beneath the cover glass to reduce the possibility of breaking it). Bakelite powder (<200 mesh) is added, first gently to cover the particle and grid, then to fill the mold; and the bakelite plug is formed in the standard manner.

The bakelite plug is removed from the press and the cover glass removed from the surface of the plug. The particle is then in an accurately specified position marked by the electron microscope grid which has been molded into the plug. The particle, which had rested on the surface of the glass, now is just below the surface of the bakelite. The grid may be removed in order to eliminate stray light reflections; however, its impression will remain in the bakelite. Careful grinding and polishing using emery papers and diamond laps complete the process. The sample can then be analysed with the electron microprobe after a conducting surface, usually carbon, has been evaporated on the surface.

This technique has been applied to the preparation of meteoritic spherules from deep sea sediments ranging from $30\mu$ to $8\mu$ in diameter. It is possible to prepare even smaller particles, but the probability of losing particles increases. If more than half a spherule has been ground away, the remainder generally will pluck out of the bakelite and be lost. Epoxy resins may provide firmer binding for particles, but bakelite is a more convenient material for electron microprobe work as it is not rapidly disintegrated by an electron beam.

Parts of the technique described here have been used previously by various workers. We are indebted to our colleagues, E. J. Dwornik, M. H. Carr, and G. A. Desborough of the U. S. Geological Survey for suggested improvements in the mounting and polishing procedures.

## MAGNESIUM ALUMINUM CARBONATE HYDROXIDE TETRAHYDRATE:

### A DISCUSSION

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A crystal structure has been proposed (Ross and Kodama, 1967) for synthetic $Mg_6Al_2CO_3(OH)_{16}\cdot4H_2O$. This paper was submitted in July 1966, so that its authors were not acquainted with the structure determinations of the two forms of $[Mg_6Fe_2(OH)_{16}][CO_3\cdot4H_2O]$, sjögrenite (hexagonal) and pyroaurite (rhombohedral), which have appeared since then Allmann and Lohse, 1966; Ingram and Taylor, 1967; Allmann, 1968).

Synthetic $[Mg_6Al_2(OH)_{16}][CO_3 \cdot 4H_2O]$ corresponds to the two minerals manasseite and hydrotalcite, which have long been reported to be isostructural with sjögrenite and pyroaurite, respectively. Unless this postulated similarity of structure were proved to be erroneous, the following comments would seem to be in order.

Both in sjögrenite and in pyroaurite all the octahedral sites of the brucite-like layer are randomly occupied by all the cations, so that there is no Fe left to go into the highly disordered interlayer. The same situation should obtain as regards Al in both manasseite and hydrotalcite.

Furthermore it has now been established that the doubling of $a$ and $c$, postulated by Frondel (1941) for the minerals of the pyroaurite series, was not justified. The true cell dimensions are near 3 and 23 Å respectively. The powder pattern of $[Mg_6Al_2(OH)_{16}][CO_3 \cdot 4H_2O]$ published by Ross and Kodama (1967) resembles that of hydrotalcite more than that of manasseite. It can be re-indexed on a rhombohedral lattice, with $a \sim$ 3.06 and $c \sim 23.6$ Å. Note that the two strong lines at about 1.54 and 1.50 Å occur on the patterns of both modifications and can be indexed 110 & 113 in the rhombohedral polymorph or 110 and 112 in the hexagonal polymorph. The new indexing follows:

| hkl | | | $d$(Å) | $I_{rel}$ | $d_{calc.}$ |
|---|---|---|---|---|---|
| 0 | 0 | 3 | 7.84 | 100 | 7.83 |
| 0 | 0 | 6 | 3.90 | 60 | 3.92 |
| 0 | 0 | 9 | 2.60 | 40 | 2.61 |
| 0 | 1 | 2 | | | 2.58 |
| 0 | 1 | 5 | 2.33 | 25 | 2.31 |
| 0 | 1 | 8 | 1.990 | 30 | 1.97 |
| 0 | 0 | 12 | 1.950 | 6 | 1.96 |
| 1 | 1 | 0 | 1.541 | 35 | 1.53 |
| 1 | 1 | 3 | 1.498 | 25 | 1.50 |
| 1 | 1 | 6 | 1.419 | 7 | 1.42 |
| 2 | 0 | 2 | 1.302 | 6 | 1.32 |
| 2 | 0 | 5 | 1.265 | 9 | 1.27 |
| 2 | 0 | 8 ? | 1.172 | 2 | 1.21 |
| 1 | 2 | 2 | 0.994 | 4 | 0.997 |
| 1 | 2 | 5 | 0.976 | 5 | 0.979 |

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## MAGNESIUM ALUMINUM CARBONATE HYDROXIDE TETRAHYDRATE:

### A REPLY

G. J. ROSS AND H. KODAMA, *Soil Research Institute, Department of Agriculture, Ottawa, Ontario, Canada.*

In proposing the one-dimensional structural scheme for the synthetic hydroxycarbonate, $Mg_6Al_2CO_3(OH)_{16} \cdot 4H_2O$, (Ross and Kodama, 1967) we had already considered among others a structure similar to the one recently determined for sjögrenite by Allmann and Lohse (1966). However, we could not revise the structural scheme which had originally been outlined by Feitknecht (1942), since our X-ray intensity data appeared to be more consistent with the proposed structural scheme as shown by a comparison in the following table.

| 00*l* | *I* obs. | $(Mg_6)(OH)_{16}[Al_2CO_3(H_2O)_4]$ | $(Mg_6Al_2)(OH)_{16}[CO_3(H_2O)_4]$ |
|:---:|:---:|:---:|:---:|
| | | *I* calc. | *I* calc. |
| 1 | 100 | 100 | 100 |
| 2 | 60 | 53 | 24 |
| 3 | 40 | 23 | 4 |
| 4 | 6 | 2 | 1 |
| 5 | — | 0.03 | 0.3 |
| 6 | 6 | 7 | 3 |
| 7 | — | 1 | 0.9 |
| 8 | 5 | 3 | 1.3 |

The comparison shows that the observed intensities agree better with the calculated intensities of a structure in which the Al is present in the disordered interlayer (column 3) than with the calculated intensities of a structure in which no Al is present in this interlayer (column 4). The agreement between the observed intensities and the calculated intensities of the latter structure is only slightly improved if the powder pattern is indexed on a rhombohedral lattice (Table shown in previous discussion). In this case the enhancement of the intensity of the third order reflection may be explained by the contribution of the prismatic reflection.

Because of the microcrystallinity of our compound we could use only X-ray powder diffraction methods. We are aware of the limitations of