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Iron(II) complexes of dimethyltriazacyclophane

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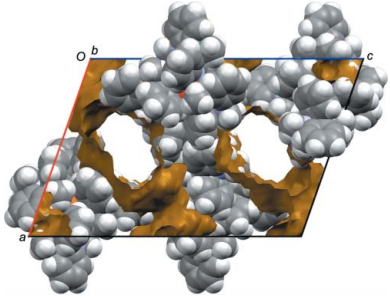
Treatment of the *ortho*-triazacyclophane 1,4-dimethyltribenzo[*b*,*e*,*h*][1,4,7]triazacyclononа-2,5,8-triene [$(C_6H_5)_3(NH)(NCH_3)_2$, **L1**] with $Fe[N(SiMe_3)_2]_2$ yields the dimeric iron(II) complex bis(μ-1,4-dimethyltribenzo[*b*,*e*,*h*][1,4,7]triazacyclononа-2,5,8-trien-7-ido)bis[(μ-1,4-dimethyltribenzo[*b*,*e*,*h*][1,4,7]triazacyclononа-2,5,8-trien-7-ido)iron(II)], $[Fe(C_{20}H_{18}N_3)_4]$ or Fe_2(**L1**)$_4$ (**9**). Dissolution of **9** in tetrahydrofuran (THF) results in solvation by two THF ligands and the formation of a simpler monoiron complex, namely bis(μ-1,4-dimethyltribenzo[*b*,*e*,*h*][1,4,7]triazacyclononа-2,5,8-trien-7-ido-κN^7)bis(tetrahydrofuran-κO)iron(II), $[Fe(C_{20}H_{18}N_3)_2(C_4H_8O)_2]$ or (**L1**)$_2$Fe(THF)$_2$ (**10**). The reaction is reversible and **10** reverts *in vacuo* to diiron complex **9**. In the structures of both **9** and **10**, the monoanionic triazacyclophane ligand **L1**$^-$ is observed in only the less-symmetric saddle conformation. No bowl-shaped crown conformers are observed in the solid state, thus preventing chelating κ^3-coordination to the metal as had been proposed earlier based on density functional theory (DFT) calculations. Instead, the **L1**$^-$ ligands are bound in either a η^2-chelating fashion through the amide and one amine donor (for one of the four ligands of **9**), or solely through their amide N atoms in an even simpler monodentate η^1-coordination mode. Density functional calculations on dimer **9** revealed nearly full cationic charges on each Fe atom and no bonding interaction between the two metal centers, consistent with the relatively long Fe···Fe distance of 2.912 (1) Å observed in the solid state.

1. Introduction

Amine-containing macrocyclic ligands have been widely used and play an important role in the field of coordination chemistry due to their ability to, on many occasions, form kinetically inert metal complexes. Common among them is 1,4,7-triazacyclononane (TACN, **1**, Fig. 1), which is often considered to structurally and electronically resemble the η^5-cyclopentadienyl (Cp) ligand in its usual facial coordination. Given the modular nature of TACN, the straightforward introduction of chemical functionality, generally through the N atoms, enables modulation of the ligand, affording a variety of multidentate ligands ranging from tri- to nonadentate TACN-based ligands for supporting main-group-, transition- (Yang & Zompa, 1976), lanthanide-, and actinide-metal complexes, and the coordination chemistry has been reviewed (Yang & Zompa, 1976; Chaudhuri & Wieghardt, 2007). Among the transition-metal compounds are several TACN complexes of iron (Turner & Schultz, 2001; Tse *et al.*, 2014; Mitra *et al.*, 2014; Boeyens *et al.*, 1985; Kindermann *et al.*, 2016; Tsitovich *et al.*, 2015; Sabenya *et al.*, 2017; Nakanishi *et al.*, 2016; Thorarinsdottir *et al.*, 2017).

Despite its vast utility and popularity, backbone-aryl modifications of TACN, such as monobenzo-annelated deri-



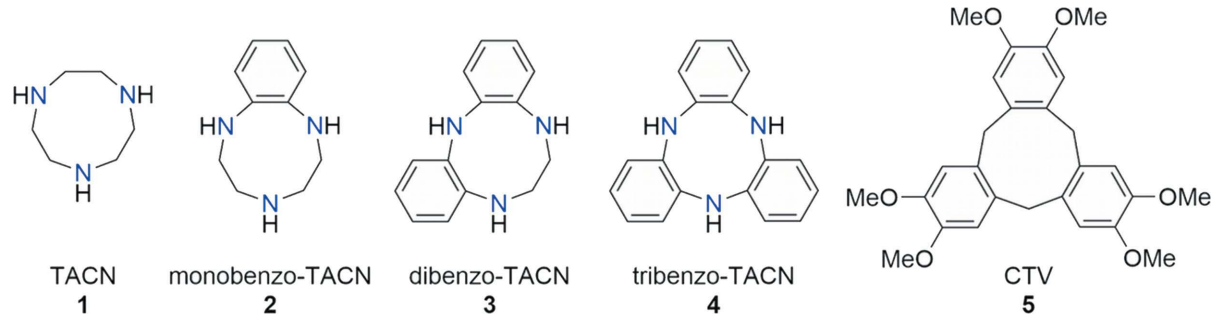


Figure 1
Triazacyclononane (TACN, **1**) and benzo-fused TACN derivatives, including monobenzo-TACN **2**, dibenzo-TACN **3**, triazacyclophane **4**, and cyclotriveratrylene (CTV, **5**).

vative **2**, dibenzoannelated derivative **3**, and triazaorthocyclophane **4** (Fig. 1) remain almost completely unexplored. It is noteworthy that ancillary ligands based on aniline or phenylenediamine moieties are considered as redox non-innocent ligands and their metal complexes have drawn much attention due to unique reactivity and catalytic applications. (Chłopek *et al.*, 2006; Lyaskovskyy & de Bruin, 2012; Hicks, 2008; Suarez *et al.*, 2013; Ghosh *et al.*, 2001; Herebian *et al.*, 2003; Van der Meer *et al.*, 2014; Leconte *et al.*, 2014, 2017; Kochem *et al.*, 2013) A literature search reveals that only two examples of benzo-annelated derivatives have been prepared, (Panagopoulos *et al.*, 2010; Samanta *et al.*, 2012), yet no well-defined metal complexes have been successfully isolated (Samanta *et al.*, 2012), although binding of the neutral ligand **4** to iron has been proposed and was the subject of modeling studies (Foscato *et al.*, 2015). In contrast, several late-transition-metal complexes employing the S-donor congener, trithiaorthocyclophane, have been reported (Von Deuten *et al.*, 1979; Von Deuten & Klar, 1981; Kopf *et al.*, 1979).

Cyclotriveratrylene (CTV, **5**) (Collet, 1987) is a bowl-shaped molecule that has been explored for applications in sensors, self-organized materials, liquid crystals, and metallo-supramolecular chemistry (Hardie, 2010). CTV and its cryptophane derivatives are of great interest in molecular recognition in host–guest chemistry (Hardie, 2012). Our interest in the supramolecular scaffold CTV includes studies of the conformational dynamics of its derivatives between the bowl-shaped crown conformer and the saddle confomer (French *et al.*, 2009), homologs of CTV (Lutz *et al.*, 2012), and apically-modified chiral derivatives for enantio-discrimination (Lutz *et al.*, 2018). Inspired by the conceptual combination of CTV and TACN, we prepared 1,4-dimethyltribenzo[*b*,*e*,*h*]-[1,4,7]triazacyclonona-2,5,8-triene (**7**), an *ortho*-triazacyclophane, and 1,4,7-trimethyltribenzo[*b*,*e*,*h*][1,4,7]triazacyclonona-2,5,8-triene (**8**) (Fig. 2). Specifically, we envisioned a supramolecular scaffold that could include a complexed metal, both to provide a cationic cyclophane for anion recognition, as well as to enable an electrochemically switchable host molecule. In light of the limited previous studies, we were interested in exploring the coordination chemistry of benzo-fused TACN ligands **7** and **8** with first-row transition metals, especially iron because of spin-crossover behavior, which was observed for $[Fe(TACN)_2]^{2+}$ (Turner & Schultz, 2001).

2. Experimental

2.1. Material and methods

All manipulations were performed under a nitrogen atmosphere using standard Schlenk techniques or in an M. Braun UNIlab Pro glove-box. Glassware was dried at 150 °C overnight. Diethyl ether, *n*-pentane, tetrahydrofuran, and toluene were purified using a Pure Process Technology solvent purification system. Deuterated benzene was first dried with CaH_2, then over Na/benzophenone, and then vacuum transferred into a storage container. Before use, an aliquot of each solvent was tested with a drop of sodium benzophenone ketyl in THF solution. All reagents were purchased from commercial vendors and used as received. $Fe[N(SiMe_3)_2]_2$ was prepared according to a literature procedure (Ohki *et al.*, 2010). ^{1}H NMR data were recorded on a Varian Inova 300 or 500 MHz spectrometer at 22 °C. Resonances in the ^{1}H NMR

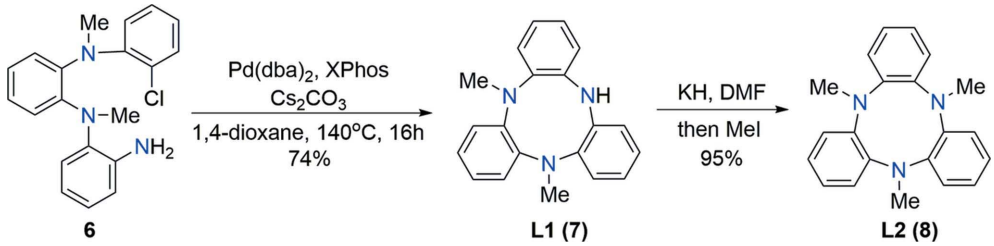


Figure 2
Optimized Buchwald–Hartwig macrocyclization of chloroaniline derivative **6** to yield **L1** (**7**) and the previously reported methylation (Panagopoulos *et al.*, 2010) to afford **L2** (**8**).

Table 1
Experimental details.

	9	**10**
Crystal data		
Chemical formula	$[Fe(C_{20}H_{18}N_3)_4]$	$[Fe(C_{20}H_{18}N_3)_2(C_4H_8O)_2]$
M_r	1313.19	800.80
Crystal system, space group	Monoclinic, $P2_1/c$	Orthorhombic, *Pbca*
Temperature (K)	100	100
a, b, c (Å)	21.758 (2), 12.7682 (13), 31.382 (3)	15.9624 (12), 15.8047 (11), 32.747 (2)
α, β, γ (°)	90, 109.191 (5), 90	90, 90, 90
V (Å^3)	8233.7 (14)	8261.5 (10)
Z	4	8
Radiation type	Cu $K\alpha$	Cu $K\alpha$
μ (mm^{-1})	3.17	3.29
Crystal size (mm)	0.11 × 0.06 × 0.05	0.11 × 0.10 × 0.06
Data collection		
Diffractometer	Bruker Prospector CCD	Bruker X8 Prospector CCD
Absorption correction	Multi-scan (*APEX2*; Bruker, 2014)	Multi-scan (*APEX2*; Bruker, 2014)
T_{min}, T_{max}	0.478, 0.753	0.569, 0.753
No. of measured, independent and observed $[I > 2\sigma(I)]$ reflections	52565, 14191, 8827	23792, 7194, 5411
R_{int}	0.133	0.055
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.597	0.596
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.076, 0.193, 1.03	0.043, 0.111, 1.05
No. of reflections	14191	7194
No. of parameters	855	555
No. of restraints	0	132
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.79, −0.54	0.45, −0.28

Computer programs: *APEX2* (Bruker, 2014), *SAINT* (Bruker, 2014), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *shelXle* (Hübschle *et al.*, 2011) and *publCIF* (Westrip, 2010).

spectra are referenced to residual C_6D_5H at d = 7.16 or C_4D_7HO at d = 3.58 ppm. Solution magnetic susceptibilities were determined by the Evans method (Schubert, 1992). Elemental analysis was conducted by Midwest Microlab, LLC (Indianapolis, IN).

2.2. Computational studies

The CIF file of **9** was opened with *SPARTAN'16* (Wavefunction, 2016). The positions of the heavy atoms were frozen, and 'Molecular Mechanics' was performed to place the H atoms. The number of unpaired electrons was set at 8, and a single-point ground-state energy in the gas phase was calculated with Hartree–Fock (3-21G* basis set), using the options CONVERGE, SCFTOLERANCE=LOW, and SCFCYCLES=900. After successful completion, this result was used as the starting point for a density functional calculation using the EDF2 method and the 6-31G* basis set, keeping the same options as above (CONVERGE, SCFTOLERANCE=LOW, and SCFCYCLES=900). After successful completion, this result was used as the starting point for a density functional calculation using the EDF2 method and the 6-31G* basis set with options CONVERGE and SCFCYCLES=900 but eliminating the low SCF tolerance option. After successful completion, this result was used to calculate orbitals and energies, as well as charges and bond orders.

The result of the density functional EDF2 method with the 6-31G* basis set was used as the starting point to calculate the single-point energy using the density functional level of theory and the B3LYP method with the 6-31G* basis set, using the option MEM_STATIC=502. Continuing from this as the starting point for the wB97X-D method with the double-basis set 6-311+G(3df,2p)[6-311G*] afforded atomic charges of all atoms, including +0.961 for Fe1 and +0.998 for Fe2, as well as all HOMO/LUMO and spin-surface maps.

For direct comparison of the crystal structure with the equilibrium geometry calculated structure, the density functional (6-31G*) result with frozen heavy atoms was thawed (heavy atoms) and recalculated with the density functional level of theory, the EDF2 method, and the 6-31G* basis set, with COMVERGE SCFCYCLES=900, to yield the geometry optimized thawed structure.

2.3. Synthesis and crystallization

2.3.1. Preparation of 1,4-dimethyltribenzo[*b*,*e*,*h*][1,4,7]triazacyclonona-2,5,8-triene (L1, 7). A 125 ml pressure flask was charged with XPhos (260 mg, 0.54 mmol) and $Pd(dba)_2$ (dba is dibenzylideneacetone; 156 mg, 0.271 mmol), and a solution of chloroaniline, **6** (Panagopoulos *et al.*, 2010) (504 mg, 1.49 mmol), in anhydrous 1,4-dioxane (26 ml). The resulting solution was stirred at room temperature for 15 min as argon was passed over the solution. Caesium carbonate (838 mg, 2.57 mmol) was added and the resulting suspension was purged with argon for 30 min. The flask was sealed and heated in a 140 °C oil bath for 16 h. The reaction mixture was

cooled to room temperature and filtered through a pad of Celite, and the filter cake was washed with 1:1 (*v*/*v*) methanol–CH_2Cl_2. The filtrate was concentrated to dryness, leaving a brown solid which was partitioned between ethyl acetate and water. The aqueous layer was then extracted once with ethyl acetate and the organic extracts were combined, dried over $MgSO_4$, filtered, and concentrated to dryness. The residue was dissolved in ethyl acetate and to this solution was added 6.6 g of Celite, and the suspension concentrated to dryness, leaving an off-white powder. This powder was then applied to the top of an 80 g column (Silica gel 60A, 40–75 µm, 200 × 400 mesh) and eluted at a 42% maximum pump rate using a gradient starting with 20% CH_2Cl_2/petroleum ether and increasing to 30% CH_2Cl_2/petroleum ether, collecting 28 ml fractions to give macrocycle **7** (**L1**) as an off-white powder (yield: 338 mg, 75%; m.p. 228–230 °C) and with ^{1}H and ^{13}C spectra identical to those reported previously (Panagopoulos *et al.*, 2010).

2.3.2. Preparation of [(L1)$_4$Fe$_2$] (9). To a stirred solution of **L1** (394 mg, 2.0 mmol) in THF (4 ml) under an N_2 atmosphere was added a solution of $Fe[N(SiMe_3)_2]_2$ (377 mg, 1.0 mmol) in THF (2 ml). After 3 h, the volatiles were removed under reduced pressure. The residue was redissolved in toluene (5 ml) and filtered through a Celite pad. The filtrate was dried *in vacuo* to yield diiron complex **9** as a dark-red solid (yield: 408 mg, 68%). Crystals suitable for X-ray diffraction were grown by slow diffusion of *n*-pentane into a toluene solution at −35 °C. μ_{eff} = 7.7 (3) μ_B. Analysis calculated (%) for $C_{80}H_{72}Fe_2N_{12}$: C 73.17, H 5.53, N 12.80; found: C 73.53, H 5.35, N 12.42.

2.3.3. Preparation of [(L1)$_2$Fe(THF)$_2$] (10). Diiron complex **9** was dissolved in THF and immediately yielded a yellow–green solution. Crystals of monoiron complex **10** suitable for X-ray diffraction were obtained by slow diffusion of *n*-pentane into a THF solution at −35 °C. μ_{eff} (THF-d_8) = 5.1 (2) μ_B. Elemental analysis for **10** was not obtained due to the conversion of monoiron complex **10** back to diiron complex **9** upon prolonged vacuum drying.

2.4. Refinement

If not specified otherwise, H atoms attached to C and N atoms were positioned geometrically and constrained to ride on their parent atoms, with C—H bond lengths of 0.95 Å for alkene and aromatic, and 0.99 and 0.98 Å for aliphatic CH_2 and CH_3 groups, respectively. Methyl H atoms were allowed to rotate but not to tip to best fit the experimental electron density. U_{iso}(H) values were set to a multiple of U_{eq}(C), *i.e.* 1.5 for CH_3 and 1.2 for CH_2 and CH units, respectively.

