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(4*E*)-1-Phenyl-4-{[(pyridin-2-yl)amino]methylidene}pyrazolidine-3,5-dione

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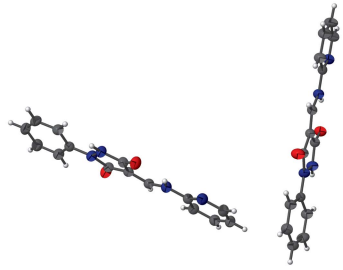
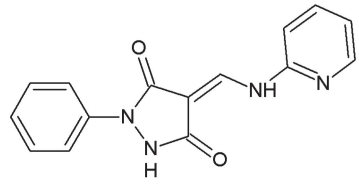
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{15}H_{12}N_4O_2$, contains two independent molecules in the asymmetric unit. These differ in terms of dihedral angles that the phenyl and 2-pyridyl rings subtend with the central five-membered ring; 15.2 (2) and 2.9 (2)°, respectively, in one molecule, 8.9 (2) and 5.1 (2)°for the second. In the crystal, the independent molecules each self-associate to form layers through N—H···O and C—H···O hydrogen bonding. The layers associate through π–π interactions between the phenyl rings and isolated carbon–carbon double bonds [shortest midpoint–centroid distance = 3.347 (4) Å]. The crystal studied was refined as a two-component twin.

3D view



Chemical scheme



Structure description

Pyrazolone scaffold compounds have been shown to possess diverse biological properties such as tranquilizing, muscle-relaxant, psycho-analeptic, anti-convulsant, anti-hypertensive, anti-depressant, anti-pyretic and analgesic activities (Turan-Zitouni *et al.*, 2000; Rajendra Prasad *et al.*, 2005). Some pyrazolone derivatives are used as anti-inflammatory (Bekhit *et al.*, 2009*a*,*b*), anti-cancer (Nitulescu *et al.*, 2010) and enzyme inhibitory (Li *et al.*, 2004) agents. In this context, we report here the crystal structure of the title compound.

The asymmetric unit contains two independent molecules which differ modestly in their conformations, Fig. 1. Thus in molecule 1, the the phenyl and 2-pyridyl rings are inclined by 15.2 (2) and 2.9 (2)°, respectively, to the central five-membered ring while in molecule 2, the corresponding angles are 8.9 (2) and 5.1 (2)°. Through N—H···O and

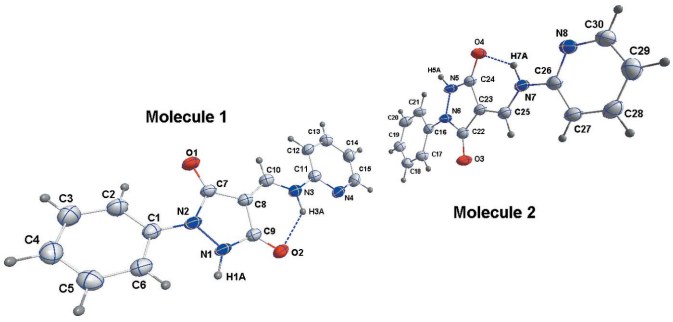


Figure 1
The asymmetric unit with atom labelling scheme and 50% probability ellipsoids.

C—H···O hydrogen bonding, each independent molecule forms layers with its symmetry-related counterparts (Table 1 and Fig. 2). These layers are inclined at approximately 81° to one another. Successive layers containing molecule 1 associate *via* π–π interactions between the C1–C6 phenyl ring and the C8═C10 π-bond in the molecule at $1-x, 2-y, 1-z$ with a midpoint–centroid distance of 3.347 (4) Å. An analogous interaction occurs between the layers comprising molecule 2 (C16–C21 ring and C23═C25 double bond) with a midpoint–centroid distance of 3.447 (4) Å.

