Appendix A
Attempted Synthesis of Pyridine Bis(anilide) Scandium Complexes

Introduction

Organoscandium complexes have attracted widespread interest due to their ability to carry out a myriad of reactions: notably sigma bond metathesis, various insertion chemistry, and olefin polymerization. With regards to olefin polymerization, scandocene alkyls have provided valuable mechanistic insight; they are isoelectronic to cationic group IV metallocenes, the presumed active species in Ziegler-Natta olefin polymerization. Though the most well-studied scandium systems are undoubtedly metallocene-based, the past decade has seen a concerted effort to discover non-metallocene systems that incorporate the advantages of metallocenes but alleviate certain drawbacks.[1–5] Herein are described attempts at using ligands of the type in Figure A.1 for organoscandium chemistry. The possible conformations of these ligands mirror symmetries exhibited by ansa-metallocenes; it is well-established that the symmetries of ansa-metallocenes are strongly related to tacticity control in the polymerization of the α-olefins.[6]

Results and Discussion

Reaction of [^{Mes}NNN]H_2 with scandium alkyls was performed to obtain the

(1) Gromada, J.; Carpentier, J.; Mortreux, A. *Coord. Chem. Rev.* **2004**, *248*, 397–410.

(2) Gibson V. C.; Spitzmesser, S. K. *Chem. Rev.* **2003**, *103,* 283–315.

(3) Mountford, P.; Ward, B. D. *Chem. Commun.* **2003**, 1797–1803.

(4) Zeimentz, P. M.; Arndt, S.; Elvidge, B. R.; Okuda, J. *Chem. Rev.* **2006**, *106*, 2404–2433.

(5) Resconi, L.; Cavallo, L.; Fait, A.; Piemontesi, F. *Chem. Rev.* **2000**, *100,* 1253–1345.

(6) Coates, G. W. *Chem. Rev.* **2000**, *100*, 1223–1252, and references contained therein.

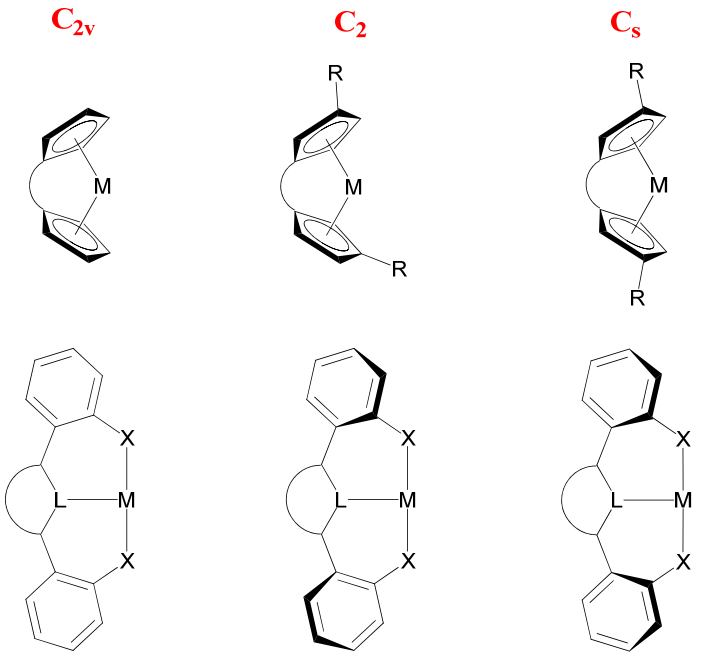
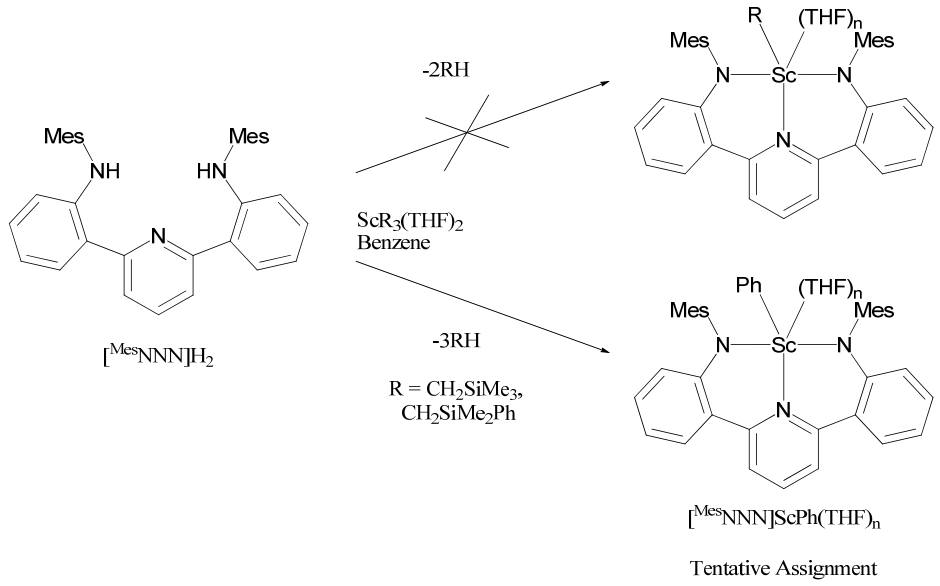


Figure A.1. Different symmetries exhibited by ansa-metallocenes (top), and analogous symmetries for the ligands herein (bottom).

desired monoalkylated product via double alkane elimination. Instead, triple alkane elimination was observed to form the scandium phenyl complex [^{Mes}NNN]ScPh(THF)$_n$ (Scheme A.1). Presumably, this reaction occurs via a transient scandium monoalkyl, which reacts via sigma bond metathesis with benzene to form the more stable scandium aryl linkage.[7] Also consistent with this explanation is the observation of the same product despite the use of different scandium alkyls. The three 6:6:6 singlets in the ^{1}H NMR spectrum of the compound were assigned as the mesityl methyl protons. This data is consistent with a structure exhibiting hindered bond rotation about the mesityl ipso carbon nitrogen bond, and

(7) Thompson, M. E.; Baxter, S. M.; Bulls, A. R.; Burger, B. J.; Nolan, M. C.; Santarsiero, B. D.; Schaefer, W. P.; Bercaw, J. E. *J. Am. Chem. Soc.* **1987**, *109*, 203–219.



Scheme A.1. Reaction of $[^{Mes}NNN]H_2$ with scandium trialkyls.

an axial phenyl on scandium. Preparation of the scandium phenyl complex using protio benzene was undertaken in an effort to observe resonances corresponding to the phenyl protons. This attempt was probably precluded by fast sigma bond metathesis with the deuterated solvent; thus samples prepared in deutero or protio solvents produced identical NMR spectra.

Though initially a promising compound, several factors prevented the aforementioned complex from being a useful synthon for organoscandium chemistry. First, the scandium alkyl precursors used above typically require very short reaction times even at subambient temperatures for alkane elimination (~30 minutes to one hour).[8–10] This amount of time is small on the timescale of their

(8) Tredget, C. S.; Lawrence, S. C.; Ward, B. D.; Howe, R. G.; Cowley, A. R.; Mountford, P. *Organometallics* **2005**, *24*, 3136–3148.

(9) Ward, B. D.; Bellemin-Laponnaz, S.; Gade, L. H. *Angew. Chem. Int. Ed.* **2005**, *44*, 1668–1671.

decomposition reactions at room temperature. The metallation described herein, however, demands almost two weeks to reach completion at room temperature and high concentration. This implies that scandium alkyl decomposition competes with ligand metallation, and free ligand is invariably obtained even with greater than 1 equivalent of scandium alkyl. Mild heating of the reaction resulted in product decomposition and other unidentified products.

Second, the high reactivity of the product with solvents made its purification prohibitively difficult. Reactivity was observed even with titanocene dihydride dried solvents, so residual water content is an unlikely culprit. It has been observed that sufficiently reactive scandium alkyls and aryls undergo sigma bond metathesis with aryl and even primary C-H bonds, which is the likely explanation for the observed decomposition when solvents other than benzene were used. Despite the fact that benzene would itself fall into the aforementioned category, sigma bond metathesis would be degenerate and thus one species would be observed. Though there is some precedent for cyclohexane as a suitably inert solvent for such early metal hydrocarbyls,[7] attempted preparation of the scandium monoalkyl in C_6D_{12} led to complete decomposition, possibly due to the extremely low solubility of the reactants in cyclohexane.

In light of the challenges observed with alkane elimination, salt metathesis was explored as an alternative method of ligand metallation. Reaction of $[^{Mes}NNN]H_2$ with n-butyllithium in tetrahydrofuran led to the dilithium salt hexakis(THF)

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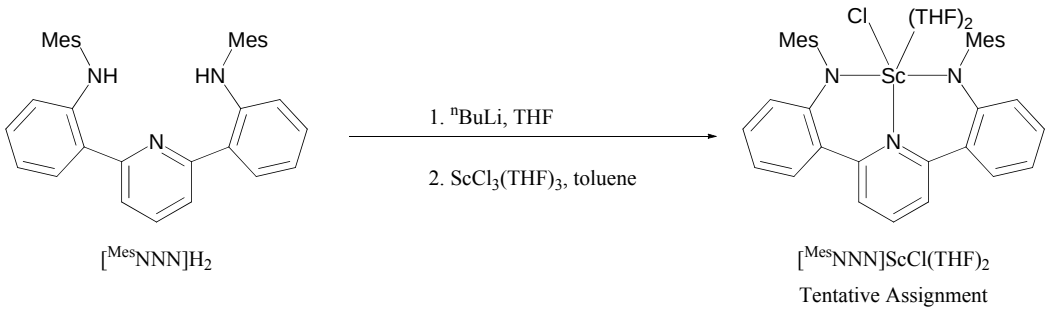
(10) Estler, F.; Eickerling, G.; Herdtweck, E.; Anwander, R. *Organometallics* **2003**, *22*, 1212–1222.

adduct. The deprotonated ligand was then treated with $ScCl_3(THF)_3$ in toluene (Scheme A.2). In contrast to the scandium phenyl complex, only two ^1H resonances in a 12:6 ratio were observed, indicating either some degree of fluxionality on the NMR timescale, a chloride ligand in the plane of the ligand, or both. Unfortunately, the putative scandium chloride begins decomposing over the course of a day, in solution or the solid state, to a dark, insoluble solid. All attempts at obtaining



Scheme A.2. Deprotonation of $[^{Mes}NNN]H_2$ and subsequent salt metathesis with $ScCl_3(THF)_3$.

crystalline material inevitably led to precipitation of this solid. The preparation of the scandium chloride also suffered from reproducibility issues, the reasons for this problem are unclear. It is possible that the aforementioned decomposition results from loss of THF and subsequent dimerization or oligomerization. For that reason, preparation of the compound was attempted in deuterated THF. Unfortunately, formation of the complex was not observed. It is possible that THF loss from the scandium starting material is required for subsequent metallation, and thus the solvent inhibits the metallation. Additionally, the *in situ* generated scandium chloride was treated with excess trimethylphosphine. Surprisingly, this led to complete decomposition to many unidentified products.

Finally, amine elimination was attempted as an entry into scandium compounds of the ligand. Although less common than other methods for scandium installation, there is some precedent for its effectiveness.[11–13] Generation of the 3-coordinate tris(bis(trimethylsilyl)amide) scandium (III) was easily accomplished using a modified procedure from the original paper by Bradley.[14] This starting material has the advantage of being base free, and double amine elimination would be anticipated to lead to the four coordinate ligand scandium amide. A high degree of coordinative unsaturation has been shown to afford highly reactive complexes.[15] Unfortunately, refluxing a benzene solution of the tris(amide) with the ligand did not lead to a reaction. Additonally, refluxing a solution of the reactants in xylenes under an argon purge eventually led to decomposition of the scandium tris(amide). This result is surprising considering the large pK_a difference between amines and anilines, and the low kinetic barrier expected for a tricoordinate scandium center. The significant steric bulk of the ligand, however, may be the cause of this result.

In light of the apparent instability of the pyridine bis(aniline) system with scandium, a new strategy was employed to impart greater stability than the

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(11) Skinner, M. E. G.; Mountford, P. *Dalton Trans*. **2002**, 1694–1703.

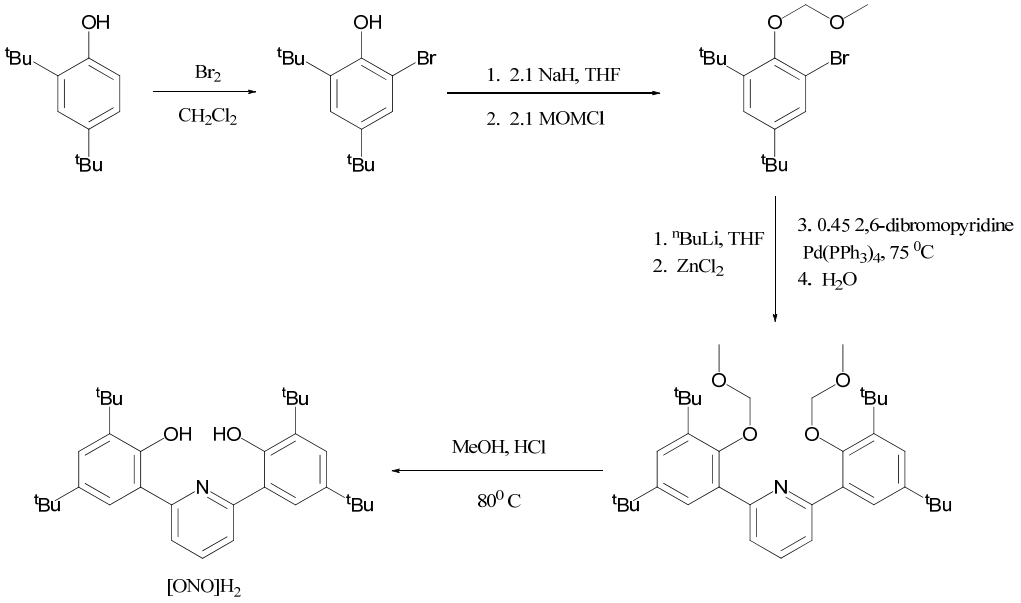
(12) Covert, K. J.; Neithamer, D. R.; Zonnevylle, M. C.; LaPointe, R. E.; Schaller, C. P.; Wolczanski, P. T. *Inorg. Chem.* **1991**, *30*, 2494–2508.

(13) Hitchcock, P. B.; Lappert, M. F.; Singh, A. *Chem. Comm.* **1983**, 1499–1501.

(14) Alyea, E. C.; Bradley, D. C.; Copperthwaite, R. G. *J. Chem. Soc., Dalton Trans.* **1972**, 1580–1584.

(15) Thompson, M. E.; Bercaw, J.E. *Pure Appl. Chem.* **1984**, *56*, 1–11.

previous system while still exploring a semi-rigid ligand as in Figure A.1. Specifically, the replacement of the anilines with phenols would be predicted to induce stability, as early metal oxygen bonds are known to be extremely strong. Additionally, the higher acidity of phenols compared to anilines could facilitate alkane and amine eliminations. Thus, the synthesis of the pyridine bis(phenol) $[ONO]H_2$ was undertaken using a known procedure (Scheme A.3).[16] Unfortunately, reaction of $[ONO]H_2$ with either $Sc(CH_2SiMe_3)_3(THF)_2$ or $Sc(N(SiMe_3)_2)_3$ led to unidentifiable mixtures. Reaction of the dilithium salt $[ONO]Li_2$ also resulted in decomposition, and the use of the bis(phenol) was not explored further.



Scheme A.3. Synthesis of $[ONO]H_2$

(16) Agapie, T. Synthetic, Reactivity, and Mechanistic Studies Relevant to Olefin Oligomerization and Polymerization. PhD Thesis, California Institute of Technology, Pasadena, CA, January 2007.

Conclusions

The surprising instability of scandium complexes based on ^{Mes}NNN or ONO leads to the conclusion that they cannot provide useful entry points into organoscandium chemistry. This may be partially due to the small binding pocket of the ligand and relatively large size of scandium. Another factor is formation of six membered rings by the chelate, which might require severe distortion of the ligand framework to give favorable bond angles.

Experimental

General Methods: Unless otherwise specified, air exposed solids were dried under vacuum prior to use, liquids were degassed or bubbled with argon, reagents were used as received from the supplier, benzene, toluene, petroleum ether, xylenes, cyclohexane and diethyl ether were dried with titanocene dihydride for at least two days prior to use, and reactions were performed under inert atmosphere or vacuum. Tetrahydrofuran was dried via disodium benzophenone. Deuterated solvents were obtained from Cambridge Isotope Laboratories. Lithium granules, lithium bis(trimethylsilyl)amide, chloromethyldimethylphenylsilane, 2,4-ditertbutylphenol, bromine, methoxymethylchloride, zinc (II) chloride, and tetrakis(triphenylphosphine) palladium (0) were obtained from Aldrich. Chloromethyldimethylphenylsilane was exposed to the atmosphere, but degassed prior to use. [ONO]H_2 was prepared as reported previously.[16] Trichloro-tris(tetrahydrofuran) scandium (III) was prepared in an analogous manner to the

published procedure.[17,18] Dimethylphenylsilylmethyllithium and tris(dimethylphenylsilylmethyl)bis(tetrahydrofuran) scandium (III) were synthesized as described previously by Piers,[19] and the toluene used for these preparations was dried via Grubbs' method. Tris(bis(dimethylsilylamido)) scandium (III) was prepared in a similar manner to the reported procedure,[14] except $ScCl_3(THF)_3$ and $LiNSi(Me_3)_2$ were prepared beforehand and not generated *in situ*. NMR spectra were recorded on Varian Mercury 300 Megahertz NMR spectrometers, and referenced according to the solvent residual peak.

Synthesis of [^{Mes}NNN]ScPh(THF)$_n$. $Sc(CH_2SiMe_3)_3(THF)_2$ was generated *in situ* by allowing $LiCH_2SiMe_3$ (5 mg, 53 μmol) and $ScCl_3(THF)_3$ (6.5 mg, 17.7 μmol) to react in C_6D_6. After ~30 minutes, no $LiCH_2SiMe_3$ was detectable by ^{1}H NMR and the solution was cloudy. [^{Mes}NNN]H_2 (8.8 mg, 17.7 μmol) was added, and the reaction was complete after 13 days. ^{1}H NMR (C_6D_6): δ 1.21 (s, $OCH_2C\underline{H}_2$), 2.07 (s, 6H, $C\underline{H}_3$), 2.24 (s, 6H, $C\underline{H}_3$), 2.37 (s, 6H, $C\underline{H}_3$), 3.50 (s, $OC\underline{H}_2CH_2$), 6.5–7.6 (m, 13H, aryl-CH). The complexity of the aryl region precludes specific assignments.

Synthesis of [^{Mes}NNN]ScCl(THF)$_2$. A chilled solution of [^{Mes}NNN]H_2 (400 mg, 0.80 mmol) in 30 mL of THF was treated with 7.4 mL of a chilled THF/hexanes solution (6:1.4 THF:hexanes) of n-butyllithium (3.5 mmol, 0.49 M) over the course of five minutes. After 2 hours, the reaction was concentrated in vacuo. The

(17) Stotz, R. W.; Melson, G. A. *Inorg. Chem.* **1972**, *11*, 1720–1721.

(18) Atwood, J. L.; Smith, K. D. *Dalton Trans* **1974**, 921–923.

(19) David J. H. Emslie, D. J. H.; Piers, W. E.; Parvez, M.; McDonald, R. *Organometallics* **2002**, *21*, 4226–4240.

remaining solid was washed with 2 mL of cold petroleum ether, then dried in vacuo. A total of 477 mg were obtained. A solution of the deprotonated ligand (200 mg, 0.21 mmol) in 6 mL of toluene was added to solid $ScCl_3(THF)_3$ (78 mg, 0.21 mmol). The solution became very dark and white solid precipitated (presumably lithium chloride). After one hour, the reaction was filtered and the remaining white solid was washed with 1 mL of toluene. The filtrate was concentrated in vacuo, giving 141 mg of $[^{Mes}NNN]ScCl(THF)_2$.

^{1}H NMR (C_6D_6): δ 1.07(s, 4H $OCH_2C\underline{H}_2$), 1.93 (s, 12H, *o*-$C\underline{H}_3$), 2.38 (s, 6H, *p*-$C\underline{H}_3$), 3.08 (s, 4H, $OC\underline{H}_2CH_2$), 6.47 (t, $J_{H\text{-}H}$ = 7 Hz, 2H, C<u>H</u>), 6.56 (d, $J_{H\text{-}H}$ = 8.7 Hz, 2H, C<u>H</u>), 6.90 (s, 4H, mesityl aryl C<u>H</u>), 7.03 (t, $J_{H\text{-}H}$ = 7.6 Hz, 2H, C<u>H</u>), 7.24–7.36 (m, 3H, C<u>H</u>), 7.62 (d, $J_{H\text{-}H}$ = 8.4 Hz, 2H, C<u>H</u>).

Appendix B
Attempted Synthesis of other Transition Metal Pyridine Bis(anilide) Complexes

The application of the ONO pincer ligand has been realized for a variety of transition metals: Ti, Zr, Hf, V, Ta, Fe, and Ir. It was attempted to similarly expand the scope of the NNN ligand beyond iron chemistry. Additionally, metallations of ONO were tried for certain previously untested metals. A brief list of unsuccessful metallation reactions with Nb, Ta, Ru, Cu, Zn, and Cd is given in this appendix.

Given the successful application of the ONO ligand set to tantalum, it was desirable to investigate the analogous NNN complexes with group 5 metals. Both metathetical and methane elimination routes were tested (Table B.1), but inevitably led to decomposition.

Table B.1. Attempted group 5 metallations.

Ligand	Metal Source	Solvent, temp	Result
$[^{Mes}NNN]Li_2$	$NbCl_5$	C_6D_6, RT	Decomposition
$[^{Mes}NNN]Li_2$	$TaCl_5$	C_6D_6, RT	Decomposition
$[^{Mes}NNN]Li_2$	Me_3Ta Cl_2	C_6D_6, RT	Decomposition
$[^{Mes}NNN]H_2$	Me_3Ta Cl_2	C_6D_6, RT	No reaction, decomposition upon heating

Investigation of ruthenium pincer complexes was inspired by the ability of iron congeners to initiate intramolecular C-H activation via a high-valent iron imido species. It was thought that a high-valent ruthenium imide would be more stable, and thus more amenable to characterization and intermolecular reactivity. With the knowledge from the iron study, it was known that the $[^{Mes}NNN]H_2$ ligand decomposed via benzylic C-H activation. Thus, metallation focused on the 3,5-

ditbutylphenyl substituted ligand, [^{tBu}NNN]H_2, which contains no reactive benzylic C-H bonds (Table B.2). Ruthenium ONO complexes were also briefly investigated.

Copper metallation was briefly attempted because of its similarity to iron in terms of redox properties (Table B.3). Zinc and cadmium complexes of [^{Mes}NNN] were attempted to investigate the electrochemical properties of the NNN ligand with a redox-inactive metal. The results could then be compared the iron system, and the peaks observed for the iron complex could be assigned as primarily metal or ligand based. No attempts to make group 10 complexes of the [^{Mes}NNN] ligand were successful, however.

Table B.2. Attempted ligation of ruthenium.

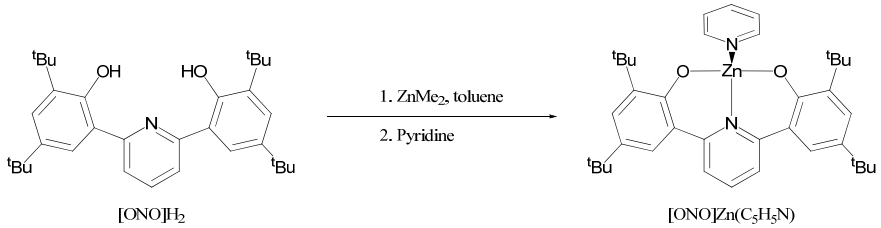
Ligand	Metal Source (base)	Solvent, temp	Result
[^{tBu}NNN]Li_2	$[RuCl_2(C_6H_6)]_2$	d_8-THF, RT	Decomposition upon heating
[^{tBu}NNN]Li_2	(*p*-cymene)$RuCl_2(PCy_3)$	C_6D_6, RT	Decomposition
[^{tBu}NNN]Li_2	$(Ph_3P)_3RuCl_2$	C_6D_6, RT	2 major products
[^{tBu}NNN]Li_2	$(DMSO)_4RuCl_2$	C_6D_6,70 °C	No reaction, eventual ligand protonation
[^{tBu}NNN]Li_2	$RuCl_3$	d_8-THF, RT	Ligand protonation
[^{tBu}NNN]H_2	(*p*-cymene)$RuCl_2(PCy_3)$ (C_5H_5N)	C_6D_6, 90 °C	No reaction
[^{tBu}NNN]H_2	Ru metal	d_8-THF, 70 °C	No reaction
[^{tBu}NNN]H_2	$[RuCl_2(C_6H_6)]_2$ (NEt_3)	C_6D_6, 90 °C	No reaction
[^{tBu}NNN]H_2	(*p*-cymene)$RuCl_2(PCy_3)$ (NEt_3)	C_6D_6, RT	Decomposition upon heating
[ONO]Li_2	$(Ph_3P)_3RuCl_2$	C_6D_6, 80 °C	Metal source decomposition
[ONO]H_2	$(Ph_3P)_3RuCl_2$ (NEt_3)	d_8-isopropanol, 85 °C	No reaction

Table B.3. Attempted metallations with groups 9 and 10.

Ligand	Metal Source	Solvent, temp	Result
[^{Mes}NNN]Li_2	$CuCl_2$	d_8-THF, RT	Ligand protonation and decomposition
[^{Mes}NNN]H_2	$ZnMe_2$	C_6D_6, 90 °C	Inconclusive
[^{Mes}NNN]H_2	$ZnEt_2$	d_8-toluene, 120 °C	Decomposition
[^{Mes}NNN]Li_2	$ZnCl_2$	d_8-THF, RT	Mulitple products

$[^{Mes}NNN]Li_2$	$CdCl_2$	THF, RT	In solution, product decomposes to a gray solid over time. Crystallization attempts unsuccessful.
$[ONO]H_2$	$ZnMe_2$	Toluene/pyridine, RT	Successful metallation

An ONO zinc complex was accessible however, as the pyridine adduct (Scheme B.1). Mixing $[ONO]H_2$ and $ZnMe_2$ in toluene produces an



Scheme B.1. Synthesis of $[ONO]Zn(C_5H_5N)$.

intermediate complex. The intermediate is unlikely monomeric, in analogy to the known non-t-butyl substituted pyridine bis(phenoxide) zinc complex $([^{H}ONO]Zn)_n$, which is proposed to be a polymer.[1] Addition of pyridine produces a new complex by ^1H NMR, assigned as $[ONO]Zn(C_5H_5N)$. ^1H and ^{13}C NMR spectra confirm the 1:1 ratio of ONO ligand to pyridine. While $[^{H}ONO]Zn(C_5H_5N)$ is a tetramer, preliminary DOSY measurements indicated that $[ONO]Zn(C_5H_5N)$ is monomeric. Some caution must be taken with this statement however, as the standard for the DOSY solution was the solvent residual signal, whose hydrodynamic radius is significantly different from the product. The complex $[ONO]Zn(C_5H_5N)$ was not explored further.

(1) Zhang, H.-Y.; Ye, K.-Q.; Jing-Ying Zhang, J.-Y.; Liu, Y.; Wang, Y. *Inorg. Chem.* **2006**, *45*, 1745–1753.

Experimental

Synthesis of [ONO]Zn(C_5H_5N). 25 mL of toluene were vac. transferred from a Cp_2TiH_2 pot onto the solid $[ONO]H_2$ ligand (300.9 mg, 0.6170 mmol). Dimethylzinc (0.50 mL, 7.3 mmol) was vac. transferred onto the solution. Bubbling occurred for several minutes, and the solution was a golden yellow. ^{1}H NMR of "$([ONO]Zn)_n$" (C_6D_6, 300 MHz): 1.33 (s, 18H, $C(CH_3)_3$), 1.63 (s, 18H, $C(CH_3)_3$), 7.02–7.12 (m, 3H, CH), 7.41 (d, J_{H-H} = 3 Hz, 2H, CH), 7.66 (d, J_{H-H} = 3 Hz, 2H, CH). After two hours, the volatiles were removed in vacuo. To remove any residual dimethylzinc trapped in the solid, 25 mL of pentane were vac. transferred onto the solid. The solid dissolved upon warming to room temperature, and the pentane was removed in vacuo. 10 mL of pyridine were vac. transferred from a sodium pot onto the solid, giving an orange-brown solution. The solution was left stirring for 12 hours, then concentrated in vacuo. 30 mL of pentane were vac. transferred onto the solid, and the resulting suspension was left stirring for 30 minutes. The pentane was removed in vacuo. In a glovebox, the solid was dissolved in 10 mL of toluene. The solution was diluted with 30 mL of heptane, and cooled to -30 °C. After 2.5 hours, the cold solution was filtered, and the light solid was dried in vacuo. 148.8 mg of $[ONO]Zn(C_5H_5N)$ were obtained in 38% yield. ^{1}H NMR (C_6D_6, 400 MHz): δ 1.36 (s, 18H, $C(C\underline{H}_3)_3$), 1.42 (s, 18H, $C(C\underline{H}_3)_3$), 6.59 (t, J_{H-H} = 6 Hz, 2H, *m*-$C_5\underline{H}_5N$), 6.89 (t, J_{H-H} = 7 Hz, 1H, *p*-$C_5\underline{H}_5N$), 7.29 (m, 3H, overlap of *m*-$C_5\underline{H}_3Ar_2N$ and *p*-$C_5\underline{H}_3Ar_2N$), 7.54 (d, J_{H-H} = 3 Hz, 2H, $C_6\underline{H}_2$), 7.66 (d, J_{H-H} = 3 Hz, 2H, $C_6\underline{H}_2$), 8.64 (d, J_{H-H} = 5 Hz, 2H, *o*-$C_5\underline{H}_5N$). ^{13}C NMR (C_6D_6, 101

MHz): δ 30.09, 31.41, 34.41, 35.53, 122.38, 124.53, 124.67, 124.84, 127.50, 138.79, 140.02, 140.09, 140.94, 149.64, 158.15, 159.79.

Appendix C
Crystallographic Tables

CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE
X-RAY CRYSTALLOGRAPHY LABORATORY

Date 10 July 2007

Crystal Structure Analysis of:

[^{Mes}NNN]H_2 (ECW01)

(shown below)

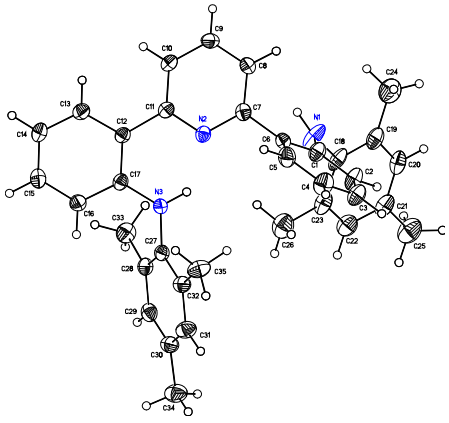
For Investigator: Edward Weintrob ext. 6576

Advisor: J. E. Bercaw ext. 6577

Account Number: JEB.65152-1DOE.651520

By Michael W. Day 116 Beckman ext. 2734
e-mail: mikeday@caltech.edu

Contents



ECW01

Note: The crystallographic data have been deposited in the Cambridge Database (CCDC) and has been placed on hold pending further instructions from me. The deposition number is 653538. Ideally the CCDC would like the publication to contain a footnote of the type: "Crystallographic data have been deposited at the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK and copies can be obtained on request, free of charge, by quoting the publication citation and the deposition number 653538."

Table 1. Crystal data and structure refinement for ECW01 (CCDC 653538).

