

DETERMINATION OF SEMIMICRO AMOUNTS OF TELLURIUM BY  
ELECTRODEPOSITION

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Several gravimetric methods for the determination of tellurium have been reported but each of the methods has some limitations. The well-established procedure for the precipitation of elemental tellurium using sulfur dioxide and hydrazine as reducing reagents does not give complete recovery of the metal. The deviation becomes critical with small amounts of tellurium as has been reported by AAREMAE AND ASSARSSON<sup>1</sup>. Precipitation of tellurium dioxide from neutral solutions as reported by CHENG<sup>2</sup> produces somewhat low results.

Electroreduction of tellurium produces colloidal or poor quality deposit and in general is not quantitative. Semiquantitative or quantitative deposition of tellurium is reported by MATHEWS AND TURNER<sup>3</sup> from hydrofluoric-sulfuric acid baths. LUCAS AND JILEK<sup>4</sup> describe quantitative tellurium deposition onto a platinum dish cathode from sulfuric, tartaric and succinic acid baths. In addition, simultaneous deposition of selenium and tellurium as copper selenide and copper telluride is described by NORWITZ<sup>5</sup>. This method is limited to small amounts of each.

The use of a platinum dish as the cathode is undesirable. Thus a development of an electrodeposition method of tellurium in the micro and semimicro range onto gauze electrodes has been investigated.

## EXPERIMENTAL

*Reagents and apparatus*

A Fisher "powerhouse" direct current power supply was used for the electrolyses. Copper gauze cathodes were constructed from fine wire screen by crimping a cylinder of the screen to a short piece of eighteen gauge copper wire. Each electrode was 2 cm in height. Copper electrodes were cleaned in dilute nitric acid. Nickel, platinum, steel and silver electrodes were prepared in a similar manner as the copper. Platinum wire was used as the anode. All potential measurements were made against the saturated mercury-mercurous sulfate reference electrode with a student type potentiometer. Current was measured with a Weston Model 45 milliammeter. Sargent Model XXI recording polarograph was used for determination of decomposition potentials.

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High purity tellurium metal and tellurium acids were obtained from the Amend Drug Company. Tellurium(IV) oxide was purified by several precipitations of tellurium dioxide from nitric acid solutions. Other chemicals used were reagent grade.

### Procedure

The best tellurium plate is obtained on a copper cathode from 2 *M* sulfuric acid containing 5 g of ammonium sulfate, 2 g of sodium tartrate and about 0.5 g of sodium nitrate per 100 ml of solution. The initial current of approximately 100 mA is applied. Electrodeposition is complete when there is no current change observed. Prolonged electrolysis beyond this point should be avoided. The applied potential is not turned off until the electrodes have been washed with distilled water and removed from the bath. Cathodes are rinsed with distilled water and acetone, and finally dried in air for 20 min and weighed.

### RESULTS AND DISCUSSION

Reproducibility and accuracy of the electrodeposition as a quantitative method for determination of tellurium were checked against known samples. The stock solutions were analyzed by sulfur dioxide-hydrazine gravimetric and dichromate titrimetric methods. Results for determination of tellurium by various methods are summarized in Table I.

TABLE I  
QUANTITATIVE ELECTRODEPOSITION OF TELLURIUM ONTO COPPER CATHODES

<i>Weight of tellurium taken as determined</i>		<i>Weight of tellurium found by electrodeposition<sup>c</sup></i> (mg)
<i>Gravimetrically<sup>a</sup></i> (mg)	<i>Volumetrically<sup>b</sup></i> (mg)	
9.92	10.01	10.00 ± 0.05
15.23	15.64	15.59 ± 0.09
30.46	30.98	30.71 ± 0.15
39.96	—	40.22 ± 0.18
—	51.72	51.63 ± 0.18
103.21	103.44	103.43 ± 0.24

<sup>a</sup> As determined by gravimetric precipitation of tellurium with sulfur dioxide and hydrazine.

<sup>b</sup> Values determined adding an excess of dichromate and back-titrating with ferrous ammonium sulfate.

<sup>c</sup> Average values determined on ten determinations.

