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# 2-Azido-*N*-(4-methylphenyl)acetamide

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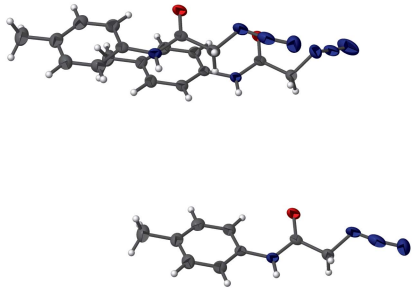
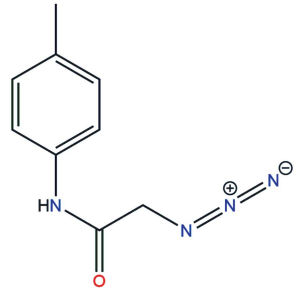
**Structural data**: full structural data are available from iucrdata.iucr.org

The asymmetric unit of the title compound, $C_9H_{10}N_4O$, comprises three independent molecules, two pairs of which differ significantly in the rotational orientation of the azido group and one pair having very similar conformations; the N—N—C—C torsion angles are −173.9 (2), −102.7 (2) and −173.6 (2)°. In the crystal, each independent molecule forms N—H···O hydrogen bonds with its glide-plane-related counterparts, forming zigzag chains extending along the *c*-axis direction.

**3D view**



**Chemical scheme**




## Structure description

*N*-arylacetamides are significant intermediates for the synthesis of medicinal, agrochemical and pharmaceutical compounds (Beccalli *et al.*, 2007; Valeur & Bradley, 2009; Allen & Williams, 2011; Missioui *et al.*, 2021, 2022*a*,*b*). Azides have found valuable applications in medicinal chemistry (Contin *et al.*, 2019), molecular biology (Ahmed & Abdallah, 2019) and attract increasing attention in the field of organic synthesis as intermediates for the preparation of heterocycles such as tetrazoles, triazolines, triazoles, *etc* (Chauhan *et al.*, 2019; Bakulev *et al.*, 2019; Abad *et al.*, 2020; Missioui, Lgaz *et al.*, 2022). Based on the aforementioned information and in continuation of our research efforts to synthesize more *N*-arylacetamides (Missioui *et al.*, 2020; Missioui, Guerrab, Nchioua *et al.*, 2022; Guerrab *et al.*, 2021), we report the synthesis and crystal structure of the title compound. The structure of the closely related compound 2-azido-*N*-(4-fluorophenyl)acetamide is reported by Missioui, Guerrab, Alsubari *et al.* (2022).

The asymmetric unit comprises three independent molecules with the azide moieties oriented in opposite directions between molecules containing O1 and O2 but with the same situation in the molecules containing O2 and O3 (Table 1). On the other hand, the

**Table 1**
Selected torsion angles (°).

| | | | |
|---|---|---|---|
| N3—N2—C9—C8 | −173.9 (2) | N11—N10—C27—C26 | −173.6 (2) |
| N7—N6—C18—C17 | −102.7 (2) | | |

**Table 2**
Hydrogen-bond geometry (Å, °).

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*A*···O1$^{i}$ | 0.86 (3) | 2.04 (3) | 2.867 (2) | 162 (2) |
| N5—H5*A*···O2$^{i}$ | 0.85 (3) | 2.01 (3) | 2.833 (2) | 163 (2) |
| C15—H15···O2 | 0.91 (3) | 2.34 (2) | 2.905 (3) | 120 (2) |
| C18—H18*B*···O2$^{i}$ | 0.99 (3) | 2.57 (3) | 3.353 (3) | 136 (2) |
| N9—H9*C*···O3$^{i}$ | 0.81 (3) | 2.05 (3) | 2.835 (2) | 163 (2) |
| C24—H24···O3 | 0.91 (3) | 2.27 (3) | 2.882 (3) | 125 (2) |

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

molecules containing O1 and O3 have very similar conformations. The rotational orientations of the phenyl groups with respect to the carboxamide moieties are partially determined by intramolecular C—H···O hydrogen bonds (Fig. 1 and Table 2).

In the crystal, each component of the asymmetric unit forms a chain with its counterparts related by the glide plane and extending along the *c*-axis direction through N1—H1*A*···O1 or N5—H5*A*···O2 or N9—H9*C*···O3 hydrogen bonds (Table 2). In the case of the molecule containing O2, the chain is reinforced by C18—H18*B*···O2 hydrogen bonds (Table 2 and Figs. 2 and 3). The chains pack through normal van der Waals contacts.

## Synthesis and crystallization

2-Chloro-*N*-(*p*-tolyl)acetamide (0.011 mol) and sodium azide (0.015 mol) were dissolved in a mixture of ethanol/water (70:30) then refluxed for 24 h at 80°C. After completion of the reaction (monitored by thin-layer chromatography, TLC), the 2-azido-*N*-(4-methylphenyl)acetamide precipitate was filtered and washed with cold water. A portion of the product was dissolved in hot ethanol, the solution was filtered and the filtrate was left undisturbed for 7 days to form colourless plate-like crystals.