The structure of diiron complex **9** contains solvent-accessible voids of 2479.0 Å^3 (*ca* 30% of the unit-cell volume). The residual electron-density peaks were not arranged in an interpretable pattern. The structure factors of the unresolved solvent molecules were estimated using reverse Fourier transform methods employing the SQUEEZE routine (van der Sluis & Spek, 1990; Spek, 2015), as implemented in the program *PLATON* (Spek, 2009). The resultant .fab file with scattering contributions from the solvent molecules was used in the further refinement in combination with the .hkl file. The SQUEEZE procedure estimated 569.8 electrons within the solvent-accessible voids, equivalent to 11.4 molecules of toluene (the solvents of crystallization were *n*-pentane and toluene).

In monoiron complex **10**, one THF ligand is disordered with two alternative orientations. The two moieties were restrained to have similar geometries, and the U^{ij} components of the anisotropic displacement parameters of the atoms were restrained to be similar if closer to each other than 1.7 Å (the s.u. value used was 0.01 Å^2). Subject to these conditions, the occupancy ratio refined to 0.790 (8):0.210 (8). Reflection 002 was affected by the beamstop and was omitted from the refinement.

Crystal data, data collection and structure refinement details are summarized in Table 1.

3. Results and discussion

3.1. Optimization of the synthesis of L1 (see Fig. 1)

Our previously reported synthesis (Panagopoulos *et al.*, 2010) of **L1** utilized a microwave-assisted intramolecular ring closure to form the tribenzotriazacyclononane ring. However, that reaction gave highly variable results, leading us to pursue reaction conditions that would yield more consistent and scalable yields. The original reaction conditions were 10 mol% of $Pd(dba)_2$, 20 mol% of racemic BINAP, and 150 mol% of Cs_2CO_3 in a solvent mixture of 1:1 (*v*/*v*) toluene–*tert*-butanol at a concentration of 0.09 molar under an argon atmosphere at 250 W and 130°C for 1 h to give the desired product in an optimum yield of 50%. We turned our attention to the use of $Pd(OAc)_2$ as a catalyst, which gave more consistent results, and varied the amount of catalyst from 10 to 60 mol%, revealing the optimal amount of catalyst to be *ca* 20 mol%. We explored alternatives to toluene/*tert*-butanol, and improved results were seen when the solvent was changed to 1,4-dioxane. The concentration of the reaction with respect to the acyclic starting material was then varied from 0.05 to 1 *M*, with

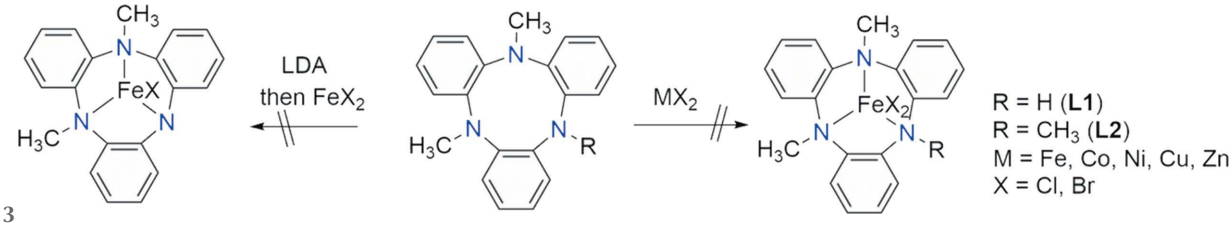


Figure 3
Attempted syntheses of *ortho*-triazacyclophane iron halide complexes.

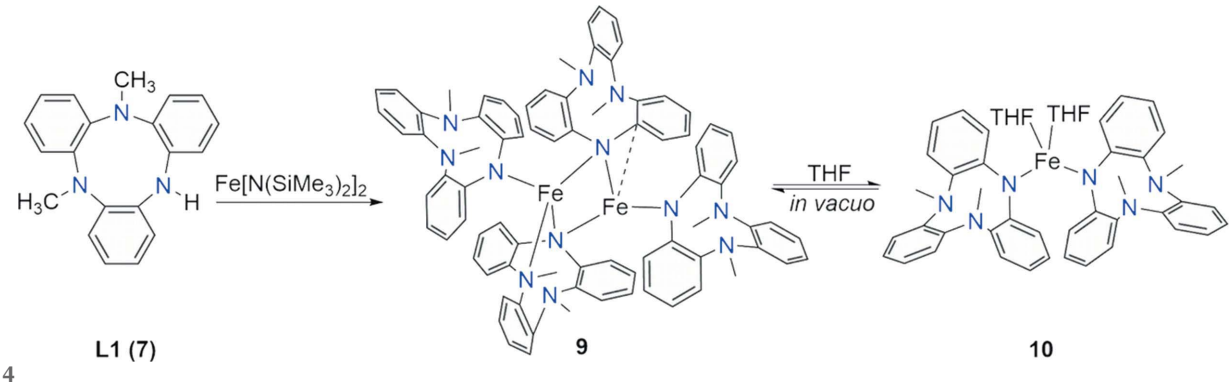


Figure 4
Synthesis of diiron complex **9** and monoiron complex **10**.

0.05 *M* producing the best results. We next turned our attention to varying the ligand, and found that *tert*-Butyl XPhos gave superior results to BINAP, JohnPhos, or tri-*tert*-butylphosphine. Microwave heating was explored relative the conventional thermal conditions. We tried performing the reaction in the microwave on a 0.1 mmol scale using 20 mol% $Pd(dba)_2$, 40 mol% XPhos, and 200 mol% Cs_2CO_3 at a concentration of 0.05 molar in 1,4-dioxane at 250 W and 105 °C. The result was consumption of the starting material, but it required four cycles in the microwave and was not scalable. Performing the reaction on the same scale under thermal conditions in a pressure tube at 160 °C also led to consumption of the starting material, but gave a number of side products. Lowering the temperature to 140 °C for 16 h gave consistently higher yields of 59–74% on a scale of 2 mmol of the acyclic amine halide, compared to yields of 50% or less on a 0.1 mmol scale (Fig. 2). Methylation as previously described (Panagopoulos *et al.*, 2010) gave the trimethyl derivative **L2**.

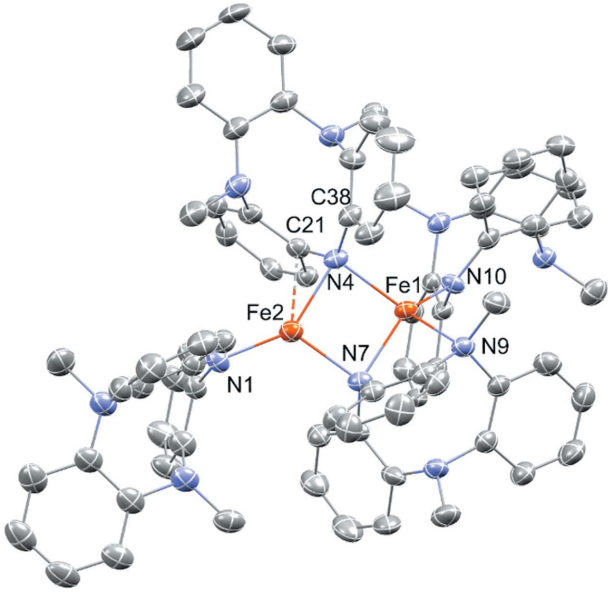


Figure 5
X-ray crystal structure of diiron complex **9**. Displacement ellipsoids are shown at the 50% probability level. Color key: orange = Fe, blue = N, and gray = C. H atoms and labels for most C and N atoms have been omitted for clarity. A displacement ellipsoid plot with H atoms and labels for all N atoms is given in the supporting information.

3.2. Synthesis of iron(II) complexes

Attempts at preparing first-row transition-metal complexes by simply mixing divalent salts of iron, cobalt, nickel, copper, or zinc with **L1**, or with **L2**, failed. A predicted molecular ion was observed when screening **L1** and **L2** with FeX_2, CoX_2, NiX_2 (X = Cl and Br), $CuCl_2$, or $ZnCl_2$; however, none of these complexes could be isolated based on ^{1}H NMR spectroscopy (Fig. 3). We attribute this to the poor availability of the nitrogen lone-pair electrons due to delocalization in the aromatic rings. We therefore pursued deprotonation of **L1**, followed by transmetallation with iron halide, which was promising in producing paramagnetic products, but the mixtures were intractable. The formation of multiple species could be attributed, at least in part, to the deprotonated ligand undergoing a Smiles rearrangement (Panagopoulos *et al.*, 2013).

Encouraged by the potential formation of inorganic complexes, an iron precursor with an internal (counter-ion) base, $Fe[N(SiMe_3)_2]_2$, was used (Fig. 4). Thus, treating $Fe[N(SiMe_3)_2]_2$ with **L1** in THF afforded a yellow–green solution and, upon the removal of the volatiles, a dark-red residue was obtained. Single crystals suitable for X-ray crystallographic analysis were harvested from a saturated toluene solution stored at −35 °C (Table 1), and the structure was revealed to be the diiron complex **9** (Fig. 5; a depiction with complete atom labeling for Fe and N atoms is given in Fig. S1 in the supporting information). The X-ray crystal structure of **9** consists of a tetrahedral Fe^{II} ion and a trigonal planar Fe^{II} center bridged by two Me_2**L1**$^-$ units. Notably, **9** is a rare example of an asymmetric diiron complex. Fe1 adopts a distorted tetrahedral geometry (τ_4 = 0.69; Yang *et al.*, 2007), coordinated by one amine, one terminal amido, and two bridging amido N atoms. Fe2 has a distorted trigonal–planar coordination, as quantified by the sum of the angles [359.8 (2)°] around the metal, with a narrow N—Fe—N angle of 93.0 (1)° between two bridging amido N atoms (N4 and N7). The Fe—N_{amido} bond lengths of 1.947 (4)–2.153 (4) Å are shorter than that of Fe—N_{amine} [2.245 (3) Å]. Not surprisingly, the Fe—N_{amido} bond lengths in the μ_2-bridging amides (N4

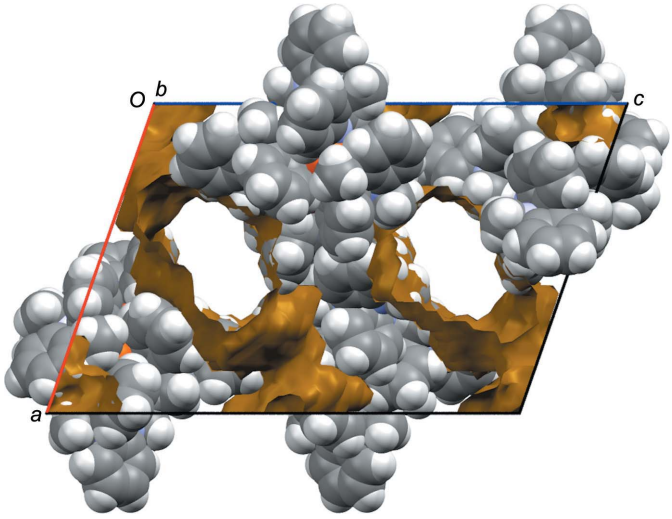


Figure 6
Solvent-accessible voids of 2479.0 Å^3 (*ca* 30% of the unit-cell volume) in the structure of diiron complex **9**.

and N7) are longer than those in the terminal ones (N1 and N9) (*vis-à-vis*, Bai *et al.*, 2017; Deschner *et al.*, 2011; Olmstead *et al.*, 1991). Additional selected bond lengths and angles are given in Table 2.

In addition, the solution magnetic moment of diiron complex **9** [7.7 (3) μ_B] agrees well with the relatively long Fe···Fe distance of 2.912 (1) Å (Malassa *et al.*, 2010; Frazier *et al.*, 2013), which may be indicative of no interaction between the dimetal core (the covalent radii sum for Fe—Fe is 2.48 Å).

Upon closer inspection of **9** we note that one of the **L1**$^-$ ligands coordinated to Fe2 is bound unsymmetrically. Besides the μ_2-bridging amides binding, there is a stabilizing *ipso* interaction between one of the anilide arms and the metal center. The geometry around atom N1 is distorted tetrahedral, with the six angles ranging from 85.6 (2) to 130.2 (3)°. The Fe2—C21 bond length is 2.425 (4) Å, more than 0.7 Å shorter than the distance to the *ipso* carbon on the other anilide arm [Fe2—C38 = 3.181 (5) Å]. In addition, the small Fe2—N4—C21 angle of 85.6 (2)° suggests the presence of a weak interaction between the $p(\pi)$ orbital on C_{ipso} and the Fe^{II} center. This *ipso* interaction is likely necessary to stabilize the highly electrophilic 16-electron Fe center. Such an interaction is often observed in early-transition or *f*-block metal complexes (Tonks *et al.*, 2012; Gountchev & Tilley, 1999; Müller–Buschbaum & Quitmann, 2003; Odom *et al.*, 1998; Vlaisavljevich *et al.*, 2013; Haftbaradaran *et al.*, 2005; Evans *et al.*, 1996; Berthet *et al.*, 2008; Arney *et al.*, 1992). To the best of our knowledge, compound **9** is the first structural report on an Fe^{II}···C_{ipso} interaction (Suess & Peters, 2013).

Significant solvent-accessible voids of 2479.0 Å^3, totalling *ca* 30% of the unit-cell volume, were observed in the structure of diiron complex **9** (see Fig. 6 and refinement details). One large and two smaller void areas are observed, located at the center of the unit cell, at the origin, and at the center of the *A*-face of the unit cell, with the latter two adjacent to each other. The electron density of the void area at the origin is partially

Table 2
Selected bond lengths (Å) and angles (°) for **9** and **10**.

Atoms are labeled as indicated in Figs. 5 and 7.

	9			**10**	
Fe1—N4	2.153 (4)	N4—Fe1—N7	89.8 (1)	Fe1—N1	2.009 (2)
Fe1—N7	2.091 (3)	N4—Fe1—N9	122.6 (1)	Fe1—N4	1.986 (2)
Fe1—N9	1.992 (4)	N4—Fe1—N10	115.7 (1)	Fe1—O1	2.093 (2)
Fe1—N10	2.245 (3)	N7—Fe1—N9	79.8 (1)	Fe1—O2	2.107 (2)
		N7—Fe1—N10	139.6 (1)		
Fe2—N1	1.947 (4)	N9—Fe1—N10	107.5 (1)	N1—Fe1—N4	126.64 (8)
Fe2—N4	2.068 (3)			O1—Fe1—O2	93.90 (7)
Fe2—N7	2.061 (4)	N1—Fe2—N4	143.6 (2)	N1—Fe1—O1	116.48 (8)
		N1—Fe2—N7	123.2 (2)	N1—Fe1—O2	97.67 (8)
Fe1···Fe2	2.912 (1)	N4—Fe2—N7	93.0 (1)	N4—Fe1—O1	100.46 (8)
Fe2···C21	2.425 (4)			N4—Fe1—O2	117.68 (8)
Fe2···C38	3.181 (5)				

resolved and could be interpreteted as mostly a pentane molecule disordered across an inversion center. The content of the larger and the second smaller void is highly disordered and no attempts were made to model it with disordered solvent molecules. The content of the solvent-accessible voids was estimated as *ca* 570 electrons, equivalent to 11.4 molecules of toluene per unit cell.

Dissolution of diiron complex **9** in THF results in solvation by two THF ligands and the formation of the simpler monoiron complex (**L1**)$_2$Fe(THF)$_2$ (**10**). The molecular structure and a partial numbering scheme of **10**, as determined by X-ray crystallography, is depicted in Fig. 7. Selected bond lengths and angles are given in Table 2. The C atoms of one of the two coordinated THF molecules are disordered, with a refined major occupancy of 0.790 (8). Disorder and complete atom labeling are given in an additional figure in the supporting information (Fig. S2 in the supporting information). The Fe atom, unaffected by the disorder, is in a distorted tetrahedral (τ_4 = 0.82) environment, with a significantly widened N—Fe—N bond angle of 126.64 (8)° due to steric and electrostatic repulsion between the bulky amide ions. As expected, the

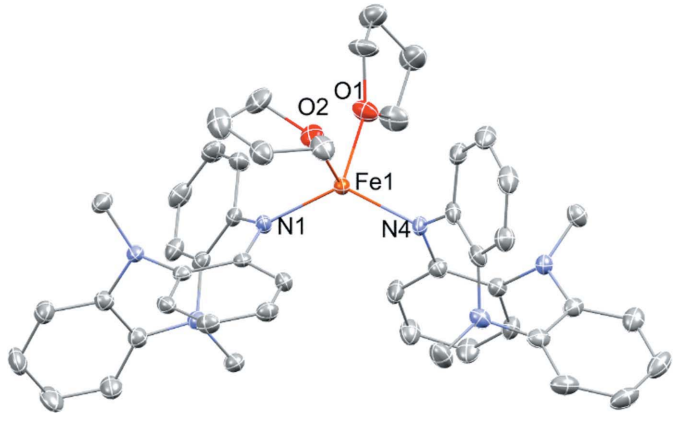


Figure 7
X-ray crystal structure of the monoiron complex **10**. Displacement ellipsoids are shown at the 50% probability level. Color key: orange = Fe, blue = N, gray = C, and red = O. H atoms, the disorder of the THF ligands, and most labels for C and N atoms have been omitted for clarity. A fully labeled displacement ellipsoid plot with disorder shown and H atoms included is given in the supporting information.

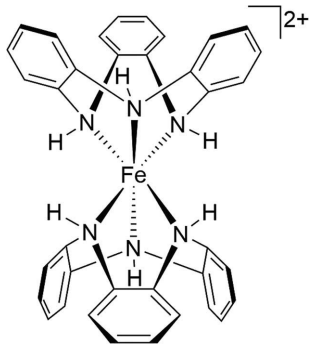


Figure 8
Calculated hypothetical structure (Foscato *et al.*, 2015) of the dicationic L_2Fe structure, where L = triazacyclophane **4**.

