SPECTROPHOTOMETRIC METHODS FOR THE DETERMINATION OF ALCOHOLS AND TELLURIC ACID

by

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INTRODUCTION

This thesis is presented in two sections, determination of alcohols and determination of telluric acid.

Alcohols

The study on alcohols was undertaken to determine whether the rate of color decay of the colored complex formed when an alcohol is mixed with ammonium-hexanitrato-cerate reagent could be used as a quantitative method of alcohol determination. Simple and rapid methods, especially for the determination of low concentrations of ethyl alcohol in small samples (e. g., blood alcohol level), are needed. It was felt that a spectrophotometric method suitable for direct determination of alcohol might prove to be more simple and rapid, and also more accurate than existing methods.

Telluric Acid

A need existed for a method of determining telluric acid in the presence of small amounts of other acids. Prior to this study, the methods which had been used for the determination of telluric acid were involved, time-consuming, and required careful control of experimental conditions. These methods were not entirely satisfactory, since occluded acids, especially nitric, gave high results. This is a problem because, in the recrystallization of telluric acid, nitric acid is commonly used as a precipitating agent. Since telluric acid solutions absorb in the ultraviolet, it was felt that a spectrophotometric method might possibly eliminate the interference by other acids and be used for rapid and accurate determinations for the pure telluric acid.

DETERMINATION OF ALCOHOLS

Literature Survey

A search of the literature revealed several qualitative and quantitative methods for determination of alcohols. These methods include boiling or freezing point changes; oxidation with bromine, potassium permanganate, or potassium dichromate under varied conditions, with determination either of the amount of oxidant used or of the products of oxidation; electrolytic oxidation; preparation of derivatives; and mass spectrometer. Because of their excessive number, references to these methods are not listed in this study.

Duke and Smith (3) reported the use of hexanitrato- or perchlorato-cerate anions in acid solution as a qualitative test for alcohols. A red complex is formed immediately when the reagent is mixed with an alcohol solution. However, this color is not stable. The color produced with tertiary butyl alcohol is more stable than the color produced with primary or secondary butyl alcohols.

In 1945, Duke (2) reported the use of aqueous ammonium hexanitrato-cerate for quantitative determination of alcohols up to 2.5 millimoles per 25 ml. of solution with approximately 5 per cent accuracy. He measured the extinction at 475 mμ at a definite time after mixing. He states that the decay of color is a straight line function for the first 15 minutes, and that the value obtained can be corrected to zero time. The amount of alcohol can then be determined from empirical calibration curves. The rate of decay varies, permitting identification of the alcohol in some instances.

Reid and Truelove (8) also reported the use of the colored complex for alcohol determination, giving data for six alcohols in concentrations from 0.1 per cent (W/V) to 0.8 per cent. Their method uses 5 ml. of alcohol and

2 ml. of acidic cerate reagent against a reagent blank and requires evaluation of the absorption exactly 5 minutes after mixing. The wave lengths used were those which pass a Hilger No. OG1 olive green filter.

Reagents and Equipment

1. Cerate reagent. Ammonium hexanitrato-cerate (200 grams) was dissolved in sufficient 2N nitric acid to make 500 ml.

2. Alcohols. Ethyl alcohol (95%) and the other alcohols used (assumed to be 100 per cent) were diluted to give stock concentrations of 5 or 10 per cent by volume. The concentration of the 10 per cent ethyl alcohol was checked with an immersion refractometer.

3. Beckman Model DU spectrophotometer equipped with a water-jacketed lamp housing, a set of thermospacers, and 1-cm. corex absorption cells.

4. Constant temperature bath (Sargent S-84860).

5. Centrifugal pump for circulating water from the constant temperature bath through the thermospacers and lamp housing cooling coils of the spectrophotometer.

Experimental Data

The initial experimental data was obtained prior to the installation of the water-jacketed lamp housing and cell compartment. After the cerate reagent was prepared, an absorption curve was determined for 1 part reagent plus 2 parts water, using a water blank. The resulting curve showed a sharp decrease in absorbancy between 450 mμ and 500 mμ, with a levelling off above that wave length. Therefore, it was considered desirable to carry out the determination at about 500 mμ.

The solutions were made up by adding 2 ml. of alcohol to 1 ml. of reagent and diluting the mixture to 10 ml. The absorbancies of the solutions were measured at given intervals after mixing. The rate of decay, which was linear up to 15 to 20 minutes, showed a definite decrease as the alcohol concentration was decreased (Fig. 1). However, since it was desired to determine lower concentrations of alcohol, other amounts of reagent and other wave lengths were tried.

