6 Crystallographic Data

6.1 CRYSTALLOGRAPHIC DATA OF RADICAL 1

Single crystals were grown by slow evaporation of a methylenchloride/hexane (1:1) mixture and used for its X-ray crystal determination.[6] An ORTEP view of **1** is shown in Figure 56. The structure reveals almost eclipsed cyclopentadienyl rings and a *trans* configuration for the CH=CH unit. Despite the available resonance pathway between the radical and ferrocene units and the *trans* configuration of the CH=CH spacer, there is a lack of planarity between the C_6Cl_4 ring and the cyclopentadiene unit. This lack of planarity is caused by packing effects for the $FeCp_2$-C(11) group in the crystal lattice. The structure has two disordered $FeCp_2$-C(11) parts (see Figure 57) and the twist angle is 44º for the major and 27º for the minor unit. Normally disordering leads to a lost of information, but in this case we can interpret the different twist angles of approximately 44 and 27 degrees in direction of a packing effect in the solid state. This means, that the distortion of the planarity depends beside the repulsion of Cl--H atoms also from the "free" space of the "attached" unit in the crystal lattice.

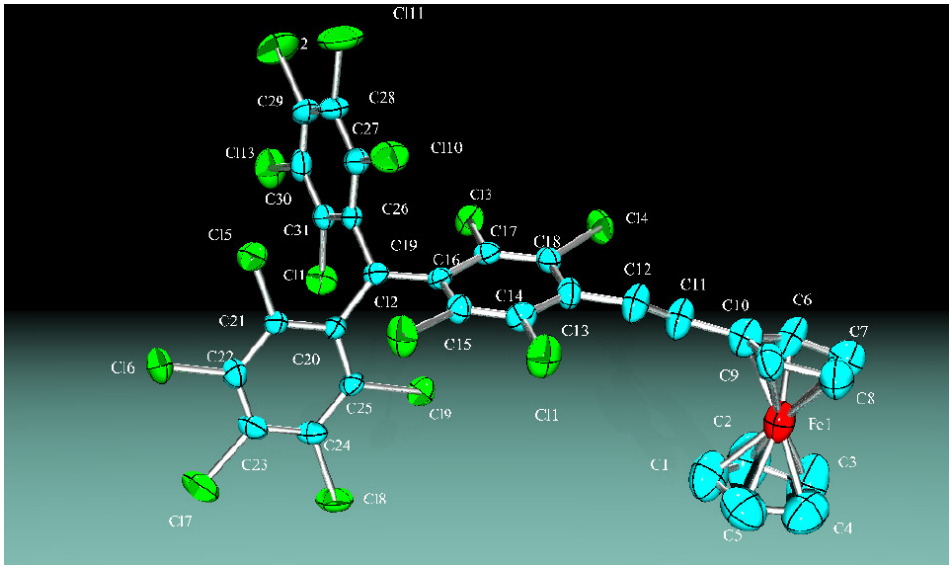

Figure 56. Molecular structure of radical **1**

Repulsion distances: Cl(1)---H(11a): 2.36 Å and Cl(4)---H(11): 2.64 Å

Dieder angles (for twisting):
From the major part
1. C(13)---C(18) : C(13)-C(12)-C(11) 44.4(7) °
2. C(6)---C(10) : C(10)-C(11)-C(12) 20(1) °
and from the minor part
3. C(13)---C(18) : C(13a)-C(12a)-C(11a) 26.9(9) °
4. C(6a)---C(10a) : C(10a)-C(11a)-C(12a) 11(2) °

The C_6Cl_4 ring is twisted by an angle of 12º with respect to the cyclopentadiene and 45 º with respect to the spacer. It is likely that this disorder and distortion of coplanarity is caused by an effect of packing.

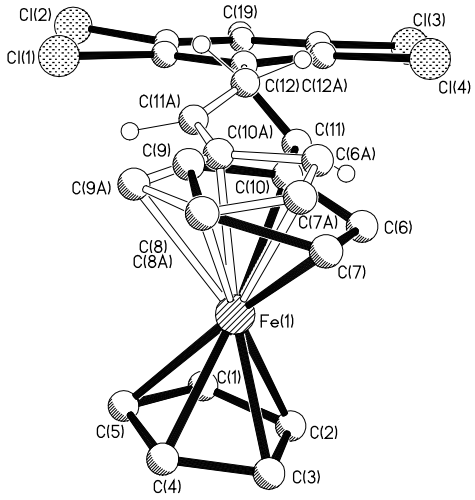

Figure 57. Disorder of C(6)-C(11):C(6a)-C(11a) with C(12)=C(12a) (for hydrogen calculation) and C(8)=C(8a) (overlying position); no separation of the Cp-ring C(1)---C(5) (nearly overlying positions, therefore great anisotropic parameters on these positions).

Solvent molecules and hydrogen atoms have been omitted for more clarity. Just the major part of the molecule have been draw, also for more clarity. Crystallographic data for this structure have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-191556.

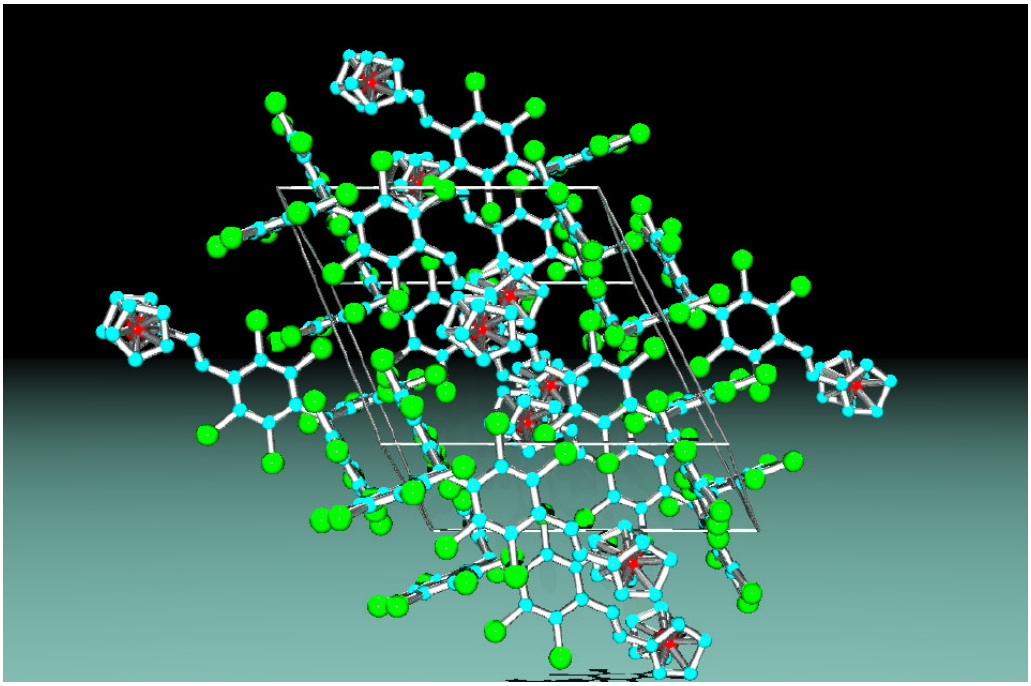

Figure 58. Crystal packing of **1**

6.1.1 Crystallographic data

Molecular formula	$C_{31}H_{11}Cl_{14}Fe$ x 0.5 CH_2Cl_2
Formula weight	978.01
Crystal system	Triclinic
Space group	*P*-1
a, pm	887.71(4)
b, pm	1441.98(8)
c, pm	1555.46(7)
α,deg	109.200(2)
β,deg	97.008(3)
γ,deg	102.408(2)
V, nm^3	1795.89(15)
Z	2
h (min-max)	0-9
k (min-max)	-15-14
l (min-max)	(-16)-(15)
cryst color,habit	Dark platelet
D(calc), Mg /m^3	1.809
F(000)	964
temp, K	223(2)
Scan range	ϕ and ω scans
Reflections collected	4408
Independent reflections	7774 (R_{int}=0.0286)
diffractometer	Kappa CCD
Radiation	MoKα (λ=0.71073 Å)

Refinement method	Full-matrix least-squares on F^2
R(F), %[a]	0.0601
R(wF^2), %[a]	0.01137

[a] Quantity minimized = $R(wF^2) = \sum\left[w\left(F_0^{\,2} - F_c^{\,2}\right)^2\right] / \sum\left[w\left(F_0^{\,2}\right)^2\right]^{1/2}$;
$R = \sum\Delta / \sum\left(F_0\right), \Delta = \left|\left(F_0 - F_c\right)\right|$

Refinement details: 3:2 disorder of the ethenyl ferrocene unit. Disorder was only solved for atoms C6-C12 : C6A-C12A. C8=C8A and C12=C12A with equal coordinates and displacement parameters were refined separately for calculation of their hydrogens. The solvent CH_2Cl_2 lies nearby an inversion centre with occupancy of 0.5.

6.1.2 Atomic coordinates (Å) and equivalent isotropic displacement parameters (pm^2)

Num	Atom	X/Å	Y/Å	Z/Å	U eqq
1	Fe1	1.28819(10)	0.43671(7)	0.32466(6)	0.0673(3)
2	Cl1	0.71347(17)	0.48048(10)	0.12938(10)	0.0647(4)
3	Cl2	0.58155(16)	0.63144(10)	0.06818(8)	0.0567(4)
4	Cl3	0.87679(14)	0.93082(10)	0.40140(7)	0.0482(3)
5	Cl4	0.98001(16)	0.77548(11)	0.46855(8)	0.0573(4)
6	Cl5	0.39117(13)	0.84448(11)	0.07462(8)	0.0542(4)
7	Cl6	0.43495(16)	0.87339(12)	-0.10842(8)	0.0624(4)
8	Cl7	0.76139(18)	0.89207(12)	-0.16392(9)	0.0677(4)
9	Cl8	1.04022(15)	0.86569(11)	-0.03964(9)	0.0596(4)
10	Cl9	1.00894(13)	0.85571(11)	0.15266(9)	0.0512(4)
11	Cl10	0.40586(15)	0.77399(11)	0.27729(10)	0.0586(4)
12	Cl11	0.27213(17)	0.93313(14)	0.40529(11)	0.0827(5)
13	Cl12	0.4099(2)	1.16356(15)	0.44548(12)	0.0961(6)
14	Cl13	0.69877(19)	1.23490(11)	0.36380(10)	0.0690(4)
15	Cl14	0.84727(14)	1.07697(10)	0.24478(8)	0.0496(3)
16	Cl15	0.9949(12)	0.6009(7)	0.0050(8)	0.190(4)
17	Cl16	0.9193(8)	0.4010(6)	-0.0691(4)	0.130(2)
18	C1	1.3471(10)	0.5125(6)	0.2385(6)	0.096(2)
19	C2	1.4651(10)	0.5481(6)	0.3179(7)	0.115(3)
20	C3	1.5223(9)	0.4643(7)	0.3225(7)	0.121(3)
21	C4	1.4399(10)	0.3805(6)	0.2474(7)	0.103(2)
22	C5	1.3336(10)	0.4088(7)	0.1941(6)	0.102(2)
23	C13	0.8542(5)	0.6337(4)	0.2939(3)	0.0433(12)
24	C14	0.7611(5)	0.6068(4)	0.2052(3)	0.0407(12)
25	C15	0.7045(5)	0.6755(4)	0.1768(3)	0.0366(11)
26	C16	0.7370(5)	0.7791(3)	0.2350(3)	0.0302(11)
27	C17	0.8227(5)	0.8052(3)	0.3258(3)	0.0330(11)
28	C18	0.8784(5)	0.7352(4)	0.3547(3)	0.0359(11)
29	C19	0.6852(4)	0.8533(3)	0.2025(3)	0.0314(11)
30	C20	0.7023(5)	0.8558(3)	0.1102(3)	0.0308(10)
31	C21	0.5768(5)	0.8602(4)	0.0497(3)	0.0354(11)
32	C22	0.5947(5)	0.8687(4)	-0.0359(3)	0.0417(12)
33	C23	0.7378(6)	0.8734(4)	-0.0613(3)	0.0419(12)
34	C24	0.8625(5)	0.8647(4)	-0.0048(3)	0.0402(12)
35	C25	0.8454(5)	0.8567(3)	0.0801(3)	0.0346(11)
36	C26	0.6196(5)	0.9304(3)	0.2629(3)	0.0308(10)

37	C27	0.4919(5)	0.9006(4)	0.3021(3)	0.0403(12)
38	C28	0.4283(5)	0.9726(4)	0.3588(3)	0.0480(14)
39	C29	0.4907(6)	1.0752(4)	0.3765(3)	0.0512(14)
40	C30	0.6173(6)	1.1067(4)	0.3392(3)	0.0453(13)
41	C31	0.6799(5)	1.0341(4)	0.2830(3)	0.0346(11)
42	C32	1.066(2)	0.498(3)	0.0358(17)	0.133(7)
43	C6	1.2513(14)	0.5037(11)	0.4486(12)	0.071(5)
44	C7	1.2801(17)	0.4110(17)	0.4420(16)	0.066(5)
45	C8	1.1618(9)	0.3238(6)	0.3587(7)	0.083(2)
46	C9	1.0640(19)	0.3782(17)	0.3261(13)	0.060(5)
47	C10	1.1241(16)	0.4888(12)	0.3838(14)	0.060(4)
48	C11	1.0574(11)	0.5697(7)	0.3679(7)	0.065(3)
49	C12	0.9192(7)	0.5606(5)	0.3232(4)	0.0573(15)
50	C6A	1.137(5)	0.466(3)	0.428(2)	0.115(14)
51	C7A	1.198(4)	0.387(3)	0.434(2)	0.087(9)
52	C8A	1.1618(9)	0.3238(6)	0.3587(7)	0.083(2)
53	C9A	1.060(4)	0.335(3)	0.283(2)	0.107(14)
54	C10A	1.052(3)	0.437(3)	0.3337(19)	0.062(6)
55	C11A	0.9562(19)	0.4879(14)	0.2870(10)	0.072(5)
56	C12A	0.9192(7)	0.5606(5)	0.3232(4)	0.0573(15)