Yield 73%, mp 360–362 K, FT–IR (ATR, $\nu$, cm$^{-1}$) 3254 $\nu$(N—H amide), 3073 $\nu$(C—H$_{arom}$), 2961 $\nu$(C—H,CH$_2$), 2109 $\nu$(N$_3$), 1027 $\nu$(N—C amide), 1660 $\nu$(C═O amide), 1175 $\upsilon$(C—N). $^1$H NMR (DMSO–$d_6$) $\delta$ p.p.m. 4.02 (2*H*, *s*, CH$_2$), 4.21 (3*H*, *s*, CH$_3$), 6.93–7.1 (4*H*, *m*, *J* = 1.3 Hz, H$_{arom}$), 10.05 (1*H*, *s*, NH), $^{13}$C NMR (DMSO–$d_6$) $\delta$ p.p.m. 51.18 (CH$_2$), 63.85 (CH$_3$), 131.47 (C$_{arom}$—N), 155.47 (C$_{arom}$–O), 113.90–120.86 (C$_{arom}$);

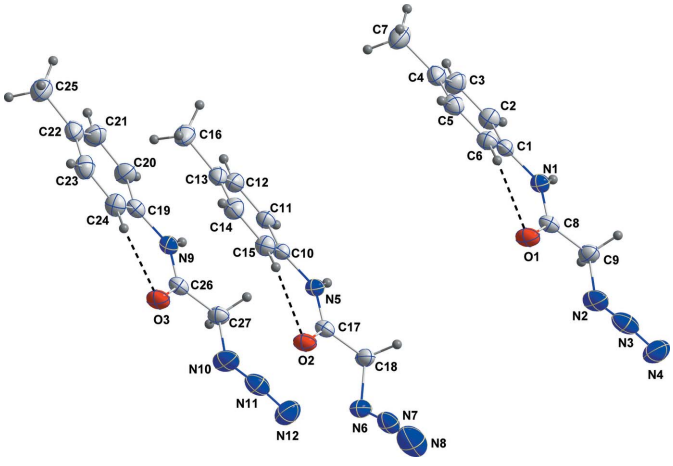



**Figure 1**
The asymmetric unit with the labelling scheme and 50% probability ellipsoids. The intramolecular C—H···O hydrogen bonds are shown as dashed lines.

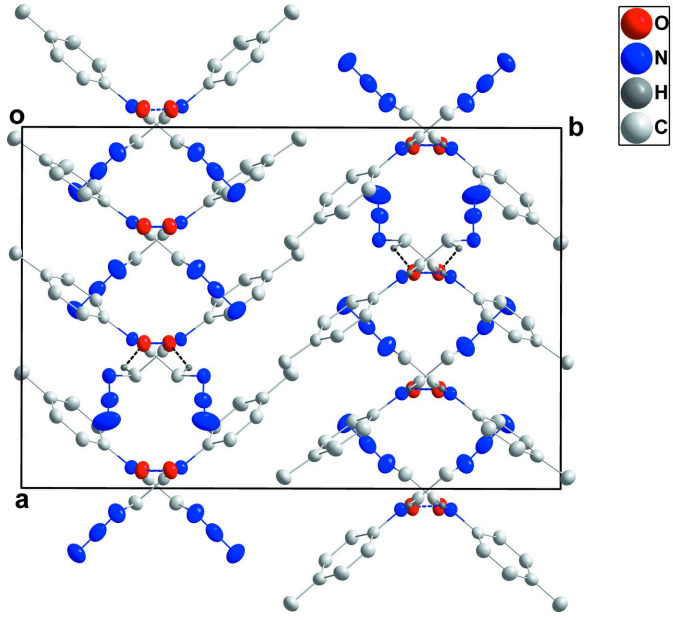



**Figure 2**
Packing viewed along the *c*-axis direction with N—H···O and C—H···O hydrogen bonds indicated, respectively, by blue and black dashed lines.

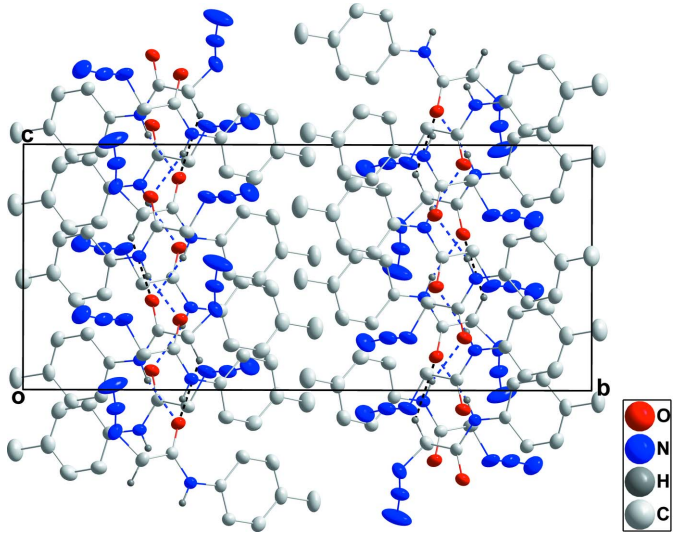



**Figure 3**
Packing viewed along the *a*-axis direction with N—H···O and C—H···O hydrogen bonds indicated, respectively, by blue and black dashed lines.

