

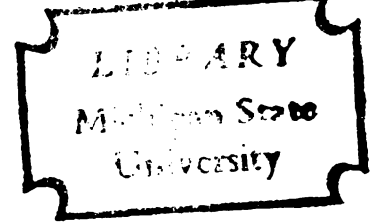


EVALUATION OF REDOX SYSTEMS AND
INDICATORS IN ACETIC ACID

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EVALUATION OF REDOX SYSTEMS AND INDICATORS
IN ACETIC ACID

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INTRODUCTION

INTRODUCTION

Previous work on oxidation in acetic acid has been done either potentiometrically or amperometrically. No work has been reported on the use of indicators (4,5). Previous workers have noted inconsistencies when using aqueous calomel electrodes for reference electrodes. It appears that the liquid junction between the acetic acid solution and aqueous potassium chloride in the asbestos fiber introduced a variable potential so that particular values observed depended on time, concentration, and other factors.

In the literature nothing was found concerning the behavior of redox indicators in acetic acid medium even though all of them were studied in aqueous media (1,2). The preparation of titanium (III) sulfate in acetic acid was not found in literature.

Ammonium hexanitratocerate (IV) has been used as an oxidizing agent because of its solubility in acetic acid, stability and oxidizing properties (4,5).

The object of this work was to develop a mercurous acetate reference electrode for the study of indicator behavior. Applications of indicators in redox titrations were studied.

EXPERIMENTAL

Reagents

1. Glacial acetic acid - Baker's "Analyzed" and Dupont "RG" both were used as solvent without further purification.
2. Sulphuric acid - Dupont "RG".
3. Perchloric acid - Mallinckrodt "AR" grade. Con. 70%, sp. gr. 1.6.
4. Sodium oxalate - Merck "reagent" primary standard purity.
5. Ferrous Ammonium sulfate - Baker's "Analyzed".
6. Anhydrous sodium perchlorate - G. Frederick Smith Chemical Co. "RG".
7. Ferric chloride - Purified "Fisher laboratory."
8. Ammonium Hexanitratocerate IV - G. Frederick Smith Chemical Co.
9. Ferrous perchlorate - G. Frederick Smith Chemical Co.
10. Iron powder - Baker's Chemical Co.
11. Potassium dichromate - Merck "reagent".
12. o-Phenanthroline - G. Frederick Smith Chemical Co.
13. Neutral red - Eastman Kodak Company (white label).
14. Methylene blue - pharmaceutical laboratories. National Aniline Division.
15. Diphenylamine - Eastman Kodak Company (white label).
16. Titanium metal - Dow Chemical Company.
17. Potassium iodide - Merck "reagent".
18. Potassium Iodate - Mallinckrodt "AR" grade.
19. Ceric Ammonium sulfate - G. Frederick Smith Chemical Co.
20. Sodium azide - "Dupont" practical grade.
21. Quinone - Eastman organic chemicals, practical grade (yellow label).

Preparation of Solutions

1. Sodium oxalate - $\text{Na}_2\text{C}_2\text{O}_4$

A 0.01 N solution of sodium oxalate in glacial acetic acid 1 N with respect to perchloric was prepared by dissolving 0.67 gram of sodium oxalate in a mixture of 89.20 ml. perchloric acid diluted to 1000 ml. with glacial acetic acid.

2. Ammonium hexanitratocerate (IV), $(\text{NH}_4)_2\text{Ce}(\text{NO}_3)_6$ -:

The reagent was dried at 105° for two hours and 5.48 grams were dissolved in one liter glacial acetic acid to give 0.01 N solution. Heating on a hot plate was required to dissolve all the salt. The solution was stored in dark bottle to prevent decomposition by light.

3. Titanous solution -:

A small amount of titanium metal was added to a mixture of 250 ml. glacial acetic acid plus 50 ml. of concentrated sulfuric acid in a round-bottomed flask fitted with a condenser and heated over a hot plate. The solution was heated until a deep violet-blue color developed. Too much heating is to be avoided because a precipitate forms on prolonged heating. The solution was poured into a dark bottle and cooled under an inert atmosphere. Nitrogen was used to prevent the oxidation of the titanous solution, which is very quickly oxidized by the oxygen of the air. The titanous solution was standardized with potassium dichromate dissolved in glacial acetic acid 2 N with respect to sulfuric acid. The titration was done potentiometrically.

