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3-(4-Phenylbenzoyl)-4,5,6,7-tetrahydro-1,2,3-triazolo[1,5-*a*]pyrimidine

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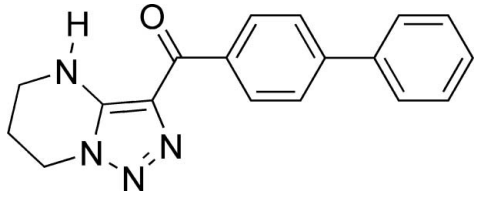
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; R factor = 0.044; wR factor = 0.106; data-to-parameter ratio = 14.4.

In the title compound, $C_{18}H_{16}N_4O$, the dihedral angle between the N_3C_2 ring and the central benzene ring is 12.62 (9)°. The dihedral angle between the central and terminal benzene rings is 36.14 (9)°. In the crystal structure, molecules interact *via* N—H···N hydrogen bonds, resulting in infinite chains. There is also an intramolecular N—H···O hydrogen bond.

Related literature

For background, see: Huang & Wang (1992); Huang & Wang (1994).



Experimental

Crystal data

$C_{18}H_{16}N_4O$
M_r = 304.35
Orthorhombic, *Pbca*
a = 12.923 (3) Å
b = 7.3253 (17) Å
c = 31.765 (7) Å
V = 3007.0 (12) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 294 (2) K
0.24 × 0.20 × 0.14 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.979, T_{max} = 0.988
16020 measured reflections
3066 independent reflections
1804 reflections with $I > 2\sigma(I)$
R_{int} = 0.074

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.106
S = 1.03
3066 reflections
213 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1	0.902 (13)	2.269 (17)	2.833 (2)	120.3 (12)
N1—H1···N3^{i}	0.902 (13)	2.452 (12)	3.152 (2)	134.6 (13)

Symmetry code: (i) $x+\frac{1}{2}, -y+\frac{1}{2}, -z+1$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; 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*.

The authors thank Mr Wang Hong Geng of Nankai University for the X-ray crystallographic determination.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2428).

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supplementary materials

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3-(4-Phenylbenzoyl)-4,5,6,7-tetrahydro-1,2,3-triazolo[1,5-*a*]pyrimidine

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Comment

Heterocyclic ketene aminals, also named cyclic 1,1-enediamines, are useful building blocks in organic synthesis, especially for the synthesis of more complex heterocycles (Huang & Wang, 1994). The title compound, (I) (Fig. 1), belongs to the family of 1, 2,3-triazole fused 1,3-diazoheterocles and its structure is described here.

The dihedral angle between the N_3C_2 ring and the C7—C12 benzene ring is 12.62 (9)°. The dihedral angle between the two bezene rings (C7—C12 and C13—C18) is 36.14 (9)°.

In the crystal, adjacent molecules interact by way of N—H···N hydrogen bonds (Table 1) to result in [100] chains. An intramolecular N—H···O link occurs at the same time.

Experimental

The title compound was prepared according to the procedure reported by Huang & Wang (1992) and purified by recrystallization from ethyl acetate in 82.7% yield; mp 438–440 K; FT—IR (KBr): 3369.03, 1636.30, 1588.09, 1526.38, 1428.03 cm^{-1}.^{1}H NMR ($CDCl_3$, δp.p.m.): 8.60–8.57 (d, 2H, aryl H), 7.74–7.71 (d, 2H, aryl H), 7.67–7.65 (d, 2H, aryl H), 7.49–7.44(t, 2H, aryl H), 7.41–7.36 (t, 1H, aryl H), 6.73 (s, 1H, NH), 4.41–4.36 (t, 2H, CH_2); 3.56–3.51 (m, 2H, CH_2), 2.25–2.18 (m, 2H, CH_2); ^{13}C NMR (CDCl3, δp.p.m.): 185.18, 146.12, 144.95, 140.35, 136.02, 130.60, 128.90, 128.74, 127.98, 127.33, 126.95, 43.23, 38.66, 20.60; MS (EI) m/*z*: 305($[M+H]^+$), 304 ($[M]^+$). Anal. Calcd. For $C_{18}H_{16}N_4O$: C, 71.04; H, 5.30; N, 18.41. Found: C, 70.53; H, 5.23; N, 21.18.50.

