

S = 1.10
3569 reflections
286 parameters
H atoms: see below
 $w = 1/[\sigma F^2 + (0.02F)^2 + 0.10]$

Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Ülkü, D., Ercan, F., Atakol, O. & Dinçer, F. N. (1997). *Acta Cryst.* C53, 1056–1057.

Ülkü, D., Tahir, M. N., Atakol, O. & Nazır, H. (1997). *Acta Cryst.* C53, 872–874.

Table 2. Selected geometric parameters (Å, °)

Ni···Mn	3.133 (2)	O1—C1	1.309 (2)
Ni—O1	2.035 (2)	O2—C17	1.320 (3)
Ni—O2	2.017 (2)	O3—C18	1.251 (3)
Ni—O4	2.046 (2)	O4—C18	1.247 (4)
Ni—O5	2.184 (2)	O5—C20	1.226 (3)
Ni—N1	2.016 (2)	N1—C10	1.465 (3)
Ni—N2	2.036 (2)	C9—C10	1.514 (3)
Mn—O1	2.163 (1)	C11—C12	1.452 (4)
Mn—O2	2.166 (2)	C18—C19	1.510 (5)
Mn—O3	2.194 (2)		
O1—Ni—O2	82.98 (7)	O5—Ni—N2	84.79 (8)
O1—Ni—O4	93.66 (8)	N1—Ni—N2	97.49 (9)
O1—Ni—O5	88.22 (8)	O1—Mn—O2	76.66 (6)
O1—Ni—N1	172.58 (8)	O1—Mn—O3	85.95 (6)
O2—Ni—O4	95.10 (7)	O2—Mn—O3	87.74 (7)
O2—Ni—O5	91.55 (7)	Ni—O1—Mn	96.49 (6)
O2—Ni—N2	171.23 (8)	Ni—O1—C1	123.4 (2)
O4—Ni—N1	90.23 (8)	Mn—O1—C1	135.9 (2)
O4—Ni—N2	88.77 (8)	Ni—O2—Mn	96.95 (7)
O5—Ni—N1	88.64 (8)	Mn—O3—C18	130.5 (2)

Acta Cryst. (1998). C54, 1270–1272

Triaqua(pyridine-2,6-dipicolinato-*O,N,O'*)-iron(III) Bis(pyridine-2,6-dipicolinato-*O,N,O'*)iron(III) Hexahydrate: an Unusual Ion-Pair Compound

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All non-H atoms were refined with anisotropic displacement parameters. The H-atom positions were taken from difference maps. A riding model was used for all H atoms, with $U_{iso}(H) = 1.3U_{eq}(C)$ of the parent atom.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1993). Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SIMPEL* in *MolEN*. Program(s) used to refine structure: *LSFM* in *MolEN*. Molecular graphics: *ORTEPII* (Johnson, 1976) in *MolEN*. Software used to prepare material for publication: *MolEN*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1111). Services for accessing these data are described at the back of the journal.

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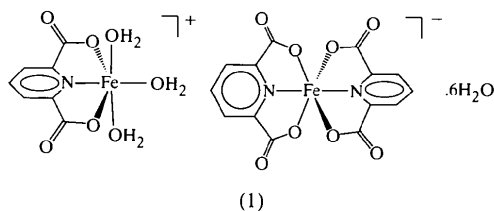
Abstract

The reaction of dipicolinic acid (2,6-pyridinedicarboxylic acid) with FeCl₃ results in the unusual title ion-pair compound, [Fe(C₇H₃NO₄)(H₂O)₃][Fe(C₇H₃NO₄)₂].6H₂O. Each Fe atom has an octahedral geometry. The dipicolinate ligands are nearly planar, with the two in the anion being perpendicular to each other and displaying a dihedral angle of 93.74 (6)°. The Fe—O(carboxylate) bond distances are comparable and range from 2.008 (2) to 2.028 (2) Å, while the Fe—N distances are 2.059 (2), 2.054 (2) and 2.054 (2) Å. The Fe—O(water) distances are 1.971 (2), 1.987 (2) and 2.004 (2) Å. The cation, anion and water molecules are involved in an extensive three-dimensional hydrogen-bonding network, with O···O distances ranging from 2.36 (2) to 2.953 (3) Å and angles ranging from 129 to 177°.