4. Ferrous o-phenanthroline complex -:

This reagent was prepared in stoichiometric ratio so that three moles of o-phenanthroline combined with one mole of ferrous.

In an attempt to use ferrous perchlorate, a 0.01 N solution was prepared by dissolving 3.6185 gram of $\text{Fe}(\text{ClO}_4)_2 \cdot 6\text{H}_2\text{O}$ in 1000 ml. glacial acetic acid and a small amount of iron powder was added to reduce the ferric perchlorate which was present due to air oxidation of some of the ferrous perchlorate. The iron powder did not appear to reduce the ferric, but it precipitated, so the solution was filtered through a Gooch crucible. This solution was standardized with potassium permanganate in the usual way. Then 0.540 gram (3 moles.) of o-phenanthroline was dissolved in an amount of ferrous solution equal to 100 ml. of 0.01 N ferrous perchlorate (1 mole.). All of the o-phenanthroline went into solution forming the red complex, but part precipitated as the perchlorate salt. Therefore, this ferrous salt is unsatisfactory.

In an attempt to use ferrous ammonium sulfate 0.3921 gram was added to 100 ml. of glacial acetic acid followed by 0.54 gram of o-phenanthroline. The ferrous ammonium sulfate is only partially soluble in glacial acetic acid, but when the o-phenanthroline is added, the intense red color of the complex developed which means that the o-phenanthroline forms a complex with the ferrous which makes it dissolve.

Apparatus

A Beckman Glass electrode pH meter, Model H2, was used for potentiometric titrations. A mercurous acetate reference electrode which is described in detail later and a platinum electrode were used for detecting the end point. All titrations were done at room temperature. Magnetic stirring was not possible because the potentials observed varied as the magnetic field in the acetic acid varied with the rate of stirring. Provision was made to pass nitrogen through the solutions in some titrations.

Mercurous Acetate Reference Electrode

For a reference electrode in potentiometric titrations in acetic acid the ordinary calomel electrode which contains potassium chloride in aqueous solution could not be used because of the liquid junction potential produced at the surface of contact between the potassium chloride solution and acetic acid in the beaker. In addition there will be some diffusion of the glacial acetic acid into the potassium chloride solution which will vary the composition. So an electrode was made using acetic acid as the solvent.

The seal on a regular fiber type calomel electrode was softened by careful heating. The inside tube was removed and the bottom cut off with a diamond blade. The inside of the tube was cleaned and the new filling introduced.

First, mercury was introduced by turning the tube upside down. The amount of mercury and the proportion to other materials is shown in Fig. (1). The amount of mercury should be enough so that the platinum wire will dip into it.

Secondly, a paste was prepared by grinding mercurous acetate with mercury in a mortar. Mercurous acetate was used instead of mercurous chloride because the solvent was acetic acid and chloride might diffuse out of the electrode. Then the paste was moistened with a 0.5 M solution of sodium perchlorate in acetic acid. The moist paste was added to the tube on top of the mercury. The last material introduced into tube B was the asbestos fibers leaving a little space so that the tube could be restricted leaving a little hole which made possible the connection between the material inside tube A and the liquid outside it in tube B.

The asbestos fibers were moistened by adding 0.5 M sodium perchlorate in acetic acid with a syringe through the small hole. Care was taken to prevent the formation of an air bubble which will prevent the connection between tube A and B. Then tube A was introduced inside tube B and the top was sealed with sealing wax. Then through the side opening C tube B was filled with 0.5 M sodium perchlorate in acetic acid to the mark shown in Fig. (1) so that during a titration the surface of liquid inside the tube B is above the liquid in the beaker used for titrations. The sodium perchlorate was used to make the acetic acid a conducting liquid. The opening was closed with a cork to prevent the introduction of dust and to prevent the evaporation of the solution.

