In the molecule, there are two separate trapezoids (not precisely planar), one defined by atoms N(1), N(1′), Cl(1), Cl(1′) [dihedral angle 1·7 (5)°] and the other defined by N(2), N(2′), Cl(2), Cl(2′) [dihedral angle 1·6 (8)°]. The two best planes are essentially orthogonal to each other [90·5 (9)°]. The four nitrogen atoms form an elongated tetrahedron, while the four chlorine atoms form a flattened tetrahedron. This is as would be expected since the most crowded positions of the polyhedra are at the corners of the elongated tetrahedron and therefore are more suitable for the smaller nitrogen atoms. The average angle between the crystallographically imposed twofold axis and the uranium–nitrogen bonds is 33·9 [1]° while the average angle between the axis and the uranium–chloride bonds is 106·8 [2]°. These structural values place the $UCl_4(CH_3CN)_4$ structure near that which has been calculated for the minimum repulsion energy of a dodecahedron (Kepert, 1978). The structure of this complex comes very close to being an ideal dodecahedron.

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Structure of (+)-(7*S*,11*S*)-11-Hydroxy-10-methylbicyclo[5.3.1]dec-1(10)-en-7-ylmethyl (+)-10-Camphorsulfonate,* $C_{23}H_{36}O_5S$

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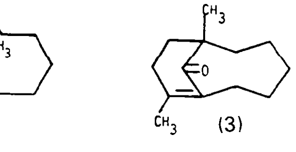
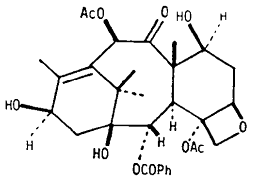
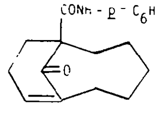
Abstract. $M_r = 424{\cdot}6$, tetragonal, $P4_1$, $a = 9{\cdot}833$ (4), $c = 23{\cdot}46$ (1) Å, $V = 2267$ (1) Å^3, $Z = 4$, $D_x = 1{\cdot}24$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu = 1{\cdot}64$ cm^{-1}, $F(000) = 920$, $T = 291$ K, final $R = 0{\cdot}065$ for 1168 observed reflections. The eight-carbon ring is in a conformation corresponding to that of minimum energy for cyclooctane with an approximate D_2 symmetry. The best planes through it and the condensed cyclohexene form an angle of 101·7 (6)°.

Introduction. In connection with studies on optically active *trans*-cycloalkenes, we recently resolved racemic 7-hydroxymethyl-10-methylbicyclo[5.3.1]dec-1(10)-en-11-ol *via* the (+)-10-camphorsulfonate (1) and converted the highly crystalline (+)-diastereomer to (−)-*trans*-1,2-dimethylcyclodecene (2) by a six-step sequence (Marshall, Konicek & Flynn, 1980). Our assignment of configuration of (+)-(1) and (−)-(2) was based upon the optical rotatory dispersion curve analysis of enone (3),





(4)



(5)

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* 10-Camphorsulfonic acid is 7,7-dimethyl-2-oxobicyclo[2.2.1]hept-1-ylmethanesulfonic acid.

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a degradation product of camphorsulfonate (1). We later found reason to question the applicability of the enone octant rule to bicyclics such as (3) owing to apparent noncoplanarity of the enone system (Marshall & Flynn, 1982; Cameron & Jamieson, 1971). Thus, additional evidence for the configuration of (1) was sought through X-ray structure analysis.

