# organic compounds

# N-(4-Acetyl-3-methyl-1-phenyl-1H-pyrazol-5-yl)-N-methyl-2-(2-methyl-4-oxo-3,4-dihydroquinazolin-3-yl)-benzamide

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In the title compound, $C_{29}H_{25}N_5O_3$, the dihedral angle between the benzene ring and the pendant quinazoline ring system (r.m.s. deviation = 0.036Å) is 87.60 (17)°. The equivalent angle between the pyrazole ring and the phenyl group is 70.0 (2)°. The dihedral angle between the benzene and pyrazole rings is 30.7 (2)° and overall, the molecular conformation approximates to a Z shape. A short intramolecular C—H···O contact occurs. In the crystal, the molecules are linked by Cπ—H···O-type hydrogen bonds and aromatic π–π stacking interactions [centroid–centroid distance = 3.860 (3) Å], generating a three-dimensional network.

## Related literature

For background to the bioactivity of methaqualone and its derivatives, see: Ionescu-Pioggia *et al.* (1988); Wolfe *et al.* (1990). For structural and molecular modeling studies of quinazolinone derivatives, see: Duke & Codding (1993). For further synthetic details, see: Plescia *et al.* (1978).
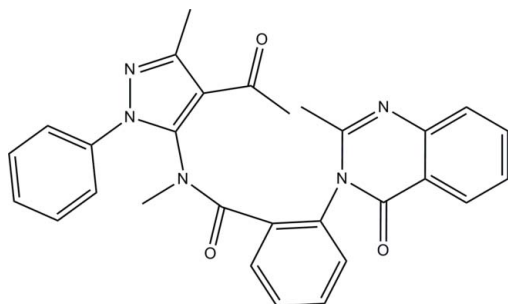


## Experimental

### Crystal data

| | |
|---|---|
| $C_{29}H_{25}N_5O_3$ | $V = 2542.3$ (16) Å$^3$ |
| $M_r = 491.54$ | $Z = 4$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation |
| $a = 8.617$ (4) Å | $\mu = 0.09$ mm$^{-1}$ |
| $b = 20.438$ (5) Å | $T = 293$ K |
| $c = 15.038$ (5) Å | $0.6 \times 0.5 \times 0.4$ mm |
| $\beta = 106.27$ (2)° | |

### Data collection

| | |
|---|---|
| Enraf–Nonius TurboCAD-4 diffractometer | 1358 reflections with $I > 2\sigma(I)$ |
| 5236 measured reflections | $R_{int} = 0.062$ |
| 4401 independent reflections | 3 standard reflections every 120 min intensity decay: −3% |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)] = 0.047$ | 338 parameters |
| $wR(F^2) = 0.120$ | H-atom parameters constrained |
| $S = 0.92$ | $\Delta\rho_{max} = 0.16$ e Å$^{-3}$ |
| 4401 reflections | $\Delta\rho_{min} = -0.16$ e Å$^{-3}$ |

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C21—H21C···O1 | 0.96 | 2.57 | 3.214 (5) | 124 |
| C3—H3···O2$^i$ | 0.93 | 2.54 | 3.351 (6) | 146 |
| C5—H5···O1$^{ii}$ | 0.93 | 2.40 | 3.276 (5) | 157 |
| C16—H16···O3$^{iii}$ | 0.93 | 2.52 | 3.305 (6) | 143 |

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

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 2012).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB7092).

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# supporting information

# *N*-(4-Acetyl-3-methyl-1-phenyl-1*H*-pyrazol-5-yl)-*N*-methyl-2-(2-methyl-4-oxo-3,4-dihydroquinazolin-3-yl)benzamide

**Fiorella Meneghetti and Benedetta Maggio**

## S1. Comment

The product obtained from 2-acetamido-*N*-methyl-N-(3-methyl-5-phenyl-1*H*-pyrazol-5-yl)benzamide by the Bischler–Napieralski reaction (Plescia *et al.*, 1978) was hydrolized by 6 N aqueous hydrochloric acid to give the metaqualone derivative 1, whose definitive structure is now reported (Fig. 1 and 2). The compound $C_{29}H_{25}N_5O_3$ crystallizes in the monoclinic P2$_1$/c space group. The overall molecular conformation has about a *Z* shape (Fig. 2). The presence of an intramolecular hydrogen bond between C2—H2⋯O1 at a distance of 2.65 (1) Å, angle 129 (1)° contributes to stabilize the folded conformation of the molecule. The 2-methyl-4-oxoquinazolin-3(4*H*)-yl)benzamide moiety is characterized by an almost planar conformation, with the maximum deviation out of its best mean plane for O2 and C21 atoms by 0.103 (4) Å and -0.198 (5) Å, respectively. The bicyclic system is nearly perpendicularly oriented with respect to the N4-attached phenyl ring (dihedral angle 87.60 (17)°), while it forms with the distal ones a dihedral angle of 41.0 (1)°. The pyrazole is inclined of 70.0 (2)° with respect to both the bicyclic moiety and the C1—C6 benzene, while it presents a dihedral angle of 30.7 (2)° with the C12—C17 phenyl ring. The two benzene are oriented each other at 51.6 (2)°. The molecular packing is stabilized by intermolecular interactions type C$\pi$—H⋯O between: C3—H3⋯O2i of 2.51 (3) Å and 146 (1)° [symmetry code: (i) *x* + 1, *y*, *z*], C5—H5⋯O1ii contact of 2.40 (3) Å and 157 (1)° [symmetry code: (ii) *x*, 1/2 - *y*, *z* - 1/2], and C16—H16⋯O3iii at distance of 2.51 (4) Å, angle 143 (1)° [symmetry code: (iii) 2 - *x*, *y* + 1/2, 1/2 - *z*] (Fig. 3). Stacking interactions between the benzene o f the oxoquinazoline systems [centroid-centroid distance = 3.860 (3) Å] further consolidate the packing.