Synthesis and crystallization

The title compound was prepared according to our reported method (Ahmed, 2015). Pale-yellow crystals suitable for X-ray analysis were grown from pyridine (m.p. 541–543 K, yield 91%).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The crystal studied was refined as a two-component twin. Several reflections, *i.e.* (0 2 6), ($\bar{2}$ $\bar{9}$ 5), ($\bar{1}$ $\bar{9}$ 5), (0 $\bar{9}$ 5), ($\bar{1}$ 9 $\bar{5}$) and (1 $\bar{9}$ 5) were omitted from the final cycles of refinement owing to poor agreement.

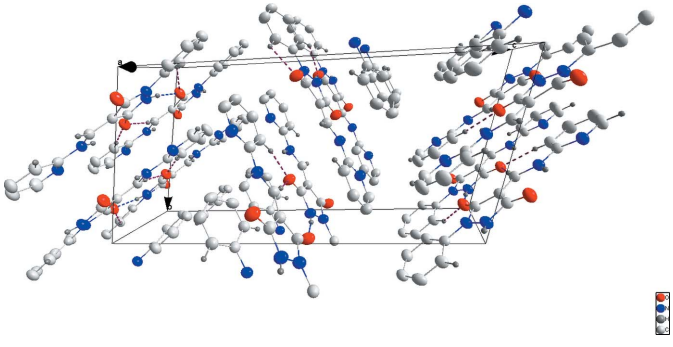


Figure 2
Packing showing the intermolecular N—H···O and C—H···O hydrogen bonds as, respectively, blue and purple dotted lines.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.91 (5)	1.97 (5)	2.869 (3)	168 (4)
N3—H3A···O2	0.96 (5)	2.10 (5)	2.812 (4)	130 (4)
C2—H2···O1	0.94	2.33	2.920 (4)	121
C6—H6···O1^{i}	0.94	2.57	3.401 (4)	147
C10—H10···O2^{ii}	0.94	2.29	3.222 (4)	170
C12—H12···O2^{ii}	0.94	2.24	3.175 (4)	170
N5—H5A···O3^{ii}	0.86 (4)	2.02 (4)	2.866 (4)	171 (4)
N7—H7A···O4	0.94 (4)	2.03 (4)	2.794 (3)	138 (3)
C17—H17···O3	0.94	2.27	2.895 (4)	124
C21—H21···O3^{ii}	0.94	2.53	3.392 (4)	153
C25—H25···O4^{i}	0.94	2.30	3.230 (4)	172
C27—H27···O4^{i}	0.94	2.29	3.222 (4)	173

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{12}N_4O_2$
M_r	280.29
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	220
a, b, c (Å)	6.2175 (2), 10.2646 (4), 20.7094 (8)
α, β, γ (°)	102.511 (2), 94.343 (2), 95.263 (2)
V (Å^3)	1278.63 (8)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.83
Crystal size (mm)	0.21 × 0.07 × 0.02
Data collection	
Diffractometer	Bruker D8 VENTURE PHOTON 100 CMOS
Absorption correction	Multi-scan (*TWINABS*; Sheldrick, 2009)
T_{min}, T_{max}	0.81, 0.98
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	24529, 12138, 10472
R_{int}	0.054
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.620
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.066, 0.171, 1.11
No. of reflections	12138
No. of parameters	396
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.26, −0.22

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014/7* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg & Putz, 2012) and *SHELXTL* (Sheldrick, 2008).

Acknowledgements

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full crystallographic data

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Crystal data

$C_{15}H_{12}N_4O_2$
M_r = 280.29
Triclinic, $P\overline{1}$
a = 6.2175 (2) Å
b = 10.2646 (4) Å
c = 20.7094 (8) Å
α = 102.511 (2)°
β = 94.343 (2)°
γ = 95.263 (2)°
V = 1278.63 (8) Å^3
Z = 4
$F(000)$ = 584
D_x = 1.456 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 9905 reflections
θ = 4.4–72.9°
μ = 0.83 mm^{-1}
T = 220 K
Column, yellow
0.21 × 0.07 × 0.02 mm

Data collection

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer
Radiation source: INCOATEC IμS micro–focus source
Mirror monochromator
Detector resolution: 10.4167 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*TWINABS*; Sheldrick, 2009)
T_{min} = 0.81, T_{max} = 0.98
24529 measured reflections
12138 independent reflections
10472 reflections with $I > 2\sigma(I)$
R_{int} = 0.054
θ_{max} = 72.8°, θ_{min} = 4.4°
h = −7→7
k = −12→12
l = −25→25

