Table 1. *Positional and thermal parameters for* Te

Wyckoff position 3(a) $(x, 0, \frac{1}{3}, 0, x, \frac{2}{3}; -x, -x, 0)$ gives the restriction for thermal factors $u_{13} = \frac{1}{2} u_{23}$ and $u_{12} = \frac{1}{2} u_{22}$.

$x = -0.2636\,(1)$ $u_{11} = 0.0193\,(6)\,\text{Å}^2$ $B_{eq} = 1.29\,(3)\,\text{Å}^2$
                  $u_{22} = 0.0179\,(5)$
                  $u_{33} = 0.0113\,(6)$
                  $u_{23} = -0.0020\,(1)$

Table 2. *Short contacts* (Å) *to different symmetry-related sites*

Type	Distance	Symmetry code
1	$2 \times 2.8345\,(8)$	$0, x, \frac{2}{3}$
		$-x, -x, 0$
2	$4 \times 3.4912\,(8)$	$-1, x, \frac{2}{3}$
		$0, x+1, \frac{2}{3}$
		$-x-1, -x-1, 0$
		$-x-1, -x, 0$
3	$2 \times 4.441\,(1)$	$0, x, -\frac{1}{3}$
		$-x, -x, 1$
4	$6 \times 4.456\,(1)$	$x-1, -1, \frac{1}{3}$
		$x-1, 0, \frac{1}{3}$
		$x, -1, \frac{1}{3}$
		$x, 1, \frac{1}{3}$
		$x+1, 0, \frac{1}{3}$
		$x+1, 1, \frac{1}{3}$
5	$4 \times 4.886\,(1)$	$-1, x, -\frac{1}{3}$
		$0, x+1, -\frac{1}{3}$
		$-x, -1, -x-1, 1$
		$-x-1, -x, 1$

Fig. 1. Coordination around the Te atom.

Type of contact:
1
2
3
4
5

visualized by Donohue (1982). The present study is of higher accuracy than the previous structure determinations, as they were based on only a few (13 to 22) reflexions in two dimensions.

Related literature. The structure of tellurium was first determined by Bradley (1924), then by Cherin & Unger (1967), who published values $x = 0.2633$ (5), contact lengths 2·835 (2), 3·495 (3) and 4·445 (1) Å and bond angle 103·2 (1)°. The effect of pressure on the structure was studied by Keller, Holzapfel & Schulz (1977). The structure description in terms of infinite helices parallel to **c**, with three atoms per turn of the helix, was
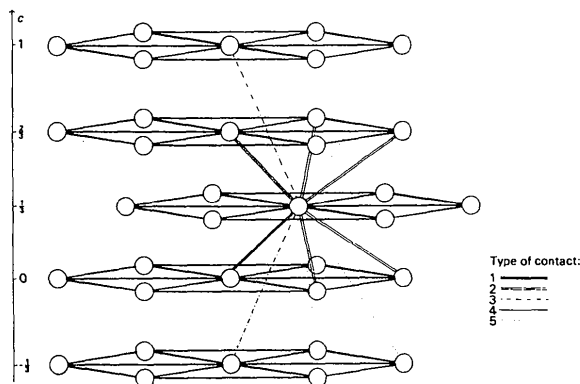
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The Structure of (ND4)2Fe(SO4)2.6D2O at 4·3 K by Neutron Diffraction

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Abstract. Di($^2H_2$)ammonium hexa[($^2H_4$)aqua]iron(II) disulfate, $[ND_4]_2[Fe(D_2O)_6](SO_4)_2$, $M_r = 410.8$, monoclinic, $P2_1/a$, $a = 9.167$ (4), $b = 12.405$ (7), $c = 6.305$ (3) Å, $\beta = 106.71$ (2)°, $V = 687$ (1) Å^3, $Z = 2$, $D_m = 1.96$ (1) (295 K), $D_x = 1.99$ Mg m^{-3}, neutrons, $\lambda = 1.176$ Å, $\mu \simeq 20$ m^{-1} (incoherent H), $F(000) = 465$ fm, $T = 4.33$ K, $R(F) = 0.024$ for 1392 reflections, $\chi = 2.92$, deuteration 92·2 (4)%. The structure is

very similar to the hydrogen-bonded network in the other isomorphous ammonium Tutton salts studied at 4 K (V, Mn, Ni). The Fe—O_6 octahedron has small but significant deviations from octahedral symmetry.

