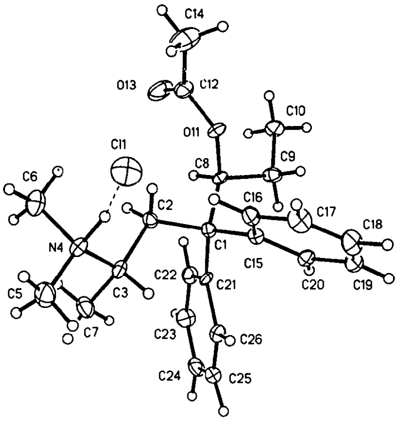

Fig. 1. A thermal ellipsoid plot of one of the two independent (−)-α-acetylmethadol molecules with ellipsoids drawn at the 20% probability level. The N—H···Cl hydrogen bond is also shown.

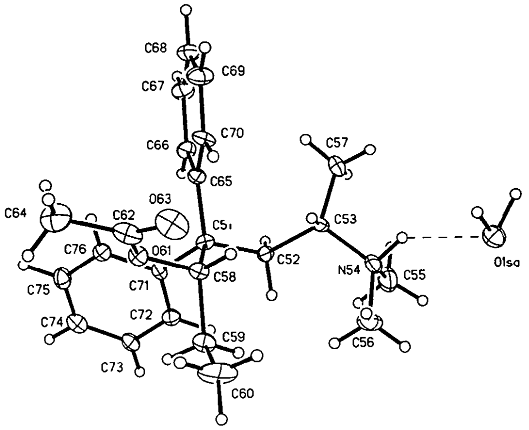

Fig. 2. A thermal ellipsoid plot of the other independent molecule of (−)-α-acetylmethadol hydrochloride with ellipsoids drawn at the 20% probability level. The N—H···O hydrogen bond is also shown.

fixed $U_{\text{iso}}(\text{H})$]. H atoms on the water molecules were not found. $(\Delta/\sigma)_{\max} = 0.35$, ratio of observations to parameters = 4.7:1, $R = 0.069$ ($R = 0.082$ for all data), $wR = 0.064$, $S = 1.49$. Final difference map excursions were 0.32 and −0.29 e Å^{-3}. Atomic scattering factors were obtained from *International Tables for X-ray Crystallography* (1974, Vol. IV). Atom numbering for Tables 1, atom coordinates, and 2, bond distances and angles, follows that shown in Figs. 1 and 2.*

Related literature. The structures of methadone and several of its derivatives have been reported (Hanson & Ahmed, 1958; Bürgi, Dunitz & Shefter, 1973; Bye, 1974; Shefter, 1974; Singh & Moreland, 1989). The structure of a racemic mixture of the title compound has also been reported (Shefter, 1974).

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* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55113 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH0604]

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Structure of 3,6-Diacetyl-8-benzoylantakyatriol

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Abstract. 3,6-Diacetyl-8-benzoylantakyatriol, (2) [4,10-dimethyl-7-(1-methylethyl)cyclodeca-4,10-diene-3,6,8-triol 3,8-diacetate 8-benzoate], was isolated during chemotaxonomical investigation of *Ferula* and closely related taxa. $C_{26}H_{34}O_6$, $M_r = 442.56$, orthorhombic, $P2_12_12_1$, $a = 7.766$ (6), $b = 11.809$ (4),

