

## Deformation Density in Complex Anions. IV. Magnesium Thiosulfate Hexahydrate, $\text{MgS}_2\text{O}_3 \cdot 6\text{H}_2\text{O}^*$

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**Abstract.** The deformation electron density in  $\text{MgS}_2\text{O}_3 \cdot 6\text{H}_2\text{O}$  has been determined at 120 K from a combination of X-ray and neutron diffraction data.  $M_r = 244.56$ ,  $Pnma$ ,  $a = 9.324$  (2),  $b = 14.461$  (4),  $c = 6.862$  (1) Å,  $V = 925.2$  (4) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.756$  Mg m<sup>-3</sup>,  $F(000) = 512$ . Neutron diffraction:  $\lambda = 0.8410$  Å,  $(\sin \theta / \lambda)_{\max} = 1.02$  Å<sup>-1</sup>,  $\mu = 0.158$  (1) mm<sup>-1</sup>,  $R_w(F) = 0.047$  for 2771 independent reflections. X-ray diffraction: Nb-filtered Mo  $K\alpha$ ,  $\lambda = 0.71069$  Å,  $(\sin \theta / \lambda)_{\max} = 0.85$  Å<sup>-1</sup>,  $\mu = 0.655$  mm<sup>-1</sup>,  $R_w(F) = 0.026$  for 2489 independent reflections. Peaks of about  $0.25 \text{ e Å}^{-3}$  are observed at the midpoints of the S–O and S–S bonds. Extended lone-pair regions up to  $0.30 \text{ e Å}^{-3}$  are found at the back of the sulfate O atoms. The lone-pair density at the terminal S atom is rather diffuse. All three independent water molecules show O–H bond peaks of  $0.35\text{--}0.40 \text{ e Å}^{-3}$ . Their O lone-pair peaks amount to  $0.30\text{--}0.37 \text{ e Å}^{-3}$  and are considerably extended normal to the HOH plane.

**Introduction.** Our interest lies in the determination of the deformation electron density in complex anions containing the second-row elements, such as perchlorates (Bats & Fuess, 1982), chlorates, silicates and sulfates (Bats, Fuess, Teng, Elerman & Joswig, 1981). In the present paper we present our results on  $\text{MgS}_2\text{O}_3 \cdot 6\text{H}_2\text{O}$ . A comparison can be made with the deformation density in  $\text{Na}_2\text{S}_2\text{O}_6 \cdot 2\text{H}_2\text{O}$  (Kirkel & Will, 1980a) and  $\text{CaSO}_4$  (Kirkel & Will, 1980b).

The crystal structure of  $\text{MgS}_2\text{O}_3 \cdot 6\text{H}_2\text{O}$  was determined by Nardelli, Fava & Giraldi (1962) and refined by Baggio, Amzel & Becka (1969). Recently the structure was refined from neutron diffraction data at 296 and 120 K (Elerman, Fuess & Joswig, 1982). The present work reports new more accurate neutron diffraction data.

**Experimental.** Single crystals grown from aqueous solutions, cell parameters at 120 K refined from setting angles of 15 reflections in the X-ray diffraction experiment.

### Neutron diffraction

Crystal  $3.2 \times 1.7 \times 1.6$  mm,  $\omega/2\theta$  scan, diffractometer D9 at ILL, Grenoble, cryostat described by Allibon, Filhol, Lehmann, Mason & Simms (1981), thermocouple calibrated with a  $\text{KH}_2\text{PO}_4$  crystal (phase transition at 122.8 K), one octant measured up to  $\sin \theta / \lambda = 1.02$  Å<sup>-1</sup>, second octant half completed, 4160 reflections, 2771 independent, two standard reflections every 50 reflections, fluctuations up to 5%, data rescaled with respect to standards, background corrections by profile analysis (Lehmann & Larsen, 1974), linear absorption coefficient determined experimentally, transmission range: 0.745 to 0.795, averaging of equivalent reflections:  $R(I)_{\text{internal}} = 0.05$ , weights assigned to individual reflections according to  $w(F) = s^{-2}$ , where  $s$  is the larger value of  $\sigma(F)$  or  $0.03F$ .

Structure refinement on  $F$  by *ORXFLS* (Busing *et al.*, 1974), extinction correction according to Coppens & Hamilton (1970), scattering lengths from Koester (1977); several very weak reflections with  $F_o \gg F_c$  were believed to be affected by multiple reflections; 88 reflections had  $F_{\text{obs}} > 2F_{\text{calc}}$  and were excluded from the refinement; final refinement included a correction for anisotropic extinction; difference from an isotropic extinction correction was not significant, final  $R$  values  $R(F) = 0.053$ ,  $R_w(F) = 0.047$ ,  $S = 1.13$ .

