dihydro-2′,3′-O-isopropylidene-3-methyluridine (Gautham, Ramakrishnan, Seshadri & Viswamitra, 1982).

Dioxolane ring: In molecule A, C(7) and O(3′) are displaced by 0·377 (3) and −0·154 (2) Å respectively whereas in molecule B they are displaced by −0·187 (3) and 0·248 (2) Å respectively from the best three-atom planes of the dioxolane rings. Thus the dioxolane ring assumes a twist conformation in both molecules.

Molecular packing: The crystal structure is stabilized by two hydrogen bonds, N(3)—H⋯O(4) [2·832 (3)Å and 174·36°] and N(3)—H⋯O(3′) [2·893 (2) Å and 175·66°]. This seems to be the first isopropylidene nucleoside to show such an H bond involving the isopropylidene group. These two hydrogen bonds are shown in Fig. 2.

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Ionic Compounds from the 2:1 Addition of Tropolone* and 4-Hydroxy-3-methoxybenzaldehyde to 1,7,10,16-Tetraoxa-4,13-diazacyclooctadecane

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Abstract. $C_{12}H_{28}N_2O_4^{2+}.2C_7H_5O_2^-$ (1), $M_r = 506·59$, triclinic, $P\bar{1}$, $a = 7·513$ (3), $b = 9·165$ (5), $c = 10·458$ (5) Å, $\alpha = 81·42$ (4), $\beta = 72·75$ (3), $\gamma = 78·47$ (4)°, $V = 670·6$ (6) Å³, $Z = 1$, $D_x = 1·25$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0·71073$ Å, $\mu = 0·87$ cm⁻¹, $F(000) = 272$, $T = 300$ K, $R = 0·047$ for 970 unique observed reflections. $C_{12}H_{28}N_2O_4^{2+}.2C_8H_7O_3^-$ (2), $M_r = 566·65$, triclinic, $P\bar{1}$, $a = 7·400$ (2), $b = 9·111$ (3), $c = 10·704$ (3) Å, $\alpha = 94·21$ (2), $\beta = 93·38$ (2), $\gamma = 94·91$ (2)°, $V = 715·6$ (3) Å³, $Z = 1$, $D_x = 1·31$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0·71073$ Å, $\mu = 0·93$ cm⁻¹, $F(000) = 304$, $T = 300$ K, $R = 0·0437$ for 1542 unique observed reflections. The 'diaza-18-crown-6' dications adopt different conformations in the two structures. The conformer in (1) resembles that of free 18-crown-6; however, the conformer in (2) has not been observed previously. The tropolonate anion is planar and

participates in a bifurcated hydrogen bond. The 4-hydroxy-3-methoxybenzaldehyde anion exhibits a quinoid-like arrangement of double bonds in the phenyl ring.

Introduction. In general, complexes between 1,7,10,16-tetraoxa-4,13-diazacyclooctadecane ('diaza-18-crown-6') and neutral organic molecules containing proton donors are stabilized by hydrogen bonds to the N atoms of the crown molecule. When Me₂SO solutions of tropolone and 'diaza-18-crown-6' (2:1 ratio) are mixed, a yellow solution is formed. This yellow solution arises because of proton transfer from the tropolone (3) to the 'diaza-18-crown-6' resulting in the formation of an ionic compound composed of a 'diaza-18-crown-6' dication and two tropolonate anions. The ¹³C spectrum of pure tropolone contains only four lines which indicates the proton transfer between the two O atoms is fast on the NMR time scale (Weiler, 1972). In the crystal structure of tropolone (Shimanouchi & Sasada,


1973), the proton is fixed and bond alternation is observed. This is a situation where there should be a significant difference between the solid-state ^{13}C spectrum and that in solution. In sodium tropolonate (Shiono, 1961), the proton is removed, the electrons are delocalized and no bond alternation is observed. Although the color of the tropolone:'diaza-18-crown-6' solution differs from that of tropolone, the ^{13}C spectrum shows only small differences in chemical shift. On the NMR time scale, the equivalence of C atoms due to rapid tautomerism is almost the same as the equivalence due to delocalization. More significant chemical shifts are observed in the ^{13}C spectrum of the 'diaza-18-crown-6' moiety. Here, we report the details of the crystal structures of the ionic compounds formed by the addition of tropolone and of 4-hydroxy-3-methoxybenzaldehyde (4) to 'diaza-18-crown-6'.

