

C(2)–C(3) and C(3)–C(4) bond distances are significantly shorter than the expected value of 1.533 Å for a C–C bond length in *n*-hydrocarbons (Bartell, 1959). The C(22)–O(3) bond adjacent to the *F* ring with a length of 1.407 (4) Å is shorter than C(16)–O(3) with length 1.432 (4) Å, probably due to the presence of two electronegative atoms, O(3) and O(4), with non-bonded electrons adjacent to C(22).

The stereochemistry of (1) is as follows: C(5)– α H *trans* to C(10)– β CH₃; C(8)– β H *trans* to both C(9)– α H and C(14)– α H; C(9)– α H *trans* to C(10)– β CH₃; C(17)– α H *cis* to both C(16)– α H and C(20)– α CH₃ and *trans* to C(13)– β CH₃.

The *A/B*, *B/C* and *C/D* ring junctions are *trans*; the *D/E* ring junction is *cis*. Rings *A*, *B*, *C* and *F* all exist in chair conformations. The average values for the endocyclic torsion angles are 55.5 (4), 55.2 (4), 52.2 (4) and 57.2 (4)°, in agreement with that obtained for cyclohexane (Kahn, Fourme, André & Renaud, 1973). The substituents at C(3) and C(25) are equatorial.

The conformation of both five-membered rings may be described by the parameters Δ and φ_m (Altona, Geise & Romers, 1968). The *D* and *E* rings have Δ and φ_m values of 12.5 (6), 47.9 (6)° and 15.7 (6), 42.9 (7)°, respectively, indicating in both cases a conformation intermediate between half-chair and β -envelope. These values are similar to those of 3 α ,6 α -dihydroxy-5 β -cholan-24-oic acid (Hall, Maslen & Cooper, 1974) ($\Delta = 14.3^\circ$ and $\varphi_m = 44.8^\circ$) and sodium 3 α ,7 α ,12 α -trihydroxy-5 β -cholan-24-oate monohydrate (Cobledick & Einstein, 1980) ($\Delta = 14.3^\circ$ and $\varphi_m = 46.6^\circ$).

The molecular arrangement, consisting of layers of molecules perpendicular to **b**, is shown in Fig. 2. Two distinct types of hydrogen-bonding sequences create a rigid three-dimensional network of molecules. The first type connects the end of one molecule to the beginning of another by means of a water molecule forming spirals consisting of two hydrogen bonds [–O(4)⋯H(5B)–O(5)–H(5A)⋯O(1)–], which run parallel to **c**

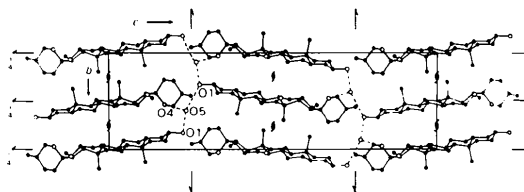


Fig. 2. The packing arrangement as viewed along **a**. Hydrogen bonds are shown by broken lines.

along the screw axes located at the edges and the centre of the unit cell (at $0,0,\frac{1}{4}$; $0,\frac{1}{2},\frac{1}{4}$ and $0,1,\frac{1}{4}$). The second type is a head-to-tail linking of adjacent layers from the C(3) hydroxy group to O(4) *via* the water molecule. Details of the hydrogen-bonding scheme have been deposited.*

We are greatly indebted to Consejo Nacional de Ciencia y Tecnología (CONACYT) de México, for funds which enabled us to obtain a single-phase uninterrupted power system to protect our diffractometer. We thank Mr Abelardo Cuellar for technical assistance. Project No. PCCBBNA-101810.

* See deposition footnote.

