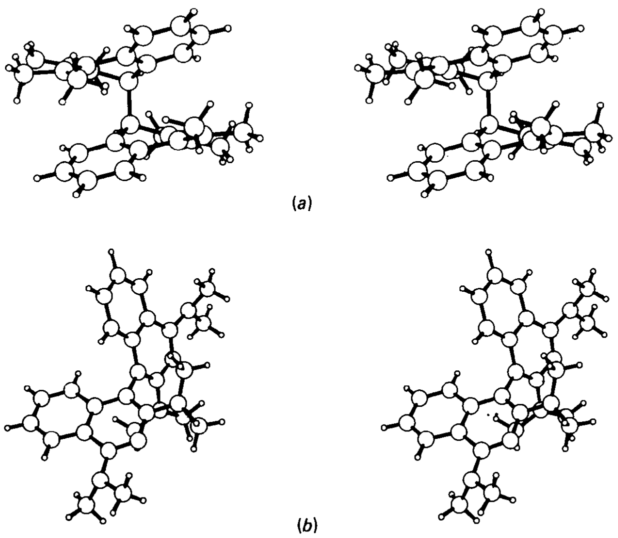

Fig. 2. (*a*) Stereoview of the dication of (II), viewed parallel to the *b* axis. (*b*) Stereoview of the molecule of (III).

In the crystal, some distances between methyl groups are significantly shorter than 4·0 Å which is twice the van der Waals radius for a methyl group. This is presumably achieved by inhibition of free rotation of the methyl groups.

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Structure of a Novel Oxygenated Dimer: Dilongicyclyl-12-ether*

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Abstract. Bis[(1,2,4-metheno-5,5,8a-trimethylperhydroazulen-1-yl)methyl] ether {Bis[(2,6,6-trimethyltetracyclo[$5.3.1.0^{2,7}.0^{10,11}$]undecyl)methyl] ether}, $C_{30}H_{46}O$, $M_r = 422·7$, orthorhombic, $P2_12_12_1$, $a = 7·450\ (2)$, $b = 15·886\ (2)$, $c = 21·466\ (2)$ Å, $V = 2540·5$ Å^3, D_m(flotation in aqueous KI solution) = 1·122 (6), $D_x = 1·105$ Mg m^{-3}, $Z = 4$, $\lambda(\text{Mo}\ K\alpha) = 0·7207$ Å, $\mu(\text{Mo}\ K\alpha) = 0·69$ mm^{-1}, $F(000) = 936$, $T = 293$ K, $R = 0·068$ for 1104 observed reflections. The oxygen atom bridges the two monomeric units, one of which has disorder in the seven-membered-ring moiety.

Introduction. While studying Lewis-acid-induced rearrangements *via* longifolene chemistry, pseudolongifolol (Mehta, Nayak & Sukh Dev, 1968) in benzene was exposed to boron trifluoride etherate; silica gel column chromatography of the resulting mixture afforded one pure crystalline compound $C_{30}H_{46}O$ (M^+ 422), in major yield (45%) (Lamture, Surayawanshi & Nayak, 1982).

The structure could not easily be deduced from spectral data and X-ray analysis was required to establish this unequivocally.

Experimental. Crystal 0·20 × 0·35 × 0·70 mm; Nonius CAD-4F-11M single-crystal X-ray diffractometer; graphite-monochromated Mo $K\alpha$ radiation; $\omega/2\theta$ scan mode; scan speed 1° min^{-1}, $\theta < 23·5°$, h 0 to 8, k 0 to 17, l 0 to 24, 2172 reflections collected, 1104 judged significant [$|F_o| > 3\sigma(|F_o|)$], lattice parameters from 21 reflections ($15 < 2\theta < 35°$), three standard reflections (124, 042 and 105) every 1000 s, 2% variation in intensity, no correction for absorption. Structure solved by direct methods using *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). During the final stages of refinement, it was realized that two of the atoms, C(4′) and C(5′), are disordered. A careful examination of a difference Fourier map at this stage indicated clearly the existence of these disordered atoms C(4″) and C(5″) which were refined with occupancies 0·4 and 0·5. Attempts to determine the absolute configuration of the molecule,

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since the space group is chiral, were unsuccessful because of the presence of disorder in the atoms C(4′) and C(5′). Full-matrix least-squares refinement of scale factor, positional and anisotropic thermal parameters converged to $R(F) = 0{\cdot}068$, $wR = 0{\cdot}068$ with unit weights; $S = 1{\cdot}66$. $(\Delta/\sigma)_{\max} = 0{\cdot}1$. Final $\Delta\rho$ excursions $< |0{\cdot}3 \pm$ e Å^{-3}. No correction for secondary extinction. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Program *LALS* (Gantzel, Sparks & Trueblood, 1961) used for refinement. H atoms were held fixed during refinement.

