been described at 293 K. In previous papers, we have reported the structures of trimethyloxosulfonium iodide, bromide, chloride, nitrate and chromate (Jannin, Puget, de Brauer & Perret, 1991 *a,b,c*; de Brauer, Jannin, Puget & Perret, 1991).

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Structures of (2*S*,3*R*)-*exo*-1,7,7-Trimethyl-3-propylsulfonylbicyclo[2.2.1]heptan-2-ol (I) and (2*S*,3*R*)-*exo*-1,3,7,7-Tetramethyl-3-propylsulfonylbicyclo[2.2.1]heptan-2-ol (II)

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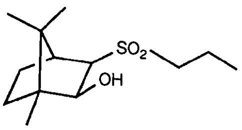
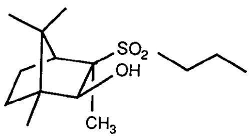
Abstract. (I), $C_{13}H_{24}O_3S$, M_r = 260·4, orthorhombic, $P2_12_12_1$, a = 11·737 (2), b = 21·244 (4), c = 22·932 (3) Å, V = 5717·9 (1·4) Å^3, Z = 16, D_x = 1·21 g cm^{-3}, Cu $K\alpha$, λ = 1·54178 Å, μ = 19·23 cm^{-1}, $F(000)$ = 2272, T = 291 K, R = 0·063 for 3245 observed reflections. (II), $C_{14}H_{26}O_3S$, M_r = 274·4, orthorhombic, $P2_12_12_1$, a = 8·651 (2), b = 11·920 (4), c = 14·256 (3) Å, V = 1470·1 (6) Å^3, Z = 4, D_x = 1·24 g cm^{-3}, Cu $K\alpha$, λ = 1·54178 Å, μ = 18·95 cm^{-1}, $F(000)$ = 600, T = 291K, R = 0·056 for 1491 observed reflections. Regio- and stereocontrolled three-carbon annulation is a valuable tool for the synthesis of natural products. In a previous communication [De Lombaert, Nemery, Roekens, Carretero, Kimmel & Ghosez (1986). *Tetrahedron Lett.* **27**, 5099-5102] we reported a novel two-step sequence yielding highly functionalized, fused cyclopentanes. The title compounds are being used to develop an enantioselective version, and were synthesized efficiently in optically pure, crystalline form from (+)-camphor. X-ray crystallography confirmed the stereochemistries shown for (I) and (II).

Experimental. D_m not measured. Crystal sizes: (I) 0·3 × 0·4 × 0·25 mm; (II) 0·3 × 0·3 × 0·2 mm. Lattice parameters refined using (I) 20, (II) 30 reflections in the range (I) $9 \leq 2\theta \leq 50°$, (II) $12 \leq 2\theta \leq 49°$. Huber four-circle diffractometer, graphite-monochromatized Cu $K\alpha$ radiation. For (I) 5735 independent reflections with $\sin\theta/\lambda \leq 0·69$ Å^{-1}; $0 \leq h \leq 14$, $0 \leq k \leq 25$, $0 \leq l \leq 27$, 3245 with $I \geq 2·5\sigma(I)$. For (II) 1567 independent reflections with $\sin\theta/\lambda \leq 0·69$ Å^{-1}; $0 \leq h \leq 10$, $0 \leq k \leq 14$, $0 \leq l \leq 17$, 1491 with $I \geq 2·5\sigma(I)$. Standard reflections (I): 303; (II): 02$\bar{7}$ checked every 50 reflections, no significant deviation.



