

Fig. 2. Stereoview showing the packing of the peptides around the water molecules.

One water molecule is bonded to the peptide carboxyl groups related by the twofold axis and acts as proton donor for both hydrogen bonds.

In the crystallographic 'a' direction, these helices are related by a twofold axis and hydrogen bonded at the level of the COO⁻ and NH₃⁺ polar groups. In the 'c' direction, the stacking of the helices is only stabilized by

weak van der Waals interactions between atoms of the hydrophobic phenyl groups.

References

- BENEDETTI, E. (1977). *Peptides* (Proc. Fifth Am. Pept. Symp.), edited by GOODMAN & MEINHOFER, pp. 257–273. New York: John Wiley.
- BENEDETTI, E., MORELLI, G., NEMETHY, G. & SCHERAGA, H. A. (1983). *Int. J. Pept. Protein Res.* **22**, 1–15.
- COTRAIT, M. & BIDEAU, J. P. (1974). *Acta Cryst.* **B30**, 1024–1028. *International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
- IUPAC–IUB COMMISSION ON BIOCHEMICAL NOMENCLATURE (1970). *Biochemistry*, **9**, 3471–3478.
- MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- MERRIFIELD, R. B. (1963). *J. Am. Chem. Soc.* **82**, 2149–2154.
- PRANGE, T. & PASCARD, C. (1979). *Acta Cryst.* **B35**, 1812–1819.
- SHEENAN, J. C. & NESS, G. P. (1955). *J. Am. Chem. Soc.* **77**, 1067–1068.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

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rel-(1*aR*,2*R*,4*aR*,7*S*,7*aR*,7*bR*)-Decahydro-7-hydroxy-1,1,7-trimethyl-4-methylene-1*H*-cycloprop[*e*]azulen-2-yl Benzoate

BY PREM C. JAIN* AND WILLIAM H. WATSON†

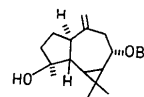
FASTBIOS Laboratory, Department of Chemistry, Texas Christian University, Fort Worth, Texas 76129, USA

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Abstract. C₂₂H₂₈O₃, *M_r* = 340.47, monoclinic, *P*2₁, *a* = 10.957 (4), *b* = 9.170 (2), *c* = 9.804 (3) Å, β = 93.23 (3)°, *V* = 983.5 (5) Å³, *Z* = 2, *D_x* = 1.150, *D_m*(floatation) = 1.148 g cm⁻³, λ(Cu *Kα*) = 1.54178 Å, μ = 6.21 cm⁻¹, *F*(000) = 368, *T* = 293 K, *R* = 0.035 for 1295 unique observed reflections. The compound is an aromadendrane-type of sesquiterpene isolated from *Ferulago antiochia* (Apiaceae). It contains a seven-membered ring in a chair conformation fused to a three-membered ring and to a five-membered ring in a half-chair conformation. The benzoate ester exhibits an extended conformation with the benzene ring planar to within 0.020 (11) Å.

Introduction. As part of a phytochemical screening of the genus *Ferula* or *Ferulago* (family Apiaceae) (Miski, Ulubelen & Mabry, 1983; Miski, Ulubelen, Mabry,

Vickovic, Watson & Holub, 1984), a newly described species *Ferulago antiochia* Saya & Miski (Saya & Miski, 1985) from Hatay province in the Southern region of Turkey has been investigated. In addition to several germacrane-type sesquiterpene esters, the petroleum ether extract of the roots of *F. antiochia* afforded a new aromadendrene ester (1). Spectral data indicated (1) to be 8β-benzoyloxyspathulenol; this structure has been confirmed by X-ray crystallography.



(1)

Experimental. A large colorless crystal was cleaved and air-ground to dimensions 0.48 × 0.49 × 0.36 mm, mounted along the *c* axis on a Syntex *P*2₁ diffractometer and data collected using a θ:2θ scan, 2θ_{max}

* On leave from Kurukshetra University, Kurukshetra, India.

† To whom correspondence should be addressed.

