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Bis(2,3,5,6-tetra-2-pyridylpyrazine-κ^2N^1,N^2,N^6)iron(II) tetrakis(thiocyanato-κN)ferrate(II) methanol solvate

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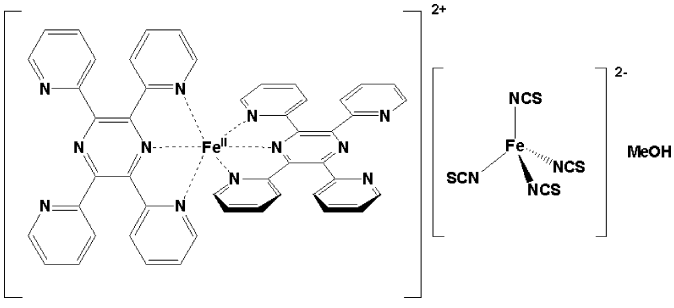
Key indicators: single-crystal X-ray study; T = 88 K; mean σ(C–C) = 0.003 Å; R factor = 0.035; wR factor = 0.075; data-to-parameter ratio = 16.8.

In the cation of the title compound, $[Fe(C_{24}H_{16}N_6)_2][Fe(NCS)_4]\cdot CH_3OH$ at 88 (2) K, the two hexadentate 2,3,5,6-tetra-2-pyridylpyrazine ligands coordinate to the Fe^{II} atom as tridentate ligands to give an octahedral mononuclear complex. In the anion, the tetrahedral Fe^{II} ion is coordinated by the N atoms of the four thiocyanate ions.

Related literature

For related literature, see: Batten *et al.* (1995); Fujita *et al.* (1995); Judge & Baker (1967); Matsuda *et al.* (2005); Schmidt *et al.* (1967).



Experimental

Crystal data

$[Fe(C_{24}H_{16}N_6)_2][Fe(NCS)_4]\cdot CH_4O$
M_r = 1152.92
Monoclinic, $P2_1/c$
a = 13.9846 (2) Å
b = 17.3102 (2) Å
c = 21.9143 (1) Å
β = 107.0252 (12)°
V = 5072.45 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.80 mm^{-1}
T = 88 (2) K
0.50 × 0.30 × 0.30 mm

Data collection

Rigaku R-AXIS RAPID imaging-plate diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.692, T_{max} = 0.796
43606 measured reflections
11576 independent reflections
8384 reflections with $I > 2\sigma(I)$
R_{int} = 0.045

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.075
S = 0.93
11576 reflections
690 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-3}

Table 1
Selected bond lengths (Å).

Fe1—N1	1.8658 (14)	Fe1—N9	1.9709 (15)
Fe1—N7	1.8676 (15)	Fe2—N16	1.9899 (18)
Fe1—N6	1.9557 (16)	Fe2—N13	1.9962 (18)
Fe1—N12	1.9680 (15)	Fe2—N15	1.997 (2)
Fe1—N3	1.9698 (16)	Fe2—N14	2.0073 (19)

Data collection: *PROCESS-AUTO* (Rigaku Corporation, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *TEXSAN* (Molecular Structure Corporation, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *TEXSAN*; molecular graphics: *Yadokari-XG* (Wakita, 2000); software used to prepare material for publication: *Yadokari-XG*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2223).

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supplementary materials

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Bis(2,3,5,6-tetra-2-pyridylpyrazine-κ^2N^1,N^2,N^6)iron(II) tetrakis(thiocyanato-κN)ferrate(II) methanol solvate

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Comment

Metal complexes are suitable compounds to construct supramolecular compounds because they can have various coordination geometries. In particular, complexes with multi-dentate ligands are interesting from a viewpoint of designing structure, because they can construct coordination polymers (Batten *et al.*, 1995; Fujita *et al.*, 1995). For example, porous compounds that can storage gas molecules are achieved by using metal complexes (Matsuda *et al.*, 2005). Moreover, such supramolecular compounds may be able to obtain more functions resulting from spin state of their central metal ions, for example, spin-crossover phenomenon when the metal ions have dynamic spin state. We focused on iron(II) complexes with terpyridine (= 2,2':6'2"-terpyridine) derivative ligands for the reasons mentioned above (Judge & Baker, 1967; Schmidt *et al.*, 1967) and here we report the synthesis and the crystal structure of the the title compound, (I) (Fig. 1). Compound (I) loses the methanol of solvation immediately on exposure to air, therefore the single-crystal measurement was performed in liquid paraffin.

The ligand tpypz is hexadentate, and there are two terpyridine (tpy) moieties on both sides of the ligand. However, one of tpy doesn't coordinate to the iron(II) ion. As a result, the cation is mononuclear with the iron atom Fe(1) coordinated to two tpypz ligands. The two tridentate ligands in the complex were found to be perpendicular to one another. The bond lengths of Fe(1)—N(tpypz) were assigned to a low-spin state of iron(II) compounds. The distance of Fe(1)—N(1, 7) is shorter than that of Fe(1)—N(3, 6, 9, 12), which exhibits a distorted FeN_6 octahedron. The coordinated terpyridine moiety of tpypz is co-planar with, for example, The torsion angle N(1)—C(1)—C(5)—N(3) = −10.83 (15)°. On the other hand, the non-coordinated terpyridine moiety is twisted out of the plane. with, for example, N(4)—C(10)—C(2)—C(1) = −49.97 (17)°.

In the anion, Fe(2) is coordinated to the nitrogen atoms of four thiocyanate ions in tetrahedral geometry. The bond lengths of Fe(2)—*N*(thiocyanate) were assigned to a high-spin state of iron(II) compounds.

Furthermore, there is intermolecular π-π stacking between the pyridine ring in non-coordinated terpyridine moiety and that in the terpyridine moiety of next complex. The bond length between C(13) and C(34) of the next complex is 3.1 Å.