In runs at 470 mμ, using a cerate reagent blank, the absorbancy was found to be a maximum (for 1 per cent ethyl alcohol) when 2 ml. of reagent plus 2 ml. of alcohol were used for 10 ml. of solution. Therefore, this amount of reagent was tried at 500 mμ and at 490 mμ with varied concentrations of ethyl alcohol down to 0.25 per cent, using a cerate reagent blank and also a water blank. For the determinations at 480 mμ, the temperature-controlling devices had been installed.

Determinations were made also with methyl alcohol, butyl alcohol, and tertiary butyl alcohol, using them in concentrations of 2 per cent to 0.2 per cent. The 2 per cent methyl alcohol gave a much higher absorbancy than the 2 per cent ethyl alcohol and 2 per cent butyl alcohol. These in turn gave a deeper color than the 2 per cent tertiary butyl alcohol. The first absorbancy readings were taken 1.5 minutes after mixing.

The initial color decay was studied by adding 1 ml. of cerate reagent to 4 ml. of alcohol with no further dilution. In this way the first absorbancy readings were obtained 30 seconds after mixing.

The absorption of the reagent was checked and the absorption curve for the alcohol-reagent complex was determined (Table 1 and Fig. 2), by using tertiary butyl alcohol (the most stable of the alcohols tested). These absorption curves show that the most satisfactory wave length for the

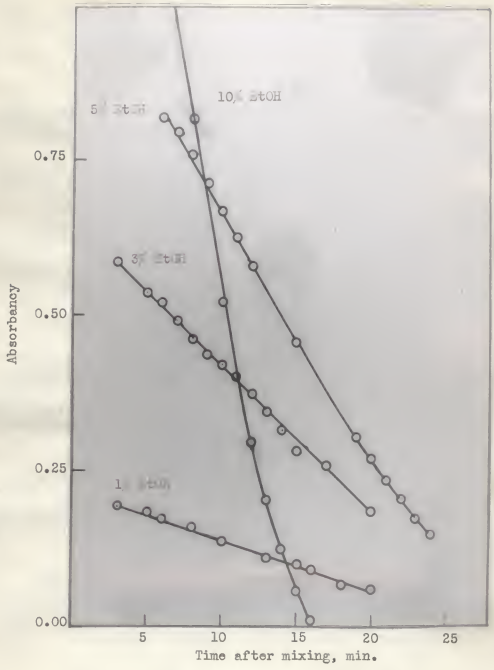

Fig. 1. Effect of alcohol concentration on rate of color decay, 500 mμ and reagent blank.

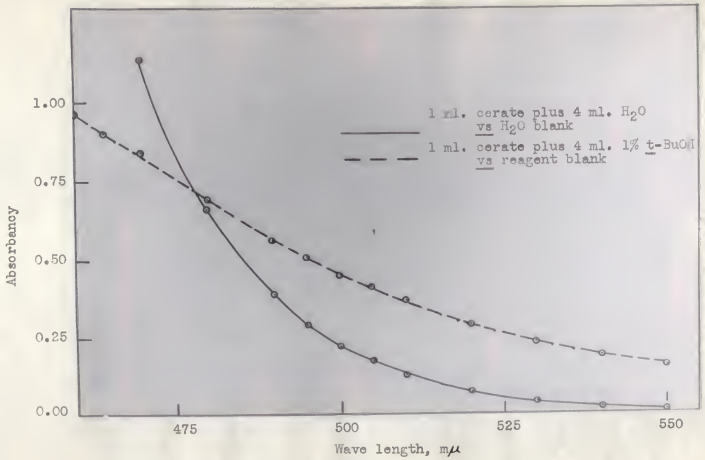

Fig. 2. Absorption curves for reagent and reagent-alcohol complex at 22° C.

determination is between 500 mμ and 510 mμ.

Table 1. Absorptions at 22° C. for reagent using H_2O blank and for alcohol-reagent complex using a reagent blank.

Wave length, mμ	Absorbancy		
	Reagent	Complex	Difference
460	1.89	0.966	-0.99
470	1.13	0.840	-0.29
480	0.660	0.690	0.030
490	0.390	0.560	0.170
495	0.295	0.505	0.210
500	0.220	0.450	0.230
505	0.174	0.410	0.236
510	0.130	0.367	0.237
520	0.072	0.293	0.221
530	0.040	0.234	0.196
540	0.019	0.188	0.169
550	0.007	0.149	0.142

With the temperature of the cell compartment at 22° C. and wave length set at 500 mμ, the absorbancy of the cerate reagent plus ethyl alcohol solutions was determined for concentrations from 0.05 per cent to 2.0 per cent, using a cerate blank, and for concentrations from 0.05 per cent to 0.8 per cent, using a water blank. The rate of decay decreased as the concentration of the alcohol decreased until, with the 0.05 per cent alcohol, the color was nearly stable. These plots of absorbancy *versus* time for ethyl alcohol show that the decrease in absorbancy is linear for the period between 2 and 5 minutes after mixing, with levelling off after 5 minutes have elapsed. Extrapolation of these plots

Table 2. Initial absorbancy of 1 ml. reagent plus 4 ml. alcohol at 500 mμ and 22° C. using reagent blank.