Discussion. The atomic parameters with their e.s.d.'s and equivalent isotropic temperature factors are given in Table 1.* Bond lengths and bond angles involving the non-hydrogen atoms are given in Table 2. Fig. 1 shows the chemical formula together with the numbering of the atoms. Fig. 2 shows a perspective view of the molecule along with the disordered atoms. It is conjectured that two molecules of pseudolongifolol are bridged (dimerized) through the O atom with the removal of a water molecule in the presence of BF_3–OEt_2.

An analysis of the flexibility of the norbornane moiety (Acharya, Tavale & Guru Row, 1984) indicates that the twist is restricted due to the formation of a three-membered ring involving the atoms C(8), C(9) and C(10) and also C(8′), C(9′) and C(10′). The cycloheptane moiety [C(1) to C(7)] has neither C_2 nor C_s symmetry and the ratio suggested (McPhail & Sim, 1973) has a value of 0·88 which is also close to the chair conformation (Bixon & Lifson, 1967).

The two monomeric units are *trans* fused at the bridge oxygen [torsional angles C(9)—C(12)—O—C(12′) = 160·7 (9) and C(9′)—C(12′)—O—C(12) = 170·1 (9)°]. The crystal structure is stabilized by van der Waals interactions.

Table 1. *Atomic coordinates* (×10⁴) *and equivalent isotropic thermal parameters for non-hydrogen atoms with e.s.d.'s in parentheses*

$$B_{\rm eq} = \tfrac{8}{3}\pi^2 \textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$(Å^2)
O	4631 (11)	3864 (4)	3695 (3)	4·16
C(1)	2541 (11)	4622 (5)	5545 (4)	3·22
C(2)	3876 (15)	4031 (6)	5197 (4)	4·33
C(3)	5787 (13)	4288 (6)	5103 (4)	3·54
C(4)	6245 (14)	5138 (6)	4778 (5)	4·73
C(5)	5532 (14)	5890 (6)	5120 (5)	5·00
C(6)	3576 (16)	6118 (6)	5042 (4)	4·51
C(7)	2189 (11)	5395 (5)	5080 (4)	2·83
C(8)	1810 (13)	4846 (6)	4539 (4)	3·68
C(9)	2713 (11)	3979 (6)	4600 (4)	2·83
C(10)	758 (15)	4068 (6)	4783 (5)	4·35
C(11)	713 (10)	4201 (5)	5484 (4)	4·37
C(12)	3192 (13)	3465 (6)	4017 (4)	3·96
C(13)	2972 (17)	6765 (7)	5555 (5)	6·95
C(14)	3232 (16)	6577 (5)	4405 (5)	6·09
C(15)	3999 (14)	3124 (5)	5510 (5)	5·21
C(1′)	8140 (20)	4445 (7)	2068 (4)	6·08
C(2′)	6357 (15)	4068 (6)	2208 (4)	4·80
C(3′)	4592 (17)	4661 (8)	2198 (6)	7·15
C(4′)	4981 (36)	5617 (18)	2252 (12)	8·64
C(5′)	5961 (32)	5922 (11)	2826 (12)	5·70
C(6′)	7937 (20)	5900 (8)	2690 (7)	4·62
C(7′)	8715 (17)	5009 (8)	2638 (5)	4·35
C(8′)	8165 (20)	4361 (8)	3147 (6)	3·89
C(9′)	6803 (16)	3774 (7)	2868 (5)	3·59
C(10′)	8847 (25)	3548 (11)	2861 (8)	7·06
C(11′)	9504 (25)	3678 (10)	2216 (8)	6·62
C(12′)	5482 (20)	3314 (8)	3296 (6)	5·22
C(13′)	8510 (27)	6243 (11)	3337 (8)	7·56
C(14′)	9201 (28)	6408 (11)	2201 (8)	9·14
C(15′)	5935 (25)	3321 (10)	1785 (8)	7·65
C(4″)*	4582 (36)	5512 (26)	2662 (16)	8·56
C(5″)*	6211 (35)	6063 (20)	2465 (14)	5·96

* Occupancies: C(4′) 0·6, C(4″) 0·4, C(5′) 0·5, C(5″) 0·5.

