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4,4′-Dimethyl-2,2′-[1,2-phenylenebis(nitrilomethylidyne)]diphenol

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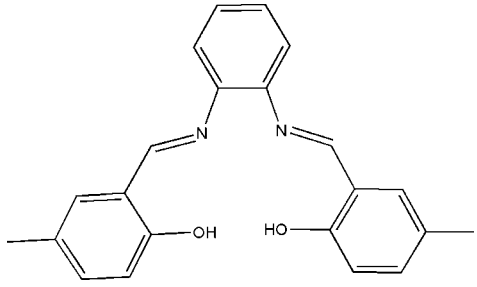
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.049; wR factor = 0.133; data-to-parameter ratio = 17.1.

In the title Schiff base, $C_{22}H_{20}N_2O_2$, the benzene ring forms dihedral angles of 53.92 (1) and 3.62 (1)° with the two salicylaldimine groups. There are two strong O—H···N intramolecular hydrogen bonds. The crystal packing is stabilized by weak intermolecular C—H···O hydrogen bonds and π–π stacking interactions (average distance 3.39 Å).

Related literature

For related literature, see: Cohen *et al.* (1964).



Experimental

Crystal data

$C_{22}H_{20}N_2O_2$
M_r = 344.40
Monoclinic, $P2_1/c$
a = 6.0835 (12) Å
b = 16.207 (3) Å
c = 18.607 (4) Å
β = 98.28 (3)°
V = 1815.4 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 296 (2) K
0.33 × 0.28 × 0.21 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.971, T_{max} = 0.985
17288 measured reflections
4063 independent reflections
2571 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.134
S = 1.03
4063 reflections
237 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.82	1.87	2.595 (2)	147
O2—H2A···N2	0.82	1.86	2.5880 (19)	147
C8—H8A···O1^{i}	0.93	2.56	3.407 (2)	152
C18—H18A···O1^{ii}	0.93	2.54	3.359 (2)	146

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

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2120).

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supplementary materials

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4,4'-Dimethyl-2,2'-[1,2-phenylenebis(nitrilomethylidyne)]diphenol

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Comment

Schiff bases have been used extensively as ligands in the field of coordination chemistry. Some of the reasons are that the intramolecular hydrogen bond between the O and N atoms plays an important role in the formation of metal complexes, and that Schiff base compounds show photochromism and thermochromism in the solid state by proton transfer from the hydroxyl O atom to the imine N atom (Cohen *et al.*, 1964). On the basis of structural studies on photochromic and thermochromic salicylaldimine derivatives it was concluded that there is a significant difference in crydtal packing of these molecules: molecules exhibiting thermochromism are planar while those showing photochromism are non-planar (Cohen *et al.*, 1964). In other words, photochromic salicylideneanilines are packed rather loosely in the crystal, in which nonplanar molecules may undergo some conformational changes, while thermochromic salicylideneanilines are packed tightly to form one-dimensional columns. With the aim of gaining a deeper insight into the structural aspects responsible for the observed phenomenon in the solid state, conformational and crystallographic analysis of the non-planar tetra-dentate title compound (I), has been carried out and the results are presented in this paper.

The molecular structure of (I) is illustrated in Fig. 1.

The title molecule is not planar. The salicylaldimine groups C1—C7 (A) and C16—C22 (B) are twisted relative to the phenylene spacer and the angles between the spacer and the salicylaldimino parts A and B are 53.92 (1) and 3.62 (1)°, respectively. The dihedral angle between the salicylaldimine groups A and B is equal to 56.23 (2)°.

In the title molecule there are intramolecular hydrogen bonds between between O1 and N1 and between O2 and N2 atoms (Table 1). Clearly, the enolimine tautomer is favoured over the ketamine form. The crystal packing is stabilized by weak intermolecular hydrogen bonds C—H···O (Table 1) and π···π stacking interactions between benzene ring and salicylaldimine group B.

Experimental

1,2-Phenylenediamine(0.01 mol, 1.08 g) and 5-methylsalicylaldehyde (0.02 mol, 2.76 g) were dissolved in ethanol and the solution was refluxed for 3 h. After evaporation, a crude product was recrystallized twice from ethanol to give a pure yellow product. Yield: 90.1%. Melting point: 494–496 K. Calcd. for $C_{22}H_{20}N_2O_2$: C, 76.72; H, 5.85; N, 8.13; Found: C, 76.44; H, 5.75; N, 8.07%.

