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 SUBJECT: High-Frequency Titration as Applied to the
 Determination of Thorium, Uranium, Sulfate,
 and Free Acid. Parts I Through V.

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FROM: Oscar Menis

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ABSTRACT

The technique of high-frequency titrimetry has been applied to the determination of thorium, uranium, sulfate, and free acid. In Part I of this report, the reproducibility of the method for the titration of standard solutions which contained 50 mg of thorium in the absence of interferences is established. The coefficient of variation of the method, under these conditions, was found to be less than one per cent. In Part II, the effect of uranium on the high-frequency titration of thorium, as well as the application of the method to actual samples, is discussed. Uranium in a ratio of 5 to 1 to thorium can be tolerated. When the method is applied to the analysis of representative samples, the coefficient of variation is one per cent.

Attempts to determine uranium by high-frequency titration with 8-quinolinol were unsuccessful. Tests on this titration and possible reasons for its failure are presented in Part III. The application of the high-frequency titration method to the determination of sulfate in solutions of uranyl sulfate is described in Part IV. The coefficient of variation, on titrating 40 to 70 mg of sulfate with barium chloride after the uranium is masked with citrate or fluoride, is two per cent.

Finally, in Part V, the high-frequency titration of free acid in solutions of uranyl sulfate is discussed. Uranium up to 350 mg, which was the highest level tested, does not interfere. The coefficient of variation of the method is about two per cent.

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PART I: HIGH-FREQUENCY TITRATION OF THORIUM

Reproducibility Studies

D. L. Manning

Purpose

To determine the reproducibility when thorium is titrated with ethylenediaminetetraacetic acid (EDTA) and the end point is detected by high-frequency oscillometry.

Apparatus and Reagents

Oscillator. A 190-Mc instrument was constructed by R. W. Stelzner of the Instrumentation Group. The parallel transmission lines are comprised of one-half inch copper tubing 7-inches long and spaced 1-1/2 inches apart. These lines run horizontally and mounted underneath them is a plastic well-type holder for the titration cell. The cell can be conveniently inserted and removed from this holder.

A 10-turn helipot having a resistance of 1000 ohms and a Coax connector were incorporated into the instrument. This permits the out-put of the oscillator to be recorded directly on a potentiometric strip recorder.

Housing. The oscillator is housed in a 6 in. x 5 in. x 9 in. metal cabinet. The cell is placed between the copper lines through a circular opening in the top of the housing.

Zero-Set Circuit. The same as described by Stelzner and Kelley.⁽¹⁾ Housed in a 6 in. x 5 in. x 9 in. metal cabinet.

Titration Cell. Glass, 1-1/2-inch diameter, 3-1/2-inches high.

Recorder. Brown potentiometric recorder, 50 Mv, 4-sec. response. Chart travel 2.00 inches per minute.

Buret. Motor-driven, syringe-type. This device delivers 0.906 ml of titrant per inch of chart travel.

Power Supply. Fluke Model 407 D.C. power supply.

Stirrer. Motor driven.

Thorium Nitrate. Approximately 25 mg of thorium per ml. Dissolve about 60 g of thorium nitrate $\text{Th}(\text{NO}_3)_2 \cdot \text{H}_2\text{O}$ in water and dilute to 1 liter. Standardize gravimetrically by taking an aliquot of the solution, precipitating the thorium as the oxalate and igniting to the oxide.

Ethylenediaminetetraacetic Acid (EDTA), 0.0712 M. Dissolve 13.28 g of Fisher reagent grade EDTA (M.W. 372.25) in water and dilute to 500 ml.

Procedure

Known amounts of thorium were transferred to the titration cell. The volume was adjusted to about 30 ml and the pH to 2.8 ± 0.2 as indicated by a pH meter. The cell was placed in the cell holder between the transmission lines. After inserting the buret tip and stirrer, the recorder was adjusted to a scale reading of approximately 80 by means of the control on the zero-set circuit. The 1000-ohm helipot was adjusted to 500 ohms. (This latter value is ascertained by a preliminary titration, and is chosen so that the oscillator response over the complete titration will not exceed full-scale deflection of the recorder.) The flow of titrant was started and the output of the oscillator during the course of the titration was recorded.