Experimental

tpypz α-Pyridoin (20 g) and ammonium acetate (90 g) were mixed in a flask fitted with a reflux in air and slowly teated to 180 °C in an oil bath. The mixture soon became molten and very intense blown-green color developed. After about half an hour, Large white crystals appeared in the melt. The heating was continued for a further 1.5 h, with occasional shaking. The product was washed with alcohol and recrystallized from pyridine. The title complex (I) was prepared by additional of solutions of $FeCl_2{\cdot}4H_2O$ (0.20 g, 1 mmol) in methanol and KSCN (0.20 g, 2 mmol) in methanol to a solution of tpypz (0.78 g, 2 mmol) in methanol. Violet single crystals were obtained by diffusion in methanol. Calcd for $[Fe(tpypz)_2][Fe(NCS)_4]$, $C_{52}H_{34}Fe_2N_{16}OS_4$; C, 55.72; H, 2.88; N, 19.99. Found: C, 54.88; H, 2.95; N, 19.70%.

Refinement

The carbon H atoms were placed in calculated positions and allowed to ride during subsequent refinement, with *Uĩso*~(H) = 1.2*U*~*eq*~(C).

Figures

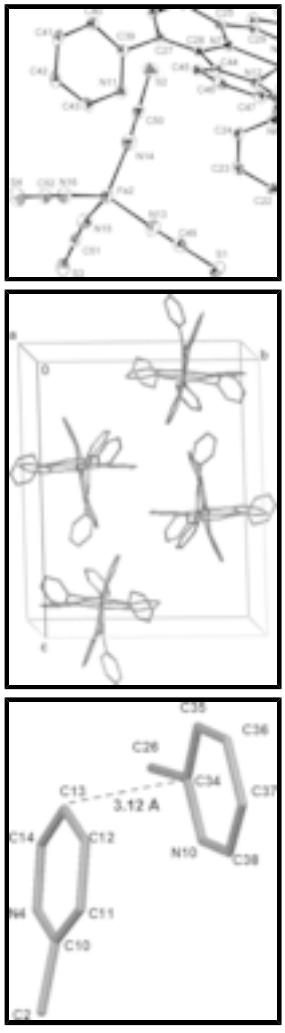

Figure 1. *ORTEP* drawing for the compound **1** showing 50% probability displacement ellipsoids.

Bis(2,3,5,6-tetra-2-pyridylpyrazine-κ^2*N*^{1},*N*^{2},*N*^{6})iron(II) tetrakis(thiocyanato-κ*N*)iron(II) methanol solvate

Crystal data

$[Fe(C_{24}H_{16}N_6)_2][Fe(NCS)_4]\cdot CH_4O$	F_{000} = 2360
M_r = 1152.92	D_x = 1.510 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71069 Å
Hall symbol: -P 2ybc	Cell parameters from 34587 reflections

a = 13.9846 (2) Å	θ = 2.4–27.5º
b = 17.3102 (2) Å	μ = 0.80 mm^{-1}
c = 21.9143 (1) Å	T = 88 (2) K
β = 107.0252 (12)º	Block, violet
V = 5072.45 (10) Å^3	0.50 × 0.30 × 0.30 mm
Z = 4	

Data collection

Rigaku R-AXIS RAPID imaging-plate diffractometer	11576 independent reflections
Radiation source: fine-focus sealed tube	8384 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.045
Detector resolution: 10.00 pixels mm^{-1}	θ_{max} = 27.5º
T = 88(2) K	θ_{min} = 1.9º
ω scans	h = 0→18
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	k = 0→22
T_{min} = 0.692, T_{max} = 0.796	l = −28→27
43606 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.035	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.075	$w = 1/[\sigma^2(F_o^2) + (0.0354P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.93	$(\Delta/\sigma)_{max}$ = 0.001
11576 reflections	$\Delta\rho_{max}$ = 0.49 e Å^{-3}
690 parameters	$\Delta\rho_{min}$ = −0.44 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
Fe1	0.453755 (19)	0.211279 (15)	0.422430 (13)	0.01215 (7)
N1	0.58227 (11)	0.20286 (9)	0.41396 (7)	0.0127 (3)
N2	0.77347 (11)	0.19119 (9)	0.40812 (8)	0.0161 (3)
N3	0.46704 (11)	0.09801 (9)	0.42791 (7)	0.0132 (3)
N4	0.78731 (11)	0.01862 (9)	0.48366 (8)	0.0177 (4)
N5	0.76085 (12)	0.36864 (10)	0.34373 (8)	0.0204 (4)
N6	0.47812 (11)	0.32104 (9)	0.41192 (7)	0.0145 (3)
N7	0.32731 (11)	0.21983 (9)	0.43436 (7)	0.0131 (3)
N8	0.14399 (11)	0.22695 (9)	0.45320 (7)	0.0136 (3)
N9	0.49583 (11)	0.23149 (9)	0.51499 (7)	0.0130 (3)
N10	0.27582 (11)	0.16279 (9)	0.60599 (7)	0.0150 (3)
N11	0.06851 (11)	0.29405 (9)	0.29740 (8)	0.0173 (4)
N12	0.37209 (11)	0.19415 (9)	0.33370 (7)	0.0140 (3)
C1	0.62430 (13)	0.13222 (11)	0.41644 (9)	0.0132 (4)
C2	0.72449 (13)	0.12733 (11)	0.41655 (9)	0.0152 (4)
C3	0.73002 (13)	0.26037 (11)	0.40467 (9)	0.0156 (4)
C4	0.63346 (13)	0.26831 (11)	0.41043 (9)	0.0141 (4)
C5	0.55292 (13)	0.07035 (11)	0.41797 (9)	0.0139 (4)
C6	0.56427 (13)	−0.00732 (11)	0.40691 (9)	0.0155 (4)
H1	0.6240	−0.0256	0.3995	0.019*
C7	0.48765 (14)	−0.05810 (11)	0.40674 (9)	0.0174 (4)
H2	0.4933	−0.1113	0.3978	0.021*
C8	0.40271 (14)	−0.03060 (11)	0.41972 (9)	0.0178 (4)
H3	0.3506	−0.0648	0.4216	0.021*
C9	0.39538 (13)	0.04745 (11)	0.42983 (9)	0.0155 (4)
H4	0.3369	0.0663	0.4386	0.019*
C10	0.78729 (13)	0.05647 (11)	0.43032 (9)	0.0153 (4)
C11	0.84838 (13)	0.03755 (11)	0.39256 (10)	0.0185 (4)
H5	0.8447	0.0656	0.3547	0.022*
C12	0.91495 (14)	−0.02353 (12)	0.41181 (11)	0.0226 (5)
H6	0.9572	−0.0388	0.3870	0.027*
C13	0.91821 (14)	−0.06132 (12)	0.46766 (10)	0.0230 (5)
H7	0.9644	−0.1022	0.4827	0.028*
C14	0.85359 (14)	−0.03918 (11)	0.50157 (10)	0.0209 (4)
H8	0.8562	−0.0664	0.5397	0.025*
C15	0.79512 (14)	0.32617 (11)	0.39661 (10)	0.0180 (4)
C16	0.88689 (14)	0.33683 (13)	0.44099 (11)	0.0262 (5)
H9	0.9081	0.3048	0.4777	0.031*
C17	0.94734 (16)	0.39561 (14)	0.43050 (13)	0.0353 (6)
H10	1.0107	0.4054	0.4604	0.042*
C18	0.91434 (16)	0.43942 (13)	0.37639 (12)	0.0321 (6)
H11	0.9550	0.4796	0.3680	0.038*
C19	0.82147 (16)	0.42467 (12)	0.33418 (11)	0.0262 (5)
H12	0.7994	0.4554	0.2968	0.031*
C20	0.57463 (13)	0.33802 (11)	0.41285 (9)	0.0144 (4)

