Discussion. Final fractional coordinates and equivalent B values for the title compound are listed in Table 1* and the geometry is described in Table 2. Fig. 1 shows the atom-labeling scheme of the molecule. The aminobenzyl ring is twist-skewed with respect to the quinoline ring as defined by the torsion angles C(7)–C(6)–C(11)–C(12) = 113·4 (3) and C(6)–C(11)–C(12)–C(17) = –26·4 (4)°. The *o*-amino group is distal to the quinoline ring and is protonated, as is the quinoline nitrogen atom. The indolyl ring is twisted –42·6 (4)° [C(6)–C(7)–C(19)–C(27)] from the quinoline ring. The molecule is involved in a network of hydrogen bonds formed between the hydrogen atoms of N(5), N(18), and N(26) and the ethanesulfonate oxygen atoms. The geometry of the complex is similar to that observed in other indole and ethanesulfonate structures (Andrianov, Struchkov & Babievsky, 1982; Cody & Zakrzewski, 1982). The only exception is the N(26)–C(27) indole-ring bond length [1·340 (4) Å] which is somewhat shortened when compared to the indole bond lengths [1·377 (3) Å] observed in other indole structures. This shortening may be indicative of a partial positive charge on the nitrogen atom.

This is the first reported occurrence of this unusual compound which may have been formed by the condensation of two indole molecules in the presence of the strong acid, ethanesulfonic acid, which then reacted in UV light and methanol to form the observed product.

This research was supported in part by a grant from the American Cancer Society, CH–210, and The Buffalo Foundation.

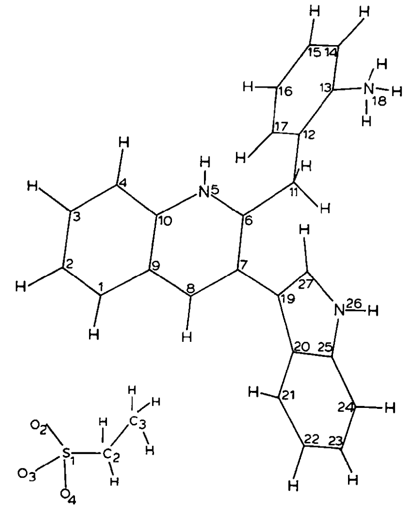

Fig. 1. Molecular structure and numbering scheme for the title compound.

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* Lists of structure factors, anisotropic thermal parameters, hydrogen-atom coordinates and intermolecular contacts have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39842 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structures of 3,8-Dihydroxy-3-methyl-3,4-dihydroisocoumarin (I), $C_{10}H_{10}O_4$, and 2-Acetonyl-6-methoxybenzoic Acid (II), $C_{11}H_{12}O_4$

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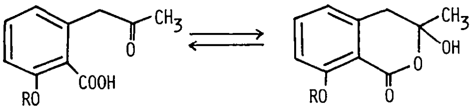
Abstract. (I): $M_r = 194·19$, monoclinic, $P2_1/c$, $a = 7·011\,(2)$, $b = 9·493\,(2)$, $c = 13·943\,(3)$ Å, $\beta = 93·87\,(2)°$, $V = 926·1\,(3)$ Å^3, $Z = 4$, $D_x = 1·39$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0·71070$ Å, $\mu = 0·10$ mm^{-1}, $F(000) = 408$, $T = 293$ K, final $R = 0·068$ for 1041 unique observed reflections. (II): $M_r = 208·22$, orthorhombic, $P2_12_12_1$, $a = 12·954\,(12)$, $b = 15·043\,(9)$, $c = 5·218\,(1)$ Å, $V = 1016·9\,(11)$ Å^3, Z

$= 4$, $D_x = 1{\cdot}36$ Mg m^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}71070$ Å, $\mu = 0{\cdot}10$ mm^{-1}, $F(000) = 440$, $T = 293$ K, final $R = 0{\cdot}065$ for 788 unique reflections. The chemical structures of (I) and (II) in the crystals have been established as the lactol and keto acid forms, respectively. There are no unusual bond distances or angles. Molecules of both (I) and (II) are held together mainly by hydrogen bonds.

Introduction. In the course of the synthesis of penioactol, the fungal metabolite isolated from *Peniophora sanguinea* Bres. (Gripenberg, 1974), 2-acetonylbenzoic acids were synthesized as model compounds in order to study the tautomerism between keto acid forms (I*a* and II*a*) and lactol forms (I*b* and II*b*). Compound (I) has been shown to exist in the lactol form (I*b*) in organic solvents predominantly on the basis of the proton magnetic resonance spectrum, while compound (II) exists mainly in the keto acid form (II*a*) (Kameda, Aoki, Tanaka & Namiki, 1973). The structures of (I) and (II) in the solid state could be determined from the infrared spectra, but the absorption band of carbonyl was too complicated to determine the structures. Thus X-ray structure analyses of (I) and (II) have been undertaken in order to establish the crystal structures.