EtOH		BuOH		iso-BuOH		t-BuOH		iso-PrOH		MeOH	
0.05%	0.075	0.1%	0.141	0.2%	0.300	0.2%	0.103	0.2%	0.168	0.1%	0.185
0.10%	0.142	0.2%	0.255	0.4%	0.560	0.4%	0.192	0.4%	0.318	0.2%	0.350
0.20%	0.270	0.3%	0.364	0.6%	0.812	1.0%	0.463	1.0%	0.765	0.4%	0.671
0.25%	0.333	0.4%	0.489	0.8%	1.093	2.0%	0.888	2.0%	1.533	1.0%	1.612
0.30%	0.398	0.5%	0.600	1.0%	1.341						
0.40%	0.530	0.6%	0.712								
0.50%	0.654	0.8%	0.934								
0.60%	0.778	1.0%	1.168								
0.80%	1.014										
1.00%	1.253										
1.25%	1.573										
1.5%	1.858										
2.0%	2.40										

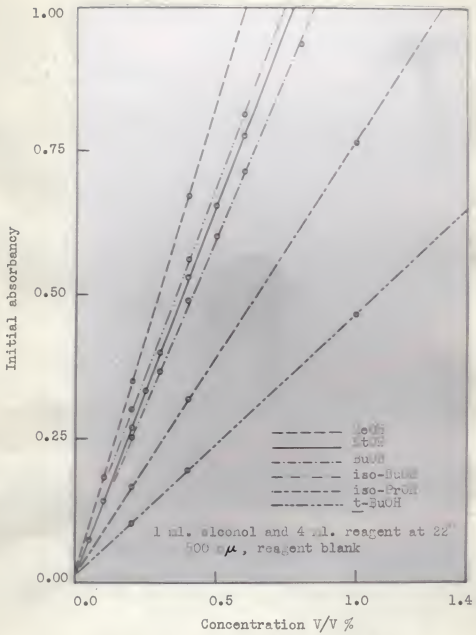

Fig. 3. Calibration curves.

back to zero time gives a value for the initial absorbancy of the alcohol-reagent mixture. The true initial absorbancies are higher than those obtained by this method, but are not readily evaluated.

Table 2 and Fig. 3 show the linear relationship of initial absorbancy to concentration from 0.05 per cent to 1.5 per cent ethyl alcohol.

Figure 4, in which per cent transmittancy was plotted against log concentration (Ayers (1) and Ringbom (9)), shows that the range for greatest relative accuracy in determination of ethyl alcohol concentration by this method is between 0.16 per cent and 0.5 per cent. The initial absorbancies with the water blank show also a linear relationship to concentration. This line is parallel to that obtained with a cerate reagent blank (Table 3 and Fig. 5), but shows higher absorbancies.

Table 3. Initial absorbancy of 1 ml. cerate reagent plus 4 ml. ethyl alcohol at 500 mμ and 22° C.

Conc EtOH, per cent	Cerate reagent blank	Water blank
0.05	0.075	0.309
0.10	0.142	0.376
0.20	0.270	0.505
0.25	0.333	0.567
0.30	0.398	0.630
0.40	0.530	0.756
0.50	0.654	-----
0.60	0.778	1.017
0.80	1.014	1.247

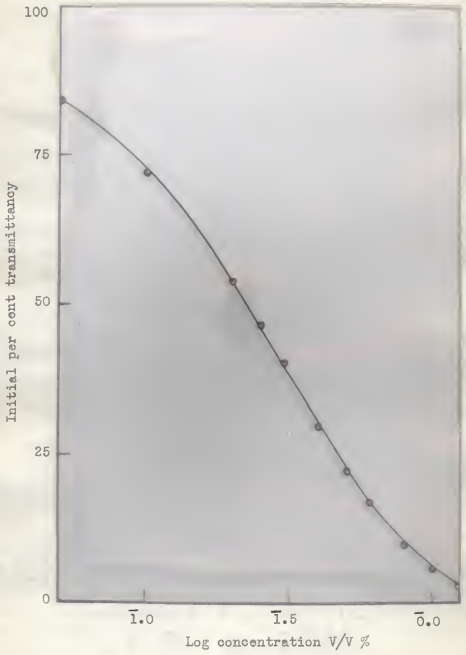

Fig. 4. Ringbom plot, EtOH, 500 mμ, 22° C.

