

Table 2. Bond lengths (Å), bond angles (°), torsion angles (°) and barium coordination distances (Å) in barium D-glucose 6-phosphate heptahydrate

O(1)—C(1)	1.375 (4)	C(2)—C(3)	1.525 (4)
O(2)—C(2)	1.427 (4)	C(3)—C(4)	1.524 (4)
O(3)—C(3)	1.431 (4)	C(4)—C(5)	1.531 (5)
O(4)—C(4)	1.418 (4)	C(5)—C(6)	1.522 (5)
O(5)—C(5)	1.432 (4)	P—O(6)	1.615 (3)
O(5)—C(1)	1.437 (4)	P—O(7)	1.514 (3)
O(6)—C(6)	1.431 (4)	P—O(8)	1.528 (3)
C(1)—C(2)	1.530 (5)	P—O(9)	1.510 (3)
O(6)—P—O(7)	101.6 (2)	O(2)—C(2)—C(3)	110.4 (3)
O(6)—P—O(8)	107.2 (2)	C(1)—C(2)—C(3)	110.4 (3)
O(6)—P—O(9)	108.0 (2)	O(3)—C(3)—C(2)	111.1 (3)
O(7)—P—O(8)	113.8 (2)	O(3)—C(3)—C(4)	111.7 (3)
O(7)—P—O(9)	113.1 (2)	C(2)—C(3)—C(4)	109.8 (3)
O(8)—P—O(9)	112.2 (2)	O(4)—C(4)—C(3)	109.2 (3)
P—O(6)—C(6)	120.1 (3)	O(4)—C(4)—C(5)	111.3 (3)
C(1)—O(5)—C(5)	113.3 (3)	C(3)—C(4)—C(5)	107.9 (3)
O(1)—C(1)—O(5)	107.2 (3)	O(5)—C(5)—C(4)	108.4 (3)
O(1)—C(1)—C(2)	109.1 (3)	O(5)—C(5)—C(6)	108.7 (3)
O(5)—C(1)—C(2)	110.0 (3)	C(4)—C(5)—C(6)	114.2 (3)
O(2)—C(2)—C(1)	107.9 (3)	O(6)—C(6)—C(5)	110.9 (3)
O(7)—P—O(6)—C(6)	169.2 (3)	P—O(6)—C(6)—C(5)	-119.6 (5)
O(8)—P—O(6)—C(6)	49.6 (4)	O(6)—C(6)—C(5)—O(5)	-68.2 (5)
O(9)—P—O(6)—C(6)	-71.6 (4)	O(6)—C(6)—C(5)—C(4)	53.0 (5)
Ba—O(3 ^a)	2.876 (3)	Ba—O(7)	2.754 (3)
W(1 ^b)	2.823 (4)	W(1 ^b)	2.865 (4)
W(2 ^b)	2.742 (3)	W(3 ^b)	2.746 (3)
W(5)	2.830 (4)	W(7 ^b)	2.934 (3)
W(7 ^b)	2.961 (3)		

Symmetry code: (i) $1-x, y-0.5, 1-z$; (ii) $x-1, y, z$; (iii) $1-x, y+0.5, 1-z$.

Final atom parameters are summarized in Table 1.* The overall molecular configuration and the atom-numbering scheme of the dianion are shown in

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and hydrogen-bond data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53488 (37 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of (μ -Sulfur dioxide)bis(dicarbonylcyclopentadienyliron) Hydrate

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Abstract. $[\text{Fe}_2(\text{CO})_4(\text{SO}_2)(\text{C}_5\text{H}_5)_2] \cdot \text{H}_2\text{O}$, $M_r = 436.0$, monoclinic, $P2_1/n$, $a = 10.884$ (2), $b = 12.424$ (1), $c = 12.658$ (5) Å, $\beta = 103.35$ (3)°, $V = 1665.3$ (13) Å³, Z

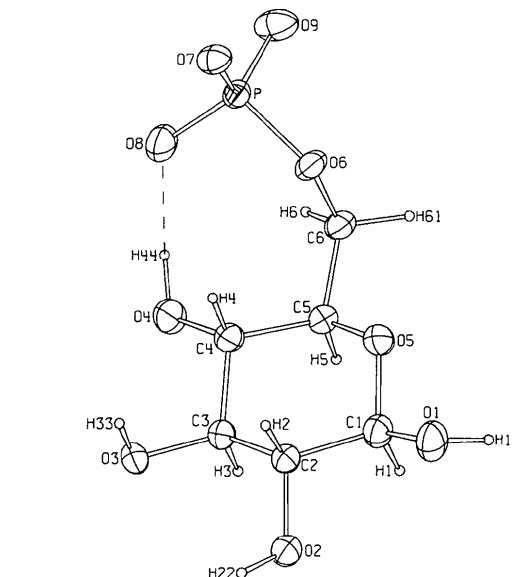


Fig. 1. The structure of the D-glucose 6-phosphate dianion, showing the numbering of the atoms in the barium salt.

