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Dichlorido[(1,2,3,3a,8b-η)-2,4-dimethylcyclopenta[*b*]indolyl](η^5-pentamethylcyclopentadienyl)zirconium(IV)

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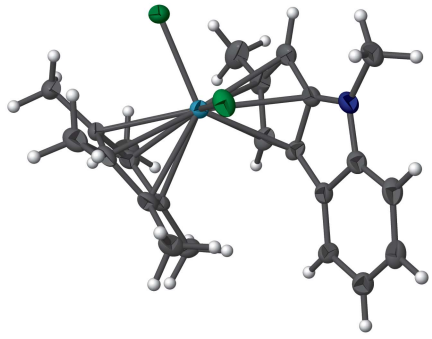
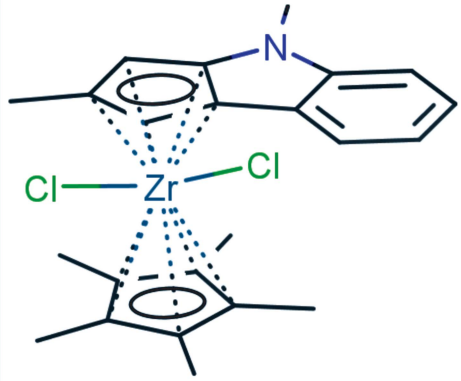
Structural data: full structural data are available from iucrdata.iucr.org

In the structure of the title compound, $[Zn(C_{13}H_{12}N)(C_{10}H_{15})Cl_2]$, the dihedral angle between the planes of rings coordinating to Zr is 51.6 (2)°. The Cl—Zr—Cl angle is 97.52 (4)°. The crystal structure is stabilized by H···Cl and C—H···π interactions.

3D view



Chemical scheme



Structure description

A view of the title molecule is given on Fig. 1. The chlorine ligands form intramolecular C—H···Cl hydrogen bonds (Table 1). The crystal structure is stabilized by C—H···π interactions.

Synthesis and crystallization

The title complex was obtained *via* treatment of $(C_5Me_5)ZrCl_3$ (Ryabov *et al.*, 2002) with the lithium salt of 2,4-dimethyl-1,4-dihydrocyclopenta[*b*]indole (van Baar *et al.*, 2003) in THF followed by crystallization of the crude product from toluene. Single crystals suitable for X-ray crystal structure analysis were grown from a toluene–hexanes solution at −30°C.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Table 1
Hydrogen-bond geometry (Å, °).

*Cg*4 is the centroid of the C6–C11 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···Cl1^{i}	1.0	2.72	3.684 (5)	162
C10—H10···Cl2^{ii}	0.95	2.77	3.699 (5)	166
C22—H22*C*···Cl1	0.98	2.67	3.158 (5)	111
C22—H22*A*···*Cg*4^{iii}	0.98	2.57	3.487 (5)	156

Symmetry codes: (i) $-x+1, -y+1, z$; (ii) $y, -x+1, -z+1$; (iii) $x+\frac{1}{2}, -y+\frac{1}{2}, -z+\frac{3}{2}$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$[Zn(C_{13}H_{12}N)(C_{10}H_{15})Cl_2]$
M_r	479.57
Crystal system, space group	Tetragonal, $P\bar{4}2_1c$
Temperature (K)	100
a, c (Å)	15.9363 (8), 16.8801 (8)
V (Å^3)	4287.0 (5)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.77
Crystal size (mm)	0.25 × 0.2 × 0.2
Data collection	
Diffractometer	Bruker *SMART* CCD area detector
Absorption correction	Multi-scan (*SADABS*; Bruker, 2001)
T_{min}, T_{max}	0.759, 0.925
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	86055, 5707, 5264
R_{int}	0.051
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.682
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.042, 0.096, 1.11
No. of reflections	5707
No. of parameters	251
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.25, −0.38
Absolute structure	Flack x determined using 2285 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	−0.056 (13)

Computer programs: *SMART* and *SAINT* (Bruker, 2001), *SHELXS* (Sheldrick, 2008), *SHELXL* (Sheldrick, 2015) and *OLEX2* (Dolomanov *et al.*, 2009).

