Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Dichloridobis[3-methoxymethyl-4-phenyl-5-(2-pyridyl)-4*H*-1,2,4-triazole-κ^2N^1,N^5]chromium(III) chloride

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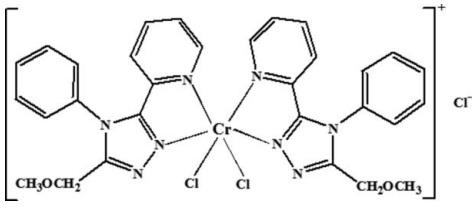
Received 19 July 2011; accepted 25 September 2011

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.005 Å; disorder in solvent or counterion; R factor = 0.039; wR factor = 0.119; data-to-parameter ratio = 13.1.

In the title complex, $[CrCl_2(C_{15}H_{14}N_4O)_2]Cl$, the Cr^{III} atom is located on a twofold rotation axis and is coordinated by two N,N'-bidentate triazole derivatives and two chloride ions in a distorted octahedral $CrN_2N'_2Cl_2$ geometry. One of the two independent Cl^- counter-anions sits on a special position (site symmetry $\overline{3}$.) and is fully occupied, whereas the other is disordered around a twofold rotation axis over two positions in a 2:3 ratio.

Related literature

For general background to the coordination chemistry of 1,2,4-triazole derivatives, see: Koningsbruggen *et al.* (1997); Garcia *et al.* (1999); Klingele & Brooker (2003); Matsukizono *et al.* (2008); Suksrichavalit *et al.* (2009); Rubio *et al.* (2011). For their biological activity, see: Tozkoparan *et al.* (2000); Grenman *et al.* (2003); Alagarsamy *et al.* (2008); Isloor *et al.* (2009).



Experimental

Crystal data

$[CrCl_2(C_{15}H_{14}N_4O)_2]Cl$
M_r = 690.95
Hexagonal, $R\overline{3}c$
a = 20.8852 (12) Å
c = 37.620 (4) Å
V = 14211.1 (19) Å^3
Z = 18
Mo $K\alpha$ radiation
μ = 0.66 mm^{-1}
T = 296 K
0.17 × 0.13 × 0.12 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.896, T_{max} = 0.925
32255 measured reflections
2785 independent reflections
2243 reflections with $I > 2\sigma(I)$
R_{int} = 0.052

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.119
S = 1.10
2785 reflections
213 parameters
18 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.68 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Table 1
Selected bond lengths (Å).

Cr1—N1	2.040 (2)	Cr1—Cl2^{i}	2.2746 (7)
Cr1—N4	2.0949 (19)		

Symmetry code: (i) $y+\frac{1}{3}, x-\frac{1}{3}, -z+\frac{1}{6}$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

We are grateful to Jingye Pharmochemical Pilot Plant for financial assistance though project 8507040052.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2515).

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supporting information

Acta Cryst. (2011). E**67**, m1492 [doi:10.1107/S1600536811039328]

Dichloridobis[3-methoxymethyl-4-phenyl-5-(2-pyridyl)-4*H*-1,2,4-triazole-κ^2N^1,N^5]chromium(III) chloride

Xiaofei Jin, Zuoxiang Wang and Shouping Cao

S1. Comment

As the 1,2,4-triazole ring posesses strong electron donors, the coordination chemistry of 1,2,4-triazoles employed as ligand is widely studied (Koningsbruggen *et al.*, 1997; Garcia *et al.*, 1999; Klingele & Brooker, 2003; Matsukizono *et al.*, 2008; Suksrichavalit *et al.*, 2009; Rubio *et al.*, 2011). Moreover, some 1,2,4-triazole compounds show biological activities (Tozkoparan *et al.*, 2000; Grenman *et al.*, 2003; Alagarsamy *et al.*, 2008; Isloor *et al.*, 2009). We report here the crystal structure analysis of the title compound, $[Cr(C_{15}H_{14}N_4O)_2Cl_2]Cl$.

In the title compouund, the chromium(III) atom is coordinated by two chelating 3-(methoxymethyl)-4-phenyl-5-(2-pyridyl)-4*H*-1,2,4- triazole ligands and two chloride anions in a distorted octahedral geometry with a $CrN_2N'_2Cl_2$ coordination set. The central Cr^{III} atom is located on a special position (site symmetry .2). The dihedral angle between the 1,2,4-triazole ring and the phenyl ring is 83.28 (16) °. One of the two non-coordinating Cl^- counter is located on a special position (site symmetry $\bar{3}$.) whereas the other shows disorder around a twofold rotation axis.

