

Phenyl 2-thioxo-1,3-thiazolidine-3-carboxylate

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Key indicators

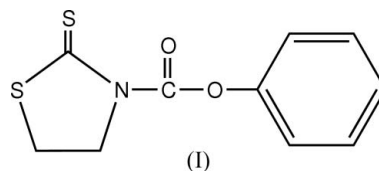
Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.009$ Å
 R factor = 0.068
 wR factor = 0.220
Data-to-parameter ratio = 18.4For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The title compound, $\text{C}_{10}\text{H}_9\text{NO}_2\text{S}_2$, was prepared from a condensation reaction of phenyl chloroformate and 1,3-thiazolidine-2-thione in the presence of triethylamine. In the crystal structure, molecules exist as the thione tautomer.

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Comment

1,3-Thiazolidine-2-thione derivatives have a high potential for biological activity, and these derivatives have been widely used in agrochemical fungicides (Takashi *et al.*, 1997). In addition, 3-acyl-1,3-thiazolidine-2-thiones can be used as active amides for peptide synthesis (Li *et al.*, 1981). In order to investigate the structure–activity and structure–property relationships, a series of new 1,3-thiazolidine-2-thione derivatives has been synthesized in our laboratory. We report here the structure of the title compound, (I), an early result in our study of this series of compounds.



The molecular structure of (I) is illustrated in Fig. 1. Selected bond lengths and angles are listed in Table 1. In solution, 1,3-thiazolidine-2-thione exists in tautomeric equilibrium with its thiol form (Atzei *et al.*, 2001). However, only the thione form is observed in the crystal structure of (I).

Experimental

1,3-Thiazolidine-2-thione (0.60 g, 5 mmol), prepared according to the procedure of Owen (1967), and triethylamine (0.62 g, 6 mmol) were dissolved in dichloromethane (15 ml) with stirring. Phenyl chloroformate (0.95 g, 6 mmol) was added dropwise to the mixture in an ice bath. The mixture was stirred at 273 K for 6 h and then dried *in vacuo* to give a yellow solid of (I) (1.15 g, yield 96.2%), which was then recrystallized from ethanol by slow evaporation to give yellow blocks (m.p. 386–388 K).

Crystal data

$\text{C}_{10}\text{H}_9\text{NO}_2\text{S}_2$
 $M_r = 239.31$
Monoclinic, $P2_1/c$
 $a = 11.5465$ (6) Å
 $b = 8.9066$ (4) Å
 $c = 11.9102$ (6) Å
 $\beta = 116.112$ (2)°
 $V = 1099.83$ (10) Å³
 $Z = 4$

$D_x = 1.445$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 7221
reflections
 $\theta = 2.3$ – 27.4 °
 $\mu = 0.46$ mm⁻¹
 $T = 293$ (1) K
Block, yellow
0.50 × 0.50 × 0.40 mm

Data collection

Rigaku R-Axis RAPID
diffractometer
 ω scans
Absorption correction: multi-scan
(*ABSCOR*; Higashi, 1995)
 $T_{\min} = 0.772$, $T_{\max} = 0.831$
9973 measured reflections

2505 independent reflections
1537 reflections with $F^2 > 2\sigma(F^2)$
 $R_{\text{int}} = 0.027$
 $\theta_{\text{max}} = 27.4^\circ$
 $h = -14 \rightarrow 14$
 $k = -11 \rightarrow 11$
 $l = -15 \rightarrow 15$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.068$
 $wR(F^2) = 0.220$
 $S = 1.00$
2505 reflections
136 parameters

H-atom parameters constrained
 $w = 4F_o^2/[0.0101F_o^2 + \sigma(F_o^2)]$
 $(\Delta/\sigma)_{\text{max}} = 0.004$
 $\Delta\rho_{\text{max}} = 0.35 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.41 \text{ e } \text{\AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

S1—C3	1.641 (4)	O2—C5	1.407 (5)
S2—C1	1.750 (8)	N1—C2	1.485 (5)
S2—C3	1.725 (4)	N1—C3	1.371 (6)
O1—C4	1.177 (5)	N1—C4	1.403 (5)
O2—C4	1.338 (5)		
S1—C3—S2	118.9 (3)	S2—C3—N1	111.5 (3)
S1—C3—N1	129.5 (3)	C3—N1—C2	115.1 (3)
C3—S2—C1	93.2 (3)	N1—C2—C1	107.7 (6)
S2—C1—C2	111.2 (5)		
C1—S2—C3—N1	−3.1 (3)	C2—N1—C3—S2	−3.9 (4)
C5—O2—C4—N1	176.9 (3)	C3—N1—C4—O1	8.6 (7)
C4—O2—C5—C6	103.7 (5)		

All H atoms were placed in idealized positions and allowed to ride on their parent atoms ($\text{C—H} = 0.97 \text{ \AA}$), with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSK, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1993); program(s) used to refine structure: *CRYSTALS* (Better-

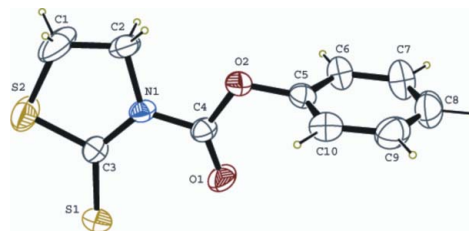


Figure 1

The molecular structure of (I), showing 30% probability displacement ellipsoids.

idge *et al.*, 2003); molecular graphics: *ORTEP3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

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References

Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. (1993). *J. Appl. Cryst.* **26**, 343–350.
Atzei, D., Filippo, D. D., Rossi, A. & Porcelli, M. (2001). *Spectrosc. Acta Sect. A*, **57**, 1073–1083.
Betteridge, P. W., Carruthers, J. R., Cooper, R. I., Prout, K. & Watkin, D. J. (2003). *J. Appl. Cryst.* **36**, 1487.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Li, C., Yieh, Y., Lin, Y., Lu, Y., Chi, A. & Hsing, C. (1981). *Tetrahedron Lett.* **36**, 3467–3470.
Owen, T. C. (1967). *J. Chem. Soc.* **15**, 1373–1376.
Rigaku (1998). *PROCESS-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSK (2004). *CrystalStructure*. Version 3.6.0. Rigaku/MSK, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA.
Takashi, J., Nakaya, M., Inami, S. & Matsuno, H. (1997). JP Patent No. 09 124 647.