An Alternately Intercalated Compound of Tantalum Disulphide

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Summary If the known second-stage intercalation compound of pyridine and tantalum disulphide is contacted with ammonia vapour, the new guest is incorporated in the previously unfilled interlayer spaces, resulting in an alternately intercalated compound; thermogravimetric and *X*-ray diffraction analyses are corroborative.

FREEMAN recently described the preparation of a doubly occupied graphite intercalation compound, one in which the carbon interlayers are regularly either unoccupied, filled with $FeCl_3$, or filled with N_2O_5.[1] In the course of our work on the inclusion chemistry of the layered transition-metal dichalcogenides, we have prepared an intercalation compound of tantalum disulphide in which the interlayers are alternately filled with pyridine and ammonia, leaving no vacant van der Waals regions.

Tantalum disulphide is known to form both a first-stage and a second-stage intercalation compound with pyridine simply by heating the appropriate amounts together.[2] Ammonia has also been shown to form both phases, the second-stage product being formed on partial deintercalation of the fully loaded compound.[3] The formation of staged layered compounds is most clearly evident in their *X*-ray diffraction patterns, which will show repeat distances

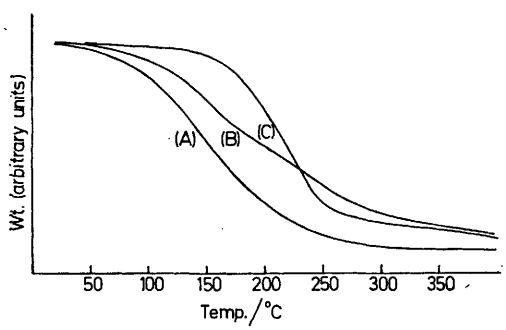

FIGURE. Thermogravimetric results. (A) $TaS_2(NH_3)$; (B) $TaS_2(pyr)_{0.25}(NH_3)_{0.5}$; (C) $TaS_2(pyr)_{0.25}$.

perpendicular to the compound slabs. (For a hexagonal lattice, these repeats are the c spacings.) Data are summarized in the Table.

TABLE. Repeat distances (c/n spacings) from low angle X-ray diffraction data

Compound	Repeat distance/Å
TaS_2	6·05
$TaS_2(pyr)_{0·5}$[a]	11·85
$TaS_2(pyr)_{0·25}$[b]	18
$TaS_2(NH_3)_{1·0}$[a]	9·1
$TaS_2(NH_3)_{0·5}$[b]	15
$TaS_2(pyr)_{0·25}(NH_3)_{0·5}$ ..	21

[a] First stage. [b] Second stage.

If the pyridine second-stage compound is contacted with ammonia vapour at 760 Torr and 23 °C, *ca.* 0·5 equiv. of NH_3 is taken up per TaS_2 over *ca.* 1 day. Ammonia normally forms a first-stage compound with TaS_2 containing a 1 : 1 ratio of guest to host in this time period.[3] The thermogravimetric curves of the normal ammonia–TaS_2, pyridine–TaS_2, and $TaS_2(pyr)_{0·25}(NH_3)_{0·5}$ product are shown in the Figure. In its X-ray powder pattern, the mixed product has reflections which are consistent with a repeat distance of *ca.* 21 Å (or 2 × 21 Å) which would be expected for an alternately intercalated compound whose component interlayer distances are *ca.* 12 and 9 Å, the values found in the respective first-stage compounds of pyridine and ammonia.

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[1] A. G. Freeman, *J.C.S. Chem. Comm.*, 1974, 746.
[2] F. R. Gamble, J. H. Osiecki, M. Cais, R. Pisharody, F. J. DiSalvo, and T. H. Geballe, *Science*, 1971, **174**, 493.
[3] M. B. Dines and R. B. Levy, to be published (presented at 168th A.C.S. Meeting, Atlantic City, New Jersey, September 1974).