C2—C11	1.528 (4)	C17—C29	1.504 (4)
C4—C5	1.461 (4)	C19—C20	1.503 (4)
C5—C10	1.384 (4)	C20—C25	1.377 (5)
C5—C6	1.397 (4)	C20—C21	1.384 (4)
C6—C7	1.371 (5)	C21—C22	1.374 (5)
C7—C8	1.375 (5)	C22—C23	1.360 (6)
C8—C9	1.366 (5)	C23—C24	1.362 (6)
C9—C10	1.398 (4)	C24—C25	1.370 (6)
C2—N1—C10	118.0 (2)	C27—C11—C2	109.5 (2)
C2—N3—C4	121.1 (2)	C18—C13—C14	119.6 (3)
C2—N3—C19	122.0 (2)	C18—C13—N12	122.4 (2)
C4—N3—C19	116.7 (2)	C14—C13—N12	117.9 (3)
C13—N12—C11	122.5 (2)	C15—C14—C13	117.6 (3)
N1—C2—N3	124.4 (3)	C15—C14—C28	121.1 (3)
N1—C2—C11	117.1 (2)	C13—C14—C28	121.4 (3)
N3—C2—C11	118.5 (2)	C16—C15—C14	122.7 (3)
O26—C4—N3	120.3 (3)	C15—C16—C17	120.4 (3)
O26—C4—C5	125.0 (3)	C16—C17—C18	118.2 (3)
N3—C4—C5	114.7 (3)	C16—C17—C29	121.0 (3)
C10—C5—C6	120.4 (3)	C18—C17—C29	120.8 (3)
C10—C5—C4	119.3 (3)	C17—C18—C13	121.4 (3)
C6—C5—C4	120.2 (3)	N3—C19—C20	115.5 (2)
C7—C6—C5	119.6 (3)	C25—C20—C21	118.4 (3)
C6—C7—C8	120.4 (3)	C25—C20—C19	120.2 (3)
C9—C8—C7	120.5 (3)	C21—C20—C19	121.3 (3)
C8—C9—C10	120.5 (3)	C22—C21—C20	120.5 (3)
C5—C10—N1	122.2 (3)	C23—C22—C21	120.6 (4)
C5—C10—C9	118.6 (3)	C22—C23—C24	119.2 (4)
N1—C10—C9	119.2 (3)	C23—C24—C25	121.2 (4)
N12—C11—C27	108.3 (2)	C24—C25—C20	120.1 (4)
N12—C11—C2	111.2 (2)		

For both compounds, data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1989); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1992). Program(s) used to solve structures: *SAPI*91 (Fan, 1991) for (I); *TEXSAN*, *SIR*88 (Burla *et al.*, 1989) for (II). For both compounds, program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); software used to prepare material for publication: *TEXSAN FINISH*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1084). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1,2-Bis-crown-5-calix[4]arene

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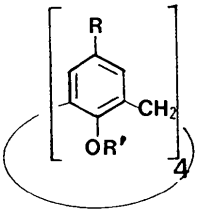
Abstract

The title compound, 13,16,19,22,25,39,42,45,48,51-decaoxahexacyclo[35.15.1.1^{11,27}.0^{5,52}.0^{7,12}.0^{26,31}.0^{33,38}]-tetrapentaconta-1(52),2,4,7(12),8,10,26(31),27,29,-33(38),34,36-dodecaene, $C_{44}H_{52}O_{10}$, is a potent and selective alkali metal carrier. Two half-independent molecules are observed in the solid state and they have the 'pinched-cone' conformation of the studied calixarenes.

Comment

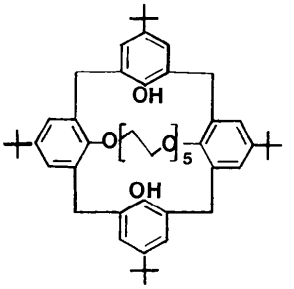
Calix[4]arenes (1) are cyclic oligomers made up from four phenol units which can be functionalized at either the 'upper rim', *R* (aromatic nuclei), or the 'lower rim', *R'* (phenolic OH groups).



