This shows that the degree of linkage of the coordination polyhedra is highest in vauxite, lowest in metavauxite. There is no similarity between the crystal structure of vauxite and the structures of the other two minerals of the vauxite group. This is interesting in view of the fact that all three minerals occur together as secondary minerals.

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ELECTRON DIFFRACTION DATA FOR SOME MEMBERS OF THE METATORBERNITE GROUP

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INTRODUCTION

The metatorbernite group of minerals is characterized by the formula A^z(UO$_2$XO$_4$)$_z$·nH$_2$O, where A may be almost any monovalent or divalent cation, and X may be P or As. These minerals have received a great deal of study, but there are still problems concerning the crystallography of some members of the group. Different authors arrive at different results in the determination of unit cell and space group (Alver and Sellevol, 1957; Donnay and Donnay, 1955; Nuffield and Milne, 1953; Ross, 1963; Ross, Evans and Appleman, 1964; Ross and Evans, 1965; Takano, 1961; Volborth, 1959).

Up to 1955 a tetragonal cell with $a = 6.9$ Å was assigned to the minerals of the group. Weak reflections showing that, for some members of the group, the cell with $a = 6.9$ Å was a pseudo cell were observed later by different authors. Donnay (1955) for instance, using the integrating precession technique, found for uranocircite a tetragonal cell with $a = 9.87$ Å.

To our knowledge, published data have been obtained exclusively by

X-ray diffraction methods. At our laboratory an electron diffraction study of synthetic samples of several members of the arsenate group has been started. It was expected that electron diffraction techniques would furnish a more reliable and sensitive method of study, permitting the recording of weak reflections not easily observed by X-ray diffraction. As stated by Ross (1963), the true cell and symmetry can be deduced only by taking into account the very weak reflections; the light oxygen atoms determine the principles of coordination, bonding and packing in these structures; the uranium atoms, usually occupying special positions, contribute scattering intensity only to certain classes of reflections corresponding to a pseudo cell.

METHOD

The compounds were obtained as thin {001} platelets, too small for X-ray single crystal work, but suitable for transmission electron diffraction; they were quantitatively analysed, and identified by their X-ray powder patterns.
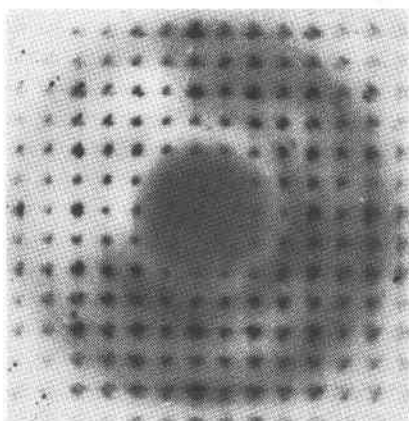
Electron diffraction patterns were taken in a Metropolitan Vickers E.D. 2 chamber, and in a Philips 100 A electron microscope. As tilting was only possible around the axis of the sample holder, our observations were limited to the $hk0$ net of the reciprocal lattice.

The state of hydration of the samples under the conditions prevailing in the electron microscope is not precisely known. The water of hydration is situated between structural layers which lie parallel to the (001) micaceous cleavage. We believe that its content does not affect the reflections observable on our selected area diffraction (SAD) patterns. Results derived from these patterns have been correlated with published X-ray diffraction data for the corresponding "metahydrates;" when space groups are suggested, the results obtained by different authors from reflections other than $hk0$ have been taken into account.
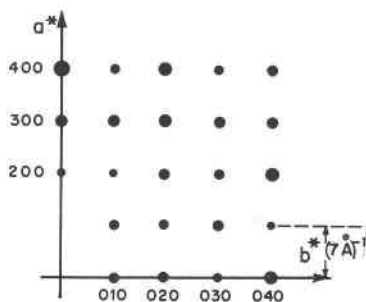
METAZEUNERITE: $Cu(UO_2AsO_4)_2 . 8H_2O$

From our S.A.D. patterns a value of 6.9 Å is obtained for the a cell edge of metazeunerite, agreeing with the results given by Donnay (1965) and by Ross, Evans and Appleman (1964). The space group given by Donnay is $P4/nmm$; that given by Ross *et al.* is $P4/n$ (probable). For both groups, the condition limiting the presence of possible $hk0$ reflections is $h+k=2n$. In all our SAD patterns reflections with indices such that $h+k=2n+1$, are observed (Fig. 1).

The crystals of metazeunerite being very thin in the c direction these spots might belong to the $hk1$ reciprocal net. If this is true space groups symmetry, $P4/nmm$ or $P/4n$ is possible. But the fact that the spots are

(a)

(b)

Fɪɢ. 1. Metazeunerite, $Cu(UO_2AsO_4)_2 \cdot 8H_2O$. (a) selected area electron diffraction pattern, (b) idealized pattern.

observed throughout the whole pattern and their intensities are not enhanced when the sample is tilted about [100], suggests that they belong to the $hk0$ net. If they do, no conditions limit the presence of the $hk0$ reflections, and indicate the absence of an n glide.
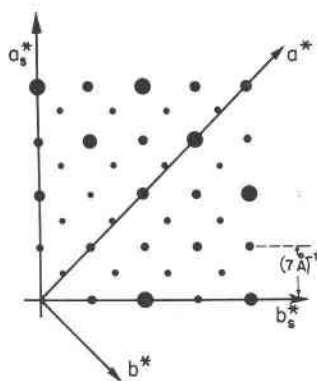
Metaheinrichite: $Ba(UO_2AsO_4)_2 \cdot 8H_2O$

