Supplementary data for:

New Diamino-Diheterophenol Ligands Coordinate Iron(III) to Make Structural and Functional Models of Protocatechuate 3,4-dioxygenase.

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Materials and General Procedures. All compounds were purchased from commercial sources and used as received. Reactions that made use of a pressure flask were conducted behind a blast shield for additional safety.

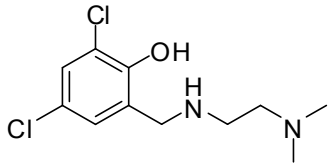
Equipment and Procedures. NMR spectra were recorded on an Oxford AS400 spectrometer and referenced to residual peaks in the deuterated solvents. MS data were collected either on a Bruker Daltonics-LC Ion trap mass spectrometer (ESI) or a JEOL MStation JMS 700 (FAB) both located at the UMass Amherst Mass Spectrometry Center. GCMS data was collected on a Hewlett Packard 6890 series GC System interfaced with the HP 5973 Mass Selective Detector. IR data was collected on a PerkinElmer Spectrum One FT-IR Spectrometer with a universal ATR sampling accessory.

Cyclic Voltammatry. Cyclic voltammatry was carried out using Hewlett-Packard Powersuite Software. A three-electrode setup was employed, using either a platinum or an ITO covered glass plate as the working electrode, a platinum gauze counter electrode, and an Ag/Ag^+ reference electrode. The reference electrode solution was prepared from a solution of 0.1 M tetrabutyl ammonium PF_6 (TBAP) and 0.01 M $AgNO_3$ in anhydrous acetonitrile, and stored in the absence of light. The supporting electrolyte for all electrochemical experiments were solutions of 0.1 M TBAP in anhydrous acetonitrile. All experiments were conducted in a MBraun glove box. Before every experiment a CV of ferrocene was run in the supporting electrolyte to check for reversibility and position of the $E_{1/2}$ of the Fc/Fc^+ couple. For all trials $E_{1/2}$ is reported with respect to the reversible oxidation of ferrocene.

X-ray crystallography. X-ray intensity data was measured at 100 K (Bruker KRYO-FLEX) on a Bruker SMART APEX CCD-based X-ray diffractometer system equipped with a Mo-target X-ray tube (λ = 0.71073 Å) operated at 2000 watts power. The crystal was mounted on a cryoloop using Paratone N-Exxon oil and placed under a stream of nitrogen. The detector was placed at a distance of 5.009 cm from the crystal. Analysis of the data set showed negligible decay during data collection. The data was corrected for absorption using the SADABS program. The structure was refined using the Bruker SHELXTL Software Package (Version 6.1), and was solved using direct methods until

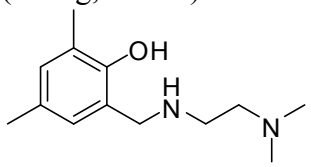
the final anisotropic full-matrix, least squares refinement of F^2 converged.[6] The density, F(000), and extinction coefficient reflect the full formula.

Synthesis of **14**. 2,4-dichloro-6-[[[2-(dimethylamino)ethyl]amino]methyl]-phenol (2.59 g, 68.4% yield)



In a 25 ml round bottom flask equipped with a magnetic stir bar and a reflux condenser, 2.0 mL (15.4 mmol; 1 eq) of *N,N'*-dimethylethylenediamine and 1.14 mL (15.4 mmol, 1 eq) of 37% aqueous formaldehyde were combined with 10.0 mL of methanol. The reaction was allowed to reflux for one hour, after which, 2.50g (15.4 mmol, 1 eq) of 2,4-dichlorophenol was added. After three days of refluxing, the reaction was cooled in an ice bath resulting in the formation of a white precipitate, which was isolated by filtration. FTIR (ATR): 2976, 2949, 2829, 2344 cm^{-1}. ^1H NMR: $CDCl_3$ δ 7.25 (s, 1 H, aromatic H), 6.89 (s, 1H, aromatic H) 3.98 (s, 2H, benzylic H), 2.69 (t, 2H, J = 11.2 Hz, $CCH_2CH_2NMe_2$), 2.45 (t, 2H, J = 11.2 Hz, $CCH_2CH_2NMe_2$), 2.23 (s, 6H, $N(CH_3)_2$). ^{13}C NMR: $CDCl_3$ δ 153.5, 152.0, 129.8, 128.6, 128.4, 126.7, 58.0, 55.9, 52.2, 45.5. HRMS (FAB^+) *m/z*: $[M + H]^+$ calcd for $C_{11}H_{17}N_2OCl_2$ 263.0718; found, 263.0739. MP 127.3-128.7

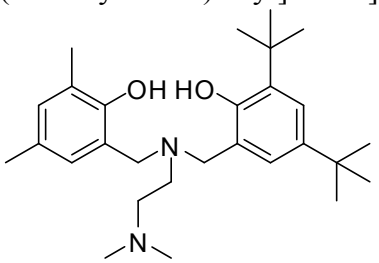
Synthesis of **15**. 2,4-dimethyl-6-[[[2-(dimethylamino)ethyl]amino]methyl]-phenol (1.38 g, 40.4%)



In a 25 ml round bottom flask equipped with a magnetic stir bar and a reflux condenser, 2.0 mL (15.4 mmol; 1 eq) of *N,N'*-dimethylethylenediamine and 1.14 mL (15.4 mmol, 1 eq) of 37% aqueous formaldehyde were combined with 10.0 mL of methanol. The reaction was allowed to reflux for one hour, after which, 1.84 mL (15.4 mmol, 1 eq) of 2,4-dimethylphenol was added. The reaction was allowed to reflux for three days. The solvent was removed by rotary evaporation; the resulting oil was dissolved in 10mL of CH_2Cl_2 and washed with 1 mL HCl. 1.0 M NaOH was added to the aqueous layer until the pH was ~8, and then the aqueous layer was extracted with CH_2Cl_2. The organic layer was dried with magnesium sulfate and the solvent removed by rotary evaporation resulting in an oil that contained both the product and a small amount of the starting *N,N'*-dimethylethylenediamine. The *N,N'*-dimethylethylenediamine. Was removed by Kugelrohr distillation and the product was isolated as an oil. FTIR (ATR): 3683, 3057, 2255 cm^{-1}. ^1H NMR ($CDCl_3$) δ 6.87 (s, 1H), 6.67 (s, 1H) 3.94 (s, 2H, Ar-CH_2-N), 2.72 (t, 2H, *J*=11.6, N-CH_2-CH_2), 2.54 (t, 2H, *J*=11.6, N-CH_2-CH_2), 2.24 (s, 6H, N-$(CH_3)_2$), 2.24 (s, 6H, Ar-CH_3). ^{13}C NMR ($CDCl_3$) δ 154.1, 130.4, 127.4, 126.5, 124.8, 121.8,

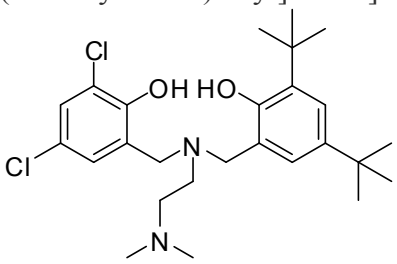
58.3, 52.7, 45.6, 45.5, 20.5, 15.7. HRMS (FAB$^+$) *m/z*: [M + H$^+$]$^+$ calcd for $C_{13}H_{23}N_2O$, 223.1810; found, 223.1780

Synthesis of **6.** 2-[[[[3,5-bis(1,1-dimethylethyl)-2-hydroxyphenyl]methyl][2-(dimethylamino)ethyl]amino]methyl]-4,6-dimethylphenol (0.080 g, 17.8%).



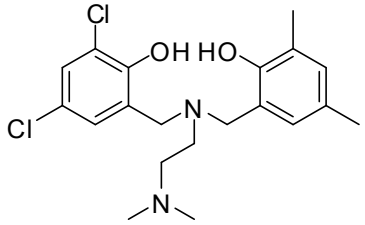
In a 25.0 ml round bottom equipped with a magnetic stir bar and reflux condenser, 0.250 g (1.2 mmol, 1 eq) of **15**, 0.232g (1.2 mmol, 1 eq) of 2,4-di-*t*-butylphenol and 0.089 mL (1.2 mmol, 1 eq) of 37% aqueous formaldehyde were combined with 10.0 ml of methanol and allowed to reflux for four days. The reaction mixture was then cooled in an ice bath, resulting in the formation a white precipitate which was collected by filtration. Spectroscopic data agreed with Groysman *et. al. (Organometallics*, **2004**, *23*, 5291-5299).

Synthesis of **7**. 2-[[[[3,5-bis-chloro-2-hydroxyphenyl]methyl][2-(dimethylamino)ethyl]amino]methyl]-4,6-di-*tert*-butylphenol (0.254 g, 54.7%).



