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Hydrazine-1,2-diium bis(3-carboxy-4-hydroxybenzenesulfonate) tetrahydrate

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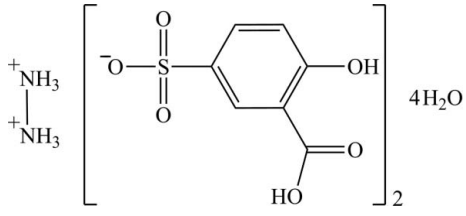
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.040; wR factor = 0.107; data-to-parameter ratio = 14.5.

Reaction of 5-sulfosalicylic acid with hydrazine hydrate at pH = 1 results in the formation of the title hydrated salt, $0.5N_2H_6^{2+}\cdot C_7H_5O_6S^-\cdot 2H_2O$. The hydrazinium dications lie on centres of inversion. They are located between 3-carboxy-4-hydroxybenzenesulfonate anions, forming intermolecular N—H···O hydrogen bonds with sulfonate ions and water molecules of crystallisation. Further intra- and intermolecular O—H···O hydrogen bonds are observed in the crystal structure.

Related literature

For general background on hydrogen bonding in proton-transfer compounds of 3-carboxy-4-hydroxybenzenesulfonate anions with Lewis bases, see: Smith *et al.* (2004, 2005). For recent related structures containing the 3-carboxy-4-hydroxybenzenesulfonate anion, see: Wang, Yang *et al.* (2008); Wang, Yao *et al.* (2008); Smith & Wermuth (2009); Hemamalini & Fun (2010); Yin *et al.* (2010). For related structures containing the $N_2H_6^{2+}$ hydrazinium dication, see: Starosta & Leciejewicz (2008); Klapotke *et al.* (1996).



Experimental

Crystal data

$0.5N_2H_6^{2+}\cdot C_7H_5O_6S^-\cdot 2H_2O$
M_r = 270.25
Triclinic, $P\overline{1}$
a = 7.0620 (5) Å
b = 7.2069 (4) Å
c = 11.5995 (8) Å
α = 78.460 (3)°
β = 75.806 (3)°
γ = 77.379 (3)°
V = 551.77 (6) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.33 mm^{-1}
T = 296 K
0.50 × 0.40 × 0.30 mm

Data collection

Bruker Kappa APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
T_{min} = 0.823, T_{max} = 0.882
9320 measured reflections
2711 independent reflections
2582 reflections with $I > 2\sigma(I)$
R_{int} = 0.057

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.107
S = 1.12
2711 reflections
187 parameters
6 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.52 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O6—H6···O5	0.82	1.88	2.6074 (18)	146
O7—H7A···O2^{i}	0.83 (2)	1.98 (2)	2.8036 (15)	177 (2)
O7—H7B···O8	0.85 (2)	1.84 (2)	2.6754 (17)	171 (2)
O4—H4···O7^{ii}	0.79 (3)	1.89 (3)	2.6758 (16)	174 (3)
O8—H8B···O2^{iii}	0.86 (2)	2.00 (2)	2.8384 (16)	165 (2)
O8—H8A···O3^{iv}	0.87 (2)	1.95 (2)	2.8213 (17)	177 (3)
N1—H1A···O3^{v}	0.82 (2)	1.93 (2)	2.7493 (17)	175.2 (19)
N1—H1B···O7	0.94 (2)	1.84 (2)	2.7798 (16)	174.5 (19)
N1—H1C···O1^{vi}	0.91 (2)	1.84 (2)	2.6813 (16)	154 (2)

Symmetry codes: (i) $-x, -y+2, -z+1$; (ii) $x, y+1, z$; (iii) $-x+1, -y+2, -z+1$; (iv) $x, y-1, z+1$; (v) $-x, -y+1, -z+1$; (vi) $-x+1, -y+1, -z+1$.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2* and *SAINT* (Bruker, 2004); data reduction: *SAINT* and *XPREP* (Bruker, 2004); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *Mercury* (Bruno *et al.*, 2002); software used to prepare material for publication: *SHELXL97*.

The authors acknowledge the services of Sophisticated Test and Instrumentation Centre, Cochin University, Kochi, for providing the single-crystal X-ray diffraction data. We thank the All India Council for Technical Education, New Delhi (grant-in-aid No. 8023/BOR/RID/RPS-012/2009-10), for financial support of this work.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZQ2096).