## S2. Experimental

A solution of the product obtained from 2-acetamido-*N*-methyl-*N*-(3-methyl-5-phenyl-1*H*-pyrazol-5-yl)benzamide by the Bischler-Napieralski reaction (Plescia *et al.*, 1978) (6 g.) in 60 ml of aqueous 6 N hydrochloric acid was refluxed for 25 minutes. The precipitated solid (3.2 g) was crystallized from ethanol-diethyl ether to give a product which was dissolved in chloroform (100 ml) and treated with triethylamine (5 ml). The solution was stirred for 1 h at room temperature, washed with water (3 × 100 ml) and dried (sodium sulfate). Removal of the solvent and the crystallization from ethanol of the residue afforded to the title compound.

## S3. Refinement

Hydrogen atoms were located by difference Fourier synthesis, except methyl and phenyl hydrogen atoms, that were introduced at calculated positions, in their described geometries and allowed to ride on the attached carbon atom with fixed isotropic thermal parameters 1.2Ueq and 1.5Ueq of the parent carbon atom for aromatic H-atoms and methyl-bound H-atoms, respectively. The crystal contains small solvent accessible voids, however, no electron density peaks were found in chemically sensible positions for solvent molecules.
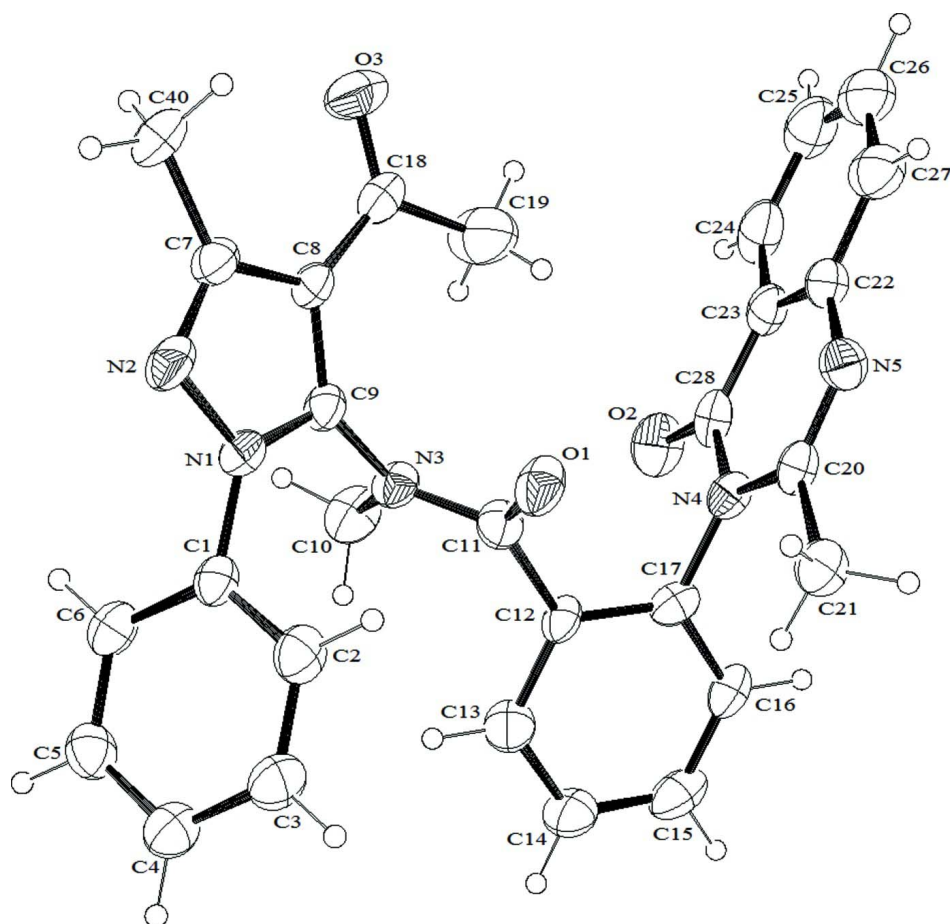
**Figure 1**

