

Lists of structure factors, anisotropic displacement parameters, bond distances and angles involving H atoms and selected torsion angles have been deposited with the IUCr (Reference: AB1116). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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N-Phenyl-2-phenyliminotetrahydro-1,3-thiazole-2-carbothioamide, C₁₆H₁₅N₃S₂

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Abstract

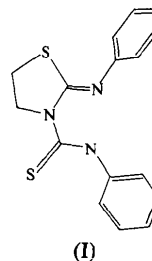
The thiazolidine ring in the title compound has an envelope conformation, *E*₄, with a C atom out of the plane of the ring. The C—S and C—N bond lengths clearly show the typical resonance of the thiourea group. The molecules are linked by van der Waals forces and an intramolecular N···N hydrogen bond is present.

Comment

A large number of thiazole or thiazine derivatives may be obtained by the reaction of haloalkylisothiocyanates with amines. Compounds containing the thiazoline or thiazine structural fragment have biological properties as potential herbicides and antifungal agents (Behner & Stendel, 1971; Sumitomo

Chemical Co. Ltd, 1982). Some of them also display medical properties (Johnson, Boukma & Kim, 1970).

The two phenyl groups in the title compound, (I), are planar (the maximum deviation from the least-squares plane is 0.007 Å). Bond lengths and angles are normal. The thiourea group is also planar (the maximum deviation from the least-squares plane is 0.006 Å). The S—C distance of 1.661 (2) Å and the C—N distances of 1.390 (3) and 1.344 (3) Å clearly show the resonance of the thiourea system (Kunchur & Truter, 1958; Valle, Cojazzi, Busetti & Mammi, 1970).



The thiazolidine ring is not planar, and in terms of the ring-puckering coordinates (Cremer & Pople, 1975) the amplitude and phase magnitudes are *Q* = 0.353 (3) Å and *φ* = −33.6 (4)° for the sequence of atoms S1, C2, N3, C4 and C5. The conformation of the ring is an envelope with C5 lying 0.316 (3) Å out of the least-squares plane of the ring. However, Nardelli (1983*a*) asymmetry parameters (Table 3) show a departure from ideal *E* symmetry. The substituent atoms C31 and N21 are −0.143 (2) and 0.296 (2) Å from the least-squares plane. Bond distances and angles agree well with those found for a

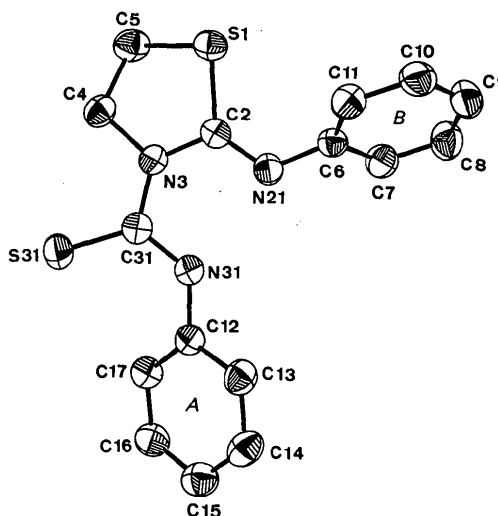


Fig. 1. An ORTEPII (Johnson, 1976) view of the molecule viewed down the *c* axis, showing the atomic numbering scheme. Displacement ellipsoids are shown at the 50% probability level.

planar thiazolidine ring (Rasmussen, Villani & Griffin, 1984).

The dihedral angles between the least-squares planes of the different groups are: phenyl ring *A*–phenyl ring *B* 66.0 (1)°, phenyl ring *A*–thiourea 54.1 (1)°, phenyl ring *A*–thiazolidine ring 56.0 (1)°, phenyl ring *B*–thiourea 38.1 (1)°, phenyl ring *B*–thiazolidine ring 21.5 (1)° and thiourea–thiazolidine ring 16.7 (1)°. The packing of the molecules is governed by van der Waals forces; there is an intramolecular hydrogen bond, N31...N21 = 2.596 (3) Å, N31—H...N21 = 138°.

