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Crystal structures and spectroscopic characterization of $MBr_2(CNXyl)_n$ (M = Fe and Co, n = 4; M = Ni, n = 2; Xyl = 2,6-dimethylphenyl), and of formally zero-valent iron as a cocrystal of $Fe(CNXyl)_5$ and $Fe_2(CNXyl)_9$

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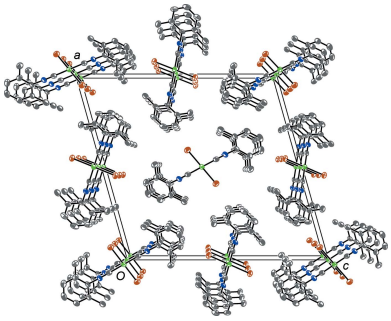
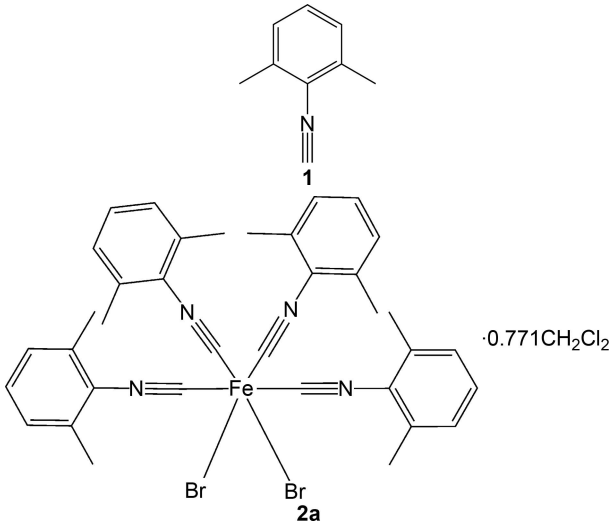
Structures and spectroscopic characterization of the divalent complexes *cis*-dibromidotetrakis(2,6-dimethylphenyl isocyanide)iron(II) dichloromethane 0.771-solvate, $[FeBr_2(C_9H_9N)_4]\cdot 0.771CH_2Cl_2$ or *cis*-$FeBr_2(CNXyl)_4\cdot 0.771CH_2Cl_2$ (Xyl = 2,6-dimethylphenyl), *trans*-dibromidotetrakis(2,6-dimethylphenyl isocyanide)-iron(II), $[FeBr_2(C_9H_9N)_4]$ or *trans*-$FeBr_2(CNXyl)_4$, *trans*-dibromidotetrakis(2,6-dimethylphenyl isocyanide)cobalt(II), $[CoBr_2(C_9H_9N)_4]$ or *trans*-$CoBr_2(CNXyl)_4$, and *trans*-dibromidobis(2,6-dimethylphenyl isocyanide)nickel(II), $[NiBr_2(C_9H_9N)_2]$ or *trans*-$NiBr_2(CNXyl)_2$, are presented. Additionally, crystals grown from a cold diethyl ether solution of zero-valent $Fe(CNXyl)_5$ produced a structure containing a cocrystallization of mononuclear $Fe(CNXyl)_5$ and the previously unknown dinuclear $[Fe(CNXyl)_3]_2(\mu_2\text{-}CNXyl)_3$, namely pentakis(2,6-dimethylphenyl isocyanide)iron(0) tris(μ_2-2,6-dimethylphenyl isocyanide)bis[tris(2,6-dimethylphenyl isocyanide)iron(0)], $[Fe(C_9H_9N)_5][Fe_2(C_9H_9N)_9]$. The ($M$)C—N—C(Xyl) angles of the isocyanide ligand are nearly linear for the metals in the +2 oxidation state, for which the ligands function essentially as pure donors. The νCN stretching frequencies for these divalent metal isocyanides are at or above that of the free ligand. Relative to Fe^{II}, in the structure containing iron in the formally zero-valent oxidation state, the Fe—C bond lengths have shortened, the C≡N bond lengths have elongated, the (M)C—N—C(Xyl) angles of the terminal CNXyl ligands are more bent, and the νCN stretching frequencies have shifted to lower energies, all indicative of substantial $M(d\pi)\rightarrow\pi^*$ backbonding.

1. Introduction

Access to isolable low-valent transition-metal complexes directly from higher-valent precursors often requires employing a synthetic strategy that minimizes or avoids the formation of unstable intermediates. One successful method involves having an electron source double as a coordinating ligand. In the synthesis of $[Ta(CO)_6]^-$ (CO is carbon monoxide), a naphthalene radical anion serves this purpose (Dewey *et al.*, 1983). Six equivalents of the naphthalene radical anion reduce the Ta center in $TaCl_5$ from an oxidation state of +V to that of formally −I. During the reduction and before CO is introduced, naphthalene also coordinates and stabilizes the metal center, as confirmed by the isolation of highly air-sensitive but thermally stable $[Ta(C_{10}H_8)_3]^-$ ($C_{10}H_8$ is naphthalene) (Brennessel *et al.*, 2002*a*). In an analogous reaction in the cobalt system, for which naphthalene alone appears to be ineffective in the stabilization of low-valent cobalt, the



homoleptic carbonyl anion $[Co(CO)_4]^-$ is formed in very low yield (<20%) {$[Co(C_{10}H_8)_2]^-$ has not been isolated as a pure substance from this reaction to date in the absence of CO; see Brennessel *et al.* (2006) and Brennessel & Ellis (2012)}.



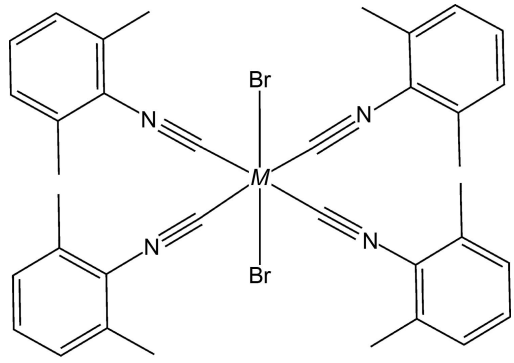
Another method to access stable low-valent species, which is an offshoot of the aforementioned method, involves starting with the metal already in the desired reduced oxidation state. $[Ta(CO)_6]^-$ can be obtained quantitatively from the reaction of carbon monoxide with $[Ta(C_{10}H_8)_3]^-$, for which $[Ta(C_{10}H_8)_3]^-$ acts as a source of Ta^{-I} monoatomic anions in the synthesis (Brennessel *et al.*, 2002*a*). The same strategy has been employed to prepare $[Co(CNXyl)_4]^-$ in high yields, *ca* 83%, by the reaction of $[Co(C_{14}H_{10})_2]^-$ ($C_{14}H_{10}$ is anthracene) with four equivalents of CNXyl (Brennessel *et al.*, 2002*b*). However, treatment of the related ferrate, *i.e.* $[Fe(C_{14}H_{10})_2]^-$ (Brennessel *et al.*, 2007), with CNXyl did not afford the desired homoleptic iron isocyanide products (Brennessel, 2009). Direct access to isoelectronic $[Fe(CNXyl)_4]^{2-}$ required a more conventional naphthalene radical anion mediated reduction of $FeBr_2(CNXyl)_4$ (Brennessel & Ellis, 2007). This article reports on the structures and spectroscopic characterizations of several CNXyl (denoted **1**) complexes of divalent iron (as the dichloromethane 0.771-solvate, **2a**, and in the unsolvated form, **2b**), cobalt (**4**), and nickel (**5**), and the unexpected structure of a cocrystal of two homoleptic CNXyl compounds of zero-valent iron, *i.e.* $Fe(CNXyl)_5$ and the previously unknown dinuclear $[Fe(CNXyl)_3]_2(\mu_2\text{-}CNXyl)_3$ (**3**) (see Schemes).

2. Experimental

2.1. Synthesis and crystallization

2.1.1. 2,6-Dimethylphenyl isocyanide (CNXyl) (1). This synthesis was modeled after published ones (Malatesta, 1947; Ugi & Meyr, 1960). Unpurified CH_2Cl_2 (300 ml) and NEt_3 (105 ml, 755 mmol) were added to a 500 ml flask containing *N*-(2,6-dimethylphenyl)formamide (25.6 g, 172 mmol) that had a dropping funnel attached. $POCl_3$ (17.6 ml, 189 mmol), previously purified by vacuum distillation, was added *via* a Teflon cannula to the dropping funnel, to which had been added CH_2Cl_2 (10 ml). The reaction flask was placed in an ice–water bath and the $POCl_3$ solution was added dropwise, which resulted in an orange–brown slurry upon completion of the addition. The reaction mixture was stirred for an additional hour, and then transferred to a 1000 ml separatory funnel. The product was washed and separated three times with a buffer solution (pH 6.27) prepared from $K_2HPO_4{\cdot}3H_2O$ (3.027 g) and KH_2PO_4 (15.54 g) in H_2O (300 ml). (The first wash produced a slight warming.) The resultant organic fraction was then washed and separated three times with a saturated brine solution prepared from NaCl in H_2O (300 ml). The product was transferred to a flask containing Na_2SO_4, stirred for 30 min, and filtered *via* gravity through a one-inch plug of Na_2SO_4. After the solvent had been removed under vacuum, the orange solid was dissolved in petroleum ether and filtered to remove the colored impurity. Removal of the solvent under vacuum left a fluffy pale-yellow solid (yield 17.6 g, 78%).

IR [tetrahydrofuran (THF), νCN, cm^{-1}]: 2117 (*vs*); IR (Nujol mull, νCN, cm^{-1}): 2114 (*vs*); m.p. 345–347 K; ^{1}H NMR (300 MHz, $CDCl_3$, 293 K, δ, ppm): 7.27 ($CDCl_3$), 7.09–7.22 (*m*, 3H, *m*- and *p*-CNXyl), 2.43 (*s*, 6H, CH_3); $^{13}C\{^1H\}$ NMR (75.4 MHz, $CDCl_3$, 293 K, δ, ppm): 167.7 (*C*NXyl), 135.1 (*o*), ~130 (*ipso*), 128.8 (*p*), 127.9 (*m*), 77.2 ($CDCl_3$), 19.1 (CH_3).



2b *M* = Fe
4 *M* = Co

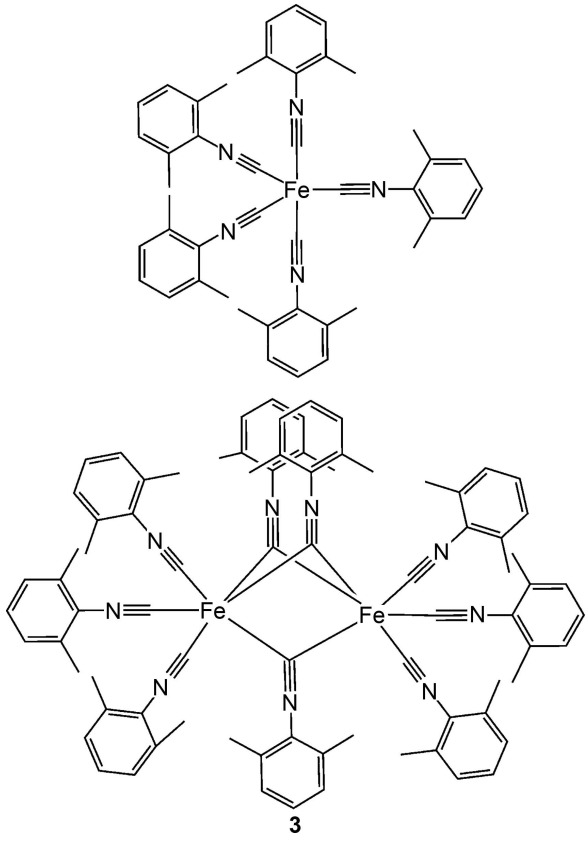
2.1.2. *cis*-$FeBr_2(CNXyl)_4{\cdot}0.771CH_2Cl_2$ (2a) and *trans*-$FeBr_2(CNXyl)_4$ (2b). CNXyl (1.217 g, 9.27 mmol) in toluene (20 ml) was added to $FeBr_2$ (0.500 g, 2.32 mmol) in toluene (100 ml). The reaction mixture was heated to 358 K for 1 h, which produced a purple solution with obvious solids. After cooling to room temperature, the mixture was filtered. The bright-orange cake of *cis* compound **2a** was dried under vacuum (yield 0.715 g, 42%). The solvent was removed under vacuum from the red–purple filtrate, which was then filtered, washed with pentane (2 × 20 ml), and dried under vacuum, yielding light-blue–purple *trans* compound **2b** (yield 0.728 g, 42%). Although no attempts to obtain bulk elemental analyses on these compounds were carried out, they were obtained as spectroscopically pure substances. Single crystals of each were obtained by evaporation from CH_2Cl_2 solutions.

cis-$FeBr_2(CNXyl)_4$ (**2a**): IR (νCN, Nujol mull, cm^{-1}): 2146 (*s*), 2127 (*s*); ^{1}H NMR (300 MHz, CD_2Cl_2, 293 K, δ, ppm): 7.11–7.19 (*m*, 12H, *m*- and *p*-CNXyl), 5.32 (CD_2Cl_2), 2.62 (*s*, 12H, CH_3), 2.48 (*s*, 12H, CH_3); $^{13}C\{^1H\}$ NMR (75.4 MHz,

CD_2Cl_2, 293 K, δ, ppm): 173.2 (*C*NXyl), 136.4, 135.6, 129.2, 128.8, 128.4, 54.0 (quintet, CD_2Cl_2), 19.3 (*C*H_3), 19.2 (*C*H_3).

trans-$FeBr_2(CNXyl)_4$ (**2b**): IR (νCN, THF, cm^{-1}): 2181 (*sh*), 2130 (*vs*); ^{1}H NMR (300 MHz, CD_2Cl_2, 293 K, δ, ppm): 7.14–7.20 (*m*, 12H, *m*- and *p*-CNXyl), 5.32 (CD_2Cl_2), 2.61 (*s*, 24H, C*H*$_3$); $^{13}C\{^1H\}$ NMR (75.4 MHz, CD_2Cl_2, 293 K, δ, ppm): 174.7 (*C*NXyl), 136.3, 128.9, 128.3, 54.0 (quintet, CD_2Cl_2), 19.2 (*C*H_3). The solution prepared for NMR analysis was stored in the NMR tube for one month, after which a ^{1}H NMR spectrum revealed that it had mostly converted to *cis* isomer **2a**.

2.1.3. $Fe(CNXyl)_5{\cdot}Fe_2(\mu_2\text{-}CNXyl)_3(CNXyl)_6$ (3). The mononuclear part of **3** was synthesized based on the literature preparation (Bassett *et al.*, 1979), and the spectroscopic data matched those reported previously. Bright-red single crystals of cocrystal **3** were grown from a saturated diethyl ether solution at 243 K.



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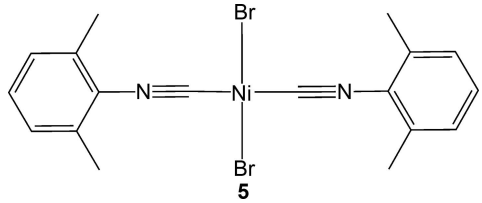
$Fe(CNXyl)_5$ (**3**, mononuclear part): IR (νCN, THF, cm^{-1}): 2033 (*sh*), 1975 (*vs br*), 1871 (*sh*); ^{1}H NMR (300 MHz, C_6D_6, 293 K, δ, ppm): 7.16 (C_6D_6), 6.78 (*s*, 15H, *m*- and *p*-CNXyl), 2.43 (*s*, 30H, C*H*$_3$); $^{13}C\{^1H\}$ NMR (75.4 MHz, C_6D_6, 293 K, δ, ppm): 197.0 (*C*NXyl), 133.7 (*o*-CNXyl), 132.5 (*ipso*-CNXyl), 128.4 (*t*, C_6D_6), 128.2 (*m*-CNXyl), 125.5 (*p*-CNXyl), 19.7 (*C*H_3).

2.1.4. *trans*-$CoBr_2(CNXyl)_4$ (4). This compound was prepared previously by a different method (Yamamoto & Yamazaki, 1977*a*). To a clear blue solution of $CoBr_2$ (0.500 g, 2.29 mmol) in THF (30 ml) was added a solution of CNXyl (1.199 g, 9.14 mmol) in THF (20 ml) at room temperature, which caused a green solid to precipitate immediately. After an additional 3 h of stirring, the slurry was filtered, and the product was dried under vacuum, yielding a bright-green powder (yield 1.633 g, 96%).

IR (νCN, CH_2Cl_2, cm^{-1}): 2182 (*vs*); IR (νCN, Nujol mull, cm^{-1}): 2180 (*sh*), 2168 (*vs*); m.p. 475 K.

2.1.5. *trans*-$NiBr_2(CNXyl)_2$ (5). This compound was prepared previously by a different method (Yamamoto *et al.*, 1991). Toluene (80 ml) was added to a flask containing $NiBr_2$ (1.030 g, 4.71 mmol) and CNXyl (1.287 g, 9.81 mmol). The suspension was heated at 353–358 K for 12 h, although most particulates had dissolved and/or reacted after 6 h. The red–brown slurry was cooled slowly to room temperature, after which half of the toluene was removed under vacuum before filtering. The filter cake was dried for a few hours at room temperature, affording a dull-orange fibrous solid (yield 1.577 g, 70%). The solvent was then removed under vacuum from the filtrate, providing another 0.357 g of product, giving a total of 1.934 g (85%). Although no attempts to obtain bulk elemental analyses on this compound were carried out, it was obtained as a spectroscopically pure substance.

IR (toluene, νCN, cm^{-1}): 2191 (*vs*); IR (Nujol mull, νCN, cm^{-1}): 2196 (*vs*); ^{1}H NMR (300 MHz, $CDCl_3$, 293 K, δ, ppm): 7.24–7.30 (*m*, 2H, *p*-CNXyl), 7.12–7.15 (*m*, 4H, *m*-CNXyl), 2.48 (*s*, 12H, C*H*$_3$); ^{13}C NMR (75.4 MHz, $CDCl_3$, 293 K, δ, ppm): 136.7 (*d*, J = 161.8 Hz, *o*-CNXyl), 135.1 (*vbr*, *ipso*-CNXyl), 130.5 (*d*, J = 161.2 Hz, *p*-CNXyl), 128.3 (*d*, J = 166.4 Hz, *m*-CNXyl), 77.2 ($CDCl_3$), 18.8 (*q*, J = 126.9 Hz, *C*H_3).



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2.2. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. In **1**, the cocrystallized dichloromethane solvent was modeled as disordered over a crystallographic twofold axis (0.50:0.50), and additionally over two general positions [0.593 (9) and 0.192 (9)]. The occupancies of the general positions were not required to sum to unity because it had been determined that there had been solvent loss during the mounting procedure. This loss was confirmed by a significant increase in $R1$ (strong data) when the occupancies were forced to sum to unity, as well as by examining the results of the SQUEEZE (Spek, 2015) routine of *PLATON* (Spek, 2009), which showed 150 electrons *versus* the expected 168. Analogous bond lengths and angles between the two positions of the dichloromethane solvent disorder were restrained to be similar. Anisotropic displacement parameters for proximal (including symmetry-equivalent) atoms were constrained to be equivalent. Additionally, anisotropic displacement parameters for all atoms of the major component were restrained to be similar.

Methyl H atoms on C17 in **2a**, C8, C9, and C17 in **2b**, and C9 in **4** were modeled as rotationally disordered.

Table 1
Experimental details.

	1	**2a**	**2b**
Crystal data			
Chemical formula	C_9H_9N	$[FeBr_2(C_9H_9N)_4]\cdot0.771CH_2Cl_2$	$[FeBr_2(C_9H_9N)_4]$
M_r	131.17	805.87	740.36
Crystal system, space group	Monoclinic, *I*2/*a*	Monoclinic, *C*2/*c*	Orthorhombic, *Pbca*
Temperature (K)	100	173	173
a, *b*, *c* (Å)	14.5535 (6), 7.5199 (2), 14.6333 (7)	16.8177 (13), 15.8912 (12), 14.1334 (11)	14.4246 (13), 14.0229 (13), 16.3444 (15)
α, β, γ (°)	90, 113.548 (5), 90	90, 92.9182 (10), 90	90, 90, 90
V (Å^3)	1468.12 (11)	3772.3 (5)	3306.1 (5)
Z	8	4	4
Radiation type	Cu *K*α	Mo *K*α	Mo *K*α
μ (mm^{-1})	0.54	2.66	2.90
Crystal size (mm)	0.15 × 0.12 × 0.06	0.44 × 0.16 × 0.08	0.25 × 0.20 × 0.15
Data collection			
Diffractometer	XtaLAB Synergy, Dualflex, HyPix	Bruker SMART CCD platform	Siemens SMART CCD platform
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2018)	Multi-scan (*SADABS*; Sheldrick, 2014)	Multi-scan (*SADABS*; Sheldrick, 2014)
T_{min}, T_{max}	0.927, 0.968	0.662, 0.746	0.592, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6065, 1521, 1375	15633, 4323, 3646	25764, 3790, 3151
R_{int}	0.032	0.020	0.023
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.634	0.650	0.650
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.040, 0.117, 1.11	0.025, 0.066, 1.03	0.025, 0.065, 1.03
No. of reflections	1521	4323	3790
No. of parameters	93	230	198
No. of restraints	0	20	0
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.20, −0.21	0.48, −0.31	0.32, −0.52

	3	**4**	**5**
Crystal data			
Chemical formula	$[Fe(C_9H_9N)_5][Fe_2(C_9H_9N)_9]$	$[Co(C_9H_9N)_4]$	$[NiBr_2(C_9H_9N)_2]$
M_r	2003.95	743.44	480.87
Crystal system, space group	Monoclinic, $P2_1/n$	Orthorhombic, *Pbca*	Monoclinic, $P2_1/n$
Temperature (K)	173	173	173
a, *b*, *c* (Å)	24.901 (3), 14.2838 (19), 30.954 (4)	14.5902 (14), 14.0206 (13), 16.2747 (15)	20.433 (7), 4.5010 (15), 21.285 (7)
α, β, γ (°)	90, 96.654 (2), 90	90, 90, 90	90, 105.682 (5), 90
V (Å^3)	10935 (3)	3329.2 (5)	1884.7 (11)
Z	4	4	4
Radiation type	Mo *K*α	Mo *K*α	Mo *K*α
μ (mm^{-1})	0.45	2.95	5.27
Crystal size (mm)	0.36 × 0.28 × 0.08	0.25 × 0.15 × 0.10	0.50 × 0.10 × 0.05
Data collection			
Diffractometer	Bruker SMART CCD platform	Siemens SMART CCD platform	Siemens SMART CCD platform
Absorption correction	Multi-scan (*SADABS*; Sheldrick, 2014)	Multi-scan (*SADABS*; Sheldrick, 2014)	Multi-scan (*SADABS*; Sheldrick, 2014)
T_{min}, T_{max}	0.662, 0.746	0.676, 0.746	0.592, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	75619, 19257, 10377	36969, 3815, 3240	9617, 3287, 2241
R_{int}	0.086	0.037	0.074
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.595	0.649	0.596
Refinement			
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.063, 0.153, 1.03	0.027, 0.057, 1.08	0.061, 0.109, 1.14
No. of reflections	19257	3815	3287
No. of parameters	1316	200	215
No. of restraints	0	0	0
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.44, −0.47	0.28, −0.45	0.94, −0.62

Computer programs: *CrysAlis PRO* (Rigaku OD, 2018), *SMART* (Bruker, 2003), *SAINT* (Bruker, 2003), *SHELXT* (Sheldrick, 2015*a*), *SHELXS97* (Sheldrick, 2008), *SHELXL2018* (Sheldrick, 2015*b*), *OLEX2* (Dolomanov *et al.*, 2009) and *SHELXTL* (Sheldrick, 2008).

Table 2
Selected geometric parameters (Å, °) for **1**.

N1—C1	1.1608 (15)
C1—N1—C2	179.47 (11)

H atoms were placed geometrically and treated as riding atoms, with methylene C—H = 0.99 Å and Csp^2—H = 0.95 Å, with U_{iso}(H) = 1.2U_{eq}(C), and methyl C—H = 0.98 Å, with U_{iso}(H) = 1.5U_{eq}(C).

For **1**, the maximum residual peak of 0.20 e$^-$ Å^{-3} and the deepest hole of −0.21 e$^-$ Å^{-3} are found 0.74 and 1.35 Å from atoms C9 and C6, respectively.

For **2a**, the maximum residual peak of 0.48 e$^-$ Å^{-3} and the deepest hole of −0.31 e$^-$ Å^{-3} are found 1.09 and 0.42 Å from atoms Cl1′ and Cl2′, respectively.

For **2b**, the maximum residual peak of 0.32 e$^-$ Å^{-3} and the deepest hole of −0.52 e$^-$ Å^{-3} are found 0.25 and 0.49 Å from atoms H9*F* and Br1, respectively.

For **3**, the maximum residual peak of 0.44 e$^-$ Å^{-3} and the deepest hole of −0.47 e$^-$ Å^{-3} are found 1.11 and 0.88 Å from atoms H60*A* and Fe3, respectively.

For **4**, the maximum residual peak of 0.28 e$^-$ Å^{-3} and the deepest hole of −0.44 e$^-$ Å^{-3} are found 0.72 and 0.46 Å from atoms C3 and Br1, respectively.

For **5**, the maximum residual peak of 0.94 e$^-$ Å^{-3} and the deepest hole of −0.62 e$^-$ Å^{-3} are found 1.14 and 1.17 Å from atoms Ni1 and Br1, respectively.

3. Results and discussion

The isocyanide molecule used for ligating metals in this study, *i.e.* CNXyl, **1**, crystallized in the space group *I*2/*a* with one molecule in the asymmetric unit (Fig. 1). The structure was intially reported in its primitive setting of one-half the volume with two molecules in the asymmetric unit (Mathieson *et al.*, 2001). Despite the potential for parameter correlations between symmetry-related atoms, the reported bond lengths closely match those of **1**. A second structure containing CNXyl was recently published in which CNXyl was cocrystallized with an iminodisilirane of interest (Tashkandi *et al.*, 2019). In **1**, the terminal C—N bond length and linear C—N—C angle (Table 2), as well as the solution IR νCN stretch of 2117 cm^{-1} and the ^{13}C NMR resonance for the isocyanide C atom of δ 167.7 ppm, are consistent with a C≡N triple bond. The molecules of **1** are aligned along [001], with pairs of offset parallel-stacked molecules (centroid–centroid distance = 3.64 Å) potentially linked by overlapped π systems in the C≡N moieties (3.47 Å) and C—H···centroid (H···centroid = 2.73 Å), donor→electron-deficient-ring-center interactions (Fig. 2).

