C14	0.4471 (2)	0.3796 (3)	0.27334 (7)	0.0388 (4)
C15	0.4104 (3)	0.4873 (4)	0.33683 (9)	0.0479 (5)
C16	0.2348 (3)	0.3800 (4)	0.36391 (7)	0.0449 (5)
C17	0.2137 (2)	0.1992 (3)	0.32745 (8)	0.0452 (5)
C18	0.5568 (3)	0.0840 (4)	0.33795 (10)	0.0525 (6)
C19	1.3937 (4)	0.2395 (5)	−0.01082 (12)	0.0689 (8)
C20	0.1855 (3)	0.3681 (4)	0.43600 (8)	0.0548 (6)
C21	0.2735 (4)	0.5146 (5)	0.48256 (10)	0.0734 (9)
C22	0.2168 (4)	0.7029 (5)	0.45177 (11)	0.0727 (8)
C23	0.0350 (4)	0.6674 (5)	0.40359 (12)	0.0698 (8)
C24	0.0411 (3)	0.4619 (4)	0.38802 (8)	0.0516 (5)

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

O3—C3	1.374 (2)	C11—C12	1.542 (3)
O3—C19	1.411 (3)	C12—C13	1.520 (3)
O17—C17	1.216 (2)	C13—C14	1.534 (3)
C1—C2	1.392 (3)	C13—C17	1.517 (2)
C1—C10	1.392 (3)	C13—C18	1.544 (3)
C2—C3	1.375 (3)	C14—C15	1.538 (3)
C3—C4	1.388 (3)	C15—C16	1.529 (3)
C4—C5	1.395 (3)	C16—C17	1.494 (3)
C5—C6	1.516 (3)	C16—C20	1.527 (2)
C5—C10	1.403 (3)	C16—C24	1.524 (3)
C6—C7	1.525 (3)	C20—C21	1.508 (4)
C7—C8	1.527 (3)	C20—C24	1.488 (3)
C8—C9	1.542 (2)	C21—C22	1.524 (5)
C8—C14	1.5211 (19)	C22—C23	1.532 (4)
C9—C10	1.521 (2)	C23—C24	1.505 (5)
C9—C11	1.537 (3)		
C3—O3—C19	117.51 (16)	C14—C13—C17	100.48 (15)
C2—C1—C10	123.0 (2)	C14—C13—C18	113.44 (15)
C1—C2—C3	118.7 (2)	C17—C13—C18	104.76 (14)
O3—C3—C2	124.29 (16)	C8—C14—C13	112.14 (15)
O3—C3—C4	115.94 (17)	C8—C14—C15	120.51 (15)
C2—C3—C4	119.77 (18)	C13—C14—C15	105.00 (15)
C3—C4—C5	121.41 (19)	C14—C15—C16	102.47 (18)
C4—C5—C6	118.31 (18)	C15—C16—C17	107.82 (16)
C4—C5—C10	119.59 (17)	C15—C16—C20	126.07 (18)
C6—C5—C10	122.04 (16)	C15—C16—C24	127.1 (2)
C5—C6—C7	113.36 (17)	C17—C16—C20	114.3 (2)
C6—C7—C8	110.83 (16)	C17—C16—C24	115.94 (18)
C7—C8—C9	108.83 (14)	C20—C16—C24	58.40 (13)
C7—C8—C14	113.70 (16)	O17—C17—C13	126.51 (19)
C9—C8—C14	108.02 (14)	O17—C17—C16	125.96 (16)
C8—C9—C10	111.13 (15)	C13—C17—C16	107.52 (15)
C8—C9—C11	112.17 (15)	C16—C20—C21	118.0 (2)
C10—C9—C11	113.47 (17)	C16—C20—C24	60.70 (13)
C1—C10—C5	117.46 (15)	C21—C20—C24	108.1 (2)
C1—C10—C9	121.43 (18)	C20—C21—C22	106.16 (19)
C5—C10—C9	121.05 (16)	C21—C22—C23	106.5 (3)
C9—C11—C12	113.20 (18)	C22—C23—C24	105.5 (2)
C11—C12—C13	109.91 (17)	C16—C24—C20	60.90 (13)
C12—C13—C14	109.41 (15)	C16—C24—C23	118.5 (2)
C12—C13—C17	117.06 (14)	C20—C24—C23	109.05 (19)
C12—C13—C18	111.34 (18)		

