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5,8,13,13-Tetrachloro-13*H*-dibenzo[*a*,*i*]fluorene cyclohexane hemisolvate

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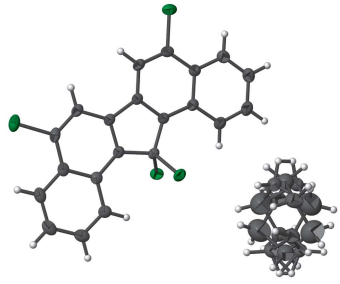
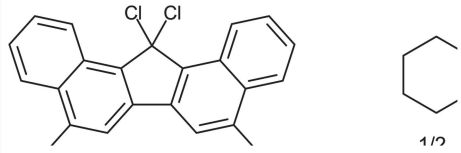
In the crystal structure of the solvated pentacyclic title compound, $C_{21}H_{10}Cl_4{\cdot}0.5C_6H_{12}$, the pentacyclic chloroaromatic rings are arranged in parallel layers, with the chlorine atoms protruding from these planes. Channels orthogonal to these layers are filled with disordered cyclohexane molecules.

3D view



Chemical scheme



Structure description

The pentacyclic compound $C_{21}H_{20}Cl_4$ (Fig. 1) appeared as a new byproduct in the synthesis of the 13,13-dichloro analogue, an intermediate for bis-(dibenzo[*a.i*])fluorenylidene (Bergmann *et al.*, 1953, Franzen & Joschek, 1961), a biradical still under discussion (Kanawati *et al.*, 2012; Wentrup *et al.*, 2016). Eight identical molecules fill the unit cell, these aromatic compounds are essentially planar, the largest deviation from the mean plane being 0.0164 (18) Å at C8. With a bond angle of 107.26 (9)°, the Cl1—C1—Cl2 unit makes an angle of 89.97 (11)° to the aromatic plane. In the crystal, the molecules are arranged in layers parallel to the *ac* plane with an interlayer spacing of 3.28 Å. Channels along the *b*-axis direction (Fig. 2) are filled with one disordered cyclohexane molecule per two dibenzofluorene molecules.

Synthesis and crystallization

Bis-α-naphthylketone (10.0 g), prepared from α-cyanonaphthalene according to Blicke (1927), was added to PCl_5 (13.0 g) and the mixture was heated to 423 K for 5 h. Following the procedure of Magidson (1925), additional PCl_5 (13.0 g) was added, and after 5 h at 423 K, cooled to ambient temperature and the residue washed with light petroleum. The yield after threefold recrystallization from toluene solution was 0.6 g of a yellow solid with m.p. = 505–509 K. Single crystals were grown by slow evaporation of a solution in chloroform/cyclohexane (1/2). IR (KBr): 3420, 3072, 1620, 1566, 1518, 1421, 1376, 1344,

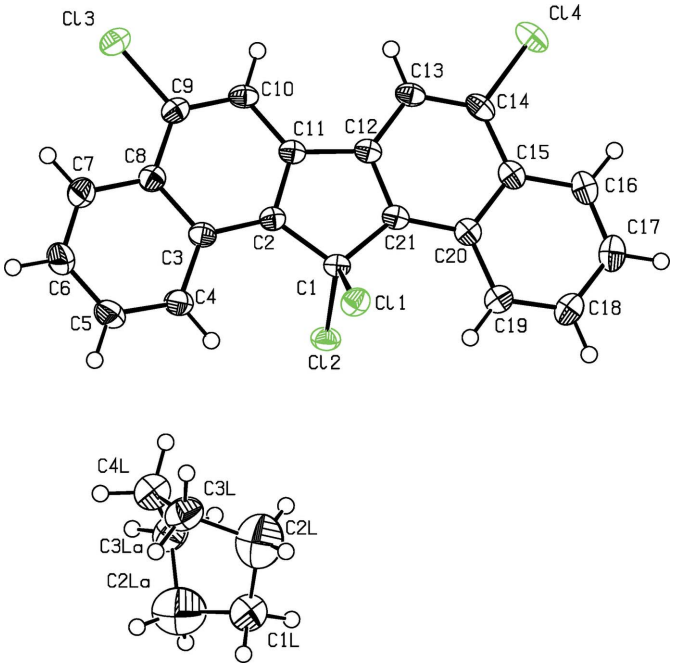


Figure 1
Perspective view of the title compound. Displacement ellipsoids are drawn at the 50% probability level. Only one component of the disordered cyclohexane solvent molecule is shown.

