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2-Hydroxyethyl 2-(2,4-dichloroanilino)-4,4-dimethyl-6-oxocyclohex-1-enecarbodithioate

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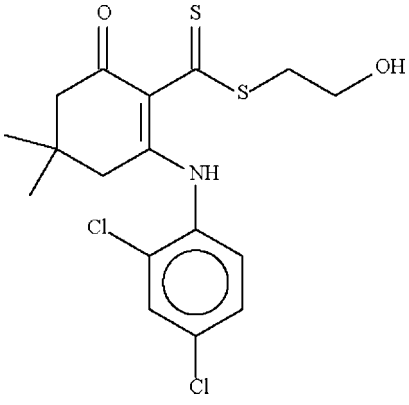
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.037; wR factor = 0.098; data-to-parameter ratio = 18.9.

The six-membered cyclohexene ring in the title compound, $C_{17}H_{19}Cl_2NOS_2$, adopts an envelope conformation, with the C atom bearing the two methyl groups representing the flap. This atom deviates by 0.716 (3) Å from the plane passing through the other five atoms of the ring (r.m.s. deviation = 0.072 Å). The molecular conformation is stabilized by an intramolecular N—H···S hydrogen bond. The hydroxy group engages in intermolecular O—H···O hydrogen bonding with adjacent acceptor atoms to generate a zigzag chain running along the c axis.

Related literature

For background, see: El Ashry *et al.* (2009).



Experimental

Crystal data

$C_{17}H_{19}Cl_2NO_2S_2$
M_r = 404.35
Monoclinic, $P2_1/c$
a = 11.7310 (2) Å
b = 14.0903 (2) Å
c = 12.0416 (2) Å
β = 111.245 (1)°
V = 1855.13 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.59 mm^{-1}
T = 100 K
0.40 × 0.10 × 0.10 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.895, T_{max} = 0.944
17368 measured reflections
4269 independent reflections
3581 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.098
S = 1.17
4269 reflections
226 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 1.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O1^{i}	0.83 (1)	1.92 (1)	2.737 (2)	165 (3)
N1—H1···S2	0.88 (1)	2.14 (2)	2.913 (2)	147 (2)

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

We thank the Higher Education Commission of Pakistan and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2881).

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supporting information

Acta Cryst. (2009). E**65**, o602 [doi:10.1107/S1600536809006199]

2-Hydroxyethyl 2-(2,4-dichloroanilino)-4,4-dimethyl-6-oxocyclohex-1-enecarbodithioate

El Sayed H. El Ashry, Mohammed R. Amer, M. Raza Shah and Seik Weng Ng

S1. Experimental

To a solution of (2,4-dichlorophenylamino)-5,5-dimethyl-cyclohex-2-en-1-one (0.1 mol) in DMSO (20 ml) and sodium hydroxide (0.4 g) in water (1 ml), carbon disulfphide (0.3 mol) was added in the course of 30 minutes. The mixture was stirred for 20 min below 283 K, and then 2-bromoethanol (0.1 mol) was added drop wise at room temperature for 30 min. The reaction mixture was left for 24 h and then diluted with water (200 ml) and acidified with 10% hydrochloric acid. The resulting precipitate was collected by filtration, dried and purified on silica gel column (40% ethyl acetate in hexane) to give yellow crystal (34.5% yield; mp.424 K).

S2. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.93 to 0.99 Å) and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2 to 1.5*U*(C).

The amino and hydroxy H-atoms were located in a difference Fourier map, and were refined with distance restraints of N–H 0.88±0.01 and O–H 0.84±0.01 Å; their isotropic displacement parameters were freely refined.

The final difference Fourier map had a large peak at 1 Å from S1, but was otherwise featureless.

