Table 3. *Hydrogen-bonding geometry* (Å, °)

D	H	*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1	H1O	O2	0.92 (2)	1.77 (2)	2.590 (1)	147 (2)
O2	H2O	O3	1.07 (3)	1.38 (3)	2.393 (1)	156 (2)

The structure was solved by direct methods in the non-centrosymmetric space group *P*1 but inspection of the atomic coordinates clearly showed the crystal structure to be centrosymmetric. All H-atom positions were found in difference syntheses. Data collection and cell refinement: Enraf–Nonius CAD-4 software. Data reduction: *MolEN* (Fair, 1990). Program used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976); *SCHAKAL*92 (Keller, 1992). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SE1049). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1994). C**50**, 1320–1323

5,11,17,23-Tetra-*tert*-butyl-25,26,27,28-[(5-methylfurfuryl)oxycarbonylmethoxy]calix[4]arene

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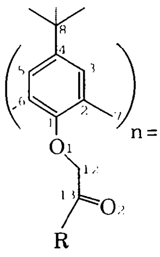
Abstract

The title molecule [tetrakis(5-methylfurfuryl) 5,11,17,23-tetra-*tert*-butylpentacyclo[19.3.1.$1^{3,7}$.$1^{9,13}$.$1^{15,19}$]octacosa-1(25),3,5,7(28),9,11,13(27),15,17,19(26),21,23-dodecaene-25,26,27,28-tetrayltetraoxytetraacetate, $C_{76}H_{88}O_{16}$], which complexes Na^+ preferentially in solution, has twofold crystallographic symmetry and adopts a distorted cone conformation in the solid state. The orientation of the aromatic rings is such that two rings are almost parallel [interplanar angle 5.6 (1)°] and the other two are almost perpendicular to each other [interplanar angle 90.6 (1)°]. This conformation precludes any solvent molecule being enclathrated within the small molecular cavity.

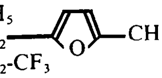
Comment

The ability of calix[4]arene tetraesters of general formula (1) to form complexes with alkali metal cations is well established (Gutsche, 1989; Vicens & Böhmer, 1990). For the ethyl ester (2) there is a distinct preference for Na^+ over the other alkali cations as revealed by extraction studies and stability-constant measurements (Arnaud-Neu *et al.*, 1989). A study of substituent effects shows that changing the alkoxy moiety of the ester group can cause significant changes in complexation selectivities for Na^+ relative to K^+ (Arnaud-Neu *et al.*, 1992). One of the objectives of this research is to maximize complexation selectivities of chosen cations, for possible applications in sensors. Preliminary extraction studies with a new member of the tetraester series, the 5-methylfurfuryl derivative (3), have shown that this compound has no detectable affinity for Li^+, K^+, Rb^+ and Cs^+, though it does maintain the characteristic response of the series to Na^+. It was of interest, therefore, to compare the molecular structure of (3) with that of the ethyl and trifluoroethyl esters, (2) and (4), respectively, whose molecular structures have been determined already by X-ray diffraction (Arnaud-Neu *et al.*, 1989; Arnaud-Neu *et al.*, 1992).



(1)

(2) R = O-C_2H_5

(3) R = O-CH_2––CH_3

(4) R = O-CH_2-CF_3

(5) R = Cl

Molecule (3) (Fig. 1) has twofold crystallographic symmetry and adopts a disorted cone shape whose conformation can be defined by the angles which the aromatic rings *A*, *B*, *A'*, *B'* make with the plane through the macrocyclic ring methylene C atoms (C7*A*, C7*B*, C7*A'*, C7*B'*): *A*, *A'* 92.8 (1), *B*, *B'* 44.7 (1)°. The aromatic rings *A* and *A'* are almost parallel [at an interplanar angle of 5.6 (1)°] and tilted so as to increase the separation of the *tert*-butyl groups; the shortest C···C cross-cup calix distance is 5.380 (5) Å (C2*A*···C6*A'*). Rings *B* and *B'* are almost normal to each other [interplanar angle 90.6 (1)°]. The O···O separations of *cis*-adjacent ether O atoms are O1*A*···O1*B* 3.276 (4), O1*A*···O1*B'* 3.117 (4) Å. An alternative way of describing calixarene conformations has been proposed by Ugozzoli & Andreetti (1992) in terms of torsion angles (to which they assign the labels φ and χ) involving the methylene C atoms. Table 2 lists the φ and χ values for (3) and the corresponding values for (2) and (4) for comparison. The torsion angle values and their sign sequence (Table 2) are typical for this kind of conformation with two opposite aromatic rings almost parallel, and two almost normal to each other.

