

The molecule has the *R,S* configuration and adopts a staggered conformation. The phenyl rings and the furoyl group are planar, the maximum deviations from the best planes being 0.005 (9), 0.001 (8) and 0.007 (10) Å respectively. The aromatic C—C lengths average 1.381 (4) Å, the C—H lengths 1.06 (2) Å (excluding methyl H's). The molecular structure displays no unusual features.

The thermal motion of the molecule appears to be governed by a libration about the P—C bond and wagging motions of the furan and phenyl groups. Atoms P and C(8) vibrate almost isotropically, the r.m.s. vibration components along the crystallographic axes being 0.214 (7), 0.209 (7), 0.226 (7) Å for P and 0.204 (27), 0.229 (25), 0.209 (26) Å for C(8).

We thank Dr G. Maas for providing the crystals and the Deutsche Forschungsgemeinschaft for financial

support. All calculations were done on a TR 440 computer at the Rechenzentrum der Universität des Saarlandes.

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*Acta Cryst.* (1985). **C41**, 1320–1322

### Structure of 3-Benzyl-5-(methoxycarbonylmethylthiomethyl)hydantoin, \* C<sub>14</sub>H<sub>16</sub>N<sub>2</sub>O<sub>4</sub>S†

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(Received 7 January 1985; accepted 11 April 1985)

**Abstract.**  $M_r = 308.4$ , monoclinic,  $P2_1$ ,  $a = 11.158$  (2),  $b = 6.010$  (1),  $c = 11.428$  (3) Å,  $\beta = 98.80$  (2)°,  $V = 757.3$  Å<sup>3</sup>,  $Z = 2$ ,  $D_m = 1.330$ ,  $D_x = 1.352$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.7107$  Å,  $\mu = 0.23$  mm<sup>-1</sup>,  $F(000) = 324$ ,  $T = 293$  K.  $R = 0.037$  for 1039 observed reflections. The hydantoin ring is planar and the dihedral angle it makes with the aromatic ring is 89.3 (7)°. There is a hydrogen bond N—H...O, 2.89 (1) Å, linking the molecules along the  $y$  axis, *via* N(1) and the C(4) carbonyl oxygen atom.

**Introduction.** As a part of the project on the synthesis of (+)-biotin from L(+)-cysteine, the optically active title compound has been prepared (Ravindranathan, Hiremath & Reddy, 1984).

**Experimental.** Crystal 0.40 × 0.12 × 0.35 mm,  $D_m$  by flotation in aqueous KBr solution, Nonius CAD-4F-11M diffractometer, graphite-monochromated Mo  $K\alpha$  radiation,  $\omega/2\theta$  scan mode, scan speed 1° min<sup>-1</sup>,  $\theta < 23.5^\circ$ ,  $h$  0 to 12,  $k$  0 to 6,  $l$  -12 to 12. 1369 reflections collected, 1039 judged significant ( $|F_o| > 3\sigma|F_o|$ ), lattice parameters from 19 reflections

( $16 < 2\theta < 38^\circ$ ), three standard reflections (400, 004 and 313) every 2000 s, 4% variation in intensity. No correction for absorption. Structure solved by direct methods using *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Full-matrix least-squares refinement of scale factor, positional and anisotropic thermal parameters (isotropic thermal parameters for hydrogen atoms, initial hydrogen positions calculated), final  $R = 0.037$ ,  $wR = 0.035$ ,  $S = 0.72$ ,  $\sum w(|F_o| - |F_c|)^2$  minimized,  $w = (2.5 + 1.0|F_o| + 0.04|F_o|^2)^{-1}$ .  $(\Delta/\sigma)_{\max} = 0.3$ . Final  $\Delta\rho$  excursions  $< 10.2$  e Å<sup>-3</sup>. No correction for secondary extinction. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Programs *LALS* (Gantzel, Sparks & Trueblood, 1961) used for refinement, *NRC-12* and *NRC-22* (Ahmed, Hall, Pippy & Huber, 1973) for bond lengths and angles and torsion angles.

**Discussion.** The atomic coordinates with their e.s.d.'s and equivalent isotropic temperature factors are given in Table 1. § Bond lengths and bond angles involving the

§ Lists of structure amplitudes, anisotropic thermal parameters and hydrogen-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42208 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

\* IUPAC name: methyl (3-benzyl-2,4-dioxo-5-imidazolidinyl-methylthio)acetate.

