)	1726(13)	-1356(6)	6579(11)	32(3)	1
H(18A)	4650(20)	-3408(10)	9708(17)	78(6)	1
H(18B)	4489(19)	-3065(9)	8401(17)	66(5)	1
H(18C)	3121(18)	-3247(8)	8891(14)	54(4)	1
H(20A)	5674(18)	1170(8)	3642(15)	63(5)	1
H(20B)	5314(17)	419(8)	4168(14)	59(4)	1
H(21A)	6560(20)	1073(10)	5730(17)	78(6)	1
H(21B)	4969(18)	1084(9)	5888(15)	63(5)	1
H(21C)	5620(20)	1747(11)	5424(18)	90(6)	1
H(22A)	3945(16)	1048(8)	1957(13)	49(4)	1
H(22B)	2339(16)	964(7)	1977(12)	44(4)	1
H(23A)	3020(20)	-81(10)	1380(18)	82(6)	1
H(23B)	2828(17)	-164(8)	2700(14)	57(4)	1
H(23C)	4370(20)	-99(10)	2501(16)	80(6)	1
H(24A)	3296(14)	1884(7)	4483(12)	38(3)	1
H(24B)	2106(15)	1747(7)	3313(12)	39(3)	1
H(25A)	3363(19)	2731(10)	3109(15)	69(5)	1
H(25B)	3507(18)	2212(9)	2126(16)	66(5)	1
H(25C)	4840(20)	2325(9)	3305(16)	73(5)	1

Table E81. Torsion angles (°) for **2D•$(C_6H_{15}N)_2$**.

Atoms	Angle
O(1) C(1) C(2) O(2)	-5.25(13)
C(3)#1 C(1) C(2) O(2)	175.63(9)
O(1) C(1) C(2) C(3)	174.27(9)
C(3)#1 C(1) C(2) C(3)	-4.85(16)
O(2) C(2) C(3) C(1)#1	-175.72(9)
C(1) C(2) C(3) C(1)#1	4.80(16)
O(2) C(2) C(3) C(4)	6.26(15)
C(1) C(2) C(3) C(4)	-173.22(8)
C(2) C(3) C(4) C(12)	-121.05(10)
C(1)#1 C(3) C(4) C(12)	61.00(13)
C(2) C(3) C(4) C(5)	106.69(10)
C(1)#1 C(3) C(4) C(5)	-71.26(12)
C(3) C(4) C(5) C(6)	70.50(12)
C(12) C(4) C(5) C(6)	-64.36(12)
C(3) C(4) C(5) C(10)	-111.47(10)
C(12) C(4) C(5) C(10)	113.68(10)
C(10) C(5) C(6) C(7)	0.30(16)
C(4) C(5) C(6) C(7)	178.39(10)
C(5) C(6) C(7) C(8)	-0.65(17)
C(6) C(7) C(8) O(8)	-178.52(10)
C(6) C(7) C(8) C(9)	0.39(16)
C(9) C(8) O(8) C(11)	-178.95(10)
C(7) C(8) O(8) C(11)	0.00(15)
O(8) C(8) C(9) C(10)	179.20(10)
C(7) C(8) C(9) C(10)	0.20(16)
C(8) C(9) C(10) C(5)	-0.56(17)
C(6) C(5) C(10) C(9)	0.30(16)
C(4) C(5) C(10) C(9)	-177.84(10)
C(3) C(4) C(12) C(17)	17.01(14)
C(5) C(4) C(12) C(17)	149.53(10)
C(3) C(4) C(12) C(13)	-169.60(9)
C(5) C(4) C(12) C(13)	-37.07(13)
C(17) C(12) C(13) C(14)	0.85(17)
C(4) C(12) C(13) C(14)	-172.99(11)
C(12) C(13) C(14) C(15)	1.03(19)
C(13) C(14) C(15) O(15)	178.61(11)
C(13) C(14) C(15) C(16)	-2.09(19)
C(14) C(15) O(15) C(18)	-177.01(13)
C(16) C(15) O(15) C(18)	3.74(19)
O(15) C(15) C(16) C(17)	-179.53(11)
C(14) C(15) C(16) C(17)	1.25(17)

Table E81. continued...

C(13) C(12) C(17) C(16)	-1.70(16)
C(4) C(12) C(17) C(16)	171.83(10)
C(15) C(16) C(17) C(12)	0.68(17)
C(24) N(1) C(20) C(21)	49.16(15)
C(22) N(1) C(20) C(21)	176.09(12)
C(24) N(1) C(22) C(23)	-162.95(12)
C(20) N(1) C(22) C(23)	68.21(15)
C(22) N(1) C(24) C(25)	-62.01(14)
C(20) N(1) C(24) C(25)	64.57(14)

Symmetry transformations used to generate equivalent atoms:
#1 -x,-y,-z+1

Table E82. Hydrogen bonds, lengths (Å) and angles(°) **2D•$(C_6H_{15}N)_2$**.

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1)...O(2)	0.928(15)	1.793(15)	2.6862(13)	160.5(13)
N(1)-H(1)...O(1)	0.928(15)	2.340(14)	2.9081(13)	119.2(11)

Table E83. Crystal and structure refinement data for $2D^{2-}\bullet(Cs^{+})_2\bullet(CH_4O)_4$.

Identification code	$2D^{2-}\bullet(Cs^{+})_2\bullet(CH_4O)_4$
Empirical formula	$C_{40}H_{46}Cs_2O_{12}$
Formula weight	984.59 g/mole
Temperature	173(2) K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group	$P^{-}1$
Unit cell dimensions	a = 9.4464(19) Å b = 10.878(2) Å c = 11.065(2) Å α = 97.76(3)° β = 113.19(3)° γ = 99.02(3)°
Volume	1007.7(4) $Å^3$
Z	1
Density (calculated)	1.622 Mg/m^3
Absorption coefficient	1.869 mm^{-1}
F(000)	492
Crystal Size	0.44 mm x 0.20 mm x 0.14 mm
Color	red
Theta range for data collection	1.94° to 28.28°
Index ranges	$-11 \le h \le 11$, $-14 \le k \le 14$, $-14 \le l \le 14$
Reflections collected / Unique	11899 / 4642 [R_{int} = 0.0205]
Refinement method	Full-matrix-least-squares on F^2
Data / Restraints / Parameters	4642 / 0 / 328
Goodness-of-fit on F^2	0.996
Final R indices [$I>2\sigma(I)$]	R_1 = 0.0200, wR_2 = 0.0469
R indices (all data)	R_1 = 0.0247, wR_2 = 0.0478
Extinction Coefficient	0.0026(4)
Largest diff. peak and hole	0.568 and -0.371 e/A^3

Table E84. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for $2D^{2-}•(Cs^+)_2•(CH_4O)_4$. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U_{eq}	Occ.
Cs(1)	5628(1)	6779(1)	6414(1)	28(1)	1
O(1)	2052(2)	6784(1)	4640(1)	28(1)	1
C(1)	1025(2)	5998(2)	4794(2)	19(1)	1
O(2)	2736(1)	4568(1)	5042(1)	22(1)	1
C(2)	1440(2)	4701(2)	5031(2)	18(1)	1
C(3)	393(2)	3757(2)	5228(2)	18(1)	1
C(4)	818(2)	2505(2)	5536(2)	20(1)	1
C(5)	1195(2)	1745(2)	4477(2)	21(1)	1
C(6)	442(2)	1783(2)	3118(2)	26(1)	1
C(7)	636(2)	1004(2)	2121(2)	31(1)	1
O(8)	1727(2)	-595(2)	1424(2)	44(1)	1
C(8)	1625(2)	158(2)	2475(2)	30(1)	1
C(9)	2406(2)	113(2)	3812(2)	30(1)	1
C(10)	2179(2)	897(2)	4795(2)	27(1)	1
C(11)	2646(4)	-1517(3)	1758(3)	55(1)	1
C(12)	2050(2)	2701(2)	6986(2)	20(1)	1
C(13)	1569(2)	2405(2)	7972(2)	28(1)	1
C(14)	2631(3)	2600(2)	9305(2)	32(1)	1
O(15)	5200(2)	3289(2)	11047(1)	35(1)	1
C(15)	4227(2)	3118(2)	9699(2)	26(1)	1
C(16)	4741(2)	3425(2)	8742(2)	24(1)	1
C(17)	3654(2)	3208(2)	7402(2)	22(1)	1
C(18)	6846(3)	3787(2)	11454(2)	37(1)	1
O(30)	7139(2)	4426(2)	7313(2)	44(1)	1
C(30)	8629(4)	5026(3)	8335(3)	59(1)	1
O(40)	3976(2)	8955(2)	6822(2)	56(1)	1
C(40)	3733(4)	9484(3)	7960(4)	66(1)	1

Table E85. Bond lengths (Å) and angles (°) for $2D^{2-}\bullet(Cs^{+})_2\bullet(CH_4O)_4$.

Atoms	Length	Angle
Cs(1)-O(2)#1	3.0268(14)	
Cs(1)-O(2)	3.039(2)	
Cs(1)-O(40)	3.1091(19)	
Cs(1)-O(1)	3.1699(17)	
Cs(1)-O(15)#2	3.2054(16)	
Cs(1)-O(30)	3.2092(19)	
Cs(1)-C(7)#1	3.560(3)	
Cs(1)-C(6)#1	3.598(2)	
Cs(1)-C(8)#1	3.607(2)	
Cs(1)-C(9)#1	3.683(2)	
Cs(1)-C(5)#1	3.715(2)	
Cs(1)-C(10)#1	3.732(2)	
Cs(1)-H(30)	3.46(3)	
Cs(1)-H(40)	3.14(4)	
O(1)-C(1)	1.269(2)	
C(1)-C(3)#3	1.398(2)	
C(1)-C(2)	1.550(2)	
O(2)-C(2)	1.251(2)	
O(2)-Cs(1)#1	3.0268(14)	
C(2)-C(3)	1.412(2)	
C(3)-C(1)#3	1.398(2)	
C(3)-C(4)	1.524(2)	
C(4)-C(12)	1.528(3)	
C(4)-C(5)	1.531(2)	
C(4)-H(4)	0.97(2)	
C(5)-C(10)	1.392(2)	
C(5)-C(6)	1.397(3)	
C(5)-Cs(1)#1	3.715(2)	
C(6)-C(7)	1.387(3)	
C(6)-Cs(1)#1	3.598(2)	
C(6)-H(6)	0.92(2)	
C(7)-C(8)	1.397(3)	
C(7)-Cs(1)#1	3.560(3)	
C(7)-H(7)	0.95(2)	
O(8)-C(8)	1.373(2)	
O(8)-C(11)	1.421(3)	
C(8)-C(9)	1.380(3)	
C(8)-Cs(1)#1	3.607(2)	
C(9)-C(10)	1.396(3)	
C(9)-Cs(1)#1	3.683(2)	
C(9)-H(9)	0.95(2)	

Table E85. continued...

C(10)-Cs(1)#1	3.732(2)
C(10)-H(10)	0.92(2)
C(11)-H(11A)	0.85(4)
C(11)-H(11B)	0.97(3)
C(11)-H(11C)	0.96(3)
C(12)-C(17)	1.390(3)
C(12)-C(13)	1.393(3)
C(13)-C(14)	1.382(3)
C(13)-H(13)	0.94(3)
C(14)-C(15)	1.390(3)
C(14)-H(14)	0.92(3)
O(15)-C(15)	1.377(2)
O(15)-C(18)	1.426(3)
O(15)-Cs(1)#2	3.2054(16)
C(15)-C(16)	1.385(3)
C(16)-C(17)	1.393(3)
C(16)-H(16)	0.87(2)
C(17)-H(17)	0.93(2)
C(18)-H(18A)	0.88(3)
C(18)-H(18B)	0.96(2)
C(18)-H(18C)	0.98(3)
O(30)-C(30)	1.395(4)
O(30)-H(30)	0.80(3)
C(30)-H(30A)	0.94(3)
C(30)-H(30B)	0.93(4)
C(30)-H(30C)	0.89(3)
O(40)-C(40)	1.429(4)
O(40)-H(40)	0.96(4)
C(40)-H(40A)	0.9600
C(40)-H(40B)	0.9600
C(40)-H(40C)	0.9600

O(2)#1-Cs(1)-O(2)	89.55(4)
O(2)#1-Cs(1)-O(40)	150.60(5)
O(2)-Cs(1)-O(40)	99.18(5)
O(2)#1-Cs(1)-O(1)	115.82(4)
O(2)-Cs(1)-O(1)	50.31(4)
O(40)-Cs(1)-O(1)	54.84(5)
O(2)#1-Cs(1)-O(15)#2	140.18(4)
O(2)-Cs(1)-O(15)#2	83.09(5)
O(40)-Cs(1)-O(15)#2	69.09(5)
O(1)-Cs(1)-O(15)#2	88.87(4)
O(2)#1-Cs(1)-O(30)	57.32(4)
O(2)-Cs(1)-O(30)	78.69(5)

Table E85. continued...

O(40)-Cs(1)-O(30)	151.89(5)
O(1)-Cs(1)-O(30)	128.98(5)
O(15)#2-Cs(1)-O(30)	82.87(5)
O(2)#1-Cs(1)-C(7)#1	81.28(5)
O(2)-Cs(1)-C(7)#1	170.27(4)
O(40)-Cs(1)-C(7)#1	90.44(5)
O(1)-Cs(1)-C(7)#1	137.31(5)
O(15)#2-Cs(1)-C(7)#1	101.89(5)
O(30)-Cs(1)-C(7)#1	93.52(5)
O(2)#1-Cs(1)-C(6)#1	59.26(5)
O(2)-Cs(1)-C(6)#1	148.80(4)
O(40)-Cs(1)-C(6)#1	107.99(5)
O(1)-Cs(1)-C(6)#1	140.11(4)
O(15)#2-Cs(1)-C(6)#1	120.56(5)
O(30)-Cs(1)-C(6)#1	84.16(5)
C(7)#1-Cs(1)-C(6)#1	22.34(5)
O(2)#1-Cs(1)-C(8)#1	96.30(5)
O(2)-Cs(1)-C(8)#1	165.30(4)
O(40)-Cs(1)-C(8)#1	69.42(5)
O(1)-Cs(1)-C(8)#1	115.23(5)
O(15)#2-Cs(1)-C(8)#1	100.59(5)
O(30)-Cs(1)-C(8)#1	115.78(5)
C(7)#1-Cs(1)-C(8)#1	22.47(5)
C(6)#1-Cs(1)-C(8)#1	39.00(5)
O(2)#1-Cs(1)-C(9)#1	90.29(4)
O(2)-Cs(1)-C(9)#1	145.48(4)
O(40)-Cs(1)-C(9)#1	66.76(5)
O(1)-Cs(1)-C(9)#1	99.75(5)
O(15)#2-Cs(1)-C(9)#1	117.13(5)
O(30)-Cs(1)-C(9)#1	128.69(5)
C(7)#1-Cs(1)-C(9)#1	38.69(5)
C(6)#1-Cs(1)-C(9)#1	44.60(5)
C(8)#1-Cs(1)-C(9)#1	21.80(5)
O(2)#1-Cs(1)-C(5)#1	52.59(4)
O(2)-Cs(1)-C(5)#1	135.11(4)
O(40)-Cs(1)-C(5)#1	104.91(5)
O(1)-Cs(1)-C(5)#1	120.13(4)
O(15)#2-Cs(1)-C(5)#1	140.92(5)
O(30)-Cs(1)-C(5)#1	95.47(4)
C(7)#1-Cs(1)-C(5)#1	39.07(5)
C(6)#1-Cs(1)-C(5)#1	21.95(4)
C(8)#1-Cs(1)-C(5)#1	45.26(4)
C(9)#1-Cs(1)-C(5)#1	38.49(4)
O(2)#1-Cs(1)-C(10)#1	69.64(4)

Table E85. continued...

O(2)-Cs(1)-C(10)#1	134.57(4)
O(40)-Cs(1)-C(10)#1	84.66(5)
O(1)-Cs(1)-C(10)#1	102.34(5)
O(15)#2-Cs(1)-C(10)#1	138.12(5)
O(30)-Cs(1)-C(10)#1	117.01(4)
C(7)#1-Cs(1)-C(10)#1	44.49(5)
C(6)#1-Cs(1)-C(10)#1	37.77(5)
C(8)#1-Cs(1)-C(10)#1	38.14(5)
C(9)#1-Cs(1)-C(10)#1	21.68(4)
C(5)#1-Cs(1)-C(10)#1	21.55(4)
O(2)#1-Cs(1)-H(30)	44.5(5)
O(2)-Cs(1)-H(30)	77.7(5)
O(40)-Cs(1)-H(30)	164.9(5)
O(1)-Cs(1)-H(30)	126.9(5)
O(15)#2-Cs(1)-H(30)	95.8(5)
O(30)-Cs(1)-H(30)	13.2(5)
C(7)#1-Cs(1)-H(30)	93.4(5)
C(6)#1-Cs(1)-H(30)	79.7(5)
C(8)#1-Cs(1)-H(30)	115.7(5)
C(9)#1-Cs(1)-H(30)	123.6(5)
C(5)#1-Cs(1)-H(30)	87.1(5)
C(10)#1-Cs(1)-H(30)	108.2(5)
O(2)#1-Cs(1)-H(40)	145.6(7)
O(2)-Cs(1)-H(40)	82.4(8)
O(40)-Cs(1)-H(40)	17.6(8)
O(1)-Cs(1)-H(40)	37.4(8)
O(15)#2-Cs(1)-H(40)	72.1(7)
O(30)-Cs(1)-H(40)	150.2(8)
C(7)#1-Cs(1)-H(40)	107.0(8)
C(6)#1-Cs(1)-H(40)	122.3(8)
C(8)#1-Cs(1)-H(40)	85.2(8)
C(9)#1-Cs(1)-H(40)	78.4(8)
C(5)#1-Cs(1)-H(40)	114.1(8)
C(10)#1-Cs(1)-H(40)	92.6(8)
H(30)-Cs(1)-H(40)	157.9(9)
C(1)-O(1)-Cs(1)	115.55(11)
O(1)-C(1)-C(3)#3	124.27(16)
O(1)-C(1)-C(2)	115.44(15)
C(3)#3-C(1)-C(2)	120.29(15)
O(1)-C(1)-Cs(1)	47.33(9)
C(3)#3-C(1)-Cs(1)	150.22(12)
C(2)-C(1)-Cs(1)	75.89(9)
C(2)-O(2)-Cs(1)#1	146.07(11)
C(2)-O(2)-Cs(1)	119.88(11)

Table E85. continued...

Cs(1)#1-O(2)-Cs(1)	90.45(4)
O(2)-C(2)-C(3)	123.66(15)
O(2)-C(2)-C(1)	115.90(15)
C(3)-C(2)-C(1)	120.44(15)
O(2)-C(2)-Cs(1)	43.62(9)
C(3)-C(2)-Cs(1)	146.76(11)
C(1)-C(2)-Cs(1)	80.93(9)
C(1)#3-C(3)-C(2)	119.27(15)
C(1)#3-C(3)-C(4)	119.42(15)
C(2)-C(3)-C(4)	121.23(15)
C(3)-C(4)-C(12)	110.99(14)
C(3)-C(4)-C(5)	114.22(15)
C(12)-C(4)-C(5)	114.77(14)
C(3)-C(4)-H(4)	105.5(12)
C(12)-C(4)-H(4)	105.1(12)
C(5)-C(4)-H(4)	105.2(12)
C(10)-C(5)-C(6)	116.85(17)
C(10)-C(5)-C(4)	121.84(16)
C(6)-C(5)-C(4)	121.03(16)
C(10)-C(5)-Cs(1)#1	79.94(11)
C(6)-C(5)-Cs(1)#1	74.31(11)
C(4)-C(5)-Cs(1)#1	120.25(11)
C(7)-C(6)-C(5)	122.18(18)
C(7)-C(6)-Cs(1)#1	77.29(12)
C(5)-C(6)-Cs(1)#1	83.74(12)
C(7)-C(6)-H(6)	118.2(13)
C(5)-C(6)-H(6)	119.6(13)
Cs(1)#1-C(6)-H(6)	107.8(13)
C(6)-C(7)-C(8)	119.50(19)
C(6)-C(7)-Cs(1)#1	80.38(12)
C(8)-C(7)-Cs(1)#1	80.68(12)
C(6)-C(7)-H(7)	122.4(13)
C(8)-C(7)-H(7)	118.0(13)
Cs(1)#1-C(7)-H(7)	106.4(13)
C(8)-O(8)-C(11)	116.5(2)
O(8)-C(8)-C(9)	124.54(18)
O(8)-C(8)-C(7)	115.77(18)
C(9)-C(8)-C(7)	119.69(18)
O(8)-C(8)-Cs(1)#1	111.62(13)
C(9)-C(8)-Cs(1)#1	82.18(12)
C(7)-C(8)-Cs(1)#1	76.84(12)
C(8)-C(9)-C(10)	119.75(18)
C(8)-C(9)-Cs(1)#1	76.02(12)
C(10)-C(9)-Cs(1)#1	81.15(12)

Table E85. continued...

C(8)-C(9)-H(9)	121.1(14)
C(10)-C(9)-H(9)	119.1(14)
Cs(1)#1-C(9)-H(9)	113.4(14)
C(5)-C(10)-C(9)	122.03(19)
C(5)-C(10)-Cs(1)#1	78.51(11)
C(9)-C(10)-Cs(1)#1	77.16(12)
C(5)-C(10)-H(10)	118.9(13)
C(9)-C(10)-H(10)	119.1(13)
Cs(1)#1-C(10)-H(10)	118.0(13)
O(8)-C(11)-H(11A)	106(2)
O(8)-C(11)-H(11B)	112.3(18)
H(11A)-C(11)-H(11B)	111(3)
O(8)-C(11)-H(11C)	112.8(18)
H(11A)-C(11)-H(11C)	109(3)
H(11B)-C(11)-H(11C)	106(3)
C(17)-C(12)-C(13)	117.01(17)
C(17)-C(12)-C(4)	123.31(16)
C(13)-C(12)-C(4)	119.65(16)
C(14)-C(13)-C(12)	121.86(18)
C(14)-C(13)-H(13)	121.0(15)
C(12)-C(13)-H(13)	117.0(15)
C(13)-C(14)-C(15)	120.12(18)
C(13)-C(14)-H(14)	121.0(16)
C(15)-C(14)-H(14)	118.9(16)
C(15)-O(15)-C(18)	116.64(16)
C(15)-O(15)-Cs(1)#2	130.73(12)
C(18)-O(15)-Cs(1)#2	111.68(12)
O(15)-C(15)-C(16)	124.39(18)
O(15)-C(15)-C(14)	116.27(17)
C(16)-C(15)-C(14)	119.35(17)
C(15)-C(16)-C(17)	119.60(18)
C(15)-C(16)-H(16)	122.0(13)
C(17)-C(16)-H(16)	118.4(13)
C(12)-C(17)-C(16)	122.05(17)
C(12)-C(17)-H(17)	119.8(12)
C(16)-C(17)-H(17)	118.2(13)
O(15)-C(18)-H(18A)	107.0(17)
O(15)-C(18)-H(18B)	110.6(13)
H(18A)-C(18)-H(18B)	108(2)
O(15)-C(18)-H(18C)	109.9(15)
H(18A)-C(18)-H(18C)	108(2)
H(18B)-C(18)-H(18C)	114(2)
C(30)-O(30)-Cs(1)	102.88(18)
C(30)-O(30)-H(30)	105(2)

Table E85. continued...

Cs(1)-O(30)-H(30)	101.5(19)
O(30)-C(30)-Cs(1)	56.01(15)
O(30)-C(30)-H(30A)	111(2)
Cs(1)-C(30)-H(30A)	95(2)
O(30)-C(30)-H(30B)	113(2)
Cs(1)-C(30)-H(30B)	70(2)
H(30A)-C(30)-H(30B)	109(3)
O(30)-C(30)-H(30C)	116(2)
Cs(1)-C(30)-H(30C)	161(2)
H(30A)-C(30)-H(30C)	104(3)
H(30B)-C(30)-H(30C)	103(3)
C(40)-O(40)-Cs(1)	130.58(18)
C(40)-O(40)-H(40)	112(2)
Cs(1)-O(40)-H(40)	83(2)
O(40)-C(40)-H(40A)	109.5
O(40)-C(40)-H(40B)	109.5
H(40A)-C(40)-H(40B)	109.5
O(40)-C(40)-H(40C)	109.5
H(40A)-C(40)-H(40C)	109.5
H(40B)-C(40)-H(40C)	109.5

Symmetry transformations used to generate equivalent atoms:

#1 -x+1, -y+1, -z+1

#2 -x+1, -y+1, -z+2

#3 -x, -y+1, -z+1

Table E86. Anisotropic displacement parameters ($Å^2 \times 10^3$) for $2D^{2-} \cdot (Cs^+)_2 \cdot (CH_4O)_4$. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+...+2hka^*b^*U_{12}]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Cs(1)	25(1)	27(1)	33(1)	2(1)	15(1)	5(1)
O(1)	23(1)	24(1)	45(1)	14(1)	18(1)	8(1)
C(1)	18(1)	18(1)	18(1)	3(1)	6(1)	5(1)
O(2)	19(1)	23(1)	28(1)	6(1)	12(1)	9(1)
C(2)	19(1)	19(1)	15(1)	2(1)	6(1)	7(1)
C(3)	18(1)	16(1)	19(1)	3(1)	6(1)	6(1)
C(4)	17(1)	17(1)	25(1)	5(1)	8(1)	5(1)
C(5)	20(1)	16(1)	25(1)	1(1)	7(1)	3(1)
C(6)	25(1)	22(1)	27(1)	2(1)	6(1)	9(1)
C(7)	33(1)	28(1)	24(1)	0(1)	7(1)	7(1)
O(8)	55(1)	36(1)	38(1)	-6(1)	21(1)	17(1)
C(8)	32(1)	21(1)	33(1)	-5(1)	15(1)	3(1)
C(9)	32(1)	20(1)	40(1)	4(1)	15(1)	12(1)
C(10)	29(1)	22(1)	28(1)	5(1)	9(1)	10(1)
C(11)	63(2)	44(2)	59(2)	-8(1)	29(2)	24(1)
C(12)	23(1)	17(1)	24(1)	6(1)	11(1)	8(1)
C(13)	25(1)	28(1)	33(1)	8(1)	15(1)	5(1)
C(14)	39(1)	39(1)	28(1)	12(1)	20(1)	10(1)
O(15)	39(1)	44(1)	20(1)	7(1)	10(1)	12(1)
C(15)	33(1)	26(1)	20(1)	6(1)	11(1)	12(1)
C(16)	21(1)	25(1)	26(1)	6(1)	9(1)	8(1)
C(17)	24(1)	24(1)	23(1)	9(1)	12(1)	9(1)
C(18)	38(1)	42(1)	22(1)	1(1)	4(1)	17(1)
O(30)	44(1)	62(1)	43(1)	22(1)	29(1)	21(1)
C(30)	66(2)	60(2)	46(2)	7(1)	18(2)	19(2)
O(40)	44(1)	41(1)	64(1)	-4(1)	8(1)	15(1)
C(40)	77(2)	44(2)	92(2)	19(2)	51(2)	15(2)

Table E87. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for $2D^{2-}•(Cs^+)_2•(CH_4O)_4$.