Gross *et al.*, (1958) gave for metaheinrichite a tetragonal unit cell with $a = 7.07$ Å and no conditions limiting the presence of $hk0$ reflections. In most of out SAD patterns, besides $hk0$ reflections belonging to that cell, weaker spots appear that cannot be indexed on the basis of a cell with $a = b = 7.07$ Å (Fig. 2). If the weak spots belong to the $hk0$ reciprocal net, two unit cells can be chosen

    1) $a = b = 9.97$ Å; $hk0$: no conditions. Any of the space groups proposed by Gross and Corey: ($P4_2$, $P4_2/m$, $P4_222$), is possible.
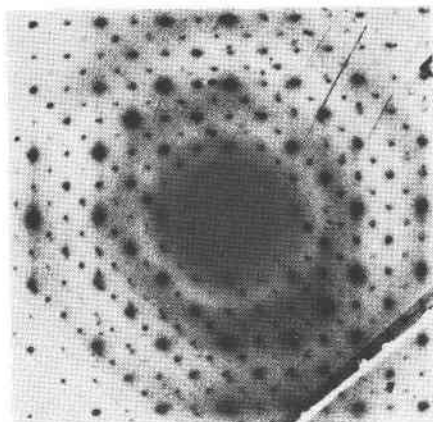
(a)

(b)

FIG. 2. Metaheinrichite, $Ba(UO_2AsO_4)_2 \cdot 8H_2O$: (a) selected area electron diffraction pattern, (b) idealized patterns. The subscript (s) refers to the axis of the pseudo cell with $a = b = 7.07$ Å.

2) $a = 14.1$ Å; $hk0: h+k = 2n$. Examples of possible diffraction symbols are: $4/mP-/n$ or $4/mmmP-/n--$.

If the weak spots belong to the $hk1$ reciprocal net, possible unit cells are:

1) a tetragonal cell with $a = 9.97$ Å; $hk0: h+k = 2n$. Possible diffraction symbols are: $4/mP-/n$ or $4/mmmP-/n---$.

2) a cell with orthorhombic symmetry and $a \simeq b \simeq 14.1$ Å; $hk0: h = 2n$, $k = 2n$; $hkl: h+k = 2n$. Possible space groups: $Cmca$, $Cmma$, $Ccca$.

The possibility of an orthorhombic cell must not be over looked. For metauranocircite, the phosphate analogue of metaheinrichite, Donnay (1955) reported a tetragonal cell with $a = 9.87$ Å, Pseudo space group:
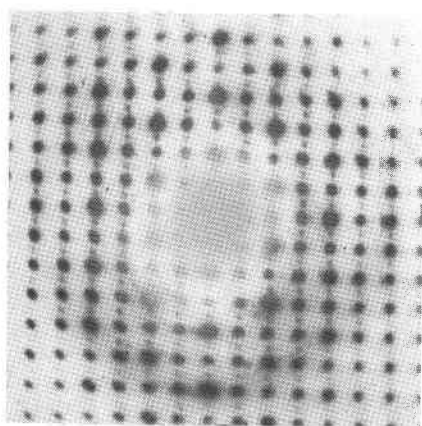
(a)
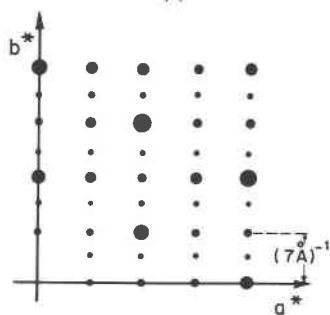
(b)

FIG. 3. Metanovaceckite, $Mg(UO_2AsO_4)_2 \cdot 4H_2O$: (a) selected area electron diffraction pattern, (b) idealized pattern.

$P4/nmm$, but Ross and Evans (1965) found the cell of metaurano circita (I) to be orthorhombic with $a = 9.81$; $b = 9.91$ Å. Our patterns would not detect differences of 0.1 Å.

METANOVACECKITE: $Mg(UO_2AsO_2)2 \cdot 4H_2O$

Donnay and Donnay (1955) reported for metanovaceckite a unit cell with $a = 7.12$ Å, space group $P4/n$. They suggest that weak reflections may have been overlooked, and that the cell they found may be really a pseudocell.

In our SAD patterns, in addition to the intense spots defining a cell with $a = 7.0$ Å, weaker spots appear showing that the symmetry of the crystals must be lower than tetragonal (Fig. 3).

If the weak spots belong to the $hk0$ reciprocal net, two orthorhombic unit cells may be chosen:

1) $a=7.0$ Å; $b=14.0$ Å. No conditions limit the presence of $hk0$ reflections, which implies the absence of an n glide plane normal to the c axis, and of screw axes parallel to a or b.

2) $a=14.0$ Å; $b=14.0$ Å. The condition limiting the presence of possible $hk0$ reflections, $h=2n$, implies an a glide plane normal to c.

If the weak spots belong to the $hk1$ reciprocal net, the cell has

$a=7.0$ Å; $b=14.0$ Å. The limiting condition for the presence of possible $hk0$ reflections, $k=2n$, implies a b glide plane. For every set of conditions, several orthorhombic space groups are possible.

These few results point to the convenience of a more complete investigation of the minerals of this group by electron diffraction methods.

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LOW-ANGLE X-RAY DIFFRACTION MAXIMUM DUE TO TEXTURAL PERIODICITY IN HEATED GIBBSITE

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Oriented specimens of powdered gibbsite (from Minas Gerais, Brazil), <1 micron, were heated for two hours at 300°, 400° and 500°C and scanned on a conventional Philips X-ray diffractometer with CuKα radiation. At each temperature only one X-ray diffraction maximum occurred. The diffraction angle decreased slightly with increasing temperature. The maxima were reproducible. At 500°C the maximum was the most pronounced and was situated between 1.5 and 2°2θ, corresponding to about 50 Å. See Figure 1.