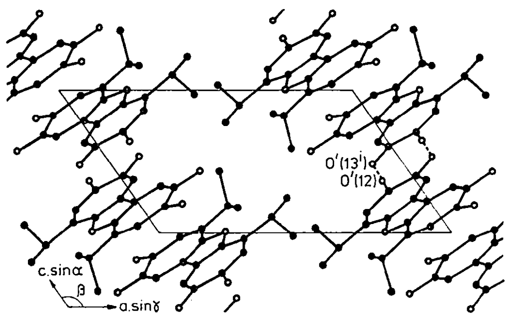

Fig. 3. The unit cell in projection along the *b* axis. Intermolecular hydrogen bonds are marked with dotted lines. ● Carbon. ○ Oxygen.

Intramolecular hydrogen bonds are observed in each tropolone moiety in utahin. The distances O(12)–O(13) and O′(12)–O′(13) are 2·510 and 2·586 Å respectively. H atoms are found at normal X-ray-measured bond distances (0·82 and 0·82 Å) from O(13) and O′(13) and at 1·94 and 2·11 Å, respectively, from O(12) and O′(12). The angles O(13)–H(O13)···O(12) and O′(13)–H(O′13)···O′(12) are 126 and 117° respectively. However, only the primed parts of the molecules are involved in intermolecular hydrogen bonding to form hydrogen-bonded dimers of centrosymmetrically related molecules. The intermolecular distance H(O′13)···O′(12^i) ($i \equiv 2-x, -y, 1-z$) is 2·04 Å and the angle O′(13)–H(O′13)···O′(12^i) is 152°. The distance O′(13)···O′(12^i) is 2·793 Å. Other intermolecular contacts are normal van der Waals distances. The crystal packing projected along the *b* axis is shown in Fig. 3.

The present investigation has received financial support from the Swedish Natural Science Research Council. The authors express their gratitude to Professor Peder Kierkegaard for his active and stimulating interest in this work. Professor Torbjörn Norin has kindly supplied the crystals used in the work. The authors also wish to thank Dr Don Koenig who corrected the English of this paper.

References

Baggaley, K. H. & Norin, T. (1968). *Chem Commun.* pp. 233–234.
Berg, J.-E., Karlsson, B., Pilotti, A.-M. & Wiehager, A.-C. (1976). *Acta Cryst.* **B32**, 3121–3123.
Derry, J. E. & Hamor, T. A. (1972). *J. Chem. Soc.* pp. 694–697.
Dewar, M. J. S. & Trinajstic, N. (1970). *Croat. Chem. Acta*, **42**, 1–12.
Freeman, A. J. (1959). *Acta Cryst.* **12**, 261–271.
Germain, G., Main, P. & Woolfson, M. M. (1970). *Acta Cryst.* **B26**, 274–285.
Hughes, E. W. (1941). *J. Amer. Chem. Soc.* **63**, 1737–1752.
Karlsson, B., Pilotti, A.-M. & Wiehager, A.-C. (1973). *Acta Cryst.* **B29**, 1209–1213.
Shimanouchi, H. & Sasada, Y. (1973). *Acta Cryst.* **B29**, 81–90.
Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
Wilson, A. J. C. (1942). *Nature, Lond.* **150**, 151–152.

Acta Cryst. (1976). **B32**, 3121

5-Isopropyltropolone, γ-Thujaplicin

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Abstract. $C_{10}H_{12}O_2$, monoclinic, $P2_1/n$, $a=7{\cdot}573$ (2), $b=18{\cdot}311$ (5), $c=6{\cdot}632$ (2) Å, $\beta=105{\cdot}1$ (2)°, $Z=4$, $V=887{\cdot}9$ Å^3, $D_c=1{\cdot}227$ g cm^{-3}. The final R value is 0·064 for 1078 observed independent reflexions. The π-electron system in the tropolone moiety is only partially delocalized with some double-bond localization both for C–C and C–O bonds. The hydroxyl H participates in a bifurcated hydrogen bond, of which one branch is intramolecular and the other intermolecular.

Introduction. Intensities were obtained from two crystals with approximate volumes 0·0045 and 0·002 mm^3 on a computer-controlled diffractometer (Philips PW 1100, graphite monochromator, Cu $K\alpha$ radiation, $\omega/2\theta$ scan, stationary background measurements at the beginning and end of each scan). 1695 unique reflexions up to $\theta=70°$ were measured. Of these, the 1078 satisfying the condition $\sigma(I)/I \leq 0{\cdot}25$ were used for subsequent refinement. During the data collection three monitor reflexions measured at intervals of approximately 1·5 h showed a decrease in intensity of 10–15%, from both crystals. Individual reflexions were corrected for this loss by fitting a linear function of time to the intensities of the monitor reflexions.

