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A novel heterometallic Fe^{II}–Na^+ phase: 1,10-phenanthrolinium aquabis(1,10-phenanthroline)sodium(I) pentacyanonitrosoiron(II) monohydrate

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The structure of the title compound, $(C_{12}H_9N_2)$-$[Na(C_{12}H_8N_2)_2(H_2O)][Fe(CN)_5(NO)]\cdot H_2O$, is built up from Fe^{II}-containing anions, Na^+-containing cations, non-coordinated 1,10-phenanthrolinium (Hphen) cations and water molecules. The sodium cation is coordinated to two *N,N'*-bidentate phen ligands and one water molecule in approximate square-based pyramidal geometry, with the O atom occupying the axial site. The non-coordinated Hphen and water molecules link the metal complexes through O—H···N and N—H···O hydrogen bonds, forming a three-dimensional network.

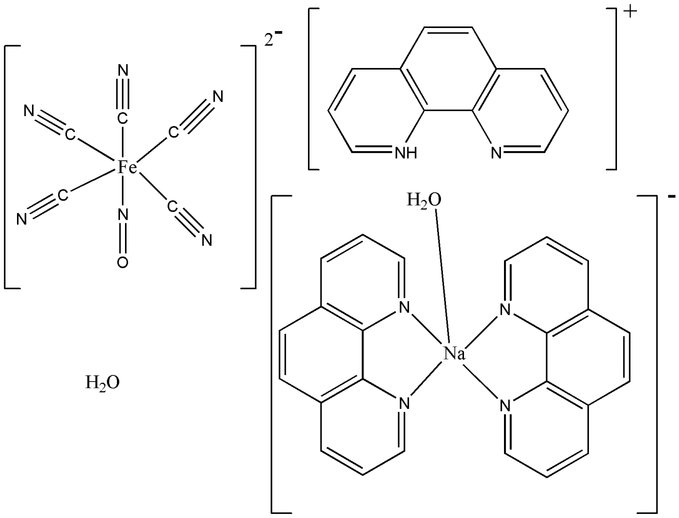
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.063
wR factor = 0.148
Data-to-parameter ratio = 14.9

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

Comment

The title compound, (I), combines an Fe^{II}-containing, $[Fe(CN)_5(NO)]^{2-}$, nitroprusside anion [see Soria *et al.* (2002) for a review of related structures] with an unusual $[Na(phen)_2(H_2O)]^+$ cation.



(I)

The asymmetric unit of (I) (Fig. 1) contains a discrete $[Na(1,10\text{-phenanthroline})_2(H_2O)]^+$ grouping, a $[Fe(CN)_5(NO)]^{2-}$ nitroprusside unit, one uncoordinated $(C_{12}H_9N_2)^+$ 1,10-phenanthrolinium cation and one uncoordinated water molecule (Table 1). The coordination environment about Na1 approximates to a square-based pyramid, with the four N atoms (N7, N8, N9 and N10) from two phen ligands occupying the equatorial positions, and one water O atom (O2) occupying the axial position. The Na—O bond is slightly shorter than the Na—N bonds (Table 1). The $[Fe(CN)_5(NO)]^{2-}$ nitroprusside ion has its usual distorted octahedral pagoda-

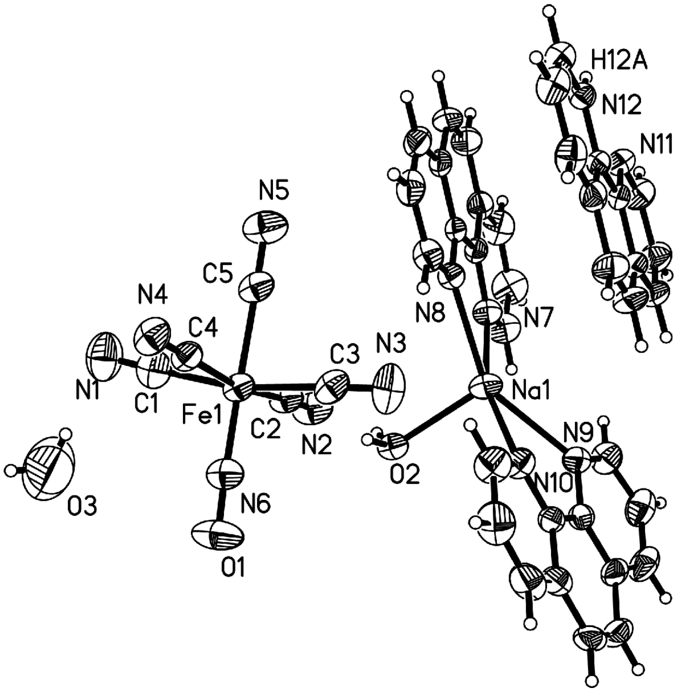


Figure 1
*ORTEP*II (Johnson, 1976) drawing of (I), with 35% probability ellipsoids, showing the atom-numbering scheme. H atoms are represented by spheres of arbitrary radius. For clarity, H and C atoms are not labelled.