Experimental. Crystals obtained by slow evaporation from methanol solution; fragment of a tetragonal needle 0·3 × 0·2 × 0·2 mm mounted in a glass capillary; Enraf–Nonius CAD-4 diffractometer, graphite monochromator; 25 general reflections used for orientation matrix and unit-cell measurements, $00l$ with $l \neq 4n$ absent, space group $P4_1$ selected to match the absolute configuration of camphor, $2\theta_{\max} = 50°$, *hkl* range 0–11, 0–11, 0–26, 3 standard reflections monitored every 3 h, no decay, 2321 reflections measured in ω–2θ scan mode, 1248 with $I > 3\sigma(I)$ observed, 1168 symmetry independent; structure solved with *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980); about half the structure was found in the best *E* map, the rest located in subsequent Fourier maps; positions of H atoms calculated except for methyl and hydroxyl groups which could not be located; all other atoms refined using anisotropic thermal-motion parameters, $\sum w(|F_o| - |F_c|)^2$ minimized, $wR = 0{\cdot}077$, $S = 1{\cdot}59$, $w = [\sigma^2(F) + 0{\cdot}0007F^2]^{-1}$, last cycle max. Δ/σ 0·08, av. 0·02; final difference Fourier map: max. = 0·33, min. = −0·41 e Å^{-3}; atomic scattering factors and f', f'' values from *International Tables for X-ray Crystallography* (1974); data reduction and structure solution carried out with the *Enraf–Nonius Structure Determination Package* (Frenz, 1982), refinement with *SHELX*76 (Sheldrick, 1976).

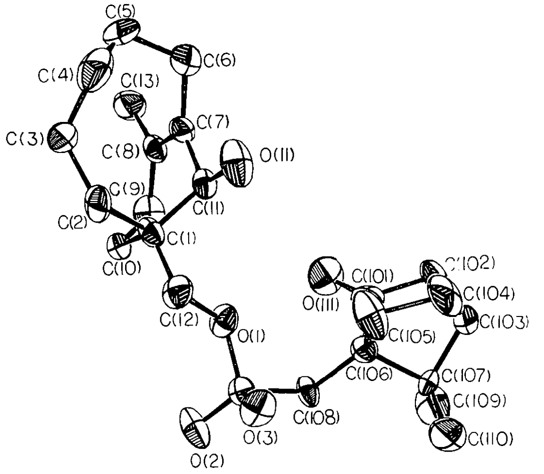

Fig. 1. An *ORTEP* (Johnson, 1970) drawing of the title molecule with the atom numbering scheme. The thermal ellipsoids are drawn at the 50% probability level.

Table 1. *Positional parameters and equivalent isotropic thermal parameters 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)
S(1)	0·3655 (2)	0·2595 (2)	0·5000	2·81
O(1)	0·3491 (7)	0·2666 (7)	0·5651 (3)	3·41
O(2)	0·2468 (7)	0·3143 (7)	0·4738 (4)	4·92
O(3)	0·4055 (8)	0·1261 (7)	0·4822 (4)	4·02
C(12)	0·2739 (11)	0·1543 (11)	0·5932 (5)	3·63
C(1)	0·2313 (10)	0·2005 (10)	0·6502 (5)	2·97
C(2)	0·1481 (11)	0·0786 (10)	0·6751 (5)	3·71
C(3)	0·0949 (11)	0·0829 (11)	0·7365 (5)	3·93
C(4)	0·1899 (13)	0·0383 (13)	0·7840 (7)	5·47
C(5)	0·2399 (12)	0·1448 (15)	0·8231 (5)	4·87
C(6)	0·3560 (12)	0·2330 (13)	0·7979 (5)	4·76
C(7)	0·3187 (10)	0·2975 (11)	0·7425 (5)	3·20
C(8)	0·2402 (9)	0·4119 (10)	0·7383 (5)	2·90
C(9)	0·2001 (11)	0·4520 (11)	0·6815 (6)	3·86
C(10)	0·1399 (10)	0·3277 (10)	0·6489 (5)	3·25
C(11)	0·3556 (10)	0·2328 (9)	0·6865 (5)	2·79
O(11)	0·4390 (8)	0·1124 (7)	0·6921 (4)	4·96
C(13)	0·1879 (11)	0·4938 (13)	0·7894 (6)	4·40
C(108)	0·5001 (8)	0·3750 (9)	0·4914 (5)	2·96
C(106)	0·6365 (9)	0·3478 (10)	0·5214 (4)	2·99
C(101)	0·6606 (12)	0·4439 (11)	0·5703 (5)	3·51
O(111)	0·5741 (8)	0·5003 (8)	0·5984 (4)	5·42
C(102)	0·8141 (11)	0·4446 (13)	0·5804 (6)	4·75
C(103)	0·8635 (10)	0·3507 (16)	0·5339 (5)	5·33
C(104)	0·8210 (12)	0·2065 (14)	0·5550 (6)	5·85
C(105)	0·6620 (11)	0·2033 (10)	0·5482 (6)	5·40
C(107)	0·7647 (10)	0·3790 (15)	0·4828 (5)	5·18
C(109)	0·7848 (13)	0·2790 (18)	0·4341 (6)	7·62
C(110)	0·7688 (13)	0·5286 (14)	0·4611 (7)	6·27