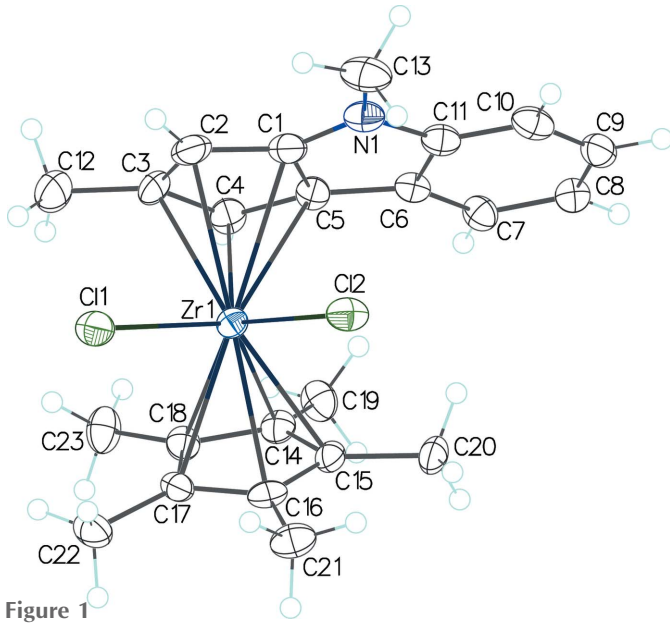


Figure 1
The structure of the title compound with displacement ellipsoids at the 50% probability level.

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x161408 [doi:10.1107/S2414314616014085]

Dichlorido[(1,2,3,3a,8b-η)-2,4-dimethylcyclopenta[*b*]indolyl)(η^5-pentamethylcyclopentadienyl)zirconium(IV)

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Crystal data

$[Zn(C_{13}H_{12}N)(C_{10}H_{15})Cl_2]$
M_r = 479.57
Tetragonal, $P\bar{4}2_1c$
a = 15.9363 (8) Å
c = 16.8801 (8) Å
V = 4287.0 (5) Å^3
Z = 8
$F(000)$ = 1968
D_x = 1.486 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1024 reflections
θ = 2.8–28.9°
μ = 0.77 mm^{-1}
T = 100 K
Prism, yellow
0.25 × 0.2 × 0.2 mm

Data collection

Bruker SMART CCD area detector diffractometer
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2001)
T_{min} = 0.759, T_{max} = 0.925
86055 measured reflections
5707 independent reflections
5264 reflections with $I > 2\sigma(I)$
R_{int} = 0.051
θ_{max} = 29.0°, θ_{min} = 1.8°
h = −21→21
k = −21→21
l = −23→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.096
S = 1.11
5707 reflections
251 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.040P)^2 + 7.P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 1.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}
Absolute structure: Flack x determined using 2285 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: −0.056 (13)