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Acta Cryst. (1984). **C40**, 2118–2120

3-Methyl-2(3H)-benzothiazolone, C₈H₇NOS

By STEVEN RUDD AND GEORGE BARANY*

Department of Chemistry, University of Minnesota, 207 Pleasant Street S.E., Minneapolis, MN 55455, USA

(Received 25 January 1984; accepted 29 August 1984)

Abstract. $M_r = 165.22$, orthorhombic, *Pbca*, $a = 16.941$ (4), $b = 10.816$ (2), $c = 8.424$ (2) Å, $V = 1544$ (1) Å³, $Z = 8$, $D_x = 1.422$ g cm⁻³, $\lambda(\text{Mo } K\alpha) =$

0.71073 Å, $\mu = 3.5$ cm⁻¹, $F(000) = 688$, $T = 297$ (1) K, $R = 0.038$ for 938 unique observed reflections. The five-membered heterocycle is flat and essentially coplanar with the fused benzene ring. Bond lengths and angles are all in accord with similar

* To whom correspondence should be addressed.

structures in the literature, and the C–N and C–S bonds of the thiazolone show partial double-bond character.

Introduction. The title compound, first made at the beginning of the century (Besthorn, 1910), can now be synthesized in a straightforward way (Barany, Schroll, Mott & Halsrud, 1983) from *N*-methylaniline and chlorocarbonylsulfonyl chloride. The X-ray structural analysis was carried out because of our interest in structure–reactivity correlations of sulfur-containing five-membered heterocycles (Barany, 1982). A search of the Cambridge Structural Database (1983) shows that there is almost no structural information on the dimensions of thiazolones.

Experimental. Thick needles, many of which were twinned, were grown by slow evaporation of a hexane solution of the title compound. A crystal of dimensions 0.35 × 0.20 × 0.70 mm was cut from a larger cluster. Enraf–Nonius CAD-4 diffractometer, θ – 2θ scans, graphite monochromator. 19 reflections with $10 < \theta < 19^\circ$ used for measuring lattice parameters. Systematic absences ($0kl$, $k = 2n+1$; $h0l$, $l = 2n+1$; $hk0$, $h = 2n+1$) uniquely determined space group. No absorption correction applied. Max. $(\sin\theta)/\lambda = 0.59 \text{ \AA}^{-1}$. One octant measured for $0 < 2\theta < 50^\circ$ ($h = 0$ to 20, $k = 0$ to 12, $l = 0$ to 10). Three check reflections measured every 5000 s of exposure time did not vary significantly over the course of the data collection. 1594 unique reflections measured, 938 with $I > \sigma(I)$ used in calculations. Structure solved by direct methods and refined using full-matrix least-squares refinement on F . Hydrogen-atom coordinates found on a difference Fourier map and refined with isotropic thermal parameters. All non-hydrogen atoms refined with anisotropic thermal parameters. Final $R = 0.038$, $wR = 0.043$, $S = 1.840$, with $w = 1/[\sigma(F)]^2$ and $\sigma(F)$ derived from $\sigma^2(I) = \sigma^2(I)_{\text{counting}} + (0.03 I)^2$. Max. $\Delta/\sigma = 0.04$. In final difference Fourier synthesis no electron density $> \pm 0.23 \text{ e \AA}^{-3}$. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). All calculations performed on a PDP 11/34 computer using Enraf–Nonius SDP program library (Frenz, 1978).

Discussion. The final atom parameters are given in Table 1.*

The structure of the title compound is shown in Fig. 1. Bond lengths and angles are all in accord with similar

* Lists of structure factors, anisotropic thermal parameters and bond angles and lengths involving hydrogen have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39693 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

structures in the literature. The C–N and C–S bonds in the thiazolone show partial double-bond character, and the heterocycle is planar. All individual atoms are within $\pm 0.015 \text{ \AA}$ of the least-squares mean plane through C(5), C(6), C(7), N and S. Moreover, the dihedral angle between the plane defined by C(1)–C(6) and the thiazolone plane is $1.0(2)^\circ$, implying that these planes are essentially coplanar.

