

electrons, which again conforms to the explanation given<sup>18</sup> for the large small-angle scattering of electrons by the helium atoms.

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## CRYSTALLOGRAPHY

### Interdiffusion Method for the Preparation of Single Crystals of Certain Hydroxides and Basic Salts

SINGLE crystals of insoluble compounds have been grown<sup>1</sup> by means of slow interdiffusion of solutions containing the constituent ions or molecules. By making use of ammonia is one of two vertically interdiffusing solutions, I have grown single crystals, with dimensions ranging from a few tenths of a mm to a few mm, of the hydroxide, the basic nitrates and a basic sulphate, as well as the isomorphous basic selenate, of cadmium. Likewise single crystals of some of the corresponding compounds of divalent manganese and cobalt have been prepared.

Cadmium hydroxide was made in the following way. By means of a pipette, a 0.5 M solution of cadmium nitrate was carefully introduced at the bottom of a 200-ml. beaker previously three-fourths filled with distilled water. The beaker was placed in a desiccator together with a second beaker containing 3 M ammonia. After a few days many crystals appeared in a ring-shaped zone about 1 cm wide at some intermediate level on the wall of the first beaker. Thin hexagonal platelets formed in the upper part of the zone and large hexagonal prisms in the lower part. Sometimes needle-shaped crystals of a basic nitrate appeared below the zone of hydroxide crystals. By gradually increasing the initial concentration of the cadmium nitrate solution I obtained single crystals of two more basic nitrates.

The other compounds already mentioned were obtained with the same or a slightly modified technique. Some basic salts, for example, were best obtained when the first beaker was filled with a single homogeneous solution. No systematic attempts were made to establish optimal growing conditions for any of the crystals.

In contrast to most other diffusion methods, this process requires only a few days' time and can be carried out in a small beaker. There is no need for taking rigorous precautions against shocks and vibrations, temperature fluctuations, etc. It would seem that this is due to the use of ammonia, which is a weak base and which, furthermore, forms complex ions with the metal ions. The complex formation causes an apparent increase in the solubility of the products. This increase ultimately led

to the dissolution of all crystals already formed if the process was not stopped in time.

Hydroxides were obtained from solutions of the nitrates but not from other salt solutions. For cadmium this may be traced to the fact that the concentration of the nitrate solution, which is in equilibrium with both the hydroxide and the basic salt, is relatively high<sup>3</sup>.

I have determined the lattice parameters of all compounds obtained and the structures of some of them are being examined. Although several of the basic salts seem to be new, others were obtained and examined before by Feitknecht<sup>2,3</sup> in his work on basic salts.

This method may also apply to hydroxides and basic salts of such metals as lead, zinc, copper and certain trivalent metals.

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### Superlattice Structure in Ga<sub>2</sub>Te<sub>3</sub>

MANY  $A^{II}_2B^{VI}_3$  compounds crystallize in structures based on zincblende or wurtzite. Because of the unequal atomic concentrations of the components, some lattice sites which are occupied in zincblende and wurtzite are vacant in these compounds. The original work on these compounds showed only crystal structures with a random distribution of the vacancies<sup>1,2</sup>. Later, superlattice structures were observed for Ga<sub>2</sub>S<sub>3</sub> (refs. 3 and 4), In<sub>2</sub>Te<sub>3</sub> (ref. 5), and In<sub>2</sub>Se<sub>3</sub> (ref. 6). No superlattice for Ga<sub>2</sub>Te<sub>3</sub> has previously been reported.

In order to be able to observe a superlattice in Ga<sub>2</sub>Te<sub>3</sub>, it was decided to form the compound at as low a temperature as practicable. Reference to the phase diagram<sup>7</sup> shows a compound GaTe<sub>3</sub>, with a peritectic decomposition at 429° C. A mixture of 77.5 at. per cent tellurium 22.5 at. per cent gallium was heated to 450° C (at which temperature it is liquid) in a sealed, evacuated, silica tube. The temperature at one end of the tube was then lowered by 5°-10° C for 5 days, and tellurium distilled off. X-ray powder patterns from the residue were obtained in an 11.46 cm diameter Debye-Scherrer camera, using copper K $\alpha$ -radiation.

Seven lines were found which could not be indexed on the zincblende unit cell, containing 4/3 molecules of Ga<sub>2</sub>Te<sub>3</sub> with random vacancies. If one of these is ascribed to the strong line of tellurium, the remainder can be indexed on the basis of an orthorhombic unit cell, with the following lattice constants:  $a = 4.17$  Å;  $b = 23.60$  Å;  $c = 12.5_2$  Å. If the lattice constant of the zincblende cell is written as  $a_0$ , then these are related to  $a_0$  as follows:  $a = a_0/\sqrt{2}$   $b = 4a_0$   $c = 3a_0/\sqrt{2}$ . By comparison with the zincblende cell, the orthorhombic unit cell contains 8 molecules of Ga<sub>2</sub>Te<sub>3</sub>. From the occurrence of a (070) reflexion, the space group must be primitive. Assuming that the point group is a sub-group of  $\bar{4}3m$  (the point group of zincblende) leaves 8 space groups, from which no further selection can be made.

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