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# 4-[(*E*)-3-Methoxy-5-nitro-4-(4-nitrobenzyloxy)benzylideneamino]-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one

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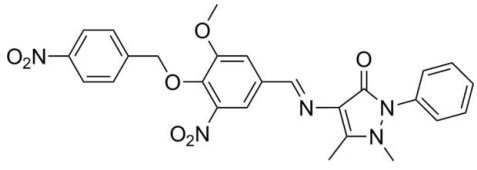
Key indicators: single-crystal X-ray study; $T$ = 294 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.048; $wR$ factor = 0.139; data-to-parameter ratio = 12.6.

In the title compound, $C_{26}H_{23}N_5O_7$, the central benzene ring makes dihedral angles of 35.08 (6), 48.75 (7) and 69.55 (8)° with the pyrazolone ring, the nitrobenzene ring and the terminal phenyl ring, respectively. An intramolecular C—H···O interaction generates an $S$(6) ring. The packing is stabilized by weak nonclassical intermolecular C—H···O=C hydrogen bonds that link adjacent molecules into chains.

## Related literature

For general background to related compounds, see: Chen & Yu (2006); Li *et al.* (2005); Santos *et al.* (2001); Zhang *et al.* (2006). For reference bond lengths, see: Allen *et al.* (1987).



## Experimental

### *Crystal data*

$C_{26}H_{23}N_5O_7$
$M_r$ = 517.49
Monoclinic, $C2/c$
$a$ = 25.671 (11) Å
$b$ = 11.933 (5) Å
$c$ = 16.617 (7) Å
$\beta$ = 103.972 (7)°
$V$ = 4940 (4) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 0.10 mm$^{-1}$
$T$ = 294 K
0.20 × 0.18 × 0.10 mm

### *Data collection*

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.938, $T_{max}$ = 0.989
12593 measured reflections
4363 independent reflections
2652 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.039

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.139
$S$ = 1.02
4363 reflections
346 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.39 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.21 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C15—H15···O7 | 0.93 | 2.35 | 2.997 (3) | 127 |
| C4—H4···O7$^{i}$ | 0.93 | 2.56 | 3.447 (3) | 159 |

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

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5658).

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# supporting information

*Acta Cryst.* (2010). E**66**, o2783 [https://doi.org/10.1107/S1600536810039152]

## 4-[(*E*)-3-Methoxy-5-nitro-4-(4-nitrobenzyloxy)benzylideneamino]-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one

**Lei Liao**

### S1. Comment

Schiff bases have extensively been studied because of their significant biological activity such as protein and enzyme mimics (Santos *et al.*, 2001). Consequently, many Schiff base derivatives have been synthesized and employed to develop protein and enzyme mimics such as models to mimic hydrolase in the hydrolysis of *p*-nitrophenyl picolinate (Li *et al.*, 2005).

Among the large number of compounds, 4-amino-1,5-dimethyl-2-phenylpyrazol-3-one forms a variety of Schiff bases with aldehydes, and the synthesis and crystal structures of some of them, such as (*E*)-4-(4-(4-chlorobenzyloxy)-3-ethoxybenzylideneamino) -1,5-dimethyl-2-phenyl-1,2-dihydropyrazol-3-one (Zhang *et al.*, 2006) and (*E*)-2-(2-((1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1*H*-pyrazol-4-ylimino) methyl)phenoxy)ethyl 4-methylbenzenesulfonate(Chen & Yu, 2006) have been reported.

Structural information is useful when investigating the coordination properties of Schiff bases functioning as ligands. We report here the synthesis and molecular structure of the title Schiff base compound, (I), (Fig. 1)

In the title molecule (Fig. 1), bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The pyrazolone ring (C16—C18/N3—N5/O7) is nearly planar, with an r.m.s. deviation for fitted atoms of 0.0379 Å. It makes a dihedral angle of 44.37 (9)° with the attached phenyl ring (C21—C26). The central benzene ring (C8—C13/C15/O3/O4) is almost planar, with an r.m.s. deviation for fitted atoms of 0.0342 Å. This group makes dihedral angles of 35.08 (6)°, 48.75 (7)° and 69.55 (8)°, respectively, with the the pyrazolone ring (C16—C18/N3—N5/O7), the nitrobenzene ring (C1—C6) and the terminal phenyl ring (C21—C26).

