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# Crystal structure of 4-[(*E*)-(4-hydroxybenzylidene)amino]-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one

**Joel T. Mague,[a] Mehmet Akkurt,[b] Shaaban K. Mohamed,[c,d] Alaa F. Mohamed[e] and Mustafa R. Albayati[f]***

[a]Department of Chemistry, Tulane University, New Orleans, LA 70118, USA, [b]Department of Physics, Faculty of Sciences, Erciyes University, 38039 Kayseri, Turkey, [c]Chemistry and Environmental Division, Manchester Metropolitan University, Manchester M1 5GD, England, [d]Chemistry Department, Faculty of Science, Minia University, 61519 El-Minia, Egypt, [e]National Organization for Drug Control and Research, Giza, Egypt, and [f]Kirkuk University, College of Science, Department of Chemistry, Kirkuk, Iraq. *Correspondence e-mail: shaabankamel@yahoo.com

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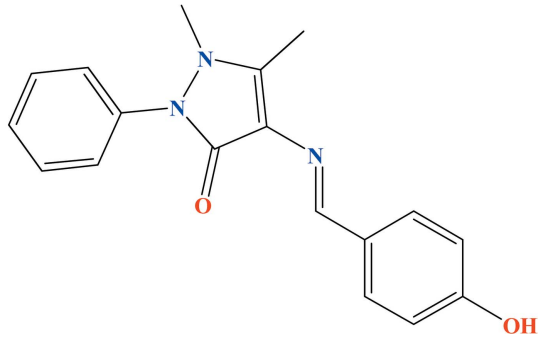
The asymmetric unit of the title compound, $C_{18}H_{17}N_3O_2$, comprises three independent molecules (1, 2 and 3). In molecule 1, the dihedral angles between the pyrazolone ring and the pendant phenyl and hydroxybenzene rings are 54.43 (6) and 28.72 (6)°, respectively. The corresponding data for molecule 2 are 86.84 (6) and 25.69 (5)°, respectively, and for molecule 3 are 47.41 (7) and 17.09 (7)°, respectively. The three molecules feature an intramolecular C—H···O interaction, which closes an *S*(6) ring in each case. In the crystal, molecules are linked by O—H···O hydrogen bonds, which generate [100] chains incorporating all three asymmetric molecules. Two weak C—H···O interactions connect three independent molecules to each other along the c-axis direction.

**Keywords:** crystal structure; pyrazolones; bio-active motifs; hydrogen bonding.

**CCDC reference**: 1436039

## 1. Related literature

For the biological activities of the pyrazolone ring system, see: Nirali & Maulik (2010); Rahat *et al.* (2008); Thakkar & Joshi (2010); Mahmoud *et al.* (2011); Tripathy *et al.* (2007); Brune (1997); Abdel-Aziz *et al.* (2009). For industrial applications, see: Karci & Ertan (2002); Khalil *et al.* (2005); Ho (2005).




## 2. Experimental

### 2.1. Crystal data

$C_{18}H_{17}N_3O_2$
$M_r$ = 307.35
Triclinic, $P\overline{1}$
$a$ = 8.1214 (7) Å
$b$ = 12.5405 (10) Å
$c$ = 23.1875 (19) Å
$\alpha$ = 91.121 (1)°
$\beta$ = 90.199 (1)°
$\gamma$ = 90.748 (1)°
$V$ = 2360.9 (3) Å$^3$
$Z$ = 6
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 150 K
0.30 × 0.18 × 0.15 mm

### 2.2. Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2015)
$T_{min}$ = 0.86, $T_{max}$ = 0.99
22491 measured reflections
11415 independent reflections
7110 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.035

### 2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.115
$S$ = 0.97
11415 reflections
628 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.25 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···$A$ |
|---|---|---|---|---|
| O1—H1···O4 | 0.84 | 1.86 | 2.6947 (14) | 170 |
| O3—H3$A$···O6$^{i}$ | 0.84 | 1.86 | 2.6993 (15) | 175 |
| O5—H5$A$···O4 | 0.84 | 1.88 | 2.7218 (15) | 179 |
| C7—H7···O2 | 0.95 | 2.30 | 3.009 (2) | 131 |
| C25—H25···O4 | 0.95 | 2.46 | 3.0612 (18) | 121 |
| C43—H43···O6 | 0.95 | 2.36 | 2.9989 (18) | 124 |
| C33—H33···O6$^{ii}$ | 0.95 | 2.42 | 3.168 (2) | 135 |
| C35—H35···O2$^{ii}$ | 0.95 | 2.31 | 3.122 (2) | 143 |

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

Data collection: *APEX2* (Bruker, 2015); cell refinement: *SAINT* (Bruker, 2015); data reduction: *SAINT*; program(s) used to solve structure: *SHELXT* (Sheldrick, 2015*b*); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015*a*); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2012); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

## Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7540).

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# supporting information

*Acta Cryst.* (2015). E**71**, o947–o948 [https://doi.org/10.1107/S2056989015021325]

## Crystal structure of 4-[(*E*)-(4-hydroxybenzylidene)amino]-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one

**Joel T. Mague, Mehmet Akkurt, Shaaban K. Mohamed, Alaa F. Mohamed and Mustafa R. Albayati**

### S1. Comment

The pyrazolone ring system is an important structural moiety found in numerous pharmaceutically active compounds. This is mainly due to the ease preparation and the important versatile pharmaceutical and industrial applications. Pyrazolones are mostly useful as anti-inflammatory and analgesic activities (Nirali & Maulik, 2010), but in recent times, they are known to exhibit anti-cancer (Rahat *et al.*, 2008), anti-bacterial (Thakkar & Joshi, 2010) and several other pharmacological actions like anti-fungal (Mahmoud *et al.*, 2011), protein kinase inhibitor (Tripathy *et al.*, 2007), anti-pyretic (Brune, 1997), anti-convulsant (Abdel-Aziz *et al.*, 2009). In addition, they have been used as plant growth regulator, herbicidal and as an azo-dyes (Karci & Ertan, 2002; Khalil *et al.*, 2005; Ho, 2005). Based on such findings and following to our on-going study, we report herein the synthesis and crystal structure of the title compound.

For the title compound there are three independent molecules in the asymmetric unit (Fig. 1) which differ in their conformations. In molecules 1 - 3, respectively, the maximum deviations from the mean planes of the pyrazolone rings are 0.0501 (8) Å (N3), 0.0305 (8) Å (N5) and -0.0367 (8) Å (N8). In molecule 1 the dihedral angles between the mean plane of the pyrazolone ring and those of the phenyl and hydroxyphenyl substituents are, respectively, 54.43 (6)° and 28.72 (6)°. In molecule 2 the corresponding angles are 86.84 (6) and 25.69 (5)° while in molecule 3 they are 47.41 (7) and 17.09 (7)°. In the asymmetric unit, molecules 1 and 3,respectively, form O1—H1···O4 and O5—H5A···O4 hydrogen bonds which tie the unit together (Fig. 1 and Table 1) while O3—H3A···O6$^{i}$ (i: *x* - 1, *y*, *z*) hydrogen bonds aid in the packing (Fig. 2 and Table 1).

### S2. Refinement

H-atoms attached to carbon were placed in calculated positions (C—H = 0.95 - 0.98 Å) while those attached to oxygen were placed in ocations derived from a difference map and their coordinates adjusted to give O—H = 0.84 Å. All were included as riding contributions with isotropic displacement parameters 1.2 - 1.5 times those of the attached atoms.