Refinement

The N-bound hydrogen atom was located in a difference map and its position and U_{iso} value were freely refined. The C-bound H atoms were geometrically placed (C—H = 0.95–1.00 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C).

Figures

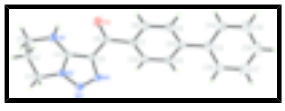

Fig. 1. Molecular structure of (I) showing 30% displacement ellipsoids (arbirary spheres for the H atoms).

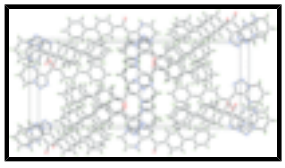

Fig. 2. Packing diagram of (I) viewed down the *a*-axis.

3-(4-Phenylbenzoyl)-4,5,6,7-tetrahydro-1,2,3-triazolo[1,5-*a*]pyrimidine

Crystal data

$C_{18}H_{16}N_4O$	$D_x = 1.345$ Mg m^{-3}
$M_r = 304.35$	Melting point: 438-440 K
Orthorhombic, *Pbca*	Mo *K*α radiation λ = 0.71073 Å
$a = 12.923$ (3) Å	Cell parameters from 2403 reflections
$b = 7.3253$ (17) Å	θ = 3.2–21.2º
$c = 31.765$ (7) Å	μ = 0.09 mm^{-1}
$V = 3007.0$ (12) Å^3	$T = 294$ (2) K
$Z = 8$	Prism, colourless
$F_{000} = 1280$	0.24 × 0.20 × 0.14 mm

Data collection

Bruker SMART CCD diffractometer	3066 independent reflections
Radiation source: fine-focus sealed tube	1804 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.074$
$T = 294$(2) K	$\theta_{max} = 26.4$º
ω scans	$\theta_{min} = 2.0$º
Absorption correction: multi-scan (SADABS; Bruker, 1997)	$h = -16 \rightarrow 16$
$T_{min} = 0.979$, $T_{max} = 0.988$	$k = -9 \rightarrow 9$
16020 measured reflections	$l = -26 \rightarrow 39$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.044$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.106$	$w = 1/[\sigma^2(F_o^2) + (0.0465P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} = 0.001$
3066 reflections	$\Delta\rho_{max} = 0.17$ e Å^{-3}
213 parameters	$\Delta\rho_{min} = -0.13$ e Å^{-3}
1 restraint	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0087 (8)

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}
O1	0.27429 (9)	0.3111 (2)	0.43106 (4)	0.0629 (4)
N1	0.20844 (11)	0.2160 (2)	0.51324 (5)	0.0447 (4)
H1	0.2688 (9)	0.216 (3)	0.4990 (5)	0.064 (6)*
N2	0.02806 (9)	0.23953 (18)	0.50904 (4)	0.0361 (4)
N3	−0.04815 (10)	0.2859 (2)	0.48069 (5)	0.0441 (4)
N4	−0.00221 (10)	0.3245 (2)	0.44527 (5)	0.0412 (4)
C1	0.20019 (13)	0.1494 (3)	0.55610 (5)	0.0438 (5)
H1A	0.2627	0.1791	0.5715	0.053*
H1B	0.1923	0.0177	0.5560	0.053*
C2	0.10745 (12)	0.2368 (3)	0.57734 (5)	0.0422 (5)
H2A	0.1179	0.3677	0.5791	0.051*
H2B	0.1010	0.1899	0.6058	0.051*
C3	0.00857 (12)	0.1979 (2)	0.55315 (5)	0.0414 (5)
H3A	−0.0108	0.0707	0.5563	0.050*
H3B	−0.0474	0.2731	0.5638	0.050*
C4	0.12182 (11)	0.2518 (2)	0.49126 (5)	0.0348 (4)
C5	0.10309 (12)	0.3075 (2)	0.45001 (5)	0.0347 (4)
C6	0.18455 (13)	0.3375 (2)	0.41932 (5)	0.0401 (5)
C7	0.16573 (13)	0.3949 (2)	0.37498 (5)	0.0364 (4)
C8	0.24826 (13)	0.3823 (3)	0.34710 (6)	0.0474 (5)
H8	0.3119	0.3404	0.3568	0.057*
C9	0.23771 (14)	0.4304 (3)	0.30551 (6)	0.0496 (5)
H9	0.2944	0.4203	0.2876	0.059*
C10	0.14424 (13)	0.4939 (2)	0.28949 (6)	0.0401 (5)
C11	0.06187 (13)	0.5085 (2)	0.31750 (6)	0.0444 (5)
H11	−0.0015	0.5517	0.3079	0.053*
C12	0.07241 (13)	0.4601 (3)	0.35947 (5)	0.0421 (5)
H12	0.0161	0.4715	0.3775	0.050*
C13	0.13337 (14)	0.5414 (2)	0.24429 (6)	0.0431 (5)
C14	0.21450 (16)	0.6219 (3)	0.22220 (6)	0.0561 (6)
H14	0.2765	0.6462	0.2360	0.067*
C15	0.20419 (19)	0.6662 (3)	0.18004 (7)	0.0658 (6)
H15	0.2592	0.7198	0.1657	0.079*