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supplementary materials

Acta Cryst. (2011). E**67**, o1236-o1237 [**doi:10.1107/S1600536811014231**]

Hydrazine-1,2-diium bis(3-carboxy-4-hydroxybenzenesulfonate) tetrahydrate

D. Selvaraju, R. Venkatesh and V. Sundararajan

Comment

5-Sulfosalicylic acid, a strong organic acid with $pK_a = 2.85$, donates its sulfonic protons to N-containing Lewis bases (Smith *et al.*, 2004, 2005) forming organic salts (Wang, Yang *et al.*, 2008; Wang, Yao *et al.*, 2008; Smith & Wermuth, 2009; Hemamalini & Fun, 2010; Yin *et al.*, 2010). Hydrazine as a diacidic base captures the H atoms of sulfonic groups from two acid molecules to form a dicationic hydrazinium salt. The molecular structure of the salt, $C_7H_5O_6S.0.5(H_6N_2).2(H_2O)$, formed by the reaction of 5-sulfosalicylic acid with hydrazine hydrate at pH = 1 is shown in Fig.1 and the various types of hydrogen bonds involved in the crystal structure are reported in Table 1.

The unit cell of the crystal structure of the title compound contains eight $(N_2H_6)^{2+}$ units located at the unit cell corners (centres of inversion) which contribute to 1/8 of the charge of each cell, two compensating 3-carboxy-4-hydroxybenzenesulfonate anions, and four isolated water molecules. The anions are held by intermolecular N—H···O hydrogen bonds in all directions with the hydrazinium ions. These N···O interactions fall in the range of 2.6813 (16)–2.7798 (16) Å. In addition, the intramolecular O6—H6···O5 hydrogen bond [$D\cdots A$ = 2.6074 (18) Å] and intermolecular O—H···O hydrogen bonds, O7—H7·····O2 [$D\cdots A$ = 2.8036 (15) Å] between sulfonyl O atoms and water molecules, O4—H4···O7 [$D\cdots A$ = 2.6758 (16) Å] between carboxyl H atoms and isolated water molecules, and O7—H7B···O8 [$D\cdots A$ = 2.6754 (17) Å] between lattice water molecules, stabilize the molecular conformation and the crystal structure.

The N1—$N1^{i}$ distance of 1.433 (2) Å (symmetry code: (i) -*x*, -*y*, -*z* + 2) is in a good agreement with the values reported by Starosta & Leciejewicz (2008) and Klapotke *et al.* (1996). The hydrazinium ion has a staggered conformation due to the symmetry imposed by the centre of inversion located in the middle of the N—N bond.

Experimental

The title compound was synthesized by dissolving 5-sulfosalicylicacid dihydrate (2 mmol, 0.508 g) and hydrazine hydrate (99.98% pure; 1 mmol, 0.05 ml) in 30 ml of distilled water at pH = 1. The mixture was stirred for 4 h at ambient temperature and then filtered. The resulting clear solution was kept for three weeks in a wooden enclosure. Colourless prismatic crystals suitable for single-crystal X-ray diffraction analysis were grown by slow evaporation of the solvent.

Refinement

All non-H atoms were refined anisotropically. H atoms bonded to C atoms were positioned geometrically with C—H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ while H atoms bonded to N and O atoms were found in difference Fourier maps and their coordinates and thermal parameters freely refined.

Figures

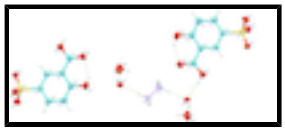

Fig. 1. View of the title compound (I), showing displacement ellipsoids at the 50% probability level. Symmetry code: 1 - *x*, 1 - *y*, 1 - *z*.

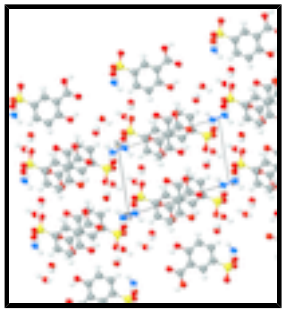

Fig. 2. The packing diagram of title compound (I).

