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DETERMINATION OF COMPLEXABLE METALS
BY CHELOMETRIC TITRATION

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ABSTRACT

A rapid, simple method for the determination of total complexable metal ions (Fe^{3+} , Al^{3+} , Mn^{2+} , Ni^{2+} , Co^{2+} , Pb^{2+} , and Rare Earths) in feed to the Hanford strontium recovery process has been developed. The method consists of complexing the metal ions with EDTA and titrating the excess with copper (II) ion. Pyrocatechol violet is used as a colorimetric end point indicator.

Samples of actual process feed were analyzed and the results compared very favorably with results by atomic absorption. Use of a synthetic feed standard showed an average accuracy of 99.7% and a relative precision of $\pm 1.9\%$ (95% C.L.).

Interference studies run on the method show that oxalate and phosphate interfere above mole ratios of 0.1 and 0.3, respectively (anion : total complexable cations). Of the complexable metal ions commonly encountered in this feed (specifically excluding alkali and alkaline-earth metals), the only complexable ion not determinable by this method is Cr(III).

INTRODUCTION

Nuclear fuel reprocessing plants, such as the Purex Plant on the Hanford reservation, generated large volumes of liquid waste containing very high levels of fission products. As part of the current Hanford Waste Management program,^(1,2) the high activity of this liquid waste, some of it self-boiling, must be reduced or removed to reduce the heat load of the storage tanks. Most of the fission products present (e.g., $^{95}\text{ZrNb}$, ^{103}Ru , $^{106}\text{RuRh}$, $^{144}\text{CePr}$, etc.) are relatively short-lived (one year or less), so the concentrations of these radionuclides are reduced by simply allowing the waste to decay for a few years. However, the quantities of ^{90}Sr and ^{137}Cs are not significantly reduced in this time period, owing to their relatively long half-lives (28.8 and 30.2 years, respectively). Consequently, the bulk of these isotopes is removed at the Waste Fractionization Processing Plant (B-Plant).

Most of the ^{90}Sr is removed by solvent extraction. The feed, which is currently PAS (Purex Acidified Sludge) is complexed with a mixture of ethylenediaminetetraacetic acid (EDTA), hydroxyethylethylenediaminetriacetic acid (HEDTA), and sodium citrate, and the pH is adjusted to 5.0 with sodium carbonate. This treated feed is then contacted with an organic solution of bis(2-ethylhexyl)phosphoric acid (HDEHP) and tributyl phosphate (TBP) in normal paraffin hydrocarbon (NPH).*

The total content of metal ions complexable at pH 5 must be known in order that the proper amount of complexant may be added. Insufficient complexant will cause some of these metals to extract, loading the solvent and suppressing extraction of strontium. Too much complexant is wasteful and can lead to decreased extraction of strontium through complex formation.⁽²⁾ Therefore, a method is needed that will determine the concentrations of all the metal ions normally complexable at pH 5 with the complexants used in the process. Ideally, the method would be rapid, simple, and determine all the metal ions simultaneously.

There have been a number of methods used in the past for determining complexable cations. Probably the simplest was titration with standard NaOH solution, since the cations of interest are hydrolyzed in aqueous solution. Two titrations were done, with an excess of oxalate ion added to one titration. The difference between the results of these two titrations was the acid due to the hydrolysis of the cations. The chief disadvantage of this method is that not all the cations are in the same valence state, so each species consumes relatively different amounts of base. Furthermore, not all the ions complexable with EDTA are complexable with oxalate.

*A mixture of straight-chain alkanes, usually from 9 to 14 carbons in length.

INTRODUCTION (Contd)

The other method which was used for a number of years was atomic absorption, in which the major complexable metals (Al, Fe, Mn) were determined. This method usually gave reliable results; however, some were often erratic, presumably because of matrix effects or poor technique, but otherwise unexplainable. Additionally, the turnaround time was very slow, sometimes as long as five shifts (~40 hours). Moreover, because of the high radioactivity of these samples (usually upwards of 1 Curie/liter ^{90}Sr), personnel exposure was a problem.

Since EDTA is one of the complexants used in the process to complex the undesirable metals, a method was sought that utilized EDTA. Kritz⁽³⁾ had used a back-titration method for determining Al and Fe in glass, and I found that this method could be expanded to include Mn, Co, Pb, and a number of other metals, as well as Al and Fe.

SUMMARY AND CONCLUSIONS

A method has been developed for the determination of total complexable cations in PAS feed solutions. An accuracy of 99.7 percent was observed on synthetic standard feed solutions, and the precision was $\pm 1.9\%$ at the 95% confidence level.