An intramolecular C15—H15···O7═C17 hydrogen bond is found in (I) (Table 1), which helps to stabilize the conformation of the molecule. Packing is stabilized by weak, non-classical intermolecular C4—H4···O7═C117 hydrogen bonds that link adjacent molecules into one-dimensional chains (Table 1, Fig. 2).

### S2. Experimental

An anhydrous ethanol solution (100 ml) of 3-methoxy-5-nitro-4-(4-nitrobenzyloxy)benzaldehyde (3.32 g, 10 mmol) was added to an anhydrous ethanol solution (50 ml) of 4-amino-1,5-dimethyl-2-phenylpyrazol-3-one (2.03 g, 10 mmol) and the mixture stirred at 350 K for 3 h under $N_2$, giving a yellow precipitate. The product was isolated, recrystallized from acetonitrile, and then dried in a vacuum to give pure compound (I) in 79% yield. Yellow blocks of (I) were obtained by slow evaporation of an acetonitrile solution.

### S3. Refinement

The H atoms were included in calculated positions and refined using a riding model approximation. Constrained C—H bond lengths and isotropic U parameters: 0.93 Å and $U_{iso}$(H) = 1.2$U_{eq}$(C) for C$sp^2$—H; 0.97 Å and $U_{iso}$(H) = 1.2$U_{eq}$(C) for

methylene C—H; 0.96 Å and $U_{iso}$(H) = 1.5$U_{eq}$(C) for methyl C—H.

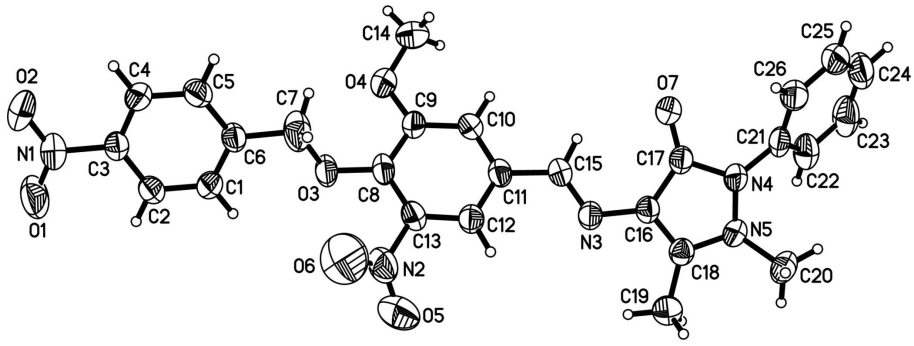


**Figure 1**

The structure of (I), with displacement ellipsoids for non-H atoms drawn at the 50% probability level.

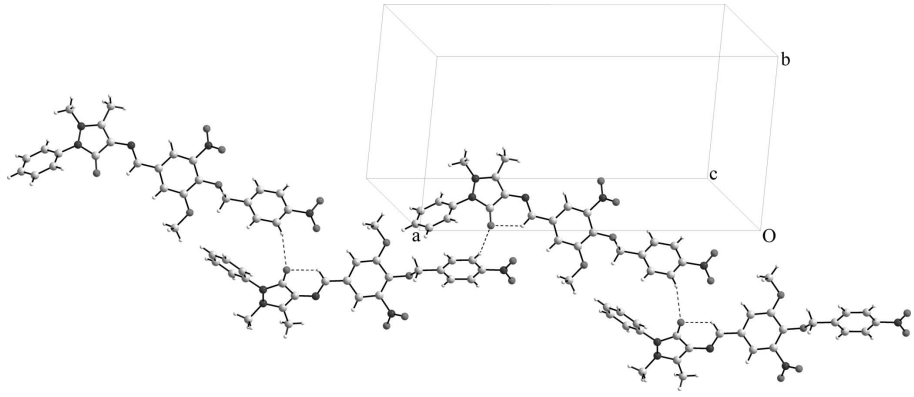


**Figure 2**

A packing diagram for (I), with hydrogen bonds drawn as dashed lines.