In a 25.0 ml round bottom equipped with a magnetic stir bar and reflux condenser, 0.250 g (1.0 mmol, 1 eq) of **14**, 0.206 g of 2,4 di-*t*-butyl phenol (1.0 mmol, 1 eq) and 0.74 mL (1.0 mmol, 1 eq) of 37% of aqueous formaldehyde were combined with 10.0 ml of methanol and allowed to reflux for four days. The reaction mixture was then cooled in an ice bath, resulting in the formation a white precipitate which was collected by filtration. FTIR (ATR): 2952 cm^{-1}.^{1}H NMR: ($CDCl_3$) δ 7.26 (d, *J* = 2.8 Hz, 1H aromatic H), 7.10 (d, *J* = 2.8 Hz, 1H aromatic H), 6.92 (d, *J* = 2.8 Hz, 1H aromatic H), 6.85 (d, *J* = 2.8 Hz, 1H aromatic H), 3.70 (s, 2H benzylic H), 3.56 (s, 2H benzylic H), 2.63 (s, 4H, $NCH_2CH_2NMe_2$), 2.37 (s, 6H, $N(CH_3)_2$), 1.35 (s 9H, Ph-$C(CH_3)_3$), 1.27 (s 9H, Ph-$C(CH_3)_3$). ^{13}C NMR ($CDCl_3$) δ 153.2, 152.5, 141.0, 136.1, 129.6, 129.1, 125.3, 124.5, 123.7, 123.0, 122.8, 121.1, 58.5, 56.1, 54.6, 49.2, 45.0, 35.1, 34.3, 31.9, 29.7. HRMS (FAB$^+$) *m/z*: [M + H$^+$]$^+$ calculated for $C_{26}H_{39}Cl_2N_2O_2$, 481.2354; found, 481.2389. MP 96.2-96.6

Synthesis of **8**. 2-[[[[3,5-bis-chloro-2-hydroxyphenyl]methyl][2-(dimethylamino)ethyl]amino]methyl]-4,6-dimethylphenol (0.740 g, 49.1 %).



In a 50mL round bottom flask equipped with a magnetic stir bar and condenser, 1.00g (3.8 mmol) of **14**, was combined with 0.283 mL (3.8 mmol) of 37% formaldehyde, 0.455 mL (3.8 mmol) of 2,4-dimethylphenol, and 25 mL of methanol. The mixture was allowed to reflux for 24 h. After the reflux the reaction flask was put into an ice bath which induced crystal formation. The white crystals were collected by vacuum filtration. FTIR (ATR): 2964, 2806 cm^{-1}. ^{1}H NMR: ($CDCl_3$) δ 7.26 (s, 1H aromatic H),6.93 (s, 1H aromatic H), 6.65 (s, 1H aromatic H), 6.66 (s, 1H aromatic H), 3.62 (s, 2H benzylic H), 3.58 (s, 2H benzylic H), 2.60 (s, 4H, $NCH_2CH_2NMe_2$), 2.35 (s, 6H, $N(CH_3)_2$), 2.20 (s 3H, Ph-CH_3), 2.16 (s 3H, Ph-CH_3). ^{13}C NMR ($CDCl_3$) δ 152.5, 152.3, 131.5, 129.5, 128.7, 128.1, 128.0, 125.5, 125.3, 123.0, 122.8, 121.0, 57.0, 56.2, 55.2, 49.2, 45.0, 20.5, 16.1. HRMS (FAB^+) *m/z*: $[M + H^+]^+$ calculated for $C_{20}H_{27}Cl_2N_2O_2$, 397.1450; found, 397.1438. MP: 169.9- 170.3

General Procedure for synthesis of **9-11**.

To a 50 mL Schlenk flask equipped with a magnetic stir bar and reflux condenser, 1.00 mmol of the appropriate ligand **5-7** was combined with 5.0 mL of methanol. Anhydrous $FeCl_3$ (0.162 g, 1.0 mmol) was dissolved in 10.0 mL of methanol and added to the ligand solution resulting in a deep purple solution. Addition of triethylamine (0.28 mL, 2.0 mmol) to the reaction was followed by two hours of reflux. The methanol was then removed *in vacuo* and the resulting purple solid was recrystallized from hot acetonitrile.

Synthesis of **9** (0.408g, 77%.).
Single crystals grown from CH_2Cl_2/Et_2O vapor diffusion. MS (FAB^+) *m/z*: $[M - Cl^-]^+$ calculated for $C_{28}H_{42}FeN_2O_2$ 495.2496; found 495.2570. Elemental Analysis: Calculated for $C_{28}H_{42}ClFeN_2O_2$: C, 63.46; H, 7.99; N, 5.29. Found: C, 63.07; H, 8.06; N, 4.87.

Synthesis of **10** (0.468g, 82%).
Single crystals grown from CH_2Cl_2/Et_2O vapor diffusion. MS (FAB^+) *m/z*: $[M - Cl^-]^+$ calculated for $C_{26}H_{36}Cl_2FeN_2O_2$: 534.1503; found: 534.1530. Elemental Analysis: Calculated for $C_{26}H_{36}Cl_3FeN_2O_2$: C, 54.71; H, 6.36; N, 4.91. Found: C, 54.35; H, 6.40; N, 4.77.

Synthesis of **11** (0.326g, 67%).

Single crystals grown from CH_2Cl_2/Et_2O vapor diffusion. MS (FAB^+) *m/z*: $[M - Cl^-]^+$ calculated for $C_{20}H_{24}Cl_2FeN_2O_2$: 450.0564; found: 450.0558. Elemental Analysis: Calculated for $C_{20}H_{24}Cl_3FeN_2O_2$: C, 49.36; H, 4.97; N, 5.76. Found: C, 49.57; H, 5.36; N, 5.37.

General Procedure for the reaction of **10-12** with O_2 and 3,5-di-*t*-butylcatechol. Compound **10-12** (0.5 mmol), 3,5-di-*t*-butylcatechol (0.11 g, 0.5 mmol), and piperdine (0.10 mL, 1.0 mmol) were combined with 10.0 mL of DMF in a round bottom flask and sealed. The solution was put under an active oxygen atmosphere (bubbling) for 15 minutes followed by a static oxygen atmosphere for 48 hours. Next, 30.0 mL of 2M HCL was added to the reaction mixture and the tan solution was transferred to a separatory funnel. The organic layer was extracted with three 50 mL portions of ether. The ether layer was dried with magnesium sulfate and filtered. The filtrate was concentrated by rotary evaporation and a GC-MS sample was prepared by dilution with $CHCl_3$. Control experiments using $FeCl_3$ instead of **10-12** resulted in no formation of compounds **16-18**.

Table S1: bond lengths and angles and Tau values for Fe(III) complexes

	11	**10**	**9**	**12**	**13**
Fe-Cl	2.292(2)	2.288(3)	2.3001(9)	2.29(1)	2.305(1)
Fe-O(1)	1.878(3)[a]	1.839(3)[c]	1.862(1)[b]	1.868(2)[b]	1.865(2)[d]
Fe-O(2)	1.851(3)[b]	1.875(2)[a]	1.853(1)[c]	1.855(2)[b]	1.845(2)[d]
Fe-N(1)	2.243(4)	2.276(4)	2.278(2)	2.288(2)	2.248(2)
Fe-N(2)	2.158(4)	2.190(4)	2.180(2)	2.152(2)	2.181(3)
Cl-Fe-O(1)	96.47(9)[a]	98.50(7)[c]	96.30(4)[b]	94.3(1)[b]	95.45(8)[d]
Cl-Fe-O(2)	96.8(1)[b]	96.41(8)[a]	98.95(4)[c]	99.4(1)[b]	97.11(7)[d]
Cl-Fe-N(1)	170.9(1)	169.37(6)	168.94(4)	170.1(1)	171.75(8)
Cl-Fe-N(2)	91.1(1)	91.39(6)	91.09(4)	92.2(1)	92.09(8)
O(1)-Fe-O(2)	118.6(1)[a, b]	118.5(1)[a, c]	118.02(6)[b, c]	121.3(1)[b]	125.3(1)[d]
O(1)-Fe-N(1)	88.9(1)[a]	89.29(8)[c]	86.34(6)[b]	85.6(1)[b]	87.9(1)[d]
O(1)-Fe-N(2)	123.4(1)[a]	117.14(9)[c]	123.36(6)[b]	123.0(1)[b]	120.4(1)[d]
O(2)-Fe-N(1)	86.9(1)[b]	86.09(9)[a]	89.19(5)[c]	89.1(1)[b]	87.0(1)[d]
O(2)-Fe-N(2)	116.0(2)[b]	121.8(1)[a]	115.99(6)[c]	113.1(1)[b]	112.1(1)[d]
N(1)-Fe-N(2)	79.9(1)	78.58(8)	78.56(5)	79.7(1)	79.7(1)
Fe-O(1)-C(1)	131.1(3)[a]	127.2(2)[c]	126.5(1)[b]	122.15[b]	129.3(2)[d]
Fe-O(2)-C(2)	130.3(3)[b]	127.5(2)[a]	126.1(1)[c]	122.05[b]	138.3(2)[d]
Tau	0.79	0.79	0.76	0.79	0.77

[a]Oxygen from 2,4-dichlorophenol, [b]Oxygen from 2,4-dimethyphenol, [c]Oxygen from 2,4-di-*t*-butylphenol, [d]Oxygen from 2-*t*-butyl-4-methylphenol. $\tau = (\beta-\alpha)/60$ where β is equal to the largest angle (θ between two axial ligands) and α is equal to second largest angle (θ between two equatorial ligands), from Addison, A. W.; Rao, T. N.; Reedijk, J.; Van Rijn, J.; Vershcoor, G. C. *J. chem.. Soc., Dalton trans.* **1984**, 1349.