(Ia) R = H
(IIa) R = CH3

(Ib) R = H
(IIb) R = CH3

Experimental. Title compounds prepared from 3-methoxyhomophthalic acid. Both crystals grown from methanol as colorless prisms, m.p. 371 K for (I), 430 K for (II). D_m not measured. Single crystals $0{\cdot}1 \times 0{\cdot}2 \times 0{\cdot}4$ mm and $0{\cdot}15 \times 0{\cdot}2 \times 0{\cdot}3$ mm for (I) and (II). Rigaku AFC-5FOS diffractometer, graphite-monochromated Mo $K\alpha$ radiation. Cell parameters from setting angles of 24 reflections with $13° < \theta < 18°$ for (I), from those of 16 reflections with $9° < \theta < 13°$ for (II). No corrections for absorption. 1922 independent reflections measured within $2\theta = 53°$, range of *hkl*: h $-8\rightarrow8$, k $0\rightarrow11$, l $0\rightarrow17$, $\omega/2\theta$ scan mode, 1041 with $F \geq 2\sigma(F)$ for (I). 1100 independent reflections measured within $2\theta = 50°$, range of *hkl*: h $0\rightarrow15$, k $0\rightarrow17$, l $0\rightarrow6$, $\omega/2\theta$ scan mode, 788 with $F \geq 2\sigma(F)$ for (II). Three standard reflections checked every 100 reflections, no significant deviation. Structure solved by direct methods (*MULTAN*, Germain, Main & Woolfson, 1970) and refined (on F) by block-diagonal least squares. H atoms from difference map. Refinement with anisotropic temperature factors for non-hydrogen atoms and isotropic thermal parameters for H atoms. $R = 0{\cdot}0680$, $R_w = 0{\cdot}0657$, $w = 1$, $(\Delta/\sigma)_{\max} = 0{\cdot}77$ ($0{\cdot}29$ for non-H atoms), $S = 1{\cdot}13$, largest peak in final ΔF map $0{\cdot}15$ e Å^{-3} for (I). $R = 0{\cdot}0653$, $R_w = 0{\cdot}0635$, $w = 1$, $(\Delta/\sigma)_{\max} = 0{\cdot}89$, $S = 1{\cdot}05$, largest peak in final ΔF map $0{\cdot}13$ e Å^{-3} for (II).*

Table 1. *Atomic positional parameters* ($\times 10^4$) *and equivalent isotropic thermal parameters* (Å^2) *for* (I) *and* (II) *with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{4}{3}\textstyle\sum_i \sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Compound (I)				
C(1)	3989 (7)	2097 (5)	4795 (3)	3·0
C(3)	7415 (7)	2050 (6)	5218 (3)	3·1
C(4)	7517 (7)	3578 (6)	4907 (4)	3·8
C(4a)	6061 (7)	3902 (5)	4108 (3)	3·3
C(5)	6328 (9)	4931 (6)	3424 (4)	4·9
C(6)	4872 (11)	5209 (7)	2722 (4)	5·9
C(7)	3166 (9)	4496 (7)	2690 (4)	5·0
C(8)	2869 (8)	3480 (6)	3357 (3)	3·7
C(8a)	4330 (7)	3175 (5)	4075 (3)	2·9
C(9)	8522 (8)	1748 (7)	6157 (4)	4·3
O(1)	2430 (5)	1560 (4)	4871 (3)	4·1
O(2)	5408 (5)	1712 (4)	5421 (2)	3·4
O(3)	7903 (5)	1215 (4)	4466 (2)	3·4
O(8)	1165 (6)	2819 (5)	3293 (3)	5·2
Compound (II)				
C(1)	4899 (4)	813 (4)	4813 (13)	2·6
C(2)	4130 (5)	667 (4)	3055 (14)	3·2
C(3)	3707 (5)	−193 (5)	2842 (17)	4·2
C(4)	4038 (6)	−842 (4)	4522 (18)	4·9
C(5)	4771 (5)	−686 (4)	6347 (16)	4·3
C(6)	5207 (5)	144 (4)	6515 (14)	3·3
C(7)	5391 (5)	1713 (4)	4997 (13)	2·9
C(8)	3806 (5)	1378 (4)	1215 (15)	4·0
C(9)	3056 (5)	2050 (4)	2336 (14)	3·3
C(10)	2955 (7)	2871 (5)	749 (17)	5·1
C(11)	6349 (6)	−274 (5)	9938 (17)	5·2
O(6)	5944 (4)	376 (3)	8244 (10)	4·7
O(9)	2561 (4)	1905 (3)	4209 (11)	4·4
O(71)	6368 (4)	1709 (3)	4367 (11)	4·7
O(72)	4947 (4)	2363 (3)	5589 (17)	7·2