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.171
S = 1.11
12138 reflections
396 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
$w = 1/[\sigma^2(F_o^2) + (0.0669P)^2 + 0.7168P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Special details

Experimental. Analysis of 1259 reflections having I/σ(I) > 13 and chosen from the full data set with *CELL_NOW* (Sheldrick, 2008) showed the crystal to belong to the triclinic system and to be twinned by a 180° rotation about the *c** axis. The raw data were processed using the multi-component version of *SAINT* under control of the two-component orientation file generated by *CELL_NOW*.

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. H-atoms attached to carbon were placed in calculated positions (C—H = 0.95 Å) and included as riding contributions with isotropic displacement parameters 1.2 times those of the attached atoms. Refined as a 2-component twin.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.2523 (4)	0.9078 (2)	0.56147 (13)	0.0414 (6)
O2	0.7975 (4)	0.6606 (3)	0.46405 (12)	0.0408 (6)
N1	0.7883 (4)	0.8648 (3)	0.53604 (14)	0.0335 (6)
H1A	0.932 (8)	0.888 (4)	0.549 (2)	0.056 (12)*
N2	0.6284 (4)	0.9343 (3)	0.56886 (13)	0.0327 (6)
N3	0.3715 (5)	0.5366 (3)	0.42450 (14)	0.0356 (6)
H3A	0.522 (8)	0.529 (5)	0.419 (2)	0.061 (13)*
N4	0.3472 (5)	0.3343 (3)	0.35087 (14)	0.0378 (6)
C1	0.6862 (5)	1.0608 (3)	0.61282 (15)	0.0315 (7)
C2	0.5388 (6)	1.1181 (4)	0.65468 (17)	0.0400 (8)
H2	0.3995	1.0731	0.6537	0.048*
C3	0.5980 (6)	1.2413 (4)	0.69752 (18)	0.0451 (8)
H3	0.4969	1.2804	0.7251	0.054*
C4	0.8024 (6)	1.3081 (4)	0.70074 (17)	0.0428 (8)
H4	0.8417	1.3914	0.7306	0.051*
C5	0.9486 (6)	1.2506 (4)	0.65940 (18)	0.0411 (8)
H5	1.0884	1.2955	0.6611	0.049*
C6	0.8923 (5)	1.1277 (3)	0.61545 (16)	0.0360 (7)
H6	0.9932	1.0896	0.5875	0.043*
C7	0.4242 (5)	0.8666 (3)	0.54520 (15)	0.0305 (6)
C8	0.4648 (5)	0.7457 (3)	0.49929 (15)	0.0309 (6)
C9	0.6952 (5)	0.7474 (3)	0.49606 (15)	0.0311 (6)
C10	0.3128 (5)	0.6440 (3)	0.46519 (15)	0.0325 (7)
H10	0.1650	0.6502	0.4706	0.039*
C11	0.2378 (5)	0.4245 (3)	0.38691 (15)	0.0328 (7)
C12	0.0137 (5)	0.4102 (3)	0.38836 (16)	0.0356 (7)
H12	−0.0566	0.4775	0.4141	0.043*
C13	−0.1015 (6)	0.2943 (4)	0.35084 (17)	0.0401 (8)
H13	−0.2531	0.2808	0.3508	0.048*
C14	0.0074 (6)	0.1972 (4)	0.31298 (17)	0.0420 (8)