The molecular structure of the title compound, showing displacement ellipsoids for non-H atoms at the 40% probability level.
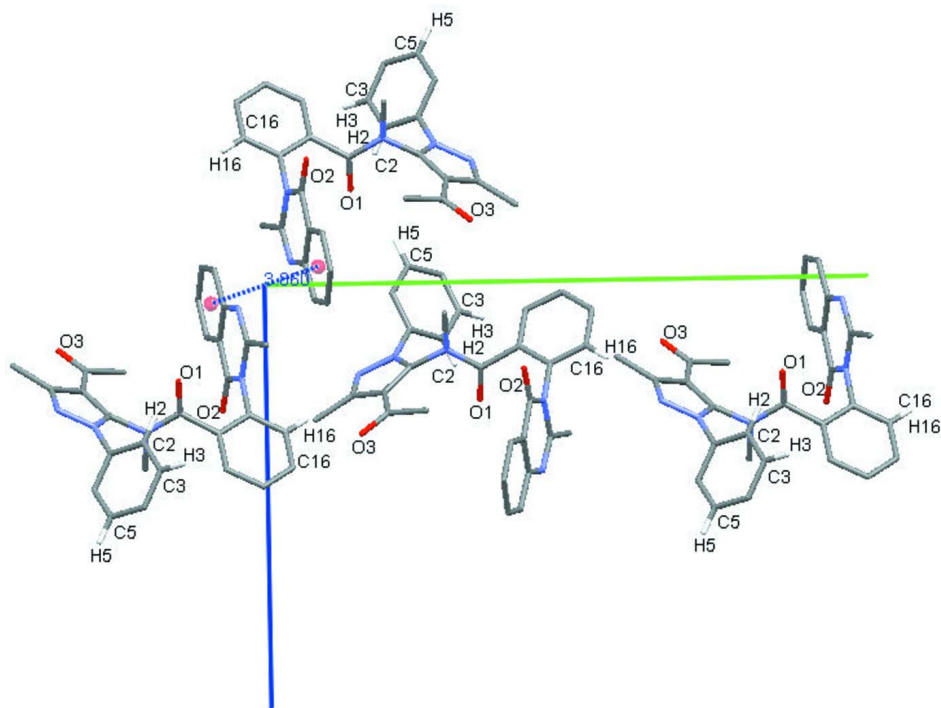
**Figure 2**

Intermolecular interactions of the title compound, viewed along the *a* axis. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

## *N*-(4-Acetyl-3-methyl-1-phenyl-1*H*-pyrazol-5-yl)-*N*-methyl-2-(2-methyl-4-oxo-3,4-dihydroquinazolin-3-yl)benzamide

*Crystal data*

| | |
|---|---|
| $C_{29}H_{25}N_5O_3$ | $F(000) = 1032$ |
| $M_r = 491.54$ | $D_x = 1.284$ Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Mo *Kα* radiation, $\lambda = 0.71073$ Å |
| Hall symbol: -P 2ybc | Cell parameters from 25 reflections |
| $a = 8.617$ (4) Å | $\theta = 9$–$10°$ |
| $b = 20.438$ (5) Å | $\mu = 0.09$ mm$^{-1}$ |
| $c = 15.038$ (5) Å | $T = 293$ K |
| $\beta = 106.27$ (2)° | Prism, colorless |
| $V = 2542.3$ (16) Å$^3$ | $0.6 \times 0.5 \times 0.4$ mm |
| $Z = 4$ | |

*Data collection*

| | |
|---|---|
| Enraf–Nonius TurboCAD-4 diffractometer | $R_{int} = 0.062$ |
| | $\theta_{max} = 24.9°$, $\theta_{min} = 2.4°$ |
| Radiation source: fine-focus sealed tube | $h = -10 \rightarrow 9$ |
| Graphite monochromator | $k = -1 \rightarrow 24$ |
| non–profiled *ω/2θ* scans | $l = -1 \rightarrow 17$ |
| 5236 measured reflections | 3 standard reflections every 120 min |
| 4401 independent reflections | intensity decay: −3% |
| 1358 reflections with $I > 2\sigma(I)$ | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | 0 restraints |
| Least-squares matrix: full | H-atom parameters constrained |
| $R[F^2 > 2\sigma(F^2)] = 0.047$ | $w = 1/[\sigma^2(F_o^2) + (0.0352P)^2]$ |
| $wR(F^2) = 0.120$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 0.92$ | $(\Delta/\sigma)_{max} = 0.003$ |
| 4401 reflections | $\Delta\rho_{max} = 0.16$ e Å⁻³ |
| 338 parameters | $\Delta\rho_{min} = -0.16$ 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 (Å²)*