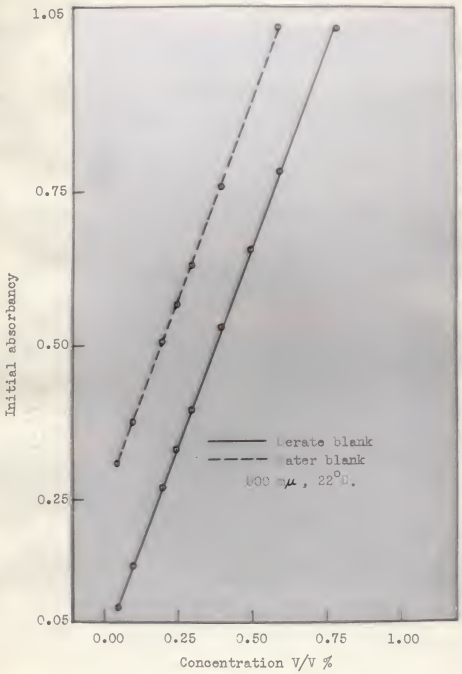

Fig. 5. Calibration curves, EtOH

The absorbancies versus time at 22° C. and 500 mμ were also determined for methyl alcohol, iso-propyl alcohol, butyl alcohol, iso-butyl alcohol, and tertiary-butyl alcohol, and these curves were extrapolated to obtain the initial absorbancy. Figure 3 and Table 2 show that a different slope was obtained for each of the alcohols determined.

Stability of Reagent. A check was made on the stability of the cerate reagent as diluted for a blank (2 ml. reagent per 10 ml. of solution), using a water blank. After the first 10 minutes, the absorbancy at 500 mμ was effectively constant for at least 2 hours in a cell compartment kept at 20° C. (Table 4).

Table 4. Stability of cerate reagent blank.

Time, min.	Absorbancy
2.0	0.232
4.5	0.230
6.0	0.229
7.5	0.228
9.5	0.228
15.	0.227
25.	0.226
33.	0.225
60.	0.225
90.	0.224
120.	0.224

The cerate reagent stock was also stable at room temperature for at least 9 months as shown by absorbancy determinations run with old and new reagent

stock on several concentrations of ethyl alcohol at 500 mμ. Data are given (Table 5) for the 1 per cent ethyl alcohol.

Importance of Temperature Control. A temperature dependence study using

Table 5. Stability of cerate reagent stock.

Time after mixing, min.	Absorbancy of 2 ml. reagent plus 2 ml. 1% EtOH diluted to 10 ml. vs reagent blank at 500 mμ and 22° C.	
	Old reagent	New reagent
1.0		
1.5	0.311	
2.0		0.311
2.5	0.308	0.310
3.0	0.305	0.309
3.5	0.304	0.305
4.0	0.302	0.302
4.5	0.299	
5.0		
5.5	0.297	0.299
6.0		0.297
6.5	0.295	
7.0	0.294	0.295
7.5		0.294
8.0	0.292	0.293
8.5	0.291	0.292
9.0	0.290	0.291
9.5	0.289	0.290
10.0	0.288	0.289

ethyl alcohol (0.6 per cent, 0.4 per cent, and 0.3 per cent) indicates the necessity of controlling temperature to obtain accurate and reproducible results. The data show that the rate of color decay and the initial absorbancy increase with increasing temperature. Figure 6 is a graphical representation of the data for the 0.6 per cent ethyl alcohol.

Discussion and Conclusions

In this study it was found that the rate of color decay is indicative of the concentration, but that the initial or zero time absorbancy of the ammonium-hexanitrato-cerate-alcohol complex provides a better method for quantitative determination.

Reid and Trueleve (8) plotted absorbtiometer drum differences, obtained exactly 5 minutes after mixing, *versus* concentrations from 0.1 to 0.8 per cent (W/V) and obtained a curvilinear plot. However, in this study the initial absorbancy was found to be a linear function of the alcohol concentration, when using 1 ml. reagent (200 g. ammonium hexanitrato-cerate in sufficient 2N nitric acid to make 500 ml.) plus 4 ml. alcohol at 500 mμ and 22° C.

The initial absorbancies were obtained by extrapolation back to zero time of the linear portion of absorbancy *versus* time plots for the various alcohols and concentrations. The slope of an initial absorbancy *versus* concentration plot (Fig. 3) differs for each of the alcohols studied (ethyl, methyl, butyl, iso-butyl, tertiary-butyl, and iso-propyl). This difference suggests that, in some instances, determination of the concentration of each alcohol in a mixture of two alcohols might prove feasible.