6.1.3 Bond distances (Angstroms)

Atom A	Atom B	Distance	Atom A	Atom B	Distance
Fe1	C6	1.955(16)	C13	C14	1.404(6)
Fe1	C10	1.968(14)	C13	C18	1.410(7)
Fe1	C7	1.98(2)	C13	C12	1.470(7)
Fe1	C9	1.994(15)	C14	C15	1.370(7)
Fe1	C8	2.029(7)	C15	C16	1.412(6)
Fe1	C2	2.031(8)	C16	C17	1.406(6)
Fe1	C4	2.034(8)	C16	C19	1.457(6)
Fe1	C3	2.037(9)	C17	C18	1.387(6)
Fe1	C1	2.038(8)	C19	C20	1.473(6)
Fe1	C5	2.044(8)	C19	C26	1.478(6)
Fe1	C9A	2.11(4)	C20	C21	1.395(6)
Cl1	C14	1.738(5)	C20	C25	1.405(6)
Cl2	C15	1.730(4)	C21	C22	1.401(6)
Cl3	C17	1.727(4)	C22	C23	1.371(6)
Cl4	C18	1.731(4)	C23	C24	1.381(7)
Cl5	C21	1.723(4)	C24	C25	1.387(6)
Cl6	C22	1.729(5)	C26	C31	1.388(6)
Cl7	C23	1.733(5)	C26	C27	1.404(6)
Cl8	C24	1.728(4)	C27	C28	1.395(6)
Cl9	C25	1.732(4)	C28	C29	1.383(7)
Cl10	C27	1.712(5)	C29	C30	1.385(7)
Cl11	C28	1.716(5)	C30	C31	1.392(6)
Cl12	C29	1.724(5)	C32	Cl15	1.31(3)
Cl13	C30	1.726(5)	C32	Cl16	1.35(3)
Cl14	C31	1.734(4)	C32	C32	1.54(4)
Cl15	Cl16	1.190(10)	C6	C10	1.35(3)
Cl15	C32	1.31(3)	C6	C7	1.39(3)
Cl15	C32	1.91(3)	C7	C8	1.55(2)
Cl16	Cl15	1.190(10)	C8	C9	1.44(2)

Cl16	C32	1.35(3)	C9	C10	1.49(2)
Cl16	C32	1.87(3)	C10	C11	1.49(2)
C1	C2	1.387(11)	C11	C12	1.291(11)
C1	C5	1.397(11)	C6A	C7A	1.38(5)
C2	C3	1.425(11)	C6A	C10A	1.45(4)
C3	C4	1.357(11)	C9A	C10A	1.45(4)
C4	C5	1.392(11)	C10A	C11A	1.50(3)

6.1.4 Bond angles (degrees)

Atom A	Atom B	Atom C	Angle	Atom A	Atom B	Atom C	Angle
C6	Fe1	C10	40.1(8)	C15	C16	C19	121.9(4)
C6	Fe1	C7	41.2(8)	C18	C17	C16	122.2(4)
C10	Fe1	C7	69.1(8)	C18	C17	Cl3	118.0(3)
C6	Fe1	C9	71.6(7)	C16	C17	Cl3	119.5(4)
C10	Fe1	C9	44.1(7)	C17	C18	C13	121.7(4)
C7	Fe1	C9	71.2(6)	C17	C18	Cl4	118.2(3)
C6	Fe1	C8	74.1(5)	C13	C18	Cl4	120.1(4)
C10	Fe1	C8	73.2(5)	C16	C19	C20	121.0(3)
C7	Fe1	C8	45.3(6)	C16	C19	C26	120.6(3)
C9	Fe1	C8	42.0(6)	C20	C19	C26	118.4(4)
C6	Fe1	C2	100.7(5)	C21	C20	C25	117.1(4)
C10	Fe1	C2	113.4(5)	C21	C20	C19	120.9(3)
C7	Fe1	C2	121.0(6)	C25	C20	C19	122.0(4)
C9	Fe1	C2	152.2(7)	C20	C21	C22	121.3(4)
C8	Fe1	C2	163.3(4)	C20	C21	Cl5	120.2(3)
C6	Fe1	C4	147.3(5)	C22	C21	Cl5	118.3(3)
C10	Fe1	C4	172.5(7)	C23	C22	C21	120.0(4)
C7	Fe1	C4	117.5(7)	C23	C22	Cl6	120.6(3)
C9	Fe1	C4	132.7(7)	C21	C22	Cl6	119.3(3)
C8	Fe1	C4	108.4(3)	C22	C23	C24	120.1(4)
C2	Fe1	C4	67.2(3)	C22	C23	Cl7	119.4(4)
C6	Fe1	C3	112.1(5)	C24	C23	Cl7	120.5(3)
C10	Fe1	C3	145.9(5)	C23	C24	C25	120.0(4)
C7	Fe1	C3	102.8(5)	C23	C24	Cl8	119.8(3)
C9	Fe1	C3	166.7(7)	C25	C24	Cl8	120.3(4)
C8	Fe1	C3	125.5(3)	C24	C25	C20	121.5(4)
C2	Fe1	C3	41.0(3)	C24	C25	Cl9	118.4(3)
C4	Fe1	C3	38.9(3)	C20	C25	Cl9	120.0(3)
C6	Fe1	C1	123.1(5)	C31	C26	C27	117.4(4)
C10	Fe1	C1	107.6(6)	C31	C26	C19	122.0(4)
C7	Fe1	C1	159.1(6)	C27	C26	C19	120.6(4)
C9	Fe1	C1	121.7(6)	C28	C27	C26	121.1(5)
C8	Fe1	C1	155.2(4)	C28	C27	Cl10	118.5(4)
C2	Fe1	C1	39.9(3)	C26	C27	Cl10	120.4(3)
C4	Fe1	C1	67.7(3)	C29	C28	C27	119.8(4)
C3	Fe1	C1	67.9(3)	C29	C28	Cl11	120.5(4)
C6	Fe1	C5	163.1(5)	C27	C28	Cl11	119.7(4)
C10	Fe1	C5	132.8(7)	C28	C29	C30	120.3(4)
C7	Fe1	C5	154.8(7)	C28	C29	Cl12	119.5(4)
C9	Fe1	C5	114.1(6)	C30	C29	Cl12	120.2(4)
C8	Fe1	C5	121.2(4)	C29	C30	C31	119.3(5)
C2	Fe1	C5	66.6(4)	C29	C30	Cl13	120.4(4)

C4	Fe1	C5	39.9(3)	C31	C30	Cl13	120.3(4)
C3	Fe1	C5	66.4(4)	C26	C31	C30	122.1(4)
C1	Fe1	C5	40.0(3)	C26	C31	Cl14	119.8(3)
C6	Fe1	C9A	91.4(7)	C30	C31	Cl14	117.9(4)
C10	Fe1	C9A	63.8(8)	Cl15	C32	Cl16	161.7(17)
C7	Fe1	C9A	82.2(10)	Cl15	C32	C32	84(2)
C9	Fe1	C9A	20.6(6)	Cl16	C32	C32	80(2)
C8	Fe1	C9A	41.5(10)	Cl15	C32	Cl16	39.1(9)
C2	Fe1	C9A	155.1(11)	Cl16	C32	Cl16	125.7(14)
C4	Fe1	C9A	112.3(7)	C32	C32	Cl16	45.3(17)
C3	Fe1	C9A	149.9(8)	Cl15	C32	Cl15	126.5(15)
C1	Fe1	C9A	115.6(11)	Cl16	C32	Cl15	38.3(8)
C5	Fe1	C9A	96.5(8)	C32	C32	Cl15	43.0(16)
Cl16	Cl15	C32	97.1(13)	Cl16	C32	Cl15	87.9(9)
Cl16	Cl15	C32	44.6(8)	C10	C6	C7	110.2(13)
C32	Cl15	C32	53.5(15)	C10	C6	Fe1	70.5(10)
Cl15	Cl16	C32	97.1(10)	C7	C6	Fe1	70.5(11)
Cl15	Cl16	C32	43.8(8)	C6	C7	C8	109.9(15)
C32	Cl16	C32	54.3(14)	C6	C7	Fe1	68.2(12)
C2	C1	C5	106.9(7)	C8	C7	Fe1	68.9(9)
C2	C1	Fe1	69.8(5)	C9	C8	C7	101.6(13)
C5	C1	Fe1	70.2(4)	C9	C8	Fe1	67.7(6)
C1	C2	C3	108.0(7)	C7	C8	Fe1	65.8(9)
C1	C2	Fe1	70.4(4)	C8	C9	C10	109.2(12)
C3	C2	Fe1	69.7(5)	C8	C9	Fe1	70.3(7)
C4	C3	C2	107.7(8)	C10	C9	Fe1	67.1(8)
C4	C3	Fe1	70.4(5)	C6	C10	C9	109.1(14)
C2	C3	Fe1	69.2(5)	C6	C10	C11	126.2(13)
C3	C4	C5	108.6(8)	C9	C10	C11	124.5(16)
C3	C4	Fe1	70.7(5)	C6	C10	Fe1	69.4(8)
C5	C4	Fe1	70.4(4)	C9	C10	Fe1	68.9(8)
C4	C5	C1	108.7(8)	C11	C10	Fe1	125.5(12)
C4	C5	Fe1	69.6(5)	C12	C11	C10	128.8(10)
C1	C5	Fe1	69.7(5)	C11	C12	C13	131.3(7)
C14	C13	C18	115.6(4)	C7A	C6A	C10A	109(3)
C14	C13	C12	122.6(5)	C7A	C6A	Fe1	72.4(18)
C18	C13	C12	121.8(5)	C10A	C6A	Fe1	67.2(13)
C15	C14	C13	122.7(4)	C6A	C7A	Fe1	71.4(17)
C15	C14	Cl1	119.0(3)	C10A	C9A	Fe1	70.5(19)
C13	C14	Cl1	118.3(4)	C9A	C10A	C6A	107(2)
C14	C15	C16	122.1(4)	C9A	C10A	C11A	120(3)
C14	C15	Cl2	118.3(3)	C6A	C10A	C11A	133(3)
C16	C15	Cl2	119.6(3)	C9A	C10A	Fe1	69.5(19)
C17	C16	C15	115.5(4)	C6A	C10A	Fe1	73.9(17)
C17	C16	C19	122.7(4)	C11A	C10A	Fe1	124.7(15)

6.2 CRYSTALLOGRAPHIC DATA OF RADICAL 2

Single crystals of **2** were grown by slow difussion of hexane over a methylenchloride solution of **2** and used for its X-Ray crystal determination. An ORTEP view of **2** is shown in Figure 59. The structure reveals totally alternated cyclopentadienyl rings, due to the high steric hindrance of their methyl groups. A trans configuration of the CH=CH unit is also observed. Due to the available resonance pathway and the trans configuration, the (H-C_5H_4)

and the ($C_6H_2Cl_4$) rings are almost in a coplanar disposition twisted by a low angle of ~11 º. It is likely that the presence of electronically attractive interactions between the CH_3 and chlorines of the benzene unit causes this coplanarity (Cl-----H = 3.167Å). The solid-state packing of **2** is best described as centrosymmetrically related pairs of molecules. Stacking of these related pairs occurs with continuous staggering along the c axis and with a head-to-tail pairing along the b axis. Under these circumstances, the largest driving forces for such molecular packing are Cl---H interaction, dipole-dipole and van der Waals interactions, which lead to an efficient filling of space.

Crystallographic data for this structure have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-191558.