### X-ray diffraction

Crystal  $0.32 \times 0.37 \times 0.25$  mm, Syntex P2<sub>1</sub> diffractometer,  $\omega/2\theta$  scan, Enraf–Nonius low-temperature device, thermocouple in the cold-gas stream calibrated using phase transition of  $\text{KH}_2\text{PO}_4$  at 122.8 K, four equivalent reflections up to  $\sin \theta / \lambda = 0.80$  Å<sup>-1</sup>, two in the range  $0.80 < \sin \theta / \lambda < 0.85$  Å<sup>-1</sup>, 13 257 reflections measured, 2489 unique; three standard reflections after every 60 reflections decreased about 20% due to fading of the primary-beam intensity; data rescaled with respect to the standards, background corrections by profile analysis (Blessing, Coppens & Becker, 1974), transmission range for absorption: 0.816–0.859, weights assigned to individual reflections according to  $w(I) = \{\sigma^2(I)_{\text{counting}} + (0.03I)^2\}^{-1}$ , equivalent reflections averaged:  $R(I)_{\text{internal}} = 0.031$ , 2375 independent reflections with  $I > 0$  used, calculations with XRAY system

\* Part III: Bats & Fuess (1982).

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(Stewart, Kruger, Ammon, Dickinson & Hall, 1972), neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974), anomalous dispersion factors from Cromer & Liberman (1970), scattering factors for H from Stewart, Davidson & Simpson (1965), RHF (relativistic Hartree-Fock) values (*International Tables for X-ray Crystallography*, 1974) used in difference syntheses, isotropic extinction correction (Larson, 1969), refinement on  $F$  resulted in  $R(F) = 0.030$ ,  $R_w(F) = 0.026$  and  $S = 1.77$ .

**Discussion.** The atomic parameters are compared with the neutron results in Table 1.\*

The bond distances and angles derived from the neutron diffraction data are given in Table 2, the hydrogen bonds in Table 3. The present result agrees with the work by Elerman, Fuess & Joswig (1982), but is a factor of 5–8 more accurate.

Comparison of the positional parameters of the X-ray and neutron refinement show, not considering the H atoms, significant differences only for the sulfate O atoms [O(1) and O(2)]. They are shifted by an extension of the S–O bonds by 0.005 (1) and 0.004 (1) Å in comparison to the neutron results. These are the asphericity shifts defined by Coppens (1974). The hydrate O atoms agree within 0.002 (1) Å within the two determinations. The H atoms from the X-ray determination are 0.12–0.25 Å too close to the O atoms.

Considerable differences are found in the thermal parameters of the two data sets. Those of the X-ray determination are on the average 15% larger than those of the neutron determination. These relative differences are, however, not the same for all atoms, but are larger for the Mg and S atoms (20%) than for the O atoms (13%). When the absolute differences are considered, no such dependence of the atom type is found. The absolute differences in thermal parameters can be calculated as

$$\Delta U_{ii} = \frac{\sum_{\text{atoms}} w [U_{ii}(X) - U_{ii}(N)]}{\sum_{\text{atoms}} w}$$

and are  $\Delta U_{11} = 0.00169$  (10),  $\Delta U_{22} = 0.00051$  (9) and  $\Delta U_{33} = 0.00180$  (11). A similar effect was observed in other combined X-ray and neutron diffraction studies (Bats & Fuess, 1982). As the thermal parameters from neutron diffraction studies are generally lower than those from X-ray diffraction studies, it seems unlikely that temperature differences in the measurements are responsible for this effect. It may result from diffuse scattering.

The deformation electron density in the title compound was calculated by a Fourier synthesis using the

\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38325(35 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Positional parameters and equivalent values of the anisotropic thermal parameters* ( $U_{\text{eq}} = \frac{1}{3} \text{ trace } U$ )

First entry from neutron data, second entry from X-ray data.

