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Structure of 5,11,17,23-Tetra-*tert*-butyl-25,27-dicyanomethoxy-26,28-dimethoxycalix[4]arene

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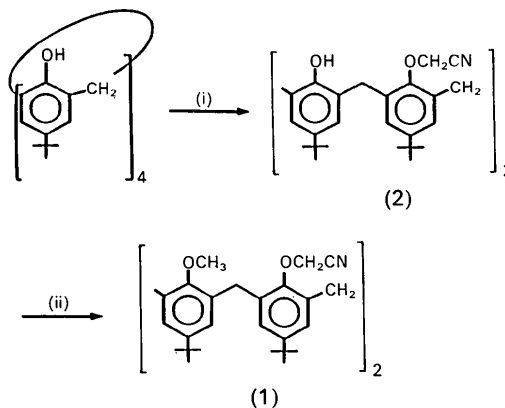
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Abstract. β,β' -{5,11,17,23-Tetra-*tert*-butyl-26,28-dimethoxypentacyclo[19.3.1.1^{3,7}.1^{9,13}.1^{15,19}]octacosal(25),3,5,7(28),9,11,13(27),15,17,19(26),21,23-dodecaene-25,27-diylidioxy}diacetonitrile, C₅₀H₆₂N₂O₄, $M_r = 755.1$, monoclinic, $C2/c$, $a = 14.659(1)$, $b = 16.591(1)$, $c = 37.558(5)$ Å, $\beta = 90.38(1)^\circ$, $V = 9134.19$ Å³, $Z = 8$, $D_x = 1.098$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.7107$ Å, $\mu = 0.64$ cm⁻¹, $F(000) = 3264.0$, $T = 293$ K, $wR = 0.060$ ($w = 1$) for 4394 observed reflections with $I > 3\sigma(I)$. The X-ray structure shows that the compound adopts a partial-cone conformation in which the two anisole moieties are parallel to each other with the methoxy groups pointing outwards. The cyano groups are in *anti* positions.

Introduction. Calix[4]arenes are cyclic tetramers extracted from the condensation products of phenol and formaldehyde in basic media (Gutsche, 1989; Vicens & Bohmer, 1991). They may exist in four different conformations: cone, partial-cone, 1,2-alternate and 1,3-alternate (Gutsche, 1989; Vicens & Bohmer, 1991). In most cases, non-functionalized

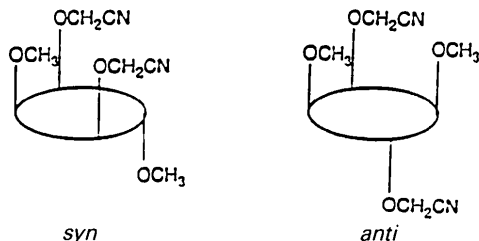
calix[4]arenes as well as their *O*-alkyl derivatives display a cone conformation. Aiming at the preparation of mixed *O*-functionalized ligands derived from *p*-*tert*-butylcalix[4]arene, we synthesized the dimethoxydinitrile ligand (1), which was shown to exist in partial-cone conformation, as deduced from NMR experiments.



(i) BrCH₂CN, K₂CO₃, acetonitrile, reflux 25h
(ii) CH₃I, K₂CO₃, acetonitrile, reflux 8h

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In order to establish the stereochemistry of the nitrile groups, *syn* or *anti*, a crystallographic study was undertaken.



Experimental. The compound was prepared by treating *p*-*tert*-butylcalix[4]arene with a tenfold excess of bromoacetonitrile in the presence of 10 equivalents of potassium carbonate in refluxing acetonitrile. This produces the cone 1,3-dinitrile derivative (2) as deduced from ¹H NMR: two *tert*-butyl singlets at 0.88 and 1.33 p.p.m. and a single *AB* system for the bridging methylene groups at 3.44 and 4.22 p.p.m. ($J_{AB} = 13.5$ Hz). Derivative (2) was then reacted with an excess of iodomethane in the presence of 10 equivalents of potassium carbonate in refluxing acetonitrile to give (1) in *ca* 30% yield. The partial-cone conformation of (1) was shown by ¹H NMR: three *tert*-butyl singlets at 1.12, 1.32 and 1.43 p.p.m. in 2:1:1 integral ratio, two *AB* systems for the bridging methylene groups at 3.21 and 4.22 p.p.m. ($J_{AB} = 13.5$ Hz), and at 3.79 and 3.84 p.p.m. ($J_{AB} = 12.5$ Hz), and two singlets for the cyanomethoxy groups at 3.65 and 4.10 p.p.m. Crystals of (1) suitable for X-ray diffraction were obtained from methanol by slow evaporation at 298 K.