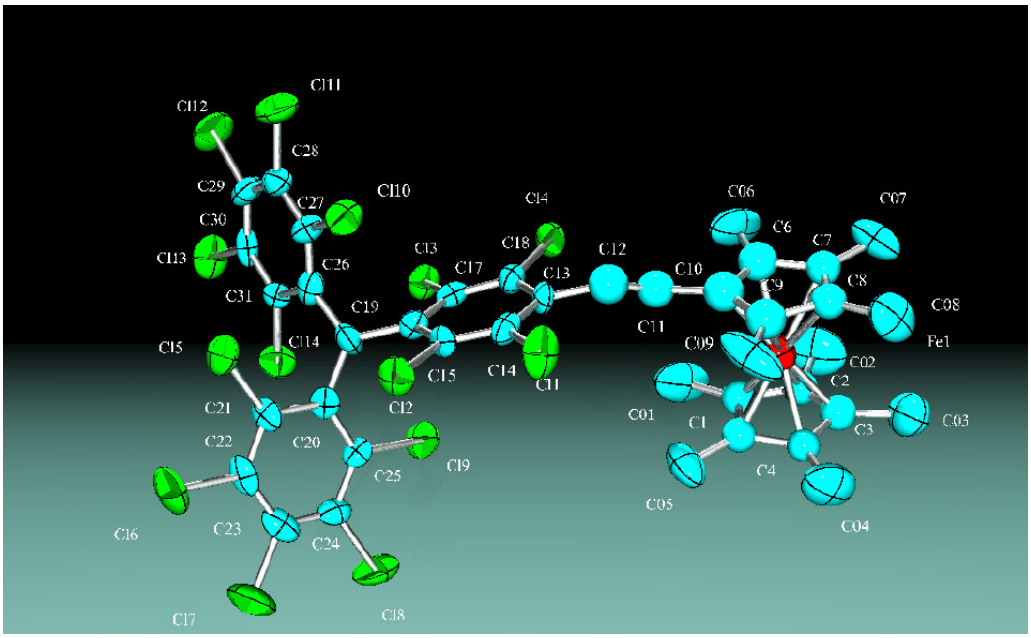

Figure 59. Molecular structure of radical **2**

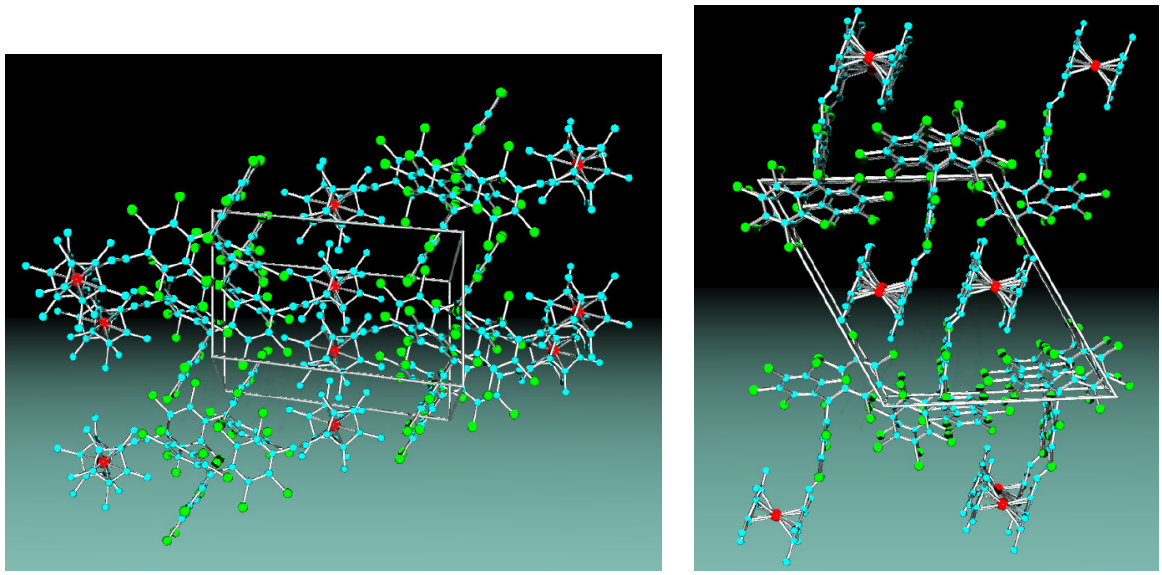

Figure 60. Crystal packing of radical **2**

6.2.1 Crystallographic data

Molecular formula	$C_{40}H_{29}Cl_{14}Fe$
Formula weight	1061.869
Crystal system	triclinic
Space group	*P-1*
a, pm	9.1198(8)
b, pm	15.805(2)
c, pm	17.310(2)
α,deg	116.150(5)
β,deg	95.495(7)
γ,deg	99.661(7)
V, nm^3	2166.9(4)
Z	2
h (min-max)	0-8
k (min-max)	-14-14
l (min-max)	-16-15
cryst color,habit	Brown plate
Size	0.25x0.06x0.03
F(000)	1066
temp, K	233(2)
Scan range	ϕ and ω scans
Reflections collected	6943
Independent reflections	3738 (R_{int}=0.0766)
diffractometer	Kappa CCD
Radiation	MoKα (λ=0.71073 Å)
Refinement method	Full-matrix least-squares on F^2
R(F), %[a]	0.1330
R(wF^2), %[a]	0.1749

[a] Quantity minimized = $R(wF^2) = \sum\left[w\left(F_0^{\,2} - F_c^{\,2}\right)^2\right] / \sum\left[w\left(F_0^{\,2}\right)^2\right]^{1/2}$;

$$R = \sum \Delta / \sum (F_0), \Delta = \left|(F_0 - F_c)\right|$$

Refinement details: small crystal and bad data quality, because of splitting reflections from grown-ups; 30 worse reflections were omitted. C1-C12 were refined isotropic. The bond C11=C12 is disordered; 1:1 C11:C11A, for hydrogen calculation: C12=C12A

6.2.2 Atomic coordinates (Å) and equivalent isotropic displacement parameters (pm^2)

Num	Atom	X/Å	Y/Å	Z/Å	Ueqq
1	Fe1	0.2020(2)	0.24949(16)	0.49968(14)	0.0527(7)
2	C1	0.1603(17)	0.2327(12)	0.6069(11)	0.064(5)
3	C2	0.0473(18)	0.1667(12)	0.5325(11)	0.064(5)
4	C3	0.1028(18)	0.1081(12)	0.4685(11)	0.068(5)
5	C4	0.2646(16)	0.1282(11)	0.4957(10)	0.055(4)
6	C5	0.3039(17)	0.2084(11)	0.5829(10)	0.060(5)
7	C6	0.1428(19)	0.3709(13)	0.5046(12)	0.078(5)
8	C7	0.0979(17)	0.2921(11)	0.4160(10)	0.059(4)
9	C8	0.2276(17)	0.2656(11)	0.3895(11)	0.061(5)
10	C9	0.357(2)	0.3235(13)	0.4602(12)	0.079(5)

11	C10	0.299(2)	0.3889(13)	0.5298(12)	0.080(5)
12	C13	0.5277(17)	0.5895(10)	0.7548(10)	0.045(4)
13	C14	0.6882(18)	0.6074(10)	0.7771(10)	0.048(4)
14	C15	0.7651(14)	0.6654(10)	0.8623(11)	0.045(4)
15	C16	0.6897(15)	0.7099(10)	0.9328(10)	0.038(4)
16	C17	0.5327(16)	0.6912(10)	0.9081(10)	0.041(4)
17	C18	0.4580(16)	0.6384(11)	0.8219(11)	0.048(4)
18	C19	0.7741(16)	0.7696(11)	1.0224(10)	0.050(4)
19	C20	0.8773(14)	0.7324(11)	1.0638(9)	0.040(4)
20	C21	1.0218(16)	0.7910(10)	1.1115(10)	0.046(4)
21	C22	1.1212(14)	0.7553(13)	1.1486(9)	0.052(4)
22	C23	1.0761(19)	0.6601(15)	1.1388(10)	0.055(4)
23	C24	0.937(2)	0.6061(10)	1.0946(10)	0.046(4)
24	C25	0.8379(14)	0.6399(10)	1.0567(9)	0.038(4)
25	C26	0.7379(14)	0.8668(11)	1.0717(11)	0.048(4)
26	C27	0.7396(15)	0.9320(12)	1.0368(10)	0.047(4)
27	C28	0.6948(16)	1.0166(12)	1.0795(13)	0.056(4)
28	C29	0.6521(16)	1.0429(11)	1.1597(13)	0.053(4)
29	C30	0.6567(14)	0.9826(12)	1.1969(9)	0.045(4)
30	C31	0.7008(13)	0.8962(10)	1.1555(10)	0.032(3)
31	C01	0.156(3)	0.3091(17)	0.6958(12)	0.147(11)
32	C02	-0.1214(19)	0.1724(18)	0.5355(17)	0.140(10)
33	C03	0.016(2)	0.0238(13)	0.3798(13)	0.103(7)
34	C04	0.372(2)	0.0766(15)	0.4458(13)	0.112(7)
35	C05	0.4592(18)	0.2484(13)	0.6382(11)	0.090(6)
36	C06	0.029(3)	0.4214(15)	0.5556(13)	0.140(10)
37	C07	-0.0577(18)	0.2521(15)	0.3598(12)	0.095(7)
38	C08	0.236(2)	0.1885(14)	0.2990(11)	0.098(7)
39	C09	0.5189(17)	0.3169(17)	0.4544(14)	0.118(9)
40	Cl1	0.7922(5)	0.5561(3)	0.6963(3)	0.0795(15)
41	Cl2	0.9618(4)	0.6908(3)	0.8843(3)	0.0592(12)
42	Cl3	0.4273(4)	0.7306(3)	0.9911(3)	0.0545(11)
43	Cl4	0.2623(4)	0.6263(3)	0.8003(3)	0.0602(12)
44	Cl5	1.0821(4)	0.9043(3)	1.1221(3)	0.0682(13)
45	Cl6	1.2982(4)	0.8273(4)	1.2067(3)	0.0872(16)
46	Cl7	1.2018(6)	0.6178(4)	1.1851(3)	0.1021(18)
47	Cl8	0.8812(6)	0.4910(3)	1.0845(3)	0.0862(15)
48	Cl9	0.6569(4)	0.5696(3)	1.0069(3)	0.0691(13)
49	Cl10	0.8017(5)	0.9048(3)	0.9399(3)	0.0627(12)
50	Cl11	0.6915(5)	1.0922(3)	1.0315(3)	0.0789(14)
51	Cl12	0.5899(5)	1.1472(3)	1.2083(3)	0.0856(15)
52	Cl13	0.6034(5)	1.0155(3)	1.2982(3)	0.0747(14)
53	Cl14	0.6950(4)	0.8221(3)	1.2043(3)	0.0621(12)
54	C11	0.339(4)	0.465(3)	0.626(2)	0.070(10)
55	C12	0.448(2)	0.5298(14)	0.6655(13)	0.078(5)
56	C11A	0.442(4)	0.451(2)	0.608(2)	0.063(9)
57	C12A	0.448(2)	0.5298(14)	0.6655(13)	0.078(5)

6.2.3 Bond distances (Angstroms)

Atom A	Atom B	Distance	Atom A	Atom B	Distance
Fe1	C5	2.033(15)	C14	C15	1.379(19)
Fe1	C10	2.037(18)	C14	Cl1	1.739(15)
Fe1	C9	2.040(18)	C15	C16	1.423(19)
Fe1	C6	2.048(17)	C15	Cl2	1.737(13)

Fe1	C1	2.049(16)	C16	C17	1.396(18)
Fe1	C2	2.053(16)	C16	C19	1.458(18)
Fe1	C8	2.062(16)	C17	C18	1.380(19)
Fe1	C4	2.065(15)	C17	Cl3	1.747(14)
Fe1	C7	2.065(15)	C18	Cl4	1.748(14)
Fe1	C3	2.067(17)	C19	C20	1.474(18)
C1	C2	1.42(2)	C19	C26	1.51(2)
C1	C5	1.47(2)	C20	C25	1.391(18)
C1	C01	1.49(2)	C20	C21	1.408(18)
C2	C3	1.30(2)	C21	C22	1.389(19)
C2	C02	1.56(2)	C21	Cl5	1.705(14)
C3	C4	1.442(19)	C22	C23	1.42(2)
C3	C03	1.54(2)	C22	Cl6	1.725(14)
C4	C5	1.44(2)	C23	C24	1.34(2)
C4	C04	1.48(2)	C23	Cl7	1.720(15)
C5	C05	1.492(19)	C24	C25	1.377(18)
C6	C10	1.39(2)	C24	Cl8	1.731(15)
C6	C7	1.45(2)	C25	Cl9	1.724(13)
C6	C06	1.52(2)	C26	C27	1.403(19)
C7	C8	1.374(19)	C26	C31	1.410(19)
C7	C07	1.50(2)	C27	C28	1.37(2)
C8	C9	1.45(2)	C27	Cl10	1.717(15)
C8	C08	1.52(2)	C28	C29	1.38(2)
C9	C10	1.42(2)	C28	Cl11	1.732(16)
C9	C09	1.51(2)	C29	C30	1.37(2)
C10	C11	1.52(4)	C29	Cl12	1.711(15)
C10	C11A	1.60(4)	C30	C31	1.386(19)
C13	C18	1.366(19)	C30	Cl13	1.739(14)
C13	C14	1.424(19)	C31	Cl14	1.718(13)
C13	C12	1.44(2)	C11	C12	1.19(3)

6.2.4 Bond angles (degrees)

Atom A	Atom B	Atom C	Angle	Atom A	Atom B	Atom C	Angle
C5	Fe1	C10	111.6(7)	C8	C7	Fe1	70.4(9)
C5	Fe1	C9	111.7(7)	C6	C7	Fe1	68.7(9)
C10	Fe1	C9	40.7(6)	C07	C7	Fe1	130.0(11)
C5	Fe1	C6	138.3(7)	C7	C8	C9	109.8(15)
C10	Fe1	C6	39.8(6)	C7	C8	C08	125.4(15)
C9	Fe1	C6	68.0(7)	C9	C8	C08	124.8(15)
C5	Fe1	C1	42.3(6)	C7	C8	Fe1	70.7(10)
C10	Fe1	C1	113.3(7)	C9	C8	Fe1	68.5(10)
C9	Fe1	C1	142.0(7)	C08	C8	Fe1	128.2(12)
C6	Fe1	C1	111.1(7)	C10	C9	C8	105.8(15)
C5	Fe1	C2	68.3(6)	C10	C9	C09	129.2(18)
C10	Fe1	C2	143.1(7)	C8	C9	C09	124.9(17)
C9	Fe1	C2	176.2(7)	C10	C9	Fe1	69.6(10)
C6	Fe1	C2	114.7(7)	C8	C9	Fe1	70.1(9)
C1	Fe1	C2	40.4(6)	C09	C9	Fe1	128.1(12)
C5	Fe1	C8	141.5(6)	C6	C10	C9	109.2(17)
C10	Fe1	C8	67.7(7)	C6	C10	C11	106(2)
C9	Fe1	C8	41.3(6)	C9	C10	C11	144(2)
C6	Fe1	C8	67.0(7)	C6	C10	C11A	145(2)
C1	Fe1	C8	175.9(6)	C9	C10	C11A	105.2(18)
C2	Fe1	C8	136.5(6)	C11	C10	C11A	39.0(15)

