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On the Crystal Chemistry of Salt Hydrates, III.
The Determination of the Crystal Structure of $FeSO_4.7H_2O$ (Melanterite)*

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$FeSO_4.7H_2O$ crystallizes in space group $P2_1/c$ with $a=14.072$, $b=6.503$, $c=11.041$ Å, $\beta=105°34'$, $Z=4$. The crystal structure has been determined from Patterson and Fourier projections using $F_{h0\ell}$ and F_{hk0} data. The refinement was done by the method of least squares employing 738 observed structure factors from the zones hk0, h0ℓ, 0kℓ, h3ℓ and hk1. The final R value, including the contribution of the hydrogen atoms is 4.5%. The Fe ions occupy special positions and are surrounded octahedrally by six water molecules each. The six water oxygens form hydrogen bonds to the tetrahedral sulfate group and to the seventh water molecule which is not coordinated to Fe^{2+}. The mean bond length of $Fe-O_w$ is 2.12_4 Å, of S-O 1.47_4 Å and of O-H---O 2.82 Å. One of the Fe-coordinated water oxygens receives an "outward" hydrogen bond from the seventh water oxygen. The bond from Fe to this water oxygen is 2.19 Å and thus significantly longer than the mean $Fe-O_w$ bond length.

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Introduction

Monoclinic $FeSO_4.7H_2O$ is the stable solid phase between -1.82° and 56.6° C in contact with a saturated water solution of $FeSO_4$ (Fraenckel, 1907). It occurs in nature as an oxidation product of Fe-containing sulfides and is called melanterite. $FeSO_4.7H_2O$ belongs to a series of compounds $Me^{2+}SO_4.nH_2O$, where Me^{2+} is a cation with an approximate ionic radius of 0.7 Å. The 1-, 4- and 5-hydrates are known to crystalize each in only one form, whereas the hexa- and the heptahydrates occur both in two different forms (Groth, 1908). The crystal structure of the hexahydrates are known. Beevers and Lipson (1932) determined the structure of the tetragonal $NiSO_4.6H_2O$; Zalkin, Ruben and Templeton (1962) reported the structure of the monoclinic $CoSO_4.6H_2O$. Of the structure of the heptahydrates but one was described: the orthorhombic form of $NiSO_4.7H_2O$ (Beevers and Schwartz, 1935). No details were known about one of the monoclinic heptahydrates, though Leonhardt and Ness (1947) published the cell constants and the space group of $FeSO_4.7H_2O$. In addition they stated essentially correct positional parameters for the sulfur atom and gave the correct positions of the iron atoms. - The present investigation has been undertaken as part of an extensive study of salt hydrates (I=Baur, 1962a; II=Baur, 1964a; IV=Baur, 1964b). A preliminary account has been published before (Baur, 1962b).

Experimental

Green crystals of $FeSO_4.7H_2O$ were grown at 35° C from an aqueous solution of sulfuric acid saturated with $FeSO_4$. Crystal plates of approximately 0.2x2.0x2.0 mm^3 were ground perpendicular to [100], [010] and [001]. $0k\ell$, $h0\ell$, $hk0$, $h3\ell$ and hkl data were collected on a precession camera using the multiple exposure technique (Mo-K_α radiation, Zr-filter). In order to prevent decomposition of the crystals they were coated with a protective layer of a synthetic resin. The intensities of 738 independent reflections were measured using a recording microphotometer. The intensities of an additional 288 reflections were below the detection limit. The cell constants were derived from the precession photographs (Mo-K_α(mean)=0.7107 Å).

The intensites were reduced to F_o using a Lorentz-polarization factor program written by the author for an IBM 650 with indexing registers and floating point arithmetic. This program is based on the formulas of Waser (1951) and Burbank (1952). The calculations for the first stages of the structure determination were carried out on an IBM 650 computer using the structure factor program by Shiono (1959) and a 2-dimensional Fourier synthesis program written by the author. This latter program is especially fast since it takes advantage of the indexing registers and also because the whole first summation is stored internally on the magnetic drum. The ultimate refinement of the structure was performed on an IBM 7094 at Brookhaven National Laboratory using local modifications of the Fortran crystallographic least squares program ORFLS and the function and error program ORFFE (Busing, Martin and Levy, 1962a, 1962b), as well as of the Fourier synthesis program Fordaper by Allan Zalkin.

Crystal Data

$FeSO_4.7H_2O$ is monoclinic, with a=14.072(10) Å*, b=6.503(7) Å, c=11.041(10) Å and β=105°34(5)'; V=973(2) Å^3, Z=4, D_x=1.897(4) g.cm^{-3}, D_m=1.895 g.cm^{-3} (Moles and Crespi, 1927). Because of the characteristic extinctions, $h0\ell$ only present with $h=2n$, 0k0 only with $k=2n$, $P2_1/c$ was chosen as space group. The a:b:c calculated from the above cell constants is 2.164:1:1.698. Groth (1908) gives a:b:c = 1.1828:1:1.5427 , β=104°16', recalculated to the present setting this corresponds

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*Throughout this paper the estimated standard deviations are given in parantheses following the value. They correspond to the last significant digits of these values.

to $a:b:c = 2.169:1:1.689$, $\beta = 105^\circ 28'$. The matrix for the transformation from Groth's setting to the present one is $(\frac{1}{2}0-\frac{1}{2}/0\frac{1}{2}0/\frac{1}{2}0\frac{1}{2})$.

Determination of Structure

Since the intensities of the reflections with h, k and ℓ all 2n or all 2n+1 were observed to be on the average stronger than those with mixed indices it was assumed that the iron atoms occupied the special positions (a) $000, 0\frac{1}{2}\frac{1}{2}$ and (d) $\frac{1}{2}\frac{1}{2}0$, $\frac{1}{2}0\frac{1}{2}$, thus forming a face-centered arrangement. Positions (a) and (d) have the point symmetry $\bar{1}$, as have the other two special positions (b) and (c). Thus (b) and (c) cannot be expected to be occupied since neither the sulfur atom or the oxygen atoms of the SO_4-group, nor a water oxygen can have a surrounding with this symmetry. Therefore all other atoms must lie in the fourfold general position (e): $\pm(xyz;\ x, \frac{1}{2}-y, \frac{1}{2}+z)$. The Patterson projection along [010] was readily interpreted on this assumption. It resembles closely a double Fourier projection, since the Fe-S and Fe-O vectors from the two different Fe-positions each form an image of the structure; the S-O and O-O vectors are too weak to change this picture substantially. A first structure factor calculation, which included the Fe-atoms and the SO_4-group with positional parameters derived from the Patterson projection yielded an $R = 0.53$ $(R = \sum \left||F_o| - |F_c|\right| / \sum |F_o|)$ for the reflections with $\sin\vartheta/\lambda < 0.38$ Å^{-1}. A Fourier projection calculated with 76 $F_{h0\ell}$ revealed the positions of all the water oxygens. The 231 $F_{h0\ell}$ (including not observed reflections) were refined by 11 cycles of consecutive Fourier or $(F_o - F_c)$ syntheses and structure factor calculations to $R = 0.071$. The last difference syntheses were performed using only the reflections with $h = 2n+1$, to which the Fe atoms do not contribute. Since the Patterson projections along [001] and [100] were not easily interpretable the y-parameters were obtained from the [010]-projection: assuming the bond length of S-O to be 1.48 Å, of

