Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,4-Dioxo-1,5-benzodiazepino-15-crown-3

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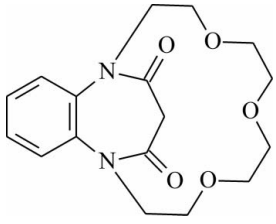
Received 7 June 2007; accepted 18 June 2007

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.049; wR factor = 0.150; data-to-parameter ratio = 19.6.

The title compound (systematic name: 4,7,10-trioxa-1,13-diazatricyclo[11.6.3.0^{14,19}]icosa-14,16,18-triene-20,22-dione), $C_{17}H_{22}N_2O_5$, is a macrocycle containing two roughly perpendicular parts, *viz*. the benzo-fused macrocycle and the dicarbonyl spacer. The carbonyl groups point out of the cavity, far away from the O atoms of the macrocycle, and so cannot be involved in coordination to a metal ion.

Related literature

For related literature, see: Allen (2002); Bürger & Seebach (1994); Basak & Shain (1998); Bourgoin *et al.* (1975); Chang *et al.* (1986); Costero & Rodriguez (1992); Cram & Ho (1986); Dietrich *et al.* (1993); Izatt *et al.* (1987, 1991, 1995); Keïta *et al.* (2003); Lazrak *et al.* (2004); Liotta & Harris (1974); Pedersen (1967); Rothermel *et al.* (1992); Sam & Simmons (1972); Takaki *et al.* (1972); Veggel *et al.* (1991).



Experimental

Crystal data

$C_{17}H_{22}N_2O_5$
M_r = 334.37
Monoclinic, $P2_1/n$
a = 11.786 (3) Å
b = 8.092 (2) Å
c = 18.062 (5) Å
β = 103.692 (17)°
V = 1673.8 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 296 (2) K
0.25 × 0.19 × 0.05 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.950, T_{max} = 0.994
58268 measured reflections
4249 independent reflections
2847 reflections with $I > 2\sigma(I)$
R_{int} = 0.056

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.151
S = 1.02
4249 reflections
217 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.63 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

The 'Fonds Européen de Développement Régional (FEDER)', 'CNRS', 'Région Nord Pas-de-Calais' and 'Ministère de l'Education Nationale de l'Enseignement Supérieur et de la Recherche' are acknowledged for their financial supports of different X-ray diffractometers.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2198).

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  doi:10.1107/S1600536807029753

supplementary materials

Acta Cryst. (2007). E**63**, o3494 [**doi:10.1107/S1600536807029753**]

2,4-Dioxo-1,5-benzodiazepino-15-crown-3

L. Cherif Alaoui, Y. Kandri Rodi, A. Haoudi, S. Obbade and E. M. Essassi

Comment

Since the pioneering work of Pederson (Pedersen, 1967), extensive research has been devoted to the preparation and study of the macrocyclic polyethers properties. Several types of ligands have been synthesized to enhance the stability of the cation-ligand complex and to achieve better selectivity (Izatt *et al.*, 1991; Izatt *et al.*, 1995; Veggel *et al.*, 1991; Rothermel *et al.* 1992; Basak *et al.*, 1998). These last years, research was focused on the synthesis of macrocycles being able to have potential applications in different fields such as the ionic and molecular recognition (Dietrich *et al.*, 1991), chemical analysis (Cram & Ho, 1986), the extraction and the metal elements transport through pecific membranes (Izatt *et al.*, 1987; Costero & Rodriguez, 1992; Chang *et al.*, 1986, Bürger & Seebach, 1994). In addition, from a reactional point of view, this type of compound is used as well in the supramolecular catalysis (Sam & Simmons, 1972; Liotta & Harris, 1974) or in the separation of the pairs of ions while behaving as base (Takaki *et al.*, 1972; Bourgoin *et al.*, 1975). In this context, we prepared the 2,4-dioxo-1,5 benzodizepino-15-crown-3, obtained by condensation of the dichlorotetraethlene-glycol with the 1,5-benzodiazepine-2,4-dione by phase transfer catalysis conditions (Keïta *et al.*, 2003; Lazrak *et al.*, 2004)using dimethylformamide as solvent. (I).

The molecular structure of (I) is built up from a benzodiazepine fragment and a crown ether as a a spacer (Fig. 1). The bond lengths and angles are within the expected range for similar structures deposited in the Cambridge Structural Database, Version 5.27, 2006(Allen, 2002). The crystal structure is stabilized by Van der Waals forces.