C16	0.11331 (19)	0.6316 (3)	0.15926 (7)	0.0665 (6)
H16	0.1061	0.6630	0.1310	0.080*
C17	0.03321 (18)	0.5504 (3)	0.18044 (7)	0.0697 (7)
H17	−0.0283	0.5251	0.1664	0.084*
C18	0.04299 (16)	0.5058 (3)	0.22242 (6)	0.0578 (6)
H18	−0.0122	0.4508	0.2363	0.069*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0297 (8)	0.1105 (13)	0.0485 (8)	0.0093 (7)	−0.0012 (6)	0.0123 (8)
N1	0.0269 (8)	0.0697 (11)	0.0377 (9)	0.0051 (7)	0.0009 (7)	0.0067 (8)
N2	0.0246 (8)	0.0471 (9)	0.0367 (8)	−0.0010 (6)	0.0008 (6)	0.0018 (7)
N3	0.0288 (8)	0.0592 (10)	0.0441 (9)	−0.0002 (7)	−0.0015 (7)	0.0047 (8)
N4	0.0298 (8)	0.0524 (9)	0.0414 (9)	−0.0004 (7)	−0.0001 (7)	0.0032 (8)
C1	0.0391 (11)	0.0499 (11)	0.0425 (11)	0.0044 (9)	−0.0029 (9)	0.0072 (9)
C2	0.0391 (11)	0.0497 (11)	0.0380 (11)	0.0010 (9)	0.0003 (8)	0.0060 (9)
C3	0.0345 (10)	0.0491 (11)	0.0407 (11)	−0.0023 (8)	0.0079 (9)	0.0042 (9)
C4	0.0262 (9)	0.0401 (10)	0.0382 (10)	0.0004 (8)	−0.0002 (8)	−0.0018 (9)
C5	0.0268 (10)	0.0415 (10)	0.0357 (10)	0.0021 (7)	0.0003 (8)	0.0012 (9)
C6	0.0306 (10)	0.0490 (11)	0.0408 (11)	0.0033 (8)	−0.0022 (8)	−0.0024 (9)
C7	0.0313 (10)	0.0403 (10)	0.0376 (10)	0.0000 (8)	−0.0003 (8)	−0.0013 (8)
C8	0.0342 (10)	0.0647 (13)	0.0433 (12)	0.0079 (9)	0.0035 (9)	0.0033 (10)
C9	0.0417 (11)	0.0667 (14)	0.0402 (12)	0.0065 (10)	0.0114 (9)	0.0015 (10)
C10	0.0404 (11)	0.0403 (11)	0.0395 (11)	−0.0009 (8)	0.0013 (9)	−0.0023 (9)
C11	0.0364 (11)	0.0568 (13)	0.0399 (11)	0.0057 (9)	−0.0043 (9)	0.0021 (10)
C12	0.0337 (10)	0.0547 (12)	0.0379 (11)	0.0031 (9)	0.0047 (8)	−0.0005 (9)
C13	0.0505 (12)	0.0409 (10)	0.0378 (11)	0.0021 (9)	0.0016 (9)	−0.0019 (9)
C14	0.0590 (13)	0.0658 (14)	0.0436 (13)	−0.0082 (11)	0.0040 (10)	0.0016 (11)
C15	0.0825 (17)	0.0672 (15)	0.0479 (14)	−0.0052 (12)	0.0184 (12)	0.0065 (12)
C16	0.0975 (19)	0.0633 (15)	0.0388 (13)	0.0091 (13)	−0.0009 (13)	0.0025 (11)
C17	0.0766 (17)	0.0841 (17)	0.0485 (15)	−0.0029 (14)	−0.0138 (12)	0.0020 (13)
C18	0.0586 (14)	0.0693 (15)	0.0457 (13)	−0.0078 (11)	−0.0065 (10)	0.0068 (11)