The crystal used for the structure determination was unusually large, but by using an Ni β-filter instead of a monochromator on the diffractometer it was ensured that the homogeneous part of the incident X-ray beam was large enough to surround the crystal completely.

Data collection: *ARGUS* (Schreurs & Duisenberg, unpublished). Cell refinement: *SET*4 (de Boer & Duisenberg, 1984). Data reduction: *HELENA* (Spek, 1993). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *PLATON*93 (Spek, 1990). User interface software: *S* (Spek, 1994) .

We would like to thank Organon International BV for supplying the compound.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, including H-atom geometry, and torsion angles have been deposited with the IUCr (Reference: AB1170). 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. (1995). C**51**, 723–726

Two Antithrombotic Quinazolone Derivatives

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(*Received* 23 *February* 1994; *accepted* 11 *April* 1994)

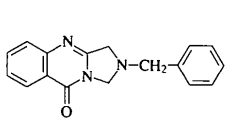
Abstract

The structures of two antithrombotic quinazolone derivatives, 1,2,3,5-tetrahydro-2-benzylimidazo[5,1-*b*]-quinazolin-5-one, (I), and 3-benzyl-2-[1-(2,5-xylidino)-

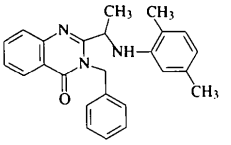
ethyl]quinazolin-4(3*H*)-one, (II), display significant differences in the bond lengths in one region of the quinazolone moiety.

Comment

The antithrombotic compounds (I) and (II) have been structurally characterized.



(I)



(II)

Compound (I) contains a new linear tricycle which has not been characterized previously, while compound (II) can be described as a substituted ring-opened analogue of (I). We have tried to consider the conformational consequences of the ring opening and the susbtitution. We can state clearly that there is a significant shortening of the bond lengths N6—C2, N6—C5 and N6—C7 in the tricyclic compound (I) as compared to the corresponding bond lengths (N3—C2, N3—C19 and N3—C4) of compound (II), which is probably a result of the ring closure in an open envelope conformation. N4 is 0.536 (2) Å above the plane of the remaining atoms of the tricycle. It is worth mentioning that no hydrogen bond was detected in the crystal structure of (II) although there are both potential hydrogen-bond acceptors and one potential hydrogen-bond donor in the molecule. This may be attributed to the N—H bond being sterically hindered by large neighbouring groups.

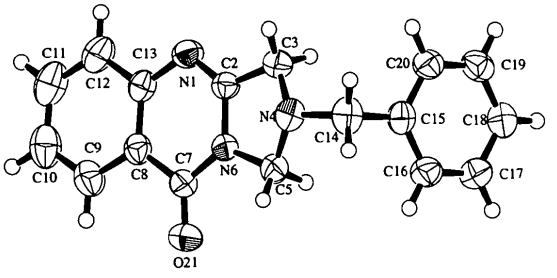


Fig. 1. Structure of (I) showing 50% probability displacement ellipsoids.