Figure 4 shows that the range for greatest relative accuracy in the determination of ethyl alcohol concentration by the proposed method is between

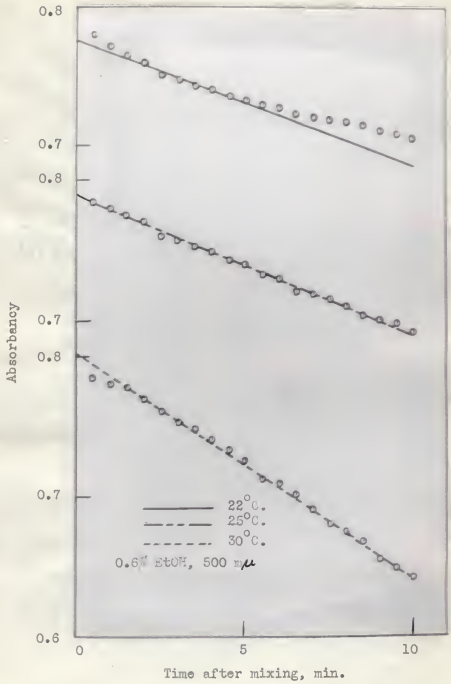

Fig. 6. Temperature dependence study.

0.16 and 0.5 per cent (V/V). This upper limit is twice the concentration given by Duke (2). Beers law is followed for ethyl alcohol from 0.05 to 1.5 per cent (V/V), permitting determinations at still higher concentrations. Duke also states that the tertiary-butyl alcohol complex is stable and the present study shows a definite, although slight, decrease in absorbancy with time for the tertiary-butyl alcohol complex. This complex is much more stable than the complexes formed with other alcohols. However, the decay of color must be considered when determining concentration.

None of the previously published works on the use of this complex for alcohol determination make any mention of the necessity for temperature control. From this study, it is evident that control of temperature within 1 or 2 degrees is essential for accurate and reproducible results in quantitative determinations.

The reagent as diluted for a blank was found to be stable for at least 2 hours at 20° C. The reagent was found to be stable for at least 9 months at room temperature.

The data of Fig. 5 on initial absorbancy suggest use of a water blank to determine ethyl alcohol at concentrations lower than 0.16 per cent. Higher absorbancy readings are obtained with the water blank than with the cerate reagent blank. A filter photometer could probably be used instead of the Beckman spectrophotometer if some means for temperature control and a filter passing wave lengths between 500 mμ and 510 mμ were available.

DETERMINATION OF TELLURIC ACID

Literature Survey

A search of the literature reveals various methods for the determination of telluric acid, including refractometric analysis, Urban and Meloche (11); acid-base titration, Rosenheim and Weinheber (10); and oxidation-reduction methods, Gooch and Howland (5) and Lingane and Niedrach (7).

Reagents and Equipment

1. Telluric acid prepared by the method of Horner and Leonard (6) and purified by repeated recrystallization from water.

2. Selenic acid prepared by the method of Gilbertson and King (4).

3. Other reagents of reagent grade.

4. Beckman Model DU spectrophotometer equipped with a water-jacketed lamp housing, a set of thermospacers, and 1-cm. silica absorption cells.

5. Constant temperature bath (Sargent S-84860).

6. Centrifugal pump for circulating water from the constant temperature bath through the thermospacers and lamp housing cooling coils of the spectrophotometer.

Experimental Data

Preliminary investigations indicated that at least 2 species of telluric acid existed in aqueous medium. The equilibrium between these species appears to be pH dependent. Figure 7 shows the shift of the telluric acid absorption spectrum with increasing concentration of sodium hydroxide. The absorbancy of a given concentration of telluric acid and, therefore, the sensitivity of

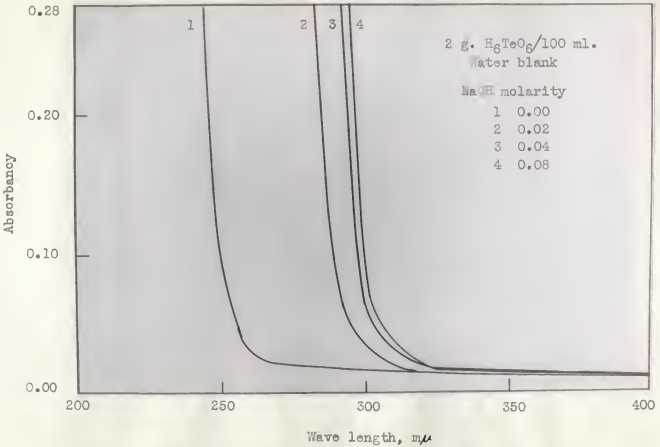

Fig. 7. Absorption curves.

a spectrophotometric determination, is increased by the addition of base. Because potassium hydroxide and sodium hydroxide were found to have greater absorbancies than ammonium hydroxide, the latter was selected for the determination.

