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# 3-Amino-5,5-diphenylimidazolidine-2,4-dione

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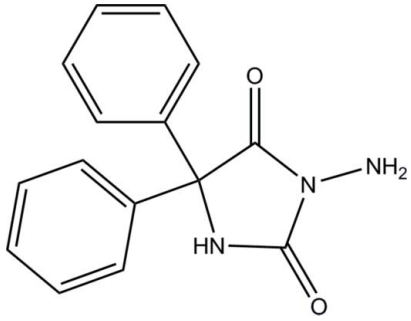
Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.054; $wR$ factor = 0.111; data-to-parameter ratio = 12.6.

The title compound, $C_{15}H_{13}N_3O_2$, crystallizes with two independent molecules in the asymmetric unit, which differ considerably in the dihedral angles made between the phenyl groups and the five-membered rings [47.19 (8) and 61.16 (9)° in one molecule and 55.04 (10) and 55.00 (8)° in the other]. In the crystal, N—H···O hydrogen bonds generate columnar units having approximate fourfold rotational symmetry about axes parallel to $b$.

## Related literature

For the biological properties of hydantoins, see: El-Deeb *et al.* (2010); Rajic *et al.* (2006); Carmi *et al.* (2006); Sergent *et al.* (2008). For the preparation of the title compound, see: Kiec-Kononowicz *et al.* (1984). For related crystal structures, see: Delgado *et al.* (2007); Roszak & Weaver (1998); Kashif *et al.* (2008); Coquerel *et al.* (1993); SethuSankar *et al.* (2002); Eknoian *et al.* (1999); Ciechanowicz-Rutkowska *et al.* (1994).




## Experimental

### Crystal data

$C_{15}H_{13}N_3O_2$
$M_r$ = 267.28
Monoclinic, $P2_1/n$
$a$ = 20.1565 (7) Å
$b$ = 6.1651 (2) Å
$c$ = 20.3250 (7) Å
$\beta$ = 97.781 (2)°
$V$ = 2502.47 (15) Å$^3$
$Z$ = 8
Cu $K\alpha$ radiation
$\mu$ = 0.79 mm$^{-1}$
$T$ = 100 K
0.22 × 0.07 × 0.05 mm

### Data collection

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2012)
$T_{min}$ = 0.84, $T_{max}$ = 0.96
32772 measured reflections
4560 independent reflections
3185 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.105

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.111
$S$ = 1.08
4560 reflections
361 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.24 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···O1$^{i}$ | 0.91 | 1.92 | 2.828 (3) | 177 |
| N3—H3A···O3 | 0.91 | 2.11 | 2.957 (3) | 154 |
| N4—H4···O4$^{ii}$ | 0.91 | 1.91 | 2.820 (3) | 176 |
| N6—H6A···O3$^{i}$ | 0.91 | 2.58 | 3.320 (3) | 139 |
| N6—H6B···O2$^{iii}$ | 0.91 | 2.46 | 3.070 (3) | 124 |
| N6—H6B···N3$^{iii}$ | 0.91 | 2.52 | 3.363 (3) | 154 |

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

Data collection: *APEX2* (Bruker, 2012); cell refinement: *SAINT* (Bruker, 2012); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2012); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

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Supporting information for this paper is available from the IUCr electronic archives (Reference: SJ5389).

## References

Brandenburg, K. & Putz, H. (2012). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Bruker (2012). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Carmi, C., Cavazzoni, A., Zuliani, V., Lodola, A., Bordi, F., Plazzi, P. V., Alfieri, R. R., Petronini, P. G. & Mor, M. (2006). *Bioorg. Med. Chem. Lett.* **16**, 4021–4025.
Ciechanowicz-Rutkowska, M., Kieć-Kononowicz, K., Howard, S. T., Lieberman, H. & Hursthouse, M. B. (1994). *Acta Cryst.* B**50**, 86–96.
Coquerel, G., Petit, M. N. & Robert, F. (1993). *Acta Cryst.* C**49**, 824–825.

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Delgado, G. E., Mora, A. J., Uzcátegui, J., Bahsas, A. & Briceño, A. (2007). *Acta Cryst.* C**63**, o448–o450.
Eknoian, M. W., Webb, T. R., Worley, S. D., Braswell, A. & Hadley, J. (1999). *Acta Cryst.* C**55**, 405–407.
El-Deeb, I. M., Bayoumi, S. M., El-Sherbeny, M. A. & Abdel-Aziz, A. A.-M. (2010). *Eur. J. Med. Chem.* **45**, 2516–2530.
Kashif, M. K., Hussain, A., Khawar Rauf, M., Ebihara, M. & Hameed, S. (2008). *Acta Cryst.* E**64**, o444.
Kiec-Kononowicz, K., Zejc, A. & Byrtus, H. (1984). *Pol. J. Chem.* **58**, 585–591.
Rajic, Z., Zorc, B., Raic-Malic, S., Ester, K., Kralj, M., Pavelic, K., Balzarini, J., De Clercq, E. & Mintas, M. (2006). *Molecules*, **11**, 837–848.
Roszak, A. W. & Weaver, D. F. (1998). *Acta Cryst.* C**54**, 1168–1170.
Sergent, D., Wang, Q., Sasaki, N. A. & Ouazzani, J. (2008). *Bioorg. Med. Chem. Lett.* **18**, 4332–4335.
SethuSankar, K., Thennarasu, S., Velmurugan, D. & Kim, M. J. (2002). *Acta Cryst.* C**58**, o715–o717.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