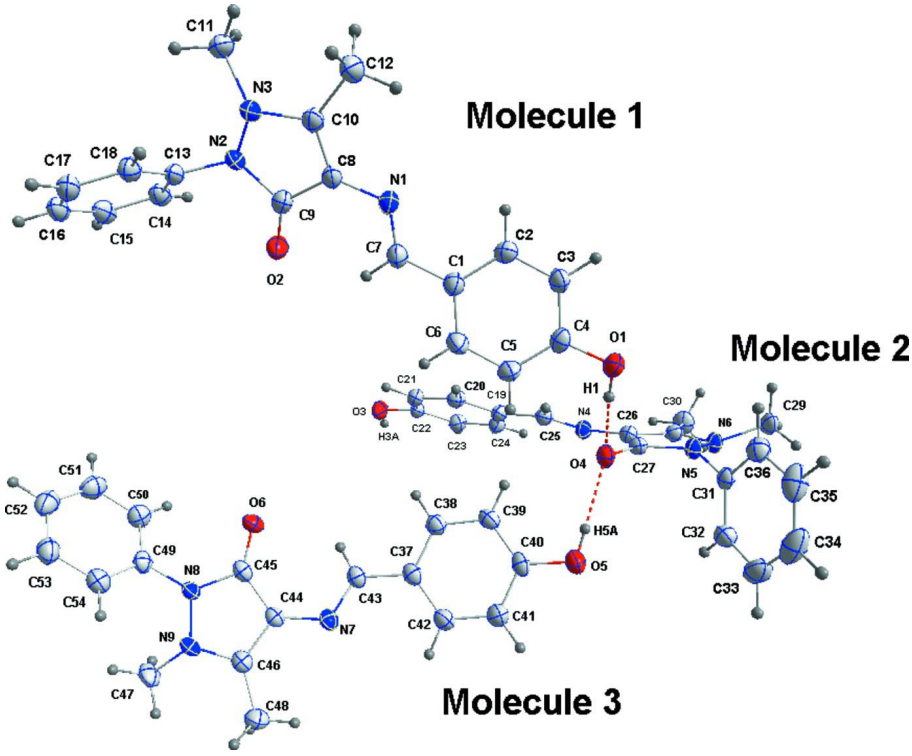


**Figure 1**

The asymmetric unit with labeling scheme and 50% probability ellipsoids. The O—H···O hydrogen bonds are shown as dotted lines.

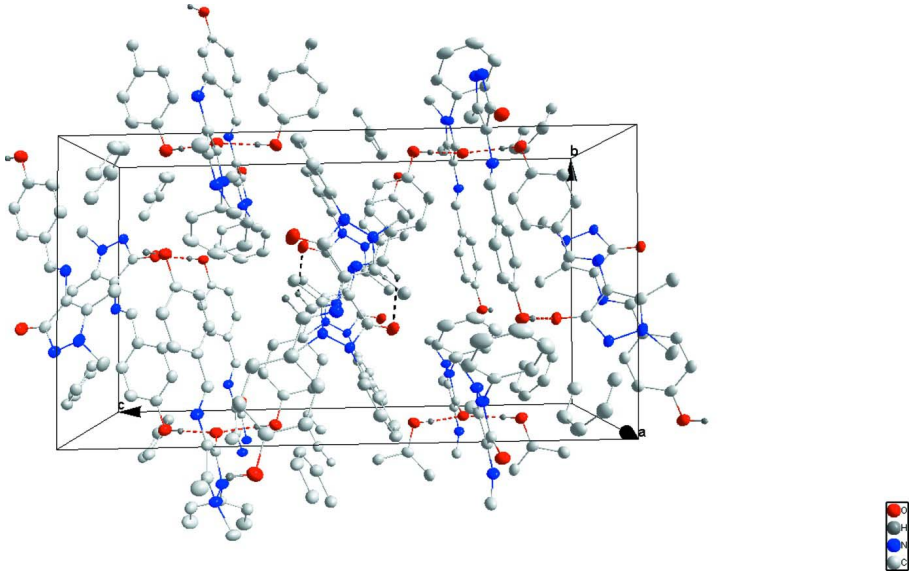


**Figure 2**

The unit-cell contents viewed down the *a* axis. Intermolecular O—H···O and C—H···O hydrogen bonds are shown, respectively, as red and black dotted lines.

**4-[(*E*)-(4-Hydroxybenzylidene)amino]-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one**

*Crystal data*

$C_{18}H_{17}N_3O_2$
$M_r = 307.35$
Triclinic, $P\overline{1}$
$a = 8.1214$ (7) Å
$b = 12.5405$ (10) Å
$c = 23.1875$ (19) Å
$\alpha = 91.121$ (1)°
$\beta = 90.199$ (1)°
$\gamma = 90.748$ (1)°
$V = 2360.9$ (3) Å$^3$
$Z = 6$
$F(000) = 972$
$D_x = 1.297$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6224 reflections
$\theta = 2.4$–$28.2°$
$\mu = 0.09$ mm$^{-1}$
$T = 150$ K
Column, colourless
0.30 × 0.18 × 0.15 mm

*Data collection*

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.3333 pixels mm$^{-1}$
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2015)
$T_{min} = 0.86$, $T_{max} = 0.99$
22491 measured reflections
11415 independent reflections
7110 reflections with $I > 2\sigma(I)$
$R_{int} = 0.035$
$\theta_{max} = 28.5°$, $\theta_{min} = 1.6°$
$h = -10 \rightarrow 10$
$k = -16 \rightarrow 16$
$l = -30 \rightarrow 31$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.115$
$S = 0.97$
11415 reflections
628 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0468P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.25$ e Å$^{-3}$
$\Delta\rho_{min} = -0.21$ e Å$^{-3}$

*Special details*

**Experimental**. The diffraction data were collected in three sets of 363 frames (0.5° width in $\omega$) at $\varphi = 0$, 120 and 240°. A scan time of 40 sec/frame was used.

