

Fig. 2. The molecular packing. (a) Projection down  $b$ . (b) Projection perpendicular to the (001) plane. Short intermolecular contacts ( $\text{\AA}$ ) within the layers of molecules are given. The upper number in each case is the  $\text{O}\cdots\text{H}$  or  $\text{N}\cdots\text{H}$  distance. The lower (underlined) number is the corresponding  $\text{O}\cdots\text{C}$  or  $\text{N}\cdots\text{C}$  distance. Molecules in the next two layers are shown in the lower right-hand corner of (b).

The same situation occurs here. The molecules occur in layers at  $z \sim \frac{1}{4}$ . The two sides of the layer make different contacts; the distance between layers is  $3.37 \text{ \AA}$  from  $z \sim \frac{1}{4}$  to  $z \sim \frac{3}{4}$ , and  $3.46 \text{ \AA}$  and  $z \sim \frac{1}{4}$  to  $z \sim -\frac{1}{4}$ . Individual molecules are tilted  $10^\circ$  with respect to the plane of the layers; this leads to corresponding perpendicular distances between the planes of the molecules of  $3.39$  and  $3.47 \text{ \AA}$ . The two kinds of contact between layers can be seen in the lower right-hand corner of Fig. 2. Within a layer all the close contacts are either  $\text{O}\cdots\text{H}$  or  $\text{N}\cdots\text{H}$ . The distances, however, are not unusually short, so that we cannot describe these as  $\text{O}\cdots\text{H}-\text{C}$  or  $\text{N}\cdots\text{H}-\text{C}$  hydrogen bonds. Perhaps a satisfactory description would be to say they are incipient hydrogen bonds; the small polarity of the  $\text{C}-\text{H}$  bond leads to enough interaction to determine the packing because the packing in layers is reasonably efficient in this case.

We thank the Graduate School and the University Computer Center of the University of Minnesota for their support of this work.

#### References

- BRITTON, D. & NOLAND, W. E. (1972). *Acta Cryst.* **B28**, 1116–1121.  
 CHOW, Y. M. & BRITTON, D. (1974). *Acta Cryst.* **B30**, 147–151.  
 GEHRZ, R. C. & BRITTON, D. (1972). *Acta Cryst.* **B28**, 1126–1130.  
 GOL'DER, G. A., TODRES-SELEKTOR, Z. V. & BOGDANOV, S. V. (1961). *Zh. Strukt. Khim.* **2**, 478–479.  
 HULME, R. (1962). *Chem. Ind. (London)*, pp. 42–43.  
 SCHOMAKER, V. & TRUEBLOOD, K. N. (1968). *Acta Cryst.* **B24**, 63–76.

*Acta Cryst.* (1979). **B35**, 3078–3080

### 3-Phenyl-1,3-thiazolidine-2,4-dione

BY S. STANKOVIĆ

*Institute of Physics, University of Novi Sad, Dr. Ilije Djuricića 4, 21000 Novi Sad, Yugoslavia*

AND G. D. ANDREETTI

*Istituto di Strutturistica Chimica dell'Università di Parma, Centro di Studio per la Strutturistica Diffraattometrica del CNR, 43100 Parma, Italy*

(Received 22 November 1978; accepted 26 June 1979)

**Abstract.**  $\text{C}_9\text{H}_7\text{NO}_2\text{S}$ , monoclinic, space group  $C2/c$ ,  $a = 25.77(2)$ ,  $b = 4.43(1)$ ,  $c = 12.73(3) \text{ \AA}$ ,  $\beta = 107.4(1)^\circ$ ,  $V = 1700 \text{ \AA}^3$ ,  $Z = 8$ ,  $M_r = 193.22$ ,  $D_m = 1.49$  (floatation),  $D_c = 1.51 \text{ Mg m}^{-3}$ ,  $\mu(\text{Cu } K\alpha) = 2.89 \text{ mm}^{-1}$ . The structure has been determined by direct

methods and refined by full-matrix least squares to an  $R$  of 0.056. The thiazolidine ring is slightly non-planar.

**Introduction.** The compound was obtained by Professor Pergal of the Institute of Chemistry, University

of Novi Sad (Pergal, Popov-Pergal & Kobilarov, 1976). Preliminary cell dimensions were obtained from Weissenberg photographs (Lazar, Ribár, Popov-Pergal & Pergal, 1976). The systematic absences indicated space groups *Cc* or *C2/c*. The statistical distribution of intensities was according to the centrosymmetric space group *C2/c* and this was confirmed by the structure analysis. A crystal  $0.07 \times 0.71 \times 0.26$  mm was used for data collection. The cell parameters were obtained by least squares from the setting of twenty reflections measured on a Siemens AED single-crystal diffractometer using Cu  $K\alpha$  radiation ( $\lambda = 1.54178$  Å). For intensity measurements the  $\theta$ - $2\theta$  scan method was used with stationary background counts taken before and after the scan. No systematic decline of reference reflections was observed. 1692 independent reflections up to  $\theta = 70^\circ$  were collected and 1678 of them were considered observed [ $I > 2\sigma(I)$ ] and included in the refinement. The intensity data were reduced to structure factors by the application of Lorentz and polarization factors, but no correction for absorption was applied. The structure was determined by direct methods with *SHELX* (Sheldrick, 1976). The *E* map gave the positions for all non-hydrogen atoms. All hydrogen atoms were found in the difference Fourier map. The refinement was carried out by full-matrix least squares, refining isotropically the H and aniso-