Part I: Table I

High-Frequency Titration of Thorium With EDTA

Reproducibility Studies

Conditions:	Oscillator, Mc	190
	Volume, ml	35
	pH (unbuffered)	2.8
	EDTA, M	0.0712

	Inches of Chart to End Point*	Thorium, mg	
		Present	Found
	3.36	50.3	50.4
	3.35		50.2
	3.36		50.4
	3.40		50.9
	3.35		50.2
	3.34		50.1
	<u>3.33</u>		<u>50.0</u>
Average	$\bar{X}$ 3.36		$\bar{X}$ 50.3
Coefficient of variation	V 0.6 per cent		V 1 per cent

* Chart travel, 2.00 ins./minute
Titrant delivery, 0.906 ml/inch

Discussion

From the data reported in Table I, it is seen that the coefficient of variation of the high-frequency titration method under favorable conditions, that is, when titrating only standard solutions of thorium, which contained 50 mg of thorium in 35 ml of solution, is of the order of 0.6 per cent. This compares favorably with visual titration methods for thorium in this concentration range. The governing factor in obtaining reproducible results is the location of the end point from an extrapolation of the two segments of the recorded titration graph. However, as illustrated in Figure 1, the end point which is obtained under favorable conditions is well-defined and an extrapolation of the graph is generally not necessary.

Conclusion

The high-frequency titration method is applicable to the determination of thorium under the conditions as outlined in this report with a coefficient of variation of the order of 0.6 per cent. This compares favorably with visual titrimetric methods. The main advantage of the high-frequency technique, however, is the applicability to solutions where indicators would not be satisfactory.

Future Work

To deduce the effect of uranium on the high-frequency titration of thorium with EDTA, and then to test the applicability of the method to the determination of thorium in mixed thorium oxide-uranium oxide slurries.

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Figure 1. High-Frequency Titration of Thorium with
Ethylenediaminetetraacetic Acid

PART II: EFFECT OF URANIUM ON THE HIGH-FREQUENCY
TITRATION OF THORIUM WITH ETHYLENEDIAMINETETRAACETIC ACID

Applicability to Samples

Purpose

To determine the effect of uranium on the high-frequency titration of thorium with ethylenediaminetetraacetic acid (EDTA), and then to apply the method to the analysis of thorium in solutions of thorium and uranium which comprise actual samples of dissolved thorium oxide-uranium oxide slurries.

Reagents and Apparatus

As previously outlined in Part I of this report.

Procedure

A 2-ml portion of the standard solution of thorium (21.7 mg/ml) was transferred to a 50-ml beaker. To the solution was added a known quantity of uranium as uranyl nitrate. The volume was adjusted to 30 ml and the pH to 3.0 ± 0.2 as indicated by a pH meter. The solution was then transferred to the titration cell, following which the cell was placed between the transmission lines of the oscillator. After inserting the buret tip and stirrer, the recorder was adjusted to a scale reading of approximately 80 by means of the control on the zero-set circuit. The 1000-ohm helipot was adjusted to 500 ohms. The chart of the recorder was started and, at a selected point on the chart, the buret motor was turned on and the response of the oscillator was recorded.

The above procedure was repeated for the analysis of samples after transferring an aliquot of the sample, usually ranging from 10 to 25 ml, to a 50-ml beaker. The aliquot taken contained from 30 to 70 mg of thorium.

Discussion

The effect of uranium on the high-frequency titration of thorium is illustrated from the data in Table II. From these results, it is evident that thorium can be titrated in the presence of up to 5 times as much uranium with an error of about one per cent. Larger quantities of uranium were not tested since the upper limit already tested far exceeds the uranium-thorium ratio that would be encountered in the analysis of samples.