The addition of four equivalents of CNXyl to anhydrous $FeBr_2$ in toluene resulted in a purple solution with solids. The solids were filtered off, dried, and washed, which resulted in an orange powder. The solvent was removed from the red–purple filtrate, to leave a light-blue–purple powder. Separately, both

Table 3
Selected geometric parameters (Å, °) for **2a**.

Fe1—C10	1.8325 (17)	C1—N1	1.153 (2)
Fe1—C1	1.8791 (17)	C10—N2	1.161 (2)
Fe1—Br1	2.4785 (3)		
C10^i—Fe1—C10	96.49 (10)	C1—Fe1—Br1	89.17 (5)
C10^i—Fe1—C1	88.99 (7)	C1^i—Fe1—Br1	88.77 (5)
C10—Fe1—C1	92.97 (7)	Br1—Fe1—Br1^i	91.520 (15)
C1—Fe1—C1^i	177.05 (10)	C1—N1—C2	177.85 (18)
C10^i—Fe1—Br1	176.96 (5)	C10—N2—C11	163.00 (17)
C10—Fe1—Br1	86.02 (5)		

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

powders were dissolved in dichloromethane and the solutions were left open to the air to evaporate. Single crystals grown from the orange solution were identified by X-ray diffraction as *cis*-$FeBr_2(CNXyl)_4\cdot 0.771CH_2Cl_2$, **2a**, and those grown from the purple solution were shown to be *trans*-$FeBr_2(CNXyl)_4$, **2b**.

The asymmetric unit of **2a** contains one-half of an iron complex (Fig. 3 and Table 3), located along a crystallographic twofold axis that includes the Fe atom, plus cocrystallized dichloromethane solvent, also located along a twofold axis. The solvent was modeled as disordered over the axis (0.50:0.50) and additionally over two general positions [0.579 (9):0.192 (9)]. The ratio does not sum to unity because it was found that the solvent, which packed in channels along [001] (Fig. 4), had partially escaped from the crystals during the mounting procedure. The structure of **2a** is isomorphous with its chlorido analog (Perry *et al.*, 2010), whose solvent channels were occupied by *n*-hexane, also of partial occupancy.

The *trans* isomer of $FeBr_2(CNXyl)_4$, **2b** (Fig. 5 and Table 4), crystallized as large purple blocks. The molecule is located on a crystallographic inversion center that coincides with the Fe atom. A three-dimensional network is generated by pairs of offset parallel stacked arene rings (centroid–centroid distance = 3.61 Å). The previously reported chlorido analog

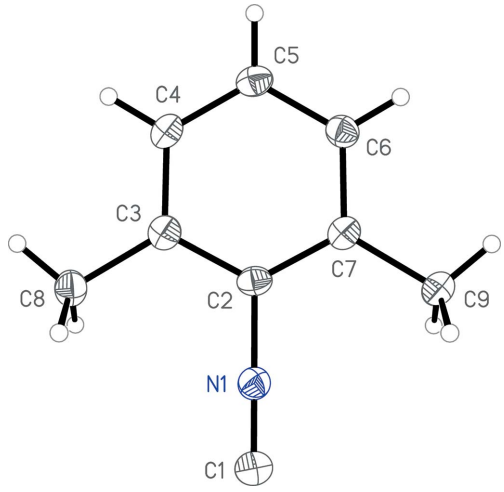

Figure 1
Anisotropic displacement ellipsoid plot of **1** drawn at the 50% probability level.

Table 4
Selected geometric parameters (Å, °) for **2b**.

Fe1—C10	1.8748 (16)	C1—N1	1.156 (2)
Fe1—C1	1.8842 (16)	C10—N2	1.156 (2)
Fe1—Br1	2.4665 (3)		
C10—Fe1—C10^{i}	180.0	C1^{i}—Fe1—Br1	86.81 (5)
C10—Fe1—C1	91.43 (7)	C1—Fe1—Br1	93.19 (5)
C1^{i}—Fe1—C1	180.0	Br1—Fe1—Br1^{i}	180.0
C10—Fe1—Br1	91.52 (5)	C1—N1—C2	167.90 (17)
C10^{i}—Fe1—Br1	88.49 (5)	C10—N2—C11	171.99 (17)

Symmetry code: (i) $-x, -y+1, -z+1$.

(Drew *et al.*, 1986) is not isomorphous or even isostructural, but has similar unit-cell constants of 13.853 (8), 13.853 (8), and 8.803 (8) Å and 90, 90, and 90° (*cf.* Table 1).

The IR stretches for **2a** and **2b** of νCN = 2137 (average of two bands) and 2130 cm^{-1}, respectively, compared to that of the free ligand, 2117 cm^{-1}, indicate a strengthening of the C≡N bonds through coordination to the metal. Indeed, it has been demonstrated that the lone pair on the isocyanide C atom of the free ligand is antibonding with respect to the C≡N bond, and that through coordination to a metal center, more bonding character is achieved, thus strengthening this bond (Crabtree, 1994). Although the average Fe—C bond lengths vary slightly between **2a** and **2b**, the average C≡N bond lengths and C—N—C angles are the same (Tables 3 and 4). However, because of the *cis* geometry of **2a**, two CNXyl ligands (one unique due to symmetry) are *trans* to an isocyanide ligand and the other two CNXyl ligands (again, one unique) are *trans* to the bromido ligands. The isocyanide ligands *trans* to the other isocyanides have the same Fe—C and C≡N bond lengths and C—N—C angles as those found in **2b** (Table 4), but those *trans* to a bromido ligand show evidence of backbonding (Table 3), specifically shorter Fe—C

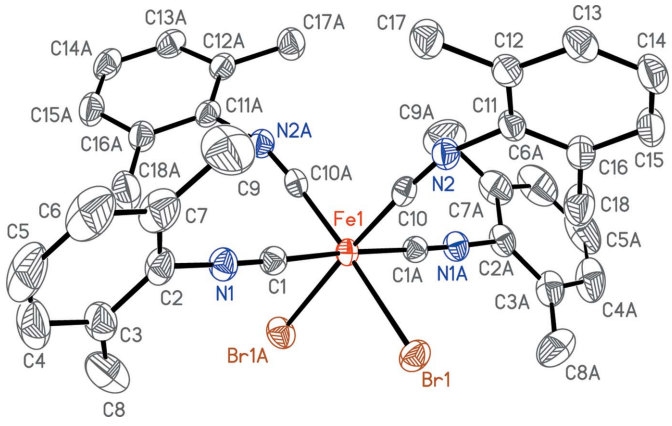

Figure 3
Anisotropic displacement ellipsoid plot of **2a**, drawn at the 50% probability level and with solvent molecules and H atoms omitted. The symmetry equivalent portion is generated by a twofold axis at $(-x+1, y, -z+\frac{1}{2})$ (symmetry code A).

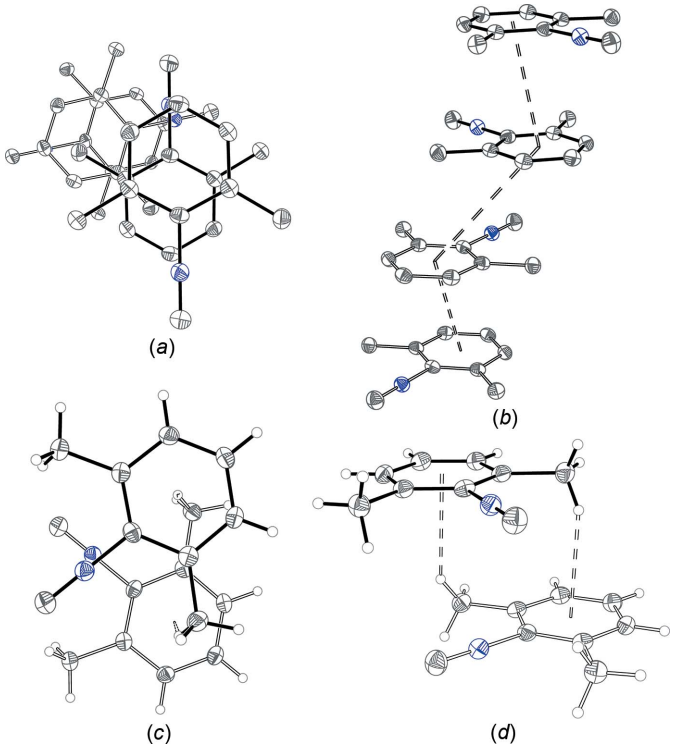

Figure 2
Anisotropic displacement ellipsoid plots of **1**, drawn at the 50% probability level. Symmetry equivalent molecules were generated by the operations $(-x+\frac{1}{2}, -y+\frac{3}{2}, -z+\frac{1}{2})$, $(-x+\frac{1}{2}, y, -z+1)$, and $(x, -y+\frac{3}{2}, z+\frac{1}{2})$. (*a*) View down [001] which shows the parallel stacking. Pairs of molecules of the same bond type (*i.e.* solid or hollow) are offset parallel stacked at centroid–centroid distances of 3.64 Å. The centroid–centroid distance between non-overlapping rings (*i.e.* different bond types) is 4.65 Å. (*b*) Side view that depicts the centroid–centroid offsets. (*c*) The pairs of molecules whose rings do not overlap have overlaps of their C≡N groups at a distance of 3.47 Å and a C≡N···N≡C pseudo-torsion angle of 78.5 (2)°. (*d*) In addition to the possible π–π interaction of the C≡N segments, there may be C—H···centroid interactions (H··· centroid distance = 2.73 Å).

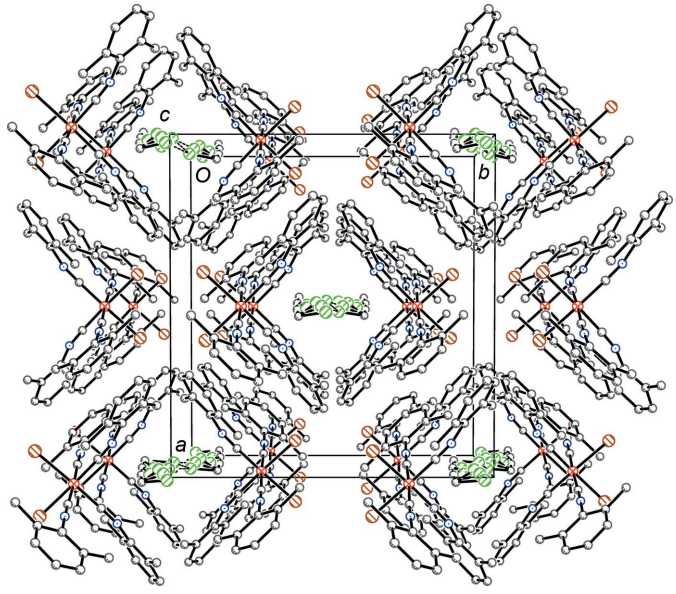

Figure 4
Packing plot of **2a**, with H atoms omitted. Disordered solvent of partial occupancy is located in channels along [001].

Table 5
Selected geometric parameters (Å, °) for **3**.

Fe1—C28	1.805 (4)	Fe2—Fe3	2.4764 (8)
Fe1—C1	1.816 (4)	Fe3—C100	1.838 (5)
Fe1—C19	1.819 (4)	Fe3—C109	1.856 (4)
Fe1—C37	1.821 (5)	Fe3—C118	1.860 (4)
Fe1—C10	1.829 (4)	Fe3—C73	1.962 (4)
N1—C1	1.196 (5)	Fe3—C82	1.978 (4)
N2—C10	1.183 (5)	Fe3—C91	2.022 (4)
N3—C19	1.177 (5)	N6—C46	1.180 (5)
N4—C28	1.192 (5)	N7—C55	1.178 (5)
N5—C37	1.178 (5)	N8—C64	1.178 (5)
Fe2—C64	1.832 (5)	N9—C73	1.245 (4)
Fe2—C55	1.840 (4)	N10—C82	1.237 (4)
Fe2—C46	1.852 (4)	N11—C91	1.235 (4)
Fe2—C91	1.967 (4)	N12—C100	1.190 (5)
Fe2—C73	1.974 (4)	N13—C109	1.176 (4)
Fe2—C82	1.974 (4)	N14—C118	1.179 (4)
C28—Fe1—C1	128.78 (18)	C46—Fe2—C82	96.16 (16)
C28—Fe1—C19	88.02 (17)	C91—Fe2—C82	84.02 (15)
C1—Fe1—C19	90.78 (17)	C73—Fe2—C82	87.43 (16)
C28—Fe1—C37	85.38 (17)	C100—Fe3—C109	93.56 (16)
C1—Fe1—C37	92.23 (17)	C100—Fe3—C118	93.66 (16)
C19—Fe1—C37	173.24 (17)	C109—Fe3—C118	90.28 (16)
C28—Fe1—C10	119.54 (18)	C100—Fe3—C73	94.61 (16)
C1—Fe1—C10	111.66 (17)	C109—Fe3—C73	171.46 (16)
C19—Fe1—C10	90.29 (17)	C118—Fe3—C73	91.68 (15)
C37—Fe1—C10	94.22 (17)	C100—Fe3—C82	84.51 (16)
C1—N1—C2	162.9 (4)	C109—Fe3—C82	90.63 (16)
C10—N2—C11	166.4 (4)	C118—Fe3—C82	178.00 (16)
C19—N3—C20	178.7 (4)	C73—Fe3—C82	87.67 (16)
C28—N4—C29	158.6 (4)	C100—Fe3—C91	166.97 (16)
C37—N5—C38	164.6 (4)	C109—Fe3—C91	87.99 (15)
C64—Fe2—C55	93.53 (17)	C118—Fe3—C91	99.28 (16)
C64—Fe2—C46	93.81 (17)	C73—Fe3—C91	83.49 (15)
C55—Fe2—C46	95.13 (17)	C82—Fe3—C91	82.53 (16)
C64—Fe2—C91	85.33 (16)	C46—N6—C47	164.1 (4)
C55—Fe2—C91	93.71 (17)	C55—N7—C56	165.8 (4)
C46—Fe2—C91	171.16 (17)	C64—N8—C65	165.4 (4)
C64—Fe2—C73	88.00 (16)	C73—N9—C74	132.9 (3)
C55—Fe2—C73	177.63 (17)	C82—N10—C83	133.0 (3)
C46—Fe2—C73	86.57 (16)	C91—N11—C92	133.7 (3)
C91—Fe2—C73	84.61 (16)	C100—N12—C101	165.2 (4)
C64—Fe2—C82	168.76 (17)	C109—N13—C110	168.6 (4)
C55—Fe2—C82	90.74 (16)	C118—N14—C119	162.7 (4)

but longer C≡N bond lengths, and more bent (*i.e.* nonlinear) C—N—C angles. This exact phenomenon was noted for the chlorido analog (Perry *et al.*, 2010). Interestingly, a ^{1}H NMR spectrum taken approximately one month later on the exact same solution of **2b** in CD_2Cl_2 showed that it had converted almost entirely to **2a**.

Although **2a** and **2b** can be synthesized simultaneously and subsequently separated by solubility differences, when employed as precursors to low-valent species, it is unnecessary to worry about isomer segregation. A mixture of **2a** and **2b** formed *in situ* in THF was used successfully to generate $K_2[Fe(CNXyl)_4]$ by reduction with four equivalents of $K^+C_{10}H_8^-$ (Brennessel & Ellis, 2007).

While a few structures of homoleptic isocyanide complexes of zero-valent iron have been reported, namely $Fe(CN^tBu)_5$, $[Fe(CNEt)_3]_2(\mu_2\text{-CNEt})_3$ (Bassett *et al.*, 1979), and $[Fe(CNPh)_3]_2(\mu_2\text{-CNPh})_3$ (Ruiz *et al.*, 1992), we wanted to evaluate a structure of $Fe(CNXyl)_5$ for direct comparison with divalent iron complexes **2a** and **2b** and $[Fe(CNXyl)_4]^{2-}$. Therefore, we synthesized $Fe(CNXyl)_5$ in a manner similar to

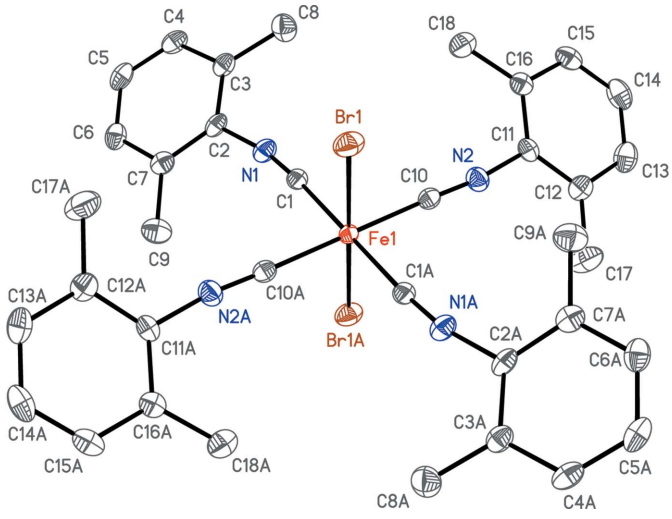

Figure 5
Anisotropic displacement ellipsoid plot of **2b**, drawn at the 50% probability level and with H atoms omitted. The symmetry-equivalent portion is generated by the inversion operation $(-x, -y + 1, -z + 1)$ (symmetry code A). Structures **2b** and **4** are isomorphous (see supporting information for an ellipsoid plot of **4**).

that of the literature procedure (Bassett *et al.*, 1979), by reduction of a mixture of $FeBr_2$ and five equivalents of CNXyl in THF with sodium amalgam {which is too weak of a reducing agent in THF to generate $[Fe(CNXyl)_4]^{2-}$}. As the goal of the synthesis was to obtain a crystal structure, after confirming the

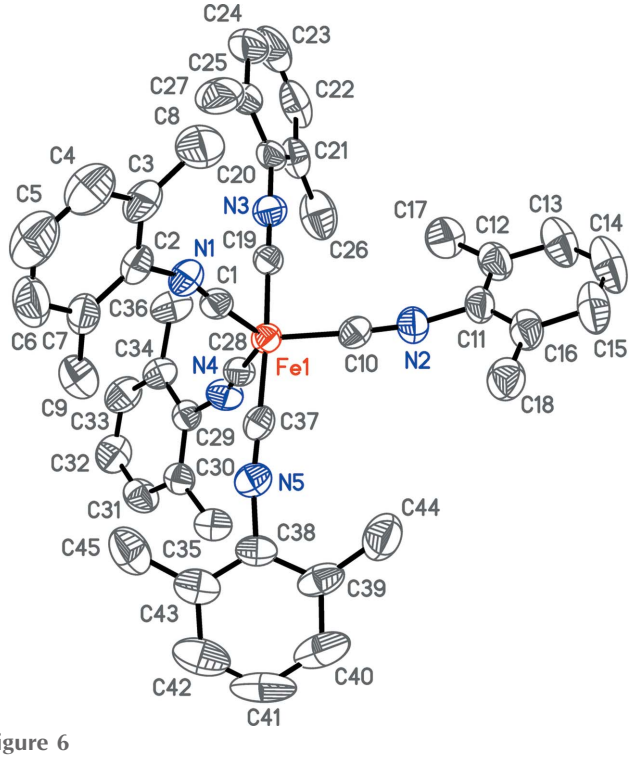

Figure 6
Anisotropic displacement ellipsoid plot of the monomeric part of **3**, drawn at the 50% probability level and with H atoms omitted. The orientation was chosen to feature the axial ligands of the trigonal–bipyramidal complex vertically (ligands containing atoms N3 and N5).

Table 6
Selected average structural and spectroscopic data.

	M—C (Å)	C≡N (Å)	C—N—C (°)	νCN (cm^{-1})[a]	δ(M—C) (ppm)
1	–	1.1608 (15)	179.47 (11)	2117 (*vs*)[b], 2114 (*vs*)[c]	167.7[d]
2a	1.856 (2)[e]	1.157 (3)[e]	170.4 (3)[e]	2146 (*s*), 2147 (*s*)[c]	173.2[f]
2b	1.880 (2)	1.156 (3)	169.9 (3)	2181 (*sh*), 2130 (*vs*)[b]	174.7[f]
3, L5[g]	1.818 (4)	1.185 (4)	166 (3)[h]	2033 (*sh*), 1975 (*vs br*), 1871 (*sh*)[b]	197.0[i]
3, L9t[g]	1.846 (5)	1.180 (2)	165.3 (8)	–	–
3, L9b[g]	1.980 (9)	1.239 (3)	133.2 (3)	–	–
$[Fe(CNXyl)_4]^{2-}$,[k]	1.765 (6)	1.237 (8)	143.8 (6)	1670 (*vs, br*)[b]	238.7[l]
4	1.867 (3)	1.151 (3)	171.7 (3)	2182 (*vs*)[j], 2180 (*sh*), 2168 (*vs*)[c]	–[m]
5	1.84 (1)	1.15 (1)	179 (1)	2191 (*vs*)[n], 2196 (*vs*)[c]	–[m]

Notes: (*a*) *vs* = very strong, *s* = strong, *sh* = shoulder, and *br* = broad; (*b*) tetrahydrofuran (THF, C_4H_8O); (*c*) Nujol (heavy mineral oil) mull; (*d*) deuterated chloroform ($CDCl_3$); (*e*) the two individual values in the asymmetric unit differ significantly, depending on which ligand is *trans* (see Table 3 for individual bond lengths and angles); (*f*) deuterated dichloromethane (CD_2Cl_2); (*g*) L5 = mononuclear $Fe(CNXyl)_5$, L9t = dinuclear $Fe_2(CNXyl)_9$, with terminal isocyanide ligands, and L9b = dinuclear $Fe_2(CNXyl)_9$, with bridging isocyanide ligands; (*h*) one axial ligand (N3) angle differs greatly from the others [178.8 (4)°] (see Table 5 for individual angles); (*i*) deuterated benzene (C_6D_6); (*j*) dichloromethane (CH_2Cl_2); (*k*) Brennessel & Ellis (2007); (*l*) deuterated THF (C_4D_8O); (*m*) not resolved; (*n*) toluene ($C_6H_5CH_3$).

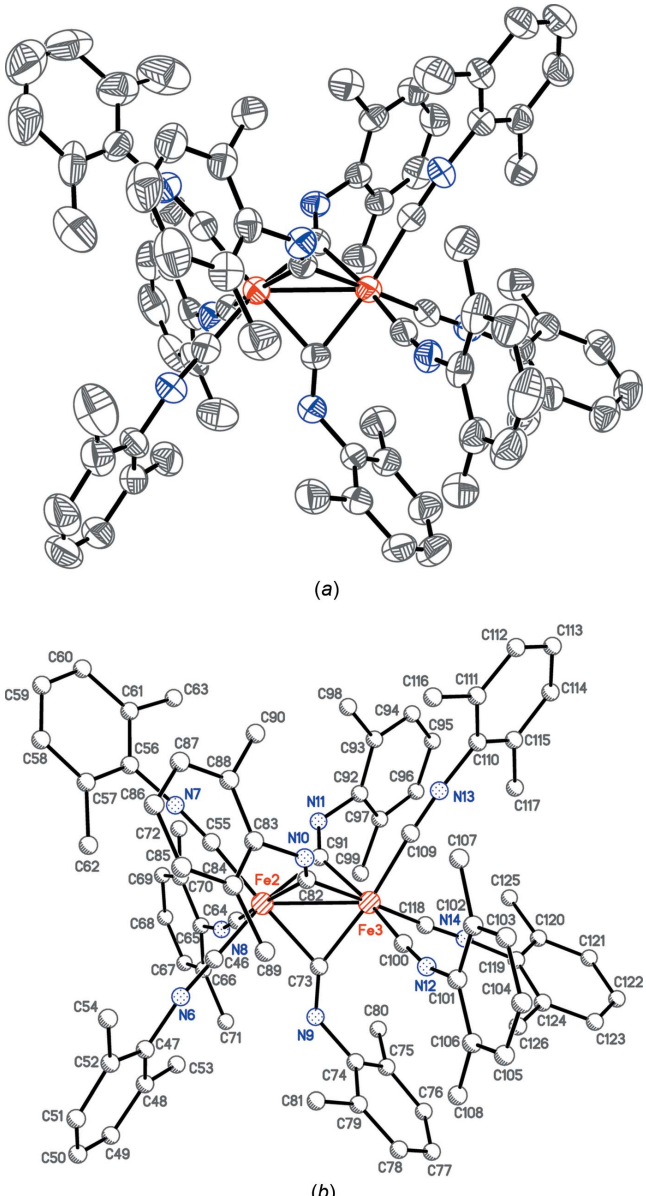

Figure 7
Plot of the dinuclear part of **3**, with H atoms omitted, showing (*a*) an anisotropic displacement ellipsoid plot drawn at the 50% probability level and (*b*) drawn in the same orientation as small spheres for labeling clarity.

product by IR and NMR spectroscopies, the isolated solid was dissolved in a minimal amount of diethyl ether and left in a 243 K freezer for approximately one month. The single crystals analyzed proved to be $[Fe(CNXyl)_5][Fe(CNXyl)_3]_2(\mu_2\text{-}CNXyl)_3$, **3** (Figs. 6 and 7), a structure containing a 1:1 cocrystallization of both forms (see examples above) of zerovalent iron. Surprisingly, despite the large number of arene rings in the asymmetric unit (14), only one obvious π–π interaction occurs, an offset parallel stacking between rings C11–C18 and C47–C54 (centroid–centroid distance = 3.77 Å).

As expected for the lower oxidation state of iron in **3** compared to isomers **2**, the average Fe—C bond length to the terminal isocyanide ligands is shorter, the C≡N bond length has increased, and the C—N—C angle has decreased (Tables 5 and 6). Spectroscopically, the IR νCN stretches have shifted to lower energy and the ^{13}C NMR resonance for the isocyanide C atom has shifted downfield. For direct comparison with **2** and **3**, $[Fe(CNXyl)_4]^{2-}$ has also been included in Table 6.