Fig. 1. Principal interatomic distances, bond and torsion angles are given in Table 2.

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$= 4$, $D_x = 1.74$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 18.9$ cm⁻¹, $F(000) = 880$, $T = 293$ K, $R = 0.0355$ for 1455 reflections with $F_o^2 > 3\sigma(F_o^2)$. Hydrogen bonds © 1991 International Union of Crystallography

between the water molecule and the SO₂ unit link complexes related by the inversion centers. The water molecule causes the complex to crystallize in a *trans* conformation as opposed to the *gauche* form of the anhydrous material. S—O distances are 1.480 (4) and 1.496 (4) Å and Fe—S distances are 2.268 (2) and 2.282 (2) Å. Fe—S—Fe and O—S—O angles are 119.64 (7) and 111.2 (2)°, respectively.

Experimental. Title compound prepared by the procedure given by Craig, Tennent & Wojcicki (1980); chromatographic elution of the product with technical grade acetone was the suspected source of the water. Data crystal obtained by slow evaporation of a 1:20 methylene chloride/pentane solution. Dark red crystal 0.17 × 0.17 × 0.46 mm mounted on a glass fiber with epoxy. Intensities measured with an Enraf-Nonius CAD-4 diffractometer using ω -2 θ scans of 4–16° min⁻¹ in θ . Unit cell determined from least-squares analysis of angle data for 25 reflections with 16 < 2 θ < 18°. Analytical absorption correction varied from 0.91 to 1.00. Data collected to $(\sin\theta)/\lambda = 0.59 \text{ \AA}^{-1}$, $-10 \leq h \leq 10$, $0 \leq k \leq 11$, $0 \leq l \leq 12$. Three standard reflections (420, 402, 034) decreased 2.5% over 21.6 h of data collection; a linear correction was applied. 2556 reflections measured, 2305 unique ($R_{\text{int}} = 0.019$), 847 reflections with $I < 3\sigma(I)$, where $\sigma^2(I) = \sigma_{\text{cs}}^2(I) + (0.05I)^2$; $\sigma_{\text{cs}}(I)$ is standard deviation of I based on counting statistics. Three of the lowest-angle reflections were given zero weight in the refinement: 011, 110 and 101. Solved by direct methods using *MULTAN11/82* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and Fourier methods. Full-matrix least squares minimized $\sum w(F_o - F_c)^2$. The ring H atoms were constrained to idealized positions (C—H = 0.95 Å), the water H atoms were held at positions obtained from a difference map. All H atoms had fixed isotropic B values of $1.2 \times B$ value of the attached atom. All non-H atoms were refined anisotropically for a total of 217 parameters. $R = 0.035$, $wR = 0.043$, $\text{GOF} = 1.12$, where non-Poisson $w^{-1} = [\sigma^2(I) + (0.05I)^2]/4F^2$. Final $(\Delta/\sigma)_{\text{max}} < 0.1$, $\Delta\rho_{\text{max}} = 0.29$ (7) and $\Delta\rho_{\text{min}} = -0.27$ (7) e Å⁻³ on final difference map. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974, Vol. IV) and programs used were those of Enraf-Nonius (1982) *SDP*.* Table 1 gives the atomic coordinates and Table 2 selected bond distances and angles. Fig. 1 shows the complex with the numbering scheme.

* Tables of anisotropic temperature factors, H-atom positions and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53511 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional coordinates and isotropic or equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

The thermal parameters are of the form $B = 8\pi^2 U$ and $B_{\text{eq}} = (8/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$.