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*Crystal data*

$C_{26}H_{23}N_5O_7$
$M_r$ = 517.49
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
*a* = 25.671 (11) Å
*b* = 11.933 (5) Å
*c* = 16.617 (7) Å
*β* = 103.972 (7)°
*V* = 4940 (4) Å$^3$
*Z* = 8
*F*(000) = 2160
$D_x$ = 1.392 Mg m$^{-3}$
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 3086 reflections
*θ* = 2.5–23.1°
*μ* = 0.10 mm$^{-1}$
*T* = 294 K
Block, yellow
0.20 × 0.18 × 0.10 mm

*Data collection*

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
*φ* and *ω* scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.938, $T_{max}$ = 0.989
12593 measured reflections
4363 independent reflections
2652 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.039
$\theta_{max}$ = 25.0°, $\theta_{min}$ = 1.6°
*h* = −30→30
*k* = −10→14
*l* = −18→19

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.139$
$S = 1.02$
4363 reflections
346 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0693P)^2 + 1.2662P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.39$ e Å$^{-3}$
$\Delta\rho_{min} = -0.21$ 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 $F^2 > 2\sigma(F^2)$ 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| N1 | 0.17786 (9) | −0.1619 (2) | −0.12948 (15) | 0.0577 (6) |
| N2 | 0.45460 (10) | 0.1575 (2) | 0.1025 (2) | 0.0746 (8) |
| N3 | 0.65269 (7) | 0.10798 (16) | 0.27094 (12) | 0.0441 (5) |
| N4 | 0.79727 (7) | 0.09102 (16) | 0.33957 (12) | 0.0442 (5) |
| N5 | 0.78530 (8) | 0.18315 (16) | 0.38510 (13) | 0.0456 (5) |
| O1 | 0.14745 (8) | −0.0959 (2) | −0.10853 (15) | 0.0924 (8) |
| O2 | 0.16274 (8) | −0.23197 (19) | −0.18340 (15) | 0.0838 (7) |
| O3 | 0.41585 (6) | −0.06411 (14) | 0.08900 (11) | 0.0522 (5) |
| O4 | 0.48334 (7) | −0.23900 (15) | 0.14053 (13) | 0.0675 (6) |
| O5 | 0.45720 (10) | 0.2406 (2) | 0.1478 (2) | 0.1103 (9) |
| O6 | 0.42595 (15) | 0.1535 (3) | 0.0360 (2) | 0.1733 (17) |
| O7 | 0.74844 (6) | −0.04709 (15) | 0.25573 (11) | 0.0559 (5) |
| C1 | 0.30688 (10) | −0.06890 (19) | 0.00891 (16) | 0.0471 (6) |
| H1 | 0.3190 | −0.0199 | 0.0530 | 0.057* |
| C2 | 0.25251 (10) | −0.0791 (2) | −0.02563 (16) | 0.0471 (6) |
| H2 | 0.2279 | −0.0369 | −0.0056 | 0.057* |
| C3 | 0.23550 (9) | −0.15309 (19) | −0.09037 (15) | 0.0402 (6) |
| C4 | 0.27074 (10) | −0.2174 (2) | −0.12056 (16) | 0.0479 (6) |
| H4 | 0.2584 | −0.2679 | −0.1636 | 0.057* |
| C5 | 0.32471 (10) | −0.2054 (2) | −0.08570 (16) | 0.0524 (7) |
| H5 | 0.3491 | −0.2483 | −0.1058 | 0.063* |
| C6 | 0.34367 (9) | −0.1309 (2) | −0.02133 (15) | 0.0442 (6) |
| C7 | 0.40340 (11) | −0.1185 (3) | 0.01048 (19) | 0.0792 (10) |
| H7A | 0.4201 | −0.1919 | 0.0156 | 0.095* |
| H7B | 0.4176 | −0.0752 | −0.0287 | 0.095* |

