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(*E*)-2-(5-Chloro-2-hydroxybenzylidene)-*N*-cyclohexylhydrazine-1-carbothioamide

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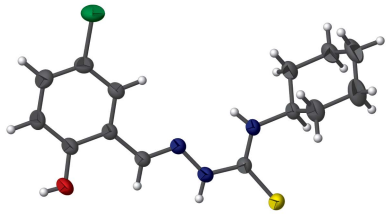
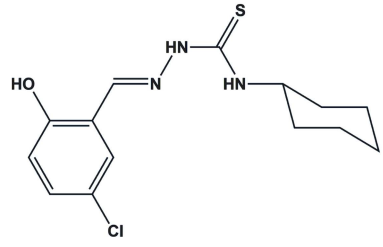
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{14}H_{18}ClN_3OS$, the phenol ring is almost coplanar with the hydrazinecarbothioamide moiety, making a dihedral angle of 6.92 (8)°. The cyclohexane ring has a chair conformation and the conformation about the C=N bond is *E*. In the crystal, molecules are linked by N—H···O and O—H···S hydrogen bonds, forming inversion dimers with an $R_2^2(14)$ ring motif flanked by two $R_2^2(6)$ ring motifs. The dimers are linked by short Cl···Cl interactions, forming layers parallel to the *ab* plane.

3D view



Chemical scheme



Structure description

In the title compound, Fig. 1, the phenol ring (C1–C6) is almost coplanar with the hydrazinecarbothioamide moiety (N1–N3/C8/S1), making a dihedral angle of 6.92 (8)°. The cyclohexane ring (C9–C14) has a chair conformation and the conformation about the C7=N1 bond is *E*. This arrangement is close to that observed in the very similar compound, (*E*)-2-(5-bromo-2-hydroxy-3-methoxybenzylidene)-N– cyclohexylhydrazine-1-carbothioamide (Jacob & Kurup, 2012).

In the crystal, molecules are linked by N—H···O and O—H···S hydrogen bonds, forming inversion dimers with an $R_2^2(14)$ ring motif flanked by two $R_2^2(6)$ ring motifs (Table 1 and Fig. 2). The dimers are linked by short Cl···Cl($-x+3, -y+1, -z+1$) contacts of 3.381 (1) Å, forming layers parallel to the *ab* plane (Fig. 2).

Synthesis and crystallization

The synthesis of the title compound is illustrated in Fig. 3. To 2-hydroxy-5-chlorobenzaldehyde (0.783 g, 5 mmol) dissolved in 20 ml of methanol, was added 0.2 ml glacial acetic acid and the mixture was refluxed for 30 min. *N*-cyclohexylhydrazinecarbothioamide (0.866 g, 5 mmol) in 20 ml methanol was added dropwise to the mixture and the

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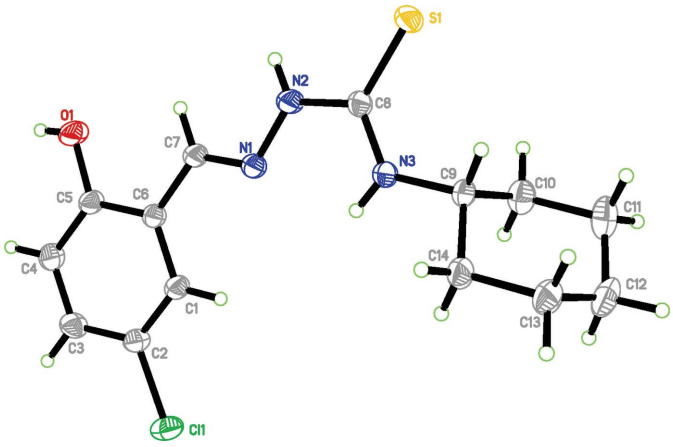

Figure 1
The molecular structure of the title compound, showing the atom labelling. Displacement ellipsoids are drawn at the 30% probability level.

