making a dihedral angle of 7·9 (6)° with the acetyl moiety. The C(2)—C(3) bond of 1·583 (2) Å is significantly longer than the value of 1·48 Å expected for a C(sp^2)—C(sp^2) single bond. As suggested by Palenik, Koziol, Katritzky & Fan (1990), this lengthening often observed in *cis*-diketones can be ascribed to non-bonded lone pair–lone pair repulsions.

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Structure of a Glycyrrhetinic Acid Derivative

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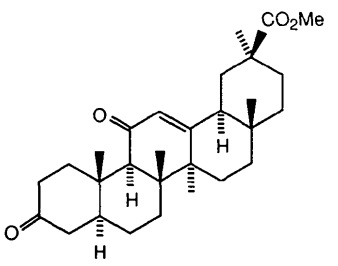
Abstract. Methyl 4,4-desmethyl-3,11-dioxo-18α-olean-12-en-30-oate, $C_{29}H_{42}O_4$, $M_r = 454.65$, orthorhombic, $P2_12_12_1$, $a = 12.521$ (1), $b = 29.271$ (3), $c = 6.779$ (1) Å, $V = 2484.3$ (3) Å^3, $Z = 4$, $D_x = 1.215$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.54178 Å, $\mu = 0.63$ mm^{-1}, $F(000) = 992$, $T = 295$ K, $R = 0.044$ for 2390 observed reflections. The ring junctions *A*/*B*, *B*/*C* and *D*/*E* are all *trans*. The conformations of rings *A*, *B*, *D* and *E* are all chair.

Experimental. Colorless plates (I) obtained from ethyl acetate. Crystal of dimensions 0.4 × 0.4 × 0.1 mm. Rigaku AFC-5R diffractometer, graphite-monochromatized Cu $K\alpha$ radiation. Cell dimensions determined from 2θ angles for 25 reflections in the range $30 < 2\theta < 45°$. Intensities measured up to $\theta = 70°$ in h 0/15, k 0/34 and l −8/0, ω–2θ scans, ω-scan width $(1 + 0.2\tan\theta)°$, three standard reflections monitored every 100 measurements showed no significant change. 2580 unique reflections measured, 2390 intensities observed [$F_o > 3\sigma(F_o)$] and two very strong reflections rejected], no absorption corrections.



(I)

Structure solved by *MULTAN*87 (Debaerdemaeker, Germain, Main, Tate & Woolfson, 1987). H atoms located on a difference density map. Positional and thermal parameters refined by block-diagonal least squares, isotropic for H and anisotropic for other atoms, 467 parameters. $\sum(w|\Delta F|^2)$ minimized, $w = 1/[\sigma^2(F_o) + 0.0022|F_o|^2]$, $w = 0$ for 59 reflections with $w^{1/2}|\Delta F| > 3$. The final $R = 0.044$, $wR = 0.057$, $S = 1.1171$. The maximum Δ/σ in the final cycle was 0.2. The highest and lowest peaks in the final difference density map were 0.6 and −0.3 e Å^{-3}. Atomic scattering factors calculated by $\sum[a_i\exp(-b_i\lambda^{-2}\sin^2\theta)] + c$ ($i = 1,...,4$) (*International Tables for X-ray Crystallography*, 1974, Vol. IV). Calculations performed on a VAX station 3100 computer. The final atomic coordinates and equivalent isotropic temperature factors are given in Table 1.* A perspective view of the molecule with the atom labeling is presented in Fig. 1.

Related literature. Structure–activity relationships of the title compound have been referred to by Terasawa, Okada, Hara & Itoh (1991).

The author thanks Drs Terasawa and Okada for supplying the crystals.

* Lists of H-atom coordinates, anisotropic temperature factors, bond lengths, bond angles and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54608 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS0539]