Structure **4** is the isomorphous cobalt analog of **2b** (Fig. S1 in the supporting information and Table 7). Although cobalt has a slightly smaller radius and is slightly less electropositive than iron, for the three metrics that we have been considering, the M—C and C≡N bond lengths are the same, and the C—N—C angles are very similar (Table 6). The IR νCN stretches between the two are different, but that of **4**, *i.e.* 2170 cm^{-1}, matches that of the iodido analog, $CoI_2(CNXyl)_4$ (Leach *et al.*, 1994). Just as the isomers of **2** were used to access $[Fe(CNXyl)_4]^{2-}$ (Brennessel & Ellis, 2007) and $CoI_2(CNXyl)_4$ could be used to generate $[Co(CNXyl)_4]^-$ (Leach *et al.*, 1994), **4** was also successfully reduced by naphthalene radical anion to afford $[Co(CNXyl)_4]^-$ in good yield (Brennessel, 2009).

Moving further to the right of the periodic table, we examined the reaction of CNXyl with nickel. Not unsurprising, even in the presence of excess CNXyl, the species formed was the four-coordinate square-planar Ni^{II} complex *trans*-$NiBr_2(CNXyl)_2$, **5** (Fig. 8 and Table 8). The asymmetric unit contains one-half each of two independent molecules located at crystallographic inversion centers that coincide with the metals. The angle between the planes of the two molecules is 75.8 (2)°. Each independent molecule stacks with symmetry equivalents of itself along [010] (Fig. 9), such that the distance

Table 7
Selected geometric parameters (Å, °) for **4**.

Co1—C10	1.8718 (17)	C1—N1	1.152 (2)
Co1—C1	1.8800 (17)	C10—N2	1.149 (2)
Co1—Br1	2.6846 (3)		
C10^{i}—Co1—C10	180.0	C10—Co1—Br1	93.72 (5)
C10—Co1—C1	91.03 (7)	C1—Co1—Br1	95.15 (5)
C1^{i}—Co1—C1	180.0	Br1^{i}—Co1—Br1	180.0
C10—Co1—Br1^{i}	86.28 (5)	C1—N1—C2	169.55 (17)
C1—Co1—Br1^{i}	84.85 (5)	C10—N2—C11	173.77 (18)

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

Table 8
Selected geometric parameters (Å, °) for **5**.

Ni1—C1	1.844 (8)	Ni2—C10	1.845 (8)
Ni1—Br1	2.3079 (9)	Ni2—Br2	2.3020 (9)
C1—N1	1.146 (8)	C10—N2	1.161 (9)
C1—Ni1—C1^{i}	180.0	C10—Ni2—C10^{ii}	180.0
C1—Ni1—Br1	90.3 (2)	C10—Ni2—Br2	90.1 (2)
Br1—Ni1—Br1^{i}	180.0	Br2—Ni2—Br2^{ii}	180.0
C1—N1—C2	178.0 (7)	C10—N2—C11	179.4 (8)

Symmetry codes: (i) $-x+2, -y, -z+2$; (ii) $-x+2, -y+2, -z+1$.

between neighboring symmetry-equivalent atoms is the *b*-axis unit-cell dimension [4.5010 (15) Å]. Selected bond lengths and angles for **5** are found in Table 8.

A few brief attempts were made to synthesize the known zero-valent nickel complex Ni(CNXyl)$_4$ from **5**, by reducing it in the presence of excess CNXyl in THF. Unfortunately, although the correct IR νCN stretches of 2033 and 1997 cm^{-1} (Hahn *et al.*, 2004) were seen in the spectrum, the amount of product was negligible, perhaps due to polymerization of the isocyanide ligand. And unlike in the iron and cobalt systems, for which percursors in the low oxidation states of Fe^{-II} and Co^{-I} are not commercially available, Ni^{0} is readily available as Ni(CO)$_4$ and Ni(cod)$_2$ (cod = cycloocta-1,5-diene). In fact, the first synthesis of Ni(CNXyl)$_4$ was obtained from the former (Yamamoto & Yamazaki, 1977*b*) and the synthesis from which the crystal structure was obtained used the latter (Hahn *et al.*, 2004). No attempts were made, however, to strategically improve the synthesis of Ni(CNXyl)$_4$ from **5**.

In conclusion, the crystal structures of several divalent dibromido metal complexes ligated by isocyanide CNXyl were presented. In the iron system, the reaction of FeBr$_2$ with CNXyl resulted in equal amounts of the *cis* and *trans* isomers, while the reaction of CoBr$_2$ and CNXyl resulted solely in the *trans* form. When NiBr$_2$ was reacted with CNXyl, the product was four-coordinate square planar and *trans*, with only two CNXyl ligands present. Crystallization of the homoleptic zero-valent iron complex Fe(CNXyl)$_5$ from a cold diethyl ether solution led to a structure with two forms cocrystallized as [Fe(CNXyl)$_5$][Fe(CNXyl)$_3$]$_2$(μ_2-CNXyl)$_3$. Comparison of the bond lengths and angles and the spectroscopic data between the divalent and zero-valent species confirmed the expected back-bonding of the isocyanide ligands. The divalent iron and cobalt complexes were shown to be appropriate precursors to low-valent Fe^{-II} and Co^{-I} isocyanide complexes in good yield, presumably due to their having the ligand set coordinated in both the starting and product materials, which allowed for stabilization of intermediate oxidation states during the reductive process. In contrast, the divalent nickel species only contained two CNXyl ligands, requiring two more to coordinate to afford the zero-valent Ni(CNXyl)$_4$ complex. In this last case, the yield was extremely poor. Further studies with different isocyanide ligands and different metals may be beneficial for accessing lower formal oxidation states of

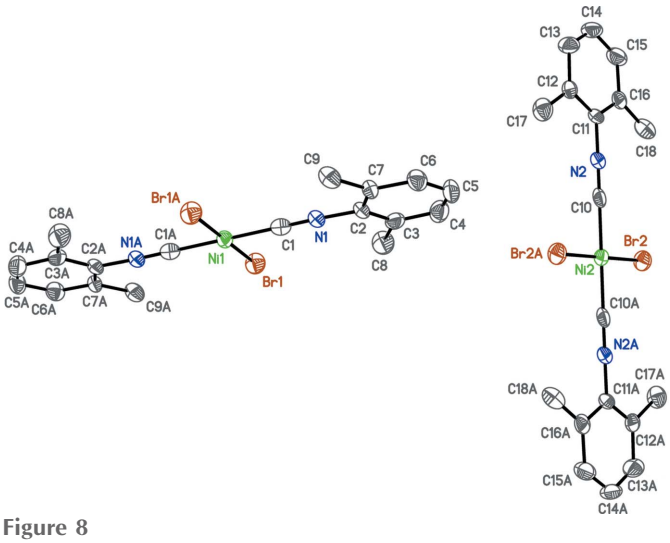

Figure 8
Anisotropic displacement ellipsoid plot of the two independent molecules of **5**, drawn at the 50% probability level and with H atoms omitted. Both independent molecules in the asymmetric unit lie on crystallographic inversion centers that coincide with the Ni atoms. The symmetry-equivalent parts of the molecules are generated by the operators ($-x+2$, $-y$, $-z+2$) (Ni1) and ($-x+2$, $-y+2$, $-z+1$) (Ni2) (symmetry code A).

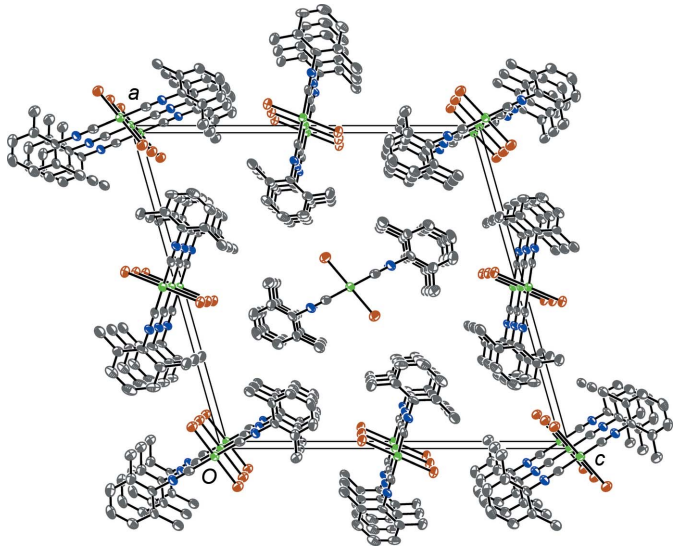

Figure 9
Anisotropic displacement ellipsoid packing plot of **5**, drawn at the 50% probability level and with H atoms omitted. Each independent nickel molecule is parallel stacked along [010] with its symmetry equivalents at distances of 4.5010 (15) Å, *i.e.* the *b*-axis unit-cell dimension. The angle between the Ni1 and Ni2 molecular planes is 75.8 (2)°.

metals, with the added benefit that the isocyanide ligand is also a great spectroscopic probe.

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Crystal structures and spectroscopic characterization of MBr$_2$(CNXyl)$_n$ (M = Fe and Co, n = 4; M = Ni, n = 2; Xyl = 2,6-dimethylphenyl), and of formally zero-valent iron as a cocrystal of Fe(CNXyl)$_5$ and Fe$_2$(CNXyl)$_9$

William W. Brennessel, Benjamin E. Kucera, Victor G. Young and John E. Ellis

Computing details

Data collection: *CrysAlis PRO* (Rigaku OD, 2018) for (1); *SMART* (Bruker, 2003) for (2a), (2b), (3), (4), (5). Cell refinement: *CrysAlis PRO* (Rigaku OD, 2018) for (1); *SAINT* (Bruker, 2003) for (2a), (2b), (3), (4), (5). Data reduction: *CrysAlis PRO* (Rigaku OD, 2018) for (1); *SAINT* (Bruker, 2003) for (2a), (2b), (3), (4), (5). Program(s) used to solve structure: SHELXT (Sheldrick, 2015a) for (1); *SHELXS97* (Sheldrick, 2008) for (2a), (2b), (3), (4), (5). For all structures, program(s) used to refine structure: *SHELXL2018* (Sheldrick, 2015b). Molecular graphics: OLEX2 (Dolomanov *et al.*, 2009) for (1); *SHELXTL* (Sheldrick, 2008) for (2a), (2b), (3), (4), (5). Software used to prepare material for publication: OLEX2 (Dolomanov *et al.*, 2009) for (1); *SHELXTL* (Sheldrick, 2008) for (2a), (2b), (3), (4), (5).

2,6-Dimethylphenyl isocyanide (1)

Crystal data

C_9H_9N
M_r = 131.17
Monoclinic, $I2/a$
a = 14.5535 (6) Å
b = 7.5199 (2) Å
c = 14.6333 (7) Å
β = 113.548 (5)°
V = 1468.12 (11) Å^3
Z = 8
$F(000)$ = 560
D_x = 1.187 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 3228 reflections
θ = 6.6–77.2°
μ = 0.54 mm^{-1}
T = 100 K
Plate, colourless
0.15 × 0.12 × 0.06 mm

Data collection

XtaLAB Synergy, Dualflex, HyPix diffractometer
Radiation source: micro-focus sealed X-ray tube, PhotonJet (Cu) X-ray Source
Mirror monochromator
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku OD, 2018)
T_{min} = 0.927, T_{max} = 0.968
6065 measured reflections
1521 independent reflections
1375 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 77.7°, θ_{min} = 6.6°
h = −17→18
k = −7→9
l = −18→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.117$
$S = 1.11$
1521 reflections
93 parameters
0 restraints
Primary atom site location: dual
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0648P)^2 + 0.6454P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

Special details

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.19375 (7)	0.52937 (12)	0.37062 (7)	0.0216 (3)
C1	0.14211 (9)	0.40998 (16)	0.36776 (9)	0.0287 (3)
C2	0.25634 (8)	0.67397 (14)	0.37500 (7)	0.0194 (3)
C3	0.21774 (8)	0.84592 (15)	0.37156 (8)	0.0206 (3)
C4	0.28185 (8)	0.98676 (14)	0.37771 (8)	0.0218 (3)
H4	0.259422	1.102821	0.376752	0.026*
C5	0.37863 (8)	0.95713 (14)	0.38524 (8)	0.0226 (3)
H5	0.420152	1.053264	0.388885	0.027*
C6	0.41410 (8)	0.78494 (14)	0.38740 (8)	0.0212 (3)
H6	0.479072	0.766913	0.392141	0.025*
C7	0.35330 (8)	0.63899 (14)	0.38252 (8)	0.0201 (3)
C8	0.11209 (8)	0.87498 (15)	0.36144 (8)	0.0242 (3)
H8A	0.066418	0.821831	0.300578	0.036*
H8B	0.098978	1.000251	0.360303	0.036*
H8C	0.103281	0.821439	0.416947	0.036*
C9	0.38993 (8)	0.45156 (14)	0.38439 (8)	0.0240 (3)
H9A	0.387081	0.389722	0.440587	0.036*
H9B	0.457847	0.453512	0.389713	0.036*
H9C	0.348271	0.391761	0.324053	0.036*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0226 (5)	0.0205 (5)	0.0228 (5)	0.0008 (4)	0.0102 (4)	0.0010 (3)
C1	0.0295 (6)	0.0240 (6)	0.0348 (6)	−0.0018 (5)	0.0150 (5)	0.0009 (5)
C2	0.0234 (6)	0.0189 (5)	0.0169 (5)	−0.0033 (4)	0.0090 (4)	−0.0001 (4)
C3	0.0242 (6)	0.0217 (6)	0.0170 (5)	0.0010 (4)	0.0093 (4)	0.0004 (4)
C4	0.0269 (6)	0.0173 (5)	0.0216 (5)	0.0022 (4)	0.0102 (4)	0.0002 (4)
C5	0.0267 (6)	0.0192 (5)	0.0232 (5)	−0.0036 (4)	0.0114 (4)	0.0002 (4)

C6	0.0211 (5)	0.0228 (5)	0.0214 (5)	−0.0008 (4)	0.0103 (4)	0.0000 (4)
C7	0.0239 (5)	0.0192 (5)	0.0178 (5)	0.0007 (4)	0.0089 (4)	0.0000 (4)
C8	0.0242 (6)	0.0244 (5)	0.0255 (5)	0.0026 (4)	0.0115 (4)	0.0002 (4)
C9	0.0261 (6)	0.0187 (5)	0.0274 (6)	0.0015 (4)	0.0110 (4)	0.0006 (4)

Geometric parameters (Å, º)

N1—C1	1.1608 (15)	C6—H6	0.9300
N1—C2	1.4035 (13)	C6—C7	1.3936 (15)
C2—C3	1.4026 (15)	C7—C9	1.5033 (14)
C2—C7	1.3959 (15)	C8—H8A	0.9600
C3—C4	1.3904 (15)	C8—H8B	0.9600
C3—C8	1.5006 (15)	C8—H8C	0.9600
C4—H4	0.9300	C9—H9A	0.9600
C4—C5	1.3861 (16)	C9—H9B	0.9600
C5—H5	0.9300	C9—H9C	0.9600
C5—C6	1.3897 (15)		
C1—N1—C2	179.47 (11)	C2—C7—C9	121.21 (10)
C3—C2—N1	117.99 (9)	C6—C7—C2	117.18 (10)
C7—C2—N1	118.35 (9)	C6—C7—C9	121.61 (10)
C7—C2—C3	123.66 (10)	C3—C8—H8A	109.5
C2—C3—C8	121.17 (10)	C3—C8—H8B	109.5
C4—C3—C2	116.81 (10)	C3—C8—H8C	109.5
C4—C3—C8	122.01 (10)	H8A—C8—H8B	109.5
C3—C4—H4	119.4	H8A—C8—H8C	109.5
C5—C4—C3	121.13 (10)	H8B—C8—H8C	109.5
C5—C4—H4	119.4	C7—C9—H9A	109.5
C4—C5—H5	119.7	C7—C9—H9B	109.5
C4—C5—C6	120.53 (10)	C7—C9—H9C	109.5
C6—C5—H5	119.7	H9A—C9—H9B	109.5
C5—C6—H6	119.7	H9A—C9—H9C	109.5
C5—C6—C7	120.67 (10)	H9B—C9—H9C	109.5
C7—C6—H6	119.7		
N1—C2—C3—C4	178.95 (9)	C3—C4—C5—C6	−0.37 (17)
N1—C2—C3—C8	−1.20 (15)	C4—C5—C6—C7	−0.31 (17)
N1—C2—C7—C6	−179.59 (9)	C5—C6—C7—C2	0.29 (16)
N1—C2—C7—C9	0.90 (15)	C5—C6—C7—C9	179.80 (10)
C2—C3—C4—C5	1.01 (16)	C7—C2—C3—C4	−1.05 (16)
C3—C2—C7—C6	0.41 (16)	C7—C2—C3—C8	178.79 (9)
C3—C2—C7—C9	−179.10 (10)	C8—C3—C4—C5	−178.83 (10)

cis-Dibromidotetrakis(2,6-dimethylphenyl isocyanide)iron(II) dichloromethane 0.771-solvate (2a)

Crystal data

$[FeBr_2(C_9H_9N)_4]\cdot 0.771CH_2Cl_2$
M_r = 805.87

Monoclinic, *C*2/*c*
a = 16.8177 (13) Å

b = 15.8912 (12) Å
c = 14.1334 (11) Å
β = 92.9182 (10)°
V = 3772.3 (5) Å^3
Z = 4
F(000) = 1634
D_x = 1.419 Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1861 reflections
θ = 2.4–27.5°
μ = 2.66 mm^{-1}
T = 173 K
Needle, orange
0.44 × 0.16 × 0.08 mm

Data collection

Bruker SMART CCD platform diffractometer
Radiation source: normal-focus sealed tube
ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2014)
T_{min} = 0.662, T_{max} = 0.746
15633 measured reflections

4323 independent reflections
3646 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 27.5°, θ_{min} = 1.8°
h = −21→21
k = −19→20
l = −18→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.066
S = 1.03
4323 reflections
230 parameters
20 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0376P)^2 + 1.3322P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Special details

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

Refinement. During the mounting procedure there was a partial desolvation of the crystal. The cocrystallized dichloromethane solvent, which is modeled as disordered (see below), is found in channels along the [001] direction. The occupancies of the two components of the disorder were refined independently and summed to an occupancy of 0.771 (13) relative to one iron complex. A test using the SQUEEZE routine of program PLATON (Spek, 2015), also showed less than full occupancy of solvent in the channel: 168 electrons were expected, but only 150 were reported. The cocrystallized dichloromethane solvent was modeled as disordered over a crystallographic two-fold axis (0.50:0.50), and additionally over two general positions (0.579 (9) and 0.192 (9), see above response to alert PLAT077 for an explanation of why the two numbers do not sum to 1.000). Analogous bond lengths and angles between the two positions of the dichloromethane solvent disordere were restrained to be similar. Anisotropic displacement parameters for proximal (including symmetry equivalent) atoms were constrained to be equivalent. Additionally, anisotropic displacement parameters for all atoms of the major component were restrained to be similar.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
Fe1	0.500000	0.22824 (2)	0.250000	0.02214 (8)	
Br1	0.40420 (2)	0.11942 (2)	0.29733 (2)	0.03483 (7)	
C1	0.45319 (9)	0.22520 (10)	0.12645 (12)	0.0264 (3)	
N1	0.42660 (9)	0.22521 (9)	0.04962 (10)	0.0309 (3)	

C2	0.39319 (11)	0.22826 (12)	−0.04309 (12)	0.0348 (4)	
C3	0.41615 (13)	0.16724 (14)	−0.10721 (14)	0.0432 (5)	
C4	0.37932 (17)	0.17251 (19)	−0.19825 (16)	0.0632 (7)	
H4A	0.393109	0.133019	−0.245145	0.076*	
C5	0.32403 (18)	0.2332 (2)	−0.22106 (17)	0.0722 (9)	
H5A	0.299385	0.234244	−0.283042	0.087*	
C6	0.30326 (17)	0.29288 (19)	−0.15603 (19)	0.0687 (8)	
H6A	0.264967	0.334674	−0.173779	0.082*	
C7	0.33798 (14)	0.29235 (15)	−0.06463 (16)	0.0499 (5)	
C8	0.47627 (15)	0.10095 (16)	−0.07896 (19)	0.0610 (7)	
H8A	0.474795	0.056186	−0.126714	0.091*	
H8B	0.463744	0.077275	−0.017445	0.091*	
H8C	0.529541	0.126070	−0.074263	0.091*	
C9	0.31701 (18)	0.35630 (18)	0.0082 (2)	0.0738 (8)	
H9A	0.297795	0.327473	0.063952	0.111*	
H9B	0.275227	0.393571	−0.018524	0.111*	
H9C	0.364306	0.389593	0.026807	0.111*	
C10	0.42775 (10)	0.30503 (11)	0.29015 (11)	0.0257 (3)	
N2	0.38106 (9)	0.35035 (10)	0.32005 (10)	0.0299 (3)	
C11	0.32158 (10)	0.38481 (11)	0.37402 (13)	0.0292 (4)	
C12	0.26934 (10)	0.44403 (11)	0.33280 (13)	0.0318 (4)	
C13	0.20789 (11)	0.47249 (12)	0.38735 (14)	0.0371 (4)	
H13A	0.170436	0.511977	0.361295	0.045*	
C14	0.20098 (11)	0.44380 (13)	0.47894 (14)	0.0404 (5)	
H14A	0.158679	0.463631	0.515018	0.048*	
C15	0.25476 (12)	0.38675 (12)	0.51853 (14)	0.0404 (5)	
H15A	0.249421	0.368456	0.581887	0.049*	
C16	0.31685 (11)	0.35549 (12)	0.46705 (13)	0.0343 (4)	
C17	0.27863 (12)	0.47510 (13)	0.23332 (14)	0.0430 (5)	
H17A	0.236722	0.516159	0.216789	0.065*	0.5
H17B	0.330863	0.501814	0.229207	0.065*	0.5
H17C	0.274360	0.427536	0.189211	0.065*	0.5
H17D	0.324574	0.447514	0.206683	0.065*	0.5
H17E	0.230434	0.461858	0.194264	0.065*	0.5
H17F	0.286937	0.536137	0.234260	0.065*	0.5
C18	0.37621 (14)	0.29283 (15)	0.50771 (15)	0.0498 (5)	
H18A	0.362542	0.277862	0.572153	0.075*	
H18B	0.429634	0.317589	0.509368	0.075*	
H18C	0.375161	0.242179	0.468101	0.075*	
Cl1	0.5113 (5)	0.4486 (7)	0.6584 (7)	0.1084 (12)	0.290 (5)
C19	0.5096 (9)	0.3872 (6)	0.7698 (10)	0.094 (8)	0.290 (5)
H19A	0.464105	0.347616	0.767632	0.113*	0.290 (5)
H19B	0.559526	0.354894	0.780593	0.113*	0.290 (5)
Cl2	0.4993 (4)	0.4653 (6)	0.8629 (8)	0.1084 (12)	0.290 (5)
Cl1′	0.4834 (9)	0.4791 (9)	0.6060 (13)	0.1084 (12)	0.096 (4)
C19′	0.471 (3)	0.3923 (15)	0.6878 (17)	0.094 (8)	0.096 (4)
H19C	0.414493	0.376005	0.686380	0.113*	0.096 (4)
H19D	0.501982	0.343358	0.666233	0.113*	0.096 (4)

Cl2′	0.5042 (11)	0.4174 (13)	0.8087 (14)	0.1084 (12)	0.096 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.02145 (15)	0.02663 (17)	0.01831 (16)	0.000	0.00083 (12)	0.000
Br1	0.03405 (10)	0.03661 (11)	0.03405 (11)	−0.01018 (7)	0.00382 (7)	0.00266 (7)
C1	0.0249 (8)	0.0276 (8)	0.0268 (9)	0.0007 (6)	0.0027 (7)	0.0006 (7)
N1	0.0327 (7)	0.0360 (8)	0.0236 (8)	−0.0015 (6)	−0.0020 (6)	0.0000 (6)
C2	0.0373 (9)	0.0448 (11)	0.0215 (9)	−0.0091 (8)	−0.0054 (7)	0.0023 (8)
C3	0.0458 (11)	0.0521 (12)	0.0319 (10)	−0.0174 (10)	0.0022 (8)	−0.0081 (9)
C4	0.0738 (16)	0.0849 (19)	0.0309 (12)	−0.0353 (16)	0.0034 (11)	−0.0162 (12)
C5	0.0807 (19)	0.102 (2)	0.0307 (12)	−0.0364 (17)	−0.0225 (12)	0.0196 (14)
C6	0.0723 (17)	0.0808 (19)	0.0499 (15)	−0.0086 (15)	−0.0258 (13)	0.0245 (14)
C7	0.0536 (13)	0.0547 (13)	0.0400 (12)	0.0011 (11)	−0.0115 (10)	0.0121 (10)
C8	0.0578 (15)	0.0572 (14)	0.0685 (17)	−0.0023 (12)	0.0085 (12)	−0.0241 (13)
C9	0.0805 (19)	0.0667 (16)	0.0730 (19)	0.0321 (15)	−0.0076 (15)	0.0016 (15)
C10	0.0263 (8)	0.0308 (9)	0.0199 (8)	−0.0024 (7)	0.0007 (6)	0.0039 (7)
N2	0.0302 (7)	0.0324 (8)	0.0278 (8)	0.0025 (6)	0.0057 (6)	0.0004 (6)
C11	0.0274 (8)	0.0305 (9)	0.0302 (9)	0.0002 (7)	0.0072 (7)	−0.0050 (7)
C12	0.0330 (9)	0.0318 (9)	0.0309 (9)	0.0007 (7)	0.0044 (7)	−0.0040 (7)
C13	0.0327 (9)	0.0364 (10)	0.0426 (11)	0.0050 (8)	0.0055 (8)	−0.0060 (8)
C14	0.0354 (10)	0.0451 (11)	0.0419 (11)	0.0004 (9)	0.0145 (8)	−0.0112 (9)
C15	0.0435 (11)	0.0481 (12)	0.0309 (10)	−0.0011 (9)	0.0128 (8)	−0.0039 (9)
C16	0.0351 (9)	0.0380 (10)	0.0303 (10)	0.0000 (8)	0.0069 (8)	−0.0007 (8)
C17	0.0461 (11)	0.0458 (11)	0.0377 (11)	0.0136 (9)	0.0090 (9)	0.0060 (9)
C18	0.0563 (13)	0.0602 (14)	0.0337 (11)	0.0147 (11)	0.0089 (9)	0.0107 (10)
Cl1	0.085 (2)	0.068 (3)	0.169 (5)	−0.006 (2)	−0.022 (2)	−0.002 (2)
C19	0.059 (14)	0.051 (5)	0.17 (2)	0.018 (6)	−0.037 (11)	−0.047 (10)
Cl2	0.085 (2)	0.068 (3)	0.169 (5)	−0.006 (2)	−0.022 (2)	−0.002 (2)
Cl1′	0.085 (2)	0.068 (3)	0.169 (5)	−0.006 (2)	−0.022 (2)	−0.002 (2)
C19′	0.059 (14)	0.051 (5)	0.17 (2)	0.018 (6)	−0.037 (11)	−0.047 (10)
Cl2′	0.085 (2)	0.068 (3)	0.169 (5)	−0.006 (2)	−0.022 (2)	−0.002 (2)