1288, 1261, 1195, 1161, 1072, 1029, 956, 930, 853, 795, 764, 728, 623, 584, 533, 519, 501, 429. ^{1}H NMR (400 MHz, $CDCl_3$): 8.77 (*dd*, 2H, 6-H), 8.41 (*d*, 2 H, 3-H), 7.91 (2, 2 H, 1-H), 7.80 (*m*, 2 H, 5-H), 7.69 (*m*, 2 H, 4-H).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. The solvent molecule (cyclohexane) is highly disordered and was refined using split positions. The s.o.f. were kept fixed due to the imposed symmetry. The displacement parameters of the solvent atoms were restrained to approximate isotropic behaviour. Equivalent bond lengths and angles involving the disordered atoms were restrained to be equal.

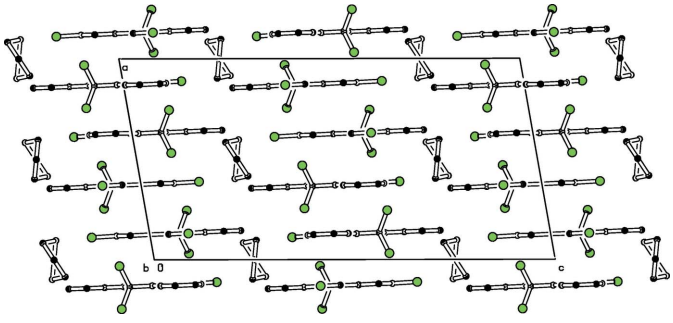


Figure 2
Part of the packing diagram. View along the *b* axis.

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_{21}H_{10}Cl_4{\cdot}0.5C_6H_{12}$
M_r	446.17
Crystal system, space group	Monoclinic, *I*2/*c*
Temperature (K)	120
a, *b*, *c* (Å)	13.7822 (6), 10.7752 (4), 27.0787 (13)
β (°)	99.901 (4)
V (Å^3)	3961.5 (3)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.61
Crystal size (mm)	0.90 × 0.37 × 0.08
Data collection	
Diffractometer	Stoe IPDS 2T
Absorption correction	Integration (*X-RED* and *X-AREA*; Stoe & Cie, 1996)
T_{min}, T_{max}	0.736, 0.952
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	11344, 4935, 4194
R_{int}	0.018
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.668
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.039, 0.102, 1.05
No. of reflections	4935
No. of parameters	290
No. of restraints	66
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.51, −0.51

Computer programs: *X-RED* and *X-AREA* (Stoe & Cie, 1996), *SIR2004* (Altomare *et al.*, 1999), *SHELXL2018* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

Acknowledgements

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full crystallographic data

IUCrData (2019). **4**, x190236 [https://doi.org/10.1107/S2414314619002360]

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Crystal data

$C_{21}H_{10}Cl_4{\cdot}0.5C_6H_{12}$
M_r = 446.17
Monoclinic, *I*2/*c*
a = 13.7822 (6) Å
b = 10.7752 (4) Å
c = 27.0787 (13) Å
β = 99.901 (4)°
V = 3961.5 (3) Å^3
Z = 8
F(000) = 1824
D_x = 1.496 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 17812 reflections
θ = 2.0–28.6°
μ = 0.61 mm^{-1}
T = 120 K
Plate, colourless
0.90 × 0.37 × 0.08 mm

