

Table 2. Bond lengths (Å) and angles (°) with e.s.d.'s in parentheses

The primed and unprimed atoms are related by a crystallographic diad axis.

C(1)—N(1)	1.48 (1)	C(1)—N(1)—C(3)	108.4 (7)
C(2)—N(2)	1.48 (1)	C(1)—N(1)—C(4)	108.8 (7)
C(3)—N(1)	1.47 (2)	C(3)—N(1)—C(4)	108.8 (11)
C(3)—N(2)'	1.51 (2)	C(2)—N(2)—C(3)'	108.7 (7)
C(4)—N(1)	1.51 (2)	C(2)—N(2)—C(4)	108.3 (8)
C(4)—N(2)	1.47 (2)	C(4)—N(2)—C(3)'	107.2 (12)
N(1)···O(1)	2.74 (2)	N(1)—C(1)—N(1)'	111.0 (10)
		N(2)—C(2)—N(2)'	112.7 (14)
		N(1)—C(3)—N(2)	111.3 (9)
		N(1)—C(4)—N(2)	111.8 (9)

gen bonds to give a three-dimensional network. The fact that the present adduct slowly decomposes upon exposure to air is explicable in terms of the lack of extensive hydrogen-bonding in the crystal (Fig. 2). 1:2 adducts of HMT with other substituted phenols (e.g. *p*-chlorophenol and *p*-bromophenol) are also relatively unstable; presumably in each case the basic structural unit is a 1:2 hydrogen-bonded molecular complex corresponding to the stoichiometric formula.

The measured dimensions of HMT in the present adduct (Table 2) are in good agreement with those in crystalline HMT (Becka & Cruickshank, 1963). The N(1)···H—O(1) bond of 2.74 (2) Å is normal and there are no unusual van der Waals contacts.

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Sendanolactone, a New Triterpenoid from *Melia azedarach* L. var. *japonica* Makino

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Abstract. C₃₀H₄₂O₄, 3,6-dioxo-13 α ,14 β ,17 α -lanosta-7,24-dien-21,16 β -olide, orthorhombic, *P*2₁2₁2₁, *a* = 30.325 (2), *b* = 12.121 (1), *c* = 7.109 (1) Å, *Z* = 4, *D*_x = 1.19, *D*_m = 1.18 g cm⁻³. The structure was solved by the direct method and refined by a block-diagonal least-squares technique to *R* = 0.075 for 1666 reflexions. Rings *A*, *C* and *D* are in the chair, boat and envelope conformations respectively.

Introduction. Crystals were obtained from a methanol solution. The systematic absences are *h*00 when *h* is

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odd, 0*k*0 when *k* is odd, and 00*l* when *l* is odd. Intensities were collected by the θ -2 θ scan technique on a Hilger & Watts Y-290 diffractometer with Zr-filtered Mo *K* α radiation and a crystal of dimensions 0.2 × 0.2 × 0.3 mm. 1998 independent reflexions with $\theta \leq 22.5^\circ$ were corrected for Lorentz and polarization effects, but not for absorption. The structure was solved by the use of the program *MULTAN* (Main, Germain & Woolfson, 1970) with local modifications on a FACOM 270-30 computer. After anisotropic refinement of the non-hydrogen atoms by the block-diagonal

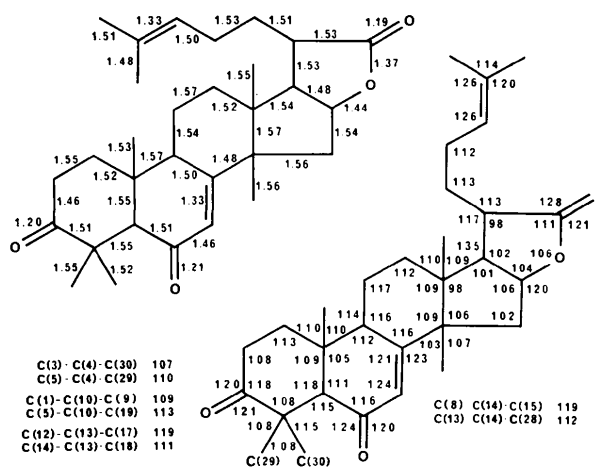
Table 1. Atomic fractional coordinates ($\times 10^4$, for H $\times 10^3$) for sendanolactone