Atom	x	y	z	U_{eq}	Occ.
H(4)	-130(20)	1978(19)	5500(20)	22(5)	1
H(6)	-170(20)	2360(20)	2870(20)	23(5)	1
H(7)	160(20)	1050(20)	1200(20)	26(5)	1
H(9)	3090(30)	-440(20)	4080(20)	36(6)	1
H(10)	2660(20)	828(19)	5680(20)	23(5)	1
H(11A)	2590(40)	-1900(30)	1020(40)	81(11)	1
H(11B)	2260(40)	-2110(30)	2210(30)	66(9)	1
H(11C)	3740(40)	-1140(30)	2350(30)	63(9)	1
H(13)	480(30)	2110(20)	7700(20)	42(7)	1
H(14)	2300(30)	2400(20)	9950(30)	42(7)	1
H(16)	5730(20)	3767(18)	8953(19)	16(5)	1
H(17)	4030(20)	3424(19)	6770(20)	20(5)	1
H(18A)	7330(30)	3790(20)	12320(30)	49(7)	1
H(18B)	7040(30)	4650(20)	11350(20)	31(6)	1
H(18C)	7250(30)	3220(20)	10970(20)	43(7)	1
H(30)	7300(30)	4160(30)	6680(30)	47(8)	1
H(30A)	8570(40)	5280(30)	9160(40)	77(10)	1
H(30B)	9130(40)	5730(40)	8140(40)	89(12)	1
H(30C)	9340(40)	4550(30)	8530(30)	75(10)	1
H(40)	3050(50)	8360(40)	6150(40)	115(14)	1
H(40A)	2968	9995	7692	260(40)	1
H(40B)	4716	10005	8644	190(30)	1
H(40C)	3354	8807	8308	144(18)	1

Table E88. Torsion angles (°) for $2D^{2-}\cdot(Cs^{+})_2\cdot(CH_4O)_4$.

Atoms	Angle
O(2)#1 Cs(1) O(1) C(1)	102.68(12)
O(2) Cs(1) O(1) C(1)	37.08(11)
O(40) Cs(1) O(1) C(1)	-109.86(13)
O(15)#2 Cs(1) O(1) C(1)	-44.87(12)
O(30) Cs(1) O(1) C(1)	35.02(14)
C(7)#1 Cs(1) O(1) C(1)	-151.29(12)
C(6)#1 Cs(1) O(1) C(1)	174.83(11)
C(8)#1 Cs(1) O(1) C(1)	-146.05(12)
C(9)#1 Cs(1) O(1) C(1)	-162.22(12)
C(5)#1 Cs(1) O(1) C(1)	162.72(11)
C(10)#1 Cs(1) O(1) C(1)	175.84(12)
Cs(1) O(1) C(1) C(3)#3	143.16(14)
Cs(1) O(1) C(1) C(2)	-36.31(18)
O(2)#1 Cs(1) C(1) O(1)	-86.05(12)
O(2) Cs(1) C(1) O(1)	-130.68(14)
O(40) Cs(1) C(1) O(1)	60.50(12)
O(15)#2 Cs(1) C(1) O(1)	133.59(13)
O(30) Cs(1) C(1) O(1)	-150.66(12)
C(7)#1 Cs(1) C(1) O(1)	42.79(16)
C(6)#1 Cs(1) C(1) O(1)	-8.54(18)
C(8)#1 Cs(1) C(1) O(1)	40.49(14)
C(9)#1 Cs(1) C(1) O(1)	19.56(13)
C(5)#1 Cs(1) C(1) O(1)	-21.83(14)
C(10)#1 Cs(1) C(1) O(1)	-4.66(13)
O(2)#1 Cs(1) C(1) C(3)#3	-172.1(2)
O(2) Cs(1) C(1) C(3)#3	143.3(3)
O(40) Cs(1) C(1) C(3)#3	-25.5(2)
O(1) Cs(1) C(1) C(3)#3	-86.0(3)
O(15)#2 Cs(1) C(1) C(3)#3	47.5(2)
O(30) Cs(1) C(1) C(3)#3	123.3(2)
C(7)#1 Cs(1) C(1) C(3)#3	-43.3(3)
C(6)#1 Cs(1) C(1) C(3)#3	-94.6(2)
C(8)#1 Cs(1) C(1) C(3)#3	-45.6(2)
C(9)#1 Cs(1) C(1) C(3)#3	-66.5(2)
C(5)#1 Cs(1) C(1) C(3)#3	-107.9(2)
C(10)#1 Cs(1) C(1) C(3)#3	-90.7(2)
O(2)#1 Cs(1) C(1) C(2)	60.49(10)
O(2) Cs(1) C(1) C(2)	15.86(8)
O(40) Cs(1) C(1) C(2)	-152.96(11)
O(1) Cs(1) C(1) C(2)	146.54(17)
O(15)#2 Cs(1) C(1) C(2)	-79.88(9)

Table E88. continued...

O(30) Cs(1) C(1) C(2)	-4.12(10)
C(7)#1 Cs(1) C(1) C(2)	-170.67(9)
C(6)#1 Cs(1) C(1) C(2)	138.00(11)
C(8)#1 Cs(1) C(1) C(2)	-172.98(8)
C(9)#1 Cs(1) C(1) C(2)	166.10(9)
C(5)#1 Cs(1) C(1) C(2)	124.71(9)
C(10)#1 Cs(1) C(1) C(2)	141.87(9)
O(2)#1 Cs(1) O(2) C(2)	-164.04(13)
O(40) Cs(1) O(2) C(2)	-12.25(13)
O(1) Cs(1) O(2) C(2)	-39.11(11)
O(15)#2 Cs(1) O(2) C(2)	55.18(12)
O(30) Cs(1) O(2) C(2)	139.26(12)
C(7)#1 Cs(1) O(2) C(2)	176.6(2)
C(6)#1 Cs(1) O(2) C(2)	-162.78(11)
C(8)#1 Cs(1) O(2) C(2)	-50.3(2)
C(9)#1 Cs(1) O(2) C(2)	-74.19(14)
C(5)#1 Cs(1) O(2) C(2)	-134.35(12)
C(10)#1 Cs(1) O(2) C(2)	-103.79(13)
O(2)#1 Cs(1) O(2) Cs(1)#1	0.0
O(40) Cs(1) O(2) Cs(1)#1	151.79(5)
O(1) Cs(1) O(2) Cs(1)#1	124.93(5)
O(15)#2 Cs(1) O(2) Cs(1)#1	-140.77(4)
O(30) Cs(1) O(2) Cs(1)#1	-56.70(4)
C(7)#1 Cs(1) O(2) Cs(1)#1	-19.4(3)
C(6)#1 Cs(1) O(2) Cs(1)#1	1.27(8)
C(8)#1 Cs(1) O(2) Cs(1)#1	113.71(15)
C(9)#1 Cs(1) O(2) Cs(1)#1	89.85(7)
C(5)#1 Cs(1) O(2) Cs(1)#1	29.69(6)
C(10)#1 Cs(1) O(2) Cs(1)#1	60.25(6)
Cs(1)#1 O(2) C(2) C(3)	70.2(3)
Cs(1) O(2) C(2) C(3)	-139.27(14)
Cs(1)#1 O(2) C(2) C(1)	-110.26(19)
Cs(1) O(2) C(2) C(1)	40.24(18)
Cs(1)#1 O(2) C(2) Cs(1)	-150.5(2)
O(1) C(1) C(2) O(2)	-0.2(2)
C(3)#3 C(1) C(2) O(2)	-179.65(15)
Cs(1) C(1) C(2) O(2)	-26.83(13)
O(1) C(1) C(2) C(3)	179.38(16)
C(3)#3 C(1) C(2) C(3)	-0.1(3)
Cs(1) C(1) C(2) C(3)	152.70(15)
O(1) C(1) C(2) Cs(1)	26.68(14)
C(3)#3 C(1) C(2) Cs(1)	-152.82(15)
O(2)#1 Cs(1) C(2) O(2)	16.57(14)
O(40) Cs(1) C(2) O(2)	167.81(13)

Table E88. continued...

O(1) Cs(1) C(2) O(2)	128.86(14)
O(15)#2 Cs(1) C(2) O(2)	-122.00(13)
O(30) Cs(1) C(2) O(2)	-39.81(12)
C(7)#1 Cs(1) C(2) O(2)	-175.1(3)
C(6)#1 Cs(1) C(2) O(2)	33.5(2)
C(8)#1 Cs(1) C(2) O(2)	155.57(12)
C(9)#1 Cs(1) C(2) O(2)	125.02(12)
C(5)#1 Cs(1) C(2) O(2)	61.69(14)
C(10)#1 Cs(1) C(2) O(2)	93.02(13)
O(2)#1 Cs(1) C(2) C(3)	98.8(2)
O(2) Cs(1) C(2) C(3)	82.2(2)
O(40) Cs(1) C(2) C(3)	-110.0(2)
O(1) Cs(1) C(2) C(3)	-148.9(2)
O(15)#2 Cs(1) C(2) C(3)	-39.8(2)
O(30) Cs(1) C(2) C(3)	42.4(2)
C(7)#1 Cs(1) C(2) C(3)	-92.9(4)
C(6)#1 Cs(1) C(2) C(3)	115.7(2)
C(8)#1 Cs(1) C(2) C(3)	-122.2(2)
C(9)#1 Cs(1) C(2) C(3)	-152.76(19)
C(5)#1 Cs(1) C(2) C(3)	143.91(19)
C(10)#1 Cs(1) C(2) C(3)	175.24(18)
O(2)#1 Cs(1) C(2) C(1)	-127.38(9)
O(2) Cs(1) C(2) C(1)	-143.95(17)
O(40) Cs(1) C(2) C(1)	23.86(9)
O(1) Cs(1) C(2) C(1)	-15.09(8)
O(15)#2 Cs(1) C(2) C(1)	94.05(9)
O(30) Cs(1) C(2) C(1)	176.24(9)
C(7)#1 Cs(1) C(2) C(1)	40.9(4)
C(6)#1 Cs(1) C(2) C(1)	-110.43(15)
C(8)#1 Cs(1) C(2) C(1)	11.62(14)
C(9)#1 Cs(1) C(2) C(1)	-18.93(12)
C(5)#1 Cs(1) C(2) C(1)	-82.26(11)
C(10)#1 Cs(1) C(2) C(1)	-50.93(11)
O(2) C(2) C(3) C(1)#3	179.61(16)
C(1) C(2) C(3) C(1)#3	0.1(3)
Cs(1) C(2) C(3) C(1)#3	124.40(18)
O(2) C(2) C(3) C(4)	2.8(3)
C(1) C(2) C(3) C(4)	-176.71(15)
Cs(1) C(2) C(3) C(4)	-52.4(3)
C(1)#3 C(3) C(4) C(12)	-102.27(18)
C(2) C(3) C(4) C(12)	74.6(2)
C(1)#3 C(3) C(4) C(5)	126.07(17)
C(2) C(3) C(4) C(5)	-57.1(2)
C(3) C(4) C(5) C(10)	153.84(17)

Table E88. continued...

C(12) C(4) C(5) C(10)	24.0(2)
C(3) C(4) C(5) C(6)	-32.5(2)
C(12) C(4) C(5) C(6)	-162.27(16)
C(3) C(4) C(5) Cs(1)#1	56.70(17)
C(12) C(4) C(5) Cs(1)#1	-73.10(16)
C(10) C(5) C(6) C(7)	0.9(3)
C(4) C(5) C(6) C(7)	-173.10(18)
Cs(1)#1 C(5) C(6) C(7)	70.69(18)
C(10) C(5) C(6) Cs(1)#1	-69.79(16)
C(4) C(5) C(6) Cs(1)#1	116.21(15)
C(5) C(6) C(7) C(8)	-0.6(3)
Cs(1)#1 C(6) C(7) C(8)	73.47(18)
C(5) C(6) C(7) Cs(1)#1	-74.09(18)
C(11) O(8) C(8) C(9)	3.2(3)
C(11) O(8) C(8) C(7)	-176.0(2)
C(11) O(8) C(8) Cs(1)#1	98.6(2)
C(6) C(7) C(8) O(8)	178.80(18)
Cs(1)#1 C(7) C(8) O(8)	-107.90(17)
C(6) C(7) C(8) C(9)	-0.4(3)
Cs(1)#1 C(7) C(8) C(9)	72.90(18)
C(6) C(7) C(8) Cs(1)#1	-73.30(18)
O(8) C(8) C(9) C(10)	-178.04(19)
C(7) C(8) C(9) C(10)	1.1(3)
Cs(1)#1 C(8) C(9) C(10)	71.05(18)
O(8) C(8) C(9) Cs(1)#1	110.9(2)
C(7) C(8) C(9) Cs(1)#1	-69.96(18)
C(6) C(5) C(10) C(9)	-0.2(3)
C(4) C(5) C(10) C(9)	173.76(18)
Cs(1)#1 C(5) C(10) C(9)	-66.76(19)
C(6) C(5) C(10) Cs(1)#1	66.57(16)
C(4) C(5) C(10) Cs(1)#1	-119.48(16)
C(8) C(9) C(10) C(5)	-0.8(3)
Cs(1)#1 C(9) C(10) C(5)	67.44(18)
C(8) C(9) C(10) Cs(1)#1	-68.25(18)
C(3) C(4) C(12) C(17)	-78.5(2)
C(5) C(4) C(12) C(17)	52.9(2)
C(3) C(4) C(12) C(13)	99.35(19)
C(5) C(4) C(12) C(13)	-129.28(17)
C(17) C(12) C(13) C(14)	-0.1(3)
C(4) C(12) C(13) C(14)	-178.11(18)
C(12) C(13) C(14) C(15)	0.7(3)
C(18) O(15) C(15) C(16)	-1.6(3)
Cs(1)#2 O(15) C(15) C(16)	166.23(13)
C(18) O(15) C(15) C(14)	178.29(18)

Table E88. continued...

Cs(1)#2 O(15) C(15) C(14)	-13.9(2)
C(13) C(14) C(15) O(15)	179.51(18)
C(13) C(14) C(15) C(16)	-0.6(3)
O(15) C(15) C(16) C(17)	179.75(17)
C(14) C(15) C(16) C(17)	-0.1(3)
C(13) C(12) C(17) C(16)	-0.6(3)
C(4) C(12) C(17) C(16)	177.27(16)
C(15) C(16) C(17) C(12)	0.8(3)
O(2)#1 Cs(1) O(30) C(30)	96.26(18)
O(2) Cs(1) O(30) C(30)	-166.93(18)
O(40) Cs(1) O(30) C(30)	-78.6(2)
O(1) Cs(1) O(30) C(30)	-165.31(17)
O(15)#2 Cs(1) O(30) C(30)	-82.59(18)
C(7)#1 Cs(1) O(30) C(30)	18.96(18)
C(6)#1 Cs(1) O(30) C(30)	39.27(18)
C(8)#1 Cs(1) O(30) C(30)	15.76(19)
C(9)#1 Cs(1) O(30) C(30)	36.66(19)
C(5)#1 Cs(1) O(30) C(30)	58.11(18)
C(10)#1 Cs(1) O(30) C(30)	58.53(19)
O(2)#1 Cs(1) C(30) O(30)	-71.49(17)
O(2) Cs(1) C(30) O(30)	12.99(18)
O(40) Cs(1) C(30) O(30)	131.24(15)
O(1) Cs(1) C(30) O(30)	22.5(3)
O(15)#2 Cs(1) C(30) O(30)	94.34(18)
C(7)#1 Cs(1) C(30) O(30)	-160.23(19)
C(6)#1 Cs(1) C(30) O(30)	-137.27(19)
C(8)#1 Cs(1) C(30) O(30)	-165.78(17)
C(9)#1 Cs(1) C(30) O(30)	-150.07(16)
C(5)#1 Cs(1) C(30) O(30)	-121.84(18)
C(10)#1 Cs(1) C(30) O(30)	-128.18(17)
O(2)#1 Cs(1) O(40) C(40)	-158.8(2)
O(2) Cs(1) O(40) C(40)	95.5(2)
O(1) Cs(1) O(40) C(40)	120.7(2)
O(15)#2 Cs(1) O(40) C(40)	16.6(2)
O(30) Cs(1) O(40) C(40)	12.4(3)
C(7)#1 Cs(1) O(40) C(40)	-85.9(2)
C(6)#1 Cs(1) O(40) C(40)	-100.0(2)
C(8)#1 Cs(1) O(40) C(40)	-94.1(2)
C(9)#1 Cs(1) O(40) C(40)	-117.4(2)
C(5)#1 Cs(1) O(40) C(40)	-122.7(2)
C(10)#1 Cs(1) O(40) C(40)	-130.1(2)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1, -y+1, -z+1 #2 -x+1, -y+1, -z+2 #3 -x,-y+1,-z+1

Table E89. Hydrogen bonds, lengths (Å) and angles (°) for $2D^{2-}\bullet(Cs^{+})_2\bullet(CH_4O)_4$.

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(30)-H(30)...O(1)#1	0.80(3)	2.01(3)	2.799(2)	166(3)
O(30)-H(30)...O(2)#1	0.80(3)	2.49(3)	2.995(2)	122(2)
O(40)-H(40)...O(1)	0.96(4)	2.02(4)	2.892(3)	150(3)

Symmetry transformations used to generate equivalent atoms:
#1 -x+1,-y+1,-z+1

Table E90. Crystal and structure refinement data for $2D^{2-}\bullet(Na^{+})_2\bullet(C_4H_8O)_4$.

Identification code	$2D^{2-}\bullet(Na^{+})_2\bullet(C_4H_8O)_4$
Empirical formula	$C_{52}H_{62}Na_2O_{12}$
Formula weight	925.00 g/mole
Temperature	173(2) K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group	$P\bar{1}$
Unit cell dimensions	a = 8.5552(17) Å b = 11.555(2) Å c = 13.216(3) Å α = 110.14(3)° β = 102.53(3)° γ = 96.15(3)°
Volume	1173.6(4) $Å^3$
Z	1
Density (calculated)	1.309 Mg/m^3
Absorption coefficient	0.107 mm^{-1}
F(000)	492
Crystal Size	0.32 mm x 0.20 mm x 0.10 mm
Color	red
Theta range for data collection	1.71° to 28.33°
Index ranges	$-11 \le h \le 11, -15 \le k \le 15, -17 \le l \le 17$
Reflections collected / Unique	14261 / 5557 [R_{int} = 0.0685]
Refinement method	Full-matrix-least-squares on F^2
Data / Restraints / Parameters	5557 / 0 / 359
Goodness-of-fit on F^2	0.975
Final R indices [$I>2\sigma(I)$]	R_1 = 0.0634, wR_2 = 0.1441
R indices (all data)	R_1 = 0.1619, wR_2 = 0.1757
Extinction Coefficient	0.007(3)
Largest diff. peak and hole	0.290 and -0.340 e/A^3

Table E91. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for $2D^{2-}•(Na^+)_2•(C_4H_8O)_4$. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U_{eq}	Occ.
Na(1)	275(1)	3704(1)	119(1)	36(1)	1
O(1)	-2655(2)	6859(2)	214(2)	34(1)	1
C(1)	-3795(3)	6019(2)	119(2)	24(1)	1
O(2)	-1847(2)	4701(2)	171(2)	30(1)	1
C(2)	-3355(3)	4755(2)	79(2)	24(1)	1
C(3)	-4581(3)	3776(2)	-56(2)	24(1)	1
C(4)	-4304(3)	2481(2)	-100(2)	24(1)	1
C(5)	-3079(3)	1971(2)	-735(2)	24(1)	1
C(6)	-3226(3)	1983(3)	-1800(3)	32(1)	1
C(7)	-2186(4)	1496(3)	-2419(3)	36(1)	1
O(8)	10(2)	505(2)	-2691(2)	42(1)	1
C(8)	-947(3)	961(3)	-1998(2)	31(1)	1
C(9)	-781(3)	925(3)	-950(2)	28(1)	1
C(10)	-1840(3)	1433(2)	-326(3)	26(1)	1
C(11)	1405(4)	67(4)	-2241(4)	44(1)	1
C(12)	-4045(3)	2386(2)	1034(2)	25(1)	1
C(13)	-2843(4)	3218(3)	1971(3)	33(1)	1
C(14)	-2645(4)	3138(3)	3001(3)	39(1)	1
O(15)	-3371(3)	2196(2)	4188(2)	53(1)	1
C(15)	-3666(4)	2190(3)	3123(3)	36(1)	1
C(16)	-4848(4)	1360(3)	2210(3)	36(1)	1
C(17)	-5028(4)	1454(3)	1181(3)	31(1)	1
C(18)	-4421(6)	1264(5)	4341(4)	64(1)	1
O(30)	1306(3)	4106(2)	2028(2)	58(1)	1
C(31)	2589(7)	5208(5)	2661(4)	105(2)	1
C(32A)	3779(15)	4910(8)	3520(8)	84(3)	0.66(2)
C(32B)	2990(30)	5151(15)	3772(14)	87(7)	0.34(2)
C(33)	2800(7)	3867(5)	3601(4)	116(2)	1
C(34)	1669(5)	3195(4)	2469(4)	74(1)	1
O(40)	-6726(4)	2209(4)	-3766(3)	112(1)	1
C(41)	-7218(7)	3216(6)	-3064(4)	119(2)	1
C(42)	-8772(7)	3287(5)	-3537(6)	149(3)	1
C(43)	-9414(6)	2138(5)	-4574(5)	105(2)	1
C(44)	-8114(5)	1430(4)	-4580(4)	84(1)	1

Table E92. Bond lengths (Å) and angles (°) for $2D^{2-}\bullet(Na^{+})_2\bullet(C_4H_8O)_4$.

Atoms	Length	Angle
Na(1)-O(2)	2.252(2)	
Na(1)-O(1)#1	2.289(2)	
Na(1)-O(30)	2.346(3)	
Na(1)-O(2)#1	2.351(2)	
Na(1)-C(10)	2.822(3)	
Na(1)-C(9)	2.971(3)	
Na(1)-C(5)	3.054(3)	
Na(1)-C(1)#1	3.089(3)	
Na(1)-C(2)#1	3.130(3)	
Na(1)-Na(1)#1	3.189(2)	
O(1)-C(1)	1.252(3)	
O(1)-Na(1)#1	2.289(2)	
C(1)-C(3)#2	1.424(3)	
C(1)-C(2)	1.533(4)	
C(1)-Na(1)#1	3.089(3)	
O(2)-C(2)	1.280(3)	
O(2)-Na(1)#1	2.351(2)	
C(2)-C(3)	1.392(4)	
C(2)-Na(1)#1	3.130(3)	
C(3)-C(1)#2	1.424(3)	
C(3)-C(4)	1.523(4)	
C(4)-C(12)	1.511(4)	
C(4)-C(5)	1.524(4)	
C(4)-H(4)	0.96(2)	
C(5)-C(10)	1.388(4)	
C(5)-C(6)	1.391(4)	
C(6)-C(7)	1.372(4)	
C(6)-H(6)	1.01(3)	
C(7)-C(8)	1.392(4)	
C(7)-H(7)	0.93(3)	
O(8)-C(8)	1.368(3)	
O(8)-C(11)	1.437(4)	
C(8)-C(9)	1.377(4)	
C(9)-C(10)	1.396(4)	
C(9)-H(9)	0.97(3)	
C(10)-H(10)	0.98(2)	
C(11)-H(11A)	0.95(4)	
C(11)-H(11B)	1.06(3)	
C(11)-H(11C)	1.03(3)	
C(12)-C(17)	1.387(4)	
C(12)-C(13)	1.390(4)	

Table E92. continued...

C(13)-C(14)	1.372(4)
C(13)-H(13)	0.94(3)
C(14)-C(15)	1.402(4)
C(14)-H(14)	1.01(3)
O(15)-C(15)	1.372(4)
O(15)-C(18)	1.425(5)
C(15)-C(16)	1.366(4)
C(16)-C(17)	1.379(4)
C(16)-H(16)	0.82(3)
C(17)-H(17)	0.95(3)
C(18)-H(18A)	0.98(4)
C(18)-H(18B)	1.02(5)
C(18)-H(18C)	1.03(4)
O(30)-C(34)	1.400(4)
O(30)-C(31)	1.447(5)
C(31)-C(32B)	1.460(16)
C(31)-C(32A)	1.512(9)
C(31)-H(31A)	0.9700
C(31)-H(31B)	0.9700
C(31)-H(31C)	0.9700
C(31)-H(31D)	0.9700
C(32A)-C(33)	1.441(9)
C(32A)-H(32A)	0.9700
C(32A)-H(32B)	0.9700
C(32B)-C(33)	1.408(16)
C(32B)-H(32C)	0.9700
C(32B)-H(32D)	0.9700
C(33)-C(34)	1.479(6)
C(33)-H(33A)	0.9700
C(33)-H(33B)	0.9700
C(33)-H(33C)	0.9700
C(33)-H(33D)	0.9700
C(34)-H(34A)	0.9700
C(34)-H(34B)	0.9700
O(40)-C(41)	1.386(5)
O(40)-C(44)	1.388(5)
C(41)-C(42)	1.365(6)
C(41)-H(41A)	0.9700
C(41)-H(41B)	0.9700
C(42)-C(43)	1.486(7)
C(42)-H(42A)	0.9700
C(42)-H(42B)	0.9700
C(43)-C(44)	1.449(6)
C(43)-H(43A)	0.9700

Table E92. continued...