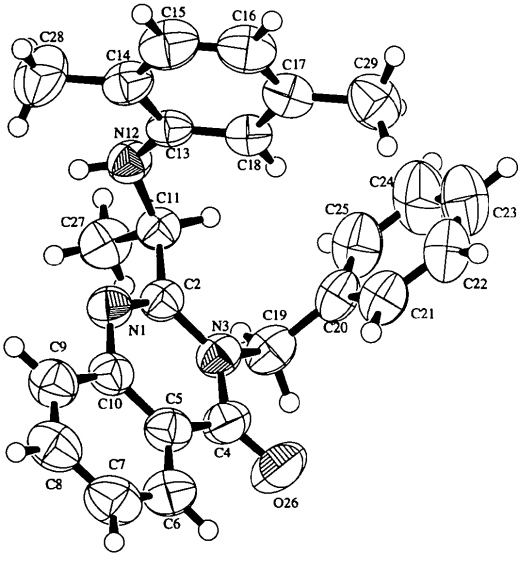


Fig. 2. Structure of (II) showing 50% probability displacement ellipsoids.

Experimental

Compounds (I) and (II) were synthesized by the methods described by Örfi, Kökösi, Hermecz, Kapui, Szabó & Szśz (1993).

Compound (I)

Crystal data

$C_{17}H_{15}N_3O$
M_r = 277.33
Monoclinic
$P2_1/c$
a = 7.423 (1) Å
b = 20.540 (1) Å
c = 9.1829 (7) Å
β = 101.448 (8)°
V = 1372.3 (3) Å^3
Z = 4
D_x = 1.342 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 25 reflections
θ = 48.08–49.88°
μ = 0.688 mm^{-1}
T = 293 (2) K
Cubic
0.60 × 0.55 × 0.40 mm
Brownish

Data collection

AFC-6S diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2959 measured reflections
2759 independent reflections
2209 observed reflections [$I > 2\sigma(I)$]
R_{int} = 0.0159
θ_{max} = 75.08°
$h = -8 \rightarrow 9$
$k = -13 \rightarrow 25$
$l = -11 \rightarrow 11$
3 standard reflections monitored every 150 reflections
intensity decay: 1.73%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.0530
$wR(F^2)$ = 0.1454
S = 1.167
2755 reflections
193 parameters
Only H-atom U's refined
$w = 1/[\sigma^2(F_o^2) + (0.0711P)^2 + 0.4583P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = −0.002
$\Delta\rho_{max}$ = 0.272 e Å^{-3}
$\Delta\rho_{min}$ = −0.293 e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0177 (13)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

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

	x	y	z	U_{eq}
O21	0.2943 (3)	0.35395 (8)	0.3598 (2)	0.0755 (5)
N1	0.2728 (3)	0.51119 (8)	0.6324 (2)	0.0552 (4)
N4	0.2911 (2)	0.35880 (8)	0.8116 (2)	0.0539 (4)
N6	0.3062 (2)	0.39911 (7)	0.5868 (2)	0.0477 (4)
C2	0.3017 (3)	0.45209 (9)	0.6770 (2)	0.0470 (4)
C3	0.3354 (3)	0.42810 (10)	0.8331 (2)	0.0549 (5)
C5	0.3427 (3)	0.33938 (9)	0.6740 (2)	0.0549 (5)
C7	0.2833 (3)	0.40249 (10)	0.4345 (2)	0.0516 (5)
C8	0.2428 (3)	0.46858 (10)	0.3788 (2)	0.0504 (5)
C9	0.2048 (3)	0.48062 (13)	0.2262 (3)	0.0653 (6)
C10	0.1660 (3)	0.54229 (14)	0.1731 (3)	0.0743 (7)
C11	0.1673 (3)	0.59358 (13)	0.2724 (3)	0.0745 (7)
C12	0.2053 (3)	0.58303 (11)	0.4227 (3)	0.0677 (6)
C13	0.2416 (3)	0.52003 (10)	0.4792 (2)	0.0512 (5)
C14	0.3497 (3)	0.31534 (11)	0.9388 (3)	0.0607 (6)
C15	0.5528 (3)	0.31133 (9)	1.0005 (2)	0.0516 (5)
C16	0.6631 (3)	0.26575 (10)	0.9477 (2)	0.0590 (5)
C17	0.8493 (3)	0.26273 (11)	1.0034 (3)	0.0632 (6)
C18	0.9292 (3)	0.30474 (11)	1.1144 (3)	0.0608 (6)
C19	0.8230 (3)	0.35064 (10)	1.1682 (2)	0.0594 (6)
C20	0.6367 (3)	0.35366 (10)	1.1124 (2)	0.0561 (5)