C21	0.60754 (14)	0.41395 (11)	0.41788 (9)	0.0172 (4)
H13	0.6751	0.4253	0.4206	0.021*
C22	0.54166 (14)	0.47321 (11)	0.41893 (10)	0.0198 (4)
H14	0.5636	0.5254	0.4222	0.024*
C23	0.44394 (14)	0.45590 (12)	0.41523 (10)	0.0198 (4)
H15	0.3972	0.4959	0.4148	0.024*
C24	0.41536 (14)	0.37947 (11)	0.41211 (9)	0.0172 (4)
H16	0.3481	0.3677	0.4100	0.021*
C25	0.32048 (13)	0.22924 (10)	0.49421 (9)	0.0125 (4)
C26	0.22612 (13)	0.22400 (10)	0.50357 (9)	0.0131 (4)
C27	0.15187 (13)	0.22736 (10)	0.39374 (9)	0.0139 (4)
C28	0.24531 (13)	0.21500 (10)	0.38325 (9)	0.0125 (4)
C29	0.41888 (13)	0.24529 (11)	0.53989 (9)	0.0136 (4)
C30	0.43471 (13)	0.27611 (11)	0.60043 (9)	0.0161 (4)
H17	0.3796	0.2869	0.6161	0.019*
C31	0.53114 (14)	0.29106 (12)	0.63790 (10)	0.0197 (4)
H18	0.5433	0.3129	0.6792	0.024*
C32	0.60994 (14)	0.27329 (11)	0.61353 (9)	0.0189 (4)
H19	0.6772	0.2809	0.6388	0.023*
C33	0.58948 (13)	0.24456 (11)	0.55259 (9)	0.0154 (4)
H20	0.6438	0.2334	0.5363	0.018*
C34	0.21025 (13)	0.21104 (11)	0.56702 (9)	0.0135 (4)
C35	0.12820 (13)	0.24239 (11)	0.58155 (9)	0.0148 (4)
H21	0.0832	0.2757	0.5523	0.018*
C36	0.11340 (14)	0.22401 (11)	0.63941 (9)	0.0168 (4)
H22	0.0587	0.2453	0.6512	0.020*
C37	0.17961 (14)	0.17393 (11)	0.68005 (9)	0.0174 (4)
H23	0.1707	0.1598	0.7199	0.021*
C38	0.25889 (14)	0.14489 (11)	0.66145 (9)	0.0170 (4)
H24	0.3038	0.1104	0.6895	0.020*
C39	0.05891 (13)	0.24365 (11)	0.34190 (9)	0.0140 (4)
C40	−0.03118 (13)	0.21050 (12)	0.34217 (9)	0.0176 (4)
H25	−0.0347	0.1755	0.3749	0.021*
C41	−0.11614 (14)	0.22990 (12)	0.29331 (9)	0.0210 (5)
H26	−0.1788	0.2068	0.2909	0.025*
C42	−0.10818 (14)	0.28334 (12)	0.24825 (9)	0.0215 (5)
H27	−0.1655	0.2988	0.2151	0.026*
C43	−0.01489 (14)	0.31398 (12)	0.25234 (9)	0.0190 (4)
H28	−0.0101	0.3511	0.2214	0.023*
C44	0.27113 (13)	0.19284 (10)	0.32537 (9)	0.0135 (4)
C45	0.20554 (13)	0.16436 (11)	0.26963 (9)	0.0151 (4)
H29	0.1356	0.1642	0.2642	0.018*
C46	0.24257 (14)	0.13616 (11)	0.22189 (9)	0.0171 (4)
H30	0.1985	0.1152	0.1839	0.021*
C47	0.34447 (14)	0.13886 (11)	0.23012 (9)	0.0179 (4)
H31	0.3714	0.1201	0.1979	0.021*
C48	0.40621 (14)	0.16937 (11)	0.28604 (9)	0.0163 (4)
H32	0.4758	0.1729	0.2909	0.020*
Fe2	0.14977 (2)	0.566702 (18)	0.346194 (14)	0.02213 (8)

