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Key indicators

Single-crystal X-ray study
T = 150 K
Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
Disorder in main residue
R factor = 0.039
wR factor = 0.047
Data-to-parameter ratio = 9.8

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Benzyl N-[1-(furan-2-yl)ethylidene]hydrazine-carbodithioate

The title compound, $\text{C}_{14}\text{H}_{12}\text{N}_2\text{S}_2\text{O}$, contains a dithiocarbazate group. The phenyl ring is disordered and perpendicular [dihedral angle of $48.0(3)^\circ$] to the rest of the molecule, which is planar.

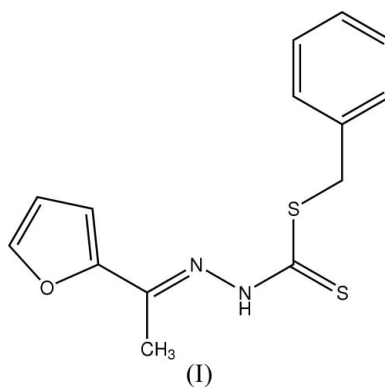
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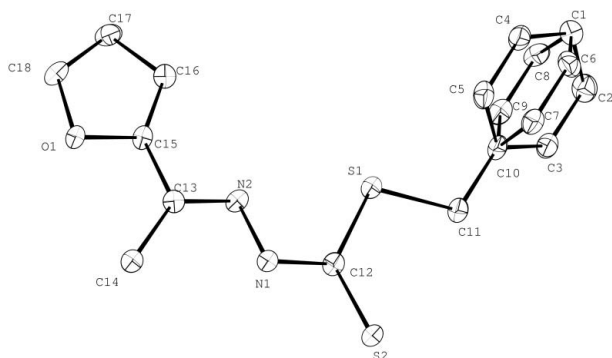
Comment

Dithiocarbazate derivatives have been widely studied and have great potential biological activity as anticancer and antimicrobial drugs (Bharti *et al.*, 2000) and in radio-pharmaceutical applications (Boschi *et al.*, 2003). This functional group is of particular interest because it is easily tuned by reaction with different aldehydes or ketones to give varied geometries for chelation to transition metals. In the structure of the title compound, (I), we were interested in studying the effect of introducing a furan ring to determine if it can also participate in chelation to a metal centre. The previously reported Cd^{II} complex of this ligand (Tarafder *et al.*, 2002b) indicated that it only forms a bis-chelating bidentate ligand without O coordination. The biological activity of the compound and its analytical characterization have also been reported (Tarafder *et al.*, 2002a).

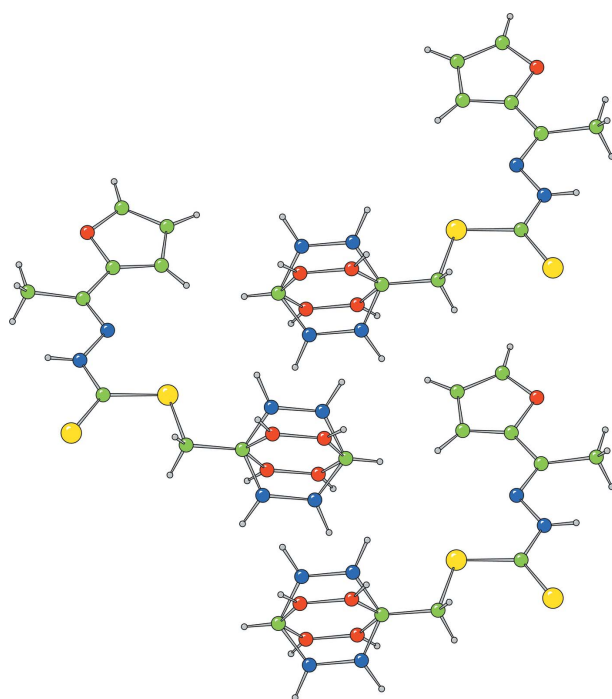


Compound (I) (Figs. 1–3) crystallizes in the unprotonated thione form with a $\text{C}=\text{S}$ bond length of $1.664(2) \text{ \AA}$, which is slightly longer than that previously reported for a dithiocarbazate Schiff base [$1.6503(17) \text{ \AA}$; Chan *et al.*, 2003]. This is in accordance with other experimental characterizations, which indicate that this type of compound forms the thione tautomer in the solid state. The formation of the Cd^{II} complex occurs through coordination at the azomethane N atom and thiolate S atom (Tarafder *et al.*, 2002b) but does not show any bond-length change: $\text{N1}-\text{N2} = 1.381(2) \text{ \AA}$ in (I).

The molecule crystallizes in the conformer in which the $\text{N1}-\text{N2}$ bond adopts a *trans* geometry with respect to $\text{C12}=\text{S2}$, while the *S*-benzyl group adopts a *cis* geometry. The


Figure 1

The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Both disorder components are shown. H atoms have been omitted.