C(43)-H(43B)	0.9700
C(44)-H(44A)	0.9700
C(44)-H(44B)	0.9700

O(2)-Na(1)-O(1)#1	160.75(8)
O(2)-Na(1)-O(30)	100.28(9)
O(1)#1-Na(1)-O(30)	90.55(9)
O(2)-Na(1)-O(2)#1	92.34(7)
O(1)#1-Na(1)-O(2)#1	69.43(7)
O(30)-Na(1)-O(2)#1	104.38(9)
O(2)-Na(1)-C(10)	89.77(8)
O(1)#1-Na(1)-C(10)	105.39(8)
O(30)-Na(1)-C(10)	94.18(10)
O(2)#1-Na(1)-C(10)	160.62(10)
O(2)-Na(1)-C(9)	112.64(8)
O(1)#1-Na(1)-C(9)	79.45(8)
O(30)-Na(1)-C(9)	104.30(10)
O(2)#1-Na(1)-C(9)	137.38(9)
C(10)-Na(1)-C(9)	27.75(8)
O(2)-Na(1)-C(5)	65.22(7)
O(1)#1-Na(1)-C(5)	126.30(8)
O(30)-Na(1)-C(5)	108.46(9)
O(2)#1-Na(1)-C(5)	142.81(8)
C(10)-Na(1)-C(5)	26.95(7)
C(9)-Na(1)-C(5)	47.63(8)
O(2)-Na(1)-C(1)#1	141.97(8)
O(1)#1-Na(1)-C(1)#1	20.87(6)
O(30)-Na(1)-C(1)#1	89.87(9)
O(2)#1-Na(1)-C(1)#1	49.66(7)
C(10)-Na(1)-C(1)#1	126.23(8)
C(9)-Na(1)-C(1)#1	99.83(9)
C(5)-Na(1)-C(1)#1	145.07(8)
O(2)-Na(1)-C(2)#1	113.50(8)
O(1)#1-Na(1)-C(2)#1	49.04(7)
O(30)-Na(1)-C(2)#1	95.52(9)
O(2)#1-Na(1)-C(2)#1	21.58(6)
C(10)-Na(1)-C(2)#1	152.60(9)
C(9)-Na(1)-C(2)#1	124.87(9)
C(5)-Na(1)-C(2)#1	155.92(8)
C(1)#1-Na(1)-C(2)#1	28.54(7)
O(2)-Na(1)-Na(1)#1	47.45(5)
O(1)#1-Na(1)-Na(1)#1	114.06(7)
O(30)-Na(1)-Na(1)#1	108.01(9)
O(2)#1-Na(1)-Na(1)#1	44.89(5)

Table E92. continued...

C(10)-Na(1)-Na(1)#1	133.84(8)
C(9)-Na(1)-Na(1)#1	144.53(9)
C(5)-Na(1)-Na(1)#1	106.94(7)
C(1)#1-Na(1)-Na(1)#1	94.53(7)
C(2)#1-Na(1)-Na(1)#1	66.17(6)
C(1)-O(1)-Na(1)#1	118.48(16)
O(1)-C(1)-C(3)#2	121.9(2)
O(1)-C(1)-C(2)	116.7(2)
C(3)#2-C(1)-C(2)	121.4(2)
O(1)-C(1)-Na(1)#1	40.65(12)
C(3)#2-C(1)-Na(1)#1	159.35(18)
C(2)-C(1)-Na(1)#1	77.21(14)
C(2)-O(2)-Na(1)	154.19(17)
C(2)-O(2)-Na(1)#1	115.89(16)
Na(1)-O(2)-Na(1)#1	87.66(7)
O(2)-C(2)-C(3)	125.5(2)
O(2)-C(2)-C(1)	115.3(2)
C(3)-C(2)-C(1)	119.2(2)
O(2)-C(2)-Na(1)#1	42.52(12)
C(3)-C(2)-Na(1)#1	162.84(19)
C(1)-C(2)-Na(1)#1	74.25(13)
C(2)-C(3)-C(1)#2	119.3(2)
C(2)-C(3)-C(4)	124.3(2)
C(1)#2-C(3)-C(4)	116.3(2)
C(12)-C(4)-C(3)	112.0(2)
C(12)-C(4)-C(5)	114.7(2)
C(3)-C(4)-C(5)	114.8(2)
C(12)-C(4)-H(4)	106.6(14)
C(3)-C(4)-H(4)	103.3(14)
C(5)-C(4)-H(4)	104.0(14)
C(10)-C(5)-C(6)	117.0(3)
C(10)-C(5)-C(4)	123.3(2)
C(6)-C(5)-C(4)	119.6(2)
C(10)-C(5)-Na(1)	67.17(15)
C(6)-C(5)-Na(1)	90.34(17)
C(4)-C(5)-Na(1)	114.23(16)
C(7)-C(6)-C(5)	121.8(3)
C(7)-C(6)-H(6)	121.0(16)
C(5)-C(6)-H(6)	117.2(16)
C(6)-C(7)-C(8)	120.6(3)
C(6)-C(7)-H(7)	118.8(17)
C(8)-C(7)-H(7)	120.5(17)
C(8)-O(8)-C(11)	116.9(3)
O(8)-C(8)-C(9)	125.7(3)

Table E92. continued...

O(8)-C(8)-C(7)	115.4(3)
C(9)-C(8)-C(7)	119.0(3)
C(8)-C(9)-C(10)	119.7(3)
C(8)-C(9)-Na(1)	91.11(17)
C(10)-C(9)-Na(1)	70.18(15)
C(8)-C(9)-H(9)	121.8(16)
C(10)-C(9)-H(9)	118.5(16)
Na(1)-C(9)-H(9)	109.1(16)
C(5)-C(10)-C(9)	121.9(3)
C(5)-C(10)-Na(1)	85.88(16)
C(9)-C(10)-Na(1)	82.08(16)
C(5)-C(10)-H(10)	119.9(14)
C(9)-C(10)-H(10)	118.2(14)
Na(1)-C(10)-H(10)	101.8(13)
O(8)-C(11)-H(11A)	99(2)
O(8)-C(11)-H(11B)	108.6(16)
H(11A)-C(11)-H(11B)	112(3)
O(8)-C(11)-H(11C)	111.1(17)
H(11A)-C(11)-H(11C)	114(3)
H(11B)-C(11)-H(11C)	111(2)
C(17)-C(12)-C(13)	117.1(3)
C(17)-C(12)-C(4)	120.5(3)
C(13)-C(12)-C(4)	122.4(2)
C(14)-C(13)-C(12)	121.7(3)
C(14)-C(13)-H(13)	116.0(16)
C(12)-C(13)-H(13)	122.2(16)
C(13)-C(14)-C(15)	119.8(3)
C(13)-C(14)-H(14)	120.3(18)
C(15)-C(14)-H(14)	119.9(18)
C(15)-O(15)-C(18)	116.2(3)
C(16)-C(15)-O(15)	125.6(3)
C(16)-C(15)-C(14)	119.2(3)
O(15)-C(15)-C(14)	115.1(3)
C(15)-C(16)-C(17)	120.2(3)
C(15)-C(16)-H(16)	122(2)
C(17)-C(16)-H(16)	117(2)
C(16)-C(17)-C(12)	122.0(3)
C(16)-C(17)-H(17)	120.5(16)
C(12)-C(17)-H(17)	117.5(16)
O(15)-C(18)-H(18A)	105(2)
O(15)-C(18)-H(18B)	114(3)
H(18A)-C(18)-H(18B)	111(3)
O(15)-C(18)-H(18C)	111(2)
H(18A)-C(18)-H(18C)	110(3)

Table E92. continued...

H(18B)-C(18)-H(18C)	106(3)
C(34)-O(30)-C(31)	106.6(3)
C(34)-O(30)-Na(1)	124.9(2)
C(31)-O(30)-Na(1)	116.1(2)
O(30)-C(31)-C(32B)	102.2(7)
O(30)-C(31)-C(32A)	108.0(4)
C(32B)-C(31)-C(32A)	33.1(8)
O(30)-C(31)-H(31A)	110.1
C(32B)-C(31)-H(31A)	82.6
C(32A)-C(31)-H(31A)	110.1
O(30)-C(31)-H(31B)	110.1
C(32B)-C(31)-H(31B)	138.8
C(32A)-C(31)-H(31B)	110.1
H(31A)-C(31)-H(31B)	108.4
O(30)-C(31)-H(31C)	111.3
C(32B)-C(31)-H(31C)	111.3
C(32A)-C(31)-H(31C)	132.4
H(31A)-C(31)-H(31C)	30.3
H(31B)-C(31)-H(31C)	80.2
O(30)-C(31)-H(31D)	111.3
C(32B)-C(31)-H(31D)	111.3
C(32A)-C(31)-H(31D)	79.2
H(31A)-C(31)-H(31D)	131.7
H(31B)-C(31)-H(31D)	32.8
H(31C)-C(31)-H(31D)	109.2
C(33)-C(32A)-C(31)	102.0(7)
C(33)-C(32A)-H(32A)	111.4
C(31)-C(32A)-H(32A)	111.4
C(33)-C(32A)-H(32B)	111.4
C(31)-C(32A)-H(32B)	111.4
H(32A)-C(32A)-H(32B)	109.2
C(33)-C(32B)-C(31)	106.3(12)
C(33)-C(32B)-H(32C)	110.5
C(31)-C(32B)-H(32C)	110.5
C(33)-C(32B)-H(32D)	110.5
C(31)-C(32B)-H(32D)	110.5
H(32C)-C(32B)-H(32D)	108.7
C(32B)-C(33)-C(32A)	34.6(8)
C(32B)-C(33)-C(34)	105.5(7)
C(32A)-C(33)-C(34)	104.9(5)
C(32B)-C(33)-H(33A)	78.8
C(32A)-C(33)-H(33A)	110.8
C(34)-C(33)-H(33A)	110.8
C(32B)-C(33)-H(33B)	136.5

Table E92. continued...

C(32A)-C(33)-H(33B)	110.8
C(34)-C(33)-H(33B)	110.8
H(33A)-C(33)-H(33B)	108.8
C(32B)-C(33)-H(33C)	110.6
C(32A)-C(33)-H(33C)	137.1
C(34)-C(33)-H(33C)	110.6
H(33A)-C(33)-H(33C)	33.6
H(33B)-C(33)-H(33C)	78.3
C(32B)-C(33)-H(33D)	110.6
C(32A)-C(33)-H(33D)	79.1
C(34)-C(33)-H(33D)	110.6
H(33A)-C(33)-H(33D)	132.7
H(33B)-C(33)-H(33D)	33.4
H(33C)-C(33)-H(33D)	108.8
O(30)-C(34)-C(33)	106.9(4)
O(30)-C(34)-H(34A)	110.3
C(33)-C(34)-H(34A)	110.3
O(30)-C(34)-H(34B)	110.3
C(33)-C(34)-H(34B)	110.3
H(34A)-C(34)-H(34B)	108.6
C(41)-O(40)-C(44)	107.5(4)
C(42)-C(41)-O(40)	111.0(4)
C(42)-C(41)-H(41A)	109.4
O(40)-C(41)-H(41A)	109.4
C(42)-C(41)-H(41B)	109.4
O(40)-C(41)-H(41B)	109.4
H(41A)-C(41)-H(41B)	108.0
C(41)-C(42)-C(43)	106.5(4)
C(41)-C(42)-H(42A)	110.4
C(43)-C(42)-H(42A)	110.4
C(41)-C(42)-H(42B)	110.4
C(43)-C(42)-H(42B)	110.4
H(42A)-C(42)-H(42B)	108.6
C(44)-C(43)-C(42)	104.4(4)
C(44)-C(43)-H(43A)	110.9
C(42)-C(43)-H(43A)	110.9
C(44)-C(43)-H(43B)	110.9
C(42)-C(43)-H(43B)	110.9
H(43A)-C(43)-H(43B)	108.9
O(40)-C(44)-C(43)	108.0(4)
O(40)-C(44)-H(44A)	110.1
C(43)-C(44)-H(44A)	110.1
O(40)-C(44)-H(44B)	110.1
C(43)-C(44)-H(44B)	110.1

Table E92. continued...

H(44A)-C(44)-H(44B)	108.4

Symmetry transformations used to generate equivalent atoms:
#1 -x, -y+1, -z
#2 -x-1, -y+1, -z

Table E93. Anisotropic displacement parameters ($Å^2 \times 10^3$) for $2D^{2-} \cdot (Na^+)_2 \cdot (C_4H_8O)_4$. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+...+2hka^*b^*U_{12}]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Na(1)	19(1)	33(1)	65(1)	25(1)	16(1)	8(1)
O(1)	18(1)	23(1)	67(2)	20(1)	17(1)	4(1)
C(1)	18(1)	22(2)	36(2)	13(1)	10(1)	6(1)
O(2)	12(1)	28(1)	53(1)	19(1)	11(1)	7(1)
C(2)	16(1)	25(2)	35(2)	14(1)	8(1)	6(1)
C(3)	17(1)	23(1)	37(2)	15(1)	11(1)	7(1)
C(4)	14(1)	22(2)	38(2)	11(1)	8(1)	5(1)
C(5)	18(1)	18(1)	38(2)	11(1)	8(1)	1(1)
C(6)	26(2)	35(2)	40(2)	18(2)	8(1)	11(1)
C(7)	34(2)	45(2)	34(2)	19(2)	10(2)	15(2)
O(8)	39(1)	50(1)	47(1)	19(1)	23(1)	25(1)
C(8)	26(2)	27(2)	39(2)	9(1)	12(1)	9(1)
C(9)	23(2)	28(2)	38(2)	15(1)	8(1)	8(1)
C(10)	22(2)	24(2)	34(2)	14(1)	10(1)	6(1)
C(11)	33(2)	51(2)	56(2)	20(2)	22(2)	23(2)
C(12)	19(1)	22(2)	40(2)	14(1)	12(1)	10(1)
C(13)	28(2)	29(2)	46(2)	16(2)	14(2)	5(1)
C(14)	32(2)	40(2)	40(2)	14(2)	6(2)	2(2)
O(15)	54(2)	66(2)	47(2)	31(1)	14(1)	5(1)
C(15)	36(2)	42(2)	40(2)	22(2)	18(2)	15(2)
C(16)	28(2)	35(2)	53(2)	25(2)	16(2)	4(2)
C(17)	25(2)	29(2)	42(2)	16(2)	12(2)	4(1)
C(18)	61(3)	92(4)	56(3)	46(3)	18(2)	9(3)
O(30)	43(1)	66(2)	71(2)	38(2)	10(1)	9(1)
C(31)	123(4)	80(3)	96(4)	43(3)	-1(3)	-23(3)
C(33)	133(5)	116(5)	89(4)	49(4)	-15(4)	46(4)
C(34)	57(3)	58(3)	104(4)	41(3)	1(2)	12(2)
O(40)	62(2)	121(3)	94(2)	-19(2)	-8(2)	39(2)
C(41)	85(4)	131(5)	80(4)	-20(3)	-10(3)	41(4)
C(42)	69(4)	67(3)	220(7)	-29(4)	-7(4)	21(3)
C(43)	65(3)	83(4)	133(5)	29(3)	-20(3)	5(3)
C(44)	65(3)	82(3)	81(3)	3(3)	20(3)	8(3)

Table E94. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for $2D^{2-}•(Na^+)_2•(C_4H_8O)_4$.

Atom	x	y	z	U_{eq}	Occ.
H(4)	-5330(30)	1940(20)	-558(19)	16(6)	1
H(6)	-4150(40)	2340(30)	-2110(20)	42(8)	1
H(7)	-2350(30)	1500(30)	-3140(20)	36(8)	1
H(9)	50(30)	540(30)	-630(20)	33(8)	1
H(10)	-1680(30)	1410(20)	420(20)	14(6)	1
H(11A)	1930(40)	-50(30)	-2820(30)	65(11)	1
H(11B)	990(40)	-790(30)	-2170(20)	50(9)	1
H(11C)	2090(40)	720(30)	-1470(30)	54(10)	1
H(13)	-2150(30)	3890(30)	1950(20)	24(7)	1
H(14)	-1740(40)	3740(30)	3670(30)	52(9)	1
H(16)	-5480(40)	790(30)	2250(20)	31(8)	1
H(17)	-5830(30)	860(30)	540(20)	34(8)	1
H(18A)	-4150(40)	1480(30)	5150(40)	71(12)	1
H(18B)	-4290(50)	370(40)	3940(40)	97(16)	1
H(18C)	-5630(50)	1280(30)	4050(30)	76(12)	1
H(31A)	2130	5917	3030	127	0.66(2)
H(31B)	3145	5423	2164	127	0.66(2)
H(31C)	2203	5971	2677	127	0.34(2)
H(31D)	3524	5159	2354	127	0.34(2)
H(32A)	4721	4672	3265	101	0.66(2)
H(32B)	4145	5618	4233	101	0.66(2)
H(32C)	4105	5585	4183	105	0.34(2)
H(32D)	2261	5542	4194	105	0.34(2)
H(33A)	2200	4162	4153	139	0.66(2)
H(33B)	3481	3322	3811	139	0.66(2)
H(33C)	2345	3704	4161	139	0.34(2)
H(33D)	3843	3603	3642	139	0.34(2)
H(34A)	2179	2597	1998	88	1
H(34B)	679	2742	2512	88	1
H(41A)	-7168	3119	-2358	143	1
H(41B)	-6479	3990	-2912	143	1
H(42A)	-8769	4035	-3714	178	1
H(42B)	-9444	3320	-3027	178	1
H(43A)	-10404	1660	-4555	126	1
H(43B)	-9647	2353	-5231	126	1
H(44A)	-8425	694	-4419	101	1
H(44B)	-7903	1154	-5309	101	1

Table E95. Torsion angles (°) for $2D^{2-}\bullet(Na^{+})_2\bullet(C_4H_8O)_4$.

Atoms	Angle
Na(1)#1 O(1) C(1) C(3)#2	-165.3(2)
Na(1)#1 O(1) C(1) C(2)	15.0(3)
O(1)#1 Na(1) O(2) C(2)	138.6(4)
O(30) Na(1) O(2) C(2)	-98.1(4)
O(2)#1 Na(1) O(2) C(2)	156.9(4)
C(10) Na(1) O(2) C(2)	-3.9(4)
C(9) Na(1) O(2) C(2)	12.3(4)
C(5) Na(1) O(2) C(2)	7.6(4)
C(1)#1 Na(1) O(2) C(2)	158.7(4)
C(2)#1 Na(1) O(2) C(2)	161.2(4)
Na(1)#1 Na(1) O(2) C(2)	156.9(4)
O(1)#1 Na(1) O(2) Na(1)#1	-18.3(3)
O(30) Na(1) O(2) Na(1)#1	105.07(10)
O(2)#1 Na(1) O(2) Na(1)#1	0.0
C(10) Na(1) O(2) Na(1)#1	-160.73(10)
C(9) Na(1) O(2) Na(1)#1	-144.59(10)
C(5) Na(1) O(2) Na(1)#1	-149.23(10)
C(1)#1 Na(1) O(2) Na(1)#1	1.86(14)
C(2)#1 Na(1) O(2) Na(1)#1	4.37(9)
Na(1) O(2) C(2) C(3)	9.4(6)
Na(1)#1 O(2) C(2) C(3)	163.5(2)
Na(1) O(2) C(2) C(1)	-170.3(3)
Na(1)#1 O(2) C(2) C(1)	-16.2(3)
Na(1) O(2) C(2) Na(1)#1	-154.1(5)
O(1) C(1) C(2) O(2)	1.3(4)
C(3)#2 C(1) C(2) O(2)	-178.3(2)
Na(1)#1 C(1) C(2) O(2)	11.3(2)
O(1) C(1) C(2) C(3)	-178.4(3)
C(3)#2 C(1) C(2) C(3)	2.0(4)
Na(1)#1 C(1) C(2) C(3)	-168.4(2)
O(1) C(1) C(2) Na(1)#1	-10.0(2)
C(3)#2 C(1) C(2) Na(1)#1	170.4(3)
O(2) C(2) C(3) C(1)#2	178.4(3)
C(1) C(2) C(3) C(1)#2	-1.9(4)
Na(1)#1 C(2) C(3) C(1)#2	-140.9(5)
O(2) C(2) C(3) C(4)	0.8(4)
C(1) C(2) C(3) C(4)	-179.5(2)
Na(1)#1 C(2) C(3) C(4)	41.4(8)
C(2) C(3) C(4) C(12)	94.3(3)
C(1)#2 C(3) C(4) C(12)	-83.3(3)
C(2) C(3) C(4) C(5)	-38.8(4)

Table E95. continued...

C(1)#2 C(3) C(4) C(5)	143.5(2)
C(12) C(4) C(5) C(10)	1.4(4)
C(3) C(4) C(5) C(10)	133.3(3)
C(12) C(4) C(5) C(6)	178.5(2)
C(3) C(4) C(5) C(6)	-49.7(3)
C(12) C(4) C(5) Na(1)	-76.3(2)
C(3) C(4) C(5) Na(1)	55.6(3)
O(2) Na(1) C(5) C(10)	-153.92(19)
O(1)#1 Na(1) C(5) C(10)	44.1(2)
O(30) Na(1) C(5) C(10)	-60.87(19)
O(2)#1 Na(1) C(5) C(10)	148.36(18)
C(9) Na(1) C(5) C(10)	31.89(17)
C(1)#1 Na(1) C(5) C(10)	57.4(2)
C(2)#1 Na(1) C(5) C(10)	113.6(2)
Na(1)#1 Na(1) C(5) C(10)	-177.12(17)
O(2) Na(1) C(5) C(6)	86.72(17)
O(1)#1 Na(1) C(5) C(6)	-75.29(19)
O(30) Na(1) C(5) C(6)	179.77(17)
O(2)#1 Na(1) C(5) C(6)	29.0(2)
C(10) Na(1) C(5) C(6)	-119.4(3)
C(9) Na(1) C(5) C(6)	-87.47(19)
C(1)#1 Na(1) C(5) C(6)	-61.9(2)
C(2)#1 Na(1) C(5) C(6)	-5.8(3)
Na(1)#1 Na(1) C(5) C(6)	63.52(18)
O(2) Na(1) C(5) C(4)	-36.26(17)
O(1)#1 Na(1) C(5) C(4)	161.73(16)
O(30) Na(1) C(5) C(4)	56.79(19)
O(2)#1 Na(1) C(5) C(4)	-94.0(2)
C(10) Na(1) C(5) C(4)	117.7(3)
C(9) Na(1) C(5) C(4)	149.5(2)
C(1)#1 Na(1) C(5) C(4)	175.07(16)
C(2)#1 Na(1) C(5) C(4)	-128.8(2)
Na(1)#1 Na(1) C(5) C(4)	-59.46(19)
C(10) C(5) C(6) C(7)	-0.5(4)
C(4) C(5) C(6) C(7)	-177.8(3)
Na(1) C(5) C(6) C(7)	63.8(3)
C(5) C(6) C(7) C(8)	0.5(5)
C(11) O(8) C(8) C(9)	-6.6(4)
C(11) O(8) C(8) C(7)	173.6(3)
C(6) C(7) C(8) O(8)	179.9(3)
C(6) C(7) C(8) C(9)	0.1(4)
O(8) C(8) C(9) C(10)	179.6(2)
C(7) C(8) C(9) C(10)	-0.7(4)
O(8) C(8) C(9) Na(1)	111.9(3)

Table E95. continued...

C(7) C(8) C(9) Na(1)	-68.3(3)
O(2) Na(1) C(9) C(8)	84.70(18)
O(1)#1 Na(1) C(9) C(8)	-79.63(17)
O(30) Na(1) C(9) C(8)	-167.49(17)
O(2)#1 Na(1) C(9) C(8)	-36.5(2)
C(10) Na(1) C(9) C(8)	121.4(3)
C(5) Na(1) C(9) C(8)	90.41(19)
C(1)#1 Na(1) C(9) C(8)	-75.09(18)
C(2)#1 Na(1) C(9) C(8)	-60.1(2)
Na(1)#1 Na(1) C(9) C(8)	37.3(2)
O(2) Na(1) C(9) C(10)	-36.66(19)
O(1)#1 Na(1) C(9) C(10)	159.01(18)
O(30) Na(1) C(9) C(10)	71.15(18)
O(2)#1 Na(1) C(9) C(10)	-157.89(17)
C(5) Na(1) C(9) C(10)	-30.95(16)
C(1)#1 Na(1) C(9) C(10)	163.55(17)
C(2)#1 Na(1) C(9) C(10)	178.53(16)
Na(1)#1 Na(1) C(9) C(10)	-84.0(2)
C(6) C(5) C(10) C(9)	0.0(4)
C(4) C(5) C(10) C(9)	177.1(2)
Na(1) C(5) C(10) C(9)	-78.0(2)
C(6) C(5) C(10) Na(1)	78.0(2)
C(4) C(5) C(10) Na(1)	-104.8(2)
C(8) C(9) C(10) C(5)	0.6(4)
Na(1) C(9) C(10) C(5)	80.1(2)
C(8) C(9) C(10) Na(1)	-79.5(3)
O(2) Na(1) C(10) C(5)	23.53(18)
O(1)#1 Na(1) C(10) C(5)	-144.45(17)
O(30) Na(1) C(10) C(5)	123.82(18)
O(2)#1 Na(1) C(10) C(5)	-72.9(3)
C(9) Na(1) C(10) C(5)	-123.0(3)
C(1)#1 Na(1) C(10) C(5)	-143.27(16)
C(2)#1 Na(1) C(10) C(5)	-125.7(2)
Na(1)#1 Na(1) C(10) C(5)	3.8(2)
O(2) Na(1) C(10) C(9)	146.56(18)
O(1)#1 Na(1) C(10) C(9)	-21.42(19)
O(30) Na(1) C(10) C(9)	-113.15(18)
O(2)#1 Na(1) C(10) C(9)	50.2(3)
C(5) Na(1) C(10) C(9)	123.0(3)
C(1)#1 Na(1) C(10) C(9)	-20.2(2)
C(2)#1 Na(1) C(10) C(9)	-2.6(3)
Na(1)#1 Na(1) C(10) C(9)	126.86(17)
C(3) C(4) C(12) C(17)	124.2(3)
C(5) C(4) C(12) C(17)	-102.6(3)

Table E95. continued...

C(3) C(4) C(12) C(13)	-55.3(3)
C(5) C(4) C(12) C(13)	77.9(3)
C(17) C(12) C(13) C(14)	-0.9(4)
C(4) C(12) C(13) C(14)	178.6(3)
C(12) C(13) C(14) C(15)	0.5(4)
C(18) O(15) C(15) C(16)	-1.3(5)
C(18) O(15) C(15) C(14)	178.3(3)
C(13) C(14) C(15) C(16)	-0.1(4)
C(13) C(14) C(15) O(15)	-179.7(3)
O(15) C(15) C(16) C(17)	179.8(3)
C(14) C(15) C(16) C(17)	0.2(4)
C(15) C(16) C(17) C(12)	-0.7(4)
C(13) C(12) C(17) C(16)	1.1(4)
C(4) C(12) C(17) C(16)	-178.5(3)
O(2) Na(1) O(30) C(34)	130.1(3)
O(1)#1 Na(1) O(30) C(34)	-65.9(3)
O(2)#1 Na(1) O(30) C(34)	-134.8(3)
C(10) Na(1) O(30) C(34)	39.6(3)
C(9) Na(1) O(30) C(34)	13.4(3)
C(5) Na(1) O(30) C(34)	63.0(3)
C(1)#1 Na(1) O(30) C(34)	-86.7(3)
C(2)#1 Na(1) O(30) C(34)	-114.8(3)
Na(1)#1 Na(1) O(30) C(34)	178.5(3)
O(2) Na(1) O(30) C(31)	-93.0(3)
O(1)#1 Na(1) O(30) C(31)	71.0(3)
O(2)#1 Na(1) O(30) C(31)	2.1(3)
C(10) Na(1) O(30) C(31)	176.5(3)
C(9) Na(1) O(30) C(31)	150.3(3)
C(5) Na(1) O(30) C(31)	-160.1(3)
C(1)#1 Na(1) O(30) C(31)	50.1(3)
C(2)#1 Na(1) O(30) C(31)	22.1(3)
Na(1)#1 Na(1) O(30) C(31)	-44.6(3)
C(34) O(30) C(31) C(32B)	-33.4(13)
Na(1) O(30) C(31) C(32B)	-177.6(12)
C(34) O(30) C(31) C(32A)	0.4(8)
Na(1) O(30) C(31) C(32A)	-143.9(7)
O(30) C(31) C(32A) C(33)	-20.7(10)
C(32B) C(31) C(32A) C(33)	64.0(13)
O(30) C(31) C(32B) C(33)	34.8(19)
C(32A) C(31) C(32B) C(33)	-69.6(17)
C(31) C(32B) C(33) C(32A)	71.1(17)
C(31) C(32B) C(33) C(34)	-23(2)
C(31) C(32A) C(33) C(32B)	-63.6(13)
C(31) C(32A) C(33) C(34)	32.0(10)

Table E95. continued...