Fe-O_w 2.12 Å and of O-H---O 2.82 Å the heights of all the atoms were calculated from their x- and z-parameters. The y-parameters thus derived were used as a starting point for refining the 143 F_{hk0} (including not observed reflections) by difference syntheses. In eight cycles R was lowered from 0.36 to 0.078. The Fourier projections parallel to [010] and [001] are shown in Figures 1(a) and (b).

The final refinement was carried out on 738 non zero $h0\ell$, $hk0$, $0k\ell$, $h3\ell$ and hkl reflections by the method of least-squares using the weighting scheme of Hughes (1941). By two cycles of refinement with isotropic temperature factors R was lowered from 0.081 to 0.072. Three more cycles of refinement employing anisotropic temperature factors, brought R down to 0.054. In the last refinement 125 parameters (36 positional parameters, 84 thermal parameters and 5 scale factors) were varied.

At this stage the hydrogen atoms were taken into account. Their positions were calculated under the following assumptions:

1) The O-H distance is 0.97 Å; $\measuredangle$H-O-H is 109.5°.
2) The hydrogen atoms lie in the plane defined by the water oxygen and the two oxygens to which this water oxygen is hydrogen bonded.
3) The hydrogen atoms are as far as possible separated from the cation to which the water oxygen is bonded. For O_{w7} which is not coordinated to a Fe^{2+}, instead the midpoint of the positions of the two hydrogens which form hydrogen bonds to O_{w7}, was chosen as "cation" position.

The assumptions 2) and 3) are not strictly valid as is known from the neutron diffraction studies of $CuSO_4.5H_2O$ (Bacon, 1962) and $MgSO_4.4H_2O$ (II). The positions derived for the hydrogen atoms in this way are therefore expected to be correct only within 0.1 to 0.2 Å. That these positions are reasonable is supported by the fact, that on inclusion of the hydrogen atoms in the structure factor calculation R was lowered to 0.045 (for all observed reflections). Moreover a difference projection along [010], computed with F_o-$F_{c(heavy)}$, shows the positions of all the hydrogen atoms as well as can be expected in the presence of an iron atom (Figure 2). The distances between the hydrogen atoms themselves are sensible. The shortest H-H distance is 2.11 Å (H_{57}-H_{74}) all other H-H distances are above 2.23 Å. But even a distance of 2.11 Å is not precluded since distances of 2.12 Å between hydrogen atoms from different water molecules have been found by neutron-diffraction in crystals of $Na_2CO_3.NaHCO_3.2H_2O$ (Bacon and Curry, 1956) and $CrK(SO_4)_2.12H_2O$ (Bacon and Gardner, 1958).

The final positional and thermal parameters are given in Table 1 and 2, the calculated positions of the hydrogen atoms in Table 3. The B's in Table 2 are the isotropic B's equivalent to the anisotropic temperature factors (Hamilton, 1959). The observed and calculated structure factors are listed in Table 4. Non-obsered reflections are marked by an asterisk, the value given here for F_o has been calculated from $\frac{1}{2}.I_{min}$. The atomic form factors used in cumputing F_c were taken from the International Tables (Ibers, 1962), The F_c include the contribution of the hydrogen atoms; the B of the isotropic temperature factor for the hydrogen atoms was chosen uniformly 1.3 $Å^2$ higher than the equivalent isotropic B of the corresponding water oxygens. The R-value for all the reflections including the 288 unobserved ones is 0.071.

Discussion of the Structure

The bond distances and angles in $FeSO_4.7H_2O$ are presented in Table 5. The estimated standard deviations given have been calculated using the least-squares variance-covariance matrix and therefore include the effects of correlations between parameters. None of the positional parameters is involved in high correlations. The highest correlation coefficient of 0.6 is one between two thermal parameters. The four components of the structure, i. e. the two different $Fe(H_2O)_6^{2+}$ octahedra, the SO_4^{2-} tetrahedron and the seventh water molecule, which is not coordinated to Fe^{2+}, are linked together by hydrogen bonds. A schematic representation of the hydrogen bonding is shown in Figure 3, the actual spatial arrangement can be gathered from Figure 4.

The average value of the O-H---O bond lengths is 2.82 Å, the spread is from 2.71 to 3.03 Å. The angles O-O_w-O around the water oxygens to the oxygen atoms to which they are hydrogen bonded scatter from 103° to 145°. Assuming the hydrogen positions of Table 3 to be correct the angles O_w-H-O range (with one exception) from 160° to 180°, the angles H-O_w-O from 0° to 13°, the angles Fe-O_w-H from 113 to 124°, the distances H----O from 1.75 to 1.83 Å. The exception is the hydrogen bond from O_{w7} to O_{w6} where O_{w6}-H_{76}-O_{w7} is 148°, H_{76}-O_{w7}-O_{w6} is 23° and H----O is 2.17 Å. The assignment of this hydrogen bond is not quite unambiguous. The water oxygens O_{w1} through O_{w6} have each only two contacts to sulfate oxygens or to O_{w7} which can be explained as hydrogen bonds (bonds distances between 2.71 and 2.95 Å, see Table 5 (d)). O_{w7} instead has three such contacts to O_4, O_3 and O_{w6}. The sulfate oxygen O_4 at a distance of 2.77 Å seems clearly to by hydrogen bonded to O_{w7}. The distances of O_3 and O_{w6} to O_{w7} are 3.02 and 3.03 Å, the angles O_4-O_{w7}-O_3 and O_4-O_{w7}-O_{w6} are 97° and 145° respectively. On pure geometrical grounds it cannot be decided whether O_3 or O_{w6} is the acceptor atom of the hydrogen bond from O_{w7}, or if the bond is a bifurcated one. It seems however more probable that the bond goes to O_{w6} since this assignment is in better accord with the difference map parallel to [010]. Further supporting evidence is to be found in the fact that the distance Fe_2-O_{w6} is 2.19 Å and thus significantly longer than the average Fe_2-O_w distance of 2.13 Å. This is similar to the differences in bond lengths of Mg-O_w in $MgSO_4.7H_2O$(IV), where the water oxygens which act as acceptor atoms of hydrogen bonds have an average Mg-O_w bond length of 2.10 Å, while for the ones which are bonded to Mg only, Mg-O_w