(3)   (4)
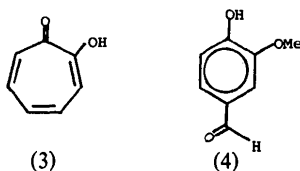
Experimental. All X-ray data were collected on a Nicolet $R3m/\mu$ update of a $P2_1$ diffractometer. Data were collected in the Wyckoff mode (2θ fixed, ω varied) [$3 \le 2\theta \le 45°$ for (1) and $3 \le 2\theta \le 50°$ for (2)] using graphite-monochromated Mo $K\alpha$ radiation. Unit-cell dimensions from a least-squares refinement of 25 centered high-angle reflections ($2\theta \ge 30°$). $\sum w(|F_o| - |F_c|)^2$ was minimized. All computer programs supplied by Nicolet for Desktop 30 Microeclipse and Nova 4/C configuration (Nicolet Instrument Corporation, 1986). Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974).

The 2:1 tropolone adduct (1) was prepared from 65·6 mg (0·25 mmol) 1,7,10,16-tetraoxa-4,13-diaza-cyclooctadecane and 56·1 mg (0·47 mmol) tropolone in 1 ml ethyl acetate under refluxing and stirring for 1 h. After hot filtration the solution was allowed to crystallize at room temperature. The yellow needles were recrystallized from ethyl acetate: yield 103 mg (87%), m.p. 411–414 K. Analysis: calcd for $C_{26}H_{38}N_2O_8$: C 61·64, H 7·56, N 5·53%; found: C 61·96, H 7·60, N 5·69%.

A transparent yellow crystal of dimensions 0·15 × 0·25 × 0·55 mm was used for all X-ray measurements. Monitored reflections 204 and $\bar{1}2\bar{1}$ showed only statistical variations during data collection; Laue symmetry $\bar{1}$, 1164 unique reflections of which $970 \ge 2\cdot5\sigma(I)$ ($0 \le h \le 8$, $-10 \le k \le 10$, $-11 \le l \le 11$); Lorentz–polarization corrections, ψ-scan empirical absorption correction (transmission factors 0·924 to 0·958); direct methods revealed the positions of most

non-H atoms, the remainder located in subsequent difference Fourier maps, block-cascade least-squares refinement; H atoms located in difference Fourier map, H atoms attached to the N atoms were refined, the remaining H atoms allowed to ride on attached atom at fixed distance (one isotropic thermal parameter for all such H atoms); final $R = 0\cdot0470$, $wR = 0\cdot0461$ for 169 parameters and 970 reflections, $R = 0\cdot0519$, $wR = 0\cdot0505$ for all 1164 reflections, $S = 1\cdot351$, $(\Delta/\sigma)_{max} = 0\cdot035$, $(\Delta/\sigma)_{mean} = 0\cdot007$, largest peaks in the final difference Fourier map of $+0\cdot14$ and $-0\cdot15$ e Å^{-3}, $w = [\sigma^2(F_o) + 0\cdot00040F_o^2]^{-1}$. Atomic positional parameters are given in Table 1 while distances, valence angles and selected torsion angles are given in Tables 3, 4 and 5.

The 2:1 4-hydroxy-3-methoxybenzaldehyde adduct (2) was prepared by a procedure analogous to that described above: yield 86 mg (61%), m.p. 379–381 K. Analysis: calcd for $C_{28}H_{42}N_2O_{10}$: C 59·35, H 7·47, N 4·94%; found: C 59·43, H 7·26, N 4·93%.

