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Ethyl 5-(ethoxycarbonyl)-3-phenyl-1*H*-pyrazole-1-acetate

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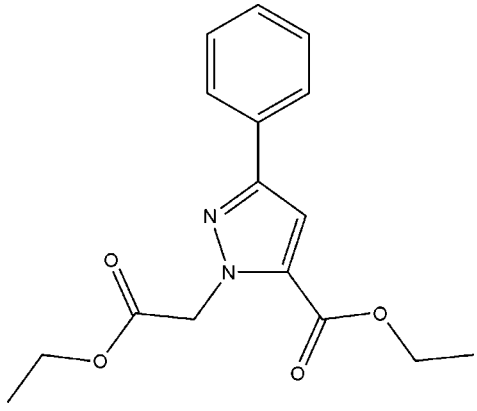
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.044; wR factor = 0.126; data-to-parameter ratio = 18.0.

In the title compound, $C_{16}H_{18}N_2O_4$, all bond lengths and angles show normal values. The dihedral angle between the pyrazole ring and the benzene ring is 10.64 (9)°. The molecules are linked into a parallel network by intermolecular C—H···π interactions.

Related literature

For related literature, see: Brough *et al.* (2005); Cheng *et al.* (2006); Sehon *et al.* (2006); Wei *et al.* (2006); Xia *et al.* (2007). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{16}H_{18}N_2O_4$
M_r = 302.32
Monoclinic, $P2_1/c$
a = 8.3898 (1) Å
b = 22.1418 (4) Å
c = 8.6374 (1) Å
β = 101.756 (1)°
V = 1570.87 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 296 (2) K
0.49 × 0.47 × 0.34 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*APEX2 Software Suite*; Bruker, 2005)
T_{min} = 0.87, T_{max} = 0.97
12410 measured reflections
3613 independent reflections
2467 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.126
S = 1.03
3613 reflections
201 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Table 1
X—H···π-ring interactions calculated by *PLATON* (Spek, 2003).

*Cg*1 is the centroid of the benzene ring.

X—H···Cg	X—H	H···Cg	X···Cg	D—H···Cg
C13—H13A···$Cg1^{i}$	0.97	2.88	3.6436 (19)	136

Symmetry code: (i) $1-x,-y,-z$.

Data collection: *APEX2 Software Suite* (Bruker, 2005); cell refinement: *APEX2 Software Suite*; data reduction: *APEX2 Software Suite*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: OM2172).

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supplementary materials

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Ethyl 5-(ethoxycarbonyl)-3-phenyl-1*H*-pyrazole-1-acetate

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Comment

Pyrazole derivatives are an important class of heteroaromatic ring systems that have found extensive use in the pharmaceutical industry. Many pyrazole derivatives are known to exhibit a wide range of biological properties such as antagonists (Sehon *et al.*, 2006), anti-inflammatory (Cheng *et al.*, 2006), inhibitors of the Hsp90 (Brough *et al.*, 2005), and antitumor (Wei *et al.*, 2006, Xia *et al.*., 2007).

In the title compound (Fig. 1), all bond lengths and angles are normal (Allen *et al.*, 1987). The dihedral angles between the rings of the pyrazole and the benzene ring is 10.64 (9)°. The two ethyl carboxylate group are inclined to the attached pyrazole ring by 3.34 (7)° and 74.15 (9)°, respectively. The molecules are linked into a parallel network by C—H···π interactions (Table 1) involving the benzene ring (centroid *Cg*1).

Experimental

A mixture of ethyl 3-phenyl-1*H*-pyrazole-5-carboxylate (0.01 mol), ethyl chloroacetate (0.015 mol) and potassium carbonate (0.02 mol) in acetonitrile (50 ml) was heated to reflux for 15 h. The solvent was removed under reduced pressure, and the residue was dissolved in the mixture of water (50 ml) and ethyl acetate (50 ml). After separation, the water phase was extracted with ethyl acetate (25 ml), and then the organic phase was combined, dried over anhydrous magnesium sulfate and filtered. The solvent was removed under reduced pressure. The solid product was recrystallized from ethyl acetate (yield 62%). Crystals suitable for X-ray diffraction were obtained by slow evaporation of a solution of the solid in ethyl acetate at room temperature for 5 d.