N13	0.25708 (13)	0.56221 (10)	0.30301 (9)	0.0261 (4)
C49	0.31767 (14)	0.55521 (12)	0.27660 (10)	0.0210 (4)
S1	0.40341 (4)	0.54587 (3)	0.24046 (3)	0.02538 (13)
N14	0.18739 (13)	0.49490 (11)	0.42138 (9)	0.0283 (4)
C50	0.20445 (14)	0.46266 (12)	0.47034 (11)	0.0214 (5)
S2	0.22749 (4)	0.41761 (3)	0.53795 (3)	0.02662 (13)
N15	0.14185 (13)	0.66939 (11)	0.38600 (9)	0.0278 (4)
C51	0.14694 (14)	0.73203 (13)	0.40514 (10)	0.0211 (5)
S3	0.15428 (4)	0.82075 (3)	0.43038 (3)	0.02546 (13)
N16	0.02412 (13)	0.54473 (11)	0.27726 (9)	0.0276 (4)
C52	−0.03876 (15)	0.52652 (12)	0.23213 (11)	0.0235 (5)
S4	−0.12829 (4)	0.50030 (3)	0.16981 (3)	0.03002 (14)
O1	0.59213 (13)	0.29637 (10)	0.26346 (8)	0.0421 (4)
H33	0.656 (3)	0.326 (2)	0.2874 (17)	0.104 (13)*
C53	0.51510 (17)	0.35184 (15)	0.24737 (11)	0.0356 (6)
H34	0.4769	0.3497	0.2784	0.053*
H35	0.4706	0.3409	0.2046	0.053*
H36	0.5442	0.4034	0.2480	0.053*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.00903 (12)	0.01469 (14)	0.01263 (13)	−0.00061 (10)	0.00299 (10)	−0.00022 (11)
N1	0.0109 (7)	0.0165 (8)	0.0103 (8)	−0.0006 (6)	0.0027 (6)	−0.0008 (7)
N2	0.0130 (8)	0.0183 (9)	0.0176 (8)	−0.0003 (6)	0.0053 (7)	0.0001 (7)
N3	0.0125 (8)	0.0173 (8)	0.0098 (7)	−0.0011 (6)	0.0035 (6)	0.0003 (7)
N4	0.0150 (8)	0.0164 (9)	0.0187 (9)	−0.0016 (6)	0.0000 (7)	−0.0017 (7)
N5	0.0220 (9)	0.0198 (9)	0.0227 (9)	0.0006 (7)	0.0119 (8)	0.0013 (8)
N6	0.0111 (7)	0.0188 (9)	0.0125 (8)	−0.0014 (6)	0.0017 (6)	0.0003 (7)
N7	0.0109 (7)	0.0135 (8)	0.0141 (8)	−0.0006 (6)	0.0024 (6)	−0.0003 (7)
N8	0.0117 (7)	0.0145 (8)	0.0135 (8)	−0.0004 (6)	0.0018 (6)	0.0001 (7)
N9	0.0111 (7)	0.0136 (8)	0.0139 (8)	−0.0002 (6)	0.0030 (6)	−0.0005 (7)
N10	0.0114 (8)	0.0178 (9)	0.0147 (8)	−0.0020 (6)	0.0021 (6)	−0.0001 (7)
N11	0.0150 (8)	0.0215 (9)	0.0149 (8)	0.0022 (7)	0.0035 (7)	0.0008 (7)
N12	0.0128 (8)	0.0152 (8)	0.0140 (8)	−0.0001 (6)	0.0040 (6)	0.0008 (7)
C1	0.0132 (9)	0.0159 (10)	0.0104 (9)	0.0001 (7)	0.0032 (7)	0.0004 (8)
C2	0.0141 (9)	0.0209 (11)	0.0107 (9)	−0.0008 (8)	0.0037 (8)	−0.0021 (8)
C3	0.0136 (9)	0.0194 (10)	0.0138 (9)	−0.0010 (8)	0.0039 (8)	0.0006 (8)
C4	0.0126 (9)	0.0166 (10)	0.0125 (9)	−0.0014 (7)	0.0030 (7)	0.0000 (8)
C5	0.0109 (9)	0.0195 (10)	0.0110 (9)	0.0000 (7)	0.0030 (7)	−0.0004 (8)
C6	0.0156 (9)	0.0183 (10)	0.0120 (9)	0.0021 (8)	0.0033 (8)	0.0013 (8)
C7	0.0199 (10)	0.0141 (10)	0.0161 (10)	−0.0007 (8)	0.0020 (8)	0.0011 (8)
C8	0.0154 (9)	0.0190 (10)	0.0172 (10)	−0.0049 (8)	0.0019 (8)	0.0025 (9)
C9	0.0121 (9)	0.0206 (10)	0.0138 (9)	−0.0003 (7)	0.0040 (8)	0.0016 (8)
C10	0.0103 (9)	0.0152 (10)	0.0188 (10)	−0.0027 (7)	0.0019 (8)	−0.0046 (8)
C11	0.0118 (9)	0.0206 (11)	0.0226 (10)	−0.0040 (8)	0.0044 (8)	−0.0049 (9)
C12	0.0092 (9)	0.0207 (11)	0.0365 (13)	−0.0051 (8)	0.0048 (9)	−0.0136 (10)
C13	0.0116 (9)	0.0165 (10)	0.0346 (12)	0.0000 (8)	−0.0031 (9)	−0.0074 (10)