Empirical formula	$C_{35}H_{35}N_3$
Formula weight	497.66
Crystallization Solvent	Diethyl ether
Crystal Habit	Blade
Crystal size	0.39 x 0.22 x 0.09 mm^3
Crystal color	Colorless

Data Collection

Type of diffractometer	Bruker SMART 1000
Wavelength	0.71073 Å MoKα
Data Collection Temperature	100(2) K
θ range for 8568 reflections used in lattice determination	2.54 to 27.95°
Unit cell dimensions	a = 12.1774(12) Å b = 8.3901(8) Å β= 93.803(2)° c = 27.039(3) Å
Volume	2756.5(5) $Å^3$
Z	4
Crystal system	Monoclinic
Space group	$P2_1/n$
Density (calculated)	1.199 Mg/m^3
F(000)	1064
Data collection program	Bruker SMART v5.630
θ range for data collection	1.51 to 28.41°
Completeness to θ = 28.41°	93.2 %
Index ranges	$-15 \le h \le 16$, $-10 \le k \le 11$, $-36 \le l \le 35$
Data collection scan type	ω scans at 5 ϕ settings
Data reduction program	Bruker SAINT v6.45A
Reflections collected	34841
Independent reflections	6474 [R_{int}= 0.0831]
Absorption coefficient	0.070 mm^{-1}
Absorption correction	None
Max. and min. transmission	0.9937 and 0.9732

Table 1 (cont.)

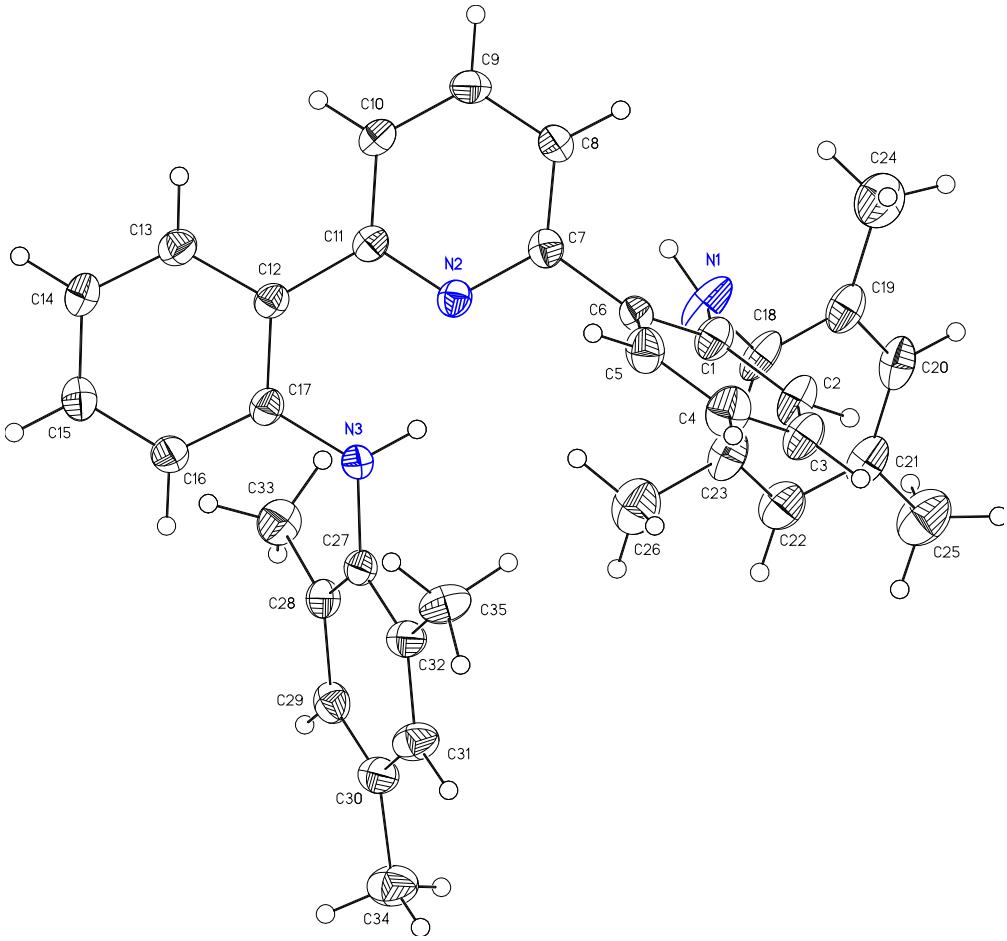
Structure solution and Refinement

Structure solution program	SHELXS-97 (Sheldrick, 1990)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	SHELXL-97 (Sheldrick, 1997)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	6474 / 0 / 349
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	1.579
Final R indices [$I>2\sigma(I)$, 4122 reflections]	R1 = 0.0554, *w*R2 = 0.0907
R indices (all data)	R1 = 0.0900, *w*R2 = 0.0939
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.000
Average shift/error	0.000
Largest diff. peak and hole	0.387 and -0.364 e.Å^{-3}

Special Refinement Details

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

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.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C34

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for ECW01 (CCDC 653538). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
N(1)	1038(1)	3989(2)	1311(1)	39(1)
N(2)	3731(1)	5158(2)	1856(1)	21(1)
N(3)	4904(1)	2425(2)	1759(1)	24(1)
C(1)	1569(1)	5009(2)	998(1)	27(1)
C(2)	1265(1)	5049(2)	493(1)	33(1)
C(3)	1798(1)	6037(2)	181(1)	34(1)
C(4)	2639(1)	7005(2)	366(1)	36(1)
C(5)	2952(1)	6967(2)	866(1)	31(1)
C(6)	2434(1)	5978(2)	1186(1)	22(1)
C(7)	2781(1)	5934(2)	1727(1)	22(1)
C(8)	2159(1)	6639(2)	2073(1)	27(1)
C(9)	2504(1)	6534(2)	2570(1)	28(1)
C(10)	3462(1)	5726(2)	2705(1)	25(1)
C(11)	4076(1)	5057(2)	2340(1)	21(1)
C(12)	5122(1)	4202(2)	2469(1)	20(1)
C(13)	5747(1)	4626(2)	2903(1)	25(1)
C(14)	6718(1)	3870(2)	3052(1)	30(1)
C(15)	7103(1)	2667(2)	2758(1)	29(1)
C(16)	6516(1)	2220(2)	2327(1)	26(1)
C(17)	5517(1)	2946(2)	2180(1)	22(1)
C(18)	458(2)	2616(2)	1119(1)	31(1)
C(19)	-685(2)	2578(2)	1105(1)	31(1)
C(20)	-1216(2)	1243(2)	903(1)	34(1)
C(21)	-652(2)	-34(2)	722(1)	33(1)
C(22)	485(1)	30(2)	744(1)	36(1)
C(23)	1059(1)	1350(2)	938(1)	34(1)
C(24)	-1328(2)	3938(2)	1312(1)	45(1)
C(25)	-1250(2)	-1478(2)	512(1)	49(1)
C(26)	2300(1)	1393(2)	948(1)	45(1)
C(27)	5218(1)	1155(2)	1452(1)	22(1)
C(28)	5264(1)	-416(2)	1626(1)	25(1)
C(29)	5539(1)	-1613(2)	1302(1)	27(1)
C(30)	5737(1)	-1313(2)	812(1)	28(1)
C(31)	5673(1)	251(2)	649(1)	30(1)
C(32)	5430(1)	1503(2)	963(1)	25(1)
C(33)	4974(1)	-839(2)	2143(1)	34(1)
C(34)	5955(1)	-2667(2)	464(1)	37(1)
C(35)	5391(1)	3205(2)	784(1)	34(1)

Table 3. Bond lengths [Å] and angles [°] for ECW01 (CCDC 653538).

N(1)-C(1)	1.3935(19)	N(1)-C(1)-C(6)	120.22(14)
N(1)-C(18)	1.430(2)	C(3)-C(2)-C(1)	120.67(16)
N(2)-C(11)	1.3511(18)	C(4)-C(3)-C(2)	120.27(16)
N(2)-C(7)	1.3525(18)	C(3)-C(4)-C(5)	119.50(16)
N(3)-C(17)	1.3900(17)	C(6)-C(5)-C(4)	121.31(16)
N(3)-C(27)	1.4191(19)	C(5)-C(6)-C(1)	118.94(15)
C(1)-C(2)	1.390(2)	C(5)-C(6)-C(7)	121.10(14)
C(1)-C(6)	1.400(2)	C(1)-C(6)-C(7)	119.96(14)
C(2)-C(3)	1.376(2)	N(2)-C(7)-C(8)	122.15(14)
C(3)-C(4)	1.374(2)	N(2)-C(7)-C(6)	116.41(14)
C(4)-C(5)	1.383(2)	C(8)-C(7)-C(6)	121.44(14)
C(5)-C(6)	1.381(2)	C(7)-C(8)-C(9)	119.20(15)
C(6)-C(7)	1.496(2)	C(10)-C(9)-C(8)	119.07(15)
C(7)-C(8)	1.376(2)	C(9)-C(10)-C(11)	119.67(14)
C(8)-C(9)	1.383(2)	N(2)-C(11)-C(10)	120.90(14)
C(9)-C(10)	1.378(2)	N(2)-C(11)-C(12)	117.71(13)
C(10)-C(11)	1.395(2)	C(10)-C(11)-C(12)	121.39(14)
C(11)-C(12)	1.483(2)	C(13)-C(12)-C(17)	117.77(14)
C(12)-C(13)	1.4016(19)	C(13)-C(12)-C(11)	118.97(14)
C(12)-C(17)	1.416(2)	C(17)-C(12)-C(11)	123.25(13)
C(13)-C(14)	1.378(2)	C(14)-C(13)-C(12)	122.38(15)
C(14)-C(15)	1.386(2)	C(13)-C(14)-C(15)	118.92(15)
C(15)-C(16)	1.376(2)	C(16)-C(15)-C(14)	120.50(15)
C(16)-C(17)	1.395(2)	C(15)-C(16)-C(17)	121.12(15)
C(18)-C(19)	1.390(2)	N(3)-C(17)-C(16)	120.52(14)
C(18)-C(23)	1.396(2)	N(3)-C(17)-C(12)	120.20(13)
C(19)-C(20)	1.387(2)	C(16)-C(17)-C(12)	119.27(14)
C(19)-C(24)	1.512(2)	C(19)-C(18)-C(23)	121.33(16)
C(20)-C(21)	1.380(2)	C(19)-C(18)-N(1)	119.83(17)
C(21)-C(22)	1.384(2)	C(23)-C(18)-N(1)	118.83(16)
C(21)-C(25)	1.505(2)	C(20)-C(19)-C(18)	117.95(17)
C(22)-C(23)	1.393(2)	C(20)-C(19)-C(24)	121.01(16)
C(23)-C(26)	1.511(2)	C(18)-C(19)-C(24)	121.03(16)
C(27)-C(32)	1.394(2)	C(21)-C(20)-C(19)	122.48(17)
C(27)-C(28)	1.400(2)	C(20)-C(21)-C(22)	118.34(17)
C(28)-C(29)	1.390(2)	C(20)-C(21)-C(25)	121.32(17)
C(28)-C(33)	1.505(2)	C(22)-C(21)-C(25)	120.34(18)
C(29)-C(30)	1.385(2)	C(21)-C(22)-C(23)	121.50(18)
C(30)-C(31)	1.385(2)	C(22)-C(23)-C(18)	118.39(16)
C(30)-C(34)	1.509(2)	C(22)-C(23)-C(26)	120.14(18)
C(31)-C(32)	1.395(2)	C(18)-C(23)-C(26)	121.47(16)
C(32)-C(35)	1.507(2)	C(32)-C(27)-C(28)	120.76(15)
		C(32)-C(27)-N(3)	118.11(14)
C(1)-N(1)-C(18)	120.80(13)	C(28)-C(27)-N(3)	121.06(14)
C(11)-N(2)-C(7)	119.00(13)	C(29)-C(28)-C(27)	118.24(15)
C(17)-N(3)-C(27)	124.37(12)	C(29)-C(28)-C(33)	119.80(15)
C(2)-C(1)-N(1)	120.47(15)	C(27)-C(28)-C(33)	121.89(15)
C(2)-C(1)-C(6)	119.30(15)	C(30)-C(29)-C(28)	122.51(16)
		C(29)-C(30)-C(31)	117.83(15)

C(29)-C(30)-C(34)	120.51(1616)
C(31)-C(30)-C(34)	121.59(16)
C(30)-C(31)-C(32)	121.95(16)
C(27)-C(32)-C(31)	118.67(15)
C(27)-C(32)-C(35)	119.94(14)
C(31)-C(32)-C(35)	121.39(15)

Table 5. Hydrogen bonds for ECW01 (CCDC 653538) [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(3)-H(3)...N(2)	0.88	2.06	2.7235(18)	130.9

Symmetry transformations used to generate equivalent atoms:

CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE

X-RAY CRYSTALLOGRAPHY LABORATORY

Date 15 February 2008

Crystal Structure Analysis of:

[^{Mes}NNN]Fe(THF) (ECW03)

(shown below)

For Investigator: Edward Weintrob ext. 6576

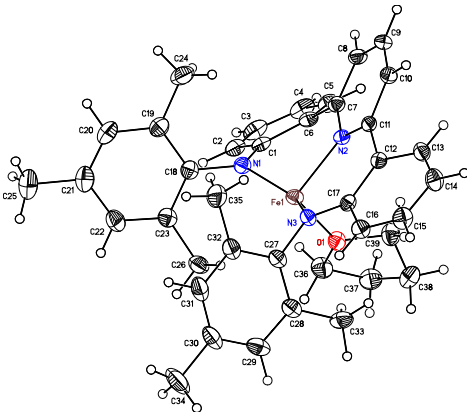
Advisor: J. E. Bercaw ext. 6577

Account Number: JEB.ENERGY-1.02-GRANT.MOOREJCP

By Michael W. Day 116 Beckman ext. 2734

e-mail: mikeday@caltech.edu

Contents



ECW03

Note: The crystallographic data have been deposited in the Cambridge Database (CCDC) and has been placed on hold pending further instructions from me. The deposition number is 678268. Ideally the CCDC would like the publication to contain a footnote of the type: "Crystallographic data have been deposited at the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK and copies can be obtained on request, free of charge, by quoting the publication citation and the deposition number 678268."

Table 1. Crystal data and structure refinement for ECW03 (CCDC 678268).

Empirical formula	$C_{39}H_{41}N_3OFe$
Formula weight	623.60
Crystallization Solvent	Toluene/petroleumether
Crystal Habit	Block
Crystal size	0.26 x 0.25 x 0.21 mm^3
Crystal color	Dark red

Data Collection

Type of diffractometer	Bruker KAPPA APEX II
Wavelength	0.71073 Å MoKα
Data Collection Temperature	100(2) K
θ range for 9851 reflections used in lattice determination	2.48 to 35.69°
Unit cell dimensions	a = 14.5396(6) Å b = 13.5644(6) Å β= 98.247(2)° c = 16.6014(7) Å
Volume	3240.3(2) $Å^3$
Z	4
Crystal system	Monoclinic
Space group	$P2_1/c$
Density (calculated)	1.278 Mg/m^3
F(000)	1320
Data collection program	Bruker APEX2 v2.1-0
θ range for data collection	1.95 to 36.53°
Completeness to θ = 36.53°	93.3 %
Index ranges	$-24 \leq h \leq 22, -22 \leq k \leq 22, -26 \leq l \leq 27$
Data collection scan type	ω scans; 17 settings
Data reduction program	Bruker SAINT-Plus v7.34A
Reflections collected	97719
Independent reflections	14879 [R_{int}= 0.0486]
Absorption coefficient	0.501 mm^{-1}
Absorption correction	None

Table 1 (cont.)

Structure solution and Refinement

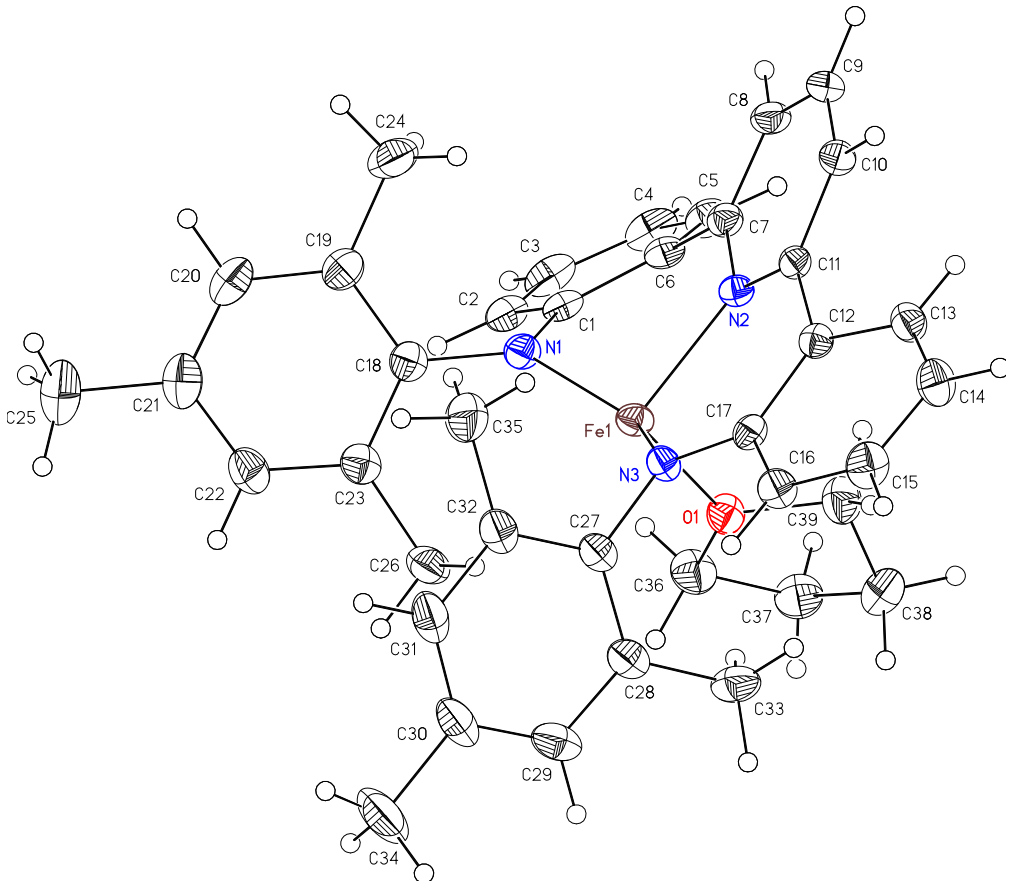
Structure solution program	SHELXS-97 (Sheldrick, 2008)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	SHELXL-97 (Sheldrick, 2008)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	14879 / 0 / 403
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	2.823
Final R indices [$I>2\sigma(I)$, 10317 reflections]	R1 = 0.0501, *w*R2 = 0.0848
R indices (all data)	R1 = 0.0792, *w*R2 = 0.0860
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.003
Average shift/error	0.000
Largest diff. peak and hole	2.279 and -1.067 e.Å^{-3}

Special Refinement Details

Crystals were mounted on a glass fiber using Paratone oil then placed on the diffractometer under a nitrogen stream at 100K.

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

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.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C37
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Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for ECW03 (CCDC 678268). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Fe(1)	2281(1)	2168(1)	8517(1)	19(1)
O(1)	2086(1)	2910(1)	7387(1)	24(1)
N(1)	1773(1)	860(1)	8316(1)	21(1)
N(2)	1160(1)	2750(1)	8954(1)	17(1)
N(3)	3188(1)	2847(1)	9282(1)	19(1)
C(1)	898(1)	889(1)	7846(1)	21(1)
C(2)	640(1)	204(1)	7222(1)	25(1)
C(3)	-198(1)	270(1)	6716(1)	30(1)
C(4)	-809(1)	1027(1)	6800(1)	30(1)
C(5)	-590(1)	1696(1)	7418(1)	27(1)
C(6)	244(1)	1635(1)	7966(1)	21(1)
C(7)	329(1)	2301(1)	8682(1)	20(1)
C(8)	-444(1)	2470(1)	9065(1)	24(1)
C(9)	-365(1)	3075(1)	9738(1)	26(1)
C(10)	466(1)	3530(1)	10008(1)	23(1)
C(11)	1234(1)	3388(1)	9596(1)	18(1)
C(12)	2101(1)	3946(1)	9852(1)	19(1)
C(13)	2009(1)	4838(1)	10263(1)	24(1)
C(14)	2748(1)	5410(1)	10593(1)	29(1)
C(15)	3637(1)	5083(1)	10530(1)	29(1)
C(16)	3769(1)	4231(1)	10121(1)	24(1)
C(17)	3023(1)	3652(1)	9734(1)	19(1)
C(18)	2226(1)	-67(1)	8422(1)	23(1)
C(19)	1844(1)	-829(1)	8840(1)	28(1)
C(20)	2299(1)	-1734(1)	8933(1)	34(1)
C(21)	3133(1)	-1905(1)	8648(1)	36(1)
C(22)	3521(1)	-1128(1)	8274(1)	31(1)
C(23)	3089(1)	-214(1)	8148(1)	25(1)
C(24)	955(1)	-666(1)	9191(1)	37(1)
C(25)	3616(1)	-2900(1)	8753(1)	49(1)
C(26)	3530(1)	588(1)	7708(1)	30(1)
C(27)	4129(1)	2569(1)	9221(1)	22(1)
C(28)	4607(1)	3015(1)	8642(1)	25(1)
C(29)	5484(1)	2647(1)	8549(1)	31(1)
C(30)	5884(1)	1864(1)	9001(1)	35(1)
C(31)	5406(1)	1449(1)	9574(1)	35(1)
C(32)	4530(1)	1782(1)	9694(1)	28(1)
C(33)	4194(1)	3870(1)	8130(1)	32(1)
C(34)	6839(1)	1497(1)	8872(1)	57(1)
C(35)	4025(1)	1312(1)	10322(1)	38(1)
C(36)	2090(1)	2548(1)	6570(1)	32(1)
C(37)	1640(1)	3365(1)	6009(1)	33(1)
C(38)	1787(1)	4274(1)	6540(1)	34(1)
C(39)	1653(1)	3871(1)	7358(1)	31(1)

Table 3. Selected bond lengths [Å] and angles [°] for ECW03 (CCDC 678268).

Fe(1)-N(3)	1.9278(10)	N(3)-Fe(1)-N(1)	139.52(4)
Fe(1)-N(1)	1.9319(10)	N(3)-Fe(1)-N(2)	94.97(4)
Fe(1)-N(2)	2.0356(10)	N(1)-Fe(1)-N(2)	96.44(4)
Fe(1)-O(1)	2.1126(9)	N(3)-Fe(1)-O(1)	110.45(4)
		N(1)-Fe(1)-O(1)	106.54(4)
		N(2)-Fe(1)-O(1)	96.85(4)

Table 4. Bond lengths [Å] and angles [°] for ECW03 (CCDC 678268).

Fe(1)-N(3)	1.9278(10)		
Fe(1)-N(1)	1.9319(10)	N(3)-Fe(1)-N(1)	139.52(4)
Fe(1)-N(2)	2.0356(10)	N(3)-Fe(1)-N(2)	94.97(4)
Fe(1)-O(1)	2.1126(9)	N(1)-Fe(1)-N(2)	96.44(4)
O(1)-C(36)	1.4435(15)	N(3)-Fe(1)-O(1)	110.45(4)
O(1)-C(39)	1.4453(15)	N(1)-Fe(1)-O(1)	106.54(4)
N(1)-C(1)	1.3950(15)	N(2)-Fe(1)-O(1)	96.85(4)
N(1)-C(18)	1.4184(16)	C(36)-O(1)-C(39)	109.65(10)
N(2)-C(11)	1.3654(15)	C(36)-O(1)-Fe(1)	130.90(8)
N(2)-C(7)	1.3706(14)	C(39)-O(1)-Fe(1)	117.46(7)
N(3)-C(17)	1.3656(15)	C(1)-N(1)-C(18)	117.65(10)
N(3)-C(27)	1.4373(15)	C(1)-N(1)-Fe(1)	111.48(8)
C(1)-C(2)	1.4027(17)	C(18)-N(1)-Fe(1)	129.32(8)
C(1)-C(6)	1.4218(17)	C(11)-N(2)-C(7)	120.38(10)
C(2)-C(3)	1.3797(18)	C(11)-N(2)-Fe(1)	123.15(8)
C(3)-C(4)	1.378(2)	C(7)-N(2)-Fe(1)	115.05(8)
C(4)-C(5)	1.3723(19)	C(17)-N(3)-C(27)	119.46(10)
C(5)-C(6)	1.4106(17)	C(17)-N(3)-Fe(1)	125.95(8)
C(6)-C(7)	1.4838(17)	C(27)-N(3)-Fe(1)	113.35(8)
C(7)-C(8)	1.3868(17)	N(1)-C(1)-C(2)	121.35(12)
C(8)-C(9)	1.3786(18)	N(1)-C(1)-C(6)	121.06(11)
C(9)-C(10)	1.3735(18)	C(2)-C(1)-C(6)	117.57(11)
C(10)-C(11)	1.4028(16)	C(3)-C(2)-C(1)	121.66(13)
C(11)-C(12)	1.4788(16)	C(4)-C(3)-C(2)	120.85(13)
C(12)-C(13)	1.4036(17)	C(5)-C(4)-C(3)	119.03(13)
C(12)-C(17)	1.4398(16)	C(4)-C(5)-C(6)	121.93(13)
C(13)-C(14)	1.3738(18)	C(5)-C(6)-C(1)	118.76(12)
C(14)-C(15)	1.3849(18)	C(5)-C(6)-C(7)	116.50(12)
C(15)-C(16)	1.3671(18)	C(1)-C(6)-C(7)	124.43(11)
C(16)-C(17)	1.4151(16)	N(2)-C(7)-C(8)	120.67(11)
C(18)-C(19)	1.4030(18)	N(2)-C(7)-C(6)	120.17(10)
C(18)-C(23)	1.4103(17)	C(8)-C(7)-C(6)	119.16(11)
C(19)-C(20)	1.3926(19)	C(9)-C(8)-C(7)	119.39(12)
C(19)-C(24)	1.5084(19)	C(10)-C(9)-C(8)	119.92(12)
C(20)-C(21)	1.382(2)	C(9)-C(10)-C(11)	120.29(12)
C(21)-C(22)	1.384(2)	N(2)-C(11)-C(10)	119.20(11)
C(21)-C(25)	1.520(2)	N(2)-C(11)-C(12)	121.14(10)
C(22)-C(23)	1.3905(19)	C(10)-C(11)-C(12)	119.64(11)
C(23)-C(26)	1.5049(18)	C(13)-C(12)-C(17)	117.37(11)
C(27)-C(32)	1.4007(18)	C(13)-C(12)-C(11)	116.38(11)
C(27)-C(28)	1.4020(18)	C(17)-C(12)-C(11)	126.24(11)
C(28)-C(29)	1.3982(18)	C(14)-C(13)-C(12)	123.88(12)
C(28)-C(33)	1.5114(19)	C(13)-C(14)-C(15)	118.24(12)
C(29)-C(30)	1.380(2)	C(16)-C(15)-C(14)	120.50(12)
C(30)-C(31)	1.376(2)	C(15)-C(16)-C(17)	122.76(12)
C(30)-C(34)	1.5190(19)	N(3)-C(17)-C(16)	120.67(11)
C(31)-C(32)	1.3919(18)	N(3)-C(17)-C(12)	122.43(11)
C(32)-C(35)	1.502(2)	C(16)-C(17)-C(12)	116.89(11)
C(36)-C(37)	1.5311(19)	C(19)-C(18)-C(23)	119.24(12)
C(37)-C(38)	1.513(2)	C(19)-C(18)-N(1)	120.38(11)
C(38)-C(39)	1.5034(19)	C(23)-C(18)-N(1)	120.30(12)

C(20)-C(19)-C(18)	119.27(13)
C(20)-C(19)-C(24)	120.45(13)
C(18)-C(19)-C(24)	120.28(12)
C(21)-C(20)-C(19)	122.43(14)
C(20)-C(21)-C(22)	117.34(14)
C(20)-C(21)-C(25)	121.52(15)
C(22)-C(21)-C(25)	121.13(14)
C(21)-C(22)-C(23)	122.83(13)
C(22)-C(23)-C(18)	118.77(13)
C(22)-C(23)-C(26)	120.19(12)
C(18)-C(23)-C(26)	121.02(12)
C(32)-C(27)-C(28)	120.32(11)
C(32)-C(27)-N(3)	119.01(11)
C(28)-C(27)-N(3)	120.39(12)
C(29)-C(28)-C(27)	118.17(13)
C(29)-C(28)-C(33)	120.43(13)
C(27)-C(28)-C(33)	121.40(11)
C(30)-C(29)-C(28)	122.26(14)
C(31)-C(30)-C(29)	118.40(13)
C(31)-C(30)-C(34)	121.75(15)
C(29)-C(30)-C(34)	119.83(15)
C(30)-C(31)-C(32)	121.93(14)
C(31)-C(32)-C(27)	118.90(13)
C(31)-C(32)-C(35)	120.65(13)
C(27)-C(32)-C(35)	120.45(12)
O(1)-C(36)-C(37)	105.45(11)
C(38)-C(37)-C(36)	102.94(11)
C(39)-C(38)-C(37)	101.77(11)
O(1)-C(39)-C(38)	104.19(11)

CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE

X-RAY CRYSTALLOGRAPHY LABORATORY

Date 18 July 2008

Crystal Structure Analysis of:

[^{Mes}NNN]FeI (ECW04)

(shown below)

For Investigator: Edward Weintrob ext. 6576

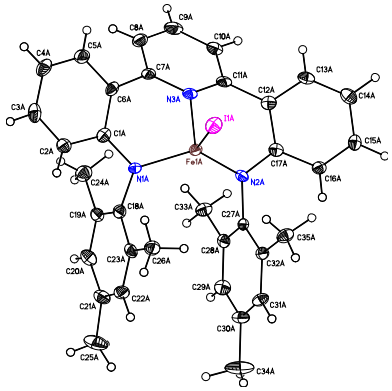
Advisor: J. E. Bercaw ext. 6577

Account Number: JEB.ENERGY-1.02-GRANT.MOOREJCP

By Michael W. Day 116 Beckman ext. 2734

e-mail: mikeday@caltech.edu

Contents



ECW04

Note: The crystallographic data have been deposited in the Cambridge Database (CCDC) and has been placed on hold pending further instructions from me. The deposition number is 695390. Ideally the CCDC would like the publication to contain a footnote of the type: "Crystallographic data have been deposited at the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK and copies can be obtained on request, free of charge, by quoting the publication citation and the deposition number 695390."

Table 1. Crystal data and structure refinement for ECW04 (CCDC 695390).

Empirical formula	$2(C_{35}H_{33}N_3FeI)\ 1.5(C_6H_6)$
Formula weight	736.97
Crystallization Solvent	Benzene/petroleumether
Crystal Habit	Flake
Crystal size	0.16 x 0.10 x 0.06 mm^3
Crystal color	Dark brown

Data Collection

Type of diffractometer	Bruker KAPPA APEX II	
Wavelength	0.71073 Å MoKα	
Data Collection Temperature	100(2) K	
θ range for 9935 reflections used in lattice determination	2.54 to 31.80°	
Unit cell dimensions	a = 14.8740(7) Å b = 14.9489(7) Å c = 16.9750(8) Å	α= 69.528(3)° β= 72.061(3)° γ = 76.207(3)°
Volume	3327.7(3) $Å^3$	
Z	4	
Crystal system	Triclinic	
Space group	P-1	
Density (calculated)	1.471 Mg/m^3	
F(000)	1498	
Data collection program	Bruker APEX2 v2.1-0	
θ range for data collection	1.66 to 32.13°	
Completeness to θ = 32.13°	94.8 %	
Index ranges	$-21 \le h \le 20, -22 \le k \le 21, -25 \le l \le 24$	
Data collection scan type	ω scans; 17 settings	
Data reduction program	Bruker SAINT-Plus v7.34A	
Reflections collected	85468	
Independent reflections	22141 [R_{int}= 0.0607]	
Absorption coefficient	1.413 mm^{-1}	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.4344 and 0.3722	

Table 1 (cont.)