Table 1. Crystal data and structure refinement for **9**. FeCl(DMDTB-DMEN).

Identification code	p21c	
Empirical formula	C28 H42 Cl Fe N2 O2	
Formula weight	529.94	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 11.477(4) Å	α= 90°.
	b = 17.017(6) Å	β= 96.999(6)°.
	c = 14.198(5) Å	γ = 90°.
Volume	2752.2(17) $Å^3$	
Z	4	
Density (calculated)	1.279 Mg/m^3	
Absorption coefficient	0.672 mm^{-1}	
F(000)	1132	
Crystal size	0.26 x 0.19 x 0.18 mm^3	
Theta range for data collection	1.79 to 27.00°.	
Index ranges	-14<=h<=14, -20<=k<=21, -18<=l<=18	
Reflections collected	22950	
Independent reflections	5984 [R(int) = 0.0482]	
Completeness to theta = 27.00°	99.6 %	
Absorption correction	SADABS	
Max. and min. transmission	0.8887 and 0.8448	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5984 / 0 / 317	
Goodness-of-fit on F^2	1.004	
Final R indices [I>2sigma(I)]	R1 = 0.0379, wR2 = 0.0933	
R indices (all data)	R1 = 0.0494, wR2 = 0.0980	
Largest diff. peak and hole	0.465 and -0.294 e.$Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **9**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	7419(2)	2419(1)	1811(1)	18(1)
C(2)	8598(2)	2244(1)	1725(1)	22(1)
C(3)	9423(2)	2846(1)	1861(1)	22(1)
C(4)	9123(2)	3612(1)	2074(1)	20(1)
C(5)	7949(2)	3772(1)	2149(1)	18(1)
C(6)	7098(2)	3187(1)	2023(1)	16(1)
C(7)	8927(2)	1420(1)	1485(2)	36(1)
C(8)	10040(2)	4248(1)	2217(1)	25(1)
C(9)	5825(2)	3354(1)	2076(1)	17(1)
C(10)	3359(2)	1977(1)	1473(1)	16(1)
C(11)	2685(2)	1725(1)	627(1)	16(1)
C(12)	2120(2)	2313(1)	52(1)	18(1)
C(13)	2175(2)	3114(1)	266(1)	17(1)
C(14)	2804(2)	3332(1)	1128(1)	17(1)
C(15)	3380(2)	2773(1)	1734(1)	16(1)
C(16)	2591(2)	854(1)	346(1)	18(1)
C(17)	3788(2)	535(1)	165(1)	25(1)
C(18)	2135(2)	370(1)	1142(1)	24(1)
C(19)	1740(2)	729(1)	-560(2)	29(1)
C(20)	1533(2)	3706(1)	-429(1)	18(1)
C(21)	1904(2)	3599(1)	-1429(1)	24(1)
C(22)	204(2)	3574(1)	-490(2)	24(1)
C(23)	1808(2)	4554(1)	-115(1)	24(1)
C(24)	4011(2)	3006(1)	2684(1)	17(1)
C(25)	5844(2)	3035(1)	3754(1)	18(1)
C(26)	5602(2)	2379(1)	4419(1)	18(1)
C(27)	5464(2)	964(1)	4567(1)	23(1)
C(28)	7285(2)	1545(1)	4225(1)	22(1)
Cl(1)	6045(1)	230(1)	2528(1)	25(1)
Fe(1)	5564(1)	1542(1)	2513(1)	15(1)
N(1)	5305(1)	2848(1)	2776(1)	15(1)

N(2)	5983(1)	1611(1)	4050(1)	17(1)
O(1)	6597(1)	1851(1)	1668(1)	21(1)
O(2)	3985(1)	1463(1)	2061(1)	17(1)

Table 3. Bond lengths [Å] and angles [°] for **9**.

C(1)-O(1)	1.349(2)
C(1)-C(6)	1.400(3)
C(1)-C(2)	1.404(3)
C(2)-C(3)	1.392(3)
C(2)-C(7)	1.502(3)
C(3)-C(4)	1.391(3)
C(4)-C(5)	1.391(3)
C(4)-C(8)	1.507(3)
C(5)-C(6)	1.392(3)
C(6)-C(9)	1.499(3)
C(9)-N(1)	1.493(2)
C(10)-O(2)	1.353(2)
C(10)-C(15)	1.403(3)
C(10)-C(11)	1.414(3)
C(11)-C(12)	1.400(3)
C(11)-C(16)	1.535(2)
C(12)-C(13)	1.397(3)
C(13)-C(14)	1.392(3)
C(13)-C(20)	1.535(3)
C(14)-C(15)	1.394(3)
C(15)-C(24)	1.505(2)
C(16)-C(17)	1.528(3)
C(16)-C(19)	1.533(3)
C(16)-C(18)	1.539(3)
C(20)-C(23)	1.532(3)
C(20)-C(22)	1.533(3)
C(20)-C(21)	1.542(3)
C(24)-N(1)	1.499(2)
C(25)-N(1)	1.485(2)
C(25)-C(26)	1.510(3)
C(26)-N(2)	1.494(2)
C(27)-N(2)	1.487(2)
C(28)-N(2)	1.488(2)
Cl(1)-Fe(1)	2.3001(9)

Fe(1)-O(2)	1.8533(14)
Fe(1)-O(1)	1.8619(14)
Fe(1)-N(2)	2.1799(17)
Fe(1)-N(1)	2.2782(16)

O(1)-C(1)-C(6)	120.16(17)
O(1)-C(1)-C(2)	119.92(17)
C(6)-C(1)-C(2)	119.91(17)
C(3)-C(2)-C(1)	118.58(17)
C(3)-C(2)-C(7)	122.13(18)
C(1)-C(2)-C(7)	119.28(18)
C(2)-C(3)-C(4)	122.44(18)
C(5)-C(4)-C(3)	117.94(18)
C(5)-C(4)-C(8)	121.07(18)
C(3)-C(4)-C(8)	120.99(18)
C(4)-C(5)-C(6)	121.39(18)
C(5)-C(6)-C(1)	119.73(17)
C(5)-C(6)-C(9)	122.09(16)
C(1)-C(6)-C(9)	118.16(16)
N(1)-C(9)-C(6)	113.30(14)
O(2)-C(10)-C(15)	118.10(16)
O(2)-C(10)-C(11)	121.42(16)
C(15)-C(10)-C(11)	120.47(16)
C(12)-C(11)-C(10)	116.45(16)
C(12)-C(11)-C(16)	121.70(16)
C(10)-C(11)-C(16)	121.85(16)
C(11)-C(12)-C(13)	124.39(17)
C(14)-C(13)-C(12)	117.14(16)
C(14)-C(13)-C(20)	123.17(16)
C(12)-C(13)-C(20)	119.68(16)
C(13)-C(14)-C(15)	121.11(17)
C(14)-C(15)-C(10)	120.26(17)
C(14)-C(15)-C(24)	120.91(16)
C(10)-C(15)-C(24)	118.81(16)
C(17)-C(16)-C(19)	107.66(16)
C(17)-C(16)-C(11)	110.48(15)

