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

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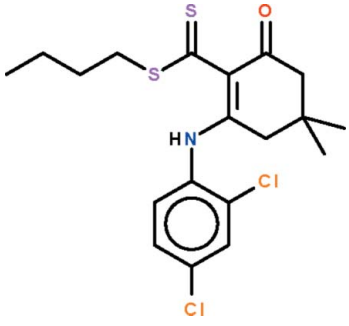
Key indicators: single-crystal X-ray study; T = 123 K; mean σ(C–C) = 0.002 Å; R factor = 0.025; wR factor = 0.070; data-to-parameter ratio = 19.8.

The cyclohexene ring in the title compound, $C_{19}H_{23}Cl_2NOS_2$, adopts an envelope conformation, with the C atom bearing the two methyl groups representing the flap. This atom deviates by 0.630 (2) Å from the plane passing through the other five atoms of the ring (r.m.s. deviation = 0.020 Å). The molecular conformation is stabilized by an intramolecular N—H···S hydrogen bond.

Related literature

For the crystal structures of the *n*-undecanyl and 2-hydroxyethyl analogues, see: El Ashry *et al.* (2009*a*,*b*).



Experimental

Crystal data

$C_{19}H_{23}Cl_2NOS_2$
M_r = 416.40
Monoclinic, $P2_1/c$
a = 9.0321 (1) Å
b = 19.4422 (2) Å
c = 11.4700 (1) Å
β = 100.331 (1)°
V = 1981.52 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.55 mm^{-1}
T = 123 K
0.30 × 0.20 × 0.10 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.853, T_{max} = 0.947
18747 measured reflections
4554 independent reflections
4279 reflections with $I > 2\sigma(I)$
R_{int} = 0.016

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.070
S = 1.00
4554 reflections
230 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···S2	0.91 (2)	2.08 (2)	2.885 (1)	147 (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).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2904).

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supporting information

Acta Cryst. (2009). E**65**, o2459 [doi:10.1107/S1600536809036320]

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

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S1. Experimental

A cooled (283 K) solution of (2,4-dichloroanilino)-4,4-dimethyl-6-oxocyclohex-1-ene (0.2 mol) and sodium hydroxide (0.2 mol) in DMSO (30 ml) and water (2 ml), was treated with carbon disulfide (0.3 mol). After 40 min, *n*-bromobutane (0.15 mol) was added and the reaction mixture was left overnight. The mixture was then diluted with water (200 ml) and acidified with 10% hydrochloric acid. The product was purified on silica gel column chromatography to give yellow crystals when recrystallized from ethanol (m.p. 401 K).

S2. Refinement

The N-bound H atom was located in a difference Fourier map and was refined freely. C-bound H atoms were placed in calculated positions (C—H = 0.95–0.99 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2 to 1.5U_{eq}(C).

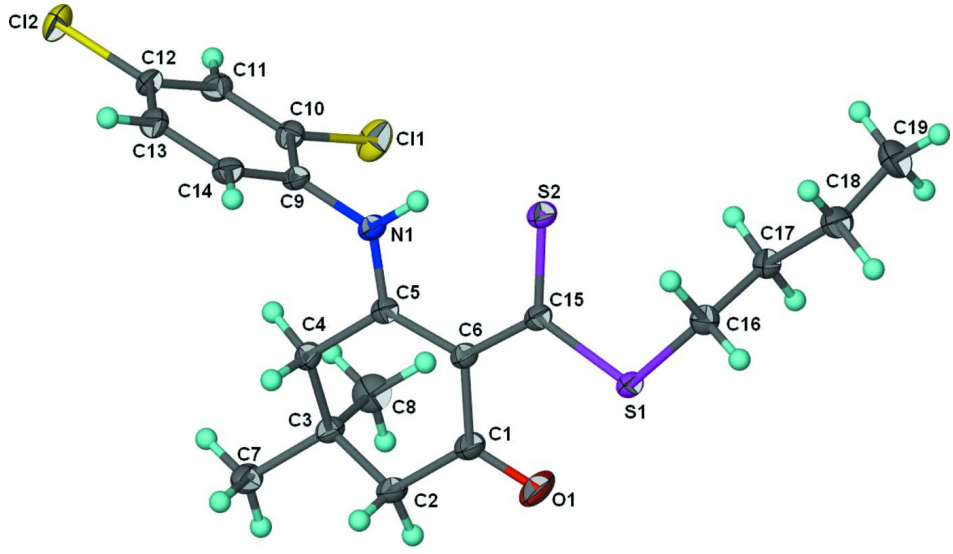

Figure 1

Displacement ellipsoid plot (Barbour, 2001) plot of $C_{19}H_{23}Cl_2NOS_2$ at the 70% probability level; H atoms are drawn as spheres of arbitrary radius.