The cell constants were refined by least squares from diffractometer-measured settings of 25 reflexions. The structure was solved by the multisolution tangent-formula refinement (Germain, Main & Woolfson, 1971)

Table 1. *Positional and anisotropic thermal parameters of the non-hydrogen atoms*

The βvalues refer to the temperature-factor expression: exp $[-(h^2\beta_{11}+k^2\beta_{22}+l^2\beta_{33}+hk\beta_{12}+hl\beta_{13}+kl\beta_{23})]$. Estimated standard deviations are given in parentheses. Values are $\times 10^4$.

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C(1)	7072 (4)	507 (2)	7936 (5)	163 (6)	28 (1)	209 (12)	22 (4)	101 (12)	17 (5)
C(2)	8106 (4)	699 (2)	6441 (5)	128 (6)	31 (1)	231 (13)	−3 (4)	106 (12)	−4 (5)
C(3)	7498 (4)	1038 (2)	4558 (5)	145 (6)	39 (2)	257 (13)	−8 (5)	169 (14)	20 (6)
C(4)	5763 (5)	1311 (2)	3516 (5)	204 (7)	31 (1)	225 (12)	15 (5)	162 (14)	40 (5)
C(5)	4134 (4)	1292 (2)	4063 (5)	165 (6)	25 (1)	208 (12)	16 (4)	114 (12)	0 (4)
C(6)	3927 (4)	944 (2)	5892 (5)	135 (6)	32 (1)	245 (13)	14 (4)	146 (12)	10 (5)
C(7)	5137 (4)	614 (2)	7496 (5)	163 (6)	33 (1)	229 (12)	22 (4)	188 (13)	40 (5)
O(8)	7929 (3)	218 (2)	9606 (4)	199 (5)	65 (2)	281 (12)	76 (4)	150 (12)	96 (5)
O(9)	9871 (3)	508 (2)	7024 (4)	136 (5)	66 (2)	286 (12)	24 (4)	128 (11)	53 (5)
C(10)	2401 (5)	1638 (2)	2725 (5)	205 (7)	39 (2)	252 (13)	51 (5)	121 (4)	24 (6)
C(11)	2425 (7)	1831 (4)	515 (7)	302 (11)	64 (3)	284 (15)	109 (8)	108 (19)	54 (8)
C(12)	1831 (7)	2290 (3)	3858 (8)	257 (10)	45 (2)	352 (15)	88 (6)	132 (19)	0 (8)

of 140 reflexions with $|E| \geq 1.60$ and refined by least-squares analysis with the weighting scheme of Hughes (1941). Table 1 lists the final coordinates and temperature factors of the C and O atoms. Positions of the H atoms are given in Table 2. The final R value was 0·064.* The atomic scattering factors used for non-H atoms are those of Freeman (1959); those of Stewart, Davidson & Simpson (1965) were used for the H atoms.

Table 2. *Positional* ($\times 10^3$) *and isotropic thermal* ($\times 10^2$) *parameters of the hydrogen atoms with estimated standard deviations in parentheses*

	x	y	z	B (Å^2)
H(C3)	835 (5)	105 (2)	383 (5)	348
H(C4)	577 (4)	154 (2)	235 (5)	361
H(C6)	266 (5)	91 (2)	589 (5)	321
H(C7)	469 (4)	42 (2)	852 (5)	319
H(O9)	1016 (5)	33 (2)	829 (7)	526
H(C10)	138 (5)	124 (2)	243 (5)	422
H1(C11)	282 (6)	138 (3)	989 (7)	637
H2(C11)	118 (6)	200 (3)	954 (6)	637
H3(C11)	342 (6)	228 (3)	89 (6)	637
H1(C12)	177 (5)	219 (2)	523 (7)	536
H2(C12)	77 (6)	252 (2)	304 (6)	536
H3(C12)	768 (6)	236 (2)	896 (6)	536

Discussion. The molecular structure is shown in Fig. 1. Bond lengths and angles are shown in Fig. 2. Standard deviations are estimated to be 0·004 Å and 0·3° respectively, but somewhat larger values of about 0·006 Å and 0·4° are expected for the bonds involving atoms C(10), C(11) and C(12).

γ-Thujaplicin exhibits bond-length alternation in the seven-membered ring, as do the related compounds tropolone (Shimanouchi & Sasada, 1973), β-thujaplicin (Derry & Hamor, 1972), chanootin (Karlsson, Pilotti & Wiehager, 1973) and utahin (Karlsson, Pilotti & Wiehager, 1976). The π-electron system in the tropolone moiety is only partially delocalized with some double-bond fixation both for C–C and C–O bonds. The C(1)–C(2) bond length (1·458 Å) is, however, significantly longer than the other C–C 'single' bonds (mean value 1·418 Å), *i.e.* this bond is only to a small extent included in the partial π-electron delocalization.

