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CONDENSATION OF TERT-BUTYL  
ALCOHOL WITH PHENOL IN THE  
PRESENCE OF ANHYDROUS ZINC  
CHLORIDE UNDER THIRTY DEGREES  
TEMPERATURE

Thesis for the Degree of M. S.  
MICHIGAN STATE COLLEGE

Albert H. Agett  
1938



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THESIS

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## INTRODUCTION

In 1916 Huston and Freedman (1) condensed benzyl alcohol and benzene by useing anhydrous aluminum chloride as the condensing agent.

In 1920 (2) benzyl alcohol and phenol were condensed by the same method.

In 1929 (3) benzyl alcohol and para-cresol were also condensed by the use of anhydrous aluminum chloride as the condensing agent.

In 1934 Huston and Fox (4) condensed tert-butyl and tert-amyl alcohols with benzene.(same method)

In 1936 tert-butyl and tert-amyl alcohols were condensed with phenol.(5).

To investigate the use of zinc chloride as a condensing agent below thirty degrees temperature the author has attempted to condense tertiary butyl alcohol with phenol.



Previous workers have used anhydrous zinc chloride as a condensing agent and as a catalyst in many instances. In all cases that were investigated an elevated temperature was required to bring about the desired reaction.

Liebmann (6) condensed benzyl alcohol with phenol in the presence of anhydrous zinc chloride. The reaction took place rapidly and with a fair yield.

Kippernberg (7) brought about the condensation of an alcohol and an amine. Here again an elevated temperature was required to bring about the reaction in the presence of anhydrous zinc chloride.

Fisher and Roser (8) (9) condensed alcohols with aromatic bases. This reaction took place only at elevated temperatures and required a long period of time.

Liebermann (10) condensed butyl, benzyl and amyl alcohols with phenol. Finding also that the higher homologues of phenol failed to give the ferric chloride tests. An elevated temperature was required to initiate the reaction.

Merz and Weith (11) also produced diphenyl ether by the action of anhydrous zinc chloride at 110° on phenol.

Auer (12) prepared ethyl phenol by heating absolute alcohol (ethyl) and phenol in the presence of anhydrous zinc chloride.

In the crotonization of aldehydes and ketones Perkins (13) discovered that in a liquid medium acetaldehyde was converted into crotonaldehyde with zinc chloride at a temperature of 97°.

Prieb (14) condensed benzaldehyde with nitromethane in the presence of anhydrous zinc chloride to give the nitro derivatives of phenyl ethylene.

Frank and Kohn (15) found that hot para-aldehyde was converted into mono-molecular aldehydes at 100°.

Knownagin (16) acetylated aldehydes in the presence of anhydrous zinc chloride at a very high temperature.

Sabateir and Mailhe (17) used zinc chloride as a dehydrating agent, especially in the dehydration of alcohols to give ethers and unsaturated (usually)



hydrocarbons. It was also found that it was impossible to use zinc chloride to dehydrate methyl alcohol to give di-methyl ether as gaseous products are produced through a very complicated reaction and a certain amount of hexamethylbenzene is also formed.

The author was unable to find any references in which anhydrous zinc chloride had been used successfully as a condensing agent at reduced temperatures.

## DISCUSSION

The condensation of tertiary butyl with phenol was attempted by the author (19). No alkylated phenol was isolated by this method. Many different methods were used to bring about this reaction at the desired temperature but they were all unsuccessful. It was found, however, that by the addition of a small quantity of anhydrous aluminum chloride to the reaction mixture that the zinc chloride was activated and the reaction took place. The percent yield of the para isomer compares favorably with that of the aluminum chloride condensations with less gummy material being formed. Upon purifying the para isomer it was noticed that there was a low and a high boiling fraction that could not be any of the original reactants. During the course of the investigation these fractions were proven to be the ortho and the dialkylated phenols which apparently exist in equilibrium with the para isomer, but in very small quantities. Attempts were made to produce the ortho and the dialkylated isomers by heating the para isomer in the presence of the aluminum chloride zinc chloride mixture (same amounts as used in condensations)

but only the ortho-tertiary butyl phenol was isolated in very small quantities. The dialkylated product was not obtained in this procedure although it was found in small amounts when tert-butyl alcohol and phenol were condensed in the presence of the zinc chloride aluminum chloride mixture. When the same method was used with aluminum chloride alone (20) both the ortho<sup>h</sup> and the dialkylated phenols were isolated in small quantities (see tables). When the ortho isomer was treated with aluminum chloride only the para tertiary butyl phenol could be isolated. The same procedure was also followed using anhydrous zinc chloride as the equilibrium producing agent, but no ortho or dialkylated phenols were isolated. With phosphoric acid as the equilibrium producing agent no ortho or dialkylated phenols were isolated. However when tertiary butyl alcohol and phenol were condensed in the presence of phosphoric acid (21) a small yield of ortho tertiary butyl phenol was found but no dialkylated isomer was isolated.

