

A Correction of the Crystal Structure of $\text{MoO}_3 \cdot 2\text{H}_2\text{O}$

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In order to refine the crystal structure of $\text{MoO}_3 \cdot 2\text{H}_2\text{O}$ a reinvestigation was started in this Institute. Prolonged exposures then showed that the crystal has a considerable superstructure. The new axes a, b, c are related to the earlier a', b', c' in the following way: $a = 4a', b = 2b', c = 2c'$. The cell dimensions are thus $a = 15.08 \text{ \AA}, b = 13.82 \text{ \AA}, c = 14.68 \text{ \AA}$ and $\beta = 90^\circ 40'$. The loss of orthorhombic symmetry which could not be detected in the intensities earlier is now obvious for the superstructure reflexions. It has been checked that the main principles of the suggested structure cannot be influenced by the superstructure but there are of course, small changes in the oxygen positions. The calculated bond distances (except for Mo-Mo) are thus of little value and therefore the basis is removed for the suggestion about the existence of an ion H_4O^{++} .

Because of change of axes during the work an obvious error has been made in the description of the structure in Ref.¹: x and z parameters and h and l indices should be interchanged all through the paper to fit the choice of axes.

A complete reinvestigation will be very time-consuming and is not planned at present.

1. Lindqvist, I. *Acta Chem. Scand.* 4 (1950) 650.

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A Nuclear Magnetic Resonance Study of the Dihydrate of Molybdenum Trioxide

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A combined proton magnetic resonance and infra-red investigation of the dihydrate of molybdenum trioxide has shown

that the formula $(\text{H}_4\text{O}^{++})\text{MoO}_4^-$ is very improbable¹.

The second moment of the proton absorption line at 77°K is 27.6 gauss². If a symmetrical H_4O^{++} ion existed in the crystal, this value would predict³ an O-H distance of 1.16 Å in H_4O^{++} , compared with 0.96 Å in water vapour, and this seems an improbably large increase. In addition, the infra-red spectrum shows frequencies at 3100 and 1592 cm^{-1} ; if these are interpreted as stretching and bending frequencies associated with O-H groups, they suggest an expansion of the order of 0.04 Å only. It would be difficult to reconcile both of these results with the existence of an H_4O^{++} ion.

We hope to publish these results in detail elsewhere.

1. Lindqvist, I. *Acta Chem. Scand.* 4 (1950) 650.
2. Van Vleck, *Phys. Rev.* 74 (1948) 1168.

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Metalation of Pyrene

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Only few strictly aromatic hydrocarbons have been metalated by organolithium compounds¹ and the yields of carboxylic acids obtained upon carbonation have been poor.

Pyrene, in view of its readiness to partake in substitution reactions, and especially of the general rule of substitution in 3-position² governing these reactions, seemed an interesting object for metalation.

It has now been found that pyrene, by the action at room temperature of a rather large excess of *n*-butyllithium in diethyl ether not only is metalated as evidenced by the isolation of pyrene carboxylic acids upon carbonation of the reaction mixture, but also is attacked by the organometallic reagent in positions 1 and 4 in addition to the usual 3-position, that is in all isomeric positions in the pyrene molecule. This was shown by the isolation from the acid mixture of three acids two of which were

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