C(31) O(30) C(34) C(33)	20.0(5)
Na(1) O(30) C(34) C(33)	160.2(3)
C(32B) C(33) C(34) O(30)	2.0(14)
C(32A) C(33) C(34) O(30)	-33.9(8)
C(44) O(40) C(41) C(42)	-15.1(8)
O(40) C(41) C(42) C(43)	7.3(9)
C(41) C(42) C(43) C(44)	2.8(8)
C(41) O(40) C(44) C(43)	16.5(6)
C(42) C(43) C(44) O(40)	-11.8(6)

Symmetry transformations used to generate equivalent atoms:

#1 -x, -y+1, -z

#2 -x-1, -y+1, -z

Table E96. Crystal and structure refinement data for **2E•$(C_3H_6O)_2$**.

Identification code	**2E•$(C_3H_6O)_2$**
Empirical formula	$C_{74}H_{108}O_{10}$
Formula weight	1157.60 g/mole
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group	P_1
Unit cell dimensions	a = 11.914(2) Å b = 12.980(3) Å c = 13.215(3) Å α = 70.89(3)° β = 77.59(3)° γ = 77.07(3)°
Volume	1859.56 $Å^3$
Z	1
Density (calculated)	1.034 Mg/m^3
Absorption coefficient	0.067 mm^{-1}
F(000)	632
Crystal Size	**?** mm x **?** mm x **?** mm
Color	yellow
Theta range for data collection	1.65° to 28.25°
Index ranges	$-15 \le h \le 15$, $-17 \le k \le 17$, $-17 \le l \le 17$
Reflections collected / Unique	22245 / 16484 [R_{int} = 0.0139]
Refinement method	Full-matrix-least-squares on F^2
Data / Restraints / Parameters	16484 / 3 / 769
Goodness-of-fit on F^2	1.180
Final R indices [$I>2\sigma(I)$]	R_1 = 0.0608, wR_2 = 0.1713
R indices (all data)	R_1 = 0.1006, wR_2 = 0.1973
Largest diff. peak and hole	0.327 and -0.216 e/A^3

Table E97. Atomic coordinates and equivalent isotropic displacement parameters ($Å^2$) for **2E•(C_3H_6O)$_2$**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U_{eq}
C1	0.8415(3)	0.2360(3)	0.4598(3)	0.0384(8)
C2	0.7983(3)	0.1954(3)	0.5645(3)	0.0385(9)
O3	0.8217(2)	0.2268(2)	0.6438(2)	0.0491(7)
C4	0.7211(3)	0.1082(3)	0.6026(3)	0.0378(8)
O5	0.6885(3)	0.0748(3)	0.7005(2)	0.0604(8)
C6	0.6880(3)	0.0657(3)	0.5247(3)	0.0368(8)
C7	0.7351(3)	0.1075(3)	0.4195(3)	0.0386(9)
O8	0.7119(3)	0.0740(3)	0.3419(2)	0.0592(8)
C9	0.8135(3)	0.1943(3)	0.3811(3)	0.0408(9)
O1	0 0.8458(3)	0.2276(3)	0.2835(2)	0.0578(8)
C11	0.9287(3)	0.3193(3)	0.4231(3)	0.0405(9)
C12	1.0412(3)	0.2755(3)	0.3554(3)	0.0448(10)
C13	1.0952(3)	0.1689(3)	0.3918(3)	0.0458(10)
C14	1.2005(4)	0.1266(4)	0.3363(4)	0.0599(13)
C15	1.2536(4)	0.2004(4)	0.2463(4)	0.0553(11)
C16	1.1964(4)	0.3094(4)	0.2000(4)	0.0580(12)
C17	1.0892(4)	0.3439(4)	0.2551(3)	0.0531(11)
C18	1.2466(4)	-0.0002(5)	0.3746(5)	0.0731(15)
C19	1.1467(7)	-0.0566(5)	0.4216(12)	0.248(8)
C20	1.2788(9)	-0.0521(6)	0.2867(7)	0.161(4)
C21	1.3184(12)	-0.0259(6)	0.4578(10)	0.293(9)
O22	1.3665(3)	0.1654(4)	0.1975(3)	0.0901(13)
C23	1.4564(5)	0.1857(8)	0.2437(7)	0.130(3)
C24	1.2416(5)	0.3835(5)	0.0914(5)	0.0881(17)
C25	1.1479(8)	0.4886(7)	0.0506(6)	0.177(4)
C26	1.3467(8)	0.4293(11)	0.0946(8)	0.247(7)
C27	1.2828(9)	0.3201(9)	0.0032(5)	0.180(4)
C28	0.8738(3)	0.4378(3)	0.3706(3)	0.0425(10)
C29	0.7717(3)	0.4653(3)	0.3265(3)	0.0490(10)
C30	0.7251(4)	0.5767(4)	0.2747(4)	0.0607(12)
C31	0.7809(4)	0.6604(3)	0.2795(3)	0.0536(11)
C32	0.8885(4)	0.6345(4)	0.3202(4)	0.0550(11)
C33	0.9300(4)	0.5232(3)	0.3667(3)	0.0492(10)
C34	0.6205(5)	0.5961(5)	0.2176(5)	0.0830(17)
C35	0.5072(6)	0.6454(11)	0.2780(10)	0.220(6)
C36	0.6159(10)	0.4965(9)	0.1886(12)	0.252(8)
C37	0.6432(10)	0.6762(11)	0.1044(7)	0.230(6)
O38	0.7356(3)	0.7686(3)	0.2402(3)	0.0804(11)
C39	0.6429(6)	0.8133(5)	0.3142(6)	0.111(2)

Table E97. continued...

C40	0.9634(5)	0.7221(4)	0.3092(5)	0.0837(17)
C41	0.9030(6)	0.8008(5)	0.3777(8)	0.138(3)
C42	1.0837(6)	0.6676(5)	0.3462(6)	0.1129(19)
C43	0.9768(8)	0.7950(7)	0.1864(7)	0.166(4)
C44	0.6060(3)	-0.0162(3)	0.5607(3)	0.0399(9)
C45	0.4938(3)	0.0229(3)	0.6298(3)	0.0407(9)
C46	0.4377(3)	0.1318(4)	0.5929(4)	0.0520(11)
C47	0.3337(3)	0.1760(4)	0.6452(4)	0.0517(11)
C48	0.2811(4)	0.1002(5)	0.7385(4)	0.0641(14)
C49	0.3394(4)	-0.0056(4)	0.7850(4)	0.0607(12)
C50	0.4448(4)	-0.0420(4)	0.7247(3)	0.0507(11)
C51	0.2875(5)	0.3005(4)	0.6051(5)	0.0712(15)
C52	0.1745(6)	0.3296(6)	0.5574(7)	0.137(2)
C53	0.2445(12)	0.3431(7)	0.7099(8)	0.222(6)
C54	0.3764(8)	0.3647(5)	0.5206(7)	0.159(3)
O55	0.1687(3)	0.1355(4)	0.7856(3)	0.0888(12)
C56	0.0781(5)	0.1156(8)	0.7432(7)	0.125(3)
C57	0.2952(4)	-0.0867(6)	0.8980(4)	0.0884(19)
C58	0.2452(12)	-0.0310(8)	0.9775(7)	0.225(7)
C59	0.3932(7)	-0.1599(12)	0.9437(6)	0.308(10)
C60	0.2092(12)	-0.1496(10)	0.8882(7)	0.240(7)
C61	0.6618(3)	-0.1363(3)	0.6102(3)	0.0423(9)
C62	0.6037(4)	-0.2202(4)	0.6158(4)	0.0534(11)
C63	0.6456(4)	-0.3311(3)	0.6645(4)	0.0569(12)
C64	0.7505(4)	-0.3542(4)	0.7027(4)	0.0621(13)
C65	0.8079(4)	-0.2735(4)	0.7058(4)	0.0576(12)
C66	0.7583(4)	-0.1649(4)	0.6587(3)	0.0547(11)
C67	0.9144(6)	-0.2935(5)	0.7668(5)	0.0906(19)
C68	0.9155(11)	-0.1990(8)	0.8041(11)	0.241(7)
C69	0.9263(9)	-0.4028(8)	0.8543(8)	0.204(5)
C70	1.0223(6)	-0.2852(9)	0.6857(8)	0.186(5)
O71	0.8016(3)	-0.4690(3)	0.7438(3)	0.0813(11)
C72	0.8765(6)	-0.5135(5)	0.6656(6)	0.103(2)
C73	0.5699(5)	-0.4197(4)	0.6768(5)	0.0711(14)
C74	0.5453(9)	-0.4873(9)	0.7877(7)	0.158(3)
C75	0.6191(7)	-0.4848(8)	0.5968(7)	0.163(4)
C76	0.4496(6)	-0.3668(6)	0.6613(11)	0.224(6)
C77	0.8463(8)	0.2040(5)	0.0613(5)	0.099(2)
O78	0.7662(5)	0.1642(5)	0.1182(4)	0.143(2)
C79	0.8416(16)	0.3016(9)	-0.0241(9)	0.283(9)
C80	0.9688(10)	0.1332(10)	0.0801(12)	0.244(7)
C81	0.6937(7)	0.0972(6)	0.9181(5)	0.098(2)
O82	0.7761(4)	0.1312(5)	0.8646(3)	0.1075(15)
C83	0.5819(7)	0.1479(9)	0.9106(7)	0.153(3)

Table E97. continued...

C84	0.7014(11)	-0.0171(8)	0.9989(8)	0.202(5)

Table E98. Bond Lengths (Å) and angles (°) for **2E•$(C_3H_6O)_2$**.

Atoms	Length	Angle
C1-C2	1.339(5)	
C1-C9	1.444(5)	
C1-C11	1.558(5)	
C2-O3	1.341(5)	
C2-C4	1.508(5)	
C4-O5	1.227(4)	
C4-C6	1.472(5)	
C6-C7	1.360(5)	
C6-C44	1.498(6)	
C7-O8	1.335(5)	
C7-C9	1.514(6)	
C9-O10	1.222(5)	
C11-C28	1.523(5)	
C11-C12	1.547(5)	
C12-C13	1.364(6)	
C12-C17	1.415(6)	
C13-C14	1.404(6)	
C14-C15	1.391(7)	
C14-C18	1.561(7)	
C15-O22	1.406(5)	
C15-C16	1.422(7)	
C16-C17	1.386(6)	
C16-C24	1.507(8)	
C18-C21	1.446(8)	
C18-C19	1.454(8)	
C18-C20	1.468(8)	
O22-C23	1.450(8)	
C24-C26	1.516(10)	
C24-C27	1.575(10)	
C24-C25	1.578(10)	
C28-C29	1.387(5)	
C28-C33	1.399(5)	
C29-C30	1.423(6)	
C30-C31	1.418(7)	
C30-C34	1.526(7)	
C31-O38	1.355(5)	
C31-C32	1.425(6)	
C32-C33	1.393(6)	
C32-C40	1.550(7)	
C34-C36	1.478(11)	
C34-C35	1.534(10)	

Table E98. continued...

C34-C37	1.525(10)
O38-C39	1.461(6)
C40-C41	1.542(8)
C40-C42	1.554(9)
C40-C43	1.582(9)
C44-C61	1.534(5)
C44-C45	1.534(5)
C45-C50	1.356(5)
C45-C46	1.397(5)
C46-C47	1.384(6)
C47-C48	1.425(7)
C47-C51	1.537(7)
C48-O55	1.393(5)
C48-C49	1.397(7)
C49-C50	1.409(6)
C49-C57	1.580(7)
C51-C54	1.529(9)
C51-C52	1.528(8)
C51-C53	1.599(10)
O55-C56	1.423(7)
C57-C58	1.420(10)
C57-C59	1.437(9)
C57-C60	1.494(10)
C61-C66	1.357(6)
C61-C62	1.391(6)
C62-C63	1.395(6)
C63-C64	1.382(7)
C63-C73	1.562(6)
C64-C65	1.388(7)
C64-O71	1.447(5)
C65-C66	1.391(6)
C65-C67	1.577(7)
C67-C68	1.467(9)
C67-C70	1.486(9)
C67-C69	1.508(10)
O71-C72	1.400(6)
C73-C74	1.446(10)
C73-C76	1.470(8)
C73-C75	1.507(8)
C77-O78	1.184(8)
C77-C79	1.393(12)
C77-C80	1.563(13)
C81-O82	1.162(7)
C81-C83	1.356(10)

Table E98. continued...

C81-C84	1.514(11)
C2-C1-C9	118.8(3)
C2-C1-C11	121.4(4)
C9-C1-C11	119.7(3)
C1-C2-O3	123.2(3)
C1-C2-C4	122.2(3)
O3-C2-C4	114.6(3)
O5-C4-C6	122.3(3)
O5-C4-C2	117.0(3)
C6-C4-C2	120.7(3)
C7-C6-C4	115.3(3)
C7-C6-C44	123.4(3)
C4-C6-C44	121.3(3)
O8-C7-C6	120.4(4)
O8-C7-C9	115.5(3)
C6-C7-C9	124.1(3)
O10-C9-C1	124.0(4)
O10-C9-C7	117.0(4)
C1-C9-C7	118.9(3)
C28-C11-C12	114.6(3)
C28-C11-C1	113.4(3)
C12-C11-C1	110.3(3)
C13-C12-C17	118.8(4)
C13-C12-C11	119.9(4)
C17-C12-C11	121.2(4)
C12-C13-C14	122.2(4)
C15-C14-C13	117.3(4)
C15-C14-C18	124.1(4)
C13-C14-C18	118.5(5)
C14-C15-O22	119.1(4)
C14-C15-C16	122.2(4)
O22-C15-C16	118.7(4)
C17-C16-C15	117.0(4)
C17-C16-C24	119.4(5)
C15-C16-C24	123.4(4)
C16-C17-C12	121.5(4)
C21-C18-C19	107.7(9)
C21-C18-C20	121.9(7)
C19-C18-C20	92.1(7)
C21-C18-C14	110.6(5)
C19-C18-C14	108.0(5)
C20-C18-C14	113.9(5)
C15-O22-C23	112.5(4)

Table E98. continued...

C16-C24-C26	112.6(5)
C16-C24-C27	111.9(6)
C26-C24-C27	105.5(8)
C16-C24-C25	111.7(4)
C26-C24-C25	104.9(8)
C27-C24-C25	109.8(6)
C29-C28-C33	118.5(4)
C29-C28-C11	123.5(3)
C33-C28-C11	118.0(3)
C28-C29-C30	122.0(4)
C29-C30-C31	117.2(4)
C29-C30-C34	117.4(4)
C31-C30-C34	125.4(4)
O38-C31-C32	117.8(4)
O38-C31-C30	120.5(4)
C32-C31-C30	121.6(4)
C33-C32-C31	117.1(4)
C33-C32-C40	119.2(4)
C31-C32-C40	123.6(4)
C32-C33-C28	123.1(4)
C36-C34-C30	110.4(5)
C36-C34-C35	116.0(8)
C30-C34-C35	112.6(6)
C36-C34-C37	99.7(8)
C30-C34-C37	109.0(5)
C35-C34-C37	108.2(8)
C31-O38-C39	115.3(4)
C41-C40-C42	107.3(6)
C41-C40-C32	112.0(5)
C42-C40-C32	111.7(4)
C41-C40-C43	107.0(6)
C42-C40-C43	111.8(5)
C32-C40-C43	107.0(5)
C6-C44-C61	114.8(3)
C6-C44-C45	111.5(3)
C61-C44-C45	114.0(3)
C50-C45-C46	117.5(4)
C50-C45-C44	124.1(3)
C46-C45-C44	118.4(4)
C47-C46-C45	124.1(4)
C46-C47-C48	115.4(4)
C46-C47-C51	119.5(4)
C48-C47-C51	125.0(4)
O55-C48-C49	119.8(5)

Table E98. continued...

O55-C48-C47	118.0(5)
C49-C48-C47	122.2(4)
C48-C49-C50	116.7(4)
C48-C49-C57	125.0(4)
C50-C49-C57	118.3(4)
C45-C50-C49	123.2(4)
C54-C51-C47	112.8(4)
C54-C51-C52	107.4(6)
C47-C51-C52	114.7(5)
C54-C51-C53	113.6(7)
C47-C51-C53	107.0(5)
C52-C51-C53	100.8(6)
C48-O55-C56	114.6(4)
C58-C57-C59	102.1(9)
C58-C57-C60	109.2(8)
C59-C57-C60	111.4(9)
C58-C57-C49	112.5(6)
C59-C57-C49	109.5(4)
C60-C57-C49	111.8(5)
C66-C61-C62	117.8(4)
C66-C61-C44	123.6(4)
C62-C61-C44	118.2(4)
C63-C62-C61	121.7(4)
C62-C63-C64	116.8(4)
C62-C63-C73	118.7(4)
C64-C63-C73	124.5(4)
C65-C64-C63	123.6(4)
C65-C64-O71	118.4(4)
C63-C64-O71	118.0(5)
C64-C65-C66	115.6(4)
C64-C65-C67	126.1(4)
C66-C65-C67	118.0(4)
C61-C66-C65	124.0(4)
C68-C67-C70	98.1(9)
C68-C67-C69	113.5(7)
C70-C67-C69	110.7(7)
C68-C67-C65	111.9(5)
C70-C67-C65	108.5(5)
C69-C67-C65	113.2(6)
C72-O71-C64	114.4(4)
C74-C73-C76	98.0(7)
C74-C73-C75	114.1(7)
C76-C73-C75	109.1(7)
C74-C73-C63	112.7(5)

Table E98. continued...

C76-C73-C63	110.4(4)
C75-C73-C63	111.7(4)
O78-C77-C79	126.8(11)
O78-C77-C80	115.2(7)
C79-C77-C80	117.8(10)
O82-C81-C83	125.7(8)
O82-C81-C84	121.8(8)
C83-C81-C84	112.3(8)

Table E99. Anisotropic displacement parameters ($Å^2$) for **2E•$(C_3H_6O)_2$**. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2a^{*2}U_{11}+...+2hka^*b^*U_{12}]$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	0.0364(19)	0.0279(18)	0.047(2)	-0.0076(16)	-0.0021(16)	-0.0080(15)
C2	0.0300(19)	0.040(2)	0.047(2)	-0.0143(17)	-0.0027(16)	-0.0080(16)
O3	0.0560(17)	0.0551(17)	0.0427(16)	-0.0187(13)	-0.0052(13)	-0.0183(14)
C4	0.035(2)	0.036(2)	0.039(2)	-0.0120(17)	-0.0004(16)	-0.0031(15)
O5	0.080(2)	0.0636(19)	0.0425(18)	-0.0108(15)	0.0012(15)	-0.0390(16)
C6	0.0344(19)	0.037(2)	0.038(2)	-0.0109(16)	-0.0065(15)	-0.0021(15)
C7	0.046(2)	0.0303(19)	0.038(2)	-0.0116(16)	-0.0093(17)	0.0004(16)
O8	0.080(2)	0.063(2)	0.0438(17)	-0.0162(14)	-0.0022(15)	-0.0371(17)
C9	0.046(2)	0.038(2)	0.037(2)	-0.0075(17)	-0.0058(17)	-0.0105(17)
O10	0.0658(19)	0.070(2)	0.0372(17)	-0.0135(14)	0.0029(14)	-0.0249(15)
C11	0.038(2)	0.036(2)	0.045(2)	-0.0094(17)	0.0016(17)	-0.0142(16)
C12	0.036(2)	0.050(2)	0.053(3)	-0.020(2)	-0.0032(18)	-0.0110(18)
C13	0.045(2)	0.042(2)	0.052(3)	-0.0157(19)	-0.0057(19)	-0.0090(18)
C14	0.053(3)	0.059(3)	0.073(3)	-0.030(3)	-0.013(2)	0.001(2)
C15	0.037(2)	0.070(3)	0.063(3)	-0.031(2)	-0.008(2)	-0.001(2)
C16	0.040(2)	0.079(3)	0.057(3)	-0.025(2)	0.003(2)	-0.016(2)
C17	0.043(2)	0.061(3)	0.055(3)	-0.018(2)	0.0015(19)	-0.013(2)
C18	0.058(3)	0.066(3)	0.104(4)	-0.039(3)	-0.029(3)	0.010(2)
C19	0.108(5)	0.056(4)	0.56(2)	-0.106(7)	0.048(8)	-0.032(3)
C20	0.242(9)	0.094(5)	0.132(5)	-0.065(4)	0.038(6)	-0.016(5)
C21	0.478(18)	0.071(4)	0.435(16)	-0.043(6)	-0.417(16)	0.029(7)
O22	0.045(2)	0.134(4)	0.101(3)	-0.065(3)	0.0026(19)	-0.002(2)
C23	0.046(4)	0.163(8)	0.169(7)	-0.042(6)	-0.007(4)	-0.016(4)
C24	0.076(4)	0.091(4)	0.083(4)	-0.015(3)	-0.004(3)	-0.011(3)
C25	0.170(7)	0.147(5)	0.094(4)	0.049(4)	0.054(4)	0.016(5)
C26	0.156(6)	0.316(13)	0.202(9)	0.113(9)	-0.051(6)	-0.169(8)
C27	0.227(9)	0.240(11)	0.058(4)	-0.062(5)	0.027(5)	-0.029(8)
C28	0.042(2)	0.036(2)	0.048(2)	-0.0119(18)	0.0037(17)	-0.0125(17)
C29	0.039(2)	0.040(2)	0.061(3)	-0.0072(19)	-0.0077(19)	-0.0034(17)
C30	0.050(3)	0.053(3)	0.068(3)	-0.009(2)	-0.002(2)	-0.007(2)
C31	0.061(3)	0.036(2)	0.053(3)	-0.0095(19)	0.000(2)	0.000(2)
C32	0.061(3)	0.043(2)	0.056(3)	-0.012(2)	0.000(2)	-0.015(2)
C33	0.055(3)	0.037(2)	0.054(3)	-0.0110(19)	-0.006(2)	-0.0092(19)
C34	0.079(4)	0.065(4)	0.101(4)	-0.008(3)	-0.043(3)	-0.002(3)
C35	0.068(4)	0.323(14)	0.333(15)	-0.206(13)	-0.067(6)	0.041(6)
C36	0.284(13)	0.149(9)	0.423(18)	-0.119(11)	-0.314(14)	0.064(9)
C37	0.293(12)	0.260(12)	0.132(6)	0.080(7)	-0.147(7)	-0.132(10)
O38	0.095(3)	0.0418(19)	0.082(2)	-0.0093(17)	0.004(2)	0.0021(17)
C39	0.104(4)	0.061(3)	0.141(5)	-0.033(3)	0.016(4)	0.009(3)

Table E99. continued...

C40	0.083(4)	0.051(3)	0.117(5)	-0.027(3)	0.008(3)	-0.028(3)
C41	0.112(4)	0.078(3)	0.255(9)	-0.092(5)	-0.047(5)	0.006(3)
C42	0.099(4)	0.070(4)	0.179(5)	-0.033(3)	-0.024(4)	-0.035(3)
C43	0.178(8)	0.133(6)	0.149(6)	0.062(5)	-0.016(5)	-0.114(6)
C44	0.042(2)	0.038(2)	0.042(2)	-0.0117(17)	-0.0125(17)	-0.0038(16)
C45	0.037(2)	0.037(2)	0.045(2)	-0.0068(17)	-0.0090(17)	-0.0051(16)
C46	0.042(2)	0.050(3)	0.064(3)	-0.015(2)	-0.014(2)	-0.0043(19)
C47	0.035(2)	0.059(3)	0.070(3)	-0.030(2)	-0.018(2)	0.001(2)
C48	0.039(2)	0.086(4)	0.075(3)	-0.044(3)	0.002(2)	-0.006(2)
C49	0.048(3)	0.073(3)	0.057(3)	-0.016(2)	-0.001(2)	-0.013(2)
C50	0.045(2)	0.048(2)	0.051(2)	-0.0053(19)	-0.0085(19)	-0.0042(19)
C51	0.067(3)	0.051(3)	0.104(4)	-0.039(3)	-0.022(3)	0.006(2)
C52	0.102(3)	0.090(4)	0.201(6)	-0.012(4)	-0.073(4)	0.021(3)
C53	0.399(16)	0.096(6)	0.185(8)	-0.095(6)	-0.119(9)	0.083(8)
C54	0.156(6)	0.046(3)	0.195(6)	0.004(3)	0.040(5)	0.018(3)
O55	0.0428(19)	0.118(3)	0.100(3)	-0.048(3)	0.0056(19)	0.007(2)
C56	0.041(3)	0.187(9)	0.157(6)	-0.060(6)	-0.017(4)	-0.022(4)
C57	0.063(3)	0.133(5)	0.046(3)	-0.008(3)	0.024(2)	-0.028(3)
C58	0.405(18)	0.125(7)	0.093(6)	-0.032(5)	0.084(8)	-0.057(8)
C59	0.111(5)	0.458(19)	0.101(5)	0.151(8)	0.021(4)	0.072(8)
C60	0.401(16)	0.228(10)	0.137(6)	0.024(6)	-0.055(8)	-0.248(12)
C61	0.040(2)	0.037(2)	0.046(2)	-0.0112(18)	-0.0039(18)	-0.0040(17)
C62	0.044(2)	0.046(3)	0.067(3)	-0.015(2)	0.001(2)	-0.013(2)
C63	0.060(3)	0.035(2)	0.068(3)	-0.013(2)	0.008(2)	-0.013(2)
C64	0.071(3)	0.035(2)	0.061(3)	-0.001(2)	0.004(2)	-0.001(2)
C65	0.061(3)	0.047(3)	0.059(3)	-0.011(2)	-0.021(2)	0.006(2)
C66	0.059(3)	0.046(3)	0.062(3)	-0.017(2)	-0.008(2)	-0.014(2)
C67	0.083(4)	0.073(4)	0.110(5)	-0.012(3)	-0.047(4)	0.006(3)
C68	0.325(14)	0.121(7)	0.371(16)	-0.124(9)	-0.280(14)	0.068(8)
C69	0.220(9)	0.153(6)	0.236(10)	0.052(6)	-0.178(9)	-0.053(6)
C70	0.063(4)	0.290(12)	0.198(7)	-0.044(7)	-0.035(4)	-0.045(5)
O71	0.086(2)	0.0416(18)	0.088(2)	0.0000(17)	-0.0083(19)	0.0132(17)
C72	0.091(4)	0.074(4)	0.147(6)	-0.060(4)	-0.017(4)	0.021(3)
C73	0.077(3)	0.037(2)	0.092(4)	-0.003(2)	-0.008(3)	-0.023(2)
C74	0.167(7)	0.176(8)	0.128(6)	-0.015(5)	0.017(5)	-0.105(6)
C75	0.186(7)	0.191(8)	0.181(7)	-0.134(6)	0.051(6)	-0.125(6)
C76	0.073(4)	0.066(4)	0.528(19)	-0.051(7)	-0.070(7)	-0.034(3)
C77	0.174(7)	0.074(4)	0.052(3)	-0.019(3)	0.005(4)	-0.047(4)
O78	0.203(5)	0.196(5)	0.062(3)	-0.037(3)	0.006(3)	-0.120(5)
C79	0.58(3)	0.171(9)	0.146(8)	-0.009(7)	-0.039(11)	-0.235(14)
C80	0.188(9)	0.140(7)	0.307(14)	-0.056(8)	0.075(9)	0.045(7)
C81	0.119(5)	0.131(6)	0.063(4)	-0.051(4)	0.013(3)	-0.050(4)
O82	0.109(3)	0.163(4)	0.059(2)	-0.020(3)	-0.007(2)	-0.065(3)
C83	0.122(5)	0.200(9)	0.143(6)	-0.093(6)	0.016(4)	-0.011(5)

Table E99. continued...