**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. H-atoms attached to carbon were placed in calculated positions (C—H = 0.95 - 0.98 Å) while those attached to oxygen were placed in ocations derived from a difference map and their coordinates adjusted to give O—H = 0.84%A. All were included as riding contributions with isotropic displacement parameters 1.2 - 1.5 times those of the attached atoms.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.43335 (13) | 0.99066 (8) | 0.36405 (5) | 0.0329 (3) |
| H1 | 0.4041 | 0.9909 | 0.3293 | 0.040* |
| O2 | 0.69023 (15) | 0.34940 (9) | 0.41279 (5) | 0.0414 (3) |
| N1 | 0.69265 (15) | 0.56857 (10) | 0.48144 (5) | 0.0282 (3) |
| N2 | 0.82894 (15) | 0.29591 (10) | 0.49432 (5) | 0.0264 (3) |
| N3 | 0.89959 (15) | 0.34786 (10) | 0.54289 (5) | 0.0261 (3) |
| C1 | 0.57962 (19) | 0.68917 (12) | 0.41316 (7) | 0.0300 (4) |
| C2 | 0.54167 (18) | 0.76897 (12) | 0.45379 (7) | 0.0282 (4) |
| H2 | 0.5501 | 0.7548 | 0.4938 | 0.034* |
| C3 | 0.49214 (18) | 0.86797 (12) | 0.43633 (7) | 0.0287 (4) |
| H3 | 0.4660 | 0.9213 | 0.4644 | 0.034* |
| C4 | 0.48012 (18) | 0.89043 (12) | 0.37808 (7) | 0.0268 (3) |
| C5 | 0.5166 (2) | 0.81240 (13) | 0.33727 (7) | 0.0338 (4) |
| H5 | 0.5087 | 0.8271 | 0.2973 | 0.041* |
| C6 | 0.5647 (2) | 0.71295 (13) | 0.35493 (7) | 0.0386 (4) |
| H6 | 0.5881 | 0.6594 | 0.3267 | 0.046* |
| C7 | 0.6392 (2) | 0.58501 (13) | 0.43047 (7) | 0.0338 (4) |
| H7 | 0.6381 | 0.5278 | 0.4030 | 0.041* |
| C8 | 0.75333 (18) | 0.46864 (12) | 0.49509 (6) | 0.0259 (3) |
| C9 | 0.74727 (19) | 0.37078 (12) | 0.46124 (7) | 0.0290 (4) |
| C10 | 0.84009 (18) | 0.44934 (12) | 0.54448 (6) | 0.0260 (3) |
| C11 | 0.9202 (2) | 0.28398 (13) | 0.59429 (6) | 0.0331 (4) |
| H11A | 0.9757 | 0.3270 | 0.6245 | 0.050* |
| H11B | 0.9867 | 0.2214 | 0.5848 | 0.050* |
| H11C | 0.8119 | 0.2606 | 0.6081 | 0.050* |
| C12 | 0.8719 (2) | 0.52260 (13) | 0.59430 (7) | 0.0379 (4) |
| H12A | 0.8126 | 0.4967 | 0.6281 | 0.057* |
| H12B | 0.8340 | 0.5942 | 0.5851 | 0.057* |
| H12C | 0.9903 | 0.5253 | 0.6027 | 0.057* |
| C13 | 0.90702 (19) | 0.20424 (11) | 0.47004 (6) | 0.0253 (3) |
| C14 | 0.80958 (19) | 0.12678 (12) | 0.44266 (6) | 0.0284 (4) |
| H14 | 0.6932 | 0.1332 | 0.4419 | 0.034* |
| C15 | 0.8844 (2) | 0.03966 (12) | 0.41634 (7) | 0.0318 (4) |
| H15 | 0.8190 | −0.0136 | 0.3972 | 0.038* |
| C16 | 1.0533 (2) | 0.03015 (12) | 0.41783 (7) | 0.0319 (4) |
| H16 | 1.1039 | −0.0295 | 0.3997 | 0.038* |
| C17 | 1.1490 (2) | 0.10741 (12) | 0.44578 (7) | 0.0321 (4) |
| H17 | 1.2652 | 0.0999 | 0.4472 | 0.038* |
| C18 | 1.07674 (19) | 0.19580 (12) | 0.47182 (6) | 0.0277 (3) |
| H18 | 1.1426 | 0.2494 | 0.4905 | 0.033* |
| O3 | −0.07276 (14) | 0.38608 (8) | 0.19190 (5) | 0.0335 (3) |
| H3A | −0.1490 | 0.3819 | 0.1674 | 0.040* |
| O4 | 0.30090 (12) | 0.99610 (8) | 0.25745 (4) | 0.0276 (2) |
| N4 | −0.05124 (15) | 0.88981 (10) | 0.24542 (5) | 0.0269 (3) |
| N5 | 0.14278 (14) | 1.14370 (9) | 0.27759 (5) | 0.0238 (3) |

| | | | | |
|---|---|---|---|---|
| N6 | −0.02363 (14) | 1.16934 (9) | 0.27561 (5) | 0.0241 (3) |
| C19 | 0.00875 (18) | 0.70312 (11) | 0.24138 (6) | 0.0250 (3) |
| C20 | 0.12014 (19) | 0.62286 (12) | 0.25460 (6) | 0.0272 (3) |
| H20 | 0.2185 | 0.6414 | 0.2749 | 0.033* |
| C21 | 0.09102 (19) | 0.51762 (12) | 0.23895 (6) | 0.0274 (3) |
| H21 | 0.1682 | 0.4645 | 0.2487 | 0.033* |
| C22 | −0.05170 (19) | 0.48969 (11) | 0.20883 (6) | 0.0254 (3) |
| C23 | −0.16547 (19) | 0.56840 (12) | 0.19590 (6) | 0.0282 (4) |
| H23 | −0.2644 | 0.5496 | 0.1760 | 0.034* |
| C24 | −0.13492 (19) | 0.67315 (12) | 0.21193 (6) | 0.0278 (3) |
| H24 | −0.2132 | 0.7259 | 0.2027 | 0.033* |
| C25 | 0.04643 (19) | 0.81384 (12) | 0.25703 (6) | 0.0267 (3) |
| H25 | 0.1470 | 0.8302 | 0.2765 | 0.032* |
| C26 | −0.00185 (18) | 0.99402 (11) | 0.25961 (6) | 0.0234 (3) |
| C27 | 0.15973 (18) | 1.03778 (11) | 0.26465 (6) | 0.0231 (3) |
| C28 | −0.10963 (18) | 1.07782 (12) | 0.26662 (6) | 0.0250 (3) |
| C29 | −0.07842 (19) | 1.26236 (11) | 0.30874 (7) | 0.0297 (4) |
| H29A | −0.1949 | 1.2749 | 0.3005 | 0.045* |
| H29B | −0.0129 | 1.3250 | 0.2980 | 0.045* |
| H29C | −0.0642 | 1.2496 | 0.3500 | 0.045* |
| C30 | −0.29252 (18) | 1.07551 (13) | 0.26618 (8) | 0.0356 (4) |
| H30A | −0.3332 | 1.0926 | 0.3050 | 0.053* |
| H30B | −0.3320 | 1.0043 | 0.2542 | 0.053* |
| H30C | −0.3330 | 1.1282 | 0.2391 | 0.053* |
| C31 | 0.26579 (17) | 1.22415 (11) | 0.26718 (6) | 0.0225 (3) |
| C32 | 0.3001 (2) | 1.25156 (12) | 0.21120 (7) | 0.0318 (4) |
| H32 | 0.2406 | 1.2189 | 0.1801 | 0.038* |
| C33 | 0.4207 (2) | 1.32638 (14) | 0.20060 (8) | 0.0422 (4) |
| H33 | 0.4461 | 1.3451 | 0.1621 | 0.051* |
| C34 | 0.5045 (2) | 1.37399 (14) | 0.24610 (9) | 0.0456 (5) |
| H34 | 0.5874 | 1.4261 | 0.2388 | 0.055* |
| C35 | 0.4699 (2) | 1.34720 (14) | 0.30215 (9) | 0.0435 (5) |
| H35 | 0.5280 | 1.3811 | 0.3332 | 0.052* |
| C36 | 0.3501 (2) | 1.27070 (12) | 0.31313 (7) | 0.0321 (4) |
| H36 | 0.3264 | 1.2507 | 0.3515 | 0.039* |
| O5 | 0.43831 (13) | 1.00160 (8) | 0.15063 (5) | 0.0359 (3) |
| H5A | 0.3963 | 1.0005 | 0.1837 | 0.043* |
| O6 | 0.69633 (13) | 0.37408 (8) | 0.10793 (4) | 0.0302 (3) |
| N7 | 0.68224 (15) | 0.57227 (10) | 0.02772 (5) | 0.0278 (3) |
| N8 | 0.82733 (15) | 0.30098 (10) | 0.02799 (5) | 0.0255 (3) |
| N9 | 0.88764 (15) | 0.34003 (10) | −0.02407 (5) | 0.0277 (3) |
| C37 | 0.53272 (18) | 0.69523 (12) | 0.08808 (7) | 0.0277 (4) |
| C38 | 0.46494 (18) | 0.71054 (13) | 0.14274 (7) | 0.0297 (4) |
| H38 | 0.4420 | 0.6503 | 0.1657 | 0.036* |
| C39 | 0.43024 (18) | 0.81153 (12) | 0.16433 (7) | 0.0286 (4) |
| H39 | 0.3844 | 0.8201 | 0.2017 | 0.034* |
| C40 | 0.46269 (18) | 0.89989 (12) | 0.13116 (7) | 0.0279 (4) |
| C41 | 0.5231 (2) | 0.88589 (13) | 0.07559 (7) | 0.0347 (4) |