Experimental

With a solution of 11.10–3 mole of the 1,5-benzodiazepine-2,4-dione in 60 ml of dimethylformamide, one adds 33. 10–3 mole of potassium carbonate, 11. 10–3 mole of the di-chloro tetraethylene glycol and 6 10–3 mole of tetra-n-butylammonium bromide. Under agitation, the mixture is heated at a temperature between 80 and 90°C during 24 h. After filtration of salts, the filtrate is concentrated under reduced pressure (1.10–2 m mH g). The compound is purified by silica gel column chromatography (eluant/chloroform/methanol: 95/5).

Refinement

All H atoms were fixed geometrically and treated as riding with C—H = 0.93 Å (aromatic) or 0.97 Å (methylene) with = U_{iso}(H) = 1.2U_{eq}(C).

Figures

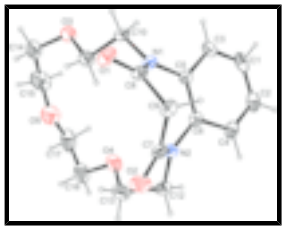


Fig. 1. : Molecular view of the title compound with the atom labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii

4,7,10-Trioxa-1,13-diazatricyclo[11.6.3.0^{14,19}]icosa-14,16,18-triene- 20,22-dione

Crystal data

$C_{17}H_{22}N_2O_5$	F_{000} = 712
M_r = 334.37	D_x = 1.327 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 8091 reflections
a = 11.786 (3) Å	θ = 2.3–22.2º
b = 8.092 (2) Å	μ = 0.10 mm^{-1}
c = 18.062 (5) Å	T = 296 (2) K
β = 103.692 (17)º	Block, colourless
V = 1673.8 (8) Å^3	0.25 × 0.19 × 0.05 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	4249 independent reflections
Radiation source: fine-focus sealed tube	2847 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.056
T = 296(2) K	θ_{max} = 28.6º
φ and ω scans	θ_{min} = 2.3º
Absorption correction: multi-scan (SADABS; Bruker, 1998)	h = −15→15
T_{min} = 0.950, T_{max} = 0.994	k = −10→10
58268 measured reflections	l = −24→24

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.049	H-atom parameters constrained
$wR(F^2)$ = 0.151	$w = 1/[\sigma^2(F_o^2) + (0.0687P)^2 + 0.6508P]$ where $P = (F_o^2 + 2F_c^2)/3$

S = 1.02	$(\Delta/\sigma)_{max} < 0.001$
4249 reflections	$\Delta\rho_{max} = 0.63$ e Å^{-3}
217 parameters	$\Delta\rho_{min} = -0.23$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.20967 (11)	0.18321 (16)	0.97933 (8)	0.0311 (3)
N2	0.30717 (12)	0.48339 (16)	0.93351 (8)	0.0346 (3)
O1	0.33317 (11)	−0.01059 (14)	0.95379 (9)	0.0491 (4)
O2	0.46850 (13)	0.39526 (18)	0.89713 (9)	0.0593 (4)
O3	−0.03654 (12)	0.00746 (18)	0.84001 (8)	0.0523 (4)
O4	0.11484 (14)	0.5203 (2)	0.78801 (8)	0.0608 (4)
O5	−0.02232 (14)	0.2477 (2)	0.71616 (9)	0.0708 (5)
C1	0.10071 (17)	0.4975 (3)	1.09477 (11)	0.0501 (5)
H1	0.0511	0.5030	1.1278	0.060*
C2	0.15493 (17)	0.6394 (3)	1.07702 (11)	0.0494 (5)
H2	0.1443	0.7393	1.0999	0.059*
C3	0.12066 (15)	0.3484 (2)	1.06322 (9)	0.0396 (4)
H3	0.0875	0.2525	1.0772	0.047*
C4	0.22434 (16)	0.6323 (2)	1.02571 (10)	0.0420 (4)
H4	0.2614	0.7276	1.0148	0.050*
C5	0.19048 (13)	0.3397 (2)	1.01025 (9)	0.0304 (3)
C6	0.24003 (14)	0.4840 (2)	0.98966 (9)	0.0322 (3)
C7	0.40197 (14)	0.3828 (2)	0.93951 (10)	0.0375 (4)
C8	0.31806 (14)	0.12811 (19)	0.97673 (10)	0.0333 (4)
C9	0.41718 (13)	0.2502 (2)	0.99990 (11)	0.0374 (4)
H9A	0.4161	0.2991	1.0488	0.045*
H9B	0.4914	0.1946	1.0046	0.045*
C10	0.10919 (14)	0.0749 (2)	0.94951 (10)	0.0355 (4)
H10A	0.0443	0.1072	0.9709	0.043*
H10B	0.1296	−0.0384	0.9645	0.043*
C11	0.07348 (16)	0.0865 (3)	0.86399 (11)	0.0474 (5)
H11A	0.0676	0.2013	0.8481	0.057*