C(19)-C(16)-C(11)	111.66(15)
C(17)-C(16)-C(18)	109.09(15)
C(19)-C(16)-C(18)	107.65(16)
C(11)-C(16)-C(18)	110.21(15)
C(23)-C(20)-C(13)	111.43(15)
C(23)-C(20)-C(22)	108.83(16)
C(13)-C(20)-C(22)	109.62(15)
C(23)-C(20)-C(21)	108.00(16)
C(13)-C(20)-C(21)	110.52(15)
C(22)-C(20)-C(21)	108.37(16)
N(1)-C(24)-C(15)	113.15(15)
N(1)-C(25)-C(26)	109.79(15)
N(2)-C(26)-C(25)	110.12(15)
O(2)-Fe(1)-O(1)	118.02(6)
O(2)-Fe(1)-N(2)	115.99(6)
O(1)-Fe(1)-N(2)	123.36(6)
O(2)-Fe(1)-N(1)	89.19(5)
O(1)-Fe(1)-N(1)	86.34(6)
N(2)-Fe(1)-N(1)	78.56(5)
O(2)-Fe(1)-Cl(1)	98.95(4)
O(1)-Fe(1)-Cl(1)	96.30(4)
N(2)-Fe(1)-Cl(1)	91.09(4)
N(1)-Fe(1)-Cl(1)	168.94(4)
C(25)-N(1)-C(9)	110.10(14)
C(25)-N(1)-C(24)	109.77(14)
C(9)-N(1)-C(24)	108.13(13)
C(25)-N(1)-Fe(1)	108.32(10)
C(9)-N(1)-Fe(1)	112.49(11)
C(24)-N(1)-Fe(1)	108.00(10)
C(27)-N(2)-C(28)	108.45(14)
C(27)-N(2)-C(26)	108.81(14)
C(28)-N(2)-C(26)	109.75(14)
C(27)-N(2)-Fe(1)	113.72(12)
C(28)-N(2)-Fe(1)	104.94(11)
C(26)-N(2)-Fe(1)	111.04(11)
C(1)-O(1)-Fe(1)	126.45(11)

C(10)-O(2)-Fe(1) 126.11(11)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **9**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	22(1)	18(1)	13(1)	0(1)	4(1)	-1(1)
C(2)	25(1)	20(1)	23(1)	0(1)	6(1)	5(1)
C(3)	18(1)	27(1)	22(1)	6(1)	5(1)	6(1)
C(4)	21(1)	24(1)	13(1)	3(1)	1(1)	-2(1)
C(5)	24(1)	17(1)	14(1)	0(1)	3(1)	1(1)
C(6)	20(1)	17(1)	12(1)	1(1)	2(1)	2(1)
C(7)	32(1)	24(1)	55(2)	-3(1)	18(1)	8(1)
C(8)	21(1)	31(1)	23(1)	1(1)	1(1)	-3(1)
C(9)	18(1)	14(1)	18(1)	1(1)	4(1)	1(1)
C(10)	17(1)	16(1)	15(1)	2(1)	3(1)	2(1)
C(11)	16(1)	16(1)	17(1)	-1(1)	3(1)	-1(1)
C(12)	17(1)	21(1)	15(1)	0(1)	0(1)	-1(1)
C(13)	14(1)	19(1)	18(1)	1(1)	4(1)	2(1)
C(14)	16(1)	15(1)	20(1)	-2(1)	4(1)	0(1)
C(15)	15(1)	17(1)	16(1)	0(1)	3(1)	0(1)
C(16)	22(1)	14(1)	17(1)	-1(1)	1(1)	0(1)
C(17)	29(1)	22(1)	25(1)	-4(1)	7(1)	2(1)
C(18)	29(1)	18(1)	27(1)	-2(1)	7(1)	-3(1)
C(19)	38(1)	19(1)	26(1)	-5(1)	-9(1)	-1(1)
C(20)	16(1)	18(1)	19(1)	2(1)	1(1)	2(1)
C(21)	26(1)	26(1)	20(1)	5(1)	4(1)	5(1)
C(22)	18(1)	26(1)	26(1)	4(1)	0(1)	3(1)
C(23)	26(1)	19(1)	25(1)	4(1)	-3(1)	3(1)
C(24)	18(1)	16(1)	17(1)	-1(1)	2(1)	2(1)
C(25)	19(1)	21(1)	14(1)	-3(1)	0(1)	0(1)
C(26)	21(1)	22(1)	11(1)	-1(1)	0(1)	3(1)
C(27)	25(1)	24(1)	22(1)	6(1)	6(1)	1(1)

C(28)	18(1)	25(1)	22(1)	-1(1)	-1(1)	4(1)
Cl(1)	33(1)	15(1)	25(1)	0(1)	1(1)	6(1)
Fe(1)	17(1)	14(1)	14(1)	0(1)	1(1)	1(1)
N(1)	17(1)	15(1)	12(1)	0(1)	1(1)	1(1)
N(2)	15(1)	18(1)	16(1)	1(1)	1(1)	2(1)
O(1)	25(1)	17(1)	21(1)	-3(1)	8(1)	-2(1)
O(2)	18(1)	14(1)	17(1)	2(1)	-1(1)	2(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **9**.

	x	y	z	U(eq)
H(3)	10222	2729	1806	27
H(5)	7724	4293	2289	22
H(7A)	8575	1293	839	54
H(7B)	8637	1052	1934	54
H(7C)	9783	1378	1525	54
H(8A)	9975	4590	1658	38
H(8B)	10823	4009	2309	38
H(8C)	9921	4560	2778	38
H(9A)	5737	3912	2251	20
H(9B)	5381	3274	1440	20
H(12)	1669	2156	-523	21
H(14)	2841	3870	1305	20
H(17A)	4076	834	-350	38
H(17B)	3713	-20	-15	38
H(17C)	4344	587	743	38
H(18A)	2076	-184	954	36
H(18B)	1359	564	1252	36
H(18C)	2680	422	1726	36
H(19A)	2029	1010	-1088	43
H(19B)	963	931	-465	43
H(19C)	1683	167	-708	43
H(21A)	1481	3977	-1866	36
H(21B)	1717	3064	-1653	36
H(21C)	2751	3689	-1405	36
H(22A)	-49	3648	138	35
H(22B)	16	3038	-711	35
H(22C)	-203	3951	-937	35
H(23A)	1419	4917	-589	35
H(23B)	2658	4639	-54	35
H(23C)	1523	4648	498	35

H(24A)	3884	3573	2786	20
H(24B)	3668	2714	3187	20
H(25A)	5515	3534	3966	22
H(25B)	6702	3103	3762	22
H(26A)	6030	2479	5056	22
H(26B)	4752	2360	4479	22
H(27A)	5788	979	5238	35
H(27B)	5652	458	4293	35
H(27C)	4610	1029	4511	35
H(28A)	7542	1602	4906	33
H(28B)	7641	1960	3876	33
H(28C)	7528	1030	4009	33

Table 1. Crystal data and structure refinement for **10**.

Identification code	p21c	
Empirical formula	C26 H37 Cl3 Fe N2 O2	
Formula weight	571.78	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 11.359(15) Å	α= 90°.
	b = 16.93(2) Å	β= 96.78(2)°.
	c = 14.284(18) Å	γ = 90°.
Volume	2728(6) $Å^3$	
Z	4	
Density (calculated)	1.392 Mg/m^3	
Absorption coefficient	0.872 mm^{-1}	
F(000)	1200	
Crystal size	0.90 x 0.80 x 0.10 mm^3	
Theta range for data collection	1.81 to 27.00°.	
Index ranges	-14<=h<=14, -21<=k<=21, -18<=l<=18	
Reflections collected	21398	
Independent reflections	5926 [R(int) = 0.0862]	
Completeness to theta = 27.00°	99.5 %	
Absorption correction	SADABS	
Max. and min. transmission	0.9178 and 0.5073	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5926 / 0 / 315	
Goodness-of-fit on F^2	0.929	
Final R indices [I>2sigma(I)]	R1 = 0.0478, wR2 = 0.0999	
R indices (all data)	R1 = 0.0771, wR2 = 0.1102	
Largest diff. peak and hole	0.450 and -0.446 e.$Å^{-3}$	

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

	x	y	z	U(eq)
C(1)	1670(2)	1978(2)	3538(2)	15(1)
C(2)	2342(2)	1720(2)	4377(2)	16(1)
C(3)	2913(2)	2306(2)	4954(2)	17(1)
C(4)	2861(2)	3116(2)	4744(2)	15(1)
C(5)	2232(2)	3337(2)	3884(2)	15(1)
C(6)	1649(2)	2778(2)	3277(2)	14(1)
C(7)	2449(2)	844(2)	4655(2)	18(1)
C(8)	3326(3)	720(2)	5552(2)	30(1)
C(9)	1246(2)	524(2)	4848(2)	24(1)
C(10)	2896(3)	363(2)	3855(2)	24(1)
C(11)	3513(2)	3709(2)	5443(2)	16(1)
C(12)	4854(2)	3567(2)	5513(2)	23(1)
C(13)	3128(3)	3602(2)	6438(2)	21(1)
C(14)	3262(3)	4563(2)	5130(2)	23(1)
C(15)	1023(2)	3007(2)	2325(2)	16(1)
C(16)	-812(2)	3353(2)	2922(2)	16(1)
C(17)	-2092(2)	3190(2)	2964(2)	14(1)
C(18)	-2936(2)	3792(2)	2861(2)	17(1)
C(19)	-4113(2)	3624(2)	2936(2)	18(1)
C(20)	-4475(3)	2869(2)	3122(2)	21(1)
C(21)	-3636(2)	2273(2)	3228(2)	19(1)
C(22)	-2439(2)	2419(2)	3155(2)	17(1)
C(23)	-2249(2)	1540(2)	764(2)	22(1)
C(24)	-416(3)	963(2)	421(2)	24(1)
C(25)	-819(2)	3041(2)	1242(2)	17(1)
C(26)	-560(2)	2383(2)	576(2)	17(1)
Cl(1)	-989(1)	215(1)	2449(1)	24(1)
Cl(2)	-5156(1)	4381(1)	2793(1)	23(1)
Cl(3)	-4062(1)	1320(1)	3468(1)	36(1)
Fe(1)	-535(1)	1532(1)	2471(1)	15(1)
N(1)	-283(2)	2846(1)	2223(2)	14(1)

N(2)	-944(2)	1609(1)	936(2)	17(1)
O(1)	1038(2)	1460(1)	2947(1)	16(1)
O(2)	-1632(2)	1837(1)	3285(1)	19(1)

Table 3. Bond lengths [Å] and angles [°] for **10**.