O1—Fe1—O2 angle between the two smaller ligands, 93.90 (7)°, is less than the N1—Fe1—N4 angle between the two large **L1**$^-$ groups. Comparable values were also observed for [(THF)$_2$Fe(SSi^{t}Bu$_3$)$_2$] with bulky trimethylsilyl thiolate ions. A lithium tris(diphenylamide)iron(II) complex was prepared previously by Francke & Francke (1988).

Iron(II) κ^3-coordinated by unmethylated **4** (Fig. 8) has been proposed from the results of DFT calculations. We were, however, unable to obtain complexes with this binding mode. A bowl-shaped crown conformation of the *ortho*-triazacyclophane ligand is essential for κ^3-coordination, but no crown conformer was observed in either diiron complex **9** or monoiron complex **10**, and only saddle conformers were observed. In **9**, all the ligands are coordinated to the Fe ions through only the unmethylated (anionic) amide N atom, while the methyl-substituted amine donors remain unbound. In **10**, one of the four ligands is coordinated in a chelating fashion involving the amide and one amine donor, but the remaining three ligands are again bound solely through their amide N atoms. An over-estimation in theoretical calculations for the degree of pyramidalization of the amine N atoms in ligands **L1** and **L2** could be responsible for the reluctance for this hypothetical complex to form (Foscato *et al.*, 2015). This hypothesis is corroborated by the absence of any substantial intermolecular interactions involving the amine N atoms. They do not act as acceptors for any of the C—H···N interactions. Indeed, intermolecular interactions in both **9** and **10** are sparse and directional interactions are limited to a number of weak C—H···π contacts, and shape recognition through van der Waals contacts.

Table 3
Bond orders for the Fe—Fe and all the Fe—N bonds in **9**.

Entry	Atom 1	Atom 2	Bond order
1	Fe1	Fe2	−0.113
2	Fe1	N4	0.342
3	Fe1	N7	0.296
4	Fe1	N9	0.253
5	Fe1	N10	0.564
6	Fe2	N1	0.556
7	Fe2	N4	0.303
8	Fe2	N7	0.383

3.3. Theoretical calculations

Bond orders in diiron complex **9** were calculated using *Spartan* at the density function level of theory using the EDF2 method with an 6-31G* basis set with heavy atoms frozen. Bond orders for Fe—Fe and for all Fe—N bonds are listed in Table 3. The Fe—Fe bond order is negative, indicating no metal–metal bond, and actually some repulsion.

For direct comparison of the X-ray crystal structure of diiron complex **9** with the calculated equilibrium geometry structure, the density functional (6-31G*) result with frozen heavy atoms was thawed and recalculated with the density functional level of theory and the EDF2 method and the 6-31G* basis set to yield a geometry-optimized structure. The key bond lengths for the X-ray crystal structure of the diiron complex are summarized in Fig. 9, alongside the distances calculated for the density functional (6-31G*) optimized structure. The Fe···Fe distance of 2.912 Å in the crystal structure (and further, the Fe···Fe distance in the geometry-optimized DF 6-31G* calculation of 3.012 Å) are longer than typical Fe—Fe bonding distances of 2.46–2.78 Å (Pauling, 1976), consistent with the calculated Fe—Fe bond order of −0.113, indicating no metal–metal bond.

Further, a density functional calculation using the wB97X-D method with the double-basis set 6-311+G(3df,2p)[6-311G*] revealed near monocationic charges on each of the Fe atoms, as well as providing HOMO/LUMO and spin-surface maps.

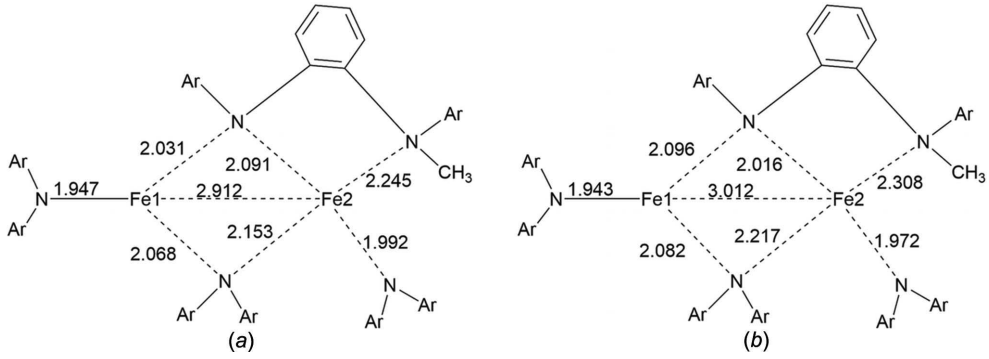


Figure 9
Key Fe bond distances in Ångströms for (*a*) the X-ray crystal structure of diiron complex **9** and (*b*) the density functional (6-31G*) optimized structure for **9** obtained by starting with the crystal structure and thawing the heavy atoms.

Specifically, these calculations revealed atomic charges of +0.998 for Fe1 and +0.961 for Fe2 in diiron complex **9**.

4. Summary

We describe an improved Buchwald–Hartwig macrocyclization reaction for the preparation of the *ortho*-triazacyclophane **L1**, which was deprotonated by the internal Fe^{II} base $Fe[N(SiMe_3)_2]_2$ to afford the unusual diiron complex **9**, as confirmed by X-ray crystallography. Diiron complex **9** was studied computationally, as well as at the density functional level of theory that, along with the longer distance between the Fe atoms, showed no evidence of metal–metal bonding. Treatment of diiron complex **9** with THF yielded the simpler $(\mathbf{L2})_2Fe(THF)_2$ complex **10**, which reverts to diiron complex **9** *in vacuo*.

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supporting information

Acta Cryst. (2018). C**74**, 1641-1649 [https://doi.org/10.1107/S2053229618015255]

Iron(II) complexes of dimethyltriazacyclophane

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Computing details

For both structures, data collection: *APEX2* (Bruker, 2014); cell refinement: *SAINT* (Bruker, 2014); data reduction: *SAINT* (Bruker, 2014); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015) and SHELXLE (Hübschle *et al.*, 2011); software used to prepare material for publication: *publCIF* (Westrip, 2010).

Bis(μ-1,4-dimethyltribenzo[*b*,*e*,*h*][1,4,7]triazacyclonona-2,5,8-trien-7-ido)-1:2κ^2*N*^{1},*N*^{7}:κ*N*^{7}; 1:2κ*N*^{7}:κ^2*N*^{7},*C*^{6}-bis[(μ-1,4-dimethyltribenzo[*b*,*e*,*h*][1,4,7]triazacyclonona-2,5,8-trien-7-ido-κ*N*^{7})iron(II)] (9)

Crystal data

$[Fe(C_{20}H_{18}N_3)_4]$
M_r = 1313.19
Monoclinic, $P2_1/c$
a = 21.758 (2) Å
b = 12.7682 (13) Å
c = 31.382 (3) Å
β = 109.191 (5)°
V = 8233.7 (14) Å^3
Z = 4
$F(000)$ = 2752
D_x = 1.059 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 8887 reflections
θ = 3.0–65.8°
μ = 3.17 mm^{-1}
T = 100 K
Rod, red
0.11 × 0.06 × 0.05 mm

Data collection

Bruker Prospector CCD diffractometer
Radiation source: I-mu-S microsource X-ray tube
Laterally graded multilayer (Goebel) mirror monochromator
ω and phi scans
Absorption correction: multi-scan (*APEX2*; Bruker, 2014)
T_{min} = 0.478, T_{max} = 0.753
52565 measured reflections
14191 independent reflections
8827 reflections with $I > 2\sigma(I)$
R_{int} = 0.133
θ_{max} = 66.9°, θ_{min} = 2.2°
h = −25→25
k = −14→15
l = −23→37

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.076
$wR(F^2)$ = 0.193
S = 1.03
14191 reflections
855 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

$w = 1/[\sigma^2(F_o^2) + (0.0816P)^2 + 5.4245P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.79$ e Å^{-3}
$\Delta\rho_{min} = -0.54$ 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. The structure contains solvent accessible voids of 2479.0 Ang3 (30% of the unit cell volume). The the residual electron density peaks were not arranged in an interpretable pattern. The hkl file was thus corrected using reverse Fourier transform methods using the SQUEEZE routine (P. van der Sluis & Spek (1990). Acta Cryst. A46, 194-201) as implemented in the program Platon. The resultant files were used in the further refinement. (The FAB file with details of the Squeeze results is appended to this cif file). The Squeeze procedure corrected for 569.8 electrons within the solvent accesible voids, equivalent of eight molecules of tetrahydro furane (the crystallization solvent).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.2847 (2)	0.3704 (4)	0.38404 (15)	0.0336 (11)
C2	0.2243 (2)	0.3887 (4)	0.35057 (15)	0.0339 (11)
H2	0.2097	0.3425	0.3255	0.041*
C3	0.1860 (2)	0.4723 (4)	0.35341 (17)	0.0418 (13)
H3	0.1453	0.4826	0.3304	0.050*
C4	0.2056 (3)	0.5413 (4)	0.38898 (18)	0.0509 (15)
H4	0.1784	0.5977	0.3914	0.061*
C5	0.2668 (2)	0.5262 (4)	0.42160 (17)	0.0462 (14)
H5	0.2819	0.5747	0.4457	0.055*
C6	0.3053 (2)	0.4428 (4)	0.41940 (16)	0.0367 (12)
C7	0.4093 (2)	0.5186 (4)	0.45291 (16)	0.0395 (12)
C8	0.4367 (2)	0.5861 (4)	0.48879 (17)	0.0457 (14)
H8	0.4317	0.5719	0.5172	0.055*
C9	0.4713 (3)	0.6743 (4)	0.48399 (19)	0.0510 (15)
H9	0.4911	0.7186	0.5091	0.061*
C10	0.4768 (3)	0.6969 (4)	0.4424 (2)	0.0552 (16)
H10	0.4971	0.7602	0.4383	0.066*
C11	0.4526 (2)	0.6272 (4)	0.40623 (18)	0.0487 (14)
H11	0.4584	0.6421	0.3781	0.058*
C12	0.4198 (2)	0.5352 (4)	0.41123 (16)	0.0398 (12)
C13	0.4224 (2)	0.3570 (4)	0.38532 (15)	0.0364 (12)
C14	0.4872 (2)	0.3433 (4)	0.39039 (16)	0.0413 (12)
H14	0.5136	0.4031	0.3915	0.050*
C15	0.5148 (3)	0.2450 (4)	0.39382 (17)	0.0494 (14)
H15	0.5597	0.2367	0.3975	0.059*
C16	0.4750 (2)	0.1579 (4)	0.39176 (16)	0.0446 (13)
H16	0.4925	0.0893	0.3932	0.053*
C17	0.4108 (2)	0.1712 (4)	0.38768 (15)	0.0362 (11)
H17	0.3846	0.1109	0.3861	0.043*
C18	0.3823 (2)	0.2705 (4)	0.38573 (15)	0.0336 (11)

C19	0.3620 (2)	0.4097 (4)	0.49871 (16)	0.0444 (13)
H19A	0.3348	0.3473	0.4966	0.067*
H19B	0.3417	0.4697	0.5083	0.067*
H19C	0.4052	0.3973	0.5208	0.067*
C20	0.3840 (3)	0.4952 (4)	0.33024 (16)	0.0550 (15)
H20A	0.3566	0.4434	0.3095	0.083*
H20B	0.4257	0.5011	0.3249	0.083*
H20C	0.3620	0.5633	0.3251	0.083*
C21	0.1671 (2)	0.1208 (3)	0.31601 (14)	0.0284 (10)
C22	0.1206 (2)	0.1744 (3)	0.32995 (14)	0.0285 (10)
H22	0.1141	0.1550	0.3574	0.034*
C23	0.0841 (2)	0.2552 (3)	0.30420 (15)	0.0304 (10)
H23	0.0532	0.2909	0.3143	0.036*
C24	0.0924 (2)	0.2842 (4)	0.26395 (15)	0.0334 (11)
H24	0.0662	0.3374	0.2457	0.040*
C25	0.1398 (2)	0.2342 (4)	0.25073 (15)	0.0320 (11)
H25	0.1467	0.2555	0.2237	0.038*
C26	0.1770 (2)	0.1544 (4)	0.27575 (14)	0.0313 (11)
C27	0.2018 (2)	0.0586 (4)	0.21864 (15)	0.0380 (12)
C28	0.2236 (3)	0.0819 (4)	0.18263 (17)	0.0528 (15)
H28	0.2593	0.1282	0.1870	0.063*
C29	0.1935 (3)	0.0383 (5)	0.14064 (18)	0.0631 (18)
H29	0.2095	0.0540	0.1166	0.076*
C30	0.1410 (3)	−0.0274 (5)	0.13302 (17)	0.0627 (18)
H30	0.1192	−0.0539	0.1036	0.075*
C31	0.1202 (3)	−0.0548 (4)	0.16902 (15)	0.0470 (14)
H31	0.0844	−0.1014	0.1641	0.056*
C32	0.1507 (2)	−0.0152 (4)	0.21207 (15)	0.0376 (12)
C33	0.1904 (2)	−0.0967 (4)	0.28417 (15)	0.0356 (11)
C34	0.2103 (3)	−0.1934 (4)	0.27291 (17)	0.0436 (13)
H34	0.1873	−0.2231	0.2443	0.052*
C35	0.2626 (3)	−0.2482 (4)	0.30196 (19)	0.0604 (17)
H35	0.2749	−0.3147	0.2939	0.072*
C36	0.2962 (3)	−0.2022 (5)	0.34327 (18)	0.0571 (16)
H36	0.3320	−0.2378	0.3638	0.069*
C37	0.2781 (2)	−0.1059 (4)	0.35466 (15)	0.0386 (12)
H37	0.3031	−0.0749	0.3825	0.046*
C38	0.2235 (2)	−0.0516 (4)	0.32614 (14)	0.0301 (10)
C39	0.2869 (2)	0.1660 (4)	0.27151 (16)	0.0429 (13)
H39A	0.3206	0.1233	0.2655	0.064*
H39B	0.3015	0.1867	0.3034	0.064*
H39C	0.2788	0.2288	0.2525	0.064*
C40	0.0735 (2)	−0.0973 (4)	0.24384 (15)	0.0366 (12)
H40A	0.0389	−0.0560	0.2226	0.055*
H40B	0.0650	−0.1033	0.2726	0.055*
H40C	0.0745	−0.1674	0.2314	0.055*
C41	0.2244 (2)	0.1888 (4)	0.46888 (15)	0.0305 (11)
C42	0.1949 (2)	0.2835 (4)	0.45118 (16)	0.0345 (11)

H42	0.1842	0.2963	0.4198	0.041*
C43	0.1809 (3)	0.3585 (4)	0.47771 (19)	0.0470 (13)
H43	0.1602	0.4217	0.4646	0.056*
C44	0.1968 (3)	0.3425 (4)	0.5234 (2)	0.0585 (16)
H44	0.1886	0.3953	0.5422	0.070*
C45	0.2250 (3)	0.2480 (4)	0.54172 (17)	0.0469 (14)
H45	0.2361	0.2370	0.5733	0.056*
C46	0.2375 (2)	0.1695 (4)	0.51516 (15)	0.0356 (11)
C47	0.2139 (2)	−0.0121 (4)	0.52683 (15)	0.0350 (11)
C48	0.1787 (2)	−0.0102 (4)	0.55643 (16)	0.0427 (12)
H48	0.1885	0.0425	0.5791	0.051*
C49	0.1303 (3)	−0.0812 (5)	0.55451 (17)	0.0517 (14)
H49	0.1075	−0.0789	0.5757	0.062*
C50	0.1159 (3)	−0.1555 (4)	0.52102 (17)	0.0467 (13)
H50	0.0826	−0.2054	0.5189	0.056*
C51	0.1492 (2)	−0.1588 (4)	0.49038 (16)	0.0427 (13)
H51	0.1375	−0.2096	0.4670	0.051*
C52	0.1998 (2)	−0.0884 (4)	0.49323 (14)	0.0319 (11)
C53	0.2967 (2)	−0.0500 (4)	0.47170 (15)	0.0334 (11)
C54	0.3533 (2)	−0.1058 (4)	0.49256 (15)	0.0400 (12)
H54	0.3512	−0.1785	0.4984	0.048*
C55	0.4128 (2)	−0.0553 (4)	0.50488 (17)	0.0474 (14)
H55	0.4515	−0.0936	0.5192	0.057*
C56	0.4163 (2)	0.0504 (4)	0.49650 (17)	0.0459 (13)
H56	0.4573	0.0849	0.5053	0.055*
C57	0.3599 (2)	0.1058 (4)	0.47530 (15)	0.0371 (12)
H57	0.3625	0.1784	0.4694	0.044*
C58	0.2997 (2)	0.0570 (4)	0.46250 (14)	0.0309 (11)
C59	0.3147 (2)	0.0667 (4)	0.57586 (15)	0.0479 (14)
H59A	0.3490	0.1159	0.5752	0.058*
H59B	0.3329	−0.0041	0.5817	0.058*
H59C	0.2973	0.0870	0.5998	0.058*
C60	0.2344 (3)	−0.2094 (4)	0.44436 (16)	0.0427 (13)
H60A	0.1900	−0.2332	0.4282	0.064*
H60B	0.2536	−0.2539	0.4709	0.064*
H60C	0.2607	−0.2139	0.4244	0.064*
C61	0.0408 (2)	0.0432 (3)	0.39382 (13)	0.0273 (10)
C62	0.0637 (2)	0.1056 (4)	0.43271 (14)	0.0309 (11)
H62	0.1019	0.0843	0.4561	0.037*
C63	0.0333 (2)	0.1961 (3)	0.43842 (14)	0.0313 (11)
H63	0.0513	0.2370	0.4649	0.038*
C64	−0.0238 (2)	0.2280 (4)	0.40555 (15)	0.0342 (11)
H64	−0.0452	0.2909	0.4088	0.041*
C65	−0.0486 (2)	0.1649 (3)	0.36772 (14)	0.0291 (10)
H65	−0.0884	0.1846	0.3455	0.035*
C66	−0.0181 (2)	0.0751 (3)	0.36100 (13)	0.0257 (10)
C67	−0.1073 (2)	−0.0313 (4)	0.31871 (14)	0.0325 (11)
C68	−0.1663 (2)	−0.0093 (4)	0.28458 (15)	0.0372 (12)