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non-hydrogen atoms are given in Table 2. Fig. 1 shows the numbering of the atoms. Fig. 2 shows the packing of the molecules in the unit cell viewed along *c*.

Table 1. Fractional atomic coordinates ( $\times 10^4$ ) with *e.s.d.*'s in parentheses and equivalent values of the isotropic temperature-factor coefficients

$$B_{\text{eq}} = \frac{4}{3}(B_{11}a^2 + B_{22}b^2 + B_{33}c^2 + B_{13}ac\cos\beta).$$

S	<i>x</i>	<i>y</i>	<i>z</i>	$B_{\text{eq}}(\text{\AA}^2)$
S	6862 (1)	2712	1968 (1)	3.98
C(2)	3890 (3)	1819 (7)	4160 (3)	3.16
C(4)	4089 (3)	-1109 (7)	2942 (3)	3.01
C(5)	4722 (4)	869 (8)	2507 (3)	2.99
C(6)	6052 (4)	338 (8)	2425 (3)	3.39
C(7)	6472 (3)	2575 (10)	369 (4)	4.14
C(8)	7086 (4)	626 (8)	-83 (3)	4.04
C(9)	8809 (7)	-588 (17)	-847 (7)	8.54
C(10)	2863 (4)	-1786 (7)	4548 (3)	3.39
C(11)	1567 (3)	-2035 (8)	3927 (3)	3.29
C(12)	933 (4)	-3942 (10)	4118 (4)	4.52
C(13)	-261 (5)	-4170 (14)	3605 (6)	5.49
C(14)	-830 (5)	-2578 (15)	2872 (7)	5.82
C(15)	-203 (6)	-676 (16)	2680 (7)	6.13
C(16)	992 (5)	-417 (12)	3203 (6)	4.89
N(1)	4571 (4)	2552 (8)	3365 (4)	3.36
N(3)	3607 (3)	-426 (8)	3899 (3)	2.88
O(1)	3556 (3)	2826 (8)	4976 (3)	4.44
O(2)	4029 (4)	-2971 (6)	2524 (3)	4.15
O(3)	6687 (4)	-1233 (9)	-167 (3)	5.39
O(4)	8156 (4)	1170 (9)	-373 (4)	5.47

Table 2. Bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ ) with *e.s.d.*'s in parentheses

C(2)—N(1)	1.344 (6)	C(2)—N(3)	1.407 (6)
C(2)—O(1)	1.218 (5)	C(4)—C(5)	1.505 (6)
C(4)—N(3)	1.354 (5)	C(4)—O(2)	1.215 (5)
C(5)—C(6)	1.536 (6)	C(5)—N(1)	1.436 (6)
C(6)—S	1.808 (5)	C(7)—C(8)	1.489 (7)
C(7)—S	1.814 (5)	C(8)—O(3)	1.201 (7)
C(8)—O(4)	1.328 (6)	C(9)—O(4)	1.436 (11)
C(10)—C(11)	1.518 (6)	C(10)—N(3)	1.448 (6)
C(11)—C(12)	1.382 (7)	C(11)—C(16)	1.371 (8)
C(12)—C(13)	1.378 (8)	C(13)—C(14)	1.363 (11)
C(14)—C(15)	1.375 (12)	C(15)—C(16)	1.383 (9)
N(1)—C(2)—N(3)	107.6 (4)	C(6)—S—C(7)	101.6 (2)
N(3)—C(2)—O(1)	123.6 (4)	N(1)—C(2)—O(1)	128.7 (4)
C(5)—C(4)—O(2)	126.8 (4)	C(5)—C(4)—N(3)	106.9 (3)
C(4)—C(5)—C(6)	111.3 (3)	N(3)—C(4)—O(2)	126.3 (4)
C(6)—C(5)—N(1)	113.8 (4)	C(4)—C(5)—N(1)	102.8 (3)
C(8)—C(7)—S	109.4 (3)	C(5)—C(6)—S	112.6 (3)
C(7)—C(8)—O(4)	111.8 (4)	C(7)—C(8)—O(3)	125.0 (4)
C(11)—C(10)—N(3)	113.3 (3)	O(3)—C(8)—O(4)	123.1 (5)
C(10)—C(11)—C(16)	122.5 (4)	C(10)—C(11)—C(12)	118.8 (4)
C(11)—C(12)—C(13)	120.0 (5)	C(12)—C(11)—C(16)	118.7 (4)
C(13)—C(14)—C(15)	118.8 (7)	C(12)—C(13)—C(14)	121.4 (6)
C(11)—C(16)—C(15)	120.8 (6)	C(14)—C(15)—C(16)	120.3 (7)
C(2)—N(3)—C(4)	111.0 (3)	C(2)—N(1)—C(5)	111.6 (4)
C(2)—N(3)—C(10)	124.1 (4)	C(8)—O(4)—C(9)	116.1 (4)
C(4)—N(3)—C(10)	124.9 (4)		