**Table 3**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $C_9H_{10}N_4O$ |
| $M_r$ | 190.21 |
| Crystal system, space group | Monoclinic, $P2_1/c$ |
| Temperature (K) | 150 |
| $a, b, c$ (Å) | 14.4362 (4), 21.3403 (6), 9.2949 (3) |
| $\beta$ (°) | 98.356 (1) |
| $V$ (Å$^3$) | 2833.11 (14) |
| $Z$ | 12 |
| Radiation type | Cu $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.77 |
| Crystal size (mm) | 0.22 × 0.16 × 0.08 |
| | |
| Data collection | |
| Diffractometer | Bruker D8 VENTURE PHOTON 100 CMOS |
| Absorption correction | Multi-scan (*SADABS*; Krause *et al.*, 2015) |
| $T_{min}$, $T_{max}$ | 0.86, 0.94 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 21650, 5486, 4161 |
| $R_{int}$ | 0.039 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.617 |
| | |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.059, 0.178, 1.05 |
| No. of reflections | 5486 |
| No. of parameters | 466 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.44, −0.39 |

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL 2018/1* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg & Putz, 2012) and *SHELXTL* (Sheldrick, 2008).

165.71 (C═O); HRMS (ESI MS) (*m*/*z*) calculated for $C_9H_{10}N_4O$ 190.21 found 190.1191.

## Refinement

Crystal data, data collection and structure and refinement details are summarized in Table 3.

## Acknowledgements

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# full crystallographic data

*IUCrData* (2022). **7**, x220621 [https://doi.org/10.1107/S2414314622006216]

## 2-Azido-*N*-(4-methylphenyl)acetamide

**Mohcine Missioui, Walid Guerrab, Abdulsalam Alsubari, Joel T. Mague and Youssef Ramli**

### 2-Azido-*N*-(4-methylphenyl)acetamide

*Crystal data*

$C_9H_{10}N_4O$
$M_r$ = 190.21
Monoclinic, $P2_1/c$
$a$ = 14.4362 (4) Å
$b$ = 21.3403 (6) Å
$c$ = 9.2949 (3) Å
$\beta$ = 98.356 (1)°
$V$ = 2833.11 (14) Å$^3$
$Z$ = 12
$F(000)$ = 1200
$D_x$ = 1.338 Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda$ = 1.54178 Å
Cell parameters from 9919 reflections
$\theta$ = 3.7–72.2°
$\mu$ = 0.77 mm$^{-1}$
$T$ = 150 K
Plate, colourless
0.22 × 0.16 × 0.08 mm

*Data collection*

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer
Radiation source: INCOATEC I$\mu$S micro–focus source
Mirror monochromator
Detector resolution: 10.4167 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Krause *et al.*, 2015)
$T_{min}$ = 0.86, $T_{max}$ = 0.94
21650 measured reflections
5486 independent reflections
4161 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.039
$\theta_{max}$ = 72.2°, $\theta_{min}$ = 3.7°
$h$ = −17→17
$k$ = −26→24
$l$ = −10→11

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.178
$S$ = 1.05
5486 reflections
466 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0988P)^2 + 1.2801P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.44 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.39 e Å$^{-3}$

*Special details*

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted R-factor wR and goodness of fit S are based on $F^2$, conventional R-factors R are based on F, with F set to zero for negative $F^2$. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on $F^2$ are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger. The methyl group hydrogen atoms were included as riding contributions in idealized positions since independent refinement yielded unsatisfactory geometries.
The H atoms were treated by a mixture of independent and constrained refinement.

