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On the crystallography of calclacite, $Ca(CH_3COO)Cl.5\,H_2O$.

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The mineral name calclacite has been given, by the author, to a silky, acicular efflorescence, occurring on fossils and limestones in wooden museum cases. The chemical composition corresponds to $Ca(CH_3COO)Cl.5\,H_2O$. The needles show a thickness of only 0·03–0·16 mm. and occasionally reach 4 cm. in length. The substance has also been observed on pottery sherds (Frondel, 1951; Gettens, 1957).

The 'natural' efflorescence is, as shown by its X-ray diffraction powder pattern, identical with the synthetic monoclinic calcium acetate chloride pentahydrate, described by Handl (1861) and Rammelsberg (1882), but is crystallographically different from the salt of same composition, previously prepared and examined by the author. Identity between the former substances was established by comparison with material from a large ($4\frac{1}{2}$ g.), {010} flattened crystal, labelled 'essigsaurer Kalk–Chlorcalcium', prepared, before 1880, by von Foullon in von Hauer's laboratory, Vienna. The availability of well developed synthetic calclacite crystals, 2 to 3 mm. long and 0·5 mm. thick, permitted further investigation. Miss E. West, Freer Gallery of Art, Washington, prepared them starting from an equimolecular solution of $CaCl_2$ and $Ca(CH_3COO)_2$ evaporated at room temperature.

Optical goniometer measurements revealed the forms {001}, {110}, {120}, {130}, {010} and {210}. The last form, observed on four faces, is new for the substance. For the obtuse angle β the value of 116° 42′ (mean of 5 measurements) was found, which is in good agreement with 116° 49′ (Handl, 1861) and 116° 59′ (Rammelsberg, 1882). The elongation direction lies along the c axis. The crystals usually show good (010) faces; the other prism faces are narrow and, as well as the top face, of rather poor quality for goniometer readings.

X-ray rotation photographs round the three crystal axes, using the morphological angle β for the a-axis, were taken. Weissenberg photographs were obtained with the elongation direction c as rotation axis, in normal-beam setting for the zero layer and in equi-inclination setting for the first layer. The following absolute parameters were observed:

$$a = 11{\cdot}51,\quad b = 13{\cdot}72,\quad c = 6{\cdot}82\ \text{Å}\,.$$

The monoclinic symmetry and the morphological angle β were confirmed. The ratio $a_0:b_0:c_0$ is 0·839:1:0·496, where $a_0:b_0$ is in good agreement with the values 0·8395 (Handl) and 0·856 (Rammelsberg). The Weissenberg photographs showed no restriction for the hkl reflections, but systematic absences were observed for $h00$ when h is odd and for $0k0$ when k is odd; hence the space group is $P2_1/a$.

Density determinations were based on following specimens:

1) 'natural' efflorescence: $> 1{\cdot}48$, $< 1{\cdot}59$ g.cm.$^{-3}$, measured by flotation (Van Tassel, 1945), 2) large crystal, prepared by von Foullon: 1·487 g.cm.$^{-3}$, measured with hydrostatic balance, 3) crystals, prepared by E. West: 1·52 g.cm.$^{-3}$, measured with Berman's precision torsion balance. The densities provided by the synthetic substances are to be considered as too small, as microscopic examination showed the presence of solution inclusions. The volume of the unit cell is 962 Å^3. Using the experimental density of 1·52, it is found that the number of molecules in the cell is 3·9 (molecular weight $Ca(CH_3COO)Cl.5\,H_2O$ = 224·6). There are thus 4 molecules per unit cell, as required for the general position in $P2_1/a$. The calculated density is 1·55 g.cm.$^{-3}$.

The powder data, obtained with a Debye–Scherrer camera of 5·73 cm. diameter and with filtered Fe-radiation, are given for identification purposes. The reflections satisfy the equation:

$$\sin^2\theta_{Cu} = 0{\cdot}005621h^2 + 0{\cdot}003156k^2 + 0{\cdot}01601l^2 - 0{\cdot}008523hl.$$

The lines in Table 1 have been indexed on this basis.