(I)



(II)

Structure solved by direct methods using *SHELXS*86 (Sheldrick, 1985). All H atoms in computed positions, except those of the hydroxyl groups [for (I) from difference Fourier synthesis, not found for (II)]. Anisotropic least-squares refinement (*SHELX*76; Sheldrick, 1976) using F; H atoms isotropic with common refined temperature factor [U = 0·0940 (I), 0·0926 (II) Å^2]. $w = 1/(\sigma^2 + gF^2)$; g =

0108-2701/91/102234-03$03.00

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* (Å^2)

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

	x	y	z	B_{eq}
Compound (I)				
C1*A*	9858 (8)	5316 (5)	1229 (4)	4·78 (18)
C2*A*	9601 (7)	5222 (4)	1890 (4)	4·33 (17)
C3*A*	8337 (7)	5457 (4)	1923 (3)	3·32 (13)
C4*A*	8008 (8)	5552 (4)	1266 (4)	4·45 (17)
C5*A*	8533 (8)	6186 (4)	1119 (4)	4·62 (17)
C6*A*	9824 (8)	6031 (5)	1130 (5)	5·43 (20)
C7*A*	8776 (8)	5086 (5)	924 (4)	4·66 (17)
C8*A*	11006 (8)	5023 (5)	1048 (5)	5·98 (21)
C9*A*	8464 (12)	4406 (4)	1005 (5)	6·27 (23)
C10*A*	8776 (11)	5210 (6)	267 (4)	6·76 (25)
S11*A*	7312 (2)	5045 (1)	2348 (1)	3·85 (3)
O12*A*	7053 (7)	4431 (3)	2143 (3)	5·85 (14)
O13*A*	6338 (5)	5464 (3)	2400 (3)	5·19 (12)
C14*A*	7963 (8)	5009 (5)	3047 (4)	5·21 (19)
C15*A*	7175 (13)	4673 (11)	3478 (5)	10·99 (41)
C16*A*	7643 (20)	4366 (8)	3924 (8)	13·74 (59)
O17*A*	9713 (5)	4587 (3)	2097 (3)	5·24 (13)
C1*B*	5141 (9)	6913 (4)	811 (4)	4·45 (17)
C2*B*	5364 (8)	6734 (5)	1450 (4)	4·80 (18)
C3*B*	4796 (7)	7312 (4)	1770 (4)	3·91 (15)
C4*B*	4157 (9)	7676 (4)	1278 (4)	4·95 (18)
C5*B*	5137 (11)	8052 (5)	981 (5)	6·26 (23)
C6*B*	5842 (10)	7527 (6)	704 (5)	6·44 (23)
C7*B*	3891 (8)	7171 (5)	813 (4)	4·61 (17)
C8*B*	5412 (11)	6394 (6)	385 (4)	6·52 (23)
C9*B*	2973 (9)	6699 (5)	981 (4)	5·59 (20)
C10*B*	3543 (11)	7463 (6)	221 (5)	6·81 (25)
S11*B*	4034 (2)	7161 (1)	2425 (1)	3·66 (3)
O12*B*	4767 (5)	6784 (3)	2794 (3)	4·75 (12)
O13*B*	2909 (5)	6911 (3)	2326 (3)	4·54 (11)
C14*B*	3900 (10)	7935 (4)	2726 (4)	5·29 (19)
C15*B*	3450 (10)	7903 (5)	3363 (5)	5·99 (22)
