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(*E*)-*N*′-(4-Pyridylmethylene)-2-(quinolin-8-yloxy)acetohydrazide sesquihydrate

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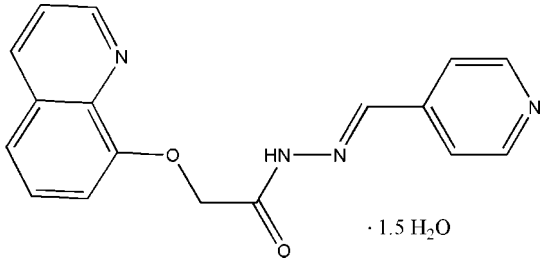
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.003 Å; R factor = 0.045; wR factor = 0.113; data-to-parameter ratio = 13.3.

In the title compound, $C_{17}H_{14}N_4O_2\cdot1.5H_2O$, the mean planes of the pyridine ring and quinoline group make a dihedral angle of 21.0 (2)°. One water molecule lies on a twofold rotation axis. The organic molecules and the three water molecules are linked into infinite chains by N—H···O, O—H···O and O—H···N hydrogen bonds.

Related literature

For related literature, see: Chen & Shi (1998); Moawad & Hanna (2002).



Experimental

Crystal data

$C_{17}H_{14}N_4O_2\cdot1.5H_2O$
M_r = 333.35
Monoclinic, $C2/c$
a = 16.3206 (19) Å
b = 15.6706 (19) Å
c = 13.5692 (16) Å
β = 105.623 (3)°
V = 3342.2 (7) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 295 K
0.26 × 0.22 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.976, T_{max} = 0.983
8726 measured reflections
2962 independent reflections
1974 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.113
S = 1.00
2962 reflections
222 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H19···N4^{i}	0.85	2.14	2.914 (2)	151
N2—H2···O3	0.86	2.10	2.890 (2)	153
O3—H18···N1	0.85	1.91	2.7582 (19)	175
O4—H20···O3^{ii}	0.85	2.48	2.825 (2)	105

Symmetry codes: (i) $-x-\frac{1}{2}, -y+\frac{3}{2}, -z$; (ii) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); 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: RK2067).

References

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supplementary materials

Acta Cryst. (2008). E**64**, o297 [**doi:10.1107/S1600536807066664**]

(*E*)-*N*'-(4-Pyridylmethylene)-2-(quinolin-8-yloxy)acetohydrazide sesquihydrate

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Comment

8–Hydroxyquinoline and its derivatives are of the most extensively investigated ligands in the coordination chemistry (Chen & Shi, 1998; Moawad & Hanna, 2002). In course of our studies on searching for good extractants of metal ions or a biologically active material, the title compound, (I), was synthesized and its crystal structure determined. The conformation along the O1–C10–C11–N2–N3–C12–C13 bond sequence is (-)*gauche–trans–trans–trans* (Fig.1). The mean planes of the pyridine ring and quinoline group make a dihedral angle of the molecules are 21.0 (2)°. The two molecules and the three water molecules are linked into infinite chains by N—H···O, O—H···O and O—H···N hydrogen bonds (Fig. 2).

Experimental

2–(Quinolin–8–yl-oxy)acetohydrazide (0.01 mol), 4–pyridylaldehyde (0.01 mol), ethanol (40 ml) and three drops of acetic acid were added to a 100 ml flask, and refluxed for 6 h. After cooling to room temperature, the mixture was filtered. Colourless single crystals suitable for *X*–ray diffraction study were obtained by slow evaporation of a tetrahydrofuran solution over a period of 1 d. (m.p. 361 K). Elemental analysis calc.: C 61.25, H 5.14, N 16.80%; found: C 61.19, H 5.21, N 16.77%.

Refinement

All H atoms were placed in idealized calculated positions with C—H = 0.93–0.97 Å, O—H = 0.85 Å, N—H = 0.86Å and refined as riding atoms with U_{iso}(H) = 1.2U_{eq}(C or N) and U_{iso}(H) = 1.5U_{eq}(O).

Figures

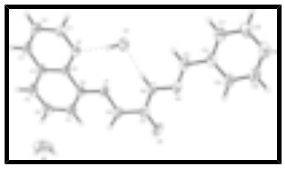

Fig. 1. The molecular structure of the title compound with the numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as small spheres of arbitrary radius. The dashed lines indicate hydrogen bonds.

