Table 2. *Equations of best molecular planes, and distances* (Å) *of atoms from these planes*

Root-mean-square deviations of the fitted atoms for planes (*a*), (*b*), (*c*) and (*d*) are 0·005 (1), 0·006 (2), 0·007 (1) and 0·004 (2), respectively. Calculations were performed with program *ORFFE*4 (Busing, Martin & Levy, 1964). X, Y and Z represent direct-axis coordinates expressed in Å.

$$\begin{aligned}
&(a)\quad 0{\cdot}1576X - 0{\cdot}1430Y + 0{\cdot}9552Z = 3{\cdot}9926\\
&(b)\quad 0{\cdot}2313X - 0{\cdot}1688Y + 0{\cdot}9287Z = 4{\cdot}5501\\
&(c)\quad -0{\cdot}1261X + 0{\cdot}1509Y - 0{\cdot}9619Z = -1{\cdot}3714\\
&(d)\quad -0{\cdot}2816X + 0{\cdot}1991Y - 0{\cdot}9040Z = -2{\cdot}0470
\end{aligned}$$

(*a*)		(*b*)		(*c*)		(*d*)	
S(11)	0·007 (1)	N(11)	−0·003 (2)	S(12)	−0·002 (1)	N(12)	−0·001 (2)
S(21)	−0·006 (2)	C(11)	−0·004 (3)	S(22)	−0·003 (1)	C(12)	−0·002 (2)
C(51)	0·004 (2)	C(21)	0·005 (3)	C(52)	0·008 (2)	C(22)	0·000 (3)
C(61)	0·002 (2)	N(21)	0·001 (3)	C(62)	−0·011 (2)	N(22)	0·004 (3)
C(71)	−0·007 (2)	C(31)	−0·008 (3)	C(72)	0·008 (2)	C(32)	−0·007 (3)
O(11)*	0·016 (5)	C(41)	0·009 (3)	O(12*	0·026 (5)	C(42)	0·005 (3)
C(81)*	−0·023 (7)	C(71)*	0·021 (5)	C(82)*	−0·034 (7)	C(72)*	−0·056 (5)
C(11)*	−0·014 (5)	H(21)*	0·08 (4)	C(12)*	0·005 (5)	H(22)*	0·04 (3)
H(8*a*1)*	0·12 (5)	H(31)*	0·06 (4)	H(8*a*2)*	0·09 (5)	H(32)*	−0·04 (4)
H(8*b*1)*	−0·74 (4)	H(41)*	0·00 (3)	H(8*b*2)*	−0·70 (4)	H(42)*	−0·03 (4)
H(8*c*1)*	0·63 (4)			H(8*c*2)*	0·73 (4)		

* Atoms not included in the calculation of the least-squares plane.

to alleviate in part the intramolecular strain, the five- and six-membered rings in each molecule are twisted away from each other by a rotation around the C(1)—C(7) bonds, resulting in an angle of 4·6 (1)° between the plane normals for molecule (1), while the corresponding value for molecule (2) is 9·6 (1)°. The difference of 5·0 (1)° in these two dihedral angles is noticeable, as is the difference between the two independent molecules of oltipraz [5·2 (1)°]. The measure of this twist between the pyrazine ring and the 1,2-dithiole ring can also be expressed in terms of the torsion angles S(1)—C(7)—C(1)—N(1). For molecules (1) and (2), these values are −4·4 (4) and 8·9 (3)°, respectively. It should be noted that the signs of these values change for the inverted molecules.

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Structure of 8-Benzyl-4-thia-1,8-diazabicyclo[4.3.0]nonane-2,7,9-trione*

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Abstract. $C_{13}H_{12}N_2O_3S$, $M_r = 276{\cdot}3$, orthorhombic, *Pcab* (non-standard setting of *Pbca*). $a = 8{\cdot}851$ (2), $b = 12{\cdot}035$ (2), $c = 23{\cdot}506$ (3) Å, $V = 2504$ Å^3, $Z = 8$, $D_m = 1{\cdot}458$, $D_x = 1{\cdot}466$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 0{\cdot}256$ mm^{-1}, $F(000) = 1152$, $T = 293$ K. $R = 0{\cdot}067$ for 1491 observed reflections. The hydantoin ring is planar while the thiazine ring adopts a

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'boat' conformation. The hydantoin ring makes a dihedral angle of 113·9 (4)° with the planar aromatic ring.