S2. Experimental

To a warm solution of 0.798 g of 3-(methoxymethyl)-4-phenyl-5-(2-pyridyl)-4*H*-1,2,4- triazole (3 mmol) in 20 ml ethanol, 0.399 g of $CrCl_3·6H_2O$ (1.5 mmol) were added. The filtrate was left to stand at room temperature for several days. The dark purple product was collected, and single crystals suitable for X-ray diffraction were selected.

S3. Refinement

Positional parameters of all the H atoms were calculated geometrically and were allowed to ride on the C atoms to which they are bonded, riding with C—H = 0.93 Å (aromatic), 0.96 Å (methyl) and 0.97 Å (methylene), with $U_{iso}(H)$ = 1.2 or 1.5 times $U_{eq}(C)$. Atom CL3 is positionally and occupationally disordered around a twofold rotation axis. One part (Cl3A, site occupation factor 0.168 (10)) sits on the twofold axis whereas the other part (Cl3) is 1.014 (19) Å away from this atom with a s.o.f. of 0.249 (5). To achieve an electroneutral compound, the overall occuipancy of the two Cl3 atoms was restrained to $0.\bar{3}$.

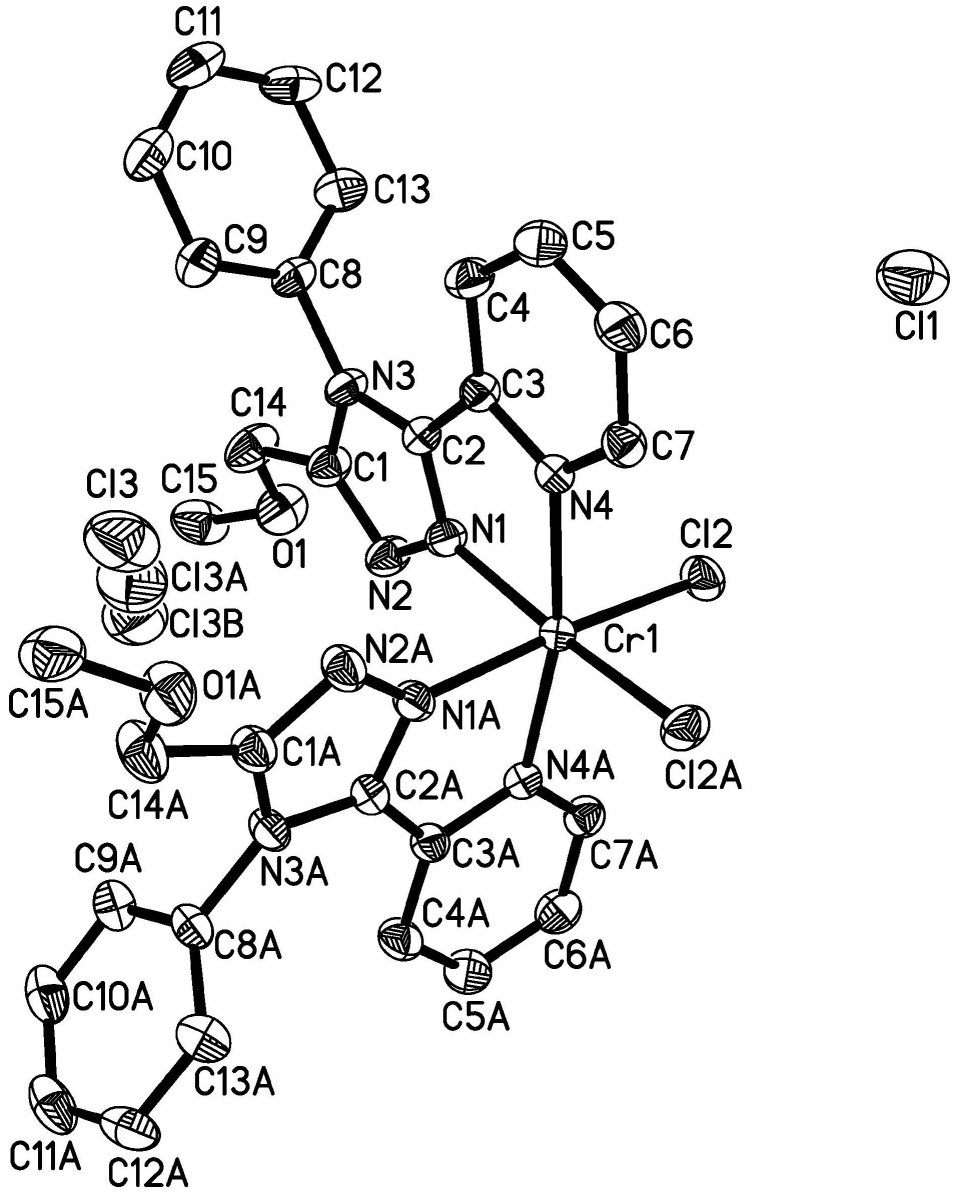

Figure 1

The molecular structure of the title compound with the atomic labelling. Displacement ellipsoids are shown at 30% probability level. [Symmetry code: A) *y* + 1/3, *x* - 1/3, -*z* + 1/6.]

