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**A NEW PROCEDURE FOR THE
COLORIMETRIC DETERMINATION OF TELLURIUM**

I. Abstract

A rapid colorimetric method for the determination of traces of tellurium (20-100 γ) depending upon the color of a colloidal suspension of the metal has been developed. It is accurate to 2-3%. Only those ions interfere which form insoluble chlorides or which are reduced to the elemental state by stannous chloride. The time required for a complete analysis is 10-15 minutes.

II. Introduction

The intended use of a tellurium brass in beta-machines produced a need for the analysis of traces of tellurium in the presence of tuballoy and the other metals encountered in plant samples. Work was done by Mr. L. B. Wick to adapt the method outlined in B.R. 193 to our needs. This procedure, involving the use of ferric hydroxide as a carrier for the tellurium, necessitates tedious separation and filtration steps. The method which was finally used employs copper as a carrier and effects a fast, clean-cut separation from the usual interfering ions.

A sample containing 20-100 γ of tellurium and 20-50 mg. of copper is treated with zinc and hydrochloric acid. The precipitated mixture is dissolved in nitric acid, and upon the addition of an excess of stannous chloride in HCl, the

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tellurium suspension is produced. The light absorption of the colloid is measured with a Beckman spectrophotometer.

III. Experimental Work

To establish the applicability of the method, the following factors were investigated:

A. Beer's Law:

The light absorption of a colloidal suspension of tellurium in an acid solution is expressible by Beer's Law when the metal concentration lies in the range 20-120 γ /25 mls. and the optical density is read at 400 $m\mu$. Experimental data are given in Table I. The graph (Figure 1) may be used to convert a density reading directly into γ tellurium present.

Table I

γ Te/25 mls.	Optical Density at 400 $m\mu$
0	.000
20	.034
40	.071
60	.104
80	.133
100	.167
120	.193
140	.208
160	.246
180	.271
200	.278

Above 120 γ Te/25 mls., the values are irreproducible, possibly because of partial coagulation and precipitation of the suspension. The readings are taken at 400 $m\mu$ since the absorption is almost constant between 390-410 $m\mu$; at lower wavelengths Beer's Law is not followed and at higher wavelengths the absorption is less.

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B. Quantitative Precipitation of Tellurium Using Copper as Carrier:

To test the completeness of precipitation of tellurium in the presence of a copper carrier, mixtures containing varying amounts of tellurium and copper were reduced with zinc and hydrochloric acid, the precipitated tellurium-copper mixture dissolved in nitric acid, the tellurium color developed by reduction with stannous chloride and the density read at $400m\mu$. The recovery of tellurium was found to be quantitative when the tellurium concentration was between 20 and 100 γ of Te/25 mls. and the copper concentration between 20 and 50 mg. of Cu/25 mls. (In each case a solution containing 20 or 50 mg. Cu respectively and treated according to procedure was used as a blank. Such a solution has an absorption of .005 for 20 mg. and of .012 for 50 mg. of Cu at $400m\mu$. The approximate amount of copper present must therefore be known to correct for its absorption.) Table II gives some experimental data to show completeness of precipitation.

Table II

γ Te/25 mls. added	Optical Density at $400m\mu$		γ Te found/25 mls.	
	20 mg. Cu/25 mls.	50mg Cu/25mls	20mg.Cu	50 mg. Cu
0	.000	.000	0	0
20	.040	.034	22	20
50	.084	.084	50	50
100	.170	.162	102	98

C. Separation from Interfering Ions:

The simultaneous reduction of 20-50 mg. of copper and 20-100 γ tellurium to the metallic state serves to separate tellurium from T, Fe, Cr, Mn, V, Mo, Ti. In each case the solution contained 10 mg. of the substance whose

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interference was to be determined, 50 mg. of copper and 100% of tellurium. The mixture was treated according to the procedure to be outlined. In each case the density expected from 100% Te/25 mls. at 400 μ was obtained. Separation from Ca, Mg, Al, Sn, etc. is complete also. They were not investigated since their solutions are colorless and would not affect the final density reading in any case. Au, Ag, Se, Pt, Pd, Hg, Sb, and large amounts of Pb should not be present since they either form insoluble chlorides or are reduced to the elemental state by stannous chloride.

D. Miscellaneous Variables:

The density readings were unchanged over the temperature range 15-35 $^{\circ}$, and remained constant for at least 3 hours. (Coagulation and precipitation occurs after approximately 24 hours.) The acidity of the solution is not a critical factor.

IV. Recommended Procedure

1. Place a sample of solution containing 20-100% tellurium in a 100 ml. centrifuge tube. (The amount of copper contained therein can be previously estimated by reducing the solution and comparing the amount of copper precipitated with that from a known solution.)
2. To this solution add enough Cu to bring the total concentration up to 20-50 mg. of copper.
3. Add 15 cc. concentrated hydrochloric acid and then add slowly with shaking 3-5 g. of granulated zinc metal. (30 mesh) Caution!
4. When the reaction is over, dilute with 25 cc. distilled water and boil for 2-3 minutes to dissolve any zinc metal and to produce a coarse copper precipitate.

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5. Allow the precipitate to settle and decant the clear, supernatant liquid, being careful not to lose any precipitate.
6. Wash 3 times with 50 cc. portions of distilled water allowing the precipitate to settle well between each washing and decantation.
(With a little practice all but a few drops of water can be decanted.)
7. Dissolve the precipitate in a minimum (1 cc. usually being sufficient) of concentrated nitric acid. Hood !
8. Dilute with 10 cc. distilled water.
9. Add slowly with shaking 5 cc. of stannous chloride solution prepared by dissolving 50 g. of stannous chloride in 50 cc. concentrated hydrochloric acid and diluting to 100 cc. with distilled water.
10. Pour into a 25 ml. volumetric flask, washing the centrifuge tube with distilled water and adding the washings to the flask. Dilute to mark with a few drops of stannous chloride solution. Shake well for 1 minute.
11. Transfer a part of the above solution to a Corex absorption cell and measure the optical density at $400m\mu$ with a Beckman spectrophotometer, using a solution containing approximately the same amount of copper as the unknown solution in the standard cell.
12. Convert optical density reading into % Te/25 mls. by reference to the graph.

The tellurium color is similar to that of a colloidal gold suspension.

V. Analysis of Some Samples

1. A sample of tellurium brass (.5% Te) was analyzed by this method. The average value of three determination was 0.528% tellurium.

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6.

2. To a solution containing about 1% T, Cr, Fe, Cu, Ca, Mn were added 100% Te. Upon analysis, 98% Te were found.
3. Two samples of T-peroxide which had been precipitated from a solution containing tellurium were analyzed, and found to contain 100% Te per gram of T.

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