



NOTE

Determination of Tellurium by Radiochemical Displacement

K. ARUNA* and N.V.S. NAIDU

Department of Chemistry, Sri Venkateswara University, Tirupati-517 502, India

*Corresponding author: E-mail: achalaarunkumar@gmail.com

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A rapid and sensitive method for the determination of tellurium(IV) by the displacement of radio cobalt from its diethyl dithiocarbamate complex in CCl_4 is reported. pH 5 in acetate buffer was found suitable for the quantitative displacement. Effect of diverse metal ions on the displacement were studied. Most of the ions generally associated with tellurium does not interfere and the interference due to Fe(III) and Sb(III) was masked with oxalate. Interference due to other ions was overcome by prior collection of tellurium on $\text{Al}(\text{OH})_3$. Calibration was linear and accurate in the range 2-16 μg Te. Samples of cadmium oxide, copper metal, stream sediments and Sb-In alloy were analyzed.

Key Words: Radiochemical displacement, Determination, Tellurium, Diethyl dithiocarbamate.

Radiochemical displacement technique applied for the displacement of a metal ion from its complex by another metal ion which forms a more stable complex with the same metal ion. The metal being released is labeled with tracer. Correct choice of the radioreagent and the conditional extraction constant of the metal chelate involved in the displacement are required^{1,2}. Determinations based on the technique are reported for the determination of several elements such as Sb^3 , Cd^4 , Pd^5 and In^6 . However, radiochemical displacement has not been employed for the determination of tellurium. Tellurium(VI) displaces Cd^{2+} , Zn^{2+} and Co^{2+} from their diethyl dithiocarbamate complexes in acid media⁷. It is proposed to examine the suitability of the displacement reaction for the determination of Te using the radiolabelled, ^{60}Co -diethyl dithiocarbamate reagent.

All the reagents used were of analytical grade and doubly distilled water was used throughout. Diethyl dithiocarbamate of 99 % purity (E. Merck) was used. 0.38 g of sodium salt of diethyl dithiocarbamate was dissolved in double distilled water and further diluted to 4.4×10^{-4} M as working solution.

50 $\mu\text{g}/\text{mL}$ Co solution containing 30 μCi of ^{60}Co was prepared. The radio reagent solution was prepared by mixing 50 mL of freshly prepared sodium salt of diethyl dithiocarbamate solution and labeled Co solution and the pH adjusted to 8 with borate buffer. The reagent formed was extracted into 100 mL CCl_4 and stored in a brown bottle.

Preparation of samples: In-Sb alloy: 1g sample was digested with 5 mL 1:1 HCl by gently warming and made up to 25 mL.

Cadmium oxide: 1 g Cadmium oxide was brought into solution by treatment with 1:1 HCl and made up to 50 mL.

Stream sediments: 1 g sample was fused with 2 g $\text{NaNO}_3 + \text{CH}_3\text{COONa}$ at 600 °C in a platinum crucible⁸. The contents were cooled and leached into water, filtered and warmed with 4N HCl and made up to 50 mL withdrawn and the activity is measured (Fig. 1).

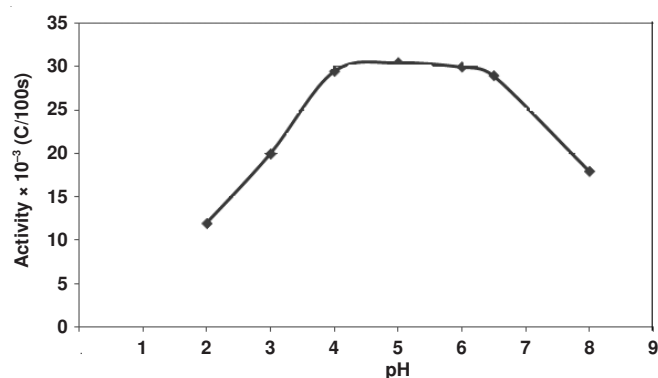


Fig.1: Effect of pH on the displacement of Co^* from Co:DDC by Te(IV)

Copper metal: 1 g Copper was dissolved in 1:1 HCl + H_2SO_4 and heated gently and evaporated to fumes of sulphur trioxide, cooled and made up to 25 mL with water and adjusted to 4N with respect to HCl.

Cobalt tracer as ^{60}Co was obtained from M/s Isotope Group, Bhabha Atomic Research Centre, Mumbai.

A well type NaI(Tl) scintillation counter was employed for the γ activity measurements of 1.3 MeV ^{60}Co .

Effect of pH on the displacement of Co^* from the radio reagent by Te: A series of solutions containing 10 μg of Te(VI) were taken and the pH adjusted to 1.0 to 6.0 with acetate buffers. 5 mL of the radio reagent was added and equilibrated. 1 mL of the aqueous solution of Te(IV).

Interference studies: Effect of diverse ions on the displacement are studied with 1 mg of each ion to 10 μg of Te(IV) at pH 5. 2% change in the activity displaced was considered to be the interference due to the corresponding species (Table-1). Interference due to the other ions can be overcome by selectively collecting Te(IV) on $\text{Al}(\text{OH})_3$.