Geometric parameters (Å, º)

Fe1—C10^{i}	1.8325 (17)	C11—C16	1.401 (3)
Fe1—C10	1.8325 (17)	C12—C13	1.396 (2)
Fe1—C1	1.8791 (17)	C12—C17	1.506 (3)
Fe1—C1^{i}	1.8791 (17)	C13—C14	1.383 (3)
Fe1—Br1	2.4785 (3)	C13—H13A	0.9500
Fe1—Br1^{i}	2.4785 (3)	C14—C15	1.379 (3)
C1—N1	1.153 (2)	C14—H14A	0.9500
N1—C2	1.400 (2)	C15—C16	1.394 (3)
C2—C3	1.395 (3)	C15—H15A	0.9500
C2—C7	1.401 (3)	C16—C18	1.503 (3)
C3—C4	1.402 (3)	C17—H17A	0.9800
C3—C8	1.500 (3)	C17—H17B	0.9800

C4—C5	1.367 (4)	C17—H17C	0.9800
C4—H4A	0.9500	C17—H17D	0.9800
C5—C6	1.378 (4)	C17—H17E	0.9800
C5—H5A	0.9500	C17—H17F	0.9800
C6—C7	1.390 (3)	C18—H18A	0.9800
C6—H6A	0.9500	C18—H18B	0.9800
C7—C9	1.502 (4)	C18—H18C	0.9800
C8—H8A	0.9800	Cl1—C19	1.854 (12)
C8—H8B	0.9800	C19—Cl2	1.823 (10)
C8—H8C	0.9800	C19—H19A	0.9900
C9—H9A	0.9800	C19—H19B	0.9900
C9—H9B	0.9800	Cl1′—C19′	1.817 (16)
C9—H9C	0.9800	C19′—Cl2′	1.813 (15)
C10—N2	1.161 (2)	C19′—H19C	0.9900
N2—C11	1.400 (2)	C19′—H19D	0.9900
C11—C12	1.394 (2)		

C10^{i}—Fe1—C10	96.49 (10)	N2—C10—Fe1	175.84 (15)
C10^{i}—Fe1—C1	88.99 (7)	C10—N2—C11	163.00 (17)
C10—Fe1—C1	92.97 (7)	C12—C11—N2	119.25 (16)
C10^{i}—Fe1—C1^{i}	92.97 (7)	C12—C11—C16	123.50 (16)
C10—Fe1—C1^{i}	88.99 (7)	N2—C11—C16	117.23 (16)
C1—Fe1—C1^{i}	177.05 (10)	C11—C12—C13	117.15 (17)
C10^{i}—Fe1—Br1	176.96 (5)	C11—C12—C17	121.21 (16)
C10—Fe1—Br1	86.02 (5)	C13—C12—C17	121.63 (17)
C1—Fe1—Br1	89.17 (5)	C14—C13—C12	120.67 (18)
C1^{i}—Fe1—Br1	88.77 (5)	C14—C13—H13A	119.7
C10^{i}—Fe1—Br1^{i}	86.02 (5)	C12—C13—H13A	119.7
C10—Fe1—Br1^{i}	176.96 (5)	C15—C14—C13	120.80 (17)
C1—Fe1—Br1^{i}	88.77 (5)	C15—C14—H14A	119.6
C1^{i}—Fe1—Br1^{i}	89.17 (5)	C13—C14—H14A	119.6
Br1—Fe1—Br1^{i}	91.520 (15)	C14—C15—C16	121.04 (18)
N1—C1—Fe1	177.55 (15)	C14—C15—H15A	119.5
C1—N1—C2	177.85 (18)	C16—C15—H15A	119.5
C3—C2—N1	118.21 (18)	C15—C16—C11	116.81 (18)
C3—C2—C7	124.50 (18)	C15—C16—C18	122.47 (17)
N1—C2—C7	117.29 (18)	C11—C16—C18	120.71 (16)
C2—C3—C4	115.5 (2)	C12—C17—H17A	109.5
C2—C3—C8	121.35 (19)	C12—C17—H17B	109.5
C4—C3—C8	123.1 (2)	H17A—C17—H17B	109.5
C5—C4—C3	121.5 (2)	C12—C17—H17C	109.5
C5—C4—H4A	119.3	H17A—C17—H17C	109.5
C3—C4—H4A	119.3	H17B—C17—H17C	109.5
C4—C5—C6	121.4 (2)	H17D—C17—H17E	109.5
C4—C5—H5A	119.3	H17D—C17—H17F	109.5
C6—C5—H5A	119.3	H17E—C17—H17F	109.5
C5—C6—C7	120.5 (3)	C16—C18—H18A	109.5
C5—C6—H6A	119.8	C16—C18—H18B	109.5

C7—C6—H6A	119.8	H18A—C18—H18B	109.5
C6—C7—C2	116.7 (2)	C16—C18—H18C	109.5
C6—C7—C9	122.1 (2)	H18A—C18—H18C	109.5
C2—C7—C9	121.28 (19)	H18B—C18—H18C	109.5
C3—C8—H8A	109.5	Cl2—C19—Cl1	105.2 (5)
C3—C8—H8B	109.5	Cl2—C19—H19A	110.7
H8A—C8—H8B	109.5	Cl1—C19—H19A	110.7
C3—C8—H8C	109.5	Cl2—C19—H19B	110.7
H8A—C8—H8C	109.5	Cl1—C19—H19B	110.7
H8B—C8—H8C	109.5	H19A—C19—H19B	108.8
C7—C9—H9A	109.5	Cl2′—C19′—Cl1′	113.2 (14)
C7—C9—H9B	109.5	Cl2′—C19′—H19C	108.9
H9A—C9—H9B	109.5	Cl1′—C19′—H19C	108.9
C7—C9—H9C	109.5	Cl2′—C19′—H19D	108.9
H9A—C9—H9C	109.5	Cl1′—C19′—H19D	108.9
H9B—C9—H9C	109.5	H19C—C19′—H19D	107.7

N1—C2—C3—C4	178.63 (18)	C10—N2—C11—C16	27.6 (7)
C7—C2—C3—C4	−0.6 (3)	N2—C11—C12—C13	175.94 (16)
N1—C2—C3—C8	−1.3 (3)	C16—C11—C12—C13	−2.3 (3)
C7—C2—C3—C8	179.5 (2)	N2—C11—C12—C17	−3.5 (3)
C2—C3—C4—C5	−0.8 (3)	C16—C11—C12—C17	178.28 (18)
C8—C3—C4—C5	179.1 (2)	C11—C12—C13—C14	1.3 (3)
C3—C4—C5—C6	1.3 (4)	C17—C12—C13—C14	−179.29 (18)
C4—C5—C6—C7	−0.5 (4)	C12—C13—C14—C15	0.3 (3)
C5—C6—C7—C2	−0.8 (4)	C13—C14—C15—C16	−0.9 (3)
C5—C6—C7—C9	180.0 (3)	C14—C15—C16—C11	0.0 (3)
C3—C2—C7—C6	1.4 (3)	C14—C15—C16—C18	−179.6 (2)
N1—C2—C7—C6	−177.9 (2)	C12—C11—C16—C15	1.7 (3)
C3—C2—C7—C9	−179.4 (2)	N2—C11—C16—C15	−176.60 (17)
N1—C2—C7—C9	1.4 (3)	C12—C11—C16—C18	−178.75 (19)
C10—N2—C11—C12	−150.8 (5)	N2—C11—C16—C18	3.0 (3)

Symmetry code: (i) $-x+1, y, -z+1/2$.

trans-Dibromidotetrakis(2,6-dimethylphenyl isocyanide)iron(II) (2b)

Crystal data

$[FeBr_2(C_9H_9N)_4]$
$M_r = 740.36$
Orthorhombic, *Pbca*
$a = 14.4246$ (13) Å
$b = 14.0229$ (13) Å
$c = 16.3444$ (15) Å
$V = 3306.1$ (5) Å^3
$Z = 4$
$F(000) = 1504$

$D_x = 1.487$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2048 reflections
$\theta = 2.4$–27.4°
$\mu = 2.90$ mm^{-1}
$T = 173$ K
Block, blue-violet
0.25 × 0.20 × 0.15 mm

Data collection

Siemens SMART CCD platform diffractometer
Radiation source: normal-focus sealed tube
ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2014)
T_{min} = 0.592, T_{max} = 0.746
25764 measured reflections
3790 independent reflections
3151 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 27.5°, θ_{min} = 2.4°
h = −17→18
k = −18→18
l = −21→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.065
S = 1.03
3790 reflections
198 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.031P)^2 + 2.1282P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.52 e Å^{-3}

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Fe1	0.000000	0.500000	0.500000	0.01608 (8)	
Br1	0.15064 (2)	0.50738 (2)	0.42887 (2)	0.02887 (7)	
C1	−0.06328 (12)	0.56121 (11)	0.41390 (10)	0.0193 (3)	
N1	−0.10460 (10)	0.60276 (10)	0.36485 (9)	0.0230 (3)	
C2	−0.15108 (12)	0.67006 (12)	0.31575 (10)	0.0209 (3)	
C3	−0.16043 (12)	0.65280 (12)	0.23198 (11)	0.0225 (4)	
C4	−0.19996 (13)	0.72519 (13)	0.18514 (11)	0.0263 (4)	
H4A	−0.206190	0.716791	0.127727	0.032*	
C5	−0.23023 (13)	0.80895 (13)	0.22070 (12)	0.0283 (4)	
H5A	−0.255244	0.858165	0.187406	0.034*	
C6	−0.22434 (12)	0.82168 (12)	0.30484 (12)	0.0263 (4)	
H6A	−0.248075	0.878373	0.328791	0.032*	
C7	−0.18419 (12)	0.75256 (12)	0.35443 (11)	0.0231 (3)	
C8	−0.13120 (14)	0.56026 (13)	0.19373 (12)	0.0314 (4)	
H8A	−0.105081	0.518397	0.235850	0.047*	0.5
H8B	−0.185127	0.529337	0.168706	0.047*	0.5
H8C	−0.084299	0.572607	0.151663	0.047*	0.5
H8D	−0.144590	0.561831	0.134963	0.047*	0.5
H8E	−0.064544	0.550890	0.202107	0.047*	0.5
H8F	−0.165373	0.507620	0.219149	0.047*	0.5

C9	−0.17650 (15)	0.76572 (15)	0.44568 (12)	0.0346 (5)	
H9A	−0.146411	0.709744	0.469840	0.052*	0.5
H9B	−0.139490	0.822717	0.457396	0.052*	0.5
H9C	−0.238583	0.773257	0.469151	0.052*	0.5
H9D	−0.203245	0.827402	0.461085	0.052*	0.5
H9E	−0.210166	0.714429	0.473529	0.052*	0.5
H9F	−0.111073	0.763888	0.461774	0.052*	0.5
C10	−0.02539 (12)	0.37964 (11)	0.45537 (10)	0.0201 (3)	
N2	−0.04306 (11)	0.30496 (10)	0.42980 (9)	0.0237 (3)	
C11	−0.06811 (12)	0.22107 (11)	0.38876 (10)	0.0205 (3)	
C12	−0.09778 (13)	0.14231 (13)	0.43430 (11)	0.0264 (4)	
C13	−0.12441 (14)	0.06187 (13)	0.39012 (13)	0.0330 (4)	
H13A	−0.144915	0.006661	0.418526	0.040*	
C14	−0.12147 (14)	0.06116 (13)	0.30577 (13)	0.0340 (4)	
H14A	−0.139892	0.005522	0.276866	0.041*	
C15	−0.09205 (13)	0.14043 (13)	0.26284 (11)	0.0277 (4)	
H15A	−0.091054	0.138693	0.204738	0.033*	
C16	−0.06396 (12)	0.22243 (12)	0.30311 (11)	0.0217 (3)	
C17	−0.10146 (17)	0.14458 (17)	0.52657 (12)	0.0408 (5)	
H17A	−0.056456	0.191126	0.547128	0.061*	0.5
H17B	−0.086430	0.081254	0.548182	0.061*	0.5
H17C	−0.163889	0.162832	0.544300	0.061*	0.5
H17D	−0.148061	0.099016	0.545946	0.061*	0.5
H17E	−0.118087	0.208888	0.544891	0.061*	0.5
H17F	−0.040628	0.127310	0.548773	0.061*	0.5
C18	−0.03274 (13)	0.31020 (13)	0.25826 (12)	0.0292 (4)	
H18A	0.031277	0.325211	0.273649	0.044*	
H18B	−0.073117	0.363829	0.272696	0.044*	
H18C	−0.036025	0.298833	0.199171	0.044*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.02056 (17)	0.01488 (15)	0.01281 (15)	0.00096 (12)	−0.00284 (12)	0.00069 (12)
Br1	0.02599 (11)	0.03546 (11)	0.02516 (11)	0.00368 (7)	0.00558 (7)	0.00650 (7)
C1	0.0228 (9)	0.0182 (7)	0.0171 (8)	−0.0021 (6)	−0.0004 (7)	−0.0007 (6)
N1	0.0239 (8)	0.0242 (7)	0.0210 (7)	−0.0017 (6)	−0.0039 (6)	0.0047 (6)
C2	0.0185 (8)	0.0224 (8)	0.0219 (8)	−0.0028 (6)	−0.0048 (7)	0.0081 (6)
C3	0.0220 (9)	0.0231 (8)	0.0225 (9)	−0.0040 (7)	−0.0035 (7)	0.0042 (6)
C4	0.0250 (10)	0.0331 (9)	0.0209 (9)	−0.0016 (7)	−0.0054 (7)	0.0079 (7)
C5	0.0241 (9)	0.0272 (9)	0.0336 (10)	0.0002 (7)	−0.0039 (8)	0.0132 (8)
C6	0.0213 (9)	0.0218 (8)	0.0356 (10)	−0.0010 (7)	0.0006 (7)	0.0037 (7)
C7	0.0183 (8)	0.0267 (8)	0.0242 (9)	−0.0045 (7)	−0.0006 (7)	0.0024 (7)
C8	0.0351 (11)	0.0289 (9)	0.0302 (10)	0.0002 (8)	−0.0087 (8)	−0.0033 (8)
C9	0.0366 (12)	0.0408 (11)	0.0266 (10)	0.0001 (9)	−0.0019 (9)	−0.0034 (8)
C10	0.0228 (9)	0.0212 (8)	0.0161 (8)	0.0020 (6)	−0.0036 (7)	0.0022 (6)
N2	0.0260 (8)	0.0217 (7)	0.0236 (7)	0.0005 (6)	−0.0027 (6)	−0.0029 (6)
C11	0.0195 (8)	0.0186 (7)	0.0235 (9)	0.0007 (6)	−0.0018 (7)	−0.0046 (6)

C12	0.0259 (9)	0.0265 (8)	0.0268 (9)	−0.0020 (7)	−0.0017 (8)	0.0027 (7)
C13	0.0336 (11)	0.0225 (8)	0.0430 (12)	−0.0075 (8)	−0.0012 (9)	0.0031 (8)
C14	0.0333 (11)	0.0253 (9)	0.0434 (12)	−0.0042 (8)	−0.0046 (9)	−0.0125 (8)
C15	0.0264 (10)	0.0327 (9)	0.0238 (9)	0.0024 (8)	−0.0017 (8)	−0.0083 (7)
C16	0.0175 (8)	0.0246 (8)	0.0228 (8)	0.0021 (6)	−0.0006 (7)	−0.0015 (7)
C17	0.0476 (14)	0.0477 (12)	0.0271 (10)	−0.0098 (10)	−0.0004 (10)	0.0086 (9)
C18	0.0268 (10)	0.0325 (9)	0.0283 (10)	0.0012 (8)	0.0018 (8)	0.0057 (8)

Geometric parameters (Å, º)

Fe1—C10	1.8748 (16)	C9—H9C	0.9800
Fe1—C10^{i}	1.8748 (16)	C9—H9D	0.9800
Fe1—C1^{i}	1.8842 (16)	C9—H9E	0.9800
Fe1—C1	1.8842 (16)	C9—H9F	0.9800
Fe1—Br1	2.4665 (3)	C10—N2	1.156 (2)
Fe1—Br1^{i}	2.4665 (3)	N2—C11	1.402 (2)
C1—N1	1.156 (2)	C11—C12	1.399 (2)
N1—C2	1.409 (2)	C11—C16	1.401 (2)
C2—C3	1.397 (2)	C12—C13	1.393 (3)
C2—C7	1.402 (2)	C12—C17	1.509 (3)
C3—C4	1.393 (2)	C13—C14	1.379 (3)
C3—C8	1.501 (2)	C13—H13A	0.9500
C4—C5	1.381 (3)	C14—C15	1.381 (3)
C4—H4A	0.9500	C14—H14A	0.9500
C5—C6	1.389 (3)	C15—C16	1.386 (2)
C5—H5A	0.9500	C15—H15A	0.9500
C6—C7	1.390 (2)	C16—C18	1.502 (2)
C6—H6A	0.9500	C17—H17A	0.9800
C7—C9	1.507 (3)	C17—H17B	0.9800
C8—H8A	0.9800	C17—H17C	0.9800
C8—H8B	0.9800	C17—H17D	0.9800
C8—H8C	0.9800	C17—H17E	0.9800
C8—H8D	0.9800	C17—H17F	0.9800
C8—H8E	0.9800	C18—H18A	0.9800
C8—H8F	0.9800	C18—H18B	0.9800
C9—H9A	0.9800	C18—H18C	0.9800
C9—H9B	0.9800		

C10—Fe1—C10^{i}	180.0	H9D—C9—H9E	109.5
C10—Fe1—C1^{i}	88.57 (7)	H9D—C9—H9F	109.5
C10^{i}—Fe1—C1^{i}	91.43 (7)	H9E—C9—H9F	109.5
C10—Fe1—C1	91.43 (7)	N2—C10—Fe1	177.84 (16)
C10^{i}—Fe1—C1	88.57 (7)	C10—N2—C11	171.99 (17)
C1^{i}—Fe1—C1	180.0	C12—C11—C16	123.74 (16)
C10—Fe1—Br1	91.52 (5)	C12—C11—N2	119.13 (16)
C10^{i}—Fe1—Br1	88.49 (5)	C16—C11—N2	117.10 (15)
C1^{i}—Fe1—Br1	86.81 (5)	C13—C12—C11	116.59 (17)
C1—Fe1—Br1	93.19 (5)	C13—C12—C17	121.68 (17)

C10—Fe1—Br1^{i}	88.49 (5)	C11—C12—C17	121.72 (17)
C10^{i}—Fe1—Br1^{i}	91.51 (5)	C14—C13—C12	121.02 (18)
C1^{i}—Fe1—Br1^{i}	93.19 (5)	C14—C13—H13A	119.5
C1—Fe1—Br1^{i}	86.81 (5)	C12—C13—H13A	119.5
Br1—Fe1—Br1^{i}	180.0	C13—C14—C15	120.76 (17)
N1—C1—Fe1	175.50 (15)	C13—C14—H14A	119.6
C1—N1—C2	167.90 (17)	C15—C14—H14A	119.6
C3—C2—C7	123.50 (15)	C14—C15—C16	121.09 (17)
C3—C2—N1	119.23 (15)	C14—C15—H15A	119.5
C7—C2—N1	117.26 (15)	C16—C15—H15A	119.5
C4—C3—C2	116.86 (16)	C15—C16—C11	116.80 (16)
C4—C3—C8	121.07 (16)	C15—C16—C18	122.41 (17)
C2—C3—C8	122.06 (15)	C11—C16—C18	120.77 (16)
C5—C4—C3	121.18 (17)	C12—C17—H17A	109.5
C5—C4—H4A	119.4	C12—C17—H17B	109.5
C3—C4—H4A	119.4	H17A—C17—H17B	109.5
C4—C5—C6	120.42 (16)	C12—C17—H17C	109.5
C4—C5—H5A	119.8	H17A—C17—H17C	109.5
C6—C5—H5A	119.8	H17B—C17—H17C	109.5
C5—C6—C7	120.87 (17)	C12—C17—H17D	109.5
C5—C6—H6A	119.6	H17A—C17—H17D	141.1
C7—C6—H6A	119.6	H17B—C17—H17D	56.3
C6—C7—C2	117.02 (16)	H17C—C17—H17D	56.3
C6—C7—C9	121.50 (17)	C12—C17—H17E	109.5
C2—C7—C9	121.48 (16)	H17A—C17—H17E	56.3
C3—C8—H8A	109.5	H17B—C17—H17E	141.1
C3—C8—H8B	109.5	H17C—C17—H17E	56.3
H8A—C8—H8B	109.5	H17D—C17—H17E	109.5
C3—C8—H8C	109.5	C12—C17—H17F	109.5
H8A—C8—H8C	109.5	H17A—C17—H17F	56.3
H8B—C8—H8C	109.5	H17B—C17—H17F	56.3
H8D—C8—H8E	109.5	H17C—C17—H17F	141.1
H8D—C8—H8F	109.5	H17D—C17—H17F	109.5
H8E—C8—H8F	109.5	H17E—C17—H17F	109.5
C7—C9—H9A	109.5	C16—C18—H18A	109.5
C7—C9—H9B	109.5	C16—C18—H18B	109.5
H9A—C9—H9B	109.5	H18A—C18—H18B	109.5
C7—C9—H9C	109.5	C16—C18—H18C	109.5
H9A—C9—H9C	109.5	H18A—C18—H18C	109.5
H9B—C9—H9C	109.5	H18B—C18—H18C	109.5

C1—N1—C2—C3	154.1 (8)	N1—C2—C7—C9	−4.2 (3)
C1—N1—C2—C7	−24.8 (9)	C16—C11—C12—C13	−0.1 (3)
C7—C2—C3—C4	4.0 (3)	N2—C11—C12—C13	178.14 (17)
N1—C2—C3—C4	−174.72 (16)	C16—C11—C12—C17	−179.69 (19)
C7—C2—C3—C8	−174.87 (17)	N2—C11—C12—C17	−1.4 (3)
N1—C2—C3—C8	6.4 (3)	C11—C12—C13—C14	−0.1 (3)
C2—C3—C4—C5	−1.5 (3)	C17—C12—C13—C14	179.4 (2)

C8—C3—C4—C5	177.44 (17)	C12—C13—C14—C15	−0.1 (3)
C3—C4—C5—C6	−1.9 (3)	C13—C14—C15—C16	0.6 (3)
C4—C5—C6—C7	2.9 (3)	C14—C15—C16—C11	−0.8 (3)
C5—C6—C7—C2	−0.5 (3)	C14—C15—C16—C18	−179.21 (17)
C5—C6—C7—C9	179.43 (17)	C12—C11—C16—C15	0.6 (3)
C3—C2—C7—C6	−3.0 (3)	N2—C11—C16—C15	−177.71 (15)
N1—C2—C7—C6	175.72 (15)	C12—C11—C16—C18	179.04 (17)
C3—C2—C7—C9	177.00 (17)	N2—C11—C16—C18	0.7 (3)

Symmetry code: (i) −*x*, −*y*+1, −*z*+1.