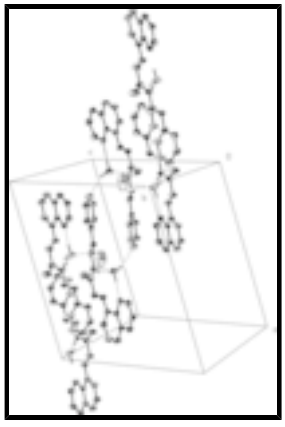

Fig. 2. The infinite chains structure *via* hydrogen bonds in the title compound. The dashed lines indicate hydrogen bonds. H atoms not involved in H bonds have been omitted for clarity.

(*E*)-*N*′-(4-Pyridylmethylene)-2-(quinolin-8-yloxy)acetohydrazide sesquihydrate

Crystal data

$C_{17}H_{14}N_4O_2 \cdot 1.5H_2O$	F_{000} = 1400
M_r = 333.35	D_x = 1.325 Mg m^{-3}
Monoclinic, *C*2/*c*	Melting point: 361 K
Hall symbol: -c 2yc	Mo *K*α radiation λ = 0.71073 Å
a = 16.3206 (19) Å	Cell parameters from 1750 reflections
b = 15.6706 (19) Å	θ = 2.6–22.1º
c = 13.5692 (16) Å	μ = 0.10 mm^{-1}
β = 105.623 (3)º	*T* = 295 K
V = 3342.2 (7) Å^3	Block, colorless
Z = 8	0.26 × 0.22 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer	2962 independent reflections
Radiation source: fine–focus sealed tube	1974 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.034
T = 295 K	θ_{max} = 25.1º
φ and ω scans	θ_{min} = 1.8º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	*h* = −18→19
T_{min} = 0.976, T_{max} = 0.983	*k* = −15→18
8726 measured reflections	*l* = −16→15

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.045	H-atom parameters constrained

$wR(F^2) = 0.113$	$w = 1/[\sigma^2(F_o^2) + (0.0416P)^2 + 1.6836P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{max} < 0.001$
2962 reflections	$\Delta\rho_{max} = 0.23$ e Å^{-3}
222 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 $F^2 > 2\sigma(F^2)$ 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}
O1	0.15472 (7)	0.61002 (8)	0.13430 (10)	0.0522 (4)
O2	−0.00480 (9)	0.45270 (10)	0.11135 (14)	0.0848 (6)
O3	0.03989 (8)	0.77138 (9)	0.10648 (12)	0.0679 (5)
H18	0.0924	0.7680	0.1089	0.102*
H19	0.0139	0.7832	0.0448	0.102*
O4	0.5000	0.38335 (19)	0.2500	0.198 (2)
H20	0.4541	0.3547	0.2364	0.297*
N1	0.21216 (9)	0.76870 (10)	0.12366 (12)	0.0480 (4)
N2	−0.01021 (9)	0.59689 (11)	0.12749 (13)	0.0563 (5)
H2	0.0178	0.6440	0.1386	0.068*
N3	−0.09687 (10)	0.59595 (12)	0.11520 (13)	0.0586 (5)
N4	−0.39872 (12)	0.69428 (16)	0.07266 (16)	0.0784 (6)
C1	0.24172 (13)	0.84681 (13)	0.12079 (16)	0.0570 (6)
H1	0.2036	0.8919	0.1147	0.068*
C2	0.32621 (13)	0.86644 (15)	0.12628 (16)	0.0621 (6)
H2A	0.3437	0.9228	0.1248	0.074*
C3	0.38204 (13)	0.80125 (15)	0.13380 (16)	0.0596 (6)
H3	0.4386	0.8127	0.1370	0.071*
C4	0.35495 (11)	0.71610 (13)	0.13688 (15)	0.0504 (5)
C5	0.40959 (12)	0.64574 (16)	0.14487 (17)	0.0642 (6)
H5	0.4662	0.6543	0.1460	0.077*
C6	0.38064 (13)	0.56572 (16)	0.15098 (18)	0.0671 (6)
H6A	0.4179	0.5198	0.1576	0.081*
C7	0.29443 (12)	0.55081 (14)	0.14739 (16)	0.0591 (6)
H7	0.2753	0.4954	0.1514	0.071*