The structure of the alkylated phenols were proven by comparison with those produced by Ferkins, Dietzler, and Lundquist (20). In addition

Swayzee (22) condensed ortho bromo phenol with tert-butyl alcohol using the aluminum chloride method. The author upon the bromination of the 4-tertiary butyl phenol found that the product formed was identical in Melting Point and Boiling Point to that of Swayzee's above. A mixed melting point was run with no depression. A bromine determination was made (23) (see tables) on both compounds and close agreement with the theoretical were obtained. One atom of bromine being attached to the ring.

A carbon and hydrogen determination (24) was run on the ortho alkylated phenol and the calculated results agree favorably with the theoretical (see tables). The ortho tertiary butyl phenol was then bromine determination made (23). It was also found to have only one atom of Br attached to the ring. Since the melting point and the boiling point of this compound does not agree with that of 2-bromo 4-tertiary butyl phenol and since there are no other likely possibilities it can be safely stated that it is 2-tertiary butyl phenol. Also when the ortho tertiary butyl phenol was treated with aluminum chloride it rearranged almost quantitatively to the 4-tertiary butyl phenol.

Carbon and hydrogen determinations (24) were made on the 2,4 ditertiary butyl phenol and the calculated results were in agreement with the theoretical (see tables)

The author also attempted to alkylate para-bromo phenol with tertiary butyl alcohol in the presence of anhydrous aluminum chloride, anhydrous zinc chloride and aluminum chloride mixture, and in the presence of phosphoric acid. In all three cases no alkylated phenols were recovered. Apparently the ortho position under these conditions is not receptive to alkylation.

Tertiary butyl bromide and phenol were condensed in the presence of metallic Sodium (25). The only alkylated phenol that was isolated was the para isomer and no ortho or dialkylated were obtained.

In order to facilitate the separation of the ortho isomer from the para isomer it was found that it was very convenient to use a 5% solution of KOH. The para tertiary butyl phenol being soluble and the ortho tertiary butyl phenol insoluble in this solution (see tables).



## EXPERIMENTAL

### A.

Condensation of Tertiary Butyl Alcohol with Phenol  
in the Presence of Zinc Chloride and Aluminum  
Chloride Under Thirty Degrees Temperature.

Eighteen and five-tenths grams (.25 mole) of Tertiary butyl alcohol and 23.5 grams (0.25 mole) of phenol were treated with 80 ml. of petroleum ether in a five hundred ml. round bottomed three necked flask equipped with mercury sealed mechanical stirrer, and a condenser which carries both a thermometer which reaches into the reaction mixture and a bent tube for the outlet of HCl gas. This apparatus was placed in the hood. Then seventeen grams (.125 mole) of Zinc Chloride was added to the mixture and stirred for one half an hour. Then to this mixture was added three grams of anhydrous aluminum chloride. No rise in temperature was noted. After the addition of the aluminum chloride the stirring was continued for four or five hours. During this time only a very little amount of HCl gas was given off. The contents became a slight pink in color and upon standing overnight became quite dark. At which time it was decomposed with ice water and HCl. The hydrolyzed product was then extracted three times with

ether and dried over  $\text{CaCl}_2$ . The ether was removed by distillation and the residue fractionated. The first fraction was collected from  $90^\circ$ - $120^\circ$  @ 14 mm. pressure and was found to be mostly unchanged phenol and ortho tertiary butyl phenol. The second fraction was collected at  $120^\circ$ - $135^\circ$  @ 14 mm. pressure and was found to be almost pure para tert-butyl phenol. The third fraction was collected at  $135^\circ$ - $150^\circ$  @ 14 mm. pressure and was found to be mostly 2,4 ditertiary butyl phenol. These three fractions were purified by fractional distillation. The para tertiary butyl phenol was recrystallized from petroleum ether and was to be a 38% yield .M.P.  $97.5^\circ$ ; B.P.  $120^\circ$ - $122^\circ$  @ 14 mm. pressure. The ortho tertiary butyl phenol was found to be a slightly yellow colored liquid upon purification. Yield 1.5%; B.P.  $115^\circ$ - $117^\circ$  @ 23 mm pressure. The 2,4 ditertiary butyl phenol was purified by fractionation. It crystallized as a white solid with M.P.  $53^\circ$  and B.P.  $143^\circ$ - $145^\circ$  @ 23 mm pressure. Less than 1% yield was obtained.

