

ics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PARST* (Nardelli, 1995).

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Diazidobis(1,10-phenanthroline-*N,N'*)-manganese(II)

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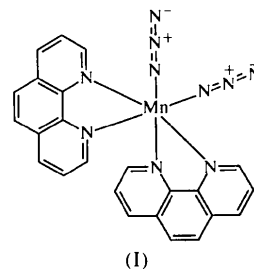
(Received 24 December 1998; accepted 28 January 1999)

Abstract

The crystal structure of the title complex, [Mn(N₃)₂·(C₁₂H₈N₂)₂], shows the two 1,10-phenanthroline ligands and the two azido anions to be in a distorted octahedral arrangement around the Mn^{II} atom, with the two azido ions in *cis* positions.

Comment

In recent years, the azido ion has been shown to be a very versatile ligand in the field of coordination chemistry. The copper–azido system has been thoroughly studied from both the synthetic and magnetic points of view (Thompson *et al.*, 1995). At present, the chemistry of manganese–azido-bridged systems has been found to be characterized by the high dimensionality of the resulting complexes; in one case only, a dinuclear compound was obtained, while all the other derivatives are one-, two- or three-dimensional compounds (Escuer *et al.*, 1998). We report here the structure of a mononuclear manganese–azido complex, (I), with the two azido ions in *cis* positions.



The asymmetric unit of (I) consists of one-half of the title complex, with the other half generated by twofold symmetry; the Mn1 atom lies on the twofold axis. The two 1,10-phenanthroline (phen) ligands and

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the two azido ions form a distorted octahedral geometry around the Mn atom, with the two azido ligands in *cis* positions. This arrangement is similar to that observed in the structures of *cis*-[Mn(phen)₂Cl₂] (Malinowski & Bologna, 1996) and [Mn(phen)₂Cl₂]NO₃·2.5CH₃COOH (Reddy & Rajasekharan, 1994). The Mn1—N1 distance of 2.332 (2) Å is slightly longer than the Mn1—N2 distance [2.284 (2) Å]. The phen ligand is planar, with the C9 atom having the greatest deviation at -0.052 (3) Å. In the crystal, one of azido N atoms (N3) is involved in a weak C8—H8···N3($\frac{1}{2} + x, \frac{1}{2} + y, \frac{1}{2} - z$) intermolecular hydrogen bond (Table 2).

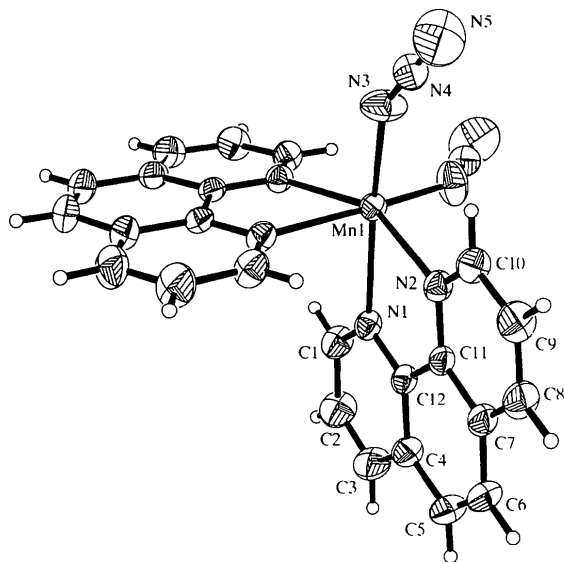


Fig. 1. The structure of (I) showing 30% probability displacement ellipsoids and the atom-numbering scheme. Only the contents of the asymmetric unit are numbered.

Experimental

The title complex was synthesized by reaction of Mn(NO₃)₂, 1,10-phenanthroline and NaN₃ in water.