A white crystal of dimensions 0.5 × 0.30 × 0.40 mm was mounted on an Enraf-Nonius CAD-4 diffractometer with graphite-monochromated Mo *K*α radiation. Lattice parameters were determined from least-squares analysis of 25 reflections with 20 < θ < 27°. Using ω-2θ scans, scan width 0.60°, scan speed 0.010° s⁻¹, 16 372 independent reflections with sinθ/λ < 0.594 Å⁻¹ were measured in the range -17 < *h* < 17, 0 < *k* < 19, 0 < *l* < 44, of which 4394 reflections satisfied $I > 3\sigma(I)$. The intensities of four reflections (467, 6,0,12, 5,1,15, 1,7,10) measured every 2 h of X-ray exposure time showed no significant variation. Lorentz-polarization corrections were applied but absorption and extinction were ignored. The structure was solved by direct methods using 499 reflections with $E > 2.0$ in *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and Fourier methods; anisotropic full-matrix refinement on *F* (*SHELX*76; Sheldrick, 1976). Phenyl H atoms were inserted at calculated positions; every methyl group was treated as a rigid CH₃ unit with its original orientation taken from the strongest H-atom peak

Table 1. Fractional atomic coordinates (× 10⁴) and equivalent isotropic thermal factors (Å² × 10³)