C(1)-O(1)	1.363(3)
C(1)-C(6)	1.403(4)
C(1)-C(2)	1.413(4)
C(2)-C(3)	1.399(4)
C(2)-C(7)	1.536(4)
C(3)-C(4)	1.403(4)
C(4)-C(5)	1.397(4)
C(4)-C(11)	1.543(4)
C(5)-C(6)	1.397(4)
C(6)-C(15)	1.509(4)
C(7)-C(9)	1.526(4)
C(7)-C(10)	1.537(4)
C(7)-C(8)	1.541(4)
C(11)-C(14)	1.531(4)
C(11)-C(12)	1.533(4)
C(11)-C(13)	1.546(4)
C(15)-N(1)	1.499(4)
C(16)-C(17)	1.489(4)
C(16)-N(1)	1.495(3)
C(17)-C(18)	1.395(4)
C(17)-C(22)	1.399(4)
C(18)-C(19)	1.384(4)
C(19)-C(20)	1.378(4)
C(19)-Cl(2)	1.742(3)
C(20)-C(21)	1.384(4)
C(21)-C(22)	1.398(4)
C(21)-Cl(3)	1.731(4)
C(22)-O(2)	1.343(3)
C(23)-N(2)	1.478(4)
C(24)-N(2)	1.485(4)
C(25)-N(1)	1.498(4)
C(25)-C(26)	1.515(4)
C(26)-N(2)	1.492(4)
Cl(1)-Fe(1)	2.288(3)

Fe(1)-O(1)	1.839(3)
Fe(1)-O(2)	1.875(2)
Fe(1)-N(2)	2.190(4)
Fe(1)-N(1)	2.276(4)

O(1)-C(1)-C(6)	117.8(2)
O(1)-C(1)-C(2)	121.2(2)
C(6)-C(1)-C(2)	121.0(2)
C(3)-C(2)-C(1)	116.5(3)
C(3)-C(2)-C(7)	121.0(3)
C(1)-C(2)-C(7)	122.5(2)
C(2)-C(3)-C(4)	124.2(3)
C(5)-C(4)-C(3)	117.1(2)
C(5)-C(4)-C(11)	123.4(3)
C(3)-C(4)-C(11)	119.5(2)
C(4)-C(5)-C(6)	121.2(3)
C(5)-C(6)-C(1)	119.8(2)
C(5)-C(6)-C(15)	121.5(3)
C(1)-C(6)-C(15)	118.6(2)
C(9)-C(7)-C(2)	110.2(2)
C(9)-C(7)-C(10)	109.1(2)
C(2)-C(7)-C(10)	110.1(2)
C(9)-C(7)-C(8)	107.8(2)
C(2)-C(7)-C(8)	111.7(2)
C(10)-C(7)-C(8)	107.9(2)
C(14)-C(11)-C(12)	108.5(2)
C(14)-C(11)-C(4)	111.6(2)
C(12)-C(11)-C(4)	109.7(2)
C(14)-C(11)-C(13)	108.4(2)
C(12)-C(11)-C(13)	107.9(2)
C(4)-C(11)-C(13)	110.7(2)
N(1)-C(15)-C(6)	113.3(2)
C(17)-C(16)-N(1)	113.0(2)
C(18)-C(17)-C(22)	119.8(3)
C(18)-C(17)-C(16)	121.4(3)
C(22)-C(17)-C(16)	118.7(2)

C(19)-C(18)-C(17)	119.9(3)
C(20)-C(19)-C(18)	121.3(3)
C(20)-C(19)-Cl(2)	119.4(2)
C(18)-C(19)-Cl(2)	119.3(2)
C(19)-C(20)-C(21)	118.6(3)
C(20)-C(21)-C(22)	121.8(3)
C(20)-C(21)-Cl(3)	119.7(2)
C(22)-C(21)-Cl(3)	118.4(2)
O(2)-C(22)-C(21)	121.0(3)
O(2)-C(22)-C(17)	120.5(3)
C(21)-C(22)-C(17)	118.5(3)
N(1)-C(25)-C(26)	109.8(2)
N(2)-C(26)-C(25)	110.0(2)
O(1)-Fe(1)-O(2)	118.46(12)
O(1)-Fe(1)-N(2)	117.14(9)
O(2)-Fe(1)-N(2)	121.76(11)
O(1)-Fe(1)-N(1)	89.29(8)
O(2)-Fe(1)-N(1)	86.09(9)
N(2)-Fe(1)-N(1)	78.58(8)
O(1)-Fe(1)-Cl(1)	98.50(7)
O(2)-Fe(1)-Cl(1)	96.41(8)
N(2)-Fe(1)-Cl(1)	91.39(6)
N(1)-Fe(1)-Cl(1)	169.37(6)
C(16)-N(1)-C(25)	110.3(2)
C(16)-N(1)-C(15)	107.6(2)
C(25)-N(1)-C(15)	109.8(2)
C(16)-N(1)-Fe(1)	112.98(17)
C(25)-N(1)-Fe(1)	108.42(16)
C(15)-N(1)-Fe(1)	107.71(15)
C(23)-N(2)-C(24)	108.4(2)
C(23)-N(2)-C(26)	109.9(2)
C(24)-N(2)-C(26)	108.9(2)
C(23)-N(2)-Fe(1)	104.58(16)
C(24)-N(2)-Fe(1)	113.68(18)
C(26)-N(2)-Fe(1)	111.20(16)
C(1)-O(1)-Fe(1)	127.16(17)

C(22)-O(2)-Fe(1) 127.53(17)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **10**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	15(2)	15(2)	15(1)	-2(1)	3(1)	0(1)
C(2)	18(2)	15(2)	15(2)	0(1)	4(1)	1(1)
C(3)	15(2)	21(2)	14(2)	1(1)	0(1)	2(1)
C(4)	13(1)	16(2)	18(2)	0(1)	4(1)	-2(1)
C(5)	16(2)	11(2)	19(2)	1(1)	2(1)	0(1)
C(6)	15(2)	15(2)	11(1)	0(1)	2(1)	1(1)
C(7)	20(2)	16(2)	17(2)	1(1)	2(1)	0(1)
C(8)	42(2)	14(2)	29(2)	4(1)	-10(2)	0(1)
C(9)	29(2)	24(2)	21(2)	4(1)	8(1)	-2(1)
C(10)	30(2)	20(2)	23(2)	3(1)	7(1)	7(1)
C(11)	16(2)	19(2)	15(2)	-2(1)	1(1)	1(1)
C(12)	17(2)	29(2)	22(2)	-3(1)	3(1)	-3(1)
C(13)	22(2)	24(2)	16(2)	-6(1)	1(1)	-7(1)
C(14)	26(2)	19(2)	22(2)	-4(1)	-1(1)	-3(1)
C(15)	20(2)	15(2)	15(2)	1(1)	3(1)	-3(1)
C(16)	20(2)	12(2)	16(2)	-1(1)	4(1)	-2(1)
C(17)	18(2)	15(2)	11(1)	0(1)	3(1)	-2(1)
C(18)	25(2)	14(2)	14(2)	1(1)	3(1)	-1(1)
C(19)	20(2)	21(2)	13(1)	-3(1)	1(1)	4(1)
C(20)	19(2)	23(2)	20(2)	-4(1)	3(1)	-3(1)
C(21)	20(2)	21(2)	17(2)	1(1)	7(1)	-4(1)
C(22)	21(2)	19(2)	10(1)	-1(1)	4(1)	3(1)
C(23)	16(2)	30(2)	20(2)	-1(1)	-1(1)	-4(1)
C(24)	31(2)	20(2)	20(2)	-6(1)	6(1)	0(1)
C(25)	20(2)	17(2)	13(1)	4(1)	-1(1)	-2(1)
C(26)	17(2)	21(2)	12(1)	0(1)	1(1)	0(1)
Cl(1)	32(1)	14(1)	25(1)	0(1)	1(1)	-4(1)

