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Bis(di-*n*-propylamine-κ*N*)bis(tri-*tert*-butoxysilanethiolato-κ*S*)chromium(II)

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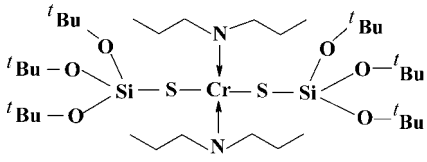
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.004 Å; R factor = 0.057; wR factor = 0.139; data-to-parameter ratio = 18.1.

The title compound, $[Cr(C_{12}H_{27}O_3SSi)_2(C_6H_{15}N)_2]$, is a molecular chromium(II) thiolate that is coordinated by two dipropylamine ligands in a square-planar environment. The molecule lies on an inversion site.

Related literature

For (tetrahydrofuran)bis(tri-*tert*-butoxysilanethiolato)-chromium(II), see: Ciborska *et al.* (2007). For the synthetic procedures, see: Perrin & Armarego (1988); Piękoś & Wojnowski (1962); Wojnowska & Wojnowski (1974). For comparison of Cr—S bond lengths, see: Okura *et al.* (1985); Ito (2002); Ciborska *et al.* (2007).



Experimental

Crystal data

$[Cr(C_{12}H_{27}O_3SSi)_2(C_6H_{15}N)_2]$
M_r = 813.35
Monoclinic, $P2_1/n$
a = 9.3573 (8) Å
b = 15.6328 (12) Å
c = 16.4333 (12) Å
β = 93.296 (7)°
V = 2399.9 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.41 mm^{-1}
T = 120 (2) K
0.52 × 0.27 × 0.22 mm

Data collection

Oxford Diffraction KM-4 CCD diffractometer
Absorption correction: none
15205 measured reflections
4230 independent reflections
3916 reflections with $I > 2\sigma(I)$
R_{int} = 0.056

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.139
S = 1.07
4230 reflections
234 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.59 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2005); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2005); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2337).

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Acta Cryst. (2008). E**64**, m46 [https://doi.org/10.1107/S1600536807050283]

Bis(di-*n*-propylamine-κN)bis(tri-*tert*-butoxysilanethiolato-κS)chromium(II)

Anna Ciborska, Katarzyna Baranowska and Wiesław Wojnowski

S1. Comment

We present here the crystal structure of the title compound (I), which is the first example of square-planar chromium(II) complex (Fig.1). It was obtained in the reaction of anhydrous Cr(II) chloride with sodium tri-*tert*-butoxysilanethiolate and dipropylamine. The Cr(II) ion is coordinated by two S atoms of the tri-*tert*-butoxysilanethiolate ligand, and two N atoms of the amine. The central Cr atom sits on an inversion centre at Wyckoff position *a* (1/2, 1/2, 1/2). The amine ligand forms intramolecular hydrogen bonds of the N–H···O type. The *trans* angles of the square base are then described by S–Cr–S and N–Cr–N. The Cr–S bond lengths are typical of Cr-thiolate complexes (Okura *et al.*, 1985; Ito 2002; Ciborska *et al.*, 2007). Selected data on important bond lengths and angles are compared in Table.1. Molecules of (I) pack in the crystal structure as discrete entities with no interactions other than von der Waals. Compound (I) is one of the few structurally defined planar, four-coordinate Cr(II) thiolate complexes.

S2. Experimental

All manipulations were conducted under an atmosphere of nitrogen using standard Schlenk techniques. Solvents and the amine were purified and dried by standard methods (Perrin & Armarego, 1988). The substrate $(^{t}BuO)_3SiSNa$ was prepared according to literature methods (Piękoś & Wojnowski, 1962; Wojnowska & Wojnowski, 1974). The compound was synthesized by addition of the $CrCl_2$ solution (0.26 g, 2.13 mmol) in tetrahydrofuran (20 ml) to $(^{t}BuO)_3SiSNa$ solution (1.24 g, 4.12 mmol) in toluene (15 ml) and stirring for 1 h. Then, to the pale-green solution dipropylamine (0.55 ml, 0.4 g, 4 mmol) was added and stirred for next 12 h. After that the mixture was filtered. The dark blue filtrate was concentrated and cooled (250 K) afford blue crystals.