is 2.05 Å. These differences in bond lengths can be understood in terms of Paulings electrostatic valence rule: a point which has been discussed at greater length in I. - The coordination octahedron around Fe_1 is distorted in a similar way, although not quite so pronounced, as in FeF_2 (Baur, 1958). In FeF_2 there are two bonds Fe-F of 1.99 Å and four of 2.12 Å; around Fe_1 are two O_{w1} at a distance of 2.07 Å and four O_{w2} and O_{w3} at 2.14 Å. Dunitz and Orgel (1960) interpret tentatively this kind of distortion around Fe^{2+} by a Jahn-Teller mechanism. In the present case however it is possible that this short distance is only apparent. Although one has to be cautious in interpreting the thermal parameters since they are much more liable than positional parameters to be affected by systematic errors in the data, it seems safe to conclude from Table 6 that the thermal motion of O_{w1} is significantly more anisotropic than that of the other water oxygens. The thermal displacements of the water oxygens O_{w2} through O_{w6} and of Fe_1 and Fe_2 can be interpreted as rigid body motions of the whole groups. The directions of the smallest thermal displacement (axis 1 in Table 6) of the water oxygens are at an average angle of 12°(2°) with the line connecting them to the Fe atoms. The displacement of O_{w2} through O_{w6} along the bond $Fe-O_w$ is of approximately the same magnitude as the displacement of the central Fe atom. The other two axes, of intermediate and largest thermal displacement (axes 2 and 3 in Table 6) are at average angles of 94°(4°) and 83°(3°) with the respective $Fe-O_w$ lines. The displacements of O_{w1} along axes 1 and 3 are very different from those of all the other water oxygens. It seems that O_{w1} is moving (at least partly) independently of the rest of the group. The correction of the bond distance for thermal motion assuming that Fe_1 and O_{w1} move independently (Busing, Martin and Levy, 1962b) changes Fe_1-O_{w1} from 2.07 to 2.11 Å. This correction brings the distance Fe_1-O_{w1} in somewhat better accord with the distances Fe_1-O_{w2} and Fe_1-O_{w3}.

The mean bond length for the six different Fe-O_w bonds in $FeSO_4.7H_2O$ is 2.12_4 Å (uncorrected for thermal motion). Reliable information on Fe^{2+}-O_w distances is scarce; the value found here can be compared with the mean value for Fe-O,O_w of 2.122(5) Å in $FeSO_4.4H_2O$ (I) and of 2.140(13) Å in $Fe(H_2O)_6.SiF_6$ (Hamilton, 1962). The differences between these values are not significant. The mean value for the Fe-O_w bond length in $FeSO_4.7H_2O$ is changed to 2.138 Å if the correction for thermal motion is applied to all the distances, namely a correction assuming independent motion for the distance Fe_1-O_{w1} and assuming in-phase motion for all the other Fe-O_w distances. The bonding angles O_w-Fe-O_w scatter from 86.0° to 94.0°, the differences between them are significant; so are the differences between the O_w-O_w distances in the octahedra, which range from 2.90 to 3.13 Å, The mean value of the S-O distances in the sulfate group is 1.47_4 Å, the corresponding value in $MgSO_4.4H_2O$ (II) is 1.47_3 Å. Since the thermal ellipsoids of the sulfate oxygens are similarly oriented in respect to the sulfur atom, as are the water oxygens to the iron atom (the average angles of the axes 1, 2 and 3 with the lines S-O are $14^\circ(3^\circ)$, $94^\circ(2^\circ)$ and $93^\circ(3^\circ)$), rigid body motion of the whole sulfate group may be assumed and the in-phase correction for thermal motion applied to the bond distances: the average S-O bond length becommes then 1.48_5 Å. - The O-S-O angles are close to the tetrahedral value; the deviations of the O-O distances within the sulfate group from their mean value are not significant.

Beevers and Schwartz (1935) made it a point in their description of the orthorhombic crystal structure of $NiSO_4.7H_2O$ that the octahedral coordination of water around a divalent cation like Ni^{2+} is always different from the one around a trivalent ion like Al^{3+}. In the first case there should always be two water oxygens in the octahedron receiving hydrogen bonds from an outside water molecule. This in fact is true for tetragonal $NiSO_4.6H_2O$ (Beevers and Lipson, 1932) and for ortorhombic $NiSO_4.7H_2O$ (the latter structure has been fully confirmed by a refinement of the isostructural $MgSO_4.7H_2O$ (IV)). But it does not hold for the monoclinic structure of $CoSO_4.6H_2O$ (Zalkin, Ruben and Templeton, 1962) and for $FeSO_4.7H_2O$, as has been demonstrated here. In both these structures there are two crystallographically different $Me^{2+}(H_2O)_6$ octahedra in special positions, and in each structure only one out of the two octahedra is of the type reported for the nickel sulfate hydrates.

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References

Bacon, G. E. (1962). Proc. Roy. Soc., London, A, 266, 95.

Bacon, G. E. & Curry, N. A. (1956). Acta Cryst., 9, 82.

Bacon, G. E. & Gardner, W. E. (1958). Proc. Roy. Soc., A, 246, 78.

Baur, W. H. (1958). Acta Cryst., 11, 488.

Baur, W. H. (1962a). Acta Cryst. 15, 815.

Baur, W. H. (1962b). Naturwiss. 49, 464.

Baur, W. H. (1964a). Acta Cryst. 17, in print.

Baur, W. H. (1964b), to be published.

Beevers, C. A. & Lipson, H. (1932). Z. Krist. 83, 123.

Beevers, C. A. & Schwartz, C. M. (1935). Z. Krist. 91, 157.

Burbank, R. D. (1952). Rev. Sci. Instr. 23, 321.

Busing, W. R., Martin, K. O. & Levy, H. A. (1962a). ORNL-TM-305.

Busing, W. R., Martin, K.O. & Levy, H. A. (1962b). ORNL-TM-306.

Dunitz, J. D. & Orgel, L.E. (1960). Adv. Inorg. Chem. Radiochem. 2, 1.

Fraenckel, F. (1907). Z. anorg. Chemie. 55, 223.

Groth, P. (1908). Chemische Kristallographie. Vol. 2. Leipzig: Engelmann.