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

O21—C7	1.223 (2)	C8—C13	1.404 (3)
N1—C2	1.286 (2)	C9—C10	1.368 (3)
N1—C13	1.391 (3)	C10—C11	1.392 (4)
N4—C5	1.447 (3)	C11—C12	1.370 (4)
N4—C3	1.465 (3)	C12—C13	1.400 (3)
N4—C14	1.466 (3)	C14—C15	1.504 (3)
N6—C2	1.372 (2)	C15—C16	1.392 (3)
N6—C7	1.377 (3)	C15—C20	1.395 (3)
N6—C5	1.460 (2)	C16—C17	1.376 (3)
C2—C3	1.489 (3)	C17—C18	1.377 (3)
C7—C8	1.461 (3)	C18—C19	1.382 (3)
C8—C9	1.396 (3)	C19—C20	1.378 (3)
C2—N1—C13	115.4 (2)	C13—C8—C7	119.8 (2)
C5—N4—C3	107.1 (2)	C10—C9—C8	120.6 (2)
C5—N4—C14	116.1 (2)	C9—C10—C11	119.6 (2)
C3—N4—C14	117.4 (2)	C12—C11—C10	120.9 (2)
C2—N6—C7	124.1 (2)	C11—C12—C13	120.3 (2)
C2—N6—C5	111.1 (2)	N1—C13—C12	118.4 (2)
C7—N6—C5	124.8 (2)	N1—C13—C8	123.0 (2)
N1—C2—N6	125.4 (2)	C12—C13—C8	118.6 (2)
N1—C2—C3	127.2 (2)	N4—C14—C15	116.8 (2)
N6—C2—C3	107.4 (2)	C16—C15—C20	117.9 (2)
N4—C3—C2	101.6 (2)	C16—C15—C14	121.4 (2)
N4—C5—N6	101.21 (15)	C20—C15—C14	120.7 (2)
O21—C7—N6	121.4 (2)	C17—C16—C15	121.2 (2)
O21—C7—C8	126.4 (2)	C16—C17—C18	120.0 (2)
N6—C7—C8	112.2 (2)	C17—C18—C19	120.0 (2)
C9—C8—C13	120.0 (2)	C20—C19—C18	120.0 (2)
C9—C8—C7	120.2 (2)	C19—C20—C15	120.9 (2)

Compound (II)

Crystal data

$C_{25}H_{25}N_3O$
M_r = 383.48
Monoclinic
$C2/c$
a = 19.053 (6) Å
b = 11.451 (3) Å
c = 19.309 (3) Å
β = 96.62 (2)°
V = 4184.6 (19) Å^3
Z = 8
D_x = 1.217 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 25 reflections
θ = 44.07–48.23°
μ = 0.590 mm^{-1}
T = 293 (2) K
Octahedral
0.50 × 0.20 × 0.20 mm
Pinkish

Data collection

AFC-6*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: none
4257 measured reflections
4130 independent reflections
2367 observed reflections [$I > 2\sigma(I)$]
R_{int} = 0.1021
θ_{max} = 75.38°
$h = -10 \rightarrow 23$
$k = -8 \rightarrow 14$
$l = -23 \rightarrow 22$
3 standard reflections monitored every 150 reflections
intensity decay: 2.24%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.0578
$wR(F^2)$ = 0.1641
S = 1.123
4124 reflections
274 parameters
Only H-atom U's refined
$w = 1/[\sigma^2(F_o^2) + (0.0892P)^2 + 3.3572P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.047
$\Delta\rho_{max}$ = 0.208 e Å^{-3}
$\Delta\rho_{min}$ = −0.206 e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0019 (2)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (II)