C14	0.0178 (10)	0.0146 (10)	0.0246 (11)	−0.0046 (8)	−0.0027 (9)	−0.0020 (9)
C15	0.0148 (9)	0.0171 (10)	0.0258 (11)	0.0001 (8)	0.0115 (8)	−0.0013 (9)
C16	0.0162 (10)	0.0287 (12)	0.0322 (13)	−0.0034 (9)	0.0048 (9)	0.0069 (10)
C17	0.0172 (11)	0.0371 (14)	0.0506 (16)	−0.0085 (10)	0.0085 (11)	0.0046 (13)
C18	0.0265 (12)	0.0277 (13)	0.0490 (15)	−0.0091 (10)	0.0220 (11)	0.0012 (12)
C19	0.0340 (12)	0.0222 (11)	0.0294 (12)	0.0009 (9)	0.0201 (10)	0.0025 (10)
C20	0.0119 (9)	0.0185 (10)	0.0129 (9)	−0.0012 (7)	0.0040 (7)	−0.0004 (8)
C21	0.0138 (9)	0.0192 (10)	0.0192 (10)	−0.0019 (8)	0.0059 (8)	−0.0007 (9)
C22	0.0216 (10)	0.0151 (10)	0.0234 (11)	−0.0031 (8)	0.0080 (9)	−0.0002 (9)
C23	0.0189 (10)	0.0177 (10)	0.0229 (11)	0.0045 (8)	0.0060 (9)	0.0004 (9)
C24	0.0115 (9)	0.0218 (11)	0.0182 (10)	−0.0001 (8)	0.0042 (8)	0.0011 (9)
C25	0.0134 (9)	0.0124 (10)	0.0120 (9)	0.0005 (7)	0.0040 (7)	−0.0009 (8)
C26	0.0124 (9)	0.0125 (9)	0.0144 (9)	0.0006 (7)	0.0038 (8)	−0.0013 (8)
C27	0.0144 (9)	0.0127 (10)	0.0144 (9)	−0.0011 (7)	0.0041 (8)	0.0006 (8)
C28	0.0124 (8)	0.0108 (9)	0.0134 (9)	−0.0022 (7)	0.0025 (7)	0.0010 (8)
C29	0.0110 (9)	0.0147 (10)	0.0148 (9)	−0.0005 (7)	0.0035 (8)	0.0005 (8)
C30	0.0138 (9)	0.0178 (10)	0.0176 (10)	−0.0006 (7)	0.0060 (8)	−0.0019 (8)
C31	0.0186 (10)	0.0221 (11)	0.0170 (10)	−0.0023 (8)	0.0031 (8)	−0.0030 (9)
C32	0.0123 (9)	0.0240 (11)	0.0173 (10)	−0.0041 (8)	−0.0003 (8)	−0.0014 (9)
C33	0.0092 (9)	0.0185 (10)	0.0181 (10)	−0.0003 (7)	0.0037 (8)	0.0027 (8)
C34	0.0111 (9)	0.0154 (10)	0.0130 (9)	−0.0049 (7)	0.0017 (7)	−0.0026 (8)
C35	0.0116 (9)	0.0158 (10)	0.0156 (9)	−0.0017 (7)	0.0019 (7)	−0.0013 (8)
C36	0.0133 (9)	0.0187 (10)	0.0190 (10)	−0.0036 (8)	0.0055 (8)	−0.0060 (9)
C37	0.0193 (10)	0.0210 (11)	0.0120 (9)	−0.0051 (8)	0.0049 (8)	−0.0019 (8)
C38	0.0144 (9)	0.0188 (10)	0.0156 (10)	−0.0015 (8)	0.0007 (8)	−0.0001 (8)
C39	0.0133 (9)	0.0169 (10)	0.0117 (9)	0.0010 (7)	0.0035 (8)	−0.0015 (8)
C40	0.0157 (9)	0.0230 (11)	0.0145 (9)	0.0002 (8)	0.0048 (8)	0.0008 (9)
C41	0.0113 (9)	0.0322 (12)	0.0189 (10)	−0.0006 (8)	0.0032 (8)	−0.0044 (10)
C42	0.0150 (9)	0.0330 (12)	0.0141 (10)	0.0070 (9)	0.0007 (8)	−0.0030 (9)
C43	0.0197 (10)	0.0244 (11)	0.0137 (9)	0.0081 (8)	0.0062 (8)	0.0030 (9)
C44	0.0136 (9)	0.0129 (9)	0.0144 (9)	0.0015 (7)	0.0047 (8)	0.0020 (8)
C45	0.0138 (9)	0.0153 (10)	0.0148 (9)	−0.0002 (8)	0.0021 (8)	0.0028 (8)
C46	0.0209 (10)	0.0164 (10)	0.0117 (9)	0.0009 (8)	0.0008 (8)	0.0009 (8)
C47	0.0219 (10)	0.0185 (10)	0.0152 (9)	0.0039 (8)	0.0082 (8)	0.0011 (9)
C48	0.0156 (9)	0.0181 (10)	0.0165 (10)	0.0009 (8)	0.0068 (8)	0.0028 (8)
Fe2	0.01638 (14)	0.02719 (18)	0.02248 (16)	−0.00018 (12)	0.00518 (12)	0.00178 (14)
N13	0.0207 (9)	0.0312 (11)	0.0271 (10)	−0.0007 (8)	0.0081 (8)	−0.0009 (9)
C49	0.0198 (10)	0.0208 (11)	0.0182 (10)	−0.0024 (8)	−0.0011 (9)	−0.0006 (9)
S1	0.0250 (3)	0.0299 (3)	0.0240 (3)	−0.0052 (2)	0.0116 (2)	−0.0049 (2)
N14	0.0216 (9)	0.0329 (11)	0.0293 (11)	−0.0009 (8)	0.0057 (8)	0.0027 (9)
C50	0.0138 (9)	0.0199 (11)	0.0300 (12)	−0.0026 (8)	0.0058 (9)	−0.0031 (10)
S2	0.0244 (3)	0.0227 (3)	0.0285 (3)	−0.0043 (2)	0.0012 (2)	0.0047 (2)
N15	0.0238 (10)	0.0320 (11)	0.0288 (10)	−0.0004 (8)	0.0097 (8)	0.0023 (9)
C51	0.0120 (9)	0.0340 (13)	0.0168 (10)	−0.0006 (8)	0.0036 (8)	0.0023 (10)
S3	0.0208 (3)	0.0298 (3)	0.0233 (3)	0.0009 (2)	0.0026 (2)	−0.0037 (2)
N16	0.0210 (9)	0.0313 (11)	0.0286 (10)	−0.0012 (8)	0.0042 (8)	0.0041 (9)
C52	0.0224 (11)	0.0228 (11)	0.0277 (12)	0.0054 (9)	0.0112 (10)	0.0041 (10)
S4	0.0296 (3)	0.0306 (3)	0.0248 (3)	0.0041 (2)	0.0001 (2)	−0.0053 (3)
O1	0.0383 (10)	0.0369 (10)	0.0378 (10)	0.0050 (8)	−0.0095 (8)	−0.0057 (8)

C53	0.0307 (13)	0.0450 (15)	0.0252 (12)	0.0057 (11)	−0.0011 (10)	0.0023 (12)

Geometric parameters (Å, °)