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Zr1	0.65206 (2)	0.33633 (2)	0.77455 (2)	0.02065 (10)
Cl1	0.64847 (7)	0.47013 (7)	0.84549 (7)	0.0319 (2)
Cl2	0.65603 (7)	0.39342 (7)	0.64001 (7)	0.0297 (2)
N1	0.4764 (2)	0.3176 (2)	0.6413 (3)	0.0273 (8)
C1	0.4985 (3)	0.3110 (3)	0.7191 (3)	0.0279 (9)
C2	0.4924 (3)	0.3562 (3)	0.7901 (3)	0.0338 (11)
H2	0.4627	0.4110	0.7973	0.041*
C3	0.5219 (3)	0.3034 (4)	0.8516 (3)	0.0362 (12)
C4	0.5521 (3)	0.2274 (3)	0.8176 (3)	0.0279 (10)
H4	0.5667	0.1752	0.8474	0.033*
C5	0.5391 (3)	0.2331 (3)	0.7334 (3)	0.0238 (9)
C6	0.5416 (3)	0.1902 (3)	0.6576 (3)	0.0242 (9)
C7	0.5689 (3)	0.1122 (3)	0.6303 (3)	0.0263 (9)
H7	0.5931	0.0730	0.6660	0.032*
C8	0.5604 (3)	0.0922 (3)	0.5505 (3)	0.0291 (10)
H8	0.5798	0.0394	0.5318	0.035*
C9	0.5237 (3)	0.1489 (4)	0.4974 (3)	0.0310 (10)
H9	0.5183	0.1339	0.4432	0.037*
C10	0.4951 (3)	0.2267 (3)	0.5229 (3)	0.0298 (10)
H10	0.4710	0.2655	0.4867	0.036*
C11	0.5027 (3)	0.2461 (3)	0.6024 (3)	0.0258 (9)
C12	0.5167 (4)	0.3213 (4)	0.9390 (3)	0.0475 (15)
H12A	0.4582	0.3169	0.9565	0.071*
H12B	0.5375	0.3781	0.9495	0.071*
H12C	0.5510	0.2806	0.9680	0.071*
C13	0.4404 (3)	0.3916 (3)	0.6035 (4)	0.0394 (13)
H13A	0.4255	0.4330	0.6441	0.059*
H13B	0.3901	0.3755	0.5739	0.059*
H13C	0.4816	0.4161	0.5671	0.059*
C14	0.7468 (3)	0.2111 (3)	0.7950 (3)	0.0245 (9)
C15	0.7796 (3)	0.2544 (3)	0.7291 (3)	0.0240 (8)
C16	0.8101 (3)	0.3336 (3)	0.7566 (3)	0.0265 (9)
C17	0.7955 (3)	0.3379 (3)	0.8390 (3)	0.0227 (8)
C18	0.7547 (3)	0.2631 (3)	0.8630 (3)	0.0247 (9)
C19	0.7246 (3)	0.1194 (3)	0.7945 (3)	0.0313 (10)
H19B	0.7736	0.0866	0.7780	0.047*
H19C	0.6784	0.1098	0.7572	0.047*
H19A	0.7073	0.1021	0.8477	0.047*
C20	0.7866 (3)	0.2202 (3)	0.6466 (3)	0.0292 (10)
H20A	0.8277	0.1745	0.6458	0.044*
H20B	0.8048	0.2648	0.6106	0.044*
H20C	0.7318	0.1988	0.6295	0.044*
C21	0.8562 (3)	0.3978 (3)	0.7079 (3)	0.0327 (11)
H21B	0.9166	0.3932	0.7179	0.049*
H21C	0.8369	0.4541	0.7224	0.049*