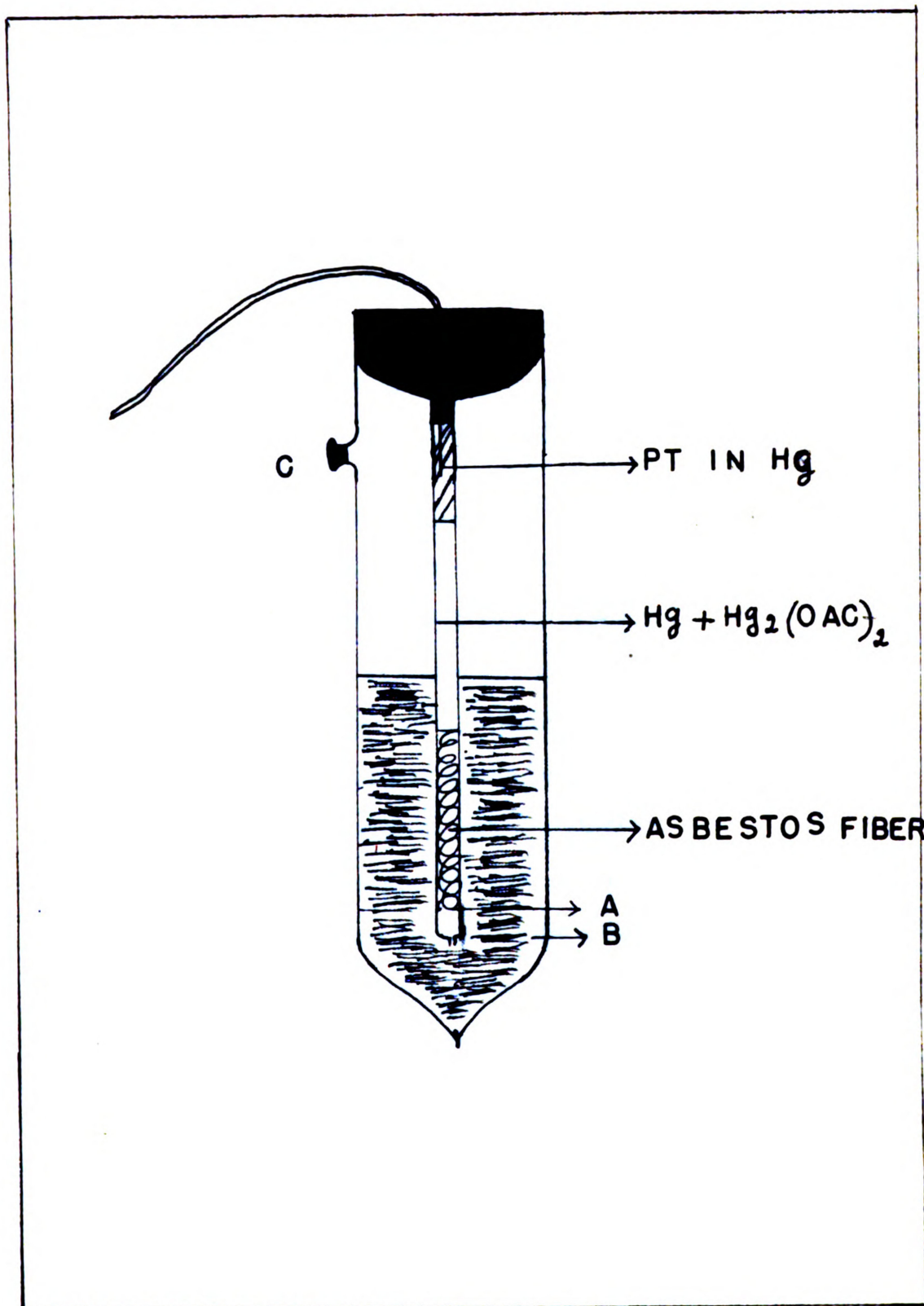


FIGURE · 1 · MERCUROUS ACETATE ELECTRODE

Indicator Studies

With the available mercurous acetate reference electrode, it was possible to study the behaviors of some indicators in glacial acetic acid medium by potentiometric titrations which was not done before. The formal potentials, the potentials at the end point and the potentials at the breaks were studied. In general indicator titrations are preferable because even if the color change coincides with the break in potential the potentiometric titrations are more time consuming.