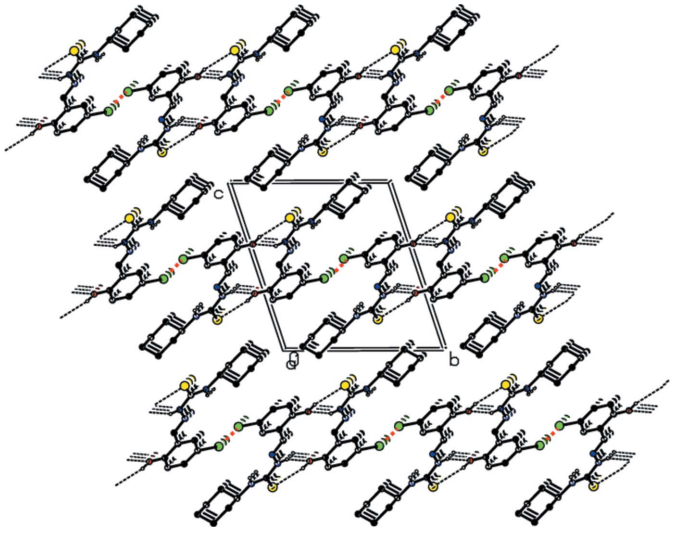

Figure 2
A view along the *a* axis of the crystal packing of the title compound. The hydrogen bonds (see Table 1) and short Cl···Cl contacts are shown as black and red dashed lines, respectively.

resulting colourless solution was refluxed for 4 h with stirring. The solution was then dried under reduced pressure overnight and the product that formed was washed with 5 ml *n*-hexane. The recovered product was dissolved in methanol for purification by recrystallization. Colourless crystals of the title compound, suitable for X-ray diffraction analysis, were obtained by slow evaporation of the solvent (yield 98%, m.p. 447–448 K).

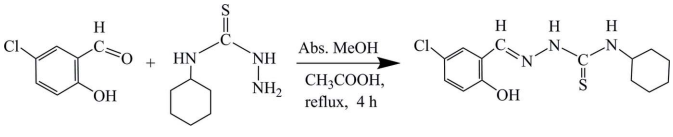

Figure 3
Synthesis of the title compound.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1*N*2···O1^{i}	0.84 (2)	2.13 (2)	2.915 (2)	156 (2)
O1—H1*O*1···S1^{i}	0.73 (3)	2.48 (3)	3.128 (2)	150 (3)

Symmetry code: (i) $-x+1, -y+2, -z+1$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{14}H_{18}ClN_3OS$
M_r	311.82
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	294
a, *b*, *c* (Å)	5.9923 (1), 11.0704 (2), 12.1128 (3)
α, β, γ (°)	107.9815 (9), 91.3414 (9), 97.2061 (9)
V (Å^3)	756.60 (3)
Z	2
Radiation type	Mo *K*α
μ (mm^{-1})	0.39
Crystal size (mm)	0.38 × 0.30 × 0.17
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2009)
T_{min}, T_{max}	0.786, 0.828
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	30649, 4824, 4127
R_{int}	0.020
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.727
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.053, 0.130, 1.12
No. of reflections	4824
No. of parameters	193
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.39, −0.22

Computer programs: *APEX2* and *SAINT* (Bruker, 2009), *SHELXS2014* (Sheldrick, 2008), *SHELXTL* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *PLATON* (Spek, 2009), and *publCIF* (Westrip, 2010).

Refinement

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

Acknowledgements

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Funding information

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The World Academy of Science

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full crystallographic data

IUCrData (2017). **2**, x161997 [https://doi.org/10.1107/S2414314616019970]