C8	0.23931 (11)	0.61774 (13)	0.13810 (14)	0.0463 (5)
C9	0.26813 (11)	0.70304 (12)	0.13252 (13)	0.0429 (5)
C10	0.12326 (11)	0.52542 (12)	0.13095 (17)	0.0561 (6)
H10A	0.1537	0.4956	0.1926	0.067*
H10B	0.1344	0.4959	0.0730	0.067*
C11	0.02969 (13)	0.52209 (15)	0.12202 (16)	0.0577 (6)
C12	−0.12998 (13)	0.66627 (15)	0.13033 (16)	0.0575 (6)
H12	−0.0953	0.7134	0.1524	0.069*
C13	−0.22215 (12)	0.67417 (14)	0.11357 (15)	0.0549 (5)
C14	−0.27696 (13)	0.60665 (16)	0.07901 (19)	0.0730 (7)
H14	−0.2559	0.5532	0.0688	0.088*
C15	−0.36330 (15)	0.62007 (19)	0.0599 (2)	0.0873 (8)
H15	−0.3993	0.5741	0.0365	0.105*
C16	−0.34569 (15)	0.75802 (17)	0.10755 (17)	0.0701 (7)
H16	−0.3684	0.8105	0.1185	0.084*
C17	−0.25845 (14)	0.75065 (15)	0.12848 (15)	0.0617 (6)
H17	−0.2240	0.7976	0.1528	0.074*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0346 (7)	0.0500 (8)	0.0732 (10)	−0.0113 (6)	0.0166 (6)	0.0027 (7)
O2	0.0579 (10)	0.0641 (11)	0.1351 (16)	−0.0255 (8)	0.0306 (10)	0.0071 (10)
O3	0.0428 (8)	0.0768 (10)	0.0849 (11)	−0.0025 (7)	0.0187 (7)	0.0064 (8)
O4	0.389 (7)	0.066 (2)	0.178 (4)	0.000	0.143 (4)	0.000
N1	0.0389 (9)	0.0527 (10)	0.0519 (10)	−0.0096 (8)	0.0112 (7)	0.0016 (8)
N2	0.0345 (9)	0.0646 (12)	0.0699 (12)	−0.0164 (8)	0.0143 (8)	0.0004 (9)
N3	0.0357 (9)	0.0750 (13)	0.0656 (12)	−0.0128 (9)	0.0143 (8)	0.0074 (10)
N4	0.0464 (12)	0.1033 (17)	0.0859 (15)	0.0024 (12)	0.0187 (10)	0.0101 (13)
C1	0.0543 (13)	0.0545 (13)	0.0609 (14)	−0.0101 (10)	0.0135 (10)	0.0021 (10)
C2	0.0570 (14)	0.0624 (14)	0.0675 (15)	−0.0236 (11)	0.0179 (11)	−0.0018 (11)
C3	0.0423 (12)	0.0770 (16)	0.0602 (14)	−0.0246 (12)	0.0151 (10)	−0.0027 (12)
C4	0.0366 (11)	0.0681 (14)	0.0476 (12)	−0.0130 (10)	0.0130 (9)	−0.0041 (10)
C5	0.0331 (11)	0.0861 (17)	0.0747 (16)	−0.0066 (11)	0.0166 (10)	−0.0016 (13)
C6	0.0426 (12)	0.0750 (16)	0.0851 (17)	0.0079 (11)	0.0192 (11)	0.0010 (13)
C7	0.0464 (12)	0.0551 (13)	0.0758 (16)	−0.0032 (10)	0.0164 (11)	0.0019 (11)
C8	0.0311 (10)	0.0576 (12)	0.0498 (12)	−0.0081 (9)	0.0103 (9)	0.0003 (9)
C9	0.0338 (10)	0.0547 (12)	0.0400 (11)	−0.0094 (9)	0.0099 (8)	−0.0001 (9)
C10	0.0401 (11)	0.0504 (12)	0.0775 (15)	−0.0104 (9)	0.0150 (10)	0.0061 (11)
C11	0.0464 (12)	0.0593 (14)	0.0683 (15)	−0.0143 (11)	0.0171 (11)	0.0073 (11)
C12	0.0435 (12)	0.0731 (16)	0.0559 (14)	−0.0153 (11)	0.0137 (10)	0.0025 (11)
C13	0.0406 (11)	0.0760 (15)	0.0486 (12)	−0.0087 (11)	0.0130 (9)	0.0102 (11)
C14	0.0436 (13)	0.0765 (16)	0.0971 (19)	−0.0079 (12)	0.0161 (12)	0.0066 (14)
C15	0.0456 (15)	0.092 (2)	0.120 (2)	−0.0133 (13)	0.0158 (14)	0.0047 (17)
C16	0.0623 (15)	0.0892 (18)	0.0631 (15)	0.0073 (14)	0.0243 (12)	0.0065 (13)
C17	0.0587 (14)	0.0788 (16)	0.0503 (13)	−0.0065 (12)	0.0192 (11)	0.0026 (11)