| | | | | |
|---|---|---|---|---|
| C8 | 0.46952 (9) | −0.0463 (2) | 0.12145 (15) | 0.0445 (6) |
| C9 | 0.50518 (9) | −0.1341 (2) | 0.14976 (16) | 0.0454 (6) |
| C10 | 0.55822 (9) | −0.1122 (2) | 0.18748 (16) | 0.0462 (6) |
| H10 | 0.5818 | −0.1716 | 0.2042 | 0.055* |
| C11 | 0.57716 (9) | −0.0028 (2) | 0.20097 (15) | 0.0411 (6) |
| C12 | 0.54226 (9) | 0.0855 (2) | 0.17466 (15) | 0.0455 (6) |
| H12 | 0.5539 | 0.1593 | 0.1839 | 0.055* |
| C13 | 0.48995 (9) | 0.0621 (2) | 0.13456 (16) | 0.0459 (6) |
| C14 | 0.51973 (13) | −0.3321 (2) | 0.1583 (2) | 0.0860 (11) |
| H14A | 0.5425 | −0.3321 | 0.1202 | 0.129* |
| H14B | 0.4997 | −0.4007 | 0.1528 | 0.129* |
| H14C | 0.5414 | −0.3256 | 0.2140 | 0.129* |
| C15 | 0.63347 (9) | 0.0136 (2) | 0.24335 (16) | 0.0452 (6) |
| H15 | 0.6563 | −0.0480 | 0.2505 | 0.054* |
| C16 | 0.70778 (9) | 0.11570 (18) | 0.30688 (14) | 0.0391 (6) |
| C17 | 0.74983 (9) | 0.0415 (2) | 0.29486 (15) | 0.0418 (6) |
| C18 | 0.73083 (9) | 0.20013 (19) | 0.35782 (15) | 0.0414 (6) |
| C19 | 0.70450 (11) | 0.3001 (2) | 0.38397 (17) | 0.0575 (7) |
| H19A | 0.7146 | 0.3658 | 0.3580 | 0.086* |
| H19B | 0.7157 | 0.3080 | 0.4431 | 0.086* |
| H19C | 0.6662 | 0.2910 | 0.3677 | 0.086* |
| C20 | 0.82277 (11) | 0.2781 (2) | 0.39304 (18) | 0.0595 (7) |
| H20A | 0.8205 | 0.3102 | 0.3394 | 0.089* |
| H20B | 0.8587 | 0.2522 | 0.4160 | 0.089* |
| H20C | 0.8135 | 0.3337 | 0.4290 | 0.089* |
| C21 | 0.84699 (9) | 0.03300 (19) | 0.36710 (16) | 0.0447 (6) |
| C22 | 0.87379 (10) | 0.0311 (2) | 0.45004 (18) | 0.0596 (7) |
| H22 | 0.8601 | 0.0695 | 0.4891 | 0.072* |
| C23 | 0.92118 (12) | −0.0284 (3) | 0.4743 (2) | 0.0748 (10) |
| H23 | 0.9394 | −0.0296 | 0.5299 | 0.090* |
| C24 | 0.94167 (12) | −0.0855 (3) | 0.4174 (3) | 0.0758 (10) |
| H24 | 0.9734 | −0.1258 | 0.4346 | 0.091* |
| C25 | 0.91524 (11) | −0.0832 (2) | 0.3348 (2) | 0.0677 (9) |
| H25 | 0.9292 | −0.1219 | 0.2962 | 0.081* |
| C26 | 0.86791 (10) | −0.0234 (2) | 0.30924 (19) | 0.0549 (7) |
| H26 | 0.8502 | −0.0212 | 0.2534 | 0.066* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.0418 (14) | 0.0746 (16) | 0.0518 (15) | −0.0035 (12) | 0.0019 (12) | 0.0040 (13) |
| N2 | 0.0522 (16) | 0.0634 (18) | 0.095 (2) | 0.0082 (13) | −0.0074 (15) | 0.0167 (16) |
| N3 | 0.0350 (12) | 0.0457 (12) | 0.0496 (13) | −0.0031 (9) | 0.0065 (10) | 0.0016 (10) |
| N4 | 0.0339 (12) | 0.0459 (12) | 0.0482 (13) | −0.0017 (9) | 0.0009 (9) | −0.0022 (10) |
| N5 | 0.0396 (12) | 0.0440 (12) | 0.0499 (13) | −0.0071 (9) | 0.0045 (10) | −0.0059 (10) |
| O1 | 0.0398 (12) | 0.141 (2) | 0.0917 (18) | 0.0139 (13) | 0.0061 (11) | −0.0275 (15) |
| O2 | 0.0578 (14) | 0.0940 (16) | 0.0865 (17) | −0.0192 (11) | −0.0082 (12) | −0.0233 (13) |
| O3 | 0.0289 (9) | 0.0750 (12) | 0.0497 (11) | 0.0002 (8) | 0.0037 (8) | −0.0143 (9) |

