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4-Methyl-*N*′-(2-oxoindolin-3-ylidene)-benzene-1-sulfonohydrazide

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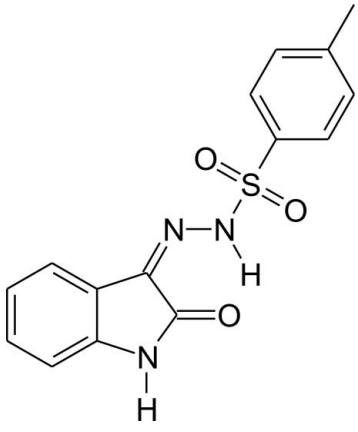
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.042; wR factor = 0.113; data-to-parameter ratio = 20.2.

In the title compound, $C_{15}H_{13}N_3O_3S$, the C—S—N(H)—N linkage is non-planar, the torsion angle being −65.12 (13)° and the S atom showing a tetrahedral environment. The compound has two almost planar fragments linked to the S atom: the isatin-derivative fragment [$(C_8H_5NO)N—N(H)$–] and the tolyl fragment [C_7H_7–] have maximum deviations from the mean plane through the non-H atoms of 0.0813 (13) and 0.0094 (16) Å, respectively, and make an interplanar angle of 80.48 (3)°. In the crystal, molecules are connected into inversion dimers *via* pairs of N—H···O hydrogen bonds. Additionally, the molecular structure is stabilized by an intramolecular N—H···O hydrogen bond.

Related literature

For the synthesis of isatin-3-tosylhydrazone, see: Cava *et al.* (1958). For the antifungal and antibacterial properties of isatin derivatives, including the title compound, see: Chohan *et al.* (2004).



Experimental

Crystal data

$C_{15}H_{13}N_3O_3S$
M_r = 315.35
Monoclinic, $P2_1/c$
a = 14.9050 (3) Å
b = 5.7849 (1) Å
c = 17.8112 (3) Å
β = 110.427 (1)°
V = 1439.18 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 293 K
0.56 × 0.16 × 0.10 mm

Data collection

Bruker X8 APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.877, T_{max} = 0.976
15630 measured reflections
4204 independent reflections
3146 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.113
S = 1.05
4204 reflections
208 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H6···O1	0.83 (2)	2.08 (2)	2.7539 (17)	138.3 (19)
N1—H1···O1^{i}	0.86 (2)	2.04 (2)	2.9029 (16)	172.9 (18)

Symmetry code: (i) $-x, -y+2, -z$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2252).

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supplementary materials

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4-Methyl-*N*'-(2-oxoindolin-3-ylidene)benzene-1-sulfonohydrazide

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Comment

Isatin derivatives have a wide range of biological properties. For example, isatin-based hydrazones show pharmacological activity against bacteria and fungi (Chohan *et al.*, 2004). As part of our study of isatin derivatives, we report herein the crystal structure of isatin-3-tosylhydrazone. In the title compound (Fig. 1) the C—S—N(H)—N linkage is non-planar with the torsion angle being 65.12 (13)° and a tetrahedral environment suggests a sp^3 hybridization for the S atom. The title structure contains additionally two planar fragments. The mean deviations from the least squares planes for the isatin-derivative fragment C1/C2/C3/C4/C5/C6/C7/C8/N1/N2/N3/O1 and for the tolyl fragment C9/C10/C11/C12/C13/C14/C15 amount to 0.0813 (13)° for C2 and 0.0094 (16)° for C11 atoms, respectively, with a dihedral angle of 80.48 (3)°. The crystal packing is stabilized by intermolecular N—H···O (Table 1; N1—H1···O1^{i}) and also by intramolecular N—H···O bonds (Table 1; N3—H6···O1) leading the isatin-3-tosylhydrazone dimers (Fig. 2). Symmetry code: (i)-*x*, -*y* + 2, -*z*.

Experimental

Starting materials were commercially available and were used without further purification. The synthesis was adapted from a procedure reported previously (Cava *et al.*, 1958). The glacial acetic acid catalyzed reaction of isatin (5 mmol) and *p*-toluenesulfonylhydrazine (5 mmol) in methanol (60 ml) was refluxed for 5 h. After cooling and filtering, crystals suitable for X-ray diffraction were obtained.