Molecular dimensions (summarized in Table 3) are unexceptional and serve to establish the structure. The shortest intermolecular contacts correspond with normal van der Waals contacts.

The pendant furfuryl ester side chains in (3) are oriented in the solid state in such a way that the four furan rings are almost in a plane normal to the molecular twofold axis, with the furan O atoms remote from the phenolic O and carboxyl O atoms. Complex formation by metal cations with molecules (2)–(4) in solution requires rotation of the carbonyls which are largely *exo* in the solid state into the *endo* position for interaction with the metal cation. The extent of rotation required is comparable for the three molecules. It appears that the disposition of the pendant chains in the solid state is the result of crystal-packing effects and the structures as such (apart from confirming the familiar distorted cone conformation) do not allow predictions of their effectiveness in complexation.

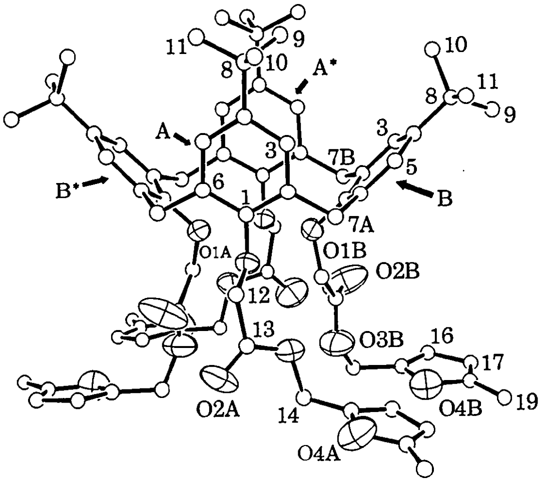


Fig. 1. A side view of (3) with the crystallographic numbering scheme; the O atoms are shown with displacement ellipsoids drawn at the 50% probability level. For clarity, the C atoms are drawn as small spheres of arbitrary size and only one of the two observed orientations of the methyl C atoms of the ring-*B* *tert*-butyl group is shown; the H atoms are omitted.

Experimental

Compound (3) was prepared from the known tetra-acid chloride (5) and 5-methylfurfuryl alcohol using a published procedure (Arnaud-Neu *et al.*, 1992). Suitable crystals of (3) were obtained from ethanol–dichloromethane.

Crystal data

$C_{76}H_{88}O_{16}$
M_r = 1257.51
Monoclinic
$C2/c$
a = 20.4313 (16) Å
b = 13.6266 (15) Å
c = 25.6804 (21) Å
β = 92.863 (6)°
V = 7140.7 (11) Å^3
Z = 4
D_x = 1.170 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71067 Å
Cell parameters from 25 reflections
θ = 9.50–12.00°
μ = 0.08 mm^{-1}
T = 293 K
Plate
0.30 × 0.30 × 0.10 mm
Colourless

Data collection

Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none

$R_{\rm int}$ = 0.034
$\theta_{\rm max}$ = 26.91°
h = −26 → 26
k = 0 → 17
l = 0 → 32

7942 measured reflections
7775 independent reflections
3161 observed reflections
[$I > 3.0\sigma(I)$]
3 standard reflections
frequency: 60 min
intensity variation: 1.0%