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.94983 (12) | 0.27610 (7) | 0.28617 (15) | 0.0374 (4) |
| N1 | 0.94218 (12) | 0.29612 (8) | 0.04434 (18) | 0.0288 (4) |
| H1A | 0.9534 (17) | 0.2808 (11) | −0.037 (3) | 0.036 (6)* |
| N2 | 1.06934 (16) | 0.17717 (11) | 0.2860 (2) | 0.0456 (5) |
| N3 | 1.12261 (17) | 0.14178 (11) | 0.2867 (2) | 0.0462 (5) |
| N4 | 1.18070 (17) | 0.10247 (12) | 0.3057 (3) | 0.0584 (6) |
| C1 | 0.87815 (13) | 0.34695 (9) | 0.0273 (2) | 0.0271 (4) |
| C2 | 0.82911 (16) | 0.35703 (10) | −0.1109 (2) | 0.0342 (5) |
| H2 | 0.8403 (17) | 0.3316 (11) | −0.190 (3) | 0.036 (6)* |
| C3 | 0.76610 (16) | 0.40609 (11) | −0.1351 (3) | 0.0395 (5) |
| H3 | 0.7357 (18) | 0.4126 (12) | −0.230 (3) | 0.044 (7)* |
| C4 | 0.75000 (15) | 0.44650 (10) | −0.0236 (3) | 0.0361 (5) |
| C5 | 0.80085 (16) | 0.43586 (10) | 0.1134 (3) | 0.0358 (5) |
| H5 | 0.7923 (17) | 0.4632 (12) | 0.195 (3) | 0.040 (7)* |
| C6 | 0.86475 (16) | 0.38713 (10) | 0.1402 (2) | 0.0324 (5) |
| H6 | 0.8968 (18) | 0.3818 (12) | 0.240 (3) | 0.051 (8)* |
| C7 | 0.68108 (17) | 0.49939 (12) | −0.0490 (3) | 0.0491 (6) |
| H7A | 0.684547 | 0.518373 | −0.144064 | 0.074* |
| H7B | 0.695979 | 0.531009 | 0.027316 | 0.074* |
| H7C | 0.617706 | 0.483320 | −0.046823 | 0.074* |
| C8 | 0.97286 (14) | 0.26489 (9) | 0.1665 (2) | 0.0278 (4) |
| C9 | 1.04041 (16) | 0.21228 (10) | 0.1437 (2) | 0.0328 (5) |
| H9A | 1.094 (2) | 0.2312 (13) | 0.113 (3) | 0.051 (8)* |
| H9B | 1.0123 (18) | 0.1829 (13) | 0.072 (3) | 0.046 (7)* |
| O2 | 0.59911 (11) | 0.27335 (7) | 0.86318 (15) | 0.0342 (4) |
| N5 | 0.58708 (12) | 0.29502 (8) | 0.62103 (18) | 0.0267 (4) |
| H5A | 0.5974 (17) | 0.2809 (11) | 0.539 (3) | 0.036 (6)* |
| N6 | 0.68901 (13) | 0.16221 (9) | 0.8282 (2) | 0.0382 (4) |
| N7 | 0.75608 (14) | 0.16342 (10) | 0.9230 (2) | 0.0404 (5) |
| N8 | 0.81422 (19) | 0.15780 (17) | 1.0172 (3) | 0.0832 (10) |
| C10 | 0.52692 (14) | 0.34800 (9) | 0.6098 (2) | 0.0256 (4) |
| C11 | 0.47922 (14) | 0.36246 (9) | 0.4726 (2) | 0.0284 (4) |
| H11 | 0.4846 (15) | 0.3348 (10) | 0.395 (3) | 0.029 (6)* |
| C12 | 0.42233 (15) | 0.41482 (10) | 0.4533 (2) | 0.0333 (5) |
| H12 | 0.3878 (18) | 0.4254 (12) | 0.350 (3) | 0.044 (7)* |
| C13 | 0.41091 (14) | 0.45438 (9) | 0.5689 (2) | 0.0330 (5) |
| C14 | 0.45925 (16) | 0.43892 (10) | 0.7051 (3) | 0.0362 (5) |