C5	Fe1	C4	41.1(6)	C6	C10	Fe1	70.5(10)
C10	Fe1	C4	139.5(6)	C9	C10	Fe1	69.8(10)
C9	Fe1	C4	111.5(6)	C11	C10	Fe1	118.7(17)
C6	Fe1	C4	179.2(7)	C11A	C10	Fe1	120.5(15)
C1	Fe1	C4	68.9(6)	C18	C13	C14	115.5(13)
C2	Fe1	C4	65.8(6)	C18	C13	C12	123.1(15)
C8	Fe1	C4	113.1(6)	C14	C13	C12	121.2(16)
C5	Fe1	C7	179.5(7)	C15	C14	C13	121.5(13)
C10	Fe1	C7	68.2(7)	C15	C14	Cl1	118.3(13)
C9	Fe1	C7	68.5(6)	C13	C14	Cl1	120.2(13)
C6	Fe1	C7	41.2(6)	C14	C15	C16	122.3(12)
C1	Fe1	C7	137.3(6)	C14	C15	Cl2	119.3(13)
C2	Fe1	C7	111.6(6)	C16	C15	Cl2	118.3(11)
C8	Fe1	C7	38.9(5)	C17	C16	C15	114.4(13)
C4	Fe1	C7	139.3(6)	C17	C16	C19	124.6(14)
C5	Fe1	C3	68.8(6)	C15	C16	C19	121.0(12)
C10	Fe1	C3	179.6(7)	C18	C17	C16	122.8(13)
C9	Fe1	C3	139.3(7)	C18	C17	Cl3	119.2(12)
C6	Fe1	C3	139.9(7)	C16	C17	Cl3	117.9(12)
C1	Fe1	C3	66.9(7)	C13	C18	C17	123.0(13)
C2	Fe1	C3	36.9(6)	C13	C18	Cl4	119.2(12)
C8	Fe1	C3	112.0(7)	C17	C18	Cl4	117.6(13)
C4	Fe1	C3	40.8(5)	C16	C19	C20	121.4(13)
C7	Fe1	C3	111.4(6)	C16	C19	C26	115.7(12)
C2	C1	C5	105.0(14)	C20	C19	C26	122.6(13)
C2	C1	C01	133.8(17)	C25	C20	C21	118.1(13)
C5	C1	C01	121.2(16)	C25	C20	C19	122.2(12)
C2	C1	Fe1	69.9(9)	C21	C20	C19	119.6(14)
C5	C1	Fe1	68.3(9)	C22	C21	C20	119.9(13)
C01	C1	Fe1	128.0(13)	C22	C21	Cl5	118.3(12)
C3	C2	C1	112.9(15)	C20	C21	Cl5	121.7(12)
C3	C2	C02	128.5(17)	C21	C22	C23	119.8(12)
C1	C2	C02	118.6(16)	C21	C22	Cl6	119.8(14)
C3	C2	Fe1	72.1(11)	C23	C22	Cl6	120.5(14)
C1	C2	Fe1	69.6(9)	C24	C23	C22	119.4(13)
C02	C2	Fe1	125.6(12)	C24	C23	Cl7	121.8(15)
C2	C3	C4	109.1(15)	C22	C23	Cl7	118.7(14)
C2	C3	C03	128.0(16)	C23	C24	C25	121.6(14)
C4	C3	C03	122.8(15)	C23	C24	Cl8	119.4(14)
C2	C3	Fe1	71.0(10)	C25	C24	Cl8	119.0(13)
C4	C3	Fe1	69.5(9)	C24	C25	C20	121.1(12)
C03	C3	Fe1	129.4(12)	C24	C25	Cl9	119.1(12)
C5	C4	C3	107.0(14)	C20	C25	Cl9	119.6(11)
C5	C4	C04	125.5(15)	C27	C26	C31	117.1(13)
C3	C4	C04	127.5(16)	C27	C26	C19	121.9(15)
C5	C4	Fe1	68.3(8)	C31	C26	C19	120.9(15)
C3	C4	Fe1	69.6(9)	C28	C27	C26	121.0(14)
C04	C4	Fe1	128.0(12)	C28	C27	Cl10	120.1(14)
C4	C5	C1	106.1(13)	C26	C27	Cl10	118.9(12)
C4	C5	C05	124.7(15)	C27	C28	C29	121.5(14)
C1	C5	C05	128.9(15)	C27	C28	Cl11	119.2(15)
C4	C5	Fe1	70.6(9)	C29	C28	Cl11	119.3(14)
C1	C5	Fe1	69.4(9)	C30	C29	C28	118.5(14)
C05	C5	Fe1	129.5(11)	C30	C29	Cl12	121.9(16)
C10	C6	C7	108.3(16)	C28	C29	Cl12	119.6(15)
C10	C6	C06	129.4(19)	C29	C30	C31	121.7(13)
C7	C6	C06	122.3(17)	C29	C30	Cl13	118.8(14)

C10	C6	Fe1	69.7(10)	C31	C30	Cl13	119.6(13)
C7	C6	Fe1	70.0(10)	C30	C31	C26	120.1(13)
C06	C6	Fe1	126.4(14)	C30	C31	Cl14	118.7(12)
C8	C7	C6	106.9(14)	C26	C31	Cl14	121.1(12)
C8	C7	C07	125.7(16)	C12	C11	C10	131(3)
C6	C7	C07	127.2(15)	C11	C12	C13	139(2)

6.3 CRYSTALLOGRAPHIC DATA OF RADICAL 3

Single crystals of **3** were grown by slow evaporation from a carbon tetrachloride/hexanes (1:1) mixture and used for its X-Ray crystal determination. An ORTEP view of **3** is shown in Figure 61. The structure reveals almost eclipsed cyclopentadienyl rings and a trans configuration of the CH=N unit. Despite the available resonance pathway and the trans configuration, the (H-C_5H_4) and the ($C_6H_2Cl_2$) rings are twisted by an angle of ~28 °. It is likely that the presence of steric interactions between the CH=N hydrogen and one of the ortho hydrogen atoms of the benzene ring causes this lack of planarity. The solid-state packing of **3** is best described as centrosymmetrically related pairs of molecules. Stacking of these related pairs occurs with continuous staggering along the c axis and with a head-to-tail pairing along the b axis. The relative arrangement and the considerably large distances between neighboring molecules excludes the presence of hydrogen bonds among neighboring CH=N units. Under these circumstances, the largest driving forces for such molecular packing are dipole-dipole and van der Waals interactions, which lead to an efficient filling of space.

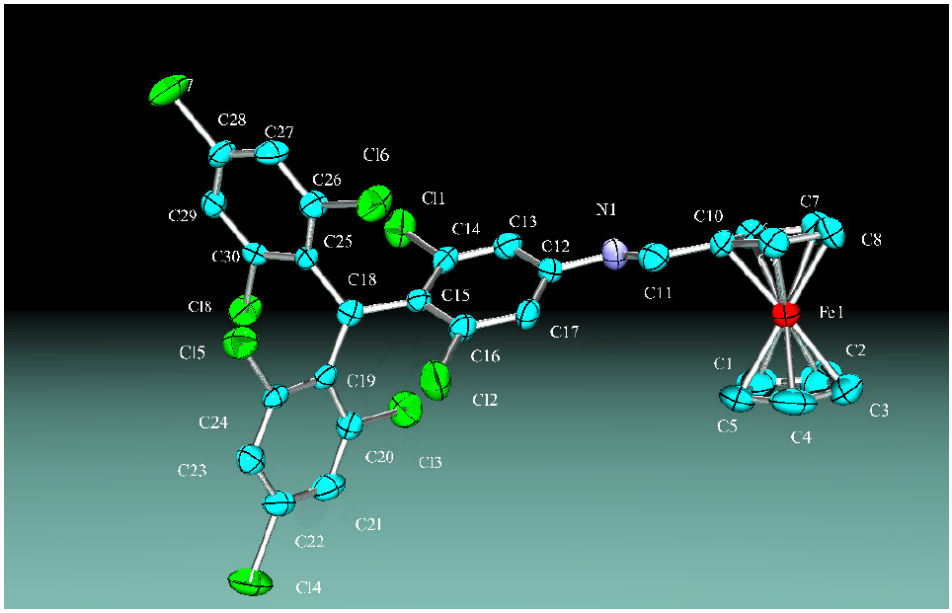

Figure 61. Molecular structure of radical **3**

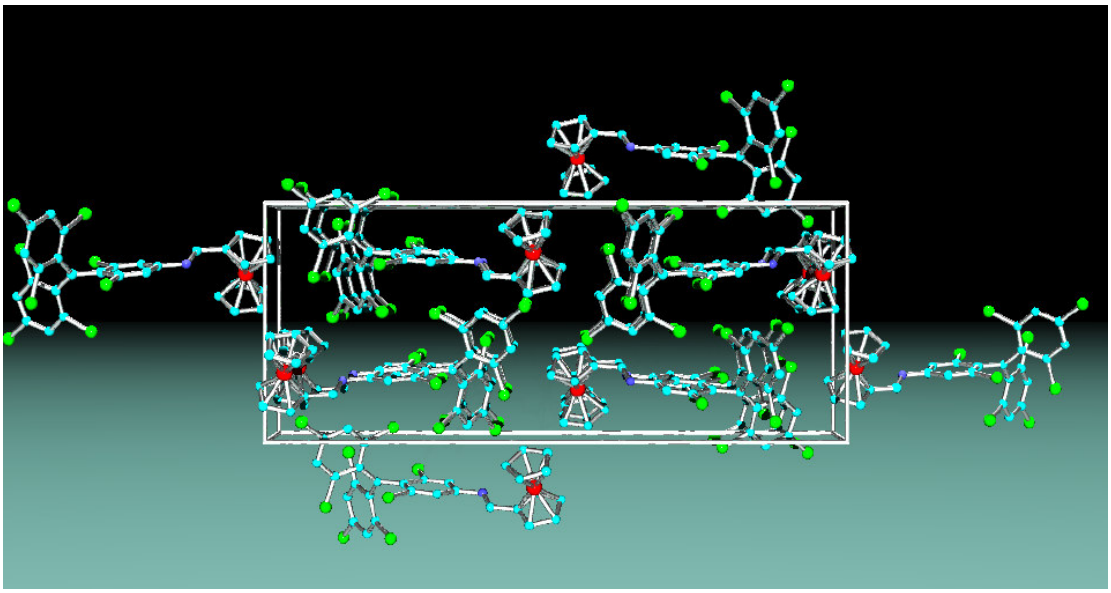

Figure 62. Crystal packing of radical **3**

Crystallographic data for this structure have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-147245.

6.3.1 Crystallographic data

Molecular formula	$C_{30}H_{16}Cl_8NFe$
Formula weight	729.89
Crystal system	monoclinic
Space group	$P2_1/n$
a, pm	819.00(4)
b, pm	2953.9(2)
c, pm	1210.65(4)
β,deg	91.021(3)
V, nm^3	2.9284(3)
Z	4
h (min-max)	0-8
k (min-max)	0-31
l (min-max)	(-12)-12
cryst color,habit	Brown platelet
D(calc), Mg /m^3	1.656
F(000)	1460
temp, K	218(2)
Scan type	*Frames in ϕ and ω*
Scan Speed	60 sec. per frame, 2 iterations
Scan range	1.4º per frame
Reflections collected	5879
Independent reflections	3038 (R_{int}=0.0456)
diffractometer	Kappa CCD
radiation	MoKα (λ=0.71073 Å)

Refinement method	Full-matrix least-squares on F^2
R(F), %[a]	3.96
R(wF^2), %[a]	7.03
$\Delta\rho_{max}$, **e.nm^{-3}**	283
$\Delta\rho_{min}$, **e.nm^{-3}**	-240

[a] Quantity minimized $= R(wF^2) = \sum\left[w\left(F_0^2 - F_c^2\right)^2\right] / \sum\left[w\left(F_0^2\right)^2\right]^{1/2}$;

$$R = \sum\Delta / \sum\left(F_0\right), \Delta = \left|\left(F_0 - F_c\right)\right|$$