H14	−0.0678	0.1169	0.2872	0.050*
C15	0.2289 (6)	0.2228 (4)	0.31449 (17)	0.0411 (8)
H15	0.3025	0.1579	0.2883	0.049*
O3	0.2981 (4)	0.1607 (3)	0.06003 (12)	0.0408 (6)
O4	−0.3107 (4)	0.3200 (3)	−0.03639 (12)	0.0377 (5)
N5	−0.2559 (4)	0.1755 (3)	0.03312 (14)	0.0332 (6)
H5A	−0.386 (7)	0.164 (4)	0.0435 (18)	0.038 (10)*
N6	−0.0752 (4)	0.1344 (3)	0.06515 (13)	0.0314 (6)
N7	0.0864 (4)	0.4084 (3)	−0.07590 (13)	0.0308 (5)
H7A	−0.065 (7)	0.406 (4)	−0.0776 (17)	0.036 (10)*
N8	0.0571 (4)	0.5406 (3)	−0.15078 (13)	0.0360 (6)
C16	−0.1045 (5)	0.0432 (3)	0.10675 (14)	0.0286 (6)
C17	0.0727 (5)	0.0106 (4)	0.14271 (16)	0.0359 (7)
H17	0.2138	0.0484	0.1394	0.043*
C18	0.0385 (6)	−0.0785 (4)	0.18350 (17)	0.0389 (7)
H18	0.1583	−0.1011	0.2076	0.047*
C19	−0.1664 (6)	−0.1346 (3)	0.18949 (17)	0.0380 (7)
H19	−0.1867	−0.1939	0.2179	0.046*
C20	−0.3419 (6)	−0.1028 (4)	0.15342 (19)	0.0418 (8)
H20	−0.4824	−0.1411	0.1571	0.050*
C21	−0.3126 (5)	−0.0150 (4)	0.11177 (17)	0.0366 (7)
H21	−0.4328	0.0054	0.0869	0.044*
C22	0.1142 (5)	0.1857 (3)	0.04333 (15)	0.0312 (6)
C23	0.0459 (5)	0.2672 (3)	−0.00137 (14)	0.0284 (6)
C24	−0.1876 (5)	0.2606 (3)	−0.00493 (15)	0.0293 (6)
C25	0.1733 (5)	0.3375 (3)	−0.03593 (14)	0.0295 (6)
H25	0.3246	0.3358	−0.0314	0.035*
C26	0.1927 (5)	0.4805 (3)	−0.11675 (14)	0.0298 (6)
C27	0.4153 (5)	0.4882 (4)	−0.12135 (17)	0.0373 (7)
H27	0.5054	0.4444	−0.0965	0.045*
C28	0.4998 (6)	0.5628 (4)	−0.16385 (18)	0.0435 (8)
H28	0.6495	0.5706	−0.1682	0.052*
C29	0.3636 (6)	0.6253 (4)	−0.19946 (19)	0.0483 (9)
H29	0.4173	0.6758	−0.2288	0.058*
C30	0.1447 (6)	0.6116 (4)	−0.19100 (18)	0.0441 (8)
H30	0.0518	0.6552	−0.2151	0.053*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0212 (11)	0.0423 (14)	0.0570 (14)	0.0073 (10)	0.0067 (10)	0.0002 (11)
O2	0.0268 (12)	0.0421 (14)	0.0498 (13)	0.0088 (10)	0.0078 (10)	−0.0013 (10)
N1	0.0182 (13)	0.0360 (15)	0.0447 (14)	0.0067 (11)	0.0059 (10)	0.0028 (12)
N2	0.0191 (12)	0.0351 (15)	0.0432 (14)	0.0061 (11)	0.0051 (10)	0.0058 (11)
N3	0.0278 (14)	0.0388 (15)	0.0391 (13)	0.0062 (11)	0.0040 (11)	0.0051 (11)
N4	0.0313 (15)	0.0400 (16)	0.0424 (14)	0.0099 (12)	0.0081 (12)	0.0060 (12)
C1	0.0291 (15)	0.0334 (16)	0.0333 (14)	0.0069 (13)	0.0023 (12)	0.0089 (12)
C2	0.0312 (17)	0.0424 (19)	0.0455 (17)	0.0038 (15)	0.0079 (14)	0.0066 (15)