We are grateful to Ms Dominique Chavatte-Kallander and Dr Andrew W. Mott for valuable contributions, and are indebted to Professor Doyle Britton and the University of Minnesota Department of Chemistry X-ray group for their help in structure solution. A literature search was performed through the courtesy of the Squibb Institute, Princeton, NJ. Purchase of the X-ray diffractometer and structure-solving equipment was aided by a grant from NSF (CHE-77-28505), and our own research was supported by a grant from NIH (GM-28934).

Table 1. Atomic coordinates and isotropic thermal parameters with e.s.d.'s in parentheses

	$B_{\text{iso}} = (B_{11} + B_{22} + B_{33})/3.$			
	x	y	z	$B_{\text{iso}}(\text{\AA}^2)$
S	0.39477 (4)	0.24640 (6)	0.50479 (8)	5.8
O	0.52740 (9)	0.3565 (2)	0.5944 (2)	6.7
N	0.4098 (1)	0.4392 (2)	0.6810 (2)	4.1
C(1)	0.2316 (1)	0.2796 (2)	0.5640 (3)	5.2
C(2)	0.1737 (1)	0.3474 (2)	0.6378 (3)	5.4
C(3)	0.1930 (1)	0.4491 (2)	0.7278 (3)	5.3
C(4)	0.2701 (1)	0.4851 (2)	0.7463 (3)	4.7
C(5)	0.3290 (1)	0.4173 (2)	0.6733 (3)	3.7
C(6)	0.3099 (1)	0.3149 (2)	0.5819 (2)	4.2
C(7)	0.4561 (1)	0.3567 (2)	0.6009 (3)	4.9
C(8)	0.4450 (1)	0.5387 (2)	0.7708 (3)	5.7
H(1)	0.223 (1)	0.215 (2)	0.509 (2)	4.9 (5)
H(2)	0.121 (1)	0.322 (2)	0.637 (3)	5.8 (5)
H(3)	0.152 (1)	0.491 (2)	0.774 (3)	5.8 (6)
H(4)	0.284 (1)	0.554 (2)	0.797 (2)	4.7 (5)
HA	0.429 (1)	0.618 (2)	0.737 (3)	8.9 (8)
HB	0.433 (1)	0.529 (2)	0.867 (3)	7.7 (7)
HC	0.501 (2)	0.533 (2)	0.761 (3)	8.0 (7)

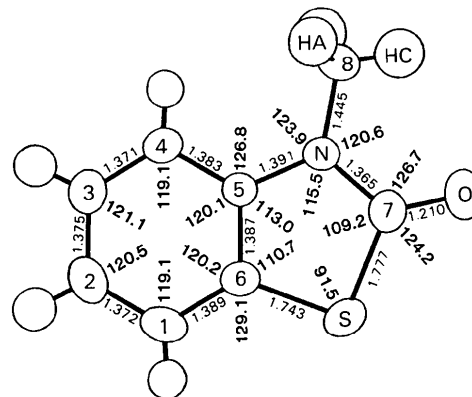


Fig. 1. A single molecule with atomic numbering, bond lengths (\AA) and angles ($^\circ$). The e.s.d.'s were all less than 0.003 \AA and 0.2° respectively.

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Acta Cryst. (1984). **C40**, 2120–2122

2-Imino-N-phenyl-3-thiazolidinecarboxamide, C₁₀H₁₁N₃OS

BY C. R. RASMUSSEN, FRANK J. VILLANI JR AND ELIZABETH A. GRIFFIN

Department of Chemical Research, McNeil Pharmaceutical, Spring House, Pennsylvania 19477, USA

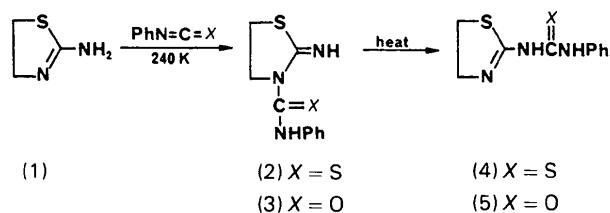
AND DAVID S. MORRISON AND R. A. OLOFSON

Department of Chemistry, The Pennsylvania State University, University Park, Pennsylvania 16802, USA