Structure solution and Refinement

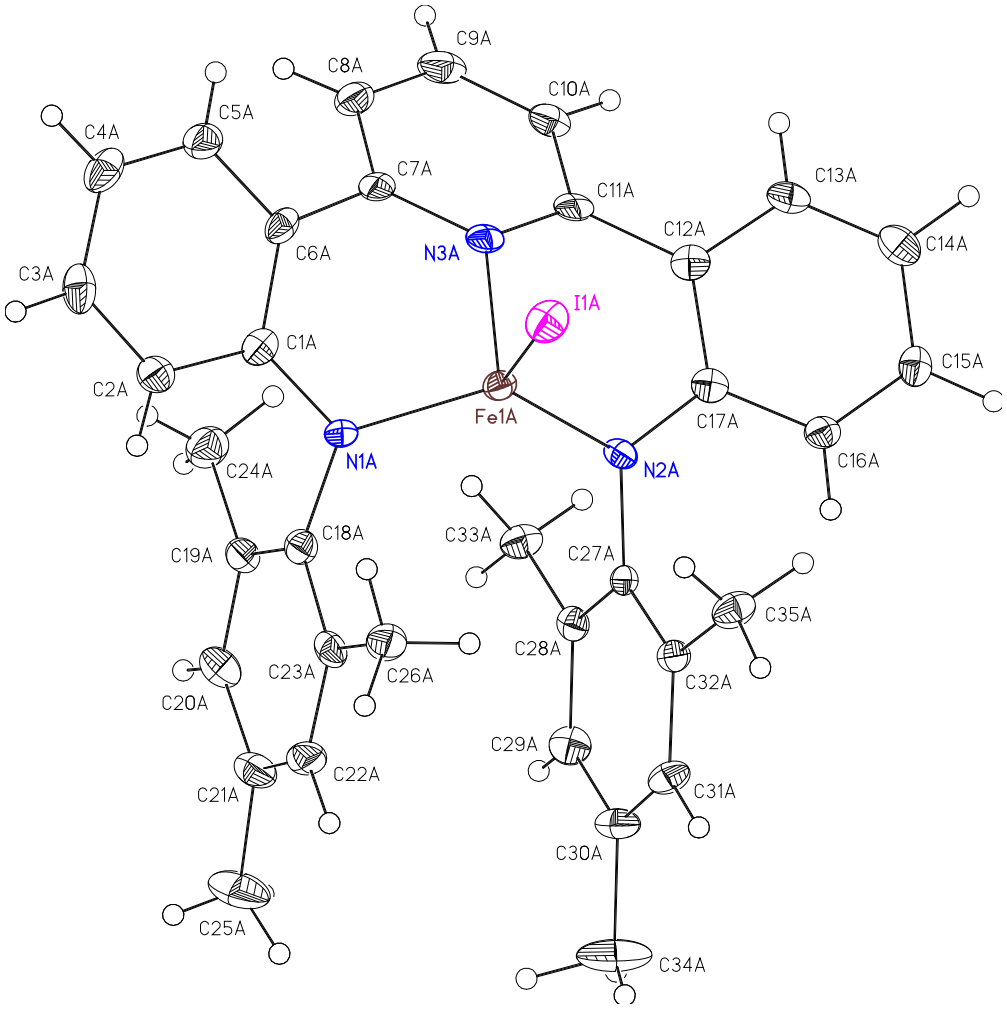
Structure solution program	SHELXS-97 (Sheldrick, 2008)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Difference Fourier map
Structure refinement program	SHELXL-97 (Sheldrick, 2008)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	22141 / 0 / 1102
Treatment of hydrogen atoms	Unrestrained
Goodness-of-fit on F^2	1.524
Final R indices [$I>2\sigma(I)$, 13476 reflections]	R1 = 0.0507, *w*R2 = 0.0602
R indices (all data)	R1 = 0.1132, *w*R2 = 0.0647
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.003
Average shift/error	0.000
Largest diff. peak and hole	1.916 and -1.795 e.Å^{-3}

Special Refinement Details

Crystals were mounted on a glass fiber using Paratone oil then placed on the diffractometer under a nitrogen stream at 100K.

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

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.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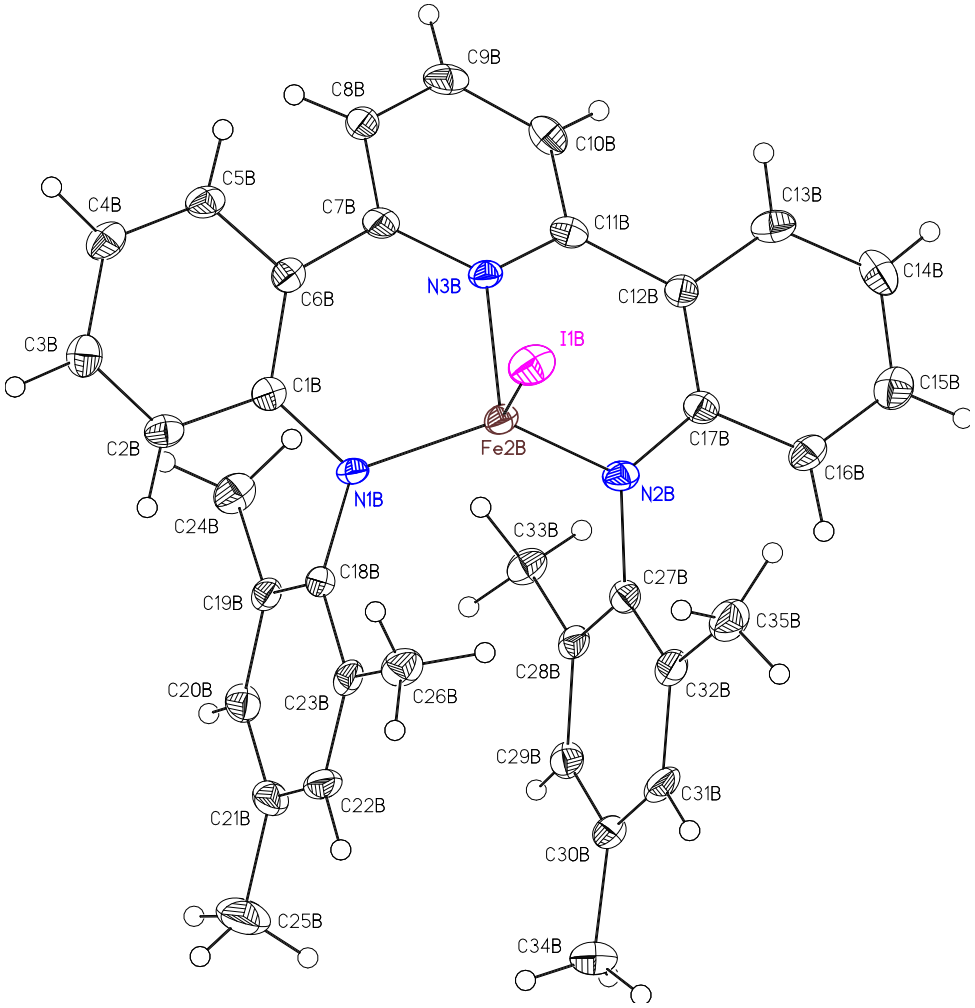
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C28B
C32B
C20B
C23B
C26B
C29B
C31B
C22B
C21B
C30B
C25B
C34B

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for ECW04 (CCDC 695390). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Fe(1A)	7733(1)	6797(1)	9594(1)	14(1)
I(1A)	9034(1)	6803(1)	10304(1)	22(1)
N(1A)	7164(2)	8029(2)	9000(1)	15(1)
N(2A)	6991(2)	5785(2)	10103(1)	14(1)
N(3A)	8373(2)	6424(2)	8490(1)	15(1)
C(1A)	7828(2)	8566(2)	8359(2)	16(1)
C(2A)	7785(2)	9546(2)	8240(2)	18(1)
C(3A)	8485(2)	10070(2)	7659(2)	23(1)
C(4A)	9263(2)	9626(2)	7158(2)	24(1)
C(5A)	9303(2)	8677(2)	7232(2)	22(1)
C(6A)	8601(2)	8124(2)	7809(2)	17(1)
C(7A)	8641(2)	7147(2)	7758(2)	17(1)
C(8A)	8926(2)	6978(2)	6952(2)	21(1)
C(9A)	8919(2)	6077(2)	6911(2)	24(1)
C(10A)	8649(2)	5360(2)	7654(2)	20(1)
C(11A)	8382(2)	5522(2)	8472(2)	16(1)
C(12A)	8097(2)	4739(2)	9261(2)	16(1)
C(13A)	8492(2)	3790(2)	9268(2)	19(1)
C(14A)	8181(2)	2995(2)	9922(2)	21(1)
C(15A)	7410(2)	3130(2)	10613(2)	17(1)
C(16A)	7027(2)	4047(2)	10647(2)	16(1)
C(17A)	7357(2)	4873(2)	10015(2)	14(1)
C(18A)	6176(2)	8444(2)	9186(2)	16(1)
C(19A)	5661(2)	8661(2)	8566(2)	18(1)
C(20A)	4674(2)	8922(2)	8799(2)	21(1)
C(21A)	4192(2)	8966(2)	9634(2)	22(1)
C(22A)	4737(2)	8802(2)	10216(2)	20(1)
C(23A)	5719(2)	8542(2)	10017(2)	17(1)
C(24A)	6135(3)	8585(3)	7663(2)	25(1)
C(25A)	3117(2)	9170(4)	9891(3)	35(1)
C(26A)	6268(2)	8374(3)	10677(2)	22(1)
C(27A)	6023(2)	5936(2)	10621(2)	13(1)
C(28A)	5287(2)	6171(2)	10211(2)	15(1)
C(29A)	4355(2)	6369(2)	10683(2)	19(1)
C(30A)	4150(2)	6325(2)	11550(2)	20(1)
C(31A)	4900(2)	6079(2)	11942(2)	18(1)
C(32A)	5846(2)	5879(2)	11494(2)	14(1)
C(33A)	5483(2)	6220(3)	9275(2)	20(1)
C(34A)	3127(2)	6531(4)	12043(3)	38(1)
C(35A)	6631(2)	5585(3)	11954(2)	21(1)
Fe(2B)	2563(1)	7908(1)	5601(1)	15(1)
I(1B)	1359(1)	9349(1)	5034(1)	25(1)
N(1B)	3345(2)	8156(2)	6179(1)	15(1)
N(2B)	3089(2)	6942(2)	5040(1)	16(1)
N(3B)	1832(2)	7032(2)	6682(1)	15(1)
C(1B)	2814(2)	8512(2)	6866(2)	15(1)

C(2B)	3128(2)	9216(2)	7047(2)	19(1)
C(3B)	2569(2)	9650(2)	7667(2)	21(1)
C(4B)	1679(2)	9394(2)	8131(2)	21(1)
C(5B)	1375(2)	8680(2)	7996(2)	19(1)
C(6B)	1930(2)	8195(2)	7384(2)	16(1)
C(7B)	1617(2)	7311(2)	7417(2)	17(1)
C(8B)	1126(2)	6738(2)	8196(2)	19(1)
C(9B)	895(2)	5884(2)	8233(2)	21(1)
C(10B)	1162(2)	5596(2)	7496(2)	20(1)
C(11B)	1613(2)	6185(2)	6707(2)	16(1)
C(12B)	1870(2)	5893(2)	5899(2)	17(1)
C(13B)	1373(2)	5194(2)	5910(2)	22(1)
C(14B)	1617(2)	4758(2)	5258(2)	25(1)
C(15B)	2389(2)	5023(2)	4563(2)	23(1)
C(16B)	2868(2)	5723(2)	4517(2)	19(1)
C(17B)	2615(2)	6199(2)	5156(2)	17(1)
C(18B)	4357(2)	8138(2)	5915(2)	15(1)
C(19B)	4925(2)	7499(2)	6459(2)	15(1)
C(20B)	5915(2)	7440(2)	6167(2)	20(1)
C(21B)	6348(2)	7993(2)	5361(2)	21(1)
C(22B)	5776(2)	8625(2)	4835(2)	21(1)
C(23B)	4783(2)	8722(2)	5090(2)	17(1)
C(24B)	4486(2)	6884(3)	7340(2)	23(1)
C(25B)	7428(2)	7925(3)	5055(3)	36(1)
C(26B)	4198(2)	9436(2)	4505(2)	23(1)
C(27B)	4033(2)	7003(2)	4476(2)	15(1)
C(28B)	4814(2)	6468(2)	4821(2)	16(1)
C(29B)	5733(2)	6607(2)	4304(2)	20(1)
C(30B)	5894(2)	7239(2)	3474(2)	20(1)
C(31B)	5110(2)	7738(2)	3144(2)	21(1)
C(32B)	4177(2)	7629(2)	3629(2)	19(1)
C(33B)	4662(2)	5779(2)	5719(2)	21(1)
C(34B)	6900(3)	7403(3)	2965(2)	31(1)
C(35B)	3349(2)	8191(3)	3241(2)	26(1)
C(41)	9959(2)	8043(2)	3955(2)	31(1)
C(42)	10483(3)	7138(3)	4013(2)	31(1)
C(43)	10445(2)	6636(3)	3490(2)	37(1)
C(44)	9886(2)	7032(3)	2909(2)	36(1)
C(45)	9370(2)	7937(3)	2841(2)	30(1)
C(46)	9411(2)	8439(2)	3367(2)	29(1)
C(51)	588(2)	421(2)	206(2)	26(1)
C(52)	117(2)	952(2)	-423(2)	25(1)
C(53)	488(2)	-534(3)	629(2)	26(1)

Table 3. Selected bond lengths [Å] and angles [°] for ECW04 (CCDC 695390).

Fe(1A)-N(2A)	1.883(2)	N(2A)-Fe(1A)-N(1A)	118.30(10)
Fe(1A)-N(1A)	1.899(2)	N(2A)-Fe(1A)-N(3A)	94.66(9)
Fe(1A)-N(3A)	2.0274(19)	N(1A)-Fe(1A)-N(3A)	93.44(9)
Fe(1A)-I(1A)	2.5784(4)	N(2A)-Fe(1A)-I(1A)	118.77(7)
Fe(2B)-N(2B)	1.888(2)	N(1A)-Fe(1A)-I(1A)	115.77(7)
Fe(2B)-N(1B)	1.902(2)	N(3A)-Fe(1A)-I(1A)	108.74(6)
Fe(2B)-N(3B)	2.008(2)	N(2B)-Fe(2B)-N(1B)	116.72(9)
Fe(2B)-I(1B)	2.5576(5)	N(2B)-Fe(2B)-N(3B)	94.66(9)
		N(1B)-Fe(2B)-N(3B)	94.36(9)
		N(2B)-Fe(2B)-I(1B)	121.38(7)
		N(1B)-Fe(2B)-I(1B)	115.03(7)
		N(3B)-Fe(2B)-I(1B)	107.04(6)

Table 4. Bond lengths [Å] and angles [°] for ECW04 (CCDC 695390).

Fe(1A)-N(2A)	1.883(2)	C(24A)-H(24B)	0.94(3)
Fe(1A)-N(1A)	1.899(2)	C(24A)-H(24C)	0.86(3)
Fe(1A)-N(3A)	2.0274(19)	C(25A)-H(25A)	0.88(3)
Fe(1A)-I(1A)	2.5784(4)	C(25A)-H(25B)	0.80(4)
N(1A)-C(1A)	1.381(3)	C(25A)-H(25C)	0.97(5)
N(1A)-C(18A)	1.435(3)	C(26A)-H(26A)	0.96(2)
N(2A)-C(17A)	1.385(3)	C(26A)-H(26B)	0.95(3)
N(2A)-C(27A)	1.449(3)	C(26A)-H(26C)	0.94(3)
N(3A)-C(7A)	1.354(3)	C(27A)-C(28A)	1.386(4)
N(3A)-C(11A)	1.356(3)	C(27A)-C(32A)	1.398(3)
C(1A)-C(2A)	1.396(4)	C(28A)-C(29A)	1.392(4)
C(1A)-C(6A)	1.426(3)	C(28A)-C(33A)	1.502(4)
C(2A)-C(3A)	1.373(4)	C(29A)-C(30A)	1.388(4)
C(2A)-H(2A)	0.85(2)	C(29A)-H(29A)	0.87(3)
C(3A)-C(4A)	1.388(4)	C(30A)-C(31A)	1.384(4)
C(3A)-H(3A)	0.89(2)	C(30A)-C(34A)	1.511(4)
C(4A)-C(5A)	1.369(4)	C(31A)-C(32A)	1.395(3)
C(4A)-H(4A)	0.91(3)	C(31A)-H(31A)	0.91(2)
C(5A)-C(6A)	1.396(4)	C(32A)-C(35A)	1.499(4)
C(5A)-H(5A)	0.82(3)	C(33A)-H(33A)	0.93(3)
C(6A)-C(7A)	1.479(4)	C(33A)-H(33B)	0.92(4)
C(7A)-C(8A)	1.398(3)	C(33A)-H(33C)	0.92(3)
C(8A)-C(9A)	1.374(4)	C(34A)-H(34A)	0.76(3)
C(8A)-H(8A)	0.87(2)	C(34A)-H(34B)	0.79(3)
C(9A)-C(10A)	1.359(4)	C(34A)-H(34C)	1.04(6)
C(9A)-H(9A)	0.90(2)	C(35A)-H(35A)	0.93(3)
C(10A)-C(11A)	1.412(3)	C(35A)-H(35B)	0.86(3)
C(10A)-H(10A)	0.90(2)	C(35A)-H(35C)	0.97(3)
C(11A)-C(12A)	1.462(4)	Fe(2B)-N(2B)	1.888(2)
C(12A)-C(13A)	1.396(4)	Fe(2B)-N(1B)	1.902(2)
C(12A)-C(17A)	1.448(3)	Fe(2B)-N(3B)	2.008(2)
C(13A)-C(14A)	1.372(4)	Fe(2B)-I(1B)	2.5576(5)
C(13A)-H(13A)	0.86(2)	N(1B)-C(1B)	1.391(3)
C(14A)-C(15A)	1.399(4)	N(1B)-C(18B)	1.430(3)
C(14A)-H(14A)	1.00(3)	N(2B)-C(17B)	1.381(3)
C(15A)-C(16A)	1.368(4)	N(2B)-C(27B)	1.438(3)
C(15A)-H(15A)	0.97(2)	N(3B)-C(11B)	1.364(3)
C(16A)-C(17A)	1.399(4)	N(3B)-C(7B)	1.374(3)
C(16A)-H(16A)	0.90(2)	C(1B)-C(2B)	1.403(4)
C(18A)-C(19A)	1.393(4)	C(1B)-C(6B)	1.422(4)
C(18A)-C(23A)	1.406(3)	C(2B)-C(3B)	1.379(3)
C(19A)-C(20A)	1.394(4)	C(2B)-H(2B)	0.93(2)
C(19A)-C(24A)	1.508(4)	C(3B)-C(4B)	1.381(4)
C(20A)-C(21A)	1.395(4)	C(3B)-H(3B)	0.95(2)
C(20A)-H(20A)	0.96(2)	C(4B)-C(5B)	1.364(4)
C(21A)-C(22A)	1.386(4)	C(4B)-H(4B)	0.96(2)
C(21A)-C(25A)	1.508(4)	C(5B)-C(6B)	1.414(3)
C(22A)-C(23A)	1.384(4)	C(5B)-H(5B)	0.88(3)
C(22A)-H(22A)	0.92(2)	C(6B)-C(7B)	1.480(4)
C(23A)-C(26A)	1.502(4)	C(7B)-C(8B)	1.388(4)
C(24A)-H(24A)	1.01(3)	C(8B)-C(9B)	1.375(4)

C(8B)-H(8B)	0.87(2)	C(35B)-H(35E)	0.98(3)
C(9B)-C(10B)	1.378(4)	C(35B)-H(35F)	0.94(3)
C(9B)-H(9B)	0.98(3)	C(41)-C(46)	1.367(4)
C(10B)-C(11B)	1.386(4)	C(41)-C(42)	1.378(4)
C(10B)-H(10B)	0.88(2)	C(41)-H(41)	0.86(2)
C(11B)-C(12B)	1.495(3)	C(42)-C(43)	1.368(4)
C(12B)-C(13B)	1.410(4)	C(42)-H(42)	0.88(3)
C(12B)-C(17B)	1.417(4)	C(43)-C(44)	1.371(5)
C(13B)-C(14B)	1.387(4)	C(43)-H(43)	1.00(3)
C(13B)-H(13B)	0.89(3)	C(44)-C(45)	1.372(5)
C(14B)-C(15B)	1.386(4)	C(44)-H(44)	0.93(3)
C(14B)-H(14B)	0.84(2)	C(45)-C(46)	1.373(4)
C(15B)-C(16B)	1.368(4)	C(45)-H(45)	0.97(3)
C(15B)-H(15B)	0.92(2)	C(46)-H(46)	0.90(2)
C(16B)-C(17B)	1.408(3)	C(51)-C(52)	1.364(4)
C(16B)-H(16B)	0.97(2)	C(51)-C(53)	1.377(4)
C(18B)-C(19B)	1.399(4)	C(51)-H(51)	0.93(3)
C(18B)-C(23B)	1.409(4)	C(52)-C(53)#1	1.392(4)
C(19B)-C(20B)	1.393(4)	C(52)-H(52)	0.91(2)
C(19B)-C(24B)	1.494(4)	C(53)-C(52)#1	1.392(4)
C(20B)-C(21B)	1.375(4)	C(53)-H(53)	0.87(3)
C(20B)-H(20B)	0.96(3)		
C(21B)-C(22B)	1.381(4)	N(2A)-Fe(1A)-N(1A)	118.30(10)
C(21B)-C(25B)	1.519(4)	N(2A)-Fe(1A)-N(3A)	94.66(9)
C(22B)-C(23B)	1.393(4)	N(1A)-Fe(1A)-N(3A)	93.44(9)
C(22B)-H(22B)	0.95(3)	N(2A)-Fe(1A)-I(1A)	118.77(7)
C(23B)-C(26B)	1.493(4)	N(1A)-Fe(1A)-I(1A)	115.77(7)
C(24B)-H(24D)	0.94(2)	N(3A)-Fe(1A)-I(1A)	108.74(6)
C(24B)-H(24E)	0.94(3)	C(1A)-N(1A)-C(18A)	120.0(2)
C(24B)-H(24F)	0.93(3)	C(1A)-N(1A)-Fe(1A)	112.52(18)
C(25B)-H(25D)	0.89(4)	C(18A)-N(1A)-Fe(1A)	127.38(18)
C(25B)-H(25E)	0.95(3)	C(17A)-N(2A)-C(27A)	118.0(2)
C(25B)-H(25F)	0.91(5)	C(17A)-N(2A)-Fe(1A)	121.37(17)
C(26B)-H(26D)	0.93(4)	C(27A)-N(2A)-Fe(1A)	120.59(16)
C(26B)-H(26E)	0.89(3)	C(7A)-N(3A)-C(11A)	122.0(2)
C(26B)-H(26F)	0.99(3)	C(7A)-N(3A)-Fe(1A)	116.75(17)
C(27B)-C(32B)	1.398(4)	C(11A)-N(3A)-Fe(1A)	120.33(18)
C(27B)-C(28B)	1.406(4)	N(1A)-C(1A)-C(2A)	121.6(2)
C(28B)-C(29B)	1.397(4)	N(1A)-C(1A)-C(6A)	120.7(2)
C(28B)-C(33B)	1.495(4)	C(2A)-C(1A)-C(6A)	117.7(3)
C(29B)-C(30B)	1.379(4)	C(3A)-C(2A)-C(1A)	122.4(3)
C(29B)-H(29B)	0.91(3)	C(3A)-C(2A)-H(2A)	117.3(18)
C(30B)-C(31B)	1.388(4)	C(1A)-C(2A)-H(2A)	120.3(18)
C(30B)-C(34B)	1.508(4)	C(2A)-C(3A)-C(4A)	119.8(3)
C(31B)-C(32B)	1.394(4)	C(2A)-C(3A)-H(3A)	118.4(16)
C(31B)-H(31B)	0.91(3)	C(4A)-C(3A)-H(3A)	121.8(16)
C(32B)-C(35B)	1.508(4)	C(5A)-C(4A)-C(3A)	119.1(3)
C(33B)-H(33D)	0.93(2)	C(5A)-C(4A)-H(4A)	123.2(16)
C(33B)-H(33E)	0.96(3)	C(3A)-C(4A)-H(4A)	117.6(16)
C(33B)-H(33F)	1.06(4)	C(4A)-C(5A)-C(6A)	122.6(3)
C(34B)-H(34D)	0.88(4)	C(4A)-C(5A)-H(5A)	121(2)
C(34B)-H(34E)	0.92(4)	C(6A)-C(5A)-H(5A)	117(2)
C(34B)-H(34F)	0.91(3)	C(5A)-C(6A)-C(1A)	118.2(3)
C(35B)-H(35D)	0.87(3)	C(5A)-C(6A)-C(7A)	118.4(2)

C(1A)-C(6A)-C(7A)	123.1(2)	H(24A)-C(24A)-H(24B)	106(2)
N(3A)-C(7A)-C(8A)	119.9(3)	C(19A)-C(24A)-H(24C)	108.4(18)
N(3A)-C(7A)-C(6A)	120.1(2)	H(24A)-C(24A)-H(24C)	107(3)
C(8A)-C(7A)-C(6A)	120.0(3)	H(24B)-C(24A)-H(24C)	113(3)
C(9A)-C(8A)-C(7A)	119.3(3)	C(21A)-C(25A)-H(25A)	115(2)
C(9A)-C(8A)-H(8A)	122.2(14)	C(21A)-C(25A)-H(25B)	115(3)
C(7A)-C(8A)-H(8A)	118.4(14)	H(25A)-C(25A)-H(25B)	107(4)
C(10A)-C(9A)-C(8A)	119.8(3)	C(21A)-C(25A)-H(25C)	114(3)
C(10A)-C(9A)-H(9A)	120.9(16)	H(25A)-C(25A)-H(25C)	105(4)
C(8A)-C(9A)-H(9A)	119.1(16)	H(25B)-C(25A)-H(25C)	99(4)
C(9A)-C(10A)-C(11A)	121.0(3)	C(23A)-C(26A)-H(26A)	114.4(16)
C(9A)-C(10A)-H(10A)	122.4(15)	C(23A)-C(26A)-H(26B)	110(2)
C(11A)-C(10A)-H(10A)	116.6(15)	H(26A)-C(26A)-H(26B)	109(2)
N(3A)-C(11A)-C(10A)	117.9(3)	C(23A)-C(26A)-H(26C)	113.7(19)
N(3A)-C(11A)-C(12A)	122.2(2)	H(26A)-C(26A)-H(26C)	105(2)
C(10A)-C(11A)-C(12A)	119.9(3)	H(26B)-C(26A)-H(26C)	105(3)
C(13A)-C(12A)-C(17A)	117.1(3)	C(28A)-C(27A)-C(32A)	121.5(2)
C(13A)-C(12A)-C(11A)	118.7(2)	C(28A)-C(27A)-N(2A)	117.6(2)
C(17A)-C(12A)-C(11A)	123.9(2)	C(32A)-C(27A)-N(2A)	120.9(2)
C(14A)-C(13A)-C(12A)	123.6(3)	C(27A)-C(28A)-C(29A)	118.6(2)
C(14A)-C(13A)-H(13A)	118.4(19)	C(27A)-C(28A)-C(33A)	121.1(2)
C(12A)-C(13A)-H(13A)	118.0(19)	C(29A)-C(28A)-C(33A)	120.2(3)
C(13A)-C(14A)-C(15A)	118.9(3)	C(30A)-C(29A)-C(28A)	121.6(3)
C(13A)-C(14A)-H(14A)	121.9(17)	C(30A)-C(29A)-H(29A)	121.1(17)
C(15A)-C(14A)-H(14A)	119.2(17)	C(28A)-C(29A)-H(29A)	117.2(17)
C(16A)-C(15A)-C(14A)	119.3(3)	C(31A)-C(30A)-C(29A)	118.3(3)
C(16A)-C(15A)-H(15A)	120.6(13)	C(31A)-C(30A)-C(34A)	121.7(3)
C(14A)-C(15A)-H(15A)	119.9(13)	C(29A)-C(30A)-C(34A)	120.0(3)
C(15A)-C(16A)-C(17A)	123.3(3)	C(30A)-C(31A)-C(32A)	122.1(2)
C(15A)-C(16A)-H(16A)	121.3(17)	C(30A)-C(31A)-H(31A)	119.4(15)
C(17A)-C(16A)-H(16A)	115.3(17)	C(32A)-C(31A)-H(31A)	118.5(15)
N(2A)-C(17A)-C(16A)	121.4(2)	C(31A)-C(32A)-C(27A)	117.8(3)
N(2A)-C(17A)-C(12A)	121.2(2)	C(31A)-C(32A)-C(35A)	120.2(2)
C(16A)-C(17A)-C(12A)	117.4(2)	C(27A)-C(32A)-C(35A)	122.0(2)
C(19A)-C(18A)-C(23A)	120.7(2)	C(28A)-C(33A)-H(33A)	110.7(16)
C(19A)-C(18A)-N(1A)	119.3(2)	C(28A)-C(33A)-H(33B)	116(2)
C(23A)-C(18A)-N(1A)	119.8(2)	H(33A)-C(33A)-H(33B)	106(3)
C(18A)-C(19A)-C(20A)	118.8(2)	C(28A)-C(33A)-H(33C)	108.1(17)
C(18A)-C(19A)-C(24A)	122.0(3)	H(33A)-C(33A)-H(33C)	106(2)
C(20A)-C(19A)-C(24A)	119.2(3)	H(33B)-C(33A)-H(33C)	110(3)
C(19A)-C(20A)-C(21A)	121.7(3)	C(30A)-C(34A)-H(34A)	116(3)
C(19A)-C(20A)-H(20A)	117.1(15)	C(30A)-C(34A)-H(34B)	113(2)
C(21A)-C(20A)-H(20A)	121.2(15)	H(34A)-C(34A)-H(34B)	129(4)
C(22A)-C(21A)-C(20A)	117.7(3)	C(30A)-C(34A)-H(34C)	108(3)
C(22A)-C(21A)-C(25A)	121.7(3)	H(34A)-C(34A)-H(34C)	98(4)
C(20A)-C(21A)-C(25A)	120.7(3)	H(34B)-C(34A)-H(34C)	78(4)
C(23A)-C(22A)-C(21A)	122.7(3)	C(32A)-C(35A)-H(35A)	114.9(19)
C(23A)-C(22A)-H(22A)	115.3(17)	C(32A)-C(35A)-H(35B)	109(2)
C(21A)-C(22A)-H(22A)	121.9(17)	H(35A)-C(35A)-H(35B)	106(2)
C(22A)-C(23A)-C(18A)	118.2(3)	C(32A)-C(35A)-H(35C)	111.9(17)
C(22A)-C(23A)-C(26A)	120.1(2)	H(35A)-C(35A)-H(35C)	105(3)
C(18A)-C(23A)-C(26A)	121.7(3)	H(35B)-C(35A)-H(35C)	109(2)
C(19A)-C(24A)-H(24A)	114.5(16)	N(2B)-Fe(2B)-N(1B)	116.72(9)
C(19A)-C(24A)-H(24B)	108.2(16)	N(2B)-Fe(2B)-N(3B)	94.66(9)