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

O—C(12)	1·425 (12)
O—C(12′)	1·378 (15)
C(1)—C(2)	1·558 (13)
C(1)—C(7)	1·603 (12)
C(1)—C(11)	1·523 (11)
C(2)—C(3)	1·495 (15)
C(2)—C(9)	1·550 (13)
C(2)—C(15)	1·592 (13)
C(3)—C(4)	1·558 (14)
C(4)—C(5)	1·500 (14)
C(5)—C(6)	1·511 (16)
C(6)—C(7)	1·547 (13)
C(6)—C(13)	1·572 (14)
C(6)—C(14)	1·570 (14)
C(7)—C(8)	1·480 (12)
C(8)—C(9)	1·540 (13)
C(8)—C(10)	1·556 (14)
C(9)—C(10)	1·516 (14)
C(9)—C(12)	1·534 (13)
C(10)—C(11)	1·520 (14)
C(1′)—C(2′)	1·488 (18)
C(1′)—C(7′)	1·576 (15)
C(1′)—C(11′)	1·619 (21)
C(2′)—C(3′)	1·617 (17)
C(2′)—C(9′)	1·528 (14)
C(2′)—C(15′)	1·529 (19)
C(3′)—C(4′)	1·551 (31)
C(4′)—C(5′)	1·512 (36)
C(5′)—C(6′)	1·501 (28)
C(6′)—C(7′)	1·533 (18)
C(6′)—C(13′)	1·551 (23)
C(6′)—C(14′)	1·625 (24)
C(7′)—C(8′)	1·556 (18)
C(8′)—C(9′)	1·503 (18)
C(8′)—C(10′)	1·517 (22)
C(9′)—C(10′)	1·565 (22)
C(9′)—C(12′)	1·530 (18)
C(10′)—C(11′)	1·484 (24)
C(12)—O—C(12′)	111·5 (8)
C(2)—C(1)—C(7)	105·6 (7)
C(2)—C(1)—C(11)	105·4 (7)
C(7)—C(1)—C(11)	97·9 (6)
C(1)—C(2)—C(3)	120·5 (8)
C(1)—C(2)—C(9)	94·1 (7)
C(1)—C(2)—C(15)	112·4 (8)
C(3)—C(2)—C(9)	115·8 (8)
C(3)—C(2)—C(15)	104·5 (8)
C(9)—C(2)—C(15)	109·4 (8)
C(2)—C(3)—C(4)	120·4 (8)
C(3)—C(4)—C(5)	113·2 (8)
C(4)—C(5)—C(6)	118·5 (9)
C(5)—C(6)—C(7)	117·4 (8)
C(5)—C(6)—C(13)	110·8 (8)
C(5)—C(6)—C(14)	111·4 (8)
C(7)—C(6)—C(13)	104·9 (8)
C(7)—C(6)—C(14)	106·4 (8)
C(13)—C(6)—C(14)	105·0 (8)
C(1)—C(7)—C(6)	119·5 (7)
C(1)—C(7)—C(8)	93·9 (6)
C(6)—C(7)—C(8)	121·5 (8)
C(7)—C(8)—C(9)	112·2 (7)
C(7)—C(8)—C(10)	107·5 (8)
C(9)—C(8)—C(10)	58·7 (6)
C(2)—C(9)—C(8)	105·4 (7)
C(2)—C(9)—C(10)	108·5 (8)
C(2)—C(9)—C(12)	124·9 (8)
C(8)—C(9)—C(10)	61·2 (6)
C(8)—C(9)—C(12)	120·6 (8)
C(10)—C(9)—C(12)	119·0 (8)
C(8)—C(10)—C(9)	60·1 (6)
C(8)—C(10)—C(11)	103·5 (8)
C(9)—C(10)—C(11)	106·9 (8)
C(1)—C(11)—C(10)	97·2 (7)
O—C(12)—C(9)	109·5 (7)
C(2′)—C(1′)—C(7′)	108·3 (9)
C(2′)—C(1′)—C(11′)	102·6 (10)
C(7′)—C(1′)—C(11′)	96·1 (10)
C(1′)—C(2′)—C(3′)	119·2 (9)
C(1′)—C(2′)—C(9′)	96·7 (8)
C(1′)—C(2′)—C(15′)	112·1 (10)
C(3′)—C(2′)—C(9′)	111·6 (9)
C(3′)—C(2′)—C(15′)	106·1 (10)
C(9′)—C(2′)—C(15′)	111·0 (10)
C(2′)—C(3′)—C(4′)	114·7 (13)
C(3′)—C(4′)—C(5′)	117·8 (20)
C(4′)—C(5′)—C(6′)	108·0 (18)
C(5′)—C(6′)—C(7′)	114·0 (13)
C(5′)—C(6′)—C(13′)	95·1 (14)
C(5′)—C(6′)—C(14′)	133·0 (14)
C(7′)—C(6′)—C(13′)	106·6 (12)
C(7′)—C(6′)—C(14′)	101·1 (11)
C(13′)—C(6′)—C(14′)	104·1 (12)
C(1′)—C(7′)—C(6′)	118·5 (10)
C(1′)—C(7′)—C(8′)	95·5 (9)
C(6′)—C(7′)—C(8′)	117·4 (11)
C(7′)—C(8′)—C(9′)	108·1 (10)
C(7′)—C(8′)—C(10′)	101·1 (11)
C(9′)—C(8′)—C(10′)	62·4 (10)
C(2′)—C(9′)—C(8′)	109·0 (9)
C(2′)—C(9′)—C(10′)	105·9 (10)
C(2′)—C(9′)—C(12′)	124·2 (10)
C(8′)—C(9′)—C(10′)	59·3 (9)
C(8′)—C(9′)—C(12′)	119·5 (10)
C(10′)—C(9′)—C(12′)	121·5 (11)
C(8′)—C(10′)—C(9′)	58·3 (9)
C(8′)—C(10′)—C(11′)	111·6 (14)
C(9′)—C(10′)—C(11′)	107·3 (13)
C(1′)—C(11′)—C(10′)	94·6 (12)
O—C(12′)—C(9′)	111·5 (10)