A transparent yellow crystal of dimensions 0·15 × 0·27 × 0·35 mm was used for all X-ray measurements. Monitored reflections 303 and 032 showed only statistical variations during data collection, Laue symmetry $\bar{1}$, 1877 unique reflections of which $1542 \ge 2\cdot5\sigma(I)$ ($0 \le h \le 7$, $-9 \le k \le 9$, $-11 \le l \le 11$); Lorentz–polarization corrections, ψ-scan empirical absorption correction (transmission factors 0·915 to 0·972); direct methods revealed the positions of most heavy atoms, the remainder were located in subsequent difference Fourier maps; block-cascade least-squares refinement, riding model for all H atoms attached to C (isotropic temperature factor refined for each atom), H atoms attached to N atoms were refined; final $R = 0\cdot0437$, $wR = 0\cdot0440$ for 1542 reflections and 208 parameters, $R = 0\cdot0516$, $wR = 0\cdot0492$ for all 1877 reflections, $(\Delta/\sigma)_{max} = 0\cdot18$, $(\Delta/\sigma)_{mean} = 0\cdot028$, largest peaks in the final difference Fourier map of $+0\cdot18$ and $-0\cdot15$ e Å^{-3}; $w = [\sigma^2(F_o) + 0\cdot00034F_o^2]^{-1}$. Atomic positional parameters are shown in Table 2 while distances, valence angles and selected torsion angles are given in Tables 3, 4 and 5.*

Discussion. The ionic complexes (1) and (2) are shown in Figs. 1 and 2. The two 'diaza-18-crown-6' dications exhibit different conformations. The crown in complex (1) adopts a conformation similar to that of the neutral uncomplexed 18-crown-6 molecule (Knöchel, Kopf, Oehler & Rudolph, 1978) which corresponds to the calculated minimum-energy conformation for neutral 18-crown-6 (Bovill, Chadwick & Sutherland, 1980).

Table 1. *Atomic coordinates* $(\times 10^4)$ *and equivalent isotropic thermal parameters* $(\text{Å}^2 \times 10^3)$ *for compound* (1)

	x	y	z	U_{eq}*
N(1)	−1986 (4)	1972 (2)	9906 (2)	39 (1)
C(2)	−1144 (6)	3300 (3)	9174 (3)	51 (2)
C(3)	914 (5)	2820 (3)	8544 (3)	48 (2)
O(4)	1662 (4)	4056 (2)	7798 (2)	50 (1)
C(5)	3643 (6)	3748 (3)	7260 (3)	50 (2)
C(6)	4434 (6)	5165 (4)	6962 (3)	57 (2)
O(7)	4096 (4)	5757 (2)	8206 (2)	60 (1)
C(8)	4505 (7)	7202 (4)	8056 (3)	62 (2)
C(9)	4054 (5)	7753 (4)	9421 (3)	54 (2)
C(10)	−2151 (6)	194 (3)	7043 (3)	45 (2)
O(10)	−2783 (4)	1348 (2)	7646 (2)	69 (1)
C(11)	−871 (5)	−974 (3)	7611 (3)	39 (2)
O(11)	−431 (4)	−624 (2)	8596 (2)	56 (1)
C(12)	−208 (6)	−2397 (3)	7166 (3)	45 (2)
C(13)	−352 (6)	−3019 (3)	6082 (3)	51 (2)
C(14)	−1226 (6)	−2414 (3)	5106 (3)	48 (2)
C(15)	−2257 (5)	−997 (3)	5021 (3)	47 (2)
C(16)	−2668 (6)	110 (3)	5854 (3)	47 (2)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 2. *Atomic coordinates* $(\times 10^4)$ *and equivalent isotropic thermal parameters* $(\text{Å}^2 \times 10^3)$ *for compound* (2)