N(1B)-Fe(2B)-N(3B)	94.36(9)	C(13B)-C(14B)-H(14B)	119.8(18)
N(2B)-Fe(2B)-I(1B)	121.38(7)	C(16B)-C(15B)-C(14B)	120.1(3)
N(1B)-Fe(2B)-I(1B)	115.03(7)	C(16B)-C(15B)-H(15B)	122.5(18)
N(3B)-Fe(2B)-I(1B)	107.04(6)	C(14B)-C(15B)-H(15B)	117.4(18)
C(1B)-N(1B)-C(18B)	119.3(2)	C(15B)-C(16B)-C(17B)	122.8(3)
C(1B)-N(1B)-Fe(2B)	112.61(17)	C(15B)-C(16B)-H(16B)	117.8(13)
C(18B)-N(1B)-Fe(2B)	127.48(15)	C(17B)-C(16B)-H(16B)	119.2(13)
C(17B)-N(2B)-C(27B)	120.1(2)	N(2B)-C(17B)-C(16B)	119.7(2)
C(17B)-N(2B)-Fe(2B)	123.15(17)	N(2B)-C(17B)-C(12B)	122.3(2)
C(27B)-N(2B)-Fe(2B)	116.77(17)	C(16B)-C(17B)-C(12B)	118.0(3)
C(11B)-N(3B)-C(7B)	121.5(2)	C(19B)-C(18B)-C(23B)	120.3(2)
C(11B)-N(3B)-Fe(2B)	122.99(16)	C(19B)-C(18B)-N(1B)	119.6(2)
C(7B)-N(3B)-Fe(2B)	115.34(18)	C(23B)-C(18B)-N(1B)	120.0(3)
N(1B)-C(1B)-C(2B)	120.6(2)	C(20B)-C(19B)-C(18B)	118.9(3)
N(1B)-C(1B)-C(6B)	120.9(2)	C(20B)-C(19B)-C(24B)	120.0(3)
C(2B)-C(1B)-C(6B)	118.5(2)	C(18B)-C(19B)-C(24B)	121.1(3)
C(3B)-C(2B)-C(1B)	121.5(3)	C(21B)-C(20B)-C(19B)	121.9(3)
C(3B)-C(2B)-H(2B)	117.2(13)	C(21B)-C(20B)-H(20B)	118.0(17)
C(1B)-C(2B)-H(2B)	121.2(13)	C(19B)-C(20B)-H(20B)	120.1(17)
C(2B)-C(3B)-C(4B)	120.2(3)	C(20B)-C(21B)-C(22B)	118.4(3)
C(2B)-C(3B)-H(3B)	117.4(14)	C(20B)-C(21B)-C(25B)	121.2(3)
C(4B)-C(3B)-H(3B)	122.4(14)	C(22B)-C(21B)-C(25B)	120.4(3)
C(5B)-C(4B)-C(3B)	119.4(3)	C(21B)-C(22B)-C(23B)	122.5(3)
C(5B)-C(4B)-H(4B)	118.7(15)	C(21B)-C(22B)-H(22B)	118.3(18)
C(3B)-C(4B)-H(4B)	121.7(15)	C(23B)-C(22B)-H(22B)	119.2(18)
C(4B)-C(5B)-C(6B)	122.6(3)	C(22B)-C(23B)-C(18B)	117.9(3)
C(4B)-C(5B)-H(5B)	122.4(16)	C(22B)-C(23B)-C(26B)	120.3(3)
C(6B)-C(5B)-H(5B)	115.0(16)	C(18B)-C(23B)-C(26B)	121.7(3)
C(5B)-C(6B)-C(1B)	117.5(2)	C(19B)-C(24B)-H(24D)	111.5(17)
C(5B)-C(6B)-C(7B)	117.4(2)	C(19B)-C(24B)-H(24E)	114.8(18)
C(1B)-C(6B)-C(7B)	124.5(2)	H(24D)-C(24B)-H(24E)	109(2)
N(3B)-C(7B)-C(8B)	119.1(3)	C(19B)-C(24B)-H(24F)	110(2)
N(3B)-C(7B)-C(6B)	120.4(2)	H(24D)-C(24B)-H(24F)	110(2)
C(8B)-C(7B)-C(6B)	120.4(2)	H(24E)-C(24B)-H(24F)	101(3)
C(9B)-C(8B)-C(7B)	120.1(3)	C(21B)-C(25B)-H(25D)	113(2)
C(9B)-C(8B)-H(8B)	122.6(17)	C(21B)-C(25B)-H(25E)	114(2)
C(7B)-C(8B)-H(8B)	117.0(17)	H(25D)-C(25B)-H(25E)	112(3)
C(8B)-C(9B)-C(10B)	119.6(3)	C(21B)-C(25B)-H(25F)	113(3)
C(8B)-C(9B)-H(9B)	119.3(14)	H(25D)-C(25B)-H(25F)	97(4)
C(10B)-C(9B)-H(9B)	120.5(14)	H(25E)-C(25B)-H(25F)	106(3)
C(9B)-C(10B)-C(11B)	120.6(3)	C(23B)-C(26B)-H(26D)	116(2)
C(9B)-C(10B)-H(10B)	119.2(16)	C(23B)-C(26B)-H(26E)	115.2(19)
C(11B)-C(10B)-H(10B)	120.2(15)	H(26D)-C(26B)-H(26E)	103(3)
N(3B)-C(11B)-C(10B)	118.9(2)	C(23B)-C(26B)-H(26F)	115.6(17)
N(3B)-C(11B)-C(12B)	120.4(2)	H(26D)-C(26B)-H(26F)	103(2)
C(10B)-C(11B)-C(12B)	120.8(3)	H(26E)-C(26B)-H(26F)	103(2)
C(13B)-C(12B)-C(17B)	117.4(2)	C(32B)-C(27B)-C(28B)	120.8(2)
C(13B)-C(12B)-C(11B)	116.6(2)	C(32B)-C(27B)-N(2B)	120.9(2)
C(17B)-C(12B)-C(11B)	125.8(2)	C(28B)-C(27B)-N(2B)	118.2(2)
C(14B)-C(13B)-C(12B)	123.2(3)	C(29B)-C(28B)-C(27B)	118.1(3)
C(14B)-C(13B)-H(13B)	117.4(17)	C(29B)-C(28B)-C(33B)	121.0(3)
C(12B)-C(13B)-H(13B)	119.3(16)	C(27B)-C(28B)-C(33B)	120.8(3)
C(15B)-C(14B)-C(13B)	118.3(3)	C(30B)-C(29B)-C(28B)	122.2(3)
C(15B)-C(14B)-H(14B)	121.7(17)	C(30B)-C(29B)-H(29B)	119.4(18)

C(28B)-C(29B)-H(29B)	118.3(18)
C(29B)-C(30B)-C(31B)	118.3(3)
C(29B)-C(30B)-C(34B)	120.0(3)
C(31B)-C(30B)-C(34B)	121.7(3)
C(30B)-C(31B)-C(32B)	122.0(3)
C(30B)-C(31B)-H(31B)	119.6(18)
C(32B)-C(31B)-H(31B)	118.4(18)
C(31B)-C(32B)-C(27B)	118.5(3)
C(31B)-C(32B)-C(35B)	120.0(3)
C(27B)-C(32B)-C(35B)	121.5(3)
C(28B)-C(33B)-H(33D)	114.1(16)
C(28B)-C(33B)-H(33E)	111.9(17)
H(33D)-C(33B)-H(33E)	106(2)
C(28B)-C(33B)-H(33F)	114(2)
H(33D)-C(33B)-H(33F)	109(2)
H(33E)-C(33B)-H(33F)	101(2)
C(30B)-C(34B)-H(34D)	111(2)
C(30B)-C(34B)-H(34E)	107(3)
H(34D)-C(34B)-H(34E)	109(3)
C(30B)-C(34B)-H(34F)	111.0(19)
H(34D)-C(34B)-H(34F)	102(3)
H(34E)-C(34B)-H(34F)	117(3)
C(32B)-C(35B)-H(35D)	112(2)
C(32B)-C(35B)-H(35E)	108.9(19)
H(35D)-C(35B)-H(35E)	115(3)
C(32B)-C(35B)-H(35F)	111(2)
H(35D)-C(35B)-H(35F)	102(3)
H(35E)-C(35B)-H(35F)	109(3)
C(46)-C(41)-C(42)	119.8(3)
C(46)-C(41)-H(41)	117(2)
C(42)-C(41)-H(41)	123(2)
C(43)-C(42)-C(41)	120.0(4)
C(43)-C(42)-H(42)	118.9(19)
C(41)-C(42)-H(42)	121.0(19)
C(42)-C(43)-C(44)	120.0(3)
C(42)-C(43)-H(43)	120.9(18)
C(44)-C(43)-H(43)	118.9(18)
C(43)-C(44)-C(45)	120.3(3)
C(43)-C(44)-H(44)	118.9(19)
C(45)-C(44)-H(44)	120.8(19)
C(44)-C(45)-C(46)	119.5(3)
C(44)-C(45)-H(45)	118.0(15)
C(46)-C(45)-H(45)	122.4(15)
C(41)-C(46)-C(45)	120.5(3)
C(41)-C(46)-H(46)	120.6(17)
C(45)-C(46)-H(46)	118.9(17)
C(52)-C(51)-C(53)	120.7(3)
C(52)-C(51)-H(51)	121(2)
C(53)-C(51)-H(51)	118(2)
C(51)-C(52)-C(53)#1	120.0(3)
C(51)-C(52)-H(52)	119.3(16)
C(53)#1-C(52)-H(52)	120.6(16)
C(51)-C(53)-C(52)#1	119.2(3)
C(51)-C(53)-H(53)	121.0(19)
C(52)#1-C(53)-H(53)	119.6(19)

Symmetry transformations used to generate equivalent atoms: #1 -x,-y,-z

CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE

X-RAY CRYSTALLOGRAPHY LABORATORY

Date 8 August 2008

Crystal Structure Analysis of:

([^{Mes}NNN]Fe)$_2$O (ECW05)

(shown below)

For Investigator: Edward Weintrob ext. 6576

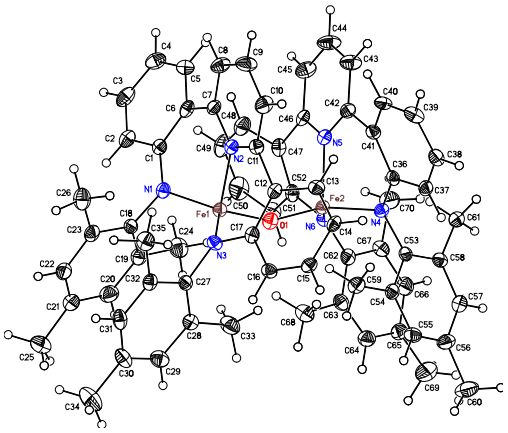
Advisor: J. E. Bercaw ext. 6577

Account Number: JEB.ENERGY-1.02-GRANT.MOOREJCP

By Michael W. Day 116 Beckman ext. 2734

e-mail: mikeday@caltech.edu

Contents



ECW05

Note: The crystallographic data have been deposited in the Cambridge Database (CCDC) and has been placed on hold pending further instructions from me. The deposition number is 697910. Ideally the CCDC would like the publication to contain a footnote of the type: "Crystallographic data have been deposited at the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK and copies can be obtained on request, free of charge, by quoting the publication citation and the deposition number 697910."

Table 1. Crystal data and structure refinement for ECW05 (CCDC 697910).

Empirical formula	$C_{70}H_{66}N_6OFe_2 \bullet 0.5(C_7H_8)$
Formula weight	1165.05
Crystallization Solvent	Toluene
Crystal Habit	Plate
Crystal size	0.38 x 0.24 x 0.07 mm^3
Crystal color	Green

Data Collection

Type of diffractometer	Bruker KAPPA APEX II
Wavelength	0.71073 Å MoKα
Data Collection Temperature	100(2) K
θ range for 9283 reflections used in lattice determination	2.30 to 31.52°
Unit cell dimensions	a = 19.8707(9) Å b = 39.4031(19) Å c = 15.1038(6) Å
Volume	11825.8(9) $Å^3$
Z	8
Crystal system	Orthorhombic
Space group	I*ba*2
Density (calculated)	1.309 Mg/m^3
F(000)	4904
Data collection program	Bruker APEX2 v2.1-0
θ range for data collection	1.86 to 32.23°
Completeness to θ = 32.23°	93.8 %
Index ranges	$-29 \le h \le 29$, $-59 \le k \le 42$, $-19 \le l \le 22$
Data collection scan type	ω scans; 12 settings
Data reduction program	Bruker SAINT-Plus v7.34A
Reflections collected	121688
Independent reflections	18216 [R_{int}= 0.0571]
Absorption coefficient	0.542 mm^{-1}
Absorption correction	None
Max. and min. transmission	0.9630 and 0.8204

Table 1 (cont.)

Structure solution and Refinement

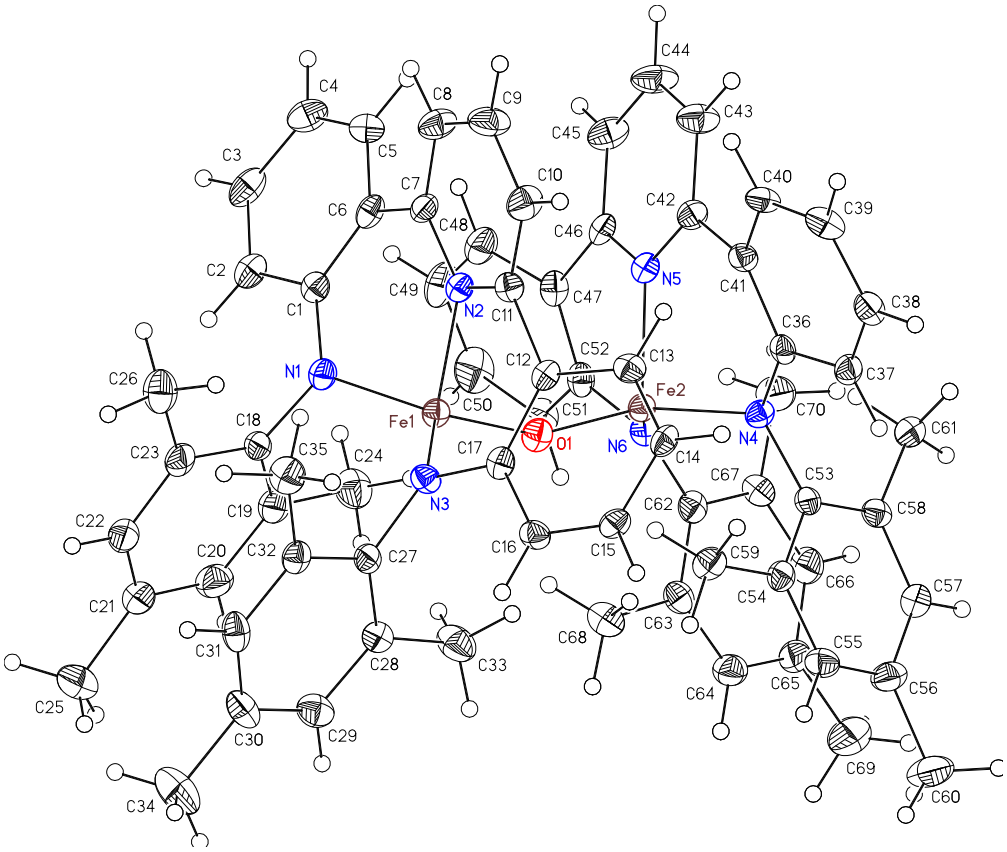
Structure solution program	SHELXS-97 (Sheldrick, 2008)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	SHELXL-97 (Sheldrick, 2008)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	18216 / 10 / 757
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	1.792
Final R indices [I>2σ(I), 14462 reflections]	R1 = 0.0416, *w*R2 = 0.0558
R indices (all data)	R1 = 0.0600, *w*R2 = 0.0567
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.003
Average shift/error	0.000
Absolute structure determination	Anomalous differences
Absolute structure parameter	-0.007(6)
Largest diff. peak and hole	1.372 and -0.704 e.$Å^{-3}$

Special Refinement Details

Crystals were mounted on a glass fiber using Paratone oil then placed on the diffractometer under a nitrogen stream at 100K.

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

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.


C44
C4
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C9
C43
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C6
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N5
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C50
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C70
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O1
N6
N4
C23
C35
C24
C17
C14
C67
C53
N3
C62
C58
C22
C19
C16
C15
C59
C20
C32
C27
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C21
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C68
C31
C28
C33
C55
C25
C65
C56
C64
C30
C29
C69
C34
C60

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for ECW05 (CCDC 697910). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Fe(1)	7380(1)	8495(1)	10926(1)	16(1)
Fe(2)	7526(1)	8565(1)	8636(1)	16(1)
O(1)	7402(1)	8618(1)	9792(1)	21(1)
N(1)	8180(1)	8518(1)	11632(1)	19(1)
N(2)	7370(1)	7983(1)	10897(1)	16(1)
N(3)	6529(1)	8538(1)	11526(1)	18(1)
N(4)	6787(1)	8425(1)	7895(1)	17(1)
N(5)	8028(1)	8124(1)	8483(1)	17(1)
N(6)	8206(1)	8840(1)	8083(1)	18(1)
C(1)	8708(1)	8299(1)	11484(1)	18(1)
C(2)	9373(1)	8391(1)	11729(1)	23(1)
C(3)	9912(1)	8190(1)	11523(1)	27(1)
C(4)	9823(1)	7887(1)	11070(1)	27(1)
C(5)	9181(1)	7785(1)	10874(1)	22(1)
C(6)	8607(1)	7975(1)	11101(1)	19(1)
C(7)	7949(1)	7801(1)	10999(1)	19(1)
C(8)	7909(1)	7449(1)	11032(1)	26(1)
C(9)	7294(1)	7289(1)	10922(2)	29(1)
C(10)	6722(1)	7481(1)	10808(1)	26(1)
C(11)	6760(1)	7832(1)	10801(1)	18(1)
C(12)	6139(1)	8040(1)	10700(1)	17(1)
C(13)	5624(1)	7897(1)	10185(1)	19(1)
C(14)	4989(1)	8035(1)	10140(1)	20(1)
C(15)	4850(1)	8326(1)	10626(1)	19(1)
C(16)	5349(1)	8483(1)	11104(1)	19(1)
C(17)	6014(1)	8356(1)	11122(1)	18(1)
C(18)	8271(1)	8820(1)	12134(1)	19(1)
C(19)	8463(1)	9122(1)	11722(1)	24(1)
C(20)	8476(1)	9417(1)	12220(1)	30(1)
C(21)	8305(1)	9422(1)	13118(1)	31(1)
C(22)	8132(1)	9116(1)	13510(1)	28(1)
C(23)	8118(1)	8816(1)	13049(1)	22(1)
C(24)	8664(1)	9124(1)	10762(1)	31(1)
C(25)	8297(1)	9749(1)	13633(2)	50(1)
C(26)	7947(1)	8485(1)	13498(1)	31(1)
C(27)	6372(1)	8808(1)	12120(1)	18(1)
C(28)	6424(1)	9147(1)	11842(1)	20(1)
C(29)	6325(1)	9406(1)	12450(1)	25(1)
C(30)	6169(1)	9340(1)	13334(1)	26(1)
C(31)	6107(1)	9004(1)	13585(1)	24(1)
C(32)	6199(1)	8739(1)	13004(1)	20(1)
C(33)	6572(1)	9232(1)	10887(1)	31(1)
C(34)	6062(1)	9631(1)	13968(1)	44(1)
C(35)	6107(1)	8376(1)	13307(1)	27(1)
C(36)	6541(1)	8101(1)	7933(1)	16(1)
C(37)	5878(1)	8026(1)	7672(1)	20(1)
C(38)	5605(1)	7708(1)	7745(1)	22(1)

C(39)	5979(1)	7445(1)	8100(1)	24(1)
C(40)	6632(1)	7506(1)	8338(1)	21(1)
C(41)	6948(1)	7821(1)	8226(1)	18(1)
C(42)	7693(1)	7830(1)	8310(1)	19(1)
C(43)	8061(1)	7535(1)	8154(1)	28(1)
C(44)	8756(1)	7544(1)	8191(1)	32(1)
C(45)	9081(1)	7842(1)	8362(1)	27(1)
C(46)	8716(1)	8137(1)	8517(1)	19(1)
C(47)	9071(1)	8461(1)	8678(1)	20(1)
C(48)	9712(1)	8436(1)	9075(1)	26(1)
C(49)	10152(1)	8702(1)	9122(1)	32(1)
C(50)	9958(1)	9011(1)	8760(1)	35(1)
C(51)	9326(1)	9055(1)	8419(1)	28(1)
C(52)	8853(1)	8785(1)	8390(1)	20(1)
C(53)	6399(1)	8693(1)	7508(1)	17(1)
C(54)	5914(1)	8866(1)	8008(1)	19(1)
C(55)	5601(1)	9149(1)	7644(1)	23(1)
C(56)	5755(1)	9263(1)	6801(1)	25(1)
C(57)	6228(1)	9085(1)	6307(1)	22(1)
C(58)	6548(1)	8800(1)	6647(1)	18(1)
C(59)	5729(1)	8747(1)	8924(1)	23(1)
C(60)	5417(1)	9575(1)	6434(1)	38(1)
C(61)	7040(1)	8606(1)	6082(1)	23(1)
C(62)	8065(1)	9128(1)	7541(1)	19(1)
C(63)	7634(1)	9382(1)	7838(1)	21(1)
C(64)	7468(1)	9649(1)	7285(1)	25(1)
C(65)	7724(1)	9677(1)	6439(1)	26(1)
C(66)	8157(1)	9422(1)	6157(1)	27(1)
C(67)	8334(1)	9149(1)	6676(1)	23(1)
C(68)	7356(1)	9380(1)	8775(1)	28(1)
C(69)	7543(1)	9972(1)	5848(1)	44(1)
C(70)	8796(1)	8877(1)	6331(1)	31(1)
C(81)	10000	10000	7764(3)	288(7)
C(82)	10000	10000	6753(3)	204(4)
C(83)	9909(2)	9699(1)	6290(3)	155(3)
C(84)	9909(2)	9699(1)	5371(3)	381(8)
C(85)	10000	10000	4906(3)	207(6)

Table 3. Selected bond lengths [Å] and angles [°] for ECW05 (CCDC 697910).

Fe(1)-O(1)	1.7802(14)	O(1)-Fe(1)-N(1)	120.08(6)
Fe(1)-N(1)	1.9174(15)	O(1)-Fe(1)-N(3)	116.84(6)
Fe(1)-N(3)	1.9262(14)	N(1)-Fe(1)-N(3)	117.50(6)
Fe(1)-N(2)	2.0161(14)	O(1)-Fe(1)-N(2)	104.57(6)
Fe(2)-O(1)	1.7761(14)	N(1)-Fe(1)-N(2)	93.86(6)
Fe(2)-N(6)	1.9234(15)	N(3)-Fe(1)-N(2)	95.14(6)
Fe(2)-N(4)	1.9262(15)	O(1)-Fe(2)-N(6)	117.27(6)
Fe(2)-N(5)	2.0153(15)	O(1)-Fe(2)-N(4)	119.97(6)
		N(6)-Fe(2)-N(4)	116.39(6)
		O(1)-Fe(2)-N(5)	106.37(6)
		N(6)-Fe(2)-N(5)	95.06(6)
		N(4)-Fe(2)-N(5)	93.67(6)

Table 4. Bond lengths [Å] and angles [°] for ECW05 (CCDC 697910).

Fe(1)-O(1)	1.7802(14)	C(29)-C(30)	1.396(3)
Fe(1)-N(1)	1.9174(15)	C(30)-C(31)	1.383(3)
Fe(1)-N(3)	1.9262(14)	C(30)-C(34)	1.508(3)
Fe(1)-N(2)	2.0161(14)	C(31)-C(32)	1.378(3)
Fe(2)-O(1)	1.7761(14)	C(32)-C(35)	1.513(3)
Fe(2)-N(6)	1.9234(15)	C(36)-C(37)	1.406(2)
Fe(2)-N(4)	1.9262(15)	C(36)-C(41)	1.437(3)
Fe(2)-N(5)	2.0153(15)	C(37)-C(38)	1.370(3)
N(1)-C(1)	1.378(2)	C(38)-C(39)	1.382(3)
N(1)-C(18)	1.421(2)	C(39)-C(40)	1.369(2)
N(2)-C(11)	1.358(2)	C(40)-C(41)	1.402(3)
N(2)-C(7)	1.365(2)	C(41)-C(42)	1.487(2)
N(3)-C(17)	1.391(2)	C(42)-C(43)	1.395(3)
N(3)-C(27)	1.428(2)	C(43)-C(44)	1.382(3)
N(4)-C(36)	1.370(2)	C(44)-C(45)	1.362(3)
N(4)-C(53)	1.431(2)	C(45)-C(46)	1.391(3)
N(5)-C(42)	1.361(2)	C(46)-C(47)	1.478(2)
N(5)-C(46)	1.371(2)	C(47)-C(48)	1.410(2)
N(6)-C(52)	1.384(2)	C(47)-C(52)	1.416(3)
N(6)-C(62)	1.427(2)	C(48)-C(49)	1.367(3)
C(1)-C(2)	1.419(2)	C(49)-C(50)	1.387(3)
C(1)-C(6)	1.414(3)	C(50)-C(51)	1.369(3)
C(2)-C(3)	1.370(3)	C(51)-C(52)	1.422(3)
C(3)-C(4)	1.386(3)	C(53)-C(58)	1.398(2)
C(4)-C(5)	1.370(2)	C(53)-C(54)	1.402(2)
C(5)-C(6)	1.408(2)	C(54)-C(55)	1.391(3)
C(6)-C(7)	1.486(2)	C(54)-C(59)	1.506(2)
C(7)-C(8)	1.389(3)	C(55)-C(56)	1.385(2)
C(8)-C(9)	1.384(2)	C(56)-C(57)	1.391(3)
C(9)-C(10)	1.376(3)	C(56)-C(60)	1.506(3)
C(10)-C(11)	1.385(3)	C(57)-C(58)	1.390(3)
C(11)-C(12)	1.487(2)	C(58)-C(61)	1.507(2)
C(12)-C(13)	1.403(2)	C(62)-C(63)	1.391(3)
C(12)-C(17)	1.421(3)	C(62)-C(67)	1.414(2)
C(13)-C(14)	1.376(2)	C(63)-C(64)	1.384(3)
C(14)-C(15)	1.387(2)	C(63)-C(68)	1.519(2)
C(15)-C(16)	1.374(2)	C(64)-C(65)	1.380(3)
C(16)-C(17)	1.413(2)	C(65)-C(66)	1.390(3)
C(18)-C(19)	1.397(3)	C(65)-C(69)	1.507(3)
C(18)-C(23)	1.415(2)	C(66)-C(67)	1.379(3)
C(19)-C(20)	1.385(3)	C(67)-C(70)	1.504(3)
C(19)-C(24)	1.503(3)	C(81)-C(82)	1.5266(9)
C(20)-C(21)	1.400(3)	C(82)-C(83)#1	1.3880(9)
C(21)-C(22)	1.384(3)	C(82)-C(83)	1.3881(9)
C(21)-C(25)	1.506(3)	C(83)-C(84)	1.3885(9)
C(22)-C(23)	1.374(3)	C(84)-C(85)	1.3897(10)
C(23)-C(26)	1.509(3)	C(85)-C(84)#1	1.3898(10)
C(27)-C(28)	1.402(3)		
C(27)-C(32)	1.406(2)	O(1)-Fe(1)-N(1)	120.08(6)
C(28)-C(29)	1.389(3)	O(1)-Fe(1)-N(3)	116.84(6)
C(28)-C(33)	1.509(2)	N(1)-Fe(1)-N(3)	117.50(6)

O(1)-Fe(1)-N(2)	104.57(6)	N(3)-C(17)-C(16)	120.89(17)
N(1)-Fe(1)-N(2)	93.86(6)	N(3)-C(17)-C(12)	121.30(16)
N(3)-Fe(1)-N(2)	95.14(6)	C(16)-C(17)-C(12)	117.80(16)
O(1)-Fe(2)-N(6)	117.27(6)	C(19)-C(18)-C(23)	120.15(19)
O(1)-Fe(2)-N(4)	119.97(6)	C(19)-C(18)-N(1)	120.67(16)
N(6)-Fe(2)-N(4)	116.39(6)	C(23)-C(18)-N(1)	119.04(18)
O(1)-Fe(2)-N(5)	106.37(6)	C(20)-C(19)-C(18)	118.61(18)
N(6)-Fe(2)-N(5)	95.06(6)	C(20)-C(19)-C(24)	120.88(19)
N(4)-Fe(2)-N(5)	93.67(6)	C(18)-C(19)-C(24)	120.51(19)
Fe(2)-O(1)-Fe(1)	156.60(7)	C(19)-C(20)-C(21)	122.3(2)
C(1)-N(1)-C(18)	120.93(15)	C(22)-C(21)-C(20)	117.6(2)
C(1)-N(1)-Fe(1)	120.81(12)	C(22)-C(21)-C(25)	121.43(19)
C(18)-N(1)-Fe(1)	116.21(12)	C(20)-C(21)-C(25)	121.0(2)
C(11)-N(2)-C(7)	122.30(15)	C(23)-C(22)-C(21)	122.49(19)
C(11)-N(2)-Fe(1)	116.62(12)	C(22)-C(23)-C(18)	118.85(19)
C(7)-N(2)-Fe(1)	121.04(12)	C(22)-C(23)-C(26)	121.44(18)
C(17)-N(3)-C(27)	120.01(14)	C(18)-C(23)-C(26)	119.71(18)
C(17)-N(3)-Fe(1)	113.20(11)	C(28)-C(27)-C(32)	119.27(18)
C(27)-N(3)-Fe(1)	123.58(12)	C(28)-C(27)-N(3)	120.36(16)
C(36)-N(4)-C(53)	120.80(15)	C(32)-C(27)-N(3)	120.28(18)
C(36)-N(4)-Fe(2)	120.97(12)	C(29)-C(28)-C(27)	119.46(17)
C(53)-N(4)-Fe(2)	115.93(12)	C(29)-C(28)-C(33)	119.71(18)
C(42)-N(5)-C(46)	121.72(16)	C(27)-C(28)-C(33)	120.81(17)
C(42)-N(5)-Fe(2)	120.95(12)	C(28)-C(29)-C(30)	121.74(19)
C(46)-N(5)-Fe(2)	117.25(12)	C(31)-C(30)-C(29)	117.50(18)
C(52)-N(6)-C(62)	120.02(15)	C(31)-C(30)-C(34)	122.66(18)
C(52)-N(6)-Fe(2)	114.75(12)	C(29)-C(30)-C(34)	119.83(19)
C(62)-N(6)-Fe(2)	123.96(12)	C(30)-C(31)-C(32)	122.70(18)
N(1)-C(1)-C(2)	120.41(17)	C(31)-C(32)-C(27)	119.28(19)
N(1)-C(1)-C(6)	121.52(16)	C(31)-C(32)-C(35)	120.62(17)
C(2)-C(1)-C(6)	118.07(17)	C(27)-C(32)-C(35)	120.09(18)
C(3)-C(2)-C(1)	121.34(19)	N(4)-C(36)-C(37)	121.31(17)
C(2)-C(3)-C(4)	120.65(18)	N(4)-C(36)-C(41)	121.83(16)
C(5)-C(4)-C(3)	118.70(18)	C(37)-C(36)-C(41)	116.85(18)
C(4)-C(5)-C(6)	122.93(18)	C(38)-C(37)-C(36)	122.77(18)
C(5)-C(6)-C(1)	117.77(16)	C(37)-C(38)-C(39)	120.26(18)
C(5)-C(6)-C(7)	116.14(17)	C(40)-C(39)-C(38)	118.66(19)
C(1)-C(6)-C(7)	125.76(16)	C(39)-C(40)-C(41)	123.27(19)
N(2)-C(7)-C(8)	118.77(16)	C(40)-C(41)-C(36)	117.70(17)
N(2)-C(7)-C(6)	120.70(16)	C(40)-C(41)-C(42)	117.14(17)
C(8)-C(7)-C(6)	120.51(16)	C(36)-C(41)-C(42)	124.69(17)
C(9)-C(8)-C(7)	120.01(17)	N(5)-C(42)-C(43)	119.14(17)
C(10)-C(9)-C(8)	119.52(18)	N(5)-C(42)-C(41)	121.49(17)
C(9)-C(10)-C(11)	120.43(18)	C(43)-C(42)-C(41)	119.24(18)
N(2)-C(11)-C(10)	118.91(16)	C(44)-C(43)-C(42)	119.6(2)
N(2)-C(11)-C(12)	120.75(16)	C(45)-C(44)-C(43)	120.3(2)
C(10)-C(11)-C(12)	120.34(16)	C(44)-C(45)-C(46)	120.32(18)
C(13)-C(12)-C(17)	118.16(16)	N(5)-C(46)-C(45)	118.86(17)
C(13)-C(12)-C(11)	116.22(17)	N(5)-C(46)-C(47)	120.98(16)
C(17)-C(12)-C(11)	125.50(16)	C(45)-C(46)-C(47)	120.10(17)
C(14)-C(13)-C(12)	122.53(18)	C(48)-C(47)-C(52)	117.91(17)
C(13)-C(14)-C(15)	118.88(17)	C(48)-C(47)-C(46)	116.22(17)
C(16)-C(15)-C(14)	120.42(17)	C(52)-C(47)-C(46)	125.57(16)
C(15)-C(16)-C(17)	121.63(17)	C(49)-C(48)-C(47)	123.2(2)

C(48)-C(49)-C(50)	118.32(18)
C(51)-C(50)-C(49)	120.99(19)
C(50)-C(51)-C(52)	121.45(19)
N(6)-C(52)-C(47)	121.93(17)
N(6)-C(52)-C(51)	120.39(18)
C(47)-C(52)-C(51)	117.68(17)
C(58)-C(53)-C(54)	120.03(17)
C(58)-C(53)-N(4)	119.14(16)
C(54)-C(53)-N(4)	120.60(16)
C(55)-C(54)-C(53)	119.01(17)
C(55)-C(54)-C(59)	120.24(17)
C(53)-C(54)-C(59)	120.74(17)
C(56)-C(55)-C(54)	121.66(18)
C(55)-C(56)-C(57)	118.59(18)
C(55)-C(56)-C(60)	120.29(18)
C(57)-C(56)-C(60)	121.12(18)
C(56)-C(57)-C(58)	121.36(18)
C(57)-C(58)-C(53)	119.32(17)
C(57)-C(58)-C(61)	119.89(17)
C(53)-C(58)-C(61)	120.78(17)
C(63)-C(62)-C(67)	119.28(18)
C(63)-C(62)-N(6)	120.59(16)
C(67)-C(62)-N(6)	120.04(17)
C(64)-C(63)-C(62)	119.96(18)
C(64)-C(63)-C(68)	118.63(17)
C(62)-C(63)-C(68)	121.39(17)
C(65)-C(64)-C(63)	122.15(19)
C(64)-C(65)-C(66)	117.03(19)
C(64)-C(65)-C(69)	121.48(19)
C(66)-C(65)-C(69)	121.49(19)
C(67)-C(66)-C(65)	123.23(18)
C(66)-C(67)-C(62)	118.35(18)
C(66)-C(67)-C(70)	120.91(17)
C(62)-C(67)-C(70)	120.73(18)
C(83)#1-C(82)-C(83)	119.49(17)
C(83)#1-C(82)-C(81)	120.26(8)
C(83)-C(82)-C(81)	120.25(8)
C(82)-C(83)-C(84)	120.28(11)
C(83)-C(84)-C(85)	120.30(11)
C(84)-C(85)-C(84)#1	119.36(18)

Symmetry transformations used to
generate equivalent atoms:
#1 -x+2,-y+2,z

CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE

X-RAY CRYSTALLOGRAPHY LABORATORY

Date 23 July 2010

Crystal Structure Analysis of:

[^{Mes}NNN]FeCH$_2$PMe$_3$ (ECW09)

(shown below)

For Investigator: Ed Weintrob ext. 6576

Advisor: J. E. Bercaw ext. 6577

Account Number: JEB.ENERGY-1.02-GRANT.MOOREJCP

By Michael W. Day 116 Beckman ext. 2734

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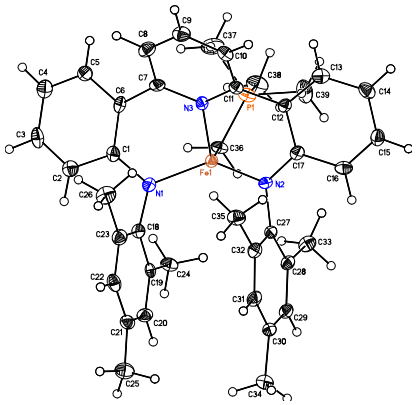
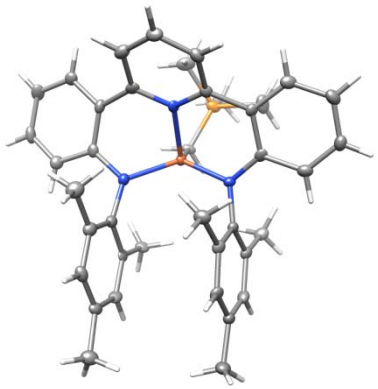
Contents





ECW09

Note: The crystallographic data have been deposited in the Cambridge Database (CCDC) and has been placed on hold pending further instructions from me. The deposition number is 785426. Ideally the CCDC would like the publication to contain a footnote of the type: "Crystallographic data have been deposited at the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK and copies can be obtained on request, free of charge, by quoting the publication citation and the deposition number 785426."

