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Rerefinement of poly[[tetra-μ-oxo-dioxobis-(1,10-phenanthroline-κ^2N,N)dimolybdenum(VI)-iron(II)]-di-μ-oxo] in the centrosymmetric space group $P2_1/m$

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.024
wR factor = 0.065
Data-to-parameter ratio = 12.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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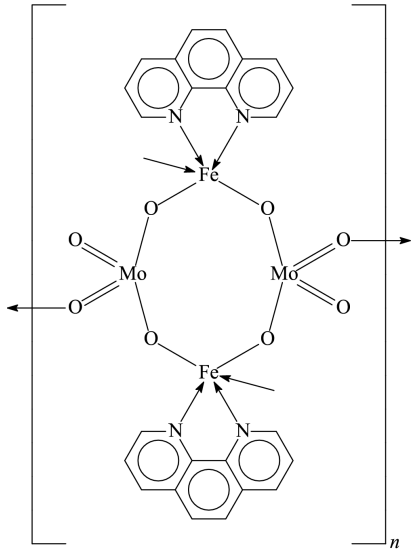
Polymeric $[Fe_2\{MoO_4\}_2(C_{12}H_8N_2)_2]_n$ adopts a chain structure that propagates linearly along the b axis of the monoclinic unit cell. The $(C_{12}H_8N_2)Fe$ moiety lies on a mirror plane, as does the MoO_4 moiety; two MoO_4 anions link two $(C_{12}H_8N_2)Fe$ entities to form an eight-membered Fe—O—Mo—O—Fe—O—Mo—O— ring; the Fe atom is five-coordinate in a trigonal bipyramidal environment; the phenanthroline ligand spans the axial–equatorial sites.

Comment

The title compound, (I), was previously refined in the space group $P2_1$ (Chu *et al.*, 2001); a check with *PLATON* (Spek, 2003) shows that the structure is better described in $P2_1/m$. In this setting, it is isostructural with the Zn analog, which was also reported earlier (Hagrman & Zubieta, 1999); both structures have been described in detail. With the refinement based on new diffraction measurements, the $(C_{12}H_8N_2)Fe$ moiety lies on a mirror plane, as does the MoO_4 moiety; two MoO_4 anions link two $(C_{12}H_8N_2)Fe$ entities to form an eight-membered Fe—O—Mo—O—Fe—O—Mo—O— ring (Fig. 1).



(I)

Experimental

A mixture of $Na_2MoO_4{\cdot}2H_2O$ (0.194 g, 0.8 mmol), $FeCl_2{\cdot}4H_2O$ (0.123 g, 0.62 mmol), 1,10-phenanthroline (0.054 g, 0.3 mmol), ethylenediamine (0.034 g, 0.58 mmol) and water (7 ml) was placed in a 15 ml Teflon-lined stainless-steel bomb, which was heated at 448 K for 120 h. The bomb was cooled slowly to room temperature and black block-shaped crystals were isolated in about 45% yield. Analysis found: C 36.35, H 2.07, N 7.02%; calculated for $C_{12}H_8FeMoN_2O_4$: C 36.40, H 2.04, N 7.07%.

Crystal data

$[Fe_2Mo_2O_8(C_{12}H_8N_2)_2]$	D_x = 2.159 Mg m^{-3}
M_r = 791.99	Mo $K\alpha$ radiation
Monoclinic, $P2_1/m$	Cell parameters from 2636 reflections
a = 8.873 (1) Å	θ = 2.3–27.6°
b = 6.567 (1) Å	μ = 2.23 mm^{-1}
c = 10.629 (2) Å	T = 293 (2) K
β = 100.437 (2)°	Block, black
V = 609.1 (2) Å^3	0.13 × 0.11 × 0.11 mm
Z = 1	