	x	y	z	U_{eq}*
N(1)	5873 (3)	−1600 (2)	836 (2)	38 (1)
C(2)	7904 (3)	−1499 (3)	1036 (2)	51 (1)
C(3)	8660 (4)	−158 (3)	1816 (2)	57 (1)
O(4)	8555 (2)	1091 (2)	1105 (1)	48 (1)
C(5)	8731 (4)	2449 (3)	1866 (2)	52 (1)
C(6)	8794 (3)	3711 (3)	1052 (2)	51 (1)
O(7)	7121 (2)	3786 (2)	334 (1)	52 (1)
C(8)	7029 (3)	3101 (3)	−910 (2)	48 (1)
C(9)	5081 (3)	2890 (2)	−1371 (2)	44 (1)
C(10)	3243 (3)	3985 (3)	3639 (2)	50 (1)
C(11)	3843 (3)	3224 (2)	2610 (2)	48 (1)
C(12)	3835 (3)	1675 (2)	2528 (2)	37 (1)
O(12)	4368 (2)	934 (2)	1559 (1)	47 (1)
C(13)	3203 (3)	940 (2)	3580 (2)	36 (1)
O(13)	3276 (2)	−563 (2)	3453 (1)	52 (1)
C(14)	2600 (3)	1716 (2)	4592 (2)	41 (1)
C(15)	2606 (3)	3254 (2)	4640 (2)	43 (1)
C(16)	1943 (4)	4075 (3)	5702 (2)	58 (1)
O(16)	1303 (3)	3558 (2)	6612 (2)	77 (1)
C(17)	2587 (4)	−1389 (3)	4432 (3)	62 (1)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

The conformer can be described as an approximate parallelogram with an internal angle of 62·1 (7)°. The crown in complex (2) does not correspond to any of the calculated lower energy conformers for 18-crown-6 and differs from the conformations observed for other 18-crown-6 and 'diaza-18-crown-6' adducts (Watson, Galloy, Grossie, Vögtle & Müller, 1984; Vögtle, Müller & Watson, 1984). The positive charges on the N atoms alter significantly the dipolar repulsions within the macrocycle and result in alternate low-energy conformers. The 'diaza-crown' in (2) can be described as

Table 3. *Bond lengths* (Å) *and hydrogen-bond distances* (Å) *and angles* (°)

	(1)		(2)
N(1)−C(2)	1·486 (4)		1·500 (3)
N(1)−C(9)	1·493 (5)		1·488 (3)
C(2)−C(3)	1·495 (5)		1·477 (3)
C(3)−O(4)	1·391 (3)		1·419 (3)
O(4)−C(5)	1·415 (5)		1·423 (3)
C(5)−C(6)	1·487 (5)		1·492 (3)
C(6)−O(7)	1·418 (4)		1·428 (3)
O(7)−C(8)	1·393 (4)		1·423 (3)
C(8)−C(9)	1·505 (5)		1·488 (3)
C(10)−O(10)	1·248 (3)	C(10)−C(11)	1·375 (3)
C(10)−C(11)	1·472 (5)	C(10)−C(15)	1·387 (3)
C(10)−C(16)	1·428 (5)	C(11)−C(12)	1·407 (3)
C(11)−O(11)	1·273 (4)	C(12)−O(12)	1·295 (3)
C(11)−C(12)	1·394 (4)	C(12)−C(13)	1·434 (3)
C(12)−C(13)	1·384 (5)	C(13)−O(13)	1·372 (2)
C(13)−C(14)	1·368 (5)	C(13)−C(14)	1·368 (3)
C(14)−C(15)	1·377 (4)	O(13)−C(17)	1·428 (3)
C(15)−C(16)	1·364 (4)	C(14)−C(15)	1·399 (3)
		C(15)−C(16)	1·448 (3)
		C(16)−O(16)	1·215 (3)
H(1B)···O(10)	2·04 (4)	H(1B^{ii})···O(12)	1·76 (2)
N(1)···O(10)	2·770 (4)	N(1^{ii})···O(12)	2·677 (3)
N(1)−H(1B)···O(10)	133 (3)	N(1^{ii})−H(1B^{ii})···O(12)	156 (2)
H(1B)···O(11)	1·96 (4)	H(1A)···O(12)	1·80 (2)
N(1)···O(11)	2·792 (3)	N(1)···O(12)	2·729 (3)
N(1)−H(1B)···O(11)	146 (3)	N(1)−H(1A)···O(12)	166 (2)
H(1A^i)···O(11)	1·68 (4)		
N(1^i)···O(11)	2·737 (4)		
N(1^i)−H(1A^i)···O(11)	174 (3)		

Symmetry code: (i) $-x, -y, 2-z$; (ii) $-x, -y, -z$.

