while the next closest distance between Cl^- and N^+ is 5·15 Å, so the only significant ionic attractions are within each ion pair.

One major packing interaction is that between hydrophobic portions of the bulky $HMTH^+$ ions (Fig. 2). The second major packing interaction is the tight surrounding of each hydrogen-bonded chloride ion by six additional non-bonded hydrogen atoms (Fig. 3). Three of these, each at a distance of 2·817 Å from Cl^-, are H(4) atoms from the ion pair translated up along **c** from the given ion pair. The three others, each 2·908 Å from Cl^-, are H(5) atoms in three separate ion pairs related to the given one by rhombohedral centering translations. These $Cl^-\cdots H$ distances are essentially equal to the sum of the ionic radius of Cl^-, 1·81 Å (Pauling, 1970), and the van der Waals radius of aliphatic H, 1·06–1·20 Å (Bondi, 1964).

This crystal is isostructural with that of HMT.HBr (Mak, Li & Yip, 1983).

Margaret Chou thanks IBM for an undergraduate summer research stipend.

References

Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.
International Tables for X-ray Crystallography (1974). Vol. IV, pp. 99, 149. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
Mak, T. C. W., Li, W.-K. & Yip, W.-H. (1983). *Acta Cryst.* C**39**, 134–136.
Pauling, L. (1970). *General Chemistry*, p. 175. San Francisco: Freeman.
Sheldrick, G. M. (1983). *SHELXTL. An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data.* Univ. of Göttingen.

Acta Cryst. (1987). C**43**, 324–326

Structure of 7a-Hydroxy-4-phenyl-1,2,4,4a,5,6,7,7a-octahydrocyclopenta[*d*][1,3]thiazine-2-thione

By Gyula Argay and Alajos Kálmán

Central Research Institute for Chemistry, Hungarian Academy of Sciences, PO Box 17, *Budapest, H*-1525 *Hungary*

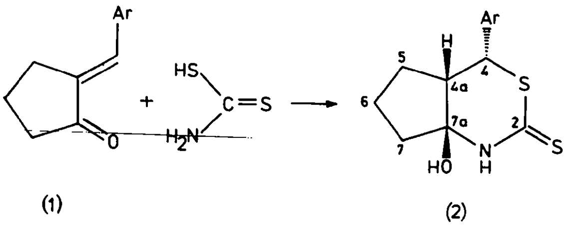
and Pál Perjési and Dezsö Szabó

Department of Chemistry, University Medical School, PO Box 99, *Pécs, H*-7601 *Hungary*

(*Received* 12 *June* 1986; *accepted* 10 *September* 1986)

Abstract. $C_{13}H_{15}NOS_2$, $M_r = 265·4$, monoclinic, $C2/c$, $a = 30·482\,(2)$, $b = 6·989\,(1)$, $c = 12·685\,(1)$ Å, $\beta = 111·09\,(1)°$, $V = 2521·4\,(5)$ Å^3, $Z = 8$, $D_x = 1·398$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0·71073$ Å, $\mu = 0·39$ mm^{-1}, $F(000) = 1120$, $T = 293\,(1)$ K. $R = 0·050$ for 2290 unique observed reflections. Only X-ray analysis could reveal the structure of the title compound obtained by the reaction of 2-benzylidenecyclopentanone with dithiocarbamic acid. It has been shown to be a *cis*-fused diastereomer in which the 4-phenyl group is in a *trans* position to the annular H atom.

Introduction. The reactions of 2-arylidenecyclopentanones (1*a*–*c*) with dithiocarbamic acid have been investigated. In these reactions stereohomogenous 4-aryl-7a-hydroxy-1,2,4,4a,5,6,7,7a-octahydrocyclopenta[*d*][1,3]thiazine-2-thiones (2*a*–*c*) were formed. The crude-product analysis (^{1}H NMR) showed that in each case the formation of only one diastereoisomer could be observed as depicted in the scheme. However, as to which of the four possible diastereomers was furnished no spectroscopic answer could be given. Consequently, an X-ray analysis of (2*a*) (melt: 431–434 K) has been performed.