Refinement

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms (C—H = 0.93 Å or 0.96 Å; O—H = 0.82 Å) and U_{iso}(H) values equal to 1.2U_{eq}(C) or 1.5U_{eq}(O).

Figures

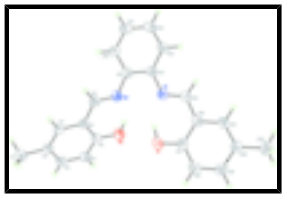

Fig. 1. The structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

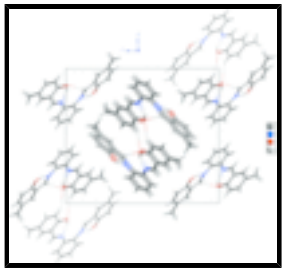

Fig. 2. A view of crystal packing of (I).

4,4'-Dimethyl-2,2'-[1,2-phenylenebis(nitrilomethylidyne)]diphenol

Crystal data

$C_{22}H_{20}N_2O_2$	F_{000} = 728
M_r = 344.40	D_x = 1.260 Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 494-496 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 6.0835 (12) Å	Cell parameters from 8652 reflections
b = 16.207 (3) Å	θ = 1.0–27.4º
c = 18.607 (4) Å	μ = 0.08 mm^{-1}
β = 98.28 (3)º	T = 296 (2) K
V = 1815.4 (6) Å^3	Block, orange
Z = 4	0.33 × 0.29 × 0.21 mm

Data collection

Rigaku R-AXIS RAPID diffractometer	4063 independent reflections
Radiation source: fine-focus sealed tube	2571 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.037
Detector resolution: 0 pixels mm^{-1}	θ_{max} = 27.4º
T = 296(2) K	θ_{min} = 3.4º
ω scans	h = −7→7
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	k = −21→21
T_{min} = 0.971, T_{max} = 0.985	l = −24→23
17288 measured reflections	

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
$wR(F^2) = 0.134$	$w = 1/[\sigma^2(F_o^2) + (0.0577P)^2 + 0.235P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} = 0.003$
4063 reflections	$\Delta\rho_{max} = 0.17$ e Å^{-3}
237 parameters	$\Delta\rho_{min} = -0.15$ 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 $F^2 > 2$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	0.4005 (2)	0.75760 (9)	0.02195 (8)	0.0530 (4)
O1	0.1506 (2)	0.88529 (8)	−0.01297 (7)	0.0731 (4)
H1	0.2520	0.8526	−0.0155	0.110*
C1	0.7385 (4)	1.07183 (14)	−0.30763 (12)	0.0790 (6)
H1B	0.8885	1.0532	−0.3072	0.118*
H1C	0.7385	1.1210	−0.2789	0.118*
H1D	0.6729	1.0834	−0.3566	0.118*
O2	0.2200 (2)	0.82823 (8)	−0.19143 (7)	0.0677 (4)
H2A	0.2921	0.8046	−0.1568	0.101*
N2	0.5699 (2)	0.78862 (9)	−0.10188 (7)	0.0535 (4)
C2	0.6061 (3)	1.00555 (11)	−0.27638 (9)	0.0562 (4)
C3	0.3863 (3)	0.99007 (11)	−0.30589 (10)	0.0606 (5)
H3A	0.3232	1.0202	−0.3462	0.073*
C4	0.2594 (3)	0.93154 (11)	−0.27725 (9)	0.0586 (5)
H4A	0.1126	0.9233	−0.2979	0.070*
C5	0.3491 (3)	0.88514 (11)	−0.21800 (9)	0.0508 (4)
C6	0.5724 (3)	0.89744 (11)	−0.18760 (8)	0.0489 (4)
C7	0.6942 (3)	0.95853 (11)	−0.21748 (9)	0.0564 (4)

