Poly[tetra-μ_2-azido-di-μ_2-4,4′-bipyridine-sodium(I)iron(III)]

Christian Näther,* Jan Greve and Inke Jeß

Institut für Anorganische Chemie, Christian-Albrechts-Universität Kiel, Olshausenstraße 40, D-24098 Kiel, Germany

Correspondence e-mail: cnaether@ac.uni-kiel.de

Key indicators

Single-crystal X-ray study

T = 150 K

Mean σ(C–C) = 0.002 Å

R factor = 0.029

wR factor = 0.075

Data-to-parameter ratio = 15.3

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

In the crystal structure of the title compound, [FeNa-$(N_3)_4(C_{10}H_8N_2)_2]_n$, the Fe and Na atoms are coordinated by four N atoms of azide anions and two N atoms of 4,4′-bipyridine ligands within distorted octahedra. The azide anions are located in general positions whereas the Fe and Na atoms, as well as the 4,4′-bipyridine ligands, are located on twofold axes. The iron and the sodium cations are connected *via* the 4,4′-bipyridine ligands into chains, which are linked by the azide anions into a three-dimensional coordination network.

Comment

The structure determination of the title compound, (I), was undertaken as part of a project on the synthesis, crystal structures and properties of new coordination polymers based on paramagnetic transition metals, small anions like cyanide, azide or thiocyanate anions and N-donor ligands (Näther & Greve, 2003). The title compound was obtained by accident and cannot be prepared as a pure phase.

(I)
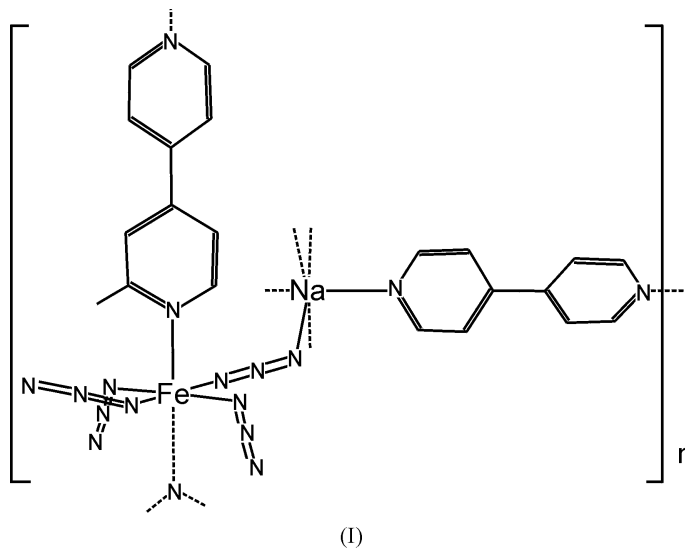
The asymmetric unit of (I) consists of one half Fe atom, one half Na atom, two half 4,4′-bipyridine ligands and two azide anions. Whereas the azide anions are located in general positions, the Fe and Na atoms, as well as the 4,4′-bipyridine ligands, are located on twofold axes. In the structure, the Fe[III] atoms are sixfold coordinated by two N atoms of two symmetry-related 4,4′-bipyridine ligands and four N atoms of four azide anions, which are related in pairs by symmetry. As expected, the Fe—N bond lengths to the negatively charged azide N atoms are shorter than those to the N atoms of the 4,4′-bipyridine ligands (Fig. 1). The N—Fe—N angles have
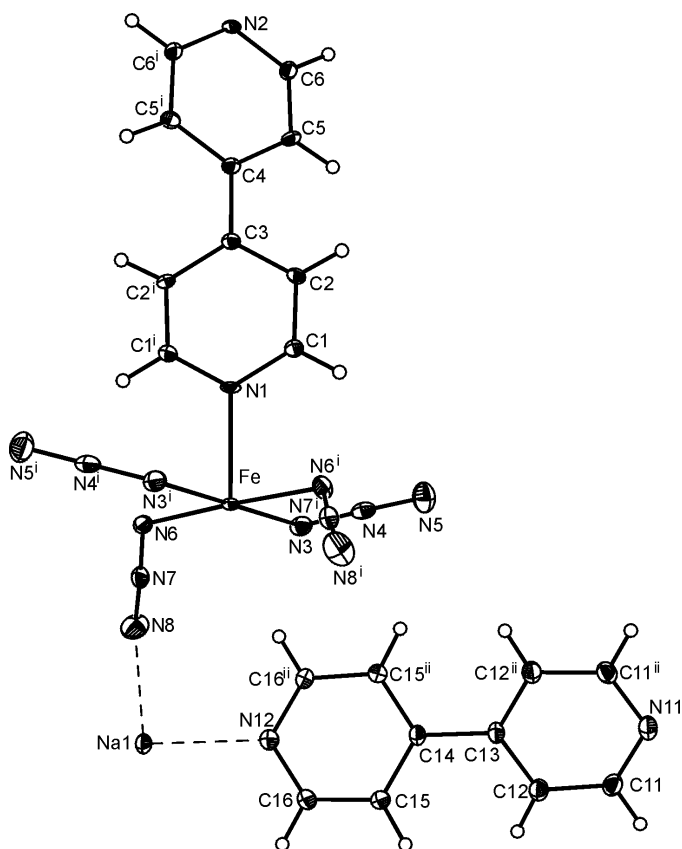
Figure 1
Part of the crystal structure of the title compound, showing the atom labelling and displacement ellipsoids at the 50% probability level. The sodium coordination is indicated by dashed lines. [Symmetry codes: (i) x, $y + 1$, $-z$; (ii) $-x + 2$, y, $-z + \frac{1}{2}$.]
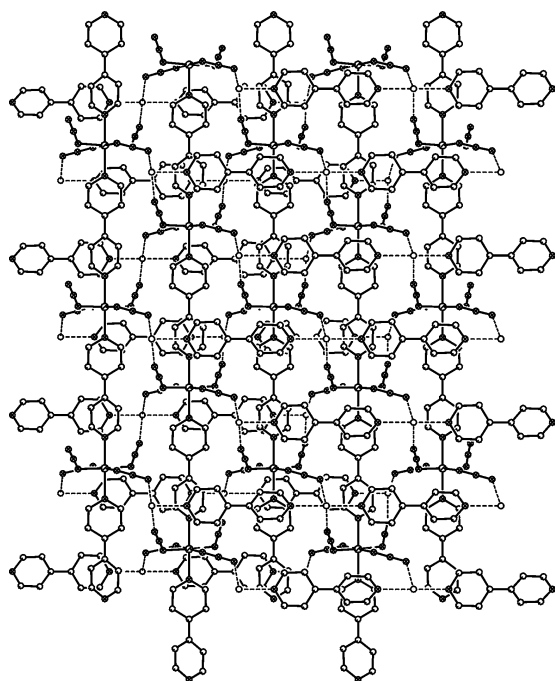