Table 1. Fractional coordinates ( $\times 10^4$ ) for the non-hydrogen atoms with estimated standard deviations in parentheses

	x	y	z
S	4705.9 (4)	3579 (1)	11072.1 (4)
O(1)	3598 (1)	8468 (3)	9952 (1)
O(2)	4413 (1)	1589 (3)	9093 (1)
N	3942 (1)	5036 (3)	9352 (1)
C(1)	4381 (1)	6452 (4)	11118 (2)
C(2)	3926 (1)	6830 (4)	10120 (2)
C(3)	4333 (1)	3202 (3)	9678 (2)
C(4)	3571 (1)	5072 (3)	8253 (1)
C(5)	3602 (1)	6988 (4)	7558 (2)
C(6)	3240 (1)	7002 (4)	6496 (2)
C(7)	2864 (1)	5138 (4)	6166 (2)
C(8)	2835 (1)	3247 (4)	6872 (2)
C(9)	3196 (1)	3199 (4)	7931 (2)

Table 2. Fractional coordinates ( $\times 10^3$ ) for the hydrogen atoms

	x	y	z
H(1)	460 (1)	794 (6)	1128 (2)
H(2)	422 (1)	648 (5)	1174 (3)
H(3)	387 (1)	834 (4)	784 (2)
H(4)	328 (1)	824 (4)	604 (3)
H(5)	262 (1)	509 (5)	545 (3)
H(6)	259 (1)	206 (5)	673 (2)
H(7)	317 (1)	187 (5)	838 (2)

tropically the non-hydrogen atoms. The final *R* was 0.056 [ $R_w = (\sum w\Delta^2 / \sum w|F_o|^2)^{1/2} = 0.067$ ] for the observed reflections. The weights were given by  $w = k/[\sigma^2(F_o) + 0.005F_o^2]$ , where *k* refined to 0.2919. The final parameters for the non-hydrogen atoms are given in Table 1 and for hydrogen atoms in Table 2. Bond distances and angles with their standard deviations, derived from the full variance-covariance matrix, are given in Table 3.\*

**Discussion.** A view of the molecule is shown in Fig. 1. The thiazolidine ring is slightly non-planar although in the related molecule, 1,3-thiazolidine-2,4-dione (Form, Raper & Downie, 1975), the ring shows essential

\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34562 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. Interatomic distances (Å) and bond angles ( $^\circ$ ) with their e.s.d.'s in parentheses

S—C(1)	1.797 (2)	C(7)—C(8)	1.381 (3)
S—C(3)	1.759 (2)	C(8)—C(9)	1.390 (3)
O(1)—C(2)	1.201 (2)	C(9)—C(4)	1.378 (2)
O(2)—C(3)	1.207 (2)	C(1)—H(1)	0.97 (3)
C(1)—C(2)	1.521 (3)	C(1)—H(2)	0.91 (3)
N—C(2)	1.389 (2)	C(5)—H(3)	1.00 (2)
N—C(3)	1.389 (2)	C(6)—H(4)	0.91 (3)
N—C(4)	1.439 (2)	C(7)—H(5)	0.95 (3)
C(4)—C(5)	1.383 (2)	C(8)—H(6)	0.88 (3)
C(5)—C(6)	1.394 (3)	C(9)—H(7)	0.94 (3)
C(6)—C(7)	1.377 (3)		
C(1)—S—C(3)	93.2 (1)	C(3)—N—C(4)	120.8 (1)
S—C(1)—C(2)	107.3 (1)	N—C(4)—C(5)	119.0 (1)
C(1)—C(2)—N	110.9 (2)	N—C(4)—C(9)	118.8 (1)
O(1)—C(2)—C(1)	124.9 (2)	C(5)—C(4)—C(9)	122.3 (2)
O(1)—C(2)—N	124.1 (2)	C(4)—C(5)—C(6)	118.3 (2)
S—C(3)—N	110.3 (1)	C(5)—C(6)—C(7)	119.8 (2)
O(2)—C(3)—S	124.4 (1)	C(6)—C(7)—C(8)	121.2 (2)
O(2)—C(3)—N	125.4 (2)	C(7)—C(8)—C(9)	119.6 (2)
C(2)—N—C(3)	117.6 (2)	C(8)—C(9)—C(4)	118.7 (2)
C(2)—N—C(4)	121.6 (1)		

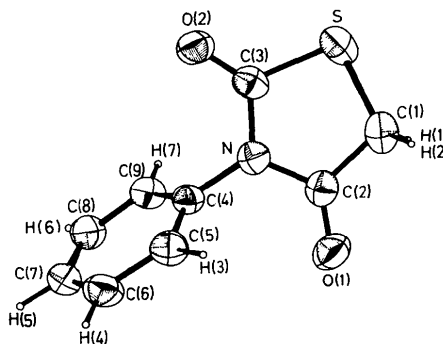


Fig. 1. ORTEP drawing of the molecule showing atomic numbering (Johnson, 1965). The 50% probability ellipsoids are shown.