| | | | | | | |
|---|---|---|---|---|---|---|
| O4 | 0.0459 (11) | 0.0504 (11) | 0.0977 (16) | −0.0049 (9) | 0.0007 (10) | −0.0121 (10) |
| O5 | 0.0948 (19) | 0.0689 (16) | 0.159 (3) | 0.0270 (14) | 0.0149 (17) | 0.0107 (17) |
| O6 | 0.175 (3) | 0.122 (2) | 0.155 (3) | 0.037 (2) | −0.093 (3) | 0.025 (2) |
| O7 | 0.0407 (10) | 0.0532 (11) | 0.0691 (13) | −0.0016 (8) | 0.0043 (9) | −0.0156 (10) |
| C1 | 0.0434 (16) | 0.0509 (15) | 0.0441 (16) | −0.0005 (12) | 0.0050 (12) | −0.0112 (12) |
| C2 | 0.0387 (15) | 0.0512 (15) | 0.0504 (16) | 0.0070 (11) | 0.0089 (12) | −0.0030 (13) |
| C3 | 0.0353 (14) | 0.0419 (14) | 0.0409 (15) | −0.0028 (11) | 0.0043 (11) | 0.0061 (11) |
| C4 | 0.0480 (16) | 0.0460 (15) | 0.0449 (16) | −0.0012 (12) | 0.0020 (12) | −0.0068 (12) |
| C5 | 0.0468 (17) | 0.0593 (17) | 0.0493 (17) | 0.0116 (12) | 0.0082 (13) | −0.0090 (13) |
| C6 | 0.0351 (14) | 0.0553 (15) | 0.0395 (15) | 0.0038 (11) | 0.0037 (11) | −0.0042 (12) |
| C7 | 0.0435 (18) | 0.134 (3) | 0.057 (2) | 0.0033 (17) | 0.0059 (15) | −0.0360 (19) |
| C8 | 0.0284 (13) | 0.0637 (17) | 0.0404 (15) | −0.0015 (12) | 0.0061 (11) | −0.0036 (12) |
| C9 | 0.0333 (14) | 0.0495 (15) | 0.0525 (17) | −0.0038 (12) | 0.0087 (12) | −0.0087 (12) |
| C10 | 0.0361 (15) | 0.0474 (15) | 0.0532 (17) | 0.0056 (11) | 0.0070 (12) | −0.0056 (12) |
| C11 | 0.0319 (13) | 0.0514 (15) | 0.0404 (15) | −0.0003 (11) | 0.0095 (11) | −0.0023 (11) |
| C12 | 0.0378 (15) | 0.0454 (15) | 0.0516 (17) | −0.0024 (11) | 0.0075 (12) | 0.0027 (12) |
| C13 | 0.0344 (14) | 0.0513 (16) | 0.0498 (16) | 0.0056 (12) | 0.0057 (12) | 0.0061 (12) |
| C14 | 0.072 (2) | 0.0483 (18) | 0.129 (3) | 0.0055 (16) | 0.009 (2) | −0.0209 (18) |
| C15 | 0.0331 (14) | 0.0484 (15) | 0.0519 (17) | 0.0025 (11) | 0.0061 (12) | −0.0009 (12) |
| C16 | 0.0342 (14) | 0.0410 (13) | 0.0400 (14) | −0.0017 (10) | 0.0050 (11) | 0.0034 (11) |
| C17 | 0.0350 (14) | 0.0425 (14) | 0.0450 (15) | −0.0034 (11) | 0.0040 (11) | 0.0025 (12) |
| C18 | 0.0394 (15) | 0.0420 (14) | 0.0420 (15) | −0.0020 (11) | 0.0081 (11) | 0.0037 (11) |
| C19 | 0.0559 (17) | 0.0566 (17) | 0.0601 (19) | 0.0004 (13) | 0.0140 (14) | −0.0072 (14) |
| C20 | 0.0500 (17) | 0.0547 (17) | 0.069 (2) | −0.0153 (13) | 0.0060 (14) | −0.0055 (14) |
| C21 | 0.0302 (14) | 0.0433 (14) | 0.0566 (18) | −0.0069 (11) | 0.0027 (12) | 0.0102 (12) |
| C22 | 0.0435 (16) | 0.0712 (19) | 0.0592 (19) | −0.0060 (14) | 0.0028 (14) | 0.0157 (15) |
| C23 | 0.0453 (18) | 0.085 (2) | 0.082 (2) | −0.0057 (16) | −0.0090 (17) | 0.0290 (19) |
| C24 | 0.0403 (18) | 0.059 (2) | 0.123 (3) | 0.0019 (14) | 0.009 (2) | 0.023 (2) |
| C25 | 0.0491 (19) | 0.0482 (17) | 0.106 (3) | −0.0026 (13) | 0.0195 (18) | 0.0006 (16) |
| C26 | 0.0462 (17) | 0.0502 (16) | 0.066 (2) | −0.0015 (12) | 0.0085 (14) | 0.0014 (14) |