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Crystal data

$[CrCl_2(C_{15}H_{14}N_4O)_2]Cl$
M_r = 690.95
Hexagonal, $R\bar{3}c$
Hall symbol: -R 3 2"c
a = 20.8852 (12) Å
c = 37.620 (4) Å
V = 14211.1 (19) Å^3
Z = 18
F(000) = 6390
D_x = 1.453 Mg m^{-3}
Mo *Kα* radiation, λ = 0.71073 Å
Cell parameters from 9999 reflections
θ = 2.4–21.0°
μ = 0.66 mm^{-1}
T = 296 K
Octahedral, purple
0.17 × 0.13 × 0.12 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.896, T_{max} = 0.925
32255 measured reflections
2785 independent reflections
2243 reflections with $I > 2\sigma(I)$
R_{int} = 0.052
θ_{max} = 25.0°, θ_{min} = 2.0°
h = −24→24
k = −24→24
l = −44→42

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.119
S = 1.10
2785 reflections
213 parameters
18 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.072P)^2 + 5.702P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.68 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Special details

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

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.91830 (15)	0.47477 (15)	0.03037 (7)	0.0461 (6)	
Cl1	1.0000	1.0000	0.0000	0.0778 (6)	
Cl2	1.12055 (4)	0.71828 (4)	0.055158 (18)	0.0496 (2)	
Cl3	0.7438 (5)	0.4643 (3)	0.07589 (15)	0.109 (2)	0.249 (5)
Cl3A	0.7727 (7)	0.4394 (7)	0.0833	0.113 (5)	0.168 (10)
Cr1	1.01043 (2)	0.67710 (2)	0.0833	0.03157 (19)	
N1	0.96416 (11)	0.58203 (11)	0.05415 (5)	0.0386 (5)	
O1	0.94481 (13)	0.37801 (11)	0.03630 (6)	0.0668 (6)	
C2	0.92596 (13)	0.58194 (13)	0.02635 (6)	0.0363 (6)	
N2	0.96048 (13)	0.51506 (11)	0.05722 (6)	0.0467 (6)	
C3	0.92437 (13)	0.64891 (13)	0.01735 (6)	0.0369 (6)	
N3	0.89587 (12)	0.51482 (11)	0.01048 (5)	0.0408 (5)	
C4	0.89089 (15)	0.65933 (16)	−0.01212 (7)	0.0484 (7)	
H4	0.8639	0.6207	−0.0278	0.058*	
N4	0.96271 (10)	0.70375 (10)	0.04105 (5)	0.0345 (5)	
C5	0.89817 (16)	0.72790 (16)	−0.01779 (8)	0.0533 (7)	

