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## Structure of Benzyl 2-Oxo-2,3-dihydro-1,3-benzothiazoleacetate

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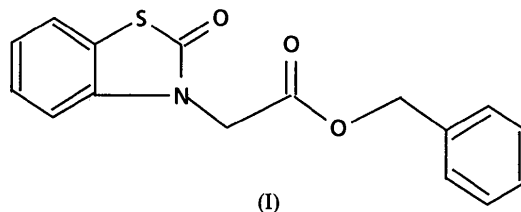
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**Abstract.** C<sub>16</sub>H<sub>13</sub>NO<sub>3</sub>S, *M<sub>r</sub>* = 299.3, monoclinic, *P*2<sub>1</sub>, *a* = 4.714 (0.5), *b* = 10.914 (1), *c* = 14.419 (2) Å, β = 94.29 (1)°, *V* = 739.7 (1) Å<sup>3</sup>, *Z* = 2, *D<sub>m</sub>* = 1.33, *D<sub>x</sub>* = 1.34 Mg m<sup>-3</sup>, λ(Mo *K*α) = 0.71069 Å, μ = 0.23 mm<sup>-1</sup>, *F*(000) = 312, room temperature. The structure was refined by full-matrix least squares to a final *R* = 0.043 for 786 observed reflections. Packing is essentially determined by the shortest CH...O = 2.28 and 2.48 Å intermolecular contacts. The atoms of the 2(3*H*)-benzothiazolone moiety are coplanar to within ±0.015 (7) Å.

**Introduction.** Derivatives of benzothiazole are a new group of compounds which exhibit biological growth activity in plants. Exceptionally, in higher concentrations they display growth retardation activity as well. Of these derivatives benzyl 2-oxo-2,3-dihydro-1,3-benzothiazoleacetate (I) has especially remarkable

biological properties (Varkonda, Hýblová, Sutoris, Konečný & Mikulášek, 1985). We now report the X-ray crystal structure determination of this compound.



**Experimental.** (I) was prepared by dissolving equimolar quantities of benzothiazole-2-ol and chloromethyl benzeneacetate in anhydrous acetone and triethylamine (Sutoris, Mikulášek, Sekerka & Konečný, 1984). Recrystallization from ethanol gave

Table 1. Final positional parameters for non-H atoms ( $\times 10^4$ ) and equivalent isotropic thermal parameters with e.s.d.'s in parentheses

	x	y	z	$B_{eq}^*$ ( $\text{\AA}^2$ )
S	1384 (5)	0	1291 (2)	7.0 (7)
O(1)	2167 (14)	910 (7)	3010 (5)	8.4 (2)
O(2)	1799 (12)	4020 (6)	2285 (4)	6.7 (2)
O(3)	-892 (13)	4492 (6)	3435 (5)	9.3 (2)
N	-1153 (12)	1841 (6)	1981 (4)	5.2 (2)
C(1)	906 (19)	978 (9)	2254 (6)	6.1 (3)
C(2)	-2184 (14)	1812 (7)	1056 (5)	4.8 (2)
C(3)	-4173 (17)	2596 (8)	615 (7)	6.4 (3)
C(4)	-4955 (21)	2379 (10)	-317 (7)	7.4 (3)
C(5)	-3834 (23)	1429 (12)	-790 (6)	7.8 (3)
C(6)	-1854 (20)	652 (9)	-348 (6)	6.8 (3)
C(7)	-1055 (15)	844 (7)	562 (6)	5.5 (2)
C(8)	-1973 (19)	2705 (8)	2679 (6)	6.0 (3)
C(9)	-124 (18)	3817 (8)	2751 (6)	5.5 (3)
C(10)	854 (21)	5580 (11)	3651 (10)	12.1 (5)
C(11)	-1043 (16)	6605 (8)	3865 (6)	5.8 (3)
C(12)	-2047 (19)	6719 (9)	4714 (6)	6.6 (3)
C(13)	-3817 (31)	7669 (16)	4898 (10)	11.0 (6)
C(14)	-4640 (36)	8483 (16)	4187 (19)	15.1 (9)
C(15)	-3431 (31)	8410 (17)	3423 (17)	14.7 (9)
C(16)	-1721 (30)	7480 (16)	3200 (8)	10.6 (5)

