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Orthorhombic diglycine sulphate

We note with interest the recent publication on the growth and properties of monoclinic diglycine sulphate monohydrate [1]. Crystals grown under similar conditions and studied here have proved to be anhydrous diglycine sulphate with an orthorhombic structure, almost certainly the same orthorhombic diglycine sulphate mentioned by Wood and Holden [2]. Large clear crystals could be grown at 30°C from water solutions of glycine and sulphuric acid in 1:1 proportions; the crystals are tabular in habit (Fig. 1) and exhibit perfect cleavage parallel to the $\{010\}$

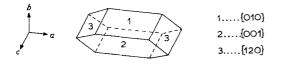


Figure 1 Diglycine sulphate; crystal habit.

face. Proportions of amino acid to sulphuric acid of 2:1 and 3:1 always gave triglycine sulphate (TGS). X-ray powder photographs (Table I) demonstrated that the crystals grown from 1:1 solution differed from TGS.

X-ray examination of the new crystals by Weissenberg techniques showed that they belong to the orthorhombic class, with parameters:

 $a_0 = 10.93$ Å $b_0 = 17.74$ Å $c_0 = 9.88$ Å

Table II compares this material with the other two glycine sulphates that have been described in the literature.

A Gieber-Scheiber test (for piezoelectricity) in powdered samples of the new compound proved negative, suggesting a crystallographic point group of *mmm*.

Chemical analysis of these crystals showed a sulphuric acid content of 39.5% by wt; the calculated sulphuric acid content for

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 $(NH_2CH_2COOH)_2.H_2SO_4$ is 39.52%. The nitrogen content, 11.5% by wt, gives a calculated nitrogen:sulphur atomic ratio of 2.04:1, confirming that the new material is diglycine sulphate. Its measured density (ρ_m) was 1.743, which compares well with the calculated density (ρ_x) (Z = 8) of 1.72 from the X-ray data for $(NH_2CH_2COOH)_2.H_2SO_4$.

Futher studies revealed that, unlike the other two glycine sulphates, anhydrous diglycine sulphate does not show any crystallographic transition between room temperature and its decomposition temperature. Differential thermal analysis indicates decomposition of the compound at temperatures above 150°C.

Dielectric measurements on plates cleaved or cut perpendicular to the crystallographic axes gave dielectric constants of 7.5, 7.0 and 14.5 in

TABLE I Powder data for diglycine sulphate

Intensity	$d_{\rm obs}$
m	9.0
w	6.9
m	5.6
VW	5.45
vw	4.95
8	4.78
8	4.68
m	4.45
ms	4.35
s	3.8
mw	3.73
mw	3.68
s	3.6
S	3.38
s	3.2
vs	2.97
w	2.81
m	2.78
m	2.52
m	2.48
m	2.39

	Triglycine sulphate	Diglycine sulphate monohydrate	Diglycine sulphate
Reference	[2]	[1]	This work
Mol wt	323.3	266.2	248.2
Structure	Monoclinic, P2 ₁	Monoclinic, P2/a	Orthorhombic
a_0	9.15	13.50	10.93Å
b_0	12.69	8.67	17.74Å
c_0	5.73	9.62	9.88Å
β	105° 40′	106° 30′	
Volume	641	1080	1920 ų
Ζ	2	4	8
ρ×	1.68	1.64	1.72
0m	1.69	1.63	1.743
Transition	49°C	72°C	None

TABLE II X-ray data for the glycine sulphates

the *a*, *b* and *c* directions at room temperature. No dielectric anomalies were noted up to 100°C. The values for the dielectric constant at 80°C were respectively 7.5, 8.0 and 16.0 in the three directions. The resistivity of the material at room temperature was measured as 1.4×10^9 ohm cm.

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Note on an etching technique for orientation confirmation in a directionally solidified eutectic

The pseudo-binary eutectic between a nichrome matrix and TaC fibres is being studied to determine fracture mechanisms under monotonic and cyclic loading conditions. This NiCrTaC alloy has been directionally solidified at 0.635 cm/h through a temperature gradient of approximately 80° C/cm to give a good fibrous structure of predominantly square TaC rods in a nichrome matrix. The resulting structure has rods 1.5 to 2.0 µm square with an average inter-rod spacing of 7.5 to 8.0 µm. The aspect ratio of the rods is 10^4 or greater.

Both the matrix and fibres are nominally oriented with the $\langle 100 \rangle$ direction parallel to the

Figure 1 Etched, transverse section of a directionally solidified eutectic showing rod shape and (111) planes in the matrix (\times 3000).