Geometric parameters (Å, °)

O1—C6	1.2333 (19)	C7—C8	1.389 (2)
N1—C4	1.345 (2)	C8—C9	1.374 (2)
N1—C1	1.450 (2)	C8—H8	0.9300
N1—H1	0.901 (9)	C9—C10	1.391 (2)
N2—C4	1.3398 (18)	C9—H9	0.9300
N2—N3	1.3772 (18)	C10—C11	1.391 (2)
N2—C3	1.456 (2)	C10—C13	1.484 (2)
N3—N4	1.3032 (19)	C11—C12	1.386 (2)
N4—C5	1.3748 (19)	C11—H11	0.9300
C1—C2	1.517 (2)	C12—H12	0.9300
C1—H1A	0.9700	C13—C18	1.384 (2)
C1—H1B	0.9700	C13—C14	1.392 (2)
C2—C3	1.518 (2)	C14—C15	1.384 (3)

C2—H2A	0.9700	C14—H14	0.9300
C2—H2B	0.9700	C15—C16	1.371 (3)
C3—H3A	0.9700	C15—H15	0.9300
C3—H3B	0.9700	C16—C17	1.370 (3)
C4—C5	1.394 (2)	C16—H16	0.9300
C5—C6	1.452 (2)	C17—C18	1.379 (3)
C6—C7	1.490 (2)	C17—H17	0.9300
C7—C12	1.387 (2)	C18—H18	0.9300
C4—N1—C1	119.45 (14)	C12—C7—C6	125.08 (15)
C4—N1—H1	117.4 (12)	C8—C7—C6	117.28 (15)
C1—N1—H1	122.2 (12)	C9—C8—C7	121.30 (17)
C4—N2—N3	110.76 (13)	C9—C8—H8	119.4
C4—N2—C3	125.19 (14)	C7—C8—H8	119.4
N3—N2—C3	123.87 (13)	C8—C9—C10	121.58 (17)
N4—N3—N2	107.00 (13)	C8—C9—H9	119.2
N3—N4—C5	109.68 (13)	C10—C9—H9	119.2
N1—C1—C2	109.48 (14)	C9—C10—C11	117.15 (17)
N1—C1—H1A	109.8	C9—C10—C13	120.98 (17)
C2—C1—H1A	109.8	C11—C10—C13	121.87 (17)
N1—C1—H1B	109.8	C12—C11—C10	121.33 (17)
C2—C1—H1B	109.8	C12—C11—H11	119.3
H1A—C1—H1B	108.2	C10—C11—H11	119.3
C1—C2—C3	111.13 (15)	C11—C12—C7	120.99 (16)
C1—C2—H2A	109.4	C11—C12—H12	119.5
C3—C2—H2A	109.4	C7—C12—H12	119.5
C1—C2—H2B	109.4	C18—C13—C14	117.54 (18)
C3—C2—H2B	109.4	C18—C13—C10	121.41 (17)
H2A—C2—H2B	108.0	C14—C13—C10	121.04 (17)
N2—C3—C2	107.59 (13)	C15—C14—C13	120.9 (2)
N2—C3—H3A	110.2	C15—C14—H14	119.5
C2—C3—H3A	110.2	C13—C14—H14	119.5
N2—C3—H3B	110.2	C16—C15—C14	120.3 (2)
C2—C3—H3B	110.2	C16—C15—H15	119.8
H3A—C3—H3B	108.5	C14—C15—H15	119.8
N2—C4—N1	121.38 (15)	C17—C16—C15	119.4 (2)
N2—C4—C5	105.01 (13)	C17—C16—H16	120.3
N1—C4—C5	133.60 (15)	C15—C16—H16	120.3
N4—C5—C4	107.54 (14)	C16—C17—C18	120.6 (2)
N4—C5—C6	129.10 (15)	C16—C17—H17	119.7
C4—C5—C6	123.35 (14)	C18—C17—H17	119.7
O1—C6—C5	117.08 (16)	C17—C18—C13	121.2 (2)
O1—C6—C7	118.91 (15)	C17—C18—H18	119.4
C5—C6—C7	124.00 (15)	C13—C18—H18	119.4
C12—C7—C8	117.64 (16)		
C4—N2—N3—N4	0.80 (19)	C5—C6—C7—C12	12.9 (3)
C3—N2—N3—N4	176.12 (15)	O1—C6—C7—C8	11.9 (3)
N2—N3—N4—C5	−1.06 (18)	C5—C6—C7—C8	−167.19 (16)
C4—N1—C1—C2	−35.0 (2)	C12—C7—C8—C9	−0.8 (3)