| | $x$ | $y$ | $z$ | $U_{iso}*/U_{eq}$ |
|---|---|---|---|---|
| C28 | −0.0045 (5) | 0.4366 (2) | 0.2801 (3) | 0.0519 (12) |
| O1 | 0.3884 (3) | 0.35430 (13) | 0.2742 (2) | 0.0663 (9) |
| O3 | 0.0937 (4) | 0.15749 (16) | 0.3520 (2) | 0.0943 (11) |
| N4 | 0.1492 (4) | 0.45942 (15) | 0.2812 (2) | 0.0441 (9) |
| N3 | 0.2159 (4) | 0.29993 (16) | 0.1566 (2) | 0.0481 (9) |
| N2 | 0.4505 (4) | 0.15359 (16) | 0.2105 (2) | 0.0594 (10) |
| N1 | 0.4060 (4) | 0.21360 (16) | 0.1706 (2) | 0.0509 (9) |
| N5 | 0.2572 (4) | 0.45536 (16) | 0.4443 (2) | 0.0538 (9) |
| C9 | 0.2816 (5) | 0.23970 (19) | 0.1957 (3) | 0.0466 (11) |
| C16 | 0.1457 (5) | 0.5305 (2) | 0.1520 (3) | 0.0548 (12) |
| H16 | 0.1038 | 0.5639 | 0.1805 | 0.066* |
| C1 | 0.4832 (5) | 0.2402 (2) | 0.1058 (3) | 0.0460 (11) |
| C23 | −0.0249 (6) | 0.42417 (19) | 0.3709 (3) | 0.0523 (12) |
| C17 | 0.1780 (5) | 0.4706 (2) | 0.1939 (3) | 0.0484 (11) |
| C20 | 0.2709 (5) | 0.46966 (18) | 0.3641 (3) | 0.0477 (11) |
| C8 | 0.2412 (5) | 0.19578 (19) | 0.2549 (3) | 0.0479 (11) |
| O2 | −0.1073 (3) | 0.42729 (14) | 0.2066 (2) | 0.0772 (10) |
| C10 | 0.0791 (5) | 0.29730 (19) | 0.0725 (3) | 0.0685 (13) |
| H10A | 0.014 | 0.3359 | 0.0689 | 0.103* |
| H10B | 0.0148 | 0.2593 | 0.0745 | 0.103* |
| H10C | 0.1191 | 0.295 | 0.019 | 0.103* |
| C40 | 0.3648 (5) | 0.08126 (19) | 0.3164 (3) | 0.0791 (15) |
| H40A | 0.4463 | 0.0537 | 0.3036 | 0.119* |
| H40B | 0.263 | 0.0587 | 0.2997 | 0.119* |
| H40C | 0.3942 | 0.0917 | 0.3812 | 0.119* |
| C12 | 0.2366 (5) | 0.42010 (18) | 0.1508 (3) | 0.0450 (11) |
| C11 | 0.2842 (5) | 0.3564 (2) | 0.1997 (3) | 0.0510 (12) |
| C4 | 0.6172 (5) | 0.2944 (2) | −0.0221 (3) | 0.0662 (13) |