Hydrazine-1,2-diium bis(3-carboxy-4-hydroxybenzenesulfonate) tetrahydrate

Crystal data

$0.5N_2H_6^{2+}\cdot C_7H_5O_6S^-\cdot 2H_2O$	$Z = 2$
$M_r = 270.25$	$F(000) = 282$
Triclinic, $P\overline{1}$	$D_x = 1.627$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 7.0620$ (5) Å	Cell parameters from 6221 reflections
$b = 7.2069$ (4) Å	$\theta = 2.5$–$26.2°$
$c = 11.5995$ (8) Å	$\mu = 0.33$ mm^{-1}
$\alpha = 78.460$ (3)°	$T = 296$ K
$\beta = 75.806$ (3)°	Block, colourless
$\gamma = 77.379$ (3)°	0.50 × 0.40 × 0.30 mm
$V = 551.77$ (6) Å^3	

Data collection

Bruker Kappa APEXII CCD area-detector diffractometer	2711 independent reflections
Radiation source: fine-focus sealed tube	2582 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.057$
ω and φ scans	$\theta_{max} = 28.2°$, $\theta_{min} = 2.9°$
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)	$h = -9\rightarrow9$
$T_{min} = 0.823$, $T_{max} = 0.882$	$k = -9\rightarrow9$
9320 measured reflections	$l = -13\rightarrow15$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map

Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.040$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.107$	$w = 1/[\sigma^2(F_o^2) + (0.0527P)^2 + 0.1344P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.12$	$(\Delta/\sigma)_{max} < 0.001$
2711 reflections	$\Delta\rho_{max} = 0.41$ e Å^{-3}
187 parameters	$\Delta\rho_{min} = -0.52$ e Å^{-3}
6 restraints	Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 1.12 (4)

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 $F^2 > \sigma(F^2)$ 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}
C1	0.28715 (19)	1.12543 (18)	0.37831 (12)	0.0277 (3)
H7	0.2576	1.2577	0.3783	0.033*
C2	0.36576 (19)	1.05458 (18)	0.27144 (12)	0.0268 (3)
C3	0.4096 (2)	0.8553 (2)	0.27042 (15)	0.0373 (3)
H1	0.4618	0.8079	0.1981	0.045*
C4	0.3755 (3)	0.7304 (2)	0.37640 (16)	0.0434 (4)
H3	0.4049	0.5983	0.3756	0.052*
C5	0.2966 (2)	0.8008 (2)	0.48587 (14)	0.0364 (3)
C6	0.2515 (2)	1.00021 (19)	0.48664 (13)	0.0290 (3)
C7	0.1686 (2)	1.0757 (2)	0.60113 (13)	0.0323 (3)
N1	0.01665 (18)	−0.00608 (18)	0.93729 (10)	0.0272 (3)
O1	0.59710 (15)	1.13796 (16)	0.06468 (10)	0.0369 (3)
O7	−0.03247 (17)	0.36335 (14)	0.80737 (10)	0.0340 (3)
O2	0.40796 (15)	1.40062 (14)	0.17028 (9)	0.0333 (3)
O8	0.19337 (19)	0.5388 (2)	0.88950 (13)	0.0505 (3)
O3	0.24335 (15)	1.22941 (15)	0.07799 (10)	0.0356 (3)
O4	0.1284 (2)	1.26416 (17)	0.58929 (11)	0.0445 (3)
O5	0.1392 (2)	0.97087 (18)	0.69881 (10)	0.0454 (3)
O6	0.2667 (2)	0.67267 (18)	0.58729 (12)	0.0568 (4)