Ar: (*a*) C_6H_5, (*b*) 4-CH_3–C_6H_4, (*c*) 4-CH_3O–C_6H_4

Experimental. Colourless crystals, *ca* 0·30 × 0·35 × 0·40 mm, CAD-4 diffractometer, $\omega/2\theta$ scan in the range $1·5 < \theta < 27·0°$ with scan width 0·40° + 0·35° tanθ using graphite-monochromated Mo $K\alpha$ radiation. Cell constants by least squares using 25

0108-2701/87/020324-03$01.50

reflections with $20{\cdot}4 \le \theta \le 22{\cdot}6°$. Three standard reflections (28,0,0, 17,1,13 and 30,0,$\bar{8}$) were monitored every hour and showed no loss or gain in intensity during 23·5 h of exposure. 2740 unique reflections were recorded with h 0–38, k 0–8, l −16–14 of which – after data correction – 2290 with $I > 3{\cdot}0\sigma(I)$ were taken as observed. The structure was solved by *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) using 240 $E > 1{\cdot}70$ normalized structure factors. An empirical absorption correction using program *DIFABS* (Walker & Stuart, 1983) (max. and min. absorption corrections: 1·27 and 0·81) reduced R from 0·15 to 0·12. Positions of H atoms bound to C atoms were generated from assumed geometries; those belonging to N or O were located in difference Fourier map at $R = 0{\cdot}06$. Full-matrix least-squares refinement. $\sum w(\Delta F)^2$ minimized for 155 parameters. Final $R = 0{\cdot}050$, $wR = 0{\cdot}070$, $S = 4{\cdot}8$, $w = [\sigma^2(F_o) + 0{\cdot}01F_o^2]^{-1}$. Extinction coefficient $3{\cdot}8 \times 10^{-7}$. Max. peak height in final $\Delta\rho$ map 0·32 e Å^{-3}. Max. Δ/σ 0·4. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1962). Program system applied: Enraf–Nonius (1982) *SDP* with local modifications adapted to a PDP 11/34 minicomputer (64 K).

Discussion. Atomic coordinates of non-H atoms are in Table 1.* The molecular geometry with atomic numbering is shown in Fig. 1. Bond lengths, angles and relevant torsion angles are given in Table 2.

In accordance with our earlier conclusions (Argay, Kálmán, Nahlovski, & Ribár, 1975), the exocyclic C(2)—S(8) double bond is somewhat polarized [1·685 (2) Å], similar bond lengths being found for example in 4-phenyl- and 1-phenylthiosemicarbazides (Kálmán, Argay & Czugler, 1972; Czugler, Kálmán & Argay, 1973), while the endocyclic C(2)—S(3) single bond – due to the delocalization of the N lone pair on

Table 1. *Final fractional coordinates and equivalent isotropic thermal parameters for non-*H *atoms, with e.s.d.'s in parentheses*

	x	y	z	B_{eq}*(Å^2)
S(3)	0·37100 (1)	0·22497 (9)	0·24335 (4)	2·93 (2)
S(8)	0·45785 (1)	0·01815 (9)	0·34878 (4)	3·02 (2)
O(9)	0·3852 (1)	0·1619 (3)	−0·0368 (1)	3·40 (5)
N(1)	0·4369 (1)	0·1904 (3)	0·1516 (1)	2·51 (5)
C(2)	0·4237 (1)	0·1473 (3)	0·2372 (2)	2·19 (5)
C(4a)	0·3776 (1)	0·4415 (3)	0·0657 (2)	2·36 (5)
C(4)	0·3420 (1)	0·3530 (3)	0·1102 (2)	2·40 (5)
C(5)	0·4107 (1)	0·5986 (4)	0·1332 (2)	3·05 (6)
C(6)	0·4508 (1)	0·5991 (5)	0·0867 (2)	4·12 (8)
C(7a)	0·4108 (1)	0·2947 (3)	0·0462 (2)	2·42 (5)
C(7)	0·4461 (1)	0·4174 (4)	0·0171 (2)	3·05 (6)
C(10)	0·3069 (1)	0·4908 (3)	0·1294 (2)	2·44 (5)
C(11)	0·2608 (1)	0·4893 (4)	0·0541 (2)	3·04 (6)
C(12)	0·2274 (1)	0·6076 (4)	0·0726 (3)	3·87 (7)
C(13)	0·2405 (1)	0·7298 (4)	0·1642 (2)	3·79 (8)
C(14)	0·2867 (1)	0·7349 (5)	0·2363 (2)	4·13 (8)
C(15)	0·3196 (1)	0·6153 (4)	0·2194 (2)	3·60 (7)

* $B_{eq} = \frac{4}{3}$ trace (B**G**) where **G** is the direct metric tensor.

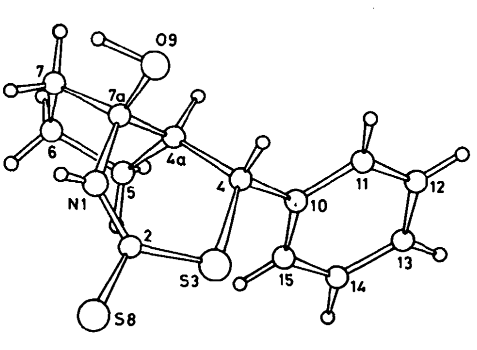


Fig. 1. A perspective view of compound (2*a*) showing the crystallographic numbering. The bare numbers are for C atoms unless indicated otherwise. The H atoms are shown but not labelled.