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

Crystal data

$C_{19}H_{23}Cl_2NOS_2$
$M_r = 416.40$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 9.0321$ (1) Å
$b = 19.4422$ (2) Å
$c = 11.4700$ (1) Å
$\beta = 100.331$ (1)°
$V = 1981.52$ (3) Å^3
$Z = 4$
$F(000) = 872$
$D_x = 1.396$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9957 reflections
$\theta = 2.5$–$28.2°$
$\mu = 0.55$ mm^{-1}
$T = 123$ K
Block, yellow
0.30 × 0.20 × 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)
$T_{min} = 0.853$, $T_{max} = 0.947$
18747 measured reflections
4554 independent reflections
4279 reflections with $I > 2\sigma(I)$
$R_{int} = 0.016$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.1°$
$h = -11 \rightarrow 11$
$k = -25 \rightarrow 25$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.025$
$wR(F^2) = 0.070$
$S = 1.00$
4554 reflections
230 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0405P)^2 + 0.9836P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.41$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ 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	0.66246 (3)	0.420713 (17)	0.99199 (3)	0.02548 (8)
Cl2	1.25630 (3)	0.391875 (17)	1.00782 (3)	0.02572 (8)
S1	0.17044 (3)	0.547076 (14)	0.56447 (2)	0.01521 (7)
S2	0.45310 (3)	0.595199 (14)	0.71292 (3)	0.01818 (8)
O1	0.18119 (12)	0.41970 (5)	0.52118 (12)	0.0435 (3)
N1	0.65717 (11)	0.47990 (5)	0.74995 (8)	0.01551 (19)
H1	0.625 (2)	0.5237 (10)	0.7584 (16)	0.033 (4)*

C1	0.31311 (14)	0.40793 (6)	0.56335 (11)	0.0194 (2)
C2	0.37252 (13)	0.33742 (6)	0.54278 (10)	0.0176 (2)
H2A	0.4118	0.3381	0.4676	0.021*
H2B	0.2878	0.3044	0.5331	0.021*
C3	0.49613 (12)	0.31158 (6)	0.64090 (10)	0.0150 (2)
C4	0.61567 (12)	0.36787 (6)	0.66192 (10)	0.0163 (2)
H4A	0.6925	0.3547	0.7312	0.020*
H4B	0.6662	0.3698	0.5922	0.020*
C5	0.55899 (12)	0.43880 (6)	0.68327 (9)	0.0133 (2)
C6	0.41136 (12)	0.46044 (6)	0.63000 (10)	0.0141 (2)
C7	0.56586 (14)	0.24599 (6)	0.60044 (11)	0.0203 (2)
H7A	0.6051	0.2554	0.5277	0.030*
H7B	0.4890	0.2100	0.5851	0.030*
H7C	0.6482	0.2306	0.6626	0.030*
C8	0.43521 (15)	0.29707 (7)	0.75460 (11)	0.0246 (3)
H8A	0.3590	0.2607	0.7397	0.037*
H8B	0.3898	0.3390	0.7800	0.037*
H8C	0.5178	0.2822	0.8170	0.037*
C9	0.80208 (12)	0.45912 (6)	0.81146 (10)	0.0144 (2)
C10	0.81793 (13)	0.43077 (6)	0.92496 (10)	0.0160 (2)
C11	0.95775 (13)	0.40992 (6)	0.98561 (10)	0.0171 (2)
H11	0.9681	0.3902	1.0624	0.021*
C12	1.08177 (13)	0.41848 (6)	0.93158 (10)	0.0171 (2)
C13	1.07029 (13)	0.44805 (6)	0.82040 (10)	0.0187 (2)
H13	1.1572	0.4543	0.7856	0.022*
C14	0.92928 (13)	0.46846 (6)	0.76063 (10)	0.0169 (2)
H14	0.9198	0.4889	0.6845	0.020*
C15	0.35496 (12)	0.53004 (6)	0.63903 (9)	0.0140 (2)
C16	0.14330 (13)	0.63819 (6)	0.58501 (10)	0.0162 (2)
H16A	0.2411	0.6618	0.5885	0.019*
H16B	0.0737	0.6561	0.5150	0.019*
C17	0.08095 (13)	0.65650 (6)	0.69585 (10)	0.0169 (2)
H17A	0.1575	0.6462	0.7669	0.020*
H17B	−0.0089	0.6280	0.6994	0.020*
C18	0.03800 (14)	0.73269 (6)	0.69634 (11)	0.0226 (2)
H18A	0.1254	0.7609	0.6846	0.027*
H18B	−0.0451	0.7418	0.6292	0.027*
C19	−0.01100 (17)	0.75431 (7)	0.81137 (13)	0.0308 (3)
H19A	−0.0374	0.8033	0.8074	0.046*
H19B	0.0716	0.7464	0.8780	0.046*
H19C	−0.0988	0.7272	0.8227	0.046*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.01664 (14)	0.03539 (18)	0.02576 (15)	−0.00287 (11)	0.00749 (11)	0.00779 (12)
Cl2	0.01535 (14)	0.03229 (17)	0.02687 (16)	0.00715 (11)	−0.00338 (11)	0.00089 (12)
S1	0.01301 (13)	0.01582 (14)	0.01536 (13)	0.00136 (9)	−0.00128 (10)	−0.00127 (9)