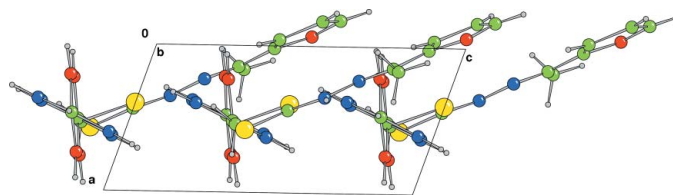

Figure 2

A projection along the *a* axis of part of the packing of (I), showing that the phenyl groups form a layer in the crystal structure. The alternative orientations of the phenyl C atoms are coloured red and blue.

furan and phenyl groups are *cis* to each other across N2/N1/C12/S1. The C13=N2 bond [1.289 (3) Å] is formed from the condensation reaction. The C14 methyl group is *cis* to the furyl O atom in this free ligand but transforms to *trans* upon chelation to Cd^{II} (Tarafer *et al.*, 2002*b*), even though the O atom does not coordinate to the metal centre. The S1–C12=S2 angle is maintained at 125.22 (12)° after coordination [125.22 (12)° in (I)].

Experimental

The Schiff base ligand was prepared according to the literature method of Tarafer *et al.* (2002*a*). *S*-Benzyldithiocarbamate (1.98 g, 0.1 mol) in absolute ethanol (40 ml) was added to an equimolar


Figure 3

A projection along the *b* axis of a section of the crystal structure of (I). The alternative orientations of the phenyl C atoms are coloured red and blue. Note that if alternate red and blue phenyl groups are selected in any layer, there are no short intermolecular clashes.

quantity of 2-furylmethylketone in absolute ethanol (50 ml). The mixture was heated over a steam bath for 10 min and then cooled to 273 K in an ice bath. The Schiff base which precipitated was filtered, washed with cold ethanol and dried *in vacuo* over silica gel, giving a dark-orange product (yield 80%, m.p 406 K). Yellow single crystals of (I), suitable for X-ray analysis, were obtained by slow evaporation of an ethanol solution over a period of three weeks.

Crystal data

C₁₄H₁₄N₂OS₂
M_r = 290.41
 Monoclinic, *P*₂₁/*c*
a = 4.7347 (1) Å
b = 32.6510 (7) Å
c = 9.3959 (2) Å
 β = 109.0305 (9)°
V = 1373.15 (5) Å³
Z = 4

D_x = 1.405 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 2559 reflections
 θ = 5–27°
 μ = 0.38 mm⁻¹
T = 150 K
 Block, yellow
 0.40 × 0.30 × 0.30 mm

Data collection

Nonius KappaCCD area-detector diffractometer
 ω scans
 Absorption correction: multi-scan (*DENZO* and *SCALEPACK*; Otwinowski & Minor, 1997)
 T_{\min} = 0.89, T_{\max} = 0.89
 5121 measured reflections

3055 independent reflections
 2036 reflections with $I > 3\sigma(I)$
 R_{int} = 0.019
 θ_{max} = 27.5°
 h = –6 → 6
 k = –38 → 42
 l = –12 → 12

Refinement

Refinement on *F*
 $R[F^2 > 2\sigma(F^2)]$ = 0.039
 $wR(F^2)$ = 0.047
 S = 1.09
 2036 reflections
 208 parameters

H-atom parameters constrained
 Chebyshev polynomial, with *A_i* coefficients 1.29, 0.798, 1.02
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}}$ = 0.37 e Å⁻³
 $\Delta\rho_{\text{min}}$ = –0.27 e Å⁻³

Table 1

Selected geometric parameters (Å, °).

C12–N1	1.343 (3)	C15–O1	1.374 (2)
C12–S1	1.749 (2)	C16–C17	1.423 (3)
C12–S2	1.664 (2)	C17–C18	1.330 (3)
C13–N2	1.289 (3)	C18–O1	1.364 (3)
C15–C16	1.344 (3)	N1–N2	1.381 (2)
N1–C12–S1	113.77 (15)	C12–N1–N2	119.35 (17)
N1–C12–S2	121.00 (16)	C12–N1–H3	119.4
S1–C12–S2	125.22 (12)	N1–N2–C13	116.37 (18)
C14–C13–N2	125.2 (2)	C15–O1–C18	106.40 (16)
C15–C13–N2	115.42 (18)	C11–S1–C12	101.75 (10)

The phenyl group was seen to be disordered. The site

occupancy factors for the two orientations refined to 0.508 (4):0.492 (4), in close agreement with the value of 0.5:0.5 which would be required by strict alternation of the two orientations in each molecular layer (Figs. 2 and 3). All H atoms (including those of the disordered phenyl group) were located in a difference map, but those attached to C atoms were repositioned geometrically. All H atoms were initially refined with soft restraints on the bond lengths and angles to regularize their geometry (C–H in the range 0.93–98 and N–H = 0.85 Å) and isotropic atomic displacement parameters [$U_{\text{iso}}(\text{H})$ in the range 1.2–1.5 times U_{eq} of the parent atom], after which they were refined with riding constraints.

Data collection: *COLLECT* (Nonius, 2001); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *CAMERON* (Watkin *et al.*, 1996); software used to prepare material for publication: *CRYSTALS*.

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