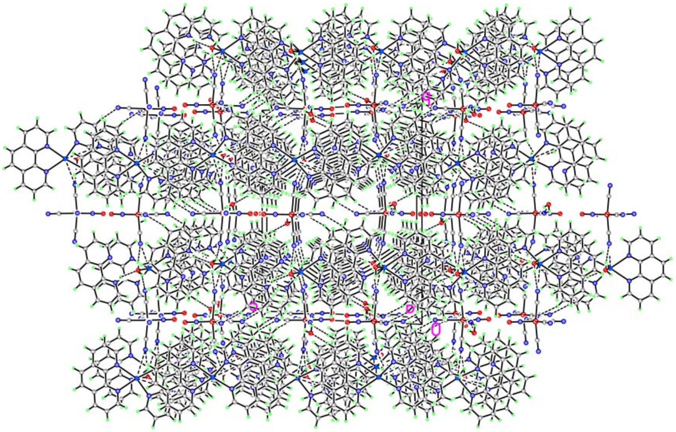


Figure 2
A view of the molecular packing in (I), viewed down the *a* axis.

like conformation, with an average Fe—C bond distance of 1.936 (2) Å and an Fe—N bond distance of 1.652 (4) Å, in agreement with literature values (Soria *et al.*, 2002). The structure is completed by an uncoordinated Hphen cation, thus ensuring charge balance, and one uncoordinated water molecule. As shown in Fig. 2, the O—H$\cdots$N and N—H$\cdots$O hydrogen bonds (Table 2) between the metal complexes, Hphen cations water molecules result in the formation of a three-dimensional network.

Experimental

Sodium nitroprusside (0.5 mmol) and phen (1.0 mmol) were dissolved in deionized water (20 ml). The resulting mixture was stirred for 4 h at room temperature and filtered and evaporated. After 7 d, red crystals of the complex suitable for X-ray single-crystal analysis were obtained. They were collected by suction filtration and air-dried (yield: 14%). All chemicals used in this experiment were purchased commercially without further purification. Found (%): C 60.13, H 3.59, N 20.51; calculated for $C_{41}H_{29}FeN_{12}NaO_3$ (%): C 60.25, H 3.55, N 20.57.

Crystal data

$(C_{12}H_9N_2)[Na(C_{12}H_8N_2)_2(H_2O)]$-
$[Fe(CN)_5(NO)]\cdot H_2O$
$M_r = 816.60$
Monoclinic, $P2_1/c$
$a = 10.429$ (3) Å
$b = 22.125$ (6) Å
$c = 19.435$ (6) Å
$\beta = 121.922$ (5)°
$V = 3806.1$ (18) Å^3
$Z = 4$

$D_x = 1.425$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1009 reflections
$\theta = 2.2$–20.9°
$\mu = 0.47$ mm^{-1}
$T = 293$ (2) K
Prism, red
0.18 × 0.16 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
$T_{min} = 0.794$, $T_{max} = 0.946$
21737 measured reflections

7801 independent reflections
3256 reflections with $I > 2\sigma(I)$
$R_{int} = 0.100$
$\theta_{max} = 26.4°$
$h = -13 \rightarrow 13$
$k = -27 \rightarrow 26$
$l = -20 \rightarrow 24$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.063$
$wR(F^2) = 0.149$
$S = 0.93$
7801 reflections
523 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0552P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.42$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Fe1—N6	1.652 (4)	Na1—O2	2.378 (3)
Fe1—C1	1.926 (6)	Na1—N7	2.413 (4)
Fe1—C5	1.927 (5)	Na1—N10	2.415 (4)
Fe1—C3	1.940 (5)	Na1—N8	2.439 (4)
Fe1—C4	1.940 (4)	Na1—N9	2.520 (4)
Fe1—C2	1.948 (4)		
O2—Na1—N7	98.81 (11)	N10—Na1—N8	115.77 (14)
O2—Na1—N10	94.87 (12)	O2—Na1—N9	102.25 (12)
N7—Na1—N10	163.19 (14)	N7—Na1—N9	99.73 (13)
O2—Na1—N8	109.19 (11)	N10—Na1—N9	67.65 (13)
N7—Na1—N8	68.65 (12)	N8—Na1—N9	147.68 (13)

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O2—H2$A\cdots$N4^{i}	0.85	2.10	2.897 (4)	157
O2—H2$B\cdots$N2	0.85	2.04	2.883 (4)	171
N12—H12$A\cdots$O2^{ii}	0.86	1.96	2.774 (4)	157
O3—H3$A\cdots$N1	0.84	1.88	2.725 (8)	179
O3—H3$B\cdots$N3^{iii}	0.86	2.27	2.806 (7)	120

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

H atoms attached to O and N atoms were positioned geometrically and the coordinates for water H atoms were calculated using the program *HYDROGEN* (Nardelli, 1999). These H atoms were constrained to ride on their parent atoms in the final refinement. H atoms attached to C atoms were placed in calculated positions (C—H = 0.93 Å) and allowed to ride on the parent atoms, with U_{iso} values constrained to be $1.2U_{eq}$ of the parent atom.

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Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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