Comment

The reaction of iron with dipicolinic acid (2,6-pyridinedicarboxylic acid, dipicH₂) is more complicated than expected, particularly in the solid state. Several crystal structures of iron(III) and iron(II) complexes with dipicH₂ and its derivatives have been reported in which mononuclear, binuclear and polynuclear structures are formed. The ligand-to-iron ratio can be one and two, and six- and seven-coordinate iron complexes have been reported (Laine *et al.*, 1995, 1995*a,b,c*; Hseu *et al.*, 1991; Marsh, 1993; Cousson *et al.*, 1992; Thich *et al.*, 1976). Interest in this area stems from the biological

activity of dipicolinic acid for enhancing the production of spore photoproducts in bacterial spores upon UV irradiation (Bailey *et al.*, 1965; Setlow & Setlow, 1993; Waterbury *et al.*, 1989), the usefulness of dipicolinic acid in analytical chemistry for the determination of iron (Morimoto & Tanaka, 1963; Kanay, 1990), and the catalytic activity of iron–dipicolinate complexes for the activation of dioxygen and hydrogen peroxide (Sheu & Sawyer, 1990; Sheu *et al.*, 1990; Tung *et al.*, 1992; Balavoine *et al.*, 1992). We report here the synthesis and crystal structure determination of the title complex, (1).



The asymmetric unit consists of an $[\text{Fe}^{\text{III}}(\text{dipic})(\text{H}_2\text{O})_3]^+$ cation, an $[\text{Fe}^{\text{III}}(\text{dipic})_2]^-$ anion and six water molecules of crystallization. Each of the Fe atoms has an octahedral coordination geometry which is distorted due to the structural requirements of the tridentate dipicolinate ligands. The two ligands in the complex anion are *trans* meridional with respect to each other and have a dihedral angle of $93.74(6)^\circ$. All three ligands are nearly individually planar, with the largest deviations observed in the positions of the carboxylic O atoms. These deviations can be described by a slight rotation of each carboxylate group from its respective ligand, with angles ranging from $1.1(3)^\circ$ for the C1 group to $3.9(4)^\circ$ for the C7 group. The Fe—O bonds are comparable in both ions [distances range from $2.008(2)$ to $2.028(2)$ Å] and are equivalent to Fe^{III}—O bonds in other Fe^{III}(dipic) complexes (Hseu *et al.*, 1991; Cousson *et al.*, 1992; Marsh, 1993; Laine *et al.*, 1995c). However, they are shorter than their counterparts in Fe^{II}(dipic) complexes. Observed Fe^{II}—O distances are in the range $2.137(6)$ to $2.213(6)$ Å (Laine *et al.*, 1995a,b). On the other hand, the Fe—N bonds are equal in both ions [$2.059(2)$ Å in the cation and $2.054(2)$ Å for each bond in the anion], and they are equivalent to Fe^{III}—N and Fe^{II}—N bond lengths in similar complexes. The shortening of the Fe—O distance with the Fe—N distance remaining unchanged cannot be explained by the smaller ionic radius of Fe^{III} compared with Fe^{II}, for this would lead to shortening of both Fe^{III}—O and Fe^{III}—N bonds. A search of the April 1997 release of the Cambridge Structural Database (Allen & Kennard, 1993) revealed that Fe^{II} and Fe^{III} have bonds to pyridinium N atoms in the wide range 1.90 to 2.75 Å.

All available protons are involved in an extensive three-dimensional network of hydrogen bonding (Table 2). Although the H atoms of the disordered O23

and O23' partial water molecules were not found, these molecules are also involved in hydrogen bonding.

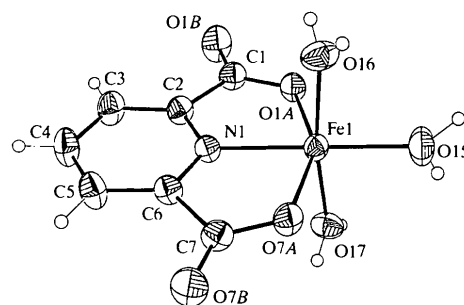


Fig. 1. Displacement ellipsoid drawing (50% probability) of the $[\text{Fe}^{\text{III}}(\text{dipic})(\text{H}_2\text{O})_3]^+$ cation, showing the atom-numbering scheme.

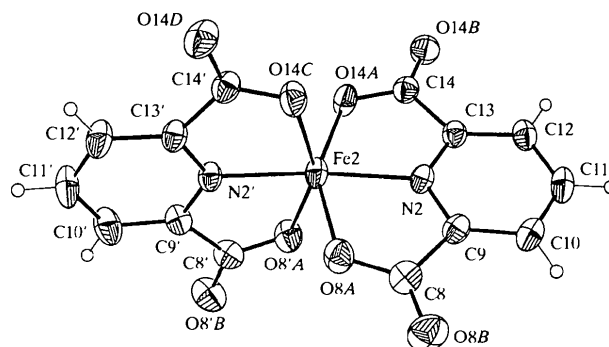
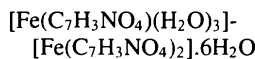


Fig. 2. Displacement ellipsoid drawing (50% probability) of the $[\text{Fe}^{\text{III}}(\text{dipic})_2]^-$ anion, showing the atom-numbering scheme.