H68	−0.1667	0.0356	0.2604	0.045*
C69	−0.2235 (2)	−0.0522 (4)	0.28602 (16)	0.0427 (13)
H69	−0.2631	−0.0365	0.2628	0.051*
C70	−0.2242 (2)	−0.1180 (4)	0.32079 (16)	0.0411 (12)
H70	−0.2641	−0.1455	0.3221	0.049*
C71	−0.1657 (2)	−0.1435 (4)	0.35389 (15)	0.0348 (11)
H71	−0.1658	−0.1907	0.3773	0.042*
C72	−0.1073 (2)	−0.1012 (4)	0.35337 (14)	0.0317 (11)
C73	−0.0028 (2)	−0.1795 (4)	0.36796 (14)	0.0320 (11)
C74	−0.0188 (2)	−0.2791 (4)	0.34968 (15)	0.0356 (11)
H74	−0.0590	−0.3087	0.3496	0.043*
C75	0.0201 (2)	−0.3376 (4)	0.33164 (15)	0.0393 (12)
H75	0.0075	−0.4055	0.3195	0.047*
C76	0.0781 (2)	−0.2930 (4)	0.33209 (14)	0.0356 (11)
H76	0.1059	−0.3306	0.3197	0.043*
C77	0.0964 (2)	−0.1947 (4)	0.35019 (14)	0.0325 (11)
H77	0.1368	−0.1667	0.3501	0.039*
C78	0.0569 (2)	−0.1339 (3)	0.36900 (13)	0.0280 (10)
C79	−0.0450 (2)	0.0685 (4)	0.27994 (14)	0.0354 (11)
H79A	−0.0621	0.0224	0.2537	0.053*
H79B	0.0001	0.0873	0.2835	0.053*
H79C	−0.0715	0.1321	0.2754	0.053*
C80	−0.0478 (2)	−0.1707 (4)	0.42861 (15)	0.0432 (13)
H80A	−0.0658	−0.2417	0.4235	0.065*
H80B	−0.0751	−0.1268	0.4408	0.065*
H80C	−0.0035	−0.1731	0.4502	0.065*
Fe1	0.17367 (3)	0.00329 (6)	0.40199 (2)	0.02762 (18)
Fe2	0.25881 (3)	0.15927 (6)	0.38298 (2)	0.03122 (19)
N1	0.31703 (17)	0.2743 (3)	0.38190 (12)	0.0315 (9)
N2	0.36834 (18)	0.4317 (3)	0.45443 (13)	0.0384 (10)
N3	0.39530 (19)	0.4622 (3)	0.37670 (13)	0.0399 (10)
N4	0.20378 (16)	0.0392 (3)	0.34477 (11)	0.0277 (8)
N5	0.22650 (18)	0.1048 (3)	0.26126 (12)	0.0374 (10)
N6	0.13593 (18)	−0.0458 (3)	0.25134 (12)	0.0330 (9)
N7	0.24007 (17)	0.1133 (3)	0.44047 (11)	0.0292 (9)
N8	0.26247 (18)	0.0687 (3)	0.53236 (12)	0.0344 (9)
N9	0.23263 (18)	−0.0987 (3)	0.45908 (11)	0.0317 (9)
N10	0.08062 (16)	−0.0371 (3)	0.38772 (11)	0.0268 (8)
N11	−0.04708 (17)	0.0144 (3)	0.32030 (11)	0.0298 (9)
N12	−0.04644 (17)	−0.1268 (3)	0.38618 (11)	0.0282 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.028 (2)	0.033 (3)	0.035 (3)	−0.002 (2)	0.004 (2)	0.002 (2)
C2	0.034 (3)	0.026 (3)	0.035 (3)	−0.001 (2)	0.002 (2)	0.001 (2)
C3	0.031 (3)	0.034 (3)	0.046 (3)	0.003 (2)	−0.007 (2)	−0.001 (2)
C4	0.039 (3)	0.038 (3)	0.061 (4)	0.016 (2)	−0.004 (3)	−0.006 (3)

C5	0.040 (3)	0.042 (3)	0.046 (3)	0.004 (2)	−0.001 (2)	−0.009 (2)
C6	0.022 (2)	0.040 (3)	0.041 (3)	0.005 (2)	0.000 (2)	0.003 (2)
C7	0.029 (3)	0.035 (3)	0.041 (3)	0.007 (2)	−0.007 (2)	0.005 (2)
C8	0.036 (3)	0.043 (3)	0.045 (3)	0.007 (3)	−0.004 (2)	−0.004 (2)
C9	0.039 (3)	0.045 (3)	0.048 (3)	0.003 (3)	−0.014 (2)	−0.006 (3)
C10	0.040 (3)	0.043 (3)	0.063 (4)	−0.003 (3)	−0.010 (3)	0.003 (3)
C11	0.034 (3)	0.051 (3)	0.046 (3)	−0.004 (3)	−0.008 (2)	0.009 (3)
C12	0.033 (3)	0.038 (3)	0.035 (3)	0.005 (2)	−0.007 (2)	0.004 (2)
C13	0.027 (2)	0.044 (3)	0.032 (3)	0.001 (2)	0.0016 (19)	0.002 (2)
C14	0.036 (3)	0.050 (3)	0.036 (3)	−0.007 (3)	0.010 (2)	0.002 (2)
C15	0.032 (3)	0.063 (4)	0.051 (3)	0.007 (3)	0.011 (2)	0.005 (3)
C16	0.038 (3)	0.055 (4)	0.040 (3)	0.012 (3)	0.012 (2)	−0.002 (3)
C17	0.032 (3)	0.042 (3)	0.032 (3)	0.000 (2)	0.007 (2)	−0.001 (2)
C18	0.031 (3)	0.038 (3)	0.029 (2)	0.001 (2)	0.0057 (19)	0.003 (2)
C19	0.038 (3)	0.050 (3)	0.036 (3)	0.002 (3)	−0.001 (2)	−0.007 (2)
C20	0.059 (4)	0.058 (4)	0.034 (3)	−0.005 (3)	−0.003 (2)	0.011 (3)
C21	0.026 (2)	0.028 (2)	0.025 (2)	0.000 (2)	0.0004 (18)	−0.0003 (19)
C22	0.026 (2)	0.035 (3)	0.025 (2)	−0.003 (2)	0.0080 (18)	−0.001 (2)
C23	0.024 (2)	0.027 (3)	0.039 (3)	0.0000 (19)	0.0082 (19)	−0.003 (2)
C24	0.024 (2)	0.036 (3)	0.036 (3)	−0.003 (2)	0.0033 (19)	0.008 (2)
C25	0.026 (2)	0.040 (3)	0.027 (2)	−0.004 (2)	0.0046 (19)	0.003 (2)
C26	0.024 (2)	0.042 (3)	0.026 (2)	−0.007 (2)	0.0050 (18)	−0.005 (2)
C27	0.038 (3)	0.050 (3)	0.027 (3)	0.002 (2)	0.012 (2)	0.001 (2)
C28	0.067 (4)	0.062 (4)	0.036 (3)	−0.006 (3)	0.025 (3)	−0.007 (3)
C29	0.093 (5)	0.073 (4)	0.030 (3)	−0.016 (4)	0.030 (3)	−0.007 (3)
C30	0.095 (5)	0.070 (4)	0.020 (3)	−0.008 (4)	0.015 (3)	−0.008 (3)
C31	0.067 (4)	0.041 (3)	0.028 (3)	−0.002 (3)	0.008 (2)	−0.006 (2)
C32	0.041 (3)	0.043 (3)	0.024 (2)	0.010 (2)	0.005 (2)	0.002 (2)
C33	0.036 (3)	0.035 (3)	0.034 (3)	0.003 (2)	0.009 (2)	−0.005 (2)
C34	0.047 (3)	0.040 (3)	0.039 (3)	0.011 (3)	0.007 (2)	−0.005 (2)
C35	0.067 (4)	0.048 (4)	0.057 (4)	0.029 (3)	0.008 (3)	−0.014 (3)
C36	0.054 (4)	0.064 (4)	0.046 (3)	0.026 (3)	0.007 (3)	−0.001 (3)
C37	0.035 (3)	0.048 (3)	0.030 (3)	0.012 (2)	0.007 (2)	0.000 (2)
C38	0.025 (2)	0.040 (3)	0.027 (2)	0.006 (2)	0.0103 (18)	0.002 (2)
C39	0.028 (3)	0.064 (4)	0.037 (3)	−0.002 (3)	0.012 (2)	−0.002 (3)
C40	0.039 (3)	0.035 (3)	0.031 (3)	0.002 (2)	0.004 (2)	0.000 (2)
C41	0.022 (2)	0.037 (3)	0.033 (3)	−0.002 (2)	0.0090 (18)	−0.008 (2)
C42	0.035 (3)	0.035 (3)	0.036 (3)	0.000 (2)	0.016 (2)	−0.003 (2)
C43	0.050 (3)	0.037 (3)	0.059 (4)	0.000 (3)	0.024 (3)	−0.011 (3)
C44	0.073 (4)	0.042 (4)	0.072 (4)	−0.008 (3)	0.039 (3)	−0.023 (3)
C45	0.064 (4)	0.047 (3)	0.036 (3)	−0.007 (3)	0.025 (3)	−0.014 (3)
C46	0.038 (3)	0.035 (3)	0.035 (3)	−0.010 (2)	0.013 (2)	−0.013 (2)
C47	0.039 (3)	0.035 (3)	0.027 (2)	0.001 (2)	0.006 (2)	0.005 (2)
C48	0.047 (3)	0.050 (3)	0.035 (3)	−0.008 (3)	0.019 (2)	−0.003 (2)
C49	0.058 (4)	0.067 (4)	0.037 (3)	−0.008 (3)	0.025 (3)	−0.001 (3)
C50	0.044 (3)	0.055 (4)	0.042 (3)	−0.007 (3)	0.015 (2)	0.011 (3)
C51	0.041 (3)	0.045 (3)	0.034 (3)	−0.003 (3)	0.001 (2)	−0.001 (2)
C52	0.034 (3)	0.036 (3)	0.023 (2)	−0.001 (2)	0.0043 (19)	0.003 (2)

C53	0.029 (2)	0.043 (3)	0.029 (2)	0.007 (2)	0.0106 (19)	0.000 (2)
C54	0.043 (3)	0.044 (3)	0.033 (3)	0.009 (2)	0.013 (2)	0.010 (2)
C55	0.024 (3)	0.058 (4)	0.054 (3)	0.012 (3)	0.004 (2)	0.012 (3)
C56	0.032 (3)	0.054 (4)	0.050 (3)	0.001 (3)	0.010 (2)	0.006 (3)
C57	0.029 (3)	0.049 (3)	0.031 (3)	0.006 (2)	0.006 (2)	0.006 (2)
C58	0.028 (2)	0.037 (3)	0.029 (2)	0.005 (2)	0.0102 (19)	−0.003 (2)
C59	0.045 (3)	0.067 (4)	0.027 (3)	−0.012 (3)	0.006 (2)	−0.007 (3)
C60	0.053 (3)	0.033 (3)	0.035 (3)	0.003 (2)	0.004 (2)	−0.002 (2)
C61	0.027 (2)	0.034 (3)	0.021 (2)	−0.002 (2)	0.0083 (18)	−0.0014 (19)
C62	0.029 (2)	0.036 (3)	0.027 (2)	0.000 (2)	0.0081 (19)	0.002 (2)
C63	0.040 (3)	0.030 (3)	0.026 (2)	−0.008 (2)	0.013 (2)	−0.007 (2)
C64	0.033 (3)	0.033 (3)	0.035 (3)	0.005 (2)	0.009 (2)	0.000 (2)
C65	0.033 (2)	0.030 (3)	0.024 (2)	0.002 (2)	0.0077 (18)	0.005 (2)
C66	0.026 (2)	0.028 (3)	0.022 (2)	−0.0020 (19)	0.0065 (17)	0.0036 (19)
C67	0.030 (2)	0.042 (3)	0.024 (2)	0.003 (2)	0.0069 (18)	−0.004 (2)
C68	0.033 (3)	0.046 (3)	0.028 (2)	0.009 (2)	0.0038 (19)	0.000 (2)
C69	0.027 (3)	0.066 (4)	0.030 (3)	0.008 (3)	0.003 (2)	−0.004 (3)
C70	0.027 (2)	0.055 (3)	0.040 (3)	−0.001 (2)	0.008 (2)	−0.010 (3)
C71	0.031 (3)	0.045 (3)	0.029 (2)	0.001 (2)	0.011 (2)	0.000 (2)
C72	0.028 (2)	0.038 (3)	0.024 (2)	0.000 (2)	0.0025 (18)	−0.005 (2)
C73	0.033 (3)	0.035 (3)	0.024 (2)	0.007 (2)	0.0031 (19)	0.002 (2)
C74	0.030 (3)	0.034 (3)	0.037 (3)	−0.003 (2)	0.003 (2)	0.003 (2)
C75	0.040 (3)	0.040 (3)	0.032 (3)	0.006 (2)	0.004 (2)	−0.007 (2)
C76	0.035 (3)	0.041 (3)	0.027 (2)	0.013 (2)	0.0050 (19)	−0.002 (2)
C77	0.030 (2)	0.040 (3)	0.024 (2)	0.005 (2)	0.0035 (18)	0.001 (2)
C78	0.029 (2)	0.033 (3)	0.020 (2)	0.006 (2)	0.0051 (18)	0.0056 (19)
C79	0.037 (3)	0.042 (3)	0.024 (2)	0.001 (2)	0.007 (2)	0.003 (2)
C80	0.038 (3)	0.056 (3)	0.032 (3)	0.004 (3)	0.007 (2)	0.009 (2)
Fe1	0.0246 (3)	0.0327 (4)	0.0226 (3)	0.0008 (3)	0.0037 (3)	−0.0012 (3)
Fe2	0.0281 (4)	0.0352 (4)	0.0279 (4)	−0.0007 (3)	0.0058 (3)	−0.0004 (3)
N1	0.0237 (19)	0.030 (2)	0.037 (2)	−0.0012 (17)	0.0041 (16)	0.0004 (17)
N2	0.026 (2)	0.043 (3)	0.035 (2)	0.0028 (19)	−0.0040 (17)	0.0025 (19)
N3	0.037 (2)	0.038 (2)	0.034 (2)	−0.0011 (19)	−0.0020 (18)	0.0014 (19)
N4	0.0224 (18)	0.036 (2)	0.0243 (19)	0.0062 (16)	0.0071 (15)	−0.0028 (16)
N5	0.031 (2)	0.050 (3)	0.030 (2)	−0.0004 (19)	0.0092 (17)	0.0023 (19)
N6	0.032 (2)	0.037 (2)	0.025 (2)	0.0025 (18)	0.0015 (16)	0.0009 (17)
N7	0.0236 (19)	0.036 (2)	0.026 (2)	0.0035 (17)	0.0058 (15)	−0.0040 (17)
N8	0.032 (2)	0.042 (2)	0.025 (2)	−0.0081 (19)	0.0049 (16)	−0.0054 (18)
N9	0.036 (2)	0.031 (2)	0.0237 (19)	−0.0001 (18)	0.0042 (16)	−0.0003 (17)
N10	0.0184 (17)	0.029 (2)	0.028 (2)	−0.0028 (16)	0.0013 (14)	−0.0057 (16)
N11	0.031 (2)	0.033 (2)	0.0239 (19)	0.0048 (17)	0.0070 (15)	0.0022 (16)
N12	0.0223 (18)	0.034 (2)	0.0244 (19)	0.0018 (16)	0.0031 (15)	0.0019 (16)

Geometric parameters (Å, º)