Recorded titration graphs of thorium in the presence and absence of uranium are shown in Figure 2. Essentially the same shape of curve is obtained in both cases and no difficulty is encountered in extrapolating the two segments to a point of intersection.

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Figure 2. High-Frequency Titration of Thorium
With Ethylenediaminetetraacetic Acid

Part II: Table II

The Effect of Uranium on the High-Frequency Titration
of Thorium With Ethylenediaminetetraacetic Acid

Conditions: Oscillator, Mc 190
Volume, ml 35
pH 3
EDTA, M 0.0712

Uranium, mg	Inches of Chart to End Point**	Thorium		
		Present	Found	Per Cent
		mg		
0	2.85	43.4	42.8	99.0
12	2.84		42.6	98.4
24	2.87		43.0	99.1
36	2.81		42.1	97.0
100	2.84		42.6	98.3
200	2.87		43.0	99.1
				$\bar{X}$ 99
				Coefficient of Variation, V 1

* Added as $UO_2(NO_3)_2$

** Titrant delivery, 0.906 ml/in. of chart travel

The results from the analyses of solutions derived by dissolving representative thorium oxide-uranium oxide slurries are shown in Table III. The uranium content of the solutions is of the order of 1 mg/ml. From these data, it appears that the error of the method is about 1 per cent. Thus the high-frequency titrimetric procedure appears to be a relatively precise method for determining thorium under conditions where the only other alternatives would be lengthy gravimetric procedures, a potentiometric titration which makes use of the Hg-EDTA electrode, or less precise colorimetric methods. The time required for a high-frequency titration, including the time for aliquoting and pH adjustment, is about 7 minutes.

Part II: Table III

Coefficient of Variation of the High-Frequency Titration
of Thorium as Determined From the Difference
Between Duplicates. Application to Samples.

<u>Sample</u>	<u>Chart Travel, Inches</u>	<u>Thorium mg/ml</u>	<u>Average $\bar{X}$</u>	<u>Difference d</u>	<u>D Per Cent</u>	<u>D²</u>
1	3.80 3.73	11.2 11.4	11.3	0.1	0.9	0.81
2	2.42 2.40	7.25 7.20	7.23	0.05	0.43	0.18
3	1.75 1.87	2.63 2.80	2.71	0.08	3	9
4	2.00 1.90	3.00 2.85	2.93	0.07	2	4
5	1.23 1.18	1.84 1.77	1.80	0.04	2	4
6	2.05 2.00	3.08 3.00	3.04	0.04	1.3	1.8

$$N = 12$$

$$P = 6$$

$$S^2 = \frac{\sum D^2}{2P} = \frac{19.79}{12} = 1.65$$

$$\sum D^2 = 19.79$$

$$V = \sqrt{1.65} = 1.3 \text{ per cent coefficient of variation}$$

Conclusion

The high-frequency titrimetric method is applicable to the determination of milligram quantities of thorium in the presence of up to 5 times as much uranium. The thorium content of dissolved thorium oxide-uranium oxide slurries can be evaluated with a coefficient of variation of the order of 1 per cent.

Future Work

An investigation will also be made of the utility of the high-frequency titration with other systems such as the determination of free acid and sulfate and, possibly, uranium in solutions of uranyl sulfate.

PART III: DETERMINATION OF URANIUM WITH
8-HYDROXYQUINOLINE BY HIGH-FREQUENCY OSCILLOMETRY

Purpose

To investigate the possibility of determining uranium volumetrically by titrating it with 8-hydroxyquinoline and detecting the end point by high-frequency oscillometry.

Introduction

Rodden⁽²⁾ states that 8-hydroxyquinoline precipitates uranium(VI) quantitatively as a reddish-orange compound which is composed of one mole of uranium and 3 moles of reagent. The precipitation occurs over the pH range of 4 to 13 and is the basis of a precise gravimetric method for the determination of uranium.