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

	x	y	z	U_{eq}
O26	0.73376 (13)	−0.2050 (2)	0.12354 (14)	0.1015 (9)
N1	0.89828 (12)	0.0228 (2)	0.14793 (12)	0.0587 (6)
N3	0.78907 (12)	−0.0482 (2)	0.17795 (11)	0.0601 (6)
N12	0.87928 (14)	0.2333 (2)	0.21058 (12)	0.0604 (6)
C2	0.84495 (14)	0.0301 (2)	0.18315 (13)	0.0552 (6)
C4	0.7849 (2)	−0.1399 (3)	0.1296 (2)	0.0708 (8)
C5	0.8449 (2)	−0.1481 (3)	0.08900 (15)	0.0654 (7)
C6	0.8472 (2)	−0.2364 (3)	0.0394 (2)	0.0866 (10)
C7	0.9046 (2)	−0.2448 (3)	0.0029 (2)	0.0897 (11)
C8	0.9600 (2)	−0.1678 (3)	0.0155 (2)	0.0846 (10)
C9	0.9580 (2)	−0.0808 (3)	0.0637 (2)	0.0735 (8)
C10	0.89979 (15)	−0.0689 (2)	0.10108 (14)	0.0597 (7)
C11	0.84485 (15)	0.1308 (2)	0.23506 (13)	0.0573 (7)
C13	0.85167 (14)	0.2954 (2)	0.15089 (13)	0.0524 (6)
C14	0.8978 (2)	0.3671 (3)	0.1185 (2)	0.0676 (8)
C15	0.8691 (2)	0.4367 (3)	0.0641 (2)	0.0857 (10)
C16	0.7988 (2)	0.4375 (3)	0.0412 (2)	0.0832 (10)
C17	0.7528 (2)	0.3648 (3)	0.07132 (14)	0.0657 (8)
C18	0.78011 (15)	0.2938 (2)	0.12613 (13)	0.0565 (7)
C19	0.7341 (2)	−0.0448 (3)	0.22612 (15)	0.0698 (8)
C20	0.67592 (15)	0.0425 (3)	0.20823 (15)	0.0666 (8)
C21	0.6423 (2)	0.0525 (3)	0.1410 (2)	0.0771 (9)
C22	0.5877 (2)	0.1303 (4)	0.1261 (2)	0.0944 (11)
C23	0.5667 (2)	0.2000 (5)	0.1769 (3)	0.1130 (15)
C24	0.5996 (2)	0.1905 (5)	0.2431 (3)	0.122 (2)
C25	0.6536 (2)	0.1126 (4)	0.2593 (2)	0.0952 (12)
C27	0.8846 (2)	0.0940 (3)	0.3049 (2)	0.0790 (9)
C28	0.9755 (2)	0.3695 (4)	0.1425 (2)	0.1049 (13)
C29	0.6751 (2)	0.3640 (3)	0.0461 (2)	0.0898 (11)

Table 4. *Selected geometric parameters* (Å, °) *for* (II)

O26—C4	1.221 (3)	C11—C27	1.527 (4)
N1—C2	1.289 (3)	C13—C18	1.392 (4)
N1—C10	1.389 (4)	C13—C14	1.403 (4)
N3—C2	1.387 (3)	C14—C15	1.380 (5)
N3—C4	1.401 (4)	C14—C28	1.499 (5)
N3—C19	1.480 (4)	C15—C16	1.361 (5)
N12—C13	1.404 (3)	C16—C17	1.384 (4)
N12—C11	1.451 (3)	C17—C18	1.387 (4)

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*.

This work was financed from the grant OTKA No. 1009. Thanks are also due to Chinoin Pharmaceuticals for supporting the project with manpower and instrumentation.

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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Acta Cryst. (1995). C**51**, 726–729

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

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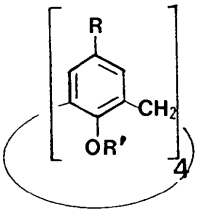
(*Received* 1 *December* 1993; *accepted* 1 *September* 1994)

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).