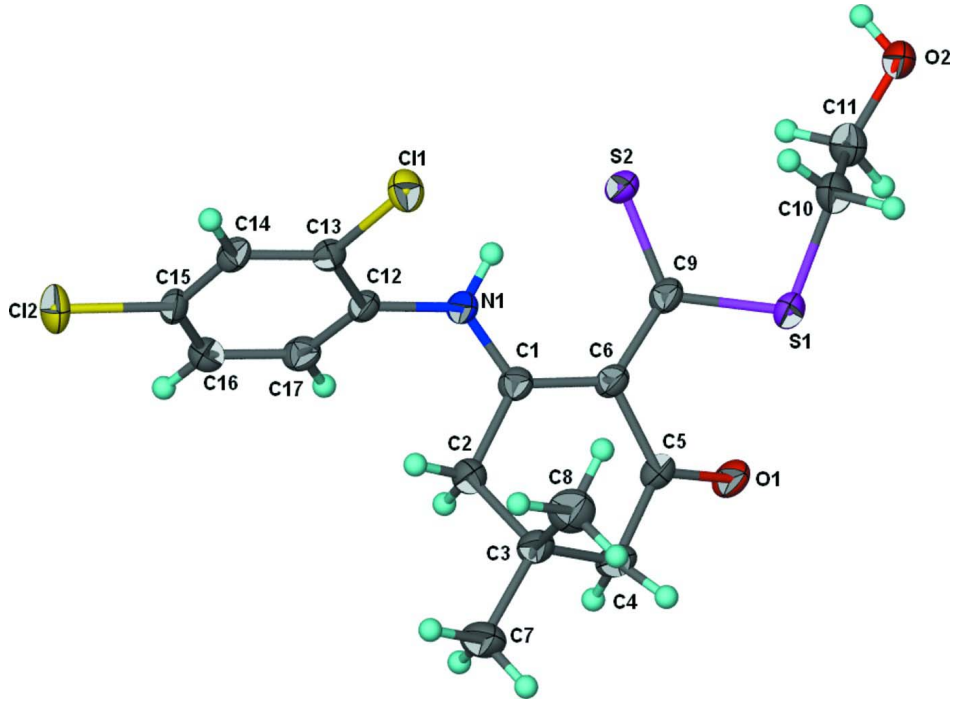

Figure 1

Anisotropic displacement ellipsoid plot (Barbour, 2001) plot of $C_{17}H_{19}Cl_2NO_2S_2$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

2-Hydroxyethyl 2-(2,4-dichloroanilino)-4,4-dimethyl-6-oxocyclohex-1-enecarbodithioate

Crystal data

$C_{17}H_{19}Cl_2NO_2S_2$
$M_r = 404.35$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 11.7310\ (2)$ Å
$b = 14.0903\ (2)$ Å
$c = 12.0416\ (2)$ Å
$\beta = 111.245\ (1)°$
$V = 1855.13\ (5)$ Å^3
$Z = 4$
$F(000) = 840$
$D_x = 1.448$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5593 reflections
$\theta = 2.3–28.2°$
$\mu = 0.59$ mm^{-1}
$T = 100$ K
Prism, orange
0.40 × 0.10 × 0.10 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.895$, $T_{max} = 0.944$
17368 measured reflections
4269 independent reflections
3581 reflections with $I > 2\sigma(I)$
$R_{int} = 0.031$
$\theta_{max} = 27.5°$, $\theta_{min} = 1.9°$
$h = -15\rightarrow15$
$k = -18\rightarrow18$
$l = -15\rightarrow15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.098$
$S = 1.17$
4269 reflections
226 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0429P)^2 + 0.8915P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.48$ e Å^{-3}
$\Delta\rho_{min} = -0.49$ 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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.50297 (4)	0.66542 (3)	0.81611 (4)	0.02438 (12)
Cl2	0.69365 (5)	0.33055 (4)	1.00864 (5)	0.02908 (13)
S1	−0.02899 (5)	0.74647 (4)	0.42522 (4)	0.02988 (14)
S2	0.11953 (5)	0.71791 (4)	0.68063 (4)	0.02937 (14)
O1	−0.05234 (12)	0.57916 (11)	0.32339 (12)	0.0255 (3)
O2	−0.14964 (14)	0.95304 (11)	0.58309 (13)	0.0302 (4)
H2	−0.123 (2)	0.954 (2)	0.6574 (9)	0.048 (8)*
N1	0.27644 (14)	0.55532 (12)	0.69084 (14)	0.0203 (3)
H1	0.250 (2)	0.6051 (12)	0.718 (2)	0.038 (7)*
C1	0.21902 (17)	0.53820 (13)	0.57550 (17)	0.0188 (4)
C2	0.26758 (19)	0.45827 (15)	0.52239 (17)	0.0239 (4)
H2A	0.2247	0.3989	0.5278	0.029*
H2B	0.3556	0.4495	0.5698	0.029*
C3	0.25180 (18)	0.47536 (15)	0.39220 (17)	0.0239 (4)
C4	0.11563 (19)	0.48819 (16)	0.32527 (18)	0.0266 (4)
H4A	0.1028	0.5110	0.2439	0.032*
H4B	0.0754	0.4255	0.3176	0.032*
C5	0.05389 (17)	0.55607 (14)	0.38206 (16)	0.0203 (4)
C6	0.11713 (16)	0.59172 (14)	0.50288 (16)	0.0191 (4)
C7	0.2972 (2)	0.38762 (17)	0.3452 (2)	0.0343 (5)
H7A	0.2836	0.3965	0.2606	0.051*
H7B	0.2523	0.3315	0.3549	0.051*
H7C	0.3848	0.3788	0.3901	0.051*
C8	0.3236 (2)	0.56243 (17)	0.3788 (2)	0.0326 (5)
H8A	0.3078	0.5734	0.2942	0.049*
H8B	0.4112	0.5516	0.4212	0.049*
H8C	0.2977	0.6181	0.4124	0.049*
C9	0.07364 (17)	0.67738 (14)	0.54093 (16)	0.0203 (4)