Table 1. Crystal data and structure refinement for ECW09 (CCDC 785426).

Empirical formula	$C_{39}H_{44}N_3PFe \bullet 1.5(C_6H_6)$
Formula weight	758.75
Crystallization Solvent	Benzene/pentane
Crystal Habit	Blade
Crystal size	0.21 x 0.11 x 0.07 mm^3
Crystal color	Dark red

Data Collection

Type of diffractometer	Bruker KAPPA APEX II	
Wavelength	0.71073 Å MoKα	
Data Collection Temperature	100(2) K	
θ range for 6619 reflections used in lattice determination	2.32 to 27.47°	
Unit cell dimensions	a = 10.0318(4) Å b = 37.7399(15) Å c = 10.8356(4) Å	α= 90° β= 98.561(2)° γ = 90°
Volume	4056.6(3) $Å^3$	
Z	4	
Crystal system	Monoclinic	
Space group	P $2_1/n$	
Density (calculated)	1.242 Mg/m^3	
F(000)	1612	
Data collection program	Bruker APEX2 v2009.7-0	
θ range for data collection	1.08 to 28.31°	
Completeness to θ = 28.31°	94.4 %	
Index ranges	$-13 \le h \le 13$, $-49 \le k \le 50$, $-12 \le l \le 14$	
Data collection scan type	ω scans; 6 settings	
Data reduction program	Bruker SAINT-Plus v7.66A	
Reflections collected	32506	
Independent reflections	9557 [R_{int}= 0.1258]	
Absorption coefficient	0.448 mm^{-1}	
Absorption correction	None	
Max. and min. transmission	0.9693 and 0.9118	

Table 1 (cont.)

Structure solution and Refinement

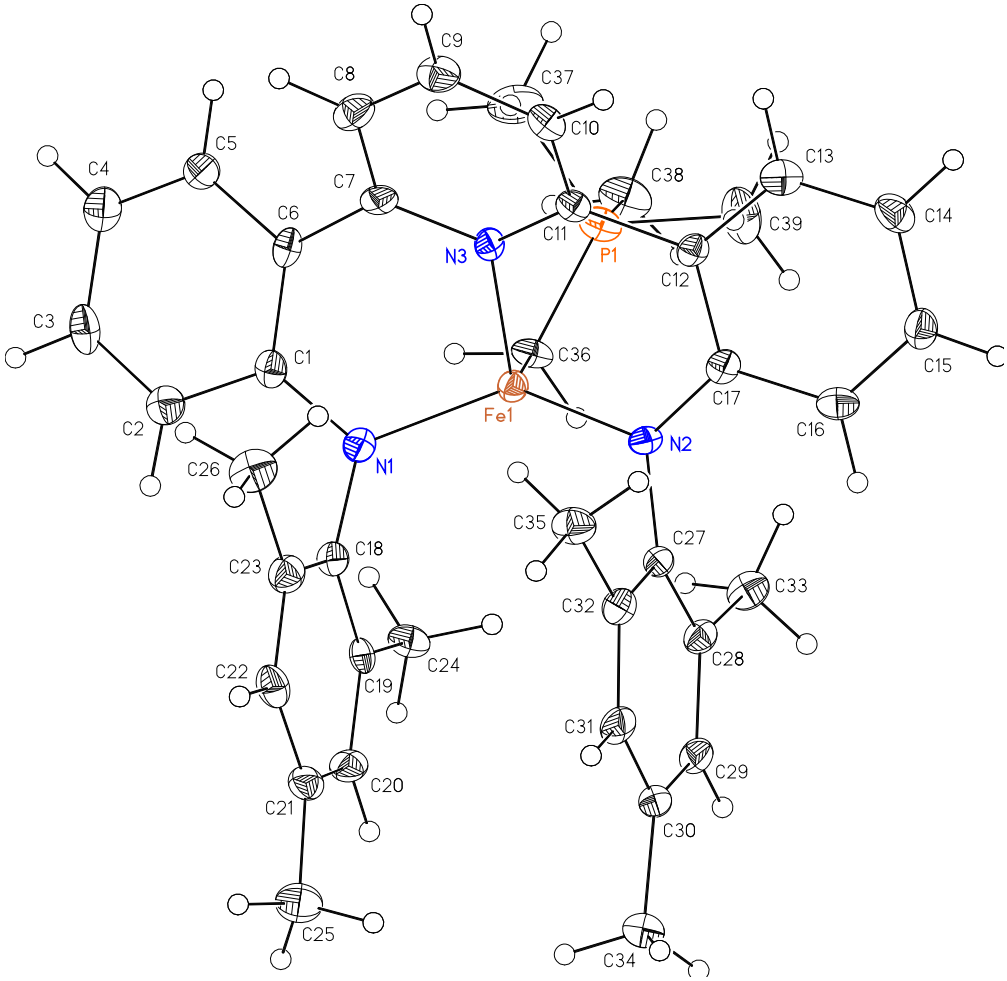
Structure solution program	SHELXS-97 (Sheldrick, 2008)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	SHELXL-97 (Sheldrick, 2008)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	9557 / 0 / 487
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	1.292
Final R indices [$I>2\sigma(I)$, 6045 reflections]	R1 = 0.0630, *w*R2 = 0.1094
R indices (all data)	R1 = 0.1102, *w*R2 = 0.1148
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.000
Average shift/error	0.000
Largest diff. peak and hole	0.775 and -0.798 e.Å^{-3}

Special Refinement Details

Crystals were mounted on a glass fiber using Paratone oil then placed on the diffractometer under a nitrogen stream at 100K.

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

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.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C37
C10
C5
C4
C7
C38
C13
C6
C11
C39
C14
N3
P1
C12
C3
C36
C15
C1
Fe1
C17
C2
C16
N2
C26
N1
C35
C27
C18
C23
C33
C32
C28
C24
C22
C19
C31
C29
C20
C21
C30
C25
C34

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for ECW09 (CCDC 785426). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Fe(1)	5324(1)	1475(1)	6796(1)	14(1)
P(1)	3260(1)	1577(1)	8838(1)	19(1)
N(1)	5064(2)	1474(1)	4954(2)	17(1)
N(2)	7192(2)	1438(1)	7640(2)	14(1)
N(3)	5080(2)	932(1)	6784(2)	13(1)
C(1)	3828(3)	1337(1)	4439(3)	15(1)
C(2)	3026(3)	1493(1)	3413(3)	21(1)
C(3)	1743(3)	1384(1)	3007(3)	23(1)
C(4)	1175(3)	1101(1)	3590(3)	23(1)
C(5)	1964(3)	929(1)	4546(3)	18(1)
C(6)	3306(3)	1032(1)	4986(3)	16(1)
C(7)	4144(3)	794(1)	5850(3)	15(1)
C(8)	4025(3)	429(1)	5702(3)	21(1)
C(9)	4895(3)	207(1)	6454(3)	22(1)
C(10)	5861(3)	347(1)	7340(3)	18(1)
C(11)	5919(3)	716(1)	7539(3)	14(1)
C(12)	6917(3)	853(1)	8561(3)	14(1)
C(13)	7297(3)	637(1)	9589(3)	20(1)
C(14)	8352(3)	714(1)	10525(3)	22(1)
C(15)	9098(3)	1018(1)	10418(3)	20(1)
C(16)	8741(3)	1248(1)	9445(3)	19(1)
C(17)	7603(3)	1188(1)	8527(3)	15(1)
C(18)	5900(3)	1642(1)	4174(3)	16(1)
C(19)	6190(3)	2004(1)	4297(3)	15(1)
C(20)	7148(3)	2149(1)	3635(3)	18(1)
C(21)	7816(3)	1948(1)	2844(3)	18(1)
C(22)	7465(3)	1595(1)	2698(3)	19(1)
C(23)	6531(3)	1435(1)	3357(3)	20(1)
C(24)	5498(3)	2231(1)	5143(3)	20(1)
C(25)	8909(3)	2106(1)	2197(3)	25(1)
C(26)	6224(3)	1043(1)	3196(3)	26(1)
C(27)	8178(3)	1673(1)	7277(3)	15(1)
C(28)	8273(3)	2028(1)	7654(3)	17(1)
C(29)	9143(3)	2252(1)	7125(3)	20(1)
C(30)	9934(3)	2132(1)	6273(3)	20(1)
C(31)	9863(3)	1776(1)	5944(3)	20(1)
C(32)	9004(3)	1544(1)	6432(3)	17(1)
C(33)	7447(3)	2169(1)	8600(3)	21(1)
C(34)	10786(3)	2385(1)	5648(3)	28(1)
C(35)	8897(3)	1163(1)	6024(3)	22(1)
C(36)	3887(3)	1781(1)	7609(3)	18(1)
C(37)	2329(3)	1190(1)	8304(3)	30(1)
C(38)	2115(3)	1841(1)	9602(3)	28(1)
C(39)	4594(3)	1456(1)	10065(3)	32(1)
C(51)	4089(3)	472(1)	883(3)	28(1)
C(52)	3734(3)	341(1)	-300(3)	26(1)

C(53)	2404(3)	273(1)	-776(3)	30(1)
C(54)	1409(3)	337(1)	-44(4)	34(1)
C(55)	1771(3)	465(1)	1153(4)	31(1)
C(56)	3100(3)	538(1)	1608(3)	26(1)
C(61)	8996(5)	212(1)	5322(5)	53(1)
C(62)	9170(5)	181(1)	4103(5)	58(1)
C(63)	10180(5)	-26(1)	3789(4)	57(1)

Table 3. Selected bond lengths [Å] and angles [°] for ECW09 (CCDC 785426).

Fe(1)-N(2)	1.964(2)
Fe(1)-N(1)	1.974(2)
Fe(1)-N(3)	2.062(2)
Fe(1)-C(36)	2.137(3)
N(2)-Fe(1)-N(1)	116.38(9)
N(2)-Fe(1)-N(3)	92.16(10)
N(1)-Fe(1)-N(3)	89.71(10)
N(2)-Fe(1)-C(36)	120.06(11)
N(1)-Fe(1)-C(36)	114.93(11)
N(3)-Fe(1)-C(36)	116.94(10)

Table 4. Bond lengths [Å] and angles [°] for ECW09 (CCDC 785426).

Fe(1)-N(2)	1.964(2)	C(51)-C(56)	1.377(4)
Fe(1)-N(1)	1.974(2)	C(52)-C(53)	1.381(5)
Fe(1)-N(3)	2.062(2)	C(53)-C(54)	1.385(4)
Fe(1)-C(36)	2.137(3)	C(54)-C(55)	1.381(5)
P(1)-C(36)	1.735(3)	C(55)-C(56)	1.378(5)
P(1)-C(37)	1.784(3)	C(61)-C(62)	1.363(6)
P(1)-C(39)	1.800(3)	C(61)-C(63)#1	1.367(6)
P(1)-C(38)	1.809(3)	C(62)-C(63)	1.361(6)
N(1)-C(1)	1.383(4)	C(63)-C(61)#1	1.367(6)
N(1)-C(18)	1.425(4)		
N(2)-C(17)	1.368(4)	N(2)-Fe(1)-N(1)	116.38(9)
N(2)-C(27)	1.427(3)	N(2)-Fe(1)-N(3)	92.16(10)
N(3)-C(11)	1.356(4)	N(1)-Fe(1)-N(3)	89.71(10)
N(3)-C(7)	1.375(4)	N(2)-Fe(1)-C(36)	120.06(11)
C(1)-C(2)	1.402(4)	N(1)-Fe(1)-C(36)	114.93(11)
C(1)-C(6)	1.429(4)	N(3)-Fe(1)-C(36)	116.94(10)
C(2)-C(3)	1.360(4)	C(36)-P(1)-C(37)	110.23(16)
C(3)-C(4)	1.406(4)	C(36)-P(1)-C(39)	111.38(15)
C(4)-C(5)	1.369(4)	C(37)-P(1)-C(39)	109.05(18)
C(5)-C(6)	1.414(4)	C(36)-P(1)-C(38)	115.80(14)
C(6)-C(7)	1.467(4)	C(37)-P(1)-C(38)	105.18(15)
C(7)-C(8)	1.390(4)	C(39)-P(1)-C(38)	104.79(16)
C(8)-C(9)	1.385(4)	C(1)-N(1)-C(18)	119.9(2)
C(9)-C(10)	1.365(4)	C(1)-N(1)-Fe(1)	112.60(17)
C(10)-C(11)	1.408(4)	C(18)-N(1)-Fe(1)	126.80(19)
C(11)-C(12)	1.473(4)	C(17)-N(2)-C(27)	118.4(2)
C(12)-C(13)	1.387(4)	C(17)-N(2)-Fe(1)	122.85(18)
C(12)-C(17)	1.440(4)	C(27)-N(2)-Fe(1)	118.73(19)
C(13)-C(14)	1.383(4)	C(11)-N(3)-C(7)	120.7(2)
C(14)-C(15)	1.386(4)	C(11)-N(3)-Fe(1)	122.14(19)
C(15)-C(16)	1.370(4)	C(7)-N(3)-Fe(1)	116.54(19)
C(16)-C(17)	1.416(4)	N(1)-C(1)-C(2)	122.5(3)
C(18)-C(23)	1.400(4)	N(1)-C(1)-C(6)	119.7(3)
C(18)-C(19)	1.400(4)	C(2)-C(1)-C(6)	117.8(3)
C(19)-C(20)	1.394(4)	C(3)-C(2)-C(1)	122.2(3)
C(19)-C(24)	1.500(4)	C(2)-C(3)-C(4)	120.8(3)
C(20)-C(21)	1.390(4)	C(5)-C(4)-C(3)	118.2(3)
C(21)-C(22)	1.381(4)	C(4)-C(5)-C(6)	122.6(3)
C(21)-C(25)	1.510(4)	C(5)-C(6)-C(1)	118.0(3)
C(22)-C(23)	1.397(4)	C(5)-C(6)-C(7)	118.3(3)
C(23)-C(26)	1.514(4)	C(1)-C(6)-C(7)	123.1(3)
C(27)-C(28)	1.399(4)	N(3)-C(7)-C(8)	119.8(3)
C(27)-C(32)	1.410(4)	N(3)-C(7)-C(6)	120.2(3)
C(28)-C(29)	1.397(4)	C(8)-C(7)-C(6)	120.0(3)
C(28)-C(33)	1.507(4)	C(9)-C(8)-C(7)	119.8(3)
C(29)-C(30)	1.380(4)	C(10)-C(9)-C(8)	119.9(3)
C(30)-C(31)	1.389(4)	C(9)-C(10)-C(11)	119.9(3)
C(30)-C(34)	1.510(4)	N(3)-C(11)-C(10)	119.7(3)
C(31)-C(32)	1.386(4)	N(3)-C(11)-C(12)	122.2(3)
C(32)-C(35)	1.505(4)	C(10)-C(11)-C(12)	118.1(3)
C(51)-C(52)	1.372(5)	C(13)-C(12)-C(17)	117.4(3)

C(13)-C(12)-C(11)	118.1(3)	C(28)-C(27)-N(2)	122.3(2)
C(17)-C(12)-C(11)	124.3(3)	C(32)-C(27)-N(2)	117.5(3)
C(14)-C(13)-C(12)	123.7(3)	C(29)-C(28)-C(27)	118.6(3)
C(13)-C(14)-C(15)	118.4(3)	C(29)-C(28)-C(33)	120.5(3)
C(16)-C(15)-C(14)	120.4(3)	C(27)-C(28)-C(33)	121.0(3)
C(15)-C(16)-C(17)	122.0(3)	C(30)-C(29)-C(28)	122.2(3)
N(2)-C(17)-C(16)	120.6(3)	C(29)-C(30)-C(31)	118.5(3)
N(2)-C(17)-C(12)	121.9(3)	C(29)-C(30)-C(34)	120.8(3)
C(16)-C(17)-C(12)	117.4(3)	C(31)-C(30)-C(34)	120.6(3)
C(23)-C(18)-C(19)	119.9(3)	C(32)-C(31)-C(30)	121.5(3)
C(23)-C(18)-N(1)	119.3(3)	C(31)-C(32)-C(27)	119.3(3)
C(19)-C(18)-N(1)	120.6(2)	C(31)-C(32)-C(35)	120.8(3)
C(20)-C(19)-C(18)	119.0(3)	C(27)-C(32)-C(35)	119.9(2)
C(20)-C(19)-C(24)	120.5(3)	P(1)-C(36)-Fe(1)	114.92(15)
C(18)-C(19)-C(24)	120.6(2)	C(52)-C(51)-C(56)	119.3(3)
C(21)-C(20)-C(19)	122.3(3)	C(51)-C(52)-C(53)	121.2(3)
C(22)-C(21)-C(20)	117.4(3)	C(52)-C(53)-C(54)	119.5(3)
C(22)-C(21)-C(25)	121.3(3)	C(55)-C(54)-C(53)	119.2(3)
C(20)-C(21)-C(25)	121.3(3)	C(56)-C(55)-C(54)	120.7(3)
C(21)-C(22)-C(23)	122.5(3)	C(51)-C(56)-C(55)	120.0(3)
C(18)-C(23)-C(22)	118.9(3)	C(62)-C(61)-C(63)#1	119.2(4)
C(18)-C(23)-C(26)	120.9(3)	C(61)-C(62)-C(63)	119.9(5)
C(22)-C(23)-C(26)	120.2(3)	C(62)-C(63)-C(61)#1	120.9(4)
C(28)-C(27)-C(32)	119.9(3)		

Symmetry transformations used to generate equivalent atoms:
#1 -x+2,-y,-z+1

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X-RAY CRYSTALLOGRAPHY LABORATORY

Crystal Structure Analysis of:

[^{Mes}NNN-NHAd]Fe (ECW16)

(shown below)

For Investigator: Edward Weintrob ext. 6576

Advisor: J. E. Bercaw ext. 6577

Account Number: JEB.ENERGY-1.02-GRANT.MOOREJCP

By Edward Weintrob 204 Noyes ext. 6576

e-mail: tw288@caltech.edu

Contents

Table 1. Crystal data and structure refinement for ecw16.

Empirical formula	$C_{45}H_{47}N_4Fe$	
Formula weight	699.72	

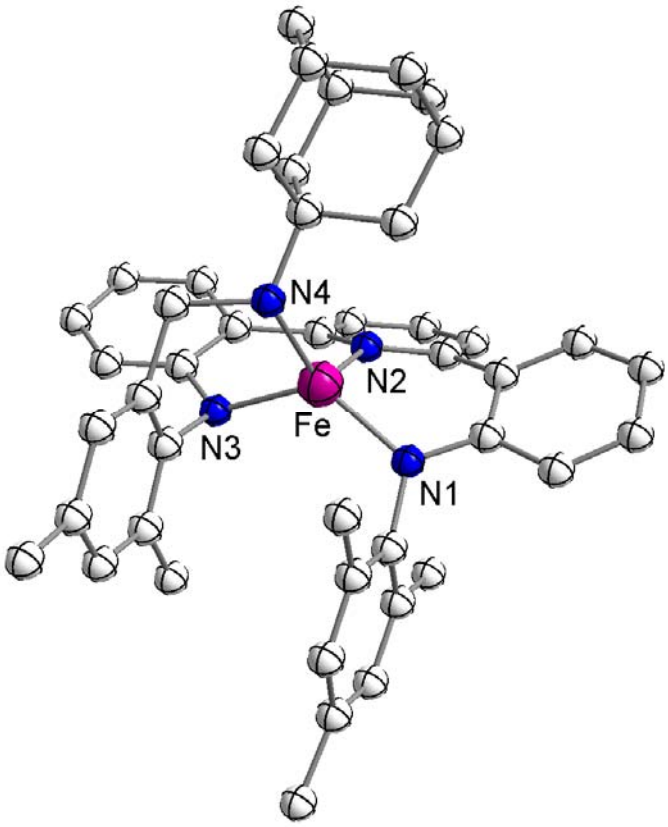
Data Collection

Type of diffractometer	Synchrotron	
Wavelength	0.72930 Å	
Data Collection Temperature	100(2) K	
Unit cell dimensions	a = 11.579(2) Å b = 13.386(3) Å c = 24.019(5) Å	α= 83.94(3)° β= 76.78(3)° γ = 79.94(3)°
Volume	3560.1(12) $Å^3$	
Z	4	
Crystal system	Triclinic	
Space group	P-1	
Density (calculated)	1.305 Mg/m^3	
F(000)	1484	
θ range for data collection	0.90 to 21.94°	
Completeness to θ = 21.94°	90.8 %	
Index ranges	-11<=h<=11, -13<=k<=13, -24<=l<=24	
Reflections collected	23622	
Independent reflections	7300 [R_{int}= 0.0895]	

Structure solution and Refinement

Structure solution program	SHELXS-97 (Sheldrick, 2008)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	SHELXL-97 (Sheldrick, 2008)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	7300 / 612 / 912
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	4.154
Final R indices [I>2σ(I), 5605 reflections]	R1 = 0.1499, *w*R2 = 0.3194
R indices (all data)	R1 = 0.1755, *w*R2 = 0.3221
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	4.790

Average shift/error	0.027
Largest diff. peak and hole	2.935 and -1.439 e.Å^{-3}



CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE

X-RAY CRYSTALLOGRAPHY LABORATORY

Date 13 September 2010

Crystal Structure Analysis of:

[^{Mes}NNN-NH_2]H_2 (ECW13)

(shown below)

For Investigator: Edward Weintrob ext. 6576

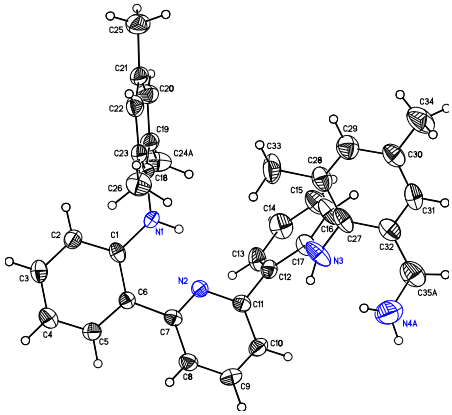
Advisor: J. E. Bercaw ext. 6577

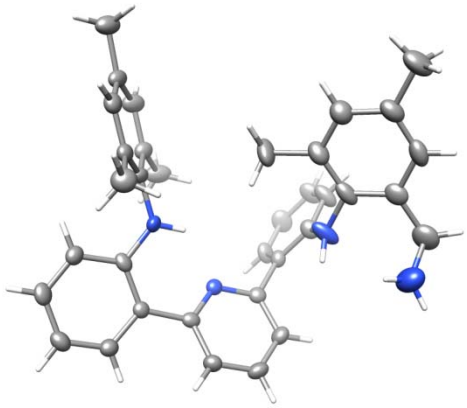
Account Number: JEB.ENERGY-1.02-GRANT.MOOREJCP

By Michael W. Day 116 Beckman ext. 2734

e-mail: mikeday@caltech.edu

Contents





ECW13

Note: The crystallographic data have been deposited in the Cambridge Database (CCDC) and has been placed on hold pending further instructions from me. The deposition number is 793154. Ideally the CCDC would like the publication to contain a footnote of the type: "Crystallographic data have been deposited at the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK and copies can be obtained on request, free of charge, by quoting the publication citation and the deposition number 793154."

Table 1. Crystal data and structure refinement for ECW13 (CCDC 793154).

Empirical formula	$C_{35}H_{36}N_4$
Formula weight	512.68
Crystallization Solvent	Dichloromethane/pentane
Crystal Habit	Fragment
Crystal size	0.24 x 0.15 x 0.12 mm^3
Crystal color	Colorless

Data Collection

Type of diffractometer	Bruker KAPPA APEX II	
Wavelength	0.71073 Å MoKα	
Data Collection Temperature	100(2) K	
θ range for 6446 reflections used in lattice determination	2.55 to 23.18°	
Unit cell dimensions	a = 12.1803(6) Å b = 8.3648(4) Å c = 27.4592(14) Å	α= 90° β= 94.852(3)° γ = 90°
Volume	2787.7(2) $Å^3$	
Z	4	
Crystal system	Monoclinic	
Space group	P $2_1/n$	
Density (calculated)	1.222 Mg/m^3	
F(000)	1096	
θ range for data collection	1.49 to 25.56°	
Completeness to θ = 25.56°	91.5 %	
Index ranges	$-13 \le h \le 13, -10 \le k \le 9, -32 \le l \le 33$	
Data collection scan type	ω scans; 8 settings	
Reflections collected	29589	
Independent reflections	4775 [R_{int}= 0.0438]	
Absorption coefficient	0.072 mm^{-1}	
Absorption correction	None	
Max. and min. transmission	0.9914 and 0.9829	

Table 1 (cont.)

Structure solution and Refinement

Structure solution program	SHELXS-97 (Sheldrick, 2008)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	SHELXL-97 (Sheldrick, 2008)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	4775 / 1 / 361
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	2.495
Final R indices [$I>2\sigma(I)$, 3224 reflections]	R1 = 0.0574, *w*R2 = 0.0734
R indices (all data)	R1 = 0.0897, *w*R2 = 0.0748
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.015
Average shift/error	0.000
Largest diff. peak and hole	0.590 and -0.422 e.Å^{-3}

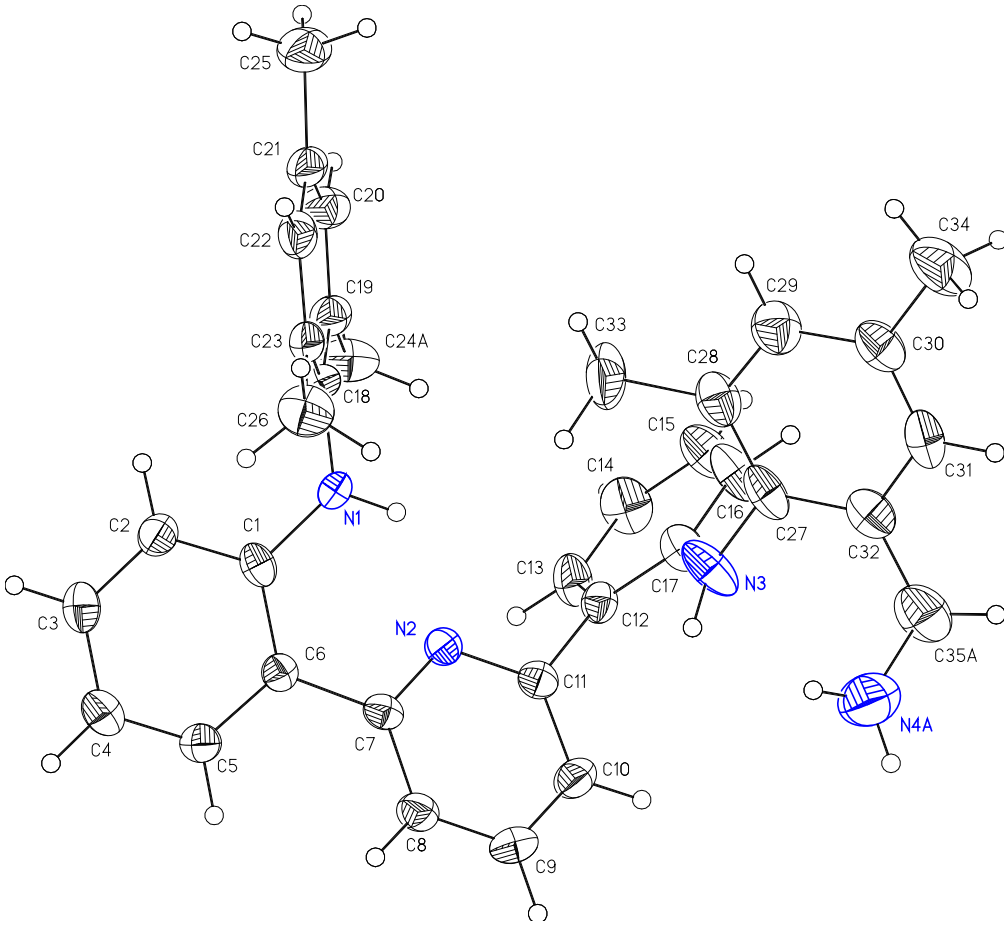
Special Refinement Details

Crystals were mounted on a glass fiber using Paratone oil then placed on the diffractometer under a nitrogen stream at 100K.