Experimental

The title compound was prepared as follows: 30 ml of an aqueous solution (pH 1.5) containing $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ (10^{-3} mol) and dipicH_2 (2×10^{-3} mol) was stirred at room temperature for 1 h. After allowing the solution to stand for 4 d, green crystals formed. Analysis calculated for $\text{C}_{21}\text{H}_{27}\text{N}_3\text{O}_{21}\text{Fe}_2$: C 32.79, H 3.54, N 5.46%; found: C 35.27, H 2.84, N 5.82%. The difference between the calculated and found values is due to the loss of water molecules from the crystal lattice. Mass-FAB (*m/e*): 367, 331, 314, 275, 239.

Crystal data



$M_r = 769.16$

Triclinic

$P\bar{1}$

$a = 8.582(2)$ Å

$b = 11.313(2)$ Å

$c = 16.251(3)$ Å

$\alpha = 74.75(3)^\circ$

$\beta = 87.29(3)^\circ$

$\gamma = 83.16(3)^\circ$

$V = 1511.2(5)$ Å³

$Z = 2$

$D_x = 1.690$ Mg m⁻³

D_m not measured

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 40 reflections

$\theta = 10\text{--}11^\circ$

$\mu = 1.056$ mm⁻¹

$T = 293(2)$ K

Prism

$0.57 \times 0.43 \times 0.27$ mm

Green

Data collection

Siemens P3/PC diffractometer	4326 reflections with $I > 2\sigma(I)$
ω scans	$R_{\text{int}} = 0.015$
Absorption correction: by integration based on measured crystal faces (Sheldrick, 1995)	$\theta_{\text{max}} = 25^\circ$ $h = 0 \rightarrow 10$ $k = -13 \rightarrow 13$ $l = -19 \rightarrow 19$
$T_{\text{min}} = 0.638$, $T_{\text{max}} = 0.783$	4 standard reflections every 100 reflections
5714 measured reflections	intensity decay: <1%
5323 independent reflections	

Refinement

Refinement on F^2	$(\Delta/\sigma)_{\text{max}} = 0.001$
$R[F^2 > 2\sigma(F^2)] = 0.035$	$\Delta\rho_{\text{max}} = 0.568 \text{ e } \text{\AA}^{-3}$
$wR(F^2) = 0.097$	$\Delta\rho_{\text{min}} = -0.566 \text{ e } \text{\AA}^{-3}$
$S = 1.037$	Extinction correction: <i>SHELXL97</i>
5323 reflections	Extinction coefficient: 0.0058 (8)
448 parameters	Scattering factors from <i>International Tables for Crystallography</i> (Vol. C)
H atoms: see below	
$w = 1/[\sigma^2(F_o^2) + (0.0547P)^2 + 0.5498P]$	
where $P = (F_o^2 + 2F_c^2)/3$	

Table 1. Selected bond lengths (Å)

Fe1—O15	1.971 (2)	Fe2—O8A	2.025 (2)
Fe1—O16	2.004 (2)	Fe2—O8'A	2.028 (2)
Fe1—O17	1.987 (2)	Fe2—O14A	2.023 (2)
Fe1—O1A	2.019 (2)	Fe2—O14C	2.023 (2)
Fe1—O7A	2.008 (2)	Fe2—N2	2.054 (2)
Fe1—N1	2.059 (2)	Fe2—N2'	2.054 (2)

Table 2. Hydrogen-bonding geometry (Å, °)