Data collection

Stoe IPDS 2T diffractometer
Radiation source: sealed X-ray tube, 12 x 0.4 mm long-fine focus
Detector resolution: 6.67 pixels mm^{-1}
rotation method scans
Absorption correction: integration (X-RED and X-AREA; Stoe & Cie, 1996)
T_{min} = 0.736, T_{max} = 0.952
11344 measured reflections
4935 independent reflections
4194 reflections with $I > 2\sigma(I)$
R_{int} = 0.018
θ_{max} = 28.4°, θ_{min} = 2.0°
h = −18→18
k = −14→14
l = −33→36

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.102
S = 1.05
4935 reflections
290 parameters
66 restraints
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.047P)^2 + 6.8104P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.50 e Å^{-3}

Special details

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

Refinement. Hydrogen atoms attached to carbons were placed at calculated positions and were refined in the riding-model approximation with isotropic displacement parameters.

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Cl1	0.74520 (3)	0.24458 (4)	0.60278 (2)	0.02905 (11)	
Cl2	0.53125 (3)	0.23873 (4)	0.58942 (2)	0.02864 (11)	
Cl3	0.63077 (3)	0.87411 (4)	0.59660 (2)	0.03174 (12)	
Cl4	0.61148 (4)	0.43078 (5)	0.35395 (2)	0.03516 (13)	
C1	0.63442 (12)	0.32199 (16)	0.57254 (6)	0.0221 (3)	
C2	0.63383 (12)	0.45706 (16)	0.58782 (7)	0.0217 (3)	
C3	0.63784 (13)	0.51050 (17)	0.63570 (7)	0.0244 (3)	
C4	0.64182 (15)	0.44129 (19)	0.68067 (7)	0.0307 (4)	
H4	0.642579	0.353188	0.679465	0.037*	
C5	0.64455 (17)	0.4998 (2)	0.72553 (8)	0.0387 (5)	
H5	0.646238	0.452034	0.755159	0.046*	
C6	0.64488 (18)	0.6299 (2)	0.72840 (8)	0.0403 (5)	
H6	0.647220	0.669447	0.759936	0.048*	
C7	0.64187 (16)	0.6999 (2)	0.68613 (8)	0.0340 (4)	
H7	0.642775	0.787869	0.688647	0.041*	
C8	0.63741 (13)	0.64337 (17)	0.63870 (7)	0.0254 (4)	
C9	0.63181 (12)	0.71300 (17)	0.59358 (7)	0.0247 (4)	
C10	0.62704 (12)	0.65911 (17)	0.54760 (7)	0.0242 (3)	
H10	0.622833	0.708019	0.518124	0.029*	
C11	0.62854 (12)	0.52906 (16)	0.54535 (6)	0.0215 (3)	
C12	0.62551 (12)	0.44863 (16)	0.50131 (7)	0.0221 (3)	
C13	0.62051 (12)	0.48153 (17)	0.45073 (7)	0.0248 (4)	
H13	0.618390	0.566095	0.440677	0.030*	
C14	0.61879 (12)	0.38833 (19)	0.41650 (7)	0.0257 (4)	
C15	0.62220 (12)	0.26011 (18)	0.42978 (7)	0.0249 (4)	
C16	0.62121 (13)	0.1634 (2)	0.39421 (7)	0.0294 (4)	
H16	0.618368	0.183259	0.359817	0.035*	
C17	0.62430 (14)	0.0420 (2)	0.40878 (8)	0.0331 (4)	
H17	0.623363	−0.021715	0.384455	0.040*	
C18	0.62885 (14)	0.01062 (19)	0.45940 (8)	0.0315 (4)	
H18	0.631065	−0.074267	0.469000	0.038*	
C19	0.63012 (13)	0.10069 (17)	0.49512 (7)	0.0272 (4)	
H19	0.632970	0.077843	0.529224	0.033*	
C20	0.62723 (12)	0.22794 (17)	0.48156 (7)	0.0237 (3)	
C21	0.62889 (12)	0.32607 (16)	0.51631 (6)	0.0224 (3)	
C1L	0.500000	−0.0571 (8)	0.750000	0.053 (2)	0.5
H1L1	0.530101	−0.111210	0.778039	0.063*	0.25
H1L2	0.469897	−0.111206	0.721960	0.063*	0.25
C2L	0.5837 (6)	0.0253 (8)	0.7298 (4)	0.098 (3)	0.5
H2L1	0.646442	−0.021233	0.733130	0.117*	0.5
H2L2	0.562314	0.047455	0.694063	0.117*	0.5
C3L	0.5967 (4)	0.1440 (5)	0.7630 (2)	0.0488 (11)	0.5
H3L1	0.609680	0.121636	0.798953	0.059*	0.5
H3L2	0.652236	0.194808	0.755464	0.059*	0.5
C4L	0.500000	0.2150 (7)	0.750000	0.0497 (19)	0.5