Fe1—N1	1.8658 (14)	C21—H13	0.9500
Fe1—N7	1.8676 (15)	C22—C23	1.378 (3)
Fe1—N6	1.9557 (16)	C22—H14	0.9500
Fe1—N12	1.9680 (15)	C23—C24	1.378 (3)
Fe1—N3	1.9698 (16)	C23—H15	0.9500
Fe1—N9	1.9709 (15)	C24—H16	0.9500
N1—C1	1.351 (2)	C25—C26	1.396 (2)
N1—C4	1.354 (2)	C25—C29	1.472 (2)
N2—C3	1.335 (2)	C26—C34	1.488 (3)
N2—C2	1.342 (2)	C27—C28	1.408 (2)
N3—C9	1.340 (2)	C27—C39	1.482 (2)
N3—C5	1.368 (2)	C28—C44	1.468 (3)
N4—C10	1.340 (2)	C29—C30	1.386 (3)
N4—C14	1.342 (2)	C30—C31	1.383 (3)
N5—C15	1.337 (3)	C30—H17	0.9500
N5—C19	1.344 (3)	C31—C32	1.392 (3)
N6—C24	1.340 (2)	C31—H18	0.9500
N6—C20	1.376 (2)	C32—C33	1.374 (3)
N7—C28	1.351 (2)	C32—H19	0.9500
N7—C25	1.353 (2)	C33—H20	0.9500
N8—C27	1.340 (2)	C34—C35	1.388 (2)
N8—C26	1.341 (2)	C35—C36	1.380 (3)
N9—C33	1.347 (2)	C35—H21	0.9500
N9—C29	1.363 (2)	C36—C37	1.385 (3)
N10—C38	1.341 (2)	C36—H22	0.9500
N10—C34	1.345 (2)	C37—C38	1.383 (3)
N11—C43	1.334 (2)	C37—H23	0.9500
N11—C39	1.344 (2)	C38—H24	0.9500
N12—C48	1.339 (2)	C39—C40	1.386 (3)
N12—C44	1.370 (2)	C40—C41	1.388 (3)
C1—C2	1.403 (2)	C40—H25	0.9500
C1—C5	1.471 (3)	C41—C42	1.381 (3)
C2—C10	1.487 (3)	C41—H26	0.9500
C3—C4	1.399 (2)	C42—C43	1.387 (3)
C3—C15	1.501 (3)	C42—H27	0.9500
C4—C20	1.470 (3)	C43—H28	0.9500
C5—C6	1.384 (3)	C44—C45	1.386 (3)
C6—C7	1.385 (3)	C45—C46	1.385 (3)
C6—H1	0.9500	C45—H29	0.9500
C7—C8	1.384 (3)	C46—C47	1.384 (3)
C7—H2	0.9500	C46—H30	0.9500
C8—C9	1.378 (3)	C47—C48	1.381 (3)
C8—H3	0.9500	C47—H31	0.9500
C9—H4	0.9500	C48—H32	0.9500
C10—C11	1.391 (3)	Fe2—N16	1.9899 (18)

C11—C12	1.390 (3)	Fe2—N13	1.9962 (18)
C11—H5	0.9500	Fe2—N15	1.997 (2)
C12—C13	1.377 (3)	Fe2—N14	2.0073 (19)
C12—H6	0.9500	N13—C49	1.164 (2)
C13—C14	1.382 (3)	C49—S1	1.627 (2)
C13—H7	0.9500	N14—C50	1.170 (3)
C14—H8	0.9500	C50—S2	1.621 (2)
C15—C16	1.377 (3)	N15—C51	1.157 (3)
C16—C17	1.384 (3)	C51—S3	1.625 (2)
C16—H9	0.9500	N16—C52	1.158 (3)
C17—C18	1.369 (3)	C52—S4	1.624 (2)
C17—H10	0.9500	O1—C53	1.409 (3)
C18—C19	1.380 (3)	O1—H33	1.03 (4)
C18—H11	0.9500	C53—H34	0.9800
C19—H12	0.9500	C53—H35	0.9800
C20—C21	1.386 (3)	C53—H36	0.9800
C21—C22	1.383 (3)		
N1—Fe1—N7	177.77 (7)	C23—C22—C21	119.45 (18)
N1—Fe1—N6	81.84 (6)	C23—C22—H14	120.3
N7—Fe1—N6	98.29 (6)	C21—C22—H14	120.3
N1—Fe1—N12	100.79 (6)	C24—C23—C22	118.67 (18)
N7—Fe1—N12	81.43 (6)	C24—C23—H15	120.7
N6—Fe1—N12	95.78 (6)	C22—C23—H15	120.7
N1—Fe1—N3	81.48 (6)	N6—C24—C23	122.97 (17)
N7—Fe1—N3	98.43 (6)	N6—C24—H16	118.5
N6—Fe1—N3	163.25 (6)	C23—C24—H16	118.5
N12—Fe1—N3	85.83 (6)	N7—C25—C26	118.21 (16)
N1—Fe1—N9	96.33 (6)	N7—C25—C29	111.39 (15)
N7—Fe1—N9	81.46 (6)	C26—C25—C29	130.39 (17)
N6—Fe1—N9	86.50 (6)	N8—C26—C25	119.68 (17)
N12—Fe1—N9	162.88 (6)	N8—C26—C34	116.67 (15)
N3—Fe1—N9	96.87 (6)	C25—C26—C34	123.57 (16)
C1—N1—C4	121.91 (15)	N8—C27—C28	120.18 (17)
C1—N1—Fe1	119.15 (12)	N8—C27—C39	116.30 (16)
C4—N1—Fe1	118.74 (12)	C28—C27—C39	123.49 (16)
C3—N2—C2	120.31 (16)	N7—C28—C27	117.29 (16)
C9—N3—C5	118.40 (16)	N7—C28—C44	111.64 (15)
C9—N3—Fe1	126.42 (13)	C27—C28—C44	130.97 (16)
C5—N3—Fe1	114.21 (12)	N9—C29—C30	121.81 (16)
C10—N4—C14	116.32 (17)	N9—C29—C25	112.67 (16)
C15—N5—C19	116.70 (18)	C30—C29—C25	125.39 (16)
C24—N6—C20	118.64 (16)	C31—C30—C29	119.68 (17)
C24—N6—Fe1	126.59 (13)	C31—C30—H17	120.2
C20—N6—Fe1	114.20 (12)	C29—C30—H17	120.2
C28—N7—C25	121.85 (15)	C30—C31—C32	118.34 (18)
C28—N7—Fe1	119.20 (12)	C30—C31—H18	120.8
C25—N7—Fe1	118.94 (12)	C32—C31—H18	120.8
C27—N8—C26	120.42 (16)	C33—C32—C31	119.32 (17)
C33—N9—C29	117.79 (16)	C33—C32—H19	120.3

