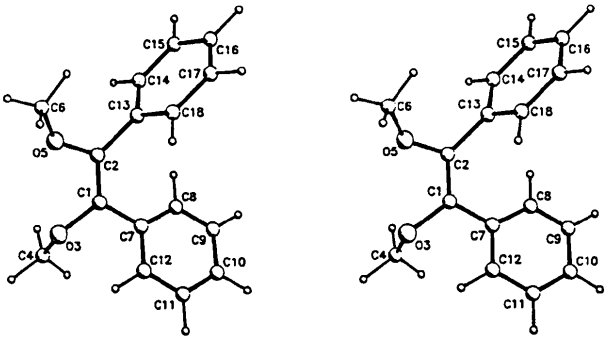

Fig. 1. Stereoscopic view of (*Z*)-α,β-dimethoxystilbene.

The atomic parameters are given in Table 1.* Bond lengths and bond angles are listed in Table 2. Fig. 1 is a stereoscopic view of the compound, showing the numbering of the atoms (*PLUTO*; Motherwell & Clegg, 1978).

Related literature. The compound has been synthetized following the procedure described by Merz & Tomahogh (1977). The structures of the sulfur analogues (*E*)- and (*Z*)-1,2-bis(methylthio)-1,2-diphenylethylene (Adiwidjaja, Kistenbrugger & Voss, 1981) and those of *cis*- and *trans*-1,2-bis(methoxyethoxy)-1,2-diphenylethylene (Soumillion, Weiler, De Man, Touillaux, Declercq & Tinant, 1989) have been reported. From dipole-moment measurements, Lumbroso, Lund & Simonet (1974) have discussed the *cis–trans* configurations and the conformation of the methoxy and phenyl groups in the title compound.

* 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 55362 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA0102]

References

ADIWIDJAJA, G., KISTENBRUGGER, L. & VOSS, J. (1981). *J. Chem. Res.* (*S*), pp. 1227–1228.

LUMBROSO, H., LUND, H. & SIMONET, J. (1974). *C. R. Acad. Sci. Sér. C*, **278**, 1449–1452.

MERZ, A. & TOMAHOGH, R. (1977). *J. Chem. Res.* (*M*), pp. 3070–3084.

MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

SHELDRICK, G. M. (1985). *SHELXS86*. In *Crystallographic Computing* 3, edited by G. M. SHELDRICK, C. KRÜGER & R. GODDARD, pp. 175–189. Oxford Univ. Press.

SOUMILLION, J. PH., WEILER, J., DE MAN, X., TOUILLAUX, R., DECLERCQ, J.-P. & TINANT, B. (1989). *Tetrahedron Lett.* **30**, 4509–4512.

Acta Cryst. (1992). C**48**, 2251–2253

Structure of Dimethyl Ceanothate

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(*Received* 10 *January* 1992; *accepted* 13 *April* 1992)

Abstract. Methyl 2α-methoxycarbonyl-3β-hydroxy-*A*(1)-norlup-20(29)-en-28-oate, $C_{32}H_{50}O_5$, M_r = 514.7, orthorhombic, $P2_12_12_1$, a = 9.795 (2), b = 16.452 (2), c = 18.835 (2) Å, V = 3035.2 (2) Å^3, Z = 4, D_x = 1.13 g cm^{-3}, λ(Cu $K\alpha$) = 1.5418 Å, μ = 5.52 cm^{-1}, $F(000)$ = 1128, T = 295 K, R = 0.049, wR = 0.057 for 2481 unique observed reflections [$I > 2\sigma(I)$]. Ring *A* is in a half-chair conformation and ring *E* is in an envelope conformation. Rings *B*, *C* and *D* have slightly distorted chair conformations with mean torsion angles of 54.8 (4), 58.4 (4) and 55.5 (4)°, respectively. The molecule is stabilized by van der Waals forces.

