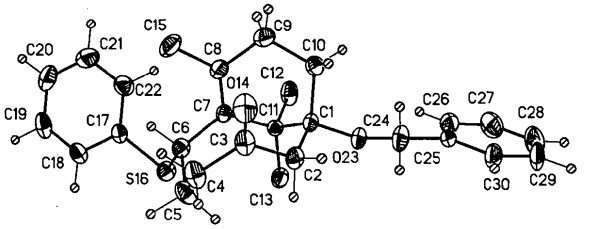

Fig. 1. View of (1) showing the atom-labeling scheme and illustrating the boat–chair conformation of the cyclooctane ring. The methyl H atoms were omitted for clarity. The non-H atoms are scaled to the 30% probability level while the H atoms are drawn to an arbitrary size.

Related literature. The structures of three molecules having the [5.3.1]undecene ring system showing similar distortions at the bridgehead alkene moiety have been reported (Lynch, Fishpaugh, Martin & Davis, 1990; Lynch, Tanaka, Fishpaugh, Martin & Davis, 1990; Lynch, Assercq, Martin & Davis, 1990).

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Structure of Phomozin: Ester of Dimethylglyceric Acid and *O*-Orsellinic Acid. A New Phytotoxin from the Phytopathogenic Fungus *Phomopsis helianthi*

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Abstract. 2,3-Dimethyl-3-(*o*-orsellinoyl)lactic acid, $C_{13}H_{16}O_7.H_2O$, $M_r = 302{\cdot}28$, triclinic, $P\bar{1}$, $a = 6{\cdot}465$ (1), $b = 6{\cdot}801$ (1), $c = 16{\cdot}951$ (4) Å, $\alpha = 89{\cdot}97$ (2), $\beta = 88{\cdot}00$ (2), $\gamma = 76{\cdot}54$ (2)°, $V = 724{\cdot}4$ (3) Å^3, $Z = 2$, $D_x = 1{\cdot}39$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}54178$ Å, $\mu = 10{\cdot}1$ cm^{-1}, $F(000) = 320$, $T = 291$ K, $R = 0{\cdot}040$ for 3315 observed reflections. A new phytotoxin, phomozin, isolated from culture filtrates of *Phomopsis helianthi* [Muntanola-Cvetkovic, Mihaljcevic & Petrov (1981). *Nova Hedwigia Z. Kryptogamenkd.* **34**, 417–435] was purified by high-pressure liquid chromatography using both liquid–solid and ion-pair liquid chromatographies. The two molecules have identical configurations. Intramolecular hydrogen bonds appear between O(15)···O(17) and O(18)···O(19).

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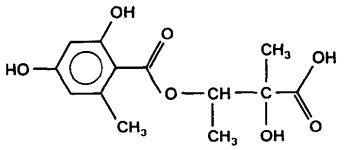
Experimental. Crystals of phomozin (1) obtained by evaporation from dichloromethane. D_m not measured. Parallelepiped crystal with dimensions 0·37 × 0·30 × 0·04 mm. Lattice parameters refined using 15



reflections in the range $13 \le 2\theta \le 27°$. Huber 4-circle diffractometer and Rigaku rotating-anode generator, graphite-monochromatized Cu $K\alpha$ radiation. 4141 independent reflections ($-7 \le h \le 6$, $-7 \le k \le 7$, $-20 \le l \le 18$) with $(\sin\theta)/\lambda \le 0{\cdot}600$ Å^{-1}, 3315 with $I \ge 2{\cdot}5\sigma(I)$. One standard reflection ($\bar{1}\bar{1}3$) checked every 50 reflections: no significant deviation. Structure solved by direct methods using *SHELXS*86 (Sheldrick, 1985). H atoms from difference Fourier synthesis. Anisotropic least-squares refinement (*SHELX*76; Sheldrick, 1976) using F; H atoms isotropic with common refined temperature factor (B = 5·7 Å^2). $w = 1/(\sigma^2 + 0{\cdot}005F^2)$, $R = 0{\cdot}040$, $wR = 0{\cdot}046$, $S = 0{\cdot}72$ for 3315 observed reflections. Final maximum shift to e.s.d. = 0·13. Maximum and minimum heights in final difference Fourier synthesis = 0·25 and −0·26 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV).