# supporting information

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## 3-Amino-5,5-diphenylimidazolidine-2,4-dione

**Joel T. Mague, Alaa A.-M. Abdel-Aziz and Adel S. El-Azab**

### S1. Comment

Hydantoins are an important class of compounds which have long attracted attention, owing to their remarkable biological and pharmacological properties. These include antitumor and antiviral activity, insulinotropic properties and use as EGFR inhibitors. (El-Deeb *et al.*, 2010; Rajic *et al.*, 2006; Carmi *et al.*, 2006; Sergent *et al.*, 2008). Of several structure determinations of hydantoins (Delgado *et al.*, 2007; Kashif *et al.*, 2008; Coquerel *et al.*, 1993; SethuSankar *et al.*, 2002; Ciechanowicz-Rutkowska *et al.*, 1994; Roszak & Weaver, 1998; Eknoian *et al.*, 1999), those in the last two reports bear the greatest similarity to the title compound. The title compound crystallizes with two independent molecules (A and B) in the asymmetric unit. The 5-membered ring in A is planar to within 0.032 Å while that in B is planar to within 0.016 Å. Molecules A and B differ most notably in the dihedral angles which the pendant phenyl rings make with the 5-membered ring. For molecule A the angles for C4/C5/C6/C7/C8/C9 and for C10/C11/C12/C13/C14/C15 are, respectively, 47.19 (8) and 61.16 (9)°. In molecule B the angles for C19/C20/C21/C22/C23/C24 and for C25/C26/C27/C28/C29/C30 are identical at 55.04 (10) and 55.00 (8)°, respectively. Both the imino and amino groups participate in intermolecular N—H···O hydrogen bonding which forms columns having approximate 4-fold rotational symmetry about axes parallel to *b* (Table 1 and Fig. 2).

### S2. Experimental

The title compound, 3-amino-5,5-diphenylimidazolidine-2,4-dione, was successfully obtained by the reaction of 5,5-diphenylhydantoin and hydrazine hydrate following the published route (Kiec-Kononowicz *et al.*, 1984).

### S3. Refinement

H-atoms attached to carbon were placed in calculated positions (C—H = 0.95 Å) while those attached to nitrogen were placed in locations derived from a difference map and their coordinates adjusted to give N—H = 0.91 Å. All were included as riding contributions with isotropic displacement parameters 1.2 times those of the attached atoms.

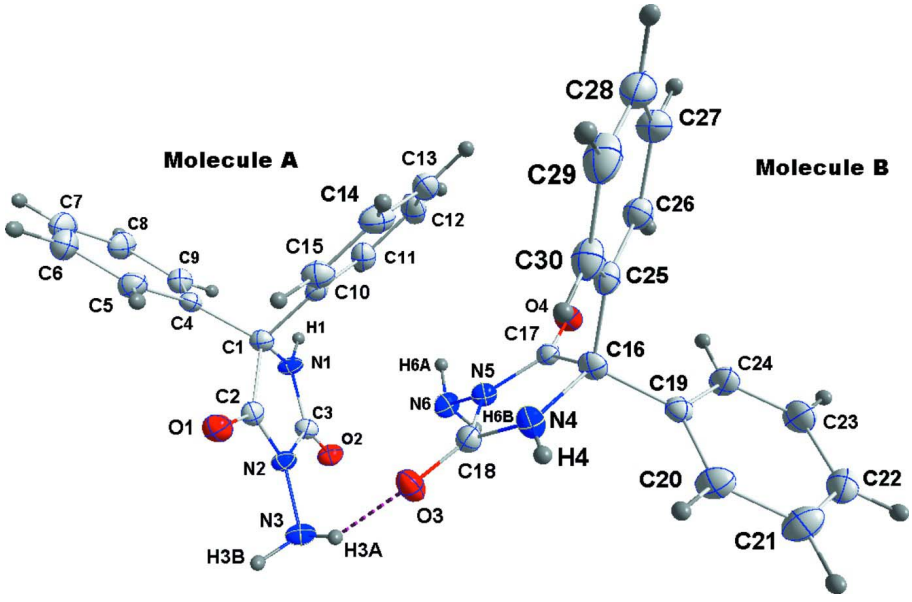


**Figure 1**

Perspective view of the asymmetric unit showing the N—H···O hydrogen bond between molecules A and B as a dashed line. Displacement ellipsoids are drawn at the 50% probability level.

