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## The crystal structure of amesite.

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Crystals of amesite $(Mg_2Al)(SiAl)O_5(OH)_4$ from Saranovskaye, U.S.S.R. (specimen BM. 1940. 77) have been found to possess a disordered form of a structure not previously reported. A small fragment was found, which, though twinned, was sufficiently ordered to permit the structure to be determined.

Oscillation and Weissenberg photographs taken with Cu $K\alpha$ radiation showed that the unit cell was very nearly rhombohedral, the related hexagonal cell having dimensions $a=5{\cdot}31$, $c=42{\cdot}1$ Å. Doubled spots due to twinning indicated that the hexagonal $c$-axis was not quite perpendicular to (001), the tilt being approximately 30′ in the (100) plane. The structure has triclinic symmetry, but approximates closely to a structure having rhombohedral symmetry $R3$ or $R\bar{3}$. The analysis has been carried out using the idealized rhombohedral space group, which shows clearly how the kaolin-type layers of amesite are stacked together.

The cell contains six amesite layers, one of which is shown in Fig. 1, and it will be assumed that they are stacked together to form a polar structure. In a structure of space group $R3$, 3-fold screw axes are perpendicular to the layers, so that the 6-layer structure can be regarded as being formed by the operation of a 3-fold screw axis on a unit of 2 layers, the 2-layer unit itself having 3-fold symmetry about the cell origin. The possible structures may therefore be found by considering first the ways in which two layers can be combined to form a unit with a 3-fold symmetry, and then considering the structures which are formed by the operation of a 3-fold screw axis on this unit.

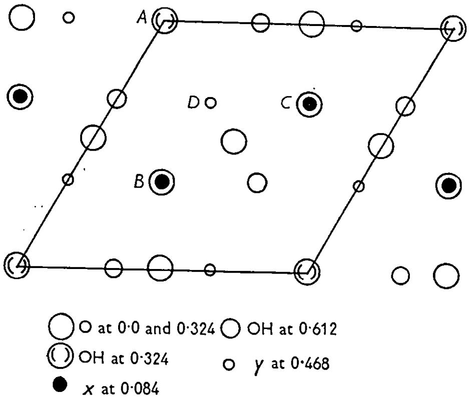

Fig. 1. Projection of one amesite layer on (001). The $z$ coordinates given are fractions of the height of one amesite layer. $X$ represents $\frac{1}{2}$(Si+Al), and $Y$ represents $\frac{1}{3}$(2Mg+Al).

In the amesite layer are three different points of 3-fold symmetry, labelled $A$, $B$ and $C$, in Fig. 1. Let $A_1B_1C_1$ be these points in one layer, and $A_2B_2C_2$ be the same points in the layer to be placed above it. Three types of double layer can be obtained, with the correct interlayer O–OH bonds, by placing the layers together, similarly orientated, with $A_2$ on $A_1$, $A_2$ on $B_1$, or $A_2$ on $C_1$, and a further three by rotating the upper layer of each of these through 60° about $A_2$. These six combinations will be referred to as $a$, $b$, $c$, $d$, $e$ and $f$.

Now consider Fig. 1 to represent the lower of the two layers in anyone of them. A 3-fold axis through the atom $D$, operating on the combination, will produce a structure with rhombohedral symmetry.

The structures formed from $a$ and $b$ give systematic absences, in addition to those required by the space group $R3$, which are not observed with the present crystal. The structure formed from $c$ is a 3-layer structure, while that from $f$ has space group $R3c$. None of these need be considered further.

Table 1. *Fractional atomic coordinates referred to the equivalent positions in Int. Tab. for the hexagonal unit cell*

| | $x$ | $y$ | $z$ |
|---|---|---|---|
| $OH_1$ | 0·667 | 0 | 0·269 |
| $OH_2$ | 0·333 | 0 | 0·102 |
| $OH_3$ | 0 | 0 | 0·221 |
| $OH_4$ | 0 | 0 | 0·054 |
| $Y_1$ | 0·333 | 0 | 0·245 |
| $Y_2$ | 0·667 | 0 | 0·078 |
| $O_1$ | 0 | 0 | 0·554 |
| $O_2$ | 0 | 0 | 0·887 |
| $O_3$ | 0 | 0 | 0·387 |
| $O_4$ | 0 | 0 | 0·721 |
| $O_5$ | 0·500 | 0 | 0·167 |
| | 0·550 | 0 | 0·167 |
| $O_6$ | 0·500 | 0 | 0 |
| | 0·450 | 0 | 0 |
| $X_1$ | 0 | 0 | 0·514 |
| $X_2$ | 0 | 0 | 0·847 |
| $X_3$ | 0 | 0 | 0·347 |
| $X_4$ | 0 | 0 | 0·681 |

$X$ and $Y$ represent $\frac{1}{2}$(Si+Al) and $\frac{1}{3}$(2Mg+Al) respectively.