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

$B_{eq} = \frac{4}{3}\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
C1	0.6918 (2)	0.5946 (1)	1.0794 (3)	3.88 (5)
C2	0.7083 (2)	0.5436 (1)	1.1332 (4)	4.85 (6)
C3	0.6065 (2)	0.5167 (1)	1.1125 (6)	5.89 (8)
C4	0.5472 (2)	0.5235 (1)	0.9284 (6)	5.50 (8)
C5	0.5270 (2)	0.5750 (1)	0.8951 (4)	3.86 (5)
C6	0.4540 (2)	0.5842 (1)	0.7205 (5)	4.69 (6)
C7	0.4206 (2)	0.6345 (1)	0.7190 (4)	4.06 (5)
C8	0.5165 (1)	0.6679 (1)	0.7119 (3)	2.85 (4)
C9	0.6010 (1)	0.6537 (1)	0.8691 (3)	2.68 (4)
C10	0.6328 (1)	0.6020 (1)	0.8831 (3)	3.05 (4)
C11	0.6950 (1)	0.6867 (1)	0.8564 (3)	2.95 (4)
C12	0.6699 (1)	0.7347 (1)	0.8153 (3)	3.03 (4)
C13	0.5720 (1)	0.7505 (1)	0.7747 (3)	2.52 (3)
C14	0.4765 (1)	0.7182 (1)	0.7601 (3)	2.84 (4)
C15	0.3956 (2)	0.7341 (1)	0.6010 (4)	4.14 (5)
C16	0.3786 (2)	0.7858 (1)	0.5926 (4)	4.33 (6)
C17	0.4839 (2)	0.8124 (1)	0.5716 (4)	3.45 (5)
C18	0.5491 (1)	0.8016 (1)	0.7606 (3)	2.88 (4)
C19	0.6479 (1)	0.8319 (1)	0.7823 (3)	3.13 (4)
C20	0.6213 (2)	0.8837 (1)	0.7744 (3)	3.49 (4)
C21	0.5561 (2)	0.8943 (1)	0.5884 (4)	4.50 (6)
C22	0.4579 (2)	0.8639 (1)	0.5689 (4)	4.62 (6)
O23	0.5744 (2)	0.4926 (1)	1.2469 (5)	9.75 (10)
O24	0.7876 (1)	0.67525 (4)	0.8844 (3)	4.09 (4)
C25	0.7044 (2)	0.5855 (1)	0.7129 (4)	4.07 (5)
C26	0.5656 (2)	0.6658 (1)	0.5032 (3)	4.02 (5)
C27	0.4205 (2)	0.7212 (1)	0.9637 (4)	4.06 (5)
C28	0.5433 (2)	0.7992 (1)	0.3843 (4)	4.48 (6)
C29	0.5635 (2)	0.8991 (1)	0.9623 (5)	5.36 (7)
C30	0.7264 (2)	0.9099 (1)	0.7662 (4)	3.96 (5)
O31	0.8133 (1)	0.8938 (1)	0.7897 (5)	7.16 (7)
O32	0.7125 (1)	0.95352 (5)	0.7245 (3)	5.37 (5)
C33	0.8071 (2)	0.9817 (1)	0.7171 (5)	5.70 (7)

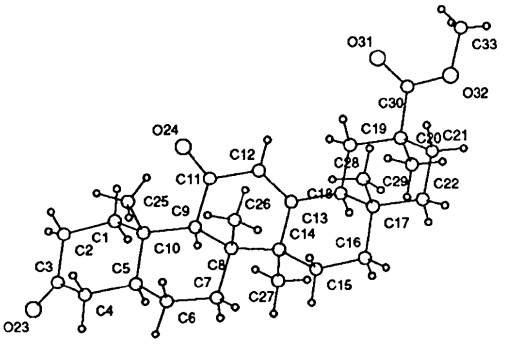

Fig. 1. Perspective view drawn by *PLUTO* (Motherwell & Clegg, 1978).

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2-[(2,6-Dihydroxyphenyl)ethynyl]benzoic Acid

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Abstract. $C_{15}H_{10}O_4$, $M_r = 254.2$, monoclinic, $P2_1/n$, $a = 12.8584$ (13), $b = 5.0051$ (2), $c = 19.381$ (2) Å, $\beta = 109.141$ (9)°, $V = 1178.3$ (2) Å^3, $Z = 4$, $D_x = 1.431$ g cm^{-3} at 295 K, λ(Cu $K\alpha$) = 1.54184 Å, $\mu = 8.28$ cm^{-1}, $F(000) = 528$, 2316 unique data measured, final $R = 0.037$ for 2065 reflections with $I > 3.0\sigma(I)$. Maximum deviations of the two aromatic rings are 0.0031 (15) Å for the ring containing the carboxy substitutent and 0.0063 (13) Å for the ring containing two hydroxy substituents. These two rings are essentially coplanar, exhibiting mean and maximum deviations of 0.007 and 0.016 (1) Å, respectively, from the 12-atom best plane. The ethynyl C atoms lie 0.014 (1) and 0.017 (1) Å in the same direction out of this plane. The triple-bond distance is 1.195 (2) Å, and the bond angles at the ethynyl C atoms are 172.3 (2) and 174.1 (2)°, which results in a *trans* kink in the three bonds that link the two aryls. One hydroxy substituent forms an intramolecular hydrogen bond of length 2.973 (2) Å with the carbonyl O atom of the carboxy group, with angle at H of 155 (2)°. The carboxyl group forms centrosymmetric hydrogen-bonded dimers, with O···O distance 2.684 (2) Å and a 170 (2)° angle at H. The other hydroxy group of the dihydroxyphenyl group forms chains of intermolecular hydrogen bonds propagated by the screw axis, having O···O distance 2.793 (2) Å and angle at H of 163 (2)°.

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