Pentakis(2,6-dimethylphenyl isocyanide)iron(0) tris(μ_2-2,6-dimethylphenyl isocyanide)bis[tris(2,6-dimethylphenyl isocyanide)iron(0)] (3)

Crystal data

$[Fe(C_9H_9N)_5][Fe_2(C_9H_9N)_9]$
M_r = 2003.95
Monoclinic, $P2_1/n$
a = 24.901 (3) Å
b = 14.2838 (19) Å
c = 30.954 (4) Å
β = 96.654 (2)°
V = 10935 (3) Å^3
Z = 4
$F(000)$ = 4232
D_x = 1.217 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4028 reflections
θ = 2.2–25.0°
μ = 0.45 mm^{-1}
T = 173 K
Plate, red
0.36 × 0.28 × 0.08 mm

Data collection

Bruker SMART CCD platform diffractometer
Radiation source: normal-focus sealed tube
ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2014)
T_{min} = 0.662, T_{max} = 0.746
75619 measured reflections
19257 independent reflections
10377 reflections with $I > 2\sigma(I)$
R_{int} = 0.086
θ_{max} = 25.0°, θ_{min} = 1.3°
h = −29→29
k = −16→16
l = −36→36

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.153
S = 1.02
19257 reflections
1316 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0675P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Fe1	0.22105 (2)	0.44139 (4)	0.03639 (2)	0.03866 (16)
N1	0.31653 (14)	0.3460 (2)	0.00409 (10)	0.0470 (9)
N2	0.21434 (14)	0.3871 (2)	0.12960 (12)	0.0543 (10)
N3	0.14953 (14)	0.2785 (2)	0.00842 (11)	0.0503 (9)
N4	0.13589 (14)	0.5677 (2)	−0.00815 (12)	0.0536 (9)
N5	0.27948 (13)	0.6227 (3)	0.05564 (11)	0.0491 (9)
C1	0.27835 (17)	0.3861 (3)	0.01537 (12)	0.0425 (10)
C2	0.36379 (16)	0.3255 (3)	−0.01425 (13)	0.0458 (11)
C3	0.38571 (18)	0.2355 (3)	−0.00936 (14)	0.0580 (12)
C4	0.4334 (2)	0.2192 (4)	−0.02749 (18)	0.0795 (16)
H4A	0.449717	0.158986	−0.024901	0.095*
C5	0.4571 (2)	0.2884 (5)	−0.0489 (2)	0.0890 (19)
H5A	0.489931	0.275914	−0.060662	0.107*
C6	0.4345 (2)	0.3753 (5)	−0.05379 (16)	0.0792 (16)
H6A	0.451290	0.422063	−0.069389	0.095*
C7	0.38719 (19)	0.3959 (3)	−0.03621 (14)	0.0546 (12)
C8	0.3592 (2)	0.1581 (3)	0.01352 (18)	0.0833 (16)
H8A	0.344875	0.183389	0.039305	0.125*
H8B	0.385871	0.109307	0.022359	0.125*
H8C	0.329546	0.131294	−0.006227	0.125*
C9	0.3605 (2)	0.4909 (3)	−0.04256 (16)	0.0835 (17)
H9A	0.383129	0.532089	−0.058207	0.125*
H9B	0.356197	0.518323	−0.014139	0.125*
H9C	0.324944	0.483925	−0.059448	0.125*
C10	0.21768 (16)	0.4112 (3)	0.09343 (14)	0.0438 (10)
C11	0.19994 (19)	0.3458 (3)	0.16815 (14)	0.0520 (12)
C12	0.1519 (2)	0.2937 (3)	0.16517 (16)	0.0617 (13)
C13	0.1386 (2)	0.2535 (4)	0.20327 (19)	0.0816 (16)
H13A	0.106560	0.217141	0.202681	0.098*
C14	0.1712 (3)	0.2654 (4)	0.2420 (2)	0.0914 (19)
H14A	0.161165	0.237742	0.267830	0.110*
C15	0.2178 (2)	0.3165 (4)	0.24370 (16)	0.0780 (16)
H15A	0.239792	0.323654	0.270716	0.094*
C16	0.23395 (19)	0.3588 (3)	0.20627 (15)	0.0575 (12)
C17	0.1167 (2)	0.2813 (4)	0.12288 (16)	0.0759 (15)
H17A	0.084003	0.246552	0.127904	0.114*
H17B	0.136486	0.246331	0.102558	0.114*
H17C	0.106461	0.342790	0.110536	0.114*
C18	0.28582 (19)	0.4142 (3)	0.20696 (15)	0.0652 (13)
H18A	0.298918	0.431227	0.237008	0.098*
H18B	0.279020	0.471104	0.189529	0.098*
H18C	0.313127	0.376004	0.194804	0.098*
C19	0.17827 (16)	0.3418 (3)	0.01932 (12)	0.0407 (10)
C20	0.11466 (17)	0.2052 (3)	−0.00479 (13)	0.0466 (11)
C21	0.0596 (2)	0.2142 (4)	−0.00154 (14)	0.0606 (13)

C22	0.0260 (2)	0.1390 (5)	−0.01518 (16)	0.0777 (17)
H22A	−0.011713	0.142358	−0.013234	0.093*
C23	0.0484 (3)	0.0589 (5)	−0.03170 (18)	0.099 (2)
H23A	0.025658	0.007596	−0.040891	0.119*
C24	0.1018 (3)	0.0533 (4)	−0.03480 (17)	0.0875 (18)
H24A	0.115507	−0.002024	−0.046533	0.105*
C25	0.1374 (2)	0.1244 (3)	−0.02172 (14)	0.0623 (13)
C26	0.0371 (2)	0.3001 (4)	0.01536 (17)	0.0859 (17)
H26A	−0.000313	0.308239	0.002230	0.129*
H26B	0.058738	0.354030	0.008097	0.129*
H26C	0.038043	0.295465	0.047023	0.129*
C27	0.1970 (2)	0.1167 (4)	−0.02383 (19)	0.0902 (18)
H27A	0.205654	0.053260	−0.032966	0.135*
H27B	0.216401	0.129506	0.004932	0.135*
H27C	0.207939	0.162113	−0.044856	0.135*
C28	0.16981 (17)	0.5161 (3)	0.00845 (13)	0.0427 (10)
C29	0.10708 (15)	0.6224 (3)	−0.04069 (13)	0.0423 (10)
C30	0.11309 (16)	0.7203 (3)	−0.03803 (14)	0.0461 (10)
C31	0.08544 (19)	0.7720 (3)	−0.07186 (16)	0.0614 (13)
H31A	0.088677	0.838300	−0.071667	0.074*
C32	0.0534 (2)	0.7290 (4)	−0.10564 (16)	0.0675 (14)
H32A	0.035772	0.765711	−0.128621	0.081*
C33	0.04705 (19)	0.6341 (4)	−0.10619 (15)	0.0669 (14)
H33A	0.024339	0.605667	−0.129292	0.080*
C34	0.07315 (17)	0.5786 (3)	−0.07361 (14)	0.0506 (11)
C35	0.14722 (19)	0.7643 (3)	−0.00035 (15)	0.0637 (13)
H35A	0.135167	0.742277	0.026925	0.096*
H35B	0.143655	0.832521	−0.002163	0.096*
H35C	0.185134	0.746762	−0.001199	0.096*
C36	0.0651 (2)	0.4748 (3)	−0.07404 (16)	0.0739 (15)
H36A	0.056710	0.453215	−0.104124	0.111*
H36B	0.035084	0.458960	−0.057484	0.111*
H36C	0.098180	0.444142	−0.060793	0.111*
C37	0.25862 (16)	0.5492 (3)	0.04894 (12)	0.0425 (10)
C38	0.29086 (16)	0.7164 (3)	0.06727 (15)	0.0479 (11)
C39	0.28553 (17)	0.7422 (3)	0.11051 (17)	0.0622 (13)
C40	0.2947 (2)	0.8368 (4)	0.1201 (2)	0.0900 (19)
H40A	0.291032	0.859020	0.148536	0.108*
C41	0.3090 (2)	0.8990 (4)	0.0890 (3)	0.094 (2)
H41A	0.314814	0.962978	0.096563	0.113*
C42	0.3151 (2)	0.8698 (4)	0.0476 (2)	0.0842 (18)
H42A	0.325212	0.913409	0.026827	0.101*
C43	0.30636 (17)	0.7762 (3)	0.03605 (17)	0.0598 (13)
C44	0.2726 (2)	0.6708 (4)	0.14287 (17)	0.0864 (17)
H44A	0.237639	0.641838	0.133016	0.130*
H44B	0.270815	0.701042	0.171093	0.130*
H44C	0.300807	0.622669	0.145769	0.130*
C45	0.3144 (2)	0.7401 (4)	−0.00862 (17)	0.0819 (16)

H45A	0.320106	0.792980	−0.027726	0.123*
H45B	0.282256	0.705059	−0.020659	0.123*
H45C	0.346027	0.698808	−0.006455	0.123*
Fe2	0.48319 (2)	0.29471 (4)	0.23064 (2)	0.03793 (16)
Fe3	0.57102 (2)	0.21657 (4)	0.22556 (2)	0.03666 (15)
N6	0.37587 (14)	0.1993 (2)	0.20430 (10)	0.0464 (9)
N7	0.44465 (14)	0.3945 (2)	0.30715 (11)	0.0488 (9)
N8	0.45217 (14)	0.4578 (2)	0.17140 (12)	0.0513 (9)
N9	0.48951 (13)	0.2150 (2)	0.14245 (11)	0.0449 (8)
N10	0.51659 (12)	0.1446 (2)	0.30016 (11)	0.0410 (8)
N11	0.57460 (13)	0.4306 (2)	0.24689 (10)	0.0418 (8)
N12	0.57532 (12)	0.0050 (3)	0.22404 (10)	0.0429 (8)
N13	0.66447 (12)	0.2235 (2)	0.29755 (10)	0.0418 (8)
N14	0.65374 (13)	0.2318 (2)	0.16143 (10)	0.0451 (9)
C46	0.41850 (17)	0.2319 (3)	0.21639 (12)	0.0412 (10)
C47	0.32775 (16)	0.1720 (3)	0.17897 (14)	0.0480 (11)
C48	0.31731 (16)	0.2094 (3)	0.13688 (14)	0.0493 (11)
C49	0.26981 (18)	0.1801 (3)	0.11257 (16)	0.0632 (13)
H49A	0.260780	0.204073	0.084023	0.076*
C50	0.2357 (2)	0.1173 (4)	0.1289 (2)	0.0765 (16)
H50A	0.204364	0.096081	0.111222	0.092*
C51	0.2466 (2)	0.0853 (3)	0.1705 (2)	0.0781 (17)
H51A	0.221760	0.043329	0.181367	0.094*
C52	0.29269 (18)	0.1117 (3)	0.19757 (17)	0.0595 (13)
C53	0.35431 (17)	0.2801 (3)	0.12026 (14)	0.0592 (13)
H53A	0.358936	0.333099	0.140453	0.089*
H53B	0.338660	0.302274	0.091572	0.089*
H53C	0.389532	0.251188	0.117877	0.089*
C54	0.3044 (2)	0.0807 (4)	0.24386 (18)	0.0861 (17)
H54A	0.270476	0.075600	0.256864	0.129*
H54B	0.328062	0.126488	0.260137	0.129*
H54C	0.322395	0.019533	0.244893	0.129*
C55	0.46174 (16)	0.3540 (3)	0.27856 (14)	0.0436 (10)
C56	0.41949 (19)	0.4209 (3)	0.34382 (14)	0.0529 (12)
C57	0.3740 (2)	0.3703 (4)	0.35267 (16)	0.0699 (15)
C58	0.3514 (2)	0.3932 (6)	0.3898 (2)	0.096 (2)
H58A	0.320550	0.360233	0.397044	0.116*
C59	0.3726 (3)	0.4613 (6)	0.4155 (2)	0.105 (2)
H59A	0.356554	0.473225	0.441313	0.126*
C60	0.4153 (3)	0.5152 (4)	0.40802 (19)	0.089 (2)
H60A	0.427635	0.564951	0.427021	0.107*
C61	0.4415 (2)	0.4934 (4)	0.36932 (18)	0.0751 (15)
C62	0.3520 (2)	0.2922 (4)	0.32309 (18)	0.0885 (17)
H62A	0.343778	0.316192	0.293399	0.133*
H62B	0.318904	0.267540	0.333198	0.133*
H62C	0.378916	0.242109	0.323433	0.133*
C63	0.4892 (3)	0.5446 (4)	0.3580 (2)	0.106 (2)
H63A	0.497263	0.596451	0.378483	0.160*

H63B	0.481753	0.569510	0.328408	0.160*
H63C	0.520204	0.502119	0.359595	0.160*
C64	0.46387 (15)	0.3933 (3)	0.19426 (13)	0.0421 (10)
C65	0.44089 (15)	0.5475 (3)	0.15406 (15)	0.0485 (11)
C66	0.42878 (16)	0.5576 (3)	0.10934 (15)	0.0543 (12)
C67	0.41855 (17)	0.6481 (4)	0.09363 (18)	0.0696 (15)
H67A	0.409979	0.658124	0.063270	0.083*
C68	0.42074 (19)	0.7228 (4)	0.1215 (2)	0.0756 (16)
H68A	0.413849	0.783834	0.110052	0.091*
C69	0.43256 (19)	0.7118 (3)	0.1655 (2)	0.0702 (14)
H69A	0.433379	0.764863	0.184082	0.084*
C70	0.44349 (17)	0.6225 (3)	0.18319 (16)	0.0570 (12)
C71	0.4263 (2)	0.4754 (4)	0.07893 (15)	0.0806 (16)
H71A	0.441062	0.493357	0.052122	0.121*
H71B	0.388631	0.455455	0.071971	0.121*
H71C	0.447602	0.423615	0.092856	0.121*
C72	0.4572 (2)	0.6066 (3)	0.23141 (15)	0.0721 (14)
H72A	0.459957	0.666999	0.246485	0.108*
H72B	0.491818	0.573458	0.236741	0.108*
H72C	0.428772	0.568871	0.242300	0.108*
C73	0.50909 (15)	0.2299 (3)	0.18069 (12)	0.0380 (9)
C74	0.50691 (16)	0.1606 (3)	0.10838 (13)	0.0442 (10)
C75	0.53932 (17)	0.2004 (3)	0.07920 (13)	0.0494 (11)
C76	0.55468 (19)	0.1432 (4)	0.04648 (15)	0.0627 (13)
H76A	0.576890	0.168436	0.026375	0.075*
C77	0.5387 (2)	0.0513 (4)	0.04234 (16)	0.0701 (14)
H77A	0.551050	0.012933	0.020459	0.084*
C78	0.50454 (19)	0.0150 (3)	0.07014 (15)	0.0609 (13)
H78A	0.492598	−0.047955	0.066515	0.073*
C79	0.48717 (17)	0.0688 (3)	0.10344 (14)	0.0492 (11)
C80	0.55509 (19)	0.3018 (3)	0.08107 (14)	0.0631 (13)
H80A	0.594497	0.307093	0.082549	0.095*
H80B	0.542865	0.330641	0.106940	0.095*
H80C	0.538218	0.333775	0.054960	0.095*
C81	0.44740 (18)	0.0334 (3)	0.13308 (16)	0.0645 (13)
H81A	0.436754	−0.030871	0.124927	0.097*
H81B	0.415330	0.073652	0.130247	0.097*
H81C	0.464362	0.034382	0.163272	0.097*
C82	0.51861 (15)	0.1951 (3)	0.26791 (13)	0.0392 (10)
C83	0.47746 (15)	0.1323 (3)	0.32913 (13)	0.0407 (10)
C84	0.44111 (17)	0.0582 (3)	0.32149 (14)	0.0511 (11)
C85	0.40438 (19)	0.0423 (4)	0.35142 (18)	0.0678 (14)
H85A	0.378699	−0.006804	0.346257	0.081*
C86	0.4045 (2)	0.0962 (4)	0.38833 (17)	0.0716 (15)
H86A	0.379091	0.084753	0.408354	0.086*
C87	0.44209 (19)	0.1669 (4)	0.39578 (15)	0.0606 (13)
H87A	0.442713	0.203257	0.421546	0.073*
C88	0.47908 (17)	0.1867 (3)	0.36669 (13)	0.0471 (11)

C89	0.44298 (19)	−0.0044 (3)	0.28213 (15)	0.0662 (13)
H89A	0.415480	−0.053418	0.282190	0.099*
H89B	0.478823	−0.033182	0.283187	0.099*
H89C	0.435846	0.033106	0.255558	0.099*
C90	0.52158 (18)	0.2613 (3)	0.37707 (14)	0.0571 (12)
H90A	0.505490	0.315231	0.390309	0.086*
H90B	0.535707	0.280928	0.350237	0.086*
H90C	0.551136	0.236064	0.397418	0.086*
C91	0.55543 (16)	0.3523 (3)	0.23772 (12)	0.0407 (10)
C92	0.62725 (15)	0.4684 (3)	0.24936 (13)	0.0387 (10)
C93	0.65795 (17)	0.4774 (3)	0.29009 (13)	0.0435 (10)
C94	0.70859 (18)	0.5188 (3)	0.29143 (15)	0.0548 (12)
H94A	0.730385	0.525246	0.318559	0.066*
C95	0.72785 (18)	0.5510 (3)	0.25394 (17)	0.0586 (13)
H95A	0.763051	0.577369	0.255498	0.070*
C96	0.69658 (18)	0.5452 (3)	0.21466 (15)	0.0548 (12)
H96A	0.709858	0.569395	0.189345	0.066*
C97	0.64535 (17)	0.5041 (3)	0.21135 (14)	0.0461 (11)
C98	0.63644 (18)	0.4466 (3)	0.33134 (14)	0.0623 (13)
H98A	0.666245	0.443052	0.354920	0.093*
H98B	0.619572	0.384785	0.326920	0.093*
H98C	0.609520	0.491798	0.339018	0.093*
C99	0.60969 (18)	0.4994 (3)	0.16868 (13)	0.0589 (12)
H99A	0.625660	0.536929	0.146932	0.088*
H99B	0.573795	0.524075	0.172435	0.088*
H99C	0.606390	0.434211	0.158888	0.088*
C100	0.57287 (15)	0.0881 (3)	0.22227 (12)	0.0401 (10)
C101	0.58373 (16)	−0.0894 (3)	0.23737 (14)	0.0440 (10)
C102	0.60646 (16)	−0.1047 (3)	0.28053 (14)	0.0498 (11)
C103	0.61455 (19)	−0.1973 (3)	0.29358 (16)	0.0615 (13)
H10I	0.630117	−0.210193	0.322457	0.074*
C104	0.6005 (2)	−0.2711 (3)	0.26565 (19)	0.0696 (14)
H10J	0.605780	−0.333778	0.275521	0.084*
C105	0.57881 (19)	−0.2533 (3)	0.22317 (18)	0.0641 (13)
H10K	0.569599	−0.304400	0.204103	0.077*
C106	0.57010 (17)	−0.1616 (3)	0.20767 (15)	0.0497 (11)
C107	0.62318 (17)	−0.0248 (3)	0.31054 (13)	0.0560 (12)
H10A	0.634405	−0.049012	0.339822	0.084*
H10B	0.592630	0.018109	0.311445	0.084*
H10C	0.653424	0.008661	0.299956	0.084*
C108	0.54868 (18)	−0.1427 (3)	0.16158 (14)	0.0613 (13)
H10D	0.545873	−0.201659	0.145285	0.092*
H10E	0.573227	−0.100140	0.148636	0.092*
H10F	0.512845	−0.113831	0.160480	0.092*
C109	0.62735 (16)	0.2221 (2)	0.27036 (12)	0.0362 (9)
C110	0.71351 (15)	0.2357 (3)	0.32387 (13)	0.0414 (10)
C111	0.71744 (16)	0.2082 (3)	0.36749 (13)	0.0460 (10)
C112	0.76614 (17)	0.2209 (3)	0.39254 (14)	0.0585 (12)

H11G	0.770084	0.201568	0.422107	0.070*
C113	0.80947 (18)	0.2612 (3)	0.37547 (16)	0.0637 (13)
H11H	0.842658	0.270596	0.393386	0.076*
C114	0.80446 (17)	0.2878 (3)	0.33233 (15)	0.0553 (12)
H11I	0.834599	0.314846	0.320771	0.066*
C115	0.75648 (16)	0.2760 (3)	0.30557 (13)	0.0426 (10)
C116	0.66989 (18)	0.1664 (4)	0.38705 (14)	0.0693 (14)
H11A	0.649506	0.125409	0.365677	0.104*
H11B	0.646300	0.216766	0.395198	0.104*
H11C	0.683072	0.129966	0.412956	0.104*
C117	0.75213 (17)	0.3015 (3)	0.25807 (13)	0.0521 (11)
H11D	0.783074	0.340481	0.252724	0.078*
H11E	0.718548	0.336351	0.250040	0.078*
H11F	0.751935	0.244344	0.240556	0.078*
C118	0.61970 (15)	0.2327 (3)	0.18491 (12)	0.0363 (9)
C119	0.69374 (16)	0.2018 (3)	0.13607 (12)	0.0436 (10)
C120	0.73373 (17)	0.2659 (3)	0.12729 (13)	0.0495 (11)
C121	0.77412 (18)	0.2317 (4)	0.10388 (14)	0.0611 (13)
H12G	0.802780	0.272001	0.098111	0.073*
C122	0.7735 (2)	0.1404 (4)	0.08887 (15)	0.0663 (14)
H12H	0.801121	0.119403	0.072490	0.080*
C123	0.73283 (19)	0.0796 (3)	0.09752 (14)	0.0586 (12)
H12I	0.732875	0.017105	0.087044	0.070*
C124	0.69179 (16)	0.1090 (3)	0.12146 (13)	0.0473 (11)
C125	0.73311 (19)	0.3655 (3)	0.14131 (15)	0.0636 (13)
H12A	0.761435	0.400123	0.128617	0.095*
H12B	0.739699	0.368867	0.173102	0.095*
H12C	0.697791	0.393138	0.131430	0.095*
C126	0.64974 (17)	0.0411 (3)	0.13299 (14)	0.0575 (12)
H12D	0.651205	−0.015825	0.115510	0.086*
H12E	0.613844	0.069641	0.127071	0.086*
H12F	0.656689	0.025192	0.163933	0.086*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.0395 (3)	0.0383 (3)	0.0386 (3)	0.0002 (3)	0.0059 (3)	−0.0026 (3)
N1	0.044 (2)	0.053 (2)	0.044 (2)	0.0076 (19)	0.0041 (17)	−0.0067 (17)
N2	0.066 (3)	0.054 (2)	0.046 (2)	0.0042 (19)	0.0147 (19)	0.0036 (19)
N3	0.052 (2)	0.046 (2)	0.054 (2)	−0.002 (2)	0.0087 (18)	−0.0046 (18)
N4	0.049 (2)	0.047 (2)	0.063 (2)	−0.0060 (19)	0.0005 (19)	0.000 (2)
N5	0.041 (2)	0.052 (2)	0.053 (2)	−0.0014 (19)	0.0005 (17)	−0.0074 (19)
C1	0.045 (3)	0.040 (3)	0.041 (2)	−0.007 (2)	0.000 (2)	−0.002 (2)
C2	0.041 (3)	0.061 (3)	0.035 (2)	−0.001 (2)	0.004 (2)	−0.010 (2)
C3	0.055 (3)	0.066 (3)	0.054 (3)	0.019 (3)	0.012 (2)	−0.012 (2)
C4	0.060 (3)	0.093 (4)	0.086 (4)	0.020 (3)	0.011 (3)	−0.018 (3)
C5	0.054 (3)	0.122 (6)	0.096 (5)	0.003 (4)	0.028 (3)	−0.030 (4)
C6	0.081 (4)	0.099 (5)	0.061 (3)	−0.031 (4)	0.024 (3)	0.002 (3)

C7	0.058 (3)	0.067 (3)	0.040 (3)	−0.009 (3)	0.010 (2)	−0.005 (2)
C8	0.096 (4)	0.061 (4)	0.096 (4)	0.022 (3)	0.024 (3)	0.005 (3)
C9	0.121 (5)	0.065 (4)	0.067 (4)	−0.003 (3)	0.023 (3)	0.015 (3)
C10	0.042 (2)	0.045 (3)	0.044 (3)	0.006 (2)	0.004 (2)	−0.006 (2)
C11	0.066 (3)	0.050 (3)	0.043 (3)	0.015 (2)	0.020 (2)	0.007 (2)
C12	0.066 (3)	0.058 (3)	0.065 (3)	0.006 (3)	0.027 (3)	0.011 (3)
C13	0.072 (4)	0.097 (4)	0.080 (4)	0.006 (3)	0.028 (3)	0.032 (3)
C14	0.079 (4)	0.119 (5)	0.081 (4)	0.015 (4)	0.032 (4)	0.047 (4)
C15	0.086 (4)	0.099 (4)	0.052 (3)	0.023 (3)	0.018 (3)	0.024 (3)
C16	0.065 (3)	0.059 (3)	0.053 (3)	0.020 (3)	0.022 (3)	0.008 (2)
C17	0.078 (4)	0.076 (4)	0.075 (4)	−0.002 (3)	0.014 (3)	0.002 (3)
C18	0.073 (3)	0.067 (3)	0.058 (3)	0.012 (3)	0.016 (3)	0.001 (2)
C19	0.042 (2)	0.038 (3)	0.043 (2)	0.002 (2)	0.010 (2)	0.001 (2)
C20	0.054 (3)	0.043 (3)	0.043 (2)	−0.017 (2)	0.004 (2)	0.003 (2)
C21	0.060 (3)	0.078 (4)	0.042 (3)	−0.007 (3)	0.003 (2)	0.016 (3)
C22	0.068 (4)	0.109 (5)	0.053 (3)	−0.037 (4)	−0.005 (3)	0.027 (3)
C23	0.127 (6)	0.103 (5)	0.061 (4)	−0.072 (5)	−0.014 (4)	0.018 (4)
C24	0.131 (6)	0.059 (4)	0.073 (4)	−0.037 (4)	0.012 (4)	−0.004 (3)
C25	0.090 (4)	0.047 (3)	0.052 (3)	−0.021 (3)	0.015 (3)	0.000 (2)
C26	0.062 (3)	0.115 (5)	0.084 (4)	0.012 (3)	0.022 (3)	0.024 (4)
C27	0.101 (5)	0.058 (3)	0.118 (5)	0.013 (3)	0.043 (4)	−0.017 (3)
C28	0.046 (3)	0.037 (2)	0.044 (2)	−0.003 (2)	0.001 (2)	0.000 (2)
C29	0.039 (2)	0.044 (3)	0.045 (3)	0.005 (2)	0.010 (2)	0.001 (2)
C30	0.037 (2)	0.048 (3)	0.055 (3)	0.000 (2)	0.014 (2)	0.000 (2)
C31	0.063 (3)	0.048 (3)	0.075 (3)	0.011 (2)	0.017 (3)	0.012 (3)
C32	0.070 (3)	0.072 (4)	0.058 (3)	0.019 (3)	−0.003 (3)	0.005 (3)
C33	0.063 (3)	0.075 (4)	0.060 (3)	0.005 (3)	−0.005 (3)	−0.012 (3)
C34	0.046 (3)	0.049 (3)	0.056 (3)	0.005 (2)	0.002 (2)	−0.006 (2)
C35	0.072 (3)	0.050 (3)	0.069 (3)	−0.004 (3)	0.008 (3)	−0.003 (2)
C36	0.078 (4)	0.061 (3)	0.081 (4)	−0.004 (3)	−0.001 (3)	−0.018 (3)
C37	0.037 (2)	0.052 (3)	0.039 (2)	0.004 (2)	0.0047 (19)	−0.006 (2)
C38	0.035 (2)	0.040 (3)	0.066 (3)	−0.002 (2)	−0.005 (2)	−0.004 (2)
C39	0.040 (3)	0.061 (3)	0.087 (4)	−0.007 (2)	0.009 (3)	−0.032 (3)
C40	0.067 (4)	0.079 (4)	0.125 (5)	−0.012 (3)	0.018 (4)	−0.054 (4)
C41	0.053 (3)	0.046 (4)	0.180 (7)	−0.010 (3)	0.003 (4)	−0.030 (4)
C42	0.059 (4)	0.051 (4)	0.140 (6)	0.002 (3)	−0.001 (4)	0.011 (4)
C43	0.043 (3)	0.051 (3)	0.081 (4)	0.000 (2)	−0.010 (2)	0.003 (3)
C44	0.089 (4)	0.107 (5)	0.067 (4)	−0.016 (4)	0.027 (3)	−0.021 (3)
C45	0.080 (4)	0.090 (4)	0.073 (4)	−0.012 (3)	−0.001 (3)	0.028 (3)
Fe2	0.0358 (3)	0.0380 (3)	0.0397 (3)	0.0001 (3)	0.0031 (2)	−0.0019 (3)
Fe3	0.0334 (3)	0.0406 (3)	0.0358 (3)	−0.0028 (3)	0.0032 (2)	−0.0029 (3)
N6	0.041 (2)	0.049 (2)	0.048 (2)	−0.0037 (18)	0.0006 (17)	−0.0008 (17)
N7	0.054 (2)	0.044 (2)	0.048 (2)	0.0021 (18)	0.0078 (19)	−0.0066 (18)
N8	0.049 (2)	0.045 (2)	0.059 (2)	0.0017 (18)	0.0007 (18)	0.0026 (19)
N9	0.042 (2)	0.048 (2)	0.043 (2)	0.0035 (17)	0.0003 (17)	−0.0042 (17)
N10	0.044 (2)	0.039 (2)	0.041 (2)	−0.0024 (16)	0.0093 (16)	0.0019 (17)
N11	0.043 (2)	0.036 (2)	0.045 (2)	−0.0060 (17)	0.0034 (16)	−0.0077 (16)
N12	0.038 (2)	0.045 (2)	0.047 (2)	0.0027 (18)	0.0098 (16)	−0.0014 (18)