Hamilton, W. C. (1959). Acta Cryst. 12, 609.

Hamilton, W. C. (1962). Acta Cryst. 15, 353.

Hughes, E. W. (1941). J. Amer. Chem. Soc. 63, 1737.

Ibers, J. A. (1962). Table 3.3.1A. International Tables for X-ray Crystallography, Vol. 3, Birmingham: Kynoch Press.

Leonhardt, J. & Ness, I. (1947). Fortschr. Mineral. Kristallogr. Petrogr. 26,83.

Moles, E. & Crespi, M. (1927). Z. physik. Chem. 130, 337.

Shiono, R. (1959). Univ. of Pittsburgh Computation and Data Processing Center, Techn. Rep. No. 9.

Waser, J. (1951). Rev. Sci. Instr. 22, 563.

Zalkin, A., Ruben, H. & Templeton, D. H. (1962). Acta Cryst. 15, 1219.

Table 1

$FeSO_4 \cdot 7H_2O$, positional parameters and their standard deviations

Atom	x	y	z
Fe_1	0.0000	0.0000	0.0000
Fe_2	0.5000	0.5000	0.0000
S	0.2267(1)	0.4709(3)	0.1763(1)
O_1	0.2045(3)	0.4705(8)	0.0368(3)
O_2	0.1371(3)	0.5369(7)	0.2116(4)
O_3	0.3075(3)	0.6157(7)	0.2267(4)
O_4	0.2556(3)	0.2643(6)	0.2247(5)
O_{w1}	0.1129(4)	0.3853(9)	0.4322(5)
O_{w2}	0.1005(3)	0.9574(7)	0.1822(5)
O_{w3}	0.0305(3)	0.7937(7)	0.4323(4)
O_{w4}	0.4797(3)	0.4590(9)	0.1797(4)
O_{w5}	0.4313(3)	0.2850(8)	0.4418(4)
O_{w6}	0.3536(3)	0.8594(7)	0.4404(5)
O_{w7}	0.3637(3)	0.0048(6)	0.1142(5)

Table 2

$FeSO_4.7H_2O$, probable positions of the hydrogen atoms

Atom	x	y	z
H_{11}	0.148	0.259	0.461
H_{12}	0.126	0.428	0.354
H_{22}	0.124	0.817	0.196
H_{24}	0.156	0.051	0.198
H_{31}	0.090	0.870	0.469
H_{32}	0.979	0.887	0.387
H_{43}	0.421	0.514	0.198
H_{47}	0.536	0.475	0.253
H_{54}	0.375	0.275	0.369
H_{57}	0.413	0.359	0.508
H_{61}	0.297	0.910	0.465
H_{63}	0.331	0.776	0.365
H_{74}	0.313	0.077	0.143
H_{76}	0.334	0.894	0.058

Table 3

$FeSO_4.7H_2O$. Thermal parameters and their standard deviations

The definition of the Debye-Waller temperature factor is:

$$\exp -(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+2\beta_{12}hk+2\beta_{13}hl+2\beta_{23}kl) \;;$$

and B is $(4/3).(\beta_{11}.a^2+\beta_{22}.b^2+\beta_{33}.c^2+2\beta_{13}a.c.\cos\beta)$

Atom	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}	B (Å^2)
Fe_1	0.0022(1)	0.0081(3)	0.0036(1)	-0.0003(1)	0.0010(1)	-0.0004(2)	1.50(5)
Fe_2	0.0020(1)	0.0105(3)	0.0028(1)	-0.0004(1)	0.0006(1)	0.0004(2)	1.50(5)
S	0.0018(1)	0.0084(3)	0.0032(1)	-0.0001(1)	0.0006(1)	-0.0005(2)	1.40(5)
O_1	0.0031(2)	0.0148(11)	0.0034(3)	-0.0015(4)	0.0011(2)	0.0002(6)	2.00(15)
O_2	0.0031(2)	0.0137(9)	0.0061(4)	0.0014(4)	0.0027(2)	0.0013(7)	2.30(15)
O_3	0.0029(2)	0.0150(10)	0.0055(3)	-0.0029(4)	0.0003(2)	-0.0033(7)	2.50(15)
O_4	0.0031(2)	0.0101(8)	0.0055(4)	0.0017(3)	0.0003(3)	0.0019(7)	2.20(15)
O_{w1}	0.0065(3)	0.0238(14)	0.0076(4)	0.0073(6)	0.0052(3)	0.0042(9)	3.70(20)
O_{w2}	0.0039(2)	0.0125(9)	0.0069(4)	-0.0001(4)	0.0002(3)	0.0019(8)	2.80(15)
O_{w3}	0.0030(2)	0.0104(8)	0.0058(3)	-0.0010(4)	0.0005(2)	0.0013(7)	2.30(15)
O_{w4}	0.0030(2)	0.0207(13)	0.0034(3)	0.0000(4)	0.0012(2)	0.0008(7)	2.40(15)
O_{w5}	0.0042(2)	0.0168(10)	0.0049(3)	0.0031(5)	0.0011(3)	0.0005(7)	2.70(15)
O_{w6}	0.0026(2)	0.0171(11)	0.0065(3)	-0.0009(4)	0.0007(2)	-0.0042(8)	2.50(15)
O_{w7}	0.0041(2)	0.0112(10)	0.0069(4)	0.0002(4)	0.0009(3)	-0.0019(8)	2.70(15)

Table 4. $FeSO_4.7H_2O$, observed and calculated structure factors. Not observed F_o are marked by an asterisk.

TABLE 4

H	K	L	OBS	CALC
0	0	0	0	604
1	0	0	1*	2
2	0	0	43	38
3	0	0	21	-19
4	0	0	76	71
5	0	0	3*	-9
6	0	0	59	59
7	0	0	35	-36
8	0	0	99	101
9	0	0	58	57
10	0	0	39	41
11	0	0	24	-23
12	0	0	20	20
13	0	0	4*	3
14	0	0	41	41
15	0	0	22	-23
16	0	0	14	13
17	0	0	6	5
18	0	0	14	14
0	0	2	52	51
1	0	2	76	-74
-1	0	2	78	79
2	0	2	121	129
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15	1	0	4*	4
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TABLE 4 (Cont.)