C33—N9—Fe1	127.11 (12)	C31—C32—H19	120.3
C29—N9—Fe1	114.23 (12)	N9—C33—C32	122.94 (17)
C38—N10—C34	116.78 (16)	N9—C33—H20	118.5
C43—N11—C39	116.97 (16)	C32—C33—H20	118.5
C48—N12—C44	118.64 (16)	N10—C34—C35	123.56 (17)
C48—N12—Fe1	125.63 (12)	N10—C34—C26	115.25 (16)
C44—N12—Fe1	114.30 (12)	C35—C34—C26	121.00 (16)
N1—C1—C2	118.50 (16)	C36—C35—C34	118.49 (18)
N1—C1—C5	111.70 (15)	C36—C35—H21	120.8
C2—C1—C5	129.79 (17)	C34—C35—H21	120.8
N2—C2—C1	120.00 (17)	C35—C36—C37	118.89 (18)
N2—C2—C10	114.37 (15)	C35—C36—H22	120.6
C1—C2—C10	125.47 (17)	C37—C36—H22	120.6
N2—C3—C4	121.23 (17)	C38—C37—C36	118.72 (18)
N2—C3—C15	113.94 (16)	C38—C37—H23	120.6
C4—C3—C15	124.80 (17)	C36—C37—H23	120.6
N1—C4—C3	117.58 (17)	N10—C38—C37	123.56 (18)
N1—C4—C20	111.96 (15)	N10—C38—H24	118.2
C3—C4—C20	130.46 (17)	C37—C38—H24	118.2
N3—C5—C6	121.27 (16)	N11—C39—C40	123.76 (17)
N3—C5—C1	112.45 (16)	N11—C39—C27	115.35 (15)
C6—C5—C1	126.16 (17)	C40—C39—C27	120.84 (17)
C5—C6—C7	119.27 (17)	C39—C40—C41	118.07 (18)
C5—C6—H1	120.4	C39—C40—H25	121.0
C7—C6—H1	120.4	C41—C40—H25	121.0
C8—C7—C6	119.40 (18)	C42—C41—C40	118.93 (18)
C8—C7—H2	120.3	C42—C41—H26	120.5
C6—C7—H2	120.3	C40—C41—H26	120.5
C9—C8—C7	118.62 (17)	C41—C42—C43	118.77 (18)
C9—C8—H3	120.7	C41—C42—H27	120.6
C7—C8—H3	120.7	C43—C42—H27	120.6
N3—C9—C8	122.93 (17)	N11—C43—C42	123.42 (19)
N3—C9—H4	118.5	N11—C43—H28	118.3
C8—C9—H4	118.5	C42—C43—H28	118.3
N4—C10—C11	124.08 (18)	N12—C44—C45	120.92 (17)
N4—C10—C2	115.44 (16)	N12—C44—C28	112.66 (16)
C11—C10—C2	120.16 (18)	C45—C44—C28	126.00 (16)
C12—C11—C10	118.15 (19)	C46—C45—C44	119.59 (17)
C12—C11—H5	120.9	C46—C45—H29	120.2
C10—C11—H5	120.9	C44—C45—H29	120.2
C13—C12—C11	118.42 (19)	C47—C46—C45	119.22 (18)
C13—C12—H6	120.8	C47—C46—H30	120.4
C11—C12—H6	120.8	C45—C46—H30	120.4
C12—C13—C14	119.33 (19)	C48—C47—C46	118.76 (18)
C12—C13—H7	120.3	C48—C47—H31	120.6
C14—C13—H7	120.3	C46—C47—H31	120.6
N4—C14—C13	123.6 (2)	N12—C48—C47	122.79 (17)
N4—C14—H8	118.2	N12—C48—H32	118.6
C13—C14—H8	118.2	C47—C48—H32	118.6

N5—C15—C16	124.19 (18)	N16—Fe2—N13	104.60 (7)
N5—C15—C3	116.33 (17)	N16—Fe2—N15	110.61 (8)
C16—C15—C3	119.39 (18)	N13—Fe2—N15	112.78 (7)
C15—C16—C17	118.0 (2)	N16—Fe2—N14	117.76 (7)
C15—C16—H9	121.0	N13—Fe2—N14	108.20 (7)
C17—C16—H9	121.0	N15—Fe2—N14	103.13 (8)
C18—C17—C16	118.9 (2)	C49—N13—Fe2	175.94 (18)
C18—C17—H10	120.5	N13—C49—S1	179.3 (2)
C16—C17—H10	120.5	C50—N14—Fe2	169.10 (18)
C17—C18—C19	119.4 (2)	N14—C50—S2	179.6 (2)
C17—C18—H11	120.3	C51—N15—Fe2	170.66 (17)
C19—C18—H11	120.3	N15—C51—S3	178.6 (2)
N5—C19—C18	122.8 (2)	C52—N16—Fe2	168.85 (18)
N5—C19—H12	118.6	N16—C52—S4	178.8 (2)
C18—C19—H12	118.6	C53—O1—H33	106 (2)
N6—C20—C21	120.35 (17)	O1—C53—H34	109.5
N6—C20—C4	112.40 (16)	O1—C53—H35	109.5
C21—C20—C4	127.22 (16)	H34—C53—H35	109.5
C22—C21—C20	119.81 (17)	O1—C53—H36	109.5
C22—C21—H13	120.1	H34—C53—H36	109.5
C20—C21—H13	120.1	H35—C53—H36	109.5

