

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° . Contacts are consistent with van der Waals radii assuming for S a radius of 1.72 – 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).

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

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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

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Abstract. $C_{10}H_{18}N_2O_2S$, orthorhombic, *Pbca*, $a = 11.922$ (4), $b = 13.483$ (4), $c = 15.431$ (12) Å, $V = 2480$ Å³, $D_x = 1.23$ Mg m⁻³, $Z = 8$. 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)-1λ⁴,2,5-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 θ values of 29 reflexions, centered diffractometrically. Intensity measurements on an automatic single-crystal diffractometer (AED, Siemens, Mo $K\alpha$ radiation, θ – 2θ scan, five-value technique, 2θ up to 56°) 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

positional parameters of the non-hydrogen atoms are listed in Table 1, those of the H atoms in Table 2.*

Calculations were performed on Siemens 301 (Anorganisch-Chemisches Institut Heidelberg) and IBM 370/168 computers with the XRAY system (Stewart, Kundell & Baldwin, 1970) and scattering factors from Hanson, Herman, Lea & Skillman (1964).

Discussion. The structure determination showed the monomeric nature of the molecules. The shape of the molecule and the numbering scheme are shown by the ORTEP plot (Johnson, 1965) (Fig. 1), which omits H atoms for clarity. Bond distances and angles between

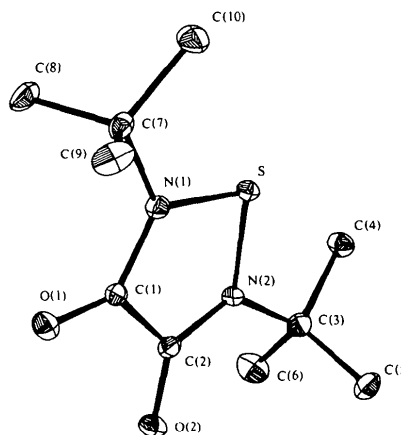


Fig. 1. Projection of the molecule with the atom numbering.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34679 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates ($\times 10^4$) for the non-hydrogen atoms

	x	y	z
S	955 (2)	1491 (1)	832 (1)
N(1)	639 (4)	1522 (4)	1906 (4)
N(2)	1675 (4)	401 (4)	972 (3)
O(1)	943 (5)	619 (4)	3155 (3)
O(2)	2290 (4)	9388 (4)	2056 (3)
C(1)	1062 (5)	757 (5)	2391 (4)
C(2)	1739 (5)	98 (5)	1784 (4)
C(3)	2313 (6)	1 (5)	191 (4)
C(4)	1833 (7)	443 (6)	9381 (5)
C(5)	3543 (7)	285 (7)	293 (5)
C(6)	2174 (8)	8872 (6)	153 (6)
C(7)	9869 (5)	2315 (5)	2248 (4)
C(8)	8791 (6)	1817 (6)	2526 (6)
C(9)	440 (7)	2832 (7)	3008 (6)
C(10)	9636 (8)	3039 (6)	1512 (6)

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

	x	y	z
H(41)	114 (7)	42 (5)	25 (5)
H(42)	226 (6)	22 (5)	881 (4)
H(43)	194 (6)	108 (5)	935 (5)
H(51)	378 (6)	7 (6)	90 (5)
H(52)	369 (6)	85 (5)	27 (5)
H(53)	412 (6)	994 (6)	973 (4)
H(61)	126 (6)	883 (5)	10 (5)
H(62)	238 (6)	860 (5)	62 (4)
H(63)	265 (6)	869 (5)	953 (4)
H(81)	836 (6)	221 (5)	265 (5)
H(82)	844 (6)	147 (5)	194 (5)
H(83)	898 (6)	156 (5)	329 (4)
H(91)	1 (6)	337 (6)	339 (4)
H(92)	74 (6)	226 (5)	368 (5)
H(93)	97 (6)	309 (5)	293 (4)
H(11)	915 (6)	273 (5)	95 (5)
H(12)	916 (7)	340 (6)	155 (5)
H(13)	31 (6)	337 (5)	137 (5)

Table 3. Bond lengths (Å)

S—N(1)	1.699 (6)	C(1)—C(2)	1.522 (9)
S—N(2)	1.715 (5)	C(3)—C(4)	1.498 (10)
N(1)—C(1)	1.522 (8)	C(3)—C(5)	1.524 (11)
N(1)—C(7)	1.505 (8)	C(3)—C(6)	1.532 (11)
N(2)—C(2)	1.320 (8)	C(7)—C(8)	1.512 (10)
N(2)—C(3)	1.524 (9)	C(7)—C(9)	1.525 (12)
O(1)—C(1)	1.202 (8)	C(7)—C(10)	1.522 (11)
O(2)—C(2)	1.235 (8)		

Table 4. Bond angles ($^\circ$)