H11B	0.1308	0.0324	0.8416	0.057*
C12	0.28953 (19)	0.6158 (2)	0.87579 (11)	0.0466 (5)
H12A	0.3647	0.6648	0.8760	0.056*
H12B	0.2413	0.7012	0.8904	0.056*
C13	0.2335 (2)	0.5609 (3)	0.79602 (13)	0.0600 (6)
H13A	0.2397	0.6486	0.7606	0.072*
H13B	0.2747	0.4652	0.7834	0.072*
C14	−0.0747 (2)	−0.0112 (3)	0.75962 (13)	0.0693 (7)
H14A	−0.1371	−0.0923	0.7487	0.083*
H14B	−0.0105	−0.0540	0.7402	0.083*
C15	−0.1168 (2)	0.1426 (4)	0.71898 (15)	0.0842 (9)
H15A	−0.1599	0.1168	0.6676	0.101*
H15B	−0.1693	0.1986	0.7447	0.101*
C16	0.0551 (3)	0.5107 (3)	0.70931 (13)	0.0714 (7)
H16A	0.1052	0.4583	0.6806	0.086*
H16B	0.0369	0.6211	0.6892	0.086*
C17	−0.0534 (2)	0.4149 (4)	0.70047 (13)	0.0763 (8)
H17A	−0.0980	0.4550	0.7355	0.092*
H17B	−0.1009	0.4260	0.6489	0.092*

Atomic displacement parameters (Å^2)