Table 4. *Bond angles* (°)

	(1)		(2)
C(2)−N(1)−C(9A)	116·3 (2)		114·3 (2)
N(1)−C(2)−C(3)	109·1 (2)		113·0 (2)
C(2)−C(3)−O(4)	108·5 (2)		109·3 (2)
C(3)−O(4)−C(5)	112·8 (2)		112·8 (2)
O(4)−C(5)−C(6)	110·0 (2)		109·7 (2)
C(5)−C(6)−O(7)	107·2 (2)		112·7 (2)
C(6)−O(7)−C(8)	113·1 (2)		115·3 (2)
O(7)−C(8)−C(9)	109·4 (2)		107·6 (2)
C(8)−C(9)−N(1A)	112·5 (4)		111·1 (2)
O(10)−C(10)−C(11)	116·4 (3)	C(11)−C(10)−C(15)	121·2 (2)
O(10)−C(10)−C(16)	118·8 (3)	C(10)−C(11)−C(12)	121·5 (2)
C(11)−C(10)−C(16)	124·9 (2)	C(11)−C(12)−O(12)	122·5 (2)
C(10)−C(11)−O(11)	115·4 (2)	C(11)−C(12)−C(13)	116·6 (2)
C(10)−C(11)−C(12)	124·1 (3)	O(12)−C(12)−C(13)	120·9 (2)
O(11)−C(11)−C(12)	120·5 (3)	C(12)−C(13)−O(13)	113·6 (2)
C(11)−C(12)−C(13)	132·0 (3)	C(12)−C(13)−C(14)	121·1 (2)
C(12)−C(13)−C(14)	130·5 (3)	O(13)−C(13)−C(14)	125·3 (2)
C(13)−C(14)−C(15)	126·2 (3)	C(13)−O(13)−C(17)	117·2 (2)
C(14)−C(15)−C(16)	129·7 (3)	C(13)−C(14)−C(15)	121·0 (2)
C(10)−C(16)−C(15)	132·0 (3)	C(10)−C(15)−C(14)	118·7 (2)
		C(10)−C(15)−C(16)	120·3 (2)
		C(14)−C(15)−C(16)	121·0 (2)
		C(15)−C(16)−O(16)	126·4 (2)

having an approximate chair conformation. There is a planar central section composed of C(2)N(1)C(9')-C(8') and symmetry-related atoms. The ends of the crown are folded in opposite directions (*anti*) with

Table 5. *Selected torsion angles (°) in the 'diaza-18-crown-6' dications*

	(1)	(2)
N(1)—C(2)—C(3)—O(4)	176·6 (3)	−73·0 (3)
C(2)—C(3)—O(4)—C(5)	174·5 (3)	162·3 (2)
C(3)—O(4)—C(5)—C(6)	−157·7 (3)	174·4 (2)
O(4)—C(5)—C(6)—O(7)	64·7 (4)	66·4 (3)
C(5)—C(6)—O(7)—C(8)	−170·6 (3)	−95·6 (2)
C(6)—O(7)—C(8)—C(9)	178·4 (3)	163·5 (2)
O(7)—C(8)—C(9)—N(1')	−69·1 (4)	−77·8 (2)
C(8)—C(9)—N(1')—C(2')	83·5 (3)	167·9 (2)
C(9)—N(1')—C(2')—C(3')	178·9 (3)	116·9 (2)

respect to this plane. The angle between the mean plane of the central section and that of the ends is 124 (1)°. Because of the differences in conformation and the resulting differences in strain energy, the distances around the crown rings differ significantly. The C—O and C—C distances in (2) average 1·423 Å [range 1·419 (3) to 1·428 (3) Å] and 1·486 Å [range 1·477 (3) to 1·492 (3) Å], respectively. The same bonds in (1) average 1·404 Å [range 1·391 (3) to 1·418 (4) Å] and 1·496 Å [range 1·487 (5) to 1·505 (5) Å].