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-9	9	1	19	19

Table 5

Interatomic distances (Å) and bond angles (degrees)

(a) Coordination octahedron around Fe_1

$Fe_1\text{-}O_{w1}$	2.068(5)
$Fe_1\text{-}O_{w2}$	2.144(5)
$Fe_1\text{-}O_{w3}$	2.136(5)

Mean value: 2.116(3)

$O_{w1}\text{-}O_{w2}$	2.992(7)	2.965(7)	$\sphericalangle O_{w1}\text{-}Fe_1\text{-}O_{w2}$	90.5°(0.2°)
$O_{w1}\text{-}O_{w3}$	3.047(7)	2.897(7)	$\sphericalangle O_{w1}\text{-}Fe_1\text{-}O_{w3}$	92.9°(0.2°)
$O_{w2}\text{-}O_{w3}$	3.129(6)	2.920(6)	$\sphericalangle O_{w2}\text{-}Fe_2\text{-}O_{w3}$	94.0°(0.2°)

(b) Coordination octahedron around Fe_2

$Fe_2\text{-}O_{w4}$	2.096(5)
$Fe_2\text{-}O_{w5}$	2.109(5)
$Fe_2\text{-}O_{w6}$	2.188(5)

Mean value: 2.131(3)

$O_{w4}\text{-}O_{w5}$	2.987(7)	2.960(7)	$\sphericalangle O_{w4}\text{-}Fe_2\text{-}O_{w5}$	90.5°(0.2°)
$O_{w4}\text{-}O_{w6}$	3.057(6)	3.002(6)	$\sphericalangle O_{w4}\text{-}Fe_2\text{-}O_{w6}$	91.0°(0.2°)
$O_{w5}\text{-}O_{w6}$	3.102(7)	2.974(7)	$\sphericalangle O_{w5}\text{-}Fe_2\text{-}O_{w6}$	92.4°(0.2°)

Table 5 cont.

(c) SO_4 tetrahedron

$S-O_1$	1.488(4)
$S-O_2$	1.481(4)
$S-O_3$	1.466(4)
$S-O_4$	1.462(4)
Mean value:	1.474(2)

O_1-O_2	2.407(6)	$\sphericalangle O_1-S-O_2$	$108.4^\circ(0.3^\circ)$
O_1-O_3	2.400(6)	$\sphericalangle O_1-S-O_3$	$108.7^\circ(0.3^\circ)$
O_1-O_4	2.412(6)	$\sphericalangle O_1-S-O_4$	$109.7^\circ(0.3^\circ)$
O_2-O_3	2.415(5)	$\sphericalangle O_2-S-O_3$	$110.1^\circ(0.3^\circ)$
O_2-O_4	2.411(6)	$\sphericalangle O_2-S-O_4$	$110.1^\circ(0.3^\circ)$
O_3-O_4	2.398(6)	$\sphericalangle O_3-S-O_4$	$109.9^\circ(0.3^\circ)$
Mean value:	2.407(3)	Mean value:	$109.5^\circ(0.1^\circ)$

Table 5 cont.

(d) Hydrogen bonds

$O_{w1}-O_1$	2.748(7)		
$O_{w1}-O_2$	2.733(7)	$\sphericalangle O_1-O_{w1}-O_2$	121.4°(0.2°)
$O_{w2}-O_2$	2.785(7)		
$O_{w2}-O_4$	2.901(6)	$\sphericalangle O_2-O_{w2}-O_4$	123.3°(0.2°)
$O_{w3}-O_1$	2.857(6)		
$O_{w3}-O_2$	2.928(6)	$\sphericalangle O_1-O_{w3}-O_2$	114.0°(0.2°)
$O_{w4}-O_3$	2.803(6)		
$O_{w4}-O_{w7}$	2.725(6)	$\sphericalangle O_3-O_{w4}-O_{w7}$	110.5°(0.2°)
$O_{w5}-O_4$	2.950(6)		
$O_{w5}-O_{w7}$	2.714(7)	$\sphericalangle O_4-O_{w5}-O_{w7}$	102.6°(0.2°)
$O_{w6}-O_1$	2.821(6)		
$O_{w6}-O_3$	2.770(6)	$\sphericalangle O_1-O_{w6}-O_3$	121.2°(0.2°)
$O_{w7}-O_4$	2.766(6)		
$O_{w7}-O_{w6}$	3.027(7)	$\sphericalangle O_4-O_{w7}-O_{w6}$	145.0°(0.2°)
Mean value:	2.82	Mean value:	120°

Table 6

$FeSO_4 \cdot 7H_2O$, root-mean-square thermal displacements along principal axes (in Å)