6.3.2 Atomic coordinates (Å) and equivalent isotropic displacement parameters (pm^2)

Num	Atom	X/Å	Y/Å	Z/Å	Ueqq
1	C1	0.1772(9)	0.0569(4)	0.4543(6)	0.079(2)
2	C2	0.2430(12)	0.0146(3)	0.4435(6)	0.083(2)
3	C3	0.3738(10)	0.0169(3)	0.3708(6)	0.079(2)
4	C4	0.3865(9)	0.0625(3)	0.3381(5)	0.076(2)
5	C5	0.2650(10)	0.0873(2)	0.3908(7)	0.075(2)
6	C6	-0.0764(7)	0.0328(2)	0.2564(4)	0.048(2)
7	C7	0.0013(7)	-0.0085(2)	0.2321(5)	0.054(2)
8	C8	0.1201(7)	0.0004(2)	0.1538(5)	0.052(2)
9	C9	0.1195(7)	0.0466(2)	0.1291(4)	0.047(2)
10	C10	-0.0038(7)	0.0672(2)	0.1921(4)	0.0385(14)
11	C11	-0.0400(7)	0.1160(2)	0.1988(4)	0.0436(15)
12	C12	-0.1816(7)	0.1780(2)	0.2623(4)	0.0376(14)
13	C13	-0.3357(7)	0.1918(2)	0.2885(4)	0.0404(14)
14	C14	-0.3721(6)	0.2369(2)	0.3010(4)	0.0367(14)
15	C15	-0.2541(6)	0.2715(2)	0.2883(4)	0.0306(13)
16	C16	-0.0971(6)	0.2560(2)	0.2662(4)	0.0370(14)
17	C17	-0.0583(6)	0.2106(2)	0.2539(4)	0.0394(14)
18	C18	-0.2972(6)	0.3195(2)	0.2959(4)	0.0320(13)
19	C19	-0.1993(6)	0.3503(2)	0.3635(4)	0.0337(14)
20	C20	-0.1446(6)	0.3399(2)	0.4720(4)	0.0389(14)
21	C21	-0.0480(7)	0.3682(2)	0.5347(4)	0.049(2)
22	C22	-0.0041(6)	0.4096(2)	0.4922(5)	0.044(2)
23	C23	-0.0544(6)	0.4225(2)	0.3889(4)	0.0398(14)
24	C24	-0.1487(6)	0.3930(2)	0.3261(4)	0.0311(13)
25	C25	-0.4360(6)	0.3362(2)	0.2294(4)	0.0276(12)
26	C26	-0.4557(6)	0.3261(2)	0.1162(4)	0.0363(14)
27	C27	-0.5838(7)	0.3425(2)	0.0518(4)	0.0413(15)
28	C28	-0.6977(6)	0.3698(2)	0.0995(4)	0.0384(14)
29	C29	-0.6885(6)	0.3803(2)	0.2105(4)	0.0368(14)
30	C30	-0.5598(6)	0.3637(2)	0.2731(4)	0.0310(13)
31	N1	-0.1544(6)	0.1306(2)	0.2538(4)	0.0443(12)
32	Cl1	-0.5710(2)	0.25001(5)	0.33733(13)	0.0574(5)
33	Cl2	0.0586(2)	0.29456(5)	0.24532(13)	0.0614(5)
34	Cl3	-0.2063(2)	0.28990(5)	0.53545(11)	0.0660(5)
35	Cl4	0.1181(2)	0.44599(6)	0.57036(12)	0.0689(5)
36	Cl5	-0.1901(2)	0.41005(5)	0.19158(10)	0.0495(4)
37	Cl6	-0.3075(2)	0.29606(5)	0.04678(10)	0.0583(5)

38	Cl7	-0.8559(2)	0.39102(6)	0.01926(12)	0.0703(5)
39	Cl8	-0.5609(2)	0.37571(5)	0.41349(10)	0.0512(4)
40	Fe1	0.16386(10)	0.03779(3)	0.29395(6)	0.0461(3)

6.3.3 Bond distances (Angstroms)

Atom A	Atom B	Distance (Å)	Atom A	Atom B	Distance (Å)
Fe1	C10	2.025(5)	C7	C8	1.395(7)
Fe1	C4	2.027(6)	C8	C9	1.397(7)
Fe1	C2	2.031(6)	C9	C10	1.414(7)
Fe1	C3	2.036(7)	C10	C11	1.474(7)
Fe1	C9	2.039(5)	C12	C13	1.369(7)
Fe1	C7	2.041(6)	C12	C17	1.400(7)
Fe1	C5	2.041(6)	C13	C14	1.376(7)
Fe1	C8	2.050(5)	C14	C15	1.417(7)
Cl1	C14	1.738(5)	C15	C16	1.395(7)
Cl2	C16	1.731(5)	C15	C18	1.465(7)
Cl3	C20	1.744(5)	C16	C17	1.388(7)
Cl4	C22	1.737(5)	C18	C19	1.454(7)
Cl5	C24	1.733(5)	C18	C25	1.467(6)
Cl6	C26	1.733(5)	C19	C24	1.404(7)
Cl7	C28	1.724(5)	C19	C20	1.415(6)
Cl8	C30	1.737(5)	C20	C21	1.371(7)
N1	C11	1.238(6)	C21	C22	1.376(7)
N1	C12	1.421(6)	C22	C23	1.364(7)
C1	C2	1.366(9)	C23	C24	1.383(7)
C1	C5	1.391(10)	C25	C30	1.409(6)
C2	C3	1.400(10)	C25	C26	1.409(6)
C3	C4	1.408(9)	C26	C27	1.383(7)
C4	C5	1.400(9)	C27	C28	1.368(7)
C6	C7	1.408(7)	C28	C29	1.379(6)
C6	C10	1.418(7)	C29	C30	1.377(7)

6.3.4 Bond angles (degrees)

Atom A	Atom B	Atom C	Angle	Atom A	Atom B	Atom C	Angle
C6	Fe1	C1	105.8(3)	C10	C6	Fe1	69.8(3)
C6	Fe1	C10	41.1(2)	C8	C7	C6	107.6(5)
C1	Fe1	C10	119.3(3)	C8	C7	Fe1	70.4(3)
C6	Fe1	C4	162.8(3)	C6	C7	Fe1	68.8(3)
C1	Fe1	C4	67.3(3)	C7	C8	C9	109.2(5)
C10	Fe1	C4	127.1(3)	C7	C8	Fe1	69.7(3)
C6	Fe1	C2	118.1(3)	C9	C8	Fe1	69.6(3)
C1	Fe1	C2	39.4(3)	C8	C9	C10	107.7(5)
C10	Fe1	C2	152.8(3)	C8	C9	Fe1	70.4(3)
C4	Fe1	C2	67.4(3)	C10	C9	Fe1	69.1(3)
C6	Fe1	C3	153.3(3)	C9	C10	C6	107.3(5)
C1	Fe1	C3	67.4(3)	C9	C10	C11	126.6(5)
C10	Fe1	C3	165.1(3)	C6	C10	C11	125.8(5)
C4	Fe1	C3	40.6(3)	C9	C10	Fe1	70.2(3)
C2	Fe1	C3	40.3(3)	C6	C10	Fe1	69.2(3)
C6	Fe1	C9	68.4(2)	C11	C10	Fe1	121.4(4)

C1	Fe1	C9	155.4(3)	N1	C11	C10	121.7(5)
C10	Fe1	C9	40.7(2)	C13	C12	C17	118.8(5)
C4	Fe1	C9	110.8(3)	C13	C12	N1	117.1(5)
C2	Fe1	C9	164.6(3)	C17	C12	N1	123.9(5)
C3	Fe1	C9	128.4(3)	C12	C13	C14	121.1(5)
C6	Fe1	C7	40.6(2)	C13	C14	C15	122.5(5)
C1	Fe1	C7	124.2(3)	C13	C14	Cl1	116.7(4)
C10	Fe1	C7	68.5(2)	C15	C14	Cl1	120.8(4)
C4	Fe1	C7	156.3(3)	C16	C15	C14	114.7(5)
C2	Fe1	C7	107.3(3)	C16	C15	C18	123.6(5)
C3	Fe1	C7	120.3(3)	C14	C15	C18	121.7(5)
C9	Fe1	C7	67.8(2)	C17	C16	C15	123.5(5)
C6	Fe1	C5	124.5(3)	C17	C16	Cl2	116.7(4)
C1	Fe1	C5	40.0(3)	C15	C16	Cl2	119.7(4)
C10	Fe1	C5	107.8(3)	C16	C17	C12	119.4(5)
C4	Fe1	C5	40.2(3)	C19	C18	C15	120.6(4)
C2	Fe1	C5	67.0(3)	C19	C18	C25	120.9(5)
C3	Fe1	C5	67.9(3)	C15	C18	C25	118.5(4)
C9	Fe1	C5	122.3(3)	C24	C19	C20	113.8(5)
C7	Fe1	C5	161.1(3)	C24	C19	C18	122.9(5)
C6	Fe1	C8	67.6(2)	C20	C19	C18	123.3(5)
C1	Fe1	C8	161.8(4)	C21	C20	C19	123.5(5)
C10	Fe1	C8	67.7(2)	C21	C20	Cl3	116.2(4)
C4	Fe1	C8	123.7(3)	C19	C20	Cl3	120.2(4)
C2	Fe1	C8	127.2(3)	C20	C21	C22	119.1(5)
C3	Fe1	C8	110.4(3)	C23	C22	C21	120.9(5)
C9	Fe1	C8	39.9(2)	C23	C22	Cl4	119.3(5)
C7	Fe1	C8	39.9(2)	C21	C22	Cl4	119.8(5)
C5	Fe1	C8	157.6(3)	C22	C23	C24	119.1(5)
C11	N1	C12	120.2(5)	C23	C24	C19	123.6(5)
C2	C1	C5	109.2(7)	C23	C24	Cl5	115.6(4)
C2	C1	Fe1	70.6(4)	C19	C24	Cl5	120.7(4)
C5	C1	Fe1	70.7(4)	C30	C25	C26	114.6(4)
C1	C2	C3	108.9(7)	C30	C25	C18	122.9(4)
C1	C2	Fe1	70.0(4)	C26	C25	C18	122.5(4)
C3	C2	Fe1	70.1(4)	C27	C26	C25	123.1(5)
C2	C3	C4	106.5(7)	C27	C26	Cl6	115.9(4)
C2	C3	Fe1	69.7(4)	C25	C26	Cl6	120.7(4)
C4	C3	Fe1	69.4(4)	C28	C27	C26	119.0(4)
C5	C4	C3	108.4(7)	C27	C28	C29	121.2(5)
C5	C4	Fe1	70.4(4)	C27	C28	Cl7	119.2(4)
C3	C4	Fe1	70.1(4)	C29	C28	Cl7	119.7(4)
C1	C5	C4	107.0(7)	C30	C29	C28	119.0(5)
C1	C5	Fe1	69.2(4)	C29	C30	C25	123.1(4)
C4	C5	Fe1	69.3(4)	C29	C30	Cl8	116.7(4)
C7	C6	C10	108.1(5)	C25	C30	Cl8	120.1(4)
C7	C6	Fe1	70.6(3)				

6.4 CRYSTALLOGRAPHIC DATA OF 18

Single crystals of **18** were grown by slow evaporation from a methylenchloride and used for its X-Ray crystal determination. An ORTEP view of **18** is shown in Figure 63. The structure reveals almost eclipsed cyclopentadienyl rings and a trans configuration of the CH=CH unit. Despite the available resonance pathway and the trans configuration, the (H-

C_5H_4) and the ($C_6H_2Cl_4$) rings are twisted by an angle of ~69 °. It is likely that the presence of steric interactions between the CH=CH hydrogen and one of the ortho hydrogen atoms of the benzene ring causes this lack of planarity. The solid-state packing of **18** is best described as ferrocenes related hexagonally. Stacking of these related molecules occurs with continuous staggering along the c axis and with a π–π aromatic interactions of the benzene rings. Solvent molecules have been omitted from the crystal packing for more clarity.

Dieder angles (for twisting):

From the major part

1. C(13)---C(18) : C(13)-C(12)-C(11) 57.0(7) °
2. C(6)---C(10) : C(10)-C(11)-C(12) 16.9(1) °

Crystallographic data for this structure have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-191557.

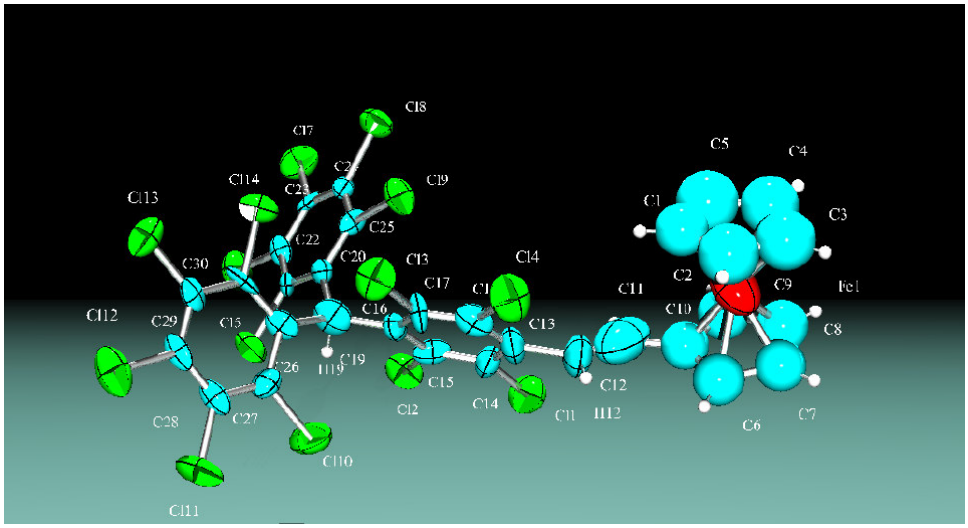

Figure 63. Molecular structure of **18**

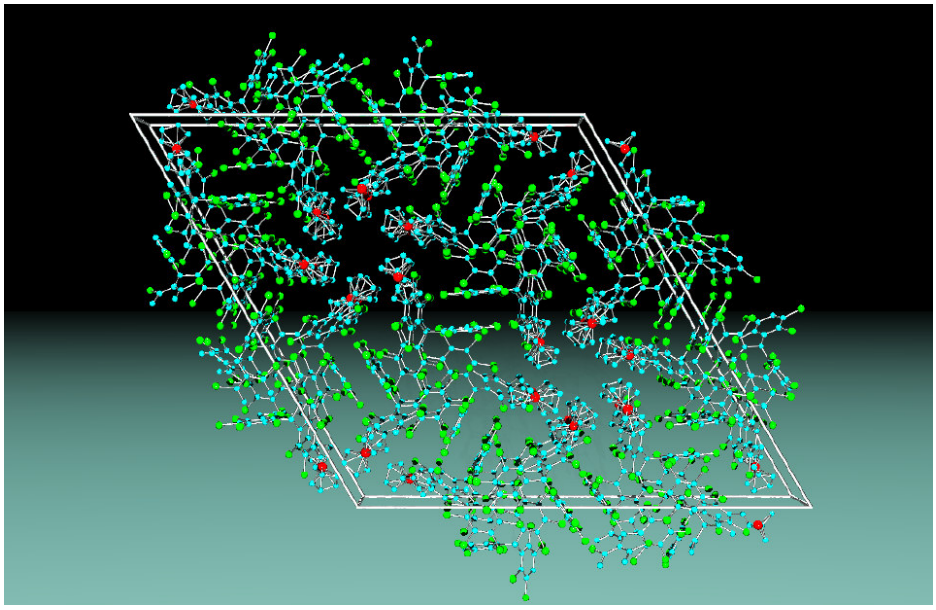

Figure 64. Crystal packing of **18**

6.4.1 Crystallographic data

Molecular formula	$C_{31}H_{12}Cl_{14}Fe$ x 1/6 CH_2Cl_2
Formula weight	950.71
Crystal system	Trigonal
Space group	*R-3*
a, pm	37.646(7)
b, pm	37.646(7)
c, pm	12.932(2)
α,deg	90.00
β,deg	90.00
γ,deg	120.00
V, nm^3	15872.1(48)
Z	18
h (min-max)	-35-19
k (min-max)	-20-34
l (min-max)	-12-11
cryst color,habit	Orange prism
D(calc), Mg /m^3	1.790
F(000)	8442
temp, K	223(2)
Scan range	ϕ and ω scans
Reflections collected	2355
Independent reflections	6093 (R_{int}=0.0766)
diffractometer	Kappa CCD
Radiation	MoKα (λ=0.71073 Å)
Refinement method	Full-matrix least-squares on F^2
R(F), %[a]	0.1181
R(wF^2), %[a]	0.2485