(*E*)-2-(5-Chloro-2-hydroxybenzylidene)-*N*-cyclohexylhydrazine-1-carbothioamide

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Crystal data

$C_{14}H_{18}ClN_3OS$
M_r = 311.82
Triclinic, $P\overline{1}$
a = 5.9923 (1) Å
b = 11.0704 (2) Å
c = 12.1128 (3) Å
α = 107.9815 (9)°
β = 91.3414 (9)°
γ = 97.2061 (9)°
V = 756.60 (3) Å^3
Z = 2
$F(000)$ = 328
D_x = 1.369 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9894 reflections
θ = 2.2–31.1°
μ = 0.39 mm^{-1}
T = 294 K
Block, colourless
0.38 × 0.30 × 0.17 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2009)
T_{min} = 0.786, T_{max} = 0.828
30649 measured reflections
4824 independent reflections
4127 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 31.1°, θ_{min} = 1.8°
h = −8→8
k = −16→15
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.130
S = 1.12
4824 reflections
193 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0404P)^2 + 0.4563P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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}
Cl1	1.32841 (10)	0.60273 (5)	0.57378 (5)	0.05951 (16)
S1	0.06031 (8)	0.65852 (5)	0.18924 (5)	0.04950 (15)
O1	0.8649 (3)	1.05436 (14)	0.65422 (15)	0.0584 (4)
N1	0.6106 (2)	0.73735 (13)	0.39402 (13)	0.0377 (3)
N2	0.4169 (3)	0.74695 (15)	0.33704 (14)	0.0439 (4)
N3	0.4146 (3)	0.54276 (14)	0.21687 (14)	0.0418 (3)
C1	1.0026 (3)	0.73197 (15)	0.53033 (15)	0.0380 (3)
H1A	0.9441	0.6553	0.4741	0.046*
C2	1.1981 (3)	0.73946 (17)	0.59555 (16)	0.0407 (4)
C3	1.2937 (3)	0.85327 (19)	0.67687 (17)	0.0454 (4)
H3A	1.4293	0.8577	0.7179	0.054*
C4	1.1854 (3)	0.96030 (18)	0.69645 (16)	0.0445 (4)
H4A	1.2482	1.0373	0.7510	0.053*
C5	0.9830 (3)	0.95343 (16)	0.63494 (15)	0.0391 (4)
C6	0.8924 (3)	0.83966 (15)	0.54870 (14)	0.0344 (3)
C7	0.6881 (3)	0.83640 (15)	0.47930 (15)	0.0374 (3)
H7A	0.6127	0.9078	0.4974	0.045*
C8	0.3097 (3)	0.64537 (16)	0.24909 (15)	0.0367 (3)
C9	0.3252 (3)	0.42105 (15)	0.12876 (15)	0.0374 (3)
H9A	0.2394	0.4398	0.0676	0.045*
C10	0.1685 (4)	0.3369 (2)	0.17991 (18)	0.0513 (5)
H10A	0.0424	0.3807	0.2111	0.062*
H10B	0.2486	0.3203	0.2431	0.062*
C11	0.0815 (4)	0.2101 (2)	0.0864 (2)	0.0643 (6)
H11A	−0.0138	0.1556	0.1206	0.077*
H11B	−0.0091	0.2265	0.0265	0.077*
C12	0.2755 (5)	0.14128 (19)	0.0318 (2)	0.0639 (6)
H12A	0.3584	0.1181	0.0902	0.077*
H12B	0.2162	0.0631	−0.0295	0.077*
C13	0.4329 (4)	0.22641 (19)	−0.01810 (19)	0.0550 (5)
H13A	0.3534	0.2429	−0.0815	0.066*
H13B	0.5591	0.1826	−0.0491	0.066*
C14	0.5210 (3)	0.35397 (18)	0.07495 (18)	0.0477 (4)
H14A	0.6126	0.3384	0.1349	0.057*
H14B	0.6149	0.4085	0.0402	0.057*
H1N3	0.535 (4)	0.548 (2)	0.253 (2)	0.051 (6)*
H1N2	0.351 (4)	0.812 (2)	0.361 (2)	0.053 (6)*
H1O1	0.923 (5)	1.112 (3)	0.697 (2)	0.067 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0599 (3)	0.0476 (3)	0.0755 (4)	0.0236 (2)	−0.0016 (3)	0.0198 (2)
S1	0.0411 (2)	0.0433 (2)	0.0526 (3)	0.01215 (18)	−0.01336 (19)	−0.0029 (2)
O1	0.0620 (9)	0.0332 (6)	0.0643 (9)	0.0163 (6)	−0.0218 (7)	−0.0092 (6)
N1	0.0354 (7)	0.0336 (6)	0.0385 (7)	0.0079 (5)	−0.0034 (5)	0.0025 (5)
N2	0.0418 (8)	0.0345 (7)	0.0455 (8)	0.0131 (6)	−0.0099 (6)	−0.0037 (6)
N3	0.0386 (8)	0.0326 (7)	0.0442 (8)	0.0073 (6)	−0.0103 (6)	−0.0024 (6)
C1	0.0413 (9)	0.0304 (7)	0.0391 (8)	0.0071 (6)	−0.0004 (7)	0.0056 (6)
C2	0.0425 (9)	0.0381 (8)	0.0437 (9)	0.0122 (7)	0.0023 (7)	0.0135 (7)
C3	0.0428 (9)	0.0497 (10)	0.0425 (9)	0.0089 (8)	−0.0074 (7)	0.0125 (8)
C4	0.0482 (10)	0.0399 (9)	0.0374 (9)	0.0035 (7)	−0.0102 (7)	0.0025 (7)
C5	0.0438 (9)	0.0315 (7)	0.0375 (8)	0.0080 (6)	−0.0032 (7)	0.0039 (6)
C6	0.0352 (8)	0.0315 (7)	0.0331 (7)	0.0060 (6)	−0.0014 (6)	0.0048 (6)
C7	0.0373 (8)	0.0304 (7)	0.0397 (8)	0.0079 (6)	−0.0031 (6)	0.0031 (6)
C8	0.0343 (8)	0.0337 (7)	0.0359 (8)	0.0053 (6)	−0.0025 (6)	0.0022 (6)
C9	0.0380 (8)	0.0299 (7)	0.0375 (8)	0.0031 (6)	−0.0052 (6)	0.0017 (6)
C10	0.0534 (11)	0.0471 (10)	0.0459 (10)	−0.0019 (8)	0.0056 (8)	0.0068 (8)
C11	0.0676 (14)	0.0500 (12)	0.0622 (14)	−0.0190 (10)	0.0030 (11)	0.0095 (10)
C12	0.0915 (18)	0.0315 (9)	0.0574 (13)	−0.0017 (10)	−0.0096 (12)	0.0026 (8)
C13	0.0571 (12)	0.0421 (10)	0.0514 (11)	0.0067 (8)	0.0023 (9)	−0.0060 (8)
C14	0.0416 (9)	0.0368 (8)	0.0520 (11)	0.0021 (7)	0.0031 (8)	−0.0031 (8)