1. o-Phenanthroline-ferrous complex :-

When the reduced form of the indicator was titrated potentiometrically with cerium(IV) the oxidised form was produced as shown by the color change from red to blue. The presence of a strong acid such as perchloric acid was necessary. When titrating the 0.01 N solution of the indicator in acetic acid alone, after two ml. of 0.01 N cerium(IV) had been added a precipitate formed, but in titrating the indicator which was 1 N with respect to perchloric acid no precipitate formed. The color change was from intense red to faint blue after the addition of two ml. of cerium(IV), and the color became brighter blue at the break which occurred at 0.920 volt. The formal potential for the indicator was 0.660 volt. The potentiometric titration curve was regular S-shaped. One milliequivalent of oxidising agent was used per millimole of indicator.

Investigations were made to find possible applications of the o-phenanthroline-ferrous complex as an indicator in oxidation-reduction titrations.

One of the attempts was the titration of 0.01 N sodium oxalate in acetic acid 1 N with perchloric acid with 0.01 N cerium(IV). One drop of 0.01 N o-phenanthroline-ferrous complex aqueous solution was added to the sodium oxalate in the beaker. The break in potential was at 0.950 volt. The formal potential for the oxalate oxidation was 0.745 volt. The indicator changed color at 0.860 volt from light red to light greenish-blue. The change in color was distinct and reversible and the oxidized form was attacked only slowly by excess reagent. The potentiometric titrations agreed with the ordinary titrations as shown in Table I.

TABLE I

TITRATION OF SODIUM OXALATE WITH CERIUM(IV)
o-PHENANTHROLINE COMPLEX AS INDICATOR

Ml. $\text{Na}_2\text{C}_2\text{O}_4$ Soln.	Ml. Ce(IV) (0.01 N) Indicator Potentiometric	
25	26.30	---
25	26.25	---
25	---	26.25
25	---	26.25

An attempt to use the 0.01 N aqueous solution of o-phenanthroline-ferrous complex as an indicator in the titration of 0.01 N ferrous perchlorate in acetic acid 1N with respect to perchloric acid with 0.01 N. Cerium(IV) was unsuccessful because there was no color change near the break in potential.

Another attempt was in the potentiometric titration of 0.01 N ferrous perchlorate in acetic acid 1 N in respect to sulphuric acid with potassium dichromate in presence of 1 drop of the indicator. This attempt was also unsuccessful because there was no color change near the break in potential.

The 0.01 N aqueous solution of o-phenanthroline-ferrous complex was successfully used as an indicator in the potentiometric titration of sodium azide with cerium IV. Before the indicator was tried, a potentiometric titration of sodium azide with cerium (IV) was made. The formal potential for the oxidation of azide was 0.600 volt and the break in potential was at 0.930 volt. It was predicted that the use of o-phenanthroline-ferrous complex as an indicator might be successful in titrating sodium azide with cerium IV. One ml. of 1 M sodium azide solution plus 50 ml. glacial acetic acid plus 4.4 ml. concentrated perchloric acid plus 1 drop of the 0.01 N aqueous solution of the o-phenanthroline ferrous complex were used. The change in color at the end point was from purple to light blue. The results are shown in Table II. These results were confirmed by the procedure of Martin (3) based on oxidation in aqueous medium.

To three ml. of the same azide solution in a glass-stoppered flask was added 50 ml. (excess) of 0.0990 N ceric sulfate standardized with sodium oxalate. The stoppered flask was shaken vigorously and allowed to stand for five minutes. Then 0.5 gram of potassium iodide was added, and the iodine released was titrated immediately with 0.1013 N sodium

thiosulfate. Near the end point one ml. of starch solution was added as an indicator. The results shown in Table II coincide with the results obtained by titration of azide in acetic acid with Cerium IV using the aqueous solution of o-phenanthroline complex as an indicator.