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}
C(1)	3255 (3)	621 (2)	1022 (1)	36 (5)
C(2)	3583 (3)	-21 (2)	826 (1)	41 (5)
C(3)	3633 (3)	69 (3)	453 (1)	46 (6)
C(4)	3352 (3)	770 (3)	284 (1)	43 (6)
C(5)	3032 (3)	1402 (3)	494 (1)	42 (5)
C(6)	2968 (3)	1336 (2)	863 (1)	38 (5)
C(7)	3366 (3)	870 (3)	-125 (1)	52 (6)
C(8)	3673 (4)	98 (4)	-313 (1)	74 (8)
C(9)	2398 (4)	1085 (4)	-255 (1)	72 (8)
C(10)	4023 (4)	1558 (4)	-222 (1)	75 (8)
O(1)	3096 (2)	529 (2)	1388 (1)	48 (4)
C(11)	3868 (4)	587 (3)	1617 (1)	55 (7)
C(12)	4140 (3)	1419 (3)	1701 (1)	53 (6)
N(1)	4397 (3)	2037 (3)	1783 (1)	68 (7)
C(13)	3840 (3)	-822 (3)	994 (1)	50 (5)
C(14)	3004 (3)	-1270 (2)	1134 (1)	44 (5)
C(15)	2346 (3)	-1546 (3)	893 (1)	45 (5)
C(16)	1564 (3)	-1942 (3)	1008 (1)	42 (6)
C(17)	1420 (3)	-2006 (2)	1375 (1)	42 (5)
C(18)	2064 (3)	-1733 (2)	1619 (1)	38 (5)
C(19)	2871 (3)	-1407 (2)	1496 (1)	38 (5)
C(20)	863 (3)	-2296 (3)	744 (1)	54 (7)
C(21)	1194 (6)	-2304 (8)	376 (2)	107 (17)
C(22)	668 (5)	-3175 (5)	845 (2)	82 (13)
C(23)	-40 (6)	-1881 (6)	789 (3)	98 (20)
O(2)	3549 (2)	-1189 (2)	1742 (1)	45 (4)
C(24)	4196 (4)	-1824 (3)	1804 (2)	77 (8)
C(25)	1858 (3)	-1756 (2)	2018 (1)	43 (5)
C(26)	1787 (3)	-916 (2)	2177 (1)	40 (5)
C(27)	2425 (3)	-658 (3)	2433 (1)	40 (5)
C(28)	2416 (3)	109 (2)	2573 (1)	37 (5)
C(29)	1744 (3)	640 (2)	2450 (1)	38 (5)
C(30)	1091 (3)	411 (2)	2200 (1)	36 (5)
C(31)	1113 (3)	-375 (3)	2074 (1)	36 (5)
C(32)	3148 (3)	423 (3)	2837 (1)	42 (6)
C(33)	2698 (4)	856 (4)	3152 (1)	67 (9)
C(34)	3752 (4)	-246 (4)	2975 (2)	74 (11)
C(35)	3738 (4)	1048 (4)	2640 (2)	67 (9)
O(3)	470 (3)	-607 (2)	1816 (1)	38 (3)
C(36)	-335 (3)	-940 (3)	1969 (1)	48 (6)
C(37)	-1018 (4)	-1001 (3)	1685 (2)	61 (8)
N(2)	-1571 (3)	-1048 (3)	1470 (2)	83 (9)
C(38)	413 (3)	1028 (3)	2056 (1)	44 (5)
C(39)	582 (3)	1250 (2)	1668 (1)	40 (5)
C(40)	-75 (3)	1100 (2)	1406 (1)	41 (5)
C(41)	58 (3)	1287 (3)	1051 (1)	42 (5)
C(42)	912 (3)	1590 (2)	954 (1)	45 (5)
C(43)	1595 (3)	1742 (2)	1205 (1)	39 (5)
C(44)	1410 (3)	1587 (2)	1560 (1)	36 (5)
C(45)	-669 (3)	1144 (3)	762 (1)	48 (6)
C(46)	-297 (5)	586 (5)	472 (2)	87 (13)
C(47)	-1537 (4)	792 (6)	912 (2)	105 (14)
C(48)	-903 (4)	1959 (4)	593 (2)	85 (10)
O(4)	2073 (2)	1746 (2)	1819 (1)	38 (3)
C(49)	2050 (4)	2563 (3)	1947 (1)	53 (7)
C(50)	2533 (3)	2001 (2)	1077 (1)	44 (5)

Table 2. Bond lengths (Å) and angles (°)

C(1)–C(2)	1.384 (5)	C(17)–C(18)	1.388 (6)
C(1)–C(6)	1.392 (5)	C(18)–C(19)	1.383 (6)
C(1)–O(1)	1.404 (5)	C(18)–C(25)	1.532 (5)
C(2)–C(3)	1.412 (5)	C(19)–O(2)	1.399 (5)
C(2)–C(13)	1.518 (6)	C(20)–C(21)	1.468 (9)
C(3)–C(4)	1.386 (7)	C(20)–C(22)	1.534 (10)
C(4)–C(5)	1.395 (6)	C(20)–C(23)	1.504 (10)
C(4)–C(7)	1.547 (5)	O(2)–C(24)	1.435 (6)
C(5)–C(6)	1.395 (5)	C(25)–C(26)	1.519 (5)
C(6)–C(50)	1.509 (5)	C(26)–C(27)	1.402 (6)
C(7)–C(8)	1.532 (8)	C(26)–C(31)	1.388 (6)
C(7)–C(9)	1.539 (7)	C(27)–C(28)	1.379 (6)
C(7)–C(10)	1.538 (8)	C(28)–C(29)	1.397 (6)
O(1)–C(11)	1.420 (7)	C(28)–C(32)	1.546 (6)
C(11)–C(12)	1.470 (7)	C(29)–C(30)	1.391 (6)
C(12)–N(1)	1.133 (7)	C(30)–C(31)	1.388 (6)
C(13)–C(14)	1.528 (6)	C(30)–C(38)	1.523 (6)
C(14)–C(15)	1.394 (6)	C(31)–O(3)	1.401 (6)
C(14)–C(19)	1.395 (5)	C(32)–C(33)	1.536 (6)
C(15)–C(16)	1.392 (6)	C(32)–C(34)	1.510 (8)
C(16)–C(17)	1.399 (5)	C(32)–C(35)	1.541 (8)
C(16)–C(20)	1.540 (6)	O(3)–C(36)	1.426 (6)

Table 2 (cont.)