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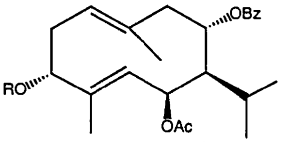
$c = 27.219\ (11)$ Å, $V = 2496.5\ (12)$ Å^3, $Z = 4$, $D_x = 1.177$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.772$ cm^{-1}, $F(000) = 952$, $T = 223$ K, $R = 0.051$, $wR = 0.068$ for 2149 observations [$I \geq 3\sigma(I)$]. Although the structure and possible conformation of this cycloundecane derivative were proposed based on the spectral data of the monoacetate, 6-acetyl-8-benzoylantakyatriol (1) and its derivatives, owing to the conformationally highly flexible nature of (1), some observed nuclear Overhauser enhancements in the NOE difference spectroscopy experiments were ambiguous. In order to determine the total stereochemistry and conformation of (1), a single crystal of the acetate derivative (2) was analyzed by X-ray diffraction methods. The ten-membered ring adopts a boat–chair conformation nearly identical in detail to that observed for similar sesquiterpenoids previously characterized. The ring methyls are *syn* and project to the α face. The *trans* double bonds are both strained, as reflected in the C2—C1—C10—C9 and C3—C4—C5—C6 torsion angles of −162.4 (4) and −159.6 (3)°.

Table 1. *Positional parameters and equivalent isotropic thermal parameters* (Å^2) *for* (2)

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

	x	y	z	B_{eq}
O3	0.3406 (4)	0.4948 (2)	0.8302 (1)	3.93 (6)
O6	0.0546 (3)	0.1193 (2)	0.94988 (8)	3.03 (5)
O8	0.0914 (3)	−0.0881 (2)	0.87180 (9)	3.11 (5)
O16	0.6244 (5)	0.4988 (3)	0.8435 (2)	6.97 (9)
O18	−0.0313 (5)	0.2608 (3)	0.9990 (1)	5.59 (7)
O20	0.3433 (4)	−0.0867 (3)	0.9117 (1)	6.20 (8)
C1	0.3258 (6)	0.1909 (3)	0.7919 (1)	3.75 (8)
C2	0.3594 (6)	0.3168 (3)	0.7860 (1)	3.75 (8)
C3	0.3384 (5)	0.3725 (3)	0.8366 (1)	3.25 (7)
C4	0.1695 (5)	0.3388 (3)	0.8598 (1)	2.92 (7)
C5	0.1722 (5)	0.2544 (3)	0.8928 (1)	2.76 (6)
C6	0.0165 (5)	0.1869 (3)	0.9067 (1)	2.61 (6)
C7	−0.0358 (5)	0.1020 (3)	0.8664 (1)	2.66 (6)
C8	0.1201 (5)	0.0254 (3)	0.8524 (1)	2.83 (7)
C9	0.1590 (6)	0.0147 (3)	0.7969 (1)	3.79 (8)
C10	0.1947 (6)	0.1305 (3)	0.7748 (1)	3.66 (8)
C11	−0.2029 (5)	0.0386 (3)	0.8788 (1)	3.18 (7)
C12	−0.2698 (6)	−0.0271 (3)	0.8342 (2)	4.40 (9)
C13	−0.3421 (5)	0.1183 (3)	0.8977 (2)	4.31 (9)
C14	0.0659 (7)	0.1697 (4)	0.7375 (2)	5.2 (1)
C15	0.0091 (6)	0.3882 (3)	0.8382 (1)	3.99 (8)
C16	0.4914 (6)	0.5475 (3)	0.8360 (2)	4.13 (8)
C17	0.4715 (7)	0.6733 (4)	0.8300 (2)	5.9 (1)
C18	0.0279 (5)	0.1674 (3)	0.9939 (1)	3.39 (7)
C19	0.0817 (7)	0.0911 (4)	1.0346 (2)	5.0 (1)
C20	0.2061 (5)	−0.1311 (3)	0.9033 (1)	3.24 (7)
C21	0.1483 (5)	−0.2392 (3)	0.9255 (1)	3.39 (7)
C22	−0.0017 (6)	−0.2926 (3)	0.9103 (1)	4.05 (9)
C23	−0.0494 (7)	−0.3942 (4)	0.9315 (2)	5.3 (1)
C24	0.0471 (8)	−0.4408 (3)	0.9683 (2)	5.7 (1)
C25	0.1985 (8)	−0.3872 (4)	0.9838 (2)	6.0 (1)
C26	0.2492 (7)	−0.2862 (4)	0.9622 (2)	5.0 (1)

Experimental. This highly oxygenated germacrane ester was isolated from *Ferulago antiochia* (Miski, Moubasher & Mabry, 1990). Colorless crystals of (2) were grown by slow evaporation from hexane/ethyl acetate.