| | | | | |
|---|---|---|---|---|
| H41 | 0.5407 | 0.9461 | 0.0520 | 0.042* |
| C42 | 0.55773 (19) | 0.78528 (13) | 0.05449 (7) | 0.0326 (4) |
| H42 | 0.5993 | 0.7770 | 0.0164 | 0.039* |
| C43 | 0.58390 (18) | 0.58873 (12) | 0.07025 (7) | 0.0278 (4) |
| H43 | 0.5431 | 0.5292 | 0.0908 | 0.033* |
| C44 | 0.74040 (17) | 0.46900 (12) | 0.01784 (6) | 0.0242 (3) |
| C45 | 0.74410 (17) | 0.38185 (12) | 0.05700 (6) | 0.0244 (3) |
| C46 | 0.82686 (18) | 0.43964 (12) | −0.03047 (6) | 0.0253 (3) |
| C47 | 0.9212 (2) | 0.26398 (13) | −0.07089 (7) | 0.0368 (4) |
| H47A | 0.8227 | 0.2200 | −0.0786 | 0.055* |
| H47B | 1.0124 | 0.2181 | −0.0598 | 0.055* |
| H47C | 0.9512 | 0.3028 | −0.1057 | 0.055* |
| C48 | 0.8547 (2) | 0.50055 (13) | −0.08359 (7) | 0.0344 (4) |
| H48A | 0.9725 | 0.5017 | −0.0926 | 0.052* |
| H48B | 0.8165 | 0.5738 | −0.0777 | 0.052* |
| H48C | 0.7935 | 0.4666 | −0.1156 | 0.052* |
| C49 | 0.90208 (19) | 0.21186 (12) | 0.05461 (6) | 0.0275 (3) |
| C50 | 0.8033 (2) | 0.14313 (13) | 0.08618 (7) | 0.0346 (4) |
| H50 | 0.6883 | 0.1546 | 0.0893 | 0.041* |
| C51 | 0.8750 (2) | 0.05781 (14) | 0.11303 (7) | 0.0413 (4) |
| H51 | 0.8091 | 0.0112 | 0.1354 | 0.050* |
| C52 | 1.0412 (2) | 0.03980 (14) | 0.10757 (7) | 0.0413 (4) |
| H52 | 1.0892 | −0.0196 | 0.1257 | 0.050* |
| C53 | 1.1383 (2) | 0.10819 (14) | 0.07575 (7) | 0.0389 (4) |
| H53 | 1.2528 | 0.0952 | 0.0718 | 0.047* |
| C54 | 1.06954 (19) | 0.19547 (13) | 0.04960 (7) | 0.0328 (4) |
| H54 | 1.1365 | 0.2435 | 0.0285 | 0.039* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0364 (6) | 0.0284 (6) | 0.0344 (6) | 0.0082 (5) | 0.0011 (5) | 0.0044 (5) |
| O2 | 0.0553 (8) | 0.0376 (7) | 0.0314 (7) | 0.0158 (6) | −0.0136 (6) | −0.0071 (5) |
| N1 | 0.0293 (7) | 0.0247 (7) | 0.0308 (7) | 0.0052 (6) | 0.0009 (6) | 0.0013 (6) |
| N2 | 0.0314 (7) | 0.0248 (7) | 0.0229 (7) | 0.0054 (6) | −0.0012 (5) | −0.0035 (5) |
| N3 | 0.0327 (7) | 0.0242 (7) | 0.0214 (7) | 0.0034 (6) | −0.0009 (5) | −0.0002 (5) |
| C1 | 0.0314 (9) | 0.0266 (9) | 0.0321 (9) | 0.0043 (7) | −0.0041 (7) | −0.0005 (7) |
| C2 | 0.0259 (8) | 0.0320 (9) | 0.0268 (8) | 0.0008 (7) | −0.0002 (7) | 0.0014 (7) |
| C3 | 0.0284 (9) | 0.0276 (9) | 0.0302 (9) | 0.0053 (7) | 0.0026 (7) | −0.0025 (7) |
| C4 | 0.0203 (8) | 0.0260 (8) | 0.0341 (9) | 0.0025 (6) | −0.0003 (6) | 0.0029 (7) |
| C5 | 0.0409 (10) | 0.0354 (10) | 0.0255 (9) | 0.0088 (8) | −0.0027 (7) | 0.0028 (7) |
| C6 | 0.0522 (11) | 0.0357 (10) | 0.0280 (9) | 0.0123 (8) | −0.0034 (8) | −0.0060 (7) |
| C7 | 0.0406 (10) | 0.0264 (9) | 0.0345 (10) | 0.0079 (7) | −0.0024 (8) | −0.0034 (7) |
| C8 | 0.0278 (8) | 0.0226 (8) | 0.0275 (8) | 0.0040 (6) | 0.0034 (7) | −0.0005 (6) |
| C9 | 0.0303 (9) | 0.0287 (9) | 0.0281 (9) | 0.0069 (7) | −0.0005 (7) | 0.0011 (7) |
| C10 | 0.0296 (8) | 0.0220 (8) | 0.0265 (8) | 0.0019 (6) | 0.0047 (7) | 0.0013 (6) |
| C11 | 0.0453 (10) | 0.0304 (9) | 0.0240 (9) | 0.0043 (8) | −0.0014 (7) | 0.0040 (7) |
| C12 | 0.0550 (11) | 0.0292 (9) | 0.0296 (9) | 0.0049 (8) | −0.0027 (8) | −0.0024 (7) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C13 | 0.0335 (9) | 0.0196 (8) | 0.0230 (8) | 0.0044 (7) | 0.0031 (6) | 0.0014 (6) |
| C14 | 0.0302 (9) | 0.0272 (9) | 0.0279 (8) | −0.0004 (7) | 0.0040 (7) | 0.0012 (7) |
| C15 | 0.0417 (10) | 0.0230 (9) | 0.0306 (9) | −0.0041 (7) | 0.0045 (7) | −0.0010 (7) |
| C16 | 0.0424 (10) | 0.0234 (9) | 0.0300 (9) | 0.0055 (7) | 0.0064 (7) | −0.0003 (7) |
| C17 | 0.0326 (9) | 0.0291 (9) | 0.0348 (9) | 0.0080 (7) | 0.0024 (7) | 0.0020 (7) |
| C18 | 0.0307 (9) | 0.0244 (8) | 0.0279 (8) | 0.0024 (7) | −0.0014 (7) | −0.0001 (7) |
| O3 | 0.0453 (7) | 0.0227 (6) | 0.0324 (6) | 0.0005 (5) | −0.0041 (5) | −0.0029 (5) |
| O4 | 0.0228 (6) | 0.0250 (6) | 0.0351 (6) | 0.0051 (4) | 0.0010 (5) | −0.0013 (5) |
| N4 | 0.0295 (7) | 0.0217 (7) | 0.0295 (7) | −0.0011 (6) | 0.0007 (6) | −0.0015 (5) |
| N5 | 0.0186 (6) | 0.0204 (7) | 0.0323 (7) | 0.0015 (5) | 0.0031 (5) | −0.0004 (5) |
| N6 | 0.0193 (6) | 0.0205 (7) | 0.0326 (7) | 0.0030 (5) | 0.0035 (5) | −0.0005 (5) |
| C19 | 0.0300 (8) | 0.0215 (8) | 0.0237 (8) | −0.0008 (6) | 0.0032 (6) | 0.0003 (6) |
| C20 | 0.0286 (8) | 0.0281 (9) | 0.0249 (8) | −0.0008 (7) | −0.0001 (6) | 0.0007 (6) |
| C21 | 0.0332 (9) | 0.0236 (8) | 0.0256 (8) | 0.0051 (7) | 0.0017 (7) | 0.0023 (6) |
| C22 | 0.0356 (9) | 0.0185 (8) | 0.0222 (8) | −0.0012 (7) | 0.0049 (7) | 0.0005 (6) |
| C23 | 0.0282 (8) | 0.0281 (9) | 0.0281 (9) | −0.0022 (7) | −0.0012 (7) | 0.0006 (7) |
| C24 | 0.0289 (9) | 0.0236 (8) | 0.0311 (9) | 0.0024 (7) | 0.0005 (7) | 0.0032 (7) |
| C25 | 0.0288 (8) | 0.0252 (9) | 0.0260 (8) | −0.0020 (7) | 0.0005 (7) | 0.0002 (6) |
| C26 | 0.0252 (8) | 0.0210 (8) | 0.0241 (8) | 0.0004 (6) | 0.0008 (6) | 0.0007 (6) |
| C27 | 0.0261 (8) | 0.0221 (8) | 0.0213 (8) | 0.0022 (6) | 0.0021 (6) | 0.0016 (6) |
| C28 | 0.0252 (8) | 0.0250 (8) | 0.0249 (8) | −0.0006 (6) | 0.0015 (6) | 0.0022 (6) |
| C29 | 0.0302 (9) | 0.0221 (8) | 0.0371 (9) | 0.0067 (7) | 0.0084 (7) | 0.0003 (7) |
| C30 | 0.0233 (8) | 0.0358 (10) | 0.0476 (11) | −0.0005 (7) | 0.0032 (7) | −0.0003 (8) |
| C31 | 0.0185 (7) | 0.0188 (8) | 0.0304 (8) | 0.0024 (6) | 0.0020 (6) | −0.0004 (6) |
| C32 | 0.0338 (9) | 0.0308 (9) | 0.0309 (9) | 0.0009 (7) | 0.0038 (7) | −0.0024 (7) |
| C33 | 0.0394 (10) | 0.0380 (10) | 0.0495 (11) | 0.0006 (8) | 0.0162 (9) | 0.0093 (9) |
| C34 | 0.0252 (9) | 0.0309 (10) | 0.0810 (15) | −0.0023 (8) | 0.0056 (9) | 0.0093 (10) |
| C35 | 0.0346 (10) | 0.0322 (10) | 0.0633 (13) | −0.0007 (8) | −0.0179 (9) | −0.0042 (9) |
| C36 | 0.0352 (9) | 0.0287 (9) | 0.0323 (9) | 0.0033 (7) | −0.0067 (7) | −0.0003 (7) |
| O5 | 0.0404 (7) | 0.0316 (7) | 0.0357 (7) | 0.0082 (5) | 0.0042 (5) | −0.0045 (5) |
| O6 | 0.0321 (6) | 0.0348 (6) | 0.0237 (6) | 0.0006 (5) | 0.0063 (5) | −0.0028 (5) |
| N7 | 0.0279 (7) | 0.0304 (7) | 0.0250 (7) | 0.0035 (6) | −0.0018 (6) | −0.0042 (6) |
| N8 | 0.0264 (7) | 0.0277 (7) | 0.0223 (7) | 0.0023 (6) | 0.0043 (5) | −0.0024 (5) |
| N9 | 0.0285 (7) | 0.0320 (8) | 0.0225 (7) | 0.0011 (6) | 0.0050 (5) | −0.0053 (6) |
| C37 | 0.0209 (8) | 0.0321 (9) | 0.0299 (9) | 0.0032 (7) | −0.0005 (6) | −0.0026 (7) |
| C38 | 0.0276 (9) | 0.0322 (9) | 0.0293 (9) | 0.0029 (7) | 0.0016 (7) | 0.0004 (7) |
| C39 | 0.0257 (8) | 0.0353 (9) | 0.0247 (8) | 0.0049 (7) | 0.0013 (6) | −0.0028 (7) |
| C40 | 0.0228 (8) | 0.0286 (9) | 0.0323 (9) | 0.0068 (7) | −0.0020 (7) | −0.0044 (7) |
| C41 | 0.0377 (10) | 0.0328 (10) | 0.0338 (9) | 0.0046 (7) | 0.0047 (7) | 0.0026 (7) |
| C42 | 0.0311 (9) | 0.0364 (10) | 0.0303 (9) | 0.0035 (7) | 0.0063 (7) | −0.0006 (7) |
| C43 | 0.0229 (8) | 0.0298 (9) | 0.0306 (9) | 0.0013 (7) | −0.0005 (7) | −0.0026 (7) |
| C44 | 0.0219 (8) | 0.0266 (8) | 0.0238 (8) | −0.0006 (6) | 0.0000 (6) | −0.0039 (6) |
| C45 | 0.0178 (7) | 0.0290 (9) | 0.0262 (8) | −0.0009 (6) | −0.0002 (6) | −0.0058 (7) |
| C46 | 0.0212 (8) | 0.0294 (9) | 0.0251 (8) | −0.0011 (6) | −0.0022 (6) | −0.0039 (6) |
| C47 | 0.0420 (10) | 0.0414 (10) | 0.0268 (9) | 0.0097 (8) | 0.0018 (7) | −0.0101 (7) |
| C48 | 0.0369 (10) | 0.0386 (10) | 0.0276 (9) | −0.0012 (8) | 0.0040 (7) | −0.0008 (7) |
| C49 | 0.0303 (9) | 0.0244 (8) | 0.0276 (8) | 0.0025 (7) | −0.0006 (7) | −0.0054 (6) |
| C50 | 0.0330 (9) | 0.0317 (9) | 0.0388 (10) | −0.0027 (7) | 0.0010 (7) | −0.0014 (8) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C51 | 0.0495 (12) | 0.0335 (10) | 0.0408 (11) | −0.0041 (8) | −0.0002 (9) | 0.0038 (8) |
| C52 | 0.0519 (12) | 0.0343 (10) | 0.0378 (10) | 0.0088 (9) | −0.0068 (9) | −0.0013 (8) |
| C53 | 0.0340 (10) | 0.0434 (11) | 0.0394 (10) | 0.0113 (8) | −0.0032 (8) | −0.0013 (8) |
| C54 | 0.0296 (9) | 0.0351 (10) | 0.0335 (9) | 0.0013 (7) | 0.0019 (7) | −0.0040 (7) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C4 | 1.3630 (17) | C25—H25 | 0.9500 |
| O1—H1 | 0.8402 | C26—C28 | 1.3839 (19) |
| O2—C9 | 1.2369 (18) | C26—C27 | 1.419 (2) |
| N1—C7 | 1.2791 (19) | C28—C30 | 1.485 (2) |
| N1—C8 | 1.3942 (18) | C29—H29A | 0.9800 |
| N2—C9 | 1.3966 (18) | C29—H29B | 0.9800 |
| N2—N3 | 1.4069 (16) | C29—H29C | 0.9800 |
| N2—C13 | 1.4272 (18) | C30—H30A | 0.9800 |
| N3—C10 | 1.3669 (18) | C30—H30B | 0.9800 |
| N3—C11 | 1.4593 (18) | C30—H30C | 0.9800 |
| C1—C6 | 1.394 (2) | C31—C32 | 1.378 (2) |
| C1—C2 | 1.399 (2) | C31—C36 | 1.380 (2) |
| C1—C7 | 1.462 (2) | C32—C33 | 1.374 (2) |
| C2—C3 | 1.378 (2) | C32—H32 | 0.9500 |
| C2—H2 | 0.9500 | C33—C34 | 1.376 (3) |
| C3—C4 | 1.388 (2) | C33—H33 | 0.9500 |
| C3—H3 | 0.9500 | C34—C35 | 1.378 (3) |
| C4—C5 | 1.383 (2) | C34—H34 | 0.9500 |
| C5—C6 | 1.381 (2) | C35—C36 | 1.385 (2) |
| C5—H5 | 0.9500 | C35—H35 | 0.9500 |
| C6—H6 | 0.9500 | C36—H36 | 0.9500 |
| C7—H7 | 0.9500 | O5—C40 | 1.3617 (17) |
| C8—C10 | 1.370 (2) | O5—H5A | 0.8402 |
| C8—C9 | 1.444 (2) | O6—C45 | 1.2496 (17) |
| C10—C12 | 1.482 (2) | N7—C43 | 1.2863 (18) |
| C11—H11A | 0.9800 | N7—C44 | 1.3992 (18) |
| C11—H11B | 0.9800 | N8—C45 | 1.3900 (18) |
| C11—H11C | 0.9800 | N8—N9 | 1.3993 (16) |
| C12—H12A | 0.9800 | N8—C49 | 1.4286 (19) |
| C12—H12B | 0.9800 | N9—C46 | 1.3597 (18) |
| C12—H12C | 0.9800 | N9—C47 | 1.4597 (18) |
| C13—C18 | 1.384 (2) | C37—C38 | 1.395 (2) |
| C13—C14 | 1.388 (2) | C37—C42 | 1.397 (2) |
| C14—C15 | 1.389 (2) | C37—C43 | 1.457 (2) |
| C14—H14 | 0.9500 | C38—C39 | 1.385 (2) |
| C15—C16 | 1.378 (2) | C38—H38 | 0.9500 |
| C15—H15 | 0.9500 | C39—C40 | 1.384 (2) |
| C16—C17 | 1.384 (2) | C39—H39 | 0.9500 |
| C16—H16 | 0.9500 | C40—C41 | 1.389 (2) |
| C17—C18 | 1.390 (2) | C41—C42 | 1.377 (2) |
| C17—H17 | 0.9500 | C41—H41 | 0.9500 |