H5	0.8766	0.7362	−0.0376	0.064*
C6	0.93721 (16)	0.78347 (16)	0.00588 (8)	0.0504 (7)
H6	0.9428	0.8301	0.0023	0.060*
C7	0.96843 (14)	0.76974 (14)	0.03532 (7)	0.0421 (6)
H7	0.9942	0.8076	0.0516	0.051*
C8	0.84783 (14)	0.49011 (14)	−0.02045 (7)	0.0413 (6)
C9	0.77403 (16)	0.46556 (15)	−0.01567 (8)	0.0521 (7)
H9	0.7551	0.4630	0.0070	0.062*
C10	0.72852 (18)	0.44481 (16)	−0.04488 (9)	0.0598 (8)
H10	0.6785	0.4287	−0.0421	0.072*
C11	0.7567 (2)	0.44776 (17)	−0.07810 (9)	0.0631 (9)
H11	0.7258	0.4332	−0.0978	0.076*
C12	0.8305 (2)	0.47214 (18)	−0.08231 (8)	0.0617 (9)
H12	0.8491	0.4741	−0.1050	0.074*
C13	0.87821 (17)	0.49400 (17)	−0.05344 (7)	0.0538 (7)
H13	0.9283	0.5105	−0.0562	0.065*
C14	0.8939 (2)	0.39585 (17)	0.02282 (9)	0.0651 (9)
H14A	0.8459	0.3644	0.0336	0.078*
H14B	0.8890	0.3874	−0.0026	0.078*
C15	0.9149 (2)	0.30032 (18)	0.04022 (9)	0.0744 (10)
H15A	0.9495	0.2911	0.0527	0.112*
H15B	0.9054	0.2776	0.0172	0.112*
H15C	0.8695	0.2799	0.0534	0.112*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0529 (16)	0.0402 (14)	0.0461 (15)	0.0241 (13)	−0.0090 (12)	−0.0049 (12)
Cl1	0.0920 (10)	0.0920 (10)	0.0494 (11)	0.0460 (5)	0.000	0.000
Cl2	0.0408 (4)	0.0593 (4)	0.0460 (4)	0.0230 (3)	0.0107 (3)	0.0075 (3)
Cl3	0.162 (5)	0.081 (3)	0.074 (3)	0.053 (3)	0.008 (3)	−0.008 (2)
Cl3A	0.121 (5)	0.121 (5)	0.089 (6)	0.053 (5)	0.013 (4)	−0.013 (4)
Cr1	0.0317 (2)	0.0317 (2)	0.0285 (3)	0.0137 (2)	−0.00026 (11)	0.00026 (11)
N1	0.0433 (12)	0.0364 (11)	0.0357 (11)	0.0195 (10)	−0.0063 (9)	−0.0019 (9)
O1	0.0815 (15)	0.0456 (12)	0.0795 (15)	0.0364 (11)	−0.0167 (12)	−0.0035 (10)
C2	0.0354 (13)	0.0355 (13)	0.0348 (13)	0.0153 (11)	−0.0035 (10)	−0.0025 (10)
N2	0.0570 (14)	0.0368 (12)	0.0464 (13)	0.0235 (11)	−0.0147 (11)	−0.0052 (10)
C3	0.0333 (13)	0.0402 (14)	0.0353 (13)	0.0170 (11)	−0.0017 (10)	0.0000 (11)
N3	0.0444 (12)	0.0385 (12)	0.0380 (12)	0.0195 (10)	−0.0119 (9)	−0.0077 (9)
C4	0.0507 (16)	0.0506 (16)	0.0436 (15)	0.0250 (13)	−0.0131 (12)	0.0004 (12)
N4	0.0347 (10)	0.0347 (11)	0.0320 (11)	0.0156 (9)	0.0018 (8)	0.0025 (8)
C5	0.0549 (17)	0.0564 (18)	0.0526 (17)	0.0309 (15)	−0.0099 (14)	0.0084 (14)
C6	0.0580 (17)	0.0441 (15)	0.0540 (18)	0.0293 (14)	0.0019 (14)	0.0102 (13)
C7	0.0449 (14)	0.0373 (14)	0.0443 (15)	0.0206 (12)	0.0012 (12)	0.0021 (11)
C8	0.0453 (15)	0.0389 (14)	0.0414 (15)	0.0222 (12)	−0.0119 (12)	−0.0096 (11)
C9	0.0483 (16)	0.0450 (16)	0.0565 (18)	0.0184 (13)	−0.0061 (13)	−0.0083 (13)
C10	0.0513 (17)	0.0476 (17)	0.077 (2)	0.0219 (14)	−0.0214 (16)	−0.0115 (15)
C11	0.076 (2)	0.0477 (17)	0.072 (2)	0.0359 (17)	−0.0394 (18)	−0.0169 (15)

C12	0.096 (3)	0.066 (2)	0.0365 (16)	0.0504 (19)	−0.0136 (16)	−0.0093 (13)
C13	0.0610 (18)	0.0626 (19)	0.0467 (17)	0.0375 (16)	−0.0064 (14)	−0.0079 (14)
C14	0.079 (2)	0.0469 (17)	0.074 (2)	0.0345 (17)	−0.0306 (18)	−0.0187 (15)
C15	0.117 (3)	0.058 (2)	0.056 (2)	0.049 (2)	−0.001 (2)	0.0027 (15)

Geometric parameters (Å, º)