Figure 2
The crystal structure of the title compound, viewed along the c axis. H atoms have been omitted for clarity and the Na coordination is indicated by dashed lines.

close to ideal octahedral values and the coordination polyhedron can be described as a slightly distorted octahedron. The Na atoms are also sixfold coordinated by two pairs of symmetry-related azide anions and two symmetry-related 4,4′-bipyridine ligands (Fig. 2). Compared to the Fe atoms, the octahedron is much more distorted. The Fe and Na atoms are connected by 4,4′-bipyridine ligands into chains which are perpendicular to each other. These chains are connected via the azide anions into a three-dimensional coordination network (Fig. 2).

Experimental

The title compound was prepared by the reaction of $FeCl_2$ (98.89 mg, 0.5 mmol), NaN_3 (65.10 mg, 1 mmol) and 4,4′-bipyridine (78.30 mg, 0.5 mmol) in acetonitrile (4 ml) at room temperature. After 6 d, the precipitate was filtered off and washed with ethanol and diethyl ether. It consisted of red crystals of the title compound and a light-pink microcrystalline powder which was not identified.

Crystal data

$[FeNa(N_3)_4(C_{10}H_8N_2)_2]$	Mo $K\alpha$ radiation
$M_r = 559.33$	Cell parameters from 8000
Orthorhombic, $C222_1$	reflections
$a = 11.4626$ (6) Å	$\theta = 16$–$26°$
$b = 12.0032$ (9) Å	$\mu = 0.72$ mm^{-1}
$c = 16.7541$ (9) Å	$T = 150$ (2) K
$V = 2305.2$ (2) Å^3	Block, red
$Z = 4$	$0.07 \times 0.06 \times 0.06$ mm
$D_x = 1.612$ Mg m^{-3}	

Data collection

Stoe Imaging Plate Diffraction System diffractometer	2576 reflections with $I > 2\sigma(I)$
φ scans	$R_{int} = 0.049$
Absorption correction: none	$\theta_{max} = 28.1°$
13 692 measured reflections	$h = -14 \rightarrow 14$
2728 independent reflections	$k = -15 \rightarrow 15$
	$l = -22 \rightarrow 22$

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} < 0.001$
$R[F^2 > 2\sigma(F^2)] = 0.029$	$\Delta\rho_{max} = 0.36$ e Å^{-3}
$wR(F^2) = 0.075$	$\Delta\rho_{min} = -0.48$ e Å^{-3}
$S = 1.09$	Extinction correction: *SHELXL97*
2728 reflections	Extinction coefficient: 0.0026 (4)
178 parameters	Absolute structure: Flack (1983),
H-atom parameters constrained	1214 Friedel pairs
$w = 1/[\sigma^2(F_o^2) + (0.043P)^2$	Flack parameter = 0.007 (15)
$+ 1.8553P]$	
where $P = (F_o^2 + 2F_c^2)/3$	

Table 1
Selected geometric parameters (Å, °).

Fe1—N3	2.0294 (15)	N5—Na1	2.572 (2)
Fe1—N6	2.0593 (16)	N6—N7	1.192 (2)
Fe1—N1	2.171 (2)	N7—N8	1.148 (3)
N3—N4	1.189 (2)	N8—Na1^{iii}	2.4476 (19)
N4—N5	1.163 (3)	Na1—N11^{iv}	2.464 (3)
N3^v—Fe1—N3	179.83 (11)	N3^v—Fe1—N2^{vi}	89.92 (5)
N3^v—Fe1—N6	89.74 (7)	N6^v—Fe1—N2^{vi}	88.84 (5)
N3—Fe1—N6	90.25 (7)	N1—Fe1—N2^{vi}	180
N6—Fe1—N6^v	177.69 (10)	N5—N4—N3	176.8 (2)
N3—Fe1—N1	90.08 (5)	N8—N7—N6	177.3 (2)
N6—Fe1—N1	91.16 (5)		

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

All H atoms were located in a difference map and positioned with idealized geometry. They were refined using a riding model [C—H = 0.95 Å and $U_{iso}(H) = 1.2U_{eq}(C)$].

Data collection: *IPDS* (Stoe & Cie, 1998); cell refinement: *IPDS*; data reduction: *IPDS*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *CIFTAB* in *SHELXL97*.

References

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