C84	0.299(13)	0.100(5)	0.158(8)	0.028(5)	-0.042(8)	-0.029(7)

Table E100. Hydrogen coordinates and isotropic displacement parameters ($Å^2$) for **2E•$(C_3H_6O)_2$**.

Atom	x	y	z	U_{eq}
H3	0.775(3)	0.183(3)	0.721(3)	0.088(10)
H8	0.7421	0.1095	0.2826	0.089
H11	0.959(2)	0.320(2)	0.500(2)	0.042(8)
H13	1.0612	0.1226	0.4555	0.055
H17	1.0478	0.4135	0.2255	0.064
H19A	1.1071	-0.0356	0.4851	0.373
H19B	1.1726	-0.1351	0.4412	0.373
H19C	1.0943	-0.0363	0.3697	0.373
H20A	1.2179	-0.0286	0.2428	0.241
H20B	1.2895	-0.1311	0.3170	0.241
H20C	1.3500	-0.0307	0.2428	0.241
H21A	1.2802	0.0118	0.5106	0.440
H21B	1.3923	-0.0025	0.4257	0.440
H21C	1.3305	-0.1042	0.4925	0.440
H23A	1.5317	0.1605	0.2080	0.195
H23B	1.4491	0.1467	0.3196	0.195
H23C	1.4480	0.2635	0.2339	0.195
H25A	1.1217	0.5260	0.1053	0.265
H25B	1.0829	0.4660	0.0363	0.265
H25C	1.1823	0.5377	-0.0146	0.265
H26A	1.3270	0.4691	0.1475	0.371
H26B	1.3701	0.4783	0.0246	0.371
H26C	1.4097	0.3695	0.1138	0.371
H27A	1.2189	0.2904	-0.0040	0.270
H27B	1.3455	0.2609	0.0248	0.270
H27C	1.3091	0.3703	-0.0651	0.270
H29	0.7324	0.4092	0.3309	0.059
H33	0.9983	0.5047	0.3967	0.059
H35A	0.4442	0.6564	0.2388	0.331
H35B	0.5157	0.7150	0.2837	0.331
H35C	0.4907	0.5956	0.3492	0.331
H36A	0.5515	0.5109	0.1503	0.377
H36B	0.6062	0.4360	0.2534	0.377
H36C	0.6872	0.4780	0.1433	0.377
H37A	0.5784	0.6877	0.0674	0.345
H37B	0.7127	0.6458	0.0646	0.345
H37C	0.6527	0.7455	0.1101	0.345
H39A	0.6150	0.8894	0.2790	0.166
H39B	0.6727	0.8079	0.3780	0.166
H39C	0.5800	0.7719	0.3342	0.166

Table E100. continued...

H41A	0.9513	0.8545	0.3678	0.207
H41B	0.8909	0.7590	0.4529	0.207
H41C	0.8293	0.8378	0.3552	0.207
H42A	1.1233	0.6170	0.3058	0.169
H42B	1.0724	0.6283	0.4221	0.169
H42C	1.1297	0.7238	0.3337	0.169
H43A	1.0094	0.7480	0.1407	0.249
H43B	1.0274	0.8471	0.1753	0.249
H43C	0.9017	0.8340	0.1688	0.249
H44	0.5887(19)	-0.0143(18)	0.501(2)	0.011(6)
H46	0.4723	0.1776	0.5292	0.062
H50	0.4825	-0.1140	0.7514	0.061
H52A	0.1511	0.4084	0.5352	0.205
H52B	0.1145	0.2963	0.6112	0.205
H52C	0.1867	0.3024	0.4959	0.205
H53A	0.2128	0.4205	0.6882	0.333
H53B	0.3093	0.3317	0.7470	0.333
H53C	0.1856	0.3027	0.7575	0.333
H54A	0.3429	0.4415	0.4974	0.238
H54B	0.3976	0.3361	0.4595	0.238
H54C	0.4446	0.3568	0.5519	0.238
H56A	0.0039	0.1416	0.7800	0.188
H56B	0.0856	0.0377	0.7539	0.188
H56C	0.0838	0.1540	0.6671	0.188
H58A	0.1770	0.0203	0.9556	0.338
H58B	0.3008	0.0082	0.9851	0.338
H58C	0.2241	-0.0840	1.0456	0.338
H59A	0.4443	-0.1174	0.9535	0.462
H59B	0.4346	-0.2022	0.8956	0.462
H59C	0.3664	-0.2090	1.0125	0.462
H60A	0.1433	-0.0987	0.8603	0.360
H60B	0.1838	-0.1975	0.9582	0.360
H60C	0.2452	-0.1930	0.8397	0.360
H62	0.5351	-0.2020	0.5863	0.064
H66	0.7934	-0.1086	0.6606	0.066
H68A	0.9076	-0.1322	0.7450	0.361
H68B	0.9878	-0.2086	0.8297	0.361
H68C	0.8519	-0.1944	0.8618	0.361
H69A	0.8563	-0.4063	0.9064	0.306
H69B	0.9912	-0.4096	0.8897	0.306
H69C	0.9389	-0.4620	0.8226	0.306
H70A	1.0136	-0.2148	0.6312	0.278
H70B	1.0358	-0.3432	0.6523	0.278
H70C	1.0873	-0.2921	0.7210	0.278

Table E100. continued...

H72A	0.9054	-0.5900	0.6986	0.155
H72B	0.9406	-0.4737	0.6358	0.155
H72C	0.8346	-0.5075	0.6089	0.155
H74A	0.6171	-0.5253	0.8137	0.237
H74B	0.4989	-0.5403	0.7909	0.237
H74C	0.5034	-0.4411	0.8320	0.237
H75A	0.6324	-0.4348	0.5251	0.245
H75B	0.5648	-0.5304	0.5990	0.245
H75C	0.6914	-0.5307	0.6152	0.245
H76A	0.4490	-0.3201	0.5880	0.336
H76B	0.4188	-0.3230	0.7103	0.336
H76C	0.4026	-0.4226	0.6754	0.336
H79A	0.7624	0.3392	-0.0240	0.424
H79B	0.8701	0.2847	-0.0915	0.424
H79C	0.8890	0.3482	-0.0156	0.424
H80A	0.9601	0.0695	0.1419	0.367
H80B	1.0167	0.1768	0.0928	0.367
H80C	1.0050	0.1098	0.0172	0.367
H83A	0.5811	0.2203	0.8596	0.229
H83B	0.5426	0.1541	0.9804	0.229
H83C	0.5428	0.1051	0.8865	0.229
H84A	0.7816	-0.0478	1.0053	0.303
H84B	0.6681	-0.0639	0.9745	0.303
H84C	0.6594	-0.0121	1.0683	0.303

Table E101. Crystal and structure refinement data for **2E•($C_6H_{15}N$)$_2$**.

Identification code	**2E•($C_6H_{15}N$)$_2$**
Empirical formula	$C_{80}H_{126}N_2O_8$
Formula weight	1243.83 g/mole
Temperature	173(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	C2/c
Unit cell dimensions	a = 27.717(6) Å b = 8.7770(18) Å c = 33.515(7) Å α = 90.00° β = 109.77(3)° γ = 90.00°
Volume	7673(3) Å^3
Z	4
Density (calculated)	1.077 Mg/m^3
Absorption coefficient	0.068 mm^{-1}
F(000)	2736
Crystal Size	1.00 mm x 0.38 mm x 0.36 mm
Color	wine red
Theta range for data collection	1.66° to 28.35°
Index ranges	$-36 \le h \le 26$, $-11 \le k \le 11$, $-43 \le l \le 44$
Reflections collected / Unique	23975 / 9054 [R_{int} = 0.0296]
Refinement method	Full-matrix-least-squares on F^2
Data / Restraints / Parameters	9054 / 0 / 500
Goodness-of-fit on F^2	1.064
Final R indices [$I>2\sigma(I)$]	R_1 = 0.0633, wR_2 = 0.1707
R indices (all data)	R_1 = 0.0951, wR_2 = 0.1887
Extinction Coefficient	0.00049(19)
Largest diff. peak and hole	0.433 and -0.370 e/A^3

Table E102. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for **2E•($C_6H_{15}N$)$_2$**. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U_{eq}	Occ.
O(1)	333(1)	7426(1)	-246(1)	29(1)	1
C(1)	163(1)	8647(2)	-154(1)	21(1)	1
O(2)	380(1)	7681(1)	551(1)	27(1)	1
C(2)	206(1)	8823(2)	314(1)	21(1)	1
C(3)	58(1)	10185(2)	453(1)	20(1)	1
C(4)	72(1)	10421(2)	906(1)	21(1)	1
C(5)	596(1)	10269(2)	1252(1)	24(1)	1
C(6)	632(1)	10407(2)	1673(1)	31(1)	1
C(7)	1094(1)	10372(3)	2009(1)	39(1)	1
O(8)	2007(1)	9926(3)	2222(1)	80(1)	1
C(8)	1536(1)	10111(3)	1906(1)	42(1)	1
C(9)	1530(1)	10092(2)	1483(1)	30(1)	1
C(10)	1051(1)	10160(2)	1165(1)	25(1)	1
C(11)	1085(1)	10706(4)	2461(1)	60(1)	1
C(12)	634(2)	11745(8)	2432(1)	163(3)	1
C(13)	1016(3)	9242(6)	2675(1)	165(3)	1
C(14)	1546(1)	11580(5)	2733(1)	94(1)	1
C(15)	2024(1)	10116(2)	1368(1)	35(1)	1
C(16)	2360(1)	11442(4)	1583(1)	72(1)	1
C(17)	2333(1)	8637(4)	1490(1)	69(1)	1
C(18)	1899(1)	10301(4)	889(1)	70(1)	1
C(19)	2088(2)	8414(7)	2380(2)	152(3)	1
C(20)	-352(1)	9493(2)	989(1)	22(1)	1
C(21)	-846(1)	10089(2)	860(1)	25(1)	1
C(22)	-1264(1)	9285(2)	893(1)	26(1)	1
O(23)	-1585(1)	6922(2)	1065(1)	42(1)	1
C(23)	-1165(1)	7812(2)	1070(1)	29(1)	1
C(24)	-666(1)	7231(2)	1248(1)	29(1)	1
C(25)	-271(1)	8084(2)	1186(1)	26(1)	1
C(26)	-1799(1)	10028(2)	748(1)	33(1)	1
C(27)	-1762(1)	11747(3)	733(2)	88(1)	1
C(28)	-2119(2)	9478(6)	317(1)	140(2)	1
C(29)	-2082(1)	9752(4)	1057(1)	96(1)	1
C(30)	-513(1)	5757(2)	1515(1)	38(1)	1
C(31)	-917(1)	5160(3)	1698(1)	70(1)	1
C(32)	-31(1)	6084(3)	1898(1)	56(1)	1
C(33)	-384(1)	4496(3)	1249(1)	52(1)	1
C(34)	-1725(1)	5844(3)	729(1)	54(1)	1
N(50)	880(1)	5147(2)	454(1)	27(1)	1

Table E102. continued...

C(51)	690(1)	4286(2)	42(1)	29(1)	1
C(52)	983(1)	2843(2)	28(1)	34(1)	1
C(53)	794(1)	4245(2)	807(1)	36(1)	1
C(54)	973(1)	5028(3)	1233(1)	47(1)	1
C(55)	1425(1)	5667(3)	572(1)	37(1)	1
C(56)	1531(1)	6524(3)	223(1)	43(1)	1

Table E103. Bond lengths (Å) and angles (°) for **2E•($C_6H_{15}N$)$_2$**.

Atoms	Length	Angle
O(1)-C(1)	1.251(2)	
C(1)-C(3)#1	1.420(2)	
C(1)-C(2)	1.540(2)	
O(2)-C(2)	1.268(2)	
C(2)-C(3)	1.394(2)	
C(3)-C(1)#1	1.420(2)	
C(3)-C(4)	1.520(2)	
C(4)-C(5)	1.529(2)	
C(4)-C(20)	1.530(2)	
C(4)-H(4)	0.997(19)	
C(5)-C(6)	1.385(3)	
C(5)-C(10)	1.390(2)	
C(6)-C(7)	1.390(3)	
C(6)-H(6)	0.93(2)	
C(7)-C(8)	1.402(3)	
C(7)-C(11)	1.552(3)	
O(8)-C(8)	1.385(3)	
O(8)-C(19)	1.418(5)	
C(8)-C(9)	1.411(3)	
C(9)-C(10)	1.396(3)	
C(9)-C(15)	1.543(3)	
C(10)-H(10)	0.958(19)	
C(11)-C(14)	1.505(4)	
C(11)-C(13)	1.514(6)	
C(11)-C(12)	1.524(5)	
C(12)-H(12A)	0.9600	
C(12)-H(12B)	0.9600	
C(12)-H(12C)	0.9600	
C(13)-H(13A)	0.9600	
C(13)-H(13B)	0.9600	
C(13)-H(13C)	0.9600	
C(14)-H(14A)	0.9600	
C(14)-H(14B)	0.9600	
C(14)-H(14C)	0.9600	
C(15)-C(16)	1.512(3)	
C(15)-C(18)	1.531(3)	
C(15)-C(17)	1.533(3)	
C(16)-H(16A)	0.9600	
C(16)-H(16B)	0.9600	
C(16)-H(16C)	0.9600	
C(17)-H(17A)	0.9600	

Table E103. continued...

C(17)-H(17B)	0.9600
C(17)-H(17C)	0.9600
C(18)-H(18A)	0.9600
C(18)-H(18B)	0.9600
C(18)-H(18C)	0.9600
C(19)-H(19A)	0.9600
C(19)-H(19B)	0.9600
C(19)-H(19C)	0.9600
C(20)-C(25)	1.383(3)
C(20)-C(21)	1.393(2)
C(21)-C(22)	1.393(2)
C(21)-H(21)	1.00(2)
C(22)-C(23)	1.410(3)
C(22)-C(26)	1.541(3)
O(23)-C(23)	1.398(2)
O(23)-C(34)	1.421(3)
C(23)-C(24)	1.404(3)
C(24)-C(25)	1.400(3)
C(24)-C(30)	1.548(3)
C(25)-H(25)	0.95(2)
C(26)-C(28)	1.498(4)
C(26)-C(29)	1.514(3)
C(26)-C(27)	1.515(3)
C(27)-H(27A)	0.9600
C(27)-H(27B)	0.9600
C(27)-H(27C)	0.9600
C(28)-H(28A)	0.9600
C(28)-H(28B)	0.9600
C(28)-H(28C)	0.9600
C(29)-H(29A)	0.9600
C(29)-H(29B)	0.9600
C(29)-H(29C)	0.9600
C(30)-C(32)	1.535(3)
C(30)-C(33)	1.537(3)
C(30)-C(31)	1.541(3)
C(31)-H(31A)	0.9600
C(31)-H(31B)	0.9600
C(31)-H(31C)	0.9600
C(32)-H(32A)	0.9600
C(32)-H(32B)	0.9600
C(32)-H(32C)	0.9600
C(33)-H(33A)	0.9600
C(33)-H(33B)	0.9600
C(33)-H(33C)	0.9600

Table E103. continued...

C(34)-H(34A)	0.9600
C(34)-H(34B)	0.9600
C(34)-H(34C)	0.9600
N(50)-C(55)	1.498(3)
N(50)-C(51)	1.505(2)
N(50)-C(53)	1.506(3)
N(50)-H(50)	0.93(3)
C(51)-C(52)	1.514(3)
C(51)-H(51A)	0.97(2)
C(51)-H(51B)	1.01(2)
C(52)-H(52A)	0.98(3)
C(52)-H(52B)	0.99(2)
C(52)-H(52C)	0.98(2)
C(53)-C(54)	1.509(3)
C(53)-H(53A)	1.02(3)
C(53)-H(53B)	0.98(3)
C(54)-H(54A)	1.04(3)
C(54)-H(54B)	1.01(3)
C(54)-H(54C)	0.95(3)
C(55)-C(56)	1.503(3)
C(55)-H(55A)	1.03(3)
C(55)-H(55B)	1.04(2)
C(56)-H(56A)	0.98(3)
C(56)-H(56B)	1.00(3)
C(56)-H(56C)	1.01(3)

O(1)-C(1)-C(3)#1	123.57(15)
O(1)-C(1)-C(2)	115.57(15)
C(3)#1-C(1)-C(2)	120.86(14)
O(2)-C(2)-C(3)	124.11(15)
O(2)-C(2)-C(1)	116.16(14)
C(3)-C(2)-C(1)	119.74(14)
C(2)-C(3)-C(1)#1	119.27(15)
C(2)-C(3)-C(4)	122.64(15)
C(1)#1-C(3)-C(4)	117.79(14)
C(3)-C(4)-C(5)	116.32(14)
C(3)-C(4)-C(20)	110.31(14)
C(5)-C(4)-C(20)	113.98(14)
C(3)-C(4)-H(4)	104.6(10)
C(5)-C(4)-H(4)	105.4(11)
C(20)-C(4)-H(4)	105.0(11)
C(6)-C(5)-C(10)	117.70(17)
C(6)-C(5)-C(4)	119.11(16)
C(10)-C(5)-C(4)	122.82(15)

Table E103. continued...

C(5)-C(6)-C(7)	123.39(18)
C(5)-C(6)-H(6)	118.4(12)
C(7)-C(6)-H(6)	118.2(12)
C(6)-C(7)-C(8)	116.61(18)
C(6)-C(7)-C(11)	118.22(19)
C(8)-C(7)-C(11)	125.09(19)
C(8)-O(8)-C(19)	112.6(3)
O(8)-C(8)-C(7)	120.47(19)
O(8)-C(8)-C(9)	117.15(19)
C(7)-C(8)-C(9)	122.32(18)
C(10)-C(9)-C(8)	116.91(17)
C(10)-C(9)-C(15)	120.23(17)
C(8)-C(9)-C(15)	122.72(17)
C(5)-C(10)-C(9)	122.48(17)
C(5)-C(10)-H(10)	120.8(12)
C(9)-C(10)-H(10)	116.7(12)
C(14)-C(11)-C(13)	111.3(3)
C(14)-C(11)-C(12)	104.1(3)
C(13)-C(11)-C(12)	107.8(4)
C(14)-C(11)-C(7)	113.4(2)
C(13)-C(11)-C(7)	110.3(3)
C(12)-C(11)-C(7)	109.5(2)
C(11)-C(12)-H(12A)	109.5
C(11)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	109.5
C(11)-C(12)-H(12C)	109.5
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5
C(11)-C(13)-H(13A)	109.5
C(11)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	109.5
C(11)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
C(11)-C(14)-H(14A)	109.5
C(11)-C(14)-H(14B)	109.5
H(14A)-C(14)-H(14B)	109.5
C(11)-C(14)-H(14C)	109.5
H(14A)-C(14)-H(14C)	109.5
H(14B)-C(14)-H(14C)	109.5
C(16)-C(15)-C(18)	107.6(2)
C(16)-C(15)-C(17)	109.1(2)
C(18)-C(15)-C(17)	106.0(2)
C(16)-C(15)-C(9)	110.20(19)

Table E103. continued...

C(18)-C(15)-C(9)	111.00(16)
C(17)-C(15)-C(9)	112.7(2)
C(15)-C(16)-H(16A)	109.5
C(15)-C(16)-H(16B)	109.5
H(16A)-C(16)-H(16B)	109.5
C(15)-C(16)-H(16C)	109.5
H(16A)-C(16)-H(16C)	109.5
H(16B)-C(16)-H(16C)	109.5
C(15)-C(17)-H(17A)	109.5
C(15)-C(17)-H(17B)	109.5
H(17A)-C(17)-H(17B)	109.5
C(15)-C(17)-H(17C)	109.5
H(17A)-C(17)-H(17C)	109.5
H(17B)-C(17)-H(17C)	109.5
C(15)-C(18)-H(18A)	109.5
C(15)-C(18)-H(18B)	109.5
H(18A)-C(18)-H(18B)	109.5
C(15)-C(18)-H(18C)	109.5
H(18A)-C(18)-H(18C)	109.5
H(18B)-C(18)-H(18C)	109.5
O(8)-C(19)-H(19A)	109.5
O(8)-C(19)-H(19B)	109.5
H(19A)-C(19)-H(19B)	109.5
O(8)-C(19)-H(19C)	109.5
H(19A)-C(19)-H(19C)	109.5
H(19B)-C(19)-H(19C)	109.5
C(25)-C(20)-C(21)	117.95(16)
C(25)-C(20)-C(4)	123.48(16)
C(21)-C(20)-C(4)	118.57(15)
C(20)-C(21)-C(22)	122.81(17)
C(20)-C(21)-H(21)	116.3(12)
C(22)-C(21)-H(21)	120.9(12)
C(21)-C(22)-C(23)	116.71(16)
C(21)-C(22)-C(26)	119.88(17)
C(23)-C(22)-C(26)	123.40(16)
C(23)-O(23)-C(34)	112.65(16)
O(23)-C(23)-C(24)	119.94(17)
O(23)-C(23)-C(22)	117.66(17)
C(24)-C(23)-C(22)	122.40(17)
C(25)-C(24)-C(23)	116.77(17)
C(25)-C(24)-C(30)	117.23(17)
C(23)-C(24)-C(30)	125.99(17)
C(20)-C(25)-C(24)	122.71(17)
C(20)-C(25)-H(25)	117.4(13)

Table E103. continued...

C(24)-C(25)-H(25)	119.9(13)
C(28)-C(26)-C(29)	109.5(3)
C(28)-C(26)-C(27)	108.3(3)
C(29)-C(26)-C(27)	103.8(2)
C(28)-C(26)-C(22)	111.2(2)
C(29)-C(26)-C(22)	112.51(19)
C(27)-C(26)-C(22)	111.22(17)
C(26)-C(27)-H(27A)	109.5
C(26)-C(27)-H(27B)	109.5
H(27A)-C(27)-H(27B)	109.5
C(26)-C(27)-H(27C)	109.5
H(27A)-C(27)-H(27C)	109.5
H(27B)-C(27)-H(27C)	109.5
C(26)-C(28)-H(28A)	109.5
C(26)-C(28)-H(28B)	109.5
H(28A)-C(28)-H(28B)	109.5
C(26)-C(28)-H(28C)	109.5
H(28A)-C(28)-H(28C)	109.5
H(28B)-C(28)-H(28C)	109.5
C(26)-C(29)-H(29A)	109.5
C(26)-C(29)-H(29B)	109.5
H(29A)-C(29)-H(29B)	109.5
C(26)-C(29)-H(29C)	109.5
H(29A)-C(29)-H(29C)	109.5
H(29B)-C(29)-H(29C)	109.5
C(32)-C(30)-C(33)	108.3(2)
C(32)-C(30)-C(31)	105.8(2)
C(33)-C(30)-C(31)	109.3(2)
C(32)-C(30)-C(24)	108.56(18)
C(33)-C(30)-C(24)	109.80(17)
C(31)-C(30)-C(24)	114.84(19)
C(30)-C(31)-H(31A)	109.5
C(30)-C(31)-H(31B)	109.5
H(31A)-C(31)-H(31B)	109.5
C(30)-C(31)-H(31C)	109.5
H(31A)-C(31)-H(31C)	109.5
H(31B)-C(31)-H(31C)	109.5
C(30)-C(32)-H(32A)	109.5
C(30)-C(32)-H(32B)	109.5
H(32A)-C(32)-H(32B)	109.5
C(30)-C(32)-H(32C)	109.5
H(32A)-C(32)-H(32C)	109.5
H(32B)-C(32)-H(32C)	109.5
C(30)-C(33)-H(33A)	109.5

Table E103. continued...

C(30)-C(33)-H(33B)	109.5
H(33A)-C(33)-H(33B)	109.5
C(30)-C(33)-H(33C)	109.5
H(33A)-C(33)-H(33C)	109.5
H(33B)-C(33)-H(33C)	109.5
O(23)-C(34)-H(34A)	109.5
O(23)-C(34)-H(34B)	109.5
H(34A)-C(34)-H(34B)	109.5
O(23)-C(34)-H(34C)	109.5
H(34A)-C(34)-H(34C)	109.5
H(34B)-C(34)-H(34C)	109.5
C(55)-N(50)-C(51)	113.81(16)
C(55)-N(50)-C(53)	111.39(16)
C(51)-N(50)-C(53)	110.92(15)
C(55)-N(50)-H(50)	105.0(15)
C(51)-N(50)-H(50)	109.4(15)
C(53)-N(50)-H(50)	105.9(15)
N(50)-C(51)-C(52)	115.14(16)
N(50)-C(51)-H(51A)	105.1(13)
C(52)-C(51)-H(51A)	111.0(13)
N(50)-C(51)-H(51B)	102.2(12)
C(52)-C(51)-H(51B)	112.1(13)
H(51A)-C(51)-H(51B)	110.8(18)
C(51)-C(52)-H(52A)	109.1(15)
C(51)-C(52)-H(52B)	111.4(13)
H(52A)-C(52)-H(52B)	106(2)
C(51)-C(52)-H(52C)	110.9(14)
H(52A)-C(52)-H(52C)	110.2(19)
H(52B)-C(52)-H(52C)	109.6(19)
N(50)-C(53)-C(54)	114.50(19)
N(50)-C(53)-H(53A)	106.7(13)
C(54)-C(53)-H(53A)	111.6(13)
N(50)-C(53)-H(53B)	104.6(14)
C(54)-C(53)-H(53B)	110.8(14)
H(53A)-C(53)-H(53B)	108(2)
C(53)-C(54)-H(54A)	108.0(14)
C(53)-C(54)-H(54B)	114.1(14)
H(54A)-C(54)-H(54B)	108(2)
C(53)-C(54)-H(54C)	109.3(16)
H(54A)-C(54)-H(54C)	113(2)
H(54B)-C(54)-H(54C)	104(2)
N(50)-C(55)-C(56)	113.11(18)
N(50)-C(55)-H(55A)	106.0(13)
C(56)-C(55)-H(55A)	110.6(13)

Table E103. continued...