	x	y	z		x	y	z
C(1)	5294 (2)	7601 (5)	5060 (11)	H(C5)	518	950	310
C(2)	5805 (2)	7630 (6)	5012 (16)	H(C7)	446	1144	520
C(3)	5942 (2)	8449 (6)	3630 (14)	H(C9)	458	856	390
C(4)	5787 (2)	9626 (5)	3874 (10)	H(C11)	450	688	550
C(5)	5280 (2)	9599 (5)	4184 (9)	H'(C11)	444	781	750
C(6)	5073 (2)	10715 (5)	4553 (9)	H(C12)	376	713	535
C(7)	4605 (2)	10707 (4)	5052 (9)	H'(C12)	378	769	750
C(8)	4376 (2)	9790 (5)	5379 (8)	H(C15)	368	1113	450
C(9)	4586 (2)	8675 (4)	5150 (8)	H'(C15)	358	1119	685
C(10)	5094 (2)	8716 (5)	5558 (9)	H(C16)	324	1028	360
C(11)	4345 (2)	7712 (5)	6123 (9)	H(C17)	319	922	690
C(12)	3826 (2)	7740 (5)	6070 (9)	H(C18)	345	844	250
C(13)	3659 (2)	8793 (4)	5162 (8)	H'(C18)	369	953	225
C(14)	3913 (2)	9803 (5)	6039 (8)	H''(C18)	395	838	260
C(15)	3623 (2)	10840 (5)	5648 (10)	H(C19)	508	963	800
C(16)	3200 (2)	10315 (5)	4851 (9)	H'(C19)	500	875	840
C(17)	3186 (2)	9173 (4)	5591 (8)	H''(C19)	544	891	790
C(18)	3719 (2)	8744 (5)	3005 (9)	H(C20)	275	859	370
C(19)	5177 (2)	8990 (6)	7635 (10)	H(C22)	256	788	815
C(20)	2725 (2)	8802 (5)	5010 (9)	H'(C22)	224	791	610
C(21)	2486 (2)	9908 (6)	5235 (8)	H(C23)	288	672	475
C(22)	2519 (2)	7889 (6)	6167 (10)	H'(C23)	250	616	625
C(23)	2588 (2)	6747 (5)	5314 (10)	H(C24)	199	656	425
C(24)	2276 (2)	6526 (6)	3722 (11)	H(C26)	175	631	100
C(25)	2381 (2)	6407 (5)	1922 (10)	H'(C26)	209	678	-35
C(26)	2020 (3)	6287 (7)	468 (12)	H''(C26)	200	563	0
C(27)	2831 (2)	6438 (7)	1137 (11)	H(C27)	288	734	100
C(28)	3958 (2)	9704 (6)	8224 (9)	H'(C27)	288	625	-15
C(29)	5898 (3)	10275 (6)	2063 (11)	H''(C27)	288	550	125
C(30)	6053 (2)	10116 (6)	5484 (13)	H(C28)	373	963	875
O(31)	6173 (3)	8189 (5)	2338 (14)	H'(C28)	408	894	875
O(32)	5270 (2)	11583 (3)	4430 (8)	H''(C28)	408	1019	880
O(33)	2777 (1)	10766 (3)	5333 (7)	H(C29)	620	1016	150
O(34)	2099 (1)	10061 (4)	5339 (7)	H'(C29)	589	1084	245
H(C1)	516	703	595	H''(C29)	568	1013	100
H'(C1)	519	719	375	H(C30)	594	1078	600
H(C2)	593	719	535	H'(C30)	625	969	600
H'(C2)	591	819	625	H''(C30)	619	1078	500

least-squares technique, the R value converged to 0.075 for 1666 reflexions with $|F_c| > \sigma(F_o)$ and $|\Delta F| < 3\sigma(F_o)$. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974).^{*} Final positional parameters are in Table 1.

Discussion. The molecular structure of sendanolactone, isolated from a methanol extract of the bark of *Melia azedarach* L. var. *japonica* Makino, has been determined on the basis of chemical and spectroscopic evidence (Ochi, Kotsuki, Tokoroyama & Kubota, 1977); the configuration at C(20) is determined here.

^{*} Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33438 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Fig. 1. Bond distances (Å) and angles ($^{\circ}$).

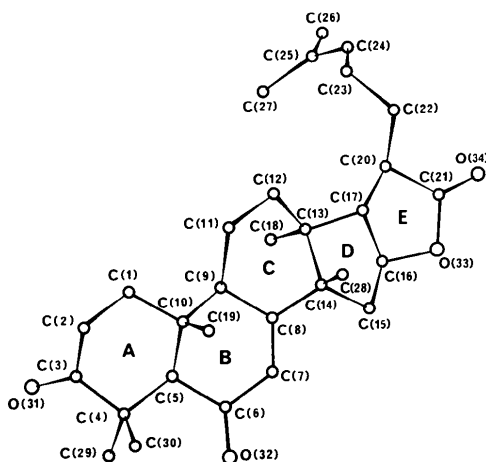


Fig. 2. Perspective view of the molecule with the atom-numbering and ring-labelling systems.

Bond distances and angles are given in Fig. 1. The mean e.s.d. values are 0.01 Å for bond lengths and 1° for angles. A perspective view of the molecule is shown

in Fig. 2. Rings *A*, *C* and *D* adopt the chair, boat and envelope conformations respectively, though they are somewhat deformed. C(10) in ring *B* is displaced 0.74 Å out of the mean plane through the rest of the atoms (mean and maximum deviations are 0.03 and 0.04 Å respectively), and C(17) in ring *E* is displaced by 0.61 Å (0.04 and 0.05 Å).

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5-(*p*-Methoxyphenyl)-3-methoxy-2,4-pentadien-4-olide

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Abstract. C₁₃H₁₂O₄, *M_r* = 232.23, orthorhombic, *Pbca*, *a* = 13.77 (2), *b* = 7.78 (2), *c* = 21.19 (2) Å, from diffractometer measurements (Mo *K*α radiation). *V* = 2270.9 Å³, *Z* = 8, *D_c* = 1.370 g cm⁻³, *F*(000) = 976, *μ* = 0.61 cm⁻¹, approximate crystal dimensions 0.22 × 0.38 × 0.22 mm. The stereochemistry about the 4,5 double bond has been established.

Introduction. The title compound (I) was recrystallized from dimethyl sulphoxide. Systematic absences (from precession photographs) *hk0 h = 2n + 1*, *h0l l = 2n + 1*, *0kl k = 2n + 1* indicated space group *Pbca*. Data were collected for *h0–7l* with *θ_{max}* = 27.5° on a Stoe STADI-2 two-circle diffractometer (graphite-monochromated Mo *K*α radiation). This gave 2266 data of which 1377 unique reflexions with *I* > 3σ(*I*) were used in subsequent calculations. Lorentz and polarization corrections (but none for extinction or absorption) were

applied, and the data scaled by a Wilson plot. The structure was solved by direct methods with *SHELX 76* (Sheldrick, 1976), which was used for all the calculations. Complex neutral scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Weighted full-matrix least-squares refinement (including isotropic H atoms) converged at *R* = 0.054 for 1377 observed reflexions (*R* = ∑||*F_o*| -

