A CARBONATO COMPLEX OF MOLYBDENUM K₆Mo(CO₃)₅:2H₂O*

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Mellor (1948, p. 659) states that no molybdenum carbonate has been reported and there appears to be nothing in more recent literature.

The carbonato complex reported in the present paper was formed when an attempt was made to produce an iodo complex with molybdenum(III).

Experimental

Potassium molybdate solution was made acid with hydriodic acid and about 2 g of potassium iodide added. This solution was electrolysed using a mercury cathode and a carbon anode. The iodine liberated, markedly corroded the carbon anode and a carbon deposit formed which was filtered off. The carbon anode was replaced by a platinium anode and electrolysis was continued. The final reduced solution was a very light brown-red colour which was quite different from the more intense red coloured solutions of molybdenum(III), chloride or bromide.

The reduced solution was evaporated under suction at 70° C to about 50 ml; ethanol was added; two layers formed, the electrolysed solution and the ethanol layer. Carbon dioxide was bubbled through the reduced electrolyte and a white precipitate formed from the aqueous layer. The precipitate was filtered, washed with ethanol, and dried at 100 °C (Found: Mo, 14 \cdot 7; CO₃, 45 \cdot 0; K, 35 \cdot 4%). This is in agreement with K₆Mo(CO₃)₅.2H₂O or K₄Mo(CO₃)₄.K₂CO₃.2H₂O.

The molecular conductivity of M/1016 at 25 °C=680 mhos. The conductivity increases slightly with time indicating that the compound undergoes some hydrolysis.

Magnetic susceptibility measurements showed this compound to be diamagnetic ($\chi \times 10^6 = -0.052$) involving Mo(IV).

^{*} Heide and Hofmann (1896) reduced an acid solution of molybdenum trioxide with potassium iodide. The excess iodine was removed by electrolysis. To this

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solution they added potassium cyanide to obtain red crystals of the red cyanide \cdot to which they assigned the formula 4KCNMoO₂.10H₂O.

This process is comparable to that just described for the preparation of the carbonato complex. In both cases the solution was completely reduced to molybdenum(III) by means of potassium iodide; in one case potassium cyanide is added and in the other carbon dioxide; both additions producing complexes.

References

HEIDE, K. VAN DER, and HOFMANN, K. A. (1896).—Z. anorg. Chem. 12: 277.
MELLOR, J. W. (1948).—" A Comprehensive Treatise on Inorganic and Theoretical Chemistry." Vol. 9. (Longmans: London.)