Results on actual process samples appear to agree quite well with those by atomic absorption (AA), and these results generally appear less erratic than the AA results.

The method is rapid and simple and appears well suited to the analysis of these feed solutions, as it determines all the complexable metal ions except Cr(III) simultaneously. In the present feed, Cr(III) is usually less than 1% of the total complexable cations and can hence be neglected without causing a significant problem or bias in the determination.

By and large, it is felt that this titrimetric method is capable of giving more reliable results than AA on strontium recovery feed samples to B-Plant. Additionally, turnaround time and radiation exposure are greatly reduced.

EXPERIMENTAL

REAGENTS

1. Ethylenediaminetetraacetic Acid (EDTA), $0.1000 \pm 0.01 \text{ M}$

Dissolve 37.2 grams of the disodium salt dihydrate in deionized water and dilute to one liter. Standardize to 0.0001 M by an accepted method (e.g., Calcium with Eriochrome Black T). Store in a polyethylene bottle.

2. Pyridine, reagent grade

3. Pyrocatechol Violet, 0.1%

Dissolve 0.1 gram of pyrocatechol violet in deionized water and dilute to 100 ml.

4. Standard Copper Solution, $0.1000 \pm 0.0100 \text{ M}$

Weigh out 6.35 grams of copper (minimum purity 99.9%) to 0.0001 gram. Dissolve in a minimum amount of 6 M nitric acid. When dissolution is complete, cool and dilute to one liter with deionized water. This is a primary standard and is calculated as follows:

$$\text{Cu}^{2+} \text{ concentration (M)} = \frac{\text{weight of Cu (grams)}}{63.54}$$

EQUIPMENT

1. Magnetic stirrer and glass-covered stir bars.
2. pH meter.
3. Manostat or ultramicroburet - calibrated to read $2 \mu\text{l}$ or less.

GENERAL PROCEDURE

Pipet sample, containing .05 to .08 millimole of complexable cations, into about 10 ml of deionized water in a glass vial. If necessary, adjust the pH to between 1 and 3 with 0.1 M NaOH or HNO_3 . While stirring, add exactly 1000 μl of standardized 0.10 M EDTA, 1 ml of pyridine, and 2 drops indicator. Titrate with standard 0.10 M copper(II) nitrate solution until the color changes from yellow to a dark bluish-green. The color change is complete over about a $5 \mu\text{l}$ addition of titrant when the stated concentrations of all reagents are used.

RESULTS AND DISCUSSION

Determination of metals by chelometric titration with EDTA is a relatively old method, often subject to numerous interferences when one particular cation is desired in the presence of others. However, the problem in this case was to determine all the complexable metal ions in solution at once. The method used is a conventional back-titration type of determination with a colorimetric end point. The color of the indicator used, pyrocatechol violet, is pH dependent and is normally yellow at pH 5-6. It forms a bright blue complex with Cu(II), as well as a number of other metals, above pH 5⁽⁴⁾. The indicator complexes of all these metals are generally weaker than the corresponding EDTA complexes, so the metal indicator complex will form in solution with only those cations not complexed with EDTA.

After sample addition and the initial pH adjustment, a known excess of standard EDTA is added, which complexes the metal ions according to the general reaction:

The pH is then raised to about 5-6 by addition of pyridine. The exact reason that Kritz chose pyridine is not known, but a possible explanation could be that it is completely miscible with water and is a very weak base ($K_b = 1.7 \times 10^{-9}$) and could therefore provide good buffering action at a near-neutral pH.

After the pH adjustment, indicator is added and the remaining free EDTA is titrated with copper (II) ion:

When all the excess free EDTA is complexed, additional copper added forms the deep blue indicator complex, and this color change is taken as the end point.

INTERFERENCES

As this was a specialized method developed for a limited variety of cations, only the ions likely to be encountered in B Plant feed solutions were studied. These included nine cations (Table I) and five anions (Table II).

In the cation studies, the indicated amounts of each cation (as the chloride or nitrate) were added, and the solution was analyzed according to the detailed procedure given in Appendix A.