N(50)-C(55)-H(55B)	105.7(13)
C(56)-C(55)-H(55B)	110.8(13)
H(55A)-C(55)-H(55B)	110.5(18)
C(55)-C(56)-H(56A)	110.6(17)
C(55)-C(56)-H(56B)	110.6(16)
H(56A)-C(56)-H(56B)	110(2)
C(55)-C(56)-H(56C)	114.6(16)
H(56A)-C(56)-H(56C)	106(2)
H(56B)-C(56)-H(56C)	105(2)

Symmetry transformations used to generate equivalent atoms:
#1 -x, -y+2, -z

Table E104. Anisotropic displacement parameters ($Å^2$ x 10^3) for **2E•($C_6H_{15}N)_2$**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+...+2hka^*b^*U_{12}]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O(1)	35(1)	24(1)	29(1)	1(1)	11(1)	10(1)
C(1)	16(1)	21(1)	27(1)	-1(1)	7(1)	-1(1)
O(2)	31(1)	22(1)	27(1)	3(1)	8(1)	6(1)
C(2)	16(1)	20(1)	27(1)	2(1)	5(1)	-1(1)
C(3)	15(1)	22(1)	24(1)	0(1)	6(1)	0(1)
C(4)	18(1)	21(1)	24(1)	-1(1)	7(1)	1(1)
C(5)	20(1)	23(1)	26(1)	0(1)	5(1)	0(1)
C(6)	22(1)	41(1)	28(1)	-3(1)	8(1)	-2(1)
C(7)	28(1)	62(1)	25(1)	-2(1)	5(1)	-7(1)
O(8)	25(1)	166(2)	37(1)	18(1)	-4(1)	7(1)
C(8)	21(1)	67(2)	31(1)	6(1)	0(1)	-1(1)
C(9)	20(1)	35(1)	33(1)	3(1)	6(1)	1(1)
C(10)	22(1)	26(1)	26(1)	1(1)	7(1)	1(1)
C(11)	39(1)	111(2)	26(1)	-15(1)	9(1)	-20(1)
C(12)	90(3)	320(8)	65(2)	-93(4)	7(2)	60(4)
C(13)	296(8)	169(5)	65(2)	-34(3)	105(4)	-131(5)
C(14)	73(2)	154(4)	53(2)	-39(2)	21(2)	-48(2)
C(15)	17(1)	47(1)	37(1)	-1(1)	6(1)	2(1)
C(16)	52(2)	82(2)	89(2)	-25(2)	33(2)	-31(2)
C(17)	43(2)	74(2)	87(2)	-1(2)	17(1)	20(1)
C(18)	29(1)	138(3)	49(1)	14(2)	20(1)	10(2)
C(19)	78(3)	245(6)	140(4)	144(4)	46(3)	77(3)
C(20)	21(1)	24(1)	22(1)	-2(1)	8(1)	-1(1)
C(21)	22(1)	24(1)	29(1)	2(1)	9(1)	1(1)
C(22)	22(1)	27(1)	31(1)	-3(1)	11(1)	-2(1)
O(23)	37(1)	31(1)	70(1)	-4(1)	34(1)	-9(1)
C(23)	28(1)	26(1)	38(1)	-2(1)	20(1)	-4(1)
C(24)	34(1)	24(1)	33(1)	1(1)	17(1)	1(1)
C(25)	24(1)	26(1)	29(1)	2(1)	10(1)	3(1)
C(26)	21(1)	34(1)	42(1)	0(1)	9(1)	1(1)
C(27)	39(2)	41(2)	195(4)	17(2)	55(2)	14(1)
C(28)	108(3)	159(4)	84(2)	-68(3)	-57(2)	96(3)
C(29)	71(2)	109(3)	142(3)	68(2)	79(2)	56(2)
C(30)	46(1)	28(1)	46(1)	9(1)	23(1)	5(1)
C(31)	82(2)	54(2)	94(2)	36(2)	58(2)	11(2)
C(32)	76(2)	47(2)	40(1)	16(1)	13(1)	3(1)
C(33)	62(2)	30(1)	64(2)	2(1)	22(1)	9(1)
C(34)	38(1)	39(1)	87(2)	-14(1)	25(1)	-15(1)
N(50)	22(1)	23(1)	36(1)	-1(1)	7(1)	4(1)

Table E104. continued...

C(51)	25(1)	24(1)	35(1)	0(1)	6(1)	3(1)
C(52)	33(1)	27(1)	43(1)	-4(1)	13(1)	5(1)
C(53)	37(1)	33(1)	40(1)	6(1)	15(1)	6(1)
C(54)	44(1)	57(2)	36(1)	2(1)	10(1)	8(1)
C(55)	21(1)	37(1)	47(1)	-2(1)	5(1)	-1(1)
C(56)	28(1)	41(1)	60(2)	1(1)	16(1)	-3(1)

Table E105. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for **2E•($C_6H_{15}N$)$_2$**.

Atom	x	y	z	U_{eq}	Occ.
H(4)	-30(7)	11510(20)	916(6)	20(5)	1
H(6)	333(8)	10570(20)	1734(6)	27(5)	1
H(10)	1048(7)	10160(20)	878(6)	22(5)	1
H(12A)	628	11950	2712	245	1
H(12B)	320	11255	2268	245	1
H(12C)	669	12686	2298	245	1
H(13A)	1012	9464	2954	248	1
H(13B)	1295	8562	2695	248	1
H(13C)	698	8771	2511	248	1
H(14A)	1519	11748	3008	141	1
H(14B)	1562	12542	2602	141	1
H(14C)	1851	11006	2763	141	1
H(16A)	2665	11445	1510	108	1
H(16B)	2450	11345	1885	108	1
H(16C)	2176	12378	1492	108	1
H(17A)	2635	8708	1412	104	1
H(17B)	2127	7796	1343	104	1
H(17C)	2430	8481	1790	104	1
H(18A)	2212	10312	826	106	1
H(18B)	1719	11241	798	106	1
H(18C)	1689	9466	744	106	1
H(19A)	2418	8343	2598	228	1
H(19B)	2074	7727	2154	228	1
H(19C)	1826	8147	2496	228	1
H(21)	-890(8)	11140(30)	735(6)	30(5)	1
H(25)	71(9)	7710(20)	1285(6)	33(5)	1
H(27A)	-2100	12179	641	131	1
H(27B)	-1590	12028	539	131	1
H(27C)	-1571	12125	1011	131	1
H(28A)	-2450	9957	234	209	1
H(28B)	-2160	8393	323	209	1
H(28C)	-1953	9731	117	209	1
H(29A)	-2413	10233	953	144	1
H(29B)	-1889	10172	1328	144	1
H(29C)	-2125	8676	1085	144	1
H(31A)	-792	4249	1859	105	1
H(31B)	-1229	4939	1471	105	1
H(31C)	-981	5921	1880	105	1
H(32A)	69	5179	2067	83(10)	1
H(32B)	-103	6881	2066	81(10)	1

Table E105. continued...

H(32C)	243	6398	1802	52(7)	1
H(33A)	-289	3585	1415	86(10)	1
H(33B)	-104	4820	1162	74(10)	1
H(33C)	-678	4296	1003	66(9)	1
H(34A)	-2015	5263	739	91(11)	1
H(34B)	-1442	5172	758	78(10)	1
H(34C)	-1814	6369	463	115(15)	1
H(50)	691(9)	6030(30)	431(7)	45(7)	1
H(51A)	709(8)	5010(20)	-171(7)	35(6)	1
H(51B)	321(9)	4070(20)	13(7)	34(5)	1
H(52A)	845(9)	2400(30)	-256(8)	49(7)	1
H(52B)	933(9)	2060(30)	222(7)	40(6)	1
H(52C)	1351(10)	3060(30)	100(7)	43(6)	1
H(53A)	972(9)	3230(30)	817(7)	46(6)	1
H(53B)	423(10)	4070(30)	711(8)	49(7)	1
H(54A)	832(10)	4420(30)	1435(8)	49(7)	1
H(54B)	856(10)	6120(30)	1225(8)	50(7)	1
H(54C)	1335(11)	5100(30)	1332(8)	53(7)	1
H(55A)	1645(9)	4700(30)	648(7)	45(6)	1
H(55B)	1490(9)	6350(30)	838(8)	44(6)	1
H(56A)	1875(12)	6970(30)	325(9)	68(8)	1
H(56B)	1271(11)	7350(30)	109(9)	62(8)	1
H(56C)	1516(11)	5890(30)	-30(9)	64(8)	1

Table E106. Torsion angles (°) for **2E•($C_6H_{15}N$)$_2$**.

Atoms	Angle
O(1) C(1) C(2) O(2)	-4.1(2)
C(3)#1 C(1) C(2) O(2)	175.81(15)
O(1) C(1) C(2) C(3)	175.80(15)
C(3)#1 C(1) C(2) C(3)	-4.3(3)
O(2) C(2) C(3) C(1)#1	-175.88(16)
C(1) C(2) C(3) C(1)#1	4.2(3)
O(2) C(2) C(3) C(4)	-2.3(3)
C(1) C(2) C(3) C(4)	177.83(14)
C(2) C(3) C(4) C(5)	60.4(2)
C(1)#1 C(3) C(4) C(5)	-125.91(16)
C(2) C(3) C(4) C(20)	-71.4(2)
C(1)#1 C(3) C(4) C(20)	102.28(17)
C(3) C(4) C(5) C(6)	-177.30(17)
C(20) C(4) C(5) C(6)	-47.2(2)
C(3) C(4) C(5) C(10)	9.8(2)
C(20) C(4) C(5) C(10)	139.92(17)
C(10) C(5) C(6) C(7)	-3.0(3)
C(4) C(5) C(6) C(7)	-176.25(19)
C(5) C(6) C(7) C(8)	-3.0(3)
C(5) C(6) C(7) C(11)	173.8(2)
C(19) O(8) C(8) C(7)	83.3(4)
C(19) O(8) C(8) C(9)	-99.5(3)
C(6) C(7) C(8) O(8)	-174.4(2)
C(11) C(7) C(8) O(8)	9.0(4)
C(6) C(7) C(8) C(9)	8.6(4)
C(11) C(7) C(8) C(9)	-168.0(2)
O(8) C(8) C(9) C(10)	175.2(2)
C(7) C(8) C(9) C(10)	-7.7(3)
O(8) C(8) C(9) C(15)	-9.2(3)
C(7) C(8) C(9) C(15)	167.9(2)
C(6) C(5) C(10) C(9)	4.0(3)
C(4) C(5) C(10) C(9)	176.93(17)
C(8) C(9) C(10) C(5)	1.2(3)
C(15) C(9) C(10) C(5)	-174.56(17)
C(6) C(7) C(11) C(14)	-143.8(3)
C(8) C(7) C(11) C(14)	32.8(4)
C(6) C(7) C(11) C(13)	90.5(4)
C(8) C(7) C(11) C(13)	-92.9(4)
C(6) C(7) C(11) C(12)	-28.0(4)
C(8) C(7) C(11) C(12)	148.6(4)
C(10) C(9) C(15) C(16)	122.1(2)

Table E106. continued...

C(8) C(9) C(15) C(16)	-53.4(3)
C(10) C(9) C(15) C(18)	3.0(3)
C(8) C(9) C(15) C(18)	-172.5(2)
C(10) C(9) C(15) C(17)	-115.8(2)
C(8) C(9) C(15) C(17)	68.7(3)
C(3) C(4) C(20) C(25)	97.89(19)
C(5) C(4) C(20) C(25)	-35.1(2)
C(3) C(4) C(20) C(21)	-81.87(19)
C(5) C(4) C(20) C(21)	145.11(16)
C(25) C(20) C(21) C(22)	-4.5(3)
C(4) C(20) C(21) C(22)	175.27(16)
C(20) C(21) C(22) C(23)	0.4(3)
C(20) C(21) C(22) C(26)	179.27(16)
C(34) O(23) C(23) C(24)	-80.5(2)
C(34) O(23) C(23) C(22)	99.6(2)
C(21) C(22) C(23) O(23)	-173.19(16)
C(26) C(22) C(23) O(23)	8.0(3)
C(21) C(22) C(23) C(24)	6.9(3)
C(26) C(22) C(23) C(24)	-171.99(17)
O(23) C(23) C(24) C(25)	170.61(16)
C(22) C(23) C(24) C(25)	-9.5(3)
O(23) C(23) C(24) C(30)	-11.0(3)
C(22) C(23) C(24) C(30)	168.98(18)
C(21) C(20) C(25) C(24)	1.7(3)
C(4) C(20) C(25) C(24)	-178.10(16)
C(23) C(24) C(25) C(20)	5.0(3)
C(30) C(24) C(25) C(20)	-173.53(17)
C(21) C(22) C(26) C(28)	100.1(3)
C(23) C(22) C(26) C(28)	-81.1(4)
C(21) C(22) C(26) C(29)	-136.7(3)
C(23) C(22) C(26) C(29)	42.1(3)
C(21) C(22) C(26) C(27)	-20.7(3)
C(23) C(22) C(26) C(27)	158.1(2)
C(25) C(24) C(30) C(32)	42.3(2)
C(23) C(24) C(30) C(32)	-136.1(2)
C(25) C(24) C(30) C(33)	-75.9(2)
C(23) C(24) C(30) C(33)	105.7(2)
C(25) C(24) C(30) C(31)	160.5(2)
C(23) C(24) C(30) C(31)	-17.9(3)
C(55) N(50) C(51) C(52)	60.1(2)
C(53) N(50) C(51) C(52)	-66.4(2)
C(55) N(50) C(53) C(54)	52.3(2)
C(51) N(50) C(53) C(54)	-179.86(18)
C(51) N(50) C(55) C(56)	51.6(2)

Table E106. continued...

C(53) N(50) C(55) C(56)	177.90(19)

Symmetry transformations used to generate equivalent atoms:
#1 -x, -y+2, -z

Table E107. Hydrogen bonds, lengths (Å) and angles (°) for **2E•($C_6H_{15}N)_2$**.

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(50)-H(50)...O(2)	0.93(3)	1.80(3)	2.697(2)	163(2)
N(50)-H(50)...O(1)	0.93(3)	2.47(2)	3.065(2)	122.2(18)

Table E108. Crystal and structure refinement data for **2E•$C_6H_{16}N_2$** (from acetone).

Identification code	**2E•$C_6H_{16}N_2$** (from acetone)
Empirical formula	$C_{37}H_{56}NO_4$
Formula weight	578.83 g/mole
Temperature	173(2) K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group	$P\bar{1}$
Unit cell dimensions	a = 12.103(2) Å b = 12.247(2) Å c = 13.028(3) Å α = 71.00(3)° β = 75.98(3)° γ = 77.91(3)°
Volume	1753.5(6) $Å^3$
Z	2
Density (calculated)	1.096 Mg/m^3
Absorption coefficient	0.070 mm^{-1}
F(000)	634
Crystal Size	1.20 mm x 1.08 mm x 0.50 mm
Color	yellow-orange
Theta range for data collection	1.75° to 28.33°
Index ranges	$-15 \le h \le 15$, $-13 \le k \le 16$, $-16 \le l \le 15$
Reflections collected / Unique	11272 / 7891 [R_{int} = 0.0254]
Refinement method	Full-matrix-least-squares on F^2
Data / Restraints / Parameters	7891 / 0 / 547
Goodness-of-fit on F^2	1.038
Final R indices [$I>2\sigma(I)$]	R_1 = 0.0645, wR_2 = 0.1858
R indices (all data)	R_1 = 0.0839, wR_2 = 0.1958
Extinction Coefficient	0.002(3)
Largest diff. peak and hole	0.703 and -0.336 $e/Å^3$

Table E109. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for **2E•$C_6H_{16}N_2$** (from acetone). U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U_{eq}	Occ.
O(1)	5471(1)	11672(1)	652(1)	32(1)	1
C(1)	5289(1)	10859(1)	385(1)	22(1)	1
O(2)	5854(1)	7730(1)	858(1)	31(1)	1
C(2)	5480(1)	8830(1)	471(1)	24(1)	1
C(3)	5752(1)	9660(1)	824(1)	22(1)	1
C(4)	6575(1)	9353(1)	1619(1)	22(1)	1
C(5)	7722(1)	8671(1)	1188(1)	24(1)	1
C(6)	8242(1)	9072(2)	97(2)	26(1)	1
C(7)	9304(1)	8545(2)	-369(2)	28(1)	1
O(8)	11018(1)	7157(1)	4(1)	39(1)	1
C(8)	9884(1)	7623(2)	365(2)	29(1)	1
C(9)	9353(2)	7121(2)	1460(2)	32(1)	1
C(10)	8268(2)	7666(2)	1841(2)	28(1)	1
C(11)	9684(2)	8995(2)	-1632(2)	35(1)	1
C(12)	9923(3)	10249(2)	-1967(2)	67(1)	1
C(13)	10697(3)	8262(3)	-2160(2)	75(1)	1
C(14)	8673(2)	8966(3)	-2152(2)	64(1)	1
C(15)	9902(2)	5987(2)	2215(2)	45(1)	1
C(16)	11054(2)	6116(2)	2437(3)	55(1)	1
C(17)	9120(3)	5626(3)	3354(3)	88(1)	1
C(18)	10100(3)	5006(2)	1663(3)	70(1)	1
C(19)	11838(2)	7873(2)	-64(2)	46(1)	1
C(20)	6054(1)	8784(1)	2819(1)	25(1)	1
C(21)	6619(2)	8741(2)	3645(1)	28(1)	1
C(22)	6212(2)	8227(2)	4757(2)	31(1)	1
O(23)	4715(1)	7338(1)	6157(1)	44(1)	1
C(23)	5166(2)	7770(2)	5036(2)	33(1)	1
C(24)	4614(2)	7717(2)	4227(2)	34(1)	1
C(25)	5073(2)	8263(2)	3130(2)	29(1)	1
C(26)	6935(2)	8113(2)	5624(2)	40(1)	1
C(27)	8077(2)	8576(3)	5075(2)	53(1)	1
C(28)	7220(3)	6814(2)	6236(2)	56(1)	1
C(29)	6311(2)	8800(3)	6452(2)	52(1)	1
C(30)	3550(2)	7096(2)	4434(2)	50(1)	1
C(31)	2470(2)	7998(4)	4370(4)	86(1)	1
C(32)	3701(4)	6503(4)	3528(3)	76(1)	1
C(33)	3406(4)	6119(4)	5521(3)	93(1)	1
C(34)	3807(3)	8133(3)	6571(2)	61(1)	1
N(50)	5927(2)	5882(1)	326(2)	39(1)	1

Table E109. continued...

C(50)	4897(2)	5587(2)	125(3)	55(1)	1
C(51)	6344(4)	5076(2)	1302(3)	92(1)	1
C(52)	6834(3)	6082(3)	-635(3)	91(1)	1

Table E110. Bond lengths (Å) and angles (°) for **2E•$C_6H_{16}N_2$** (from acetone).

Atoms	Length	Angle
O(1)-C(1)	1.2318(19)	
C(1)-C(3)	1.437(2)	
C(1)-C(2)#1	1.527(2)	
O(2)-C(2)	1.299(2)	
O(2)-H(50)	1.26(3)	
C(2)-C(3)	1.367(2)	
C(2)-C(1)#1	1.527(2)	
C(3)-C(4)	1.515(2)	
C(4)-C(20)	1.525(2)	
C(4)-C(5)	1.543(2)	
C(4)-H(4)	0.97(2)	
C(5)-C(6)	1.378(3)	
C(5)-C(10)	1.389(2)	
C(6)-C(7)	1.402(2)	
C(6)-H(6)	0.95(2)	
C(7)-C(8)	1.404(3)	
C(7)-C(11)	1.540(3)	
O(8)-C(8)	1.401(2)	
O(8)-C(19)	1.426(2)	
C(8)-C(9)	1.406(3)	
C(9)-C(10)	1.396(2)	
C(9)-C(15)	1.542(3)	
C(10)-H(10)	0.93(2)	
C(11)-C(13)	1.521(3)	
C(11)-C(12)	1.524(3)	
C(11)-C(14)	1.546(3)	
C(12)-H(12A)	0.9600	
C(12)-H(12B)	0.9600	
C(12)-H(12C)	0.9600	
C(13)-H(13A)	0.9600	
C(13)-H(13B)	0.9600	
C(13)-H(13C)	0.9600	
C(14)-H(14A)	0.9600	
C(14)-H(14B)	0.9600	
C(14)-H(14C)	0.9600	
C(15)-C(17)	1.536(4)	
C(15)-C(16)	1.540(3)	
C(15)-C(18)	1.542(4)	
C(16)-H(16A)	1.01(3)	
C(16)-H(16B)	1.00(3)	
C(16)-H(16C)	1.01(2)	

Table E110. continued...

C(17)-H(17A)	0.98(4)
C(17)-H(17B)	1.12(5)
C(17)-H(17C)	1.05(4)
C(18)-H(18A)	1.09(4)
C(18)-H(18B)	1.11(4)
C(18)-H(18C)	1.00(3)
C(19)-H(19A)	0.95(3)
C(19)-H(19B)	1.01(3)
C(19)-H(19C)	1.06(3)
C(20)-C(25)	1.378(2)
C(20)-C(21)	1.391(2)
C(21)-C(22)	1.389(3)
C(21)-H(21)	0.97(2)
C(22)-C(23)	1.411(3)
C(22)-C(26)	1.545(3)
O(23)-C(23)	1.394(2)
O(23)-C(34)	1.433(3)
C(23)-C(24)	1.402(3)
C(24)-C(25)	1.395(3)
C(24)-C(30)	1.556(3)
C(25)-H(25)	0.92(2)
C(26)-C(27)	1.527(3)
C(26)-C(28)	1.537(3)
C(26)-C(29)	1.539(3)
C(27)-H(27A)	1.00(3)
C(27)-H(27B)	1.04(2)
C(27)-H(27C)	0.94(3)
C(28)-H(28A)	1.03(3)
C(28)-H(28B)	0.90(3)
C(28)-H(28C)	1.05(3)
C(29)-H(29A)	1.06(3)
C(29)-H(29B)	1.05(3)
C(29)-H(29C)	0.92(3)
C(30)-C(31)	1.524(4)
C(30)-C(33)	1.527(4)
C(30)-C(32)	1.533(4)
C(31)-H(31A)	1.02(4)
C(31)-H(31B)	0.99(5)
C(31)-H(31C)	1.05(4)
C(32)-H(32A)	0.82(5)
C(32)-H(32B)	0.94(4)
C(32)-H(32C)	0.91(4)
C(33)-H(33A)	1.13(4)
C(33)-H(33B)	0.95(5)

Table E110. continued...

C(33)-H(33C)	0.94(4)
C(34)-H(34A)	0.93(3)
C(34)-H(34B)	0.96(4)
C(34)-H(34C)	0.97(3)
N(50)-C(52)	1.442(3)
N(50)-C(51)	1.459(3)
N(50)-C(50)	1.470(3)
N(50)-H(50)	1.37(3)
C(50)-C(50)#2	1.534(4)
C(50)-H(50A)	0.9700
C(50)-H(50B)	0.9700
C(51)-H(51A)	0.9600
C(51)-H(51B)	0.9600
C(51)-H(51C)	0.9600
C(52)-H(52A)	0.9600
C(52)-H(52B)	0.9600
C(52)-H(52C)	0.9600

O(1)-C(1)-C(3)	124.22(14)
O(1)-C(1)-C(2)#1	116.78(14)
C(3)-C(1)-C(2)#1	119.00(14)
C(2)-O(2)-H(50)	124.3(14)
O(2)-C(2)-C(3)	121.55(14)
O(2)-C(2)-C(1)#1	116.41(14)
C(3)-C(2)-C(1)#1	122.03(14)
C(2)-C(3)-C(1)	118.97(14)
C(2)-C(3)-C(4)	121.67(14)
C(1)-C(3)-C(4)	119.29(13)
C(3)-C(4)-C(20)	114.30(13)
C(3)-C(4)-C(5)	110.82(13)
C(20)-C(4)-C(5)	113.84(13)
C(3)-C(4)-H(4)	107.3(12)
C(20)-C(4)-H(4)	101.9(12)
C(5)-C(4)-H(4)	107.9(12)
C(6)-C(5)-C(10)	117.53(16)
C(6)-C(5)-C(4)	118.94(15)
C(10)-C(5)-C(4)	123.53(15)
C(5)-C(6)-C(7)	123.59(17)
C(5)-C(6)-H(6)	117.3(12)
C(7)-C(6)-H(6)	119.1(12)
C(6)-C(7)-C(8)	116.19(16)
C(6)-C(7)-C(11)	116.86(16)
C(8)-C(7)-C(11)	126.88(16)
C(8)-O(8)-C(19)	112.93(14)

Table E110. continued...

O(8)-C(8)-C(7)	119.94(17)
O(8)-C(8)-C(9)	118.04(16)
C(7)-C(8)-C(9)	122.00(16)
C(10)-C(9)-C(8)	117.34(16)
C(10)-C(9)-C(15)	120.10(17)
C(8)-C(9)-C(15)	122.52(17)
C(5)-C(10)-C(9)	122.47(17)
C(5)-C(10)-H(10)	117.7(12)
C(9)-C(10)-H(10)	119.8(12)
C(13)-C(11)-C(12)	109.6(2)
C(13)-C(11)-C(7)	116.52(19)
C(12)-C(11)-C(7)	110.29(17)
C(13)-C(11)-C(14)	104.1(2)
C(12)-C(11)-C(14)	108.8(2)
C(7)-C(11)-C(14)	107.05(16)
C(11)-C(12)-H(12A)	109.5
C(11)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	109.5
C(11)-C(12)-H(12C)	109.5
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5
C(11)-C(13)-H(13A)	109.5
C(11)-C(13)-H(13B)	109.5
H(13A)-C(13)-H(13B)	109.5
C(11)-C(13)-H(13C)	109.5
H(13A)-C(13)-H(13C)	109.5
H(13B)-C(13)-H(13C)	109.5
C(11)-C(14)-H(14A)	109.5
C(11)-C(14)-H(14B)	109.5
H(14A)-C(14)-H(14B)	109.5
C(11)-C(14)-H(14C)	109.5
H(14A)-C(14)-H(14C)	109.5
H(14B)-C(14)-H(14C)	109.5
C(17)-C(15)-C(16)	106.0(3)
C(17)-C(15)-C(18)	109.0(3)
C(16)-C(15)-C(18)	109.2(2)
C(17)-C(15)-C(9)	111.27(18)
C(16)-C(15)-C(9)	112.46(18)
C(18)-C(15)-C(9)	108.8(2)
C(15)-C(16)-H(16A)	108.6(18)
C(15)-C(16)-H(16B)	109.4(18)
H(16A)-C(16)-H(16B)	117(3)
C(15)-C(16)-H(16C)	106.5(14)
H(16A)-C(16)-H(16C)	110(2)

Table E110. continued...