S3. Refinement

All H atoms were refined as riding on C atoms with methyl C—H = 0.98 Å, methylene C—H = 0.99 Å, N–H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for CH_2 and amino groups and $1.5U_{eq}(C)$ for CH_3 groups.

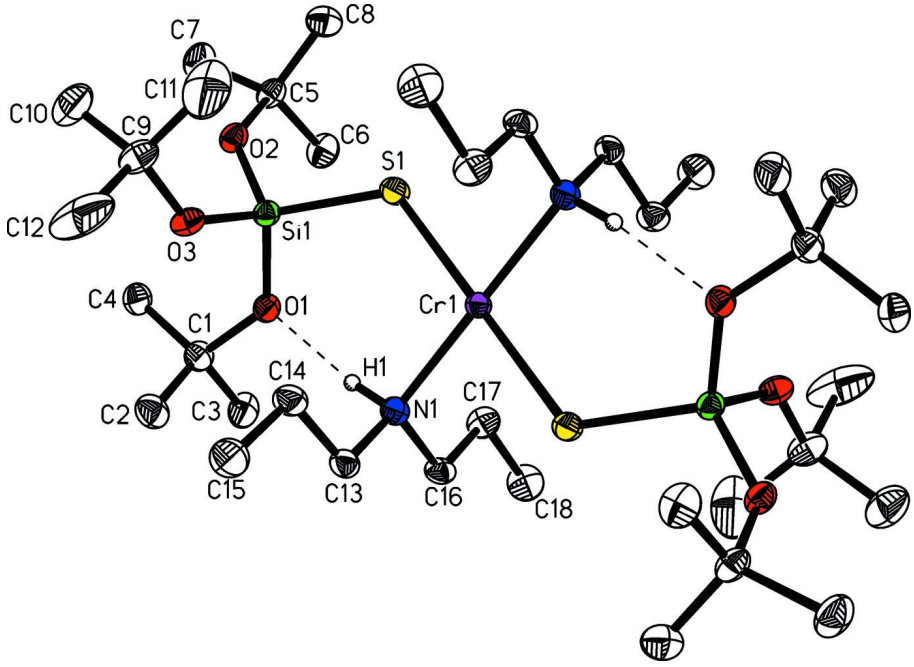

Figure 1

A view of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. C-bound H atoms have been omitted.