The tropolone system is not strictly planar (Table 3). The atoms are out of the mean plane by amounts that are large relative to the positional standard deviations.

Table 3. *Deviations* (Å) *of atoms from the least-squares plane through the seven-membered ring* C(1)–C(7)

Equation of plane: $0.2000m+0.8817n+0.4274p=3.461$ (where $m\|a^*$, $n\|b$, and $p\|c$).

C(1)	0·044	C(5)	0·032	O(9)	−0·657
C(2)	−0·006	C(6)	−0·024	C(10)	0·104
C(3)	−0·029	C(7)	−0·027	C(11)	−0·209
C(4)	0·009	O(8)	0·104	C(12)	1·443

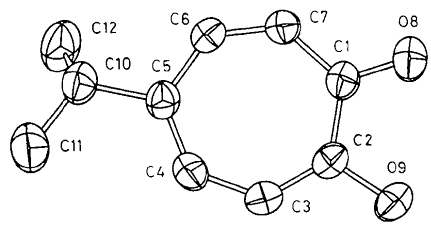
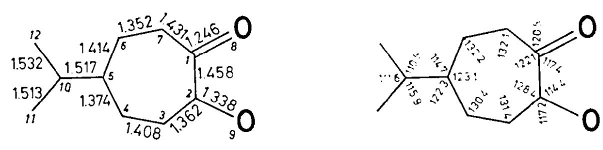


Fig. 1. A perspective view of the molecule.



(*a*) (*b*)

Fig. 2. (*a*) Bond distances in the molecule. (*b*) Bond angles in the molecule.

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31883 (3 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

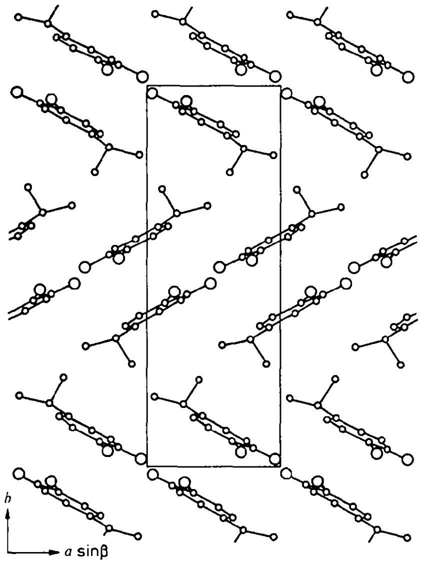


Fig. 3. The unit cell in projection along the c axis. ○ Carbon. ○ Oxygen.

The interior angles of the ring range from 122·1 to 132·2° (mean value 128·5°). The smallest angle is at the carbonyl C atom. The conformation of the isopropyl group relative to the seven-membered ring is given in Table 3 and torsion angles in Table 4.

Table 4. *Torsion angles (°) for the isopropyl group*

C(4)–C(5)–C(10)–C(11)	15·3
C(4)–C(5)–C(10)–C(12)	−113·1
C(6)–C(5)–C(10)–C(11)	−164·1
C(6)–C(5)–C(10)–C(12)	67·6

The crystal packing is shown in Fig. 3 projected along the c axis. Two molecules, related to each other by a centre of symmetry, at (0,0,0) and $(\frac{1}{2},\frac{1}{2},\frac{1}{2})$ form hydrogen-bonded dimers. The hydroxyl H participates in a bifurcated hydrogen-bonding system, of which one branch is intramolecular and the other intermolecular. A H atom is at the normal bond distance (0·88 Å) from O(9) and 2·11 Å from O(8). The angle O(9)–H(O9)···O(8) is 114°. The intermolecular distance H(O9)···O(8^{i}) ($\mathrm{i} \equiv 2-x, -y, 2-z$) is 2·0 Å and the angle O(9)–H(O9)···O(8^{i}) is 144°. The distance O(9)···O(8^{i}) is 2·76 Å. Other intermolecular contacts are normal van der Waals distances.

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References

Derry, J. E. & Hamor, T. A. (1972). *J. Chem. Soc.* pp. 694–697.

Freeman, A. J. (1959). *Acta Cryst.* **12**, 261–271.

Germain, G., Main, P. & Woolfson, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

Hughes, E. W. (1941). *J. Amer. Chem. Soc.* **63**, 1737–1752.

Karlsson, B., Pilotti, A.-M. & Wiehager, A.-C. (1973). *Acta Cryst.* B**29**, 1209–1213.

Karlsson, B., Pilotti, A.-M. & Wiehager, A.-C. (1976). *Acta Cryst.* B**32**, 3118–3121.

Shimanouchi, H. & Sasada, Y. (1973). *Acta Cryst.* B**29**, 81–90.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.