*Geometric parameters (Å, º)*

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| N1—O1 | 1.218 (3) | C8—C9 | 1.396 (3) |
| N1—O2 | 1.218 (3) | C9—C10 | 1.379 (3) |
| N1—C3 | 1.469 (3) | C10—C11 | 1.392 (3) |
| N2—O6 | 1.171 (4) | C10—H10 | 0.9300 |
| N2—O5 | 1.238 (3) | C11—C12 | 1.385 (3) |
| N2—C13 | 1.473 (3) | C11—C15 | 1.460 (3) |
| N3—C15 | 1.270 (3) | C12—C13 | 1.376 (3) |
| N3—C16 | 1.399 (3) | C12—H12 | 0.9300 |
| N4—C17 | 1.396 (3) | C14—H14A | 0.9600 |
| N4—N5 | 1.410 (3) | C14—H14B | 0.9600 |
| N4—C21 | 1.427 (3) | C14—H14C | 0.9600 |
| N5—C18 | 1.377 (3) | C15—H15 | 0.9300 |
| N5—C20 | 1.471 (3) | C16—C18 | 1.356 (3) |
| O3—C8 | 1.370 (3) | C16—C17 | 1.447 (3) |
| O3—C7 | 1.423 (3) | C18—C19 | 1.487 (3) |

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|---|---|---|---|
| O4—C9 | 1.365 (3) | C19—H19A | 0.9600 |
| O4—C14 | 1.435 (3) | C19—H19B | 0.9600 |
| O7—C17 | 1.237 (3) | C19—H19C | 0.9600 |
| C1—C2 | 1.380 (3) | C20—H20A | 0.9600 |
| C1—C6 | 1.386 (3) | C20—H20B | 0.9600 |
| C1—H1 | 0.9300 | C20—H20C | 0.9600 |
| C2—C3 | 1.379 (3) | C21—C22 | 1.384 (4) |
| C2—H2 | 0.9300 | C21—C26 | 1.384 (4) |
| C3—C4 | 1.371 (3) | C22—C23 | 1.382 (4) |
| C4—C5 | 1.374 (3) | C22—H22 | 0.9300 |
| C4—H4 | 0.9300 | C23—C24 | 1.369 (5) |
| C5—C6 | 1.385 (3) | C23—H23 | 0.9300 |
| C5—H5 | 0.9300 | C24—C25 | 1.376 (5) |
| C6—C7 | 1.504 (3) | C24—H24 | 0.9300 |
| C7—H7A | 0.9700 | C25—C26 | 1.385 (4) |
| C7—H7B | 0.9700 | C25—H25 | 0.9300 |
| C8—C13 | 1.393 (3) | C26—H26 | 0.9300 |