H4L1	0.493161	0.269490	0.778629	0.060*	0.25
H4L2	0.506841	0.269488	0.721371	0.060*	0.25
C1M	0.4912 (6)	−0.0506 (8)	0.7717 (3)	0.0326 (17)	0.25
H1M1	0.492958	−0.136370	0.757813	0.039*	0.25
H1M2	0.479821	−0.055842	0.806770	0.039*	0.25
C2M	0.5902 (3)	0.0093 (4)	0.76390 (19)	0.0419 (10)	0.5
H2M1	0.620698	0.029383	0.798809	0.050*	0.5
H2M2	0.634240	−0.049693	0.750461	0.050*	0.5
C3M	0.5827 (7)	0.1294 (8)	0.7328 (4)	0.094 (2)	0.5
H3M1	0.595833	0.107738	0.698994	0.112*	0.5
H3M2	0.635843	0.186338	0.748122	0.112*	0.5
C4M	0.4818 (7)	0.2036 (8)	0.7269 (4)	0.0376 (19)	0.25
H4M1	0.447575	0.199768	0.691691	0.045*	0.25
H4M2	0.493363	0.291640	0.736596	0.045*	0.25

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0292 (2)	0.0266 (2)	0.0289 (2)	0.00530 (16)	−0.00196 (17)	0.00312 (17)
Cl2	0.0305 (2)	0.0274 (2)	0.0290 (2)	−0.00694 (16)	0.00791 (17)	0.00400 (17)
Cl3	0.0293 (2)	0.0222 (2)	0.0446 (3)	−0.00073 (16)	0.00885 (19)	0.00088 (18)
Cl4	0.0333 (2)	0.0510 (3)	0.0216 (2)	−0.0015 (2)	0.00606 (17)	0.0058 (2)
C1	0.0207 (8)	0.0226 (8)	0.0227 (8)	−0.0010 (6)	0.0029 (6)	0.0036 (6)
C2	0.0179 (7)	0.0224 (8)	0.0246 (8)	−0.0006 (6)	0.0036 (6)	0.0016 (6)
C3	0.0215 (8)	0.0261 (8)	0.0255 (8)	−0.0015 (6)	0.0038 (6)	0.0025 (7)
C4	0.0364 (10)	0.0289 (9)	0.0268 (9)	−0.0042 (8)	0.0058 (8)	0.0025 (7)
C5	0.0519 (13)	0.0398 (11)	0.0250 (9)	−0.0052 (10)	0.0086 (9)	0.0034 (9)
C6	0.0541 (13)	0.0402 (12)	0.0275 (10)	−0.0079 (10)	0.0098 (9)	−0.0065 (9)
C7	0.0385 (11)	0.0307 (10)	0.0333 (10)	−0.0060 (8)	0.0080 (8)	−0.0051 (8)
C8	0.0215 (8)	0.0261 (8)	0.0290 (9)	−0.0025 (7)	0.0052 (7)	−0.0006 (7)