C10	−0.0347 (2)	0.85810 (15)	0.49607 (19)	0.0289 (5)
H10A	0.0452	0.8699	0.5604	0.035*
H10B	−0.0496	0.9099	0.4370	0.035*
C11	−0.13386 (19)	0.85877 (16)	0.5476 (2)	0.0293 (5)
H11A	−0.2114	0.8358	0.4873	0.035*
H11B	−0.1115	0.8158	0.6173	0.035*
C12	0.37500 (16)	0.50105 (14)	0.76960 (16)	0.0182 (4)
C13	0.48643 (17)	0.54444 (13)	0.83201 (16)	0.0175 (4)
C14	0.58495 (16)	0.49276 (14)	0.90679 (16)	0.0183 (4)
H14	0.6609	0.5228	0.9489	0.022*
C15	0.56972 (17)	0.39647 (14)	0.91836 (16)	0.0189 (4)
C16	0.45843 (18)	0.35196 (14)	0.86133 (17)	0.0216 (4)
H16	0.4491	0.2861	0.8727	0.026*
C17	0.36107 (17)	0.40498 (14)	0.78745 (17)	0.0217 (4)
H17	0.2841	0.3754	0.7486	0.026*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0248 (2)	0.0197 (2)	0.0274 (3)	0.00018 (18)	0.00800 (19)	0.00396 (18)
Cl2	0.0231 (2)	0.0295 (3)	0.0298 (3)	0.0102 (2)	0.0038 (2)	0.0088 (2)
S1	0.0367 (3)	0.0309 (3)	0.0171 (2)	0.0127 (2)	0.0039 (2)	0.0018 (2)
S2	0.0306 (3)	0.0345 (3)	0.0157 (2)	0.0153 (2)	−0.0004 (2)	−0.0050 (2)
O1	0.0188 (7)	0.0343 (8)	0.0179 (7)	0.0032 (6)	0.0001 (5)	−0.0036 (6)
O2	0.0316 (8)	0.0319 (8)	0.0202 (8)	0.0137 (7)	0.0011 (6)	−0.0012 (6)
N1	0.0167 (8)	0.0233 (9)	0.0166 (8)	0.0055 (7)	0.0009 (6)	−0.0029 (6)
C1	0.0163 (8)	0.0212 (9)	0.0179 (9)	0.0001 (7)	0.0051 (7)	−0.0012 (7)
C2	0.0247 (10)	0.0254 (10)	0.0199 (10)	0.0071 (8)	0.0062 (8)	−0.0008 (8)
C3	0.0234 (10)	0.0294 (11)	0.0178 (9)	0.0062 (8)	0.0063 (8)	−0.0033 (8)
C4	0.0253 (10)	0.0339 (11)	0.0178 (9)	0.0047 (9)	0.0045 (8)	−0.0074 (8)
C5	0.0199 (9)	0.0237 (10)	0.0152 (9)	−0.0006 (8)	0.0037 (7)	0.0002 (7)
C6	0.0165 (9)	0.0247 (10)	0.0142 (9)	0.0017 (7)	0.0031 (7)	−0.0015 (7)
C7	0.0358 (12)	0.0372 (13)	0.0290 (11)	0.0103 (10)	0.0107 (10)	−0.0094 (10)
C8	0.0306 (11)	0.0395 (13)	0.0313 (12)	0.0024 (10)	0.0155 (9)	−0.0023 (10)
C9	0.0154 (9)	0.0271 (10)	0.0160 (9)	0.0029 (7)	0.0027 (7)	−0.0006 (7)
C10	0.0365 (12)	0.0255 (10)	0.0234 (10)	0.0071 (9)	0.0093 (9)	0.0036 (8)
C11	0.0247 (10)	0.0307 (11)	0.0279 (11)	0.0055 (9)	0.0038 (9)	0.0000 (9)
C12	0.0157 (9)	0.0236 (9)	0.0137 (8)	0.0038 (7)	0.0033 (7)	−0.0014 (7)
C13	0.0192 (9)	0.0192 (9)	0.0152 (8)	0.0006 (7)	0.0075 (7)	0.0003 (7)
C14	0.0136 (8)	0.0248 (10)	0.0156 (9)	−0.0002 (7)	0.0043 (7)	−0.0001 (7)
C15	0.0182 (9)	0.0233 (9)	0.0144 (9)	0.0071 (7)	0.0048 (7)	0.0033 (7)
C16	0.0246 (10)	0.0188 (9)	0.0212 (10)	0.0015 (8)	0.0081 (8)	−0.0010 (7)
C17	0.0184 (9)	0.0245 (10)	0.0198 (9)	−0.0018 (8)	0.0042 (7)	−0.0039 (8)