C3	0.042 (2)	0.044 (2)	0.0460 (18)	0.0065 (16)	0.0089 (15)	0.0023 (15)
C4	0.048 (2)	0.0339 (18)	0.0428 (17)	0.0052 (16)	−0.0029 (15)	0.0029 (14)
C5	0.0360 (19)	0.0374 (19)	0.0488 (18)	−0.0034 (15)	−0.0025 (14)	0.0124 (15)
C6	0.0290 (16)	0.0388 (18)	0.0415 (16)	0.0063 (13)	0.0046 (13)	0.0104 (14)
C7	0.0202 (14)	0.0348 (17)	0.0370 (15)	0.0049 (12)	0.0046 (11)	0.0079 (12)
C8	0.0241 (15)	0.0350 (17)	0.0354 (15)	0.0069 (12)	0.0048 (11)	0.0097 (12)
C9	0.0238 (15)	0.0365 (17)	0.0334 (14)	0.0077 (12)	0.0040 (11)	0.0066 (12)
C10	0.0276 (16)	0.0347 (17)	0.0362 (15)	0.0053 (13)	0.0045 (12)	0.0087 (13)
C11	0.0296 (16)	0.0344 (17)	0.0349 (15)	0.0065 (13)	0.0034 (12)	0.0078 (12)
C12	0.0303 (16)	0.0360 (17)	0.0417 (16)	0.0116 (13)	0.0078 (13)	0.0065 (13)
C13	0.0348 (18)	0.0432 (19)	0.0418 (17)	0.0043 (15)	0.0038 (14)	0.0086 (14)
C14	0.043 (2)	0.0381 (19)	0.0410 (17)	0.0019 (15)	0.0035 (14)	0.0030 (14)
C15	0.044 (2)	0.0365 (18)	0.0418 (17)	0.0107 (15)	0.0070 (15)	0.0029 (14)
O3	0.0229 (11)	0.0524 (15)	0.0549 (14)	0.0105 (10)	0.0052 (10)	0.0261 (12)
O4	0.0228 (11)	0.0484 (14)	0.0474 (12)	0.0089 (10)	0.0013 (9)	0.0218 (11)
N5	0.0172 (13)	0.0410 (16)	0.0466 (14)	0.0076 (11)	0.0040 (11)	0.0190 (12)
N6	0.0211 (13)	0.0354 (14)	0.0417 (13)	0.0080 (11)	0.0025 (10)	0.0158 (11)
N7	0.0222 (13)	0.0358 (14)	0.0374 (13)	0.0072 (10)	0.0041 (10)	0.0125 (11)
N8	0.0287 (14)	0.0420 (16)	0.0406 (14)	0.0074 (12)	0.0017 (11)	0.0158 (12)
C16	0.0278 (15)	0.0265 (15)	0.0320 (13)	0.0056 (12)	0.0052 (11)	0.0057 (11)
C17	0.0277 (16)	0.0405 (18)	0.0428 (16)	0.0045 (14)	0.0047 (13)	0.0158 (14)
C18	0.0344 (17)	0.0436 (19)	0.0416 (17)	0.0100 (15)	0.0010 (13)	0.0145 (14)
C19	0.0393 (18)	0.0355 (18)	0.0427 (17)	0.0065 (14)	0.0071 (14)	0.0143 (14)
C20	0.0290 (17)	0.0409 (19)	0.059 (2)	0.0014 (14)	0.0101 (15)	0.0172 (16)
C21	0.0236 (15)	0.0413 (19)	0.0486 (17)	0.0061 (13)	0.0055 (13)	0.0163 (14)
C22	0.0257 (15)	0.0344 (17)	0.0348 (14)	0.0044 (12)	0.0042 (12)	0.0097 (12)
C23	0.0220 (14)	0.0318 (16)	0.0329 (14)	0.0065 (12)	0.0036 (11)	0.0086 (12)
C24	0.0244 (15)	0.0311 (16)	0.0344 (14)	0.0073 (12)	0.0035 (11)	0.0094 (12)
C25	0.0226 (14)	0.0342 (16)	0.0329 (14)	0.0052 (12)	0.0041 (11)	0.0092 (12)
C26	0.0286 (15)	0.0298 (16)	0.0315 (14)	0.0066 (12)	0.0022 (11)	0.0067 (12)
C27	0.0257 (16)	0.0446 (19)	0.0466 (17)	0.0090 (14)	0.0061 (13)	0.0184 (15)
C28	0.0269 (17)	0.054 (2)	0.054 (2)	0.0039 (15)	0.0116 (14)	0.0197 (17)
C29	0.044 (2)	0.057 (2)	0.0507 (19)	0.0005 (18)	0.0082 (16)	0.0290 (18)
C30	0.0353 (18)	0.057 (2)	0.0472 (18)	0.0104 (16)	0.0016 (14)	0.0258 (17)