Structures formed from $d$ and $e$ are enantiomorphous, with space group $R3$. Atomic co-ordinates for structure $d$ in terms of the hexagonal cell outlined in Fig. 1, containing six amesite layers, are given in Table 1, the values of $z$ being derived from those given by Steinfink & Brunton (1956). The two sets of co-ordinates for basal oxygens $O_5$ and $O_6$ are for an ideal kaolin-type layer and for one in which the tetrahedra are rotated through approximately 10°. Tetrahedra in successive layers, such as those containing $X_1$ and $X_3$, rotate in opposite directions. The disagreement factor $R$ for various groups of reflexions for the ideal and modified structures are given in Table 3. The necessity for rotating the tetrahedra was found by examination of the series $12l$, $21l$, $13l$ and $31l$, and it is in these series that the improvement caused by rotation is most marked, the principal zones being relatively unaffected. Table 2 gives the $F$ values for the modified structure, with some for the ideal structure in parenthesis.

The value of 10° was obtained by adjusting the coordinates of $O_5$ and $O_6$ to obtain the smallest $R$ value for reflexions with $h-k\neq 3n$, these being most sensitive

Table 2. *Observed and calculated structure factors*

| *hkil* | $F_o$ | $F_c$ | |
|---|---|---|---|
| 0,0,0,6 | > 160 | 179 | |
| 12 | > 160 | 302 | |
| 18 | 154 | 153 | |
| 24 | 103 | 79 | |
| 30 | 110 | 150 | |
| 36 | 139 | 152 | |
| 42 | 146 | 131 | |
| 48 | 135 | 149 | |
| $0,1,\bar{1},2$ | 49 | 54 | |
| 5 | 76 | 89 | |
| 8 | 47 | 49 | |
| 11 | 66 | 73 | |
| 14 | 37 | 36 | |
| 17 | 45 | 53 | |
| 20 | 27 | 27 | |
| 23 | 30 | 38 | |
| 26 | < 15 | 22 | |
| 29 | 21 | 26 | |
| $\bar{1}$ | 79 | 93 | |
| $\bar{4}$ | 41 | 53 | |
| $\bar{7}$ | 57 | 85 | |
| $\overline{10}$ | 37 | 46 | |
| $\overline{13}$ | 49 | 66 | |
| $\overline{16}$ | 26 | 31 | |
| $\overline{19}$ | 31 | 46 | |
| $\overline{22}$ | < 24 | 22 | |
| $\overline{25}$ | 24 | 31 | |
| $0,2,\bar{2},1$ | 44 | 23 | |
| 4 | 29 | 16 | |
| 7 | 43 | 39 | |
| 10 | 39 | 27 | |
| 13 | 57 | 55 | |
| 16 | 31 | 34 | |
| 19 | 52 | 64 | |
| 22 | 28 | 34 | |
| 25 | 36 | 63 | |
| 28 | < 30 | 35 | |
| 31 | 30 | 59 | |
| $0,2,\bar{2},\bar{2}$ | 29 | 14 | |
| $\bar{5}$ | 40 | 32 | |
| $\bar{8}$ | < 30 | 23 | |
| $\overline{11}$ | 60 | 50 | |
| $\overline{14}$ | 38 | 33 | |
| $0,2,\bar{2},\overline{17}$ | 45 | 64 | |
| $\overline{20}$ | 30 | 34 | |
| $\overline{23}$ | 50 | 67 | |
| $\overline{26}$ | 23 | 37 | |
| $\overline{29}$ | 46 | 63 | |
| $0,3,\bar{3},0$ | > 170 | 329 | |
| 6 | 141 | 144 | |
| 12 | 152 | 125 | |
| 18 | 101 | 97 | |
| 24 | 57 | 43 | |
| 30 | 110 | 144 | |
| 36 | 71 | 113 | |
| 42 | 98 | 109 | |
| $1,1,\bar{2},0$ | 57 | 50 | |
| 3 | 118 | 47 | |
| 6 | 170 | 210 | |
| 9 | 116 | 174 | |
| 12 | 116 | 106 | |
| 15 | 187 | 153 | |
| 18 | 114 | 109 | |
| 21 | 144 | 183 | |
| 24 | 157 | 194 | |
| 27 | 154 | 118 | |
| 30 | 110 | 115 | |
| 33 | 85 | 95 | |
| 36 | 82 | 77 | |
| 39 | 76 | 44 | |
