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10,11-Dihydrodibenzo[*b*,*f*]azepine-5-carbothioic *S*-acid

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.050
wR factor = 0.123
Data-to-parameter ratio = 13.2

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

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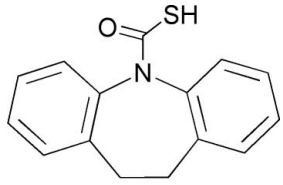
In the title molecule, $C_{15}H_{13}NOS$, the bond lengths and angles show normal values. No classical hydrogen bonds are found in the crystal packing, which is stabilized by van der Waals forces.

Comment

Molecules containing a modified azepine ring encompass a wide range of biological activities and are known to be anti-depressants (Syeda *et al.*, 2005) and to exhibit anticancer activity (Link & Kunick, 1998). Recently, the photochemistry of a series of *N*-substituted dibenzo[*b*,*f*]azepines has been studied (Querner *et al.*, 2004).



(I)

In the title molecule, (I), (Fig. 1), the bond lengths and angles show normal values (Allen *et al.*, 1987). The mean planes formed by the atoms C1–C7/N1 [r.m.s. deviation of 0.0219 (2) Å] and C7–C14/N1 [r.m.s. deviation of 0.1972 (3) Å] make a dihedral angle of 61.0 (2)°. The crystal packing (Fig. 2) demonstrates no classical hydrogen bonds and is mainly stabilized by van der Waals forces.

Experimental

Sulfur (30 mmol, 0.96 g) and Se powder (0.05 g) were added to a solution of 10,11-dihydro-5*H*-dibenzo[*b*,*f*]azepine (10 mmol, 1.95 g)

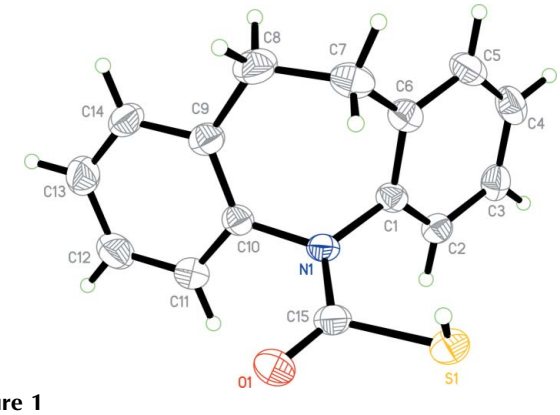


Figure 1
View of the title compound, showing the atom numbering and displacement ellipsoids drawn at the 30% probability level.

in chlorobenzene (20 ml) at 373 K; carbon monoxide was introduced (0.2 ml min^{-1}) over a period of 12 h. After cooling, a white solid was collected, treated with NaOH solution (*w*/*w* 10%, 10 ml) and filtered. The aqueous solution was neutralized with dilute HCl to pH 8, giving the title compound in 41% yield. Single crystals suitable for X-ray data collection were obtained by slow evaporation of a solution in ethanol.

Crystal data

$C_{15}H_{13}NOS$
M_r = 255.32
Orthorhombic, $Pca2_1$
a = 16.6550 (15) Å
b = 10.6438 (10) Å
c = 7.2559 (7) Å
V = 1286.3 (2) $Å^3$
Z = 4
D_x = 1.318 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 298 (2) K
Block, colourless
0.25 × 0.15 × 0.13 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.943, T_{max} = 0.968
6593 measured reflections
2189 independent reflections
2094 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 25.2°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.123
S = 1.18
2189 reflections
166 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0605P)^2 + 0.2276P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e $Å^{-3}$
$\Delta\rho_{min}$ = −0.22 e $Å^{-3}$
Absolute structure: Flack (1983), 2769 Friedel pairs
Flack parameter: −0.06 (13)

The S-bound H atom was located in a difference map and refined with the bond restraint S—H = 1.10 (3) Å and U_{iso}(H) = 1.2U_{eq}(S). The remaining H atoms were positioned geometrically (C—H = 0.93–0.96 Å) and refined as riding, with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXL97*.

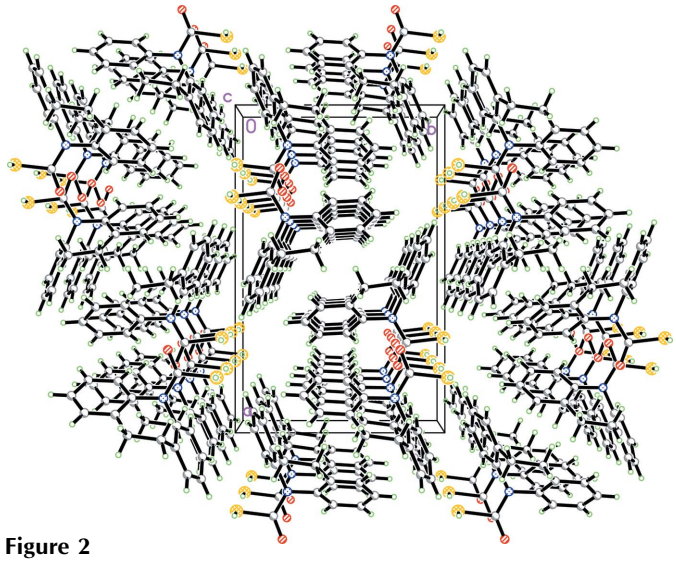


Figure 2
A perspective view of the crystal packing.

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