C(36)—C(37)	1.463 (8)	C(42)—C(43)	1.394 (6)
C(37)—N(2)	1.143 (9)	C(43)—C(44)	1.385 (5)
C(38)—C(39)	1.525 (5)	C(43)—C(50)	1.522 (6)
C(39)—C(40)	1.396 (6)	C(44)—O(4)	1.396 (5)
C(39)—C(44)	1.399 (6)	C(45)—C(46)	1.532 (9)
C(40)—C(41)	1.385 (5)	C(45)—C(47)	1.512 (8)
C(41)—C(42)	1.399 (6)	C(45)—C(48)	1.532 (8)
C(41)—C(45)	1.533 (6)	O(4)—C(49)	1.439 (6)
C(2)—C(1)—C(6)	122.2 (4)	C(11)—C(12)—N(1)	174.9 (5)
C(2)—C(1)—O(1)	119.9 (4)	C(2)—C(13)—C(14)	111.7 (3)
C(6)—C(1)—O(1)	117.5 (3)	C(13)—C(14)—C(15)	119.4 (4)
C(1)—C(2)—C(3)	117.8 (4)	C(13)—C(14)—C(19)	122.1 (3)
C(1)—C(2)—C(13)	122.6 (4)	C(15)—C(14)—C(19)	118.6 (4)
C(3)—C(2)—C(13)	119.5 (4)	C(14)—C(15)—C(16)	121.4 (4)
C(2)—C(3)—C(4)	121.9 (4)	C(15)—C(16)—C(17)	118.1 (4)
C(3)—C(4)—C(5)	118.1 (4)	C(15)—C(16)—C(20)	121.7 (4)
C(3)—C(4)—C(7)	122.7 (4)	C(17)—C(16)—C(20)	120.2 (4)
C(5)—C(4)—C(7)	119.2 (4)	C(16)—C(17)—C(18)	121.3 (4)
C(4)—C(5)—C(6)	121.9 (4)	C(17)—C(18)—C(19)	119.1 (4)
C(1)—C(6)—C(5)	118.2 (4)	C(17)—C(18)—C(25)	120.0 (3)
C(1)—C(6)—C(50)	121.4 (4)	C(19)—C(18)—C(25)	120.8 (3)
C(5)—C(6)—C(50)	120.3 (4)	C(14)—C(19)—C(18)	121.0 (4)
C(4)—C(7)—C(8)	112.0 (4)	C(14)—C(19)—O(2)	119.9 (3)
C(4)—C(7)—C(9)	108.8 (4)	C(18)—C(19)—O(2)	119.1 (3)
C(4)—C(7)—C(10)	109.1 (4)	C(16)—C(20)—C(21)	112.8 (5)
C(8)—C(7)—C(9)	108.7 (4)	C(16)—C(20)—C(22)	109.0 (4)
C(8)—C(7)—C(10)	108.9 (4)	C(16)—C(20)—C(23)	109.6 (4)
C(9)—C(7)—C(10)	109.3 (4)	C(21)—C(20)—C(22)	106.8 (6)
C(1)—O(1)—C(11)	116.7 (4)	C(21)—C(20)—C(23)	114.1 (6)
O(1)—C(11)—C(12)	114.1 (4)	C(22)—C(20)—C(23)	104.0 (5)
C(19)—O(2)—C(24)	112.4 (4)	C(31)—O(3)—C(36)	112.5 (4)
C(18)—C(25)—C(26)	112.1 (3)	O(3)—C(36)—C(37)	107.3 (4)
C(25)—C(26)—C(27)	120.2 (3)	C(36)—C(37)—N(2)	178.0 (6)
C(25)—C(26)—C(31)	122.2 (4)	C(30)—C(38)—C(39)	113.0 (3)
C(27)—C(26)—C(31)	117.6 (4)	C(38)—C(39)—C(40)	121.0 (3)
C(26)—C(27)—C(28)	122.5 (4)	C(38)—C(39)—C(44)	121.4 (3)
C(27)—C(28)—C(29)	117.6 (4)	C(40)—C(39)—C(44)	117.5 (4)
C(27)—C(28)—C(32)	123.3 (4)	C(39)—C(40)—C(41)	122.6 (4)
C(29)—C(28)—C(32)	119.0 (3)	C(40)—C(41)—C(42)	117.6 (4)
C(28)—C(29)—C(30)	122.2 (4)	C(40)—C(41)—C(45)	122.9 (4)
C(29)—C(30)—C(31)	117.9 (4)	C(42)—C(41)—C(45)	119.5 (4)
C(29)—C(30)—C(38)	120.1 (3)	C(41)—C(42)—C(43)	121.9 (4)
C(31)—C(30)—C(38)	121.9 (4)	C(42)—C(43)—C(44)	118.3 (4)
C(26)—C(31)—O(3)	122.1 (4)	C(42)—C(43)—C(50)	118.9 (3)
C(30)—C(31)—O(3)	119.3 (4)	C(44)—C(43)—C(50)	122.6 (3)
C(28)—C(32)—C(33)	110.6 (4)	C(39)—C(44)—C(43)	121.9 (4)
C(28)—C(32)—C(34)	112.0 (4)	C(39)—C(44)—O(4)	118.3 (3)
C(28)—C(32)—C(35)	107.9 (4)	C(43)—C(44)—O(4)	119.8 (3)
C(33)—C(32)—C(34)	109.5 (4)	C(41)—C(45)—C(46)	110.3 (4)
C(33)—C(32)—C(35)	107.5 (4)	C(41)—C(45)—C(47)	112.4 (4)
C(34)—C(32)—C(35)	109.3 (4)	C(41)—C(45)—C(48)	108.1 (4)
C(46)—C(45)—C(48)	108.5 (5)	C(46)—C(45)—C(47)	109.6 (5)
C(47)—C(45)—C(48)	107.9 (5)	C(44)—O(4)—C(49)	113.1 (3)
		C(6)—C(50)—C(43)	110.3 (3)

