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# 1,1′,5,5′-Tetramethyl-2,2′-diphenyl-4,4′-[*p*-phenylenebis(methylidynenitrilo)]di-1*H*-pyrazol-3(2*H*)-one

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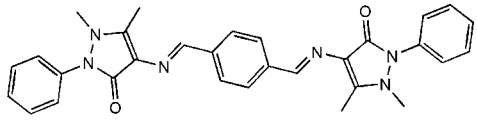
Key indicators: single-crystal X-ray study; $T$ = 292 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.049; $wR$ factor = 0.142; data-to-parameter ratio = 17.4.

In the centrosymmetric title compound, $C_{30}H_{28}N_6O_2$, the dihedral angles between the antipyrine ring and the terminal phenyl and central benzene rings are 50.55 (10) and 14.62 (9)°, respectively. Some short intermolecular C—H···O interactions may help to establish the packing. An intramolecular C—H···O hydrogen bond is also present.

## Related literature

For related structures, see: Guo *et al.* (2007); Selvakumar *et al.* (2007). For bond-length data, see: Allen *et al.* (1987).



## Experimental

### *Crystal data*

$C_{30}H_{28}N_6O_2$
$M_r$ = 504.58
Monoclinic, $P2_1/c$
$a$ = 6.0710 (2) Å
$b$ = 22.2948 (7) Å
$c$ = 9.8712 (3) Å
$\beta$ = 95.147 (2)°
$V$ = 1330.70 (7) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.08 mm$^{-1}$
$T$ = 292 (2) K
0.18 × 0.10 × 0.09 mm

### *Data collection*

Bruker APEX2 CCD diffractometer
Absorption correction: none
9162 measured reflections
3034 independent reflections
1545 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.027

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.141
$S$ = 1.03
3034 reflections
174 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.12 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.18 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C11—H11$C$···O1$^{i}$ | 0.96 | 2.36 | 3.321 (2) | 179 |
| C11—H11$A$···O1$^{ii}$ | 0.96 | 2.47 | 3.375 (3) | 157 |
| C12—H12···O1 | 0.93 | 2.30 | 3.002 (2) | 132 |

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

We thank Jian Dong Fan for collecting the data and Wei Huang for giving us a lot of help.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2742).

## References

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# supplementary materials

*Acta Cryst.* (2008). E**64**, o1479 [ **doi:10.1107/S1600536808021028** ]

# 1,1',5,5'-Tetramethyl-2,2'-diphenyl-4,4'-[*p*-phenylenebis(methylidynenitrilo)]di-1*H*-pyrazol-3(2*H*)-one

**Y. Xiao, C. F. Bi, A. D. Wang, Y. H. Fan, X. Zhang, J. K. Xu and S. T. Xie**

## Comment

Recently, some new Schiff bases of 4-aminoantipyrine have been reported (Guo *et al.*, 2007; Selvakumar *et al.*, 2007). We herein report the crystal structure of the related title compound, (I).

The complete molecule of (I) is generated by inversion and its bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The maximum deviation from the mean plane for the antipyrine ring (N1/N2/C7—C9) is 0.039 (2)Å for N2. The dihedral angles between the mean planes of the antipyrine ring and the terminal and central benzene rings are 50.55 (10)° and 14.62 (9)°, respectively.

In the crystal, weak intermolecular C—H···O hydrogen bonds (Table 1) lead to chains of molecules (Fig. 2).

## Experimental

The title compound was synthesized according to the literature method (Selvakumar *et al.*, 2007). Orange plates of (I) were obtained by slow evaporation of a dichloromethane solution at 292 K.

## Refinement

All H atoms were positioned geometrically, with C—H = 0.93 and 0.96 Å for aromatic and methyl H, respectively, and constrained to ride on their parent atoms, with $U_{iso}$(H) = 1.2$U_{eq}$(C) or 1.5$U_{eq}$(methyl C).

## Figures

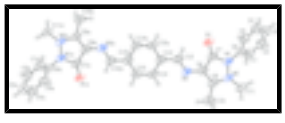

Fig. 1. The molecular structure of (I) with displacement ellipsoids drawn at the 50% probability level for the non-hydrogen atoms. Atoms with the suffix a are generated by the symmetry operation (2-x, 1-y, 1-z).

