

**Determination of Molybdenum In Soil using
Sodium Pentamethylene Dithiocarbamate**

V. V. RAMANA, K. SANTHA, M. DASARATHA RAMAIAH
and

K. SARASWATHI*

Department of Chemistry, S. V. University,
Tirupati-517 502

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SODIUM pentamethylene dithiocarbamate (pipdte) has been widely used for the determination of many metal ions¹⁻³. A simple procedure is adopted for the determination of molybdenum in the soil of Tirupati area. The results are comparable with standard thiocyanate method.

Na(pipdte) was prepared, recrystallised from benzene, and a 1% stock solution was made in double-distilled water and stored in dark. Standard molybdenum solution (0.001 M) was prepared by dissolving ammonium molybdate in double-distilled water, and standardised by oxine method. Standard solutions of diverse ions were prepared from their chlorides, sulphates or from sodium, potassium or ammonium salts. Chloroform was distilled before use. All other chemicals were either chemically pure or reagent grade unless otherwise mentioned. Magnesium sulphate was used as a salting-out agent. Absorbance measurements are made using a Shimadzu PR-1 spectrophotometer and pH adjustments are made on an Elico LI-10 pH-meter.

Results and Discussion

An aliquot of Mo^{VI} solution, 3% hydrazine sulphate solution (2 ml) to reduce Mo^{VI} to Mo^{IV}, 10 M HCl (2 ml), 1% pipdte (1 ml) and 0.2 M MgSO₄ solution (1 ml) were mixed and made upto 10 ml by adding double-distilled water. The complex formed was extracted with chloroform (10 ml). After equilibration the organic layer was separated and the absorbance was measured at 460 nm against reagent blank. Neither the reagent nor the metal ion had any absorbance in the range studied. The complex was stable and the extraction was complete in

8–10 M HCl The influence of reagent concentration for complete complex formation and influence of salting-out agent for complete extraction in single step have been examined. It was noticed that 2 ml of 2% aqueous solution of pipdte and 1 ml of 0.2 M MgSO₄ as salting-out agent caused quantitative extraction within 1–2 min of shaking. Beer's law was found to be obeyed in the concentration range 1–17 µg ml⁻¹ of Mo^{VI}, the complex being stable for more than 6 h. The molar absorptivity of the complex was found to be $4.0 \times 10^3 \text{ dm}^3 \text{ mol}^{-1} \text{ cm}^{-1}$ and the Sandell's sensitivity (absorbance=0.01) 2.1 µg cm⁻². The composition of complex found by Job's method of continuous variation, molar-ratio method and Asmus method was 1 : 2.

The instability constant of the complex calculated by Edmonds Birnbaum's method (2.7×10^{-9}) at room temperature was in good agreement with the calculated value (2.84×10^{-9}) using Asmus method.

Traces of iron, vanadium, copper, cobalt, nickel, bromate, iodate, periodate ions did not interfere. The interference of iron, vanadium, cobalt and nickel was masked by ascorbic acid. The anions like oxalate, acetate, tartrate, nitrate, sulphate, silicate and EDTA did not interfere. Phosphate also did not interfere upto 100 folds.

Estimation of Mo^{IV} in soil samples : The procedure was applied for the determination of Mo^{VI} in soil samples of Tirupati area. The soil samples were extracted and brought to solution using the standard methods^{4,5}. The amounts of Mo^{VI} in the samples determined by the proposed method were in good agreement with the values obtained by standard potassium thiocyanate method⁶ (shown in the parenthesis) : 0.14 (0.15), 0.18 (0.18), 0.22 (0.22) µg/10 g of soil.

References

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