Cl(2)	24(1)	24(1)	22(1)	-1(1)	0(1)	7(1)
Cl(3)	29(1)	21(1)	59(1)	8(1)	14(1)	-4(1)
Fe(1)	18(1)	14(1)	14(1)	0(1)	1(1)	-1(1)
N(1)	17(1)	16(1)	9(1)	-2(1)	2(1)	-2(1)
N(2)	18(1)	15(1)	17(1)	-1(1)	2(1)	-1(1)
O(1)	18(1)	14(1)	16(1)	-3(1)	0(1)	-1(1)
O(2)	23(1)	15(1)	21(1)	4(1)	8(1)	5(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **10**.

	x	y	z	U(eq)
H(3)	3364	2146	5525	20
H(5)	2200	3879	3708	18
H(8A)	4109	921	5446	44
H(8B)	3384	155	5700	44
H(8C)	3043	1004	6079	44
H(9A)	941	842	5339	36
H(9B)	1334	-26	5061	36
H(9C)	691	548	4270	36
H(10A)	2312	389	3294	36
H(10B)	3010	-188	4055	36
H(10C)	3652	582	3709	36
H(12A)	5131	3676	4902	34
H(12B)	5027	3016	5689	34
H(12C)	5261	3917	5993	34
H(13A)	3555	3981	6872	31
H(13B)	3313	3063	6660	31
H(13C)	2273	3694	6412	31
H(14A)	3683	4923	5591	34
H(14B)	2408	4665	5087	34
H(14C)	3535	4647	4512	34
H(15A)	1151	3577	2221	20
H(15B)	1381	2712	1830	20
H(16A)	-372	3267	3554	19
H(16B)	-716	3915	2753	19
H(18)	-2702	4317	2739	21
H(20)	-5285	2760	3176	25
H(23A)	-2505	1586	87	34
H(23B)	-2612	1963	1101	34
H(23C)	-2494	1026	990	34
H(24A)	-615	453	685	35

H(24B)	448	1025	485	35
H(24C)	-731	984	-248	35
H(25A)	-484	3543	1038	20
H(25B)	-1686	3107	1228	20
H(26A)	-987	2485	-58	20
H(26B)	300	2368	520	20

Table 1. Crystal data and structure refinement for **11**·1/2 CH_2Cl_2.

Identification code	p21c	
Empirical formula	C20.50 H25 Cl4 Fe N2 O2	
Formula weight	529.07	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 8.471(6) Å	α= 90°.
	b = 24.118(16) Å	β= 109.195(13)°.
	c = 12.526(9) Å	γ = 90°.
Volume	2417(3) $Å^3$	
Z	4	
Density (calculated)	1.454 Mg/m^3	
Absorption coefficient	1.085 mm^{-1}	
F(000)	1088	
Crystal size	0.12 x 0.09 x 0.03 mm^3	
Theta range for data collection	1.69 to 27.00°.	
Index ranges	-10<=h<=10, -30<=k<=30, -15<=l<=15	
Reflections collected	20152	
Independent reflections	5257 [R(int) = 0.1241]	
Completeness to theta = 27.00°	99.8 %	
Absorption correction	SADABS	
Max. and min. transmission	0.9682 and 0.8808	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5257 / 0 / 257	
Goodness-of-fit on F^2	0.893	
Final R indices [I>2sigma(I)]	R1 = 0.0634, wR2 = 0.1355	
R indices (all data)	R1 = 0.1282, wR2 = 0.1561	
Largest diff. peak and hole	0.876 and -0.740 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **11**·1/2 CH_2Cl_2. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	8593(5)	1499(2)	6479(4)	28(1)
C(2)	8463(5)	982(2)	6954(4)	28(1)
C(3)	9799(5)	625(2)	7341(4)	31(1)
C(4)	11308(5)	778(2)	7247(4)	32(1)
C(5)	11500(5)	1284(2)	6751(4)	28(1)
C(6)	10139(5)	1636(2)	6362(4)	27(1)
C(7)	10229(5)	2151(2)	5725(4)	30(1)
C(8)	10925(5)	2744(2)	7403(4)	25(1)
C(9)	8923(5)	3345(2)	7963(4)	25(1)
C(10)	8534(5)	3829(2)	8475(4)	25(1)
C(11)	9799(6)	4206(2)	8954(4)	30(1)
C(12)	11425(6)	4128(2)	8955(4)	29(1)
C(13)	11777(5)	3656(2)	8443(4)	29(1)
C(14)	10538(5)	3265(2)	7946(4)	24(1)
C(15)	6752(5)	3909(2)	8493(4)	29(1)
C(16)	12813(5)	4547(2)	9533(4)	33(1)
C(17)	9899(5)	3156(2)	5509(4)	29(1)
C(18)	8418(5)	3155(2)	4421(4)	34(1)
C(19)	5464(6)	2945(2)	3641(4)	38(1)
C(20)	6379(6)	3697(2)	4996(4)	40(1)
Cl(1)	4260(2)	2635(1)	5808(1)	42(1)
Cl(2)	6525(1)	788(1)	7038(1)	39(1)
Cl(3)	13045(2)	341(1)	7748(1)	43(1)
Fe(1)	7084(1)	2608(1)	6133(1)	28(1)
N(1)	9764(4)	2673(1)	6216(3)	25(1)
N(2)	6815(4)	3132(2)	4687(3)	33(1)
O(2)	7267(3)	1833(1)	6098(3)	30(1)
O(1)	7722(4)	2961(1)	7522(3)	31(1)

Table 3. Bond lengths [Å] and angles [°] for **11**·1/2 CH_2Cl_2.

C(1)-O(2)	1.337(5)
C(1)-C(2)	1.400(6)
C(1)-C(6)	1.404(6)
C(2)-C(3)	1.377(6)
C(2)-Cl(2)	1.743(4)
C(3)-C(4)	1.373(6)
C(4)-C(5)	1.402(6)
C(4)-Cl(3)	1.750(5)
C(5)-C(6)	1.386(6)
C(6)-C(7)	1.492(6)
C(7)-N(1)	1.507(5)
C(8)-N(1)	1.498(5)
C(8)-C(14)	1.517(6)
C(9)-O(1)	1.352(5)
C(9)-C(14)	1.389(6)
C(9)-C(10)	1.421(6)
C(10)-C(11)	1.383(6)
C(10)-C(15)	1.529(6)
C(11)-C(12)	1.389(6)
C(12)-C(13)	1.387(6)
C(12)-C(16)	1.538(6)
C(13)-C(14)	1.396(6)
C(17)-N(1)	1.491(5)
C(17)-C(18)	1.519(6)
C(18)-N(2)	1.502(5)
C(19)-N(2)	1.497(5)
C(20)-N(2)	1.498(6)
Cl(1)-Fe(1)	2.292(2)
Fe(1)-O(1)	1.851(3)
Fe(1)-O(2)	1.878(3)
Fe(1)-N(2)	2.158(4)
Fe(1)-N(1)	2.243(4)
O(2)-C(1)-C(2)	120.9(4)

O(2)-C(1)-C(6)	121.6(4)
C(2)-C(1)-C(6)	117.4(4)
C(3)-C(2)-C(1)	122.4(4)
C(3)-C(2)-Cl(2)	119.4(4)
C(1)-C(2)-Cl(2)	118.2(3)
C(4)-C(3)-C(2)	118.8(5)
C(3)-C(4)-C(5)	121.3(4)
C(3)-C(4)-Cl(3)	120.3(4)
C(5)-C(4)-Cl(3)	118.4(3)
C(6)-C(5)-C(4)	119.0(4)
C(5)-C(6)-C(1)	121.0(4)
C(5)-C(6)-C(7)	121.1(4)
C(1)-C(6)-C(7)	117.7(4)
C(6)-C(7)-N(1)	114.3(4)
N(1)-C(8)-C(14)	111.8(3)
O(1)-C(9)-C(14)	120.7(4)
O(1)-C(9)-C(10)	119.5(4)
C(14)-C(9)-C(10)	119.8(4)
C(11)-C(10)-C(9)	118.3(4)
C(11)-C(10)-C(15)	122.6(4)
C(9)-C(10)-C(15)	119.1(4)
C(10)-C(11)-C(12)	122.6(4)
C(13)-C(12)-C(11)	118.3(4)
C(13)-C(12)-C(16)	120.6(4)
C(11)-C(12)-C(16)	121.1(4)
C(12)-C(13)-C(14)	121.1(4)
C(9)-C(14)-C(13)	120.0(4)
C(9)-C(14)-C(8)	118.8(4)
C(13)-C(14)-C(8)	121.2(4)
N(1)-C(17)-C(18)	109.2(4)
N(2)-C(18)-C(17)	110.0(4)
O(1)-Fe(1)-O(2)	118.61(14)
O(1)-Fe(1)-N(2)	115.98(15)
O(2)-Fe(1)-N(2)	123.35(14)
O(1)-Fe(1)-N(1)	86.94(13)
O(2)-Fe(1)-N(1)	88.88(12)