The amount of tellurium found by electrodeposition on the whole is somewhat lower than that obtained by the titrimetric method, but is definitely higher than determined by sulfur dioxide-hydrazine reduction. The higher values obtained by the titrimetric procedure are expected as compared to the gravimetric methods. The results obtained by electrodeposition can be reproduced to within 0.6% at the confidence level of 90%.

During electrolysis two precautions should be taken: (1) the initial current should not exceed much over 100 mA, and (2) the deposition should be terminated shortly after the potential and current reach the minimum value as shown in Fig. 1. If the initial current is too high, the tellurium deposit becomes flaky and dark and may be lost upon rubbing. If the deposition is continued much beyond the minimum current value,

the deposit becomes dark and pitted. In both cases low results are obtained. Addition of small amounts of nitrate to the electrodeposition bath prevents hydrogen deposition on the cathode and preserves the metallic nature of the tellurium deposit for longer electrolysis time than without nitrate.

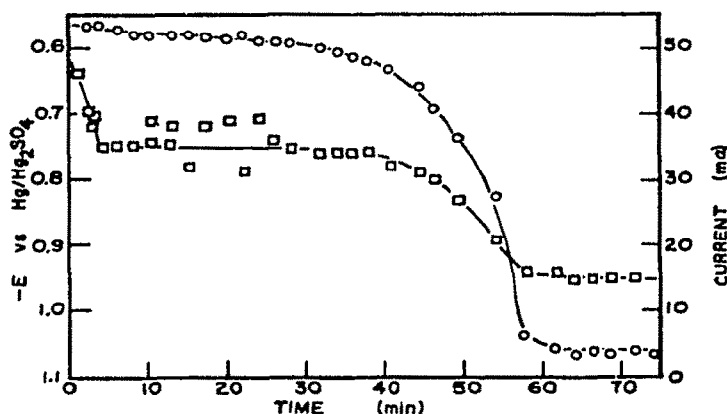


Fig. 1. Polarization curve. Current and cathode potential as a function of time for deposition of tellurium onto a copper gauze electrode from tellurium(IV) solution with sulfuric acid, sodium tartrate, ammonium sulfate supporting electrolyte. Potential  $\circ$ — $\circ$ ; current  $\square$ — $\square$ .

The nature of the cathode is very important in determining the metal on metal polarization potential and the bonding of tellurium on the particular cathode. Copper or copper-plated platinum cathodes are quite satisfactory compared to platinum, silver, nickel or steel.

Separation of tellurium from bismuth, selenium, antimony and copper is unsatisfactory because of the relatively small deposition potential differences, which were determined by the method of LINGANE AND JONES<sup>6</sup>. Addition of chelating agents such as ethylenediaminetetraacetic or tartaric acid and changing the pH does not alter the situation appreciably. In the presence of interfering elements, tellurium must be separated before the electrodeposition.

#### SUMMARY

Electrodeposition of tellurium in micro or semimicro amounts can be achieved using copper or copper-plated platinum cathodes. Best results are obtained from electrolytic baths of approximately 2 *M* sulfuric acid, 0.4 *M* ammonium sulfate, 0.1 *M* sodium tartrate and 0.06 *M* sodium nitrate. Electrodeposition of tellurium can be carried out with an accuracy better than 1% at the 90% confidence level.

#### RÉSUMÉ

L'électrodeposition de micro- et semimicroquantités de tellure peut être effectuée en utilisant des cathodes de cuivre ou de platine cuivré. Les résultats les meilleurs sont obtenus avec des bains renfermant: acide sulfurique 2 *M*, sulfate d'ammonium 0.4 *M*, tartrate de sodium 0.1 *M* et nitrate de sodium 0.06 *M*.

#### ZUSAMMENFASSUNG

Die elektrochemische Abscheidung von Mikro- oder Halbmikromengen Tellur lässt sich mit Hilfe von Kupfer- oder kupferplattierten Platinkathoden durchführen. Die besten Ergebnisse wurden mit einem Elektrolyten aus 2 *M* Schwefelsäure, 0.4 *M* Ammoniumsulfat, 0.1 *M* Natriumtartrat und 0.06 *M* Natriumnitrat erzielt. Die Genauigkeit, mit der die elektrochemische Abscheidung von Tellur ausgeführt werden kann, ist besser als 1 Prozent.

## REFERENCES

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