| | | | | |
|---|---|---|---|---|
| H14 | 0.451 (2) | 0.4667 (14) | 0.782 (3) | 0.063 (9)* |
| C15 | 0.51679 (15) | 0.38677 (10) | 0.7267 (2) | 0.0320 (5) |
| H15 | 0.5482 (16) | 0.3763 (11) | 0.815 (3) | 0.036 (6)* |
| C16 | 0.35033 (16) | 0.51175 (11) | 0.5467 (3) | 0.0451 (6) |
| H16A | 0.371814 | 0.538444 | 0.472383 | 0.068* |
| H16B | 0.354393 | 0.534995 | 0.638346 | 0.068* |
| H16C | 0.285253 | 0.499254 | 0.514934 | 0.068* |
| C17 | 0.61908 (14) | 0.26242 (9) | 0.7411 (2) | 0.0262 (4) |
| C18 | 0.68726 (16) | 0.21025 (10) | 0.7153 (2) | 0.0318 (5) |
| H18A | 0.7514 (19) | 0.2291 (12) | 0.716 (3) | 0.043 (7)* |
| H18B | 0.666 (2) | 0.1898 (13) | 0.621 (3) | 0.056 (8)* |
| O3 | 0.27488 (11) | 0.27395 (7) | 0.57857 (15) | 0.0359 (4) |
| N9 | 0.26399 (12) | 0.29490 (8) | 0.33643 (18) | 0.0276 (4) |
| H9C | 0.2770 (17) | 0.2798 (11) | 0.262 (3) | 0.034 (6)* |
| N10 | 0.39415 (17) | 0.17693 (11) | 0.5742 (2) | 0.0497 (6) |
| N11 | 0.45011 (15) | 0.13998 (10) | 0.5754 (2) | 0.0418 (5) |
| N12 | 0.50716 (17) | 0.10135 (11) | 0.5955 (2) | 0.0545 (6) |
| C19 | 0.20449 (14) | 0.34791 (9) | 0.3227 (2) | 0.0272 (4) |
| C20 | 0.16642 (16) | 0.36623 (11) | 0.1823 (2) | 0.0370 (5) |
| H20 | 0.1734 (19) | 0.3414 (13) | 0.104 (3) | 0.049 (7)* |
| C21 | 0.11114 (17) | 0.41922 (12) | 0.1608 (3) | 0.0430 (5) |
| H21 | 0.0838 (19) | 0.4294 (13) | 0.063 (3) | 0.053 (8)* |
| C22 | 0.09127 (15) | 0.45572 (10) | 0.2763 (3) | 0.0381 (5) |
| C23 | 0.12924 (16) | 0.43601 (11) | 0.4155 (3) | 0.0395 (5) |
| H23 | 0.118 (2) | 0.4638 (13) | 0.502 (3) | 0.055 (8)* |
| C24 | 0.18568 (16) | 0.38350 (11) | 0.4397 (2) | 0.0351 (5) |
| H24 | 0.2128 (19) | 0.3704 (13) | 0.529 (3) | 0.050 (8)* |
| C25 | 0.03161 (18) | 0.51349 (12) | 0.2526 (3) | 0.0531 (7) |
| H25A | 0.044391 | 0.534972 | 0.164345 | 0.080* |
| H25B | 0.046129 | 0.541573 | 0.336221 | 0.080* |
| H25C | −0.034625 | 0.501632 | 0.241599 | 0.080* |
| C26 | 0.29622 (14) | 0.26290 (9) | 0.4575 (2) | 0.0270 (4) |
| C27 | 0.36337 (16) | 0.21023 (10) | 0.4325 (2) | 0.0316 (5) |
| H27A | 0.4158 (18) | 0.2284 (11) | 0.399 (3) | 0.037 (6)* |
| H27B | 0.333 (2) | 0.1813 (13) | 0.366 (3) | 0.053 (8)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0544 (10) | 0.0360 (8) | 0.0244 (7) | 0.0067 (7) | 0.0146 (7) | 0.0036 (6) |
| N1 | 0.0348 (9) | 0.0308 (9) | 0.0221 (8) | 0.0018 (7) | 0.0092 (7) | −0.0002 (6) |
| N2 | 0.0641 (14) | 0.0517 (12) | 0.0258 (10) | 0.0102 (11) | 0.0228 (9) | 0.0092 (8) |
| N3 | 0.0592 (14) | 0.0516 (13) | 0.0277 (10) | −0.0166 (12) | 0.0066 (9) | 0.0056 (9) |
| N4 | 0.0638 (15) | 0.0573 (14) | 0.0541 (14) | 0.0200 (12) | 0.0080 (11) | 0.0149 (11) |
| C1 | 0.0274 (9) | 0.0265 (10) | 0.0287 (10) | −0.0027 (8) | 0.0083 (8) | 0.0028 (7) |
| C2 | 0.0373 (11) | 0.0365 (11) | 0.0288 (11) | −0.0003 (9) | 0.0050 (9) | −0.0025 (9) |
| C3 | 0.0377 (12) | 0.0432 (13) | 0.0359 (13) | 0.0038 (10) | −0.0009 (10) | 0.0036 (9) |
| C4 | 0.0290 (10) | 0.0327 (11) | 0.0466 (13) | −0.0024 (9) | 0.0058 (9) | 0.0012 (9) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C5 | 0.0375 (11) | 0.0316 (11) | 0.0397 (12) | −0.0028 (9) | 0.0107 (9) | −0.0030 (9) |
| C6 | 0.0383 (11) | 0.0300 (10) | 0.0298 (11) | −0.0016 (9) | 0.0078 (9) | −0.0001 (8) |
| C7 | 0.0408 (13) | 0.0408 (13) | 0.0640 (17) | 0.0075 (11) | 0.0014 (12) | 0.0005 (11) |
| C8 | 0.0334 (10) | 0.0275 (9) | 0.0230 (10) | −0.0058 (8) | 0.0056 (8) | 0.0012 (7) |
| C9 | 0.0382 (12) | 0.0355 (11) | 0.0256 (10) | 0.0047 (9) | 0.0074 (9) | 0.0036 (8) |
| O2 | 0.0474 (9) | 0.0323 (8) | 0.0246 (7) | 0.0015 (6) | 0.0107 (6) | 0.0040 (6) |
| N5 | 0.0342 (9) | 0.0254 (8) | 0.0218 (8) | 0.0029 (7) | 0.0089 (7) | 0.0001 (6) |
| N6 | 0.0411 (10) | 0.0329 (10) | 0.0402 (10) | 0.0004 (8) | 0.0046 (9) | 0.0099 (8) |
| N7 | 0.0440 (11) | 0.0531 (12) | 0.0256 (10) | −0.0055 (9) | 0.0095 (9) | 0.0069 (8) |
| N8 | 0.0636 (16) | 0.136 (3) | 0.0447 (15) | −0.0191 (17) | −0.0087 (13) | 0.0369 (16) |
| C10 | 0.0290 (9) | 0.0224 (9) | 0.0272 (10) | −0.0019 (7) | 0.0097 (8) | 0.0010 (7) |
| C11 | 0.0316 (10) | 0.0279 (10) | 0.0261 (10) | −0.0016 (8) | 0.0057 (8) | −0.0003 (8) |
| C12 | 0.0299 (10) | 0.0327 (11) | 0.0372 (12) | −0.0009 (9) | 0.0039 (9) | 0.0051 (9) |
| C13 | 0.0290 (10) | 0.0246 (10) | 0.0468 (12) | −0.0013 (8) | 0.0105 (9) | 0.0008 (8) |
| C14 | 0.0387 (12) | 0.0312 (11) | 0.0405 (12) | 0.0005 (9) | 0.0118 (10) | −0.0071 (9) |
| C15 | 0.0370 (11) | 0.0303 (11) | 0.0298 (11) | 0.0004 (9) | 0.0085 (9) | −0.0029 (8) |
| C16 | 0.0371 (12) | 0.0292 (11) | 0.0695 (17) | 0.0033 (10) | 0.0090 (11) | −0.0001 (11) |
| C17 | 0.0314 (10) | 0.0237 (9) | 0.0248 (9) | −0.0043 (8) | 0.0085 (8) | 0.0003 (7) |
| C18 | 0.0357 (11) | 0.0294 (10) | 0.0320 (11) | 0.0052 (9) | 0.0100 (9) | 0.0060 (8) |
| O3 | 0.0493 (9) | 0.0376 (8) | 0.0231 (7) | 0.0027 (7) | 0.0130 (6) | 0.0032 (6) |
| N9 | 0.0322 (9) | 0.0312 (9) | 0.0208 (8) | −0.0001 (7) | 0.0083 (7) | −0.0015 (7) |
| N10 | 0.0677 (14) | 0.0583 (13) | 0.0273 (10) | 0.0166 (12) | 0.0217 (10) | 0.0144 (9) |
| N11 | 0.0510 (12) | 0.0495 (12) | 0.0256 (9) | −0.0109 (11) | 0.0077 (8) | 0.0030 (8) |
| N12 | 0.0575 (14) | 0.0592 (14) | 0.0462 (13) | 0.0170 (12) | 0.0062 (10) | 0.0083 (10) |
| C19 | 0.0267 (9) | 0.0277 (10) | 0.0280 (10) | −0.0053 (8) | 0.0065 (8) | 0.0006 (7) |
| C20 | 0.0426 (12) | 0.0409 (12) | 0.0285 (11) | 0.0046 (10) | 0.0081 (9) | 0.0007 (9) |
| C21 | 0.0431 (13) | 0.0475 (14) | 0.0381 (13) | 0.0075 (11) | 0.0045 (10) | 0.0076 (10) |
| C22 | 0.0284 (10) | 0.0334 (11) | 0.0531 (14) | −0.0032 (9) | 0.0080 (10) | 0.0006 (10) |
| C23 | 0.0355 (11) | 0.0385 (12) | 0.0456 (13) | −0.0005 (10) | 0.0092 (10) | −0.0086 (10) |
| C24 | 0.0375 (11) | 0.0390 (12) | 0.0292 (11) | 0.0012 (9) | 0.0060 (9) | −0.0048 (9) |
| C25 | 0.0414 (13) | 0.0407 (14) | 0.077 (2) | 0.0072 (11) | 0.0082 (13) | 0.0038 (12) |
| C26 | 0.0305 (10) | 0.0269 (9) | 0.0245 (9) | −0.0066 (8) | 0.0066 (8) | 0.0006 (7) |
| C27 | 0.0383 (11) | 0.0311 (11) | 0.0260 (10) | 0.0001 (9) | 0.0071 (9) | 0.0022 (8) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C8 | 1.230 (2) | C12—H12 | 1.04 (3) |
| N1—C8 | 1.335 (3) | C13—C14 | 1.393 (3) |
| N1—C1 | 1.419 (3) | C13—C16 | 1.501 (3) |
| N1—H1A | 0.86 (3) | C14—C15 | 1.386 (3) |
| N2—N3 | 1.077 (3) | C14—H14 | 0.95 (3) |
| N2—C9 | 1.525 (3) | C15—H15 | 0.91 (3) |
| N3—N4 | 1.181 (3) | C16—H16A | 0.9800 |
| C1—C6 | 1.390 (3) | C16—H16B | 0.9800 |
| C1—C2 | 1.390 (3) | C16—H16C | 0.9800 |
| C2—C3 | 1.384 (3) | C17—C18 | 1.528 (3) |
| C2—H2 | 0.95 (2) | C18—H18A | 1.01 (3) |
| C3—C4 | 1.394 (3) | C18—H18B | 0.99 (3) |