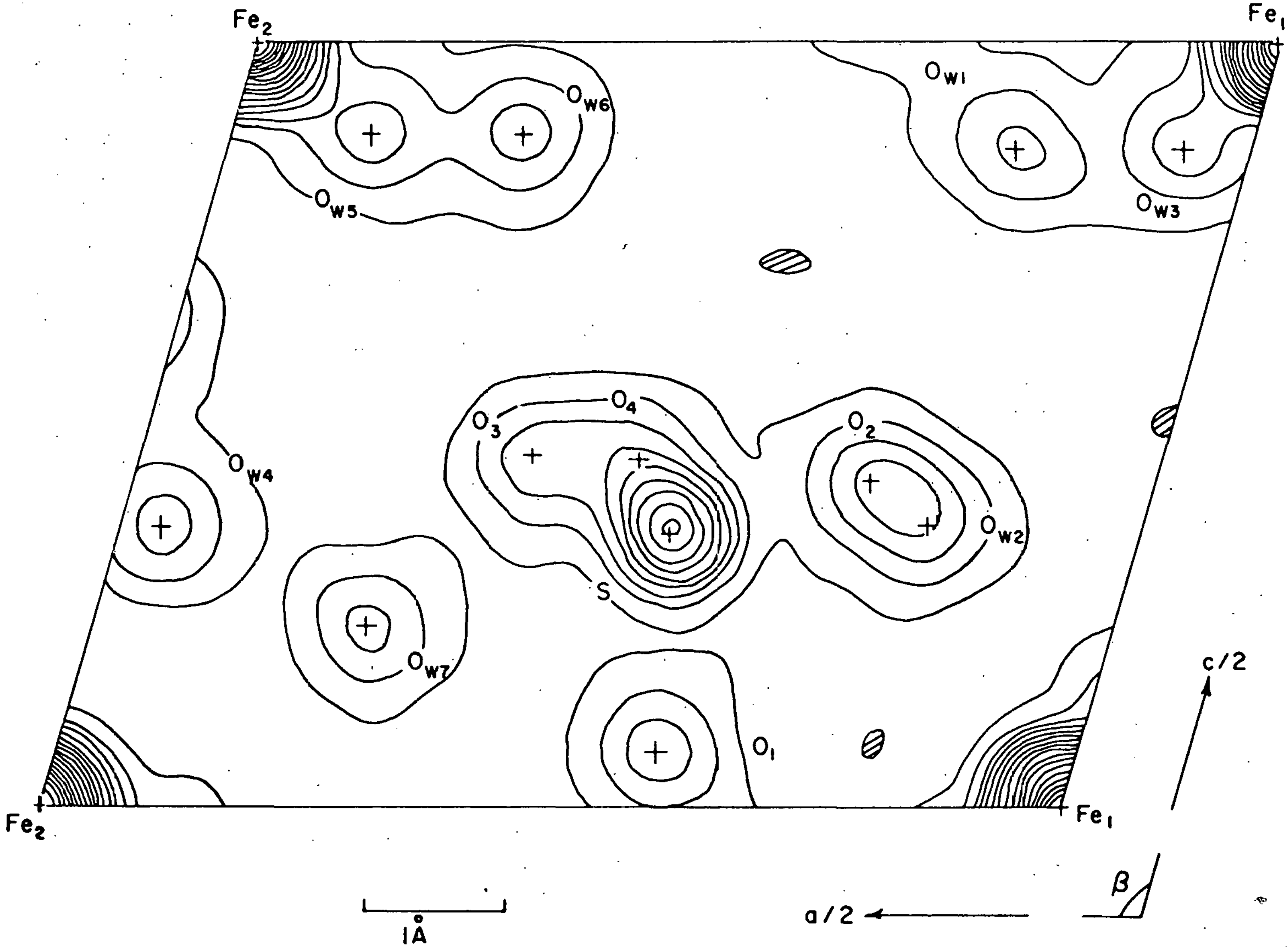
Atom	Axis 1	Axis 2	Axis 3
Fe_1	0.130(2)	0.135(2)	0.150(2)
Fe_2	0.127(2)	0.135(2)	0.154(2)
S	0.128(2)	0.129(3)	0.141(3)
O_1	0.133(7)	0.154(6)	0.194(7)
O_2	0.125(7)	0.164(8)	0.210(6)
O_3	0.110(8)	0.194(6)	0.210(6)
O_4	0.121(8)	0.181(7)	0.196(7)
O_{w1}	0.115(10)	0.180(8)	0.309(7)
O_{w2}	0.154(8)	0.180(6)	0.227(7)
O_{w3}	0.138(7)	0.163(6)	0.200(8)
O_{w4}	0.134(6)	0.167(5)	0.212(6)
O_{w5}	0.154(7)	0.169(6)	0.228(6)
O_{w6}	0.140(7)	0.166(7)	0.228(8)
O_{w7}	0.147(9)	0.191(6)	0.215(7)

Figure 1. Electron density projections of $FeSO_4.7H_2O$. F_{000} included. Negative areas shaded. Atomic positions marked by crosses. (a) Projection parallel to [010], contour interval 2.5e.$Å^{-2}$. Convergence factor with B=2.0 $Å^2$ applied to the F_o. (b) Projection parallel to [001]. Convergence factor B=1.6 $Å^2$. Contour interval 3.0e.$Å^{-2}$.

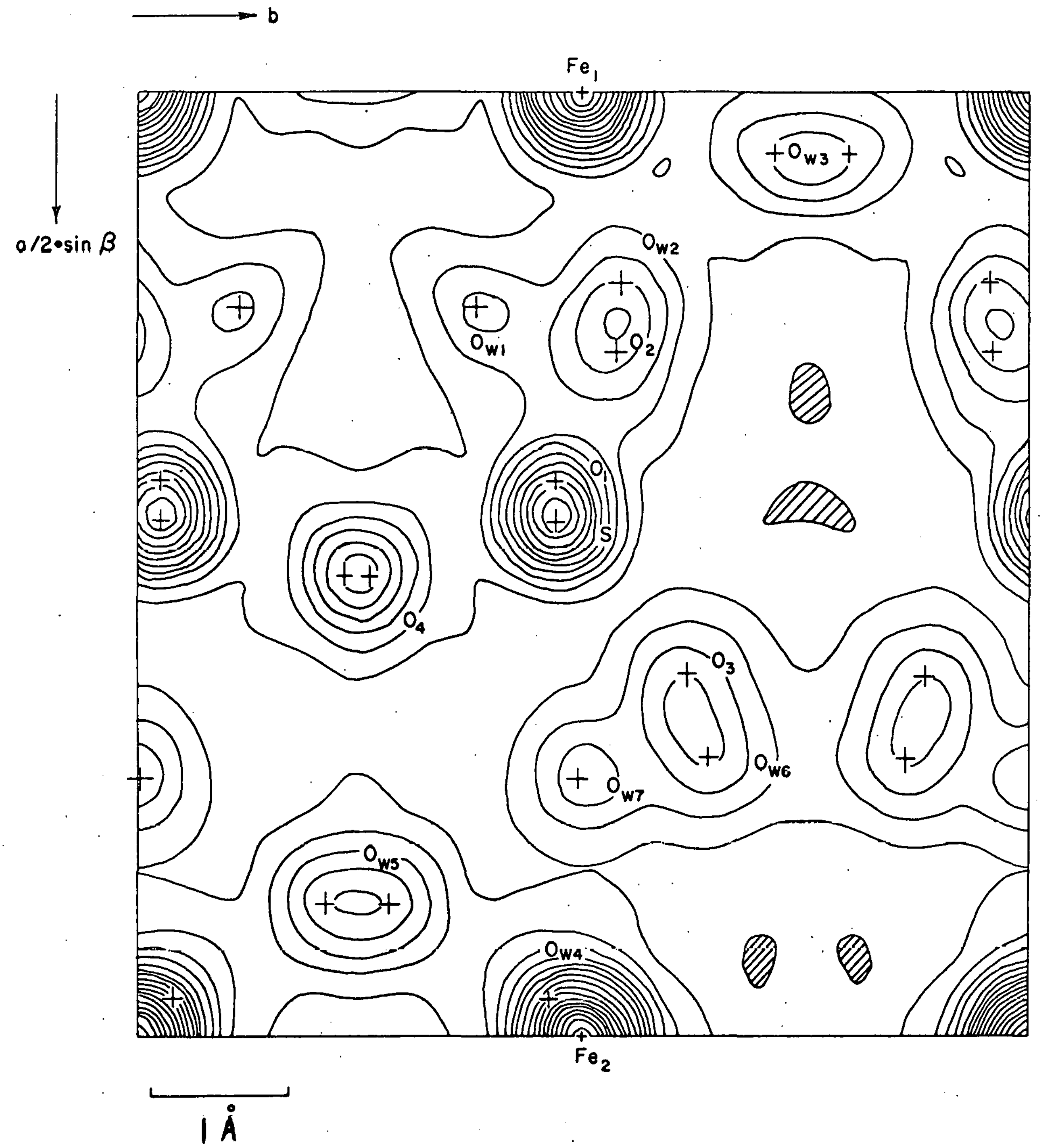
Figure 2. $FeSO_4.7H_2O$, difference density projection parallel to [010] computed with $F_o-F_{c(heavy)}$, thus showing the contributions of the hydrogen atoms only. 70 $F_{h0\ell}$ with $\sin\vartheta/\lambda<0.38$ $Å^{-1}$ were used in the summation. Atomic positions marked by crosses; water oxygens connected by lines to their corresponding hydrogen atoms. Negative areas shaded, negative contours omitted, first drawn contour is the zero contour. Largest negative peak is -0.50e.$Å^{-2}$. Contour interval 0.09e.$Å^{-2}$.