Geometric parameters (Å, °)

O1—C8	1.373 (2)	C4—C9	1.417 (2)
O1—C10	1.418 (2)	C5—C6	1.351 (3)
O2—C11	1.215 (2)	C5—H5	0.9300
O3—H18	0.8499	C6—C7	1.414 (3)
O3—H19	0.8500	C6—H6A	0.9300
O4—H20	0.8501	C7—C8	1.365 (3)
N1—C1	1.320 (2)	C7—H7	0.9300
N1—C9	1.360 (2)	C8—C9	1.426 (3)
N2—C11	1.353 (3)	C10—C11	1.500 (3)
N2—N3	1.379 (2)	C10—H10A	0.9700
N2—H2	0.8598	C10—H10B	0.9700
N3—C12	1.268 (3)	C12—C13	1.465 (3)
N4—C16	1.323 (3)	C12—H12	0.9300
N4—C15	1.331 (3)	C13—C17	1.376 (3)
C1—C2	1.395 (3)	C13—C14	1.383 (3)
C1—H1	0.9300	C14—C15	1.378 (3)
C2—C3	1.354 (3)	C14—H14	0.9300
C2—H2A	0.9300	C15—H15	0.9300
C3—C4	1.410 (3)	C16—C17	1.380 (3)
C3—H3	0.9300	C16—H16	0.9300
C4—C5	1.404 (3)	C17—H17	0.9300
C8—O1—C10	115.81 (15)	O1—C8—C9	115.05 (16)
H18—O3—H19	106.4	N1—C9—C4	122.34 (17)
C1—N1—C9	117.52 (16)	N1—C9—C8	119.39 (15)
C11—N2—N3	118.52 (17)	C4—C9—C8	118.27 (17)
C11—N2—H2	120.8	O1—C10—C11	112.77 (17)
N3—N2—H2	120.6	O1—C10—H10A	109.0
C12—N3—N2	116.05 (17)	C11—C10—H10A	109.0
C16—N4—C15	116.2 (2)	O1—C10—H10B	109.0
N1—C1—C2	124.5 (2)	C11—C10—H10B	109.0
N1—C1—H1	117.7	H10A—C10—H10B	107.8
C2—C1—H1	117.7	O2—C11—N2	124.59 (19)
C3—C2—C1	118.2 (2)	O2—C11—C10	118.0 (2)
C3—C2—H2A	120.9	N2—C11—C10	117.40 (18)
C1—C2—H2A	120.9	N3—C12—C13	120.9 (2)
C2—C3—C4	120.42 (18)	N3—C12—H12	119.6
C2—C3—H3	119.8	C13—C12—H12	119.6
C4—C3—H3	119.8	C17—C13—C14	116.83 (19)
C5—C4—C3	123.23 (18)	C17—C13—C12	121.0 (2)
C5—C4—C9	119.81 (18)	C14—C13—C12	122.1 (2)
C3—C4—C9	116.96 (19)	C15—C14—C13	118.8 (2)
C6—C5—C4	120.57 (19)	C15—C14—H14	120.6
C6—C5—H5	119.7	C13—C14—H14	120.6
C4—C5—H5	119.7	N4—C15—C14	124.5 (2)
C5—C6—C7	120.8 (2)	N4—C15—H15	117.7
C5—C6—H6A	119.6	C14—C15—H15	117.7