In this experiment, attempts were made to utilize the same reaction in a volumetric procedure and to detect the end point by high-frequency oscillometry on titrating uranium with 8-hydroxyquinoline.

Reagents

8-Hydroxyquinoline, 0.27 M. Dissolve 10.00 grams on Matheson reagent-grade 8-quinolinol (M.W. 145.1) in 35 ml of glacial acetic acid. Dilute to 250 ml with water.

Uranyl Sulfate, 564 mg U/ml. This stock solution of uranyl sulfate was standardized by a potentiometric titration with ceric sulfate. Less concentrated solutions were prepared by appropriate dilutions of the stock solution.

Experimental and Observations

Solutions were prepared which contained from 30 to 70 mg of uranium in a volume of approximately 35 ml. The pH of the solutions varied from about 4.5 to 6.0. When the uranium in these solutions was titrated with the 8-hydroxyquinoline, the resulting high-frequency titration graph (Figure 3) consisted essentially of one continuous straight line instead of two segments which could be extrapolated to a point of intersection. The same results were obtained when the titration was conducted in aqueous-methanol and aqueous-ethanol solutions. The failure of the 190-Mc oscillator to register an end point for this reaction is not clearly understood; however, the following three factors probably influence the oscillator response and, consequently, their effect must be considered.

Figure 3. High-Frequency Titration of Uranium with 8-Hydroxyquinoline

1. The reaction rate of the uranium-8-quinolinol reaction may be too slow for equilibrium to be reached as the titration progresses.
2. The difference in the "loading characteristics" between the reactants and products may be too small to produce any change in the response of the oscillator.
3. The loading of the oscillator by the acetic acid in the 8-quinolinol titrant, as the latter is added to the titration cell, may tend to mask or distort the titration graph to such an extent that the two segments of different slopes can not be noted.

Conclusion

At the present stage of development in the HR laboratory, the high-frequency titration technique is not satisfactory for the volumetric determination of uranium with 8-quinolinol. Possible factors involved are presented; however, further experiments will have to be performed before definite conclusions can be reached as to whether, in a precipitation titration, the end point can be detected by means of this apparatus.

Further Work

Further study of this type of titration is to be temporarily postponed and the possibility of determining both uranium and free acid in solutions of uranyl sulfate by high-frequency titrations with NaOH is to be investigated.

PART IV: HIGH-FREQUENCY TITRATION OF SULFATE

Application to Solutions of Uranyl Sulfate

Purpose

To apply high-frequency titrimetry to the determination of sulfate, utilizing BaCl_2 as the titrant, with emphasis on the application of this method to the estimation of sulfate in solutions of uranyl sulfate.

Reagents

Barium Chloride, 0.251 M. Dissolve 10.43 g of Baker's reagent-grade BaCl_2 in 200 ml of water.

Sodium Sulfate, 5.4 mg $\text{SO}_4^{=}$ /ml. Dissolve 2.00 g of reagent-grade Na_2SO_4 in 250 ml of water.

Sodium Citrate, 2 M. Dissolve 60 g of $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7$ in 100 ml of water. This is a saturated solution.

Sodium Fluoride, 1 M. Dissolve 10 g of NaF in 250 ml of water.

Uranyl Sulfate Solution. Same as described in Part III of this report.

Procedure

Solutions were prepared which contained milligram quantities of sulfate either as Na_2SO_4 or UO_2SO_4 in a titration volume of 30 ml. In some cases, enough sodium citrate or sodium fluoride was added to produce a solution of a specific molarity with respect to these substances when diluted to 30 ml. The solutions were then titrated with standard barium chloride. Representative high-frequency titration graphs are shown in Figure 4 and the data are presented in Table IV.