Geometric parameters (Å, º)

Cl1—C2	1.7408 (18)	C6—C7	1.459 (2)
S1—C8	1.6880 (17)	C7—H7A	0.9300
O1—C5	1.360 (2)	C9—C10	1.516 (3)
O1—H1O1	0.72 (3)	C9—C14	1.520 (2)
N1—C7	1.279 (2)	C9—H9A	0.9800
N1—N2	1.369 (2)	C10—C11	1.527 (3)
N2—C8	1.363 (2)	C10—H10A	0.9700
N2—H1N2	0.84 (2)	C10—H10B	0.9700
N3—C8	1.324 (2)	C11—C12	1.522 (4)
N3—C9	1.465 (2)	C11—H11A	0.9700
N3—H1N3	0.82 (2)	C11—H11B	0.9700
C1—C2	1.377 (2)	C12—C13	1.516 (3)
C1—C6	1.395 (2)	C12—H12A	0.9700
C1—H1A	0.9300	C12—H12B	0.9700
C2—C3	1.383 (3)	C13—C14	1.532 (3)
C3—C4	1.381 (3)	C13—H13A	0.9700
C3—H3A	0.9300	C13—H13B	0.9700
C4—C5	1.390 (2)	C14—H14A	0.9700
C4—H4A	0.9300	C14—H14B	0.9700
C5—C6	1.401 (2)		
C5—O1—H1O1	112 (2)	C10—C9—C14	111.40 (16)