Refinement

All H atoms were placed in geometrically calculated positions and refined using a riding model with C—H = 0.97Å for CH groups, 0.93 Å (for CH_2 groups) and 0.96 Å (for CH_3 groups). Their isotropic displacement parameters were set to 1.2 times (1.5 times for CH_3 groups) the equivalent displacement parameter of their parent atoms. The anisotropic thermal parameters of C3 and C4 atoms were restrained with DELU in the final cycles of refinement.

Figures

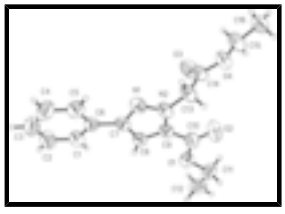

Fig. 1. The structure of the title molecule showing displacement ellipsoids drawn at the 50% probability level. The H atoms are depicted as spheres of arbitrary radii.

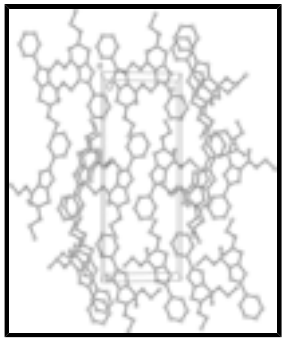

Fig. 2. Packing view shown down the *a* axis.

Ethyl 5-(ethoxycarbonyl)-3-phenyl-1*H*-pyrazole-1-acetate

Crystal data

$C_{16}H_{18}N_2O_4$	F_{000} = 640
M_r = 302.32	D_x = 1.278 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 3570 reflections
a = 8.3898 (1) Å	θ = 2.7–24.9º
b = 22.1418 (4) Å	μ = 0.09 mm^{-1}
c = 8.6374 (1) Å	T = 296 (2) K
β = 101.756 (1)º	Prism, colourless
V = 1570.87 (4) Å^3	0.49 × 0.47 × 0.34 mm
Z = 4	

Data collection

Bruker APEXII CCD diffractometer	3613 independent reflections
Radiation source: fine-focus sealed tube	2467 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.022
T = 296(2) K	θ_{max} = 27.5º
φ and ω scans	θ_{min} = 1.8º
Absorption correction: multi-scan (APEX2 Software Suite; Bruker, 2005)	h = −10→10
T_{min} = 0.87, T_{max} = 0.97	k = −28→26
12410 measured reflections	l = −10→11

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-atom parameters constrained
$wR(F^2)$ = 0.126	$w = 1/[\sigma^2(F_o^2) + (0.0596P)^2 + 0.2035P]$ where $P = (F_o^2 + 2F_c^2)/3$

S = 1.03	$(\Delta/\sigma)_{max}$ = 0.001
3613 reflections	$\Delta\rho_{max}$ = 0.15 e Å^{-3}
201 parameters	$\Delta\rho_{min}$ = −0.21 e Å^{-3}
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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}
C1	0.5549 (2)	1.14929 (7)	0.8148 (2)	0.0617 (4)
H1	0.4771	1.1463	0.7215	0.074*
C2	0.6001 (2)	1.20574 (8)	0.8781 (3)	0.0764 (5)
H2	0.5530	1.2402	0.8268	0.092*
C3	0.7135 (3)	1.21097 (9)	1.0154 (3)	0.0820 (5)
H3	0.7427	1.2489	1.0584	0.098*
C4	0.7840 (3)	1.15978 (10)	1.0895 (2)	0.0806 (5)
H4	0.8620	1.1633	1.1825	0.097*
C5	0.7407 (2)	1.10325 (8)	1.0276 (2)	0.0648 (4)
H5	0.7897	1.0690	1.0789	0.078*
C6	0.62402 (17)	1.09726 (7)	0.88887 (17)	0.0498 (3)
C7	0.57432 (17)	1.03747 (6)	0.82124 (16)	0.0464 (3)
C8	0.44394 (16)	1.02254 (6)	0.69906 (17)	0.0471 (3)
H8	0.3682	1.0488	0.6409	0.057*
C9	0.45055 (17)	0.96123 (6)	0.68258 (17)	0.0487 (3)
C10	0.34296 (19)	0.91989 (7)	0.57674 (18)	0.0557 (4)
C11	0.0989 (2)	0.91559 (9)	0.3812 (2)	0.0718 (5)
H11A	0.0317	0.8928	0.4395	0.086*
H11B	0.1512	0.8875	0.3212	0.086*
C12	−0.0026 (2)	0.95940 (10)	0.2730 (3)	0.0865 (6)
H12A	−0.0511	0.9877	0.3339	0.130*
H12B	−0.0867	0.9381	0.2018	0.130*
H12C	0.0645	0.9807	0.2135	0.130*
C13	0.6433 (2)	0.88134 (7)	0.82480 (19)	0.0614 (4)
H13A	0.5545	0.8548	0.8366	0.074*
H13B	0.7222	0.8813	0.9240	0.074*