Refinement

Refinement on F
$R = 0.070$
$wR = 0.093$
$S = 2.32$
3161 reflections
424 parameters
H atoms refined as riding (C—H 0.95 Å)
$w = 1/[\sigma^2(F) + 0.0008F^2]$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.50$ e Å^{-3}
$\Delta\rho_{\min} = -0.43$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\text{eq}} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
C1*A*	0.38396 (18)	0.2133 (3)	0.19485 (15)	0.0349 (21)
C2*A*	0.41284 (18)	0.1653 (3)	0.15469 (14)	0.0364 (21)
C3*A*	0.41002 (20)	0.0643 (3)	0.15289 (16)	0.0441 (24)
C4*A*	0.37931 (20)	0.0092 (3)	0.19068 (16)	0.0455 (23)
C5*A*	0.35424 (20)	0.0600 (3)	0.23145 (16)	0.0460 (23)
C6*A*	0.35609 (18)	0.1617 (3)	0.23527 (15)	0.0381 (21)
C7*A*	0.45080 (20)	0.2200 (3)	0.11398 (15)	0.0425 (23)
C8*A*	0.3738 (3)	−0.1019 (3)	0.18553 (20)	0.066 (3)
C9*A*	0.4387 (4)	−0.1460 (5)	0.1746 (5)	0.180 (9)
C10*A*	0.3232 (5)	−0.1269 (5)	0.1425 (4)	0.162 (7)
C11*A*	0.3516 (5)	−0.1504 (5)	0.2337 (3)	0.157 (8)
C12*A*	0.32395 (22)	0.3565 (3)	0.17553 (18)	0.057 (3)
C13*A*	0.33230 (23)	0.4645 (3)	0.16816 (18)	0.058 (3)
C14*A*	0.3963 (3)	0.5882 (4)	0.13047 (24)	0.094 (5)
C15*A*	0.4093 (3)	0.6038 (4)	0.07612 (24)	0.090 (4)
C16*A*	0.4624 (3)	0.6149 (7)	0.0510 (3)	0.148 (7)
C17*A*	0.4430 (4)	0.6312 (8)	−0.0013 (3)	0.166 (8)
C18*A*	0.3805 (3)	0.6314 (6)	−0.0069 (3)	0.114 (5)
C19*A*	0.3328 (5)	0.6437 (7)	−0.0509 (3)	0.163 (7)
O1*A*	0.38440 (12)	0.31598 (19)	0.19519 (10)	0.0419 (15)
O2*A*	0.29622 (21)	0.5242 (3)	0.18461 (18)	0.111 (3)
O3*A*	0.38149 (18)	0.48499 (23)	0.13950 (15)	0.0826 (24)
O4*A*	0.35639 (20)	0.6146 (3)	0.04148 (16)	0.099 (3)
C1*B*	0.56480 (19)	0.2257 (3)	0.15942 (14)	0.0386 (21)
C2*B*	0.62650 (19)	0.1857 (3)	0.16886 (15)	0.0417 (23)
C3*B*	0.64752 (21)	0.1146 (4)	0.13497 (16)	0.054 (3)
C4*B*	0.60898 (22)	0.0815 (4)	0.09212 (17)	0.060 (3)
C5*B*	0.54650 (22)	0.1226 (4)	0.08607 (16)	0.054 (3)
C6*B*	0.52276 (19)	0.1928 (3)	0.11892 (15)	0.0393 (22)
C7*B*	0.66821 (19)	0.2131 (3)	0.21726 (16)	0.046 (3)
C8*B*	0.6330 (3)	0.0043 (5)	0.05589 (22)	0.097 (4)
C9*B*†	0.69475	0.04042	0.03137	0.34 (3)
C10*B*†	0.64807	−0.08934	0.08631	0.232 (21)
C11*B*†	0.58039	−0.01617	0.01331	0.266 (21)
C9*C*†	0.58863	−0.08511	0.05726	0.222 (18)
C10*C*†	0.63202	0.04489	0.00072	0.192 (17)
C11*C*†	0.70256	−0.02489	0.07302	0.294 (23)
C12*B*	0.53066 (24)	0.3902 (3)	0.17627 (19)	0.061 (3)
C13*B*	0.5855 (3)	0.4613 (3)	0.18670 (19)	0.070 (3)
C14*B*	0.6146 (4)	0.6295 (4)	0.17881 (25)	0.110 (5)
C15*B*	0.6587 (3)	0.6303 (4)	0.13434 (21)	0.082 (4)
C16*B*	0.7228 (3)	0.6233 (5)	0.1290 (3)	0.102 (4)
C17*B*	0.7340 (3)	0.6349 (5)	0.0755 (3)	0.097 (5)
C18*B*	0.6768 (3)	0.6448 (4)	0.05088 (23)	0.084 (4)
C19*B*	0.6533 (4)	0.6553 (6)	−0.0040 (3)	0.126 (6)
O1*B*	0.54407 (13)	0.29438 (20)	0.19532 (10)	0.0467 (16)
O2*B*	0.63959 (21)	0.4431 (3)	0.20140 (19)	0.122 (3)
O3*B*	0.56452 (20)	0.5510 (3)	0.17485 (15)	0.092 (3)
O4*B*	0.62795 (18)	0.6442 (3)	0.08661 (14)	0.0816 (24)