	$x$	$y$	$z$	$U_{\text{eq}}(\text{\AA}^2)$
Mg	0.0	0.0	0.0	0.0059 (2)
	0.0	0.0	0.0	0.0072 (1)
S(1)	0.04096 (14)	0.25	0.43957 (20)	0.0067 (5)
	0.04105 (2)	0.25	0.43971 (3)	0.00811 (6)
S(2)	0.21649 (15)	0.25	0.61198 (22)	0.0088 (5)
	0.21639 (2)	0.25	0.61200 (3)	0.01029 (7)
O(1)	−0.04314 (6)	0.16614 (4)	0.47737 (9)	0.0138 (2)
	−0.04325 (7)	0.16579 (4)	0.47767 (8)	0.0154 (2)
O(2)	0.09119 (9)	0.25	0.23547 (11)	0.0121 (3)
	0.09132 (9)	0.25	0.23491 (10)	0.0136 (2)
O(3)	−0.15483 (5)	0.08144 (3)	0.14763 (8)	0.0088 (2)
	−0.15484 (5)	0.08130 (3)	0.14755 (7)	0.0099 (2)
O(4)	0.12792 (5)	0.11398 (3)	−0.03015 (8)	0.0103 (2)
	0.12791 (6)	0.11387 (3)	−0.03032 (7)	0.0115 (2)
O(5)	−0.09866 (6)	0.02623 (4)	−0.25960 (8)	0.0113 (2)
	−0.09855 (6)	0.02614 (4)	−0.25936 (8)	0.0126 (2)
H(1)	0.87919 (14)	0.11058 (9)	0.26887 (17)	0.0233 (5)
	0.8770 (15)	0.1047 (10)	0.2512 (20)	0.034 (4)
H(2)	0.80934 (14)	0.13264 (8)	0.06707 (19)	0.0239 (5)
	0.8129 (14)	0.1231 (9)	0.0793 (19)	0.022 (3)
H(3)	0.11983 (15)	0.16151 (8)	0.07098 (19)	0.0242 (5)
	0.1278 (17)	0.1542 (12)	0.0629 (24)	0.045 (4)
H(4)	0.15472 (16)	0.14679 (9)	0.85137 (19)	0.0269 (6)
	0.1521 (15)	0.1442 (10)	0.8618 (20)	0.030 (3)
H(5)	0.91993 (15)	0.07736 (9)	0.65198 (19)	0.0256 (5)
	0.9166 (14)	0.0680 (11)	0.6646 (22)	0.034 (4)
H(6)	0.81742 (14)	0.99294 (9)	0.69599 (21)	0.0257 (5)
	0.8352 (14)	0.9986 (10)	0.7076 (22)	0.037 (4)

Table 2. *Bond distances* (Å) *and angles* (°) *from the neutron refinement*

Values in square brackets include a libration correction.

2 × Mg–O(3)	2.1208 (6)	O(3)–H(1)	0.985 (2)
2 × Mg–O(4)	2.0453 (7)	O(3)–H(2)	0.982 (2)
2 × Mg–O(5)	2.0404 (7)	O(4)–H(3)	0.979 (2)
S(1)–S(2)	2.019 (2)	O(4)–H(4)	0.974 (2)
	[2.024 (2)]	O(5)–H(5)	0.972 (2)
2 × S(1)–O(1)	1.468 (1)	O(5)–H(6)	0.967 (2)
	[1.474 (1)]		
S(1)–O(2)	1.477 (2)		
	[1.481 (2)]		
2 × O(3)–Mg–O(4)	89.88 (3)	2 × O(1)–S(1)–S(2)	109.26 (7)
2 × O(3)–Mg–O(5)	89.60 (3)	O(1)–S(1)–O(1)	111.45 (10)
2 × O(4)–Mg–O(5)	88.58 (3)	2 × O(1)–S(1)–O(2)	109.67 (7)
Mg–O(3)–H(1)	114.9 (1)	O(2)–S(1)–S(2)	107.43 (10)
Mg–O(3)–H(2)	112.4 (1)	H(1)–O(3)–H(2)	105.20 (13)
Mg–O(4)–H(3)	116.7 (1)	H(3)–O(4)–H(4)	105.62 (13)
Mg–O(4)–H(4)	128.8 (1)	H(5)–O(5)–H(6)	109.11 (14)
Mg–O(5)–H(5)	127.2 (1)		
Mg–O(5)–H(6)	123.2 (1)		

Table 3. *Hydrogen bonds*

O–H...O,S	H...O,S (Å)	O...O,S (Å)	O–H...O,S (°)
O(3)–H(1)...O(1)	1.793 (2)	2.775 (1)	174.5 (1)
O(3)–H(2)...S(2 <sup>l</sup> )	2.268 (2)	3.250 (1)	177.3 (1)
O(4)–H(3)...O(2)	1.727 (2)	2.703 (1)	174.1 (1)
O(4)–H(4)...S(2 <sup>ll</sup> )	2.292 (2)	3.252 (1)	168.4 (1)
O(5)–H(5)...O(1 <sup>ll</sup> )	1.790 (2)	2.760 (1)	176.3 (1)
O(5)–H(6)...O(3 <sup>lll</sup> )	1.888 (2)	2.848 (1)	171.0 (1)