The atomic parameters are given in Table 1.* There are two independent molecules in the asymmetric unit. Fig. 1 is a stereoscopic view of molecule *A*, showing the numbering of the atoms (*PLUTO;* Motherwell & Clegg, 1978). Bond distances and angles are given in Table 2. The two molecules have identical configurations and very similar conformations. Attempts were made to determine the absolute configuration. Unfortunately, the space group $P1$ does not allow the measurement of symmetry-related reflections, and the shape and quality of the available crystals were poor. At the end of the refinement, reflections were sorted according to $w^{1/2}||F_c(hkl)| - |F_c(\bar{h}\bar{k}\bar{l})||$ and among the seven first pairs of reflections, the signs of six observed and calculated differences were consistent with the absolute configuration given here. On this basis, it is probably the correct one. Each molecule creates intramolecular hydrogen bonds O(19)···H—O(18) (2·56, 2·57 Å) and O(17)···H—O(15) (2·64, 2·65 Å).

* 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 53484 (23 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic temperature factors* (Å^2)

$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}
Molecule *A*				
C1	7413 (5)	−22 (4)	2766 (2)	3·13 (4)
C2	6253 (5)	−908 (4)	3321 (2)	3·53 (5)
C3	7049 (6)	−2821 (5)	3607 (2)	4·25 (5)
C4	9068 (6)	−3836 (5)	3372 (2)	4·35 (5)
C5	10311 (6)	−2947 (5)	2876 (2)	4·18 (5)
C6	9511 (5)	−1059 (4)	2553 (2)	3·30 (5)
C7	10976 (6)	−202 (6)	2014 (2)	4·63 (6)
C8	6277 (5)	1901 (4)	2415 (2)	3·39 (5)
O9	7071 (4)	2337 (3)	1731 (1)	4·30 (4)
C10	5941 (6)	4111 (4)	1311 (2)	4·10 (5)
C11	5621 (6)	3394 (4)	482 (2)	3·59 (5)
C12	4355 (5)	5204 (4)	36 (2)	3·77 (5)
C13	7201 (11)	5687 (7)	1324 (4)	7·35 (11)
C14	4379 (10)	1698 (7)	496 (3)	6·29 (9)
O15	4268 (4)	34 (4)	3601 (1)	4·66 (4)
O16	9877 (6)	−5730 (4)	3637 (2)	6·98 (6)
O17	4686 (4)	2982 (4)	2723 (1)	5·55 (4)
O18	7604 (4)	2652 (4)	90 (1)	5·29 (4)
O19	5072 (4)	5786 (4)	−564 (2)	5·64 (5)
O20	2515 (4)	6018 (4)	350 (2)	5·60 (5)
Molecule *B*				
C1	6589 (5)	96 (4)	−4454 (2)	3·06 (4)
C2	7701 (5)	1121 (4)	−4985 (2)	3·55 (5)
C3	9740 (6)	260 (5)	−5258 (2)	4·13 (5)
C4	10702 (5)	−1665 (5)	−5039 (2)	3·96 (5)
C5	9612 (5)	−2771 (5)	−4562 (2)	3·83 (5)
C6	7605 (4)	−1932 (4)	−4256 (2)	2·91 (4)
C7	6514 (6)	−3236 (4)	−3755 (2)	3·86 (5)
C8	4585 (5)	1244 (4)	−4116 (2)	3·44 (5)
O9	3965 (3)	523 (3)	−3431 (1)	3·91 (3)
C10	2086 (5)	1660 (5)	−3006 (2)	3·75 (5)
C11	2816 (5)	2451 (4)	−2238 (2)	3·42 (5)
C12	893 (5)	3902 (4)	−1837 (2)	3·73 (5)
C13	615 (7)	300 (8)	−2861 (3)	6·19 (9)
C14	4588 (7)	3534 (7)	−2385 (3)	5·57 (8)
O15	6842 (4)	3023 (3)	−5252 (1)	4·78 (4)
O16	12712 (4)	−2533 (4)	−5287 (2)	5·80 (5)
O17	3547 (4)	2799 (3)	−4399 (1)	5·10 (4)
O18	3544 (4)	834 (4)	−1717 (1)	4·89 (4)
O19	204 (4)	3632 (4)	−1197 (1)	5·52 (4)
O20	148 (5)	5473 (4)	−2275 (2)	5·67 (5)
Water				
O1*W*	402 (4)	9100 (4)	−473 (2)	6·18 (5)
O2*W*	−2750 (4)	8064 (3)	−1445 (1)	4·52 (4)

Table 2. *Bond distances* (Å) *and angles* (°)