Geometric parameters (Å, º)

Cl1—C13	1.7341 (19)	C5—C6	1.462 (3)
Cl2—C15	1.7358 (18)	C6—C9	1.448 (3)

S1—C9	1.7671 (19)	C7—H7A	0.9800
S1—C10	1.802 (2)	C7—H7B	0.9800
S2—C9	1.6701 (19)	C7—H7C	0.9800
O1—C5	1.233 (2)	C8—H8A	0.9800
O2—C11	1.428 (3)	C8—H8B	0.9800
O2—H2	0.834 (10)	C8—H8C	0.9800
N1—C1	1.328 (2)	C10—C11	1.504 (3)
N1—C12	1.424 (2)	C10—H10A	0.9900
N1—H1	0.879 (10)	C10—H10B	0.9900
C1—C6	1.416 (3)	C11—H11A	0.9900
C1—C2	1.505 (3)	C11—H11B	0.9900
C2—C3	1.530 (3)	C12—C17	1.389 (3)
C2—H2A	0.9900	C12—C13	1.391 (3)
C2—H2B	0.9900	C13—C14	1.387 (3)
C3—C4	1.517 (3)	C14—C15	1.382 (3)
C3—C8	1.529 (3)	C14—H14	0.9500
C3—C7	1.533 (3)	C15—C16	1.385 (3)
C4—C5	1.505 (3)	C16—C17	1.385 (3)
C4—H4A	0.9900	C16—H16	0.9500
C4—H4B	0.9900	C17—H17	0.9500

C9—S1—C10	103.83 (10)	C3—C8—H8B	109.5
C11—O2—H2	106.8 (19)	H8A—C8—H8B	109.5
C1—N1—C12	125.53 (16)	C3—C8—H8C	109.5
C1—N1—H1	115.2 (17)	H8A—C8—H8C	109.5
C12—N1—H1	119.3 (17)	H8B—C8—H8C	109.5
N1—C1—C6	122.96 (17)	C6—C9—S2	125.58 (14)
N1—C1—C2	116.98 (16)	C6—C9—S1	115.29 (13)
C6—C1—C2	120.05 (16)	S2—C9—S1	118.99 (11)
C1—C2—C3	113.19 (16)	C11—C10—S1	111.57 (16)
C1—C2—H2A	108.9	C11—C10—H10A	109.3
C3—C2—H2A	108.9	S1—C10—H10A	109.3
C1—C2—H2B	108.9	C11—C10—H10B	109.3
C3—C2—H2B	108.9	S1—C10—H10B	109.3
H2A—C2—H2B	107.8	H10A—C10—H10B	108.0
C4—C3—C8	111.24 (18)	O2—C11—C10	109.44 (18)
C4—C3—C2	106.04 (16)	O2—C11—H11A	109.8
C8—C3—C2	111.58 (17)	C10—C11—H11A	109.8
C4—C3—C7	109.87 (17)	O2—C11—H11B	109.8
C8—C3—C7	109.25 (18)	C10—C11—H11B	109.8
C2—C3—C7	108.80 (17)	H11A—C11—H11B	108.2
C5—C4—C3	114.95 (16)	C17—C12—C13	118.99 (17)
C5—C4—H4A	108.5	C17—C12—N1	120.92 (17)
C3—C4—H4A	108.5	C13—C12—N1	120.07 (17)
C5—C4—H4B	108.5	C14—C13—C12	121.26 (17)
C3—C4—H4B	108.5	C14—C13—Cl1	119.19 (14)
H4A—C4—H4B	107.5	C12—C13—Cl1	119.55 (14)
O1—C5—C6	121.51 (17)	C15—C14—C13	118.24 (17)