N13	0.0319 (19)	0.045 (2)	0.047 (2)	0.0000 (16)	−0.0003 (17)	0.0031 (16)
N14	0.039 (2)	0.056 (2)	0.041 (2)	−0.0039 (18)	0.0067 (17)	−0.0004 (17)
C46	0.043 (3)	0.040 (3)	0.040 (2)	0.003 (2)	0.007 (2)	0.0040 (19)
C47	0.033 (2)	0.044 (3)	0.065 (3)	0.000 (2)	−0.004 (2)	−0.011 (2)
C48	0.041 (2)	0.052 (3)	0.054 (3)	0.010 (2)	−0.001 (2)	−0.010 (2)
C49	0.047 (3)	0.066 (3)	0.073 (3)	0.007 (3)	−0.009 (3)	−0.018 (3)
C50	0.050 (3)	0.066 (4)	0.107 (5)	−0.001 (3)	−0.018 (3)	−0.030 (3)
C51	0.050 (3)	0.048 (3)	0.135 (6)	−0.008 (2)	0.003 (3)	−0.018 (3)
C52	0.049 (3)	0.042 (3)	0.087 (4)	−0.004 (2)	0.007 (3)	−0.003 (3)
C53	0.051 (3)	0.076 (3)	0.049 (3)	0.009 (3)	−0.003 (2)	0.007 (2)
C54	0.072 (4)	0.078 (4)	0.110 (5)	−0.016 (3)	0.016 (3)	0.033 (3)
C55	0.038 (2)	0.046 (3)	0.046 (3)	0.001 (2)	0.003 (2)	−0.001 (2)
C56	0.056 (3)	0.059 (3)	0.043 (3)	0.020 (2)	0.006 (2)	−0.006 (2)
C57	0.053 (3)	0.107 (4)	0.053 (3)	0.034 (3)	0.020 (3)	0.027 (3)
C58	0.072 (4)	0.149 (6)	0.072 (4)	0.045 (4)	0.021 (4)	0.036 (4)
C59	0.108 (6)	0.129 (7)	0.075 (5)	0.072 (5)	0.001 (4)	−0.001 (5)
C60	0.126 (6)	0.073 (4)	0.060 (4)	0.045 (4)	−0.029 (4)	−0.022 (3)
C61	0.095 (4)	0.056 (3)	0.069 (4)	0.022 (3)	−0.012 (3)	−0.013 (3)
C62	0.064 (4)	0.108 (5)	0.090 (4)	−0.010 (3)	−0.007 (3)	0.021 (4)
C63	0.134 (6)	0.058 (4)	0.117 (5)	−0.005 (4)	−0.029 (4)	−0.014 (3)
C64	0.037 (2)	0.047 (3)	0.043 (3)	−0.001 (2)	0.0038 (19)	−0.007 (2)
C65	0.034 (2)	0.040 (3)	0.069 (3)	0.001 (2)	−0.001 (2)	0.011 (2)
C66	0.039 (3)	0.060 (3)	0.062 (3)	−0.001 (2)	−0.003 (2)	0.020 (3)
C67	0.044 (3)	0.073 (4)	0.089 (4)	−0.008 (3)	−0.006 (3)	0.029 (3)
C68	0.052 (3)	0.059 (4)	0.114 (5)	0.006 (3)	0.000 (3)	0.031 (4)
C69	0.058 (3)	0.047 (3)	0.108 (5)	0.002 (3)	0.020 (3)	0.005 (3)
C70	0.044 (3)	0.049 (3)	0.080 (4)	0.002 (2)	0.014 (2)	0.005 (3)
C71	0.095 (4)	0.086 (4)	0.057 (3)	−0.004 (3)	−0.010 (3)	0.009 (3)
C72	0.081 (4)	0.063 (3)	0.074 (4)	−0.004 (3)	0.020 (3)	−0.015 (3)
C73	0.042 (2)	0.036 (2)	0.036 (2)	−0.0059 (19)	0.0052 (19)	−0.0001 (18)
C74	0.039 (2)	0.048 (3)	0.044 (3)	0.008 (2)	−0.003 (2)	−0.009 (2)
C75	0.049 (3)	0.057 (3)	0.042 (3)	0.008 (2)	0.001 (2)	−0.003 (2)
C76	0.068 (3)	0.071 (4)	0.049 (3)	0.004 (3)	0.008 (2)	−0.004 (3)
C77	0.079 (4)	0.078 (4)	0.054 (3)	0.015 (3)	0.009 (3)	−0.019 (3)
C78	0.066 (3)	0.055 (3)	0.058 (3)	0.008 (3)	−0.005 (3)	−0.021 (3)
C79	0.045 (3)	0.051 (3)	0.049 (3)	0.000 (2)	−0.004 (2)	−0.009 (2)
C80	0.075 (3)	0.063 (3)	0.051 (3)	0.000 (3)	0.008 (2)	0.003 (2)
C81	0.054 (3)	0.051 (3)	0.087 (4)	−0.005 (2)	0.001 (3)	−0.014 (3)
C82	0.036 (2)	0.041 (2)	0.041 (2)	−0.0072 (19)	0.0066 (18)	−0.010 (2)
C83	0.037 (2)	0.042 (3)	0.043 (3)	0.007 (2)	0.0068 (19)	0.010 (2)
C84	0.053 (3)	0.047 (3)	0.055 (3)	0.002 (2)	0.010 (2)	0.010 (2)
C85	0.054 (3)	0.068 (4)	0.085 (4)	−0.007 (3)	0.020 (3)	0.021 (3)
C86	0.060 (3)	0.097 (4)	0.063 (4)	0.008 (3)	0.028 (3)	0.026 (3)
C87	0.055 (3)	0.079 (4)	0.049 (3)	0.019 (3)	0.012 (2)	0.005 (3)
C88	0.045 (3)	0.053 (3)	0.044 (3)	0.011 (2)	0.011 (2)	0.010 (2)
C89	0.067 (3)	0.054 (3)	0.077 (4)	−0.014 (3)	0.007 (3)	−0.003 (3)
C90	0.059 (3)	0.061 (3)	0.051 (3)	0.005 (2)	0.002 (2)	−0.006 (2)
C91	0.042 (2)	0.044 (3)	0.035 (2)	0.006 (2)	−0.0009 (18)	0.006 (2)

C92	0.041 (2)	0.028 (2)	0.047 (3)	−0.0021 (18)	0.005 (2)	−0.0067 (19)
C93	0.051 (3)	0.034 (2)	0.046 (3)	−0.003 (2)	0.009 (2)	−0.0053 (19)
C94	0.051 (3)	0.055 (3)	0.056 (3)	−0.003 (2)	−0.003 (2)	−0.012 (2)
C95	0.045 (3)	0.044 (3)	0.086 (4)	−0.010 (2)	0.002 (3)	−0.011 (3)
C96	0.058 (3)	0.039 (3)	0.070 (3)	−0.007 (2)	0.017 (3)	−0.001 (2)
C97	0.050 (3)	0.034 (2)	0.054 (3)	−0.002 (2)	0.006 (2)	−0.009 (2)
C98	0.061 (3)	0.067 (3)	0.057 (3)	−0.003 (3)	0.002 (2)	−0.006 (3)
C99	0.069 (3)	0.055 (3)	0.052 (3)	−0.001 (2)	0.002 (2)	0.001 (2)
C100	0.033 (2)	0.057 (3)	0.030 (2)	0.003 (2)	0.0035 (17)	−0.001 (2)
C101	0.041 (2)	0.038 (3)	0.054 (3)	0.009 (2)	0.011 (2)	0.001 (2)
C102	0.048 (3)	0.043 (3)	0.061 (3)	0.010 (2)	0.017 (2)	0.006 (2)
C103	0.063 (3)	0.064 (3)	0.059 (3)	0.010 (3)	0.014 (2)	0.005 (3)
C104	0.075 (4)	0.050 (3)	0.087 (4)	0.018 (3)	0.025 (3)	0.013 (3)
C105	0.063 (3)	0.045 (3)	0.086 (4)	0.003 (2)	0.020 (3)	−0.013 (3)
C106	0.047 (3)	0.037 (3)	0.067 (3)	0.001 (2)	0.011 (2)	−0.005 (2)
C107	0.057 (3)	0.059 (3)	0.052 (3)	0.009 (2)	0.005 (2)	−0.004 (2)
C108	0.056 (3)	0.058 (3)	0.069 (3)	−0.002 (2)	0.003 (2)	−0.011 (2)
C109	0.040 (2)	0.032 (2)	0.037 (2)	−0.0012 (19)	0.0090 (19)	0.0014 (18)
C110	0.036 (2)	0.045 (3)	0.042 (3)	0.002 (2)	0.000 (2)	−0.0039 (19)
C111	0.037 (2)	0.054 (3)	0.045 (3)	0.007 (2)	−0.001 (2)	0.000 (2)
C112	0.047 (3)	0.081 (4)	0.045 (3)	0.004 (3)	−0.005 (2)	−0.001 (2)
C113	0.041 (3)	0.083 (4)	0.063 (3)	0.001 (3)	−0.011 (2)	−0.002 (3)
C114	0.039 (3)	0.061 (3)	0.065 (3)	−0.004 (2)	0.004 (2)	−0.002 (3)
C115	0.041 (2)	0.038 (2)	0.049 (3)	0.004 (2)	0.006 (2)	−0.003 (2)
C116	0.058 (3)	0.096 (4)	0.053 (3)	−0.017 (3)	0.007 (2)	0.008 (3)
C117	0.057 (3)	0.051 (3)	0.050 (3)	0.000 (2)	0.013 (2)	0.005 (2)
C118	0.032 (2)	0.034 (2)	0.041 (2)	−0.0016 (18)	−0.0011 (19)	0.0041 (18)
C119	0.038 (2)	0.063 (3)	0.032 (2)	0.008 (2)	0.0093 (18)	0.002 (2)
C120	0.040 (2)	0.071 (3)	0.038 (2)	−0.003 (2)	0.007 (2)	0.000 (2)
C121	0.050 (3)	0.082 (4)	0.054 (3)	−0.001 (3)	0.017 (2)	0.009 (3)
C122	0.058 (3)	0.088 (4)	0.058 (3)	0.018 (3)	0.028 (3)	0.011 (3)
C123	0.062 (3)	0.066 (3)	0.050 (3)	0.012 (3)	0.016 (2)	0.004 (2)
C124	0.043 (3)	0.060 (3)	0.039 (2)	0.009 (2)	0.007 (2)	0.010 (2)
C125	0.066 (3)	0.064 (3)	0.061 (3)	−0.015 (3)	0.010 (2)	−0.001 (3)
C126	0.061 (3)	0.059 (3)	0.054 (3)	0.006 (2)	0.013 (2)	0.002 (2)

Geometric parameters (Å, º)

Fe1—C28	1.805 (4)	C53—H53A	0.9800
Fe1—C1	1.816 (4)	C53—H53B	0.9800
Fe1—C19	1.819 (4)	C53—H53C	0.9800
Fe1—C37	1.821 (5)	C54—H54A	0.9800
Fe1—C10	1.829 (4)	C54—H54B	0.9800
N1—C1	1.196 (5)	C54—H54C	0.9800
N1—C2	1.396 (5)	C56—C61	1.376 (6)
N2—C10	1.183 (5)	C56—C57	1.397 (6)
N2—C11	1.414 (5)	C57—C58	1.377 (7)
N3—C19	1.177 (5)	C57—C62	1.505 (7)

N3—C20	1.391 (5)	C58—C59	1.327 (9)
N4—C28	1.192 (5)	C58—H58A	0.9500
N4—C29	1.404 (5)	C59—C60	1.353 (9)
N5—C37	1.178 (5)	C59—H59A	0.9500
N5—C38	1.406 (5)	C60—C61	1.462 (8)
C2—C7	1.380 (6)	C60—H60A	0.9500
C2—C3	1.398 (6)	C61—C63	1.471 (8)
C3—C4	1.391 (6)	C62—H62A	0.9800
C3—C8	1.506 (6)	C62—H62B	0.9800
C4—C5	1.361 (7)	C62—H62C	0.9800
C4—H4A	0.9500	C63—H63A	0.9800
C5—C6	1.364 (7)	C63—H63B	0.9800
C5—H5A	0.9500	C63—H63C	0.9800
C6—C7	1.384 (7)	C65—C66	1.390 (6)
C6—H6A	0.9500	C65—C70	1.397 (6)
C7—C9	1.514 (6)	C66—C67	1.395 (6)
C8—H8A	0.9800	C66—C71	1.502 (6)
C8—H8B	0.9800	C67—C68	1.369 (7)
C8—H8C	0.9800	C67—H67A	0.9500
C9—H9A	0.9800	C68—C69	1.367 (7)
C9—H9B	0.9800	C68—H68A	0.9500
C9—H9C	0.9800	C69—C70	1.403 (6)
C11—C16	1.383 (6)	C69—H69A	0.9500
C11—C12	1.404 (6)	C70—C72	1.509 (6)
C12—C13	1.386 (6)	C71—H71A	0.9800
C12—C17	1.499 (6)	C71—H71B	0.9800
C13—C14	1.379 (8)	C71—H71C	0.9800
C13—H13A	0.9500	C72—H72A	0.9800
C14—C15	1.368 (7)	C72—H72B	0.9800
C14—H14A	0.9500	C72—H72C	0.9800
C15—C16	1.405 (6)	C74—C75	1.400 (5)
C15—H15A	0.9500	C74—C79	1.403 (5)
C16—C18	1.512 (6)	C75—C76	1.389 (6)
C17—H17A	0.9800	C75—C80	1.499 (6)
C17—H17B	0.9800	C76—C77	1.374 (6)
C17—H17C	0.9800	C76—H76A	0.9500
C18—H18A	0.9800	C77—C78	1.378 (6)
C18—H18B	0.9800	C77—H77A	0.9500
C18—H18C	0.9800	C78—C79	1.394 (6)
C20—C21	1.393 (6)	C78—H78A	0.9500
C20—C25	1.413 (6)	C79—C81	1.513 (6)
C21—C22	1.395 (7)	C80—H80A	0.9800
C21—C26	1.470 (7)	C80—H80B	0.9800
C22—C23	1.396 (8)	C80—H80C	0.9800
C22—H22A	0.9500	C81—H81A	0.9800
C23—C24	1.346 (8)	C81—H81B	0.9800
C23—H23A	0.9500	C81—H81C	0.9800
C24—C25	1.378 (7)	C83—C84	1.395 (5)

C24—H24A	0.9500	C83—C88	1.396 (5)
C25—C27	1.496 (7)	C84—C85	1.394 (6)
C26—H26A	0.9800	C84—C89	1.516 (6)
C26—H26B	0.9800	C85—C86	1.377 (7)
C26—H26C	0.9800	C85—H85A	0.9500
C27—H27A	0.9800	C86—C87	1.379 (6)
C27—H27B	0.9800	C86—H86A	0.9500
C27—H27C	0.9800	C87—C88	1.390 (6)
C29—C34	1.394 (5)	C87—H87A	0.9500
C29—C30	1.407 (5)	C88—C90	1.509 (6)
C30—C31	1.397 (6)	C89—H89A	0.9800
C30—C35	1.499 (6)	C89—H89B	0.9800
C31—C32	1.383 (6)	C89—H89C	0.9800
C31—H31A	0.9500	C90—H90A	0.9800
C32—C33	1.364 (6)	C90—H90B	0.9800
C32—H32A	0.9500	C90—H90C	0.9800
C33—C34	1.384 (6)	C92—C93	1.402 (5)
C33—H33A	0.9500	C92—C97	1.403 (5)
C34—C36	1.497 (6)	C93—C94	1.390 (5)
C35—H35A	0.9800	C93—C98	1.506 (6)
C35—H35B	0.9800	C94—C95	1.385 (6)
C35—H35C	0.9800	C94—H94A	0.9500
C36—H36A	0.9800	C95—C96	1.368 (6)
C36—H36B	0.9800	C95—H95A	0.9500
C36—H36C	0.9800	C96—C97	1.397 (6)
C38—C43	1.378 (6)	C96—H96A	0.9500
C38—C39	1.410 (6)	C97—C99	1.505 (5)
C39—C40	1.396 (7)	C98—H98A	0.9800
C39—C44	1.490 (7)	C98—H98B	0.9800
C40—C41	1.385 (8)	C98—H98C	0.9800
C40—H40A	0.9500	C99—H99A	0.9800
C41—C42	1.373 (8)	C99—H99B	0.9800
C41—H41A	0.9500	C99—H99C	0.9800
C42—C43	1.396 (7)	C101—C106	1.398 (5)
C42—H42A	0.9500	C101—C102	1.406 (6)
C43—C45	1.511 (7)	C102—C103	1.391 (6)
C44—H44A	0.9800	C102—C107	1.500 (6)
C44—H44B	0.9800	C103—C104	1.382 (6)
C44—H44C	0.9800	C103—H10I	0.9500
C45—H45A	0.9800	C104—C105	1.386 (6)
C45—H45B	0.9800	C104—H10J	0.9500
C45—H45C	0.9800	C105—C106	1.403 (6)
Fe2—C64	1.832 (5)	C105—H10K	0.9500
Fe2—C55	1.840 (4)	C106—C108	1.489 (6)
Fe2—C46	1.852 (4)	C107—H10A	0.9800
Fe2—C91	1.967 (4)	C107—H10B	0.9800
Fe2—C73	1.974 (4)	C107—H10C	0.9800
Fe2—C82	1.974 (4)	C108—H10D	0.9800

Fe2—Fe3	2.4764 (8)	C108—H10E	0.9800
Fe3—C100	1.838 (5)	C108—H10F	0.9800
Fe3—C109	1.856 (4)	C110—C115	1.392 (5)
Fe3—C118	1.860 (4)	C110—C111	1.399 (5)
Fe3—C73	1.962 (4)	C111—C112	1.374 (5)
Fe3—C82	1.978 (4)	C111—C116	1.514 (6)
Fe3—C91	2.022 (4)	C112—C113	1.381 (6)
N6—C46	1.180 (5)	C112—H11G	0.9500
N6—C47	1.409 (5)	C113—C114	1.380 (6)
N7—C55	1.178 (5)	C113—H11H	0.9500
N7—C56	1.410 (5)	C114—C115	1.383 (5)
N8—C64	1.178 (5)	C114—H11I	0.9500
N8—C65	1.404 (5)	C115—C117	1.507 (5)
N9—C73	1.245 (4)	C116—H11A	0.9800
N9—C74	1.418 (5)	C116—H11B	0.9800
N10—C82	1.237 (4)	C116—H11C	0.9800
N10—C83	1.410 (5)	C117—H11D	0.9800
N11—C91	1.235 (4)	C117—H11E	0.9800
N11—C92	1.412 (5)	C117—H11F	0.9800
N12—C100	1.190 (5)	C119—C124	1.399 (6)
N12—C101	1.418 (5)	C119—C120	1.403 (6)
N13—C109	1.176 (4)	C120—C121	1.394 (6)
N13—C110	1.398 (5)	C120—C125	1.488 (6)
N14—C118	1.179 (4)	C121—C122	1.383 (6)
N14—C119	1.405 (5)	C121—H12G	0.9500
C47—C52	1.397 (6)	C122—C123	1.383 (6)
C47—C48	1.404 (6)	C122—H12H	0.9500
C48—C49	1.391 (6)	C123—C124	1.395 (5)
C48—C53	1.497 (6)	C123—H12I	0.9500
C49—C50	1.372 (7)	C124—C126	1.500 (6)
C49—H49A	0.9500	C125—H12A	0.9800
C50—C51	1.362 (7)	C125—H12B	0.9800
C50—H50A	0.9500	C125—H12C	0.9800
C51—C52	1.394 (6)	C126—H12D	0.9800
C51—H51A	0.9500	C126—H12E	0.9800
C52—C54	1.496 (7)	C126—H12F	0.9800

C28—Fe1—C1	128.78 (18)	C61—C56—C57	123.7 (5)
C28—Fe1—C19	88.02 (17)	C61—C56—N7	118.6 (5)
C1—Fe1—C19	90.78 (17)	C57—C56—N7	117.7 (4)
C28—Fe1—C37	85.38 (17)	C58—C57—C56	117.4 (6)
C1—Fe1—C37	92.23 (17)	C58—C57—C62	121.9 (6)
C19—Fe1—C37	173.24 (17)	C56—C57—C62	120.7 (4)
C28—Fe1—C10	119.54 (18)	C59—C58—C57	120.1 (7)
C1—Fe1—C10	111.66 (17)	C59—C58—H58A	120.0
C19—Fe1—C10	90.29 (17)	C57—C58—H58A	120.0
C37—Fe1—C10	94.22 (17)	C58—C59—C60	125.5 (7)
C1—N1—C2	162.9 (4)	C58—C59—H59A	117.2

C10—N2—C11	166.4 (4)	C60—C59—H59A	117.2
C19—N3—C20	178.7 (4)	C59—C60—C61	116.8 (6)
C28—N4—C29	158.6 (4)	C59—C60—H60A	121.6
C37—N5—C38	164.6 (4)	C61—C60—H60A	121.6
N1—C1—Fe1	175.5 (4)	C56—C61—C60	116.4 (6)
C7—C2—N1	118.1 (4)	C56—C61—C63	121.1 (5)
C7—C2—C3	122.8 (4)	C60—C61—C63	122.5 (5)
N1—C2—C3	119.1 (4)	C57—C62—H62A	109.5
C4—C3—C2	116.8 (5)	C57—C62—H62B	109.5
C4—C3—C8	120.6 (5)	H62A—C62—H62B	109.5
C2—C3—C8	122.6 (4)	C57—C62—H62C	109.5
C5—C4—C3	120.8 (5)	H62A—C62—H62C	109.5
C5—C4—H4A	119.6	H62B—C62—H62C	109.5
C3—C4—H4A	119.6	C61—C63—H63A	109.5
C4—C5—C6	121.2 (5)	C61—C63—H63B	109.5
C4—C5—H5A	119.4	H63A—C63—H63B	109.5
C6—C5—H5A	119.4	C61—C63—H63C	109.5
C5—C6—C7	120.6 (5)	H63A—C63—H63C	109.5
C5—C6—H6A	119.7	H63B—C63—H63C	109.5
C7—C6—H6A	119.7	N8—C64—Fe2	178.6 (4)
C2—C7—C6	117.7 (5)	C66—C65—C70	123.5 (4)
C2—C7—C9	120.9 (4)	C66—C65—N8	119.1 (4)
C6—C7—C9	121.3 (5)	C70—C65—N8	117.4 (4)
C3—C8—H8A	109.5	C65—C66—C67	117.0 (5)
C3—C8—H8B	109.5	C65—C66—C71	122.1 (4)
H8A—C8—H8B	109.5	C67—C66—C71	120.9 (4)
C3—C8—H8C	109.5	C68—C67—C66	120.6 (5)
H8A—C8—H8C	109.5	C68—C67—H67A	119.7
H8B—C8—H8C	109.5	C66—C67—H67A	119.7
C7—C9—H9A	109.5	C69—C68—C67	121.8 (5)
C7—C9—H9B	109.5	C69—C68—H68A	119.1
H9A—C9—H9B	109.5	C67—C68—H68A	119.1
C7—C9—H9C	109.5	C68—C69—C70	120.1 (5)
H9A—C9—H9C	109.5	C68—C69—H69A	119.9
H9B—C9—H9C	109.5	C70—C69—H69A	119.9
N2—C10—Fe1	176.5 (4)	C65—C70—C69	117.0 (5)
C16—C11—C12	124.1 (4)	C65—C70—C72	120.7 (4)
C16—C11—N2	118.3 (4)	C69—C70—C72	122.3 (5)
C12—C11—N2	117.6 (4)	C66—C71—H71A	109.5
C13—C12—C11	116.7 (5)	C66—C71—H71B	109.5
C13—C12—C17	121.4 (5)	H71A—C71—H71B	109.5
C11—C12—C17	121.9 (4)	C66—C71—H71C	109.5
C14—C13—C12	121.0 (5)	H71A—C71—H71C	109.5
C14—C13—H13A	119.5	H71B—C71—H71C	109.5
C12—C13—H13A	119.5	C70—C72—H72A	109.5
C15—C14—C13	120.7 (5)	C70—C72—H72B	109.5
C15—C14—H14A	119.7	H72A—C72—H72B	109.5
C13—C14—H14A	119.7	C70—C72—H72C	109.5

C14—C15—C16	121.3 (5)	H72A—C72—H72C	109.5
C14—C15—H15A	119.3	H72B—C72—H72C	109.5
C16—C15—H15A	119.3	N9—C73—Fe3	147.8 (3)
C11—C16—C15	116.2 (5)	N9—C73—Fe2	134.2 (3)
C11—C16—C18	121.3 (4)	Fe3—C73—Fe2	78.00 (14)
C15—C16—C18	122.5 (5)	C75—C74—C79	122.1 (4)
C12—C17—H17A	109.5	C75—C74—N9	120.4 (4)
C12—C17—H17B	109.5	C79—C74—N9	117.4 (4)
H17A—C17—H17B	109.5	C76—C75—C74	117.4 (4)
C12—C17—H17C	109.5	C76—C75—C80	120.2 (4)
H17A—C17—H17C	109.5	C74—C75—C80	122.4 (4)
H17B—C17—H17C	109.5	C77—C76—C75	121.9 (5)
C16—C18—H18A	109.5	C77—C76—H76A	119.0
C16—C18—H18B	109.5	C75—C76—H76A	119.0
H18A—C18—H18B	109.5	C76—C77—C78	119.6 (5)
C16—C18—H18C	109.5	C76—C77—H77A	120.2
H18A—C18—H18C	109.5	C78—C77—H77A	120.2
H18B—C18—H18C	109.5	C77—C78—C79	121.4 (5)
N3—C19—Fe1	178.4 (4)	C77—C78—H78A	119.3
N3—C20—C21	119.7 (4)	C79—C78—H78A	119.3
N3—C20—C25	117.4 (4)	C78—C79—C74	117.4 (4)
C21—C20—C25	122.9 (4)	C78—C79—C81	123.1 (4)
C20—C21—C22	117.9 (5)	C74—C79—C81	119.5 (4)
C20—C21—C26	121.3 (5)	C75—C80—H80A	109.5
C22—C21—C26	120.8 (5)	C75—C80—H80B	109.5
C21—C22—C23	119.4 (5)	H80A—C80—H80B	109.5
C21—C22—H22A	120.3	C75—C80—H80C	109.5
C23—C22—H22A	120.3	H80A—C80—H80C	109.5
C24—C23—C22	121.0 (6)	H80B—C80—H80C	109.5
C24—C23—H23A	119.5	C79—C81—H81A	109.5
C22—C23—H23A	119.5	C79—C81—H81B	109.5
C23—C24—C25	122.9 (6)	H81A—C81—H81B	109.5
C23—C24—H24A	118.6	C79—C81—H81C	109.5
C25—C24—H24A	118.6	H81A—C81—H81C	109.5
C24—C25—C20	116.0 (5)	H81B—C81—H81C	109.5
C24—C25—C27	122.7 (5)	N10—C82—Fe2	146.6 (3)
C20—C25—C27	121.3 (4)	N10—C82—Fe3	135.8 (3)
C21—C26—H26A	109.5	Fe2—C82—Fe3	77.60 (15)
C21—C26—H26B	109.5	C84—C83—C88	121.3 (4)
H26A—C26—H26B	109.5	C84—C83—N10	118.0 (4)
C21—C26—H26C	109.5	C88—C83—N10	120.4 (4)
H26A—C26—H26C	109.5	C85—C84—C83	118.3 (4)
H26B—C26—H26C	109.5	C85—C84—C89	121.5 (4)
C25—C27—H27A	109.5	C83—C84—C89	120.3 (4)
C25—C27—H27B	109.5	C86—C85—C84	121.4 (5)
H27A—C27—H27B	109.5	C86—C85—H85A	119.3
C25—C27—H27C	109.5	C84—C85—H85A	119.3
H27A—C27—H27C	109.5	C85—C86—C87	119.0 (5)