† Site occupancy = 0.5.

Table 2. *Torsion angles* (*Ugozzoli & Andreetti*, 1992) (°)

Calix φ,χ sign sequence +−,+−,+−,+− in all three structures.

	Molecule (2)		Molecule (3)		Molecule (4)	
	φ	χ	φ	χ	φ	χ
A′—*B*	+60 (1),	−103 (1)	+63.8 (3),	−99.2 (4)	+52 (1),	−97 (1)
B—*A*	+101 (1),	−66 (1)	+98.2 (4),	−63.0 (4)	+93 (1),	−68 (1)
A—*B′*	+65 (1),	−100 (1)	+63.8 (3),	−99.2 (4)	+72 (1),	−104 (1)
B′—*A′*	+100 (1),	−58 (1)	+98.2 (4),	−63.0 (4)	+101 (1),	−52 (1)

Table 3. *Selected bond lengths* (Å) (*range and mean*) *for molecule* (3)

	Range	Mean
C_{ar}—O_{ether}	1.395–1.399 (5)	1.397 (5)
C_{sp3}—O_{ether}	1.417–1.422 (5)	1.420 (5)
C_{sp3}—O_{ester}	1.460–1.480 (6)	1.470 (6)
C_{sp2}—O_{ester}	1.305–1.325 (6)	1.315 (6)
C_{sp2}—O_{furan}	1.362–1.389 (7)	1.376 (6)
C_{ar}—C_{ar}	1.375–1.400 (6)	1.387 (6)
C_{ar}—C_{sp3}	1.502–1.527 (6)	1.516 (6)
C_{sp2}=O	1.176–1.189 (6)	1.182(6)
C_{sp2}=C_{sp2}	1.28–1.33 (1)	1.30 (1)

Compound (3) crystallized in the monoclinic system; the systematic absences (*hkl* absent if $h + k = 2n + 1$, *h*0*l* absent of $l = 2n + 1$) are consistent with space groups *C*2/*c* or *Cc*; the former was assumed and confirmed by the analysis. The molecule has twofold crystallographic symmetry and it became obvious (from difference maps which were examined during the structure determination) that the methyl C atoms of the *tert*-butyl group of ring *B* were disordered over two orientations. These disordered C atoms were positioned geometrically (C—C 1.52 Å) and treated as riding atoms during the subsequent refinement. All H atoms (except those of the disordered *tert*-butyl group) were clearly visible in difference maps; they were positioned geometrically (C—H 0.95 Å) and included as riding atoms in the structure-factor calculations. The distinction between O or C in the furan rings was made easily after examining difference maps which showed clearly all the furan C—H H atoms. Examination of the structure with *PLATON*92 (Spek, 1992*a*) showed that there were no solvent-accessible voids in the crystal lattice. Data collection and cell refinement: *CAD*-4 *Software* (Enraf–Nonius, 1989). Data reduction: *NRCVAX DATRD*2 (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *NRCVAX SOLVER*. Program(s) used to refine structure: *NRCVAX LSTSQ*. Molecular graphics: *NRCVAX*; *ORTEP*II (Johnson, 1976); *PLUTON*92 (Spek, 1992*b*). Software used to prepare material for publication: *NRCVAX TABLES*.