Discussion. The final atomic parameters for (I) and (II) are listed in Table 1. The structures of (I) and (II) in the crystals are shown to be the same as those present in organic solvents. The molecular structures are shown in Figs. 1 and 2. The bond lengths and angles for (I) and (II) are also given in Figs. 1 and 2. These values are not different from the expected ones. The mean standard deviations of the bond lengths and angles are estimated as: σ(C—C or C—O) $= 0{\cdot}010$ and σ(C—H or O—H) $= 0{\cdot}056$ Å, and σ(C—C—C, C—C—O or C—O—C) $= 0{\cdot}7$, σ(C—C—H or C—O—H) $= 3{\cdot}4$ and σ(H—C—H) $= 4{\cdot}1°$ for (I). These are estimated as: σ(C—C or C—O) $= 0{\cdot}009$ and σ(C—H or O—H) $= 0{\cdot}074$ Å, and σ(C—C—C, C—C—O, C—O—C or O—C—O) $= 0{\cdot}6$, σ(C—C—H, C—O—H or O—C—H) $= 4{\cdot}3$ and σ(H—C—H) $= 6{\cdot}1°$ for (II).

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* Lists of structure factors, anisotropic temperature factors and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39868 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

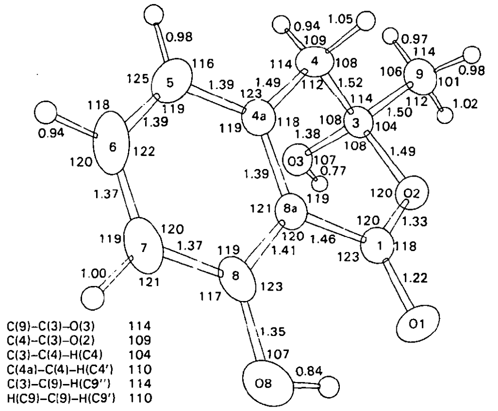

Fig. 1. The molecular structure of (I). Bond lengths (Å) and angles (°) are shown.

The dihydropyran ring of (I) adopts a half-chair form and the hydroxyl group at C(3) is in an axial conformation. Intramolecular hydrogen bonds may be formed between O(1) and H(O8)–O(8) (2·607 Å for O—O and 1·868 Å for O—H). The non-bonding distance of 3·017 Å found between C(9) and O(72) in the molecule of (II) is shorter than that expected. This will cause ring formation at C(9) by attacking the carbonyl group. The aromatic rings of (I) and (II) are almost planar as expected. The molecules are packed together mainly by the hydrogen bonding in both crystals. Non-bonding distances of 2·808 and 2·669 Å are found between O(1) and O(3) in (I) and O(9) and O(71) in (II), respectively.

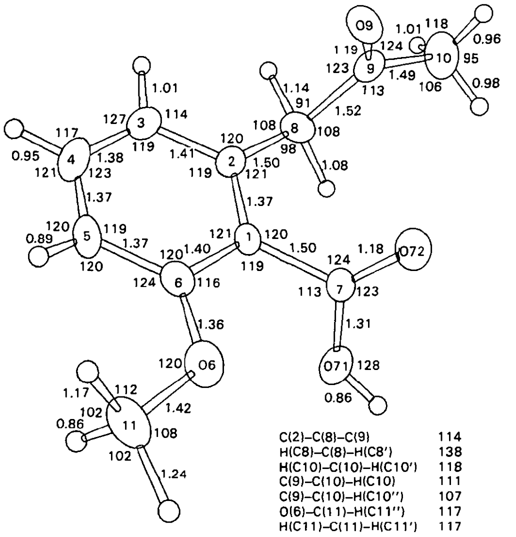

Fig. 2. The molecular structure of (II). Bond lengths (Å) and angles (°) are shown.

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Structure and Conformation of Two Diastereomers of (±)-1-(2,3-Dichlorophenyl)-3,5,6,7-tetrahydro-3-hydroxy-3,6,6-trimethyl-1*H*-indole-2,4-dione, $C_{17}H_{17}Cl_2NO_3$

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Abstract. The two diastereomers A and B of the title compound ($M_r = 354{\cdot}2$) crystallized in two different crystal systems: (A) monoclinic, $P2_1/c$, $a = 9{\cdot}319$ (1), $b = 16{\cdot}098$ (2), $c = 12{\cdot}013$ (1) Å, $\beta = 109{\cdot}11$ (1)°, $V = 1703$ Å^3, $Z = 4$, $D_x = 1{\cdot}381$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 3{\cdot}603$ mm^{-1}, $F(000) = 736$, room temperature, $R = 0{\cdot}049$ for 2889 observed reflections; (B) triclinic, $P\bar{1}$, $a = 8{\cdot}861$ (1), $b = 9{\cdot}855$ (1), $c = 11{\cdot}020$ (1) Å, $\alpha = 115{\cdot}47$ (1), $\beta = 103{\cdot}05$ (1), $\gamma = 88{\cdot}13$ (1)°, $V = 844$ Å^3, $Z = 2$, $D_x = 1{\cdot}394$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 3{\cdot}636$ mm^{-1}, $F(000) = 368$, room temperature, $R = 0{\cdot}050$ for 3057 observed reflections. The two structures show that diastereomer A has the OH at the asymmetric carbon on the same side as the Cl in the phenyl ring while B has the CH_3 at the asymmetric carbon on the same side as the Cl. In A,

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