D—H...A	D—H	H...A	D...A	D—H...A
O15—H15A...O19	0.95 (5)	1.64 (5)	2.587 (4)	175 (4)
O15—H15B...O23 ⁱ	0.68 (4)	1.87 (4)	2.55 (2)	171 (5)
O15—H15B...O23 ⁱⁱ	0.68 (4)	2.23 (5)	2.85 (2)	153 (5)
O16—H16B...O8'B ⁱⁱ	0.75 (4)	1.91 (4)	2.625 (3)	160 (4)
O16—H16A...O14B	0.77 (4)	1.91 (4)	2.675 (3)	175 (4)
O17—H17B...O18	0.90 (4)	1.70 (4)	2.592 (3)	176 (4)
O17—H17A...O14D ⁱⁱⁱ	0.73 (3)	1.89 (3)	2.610 (3)	171 (3)
O18—H18A...O7B ⁱⁱ	0.87	1.89	2.739 (3)	163
O18—H18B...O8A ⁱ	0.83	2.06	2.881 (3)	169
O19—H19A...O22	0.86	2.12	2.762 (5)	131
O19—H19B...O14B	0.84	2.14	2.822 (4)	138
O20—H20A...O14A	0.77	2.28	2.953 (3)	147
O20—H20B...O1B ^v	0.72	2.08	2.793 (3)	168
O21—H21A...O8'B ^v	1.04	1.83	2.857 (4)	170
O21—H21B...O20 ^{vi}	1.21	1.57	2.762 (5)	166
O22—H22A...O14C ^{viii}	1.12	1.72	2.843 (4)	177
O22—H22B...O23 ^{viii}	0.85	1.72	2.36 (2)	129
O22—H22B...O23 ^{viii}	0.85	2.08	2.84 (2)	147

Symmetry codes: (i) $x - 1, y - 1, z$; (ii) $x - 1, y, z$; (iii) $x, y - 1, z$; (iv) $1 + x, y, z$; (v) $1 - x, 1 - y, -1 - z$; (vi) $-x, 1 - y, -1 - z$; (vii) $-x, 1 - y, -2 - z$; (viii) $1 - x, 1 - y, -2 - z$.

The aromatic H atoms were placed in idealized positions and refined riding on their parent C atoms (C—H 0.93 Å). The H atoms of the coordinated water molecules were refined without constraints, while the H atoms of the uncoordinated water molecules were located from a difference Fourier map and fixed. The displacement parameters were $1.2U_{\text{eq}}$ of the parent C atom for the aromatic H atoms and $1.5U_{\text{eq}}$ for the water H atoms. One of the water molecules was found to be disordered over two positions, O23 and O23', and these sites were refined with isotropic displacement parameters. Their site-occupation factors were independently refined until the

last cycle of refinement, when they were fixed at 0.80 (3) and 0.20 (3) for O23 and O23', respectively. The H atoms of the disordered partial water molecules were not found in a difference Fourier map and were not included in the final refinement. A hemisphere of data was collected using ω scans over 1.2° and a variable speed of $3\text{--}6^\circ \text{ min}^{-1}$, depending on intensity.

Data collection: P3/PC (Siemens, 1993). Cell refinement: P3/PC. Data reduction: *SHELXTL-Plus* (Sheldrick, 1990). Program(s) used to solve structure: *SHELX97* (Sheldrick, 1997). Program(s) used to refine structure: *SHELX97*. Molecular graphics: *SHELXTL* (Sheldrick, 1995). Software used to prepare material for publication: *SHELX97*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1421). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1998). **C54**, 1273–1275

A Triangular $[\text{Mo}_3\text{S}_7]^{4+}$ Complex: Tris-(diethyldithiocarbamato- S,S')tris(μ_2 - η^2 -disulfido)(μ_3 -sulfido)trimolybdenum(IV)- (3 Mo—Mo) Diethyldithiocarbamate

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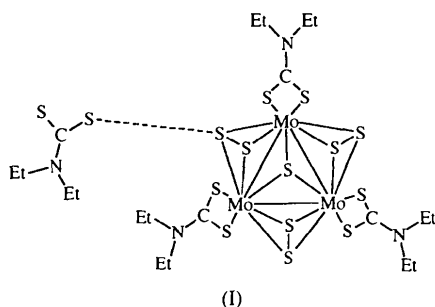
(Received 3 November 1997; accepted 3 March 1998)

Abstract

In the cluster cation of the title compound, $[\text{Mo}_3\text{S}_7(\text{Et}_2\text{NCS}_2)_3](\text{Et}_2\text{NCS}_2)$, three Mo atoms form an approximately equilateral triangle, with Mo—Mo distances ranging from 2.7140 (5) to 2.7277 (5) Å, and Mo—Mo—Mo angles of *ca* 60°. The triangle is capped by the μ_3 -S atom, and each Mo—Mo bond is bridged by a disulfido group in μ_2 - η^2 mode. Diethyldithiocarbamate ligands (Et_2dtc) are chelated to the Mo atoms with an average S—Mo—S bite angle of 69.98 (1)°. There is a weak S...S interaction between the cluster cation and the anion (Et_2dtc^-).

Comment

Compounds containing the $[\text{Mo}_3\text{S}_7]^{4+}$ core have been studied extensively (Müller *et al.*, 1979; Keck *et al.*, 1981). Fedin, Sokolov, Virovets *et al.* (1991) have synthesized the title compound, (I), using $(\text{Et}_4\text{N})_2\text{Mo}_3\text{S}_7\text{Br}$ as the starting material, but the crystal structure of the compound has not yet been reported.