Foreign ion added (1 mg)	Remarks
Ni^{2+} , Cd^{2+} , Mn^{2+} , Ba^{2+} , Zn^{2+} , Ca^{2+} , Mg^{2+} , Al^{3+} , SeO_4^{2-} , F^- , Cl^- , Br^- , I^- , NO_3^- , SO_4^{2-} , PO_4^{3-} , Oxalate and EDTA	No interference
Hg^{2+} , Cu^{2+} , Pb^{2+} , Cd^{2+} , Fe^{2+} , Fe^{3+} , Bi^{3+} , AsO_3^{3-} , VO_3^- , Sb^{3+} , tartrate and citrate	Interfere by increasing the activity displaced

* Can be masked with 1 mL of 2 % sodium oxalate

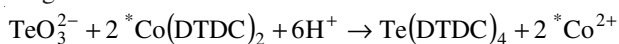
Calibration: Aliquots of Te(IV) varying from 0 to 15 μg were taken in a series of equilibrium tubes and pH adjusted to 5 with acetate buffer. The solutions are equilibrated with 5 mL of the radio reagent in CCl_4 . Two mL of the aqueous layer was withdrawn into counting tube and the activity ascertained to obtain calibration determination of Te(IV).

Synthetic mixtures: The method was initially tested with synthetic mixtures containing diverse metal ions. In the case of samples containing Cu(II), Cd(II), In(II), Zn(II) and Te(IV) was precollected on $\text{Al}(\text{OH})_3$ and the precipitate washed 0.1 M EDTA solution and buffer solution of pH 5. The precipitate was then dissolved in 0.5 N H_2SO_4 and 1 % solution of oxalic acid. The pH of the solution was adjusted to 5.0 and the Te(IV) determination was made as earlier (Table-2).

Added ions	Te (IV) found	% Recovery
Cd (II) + Cu (II)	9.95 \pm 0.05	99.5
Fe (III) + Zn (II)	9.85 \pm 0.15	98.5
Sb (III) + In (III)	9.90 \pm 0.10	99.0
Se (IV) (1000 μg)	10.00 \pm 0.05	100.0
Se (III) (2000 μg)	10.10 \pm 0/18	101.0

Samples: Suitable aliquots of the sample solution and samples spiked with known amount of Te(IV) were analyzed after $\text{Al}(\text{OH})_3$ precollection. The results were compared with the analysis by ICP-AES (Table-2).

A simple and highly sensitive radiochemical displacement for the displacement of Te(IV) from labeled Co (DTDC)₂ in CCl_4 has been developed. The displacement probably proceeds through



pH 5 was suitable for the reaction. Calibration curve was linear in the range 2-16 μg of Te and the detection limit was 2.5 ppb and

Sample (1g/50 mL)	Tellurium found* (%)	Tellurium added (μg)	Tellurium found (Present method) ^a		Recovery %
			μg	%	
Cadmium oxide	0.00035	-	3.5 \pm 0.1	-	-
		10	13.4 \pm 0.2	0.0003	99.0
		20	23.6 \pm 0.6	-	100.5
Copper metal	0.0192	-	190.0 \pm 3.4	-	-
		50	144.0 \pm 2.2	0.0190	98.4
		100	193.0 \pm 3.1	-	99.0
Stream sediment	-	-	-	-	-
Sb-In alloy (1g/25 mL)	0.0033	50	51.0 \pm 0.9	-	102.0
		-	15.5 \pm 0.2	-	-
		25	40.4 \pm 0.7	0.0031	99.6
		50	66.0 \pm 1.2	-	101.0

a. Average of 3 determinations
*. ICP – AES values

the relative standard deviation was 2.4 % (Fig. 2). Many ions generally associated with Te do not interfere and the interference due to Fe(III) and Sb(III) can be masked with oxalate. Interference due to other species is hard to remove and prior collection of Te with $\text{Al}(\text{OH})_3$ is adopted for their suppression (Table-1). Relatively, 300 folds of Se could be tolerated. The success of the developed method is illustrated in the analysis of synthetic mixtures and samples of cadmium oxide, copper metal, stream sediments and Sb-In alloy. The results compare well with ICP-AES values.

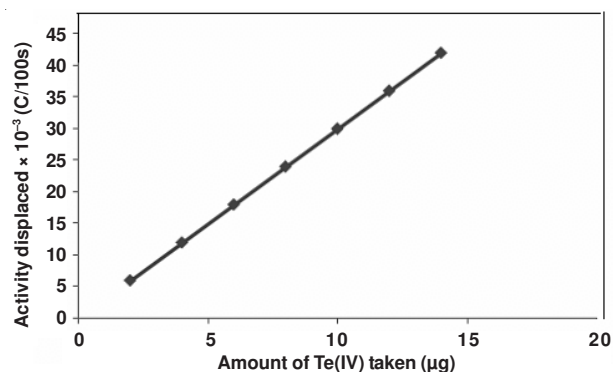


Fig. 2: Calibration curve

The method is simple and sensitive and gives accurate and precise results. The method developed for the determination of Te can be expected to find application for its determination in wide range of materials.

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