$$* B_{eq} = (4/3) \sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

colourless prismatic crystals. Found: N, 4.64; C, 64.28; S, 10.84; H, 4.41%. Calc.: N, 4.68; C, 64.20; S, 10.71; H, 4.38%. To obtain the intensity data a crystal  $0.2 \times 0.1 \times 0.3$  mm was chosen. Density measured by flotation (in  $\text{ZnSO}_4$  solution). Cell parameters by least squares from setting angles of 24 reflections with  $14 < 2\theta < 31^\circ$ . Intensities of 1006 independent reflections ( $0 \leq h \leq 5$ ,  $0 \leq k \leq 14$ ,  $-18 \leq l \leq 18$ ) were measured on a Hilger & Watts Y/290 diffractometer (Mo  $K\alpha$ , graphite monochromator,  $\omega/2\theta$  scan,  $2\theta_{\max} = 57^\circ$ ). No significant variation in intensities of three standard reflections (024, 021, 221) measured after every 100 reflections. All intensities corrected for Lorentz and polarization effects. No corrections for absorption or secondary extinction. Structure solved by direct methods (*MULTAN*; Germain, Main & Woolfson, 1971) employing 151 reflections with  $|E| > 1.3$ , refined by full-matrix least squares with anisotropic thermal parameters for non-H atoms, using 786 reflections with  $I \geq 2.0\sigma(I)$  and minimizing  $\sum w(|F_o| - |F_c|)^2$ ,  $w = 1/[\sigma^2|F_o| + (0.01|F_o|)^2]$ . Scattering factors from *International Tables for X-ray Crystallography* (1974). No correction for anomalous dispersion. H atoms located by a difference synthesis (coordinates and thermal parameters have not been refined). Final  $R = 0.043$ ,  $wR = 0.041$ , 189 variables, maximum shift/ $\sigma = 0.12$ , final electron-density fluctuations  $\pm 0.41 \text{ e \AA}^{-3}$ . All calculations performed with an Eclipse S/200 computer using the *INEXTL* programs (Gerr, Yanovsky & Struchkov, 1983).

**Discussion.** Final positional parameters for non-H atoms are given in Table 1 and selected distances and

Table 2. Selected distances ( $\text{\AA}$ ), bond angles ( $^\circ$ ) and torsion angles ( $^\circ$ ) with e.s.d.'s in parentheses