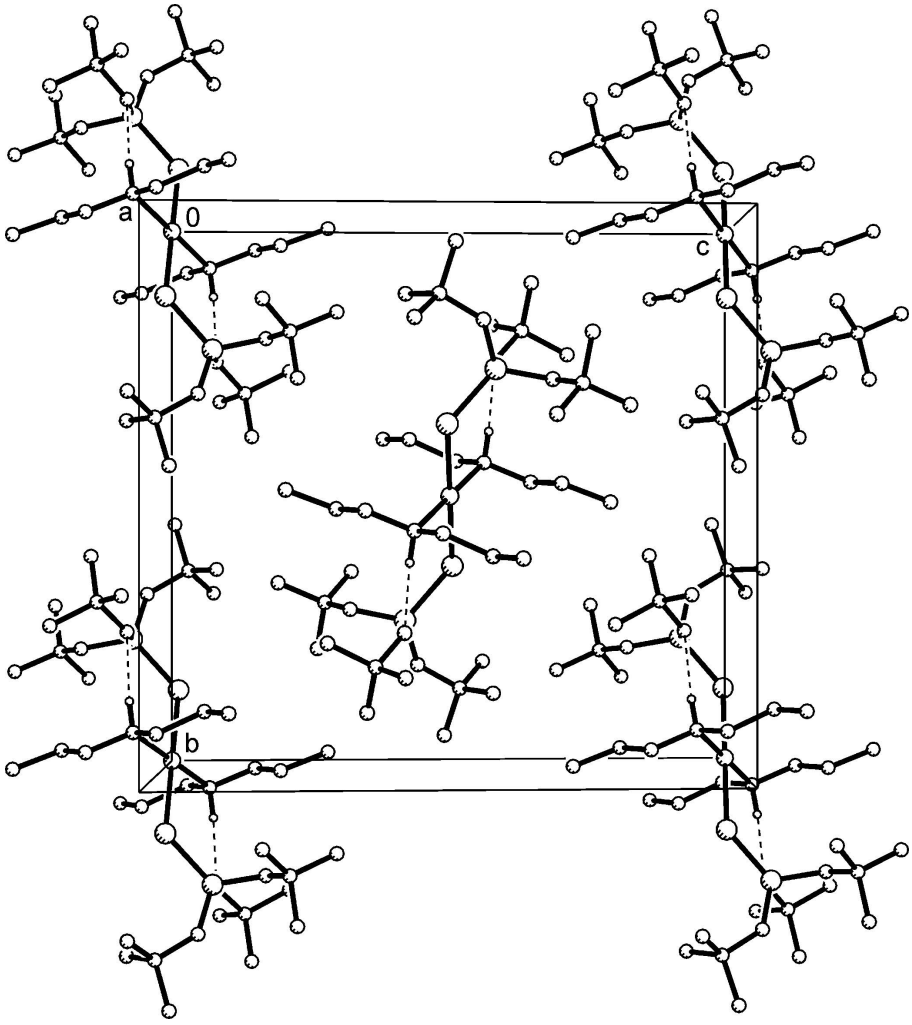

Figure 2

The crystal packing of the title compound, viewed along the *a*-axis.

Bis(di-*n*-propylamine-κ*N*)bis(tri-*tert*- butoxysilanethiolato-κ*S*)chromium(II)

Crystal data

$[Cr(C_{12}H_{27}O_3SSi)_2(C_6H_{15}N)_2]$
$M_r = 813.35$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 9.3573\ (8)$ Å
$b = 15.6328\ (12)$ Å
$c = 16.4333\ (12)$ Å
$\beta = 93.296\ (7)°$
$V = 2399.9\ (3)$ Å^3
$Z = 2$
$F(000) = 892$
$D_x = 1.126$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 8752 reflections
$\theta = 2.9–32.5°$
$\mu = 0.41$ mm^{-1}
$T = 120$ K
Prism, blue
0.52 × 0.27 × 0.22 mm

Data collection

Oxford Diffraction KM-4 CCD diffractometer
Graphite monochromator
Detector resolution: 8.1883 pixels mm^{-1}
ω (0.75° width) scans
15205 measured reflections
4230 independent reflections
3916 reflections with $I > 2\sigma(I)$
$R_{int} = 0.056$
$\theta_{max} = 25.1°$, $\theta_{min} = 2.8°$
$h = -11 \rightarrow 11$
$k = -18 \rightarrow 16$
$l = -19 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.139$
$S = 1.07$
4230 reflections
234 parameters
0 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
$w = 1/[\sigma^2(F_o^2) + (0.0625P)^2 + 4.3543P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.59$ e Å^{-3}
$\Delta\rho_{min} = -0.37$ 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.8592 (3)	0.30282 (17)	0.12476 (17)	0.0227 (6)
C2	0.8284 (3)	0.25962 (19)	0.20505 (17)	0.0282 (6)
H2A	0.775	0.2066	0.1939	0.042*
H2B	0.7715	0.2981	0.2374	0.042*
H2C	0.9189	0.2464	0.2354	0.042*
C3	0.7208 (3)	0.31714 (19)	0.07329 (19)	0.0300 (7)
H3A	0.7428	0.3422	0.0208	0.045*
H3B	0.6588	0.3562	0.1019	0.045*
H3C	0.6717	0.2623	0.0641	0.045*
C4	0.9378 (3)	0.38698 (18)	0.14046 (17)	0.0264 (6)
H4A	1.0292	0.376	0.1708	0.04*
H4B	0.8791	0.4249	0.1723	0.04*
H4C	0.9558	0.4143	0.0883	0.04*
C5	1.2379 (3)	0.35502 (17)	−0.01745 (16)	0.0212 (6)
C6	1.1047 (3)	0.35943 (19)	−0.07507 (17)	0.0288 (6)
H6A	1.0755	0.3014	−0.0914	0.043*
H6B	1.1258	0.3926	−0.1235	0.043*