The seven-membered tropolone ring is planar (0·029 Å r.m.s.d.) with O(10) and O(11) lying −0·108 and +0·153 Å out of the least-squares plane. The tropolonate ion makes an angle of 93·2 (8)° with the least-squares plane through the dication. A bifurcated hydrogen bond is formed between N(1)—H(1B) and O(10) and O(11) while a single hydrogen bond is formed between N(1)—H(1A) and O(11) of an adjacent molecule, see Table 3. The structure may be compared with that of sodium tropolonate (Sasada & Nitta, 1956; Shiono, 1961) and tropolone (Shimanouchi & Sasada, 1973). In tropolone the C=O and C—OH distances are 1·261 (3) and 1·333 (3) Å, respectively. The molecule is planar (0·036 Å maximum deviation) and there is significant bond alternation around the seven-membered ring which is consistent with a localized heptatrienone system. In sodium tropolonate the two C—O distances are statistically equivalent, 1·290 (5) and 1·278 (5) Å, and the O atoms are coordinated to the sodium ion. The anion is planar within 0·011 Å with one O being out of the plane by 0·03 Å (Shimanouchi & Sasada, 1973). Although statistically significant differences in bond lengths are observed, there is no indication of bond alternation and chemically equivalent bonds are statistically equivalent. In complex (1) the two C—O distances are nonequivalent, 1·248 (3) and 1·273 (4) Å, but more closely approximate the values in the sodium tropolonate structure. The chemically equivalent bonds are not all statistically equivalent. Some bond alternation might be indicated by the data, *i.e.* C(11)—C(12), C(13)—C(14) and C(15)—C(16) show greater double-bond character; however, the structure is consistent with some delocalization and appears to be intermediate between the two extremes.
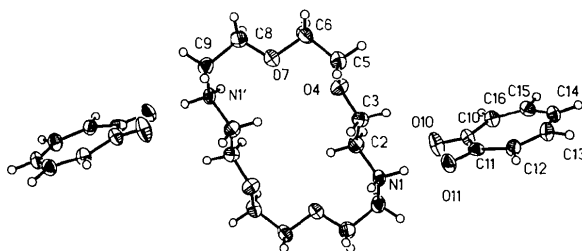

Fig. 1. The 2:1 ionic compound (1) formed between tropolone and 'diaza-18-crown-6'. Thermal ellipsoids are drawn at the 35% probability level and H atoms are represented by spheres of arbitrary size.
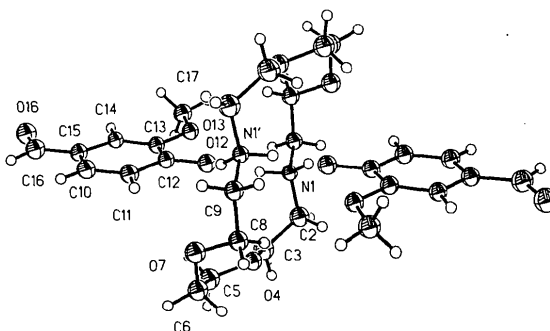

Fig. 2. The 2:1 ionic compound (2) formed between 4-hydroxy-3-methoxybenzaldehyde and 'diaza-18-crown-6'. Thermal ellipsoids are drawn at the 35% probability level and H atoms are represented by spheres of arbitrary size.