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|---|---|---|---|
| O1—N1—O2 | 122.8 (2) | C11—C12—H12 | 120.7 |
| O1—N1—C3 | 118.3 (2) | C12—C13—C8 | 123.4 (2) |
| O2—N1—C3 | 118.9 (2) | C12—C13—N2 | 117.6 (2) |
| O6—N2—O5 | 122.4 (3) | C8—C13—N2 | 119.0 (2) |
| O6—N2—C13 | 120.1 (3) | O4—C14—H14A | 109.5 |
| O5—N2—C13 | 117.5 (3) | O4—C14—H14B | 109.5 |
| C15—N3—C16 | 118.5 (2) | H14A—C14—H14B | 109.5 |
| C17—N4—N5 | 109.92 (18) | O4—C14—H14C | 109.5 |
| C17—N4—C21 | 124.28 (19) | H14A—C14—H14C | 109.5 |
| N5—N4—C21 | 120.02 (19) | H14B—C14—H14C | 109.5 |
| C18—N5—N4 | 105.89 (18) | N3—C15—C11 | 123.1 (2) |
| C18—N5—C20 | 120.4 (2) | N3—C15—H15 | 118.5 |
| N4—N5—C20 | 115.20 (19) | C11—C15—H15 | 118.5 |
| C8—O3—C7 | 114.45 (18) | C18—C16—N3 | 124.0 (2) |
| C9—O4—C14 | 117.2 (2) | C18—C16—C17 | 108.3 (2) |
| C2—C1—C6 | 120.7 (2) | N3—C16—C17 | 127.7 (2) |
| C2—C1—H1 | 119.6 | O7—C17—N4 | 123.5 (2) |
| C6—C1—H1 | 119.6 | O7—C17—C16 | 131.9 (2) |
| C3—C2—C1 | 118.6 (2) | N4—C17—C16 | 104.6 (2) |
| C3—C2—H2 | 120.7 | C16—C18—N5 | 110.6 (2) |
| C1—C2—H2 | 120.7 | C16—C18—C19 | 128.2 (2) |
| C4—C3—C2 | 122.1 (2) | N5—C18—C19 | 121.3 (2) |
| C4—C3—N1 | 118.8 (2) | C18—C19—H19A | 109.5 |
| C2—C3—N1 | 119.1 (2) | C18—C19—H19B | 109.5 |
| C3—C4—C5 | 118.3 (2) | H19A—C19—H19B | 109.5 |
| C3—C4—H4 | 120.8 | C18—C19—H19C | 109.5 |
| C5—C4—H4 | 120.8 | H19A—C19—H19C | 109.5 |
| C4—C5—C6 | 121.5 (2) | H19B—C19—H19C | 109.5 |
| C4—C5—H5 | 119.2 | N5—C20—H20A | 109.5 |
| C6—C5—H5 | 119.2 | N5—C20—H20B | 109.5 |

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| C5—C6—C1 | 118.6 (2) | H20A—C20—H20B | 109.5 |
| C5—C6—C7 | 118.2 (2) | N5—C20—H20C | 109.5 |
| C1—C6—C7 | 123.1 (2) | H20A—C20—H20C | 109.5 |
| O3—C7—C6 | 110.6 (2) | H20B—C20—H20C | 109.5 |
| O3—C7—H7A | 109.5 | C22—C21—C26 | 120.1 (2) |
| C6—C7—H7A | 109.5 | C22—C21—N4 | 121.2 (2) |
| O3—C7—H7B | 109.5 | C26—C21—N4 | 118.7 (2) |
| C6—C7—H7B | 109.5 | C23—C22—C21 | 119.3 (3) |
| H7A—C7—H7B | 108.1 | C23—C22—H22 | 120.3 |
| O3—C8—C13 | 120.7 (2) | C21—C22—H22 | 120.3 |
| O3—C8—C9 | 122.1 (2) | C24—C23—C22 | 120.9 (3) |
| C13—C8—C9 | 116.9 (2) | C24—C23—H23 | 119.6 |
| O4—C9—C10 | 123.9 (2) | C22—C23—H23 | 119.6 |
| O4—C9—C8 | 115.6 (2) | C23—C24—C25 | 119.9 (3) |
| C10—C9—C8 | 120.4 (2) | C23—C24—H24 | 120.0 |
| C9—C10—C11 | 121.3 (2) | C25—C24—H24 | 120.0 |
| C9—C10—H10 | 119.4 | C24—C25—C26 | 120.1 (3) |
| C11—C10—H10 | 119.4 | C24—C25—H25 | 120.0 |
| C12—C11—C10 | 119.3 (2) | C26—C25—H25 | 120.0 |
| C12—C11—C15 | 122.7 (2) | C21—C26—C25 | 119.8 (3) |
| C10—C11—C15 | 118.0 (2) | C21—C26—H26 | 120.1 |
| C13—C12—C11 | 118.7 (2) | C25—C26—H26 | 120.1 |
| C13—C12—H12 | 120.7 | | |