| | | | |
|---|---|---|---|
| C18—H18 | 0.9500 | C42—H42 | 0.9500 |
| O3—C22 | 1.3584 (17) | C43—H43 | 0.9500 |
| O3—H3A | 0.8402 | C44—C46 | 1.371 (2) |
| O4—C27 | 1.2763 (16) | C44—C45 | 1.435 (2) |
| N4—C25 | 1.2798 (18) | C46—C48 | 1.479 (2) |
| N4—C26 | 1.3949 (18) | C47—H47A | 0.9800 |
| N5—C27 | 1.3648 (18) | C47—H47B | 0.9800 |
| N5—N6 | 1.3941 (16) | C47—H47C | 0.9800 |
| N5—C31 | 1.4350 (18) | C48—H48A | 0.9800 |
| N6—C28 | 1.3482 (18) | C48—H48B | 0.9800 |
| N6—C29 | 1.4593 (17) | C48—H48C | 0.9800 |
| C19—C24 | 1.394 (2) | C49—C54 | 1.383 (2) |
| C19—C20 | 1.400 (2) | C49—C50 | 1.390 (2) |
| C19—C25 | 1.457 (2) | C50—C51 | 1.382 (2) |
| C20—C21 | 1.379 (2) | C50—H50 | 0.9500 |
| C20—H20 | 0.9500 | C51—C52 | 1.378 (2) |
| C21—C22 | 1.389 (2) | C51—H51 | 0.9500 |
| C21—H21 | 0.9500 | C52—C53 | 1.383 (2) |
| C22—C23 | 1.397 (2) | C52—H52 | 0.9500 |
| C23—C24 | 1.377 (2) | C53—C54 | 1.385 (2) |
| C23—H23 | 0.9500 | C53—H53 | 0.9500 |
| C24—H24 | 0.9500 | C54—H54 | 0.9500 |