C7—C6—H6A	119.6	N4—C16—C17	123.4 (2)
C8—C7—C6	120.1 (2)	N4—C16—H16	118.3
C8—C7—H7	120.0	C17—C16—H16	118.3
C6—C7—H7	120.0	C13—C17—C16	120.3 (2)
C7—C8—O1	124.51 (17)	C13—C17—H17	119.9
C7—C8—C9	120.43 (17)	C16—C17—H17	119.9
C11—N2—N3—C12	173.72 (19)	C7—C8—C9—N1	−179.76 (18)
C9—N1—C1—C2	0.0 (3)	O1—C8—C9—N1	−0.3 (3)
N1—C1—C2—C3	0.9 (3)	C7—C8—C9—C4	0.0 (3)
C1—C2—C3—C4	−0.6 (3)	O1—C8—C9—C4	179.47 (16)
C2—C3—C4—C5	−179.8 (2)	C8—O1—C10—C11	−177.36 (17)
C2—C3—C4—C9	−0.4 (3)	N3—N2—C11—O2	−3.4 (3)
C3—C4—C5—C6	177.7 (2)	N3—N2—C11—C10	177.31 (17)
C9—C4—C5—C6	−1.6 (3)	O1—C10—C11—O2	174.94 (19)
C4—C5—C6—C7	1.3 (4)	O1—C10—C11—N2	−5.7 (3)
C5—C6—C7—C8	−0.3 (4)	N2—N3—C12—C13	176.33 (17)
C6—C7—C8—O1	−179.76 (19)	N3—C12—C13—C17	−179.1 (2)
C6—C7—C8—C9	−0.3 (3)	N3—C12—C13—C14	−1.2 (3)
C10—O1—C8—C7	−5.7 (3)	C17—C13—C14—C15	1.3 (3)
C10—O1—C8—C9	174.83 (16)	C12—C13—C14—C15	−176.6 (2)
C1—N1—C9—C4	−1.1 (3)	C16—N4—C15—C14	−1.0 (4)
C1—N1—C9—C8	178.67 (17)	C13—C14—C15—N4	−0.2 (4)
C5—C4—C9—N1	−179.27 (18)	C15—N4—C16—C17	1.2 (4)
C3—C4—C9—N1	1.3 (3)	C14—C13—C17—C16	−1.1 (3)
C5—C4—C9—C8	1.0 (3)	C12—C13—C17—C16	176.83 (19)
C3—C4—C9—C8	−178.43 (18)	N4—C16—C17—C13	−0.1 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H19···N4^{i}	0.85	2.14	2.914 (2)	151
N2—H2···O3	0.86	2.10	2.890 (2)	153
O3—H18···N1	0.85	1.91	2.7582 (19)	175
O4—H20···O3^{ii}	0.85	2.48	2.825 (2)	105

Symmetry codes: (i) −*x*−1/2, −*y*+3/2, −*z*; (ii) −*x*+1/2, *y*−1/2, −*z*+1/2.

Fig. 1

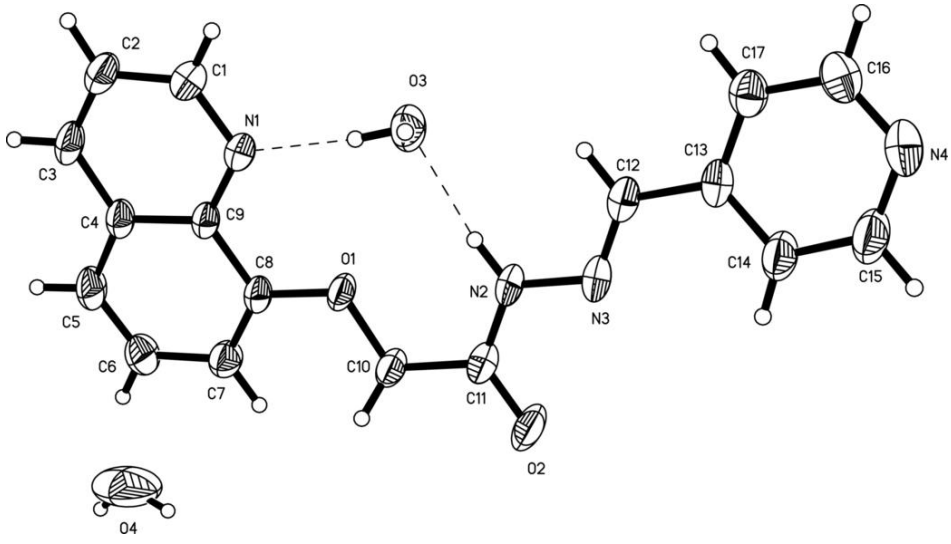
C2
C1
O3
N1
C17
C16
C3
C12
C13
N4
C4
C9
C8
O1
N2
N3
C14
C15
C5
C6
C7
C10
C11
O2
O4

Fig. 2