In complex (2) the phenyl ring and attached atoms of the 4-hydroxy-3-methoxybenzaldehyde anion are planar (0·016 Å r.m.s.d.). The anion makes an angle of 76·5 (8)° with a least-squares plane through the central portion of the dication. The bonds in the benzene ring are statistically nonequivalent and are consistent with a quinoid-like contribution to the bonding, *i.e.* C(13)—C(14) = 1·368 (3) Å and C(10)—C(11) = 1·375 (3) Å are shorter than the other bonds. The C(12)—O(12) bond is 1·295 (3) Å with O(12) forming a hydrogen bond to N(1ii)—H(1Bii) and to N(1)—H(1A), see Table 3.

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Structure and Absolute Configuration of (Z)-(+)-2-Bromo-2-cycloundecenyl Camphanate

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Abstract. $C_{21}H_{31}BrO_4$, $M_r = 427.38$, monoclinic, $P2_1$, $a = 8.050$ (1), $b = 12.703$ (3), $c = 10.658$ (1) Å, $\beta = 102.56$ (2)°, $V = 1063.80$ Å^3, $Z = 2$, $D_x = 1.334$ g cm^{-3}, $\lambda(Cu K\alpha) = 1.5418$ Å, $\mu = 28.09$ cm^{-1}, experimental temperature 296 K. $F(000) = 448$, $R = 0.045$ for 2015 unique reflections with $I \geq \sigma(I)$. The conformation of the 11-membered ring in this compound and its absolute configuration (1R) have been determined.

Introduction. As part of a study of the chiroptical properties of medium-ring 2-cycloalkenyl *p*-bromobenzoates, several *E*- and *Z*- 2-substituted 2-cycloalkenyl esters were required (Ito, Kasai & Ziffer, 1987, 1988). In order to simplify the chemistry necessary to assign the absolute stereochemistry of some of these compounds we prepared several 2-bromo derivatives, using a procedure employing silver salt solvolyses of the appropriate dibromobicyclo[*n*.1.0]alkane (Ito, Ziffer & Bax, 1986). While our reaction conditions yielded the expected 2-bromo-2-cycloalkenyl acetates, we were surprised to find that the geometry about the double bond differed in some cases from that anticipated for a concerted reaction. In those cases where it was not possible to determine the geometry about the double bond by comparing the physical properties of the compound with data in the literature, it became necessary to make the assignment from data on $^3J(C-H)$ coupling constants (Ito, Ziffer & Bax, 1986) rather than invoking arguments concerning the mechanism of the solvolysis. There were few model

compounds in which the geometry about such trisubstituted double bonds had been established rigorously. We converted one critical compound that had been prepared by this reaction sequence, 2-bromo-2-cycloundecenyl acetate, to the corresponding camphanate ester (1) which had been employed for resolving the allylic alcohol. The crystals were well formed and were chosen for an X-ray structure determination to establish the geometry about the double bond, the absolute configuration about the carbinol C atom and the conformation of the 11-membered ring, since to our knowledge there has been no previous crystallographic work on such compounds. Camphanate derivatives have been used several times to determine absolute configuration, *e.g.* Cane, Nachbar, Clardy & Finer (1977); Parker (1983); Dung, Armstrong, Anderson & Williams (1983); Parker, Taylor, Ferguson & Tonge (1986).

(1)
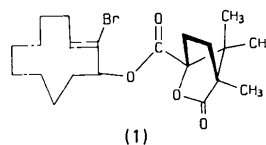
Experimental. Preparation of (1): Cyclodecene (10 g), prepared by dehydrating cyclodecanol with phosphoric acid, was treated with bromoform (17 g) and potassium *tert*-butoxide (8.4 g) in 15 ml of hexane at 273 K as described by Skattebol & Solomon (1973). The resulting dibromobicyclo[10.1.0]undecane was rearranged to 2-bromo-2-cycloundecenyl acetate by treating 2.03 g of the compound in 5 ml of acetonitrile with silver acetate (1.33 g) and acetic acid (2 ml). The mixture was refluxed for 1 h and cooled to room temperature. The solution was filtered and the filtrate

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