(Received 22 May 1984; accepted 29 August 1984)

Abstract. $M_r = 221.28$, orthorhombic, $P2_12_12_1$, $a = 7.550$ (2), $b = 12.684$ (3), $c = 22.095$ (3) Å, $V = 2116$ (1) Å³, $Z = 8$, $D_x = 1.389$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 2.79$ cm⁻¹, $F(000) = 928$, $T = 295$ K, $R = 0.072$ for 1625 observed reflections. The extremely labile kinetic adduct from reaction of 2-aminothiazoline with PhN=C=O has been identified thus also establishing several related structures. The geometry of the nearly flat molecule is determined by a hydrogen bond between the anilide NH and the imino nitrogen (imino NH not hydrogen-bonded to urea oxygen).

Introduction. Reaction of 2-amino-4,5-dihydrothiazoline (1) with phenyl isothiocyanate was first reported (Fromm & Kapeller-Adler, 1928) to give a low-melting adduct (2) which rearranged on melting (or more easily in solution) to an isomer (4). Later workers failed to find the low-melting adduct and assigned structure (2) to the high-melting isomer (m.p. 421 K) based on an 'unequivocal synthesis' (Klayman, Maul & Milne, 1967). In a subsequent retraction, the m.p. 421 K isomer was reassigned structure (4) (Klayman, Maul & Milne, 1968). A similar product (depicted as 5) was obtained (no 3 found) in the analogous treatment of (1) with phenyl isocyanate (Klayman, Maul & Milne, 1968). As part of a major investigation of the pharmaceutical utility of 2-pyrrolidinyldene ureas (Rasmussen, Gardocki, Plampin, Twardzik, Reynolds, Molinari, Schwartz, Bennetts, Price & Marakowski, 1978) and aryl urea derivatives of creatinine, the reactions of (1) with both PhN=C=S and PhN=C=O have been repeated and compounds identified as (2)–(5) isolated. All four structure proofs rest in part on the single-crystal X-ray structure analysis of the very unstable (3) presented here.



Experimental. Irregularly shaped crystal (0.25 × 0.2 × 0.3 mm) cleaved from larger crystal grown from ether–hexane at 195 K (Rasmussen, Villani, Mutter & Griffin, 1984); unit-cell parameters from 25 reflections in θ range 1.6–30°; Enraf–Nonius CAD-4 diffractometer (graphite monochromator); systematic absences $h00$ h odd, $0k0$ k odd, $00l$ l odd; data corrected for Lorentz and polarization factors but not for absorption; max. $2\theta = 60^\circ$, h, k, l max. 10, 17, 31; variable scan rate; three standard reflections every 1 h (anisotropic decay correction: 0.974–1.060, av. correction = 1.019); 3706 reflections measured, 2733 unique, 1108 unobserved [$I < 1.75\sigma(I)$]; structure by direct methods using *MULTAN78* (Main, Hull, Lesinger, Germain, Declercq & Woolfson, 1978) with all non-H atoms refined anisotropically on F ; H(N1) and H(N3) found by difference Fourier maps and their positions refined; remaining H atoms placed in calculated positions ($B_{\text{iso}} = 5.0$ Å²); 283 parameters refined; $R = 0.072$, $wR = 0.068$, $S = 1.33$; Corfield, Doedens & Ibers (1967) weighting scheme with $p = 0.07$ and $w = F^2/\sigma^2(F^2)$; max. $\Delta/\sigma = 0.02$; max. height in final difference Fourier map = 0.212 e Å⁻³; f, f' from Cromer & Waber (1974); programs: *MULTAN78*, *SDP Plus* version 1.0 (Enraf–Nonius, 1979), *ORTEPII* (Johnson, 1976).