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Structure of Hexaaquairon(II) Bis{tris[1,3-dimethyl-2,4,5,6(1*H*,3*H*)-pyrimidinetetrone 5-oximato]ferrate(II)} Dodecahydrate, $[Fe(H_2O)_6]^{2+}.2[Fe(C_6H_6N_3O_4)_3]^{-}.12H_2O$

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Abstract. $M_r = 1596{\cdot}6$, hexagonal, $R\bar{3}$, $a = 16{\cdot}329\ (3)$, $c = 21{\cdot}275\ (9)$ Å, $V = 4913\ (2)$ Å^3, $Z = 3$, $D_x = 1{\cdot}62$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu(\text{Mo } K\alpha) = 0{\cdot}766$ mm^{-1}, $T = 298$ K, $F(000) = 2484$, $R = 0{\cdot}049$ for 2078 observed reflexions. The two Fe atoms are located in special positions along the threefold axis, and exhibit octahedral coordination. An array of cations, each lying between two anions, is repeated along the threefold axis. Coordinated and uncoordinated water molecules contribute to structural packing through hydrogen bonds.

Introduction. Relatively little work has been carried out on complexes of organic nitroso ligands with transition-metal ions, and this has been almost entirely concerned with ligands of the *o*-nitrosophenolate or isonitroso ketone type.

The available structural data are about salts of the type $M^+[FeV_3]^-.xH_2O$ where M^+ is an alkali-metal cation and V the violurate anion (Raston & White, 1976).

We now report the crystal structure of the title compound, prepared by J. D. López González and co-workers, Department of Inorganic Chemistry, University of Granada (Spain).

Experimental. Black metallic crystal, $0{\cdot}7 \times 0{\cdot}4 \times 0{\cdot}3$ mm. Enraf–Nonius CAD-4 F automatic diffractometer. Cell dimensions by least-squares fitting of the θ values of 25 reflexions. $\omega/2\theta$ scans. No appreciable drop in intensity of three standard reflections, checked every hour. 6050 reflexions within $1° < \theta < 30°$ and hkl range 0,0,−29 to 22,22,29 collected; 3417 unique reflections; $R_{int} = 0{\cdot}039$; 2078 considered observed $[I > 2\sigma(I)]$. Lorentz–polarization correction; no absorption correction, $\mu R = 0{\cdot}54$. Scattering factors for neutral atoms and anomalous-dispersion corrections for

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Table 1. *Atomic coordinates and isotropic thermal parameters*

For non-hydrogen atoms $U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a^*_i a^*_j \mathbf{a}_i.\mathbf{a}_j$. For H atoms U_{iso} values are listed.

	x	y	z	U_{eq}/U_{iso}(Å² × 10³)
Fe(1)	0·0000	0·0000	0·0000	34·9 (8)
O(11)	0·0401 (3)	0·1219 (3)	0·0571 (2)	47 (2)
Fe(2)	0·0000	0·0000	0·27070 (5)	17·1 (8)
O(21)	0·1224 (2)	0·1156 (2)	0·1763 (1)	30·9 (9)
O(22)	0·1772 (2)	0·3077 (2)	0·1743 (2)	40 (2)
O(23)	0·0493 (3)	0·3942 (2)	0·3324 (2)	42 (2)
O(24)	−0·0256 (2)	0·0852 (2)	0·3236 (1)	23 (1)
N(21)	0·0741 (2)	0·1108 (2)	0·2239 (1)	21 (1)
N(22)	0·1203 (2)	0·3519 (2)	0·2574 (2)	25 (1)
N(23)	0·0086 (2)	0·2389 (2)	0·3288 (2)	24 (1)
C(21)	0·0755 (3)	0·1891 (3)	0·2469 (2)	22 (1)
C(22)	0·1278 (3)	0·2838 (3)	0·2216 (2)	25 (2)
C(23)	0·0581 (3)	0·3321 (3)	0·3069 (2)	26 (2)
C(24)	0·0172 (3)	0·1681 (3)	0·3010 (2)	21 (1)
C(25)	0·1658 (4)	0·4495 (3)	0·2330 (3)	34 (2)
C(26)	−0·0573 (4)	0·2176 (4)	0·3818 (2)	40 (2)
O(1)	−0·0840 (3)	0·1834 (3)	0·0876 (2)	54 (2)
O(2)	−0·0707 (3)	0·2784 (3)	0·2015 (2)	50 (2)
H(111)	0·074 (5)	0·127 (5)	0·086 (3)	48
H(112)	0·011 (6)	0·138 (6)	0·068 (4)	48
H(11)	−0·086 (5)	0·214 (5)	0·058 (3)	51
H(12)	−0·129 (6)	0·147 (6)	0·093 (4)	51
H(21)	−0·073 (6)	0·314 (5)	0·192 (4)	47
H(22)	−0·081 (6)	0·247 (5)	0·178 (4)	47
H(251)	0·126 (4)	0·461 (4)	0·205 (3)	33
H(252)	0·194 (4)	0·491 (5)	0·265 (3)	33
H(253)	0·217 (4)	0·463 (4)	0·197 (3)	33
H(261)	−0·024 (5)	0·248 (5)	0·420 (3)	40
H(262)	−0·111 (5)	0·224 (5)	0·371 (3)	40
H(263)	−0·080 (4)	0·152 (5)	0·407 (3)	40

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

E.s.d.'s are *ca* 0·005 Å for lengths and 0·3° for angles.