| | | | |
|---|---|---|---|
| C3—H3 | 0.94 (3) | O3—C26 | 1.232 (2) |
| C4—C5 | 1.393 (3) | N9—C26 | 1.341 (3) |
| C4—C7 | 1.500 (3) | N9—C19 | 1.415 (3) |
| C5—C6 | 1.388 (3) | N9—H9C | 0.81 (3) |
| C5—H5 | 0.98 (3) | N10—N11 | 1.128 (3) |
| C6—H6 | 0.98 (3) | N10—C27 | 1.505 (3) |
| C7—H7A | 0.9800 | N11—N12 | 1.161 (3) |
| C7—H7B | 0.9800 | C19—C24 | 1.386 (3) |
| C7—H7C | 0.9800 | C19—C20 | 1.396 (3) |
| C8—C9 | 1.522 (3) | C20—C21 | 1.382 (3) |
| C9—H9A | 0.96 (3) | C20—H20 | 0.92 (3) |
| C9—H9B | 0.96 (3) | C21—C22 | 1.390 (3) |
| O2—C17 | 1.233 (2) | C21—H21 | 0.96 (3) |
| N5—C17 | 1.339 (3) | C22—C23 | 1.394 (4) |
| N5—C10 | 1.420 (2) | C22—C25 | 1.502 (3) |
| N5—H5A | 0.85 (3) | C23—C24 | 1.384 (3) |
| N6—N7 | 1.211 (3) | C23—H23 | 1.03 (3) |
| N6—C18 | 1.465 (3) | C24—H24 | 0.91 (3) |
| N7—N8 | 1.128 (3) | C25—H25A | 0.9800 |
| C10—C15 | 1.390 (3) | C25—H25B | 0.9800 |
| C10—C11 | 1.393 (3) | C25—H25C | 0.9800 |
| C11—C12 | 1.383 (3) | C26—C27 | 1.524 (3) |
| C11—H11 | 0.95 (2) | C27—H27A | 0.94 (3) |
| C12—C13 | 1.395 (3) | C27—H27B | 0.93 (3) |