(1)

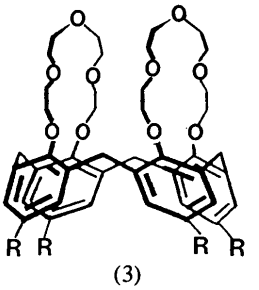
The corresponding calixarene podands, calix crowns and calix spherands, are neutral ligands which are interesting as host molecules and because of their ability to act as selective alkali metal receptors and carriers (Vicens & Bhömer, 1991).

The macrocycle (2) was the first reported 'crowned' calixarene which exhibits a 1,3-functionalization with a polyetheral chain linking two opposite O atoms of *p-tert*-butylcalix[4]arene (Alfieri, Dradi, Pochini, Ungaro & Andreetti, 1983).



(2)

The subsequent selective 1,2-functionalization of *p-tert*-butylcalix[4]arene by a Ti^{IV}-assisted reaction allowed the stepwise synthesis of a new type of calixarene-based bis-crown ether [(3) *R* = *tert*-butyl], a potentially highly preorganized molecular receptor (Arduini, Casnati, Dodi, Pochini & Ungaro, 1990).



(3)

An analogous product [(3) *R* = H], the title compound, was obtained by direct reaction of calix[4]arene with tetraethylene glycol ditosylate in the presence of caesium carbonate in acetonitrile in reflux at 9% yield (Asfari, Astier, Bressot, Estienne, Pèpe & Vicens, 1994). Its X-ray structure analysis was undertaken to determine the molecular conformation of the free ligand and to predict, using molecular-mechanics calculations (*GENMOL*; Pèpe & Siri, 1990), the conformations adopted by the corresponding alkali metal complexes.

The small number of observed reflections [1851 with $I > 3\sigma(I)$], compared to the large number of parameters to be refined in order to understand the behaviour of the molecule from analysis of anisotropic displacement parameters, led us to refine atomic coordinates (163) and anisotropic displacement parameters (325) separately. The final *R* value is 0.11, which is of the same order as values found for the determination of similar structures [0.15 reported by Atwood, Coleman, Zhang & Bott (1989), 0.14 by Iwamoto, Araki & Shinkai (1991) and 0.16 by Grootenhuis, Kollman, Grönen, Reinhoudt, van Hummel, Ugozzoli & Andreetti (1990)].

The asymmetric unit contains two half molecules related by pseudo-mirror symmetry (at the molecular level, see below) as displayed in Fig. 1. The complete molecules are generated by the binary axis of the *P2/c* space group. Fig. 2 shows the complete molecules in a projection along the *b* axis. The polyetheral chains form an open 'mouth' ready to 'swallow' the cations.

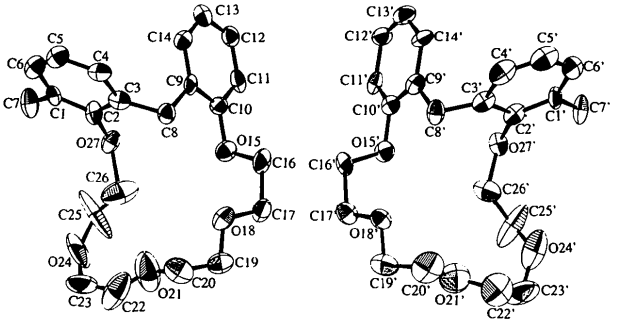


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of the two half-molecules *A* and *A'* in the asymmetric unit cell, with displacement ellipsoids at 50% probability.