Table 2. *Bond lengths* (Å) *with e.s.d.'s in parentheses*

S(1)–O(1)	1·537 (7)	S(1)–O(2)	1·425 (8)
S(1)–O(3)	1·432 (7)	S(1)–C(108)	1·756 (9)
O(1)–C(12)	1·48 (1)	C(12)–C(1)	1·47 (2)
C(1)–C(2)	1·56 (2)	C(1)–C(10)	1·54 (1)
C(1)–C(11)	1·52 (2)	C(2)–C(3)	1·53 (2)
C(3)–C(4)	1·52 (2)	C(4)–C(5)	1·48 (2)
C(5)–C(6)	1·55 (2)	C(6)–C(7)	1·49 (2)
C(7)–C(8)	1·37 (1)	C(7)–C(11)	1·50 (2)
C(8)–C(9)	1·44 (2)	C(8)–C(13)	1·53 (2)
C(9)–C(10)	1·56 (2)	C(11)–O(11)	1·45 (1)
C(108)–C(106)	1·54 (1)	C(106)–C(101)	1·51 (2)
C(106)–C(105)	1·57 (1)	C(106)–C(107)	1·58 (1)
C(101)–O(111)	1·21 (1)	C(101)–C(102)	1·53 (2)
C(102)–C(103)	1·51 (2)	C(103)–C(104)	1·56 (2)
C(103)–C(107)	1·57 (2)	C(104)–C(105)	1·57 (2)
C(107)–C(109)	1·52 (2)	C(107)–C(110)	1·56 (2)

Discussion. The non-H atomic coordinates are listed in Table 1.* The bond distances given in Table 2 and angles (deposited) are normal and show no unexpected values. An *ORTEP* (Johnson, 1970) drawing of the molecule is shown in Fig. 1. The enantiomorph conforming to the known absolute configuration of the camphor moiety was selected. It shows the configuration of (+)-(1) to be 7*S*, in contradiction to earlier conclusions (Marshall, Konicek & Flynn, 1980).

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* Lists of structure factors, anisotropic thermal parameters, coordinates of H atoms, least-squares planes and bond angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39108 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Comparison of selected torsion angles in* (1), (4) *and* (5) (°)

E.s.d.'s for (1) are given in parentheses; data for (4) and (5) are taken from the Cambridge Crystallographic Data File, May 1982 update (Allen *et al.*, 1979). *R* factors are 0·065, 0·091 and 0·092, respectively.