The optic axial plane is (100), with the c crystallo-

Table 1. *Powder lines*

d (Å)	I	hkl	d (Å)	I	hkl	d (Å)	I	hkl
8·27	*s*	110	3·00	*w*	222	2·04	*m*	510, 003, 052, $41\bar{1}$
6·87	*m*	020	2·94	*vw*	012, $22\bar{1}$	1·907	*vw*	360, $15\bar{2}$
6·15	*w*	001, 111	2·65	*w*	$23\bar{1}$	1·876	*vw*	450, 530, 171, 601
4·86	*vw*	210, 121	2·43	*s*	340	1·842	*w*	270, $10\bar{3}$
4·16	*m*	221, 220	2·30	*m*	060	1·704	*w*	460, 600
3·67	*w*	031	2·22	*w*	$33\bar{1}$	1·560	*vw*	470, $35\bar{2}$
3·24	*s*	112, 212	2·14	*w*	113	1·486	*w*	024
3·06	*w*	002						

(Visually estimated intensities: *s* = strong; *m* = medium; *w* = weak; *vw* = very weak.)

graphic axis parallel to the Z optical direction and with b the obtuse bisectrix. The indices of refraction,

$$\alpha = 1{\cdot}468, \quad \beta = 1{\cdot}484 \quad \text{and} \quad \gamma = 1{\cdot}515$$

and the axial angle $2V_{\text{obs.}} = +80°$, have been previously recorded (Van Tassel, 1945).

Thanks are due to Prof. F. Halla for advice, to Dr R. J. Gettens for providing the crystals prepared by Miss E. West and to Dr M. Van Meerssche for Weissenberg technique facilities.

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The monoclinic modification of gadolinium sesquioxide Gd_2O_3.

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Powder samples of Gd_2O_3 form B were prepared by heating commercially available cubic Gd_2O_3 of 99·9% purity at 1400–1500 °C. for several hours. X-ray diffraction patterns were run at room temperature on a Norelco diffractometer using Cu $K\alpha$-radiation and calibrating with a silicon standard. The patterns suggest that Gd_2O_3 form B is isomorphous with the monoclinic Sm_2O_3 form B reported recently by Douglass & Staritzky (1956) and investigated in more detail by Cromer (1957). The observed powder lines and their relative peak intensities are given in Table 1. The cell dimensions as determined by a least-squares method are:

$$a = 14{\cdot}061 \pm 0{\cdot}013, \quad b = 3{\cdot}566 \pm 0{\cdot}006, \quad c = 8{\cdot}760 \pm 0{\cdot}007 \text{ Å},$$
$$\beta = 100{\cdot}10 \pm 0{\cdot}08°.$$

Table 1. *Powder pattern of monoclinic* Gd_2O_3

hkl	$d_{\text{calc.}}$	$d_{\text{obs.}}$	$I_{\text{rel.}}$
$20\bar{1}$	5·930 Å	5·900 Å	3
$20\bar{2}$	3·987	3·965	4
202	3·402	3·396	10
111	3·158	3·155	70
401	3·033	3·028	60
$40\bar{2}$	2·965	2·961	100
003	2·875	2·868	50
310	2·822	2·820	75
$11\bar{2}$	2·755	2·752	75
600	2·307	2·308	10
$11\bar{3}$	2·258	2·258	5
312	2·253		
$60\bar{2}$	2·201	2·195	20
$51\bar{1}$	2·193		
510	2·187		
$31\bar{3}$	2·131	2·131	45
313	1·914	1·915	40
020	1·783	1·784	25
$80\bar{1}$	1·757	1·759	10
404	1·701	1·700	30
$71\bar{2}$	1·698		
$40\bar{5}$	1·665	1·665	20
603	1·663		
$51\bar{4}$	1·654	1·651	30
022	1·648		
711	1·648		
$11\bar{5}$	1·571	1·571	10
421	1·537	1·539	15
$42\bar{2}$	1·528	1·530	20
115	1·517	1·518	15
802	1·517		
023	1·515		
$60\bar{5}$	1·515		
$80\bar{4}$	1·482	1·483	7

With six formula units per cell the calculated density is 8·33 g.cm.$^{-3}$ which compares satisfactorily with the experimental value of 8·22 g.cm.$^{-3}$ determined by pycnometric measurements.

Goldschmidt, Ulrich & Barth (1925) reported for Sm_2O_3 and Gd_2O_3 the two separate forms B_1 and B_2 in the temperature range of the B-modification. B_1 is described as a low symmetry form (presumably the now identified monoclinic form) whereas B_2 is supposedly a higher symmetry form which was observed in a temperature range somewhat below that of B_1. In order to check this point a powder sample of cubic Gd_2O_3 was heated in a high temperature X-ray camera designed to fit on the Norelco diffractometer and patterns were obtained at successively higher temperatures. The runs taken after six hours of heating at 900 °C. and after additional half-hour steps at about 1050° and 1200 °C. showed only the cubic pattern. Further heating of one hour at about 1400 °C. produced the monoclinic pattern with the cubic lines still strongly present. The next run taken one hour later at about 1600 °C. showed only the monoclinic pattern. These results indicate a direct transition from the cubic to the monoclinic form without any evidence for the existence of B_2. It seems likely, however, that the impurities in the samples investigated and the duration of the heat treatment affect the transition characteristics which may account for the observation of the intermediate form B_2 by Goldschmidt *et al.* (1925).

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