H7A	0.8406	0.9678	−0.1968	0.068*
C8	0.6787 (3)	0.84472 (11)	−0.12984 (9)	0.0534 (4)
H8A	0.8289	0.8518	−0.1128	0.064*
C9	0.6778 (3)	0.72943 (10)	−0.05323 (9)	0.0509 (4)
C10	0.8666 (3)	0.68766 (11)	−0.06801 (10)	0.0592 (5)
H10A	0.9340	0.7028	−0.1078	0.071*
C11	0.9537 (3)	0.62385 (12)	−0.02361 (10)	0.0645 (5)
H11A	1.0801	0.5962	−0.0334	0.077*
C12	0.8537 (3)	0.60120 (12)	0.03489 (11)	0.0677 (5)
H12A	0.9106	0.5572	0.0638	0.081*
C13	0.6707 (3)	0.64270 (11)	0.05125 (10)	0.0638 (5)
H13A	0.6063	0.6271	0.0916	0.077*
C14	0.5802 (3)	0.70811 (10)	0.00797 (9)	0.0510 (4)
C15	0.2992 (3)	0.74716 (11)	0.07654 (9)	0.0540 (4)
H15A	0.3431	0.7042	0.1086	0.065*
C16	0.1188 (3)	0.79963 (10)	0.09036 (9)	0.0513 (4)
C17	0.0497 (3)	0.86653 (11)	0.04521 (10)	0.0562 (4)
C18	−0.1276 (3)	0.91440 (12)	0.05974 (11)	0.0683 (5)
H18A	−0.1730	0.9594	0.0303	0.082*
C19	−0.2366 (3)	0.89569 (12)	0.11747 (11)	0.0653 (5)
H19A	−0.3555	0.9284	0.1263	0.078*
C20	−0.1739 (3)	0.82931 (11)	0.16301 (10)	0.0588 (5)
C21	0.0043 (3)	0.78327 (11)	0.14885 (9)	0.0570 (4)
H21A	0.0510	0.7393	0.1794	0.068*
C22	−0.2979 (4)	0.80766 (14)	0.22487 (11)	0.0806 (6)
H22A	−0.4540	0.8163	0.2102	0.121*
H22B	−0.2470	0.8421	0.2659	0.121*
H22C	−0.2716	0.7508	0.2379	0.121*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0519 (8)	0.0496 (8)	0.0578 (9)	0.0005 (6)	0.0092 (7)	0.0059 (7)
O1	0.0760 (9)	0.0649 (9)	0.0824 (9)	0.0113 (7)	0.0251 (8)	0.0276 (7)
C1	0.0820 (14)	0.0738 (14)	0.0866 (15)	0.0036 (11)	0.0310 (12)	0.0169 (11)
O2	0.0551 (7)	0.0724 (9)	0.0716 (8)	−0.0099 (6)	−0.0041 (6)	0.0130 (7)
N2	0.0525 (8)	0.0601 (9)	0.0469 (8)	0.0036 (7)	0.0038 (7)	0.0036 (7)
C2	0.0619 (11)	0.0549 (10)	0.0544 (10)	0.0081 (8)	0.0171 (9)	0.0018 (8)
C3	0.0725 (12)	0.0574 (11)	0.0504 (10)	0.0143 (9)	0.0041 (9)	0.0025 (8)
C4	0.0548 (10)	0.0607 (11)	0.0565 (10)	0.0056 (8)	−0.0045 (9)	−0.0004 (9)
C5	0.0512 (9)	0.0506 (10)	0.0500 (9)	0.0011 (8)	0.0053 (8)	−0.0026 (7)
C6	0.0475 (9)	0.0560 (10)	0.0437 (8)	0.0048 (7)	0.0078 (7)	−0.0014 (7)
C7	0.0471 (9)	0.0641 (11)	0.0589 (10)	0.0022 (8)	0.0107 (8)	−0.0010 (9)
C8	0.0473 (9)	0.0643 (11)	0.0475 (9)	0.0049 (8)	0.0036 (8)	−0.0017 (8)
C9	0.0511 (9)	0.0503 (10)	0.0490 (9)	0.0011 (8)	−0.0009 (8)	−0.0022 (8)
C10	0.0597 (11)	0.0635 (12)	0.0533 (10)	0.0061 (9)	0.0047 (9)	−0.0083 (9)
C11	0.0679 (12)	0.0572 (11)	0.0660 (12)	0.0145 (9)	0.0013 (10)	−0.0120 (9)
C12	0.0792 (13)	0.0463 (10)	0.0740 (13)	0.0137 (9)	−0.0010 (11)	0.0017 (9)