Experimental

The title compound was obtained by the reaction of 2-phenylamino-2-thiazoline with phenyl isothiocyanate. Crystals suitable for data collection were grown from a benzene/methanol solution.

Crystal data

C₁₆H₁₅N₃S₂
M_r = 313.43
 Orthorhombic
*P*2₁2₁2₁
a = 11.040 (1) Å
b = 18.373 (1) Å
c = 7.474 (1) Å
V = 1516.0 (3) Å³
Z = 4
D_x = 1.36 Mg m^{−3}
D_m = 1.37 Mg m^{−3}
D_m measured by flotation in
 bromobenzene/ethanol

Data collection

Enraf–Nonius CAD-4
 diffractometer
 $\omega/2\theta$ scans
 Absorption correction:
 empirical (Walker &
 Stuart, 1983)
T_{min} = 0.655, *T_{max}* =
 1.360
 2515 measured reflections
 2515 independent reflections
 2394 observed reflections
 [*I* ≥ 3σ(*I*)]

Refinement

Refinement on *F*
R = 0.032
wR = 0.033
S = 1.67
 2394 reflections
 190 parameters
 H-atom parameters not
 refined
 Unit weights applied

Mo *K*α radiation
 λ = 0.71069 Å
 Cell parameters from 25
 reflections
 θ = 2–30°
 μ = 0.332 mm^{−1}
T = 293 K
 Prism
 0.5 × 0.4 × 0.3 mm
 Colourless

$\theta_{\max}$ = 20°
h = 0 → 15
k = 0 → 25
l = 0 → 7
 3 standard reflections
 monitored every 100
 reflections
 intensity variation:
 insignificant

(Δ/σ)_{max} = 0.004
 $\Delta\rho_{\max}$ = 0.2 e Å^{−3}
 $\Delta\rho_{\min}$ = −0.2 e Å^{−3}
 Extinction correction: none
 Atomic scattering factors
 from *International Tables*
 for *X-ray Crystallography*
 (1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j.$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i>
S1	0.92022 (7)	0.17050 (3)	0.95164 (9)	0.0413 (2)
S31	0.64341 (6)	−0.05540 (4)	0.91507 (12)	0.0486 (2)
N3	0.8163 (2)	0.0440 (1)	0.9032 (3)	0.0338 (6)
N21	1.0266 (2)	0.0367 (1)	0.9473 (3)	0.0392 (6)
N31	0.8838 (2)	−0.0746 (1)	0.9103 (3)	0.0388 (6)
C2	0.9305 (2)	0.0749 (1)	0.9342 (3)	0.0342 (6)
C4	0.7172 (2)	0.0972 (1)	0.8937 (4)	0.0422 (8)
C5	0.7736 (3)	0.1695 (2)	0.8462 (4)	0.0507 (9)
C6	1.1413 (2)	0.0673 (1)	0.9842 (3)	0.0355 (7)
C7	1.2382 (3)	0.0445 (2)	0.8817 (4)	0.0466 (8)
C8	1.3538 (3)	0.0691 (2)	0.9179 (5)	0.0568 (10)
C9	1.3730 (3)	0.1165 (2)	1.0607 (5)	0.0579 (10)
C10	1.2776 (3)	0.1384 (2)	1.1632 (5)	0.0520 (10)
C11	1.1608 (3)	0.1139 (2)	1.1279 (4)	0.0436 (8)
C12	0.8857 (2)	−0.1514 (1)	0.9199 (3)	0.0341 (6)
C13	0.9780 (3)	−0.1829 (2)	1.0201 (4)	0.0453 (8)
C14	0.9876 (3)	−0.2587 (2)	1.0298 (4)	0.0522 (9)
C15	0.9069 (3)	−0.3019 (1)	0.9396 (4)	0.0492 (9)
C16	0.8166 (3)	−0.2706 (2)	0.8383 (4)	0.0458 (8)
C17	0.8048 (3)	−0.1956 (1)	0.8282 (4)	0.0390 (7)
C31	0.7876 (2)	−0.0297 (1)	0.9084 (3)	0.0344 (6)