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0108-2701/92/122251-03$06.00

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^3$) *for non-H atoms with e.s.d.'s in parentheses*

$$U_{eq} = (U_{11} + U_{22} + U_{33})/3.$$

	x	y	z	U_{eq}
C1	1670 (4)	3655 (2)	−486 (2)	51 (1)
C2	2837 (4)	4184 (2)	−248 (2)	47 (1)
C3	3765 (4)	4430 (3)	−892 (2)	53 (1)
C4	3418 (4)	5338 (2)	−1080 (2)	55 (1)
C5	2177 (4)	5517 (2)	−611 (2)	45 (1)
C6	1729 (4)	6373 (2)	−457 (2)	58 (1)
C7	331 (4)	6349 (2)	−91 (2)	55 (1)
C8	290 (3)	5846 (2)	603 (2)	39 (1)
C9	966 (3)	4989 (2)	465 (1)	34 (1)
C10	2357 (3)	5013 (2)	72 (2)	39 (1)
C11	958 (4)	4471 (2)	1140 (2)	45 (1)
C12	−469 (4)	4331 (2)	1405 (2)	45 (1)
C13	−1204 (3)	5135 (2)	1541 (1)	37 (1)
C14	−1240 (3)	5677 (2)	853 (2)	38 (1)
C15	−1996 (4)	6488 (2)	1017 (2)	48 (1)
C16	−3368 (4)	6392 (2)	1391 (2)	50 (1)
C17	−3223 (3)	5880 (2)	2064 (2)	45 (1)
C18	−2633 (3)	5053 (2)	1864 (1)	39 (1)
C19	−2858 (4)	4495 (2)	2516 (2)	48 (1)
C20	−3155 (5)	3610 (2)	2372 (2)	65 (1)
C21	−4096 (5)	4906 (3)	2910 (2)	67 (1)
C22	−4533 (4)	5630 (2)	2459 (2)	57 (1)
C23	4612 (5)	5925 (3)	−973 (3)	82 (2)
C24	3010 (5)	5368 (3)	−1867 (2)	78 (2)
C25	3550 (4)	5286 (3)	555 (2)	56 (1)
C26	1057 (4)	6331 (2)	1181 (2)	57 (1)
C27	−2083 (4)	5256 (2)	261 (2)	48 (1)
C28	−2362 (4)	6323 (2)	2615 (2)	55 (1)
C29	−2695 (12)	3049 (4)	2825 (6)	175 (5)
C30	−4012 (12)	3392 (5)	1823 (4)	178 (4)
O31	−2693 (5)	7104 (2)	2648 (2)	95 (1)
C32	−2041 (9)	7586 (3)	3196 (3)	130 (3)
O33	1395 (4)	3079 (2)	−12 (2)	82 (2)
C34	173 (9)	2605 (4)	−130 (3)	123 (3)
O35	5141 (3)	4295 (2)	−680 (2)	75 (1)
O36	1023 (4)	3739 (2)	1020 (1)	76 (1)
O37	−1524 (3)	6027 (2)	3000 (1)	73 (1)

Experimental. Ceanothic acid was isolated (Kundu, Barik, Mondal, Dey & Banerji, 1989) from both the bark and the roots of *Zizyphus jujuba* as an intermediate product while extracting a pentacyclic triterpenoid zizyberanalic acid. It was then methylated with ethereal CH_2N_2 to yield the title compound. Crystals were grown from an ethanol/acetone mixture at room temperature. Data were collected for a colourless transparent crystal ($0.30 \times 0.30 \times 0.35$ mm) with an Enraf–Nonius CAD-4 diffractometer using Ni-filtered Cu $K\alpha$ radiation. Unit-cell parameters were derived from a least-squares analysis of 25 reflections with $25 \le 2\theta \le 35°$. Intensity data were collected with the ω–2θ scan technique, 2986 unique reflections (h 0 to 11, k 0 to 20, l 0 to 22) up to $2\theta = 140°$ were measured, of which 2481 were considered observed with $I > 2\sigma(I)$. During data collection three standard reflections, monitored after every 2 h of X-ray exposure, indicated no decay over the full 34 h period. The intensity data were corrected for Lorentz, polarization and absorption effects (ψ-scan method, transmission-factor range 0.951–0.996). The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1990) and refined on F by weighted full-matrix least squares on a MicroVAX II computer with *SHELX*76 (Sheldrick, 1976).