(1) R = H
(2) R = Ac

The data crystal had approximate dimensions 0.90 × 0.75 × 0.25 mm and was mounted with epoxy on a glass fiber. Cell constants were derived from least-squares refinement of the setting angles for 25 reflections ($30 \leq 2\theta \leq 35°$) located using the *SEARCH* routine on an Enraf–Nonius CAD-4 diffractometer equipped with Mo source and graphite monochromator. The systematic absences of $h00$, $0k0$ and $00l$ for h, k and l odd uniquely determined the space group. Intensity data were collected in an ω–2θ-scan mode using variable speeds (2.5–12.4° min^{-1}). A total of 3423 intensities were scanned ($0 \leq h \leq 10$, $0 \leq k \leq 15$, $0 \leq l \leq 36$, $2\theta_{max} = 56°$). Data were corrected for Lorentz and polarization effects and for absorption. Intensities of three standard reflections (1,7,15, 3,2,18, 464) measured every 3 h of exposure (16 times) showed a non-systematic overall increase of 6.3%. A correction was made for these changes; correction factors 0.968 minimum, 1.000 maximum. The absorption correction was made using the *DIFABS* algorithm (Walker & Stuart, 1983); correction factors 0.636 minimum, 1.216 maximum and 0.943 average.

The structure was solved using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Refinement by full-matrix least squares minimized the function $\Sigma w(|F_o| - |F_c|)^2$ where the weights, w, eventually were defined as $4F_o^2/\sigma^2(I)$ with $\sigma^2(I)$ defined as $[\sigma^2(I_c) + (0.06I)^2]$. Non-H atoms were refined with anisotropic displacement parameters. H-atom positions were located from difference Fourier maps but were subsequently assigned based on geometric considerations assuming a C—H bond distance of 1 Å, and were held fixed along with isotropic temperature factors assigned as 1.3 times the B_{eq} value of the attached atom. The refinement converged (maximum $\Delta/\sigma = 0.02$) to values of the standard crystallographic residuals $R = 0.051$, $wR = 0.068$, $S = 1.519$ for 2149 observations with $I > 3\sigma(I)$, 289 variables. A final difference Fourier map showed maximum excursions of ±0.315 e Å^{-3}. Neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV), for H from Stewart, Davidson & Simpson (1965), were used as incorporated in a locally modified version of the Enraf–Nonius *SDP-Plus* (Frenz, 1987) which was the source of all programs. A refinement using all 2981 observations not marked as weak in a prescan led to $R = 0.073$, $wR = 0.073$, $S = 1.370$. Atomic coordi-

Table 2. *Principal bond distances* (Å) *and angles* (°) *for* (2)

