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PROGRESS REPORT NO. 14

CONTROL INDICES AND ANALYTICAL PROCEDURES

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SUMMARY

The procedures summarized in Progress Report No. 7, September, 1953, for the amperometric titration of zirconium, photometric determination of aluminum, and indirect polarographic determination of fluoride have been applied to the two specific solutions described in that report in the presence of small amounts of tin (II) or of uranium (IV). The presence of the latter elements had no effect.

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SUMMARY OF RESULTS

ANALYTICAL PROCEDURE STUDIES. I. DETERMINATION OF ANALYTICAL PROCEDURES FOR ZIRCONIUM AND FLUORIDE IN ZIRCONIUM FLUORIDE - HYDROFLUORIC ACID SOLUTIONS.

II. DETERMINATION OF ANALYTICAL PROCEDURES

FOR ZIRCONIUM, ALUMINUM, AND FLUORIDE IN A MIXED SALT SOLUTION

A. Objective and Implementation

Objective. Suitable methods have been developed for (1) the amperometric titration of zirconium (Progress Report No. 4, September, 1953), (2) the indirect polarographic determination of fluoride (Progress Report No. 6, September, 1953), and (3) the spectrophotometric determination of aluminum (Progress Report No. 5, September, 1953). The application of these procedures to the analysis of two specific solutions has been described in Progress Report No. 7, September, 1953.

It was requested that these methods be investigated for their applicability to the solutions discussed in Progress Report No. 7 when these solutions contained small amounts of tin or uranium.

B. Experimental Procedure

Chemicals and Reagents. These were essentially the same as described in Progress Reports Nos. 4, 5, and 6.

Solutions. With the exceptions subsequently indicated, the test solutions were the same as those described on pages 2 and 3 of Progress Reports Nos. 4, 5, and 6.

Stannous chloride: Two solutions were prepared by dissolving reagent-grade stannous chloride in 1:10 (by volume) sulfuric acid. The tin concentrations were 0.075 g/l and 0.225 g/l.

Uranium nitrate: Two solutions were prepared by dissolving reagent-grade uranium (IV) nitrate in 1:10 (by volume) sulfuric acid to give uranium concentrations of 0.075 g/l and 0.225 g/l.

Solution 1: This solution was described on page 2 of Progress Report No. 4.

Solution 1A: The zirconium and fluoride concentrations were the same as in Solution 1. A tin concentration of 2.25 g/l was introduced during the dilutions.

Solution 1B: This solution contained the same zirconium and fluoride concentrations as Solution 1. A uranium concentration of 2.25 g/l was introduced during the dilutions.

Solution 2: This solution was described on page 3 of Progress Report No. 4.

Solution 2A: The zirconium and fluoride were present in the same concentrations as in Solution 2. A tin concentration of 0.75 g/l was introduced.

Solution 2B: The zirconium and fluoride concentrations were the same as in Solution 2. A uranium concentration of 0.75 g/l was introduced.

Apparatus. The apparatus was essentially the same as that described on pages 3 of Progress Reports Nos. 4, 5, and 6. A thermostated ($25.0 \pm 0.1^\circ\text{C}$) H-cell was used for some of the polarographic fluoride determinations.

Analytical Procedure. The procedures were essentially the same as those given in Progress Report No. 7 for Solutions 1 and 2. Solutions 1A and 1B were analyzed exactly the same as Solution 1, while Solutions 2A and 2B were analyzed exactly the same as Solution 2.

C. Data Obtained and Interpretation

The results obtained for the determination of zirconium are given in Tables I and II. Inspection of these results and comparison with those given in Progress Report No. 4, September, 1953, indicate that the added tin and uranium have no appreciable effect on the accuracy of the zirconium

determination. In another series of similar samples which were analyzed in triplicate, the average results for zirconium in the 1A and 1B samples differed from that for Solution 1 by -1.1 and -0.6 relative percent, respectively, and those for the 2A and 2B samples differed from that for Solution 2 by -0.2 and -0.8 relative percent, respectively.

Comparative results for fluoride and aluminum also seem to indicate no effect due to tin and uranium at the levels used. The fluoride determination, however, was shown to be in need of further development.

TABLE I

DETERMINATION OF ZIRCONIUM
IN THE PRESENCE OF TIN AND URANIUM

Zr Content, g/l	Zr Found		
	Soln. 1, g/l	Soln. 1A, g/l	Soln. 1B, g/l
159.1	158.9	160.9	157.0
	159.2	160.1	
	159.5	160.6	
		160.1	
Average	159.2	160.4	
Error	+0.1%	+0.8%	-1.3%

TABLE II

DETERMINATION OF ZIRCONIUM
IN THE PRESENCE OF TIN AND URANIUM

Zr Content g/l	Zr Found		
	Soln. 2 g/l	Soln. 2A, g/l	Soln. 2B, g/l
50.7	51.4	51.4	51.2
	51.2	51.2	51.7
Average	51.3	51.3	51.5
Error	+1.2%	+1.2%	+1.6%

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