| | | | |
|---|---|---|---|
| H4 | 0.6613 | 0.3129 | −0.066 | 0.079* |
| C7 | 0.3506 (5) | 0.1433 (2) | 0.2608 (3) | 0.0515 (11) |
| C5 | 0.5239 (6) | 0.2388 (2) | −0.0431 (3) | 0.0622 (13) |
| H5 | 0.5063 | 0.2196 | −0.1011 | 0.075* |
| C3 | 0.6448 (5) | 0.3221 (2) | 0.0637 (4) | 0.0682 (13) |
| H3 | 0.7082 | 0.3596 | 0.0781 | 0.082* |
| C21 | 0.4242 (5) | 0.50044 (19) | 0.3564 (3) | 0.0629 (13) |
| H21A | 0.4986 | 0.5048 | 0.417 | 0.094* |
| H21B | 0.401 | 0.5429 | 0.3285 | 0.094* |
| H21C | 0.4714 | 0.4734 | 0.3187 | 0.094* |
| C13 | 0.2639 (5) | 0.4318 (2) | 0.0658 (3) | 0.0611 (12) |
| H13 | 0.3029 | 0.3983 | 0.036 | 0.073* |
| C22 | 0.1086 (6) | 0.4322 (2) | 0.4488 (3) | 0.0531 (11) |
| C2 | 0.5792 (5) | 0.2949 (2) | 0.1288 (3) | 0.0600 (12) |
| H2 | 0.5996 | 0.3132 | 0.1876 | 0.072* |
| C24 | −0.1740 (6) | 0.4032 (2) | 0.3805 (4) | 0.0768 (15) |
| H24 | −0.2624 | 0.3981 | 0.3288 | 0.092* |
| C26 | −0.0550 (8) | 0.3968 (2) | 0.5449 (4) | 0.0877 (17) |
| H26 | −0.0652 | 0.3871 | 0.6034 | 0.105* |
| C15 | 0.1749 (5) | 0.5416 (2) | 0.0680 (3) | 0.0697 (14) |
| H15 | 0.1544 | 0.5826 | 0.0405 | 0.084* |
| C6 | 0.4571 (5) | 0.2117 (2) | 0.0202 (3) | 0.0591 (12) |
| H6 | 0.3942 | 0.1741 | 0.0056 | 0.071* |
| C14 | 0.2338 (5) | 0.4928 (2) | 0.0249 (3) | 0.0660 (13) |
| H14 | 0.2536 | 0.5006 | −0.0319 | 0.079* |
| C18 | 0.1174 (5) | 0.2017 (2) | 0.3040 (3) | 0.0612 (13) |
| C25 | −0.1877 (7) | 0.3903 (2) | 0.4688 (5) | 0.0877 (18) |
| H25 | −0.2864 | 0.3772 | 0.4765 | 0.105* |
| C19 | 0.0256 (6) | 0.2638 (2) | 0.3005 (4) | 0.113 (2) |
| H19A | −0.0472 | 0.2597 | 0.3382 | 0.17* |
| H19B | −0.0349 | 0.2728 | 0.2377 | 0.17* |
| H19C | 0.0997 | 0.2991 | 0.3234 | 0.17* |
| C27 | 0.0913 (6) | 0.4173 (2) | 0.5363 (4) | 0.0730 (15) |
| H27 | 0.1791 | 0.4213 | 0.5886 | 0.088* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C28 | 0.045 (3) | 0.040 (3) | 0.064 (3) | 0.001 (2) | 0.003 (3) | −0.015 (3) |
| O1 | 0.085 (2) | 0.0449 (19) | 0.056 (2) | 0.0109 (16) | −0.0015 (18) | 0.0038 (16) |
| O3 | 0.121 (3) | 0.063 (2) | 0.118 (3) | −0.003 (2) | 0.065 (2) | 0.020 (2) |
| N4 | 0.034 (2) | 0.042 (2) | 0.049 (2) | −0.0039 (17) | −0.001 (2) | −0.0022 (18) |
| N3 | 0.049 (2) | 0.041 (2) | 0.048 (2) | 0.0066 (19) | 0.0030 (18) | 0.007 (2) |
| N2 | 0.072 (3) | 0.037 (2) | 0.068 (3) | 0.011 (2) | 0.017 (2) | 0.009 (2) |
| N1 | 0.061 (2) | 0.036 (2) | 0.057 (2) | 0.005 (2) | 0.018 (2) | 0.0066 (19) |
| N5 | 0.050 (2) | 0.058 (2) | 0.053 (2) | 0.000 (2) | 0.013 (2) | −0.004 (2) |
| C9 | 0.054 (3) | 0.036 (3) | 0.048 (3) | 0.009 (2) | 0.011 (2) | 0.003 (2) |
| C16 | 0.059 (3) | 0.030 (3) | 0.068 (3) | 0.003 (2) | 0.005 (3) | 0.001 (3) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C1 | 0.050 (3) | 0.038 (3) | 0.046 (3) | 0.004 (2) | 0.008 (2) | −0.003 (2) |
| C23 | 0.050 (3) | 0.040 (3) | 0.070 (4) | −0.001 (2) | 0.021 (3) | −0.010 (2) |
| C17 | 0.044 (3) | 0.044 (3) | 0.050 (3) | 0.000 (2) | 0.001 (2) | 0.010 (2) |
| C20 | 0.039 (3) | 0.037 (3) | 0.063 (3) | 0.004 (2) | 0.007 (3) | −0.007 (3) |
| C8 | 0.058 (3) | 0.035 (3) | 0.051 (3) | 0.006 (2) | 0.015 (2) | 0.004 (2) |
| O2 | 0.055 (2) | 0.084 (2) | 0.079 (2) | −0.0142 (17) | −0.0042 (19) | −0.0180 (19) |
| C10 | 0.067 (3) | 0.061 (3) | 0.062 (3) | 0.007 (3) | −0.007 (3) | 0.011 (3) |
| C40 | 0.105 (4) | 0.038 (3) | 0.093 (4) | 0.012 (3) | 0.026 (3) | 0.012 (3) |
| C12 | 0.051 (3) | 0.032 (3) | 0.046 (3) | 0.007 (2) | 0.004 (2) | 0.006 (2) |
| C11 | 0.056 (3) | 0.050 (3) | 0.042 (3) | 0.007 (3) | 0.006 (2) | 0.002 (3) |
| C4 | 0.067 (3) | 0.060 (3) | 0.070 (4) | −0.006 (3) | 0.018 (3) | 0.001 (3) |
| C7 | 0.068 (3) | 0.036 (3) | 0.046 (3) | 0.002 (3) | 0.008 (2) | 0.005 (2) |
| C5 | 0.073 (3) | 0.060 (3) | 0.051 (3) | 0.001 (3) | 0.012 (3) | −0.006 (3) |
| C3 | 0.064 (3) | 0.059 (3) | 0.084 (4) | −0.019 (2) | 0.023 (3) | −0.011 (3) |
| C21 | 0.051 (3) | 0.071 (3) | 0.061 (3) | −0.012 (3) | 0.006 (2) | −0.011 (3) |
| C13 | 0.072 (3) | 0.056 (3) | 0.052 (3) | 0.008 (3) | 0.011 (3) | 0.006 (3) |
| C22 | 0.060 (3) | 0.043 (3) | 0.060 (3) | 0.006 (2) | 0.022 (3) | −0.002 (2) |
| C2 | 0.064 (3) | 0.061 (3) | 0.050 (3) | −0.011 (3) | 0.006 (2) | −0.010 (3) |
| C24 | 0.066 (4) | 0.054 (3) | 0.116 (5) | −0.008 (3) | 0.036 (3) | −0.027 (3) |
| C26 | 0.109 (5) | 0.061 (4) | 0.110 (5) | 0.013 (4) | 0.058 (5) | 0.005 (3) |
| C15 | 0.073 (3) | 0.051 (3) | 0.071 (4) | −0.008 (3) | −0.003 (3) | 0.018 (3) |
| C6 | 0.059 (3) | 0.046 (3) | 0.070 (3) | −0.012 (2) | 0.015 (3) | −0.009 (3) |
| C14 | 0.081 (4) | 0.059 (3) | 0.053 (3) | −0.001 (3) | 0.010 (3) | 0.015 (3) |
| C18 | 0.074 (3) | 0.035 (3) | 0.074 (4) | −0.003 (3) | 0.020 (3) | 0.002 (3) |
| C25 | 0.083 (5) | 0.064 (3) | 0.135 (6) | −0.015 (3) | 0.063 (5) | −0.016 (4) |
| C19 | 0.138 (5) | 0.084 (4) | 0.148 (5) | 0.043 (4) | 0.089 (4) | 0.040 (4) |
| C27 | 0.078 (4) | 0.060 (3) | 0.084 (4) | 0.011 (3) | 0.027 (3) | 0.005 (3) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C28—O2 | 1.223 (4) | C40—H40B | 0.96 |
| C28—N4 | 1.400 (4) | C40—H40C | 0.96 |
| C28—C23 | 1.446 (5) | C12—C13 | 1.383 (5) |
| O1—C11 | 1.225 (4) | C12—C11 | 1.495 (5) |
| O3—C18 | 1.210 (4) | C4—C3 | 1.368 (5) |
| N4—C20 | 1.402 (4) | C4—C5 | 1.375 (5) |
| N4—C17 | 1.421 (4) | C4—H4 | 0.93 |
| N3—C11 | 1.373 (5) | C5—C6 | 1.361 (5) |
| N3—C9 | 1.412 (4) | C5—H5 | 0.93 |
| N3—C10 | 1.469 (4) | C3—C2 | 1.379 (5) |
| N2—C7 | 1.312 (4) | C3—H3 | 0.93 |
| N2—N1 | 1.372 (4) | C21—H21A | 0.96 |
| N1—C9 | 1.343 (4) | C21—H21B | 0.96 |
| N1—C1 | 1.431 (5) | C21—H21C | 0.96 |
| N5—C20 | 1.278 (4) | C13—C14 | 1.384 (5) |
| N5—C22 | 1.385 (5) | C13—H13 | 0.93 |
| C9—C8 | 1.376 (5) | C22—C27 | 1.399 (5) |
| C16—C17 | 1.370 (5) | C2—H2 | 0.93 |