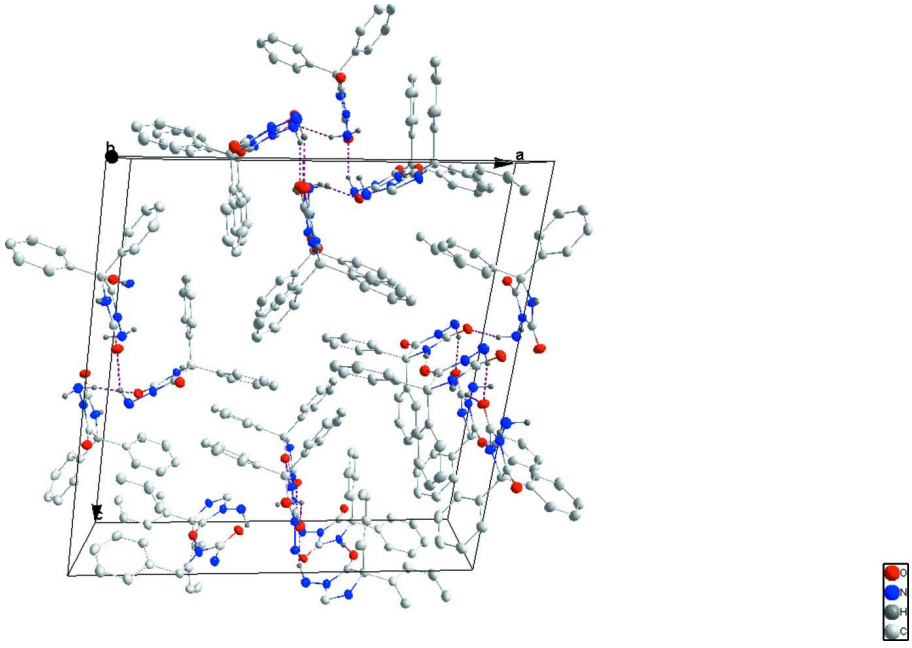


**Figure 2**

Packing diagram viewed perpendicular to (-1,6,-4) along with intermolecular N—H···O interactions shown as dotted lines.

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*Crystal data*

$C_{15}H_{13}N_3O_2$
$M_r = 267.28$
Monoclinic, $P2_1/n$
$a = 20.1565$ (7) Å
$b = 6.1651$ (2) Å
$c = 20.3250$ (7) Å
$\beta = 97.781$ (2)°
$V = 2502.47$ (15) Å$^3$
$Z = 8$
$F(000) = 1120$
$D_x = 1.419$ Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Cell parameters from 9869 reflections
$\theta = 2.9$–$68.2°$
$\mu = 0.79$ mm$^{-1}$
$T = 100$ K
Needle, clear colourless
0.22 × 0.07 × 0.05 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*; Bruker, 2012)
$T_{min} = 0.84$, $T_{max} = 0.96$
32772 measured reflections
4560 independent reflections
3185 reflections with $I > 2\sigma(I)$
$R_{int} = 0.105$
$\theta_{max} = 68.4°$, $\theta_{min} = 2.9°$
$h = -23\rightarrow24$
$k = -7\rightarrow7$
$l = -24\rightarrow24$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.111$
$S = 1.08$
4560 reflections
361 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
$w = 1/[\sigma^2(F_o^2) + (0.021P)^2 + 2.7033P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.22$ e Å$^{-3}$
$\Delta\rho_{min} = -0.24$ 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. H-atoms attached to carbon were placed in calculated positions (C—H = 0.95 Å) while those attached to nitrogen were placed in locations derived from a difference map and their coordinates adjusted to give N—H = 0.91 Å. All were included as riding contributions with isotropic displacement parameters 1.5 times those of the attached atoms.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.78438 (9) | 0.0796 (3) | 0.48058 (9) | 0.0231 (4) |
| O2 | 0.90255 (9) | 0.6421 (3) | 0.40602 (9) | 0.0254 (4) |