Refinement

H atoms attached to C atoms were positioned with idealized geometry and were refined isotropic with U_{eq}(H) set to 1.2 times of the U_{eq}(C) for the aromatic and 1.5 times of the U_{eq}(C) for methyl H atoms using a riding model with C—H = 0.93 Å and C—H = 0.96 Å, respectively. H atoms attached to N atoms were located in difference Fourier maps and included in the subsequent refinement using restraints (N1—H1 = 0.86 (2) Å and N3—H6 = 0.83 (2) Å) with U_{iso}(H) = 1.2 times of the U_{eq}(N). In the last stage of refinement, they were treated as riding on their parent N atoms.

Figures

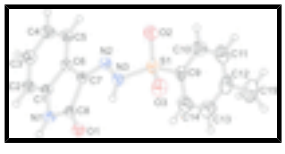

Fig. 1. : The molecular structure of the title compound with labeling and displacement ellipsoids drawn at the 40% probability level.

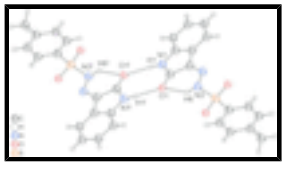

Fig. 2. : Crystal structure of the title compound showing the dimers. Intermolecular hydrogen bonding is indicated as dashed lines. Symmetry code: (i)-*x*, -*y* + 2, -*z*.

4-Methyl-*N*'-(2-oxoindolin-3-ylidene)benzene-1-sulfonohydrazide

Crystal data

$C_{15}H_{13}N_3O_3S$	$F(000) = 656$
$M_r = 315.35$	$D_x = 1.455$ Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 476 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 14.9050$ (3) Å	Cell parameters from 4508 reflections
$b = 5.7849$ (1) Å	$\theta = 2.9$–$26.0°$
$c = 17.8112$ (3) Å	$\mu = 0.24$ mm^{-1}
$\beta = 110.427$ (1)°	$T = 293$ K
$V = 1439.18$ (5) Å^3	Block, yellow
$Z = 4$	0.56 × 0.16 × 0.10 mm

Data collection

Bruker X8 APEXII CCD area-detector diffractometer	4204 independent reflections
Radiation source: fine-focus sealed tube, Bruker X8 APEXII	3146 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.026$
φ and ω scans	$\theta_{max} = 30.1°$, $\theta_{min} = 2.9°$
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	$h = -20\rightarrow21$
$T_{min} = 0.877$, $T_{max} = 0.976$	$k = -8\rightarrow5$
15630 measured reflections	$l = -25\rightarrow23$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.042$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.113$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.05$	$w = 1/[\sigma^2(F_o^2) + (0.0476P)^2 + 0.4436P]$ where $P = (F_o^2 + 2F_c^2)/3$
4204 reflections	$(\Delta/\sigma)_{max} = 0.001$
208 parameters	$\Delta\rho_{max} = 0.28$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.44$ e Å^{-3}