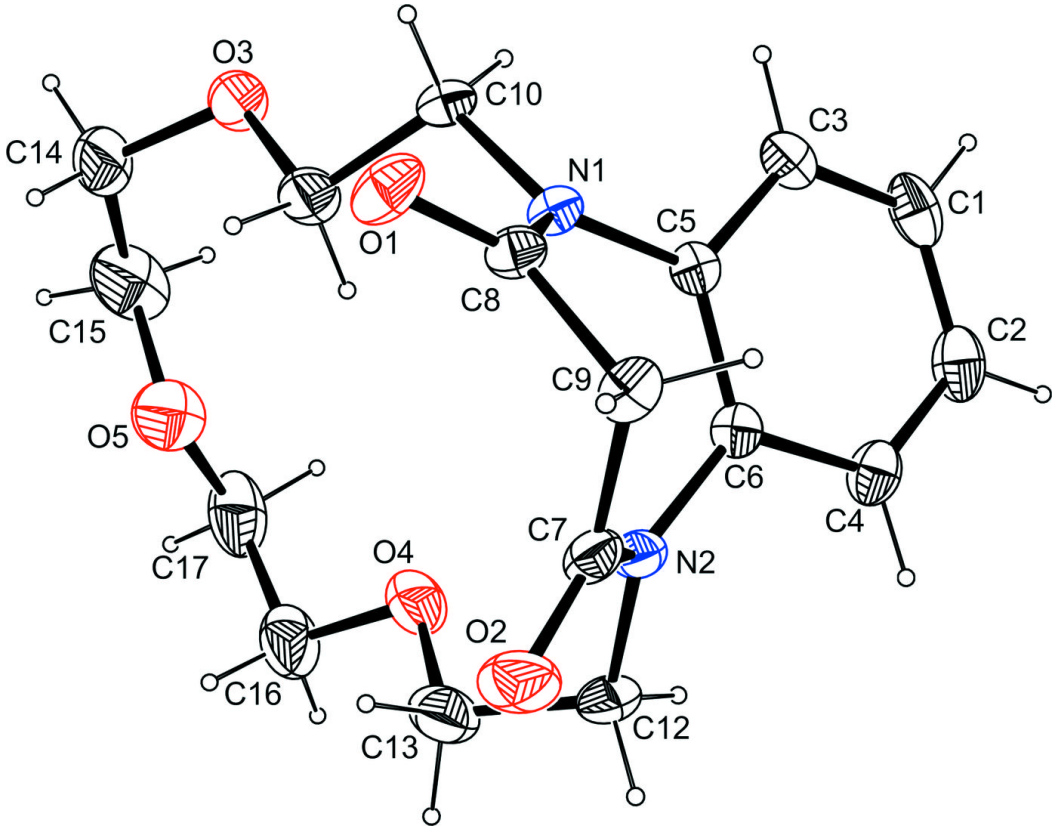
	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0283 (6)	0.0268 (7)	0.0385 (7)	−0.0017 (5)	0.0088 (5)	0.0010 (5)
N2	0.0387 (7)	0.0269 (7)	0.0406 (8)	−0.0030 (6)	0.0144 (6)	0.0008 (6)
O1	0.0446 (7)	0.0277 (6)	0.0760 (10)	0.0037 (5)	0.0165 (7)	−0.0054 (6)
O2	0.0534 (8)	0.0561 (9)	0.0809 (11)	−0.0055 (7)	0.0410 (8)	−0.0002 (8)
O3	0.0448 (7)	0.0668 (9)	0.0454 (7)	−0.0242 (7)	0.0106 (6)	−0.0088 (6)
O4	0.0686 (10)	0.0751 (10)	0.0376 (7)	−0.0089 (8)	0.0106 (7)	0.0001 (7)
O5	0.0629 (10)	0.0820 (12)	0.0626 (10)	−0.0037 (9)	0.0053 (8)	0.0052 (9)
C1	0.0423 (10)	0.0748 (14)	0.0338 (9)	0.0152 (10)	0.0104 (8)	−0.0082 (9)
C2	0.0498 (11)	0.0513 (11)	0.0448 (10)	0.0172 (9)	0.0065 (8)	−0.0129 (9)
C3	0.0343 (8)	0.0531 (11)	0.0317 (8)	0.0013 (8)	0.0086 (7)	0.0026 (7)
C4	0.0429 (9)	0.0334 (9)	0.0470 (10)	0.0066 (7)	0.0050 (8)	−0.0062 (7)
C5	0.0282 (7)	0.0325 (8)	0.0296 (7)	0.0037 (6)	0.0048 (6)	0.0006 (6)
C6	0.0308 (8)	0.0313 (8)	0.0338 (8)	0.0042 (6)	0.0061 (6)	−0.0003 (6)
C7	0.0333 (8)	0.0314 (8)	0.0507 (10)	−0.0078 (7)	0.0161 (7)	−0.0073 (7)
C8	0.0330 (8)	0.0269 (8)	0.0404 (9)	0.0018 (6)	0.0091 (7)	0.0043 (7)
C9	0.0265 (7)	0.0337 (9)	0.0513 (10)	0.0031 (7)	0.0078 (7)	−0.0041 (7)
C10	0.0323 (8)	0.0303 (8)	0.0448 (9)	−0.0066 (7)	0.0112 (7)	0.0036 (7)
C11	0.0379 (9)	0.0599 (12)	0.0456 (10)	−0.0163 (9)	0.0121 (8)	−0.0031 (9)
C12	0.0590 (11)	0.0300 (9)	0.0537 (11)	−0.0079 (8)	0.0187 (9)	0.0070 (8)
C13	0.0777 (15)	0.0543 (12)	0.0555 (12)	0.0009 (11)	0.0306 (11)	0.0118 (10)
C14	0.0695 (15)	0.0887 (18)	0.0486 (12)	−0.0384 (14)	0.0122 (11)	−0.0221 (12)
C15	0.0585 (14)	0.133 (3)	0.0515 (14)	−0.0227 (16)	−0.0061 (11)	0.0033 (15)
C16	0.109 (2)	0.0618 (15)	0.0395 (12)	0.0099 (14)	0.0106 (12)	0.0042 (10)
C17	0.0862 (18)	0.0896 (19)	0.0407 (12)	0.0155 (16)	−0.0099 (11)	−0.0094 (12)

Geometric parameters (Å, °)