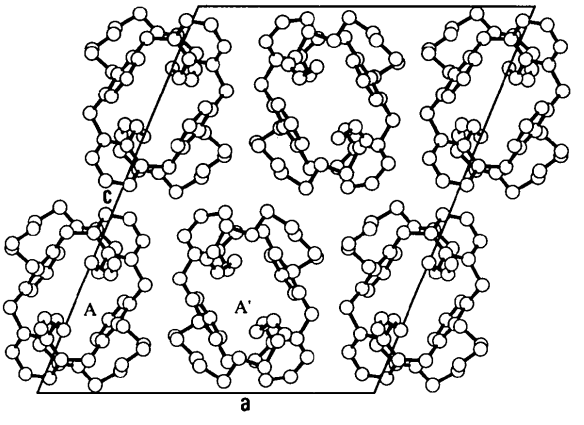


Fig. 2. Projection along the *b* axis of the two complete independent molecules.

Atoms C20–C26 have high displacement parameters (mean $B_{eq} \simeq 17.8$ Å^2) compared with atoms O15–C19. The two molecules have the 'pinched-cone' conformation. A best molecular fit performed on the complete molecule *A* and the complete mirrored molecule *A'* is 0.04 Å, which indicates a good geometrical agreement between the two observed molecules.

The angles between the phenyl rings (C1–C6 and C9–C14) and the plane through the methylene atoms C7, C8, $C7^i$ and $C8^i$ [symmetry code (i) is $-x, y, \frac{1}{2}-z$ for *A* and $-1-x, y, \frac{1}{2}-z$ for *A'*] are 140 (2) and 76 (2)° for molecule *A*, and 140 (2) and 80 (2)° for molecule *A'*, which are very similar. The 'mouth' aperture can be measured by the distances between the O atoms (Table 3) and by the angle between the plane passing through the O atoms O15, O18, O21, O24 and O27, and the plane through their symmetry equivalents [133 (2) and 110 (2)° for *A* and *A'*, respectively]. The parameters indicate that the slight geometrical differences between the two molecules result from the 'mouth' aperture, which is greater in *A* than in *A'*.

The packing analysis indicates alternate columns of molecules parallel to the *b* axis. The molecular contacts

in the columns and between the columns (between the polyetheral chains and the phenyl rings) are of van der Waals type. A pseudo-glide plane perpendicular to the *a* axis, with a translation parallel to the *b* axis, and almost equal to *b*/2, existing between the two independent half molecules, explains the observed lateral contacts.

Experimental

Crystal data

$C_{44}H_{52}O_{10}$
M_r = 740.89
Monoclinic
$P2/c$
a = 18.060 (2) Å
b = 10.716 (1) Å
c = 22.338 (2) Å
β = 112.89 (2)°
V = 3982.7 (9) Å^3
Z = 4
D_x = 1.24 Mg m^{-3}
D_m = 1.22 (2) Mg m^{-3}
D_m measured by flotation

Cu $K\alpha$ radiation
λ = 1.5416 Å
Cell parameters from 25 reflections
θ = 15.00–45.0°
μ = 0.67 mm^{-1}
T = 295 K
Square prism
0.4 × 0.3 × 0.3 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
θ scans
Absorption correction: none
7736 measured reflections
2901 independent reflections
1851 observed reflections [$I > 3.0\sigma(I)$]

R_{int} = 0.05
θ_{max} = 45°
$h = -15 \rightarrow 15$
$k = 0 \rightarrow 9$
$l = 0 \rightarrow 20$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F
R = 0.11
wR = 0.11
S = 1.64
1851 reflections
325 parameters
H atoms not located
Unit weights applied
$(\Delta/\sigma)_{max}$ = 0.35

$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.43 e Å^{-3}
Extinction correction: none
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)