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}
C3	0.11277 (11)	1.0172 (3)	0.32467 (9)	0.0394 (4)
H3	0.1057	1.1188	0.3627	0.047*
C11	0.53807 (13)	0.3670 (4)	0.17689 (12)	0.0566 (5)
H8	0.5908	0.3119	0.2187	0.068*
C12	0.54616 (13)	0.5683 (4)	0.13898 (12)	0.0527 (5)
C10	0.45279 (12)	0.2441 (3)	0.15401 (11)	0.0490 (4)
H7	0.4482	0.1086	0.1805	0.059*
C13	0.46719 (15)	0.6454 (4)	0.07640 (14)	0.0606 (5)
H9	0.4717	0.7815	0.0501	0.073*
C14	0.38184 (13)	0.5253 (3)	0.05197 (12)	0.0540 (5)
H10	0.3296	0.5786	0.0094	0.065*
C15	0.63816 (15)	0.7038 (5)	0.16498 (16)	0.0760 (7)
H11	0.6298	0.8449	0.1901	0.114*
H12	0.6553	0.7391	0.1191	0.114*
H13	0.6881	0.6138	0.2023	0.114*
H6	0.1551 (15)	0.431 (4)	0.0260 (12)	0.059 (6)*
H1	0.0168 (14)	1.043 (4)	0.0776 (12)	0.055 (6)*
S1	0.26748 (3)	0.16782 (7)	0.06008 (2)	0.03535 (12)
N3	0.18364 (9)	0.3472 (2)	0.06478 (8)	0.0352 (3)
N2	0.19062 (8)	0.4241 (2)	0.13897 (7)	0.0320 (3)
C8	0.08200 (10)	0.7533 (3)	0.07301 (8)	0.0305 (3)
O1	0.06596 (8)	0.71841 (19)	0.00121 (6)	0.0382 (3)
C9	0.37519 (11)	0.3244 (3)	0.09182 (9)	0.0347 (3)
N1	0.05136 (9)	0.9330 (2)	0.10573 (7)	0.0329 (3)
C7	0.14177 (10)	0.6049 (2)	0.14176 (8)	0.0292 (3)
C1	0.08633 (10)	0.9203 (2)	0.19011 (8)	0.0295 (3)
O3	0.23997 (9)	0.1214 (2)	−0.02344 (7)	0.0511 (3)
C5	0.17706 (11)	0.6607 (3)	0.29508 (9)	0.0353 (3)
H5	0.2115	0.5247	0.3121	0.042*
C2	0.07358 (11)	1.0743 (3)	0.24401 (9)	0.0351 (3)
H2	0.0400	1.2115	0.2272	0.042*
C6	0.13941 (10)	0.7169 (2)	0.21428 (8)	0.0298 (3)
C4	0.16212 (11)	0.8120 (3)	0.34970 (9)	0.0398 (4)
H4	0.1855	0.7755	0.4040	0.048*
O2	0.27543 (8)	−0.0145 (2)	0.11553 (8)	0.0481 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C3	0.0415 (8)	0.0398 (9)	0.0366 (8)	0.0005 (7)	0.0132 (7)	−0.0057 (7)
C11	0.0392 (9)	0.0670 (13)	0.0552 (11)	−0.0021 (9)	0.0060 (8)	−0.0073 (10)
C12	0.0403 (9)	0.0587 (12)	0.0630 (12)	−0.0114 (8)	0.0231 (8)	−0.0226 (10)
C10	0.0454 (9)	0.0506 (10)	0.0469 (10)	−0.0021 (8)	0.0109 (8)	0.0040 (8)
C13	0.0556 (12)	0.0479 (11)	0.0792 (15)	−0.0130 (9)	0.0247 (10)	0.0044 (10)
C14	0.0423 (9)	0.0485 (10)	0.0646 (12)	−0.0034 (8)	0.0105 (8)	0.0089 (9)
C15	0.0501 (12)	0.0853 (17)	0.0963 (18)	−0.0272 (12)	0.0300 (12)	−0.0305 (14)
S1	0.0344 (2)	0.0308 (2)	0.0424 (2)	−0.00097 (15)	0.01533 (16)	−0.00615 (15)
N3	0.0354 (7)	0.0364 (7)	0.0338 (7)	0.0062 (5)	0.0122 (5)	0.0009 (6)
N2	0.0333 (6)	0.0304 (6)	0.0335 (6)	0.0027 (5)	0.0133 (5)	0.0016 (5)
C8	0.0282 (6)	0.0315 (7)	0.0314 (7)	0.0007 (6)	0.0101 (5)	0.0068 (6)
O1	0.0424 (6)	0.0417 (6)	0.0294 (5)	0.0047 (5)	0.0111 (4)	0.0051 (5)
C9	0.0322 (7)	0.0333 (8)	0.0411 (8)	−0.0007 (6)	0.0158 (6)	−0.0063 (6)
N1	0.0360 (6)	0.0308 (6)	0.0313 (6)	0.0080 (5)	0.0109 (5)	0.0087 (5)
C7	0.0283 (6)	0.0292 (7)	0.0303 (7)	0.0008 (5)	0.0103 (5)	0.0059 (6)
C1	0.0287 (6)	0.0283 (7)	0.0317 (7)	0.0007 (5)	0.0107 (5)	0.0053 (6)
O3	0.0516 (7)	0.0557 (8)	0.0466 (7)	−0.0074 (6)	0.0177 (6)	−0.0220 (6)
C5	0.0342 (7)	0.0371 (8)	0.0319 (7)	0.0068 (6)	0.0081 (6)	0.0063 (6)
C2	0.0364 (7)	0.0288 (7)	0.0400 (8)	0.0040 (6)	0.0134 (6)	0.0024 (6)
C6	0.0288 (6)	0.0295 (7)	0.0315 (7)	0.0033 (5)	0.0109 (5)	0.0036 (6)
C4	0.0397 (8)	0.0460 (9)	0.0293 (7)	0.0023 (7)	0.0066 (6)	0.0014 (7)
O2	0.0449 (6)	0.0336 (6)	0.0689 (8)	0.0029 (5)	0.0239 (6)	0.0083 (6)