H6C	1.027	0.3871	−0.0474	0.043*
C7	1.2852 (4)	0.44407 (18)	0.00984 (18)	0.0310 (7)
H7A	1.2078	0.472	0.0375	0.046*
H7B	1.3084	0.478	−0.0378	0.046*
H7C	1.3701	0.4397	0.0474	0.046*
C8	1.3579 (3)	0.30911 (19)	−0.05804 (18)	0.0292 (6)
H8A	1.4401	0.3025	−0.0188	0.044*
H8B	1.3864	0.3426	−0.1048	0.044*
H8C	1.3246	0.2526	−0.0767	0.044*
C9	1.2921 (3)	0.1975 (2)	0.22075 (18)	0.0282 (6)
C10	1.3575 (4)	0.2848 (2)	0.2355 (2)	0.0414 (8)
H10A	1.3828	0.3095	0.1835	0.062*
H10B	1.4438	0.2794	0.2718	0.062*
H10C	1.2883	0.322	0.2607	0.062*
C11	1.3932 (4)	0.1416 (3)	0.1761 (3)	0.0706 (15)
H11A	1.3511	0.0846	0.1682	0.106*
H11B	1.4844	0.1368	0.2083	0.106*
H11C	1.4098	0.1671	0.123	0.106*
C12	1.2512 (4)	0.1601 (3)	0.3016 (2)	0.0618 (13)
H12A	1.1754	0.1949	0.3236	0.093*
H12B	1.3351	0.1598	0.3401	0.093*
H12C	1.2167	0.1014	0.2931	0.093*
C13	0.8096 (3)	0.02560 (17)	0.14156 (15)	0.0203 (5)
H13A	0.7703	−0.033	0.136	0.024*
H13B	0.7394	0.061	0.1693	0.024*
C14	0.9489 (3)	0.02298 (18)	0.19315 (16)	0.0233 (6)
H14A	0.9898	0.0813	0.1973	0.028*
H14B	1.018	−0.014	0.1664	0.028*
C15	0.9268 (3)	−0.0110 (2)	0.27823 (17)	0.0311 (7)
H15A	0.8585	0.0256	0.3049	0.047*
H15B	1.0184	−0.011	0.3103	0.047*
H15C	0.8893	−0.0695	0.2744	0.047*
C16	0.6911 (3)	0.06510 (17)	0.01127 (16)	0.0203 (5)
H16A	0.622	0.0987	0.0416	0.024*
H16B	0.6525	0.0064	0.0042	0.024*
C17	0.7052 (3)	0.10509 (19)	−0.07170 (17)	0.0254 (6)
H17A	0.7743	0.0717	−0.1023	0.03*
H17B	0.7425	0.1641	−0.065	0.03*
C18	0.5605 (3)	0.1073 (2)	−0.12003 (19)	0.0324 (7)
H18A	0.529	0.0487	−0.1325	0.049*
H18B	0.5701	0.1389	−0.171	0.049*
H18C	0.4898	0.1358	−0.0876	0.049*
N1	0.8296 (2)	0.06135 (14)	0.05944 (13)	0.0187 (5)
H1	0.8581	0.1178	0.0677	0.022*
O1	0.94135 (19)	0.24496 (11)	0.07689 (11)	0.0196 (4)
O2	1.2078 (2)	0.31042 (11)	0.05632 (11)	0.0209 (4)
O3	1.15806 (19)	0.20698 (12)	0.17357 (11)	0.0217 (4)
Si1	1.11443 (7)	0.22632 (4)	0.07776 (4)	0.01703 (19)