Crystal data

[Mn(N ₃) ₂ (C ₁₂ H ₈ N ₂) ₂]	Mo K α radiation
$M_r = 499.41$	$\lambda = 0.71073$ Å
Orthorhombic	Cell parameters from 8192 reflections
<i>Pbcn</i>	$\theta = 2.40$ – 38.62°
$a = 13.4920$ (2) Å	$\mu = 0.634$ mm ⁻¹
$b = 9.6317$ (1) Å	$T = 293$ (2) K
$c = 16.9975$ (3) Å	Plate
$V = 2208.84$ (6) Å ³	$0.40 \times 0.30 \times 0.18$ mm
$Z = 4$	Yellow
$D_x = 1.502$ Mg m ⁻³	
D_m not measured	

Data collection

Siemens SMART CCD area-detector diffractometer	2002 reflections with $I > 2\sigma(I)$
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ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.785, T_{\max} = 0.894$
 14 217 measured reflections
 2540 independent reflections

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.118$
 $S = 1.063$
 2540 reflections
 159 parameters
 H atoms constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0619P)^2 + 0.6583P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$R_{\text{int}} = 0.031$

$\theta_{\max} = 27.5^\circ$
 $h = 0 \rightarrow 17$
 $k = 0 \rightarrow 12$
 $l = 0 \rightarrow 22$

$(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.42$ e Å⁻³
 $\Delta\rho_{\min} = -0.30$ e Å⁻³
 Extinction correction: none
 Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. Selected geometric parameters (Å, °)

Mn1—N3 ⁱ	2.140 (2)	N1—C1	1.327 (3)
Mn1—N3	2.140 (2)	N1—C12	1.358 (2)
Mn1—N2	2.284 (2)	N2—C10	1.329 (3)
Mn1—N2 ⁱ	2.284 (2)	N2—C11	1.355 (2)
Mn1—N1	2.332 (2)	N3—N4	1.136 (3)
Mn1—N1 ⁱ	2.332 (2)	N4—N5	1.142 (3)
N3 ⁱ —Mn1—N3	99.2 (2)	N2—Mn1—N1	72.21 (5)
N3 ⁱ —Mn1—N2	102.11 (8)	N2 ⁱ —Mn1—N1	88.46 (5)
N3—Mn1—N2	94.94 (7)	N3 ⁱ —Mn1—N1 ⁱ	166.30 (7)
N3 ⁱ —Mn1—N2 ⁱ	94.94 (7)	N3—Mn1—N1 ⁱ	88.43 (9)
N3—Mn1—N2 ⁱ	102.11 (8)	N2—Mn1—N1 ⁱ	88.46 (5)
N2—Mn1—N2 ⁱ	153.62 (8)	N2 ⁱ —Mn1—N1 ⁱ	72.21 (5)
N3 ⁱ —Mn1—N1	88.43 (9)	N1—Mn1—N1 ⁱ	86.53 (8)
N3—Mn1—N1	166.30 (7)	N3—N4—N5	177.6 (3)

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

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

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
C8—H8···N3 ⁱ	0.93	2.53	3.337 (3)	146

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

The data collection covered a hemisphere of reciprocal space by a combination of three sets of exposures; each set had a different φ angle (0, 88 and 180°) for the crystal and each exposure of 30 s covered 0.3° in ω . The crystal-to-detector distance was 3 cm. Crystal decay was monitored by repeating 30 initial frames at the end of data collection and analysing the intensity of duplicate reflections; it was found to be negligible. All H atoms were fixed geometrically and a riding model was used for their refinement.

Data collection: SMART (Siemens, 1996). Cell refinement: SAINT (Siemens, 1996). Data reduction: SAINT. Program(s) used to solve structure: SHELXTL (Sheldrick, 1997). Program(s) used to refine structure: SHELXTL. Molecular graphics: SHELXTL. Software used to prepare material for publication: SHELXTL and PLATON (Spek, 1990).