on a difference Fourier synthesis; other H atoms from $\Delta\rho$ synthesis and theoretically adjusted. No H-atom coordinates were refined. Final $R = 0.060$ and $(\Delta/\sigma)_{\max} = 0.025$. The largest peaks in a final difference map were 0.35 and $-0.26 \text{ e } \text{\AA}^{-3}$. Atomic scattering factors in the analytical forms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV) and for H atoms from Stewart, Davidson & Simpson (1965). All calculations were made on a MicroVAX II.

Discussion. Final atomic coordinates of non-H atoms and equivalent isotropic thermal parameters are listed in Table 1.* Bond lengths and angles are

* 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 55557 (29 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: PA1003]

shown in Table 2. A *PLUTO* (Motherwell & Clegg, 1978) drawing of the molecule is shown in Fig. 1. The structure exhibits a partial-cone conformation as deduced from NMR experiments. The two anisole moieties are nearly parallel to each other with the methoxy groups pointing outwards while the cyano groups are in *anti* positions (Fig. 2). This indicates that during the methylation process, one of the cyano groups rotates through 'the oxygen-annulus ring' (Iwamoto, Araki & Shinkai, 1991). The rotation mechanism implies a low steric demand for the *syn-to-anti* and cone-to-partial cone transformations. A driving force for this transformation could result from a dipole-dipole repulsion between the two cyano groups. The dihedral angles between the phenyl ring and the mean plane of the methylene groups are $93, 95^\circ$ for the anisole and $37, 90^\circ$ for the nitrile groups. It appears, as shown in Fig. 2, that one of these nitrile groups is in an inside position and, thus, it partly fills the cavity of the molecule.

We are currently investigating the possibility of using such difunctional nitriles for preparation of organometallic polymers.