C13	0.0757 (13)	0.0504 (10)	0.0653 (11)	0.0052 (9)	0.0107 (10)	0.0085 (9)
C14	0.0522 (9)	0.0442 (9)	0.0549 (10)	−0.0001 (7)	0.0023 (8)	−0.0009 (7)
C15	0.0586 (10)	0.0496 (10)	0.0521 (10)	0.0027 (8)	0.0022 (9)	0.0041 (8)
C16	0.0555 (10)	0.0460 (9)	0.0512 (9)	−0.0030 (7)	0.0031 (8)	0.0001 (7)
C17	0.0591 (10)	0.0482 (10)	0.0620 (11)	−0.0025 (8)	0.0109 (9)	0.0058 (8)
C18	0.0733 (12)	0.0493 (11)	0.0827 (13)	0.0088 (9)	0.0126 (11)	0.0116 (10)
C19	0.0643 (11)	0.0550 (11)	0.0779 (13)	0.0041 (9)	0.0152 (10)	−0.0081 (10)
C20	0.0677 (11)	0.0542 (11)	0.0555 (10)	−0.0013 (9)	0.0120 (9)	−0.0073 (8)
C21	0.0695 (11)	0.0522 (10)	0.0484 (10)	0.0027 (9)	0.0052 (9)	0.0031 (8)
C22	0.0953 (16)	0.0850 (16)	0.0665 (13)	0.0006 (12)	0.0286 (12)	−0.0062 (11)

Geometric parameters (Å, °)

N1—C15	1.273 (2)	C9—C14	1.401 (2)
N1—C14	1.410 (2)	C10—C11	1.381 (3)
O1—C17	1.353 (2)	C10—H10A	0.9300
O1—H1	0.8200	C11—C12	1.371 (3)
C1—C2	1.509 (3)	C11—H11A	0.9300
C1—H1B	0.9600	C12—C13	1.372 (3)
C1—H1C	0.9600	C12—H12A	0.9300
C1—H1D	0.9600	C13—C14	1.396 (2)
O2—C5	1.350 (2)	C13—H13A	0.9300
O2—H2A	0.8200	C15—C16	1.440 (2)
N2—C8	1.279 (2)	C15—H15A	0.9300
N2—C9	1.414 (2)	C16—C17	1.399 (2)
C2—C7	1.379 (2)	C16—C21	1.399 (2)
C2—C3	1.393 (3)	C17—C18	1.387 (3)
C3—C4	1.378 (3)	C18—C19	1.375 (3)
C3—H3A	0.9300	C18—H18A	0.9300
C4—C5	1.380 (2)	C19—C20	1.388 (3)
C4—H4A	0.9300	C19—H19A	0.9300
C5—C6	1.409 (2)	C20—C21	1.373 (3)
C6—C7	1.399 (2)	C20—C22	1.505 (3)
C6—C8	1.450 (2)	C21—H21A	0.9300
C7—H7A	0.9300	C22—H22A	0.9600
C8—H8A	0.9300	C22—H22B	0.9600
C9—C10	1.394 (2)	C22—H22C	0.9600
C15—N1—C14	123.29 (15)	C12—C11—H11A	120.0
C17—O1—H1	109.5	C10—C11—H11A	120.0
C2—C1—H1B	109.5	C11—C12—C13	120.79 (18)
C2—C1—H1C	109.5	C11—C12—H12A	119.6
H1B—C1—H1C	109.5	C13—C12—H12A	119.6
C2—C1—H1D	109.5	C12—C13—C14	120.65 (18)
H1B—C1—H1D	109.5	C12—C13—H13A	119.7
H1C—C1—H1D	109.5	C14—C13—H13A	119.7
C5—O2—H2A	109.5	C13—C14—C9	118.56 (16)
C8—N2—C9	121.53 (15)	C13—C14—N1	125.29 (16)
C7—C2—C3	117.08 (17)	C9—C14—N1	116.11 (15)
C7—C2—C1	122.12 (18)	N1—C15—C16	122.20 (16)