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catena-Poly[[*(2,2'*-bipyridine)iron(II)]- μ -oxalato]

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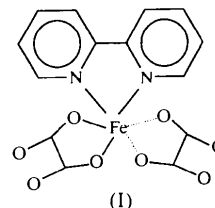
Abstract

The crystal structure of catena-poly[[*(2,2'*-bipyridine-*N,N'*)iron(II)]- μ -(oxalato-*O*¹,*O*²:*O*¹,*O*^{2'})], [Fe(C₂O₄)(C₁₀H₈N₂)]_n, is reported. The Fe^{II} atom is six-coordinated with distorted octahedral geometry and the bipyridine ring is in a planar form. The oxalate anion bridges the metal ions forming infinite chains.

Comment

Recently, oxalate-bridged polymeric compounds, especially those with higher dimensionalities, have attracted much attention due to their interesting magnetic properties (Decurtins *et al.*, 1994, and references therein). Using *2,2'*-bipyridine (*2,2'*-bipy) as a terminal ligand, chain compounds can be obtained which contain ox-

alate anions as bridging ligands. So far, there have been only two such compounds reported, viz. [Cu(C₂O₄)(*2,2'*-bipy)]·2H₂O (Fitzgerald *et al.*, 1982) and [Mn(C₂O₄)(*2,2'*-bipy)] (Deguenon *et al.*, 1990). We report herein an Fe^{II} compound, [Fe(C₂O₄)(*2,2'*-bipy)], (I), which is isostructural with the Mn^{II} compound.



An examination of the relative geometrical positions of the planar *2,2'*-bipy and oxalate ligands (Fig. 1) shows no particular kind of stacking, although the planes are nearly coplanar. The crystal structure of the title compound consists of neutral [Fe(C₂O₄)(*2,2'*-bipy)] units, with the Fe^{II} ions linked by C₂O₄ ligands to form infinite 'zigzag' chains along the *a* axis. The Fe^{II} atom has a distorted octahedral coordination environment which is made up of two N atoms from the *2,2'*-bipyridine ligand and four O atoms from the oxalate ligands. The Fe—N bond lengths [2.159 (4) and 2.173 (4) Å] and the N—Fe—N bite angle [75.7 (1)°] are comparable to corresponding values in [Fe₄O₂(O₂CCH₃)₇(bipy)₂]ClO₄·0.25CH₂Cl₂·H₂O [2.145 (6), 2.185 (6) Å and 74.9 (2)°; McCusker *et al.*,

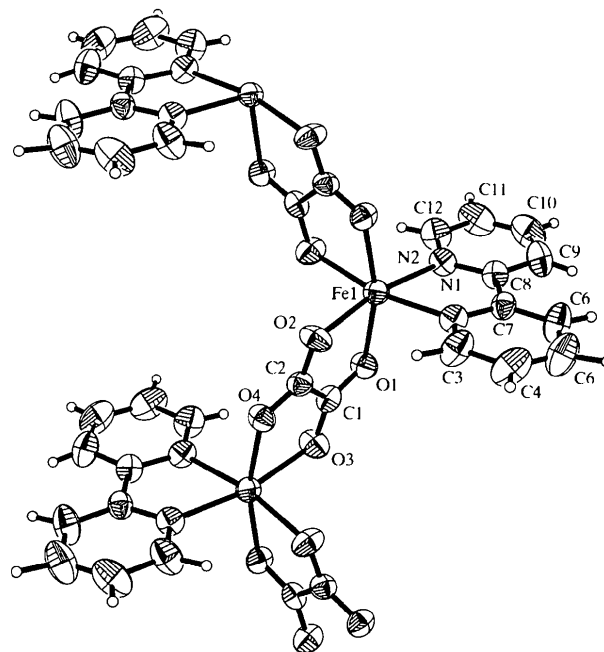


Fig. 1. Part of the polymer chain of (I), showing 50% probability displacement ellipsoids, with the asymmetric unit labelled.