seven-membered heterocyclic ring adopts a twist-chair conformation (Toromanoff, 1980).

All internal bond angles in the cage structure are less than 109°, and eight angles are less than 100°. A molecular-mechanics calculation (Allinger & Yu, 1980) with the acid side chain at C(11) replaced with an OH group gave a heat of formation of $-$397·4 kJ mol^{-1} and a strain energy of 281·6 kJ mol^{-1} with 163·1 kJ mol^{-1} partitioned to angle strain and 121·4 kJ mol^{-1} to torsional strain. The cage structure contains a number of eclipsed bonds which leads to the large torsional contribution. In general, the bond-length pattern resembles that found experimentally; however, the differences between the calculated bond types are not as great as those observed, *i.e.* ethane bonds average 1·557 (5) Å, methano bonds 1·533 (5) Å and the pure connecting bonds 1·540 (4) Å. Librational correction of X-ray bond lengths leads to poorer agreement between calculated and observed values.

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Structure of 3-(4-Hydroxy-2-methylphenyl)-3-phenylphthalide

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Abstract. $C_{21}H_{16}O_3$, M_r = 316·36, monoclinic, $P2_1/c$, a = 7·4826 (8), b = 19·446 (2), c = 11·119 (2) Å, β = 102·041 (8)°, V = 1582·3 (4) Å^3, Z = 4, D_x = 1·328 Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, μ = 0·72 mm^{-1}, F(000) = 664, T = 293 K, final R = 0·040 for 2894 unique observed reflections. The overall conformation of the title molecule is very similar to the conformation of its isomer, 3-(2-hydroxy-4-methylphenyl)-3-phenylphthalide [Skrzat, Roszak & Engelen (1988). *Acta Cryst.* C**44**, 537–540]. The tetrahedral geometry of the central C atom is significantly distorted: valence angles range from 102·6 (1) to 116·2 (1)°. The orientation of the substituted phenyl ring relative to the other parts of the molecule is slightly different in the two isomers but their *ortho* substituents are similarly oriented. The packing is determined by an intermolecular hydrogen bond between the hydroxyl and carbonyl groups with O···O = 2·789 (2) Å and ∠O—H···O = 176 (2)°.

Introduction. The present paper is a continuation of our X-ray crystallographic studies of 3,3-diarylphthalides undertaken to confirm their chemical structure and to obtain more information on relationships between their structures and reactivity. The title compound (II) is one of the isomers obtained in the condensation of 2-benzoylbenzoic acid with *m*-cresol. Previously it was reported (Dutt, 1940; Rumiński, 1973) that only isomer (II) was obtained in this reaction. Isomer (I) [3-(2-hydroxy-4-

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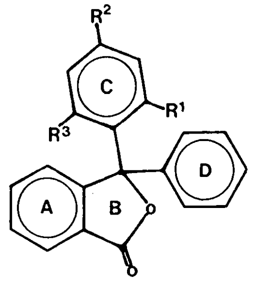
methylphenyl)-3-phenylphthalide] was also isolated and its crystal structure has been presented (Skrzat, Roszak & Engelen, 1988). The theoretically possible isomer (III) [3-(2-hydroxy-6-methylphenyl)-3-phenylphthalide] was not formed in this reaction. Isomers (I) and (II) have different melting points and only isomer (I), after reduction, undergoes facile cyclization and acetylation to form 9-acetoxy-10-(2-acetoxy-4-methylphenyl)anthracene (Gronowska, 1988). We now report the crystal structure of isomer (II).



(I) R^1 = OH, R^2 = Me, R^3 = H
(II) R^1 = Me, R^2 = OH, R^3 = H
(III) R^1 = Me, R^2 = H, R^3 = OH

Experimental. Colourless needle-shaped crystals grown from acetic acid by slow evaporation, m.p. 498·6–499·6 K, crystal specimen: 0·46 × 0·18 × 0·14 mm, Enraf–Nonius CAD-4F diffractometer, Cu radiation with Ni filter, ω–2θ scan mode, scan range $\Delta\omega = 1{\cdot}5(0{\cdot}7 + 0{\cdot}14\tan\theta)^\circ$; accurate cell parameters from least-squares refinement for 25 reflections with $30 < \theta < 40^\circ$; three standards [$\bar{5}00$: 283 (4), $\bar{2}0\bar{6}$: 156 (2), $\bar{2}8\bar{4}$: 334 (4)]; h 0/9, k 0/24, l −13/13; max. $(\sin\theta)/\lambda = 0{\cdot}6259$ Å^{-1}; 6930 reflections measured, 3191 unique reflections ($R_{\rm int} = 0{\cdot}09$), 297 unobserved reflections [$I < 2{\cdot}5\sigma(I)$]; no absorption correction.