	<i>x</i>	<i>y</i>	<i>z</i>	$B/B_{\text{eq}}(\text{Å}^2)$
Fe(1)	0.17465 (7)	0.12549 (7)	0.71178 (6)	3.38 (2)
Fe(2)	-0.14463 (8)	0.24611 (8)	0.75408 (7)	3.86 (2)
S	0.0603 (1)	0.2013 (1)	0.8225 (1)	3.07 (3)
O(1)	0.0044 (5)	-0.0507 (4)	0.6304 (4)	7.7 (1)
O(2)	0.0827 (5)	0.2664 (4)	0.5263 (3)	6.6 (1)
O(3)	-0.0875 (4)	0.4190 (4)	0.6186 (3)	5.7 (1)
O(4)	-0.1239 (5)	0.3923 (5)	0.9376 (4)	8.5 (2)
O(5)	0.0610 (4)	0.1230 (3)	0.9125 (3)	4.3 (1)
O(6)	0.1272 (4)	0.3018 (3)	0.8642 (3)	4.2 (1)
O(7)	0.5780 (5)	0.6049 (4)	0.4199 (4)	7.2 (1)
C(1)	0.0675 (6)	0.0204 (5)	0.6644 (5)	5.1 (2)
C(2)	0.1161 (6)	0.2126 (5)	0.6008 (5)	4.3 (1)
C(3)	-0.1064 (6)	0.3493 (5)	0.6711 (5)	4.2 (2)
C(4)	-0.1293 (6)	0.3332 (6)	0.8674 (5)	5.5 (2)
C(5)	0.3566 (6)	0.1510 (7)	0.6892 (6)	6.6 (2)
C(6)	0.3472 (6)	0.1932 (6)	0.7859 (7)	7.2 (2)
C(7)	0.3205 (6)	0.1112 (9)	0.8497 (5)	9.0 (3)
C(8)	0.3129 (7)	0.0173 (7)	0.7879 (7)	9.1 (2)
C(9)	0.3361 (6)	0.0461 (6)	0.6889 (6)	6.7 (2)
C(10)	-0.2000 (6)	0.0838 (6)	0.7438 (6)	5.7 (2)
C(11)	-0.2284 (6)	0.1296 (6)	0.6403 (6)	6.0 (2)
C(12)	-0.3141 (6)	0.2133 (7)	0.6391 (6)	7.0 (2)
C(13)	-0.3378 (6)	0.2209 (7)	0.7428 (6)	6.9 (2)
C(14)	-0.2673 (6)	0.1406 (7)	0.8074 (6)	6.6 (2)
H(1)	0.5703	0.5254	0.4062	
H(2)	0.5430	0.5977	0.4883	

Table 2. *Some bond lengths (Å) and bond angles (°) with e.s.d.'s in parentheses*

Fe(1)—S	2.282 (2)	O(7)—H(2)	1.028
Fe(2)—S	2.268 (2)	C(5)—C(6)	1.356 (12)
Fe(1)—C(1)	1.761 (7)	C(5)—C(9)	1.322 (11)
Fe(1)—C(2)	1.771 (7)	C(6)—C(7)	1.372 (14)
Fe(2)—C(3)	1.767 (7)	C(7)—C(8)	1.396 (14)
Fe(2)—C(4)	1.773 (8)	C(8)—C(9)	1.381 (13)
S—O(5)	1.496 (4)	C(10)—C(11)	1.397 (10)
S—O(6)	1.480 (4)	C(10)—C(14)	1.398 (10)
O(1)—C(1)	1.141 (7)	C(11)—C(12)	1.395 (11)
O(2)—C(2)	1.145 (7)	C(12)—C(13)	1.399 (11)
O(3)—C(3)	1.138 (7)	C(13)—C(14)	1.401 (11)
O(4)—C(4)	1.144 (8)	O(7)···O(5 ^a)	2.838 (6)
O(7)—H(1)	1.003	O(7)···O(5 ^b)	2.886 (6)
C(1)—Fe(1)—C(2)	95.4 (3)	C(6)—C(7)—C(8)	106.5 (8)
C(3)—Fe(2)—C(4)	92.7 (3)	C(7)—C(8)—C(9)	107.0 (9)
Fe(1)—S—Fe(2)	119.64 (7)	C(5)—C(9)—C(8)	108.6 (9)
O(5)—S—O(6)	111.2 (2)	C(11)—C(10)—C(14)	107.6 (7)
H(1)—O(7)—H(2)	91.5 (4)	C(10)—C(11)—C(12)	108.4 (7)
Fe(1)—C(1)—O(1)	175.2 (7)	C(11)—C(12)—C(13)	108.0 (7)
Fe(1)—C(2)—O(2)	176.3 (6)	C(12)—C(13)—C(14)	107.7 (8)
Fe(2)—C(3)—O(3)	176.1 (6)	C(10)—C(14)—C(13)	108.3 (7)
Fe(2)—C(4)—O(4)	176.7 (7)	O(5)···O(7)···O(5 ^b)	93.5 (2)
C(6)—C(5)—C(9)	109.6 (8)	O(7)—H(1)···O(5)	167.9
C(5)—C(6)—C(7)	108.3 (9)	O(7)—H(2)···O(5)	159.3