C1—C6	1.401 (6)	C42—H42	0.9500
C1—C2	1.406 (6)	C43—C44	1.375 (8)
C1—N1	1.426 (6)	C43—H43	0.9500

C2—C3	1.376 (6)	C44—C45	1.390 (7)
C2—H2	0.9500	C44—H44	0.9500
C3—C4	1.375 (7)	C45—C46	1.386 (6)
C3—H3	0.9500	C45—H45	0.9500
C4—C5	1.401 (6)	C46—N8	1.432 (6)
C4—H4	0.9500	C47—C48	1.384 (6)
C5—C6	1.370 (7)	C47—C52	1.394 (6)
C5—H5	0.9500	C47—N8	1.446 (6)
C6—N2	1.456 (5)	C48—C49	1.378 (7)
C7—C8	1.388 (7)	C48—H48	0.9500
C7—C12	1.416 (7)	C49—C50	1.373 (7)
C7—N2	1.434 (6)	C49—H49	0.9500
C8—C9	1.391 (7)	C50—C51	1.381 (7)
C8—H8	0.9500	C50—H50	0.9500
C9—C10	1.378 (8)	C51—C52	1.401 (7)
C9—H9	0.9500	C51—H51	0.9500
C10—C11	1.403 (7)	C52—N9	1.476 (6)
C10—H10	0.9500	C53—C54	1.386 (6)
C11—C12	1.410 (7)	C53—C58	1.401 (6)
C11—H11	0.9500	C53—N9	1.458 (6)
C12—N3	1.395 (6)	C54—C55	1.382 (7)
C13—C14	1.377 (7)	C54—H54	0.9500
C13—C18	1.411 (7)	C55—C56	1.382 (7)
C13—N3	1.456 (6)	C55—H55	0.9500
C14—C15	1.381 (7)	C56—C57	1.383 (6)
C14—H14	0.9500	C56—H56	0.9500
C15—C16	1.397 (7)	C57—C58	1.385 (6)
C15—H15	0.9500	C57—H57	0.9500
C16—C17	1.372 (7)	C58—N7	1.446 (5)
C16—H16	0.9500	C59—N8	1.461 (5)
C17—C18	1.403 (6)	C59—H59A	0.9800
C17—H17	0.9500	C59—H59B	0.9800
C18—N1	1.386 (6)	C59—H59C	0.9800
C19—N2	1.468 (6)	C60—N9	1.492 (6)
C19—H19A	0.9800	C60—H60A	0.9800
C19—H19B	0.9800	C60—H60B	0.9800
C19—H19C	0.9800	C60—H60C	0.9800
C20—N3	1.459 (6)	C61—N10	1.395 (5)
C20—H20A	0.9800	C61—C62	1.405 (6)
C20—H20B	0.9800	C61—C66	1.414 (6)
C20—H20C	0.9800	C62—C63	1.372 (6)
C21—C22	1.404 (6)	C62—H62	0.9500
C21—C26	1.417 (6)	C63—C64	1.389 (6)
C21—N4	1.436 (5)	C63—H63	0.9500
C21—Fe2	2.426 (4)	C64—C65	1.389 (6)
C22—C23	1.388 (6)	C64—H64	0.9500
C22—H22	0.9500	C65—C66	1.375 (6)
C23—C24	1.384 (6)	C65—H65	0.9500

C23—H23	0.9500	C66—N11	1.449 (5)
C24—C25	1.386 (6)	C67—C68	1.403 (6)
C24—H24	0.9500	C67—C72	1.407 (6)
C25—C26	1.376 (6)	C67—N11	1.421 (6)
C25—H25	0.9500	C68—C69	1.375 (7)
C26—N5	1.445 (6)	C68—H68	0.9500
C27—C28	1.394 (7)	C69—C70	1.381 (7)
C27—N5	1.398 (6)	C69—H69	0.9500
C27—C32	1.419 (7)	C70—C71	1.391 (6)
C28—C29	1.381 (7)	C70—H70	0.9500
C28—H28	0.9500	C71—C72	1.387 (6)
C29—C30	1.373 (8)	C71—H71	0.9500
C29—H29	0.9500	C72—N12	1.422 (5)
C30—C31	1.393 (7)	C73—C74	1.392 (6)
C30—H30	0.9500	C73—C78	1.414 (6)
C31—C32	1.390 (6)	C73—N12	1.428 (6)
C31—H31	0.9500	C74—C75	1.383 (7)
C32—N6	1.427 (6)	C74—H74	0.9500
C33—C34	1.392 (6)	C75—C76	1.381 (7)
C33—C38	1.401 (6)	C75—H75	0.9500
C33—N6	1.444 (6)	C76—C77	1.382 (6)
C34—C35	1.390 (7)	C76—H76	0.9500
C34—H34	0.9500	C77—C78	1.423 (6)
C35—C36	1.392 (7)	C77—H77	0.9500
C35—H35	0.9500	C78—N10	1.393 (5)
C36—C37	1.373 (7)	C79—N11	1.456 (5)
C36—H36	0.9500	C79—H79A	0.9800
C37—C38	1.413 (6)	C79—H79B	0.9800
C37—H37	0.9500	C79—H79C	0.9800
C38—N4	1.426 (6)	C80—N12	1.454 (6)
C39—N5	1.470 (6)	C80—H80A	0.9800
C39—H39A	0.9800	C80—H80B	0.9800
C39—H39B	0.9800	C80—H80C	0.9800
C39—H39C	0.9800	Fe1—N10	1.992 (3)
C40—N6	1.458 (6)	Fe1—N7	2.091 (4)
C40—H40A	0.9800	Fe1—N4	2.154 (4)
C40—H40B	0.9800	Fe1—N9	2.245 (3)
C40—H40C	0.9800	Fe1—Fe2	2.9121 (10)
C41—C42	1.396 (6)	Fe2—N1	1.947 (4)
C41—C46	1.407 (6)	Fe2—N7	2.061 (4)
C41—N7	1.429 (5)	Fe2—N4	2.068 (4)
C42—C43	1.367 (6)		

C6—C1—C2	117.3 (4)	C50—C51—H51	119.4
C6—C1—N1	124.8 (4)	C52—C51—H51	119.4
C2—C1—N1	117.5 (4)	C47—C52—C51	118.0 (5)
C3—C2—C1	121.2 (4)	C47—C52—N9	124.9 (4)
C3—C2—H2	119.4	C51—C52—N9	117.0 (4)

C1—C2—H2	119.4	C54—C53—C58	120.2 (5)
C4—C3—C2	121.1 (4)	C54—C53—N9	122.0 (4)
C4—C3—H3	119.4	C58—C53—N9	117.7 (4)
C2—C3—H3	119.4	C55—C54—C53	119.8 (5)
C3—C4—C5	118.1 (5)	C55—C54—H54	120.1
C3—C4—H4	120.9	C53—C54—H54	120.1
C5—C4—H4	120.9	C56—C55—C54	120.5 (5)
C6—C5—C4	121.4 (5)	C56—C55—H55	119.8
C6—C5—H5	119.3	C54—C55—H55	119.8
C4—C5—H5	119.3	C55—C56—C57	119.7 (5)
C5—C6—C1	120.8 (4)	C55—C56—H56	120.2
C5—C6—N2	118.6 (4)	C57—C56—H56	120.2
C1—C6—N2	120.7 (4)	C56—C57—C58	120.9 (5)
C8—C7—C12	120.0 (5)	C56—C57—H57	119.5
C8—C7—N2	124.0 (5)	C58—C57—H57	119.5
C12—C7—N2	115.9 (4)	C57—C58—C53	118.9 (4)
C7—C8—C9	121.2 (5)	C57—C58—N7	122.0 (4)
C7—C8—H8	119.4	C53—C58—N7	119.2 (4)
C9—C8—H8	119.4	N8—C59—H59A	109.5
C10—C9—C8	119.4 (5)	N8—C59—H59B	109.5
C10—C9—H9	120.3	H59A—C59—H59B	109.5
C8—C9—H9	120.3	N8—C59—H59C	109.5
C9—C10—C11	120.5 (5)	H59A—C59—H59C	109.5
C9—C10—H10	119.8	H59B—C59—H59C	109.5
C11—C10—H10	119.8	N9—C60—H60A	109.5
C10—C11—C12	120.5 (5)	N9—C60—H60B	109.5
C10—C11—H11	119.7	H60A—C60—H60B	109.5
C12—C11—H11	119.7	N9—C60—H60C	109.5
N3—C12—C11	123.3 (5)	H60A—C60—H60C	109.5
N3—C12—C7	118.7 (4)	H60B—C60—H60C	109.5
C11—C12—C7	118.0 (5)	N10—C61—C62	118.5 (4)
C14—C13—C18	120.8 (5)	N10—C61—C66	124.5 (4)
C14—C13—N3	118.4 (4)	C62—C61—C66	116.5 (4)
C18—C13—N3	120.7 (4)	C63—C62—C61	122.9 (4)
C13—C14—C15	121.9 (5)	C63—C62—H62	118.5
C13—C14—H14	119.1	C61—C62—H62	118.5
C15—C14—H14	119.1	C62—C63—C64	120.1 (4)
C14—C15—C16	118.2 (5)	C62—C63—H63	120.0
C14—C15—H15	120.9	C64—C63—H63	120.0
C16—C15—H15	120.9	C65—C64—C63	117.8 (4)
C17—C16—C15	120.1 (5)	C65—C64—H64	121.1
C17—C16—H16	119.9	C63—C64—H64	121.1
C15—C16—H16	119.9	C66—C65—C64	122.9 (4)
C16—C17—C18	122.6 (5)	C66—C65—H65	118.6
C16—C17—H17	118.7	C64—C65—H65	118.6
C18—C17—H17	118.7	C65—C66—C61	119.8 (4)
N1—C18—C17	117.5 (4)	C65—C66—N11	119.2 (4)
N1—C18—C13	126.2 (4)	C61—C66—N11	121.0 (4)

C17—C18—C13	116.2 (4)	C68—C67—C72	119.0 (4)
N2—C19—H19A	109.5	C68—C67—N11	123.1 (4)
N2—C19—H19B	109.5	C72—C67—N11	117.9 (4)
H19A—C19—H19B	109.5	C69—C68—C67	120.4 (5)
N2—C19—H19C	109.5	C69—C68—H68	119.8
H19A—C19—H19C	109.5	C67—C68—H68	119.8
H19B—C19—H19C	109.5	C68—C69—C70	120.9 (4)
N3—C20—H20A	109.5	C68—C69—H69	119.5
N3—C20—H20B	109.5	C70—C69—H69	119.5
H20A—C20—H20B	109.5	C69—C70—C71	119.2 (5)
N3—C20—H20C	109.5	C69—C70—H70	120.4
H20A—C20—H20C	109.5	C71—C70—H70	120.4
H20B—C20—H20C	109.5	C72—C71—C70	121.2 (5)
C22—C21—C26	117.8 (4)	C72—C71—H71	119.4
C22—C21—N4	117.8 (4)	C70—C71—H71	119.4
C26—C21—N4	124.3 (4)	C71—C72—C67	119.2 (4)
C22—C21—Fe2	95.9 (3)	C71—C72—N12	122.8 (4)
C26—C21—Fe2	112.8 (3)	C67—C72—N12	118.0 (4)
N4—C21—Fe2	58.2 (2)	C74—C73—C78	119.3 (4)
C23—C22—C21	120.9 (4)	C74—C73—N12	119.1 (4)
C23—C22—H22	119.5	C78—C73—N12	121.6 (4)
C21—C22—H22	119.5	C75—C74—C73	123.9 (5)
C24—C23—C22	120.6 (4)	C75—C74—H74	118.0
C24—C23—H23	119.7	C73—C74—H74	118.0
C22—C23—H23	119.7	C76—C75—C74	117.0 (5)
C23—C24—C25	118.9 (4)	C76—C75—H75	121.5
C23—C24—H24	120.6	C74—C75—H75	121.5
C25—C24—H24	120.6	C75—C76—C77	121.2 (5)
C26—C25—C24	121.8 (4)	C75—C76—H76	119.4
C26—C25—H25	119.1	C77—C76—H76	119.4
C24—C25—H25	119.1	C76—C77—C78	122.3 (4)
C25—C26—C21	119.9 (4)	C76—C77—H77	118.9
C25—C26—N5	120.3 (4)	C78—C77—H77	118.9
C21—C26—N5	119.7 (4)	N10—C78—C73	125.8 (4)
C28—C27—N5	124.0 (5)	N10—C78—C77	117.9 (4)
C28—C27—C32	118.9 (4)	C73—C78—C77	116.3 (4)
N5—C27—C32	117.1 (4)	N11—C79—H79A	109.5
C29—C28—C27	120.5 (6)	N11—C79—H79B	109.5
C29—C28—H28	119.8	H79A—C79—H79B	109.5
C27—C28—H28	119.8	N11—C79—H79C	109.5
C30—C29—C28	121.2 (5)	H79A—C79—H79C	109.5
C30—C29—H29	119.4	H79B—C79—H79C	109.5
C28—C29—H29	119.4	N12—C80—H80A	109.5
C29—C30—C31	119.1 (5)	N12—C80—H80B	109.5
C29—C30—H30	120.5	H80A—C80—H80B	109.5
C31—C30—H30	120.5	N12—C80—H80C	109.5
C32—C31—C30	121.2 (5)	H80A—C80—H80C	109.5
C32—C31—H31	119.4	H80B—C80—H80C	109.5

C30—C31—H31	119.4	N10—Fe1—N7	139.63 (14)
C31—C32—C27	119.0 (5)	N10—Fe1—N4	115.65 (13)
C31—C32—N6	124.6 (5)	N7—Fe1—N4	89.81 (14)
C27—C32—N6	116.4 (4)	N10—Fe1—N9	107.53 (14)
C34—C33—C38	119.8 (4)	N7—Fe1—N9	79.85 (13)
C34—C33—N6	118.0 (4)	N4—Fe1—N9	122.57 (14)
C38—C33—N6	122.3 (4)	N10—Fe1—Fe2	143.01 (11)
C35—C34—C33	122.4 (5)	N7—Fe1—Fe2	45.05 (10)
C35—C34—H34	118.8	N4—Fe1—Fe2	45.18 (10)
C33—C34—H34	118.8	N9—Fe1—Fe2	108.97 (10)
C34—C35—C36	117.7 (5)	N1—Fe2—N7	123.16 (15)
C34—C35—H35	121.1	N1—Fe2—N4	143.61 (15)
C36—C35—H35	121.1	N7—Fe2—N4	93.04 (14)
C37—C36—C35	120.9 (5)	N1—Fe2—C21	119.49 (15)
C37—C36—H36	119.6	N7—Fe2—C21	110.96 (15)
C35—C36—H36	119.6	N4—Fe2—C21	36.19 (14)
C36—C37—C38	121.8 (5)	N1—Fe2—Fe1	168.73 (11)
C36—C37—H37	119.1	N7—Fe2—Fe1	45.87 (10)
C38—C37—H37	119.1	N4—Fe2—Fe1	47.62 (10)
C33—C38—C37	117.4 (4)	C21—Fe2—Fe1	68.99 (11)
C33—C38—N4	126.0 (4)	C18—N1—C1	122.3 (4)
C37—C38—N4	116.3 (4)	C18—N1—Fe2	128.7 (3)
N5—C39—H39A	109.5	C1—N1—Fe2	108.3 (3)
N5—C39—H39B	109.5	C7—N2—C6	109.9 (4)
H39A—C39—H39B	109.5	C7—N2—C19	115.6 (4)
N5—C39—H39C	109.5	C6—N2—C19	112.0 (4)
H39A—C39—H39C	109.5	C12—N3—C13	116.3 (4)
H39B—C39—H39C	109.5	C12—N3—C20	118.6 (4)
N6—C40—H40A	109.5	C13—N3—C20	112.2 (4)
N6—C40—H40B	109.5	C38—N4—C21	120.8 (3)
H40A—C40—H40B	109.5	C38—N4—Fe2	130.2 (3)
N6—C40—H40C	109.5	C21—N4—Fe2	85.6 (2)
H40A—C40—H40C	109.5	C38—N4—Fe1	112.6 (3)
H40B—C40—H40C	109.5	C21—N4—Fe1	114.8 (3)
C42—C41—C46	118.2 (4)	Fe2—N4—Fe1	87.20 (13)
C42—C41—N7	120.6 (4)	C27—N5—C26	113.2 (4)
C46—C41—N7	121.1 (4)	C27—N5—C39	117.9 (4)
C43—C42—C41	122.0 (5)	C26—N5—C39	113.7 (4)
C43—C42—H42	119.0	C32—N6—C33	112.0 (4)
C41—C42—H42	119.0	C32—N6—C40	116.6 (4)
C42—C43—C44	120.0 (5)	C33—N6—C40	114.5 (4)
C42—C43—H43	120.0	C41—N7—C58	113.5 (3)
C44—C43—H43	120.0	C41—N7—Fe2	120.7 (3)
C43—C44—C45	119.1 (5)	C58—N7—Fe2	98.9 (3)
C43—C44—H44	120.4	C41—N7—Fe1	123.5 (3)
C45—C44—H44	120.4	C58—N7—Fe1	106.5 (3)
C46—C45—C44	121.7 (5)	Fe2—N7—Fe1	89.07 (13)
C46—C45—H45	119.1	C46—N8—C47	115.1 (4)

C44—C45—H45	119.1	C46—N8—C59	116.8 (4)
C45—C46—C41	118.7 (5)	C47—N8—C59	114.4 (4)
C45—C46—N8	123.3 (4)	C53—N9—C52	114.4 (3)
C41—C46—N8	118.0 (4)	C53—N9—C60	111.7 (4)
C48—C47—C52	119.2 (5)	C52—N9—C60	112.1 (4)
C48—C47—N8	116.8 (4)	C53—N9—Fe1	102.3 (3)
C52—C47—N8	124.1 (4)	C52—N9—Fe1	104.0 (3)
C49—C48—C47	122.9 (5)	C60—N9—Fe1	111.6 (3)
C49—C48—H48	118.6	C78—N10—C61	123.3 (3)
C47—C48—H48	118.6	C78—N10—Fe1	122.2 (3)
C50—C49—C48	117.8 (5)	C61—N10—Fe1	114.2 (3)
C50—C49—H49	121.1	C67—N11—C66	112.4 (3)
C48—C49—H49	121.1	C67—N11—C79	117.2 (3)
C49—C50—C51	121.0 (5)	C66—N11—C79	112.6 (3)
C49—C50—H50	119.5	C72—N12—C73	113.7 (3)
C51—C50—H50	119.5	C72—N12—C80	117.2 (4)
C50—C51—C52	121.1 (5)	C73—N12—C80	113.6 (4)