| | | | | |
|---|---|---|---|---|
| N1 | 0.80979 (10) | 0.6347 (3) | 0.46106 (10) | 0.0201 (5) |
| H1 | 0.8013 | 0.7785 | 0.4655 | 0.024* |
| N2 | 0.85417 (10) | 0.3212 (3) | 0.43843 (10) | 0.0198 (5) |
| N3 | 0.90346 (11) | 0.1794 (4) | 0.42044 (11) | 0.0255 (5) |
| H3A | 0.9247 | 0.1205 | 0.4586 | 0.031* |
| H3B | 0.8834 | 0.0767 | 0.3923 | 0.031* |
| C1 | 0.77157 (13) | 0.4751 (4) | 0.49361 (12) | 0.0189 (6) |
| C2 | 0.80275 (13) | 0.2652 (4) | 0.47094 (12) | 0.0194 (6) |
| C3 | 0.86045 (13) | 0.5491 (4) | 0.43294 (12) | 0.0205 (6) |
| C4 | 0.69599 (13) | 0.4923 (4) | 0.47123 (12) | 0.0191 (6) |
| C5 | 0.65335 (13) | 0.3213 (5) | 0.48164 (13) | 0.0255 (6) |
| H5 | 0.6714 | 0.1904 | 0.5012 | 0.031* |
| C6 | 0.58517 (14) | 0.3413 (5) | 0.46371 (14) | 0.0303 (7) |
| H6C | 0.5566 | 0.2234 | 0.4708 | 0.036* |
| C7 | 0.55780 (15) | 0.5314 (5) | 0.43552 (14) | 0.0316 (7) |
| H7 | 0.5108 | 0.5442 | 0.4231 | 0.038* |
| C8 | 0.59973 (14) | 0.7018 (5) | 0.42579 (14) | 0.0299 (7) |
| H8 | 0.5814 | 0.8336 | 0.4071 | 0.036* |
| C9 | 0.66835 (14) | 0.6820 (4) | 0.44302 (13) | 0.0246 (6) |
| H9 | 0.6968 | 0.7998 | 0.4354 | 0.030* |
| C10 | 0.78600 (12) | 0.4917 (4) | 0.56998 (13) | 0.0201 (6) |
| C11 | 0.80444 (13) | 0.6912 (4) | 0.59968 (13) | 0.0255 (6) |
| H11 | 0.8090 | 0.8148 | 0.5727 | 0.031* |
| C12 | 0.81613 (14) | 0.7099 (5) | 0.66814 (14) | 0.0309 (7) |
| H12 | 0.8284 | 0.8466 | 0.6877 | 0.037* |
| C13 | 0.81028 (14) | 0.5327 (5) | 0.70839 (14) | 0.0294 (7) |
| H13 | 0.8190 | 0.5462 | 0.7553 | 0.035* |
| C14 | 0.79153 (14) | 0.3359 (5) | 0.67955 (14) | 0.0291 (7) |
| H14 | 0.7872 | 0.2131 | 0.7069 | 0.035* |
| C15 | 0.77886 (14) | 0.3151 (5) | 0.61077 (13) | 0.0259 (6) |
| H15 | 0.7652 | 0.1789 | 0.5916 | 0.031* |
| O3 | 0.93849 (10) | 0.0679 (3) | 0.56241 (9) | 0.0279 (5) |
| O4 | 0.99521 (9) | 0.6386 (3) | 0.70282 (9) | 0.0235 (4) |
| N4 | 0.97628 (11) | 0.0837 (3) | 0.67430 (10) | 0.0218 (5) |
| H4 | 0.9832 | −0.0606 | 0.6816 | 0.026* |
| N5 | 0.96189 (10) | 0.3934 (3) | 0.62039 (10) | 0.0195 (5) |
| N6 | 0.95376 (11) | 0.5316 (3) | 0.56462 (10) | 0.0240 (5) |
| H6A | 0.9316 | 0.6529 | 0.5750 | 0.029* |
| H6B | 0.9960 | 0.5739 | 0.5595 | 0.029* |
| C16 | 0.99947 (13) | 0.2460 (4) | 0.72474 (12) | 0.0200 (6) |
| C17 | 0.98609 (12) | 0.4523 (4) | 0.68303 (12) | 0.0181 (6) |
| C18 | 0.95735 (13) | 0.1661 (4) | 0.61355 (13) | 0.0206 (6) |
| C19 | 1.07440 (13) | 0.2216 (4) | 0.75053 (12) | 0.0195 (6) |
| C20 | 1.10590 (14) | 0.0225 (4) | 0.74733 (14) | 0.0264 (6) |
| H20 | 1.0815 | −0.0973 | 0.7270 | 0.032* |
| C21 | 1.17274 (14) | −0.0036 (5) | 0.77354 (15) | 0.0303 (7) |
| H21 | 1.1935 | −0.1411 | 0.7710 | 0.036* |
| C22 | 1.20901 (14) | 0.1669 (5) | 0.80305 (13) | 0.0283 (7) |