O1—C5—C4	117.40 (17)	C15—C14—H14	120.9
C6—C5—C4	121.08 (16)	C13—C14—H14	120.9
C1—C6—C9	124.33 (17)	C14—C15—C16	121.83 (17)
C1—C6—C5	116.24 (17)	C14—C15—Cl2	118.61 (14)
C9—C6—C5	119.43 (16)	C16—C15—Cl2	119.55 (15)
C3—C7—H7A	109.5	C15—C16—C17	118.94 (18)
C3—C7—H7B	109.5	C15—C16—H16	120.5
H7A—C7—H7B	109.5	C17—C16—H16	120.5
C3—C7—H7C	109.5	C16—C17—C12	120.60 (18)
H7A—C7—H7C	109.5	C16—C17—H17	119.7
H7B—C7—H7C	109.5	C12—C17—H17	119.7
C3—C8—H8A	109.5		

C12—N1—C1—C6	176.51 (18)	C1—C6—C9—S1	164.93 (16)
C12—N1—C1—C2	−4.0 (3)	C5—C6—C9—S1	−15.1 (2)
N1—C1—C2—C3	−147.64 (18)	C10—S1—C9—C6	−166.31 (15)
C6—C1—C2—C3	31.9 (3)	C10—S1—C9—S2	9.62 (15)
C1—C2—C3—C4	−58.6 (2)	C9—S1—C10—C11	−89.89 (16)
C1—C2—C3—C8	62.7 (2)	S1—C10—C11—O2	−169.55 (13)
C1—C2—C3—C7	−176.75 (17)	C1—N1—C12—C17	−59.2 (3)
C8—C3—C4—C5	−73.5 (2)	C1—N1—C12—C13	122.2 (2)
C2—C3—C4—C5	48.0 (2)	C17—C12—C13—C14	3.2 (3)
C7—C3—C4—C5	165.45 (19)	N1—C12—C13—C14	−178.21 (17)
C3—C4—C5—O1	170.11 (18)	C17—C12—C13—Cl1	−176.94 (14)
C3—C4—C5—C6	−10.9 (3)	N1—C12—C13—Cl1	1.7 (2)
N1—C1—C6—C9	8.1 (3)	C12—C13—C14—C15	−0.2 (3)
C2—C1—C6—C9	−171.37 (18)	Cl1—C13—C14—C15	179.86 (14)
N1—C1—C6—C5	−171.89 (18)	C13—C14—C15—C16	−2.5 (3)
C2—C1—C6—C5	8.6 (3)	C13—C14—C15—Cl2	178.31 (14)
O1—C5—C6—C1	159.28 (18)	C14—C15—C16—C17	2.3 (3)
C4—C5—C6—C1	−19.6 (3)	Cl2—C15—C16—C17	−178.56 (15)
O1—C5—C6—C9	−20.7 (3)	C15—C16—C17—C12	0.7 (3)
C4—C5—C6—C9	160.35 (18)	C13—C12—C17—C16	−3.4 (3)
C1—C6—C9—S2	−10.7 (3)	N1—C12—C17—C16	177.98 (17)
C5—C6—C9—S2	169.31 (15)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O1^{i}	0.83 (1)	1.92 (1)	2.737 (2)	165 (3)
N1—H1···S2	0.88 (1)	2.14 (2)	2.913 (2)	147 (2)

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