TABLE I

DETERMINATION OF VARIOUS CATIONS BY CHELOMETRIC TITRATION (APPENDIX A)

Run No.	No. of Trials	Micromoles Added:				Total μ Moles:		% Rec.
		Al	Fe	Mn	Other	Added	Found	
1	3	25	25	0	0	50	49.9	99.9
2	2	25	25	10	0	60	59.8	99.0
3	2	25	25	0	Mg-10	60	50.2	84.7
4	2	20	20	10	0	50	49.8	99.6
5	2	20	20	10	Cr-10	60	49.7	82.9 ^a
6	2	20	20	10	Co-10	60	59.8	99.7
7	2	20	20	10	Ni-10	60	59.2	98.7
8	2	20	20	10	Pb-10	60	59.8	99.7
9	2	20	20	10	RE ^b -6.25	56.25	55.9	99.4

^aPrecipitate formed upon addition of pyridine.^bRE - Mixture of four lanthanides (Sm, Eu, Gd, Dy).UNCLASSIFIED
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For the anion interference studies, a 100 μ l of aliquot of Synthetic B-Plant Feed Standard (Table III) was used and the indicated amount of anion added. Anions used were solutions of either the sodium or potassium salts. As in the cation studies, the procedure given in Appendix A was used.

TABLE II

ANION INTERFERENCE STUDIES

Anion ^(a)	μ Moles Added	Mole Ratio ^(b)	% Recovery
F ⁻	1.0	0.023	97.5
	5.0	0.11	97.8
	10.0	0.23	98.2
	50.0	1.10	96.2
Cl ⁻	5.0	0.11	99.8
	10.0	0.23	99.4
	59.9	1.10	99.2
C ₂ O ₄ ²⁻	1.0	0.023	99.6 ^(c)
	5.0	0.11	94.5
	10.0	0.23	85.8
SO ₄ ²⁻	1.1	0.025	100.1
	5.1	0.125	99.2
	11.0	0.25	98.2
	55.0	1.25	98.9
PO ₄ ³⁻	1.5	0.034	99.4
	7.5	0.170	99.6
	15.0	0.340	100.8
	30.0	0.68	82.3
	37.5	0.85	76.6 ^(d)
	75.0	1.70	65.7 ^(e)

(a) Anion solutions were used as follows:

F ⁻	- 0.1 M NaF
Cl ⁻	- 0.1 M KCl
C ₂ O ₄ ²⁻	- 0.1 M K ₂ C ₂ O ₄
SO ₄ ²⁻	- 0.11 M Na ₂ SO ₄
PO ₄ ³⁻	- 0.15 M Na ₂ HPO ₄

(b) Mole Ratio = $\frac{\text{Moles Anion}}{\text{Moles Total Complexable Cations}}$

(c) End point was not as sharp with oxalate.

(d) Sample turned slightly turbid upon addition of EDTA.

(e) Sample turned very turbid upon addition of anion.

TABLE III

SYNTHETIC FEED COMPOSITION

Cations: Na^+	1.76 <u>M</u>
H^+	1.27
* Fe^{3+}	0.2000 <u>M</u>
* Al^{3+}	0.1500
* Mn^{2+}	0.0604
Ca^{2+}	0.04
* Ni^{2+}	0.02
K^+	0.005
Mg^{2+}	0.004
* Cr^{3+}	0.003
* Co^{2+}	0.003
* Pb^{2+}	0.003
* La^{3+}	0.003
Anions: Cl^-	0.07
NO_3^-	Balance ($\sim 4:3$ <u>M</u>)

*Calculated Concentration of Total Complexable Cations (TCC) = 0.4424 M

From Table I, you see that the only cation considered "complexable" which is not determinable by this method is Cr(III). Other cations not included in this study may, of course, also fail to be complexed. (Alkali and alkaline-earth metal ions are not normally complexed by EDTA at pH < 9-10.)

According to Welcher ⁽⁵⁾, Cr(III) forms a complex with EDTA only after boiling the solution for 15 minutes, so the Cr(III) complex would not form under the conditions used in this method.

Of the anions studied, (Table II), you can see that phosphate (PO_4^{3-}) and oxalate ($\text{C}_2\text{O}_4^{2-}$) are the only anions which interfere when present in significant amounts. I infer that nitrate does not interfere, as it is the major anion present and so the anion:TCC ratio is very large ($\sim 10:1$).

Since determination of cations by this technique is based on complexation with EDTA, cations already complexed by EDTA or some other strong chelating agent in the original sample will not be determined.

ACCURACY AND PRECISION

Repeated runs (Table IV) with the synthetic feed standard show an average accuracy of 99.7% and a relative precision of $\pm 1.9\%$ at the 95% confidence level.

TABLE IVDETERMINATION OF ACCURACY AND PRECISION CAPABILITIES^a

<u>Trial No.</u>	<u>Result (M)</u>	<u>% Recovery</u>
1	0.449	101.5
2	0.441	99.6
3	0.442	99.8
4	0.429	97.1
5	0.440	99.6
6	0.444	100.3
7	0.438	99.0
8	0.446	100.8
9	0.440	99.4

^aStandard Value = 0.4424 M

COMPARISON WITH ATOMIC ABSORPTION

Prior to development of this titrimetric method, atomic absorption (AA) was used for analysis of the three primary metallic constituents in the PAS B-Plant feed (Al, Fe, and Mn). However, AA sometimes gave erratic, unreliable results, possibly due to poor technique, matrix problems, or excessive spike corrections.