C9	0.0178 (7)	0.0223 (8)	0.0343 (9)	−0.0009 (6)	0.0051 (7)	0.0010 (7)
C10	0.0182 (8)	0.0254 (8)	0.0294 (9)	−0.0007 (6)	0.0049 (7)	0.0067 (7)
C11	0.0157 (7)	0.0244 (8)	0.0246 (8)	0.0002 (6)	0.0036 (6)	0.0033 (6)
C12	0.0164 (7)	0.0258 (8)	0.0240 (8)	−0.0004 (6)	0.0031 (6)	0.0034 (7)
C13	0.0189 (8)	0.0286 (9)	0.0272 (9)	−0.0004 (6)	0.0048 (6)	0.0071 (7)
C14	0.0177 (8)	0.0391 (10)	0.0203 (8)	−0.0003 (7)	0.0035 (6)	0.0042 (7)
C15	0.0163 (7)	0.0335 (9)	0.0246 (8)	−0.0004 (6)	0.0029 (6)	−0.0005 (7)
C16	0.0206 (8)	0.0415 (11)	0.0255 (9)	−0.0002 (7)	0.0029 (7)	−0.0059 (8)
C17	0.0229 (9)	0.0390 (11)	0.0364 (10)	−0.0001 (8)	0.0017 (8)	−0.0123 (9)
C18	0.0248 (9)	0.0283 (9)	0.0399 (11)	−0.0002 (7)	0.0012 (8)	−0.0045 (8)
C19	0.0235 (8)	0.0271 (9)	0.0300 (9)	−0.0005 (7)	0.0018 (7)	0.0002 (7)
C20	0.0168 (7)	0.0281 (8)	0.0257 (8)	−0.0001 (6)	0.0019 (6)	0.0001 (7)
C21	0.0180 (7)	0.0253 (8)	0.0236 (8)	0.0005 (6)	0.0030 (6)	0.0033 (7)
C1L	0.055 (3)	0.049 (3)	0.055 (3)	0.000	0.0094 (19)	0.000
C2L	0.096 (3)	0.096 (3)	0.101 (3)	0.0071 (19)	0.0155 (19)	−0.0107 (19)
C3L	0.0497 (18)	0.0403 (17)	0.0541 (19)	−0.0057 (15)	0.0023 (15)	0.0018 (15)
C4L	0.051 (2)	0.046 (2)	0.052 (2)	0.000	0.0086 (18)	0.000
C1M	0.034 (2)	0.032 (2)	0.032 (2)	−0.0033 (17)	0.0066 (18)	0.0050 (18)

C2M	0.0451 (17)	0.0398 (17)	0.0400 (17)	−0.0001 (14)	0.0049 (14)	0.0038 (14)
C3M	0.094 (3)	0.092 (3)	0.097 (3)	0.0002 (19)	0.0210 (19)	0.0097 (19)
C4M	0.042 (2)	0.035 (2)	0.037 (3)	−0.0017 (18)	0.0076 (18)	0.0019 (19)

Geometric parameters (Å, º)