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| C16—C15 | 1.374 (5) | C24—C25 | 1.390 (6) |
| C16—H16 | 0.93 | C24—H24 | 0.93 |
| C1—C6 | 1.372 (5) | C26—C27 | 1.368 (6) |
| C1—C2 | 1.377 (5) | C26—C25 | 1.379 (6) |
| C23—C24 | 1.399 (5) | C26—H26 | 0.93 |
| C23—C22 | 1.403 (5) | C15—C14 | 1.363 (5) |
| C17—C12 | 1.387 (5) | C15—H15 | 0.93 |
| C20—C21 | 1.497 (5) | C6—H6 | 0.93 |
| C8—C7 | 1.414 (5) | C14—H14 | 0.93 |
| C8—C18 | 1.462 (5) | C18—C19 | 1.489 (5) |
| C10—H10A | 0.96 | C25—H25 | 0.93 |
| C10—H10B | 0.96 | C19—H19A | 0.96 |
| C10—H10C | 0.96 | C19—H19B | 0.96 |
| C40—C7 | 1.505 (5) | C19—H19C | 0.96 |
| C40—H40A | 0.96 | C27—H27 | 0.93 |
| | | | |
| O2—C28—N4 | 120.4 (4) | C5—C4—H4 | 120.2 |
| O2—C28—C23 | 125.2 (4) | N2—C7—C8 | 112.1 (4) |
| N4—C28—C23 | 114.4 (4) | N2—C7—C40 | 119.4 (4) |
| C28—N4—C20 | 122.0 (4) | C8—C7—C40 | 128.5 (4) |
| C28—N4—C17 | 116.9 (3) | C6—C5—C4 | 120.7 (4) |
| C20—N4—C17 | 121.1 (4) | C6—C5—H5 | 119.6 |
| C11—N3—C9 | 117.9 (3) | C4—C5—H5 | 119.6 |
| C11—N3—C10 | 124.9 (3) | C4—C3—C2 | 120.4 (4) |
| C9—N3—C10 | 117.2 (3) | C4—C3—H3 | 119.8 |
| C7—N2—N1 | 104.3 (3) | C2—C3—H3 | 119.8 |
| C9—N1—N2 | 112.3 (3) | C20—C21—H21A | 109.5 |
| C9—N1—C1 | 126.9 (3) | C20—C21—H21B | 109.5 |
| N2—N1—C1 | 120.7 (3) | H21A—C21—H21B | 109.5 |
| C20—N5—C22 | 117.1 (4) | C20—C21—H21C | 109.5 |
| N1—C9—C8 | 106.8 (3) | H21A—C21—H21C | 109.5 |
| N1—C9—N3 | 119.2 (4) | H21B—C21—H21C | 109.5 |
| C8—C9—N3 | 133.9 (4) | C12—C13—C14 | 120.5 (4) |
| C17—C16—C15 | 120.3 (4) | C12—C13—H13 | 119.7 |
| C17—C16—H16 | 119.8 | C14—C13—H13 | 119.7 |
| C15—C16—H16 | 119.8 | N5—C22—C27 | 117.7 (5) |
| C6—C1—C2 | 120.7 (4) | N5—C22—C23 | 123.5 (4) |
| C6—C1—N1 | 119.5 (4) | C27—C22—C23 | 118.8 (5) |
| C2—C1—N1 | 119.8 (4) | C1—C2—C3 | 119.1 (4) |
| C24—C23—C22 | 120.7 (5) | C1—C2—H2 | 120.5 |
| C24—C23—C28 | 120.6 (5) | C3—C2—H2 | 120.5 |
| C22—C23—C28 | 118.7 (4) | C25—C24—C23 | 118.9 (5) |
| C16—C17—C12 | 120.2 (4) | C25—C24—H24 | 120.6 |
| C16—C17—N4 | 120.2 (4) | C23—C24—H24 | 120.6 |
| C12—C17—N4 | 119.6 (4) | C27—C26—C25 | 121.5 (6) |
| N5—C20—N4 | 124.1 (4) | C27—C26—H26 | 119.2 |
| N5—C20—C21 | 119.2 (4) | C25—C26—H26 | 119.2 |
| N4—C20—C21 | 116.7 (4) | C14—C15—C16 | 120.4 (4) |