S2	0.01510 (14)	0.01493 (14)	0.02250 (15)	−0.00004 (10)	−0.00210 (11)	−0.00381 (10)
O1	0.0189 (5)	0.0256 (5)	0.0756 (8)	0.0054 (4)	−0.0192 (5)	−0.0213 (5)
N1	0.0130 (4)	0.0142 (5)	0.0177 (5)	−0.0003 (3)	−0.0015 (3)	−0.0014 (4)
C1	0.0162 (5)	0.0169 (5)	0.0229 (6)	0.0000 (4)	−0.0023 (4)	−0.0034 (4)
C2	0.0170 (5)	0.0148 (5)	0.0188 (5)	−0.0003 (4)	−0.0028 (4)	−0.0027 (4)
C3	0.0135 (5)	0.0141 (5)	0.0162 (5)	−0.0014 (4)	0.0000 (4)	0.0001 (4)
C4	0.0123 (5)	0.0148 (5)	0.0210 (5)	−0.0003 (4)	0.0012 (4)	−0.0022 (4)
C5	0.0128 (5)	0.0150 (5)	0.0125 (5)	−0.0017 (4)	0.0028 (4)	0.0006 (4)
C6	0.0122 (5)	0.0145 (5)	0.0150 (5)	−0.0011 (4)	0.0008 (4)	−0.0008 (4)
C7	0.0188 (5)	0.0145 (5)	0.0259 (6)	−0.0003 (4)	−0.0010 (4)	−0.0021 (4)
C8	0.0264 (6)	0.0257 (6)	0.0225 (6)	−0.0027 (5)	0.0066 (5)	0.0050 (5)
C9	0.0124 (5)	0.0139 (5)	0.0156 (5)	−0.0009 (4)	−0.0009 (4)	−0.0023 (4)
C10	0.0138 (5)	0.0169 (5)	0.0175 (5)	−0.0024 (4)	0.0035 (4)	−0.0003 (4)
C11	0.0181 (6)	0.0169 (5)	0.0153 (5)	−0.0003 (4)	0.0002 (4)	0.0009 (4)
C12	0.0128 (5)	0.0178 (5)	0.0186 (5)	0.0025 (4)	−0.0028 (4)	−0.0027 (4)
C13	0.0142 (5)	0.0239 (6)	0.0183 (5)	−0.0002 (4)	0.0037 (4)	−0.0027 (4)
C14	0.0169 (5)	0.0197 (5)	0.0137 (5)	−0.0016 (4)	0.0010 (4)	−0.0012 (4)
C15	0.0126 (5)	0.0166 (5)	0.0125 (5)	−0.0006 (4)	0.0013 (4)	0.0003 (4)
C16	0.0176 (5)	0.0144 (5)	0.0158 (5)	0.0018 (4)	0.0007 (4)	0.0019 (4)
C17	0.0156 (5)	0.0163 (5)	0.0184 (5)	0.0011 (4)	0.0020 (4)	−0.0009 (4)
C18	0.0224 (6)	0.0180 (6)	0.0251 (6)	0.0033 (4)	−0.0016 (5)	−0.0030 (4)
C19	0.0309 (7)	0.0264 (7)	0.0340 (7)	0.0087 (5)	0.0030 (6)	−0.0091 (6)