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|---|---|---|---|
| C8—N1—C1 | 127.34 (17) | C13—C14—H14 | 115.7 (19) |
| C8—N1—H1A | 118.5 (16) | C14—C15—C10 | 119.6 (2) |
| C1—N1—H1A | 113.2 (16) | C14—C15—H15 | 122.6 (16) |
| N3—N2—C9 | 117.05 (19) | C10—C15—H15 | 117.7 (16) |
| N2—N3—N4 | 171.7 (3) | C13—C16—H16A | 109.5 |
| C6—C1—C2 | 119.58 (19) | C13—C16—H16B | 109.5 |
| C6—C1—N1 | 123.19 (18) | H16A—C16—H16B | 109.5 |
| C2—C1—N1 | 117.21 (18) | C13—C16—H16C | 109.5 |
| C3—C2—C1 | 120.1 (2) | H16A—C16—H16C | 109.5 |
| C3—C2—H2 | 119.1 (15) | H16B—C16—H16C | 109.5 |
| C1—C2—H2 | 120.7 (15) | O2—C17—N5 | 124.77 (18) |
| C2—C3—C4 | 121.6 (2) | O2—C17—C18 | 121.65 (18) |
| C2—C3—H3 | 118.1 (16) | N5—C17—C18 | 113.54 (16) |
| C4—C3—H3 | 120.3 (16) | N6—C18—C17 | 109.98 (17) |
| C5—C4—C3 | 117.2 (2) | N6—C18—H18A | 110.7 (15) |
| C5—C4—C7 | 121.0 (2) | C17—C18—H18A | 108.6 (15) |
| C3—C4—C7 | 121.8 (2) | N6—C18—H18B | 107.3 (17) |
| C6—C5—C4 | 122.3 (2) | C17—C18—H18B | 109.6 (16) |
| C6—C5—H5 | 117.6 (15) | H18A—C18—H18B | 111 (2) |
| C4—C5—H5 | 120.1 (15) | C26—N9—C19 | 127.96 (17) |
| C5—C6—C1 | 119.3 (2) | C26—N9—H9C | 114.8 (17) |
| C5—C6—H6 | 117.8 (16) | C19—N9—H9C | 117.0 (17) |
| C1—C6—H6 | 122.9 (16) | N11—N10—C27 | 117.32 (19) |

| | | | |
|---|---|---|---|
| C4—C7—H7A | 109.5 | N10—N11—N12 | 171.3 (2) |
| C4—C7—H7B | 109.5 | C24—C19—C20 | 119.0 (2) |
| H7A—C7—H7B | 109.5 | C24—C19—N9 | 123.58 (19) |
| C4—C7—H7C | 109.5 | C20—C19—N9 | 117.38 (18) |
| H7A—C7—H7C | 109.5 | C21—C20—C19 | 120.4 (2) |
| H7B—C7—H7C | 109.5 | C21—C20—H20 | 118.9 (17) |
| O1—C8—N1 | 124.90 (19) | C19—C20—H20 | 120.5 (17) |
| O1—C8—C9 | 122.14 (18) | C20—C21—C22 | 121.8 (2) |
| N1—C8—C9 | 112.96 (17) | C20—C21—H21 | 118.0 (17) |
| C8—C9—N2 | 110.01 (17) | C22—C21—H21 | 120.2 (17) |
| C8—C9—H9A | 107.2 (17) | C21—C22—C23 | 116.8 (2) |
| N2—C9—H9A | 109.4 (16) | C21—C22—C25 | 121.7 (2) |
| C8—C9—H9B | 111.3 (16) | C23—C22—C25 | 121.6 (2) |
| N2—C9—H9B | 108.4 (16) | C24—C23—C22 | 122.5 (2) |
| H9A—C9—H9B | 110 (2) | C24—C23—H23 | 119.6 (16) |
| C17—N5—C10 | 127.42 (17) | C22—C23—H23 | 117.8 (16) |
| C17—N5—H5A | 118.5 (16) | C23—C24—C19 | 119.6 (2) |
| C10—N5—H5A | 113.6 (16) | C23—C24—H24 | 124.4 (18) |
| N7—N6—C18 | 115.80 (19) | C19—C24—H24 | 116.0 (18) |
| N8—N7—N6 | 171.4 (3) | C22—C25—H25A | 109.5 |
| C15—C10—C11 | 119.29 (19) | C22—C25—H25B | 109.5 |
| C15—C10—N5 | 123.33 (18) | H25A—C25—H25B | 109.5 |
| C11—C10—N5 | 117.33 (17) | C22—C25—H25C | 109.5 |
| C12—C11—C10 | 120.22 (19) | H25A—C25—H25C | 109.5 |
| C12—C11—H11 | 121.2 (14) | H25B—C25—H25C | 109.5 |
| C10—C11—H11 | 118.5 (14) | O3—C26—N9 | 124.54 (19) |
| C11—C12—C13 | 121.6 (2) | O3—C26—C27 | 121.98 (18) |
| C11—C12—H12 | 119.5 (14) | N9—C26—C27 | 113.48 (17) |
| C13—C12—H12 | 118.9 (14) | N10—C27—C26 | 109.16 (17) |
| C14—C13—C12 | 117.18 (19) | N10—C27—H27A | 109.5 (15) |
| C14—C13—C16 | 121.5 (2) | C26—C27—H27A | 107.8 (15) |
| C12—C13—C16 | 121.3 (2) | N10—C27—H27B | 108.3 (18) |
| C15—C14—C13 | 122.1 (2) | C26—C27—H27B | 109.8 (17) |
| C15—C14—H14 | 122.1 (19) | H27A—C27—H27B | 112 (2) |