C6—C1—C2—C3	−2.4 (7)	N11—C67—C72—N12	−3.9 (6)
N1—C1—C2—C3	171.3 (4)	C78—C73—C74—C75	−0.7 (7)
C1—C2—C3—C4	0.4 (8)	N12—C73—C74—C75	−179.2 (4)
C2—C3—C4—C5	2.1 (9)	C73—C74—C75—C76	−0.1 (7)
C3—C4—C5—C6	−2.6 (9)	C74—C75—C76—C77	0.6 (7)
C4—C5—C6—C1	0.6 (8)	C75—C76—C77—C78	−0.4 (7)
C4—C5—C6—N2	−180.0 (5)	C74—C73—C78—N10	−177.4 (4)
C2—C1—C6—C5	1.9 (7)	N12—C73—C78—N10	1.2 (6)
N1—C1—C6—C5	−171.3 (5)	C74—C73—C78—C77	0.8 (6)
C2—C1—C6—N2	−177.6 (4)	N12—C73—C78—C77	179.4 (4)
N1—C1—C6—N2	9.2 (8)	C76—C77—C78—N10	178.0 (4)
C12—C7—C8—C9	4.0 (7)	C76—C77—C78—C73	−0.4 (6)
N2—C7—C8—C9	−174.4 (4)	C17—C18—N1—C1	173.8 (4)
C7—C8—C9—C10	2.1 (8)	C13—C18—N1—C1	−10.1 (7)
C8—C9—C10—C11	−5.5 (8)	C17—C18—N1—Fe2	4.8 (6)
C9—C10—C11—C12	2.8 (8)	C13—C18—N1—Fe2	−179.1 (3)
C10—C11—C12—N3	−179.3 (5)	C6—C1—N1—C18	−54.7 (7)
C10—C11—C12—C7	3.2 (7)	C2—C1—N1—C18	132.1 (5)
C8—C7—C12—N3	175.8 (4)	C6—C1—N1—Fe2	116.3 (5)
N2—C7—C12—N3	−5.6 (6)	C2—C1—N1—Fe2	−56.9 (5)
C8—C7—C12—C11	−6.6 (7)	C8—C7—N2—C6	121.6 (5)
N2—C7—C12—C11	171.9 (4)	C12—C7—N2—C6	−56.9 (5)
C18—C13—C14—C15	3.2 (7)	C8—C7—N2—C19	−6.4 (6)
N3—C13—C14—C15	−173.0 (4)	C12—C7—N2—C19	175.1 (4)
C13—C14—C15—C16	0.4 (7)	C5—C6—N2—C7	−63.7 (6)
C14—C15—C16—C17	−1.8 (7)	C1—C6—N2—C7	115.8 (5)
C15—C16—C17—C18	−0.4 (7)	C5—C6—N2—C19	66.3 (6)
C16—C17—C18—N1	−179.6 (4)	C1—C6—N2—C19	−114.2 (5)
C16—C17—C18—C13	3.9 (7)	C11—C12—N3—C13	118.5 (5)
C14—C13—C18—N1	178.6 (4)	C7—C12—N3—C13	−64.1 (6)

N3—C13—C18—N1	−5.3 (7)	C11—C12—N3—C20	−20.1 (7)
C14—C13—C18—C17	−5.2 (7)	C7—C12—N3—C20	157.4 (4)
N3—C13—C18—C17	170.9 (4)	C14—C13—N3—C12	−65.1 (6)
C26—C21—C22—C23	2.2 (6)	C18—C13—N3—C12	118.7 (5)
N4—C21—C22—C23	179.0 (4)	C14—C13—N3—C20	76.0 (5)
Fe2—C21—C22—C23	121.9 (4)	C18—C13—N3—C20	−100.2 (5)
C21—C22—C23—C24	0.5 (6)	C33—C38—N4—C21	−30.0 (7)
C22—C23—C24—C25	−2.7 (6)	C37—C38—N4—C21	156.3 (4)
C23—C24—C25—C26	2.2 (7)	C33—C38—N4—Fe2	−142.7 (4)
C24—C25—C26—C21	0.5 (7)	C37—C38—N4—Fe2	43.7 (6)
C24—C25—C26—N5	−179.3 (4)	C33—C38—N4—Fe1	110.9 (5)
C22—C21—C26—C25	−2.7 (6)	C37—C38—N4—Fe1	−62.7 (5)
N4—C21—C26—C25	−179.3 (4)	C22—C21—N4—C38	145.9 (4)
Fe2—C21—C26—C25	−113.1 (4)	C26—C21—N4—C38	−37.5 (6)
C22—C21—C26—N5	177.1 (4)	Fe2—C21—N4—C38	−135.0 (4)
N4—C21—C26—N5	0.5 (6)	C22—C21—N4—Fe2	−79.1 (4)
Fe2—C21—C26—N5	66.7 (5)	C26—C21—N4—Fe2	97.5 (4)
N5—C27—C28—C29	−175.5 (5)	C22—C21—N4—Fe1	5.8 (5)
C32—C27—C28—C29	3.5 (8)	C26—C21—N4—Fe1	−177.7 (3)
C27—C28—C29—C30	1.1 (10)	Fe2—C21—N4—Fe1	84.8 (2)
C28—C29—C30—C31	−3.6 (10)	C28—C27—N5—C26	124.2 (5)
C29—C30—C31—C32	1.3 (9)	C32—C27—N5—C26	−54.8 (6)
C30—C31—C32—C27	3.3 (8)	C28—C27—N5—C39	−12.1 (7)
C30—C31—C32—N6	−174.6 (5)	C32—C27—N5—C39	168.9 (4)
C28—C27—C32—C31	−5.7 (7)	C25—C26—N5—C27	−59.5 (6)
N5—C27—C32—C31	173.4 (4)	C21—C26—N5—C27	120.7 (4)
C28—C27—C32—N6	172.5 (4)	C25—C26—N5—C39	78.7 (5)
N5—C27—C32—N6	−8.5 (6)	C21—C26—N5—C39	−101.1 (5)
C38—C33—C34—C35	−0.1 (8)	C31—C32—N6—C33	116.0 (5)
N6—C33—C34—C35	179.3 (5)	C27—C32—N6—C33	−62.0 (5)
C33—C34—C35—C36	−1.2 (9)	C31—C32—N6—C40	−18.5 (7)
C34—C35—C36—C37	−0.2 (10)	C27—C32—N6—C40	163.5 (4)
C35—C36—C37—C38	2.7 (9)	C34—C33—N6—C32	−66.3 (6)
C34—C33—C38—C37	2.5 (7)	C38—C33—N6—C32	113.1 (5)
N6—C33—C38—C37	−176.8 (4)	C34—C33—N6—C40	69.3 (6)
C34—C33—C38—N4	−171.1 (5)	C38—C33—N6—C40	−111.4 (5)
N6—C33—C38—N4	9.6 (7)	C42—C41—N7—C58	144.3 (4)
C36—C37—C38—C33	−3.9 (8)	C46—C41—N7—C58	−36.4 (6)
C36—C37—C38—N4	170.3 (5)	C42—C41—N7—Fe2	27.3 (5)
C46—C41—C42—C43	2.5 (7)	C46—C41—N7—Fe2	−153.4 (3)
N7—C41—C42—C43	−178.2 (4)	C42—C41—N7—Fe1	−84.5 (5)
C41—C42—C43—C44	1.0 (8)	C46—C41—N7—Fe1	94.8 (4)
C42—C43—C44—C45	−2.2 (8)	C57—C58—N7—C41	−67.4 (6)
C43—C44—C45—C46	−0.1 (9)	C53—C58—N7—C41	111.8 (5)
C44—C45—C46—C41	3.6 (8)	C57—C58—N7—Fe2	61.8 (4)
C44—C45—C46—N8	−175.8 (5)	C53—C58—N7—Fe2	−119.0 (4)
C42—C41—C46—C45	−4.7 (7)	C57—C58—N7—Fe1	153.5 (4)
N7—C41—C46—C45	176.0 (4)	C53—C58—N7—Fe1	−27.3 (5)

C42—C41—C46—N8	174.7 (4)	C45—C46—N8—C47	95.8 (5)
N7—C41—C46—N8	−4.6 (6)	C41—C46—N8—C47	−83.6 (5)
C52—C47—C48—C49	0.5 (8)	C45—C46—N8—C59	−42.5 (7)
N8—C47—C48—C49	178.8 (5)	C41—C46—N8—C59	138.1 (4)
C47—C48—C49—C50	−1.3 (8)	C48—C47—N8—C46	−77.2 (5)
C48—C49—C50—C51	0.1 (8)	C52—C47—N8—C46	101.1 (5)
C49—C50—C51—C52	1.8 (8)	C48—C47—N8—C59	62.1 (6)
C48—C47—C52—C51	1.4 (7)	C52—C47—N8—C59	−119.6 (5)
N8—C47—C52—C51	−176.8 (4)	C54—C53—N9—C52	93.7 (5)
C48—C47—C52—N9	179.1 (4)	C58—C53—N9—C52	−85.6 (5)
N8—C47—C52—N9	0.8 (7)	C54—C53—N9—C60	−34.9 (6)
C50—C51—C52—C47	−2.6 (7)	C58—C53—N9—C60	145.8 (4)
C50—C51—C52—N9	179.6 (4)	C54—C53—N9—Fe1	−154.5 (4)
C58—C53—C54—C55	0.9 (7)	C58—C53—N9—Fe1	26.2 (4)
N9—C53—C54—C55	−178.4 (4)	C47—C52—N9—C53	21.8 (6)
C53—C54—C55—C56	−0.1 (8)	C51—C52—N9—C53	−160.5 (4)
C54—C55—C56—C57	−0.6 (8)	C47—C52—N9—C60	150.3 (4)
C55—C56—C57—C58	0.3 (8)	C51—C52—N9—C60	−32.1 (5)
C56—C57—C58—C53	0.5 (7)	C47—C52—N9—Fe1	−89.0 (4)
C56—C57—C58—N7	179.7 (4)	C51—C52—N9—Fe1	88.7 (4)
C54—C53—C58—C57	−1.1 (7)	C73—C78—N10—C61	−23.2 (6)
N9—C53—C58—C57	178.2 (4)	C77—C78—N10—C61	158.6 (4)
C54—C53—C58—N7	179.6 (4)	C73—C78—N10—Fe1	163.5 (3)
N9—C53—C58—N7	−1.0 (6)	C77—C78—N10—Fe1	−14.7 (5)
N10—C61—C62—C63	169.3 (4)	C62—C61—N10—C78	142.7 (4)
C66—C61—C62—C63	−3.2 (7)	C66—C61—N10—C78	−45.4 (6)
C61—C62—C63—C64	2.0 (7)	C62—C61—N10—Fe1	−43.5 (5)
C62—C63—C64—C65	0.8 (7)	C66—C61—N10—Fe1	128.4 (4)
C63—C64—C65—C66	−2.2 (7)	C68—C67—N11—C66	119.5 (5)
C64—C65—C66—C61	0.9 (7)	C72—C67—N11—C66	−59.9 (5)
C64—C65—C66—N11	−179.4 (4)	C68—C67—N11—C79	−13.2 (6)
N10—C61—C66—C65	−170.3 (4)	C72—C67—N11—C79	167.4 (4)
C62—C61—C66—C65	1.7 (6)	C65—C66—N11—C67	−66.3 (5)
N10—C61—C66—N11	10.0 (7)	C61—C66—N11—C67	113.4 (5)
C62—C61—C66—N11	−178.0 (4)	C65—C66—N11—C79	68.6 (5)
C72—C67—C68—C69	2.5 (7)	C61—C66—N11—C79	−111.7 (4)
N11—C67—C68—C69	−176.9 (4)	C71—C72—N12—C73	116.0 (5)
C67—C68—C69—C70	−0.2 (8)	C67—C72—N12—C73	−63.0 (5)
C68—C69—C70—C71	−2.1 (8)	C71—C72—N12—C80	−19.9 (6)
C69—C70—C71—C72	2.2 (7)	C67—C72—N12—C80	161.1 (4)
C70—C71—C72—C67	0.0 (7)	C74—C73—N12—C72	−65.5 (5)
C70—C71—C72—N12	−178.9 (4)	C78—C73—N12—C72	116.0 (4)
C68—C67—C72—C71	−2.4 (7)	C74—C73—N12—C80	72.0 (5)
N11—C67—C72—C71	177.1 (4)	C78—C73—N12—C80	−106.6 (5)
C68—C67—C72—N12	176.6 (4)		

Bis(μ-1,4-dimethyltribenzo[*b*,*e*,*h*][1,4,7]triazacyclonona-2,5,8-trien-7-ido-κ*N*^{7})bis(tetrahydrofuran-κ*O*)iron(II) (10)

Crystal data

$[Fe(C_{20}H_{18}N_3)_2(C_4H_8O)_2]$
M_r = 800.80
Orthorhombic, *Pbca*
a = 15.9624 (12) Å
b = 15.8047 (11) Å
c = 32.747 (2) Å
V = 8261.5 (10) Å^3
Z = 8
F(000) = 3392
D_x = 1.288 Mg m^{-3}
Cu *Kα* radiation, λ = 1.54178 Å
Cell parameters from 9933 reflections
θ = 3.9–66.7°
μ = 3.29 mm^{-1}
T = 100 K
Block, green
0.11 × 0.10 × 0.06 mm

Data collection

Bruker X8 Prospector CCD diffractometer
Radiation source: I-mu-S microsource X-ray tube
Laterally graded multilayer (Goebel) mirror monochromator
ω and phi scans
Absorption correction: multi-scan (APEX2; Bruker, 2014)
T_{min} = 0.569, T_{max} = 0.753
23792 measured reflections
7194 independent reflections
5411 reflections with $I > 2\sigma(I)$
R_{int} = 0.055
θ_{max} = 66.7°, θ_{min} = 3.9°
h = −10→18
k = −18→18
l = −31→38

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.111
S = 1.05
7194 reflections
555 parameters
132 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
$w = 1/[\sigma^2(F_o^2) + (0.0545P)^2 + 1.2424P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 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. One THF ligand is disordered with two alternative orientations. The two moieties were restrained to have similar geometries, and the Uij components of their atoms′ ADPs were restrained to be similar if closer than 1.7 Angstrom. Subject to these conditions the occupancy ratio refined to 0.790 (8) to 0.210 (8). Reflection 0 0 2 was affected by the beam stop and was omitted from the refinement.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.01642 (16)	0.58940 (13)	0.32908 (7)	0.0168 (5)	
C2	0.09943 (16)	0.58486 (14)	0.31399 (7)	0.0200 (5)	
H2	0.1417	0.5615	0.3310	0.024*	

C3	0.12151 (18)	0.61286 (14)	0.27560 (7)	0.0226 (5)
H3	0.1776	0.6063	0.2664	0.027*
C4	0.06276 (18)	0.65050 (15)	0.25038 (7)	0.0234 (6)
H4	0.0775	0.6699	0.2239	0.028*
C5	−0.01885 (17)	0.65892 (14)	0.26520 (7)	0.0208 (5)
H5	−0.0596	0.6861	0.2486	0.025*
C6	−0.04262 (16)	0.62894 (13)	0.30332 (7)	0.0175 (5)
C7	−0.15464 (17)	0.72340 (15)	0.31940 (7)	0.0210 (5)
C8	−0.21592 (18)	0.76162 (16)	0.29572 (8)	0.0272 (6)
H8	−0.2452	0.7293	0.2759	0.033*
C9	−0.2349 (2)	0.84726 (17)	0.30086 (9)	0.0355 (7)
H9	−0.2776	0.8730	0.2849	0.043*
C10	−0.1909 (2)	0.89430 (17)	0.32932 (9)	0.0378 (7)
H10	−0.2019	0.9531	0.3321	0.045*
C11	−0.13083 (19)	0.85648 (16)	0.35387 (8)	0.0305 (6)
H11	−0.1012	0.8895	0.3733	0.037*
C12	−0.11369 (17)	0.77097 (15)	0.35020 (7)	0.0216 (5)
C13	−0.09703 (16)	0.65585 (15)	0.39656 (7)	0.0191 (5)
C14	−0.16313 (17)	0.67221 (16)	0.42292 (7)	0.0242 (6)
H14	−0.1838	0.7284	0.4252	0.029*
C15	−0.19976 (17)	0.60877 (17)	0.44604 (8)	0.0286 (6)
H15	−0.2445	0.6212	0.4642	0.034*
C16	−0.17007 (18)	0.52737 (17)	0.44225 (8)	0.0288 (6)
H16	−0.1940	0.4833	0.4582	0.035*
C17	−0.10565 (17)	0.50939 (15)	0.41537 (8)	0.0247 (6)
H17	−0.0867	0.4526	0.4130	0.030*
C18	−0.06699 (15)	0.57262 (15)	0.39130 (7)	0.0181 (5)
C19	−0.18646 (17)	0.57902 (15)	0.29672 (8)	0.0265 (6)
H19A	−0.1868	0.5873	0.2671	0.040*
H19B	−0.2429	0.5884	0.3075	0.040*
H19C	−0.1686	0.5211	0.3030	0.040*
C20	−0.00600 (17)	0.77649 (15)	0.40344 (7)	0.0227 (6)
H20A	0.0224	0.8212	0.3880	0.034*
H20B	0.0359	0.7399	0.4164	0.034*
H20C	−0.0416	0.8020	0.4245	0.034*
C21	0.22608 (17)	0.53580 (14)	0.42264 (7)	0.0206 (5)
C22	0.16839 (18)	0.59961 (15)	0.43274 (8)	0.0264 (6)
H22	0.1155	0.6001	0.4194	0.032*
C23	0.18557 (19)	0.66182 (16)	0.46131 (8)	0.0295 (6)
H23	0.1447	0.7039	0.4671	0.035*
C24	0.26167 (19)	0.66321 (16)	0.48145 (8)	0.0307 (6)
H24	0.2739	0.7057	0.5011	0.037*
C25	0.31964 (18)	0.60072 (15)	0.47213 (7)	0.0244 (6)
H25	0.3722	0.6011	0.4857	0.029*
C26	0.30359 (16)	0.53794 (14)	0.44382 (7)	0.0191 (5)
C27	0.44191 (16)	0.50477 (14)	0.42074 (7)	0.0205 (5)
C28	0.51903 (18)	0.49602 (16)	0.43994 (8)	0.0269 (6)
H28	0.5225	0.4653	0.4648	0.032*