Geometric parameters (Å, °)

C3—C4	1.385 (2)	S1—N3	1.6488 (13)
C3—C2	1.389 (2)	S1—C9	1.7560 (15)
C3—H3	0.9300	N3—N2	1.3636 (17)
C11—C12	1.372 (3)	N3—H6	0.83 (2)
C11—C10	1.388 (3)	N2—C7	1.2853 (18)
C11—H8	0.9300	C8—O1	1.2329 (17)
C12—C13	1.383 (3)	C8—O1	1.2329 (17)
C12—C15	1.505 (3)	C8—N1	1.3471 (19)
C10—C9	1.373 (2)	C8—C7	1.5060 (19)
C10—H7	0.9300	N1—C1	1.4103 (18)
C13—C14	1.380 (3)	N1—H1	0.86 (2)
C13—H9	0.9300	C7—C6	1.456 (2)
C14—C9	1.383 (2)	C1—C2	1.371 (2)
C14—H10	0.9300	C1—C6	1.3991 (19)
C15—H11	0.9600	C5—C4	1.383 (2)
C15—H12	0.9600	C5—C6	1.3886 (19)
C15—H13	0.9600	C5—H5	0.9300
S1—O2	1.4212 (12)	C2—H2	0.9300
S1—O3	1.4239 (12)	C4—H4	0.9300

C4—C3—C2	121.40 (15)	S1—N3—H6	120.1 (14)
C4—C3—H3	119.3	C7—N2—N3	116.77 (12)
C2—C3—H3	119.3	O1—C8—N1	127.08 (13)
C12—C11—C10	121.27 (18)	O1—C8—N1	127.08 (13)
C12—C11—H8	119.4	O1—C8—C7	126.55 (14)
C10—C11—H8	119.4	O1—C8—C7	126.55 (14)
C11—C12—C13	118.32 (17)	N1—C8—C7	106.37 (12)
C11—C12—C15	121.2 (2)	C10—C9—C14	120.46 (16)
C13—C12—C15	120.5 (2)	C10—C9—S1	120.21 (13)
C9—C10—C11	119.39 (18)	C14—C9—S1	119.30 (13)
C9—C10—H7	120.3	C8—N1—C1	111.45 (12)
C11—C10—H7	120.3	C8—N1—H1	122.9 (13)
C14—C13—C12	121.56 (19)	C1—N1—H1	125.6 (13)
C14—C13—H9	119.2	N2—C7—C6	125.84 (13)
C12—C13—H9	119.2	N2—C7—C8	127.94 (13)
C13—C14—C9	118.99 (18)	C6—C7—C8	106.08 (12)
C13—C14—H10	120.5	C2—C1—C6	122.19 (13)
C9—C14—H10	120.5	C2—C1—N1	128.49 (13)
C12—C15—H11	109.5	C6—C1—N1	109.32 (12)
C12—C15—H12	109.5	C4—C5—C6	118.32 (14)
H11—C15—H12	109.5	C4—C5—H5	120.8
C12—C15—H13	109.5	C6—C5—H5	120.8
H11—C15—H13	109.5	C1—C2—C3	117.32 (14)
H12—C15—H13	109.5	C1—C2—H2	121.3
O2—S1—O3	120.64 (8)	C3—C2—H2	121.3
O2—S1—N3	108.22 (7)	C5—C6—C1	119.77 (13)
O3—S1—N3	102.99 (7)	C5—C6—C7	133.54 (13)
O2—S1—C9	108.30 (7)	C1—C6—C7	106.69 (12)
O3—S1—C9	109.34 (8)	C5—C4—C3	120.90 (14)
N3—S1—C9	106.44 (7)	C5—C4—H4	119.5
N2—N3—S1	116.86 (10)	C3—C4—H4	119.5
N2—N3—H6	117.5 (14)		