Geometric parameters (Å, º)

O1—C7	1.229 (4)	O3—C22	1.235 (4)
O2—C9	1.245 (4)	O4—C24	1.251 (4)
N1—C9	1.356 (4)	N5—C24	1.358 (4)
N1—N2	1.408 (4)	N5—N6	1.406 (3)
N1—H1A	0.91 (5)	N5—H5A	0.86 (4)
N2—C7	1.390 (4)	N6—C22	1.392 (4)
N2—C1	1.413 (4)	N6—C16	1.412 (4)
N3—C10	1.333 (4)	N7—C25	1.331 (4)
N3—C11	1.400 (4)	N7—C26	1.402 (4)
N3—H3A	0.96 (5)	N7—H7A	0.94 (4)
N4—C11	1.330 (4)	N8—C30	1.334 (4)

N4—C15	1.345 (4)	N8—C26	1.335 (4)
C1—C6	1.389 (5)	C16—C17	1.390 (4)
C1—C2	1.391 (5)	C16—C21	1.395 (4)
C2—C3	1.378 (5)	C17—C18	1.386 (5)
C2—H2	0.9400	C17—H17	0.9400
C3—C4	1.377 (6)	C18—C19	1.375 (5)
C3—H3	0.9400	C18—H18	0.9400
C4—C5	1.380 (5)	C19—C20	1.379 (5)
C4—H4	0.9400	C19—H19	0.9400
C5—C6	1.384 (5)	C20—C21	1.386 (5)
C5—H5	0.9400	C20—H20	0.9400
C6—H6	0.9400	C21—H21	0.9400
C7—C8	1.445 (4)	C22—C23	1.441 (4)
C8—C10	1.371 (4)	C23—C25	1.366 (4)
C8—C9	1.438 (4)	C23—C24	1.442 (4)
C10—H10	0.9400	C25—H25	0.9400
C11—C12	1.391 (5)	C26—C27	1.391 (4)
C12—C13	1.375 (5)	C27—C28	1.388 (5)
C12—H12	0.9400	C27—H27	0.9400
C13—C14	1.390 (5)	C28—C29	1.372 (5)
C13—H13	0.9400	C28—H28	0.9400
C14—C15	1.375 (5)	C29—C30	1.383 (5)
C14—H14	0.9400	C29—H29	0.9400
C15—H15	0.9400	C30—H30	0.9400

C9—N1—N2	109.8 (2)	C24—N5—N6	109.6 (2)
C9—N1—H1A	126 (3)	C24—N5—H5A	125 (3)
N2—N1—H1A	122 (3)	N6—N5—H5A	124 (3)
C7—N2—N1	109.7 (2)	C22—N6—N5	109.5 (2)
C7—N2—C1	129.7 (3)	C22—N6—C16	129.9 (3)
N1—N2—C1	120.2 (2)	N5—N6—C16	120.2 (2)
C10—N3—C11	128.0 (3)	C25—N7—C26	127.9 (3)
C10—N3—H3A	120 (3)	C25—N7—H7A	115 (2)
C11—N3—H3A	112 (3)	C26—N7—H7A	117 (2)
C11—N4—C15	116.3 (3)	C30—N8—C26	116.8 (3)
C6—C1—C2	119.5 (3)	C17—C16—C21	119.7 (3)
C6—C1—N2	120.1 (3)	C17—C16—N6	120.4 (3)
C2—C1—N2	120.4 (3)	C21—C16—N6	119.9 (3)
C3—C2—C1	119.7 (3)	C18—C17—C16	119.1 (3)
C3—C2—H2	120.2	C18—C17—H17	120.4
C1—C2—H2	120.2	C16—C17—H17	120.4
C4—C3—C2	121.3 (3)	C19—C18—C17	121.6 (3)
C4—C3—H3	119.4	C19—C18—H18	119.2
C2—C3—H3	119.4	C17—C18—H18	119.2
C3—C4—C5	118.9 (3)	C18—C19—C20	119.2 (3)
C3—C4—H4	120.6	C18—C19—H19	120.4
C5—C4—H4	120.6	C20—C19—H19	120.4
C4—C5—C6	120.9 (3)	C19—C20—C21	120.5 (3)