The sample appears to be multiple crystals with at least four discernable domains. The model presented here is the result of refinement against the major domain accounting for approximately 65% of the diffraction. Further problems rise from disorder in the NH_2 where it occupies two chemically equivalent positions.

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

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.


C25
C21
C20
C22
C19
C23
C24A
C18
C26
N1
C2
C1
C3
C6
N2
C11
C4
C5
C7
C10
C8
C9
C34
C29
C33
C28
C30
C15
C31
C14
C16
C27
C32
C13
C17
N3
C12
C35A
N4A

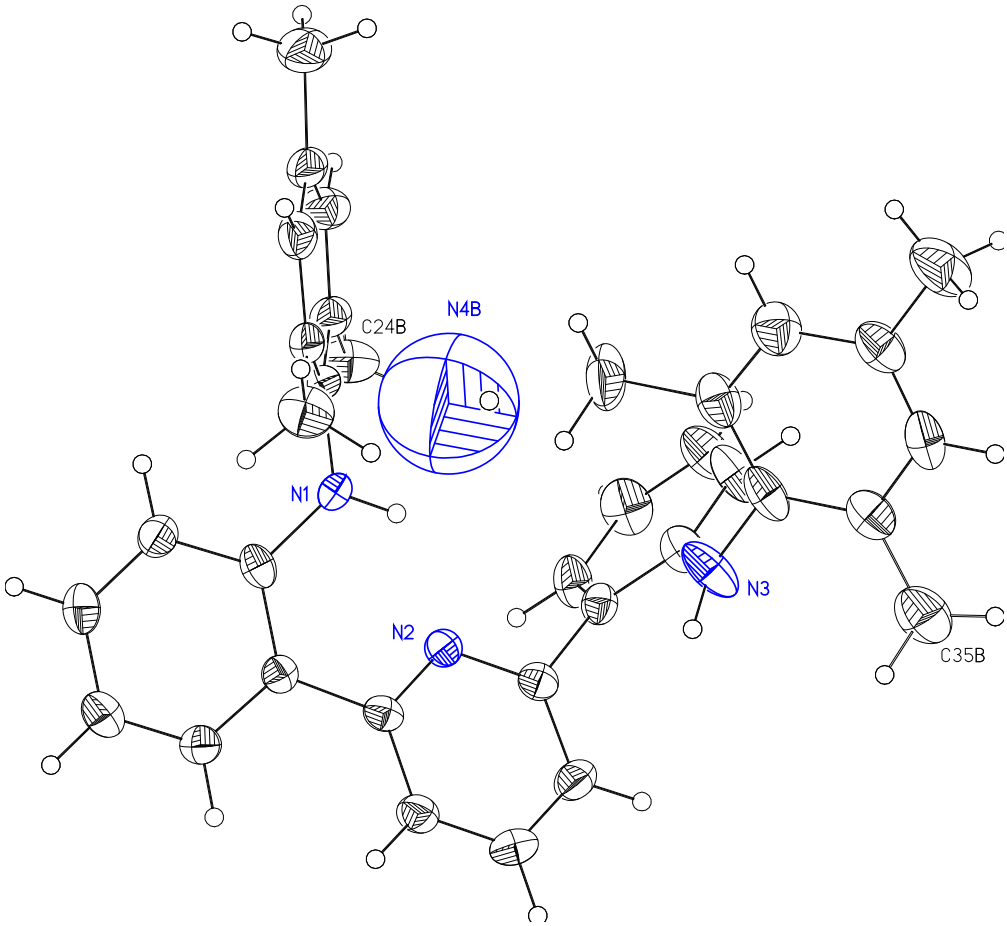

C24B
N4B
N1
N2
N3
C35B

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for ECW13 (CCDC 793154). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}	Occ
N(1)	4914(1)	2590(2)	1735(1)	29(1)	1
N(2)	3702(2)	5238(2)	1848(1)	24(1)	1
N(3)	1028(2)	3989(2)	1295(1)	45(1)	1
N(4A)	-1033(2)	4282(3)	1739(1)	70(1)	0.806(3)
N(4B)	4307(8)	3930(30)	755(10)	342(19)	0.194(3)
C(1)	5519(2)	3083(3)	2158(1)	26(1)	1
C(2)	6529(2)	2361(3)	2303(1)	31(1)	1
C(3)	7121(2)	2779(3)	2732(1)	33(1)	1
C(4)	6724(2)	3943(3)	3030(1)	34(1)	1
C(5)	5744(2)	4688(3)	2885(1)	29(1)	1
C(6)	5120(2)	4304(3)	2455(1)	23(1)	1
C(7)	4066(2)	5162(3)	2326(1)	24(1)	1
C(8)	3471(2)	5867(3)	2682(1)	29(1)	1
C(9)	2507(2)	6667(3)	2542(1)	35(1)	1
C(10)	2134(2)	6741(3)	2053(1)	33(1)	1
C(11)	2745(2)	6014(3)	1717(1)	25(1)	1
C(12)	2400(2)	6030(3)	1182(1)	26(1)	1
C(13)	2929(2)	6999(3)	869(1)	36(1)	1
C(14)	2646(2)	6980(3)	371(1)	44(1)	1
C(15)	1839(2)	5963(3)	180(1)	44(1)	1
C(16)	1302(2)	4985(3)	484(1)	43(1)	1
C(17)	1568(2)	5007(3)	985(1)	32(1)	1
C(18)	5221(2)	1324(3)	1428(1)	28(1)	1
C(19)	5439(2)	1699(3)	954(1)	31(1)	1
C(20)	5673(2)	450(3)	637(1)	39(1)	1
C(21)	5724(2)	-1116(3)	792(1)	34(1)	1
C(22)	5516(2)	-1440(3)	1268(1)	35(1)	1
C(23)	5241(2)	-258(3)	1590(1)	30(1)	1
C(24A)	5404(2)	3412(3)	784(1)	46(1)	0.806(3)
C(24B)	5404(2)	3412(3)	784(1)	46(1)	0.194(3)
C(25)	5944(2)	-2455(3)	441(1)	49(1)	1
C(26)	4916(2)	-707(3)	2088(1)	46(1)	1
C(27)	479(2)	2600(3)	1102(1)	37(1)	1
C(28)	1088(2)	1328(3)	939(1)	41(1)	1
C(29)	519(2)	6(3)	752(1)	45(1)	1
C(30)	-631(2)	-92(3)	723(1)	42(1)	1
C(31)	-1204(2)	1181(3)	892(1)	41(1)	1
C(32)	-680(2)	2543(3)	1080(1)	36(1)	1
C(33)	2336(2)	1383(3)	960(1)	59(1)	1
C(34)	-1220(2)	-1561(3)	521(1)	63(1)	1
C(35A)	-1332(2)	3949(3)	1246(1)	54(1)	0.806(3)
C(35B)	-1332(2)	3949(3)	1246(1)	54(1)	0.194(3)

Table 3. Bond lengths [Å] and angles [°] for ECW13 (CCDC 793154).

N(1)-C(1)	1.385(2)	C(3)-C(2)-C(1)	121.4(2)
N(1)-C(18)	1.423(3)	C(2)-C(3)-C(4)	120.1(2)
N(2)-C(7)	1.351(3)	C(5)-C(4)-C(3)	119.0(2)
N(2)-C(11)	1.356(2)	C(4)-C(5)-C(6)	122.8(2)
N(3)-C(17)	1.404(3)	C(5)-C(6)-C(1)	117.8(2)
N(3)-C(27)	1.421(3)	C(5)-C(6)-C(7)	119.4(2)
N(4A)-C(35A)	1.400(3)	C(1)-C(6)-C(7)	122.8(2)
N(4B)-C(24B)	1.400(3)	N(2)-C(7)-C(8)	120.8(2)
C(1)-C(2)	1.398(3)	N(2)-C(7)-C(6)	117.4(2)
C(1)-C(6)	1.419(3)	C(8)-C(7)-C(6)	121.8(2)
C(2)-C(3)	1.373(3)	C(9)-C(8)-C(7)	119.4(2)
C(3)-C(4)	1.385(3)	C(8)-C(9)-C(10)	119.7(2)
C(4)-C(5)	1.375(3)	C(11)-C(10)-C(9)	118.7(2)
C(5)-C(6)	1.387(3)	N(2)-C(11)-C(10)	122.4(2)
C(6)-C(7)	1.486(3)	N(2)-C(11)-C(12)	115.4(2)
C(7)-C(8)	1.396(3)	C(10)-C(11)-C(12)	122.2(2)
C(8)-C(9)	1.378(3)	C(13)-C(12)-C(17)	118.6(2)
C(9)-C(10)	1.382(3)	C(13)-C(12)-C(11)	120.7(2)
C(10)-C(11)	1.375(3)	C(17)-C(12)-C(11)	120.6(2)
C(11)-C(12)	1.493(3)	C(12)-C(13)-C(14)	121.3(2)
C(12)-C(13)	1.380(3)	C(15)-C(14)-C(13)	119.7(2)
C(12)-C(17)	1.400(3)	C(14)-C(15)-C(16)	120.1(2)
C(13)-C(14)	1.383(3)	C(15)-C(16)-C(17)	120.8(2)
C(14)-C(15)	1.370(3)	C(16)-C(17)-C(12)	119.5(2)
C(15)-C(16)	1.373(3)	C(16)-C(17)-N(3)	120.6(2)
C(16)-C(17)	1.388(3)	C(12)-C(17)-N(3)	119.9(2)
C(18)-C(19)	1.386(3)	C(19)-C(18)-C(23)	121.0(2)
C(18)-C(23)	1.396(3)	C(19)-C(18)-N(1)	118.0(2)
C(19)-C(20)	1.404(3)	C(23)-C(18)-N(1)	121.0(2)
C(19)-C(24A)	1.506(3)	C(18)-C(19)-C(20)	118.6(2)
C(20)-C(21)	1.377(3)	C(18)-C(19)-C(24A)	120.1(2)
C(21)-C(22)	1.377(3)	C(20)-C(19)-C(24A)	121.3(2)
C(21)-C(25)	1.517(3)	C(21)-C(20)-C(19)	121.4(2)
C(22)-C(23)	1.387(3)	C(22)-C(21)-C(20)	118.3(2)
C(23)-C(26)	1.505(3)	C(22)-C(21)-C(25)	120.9(2)
C(27)-C(28)	1.392(3)	C(20)-C(21)-C(25)	120.7(2)
C(27)-C(32)	1.409(3)	C(21)-C(22)-C(23)	122.5(2)
C(28)-C(29)	1.381(3)	C(22)-C(23)-C(18)	118.1(2)
C(28)-C(33)	1.517(3)	C(22)-C(23)-C(26)	119.9(2)
C(29)-C(30)	1.398(3)	C(18)-C(23)-C(26)	121.9(2)
C(30)-C(31)	1.375(3)	N(4B)-C(24B)-C(19)	108(1)
C(30)-C(34)	1.506(3)	C(28)-C(27)-C(32)	121.3(2)
C(31)-C(32)	1.385(3)	C(28)-C(27)-N(3)	119.9(3)
C(32)-C(35A)	1.510(3)	C(32)-C(27)-N(3)	118.8(3)
		C(29)-C(28)-C(27)	117.9(3)
C(1)-N(1)-C(18)	124.8(2)	C(29)-C(28)-C(33)	120.6(3)
C(7)-N(2)-C(11)	119.0(2)	C(27)-C(28)-C(33)	121.5(2)
C(17)-N(3)-C(27)	120.0(2)	C(28)-C(29)-C(30)	122.4(3)
N(1)-C(1)-C(2)	120.2(2)	C(31)-C(30)-C(29)	118.1(3)
N(1)-C(1)-C(6)	120.9(2)	C(31)-C(30)-C(34)	121.0(3)
C(2)-C(1)-C(6)	118.9(2)	C(29)-C(30)-C(34)	120.8(3)

C(30)-C(31)-C(32)	122.1(3)
C(31)-C(32)-C(27)	118.1(3)
C(31)-C(32)-C(35A)	121.1(3)
C(27)-C(32)-C(35A)	120.8(3)
N(4A)-C(35A)-C(32)	110.2(2)

Table 5. Hydrogen bonds for ECW13 (CCDC 793154) [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1)...N(2)	0.88	2.02	2.695(2)	132.2

CALIFORNIA INSTITUTE OF TECHNOLOGY

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X-RAY CRYSTALLOGRAPHY LABORATORY

Date 13 September 2010

Crystal Structure Analysis of:

[^{tBu}NNN]H_2 (ECW18)

(shown below)

For Investigator: Edward Weintrob ext. 6576

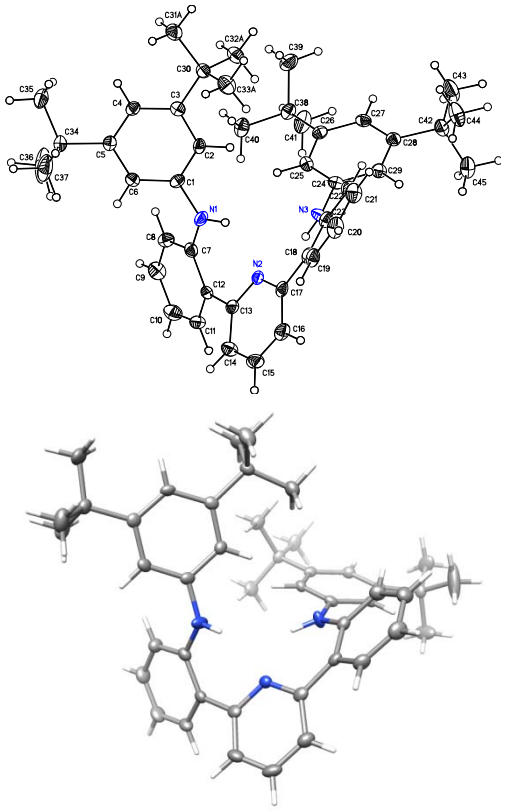
Advisor: J. E. Bercaw ext. 6577

Account Number: JEB.ENERGY-1.02-GRANT.MOOREJCP

By Michael W. Day 116 Beckman ext. 2734

e-mail: mikeday@caltech.edu

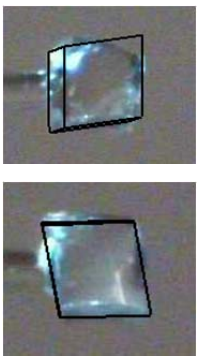
Contents



ECW18

Note: The crystallographic data have been deposited in the Cambridge Database (CCDC) and has been placed on hold pending further instructions from me. The deposition number is 793155. Ideally the CCDC would like the publication to contain a footnote of the type: "Crystallographic data have been deposited at the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK and copies can be obtained on request, free of charge, by quoting the publication citation and the deposition number 793155."

Table 1. Crystal data and structure refinement for ECW18 (CCDC 793155).



Empirical formula	$C_{45}H_{55}N_3 \bullet C_6H_{12}$
Formula weight	722.08
Crystallization Solvent	Hexanes
Crystal Habit	Block
Crystal size	0.19 x 0.17 x 0.16 mm^3
Crystal color	Colorless

Data Collection

Type of diffractometer	Bruker KAPPA APEX II	
Wavelength	0.71073 Å MoKα	
Data Collection Temperature	100(2) K	
θ range for 9933 reflections used in lattice determination	2.39 to 25.70°	
Unit cell dimensions	a = 13.2349(5) Å b = 17.6560(8) Å c = 19.0978(8) Å	α= 90° β= 102.355(2)° γ = 90°
Volume	4359.3(3) $Å^3$	
Z	4	
Crystal system	Monoclinic	
Space group	P $2_1/c$	
Density (calculated)	1.100 Mg/m^3	
F(000)	1576	
θ range for data collection	1.95 to 27.52°	
Completeness to θ = 27.52°	99.8 %	
Index ranges	$-17 \le h \le 16$, $-22 \le k \le 22$, $-24 \le l \le 24$	
Data collection scan type	ω scans; 8 settings	
Reflections collected	63493	
Independent reflections	10009 [R_{int}= 0.0554]	
Absorption coefficient	0.063 mm^{-1}	
Absorption correction	None	
Max. and min. transmission	0.9900 and 0.9881	

Table 1 (cont.)

Structure solution and Refinement

Structure solution program	SHELXS-97 (Sheldrick, 2008)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	SHELXL-97 (Sheldrick, 2008)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	10009 / 0 / 537
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	2.513
Final R indices [$I>2\sigma(I)$, 6629 reflections]	R1 = 0.0641, *w*R2 = 0.0867
R indices (all data)	R1 = 0.0986, *w*R2 = 0.0886
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.000
Average shift/error	0.000
Largest diff. peak and hole	1.093 and -0.828 e.Å^{-3}

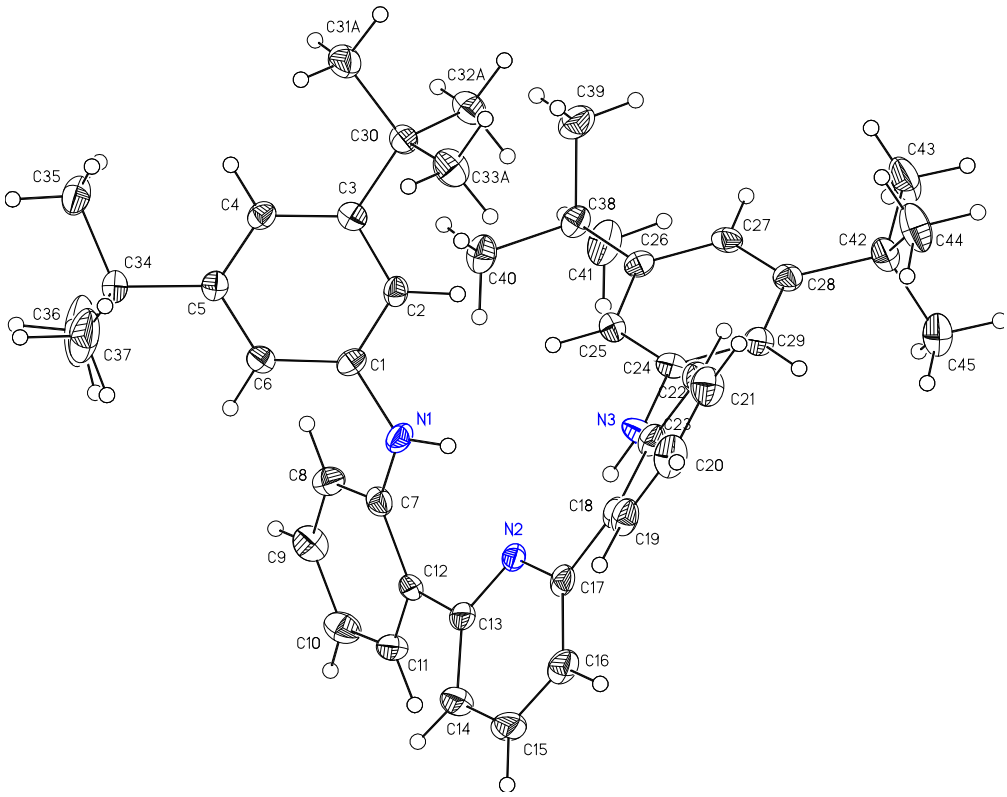
Special Refinement Details

Crystals were mounted on a glass fiber using Paratone oil then placed on the diffractometer under a nitrogen stream at 100K.

One of the t-butyl groups is disordered by rotation around the tertiary C-C bond as is common for t-butyl groups. Additional disorder occurs in the solvent of crystallization, methylcylcopentane, with the methyl group disordered over two carbon sites.

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

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.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C26
C28
C36
C37
C6
C1
C25
C24
C29
C45
C22
C21
N1
N3
C23
C20
C8
C7
C18
C19
N2
C9
C12
C17
C13
C10
C11
C16
C14
C15

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for ECW18 (CCDC 793155). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}	Occ
N(1)	3974(1)	8271(1)	4141(1)	22(1)	1
N(2)	3363(1)	9602(1)	4650(1)	19(1)	1
N(3)	2370(1)	8733(1)	5492(1)	25(1)	1
C(1)	4677(1)	7661(1)	4308(1)	18(1)	1
C(2)	4719(1)	7273(1)	4946(1)	18(1)	1
C(3)	5436(2)	6695(1)	5167(1)	18(1)	1
C(4)	6113(1)	6524(1)	4722(1)	19(1)	1
C(5)	6104(2)	6910(1)	4085(1)	18(1)	1
C(6)	5372(1)	7482(1)	3879(1)	18(1)	1
C(7)	3521(2)	8552(1)	3468(1)	20(1)	1
C(8)	3363(2)	8084(1)	2864(1)	24(1)	1
C(9)	2885(2)	8353(1)	2194(1)	28(1)	1
C(10)	2544(2)	9099(1)	2112(1)	29(1)	1
C(11)	2696(2)	9559(1)	2705(1)	25(1)	1
C(12)	3192(2)	9318(1)	3388(1)	19(1)	1
C(13)	3352(2)	9866(1)	3987(1)	21(1)	1
C(14)	3500(2)	10632(1)	3869(1)	27(1)	1
C(15)	3600(2)	11128(1)	4437(1)	31(1)	1
C(16)	3585(2)	10860(1)	5114(1)	27(1)	1
C(17)	3496(2)	10079(1)	5214(1)	21(1)	1
C(18)	3558(2)	9757(1)	5939(1)	20(1)	1
C(19)	4210(2)	10096(1)	6522(1)	26(1)	1
C(20)	4336(2)	9830(1)	7213(1)	30(1)	1
C(21)	3793(2)	9191(1)	7334(1)	30(1)	1
C(22)	3141(2)	8833(1)	6771(1)	26(1)	1
C(23)	3011(2)	9095(1)	6067(1)	22(1)	1
C(24)	1772(2)	8074(1)	5509(1)	19(1)	1
C(25)	1853(1)	7499(1)	5024(1)	19(1)	1
C(26)	1253(1)	6847(1)	4986(1)	18(1)	1
C(27)	575(1)	6789(1)	5453(1)	19(1)	1
C(28)	470(2)	7356(1)	5936(1)	20(1)	1
C(29)	1081(2)	8007(1)	5957(1)	21(1)	1
C(30)	5452(2)	6275(1)	5869(1)	20(1)	1
C(31A)	6273(2)	5639(2)	6012(1)	30(1)	0.849(3)
C(32A)	4393(2)	5904(2)	5835(1)	27(1)	0.849(3)
C(33A)	5693(2)	6833(2)	6488(1)	31(1)	0.849(3)
C(31B)	6545(11)	6330(10)	6405(8)	44(5)	0.151(3)
C(32B)	5142(13)	5441(9)	5736(8)	43(5)	0.151(3)
C(33B)	4651(12)	6629(9)	6323(8)	39(5)	0.151(3)
C(34)	6925(2)	6765(1)	3642(1)	22(1)	1
C(35)	7413(2)	5985(1)	3780(1)	44(1)	1
C(36)	6456(2)	6806(2)	2840(1)	79(1)	1
C(37)	7762(2)	7364(1)	3841(2)	59(1)	1
C(38)	1318(2)	6211(1)	4453(1)	21(1)	1
C(39)	1588(2)	5459(1)	4857(1)	32(1)	1
C(40)	2134(1)	6362(1)	4005(1)	29(1)	1
C(41)	260(1)	6124(1)	3931(1)	34(1)	1

C(42)	-307(2)	7315(1)	6430(1)	25(1)	1
C(43)	-857(2)	6548(1)	6377(1)	42(1)	1
C(44)	250(2)	7437(1)	7213(1)	40(1)	1
C(45)	-1133(2)	7936(1)	6206(1)	42(1)	1
C(51)	1385(2)	4424(1)	1794(1)	45(1)	1
C(52)	1090(2)	4984(1)	2341(1)	49(1)	1
C(53)	-141(2)	4988(2)	2108(2)	82(1)	1
C(54)	-426(2)	4637(2)	1296(2)	69(1)	1
C(55)	633(2)	4593(2)	1085(1)	45(1)	1
C(56A)	639(2)	4039(2)	498(1)	50(1)	0.886(3)
C(56B)	-745(12)	3769(10)	1281(10)	21(5)	0.114(3)

Table 3. Bond lengths [Å] and angles [°] for ECW18 (CCDC 793155).

N(1)-C(7)	1.388(2)	C(38)-C(41)	1.543(3)
N(1)-C(1)	1.414(2)	C(42)-C(43)	1.530(3)
N(2)-C(13)	1.348(2)	C(42)-C(44)	1.535(3)
N(2)-C(17)	1.350(2)	C(42)-C(45)	1.544(3)
N(3)-C(23)	1.391(2)	C(51)-C(55)	1.529(3)
N(3)-C(24)	1.413(2)	C(51)-C(52)	1.548(3)
C(1)-C(2)	1.389(3)	C(52)-C(53)	1.595(3)
C(1)-C(6)	1.392(2)	C(53)-C(54)	1.638(4)
C(2)-C(3)	1.396(3)	C(54)-C(56B)	1.590(17)
C(3)-C(4)	1.393(3)	C(54)-C(55)	1.541(3)
C(3)-C(30)	1.529(3)	C(55)-C(56A)	1.489(3)
C(4)-C(5)	1.391(3)		
C(5)-C(6)	1.397(3)	C(7)-N(1)-C(1)	127.91(18)
C(5)-C(34)	1.534(3)	C(13)-N(2)-C(17)	120.47(19)
C(7)-C(8)	1.398(3)	C(23)-N(3)-C(24)	127.63(18)
C(7)-C(12)	1.417(3)	C(2)-C(1)-C(6)	119.75(19)
C(8)-C(9)	1.386(3)	C(2)-C(1)-N(1)	118.37(18)
C(9)-C(10)	1.390(3)	C(6)-C(1)-N(1)	121.71(19)
C(10)-C(11)	1.373(3)	C(1)-C(2)-C(3)	121.53(19)
C(11)-C(12)	1.396(3)	C(4)-C(3)-C(2)	117.3(2)
C(12)-C(13)	1.479(3)	C(4)-C(3)-C(30)	122.72(19)
C(13)-C(14)	1.391(3)	C(2)-C(3)-C(30)	120.00(19)
C(14)-C(15)	1.379(3)	C(5)-C(4)-C(3)	122.7(2)
C(15)-C(16)	1.381(3)	C(4)-C(5)-C(6)	118.43(19)
C(16)-C(17)	1.399(3)	C(4)-C(5)-C(34)	121.66(19)
C(17)-C(18)	1.483(3)	C(6)-C(5)-C(34)	119.74(19)
C(18)-C(19)	1.390(3)	C(1)-C(6)-C(5)	120.3(2)
C(18)-C(23)	1.422(3)	N(1)-C(7)-C(8)	120.7(2)
C(19)-C(20)	1.376(3)	N(1)-C(7)-C(12)	120.2(2)
C(20)-C(21)	1.384(3)	C(8)-C(7)-C(12)	119.1(2)
C(21)-C(22)	1.381(3)	C(9)-C(8)-C(7)	121.1(2)
C(22)-C(23)	1.398(3)	C(8)-C(9)-C(10)	120.2(2)
C(24)-C(29)	1.385(3)	C(11)-C(10)-C(9)	118.7(2)
C(24)-C(25)	1.393(3)	C(10)-C(11)-C(12)	123.2(2)
C(25)-C(26)	1.393(3)	C(11)-C(12)-C(7)	117.7(2)
C(26)-C(27)	1.397(3)	C(11)-C(12)-C(13)	118.93(19)
C(26)-C(38)	1.530(3)	C(7)-C(12)-C(13)	123.4(2)
C(27)-C(28)	1.388(3)	N(2)-C(13)-C(14)	121.0(2)
C(28)-C(29)	1.399(3)	N(2)-C(13)-C(12)	118.10(19)
C(28)-C(42)	1.539(3)	C(14)-C(13)-C(12)	120.9(2)
C(30)-C(33A)	1.520(3)	C(15)-C(14)-C(13)	118.9(2)
C(30)-C(32B)	1.536(15)	C(14)-C(15)-C(16)	120.0(2)
C(30)-C(32A)	1.536(3)	C(15)-C(16)-C(17)	119.0(2)
C(30)-C(31A)	1.546(3)	N(2)-C(17)-C(16)	120.4(2)
C(30)-C(31B)	1.586(15)	N(2)-C(17)-C(18)	118.45(19)
C(30)-C(33B)	1.631(14)	C(16)-C(17)-C(18)	121.1(2)
C(34)-C(35)	1.520(3)	C(19)-C(18)-C(23)	117.9(2)
C(34)-C(37)	1.520(3)	C(19)-C(18)-C(17)	118.87(19)
C(34)-C(36)	1.527(3)	C(23)-C(18)-C(17)	123.18(19)
C(38)-C(40)	1.538(2)	C(20)-C(19)-C(18)	123.2(2)
C(38)-C(39)	1.538(3)	C(19)-C(20)-C(21)	118.6(2)

C(22)-C(21)-C(20)	120.2(2)	C(44)-C(42)-C(45)	109.46(19)
C(21)-C(22)-C(23)	121.7(2)	C(28)-C(42)-C(45)	108.84(18)
N(3)-C(23)-C(22)	122.41(19)	C(55)-C(51)-C(52)	105.2(2)
N(3)-C(23)-C(18)	119.2(2)	C(51)-C(52)-C(53)	101.8(2)
C(22)-C(23)-C(18)	118.3(2)	C(52)-C(53)-C(54)	106.1(2)
C(29)-C(24)-C(25)	120.24(19)	C(56B)-C(54)-C(55)	101.5(6)
C(29)-C(24)-N(3)	122.17(19)	C(56B)-C(54)-C(53)	112.8(7)
C(25)-C(24)-N(3)	117.49(18)	C(55)-C(54)-C(53)	103.2(2)
C(26)-C(25)-C(24)	120.82(19)	C(56A)-C(55)-C(51)	115.6(2)
C(25)-C(26)-C(27)	117.6(2)	C(56A)-C(55)-C(54)	112.4(2)
C(25)-C(26)-C(38)	121.92(19)	C(51)-C(55)-C(54)	103.5(2)
C(27)-C(26)-C(38)	120.45(19)		
C(28)-C(27)-C(26)	122.7(2)		
C(27)-C(28)-C(29)	118.16(19)		
C(27)-C(28)-C(42)	123.18(19)		
C(29)-C(28)-C(42)	118.63(19)		
C(24)-C(29)-C(28)	120.4(2)		
C(33A)-C(30)-C(3)	109.25(19)		
C(33A)-C(30)-C(32B)	139.0(6)		
C(3)-C(30)-C(32B)	111.5(6)		
C(33A)-C(30)-C(32A)	110.32(19)		
C(3)-C(30)-C(32A)	109.19(18)		
C(32B)-C(30)-C(32A)	51.3(6)		
C(33A)-C(30)-C(31A)	108.1(2)		
C(3)-C(30)-C(31A)	112.62(18)		
C(32B)-C(30)-C(31A)	59.0(6)		
C(32A)-C(30)-C(31A)	107.4(2)		
C(33A)-C(30)-C(31B)	56.5(6)		
C(3)-C(30)-C(31B)	112.1(6)		
C(32B)-C(30)-C(31B)	109.9(9)		
C(32A)-C(30)-C(31B)	138.7(6)		
C(31A)-C(30)-C(31B)	54.6(6)		
C(33A)-C(30)-C(33B)	52.4(6)		
C(3)-C(30)-C(33B)	113.1(5)		
C(32B)-C(30)-C(33B)	105.9(8)		
C(32A)-C(30)-C(33B)	59.7(6)		
C(31A)-C(30)-C(33B)	134.1(5)		
C(31B)-C(30)-C(33B)	103.9(8)		
C(35)-C(34)-C(37)	109.08(18)		
C(35)-C(34)-C(36)	106.4(2)		
C(37)-C(34)-C(36)	109.6(2)		
C(35)-C(34)-C(5)	112.41(18)		
C(37)-C(34)-C(5)	108.10(18)		
C(36)-C(34)-C(5)	111.23(18)		
C(26)-C(38)-C(40)	112.91(17)		
C(26)-C(38)-C(39)	109.86(17)		
C(40)-C(38)-C(39)	108.26(17)		
C(26)-C(38)-C(41)	109.30(17)		
C(40)-C(38)-C(41)	107.81(18)		
C(39)-C(38)-C(41)	108.60(17)		
C(43)-C(42)-C(44)	108.1(2)		
C(43)-C(42)-C(28)	111.93(19)		
C(44)-C(42)-C(28)	110.26(17)		
C(43)-C(42)-C(45)	108.19(18)		

Table 5. Hydrogen bonds for ECW18 (CCDC 793155) [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1)...N(2)	0.88	2.06	2.730(2)	131.7
N(3)-H(3)...N(2)	0.88	2.08	2.750(2)	132.3

CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE

X-RAY CRYSTALLOGRAPHY LABORATORY

Date 14 September 2010

Crystal Structure Analysis of:

[^{tBu}NNN]H_2 (ECW19)

(shown below)

For Investigator: Edward Weintrob ext. 6576

Advisor: J. E. Bercaw ext. 6577

Account Number: JEB.ENERGY-1.02-GRANT.MOOREJCP

By Michael W. Day 116 Beckman ext. 2734

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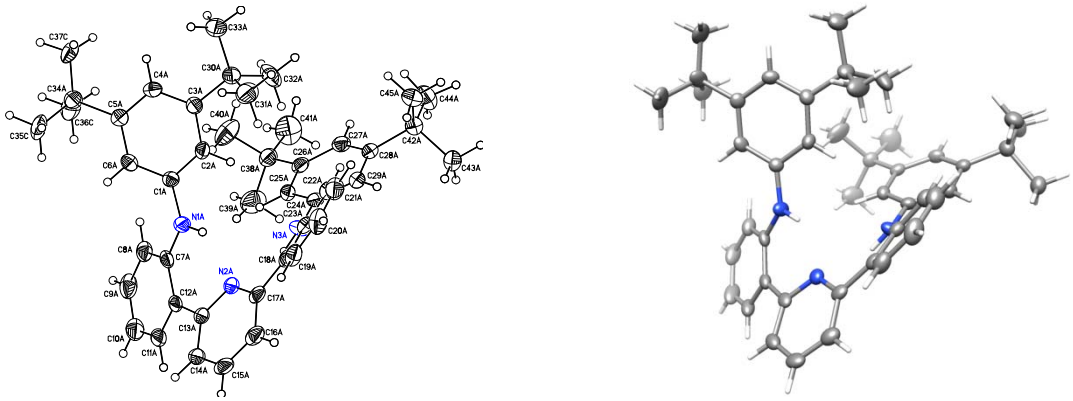
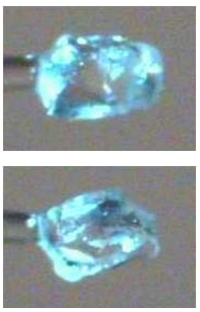
Contents



ECW19

Note: The crystallographic data have been deposited in the Cambridge Database (CCDC) and has been placed on hold pending further instructions from me. The deposition number is 793156. Ideally the CCDC would like the publication to contain a footnote of the type: "Crystallographic data have been deposited at the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK and copies can be obtained on request, free of charge, by quoting the publication citation and the deposition number 793156."