[a] Quantity minimized = $R(wF^2) = \sum\left[w\left(F_0^{\,2} - F_c^{\,2}\right)^2\right] / \sum\left[w\left(F_0^{\,2}\right)^2\right]^{1/2}$;

$$R = \sum \Delta / \sum (F_0), \Delta = \left|(F_0 - F_c)\right|$$

Refinement details: small crystal with weak diffraction, the ferrocenyl-unit is slightly disordered (disordering not solved), bond lengths of the cp-rings were refined with restraints (d = 1.4ang.) and these carbon atoms were refined isotropically. The bond lengths C12=C11 and C11-C10 must also be refined with restraints (d = 1.35 and 1.45 ang.). The Cl-atom of CH_2Cl_2 has an occupancy of 0.33333.

6.4.2 Atomic coordinates (Å) and equivalent isotropic displacement parameters (pm^2)

Num	Atom	X/Å	Y/Å	Z/Å	Ueqq
1	Fe1	-0.1982(2)	0.38531(15)	-0.4675(3)	0.140(2)
2	Cl1	-0.1717(2)	0.4202(2)	0.0187(4)	0.095(2)
3	Cl2	-0.1132(2)	0.4223(2)	0.1836(4)	0.075(2)
4	Cl3	-0.0898(2)	0.3142(2)	-0.0382(4)	0.077(2)
5	Cl4	-0.1455(2)	0.3155(2)	-0.2085(4)	0.093(2)
6	Cl5	-0.0487(2)	0.4033(2)	0.3693(3)	0.075(2)
7	Cl6	0.0272(2)	0.4877(2)	0.4041(3)	0.075(2)
8	Cl7	0.0792(2)	0.5389(2)	0.2169(4)	0.081(2)

9	Cl8	0.0557(2)	0.50383(15)	-0.0046(3)	0.066(2)
10	Cl9	-0.0205(2)	0.42153(15)	-0.0441(3)	0.064(2)
11	Cl10	-0.1476(2)	0.3113(2)	0.2642(4)	0.089(2)
12	Cl11	-0.1474(2)	0.2379(2)	0.3647(4)	0.094(2)
13	Cl12	-0.0733(2)	0.2251(2)	0.3388(4)	0.095(2)
14	Cl13	0.0014(2)	0.2855(2)	0.2060(4)	0.068(2)
15	Cl14	0.0028(2)	0.3597(2)	0.1054(3)	0.068(2)
16	C1	-0.1555(10)	0.3656(11)	-0.4802(23)	0.150(11)
17	C2	-0.1901(11)	0.3420(11)	-0.5409(28)	0.190(15)
18	C3	-0.1958(11)	0.3666(12)	-0.6128(27)	0.188(14)
19	C4	-0.1623(12)	0.4054(12)	-0.5950(27)	0.182(14)
20	C5	-0.1385(12)	0.4071(11)	-0.5085(30)	0.212(16)
21	C6	-0.2464(9)	0.3595(9)	-0.3606(22)	0.147(11)
22	C7	-0.2561(9)	0.3770(9)	-0.4486(21)	0.145(10)
23	C8	-0.2261(9)	0.4194(9)	-0.4560(22)	0.154(11)
24	C9	-0.1972(9)	0.4275(9)	-0.3757(22)	0.154(12)
25	C10	-0.2096(9)	0.3906(9)	-0.3188(21)	0.129(9)
26	C11	-0.1839(10)	0.3938(10)	-0.2282(23)	0.186(17)
27	C12	-0.1839(7)	0.3663(7)	-0.1749(19)	0.084(8)
28	C13	-0.1550(6)	0.3683(8)	-0.0884(18)	0.068(6)
29	C14	-0.1480(6)	0.3914(6)	-0.0007(21)	0.060(6)
30	C15	-0.1220(6)	0.3924(6)	0.0744(15)	0.054(5)
31	C16	-0.1032(5)	0.3687(7)	0.0668(15)	0.048(5)
32	C17	-0.1118(7)	0.3450(6)	-0.0187(18)	0.056(6)
33	C18	-0.1354(7)	0.3442(6)	-0.0930(17)	0.062(6)
34	C19	-0.0739(7)	0.3725(6)	0.1543(16)	0.079(7)
35	C20	-0.0329(5)	0.4133(6)	0.1658(17)	0.046(5)
36	C21	-0.0204(6)	0.4304(7)	0.2622(14)	0.040(5)
37	C22	0.0134(7)	0.4689(6)	0.2779(12)	0.041(5)
38	C23	0.0362(4)	0.4914(5)	0.1945(16)	0.033(4)
39	C24	0.0241(6)	0.4744(6)	0.0979(12)	0.040(5)
40	C25	-0.0102(6)	0.4379(6)	0.0832(13)	0.039(5)
41	C26	-0.0720(7)	0.3347(5)	0.1895(13)	0.050(5)
42	C27	-0.1057(6)	0.3050(7)	0.2487(14)	0.050(5)
43	C28	-0.1054(7)	0.2718(6)	0.2944(12)	0.051(5)
44	C29	-0.0722(8)	0.2660(5)	0.2804(13)	0.047(5)
45	C30	-0.0396(6)	0.2935(7)	0.2237(13)	0.045(5)
46	C31	-0.0387(6)	0.3277(6)	0.1802(11)	0.045(5)
47	C32	-0.3333	0.3333	-0.1667	0.360(82)
48	Cl15	-0.2921(10)	0.3623(15)	-0.2376(29)	0.324(24)
49	H19	-0.0890	0.3752	0.2138	0.095

6.4.3 Bond distances (Angstroms)

Atom A	Atom B	Distance	Atom A	Atom B	Distance
Fe1	C9	1.97(3)	C10	C11	1.48(2)
Fe1	C10	2.00(3)	C11	C12	1.24(2)
Fe1	C4	2.02(4)	C12	C13	1.54(3)
Fe1	C3	2.02(3)	C13	C14	1.37(3)
Fe1	C8	2.03(3)	C13	C18	1.43(3)
Fe1	C2	2.04(4)	C14	C15	1.37(3)
Fe1	C5	2.04(4)	C15	C16	1.39(2)
Fe1	C7	2.06(3)	C16	C17	1.35(2)
Fe1	C1	2.09(3)	C16	C19	1.54(3)

Fe1	C6	2.09(3)	C17	C18	1.30(3)
Cl1	C14	1.73(2)	C19	C26	1.53(3)
Cl2	C15	1.73(2)	C19	C20	1.55(3)
Cl3	C17	1.75(2)	C20	C21	1.37(2)
Cl4	C18	1.77(2)	C20	C25	1.39(2)
Cl5	C21	1.73(2)	C21	C22	1.39(2)
Cl6	C22	1.75(2)	C22	C23	1.37(2)
Cl7	C23	1.73(2)	C23	C24	1.37(2)
Cl8	C24	1.76(2)	C24	C25	1.35(2)
Cl9	C25	1.73(2)	C26	C31	1.41(2)
Cl10	C27	1.72(2)	C26	C27	1.42(2)
Cl11	C28	1.71(2)	C27	C28	1.39(2)
Cl12	C29	1.70(2)	C28	C29	1.38(2)
Cl13	C30	1.73(2)	C29	C30	1.36(2)
Cl14	C31	1.72(2)	C30	C31	1.39(2)
C1	C2	1.39(2)	C32	Cl15	1.66(2)
C1	C5	1.41(2)	C32	Cl15	1.66(2)
C2	C3	1.40(2)	C32	Cl15	1.66(2)
C3	C4	1.39(2)	C32	Cl15	1.66(2)
C4	C5	1.41(2)	C32	Cl15	1.66(2)
C6	C10	1.40(2)	C32	Cl15	1.66(2)
C6	C7	1.45(2)	Cl15	Cl15	2.30(5)
C7	C8	1.42(2)	Cl15	Cl15	2.30(5)
C8	C9	1.42(2)	Cl15	Cl15	2.39(5)
C9	C10	1.43(2)	Cl15	Cl15	2.39(5)

6.4.4 Bond angles (degrees) of 18

Atom A	Atom B	Atom C	Angle	Atom A	Atom B	Atom C	Angle
C9	Fe1	C10	42.2(6)	C14	C13	C12	122.7(27)
C9	Fe1	C4	116.7(14)	C18	C13	C12	121.1(27)
C10	Fe1	C4	151.9(14)	C15	C14	C13	120.8(19)
C9	Fe1	C3	148.7(14)	C15	C14	Cl1	117.9(22)
C10	Fe1	C3	167.2(14)	C13	C14	Cl1	121.3(21)
C4	Fe1	C3	40.2(7)	C14	C15	C16	121.3(17)
C9	Fe1	C8	41.6(7)	C14	C15	Cl2	119.8(22)
C10	Fe1	C8	70.3(11)	C16	C15	Cl2	118.8(20)
C4	Fe1	C8	106.4(13)	C17	C16	C15	116.8(16)
C3	Fe1	C8	114.5(13)	C17	C16	C19	125.8(22)
C9	Fe1	C2	167.7(13)	C15	C16	C19	117.3(21)
C10	Fe1	C2	130.7(13)	C18	C17	C16	123.5(18)
C4	Fe1	C2	65.2(14)	C18	C17	Cl3	115.2(19)
C3	Fe1	C2	40.3(7)	C16	C17	Cl3	121.3(19)
C8	Fe1	C2	150.7(13)	C17	C18	C13	121.4(18)
C9	Fe1	C5	105.0(14)	C17	C18	Cl4	124.8(21)
C10	Fe1	C5	117.2(14)	C13	C18	Cl4	113.8(23)
C4	Fe1	C5	40.7(7)	C26	C19	C16	119.9(18)
C3	Fe1	C5	70.6(14)	C26	C19	C20	113.9(18)
C8	Fe1	C5	125.3(14)	C16	C19	C20	118.5(16)
C2	Fe1	C5	68.0(13)	C21	C20	C25	115.7(16)
C9	Fe1	C7	69.0(12)	C21	C20	C19	119.3(18)
C10	Fe1	C7	68.8(11)	C25	C20	C19	124.2(19)
C4	Fe1	C7	127.7(13)	C20	C21	C22	122.9(15)
C3	Fe1	C7	106.6(13)	C20	C21	Cl5	119.0(17)
C8	Fe1	C7	40.8(6)	C22	C21	Cl5	118.1(16)

