The Crystal Structure of Iron(II) Sulphate Pentahydrate Glycine

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The bonding between iron and glycine in this compound has never been elucidated, and because of the clinical interest of the compound, AB Pharmacia, Uppsala, has suggested to us to determine the crystal structure so far that the coordination in the compound could be considered as definitely established.

Experimental. Crystals were prepared from a hot concentrated solution of the salt plus an excess of sulphuric acid. The crystallization is best done in an inert atmosphere to prevent oxidation. The carefully dried crystals, which have a pale greenish-blue colour are, however, stable in air.

Roughly prismatic crystals with a cross-section of about 0.02 × 0.02 mm^2 were used for the X-ray work. Weissenberg photographs were taken of three such crystals around the three almost perpendicular triclinic axes. Lattice parameters were obtained from high-order reflections in the equatorial zones. $a = 6.86$ Å, $b = 13.6$ Å, $c = 6.07$ Å, $\alpha = 96.1°$ $\beta = 96.8°$, $\gamma = 92.5°$. Probable space-group is $P\bar{1}$. Assuming two formula units per cell, the calculated density is 1.92 gcm^{-3}, obs. 1.87 gcm^{-3}, measured by a pycnometric method.

Cu-K radiation could be employed, in spite of the iron content. The intensities were estimated visually by comparison with a standard scale.

Crystal structure. The Patterson projections $P(xy)$ and $P(yz)$ were calculated and the Fe and S positions were derived from these projections. The positions of the light atoms were determined by successive electron density and difference maps. The approximate parameters determined in this way are:

O_1	(0.178, 0.055, 0.708)
O_2	(0.104, 0.133, 0.205)
O_3	(0.759, 0.085, 0.958)
O_4	(0.488, 0.123, 0.287)
O_5	(0.770, 0.140, 0.560)
O_6	(0.655, 0.285, 0.403)
O_7	(0.460, 0.205, 0.670)
O_8	(0.169, 0.383, 0.535)
O_9	(0.633, 0.463, 0.681)
O_{10}	(0.169, 0.343, 0.067)
O_{11}	(0.663, 0.472, 0.869)
C_1	(0.023, 0.322, 0.858)
C_2	(0.149, 0.398, 0.737)
N	(0.003, 0.345, 0.100)
S	(0.588, 0.1885, 0.487)
Fe_1	(0,0,0)
Fe_2	($\frac{1}{2}$,$\frac{1}{2}$,0)

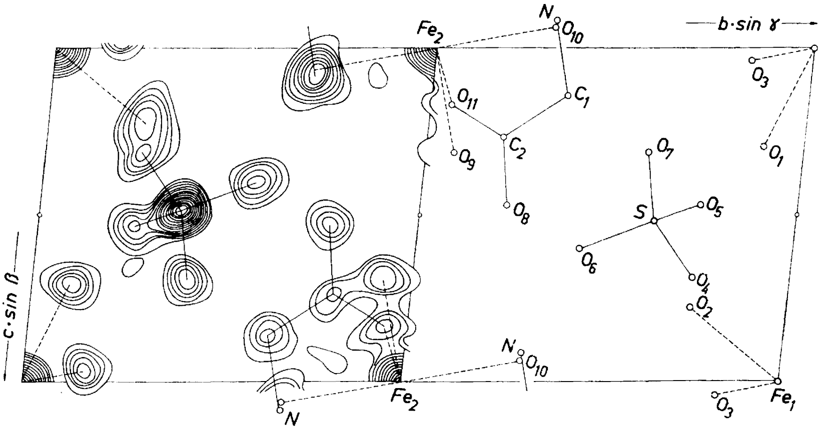

Fig. 1a. Iron (II) sulfate pentahydrate glycine: *a* axis projection, showing the complete unit cell. Electron density contours are drawn for the unique portion to the left and the corresponding atomic positions are indicated to the right across the center of symmetry.

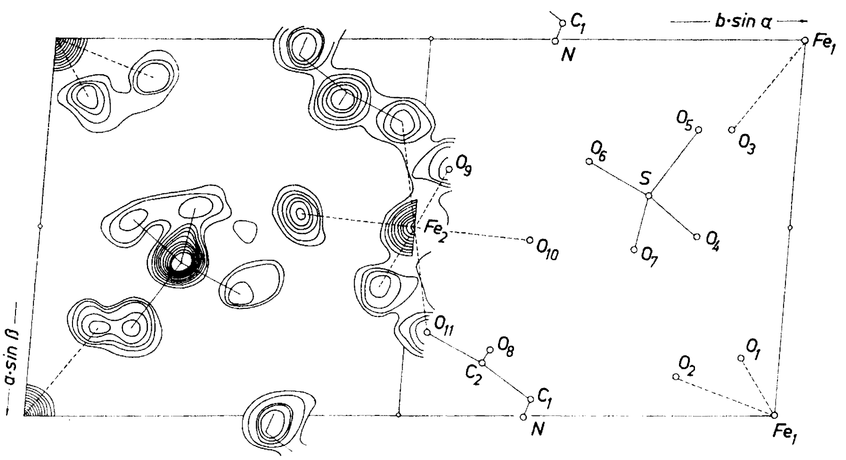

Fig. 1b Iron (II) sulfate pentahydrate glycine: *c* axis projection, showing the complete unit cell. Electron density contours are drawn for the unique portion to the left and the corresponding atomic positions are indicated to the right across the center of symmetry.

Average temperature factors for the two projections, (*B hk*0) : oxygen 3.0, carbon 3.8, nitrogen 3.4, sulfur 1.51, iron 1.62 Å^2, *B* (0*kl*): oxygen 2.5, carbon 3.3, nitrogen 2.9, sulfur 0.89, iron 1.00 Å^2. The residual error indices are *R* (*kh*0) = 0.14, *R* (0*kl*) = 0.18.

The structure is shown together with two electron density maps in Fig. 1*a* and *b*. It is built up of two different kinds of complex cations: $Fe(H_2O)_6^{2+}$ and $Fe(H_2O)_4({}^-OOCCH_2NH_3{}^+)_2^{2+}$. In the latter ion one of the oxygen atoms of the carboxyl group in the glycine »Zwitterion» is bonded to the central Fe^{2+} ion. The two glycine molecules are in trans positions. These complex cations obviously can build up a structure, together with the sulphate ions, with more favorable lattice energy than one containing only one type of cation $Fe(H_2O)_5({}^-OOCCH_2NH_3{}^+)^{2+}$. The accuracy of the structure determination does not permit a detailed discussion of the bond lengths but it is quite clear that a complicated hydrogen bridge system joins the different parts of the structure. The aim with this structure determination has, however, only been to establish the co-ordination around the Fe^{2+} ions and this problem has been definitely solved. Every suggestion about chelate bonding with the amino group can thus be rejected.

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1. H. Flood and V. Lorås, *Tidskr. Kjemi, Bergvesen Met.* **4** (1944) 35.

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A Note on the Structure of Barium Tetraborate

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Crystalline barium tetraborate, $BaO \cdot 4B_2O_3$, was obtained by Levin and McMurdie as a congruently melting phase in the system barium oxide — boron oxide[1]. Single crystals of this phase were prepared during the present study by fusing barium carbonate with the calculated amount of boric acid in a platinum