N1—C8	1.364 (2)	C7—C9	1.510 (3)
N1—C5	1.423 (2)	C8—C9	1.511 (2)
N1—C10	1.469 (2)	C9—H9A	0.9700
N2—C7	1.366 (2)	C9—H9B	0.9700
N2—C6	1.426 (2)	C10—C11	1.505 (3)
N2—C12	1.475 (2)	C10—H10A	0.9700
O1—C8	1.2240 (19)	C10—H10B	0.9700
O2—C7	1.223 (2)	C11—H11A	0.9700
O3—C11	1.419 (2)	C11—H11B	0.9700
O3—C14	1.423 (3)	C12—C13	1.503 (3)
O4—C13	1.410 (3)	C12—H12A	0.9700
O4—C16	1.431 (3)	C12—H12B	0.9700
O5—C15	1.411 (3)	C13—H13A	0.9700
O5—C17	1.413 (3)	C13—H13B	0.9700
C1—C3	1.378 (3)	C14—C15	1.470 (4)
C1—C2	1.388 (3)	C14—H14A	0.9700
C1—H1	0.9300	C14—H14B	0.9700
C2—C4	1.375 (3)	C15—H15A	0.9700
C2—H2	0.9300	C15—H15B	0.9700
C3—C5	1.404 (2)	C16—C17	1.471 (4)
C3—H3	0.9300	C16—H16A	0.9700
C4—C6	1.398 (2)	C16—H16B	0.9700
C4—H4	0.9300	C17—H17A	0.9700
C5—C6	1.395 (2)	C17—H17B	0.9700
C8—N1—C5	122.77 (13)	H10A—C10—H10B	108.1
C8—N1—C10	118.20 (13)	O3—C11—C10	107.38 (14)
C5—N1—C10	119.03 (13)	O3—C11—H11A	110.2
C7—N2—C6	121.80 (14)	C10—C11—H11A	110.2
C7—N2—C12	117.70 (15)	O3—C11—H11B	110.2
C6—N2—C12	119.39 (14)	C10—C11—H11B	110.2
C11—O3—C14	113.89 (15)	H11A—C11—H11B	108.5
C13—O4—C16	110.95 (18)	N2—C12—C13	114.77 (16)
C15—O5—C17	114.5 (2)	N2—C12—H12A	108.6
C3—C1—C2	119.72 (17)	C13—C12—H12A	108.6
C3—C1—H1	120.1	N2—C12—H12B	108.6
C2—C1—H1	120.1	C13—C12—H12B	108.6
C4—C2—C1	120.10 (17)	H12A—C12—H12B	107.6
C4—C2—H2	120.0	O4—C13—C12	111.57 (17)
C1—C2—H2	120.0	O4—C13—H13A	109.3
C1—C3—C5	120.71 (17)	C12—C13—H13A	109.3
C1—C3—H3	119.6	O4—C13—H13B	109.3
C5—C3—H3	119.6	C12—C13—H13B	109.3
C2—C4—C6	120.98 (18)	H13A—C13—H13B	108.0
C2—C4—H4	119.5	O3—C14—C15	114.1 (2)
C6—C4—H4	119.5	O3—C14—H14A	108.7
C6—C5—C3	119.31 (15)	C15—C14—H14A	108.7

C6—C5—N1	121.65 (13)	O3—C14—H14B	108.7
C3—C5—N1	119.03 (15)	C15—C14—H14B	108.7
C5—C6—C4	118.99 (15)	H14A—C14—H14B	107.6
C5—C6—N2	121.60 (14)	O5—C15—C14	110.6 (2)
C4—C6—N2	119.41 (15)	O5—C15—H15A	109.5
O2—C7—N2	122.34 (17)	C14—C15—H15A	109.5
O2—C7—C9	121.79 (16)	O5—C15—H15B	109.5
N2—C7—C9	115.83 (14)	C14—C15—H15B	109.5
O1—C8—N1	121.59 (15)	H15A—C15—H15B	108.1
O1—C8—C9	121.89 (15)	O4—C16—C17	110.3 (2)
N1—C8—C9	116.45 (14)	O4—C16—H16A	109.6
C7—C9—C8	108.22 (14)	C17—C16—H16A	109.6
C7—C9—H9A	110.1	O4—C16—H16B	109.6
C8—C9—H9A	110.1	C17—C16—H16B	109.6
C7—C9—H9B	110.1	H16A—C16—H16B	108.1
C8—C9—H9B	110.1	O5—C17—C16	107.8 (2)
H9A—C9—H9B	108.4	O5—C17—H17A	110.2
N1—C10—C11	110.15 (13)	C16—C17—H17A	110.2
N1—C10—H10A	109.6	O5—C17—H17B	110.2
C11—C10—H10A	109.6	C16—C17—H17B	110.2
N1—C10—H10B	109.6	H17A—C17—H17B	108.5
C11—C10—H10B	109.6		

Fig. 1


O3
C10
C14
C3
N1
C1
O1
C5
C8
C15
C2
C9
O5
C6
C4
C7
C17
O4
N2
O2
C16
C12
C13