= 114.7°, graphite-monochromated Cu *K* α radiation; lattice parameters from a least-squares refinement of 15 reflections ($9.03 \leq 2\theta \leq 39.3^\circ$), angles measured by a centering routine associated with the diffractometer, systematic absences ($0k0$ $k = 2n + 1$) consistent with space group $P2_1$, Laue symmetry 2, a monitored reflection (011) showed no change greater than 2%; 1436 independent reflections measured ($0 \leq h \leq 11$, $0 \leq k \leq 10$, $-10 \leq l \leq 10$) with 1295 intensities greater than $3\sigma(I)$, accurate measurement of 56 equivalent reflections ($R_{\text{int}} = 0.017$), Lorentz-polarization corrections applied, consideration of crystal shape, transmission factors, and R_{int} resulted in neglect of absorption; direct methods (*MULTAN78*: Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) revealed the positions of ten atoms. Least-squares refinement and a weighted Fourier synthesis yielded the additional non-H atoms; least-squares refinement followed by a difference Fourier synthesis revealed the positions of all H atoms, least-squares refinement with all atoms (H atoms isotropic) reduced R to 0.039, further refinement with H atoms fixed led to a final $R = 0.035$, $wR = 0.044$, $S = 1.98$, $(\Delta/\sigma)_{\text{av}} = 0.017$, $(\Delta/\sigma)_{\text{max}} = 0.11$ and a largest peak in final difference Fourier map of $|0.14| e \text{ \AA}^{-3}$; $\sum w(|F_o| - |F_c|)^2$ minimized, $w = 1/\sigma^2(F_o)$ from counting statistics, 226 parameters refined using 1295 reflections; all calculations performed with *XRAY76* (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976); atomic scattering factors for C and O those of Cromer & Mann (1968), those for H from

Stewart, Davidson & Simpson (1965). Table 1 lists atomic positional parameters and U_{eq} values while Table 2 gives interatomic distances, valence angles and selected torsion angles.*

Discussion. Fig. 1 is an *ORTEP* (Johnson, 1971) drawing showing the relative absolute configuration of the title compound. The torsion angles (Table 2) show the seven-membered ring to be in a chair conformation (Toromonoff, 1980) which is stabilized by the *cis*-fused cyclopropane ring and a *trans*-fused five-membered

* Lists of H-atom coordinates, anisotropic thermal parameters and structure factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42502 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional atomic coordinates ($\times 10^4$) for non-H atoms and their equivalent isotropic thermal parameters U_{eq} ($\text{\AA}^2 \times 10^3$)

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}^*
C(1)	7446 (2)	3026 (4)	5327 (3)	52 (2)
C(1a)	8269 (2)	4207 (4)	4814 (3)	50 (2)
C(2)	8170 (2)	5776 (4)	5210 (3)	55 (2)
O(2)	9155 (2)	5975 (3)	6274 (2)	59 (1)
C(3)	8375 (3)	6847 (4)	4045 (3)	66 (2)
C(4)	7259 (3)	7014 (4)	3050 (3)	59 (2)
C(4a)	6818 (3)	5659 (4)	2317 (3)	56 (2)
C(5)	5814 (3)	5805 (5)	1172 (3)	75 (2)
C(6)	5352 (3)	4265 (5)	1015 (3)	75 (2)
C(7)	5213 (3)	3756 (4)	2473 (3)	59 (2)
O(7)	4120 (2)	4403 (4)	2980 (3)	84 (2)
C(7a)	6291 (2)	4522	3294 (3)	48 (2)
C(7b)	7299 (2)	3540 (4)	3859 (3)	51 (2)
C(8)	8023 (3)	1547 (5)	5553 (3)	65 (2)
C(9)	6492 (3)	3388 (5)	6323 (3)	67 (2)
C(10)	6729 (3)	8276 (4)	2898 (4)	70 (2)
C(11)	5149 (3)	2126 (4)	2648 (4)	72 (2)
C(12)	9005 (3)	6892 (4)	7287 (4)	65 (2)
O(12)	8135 (2)	7676 (4)	7360 (3)	108 (2)
C(13)	10033 (3)	6824 (4)	8338 (3)	62 (2)
C(14)	10913 (3)	5765 (4)	8328 (3)	55 (2)
C(15)	11846 (3)	5718 (5)	9336 (3)	63 (2)
C(16)	11899 (3)	6722 (6)	10358 (4)	87 (3)
C(17)	11066 (4)	7796 (8)	10340 (5)	138 (4)
C(18)	10127 (4)	7857 (6)	9341 (5)	116 (4)

* $U_{\text{eq}} = \frac{1}{3}[U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}aba^*b^*\cos\gamma + 2U_{13}aca^*c^*\cos\alpha + 2U_{23}bcb^*c^*\cos\beta]$.

Table 2. Interatomic distances (\AA), valence angles ($^\circ$) and selected torsion angles ($^\circ$)