O3—C3	1.454 (3)	C7—C8	1.558 (4)
O3—C16	1.336 (4)	C7—C11	1.536 (4)
O6—C6	1.451 (3)	C8—C9	1.544 (4)
O6—C18	1.342 (3)	C9—C10	1.519 (4)
O8—C8	1.457 (3)	C10—C14	1.500 (5)
O8—C20	1.336 (4)	C11—C12	1.529 (4)
O16—C16	1.200 (5)	C11—C13	1.524 (5)
O18—C18	1.203 (4)	C16—C17	1.502 (5)
O20—C20	1.209 (4)	C18—C19	1.488 (5)
C1—C2	1.517 (4)	C20—C21	1.483 (4)
C1—C10	1.327 (5)	C21—C22	1.388 (5)
C2—C3	1.535 (5)	C21—C26	1.386 (5)
C3—C4	1.509 (5)	C22—C23	1.383 (5)
C4—C5	1.342 (4)	C23—C24	1.366 (6)
C4—C15	1.496 (5)	C24—C25	1.400 (7)
C5—C6	1.497 (4)	C25—C26	1.387 (5)
C6—C7	1.541 (4)		
C3—O3—C16	117.3 (3)	C1—C10—C14	125.6 (3)
C6—O6—C18	117.3 (2)	C9—C10—C14	115.1 (3)
C8—O8—C20	118.7 (2)	C7—C11—C12	111.2 (3)
C2—C1—C10	128.5 (4)	C7—C11—C13	111.9 (2)
C1—C2—C3	107.9 (2)	C12—C11—C13	109.9 (3)
O3—C3—C2	108.5 (3)	O3—C16—O16	123.5 (3)
O3—C3—C4	108.8 (3)	O3—C16—C17	110.9 (4)
C2—C3—C4	110.8 (3)	O16—C16—C17	125.5 (4)
C3—C4—C5	117.6 (3)	O6—C18—O18	123.4 (3)
C3—C4—C15	117.2 (2)	O6—C18—C19	111.4 (3)
C5—C4—C15	124.4 (3)	O18—C18—C19	125.2 (3)
C4—C5—C6	123.6 (3)	O8—C20—O20	123.0 (3)
O6—C6—C5	109.5 (2)	O8—C20—C21	112.8 (3)
O6—C6—C7	105.8 (2)	O20—C20—C21	124.2 (3)
C5—C6—C7	112.3 (2)	C20—C21—C22	121.6 (3)
C6—C7—C8	110.3 (3)	C20—C21—C26	117.9 (3)
C6—C7—C11	112.6 (2)	C22—C21—C26	120.5 (3)
C8—C7—C11	115.3 (2)	C21—C22—C23	119.6 (4)
O8—C8—C7	109.0 (2)	C22—C23—C24	120.6 (4)
O8—C8—C9	108.0 (2)	C23—C24—C25	119.9 (4)
C7—C8—C9	116.0 (3)	C24—C25—C26	120.0 (4)
C8—C9—C10	110.5 (2)	C21—C26—C25	119.3 (4)
C1—C10—C9	119.1 (3)		

nates are presented in Table 1;* bond distances and angles are listed in Table 2. A view of the molecule is provided in Fig. 1.

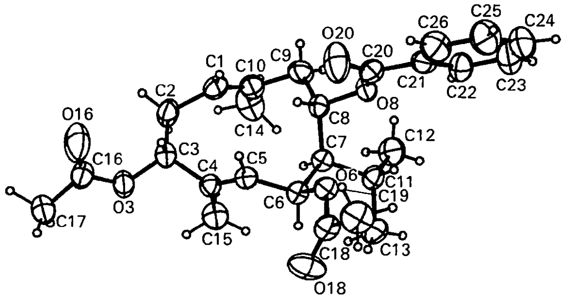


Fig. 1. *ORTEP*II (Johnson, 1976) view of (2) with non-H atoms as principal ellipses at the 50% probability level; H atoms as small spheres of arbitrary size.

Related literature. The absolute configuration of the related germacrane ester shiromodiol has been reported from its 6-*O*-acetate 8-*O*-*p*-bromobenzoate derivative (Sim, 1987). The structure of chimganidin has been determined by Makhmudov, Tashkhodzaev, Saidkhodzaev, Yagudaev & Malikov (1986).

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* Lists of structure factors, anisotropic displacement parameters, bond distances and angles involving H atoms, and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55128 (26 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST0563]

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Structure of 7-Diethylamino-4-trifluoromethylcoumarin

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Abstract. 7-Diethylamino-4-trifluoromethyl-2*H*-1-benzopyran-2-one, $C_{14}H_{14}F_3NO_2$, $M_r = 285.26$, triclinic, $P\bar{1}$, $a = 14.392$ (2), $b = 18.837$ (3), $c = 10.191$ (2) Å, $\alpha = 90.15$ (2), $\beta = 98.72$ (2), $\gamma = 85.49$ (2)°, $V = 2722.3$ (8) Å^3, $Z = 8$, $D_m = 1.405$, $D_x = 1.392$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, μ =

0108-2701/92/101859-04$06.00