TABLE II
DETERMINATION OF AZIDE; OXIDATION WITH CERIUM (IV)

Azide Solution ml.	Ml. Ce IV 0.0197 N	Meq. $\text{Ce}(\text{SO}_4)_2$ used	NaN_3 meq./ml.
o-Phenanthroline Complex as Indicator			
1	13.20		0.2600
1	13.15		0.2591
1	13.10		0.2581
Martin's Method			
3		0.7752	0.2584
3		0.7752	0.2584
Nitro-o-phenanthroline Complex as Indicator			
1	17.2		0.3388
1	17.2		0.3388

2. Diphenylamine -:

Diphenylamine was titrated potentiometrically with 0.01 N cerium IV. An oxidized form was produced which was supposed to be diphenylbenzidine. The titration was unsuccessful because a precipitate formed even in the presence of perchloric acid and in addition no regular S-shaped curve was obtained. An attempt was made to titrate the same

0.01 N solution of diphenylamine in glacial acetic acid with 0.01 N potassium dichromate which is of lower potential than the cerium IV. The potentiometric titration was successful, but the presence of a strong acid such as perchloric acid was necessary. Because in the absence of perchloric acid a precipitate formed. Twenty-five ml. of 0.01 N indicator solution was diluted to 50 ml. with acetic acid made 1 N with respect to perchloric acid. It was found that the indicator was oxidized and produced a regular S-shaped curve with either 1 N or 0.5 N perchloric acid solutions.

In 1 N perchloric acid solution the formal potential for the indicator was 0.400 volt and the break in potential occurred at 0.600 volt. In 0.5 N perchloric acid solution the formal potential for the indicator was 0.360 volt and the break in potential occurred at 0.560 volt. The change in color was from colorless to dark violet after the addition of two ml. of dichromate solution. The solution became darker and darker near the break but there was no distinct color change near the break. The indicator consumed two milliequivalents of dichromate solution per millimole of indicator with either 1 N or 0.5 N perchloric solutions.

An attempt was made using diphenyl amine as an indicator in the potentiometric titration of ferrous perchlorate in acetic acid 1 N in sulfuric acid with potassium dichromate. The indicator was unsuccessful because there was no distinct color change near the break in potential.

Another attempt was done using diphenylamine as an indicator in the potentiometric titration of ferrous perchlorate in acetic acid 1 N

in perchloric acid with 0.01 N cerium(IV). The indicator failed to work because there was no color change near the break in potential.

A third attempt was tried using 1 drop of diphenylamine solution as an indicator in the potentiometric titration of potassium dichromate in acetic acid 1 N in sulfuric acid with titanous sulfate under an inert atmosphere of nitrogen. One drop of ferrous perchlorate solution in acetic acid was added to the beaker before the titration which was supposed to help the action of the indicator. The indicator was unsatisfactory because there was no color change near the break in potential.

Diphenylamine sodium sulfonate was tried instead of diphenylamine in the titration of the potassium dichromate system previously used with titanous sulfate. The prediction was that it might work better than diphenylamine, but it was found that this indicator was unsatisfactory also.

3. Methylene blue -:

Methylene blue chloride was titrated potentiometrically with titanous sulfate in acetic acid under an atmosphere of nitrogen. The presence of a strong acid such as sulphuric acid was necessary because in the absence of sulphuric acid a precipitate formed. Twenty-five ml. of 0.01 N methylene blue solution in acetic acid was diluted to 50 ml. with acetic acid and the solution was made 1 N with sulphuric acid. The titration curve was S-shaped. The formal potential was 0.165 volt and the break in potential was at 0.020 volt. For 2 N sulfuric acid

solution the formal potential was 0.175 volt, and the break in the potential was at 0.025 volt. The change in color was from blue to green and then to brownish purple. There was no distinct color change near the break in potential. Two milliequivalents of the reducing agent, titanium (III) sulfate were consumed per millimole of the indicator.