H21A	0.8451	0.3879	0.6516	0.049*
C22	0.8289 (3)	0.4052 (3)	0.8927 (3)	0.0335 (11)
H22A	0.8902	0.4010	0.8955	0.050*
H22B	0.8052	0.3981	0.9459	0.050*
H22C	0.8132	0.4604	0.8719	0.050*
C23	0.7349 (4)	0.2407 (3)	0.9467 (3)	0.0341 (11)
H23A	0.7870	0.2283	0.9752	0.051*
H23B	0.6983	0.1913	0.9476	0.051*
H23C	0.7063	0.2879	0.9723	0.051*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zr1	0.02017 (18)	0.01997 (18)	0.02183 (17)	0.00016 (14)	0.00149 (16)	0.00073 (16)
Cl1	0.0272 (5)	0.0237 (5)	0.0448 (6)	0.0029 (4)	−0.0035 (5)	−0.0081 (4)
Cl2	0.0245 (5)	0.0343 (5)	0.0303 (5)	−0.0008 (5)	−0.0015 (5)	0.0120 (4)
N1	0.0210 (17)	0.0251 (18)	0.036 (2)	0.0016 (14)	−0.0022 (16)	0.0037 (16)
C1	0.0195 (19)	0.026 (2)	0.038 (3)	0.0002 (15)	−0.001 (2)	−0.003 (2)
C2	0.0191 (19)	0.036 (3)	0.046 (3)	−0.0020 (18)	0.0064 (18)	−0.009 (2)
C3	0.025 (2)	0.055 (3)	0.028 (2)	−0.009 (2)	0.012 (2)	−0.012 (2)
C4	0.029 (2)	0.029 (2)	0.026 (2)	−0.0101 (19)	0.0053 (18)	0.0029 (19)
C5	0.025 (2)	0.0193 (18)	0.027 (2)	−0.0078 (16)	0.0036 (18)	−0.0006 (17)
C6	0.020 (2)	0.025 (2)	0.027 (2)	−0.0019 (16)	−0.0019 (17)	0.0013 (17)
C7	0.028 (2)	0.023 (2)	0.028 (2)	−0.0005 (18)	−0.0034 (18)	−0.0005 (18)
C8	0.026 (2)	0.033 (2)	0.029 (2)	−0.0013 (18)	0.0008 (18)	−0.0036 (19)
C9	0.026 (2)	0.042 (3)	0.025 (2)	−0.008 (2)	−0.0008 (17)	0.000 (2)
C10	0.026 (2)	0.032 (2)	0.032 (2)	−0.0043 (18)	−0.0061 (19)	0.009 (2)
C11	0.022 (2)	0.025 (2)	0.031 (2)	−0.0026 (17)	−0.0013 (18)	0.0053 (18)
C12	0.039 (3)	0.065 (4)	0.038 (3)	−0.014 (3)	0.013 (2)	−0.014 (3)
C13	0.029 (3)	0.029 (3)	0.060 (4)	0.004 (2)	−0.011 (3)	0.005 (2)
C14	0.025 (2)	0.022 (2)	0.027 (2)	0.0073 (17)	−0.0012 (17)	0.0012 (17)
C15	0.0222 (19)	0.030 (2)	0.020 (2)	0.0079 (16)	−0.0002 (18)	−0.0013 (19)
C16	0.0164 (17)	0.029 (2)	0.034 (2)	0.0052 (17)	0.0001 (16)	0.0033 (19)
C17	0.0199 (18)	0.021 (2)	0.028 (2)	0.0010 (16)	−0.0052 (15)	−0.0031 (17)
C18	0.027 (2)	0.025 (2)	0.023 (2)	0.0048 (17)	−0.0022 (17)	0.0015 (17)
C19	0.038 (3)	0.023 (2)	0.033 (3)	0.0018 (19)	−0.004 (2)	−0.0025 (18)
C20	0.032 (2)	0.037 (3)	0.018 (2)	0.010 (2)	0.0025 (18)	−0.0015 (19)
C21	0.026 (2)	0.028 (2)	0.045 (3)	0.0010 (18)	0.004 (2)	0.0073 (19)
C22	0.029 (2)	0.028 (2)	0.043 (3)	0.0016 (19)	−0.009 (2)	−0.009 (2)
C23	0.049 (3)	0.031 (2)	0.023 (2)	0.007 (2)	0.002 (2)	0.0031 (19)

Geometric parameters (Å, º)

Zr1—Cl1	2.4463 (12)	C3—C12	1.505 (7)
Zr1—Cl2	2.4473 (12)	C4—C5	1.439 (6)
Zr1—C1	2.651 (4)	C5—C6	1.451 (7)
Zr1—C2	2.578 (5)	C6—C7	1.394 (6)
Zr1—C3	2.504 (5)	C6—C11	1.430 (6)