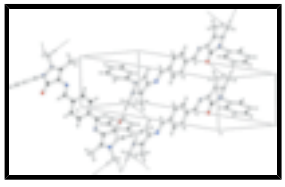

Fig. 2. A packing diagram for (I). Hydrogen bonds are shown as dashed lines.

## 1,1',5,5'-Tetramethyl-2,2'-diphenyl-4,4'-[p- phenylenebis(methylidynenitrilo)]di-1*H*-pyrazol-3(2*H*)-one

*Crystal data*

$C_{30}H_{28}N_6O_2$ $F_{000}$ = 532

| | |
|---|---|
| $M_r$ = 504.58 | $D_x$ = 1.259 Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation<br>λ = 0.71073 Å |
| Hall symbol: -P 2ybc | Cell parameters from 1828 reflections |
| $a$ = 6.0710 (2) Å | θ = 2.3–22.5º |
| $b$ = 22.2948 (7) Å | μ = 0.08 mm$^{-1}$ |
| $c$ = 9.8712 (3) Å | $T$ = 292 (2) K |
| β = 95.147 (2)º | Plate, orange |
| $V$ = 1330.70 (7) Å$^3$ | 0.18 × 0.10 × 0.09 mm |
| $Z$ = 2 | |

*Data collection*

| | |
|---|---|
| Bruker APEX2 CCD diffractometer | 1545 reflections with $I > 2\sigma(I)$ |
| Radiation source: fine-focus sealed tube | $R_{int}$ = 0.027 |
| Monochromator: graphite | $\theta_{max}$ = 27.5º |
| $T$ = 292(2) K | $\theta_{min}$ = 1.8º |
| ω scans | $h = -7 \rightarrow 7$ |
| Absorption correction: none | $k = -28 \rightarrow 17$ |
| 9162 measured reflections | $l = -12 \rightarrow 9$ |
| 3034 independent 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 |
| $wR(F^2)$ = 0.141 | $w = 1/[\sigma^2(F_o^2) + (0.0656P)^2 + 0.0153P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S$ = 1.03 | $(\Delta/\sigma)_{max}$ = 0.001 |
| 3034 reflections | $\Delta\rho_{max}$ = 0.12 e Å$^{-3}$ |
| 174 parameters | $\Delta\rho_{min}$ = −0.18 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 $F^2 > \sigma(F^2)$ 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 | 0.5949 (3) | 0.12139 (11) | 0.32324 (19) | 0.0767 (6) |
| H1 | 0.5483 | 0.1411 | 0.2427 | 0.092* |
| C2 | 0.6689 (3) | 0.06296 (12) | 0.3206 (2) | 0.0898 (7) |
| H2 | 0.6696 | 0.0428 | 0.2382 | 0.108* |
| C3 | 0.7414 (4) | 0.03451 (11) | 0.4398 (3) | 0.0976 (7) |
| H3 | 0.7902 | −0.0050 | 0.4379 | 0.117* |
| C4 | 0.7420 (4) | 0.06412 (12) | 0.5614 (2) | 0.0936 (7) |
| H4 | 0.7959 | 0.0450 | 0.6412 | 0.112* |
| C5 | 0.6643 (3) | 0.12151 (11) | 0.56657 (19) | 0.0777 (6) |
| H5 | 0.6608 | 0.1410 | 0.6497 | 0.093* |
| C6 | 0.5907 (3) | 0.15033 (9) | 0.44679 (17) | 0.0638 (5) |
| C7 | 0.6507 (3) | 0.25841 (8) | 0.49729 (18) | 0.0680 (5) |
| C8 | 0.5528 (3) | 0.31031 (9) | 0.42952 (16) | 0.0657 (5) |
| C9 | 0.3701 (3) | 0.29191 (11) | 0.34882 (16) | 0.0677 (5) |
| C10 | 0.2076 (3) | 0.32926 (10) | 0.26424 (18) | 0.0837 (6) |
| H10A | 0.1999 | 0.3158 | 0.1715 | 0.126* |
| H10B | 0.2536 | 0.3705 | 0.2689 | 0.126* |
| H10C | 0.0646 | 0.3256 | 0.2978 | 0.126* |
| C11 | 0.1418 (3) | 0.19847 (10) | 0.3420 (2) | 0.0868 (6) |
| H11A | 0.0685 | 0.2043 | 0.4232 | 0.130* |
| H11B | 0.1695 | 0.1565 | 0.3300 | 0.130* |
| H11C | 0.0497 | 0.2133 | 0.2650 | 0.130* |
| C12 | 0.8174 (3) | 0.38194 (9) | 0.49480 (17) | 0.0709 (5) |
| H12 | 0.8987 | 0.3517 | 0.5407 | 0.085* |
| C13 | 0.9078 (3) | 0.44231 (9) | 0.49655 (17) | 0.0647 (5) |
| C14 | 0.7891 (3) | 0.49092 (10) | 0.44065 (18) | 0.0758 (6) |
| H14 | 0.6464 | 0.4852 | 0.4000 | 0.091* |
| C15 | 0.8790 (3) | 0.54686 (9) | 0.44463 (19) | 0.0782 (6) |
| H15 | 0.7957 | 0.5787 | 0.4070 | 0.094* |
| N1 | 0.5147 (2) | 0.21053 (8) | 0.45312 (14) | 0.0699 (5) |
| N2 | 0.3520 (2) | 0.23112 (8) | 0.35370 (14) | 0.0709 (5) |
| N3 | 0.6298 (2) | 0.36905 (8) | 0.43221 (14) | 0.0692 (4) |