C10—C11—C12—C13	−0.7 (3)	N3—N2—C7—C6	−179.42 (13)
C10—C11—C12—C15	178.77 (19)	N3—N2—C7—C8	−4.3 (2)
C12—C11—C10—C9	0.5 (3)	O1—C8—C7—N2	4.0 (2)
C11—C12—C13—C14	0.1 (3)	O1—C8—C7—N2	4.0 (2)
C15—C12—C13—C14	−179.4 (2)	N1—C8—C7—N2	−174.84 (14)
C12—C13—C14—C9	0.7 (3)	O1—C8—C7—C6	179.91 (14)
O2—S1—N3—N2	51.10 (13)	O1—C8—C7—C6	179.91 (14)
O3—S1—N3—N2	179.90 (11)	N1—C8—C7—C6	1.06 (15)
C9—S1—N3—N2	−65.12 (13)	C8—N1—C1—C2	177.47 (14)
S1—N3—N2—C7	162.65 (11)	C8—N1—C1—C6	−2.60 (16)
N1—C8—O1—O1	0.00 (10)	C6—C1—C2—C3	−2.3 (2)
C7—C8—O1—O1	0.00 (14)	N1—C1—C2—C3	177.61 (14)
C11—C10—C9—C14	0.3 (3)	C4—C3—C2—C1	−0.5 (2)
C11—C10—C9—S1	178.32 (14)	C4—C5—C6—C1	−1.2 (2)
C13—C14—C9—C10	−0.9 (3)	C4—C5—C6—C7	178.95 (15)
C13—C14—C9—S1	−178.95 (16)	C2—C1—C6—C5	3.2 (2)
O2—S1—C9—C10	8.34 (16)	N1—C1—C6—C5	−176.70 (13)

O3—S1—C9—C10	−124.90 (15)	C2—C1—C6—C7	−176.92 (13)
N3—S1—C9—C10	124.50 (14)	N1—C1—C6—C7	3.15 (15)
O2—S1—C9—C14	−173.62 (14)	N2—C7—C6—C5	−6.7 (3)
O3—S1—C9—C14	53.14 (16)	C8—C7—C6—C5	177.26 (16)
N3—S1—C9—C14	−57.46 (15)	N2—C7—C6—C1	173.44 (14)
O1—C8—N1—C1	−177.97 (14)	C8—C7—C6—C1	−2.56 (15)
O1—C8—N1—C1	−177.97 (14)	C6—C5—C4—C3	−1.5 (2)
C7—C8—N1—C1	0.88 (16)	C2—C3—C4—C5	2.4 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H6···O1	0.83 (2)	2.08 (2)	2.7539 (17)	138.3 (19)
N1—H1···O1^{i}	0.86 (2)	2.04 (2)	2.9029 (16)	172.9 (18)

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

Fig. 1

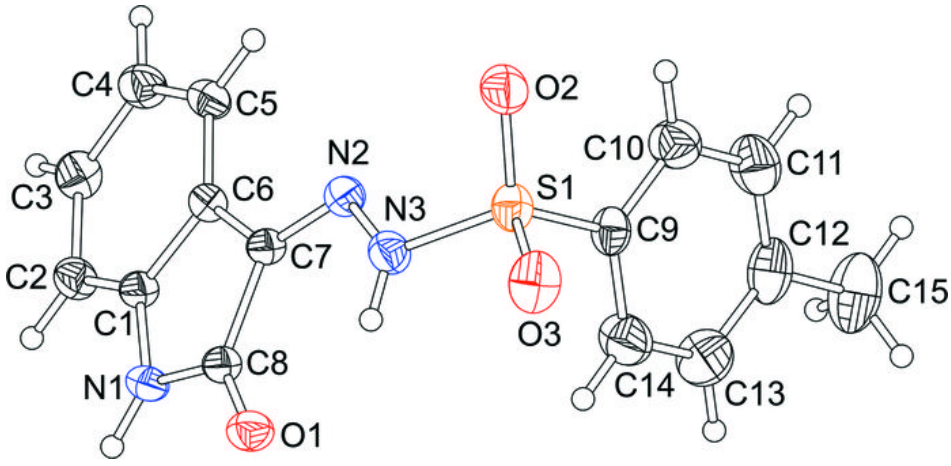
C4
C5
O2
C10
C11
N2
C3
C6
N3
S1
C9
C12
C2
C7
C15
C1
O3
C8
N1
C14
C13
O1

Fig. 2