Structure solved by direct methods with *SHELXS*86 (Sheldrick, 1985); full-matrix least squares using F magnitudes with *SHELX*76 (Sheldrick, 1976); all H atoms located on difference Fourier maps and refined isotropically; anisotropic displacement parameters for non-H atoms; 282 parameters refined.

Final $R = 0{\cdot}040$, $wR = 0{\cdot}084$ ($w^{-1} = \sigma_F^2 + 0{\cdot}0079|F|^2$) and $S = 0{\cdot}91$ for 2894 observed reflections; empirical isotropic extinction parameter x (*SHELX*76, Sheldrick, 1976) converged at 0·009 (3); max. $(\Delta/\sigma) = 0{\cdot}011$ in final cycle; max. $(\Delta\rho) = 0{\cdot}23$, min. $(\Delta\rho) = -0{\cdot}15$ e Å^{-3} in final ΔF map. Scattering factors from *International Tables for X-ray Crystallography* (1974). Other computer programs used: *ORTEP*II (Johnson, 1976), *PLUTO* (Motherwell & Clegg, 1978) and *CRYPOZ* system (Jaskólski, 1982).

Discussion. The final fractional coordinates and $U_{\rm eq}$ values for the non-H atoms of isomer (II) are given in Table 1.* Bond lengths, bond angles and selected torsion angles are listed in Table 2. A perspective view of the molecule with the atom-numbering scheme is shown in Fig. 1.

The geometry of this molecule is very similar to that of isomer (I) (Skrzat *et al.*, 1988). When the whole molecule is treated as a distorted propeller with an axis along the bond C(1)—O(1), the angles between the planes of rings *A*, *C* and *D*, and the appropriate O(1)—C(1)—C(Ph) planes are 3·5 (1), 57·2 (1) and 18·0 (1)°. The corresponding angles in isomer (I) were 4·2 (3), 64·8 (3) and 21·0 (3)°. The fused ring system is not exactly planar. The dihedral angle between rings *A* and *B* is 3·7 (1)° in isomer (II) compared with 2·8 (3)° in isomer (I).

Fig. 2 shows a superposition of isomers (I) and (II) achieved by least-squares fitting of their phthalide ring systems. The unsubstituted phenyl rings (*D*) are almost in the same position. The largest difference is observed for the substituted phenyl ring (*C*). The *ortho* substituents in the two structures have similar orientations suggesting that this position is energetically favoured for steric reasons. However, the substitution of a methyl group for the hydroxyl in the *ortho* position of the ring is associated with a significant displacement of the ring involving a 7° rotation about C(1)—C(15) and an alteration in the tetrahedral geometry about the C(1) atom. The net effect of these changes is seen in Fig. 2.

The similarity in position of the *ortho* substituent in both isomers suggests that it is an optimal position in relation to the other parts of the molecule. Rotation about C(1)—C(15) generates unfavourable van der Waals contacts to the *ortho* substituent in any orientation other than that observed in the X-ray study. The probable reason why isomer (III) does not form is that there is no conformation in which *ortho* substituents at C(2) and C(6) could be tolerated without severe strain.

A comparison of the solid-state three-dimensional structures of isomers (I) and (II) does not fully explain their different reactivity; however, a further study of this problem (Gronowska, 1988) shows that both isomers undergo reduction to intermediate compounds which in turn behave differently in the later stages of reaction. In order to explain this the X-ray structures of the intermediate compounds must be determined.

