

NOTE

DACs—New Amperometric Reagent for Thorium

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3-3' Bis [N-N' dicarboxy methyl] *o*-cresol sulphothalein known as DACs has been used as reagent for the amperometric titrations of chromium(IV) at pH 3.10 ± 0.02 in Britton-Robinson buffer. The minimum amount of thorium about 10 μ g. could be detected successfully with an error less than $\pm 1.0\%$. The results of amperometric titrations are also supported by spectral studies. Job's method and mole ratio method were applied to confirm the stoichiometry of thorium : DACs complex formation. The result revealed that 1 : 2 complex with stability constant value of $\log \beta = 6.3$ has been formed in the prescribed experimental condition. The effect of diverse ions on the amperometric titration of thorium with DACs revealed that small amount of rare metals and rare earths hampered the titrimetric procedure.

In continuation to our work¹⁻³ the present paper reports the results of spectrophotometric and amperometric determination of thorium with 3-3' bis (N-N' dicarboxy methyl aminomethyl) *o*-cresol sulphothalein (DACs). The existing literature revealed that the application of DACs as spectrophotometric and analytical reagent^{4,5}. However, the present study deals with the determination of small amount of thorium with DACs.

The chemicals used were of AnalaR/extra-pure quality. The solution of thorium nitrate was prepared by dissolving its requisite quantity in double distilled water and standardized⁶. A fresh solution of xylenol orange and DACs was prepared whenever required. Britton-Robinson buffer was prepared with usual method. The polarography of DACs has been made on CLO-2B Toshniwal digital polarograph. The capillary of $m^{2/3} t^{1/6} = 2.13 \text{ mg}^{2/3} \text{ sec.}^{-1/2}$ at 40 cm., effective height of mercury column was used. Toshniwal digital pH meter (Model CL-46) was used for pH measurements. Photometric observations were made on Milton and Roy spectrophotometer. The absorption was measured at 625 nm.

DACS gives single reduction wave at pH < 4.0 and two step reduction wave at pH > 4.2. For amperometric titrations, sets of solution containing known amount of thorium in 10 ml. Britton-Robinson buffer at pH 3.10 was prepared and each of the solutions was taken into the titration cell, the plateau potential of -1.2 V vs Hg pool was applied and then DACs was added drop by drop and current changes were noted. The current vs.

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volume plots revealed reversed L shaped curve indicating 1 : 2 stoichiometric ratio of thorium (IV) DACS complexation. Thorium (IV) gives red coloured complex with DACS. Job's method⁷ and mole ratio method⁸ was applied to determine the stoichiometry of said complexation, which indicates the formation of 1 : 2 thorium-DACS complex, which supports the observed results of the amperometric titrations. The results of amperometric studies and spectral studies are given in Table 1 and Table 2 respectively. The statistical methods have been successfully applied. The standard deviations were calculated from the replicable sets of observations and the coefficient of variation (Table 1 and 2) reveals that the ampero-

TABLE 1
AMPEROMETRIC DETERMINATION OF THORIUM WITH DACS
Plateau potential = - 1.2 V Vs Hg pool, pH = 3.15

S. No.	Amount of Thorium		% error	S. D.*	C.V
	Taken (Mg)	Found (Mg)			
1.	0.094	0.0931	-0.957	0.00658	0.70
2.	0.188	0.1878	-0.106	0.00168	0.89
3.	0.282	0.2829	+0.319	0.00240	0.84
4.	0.376	0.3759	-0.026	0.00294	0.78
5.	0.470	0.4720	+0.425	0.00235	0.49

N.B. 1. *Values determined from replicate sets of observations.

2. % error is < 1 and the values of C.V. lie in the range 0.5 to 0.9.

TABLE 2
SPECTROPHOTOMETRIC DETERMINATION OF
THORIUM WITH DACS
pH = 3.10 λ_{\max} = 625 nm

S.N.	Amount of Thorium		% error	S.D.*	C.V.
	Taken (Mg)	Found (Mg)			
1.	0.140	0.1420	+1.42	0.0015	1.05
2.	0.188	0.1911	+1.64	0.0023	1.20
3.	0.282	0.2830	+1.02	0.0031	1.09
4.	0.376	0.3754	-1.59	0.0045	1.19
5.	0.470	0.4650	-1.06	0.0058	1.24

N.B. 1. *The values obtained from replicate sets of titration.

2. % error is > 1 and the values of C.V. lie in the range 1.05 to 1.25.

metric method of determination has low detection limit with accuracy. Various cations and anions do not hamper the titration of thorium with DACS. However, small amounts of rare earths and rare metals if present affect it seriously.

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(Received: 1 May 1991; Accepted: 15 June 1991)

AJC-330

Coordination Chemistry

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July 19–24, 1992

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