C(1)–C(1a)	1.514 (5)	C(6)–C(7)	1.519 (5)
C(1)–C(7b)	1.514 (4)	C(7)–C(7a)	1.559 (4)
C(1)–C(8)	1.508 (5)	C(7)–O(7)	1.450 (4)
C(1)–C(9)	1.506 (4)	C(7)–C(11)	1.506 (5)
C(1a)–C(2)	1.496 (6)	C(7a)–C(7b)	1.507 (4)
C(1a)–C(7b)	1.506 (4)	C(12)–O(2)	1.318 (4)
C(2)–O(2)	1.470 (3)	C(12)–O(12)	1.199 (5)
C(2)–C(3)	1.533 (5)	C(12)–C(13)	1.484 (4)
C(3)–C(4)	1.528 (4)	C(13)–C(14)	1.369 (5)
C(4)–C(4a)	1.502 (5)	C(13)–C(18)	1.365 (6)
C(4)–C(10)	1.299 (5)	C(14)–C(15)	1.382 (4)
C(4a)–C(5)	1.532 (4)	C(15)–C(16)	1.360 (6)
C(4a)–C(7a)	1.549 (4)	C(16)–C(17)	1.342 (8)
C(5)–C(6)	1.505 (6)	C(17)–C(18)	1.382 (7)
C(1a)–C(1)–C(9)	120.4 (3)	C(6)–C(7)–O(7)	109.0 (3)
C(1a)–C(1)–C(7b)	59.6 (2)	C(6)–C(7)–C(11)	114.8 (3)
C(7b)–C(1)–C(8)	116.1 (3)	O(7)–C(7)–C(7a)	104.8 (2)
C(8)–C(1)–C(9)	113.8 (3)	C(7a)–C(7)–C(11)	115.3 (2)
C(1)–C(1a)–C(7b)	60.2 (2)	O(7)–C(7)–C(11)	108.8 (3)
C(2)–C(1a)–C(7b)	119.6 (3)	C(4a)–C(7a)–C(7b)	109.8 (2)
C(1)–C(1a)–C(2)	123.2 (2)	C(4a)–C(7a)–C(7)	106.4 (2)
C(1a)–C(2)–C(3)	114.0 (3)	C(7)–C(7a)–C(7b)	116.0 (2)
C(1a)–C(2)–O(2)	104.1 (2)	C(1a)–C(7b)–C(7a)	117.5 (3)
O(2)–C(2)–C(3)	108.5 (3)	C(1)–C(7b)–C(1a)	60.2 (2)
C(2)–O(2)–C(12)	119.7 (2)	C(1)–C(7b)–C(7a)	124.9 (2)
C(2)–C(3)–C(4)	113.2 (3)	O(2)–C(12)–O(12)	124.1 (3)
C(3)–C(4)–C(4a)	116.5 (3)	O(2)–C(12)–C(13)	111.9 (3)
C(3)–C(4)–C(10)	119.9 (3)	C(13)–C(12)–O(12)	124.0 (3)
C(10)–C(4)–C(4a)	123.5 (3)	C(12)–C(13)–C(14)	122.2 (3)
C(4)–C(4a)–C(7a)	112.5 (2)	C(12)–C(13)–C(18)	119.6 (3)
C(4)–C(4a)–C(5)	118.5 (3)	C(14)–C(13)–C(18)	118.2 (3)
C(5)–C(4a)–C(7a)	103.6 (2)	C(13)–C(14)–C(15)	120.6 (3)
C(4a)–C(5)–C(6)	102.5 (3)	C(14)–C(15)–C(16)	120.4 (3)
C(5)–C(6)–C(7)	104.1 (3)	C(15)–C(16)–C(17)	119.1 (3)
C(6)–C(7)–C(7a)	103.5 (2)	C(16)–C(17)–C(18)	121.2 (5)
		C(17)–C(18)–C(13)	120.4 (5)
C(1a)C(2)C(3)C(4)	78.8 (3)	C(7a)C(4a)C(5)C(6)	–37.4 (3)
C(2)C(3)C(4)C(4a)	–60.4 (4)	C(13)C(14)C(15)C(16)	0.3 (6)
C(3)C(4)C(4a)C(7a)	67.2 (3)	C(14)C(15)C(16)C(17)	–3.0 (6)
C(4)C(4a)C(7a)C(7b)	–88.7 (3)	C(15)C(16)C(17)C(18)	3.1 (8)
C(4a)C(7a)C(7b)C(1a)	67.4 (3)	C(16)C(17)C(18)C(13)	–0.5 (8)
C(7a)C(7b)C(1a)C(2)	2.7 (4)	C(17)C(18)C(13)C(14)	–2.1 (7)
C(7b)C(1a)C(2)C(3)	–69.7 (3)	C(18)C(13)C(14)C(15)	2.2 (5)
C(4a)C(5)C(6)C(7)	45.6 (3)	C(2)O(2)C(12)C(13)	175.6 (6)
C(5)C(6)C(7)C(7a)	–34.9 (3)	C(2)O(2)C(12)O(12)	–5.6 (6)
C(6)C(7)C(7a)C(4a)	11.2 (3)	O(2)C(12)C(13)C(14)	–9.5 (6)
C(7)C(7a)C(4a)C(5)	16.0 (3)		