C29	0.59142 (19)	0.53139 (17)	0.42340 (9)	0.0333 (7)	
H29	0.6437	0.5242	0.4368	0.040*	
C30	0.58688 (19)	0.57695 (18)	0.38751 (9)	0.0348 (7)	
H30	0.6354	0.6041	0.3770	0.042*	
C31	0.51092 (19)	0.58309 (15)	0.36667 (9)	0.0310 (6)	
H31	0.5084	0.6136	0.3417	0.037*	
C32	0.43867 (17)	0.54505 (14)	0.38190 (8)	0.0224 (5)	
C33	0.33363 (17)	0.45479 (15)	0.35471 (7)	0.0212 (5)	
C34	0.38313 (18)	0.40175 (16)	0.33094 (7)	0.0261 (6)	
H34	0.4316	0.4240	0.3180	0.031*	
C35	0.36331 (19)	0.31719 (17)	0.32570 (8)	0.0312 (6)	
H35	0.3968	0.2821	0.3087	0.037*	
C36	0.29458 (19)	0.28494 (16)	0.34544 (8)	0.0311 (6)	
H36	0.2814	0.2265	0.3430	0.037*	
C37	0.24396 (18)	0.33734 (15)	0.36908 (8)	0.0252 (6)	
H37	0.1965	0.3139	0.3824	0.030*	
C38	0.26102 (17)	0.42343 (15)	0.37376 (7)	0.0207 (5)	
C39	0.36655 (18)	0.40468 (15)	0.46536 (7)	0.0243 (6)	
H39A	0.3106	0.3792	0.4665	0.036*	
H39B	0.3818	0.4259	0.4925	0.036*	
H39C	0.4074	0.3620	0.4567	0.036*	
C40	0.3533 (2)	0.59413 (17)	0.32455 (8)	0.0328 (7)	
H40A	0.3929	0.5744	0.3038	0.049*	
H40B	0.3655	0.6532	0.3314	0.049*	
H40C	0.2961	0.5899	0.3139	0.049*	
C41	0.0552 (6)	0.3854 (4)	0.46578 (13)	0.0380 (12)	0.790 (8)
H41A	0.0162	0.4308	0.4744	0.046*	0.790 (8)
H41B	0.1132	0.4036	0.4719	0.046*	0.790 (8)
C42	0.0353 (3)	0.3049 (4)	0.48744 (12)	0.0405 (12)	0.790 (8)
H42A	0.0039	0.3162	0.5129	0.049*	0.790 (8)
H42B	0.0872	0.2736	0.4942	0.049*	0.790 (8)
C43	−0.0177 (3)	0.2556 (3)	0.45750 (14)	0.0388 (11)	0.790 (8)
H43A	−0.0122	0.1940	0.4621	0.047*	0.790 (8)
H43B	−0.0775	0.2717	0.4597	0.047*	0.790 (8)
C44	0.0174 (4)	0.2806 (3)	0.41696 (15)	0.0356 (12)	0.790 (8)
H44A	0.0648	0.2434	0.4093	0.043*	0.790 (8)
H44B	−0.0260	0.2771	0.3954	0.043*	0.790 (8)
C41B	0.063 (2)	0.4049 (16)	0.4631 (5)	0.038 (3)	0.210 (8)
H41C	0.0464	0.4653	0.4641	0.046*	0.210 (8)
H41D	0.1227	0.3996	0.4703	0.046*	0.210 (8)
C42B	0.0085 (12)	0.3520 (12)	0.4910 (4)	0.040 (2)	0.210 (8)
H42C	0.0338	0.3477	0.5186	0.048*	0.210 (8)
H42D	−0.0482	0.3769	0.4936	0.048*	0.210 (8)
C43B	0.0048 (15)	0.2675 (11)	0.4709 (6)	0.040 (2)	0.210 (8)
H43C	−0.0529	0.2447	0.4721	0.048*	0.210 (8)
H43D	0.0428	0.2273	0.4848	0.048*	0.210 (8)
C44B	0.0314 (18)	0.2792 (9)	0.4274 (5)	0.038 (3)	0.210 (8)
H44C	0.0832	0.2468	0.4217	0.045*	0.210 (8)

H44D	−0.0131	0.2596	0.4085	0.045*	0.210 (8)
C45	0.1079 (2)	0.36793 (17)	0.29266 (7)	0.0310 (7)	
H45A	0.1274	0.3086	0.2904	0.037*	
H45B	0.1574	0.4056	0.2934	0.037*	
C46	0.0515 (2)	0.39048 (17)	0.25683 (8)	0.0322 (7)	
H46A	0.0642	0.4480	0.2466	0.039*	
H46B	0.0588	0.3496	0.2342	0.039*	
C47	−0.0369 (2)	0.3863 (2)	0.27371 (8)	0.0408 (8)	
H47A	−0.0640	0.4425	0.2725	0.049*	
H47B	−0.0711	0.3454	0.2580	0.049*	
C48	−0.0273 (2)	0.35759 (19)	0.31744 (8)	0.0374 (7)	
H48A	−0.0682	0.3869	0.3352	0.045*	
H48B	−0.0364	0.2958	0.3197	0.045*	
N1	0.00017 (13)	0.54988 (11)	0.36630 (6)	0.0176 (4)	
N2	−0.05762 (14)	0.72633 (12)	0.37581 (6)	0.0194 (4)	
N3	−0.12926 (13)	0.63808 (11)	0.31516 (6)	0.0182 (4)	
N4	0.20224 (13)	0.47367 (12)	0.39503 (6)	0.0184 (4)	
N5	0.36636 (14)	0.47409 (12)	0.43652 (6)	0.0192 (4)	
N6	0.36110 (14)	0.54189 (12)	0.36104 (6)	0.0231 (5)	
Fe1	0.08313 (2)	0.45812 (2)	0.37925 (2)	0.01653 (11)	
O1	0.04577 (13)	0.36799 (10)	0.42238 (5)	0.0287 (4)	
O2	0.05724 (12)	0.37914 (11)	0.32899 (5)	0.0272 (4)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0183 (14)	0.0122 (10)	0.0199 (12)	−0.0029 (9)	−0.0020 (10)	−0.0015 (9)
C2	0.0200 (15)	0.0189 (11)	0.0212 (12)	0.0012 (10)	−0.0024 (10)	0.0023 (9)
C3	0.0215 (15)	0.0225 (11)	0.0238 (13)	−0.0024 (11)	0.0046 (10)	−0.0007 (10)
C4	0.0290 (16)	0.0240 (12)	0.0172 (11)	−0.0043 (11)	0.0014 (10)	0.0021 (10)
C5	0.0253 (15)	0.0167 (11)	0.0202 (12)	−0.0013 (10)	−0.0050 (10)	0.0020 (9)
C6	0.0187 (14)	0.0131 (10)	0.0209 (12)	−0.0021 (9)	−0.0004 (10)	−0.0033 (9)
C7	0.0200 (15)	0.0211 (11)	0.0220 (12)	0.0013 (10)	0.0006 (10)	0.0000 (10)
C8	0.0245 (16)	0.0287 (13)	0.0283 (14)	0.0004 (11)	−0.0067 (11)	−0.0022 (11)
C9	0.0319 (19)	0.0312 (14)	0.0435 (17)	0.0130 (13)	−0.0122 (13)	0.0004 (12)
C10	0.045 (2)	0.0208 (12)	0.0480 (18)	0.0117 (13)	−0.0108 (15)	−0.0061 (12)
C11	0.0326 (18)	0.0245 (13)	0.0344 (15)	0.0016 (12)	−0.0078 (13)	−0.0062 (11)
C12	0.0194 (15)	0.0224 (12)	0.0230 (12)	0.0010 (10)	0.0004 (10)	−0.0012 (10)
C13	0.0176 (14)	0.0238 (12)	0.0159 (11)	−0.0032 (10)	−0.0029 (9)	0.0004 (9)
C14	0.0183 (15)	0.0304 (13)	0.0238 (13)	−0.0014 (11)	−0.0021 (10)	−0.0058 (11)
C15	0.0180 (15)	0.0432 (15)	0.0246 (13)	−0.0055 (12)	0.0062 (11)	−0.0049 (12)
C16	0.0231 (16)	0.0346 (14)	0.0288 (14)	−0.0081 (12)	0.0028 (11)	0.0063 (11)
C17	0.0220 (15)	0.0222 (12)	0.0299 (13)	−0.0017 (10)	−0.0001 (11)	0.0064 (10)
C18	0.0131 (14)	0.0226 (11)	0.0185 (12)	−0.0010 (10)	−0.0027 (9)	0.0001 (9)
C19	0.0186 (15)	0.0253 (12)	0.0356 (15)	−0.0028 (11)	−0.0017 (11)	−0.0036 (11)
C20	0.0232 (15)	0.0211 (12)	0.0237 (13)	−0.0024 (10)	−0.0027 (10)	−0.0006 (10)
C21	0.0224 (15)	0.0201 (11)	0.0192 (12)	0.0013 (10)	−0.0010 (10)	−0.0013 (10)
C22	0.0200 (15)	0.0280 (13)	0.0311 (14)	0.0033 (11)	−0.0092 (11)	−0.0051 (11)

C23	0.0271 (17)	0.0286 (13)	0.0330 (14)	0.0093 (12)	−0.0047 (12)	−0.0085 (11)
C24	0.0337 (18)	0.0279 (13)	0.0307 (14)	0.0028 (12)	−0.0056 (12)	−0.0134 (11)
C25	0.0204 (15)	0.0297 (13)	0.0230 (13)	0.0007 (11)	−0.0041 (10)	−0.0028 (10)
C26	0.0192 (14)	0.0196 (11)	0.0184 (11)	0.0009 (10)	0.0022 (9)	−0.0006 (9)
C27	0.0178 (14)	0.0192 (11)	0.0245 (13)	0.0010 (10)	0.0041 (10)	−0.0058 (10)
C28	0.0255 (16)	0.0300 (13)	0.0252 (13)	0.0030 (11)	−0.0007 (11)	−0.0105 (11)
C29	0.0183 (16)	0.0388 (15)	0.0428 (16)	−0.0010 (12)	0.0025 (12)	−0.0202 (13)
C30	0.0248 (17)	0.0324 (14)	0.0473 (17)	−0.0103 (13)	0.0118 (13)	−0.0127 (13)
C31	0.0358 (19)	0.0219 (12)	0.0351 (15)	−0.0038 (12)	0.0124 (13)	−0.0030 (11)
C32	0.0233 (15)	0.0164 (11)	0.0274 (13)	0.0021 (10)	0.0073 (11)	−0.0048 (10)
C33	0.0271 (16)	0.0224 (12)	0.0142 (11)	0.0056 (11)	−0.0040 (10)	0.0014 (9)
C34	0.0258 (16)	0.0316 (13)	0.0208 (12)	0.0042 (12)	−0.0025 (11)	−0.0022 (10)
C35	0.0303 (18)	0.0332 (14)	0.0301 (14)	0.0117 (13)	−0.0054 (12)	−0.0118 (12)
C36	0.0275 (17)	0.0230 (13)	0.0430 (16)	0.0034 (11)	−0.0079 (13)	−0.0121 (12)
C37	0.0172 (14)	0.0246 (13)	0.0339 (14)	0.0017 (11)	−0.0047 (11)	−0.0044 (11)
C38	0.0198 (14)	0.0229 (12)	0.0195 (12)	0.0061 (10)	−0.0063 (10)	−0.0032 (10)
C39	0.0245 (16)	0.0252 (12)	0.0232 (13)	0.0057 (11)	0.0027 (11)	0.0020 (10)
C40	0.0338 (18)	0.0329 (14)	0.0316 (15)	0.0056 (12)	0.0033 (12)	0.0116 (12)
C41	0.056 (3)	0.038 (3)	0.0193 (19)	−0.005 (2)	−0.0013 (19)	0.0042 (18)
C42	0.045 (3)	0.047 (3)	0.0293 (19)	−0.012 (2)	−0.0061 (17)	0.0139 (19)
C43	0.042 (3)	0.044 (2)	0.030 (2)	−0.0157 (19)	−0.0056 (18)	0.0096 (18)
C44	0.053 (3)	0.0231 (17)	0.031 (2)	−0.0130 (17)	−0.002 (2)	0.0099 (16)
C41B	0.048 (5)	0.045 (5)	0.021 (5)	−0.006 (5)	0.002 (4)	0.016 (4)
C42B	0.047 (5)	0.045 (5)	0.028 (4)	−0.009 (4)	−0.003 (4)	0.010 (4)
C43B	0.049 (5)	0.040 (4)	0.031 (5)	−0.009 (4)	−0.002 (4)	0.013 (4)
C44B	0.050 (5)	0.036 (5)	0.028 (5)	0.000 (4)	−0.005 (5)	0.014 (4)
C45	0.041 (2)	0.0278 (13)	0.0241 (13)	0.0064 (12)	−0.0021 (12)	−0.0067 (11)
C46	0.0403 (19)	0.0287 (13)	0.0277 (14)	0.0020 (12)	−0.0069 (12)	−0.0015 (11)
C47	0.047 (2)	0.0438 (16)	0.0322 (15)	0.0122 (15)	−0.0095 (14)	−0.0108 (13)
C48	0.0323 (19)	0.0430 (16)	0.0369 (16)	−0.0113 (14)	−0.0083 (13)	−0.0070 (13)
N1	0.0148 (11)	0.0189 (9)	0.0189 (9)	0.0010 (8)	−0.0010 (8)	0.0041 (8)
N2	0.0216 (12)	0.0185 (9)	0.0180 (10)	−0.0009 (8)	−0.0027 (8)	−0.0007 (8)
N3	0.0170 (12)	0.0162 (9)	0.0215 (10)	−0.0005 (8)	−0.0035 (8)	−0.0012 (8)
N4	0.0136 (12)	0.0175 (9)	0.0242 (10)	0.0030 (8)	−0.0037 (8)	−0.0020 (8)
N5	0.0173 (12)	0.0210 (9)	0.0193 (10)	0.0014 (8)	−0.0009 (8)	0.0003 (8)
N6	0.0270 (13)	0.0207 (10)	0.0217 (10)	0.0014 (9)	0.0017 (9)	0.0020 (8)
Fe1	0.0167 (2)	0.01553 (17)	0.01733 (19)	0.00079 (15)	−0.00282 (15)	0.00016 (15)
O1	0.0407 (13)	0.0233 (8)	0.0220 (9)	−0.0009 (8)	0.0016 (8)	0.0036 (7)
O2	0.0302 (12)	0.0271 (9)	0.0242 (9)	−0.0020 (8)	−0.0053 (8)	−0.0073 (7)

Geometric parameters (Å, º)

C1—N1	1.394 (3)	C31—H31	0.9500
C1—C6	1.411 (3)	C32—N6	1.415 (4)
C1—C2	1.416 (4)	C33—C34	1.390 (4)
C2—C3	1.378 (3)	C33—C38	1.406 (4)
C2—H2	0.9500	C33—N6	1.460 (3)
C3—C4	1.384 (4)	C34—C35	1.384 (4)

C3—H3	0.9500	C34—H34	0.9500
C4—C5	1.397 (4)	C35—C36	1.372 (4)
C4—H4	0.9500	C35—H35	0.9500
C5—C6	1.388 (3)	C36—C37	1.392 (4)
C5—H5	0.9500	C36—H36	0.9500
C6—N3	1.444 (3)	C37—C38	1.396 (3)
C7—C8	1.387 (4)	C37—H37	0.9500
C7—N3	1.415 (3)	C38—N4	1.413 (3)
C7—C12	1.418 (3)	C39—N5	1.448 (3)
C8—C9	1.397 (4)	C39—H39A	0.9800
C8—H8	0.9500	C39—H39B	0.9800
C9—C10	1.384 (4)	C39—H39C	0.9800
C9—H9	0.9500	C40—N6	1.458 (3)
C10—C11	1.386 (4)	C40—H40A	0.9800
C10—H10	0.9500	C40—H40B	0.9800
C11—C12	1.384 (4)	C40—H40C	0.9800
C11—H11	0.9500	C41—O1	1.455 (5)
C12—N2	1.415 (3)	C41—C42	1.490 (6)
C13—C14	1.388 (4)	C41—H41A	0.9900
C13—C18	1.411 (3)	C41—H41B	0.9900
C13—N2	1.448 (3)	C42—C43	1.512 (6)
C14—C15	1.386 (4)	C42—H42A	0.9900
C14—H14	0.9500	C42—H42B	0.9900
C15—C16	1.377 (4)	C43—C44	1.495 (5)
C15—H15	0.9500	C43—H43A	0.9900
C16—C17	1.383 (4)	C43—H43B	0.9900
C16—H16	0.9500	C44—O1	1.464 (4)
C17—C18	1.414 (3)	C44—H44A	0.9900
C17—H17	0.9500	C44—H44B	0.9900
C18—N1	1.396 (3)	C41B—O1	1.479 (14)
C19—N3	1.439 (3)	C41B—C42B	1.512 (17)
C19—H19A	0.9800	C41B—H41C	0.9900
C19—H19B	0.9800	C41B—H41D	0.9900
C19—H19C	0.9800	C42B—C43B	1.492 (16)
C20—N2	1.458 (3)	C42B—H42C	0.9900
C20—H20A	0.9800	C42B—H42D	0.9900
C20—H20B	0.9800	C43B—C44B	1.497 (15)
C20—H20C	0.9800	C43B—H43C	0.9900
C21—N4	1.388 (3)	C43B—H43D	0.9900
C21—C22	1.405 (4)	C44B—O1	1.431 (14)
C21—C26	1.419 (4)	C44B—H44C	0.9900
C22—C23	1.385 (3)	C44B—H44D	0.9900
C22—H22	0.9500	C45—O2	1.449 (3)
C23—C24	1.382 (4)	C45—C46	1.522 (4)
C23—H23	0.9500	C45—H45A	0.9900
C24—C25	1.387 (4)	C45—H45B	0.9900
C24—H24	0.9500	C46—C47	1.516 (5)
C25—C26	1.382 (3)	C46—H46A	0.9900