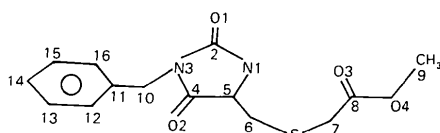


Fig. 1. Atom numbering.

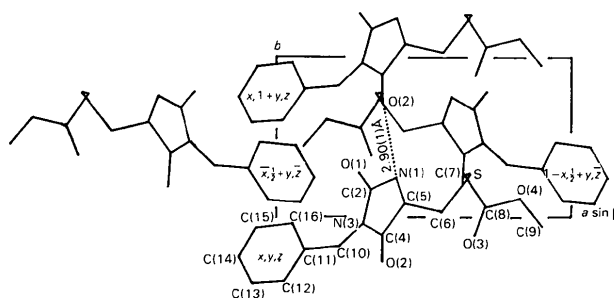


Fig. 2. The (001) projection.

The geometry of the five-membered hydantoin ring is very similar to that in the structure of DL-allantoin (Mootz, 1965). The hydantoin ring is planar with the carbonyl oxygen atoms lying in the plane of the ring to within 0.055  $\text{\AA}$ . The C—N bond, involving the tetrahedral atom C(5), of 1.436 (6)  $\text{\AA}$  is fairly close to the corresponding value of 1.461  $\text{\AA}$  in DL-allantoin as is the C(4)—C(5) bond distance of 1.505 (6)  $\text{\AA}$  to the value proposed for an  $sp^2-sp^3$  C—C single bond (Lide, 1962). The two hydantoin C=O distances both agree to well within the limits of experimental error with those in DL-allantoin and also with the average value of 1.21  $\text{\AA}$  in barbiturates (Craven, Cusatis, Gartland & Vizzini, 1973).

The bonds involving sulphur, C(7)—S and C(6)—S, 1.814 (5) and 1.808 (5)  $\text{\AA}$ , agree well with the C—S single-bond distance of 1.82  $\text{\AA}$  (Sutton, 1965). The carbonyl carbon to nitrogen bond distances are 1.344 (6), 1.354 (5) and 1.407 (6)  $\text{\AA}$  which agree with the average value of 1.37  $\text{\AA}$  in barbiturates (Craven *et al.*, 1973).

There is a hydrogen bond, N(1)—H...O(2)(*x, y + 1, z*), linking the molecules along the 2<sub>1</sub> screw axis (Fig. 2) with N...O = 2.89 (1)  $\text{\AA}$  (N—H...O = 145°, H...O = 2.15  $\text{\AA}$ ). The dihedral angle between the aromatic plane and the plane of the five-membered hydantoin ring is 89.3 (7)°.

We thank Drs A. P. B. Sinha, L. M. Pant and V. G. Puranik for their keen interest and constant encouragement, and Drs T. Ravindranathan and S. V. Hiremath and Mr D. R. Reddy for the crystals.

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*Acta Cryst.* (1985). **C41**, 1322–1324

## The Structure of 13,14-Dimethoxy-3,10-diazabicyclo[10.2.2]hexadeca-12,14,15-triene-2,11-dione Monohydrate, C<sub>16</sub>H<sub>22</sub>N<sub>2</sub>O<sub>4</sub>·H<sub>2</sub>O

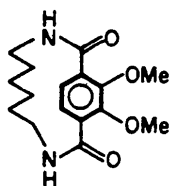
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(Received 20 August 1984; accepted 9 April 1985)