C3—C2—C1	120.80 (17)	N1—C15—H15A	118.9
C4—C3—C2	122.07 (17)	C16—C15—H15A	118.9
C4—C3—H3A	119.0	C17—C16—C21	118.29 (16)
C2—C3—H3A	119.0	C17—C16—C15	121.41 (16)
C3—C4—C5	120.33 (17)	C21—C16—C15	120.28 (16)
C3—C4—H4A	119.8	O1—C17—C18	119.03 (16)
C5—C4—H4A	119.8	O1—C17—C16	121.53 (16)
O2—C5—C4	118.72 (15)	C18—C17—C16	119.43 (16)
O2—C5—C6	121.86 (15)	C19—C18—C17	120.35 (18)
C4—C5—C6	119.41 (16)	C19—C18—H18A	119.8
C7—C6—C5	118.43 (15)	C17—C18—H18A	119.8
C7—C6—C8	120.42 (16)	C18—C19—C20	121.79 (18)
C5—C6—C8	121.03 (16)	C18—C19—H19A	119.1
C2—C7—C6	122.65 (17)	C20—C19—H19A	119.1
C2—C7—H7A	118.7	C21—C20—C19	117.33 (17)
C6—C7—H7A	118.7	C21—C20—C22	121.10 (18)
N2—C8—C6	121.25 (16)	C19—C20—C22	121.57 (18)
N2—C8—H8A	119.4	C20—C21—C16	122.80 (17)
C6—C8—H8A	119.4	C20—C21—H21A	118.6
C10—C9—C14	119.88 (16)	C16—C21—H21A	118.6
C10—C9—N2	121.52 (15)	C20—C22—H22A	109.5
C14—C9—N2	118.39 (15)	C20—C22—H22B	109.5
C11—C10—C9	120.10 (17)	H22A—C22—H22B	109.5
C11—C10—H10A	119.9	C20—C22—H22C	109.5
C9—C10—H10A	119.9	H22A—C22—H22C	109.5
C12—C11—C10	119.97 (18)	H22B—C22—H22C	109.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.82	1.87	2.595 (2)	147
O2—H2A···N2	0.82	1.86	2.5880 (19)	147
C8—H8A···O1^{i}	0.93	2.56	3.407 (2)	152
C18—H18A···O1^{ii}	0.93	2.54	3.359 (2)	146

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) −*x*, −*y*+2, −*z*.

Fig. 1

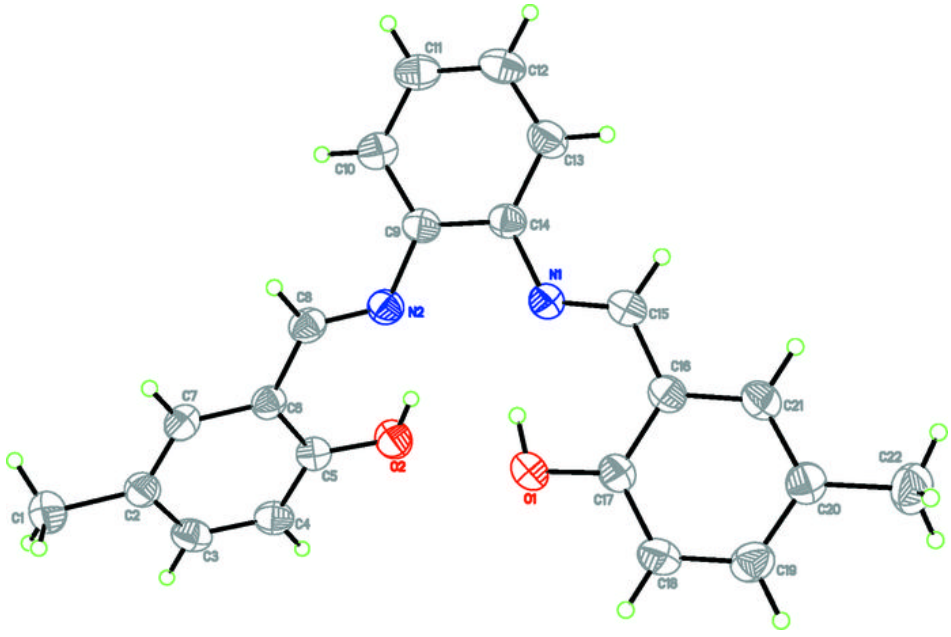
C11
C12
C13
C10
C9
C14
N1
C8
N2
C15
C16
C7
C6
C21
O2
C5
O1
C17
C22
C1
C2
C4
C20
C3
C19
C18

Fig. 2