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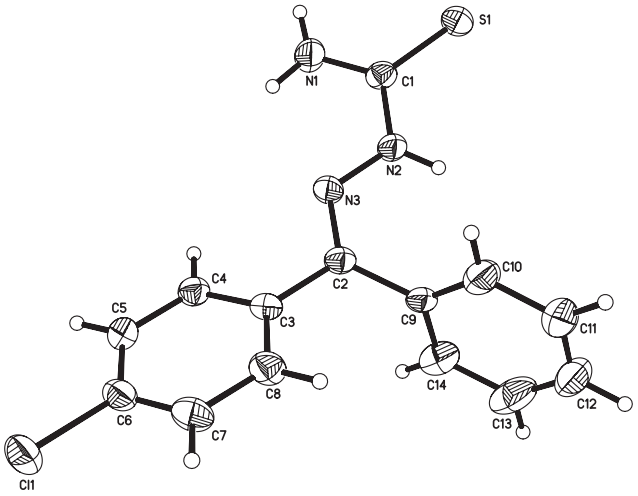
Crystal structure of (*E*)-2-((4-chlorophenyl)(phenyl)methylene)hydrazine-1-carbothioamide, $C_{14}H_{12}ClN_3S$



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Abstract

$C_{14}H_{12}ClN_3S$, monoclinic, $C2/c$ (no. 15), $a = 27.967(7)$ Å, $b = 5.9290(13)$ Å, $c = 19.698(5)$ Å, $\beta = 118.122(3)°$, $V = 2880.6(11)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0369$, $wR_{ref}(F^2) = 0.1082$, $T = 293(2)$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

All reagents and solvents were used as obtained commercially without further purification. A solution of hydrazinecarbothioamide (1 mmol, 91.1 mg) in anhydrous ethanol (10 mL) was added slowly to a solution containing (4-chlorophenyl)(phenyl)methanone (1 mmol, 216.7 mg) in 5 mL anhydrous ethanol and catalytic amounts of glacial

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.25 × 0.20 × 0.18 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.40 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7610, 2528, 0.032
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1919
$N(param)_{refined}$:	172
Programs:	Bruker [1], SHELX [2]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	x	y	z	U_{iso}*/U_{eq}
C1	0.17858(8)	0.6294(3)	0.38181(10)	0.0445(5)
C2	0.11838(8)	0.2883(3)	0.21692(10)	0.0405(5)
C3	0.13575(8)	0.1064(3)	0.18250(11)	0.0408(5)
C4	0.17680(8)	−0.0409(3)	0.22817(11)	0.0457(5)
H4	0.1932	−0.0267	0.2814	0.055*
C5	0.19379(8)	−0.2081(4)	0.19597(13)	0.0528(5)
H5	0.2210	−0.3077	0.2272	0.063*
C6	0.17002(9)	−0.2261(4)	0.11705(13)	0.0530(6)
C7	0.12979(10)	−0.0814(4)	0.07047(12)	0.0598(6)
H7	0.1142	−0.0943	0.0172	0.072*
C8	0.11249(9)	0.0839(4)	0.10318(11)	0.0532(6)
H8	0.0849	0.1814	0.0716	0.064*
C9	0.06125(8)	0.3692(3)	0.17549(11)	0.0443(5)
C10	0.04800(10)	0.5742(4)	0.13982(15)	0.0735(8)
H10	0.0751	0.6686	0.1416	0.088*
C11	−0.00543(12)	0.6422(5)	0.10108(18)	0.0981(11)
H11	−0.0141	0.7815	0.0765	0.118*
C12	−0.04521(12)	0.5083(7)	0.09853(17)	0.0954(11)
H12	−0.0812	0.5550	0.0720	0.115*
C13	−0.03279(11)	0.3060(7)	0.13455(17)	0.0930(10)
H13	−0.0602	0.2148	0.1334	0.112*
C14	0.02058(10)	0.2340(5)	0.17315(14)	0.0688(7)
H14	0.0289	0.0943	0.1975	0.083*
Cl1	0.19085(3)	−0.43752(12)	0.07611(4)	0.0824(3)
N1	0.22524(7)	0.5243(3)	0.41434(10)	0.0726(6)
H1A	0.2300	0.4051	0.3934	0.087*
H1B	0.2512	0.5746	0.4567	0.087*
N2	0.14043(6)	0.5361(3)	0.31515(9)	0.0471(4)
H2	0.1076	0.5844	0.2942	0.056*
N3	0.15454(6)	0.3656(3)	0.28168(9)	0.0431(4)
S1	0.16386(2)	0.86225(10)	0.41576(3)	0.0548(2)

acetic acid. The mixture was refluxed for 2.5 h, then cooled to room temperature. The solvent was removed to give the white product. Colourless crystals suitable for X-ray diffraction study were obtained by recrystallization from anhydrous ethanol.

Experimental details

Hydrogen atoms attached were placed geometrically and refined using a riding model approximation, with C—H = 0.93 Å, N—H = 0.86 Å and $U_{iso} = 1.2U_{eq}$ of the parent atoms.

Comment

Much attention has been paid to the studies on thiosemicarbazone compounds and their metal complexes. As important Schiff-bases they show excellent coordination properties and biological activities [3–7]. In order to search new thiosemicarbazones with higher biological activities, the title compound was synthesized and its crystal structure is reported here.

The title compound has an *E* configuration with the sulfur atom *trans* to the iminic nitrogen atom (cf. the figure), which is similar to the previous reported structure, 2-((4-fluorophenyl)(phenyl)methylene)hydrazinecarbothioamide [7]. But it is obvious different that the sulfur atoms are *trans* to the chloro atom and *cis* to the fluoro atom. In the crystal structure of title compound, the short distance *d*(N3—C2) = 1.282(2) Å has a value of a typical C=N double bond. The C1 = S1 bond distance is 1.668(2) Å, thus showing a partial double bond character. The phenyl ring and the chlorophenyl moiety are almost vertical to each other with the dihedral angle of ~89°. In the crystal structure, a hydrogen bonded dimer structure is formed by two classical N—H···S hydrogen bonds. Bond lengths and other geometric parameters are all in the expectations [8].

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