Many attempts were made to find applications of methylene blue as an indicator. One attempt was in the potentiometric titration of potassium dichromate with titanium (III). The attempt was unsuccessful because the change in color occurred earlier than the jump in potential. Another attempt was the potentiometric titration of ferric chloride with titanium (III) sulfate, but no jump was observed. The ferric chloride was insoluble in acetic acid so one gram of tartaric acid was added to form the complex ferric tartarate which was soluble in acetic acid. The third attempt was the titration of quinone with titanium (III) sulfate in the presence of three drops of methylene blue solution as an indicator. The indicator did not work because there was no color change near the break in potential.

4. Nitro-o-phenanthroline-ferrous complex :-

Investigation was made to find the possibility of using an 0.01 M aqueous solution of nitro-1-phenanthroline-ferrous complex as an indicator in the potentiometric titration of sodium oxalate 0.01 N in acetic acid made 1 N in respect to perchloric acid with 0.01 N cerium(IV). The result was satisfactory because the indicator changes color distinctly from red to colorless at 0.965 volt. The break in potential was at

0.950 volt. The formal potential for the oxalate oxidation was 0.635 volt. The potentiometric titrations agreed with the indicator titrations as shown in Table III.

TABLE III
TITRATION OF SODIUM OXALATE WITH CERIUM (IV)
NITRO-o-PHENANTHROLINE AS INDICATOR

Ml. $\text{Na}_2\text{C}_2\text{O}_4$	Ml. Ce (IV)	
	Indicator	Potentiometric
25	25.85	---
25	25.80	---
25		25.80
25		25.85

An attempt was made to use nitro-1-phenanthroline-ferrous complex as an indicator in titrating sodium azide with cerium(IV). It was found that the number of meq./ml. obtained by ordinary indicator titrations (Table II) did not coincide with the number of meq./ml. obtained by Martin's procedure (Table II).

5. Miscellaneous indicators -:

Phenosafranine was tried as an indicator in titrating ferric chloride with titanous sulfate. The indicator did not work because the change in color was unstable and there was no jump in potential during the titration. The ferric chloride solution was obtained by suspending ferric chloride in acetic acid and then adding perchloric acid in small

portions to help dissolve the ferric chloride which was incompletely soluble in acetic acid alone.

An 0.01 N solution of neutral red in acetic acid made 1 N and 2 N in respect to perchloric acid was titrated with titanous sulfate. It was found that in either solution the break in potential was too early and that was after the addition of one ml. of titanous sulfate. So this indicator was neglected because we could not get any information about the formal potential or the potential at the break which enables the prediction of using the neutral red as an indicator in various systems.

TABLE IV
FORMAL POTENTIAL OF REDOX SYSTEMS STUDIED

System	Formal Potential, mv.*	Potential at Color Change
$\text{Ce}^{+4}-\text{Ce}^{+3}$	1295	
Nitro-o-phenanthroline ferrous complex		965
$\text{Cr}^{+6}-\text{Cr}^{+3}$ (1 N H_2SO_4)	930	
$\text{Fe}^{+3}-\text{Fe}^{+2}$ (1 N HClO_4)	690	
$\text{C}_2\text{O}_4^{2-} \rightarrow 2\text{CO}_2$	620	
o-Phenanthroline ferrous complex	660	860
$2\text{H}_2^+ \rightarrow 3 \text{H}_2$	610	
$\text{Fe}^{+3} - \text{Fe}^{+2}$ (1 N H_2SO_4)	440	
Diphenylamine (1 N HClO_4)	400	
Quinone-hydroquinone (1 N H_2SO_4)	335	
Methylene blue (1 N HClO_4)	175	
$\text{Ti}^{+4} - \text{Ti}^{+3}$	-150	

*Vs. mercurous acetate electrode.

SUMMARY

SUMMARY

This work was done to study some reduction-oxidation systems, indicators, and applications to systems studied. From the titration curve the formal potential for both systems and indicators, the potential at the equivalence point, and the potential at which the indicator changed color were read.

Ammonium hexanitrate-oerate and potassium dichromate were used as oxidizing agents because of their solubility in acetic acid.

Titanous sulfate was used as a reducing agent and it was prepared by dissolving titanium metal in a mixture of acetic acid plus sulphuric acid. It was possible to obtain a variety of concentrations by varying the time of refluxing.