C14	0.72291 (19)	0.85706 (7)	0.69551 (18)	0.0550 (4)
C15	0.8330 (3)	0.76827 (8)	0.6072 (2)	0.0732 (5)
H15A	0.9203	0.7925	0.5811	0.088*
H15B	0.7519	0.7617	0.5113	0.088*
C16	0.8972 (2)	0.71005 (8)	0.6745 (2)	0.0787 (5)
H16A	0.9800	0.7170	0.7673	0.118*
H16B	0.9430	0.6883	0.5979	0.118*
H16C	0.8106	0.6868	0.7022	0.118*
N1	0.65788 (15)	0.98781 (6)	0.87805 (15)	0.0539 (3)
N2	0.58104 (15)	0.94193 (5)	0.79247 (14)	0.0537 (3)
O1	0.22050 (13)	0.95017 (5)	0.48930 (14)	0.0636 (3)
O2	0.36269 (18)	0.86606 (5)	0.57036 (15)	0.0820 (4)
O3	0.75104 (17)	0.88472 (6)	0.58571 (16)	0.0822 (4)
O4	0.76022 (14)	0.79933 (5)	0.72446 (12)	0.0623 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0662 (10)	0.0530 (9)	0.0665 (10)	0.0059 (7)	0.0148 (8)	−0.0020 (7)
C2	0.0865 (13)	0.0517 (10)	0.0951 (14)	0.0013 (9)	0.0279 (11)	−0.0036 (9)
C3	0.0875 (13)	0.0659 (11)	0.0973 (15)	−0.0191 (9)	0.0296 (12)	−0.0195 (10)
C4	0.0762 (12)	0.0895 (13)	0.0738 (12)	−0.0223 (10)	0.0097 (10)	−0.0167 (10)
C5	0.0616 (9)	0.0700 (11)	0.0618 (10)	−0.0035 (8)	0.0103 (8)	−0.0001 (8)
C6	0.0482 (7)	0.0529 (9)	0.0517 (8)	0.0004 (6)	0.0182 (6)	−0.0011 (6)
C7	0.0487 (7)	0.0469 (8)	0.0463 (8)	0.0068 (6)	0.0160 (6)	0.0043 (6)
C8	0.0461 (7)	0.0456 (8)	0.0511 (8)	0.0060 (6)	0.0137 (6)	0.0048 (6)
C9	0.0534 (8)	0.0462 (8)	0.0488 (8)	0.0053 (6)	0.0154 (6)	0.0038 (6)
C10	0.0675 (10)	0.0487 (9)	0.0548 (9)	−0.0018 (7)	0.0215 (8)	−0.0008 (7)
C11	0.0622 (10)	0.0781 (12)	0.0759 (11)	−0.0222 (9)	0.0160 (9)	−0.0213 (10)
C12	0.0546 (10)	0.1118 (17)	0.0885 (14)	−0.0013 (10)	0.0038 (10)	−0.0248 (12)
C13	0.0820 (11)	0.0473 (9)	0.0563 (9)	0.0178 (8)	0.0173 (8)	0.0105 (7)
C14	0.0611 (9)	0.0485 (9)	0.0544 (9)	0.0111 (7)	0.0093 (7)	0.0088 (7)
C15	0.0983 (13)	0.0651 (11)	0.0597 (10)	0.0174 (10)	0.0240 (9)	−0.0052 (8)
C16	0.0852 (13)	0.0645 (11)	0.0877 (13)	0.0173 (9)	0.0209 (10)	−0.0100 (10)
N1	0.0593 (7)	0.0531 (7)	0.0494 (7)	0.0097 (6)	0.0112 (6)	0.0022 (6)
N2	0.0661 (8)	0.0433 (7)	0.0519 (7)	0.0117 (6)	0.0128 (6)	0.0051 (5)
O1	0.0533 (6)	0.0584 (7)	0.0757 (7)	−0.0044 (5)	0.0055 (5)	−0.0126 (5)
O2	0.1163 (11)	0.0464 (7)	0.0798 (9)	0.0013 (7)	0.0116 (8)	−0.0051 (6)
O3	0.1095 (10)	0.0694 (8)	0.0780 (9)	0.0284 (7)	0.0433 (8)	0.0283 (7)
O4	0.0876 (8)	0.0459 (6)	0.0560 (6)	0.0150 (5)	0.0210 (6)	0.0039 (5)