S—C(1)	1.779 (9)	O(1)—C(1)	1.205 (10)
S—C(7)	1.759 (8)	O(2)—C(9)	1.189 (11)
N—C(1)	1.388 (10)	O(3)—C(9)	1.304 (10)
N—C(2)	1.385 (9)	O(3)—C(10)	1.465 (12)
N—C(8)	1.453 (10)	C(8)—C(9)	1.493 (13)
C(2)—C(3)	1.388 (10)	C(10)—C(11)	1.478 (13)
C(3)—C(4)	1.387 (11)	C(11)—C(12)	1.352 (14)
C(4)—C(5)	1.369 (16)	C(12)—C(13)	1.369 (18)
C(5)—C(6)	1.381 (16)	C(13)—C(14)	1.390 (26)
C(6)—C(7)	1.353 (14)	C(14)—C(15)	1.280 (28)
C(7)—C(2)	1.401 (10)	C(15)—C(16)	1.350 (18)
		C(16)—C(11)	1.374 (17)
O(1)⋯H(C12 <sup>i</sup> )	2.59 (2)	O(2)⋯H(C3 <sup>iii</sup> )	2.62 (2)
O(1)⋯H(C14 <sup>ii</sup> )	2.77 (3)	O(2)⋯H(C6 <sup>iv</sup> )	2.48 (2)
O(1)⋯H(C15 <sup>iii</sup> )	2.76 (3)	O(2)⋯H <sup>i</sup> (C8 <sup>iii</sup> )	2.28 (1)
C(1)—S—C(7)	92.1 (4)	S—C(7)—C(6)	128.3 (7)
C(9)—O(3)—C(10)	115.8 (8)	C(2)—C(7)—C(6)	121.4 (8)
C(1)—N—C(2)	116.4 (7)	N—C(8)—C(9)	113.1 (7)
C(1)—N—C(8)	117.4 (7)	O(2)—C(9)—O(3)	126.1 (8)
C(2)—N—C(8)	126.1 (6)	O(2)—C(9)—C(8)	125.3 (8)
S—C(1)—O(1)	126.0 (7)	O(3)—C(9)—C(8)	108.6 (7)
S—C(1)—N	108.2 (6)	O(3)—C(10)—C(11)	108.6 (9)
O(1)—C(1)—N	125.8 (8)	C(10)—C(11)—C(12)	121.1 (9)
N—C(2)—C(3)	127.1 (7)	C(10)—C(11)—C(16)	119.5 (9)
N—C(2)—C(7)	112.8 (7)	C(12)—C(11)—C(16)	119.3 (9)
C(3)—C(2)—C(7)	120.1 (7)	C(11)—C(12)—C(13)	120.3 (9)
C(2)—C(3)—C(4)	117.4 (8)	C(12)—C(13)—C(14)	119.4 (10)
C(3)—C(4)—C(5)	121.9 (9)	C(13)—C(14)—C(15)	119.2 (20)
C(4)—C(5)—C(6)	121.2 (10)	C(14)—C(15)—C(16)	124.3 (22)
C(5)—C(6)—C(7)	118.8 (6)	C(11)—C(16)—C(15)	118.4 (11)
S—C(7)—C(2)	110.3 (6)		
N—C(8)—C(9)—O(2)	1.5 (7)	O(3)—C(10)—C(11)—C(12)	81.1 (9)
C(1)—N—C(8)—C(9)	87.1 (8)	C(9)—O(3)—C(10)—C(11)	139.9 (8)

Symmetry code: (i)  $-x, y - 0.5, -z$ ; (ii)  $x + 1, y - 1, z$ ; (iii)  $x + 1, y, z$ ; (iv)  $-x, y + 0.5, -z$ .

angles in Table 2. A projection of the structure along the  $a$  axis is presented in Fig. 1.\* The crystals contain discrete molecules of the title compound. The shortest intermolecular non-bonded contacts are  $\text{O}(2)\cdots\text{H}(82)$  2.28 and  $\text{O}(2)\cdots\text{H}(61)$  2.48  $\text{\AA}$  (see Table 2). None of these distances differ significantly from 2.4  $\text{\AA}$ , the sum of the van der Waals radii for  $\text{H}\cdots\text{O}$  systems. Similar non-bonded distances are found in 2-methyl-6-nitroso-7-phenethyl-1,3-benzothiazole (Prout & Miao, 1982). The dihydrobenzothiazole system is practically planar, the maximum deviation from the least-squares plane through S, O(1), N, C(1)—C(7) being 0.051 (7)  $\text{\AA}$  for O(1). This plane is tilted by  $145.4^\circ$  with respect to the plane of the phenyl ring. The six C—C bonds in the phenyl ring vary in length from 1.280 (28) to 1.390 (26)  $\text{\AA}$ , thus indicating a significant distortion from a standard

\* Lists of anisotropic thermal parameters for the non-H atoms, isotropic thermal parameters and coordinates of the H atoms, least-squares planes and deviations from them and lists of structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52351 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