Titration were done potentiometrically using a specially prepared mercurous acetate reference electrode and a platinum measuring electrode. In some cases it was desirable to check the results by ordinary indicator titrations.

The following observations were made:

1. It was possible to work with titanous solution under an atmosphere of nitrogen in order to reduce the rate of oxidation of titanous by the atmospheric oxygen.
2. The cerium redox couples were affected by the presence of strong acids such as perchloric acid and sulphuric acid. As the amount of acid increased the break in potential of

1

2

3

the system increased. In case of the dichromate-chromic system sulphuric acid had the same effect. The titanium system was also affected by the presence of strong acid such as sulphuric acid.

3. Titanous sulfate was standardized potentiometrically with potassium dichromate.
4. All the indicators used were soluble in acetic acid and the solutions were stable for a long time.
5. The indicators which show possible applications are o-phenanthroline ferrous complex in the titration of sodium oxalate with cerium IV in 1 N perchloric acid and in the titration of sodium azide with cerium IV. Nitro-o-phenanthroline-ferrous complex applied in titration of sodium oxalate with cerium IV. Both indicators show a distinct and sharp color change at the end point.

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LITERATURE CITED

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APPENDICES

**Experimental Results
Arranged in the Order Mentioned
in the Thesis**

**Titration of o-Phenanthroline-Ferrous Complex 1 N in Respect to
Perchloric Acid with Cerium IV**

Ml.	Mvolt	Ml.	Mvolt	Ml.	Mvolt
0	370	20.1	689	22.8	990
5	640	20.2	690	23	1035
10	655	20.4	691	23.2	1100
11	658	20.7	699	23.4	1111
12	660	21	702	23.6	1128
13	661	21.2	710	24	1140
14	662	21.4	711	24.2	1142
15	667	21.6	718	24.4	1146
16	670	21.8	724	25	1150
17	671	22	750	26	1155
18	676	22.2	820	27	1160
19	681	22.4	890	28	1162
20	689	22.6	940	29	1163
				30	1163

**Titration of Sodium Oxalate with Ce IV in Presence of
o-Phenanthroline-ferrous complex as an Indicator**

Ml.	Mvolt	Ml.	Mvolt	Ml.	Mvolt
0	480	20	746	26.8	1125
0.5	490	25	770	27	1150
1	729	26.1	790	27.5	1160
2	735	26.2	825	28	1210
5	745	26.3	910	30	1250
6	745	26.4	970	35	1280
10	745	26.5	1020		
15	745	26.7	1080		



**Titration of Ferrous Perchlorate in Acetic Acid 1 N
in Perchloric Acid with Cerium IV**

ml.	mvolt	ml.	mvolt	ml.	mvolt
0	350	9.3	715	12.6	840
1	560	9.4	718	12.7	910
2	660	9.5	719	12.8	910
3	672	9.7	720	12.9	970
5	682	9.8	720	13	1010
7	690	10.1	721	13.1	1048
8	690	11.9	735	13.3	1130
8.2	692	12	739	13.5	1180
8.5	694	12.2	742	13.7	1200
9	700	12.3	760	14.2	1230
9.1	711	12.4	765	15	1255
9.2	714	12.5	765	20	1290

**Titration of Ferrous Perchlorate in Acetic Acid 1 N With
Sulphuric Acid with Potassium Dichromate**

ml.	mvolt	ml.	mvolt	ml.	mvolt
0	350	23	520	24	785
1	390	23.2	535	24.2	820
2	400	23.4	550	24.5	835
10	430	23.5	570	25	860
15	450	23.6	675	27	880
20	470	23.8	725	30	900
22	490	23.9	765	40	930



**Titration of Sodium Azide in Acetic Acid 2 N
in Perchloric Acid with Cerium IV**

Ml.	Mvolt	Ml.	Mvolt	Ml.	Mvolt
0	603	12.2	610	13.6	1250
2	608	12.4	620	13.8	1255
4	610	12.6	630	14	1260
6	610	12.8	630	15	1275
8	610	13	635	20	1300
10	610	13.2	1000	25	1310
12	610	13.4	1230		