C16*B*	2156 (10)	7863 (6)	3398 (5)	6·09 (22)
O17*B*	4888 (6)	6146 (3)	1608 (3)	5·46 (14)
C1*C*	2824 (8)	5556 (4)	3756 (4)	4·49 (17)
C2*C*	3261 (8)	5455 (4)	3117 (4)	4·44 (17)
C3*C*	3514 (7)	4735 (4)	3143 (4)	3·61 (14)
C4*C*	2994 (8)	4503 (4)	3720 (4)	4·17 (16)
C5*C*	3903 (9)	4696 (5)	4169 (5)	5·16 (19)
C6*C*	3866 (10)	5419 (5)	4150 (4)	5·47 (20)
C7*C*	2045 (8)	4979 (4)	3850 (4)	4·70 (17)
C8*C*	2298 (11)	6215 (4)	3836 (5)	6·18 (22)
C9*C*	1002 (8)	4927 (5)	3454 (5)	5·43 (19)
C10*C*	1618 (10)	4929 (6)	4487 (4)	6·10 (22)
S11*C*	3196 (2)	4273 (1)	2504 (1)	3·88 (3)
O12*C*	2011 (5)	4272 (3)	2338 (3)	5·16 (12)
O13*C*	3685 (6)	3664 (3)	2618 (3)	5·45 (13)
C14*C*	3985 (9)	4636 (5)	1931 (4)	4·80 (17)
C15*C*	4012 (10)	4258 (5)	1395 (4)	5·98 (21)
C16*C*	4734 (13)	4548 (6)	930 (4)	7·21 (26)
O17*C*	2518 (6)	5628 (3)	2663 (3)	5·42 (13)
C1*D*	2882 (8)	2108 (5)	708 (4)	4·98 (18)
C2*D*	3071 (8)	2098 (4)	1366 (4)	4·38 (16)
C3*D*	2038 (7)	2470 (4)	1593 (4)	4·29 (16)
C4*D*	1250 (7)	2530 (4)	1044 (4)	4·60 (17)
C5*D*	1793 (9)	3084 (5)	723 (4)	5·31 (19)
C6*D*	2982 (9)	2817 (4)	532 (4)	5·20 (19)
C7*D*	1568 (9)	1974 (5)	645 (5)	5·16 (19)
C8*D*	3699 (11)	1676 (5)	362 (6)	6·82 (26)
C9*D*	1156 (10)	1332 (5)	848 (6)	6·84 (25)
C10*D*	1141 (12)	2041 (7)	11 (5)	7·93 (29)
S11*D*	1426 (2)	2180 (1)	2251 (1)	5·69 (5)
O12*D*	2323 (8)	2088 (4)	2667 (3)	7·90 (19)
O13*D*	657 (7)	1660 (4)	2170 (4)	7·31 (17)
C14*D*	604 (10)	2841 (5)	2488 (5)	6·38 (22)
C15*D*	−70 (16)	2701 (9)	3031 (7)	10·77 (45)
C16*D*	−207 (29)	3018 (11)	3411 (12)	29·86 (143)
O17*D*	3174 (6)	1466 (3)	1583 (3)	5·85 (14)
Compound (II)				
C1	2935 (3)	−8422 (3)	−8562 (2)	3·24 (5)
C2	1419 (4)	−8036 (2)	−8069 (2)	2·87 (4)
C3	327 (3)	−9074 (2)	−8200 (2)	2·40 (4)
C4	1453 (4)	−9975 (2)	−8615 (2)	2·87 (5)
C5	1630 (4)	−9683 (3)	−9666 (2)	3·77 (5)
C6	2509 (5)	−8536 (3)	−9616 (2)	3·93 (6)
C7	3095 (4)	−9642 (3)	−8252 (2)	3·31 (5)
C8	4269 (5)	−7607 (4)	−8394 (3)	5·11 (7)
C9	3434 (4)	−9818 (4)	−7202 (2)	4·29 (6)
C10	4403 (4)	−10289 (4)	−8765 (3)	5·15 (7)