Geometric parameters (Å, º)

Cl1—C10	1.7287 (11)	C8—H8A	0.98
Cl2—C12	1.7387 (12)	C8—H8B	0.98
S1—C15	1.7628 (11)	C8—H8C	0.98
S1—C16	1.8094 (12)	C9—C14	1.3903 (16)
S2—C15	1.6851 (11)	C9—C10	1.3973 (16)
O1—C1	1.2244 (16)	C10—C11	1.3885 (16)
N1—C5	1.3291 (14)	C11—C12	1.3840 (17)
N1—C9	1.4293 (14)	C11—H11	0.95
N1—H1	0.911 (18)	C12—C13	1.3856 (17)
C1—C6	1.4733 (15)	C13—C14	1.3916 (16)
C1—C2	1.5061 (16)	C13—H13	0.95
C2—C3	1.5214 (15)	C14—H14	0.95
C2—H2A	0.99	C16—C17	1.5222 (16)
C2—H2B	0.99	C16—H16A	0.99
C3—C4	1.5258 (15)	C16—H16B	0.99
C3—C8	1.5300 (16)	C17—C18	1.5315 (16)
C3—C7	1.5307 (16)	C17—H17A	0.99
C4—C5	1.5063 (15)	C17—H17B	0.99
C4—H4A	0.99	C18—C19	1.5240 (19)
C4—H4B	0.99	C18—H18A	0.99
C5—C6	1.4269 (15)	C18—H18B	0.99
C6—C15	1.4562 (15)	C19—H19A	0.98
C7—H7A	0.98	C19—H19B	0.98

