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## Neutron-diffraction study of $Al_2Th$.

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The following structure has been suggested for $Al_2Th$ on the X-ray powder-pattern evidence (Braun & van Vucht, 1955):

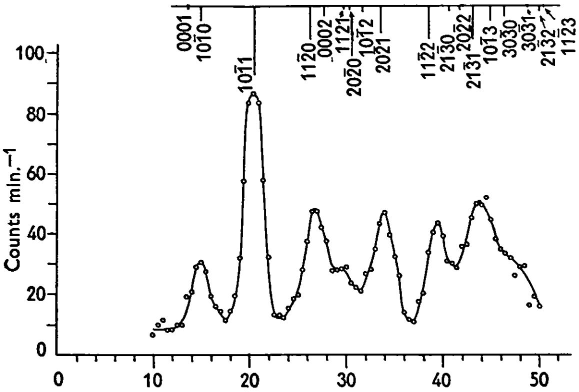

Fig. 1. Neutron-diffraction pattern of $Al_2Th$, with calculated intensities indicated.

Hexagonal, $a = 4{\cdot}393$ Å, $c = 4{\cdot}164$ Å;
Th at $(0, 0, 0)$; Al at $\pm(\frac{2}{3}, \frac{1}{3}, \frac{1}{2})$.

However, the X-ray scattering is dominated by the Th atom, and so is rather insensitive to the Al position. The neutron-scattering amplitudes are more favourable in this respect (Th = 1·01, Al = $0{\cdot}35 \times 10^{-12}$ cm.), and therefore the neutron powder diagram was taken as a further check.

11 g. of the alloy were used in a cylindrical specimen container of 1 cm. diameter made of aluminium. Counts were taken at intervals of 0·5° in $2\theta$ over 20 min. The wavelength was 1·00 Å.

Good agreement was obtained between the observed intensities and those calculated for the suggested structure. This is illustrated by Fig. 1.

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## The crystal structure of copper hydride.

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### Introduction

If solutions of hypophosphorous acid and copper sulphate are warmed together, a brown solid precipitates which gives off hydrogen when heated above 60° C. Its main constituent is believed to be copper hydride, CuH, although one can find doubt expressed as to whether this exists as a well-defined compound.

Hüttig & Brodkorb (1926) took an X-ray diagram of the precipitate from which they concluded that in the hydride the copper atoms form a face-centred cubic lattice, $a = 4{\cdot}33$ Å, although they admit that this interpretation accounts for only part of the lines they observed. In what appears to be a much more careful study, Müller & Bradley (1926) conclude that the unit cell is hexagonal, $a = 2{\cdot}89$, $c = 4{\cdot}61$ Å, with Cu at $0, 0, 0$; $\frac{1}{3}, \frac{2}{3}, \frac{1}{2}$.

Nevertheless, some recent compilations, such as Sidgwick (1950), and also the ASTM card index, give the cubic structure. Wyckoff (1951), on the other hand, follows Müller & Bradley, and tentatively assigns the wurtzite (zincite) structure to the compound.

In view of this discrepancy, and the still somewhat dubious nature of the precipitate, we have taken up the problem once more, but now also used neutrons to verify the positions of the hydrogen atoms. Because of the large incoherent neutron scattering cross-section of hydrogen, we also prepared and investigated the deuteride.

### Preparation of samples

Copper hydride was made according to Vanino (1926). The deuteride was prepared in an analogous way, using deutero-hypophosphorous acid, made from its calcium salt, which was synthesized from CaO, heavy water and white phosphorus. No attempt was made to remove all water as the precipitate becomes pyrophoric when completely dry.

X-ray analysis showed that the precipitates made in this way always contained some metallic copper, and sometimes also $Cu_2O$. The amounts of these impurities could be estimated from the X-ray diagrams. This, together with an electrolytic determination of the total copper content, gave the amount of water. The composition of the samples, taking total Cu as unity, was as follows:

| | CuH(CuD) | Cu | $Cu_{\frac{1}{2}}O$ | $H_2O(D_2O)$ |
|---|---|---|---|---|
| Copper hydride | 0·89 | 0·06 | 0·05 | 0·73 |
| Copper deuteride | 0·91 | 0·09 | 0·00 | 1·01 |

### X-ray work

A diffractometer study of the hydride completely confirmed the results of Müller & Bradley. It was noted that the line broadening they reported is particularly pronounced for high values of $l$. In fact, using Jones's

(1938) method for eliminating instrumental line width, the broadening could be very well accounted for by assuming the particles to be hexagonal prisms, 60 Å high, with an 85 Å base.