Table 1 (*cont.*)

	x	y	z	B_{eq}
S11	−634 (1)	−9677 (1)	−7170 (1)	3·02 (1)
O12	396 (3)	−10217 (2)	−6532 (2)	4·60 (5)
O13	−1830 (3)	−10397 (2)	−7557 (2)	4·15 (4)
C14	−1608 (6)	−8569 (3)	−6556 (3)	4·60 (7)
C15	−2481 (5)	−9067 (4)	−5732 (3)	4·43 (6)
C16	−3279 (9)	−8199 (5)	−5153 (4)	7·49 (11)
O17	1602 (3)	−7710 (2)	−7113 (2)	3·81 (4)
C18	−1037 (4)	−8739 (3)	−8837 (2)	3·74 (5)

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

	(I)*	(II)		(I)*	(II)
C2—C1	1·547 (13)	1·557 (4)	C6—C1	1·552 (14)	1·553 (4)
C7—C1	1·554 (13)	1·527 (5)	C8—C1	1·534 (14)	1·528 (5)
C3—C2	1·560 (12)	1·568 (4)	O17—C2	1·423 (11)	1·425 (3)
C4—C3	1·559 (12)	1·565 (4)	S11—C3	1·785 (8)	1·833 (3)
C18—C3	—	1·542 (4)	C5—C4	1·536 (14)	1·545 (4)
C7—C4	1·542 (13)	1·563 (4)	C6—C5	1·547 (15)	1·566 (5)
C9—C7	1·517 (14)	1·539 (4)	C10—C7	1·543 (14)	1·553 (4)
O12—S11	1·436 (7)	1·427 (2)	O13—S11	1·443 (7)	1·453 (3)
C14—S11	1·785 (10)	1·795 (4)	C15—C14	1·512 (14)†	1·518 (5)
C16—C15	1508 (17)†	1·493 (6)			
	(I)*	(II)		(I)*	(II)
C6—C1—C2	105·2 (8)	105·3 (2)	C7—C1—C2	103·5 (7)	103·1 (2)
C7—C1—C6	102·8 (8)	102·6 (3)	C8—C1—C2	112·9 (8)	112·2 (3)
C8—C1—C6	113·4 (8)	112·7 (3)	C8—C1—C7	117·5 (8)	119·5 (3)
C3—C2—C1	100·7 (7)	102·7 (2)	O17—C2—C1	114·2 (7)	114·7 (3)
O17—C2—C3	113·6 (7)	113·3 (2)	C4—C3—C2	104·3 (7)	102·2 (2)
S11—C3—C2	117·8 (6)	119·1 (2)	S11—C3—C4	116·6 (6)	108·4 (2)
C18—C3—C2	—	109·0 (2)	C18—C3—C4	—	115·5 (2)
C18—C3—S11	—	103·1 (2)	C5—C4—C3	102·4 (7)	105·9 (2)
C7—C4—C3	104·9 (7)	105·4 (2)	C7—C4—C5	101·9 (7)	100·0 (3)
C6—C5—C4	102·5 (7)	101·6 (2)	C5—C6—C1	103·5 (8)	103·6 (3)
C4—C7—C1	93·1 (7)	93·6 (2)	C9—C7—C1	116·5 (8)	115·3 (3)
C9—C7—C4	114·9 (8)	117·5 (3)	C10—C7—C1	112·7 (8)	113·8 (3)
C10—C7—C4	112·8 (8)	112·4 (3)	C10—C7—C9	106·7 (8)	104·6 (3)
O12—S11—C3	111·1 (4)	113·9 (1)	O13—S11—C3	109·6 (4)	104·5 (1)
O13—S11—O12	115·6 (4)	114·9 (2)	C14—S11—C3	102·8 (4)	108·4 (1)
C14—S11—O12	108·9 (5)	108·3 (2)	C14—S11—O13	107·9 (4)	106·6 (2)
C15—C14—S11	111·8 (7)†	108·9 (2)	C16—C15—C14	112·8 (9)†	112·7 (4)

* Mean values.

† Except molecules *A* and *D*.

0·02120 for (I) and 0·06482 for (II). (I): $R = 0{\cdot}063$, $wR = 0{\cdot}072$, $S = 0{\cdot}59$ for 3245 observed reflections; (II): $R = 0{\cdot}055$, $wR = 0{\cdot}055$, $S = 0{\cdot}33$ for 1491 observed reflections. In molecules (I*A*) and (I*D*) the terminal atoms (C15 and C16) of the propylsulfonyl group present a very high degree of vibration and were not taken into account in the calculations of the max. shift/e.s.d., nor for the mean values of bond distances and angles. Final max. shift/e.s.d. = 0·25 (I), 0·10 (II). Max. and min. heights in final difference Fourier synthesis: (I) 0·43 and −0·33 e Å^{-3}, (II) 0·27 and −0·58 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV).