Cl1—C1	1.8077 (17)	C17—H17	0.9500
Cl2—C1	1.8052 (17)	C18—C19	1.368 (3)
Cl3—C9	1.7380 (19)	C18—H18	0.9500
Cl4—C14	1.7407 (18)	C19—C20	1.418 (3)
C1—C21	1.512 (2)	C19—H19	0.9500
C1—C2	1.513 (2)	C20—C21	1.413 (3)
C2—C11	1.379 (2)	C1L—C2L^{i}	1.624 (9)
C2—C3	1.411 (2)	C1L—C2L	1.624 (9)
C3—C4	1.421 (3)	C1L—H1L1	0.9900
C3—C8	1.434 (3)	C1L—H1L2	0.9900
C4—C5	1.363 (3)	C2L—C3L	1.556 (8)
C4—H4	0.9500	C2L—H2L1	0.9900
C5—C6	1.404 (3)	C2L—H2L2	0.9900
C5—H5	0.9500	C3L—C4L	1.524 (6)
C6—C7	1.366 (3)	C3L—H3L1	0.9900
C6—H6	0.9500	C3L—H3L2	0.9900
C7—C8	1.414 (3)	C4L—H4L1	0.9900
C7—H7	0.9500	C4L—H4L2	0.9900
C8—C9	1.424 (3)	C1M—C1M^{i}	1.241 (18)
C9—C10	1.365 (3)	C1M—C2M^{i}	1.495 (10)
C10—C11	1.403 (2)	C1M—C2M	1.556 (8)
C10—H10	0.9500	C1M—H1M1	1.0003
C11—C12	1.469 (2)	C1M—H1M2	0.9900
C12—C21	1.380 (2)	C2M—C3M	1.539 (8)
C12—C13	1.405 (2)	C2M—H2M1	0.9900
C13—C14	1.364 (3)	C2M—H2M2	0.9900
C13—H13	0.9500	C3M—C4M	1.588 (10)
C14—C15	1.426 (3)	C3M—C4M^{i}	1.719 (13)
C15—C16	1.418 (3)	C3M—H3M1	0.9900
C15—C20	1.435 (2)	C3M—H3M2	0.9900
C16—C17	1.364 (3)	C4M—C4M^{i}	1.264 (19)
C16—H16	0.9500	C4M—H4M1	0.9900
C17—C18	1.403 (3)	C4M—H4M2	0.9900
C21—C1—C2	104.22 (14)	C20—C21—C1	129.87 (16)
C21—C1—Cl2	111.34 (12)	C2L^{i}—C1L—C2L	113.7 (7)
C2—C1—Cl2	111.54 (12)	C2L^{i}—C1L—H1L1	107.0
C21—C1—Cl1	111.22 (12)	C2L—C1L—H1L1	110.6
C2—C1—Cl1	111.32 (12)	C2L^{i}—C1L—H1L2	110.6
Cl2—C1—Cl1	107.26 (9)	C2L—C1L—H1L2	106.9
C11—C2—C3	121.66 (16)	H1L1—C1L—H1L2	107.9
C11—C2—C1	108.36 (15)	C3L—C2L—C1L	105.8 (6)