H27B—C27—H27C	109.5	C85—C86—H86A	120.5
N4—C28—Fe1	176.8 (4)	C87—C86—H86A	120.5
C34—C29—N4	119.4 (4)	C86—C87—C88	121.9 (5)
C34—C29—C30	122.6 (4)	C86—C87—H87A	119.0
N4—C29—C30	117.9 (4)	C88—C87—H87A	119.0
C31—C30—C29	116.2 (4)	C87—C88—C83	118.0 (4)
C31—C30—C35	123.2 (4)	C87—C88—C90	120.4 (4)
C29—C30—C35	120.6 (4)	C83—C88—C90	121.5 (4)
C32—C31—C30	121.5 (4)	C84—C89—H89A	109.5
C32—C31—H31A	119.3	C84—C89—H89B	109.5
C30—C31—H31A	119.3	H89A—C89—H89B	109.5
C33—C32—C31	120.5 (5)	C84—C89—H89C	109.5
C33—C32—H32A	119.8	H89A—C89—H89C	109.5
C31—C32—H32A	119.8	H89B—C89—H89C	109.5
C32—C33—C34	121.0 (4)	C88—C90—H90A	109.5
C32—C33—H33A	119.5	C88—C90—H90B	109.5
C34—C33—H33A	119.5	H90A—C90—H90B	109.5
C33—C34—C29	118.0 (4)	C88—C90—H90C	109.5
C33—C34—C36	120.6 (4)	H90A—C90—H90C	109.5
C29—C34—C36	121.3 (4)	H90B—C90—H90C	109.5
C30—C35—H35A	109.5	N11—C91—Fe2	136.7 (3)
C30—C35—H35B	109.5	N11—C91—Fe3	146.4 (3)
H35A—C35—H35B	109.5	Fe2—C91—Fe3	76.74 (15)
C30—C35—H35C	109.5	C93—C92—C97	121.7 (4)
H35A—C35—H35C	109.5	C93—C92—N11	119.3 (4)
H35B—C35—H35C	109.5	C97—C92—N11	118.7 (3)
C34—C36—H36A	109.5	C94—C93—C92	117.6 (4)
C34—C36—H36B	109.5	C94—C93—C98	120.6 (4)
H36A—C36—H36B	109.5	C92—C93—C98	121.7 (4)
C34—C36—H36C	109.5	C95—C94—C93	121.2 (4)
H36A—C36—H36C	109.5	C95—C94—H94A	119.4
H36B—C36—H36C	109.5	C93—C94—H94A	119.4
N5—C37—Fe1	174.9 (4)	C96—C95—C94	120.5 (4)
C43—C38—N5	118.4 (4)	C96—C95—H95A	119.7
C43—C38—C39	124.6 (4)	C94—C95—H95A	119.7
N5—C38—C39	117.0 (4)	C95—C96—C97	120.7 (4)
C40—C39—C38	115.1 (5)	C95—C96—H96A	119.6
C40—C39—C44	124.2 (5)	C97—C96—H96A	119.6
C38—C39—C44	120.7 (4)	C96—C97—C92	118.1 (4)
C41—C40—C39	121.5 (6)	C96—C97—C99	121.6 (4)
C41—C40—H40A	119.3	C92—C97—C99	120.3 (4)
C39—C40—H40A	119.3	C93—C98—H98A	109.5
C42—C41—C40	121.2 (5)	C93—C98—H98B	109.5
C42—C41—H41A	119.4	H98A—C98—H98B	109.5
C40—C41—H41A	119.4	C93—C98—H98C	109.5
C41—C42—C43	120.0 (6)	H98A—C98—H98C	109.5
C41—C42—H42A	120.0	H98B—C98—H98C	109.5
C43—C42—H42A	120.0	C97—C99—H99A	109.5

C38—C43—C42	117.5 (5)	C97—C99—H99B	109.5
C38—C43—C45	120.5 (4)	H99A—C99—H99B	109.5
C42—C43—C45	121.9 (5)	C97—C99—H99C	109.5
C39—C44—H44A	109.5	H99A—C99—H99C	109.5
C39—C44—H44B	109.5	H99B—C99—H99C	109.5
H44A—C44—H44B	109.5	N12—C100—Fe3	174.2 (3)
C39—C44—H44C	109.5	C106—C101—C102	123.4 (4)
H44A—C44—H44C	109.5	C106—C101—N12	119.6 (4)
H44B—C44—H44C	109.5	C102—C101—N12	117.0 (4)
C43—C45—H45A	109.5	C103—C102—C101	117.0 (4)
C43—C45—H45B	109.5	C103—C102—C107	121.6 (4)
H45A—C45—H45B	109.5	C101—C102—C107	121.4 (4)
C43—C45—H45C	109.5	C104—C103—C102	121.7 (5)
H45A—C45—H45C	109.5	C104—C103—H10I	119.1
H45B—C45—H45C	109.5	C102—C103—H10I	119.1
C64—Fe2—C55	93.53 (17)	C103—C104—C105	119.7 (5)
C64—Fe2—C46	93.81 (17)	C103—C104—H10J	120.1
C55—Fe2—C46	95.13 (17)	C105—C104—H10J	120.1
C64—Fe2—C91	85.33 (16)	C104—C105—C106	121.6 (4)
C55—Fe2—C91	93.71 (17)	C104—C105—H10K	119.2
C46—Fe2—C91	171.16 (17)	C106—C105—H10K	119.2
C64—Fe2—C73	88.00 (16)	C101—C106—C105	116.6 (4)
C55—Fe2—C73	177.63 (17)	C101—C106—C108	121.9 (4)
C46—Fe2—C73	86.57 (16)	C105—C106—C108	121.4 (4)
C91—Fe2—C73	84.61 (16)	C102—C107—H10A	109.5
C64—Fe2—C82	168.76 (17)	C102—C107—H10B	109.5
C55—Fe2—C82	90.74 (16)	H10A—C107—H10B	109.5
C46—Fe2—C82	96.16 (16)	C102—C107—H10C	109.5
C91—Fe2—C82	84.02 (15)	H10A—C107—H10C	109.5
C73—Fe2—C82	87.43 (16)	H10B—C107—H10C	109.5
C64—Fe2—Fe3	118.57 (12)	C106—C108—H10D	109.5
C55—Fe2—Fe3	126.85 (12)	C106—C108—H10E	109.5
C46—Fe2—Fe3	121.11 (12)	H10D—C108—H10E	109.5
C91—Fe2—Fe3	52.62 (12)	C106—C108—H10F	109.5
C73—Fe2—Fe3	50.78 (11)	H10D—C108—H10F	109.5
C82—Fe2—Fe3	51.27 (11)	H10E—C108—H10F	109.5
C100—Fe3—C109	93.56 (16)	N13—C109—Fe3	177.0 (3)
C100—Fe3—C118	93.66 (16)	C115—C110—N13	118.4 (3)
C109—Fe3—C118	90.28 (16)	C115—C110—C111	122.5 (4)
C100—Fe3—C73	94.61 (16)	N13—C110—C111	119.1 (4)
C109—Fe3—C73	171.46 (16)	C112—C111—C110	117.8 (4)
C118—Fe3—C73	91.68 (15)	C112—C111—C116	120.4 (4)
C100—Fe3—C82	84.51 (16)	C110—C111—C116	121.8 (4)
C109—Fe3—C82	90.63 (16)	C111—C112—C113	121.1 (4)
C118—Fe3—C82	178.00 (16)	C111—C112—H11G	119.4
C73—Fe3—C82	87.67 (16)	C113—C112—H11G	119.4
C100—Fe3—C91	166.97 (16)	C114—C113—C112	119.9 (4)
C109—Fe3—C91	87.99 (15)	C114—C113—H11H	120.1

C118—Fe3—C91	99.28 (16)	C112—C113—H11H	120.1
C73—Fe3—C91	83.49 (15)	C113—C114—C115	121.4 (4)
C82—Fe3—C91	82.53 (16)	C113—C114—H11I	119.3
C100—Fe3—Fe2	118.77 (12)	C115—C114—H11I	119.3
C109—Fe3—Fe2	122.00 (11)	C114—C115—C110	117.3 (4)
C118—Fe3—Fe2	129.54 (11)	C114—C115—C117	120.9 (4)
C73—Fe3—Fe2	51.22 (11)	C110—C115—C117	121.7 (4)
C82—Fe3—Fe2	51.14 (12)	C111—C116—H11A	109.5
C91—Fe3—Fe2	50.64 (11)	C111—C116—H11B	109.5
C46—N6—C47	164.1 (4)	H11A—C116—H11B	109.5
C55—N7—C56	165.8 (4)	C111—C116—H11C	109.5
C64—N8—C65	165.4 (4)	H11A—C116—H11C	109.5
C73—N9—C74	132.9 (3)	H11B—C116—H11C	109.5
C82—N10—C83	133.0 (3)	C115—C117—H11D	109.5
C91—N11—C92	133.7 (3)	C115—C117—H11E	109.5
C100—N12—C101	165.2 (4)	H11D—C117—H11E	109.5
C109—N13—C110	168.6 (4)	C115—C117—H11F	109.5
C118—N14—C119	162.7 (4)	H11D—C117—H11F	109.5
N6—C46—Fe2	173.1 (4)	H11E—C117—H11F	109.5
C52—C47—C48	124.0 (4)	N14—C118—Fe3	170.9 (3)
C52—C47—N6	118.2 (4)	C124—C119—C120	123.7 (4)
C48—C47—N6	117.7 (4)	C124—C119—N14	118.0 (4)
C49—C48—C47	116.2 (4)	C120—C119—N14	118.3 (4)
C49—C48—C53	122.4 (4)	C121—C120—C119	116.3 (4)
C47—C48—C53	121.3 (4)	C121—C120—C125	121.3 (4)
C50—C49—C48	121.4 (5)	C119—C120—C125	122.5 (4)
C50—C49—H49A	119.3	C122—C121—C120	121.6 (4)
C48—C49—H49A	119.3	C122—C121—H12G	119.2
C51—C50—C49	120.3 (5)	C120—C121—H12G	119.2
C51—C50—H50A	119.8	C121—C122—C123	120.5 (4)
C49—C50—H50A	119.8	C121—C122—H12H	119.8
C50—C51—C52	122.5 (5)	C123—C122—H12H	119.8
C50—C51—H51A	118.8	C122—C123—C124	120.7 (4)
C52—C51—H51A	118.8	C122—C123—H12I	119.6
C51—C52—C47	115.4 (5)	C124—C123—H12I	119.6
C51—C52—C54	123.5 (5)	C123—C124—C119	117.2 (4)
C47—C52—C54	121.0 (4)	C123—C124—C126	120.4 (4)
C48—C53—H53A	109.5	C119—C124—C126	122.3 (4)
C48—C53—H53B	109.5	C120—C125—H12A	109.5
H53A—C53—H53B	109.5	C120—C125—H12B	109.5
C48—C53—H53C	109.5	H12A—C125—H12B	109.5
H53A—C53—H53C	109.5	C120—C125—H12C	109.5
H53B—C53—H53C	109.5	H12A—C125—H12C	109.5
C52—C54—H54A	109.5	H12B—C125—H12C	109.5
C52—C54—H54B	109.5	C124—C126—H12D	109.5
H54A—C54—H54B	109.5	C124—C126—H12E	109.5
C52—C54—H54C	109.5	H12D—C126—H12E	109.5
H54A—C54—H54C	109.5	C124—C126—H12F	109.5

H54B—C54—H54C	109.5	H12D—C126—H12F	109.5
N7—C55—Fe2	174.9 (4)	H12E—C126—H12F	109.5
C1—N1—C2—C7	−0.8 (14)	C70—C65—C66—C71	−180.0 (4)
C7—C2—C3—C4	0.6 (6)	N8—C65—C66—C71	1.2 (6)
N1—C2—C3—C4	−178.7 (4)	C65—C66—C67—C68	0.2 (7)
C7—C2—C3—C8	−178.2 (4)	C71—C66—C67—C68	179.8 (4)
N1—C2—C3—C8	2.5 (6)	C66—C67—C68—C69	−0.4 (8)
C2—C3—C4—C5	0.0 (7)	C67—C68—C69—C70	0.7 (8)
C8—C3—C4—C5	178.8 (5)	C66—C65—C70—C69	0.7 (6)
C3—C4—C5—C6	−0.9 (9)	N8—C65—C70—C69	179.5 (4)
C4—C5—C6—C7	1.3 (9)	C66—C65—C70—C72	−179.6 (4)
N1—C2—C7—C6	179.1 (4)	N8—C65—C70—C72	−0.8 (6)
C3—C2—C7—C6	−0.2 (6)	C68—C69—C70—C65	−0.8 (7)
N1—C2—C7—C9	−3.7 (6)	C68—C69—C70—C72	179.5 (4)
C3—C2—C7—C9	177.0 (4)	C74—N9—C73—Fe3	9.4 (9)
C5—C6—C7—C2	−0.8 (7)	C74—N9—C73—Fe2	−172.9 (3)
C5—C6—C7—C9	−178.0 (5)	C73—N9—C74—C75	−90.7 (5)
C10—N2—C11—C16	171.8 (16)	C73—N9—C74—C79	93.8 (5)
C10—N2—C11—C12	−8.0 (19)	C79—C74—C75—C76	−4.5 (6)
C16—C11—C12—C13	0.0 (7)	N9—C74—C75—C76	−179.7 (4)
N2—C11—C12—C13	179.7 (4)	C79—C74—C75—C80	172.5 (4)
C16—C11—C12—C17	−179.6 (4)	N9—C74—C75—C80	−2.7 (6)
N2—C11—C12—C17	0.2 (6)	C74—C75—C76—C77	0.5 (6)
C11—C12—C13—C14	0.5 (8)	C80—C75—C76—C77	−176.6 (4)
C17—C12—C13—C14	−179.9 (5)	C75—C76—C77—C78	2.8 (7)
C12—C13—C14—C15	−0.7 (9)	C76—C77—C78—C79	−2.1 (7)
C13—C14—C15—C16	0.3 (9)	C77—C78—C79—C74	−1.7 (6)
C12—C11—C16—C15	−0.4 (7)	C77—C78—C79—C81	176.6 (4)
N2—C11—C16—C15	179.9 (4)	C75—C74—C79—C78	5.1 (6)
C12—C11—C16—C18	178.6 (4)	N9—C74—C79—C78	−179.6 (3)
N2—C11—C16—C18	−1.1 (6)	C75—C74—C79—C81	−173.3 (4)
C14—C15—C16—C11	0.2 (7)	N9—C74—C79—C81	2.1 (5)
C14—C15—C16—C18	−178.8 (5)	C83—N10—C82—Fe2	4.3 (8)
N3—C20—C21—C22	180.0 (4)	C83—N10—C82—Fe3	−179.7 (3)
C25—C20—C21—C22	0.9 (6)	C82—N10—C83—C84	96.4 (5)
N3—C20—C21—C26	0.4 (6)	C82—N10—C83—C88	−90.3 (5)
C25—C20—C21—C26	−178.6 (4)	C88—C83—C84—C85	3.0 (6)
C20—C21—C22—C23	−0.5 (7)	N10—C83—C84—C85	176.3 (4)
C26—C21—C22—C23	179.0 (5)	C88—C83—C84—C89	−175.4 (4)
C21—C22—C23—C24	−0.3 (8)	N10—C83—C84—C89	−2.1 (6)
C22—C23—C24—C25	0.7 (9)	C83—C84—C85—C86	−1.8 (7)
C23—C24—C25—C20	−0.4 (8)	C89—C84—C85—C86	176.7 (4)
C23—C24—C25—C27	177.9 (5)	C84—C85—C86—C87	−0.4 (7)
N3—C20—C25—C24	−179.6 (4)	C85—C86—C87—C88	1.4 (7)
C21—C20—C25—C24	−0.5 (6)	C86—C87—C88—C83	−0.2 (6)
N3—C20—C25—C27	2.2 (6)	C86—C87—C88—C90	−176.7 (4)
C21—C20—C25—C27	−178.7 (4)	C84—C83—C88—C87	−2.1 (6)

C28—N4—C29—C34	75.3 (11)	N10—C83—C88—C87	−175.2 (3)
C28—N4—C29—C30	−105.9 (11)	C84—C83—C88—C90	174.4 (4)
C34—C29—C30—C31	−3.6 (6)	N10—C83—C88—C90	1.3 (6)
N4—C29—C30—C31	177.6 (4)	C92—N11—C91—Fe2	−176.3 (3)
C34—C29—C30—C35	175.9 (4)	C92—N11—C91—Fe3	9.0 (9)
N4—C29—C30—C35	−2.8 (6)	C91—N11—C92—C93	−99.7 (5)
C29—C30—C31—C32	0.9 (6)	C91—N11—C92—C97	86.2 (5)
C35—C30—C31—C32	−178.7 (4)	C97—C92—C93—C94	−3.2 (6)
C30—C31—C32—C33	1.6 (7)	N11—C92—C93—C94	−177.1 (3)
C31—C32—C33—C34	−1.3 (7)	C97—C92—C93—C98	174.6 (4)
C32—C33—C34—C29	−1.3 (7)	N11—C92—C93—C98	0.7 (6)
C32—C33—C34—C36	178.8 (4)	C92—C93—C94—C95	0.6 (6)
N4—C29—C34—C33	−177.4 (4)	C98—C93—C94—C95	−177.2 (4)
C30—C29—C34—C33	3.9 (6)	C93—C94—C95—C96	2.0 (7)
N4—C29—C34—C36	2.4 (6)	C94—C95—C96—C97	−2.0 (6)
C30—C29—C34—C36	−176.3 (4)	C95—C96—C97—C92	−0.5 (6)
C37—N5—C38—C43	−122.1 (15)	C95—C96—C97—C99	178.3 (4)
C37—N5—C38—C39	58.0 (16)	C93—C92—C97—C96	3.2 (6)
C43—C38—C39—C40	3.0 (6)	N11—C92—C97—C96	177.1 (3)
N5—C38—C39—C40	−177.1 (4)	C93—C92—C97—C99	−175.6 (4)
C43—C38—C39—C44	−175.3 (4)	N11—C92—C97—C99	−1.7 (5)
N5—C38—C39—C44	4.6 (6)	C100—N12—C101—C102	−1.1 (16)
C38—C39—C40—C41	−1.3 (7)	C106—C101—C102—C103	−1.0 (6)
C44—C39—C40—C41	177.0 (5)	N12—C101—C102—C103	179.8 (4)
C39—C40—C41—C42	−0.3 (9)	C106—C101—C102—C107	176.7 (4)
C40—C41—C42—C43	0.5 (8)	N12—C101—C102—C107	−2.5 (6)
N5—C38—C43—C42	177.1 (4)	C101—C102—C103—C104	−0.5 (7)
C39—C38—C43—C42	−2.9 (6)	C107—C102—C103—C104	−178.2 (4)
N5—C38—C43—C45	−3.9 (6)	C102—C103—C104—C105	1.3 (7)
C39—C38—C43—C45	176.0 (4)	C103—C104—C105—C106	−0.6 (7)
C41—C42—C43—C38	1.1 (7)	C102—C101—C106—C105	1.7 (6)
C41—C42—C43—C45	−177.8 (5)	N12—C101—C106—C105	−179.2 (4)
C46—N6—C47—C52	174.3 (13)	C102—C101—C106—C108	−176.8 (4)
C46—N6—C47—C48	−7.5 (16)	N12—C101—C106—C108	2.3 (6)
C52—C47—C48—C49	−2.6 (6)	C104—C105—C106—C101	−0.9 (7)
N6—C47—C48—C49	179.2 (4)	C104—C105—C106—C108	177.7 (4)
C52—C47—C48—C53	174.7 (4)	C109—N13—C110—C115	−8 (2)
N6—C47—C48—C53	−3.5 (6)	C109—N13—C110—C111	171.1 (18)
C47—C48—C49—C50	−0.8 (6)	C115—C110—C111—C112	−0.9 (6)
C53—C48—C49—C50	−178.1 (4)	N13—C110—C111—C112	179.9 (4)
C48—C49—C50—C51	3.0 (7)	C115—C110—C111—C116	178.7 (4)
C49—C50—C51—C52	−1.9 (8)	N13—C110—C111—C116	−0.5 (6)
C50—C51—C52—C47	−1.2 (7)	C110—C111—C112—C113	1.3 (7)
C50—C51—C52—C54	177.4 (5)	C116—C111—C112—C113	−178.3 (4)
C48—C47—C52—C51	3.6 (6)	C111—C112—C113—C114	−1.2 (7)
N6—C47—C52—C51	−178.3 (4)	C112—C113—C114—C115	0.7 (7)
C48—C47—C52—C54	−175.1 (4)	C113—C114—C115—C110	−0.3 (6)
N6—C47—C52—C54	3.1 (6)	C113—C114—C115—C117	−177.4 (4)

C55—N7—C56—C61	148.0 (15)	N13—C110—C115—C114	179.6 (4)
C55—N7—C56—C57	−30.7 (18)	C111—C110—C115—C114	0.4 (6)
C61—C56—C57—C58	−2.0 (7)	N13—C110—C115—C117	−3.3 (6)
N7—C56—C57—C58	176.6 (4)	C111—C110—C115—C117	177.5 (4)
C61—C56—C57—C62	179.6 (4)	C118—N14—C119—C124	24.1 (14)
N7—C56—C57—C62	−1.8 (6)	C118—N14—C119—C120	−155.0 (11)
C56—C57—C58—C59	0.3 (8)	C124—C119—C120—C121	−2.3 (6)
C62—C57—C58—C59	178.7 (5)	N14—C119—C120—C121	176.9 (4)
C57—C58—C59—C60	2.3 (10)	C124—C119—C120—C125	176.7 (4)
C58—C59—C60—C61	−2.9 (9)	N14—C119—C120—C125	−4.2 (6)
C57—C56—C61—C60	1.2 (7)	C119—C120—C121—C122	2.3 (6)
N7—C56—C61—C60	−177.4 (4)	C125—C120—C121—C122	−176.7 (4)
C57—C56—C61—C63	−179.4 (5)	C120—C121—C122—C123	−1.3 (7)
N7—C56—C61—C63	2.0 (7)	C121—C122—C123—C124	0.1 (7)
C59—C60—C61—C56	1.1 (7)	C122—C123—C124—C119	0.0 (6)
C59—C60—C61—C63	−178.2 (5)	C122—C123—C124—C126	−176.3 (4)
C64—N8—C65—C66	175.5 (15)	C120—C119—C124—C123	1.1 (6)
C64—N8—C65—C70	−3.4 (18)	N14—C119—C124—C123	−178.0 (3)
C70—C65—C66—C67	−0.4 (6)	C120—C119—C124—C126	177.4 (4)
N8—C65—C66—C67	−179.2 (4)	N14—C119—C124—C126	−1.7 (6)

trans-Dibromidotetrakis(2,6-dimethylphenylisocyanide)cobalt(II) (4)

Crystal data

$[Co(C_9H_9N)_4]$
M_r = 743.44
Orthorhombic, *Pbca*
a = 14.5902 (14) Å
b = 14.0206 (13) Å
c = 16.2747 (15) Å
V = 3329.2 (5) Å^3
Z = 4
F(000) = 1508
D_x = 1.483 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 3950 reflections
θ = 2.4–27.5°
μ = 2.95 mm^{-1}
T = 173 K
Block, green
0.25 × 0.15 × 0.10 mm

Data collection

Siemens SMART CCD platform diffractometer
Radiation source: normal-focus sealed tube
ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2014)
T_{min} = 0.676, T_{max} = 0.746
36969 measured reflections
3815 independent reflections
3240 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
θ_{max} = 27.5°, θ_{min} = 2.4°
h = −18→18
k = −18→18
l = −21→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.057
S = 1.08
3815 reflections
200 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0214P)^2 + 2.1684P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.004$
$\Delta\rho_{max} = 0.28$ e Å^{-3}
$\Delta\rho_{min} = -0.44$ e Å^{-3}