- ↑

B.

Reaction of Para Tertiary Butyl Phenol with Zinc Chloride and Aluminum Chloride.

Seventy-five grams (.5 mole) of pure tert butyl phenol (para isomer) were placed in a 500ml. modified Claisen flask fitted with a capillary, thermometer, and a condensing flask. Thirty-four grams of anhydrous zinc chloride and six grams of anhydrous aluminum chloride was added cautiously to the warmed para tertiary butyl phenol. A small amount of HCl was given off at this time and some iso-butylene. The reaction was carried out under reduced pressure, water pump, and the mixture was heated for three hours at 35 mm pressure. At the end of the three hour period the heat was gradually increased and about twenty grams of phenol was distilled fractionally from the mixture. A solution made up of five ml. of distilled water and eleven grams of sodium carbonate was then added to the residue and filtered immediately, while hot. Upon purification by fractional distillation obtained 54.5 grams (73%) yield of para tertiary butyl phenol M.P.  $97^{\circ}$ . One and five tenths grams (2%) of 2,4 di-tertiary butyl phenol was obtained M.P.  $54^{\circ}$ .

C.

Condensation of Tertiary Butyl Alcohol with Para Bromo Phenol in the Presence of Anhydrous Zinc Chloride and Aluminum Chloride.

The procedure the same as in A above and upon purification obtained 35.5 grams (82%) of para bromo phenol and 5 grams (3%) of an unidentified compound. B.P.  $125^{\circ}$ - $127^{\circ}$  @14mm pressure.

D.

Condensation of Tertiary Butyl Alcohol and Para Tertiary Butyl Phenol in the Presence Of Zinc Chloride and Aluminum Chloride.

The procedure the same as in A above. Upon purification recovered thirty grams (80%) of para tertiary butyl phenol. No other alkylated phenol was isolated.

E.

Condensation of Tertiary Butyl Alcohol and Phenol in the Presence of Anhydrous Zinc Chloride Under Thirty Degrees Temperature.

The procedure the same as in A above with the exception that 17 grams of anhydrous (fused) Zinc Chloride was used as the condensing agent. No alkalyated phenols were isolated. Ninety % (21 grams) of para teritary butyl phenol was recovered.

F.

Condensation of Tertiary Butyl Alcohol with Phenol in the Presence of Anhydrous Aluminum Chloride.

The procedure the same as in A above. Upon purification a 41% yield of para tertiary butyl phenol was obtained. M.P.  $97.5^{\circ}$ ; B.P.  $120^{\circ}$ - $122^{\circ}$  @ 14 mm. pressure. The ortho tertiary butyl phenol upon purification was 1.5% yield of a slight yellow liquid with a B.P.  $115^{\circ}$ - $117^{\circ}$  @ 23 mm. pressure. The 2,4 ditertiary butyl phenol was purified by fractional distillation and crystallized as a white solid with a M.P.  $53.5^{\circ}$  and B.P.  $143^{\circ}$ - $145^{\circ}$  @ 23 mm pressure. A 5 % yield of the pure product was obtained.

G.

Reaction of Para Tertiary Butyl Phenol in the Presence of Anhydrous Aluminum Chloride.

The procedure the same as in B above. Thirty-four grams of anhydrous aluminum Chloride was added. A great deal of HCl was evolved at this time. The filtrate was fractionated and obtained 58% (43.5 grams) of para tertiary butyl phenol, five grams (6.6%) of 2,4 ditertiary butyl phenol, two grams (2.6%) of ortho tertiary butyl phenol.

H.

Reaction of Tertiary Butyl Alcohol with Para Bromo Phenol in the Presence of Anhydrous Aluminum Chloride.

Eighteen and five-tenths grams of (.25 mole) tertiary butyl alcohol and 43.5 grams (.25 mole) of para bromo phenol was treated with 80 ml. of petroleum ether in a 500 ml. round bottomed three necked flask. The procedure the same as in A above. Upon hydrolysis, extraction, and purification obtained 39 grams of para bromo phenol and no alkylated product was identified.



I.