Fe(1)–O(11)	2·136	N(23)–C(24)	1·367
Fe(2)–N(21)	1·881	N(23)–C(26)	1·475
Fe(2)–O(24)	1·990	C(21)–C(22)	1·446
O(24)–C(24)	1·267	C(21)–C(24)	1·422
N(21)–O(21)	1·262	C(22)–N(22)	1·403
N(21)–C(21)	1·358	C(22)–O(22)	1·225
N(22)–C(23)	1·384	C(23)–N(23)	1·399
N(22)–C(25)	1·476	C(23)–O(23)	1·220
O(24)–Fe(2)–N(21)	83·4	N(21)–C(21)–C(24)	111·7
Fe(2)–O(24)–C(24)	109·6	N(21)–C(21)–C(22)	126·6
Fe(2)–N(21)–O(21)	124·5	C(22)–C(21)–C(24)	121·7
O(21)–N(21)–C(21)	121·0	O(22)–C(22)–C(21)	126·1
Fe(2)–N(21)–C(21)	114·5	C(21)–C(22)–N(22)	114·0
C(25)–N(22)–C(22)	118·0	O(22)–C(22)–N(22)	119·9
C(23)–N(22)–C(22)	124·9	O(23)–C(23)–N(23)	120·8
C(23)–N(22)–C(25)	115·7	N(23)–C(23)–N(22)	117·7
C(26)–N(23)–C(24)	120·2	O(23)–C(23)–N(22)	121·4
C(23)–N(23)–C(24)	121·9	N(23)–C(24)–C(21)	119·1
C(23)–N(23)–C(26)	117·9	O(24)–C(24)–C(21)	120·6
		O(24)–C(24)–N(23)	120·3

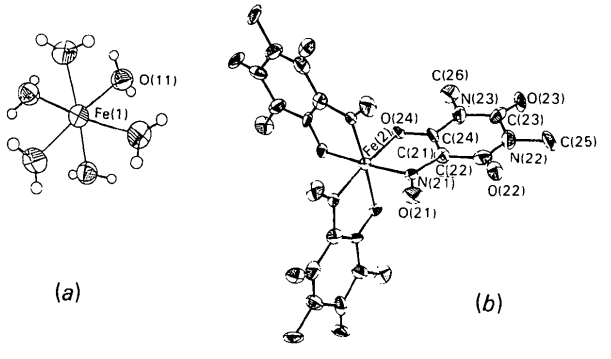

Fig. 1. Coordination polyhedra of (*a*) the $[Fe(H_2O)_6]^{2+}$ cation, and (*b*) the $[Fe(C_6H_6N_3O_4)_3]^{-}$ anion.

Fe from *International Tables for X-ray Crystallography* (1974). Structure solved by three-dimensional Patterson and Fourier syntheses allowing the location of all non-hydrogen atoms. Anisotropic full-matrix least-squares refinement with F's and unit weights; $R = 0{\cdot}058$. A difference synthesis calculated with those reflexions having $\sin\theta/\lambda < 0{\cdot}5$ Å^{-1} showed all H atoms as the highest peaks. Final refinement with fixed isotropic temperature factors and fixed coordinates for the H atoms of the methyl groups gave $R = 0{\cdot}049$. Maximum and average Δ/σ 0·5613 and 0·0267 respectively. Residual electron density in final difference synthesis within $\pm 0{\cdot}3$ e Å^{-3}. Programs: *XRAY*70 (Stewart, Kundell & Baldwin, 1970), *ORTEP* (drawings) (Johnson, 1965).

Discussion. Atomic parameters are listed in Table 1.* Table 2 lists the bond lengths and angles.

The two Fe atoms are located in special positions along the threefold axis, and exhibit octahedral coordination; Fe(1) lies on a centre of symmetry [Wyckoff position 3(*a*)].

The structure consists of an array of $[Fe(H_2O)_6]^{2+}$ cations (Fig. 1*a*), each lying between two $[Fe(C_6H_6N_3O_4)_3]^{-}$ anions (Fig. 1*b*), repeated along the threefold axis. There are also free molecules of water.

Water molecules of both types, *i.e.* coordinated and uncoordinated, contribute to structural packing through hydrogen bonds. One of these hydrogen bonds, asymmetrically bifurcated, is similar to those found by Ruiz-Valero, Monge, Gutiérrez-Puebla & Gutiérrez-Rios (1983) in $[Ag(C_6H_6N_3O_4)(C_6H_7N_3O_4)]$.

Thanks are due to the staff of the Computing Center of JEN (Madrid) for the facilities provided on the Univac 1100/80 computer.

* Lists of structure factors, anisotropic thermal parameters, hydrogen-bond parameters and bond lengths and angles involving H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39196 (27 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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