Data collection

Bruker SMART APEX area-detector diffractometer	1469 independent reflections
	1360 reflections with $I > 2\sigma(I)$
φ and ω scans	R_{int} = 0.025
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)	θ_{max} = 27.5°
	$h = -11 \rightarrow 11$
T_{min} = 0.697, T_{max} = 0.791	$k = -8 \rightarrow 8$
4906 measured reflections	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0414P)^2 + 0.0379P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.024	
$wR(F^2)$ = 0.065	
S = 1.04	$(\Delta/\sigma)_{max}$ = 0.001
1469 reflections	$\Delta\rho_{max}$ = 0.39 e Å^{-3}
118 parameters	$\Delta\rho_{min}$ = −0.33 e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Mo1—O1	1.778 (2)	Fe1—O1^{ii}	1.980 (2)
Mo1—O1^{i}	1.778 (2)	Fe1—O2^{iii}	2.068 (3)
Mo1—O2	1.748 (2)	Fe1—N1	2.143 (3)
Mo1—O3	1.708 (3)	Fe1—N2	2.190 (3)
Fe1—O1	1.980 (2)		
O1—Mo1—O1^{i}	109.1 (1)	O1—Fe1—N2	90.8 (1)
O1—Mo1—O2	110.6 (1)	O1^{ii}—Fe1—O2^{iii}	91.2 (1)
O1—Mo1—O3	109.2 (1)	O1^{ii}—Fe1—N1	111.6 (1)
O1^{i}—Mo1—O2	110.6 (1)	O1^{ii}—Fe1—N2	90.8 (1)
O1^{i}—Mo1—O3	109.2 (1)	O2^{iii}—Fe1—N1	97.9 (1)
O2—Mo1—O3	108.4 (2)	O2^{iii}—Fe1—N2	174.8 (1)
O1—Fe1—O1^{ii}	135.9 (1)	N1—Fe1—N2	76.9 (1)
O1—Fe1—O2^{iii}	91.2 (1)	Mo1—O1—Fe1	153.4 (1)
O1—Fe1—N1	111.6 (1)	Mo1—O2—Fe1^{iii}	170.2 (2)

Symmetry codes: (i) $x, \frac{3}{2} - y, z$; (ii) $x, \frac{1}{2} - y, z$; (iii) $2 - x, 1 - y, 2 - z$.

H atoms were positioned geometrically [C—H = 0.93 Å; U_{iso}(H) = 1.2U_{eq}(C)] and were included in the refinement in the riding-model approximation.

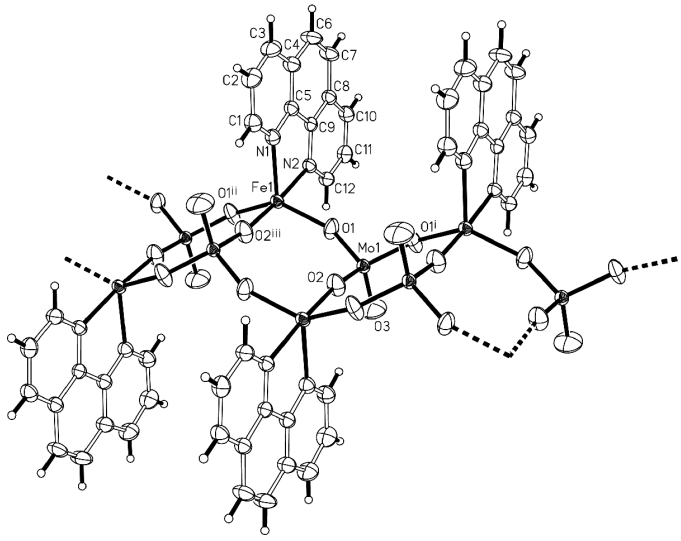


Figure 1
*ORTEP*II (Johnson, 1976) plot (50% probability displacement ellipsoids) of a fragment of the polymeric chain of the title compound. H atoms are drawn as spheres of arbitrary radii. [Symmetry codes: (i) $x, \frac{3}{2} - y, z$; (ii) $x, \frac{1}{2} - y, z$; (iii) $2 - x, 1 - y, 2 - z$.]

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; method used to solve structure: atomic coordinates taken from the Zn analog (Hagrman & Zubieta, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *OPTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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