The difference in melting points (of about 11 K) between the two isomers could be the result of slightly different hydrogen-bonding schemes in the

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* 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 51977 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic fractional coordinates and equivalent isotropic displacement parameters* (Å^2) *with e.s.d.'s in parentheses*

$$U_{eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
C(1)	0·1706 (2)	0·13586 (6)	0·8604 (1)	0·0345 (3)
C(2)	0·0747 (2)	0·05223 (6)	0·7093 (1)	0·0403 (3)
C(3)	0·2369 (2)	0·08546 (6)	0·6810 (1)	0·0388 (3)
C(4)	0·2981 (2)	0·13438 (6)	0·7706 (1)	0·0357 (3)
C(5)	0·4609 (2)	0·16895 (7)	0·7733 (1)	0·0423 (3)
C(6)	0·5565 (2)	0·15386 (9)	0·6814 (1)	0·0507 (4)
C(7)	0·4903 (2)	0·10559 (9)	0·5890 (1)	0·0551 (5)
C(8)	0·3306 (2)	0·07088 (8)	0·5880 (1)	0·0501 (4)
C(9)	0·2745 (2)	0·11197 (6)	0·9870 (1)	0·0364 (3)
C(10)	0·2768 (2)	0·04345 (7)	1·0228 (1)	0·0522 (4)
C(11)	0·3750 (3)	0·02416 (9)	1·1388 (2)	0·0683 (6)
C(12)	0·4738 (3)	0·07160 (9)	1·2166 (1)	0·0586 (5)
C(13)	0·4749 (2)	0·13972 (9)	1·1805 (1)	0·0507 (4)
C(14)	0·3749 (2)	0·16023 (7)	1·0665 (1)	0·0432 (3)
C(15)	0·0723 (2)	0·20381 (6)	0·8702 (1)	0·0342 (3)
C(16)	−0·0593 (2)	0·20871 (6)	0·9440 (1)	0·0367 (3)
C(17)	−0·1301 (2)	0·27297 (7)	0·9617 (1)	0·0431 (4)
C(18)	−0·0720 (2)	0·33240 (7)	0·9108 (1)	0·0430 (3)
C(19)	0·0530 (2)	0·32748 (7)	0·8360 (1)	0·0437 (4)
C(20)	0·1223 (2)	0·26301 (7)	0·8155 (1)	0·0408 (3)
C(21)	−0·1277 (2)	0·14793 (8)	1·0054 (1)	0·0470 (4)
O(1)	0·0337 (1)	0·08317 (4)	0·80971 (9)	0·0402 (2)
O(2)	−0·0139 (2)	0·00384 (6)	0·6609 (1)	0·0552 (3)
O(3)	−0·1430 (2)	0·39346 (6)	0·9395 (1)	0·0569 (4)

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

C(1)—O(1)	1·475 (1)	C(9)—C(14)	1·396 (2)
C(1)—C(4)	1·517 (2)	C(10)—C(11)	1·396 (3)
C(1)—C(9)	1·532 (2)	C(11)—C(12)	1·371 (3)
C(1)—C(15)	1·527 (2)	C(12)—C(13)	1·389 (2)
C(2)—O(1)	1·359 (2)	C(13)—C(14)	1·389 (2)
C(2)—O(2)	1·210 (2)	C(15)—C(16)	1·410 (2)
C(2)—C(3)	1·466 (2)	C(15)—C(20)	1·389 (2)
C(3)—C(4)	1·385 (2)	C(16)—C(17)	1·388 (2)
C(3)—C(8)	1·394 (2)	C(16)—C(21)	1·507 (2)
C(4)—C(5)	1·387 (2)	C(17)—C(18)	1·396 (2)
C(6)—C(7)	1·403 (2)	C(18)—C(19)	1·379 (2)
C(7)—C(8)	1·370 (2)	C(18)—O(3)	1·366 (2)
C(5)—C(6)	1·396 (2)	C(19)—C(20)	1·393 (2)
C(9)—C(10)	1·390 (2)		