$B_{eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
Molecule *A*				
C1	−0.1148 (8)	0.0629 (13)	0.3483 (7)	6.9 (10)
C2	−0.0387 (8)	0.0169 (14)	0.3599 (6)	6.5 (10)
C3	0.0309 (8)	0.0527 (12)	0.4147 (6)	6.2 (11)
C4	0.0178 (10)	0.1317 (13)	0.4609 (6)	7.4 (12)
C5	−0.0602 (10)	0.1729 (14)	0.4508 (7)	8.1 (12)
C6	−0.1269 (9)	0.1428 (15)	0.3932 (7)	7.7 (12)
C7	−0.1889 (8)	0.0407 (16)	0.2800 (6)	7.9 (13)
C8	0.1134 (7)	0.0187 (13)	0.4174 (6)	6.0 (10)
C9	0.1299 (6)	0.1008 (13)	0.3670 (6)	5.4 (12)
C10	0.1573 (7)	0.0429 (12)	0.3216 (6)	5.7 (11)
C11	0.1652 (7)	0.1126 (15)	0.2706 (7)	6.8 (12)
C12	0.1478 (7)	0.2409 (14)	0.2655 (7)	6.7 (11)
C13	0.1224 (8)	0.2999 (14)	0.3110 (7)	7.3 (11)
C14	0.1117 (7)	0.2279 (14)	0.3601 (6)	6.5 (11)
O15	0.1739 (5)	−0.0845 (9)	0.3289 (4)	7.1 (7)
C16	0.2582 (8)	−0.1109 (16)	0.3758 (8)	9.0 (16)
C17	0.2629 (9)	−0.2511 (15)	0.3852 (7)	8.6 (12)
O18	0.2178 (5)	−0.2768 (10)	0.4259 (5)	8.6 (8)
C19	0.2220 (11)	−0.4041 (16)	0.4435 (9)	10.5 (17)
C20	0.1678 (13)	−0.4272 (18)	0.4787 (8)	11.7 (18)
O21	0.0893 (11)	−0.4305 (20)	0.4259 (13)	24.0 (24)
C22	0.0374 (17)	−0.4884 (29)	0.4278 (12)	20.4 (31)
C23	−0.0433 (19)	−0.4710 (19)	0.3855 (17)	20.6 (38)
O24	−0.0719 (8)	−0.3745 (17)	0.3401 (8)	17.6 (17)
C25	−0.0575 (16)	−0.2710 (34)	0.3094 (14)	26.3 (37)
C26	−0.0012 (15)	−0.2072 (20)	0.3415 (10)	15.8 (24)
O27	−0.0308 (5)	−0.0645 (9)	0.3140 (4)	6.8 (7)
Molecule *A′*				
C1′	−0.2973 (7)	−0.3815 (14)	0.3588 (7)	6.8 (13)
C2′	−0.3631 (8)	−0.4285 (13)	0.3691 (6)	6.5 (12)
C3′	−0.3925 (8)	−0.3937 (13)	0.4164 (6)	6.6 (12)
C4′	−0.3370 (10)	−0.3099 (17)	0.4660 (7)	9.2 (15)
C5′	−0.2670 (11)	−0.2707 (16)	0.4608 (8)	10.7 (17)
C6′	−0.2444 (8)	−0.2969 (15)	0.4077 (7)	8.7 (15)
C7′	−0.2821 (7)	−0.4137 (16)	0.2976 (6)	7.7 (14)
C8′	−0.4724 (8)	−0.4237 (14)	0.4188 (6)	7.3 (11)
C9′	−0.5343 (8)	−0.3462 (13)	0.3620 (6)	5.8 (10)
C10′	−0.5985 (8)	−0.4053 (12)	0.3130 (6)	5.5 (10)
C11′	−0.6530 (7)	−0.3428 (14)	0.2580 (6)	6.2 (11)
C12′	−0.6428 (9)	−0.2111 (14)	0.2536 (7)	7.2 (12)
C13′	−0.5795 (10)	−0.1487 (13)	0.3060 (7)	7.5 (12)
C14′	−0.5240 (8)	−0.2163 (13)	0.3591 (7)	6.6 (11)
O15′	−0.6073 (5)	−0.5355 (9)	0.3168 (4)	6.8 (6)
C16′	−0.6585 (8)	−0.5701 (14)	0.3524 (7)	7.6 (10)
C17′	−0.6488 (9)	−0.7101 (15)	0.3634 (7)	7.9 (12)
O18′	−0.5675 (6)	−0.7270 (9)	0.4112 (5)	7.7 (7)
C19′	−0.5527 (12)	−0.8561 (17)	0.4270 (9)	11.7 (18)
C20′	−0.4614 (14)	−0.8629 (22)	0.4669 (9)	14.6 (22)
O21′	−0.4438 (10)	−0.8934 (15)	0.4086 (9)	18.1 (18)
C22′	−0.3603 (14)	−0.9247 (24)	0.4350 (10)	15.6 (25)
C23′	−0.3478 (18)	−0.9339 (21)	0.3721 (15)	19.3 (30)
O24′	−0.3391 (9)	−0.8114 (20)	0.3458 (7)	18.5 (19)
C25′	−0.3935 (13)	−0.7425 (27)	0.3093 (9)	17.6 (27)
C26′	−0.4222 (11)	−0.6432 (15)	0.3422 (7)	10.1 (15)
O27′	−0.4131 (5)	−0.5120 (9)	0.3208 (3)	6.4 (7)