| | | | | |
|---|---|---|---|---|
| H22 | 1.2547 | 0.1484 | 0.8209 | 0.034* |
| C23 | 1.17813 (14) | 0.3665 (5) | 0.80648 (14) | 0.0307 (7) |
| H23 | 1.2029 | 0.4858 | 0.8266 | 0.037* |
| C24 | 1.11154 (14) | 0.3936 (4) | 0.78078 (13) | 0.0264 (6) |
| H24 | 1.0909 | 0.5311 | 0.7838 | 0.032* |
| C25 | 0.95806 (13) | 0.2439 (4) | 0.78283 (12) | 0.0216 (6) |
| C26 | 0.95771 (14) | 0.4223 (5) | 0.82471 (13) | 0.0264 (6) |
| H26 | 0.9832 | 0.5473 | 0.8173 | 0.032* |
| C27 | 0.92022 (14) | 0.4189 (5) | 0.87740 (14) | 0.0322 (7) |
| H27 | 0.9204 | 0.5415 | 0.9057 | 0.039* |
| C28 | 0.88276 (14) | 0.2383 (5) | 0.88873 (14) | 0.0355 (8) |
| H28 | 0.8567 | 0.2373 | 0.9243 | 0.043* |
| C29 | 0.88340 (15) | 0.0586 (5) | 0.84782 (15) | 0.0347 (7) |
| H29 | 0.8579 | −0.0663 | 0.8555 | 0.042* |
| C30 | 0.92130 (13) | 0.0610 (5) | 0.79549 (13) | 0.0267 (6) |
| H30 | 0.9221 | −0.0635 | 0.7681 | 0.032* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0258 (10) | 0.0146 (10) | 0.0292 (10) | −0.0005 (8) | 0.0052 (8) | −0.0005 (8) |
| O2 | 0.0259 (10) | 0.0235 (10) | 0.0284 (10) | −0.0002 (8) | 0.0102 (9) | 0.0043 (8) |
| N1 | 0.0208 (12) | 0.0124 (11) | 0.0292 (12) | 0.0016 (9) | 0.0107 (10) | 0.0000 (9) |
| N2 | 0.0195 (12) | 0.0135 (11) | 0.0270 (12) | 0.0036 (9) | 0.0055 (10) | −0.0002 (9) |
| N3 | 0.0281 (13) | 0.0198 (12) | 0.0298 (13) | 0.0066 (10) | 0.0081 (11) | −0.0010 (10) |
| C1 | 0.0213 (14) | 0.0136 (13) | 0.0226 (13) | −0.0012 (11) | 0.0060 (11) | −0.0004 (10) |
| C2 | 0.0205 (14) | 0.0168 (14) | 0.0201 (13) | 0.0014 (11) | −0.0007 (11) | −0.0001 (11) |
| C3 | 0.0241 (15) | 0.0180 (14) | 0.0198 (13) | 0.0009 (12) | 0.0045 (12) | 0.0005 (11) |
| C4 | 0.0206 (14) | 0.0190 (14) | 0.0183 (13) | 0.0000 (11) | 0.0052 (11) | −0.0046 (11) |
| C5 | 0.0236 (15) | 0.0216 (15) | 0.0328 (16) | −0.0013 (12) | 0.0088 (13) | −0.0022 (12) |
| C6 | 0.0254 (16) | 0.0313 (17) | 0.0346 (17) | −0.0070 (13) | 0.0057 (13) | −0.0034 (13) |
| C7 | 0.0234 (16) | 0.0431 (19) | 0.0278 (16) | 0.0010 (14) | 0.0014 (13) | −0.0044 (14) |
| C8 | 0.0256 (16) | 0.0341 (17) | 0.0287 (15) | 0.0066 (13) | −0.0006 (13) | 0.0045 (13) |
| C9 | 0.0257 (15) | 0.0241 (15) | 0.0239 (14) | −0.0011 (12) | 0.0026 (12) | 0.0022 (11) |
| C10 | 0.0153 (13) | 0.0207 (14) | 0.0247 (14) | 0.0014 (11) | 0.0040 (11) | −0.0021 (11) |
| C11 | 0.0267 (15) | 0.0204 (15) | 0.0281 (15) | 0.0003 (12) | −0.0006 (13) | 0.0005 (12) |
| C12 | 0.0323 (17) | 0.0249 (16) | 0.0331 (16) | 0.0027 (13) | −0.0041 (14) | −0.0077 (13) |
| C13 | 0.0242 (16) | 0.0375 (18) | 0.0254 (15) | 0.0082 (13) | −0.0001 (12) | −0.0067 (13) |
| C14 | 0.0291 (16) | 0.0319 (17) | 0.0282 (15) | 0.0054 (13) | 0.0101 (13) | 0.0050 (13) |
| C15 | 0.0283 (16) | 0.0215 (15) | 0.0292 (15) | 0.0014 (12) | 0.0088 (13) | 0.0004 (12) |
| O3 | 0.0360 (12) | 0.0214 (10) | 0.0247 (10) | −0.0002 (9) | −0.0018 (9) | −0.0042 (8) |
| O4 | 0.0294 (11) | 0.0136 (10) | 0.0275 (10) | −0.0019 (8) | 0.0035 (8) | −0.0010 (8) |
| N4 | 0.0302 (13) | 0.0122 (11) | 0.0214 (12) | −0.0008 (10) | −0.0024 (10) | 0.0014 (9) |
| N5 | 0.0217 (12) | 0.0153 (11) | 0.0219 (11) | 0.0006 (9) | 0.0040 (10) | 0.0016 (9) |
| N6 | 0.0259 (13) | 0.0206 (12) | 0.0254 (12) | 0.0031 (10) | 0.0040 (10) | 0.0070 (10) |
| C16 | 0.0246 (15) | 0.0122 (13) | 0.0226 (13) | −0.0020 (11) | 0.0017 (11) | −0.0024 (11) |
| C17 | 0.0150 (13) | 0.0187 (14) | 0.0208 (13) | 0.0000 (11) | 0.0036 (11) | −0.0008 (11) |
| C18 | 0.0194 (14) | 0.0177 (14) | 0.0244 (14) | −0.0005 (11) | 0.0014 (12) | −0.0004 (11) |