C25—H25	0.9500	C46—H46B	0.9900
C26—N5	1.442 (3)	C47—C48	1.510 (4)
C27—C28	1.389 (4)	C47—H47A	0.9900
C27—N5	1.399 (3)	C47—H47B	0.9900
C27—C32	1.423 (4)	C48—O2	1.442 (3)
C28—C29	1.393 (4)	C48—H48A	0.9900
C28—H28	0.9500	C48—H48B	0.9900
C29—C30	1.380 (4)	N1—Fe1	2.0092 (19)
C29—H29	0.9500	N4—Fe1	1.985 (2)
C30—C31	1.395 (4)	Fe1—O1	2.0926 (17)
C30—H30	0.9500	Fe1—O2	2.1068 (16)
C31—C32	1.393 (4)		

N1—C1—C6	126.7 (2)	C33—C38—N4	124.6 (2)
N1—C1—C2	117.2 (2)	N5—C39—H39A	109.5
C6—C1—C2	116.0 (2)	N5—C39—H39B	109.5
C3—C2—C1	122.8 (2)	H39A—C39—H39B	109.5
C3—C2—H2	118.6	N5—C39—H39C	109.5
C1—C2—H2	118.6	H39A—C39—H39C	109.5
C2—C3—C4	120.6 (3)	H39B—C39—H39C	109.5
C2—C3—H3	119.7	N6—C40—H40A	109.5
C4—C3—H3	119.7	N6—C40—H40B	109.5
C3—C4—C5	117.7 (2)	H40A—C40—H40B	109.5
C3—C4—H4	121.1	N6—C40—H40C	109.5
C5—C4—H4	121.1	H40A—C40—H40C	109.5
C6—C5—C4	122.3 (2)	H40B—C40—H40C	109.5
C6—C5—H5	118.8	O1—C41—C42	106.3 (3)
C4—C5—H5	118.8	O1—C41—H41A	110.5
C5—C6—C1	120.4 (2)	C42—C41—H41A	110.5
C5—C6—N3	118.0 (2)	O1—C41—H41B	110.5
C1—C6—N3	121.6 (2)	C42—C41—H41B	110.5
C8—C7—N3	124.2 (2)	H41A—C41—H41B	108.7
C8—C7—C12	119.5 (2)	C41—C42—C43	104.5 (4)
N3—C7—C12	116.3 (2)	C41—C42—H42A	110.9
C7—C8—C9	120.5 (3)	C43—C42—H42A	110.9
C7—C8—H8	119.7	C41—C42—H42B	110.9
C9—C8—H8	119.7	C43—C42—H42B	110.9
C10—C9—C8	119.4 (3)	H42A—C42—H42B	108.9
C10—C9—H9	120.3	C44—C43—C42	103.3 (3)
C8—C9—H9	120.3	C44—C43—H43A	111.1
C9—C10—C11	120.7 (2)	C42—C43—H43A	111.1
C9—C10—H10	119.7	C44—C43—H43B	111.1
C11—C10—H10	119.7	C42—C43—H43B	111.1
C12—C11—C10	120.5 (3)	H43A—C43—H43B	109.1
C12—C11—H11	119.8	O1—C44—C43	104.9 (3)
C10—C11—H11	119.8	O1—C44—H44A	110.8
C11—C12—N2	124.1 (2)	C43—C44—H44A	110.8
C11—C12—C7	119.2 (2)	O1—C44—H44B	110.8

N2—C12—C7	116.7 (2)	C43—C44—H44B	110.8
C14—C13—C18	120.5 (2)	H44A—C44—H44B	108.8
C14—C13—N2	118.6 (2)	O1—C41B—C42B	102.9 (12)
C18—C13—N2	120.8 (2)	O1—C41B—H41C	111.2
C15—C14—C13	121.7 (2)	C42B—C41B—H41C	111.2
C15—C14—H14	119.1	O1—C41B—H41D	111.2
C13—C14—H14	119.1	C42B—C41B—H41D	111.2
C16—C15—C14	118.8 (2)	H41C—C41B—H41D	109.1
C16—C15—H15	120.6	C43B—C42B—C41B	104.4 (13)
C14—C15—H15	120.6	C43B—C42B—H42C	110.9
C15—C16—C17	120.4 (2)	C41B—C42B—H42C	110.9
C15—C16—H16	119.8	C43B—C42B—H42D	110.9
C17—C16—H16	119.8	C41B—C42B—H42D	110.9
C16—C17—C18	122.3 (2)	H42C—C42B—H42D	108.9
C16—C17—H17	118.9	C42B—C43B—C44B	107.4 (12)
C18—C17—H17	118.9	C42B—C43B—H43C	110.2
N1—C18—C13	124.9 (2)	C44B—C43B—H43C	110.2
N1—C18—C17	118.7 (2)	C42B—C43B—H43D	110.2
C13—C18—C17	116.3 (2)	C44B—C43B—H43D	110.2
N3—C19—H19A	109.5	H43C—C43B—H43D	108.5
N3—C19—H19B	109.5	O1—C44B—C43B	106.0 (12)
H19A—C19—H19B	109.5	O1—C44B—H44C	110.5
N3—C19—H19C	109.5	C43B—C44B—H44C	110.5
H19A—C19—H19C	109.5	O1—C44B—H44D	110.5
H19B—C19—H19C	109.5	C43B—C44B—H44D	110.5
N2—C20—H20A	109.5	H44C—C44B—H44D	108.7
N2—C20—H20B	109.5	O2—C45—C46	105.9 (2)
H20A—C20—H20B	109.5	O2—C45—H45A	110.6
N2—C20—H20C	109.5	C46—C45—H45A	110.6
H20A—C20—H20C	109.5	O2—C45—H45B	110.6
H20B—C20—H20C	109.5	C46—C45—H45B	110.6
N4—C21—C22	118.8 (2)	H45A—C45—H45B	108.7
N4—C21—C26	125.1 (2)	C47—C46—C45	105.0 (2)
C22—C21—C26	116.1 (2)	C47—C46—H46A	110.7
C23—C22—C21	122.6 (3)	C45—C46—H46A	110.7
C23—C22—H22	118.7	C47—C46—H46B	110.7
C21—C22—H22	118.7	C45—C46—H46B	110.7
C24—C23—C22	120.5 (2)	H46A—C46—H46B	108.8
C24—C23—H23	119.7	C48—C47—C46	105.4 (3)
C22—C23—H23	119.7	C48—C47—H47A	110.7
C23—C24—C25	118.0 (2)	C46—C47—H47A	110.7
C23—C24—H24	121.0	C48—C47—H47B	110.7
C25—C24—H24	121.0	C46—C47—H47B	110.7
C26—C25—C24	122.4 (3)	H47A—C47—H47B	108.8
C26—C25—H25	118.8	O2—C48—C47	105.8 (2)
C24—C25—H25	118.8	O2—C48—H48A	110.6
C25—C26—C21	120.5 (2)	C47—C48—H48A	110.6
C25—C26—N5	119.0 (2)	O2—C48—H48B	110.6

C21—C26—N5	120.5 (2)	C47—C48—H48B	110.6
C28—C27—N5	124.2 (2)	H48A—C48—H48B	108.7
C28—C27—C32	118.8 (2)	C1—N1—C18	122.71 (19)
N5—C27—C32	117.0 (2)	C1—N1—Fe1	112.67 (16)
C27—C28—C29	121.3 (3)	C18—N1—Fe1	124.62 (15)
C27—C28—H28	119.4	C12—N2—C13	112.8 (2)
C29—C28—H28	119.4	C12—N2—C20	117.01 (19)
C30—C29—C28	119.8 (3)	C13—N2—C20	111.86 (17)
C30—C29—H29	120.1	C7—N3—C19	118.6 (2)
C28—C29—H29	120.1	C7—N3—C6	113.33 (19)
C29—C30—C31	119.9 (3)	C19—N3—C6	115.48 (19)
C29—C30—H30	120.0	C21—N4—C38	122.4 (2)
C31—C30—H30	120.0	C21—N4—Fe1	121.30 (16)
C32—C31—C30	121.0 (3)	C38—N4—Fe1	115.99 (15)
C32—C31—H31	119.5	C27—N5—C26	114.68 (19)
C30—C31—H31	119.5	C27—N5—C39	120.1 (2)
C31—C32—N6	124.5 (2)	C26—N5—C39	115.07 (19)
C31—C32—C27	118.9 (3)	C32—N6—C40	116.8 (2)
N6—C32—C27	116.6 (2)	C32—N6—C33	111.40 (19)
C34—C33—C38	120.3 (2)	C40—N6—C33	113.1 (2)
C34—C33—N6	118.5 (2)	N4—Fe1—N1	126.64 (8)
C38—C33—N6	121.1 (2)	N4—Fe1—O1	100.46 (8)
C35—C34—C33	121.5 (3)	N1—Fe1—O1	116.49 (8)
C35—C34—H34	119.3	N4—Fe1—O2	117.67 (8)
C33—C34—H34	119.3	N1—Fe1—O2	97.67 (8)
C36—C35—C34	118.9 (3)	O1—Fe1—O2	93.90 (7)
C36—C35—H35	120.6	C41—O1—C44	109.2 (3)
C34—C35—H35	120.6	C44B—O1—C41B	108.2 (11)
C35—C36—C37	120.3 (2)	C44B—O1—Fe1	142.2 (8)
C35—C36—H36	119.8	C41—O1—Fe1	120.1 (2)
C37—C36—H36	119.8	C44—O1—Fe1	130.4 (2)
C36—C37—C38	121.8 (3)	C41B—O1—Fe1	106.7 (7)
C36—C37—H37	119.1	C48—O2—C45	106.1 (2)
C38—C37—H37	119.1	C48—O2—Fe1	121.89 (17)
C37—C38—C33	117.1 (2)	C45—O2—Fe1	127.18 (16)
C37—C38—N4	118.2 (2)		

N1—C1—C2—C3	172.5 (2)	C42—C43—C44—O1	−32.1 (5)
C6—C1—C2—C3	−3.5 (3)	O1—C41B—C42B—C43B	30 (3)
C1—C2—C3—C4	2.7 (4)	C41B—C42B—C43B—C44B	−18 (3)
C2—C3—C4—C5	0.0 (3)	C42B—C43B—C44B—O1	−3 (2)
C3—C4—C5—C6	−1.9 (3)	O2—C45—C46—C47	−17.9 (3)
C4—C5—C6—C1	1.0 (3)	C45—C46—C47—C48	−2.7 (3)
C4—C5—C6—N3	−177.8 (2)	C46—C47—C48—O2	22.5 (3)
N1—C1—C6—C5	−173.9 (2)	C6—C1—N1—C18	−27.5 (3)
C2—C1—C6—C5	1.6 (3)	C2—C1—N1—C18	157.1 (2)
N1—C1—C6—N3	4.8 (3)	C6—C1—N1—Fe1	152.43 (18)
C2—C1—C6—N3	−179.66 (19)	C2—C1—N1—Fe1	−23.0 (2)

N3—C7—C8—C9	−177.5 (3)	C13—C18—N1—C1	−36.1 (3)
C12—C7—C8—C9	2.7 (4)	C17—C18—N1—C1	147.9 (2)
C7—C8—C9—C10	1.2 (5)	C13—C18—N1—Fe1	144.0 (2)
C8—C9—C10—C11	−2.7 (5)	C17—C18—N1—Fe1	−31.9 (3)
C9—C10—C11—C12	0.2 (5)	C11—C12—N2—C13	122.2 (3)
C10—C11—C12—N2	−175.6 (3)	C7—C12—N2—C13	−57.1 (3)
C10—C11—C12—C7	3.7 (4)	C11—C12—N2—C20	−9.6 (4)
C8—C7—C12—C11	−5.2 (4)	C7—C12—N2—C20	171.1 (2)
N3—C7—C12—C11	175.0 (2)	C14—C13—N2—C12	−61.2 (3)
C8—C7—C12—N2	174.2 (2)	C18—C13—N2—C12	121.1 (2)
N3—C7—C12—N2	−5.6 (3)	C14—C13—N2—C20	73.2 (3)
C18—C13—C14—C15	2.5 (4)	C18—C13—N2—C20	−104.5 (3)
N2—C13—C14—C15	−175.2 (2)	C8—C7—N3—C19	−23.0 (4)
C13—C14—C15—C16	−0.7 (4)	C12—C7—N3—C19	156.8 (2)
C14—C15—C16—C17	−0.9 (4)	C8—C7—N3—C6	117.2 (3)
C15—C16—C17—C18	0.9 (4)	C12—C7—N3—C6	−63.0 (3)
C14—C13—C18—N1	−178.5 (2)	C5—C6—N3—C7	−64.9 (3)
N2—C13—C18—N1	−0.8 (4)	C1—C6—N3—C7	116.4 (2)
C14—C13—C18—C17	−2.4 (3)	C5—C6—N3—C19	76.6 (3)
N2—C13—C18—C17	175.2 (2)	C1—C6—N3—C19	−102.2 (2)
C16—C17—C18—N1	177.1 (2)	C22—C21—N4—C38	160.3 (2)
C16—C17—C18—C13	0.8 (4)	C26—C21—N4—C38	−23.6 (4)
N4—C21—C22—C23	177.1 (2)	C22—C21—N4—Fe1	−13.7 (3)
C26—C21—C22—C23	0.6 (4)	C26—C21—N4—Fe1	162.43 (19)
C21—C22—C23—C24	−0.2 (4)	C37—C38—N4—C21	139.0 (2)
C22—C23—C24—C25	0.0 (4)	C33—C38—N4—C21	−45.0 (3)
C23—C24—C25—C26	−0.2 (4)	C37—C38—N4—Fe1	−46.7 (3)
C24—C25—C26—C21	0.6 (4)	C33—C38—N4—Fe1	129.3 (2)
C24—C25—C26—N5	−178.6 (2)	C28—C27—N5—C26	120.0 (2)
N4—C21—C26—C25	−177.0 (2)	C32—C27—N5—C26	−62.0 (3)
C22—C21—C26—C25	−0.8 (3)	C28—C27—N5—C39	−23.7 (3)
N4—C21—C26—N5	2.2 (4)	C32—C27—N5—C39	154.3 (2)
C22—C21—C26—N5	178.4 (2)	C25—C26—N5—C27	−62.8 (3)
N5—C27—C28—C29	−177.1 (2)	C21—C26—N5—C27	118.0 (2)
C32—C27—C28—C29	4.9 (3)	C25—C26—N5—C39	82.8 (3)
C27—C28—C29—C30	0.8 (4)	C21—C26—N5—C39	−96.4 (3)
C28—C29—C30—C31	−3.9 (4)	C31—C32—N6—C40	−10.8 (3)
C29—C30—C31—C32	1.2 (4)	C27—C32—N6—C40	170.5 (2)
C30—C31—C32—N6	−174.2 (2)	C31—C32—N6—C33	121.3 (2)
C30—C31—C32—C27	4.5 (4)	C27—C32—N6—C33	−57.5 (3)
C28—C27—C32—C31	−7.5 (3)	C34—C33—N6—C32	−62.3 (3)
N5—C27—C32—C31	174.4 (2)	C38—C33—N6—C32	114.5 (3)
C28—C27—C32—N6	171.4 (2)	C34—C33—N6—C40	71.6 (3)
N5—C27—C32—N6	−6.7 (3)	C38—C33—N6—C40	−111.6 (3)
C38—C33—C34—C35	−0.7 (4)	C43B—C44B—O1—C41B	23 (3)
N6—C33—C34—C35	176.1 (2)	C43B—C44B—O1—Fe1	179.4 (10)
C33—C34—C35—C36	−1.9 (4)	C42—C41—O1—C44	2.4 (8)
C34—C35—C36—C37	2.4 (4)	C42—C41—O1—Fe1	−171.7 (4)

C35—C36—C37—C38	−0.3 (4)	C43—C44—O1—C41	18.9 (6)
C36—C37—C38—C33	−2.3 (4)	C43—C44—O1—Fe1	−167.9 (2)
C36—C37—C38—N4	174.1 (2)	C42B—C41B—O1—C44B	−34 (3)
C34—C33—C38—C37	2.7 (4)	C42B—C41B—O1—Fe1	161.2 (16)
N6—C33—C38—C37	−174.0 (2)	C47—C48—O2—C45	−34.6 (3)
C34—C33—C38—N4	−173.3 (2)	C47—C48—O2—Fe1	122.5 (2)
N6—C33—C38—N4	9.9 (4)	C46—C45—O2—C48	32.7 (3)
O1—C41—C42—C43	−22.4 (7)	C46—C45—O2—Fe1	−122.7 (2)
C41—C42—C43—C44	33.5 (6)		