Absorption data were obtained on solutions of nitric acid, hydrochloric acid, perchloric acid, and sulfuric acid. Curves of this data show that the absorbancy of chloride, perchlorate, and sulfate ions should not interfere to any extent above 250 mμ . The nitrate ion showed a definite absorbancy up to 330 mμ . However, the nitrate ion absorbancy curve shows a minimum at 260 mμ and a maximum at 300 mμ . Since the absorbancy of tellurate, even in the presence of base, is too low at wave lengths which would permit determination without interference from nitrate ion absorbancy, 260 mμ was selected for this study.

To determine the optimum concentration of base, data were obtained on two different concentrations of telluric acid (Fig. 8). Curves from this data showed that the absorbancies of the 2 concentrations of telluric acid, with increasing concentration of ammonium hydroxide, became essentially constant at 0.66 M ammonium hydroxide, although there is a twenty-fold difference in the concentrations of telluric acid. The 0.08 M telluric acid is about 6 times more concentrated than the highest concentration that could be measured at 260 mμ with 0.66 M ammonium hydroxide.

The method used for the remaining studies was as follows:

The amount of stock telluric acid to give the final concentration desired was placed in a 100 ml. flask and 10 ml. of 6.6 M ammonium hydroxide added. After mixing, the flask was filled to the mark with distilled water and placed in a constant temperature bath until temperature equilibrium was reached. The absorbancy was then measured at 260 mμ with the cell compartment at the same

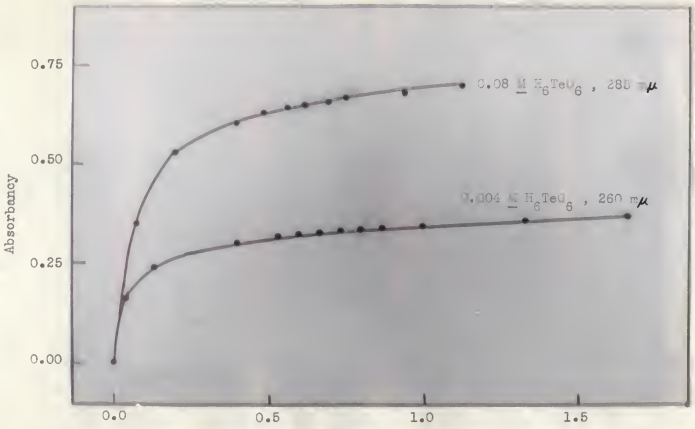

Fig. 8. Saturation curves.

temperature as the bath. 0.66 M ammonium hydroxide was used as a blank. Table 6 gives data for Fig. 9, a typical calibration plot which shows that the absorbancy of telluric acid solutions in 0.66 M ammonium hydroxide follows Beers law up to a concentration of 156 mg/100 ml. with only a slight steepening of the slope above that concentration.

Figure 10, in which per cent transmittancy was plotted against log concentration (Ayers (1) and Ringbom (9)) shows that the optimum concentration range for the conditions used is from 65 to 210 mg. of telluric acid per 100 ml. In this range, the relative analysis error is less than 0.8 per cent for a precision of 0.2 per cent in measuring the transmittancy. Concentrations of telluric acid lower than 65 mg/100 ml. can be determined at 260 mμ , but the relative analysis error will increase as the concentration of the sample is decreased. Analyses of 10 separately prepared samples, each containing 92 mg. telluric acid, gave an average value of 91.94 mg. with an average deviation of 0.11 mg., a range of 0.52 mg., and a standard deviation of 0.16 mg.

Effect of Temperature. Determination of the effect of temperature on absorbancy (Fig. 11) shows that there is a change of 0.002 in the absorbancy per degree change in temperature. The absorbancy was determined at 21° C. before and after the determinations at the higher temperatures and the same value obtained. This indicated that no permanent change in the adsorbing species took place when the temperature was raised. Since the temperature of the Beckman cell compartment was easily maintained within plus or minus 0.5 degree, the error due to temperature fluctuation was well within instrumental reading errors.

Interferences. The maximum amount of several possible contaminants which could be present without interference was determined (Table 7). The absorbancies of series of solutions containing the same concentration of

Table 6. Absorbancy at 22° ± 0.5° and 260 mμ of H_6TeO_6 solutions in 0.66 M NH_4OH, using an 0.66 M NH_4OH blank.