The structure of complex (I) consists of an $[\text{Mo}_3\text{S}_7(\text{Et}_2\text{dtc})_3]^+$ cation and an Et_2dtc^- anion (Fig. 1). The cluster cation contains an Mo_3 triangle capped by a μ_3 -S atom, with Mo—Mo distances and Mo—Mo—Mo bond angles ranging from 2.7140 (5) to 2.7277 (5) Å

and from 59.68 (1) to 60.18 (1)°, respectively. There are three parallel planes in the Mo_3S_7 core: the Mo_3 plane, S1/S3/S5 and S2/S4/S6, with a maximum dihedral angle of 1.34 (3)°. In the three S_2 groups, the equatorial S2, S4 and S6 atoms, and the axial S1, S3 and S5 atoms, deviate from the Mo_3 plane by not more than 0.17 Å for the former, and by not less than 1.63 Å for the latter. The Mo_3S_7 core of the cation has idealized C_{3v} symmetry, with a C_3 axis passing through the μ_3 -S atom, and this symmetry also includes the NCS_2 groups of three Et_2dtc^- ligands. All these structural features of the $[\text{Mo}_3\text{S}_7]^{4+}$ core are very similar to those of other complexes reported with an $[\text{M}_3\text{X}_7]^{n+}$ core, for instance, $M = \text{Mo}$ or W , $X = \text{S}$ or Se , $n = 4$ (Shang *et al.*, 1984; Lu *et al.*, 1994; Fedin, Sokolov, Geras'ko *et al.*, 1991; Fedin, Sokolov, Virovets *et al.*, 1991; Hegetchweiler *et al.*, 1991; Zimmermann *et al.*, 1991) or $M = \text{V}$, $X = \text{S}$, $n = 2$ (Yang *et al.*, 1993; Dean *et al.*, 1993).

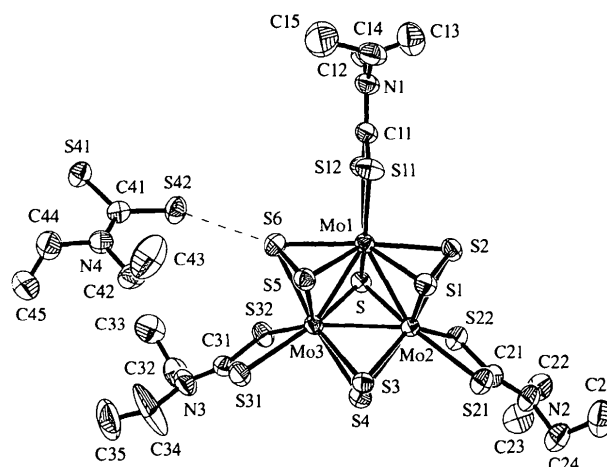


Fig. 1. ORTEP (Johnson, 1976) plot of the molecular structure of $[\text{Mo}_3\text{S}_7(\text{Et}_2\text{dtc})_3](\text{Et}_2\text{dtc})$, showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

All the $[\text{Mo}_3\text{S}_7]^{4+}$ cores of these complexes, including (I), exhibit almost the same structural parameters: (i) Mo—Mo distances in the range 2.700–2.782 Å and Mo—Mo—Mo bond angles of *ca* 60°; (ii) Mo— μ_3 -S bond distances in the range 2.336–2.386 Å; (iii) Mo— S_{eq} distances in the range 2.406–2.416 Å and Mo— S_{ax} distances of 2.487–2.511 Å; (iv) $\text{S}_{\text{eq}}-\text{S}_{\text{ax}}$ distances in the range 2.021–2.069 Å.

Compared with the selenium analogue, $[\text{Mo}_3\text{Se}_7(\text{Et}_2\text{dtc})_3](\text{Et}_2\text{dtc})$, (II) (Fedin, Sokolov, Geras'ko *et al.*, 1991), complex (I) has an obviously smaller $[\text{Mo}_3\text{S}_7]$ core than the $[\text{Mo}_3\text{Se}_7]$ core of (II). Specifically, all the bond distances in the cluster core of (I) are shorter than those of (II). Examples are: the average Mo—Mo distance [(I) 2.723; (II) 2.778 Å], Mo— μ_3 -X [(I) $X = \text{S}$, 2.386 Å; (II) $X = \text{Se}$, 2.498 Å], Mo— X_{ax} [(I) 2.490; (II) 2.617 Å] and Mo— X_{eq} [(I) 2.406; (II)