Zr1—C4	2.466 (5)	C7—C8	1.391 (7)
Zr1—C5	2.535 (4)	C8—C9	1.401 (7)
Zr1—C14	2.525 (4)	C9—C10	1.389 (8)
Zr1—C15	2.534 (4)	C10—C11	1.383 (7)
Zr1—C16	2.538 (4)	C14—C15	1.409 (7)
Zr1—C17	2.532 (4)	C14—C18	1.421 (7)
Zr1—C18	2.504 (4)	C14—C19	1.504 (6)
N1—C1	1.364 (7)	C15—C16	1.429 (7)
N1—C11	1.381 (6)	C15—C20	1.501 (6)
N1—C13	1.459 (6)	C16—C17	1.413 (6)
C1—C2	1.402 (7)	C16—C21	1.504 (6)
C1—C5	1.420 (6)	C17—C18	1.418 (6)
C2—C3	1.418 (8)	C17—C22	1.501 (6)
C3—C4	1.424 (7)	C18—C23	1.491 (7)
Cl1—Zr1—Cl2	97.52 (4)	C1—N1—C13	125.8 (4)
Cl1—Zr1—C1	106.50 (11)	C11—N1—C13	125.4 (4)
Cl1—Zr1—C2	79.62 (12)	N1—C1—Zr1	124.5 (3)
Cl1—Zr1—C3	84.79 (13)	N1—C1—C2	139.9 (5)
Cl1—Zr1—C4	117.03 (12)	N1—C1—C5	110.4 (4)
Cl1—Zr1—C5	133.10 (11)	C2—C1—Zr1	71.6 (3)
Cl1—Zr1—C14	129.47 (11)	C2—C1—C5	109.7 (5)
Cl1—Zr1—C15	128.01 (11)	C5—C1—Zr1	69.7 (2)
Cl1—Zr1—C16	95.56 (11)	C1—C2—Zr1	77.3 (3)
Cl1—Zr1—C17	78.57 (10)	C1—C2—C3	107.3 (4)
Cl1—Zr1—C18	97.46 (11)	C3—C2—Zr1	70.9 (3)
Cl2—Zr1—C1	75.69 (12)	C2—C3—Zr1	76.7 (3)
Cl2—Zr1—C2	94.25 (13)	C2—C3—C4	108.8 (4)
Cl2—Zr1—C3	125.56 (14)	C2—C3—C12	125.9 (5)
Cl2—Zr1—C4	123.48 (12)	C4—C3—Zr1	71.9 (3)
Cl2—Zr1—C5	90.29 (12)	C4—C3—C12	125.2 (6)
Cl2—Zr1—C14	113.90 (11)	C12—C3—Zr1	121.0 (4)
Cl2—Zr1—C15	83.69 (11)	C3—C4—Zr1	74.8 (3)
Cl2—Zr1—C16	82.53 (11)	C3—C4—C5	107.2 (4)
Cl2—Zr1—C17	111.83 (11)	C5—C4—Zr1	75.9 (3)
Cl2—Zr1—C18	135.23 (11)	C1—C5—Zr1	78.7 (3)
C2—Zr1—C1	31.06 (15)	C1—C5—C4	106.7 (4)
C3—Zr1—C1	52.18 (17)	C1—C5—C6	106.0 (4)
C3—Zr1—C2	32.36 (18)	C4—C5—Zr1	70.7 (3)
C3—Zr1—C5	54.44 (16)	C4—C5—C6	146.8 (5)
C3—Zr1—C14	104.96 (18)	C6—C5—Zr1	121.9 (3)
C3—Zr1—C15	135.51 (18)	C7—C6—C5	136.0 (4)
C3—Zr1—C16	151.72 (17)	C7—C6—C11	118.4 (4)
C3—Zr1—C17	121.77 (16)	C11—C6—C5	105.6 (4)
C3—Zr1—C18	97.67 (18)	C8—C7—C6	119.6 (5)
C4—Zr1—C1	53.15 (16)	C7—C8—C9	120.8 (5)
C4—Zr1—C2	54.47 (17)	C10—C9—C8	121.0 (5)
C4—Zr1—C3	33.29 (17)	C11—C10—C9	118.1 (5)