Mg H_6TeO_6/100 ml.	Corrected Absorbancy	Mg H_6TeO_6/100 ml.	Corrected Absorbancy
9.2	0.038	174.8	0.685
18.4	0.073	184.0	0.723
27.6	0.110	193.2	0.762
36.8	0.141	202.4	0.804
46.0	0.175	211.6	0.845
55.2	0.208	220.8	0.887
64.4	0.242	230.0	0.937
73.6	0.277	239.2	0.984
82.8	0.315	248.4	1.034
92.0	0.351	257.6	1.075
101.2	0.385	266.8	1.135
110.4	0.418	276.0	1.180
119.6	0.455	285.2	1.235
128.8	0.495	294.4	1.298
138.0	0.527	303.6	1.337
147.2	0.565	312.8	1.399
156.4	0.599	322.0	1.460
165.6	0.645		

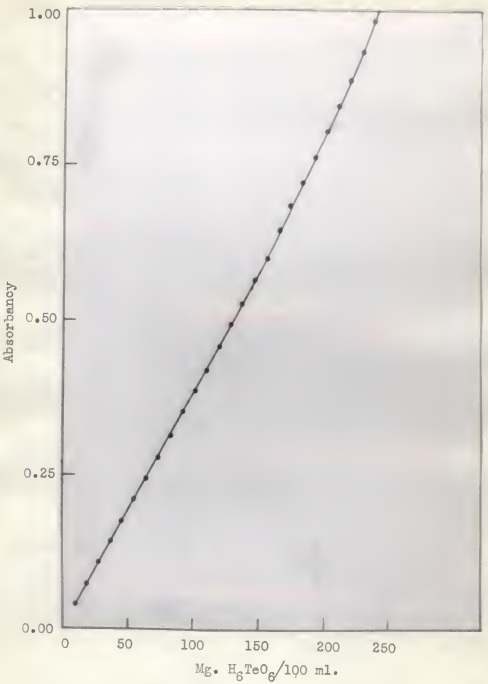

Fig. 9. Calibration curve for H_6TeO_6 at 260 mμ and 21° C.

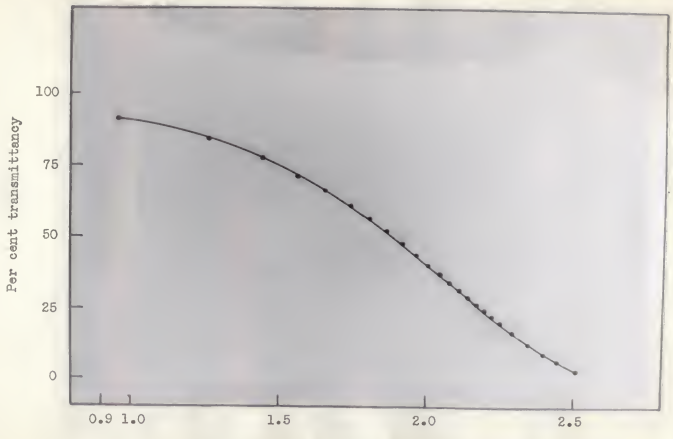

Fig. 10. Ringbom plot H_6TeO_6 , 260 mμ , 21°C.

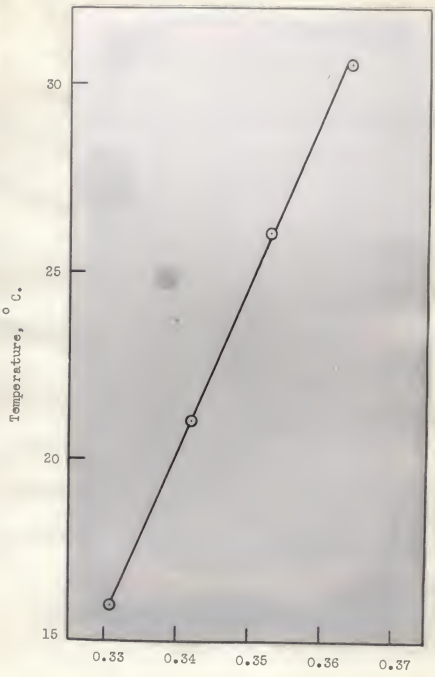

Fig. 11. Temperature dependence study, 0.004 M H_6TeO_6 in 0.66 M NH_4OH at 260 mμ, using a 0.66 M NH_4OH blank.

telluric acid and ammonium hydroxide with increasing concentrations of contaminant were determined. The maximum concentration of each impurity which did not interfere is given in Table 7. No study was made on the interaction of these contaminants when present in the same solution.

Table 7. Impurity levels above which interference occurred in determination of H_6TeO_6 (92 mg/100 ml.) at 260 mμ and 21° C. in 0.66 M NH_4OH.

Impurity	Allowable concentration, mg/100 ml.
HNO_3	9.6
H_2SO_4	9.8
H_2SeO_4	2.9
HCl	3.6
$HClO_4$	15.0
H_3PO_4	4.0
$HC_2H_3O_2$	6.0
NaOH	3.0

Discussion and Conclusions

The procedure developed in this study provides a simple, rapid, direct, and precise method for the determination of telluric acid. It is applicable within the limits given in Table 7, to the determination of telluric acid in the presence of other acids or bases.