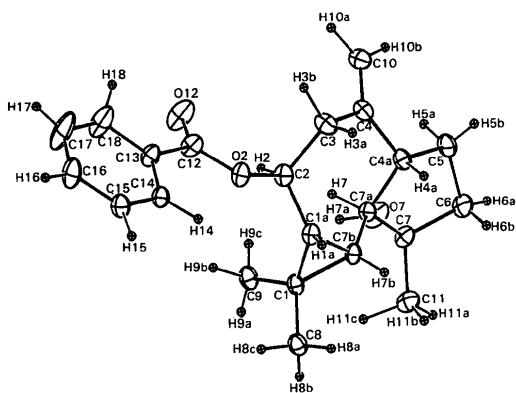


Fig. 1. ORTEP drawing of the title compound (relative configuration). Thermal ellipsoids are drawn at the 35% probability level. H atoms represented by spheres of arbitrary size.

ring. The five-membered ring is in a half-chair conformation. The benzoate ester exhibits an extended conformation, and the molecules lie in rows parallel to the body diagonal of the unit cell. The thermal motion associated with the benzoate group is much larger than that of the rigid aromadendrane framework. This is consistent with the observed molecular packing and the weak intermolecular interactions. The C—C distances in the benzene ring range from 1.342 (8) to 1.382 (4) Å which reflects the relatively large anisotropic thermal motion. The valence angles all lie within 3σ of the ideal value of 120.0° . A least-squares plane fitted to the six atoms of the benzene ring shows a maximum deviation of 0.020 (11) Å from the plane while C(12) and O(12) lie out of the plane by 0.045 (8) and 0.245 (8) Å respectively. The C(4a)—C(7a) bond joining the five- and seven-membered rings is 1.549 (4) Å while the

C(1a)—C(7b) bond joining the three- and seven-membered rings is 1.506 (4) Å. The remaining distances and angles around the five- and seven-membered rings can be rationalized in terms of conformational symmetry and substitution pattern.

There are two H...H intermolecular contacts of less than 2.50 Å, one O...H of less than 2.80 Å and one O...O of less than 3.00 Å; H(8b)...H(7)(1-x, y-1/2, 1-z) = 2.30 (5), H(16)...H(6b)(2-x, 1/2+y, 1-z) = 2.40 (4), O(7)...H(8b)(1-x, 1/2+y, 1-z) = 2.62 (3) and O(12)...O(7)(1-x, 1/2+y, 1-z) = 2.938 (4) Å.

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References

- CROMER, D. T. & MANN, J. P. (1968). *Acta Cryst.* **A24**, 321–324.
 JOHNSON, C. K. (1971). *ORTEPII*. Report ORNL-3794, revised. Oak Ridge National Laboratory, Tennessee.
 MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCO, J.-P. & WOOLFSON, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
 MISKI, M., ULUBELEN, A. & MABRY, T. J. (1983). *Phytochemistry*, **22**, 2231–2233.
 MISKI, M., ULUBELEN, A., MABRY, T. J., VICKOVIC, I., WATSON, W. H. & HOLUB, M. (1984). *Tetrahedron*, **40**, 5197–5201.
 SAYA, O. & MISKI, M. (1985). *Plant Syst. Evol.* Submitted.
 STEWART, J. M., MACHIN, P. A., DICKINSON, C. W., AMMON, H. L., HECK, H. & FLACK, H. (1976). The *XRAY76* System. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland.
 STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
 TOROMONOFF, E. (1980). *Tetrahedron*, **36**, 2809–2931.

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Die Strukturen von *N*-Mesylhydroxylamin, $\text{CH}_3\text{SO}_2\text{N}(\text{H})\text{OH}$, und *N*-Mesyl-*O*-methylhydroxylamin, $\text{CH}_3\text{SO}_2\text{N}(\text{H})\text{OCH}_3$

VON KLAUS BRINK UND RAINER MATTES

Anorganisch-Chemisches Institut der Universität Münster, Corrensstrasse 36, 4400 Münster, Bundesrepublik Deutschland

(Eingegangen am 8. Februar 1985; angenommen am 20. September 1985)

Abstract. $\text{CH}_5\text{NO}_3\text{S}$ (I): $M_r = 111.12$, triclinic, $P\bar{1}$, $a = 5.621$ (3), $b = 5.791$ (3), $c = 6.922$ (4) Å, $\alpha = 97.31$ (7), $\beta = 100.33$ (8), $\gamma = 104.29$ (5)°, $V = 211.3$ Å³, $Z = 2$, $D_m = 1.70$, $D_x = 1.75$ Mg m⁻³,

Mo $K\alpha$, $\lambda = 0.71069$ Å, $\mu = 0.31$ mm⁻¹, $F(000) = 116$, $T = 160$ K, $R = 0.058$ for 886 independent reflections. $\text{C}_2\text{H}_7\text{NO}_3\text{S}$ (II): $M_r = 125.15$, monoclinic, $P2_1/n$, $a = 8.018$ (3), $b = 4.902$ (2), $c = 13.629$ (6) Å, $\beta =$

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