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* 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 43738 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

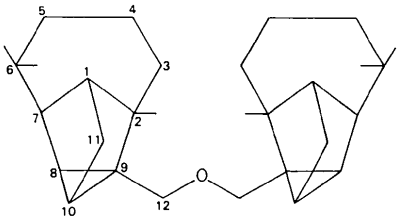

Fig. 1. Chemical formula of the title molecule, with atom numbering.

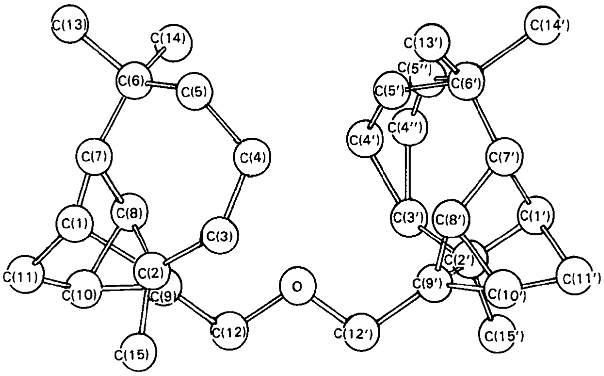

Fig. 2. Perspective view of the molecule.

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Structure of Dichloro(*p*-methoxyphenyl)(8-oxo-7-oxabicyclo[4.3.0]non-5-yl)-tellurium(IV)–Chloroform (1/0·27)

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Abstract. $[\mathrm{TeCl_2(CH_3OC_6H_4)(C_8H_{11}O_2)}].0{\cdot}27\mathrm{CHCl_3}$, $M_r = 447{\cdot}0$, hexagonal, $P6_5$, $a = 15{\cdot}061\ (3)$, $c = 14{\cdot}409\ (3)$ Å, $Z = 6$, $V = 2831\ (1)$ Å^3, $D_x = 1{\cdot}68$ Mg m^{-3}, $\lambda(\mathrm{Mo}\ K\alpha) = 0{\cdot}71069$ Å, $\mu = 1{\cdot}99$ mm^{-1}, $F(000) = 1402$, $T = 296\ (1)$ K, final $R = 0{\cdot}0471$ for 2493 observed reflections. The central Te atom is bonded to the *p*-methoxyphenyl group, to the cyclohexane moiety of the *cis*-fused γ-butyrolactone and to the two Cl atoms in a pseudo-trigonal-bipyramidal arrangement with axial halogens and one equatorial position vacant. Bond lengths are Te—Cl(1) = 2·525 (4), Te—Cl(2) = 2·499 (3), Te—C(ph) = 2·111 (8) and Te—C(lactone) = 2·184 (9) Å. If an intermolecular, secondary Te···O bond of 3·29 Å, *trans* to Te—C(lactone), is taken into consideration, the structure may be regarded as pseudo-octahedral. This weak bond ties the molecules together in spirals around the sixfold screw axes. Inside the resulting tunnels, disordered $\mathrm{CHCl_3}$ molecules are trapped.

Introduction. The tellurium compound (I) is one of several aryltellurium dichloride lactones made by the

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