N(2)-Fe(1)-N(1)	79.88(13)
O(1)-Fe(1)-Cl(1)	96.83(10)
O(2)-Fe(1)-Cl(1)	96.47(9)
N(2)-Fe(1)-Cl(1)	91.05(10)
N(1)-Fe(1)-Cl(1)	170.93(10)
C(17)-N(1)-C(8)	110.3(3)
C(17)-N(1)-C(7)	109.2(3)
C(8)-N(1)-C(7)	109.0(3)
C(17)-N(1)-Fe(1)	107.5(2)
C(8)-N(1)-Fe(1)	112.2(3)
C(7)-N(1)-Fe(1)	108.6(3)
C(19)-N(2)-C(20)	108.5(4)
C(19)-N(2)-C(18)	107.7(4)
C(20)-N(2)-C(18)	109.9(4)
C(19)-N(2)-Fe(1)	113.5(3)
C(20)-N(2)-Fe(1)	106.4(3)
C(18)-N(2)-Fe(1)	110.8(3)
C(1)-O(2)-Fe(1)	131.1(3)
C(9)-O(1)-Fe(1)	130.3(3)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **11**·1/2 CH_2Cl_2. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	25(3)	31(3)	26(3)	-8(2)	5(2)	-6(2)
C(2)	24(2)	32(3)	31(3)	-15(2)	12(2)	-6(2)
C(3)	36(3)	25(3)	33(3)	-7(2)	12(2)	-7(2)
C(4)	24(3)	33(3)	35(3)	-11(2)	4(2)	-2(2)
C(5)	24(2)	33(3)	26(3)	-10(2)	8(2)	-5(2)
C(6)	28(3)	29(3)	23(3)	-9(2)	7(2)	-3(2)
C(7)	21(2)	37(3)	34(3)	-6(2)	11(2)	2(2)
C(8)	25(2)	22(3)	24(3)	-1(2)	5(2)	3(2)
C(9)	27(2)	25(3)	24(3)	3(2)	9(2)	-1(2)
C(10)	31(3)	24(3)	22(3)	1(2)	12(2)	6(2)
C(11)	39(3)	19(3)	28(3)	1(2)	6(2)	1(2)
C(12)	39(3)	19(3)	28(3)	5(2)	9(2)	4(2)
C(13)	28(3)	27(3)	27(3)	6(2)	4(2)	1(2)
C(14)	30(3)	21(3)	22(2)	3(2)	9(2)	0(2)
C(15)	36(3)	19(3)	32(3)	-2(2)	11(2)	0(2)
C(16)	38(3)	23(3)	35(3)	-2(2)	5(2)	-5(2)
C(17)	29(3)	34(3)	28(3)	0(2)	14(2)	-2(2)
C(18)	30(3)	41(3)	32(3)	7(2)	10(2)	8(2)
C(19)	34(3)	43(3)	31(3)	5(2)	3(2)	6(2)
C(20)	44(3)	36(3)	41(3)	12(3)	14(3)	11(2)
Cl(1)	29(1)	51(1)	50(1)	7(1)	15(1)	3(1)
Cl(2)	34(1)	35(1)	52(1)	-10(1)	17(1)	-7(1)
Cl(3)	39(1)	32(1)	56(1)	-2(1)	12(1)	4(1)
Fe(1)	26(1)	29(1)	30(1)	0(1)	10(1)	1(1)
N(1)	27(2)	24(2)	22(2)	3(2)	8(2)	5(2)
N(2)	30(2)	37(2)	29(2)	-5(2)	6(2)	1(2)
O(2)	22(2)	28(2)	36(2)	-3(2)	7(1)	1(1)
O(1)	32(2)	29(2)	36(2)	-4(2)	17(2)	-4(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^{-3}) for **11**·1/2 CH_2Cl_2.

	x	y	z	U(eq)
H(3)	9677	279	7667	37
H(5)	12548	1383	6682	33
H(7A)	9473	2110	4937	36
H(7B)	11381	2192	5704	36
H(8A)	12091	2761	7398	30
H(8B)	10830	2418	7858	30
H(11)	9547	4531	9296	36
H(13)	12880	3598	8430	34
H(15A)	6512	3629	8986	44
H(15B)	5960	3868	7726	44
H(15C)	6642	4279	8781	44
H(16A)	13346	4442	10326	50
H(16B)	12328	4918	9493	50
H(16C)	13650	4547	9147	50
H(17A)	10954	3133	5332	35
H(17B)	9912	3505	5928	35
H(18A)	8438	3495	3982	41
H(18B)	8491	2831	3955	41
H(19A)	4383	2960	3768	57
H(19B)	5688	2564	3461	57
H(19C)	5437	3189	3010	57
H(20A)	7285	3835	5653	60
H(20B)	5342	3678	5181	60
H(20C)	6224	3950	4356	60

Supplemental Figure 1. ORTEP plot of the single crystal X-ray crystallographic structure of **8**. Ellipsoids are drawn at 50% and hydrogens are omitted for clarity.

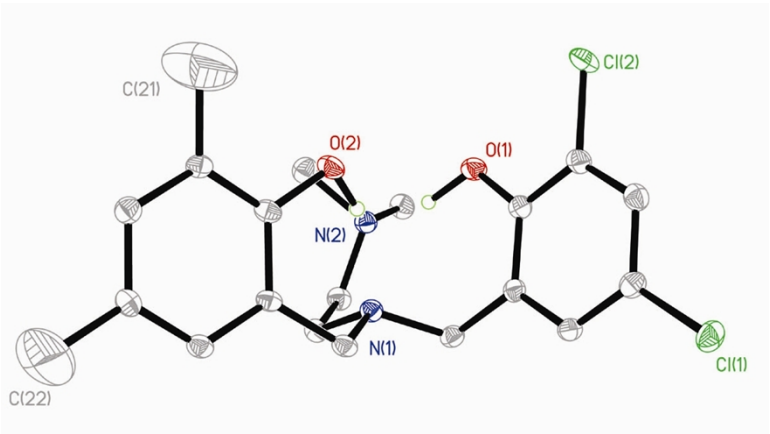


Table 1. Crystal data and structure refinement for **8**.

Identification code	MVAI093	
Empirical formula	C20 H26 Cl2 N2 O2	
Formula weight	397.33	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2(1)	
Unit cell dimensions	a = 8.3776(17) Å	α= 90°.
	b = 13.897(3) Å	β= 90°.
	c = 16.716(3) Å	γ = 90°.
Volume	1946.2(7) $Å^3$	
Z	4	
Density (calculated)	1.356 Mg/m^3	
Absorption coefficient	0.351 mm^{-1}	
F(000)	840	
Crystal size	0.50 x 0.30 x 0.30 mm^3	
Theta range for data collection	1.91 to 28.28°.	
Index ranges	-10<=h<=10, -18<=k<=18, -21<=l<=22	
Reflections collected	16659	
Independent reflections	4662 [R(int) = 0.0757]	
Completeness to theta = 28.28°	98.5 %	
Absorption correction	None	
Max. and min. transmission	0.9021 and 0.8442	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4662 / 0 / 288	
Goodness-of-fit on F^2	0.974	
Final R indices [I>2sigma(I)]	R1 = 0.0461, wR2 = 0.0985	
R indices (all data)	R1 = 0.0563, wR2 = 0.1024	
Absolute structure parameter	0.16(8)	
Largest diff. peak and hole	0.268 and -0.223 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **8**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	4570(3)	8303(2)	4074(1)	22(1)
C(2)	5198(3)	7541(2)	4499(1)	24(1)
C(3)	4494(3)	6647(2)	4494(1)	25(1)
C(4)	3134(3)	6513(2)	4038(1)	25(1)
C(5)	2485(3)	7258(2)	3614(1)	24(1)
C(6)	3171(3)	8164(2)	3635(1)	22(1)
C(7)	5362(3)	9262(2)	4075(1)	23(1)
C(8)	7704(3)	8956(2)	2191(1)	21(1)
C(9)	6476(3)	8747(2)	1650(1)	23(1)
C(10)	6820(3)	8622(2)	849(1)	21(1)
C(11)	8363(3)	8728(2)	580(1)	25(1)
C(12)	9578(3)	8952(2)	1108(1)	26(1)
C(14)	7361(2)	8977(2)	3070(1)	23(1)
C(15)	6171(3)	10547(2)	3166(1)	26(1)
C(16)	4713(3)	11144(2)	3351(1)	26(1)
C(17)	1852(3)	11177(2)	3405(1)	30(1)
C(18)	3165(3)	10850(2)	2167(1)	32(1)
N(1)	5907(2)	9518(1)	3268(1)	22(1)
N(2)	3245(2)	10725(1)	3031(1)	24(1)
O(1)	2445(2)	8866(1)	3216(1)	27(1)
O(2)	4948(2)	8633(1)	1895(1)	26(1)
C(13)	9237(3)	9058(2)	1909(1)	23(1)
Cl(1)	5260(8)	5739(3)	5025(3)	28(1)
Cl(2)	734(9)	7101(6)	3065(5)	31(1)
C(22)	11278(19)	9035(13)	838(11)	31(4)
C(21)	5528(17)	8376(11)	263(9)	18(3)
C(19)	1029(18)	7115(14)	3139(10)	24(3)
C(20)	5210(20)	5823(9)	4993(8)	48(5)
Cl(3)	5289(8)	8246(5)	215(4)	21(1)
Cl(4)	11473(9)	9056(5)	739(5)	31(1)

Table 3. Bond lengths [Å] and angles [°] for **8**.