S1	1.14306 (7)	0.12779 (4)	−0.00428 (4)	0.02372 (19)
Cr1	1	0	0	0.01522 (17)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0202 (13)	0.0198 (13)	0.0281 (14)	0.0042 (11)	0.0011 (11)	−0.0043 (11)
C2	0.0295 (15)	0.0251 (15)	0.0303 (15)	0.0039 (12)	0.0060 (12)	−0.0023 (12)
C3	0.0247 (15)	0.0274 (15)	0.0374 (16)	0.0083 (12)	−0.0041 (12)	−0.0095 (13)
C4	0.0271 (15)	0.0223 (14)	0.0299 (15)	0.0012 (11)	0.0033 (11)	−0.0072 (11)
C5	0.0239 (14)	0.0159 (13)	0.0234 (13)	−0.0055 (11)	−0.0009 (10)	0.0030 (10)
C6	0.0293 (16)	0.0282 (15)	0.0280 (14)	0.0020 (12)	−0.0056 (12)	0.0024 (12)
C7	0.0415 (18)	0.0208 (14)	0.0306 (15)	−0.0107 (13)	0.0018 (13)	0.0018 (12)
C8	0.0266 (15)	0.0293 (15)	0.0321 (15)	−0.0011 (12)	0.0057 (12)	0.0033 (12)
C9	0.0190 (14)	0.0318 (16)	0.0327 (15)	0.0001 (12)	−0.0083 (11)	0.0045 (12)
C10	0.0363 (18)	0.047 (2)	0.0396 (18)	−0.0114 (16)	−0.0104 (14)	−0.0010 (15)
C11	0.040 (2)	0.081 (3)	0.087 (3)	0.033 (2)	−0.028 (2)	−0.038 (3)
C12	0.040 (2)	0.090 (3)	0.052 (2)	−0.025 (2)	−0.0263 (17)	0.041 (2)
C13	0.0223 (13)	0.0174 (13)	0.0218 (13)	−0.0021 (10)	0.0066 (10)	−0.0014 (10)
C14	0.0236 (14)	0.0256 (14)	0.0212 (13)	−0.0027 (11)	0.0044 (10)	0.0032 (11)
C15	0.0342 (17)	0.0349 (17)	0.0243 (14)	−0.0046 (13)	0.0021 (12)	0.0041 (12)
C16	0.0152 (12)	0.0195 (13)	0.0265 (13)	0.0034 (10)	0.0041 (10)	−0.0005 (10)
C17	0.0240 (14)	0.0258 (14)	0.0260 (14)	−0.0004 (11)	−0.0016 (11)	0.0027 (11)
C18	0.0288 (16)	0.0348 (17)	0.0330 (15)	0.0069 (13)	−0.0036 (12)	0.0028 (13)
N1	0.0197 (11)	0.0154 (11)	0.0211 (11)	0.0002 (9)	0.0034 (8)	0.0000 (8)
O1	0.0179 (9)	0.0176 (9)	0.0231 (9)	0.0021 (7)	−0.0002 (7)	−0.0033 (7)
O2	0.0230 (10)	0.0175 (9)	0.0220 (9)	−0.0046 (7)	0.0002 (7)	0.0003 (7)
O3	0.0159 (9)	0.0234 (10)	0.0253 (10)	−0.0014 (8)	−0.0023 (7)	0.0034 (8)
Si1	0.0162 (4)	0.0147 (4)	0.0202 (4)	−0.0003 (3)	0.0011 (3)	−0.0008 (3)
S1	0.0239 (4)	0.0162 (3)	0.0321 (4)	−0.0046 (3)	0.0113 (3)	−0.0061 (3)
Cr1	0.0152 (3)	0.0136 (3)	0.0171 (3)	−0.0002 (2)	0.0027 (2)	−0.0008 (2)