| O1 | 0.8196 (2) | 0.25210 (6) | 0.57570 (14) | 0.0871 (5) |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0660 (11) | 0.1017 (18) | 0.0610 (13) | −0.0126 (11) | −0.0015 (9) | −0.0058 (11) |
| C2 | 0.0811 (14) | 0.1034 (19) | 0.0843 (16) | −0.0086 (13) | 0.0044 (12) | −0.0195 (14) |
| C3 | 0.0867 (15) | 0.0909 (17) | 0.113 (2) | −0.0014 (12) | −0.0011 (14) | −0.0023 (16) |
| C4 | 0.0888 (15) | 0.107 (2) | 0.0824 (17) | 0.0020 (13) | −0.0072 (12) | 0.0115 (14) |
| C5 | 0.0653 (11) | 0.1053 (18) | 0.0605 (13) | −0.0029 (11) | −0.0064 (9) | −0.0020 (11) |
| C6 | 0.0466 (9) | 0.0866 (15) | 0.0563 (11) | −0.0086 (9) | −0.0051 (8) | −0.0048 (10) |
| C7 | 0.0526 (9) | 0.0930 (15) | 0.0554 (11) | −0.0021 (9) | −0.0110 (8) | −0.0130 (10) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C8 | 0.0527 (9) | 0.0910 (15) | 0.0509 (10) | 0.0064 (10) | −0.0094 (8) | −0.0077 (9) |
| C9 | 0.0487 (9) | 0.1015 (17) | 0.0505 (10) | 0.0041 (10) | −0.0086 (8) | −0.0064 (10) |
| C10 | 0.0628 (11) | 0.1145 (17) | 0.0696 (13) | 0.0125 (10) | −0.0170 (9) | −0.0040 (11) |
| C11 | 0.0511 (10) | 0.1288 (18) | 0.0764 (13) | −0.0119 (10) | −0.0170 (9) | −0.0014 (12) |
| C12 | 0.0620 (11) | 0.0919 (15) | 0.0562 (11) | 0.0073 (10) | −0.0099 (9) | −0.0055 (10) |
| C13 | 0.0590 (10) | 0.0832 (14) | 0.0497 (10) | 0.0020 (9) | −0.0070 (8) | −0.0066 (9) |
| C14 | 0.0555 (10) | 0.0959 (17) | 0.0716 (12) | 0.0021 (10) | −0.0187 (9) | −0.0014 (11) |
| C15 | 0.0643 (11) | 0.0872 (16) | 0.0781 (13) | 0.0062 (11) | −0.0210 (10) | 0.0021 (11) |
| N1 | 0.0542 (8) | 0.0945 (13) | 0.0571 (9) | 0.0002 (8) | −0.0174 (7) | −0.0081 (8) |
| N2 | 0.0477 (8) | 0.1039 (14) | 0.0570 (9) | 0.0000 (8) | −0.0175 (7) | −0.0055 (8) |
| N3 | 0.0579 (9) | 0.0914 (13) | 0.0557 (9) | 0.0025 (8) | −0.0085 (7) | −0.0079 (8) |
| O1 | 0.0706 (8) | 0.0991 (11) | 0.0832 (9) | 0.0021 (7) | −0.0401 (7) | −0.0085 (7) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—C2 | 1.379 (3) | C9—C10 | 1.488 (2) |
| C1—C6 | 1.382 (2) | C10—H10A | 0.9600 |
| C1—H1 | 0.9300 | C10—H10B | 0.9600 |
| C2—C3 | 1.373 (3) | C10—H10C | 0.9600 |
| C2—H2 | 0.9300 | C11—N2 | 1.464 (2) |
| C3—C4 | 1.370 (3) | C11—H11A | 0.9600 |
| C3—H3 | 0.9300 | C11—H11B | 0.9600 |
| C4—C5 | 1.366 (3) | C11—H11C | 0.9600 |
| C4—H4 | 0.9300 | C12—N3 | 1.279 (2) |
| C5—C6 | 1.384 (2) | C12—C13 | 1.453 (3) |
| C5—H5 | 0.9300 | C12—H12 | 0.9300 |
| C6—N1 | 1.422 (2) | C13—C14 | 1.388 (2) |
| C7—O1 | 1.2360 (19) | C13—C15$^{i}$ | 1.391 (2) |
| C7—N1 | 1.395 (2) | C14—C15 | 1.361 (2) |
| C7—C8 | 1.438 (2) | C14—H14 | 0.9300 |
| C8—C9 | 1.369 (2) | C15—C13$^{i}$ | 1.391 (2) |
| C8—N3 | 1.390 (2) | C15—H15 | 0.9300 |
| C9—N2 | 1.361 (2) | N1—N2 | 1.4056 (17) |
| C2—C1—C6 | 119.3 (2) | H10A—C10—H10B | 109.5 |
| C2—C1—H1 | 120.4 | C9—C10—H10C | 109.5 |
| C6—C1—H1 | 120.4 | H10A—C10—H10C | 109.5 |
| C3—C2—C1 | 120.0 (2) | H10B—C10—H10C | 109.5 |
| C3—C2—H2 | 120.0 | N2—C11—H11A | 109.5 |
| C1—C2—H2 | 120.0 | N2—C11—H11B | 109.5 |
| C4—C3—C2 | 120.2 (2) | H11A—C11—H11B | 109.5 |
| C4—C3—H3 | 119.9 | N2—C11—H11C | 109.5 |
| C2—C3—H3 | 119.9 | H11A—C11—H11C | 109.5 |
| C5—C4—C3 | 120.7 (2) | H11B—C11—H11C | 109.5 |
| C5—C4—H4 | 119.7 | N3—C12—C13 | 122.21 (17) |
| C3—C4—H4 | 119.7 | N3—C12—H12 | 118.9 |
| C4—C5—C6 | 119.3 (2) | C13—C12—H12 | 118.9 |
| C4—C5—H5 | 120.4 | C14—C13—C15$^{i}$ | 117.43 (17) |
| C6—C5—H5 | 120.4 | C14—C13—C12 | 122.44 (16) |