Experimental. A. R. grade ferrous ammonium sulfate twice recrystallized from hot D_2O, large crystals readily grown by slow evaporation of D_2O solution. Crystals aligned by optical goniometry, density by flotation. Two pale-green tabular crystals mounted (2·4 × 2·7 × 3·5 mm, b axis vertical, 1·5 × 3·5 × 2·8 mm c^* axis vertical) in an He-gas-flow cryostat on the D15 lifting-arm normal-beam Weissenberg neutron diffractometer at the Institut Laue–Langevin. Cell parameters from 17 (b) and 11 (c^*) reflections separated in angle, refining both sets to a mutually consistent cell. 1771 reflections measured in the b orientation and 732 in c^*, ω scan, $(\sin\theta)/\lambda < 0.75 \text{ Å}^{-1}$, $|h| < 14$, $|k| < 18$, $|l| < 9$, some omitted due to limits on lifting-arm angle and the use of normal-beam diffraction geometry. The 4 standards collected every 40 reflections showed no systematic time variation. Scaled squares of structure-factor moduli obtained using profile analysis program *COLL5N* (Lehmann & Larsen, 1974; Allibon & Lehmann, 1982), merged, $R_{int} = 0.015$ and 0·027, unique sets 852 [824 $I > 3\sigma(I)$] for b and 540 [502 $I > 3\sigma(I)$] for c^*. Absorption correction unnecessary. Data refined using *ASRED* (Figgis, Reynolds & Williams, 1980), starting with the V^{II} structure (Deeth, Figgis, Forsyth, Kucharski & Reynolds, 1988). Both data sets refined together on $|F|^2$, weights $w = \sigma(|F|^2)^{-2}$, with separate scale and isotropic type-II extinction (Becker & Coppens, 1974, 1975), and multiple scattering corrections, but common anisotropic thermal and positional parameters. Deuterium-site scattering length refined to estimate deuteration. Other scattering lengths from Koester & Steyerl (1980). Largest correction factor for extinction was 1·5 (b) and 2·4 (c^*). Refinement converged $[(\Delta/\sigma)_{max} < 0.05]$ to $R(F^2)=0.032$, $wR(F^2) = 0.051$, $\chi = 2.92$, $R(F) = 0.024$. Refinement of the b and c^* data separately produced no significant changes in derived parameters. Refinement of NH_4 and H_2O deuteration separately gave insignificantly different values [92·7 (4) and 91·7 (4)%] as expected from exchange rates in solution. Deuterium scattering-length correlation coefficients with other parameters are small (< 0·35). The correlation is expected to be noticeably lessened, relative to high-temperature experiments, by the low thermal motion at 4·3 K. Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1.† The principal interatomic distances and

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† Lists of least-squares planes for the $|Fe(OD_2)_6|$ fragment, anisotropic thermal parameters and F^2(obs) and F^2(calc) have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51657 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic fractional positional coordinates* (× 10^4) *and equivalent isotropic thermal parameters* ($\text{Å}^2 \times 10^4$)

$$U_{eq} = \tfrac{1}{3}(U_{11} + U_{22} + U_{33}).$$

	x	y	z	U_{eq}
Fe	0	0	0	24
S	4126 (2)	1300 (3)	7349 (3)	17
O(3)	4209 (1)	2217 (1)	5868 (1)	48
O(4)	5526 (1)	662 (1)	7807 (1)	54
O(5)	2802 (1)	622 (1)	6190 (1)	43
O(6)	3898 (1)	1726 (1)	9430 (1)	49
O(7)	1799 (1)	1075 (2)	1704 (1)	58
O(8)	−1643 (1)	1171 (1)	298 (2)	54
O(9)	−6 (1)	−707 (2)	3023 (2)	58
N	1362 (1)	3403 (1)	3518 (1)	53
H(11)	704 (1)	3299 (2)	1922 (2)	188
H(12)	2295 (1)	2916 (2)	3883 (2)	189
H(13)	698 (1)	3221 (2)	4534 (2)	176
H(14)	1700 (1)	4199 (2)	3729 (2)	175
H(15)	2268 (1)	878 (2)	3245 (2)	153
H(16)	2606 (1)	1174 (2)	1005 (2)	178
H(17)	−2684 (1)	1001 (2)	−589 (2)	147
H(18)	−1449 (1)	1912 (2)	−63 (2)	152
H(19)	−956 (1)	−610 (2)	3411 (2)	179
H(20)	250 (1)	−1473 (2)	3285 (2)	154

Table 2. *Bond lengths* (Å) *and angles* (°)