Symmetry code: (i) $\frac{1}{2} + x, \frac{1}{2} - y, -\frac{1}{2} + z$; (ii) $\frac{1}{2} - x, \frac{1}{2} + y, 1\frac{1}{2} - z$.

Related literature. The chemistry of the title complex has been reported by Craig, Tennent & Wojcicki (1980) and by Reich-Rohrwig, Clark, Downs & Wojcicki (1978). The structure of the anhydrous form of the title compound has been published (Churchill, DeBoer & Kalra, 1973) and the structural

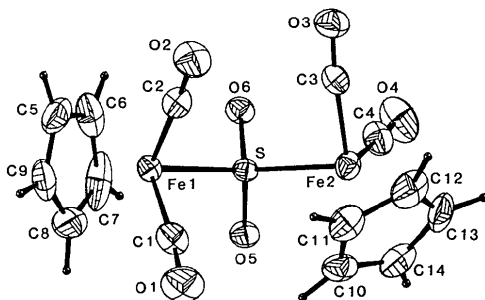


Fig. 1. ORTEP (Johnson, 1976) drawing and numbering scheme. The ellipsoids are drawn at the 30% probability surface and H atoms have been given arbitrary radii for figure clarity.

features of sulfur dioxide complexes in general have been reviewed by Ryan, Kubas, Moody & Eller (1981).

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Structures of Two Bis(ethylenedithio)tetrathiafulvalene Hexamolybdate and Hexatungstate Salts: (BEDT-TTF)₂M₆O₁₉, M = Mo, W

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Abstract. $2C_{10}H_8S_8^+ \cdot Mo_6O_{19}^{2-}$ (1), $M_r = 1649$, monoclinic, $P2_1/c$, $a = 8.898$ (8), $b = 11.265$ (4), $c = 21.098$ (8) Å, $\beta = 96.34$ (5)°, $V = 2102$ Å³, $Z = 2$, $D_x = 2.605$ g cm⁻³, $F(000) = 1592$, $\lambda(Mo K\alpha) = 0.71073$ Å, $\mu = 25.33$ cm⁻¹, $T = 293$ K, $R = 0.036$ based on 1527 observed reflections with $I \geq 3\sigma(I)$. $2C_{10}H_8S_8^+ \cdot W_6O_{19}^{2-}$ (2), $M_r = 2176.46$, monoclinic, $P2_1/c$, $a = 8.908$ (7), $b = 11.310$ (5), $c = 21.111$ (8) Å, $\beta = 96.37$ (7)°, $V = 2113.8$ Å³, $Z = 2$, $D_x = 3.419$ g cm⁻³. Only the structure of (1) has been determined since the two compounds are isostructural. The central C=C (1.388 Å) and the two types of C—S (average 1.720 and 1.737 Å) bond lengths of the BEDT-TTF molecule compare well with those

observed for one which is fully oxidized. As in the κ -(BEDT-TTF)₂X salts, the organic radical cations form orthogonalized dimers (dihedral angle 76°). Short intra-dimer (S1...S4 3.551 and S2...S3 3.553 Å) and inter-dimer (S7...S8 3.508 Å) S...S contacts are observed. The crystal structure is built by alternating ABABA... organic and inorganic layers along the [100] direction.

Experimental. The compounds were obtained on a platinum-wire electrode by anodic oxidation of DMF (DMF = *N,N*-dimethylformamide) solutions of the organic donor (BEDT-TTF) (2×10^{-3} M), under low constant current ($I = 1.25$ μA) in the presence of the tetrabutylammonium salts of the [M₆O₁₉]²⁻ dianions (10^{-2} M) as supporting electro-

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