H6	0.2187	0.7301	0.6450	0.085*
S1	0.40819 (4)	1.21604 (4)	0.13623 (3)	0.02543 (16)
H7A	−0.144 (2)	0.430 (3)	0.8122 (19)	0.046 (5)*
H7B	0.043 (3)	0.425 (3)	0.825 (2)	0.062 (7)*
H4	0.086 (4)	1.300 (4)	0.653 (2)	0.058 (7)*
H8B	0.312 (3)	0.559 (4)	0.858 (2)	0.069 (7)*
H8A	0.208 (4)	0.446 (3)	0.949 (2)	0.091 (10)*
H1A	−0.060 (3)	−0.070 (3)	0.9285 (17)	0.035 (5)*
H1B	−0.008 (3)	0.118 (3)	0.8930 (19)	0.045 (5)*
H1C	0.144 (3)	−0.067 (3)	0.9181 (19)	0.047 (5)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0271 (6)	0.0250 (6)	0.0312 (6)	−0.0017 (5)	−0.0065 (5)	−0.0078 (5)
C2	0.0240 (6)	0.0262 (6)	0.0300 (6)	−0.0008 (4)	−0.0065 (5)	−0.0067 (5)
C3	0.0428 (8)	0.0286 (7)	0.0387 (8)	0.0024 (6)	−0.0068 (6)	−0.0130 (6)
C4	0.0567 (10)	0.0237 (6)	0.0467 (9)	0.0024 (6)	−0.0108 (8)	−0.0092 (6)
C5	0.0438 (8)	0.0273 (7)	0.0374 (7)	−0.0021 (6)	−0.0134 (6)	−0.0018 (6)
C6	0.0289 (6)	0.0291 (6)	0.0303 (6)	−0.0027 (5)	−0.0090 (5)	−0.0069 (5)
C7	0.0331 (7)	0.0350 (7)	0.0300 (7)	−0.0061 (5)	−0.0079 (5)	−0.0063 (5)
N1	0.0263 (6)	0.0313 (6)	0.0243 (6)	−0.0042 (4)	−0.0055 (4)	−0.0061 (4)
O1	0.0251 (5)	0.0461 (6)	0.0361 (5)	−0.0001 (4)	0.0002 (4)	−0.0140 (5)
O7	0.0362 (6)	0.0295 (5)	0.0361 (5)	−0.0013 (4)	−0.0069 (4)	−0.0105 (4)
O2	0.0336 (5)	0.0292 (5)	0.0376 (5)	−0.0074 (4)	−0.0033 (4)	−0.0094 (4)
O8	0.0391 (7)	0.0573 (8)	0.0577 (8)	−0.0184 (6)	−0.0190 (6)	0.0066 (6)
O3	0.0306 (5)	0.0376 (5)	0.0417 (6)	−0.0068 (4)	−0.0152 (4)	−0.0031 (4)
O4	0.0646 (8)	0.0348 (6)	0.0307 (6)	−0.0039 (5)	−0.0029 (5)	−0.0115 (4)
O5	0.0622 (8)	0.0433 (6)	0.0285 (5)	−0.0086 (5)	−0.0079 (5)	−0.0034 (5)
O6	0.0906 (11)	0.0314 (6)	0.0418 (7)	−0.0058 (6)	−0.0132 (7)	0.0033 (5)
S1	0.0201 (2)	0.0276 (2)	0.0285 (2)	−0.00186 (12)	−0.00404 (13)	−0.00819 (13)

Geometric parameters (Å, °)

C1—C2	1.3775 (19)	N1—N1^{i}	1.433 (2)
C1—C6	1.3954 (19)	N1—H1A	0.82 (2)
C1—H7	0.9300	N1—H1B	0.94 (2)
C2—C3	1.4030 (18)	N1—H1C	0.91 (2)
C2—S1	1.7594 (14)	O1—S1	1.4495 (10)
C3—C4	1.374 (2)	O7—H7A	0.826 (15)
C3—H1	0.9300	O7—H7B	0.847 (15)
C4—C5	1.403 (2)	O2—S1	1.4610 (10)
C4—H3	0.9300	O8—H8B	0.859 (16)
C5—O6	1.3453 (19)	O8—H8A	0.874 (17)
C5—C6	1.404 (2)	O3—S1	1.4599 (10)
C6—C7	1.473 (2)	O4—H4	0.79 (3)
C7—O5	1.2282 (18)	O6—H6	0.8200
C7—O4	1.3115 (19)		