Fe—O(7)	2·152 (2)	O(7)—Fe—O(8)	90·4 (1)
Fe—O(8)	2·140 (2)	O(7)—Fe—O(9)	90·4 (1)
Fe—O(9)	2·100 (2)	O(8)—Fe—O(9)	91·1 (1)
O(7)—H(15)	0·974 (2)	Fe—O(7)—H(15)	113·5 (2)
O(7)—H(16)	0·972 (2)	Fe—O(7)—H(16)	115·2 (2)
O(8)—H(17)	0·980 (2)	Fe—O(8)—H(17)	113·4 (2)
O(8)—H(18)	0·975 (3)	Fe—O(8)—H(18)	116·0 (1)
O(9)—H(19)	0·977 (2)	Fe—O(9)—H(19)	114·3 (1)
O(9)—H(20)	0·981 (3)	Fe—O(9)—H(20)	119·6 (1)
S—O(3)	1·488 (3)	H(15)—O(7)—H(16)	108·0 (1)
S—O(4)	1·463 (2)	H(17)—O(8)—H(18)	106·4 (2)
S—O(5)	1·485 (3)	H(19)—O(9)—H(20)	105·3 (2)
S—O(6)	1·484 (2)	O(3)—S—O(4)	109·7 (1)
N—H(11)	1·022 (2)	O(3)—S—O(5)	107·8 (1)
N—H(12)	1·018 (2)	O(3)—S—O(6)	109·2 (2)
N—H(13)	1·028 (2)	O(4)—S—O(5)	109·7 (2)
N—H(14)	1·032 (2)	O(4)—S—O(6)	110·9 (1)
		O(5)—S—O(6)	109·5 (1)
H(15)—O(5)	1·807 (2)	H(11)—N—H(12)	111·5 (2)
H(16)—O(6)	1·880 (2)	H(11)—N—H(13)	107·3 (1)
H(17)—O(4)	1·714 (2)	H(11)—N—H(14)	108·6 (2)
H(18)—O(6)	1·766 (3)	H(12)—N—H(13)	109·7 (2)
H(19)—O(5)	1·781 (2)	H(12)—N—H(14)	109·8 (1)
H(20)—O(3)	1·738 (3)	H(13)—N—H(14)	109·9 (2)
H(11)—O(6)	1·927 (2)		
H(13)—O(3)	1·877 (2)		
H(14)—O(5)	1·819 (3)		

angles obtained using the *XTAL* system (Stewart & Hall, 1986) are given in Table 2.

Related literature. The structure is consistent with that of the V^{II} (Deeth *et al.*, 1988), Ni^{II} (Fender, Figgis & Forsyth, 1986a), and Mn^{II} (Fender, Figgis, Forsyth, Reynolds & Stevens, 1986) ammonium Tutton salts measured at helium temperatures by neutron diffraction. There are $M(OD_2)$ octahedra and ND_4 and SO_4 tetrahedra, all linked by a complete network of strong hydrogen bonds. These are a precursor, as in this

case, to the spin density measurements by polarized neutron diffraction experiments (Deeth, Figgis, Forsyth, Kucharski & Reynolds, 1989; Fender, Figgis & Forsyth, 1986b; Fender, Figgis, Forsyth, Reynolds & Stevens, 1986). The ferrous Tutton salt structure has been determined at room temperature by X-ray film methods (Montgomery, Chastain, Natt, Witkowska & Lingafelter, 1967).

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β-Hydroquinone Xenon Clathrate

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Abstract. $3C_6H_4(OH)_2.xXe$ ($x = 0.866$), $M_r = 444.03$, rhombohedral, $R\bar{3}$ (obverse setting), $a = 16.610$ (3), $c = 5.524$ (1) Å, $V = 1319.8$ (4) Å^3, $Z = 3$, $D_m = 1.67$ (1), $D_x = 1.68$ g cm^{-3} (for $x = 0.866$), graphite-monochromatized Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å, $\mu = 17.88$ cm^{-1}, $F(000) = 660.79$, $T = 298$ K, $R = 0.0242$, $wR = 0.0253$ for 594 unique reflections and 54 parameters. The encaged xenon atom occupies a near-spherical cavity (ca 4.8 Å diameter), formed by six interlocking C_6H_4 moieties and two hydrogen-bonded $[OH]_6$ hexagonal rings above and below the plane of the guest atom.

Experimental. The complex was isolated from an aqueous solution of hydroquinone with a modification of the procedure described by Powell (1950). The salient change is an increase in the initial pressure of xenon from 14 to 21 atm (1 atm = 1.01×10^5 Pa). The density of the crystals was determined by flotation in $CCl_4/CHBr_3$ solution. Crystal chosen was ground to a sphere 0.3 mm in diameter with a Nonius crystal grinder and was sealed in a Lindemann capillary. Unit-cell parameters determined from the least-squares refinement of positional angles for 15 strong independent reflections in the range $21.8° < 2\theta < 31.2°$ on a Nicolet $P2_1$ diffractometer at 298 K with graphite-monochromated Mo $K\alpha$ radiation ($\lambda = 0.71069$ Å). Rhombohedral, space group $R\bar{3}$ (No. 148) from systematic absences: $-h+k+l \neq 3n$. Intensities of 4974 reflections with $2\theta \leq 55°$, $-15 \leq h \leq 12$, $0 \leq k \leq 15$, $-7 \leq l \leq 7$, were measured with a θ–2θ scan technique. Range of scan rates used was 5.86 to 29.30° min^{-1} in 2θ. The total background time to scan time is 1:1. The intensities of two standard reflections ($3\bar{8}1$: 1.59%, and $\bar{1}\bar{1}31$; 1.48%) were monitored every 48 reflections and showed no sign of crystal decomposition or instrument instability. The data were averaged to give 594 independent reflections; $R_{int} = 0.0157$. Reflections with $3\sigma I \geq I \geq -3\sigma I$ were treated by the method of French & Wilson (1978). Corrections were made for Lorentz–polarization effects but not for absorption. The coordinates of the xenon atom were found from a three-dimensional Patterson synthesis, and a series of full-matrix least-squares