C4—C5—H5	119.5	C19—C20—H20	119.7
C6—C5—H5	119.5	C21—C20—H20	119.7
C5—C6—C1	119.8 (3)	C20—C21—C16	119.9 (3)
C5—C6—H6	120.1	C20—C21—H21	120.0
C1—C6—H6	120.1	C16—C21—H21	120.0
O1—C7—N2	124.5 (3)	O3—C22—N6	124.4 (3)
O1—C7—C8	130.4 (3)	O3—C22—C23	129.9 (3)
N2—C7—C8	105.1 (3)	N6—C22—C23	105.8 (3)
C10—C8—C9	124.9 (3)	C25—C23—C22	127.7 (3)
C10—C8—C7	126.7 (3)	C25—C23—C24	124.6 (3)
C9—C8—C7	108.4 (3)	C22—C23—C24	107.7 (3)
O2—C9—N1	124.4 (3)	O4—C24—N5	124.4 (3)
O2—C9—C8	128.8 (3)	O4—C24—C23	128.3 (3)
N1—C9—C8	106.8 (3)	N5—C24—C23	107.3 (3)
N3—C10—C8	121.0 (3)	N7—C25—C23	120.9 (3)
N3—C10—H10	119.5	N7—C25—H25	119.5
C8—C10—H10	119.5	C23—C25—H25	119.5
N4—C11—C12	124.3 (3)	N8—C26—C27	123.7 (3)
N4—C11—N3	113.2 (3)	N8—C26—N7	112.7 (3)
C12—C11—N3	122.5 (3)	C27—C26—N7	123.5 (3)
C13—C12—C11	117.7 (3)	C28—C27—C26	117.7 (3)
C13—C12—H12	121.1	C28—C27—H27	121.1
C11—C12—H12	121.1	C26—C27—H27	121.1
C12—C13—C14	119.7 (3)	C29—C28—C27	119.6 (3)
C12—C13—H13	120.2	C29—C28—H28	120.2
C14—C13—H13	120.2	C27—C28—H28	120.2
C15—C14—C13	117.7 (3)	C28—C29—C30	118.0 (3)
C15—C14—H14	121.2	C28—C29—H29	121.0
C13—C14—H14	121.2	C30—C29—H29	121.0
N4—C15—C14	124.4 (3)	N8—C30—C29	124.2 (3)
N4—C15—H15	117.8	N8—C30—H30	117.9
C14—C15—H15	117.8	C29—C30—H30	117.9

C9—N1—N2—C7	−5.5 (4)	C24—N5—N6—C22	−4.7 (3)
C9—N1—N2—C1	−179.2 (3)	C24—N5—N6—C16	−178.8 (3)
C7—N2—C1—C6	−161.3 (3)	C22—N6—C16—C17	12.5 (5)
N1—N2—C1—C6	11.0 (4)	N5—N6—C16—C17	−174.8 (3)
C7—N2—C1—C2	19.9 (5)	C22—N6—C16—C21	−167.4 (3)
N1—N2—C1—C2	−167.8 (3)	N5—N6—C16—C21	5.3 (4)
C6—C1—C2—C3	0.9 (5)	C21—C16—C17—C18	−0.6 (5)
N2—C1—C2—C3	179.7 (3)	N6—C16—C17—C18	179.5 (3)
C1—C2—C3—C4	−1.2 (6)	C16—C17—C18—C19	−0.4 (5)
C2—C3—C4—C5	0.9 (6)	C17—C18—C19—C20	0.9 (5)
C3—C4—C5—C6	−0.2 (5)	C18—C19—C20—C21	−0.3 (5)
C4—C5—C6—C1	−0.1 (5)	C19—C20—C21—C16	−0.7 (5)
C2—C1—C6—C5	−0.2 (5)	C17—C16—C21—C20	1.2 (5)
N2—C1—C6—C5	−179.0 (3)	N6—C16—C21—C20	−179.0 (3)
N1—N2—C7—O1	−175.1 (3)	N5—N6—C22—O3	−176.0 (3)