C7—H7B	0.98	C19—H19C	0.98
C7—H7C	0.98		
C15—S1—C16	105.02 (5)	C14—C9—N1	120.57 (10)
C5—N1—C9	124.86 (10)	C10—C9—N1	120.21 (10)
C5—N1—H1	115.5 (11)	C11—C10—C9	120.93 (10)
C9—N1—H1	119.6 (11)	C11—C10—Cl1	118.85 (9)
O1—C1—C6	121.90 (11)	C9—C10—Cl1	120.22 (9)
O1—C1—C2	117.19 (11)	C12—C11—C10	118.49 (10)
C6—C1—C2	120.90 (10)	C12—C11—H11	120.8
C1—C2—C3	114.80 (9)	C10—C11—H11	120.8
C1—C2—H2A	108.6	C11—C12—C13	121.94 (11)
C3—C2—H2A	108.6	C11—C12—Cl2	118.20 (9)
C1—C2—H2B	108.6	C13—C12—Cl2	119.85 (9)
C3—C2—H2B	108.6	C12—C13—C14	118.85 (11)
H2A—C2—H2B	107.5	C12—C13—H13	120.6
C2—C3—C4	106.54 (9)	C14—C13—H13	120.6
C2—C3—C8	111.29 (10)	C9—C14—C13	120.55 (11)
C4—C3—C8	110.49 (10)	C9—C14—H14	119.7
C2—C3—C7	109.76 (9)	C13—C14—H14	119.7
C4—C3—C7	109.11 (9)	C6—C15—S2	125.12 (8)
C8—C3—C7	109.58 (10)	C6—C15—S1	116.90 (8)
C5—C4—C3	115.52 (9)	S2—C15—S1	117.97 (6)
C5—C4—H4A	108.4	C17—C16—S1	114.63 (8)
C3—C4—H4A	108.4	C17—C16—H16A	108.6
C5—C4—H4B	108.4	S1—C16—H16A	108.6
C3—C4—H4B	108.4	C17—C16—H16B	108.6
H4A—C4—H4B	107.5	S1—C16—H16B	108.6
N1—C5—C6	123.03 (10)	H16A—C16—H16B	107.6
N1—C5—C4	115.65 (10)	C16—C17—C18	111.18 (10)
C6—C5—C4	121.26 (10)	C16—C17—H17A	109.4
C5—C6—C15	123.74 (10)	C18—C17—H17A	109.4
C5—C6—C1	116.59 (10)	C16—C17—H17B	109.4
C15—C6—C1	119.67 (10)	C18—C17—H17B	109.4
C3—C7—H7A	109.5	H17A—C17—H17B	108.0
C3—C7—H7B	109.5	C19—C18—C17	112.52 (11)
H7A—C7—H7B	109.5	C19—C18—H18A	109.1
C3—C7—H7C	109.5	C17—C18—H18A	109.1
H7A—C7—H7C	109.5	C19—C18—H18B	109.1
H7B—C7—H7C	109.5	C17—C18—H18B	109.1
C3—C8—H8A	109.5	H18A—C18—H18B	107.8
C3—C8—H8B	109.5	C18—C19—H19A	109.5
H8A—C8—H8B	109.5	C18—C19—H19B	109.5
C3—C8—H8C	109.5	H19A—C19—H19B	109.5
H8A—C8—H8C	109.5	C18—C19—H19C	109.5
H8B—C8—H8C	109.5	H19A—C19—H19C	109.5
C14—C9—C10	119.20 (10)	H19B—C19—H19C	109.5

O1—C1—C2—C3	−149.43 (13)	C14—C9—C10—C11	−2.07 (17)
C6—C1—C2—C3	31.47 (16)	N1—C9—C10—C11	179.58 (10)
C1—C2—C3—C4	−52.05 (13)	C14—C9—C10—Cl1	178.25 (9)
C1—C2—C3—C8	68.46 (13)	N1—C9—C10—Cl1	−0.10 (15)
C1—C2—C3—C7	−170.06 (10)	C9—C10—C11—C12	0.65 (17)
C2—C3—C4—C5	52.18 (12)	Cl1—C10—C11—C12	−179.66 (9)
C8—C3—C4—C5	−68.83 (13)	C10—C11—C12—C13	1.04 (17)
C7—C3—C4—C5	170.62 (9)	C10—C11—C12—Cl2	−179.71 (9)
C9—N1—C5—C6	175.52 (10)	C11—C12—C13—C14	−1.26 (18)
C9—N1—C5—C4	−7.29 (16)	Cl2—C12—C13—C14	179.50 (9)
C3—C4—C5—N1	151.63 (10)	C10—C9—C14—C13	1.84 (17)
C3—C4—C5—C6	−31.13 (15)	N1—C9—C14—C13	−179.82 (10)
N1—C5—C6—C15	2.50 (17)	C12—C13—C14—C9	−0.21 (17)
C4—C5—C6—C15	−174.53 (10)	C5—C6—C15—S2	−0.17 (16)
N1—C5—C6—C1	−177.12 (10)	C1—C6—C15—S2	179.44 (9)
C4—C5—C6—C1	5.84 (15)	C5—C6—C15—S1	178.79 (8)
O1—C1—C6—C5	174.85 (13)	C1—C6—C15—S1	−1.60 (14)
C2—C1—C6—C5	−6.09 (16)	C16—S1—C15—C6	−175.86 (8)
O1—C1—C6—C15	−4.79 (19)	C16—S1—C15—S2	3.18 (8)
C2—C1—C6—C15	174.27 (10)	C15—S1—C16—C17	−89.34 (9)
C5—N1—C9—C14	95.10 (14)	S1—C16—C17—C18	−170.96 (8)
C5—N1—C9—C10	−86.58 (14)	C16—C17—C18—C19	−174.62 (10)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···S2	0.91 (2)	2.08 (2)	2.885 (1)	147 (2)