C3—C2—C1	129.98 (16)	C3L—C2L—H2L1	110.6
C2—C3—C4	124.26 (17)	C1L—C2L—H2L1	110.6
C2—C3—C8	117.35 (16)	C3L—C2L—H2L2	110.6
C4—C3—C8	118.39 (17)	C1L—C2L—H2L2	110.6
C5—C4—C3	120.80 (19)	H2L1—C2L—H2L2	108.7
C5—C4—H4	119.6	C4L—C3L—C2L	105.7 (5)
C3—C4—H4	119.6	C4L—C3L—H3L1	110.6
C4—C5—C6	120.7 (2)	C2L—C3L—H3L1	110.6
C4—C5—H5	119.7	C4L—C3L—H3L2	110.6
C6—C5—H5	119.7	C2L—C3L—H3L2	110.6
C7—C6—C5	120.4 (2)	H3L1—C3L—H3L2	108.7
C7—C6—H6	119.8	C3L—C4L—C3L^{i}	119.8 (6)
C5—C6—H6	119.8	C3L—C4L—H4L1	108.3
C6—C7—C8	120.9 (2)	C3L^{i}—C4L—H4L1	106.3
C6—C7—H7	119.5	C3L—C4L—H4L2	106.3
C8—C7—H7	119.5	C3L^{i}—C4L—H4L2	108.3
C7—C8—C9	122.67 (18)	H4L1—C4L—H4L2	107.2
C7—C8—C3	118.80 (17)	C1M^{i}—C1M—C2M	63.4 (6)
C9—C8—C3	118.53 (17)	C2M^{i}—C1M—C2M	108.2 (6)
C10—C9—C8	123.03 (17)	C1M^{i}—C1M—H1M1	67.8
C10—C9—Cl3	117.92 (14)	C2M^{i}—C1M—H1M1	102.7
C8—C9—Cl3	119.05 (14)	C2M—C1M—H1M1	104.7
C9—C10—C11	117.76 (17)	C1M^{i}—C1M—H1M2	176.1
C9—C10—H10	121.1	C2M^{i}—C1M—H1M2	114.7
C11—C10—H10	121.1	C2M—C1M—H1M2	116.2
C2—C11—C10	121.66 (17)	H1M1—C1M—H1M2	109.0
C2—C11—C12	109.59 (15)	H1L1^{i}—C1M—H1L2^{i}	82.1
C10—C11—C12	128.75 (16)	C1M^{i}—C2M—C3M	92.1 (6)
C21—C12—C13	121.45 (17)	C1M^{i}—C2M—C1M	47.9 (7)
C21—C12—C11	109.32 (15)	C3M—C2M—C1M	116.4 (5)
C13—C12—C11	129.22 (16)	C1M^{i}—C2M—H2M1	149.1
C14—C13—C12	117.96 (17)	C3M—C2M—H2M1	108.5
C14—C13—H13	121.0	C1M—C2M—H2M1	101.4
C12—C13—H13	121.0	C1M^{i}—C2M—H2M2	85.7
C13—C14—C15	123.09 (16)	C3M—C2M—H2M2	109.1
C13—C14—Cl4	117.32 (15)	C1M—C2M—H2M2	113.0
C15—C14—Cl4	119.58 (14)	H2M1—C2M—H2M2	107.8
C16—C15—C14	123.03 (17)	C2M—C3M—C4M	116.9 (7)
C16—C15—C20	118.65 (17)	C2M—C3M—C4M^{i}	92.1 (6)
C14—C15—C20	118.31 (16)	C4M—C3M—C4M^{i}	44.7 (7)
C17—C16—C15	120.81 (18)	C2M—C3M—H3M1	107.6
C17—C16—H16	119.6	C4M—C3M—H3M1	108.8
C15—C16—H16	119.6	C4M^{i}—C3M—H3M1	153.2
C16—C17—C18	120.50 (18)	C2M—C3M—H3M2	108.4
C16—C17—H17	119.8	C4M—C3M—H3M2	107.6
C18—C17—H17	119.8	C4M^{i}—C3M—H3M2	82.6
C19—C18—C17	120.85 (19)	H3M1—C3M—H3M2	107.2
C19—C18—H18	119.6	C4M^{i}—C4M—C3M	73.1 (8)

C17—C18—H18	119.6	C4M^{i}—C4M—H4M1	174.5
C18—C19—C20	120.42 (18)	C3M—C4M—H4M1	109.8
C18—C19—H19	119.8	C3M^{i}—C4M—H4M1	112.3
C20—C19—H19	119.8	C4M^{i}—C4M—H4M2	73.8
C21—C20—C19	123.69 (17)	C3M—C4M—H4M2	110.9
C21—C20—C15	117.56 (16)	C3M^{i}—C4M—H4M2	110.8
C19—C20—C15	118.76 (17)	H4M1—C4M—H4M2	108.8
C12—C21—C20	121.63 (16)	H4L1^{i}—C4M—H4L2^{i}	77.7
C12—C21—C1	108.50 (15)		