Table 1. Crystal data and structure refinement for ECW19 (CCDC 793156).



Empirical formula	$C_{45}H_{55}N_3$
Formula weight	637.92
Crystallization Solvent	Acetonitrile
Crystal Habit	Fragment
Crystal size	0.23 x 0.16 x 0.15 mm^3
Crystal color	Colorless

Data Collection

Type of diffractometer	Bruker KAPPA APEX II	
Wavelength	0.71073 Å MoKα	
Data Collection Temperature	100(2) K	
θ range for 9914 reflections used in lattice determination	2.19 to 24.21°	
Unit cell dimensions	a = 14.7469(5) Å b = 17.4293(6) Å c = 18.0411(7) Å	α= 64.238(2)° β= 76.593(2)° γ = 66.808(2)°
Volume	3828.4(2) $Å^3$	
Z	4	
Crystal system	Triclinic	
Space group	P-1	
Density (calculated)	1.107 Mg/m^3	
F(000)	1384	
θ range for data collection	1.89 to 27.51°	
Completeness to θ = 27.51°	93.4 %	
Index ranges	$-17 \leq h \leq 18, -22 \leq k \leq 22, -23 , l \leq 22$	
Data collection scan type	ω scans; 11 settings	
Reflections collected	75730	
Independent reflections	16451 [R_{int}= 0.0496]	
Absorption coefficient	0.064 mm^{-1}	
Absorption correction	None	
Max. and min. transmission	0.9905 and 0.9855	

Table 1 (cont.)

Structure solution and Refinement

Structure solution program	SHELXS-97 (Sheldrick, 2008)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	SHELXL-97 (Sheldrick, 2008)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	16451 / 0 / 976
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	1.817
Final R indices [$I>2\sigma(I)$, 9925 reflections]	R1 = 0.0528, *w*R2 = 0.0721
R indices (all data)	R1 = 0.0981, *w*R2 = 0.0743
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.001
Average shift/error	0.000
Largest diff. peak and hole	0.443 and -0.396 e.Å^{-3}

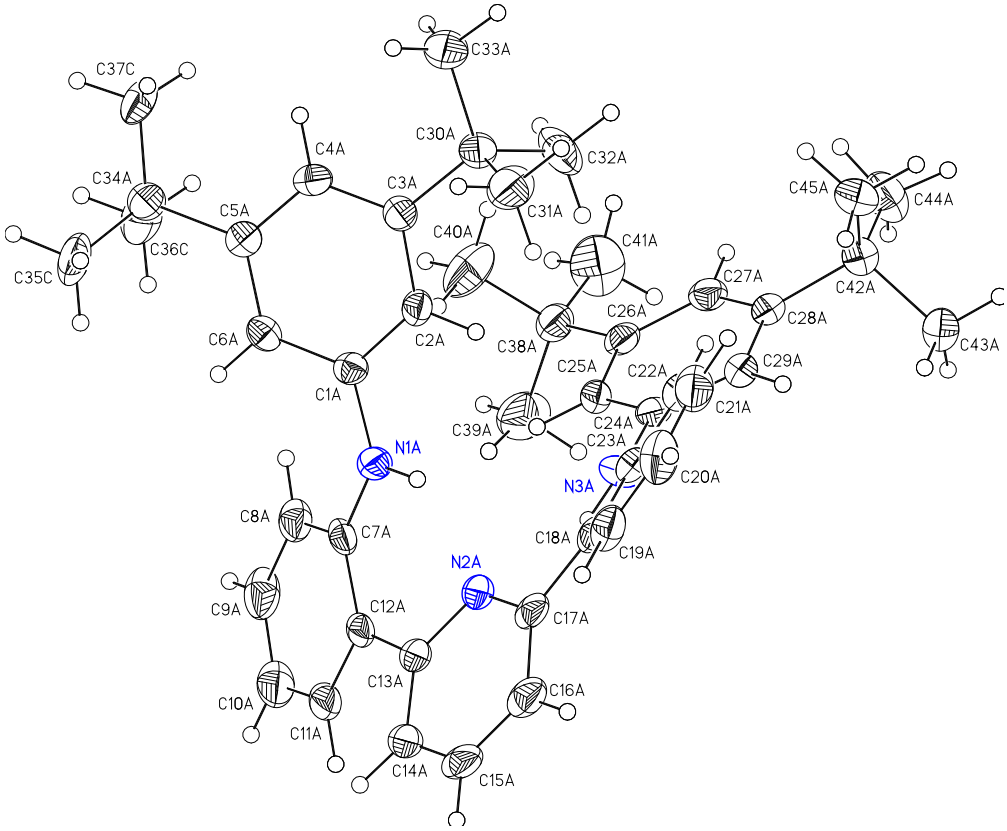
Special Refinement Details

Crystals were mounted on a glass fiber using Paratone oil then placed on the diffractometer under a nitrogen stream at 100K.

There are two molecules in the asymmetric unit and each contains disorder common to t-butyl groups. A fully labeled figure is shown for molecule A only and the overlap of A and B is shown

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

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.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C31A
C45A
C44A
C5A
C40A
C41A
C27A
C36C
C35C
C42A
C26A
C28A
C6A
C2A
C38A
C43A
C25A
C22A
C29A
C1A
C24A
C21A
C39A
C23A
N1A
C20A
N3A
C8A
C7A
C18A
C19A
N2A
C9A
C12A
C17A
C13A
C16A
C10A
C11A
C14A
C15A

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for ECW19 (CCDC 793156). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}	Occ
N(1A)	2376(1)	1612(1)	5813(1)	28(1)	1
N(2A)	3767(1)	32(1)	5856(1)	27(1)	1
N(3A)	4586(1)	322(1)	6898(1)	31(1)	1
C(1A)	1694(1)	2044(1)	6316(1)	24(1)	1
C(2A)	1932(1)	1728(1)	7122(1)	27(1)	1
C(3A)	1297(1)	2059(1)	7692(1)	26(1)	1
C(4A)	388(1)	2716(1)	7442(1)	27(1)	1
C(5A)	113(1)	3042(1)	6638(1)	26(1)	1
C(6A)	781(1)	2704(1)	6079(1)	25(1)	1
C(7A)	2550(1)	1907(1)	4962(1)	27(1)	1
C(8A)	2210(1)	2824(1)	4459(1)	34(1)	1
C(9A)	2368(1)	3112(1)	3612(1)	45(1)	1
C(10A)	2876(1)	2494(2)	3247(1)	50(1)	1
C(11A)	3248(1)	1592(1)	3738(1)	40(1)	1
C(12A)	3112(1)	1270(1)	4594(1)	28(1)	1
C(13A)	3566(1)	289(1)	5071(1)	29(1)	1
C(14A)	3797(1)	-365(1)	4740(1)	36(1)	1
C(15A)	4264(1)	-1261(1)	5206(1)	40(1)	1
C(16A)	4484(1)	-1509(1)	5998(1)	37(1)	1
C(17A)	4200(1)	-847(1)	6323(1)	29(1)	1
C(18A)	4317(1)	-1074(1)	7200(1)	28(1)	1
C(19A)	4229(1)	-1893(1)	7789(1)	37(1)	1
C(20A)	4273(1)	-2146(1)	8621(1)	44(1)	1
C(21A)	4376(1)	-1553(1)	8881(1)	41(1)	1
C(22A)	4468(1)	-733(1)	8318(1)	33(1)	1
C(23A)	4462(1)	-484(1)	7474(1)	27(1)	1
C(24A)	4744(1)	1004(1)	7023(1)	27(1)	1
C(25A)	4298(1)	1897(1)	6510(1)	28(1)	1
C(26A)	4411(1)	2615(1)	6587(1)	27(1)	1
C(27A)	4968(1)	2404(1)	7216(1)	30(1)	1
C(28A)	5439(1)	1521(1)	7729(1)	29(1)	1
C(29A)	5343(1)	821(1)	7611(1)	28(1)	1
C(30A)	1626(1)	1692(1)	8566(1)	36(1)	1
C(31A)	1911(1)	651(1)	8948(1)	57(1)	1
C(32A)	2528(1)	1949(1)	8513(1)	68(1)	1
C(33A)	817(1)	2070(1)	9138(1)	44(1)	1
C(34A)	-917(1)	3721(1)	6397(1)	39(1)	1
C(35A)	-1216(3)	4555(3)	6557(3)	40(1)	0.493(2)
C(36A)	-1735(2)	3184(2)	7025(2)	35(1)	0.493(2)
C(37A)	-1199(4)	3928(3)	5556(3)	41(1)	0.493(2)
C(35C)	-1378(3)	3455(3)	5973(3)	50(2)	0.507(2)
C(36C)	-642(3)	4634(2)	5677(2)	46(1)	0.507(2)
C(37C)	-1497(3)	4018(3)	7048(3)	51(1)	0.507(2)
C(38A)	3879(1)	3582(1)	6024(1)	32(1)	1
C(39A)	4132(1)	3696(1)	5116(1)	59(1)	1
C(40A)	2761(1)	3809(1)	6207(1)	59(1)	1
C(41A)	4159(2)	4289(1)	6125(1)	65(1)	1

C(42A)	6008(1)	1300(2)	8446(1)	38(1)	1
C(43A)	7066(3)	584(2)	8385(2)	40(1)	0.625(4)
C(44A)	6117(3)	2052(3)	8514(3)	50(1)	0.625(4)
C(45A)	5491(2)	758(3)	9284(2)	44(1)	0.625(4)
C(43C)	5181(4)	1729(5)	9038(3)	64(3)	0.375(4)
C(44C)	6562(6)	426(5)	8856(4)	60(2)	0.375(4)
C(45C)	6708(5)	1953(5)	8126(4)	53(2)	0.375(4)
N(1B)	2202(1)	7918(1)	6496(1)	29(1)	1
N(2B)	916(1)	9601(1)	5942(1)	27(1)	1
N(3B)	-349(1)	8848(1)	7182(1)	27(1)	1
C(1B)	2664(1)	7235(1)	7211(1)	26(1)	1
C(2B)	2167(1)	7260(1)	7958(1)	27(1)	1
C(3B)	2566(1)	6646(1)	8704(1)	31(1)	1
C(4B)	3492(1)	6004(1)	8675(1)	38(1)	1
C(5B)	4015(1)	5966(1)	7940(1)	34(1)	1
C(6B)	3595(1)	6590(1)	7204(1)	29(1)	1
C(7B)	2316(1)	7969(1)	5693(1)	28(1)	1
C(8B)	2770(1)	7199(1)	5508(1)	34(1)	1
C(9B)	2908(1)	7261(1)	4705(1)	42(1)	1
C(10B)	2587(1)	8088(1)	4066(1)	43(1)	1
C(11B)	2094(1)	8849(1)	4247(1)	36(1)	1
C(12B)	1934(1)	8825(1)	5047(1)	28(1)	1
C(13B)	1371(1)	9672(1)	5187(1)	27(1)	1
C(14B)	1307(1)	10525(1)	4570(1)	32(1)	1
C(15B)	733(1)	11285(1)	4735(1)	35(1)	1
C(16B)	253(1)	11204(1)	5508(1)	33(1)	1
C(17B)	376(1)	10339(1)	6115(1)	27(1)	1
C(18B)	-21(1)	10184(1)	6986(1)	27(1)	1
C(19B)	-44(1)	10777(1)	7319(1)	35(1)	1
C(20B)	-321(1)	10650(1)	8135(1)	43(1)	1
C(21B)	-564(1)	9884(1)	8656(1)	41(1)	1
C(22B)	-561(1)	9282(1)	8342(1)	33(1)	1
C(23B)	-317(1)	9425(1)	7514(1)	26(1)	1
C(24B)	-590(1)	8044(1)	7602(1)	24(1)	1
C(25B)	-8(1)	7308(1)	7394(1)	23(1)	1
C(26B)	-198(1)	6489(1)	7774(1)	23(1)	1
C(27B)	-975(1)	6439(1)	8378(1)	26(1)	1
C(28B)	-1586(1)	7173(1)	8591(1)	24(1)	1
C(29B)	-1396(1)	7987(1)	8176(1)	25(1)	1
C(30B)	5059(2)	5276(1)	7950(1)	45(1)	1
C(31B)	5098(6)	4366(5)	8479(5)	68(2)	0.708(9)
C(32B)	5750(5)	5608(5)	8207(4)	43(1)	0.708(9)
C(33B)	5498(3)	5328(4)	7052(3)	60(2)	0.708(9)
C(31D)	5140(9)	4773(10)	7365(7)	71(4)	0.292(9)
C(32D)	5140(14)	4357(12)	8859(9)	45(4)	0.292(9)
C(33D)	5822(13)	5554(13)	7868(14)	90(7)	0.292(9)
C(34B)	1969(1)	6710(1)	9506(1)	38(1)	1
C(35B)	2534(2)	6055(1)	10267(1)	69(1)	1
C(36B)	1007(1)	6532(1)	9586(1)	51(1)	1
C(37B)	1709(1)	7670(1)	9490(1)	53(1)	1
C(38B)	476(1)	5695(1)	7526(1)	26(1)	1
C(39B)	138(1)	4869(1)	7970(1)	41(1)	1
C(40B)	1534(1)	5420(1)	7745(1)	37(1)	1

C(41B)	479(1)	5977(1)	6593(1)	36(1)	1
C(42B)	-2401(1)	7060(1)	9292(1)	29(1)	1
C(43B)	-3081(1)	6664(1)	9156(1)	54(1)	1
C(44B)	-1906(1)	6412(1)	10104(1)	51(1)	1
C(45B)	-3042(1)	7941(1)	9394(1)	42(1)	1

Table 3. Bond lengths [Å] and angles [°] for ECW19 (CCDC 793156).

N(1A)-C(7A)	1.3864(19)	C(38A)-C(39A)	1.534(2)
N(1A)-C(1A)	1.405(2)	C(42A)-C(44C)	1.362(6)
N(2A)-C(17A)	1.3500(19)	C(42A)-C(44A)	1.439(4)
N(2A)-C(13A)	1.3535(19)	C(42A)-C(43A)	1.584(4)
N(3A)-C(23A)	1.3896(18)	C(42A)-C(45A)	1.598(3)
N(3A)-C(24A)	1.4117(18)	C(42A)-C(43C)	1.600(6)
C(1A)-C(2A)	1.390(2)	C(42A)-C(45C)	1.664(6)
C(1A)-C(6A)	1.393(2)	N(1B)-C(7B)	1.3850(19)
C(2A)-C(3A)	1.382(2)	N(1B)-C(1B)	1.4076(19)
C(3A)-C(4A)	1.392(2)	N(2B)-C(17B)	1.347(2)
C(3A)-C(30A)	1.537(2)	N(2B)-C(13B)	1.3471(19)
C(4A)-C(5A)	1.401(2)	N(3B)-C(23B)	1.3933(18)
C(5A)-C(6A)	1.390(2)	N(3B)-C(24B)	1.4173(17)
C(5A)-C(34A)	1.530(2)	C(1B)-C(2B)	1.387(2)
C(7A)-C(8A)	1.396(2)	C(1B)-C(6B)	1.393(2)
C(7A)-C(12A)	1.421(2)	C(2B)-C(3B)	1.386(2)
C(8A)-C(9A)	1.379(2)	C(3B)-C(4B)	1.391(2)
C(9A)-C(10A)	1.380(3)	C(3B)-C(34B)	1.534(2)
C(10A)-C(11A)	1.376(2)	C(4B)-C(5B)	1.385(2)
C(11A)-C(12A)	1.392(2)	C(5B)-C(6B)	1.388(2)
C(12A)-C(13A)	1.483(2)	C(5B)-C(30B)	1.533(2)
C(13A)-C(14A)	1.401(2)	C(7B)-C(8B)	1.394(2)
C(14A)-C(15A)	1.378(2)	C(7B)-C(12B)	1.426(2)
C(15A)-C(16A)	1.379(2)	C(8B)-C(9B)	1.377(2)
C(16A)-C(17A)	1.394(2)	C(9B)-C(10B)	1.380(2)
C(17A)-C(18A)	1.489(2)	C(10B)-C(11B)	1.378(2)
C(18A)-C(19A)	1.396(2)	C(11B)-C(12B)	1.391(2)
C(18A)-C(23A)	1.419(2)	C(12B)-C(13B)	1.482(2)
C(19A)-C(20A)	1.378(2)	C(13B)-C(14B)	1.399(2)
C(20A)-C(21A)	1.376(2)	C(14B)-C(15B)	1.380(2)
C(21A)-C(22A)	1.386(2)	C(15B)-C(16B)	1.385(2)
C(22A)-C(23A)	1.392(2)	C(16B)-C(17B)	1.395(2)
C(24A)-C(29A)	1.387(2)	C(17B)-C(18B)	1.488(2)
C(24A)-C(25A)	1.392(2)	C(18B)-C(19B)	1.390(2)
C(25A)-C(26A)	1.392(2)	C(18B)-C(23B)	1.419(2)
C(26A)-C(27A)	1.389(2)	C(19B)-C(20B)	1.376(2)
C(26A)-C(38A)	1.526(2)	C(20B)-C(21B)	1.386(2)
C(27A)-C(28A)	1.388(2)	C(21B)-C(22B)	1.390(2)
C(28A)-C(29A)	1.389(2)	C(22B)-C(23B)	1.387(2)
C(28A)-C(42A)	1.530(2)	C(24B)-C(25B)	1.387(2)
C(30A)-C(33A)	1.525(2)	C(24B)-C(29B)	1.389(2)
C(30A)-C(32A)	1.534(2)	C(25B)-C(26B)	1.4015(19)
C(30A)-C(31A)	1.546(2)	C(26B)-C(27B)	1.387(2)
C(34A)-C(35C)	1.436(5)	C(26B)-C(38B)	1.533(2)
C(34A)-C(37C)	1.447(4)	C(27B)-C(28B)	1.398(2)
C(34A)-C(35A)	1.481(4)	C(28B)-C(29B)	1.3946(19)
C(34A)-C(37A)	1.517(5)	C(28B)-C(42B)	1.532(2)
C(34A)-C(36C)	1.688(4)	C(30B)-C(33D)	1.35(2)
C(34A)-C(36A)	1.718(3)	C(30B)-C(31B)	1.437(8)
C(38A)-C(40A)	1.527(2)	C(30B)-C(33B)	1.575(4)
C(38A)-C(41A)	1.533(2)	C(30B)-C(32B)	1.586(8)

C(30B)-C(31D)	1.599(10)
C(30B)-C(32D)	1.711(17)
C(34B)-C(35B)	1.516(2)
C(34B)-C(36B)	1.531(2)
C(34B)-C(37B)	1.550(2)
C(38B)-C(39B)	1.5300(19)
C(38B)-C(40B)	1.534(2)
C(38B)-C(41B)	1.535(2)
C(42B)-C(45B)	1.518(2)
C(42B)-C(43B)	1.533(2)
C(42B)-C(44B)	1.537(2)

C(7A)-N(1A)-C(1A)	130.97(15)
C(17A)-N(2A)-C(13A)	120.85(16)
C(23A)-N(3A)-C(24A)	129.37(14)
C(2A)-C(1A)-C(6A)	118.90(17)
C(2A)-C(1A)-N(1A)	116.36(16)
C(6A)-C(1A)-N(1A)	124.54(16)
C(3A)-C(2A)-C(1A)	122.07(17)
C(2A)-C(3A)-C(4A)	117.96(16)
C(2A)-C(3A)-C(30A)	118.92(16)
C(4A)-C(3A)-C(30A)	123.12(16)
C(3A)-C(4A)-C(5A)	121.75(17)
C(6A)-C(5A)-C(4A)	118.50(16)
C(6A)-C(5A)-C(34A)	120.91(16)
C(4A)-C(5A)-C(34A)	120.53(17)
C(5A)-C(6A)-C(1A)	120.81(16)
N(1A)-C(7A)-C(8A)	121.19(17)
N(1A)-C(7A)-C(12A)	119.64(16)
C(8A)-C(7A)-C(12A)	119.14(17)
C(9A)-C(8A)-C(7A)	120.95(19)
C(8A)-C(9A)-C(10A)	120.3(2)
C(11A)-C(10A)-C(9A)	119.34(19)
C(10A)-C(11A)-C(12A)	122.44(19)
C(11A)-C(12A)-C(7A)	117.74(18)
C(11A)-C(12A)-C(13A)	118.47(17)
C(7A)-C(12A)-C(13A)	123.79(16)
N(2A)-C(13A)-C(14A)	119.82(18)
N(2A)-C(13A)-C(12A)	117.37(16)
C(14A)-C(13A)-C(12A)	122.81(17)
C(15A)-C(14A)-C(13A)	119.54(18)
C(14A)-C(15A)-C(16A)	119.95(18)
C(15A)-C(16A)-C(17A)	118.99(19)
N(2A)-C(17A)-C(16A)	120.72(17)
N(2A)-C(17A)-C(18A)	117.09(16)
C(16A)-C(17A)-C(18A)	122.14(18)
C(19A)-C(18A)-C(23A)	118.26(17)
C(19A)-C(18A)-C(17A)	118.06(16)
C(23A)-C(18A)-C(17A)	123.59(16)
C(20A)-C(19A)-C(18A)	122.66(17)
C(21A)-C(20A)-C(19A)	118.49(18)
C(20A)-C(21A)-C(22A)	120.81(18)
C(21A)-C(22A)-C(23A)	121.27(17)
N(3A)-C(23A)-C(22A)	122.17(16)
N(3A)-C(23A)-C(18A)	119.42(16)
C(22A)-C(23A)-C(18A)	118.41(16)
C(29A)-C(24A)-C(25A)	119.11(15)
C(29A)-C(24A)-N(3A)	123.20(16)
C(25A)-C(24A)-N(3A)	117.66(15)
C(24A)-C(25A)-C(26A)	121.83(16)
C(27A)-C(26A)-C(25A)	117.10(16)
C(27A)-C(26A)-C(38A)	123.27(16)
C(25A)-C(26A)-C(38A)	119.53(16)
C(28A)-C(27A)-C(26A)	122.62(16)
C(27A)-C(28A)-C(29A)	118.56(16)
C(27A)-C(28A)-C(42A)	122.02(16)
C(29A)-C(28A)-C(42A)	119.33(17)
C(24A)-C(29A)-C(28A)	120.59(16)
C(33A)-C(30A)-C(32A)	108.37(15)
C(33A)-C(30A)-C(3A)	112.52(15)
C(32A)-C(30A)-C(3A)	109.03(14)
C(33A)-C(30A)-C(31A)	108.04(15)
C(32A)-C(30A)-C(31A)	109.29(16)
C(3A)-C(30A)-C(31A)	109.53(15)
C(35C)-C(34A)-C(37C)	118.2(3)
C(35C)-C(34A)-C(35A)	131.7(3)
C(37C)-C(34A)-C(35A)	44.82(19)
C(35C)-C(34A)-C(37A)	36.40(19)
C(37C)-C(34A)-C(37A)	130.1(3)
C(35A)-C(34A)-C(37A)	111.3(3)
C(35C)-C(34A)-C(5A)	111.7(2)
C(37C)-C(34A)-C(5A)	113.1(2)
C(35A)-C(34A)-C(5A)	116.4(2)
C(37A)-C(34A)-C(5A)	116.6(2)
C(35C)-C(34A)-C(36C)	105.7(3)
C(37C)-C(34A)-C(36C)	104.6(3)
C(35A)-C(34A)-C(36C)	59.9(2)
C(37A)-C(34A)-C(36C)	69.3(2)
C(5A)-C(34A)-C(36C)	101.57(18)
C(35C)-C(34A)-C(36A)	66.6(2)
C(37C)-C(34A)-C(36A)	61.9(2)
C(35A)-C(34A)-C(36A)	104.3(2)
C(37A)-C(34A)-C(36A)	100.2(2)
C(5A)-C(34A)-C(36A)	105.75(17)
C(36C)-C(34A)-C(36A)	152.5(2)
C(26A)-C(38A)-C(40A)	110.15(14)
C(26A)-C(38A)-C(41A)	113.23(15)
C(40A)-C(38A)-C(41A)	107.33(16)
C(26A)-C(38A)-C(39A)	110.36(15)
C(40A)-C(38A)-C(39A)	108.57(15)
C(41A)-C(38A)-C(39A)	107.03(15)
C(44C)-C(42A)-C(44A)	122.6(3)
C(44C)-C(42A)-C(28A)	119.0(3)
C(44A)-C(42A)-C(28A)	116.6(2)
C(44C)-C(42A)-C(43A)	39.1(3)
C(44A)-C(42A)-C(43A)	109.5(2)
C(28A)-C(42A)-C(43A)	108.32(19)
C(44C)-C(42A)-C(45A)	64.4(3)

C(44A)-C(42A)-C(45A)	109.9(2)	C(23B)-C(22B)-C(21B)	121.58(16)
C(28A)-C(42A)-C(45A)	108.18(16)	C(22B)-C(23B)-N(3B)	122.36(15)
C(43A)-C(42A)-C(45A)	103.5(2)	C(22B)-C(23B)-C(18B)	118.73(16)
C(44C)-C(42A)-C(43C)	112.9(4)	N(3B)-C(23B)-C(18B)	118.92(16)
C(44A)-C(42A)-C(43C)	64.7(2)	C(25B)-C(24B)-C(29B)	120.06(14)
C(28A)-C(42A)-C(43C)	103.8(2)	C(25B)-C(24B)-N(3B)	117.35(15)
C(43A)-C(42A)-C(43C)	145.6(3)	C(29B)-C(24B)-N(3B)	122.53(15)
C(45A)-C(42A)-C(43C)	54.0(2)	C(24B)-C(25B)-C(26B)	121.12(16)
C(44C)-C(42A)-C(45C)	109.4(4)	C(27B)-C(26B)-C(25B)	117.29(16)
C(44A)-C(42A)-C(45C)	36.3(2)	C(27B)-C(26B)-C(38B)	123.79(14)
C(28A)-C(42A)-C(45C)	109.2(3)	C(25B)-C(26B)-C(38B)	118.89(15)
C(43A)-C(42A)-C(45C)	79.8(3)	C(26B)-C(27B)-C(28B)	122.99(15)
C(45A)-C(42A)-C(45C)	139.2(3)	C(29B)-C(28B)-C(27B)	117.92(16)
C(43C)-C(42A)-C(45C)	101.0(3)	C(29B)-C(28B)-C(42B)	121.63(15)
C(7B)-N(1B)-C(1B)	131.55(15)	C(27B)-C(28B)-C(42B)	120.37(14)
C(17B)-N(2B)-C(13B)	121.33(15)	C(24B)-C(29B)-C(28B)	120.45(16)
C(23B)-N(3B)-C(24B)	127.92(14)	C(33D)-C(30B)-C(31B)	120.6(9)
C(2B)-C(1B)-C(6B)	119.57(16)	C(33D)-C(30B)-C(5B)	117.1(7)
C(2B)-C(1B)-N(1B)	116.25(16)	C(31B)-C(30B)-C(5B)	112.8(4)
C(6B)-C(1B)-N(1B)	124.07(17)	C(33D)-C(30B)-C(33B)	79.6(8)
C(3B)-C(2B)-C(1B)	121.63(17)	C(31B)-C(30B)-C(33B)	110.2(3)
C(2B)-C(3B)-C(4B)	117.32(18)	C(5B)-C(30B)-C(33B)	111.37(19)
C(2B)-C(3B)-C(34B)	118.73(16)	C(33D)-C(30B)-C(32B)	23.4(9)
C(4B)-C(3B)-C(34B)	123.94(17)	C(31B)-C(30B)-C(32B)	112.9(4)
C(5B)-C(4B)-C(3B)	122.63(17)	C(5B)-C(30B)-C(32B)	106.2(3)
C(4B)-C(5B)-C(6B)	118.65(17)	C(33B)-C(30B)-C(32B)	102.9(3)
C(4B)-C(5B)-C(30B)	119.99(17)	C(33D)-C(30B)-C(31D)	116.0(7)
C(6B)-C(5B)-C(30B)	121.29(18)	C(31B)-C(30B)-C(31D)	73.1(5)
C(5B)-C(6B)-C(1B)	120.18(17)	C(5B)-C(30B)-C(31D)	109.1(4)
N(1B)-C(7B)-C(8B)	121.45(17)	C(33B)-C(30B)-C(31D)	42.5(5)
N(1B)-C(7B)-C(12B)	119.25(17)	C(32B)-C(30B)-C(31D)	137.9(5)
C(8B)-C(7B)-C(12B)	119.27(17)	C(33D)-C(30B)-C(32D)	107.6(11)
C(9B)-C(8B)-C(7B)	120.94(18)	C(31B)-C(30B)-C(32D)	23.6(6)
C(8B)-C(9B)-C(10B)	120.58(19)	C(5B)-C(30B)-C(32D)	108.4(6)
C(11B)-C(10B)-C(9B)	118.88(19)	C(33B)-C(30B)-C(32D)	130.1(5)
C(10B)-C(11B)-C(12B)	122.90(19)	C(32B)-C(30B)-C(32D)	93.5(6)
C(11B)-C(12B)-C(7B)	117.26(18)	C(31D)-C(30B)-C(32D)	96.5(6)
C(11B)-C(12B)-C(13B)	119.23(17)	C(35B)-C(34B)-C(36B)	109.44(16)
C(7B)-C(12B)-C(13B)	123.50(17)	C(35B)-C(34B)-C(3B)	112.94(16)
N(2B)-C(13B)-C(14B)	120.03(17)	C(36B)-C(34B)-C(3B)	109.22(15)
N(2B)-C(13B)-C(12B)	117.70(15)	C(35B)-C(34B)-C(37B)	106.41(16)
C(14B)-C(13B)-C(12B)	122.27(17)	C(36B)-C(34B)-C(37B)	108.71(15)
C(15B)-C(14B)-C(13B)	118.97(18)	C(3B)-C(34B)-C(37B)	110.01(14)
C(14B)-C(15B)-C(16B)	120.47(17)	C(39B)-C(38B)-C(26B)	112.10(14)
C(15B)-C(16B)-C(17B)	118.40(18)	C(39B)-C(38B)-C(40B)	108.19(13)
N(2B)-C(17B)-C(16B)	120.71(17)	C(26B)-C(38B)-C(40B)	109.43(13)
N(2B)-C(17B)-C(18B)	116.88(15)	C(39B)-C(38B)-C(41B)	108.30(13)
C(16B)-C(17B)-C(18B)	122.30(17)	C(26B)-C(38B)-C(41B)	109.79(14)
C(19B)-C(18B)-C(23B)	118.00(16)	C(40B)-C(38B)-C(41B)	108.96(13)
C(19B)-C(18B)-C(17B)	118.60(15)	C(45B)-C(42B)-C(28B)	113.12(13)
C(23B)-C(18B)-C(17B)	123.26(16)	C(45B)-C(42B)-C(43B)	107.85(15)
C(20B)-C(19B)-C(18B)	122.99(16)	C(28B)-C(42B)-C(43B)	111.15(14)
C(19B)-C(20B)-C(21B)	118.66(17)	C(45B)-C(42B)-C(44B)	107.41(15)
C(20B)-C(21B)-C(22B)	119.92(18)	C(28B)-C(42B)-C(44B)	108.22(14)