Symmetry code

- (i)  $-\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} - z$
- (ii)  $x, y, z - 1$
- (iii)  $-\frac{1}{2} - x, -y, -\frac{1}{2} + z$

X-ray data up to  $\sin\theta/\lambda = 0.80 \text{ \AA}^{-1}$ . The atomic parameters of the O and H atoms in the structure-factor calculation were taken from the neutron determination. To account for the systematic differences in thermal parameters, modified thermal parameters were used, defined as  $U_{ii}(\text{modified}) = U_{ii}(\text{neutron}) + \Delta U_{ii}$ . It was noted that the atomic parameters of Mg and S have larger errors in the neutron than in the X-ray determination. This may lead to additional errors in the  $X-N$  density near those atoms. As the valence density of Mg and S is known to be rather diffuse, atomic parameters for those atoms were taken from a refinement of the X-ray data with  $\sin\theta/\lambda > 0.50 \text{ \AA}^{-1}$ . The deformation density obtained in this way is shown in Fig. 1 in a number of sections through the thiosulfate group. The average error in the synthesis is calculated by  $2\sqrt{2/V\{\sum\sigma^2(F)\}^{1/2}}$  and is  $0.053 \text{ e \AA}^{-3}$  for the present result.

An accumulation of electron density with single peaks of about  $0.25 \text{ e \AA}^{-3}$  is found at the midpoints of both S—O bonds and the S—S bond and result from covalent bonding. The S—S bond shows little resemblance to the S—S bonds in cyclic octasulfur (Coppens, Young, Blessing, Cooper & Larsen, 1977), showing that the bonding in that compound is of a different character. The area near both S atoms is electron deficient.

Troughs ranging from  $-0.30$  to  $-0.40 \text{ e \AA}^{-3}$  are found in the extension of the O—S and the S(2)—S(1) bonds. They show that a considerable charge transfer has taken place at the  $sp^3$  hybridized S(1) atom, from the back of the atom into each of the bonds. A similar feature was observed for the Cl atom in chlorates and perchlorates (Bats & Fues, 1982; Bats, Fues, Teng, Elerman & Joswig, 1981). Extended regions with lone-pair density are found at both the sulfate O atoms.

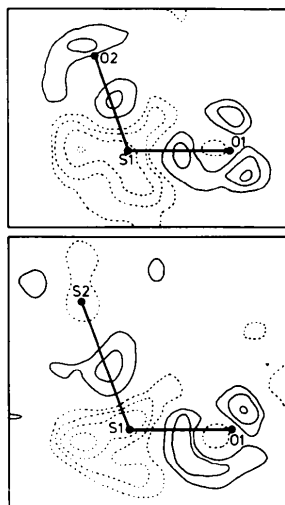


Fig. 1. Deformation density in the O(1)–S(1)–O(2) and O(1)–S(1)–S(2) sections. Contour interval  $0.1 \text{ e \AA}^{-3}$ , negative contours dashed, zero contour omitted.

At O(1) maxima up to  $0.30 \text{ e \AA}^{-3}$  are found under angles of  $100\text{--}120^\circ$  with the S—O bond. For O(2) one smeared peak with a maximum of  $0.25 \text{ e \AA}^{-3}$  is observed at the back of the atom and extends to the sides of the atom. The density at O(2) shows a similarity with the single broad lone-pair peaks in sulfamic acid (Bats, Coppens & Koetzle, 1977). Good agreement is also found with other sulfate derivatives, notably sodium sulfanilate dihydrate (Bats, 1977),  $\text{CaSO}_4$  (Kirfel & Will, 1980*b*, 1981) and  $\text{Na}_2\text{S}_2\text{O}_6 \cdot 6\text{H}_2\text{O}$  (Kirfel & Will, 1980*a*).

In all these compounds extended lone-pair density is observed at the O atoms in either a broad single maximum or in two separate maxima. No lone-pair density is observed at the back of the S(2) atoms. Instead a rather diffuse ring of density up to  $0.13 \text{ e \AA}^{-3}$  is observed under angles of  $95\text{--}125^\circ$  with the S(1)—S(2) bond. This density does not exceed  $2.5\sigma$  when the error in the map is considered. A similar ring of diffuse lone-pair density was observed at the S atom in NaSCN (Bats, Coppens & Kvik, 1977), although the present peak appears slightly farther away from the S atom ( $1.00$  vs  $0.77 \text{ \AA}$ ).