Table 1. *Fractional atomic coordinates* (× 10⁴) *with e.s.d.'s in parentheses and equivalent isotropic temperature factors* (Å²)

$$B_{eq} = \tfrac{4}{3}(b_{11}a^2 + b_{22}b^2 + b_{33}c^2).$$

	x	*y*	*z*	B_{eq}
C(2)	5706 (4)	637 (3)	2997 (1)	1·92
C(4)	4105 (4)	2132 (3)	2981 (2)	2·20
C(5)	4201 (4)	1706 (3)	2378 (2)	2·14
C(6)	4753 (5)	2608 (3)	1970 (2)	3·21
C(8)	5072 (5)	568 (3)	1421 (2)	2·97
C(9)	5749 (4)	175 (3)	1969 (2)	2·47
C(10)	5287 (4)	1637 (3)	3914 (2)	2·76
C(11)	4155 (4)	1053 (3)	4294 (2)	2·46
C(12)	3242 (6)	1644 (4)	4654 (2)	4·54
C(13)	2240 (6)	1097 (5)	5013 (2)	5·53
C(14)	2149 (5)	−26 (5)	5014 (2)	4·52
C(15)	3061 (5)	−630 (4)	4652 (2)	3·92
C(16)	4055 (5)	−91 (3)	4293 (2)	3·35
O(1)	6523 (3)	−69 (2)	3191 (1)	2·61
O(2)	3387 (3)	2925 (2)	3147 (1)	3·86
O(3)	6632 (4)	−583 (3)	2002 (1)	4·37
N(1)	5262 (3)	786 (2)	2434 (1)	2·36
N(3)	4991 (3)	1463 (2)	3305 (1)	2·48
S	5489 (1)	2032 (1)	1316 (1)	4·18

Table 2. *Bond lengths* (Å), *bond angles* (°) *and selected torsion angles* (°) *with e.s.d.'s in parentheses*

C(2)–O(1)	1·205 (4)	C(2)–N(1)	1·391 (3)
C(2)–N(3)	1·384 (4)	C(4)–C(5)	1·509 (7)
C(4)–O(2)	1·211 (5)	C(4)–N(3)	1·358 (5)
C(5)–C(6)	1·530 (6)	C(5)–N(1)	1·459 (4)
C(6)–S	1·806 (5)	C(8)–C(9)	1·498 (6)
C(8)–S	1·817 (4)	C(9)–O(3)	1·204 (5)
C(9)–N(1)	1·386 (5)	C(10)–C(11)	1·515 (6)
C(10)–N(3)	1·469 (5)	C(11)–C(12)	1·370 (6)
C(11)–C(16)	1·380 (5)	C(12)–C(13)	1·391 (7)
C(13)–C(14)	1·354 (9)	C(14)–C(15)	1·379 (7)
C(15)–C(16)	1·381 (6)		
O(1)–C(2)–N(1)	128·3 (3)	O(1)–C(2)–N(3)	125·6 (3)
N(1)–C(2)–N(3)	106·0 (3)	C(5)–C(4)–O(2)	126·8 (4)
C(5)–C(4)–N(3)	107·1 (3)	O(2)–C(4)–N(3)	126·1 (4)
C(4)–C(5)–C(6)	111·5 (3)	C(4)–C(5)–N(1)	102·9 (3)
C(6)–C(5)–N(1)	113·0 (3)	C(5)–C(6)–S	112·1 (3)
C(9)–C(8)–S	110·0 (3)	C(8)–C(9)–O(3)	123·6 (4)
C(8)–C(9)–N(1)	112·8 (3)	O(3)–C(9)–N(1)	123·6 (4)
C(11)–C(10)–N(3)	112·9 (3)	C(10)–C(11)–C(12)	120·8 (4)
C(10)–C(11)–C(16)	120·4 (4)	C(12)–C(11)–C(16)	118·7 (4)
C(11)–C(12)–C(13)	120·4 (5)	C(12)–C(13)–C(14)	120·8 (5)
C(13)–C(14)–C(15)	119·4 (5)	C(14)–C(15)–C(16)	120·1 (4)
C(11)–C(16)–C(15)	120·6 (4)	C(2)–N(1)–C(5)	111·4 (3)
C(2)–N(1)–C(9)	126·4 (3)	C(5)–N(1)–C(9)	122·1 (3)
C(2)–N(3)–C(4)	113·3 (3)	C(2)–N(3)–C(10)	122·1 (3)
C(4)–N(3)–C(10)	124·4 (3)	C(6)–S–C(8)	100·6 (2)
Thiazine ring			
C(9) N(1) C(5) -C(6)	−56·9 (4)		
N(1)–C(5)–C(6)–S	45·8 (4)		
C(5)–C(6)–S–C(8)	4·8 (3)		
C(6)–S–C(8)–C(9)	−55·1 (3)		
S–C(8)–C(9)–N(1)	56·6 (4)		
C(8)–C(9)–N(1)–C(5)	0·1 (5)		

Introduction. As a part of a project on the synthesis of (+)-biotin from L(+)-cysteine, the title compound was formed by the acid-catalyzed cyclization of 3-benzyl-5-(methoxycarbonylmethylthiomethyl)hydantoin (Ravindranathan, Hiremath & Reddy, 1984).