| | | | |
|---|---|---|---|
| C1—C6—C5 | 120.5 (2) | C15$^{i}$—C13—C12 | 120.13 (17) |
| C1—C6—N1 | 120.73 (17) | C15—C14—C13 | 120.76 (16) |
| C5—C6—N1 | 118.80 (17) | C15—C14—H14 | 119.6 |
| O1—C7—N1 | 122.90 (17) | C13—C14—H14 | 119.6 |
| O1—C7—C8 | 131.90 (17) | C14—C15—C13$^{i}$ | 121.81 (17) |
| N1—C7—C8 | 105.18 (15) | C14—C15—H15 | 119.1 |
| C9—C8—N3 | 123.15 (18) | C13$^{i}$—C15—H15 | 119.1 |
| C9—C8—C7 | 108.00 (18) | C7—N1—N2 | 109.12 (15) |
| N3—C8—C7 | 128.68 (15) | C7—N1—C6 | 123.38 (14) |
| N2—C9—C8 | 109.98 (16) | N2—N1—C6 | 119.21 (14) |
| N2—C9—C10 | 121.68 (16) | C9—N2—N1 | 107.21 (13) |
| C8—C9—C10 | 128.3 (2) | C9—N2—C11 | 124.40 (15) |
| C9—C10—H10A | 109.5 | N1—N2—C11 | 116.47 (16) |
| C9—C10—H10B | 109.5 | C12—N3—C8 | 120.28 (16) |
| C6—C1—C2—C3 | −1.3 (3) | C13—C14—C15—C13$^{i}$ | 0.4 (3) |
| C1—C2—C3—C4 | −0.5 (3) | O1—C7—N1—N2 | −173.36 (16) |
| C2—C3—C4—C5 | 2.1 (3) | C8—C7—N1—N2 | 4.94 (18) |
| C3—C4—C5—C6 | −2.0 (3) | O1—C7—N1—C6 | −25.5 (3) |
| C2—C1—C6—C5 | 1.4 (3) | C8—C7—N1—C6 | 152.81 (15) |
| C2—C1—C6—N1 | −179.78 (16) | C1—C6—N1—C7 | −114.23 (18) |
| C4—C5—C6—C1 | 0.3 (3) | C5—C6—N1—C7 | 64.6 (2) |
| C4—C5—C6—N1 | −178.63 (17) | C1—C6—N1—N2 | 30.6 (2) |
| O1—C7—C8—C9 | 177.28 (19) | C5—C6—N1—N2 | −150.49 (15) |
| N1—C7—C8—C9 | −0.80 (19) | C8—C9—N2—N1 | 6.81 (18) |
| O1—C7—C8—N3 | 2.0 (3) | C10—C9—N2—N1 | −173.58 (15) |
| N1—C7—C8—N3 | −176.11 (16) | C8—C9—N2—C11 | 147.84 (16) |
| N3—C8—C9—N2 | 171.85 (15) | C10—C9—N2—C11 | −32.6 (2) |
| C7—C8—C9—N2 | −3.78 (19) | C7—N1—N2—C9 | −7.32 (18) |
| N3—C8—C9—C10 | −7.7 (3) | C6—N1—N2—C9 | −156.74 (15) |
| C7—C8—C9—C10 | 176.65 (17) | C7—N1—N2—C11 | −151.89 (16) |
| N3—C12—C13—C14 | 5.7 (3) | C6—N1—N2—C11 | 58.7 (2) |
| N3—C12—C13—C15$^{i}$ | −174.14 (16) | C13—C12—N3—C8 | 177.92 (15) |
| C15$^{i}$—C13—C14—C15 | −0.4 (3) | C9—C8—N3—C12 | −170.27 (17) |
| C12—C13—C14—C15 | 179.78 (18) | C7—C8—N3—C12 | 4.4 (3) |