C4—Zr1—C5	33.41 (14)	N1—C11—C6	109.6 (4)
C4—Zr1—C14	77.85 (16)	N1—C11—C10	128.3 (4)
C4—Zr1—C15	104.16 (16)	C10—C11—C6	122.1 (5)
C4—Zr1—C16	131.63 (16)	C15—C14—Zr1	74.2 (2)
C4—Zr1—C17	117.64 (16)	C15—C14—C18	108.6 (4)
C4—Zr1—C18	85.31 (16)	C15—C14—C19	123.9 (4)
C5—Zr1—C1	31.69 (14)	C18—C14—Zr1	72.8 (2)
C5—Zr1—C2	53.63 (15)	C18—C14—C19	126.3 (4)
C5—Zr1—C16	131.34 (15)	C19—C14—Zr1	128.8 (3)
C14—Zr1—C1	118.66 (15)	C14—C15—Zr1	73.5 (3)
C14—Zr1—C2	132.32 (17)	C14—C15—C16	107.6 (4)
C14—Zr1—C5	87.08 (15)	C14—C15—C20	125.6 (4)
C14—Zr1—C15	32.35 (15)	C16—C15—Zr1	73.8 (2)
C14—Zr1—C16	53.80 (15)	C16—C15—C20	126.6 (4)
C14—Zr1—C17	53.82 (15)	C20—C15—Zr1	121.9 (3)
C15—Zr1—C1	123.72 (15)	C15—C16—Zr1	73.5 (2)
C15—Zr1—C2	152.35 (16)	C15—C16—C21	126.1 (4)
C15—Zr1—C5	98.75 (14)	C17—C16—Zr1	73.6 (2)
C15—Zr1—C16	32.73 (15)	C17—C16—C15	107.9 (4)
C16—Zr1—C1	150.64 (16)	C17—C16—C21	125.8 (4)
C16—Zr1—C2	173.85 (16)	C21—C16—Zr1	122.5 (3)
C17—Zr1—C1	170.66 (15)	C16—C17—Zr1	74.0 (2)
C17—Zr1—C2	147.81 (15)	C16—C17—C18	108.4 (4)
C17—Zr1—C5	139.93 (14)	C16—C17—C22	124.9 (4)
C17—Zr1—C15	53.93 (14)	C18—C17—Zr1	72.6 (2)
C17—Zr1—C16	32.36 (14)	C18—C17—C22	126.2 (4)
C18—Zr1—C1	137.96 (15)	C22—C17—Zr1	125.9 (3)
C18—Zr1—C2	129.92 (17)	C14—C18—Zr1	74.4 (3)
C18—Zr1—C5	108.95 (15)	C14—C18—C23	127.4 (4)
C18—Zr1—C14	32.82 (15)	C17—C18—Zr1	74.7 (2)
C18—Zr1—C15	54.29 (15)	C17—C18—C14	107.5 (4)
C18—Zr1—C16	54.17 (15)	C17—C18—C23	124.7 (4)
C18—Zr1—C17	32.70 (15)	C23—C18—Zr1	122.5 (3)
C1—N1—C11	108.4 (4)		

Hydrogen-bond geometry (Å, º)

Cg4 is the centroid of the C6–C11 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···Cl1^{i}	1.0	2.72	3.684 (5)	162
C10—H10···Cl2^{ii}	0.95	2.77	3.699 (5)	166
C22—H22*C*···Cl1	0.98	2.67	3.158 (5)	111
C22—H22*A*···*Cg*4^{iii}	0.98	2.57	3.487 (5)	156

Symmetry codes: (i) −*x*+1, −*y*+1, *z*; (ii) *y*, −*x*+1, −*z*+1; (iii) *x*+1/2, −*y*+1/2, −*z*+3/2.