Geometric parameters (Å, º)

C1—O1	1.448 (3)	C11—H11B	0.98
C1—C3	1.522 (4)	C11—H11C	0.98
C1—C4	1.522 (4)	C12—H12A	0.98
C1—C2	1.524 (4)	C12—H12B	0.98
C2—H2A	0.98	C12—H12C	0.98
C2—H2B	0.98	C13—N1	1.482 (3)
C2—H2C	0.98	C13—C14	1.514 (4)
C3—H3A	0.98	C13—H13A	0.99
C3—H3B	0.98	C13—H13B	0.99
C3—H3C	0.98	C14—C15	1.521 (4)
C4—H4A	0.98	C14—H14A	0.99
C4—H4B	0.98	C14—H14B	0.99
C4—H4C	0.98	C15—H15A	0.98
C5—O2	1.440 (3)	C15—H15B	0.98

C5—C8	1.519 (4)	C15—H15C	0.98
C5—C7	1.521 (4)	C16—N1	1.480 (3)
C5—C6	1.523 (4)	C16—C17	1.513 (4)
C6—H6A	0.98	C16—H16A	0.99
C6—H6B	0.98	C16—H16B	0.99
C6—H6C	0.98	C17—C18	1.530 (4)
C7—H7A	0.98	C17—H17A	0.99
C7—H7B	0.98	C17—H17B	0.99
C7—H7C	0.98	C18—H18A	0.98
C8—H8A	0.98	C18—H18B	0.98
C8—H8B	0.98	C18—H18C	0.98
C8—H8C	0.98	N1—Cr1	2.144 (2)
C9—O3	1.444 (3)	N1—H1	0.93
C9—C11	1.508 (5)	O1—Si1	1.6448 (19)
C9—C10	1.509 (4)	O2—Si1	1.6283 (19)
C9—C12	1.520 (4)	O3—Si1	1.6320 (19)
C10—H10A	0.98	Si1—S1	2.0744 (9)
C10—H10B	0.98	S1—Cr1	2.4080 (7)
C10—H10C	0.98	Cr1—N1^{i}	2.144 (2)
C11—H11A	0.98	Cr1—S1^{i}	2.4080 (7)

O1—C1—C3	104.6 (2)	H11B—C11—H11C	109.5
O1—C1—C4	111.4 (2)	C9—C12—H12A	109.5
C3—C1—C4	110.8 (2)	C9—C12—H12B	109.5
O1—C1—C2	109.0 (2)	H12A—C12—H12B	109.5
C3—C1—C2	110.4 (2)	C9—C12—H12C	109.5
C4—C1—C2	110.5 (2)	H12A—C12—H12C	109.5
C1—C2—H2A	109.5	H12B—C12—H12C	109.5
C1—C2—H2B	109.5	N1—C13—C14	111.8 (2)
H2A—C2—H2B	109.5	N1—C13—H13A	109.3
C1—C2—H2C	109.5	C14—C13—H13A	109.3
H2A—C2—H2C	109.5	N1—C13—H13B	109.3
H2B—C2—H2C	109.5	C14—C13—H13B	109.3
C1—C3—H3A	109.5	H13A—C13—H13B	107.9
C1—C3—H3B	109.5	C13—C14—C15	111.5 (2)
H3A—C3—H3B	109.5	C13—C14—H14A	109.3
C1—C3—H3C	109.5	C15—C14—H14A	109.3
H3A—C3—H3C	109.5	C13—C14—H14B	109.3
H3B—C3—H3C	109.5	C15—C14—H14B	109.3
C1—C4—H4A	109.5	H14A—C14—H14B	108
C1—C4—H4B	109.5	C14—C15—H15A	109.5
H4A—C4—H4B	109.5	C14—C15—H15B	109.5
C1—C4—H4C	109.5	H15A—C15—H15B	109.5
H4A—C4—H4C	109.5	C14—C15—H15C	109.5
H4B—C4—H4C	109.5	H15A—C15—H15C	109.5
O2—C5—C8	109.0 (2)	H15B—C15—H15C	109.5
O2—C5—C7	105.2 (2)	N1—C16—C17	112.3 (2)
C8—C5—C7	110.6 (2)	N1—C16—H16A	109.1