C(43B)-C(42B)-C(44B) 108.97(14)

Table 5. Hydrogen bonds for ECW19 (CCDC 793156) [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1A)-H(1A)...N(2A)	0.88	1.96	2.690(2)	139.0
N(3A)-H(3A)...N(2A)	0.88	2.10	2.7575(18)	130.4
N(1B)-H(1B)...N(2B)	0.88	1.94	2.6732(19)	139.9
N(3B)-H(3B)...N(2B)	0.88	2.16	2.7660(18)	125.8

CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE

X-RAY CRYSTALLOGRAPHY LABORATORY

Date 23 July 2010

Crystal Structure Analysis of:

[ONO]Fe(NC_5H_5)$_2$ (ECW17)

(shown below)

For Investigator: Ed Weintrob ext. 6576

Advisor: J. E. Bercaw ext. 6577

Account Number: JEB.ENERGY-1.02-GRANT.MOOREJCP

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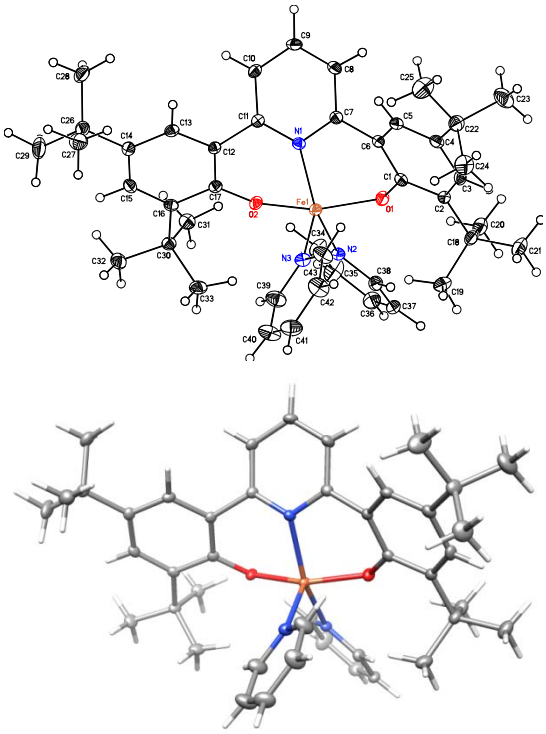
Contents



ECW17

Note: The crystallographic data have been deposited in the Cambridge Database (CCDC) and has been placed on hold pending further instructions from me. The deposition number is 785427. Ideally the CCDC would like the publication to contain a footnote of the type: "Crystallographic data have been deposited at the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK and copies can be obtained on request, free of charge, by quoting the publication citation and the deposition number 785427."

Table 1. Crystal data and structure refinement for ECW17 (CCDC 785427).

Empirical formula	$C_{43}H_{53}N_3O_2Fe \bullet C_7H_8$
Formula weight	791.87
Crystallization Solvent	Toluene/*n*-heptane
Crystal Habit	Chunk
Crystal size	0.22 x 0.21 x 0.18 mm^3
Crystal color	Dark brown

Data Collection

Type of diffractometer	Bruker KAPPA APEX II	
Wavelength	0.71073 Å MoKα	
Data Collection Temperature	100(2) K	
θ range for 9221 reflections used in lattice determination	2.50 to 27.94°	
Unit cell dimensions	a = 15.8750(6) Å b = 28.4782(10) Å c = 9.7853(4) Å	α= 90° β= 90.514(2)° γ = 90°
Volume	4423.7(3) $Å^3$	
Z	4	
Crystal system	Monoclinic	
Space group	P $2_1/c$	
Density (calculated)	1.189 Mg/m^3	
F(000)	1696	
Data collection program	Bruker APEX2 v2009.7-0	
θ range for data collection	1.92 to 29.74°	
Completeness to θ = 29.74°	95.6 %	
Index ranges	$-21 \le h \le 22$, $-39 \le k \le 38$, $-12 \le l \le 13$	
Data collection scan type	ω scans; 12 settings	
Data reduction program	Bruker SAINT-Plus v7.66A	
Reflections collected	96663	
Independent reflections	12036 [R_{int}= 0.0514]	
Absorption coefficient	0.382 mm^{-1}	
Absorption correction	None	
Max. and min. transmission	0.9344 and 0.9206	

Table 1 (cont.)

Structure solution and Refinement

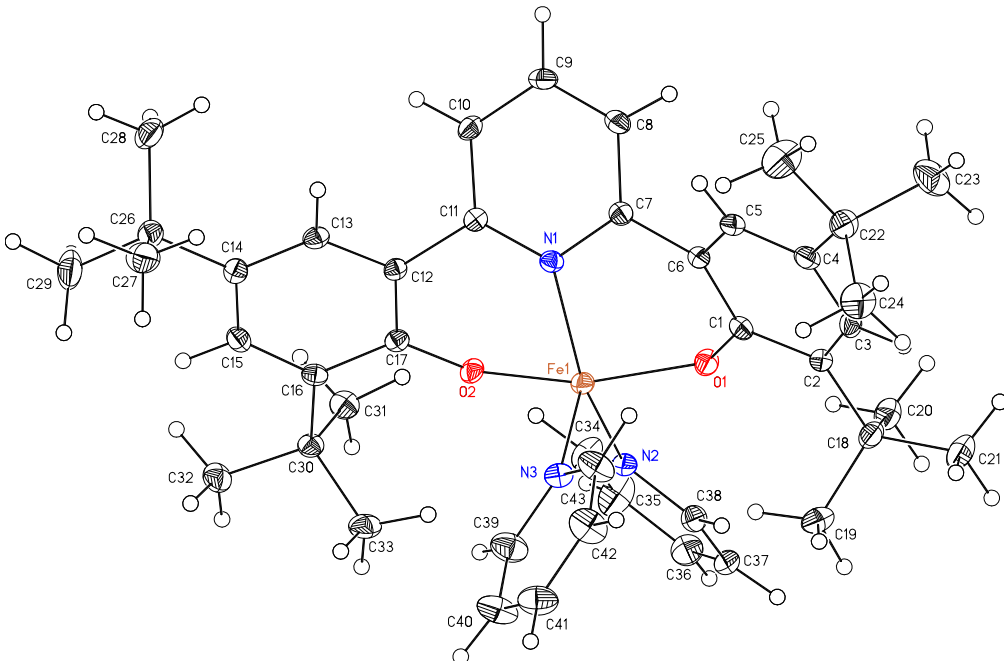
Structure solution program	SHELXS-97 (Sheldrick, 2008)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Difference Fourier map
Structure refinement program	SHELXL-97 (Sheldrick, 2008)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	12036 / 0 / 749
Treatment of hydrogen atoms	Unrestrained
Goodness-of-fit on F^2	2.235
Final R indices [$I>2\sigma(I)$, 9399 reflections]	R1 = 0.0444, *w*R2 = 0.0613
R indices (all data)	R1 = 0.0614, *w*R2 = 0.0621
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.001
Average shift/error	0.000
Largest diff. peak and hole	0.804 and -0.672 e.Å^{-3}

Special Refinement Details

Crystals were mounted on a glass fiber using Paratone oil then placed on the diffractometer under a nitrogen stream at 100K.

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

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.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C10
C8
C28
C25
C23
C26
C11
C7
C5
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C13
N1
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C29
C27
C12
C24
C1
C3
C15
C17
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C2
C16
O2
C31
C34
C20
C18
N2
C21
N3
C32
C30
C43
C35
C38
C39
C33
C42
C19
C37
C36
C40
C41

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for ECW17 (CCDC 785427). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Fe(1)	4229(1)	6372(1)	5559(1)	13(1)
O(1)	3041(1)	6269(1)	5136(1)	16(1)
O(2)	5275(1)	6724(1)	5651(1)	15(1)
N(1)	4539(1)	6184(1)	3431(1)	13(1)
N(2)	3797(1)	6837(1)	7263(1)	15(1)
N(3)	4495(1)	5708(1)	6507(1)	17(1)
C(1)	2725(1)	5940(1)	4309(1)	14(1)
C(2)	1969(1)	5694(1)	4627(1)	14(1)
C(3)	1705(1)	5340(1)	3748(2)	17(1)
C(4)	2132(1)	5206(1)	2562(1)	16(1)
C(5)	2837(1)	5462(1)	2239(2)	16(1)
C(6)	3140(1)	5830(1)	3072(1)	13(1)
C(7)	3876(1)	6088(1)	2575(1)	13(1)
C(8)	3901(1)	6217(1)	1196(2)	17(1)
C(9)	4608(1)	6422(1)	670(2)	18(1)
C(10)	5309(1)	6479(1)	1512(2)	15(1)
C(11)	5262(1)	6356(1)	2894(1)	12(1)
C(12)	6032(1)	6416(1)	3751(1)	12(1)
C(13)	6802(1)	6311(1)	3152(1)	14(1)
C(14)	7572(1)	6410(1)	3774(1)	14(1)
C(15)	7529(1)	6624(1)	5060(2)	16(1)
C(16)	6790(1)	6733(1)	5723(1)	13(1)
C(17)	6005(1)	6620(1)	5076(1)	12(1)
C(18)	1480(1)	5807(1)	5930(2)	18(1)
C(19)	2006(1)	5639(1)	7172(2)	22(1)
C(20)	1292(1)	6336(1)	6029(2)	22(1)
C(21)	632(1)	5552(1)	5999(2)	26(1)
C(22)	1802(1)	4798(1)	1683(2)	20(1)
C(23)	950(1)	4936(1)	1042(2)	32(1)
C(24)	1691(1)	4359(1)	2562(2)	27(1)
C(25)	2410(1)	4676(1)	528(2)	34(1)
C(26)	8399(1)	6289(1)	3072(1)	19(1)
C(27)	8502(1)	5756(1)	3020(2)	27(1)
C(28)	8401(1)	6487(1)	1610(2)	24(1)
C(29)	9165(1)	6498(1)	3828(2)	33(1)
C(30)	6813(1)	7003(1)	7091(1)	15(1)
C(31)	6460(1)	7497(1)	6842(2)	19(1)
C(32)	7711(1)	7061(1)	7661(2)	21(1)
C(33)	6305(1)	6751(1)	8193(2)	21(1)
C(34)	4135(1)	7264(1)	7435(2)	23(1)
C(35)	3834(1)	7586(1)	8369(2)	31(1)
C(36)	3159(1)	7471(1)	9169(2)	23(1)
C(37)	2801(1)	7033(1)	9008(2)	20(1)
C(38)	3134(1)	6730(1)	8049(2)	18(1)
C(39)	4977(1)	5672(1)	7646(2)	26(1)
C(40)	5114(1)	5254(1)	8309(2)	30(1)
C(41)	4746(1)	4851(1)	7827(2)	27(1)

C(42)	4260(1)	4879(1)	6644(2)	27(1)
C(43)	4159(1)	5308(1)	6033(2)	25(1)
C(51)	463(1)	6917(1)	7(2)	21(1)
C(52)	740(1)	6479(1)	417(2)	24(1)
C(53)	1305(1)	6427(1)	1509(2)	26(1)
C(54)	1600(1)	6819(1)	2178(2)	26(1)
C(55)	1335(1)	7260(1)	1778(2)	28(1)
C(56)	773(1)	7309(1)	689(2)	25(1)
C(57)	-171(1)	6973(1)	-1147(2)	31(1)

Table 3. Selected bond lengths [Å] and angles [°] for ECW17 (CCDC 785427).

Fe(1)-O(2)	1.9413(10)	O(2)-Fe(1)-O(1)	155.92(4)
Fe(1)-O(1)	1.9490(9)	O(2)-Fe(1)-N(3)	105.65(4)
Fe(1)-N(3)	2.1450(12)	O(1)-Fe(1)-N(3)	98.40(4)
Fe(1)-N(1)	2.2091(12)	O(2)-Fe(1)-N(1)	88.36(4)
Fe(1)-N(2)	2.2433(12)	O(1)-Fe(1)-N(1)	89.26(4)
		N(3)-Fe(1)-N(1)	98.64(4)
		O(2)-Fe(1)-N(2)	85.82(4)
		O(1)-Fe(1)-N(2)	86.82(4)
		N(3)-Fe(1)-N(2)	105.02(4)
		N(1)-Fe(1)-N(2)	156.33(4)

Table 4. Bond lengths [Å] and angles [°] for ECW17 (CCDC 785427).

Fe(1)-O(2)	1.9413(10)	C(21)-H(21A)	0.975(15)
Fe(1)-O(1)	1.9490(9)	C(21)-H(21B)	0.985(17)
Fe(1)-N(3)	2.1450(12)	C(21)-H(21C)	0.975(15)
Fe(1)-N(1)	2.2091(12)	C(22)-C(25)	1.532(2)
Fe(1)-N(2)	2.2433(12)	C(22)-C(24)	1.528(2)
O(1)-C(1)	1.3336(15)	C(22)-C(23)	1.536(2)
O(2)-C(17)	1.3255(16)	C(23)-H(23A)	0.992(17)
N(1)-C(11)	1.3589(17)	C(23)-H(23B)	1.011(16)
N(1)-C(7)	1.3659(15)	C(23)-H(23C)	0.968(15)
N(2)-C(34)	1.3386(19)	C(24)-H(24A)	0.958(16)
N(2)-C(38)	1.3442(19)	C(24)-H(24B)	0.996(16)
N(3)-C(43)	1.3402(18)	C(24)-H(24C)	0.972(15)
N(3)-C(39)	1.3493(17)	C(25)-H(25A)	0.995(15)
C(1)-C(6)	1.418(2)	C(25)-H(25B)	0.967(16)
C(1)-C(2)	1.4260(19)	C(25)-H(25C)	1.002(18)
C(2)-C(3)	1.3883(19)	C(26)-C(27)	1.528(2)
C(2)-C(18)	1.533(2)	C(26)-C(29)	1.538(2)
C(3)-C(4)	1.402(2)	C(26)-C(28)	1.538(2)
C(3)-H(3)	0.953(13)	C(27)-H(27A)	1.002(16)
C(4)-C(5)	1.375(2)	C(27)-H(27B)	1.021(14)
C(4)-C(22)	1.5369(19)	C(27)-H(27C)	0.951(16)
C(5)-C(6)	1.4081(19)	C(28)-H(28A)	0.975(16)
C(5)-H(5)	0.910(13)	C(28)-H(28B)	1.027(13)
C(6)-C(7)	1.468(2)	C(28)-H(28C)	0.941(15)
C(7)-C(8)	1.3989(19)	C(29)-H(29A)	0.972(16)
C(8)-C(9)	1.370(2)	C(29)-H(29B)	1.005(16)
C(8)-H(8)	0.931(13)	C(29)-H(29C)	1.047(17)
C(9)-C(10)	1.3870(18)	C(30)-C(32)	1.5343(19)
C(9)-H(9)	0.990(14)	C(30)-C(31)	1.532(2)
C(10)-C(11)	1.4000(19)	C(30)-C(33)	1.531(2)
C(10)-H(10)	0.967(13)	C(31)-H(31A)	1.063(15)
C(11)-C(12)	1.4854(16)	C(31)-H(31B)	0.952(15)
C(12)-C(13)	1.3932(19)	C(31)-H(31C)	0.988(14)
C(12)-C(17)	1.4213(18)	C(32)-H(32A)	1.020(15)
C(13)-C(14)	1.3892(17)	C(32)-H(32B)	0.982(15)
C(13)-H(13)	0.974(12)	C(32)-H(32C)	1.017(16)
C(14)-C(15)	1.3996(19)	C(33)-H(33A)	0.989(13)
C(14)-C(26)	1.527(2)	C(33)-H(33B)	0.941(16)
C(15)-C(16)	1.382(2)	C(33)-H(33C)	1.014(14)
C(15)-H(15)	0.954(12)	C(34)-C(35)	1.383(2)
C(16)-C(17)	1.4309(17)	C(34)-H(34)	0.976(16)
C(16)-C(30)	1.5437(19)	C(35)-C(36)	1.372(2)
C(18)-C(21)	1.532(2)	C(35)-H(35)	0.912(17)
C(18)-C(20)	1.539(2)	C(36)-C(37)	1.378(2)
C(18)-C(19)	1.544(2)	C(36)-H(36)	0.975(15)
C(19)-H(19A)	1.022(15)	C(37)-C(38)	1.382(2)
C(19)-H(19B)	0.980(16)	C(37)-H(37)	0.941(16)
C(19)-H(19C)	0.997(14)	C(38)-H(38)	0.968(14)
C(20)-H(20A)	0.975(14)	C(39)-C(40)	1.374(2)
C(20)-H(20B)	1.011(16)	C(39)-H(39)	1.005(14)
C(20)-H(20C)	0.986(15)	C(40)-C(41)	1.369(2)

C(40)-H(40)	0.926(15)	C(3)-C(4)-C(22)	120.27(13)
C(41)-C(42)	1.388(2)	C(4)-C(5)-C(6)	122.42(15)
C(41)-H(41)	0.966(15)	C(4)-C(5)-H(5)	121.5(9)
C(42)-C(43)	1.369(2)	C(6)-C(5)-H(5)	115.9(9)
C(42)-H(42)	0.918(15)	C(5)-C(6)-C(1)	119.92(14)
C(43)-H(43)	0.934(14)	C(5)-C(6)-C(7)	116.76(13)
C(51)-C(52)	1.380(2)	C(1)-C(6)-C(7)	123.31(13)
C(51)-C(56)	1.389(2)	N(1)-C(7)-C(8)	120.75(13)
C(51)-C(57)	1.515(2)	N(1)-C(7)-C(6)	120.54(12)
C(52)-C(53)	1.397(2)	C(8)-C(7)-C(6)	118.65(12)
C(52)-H(52)	0.938(14)	C(9)-C(8)-C(7)	120.24(13)
C(53)-C(54)	1.376(2)	C(9)-C(8)-H(8)	124.0(9)
C(53)-H(53)	0.961(15)	C(7)-C(8)-H(8)	115.7(9)
C(54)-C(55)	1.379(2)	C(8)-C(9)-C(10)	118.87(14)
C(54)-H(54)	0.920(14)	C(8)-C(9)-H(9)	119.9(8)
C(55)-C(56)	1.391(2)	C(10)-C(9)-H(9)	121.2(8)
C(55)-H(55)	0.936(14)	C(9)-C(10)-C(11)	119.67(14)
C(56)-H(56)	0.937(14)	C(9)-C(10)-H(10)	120.4(7)
C(57)-H(57A)	0.991(19)	C(11)-C(10)-H(10)	119.8(7)
C(57)-H(57B)	0.95(2)	N(1)-C(11)-C(10)	121.05(12)
C(57)-H(57C)	0.965(19)	N(1)-C(11)-C(12)	121.14(12)
		C(10)-C(11)-C(12)	117.80(12)
O(2)-Fe(1)-O(1)	155.92(4)	C(13)-C(12)-C(17)	120.38(12)
O(2)-Fe(1)-N(3)	105.65(4)	C(13)-C(12)-C(11)	117.27(12)
O(1)-Fe(1)-N(3)	98.40(4)	C(17)-C(12)-C(11)	122.02(13)
O(2)-Fe(1)-N(1)	88.36(4)	C(14)-C(13)-C(12)	122.92(13)
O(1)-Fe(1)-N(1)	89.26(4)	C(14)-C(13)-H(13)	119.1(8)
N(3)-Fe(1)-N(1)	98.64(4)	C(12)-C(13)-H(13)	118.0(8)
O(2)-Fe(1)-N(2)	85.82(4)	C(13)-C(14)-C(15)	115.67(13)
O(1)-Fe(1)-N(2)	86.82(4)	C(13)-C(14)-C(26)	120.89(13)
N(3)-Fe(1)-N(2)	105.02(4)	C(15)-C(14)-C(26)	123.44(12)
N(1)-Fe(1)-N(2)	156.33(4)	C(16)-C(15)-C(14)	124.64(13)
C(1)-O(1)-Fe(1)	126.31(9)	C(16)-C(15)-H(15)	120.4(8)
C(17)-O(2)-Fe(1)	127.98(9)	C(14)-C(15)-H(15)	115.0(8)
C(11)-N(1)-C(7)	118.94(11)	C(15)-C(16)-C(17)	118.78(13)
C(11)-N(1)-Fe(1)	118.23(8)	C(15)-C(16)-C(30)	120.44(12)
C(7)-N(1)-Fe(1)	116.69(9)	C(17)-C(16)-C(30)	120.65(13)
C(34)-N(2)-C(38)	116.65(14)	O(2)-C(17)-C(12)	120.81(12)
C(34)-N(2)-Fe(1)	120.36(11)	O(2)-C(17)-C(16)	121.55(12)
C(38)-N(2)-Fe(1)	122.55(10)	C(12)-C(17)-C(16)	117.54(13)
C(43)-N(3)-C(39)	116.24(13)	C(21)-C(18)-C(2)	112.85(13)
C(43)-N(3)-Fe(1)	121.55(10)	C(21)-C(18)-C(20)	106.88(14)
C(39)-N(3)-Fe(1)	122.15(11)	C(2)-C(18)-C(20)	110.95(13)
O(1)-C(1)-C(6)	119.87(13)	C(21)-C(18)-C(19)	106.64(14)
O(1)-C(1)-C(2)	121.69(13)	C(2)-C(18)-C(19)	108.41(13)
C(6)-C(1)-C(2)	118.44(13)	C(20)-C(18)-C(19)	111.04(13)
C(3)-C(2)-C(1)	118.12(14)	C(18)-C(19)-H(19A)	110.6(8)
C(3)-C(2)-C(18)	121.06(13)	C(18)-C(19)-H(19B)	109.6(9)
C(1)-C(2)-C(18)	120.78(12)	H(19A)-C(19)-H(19B)	106.2(12)
C(2)-C(3)-C(4)	124.36(14)	C(18)-C(19)-H(19C)	111.7(8)
C(2)-C(3)-H(3)	118.4(8)	H(19A)-C(19)-H(19C)	108.9(11)
C(4)-C(3)-H(3)	117.3(8)	H(19B)-C(19)-H(19C)	109.6(12)
C(5)-C(4)-C(3)	116.54(14)	C(18)-C(20)-H(20A)	111.9(8)
C(5)-C(4)-C(22)	123.18(14)	C(18)-C(20)-H(20B)	109.2(9)

H(20A)-C(20)-H(20B)	110.4(11)	H(29A)-C(29)-H(29B)	112.0(14)
C(18)-C(20)-H(20C)	110.8(9)	C(26)-C(29)-H(29C)	109.9(8)
H(20A)-C(20)-H(20C)	110.4(12)	H(29A)-C(29)-H(29C)	106.5(13)
H(20B)-C(20)-H(20C)	103.8(12)	H(29B)-C(29)-H(29C)	106.0(13)
C(18)-C(21)-H(21A)	112.7(9)	C(32)-C(30)-C(31)	107.27(13)
C(18)-C(21)-H(21B)	109.7(9)	C(32)-C(30)-C(33)	106.76(12)
H(21A)-C(21)-H(21B)	103.4(12)	C(31)-C(30)-C(33)	110.35(14)
C(18)-C(21)-H(21C)	112.3(9)	C(32)-C(30)-C(16)	112.52(13)
H(21A)-C(21)-H(21C)	110.9(12)	C(31)-C(30)-C(16)	108.24(11)
H(21B)-C(21)-H(21C)	107.2(13)	C(33)-C(30)-C(16)	111.62(12)
C(25)-C(22)-C(24)	107.86(16)	C(30)-C(31)-H(31A)	110.9(8)
C(25)-C(22)-C(4)	111.77(13)	C(30)-C(31)-H(31B)	113.0(9)
C(24)-C(22)-C(4)	110.13(12)	H(31A)-C(31)-H(31B)	106.6(12)
C(25)-C(22)-C(23)	108.36(14)	C(30)-C(31)-H(31C)	112.0(8)
C(24)-C(22)-C(23)	109.48(14)	H(31A)-C(31)-H(31C)	108.2(11)
C(4)-C(22)-C(23)	109.19(14)	H(31B)-C(31)-H(31C)	105.8(12)
C(22)-C(23)-H(23A)	111.6(9)	C(30)-C(32)-H(32A)	110.7(8)
C(22)-C(23)-H(23B)	112.8(8)	C(30)-C(32)-H(32B)	105.5(9)
H(23A)-C(23)-H(23B)	104.0(13)	H(32A)-C(32)-H(32B)	114.3(12)
C(22)-C(23)-H(23C)	108.9(9)	C(30)-C(32)-H(32C)	113.1(8)
H(23A)-C(23)-H(23C)	112.0(12)	H(32A)-C(32)-H(32C)	107.2(12)
H(23B)-C(23)-H(23C)	107.5(13)	H(32B)-C(32)-H(32C)	106.1(12)
C(22)-C(24)-H(24A)	113.4(9)	C(30)-C(33)-H(33A)	114.2(8)
C(22)-C(24)-H(24B)	110.7(9)	C(30)-C(33)-H(33B)	108.5(10)
H(24A)-C(24)-H(24B)	104.7(13)	H(33A)-C(33)-H(33B)	105.2(13)
C(22)-C(24)-H(24C)	111.5(10)	C(30)-C(33)-H(33C)	108.1(8)
H(24A)-C(24)-H(24C)	104.1(13)	H(33A)-C(33)-H(33C)	109.0(11)
H(24B)-C(24)-H(24C)	112.1(12)	H(33B)-C(33)-H(33C)	111.9(12)
C(22)-C(25)-H(25A)	112.6(9)	N(2)-C(34)-C(35)	122.97(17)
C(22)-C(25)-H(25B)	111.1(10)	N(2)-C(34)-H(34)	117.2(9)
H(25A)-C(25)-H(25B)	105.8(13)	C(35)-C(34)-H(34)	119.8(9)
C(22)-C(25)-H(25C)	106.9(10)	C(36)-C(35)-C(34)	119.68(18)
H(25A)-C(25)-H(25C)	106.9(13)	C(36)-C(35)-H(35)	123.9(11)
H(25B)-C(25)-H(25C)	113.6(13)	C(34)-C(35)-H(35)	116.4(12)
C(14)-C(26)-C(27)	109.41(14)	C(35)-C(36)-C(37)	118.31(17)
C(14)-C(26)-C(29)	112.06(13)	C(35)-C(36)-H(36)	120.4(10)
C(27)-C(26)-C(29)	108.45(14)	C(37)-C(36)-H(36)	121.3(10)
C(14)-C(26)-C(28)	110.11(12)	C(36)-C(37)-C(38)	118.76(17)
C(27)-C(26)-C(28)	109.43(14)	C(36)-C(37)-H(37)	122.8(10)
C(29)-C(26)-C(28)	107.33(14)	C(38)-C(37)-H(37)	118.3(10)
C(26)-C(27)-H(27A)	109.8(9)	N(2)-C(38)-C(37)	123.63(16)
C(26)-C(27)-H(27B)	111.4(8)	N(2)-C(38)-H(38)	116.2(9)
H(27A)-C(27)-H(27B)	113.4(12)	C(37)-C(38)-H(38)	120.1(9)
C(26)-C(27)-H(27C)	114.8(10)	N(3)-C(39)-C(40)	122.85(15)
H(27A)-C(27)-H(27C)	105.1(13)	N(3)-C(39)-H(39)	115.5(8)
H(27B)-C(27)-H(27C)	102.2(13)	C(40)-C(39)-H(39)	121.7(8)
C(26)-C(28)-H(28A)	110.7(9)	C(39)-C(40)-C(41)	119.93(15)
C(26)-C(28)-H(28B)	111.8(8)	C(39)-C(40)-H(40)	118.6(11)
H(28A)-C(28)-H(28B)	108.4(11)	C(41)-C(40)-H(40)	121.4(11)
C(26)-C(28)-H(28C)	108.7(9)	C(40)-C(41)-C(42)	118.13(16)
H(28A)-C(28)-H(28C)	106.4(13)	C(40)-C(41)-H(41)	123.4(9)
H(28B)-C(28)-H(28C)	110.7(11)	C(42)-C(41)-H(41)	118.4(9)
C(26)-C(29)-H(29A)	111.4(10)	C(43)-C(42)-C(41)	118.53(16)
C(26)-C(29)-H(29B)	110.9(9)	C(43)-C(42)-H(42)	122.9(9)

C(41)-C(42)-H(42)	118.6(9)
N(3)-C(43)-C(42)	124.29(14)
N(3)-C(43)-H(43)	114.9(10)
C(42)-C(43)-H(43)	120.8(10)
C(52)-C(51)-C(56)	118.42(14)
C(52)-C(51)-C(57)	121.40(16)
C(56)-C(51)-C(57)	120.18(16)
C(51)-C(52)-C(53)	121.25(16)
C(51)-C(52)-H(52)	119.9(9)
C(53)-C(52)-H(52)	118.7(9)
C(54)-C(53)-C(52)	119.49(16)
C(54)-C(53)-H(53)	122.1(9)
C(52)-C(53)-H(53)	118.4(9)
C(53)-C(54)-C(55)	120.12(15)
C(53)-C(54)-H(54)	117.7(10)
C(55)-C(54)-H(54)	122.1(10)
C(54)-C(55)-C(56)	120.06(17)
C(54)-C(55)-H(55)	121.9(9)
C(56)-C(55)-H(55)	118.0(9)
C(51)-C(56)-C(55)	120.64(16)
C(51)-C(56)-H(56)	121.0(8)
C(55)-C(56)-H(56)	118.4(8)
C(51)-C(57)-H(57A)	110.1(10)
C(51)-C(57)-H(57B)	110.1(12)
H(57A)-C(57)-H(57B)	110.9(16)
C(51)-C(57)-H(57C)	111.1(10)
H(57A)-C(57)-H(57C)	107.0(15)
H(57B)-C(57)-H(57C)	107.6(16)