C7—N1—N2	115.26 (14)	N3—C9—H9A	108.3
C8—N2—N1	121.03 (14)	C10—C9—H9A	108.3
C8—N2—H1N2	117.6 (16)	C14—C9—H9A	108.3
N1—N2—H1N2	120.8 (16)	C9—C10—C11	110.06 (17)
C8—N3—C9	125.07 (15)	C9—C10—H10A	109.6
C8—N3—H1N3	116.3 (16)	C11—C10—H10A	109.6
C9—N3—H1N3	118.5 (16)	C9—C10—H10B	109.6
C2—C1—C6	119.89 (15)	C11—C10—H10B	109.6
C2—C1—H1A	120.1	H10A—C10—H10B	108.2
C6—C1—H1A	120.1	C12—C11—C10	111.10 (19)
C1—C2—C3	121.44 (16)	C12—C11—H11A	109.4
C1—C2—Cl1	119.06 (14)	C10—C11—H11A	109.4
C3—C2—Cl1	119.50 (14)	C12—C11—H11B	109.4
C4—C3—C2	119.17 (17)	C10—C11—H11B	109.4
C4—C3—H3A	120.4	H11A—C11—H11B	108.0
C2—C3—H3A	120.4	C13—C12—C11	110.60 (18)
C3—C4—C5	120.28 (16)	C13—C12—H12A	109.5
C3—C4—H4A	119.9	C11—C12—H12A	109.5
C5—C4—H4A	119.9	C13—C12—H12B	109.5
O1—C5—C4	122.65 (16)	C11—C12—H12B	109.5
O1—C5—C6	117.04 (15)	H12A—C12—H12B	108.1
C4—C5—C6	120.31 (15)	C12—C13—C14	111.10 (18)
C1—C6—C5	118.78 (15)	C12—C13—H13A	109.4
C1—C6—C7	121.50 (14)	C14—C13—H13A	109.4
C5—C6—C7	119.72 (14)	C12—C13—H13B	109.4
N1—C7—C6	121.37 (15)	C14—C13—H13B	109.4
N1—C7—H7A	119.3	H13A—C13—H13B	108.0
C6—C7—H7A	119.3	C9—C14—C13	110.17 (16)
N3—C8—N2	115.86 (15)	C9—C14—H14A	109.6
N3—C8—S1	125.09 (13)	C13—C14—H14A	109.6
N2—C8—S1	119.05 (13)	C9—C14—H14B	109.6
N3—C9—C10	111.42 (15)	C13—C14—H14B	109.6
N3—C9—C14	108.90 (14)	H14A—C14—H14B	108.1

C7—N1—N2—C8	−176.04 (17)	C5—C6—C7—N1	174.53 (17)
C6—C1—C2—C3	−2.4 (3)	C9—N3—C8—N2	176.67 (17)
C6—C1—C2—Cl1	178.29 (14)	C9—N3—C8—S1	−3.9 (3)
C1—C2—C3—C4	2.8 (3)	N1—N2—C8—N3	−5.5 (3)
Cl1—C2—C3—C4	−177.85 (16)	N1—N2—C8—S1	175.02 (14)
C2—C3—C4—C5	−0.1 (3)	C8—N3—C9—C10	−83.7 (2)
C3—C4—C5—O1	177.23 (19)	C8—N3—C9—C14	153.00 (19)
C3—C4—C5—C6	−3.0 (3)	N3—C9—C10—C11	−179.08 (18)
C2—C1—C6—C5	−0.8 (3)	C14—C9—C10—C11	−57.2 (2)
C2—C1—C6—C7	178.40 (17)	C9—C10—C11—C12	56.9 (3)
O1—C5—C6—C1	−176.81 (17)	C10—C11—C12—C13	−56.7 (3)
C4—C5—C6—C1	3.4 (3)	C11—C12—C13—C14	56.4 (3)
O1—C5—C6—C7	4.0 (3)	N3—C9—C14—C13	−179.75 (17)
C4—C5—C6—C7	−175.77 (17)	C10—C9—C14—C13	57.0 (2)

N2—N1—C7—C6	179.48 (16)	C12—C13—C14—C9	−56.3 (3)
C1—C6—C7—N1	−4.6 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1*N*2···O1^{i}	0.84 (2)	2.13 (2)	2.915 (2)	156 (2)
O1—H1*O*1···S1^{i}	0.73 (3)	2.48 (3)	3.128 (2)	150 (3)

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