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| C9—C8—C7 | 104.5 (4) | C14—C15—H15 | 119.8 |
| C9—C8—C18 | 128.7 (4) | C16—C15—H15 | 119.8 |
| C7—C8—C18 | 126.8 (4) | C5—C6—C1 | 119.5 (4) |
| N3—C10—H10A | 109.5 | C5—C6—H6 | 120.2 |
| N3—C10—H10B | 109.5 | C1—C6—H6 | 120.2 |
| H10A—C10—H10B | 109.5 | C15—C14—C13 | 119.7 (4) |
| N3—C10—H10C | 109.5 | C15—C14—H14 | 120.1 |
| H10A—C10—H10C | 109.5 | C13—C14—H14 | 120.1 |
| H10B—C10—H10C | 109.5 | O3—C18—C8 | 120.6 (4) |
| C7—C40—H40A | 109.5 | O3—C18—C19 | 118.7 (4) |
| C7—C40—H40B | 109.5 | C8—C18—C19 | 120.6 (4) |
| H40A—C40—H40B | 109.5 | C26—C25—C24 | 120.1 (5) |
| C7—C40—H40C | 109.5 | C26—C25—H25 | 120 |
| H40A—C40—H40C | 109.5 | C24—C25—H25 | 120 |
| H40B—C40—H40C | 109.5 | C18—C19—H19A | 109.5 |
| C13—C12—C17 | 118.8 (4) | C18—C19—H19B | 109.5 |
| C13—C12—C11 | 120.7 (4) | H19A—C19—H19B | 109.5 |
| C17—C12—C11 | 120.2 (4) | C18—C19—H19C | 109.5 |
| O1—C11—N3 | 120.6 (4) | H19A—C19—H19C | 109.5 |
| O1—C11—C12 | 120.7 (4) | H19B—C19—H19C | 109.5 |
| N3—C11—C12 | 118.5 (4) | C26—C27—C22 | 119.9 (5) |
| C3—C4—C5 | 119.6 (4) | C26—C27—H27 | 120 |
| C3—C4—H4 | 120.2 | C22—C27—H27 | 120 |
| | | | |
| O2—C28—N4—C20 | −178.9 (4) | C10—N3—C11—O1 | 173.4 (4) |
| C23—C28—N4—C20 | −0.7 (5) | C9—N3—C11—C12 | 169.3 (4) |
| O2—C28—N4—C17 | −0.1 (5) | C10—N3—C11—C12 | −10.6 (6) |
| C23—C28—N4—C17 | 178.1 (3) | C13—C12—C11—O1 | 115.6 (5) |
| C7—N2—N1—C9 | 0.0 (4) | C17—C12—C11—O1 | −58.4 (6) |
| C7—N2—N1—C1 | −176.5 (4) | C13—C12—C11—N3 | −60.4 (5) |
| N2—N1—C9—C8 | 0.2 (4) | C17—C12—C11—N3 | 125.5 (4) |
| C1—N1—C9—C8 | 176.4 (4) | N1—N2—C7—C8 | −0.2 (4) |
| N2—N1—C9—N3 | −176.7 (3) | N1—N2—C7—C40 | −179.4 (3) |
| C1—N1—C9—N3 | −0.5 (6) | C9—C8—C7—N2 | 0.3 (5) |
| C11—N3—C9—N1 | −87.6 (4) | C18—C8—C7—N2 | −177.8 (4) |
| C10—N3—C9—N1 | 92.3 (4) | C9—C8—C7—C40 | 179.4 (4) |
| C11—N3—C9—C8 | 96.5 (5) | C18—C8—C7—C40 | 1.2 (7) |
| C10—N3—C9—C8 | −83.6 (6) | C3—C4—C5—C6 | 0.8 (6) |
| C9—N1—C1—C6 | −106.9 (5) | C5—C4—C3—C2 | −0.2 (7) |
| N2—N1—C1—C6 | 69.0 (5) | C17—C12—C13—C14 | 0.2 (6) |
| C9—N1—C1—C2 | 71.4 (5) | C11—C12—C13—C14 | −173.9 (4) |
| N2—N1—C1—C2 | −112.6 (4) | C20—N5—C22—C27 | −179.1 (4) |
| O2—C28—C23—C24 | −3.9 (6) | C20—N5—C22—C23 | −0.3 (6) |
| N4—C28—C23—C24 | 177.9 (3) | C24—C23—C22—N5 | −177.1 (4) |
| O2—C28—C23—C22 | 174.7 (4) | C28—C23—C22—N5 | 4.2 (6) |
| N4—C28—C23—C22 | −3.4 (5) | C24—C23—C22—C27 | 1.6 (6) |
| C15—C16—C17—C12 | −1.6 (6) | C28—C23—C22—C27 | −177.1 (4) |
| C15—C16—C17—N4 | 179.4 (4) | C6—C1—C2—C3 | 2.2 (6) |