C(4)—C(1)—C(9)	109·3 (1)	C(1)—C(9)—C(10)	121·8 (1)
C(4)—C(1)—C(15)	116·2 (1)	C(1)—C(9)—C(14)	118·7 (1)
C(4)—C(1)—O(1)	102·6 (1)	C(10)—C(9)—C(14)	119·4 (1)
C(9)—C(1)—C(15)	110·9 (1)	C(9)—C(10)—C(11)	119·7 (1)
C(9)—C(1)—O(1)	108·6 (1)	C(10)—C(11)—C(12)	120·8 (1)
C(1)—O(1)—C(2)	111·3 (1)	C(12)—C(13)—C(14)	120·3 (1)
C(3)—C(2)—O(1)	108·6 (1)	C(13)—C(14)—C(9)	120·0 (1)
C(3)—C(2)—O(2)	130·4 (1)	C(1)—C(15)—C(16)	120·3 (1)
O(1)—C(2)—O(2)	120·9 (1)	C(1)—C(15)—C(20)	120·5 (1)
C(2)—C(3)—C(4)	108·2 (1)	C(16)—C(15)—C(20)	118·9 (1)
C(2)—C(3)—C(8)	129·5 (1)	C(15)—C(16)—C(17)	118·5 (1)
C(4)—C(3)—C(8)	122·1 (1)	C(15)—C(16)—C(21)	123·7 (1)
C(1)—C(4)—C(3)	109·1 (1)	C(17)—C(16)—C(21)	117·7 (1)
C(1)—C(4)—C(5)	130·3 (1)	C(16)—C(17)—C(18)	121·8 (1)
C(3)—C(4)—C(5)	120·4 (1)	C(17)—C(18)—C(19)	119·7 (1)
C(4)—C(5)—C(6)	117·7 (1)	C(17)—C(18)—O(3)	117·1 (1)
C(5)—C(6)—C(7)	121·3 (1)	C(19)—C(18)—O(3)	123·2 (1)
C(6)—C(7)—C(8)	120·7 (1)	C(18)—C(19)—C(20)	118·9 (1)
C(7)—C(8)—C(3)	117·8 (1)	C(19)—C(20)—C(15)	122·1 (1)

O(1)—C(1)—C(9)—C(10)	19·5 (1)	C(4)—C(1)—C(15)—C(16)	175·2 (1)
O(1)—C(1)—C(9)—C(14)	−162·9 (1)	C(4)—C(1)—C(15)—C(20)	−10·5 (1)
O(1)—C(1)—C(15)—C(16)	60·1 (1)	C(9)—C(1)—C(15)—C(16)	−59·3 (2)
O(1)—C(1)—C(15)—C(20)	−125·6 (1)	C(9)—C(1)—C(15)—C(20)	115·1 (1)
C(4)—C(1)—C(9)—C(10)	−91·7 (1)	C(15)—C(1)—C(9)—C(10)	138·9 (1)
C(4)—C(1)—C(9)—C(14)	85·9 (1)	C(15)—C(1)—C(9)—C(14)	−43·4 (2)

two structures caused by the different positions of the hydrogen-bond donor. The molecules of isomer (II), as in isomer (I), are linked by hydrogen bonds between the hydroxyl group, O(3)—H(O3), and the carbonyl O atom, O(2$^{\rm i}$) [(i) $-x$, $\tfrac{1}{2}+y$, $\tfrac{3}{2}-z$]. Hydrogen-bond geometry: $O_D\cdots O_A = 2{\cdot}789\,(2)$, O_D—H = 0·92 (2), $O_A\cdots$H = 1·86 (3) Å, $\angle O_D$—H$\cdots O_A$ = 176 (2)°. The molecular packing with hydrogen

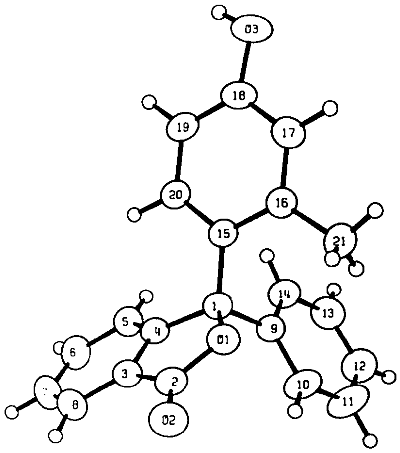

Fig. 1. Perspective view of the molecule with the numbering of non-H atoms. Thermal ellipsoids are drawn at the 50% probability level, H atoms as small spheres of arbitrary size.