Geometric parameters (Å, °)

C1—C2	1.386 (2)	C11—C12	1.488 (3)
C1—C6	1.386 (2)	C11—H11A	0.9700
C1—H1	0.9300	C11—H11B	0.9700
C2—C3	1.366 (3)	C12—H12A	0.9600
C2—H2	0.9300	C12—H12B	0.9600
C3—C4	1.375 (3)	C12—H12C	0.9600

C3—H3	0.9300	C13—N2	1.4460 (18)
C4—C5	1.380 (2)	C13—C14	1.512 (2)
C4—H4	0.9300	C13—H13A	0.9700
C5—C6	1.391 (2)	C13—H13B	0.9700
C5—H5	0.9300	C14—O3	1.1921 (18)
C6—C7	1.472 (2)	C14—O4	1.3276 (17)
C7—N1	1.3422 (17)	C15—O4	1.4576 (19)
C7—C8	1.396 (2)	C15—C16	1.471 (2)
C8—C9	1.3673 (19)	C15—H15A	0.9700
C8—H8	0.9300	C15—H15B	0.9700
C9—N2	1.3633 (19)	C16—H16A	0.9600
C9—C10	1.467 (2)	C16—H16B	0.9600
C10—O2	1.2061 (19)	C16—H16C	0.9600
C10—O1	1.3263 (19)	N1—N2	1.3415 (18)
C11—O1	1.4530 (19)		
C2—C1—C6	120.81 (17)	H11A—C11—H11B	108.5
C2—C1—H1	119.6	C11—C12—H12A	109.5
C6—C1—H1	119.6	C11—C12—H12B	109.5
C3—C2—C1	120.35 (19)	H12A—C12—H12B	109.5
C3—C2—H2	119.8	C11—C12—H12C	109.5
C1—C2—H2	119.8	H12A—C12—H12C	109.5
C2—C3—C4	119.48 (18)	H12B—C12—H12C	109.5
C2—C3—H3	120.3	N2—C13—C14	112.57 (12)
C4—C3—H3	120.3	N2—C13—H13A	109.1
C3—C4—C5	120.83 (19)	C14—C13—H13A	109.1
C3—C4—H4	119.6	N2—C13—H13B	109.1
C5—C4—H4	119.6	C14—C13—H13B	109.1
C4—C5—C6	120.27 (17)	H13A—C13—H13B	107.8
C4—C5—H5	119.9	O3—C14—O4	124.55 (15)
C6—C5—H5	119.9	O3—C14—C13	126.43 (14)
C1—C6—C5	118.25 (15)	O4—C14—C13	109.02 (12)
C1—C6—C7	120.39 (14)	O4—C15—C16	108.09 (14)
C5—C6—C7	121.36 (14)	O4—C15—H15A	110.1
N1—C7—C8	110.65 (13)	C16—C15—H15A	110.1
N1—C7—C6	120.46 (13)	O4—C15—H15B	110.1
C8—C7—C6	128.88 (13)	C16—C15—H15B	110.1
C9—C8—C7	105.76 (12)	H15A—C15—H15B	108.4
C9—C8—H8	127.1	C15—C16—H16A	109.5
C7—C8—H8	127.1	C15—C16—H16B	109.5
N2—C9—C8	106.42 (13)	H16A—C16—H16B	109.5
N2—C9—C10	122.90 (13)	C15—C16—H16C	109.5
C8—C9—C10	130.65 (14)	H16A—C16—H16C	109.5
O2—C10—O1	124.82 (15)	H16B—C16—H16C	109.5
O2—C10—C9	124.91 (15)	N2—N1—C7	105.21 (12)
O1—C10—C9	110.27 (13)	N1—N2—C9	111.95 (11)
O1—C11—C12	107.29 (15)	N1—N2—C13	118.81 (13)
O1—C11—H11A	110.3	C9—N2—C13	129.23 (13)
C12—C11—H11A	110.3	C10—O1—C11	117.43 (13)
O1—C11—H11B	110.3	C14—O4—C15	116.07 (12)