Experimental. Crystal ~0·27 × 0·70 × 0·90 mm. D_m by flotation in aqueous KI, Nonius CAD-4*F*-11M diffractometer, graphite-monochromated Mo *Kα* radiation, $\omega/2\theta$ scan mode, scan speed 1° min^{-1}, $\theta \leq 24°$, *h* 0 to 10, *k* 0 to 13, *l* 0 to 26. 2380 reflections collected, 1491 judged significant ($|F_o| > 3\sigma|F_o|$), lattice parameters from 24 reflections ($15·5 < 2\theta < 36·3°$), three standard reflections (0,4,12, 241 and $0,\bar{1},16$) every 2000 s, 4% variation in intensity. No correction for absorption. Structure solved by direct methods using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Full-matrix least-squares refinement (on *F*) of scale factor, positional and anisotropic thermal parameters (isotropic thermal parameters for H atoms, initial H positions calculated by stereochemistry) converged to $R = 0·067$ and $wR = 0·062$, $S = 3·67$, $\sum w(|F_o| - |F_c|)^2$ minimized, $w = (10·0 + 1·0|F_o| + 0·009|F_o|^2 + 0·0001|F_o|^3)^{-1}$. $(\Delta/\sigma)_{max} = 0·25$. Final $\Delta\rho$ excursions $< |0·2|$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Program *LALS* (Gantzel, Sparks & Trueblood, 1961) used for refinement. An appropriate extinction correction was applied to seven reflections (Dunitz, 1979). Of these the two strongest reflections, namely 200 (149·8, 221·9) and 202 (174·2, 218·2), still show large discrepancies even after the application of the correction.

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 non-H atoms and selected torsion angles are given in Table 2. Fig. 1 shows the chemical formula and numbering of the atoms. Fig. 2 shows a perspective view of the molecule.

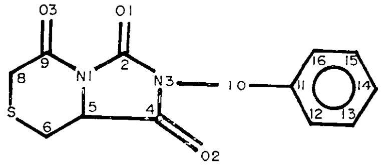

Fig. 1. Atom numbering.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and torsion angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42332 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

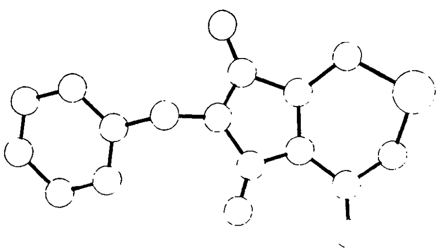

Fig. 2. Perspective view of the molecule.

The bond lengths and angles in the hydantoin ring are comparable with those in DL-allantoin (Mootz, 1965) and 3-benzyl-5-(methoxycarbonylmethylthiomethyl)hydantoin (hereafter 5CM3BH) (Dhaneshwar, Tavale & Guru Row, 1985). The C(5)—N(1) bond involving the tetrahedral C atom has a length of 1·459 (4) Å, close to the corresponding values of 1·461 Å in DL-allantoin and 1·46 (1) Å in 5CM3BH. The C(4)—C(5) bond distance of 1·509 (7) Å is close to the *sp*^{2}—*sp*^{3} C—C single-bond value of 1·501 (4) Å (Lide, 1962). The three carbonyl bonds are shorter than the mean value of 1·216 Å quoted for 12 barbiturates (Craven, Cusatis, Gartland & Vizzini, 1973) due to the absence of any hydrogen bonding, as in the case of phenobarbitone (Williams, 1974). The hydantoin ring is planar in the present case as in DL-allantoin and 5CM3BH. Atoms O(1), O(2) and C(10) lie within 0·01 Å of the ring plane, while O(3), C(8) and C(9) deviate from the ring plane by 0·1 Å.

The thiazine ring has a boat form (Hendrickson, 1964). Angles C(6)—S—C(8) and C(5)—N(1)—C(9) are 100·6 (2) and 122·1 (3)°, comparable with the corresponding angles 100·2 (1) and 122·1 (2)° in 10-(2-nitrophenyl)phenothiazine (Jovanovic, Biehl, de Meester & Chu, 1984). The two C—S bonds, C(6)—S and C(8)—S are 1·806 (5) and 1·817 (4) Å, very close to the C—S single-bond value of 1·82 Å (Sutton, 1965).

The aromatic ring is planar and makes a dihedral angle of 113·9 (4)° with the hydantoin ring.

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10β-Hydroxy-1,4-estradiene-3,17-dione

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Abstract. $C_{18}H_{22}O_3$, $M_r = 286·4$, orthorhombic, $P2_12_12_1$, $a = 7·014$ (2), $b = 12·176$ (4), $c = 17·648$ (4) Å, $V = 1507·2$ Å^3, $Z = 4$, $D_x = 1·26$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0·079$ mm^{-1}, $F(000) = 616$, T *ca* 130 K, $R = 0·109$ for all 2506 unique reflections within $\lambda^{-1}\sin\theta = 0·70$ Å^{-1}; the absolute configuration was not determined. The relative stereochemistry at C(10) is established. The

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