Figure 3. Hydrogen bonding scheme of $FeSO_4.7H_2O$.

Figure 4. $FeSO_4.7H_2O$, schematic projection parallel to [010] illustrating the hydrogen bonding system. The numbers in or near the circles respresenting the atoms are the y-parameters of these atoms. Hydrogen bonds shown by dashed lines.



1(a)



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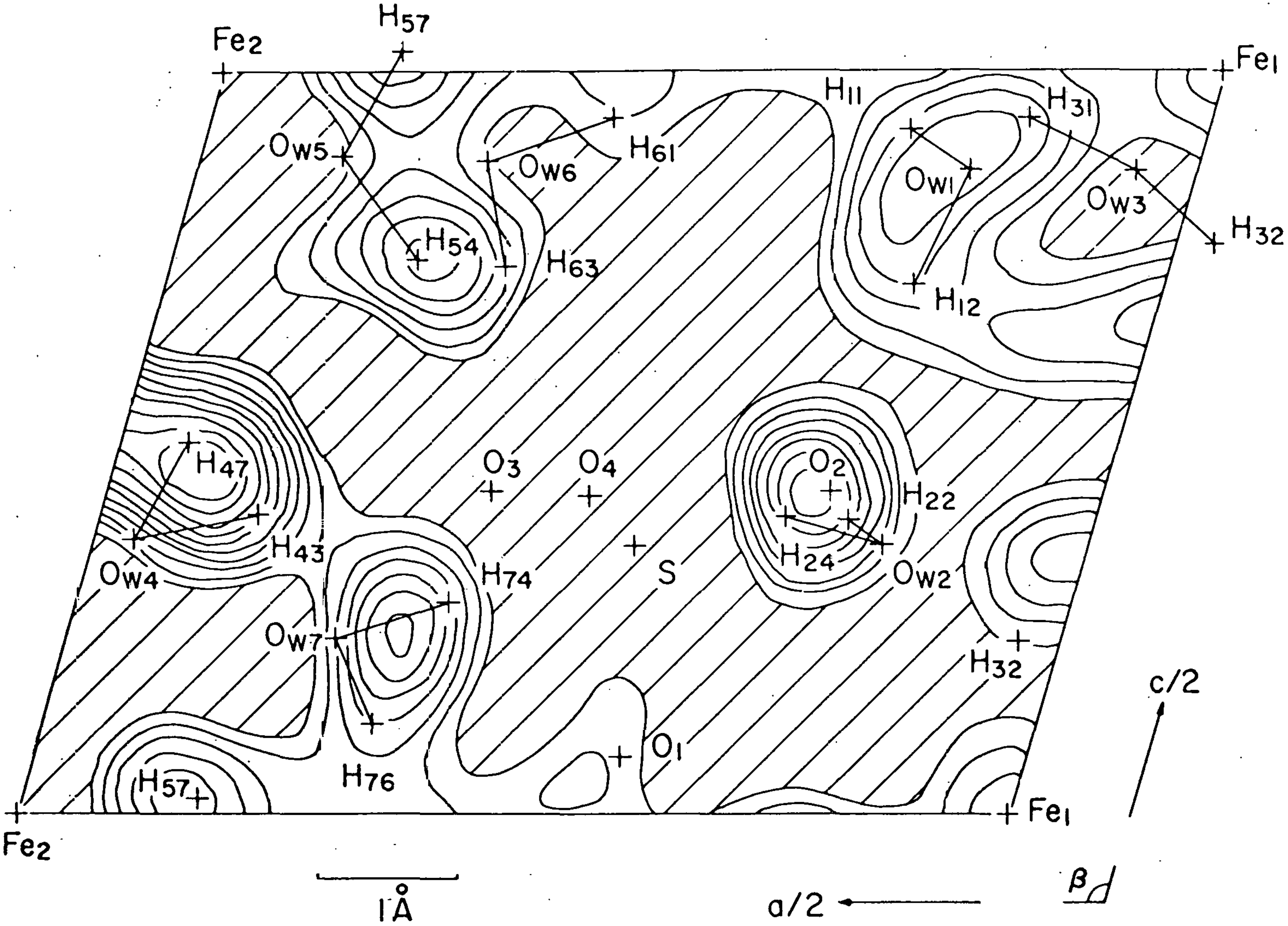
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Fe1
H11
H31
Ow5
H61
Ow6
Ow1
Ow3
H32
H54
H63
H12
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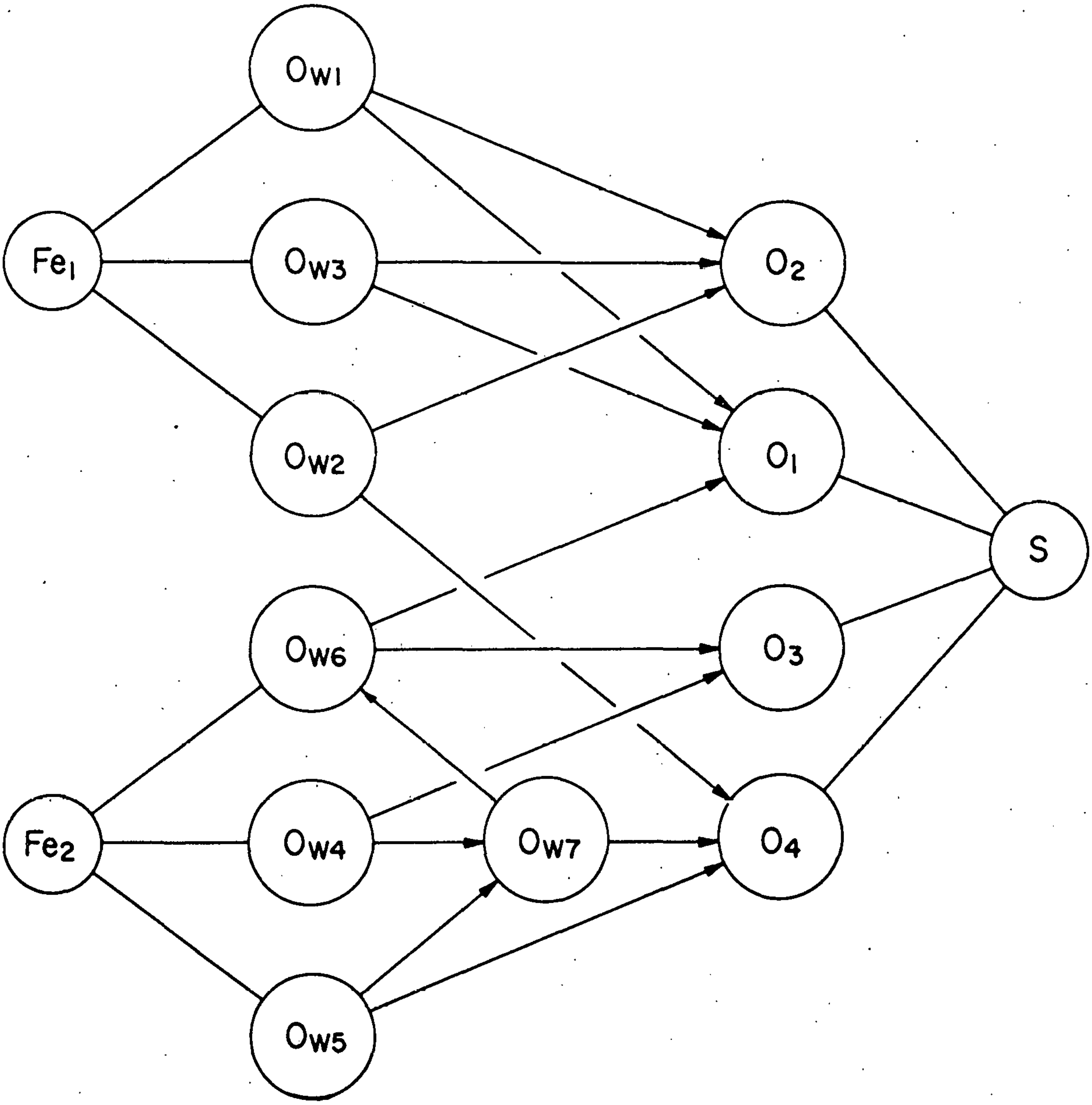
O2
H47
H22
H43
S
H24
Ow2
Ow4
H74
Ow7
H32
c/2
O1
H57
H76
Fe1
Fe2
1Å
a/2
β