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Co1	1.000000	0.500000	0.500000	0.01630 (8)	
Br1	1.16858 (2)	0.48649 (2)	0.43493 (2)	0.02843 (6)	
C1	0.94105 (12)	0.43581 (12)	0.41343 (10)	0.0193 (4)	
N1	0.90141 (10)	0.39272 (10)	0.36473 (9)	0.0216 (3)	
C2	0.85537 (12)	0.32585 (12)	0.31491 (11)	0.0202 (4)	
C3	0.82015 (12)	0.24477 (13)	0.35419 (11)	0.0229 (4)	
C4	0.78053 (13)	0.17621 (13)	0.30419 (12)	0.0268 (4)	
H4A	0.755319	0.120366	0.328316	0.032*	
C5	0.77698 (13)	0.18757 (14)	0.21948 (12)	0.0293 (4)	
H5A	0.752326	0.138253	0.186106	0.035*	
C6	0.80919 (13)	0.27036 (14)	0.18356 (12)	0.0268 (4)	
H6A	0.804256	0.278191	0.125748	0.032*	
C7	0.84881 (12)	0.34267 (13)	0.23054 (11)	0.0215 (4)	
C8	0.82543 (15)	0.23332 (15)	0.44612 (12)	0.0334 (5)	
H8A	0.785087	0.181145	0.463367	0.050*	
H8B	0.805971	0.292672	0.472712	0.050*	
H8C	0.888655	0.218700	0.462062	0.050*	
C9	0.88025 (14)	0.43415 (14)	0.19205 (12)	0.0303 (4)	
H9A	0.934578	0.457632	0.221106	0.046*	0.5
H9B	0.831180	0.481694	0.195670	0.046*	0.5
H9C	0.895532	0.422904	0.134222	0.046*	0.5
H9D	0.839615	0.450521	0.146226	0.046*	0.5
H9E	0.943013	0.426459	0.171662	0.046*	0.5
H9F	0.878662	0.485249	0.233110	0.046*	0.5
C10	0.97606 (12)	0.61811 (12)	0.45087 (10)	0.0194 (4)	
N2	0.95741 (10)	0.69076 (10)	0.42283 (9)	0.0216 (3)	
C11	0.92893 (12)	0.77415 (12)	0.38220 (11)	0.0194 (4)	
C12	0.93025 (12)	0.77252 (12)	0.29626 (11)	0.0207 (4)	
C13	0.89904 (13)	0.85373 (14)	0.25628 (12)	0.0273 (4)	
H13A	0.898734	0.855701	0.197936	0.033*	
C14	0.86837 (14)	0.93189 (14)	0.30042 (13)	0.0326 (5)	
H14A	0.847565	0.986972	0.271993	0.039*	
C15	0.86765 (14)	0.93073 (14)	0.38533 (13)	0.0316 (5)	
H15A	0.845846	0.984878	0.414474	0.038*	
C16	0.89841 (13)	0.85136 (14)	0.42887 (11)	0.0261 (4)	
C17	0.96157 (14)	0.68566 (13)	0.25024 (12)	0.0284 (4)	

H17A	0.960780	0.698844	0.191107	0.043*
H17B	0.920368	0.632255	0.262282	0.043*
H17C	1.024012	0.669161	0.267278	0.043*
C18	0.89717 (16)	0.84881 (17)	0.52141 (12)	0.0384 (5)
H18A	0.851547	0.894239	0.541927	0.058*
H18B	0.957850	0.866121	0.542493	0.058*
H18C	0.881320	0.784406	0.539998	0.058*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.02112 (16)	0.01458 (14)	0.01321 (15)	0.00015 (12)	−0.00401 (12)	−0.00021 (12)
Br1	0.02527 (10)	0.03428 (11)	0.02574 (10)	−0.00235 (8)	0.00502 (8)	−0.00528 (8)
C1	0.0221 (9)	0.0192 (8)	0.0166 (8)	0.0024 (7)	−0.0013 (7)	0.0008 (7)
N1	0.0223 (8)	0.0232 (8)	0.0194 (8)	0.0023 (6)	−0.0032 (6)	−0.0039 (6)
C2	0.0176 (9)	0.0214 (9)	0.0216 (9)	0.0025 (6)	−0.0052 (7)	−0.0072 (7)
C3	0.0206 (9)	0.0253 (9)	0.0229 (9)	0.0051 (7)	−0.0019 (7)	−0.0021 (7)
C4	0.0209 (9)	0.0221 (9)	0.0375 (11)	−0.0001 (7)	−0.0008 (8)	−0.0033 (8)
C5	0.0259 (10)	0.0289 (10)	0.0332 (11)	0.0006 (8)	−0.0061 (8)	−0.0143 (8)
C6	0.0264 (10)	0.0345 (10)	0.0195 (9)	0.0025 (8)	−0.0046 (7)	−0.0086 (8)
C7	0.0200 (9)	0.0239 (9)	0.0206 (9)	0.0045 (7)	−0.0039 (7)	−0.0024 (7)
C8	0.0349 (11)	0.0400 (11)	0.0252 (10)	−0.0022 (9)	−0.0012 (9)	0.0044 (8)
C9	0.0335 (11)	0.0298 (10)	0.0277 (10)	0.0010 (8)	−0.0073 (9)	0.0031 (8)
C10	0.0202 (9)	0.0232 (9)	0.0148 (8)	−0.0005 (7)	−0.0022 (7)	−0.0014 (7)
N2	0.0224 (8)	0.0210 (8)	0.0213 (8)	−0.0003 (6)	−0.0020 (6)	0.0038 (6)
C11	0.0183 (8)	0.0174 (8)	0.0226 (9)	0.0005 (7)	−0.0024 (7)	0.0047 (7)
C12	0.0176 (9)	0.0221 (8)	0.0224 (9)	−0.0036 (7)	−0.0010 (7)	0.0017 (7)
C13	0.0276 (10)	0.0305 (10)	0.0237 (10)	−0.0030 (8)	−0.0029 (8)	0.0083 (8)
C14	0.0337 (11)	0.0240 (10)	0.0400 (12)	0.0042 (8)	−0.0077 (9)	0.0114 (9)
C15	0.0332 (11)	0.0233 (10)	0.0382 (12)	0.0099 (8)	−0.0024 (9)	−0.0029 (8)
C16	0.0248 (10)	0.0279 (10)	0.0254 (10)	0.0031 (7)	−0.0029 (8)	−0.0017 (8)
C17	0.0298 (10)	0.0298 (10)	0.0257 (10)	0.0000 (8)	0.0001 (8)	−0.0051 (8)
C18	0.0427 (13)	0.0483 (13)	0.0242 (11)	0.0122 (10)	−0.0003 (9)	−0.0053 (9)

Geometric parameters (Å, º)

Co1—C10^{i}	1.8718 (17)	C9—H9C	0.9800
Co1—C10	1.8718 (17)	C9—H9D	0.9800
Co1—C1^{i}	1.8800 (17)	C9—H9E	0.9800
Co1—C1	1.8800 (17)	C9—H9F	0.9800
Co1—Br1^{i}	2.6846 (3)	C10—N2	1.149 (2)
Co1—Br1	2.6846 (3)	N2—C11	1.406 (2)
C1—N1	1.152 (2)	C11—C16	1.395 (3)
N1—C2	1.410 (2)	C11—C12	1.399 (2)
C2—C7	1.397 (2)	C12—C13	1.388 (2)
C2—C3	1.402 (3)	C12—C17	1.501 (2)
C3—C4	1.386 (2)	C13—C14	1.385 (3)
C3—C8	1.507 (3)	C13—H13A	0.9500

C4—C5	1.389 (3)	C14—C15	1.382 (3)
C4—H4A	0.9500	C14—H14A	0.9500
C5—C6	1.382 (3)	C15—C16	1.393 (3)
C5—H5A	0.9500	C15—H15A	0.9500
C6—C7	1.395 (2)	C16—C18	1.507 (3)
C6—H6A	0.9500	C17—H17A	0.9800
C7—C9	1.499 (3)	C17—H17B	0.9800
C8—H8A	0.9800	C17—H17C	0.9800
C8—H8B	0.9800	C18—H18A	0.9800
C8—H8C	0.9800	C18—H18B	0.9800
C9—H9A	0.9800	C18—H18C	0.9800
C9—H9B	0.9800		

C10^{i}—Co1—C10	180.0	C7—C9—H9D	109.5
C10^{i}—Co1—C1^{i}	91.03 (7)	H9A—C9—H9D	141.1
C10—Co1—C1^{i}	88.96 (7)	H9B—C9—H9D	56.3
C10^{i}—Co1—C1	88.97 (7)	H9C—C9—H9D	56.3
C10—Co1—C1	91.03 (7)	C7—C9—H9E	109.5
C1^{i}—Co1—C1	180.0	H9A—C9—H9E	56.3
C10^{i}—Co1—Br1^{i}	93.72 (5)	H9B—C9—H9E	141.1
C10—Co1—Br1^{i}	86.28 (5)	H9C—C9—H9E	56.3
C1^{i}—Co1—Br1^{i}	95.15 (5)	H9D—C9—H9E	109.5
C1—Co1—Br1^{i}	84.85 (5)	C7—C9—H9F	109.5
C10^{i}—Co1—Br1	86.28 (5)	H9A—C9—H9F	56.3
C10—Co1—Br1	93.72 (5)	H9B—C9—H9F	56.3
C1^{i}—Co1—Br1	84.85 (5)	H9C—C9—H9F	141.1
C1—Co1—Br1	95.15 (5)	H9D—C9—H9F	109.5
Br1^{i}—Co1—Br1	180.0	H9E—C9—H9F	109.5
N1—C1—Co1	174.92 (16)	N2—C10—Co1	176.65 (17)
C1—N1—C2	169.55 (17)	C10—N2—C11	173.77 (18)
C7—C2—C3	124.07 (16)	C16—C11—C12	124.14 (16)
C7—C2—N1	119.07 (16)	C16—C11—N2	118.92 (16)
C3—C2—N1	116.85 (16)	C12—C11—N2	116.91 (16)
C4—C3—C2	116.59 (17)	C13—C12—C11	116.79 (17)
C4—C3—C8	122.04 (18)	C13—C12—C17	122.11 (17)
C2—C3—C8	121.37 (16)	C11—C12—C17	121.08 (16)
C3—C4—C5	121.26 (18)	C14—C13—C12	120.80 (18)
C3—C4—H4A	119.4	C14—C13—H13A	119.6
C5—C4—H4A	119.4	C12—C13—H13A	119.6
C6—C5—C4	120.23 (17)	C15—C14—C13	120.78 (18)
C6—C5—H5A	119.9	C15—C14—H14A	119.6
C4—C5—H5A	119.9	C13—C14—H14A	119.6
C5—C6—C7	121.29 (18)	C14—C15—C16	121.02 (18)
C5—C6—H6A	119.4	C14—C15—H15A	119.5
C7—C6—H6A	119.4	C16—C15—H15A	119.5
C6—C7—C2	116.39 (17)	C15—C16—C11	116.46 (17)
C6—C7—C9	121.29 (17)	C15—C16—C18	121.56 (18)
C2—C7—C9	122.30 (16)	C11—C16—C18	121.97 (17)

C3—C8—H8A	109.5	C12—C17—H17A	109.5
C3—C8—H8B	109.5	C12—C17—H17B	109.5
H8A—C8—H8B	109.5	H17A—C17—H17B	109.5
C3—C8—H8C	109.5	C12—C17—H17C	109.5
H8A—C8—H8C	109.5	H17A—C17—H17C	109.5
H8B—C8—H8C	109.5	H17B—C17—H17C	109.5
C7—C9—H9A	109.5	C16—C18—H18A	109.5
C7—C9—H9B	109.5	C16—C18—H18B	109.5
H9A—C9—H9B	109.5	H18A—C18—H18B	109.5
C7—C9—H9C	109.5	C16—C18—H18C	109.5
H9A—C9—H9C	109.5	H18A—C18—H18C	109.5
H9B—C9—H9C	109.5	H18B—C18—H18C	109.5

C1—N1—C2—C7	−155.7 (9)	N1—C2—C7—C9	−6.6 (3)
C1—N1—C2—C3	23.2 (10)	C16—C11—C12—C13	0.1 (3)
C7—C2—C3—C4	3.1 (3)	N2—C11—C12—C13	177.80 (16)
N1—C2—C3—C4	−175.71 (15)	C16—C11—C12—C17	−178.22 (18)
C7—C2—C3—C8	−176.91 (17)	N2—C11—C12—C17	−0.5 (3)
N1—C2—C3—C8	4.3 (3)	C11—C12—C13—C14	0.0 (3)
C2—C3—C4—C5	0.6 (3)	C17—C12—C13—C14	178.33 (18)
C8—C3—C4—C5	−179.43 (18)	C12—C13—C14—C15	−0.3 (3)
C3—C4—C5—C6	−3.2 (3)	C13—C14—C15—C16	0.5 (3)
C4—C5—C6—C7	2.3 (3)	C14—C15—C16—C11	−0.4 (3)
C5—C6—C7—C2	1.1 (3)	C14—C15—C16—C18	−179.4 (2)
C5—C6—C7—C9	−177.47 (18)	C12—C11—C16—C15	0.1 (3)
C3—C2—C7—C6	−3.9 (3)	N2—C11—C16—C15	−177.56 (17)
N1—C2—C7—C6	174.89 (16)	C12—C11—C16—C18	179.09 (19)
C3—C2—C7—C9	174.63 (17)	N2—C11—C16—C18	1.5 (3)

Symmetry code: (i) −*x*+2, −*y*+1, −*z*+1.

trans-Dibromidobis(2,6-dimethylphenyl isocyanide)nickel(II) (5)

Crystal data

$[NiBr_2(C_9H_9N)_2]$
M_r = 480.87
Monoclinic, $P2_1/n$
a = 20.433 (7) Å
b = 4.5010 (15) Å
c = 21.285 (7) Å
β = 105.682 (5)°
V = 1884.7 (11) Å^3
Z = 4
$F(000)$ = 952
D_x = 1.695 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2252 reflections
θ = 2.9–25.1°
μ = 5.27 mm^{-1}
T = 173 K
Rod, orange-brown
0.50 × 0.10 × 0.05 mm

Data collection

Siemens SMART CCD platform diffractometer
Radiation source: normal-focus sealed tube
ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2014)
T_{min} = 0.592, T_{max} = 0.746
9617 measured reflections
3287 independent reflections
2241 reflections with $I > 2\sigma(I)$
R_{int} = 0.074
θ_{max} = 25.1°, θ_{min} = 1.2°

$h = -24 \rightarrow 22$
$k = -5 \rightarrow 5$
$l = -24 \rightarrow 25$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.109$
$S = 1.14$
3287 reflections
215 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0243P)^2 + 4.2164P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.94$ e Å^{-3}
$\Delta\rho_{min} = -0.62$ e Å^{-3}

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ni1	1.000000	0.000000	1.000000	0.0276 (3)
Br1	1.08394 (4)	−0.1766 (2)	0.95588 (4)	0.0397 (2)
C1	0.9623 (3)	0.2110 (16)	0.9247 (4)	0.0280 (17)
N1	0.9390 (3)	0.3399 (14)	0.8775 (3)	0.0304 (15)
C2	0.9126 (3)	0.5015 (18)	0.8193 (3)	0.0301 (17)
C3	0.9466 (4)	0.4754 (18)	0.7704 (3)	0.0349 (19)
C4	0.9187 (4)	0.6311 (19)	0.7131 (4)	0.042 (2)
H4A	0.940261	0.622905	0.678815	0.051*
C5	0.8608 (4)	0.7960 (19)	0.7050 (4)	0.044 (2)
H5A	0.842345	0.897487	0.664973	0.053*
C6	0.8288 (4)	0.8168 (18)	0.7545 (4)	0.0371 (19)
H6A	0.789015	0.935114	0.747880	0.045*
C7	0.8532 (4)	0.6699 (17)	0.8133 (3)	0.0296 (17)
C8	1.0111 (4)	0.301 (2)	0.7816 (4)	0.049 (2)
H8A	1.003809	0.099341	0.795851	0.073*
H8B	1.046981	0.398147	0.815317	0.073*
H8C	1.024815	0.290244	0.740916	0.073*
C9	0.8196 (4)	0.6850 (19)	0.8679 (3)	0.040 (2)
H9A	0.776709	0.794649	0.853356	0.060*
H9B	0.849723	0.786953	0.905414	0.060*
H9C	0.810442	0.483274	0.880695	0.060*
Ni2	1.000000	1.000000	0.500000	0.0261 (3)
Br2	1.04377 (4)	0.88606 (19)	0.41400 (3)	0.0369 (2)
C10	0.9259 (4)	0.7604 (18)	0.4649 (3)	0.0303 (18)
N2	0.8800 (3)	0.6054 (14)	0.4427 (3)	0.0283 (14)
C11	0.8253 (3)	0.4165 (17)	0.4161 (3)	0.0294 (18)

C12	0.7765 (3)	0.3712 (17)	0.4513 (3)	0.0314 (18)
C13	0.7222 (4)	0.1865 (19)	0.4234 (4)	0.042 (2)
H13A	0.687754	0.154628	0.445056	0.050*
C14	0.7176 (4)	0.0498 (19)	0.3651 (4)	0.045 (2)
H14A	0.680554	−0.080039	0.347475	0.054*
C15	0.7658 (4)	0.0956 (19)	0.3309 (4)	0.042 (2)
H15A	0.760984	−0.000329	0.290088	0.051*
C16	0.8212 (4)	0.2812 (17)	0.3560 (3)	0.0305 (18)
C17	0.7838 (4)	0.523 (2)	0.5167 (3)	0.042 (2)
H17A	0.797115	0.731026	0.513827	0.063*
H17B	0.818735	0.421277	0.550474	0.063*
H17C	0.740339	0.515522	0.527775	0.063*
C18	0.8747 (4)	0.339 (2)	0.3211 (4)	0.043 (2)
H18A	0.919338	0.283240	0.349374	0.065*
H18B	0.874819	0.550877	0.310311	0.065*
H18C	0.864908	0.221556	0.280917	0.065*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0253 (7)	0.0318 (8)	0.0249 (7)	0.0010 (6)	0.0057 (6)	0.0028 (6)
Br1	0.0346 (5)	0.0464 (6)	0.0411 (5)	0.0056 (4)	0.0154 (4)	0.0012 (4)
C1	0.025 (4)	0.023 (5)	0.037 (4)	0.002 (3)	0.009 (3)	−0.006 (4)
N1	0.029 (3)	0.035 (4)	0.025 (3)	−0.004 (3)	0.001 (3)	0.003 (3)
C2	0.028 (4)	0.035 (5)	0.022 (4)	−0.005 (4)	−0.002 (3)	0.003 (4)
C3	0.038 (5)	0.035 (5)	0.033 (4)	−0.014 (4)	0.012 (4)	−0.006 (4)
C4	0.055 (6)	0.041 (6)	0.033 (4)	−0.008 (5)	0.017 (4)	−0.001 (4)
C5	0.057 (6)	0.042 (6)	0.029 (4)	−0.005 (5)	0.003 (4)	0.011 (4)
C6	0.036 (4)	0.035 (5)	0.037 (4)	0.004 (4)	0.003 (4)	0.005 (4)
C7	0.033 (4)	0.033 (5)	0.021 (3)	−0.003 (4)	0.003 (3)	0.002 (3)
C8	0.040 (5)	0.063 (7)	0.047 (5)	0.007 (5)	0.018 (4)	0.002 (5)
C9	0.035 (5)	0.047 (6)	0.034 (4)	0.004 (4)	0.002 (4)	−0.002 (4)
Ni2	0.0251 (7)	0.0303 (8)	0.0229 (6)	−0.0018 (6)	0.0066 (6)	0.0044 (6)
Br2	0.0399 (5)	0.0448 (5)	0.0296 (4)	−0.0011 (4)	0.0159 (4)	0.0011 (4)
C10	0.033 (4)	0.041 (5)	0.016 (3)	0.004 (4)	0.005 (3)	0.007 (3)
N2	0.026 (4)	0.033 (4)	0.023 (3)	0.000 (3)	0.002 (3)	0.005 (3)
C11	0.019 (4)	0.034 (5)	0.029 (4)	0.000 (3)	−0.004 (3)	0.008 (4)
C12	0.028 (4)	0.028 (5)	0.033 (4)	0.004 (4)	0.001 (3)	0.012 (4)
C13	0.023 (4)	0.044 (6)	0.055 (5)	−0.002 (4)	0.006 (4)	0.008 (5)
C14	0.036 (5)	0.033 (6)	0.056 (5)	−0.014 (4)	−0.005 (4)	0.004 (4)
C15	0.040 (5)	0.040 (6)	0.040 (4)	−0.005 (4)	−0.004 (4)	−0.003 (4)
C16	0.026 (4)	0.031 (5)	0.030 (4)	0.008 (3)	0.002 (3)	0.010 (4)
C17	0.044 (5)	0.050 (6)	0.035 (4)	0.003 (4)	0.017 (4)	0.002 (4)
C18	0.040 (5)	0.051 (6)	0.034 (4)	0.004 (4)	0.002 (4)	−0.008 (4)

Geometric parameters (Å, º)

Ni1—C1	1.844 (8)	Ni2—C10	1.845 (8)
Ni1—C1^{i}	1.844 (8)	Ni2—C10^{ii}	1.845 (8)
Ni1—Br1	2.3079 (9)	Ni2—Br2	2.3020 (9)
Ni1—Br1^{i}	2.3080 (9)	Ni2—Br2^{ii}	2.3020 (9)
C1—N1	1.146 (8)	C10—N2	1.161 (9)
N1—C2	1.411 (9)	N2—C11	1.397 (9)
C2—C3	1.404 (9)	C11—C16	1.398 (9)
C2—C7	1.409 (10)	C11—C12	1.415 (9)
C3—C4	1.387 (10)	C12—C13	1.385 (10)
C3—C8	1.498 (11)	C12—C17	1.522 (10)
C4—C5	1.367 (11)	C13—C14	1.366 (11)
C4—H4A	0.9500	C13—H13A	0.9500
C5—C6	1.385 (10)	C14—C15	1.390 (11)
C5—H5A	0.9500	C14—H14A	0.9500
C6—C7	1.383 (10)	C15—C16	1.391 (10)
C6—H6A	0.9500	C15—H15A	0.9500
C7—C9	1.504 (9)	C16—C18	1.502 (10)
C8—H8A	0.9800	C17—H17A	0.9800
C8—H8B	0.9800	C17—H17B	0.9800
C8—H8C	0.9800	C17—H17C	0.9800
C9—H9A	0.9800	C18—H18A	0.9800
C9—H9B	0.9800	C18—H18B	0.9800
C9—H9C	0.9800	C18—H18C	0.9800
C1—Ni1—C1^{i}	180.0	C10—Ni2—C10^{ii}	180.0
C1—Ni1—Br1	90.3 (2)	C10—Ni2—Br2	90.1 (2)
C1^{i}—Ni1—Br1	89.7 (2)	C10^{ii}—Ni2—Br2	89.9 (2)
C1—Ni1—Br1^{i}	89.7 (2)	C10—Ni2—Br2^{ii}	89.9 (2)
C1^{i}—Ni1—Br1^{i}	90.3 (2)	C10^{ii}—Ni2—Br2^{ii}	90.1 (2)
Br1—Ni1—Br1^{i}	180.00 (4)	Br2—Ni2—Br2^{ii}	180.0
N1—C1—Ni1	179.3 (7)	N2—C10—Ni2	178.7 (7)
C1—N1—C2	178.0 (7)	C10—N2—C11	179.4 (8)
C3—C2—C7	124.4 (6)	N2—C11—C16	118.7 (6)
C3—C2—N1	117.4 (7)	N2—C11—C12	118.3 (6)
C7—C2—N1	118.2 (6)	C16—C11—C12	123.0 (7)
C4—C3—C2	116.2 (7)	C13—C12—C11	117.3 (7)
C4—C3—C8	122.7 (7)	C13—C12—C17	122.1 (7)
C2—C3—C8	121.0 (7)	C11—C12—C17	120.7 (7)
C5—C4—C3	121.4 (7)	C14—C13—C12	120.6 (7)
C5—C4—H4A	119.3	C14—C13—H13A	119.7
C3—C4—H4A	119.3	C12—C13—H13A	119.7
C4—C5—C6	120.7 (7)	C13—C14—C15	121.7 (8)
C4—C5—H5A	119.6	C13—C14—H14A	119.1
C6—C5—H5A	119.6	C15—C14—H14A	119.1
C7—C6—C5	121.8 (7)	C14—C15—C16	120.3 (8)
C7—C6—H6A	119.1	C14—C15—H15A	119.8

C5—C6—H6A	119.1	C16—C15—H15A	119.8
C6—C7—C2	115.5 (6)	C15—C16—C11	117.1 (7)
C6—C7—C9	123.4 (7)	C15—C16—C18	122.6 (7)
C2—C7—C9	121.1 (6)	C11—C16—C18	120.3 (7)
C3—C8—H8A	109.5	C12—C17—H17A	109.5
C3—C8—H8B	109.5	C12—C17—H17B	109.5
H8A—C8—H8B	109.5	H17A—C17—H17B	109.5
C3—C8—H8C	109.5	C12—C17—H17C	109.5
H8A—C8—H8C	109.5	H17A—C17—H17C	109.5
H8B—C8—H8C	109.5	H17B—C17—H17C	109.5
C7—C9—H9A	109.5	C16—C18—H18A	109.5
C7—C9—H9B	109.5	C16—C18—H18B	109.5
H9A—C9—H9B	109.5	H18A—C18—H18B	109.5
C7—C9—H9C	109.5	C16—C18—H18C	109.5
H9A—C9—H9C	109.5	H18A—C18—H18C	109.5
H9B—C9—H9C	109.5	H18B—C18—H18C	109.5

C7—C2—C3—C4	0.7 (11)	N2—C11—C12—C13	179.1 (7)
N1—C2—C3—C4	178.5 (7)	C16—C11—C12—C13	−0.8 (11)
C7—C2—C3—C8	177.7 (8)	N2—C11—C12—C17	−0.4 (10)
N1—C2—C3—C8	−4.4 (11)	C16—C11—C12—C17	179.7 (7)
C2—C3—C4—C5	−1.0 (12)	C11—C12—C13—C14	1.6 (11)
C8—C3—C4—C5	−178.0 (8)	C17—C12—C13—C14	−179.0 (7)
C3—C4—C5—C6	1.1 (13)	C12—C13—C14—C15	−1.6 (13)
C4—C5—C6—C7	−0.9 (13)	C13—C14—C15—C16	0.9 (12)
C5—C6—C7—C2	0.5 (11)	C14—C15—C16—C11	−0.2 (11)
C5—C6—C7—C9	−179.5 (7)	C14—C15—C16—C18	−179.9 (7)
C3—C2—C7—C6	−0.4 (11)	N2—C11—C16—C15	−179.7 (7)
N1—C2—C7—C6	−178.3 (7)	C12—C11—C16—C15	0.2 (11)
C3—C2—C7—C9	179.6 (7)	N2—C11—C16—C18	0.0 (10)
N1—C2—C7—C9	1.8 (11)	C12—C11—C16—C18	179.9 (7)

Symmetry codes: (i) −*x*+2, −*y*, −*z*+2; (ii) −*x*+2, −*y*+2, −*z*+1.