C12—C11—H11B	110.3		
C6—C1—C2—C3	−0.3 (3)	C8—C9—C10—O1	−1.7 (2)
C1—C2—C3—C4	0.8 (3)	N2—C13—C14—O3	8.0 (3)
C2—C3—C4—C5	−0.6 (3)	N2—C13—C14—O4	−173.03 (14)
C3—C4—C5—C6	−0.2 (3)	C8—C7—N1—N2	0.04 (15)
C2—C1—C6—C5	−0.4 (2)	C6—C7—N1—N2	179.59 (12)
C2—C1—C6—C7	179.60 (14)	C7—N1—N2—C9	−0.01 (15)
C4—C5—C6—C1	0.7 (2)	C7—N1—N2—C13	179.56 (12)
C4—C5—C6—C7	−179.34 (15)	C8—C9—N2—N1	−0.02 (15)
C1—C6—C7—N1	169.66 (13)	C10—C9—N2—N1	−178.18 (12)
C5—C6—C7—N1	−10.3 (2)	C8—C9—N2—C13	−179.53 (14)
C1—C6—C7—C8	−10.9 (2)	C10—C9—N2—C13	2.3 (2)
C5—C6—C7—C8	169.16 (14)	C14—C13—N2—N1	−108.46 (16)
N1—C7—C8—C9	−0.05 (15)	C14—C13—N2—C9	71.0 (2)
C6—C7—C8—C9	−179.56 (13)	O2—C10—O1—C11	1.8 (2)
C7—C8—C9—N2	0.04 (15)	C9—C10—O1—C11	−177.99 (12)
C7—C8—C9—C10	178.01 (13)	C12—C11—O1—C10	−167.62 (14)
N2—C9—C10—O2	−3.8 (2)	O3—C14—O4—C15	−3.2 (2)
C8—C9—C10—O2	178.49 (15)	C13—C14—O4—C15	177.84 (15)
N2—C9—C10—O1	175.99 (12)	C16—C15—O4—C14	168.07 (15)

X—H···π-ring interactions calculated by PLATON (Spek, 2003). Cg1 is the centroid of the benzene ring.

X—H···*Cg*	*X*—H	H···*Cg*	*X*···*Cg*	D—H···*Cg*
C13—H13A···*Cg*1^{i}	0.97	2.88	3.6436 (19)	136

Symmetry code: (i) 1 − *x*,-*y*,-*z*.

Fig. 1

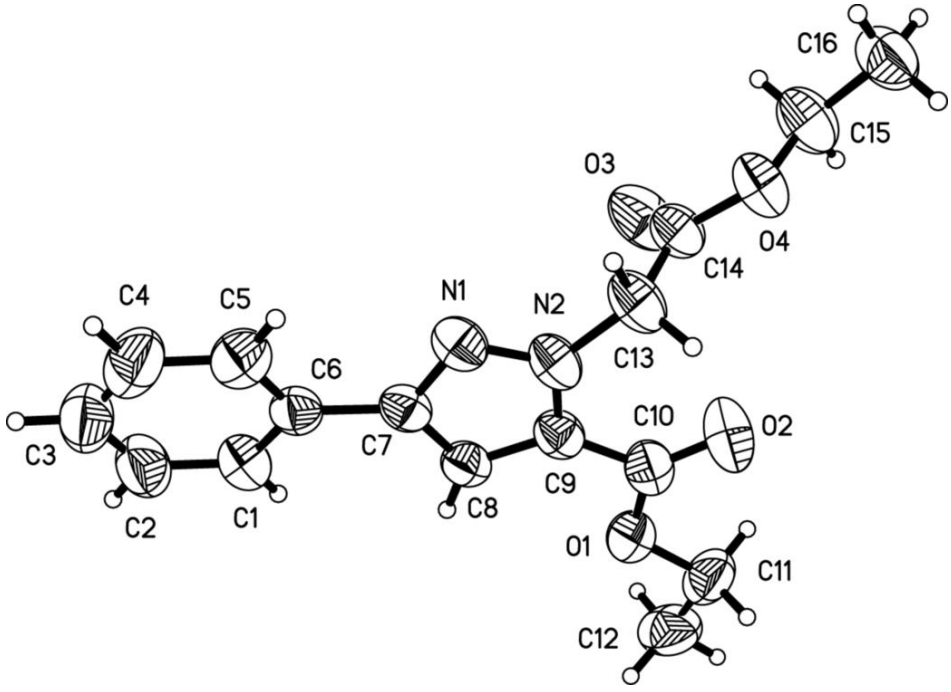
C16
C15
O3
O4
C14
C4
C5
N1
N2
C13
C6
C10
O2
C3
C7
C9
C8
C2
C1
O1
C11
C12

Fig. 2