planarity. The least-squares plane through S, N, C(1), C(2), C(3), O(1), O(2) and C(4) is  $0.6423X + 0.5645Y - 0.5184Z = 3.1698$ .† The atoms C(1) and S show the greatest deviations from the least-squares plane [0.085 (4) and  $-0.076$  (2) Å, respectively]. The phenyl ring is essentially planar. The least-squares plane through C(4), C(5), C(6), C(7), C(8) and C(9) is:  $-0.6515X + 0.5272Y + 0.5456Z = -0.0293$ . The dihedral angle formed by the two rings is  $113.8^\circ$ . Contacts are consistent with van der Waals radii assuming for S a radius of  $1.72\text{--}1.73$  Å as pointed out by several authors (van der Helm, Lessor & Merritt, 1962; Cavalca, Gaetani, Mangia & Pelizzi, 1970; Fava Gasparri, Nardelli & Villa, 1967).

The authors are greatly indebted to Professors B. Ribár, L. Cavalca and M. Nardelli for their interest, to Dr Gy. Argay for some computational work and to

† The coordinates (in Å)  $X$ ,  $Y$ ,  $Z$  are referred to the orthogonal axes  $x, y, z^*$ .

Professor M. Pergal who supplied the crystals of the compound.

#### References

- CAVALCA, L., GAETANI, A., MANGIA, A. & PELIZZI, G. C. (1970). *Gazz. Chim. Ital.* **100**, 629–638.  
 FAVA GASPARRI, G., NARDELLI, M. & VILLA, A. (1967). *Acta Cryst.* **23**, 384–391.  
 FORM, G. R., RAPER, E. S. & DOWNIE, T. C. (1975). *Acta Cryst.* **B31**, 2181–2184.  
 JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.  
 LAZAR, D., RIBÁR, B., POPOV-PERGAL, K. & PERGAL, M. (1976). *Z. Kristallogr.* **144**, 144.  
 PERGAL, M., POPOV-PERGAL, K. & KOBILAROV, N. (1976). *Synthesis*, **3**, 190–191.  
 SHELDRIK, G. M. (1976). *The SHELX System of Crystallographic Computer Programs*. Univ. of Cambridge, England.  
 VAN DER HELM, D., LESSOR, A. E. & MERRITT, L. L. (1962). *Acta Cryst.* **15**, 1227–1232.

*Acta Cryst.* (1979). **B35**, 3080–3082

## 2,5-Di-*tert*-butyl-1,2,5-thiadiazolidine-3,4-dione

BY M. SCHERZ AND J. WEISS

*Anorganisch-Chemisches Institut der Universität Heidelberg, Im Neuenheimer Feld 270, D 6900 Heidelberg 1, Federal Republic of Germany*

(Received 17 April 1979; accepted 13 August 1979)

**Abstract.**  $C_{10}H_{18}N_2O_2S$ , orthorhombic, *Pbca*,  $a = 11.922$  (4),  $b = 13.483$  (4),  $c = 15.431$  (12) Å,  $V = 2480$  Å<sup>3</sup>,  $D_x = 1.23$  Mg m<sup>-3</sup>,  $Z = 8$ . The final  $R = 0.076$  for 1346 reflexions. The five-membered ring and its attached C and O atoms are coplanar.

**Introduction.** Neidlein, Leinberger, Gieren & Dederer (1977) described a series of 1,2,5-thiadiazolidines. An X-ray structure determination has only been carried out for 2,5-diphenyl-1-(phenylimino)-1λ<sup>4</sup>,2,5-thiadiazolidine-3,4-dione. These compounds contain the interesting N–S–N group in a five-membered ring, where, in the case of the title compound, the S atom is not bonded to an additional organic group. The structure determination was undertaken to solve the question of a possible dimerization *via* an S–S bond, and to show the exact geometry of the five-membered heterocycle.

Rotation and Weissenberg photographs showed the orthorhombic symmetry of the crystals and yielded

approximate lattice constants. The systematic absences were characteristic for space group *Pbca*. Lattice constants were calculated by least squares (Berdesinski & Nuber, 1966) from the  $\theta$  values of 29 reflexions, centered diffractometrically. Intensity measurements on an automatic single-crystal diffractometer (AED, Siemens, Mo  $K\alpha$  radiation,  $\theta$ – $2\theta$  scan, five-value technique,  $2\theta$  up to  $56^\circ$ ) yielded 1346 observed reflexions with  $I > 2.58\sigma(I)$ . These were corrected with Lorentz and polarization factors only.

The asymmetric unit consists of one molecule. The positions of the ring and O atoms were obtained from a Patterson map. A Fourier synthesis revealed the positions of all other non-hydrogen atoms. Isotropic then anisotropic refinement resulted in  $R = 0.107$ . The positions of all H atoms could be detected on a difference map. In the final cycles all atomic parameters were varied, the H atoms with one common isotropic temperature factor, yielding  $R = 0.076$ . The