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

S(3)—C(2)	1·725 (1)	C(5)—C(6)	1·538 (3)
S(3)—C(4)	1·833 (2)	C(6)—C(7)	1·524 (4)
S(8)—C(2)	1·685 (2)	C(7a)—C(7)	1·520 (3)
O(9)—C(7a)	1·410 (3)	C(10)—C(11)	1·385 (3)
N(1)—C(2)	1·321 (2)	C(10)—C(15)	1·376 (3)
N(1)—C(7a)	1·479 (3)	C(11)—C(12)	1·395 (3)
C(4a)—C(4)	1·521 (2)	C(12)—C(13)	1·380 (4)
C(4a)—C(5)	1·529 (3)	C(13)—C(14)	1·375 (3)
C(4a)—C(7a)	1·524 (3)	C(14)—C(15)	1·380 (3)
C(4)—C(10)	1·523 (3)		
C(2)—S(3)—C(4)	105·1 (2)	O(9)—C(7a)—C(4a)	110·0 (3)
C(2)—N(1)—C(7a)	129·2 (3)	O(9)—C(7a)—C(7)	116·1 (3)
S(3)—C(2)—S(8)	115·7 (2)	N(1)—C(7a)—C(4a)	111·0 (3)
S(3)—C(2)—N(1)	122·0 (3)	N(1)—C(7a)—C(7)	107·5 (3)
S(8)—C(2)—N(1)	122·3 (3)	C(4a)—C(7a)—C(7)	103·2 (3)
C(4)—C(4a)—C(5)	120·0 (3)	C(6)—C(7)—C(7a)	104·9 (3)
C(4)—C(4a)—C(7a)	113·0 (3)	C(4)—C(10)—C(11)	118·8 (3)
C(5)—C(4a)—C(7a)	103·3 (3)	C(4)—C(10)—C(15)	122·0 (3)
S(3)—C(4)—C(4a)	111·6 (2)	C(11)—C(10)—C(15)	119·2 (3)
S(3)—C(4)—C(10)	107·1 (2)	C(10)—C(11)—C(12)	120·0 (4)
C(4a)—C(4)—C(10)	115·9 (3)	C(11)—C(12)—C(13)	120·0 (4)
C(4a)—C(5)—C(6)	104·1 (3)	C(12)—C(13)—C(14)	119·6 (4)
C(5)—C(6)—C(7)	107·3 (3)	C(13)—C(14)—C(15)	120·5 (4)
O(9)—C(7a)—N(1)	108·9 (3)	C(10)—C(15)—C(14)	120·6 (4)
C(4)—S(3)—C(2)—N(1)	3·1 (2)	C(5)—C(4a)—C(4)—S(3)	−64·1 (3)
C(4a)—C(4)—S(3)—C(2)	−29·1 (2)	C(7)—C(7a)—N(1)—C(2)	142·8 (3)
C(7a)—C(4a)—C(4)—S(3)	58·2 (2)		
C(4)—C(4a)—C(7a)—N(1)	−58·3 (3)	C(5)—C(4a)—C(7a)—O(9)	−166·5 (3)
C(4a)—C(7a)—N(1)—C(2)	30·6 (3)	C(4)—C(4a)—C(7a)—O(9)	62·3 (3)
C(7a)—N(1)—C(2)—S(3)	−4·3 (3)	C(2)—N(1)—C(7a)—O(9)	−90·6 (4)
		C(6)—C(7)—C(7a)—O(9)	154·0 (3)
C(6)—C(7)—C(7a)—C(4a)	33·5 (3)		
C(7a)—C(7)—C(6)—C(5)	−12·6 (3)	C(10)—C(4)—C(4a)—C(5)	59·0 (3)
C(7)—C(6)—C(5)—C(4a)	−13·1 (3)	C(10)—C(4)—S(3)—C(2)	−157·0 (3)
C(7a)—C(4a)—C(5)—C(6)	33·7 (3)		
C(7)—C(7a)—C(4a)—C(5)	−42·0 (3)		