Table 2. Selected geometric parameters (Å, °)

S1—C2	1.764 (2)	S31—C31	1.661 (2)
S1—C5	1.800 (3)	N3—C4	1.468 (3)
N3—C2	1.402 (3)	N21—C2	1.276 (3)
N3—C31	1.390 (3)	N31—C12	1.414 (3)
N21—C6	1.413 (3)	C4—C5	1.509 (4)
N31—C31	1.344 (3)	C6—C11	1.389 (3)
C6—C7	1.381 (3)	C8—C9	1.393 (5)
C7—C8	1.381 (4)	C10—C11	1.391 (4)
C9—C10	1.363 (4)	C12—C17	1.388 (3)
C12—C13	1.389 (3)	C14—C15	1.371 (4)
C13—C14	1.398 (4)	C16—C17	1.387 (3)
C15—C16	1.377 (4)		
C2—S1—C5	90.8 (1)	C4—N3—C31	118.7 (2)
C2—N3—C31	126.4 (2)	C2—N3—C4	114.0 (1)
C2—N21—C6	122.7 (2)	C12—N31—C31	128.6 (2)
N3—C2—N21	122.5 (2)	S1—C2—N21	126.5 (1)
S1—C2—N3	110.9 (1)	N3—C4—C5	106.9 (2)
S1—C5—C4	106.0 (2)	N21—C6—C11	122.3 (2)
N21—C6—C7	117.6 (2)	C7—C6—C11	119.7 (2)
C6—C7—C8	120.4 (2)	C7—C8—C9	119.6 (2)
C8—C9—C10	119.8 (3)	C9—C10—C11	120.9 (3)
C6—C11—C10	119.2 (2)	N31—C12—C17	123.2 (3)
N31—C12—C13	116.9 (2)	C13—C12—C17	119.6 (2)
C12—C13—C14	119.8 (2)	C13—C14—C15	120.1 (2)
C14—C15—C16	119.8 (2)	C15—C16—C17	120.9 (2)
C12—C17—C16	119.5 (2)	N3—C31—N31	114.6 (2)
S31—C31—N31	125.6 (1)	S31—C31—N3	119.6 (1)

Table 3. Asymmetry parameters for the thiazolidine ring

ΔC_1 (S1)	0.1511 (15)	ΔC_2 (S1)	0.1583 (10)
ΔC_2 (C2)	0.2472 (14)	ΔC_2 (C2)	0.0588 (10)
ΔC_3 (N3)	0.2488 (13)	ΔC_2 (N3)	0.0580 (10)
ΔC_4 (C4)	0.1553 (12)	ΔC_2 (C4)	0.1522 (11)
ΔC_5 (C5)	0.0118 (13)	ΔC_2 (C5)	0.1893 (10)

Preliminary Weissenberg photographs indicated that the crystal belonged to the orthorhombic system with systematic absences *h*00 *h* odd, 0*k*0 *k* odd, 00*l* *l* odd, consistent with *P*2₁2₁2₁. Corrections were made for Lorentz–polarization effects. The structure was solved by direct methods using *MULTAN*80 (Main *et al.*, 1980). The H atoms were assigned the same isotropic displacement parameters as the atoms to which they were bonded. All calculations were carried out using the crystallographic programs of the *XRAY70* System (Stewart,

Kundell & Baldwin, 1970). Bond lengths and angles were calculated using *PARST* (Nardelli, 1983*b*).

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates, and a plot of the crystal packing have been deposited with the IUCr (Reference: AB1103). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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