Electron-optical data for crystals of scarbroite.[1]

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Summary. Electron micrographs of scarbroite show thin platy crystals of about 1 μ size, having rhombic outlines with angles $66° \pm 1°$ and $113° \pm 1°$. Single-crystal electron-diffraction patterns show rectangular net patterns, with $d_{100} = 9{\cdot}90$ Å., $d_{010} = 14{\cdot}67$ Å., $\gamma^* = 90° \pm 0{\cdot}05°$. Strong $hk0$ reflections show a pseudohexagonal arrangement, but true symmetry is probably orthorhombic or monoclinic. Faces outlining rhombic forms are of type $\{11l\}$, $\{1\bar{1}l\}$.

A SAMPLE of scarbroite was made available by Drs. Goodyear and Duffin for examination by electron microscope and diffraction methods. With the electron microscope at present available to the writers (R.C.A. model: E.M.U. type 2D, fitted with variable diaphragm for selected area electron diffraction) it was possible to examine fine dispersions of scarbroite and to take single-crystal diffraction patterns for the preferred orientation taken up by the crystals on the stage of the microscope.

Scarbroite is readily dispersed in water and weak suspensions dried on collodion films show clearly the morphology of the individual crystals. Fig. 1 is a typical electron micrograph. The crystals are in the form of thin plates of uniform thickness, with well-defined rhombic outlines, and are about 1 μ in size. The crystals vary in thickness from one to another and a rough estimate of the order of the thickness is 0·01–0·05 μ.

The thinnest crystals have a mottled appearance and the edges sometimes exhibit a rim. The corners of most crystals are not geometrically sharp. These observations suggest that the crystals may be unstable under the conditions existing in the microscope and this would not be surprising in the light of the dehydration data discussed by Goodyear and Duffin in the preceding paper.

The outlines of the crystals are sufficiently sharp and linear to permit the angles between the edges to be measured to within $\pm 1°$. The mean values obtained for eleven crystals are $66° \pm 1°$ and $113° \pm 1°$, the sum

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of which is 179°±2°. These angles are significantly different from 60° and 120° and do not support the tentative hexagonal unit cell suggested by Duffin and Goodyear (1957).

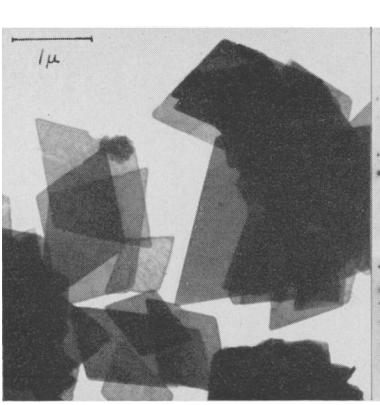

FIG. 1.

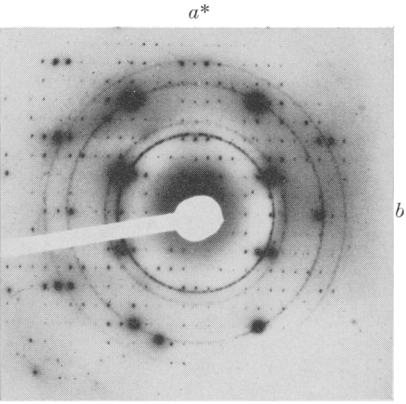

FIG. 2.

FIG. 1. Electron micrograph of scarbroite.

FIG. 2. Single crystal, electron diffraction pattern of scarbroite, with a superimposed ring pattern of aluminium for calibration.

Using thin dispersions, single crystals could readily be selected in the field of the microscope for single-crystal diffraction study. A spot pattern of the type shown in fig. 2 is easily obtained from the thinnest platelets. The pattern shows a rectangular array suggesting an orthorhombic or monoclinic unit cell. The diffraction patterns, when calibrated with respect to MgO and NaCl powder patterns (ring diagrams) taken independently but with identical instrument adjustments, gave parameter variations as large as 1–2 %. More consistent results were obtained by 'shadowing' the scarbroite lightly with aluminium, so that a combined spot pattern with superimposed Al calibration was obtained (see fig. 2). Ring diameters were found to vary by as much as 1–2 % in a single pattern, presumably due to some instrumental defects. Calibrations were made parallel to the principal axes of the rectangular spot patterns and results were then obtained for d_{100} and d_{010} that appear to be reliable to about 0·3 %. The more widely spaced and the less widely spaced point rows were labelled respectively the a^* and b^* axes and the following results were obtained: d_{100} 9·90±0·02 Å., d_{010} 14·67±0·04 Å., d_{010}/d_{100} 1·482±0·007, γ^* 90°±0·5°.

The pseudo-hexagonal character of the strongest reflections is readily

seen in fig. 2, and it is apparent why Duffin and Goodyear came to select an hexagonal cell from X-ray powder data.

The crystal morphology and the diffraction data can be correlated as follows: The diffraction patterns suggest that the *c*-axis, [001], lies parallel or nearly parallel to the electron beam. It follows that planes of type (*hk*0) also have this orientation, and hence the observed angle between the edges of the plates, as seen in the micrographs, will be equal to some interfacial angle $(h_1k_1 0):(h_2k_2 0)$ even if the bounding facets are in fact (*hkl*) planes and oblique to the *c*-axis. And since

$$(100):(010) = 90°,$$

$$(100):(1\bar{1}0) = (100):(110) = \tan^{-1}(d_{100}/d_{010}) = 34{\cdot}0°,$$

making $(110):(1\bar{1}0)$ 68·0°, close to the measured value of $66° \pm 1°$. We conclude that the plates are bounded by faces of the forms $\{11l\}$, $\{1\bar{1}l\}$, where l may be zero.

References.

DUFFIN (W. J.) and GOODYEAR (J.), 1957. Nature, vol. 180, p. 977.
—— —— 1960. Min. Mag., vol. 32, p. 353.