H(16B)-C(16)-H(16C)	105(2)
C(15)-C(17)-H(17A)	103(2)
C(15)-C(17)-H(17B)	121(3)
H(17A)-C(17)-H(17B)	109(3)
C(15)-C(17)-H(17C)	113(2)
H(17A)-C(17)-H(17C)	103(3)
H(17B)-C(17)-H(17C)	107(3)
C(15)-C(18)-H(18A)	106.8(17)
C(15)-C(18)-H(18B)	109.4(17)
H(18A)-C(18)-H(18B)	116(2)
C(15)-C(18)-H(18C)	106.1(16)
H(18A)-C(18)-H(18C)	105(2)
H(18B)-C(18)-H(18C)	112(2)
O(8)-C(19)-H(19A)	104.9(14)
O(8)-C(19)-H(19B)	112.9(19)
H(19A)-C(19)-H(19B)	115(2)
O(8)-C(19)-H(19C)	113.3(16)
H(19A)-C(19)-H(19C)	104(2)
H(19B)-C(19)-H(19C)	106(2)
C(25)-C(20)-C(21)	117.85(16)
C(25)-C(20)-C(4)	123.36(15)
C(21)-C(20)-C(4)	118.73(15)
C(22)-C(21)-C(20)	122.46(17)
C(22)-C(21)-H(21)	122.6(13)
C(20)-C(21)-H(21)	115.0(13)
C(21)-C(22)-C(23)	117.41(16)
C(21)-C(22)-C(26)	119.84(17)
C(23)-C(22)-C(26)	122.67(17)
C(23)-O(23)-C(34)	113.32(17)
O(23)-C(23)-C(24)	120.95(17)
O(23)-C(23)-C(22)	117.21(16)
C(24)-C(23)-C(22)	121.81(17)
C(25)-C(24)-C(23)	116.86(17)
C(25)-C(24)-C(30)	116.86(17)
C(23)-C(24)-C(30)	126.27(18)
C(20)-C(25)-C(24)	123.20(17)
C(20)-C(25)-H(25)	118.0(14)
C(24)-C(25)-H(25)	118.5(14)
C(27)-C(26)-C(28)	107.1(2)
C(27)-C(26)-C(29)	107.1(2)
C(28)-C(26)-C(29)	110.38(19)
C(27)-C(26)-C(22)	111.20(17)
C(28)-C(26)-C(22)	108.79(18)
C(29)-C(26)-C(22)	112.17(17)

Table E110. continued...

C(26)-C(27)-H(27A)	108.1(17)
C(26)-C(27)-H(27B)	113.9(13)
H(27A)-C(27)-H(27B)	109(2)
C(26)-C(27)-H(27C)	106.6(19)
H(27A)-C(27)-H(27C)	107(2)
H(27B)-C(27)-H(27C)	111(2)
C(26)-C(28)-H(28A)	108.5(14)
C(26)-C(28)-H(28B)	114(2)
H(28A)-C(28)-H(28B)	101(2)
C(26)-C(28)-H(28C)	110.4(14)
H(28A)-C(28)-H(28C)	111(2)
H(28B)-C(28)-H(28C)	112(2)
C(26)-C(29)-H(29A)	111.9(15)
C(26)-C(29)-H(29B)	111.3(16)
H(29A)-C(29)-H(29B)	107(2)
C(26)-C(29)-H(29C)	110.9(16)
H(29A)-C(29)-H(29C)	107(2)
H(29B)-C(29)-H(29C)	108(2)
C(31)-C(30)-C(33)	110.8(3)
C(31)-C(30)-C(32)	108.4(3)
C(33)-C(30)-C(32)	105.3(3)
C(31)-C(30)-C(24)	109.5(2)
C(33)-C(30)-C(24)	113.8(2)
C(32)-C(30)-C(24)	108.9(2)
C(30)-C(31)-H(31A)	115.3(19)
C(30)-C(31)-H(31B)	113(2)
H(31A)-C(31)-H(31B)	106(3)
C(30)-C(31)-H(31C)	106(2)
H(31A)-C(31)-H(31C)	110(3)
H(31B)-C(31)-H(31C)	106(3)
C(30)-C(32)-H(32A)	114(4)
C(30)-C(32)-H(32B)	116(2)
H(32A)-C(32)-H(32B)	96(4)
C(30)-C(32)-H(32C)	108(2)
H(32A)-C(32)-H(32C)	110(4)
H(32B)-C(32)-H(32C)	112(3)
C(30)-C(33)-H(33A)	104(2)
C(30)-C(33)-H(33B)	115(3)
H(33A)-C(33)-H(33B)	122(4)
C(30)-C(33)-H(33C)	109(2)
H(33A)-C(33)-H(33C)	107(3)
H(33B)-C(33)-H(33C)	100(3)
O(23)-C(34)-H(34A)	109.2(19)
O(23)-C(34)-H(34B)	110(2)

Table E110. continued...

H(34A)-C(34)-H(34B)	108(3)
O(23)-C(34)-H(34C)	112.3(18)
H(34A)-C(34)-H(34C)	108(3)
H(34B)-C(34)-H(34C)	109(3)
C(52)-N(50)-C(51)	112.0(3)
C(52)-N(50)-C(50)	113.4(2)
C(51)-N(50)-C(50)	113.8(2)
C(52)-N(50)-H(50)	104.3(13)
C(51)-N(50)-H(50)	104.1(13)
C(50)-N(50)-H(50)	108.2(13)
N(50)-C(50)-C(50)#2	113.4(2)
N(50)-C(50)-H(50A)	108.9
C(50)#2-C(50)-H(50A)	108.9
N(50)-C(50)-H(50B)	108.9
C(50)#2-C(50)-H(50B)	108.9
H(50A)-C(50)-H(50B)	107.7
N(50)-C(51)-H(51A)	109.5
N(50)-C(51)-H(51B)	109.5
H(51A)-C(51)-H(51B)	109.5
N(50)-C(51)-H(51C)	109.5
H(51A)-C(51)-H(51C)	109.5
H(51B)-C(51)-H(51C)	109.5
N(50)-C(52)-H(52A)	109.5
N(50)-C(52)-H(52B)	109.5
H(52A)-C(52)-H(52B)	109.5
N(50)-C(52)-H(52C)	109.5
H(52A)-C(52)-H(52C)	109.5
H(52B)-C(52)-H(52C)	109.5

Symmetry transformations used to generate equivalent atoms:

#1 -x+1, -y+2, -z

#2 -x+1, -y+1, -z

Table E111. Anisotropic displacement parameters ($Å^2 \times 10^3$) for **2E•$C_6H_{16}N_2$** (from acetone). The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+...+2hka^*b^*U_{12}]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O(1)	36(1)	25(1)	41(1)	-8(1)	-21(1)	-4(1)
C(1)	21(1)	24(1)	22(1)	-5(1)	-5(1)	-6(1)
O(2)	38(1)	21(1)	38(1)	-6(1)	-22(1)	1(1)
C(2)	23(1)	25(1)	23(1)	-4(1)	-6(1)	-4(1)
C(3)	20(1)	24(1)	21(1)	-4(1)	-6(1)	-4(1)
C(4)	22(1)	22(1)	23(1)	-4(1)	-9(1)	-5(1)
C(5)	20(1)	25(1)	28(1)	-6(1)	-8(1)	-6(1)
C(6)	22(1)	28(1)	28(1)	-6(1)	-9(1)	-5(1)
C(7)	25(1)	31(1)	34(1)	-14(1)	-3(1)	-10(1)
O(8)	23(1)	36(1)	61(1)	-22(1)	-3(1)	-3(1)
C(8)	21(1)	28(1)	44(1)	-15(1)	-6(1)	-4(1)
C(9)	25(1)	26(1)	45(1)	-6(1)	-11(1)	-4(1)
C(10)	25(1)	27(1)	29(1)	-2(1)	-7(1)	-5(1)
C(11)	33(1)	44(1)	31(1)	-14(1)	-1(1)	-13(1)
C(12)	95(2)	67(2)	42(1)	-10(1)	2(1)	-46(2)
C(13)	75(2)	88(2)	53(2)	-32(2)	5(1)	6(2)
C(14)	60(2)	97(2)	42(1)	-22(1)	-11(1)	-24(2)
C(15)	33(1)	29(1)	64(1)	2(1)	-14(1)	1(1)
C(16)	49(1)	43(1)	70(2)	-4(1)	-32(1)	3(1)
C(17)	55(2)	65(2)	87(2)	38(2)	-6(2)	11(2)
C(18)	57(2)	28(1)	127(3)	-16(1)	-39(2)	2(1)
C(19)	25(1)	49(1)	69(2)	-19(1)	-6(1)	-10(1)
C(20)	25(1)	25(1)	23(1)	-5(1)	-8(1)	-1(1)
C(21)	27(1)	31(1)	26(1)	-5(1)	-10(1)	-4(1)
C(22)	33(1)	33(1)	26(1)	-6(1)	-11(1)	1(1)
O(23)	48(1)	47(1)	25(1)	1(1)	0(1)	-6(1)
C(23)	36(1)	30(1)	24(1)	1(1)	-4(1)	-2(1)
C(24)	31(1)	32(1)	34(1)	-1(1)	-6(1)	-8(1)
C(25)	27(1)	32(1)	27(1)	-5(1)	-9(1)	-7(1)
C(26)	44(1)	49(1)	26(1)	-7(1)	-17(1)	0(1)
C(27)	48(1)	79(2)	40(1)	-14(1)	-25(1)	-9(1)
C(28)	65(2)	57(2)	38(1)	-7(1)	-26(1)	15(1)
C(29)	57(1)	66(2)	38(1)	-22(1)	-16(1)	-2(1)
C(30)	42(1)	56(1)	46(1)	1(1)	-5(1)	-27(1)
C(31)	35(1)	98(3)	122(3)	-26(3)	-9(2)	-21(2)
C(32)	82(2)	85(2)	75(2)	-18(2)	-7(2)	-59(2)
C(33)	109(3)	92(3)	76(2)	19(2)	-19(2)	-74(3)
C(34)	53(2)	79(2)	36(1)	-13(1)	6(1)	-2(1)
N(50)	43(1)	34(1)	45(1)	-12(1)	-14(1)	-8(1)

Table E111. continued...

C(50)	49(1)	35(1)	90(2)	-25(1)	-26(1)	-3(1)
C(51)	154(3)	38(1)	106(3)	-18(2)	-88(3)	6(2)
C(52)	79(2)	88(2)	120(3)	-72(2)	40(2)	-41(2)

Table E112. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for **2E•$C_6H_{16}N_2$** (from acetone).

Atom	x	y	z	U_{eq}	Occ.
H(4)	6738(17)	10079(18)	1656(16)	31(5)	1
H(6)	7836(17)	9718(17)	-361(16)	27(5)	1
H(10)	7890(17)	7360(17)	2559(17)	27(5)	1
H(12A)	10691	10251	-1890	133(16)	1
H(12B)	9384	10662	-1499	143(17)	1
H(12C)	9843	10624	-2722	180(20)	1
H(13A)	11398	8381	-2018	112	1
H(13B)	10726	8489	-2943	81(10)	1
H(13C)	10605	7452	-1851	96(12)	1
H(14A)	8860	9287	-2940	74(9)	1
H(14B)	7993	9419	-1863	79(9)	1
H(14C)	8539	8175	-1974	119	1
H(16A)	11640(30)	6230(30)	1720(30)	77(9)	1
H(16B)	11270(30)	5450(30)	3080(30)	77(9)	1
H(16C)	10910(20)	6830(20)	2700(20)	48(6)	1
H(17A)	8420(30)	5510(30)	3170(30)	84(10)	1
H(17B)	8870(50)	6210(50)	3900(40)	156(19)	1
H(17C)	9410(40)	4800(40)	3840(30)	119(13)	1
H(18A)	10730(30)	5250(30)	900(30)	85(10)	1
H(18B)	9260(30)	4850(30)	1570(30)	95(11)	1
H(18C)	10500(30)	4300(30)	2150(20)	68(8)	1
H(19A)	12530(20)	7580(20)	-480(20)	48(6)	1
H(19B)	11900(30)	7910(30)	680(30)	84(10)	1
H(19C)	11650(30)	8750(30)	-540(20)	71(8)	1
H(21)	7317(19)	9097(18)	3387(17)	37(5)	1
H(25)	4752(18)	8205(18)	2584(18)	35(5)	1
H(27A)	7910(20)	9440(30)	4730(20)	66(8)	1
H(27B)	8570(20)	8190(20)	4480(20)	48(6)	1
H(27C)	8480(30)	8460(30)	5650(30)	76(9)	1
H(28A)	7770(20)	6740(20)	6750(20)	56(7)	1
H(28B)	7650(30)	6390(30)	5800(30)	78(9)	1
H(28C)	6470(20)	6470(20)	6690(20)	53(7)	1
H(29A)	5970(20)	9660(20)	6050(20)	62(7)	1
H(29B)	6870(30)	8860(20)	6930(20)	72(8)	1
H(29C)	5710(20)	8440(20)	6930(20)	53(7)	1
H(31A)	2290(30)	8460(30)	4930(30)	88(11)	1
H(31B)	1770(40)	7660(40)	4460(30)	124(14)	1
H(31C)	2590(30)	8560(40)	3570(40)	108(13)	1
H(32A)	4350(50)	6150(50)	3390(40)	150(20)	1
H(32B)	3700(30)	7010(30)	2810(30)	84(11)	1

Table E112. continued...

H(32C)	3170(30)	6020(30)	3730(30)	90(10)	1
H(33A)	2980(40)	6590(40)	6160(40)	120(14)	1
H(33B)	4060(40)	5540(40)	5620(40)	127(18)	1
H(33C)	2880(30)	5660(30)	5510(30)	89(10)	1
H(34A)	3730(30)	7940(30)	7330(30)	75(9)	1
H(34B)	3100(30)	8070(30)	6400(30)	97(12)	1
H(34C)	3960(30)	8930(30)	6260(20)	70(9)	1
H(50)	5640(30)	6930(30)	560(30)	84(9)	1
H(50A)	4634	6197	-493	98(11)	1
H(50B)	4291	5566	771	112(13)	1
H(51A)	7021	5311	1377	137	1
H(51B)	6526	4302	1222	137	1
H(51C)	5758	5083	1947	137	1
H(52A)	7484	6260	-449	94(11)	1
H(52B)	6565	6724	-1215	141	1
H(52C)	7058	5394	-879	96(11)	1

Table E113. Torsion angles (°) for **2E•$C_6H_{16}N_2$** (from acetone).

Atoms	Angle
O(2) C(2) C(3) C(1)	-178.44(14)
C(1)#1 C(2) C(3) C(1)	0.8(3)
O(2) C(2) C(3) C(4)	4.7(3)
C(1)#1 C(2) C(3) C(4)	-176.09(14)
O(1) C(1) C(3) C(2)	179.39(16)
C(2)#1 C(1) C(3) C(2)	-0.8(3)
O(1) C(1) C(3) C(4)	-3.6(2)
C(2)#1 C(1) C(3) C(4)	176.19(13)
C(2) C(3) C(4) C(20)	-77.1(2)
C(1) C(3) C(4) C(20)	106.06(17)
C(2) C(3) C(4) C(5)	53.2(2)
C(1) C(3) C(4) C(5)	-123.69(15)
C(3) C(4) C(5) C(6)	46.26(19)
C(20) C(4) C(5) C(6)	176.75(14)
C(3) C(4) C(5) C(10)	-133.73(16)
C(20) C(4) C(5) C(10)	-3.2(2)
C(10) C(5) C(6) C(7)	-2.5(2)
C(4) C(5) C(6) C(7)	177.55(15)
C(5) C(6) C(7) C(8)	-5.4(2)
C(5) C(6) C(7) C(11)	171.68(15)
C(19) O(8) C(8) C(7)	77.2(2)
C(19) O(8) C(8) C(9)	-104.2(2)
C(6) C(7) C(8) O(8)	-170.90(14)
C(11) C(7) C(8) O(8)	12.3(3)
C(6) C(7) C(8) C(9)	10.6(2)
C(11) C(7) C(8) C(9)	-166.16(16)
O(8) C(8) C(9) C(10)	173.78(15)
C(7) C(8) C(9) C(10)	-7.7(3)
O(8) C(8) C(9) C(15)	-8.7(3)
C(7) C(8) C(9) C(15)	169.77(17)
C(6) C(5) C(10) C(9)	5.7(2)
C(4) C(5) C(10) C(9)	-174.27(16)
C(8) C(9) C(10) C(5)	-0.8(3)
C(15) C(9) C(10) C(5)	-178.36(17)
C(6) C(7) C(11) C(13)	-167.4(2)
C(8) C(7) C(11) C(13)	9.3(3)
C(6) C(7) C(11) C(12)	66.8(2)
C(8) C(7) C(11) C(12)	-116.5(2)
C(6) C(7) C(11) C(14)	-51.4(2)
C(8) C(7) C(11) C(14)	125.3(2)
C(10) C(9) C(15) C(17)	-1.2(3)

Table E113. continued...

C(8) C(9) C(15) C(17)	-178.6(3)
C(10) C(9) C(15) C(16)	-119.9(2)
C(8) C(9) C(15) C(16)	62.7(3)
C(10) C(9) C(15) C(18)	119.0(2)
C(8) C(9) C(15) C(18)	-58.4(3)
C(3) C(4) C(20) C(25)	19.2(2)
C(5) C(4) C(20) C(25)	-109.57(18)
C(3) C(4) C(20) C(21)	-163.72(14)
C(5) C(4) C(20) C(21)	67.54(19)
C(25) C(20) C(21) C(22)	-2.0(3)
C(4) C(20) C(21) C(22)	-179.22(15)
C(20) C(21) C(22) C(23)	-1.9(3)
C(20) C(21) C(22) C(26)	174.71(16)
C(34) O(23) C(23) C(24)	-81.4(3)
C(34) O(23) C(23) C(22)	100.5(2)
C(21) C(22) C(23) O(23)	-175.16(15)
C(26) C(22) C(23) O(23)	8.3(3)
C(21) C(22) C(23) C(24)	6.8(3)
C(26) C(22) C(23) C(24)	-169.73(17)
O(23) C(23) C(24) C(25)	174.54(16)
C(22) C(23) C(24) C(25)	-7.4(3)
O(23) C(23) C(24) C(30)	-6.2(3)
C(22) C(23) C(24) C(30)	171.81(19)
C(21) C(20) C(25) C(24)	1.2(3)
C(4) C(20) C(25) C(24)	178.30(16)
C(23) C(24) C(25) C(20)	3.4(3)
C(30) C(24) C(25) C(20)	-175.93(18)
C(21) C(22) C(26) C(27)	-2.2(3)
C(23) C(22) C(26) C(27)	174.20(19)
C(21) C(22) C(26) C(28)	-119.9(2)
C(23) C(22) C(26) C(28)	56.5(2)
C(21) C(22) C(26) C(29)	117.7(2)
C(23) C(22) C(26) C(29)	-65.9(3)
C(25) C(24) C(30) C(31)	-80.1(3)
C(23) C(24) C(30) C(31)	100.6(3)
C(25) C(24) C(30) C(33)	155.3(3)
C(23) C(24) C(30) C(33)	-23.9(4)
C(25) C(24) C(30) C(32)	38.2(3)
C(23) C(24) C(30) C(32)	-141.0(3)
C(52) N(50) C(50) C(50)#2	66.0(4)
C(51) N(50) C(50) C(50)#2	-63.6(4)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1, -y+2, -z #2 -x+1, -y+1, -z

Table E114. Hydrogen bonds, lengths (Å) and angles (°) for **2E•$C_6H_{16}N_2$** (from acetone).

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(2)-H(50)...N(50)	1.26(3)	1.37(3)	2.552(2)	152(3)

Table E115. Crystal and structure refinement data for **2E•$C_6H_{16}N_2$** (from ethyl acetate).

Identification code	**2E•$C_6H_{16}N_2$** (from ethyl acetate)
Empirical formula	$C_{37}H_{56}NO_4$
Formula weight	578.83 g/mole
Temperature	173(2) K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group	$P\bar{1}$
Unit cell dimensions	a = 12.115(2) Å b = 12.243(2) Å c = 13.062(3) Å α = 71.00(3)° β = 75.90(3)° γ = 77.72(3)°
Volume	1757.8(6) $Å^3$
Z	2
Density (calculated)	1.094 Mg/m^3
Absorption coefficient	0.069 mm^{-1}
F(000)	634
Crystal Size	1.00 mm x 0.76 mm x 0.52 mm
Color	yellow-orange
Theta range for data collection	1.68° to 28.26°
Index ranges	$-14 \le h \le 15$, $-15 \le k \le 16$, $-17 \le l \le 11$
Reflections collected / Unique	11262 / 7895 [R_{int} = 0.0111]
Refinement method	Full-matrix-least-squares on F^2
Data / Restraints / Parameters	7895 / 0 / 546
Goodness-of-fit on F^2	1.095
Final R indices [$I>2\sigma(I)$]	R_1 = 0.0539, wR_2 = 0.1563
R indices (all data)	R_1 = 0.0663, wR_2 = 0.1643
Extinction Coefficient	0.010(3)
Largest diff. peak and hole	0.664 and -0.342 e/A^3

In order to compare bond distances, angles and torsions within the dihydroxyquinone core from **2E•$C_6H_{16}N_2$** (from ethyl acetate) *to* **2E•$C_6H_{16}N_2$** (from ethyl acetate) *you must exchange C_1 with C_2 and O_1 with O_2. This has been done in Table 7 and Table 8 already.*

Table E116. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$ x 10^3) for **2E•$C_6H_{16}N_2$** (from ethyl acetate). U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	y	z	U_{eq}	Occ.
O(1)	9146(1)	2272(1)	4140(1)	30(1)	1
C(1)	9521(1)	1171(1)	4530(1)	23(1)	1
O(2)	9529(1)	-1675(1)	4348(1)	31(1)	1
C(2)	9712(1)	-860(1)	4615(1)	21(1)	1
C(3)	9247(1)	340(1)	4178(1)	21(1)	1
C(4)	8425(1)	647(1)	3381(1)	22(1)	1
C(5)	8944(1)	1216(1)	2185(1)	24(1)	1
C(6)	9928(1)	1737(1)	1873(1)	28(1)	1
C(7)	10383(1)	2287(1)	775(1)	33(1)	1
O(8)	10282(1)	2667(1)	-1153(1)	44(1)	1
C(8)	9832(1)	2234(1)	-32(1)	33(1)	1
C(9)	8786(1)	1770(1)	246(1)	30(1)	1
C(10)	8380(1)	1258(1)	1357(1)	27(1)	1
C(11)	11444(2)	2908(2)	570(2)	48(1)	1
C(12)	12531(2)	2008(3)	634(4)	83(1)	1
C(13)	11594(4)	3886(4)	-513(3)	88(1)	1
C(14)	11293(3)	3502(3)	1481(3)	73(1)	1
C(15)	8061(2)	1884(2)	-618(1)	38(1)	1
C(16)	7772(2)	3183(2)	-1227(2)	53(1)	1
C(17)	8689(2)	1197(2)	-1445(2)	51(1)	1
C(18)	6918(2)	1420(2)	-72(2)	53(1)	1
C(19)	11189(2)	1877(3)	-1570(2)	60(1)	1
C(20)	7278(1)	1328(1)	3814(1)	23(1)	1
C(21)	6731(1)	2335(1)	3162(1)	28(1)	1
C(22)	5645(1)	2879(1)	3548(1)	31(1)	1
O(23)	3981(1)	2843(1)	5004(1)	38(1)	1
C(23)	5113(1)	2378(1)	4643(1)	29(1)	1
C(24)	5694(1)	1453(1)	5372(1)	28(1)	1
C(25)	6757(1)	923(1)	4906(1)	25(1)	1
C(26)	5096(2)	4015(2)	2794(2)	44(1)	1
C(27)	5882(2)	4381(3)	1664(3)	86(1)	1
C(28)	4896(2)	4997(2)	3349(3)	67(1)	1
C(29)	3944(2)	3888(2)	2571(2)	53(1)	1
C(30)	5317(1)	1000(2)	6638(1)	34(1)	1
C(31)	5086(2)	-257(2)	6968(2)	62(1)	1
C(32)	6324(2)	1031(2)	7154(2)	61(1)	1
C(33)	4291(2)	1729(3)	7164(2)	71(1)	1
C(34)	3160(2)	2128(2)	5071(2)	45(1)	1
N(50)	9077(1)	4109(1)	4682(1)	36(1)	1

Table E116. continued...

C(50)	10102(2)	4413(2)	4871(2)	49(1)	1
C(51)	8639(3)	4919(2)	3715(3)	88(1)	1
C(52)	8170(3)	3906(3)	5650(3)	90(1)	1

Table E117. Bond lengths (Å) and angles (°) for **2E•$C_6H_{16}N_2$** (from ethyl acetate).

Atoms	Length	Angle
O(1)-C(1)	1.2990(17)	
O(1)-H(50)	1.26(3)	
C(1)-C(3)	1.3704(19)	
C(1)-C(2)#1	1.5283(18)	
O(2)-C(2)	1.2342(17)	
C(2)-C(3)	1.4355(19)	
C(2)-C(1)#1	1.5284(18)	
C(3)-C(4)	1.5188(18)	
C(4)-C(5)	1.5238(19)	
C(4)-C(20)	1.5426(19)	
C(4)-H(4)	0.975(18)	
C(5)-C(6)	1.382(2)	
C(5)-C(10)	1.397(2)	
C(6)-C(7)	1.400(2)	
C(6)-H(6)	0.950(18)	
C(7)-C(8)	1.402(2)	
C(7)-C(11)	1.555(2)	
O(8)-C(8)	1.3975(19)	
O(8)-C(19)	1.428(3)	
C(8)-C(9)	1.417(2)	
C(9)-C(10)	1.390(2)	
C(9)-C(15)	1.547(2)	
C(10)-H(10)	0.966(19)	
C(11)-C(13)	1.527(3)	
C(11)-C(12)	1.527(4)	
C(11)-C(14)	1.542(4)	
C(12)-H(12A)	1.02(4)	
C(12)-H(12B)	1.06(4)	
C(12)-H(12C)	0.99(4)	
C(13)-H(13A)	1.09(4)	
C(13)-H(13B)	0.95(4)	
C(13)-H(13C)	0.89(4)	
C(14)-H(14A)	0.95(3)	
C(14)-H(14B)	0.98(4)	
C(14)-H(14C)	0.89(4)	
C(15)-C(18)	1.530(3)	
C(15)-C(16)	1.536(3)	
C(15)-C(17)	1.541(3)	
C(16)-H(16A)	1.00(2)	
C(16)-H(16B)	0.98(2)	

Table E117. continued...