Although temperature control is required when using this method, readily available thermostatic devices are suitable for maintaining temperatures within plus or minus 0.5 degree. The refractometric method suggested for telluric acid requires more accurate temperature control apparatus. The

reproducibility of absorbancy readings at a given temperature after heating and cooling the solutions shows that solutions could be left at room temperature after mixing until the analyst was ready to complete the determination.

The permissible concentration of impurities may be extended by a study of the particular ions present. With nitrate ion, absorbancy readings at 260 mμ and 300 mμ would permit determination of nitrate ion and telluric acid. The usual calculations are simplified in this case because telluric acid does not absorb at 300 mμ. The use of a buffer of ammonium hydroxide and ammonium sulfate, instead of just ammonium hydroxide, will help to extend the permissible limits of impurities. These modifications would not be general, but would apply only to the conditions for which they were developed.

ACKNOWLEDGMENT

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SPECTROPHOTOMETRIC METHODS FOR THE DETERMINATION OF ALCOHOLS AND TELLURIC ACID

by

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The thesis is presented in two sections, determination of alcohols and determination of telluric acid. Both studies were done with the Beckman spectrophotometer equipped with a water-jacketed lamp housing and a set of thermospacers.

Alcohols

The study on alcohols was undertaken to determine whether the rate of color decay of the colored complex formed when an alcohol is mixed with ammonium-hexanitrato-cerate reagent could be used as a quantitative method of alcohol determination. Simple and rapid methods, especially for the determination of low concentrations of ethyl alcohol in small samples (e. g., blood alcohol level), are needed. It was felt that a spectrophotometric method suitable for direct determination of alcohol might prove to be more simple and rapid, and also more accurate than existing methods.

The reagent, as used in this study, was 200 g. ammonium hexanitrato-cerate in sufficient 2N nitric acid to make 500 ml. Both reagent and alcohol were equilibriated in a constant temperature bath before mixing. The method developed was the addition of 1 ml. of cerate reagent to 4 ml. of alcohol, with no further dilution. With the temperature of the cell compartment at 22° C. and wave length set at 500 mμ, the absorbancy of cerate plus alcohol solutions was determined, using a cerate blank. The rate of decay decreased as the concentration of alcohol decreased. These plots of absorbancy versus time showed that the decrease in absorbancy was linear for the period between 2 and 5 minutes after mixing, permitting extrapolation back to zero time.

It was found that the rate of color decay was indicative of the alcohol concentration, but that the initial or zero time absorbancy provided

a better method for quantitative determination. The initial absorbancy followed Beer's law for ethyl alcohol from 0.05 per cent to 1.5 per cent (V/V), with the range for greatest relative accuracy between 0.18 per cent and 0.50 per cent.

The reagent as prepared was found to be stable at room temperatures for at least 9 months and, when diluted for use as a blank, to be stable at 22° C. in the spectrophotometer cell compartment for at least 2 hours.

None of the previously published works on the use of this complex for alcohol determination make any mention of the necessity for temperature control. From this study, it is evident that control of temperature within 1 or 2 degrees is essential for accurate and reproducible results in quantitative determinations.

Telluric Acid

A need existed for a method of determining telluric acid in the presence of small amounts of other acids. Prior to this study, the methods which had been used for the determination of telluric acid were involved, time-consuming, and required careful control of experimental conditions. Since telluric acid solutions absorb in the ultraviolet, it was felt that a spectrophotometric method might provide rapid and accurate determinations for the pure telluric acid, and possibly eliminate the interference by other acids.

The method developed was dependent upon the shift of the tellurate's absorption spectrum in the ultraviolet region as the pH of the solution was increased. The sample of telluric acid was placed in a 100 ml. volumetric flask, 10 ml. of 6.6 M ammonium hydroxide was added and mixed thoroughly. After mixing, the flask was filled to the mark with distilled water and

placed in a constant temperature bath until temperature equilibrium was reached. Then, the absorbancy at 260 mμ with the cell compartment at the same temperature as the bath was measured. 0.66 M ammonium hydroxide was used as a blank.

Under the conditions of the determination, Beer's law was followed up to 156 mg. of telluric acid per 100 ml. of solution. For minimum relative error, the concentration range of telluric acid should be between 65 and 210 mg. per 100 ml.

Although temperature control is required when using this method, readily available thermostatic devices are suitable. Absorbancy increases only 0.002 per degree C., and with variations of plus or minus 0.5°, the error due to temperature fluctuation is within instrumental reading errors.

Limited amounts of other acids or sodium hydroxide may be present without interference. The permissible concentration of impurities may be extended by a study of the particular ions present. With nitrate ion, absorbancy readings at 260 mμ and 300 mμ would permit determination of both nitrate ion and telluric acid.