A series of actual feed samples from the routine sample point, tank 33-1, were run by both methods and the results compared (Table V).

TABLE V

ANALYTICAL RESULTS OF TK-33-1 SAMPLES

<u>Sample Number</u>	<u>Number of Trials</u>	<u>Results: (M)</u> <u>This Method</u>	<u>AA</u>	<u>Percent Diff.</u> <u>(This Method Vs. AA)</u>
1	3	0.516	0.510	+1.1%
2	2	0.571	0.553	+3.2
3	2	0.571	0.564	+1.2
4	2	0.668	0.665	+0.5
5	2	0.673	0.703	-4.3
6	2	0.662	0.698	-5.2

The titrimetric results compare quite favorably with the AA results, and in fact should be biased slightly higher, since there are other complexable cations in the feed besides Al^{3+} , Fe^{3+} , and Mn^{2+} .

As a further check, 25 samples were analyzed by shift personnel over a period of about one month using both methods, and the results were again compared (Table VI and Figure 1).

TABLE VI
RESULTS OF LONG-TERM SAMPLE STUDY

Sample No.	Results ^a		Percent Diff. (TCC Vs. AA)
	TCC	AA	
1	.415	.395	+5.1
2	.474	.518	-8.5
3	.534	.507	+5.3
4	.579	.634	-8.7
5	.496	.449	+10.5
6	.611 ^b	.551	+10.9
7	I.S. ^b	.634	----
8	.648	.563	+15.1
9	.657	.703	-6.5
10 ^c	.680	.663	+2.6
11	.679	.711	-4.5
12	.681	.666	+2.2
13	.676	.586	+15.4
14	.622	.645 ^d	-3.6
15	.680	.424 ^d	+60
16	.673	.201 ^d	+235
17	.638	.698	-8.6
18	.621	.494 ^d	+25.7
19	.667	1.072 ^d	-37.8
20	.672	.638	+5.3
21	.551	.444	+24.1
22	.614	.537	+14.3
23	.568	.676	-16.0
24	.632 ^e	.216	+22.0
25	N.D. ^e	.608	----

^aAverage Standard Recovery (TCC) = $101.2 \pm 6.0\%$ on 23 determinations (95% C.L.)

^bI.S. = Insufficient Sample. First titration of this sample turned green and a precipitate formed upon addition of indicator. There wasn't enough sample to rerun.

^cSamples 10, 11, and 12 are from same batch of feed, taken at different times.

^dQuestionable AA Results: 15 - Low Fe and Mn

16 - Low Al

19 - High Al (43% spike recovery)

^eN.S. = Not Determinable. This sample contained ~25-50% solids, making the dilution dark enough to mask any color change.

As seen from Figure 1, the results by this method (TCC) follow a much smoother variation and appear less erratic than some of the AA results, noting especially samples 16 and 19.

FIGURE 1

RESULTS OF LONG-TERM SAMPLE STUDY
CHELOMETRIC TITRATION (TCC) VS. ATOMIC ABSORPTION (AA)

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APPENDIX A

PROCEDURE FOR DETERMINATION OF TOTAL COMPLEXABLE
CATIONS BY CHELOMETRIC TITRATION

Procedure	Comment
1. Pipet the required sample aliquot into a prerinsed 15 ml vial containing ~10 ml of deionized water and a glass-covered stir bar.	1a. Sample size should contain ~0.05 to 0.08 millimole of complexable metal ions.
	1b. Radiation hazard exists with hot samples. Check radiation readings and use appropriate shielding as necessary.
2. Check pH. If necessary, add 0.1 M NaOH or HNO ₃ dropwise until the pH is between 1 and 3.	2a. This step may be omitted on routine B-Plant feed samples known to be acid (0.5 - 1.5 M H ⁺). Caustic samples should not be run without preliminary acidification.
3. While stirring, pipet exactly 1000 μ l of standard 0.1000 M EDTA solution into the vial. Record exact concentration used.	3a. Solution will turn light yellow if iron is present.
4. Continue stirring and add ~1 ml of pyridine.	4a. If a precipitate forms, either there was too much sample or it contained a large amount of chromium (III) or some other noncomplexable cation. Check the estimated metal content of the sample and rerun with a smaller sample size.
	4b. The pH should now be between 6 and 7.
5. Add 2 drops of 0.1% pyrocatechol violet indicator.	5a. Solution should now be an intense yellow.