	A	*B*		*A*	*B*
C2—C1	1·404 (4)	1·415 (4)	C6—C1	1·410 (4)	1·428 (3)
C8—C1	1·478 (4)	1·447 (4)	C3—C2	1·378 (4)	1·376 (4)
O15—C2	1·363 (4)	1·367 (3)	C4—C3	1·373 (5)	1·369 (4)
C5—C4	1·377 (5)	1·387 (5)	O16—C4	1·356 (4)	1·349 (4)
C6—C5	1·388 (4)	1·374 (4)	C7—C6	1·505 (5)	1·501 (4)
O9—C8	1·313 (3)	1·344 (3)	O17—C8	1·217 (3)	1·221 (3)
C10—O9	1·456 (3)	1·447 (3)	C11—C10	1·525 (4)	1·540 (4)
C13—C10	1·489 (7)	1·488 (7)	C12—C11	1·526 (4)	1·533 (4)
C14—C11	1·550 (7)	1·514 (6)	O18—C11	1·407 (4)	1·413 (3)
O19—C12	1·207 (4)	1·189 (4)	O20—C12	1·286 (4)	1·307 (4)
C6—C1—C2	118·5 (2)	117·3 (2)	C8—C1—C2	116·6 (2)	117·0 (2)
C8—C1—C6	124·8 (3)	125·6 (3)	C3—C2—C1	121·6 (3)	121·5 (3)
O15—C2—C1	122·8 (2)	122·6 (3)	O15—C2—C3	115·6 (3)	115·9 (3)
C4—C3—C2	118·9 (3)	119·8 (3)	C5—C4—C3	120·9 (3)	120·4 (3)
O16—C4—C3	120·0 (3)	121·0 (3)	O16—C4—C5	119·1 (3)	118·6 (3)
C6—C5—C4	121·1 (3)	121·3 (3)	C5—C6—C1	118·6 (3)	119·5 (3)
C7—C6—C1	123·7 (2)	122·3 (2)	C7—C6—C5	117·7 (3)	118·1 (2)
O9—C8—C1	115·1 (2)	114·8 (2)	O17—C8—C1	122·9 (3)	124·0 (3)
O17—C8—O9	122·0 (2)	121·1 (3)	C10—O9—C8	118·7 (2)	119·3 (2)
C11—C10—O9	106·4 (2)	107·5 (2)	C13—C10—O9	109·1 (3)	108·2 (3)
C13—C10—C11	113·5 (3)	112·8 (3)	C12—C11—C10	107·8 (2)	108·2 (2)
C14—C11—C10	111·9 (3)	112·3 (3)	C14—C11—C12	109·0 (3)	109·6 (3)
O18—C11—C10	110·0 (3)	110·3 (2)	O18—C11—C12	109·6 (2)	108·1 (2)
O18—C11—C14	108·5 (3)	108·3 (3)	O19—C12—C11	121·0 (3)	123·4 (3)
O20—C12—C11	114·8 (3)	112·0 (3)	O20—C12—O19	124·2 (3)	124·6 (3)

Related literature. This phytotoxin is an ester of orsellinic acid and the dihydroxy acid dimethylglyceric acid. Orsellinic acid is one of the β-resorcylic acids which are known as constituents of depsides produced by lichens (Asahina & Shibata, 1954; Stoessl, 1981). In addition, orsellinic acid is metabolized by fungi (Bentley, Ghaphery & Keil, 1965) and biosynthesized from tetraketo acid (Mosbach, 1960). Dimethylglyceric acid has been found in wine (Carles, Layole & Lattes, 1966), in cider (Whiting, 1958) and as esters in alkaloids of *Veratum viride* (Myers, Morozovitch, Glen, Barber, Papineau-Couture & Grant, 1955) as powerful antihypertensive agents.

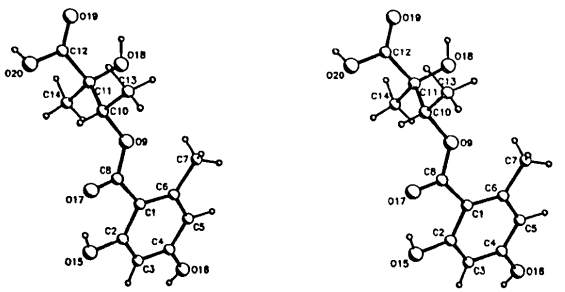


Fig. 1. Stereoview of molecule *A* and atom numbering.

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