N1—C1—C2—C3	57.67 (19)	C6—C7—C8—C9	179.30 (17)
C4—N2—C3—C2	19.5 (2)	C7—C8—C9—C10	0.0 (3)
N3—N2—C3—C2	−155.16 (15)	C8—C9—C10—C11	0.7 (3)
C1—C2—C3—N2	−48.84 (19)	C8—C9—C10—C13	−178.63 (18)
N3—N2—C4—N1	178.77 (15)	C9—C10—C11—C12	−0.6 (3)
C3—N2—C4—N1	3.5 (3)	C13—C10—C11—C12	178.66 (17)
N3—N2—C4—C5	−0.22 (18)	C10—C11—C12—C7	−0.1 (3)
C3—N2—C4—C5	−175.46 (15)	C8—C7—C12—C11	0.8 (3)
C1—N1—C4—N2	4.8 (3)	C6—C7—C12—C11	−179.28 (17)
C1—N1—C4—C5	−176.54 (18)	C9—C10—C13—C18	143.17 (19)
N3—N4—C5—C4	0.94 (19)	C11—C10—C13—C18	−36.1 (3)
N3—N4—C5—C6	−179.67 (17)	C9—C10—C13—C14	−36.1 (3)
N2—C4—C5—N4	−0.42 (18)	C11—C10—C13—C14	144.62 (19)
N1—C4—C5—N4	−179.23 (18)	C18—C13—C14—C15	0.7 (3)
N2—C4—C5—C6	−179.84 (15)	C10—C13—C14—C15	−179.97 (18)
N1—C4—C5—C6	1.3 (3)	C13—C14—C15—C16	0.1 (3)
N4—C5—C6—O1	−179.20 (16)	C14—C15—C16—C17	−0.9 (3)
C4—C5—C6—O1	0.1 (3)	C15—C16—C17—C18	0.9 (3)
N4—C5—C6—C7	−0.1 (3)	C16—C17—C18—C13	0.0 (3)
C4—C5—C6—C7	179.24 (16)	C14—C13—C18—C17	−0.8 (3)
O1—C6—C7—C12	−167.97 (17)	C10—C13—C18—C17	179.95 (19)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1	0.902 (13)	2.269 (17)	2.833 (2)	120.3 (12)
N1—H1···N3^{i}	0.902 (13)	2.452 (12)	3.152 (2)	134.6 (13)

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

Fig. 1

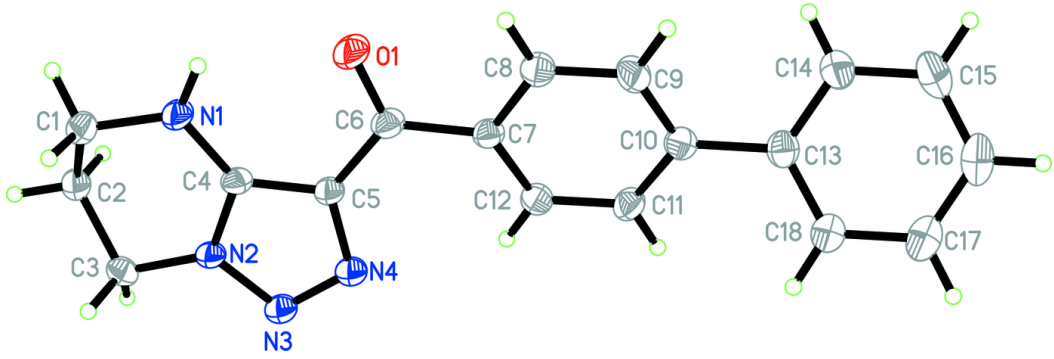
O1
C8
C9
C14
C15
C1
N1
C6
C7
C10
C13
C16
C4
C5
C12
C11
C2
C18
C17
N2
C3
N4
N3

Fig. 2