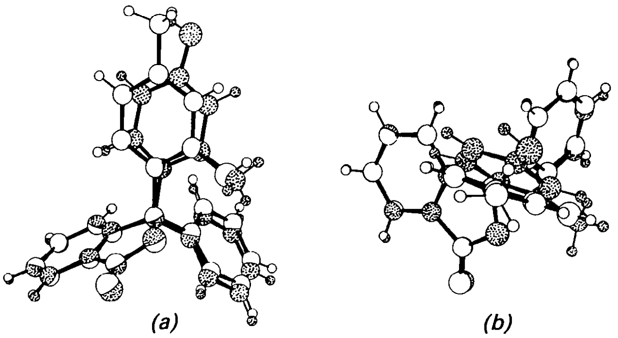
(*a*) (*b*)

Fig. 2. The superposition of the crystal conformations of the two isomers (see text) – isomer (II) represented by stippled circles: (*a*) projection similar to that used in Fig. 1; (*b*) projection along the bond C(15)—C(1) in isomer (I).

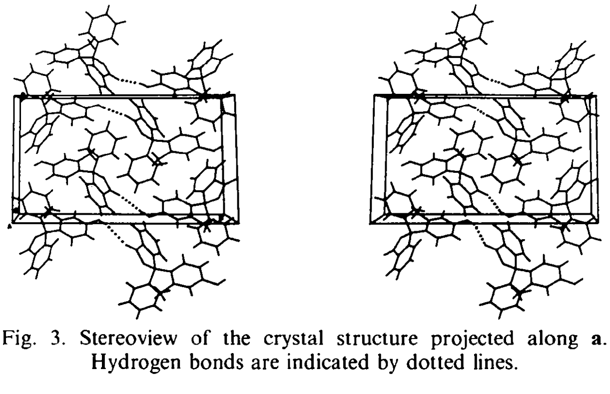

Fig. 3. Stereoview of the crystal structure projected along **a**. Hydrogen bonds are indicated by dotted lines.

bonds is shown in Fig. 3. Additionally, some short intramolecular contacts [O(1)···C(10) = 2·779 (2), O(1)···H(C10) = 2·44 (2) Å] are observed in isomer (II) which can be most probably ascribed to steric constraints in the molecule.

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Structure of 4-Triphenylmethylthio-2-azetidinone

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Abstract. $C_{22}H_{19}NOS$, $M_r = 345·5$, monoclinic, $C2/c$, $a = 27·592$ (2), $b = 8·021$ (1), $c = 16·359$ (1) Å, $\beta = 96·89$ (1)°, $V = 3594·4$ (7) Å^3, $Z = 8$, $D_m = 1·28$ (1), $D_x = 1·28$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1·54178$ Å, $\mu = 16·14$ cm^{-1}, $F(000) = 1456$, room temperature, $R = 0·033$ for 2793 reflections with $|F_o| > 3\sigma(F)$. The isolated β-lactam ring is planar to within 0·006 Å and the dimensions of the ring are similar to those found in penicillins and cephalosporins.

Introduction. The structures of β-lactam compounds with bicyclic skeletons such as penicillins and cephalosporins have been well investigated, but only a little is known about the structure of monocyclic β-lactams, especially of *N*-aryl-substituted monocyclic β-lactams whose β-lactam rings have been found to be flat (Fujiwara, Varley & van der Veen, 1977; Kartha & Ambady, 1973). It is known that aldol-type reactions between the α-position H and the β-lactam carbonyl give stereochemically different structures for bicyclic and monocyclic compounds (Yoshida, Hayashi, Takeda, Oida & Ohki, 1981; DiNinno, Beattie & Christensen, 1977), which suggests that there are reaction intermediates with different conformations for each structure.

The present work attempts to reveal details of the monocyclic β-lactam structure as part of a study of the stereochemistry in the aldol reaction of the β-lactam ring.

Experimental. The synthesized material was crystallized from dichloromethane/*n*-hexane solution. D_m by flotation in KI/H_2O. Diffraction intensities were measured from a colorless prismatic crystal 0·20 × 0·14 × 0·44 mm, in the ω–2θ scan mode with variable scan width to a maximum $2\theta = 125°$ on a Rigaku-AFC diffractometer using Ni-filtered Cu $K\alpha$ radiation. Lattice parameters and orientation matrix refined with 20 reflections in range $21 < \theta < 23°$ by least-squares method. 2934 unique reflections meas-

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