Table 2. *Bond lengths* (Å), *bond angles* (°) *and ring torsion angles* (°)

C1—C2	1.505 (5)	C12—C13	1.528 (5)
C1—O36	1.197 (5)	C13—C14	1.573 (4)
C1—O33	1.330 (5)	C13—C18	1.532 (4)
C2—C3	1.569 (5)	C14—C15	1.557 (5)
C2—C10	1.564 (5)	C14—C27	1.551 (5)
C3—C4	1.572 (6)	C15—C16	1.526 (6)
C3—O35	1.432 (5)	C16—C17	1.529 (5)
C4—C23	1.530 (6)	C17—C18	1.526 (5)
C4—C24	1.536 (5)	C17—C28	1.523 (5)
C4—C5	1.531 (5)	C17—C22	1.539 (5)
C5—C10	1.541 (5)	C18—C19	1.549 (4)
C5—C6	1.503 (5)	C19—C21	1.574 (6)
C6—C7	1.534 (6)	C19—C20	1.509 (5)
C7—C8	1.548 (5)	C20—C29	1.335 (10)
C8—C9	1.579 (5)	C20—C30	1.379 (10)
C8—C26	1.545 (5)	C21—C22	1.524 (6)
C8—C14	1.595 (4)	C28—O37	1.199 (5)
C9—C10	1.551 (4)	C28—O31	1.327 (5)
C9—C11	1.531 (4)	O31—C32	1.450 (7)
C11—C12	1.502 (6)	O33—C34	1.446 (9)
C10—C25	1.548 (5)		

O36—C1—O33	122.6 (4)	C14—C13—C18	110.9 (2)
C2—C1—O33	111.5 (3)	C14—C13—C12	111.3 (3)
O36—C1—C2	125.9 (3)	C9—C11—C12	111.5 (3)
C1—C2—C3	111.0 (3)	C13—C14—C27	110.6 (3)
C10—C2—C3	104.3 (3)	C13—C14—C15	109.5 (3)
C10—C2—C1	113.0 (3)	C13—C14—C8	108.7 (2)
O35—C3—C2	107.1 (3)	C15—C14—C27	105.8 (3)
C4—C3—O35	114.5 (4)	C15—C14—C8	110.9 (3)
C4—C3—C2	107.1 (3)	C8—C14—C27	111.5 (3)
C23—C4—C24	107.8 (3)	C14—C15—C16	115.0 (3)
C23—C4—C3	113.9 (3)	C16—C17—C18	108.8 (3)
C24—C4—C3	107.7 (3)	C16—C17—C28	110.7 (3)
C23—C4—C5	114.2 (3)	C16—C17—C22	118.1 (3)
C24—C4—C5	110.1 (3)	C18—C17—C28	112.7 (3)
C3—C4—C5	103.0 (3)	C18—C17—C22	101.4 (3)
C4—C5—C10	106.7 (3)	C22—C17—C28	105.1 (3)
C4—C5—C6	121.6 (3)	C17—C18—C19	106.2 (3)
C6—C5—C10	112.1 (3)	C17—C18—C13	111.5 (2)
C5—C6—C7	108.9 (3)	C13—C18—C19	119.8 (3)
C6—C7—C8	114.6 (3)	C18—C19—C20	117.2 (3)
C7—C8—C9	109.1 (3)	C18—C19—C21	103.2 (3)
C7—C8—C26	107.8 (3)	C20—C19—C21	110.5 (3)
C7—C8—C14	111.5 (3)	C19—C20—C29	119.1 (5)
C26—C8—C14	109.8 (3)	C19—C20—C30	120.2 (4)
C8—C9—C10	115.1 (3)	C30—C20—C29	120.3 (6)
C10—C9—C11	114.5 (3)	C21—C22—C17	104.1 (3)
C9—C10—C25	113.0 (3)	C17—C28—O37	126.7 (3)
C9—C10—C5	108.2 (3)	C17—C28—O31	111.1 (3)
C9—C10—C2	115.2 (3)	O31—C28—O37	122.2 (3)
C5—C10—C25	114.9 (3)	C28—O31—C32	117.1 (4)
C5—C10—C2	100.5 (3)	C1—O33—C34	116.7 (4)
C25—C10—C2	104.6 (3)	C11—C12—C13	111.2 (3)
C15—C16—C17	111.0 (3)	C8—C9—C11	111.0 (3)
C9—C8—C14	106.7 (3)	C19—C21—C22	106.8 (3)
C5—C4—C23	114.2 (3)		