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|---|---|---|---|---|---|---|
| C19 | 0.0224 (14) | 0.0196 (14) | 0.0171 (13) | 0.0010 (11) | 0.0045 (11) | 0.0010 (11) |
| C20 | 0.0256 (16) | 0.0180 (15) | 0.0361 (16) | −0.0013 (12) | 0.0060 (13) | −0.0017 (12) |
| C21 | 0.0273 (16) | 0.0201 (15) | 0.0444 (18) | 0.0040 (12) | 0.0074 (14) | 0.0044 (13) |
| C22 | 0.0234 (15) | 0.0331 (17) | 0.0277 (15) | 0.0026 (13) | 0.0009 (13) | 0.0051 (12) |
| C23 | 0.0282 (16) | 0.0295 (17) | 0.0331 (16) | −0.0020 (13) | −0.0006 (13) | −0.0063 (13) |
| C24 | 0.0259 (16) | 0.0214 (15) | 0.0306 (15) | 0.0015 (12) | −0.0010 (13) | −0.0049 (12) |
| C25 | 0.0196 (14) | 0.0218 (14) | 0.0223 (13) | 0.0008 (11) | −0.0005 (11) | 0.0048 (11) |
| C26 | 0.0273 (16) | 0.0259 (15) | 0.0259 (15) | 0.0023 (13) | 0.0025 (12) | 0.0013 (12) |
| C27 | 0.0294 (17) | 0.0413 (19) | 0.0266 (15) | 0.0058 (14) | 0.0064 (13) | 0.0005 (13) |
| C28 | 0.0249 (16) | 0.055 (2) | 0.0269 (16) | 0.0029 (15) | 0.0056 (13) | 0.0110 (15) |
| C29 | 0.0253 (16) | 0.0420 (19) | 0.0357 (17) | −0.0092 (14) | −0.0004 (14) | 0.0117 (15) |
| C30 | 0.0225 (15) | 0.0284 (16) | 0.0284 (15) | −0.0046 (12) | 0.0001 (12) | 0.0059 (12) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C2 | 1.226 (3) | O3—C18 | 1.218 (3) |
| O2—C3 | 1.214 (3) | O4—C17 | 1.222 (3) |
| N1—C3 | 1.344 (3) | N4—C18 | 1.342 (3) |
| N1—C1 | 1.462 (3) | N4—C16 | 1.463 (3) |
| N1—H1 | 0.9100 | N4—H4 | 0.9100 |
| N2—C2 | 1.347 (3) | N5—C17 | 1.350 (3) |
| N2—N3 | 1.408 (3) | N5—N6 | 1.410 (3) |
| N2—C3 | 1.417 (3) | N5—C18 | 1.410 (3) |
| N3—H3A | 0.9100 | N6—H6A | 0.9101 |
| N3—H3B | 0.9100 | N6—H6B | 0.9099 |
| C1—C4 | 1.533 (4) | C16—C17 | 1.532 (3) |
| C1—C2 | 1.536 (3) | C16—C25 | 1.536 (3) |
| C1—C10 | 1.543 (3) | C16—C19 | 1.537 (4) |
| C4—C9 | 1.386 (4) | C19—C20 | 1.388 (4) |
| C4—C5 | 1.394 (4) | C19—C24 | 1.392 (4) |
| C5—C6 | 1.379 (4) | C20—C21 | 1.389 (4) |
| C5—H5 | 0.9500 | C20—H20 | 0.9500 |
| C6—C7 | 1.386 (4) | C21—C22 | 1.371 (4) |
| C6—H6C | 0.9500 | C21—H21 | 0.9500 |
| C7—C8 | 1.379 (4) | C22—C23 | 1.385 (4) |
| C7—H7 | 0.9500 | C22—H22 | 0.9500 |
| C8—C9 | 1.385 (4) | C23—C24 | 1.383 (4) |
| C8—H8 | 0.9500 | C23—H23 | 0.9500 |
| C9—H9 | 0.9500 | C24—H24 | 0.9500 |
| C10—C15 | 1.388 (4) | C25—C26 | 1.392 (4) |
| C10—C11 | 1.398 (4) | C25—C30 | 1.392 (4) |
| C11—C12 | 1.384 (4) | C26—C27 | 1.392 (4) |
| C11—H11 | 0.9500 | C26—H26 | 0.9500 |
| C12—C13 | 1.379 (4) | C27—C28 | 1.382 (4) |
| C12—H12 | 0.9500 | C27—H27 | 0.9500 |
| C13—C14 | 1.378 (4) | C28—C29 | 1.386 (4) |
| C13—H13 | 0.9500 | C28—H28 | 0.9500 |
| C14—C15 | 1.392 (4) | C29—C30 | 1.391 (4) |