Table 2. *Selected geometric parameters* (Å, °)

C1—C2	1.386 (21)	C1′—C2′	1.390 (22)
C1—C6	1.399 (24)	C1′—C6′	1.455 (19)
C1—C7	1.608 (16)	C1′—C7′	1.535 (22)
C2—C3	1.424 (18)	C2′—C3′	1.405 (22)
C2—O27	1.395 (14)	C2′—O27′	1.422 (14)
C3—C4	1.424 (21)	C3′—C4′	1.475 (19)
C3—C8	1.512 (20)	C3′—C8′	1.500 (22)
C4—C5	1.408 (25)	C4′—C5′	1.379 (29)
C5—C6	1.416 (18)	C5′—C6′	1.424 (27)
C8—C9	1.546 (21)	C8′—C9′	1.564 (17)
C9—C14	1.396 (20)	C9′—C14′	1.409 (20)
C10—C11	1.415 (22)	C10′—C11′	1.409 (16)
C10—O15	1.394 (16)	C10′—O15′	1.411 (16)
C11—C12	1.405 (22)	C11′—C12′	1.432 (21)
C12—C13	1.416 (24)	C12′—C13′	1.443 (19)
C13—C14	1.414 (22)	C13′—C14′	1.418 (18)
O15—C16	1.502 (15)	C16′—O15′	1.482 (20)
C16—C17	1.515 (23)	C16′—C17′	1.519 (22)
C17—O18	1.464 (22)	C17′—O18′	1.452 (16)
O18—C19	1.414 (20)	C19′—O18′	1.427 (21)
C19—C20	1.495 (34)	C19′—C20′	1.541 (29)
C20—O21	1.450 (25)	C20′—O21′	1.492 (33)
C22—O21	1.139 (40)	C22′—O21′	1.429 (29)
C22—C23	1.407 (38)	C22′—C23′	1.510 (45)
C23—O24	1.399 (32)	C23′—O24′	1.471 (34)
O24—C25	1.381 (41)	O24′—C25′	1.246 (28)
C25—C26	1.203 (36)	C25′—C26′	1.496 (33)
C26—O27	1.657 (23)	C26′—O27′	1.514 (19)