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| C28—N4—C17—C16 | 89.6 (4) | N1—C1—C2—C3 | −176.2 (4) |
| C20—N4—C17—C16 | −91.6 (5) | C4—C3—C2—C1 | −1.2 (6) |
| C28—N4—C17—C12 | −89.4 (4) | C22—C23—C24—C25 | −0.1 (6) |
| C20—N4—C17—C12 | 89.4 (5) | C28—C23—C24—C25 | 178.5 (4) |
| C22—N5—C20—N4 | −4.2 (6) | C17—C16—C15—C14 | 1.1 (7) |
| C22—N5—C20—C21 | 174.7 (3) | C4—C5—C6—C1 | 0.1 (6) |
| C28—N4—C20—N5 | 4.9 (6) | C2—C1—C6—C5 | −1.6 (6) |
| C17—N4—C20—N5 | −173.9 (4) | N1—C1—C6—C5 | 176.8 (4) |
| C28—N4—C20—C21 | −174.0 (3) | C16—C15—C14—C13 | 0.1 (7) |
| C17—N4—C20—C21 | 7.2 (5) | C12—C13—C14—C15 | −0.8 (6) |
| N1—C9—C8—C7 | −0.3 (4) | C9—C8—C18—O3 | 177.9 (4) |
| N3—C9—C8—C7 | 176.0 (4) | C7—C8—C18—O3 | −4.3 (7) |
| N1—C9—C8—C18 | 177.8 (4) | C9—C8—C18—C19 | −5.9 (7) |
| N3—C9—C8—C18 | −5.9 (8) | C7—C8—C18—C19 | 171.8 (4) |
| C16—C17—C12—C13 | 1.0 (6) | C27—C26—C25—C24 | 1.4 (8) |
| N4—C17—C12—C13 | 180.0 (4) | C23—C24—C25—C26 | −1.3 (7) |
| C16—C17—C12—C11 | 175.1 (4) | C25—C26—C27—C22 | 0.1 (7) |
| N4—C17—C12—C11 | −5.9 (6) | N5—C22—C27—C26 | 177.3 (4) |
| C9—N3—C11—O1 | −6.7 (5) | C23—C22—C27—C26 | −1.5 (6) |

*Hydrogen-bond geometry (Å, º)*

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C21—H21$C$···O1 | 0.96 | 2.57 | 3.214 (5) | 124 |
| C3—H3···O2[i] | 0.93 | 2.54 | 3.351 (6) | 146 |
| C5—H5···O1[ii] | 0.93 | 2.40 | 3.276 (5) | 157 |
| C16—H16···O3[iii] | 0.93 | 2.52 | 3.305 (6) | 143 |

Symmetry codes: (i) $x+1, y, z$; (ii) $x, -y+1/2, z-1/2$; (iii) $-x, y+1/2, -z+1/2$.