O2—C5—C6	110.6 (2)	C17—C16—H16A	109.1
C8—C5—C6	110.3 (2)	N1—C16—H16B	109.1
C7—C5—C6	110.9 (2)	C17—C16—H16B	109.1
C5—C6—H6A	109.5	H16A—C16—H16B	107.9
C5—C6—H6B	109.5	C16—C17—C18	110.9 (2)
H6A—C6—H6B	109.5	C16—C17—H17A	109.5
C5—C6—H6C	109.5	C18—C17—H17A	109.5
H6A—C6—H6C	109.5	C16—C17—H17B	109.5
H6B—C6—H6C	109.5	C18—C17—H17B	109.5
C5—C7—H7A	109.5	H17A—C17—H17B	108
C5—C7—H7B	109.5	C17—C18—H18A	109.5
H7A—C7—H7B	109.5	C17—C18—H18B	109.5
C5—C7—H7C	109.5	H18A—C18—H18B	109.5
H7A—C7—H7C	109.5	C17—C18—H18C	109.5
H7B—C7—H7C	109.5	H18A—C18—H18C	109.5
C5—C8—H8A	109.5	H18B—C18—H18C	109.5
C5—C8—H8B	109.5	C16—N1—C13	110.5 (2)
H8A—C8—H8B	109.5	C16—N1—Cr1	115.17 (15)
C5—C8—H8C	109.5	C13—N1—Cr1	112.46 (16)
H8A—C8—H8C	109.5	C16—N1—H1	106
H8B—C8—H8C	109.5	C13—N1—H1	106
O3—C9—C11	110.4 (3)	Cr1—N1—H1	106
O3—C9—C10	109.0 (2)	C1—O1—Si1	131.23 (16)
C11—C9—C10	110.0 (3)	C5—O2—Si1	134.93 (16)
O3—C9—C12	104.7 (2)	C9—O3—Si1	134.30 (17)
C11—C9—C12	113.5 (4)	O2—Si1—O3	104.52 (10)
C10—C9—C12	109.1 (3)	O2—Si1—O1	113.29 (10)
C9—C10—H10A	109.5	O3—Si1—O1	103.46 (9)
C9—C10—H10B	109.5	O2—Si1—S1	111.60 (7)
H10A—C10—H10B	109.5	O3—Si1—S1	117.06 (8)
C9—C10—H10C	109.5	O1—Si1—S1	106.85 (7)
H10A—C10—H10C	109.5	Si1—S1—Cr1	120.28 (3)
H10B—C10—H10C	109.5	N1^{i}—Cr1—N1	180.00 (15)
C9—C11—H11A	109.5	N1^{i}—Cr1—S1	85.90 (6)
C9—C11—H11B	109.5	N1—Cr1—S1	94.10 (6)
H11A—C11—H11B	109.5	N1^{i}—Cr1—S1^{i}	94.10 (6)
C9—C11—H11C	109.5	N1—Cr1—S1^{i}	85.90 (6)
H11A—C11—H11C	109.5	S1—Cr1—S1^{i}	180.000 (17)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1	0.93	2.14	3.063 (3)	174