Ring *A*

C2—C3—C4—C5	−8.0 (4)
C3—C4—C5—C10	32.3 (4)
C4—C5—C10—C2	−43.7 (3)
C5—C10—C2—C3	36.8 (3)
C10—C2—C3—C4	−18.2 (4)

Ring *B*

C5—C6—C7—C8	57.3 (4)
C6—C7—C8—C9	−49.2 (4)
C7—C8—C9—C10	47.5 (3)
C8—C9—C10—C5	−52.6 (3)
C9—C10—C5—C6	59.9 (4)
C10—C5—C6—C7	−62.2 (4)

Ring *C*

C8—C9—C11—C12	59.6 (4)
C9—C11—C12—C13	−57.0 (4)
C11—C12—C13—C14	57.2 (4)
C12—C13—C14—C8	−58.8 (3)
C13—C14—C8—C9	58.7 (3)
C14—C8—C9—C11	−59.8 (3)

Ring *D*

C13—C14—C15—C16	−49.1 (4)
C14—C15—C16—C17	53.5 (4)
C15—C16—C17—C18	−57.8 (4)
C16—C17—C18—C13	62.2 (3)
C17—C18—C13—C14	−59.5 (3)
C18—C13—C14—C15	50.8 (3)

Ring *E*

C17—C18—C19—C21	23.8 (3)
C18—C19—C21—C22	2.3 (4)
C19—C21—C22—C17	−27.2 (4)
C21—C22—C17—C18	41.4 (3)
C22—C17—C18—C19	−40.6 (3)

38 H atoms were located from a $\Delta\rho$ map while others were fixed from stereochemical considerations. All the H atoms were refined with isotropic displacement parameters in the final cycles. Final maximum $\Delta/\sigma = 0.08$ and maximum and minimum heights in final $\Delta\rho$ maps were 0.19 and -0.26 e Å^{-3}, respectively. Refinement of 534 parameters with weights given by $w = [\sigma^2(F) + 0.004896(F_o^2)]^{-1}$ converged at $R = 0.049$, $wR = 0.057$ and $S = 0.97$. Atomic scattering factors were those of *SHELX76* taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Final positional and displacement parameters are listed in Table 1* and the bond lengths and angles obtained using *PARST* (Nardelli, 1983) are in Table 2. A *PLUTO* (Motherwell & Clegg, 1978) drawing of the molecule with atom numbering and ring labelling is presented in Fig. 1.

Related literature. Several C—C bonds and C—C—C angles deviate by more than 3σ from their respective expected values. Similar features are also observed in the fused ring systems with axial substitutions by bulky methyl groups (Hall & Maslen, 1965; Gzella, Zaprutko, Wrzeciono & Jaskólski, 1987). The C20—C30 bond distance is unusually short as in the isopropyl side chain of methyl melaleucate iodoacetate (Hall & Maslen, 1965).

* Lists of structure factors, anisotropic displacement parameters, H-atom parameters and least-squares-planes calculations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55378 (24 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1003]

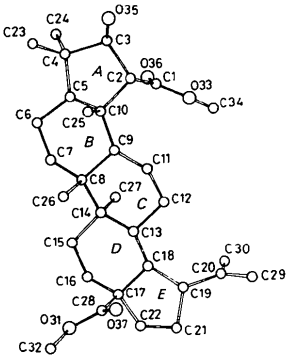

Fig. 1. Molecular structure of the title compound with ring labelling.

Thanks are due to the Council of Scientific and Industrial Research, India, for the award of a Senior Research Fellowship to KS.

References

GZELLA, A., ZAPRUTKO, L., WRZECIONO, U. & JASKÓLSKI, M. (1987). *Acta Cryst.* C**43**, 759–762.

HALL, S. R. & MASLEN, E. N. (1965). *Acta Cryst.* **18**, 265–279.

KUNDU, A. B., BARIK, B. R., MONDAL, D. N., DEY, A. K. & BANERJI, A. (1989). *Phytochemistry*, **28**, 3155–3158.

MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

NARDELLI, M. (1983). *Comput. Chem.* **7**, 95–98.

SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

SHELDRICK, G. M. (1990). *Acta Cryst.* A**46**, 467–473.