| | | | |
|---|---|---|---|
| C8—N1—C1—C6 | 25.6 (3) | C13—C14—C15—C10 | −0.4 (3) |
| C8—N1—C1—C2 | −156.2 (2) | C11—C10—C15—C14 | 0.3 (3) |
| C6—C1—C2—C3 | −1.2 (3) | N5—C10—C15—C14 | 177.55 (19) |
| N1—C1—C2—C3 | −179.52 (19) | C10—N5—C17—O2 | 1.3 (3) |
| C1—C2—C3—C4 | 0.1 (3) | C10—N5—C17—C18 | −176.63 (18) |
| C2—C3—C4—C5 | 0.7 (3) | N7—N6—C18—C17 | −102.7 (2) |
| C2—C3—C4—C7 | −179.4 (2) | O2—C17—C18—N6 | 25.1 (3) |
| C3—C4—C5—C6 | −0.4 (3) | N5—C17—C18—N6 | −156.85 (18) |
| C7—C4—C5—C6 | 179.7 (2) | C26—N9—C19—C24 | 14.4 (3) |
| C4—C5—C6—C1 | −0.7 (3) | C26—N9—C19—C20 | −168.4 (2) |
| C2—C1—C6—C5 | 1.5 (3) | C24—C19—C20—C21 | 0.2 (3) |
| N1—C1—C6—C5 | 179.71 (19) | N9—C19—C20—C21 | −177.1 (2) |
| C1—N1—C8—O1 | −0.4 (3) | C19—C20—C21—C22 | −0.2 (4) |

| | | | |
|---|---|---|---|
| C1—N1—C8—C9 | 179.19 (18) | C20—C21—C22—C23 | −0.5 (4) |
| O1—C8—C9—N2 | 1.9 (3) | C20—C21—C22—C25 | 179.6 (2) |
| N1—C8—C9—N2 | −177.75 (18) | C21—C22—C23—C24 | 1.3 (3) |
| N3—N2—C9—C8 | −173.9 (2) | C25—C22—C23—C24 | −178.9 (2) |
| C17—N5—C10—C15 | 22.5 (3) | C22—C23—C24—C19 | −1.3 (4) |
| C17—N5—C10—C11 | −160.22 (19) | C20—C19—C24—C23 | 0.5 (3) |
| C15—C10—C11—C12 | −0.1 (3) | N9—C19—C24—C23 | 177.64 (19) |
| N5—C10—C11—C12 | −177.53 (18) | C19—N9—C26—O3 | 3.1 (3) |
| C10—C11—C12—C13 | 0.0 (3) | C19—N9—C26—C27 | −176.86 (18) |
| C11—C12—C13—C14 | −0.1 (3) | N11—N10—C27—C26 | −173.6 (2) |
| C11—C12—C13—C16 | 178.9 (2) | O3—C26—C27—N10 | 0.8 (3) |
| C12—C13—C14—C15 | 0.2 (3) | N9—C26—C27—N10 | −179.23 (18) |
| C16—C13—C14—C15 | −178.8 (2) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*A*···O1$^{i}$ | 0.86 (3) | 2.04 (3) | 2.867 (2) | 162 (2) |
| N5—H5*A*···O2$^{i}$ | 0.85 (3) | 2.01 (3) | 2.833 (2) | 163 (2) |
| C15—H15···O2 | 0.91 (3) | 2.34 (2) | 2.905 (3) | 120 (2) |
| C18—H18*B*···O2$^{i}$ | 0.99 (3) | 2.57 (3) | 3.353 (3) | 136 (2) |
| N9—H9*C*···O3$^{i}$ | 0.81 (3) | 2.05 (3) | 2.835 (2) | 163 (2) |
| C24—H24···O3 | 0.91 (3) | 2.27 (3) | 2.882 (3) | 125 (2) |

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