	(1)	(4)	(5)
C(11)C(1)C(2)C(3)	56·2 (1·3)	63·7	+41·7
C(1)C(2)C(3)C(4)	−85·0 (1·3)	−103·0	−94·4
C(2)C(3)C(4)C(5)	110·5 (1·3)	70·0	−94·3
C(3)C(4)C(5)C(6)	−77·7 (1·4)	29·7	−89·9
C(4)C(5)C(6)C(7)	55·3 (1·4)	−50·0	+44·0
C(5)C(6)C(7)C(11)	−95·3 (1·2)	−50·7	+50·1
C(6)C(7)C(11)C(1)	120·2 (1·1)	121·6	+137·1
C(7)C(11)C(1)C(2)	−71·7 (1·1)	−73·2	+58·6
C(10)C(1)C(11)C(7)	48·2 (1·1)	52·8	−59·3
C(1)C(11)C(7)C(8)	−54·8 (1·2)	−51·6	+34·5
C(11)C(7)C(8)C(9)	2·0 (1·4)	5·4	−0·3
C(7)C(8)C(9)C(10)	49·4 (1·3)	37·9	−3·6
C(8)C(9)C(10)C(1)	−51·3 (1·2)	−32·2	−24·9
C(9)C(10)C(1)C(11)	2·6 (1·2)	−13·6	+55·3

The molecule consists of the camphorsulfonate moiety and the bicyclo[5.3.1]decene moiety which, except for the covalent link, do not interact. The shortest non-bonded contact is 3·11 (1) Å between O(1) and C(106). The cyclooctane ring is in a conformation similar to that of the minimum-energy form of symmetry D_2 [C(1), C(2)] (Bucourt, 1974). The average square deviation of the torsion angles from theoretical values is 9·2°. The asymmetry parameter (Duax, Weeks & Rohrer, 1976) for D_2 operators is 14·1°. The condensed cyclohexane adopts a boat conformation with the mirror plane through C(9), with Cremer–Pople (1975) puckering parameters $q_2 = 0{\cdot}700$, $q_3 = 0{\cdot}004$ Å, $\varphi_2 = 177°$, $Q = 0{\cdot}700$ Å and $\theta = 90°$. The best planes through these two rings are close to orthogonal with an angle of 101·7 (6)° between them.

Comparison with two other bicyclo[5.3.1]dec-1(10)-enes which were found in the Cambridge Crystallographic Data File, May 1982 update (Allen *et al.*, 1979), revealed significant conformational differences, presented in Table 3. In the highly substituted analog (4) (Castellano & Hodder, 1973) cyclooctane adopts a boat–chair conformation with the twofold axis passing through C(5). In the related (5) (Cameron & Jamieson, 1971) the cyclooctane has a chair conformation with D_2 symmetry [C(7), C(1)] while the cyclohexene ring has the half-boat conformation with the mirror plane through C(1). The observed differences indicate that in this condensed system the cyclooctane also has conformational flexibility. The differences in the conformation of the cyclohexene rings between (4), (1) and (5) can probably be attributed to conjugation in the enone system of (5).

The packing diagram is shown in Fig. 2. Intermolecular contacts are normal. The short distance of 2·86 (1) Å between O(11) and O(2) of a neighboring molecule suggests a hydrogen bond.

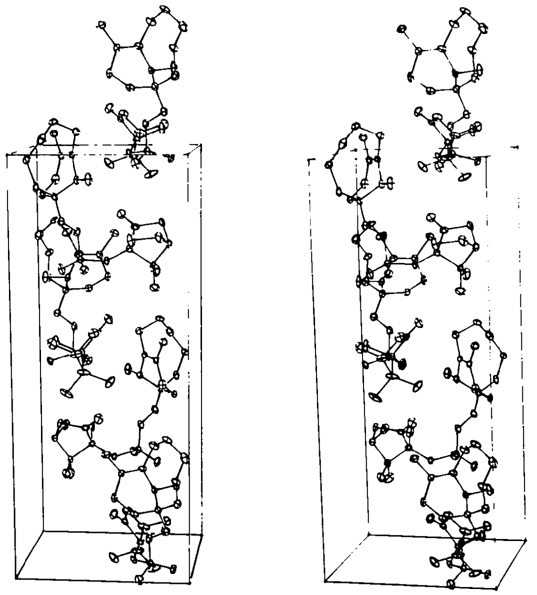

Fig. 2. Molecular packing.

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