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|---|---|---|---|
| C14—H14 | 0.9500 | C29—H29 | 0.9500 |
| C15—H15 | 0.9500 | C30—H30 | 0.9500 |
| | | | |
| C3—N1—C1 | 113.9 (2) | C18—N4—C16 | 114.2 (2) |
| C3—N1—H1 | 126.1 | C18—N4—H4 | 122.6 |
| C1—N1—H1 | 119.5 | C16—N4—H4 | 121.8 |
| C2—N2—N3 | 125.8 (2) | C17—N5—N6 | 125.7 (2) |
| C2—N2—C3 | 112.1 (2) | C17—N5—C18 | 111.8 (2) |
| N3—N2—C3 | 121.4 (2) | N6—N5—C18 | 121.5 (2) |
| N2—N3—H3A | 107.0 | N5—N6—H6A | 108.6 |
| N2—N3—H3B | 109.0 | N5—N6—H6B | 104.8 |
| H3A—N3—H3B | 112.1 | H6A—N6—H6B | 106.7 |
| N1—C1—C4 | 112.5 (2) | N4—C16—C17 | 99.47 (19) |
| N1—C1—C2 | 99.74 (19) | N4—C16—C25 | 112.2 (2) |
| C4—C1—C2 | 113.6 (2) | C17—C16—C25 | 111.1 (2) |
| N1—C1—C10 | 111.8 (2) | N4—C16—C19 | 112.2 (2) |
| C4—C1—C10 | 109.8 (2) | C17—C16—C19 | 111.2 (2) |
| C2—C1—C10 | 109.2 (2) | C25—C16—C19 | 110.4 (2) |
| O1—C2—N2 | 125.9 (2) | O4—C17—N5 | 125.6 (2) |
| O1—C2—C1 | 126.4 (2) | O4—C17—C16 | 126.2 (2) |
| N2—C2—C1 | 107.7 (2) | N5—C17—C16 | 108.2 (2) |
| O2—C3—N1 | 128.6 (2) | O3—C18—N4 | 127.9 (2) |
| O2—C3—N2 | 125.4 (2) | O3—C18—N5 | 125.9 (2) |
| N1—C3—N2 | 105.9 (2) | N4—C18—N5 | 106.2 (2) |
| C9—C4—C5 | 118.6 (3) | C20—C19—C24 | 118.2 (2) |
| C9—C4—C1 | 120.6 (2) | C20—C19—C16 | 120.3 (2) |
| C5—C4—C1 | 120.7 (2) | C24—C19—C16 | 121.4 (2) |
| C6—C5—C4 | 120.3 (3) | C19—C20—C21 | 120.7 (3) |
| C6—C5—H5 | 119.8 | C19—C20—H20 | 119.6 |
| C4—C5—H5 | 119.8 | C21—C20—H20 | 119.6 |
| C5—C6—C7 | 120.8 (3) | C22—C21—C20 | 120.7 (3) |
| C5—C6—H6C | 119.6 | C22—C21—H21 | 119.6 |
| C7—C6—H6C | 119.6 | C20—C21—H21 | 119.6 |
| C8—C7—C6 | 119.1 (3) | C21—C22—C23 | 119.1 (3) |
| C8—C7—H7 | 120.5 | C21—C22—H22 | 120.4 |
| C6—C7—H7 | 120.5 | C23—C22—H22 | 120.4 |
| C7—C8—C9 | 120.5 (3) | C24—C23—C22 | 120.5 (3) |
| C7—C8—H8 | 119.8 | C24—C23—H23 | 119.7 |
| C9—C8—H8 | 119.8 | C22—C23—H23 | 119.7 |
| C8—C9—C4 | 120.7 (3) | C23—C24—C19 | 120.8 (3) |
| C8—C9—H9 | 119.7 | C23—C24—H24 | 119.6 |
| C4—C9—H9 | 119.7 | C19—C24—H24 | 119.6 |
| C15—C10—C11 | 118.4 (2) | C26—C25—C30 | 118.7 (3) |
| C15—C10—C1 | 121.9 (2) | C26—C25—C16 | 120.8 (2) |
| C11—C10—C1 | 119.7 (2) | C30—C25—C16 | 120.5 (2) |
| C12—C11—C10 | 120.4 (3) | C25—C26—C27 | 120.5 (3) |
| C12—C11—H11 | 119.8 | C25—C26—H26 | 119.8 |
| C10—C11—H11 | 119.8 | C27—C26—H26 | 119.8 |

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| C13—C12—C11 | 120.9 (3) | C28—C27—C26 | 120.4 (3) |
| C13—C12—H12 | 119.5 | C28—C27—H27 | 119.8 |
| C11—C12—H12 | 119.5 | C26—C27—H27 | 119.8 |
| C14—C13—C12 | 119.0 (3) | C27—C28—C29 | 119.6 (3) |
| C14—C13—H13 | 120.5 | C27—C28—H28 | 120.2 |
| C12—C13—H13 | 120.5 | C29—C28—H28 | 120.2 |
| C13—C14—C15 | 120.7 (3) | C28—C29—C30 | 120.0 (3) |
| C13—C14—H14 | 119.6 | C28—C29—H29 | 120.0 |
| C15—C14—H14 | 119.6 | C30—C29—H29 | 120.0 |
| C10—C15—C14 | 120.5 (3) | C29—C30—C25 | 120.7 (3) |
| C10—C15—H15 | 119.7 | C29—C30—H30 | 119.6 |
| C14—C15—H15 | 119.7 | C25—C30—H30 | 119.6 |

