SHORT STRUCTURAL PAPERS

Papers intended for publication under this heading must be in the format prescribed in Notes for Authors, *Acta Cryst.* (1973). B**29**, 154.

Acta Cryst. (1975). B**31**, 1476

The Crystal and Molecular Structure of Phomenone

BY CLAUDE RICHE AND CLAUDINE PASCARD-BILLY

Institut de Chimie des Substances Naturelles, CNRS, 91190-Gif/Yvette, France

(*Received* 10 *January* 1975; *accepted* 11 *January* 1975)

Abstract. $C_{15}H_{20}O_4$, orthorhombic, $P2_12_12_1$, $a=9{\cdot}478$ (9), $b=10{\cdot}130$ (9), $c=14{\cdot}833$ (12) Å, $Z=4$. Final $R=0{\cdot}072$.

Introduction. Preliminary photographs showed the crystal to be orthorhombic, space group $P2_12_12_1$. A crystal with dimensions $0{\cdot}2\times0{\cdot}3\times0{\cdot}5$ mm was used to measure the cell parameters and intensities on a Philips PW 1100 four-circle automatic diffractometer. The $\omega/2\theta$-scan method was employed (scan speed $0{\cdot}02°\ s^{-1}$, scan width $1°+0{\cdot}16°\tan\theta$). Background was measured for 5 s on either side of the peak.

Data were collected to a maximum θ of 68° with graphite-monochromated Cu $K\alpha$ radiation ($\lambda=1{\cdot}5418$ Å).

Of the 1504 independent measured reflexions 1117 had intensities above $2\sigma(I)$ where $\sigma(I)$ is the standard deviation derived from counting statistics. No correction for absorption was made.

The structure was solved by a routine application of the phase function (Riche, 1973) with the symbolic addition procedure (Karle & Karle, 1966). The computer program *DEVIN* (Riche, 1973) was used. All non-hydrogen atoms appeared on the first E map.

The atomic positions and anisotropic thermal parameters of the carbon and oxygen atoms were refined by the full-matrix least-squares method (Busing, Martin & Levy, 1962); the function minimized was $\sum(\omega\Delta F)^2$ where $\omega=1/\sigma(F_o)^2$.

A difference synthesis gave the location of 14 of the hydrogen atoms. Those attached to the methyl C(15), hydroxyls O(18) and O(19) and carbon C(12) could not be located.

The contributions of the hydrogen atoms were included in the refinement with a C–H distance of 1·0 Å, but the positions were not refined. The final R value

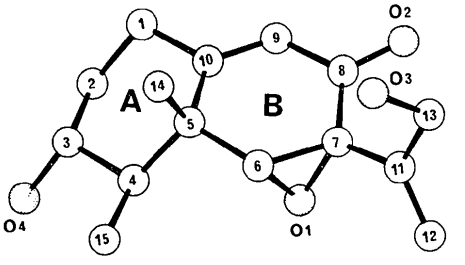


Fig. 1. Molecular structure and atom numbering.

Table 1. *The atomic parameters* ($\times10^4$) *and their e.s.d.'s* (*in parentheses*)

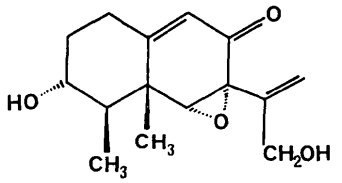
Thermal parameters are in the form $\exp[-(\beta_{11}h^2+\beta_{22}h^2+\beta_{33}h^2+2\beta_{12}hk+2\beta_{13}hl+2\beta_{23}kl)$.

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C(1)	8849 (6)	6497 (6)	2004 (4)	160 (10)	123 (8)	40 (3)	−6 (7)	6 (5)	17 (4)
C(2)	8713 (6)	6994 (6)	2972 (3)	150 (10)	114 (7)	40 (3)	−35 (7)	−17 (5)	15 (4)
C(3)	8016 (6)	5946 (6)	3547 (3)	131 (10)	111 (7)	27 (3)	11 (7)	2 (4)	3 (4)
C(4)	6577 (7)	5536 (5)	3194 (3)	165 (11)	76 (6)	30 (3)	18 (7)	−3 (5)	−1 (4)
C(5)	6628 (6)	5129 (5)	2178 (3)	170 (10)	53 (6)	26 (3)	21 (7)	13 (5)	2 (3)
C(6)	5117 (7)	5007 (5)	1870 (3)	195 (10)	76 (6)	18 (2)	13 (7)	3 (4)	−9 (3)
C(7)	4556 (7)	5577 (5)	1005 (4)	165 (11)	61 (6)	38 (3)	41 (7)	19 (5)	19 (4)
C(8)	5612 (7)	6348 (5)	476 (4)	219 (13)	71 (6)	34 (3)	18 (8)	−2 (5)	8 (4)
C(9)	6966 (6)	6661 (5)	886 (3)	141 (9)	92 (7)	39 (3)	−17 (7)	8 (5)	14 (4)
C(10)	7435 (6)	6144 (5)	1643 (3)	154 (10)	71 (7)	26 (3)	1 (7)	6 (4)	4 (4)
C(11)	3297 (7)	5033 (5)	556 (3)	159 (10)	101 (7)	33 (3)	−27 (8)	−19 (5)	17 (4)
C(12)	1972 (7)	5352 (6)	842 (4)	145 (11)	151 (9)	74 (4)	22 (9)	10 (7)	24 (6)
C(13)	3538 (7)	4117 (6)	−252 (3)	169 (11)	128 (8)	38 (3)	−30 (8)	−11 (5)	−1 (4)
C(14)	7308 (7)	3758 (5)	2043 (3)	220 (11)	98 (8)	43 (3)	24 (9)	2 (5)	−4 (4)
C(15)	5912 (7)	4467 (6)	3812 (3)	233 (12)	133 (8)	30 (3)	−39 (9)	26 (5)	21 (4)
O(1)	4285 (4)	6199 (4)	1876 (2)	177 (7)	100 (5)	36 (2)	36 (5)	5 (3)	−11 (3)
O(2)	5323 (5)	6699 (4)	−308 (2)	239 (8)	120 (5)	45 (2)	−5 (6)	−26 (4)	29 (3)
O(3)	4377 (4)	3016 (3)	−13 (3)	179 (7)	101 (5)	57 (2)	25 (5)	−6 (4)	−4 (3)
O(4)	7849 (4)	6434 (3)	4469 (2)	184 (7)	111 (5)	28 (2)	5 (5)	13 (3)	−6 (2)