N(1)—S—N(2)	90.5 (3)	C(4)—C(3)—N(3)	109.2 (6)
C(1)—N(1)—S	115.6 (4)	C(5)—C(3)—N(2)	108.0 (6)
C(7)—N(1)—S	119.6 (4)	C(6)—C(3)—N(2)	109.2 (6)
C(1)—N(1)—C(7)	124.6 (5)	C(4)—C(3)—C(5)	110.7 (6)
C(2)—N(2)—S	114.4 (4)	C(4)—C(3)—C(6)	108.8 (6)
C(3)—N(2)—S	117.0 (4)	C(5)—C(3)—C(6)	110.9 (6)
C(2)—N(2)—C(3)	127.8 (5)	C(8)—C(7)—N(1)	107.6 (5)
O(1)—C(1)—N(1)	127.5 (6)	C(9)—C(7)—N(1)	108.8 (5)
C(2)—C(1)—N(1)	107.4 (5)	C(10)—C(7)—N(1)	107.8 (6)
O(1)—C(1)—C(2)	125.2 (6)	C(8)—C(7)—C(9)	111.3 (7)
O(2)—C(2)—N(2)	126.3 (6)	C(8)—C(7)—C(10)	110.0 (6)
C(1)—C(2)—N(2)	111.9 (5)	C(9)—C(7)—C(10)	111.3 (6)
O(2)—C(2)—C(1)	121.7 (6)		

Table 5. Distances of atoms from a least-squares plane (Å)

The e.s.d.'s are 0.034 Å.

S	0.023	O(1)	-0.014
N(1)	0.037	O(2)	0.043
N(2)	-0.078	C(3)	0.019
C(1)	-0.004	C(7)	-0.006
C(2)	-0.021		

non-hydrogen atoms are compiled in Tables 3 and 4. The five-membered ring is planar; the plane also includes the O and C atoms bonded to the ring. Deviations from the common least-squares plane are shown in Table 5. Corresponding bond distances are equal within experimental error. The S–N distances are characteristic for single bonds; the C–C distance within the ring shows partial double-bond character.

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Structure of ($\alpha R, 1R, 2R$ - $\alpha S, 1S, 2S$)-1-Hydroxy- α -methyl-2-phenylcyclohexaneacetic Acid

BY G. HITE* AND JAMES B. ANDERSON

Section of Medicinal Chemistry, Pharmacognosy and Immunology, School of Pharmacy and Institute of Materials Science, University of Connecticut, Storrs, Connecticut 06268, USA

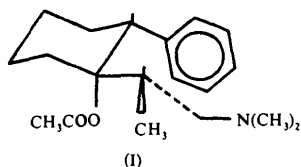
AND TELFER L. THOMAS, THOMAS A. DAVIDSON, RONALD C. GRIFFITH AND FRANCIS L. SCOTT

Chemistry Department, Pennwalt Corporation, Pharmaceutical Division, PO Box 1710, Rochester, New York 14603, USA

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Abstract. C₁₅H₂₀O₃, m.p. 402–403 K, space group $P2_1/a$, $a = 11.389$ (3), $b = 19.653$ (2), $c = 5.892$ (1) Å, $\beta = 92.76$ (2)°, $Z = 4$, $D_x = 1.252$, D_m (flotation: KI–H₂O) = 1.25 Mg m⁻³. The structure was solved by *MULTAN*. Full-matrix least-squares refinement converged with $R(F) = 0.04$. The ($\alpha R, 1R, 2R$ - $\alpha S, 1S, 2S$) stereochemistry confirms the *cis-threo* assignment of structure deduced earlier on the basis of PMR and chemical evidence.

Introduction. The synthesis of the ethyl ester of the title compound proceeds stereospecifically to give only one of four racemic diastereoisomers. Chemical and PMR data established the *cis* relationship of the phenyl and tertiary hydroxyl groups and led to the assignment of the *threo* stereochemistry about the acetate-to-ring bond (Thomas, Davidson, Griffith & Scott, 1976). Since this is a precursor of Nexeridine (I), an analgetic



* To whom correspondence should be addressed.

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currently undergoing clinical trials, it was deemed advisable to confirm the structural assignments by single-crystal X-ray analysis.

Photographs of a crystal (from toluene) about 0.2 mm on an edge revealed monoclinic symmetry. Systematic absences and molecular asymmetry led to the assignment of space group $P2_1/a$. Lattice parameters were refined by least-squares fitting of 12 automatically centered reflections ($32^\circ < 2\theta < 36^\circ$). Diffraction intensities were measured with Zr-filtered Mo $K\alpha_1$ radiation ($\lambda = 0.7093$ Å) on an automated Picker FACS-I diffractometer. Three standard reflections remained constant [$\pm 2\sigma(I_{avg})$] throughout data collection. Of 1924 independent reflections ($2\theta < 50^\circ$) of the form hkl and $h\bar{k}l$ with respect to a right-handed crystal axial system, 444 were considered unobserved according to the criterion: $|F_o| > 3.0\sigma$. Integrated intensities were corrected for Lorentz and polarization effects but no absorption correction was applied. The structure was solved with *MULTAN* (Germain, Main & Woolfson, 1971). Normalized structure factors were calculated from the observed data and 200 $|E|$'s (> 1.66) were used to calculate a map which revealed the positions of all non-hydrogen atoms. Full-matrix least-squares refinement (Busing, Martin & Levy, 1962) with a $1/\sigma^2$ weighting scheme, zerovalent