APPENDIX A (Contd)

Procedure	Comment
6. Before titrating sample, expel to waste a few drops of titrant (Standard 0.1000 M copper) from the delivery buret and reset the manostat to 0. Rinse tip of buret with deionized water.	6a. This is to take up any slack in the buret piston drive and to discard the titrant from the tip of the buret which may be contaminated or concentrated. 6b. Be sure buret is filled with titrant.
7. Lower tip of delivery buret carefully into the solution.	7a. Use care not to break the tip.
8. Titrate with the standard copper solution until a definite blue-green color persists for more than 5 seconds.	8a. Flashes of blue will occur as titrant is added and will be more persistent as the end point is approached. 8b. Solutions containing a high percentage of iron will slowly turn from yellow to green as the titration progresses. This is normal and should not be taken as the end point.

APPENDIX B

PREPARATION OF THE SYNTHETIC 33-1 STANDARD

Procedure	Comment
1. Weigh 5.4 g of Al wire to 0.0001 g and dissolve in 100 ml of 5 <u>M</u> NaOH.	1a. All metals used should be at least 99.5% pure.
2. Weigh 8.7 g of reagent grade MnO ₂ to 0.0001 g and place in glass beaker. Add 30% H ₂ O ₂ slowly dropwise until mostly dissolved, then add a minimum of 12 <u>M</u> HCl to complete the dissolution. Solution must be boiled to expel the oxygen generated. Save this solution for Step 6.	
3. When Al is completely dissolved in the caustic, add a teflon stir bar. Stir gently and slowly add 170 ml of concentrated (15.6 <u>M</u>) HNO ₃ , about 10 ml at a time.	3a. A thick precipitate of Al(OH) ₃ will form, but should redissolve after all the acid is added. 3b. Solution should now be 4-5 <u>M</u> H ⁺ .
4. Weigh 1.18 g of Ni wire and 11.7 g of Fe wire to 0.0001 g and add to the solution in Step 3, heating if necessary.	
5. Add the following reagents to a 1000 ml volumetric flask: 110.0 g NaNO ₃ 9.4 g Ca(NO ₃) ₂ ·4H ₂ O 1.0 g Mg(NO ₃) ₂ ·6H ₂ O 0.6 g Sr(NO ₃) ₂ 0.5 g KNO ₃	5a. These reagents may be weighed to 0.01 g, as the concentrations these ions do not have to be exact.
6. Transfer the solution of Mn from Step 2 quantitatively to the flask. When the metals are completely dissolved, transfer the solution from Step 4, also quantitatively. Swirl gently to dissolve the nitrate salts.	

APPENDIX B (Contd)

Procedure	Comment
7. Pipet 3.000 ml of each of the following stock standards to the solution in the flask:	7a. The stock standards may be prepared by weighing out sufficient nitrate salt to make 100 ml of solution. The amounts are (all weighings to .0001 g):
1.00 M Co^{2+}	29.1 g $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$
1.00 M Cr^{3+}	40.0 g $\text{Cr}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$
1.00 M La^{3+}	43.3 g $\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$
1.00 M Pb^{2+}	33.1 g $\text{Pb}(\text{NO}_3)_2$
	7b. It is understood that the above nitrate salts are normally not acceptable as primary standards. In this case, however, the final concentration of these metals is under 3 percent of the total cation concentration. Relatively large errors in these ions (~10%) can then be tolerated without a significant effect on the overall complexable cation content.
8. Dilute to the mark with deionized water. Add a stir bar and stir thoroughly to mix the solution.	
9. Calculate the concentration of each cation in the solution. Express the accurately determined concentrations (Al, Fe, Mn, Ni, Co, Cr, La and Pb) to four (4) places beyond the decimal point.	
10. Calculate the total complexable cation content (TCC) as the sum of the eight cations listed in the above step. An example follows:	
Na^+ 1.8 M $*\text{Mn}^{2+}$ 0.1072 M Mg^{2+} 0.004 M $*\text{Pb}^{3+}$ 0.0028 M H^+ 0.76 Ca^{2+} 0.04 $*\text{Co}^{2+}$ 0.0030 Sr^{2+} 0.003 $*\text{Al}^{3+}$ 0.2137 $*\text{Ni}^{2+}$ 0.0231 $*\text{Cr}^{3+}$ 0.0029 $*\text{Fe}^{3+}$ 0.2001 K^+ 0.005 $*\text{La}^{3+}$ 0.0030 $*\text{TCC} = 0.5558 \text{ M}$	

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