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

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C11—H11C···O1$^{ii}$ | 0.96 | 2.36 | 3.321 (2) | 179 |
| C11—H11A···O1$^{iii}$ | 0.96 | 2.47 | 3.375 (3) | 157 |
| C12—H12···O1 | 0.93 | 2.30 | 3.002 (2) | 132 |

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

**Fig. 1**

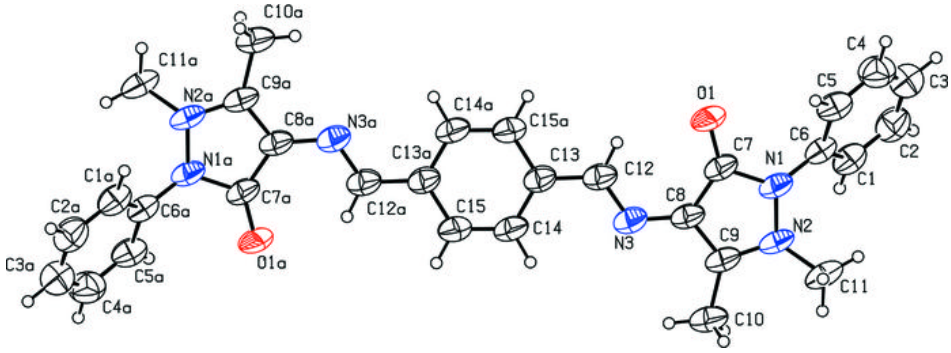
C10a
C11a
N2a
C9a
C8a
N3a
N1a
C1a
C2a
C6a
C7a
O1a
C3a
C5a
C4a
C14a
C15a
C13a
C13
C12
C12a
C15
C14
N3
O1
C7
C8
C4
C5
C3
C6
N1
C2
C1
C9
N2
C11
C10

**Fig. 2**