---

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43390 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

the N(1)C(2)S(3)S(8) moiety – gains some π-bond character [1·725 (1) Å and it is accompanied by a strong N(1)—C(2) multiple bond 1·321 (2) Å]. The nearly coplanar N(1)C(2)S(3)S(8) moiety imposes an envelope (1,2-diplanar) conformation on the 1,3-thiazine ring with C(4a) at the flap. A mirror plane (C_s) indicated by the low asymmetry factor (Kálmán, Czugler & Simon, 1982) fC_s = 0·017 (4) Å [computed from the puckering parameters of Cremer & Pople (1975): Q = 0·488 (2) Å, φ = 236·8 (4) and θ = 50·3 (3)°] bisects C(2) and C(4a). In contrast with the analogous tetrahydro-1,3-oxazin-2-one and -thione derivatives fused with cyclopentane rings (Argay, Kálmán, Kapor, Stájer & Bernáth 1985; Stanković, Kapor, Ribár, Kálmán, Argay, Karanović, Stájer & Bernáth, 1985), in compound (*2a*) the *cis*-fused cyclopentane ring (Fig. 1), albeit assuming an almost perfect half-chair conformation [Q = 0·403 (3) Å, φ = 162·6 (5)°] with twofold symmetry (C_2) bisecting C(6) [fC_2 = 0·003 (4) Å for C(6)], does not shift the conformation of the hetero ring towards a C(4a)—C(7a) half chair. The least-squares plane of the phenyl ring bound *pseudoequatorially* to the hetero ring makes a dihedral angle of −62·45 (8)° with that of the N(1)-C(2)S(3)S(8) moiety. The hydroxyl group orientated *pseudoaxially* to the hetero ring forms an intermolecular hydrogen bond with S(8) at $x, -y, z-\frac{1}{2}$ with the following parameters: O···S = 3·303 (2), H···S = 2·21 (1) Å, ∠OH···S = 154 (2)°.

References

Argay, Gy., Kálmán, A., Kapor, Á., Stájer, G. & Bernáth, G. (1985). *J. Mol. Struct.* **131**, 31–43.

Argay, Gy., Kálmán, A., Nahlovski, Á. & Ribár, B. (1975). *Acta Cryst.* **B31**, 1956–1961.

Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.

Czugler, M., Kálmán, A. & Argay, Gy. (1973). *Cryst. Struct. Commun.* **2**, 655–658.

Enraf–Nonius (1982). *Structure Determination Package*. Enraf–Nonius, Delft.

International Tables for X-ray Crystallography (1962). Vol. III. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

Kálmán, A., Argay, Gy. & Czugler, M. (1972). *Cryst. Struct. Commun.* **1**, 375–378.

Kálmán, A., Czugler, M. & Simon, K. (1982). *Molecular Structures and Biological Activity*, edited by J. F. Griffin & W. L. Duax, pp. 367–376. New York: Elsevier Biomedical.

Main, P., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

Stanković, S., Kapor, Á., Ribár, B., Kálmán, A., Argay, Gy., Karanović, Lj., Stájer, G. & Bernáth, G. (1985). *J. Mol. Struct.* **131**, 45–53.

Walker, N. & Stuart, D. (1983). *Acta Cryst.* **A39**, 158–166.

Acta Cryst. (1987). C**43**, 326–329

Structure of 1,2-Dihydro-1,2,2-tris[(2-hydroxyphenyl)methyl]-3*H*-indol-3-one (Uvarindole D)*

By Michelle A. Meek and Carl H. Schwalbe†

Drug Development Research Group, Pharmaceutical Sciences Institute, Department of Pharmaceutical Sciences, Aston University, Aston Triangle, Birmingham B4 7ET, England

and Peter G. Waterman and Ilias Mohammad

Phytochemistry Research Laboratories, Department of Pharmacy (Pharmaceutical Chemistry), University of Strathclyde, Glasgow G1 1XW, Scotland

(*Received* 3 *March* 1986; *accepted* 4 *September* 1986)

Abstract. $C_{29}H_{25}NO_4$, M_r=451·53, monoclinic, $P2_1/n$, a = 14·031 (3), b = 10·031 (4), c = 16·491 (5) Å, β = 96·51 (3)°, U = 2306·1 Å^3, Z = 4, D_x = 1·301 g cm^{-3}, Mo $K\alpha$ radiation, λ = 0·71069 Å, μ = 0·49 cm^{-1}, $F(000)$ = 952, T = 293 (2) K, final R = 0·044 for 2673 unique reflections considered observed with $F_o > 3\sigma(F_o)$. Three *o*-hydroxybenzyl groups with nearly planar rings are 1,2,2-attached to the indole nucleus, which has longer than normal C(2)—C(3) and C(3)—O(3) bonds and twists of up to 15° about heterocyclic ring bonds. Exit bonds from the heterocycle take up conformations that reduce crowding. Two OH groups hydrogen-bond to the carbonyl oxygen atom, while one of these phenolic oxygen atoms also accepts a hydrogen bond from the third OH.

---

* Contribution from the Joint Crystallography Unit, Universities of Aston and Birmingham. *Chemistry in the Annonaceae*. 16. Part 15: Waterman & Mohammad (1984).

† To whom correspondence should be addressed.

0108-2701/87/020326-04$01.50