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|---|---|---|---|
| C3—N1—C1—C4 | −128.7 (2) | C18—N4—C16—C17 | 3.9 (3) |
| C3—N1—C1—C2 | −8.0 (3) | C18—N4—C16—C25 | 121.4 (2) |
| C3—N1—C1—C10 | 107.3 (2) | C18—N4—C16—C19 | −113.7 (2) |
| N3—N2—C2—O1 | −12.5 (4) | N6—N5—C17—O4 | 11.6 (4) |
| C3—N2—C2—O1 | 177.1 (2) | C18—N5—C17—O4 | 180.0 (2) |
| N3—N2—C2—C1 | 167.5 (2) | N6—N5—C17—C16 | −168.9 (2) |
| C3—N2—C2—C1 | −2.9 (3) | C18—N5—C17—C16 | −0.5 (3) |
| N1—C1—C2—O1 | −173.8 (2) | N4—C16—C17—O4 | 177.7 (3) |
| C4—C1—C2—O1 | −53.9 (3) | C25—C16—C17—O4 | 59.4 (3) |
| C10—C1—C2—O1 | 68.9 (3) | C19—C16—C17—O4 | −63.9 (3) |
| N1—C1—C2—N2 | 6.2 (2) | N4—C16—C17—N5 | −1.9 (3) |
| C4—C1—C2—N2 | 126.1 (2) | C25—C16—C17—N5 | −120.2 (2) |
| C10—C1—C2—N2 | −111.1 (2) | C19—C16—C17—N5 | 116.5 (2) |
| C1—N1—C3—O2 | −175.5 (3) | C16—N4—C18—O3 | 177.1 (3) |
| C1—N1—C3—N2 | 6.7 (3) | C16—N4—C18—N5 | −4.4 (3) |
| C2—N2—C3—O2 | 180.0 (3) | C17—N5—C18—O3 | −178.5 (3) |
| N3—N2—C3—O2 | 9.1 (4) | N6—N5—C18—O3 | −9.5 (4) |
| C2—N2—C3—N1 | −2.1 (3) | C17—N5—C18—N4 | 2.9 (3) |
| N3—N2—C3—N1 | −173.0 (2) | N6—N5—C18—N4 | 171.9 (2) |
| N1—C1—C4—C9 | −21.0 (3) | N4—C16—C19—C20 | −25.0 (3) |
| C2—C1—C4—C9 | −133.3 (2) | C17—C16—C19—C20 | −135.4 (2) |
| C10—C1—C4—C9 | 104.2 (3) | C25—C16—C19—C20 | 100.9 (3) |
| N1—C1—C4—C5 | 162.1 (2) | N4—C16—C19—C24 | 158.5 (2) |
| C2—C1—C4—C5 | 49.7 (3) | C17—C16—C19—C24 | 48.1 (3) |
| C10—C1—C4—C5 | −72.8 (3) | C25—C16—C19—C24 | −75.6 (3) |
| C9—C4—C5—C6 | 0.4 (4) | C24—C19—C20—C21 | −0.1 (4) |
| C1—C4—C5—C6 | 177.5 (2) | C16—C19—C20—C21 | −176.8 (2) |
| C4—C5—C6—C7 | −0.4 (4) | C19—C20—C21—C22 | −0.1 (4) |
| C5—C6—C7—C8 | −0.3 (4) | C20—C21—C22—C23 | 0.0 (4) |
| C6—C7—C8—C9 | 1.0 (4) | C21—C22—C23—C24 | 0.3 (4) |
| C7—C8—C9—C4 | −1.0 (4) | C22—C23—C24—C19 | −0.5 (4) |
| C5—C4—C9—C8 | 0.3 (4) | C20—C19—C24—C23 | 0.4 (4) |
| C1—C4—C9—C8 | −176.8 (2) | C16—C19—C24—C23 | 177.0 (2) |
| N1—C1—C10—C15 | −153.5 (2) | N4—C16—C25—C26 | −160.2 (2) |
| C4—C1—C10—C15 | 80.9 (3) | C17—C16—C25—C26 | −49.8 (3) |

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| C2—C1—C10—C15 | −44.2 (3) | C19—C16—C25—C26 | 73.9 (3) |
| N1—C1—C10—C11 | 28.9 (3) | N4—C16—C25—C30 | 21.3 (3) |
| C4—C1—C10—C11 | −96.7 (3) | C17—C16—C25—C30 | 131.6 (3) |
| C2—C1—C10—C11 | 138.3 (2) | C19—C16—C25—C30 | −104.6 (3) |
| C15—C10—C11—C12 | 0.9 (4) | C30—C25—C26—C27 | −1.2 (4) |
| C1—C10—C11—C12 | 178.6 (2) | C16—C25—C26—C27 | −179.8 (2) |
| C10—C11—C12—C13 | 0.4 (4) | C25—C26—C27—C28 | −0.1 (4) |
| C11—C12—C13—C14 | −1.0 (4) | C26—C27—C28—C29 | 0.9 (4) |
| C12—C13—C14—C15 | 0.2 (4) | C27—C28—C29—C30 | −0.4 (4) |
| C11—C10—C15—C14 | −1.6 (4) | C28—C29—C30—C25 | −1.0 (4) |
| C1—C10—C15—C14 | −179.3 (2) | C26—C25—C30—C29 | 1.8 (4) |
| C13—C14—C15—C10 | 1.1 (4) | C16—C25—C30—C29 | −179.6 (2) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···O1$^{i}$ | 0.91 | 1.92 | 2.828 (3) | 177 |
| N3—H3*A*···O3 | 0.91 | 2.11 | 2.957 (3) | 154 |
| N4—H4···O4$^{ii}$ | 0.91 | 1.91 | 2.820 (3) | 176 |
| N6—H6*A*···O3$^{i}$ | 0.91 | 2.58 | 3.320 (3) | 139 |
| N6—H6*B*···O2$^{iii}$ | 0.91 | 2.46 | 3.070 (3) | 124 |
| N6—H6*B*···N3$^{iii}$ | 0.91 | 2.52 | 3.363 (3) | 154 |

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