was 0·072 (weighted R: 0·058). The scattering factors used for carbon and oxygen are those of Doyle & Turner (1968) and for hydrogen, those of Stewart, Davidson & Simpson (1965).

Atomic coordinates and temperature factors are listed in Table 1.*

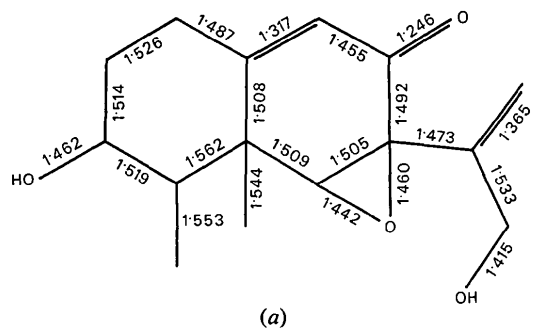
Discussion. Phomenone was extracted from cultures of *Phoma exigua* var. *non oxydabilis*. As no suitable structure could be proposed for this compound by chemical physical means, the planar formula and its relative configuration:



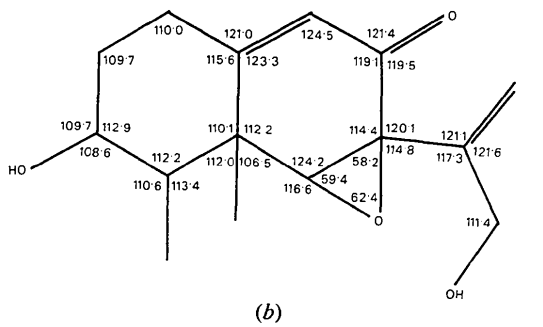
have been uniquely established by this X-ray crystal-structure determination (Riche, Pascard-Billy, Devys, Gaudemer, Barbier & Bousquet, 1974).

The molecule and the atom numbering are shown in Fig. 1. Bond lengths, interbonded angles and principal torsional angles are reported in Fig. 2. Cycle A is in a chair conformation. Cycle B is nearly planar and boat shaped. This cycle is puckered along C(5)–C(8) with a dihedral angle of 167°. The angle between the normal to the epoxy ring and the plane determined by C(5)–C(6)–C(7)–C(8) is 78°.

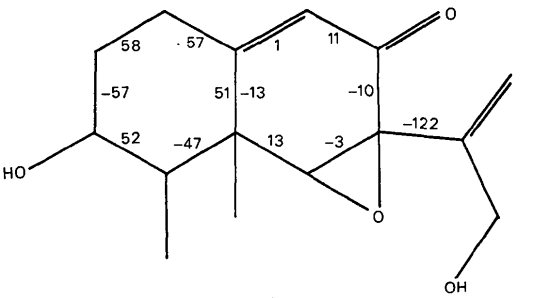
There is no intramolecular hydrogen bond but in the crystal a molecule is linked to two different symmetry-related molecules through two hydrogen bonds: O(3)···O′(4): 2·79 Å and O(3)···O″(4): 2·77 Å.



(*a*)



(*b*)



(*c*)

Fig. 2. (*a*) Bond distances, (*b*) angles [C(4)–C(5)–C(6) 106·5; C(10)–C(5)–C(14) 109·5; C(6)–C(7)–C(11) 121·9; C(8)–C(7)–C(16) 110·9] and (*c*) torsional angles. Standard deviations in bond lengths are ±0·008 Å, in bond angles 0·5°.

* A list of observed and calculated structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30861 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

References

Busing, W. R., Martin, K. O. & Levy, H. A. (1962). *ORFLS*. Oak Ridge National Laboratory Report ORNL-TM-305.

Doyle, P. A. & Turner, P. S. (1968). *Acta Cryst.* A**24**, 390–397.

Karle, I. L. & Karle, J. (1966). *Acta Cryst.* **20**, 849–859.

Riche, C. (1973). *Acta Cryst.* A**29**, 133–137.

Riche, C., Pascard-Billy, C., Devys, M., Gaudemer, A., Barbier, M. & Bousquet, J.-F. (1974). *Tetrahedron Lett.* **32**, 2765–2766.

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