C1—N2	1.328 (3)	C5—C6	1.363 (4)
C1—N3	1.368 (3)	C5—H5	0.9300
C1—C14	1.489 (4)	C6—C7	1.386 (4)
Cl2—Cr1	2.2747 (7)	C6—H6	0.9300
Cl3—Cl3^{i}	2.028 (19)	C7—H7	0.9300
Cr1—N1^{i}	2.040 (2)	C8—C9	1.371 (4)
Cr1—N1	2.040 (2)	C8—C13	1.378 (4)
Cr1—N4^{i}	2.0948 (19)	C9—C10	1.374 (4)
Cr1—N4	2.0949 (19)	C9—H9	0.9300
Cr1—Cl2^{i}	2.2746 (7)	C10—C11	1.370 (4)
N1—C2	1.315 (3)	C10—H10	0.9300
N1—N2	1.367 (3)	C11—C12	1.369 (5)
O1—C14	1.387 (4)	C11—H11	0.9300
O1—C15	1.425 (4)	C12—C13	1.387 (4)
C2—N3	1.355 (3)	C12—H12	0.9300
C2—C3	1.456 (3)	C13—H13	0.9300
C3—N4	1.353 (3)	C14—H14A	0.9700
C3—C4	1.384 (3)	C14—H14B	0.9700
N3—C8	1.452 (3)	C15—H15A	0.9600
C4—C5	1.379 (4)	C15—H15B	0.9600
C4—H4	0.9300	C15—H15C	0.9600
N4—C7	1.340 (3)		

N2—C1—N3	110.5 (2)	C6—C5—H5	120.3
N2—C1—C14	126.8 (2)	C4—C5—H5	120.3
N3—C1—C14	122.7 (2)	C5—C6—C7	119.3 (3)
N1^{i}—Cr1—N1	87.33 (12)	C5—C6—H6	120.4
N1^{i}—Cr1—N4^{i}	78.06 (8)	C7—C6—H6	120.4
N1—Cr1—N4^{i}	93.14 (8)	N4—C7—C6	122.1 (2)
N1^{i}—Cr1—N4	93.15 (8)	N4—C7—H7	118.9
N1—Cr1—N4	78.06 (8)	C6—C7—H7	118.9
N4^{i}—Cr1—N4	167.94 (10)	C9—C8—C13	122.6 (3)
N1^{i}—Cr1—Cl2^{i}	90.84 (6)	C9—C8—N3	118.4 (2)
N1—Cr1—Cl2^{i}	172.01 (6)	C13—C8—N3	119.0 (2)
N4^{i}—Cr1—Cl2^{i}	94.08 (5)	C8—C9—C10	119.0 (3)
N4—Cr1—Cl2^{i}	94.29 (6)	C8—C9—H9	120.5
N1^{i}—Cr1—Cl2	172.01 (6)	C10—C9—H9	120.5
N1—Cr1—Cl2	90.83 (6)	C11—C10—C9	120.1 (3)
N4^{i}—Cr1—Cl2	94.29 (6)	C11—C10—H10	120.0
N4—Cr1—Cl2	94.07 (5)	C9—C10—H10	120.0
Cl2^{i}—Cr1—Cl2	92.02 (4)	C12—C11—C10	120.1 (3)

C2—N1—N2	110.0 (2)	C12—C11—H11	120.0
C2—N1—Cr1	114.91 (16)	C10—C11—H11	120.0
N2—N1—Cr1	135.02 (15)	C11—C12—C13	121.4 (3)
C14—O1—C15	112.6 (3)	C11—C12—H12	119.3
N1—C2—N3	108.6 (2)	C13—C12—H12	119.3
N1—C2—C3	119.3 (2)	C8—C13—C12	116.9 (3)
N3—C2—C3	132.1 (2)	C8—C13—H13	121.5
C1—N2—N1	105.3 (2)	C12—C13—H13	121.6
N4—C3—C4	121.7 (2)	O1—C14—C1	110.1 (2)
N4—C3—C2	111.8 (2)	O1—C14—H14A	109.6
C4—C3—C2	126.4 (2)	C1—C14—H14A	109.6
C2—N3—C1	105.6 (2)	O1—C14—H14B	109.6
C2—N3—C8	127.0 (2)	C1—C14—H14B	109.6
C1—N3—C8	127.4 (2)	H14A—C14—H14B	108.2
C5—C4—C3	119.0 (3)	O1—C15—H15A	109.5
C5—C4—H4	120.5	O1—C15—H15B	109.5
C3—C4—H4	120.5	H15A—C15—H15B	109.5
C7—N4—C3	118.4 (2)	O1—C15—H15C	109.5
C7—N4—Cr1	125.86 (17)	H15A—C15—H15C	109.5
C3—N4—Cr1	115.74 (15)	H15B—C15—H15C	109.5
C6—C5—C4	119.4 (3)		

Symmetry code: (i) $y+1/3, x-1/3, -z+1/6$.