To analyze the deformation density in the water molecules, two sections have been calculated for each of the three independent hydrate groups. The density in the HOH plane is shown on the left-hand side of Fig. 2. Sections in the plane bisecting the HOH angle are shown on the right-hand side of the same figure. The HOH sections show all six hydrogen bonds. The three water molecules look rather similar. Peaks with maxima of  $0.35\text{--}0.40 \text{ e \AA}^{-3}$  are found in all the O—H bonds near the H atoms. The regions at the back of the H atoms are electron deficient. All hydrogen bonds point closely to the lone-pair lobes at the acceptor O and S atoms. The HOH sections show pear-shaped lone-pair peaks at the back of the hydrate O atoms which slightly extend over the atomic positions. These oxygen lone-pair peaks are, however, considerably extended in the direction normal to the molecular plane. The broad lone-pair peak of O(3) clearly shows two maxima with heights of  $0.37 \text{ e \AA}^{-3}$ . One would expect these two maxima for ideal  $sp^3$  hybridization. Each of the two lobes acts as an acceptor to a bond: the Mg—O(3) bond and the O(5)—H(6)···O(3) hydrogen bond. For the O(4) and O(5) atoms which are both involved in one Mg—O bond and do not accept hydrogen bonds, the two lone-pair lobes are no longer separated but one broad peak is observed. It is not clear whether the occurrence of this one peak is due to the influence of the surrounding  $\text{Mg}^{2+}$  atoms or it results from thermal smearing of the electron density. The deformation density at the water molecules shows a good resemblance to the water molecules in oxalic acid dihydrate (Stevens & Coppens, 1980) and  $\text{LiOH} \cdot \text{H}_2\text{O}$  (Hermansson & Thomas, 1982) and to theoretical calculations of  $\text{H}_2\text{O}$  molecules (Johansen, 1979; Stevens, 1980; Hermansson & Lunell, 1981).

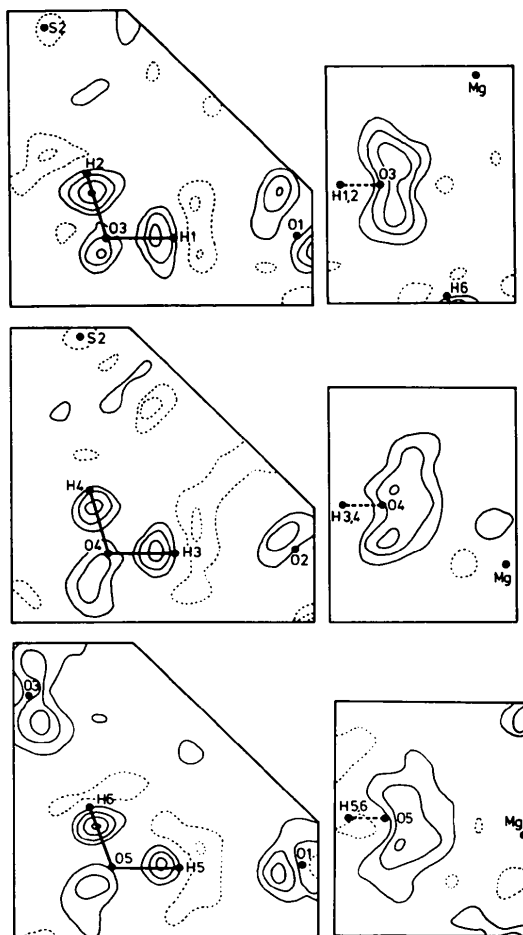


Fig. 2. Deformation density in sections containing the hydrate groups. Contours as in Fig. 1. At left: the plane defined by the HOH group; at right: the plane bisecting the OH bonds.

Less density is found in the hydrates reported by Tellgren, Thomas & Olovsson (1977) and Thomas (1978). This may result from the use of contracted H-atom scattering factors by those authors, and moreover those studies were done at room temperature.

The Mg—O bonds are characterized by the presence of the negative O lone-pair regions which point toward the Mg cation. Near the  $Mg^{2+}$  atom an electron-deficient region is observed with an average height of about  $-0.10 e \text{ \AA}^{-3}$ . Thus evidence is obtained for ionic bonding in the  $Mg(OH)_6^{2+}$  complex.

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