The atomic parameters are given in Table 1.* Figs. 1 and 2 are stereoscopic views of the molecules, showing the numbering of the atoms (*PLUTO*;

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54008 (32 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

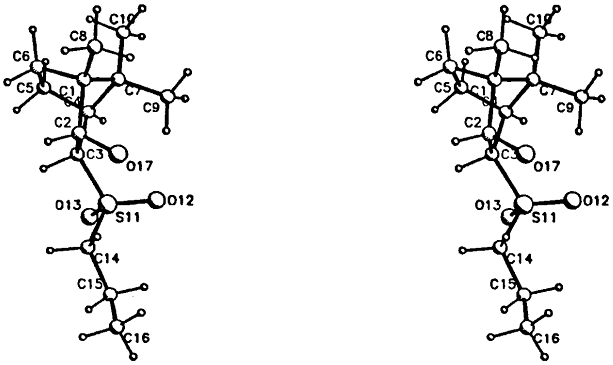

Fig. 1. Stereoscopic view of molecule *A* of compound (I) and atom-numbering scheme.

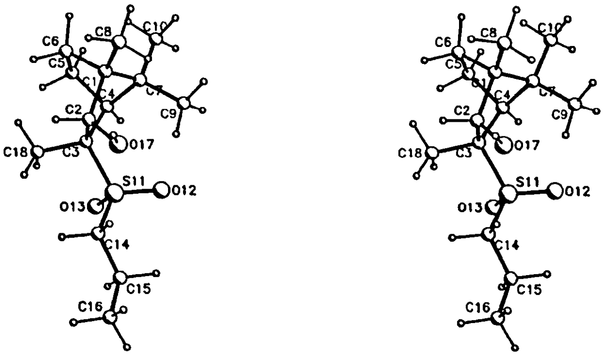

Fig. 2. Stereoscopic view of the molecule of compound (II) and atom-numbering scheme.

Motherwell & Clegg, 1978). Bond distances and angles are given in Table 2.

Related literature. The structure of (1*R*,2*S*,*S*)-3-*exo*-(prop-2-enylsulfinyl)-1,7,7-trimethylbicyclo[2.2.1]-heptan-2-ol (Goodridge, Hambley, Haynes & Ridley, 1988) is closely related to the title compounds.

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Structures of *o*-Aminobenzamide and *p*-Hydroxybenzamide Monohydrate

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Abstract. (I) *o*-Aminobenzamide, $C_7H_8N_2O$, $M_r = 136{\cdot}15$, monoclinic, $P2_1/c$, $a = 14{\cdot}173\,(3)$, $b = 6{\cdot}327\,(2)$, $c = 7{\cdot}803\,(2)$ Å, $\beta = 97{\cdot}98\,(2)°$, $V = 692{\cdot}9\,(3)$ Å^3, $Z = 4$, $D_m = 1{\cdot}31$, $D_x = 1{\cdot}306$ Mg m^{-3}, $\lambda(\text{Cu }K\alpha) = 1{\cdot}5418$ Å, $\mu = 0{\cdot}71$ mm^{-1}, $F(000) = 288$, $T = 295$ K, $R = 0{\cdot}062$ for 1031 unique reflections. (II) *p*-Hydroxybenzamide monohydrate, $C_7H_7NO_2.H_2O$, $M_r = 155{\cdot}14$, monoclinic, $P2_1/c$, $a = 16{\cdot}546\,(3)$, $b = 11{\cdot}798\,(2)$, $c = 3{\cdot}8889\,(7)$ Å, $\beta = 100{\cdot}40\,(2)°$, $V = 746{\cdot}7\,(2)$ Å^3, $Z = 4$, $D_m = 1{\cdot}35$, $D_x = 1{\cdot}381$ Mg m^{-3}, $\lambda(\text{Cu }K\alpha) = 1{\cdot}5418$ Å, $\mu = 0{\cdot}93$ mm^{-1}, $F(000) = 328$, $T = 295$ K, $R = 0{\cdot}040$ for 1024 unique reflections. Dihedral angles between the benzene ring and amide planes are 32·5 (2) for (I) and 14·6 (2)° for (II). In (I) an intramolecular hydrogen bond is formed between the *o*-amino and the amide groups. An intermolecular hydrogen bond is formed between the amide groups related by a 2_1 axis. The *o*-amino group accepts and donates the other intermolecular hydrogen bonds. In (II) a centrosymmetric dimer is formed by hydrogen bonds between the amide groups. The hydroxyl group