**Titration of Diphenylamine in Acetic Acid 1 N in
Perchloric Acid with Cerium IV**

Ml.	Mvolt	Ml.	Mvolt	Ml.	Mvolt
0	380	20.7	500	23	825
2	381	20.9	570	24	840
5	382	21	640	25	850
10	392	21.2	705	26	855
12	400	21.4	749	28	860
13	400	21.6	770	30	865
14	402	21.8	780	32	870
15	410	22	790	35	875
16	410	22.3	805	50	890
18	422	22.4	810		
20	450	22.6	812		
20.2	460	22.8	820		
20.4	468				



**Titration of Potassium Dichromate in Acetic Acid 1 N in
Respect to Sulphuric Acid with Titanous Sulfate**

ml.	Mvolt	ml.	Mvolt	ml.	Mvolt
0	930	43.2	868	44.55	440
10	950	43.4	860	44.6	320
15	942	43.6	855	44.65	200
20	939	43.8	849	44.70	80
25	932	44	840	44.8	-58
30	925	44.1	840	44.9	-60
35	910	44.2	838	45.5	-75
40	900	44.35	650	47	-100
42	880	44.45	615	49	-120
43	870	44.5	560	51	-130

**Titration of Methylene Blue in Acetic Acid 1 N in
Perchloric Acid with Titanous Sulfate**

ml.	Mvolt	ml.	Mvolt	ml.	Mvolt
0	250	35.7	130	37.2	60
5	200	35.9	129	37.3	-40
10	185	36	125	37.5	-48
15	175	36.2	121	37.7	-50
20	170	36.4	119	37.8	-55
25	162	36.8	105	38	-60
30	152	36.9	100	39	-78
35	138	37	90	40	-85
35.5	131	37.1	75	42	-100

Titration of Ferric Chloride with Titanous Sulfate

Ml.	Mvolt	Ml.	Mvolt	Ml.	Mvolt
0	340	7	275	13	210
2	315	8	260	14	185
3	292	9	260	15	170
4	285	10	259	16	150
5	280	11	245	18	120
6	280	12	230	22	80

Titration of Quinone in Acetic Acid 1 N in Respect to Sulphuric Acid with Titanous Sulfate

Ml.	Mvolt	Ml.	Mvolt	Ml.	Mvolt
0	375	26	318	27.6	250
1	370	26.2	312	27.8	80
2	355	26.4	310	28	50
5	350	26.6	308	28.2	-55
10	345	26.8	305	28.4	-62
15	340	27	300	29	-75
20	335	27.2	290	30	-90
25	321	27.4	280	33	-105
				35	-110

Titration of Sodium Oxalate with Ce IV in Presence of Nitro-o-Phenanthroline-Ferrous Complex as an Indicator

Ml.	Mvolt	Ml.	Mvolt	Ml.	Mvolt
0	640	23	720	25.85	885
5	660	24	730	26	1140
10	680	25	740	26.4	1185
15	690	25.3	745	27	1210
20	710	25.5	750	30	1250
21	712	25.7	760	35	1270
22	715	25.8	790		

Titration of Titanous Sulfate with Potassium Dichromate

Ml.	Mvolt	Ml.	Mvolt	Ml.	Mvolt
0	-180	23	235	24.5	600
10	-150	23.1	270	24.8	650
15	-150	23.3	245	25	655
20	-120	23.5	361	25.5	730
22	- 89	23.7	450	26	750
22.4	- 65	23.9	500	27	790
22.6	- 45	24.1	540	29	810
22.8	130	24.3	560	31	830
				40	855

**Titration of Cerium IV with Sodium Oxalate in
Acetic Acid 1 N in Perchloric Acid**

Ml.	Mvolt	Ml.	Mvolt	Ml.	Mvolt
0	810	15.8	1200	17	780
3	1270	16	1160	17.5	770
5	1280	16.2	950	19	760
10	1280	16.4	860	25	750
15	1250	16.6	810	30	740
15.5	1230	16.8	600		

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