**Abstract.**  $M_r = 324.37$ , orthorhombic,  $P2_12_12_1$ ,  $a = 10.6103$  (11),  $b = 12.6144$  (10),  $c = 12.7713$  (13) Å,  $V = 1709.3$  (5) Å<sup>3</sup>,  $Z = 4$ ,  $D_m = 1.258$  (4),  $D_x = 1.261$  g cm<sup>-3</sup>, Mo  $K\alpha$ ,  $\lambda = 0.71073$  Å,  $\mu = 0.877$  cm<sup>-1</sup>,  $F(000) = 696$ ,  $T = 298$  K,  $R = 0.0291$  for 2102 independent reflections with  $F^2 > 3\sigma(F^2)$ . The crystal structure shows the asymmetric orientation of the hexamethylene bridge. This bridge is tilted toward the methoxy groups. The benzene ring is slightly distorted, and the water molecule forms an intermolecular bridge between amide and carbonyl groups of adjacent molecules *via* hydrogen bonding.

**Introduction.** Macrocyclic ligands containing pyrocatechol moieties (1,2-benzenediol) are of interest as metal chelating agents, owing to their potentially enhanced strength and selectivity of bonding. The title compound (1) was prepared in an intermediate step of a program to synthesize such macrocyclic ligands. It was isolated along with higher-order cyclic oligomers (dimers, trimers *etc.*) from the high-dilution reaction of 2,3-dimethoxyterephthaloyl dichloride and 1,6-hexanediamine. Unlike the smaller-chain diamines, 1,6-hexanediamine was of sufficient length to bridge across one terephthalate moiety, yielding this monomeric compound whose physical properties were substantially different from the other cyclic oligomers. In particular, <sup>1</sup>H NMR shows a considerable upfield shift of the bridging methylene hydrogens, indicating that these protons are constrained to lie atop the benzene ring.



**Experimental.** Colorless hexagonal prisms, crystallized from water, provided by SJR, Dr Chui Ng and Dr K. N. Raymond;  $D_m$  by flotation in salt (K<sub>2</sub>CO<sub>3</sub>) water; crystal 0.45 × 0.34 × 0.40 mm; precession photographs, orthorhombic symmetry and systematic absences  $h00$  for  $h$  odd,  $0k0$  for  $k$  odd,  $00l$  for  $l$  odd; Enraf–Nonius CAD-4 diffractometer, unit cell: least squares on 24 high-angle reflections; graphite-monochromatized Mo  $K\alpha$ ,  $\theta$ – $2\theta$  scans; 2507 intensities in quadrant  $+h +k \pm l$  ( $h = 0$ –11,  $k = 0$ –13,  $l = 0$ –13),  $3 < 2\theta < 45^\circ$ ; no decrease in intensity in 3 standards nor did crystal orientation need to be redetermined during data collection; data corrected for background, scan speed, Lorentz and polarization factors (Frenz, 1982); empirical absorption correction,  $T_{\min} = 97.81$ ,  $T_{\max} = 99.87$ ,  $\sin\theta/\lambda_{\max} = 0.5233$  Å<sup>-1</sup>; systematically absent reflections and redundant data rejected yielding 2230 data, 2102 of which had  $F^2 > 3\sigma(F^2)$ . Structure solved by direct methods; first run of *MULTAN11/82* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) unambiguously yielded positions of nitrogen atoms, four of five oxygen atoms and fourteen of sixteen carbon atoms; subsequent least-squares refinement and Fourier syntheses yielded positions of remaining carbon and oxygen atoms; majority of hydrogen atoms located following full anisotropic refinement of non-hydrogen atoms. All hydrogen atoms, except water hydrogens, assigned idealized coordinates, with bond lengths 0.95 Å; hydrogen atoms assigned thermal factors  $B = 4.3$  to  $10.8$  Å<sup>2</sup>, based on magnitude of thermal parameters of parent atoms; water hydrogens assigned coordinates of their associated peaks in difference Fourier map. Extinction coefficient refined to  $8.033 \times 10^{-7}$  (Darwin, 1922).  $w = \{4F_o^2/\sigma^2(F_o^2)\}$ ,  $\sigma^2(F_o^2) = \{\sigma_o^2(F_o^2) + (pF_o^2)^2\}$ ,  $p = 0.02$ ,  $\sum w(\Delta F)^2$  minimized; final least-squares cycle  $R = 2.91$ ,  $wR = 4.21\%$ , enantiomeric structure refined to same  $R$  values; max.  $\Delta/\sigma = 0.00$ ; final difference Fourier  $\Delta\rho$