| | | | |
|---|---|---|---|
| C4—O1—H1 | 109.3 | N5—C27—C26 | 106.62 (12) |
| C7—N1—C8 | 119.66 (13) | N6—C28—C26 | 109.55 (13) |
| C9—N2—N3 | 109.04 (12) | N6—C28—C30 | 121.58 (13) |
| C9—N2—C13 | 123.24 (12) | C26—C28—C30 | 128.85 (14) |
| N3—N2—C13 | 119.37 (12) | N6—C29—H29A | 109.5 |
| C10—N3—N2 | 106.96 (11) | N6—C29—H29B | 109.5 |
| C10—N3—C11 | 123.17 (12) | H29A—C29—H29B | 109.5 |
| N2—N3—C11 | 116.63 (12) | N6—C29—H29C | 109.5 |
| C6—C1—C2 | 117.94 (14) | H29A—C29—H29C | 109.5 |
| C6—C1—C7 | 120.32 (15) | H29B—C29—H29C | 109.5 |
| C2—C1—C7 | 121.70 (14) | C28—C30—H30A | 109.5 |
| C3—C2—C1 | 120.59 (14) | C28—C30—H30B | 109.5 |
| C3—C2—H2 | 119.7 | H30A—C30—H30B | 109.5 |
| C1—C2—H2 | 119.7 | C28—C30—H30C | 109.5 |
| C2—C3—C4 | 120.53 (14) | H30A—C30—H30C | 109.5 |
| C2—C3—H3 | 119.7 | H30B—C30—H30C | 109.5 |
| C4—C3—H3 | 119.7 | C32—C31—C36 | 121.27 (14) |
| O1—C4—C5 | 123.04 (14) | C32—C31—N5 | 119.12 (13) |
| O1—C4—C3 | 117.26 (13) | C36—C31—N5 | 119.59 (14) |
| C5—C4—C3 | 119.70 (14) | C33—C32—C31 | 119.66 (16) |
| C6—C5—C4 | 119.61 (15) | C33—C32—H32 | 120.2 |
| C6—C5—H5 | 120.2 | C31—C32—H32 | 120.2 |
| C4—C5—H5 | 120.2 | C32—C33—C34 | 119.56 (17) |
| C5—C6—C1 | 121.62 (15) | C32—C33—H33 | 120.2 |
| C5—C6—H6 | 119.2 | C34—C33—H33 | 120.2 |