Condensation of Tertiary Butyl Alcohol and Para Tertiary Butyl Phenol with Anhydrous Aluminum Chloride.

The procedure is identical to that in A above. Upon hydrolysis, extraction and purification twenty grams (80%) of para tertiary butyl phenol were recovered. Five grams (1.5%) of 2,4 ditertiary butyl phenol were obtained M.P. 53.5°.

J. Reaction of Para Tertiary Butyl Phenol with Tert. Butyl Alcohol in the presence of Anhydrous Zinc Chloride.

The procedure the same as in A above .25 mole of the reactants being used. No dialkylated phenols were isolated. Twenty-two grams (92%) of para tertiary butyl phenol recovered.

K.

Condensation of Tertiary Butyl Alcohol with Phenol in the Presence of Phosphoric Acid. (26)

Eighteen and five-tenths grams (.25 mole) of tertiary butyl alcohol and 23.5 grams (.25 mole) of phenol and 200 grams of phosphoric acid were placed in a 500 ml. round bottomed flask fitted with a

mercury sealed mechanical stirrer and reflux condenser. The flask was then placed on the steam bath and heated for eight hours at  $100^{\circ}$  with constant stirring. At the end of this period the phosphoric acid was siphoned off and the residue was fractionally distilled. Obtained 19 grams (80%) of para tertiary butyl phenol, M.P.  $95^{\circ}$ ; .5 grams (2%) of 2,4 ditertiary butyl phenol, M.P.  $53.5^{\circ}$ .

L.

Condensation of Tertiary Butyl Alcohol with Para Bromo Phenol in the Presence of Phosphoric Acid

The procedure the same as in K above. Forty-three grams of para bromo phenol (.25 mole) was used. Upon purification 95% (41.5 grams) of para bromo phenol was recovered. No alkylated phenol was obtained.

M.

Reaction of Tertiary Butyl Bromide and Phenol in the Presence of Metallic Sodium.

Eleven and five-tenths grams of (.5 mole) metallic sodium was suspended in one hundred mls. of toluene in 500ml. round bottomed three necked flask

fitted with a mercury sealed mechanical stirrer and reflux condenser. Forty-seven grams (.5mole) of phenol were added carefully and the mixture stirred until the reaction was completed. Then 68.5 grams (.5 mole) of tertiary butyl bromide added slowly by means of a separatory funnel. The reaction mixture was stirred for six hours and allowed to stand overnight. Upon purification 42.5% (20 grams) of para tertiary butyl phenol were isolated, M.P.  $97.5^{\circ}$ . No other alkylated phenol was obtained.

N.

#### Bromination of Para Tertiary Butyl Phenol

Twenty-three grams (.25 mole) of para-tertiary butyl phenol was dissolved in sixty mls of carbon tetra chloride in a two hundred ml. round bottomed three necked flask, to which was fitted a mercury sealed mechanical stirrer, reflux condenser fitted with a thermometer reaching below the surface of the liquid, and a separatory funnel. Forty grams (.25 mole) of Bromine was added by use of the separatory funnel. During the addition of the Bromine the mixture was cooled with an ice and salt mixture and kept around  $10^{\circ}$ . The mixture was then allowed to come to room temperature with stirring for four hours after the Bromine was

added. It was allowed to stand overnight and then it was purified by fractional distillation under reduced pressure. Obtained a white crystalline solid, B.P. 121-123° @ 16mm/pressure and M.P. 45°.

O.

#### Bromination of Ortho Tertiary Butyl Phenol

The procedure indetical to that of N above. Upon purification a white colorless solid M.P. 55-56° and a B.P. 145-148° @ 14mm. pressure was obtained.