C21—C1—C2—C11	0.04 (18)	Cl4—C14—C15—C16	0.8 (2)
Cl2—C1—C2—C11	−120.21 (13)	C13—C14—C15—C20	0.3 (3)
Cl1—C1—C2—C11	120.02 (13)	Cl4—C14—C15—C20	−179.39 (12)
C21—C1—C2—C3	−179.87 (17)	C14—C15—C16—C17	−179.80 (17)
Cl2—C1—C2—C3	59.9 (2)	C20—C15—C16—C17	0.4 (3)
Cl1—C1—C2—C3	−59.9 (2)	C15—C16—C17—C18	−0.2 (3)
C11—C2—C3—C4	178.98 (17)	C16—C17—C18—C19	0.1 (3)
C1—C2—C3—C4	−1.1 (3)	C17—C18—C19—C20	−0.2 (3)
C11—C2—C3—C8	−0.7 (2)	C18—C19—C20—C21	−179.61 (17)
C1—C2—C3—C8	179.16 (16)	C18—C19—C20—C15	0.5 (3)
C2—C3—C4—C5	−179.32 (19)	C16—C15—C20—C21	179.52 (15)
C8—C3—C4—C5	0.4 (3)	C14—C15—C20—C21	−0.3 (2)
C3—C4—C5—C6	−0.9 (3)	C16—C15—C20—C19	−0.6 (2)
C4—C5—C6—C7	0.4 (4)	C14—C15—C20—C19	179.67 (16)
C5—C6—C7—C8	0.6 (4)	C13—C12—C21—C20	−0.2 (3)
C6—C7—C8—C9	178.47 (19)	C11—C12—C21—C20	−179.88 (15)
C6—C7—C8—C3	−1.1 (3)	C13—C12—C21—C1	179.72 (15)
C2—C3—C8—C7	−179.68 (16)	C11—C12—C21—C1	0.01 (19)
C4—C3—C8—C7	0.6 (3)	C19—C20—C21—C12	−179.72 (16)
C2—C3—C8—C9	0.7 (2)	C15—C20—C21—C12	0.2 (2)
C4—C3—C8—C9	−178.99 (16)	C19—C20—C21—C1	0.4 (3)
C7—C8—C9—C10	−179.69 (17)	C15—C20—C21—C1	−179.66 (16)
C3—C8—C9—C10	−0.1 (3)	C2—C1—C21—C12	−0.03 (18)
C7—C8—C9—Cl3	0.2 (2)	Cl2—C1—C21—C12	120.35 (13)
C3—C8—C9—Cl3	179.77 (13)	Cl1—C1—C21—C12	−120.08 (13)
C8—C9—C10—C11	−0.5 (3)	C2—C1—C21—C20	179.85 (17)
Cl3—C9—C10—C11	179.60 (12)	Cl2—C1—C21—C20	−59.8 (2)
C3—C2—C11—C10	0.1 (3)	Cl1—C1—C21—C20	59.8 (2)
C1—C2—C11—C10	−179.81 (15)	C2L^{i}—C1L—C2L—C3L	33.3 (4)
C3—C2—C11—C12	179.88 (15)	C1L—C2L—C3L—C4L	−66.8 (7)
C1—C2—C11—C12	−0.03 (19)	C2L—C3L—C4L—C3L^{i}	34.5 (4)
C9—C10—C11—C2	0.5 (3)	C2M^{i}—C1M—C2M—C1M^{i}	−53.6 (7)
C9—C10—C11—C12	−179.21 (16)	C1M^{i}—C1M—C2M—C3M	66.9 (6)
C2—C11—C12—C21	0.02 (19)	C2M^{i}—C1M—C2M—C3M	13.3 (9)
C10—C11—C12—C21	179.78 (16)	C1M^{i}—C2M—C3M—C4M	62.5 (9)
C2—C11—C12—C13	−179.67 (16)	C1M—C2M—C3M—C4M	19.4 (11)
C10—C11—C12—C13	0.1 (3)	C1M^{i}—C2M—C3M—C4M^{i}	101.3 (6)
C21—C12—C13—C14	0.2 (2)	C1M—C2M—C3M—C4M^{i}	58.2 (8)

C11—C12—C13—C14	179.83 (16)	C2M—C3M—C4M—C4M^{i}	62.9 (8)
C12—C13—C14—C15	−0.2 (3)	C2M—C3M—C4M—C3M^{i}	8.3 (10)
C12—C13—C14—Cl4	179.44 (12)	C4M^{i}—C3M—C4M—C3M^{i}	−54.6 (6)
C13—C14—C15—C16	−179.47 (17)		

Symmetry code: (i) $-x+1, y, -z+3/2$.