C2	Fe1	C7	120.4(13)	C23	C22	C21	119.5(14)
C5	Fe1	C7	164.3(13)	C23	C22	Cl6	121.0(17)
C9	Fe1	C1	128.9(13)	C21	C22	Cl6	119.4(16)
C10	Fe1	C1	110.6(12)	C24	C23	C22	117.9(15)
C4	Fe1	C1	65.2(13)	C24	C23	Cl7	123.6(16)
C3	Fe1	C1	68.1(12)	C22	C23	Cl7	118.5(16)
C8	Fe1	C1	164.8(13)	C25	C24	C23	121.9(14)
C2	Fe1	C1	39.5(7)	C25	C24	Cl8	122.5(15)
C5	Fe1	C1	40.0(7)	C23	C24	Cl8	115.6(16)
C7	Fe1	C1	154.4(13)	C24	C25	C20	121.8(15)
C9	Fe1	C6	68.8(11)	C24	C25	Cl9	115.2(15)
C10	Fe1	C6	39.9(6)	C20	C25	Cl9	122.7(16)
C4	Fe1	C6	166.7(13)	C31	C26	C27	115.2(16)
C3	Fe1	C6	129.0(14)	C31	C26	C19	127.6(18)
C8	Fe1	C6	69.1(11)	C27	C26	C19	116.8(21)
C2	Fe1	C6	112.2(13)	C28	C27	C26	122.1(16)
C5	Fe1	C6	152.1(13)	C28	C27	Cl10	119.8(18)
C7	Fe1	C6	40.9(6)	C26	C27	Cl10	118.2(18)
C1	Fe1	C6	122.0(12)	C29	C28	C27	120.2(16)
C2	C1	C5	108.6(31)	C29	C28	Cl11	121.2(18)
C2	C1	Fe1	68.3(21)	C27	C28	Cl11	118.6(19)
C5	C1	Fe1	68.1(23)	C30	C29	C28	119.5(16)
C1	C2	C3	110.9(32)	C30	C29	Cl12	122.3(20)
C1	C2	Fe1	72.2(21)	C28	C29	Cl12	118.2(18)
C3	C2	Fe1	69.4(22)	C29	C30	C31	121.3(18)
C4	C3	C2	103.1(32)	C29	C30	Cl13	118.9(18)
C4	C3	Fe1	69.8(22)	C31	C30	Cl13	119.8(18)
C2	C3	Fe1	70.3(22)	C30	C31	C26	121.7(15)
C3	C4	C5	113.5(34)	C30	C31	Cl14	118.1(20)
C3	C4	Fe1	69.9(22)	C26	C31	Cl14	119.9(17)
C5	C4	Fe1	70.2(24)	Cl15	C32	Cl15	92.3(21)
C1	C5	C4	103.3(33)	Cl15	C32	Cl15	87.6(21)
C1	C5	Fe1	71.9(23)	Cl15	C32	Cl15	87.6(21)
C4	C5	Fe1	69.1(23)	Cl15	C32	Cl15	87.6(21)
C10	C6	C7	107.0(25)	Cl15	C32	Cl15	179.994(8)
C10	C6	Fe1	66.5(17)	Cl15	C32	Cl15	92.3(21)
C7	C6	Fe1	68.2(17)	Cl15	C32	Cl15	179.996(7)
C8	C7	C6	108.7(26)	Cl15	C32	Cl15	87.6(21)
C8	C7	Fe1	68.5(19)	Cl15	C32	Cl15	92.3(21)
C6	C7	Fe1	70.8(17)	Cl15	C32	Cl15	92.4(21)
C9	C8	C7	106.7(27)	Cl15	C32	Cl15	92.3(21)
C9	C8	Fe1	67.0(18)	Cl15	C32	Cl15	92.3(21)
C7	C8	Fe1	70.8(19)	Cl15	C32	Cl15	179.995(7)
C8	C9	C10	108.9(26)	Cl15	C32	Cl15	87.7(21)
C8	C9	Fe1	71.4(19)	Cl15	C32	Cl15	87.7(21)
C10	C9	Fe1	70.1(17)	C32	Cl15	Cl15	46.2(10)
C6	C10	C9	108.6(25)	C32	Cl15	Cl15	46.2(10)
C6	C10	C11	134.9(29)	Cl15	Cl15	Cl15	62.8(25)
C9	C10	C11	116.4(26)	C32	Cl15	Cl15	43.8(10)
C6	C10	Fe1	73.6(17)	Cl15	Cl15	Cl15	58.6(13)
C9	C10	Fe1	67.7(17)	Cl15	Cl15	Cl15	90.003(5)
C11	C10	Fe1	127.2(22)	C32	Cl15	Cl15	43.8(10)
C12	C11	C10	129.8(32)	Cl15	Cl15	Cl15	89.999(5)
C11	C12	C13	131.2(25)	Cl15	Cl15	Cl15	58.6(13)
C14	C13	C18	116.1(17)	Cl15	Cl15	Cl15	60.000(3)

6.5 CRYSTALLOGRAPHIC DATA OF 21

Single crystals of **21** were grown by slow evaporation of a chloroform solution of **21** and used for its X-Ray crystal determination. An ORTEP view of **21** is shown. The structure reveals totally alternated cyclopentadienyl rings, due to the high steric hindrance of their methyl groups. A trans configuration of the CH=CH unit is also observed. Due to the available resonance pathway and the trans configuration, the ($H\text{-}C_5H_4$) and the ($C_6H_2Cl_4$) rings are almost in a coplanar disposition twisted by a low angle of ~11.9 °. It is likely that the presence of electronically attractive interactions between the CH_3 and chlorines of the benzene unit also causes this coplanarity (Cl-----H = 3.167Å). The solid-state packing of **21** (see section 8.1.5.4) is best described as centrosymmetrically related pairs of molecules. Stacking of these related pairs occurs with continuous staggering along the c axis and with a head-to-tail pairing along the b axis. Under these circumstances, the largest driving forces for such molecular packing are Cl---H interaction, dipole-dipole and van der Waals interactions, which lead to an efficient filling of space. Solvent molecules have been omitted from the crystal packing for more clarity.

Dieder angles (for twisting):
From the major part
1. C(13)---C(18) : C(13)-C(12)-C(11) 45(7) °
2. C(9)---C(10) : C(10)-C(11)-C(12) 42(1) °

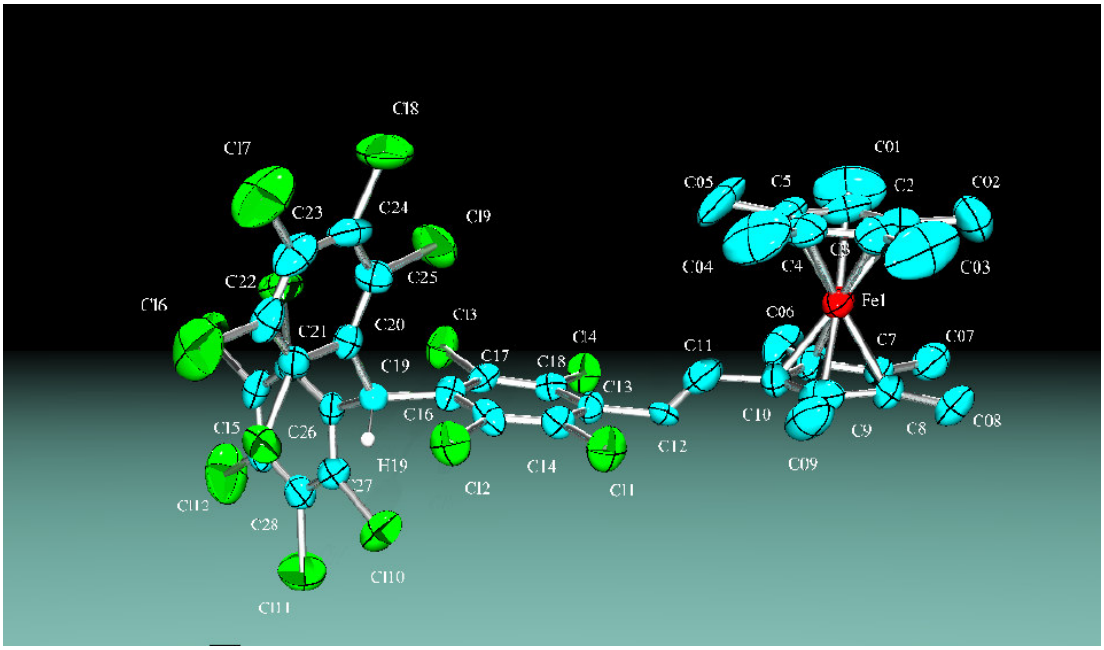

Figure 65. Molecular structure of **21**

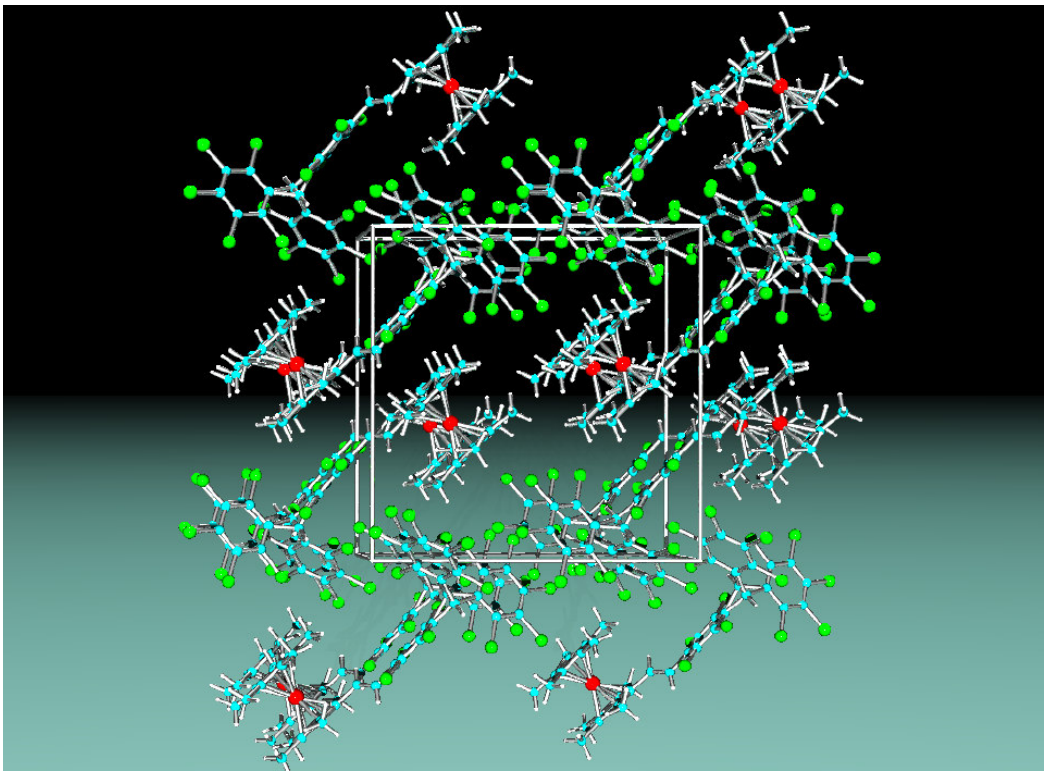

Figure 66. Crystal packing of **21**

6.5.1 Crystallographic data

Molecular formula	$C_{40}H_{30}Cl_{14}Fe$ x $CHCl_3$
Formula weight	1182.16
Crystal system	triclinic
Space group	*P-1*
a, pm	8.9933(5)
b, pm	16.308(1)
c, pm	16.688(1)
α,deg	89.893(3)
β,deg	88.832(5)
γ,deg	81.872(5)
V, nm^3	2422.4(2)
Z	2
h (min-max)	0-8
k (min-max)	-15-16
l (min-max)	-16-16
cryst color,habit	Red plate
Size	0.29x0.14x0.05
F(000)	1184
temp, K	233(2)
Scan range	ϕ and ω scans
Reflections collected	8980

Independent reflections	4840 (R_{int}=0.0766)
diffractometer	Kappa CCD
Radiation	MoKα (λ=0.71073 Å)
Refinement method	Full-matrix least-squares on F^2
R(F), %[a]	0.105
R(wF^2), %[a]	0.1612

[a] Quantity minimized = $R(wF^2) = \sum\left[w\left(F_0^{\,2} - F_c^{\,2}\right)^2\right] / \sum\left[w\left(F_0^{\,2}\right)^2\right]^{1/2}$;

$$R = \sum \Delta / \sum (F_0), \Delta = \left|(F_0 - F_c)\right|$$

Refinement details: disorder of solvent molecule $CHCl_3$ (1:1 Cl17:Cl1A) and disorder of the C-H group C19:C19A.

6.5.2 Atomic coordinates (Å) and equivalent isotropic displacement parameters (pm^2)

Num	Atom	X/Å	Y/Å	Z/Å	Ueqq
1	Fe1	0.07100(13)	0.23136(7)	0.41087(7)	0.0511(4)
2	Cl1	-0.3540(2)	-0.02303(14)	0.33926(14)	0.0650(7)
3	Cl2	-0.4166(2)	-0.14742(14)	0.21343(14)	0.0638(7)
4	Cl3	0.1808(2)	-0.21198(14)	0.14379(13)	0.0611(7)
5	Cl4	0.2427(2)	-0.10836(14)	0.28653(13)	0.0580(7)
6	Cl5	-0.4174(3)	-0.32192(16)	0.07223(16)	0.0800(8)
7	Cl6	-0.6210(3)	-0.2479(3)	-0.0620(2)	0.1259(13)
8	Cl7	-0.5712(4)	-0.0795(2)	-0.1415(2)	0.1334(14)
9	Cl8	-0.3053(4)	0.00670(18)	-0.08938(18)	0.1086(11)
10	Cl9	-0.0878(3)	-0.07170(17)	0.03526(18)	0.0905(9)
11	Cl10	-0.1579(3)	-0.35220(17)	0.25349(14)	0.0803(8)
12	Cl11	0.0445(4)	-0.51953(18)	0.23939(18)	0.1030(10)
13	Cl12	0.2450(4)	-0.56220(19)	0.0890(2)	0.1229(13)
14	Cl13	0.2168(3)	-0.44454(19)	-0.05750(17)	0.0941(10)
15	Cl14	0.0127(3)	-0.27757(16)	-0.04805(14)	0.0726(8)
16	C1	0.2012(12)	0.2948(8)	0.3376(6)	0.080(3)
17	C2	0.1047(16)	0.3501(6)	0.3869(6)	0.082(3)
18	C3	-0.0411(12)	0.3401(6)	0.3695(6)	0.073(3)
19	C4	-0.0403(11)	0.2778(7)	0.3108(6)	0.071(3)
20	C5	0.1134(12)	0.2493(6)	0.2905(5)	0.068(3)
21	C6	0.2033(11)	0.1325(6)	0.4590(5)	0.062(3)
22	C7	0.1696(9)	0.1956(5)	0.5169(5)	0.048(2)
23	C8	0.0127(10)	0.2144(5)	0.5298(5)	0.055(2)
24	C9	-0.0521(10)	0.1596(6)	0.4769(6)	0.068(3)
25	C10	0.0641(12)	0.1085(5)	0.4347(5)	0.061(3)
26	C11	0.0519(12)	0.0442(7)	0.3734(6)	0.081(3)
27	C12	-0.0338(9)	-0.0091(5)	0.3722(5)	0.044(2)
28	C13	-0.0589(8)	-0.0669(5)	0.3079(4)	0.040(2)
29	C14	-0.2036(8)	-0.0783(5)	0.2873(5)	0.045(2)
30	C15	-0.2305(8)	-0.1330(5)	0.2286(5)	0.047(2)
31	C16	-0.1158(8)	-0.1770(5)	0.1818(5)	0.044(2)
32	C17	0.0284(8)	-0.1643(5)	0.1999(4)	0.039(2)