C2—C1—C7	121.6 (11)	C2′—C1′—C7′	121.8 (11)
C6—C1—C7	117.8 (11)	C6′—C1′—C7′	121.1 (11)
C1—C2—C3	123.0 (11)	C1′—C2′—C3′	128.8 (11)
C1—C2—O27	117.5 (11)	C1′—C2′—O27′	115.7 (10)
C3—C2—O27	119.5 (10)	C3′—C2′—O27′	115.0 (10)
C2—C3—C4	116.3 (11)	C2′—C3′—C4′	112.4 (11)
C2—C3—C8	119.7 (10)	C2′—C3′—C8′	128.3 (11)
C4—C3—C8	123.6 (10)	C4′—C3′—C8′	119.1 (11)
C3—C4—C5	120.5 (12)	C3′—C4′—C5′	120.0 (13)
C4—C5—C6	121.2 (12)	C4′—C5′—C6′	125.4 (14)
C1—C6—C5	118.4 (12)	C1′—C6′—C5′	115.5 (12)
C3—C8—C9	107.4 (9)	C3′—C8′—C9′	104.6 (10)
C10—C9—C8	119.1 (9)	C10′—C9′—C8′	120.5 (10)
C10—C9—C14	118.4 (10)	C10′—C9′—C14′	119.7 (11)
C14—C9—C8	122.1 (10)	C14′—C9′—C8′	119.8 (10)
C11—C10—C9	120.8 (10)	C11′—C10′—C9′	123.5 (11)
C11—C10—O15	121.8 (10)	C11′—C10′—O15′	117.8 (10)
C9—C10—O15	117.4 (9)	C9′—C10′—O15′	118.6 (10)
C10—C11—C12	119.5 (11)	C10′—C11′—C12′	117.6 (11)
C11—C12—C13	120.2 (11)	C11′—C12′—C13′	118.9 (12)
C12—C13—C14	119.5 (11)	C12′—C13′—C14′	121.4 (12)
C13—C14—C9	121.4 (11)	C13′—C14′—C9′	118.7 (11)
C10—O15—C16	112.3 (9)	C10′—O15′—C16′	112.8 (9)
C17—C16—O15	105.7 (10)	C17′—C16′—O15′	106.1 (10)
C16—C17—O18	105.0 (10)	C16′—C17′—O18′	105.2 (10)
C17—O18—C19	111.8 (10)	C17′—O18′—C19′	110.0 (10)
C20—C19—O18	109.3 (13)	C20′—C19′—O18′	104.1 (13)
C19—C20—O21	102.1 (14)	C19′—C20′—O21′	92.8 (14)
C20—O21—C22	122.8 (21)	C20′—O21′—C22′	103.9 (15)
C23—C22—O21	123.7 (25)	C23′—C22′—O21′	98.5 (17)
C22—C23—O24	125.4 (22)	C22′—C23′—O24′	113.0 (19)
C26—C25—O24	116.0 (24)	C26′—C25′—O24′	115.8 (18)
C25—C26—O27	103.1 (19)	C25′—C26′—O27′	113.9 (13)
C23—O24—C25	150.0 (19)	C23′—O24′—C25′	127.6 (18)
C2—O27—C26	114.5 (10)	C2′—O27′—C26′	117.4 (9)

Table 3. *Distances* (Å) *between* O *atoms of the etheral chains characterizing the aperture of the crowned calixarene*

	A	*A′*
O15···O27	3.24 (4)	3.25 (4)
O18···O24^{i}	5.58 (6)	5.39 (6)
O21···O21^{i}	7.24 (8)	6.57 (8)

Symmetry code: (i) indicates the equivalent atom generated by the twofold axis.

As the crystals of the studied calixarene were of poor quality, half the reciprocal space was measured and averaged values were calculated to increase the measurement accuracy.

Data collection: *Structure Determination Package* (Enraf–Nonius, 1979). Cell refinement: *Structure Determination Package*. Data reduction: local program (CRMC2, France). Program(s) used to solve structure: *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *ORTEP*II (Johnson, 1976) and *GENMOL* (Pèpe & Siri, 1990).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PA1099). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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3-Oxo-2-phenyl-3a,4,5,7a-tetrahydro-5,7a-epoxyisoindoline-4-carboxylic Acid in Two Crystalline Phases, Solvated with DMSO and Unsolvated

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Abstract

The title compound is a precursor of optically active γ-lactams. Two crystalline forms, solvated with DMSO ($C_{15}H_{13}NO_4.C_2H_6OS$) and unsolvated ($C_{15}H_{13}NO_4$), were found and analyzed.

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