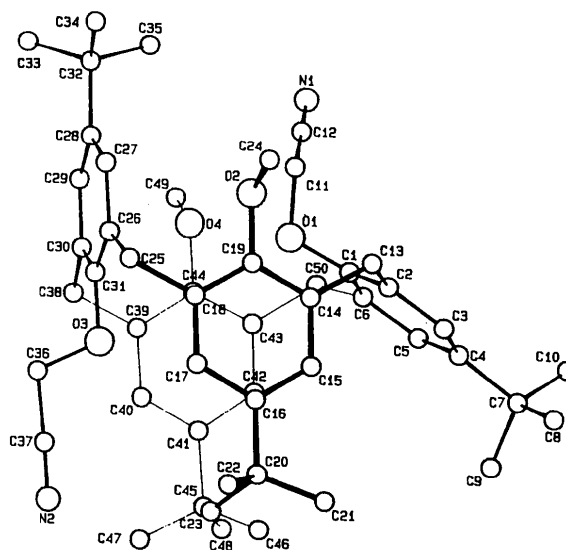


Fig. 1. *PLUTO* (Motherwell & Clegg, 1978) perspective view of (1) with the atom-labelling scheme.

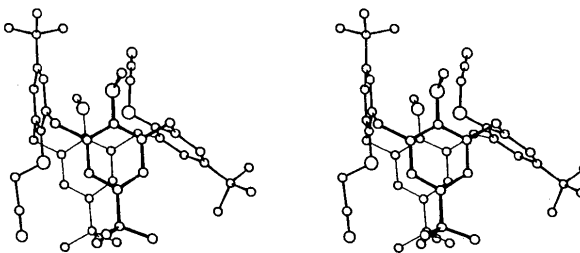


Fig. 2. Stereoscopic view of (1) viewed in the same orientation as Fig. 1.

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Structure of Dimethyl (2*R*,3*R*)-2-[(*S*)-Bromo(cyclohexyl)methyl]oxy}-3-[(diphenylboryl)oxy]-1,4-butanedioate

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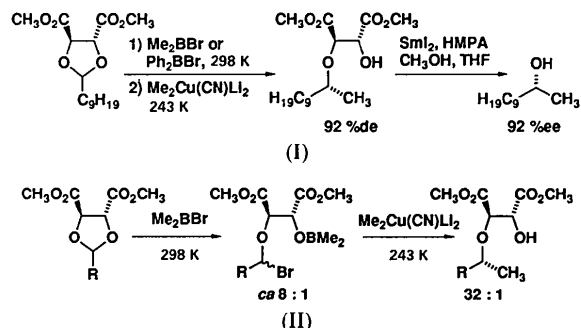
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Abstract. C₂₅H₃₀BBrO₆, *M_r* = 517.23, monoclinic, *P*2₁, *a* = 9.1584 (14), *b* = 6.910 (3), *c* = 20.224 (4) Å, β = 96.241 (14)°, *V* = 1272.3 (6) Å³, *Z* = 2, *D_x* = 1.350 g cm⁻³, λ(Mo *K*α) = 0.71069 Å, μ = 16.3 cm⁻¹, *F*(000) = 535.62, *T* = 230 (1) K, final *R* = 0.041 for 1531 observed reflections. A reactive intermediate in the dialkylboron bromide promoted opening of tartrate acetals by higher-order cuprates has been isolated and characterized by X-ray crystallography.

Introduction. The Lewis-acid-mediated reaction of acetals with nucleophiles such as allylsilanes has proven to be an extremely effective and often highly stereoselective method for the formation of carbon-carbon bonds (Alexakis & Mangeney, 1990). Recently, we described a novel reaction in which acetals of dimethyl tartrate are treated with dimethyl- or diphenylboron bromides and higher-order cuprates to produce, after auxiliary removal, secondary alcohols in high enantiomeric excess (I)

(Guindon, Simoneau, Yoakim, Gorys, Lemieux & Ogilvie, 1991). Our studies of the mechanism of this reaction have revealed that the process involves two steps with several unusual features (II). In order to better understand the reaction, it became necessary to determine the structure of the reactive bromoether intermediates. The structure of one of these highly reactive intermediates was elucidated by single-crystal X-ray diffraction (Fig. 1). We are continuing to investigate the mechanism of this process and will disclose our results in due course.



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