| | | | |
|---|---|---|---|
| C1—C6—H6 | 119.2 | C33—C34—C35 | 120.93 (16) |
| N1—C7—C1 | 122.03 (15) | C33—C34—H34 | 119.5 |
| N1—C7—H7 | 119.0 | C35—C34—H34 | 119.5 |
| C1—C7—H7 | 119.0 | C34—C35—C36 | 119.84 (16) |
| C10—C8—N1 | 123.46 (14) | C34—C35—H35 | 120.1 |
| C10—C8—C9 | 107.64 (13) | C36—C35—H35 | 120.1 |
| N1—C8—C9 | 128.82 (13) | C31—C36—C35 | 118.73 (16) |
| O2—C9—N2 | 122.70 (14) | C31—C36—H36 | 120.6 |
| O2—C9—C8 | 131.93 (14) | C35—C36—H36 | 120.6 |
| N2—C9—C8 | 105.34 (13) | C40—O5—H5A | 109.6 |
| N3—C10—C8 | 110.19 (13) | C43—N7—C44 | 118.60 (13) |
| N3—C10—C12 | 121.57 (13) | C45—N8—N9 | 109.06 (12) |
| C8—C10—C12 | 128.24 (14) | C45—N8—C49 | 125.18 (12) |
| N3—C11—H11A | 109.5 | N9—N8—C49 | 120.77 (11) |
| N3—C11—H11B | 109.5 | C46—N9—N8 | 107.53 (11) |
| H11A—C11—H11B | 109.5 | C46—N9—C47 | 125.54 (13) |
| N3—C11—H11C | 109.5 | N8—N9—C47 | 118.42 (12) |
| H11A—C11—H11C | 109.5 | C38—C37—C42 | 117.58 (14) |
| H11B—C11—H11C | 109.5 | C38—C37—C43 | 119.10 (14) |
| C10—C12—H12A | 109.5 | C42—C37—C43 | 123.19 (14) |
| C10—C12—H12B | 109.5 | C39—C38—C37 | 121.56 (15) |
| H12A—C12—H12B | 109.5 | C39—C38—H38 | 119.2 |
| C10—C12—H12C | 109.5 | C37—C38—H38 | 119.2 |
| H12A—C12—H12C | 109.5 | C40—C39—C38 | 119.73 (14) |
| H12B—C12—H12C | 109.5 | C40—C39—H39 | 120.1 |
| C18—C13—C14 | 121.21 (14) | C38—C39—H39 | 120.1 |
| C18—C13—N2 | 120.28 (14) | O5—C40—C39 | 122.79 (14) |
| C14—C13—N2 | 118.45 (13) | O5—C40—C41 | 117.71 (14) |
| C13—C14—C15 | 119.14 (14) | C39—C40—C41 | 119.50 (14) |
| C13—C14—H14 | 120.4 | C42—C41—C40 | 120.38 (15) |
| C15—C14—H14 | 120.4 | C42—C41—H41 | 119.8 |
| C16—C15—C14 | 120.28 (15) | C40—C41—H41 | 119.8 |
| C16—C15—H15 | 119.9 | C41—C42—C37 | 121.12 (15) |
| C14—C15—H15 | 119.9 | C41—C42—H42 | 119.4 |
| C15—C16—C17 | 120.02 (15) | C37—C42—H42 | 119.4 |
| C15—C16—H16 | 120.0 | N7—C43—C37 | 122.46 (15) |
| C17—C16—H16 | 120.0 | N7—C43—H43 | 118.8 |
| C16—C17—C18 | 120.62 (15) | C37—C43—H43 | 118.8 |
| C16—C17—H17 | 119.7 | C46—C44—N7 | 123.26 (14) |
| C18—C17—H17 | 119.7 | C46—C44—C45 | 107.84 (13) |
| C13—C18—C17 | 118.72 (15) | N7—C44—C45 | 128.33 (13) |
| C13—C18—H18 | 120.6 | O6—C45—N8 | 122.91 (14) |
| C17—C18—H18 | 120.6 | O6—C45—C44 | 131.57 (14) |
| C22—O3—H3A | 109.0 | N8—C45—C44 | 105.39 (13) |
| C25—N4—C26 | 118.39 (13) | N9—C46—C44 | 109.75 (13) |
| C27—N5—N6 | 109.15 (11) | N9—C46—C48 | 121.71 (13) |
| C27—N5—C31 | 124.66 (12) | C44—C46—C48 | 128.54 (14) |
| N6—N5—C31 | 119.98 (11) | N9—C47—H47A | 109.5 |

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| C28—N6—N5 | 107.53 (11) | N9—C47—H47B | 109.5 |
| C28—N6—C29 | 126.16 (12) | H47A—C47—H47B | 109.5 |
| N5—N6—C29 | 118.27 (12) | N9—C47—H47C | 109.5 |
| C24—C19—C20 | 117.65 (14) | H47A—C47—H47C | 109.5 |
| C24—C19—C25 | 122.16 (14) | H47B—C47—H47C | 109.5 |
| C20—C19—C25 | 120.16 (14) | C46—C48—H48A | 109.5 |
| C21—C20—C19 | 121.82 (15) | C46—C48—H48B | 109.5 |
| C21—C20—H20 | 119.1 | H48A—C48—H48B | 109.5 |
| C19—C20—H20 | 119.1 | C46—C48—H48C | 109.5 |
| C20—C21—C22 | 119.61 (14) | H48A—C48—H48C | 109.5 |
| C20—C21—H21 | 120.2 | H48B—C48—H48C | 109.5 |
| C22—C21—H21 | 120.2 | C54—C49—C50 | 120.93 (15) |
| O3—C22—C21 | 117.97 (13) | C54—C49—N8 | 120.48 (14) |
| O3—C22—C23 | 122.58 (14) | C50—C49—N8 | 118.58 (14) |
| C21—C22—C23 | 119.44 (14) | C51—C50—C49 | 119.05 (16) |
| C24—C23—C22 | 120.30 (14) | C51—C50—H50 | 120.5 |
| C24—C23—H23 | 119.9 | C49—C50—H50 | 120.5 |
| C22—C23—H23 | 119.9 | C52—C51—C50 | 120.49 (17) |
| C23—C24—C19 | 121.16 (14) | C52—C51—H51 | 119.8 |
| C23—C24—H24 | 119.4 | C50—C51—H51 | 119.8 |
| C19—C24—H24 | 119.4 | C51—C52—C53 | 120.06 (16) |
| N4—C25—C19 | 122.26 (14) | C51—C52—H52 | 120.0 |
| N4—C25—H25 | 118.9 | C53—C52—H52 | 120.0 |
| C19—C25—H25 | 118.9 | C52—C53—C54 | 120.32 (16) |
| C28—C26—N4 | 123.84 (13) | C52—C53—H53 | 119.8 |
| C28—C26—C27 | 106.85 (13) | C54—C53—H53 | 119.8 |
| N4—C26—C27 | 129.10 (13) | C49—C54—C53 | 119.14 (16) |
| O4—C27—N5 | 121.76 (13) | C49—C54—H54 | 120.4 |
| O4—C27—C26 | 131.57 (14) | C53—C54—H54 | 120.4 |

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| C9—N2—N3—C10 | 9.42 (15) | N4—C26—C27—O4 | 0.6 (3) |
| C13—N2—N3—C10 | 158.68 (12) | C28—C26—C27—N5 | 3.35 (16) |
| C9—N2—N3—C11 | 151.86 (13) | N4—C26—C27—N5 | 178.09 (14) |
| C13—N2—N3—C11 | −58.87 (17) | N5—N6—C28—C26 | −3.20 (16) |
| C6—C1—C2—C3 | −0.4 (2) | C29—N6—C28—C26 | −150.99 (14) |
| C7—C1—C2—C3 | 177.34 (15) | N5—N6—C28—C30 | 175.78 (13) |
| C1—C2—C3—C4 | −0.4 (2) | C29—N6—C28—C30 | 28.0 (2) |
| C2—C3—C4—O1 | −178.79 (13) | N4—C26—C28—N6 | −175.15 (13) |
| C2—C3—C4—C5 | 0.6 (2) | C27—C26—C28—N6 | −0.06 (16) |
| O1—C4—C5—C6 | 179.40 (15) | N4—C26—C28—C30 | 6.0 (2) |
| C3—C4—C5—C6 | 0.0 (2) | C27—C26—C28—C30 | −178.94 (15) |
| C4—C5—C6—C1 | −0.9 (3) | C27—N5—C31—C32 | 69.87 (18) |
| C2—C1—C6—C5 | 1.1 (3) | N6—N5—C31—C32 | −79.37 (17) |
| C7—C1—C6—C5 | −176.72 (16) | C27—N5—C31—C36 | −108.60 (17) |
| C8—N1—C7—C1 | −177.79 (14) | N6—N5—C31—C36 | 102.17 (16) |
| C6—C1—C7—N1 | 162.55 (16) | C36—C31—C32—C33 | 0.2 (2) |
| C2—C1—C7—N1 | −15.1 (3) | N5—C31—C32—C33 | −178.24 (14) |
| C7—N1—C8—C10 | 167.52 (15) | C31—C32—C33—C34 | −0.8 (2) |