GF thanks NSERC Canada for Research Grants.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and bond distances and angles involving non-H atoms have been deposited with the IUCr (Reference: HA1090). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of a Triazaoxatricyclodecadiene Derivative. Partial Disorder and a Packing Energy Study

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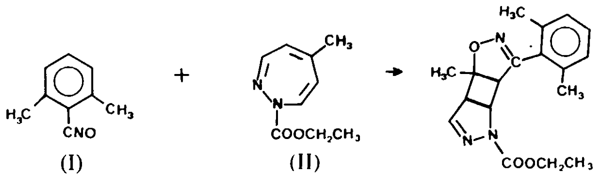
Abstract

The crystal structure of ethyl 5-(2,6-dimethylphenyl)-2-methyl-3-oxa-4,8,9-triazatricyclo[5.3.0.0^{2,6}]deca-4,9-diene-8-carboxylate, $C_{18}H_{21}N_3O_3$ (III), has been determined. The reaction between 2,6-dimethylbenzonitrile oxide (I) and ethyl 5-methyl-1,2-diazepine-1-carboxylate (II) gives rise to an *anti* tricyclo derivative with a rigid skeleton formed by 2-pyrazoline, cyclobutane and 2-isoxazoline planar rings. With respect to the central cyclobutane system, the bond lengths of which range from 1.539 (5) to 1.558 (5) Å and the bond angles of which range from 89.7 (2) to 90.7 (2)°, the 2-pyrazoline and the 2-isoxazoline planes form dihedral angles of 116 and 112°, respectively. The methyl group at the end of the ethyl carboxylate chain is disordered. Both of the corresponding orientations of the final part of the chain are consistent with the results of packing energy and semi-empirical molecular-orbital calculations.

Comment

1,3-Cycloadditions of arylnitrile oxides to ethyl 5-methyl-1,2-diazepine-1-carboxylate (II) give rise to three different types of products, one of which is definitely predominant (Beltrame, Cadoni, Carnasciali, Gelli, Lai, Mugnoli & Pani, 1992). The crystal structure of the main adduct from this type of reaction has been reported (Beltrame, Gelli, Cadoni, Carnasciali, Pani & Mugnoli, 1993). The present work deals with the determination of the structure of compound (III), one of the two minor products obtained by the 1,3-cycloaddition of the arylnitrile oxide (I) to the 1,2-diazepine (II) with concomitant [2+2] internal cycloaddition (Beltrame *et al.*, 1992).



(I) (II) (III)

Three condensed cycles are present in compound (III), as shown in Fig. 1, where atoms have been numbered in agreement with our previous work (Beltrame *et al.*, 1992). To the best of our knowledge no previous crystal structure determination has been performed on similar triazaoxatricyclodecadiene derivatives. In the rigid part of the molecule each of the three condensed rings is planar within 0.012 (3) Å (for the 2-isoxazoline ring), 0.019 (4) Å (for the 2-pyrazoline ring) and 0.002 (3) Å (for the cyclobutane ring); the dihedral angles between the cyclobutane ring mean plane and those of the two adjacent rings are 112.3 (1) and 116.0 (1)°, respectively. The phenyl ring, which is planar to within 0.014 (6) Å, is rotated with respect to the 2-isoxazoline ring, the torsion angle C13—C12—C4—N3 being 73.3 (4)°. The geometric parameters of the four-membered ring are in good agreement with the corresponding values found in seven similar three-ring derivatives in which planar five-membered rings are fused to a central cyclobutane ring, as retrieved from the Cambridge Structural Database Version 5.05 (Allen, Kennard & Taylor, 1983).

In view of the unexpected structure of compound (III), some further particular geometry checks seemed appropriate. The distances N3═C4 [1.276 (4) Å] and