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Figure 4. High-Frequency Titration of Sulfate with Barium Chloride

Part IV: Table IV

High-Frequency Titration of Sulfate With Barium Chloride

Conditions: Barium chloride, M 0.251
 Delivery, ml/in. .906
 Volume, ml 30

Substance	Medium for Titration	Chart to End Point, Inches	Sulfate		Per Cent	
			Present	Found		
			mg			
Na ₂ SO ₄	Aqueous	3.8	82.3	82.9	100	
		2.5	54	54.6	100	
		2.45	54	53.5	.99	
				$\bar{X}$	100	
Coefficient of Variation, V, %					1	
UO ₂ SO ₄	Aqueous	No end point	68.4	-	-	
		3.2	68.4	69.8	102	
		2.10	45.6	45.8	101	
	0.03 M Sodium Citrate					
		2.16	45.6	47.1	103	
	0.03 M NaF	2.12	45.6	46.2	102	
		3.17	68.4	69.2	101	
		2.10	45.6	45.8	100	
		2.10	45.6	45.8	100	
		2.45	54	53.5	.99	
		2.45	54	53.5	.99	
					$\bar{X}$	101
Coefficient of Variation, V, %					2	

Discussion

The data for the high-frequency titration of sulfate as a simple salt such as Na₂SO₄ and also as uranyl sulfate are presented in Table IV. No difficulty was encountered in titrating aqueous solutions of Na₂SO₄. Well-defined titration curves were recorded as illustrated by the top graph in Figure 4. The coefficient of variation from a limited amount of data is of the order of 1 per cent.

When an aqueous solution of uranyl sulfate was titrated with barium chloride, the resulting high-frequency titration curve (middle graph, Figure 4) consisted essentially of one continuous straight line instead of the customary two segments of different slopes. The same type of curve was obtained when attempts were made to titrate sulfate in the presence of uranyl nitrate. In this situation where the reaction is

the change in the response of the oscillator at the start of the titration, is undoubtedly due to the difference between the "loading characteristics" of UO_2SO_4 and UO_2Cl_2 . But beyond the end point where excess BaCl_2 is present, its loading effect appears to be the same as that of UO_2Cl_2 ; consequently, the response of the oscillator is constant and the graph consists of just one continuous straight line.

In the presence of a complexing agent such as sodium citrate or sodium fluoride, it is possible to titrate the sulfate which is present in a UO_2SO_4 solution. This is because the uranium is masked prior to the titration and remains inactivated throughout the precipitation reaction, so, in essence, the sulfate undergoing reaction is present as uncomplexed Na_2SO_4 . The resulting titration graphs (curves A and B, Figure 4) resemble the curve obtained for sodium sulfate. Of the two complexing agents tried, sodium fluoride is preferable to sodium citrate as a masking agent for uranium. The resulting titration curves in the presence of the former salt are better defined and the concentration of fluoride in the solution being titrated is less critical. Whereas 50 mg of sulfate as UO_2SO_4 can be titrated in 0.03 to 0.1 M solutions of NaF , the concentration of sodium citrate must be controlled to approximately 0.03 to 0.04 M for satisfactory results. An advantage of the high-frequency titration procedure over conventional volumetric methods for the determination of sulfate⁽³⁾ is that it is not necessary to remove the uranium prior to the titration. The addition of fluoride or citrate to mask the uranium prior to titrating the sulfate is the only requirement.

Conclusion

The high-frequency titration method is applicable to the titration of milligram quantities of sulfate as UO_2SO_4 with BaCl_2 . In order to achieve satisfactory results, however, it is essential to mask the uranium by the addition of either sodium citrate or sodium fluoride prior to the titration. Sodium fluoride is the preferred masking agent. The coefficient of variation is of the order of 2 per cent.

PART V: HIGH-FREQUENCY TITRATION OF FREE ACID AND
URANIUM IN SOLUTIONS OF URANYL SULFATE WITH SODIUM HYDROXIDE

Purpose

To study the feasibility of titrating both free acid and uranium in uranyl sulfate with sodium hydroxide and detecting the end points by high-frequency oscillometry.