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| C7—N1—C8—C9 | −8.9 (2) | C32—C33—C34—C35 | 0.4 (3) |
| N3—N2—C9—O2 | 171.47 (15) | C33—C34—C35—C36 | 0.5 (3) |
| C13—N2—C9—O2 | 23.6 (2) | C32—C31—C36—C35 | 0.7 (2) |
| N3—N2—C9—C8 | −6.85 (16) | N5—C31—C36—C35 | 179.17 (13) |
| C13—N2—C9—C8 | −154.68 (13) | C34—C35—C36—C31 | −1.1 (2) |
| C10—C8—C9—O2 | −176.28 (18) | C45—N8—N9—C46 | −6.79 (15) |
| N1—C8—C9—O2 | 0.5 (3) | C49—N8—N9—C46 | −163.01 (13) |
| C10—C8—C9—N2 | 1.81 (17) | C45—N8—N9—C47 | −156.57 (13) |
| N1—C8—C9—N2 | 178.63 (14) | C49—N8—N9—C47 | 47.21 (18) |
| N2—N3—C10—C8 | −8.28 (16) | C42—C37—C38—C39 | −2.9 (2) |
| C11—N3—C10—C8 | −147.67 (14) | C43—C37—C38—C39 | 173.08 (14) |
| N2—N3—C10—C12 | 172.00 (14) | C37—C38—C39—C40 | 0.2 (2) |
| C11—N3—C10—C12 | 32.6 (2) | C38—C39—C40—O5 | −177.08 (14) |
| N1—C8—C10—N3 | −172.97 (13) | C38—C39—C40—C41 | 2.8 (2) |
| C9—C8—C10—N3 | 4.06 (17) | O5—C40—C41—C42 | 176.88 (14) |
| N1—C8—C10—C12 | 6.7 (3) | C39—C40—C41—C42 | −3.0 (2) |
| C9—C8—C10—C12 | −176.24 (15) | C40—C41—C42—C37 | 0.2 (2) |
| C9—N2—C13—C18 | 112.19 (17) | C38—C37—C42—C41 | 2.7 (2) |
| N3—N2—C13—C18 | −32.53 (19) | C43—C37—C42—C41 | −173.10 (15) |
| C9—N2—C13—C14 | −65.07 (19) | C44—N7—C43—C37 | 172.49 (13) |
| N3—N2—C13—C14 | 150.21 (13) | C38—C37—C43—N7 | −162.46 (14) |
| C18—C13—C14—C15 | −0.4 (2) | C42—C37—C43—N7 | 13.3 (2) |
| N2—C13—C14—C15 | 176.85 (13) | C43—N7—C44—C46 | 170.79 (14) |
| C13—C14—C15—C16 | 0.5 (2) | C43—N7—C44—C45 | −19.0 (2) |
| C14—C15—C16—C17 | 0.2 (2) | N9—N8—C45—O6 | −170.23 (13) |
| C15—C16—C17—C18 | −0.9 (2) | C49—N8—C45—O6 | −15.3 (2) |
| C14—C13—C18—C17 | −0.3 (2) | N9—N8—C45—C44 | 6.01 (15) |
| N2—C13—C18—C17 | −177.52 (13) | C49—N8—C45—C44 | 160.93 (13) |
| C16—C17—C18—C13 | 1.0 (2) | C46—C44—C45—O6 | 172.67 (15) |
| C27—N5—N6—C28 | 5.40 (15) | N7—C44—C45—O6 | 1.2 (3) |
| C31—N5—N6—C28 | 158.95 (13) | C46—C44—C45—N8 | −3.10 (16) |
| C27—N5—N6—C29 | 156.14 (12) | N7—C44—C45—N8 | −174.53 (14) |
| C31—N5—N6—C29 | −50.31 (17) | N8—N9—C46—C44 | 4.76 (16) |
| C24—C19—C20—C21 | 0.5 (2) | C47—N9—C46—C44 | 151.81 (14) |
| C25—C19—C20—C21 | −178.02 (13) | N8—N9—C46—C48 | −174.57 (13) |
| C19—C20—C21—C22 | 0.5 (2) | C47—N9—C46—C48 | −27.5 (2) |
| C20—C21—C22—O3 | 177.13 (13) | N7—C44—C46—N9 | 170.93 (13) |
| C20—C21—C22—C23 | −1.4 (2) | C45—C44—C46—N9 | −1.03 (16) |
| O3—C22—C23—C24 | −177.20 (13) | N7—C44—C46—C48 | −9.8 (2) |
| C21—C22—C23—C24 | 1.2 (2) | C45—C44—C46—C48 | 178.24 (14) |
| C22—C23—C24—C19 | −0.2 (2) | C45—N8—C49—C54 | −119.28 (16) |
| C20—C19—C24—C23 | −0.6 (2) | N9—N8—C49—C54 | 32.9 (2) |
| C25—C19—C24—C23 | 177.84 (14) | C45—N8—C49—C50 | 59.9 (2) |
| C26—N4—C25—C19 | −177.08 (13) | N9—N8—C49—C50 | −147.93 (14) |
| C24—C19—C25—N4 | 1.1 (2) | C54—C49—C50—C51 | 0.4 (2) |
| C20—C19—C25—N4 | 179.49 (14) | N8—C49—C50—C51 | −178.77 (14) |
| C25—N4—C26—C28 | −158.83 (14) | C49—C50—C51—C52 | −1.4 (3) |
| C25—N4—C26—C27 | 27.2 (2) | C50—C51—C52—C53 | 1.0 (3) |

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| N6—N5—C27—O4 | 172.41 (12) | C51—C52—C53—C54 | 0.5 (3) |
| C31—N5—C27—O4 | 20.4 (2) | C50—C49—C54—C53 | 1.1 (2) |
| N6—N5—C27—C26 | −5.35 (15) | N8—C49—C54—C53 | −179.82 (14) |
| C31—N5—C27—C26 | −157.38 (13) | C52—C53—C54—C49 | −1.5 (2) |
| C28—C26—C27—O4 | −174.11 (15) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
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| O1—H1···O4 | 0.84 | 1.86 | 2.6947 (14) | 170 |
| O3—H3*A*···O6$^{i}$ | 0.84 | 1.86 | 2.6993 (15) | 175 |
| O5—H5*A*···O4 | 0.84 | 1.88 | 2.7218 (15) | 179 |
| C7—H7···O2 | 0.95 | 2.30 | 3.009 (2) | 131 |
| C25—H25···O4 | 0.95 | 2.46 | 3.0612 (18) | 121 |
| C43—H43···O6 | 0.95 | 2.36 | 2.9989 (18) | 124 |
| C33—H33···O6$^{ii}$ | 0.95 | 2.42 | 3.168 (2) | 135 |
| C35—H35···O2$^{ii}$ | 0.95 | 2.31 | 3.122 (2) | 143 |

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