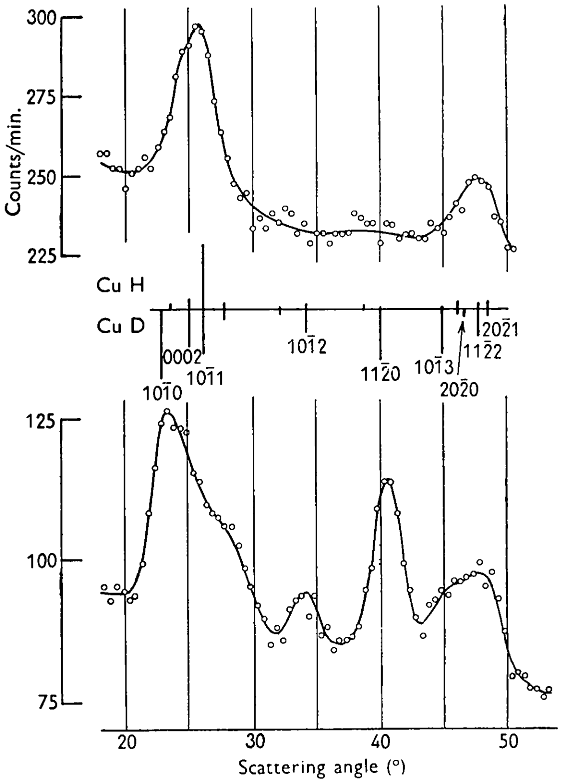

Fig. 1. Diffracted neutron intensities for CuH and CuD.

The temperature factor was found to be high, exp $-4\cdot06\ (\sin\theta/\lambda)^2$ in the structure factor.

## Neutron work

Neutrons were obtained from the Kjeller reactor running at 250 kW. A lead single-crystal monochromator was used, set for 1·00 Å. The data were recorded on an automatic goniometer, which will be described elsewhere. Counts were taken over 20 min. at intervals of 0·5° scattering angle.

For the hydride, a flat cell of 8 mm. = 0·62 g.cm.$^{-1}$ was used in transmission. Because only a small amount of deuteride was available, it was placed in a cylindrical container of 1 cm. diameter.

The observed counting rates are shown in Fig. 1. Intensities calculated for the wurtzite arrangement, i.e.

$$\text{Cu at } 0, 0, 0;\ \tfrac{1}{3}, \tfrac{2}{3}, \tfrac{1}{2},\ \text{H at } 0, 0, \tfrac{3}{8};\ \tfrac{1}{3}, \tfrac{2}{3}, \tfrac{7}{8},$$

are also shown. The small, unmarked intensities are those due to Cu and $Cu_2O$. Although there is considerable overlapping, especially where peaks of high $l$ are involved, the general agreement leaves no doubt that the wurtzite structure is correct.

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## The unit-cell dimensions and space group of 1,1'-diethyl-2,2'-pyridocyanine iodine.

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The crystals used in this study were obtained from a sample prepared by Mr G. S. Levinson, using the method of Brooker & Keyes (1935), and had been crystallized from aqueous solution. Although most of the crystals were multiply twinned or poorly formed, a few suitable for single-crystal X-ray photographs were found. These were small needles, bounded on the sides by prominent 110 and $1\bar{1}0$ and small 100 faces, and on the ends by 001 faces. The crystals are deep orange in color and under the polarizing microscope show distinct pleochroism, the color varying from yellow to deep red, with the direction of maximum absorption in the 010 plane approximately −35° from the c axis.

Oscillation, rotation, equi-inclination Weissenberg and precession photographs were taken, using Cu $K\alpha$ radiation. The cell dimensions were determined from the rotation and zero-level Weissenberg photographs, the camera radius of 57·074 mm. having been determined by calibration with sodium chloride.

The dimensions of the monoclinic unit cell were found to be:

$$a_0 = 11\cdot94\pm0\cdot01,\ b_0 = 10\cdot66\pm0\cdot02,\ c_0 = 12\cdot97\pm0\cdot02\ \text{Å},\ \beta = 114\cdot6\pm0\cdot1^\circ.$$

Systematic extinctions were found for $hkl$ reflections with $h+k$ odd and for $h0l$ reflections with $l$ odd. The space group must then be either $Cc$–$C_s^4$ or $C2/c$–$C_{2h}^6$. The observed density of 1·557 g.cm.$^{-3}$ agrees well with the density of 1·567 g.cm.$^{-3}$ calculated on the basis of four formula units per unit cell.

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