TABLE OF RESULT

ALCOHOL	PHENOL	CONDENSING AGENT	PRODUCT	% YIELD	M.P.	B.P.
TERT/BUTYL	phenol	$ZnCl_2$ (17gr.). $AlCl_3$ (3gr.)	p-tert.butyl phenol 2,4 ditert.butyl phenol o-tert.butyl phenol	38.0% -1.5% -1.0%	97° 54° liquid	120-122°/14mm 143-144°/23mm 115-117°/23mm
-----	p-tert.butyl phenol	above	p-tert.butyl phenol 2,4 ditert.butyl phenol o-tert.butyl phenol	73.0% 2.0% None	97° 53.5° -----	120-122°/14mm 143-145°/23mm -----
tert.butyl	p-bromo phenol	above	para bromo phenol unidentified product	82.0% 3.0%	62.5 solid	230°/760 mm 125-127°/14mm
Tert.butyl	p-tert.butyl phenol	above	p-tert.butyl phenol 2,4 ditert.butyl phenol o-tert.butyl phenol	87.0% None None	96.5° ----- -----	122-123°/14mm ----- -----
TERT/Butyl	phenol	$AlCl_3$ (17gr. 125 m)	p-tert.butyl phenol 2,4 ditert.butyl phenol o-tert.butyl phenol	41.0% 5.0% 1.5%	97.5° 53.5° liquid	120-123°/14mm 143-144°/23mm 115-117°/23mm
-----	P-TERT. Butyl	$AlCl_3$ (17gr. 125m)	p-tert.butyl phenol 2,4 ditert.butyl phenol o-tert.butyl phenol	58.0% 6.6 % 2.6 %	97° 54.0° liquid	120-122°/14mm 144-145°/23mm 116-117°/23mm
tert.butyl	p-bromo phenol	above	p-bromo phenol No Condensation	75.0% -----	63.0° -----	230°/760 mm -----
tert.butyl	p-tert.butyl	above	p-tert.butyl phenol 2,4 ditert. butyl phenol	80.0% 1.5%	97.0° 53.0°	120-123°/14mm 140-145°/24mm
-----	p-tert.butyl	$ZnCl_2$ (17gr)	p-tert.butyl phenol No Condensation	89.0% -----	97.0° -----	120-125°/14mm -----
tert.butyl	phenol	$ZnCl_2$ (17gr)	No Condensation	-----	-----	-----
tert.butyl	phenol	$H_3PO_4$ (200)	p-tert.butyl phenol 2,4 ditert.butyl phenol o-tert.butyl phenol	80.0% 2.0% None	96.5% 52.5% -----	120-122°/14mm 142-145°/23mm -----
tert.butyl	p-bromo phenol	$H_3PO_4$	No Condensation	-----	-----	-----
TERT. Butyl Bromide	phenol	Metallic Na	o-tert.butyl phenol p-tert.butyl phenol 2,4 ditert.butyl phenol	42.5% ----- -----	98.0° ----- -----	120-125°/14mm ----- -----





TABLE OF RESULTS  
cont.

SOLUBILITY OF ALKYLATED PHENOLS  
IN 5% KOH

COMPOUND	SOLUBILITY (5%KOH)
p-bromo o-tert.butyl phenol	insoluble
Ortho tert.butyl phenol	insoluble
o-bromo p-tert.butyl phenol	soluble
Para tert.butyl phenol	soluble

PERCENT CARBON AND HYDROGEN

COMPOUND	% C THEO.	% C CALC.	% H THEO.	% H CALC.
P-tert.butyl phenol	80.00	79.85	9.33	10.250
O-tert.butyl phenol	80.00	78.35	9.33	10.305
2,4 ditert,tert.butyl phenol	81.55	82.01	10.67	10.790

PERCENT BROMINE

COMPOUND	Calc. % Br	THEO. % Br
2 bromo 4 tert.butyl phenol M.P.	36.38	34.93
2 tert.butyl 4 bromo phenol M.P.	35.85	34.93

# SUMMARY

1. Tertiary butyl alcohol will not condense in the presence of zinc chloride at or below 30° as with aluminum chloride.
2. Tertiary butyl alcohol and phenol will condense in the presence of zinc chloride and aluminum chloride mixture to give para tertiary butyl phenol, ortho tertiary butyl phenol, and 2,4 ditertiary butyl phenol.
3. Ortho and 2,4 ditertiary butyl phenol apparently exist in equilibrium with para tertiary butyl phenol when in the presence of zinc chloride and aluminum chloride mixture. It is possible to isolate them from this reaction mixture.
4. ~~Ortho~~ tertiary butyl phenol in the presence of aluminum chloride rearranges almost quantitatively to give para tertiary butyl phenol.
5. Para bromo phenol will not condense with tertiary butyl alcohol in the presence of aluminum chloride, zinc chloride, zinc chloride and aluminum chloride mixture, or in the presence of phosphoric acid.

SUMMARY Cont.

6. Apparently the para position is strongly favored when both the ortho and para positions are open for alkalyation.

7. The ortho alkalyated phenols are insoluble in 5% KOH while the para alkalyated isomers are soluble in this solution.

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Condensation of tert-butyl  
alcohol with phenol in the  
presence of anhydrous zinc  
chloride under thirty  
degrees temperature.

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