

NOTES

A High Frequency Titrimetric Study of the Reaction between Thorium and Alkali Molybdate

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The reaction between Th^{4+} and MoO_4^{2-} ions has been investigated by means of high frequency titrations of thorium nitrate with sodium molybdate, by both the direct and reverse methods, in 30% ethanolic media. The end-points obtained from the well-defined breaks in titration curves provide cogent evidence for the formation of normal thorium molybdate, $\text{ThO}_2 \cdot 2\text{MoO}_3$, in the pH range from 4.2 to 5.8. The precipitation of the compound has been found to be quantitative. The reaction offers a simple, rapid, and accurate method for the determination of thorium as molybdate.

One of the present authors, Mittal,¹⁻³ has made a detailed study of this reaction by several electro-metric methods. The present investigation has been undertaken with a view of substantiating the previous results and studying the possibility of developing a reaction for the analytical determination of thorium. There is, however, no reference in the literature to any high-frequency titrimetric study of this reaction.

Anal. R. (BDH) reagents, thorium nitrate, and sodium molybdate were used; their solutions were prepared in air-free conductivity water. A high-frequency titrimeter of the HFT 30 C type (WTW Germany) with a working frequency of 10 Mc./s. $\pm 1\%$ and a capacity range of 0.25 pF was used; all the high-frequency titrations were carried out by adopting the deflection method. One hundred milliliters of a 30% ethanolic solution of titre was taken into the cell each time. After each addition of a titrant, the cell solution was stirred by an electrically-driven stirrer and the corresponding scale reading was noted and plotted against the volume of titrant added. Only one figure has been given for the sake of brevity (Fig. 1).

Using different concentrations of reactants, thorium nitrate and sodium molybdate a series of

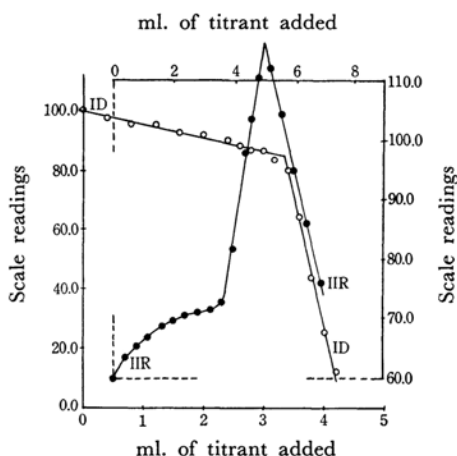


Fig. 1. Direct (curve ID; $\text{M}/10 \text{ Th}(\text{NO}_3)_4$ vs. $\text{M}/150 \text{ Na}_2\text{MoO}_4$) and reverse (curve IIR; $\text{M}/10 \text{ Na}_2\text{MoO}_4$ vs. $\text{M}/400 \text{ Th}(\text{NO}_3)_4$) high frequency titrations between thorium nitrate and sodium molybdate.

high-frequency titrations were carried out by both the direct and inverse methods. Table I illustrates the concentrations employed and the results of study.

In direct titrations, when sodium molybdate is used as the titre (Fig. 1, curve ID), it may be noted that, on the addition of the titrant (thorium

Table I. A SUMMARY OF THE RESULTS OF HIGH FREQUENCY TITRATIONS

Concentrations		Equivalence points (ml.)	
$\text{Th}(\text{NO}_3)_4$	Na_2MoO_4	Calcd.	Obs.
$\text{M}/10$	$\text{M}/150$	3.33	3.35
$\text{M}/40$	$\text{M}/500$	4.00	4.00
$\text{M}/75$	$\text{M}/750$	5.00	5.05
$\text{M}/100$	$\text{M}/1254$	4.00	3.95
Na_2MoO_4	$\text{Th}(\text{NO}_3)_4$	Reverse titrations Fig. 1, curve IIR	
$\text{M}/10$	$\text{M}/400$	5.00	5.00
$\text{M}/20$	$\text{M}/1000$	4.00	4.02
$\text{M}/25$	$\text{M}/1500$	3.33	3.30
$\text{M}/40$	$\text{M}/2000$	4.00	4.05

1) R. S. Saxena and M. L. Mittal, *J. Sci. Ind. Res. (India)*, **21B**, 92 (1962).

2) R. S. Saxena and M. L. Mittal, *Z. Phys. Chem. (N. F.)*, **34**, 319 (1962).

3) R. S. Saxena and M. L. Mittal, *Naturwissenschaften*, **50**, 373 (1963).

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nitrate), the scale reading decreases slightly until the stoichiometric end-point is reached; then, when all the MoO_4^{2-} ions have been removed by precipitation as thorium molybdate, there is a sudden decrease in the scale reading. In the case of reverse titrations when thorium nitrate solutions were used as titre, upon the addition of sodium molybdate the scale reading first increases slightly and then, in the vicinity of the end-point, appreciably until the point corresponding to the $\text{ThO}_2 : \text{MoO}_3$ ratio of 1:2 is reached, after which there is a rapid decrease. Both direct and reverse titrations yield well-defined breaks and suggest the formation of normal thorium molybdate, $\text{ThO}_2 \cdot 2\text{MoO}_3$. The accuracy

and reproducibility of these titrations have been found to be excellent, even at considerably low dilutions of thorium salt (0.5 mm). In view of the accuracy of the method and its applicability to dilute solutions, this reaction can be recommended for the quantitative determination of thorium as molybdate.

The results of high-frequency titrations fully support those obtained with other electrometric methods (*loc. cit.*).

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