C2—C1—C6	120.48 (12)	O5—C7—C6	122.77 (14)
C2—C1—H7	119.8	O4—C7—C6	114.08 (13)
C6—C1—H7	119.8	N1^{i}—N1—H1A	108.5 (13)
C1—C2—C3	120.22 (13)	N1^{i}—N1—H1B	109.8 (13)
C1—C2—S1	119.44 (10)	H1A—N1—H1B	109.2 (19)
C3—C2—S1	120.34 (11)	N1^{i}—N1—H1C	104.8 (14)
C4—C3—C2	119.94 (14)	H1A—N1—H1C	110.6 (18)
C4—C3—H1	120.0	H1B—N1—H1C	113.7 (18)
C2—C3—H1	120.0	H7A—O7—H7B	108.4 (18)
C3—C4—C5	120.36 (13)	H8B—O8—H8A	104 (2)
C3—C4—H3	119.8	C7—O4—H4	111.4 (18)
C5—C4—H3	119.8	C5—O6—H6	109.5
O6—C5—C4	118.15 (13)	O1—S1—O3	112.19 (7)
O6—C5—C6	122.23 (14)	O1—S1—O2	112.97 (6)
C4—C5—C6	119.63 (14)	O3—S1—O2	110.87 (6)
C1—C6—C5	119.37 (13)	O1—S1—C2	107.30 (6)
C1—C6—C7	120.56 (12)	O3—S1—C2	106.83 (6)
C5—C6—C7	120.07 (13)	O2—S1—C2	106.24 (6)
O5—C7—O4	123.15 (14)		
C6—C1—C2—C3	0.4 (2)	C4—C5—C6—C7	180.00 (15)
C6—C1—C2—S1	179.78 (10)	C1—C6—C7—O5	−178.27 (14)
C1—C2—C3—C4	−0.5 (2)	C5—C6—C7—O5	1.3 (2)
S1—C2—C3—C4	−179.82 (13)	C1—C6—C7—O4	2.0 (2)
C2—C3—C4—C5	0.1 (3)	C5—C6—C7—O4	−178.35 (14)
C3—C4—C5—O6	−179.70 (16)	C1—C2—S1—O1	139.04 (11)
C3—C4—C5—C6	0.3 (3)	C3—C2—S1—O1	−41.61 (14)
C2—C1—C6—C5	0.0 (2)	C1—C2—S1—O3	−100.46 (12)
C2—C1—C6—C7	179.62 (12)	C3—C2—S1—O3	78.89 (13)
O6—C5—C6—C1	179.65 (14)	C1—C2—S1—O2	17.95 (13)
C4—C5—C6—C1	−0.4 (2)	C3—C2—S1—O2	−162.70 (12)
O6—C5—C6—C7	0.0 (2)		

Symmetry codes: (i) −*x*, −*y*, −*z*+2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O6—H6···O5	0.82	1.88	2.6074 (18)	146.
O7—H7A···O2^{ii}	0.83 (2)	1.98 (2)	2.8036 (15)	177 (2)
O7—H7B···O8	0.85 (2)	1.84 (2)	2.6754 (17)	171 (2)
O4—H4···O7^{iii}	0.79 (3)	1.89 (3)	2.6758 (16)	174 (3)
O8—H8B···O2^{iv}	0.86 (2)	2.00 (2)	2.8384 (16)	165 (2)
O8—H8A···O3^{v}	0.87 (2)	1.95 (2)	2.8213 (17)	177 (3)
N1—H1A···O3^{vi}	0.82 (2)	1.93 (2)	2.7493 (17)	175.2 (19)
N1—H1B···O7	0.94 (2)	1.84 (2)	2.7798 (16)	174.5 (19)
N1—H1C···O1^{vii}	0.91 (2)	1.84 (2)	2.6813 (16)	154 (2)

Symmetry codes: (ii) −*x*, −*y*+2, −*z*+1; (iii) *x*, *y*+1, *z*; (iv) −*x*+1, −*y*+2, −*z*+1; (v) *x*, *y*−1, *z*+1; (vi) −*x*, −*y*+1, −*z*+1; (vii) −*x*+1, −*y*+1, −*z*+1.

Fig. 1

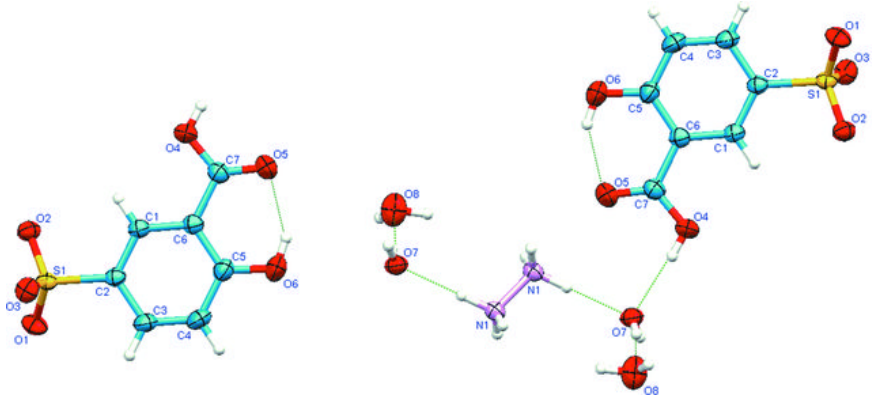
O4
C7
O5
O2
C1
C6
S1
C2
C5
O6
O3
C3
O1
C4
O1
O3
C4
C3
C2
S1
O6
C5
C6
O2
C1
O5
C7
O4
O8
O7
N1
N1
O7
O8

Fig. 2