Fig. 1

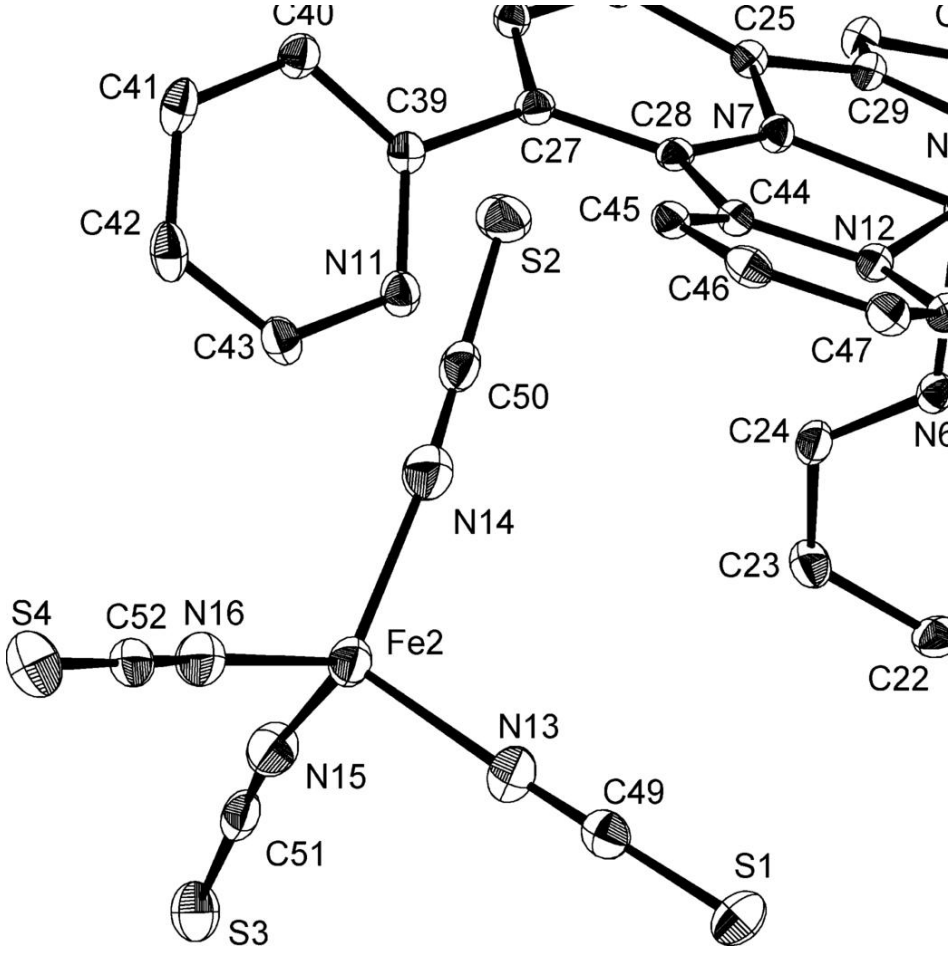
C40
C25
C41
C39
C28
N7
C29
C27
C44
C45
C42
N12
N11
S2
C46
C43
C47
C50
C24
N14
C23
S4
C52
N16
Fe2
C22
N13
N15
C49
C51
S1
S3

Fig. 2

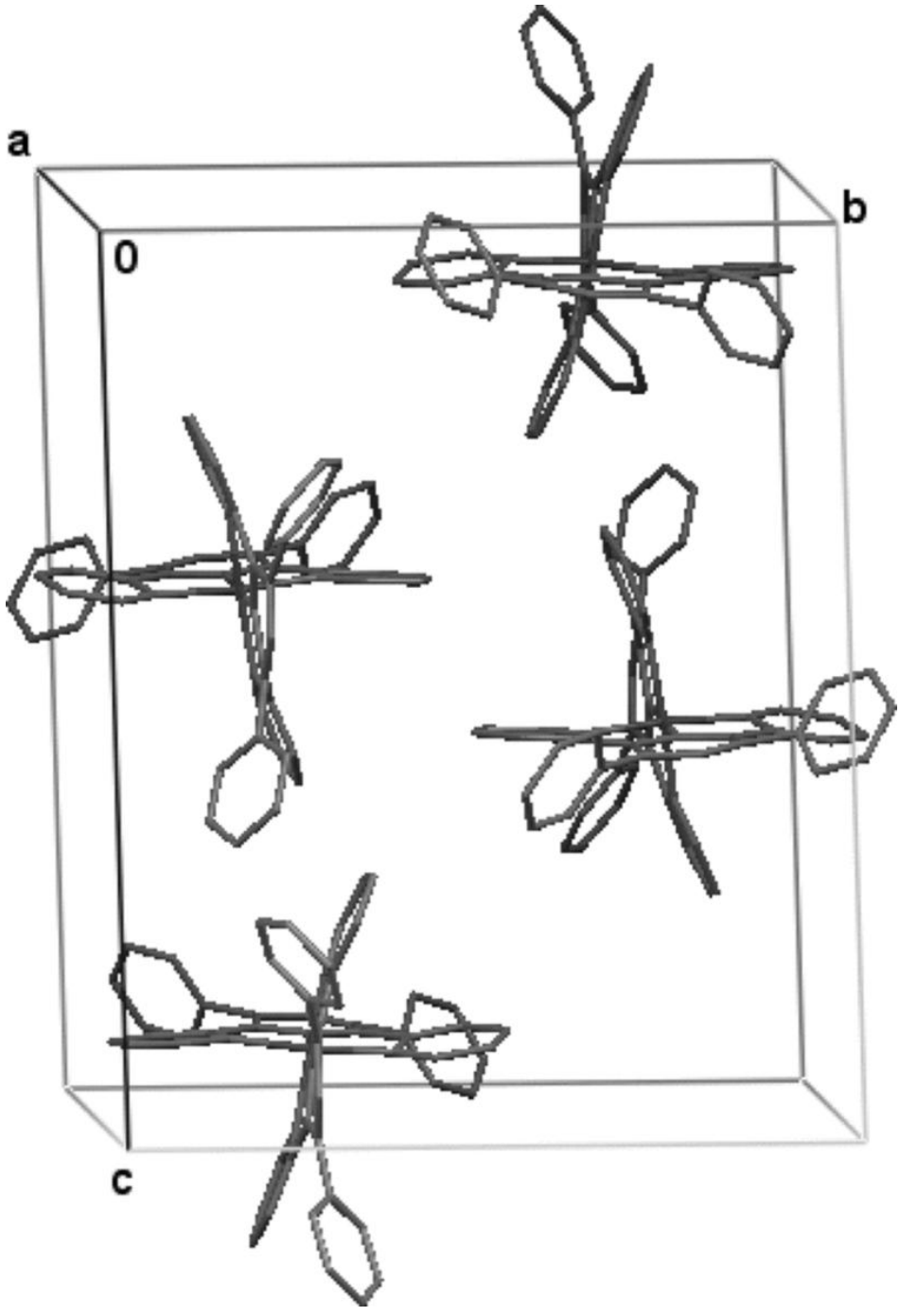

Fig. 3