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|---|---|---|---|
| C17—N4—N5—C18 | −9.2 (2) | O3—C8—C13—N2 | −8.4 (4) |
| C21—N4—N5—C18 | −163.58 (19) | C9—C8—C13—N2 | 177.2 (2) |
| C17—N4—N5—C20 | −145.0 (2) | O6—N2—C13—C12 | 135.8 (4) |
| C21—N4—N5—C20 | 60.7 (3) | O5—N2—C13—C12 | −43.2 (4) |
| C6—C1—C2—C3 | 0.6 (4) | O6—N2—C13—C8 | −42.7 (5) |
| C1—C2—C3—C4 | 0.8 (4) | O5—N2—C13—C8 | 138.2 (3) |
| C1—C2—C3—N1 | −178.0 (2) | C16—N3—C15—C11 | −176.8 (2) |
| O1—N1—C3—C4 | −172.0 (2) | C12—C11—C15—N3 | 11.5 (4) |
| O2—N1—C3—C4 | 5.4 (3) | C10—C11—C15—N3 | −168.0 (2) |
| O1—N1—C3—C2 | 6.8 (3) | C15—N3—C16—C18 | −161.3 (2) |
| O2—N1—C3—C2 | −175.8 (2) | C15—N3—C16—C17 | 21.5 (4) |
| C2—C3—C4—C5 | −1.3 (4) | N5—N4—C17—O7 | −173.0 (2) |
| N1—C3—C4—C5 | 177.5 (2) | C21—N4—C17—O7 | −20.0 (4) |
| C3—C4—C5—C6 | 0.3 (4) | N5—N4—C17—C16 | 6.8 (2) |
| C4—C5—C6—C1 | 1.0 (4) | C21—N4—C17—C16 | 159.8 (2) |
| C4—C5—C6—C7 | −176.9 (3) | C18—C16—C17—O7 | 178.0 (3) |
| C2—C1—C6—C5 | −1.5 (4) | N3—C16—C17—O7 | −4.5 (4) |
| C2—C1—C6—C7 | 176.4 (3) | C18—C16—C17—N4 | −1.8 (3) |
| C8—O3—C7—C6 | −178.1 (2) | N3—C16—C17—N4 | 175.7 (2) |
| C5—C6—C7—O3 | −164.2 (2) | N3—C16—C18—N5 | 178.4 (2) |
| C1—C6—C7—O3 | 17.9 (4) | C17—C16—C18—N5 | −4.0 (3) |
| C7—O3—C8—C13 | 116.4 (3) | N3—C16—C18—C19 | −1.7 (4) |
| C7—O3—C8—C9 | −69.5 (3) | C17—C16—C18—C19 | 175.9 (2) |
| C14—O4—C9—C10 | −11.0 (4) | N4—N5—C18—C16 | 8.1 (3) |

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|---|---|---|---|
| C14—O4—C9—C8 | 171.8 (3) | C20—N5—C18—C16 | 141.0 (2) |
| O3—C8—C9—O4 | 2.0 (3) | N4—N5—C18—C19 | −171.8 (2) |
| C13—C8—C9—O4 | 176.2 (2) | C20—N5—C18—C19 | −39.0 (3) |
| O3—C8—C9—C10 | −175.3 (2) | C17—N4—C21—C22 | −123.6 (3) |
| C13—C8—C9—C10 | −1.1 (4) | N5—N4—C21—C22 | 26.8 (3) |
| O4—C9—C10—C11 | −174.8 (2) | C17—N4—C21—C26 | 55.4 (3) |
| C8—C9—C10—C11 | 2.3 (4) | N5—N4—C21—C26 | −154.1 (2) |
| C9—C10—C11—C12 | −1.2 (4) | C26—C21—C22—C23 | −0.6 (4) |
| C9—C10—C11—C15 | 178.3 (2) | N4—C21—C22—C23 | 178.4 (2) |
| C10—C11—C12—C13 | −1.1 (4) | C21—C22—C23—C24 | −0.2 (4) |
| C15—C11—C12—C13 | 179.4 (2) | C22—C23—C24—C25 | 0.6 (4) |
| C11—C12—C13—C8 | 2.3 (4) | C23—C24—C25—C26 | −0.1 (4) |
| C11—C12—C13—N2 | −176.2 (2) | C22—C21—C26—C25 | 1.1 (4) |
| O3—C8—C13—C12 | 173.1 (2) | N4—C21—C26—C25 | −178.0 (2) |
| C9—C8—C13—C12 | −1.2 (4) | C24—C25—C26—C21 | −0.7 (4) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C15—H15···O7 | 0.93 | 2.35 | 2.997 (3) | 127 |
| C4—H4···O7$^{i}$ | 0.93 | 2.56 | 3.447 (3) | 159 |

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