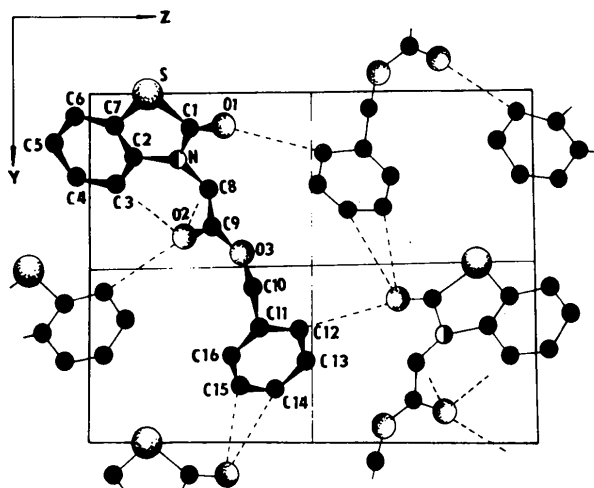


Fig. 1. Projection of the structure along the *a* axis. The O...H contacts listed in Table 2 are indicated by broken lines.

benzene ring. The mean value of 1.352 Å for these bond distances is significantly shorter than the value 1.397 Å expected from neutron diffraction (Bacon, Curry & Wilson, 1964). The C—S distances of 1.779 and 1.759 Å can be considered as pure single bonds (if the shortening of the bonds due to hybridization is taken into account); this cannot be said of the C(1)—N [1.388 (10) Å] and C(2)—N [1.385 (9) Å] distances. These lengths suggest delocalization along the system C(1)—N—C(2). The C(1)—S—C(7) angle (92.1°) is typical of S-containing five-membered heterocyclic molecules (Chesick & Donohue, 1971; Prout & Miao, 1982). Corresponding bond distances and angles found here and by Prout & Miao (1982) for free benzothiazole (bttz) molecules differ from those in coordinated bttz ligands. The free bttz mol-

ecule involves elongation of the C—N bond of the N—C—S system, in comparison with 1.320 (8) Å (Kubiak, 1985) and 1.309 (7) Å (Oughtred, Raper, Nowell & March, 1982) in bttz ligands (see Table 2). This change may be related to a strong metal–ligand interaction (Jeannin, Jeannin & Lavigne, 1979). Electron donation from the *X<sub>exo</sub>* atom to the metal (*X* = O or S) may induce a decrease in the  $\pi$ -electron density along the C—*X<sub>exo</sub>* bond and this would result in a charge displacement from the N lone pair to the C—N bond, in agreement with the observed C—N shortening in coordinated bttz molecules. The bond angles in the bttz ligand are distorted because of steric hindrance arising from coordination. The coordination alters the bttz ring geometry by decreasing the C—N—C angle and opening up the S—C—N angle.

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## Structure of Bis(cyclohexylammonium) Phosphoenolpyruvate Methanol Solvate (1/0.5)

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(Received 28 March 1989; accepted 19 June 1989)

**Abstract.** 2C<sub>6</sub>H<sub>14</sub>N<sup>+</sup>·C<sub>3</sub>H<sub>3</sub>O<sub>6</sub>P<sup>2-</sup>·0.5CH<sub>3</sub>OH, *M<sub>r</sub>* = 382.42, monoclinic, *C2/c*, *a* = 27.065 (15), *b* = 15.405 (6), *c* = 21.291 (16) Å,  $\beta$  = 112.30 (6)°, *V* = 8213 (10) Å<sup>3</sup>, *Z* = 16, *D<sub>m</sub>* = 1.239, *D<sub>x</sub>* = 1.237 (2) g cm<sup>-3</sup>, *Mo K $\alpha$* ,  $\lambda$  = 0.71069 Å,  $\mu$  =

1.73 cm<sup>-1</sup>, *F*(000) = 3312, *T* = 299 (1) K, final *R* = 0.066 for 4845 non-zero reflections. The crystal contains two crystallographically independent phosphoenolpyruvate dianions (*A* and *B*). In *B* the acidic H atom is attached to the phosphate group whereas

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