C1—N2—C7—O1	−2.2 (5)	C16—N6—C22—O3	−2.6 (5)
N1—N2—C7—C8	4.0 (3)	N5—N6—C22—C23	3.0 (3)
C1—N2—C7—C8	177.0 (3)	C16—N6—C22—C23	176.3 (3)
O1—C7—C8—C10	−3.6 (6)	O3—C22—C23—C25	−0.4 (6)
N2—C7—C8—C10	177.3 (3)	N6—C22—C23—C25	−179.3 (3)
O1—C7—C8—C9	177.7 (3)	O3—C22—C23—C24	178.5 (3)
N2—C7—C8—C9	−1.4 (3)	N6—C22—C23—C24	−0.3 (3)
N2—N1—C9—O2	−175.1 (3)	N6—N5—C24—O4	−175.7 (3)
N2—N1—C9—C8	4.4 (4)	N6—N5—C24—C23	4.3 (3)
C10—C8—C9—O2	−1.1 (6)	C25—C23—C24—O4	−3.5 (5)
C7—C8—C9—O2	177.7 (3)	C22—C23—C24—O4	177.5 (3)
C10—C8—C9—N1	179.4 (3)	C25—C23—C24—N5	176.5 (3)
C7—C8—C9—N1	−1.9 (4)	C22—C23—C24—N5	−2.5 (3)
C11—N3—C10—C8	179.3 (3)	C26—N7—C25—C23	−176.9 (3)
C9—C8—C10—N3	−0.9 (5)	C22—C23—C25—N7	179.7 (3)
C7—C8—C10—N3	−179.4 (3)	C24—C23—C25—N7	0.9 (5)
C15—N4—C11—C12	−0.6 (5)	C30—N8—C26—C27	−0.1 (5)
C15—N4—C11—N3	178.8 (3)	C30—N8—C26—N7	−180.0 (3)
C10—N3—C11—N4	179.6 (3)	C25—N7—C26—N8	179.9 (3)
C10—N3—C11—C12	−1.0 (5)	C25—N7—C26—C27	0.0 (5)
N4—C11—C12—C13	1.1 (5)	N8—C26—C27—C28	0.0 (5)
N3—C11—C12—C13	−178.2 (3)	N7—C26—C27—C28	179.8 (3)
C11—C12—C13—C14	−0.5 (5)	C26—C27—C28—C29	−0.2 (6)
C12—C13—C14—C15	−0.5 (5)	C27—C28—C29—C30	0.4 (6)
C11—N4—C15—C14	−0.5 (5)	C26—N8—C30—C29	0.4 (6)
C13—C14—C15—N4	1.1 (6)	C28—C29—C30—N8	−0.6 (7)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O1^{i}	0.91 (5)	1.97 (5)	2.869 (3)	168 (4)
N3—H3*A*···O2	0.96 (5)	2.10 (5)	2.812 (4)	130 (4)
C2—H2···O1	0.94	2.33	2.920 (4)	121
C6—H6···O1^{i}	0.94	2.57	3.401 (4)	147
C10—H10···O2^{ii}	0.94	2.29	3.222 (4)	170
C12—H12···O2^{ii}	0.94	2.24	3.175 (4)	170
N5—H5*A*···O3^{ii}	0.86 (4)	2.02 (4)	2.866 (4)	171 (4)
N7—H7*A*···O4	0.94 (4)	2.03 (4)	2.794 (3)	138 (3)
C17—H17···O3	0.94	2.27	2.895 (4)	124
C21—H21···O3^{ii}	0.94	2.53	3.392 (4)	153
C25—H25···O4^{i}	0.94	2.30	3.230 (4)	172
C27—H27···O4^{i}	0.94	2.29	3.222 (4)	173

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