C(16)-H(16C)	0.94(3)
C(17)-H(17A)	1.01(3)
C(17)-H(17B)	1.05(3)
C(17)-H(17C)	0.96(3)
C(18)-H(18A)	0.97(2)
C(18)-H(18B)	1.04(2)
C(18)-H(18C)	1.02(3)
C(19)-H(19A)	0.95(3)
C(19)-H(19B)	0.97(3)
C(19)-H(19C)	0.98(3)
C(20)-C(25)	1.383(2)
C(20)-C(21)	1.388(2)
C(21)-C(22)	1.397(2)
C(21)-H(21)	0.952(19)
C(22)-C(23)	1.409(2)
C(22)-C(26)	1.543(2)
O(23)-C(23)	1.3984(18)
O(23)-C(34)	1.430(2)
C(23)-C(24)	1.403(2)
C(24)-C(25)	1.403(2)
C(24)-C(30)	1.547(2)
C(25)-H(25)	0.942(17)
C(26)-C(27)	1.533(3)
C(26)-C(29)	1.542(3)
C(26)-C(28)	1.545(3)
C(27)-H(27A)	0.97(4)
C(27)-H(27B)	1.12(4)
C(27)-H(27C)	1.00(3)
C(28)-H(28A)	1.07(3)
C(28)-H(28B)	1.00(3)
C(28)-H(28C)	1.07(3)
C(29)-H(29A)	1.02(3)
C(29)-H(29B)	1.01(3)
C(29)-H(29C)	1.02(3)
C(30)-C(31)	1.525(3)
C(30)-C(33)	1.525(3)
C(30)-C(32)	1.543(3)
C(31)-H(31A)	0.9600
C(31)-H(31B)	0.9600
C(31)-H(31C)	0.9600
C(32)-H(32A)	0.9600
C(32)-H(32B)	0.9600
C(32)-H(32C)	0.9600
C(33)-H(33A)	0.9600

Table E117. continued...

C(33)-H(33B)	0.9600
C(33)-H(33C)	0.9600
C(34)-H(34A)	0.97(3)
C(34)-H(34B)	1.04(3)
C(34)-H(34C)	1.00(3)
N(50)-C(52)	1.450(3)
N(50)-C(51)	1.465(3)
N(50)-C(50)	1.465(2)
N(50)-H(50)	1.36(3)
C(50)-C(50)#2	1.537(3)
C(50)-H(50A)	0.9700
C(50)-H(50B)	0.9700
C(51)-H(51A)	0.9600
C(51)-H(51B)	0.9600
C(51)-H(51C)	0.9600
C(52)-H(52A)	0.9600
C(52)-H(52B)	0.9600
C(52)-H(52C)	0.9600

C(1)-O(1)-H(50)	124.2(14)
O(1)-C(1)-C(3)	121.34(12)
O(1)-C(1)-C(2)#1	116.46(12)
C(3)-C(1)-C(2)#1	122.20(12)
O(2)-C(2)-C(3)	124.11(12)
O(2)-C(2)-C(1)#1	116.97(12)
C(3)-C(2)-C(1)#1	118.92(12)
C(1)-C(3)-C(2)	118.88(12)
C(1)-C(3)-C(4)	121.84(12)
C(2)-C(3)-C(4)	119.23(12)
C(3)-C(4)-C(5)	114.42(11)
C(3)-C(4)-C(20)	110.59(11)
C(5)-C(4)-C(20)	113.89(11)
C(3)-C(4)-H(4)	106.8(10)
C(5)-C(4)-H(4)	103.9(10)
C(20)-C(4)-H(4)	106.5(10)
C(6)-C(5)-C(10)	117.79(13)
C(6)-C(5)-C(4)	123.40(12)
C(10)-C(5)-C(4)	118.77(12)
C(5)-C(6)-C(7)	123.14(14)
C(5)-C(6)-H(6)	118.8(11)
C(7)-C(6)-H(6)	117.9(11)
C(6)-C(7)-C(8)	116.88(14)
C(6)-C(7)-C(11)	116.71(15)
C(8)-C(7)-C(11)	126.40(15)

Table E117. continued...

C(8)-O(8)-C(19)	113.56(15)
O(8)-C(8)-C(7)	121.02(15)
O(8)-C(8)-C(9)	116.97(14)
C(7)-C(8)-C(9)	121.99(14)
C(10)-C(9)-C(8)	117.23(13)
C(10)-C(9)-C(15)	119.83(14)
C(8)-C(9)-C(15)	122.83(14)
C(9)-C(10)-C(5)	122.59(14)
C(9)-C(10)-H(10)	121.4(11)
C(5)-C(10)-H(10)	116.0(11)
C(13)-C(11)-C(12)	110.4(3)
C(13)-C(11)-C(14)	105.3(2)
C(12)-C(11)-C(14)	108.0(3)
C(13)-C(11)-C(7)	114.1(2)
C(12)-C(11)-C(7)	109.68(18)
C(14)-C(11)-C(7)	109.09(17)
C(11)-C(12)-H(12A)	112(2)
C(11)-C(12)-H(12B)	107.4(19)
H(12A)-C(12)-H(12B)	115(3)
C(11)-C(12)-H(12C)	111(2)
H(12A)-C(12)-H(12C)	101(3)
H(12B)-C(12)-H(12C)	111(3)
C(11)-C(13)-H(13A)	104.7(19)
C(11)-C(13)-H(13B)	106(2)
H(13A)-C(13)-H(13B)	107(3)
C(11)-C(13)-H(13C)	111(3)
H(13A)-C(13)-H(13C)	121(4)
H(13B)-C(13)-H(13C)	107(4)
C(11)-C(14)-H(14A)	109.9(18)
C(11)-C(14)-H(14B)	116(2)
H(14A)-C(14)-H(14B)	110(3)
C(11)-C(14)-H(14C)	108(2)
H(14A)-C(14)-H(14C)	109(3)
H(14B)-C(14)-H(14C)	104(3)
C(18)-C(15)-C(16)	107.03(17)
C(18)-C(15)-C(17)	107.02(17)
C(16)-C(15)-C(17)	110.34(16)
C(18)-C(15)-C(9)	111.45(14)
C(16)-C(15)-C(9)	108.79(16)
C(17)-C(15)-C(9)	112.09(14)
C(15)-C(16)-H(16A)	110.0(14)
C(15)-C(16)-H(16B)	110.9(14)
H(16A)-C(16)-H(16B)	105.7(19)
C(15)-C(16)-H(16C)	111.3(17)

Table E117. continued...

H(16A)-C(16)-H(16C)	106(2)
H(16B)-C(16)-H(16C)	113(2)
C(15)-C(17)-H(17A)	110.6(14)
C(15)-C(17)-H(17B)	112.4(13)
H(17A)-C(17)-H(17B)	108.9(19)
C(15)-C(17)-H(17C)	111.9(14)
H(17A)-C(17)-H(17C)	105(2)
H(17B)-C(17)-H(17C)	107.9(19)
C(15)-C(18)-H(18A)	109.3(14)
C(15)-C(18)-H(18B)	113.3(12)
H(18A)-C(18)-H(18B)	108.0(18)
C(15)-C(18)-H(18C)	109.8(15)
H(18A)-C(18)-H(18C)	108(2)
H(18B)-C(18)-H(18C)	108.6(19)
O(8)-C(19)-H(19A)	108.1(17)
O(8)-C(19)-H(19B)	111.9(15)
H(19A)-C(19)-H(19B)	106(2)
O(8)-C(19)-H(19C)	111(2)
H(19A)-C(19)-H(19C)	109(3)
H(19B)-C(19)-H(19C)	110(2)
C(25)-C(20)-C(21)	117.73(13)
C(25)-C(20)-C(4)	118.90(12)
C(21)-C(20)-C(4)	123.37(13)
C(20)-C(21)-C(22)	122.21(14)
C(20)-C(21)-H(21)	116.8(11)
C(22)-C(21)-H(21)	121.0(11)
C(21)-C(22)-C(23)	117.62(14)
C(21)-C(22)-C(26)	119.78(15)
C(23)-C(22)-C(26)	122.55(14)
C(23)-O(23)-C(34)	112.96(12)
O(23)-C(23)-C(24)	119.99(14)
O(23)-C(23)-C(22)	118.14(14)
C(24)-C(23)-C(22)	121.86(13)
C(25)-C(24)-C(23)	116.30(14)
C(25)-C(24)-C(30)	116.74(14)
C(23)-C(24)-C(30)	126.88(13)
C(20)-C(25)-C(24)	123.44(14)
C(20)-C(25)-H(25)	118.5(10)
C(24)-C(25)-H(25)	118.0(10)
C(27)-C(26)-C(29)	106.4(2)
C(27)-C(26)-C(22)	111.31(15)
C(29)-C(26)-C(22)	112.44(15)
C(27)-C(26)-C(28)	108.7(2)
C(29)-C(26)-C(28)	109.35(17)

Table E117. continued...

C(22)-C(26)-C(28)	108.58(17)
C(26)-C(27)-H(27A)	108(2)
C(26)-C(27)-H(27B)	116(2)
H(27A)-C(27)-H(27B)	108(3)
C(26)-C(27)-H(27C)	104.5(18)
H(27A)-C(27)-H(27C)	107(3)
H(27B)-C(27)-H(27C)	112(3)
C(26)-C(28)-H(28A)	107.9(17)
C(26)-C(28)-H(28B)	107.0(16)
H(28A)-C(28)-H(28B)	108(2)
C(26)-C(28)-H(28C)	108.0(16)
H(28A)-C(28)-H(28C)	115(2)
H(28B)-C(28)-H(28C)	110(2)
C(26)-C(29)-H(29A)	111.2(15)
C(26)-C(29)-H(29B)	105.6(14)
H(29A)-C(29)-H(29B)	112(2)
C(26)-C(29)-H(29C)	111.1(15)
H(29A)-C(29)-H(29C)	112(2)
H(29B)-C(29)-H(29C)	105(2)
C(31)-C(30)-C(33)	109.36(19)
C(31)-C(30)-C(32)	108.87(18)
C(33)-C(30)-C(32)	104.57(18)
C(31)-C(30)-C(24)	110.11(14)
C(33)-C(30)-C(24)	116.42(16)
C(32)-C(30)-C(24)	107.14(14)
C(30)-C(31)-H(31A)	109.5
C(30)-C(31)-H(31B)	109.5
H(31A)-C(31)-H(31B)	109.5
C(30)-C(31)-H(31C)	109.5
H(31A)-C(31)-H(31C)	109.5
H(31B)-C(31)-H(31C)	109.5
C(30)-C(32)-H(32A)	109.5
C(30)-C(32)-H(32B)	109.5
H(32A)-C(32)-H(32B)	109.5
C(30)-C(32)-H(32C)	109.5
H(32A)-C(32)-H(32C)	109.5
H(32B)-C(32)-H(32C)	109.5
C(30)-C(33)-H(33A)	109.5
C(30)-C(33)-H(33B)	109.5
H(33A)-C(33)-H(33B)	109.5
C(30)-C(33)-H(33C)	109.5
H(33A)-C(33)-H(33C)	109.5
H(33B)-C(33)-H(33C)	109.5
O(23)-C(34)-H(34A)	106.7(14)

Table E117. continued...

O(23)-C(34)-H(34B)	112.8(15)
H(34A)-C(34)-H(34B)	107(2)
O(23)-C(34)-H(34C)	110.0(16)
H(34A)-C(34)-H(34C)	111(2)
H(34B)-C(34)-H(34C)	110(2)
C(52)-N(50)-C(51)	111.5(2)
C(52)-N(50)-C(50)	113.46(19)
C(51)-N(50)-C(50)	113.86(18)
C(52)-N(50)-H(50)	104.7(13)
C(51)-N(50)-H(50)	103.5(13)
C(50)-N(50)-H(50)	108.8(13)
N(50)-C(50)-C(50)#2	113.99(19)
N(50)-C(50)-H(50A)	108.8
C(50)#2-C(50)-H(50A)	108.8
N(50)-C(50)-H(50B)	108.8
C(50)#2-C(50)-H(50B)	108.8
H(50A)-C(50)-H(50B)	107.6
N(50)-C(51)-H(51A)	109.5
N(50)-C(51)-H(51B)	109.5
H(51A)-C(51)-H(51B)	109.5
N(50)-C(51)-H(51C)	109.5
H(51A)-C(51)-H(51C)	109.5
H(51B)-C(51)-H(51C)	109.5
N(50)-C(52)-H(52A)	109.5
N(50)-C(52)-H(52B)	109.5
H(52A)-C(52)-H(52B)	109.5
N(50)-C(52)-H(52C)	109.5
H(52A)-C(52)-H(52C)	109.5
H(52B)-C(52)-H(52C)	109.5

Symmetry transformations used to generate equivalent atoms:

#1 -x+2, -y, -z+1

#2 -x+2, -y+1, -z+1

Table E118. Anisotropic displacement parameters ($Å^2 \times 10^3$) for **2E•$C_6H_{16}N_2$** (from ethyl acetate). The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+...+2hka^*b^*U_{12}]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O(1)	37(1)	20(1)	36(1)	-7(1)	-18(1)	2(1)
C(1)	21(1)	23(1)	24(1)	-7(1)	-4(1)	-1(1)
O(2)	35(1)	23(1)	41(1)	-10(1)	-18(1)	-2(1)
C(2)	19(1)	23(1)	22(1)	-7(1)	-3(1)	-3(1)
C(3)	18(1)	22(1)	21(1)	-5(1)	-4(1)	-1(1)
C(4)	20(1)	22(1)	23(1)	-6(1)	-6(1)	-2(1)
C(5)	23(1)	24(1)	23(1)	-6(1)	-5(1)	0(1)
C(6)	26(1)	30(1)	27(1)	-6(1)	-5(1)	-4(1)
C(7)	29(1)	31(1)	33(1)	-4(1)	-2(1)	-6(1)
O(8)	47(1)	44(1)	25(1)	1(1)	3(1)	-3(1)
C(8)	34(1)	30(1)	24(1)	-1(1)	-1(1)	0(1)
C(9)	32(1)	31(1)	24(1)	-7(1)	-8(1)	3(1)
C(10)	26(1)	29(1)	25(1)	-6(1)	-6(1)	-2(1)
C(11)	40(1)	53(1)	46(1)	-1(1)	-2(1)	-23(1)
C(12)	34(1)	93(2)	116(3)	-23(2)	-4(1)	-17(1)
C(13)	102(3)	85(2)	70(2)	17(2)	-13(2)	-64(2)
C(14)	77(2)	80(2)	76(2)	-19(2)	-6(1)	-52(2)
C(15)	42(1)	46(1)	26(1)	-10(1)	-14(1)	3(1)
C(16)	60(1)	53(1)	38(1)	-7(1)	-21(1)	14(1)
C(17)	58(1)	62(1)	37(1)	-23(1)	-15(1)	0(1)
C(18)	45(1)	80(2)	39(1)	-17(1)	-21(1)	-6(1)
C(19)	52(1)	75(2)	37(1)	-14(1)	9(1)	3(1)
C(20)	20(1)	24(1)	27(1)	-8(1)	-5(1)	-4(1)
C(21)	24(1)	25(1)	31(1)	-4(1)	-5(1)	-3(1)
C(22)	24(1)	23(1)	44(1)	-7(1)	-8(1)	-1(1)
O(23)	22(1)	34(1)	59(1)	-23(1)	0(1)	-1(1)
C(23)	20(1)	26(1)	44(1)	-16(1)	-3(1)	-2(1)
C(24)	24(1)	30(1)	31(1)	-14(1)	-1(1)	-8(1)
C(25)	23(1)	26(1)	28(1)	-8(1)	-6(1)	-3(1)
C(26)	32(1)	27(1)	61(1)	0(1)	-10(1)	3(1)
C(27)	55(2)	61(2)	83(2)	36(2)	-1(1)	12(1)
C(28)	55(1)	27(1)	122(2)	-17(1)	-34(2)	3(1)
C(29)	48(1)	40(1)	68(1)	-5(1)	-28(1)	5(1)
C(30)	32(1)	41(1)	31(1)	-16(1)	2(1)	-10(1)
C(31)	89(2)	58(1)	40(1)	-10(1)	3(1)	-37(1)
C(32)	57(1)	93(2)	39(1)	-24(1)	-7(1)	-21(1)
C(33)	72(2)	82(2)	49(1)	-32(1)	7(1)	8(1)
C(34)	24(1)	46(1)	66(1)	-20(1)	-3(1)	-8(1)
N(50)	41(1)	28(1)	44(1)	-13(1)	-12(1)	-4(1)

Table E118. continued...

C(50)	45(1)	31(1)	79(1)	-24(1)	-20(1)	0(1)
C(51)	150(3)	33(1)	105(2)	-17(1)	-89(2)	7(1)
C(52)	77(2)	82(2)	119(2)	-71(2)	44(2)	-39(2)

Table E119. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for **2E•$C_6H_{16}N_2$** (from ethyl acetate).

Atom	x	y	z	U_{eq}	Occ.
H(4)	8247(15)	-89(15)	3370(14)	27(4)	1
H(6)	10283(15)	1778(15)	2430(15)	28(4)	1
H(10)	7676(16)	919(16)	1601(15)	33(5)	1
H(12A)	12720(30)	1640(30)	0(30)	102(11)	1
H(12B)	12420(30)	1410(30)	1440(30)	106(11)	1
H(12C)	13220(30)	2400(30)	480(30)	112(11)	1
H(13A)	11990(30)	3440(30)	-1140(30)	103(11)	1
H(13B)	12150(30)	4300(30)	-460(30)	108(11)	1
H(13C)	10950(40)	4390(40)	-580(40)	131(17)	1
H(14A)	11860(30)	3990(30)	1300(20)	82(8)	1
H(14B)	11280(30)	2970(30)	2230(30)	100(11)	1
H(14C)	10600(30)	3940(30)	1520(30)	103(12)	1
H(16A)	7300(20)	3280(20)	-1780(20)	58(6)	1
H(16B)	8470(20)	3520(20)	-1644(19)	54(6)	1
H(16C)	7320(20)	3600(20)	-740(20)	75(8)	1
H(17A)	8160(20)	1180(20)	-1930(20)	64(7)	1
H(17B)	9020(20)	340(20)	-1060(20)	60(6)	1
H(17C)	9310(20)	1560(20)	-1950(20)	57(6)	1
H(18A)	6470(20)	1550(20)	-630(20)	60(7)	1
H(18B)	6428(19)	1821(19)	514(19)	50(6)	1
H(18C)	7070(20)	550(20)	300(20)	71(8)	1
H(19A)	11230(20)	2060(30)	-2340(30)	80(8)	1
H(19B)	11040(20)	1070(20)	-1260(20)	65(7)	1
H(19C)	11930(30)	1950(30)	-1440(30)	99(10)	1
H(21)	7138(16)	2651(16)	2436(16)	32(4)	1
H(25)	7151(14)	280(15)	5372(14)	23(4)	1
H(27A)	5540(30)	5130(30)	1240(30)	104(10)	1
H(27B)	6060(40)	3770(40)	1150(30)	135(15)	1
H(27C)	6600(30)	4500(30)	1830(20)	84(9)	1
H(28A)	4300(30)	4760(30)	4110(30)	91(10)	1
H(28B)	4520(20)	5720(30)	2850(20)	73(8)	1
H(28C)	5720(30)	5130(30)	3420(20)	92(9)	1
H(29A)	3330(20)	3790(20)	3270(20)	69(7)	1
H(29B)	4110(20)	3190(20)	2280(20)	60(7)	1
H(29C)	3690(20)	4570(20)	1940(20)	73(7)	1
H(31A)	4356	-265	6807	91(10)	1
H(31B)	5074	-604	7743	131(14)	1
H(31C)	5682	-693	6561	142(15)	1
H(32A)	6149	687	7937	75(8)	1
H(32B)	6438	1825	6997	113	1

Table E119. continued...

H(32C)	7012	598	6848	72(8)	1
H(33A)	3596	1612	7011	123(14)	1
H(33B)	4380	2539	6867	85(9)	1
H(33C)	4252	1493	7947	74(8)	1
H(34A)	2430(20)	2440(20)	5460(20)	59(6)	1
H(34B)	3360(20)	1270(30)	5530(20)	75(8)	1
H(34C)	3100(20)	2160(20)	4310(20)	80(9)	1
H(50)	9360(30)	3070(30)	4430(20)	93(9)	1
H(50A)	10701	4441	4221	77(8)	1
H(50B)	10376	3801	5481	86(9)	1
H(51A)	9269	5089	3106	132	1
H(51B)	8253	5629	3880	132	1
H(51C)	8109	4570	3524	132	1
H(52A)	8483	3370	6267	134	1
H(52B)	7583	3584	5514	134	1
H(52C)	7844	4632	5812	134	1

Table E120. Torsion angles (°) for **2E•$C_6H_{16}N_2$** (from ethyl acetate).

Atoms	Angle
O(1) C(1) C(3) C(2)	178.30(12)
C(2)#1 C(1) C(3) C(2)	-1.0(2)
O(1) C(1) C(3) C(4)	-4.6(2)
C(2)#1 C(1) C(3) C(4)	176.12(11)
O(2) C(2) C(3) C(1)	-179.39(13)
C(1)#1 C(2) C(3) C(1)	1.0(2)
O(2) C(2) C(3) C(4)	3.4(2)
C(1)#1 C(2) C(3) C(4)	-176.23(11)
C(1) C(3) C(4) C(5)	76.73(16)
C(2) C(3) C(4) C(5)	-106.13(14)
C(1) C(3) C(4) C(20)	-53.48(17)
C(2) C(3) C(4) C(20)	123.65(13)
C(3) C(4) C(5) C(6)	-18.88(19)
C(20) C(4) C(5) C(6)	109.69(15)
C(3) C(4) C(5) C(10)	163.64(12)
C(20) C(4) C(5) C(10)	-67.79(16)
C(10) C(5) C(6) C(7)	-0.7(2)
C(4) C(5) C(6) C(7)	-178.18(13)
C(5) C(6) C(7) C(8)	-3.7(2)
C(5) C(6) C(7) C(11)	175.68(15)
C(19) O(8) C(8) C(7)	81.3(2)
C(19) O(8) C(8) C(9)	-100.3(2)
C(6) C(7) C(8) O(8)	-174.40(14)
C(11) C(7) C(8) O(8)	6.3(3)
C(6) C(7) C(8) C(9)	7.3(2)
C(11) C(7) C(8) C(9)	-171.99(16)
O(8) C(8) C(9) C(10)	175.25(13)
C(7) C(8) C(9) C(10)	-6.4(2)
O(8) C(8) C(9) C(15)	-8.6(2)
C(7) C(8) C(9) C(15)	169.70(15)
C(8) C(9) C(10) C(5)	1.7(2)
C(15) C(9) C(10) C(5)	-174.51(14)
C(6) C(5) C(10) C(9)	1.7(2)
C(4) C(5) C(10) C(9)	179.31(13)
C(6) C(7) C(11) C(13)	-155.6(3)
C(8) C(7) C(11) C(13)	23.7(3)
C(6) C(7) C(11) C(12)	79.9(3)
C(8) C(7) C(11) C(12)	-100.8(3)
C(6) C(7) C(11) C(14)	-38.2(2)
C(8) C(7) C(11) C(14)	141.2(2)
C(10) C(9) C(15) C(18)	2.1(2)

Table E120. continued...

C(8) C(9) C(15) C(18)	-173.97(16)
C(10) C(9) C(15) C(16)	119.83(18)
C(8) C(9) C(15) C(16)	-56.2(2)
C(10) C(9) C(15) C(17)	-117.87(18)
C(8) C(9) C(15) C(17)	66.1(2)
C(3) C(4) C(20) C(25)	-46.36(16)
C(5) C(4) C(20) C(25)	-176.85(12)
C(3) C(4) C(20) C(21)	133.75(13)
C(5) C(4) C(20) C(21)	3.26(18)
C(25) C(20) C(21) C(22)	-5.5(2)
C(4) C(20) C(21) C(22)	174.36(13)
C(20) C(21) C(22) C(23)	0.8(2)
C(20) C(21) C(22) C(26)	178.39(14)
C(34) O(23) C(23) C(24)	-77.22(19)
C(34) O(23) C(23) C(22)	104.02(17)
C(21) C(22) C(23) O(23)	-173.70(13)
C(26) C(22) C(23) O(23)	8.8(2)
C(21) C(22) C(23) C(24)	7.6(2)
C(26) C(22) C(23) C(24)	-169.97(15)
O(23) C(23) C(24) C(25)	170.80(12)
C(22) C(23) C(24) C(25)	-10.5(2)
O(23) C(23) C(24) C(30)	-12.6(2)
C(22) C(23) C(24) C(30)	166.13(14)
C(21) C(20) C(25) C(24)	2.3(2)
C(4) C(20) C(25) C(24)	-177.65(12)
C(23) C(24) C(25) C(20)	5.5(2)
C(30) C(24) C(25) C(20)	-171.46(13)
C(21) C(22) C(26) C(27)	0.5(3)
C(23) C(22) C(26) C(27)	178.0(2)
C(21) C(22) C(26) C(29)	119.74(19)
C(23) C(22) C(26) C(29)	-62.8(2)
C(21) C(22) C(26) C(28)	-119.13(19)
C(23) C(22) C(26) C(28)	58.3(2)
C(25) C(24) C(30) C(31)	-66.59(19)
C(23) C(24) C(30) C(31)	116.81(19)
C(25) C(24) C(30) C(33)	168.23(18)
C(23) C(24) C(30) C(33)	-8.4(2)
C(25) C(24) C(30) C(32)	51.66(19)
C(23) C(24) C(30) C(32)	-124.94(18)
C(52) N(50) C(50) C(50)#2	-65.4(3)
C(51) N(50) C(50) C(50)#2	63.6(3)

Symmetry transformations used to generate equivalent atoms:

#1 -x+2, -y, -z+1 #2 -x+2, -y+1, -z+1

Table E121. Hydrogen bonds, lengths (Å) and angles (°) for **2E•$C_6H_{16}N_2$** (from ethyl acetate).

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(1)-H(50)...N(50)	1.26(3)	1.36(3)	2.5478(17)	153(3)

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