| $2,2,\bar{4},0$ | 76 | 81 | |
| 3 | 92 | 59 | |
| 6 | 168 | 170 | |
| 9 | 106 | 96 | |
| 12 | 67 | 31 | |
| 15 | 123 | 115 | |
| 18 | 105 | 104 | |
| 21 | 106 | 115 | |
| 29 | 125 | 122 | |
| 27 | 97 | 94 | |
| 30 | 53 | 55 | |
| $2,1,\bar{3},1$ | 86 | 68 | (61) |
| 4 | < 20 | 12 | (35) |
| 7 | 64 | 63 | (57) |
| 10 | 40 | 18 | (31) |
| 13 | 58 | 51 | (47) |
| $2,1,\bar{3},16$ | 29 | 21 | (24) |
| 19 | 42 | 38 | (36) |
| $2,1,\bar{3},\bar{2}$ | < 20 | 11 | (35) |
| $\bar{5}$ | 69 | 66 | (59) |
| $\bar{8}$ | < 20 | 16 | (33) |
| $\overline{11}$ | 59 | 56 | (51) |
| $\overline{14}$ | 36 | 20 | (27) |
| $\overline{17}$ | 44 | 43 | (41) |
| $3,1,\bar{4},2$ | < 20 | 3 | (27) |
| 5 | 71 | 63 | (45) |
| 8 | < 20 | 13 | (26) |
| 11 | 52 | 54 | (40) |
| 14 | < 20 | 19 | (21) |
| 17 | 49 | 42 | (33) |
| $3,1,\bar{4},\bar{1}$ | 76 | 64 | (46) |
| $\bar{4}$ | < 20 | 6 | (26) |
| $\bar{7}$ | 69 | 61 | (44) |
| $\overline{10}$ | < 20 | 15 | (24) |
| $\overline{13}$ | 61 | 51 | (38) |
| $1,2,\bar{3},2$ | 79 | 54 | (35) |
| 5 | 42 | 43 | (59) |
| 8 | 43 | 49 | (33) |
| 11 | 40 | 40 | (51) |
| 14 | 42 | 39 | (27) |
| $1,2,\bar{3},\bar{1}$ | 43 | 43 | (61) |
| $\bar{4}$ | 56 | 53 | (35) |
| $\bar{7}$ | 47 | 42 | (57) |
| $\overline{10}$ | 47 | 46 | (31) |
| $\overline{13}$ | 41 | 39 | (47) |
| $\overline{16}$ | 41 | 34 | (24) |
| $\overline{19}$ | 26 | 35 | (36) |
| $\overline{22}$ | 28 | 24 | (18) |
| $1,3,\bar{4},1$ | 44 | 33 | (46) |
| 4 | 64 | 55 | (26) |
| 7 | 27 | 33 | (44) |
| 10 | 43 | 49 | (24) |
| $\bar{2}$ | 76 | 55 | (27) |
| $\bar{5}$ | 44 | 34 | (45) |
| $\bar{8}$ | 54 | 51 | (26) |
| $\overline{11}$ | 27 | 33 | (40) |
| $\overline{14}$ | 46 | 42 | (21) |

Table 3. *Disagreement factors*

| | Ideal structure | Modified structure |
|---|---|---|
| $h0\bar{h}l$ zone | 0·22 | 0·21 |
| $hh\overline{2h}l$ zone | 0·15 | 0·17 |
| $12l13l$ | 0·40 | 0·15 |
| $21l31l$ | 0·20 | 0·14 |
| Overall | 0·21 | 0·18 |

to this movement. Rotation of the tetrahedra in the opposite direction caused $R$ to increase. It is permissible to rotate the tetrahedra containing $X_1$ and $X_3$ in the same sense, but this did not reduce the value of $R$.

The rotations of the tetrahedra in this structure cause all the interlayer O–OH bonds to be longer than they would be between ideal layers. The same effect was found by Steinfink & Brunton in their amesite structure. Until a detailed determination is carried out, regarding the structure as a 2-layer triclinic one, no conclusions should be drawn concerning the relation between these rotations and the crystal structure.

The stacking sequence occurring in this structure may be described concisely in terms of the vectors used by Oughton (1957). Using this terminology, the rotation vectors $\mathbf{S}_1$ or $\mathbf{S}_2$ operate on all layers, and the translational vectors $\mathbf{S}_9$ and $\mathbf{S}_4$ operate on alternate layers.

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## References

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