2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S
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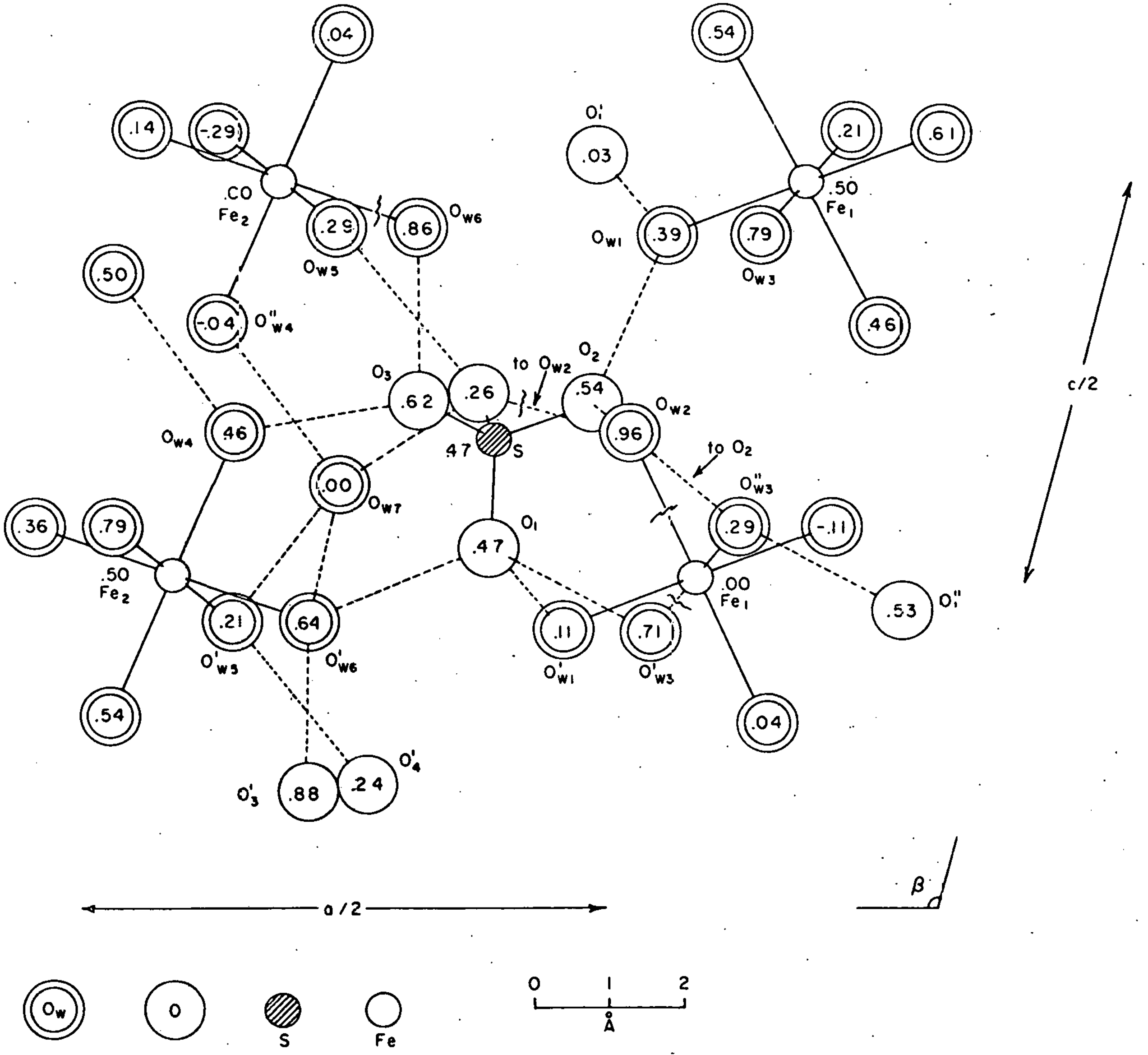
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4