33	C18	0.0557(7)	-0.1130(5)	0.2629(5)	0.040(2)
34	C20	-0.2571(8)	-0.1953(5)	0.0498(5)	0.044(2)
35	C21	-0.3794(9)	-0.2330(5)	0.0250(5)	0.049(2)
36	C22	-0.4756(9)	-0.1987(7)	-0.0341(6)	0.061(3)
37	C23	-0.4543(11)	-0.1253(7)	-0.0690(6)	0.070(3)
38	C24	-0.3353(11)	-0.0847(6)	-0.0459(5)	0.064(3)
39	C25	-0.2386(8)	-0.1209(6)	0.0122(5)	0.052(2)
40	C26	-0.0622(8)	-0.3166(5)	0.1043(5)	0.042(2)
41	C27	-0.0521(9)	-0.3742(6)	0.1662(5)	0.050(2)
42	C28	0.0408(11)	-0.4495(6)	0.1615(6)	0.062(3)
43	C29	0.1233(10)	-0.4702(5)	0.0934(7)	0.063(3)
44	C30	0.1117(9)	-0.4173(6)	0.0289(5)	0.059(3)
45	C31	0.0193(8)	-0.3410(5)	0.0349(5)	0.044(2)
46	C01	0.3731(12)	0.2879(10)	0.3322(9)	0.151(6)
47	C02	0.1543(18)	0.4136(7)	0.4429(7)	0.145(6)
48	C03	-0.1835(15)	0.3902(9)	0.4068(8)	0.171(7)
49	C04	-0.1757(13)	0.2500(9)	0.2723(7)	0.144(6)
50	C05	0.1719(15)	0.1873(8)	0.2288(6)	0.130(5)
51	C06	0.3563(11)	0.0901(6)	0.4321(6)	0.091(4)
52	C07	0.2881(10)	0.2343(6)	0.5600(5)	0.075(3)
53	C08	-0.0649(10)	0.2777(6)	0.5863(5)	0.074(3)
54	C09	-0.2221(11)	0.1612(7)	0.4741(6)	0.098(4)
55	C32	0.429(2)	-0.4449(14)	0.3010(11)	0.225(11)
56	Cl15	0.4854(6)	-0.4443(3)	0.3989(3)	0.200(2)
57	Cl16	0.5473(7)	-0.5069(4)	0.2400(3)	0.215(2)
58	Cl17	0.4248(13)	-0.3282(7)	0.2938(8)	0.189(5)
59	Cl1A	0.3512(10)	-0.3717(6)	0.2518(5)	0.129(3)
60	C19	-0.1746(13)	-0.2389(7)	0.1232(7)	0.035(3)
61	C19A	-0.114(3)	-0.2214(16)	0.0964(16)	0.047(7)

6.5.3 Bond distances (Angstoms)

Atom A	Atom B	Distance	Atom A	Atom B	Distance
Fe1	C9	2.030(9)	C7	C07	1.508(11)
Fe1	C6	2.038(8)	C8	C9	1.445(12)
Fe1	C2	2.039(10)	C8	C08	1.488(11)
Fe1	C3	2.039(9)	C9	C10	1.418(12)
Fe1	C7	2.042(8)	C9	C09	1.527(12)
Fe1	C10	2.051(9)	C10	C11	1.481(13)
Fe1	C4	2.053(9)	C11	C12	1.242(12)
Fe1	C1	2.053(10)	C12	C13	1.470(11)
Fe1	C5	2.065(9)	C13	C14	1.390(10)
Fe1	C8	2.071(8)	C13	C18	1.395(10)
Cl1	C14	1.734(7)	C14	C15	1.374(10)
Cl2	C15	1.746(7)	C15	C16	1.397(10)
Cl3	C17	1.735(7)	C16	C17	1.381(10)
Cl4	C18	1.748(7)	C16	C19	1.559(13)
Cl5	C21	1.723(9)	C16	C19A	1.60(3)
Cl6	C22	1.701(9)	C17	C18	1.391(10)

Cl7	C23	1.719(9)	C20	C25	1.393(11)
Cl8	C24	1.709(9)	C20	C21	1.403(10)
Cl9	C25	1.721(8)	C20	C19A	1.53(2)
Cl10	C27	1.733(8)	C20	C19	1.562(13)
Cl11	C28	1.727(9)	C21	C22	1.388(11)
Cl12	C29	1.728(9)	C22	C23	1.367(13)
Cl13	C30	1.732(9)	C23	C24	1.397(13)
Cl14	C31	1.724(8)	C24	C25	1.390(12)
C1	C5	1.409(13)	C26	C31	1.386(10)
C1	C2	1.412(14)	C26	C27	1.392(11)
C1	C01	1.535(14)	C26	C19	1.533(13)
C2	C3	1.380(14)	C26	C19A	1.56(3)
C2	C02	1.515(14)	C27	C28	1.385(11)
C3	C4	1.412(13)	C28	C29	1.361(12)
C3	C03	1.539(13)	C29	C30	1.374(12)
C4	C5	1.429(12)	C30	C31	1.398(11)
C4	C04	1.514(14)	C32	Cl1A	1.541(18)
C5	C05	1.480(12)	C32	Cl16	1.686(19)
C6	C7	1.410(11)	C32	Cl15	1.720(18)
C6	C10	1.430(12)	C32	Cl17	1.90(2)
C6	C06	1.510(12)	Cl17	Cl1A	1.259(14)
C7	C8	1.413(11)			

6.5.4 Bond angles (degrees)

Atom A	Atom B	Atom C	Angle	Atom A	Atom B	Atom C	Angle
C9	Fe1	C6	68.5(4)	C08	C8	Fe1	127.3(6)
C9	Fe1	C2	144.6(5)	C10	C9	C8	109.6(7)
C6	Fe1	C2	132.9(5)	C10	C9	C09	129.6(9)
C9	Fe1	C3	116.3(4)	C8	C9	C09	120.8(9)
C6	Fe1	C3	172.0(5)	C10	C9	Fe1	70.5(5)
C2	Fe1	C3	39.5(4)	C8	C9	Fe1	70.9(5)
C9	Fe1	C7	67.8(3)	C09	C9	Fe1	126.1(7)
C6	Fe1	C7	40.4(3)	C9	C10	C6	107.0(8)
C2	Fe1	C7	108.8(4)	C9	C10	C11	129.0(9)
C3	Fe1	C7	133.9(4)	C6	C10	C11	123.9(9)
C9	Fe1	C10	40.7(4)	C9	C10	Fe1	68.9(5)
C6	Fe1	C10	40.9(3)	C6	C10	Fe1	69.0(5)
C2	Fe1	C10	173.3(5)	C11	C10	Fe1	125.0(7)
C3	Fe1	C10	146.8(5)	C12	C11	C10	129.2(10)
C7	Fe1	C10	68.0(3)	C11	C12	C13	129.7(8)
C9	Fe1	C4	111.9(4)	C14	C13	C18	114.9(7)
C6	Fe1	C4	145.6(4)	C14	C13	C12	120.9(7)
C2	Fe1	C4	67.8(4)	C18	C13	C12	124.2(7)
C3	Fe1	C4	40.4(4)	C15	C14	C13	122.2(7)
C7	Fe1	C4	173.9(4)	C15	C14	Cl1	119.3(6)
C10	Fe1	C4	115.9(4)	C13	C14	Cl1	118.5(6)
C9	Fe1	C1	174.9(5)	C14	C15	C16	122.7(7)
C6	Fe1	C1	108.9(4)	C14	C15	Cl2	117.8(6)
C2	Fe1	C1	40.4(4)	C16	C15	Cl2	119.5(6)
C3	Fe1	C1	66.8(4)	C17	C16	C15	115.7(7)
C7	Fe1	C1	113.4(4)	C17	C16	C19	131.3(7)

C10	Fe1	C1	134.6(4)	C15	C16	C19	112.7(7)
C4	Fe1	C1	67.5(4)	C17	C16	C19A	109.2(11)
C9	Fe1	C5	136.3(4)	C15	C16	C19A	132.9(11)
C6	Fe1	C5	113.9(4)	C19	C16	C19A	28.7(9)
C2	Fe1	C5	67.9(4)	C16	C17	C18	121.4(6)
C3	Fe1	C5	67.6(4)	C16	C17	Cl3	120.4(6)
C7	Fe1	C5	143.7(4)	C18	C17	Cl3	118.2(6)
C10	Fe1	C5	110.8(4)	C17	C18	C13	123.0(6)
C4	Fe1	C5	40.6(4)	C17	C18	Cl4	117.7(5)
C1	Fe1	C5	40.0(4)	C13	C18	Cl4	119.3(6)
C9	Fe1	C8	41.3(3)	C25	C20	C21	116.1(7)
C6	Fe1	C8	68.8(3)	C25	C20	C19A	106.1(12)
C2	Fe1	C8	112.6(4)	C21	C20	C19A	136.4(13)
C3	Fe1	C8	110.1(4)	C25	C20	C19	129.9(8)
C7	Fe1	C8	40.2(3)	C21	C20	C19	113.5(8)
C10	Fe1	C8	69.2(3)	C19A	C20	C19	29.4(10)
C4	Fe1	C8	135.6(4)	C22	C21	C20	122.1(8)
C1	Fe1	C8	142.7(4)	C22	C21	Cl5	118.6(7)
C5	Fe1	C8	176.0(4)	C20	C21	Cl5	119.3(7)
C5	C1	C2	108.8(9)	C23	C22	C21	120.0(8)
C5	C1	C01	125.0(11)	C23	C22	Cl6	120.2(8)
C2	C1	C01	126.2(12)	C21	C22	Cl6	119.8(9)
C5	C1	Fe1	70.4(6)	C22	C23	C24	120.3(8)
C2	C1	Fe1	69.3(6)	C22	C23	Cl7	122.0(9)
C01	C1	Fe1	128.7(8)	C24	C23	Cl7	117.7(9)
C3	C2	C1	107.6(9)	C25	C24	C23	118.7(9)
C3	C2	C02	126.9(12)	C25	C24	Cl8	120.2(8)
C1	C2	C02	125.3(13)	C23	C24	Cl8	121.1(8)
C3	C2	Fe1	70.2(6)	C24	C25	C20	122.8(8)
C1	C2	Fe1	70.4(6)	C24	C25	Cl9	117.3(8)
C02	C2	Fe1	128.8(7)	C20	C25	Cl9	119.9(6)
C2	C3	C4	109.6(8)	C31	C26	C27	115.9(7)
C2	C3	C03	125.5(12)	C31	C26	C19	131.1(9)
C4	C3	C03	124.8(12)	C27	C26	C19	112.7(8)
C2	C3	Fe1	70.2(6)	C31	C26	C19A	106.4(12)
C4	C3	Fe1	70.3(5)	C27	C26	C19A	135.9(12)
C03	C3	Fe1	126.7(8)	C19	C26	C19A	29.4(9)
C3	C4	C5	107.0(8)	C28	C27	C26	122.5(8)
C3	C4	C04	126.9(11)	C28	C27	Cl10	117.8(7)
C5	C4	C04	126.0(11)	C26	C27	Cl10	119.7(6)
C3	C4	Fe1	69.3(5)	C29	C28	C27	119.9(8)
C5	C4	Fe1	70.2(5)	C29	C28	Cl11	119.8(8)
C04	C4	Fe1	128.6(8)	C27	C28	Cl11	120.2(8)
C1	C5	C4	107.0(8)	C28	C29	C30	119.9(8)
C1	C5	C05	125.5(11)	C28	C29	Cl12	119.9(8)
C4	C5	C05	127.4(11)	C30	C29	Cl12	120.2(8)
C1	C5	Fe1	69.5(5)	C29	C30	C31	119.6(8)
C4	C5	Fe1	69.2(5)	C29	C30	Cl13	119.5(8)
C05	C5	Fe1	128.8(7)	C31	C30	Cl13	120.8(8)
C7	C6	C10	107.5(8)	C26	C31	C30	122.0(8)
C7	C6	C06	127.8(9)	C26	C31	Cl14	121.2(7)
C10	C6	C06	124.5(8)	C30	C31	Cl14	116.8(7)
C7	C6	Fe1	70.0(5)	Cl1A	C32	Cl16	108.9(11)
C10	C6	Fe1	70.0(5)	Cl1A	C32	Cl15	127.8(15)
C06	C6	Fe1	129.9(7)	Cl16	C32	Cl15	114.2(10)
C6	C7	C8	110.7(7)	Cl1A	C32	Cl17	41.2(7)
C6	C7	C07	123.4(8)	Cl16	C32	Cl17	119.1(13)

C8	C7	C07	125.9(7)	Cl15	C32	Cl17	91.0(12)
C6	C7	Fe1	69.6(5)	Cl1A	Cl17	C32	53.8(9)
C8	C7	Fe1	71.0(5)	Cl17	Cl1A	C32	84.9(11)
C07	C7	Fe1	128.0(6)	C26	C19	C16	114.4(8)
C7	C8	C9	105.1(7)	C26	C19	C20	116.0(8)
C7	C8	C08	126.1(8)	C16	C19	C20	113.1(8)
C9	C8	C08	128.8(9)	C20	C19A	C26	116.2(17)
C7	C8	Fe1	68.8(5)	C20	C19A	C16	112.7(16)
C9	C8	Fe1	67.9(5)	C26	C19A	C16	110.8(16)