C(1)-C(2)	1.379(3)
C(1)-C(6)	1.396(3)
C(1)-C(7)	1.489(3)
C(2)-C(3)	1.376(3)
C(3)-C(4)	1.383(3)
C(3)-C(20)	1.537(12)
C(3)-Cl(1)	1.671(5)
C(4)-C(5)	1.367(3)
C(5)-C(6)	1.385(3)
C(5)-C(19)	1.470(14)
C(5)-Cl(2)	1.744(8)
C(6)-O(1)	1.346(3)
C(7)-N(1)	1.467(3)
C(8)-C(13)	1.376(3)
C(8)-C(9)	1.400(3)
C(8)-C(14)	1.497(3)
C(9)-O(2)	1.353(3)
C(9)-C(10)	1.382(3)
C(10)-C(11)	1.376(3)
C(10)-C(21)	1.499(13)
C(10)-Cl(3)	1.743(7)
C(11)-C(12)	1.382(3)
C(12)-C(13)	1.377(3)
C(12)-C(22)	1.499(16)
C(12)-Cl(4)	1.709(8)
C(14)-N(1)	1.469(3)
C(15)-N(1)	1.458(3)
C(15)-C(16)	1.508(3)
C(16)-N(2)	1.462(3)
C(17)-N(2)	1.465(3)
C(18)-N(2)	1.457(3)
C(2)-C(1)-C(6)	119.0(2)
C(2)-C(1)-C(7)	121.1(2)

C(6)-C(1)-C(7)	119.87(19)
C(3)-C(2)-C(1)	121.8(2)
C(2)-C(3)-C(4)	118.6(2)
C(2)-C(3)-C(20)	120.2(7)
C(4)-C(3)-C(20)	121.2(7)
C(2)-C(3)-Cl(1)	120.9(3)
C(4)-C(3)-Cl(1)	120.5(3)
C(20)-C(3)-Cl(1)	0.9(8)
C(5)-C(4)-C(3)	120.7(2)
C(4)-C(5)-C(6)	120.7(2)
C(4)-C(5)-C(19)	120.5(8)
C(6)-C(5)-C(19)	118.7(8)
C(4)-C(5)-Cl(2)	120.8(3)
C(6)-C(5)-Cl(2)	118.4(3)
C(19)-C(5)-Cl(2)	1.2(10)
O(1)-C(6)-C(5)	117.3(2)
O(1)-C(6)-C(1)	123.6(2)
C(5)-C(6)-C(1)	119.1(2)
N(1)-C(7)-C(1)	110.77(17)
C(13)-C(8)-C(9)	119.1(2)
C(13)-C(8)-C(14)	120.92(18)
C(9)-C(8)-C(14)	119.77(19)
O(2)-C(9)-C(10)	118.41(19)
O(2)-C(9)-C(8)	121.65(19)
C(10)-C(9)-C(8)	119.9(2)
C(11)-C(10)-C(9)	120.0(2)
C(11)-C(10)-C(21)	119.3(6)
C(9)-C(10)-C(21)	120.7(6)
C(11)-C(10)-Cl(3)	121.7(3)
C(9)-C(10)-Cl(3)	118.2(3)
C(21)-C(10)-Cl(3)	4.9(8)
C(10)-C(11)-C(12)	120.5(2)
C(13)-C(12)-C(11)	119.5(2)
C(13)-C(12)-C(22)	118.8(8)
C(11)-C(12)-C(22)	121.7(7)
C(13)-C(12)-Cl(4)	122.3(3)

C(11)-C(12)-Cl(4)	118.2(3)
C(22)-C(12)-Cl(4)	3.7(10)
N(1)-C(14)-C(8)	113.01(17)
N(1)-C(15)-C(16)	113.10(18)
N(2)-C(16)-C(15)	112.82(17)
C(15)-N(1)-C(7)	113.12(17)
C(15)-N(1)-C(14)	110.44(17)
C(7)-N(1)-C(14)	109.97(17)
C(18)-N(2)-C(16)	110.74(18)
C(18)-N(2)-C(17)	109.56(18)
C(16)-N(2)-C(17)	110.11(17)
C(8)-C(13)-C(12)	121.1(2)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **8**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	20(1)	26(1)	19(1)	1(1)	2(1)	0(1)
C(2)	22(1)	29(1)	21(1)	-1(1)	-2(1)	-1(1)
C(3)	27(1)	26(1)	22(1)	-1(1)	1(1)	2(1)
C(4)	25(1)	24(1)	27(1)	-2(1)	5(1)	-3(1)
C(5)	17(1)	31(1)	25(1)	-2(1)	3(1)	-1(1)
C(6)	18(1)	26(1)	21(1)	-1(1)	2(1)	2(1)
C(7)	22(1)	28(1)	20(1)	-1(1)	-1(1)	1(1)
C(8)	17(1)	20(1)	26(1)	-1(1)	-4(1)	1(1)
C(9)	17(1)	23(1)	29(1)	-3(1)	-2(1)	2(1)
C(10)	17(1)	21(1)	25(1)	1(1)	-5(1)	4(1)
C(11)	26(1)	27(1)	23(1)	-1(1)	0(1)	1(1)
C(12)	20(1)	28(1)	29(1)	0(1)	2(1)	0(1)
C(14)	16(1)	24(1)	28(1)	1(1)	-3(1)	2(1)
C(15)	24(1)	27(1)	28(1)	1(1)	0(1)	-3(1)
C(16)	28(1)	22(1)	29(1)	-1(1)	-2(1)	0(1)
C(17)	25(1)	34(1)	33(1)	0(1)	3(1)	5(1)
C(18)	33(1)	32(1)	30(1)	2(1)	0(1)	2(1)
N(1)	19(1)	24(1)	22(1)	0(1)	0(1)	1(1)
N(2)	21(1)	27(1)	22(1)	-3(1)	1(1)	0(1)
O(1)	22(1)	28(1)	30(1)	3(1)	-5(1)	1(1)
O(2)	17(1)	33(1)	30(1)	-4(1)	0(1)	-1(1)
C(13)	18(1)	25(1)	26(1)	1(1)	-3(1)	1(1)
Cl(1)	35(2)	20(1)	29(2)	10(1)	-3(1)	6(1)
Cl(2)	16(2)	33(1)	45(2)	-1(1)	-12(1)	-8(1)
C(22)	12(5)	64(6)	16(4)	-7(3)	12(3)	-6(3)
C(21)	14(5)	19(4)	23(4)	-10(2)	-2(3)	-2(3)
C(19)	13(6)	22(3)	37(4)	-2(3)	-15(3)	-8(3)
C(20)	28(7)	53(8)	64(8)	-10(5)	-5(5)	-2(6)
Cl(3)	18(2)	23(2)	23(1)	-4(1)	-8(1)	-5(1)
Cl(4)	14(2)	50(2)	29(2)	-8(1)	8(2)	-7(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^{-3}) for **8**.

	x	y	z	U(eq)
H(2)	6142	7637	4804	29
H(4)	2644	5896	4020	30
H(7A)	4603	9755	4269	28
H(7B)	6286	9249	4444	28
H(11)	8595	8645	28	30
H(14A)	7247	8308	3266	27
H(14B)	8279	9272	3351	27
H(15A)	6504	10672	2608	31
H(15B)	7054	10751	3522	31
H(16A)	4609	11211	3938	31
H(16B)	4856	11796	3123	31
H(17A)	1832	11863	3270	46
H(17B)	1915	11101	3987	46
H(17C)	876	10869	3207	46
H(18A)	2165	10575	1966	48
H(18B)	4070	10520	1916	48
H(18C)	3205	11537	2037	48
H(13)	10075	9203	2272	28
H(22A)	11991	8864	1280	46
H(22B)	11493	9698	670	46
H(22C)	11462	8598	387	46
H(21A)	4604	8795	354	28
H(21B)	5210	7703	336	28
H(21C)	5924	8469	-283	28
H(19A)	1198	7360	2596	36
H(19B)	141	7462	3389	36
H(19C)	776	6427	3115	36
H(20A)	6099	5532	4702	72
H(20B)	4384	5334	5090	72
H(20C)	5587	6076	5505	72

H(2O)	4860(40)	8930(20)	2420(17)	58(9)
H(1O)	3020(40)	9490(30)	3200(20)	90(12)