Introduction

The method currently in use for the determination of free acid involves an acidimetric titration utilizing the Dow recordomatic titrator. The titration curve exhibits two inflection points. The first, which occurs at a pH of about 3.5, represents the neutralization of the free acid, whereas the second, which occurs at a pH of approximately 9, indicates the completion of the hydrolysis of the uranium. Due to the buffering action of the uranium, however, the first end point, in the case of high ratios of uranium to free acid, is poorly defined, and, as a consequence, the result for free acid may be significantly in error. For this reason, it is of interest to apply high-frequency titrimetry to this determination and to establish if it has any advantages over the potentiometric titration procedure.

Reagents

Sodium Hydroxide, 0.100 N. Dilute 10 ml of Baker's standard solution of NaOH (1.000 N) to 100 ml with water.

Uranyl Sulfate Solution. Same as described in Part III of this report.

Sulfuric Acid, 0.07 N. Dilute 1 ml of concentrated H_2SO_4 to 500 ml with water. Standardize by titrating a portion with the standard NaOH to a potentiometric end point, utilizing a glass and saturated calomel electrode combination.

Discussion

From the data recorded in Table V, it is demonstrated that the high-frequency titration method is applicable to simple acid-base titrations and also to the measurement of free acid in the presence of uranyl sulfate. Relatively large amounts of uranium can be present without introducing a significant error in the free acid measurement. The coefficient of variation is of the order of 2 per cent which is comparable to the potentiometric method.

Part V: Table V

Effect of Uranium on the
High-Frequency Titration of Free Acid

Conditions: NaOH, N 0.100
Delivery, ml/in. .906
Titration volume, ml 35

Uranium, mg	Chart to End Point, Inches	Free Acid		Per Cent
		Present	Found	
		Meq		
None	2.30	0.21	0.209	99
	2.32		.21	100
	2.10	0.187	.190	101
	2.10		.190	<u>101</u>
				$\bar{X}$ 100
		Coefficient of Variation, V, %		1
14	2.00	0.187	0.181	97
	2.05		.186	100
35	2.00		.181	97
	2.10		.190	101
165	2.15		.195	104
	2.10		.190	101
	2.15		.195	104
	2.10		.190	101
350	2.10		.190	101
	2.10		.190	<u>101</u>
				$\bar{X}$ 101
		Coefficient of Variation, V, %		2

The values for the determination of uranium from the volume of standard base required for hydrolysis are shown in Table VI. Since the preliminary results indicated that the titration serves only as an approximation of the uranium present, further tests were suspended. Rodden⁽⁴⁾ has also pointed out the feasibility of estimating uranium by a sodium hydroxide titration, but he further emphasized the necessity of utilizing empirical factors inasmuch as the reaction is not stoichiometric.

Figure 5. High-Frequency Titration of Free Acid and Uranium with Sodium Hydroxide

Part V: Table VI

High-Frequency Titration of Uranium

Conditions: NaOH, N 0.100
 Delivery, ml/in. .906
 Titration volume, ml 35

<u>Test</u>	<u>End Point, Inches*</u>	<u>Uranium</u>		<u>Per Cent</u>
		<u>Present</u>	<u>Found</u>	
		mg		
1	2.00	22	21	96
2	2.40	22	25	113
3	5.58	56	59	105

* Corrected for the presence of 0.1 meq of free acid

A representative high-frequency titration curve is shown in Figure 5. No difficulty was encountered in any of the tests in extrapolating the segments of the graph having different slopes to points of intersection.

Conclusion

The high-frequency titration procedure is applicable to the determination of free acid in solutions of uranyl sulfate. Uranium up to 350 mg does not interfere with the titration of free acid in amounts of the order of 0.2 milliequivalents. Although the same titration can be used to approximate the uranium content of samples, it is recommended that alternate methods be used for a precise determination of the uranium in view of the non-stoichiometric nature of the uranium-sodium hydroxide reaction.

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