Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(2*Z*)-1-(5-Hydroxy-3-methyl-1-phenyl-1*H*-pyrazol-4-yl)-3-(4-methoxyanilino)-but-2-en-1-one

Abdullah M. Asiri,[a,b]‡ Abdulrahman O. Al-Youbi,[a] Hassan M. Faidallah,[a] Seik Weng Ng[c,a] and Edward R. T. Tiekink[c]*

[a]Chemistry Department, Faculty of Science, King Abdulaziz University, PO Box 80203, Jeddah, Saudi Arabia, [b]The Center of Excellence for Advanced Materials Research, King Abdulaziz University, Jeddah, PO Box 80203, Saudi Arabia, and [c]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: edward.tiekink@gmail.com

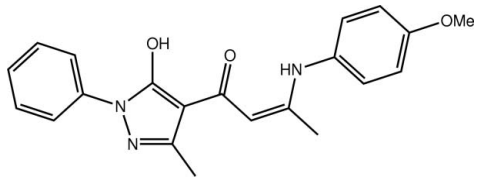
Received 8 August 2011; accepted 10 August 2011

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.042; wR factor = 0.104; data-to-parameter ratio = 15.4.

The central residue in the title compound, $C_{21}H_{21}N_3O_3$, is close to planar (r.m.s. deviation = 0.0753 Å for all non-H atoms from OH to NH inclusive): the hydroxy, amino and carbonyl groups all lie to the same side of the molecule (the conformation about the ethene bond is *Z*), facilitating the formation of intramolecular O—H···O and N—H···O hydrogen bonds that close *S*(6) rings. However, overall the molecule is twisted as the terminal aromatic rings are not coplanar with the central plane [dihedral angles = 20.55 (5) and 80.90 (4)° for the N-bound phenyl ring and the methoxybenzene ring, respectively]. The dihedral angle between the rings is 82.14 (7)°. Supramolecular layers in the *ac* plane mediated by C—H···π interactions are found in the crystal.

Related literature

For background to the synthesis, see: Gelin *et al.* (1983); Bendaas *et al.* (1999). For the structure of the 4-chloro derivative, see: Asiri *et al.* (2011).



Experimental

Crystal data

$C_{21}H_{21}N_3O_3$
M_r = 363.41
Monoclinic, $P2_1/n$
a = 9.5717 (3) Å
b = 16.9516 (6) Å
c = 11.3143 (4) Å
β = 104.946 (4)°
V = 1773.70 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 100 K
0.30 × 0.25 × 0.20 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
T_{min} = 0.837, T_{max} = 1.000
8486 measured reflections
3939 independent reflections
3145 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.104
S = 1.05
3939 reflections
255 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

*Cg*1 and *Cg*2 are the centroids of the N1,N2,C1–C3 and C15–C20 rings, respectively.

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2	0.86 (1)	1.68 (1)	2.4963 (15)	156 (2)
N3—H3···O2	0.89 (1)	1.92 (1)	2.6447 (16)	138 (2)
C14—H14b···*Cg*1^{i}	0.98	2.88	3.5542 (18)	127
C21—H21c···*Cg*2^{ii}	0.98	2.76	3.5195 (17)	134

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

Data collection: *CrysAlis PRO* (Agilent, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

The authors are thankful to the Center of Excellence for Advanced Materials Research and the Chemistry Department at King Abdulaziz University for providing research facilities. Dr Al-Amry is thanked for support. The authors also thank the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6355).

References

Agilent (2010). *CrysAlis PRO*. Agilent Technologies, Yarnton, England.
Asiri, A. M., Al-Youbi, A. O., Alamry, K. A., Faidallah, H. M., Ng, S. W. & Tiekink, E. R. T. (2011). *Acta Cryst.* E**67**, o2157.
Bendaas, A., Hamdi, M. & Sellier, N. (1999). *J. Heterocycl. Chem.* **36**, 1291–1294.
Brandenburg, K. (2006). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Gelin, S., Chantegrel, B. & Nadi, A. I. (1983). *J. Org. Chem.* **48**, 4078–4082.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

‡ Additional correspondence author, e-mail: aasiri2@kau.edu.sa.

supporting information

Acta Cryst. (2011). E**67**, o2353 [doi:10.1107/S1600536811032491]

(2*Z*)-1-(5-Hydroxy-3-methyl-1-phenyl-1*H*-pyrazol-4-yl)-3-(4-methoxyanilino)but-2-en-1-one

Abdullah M. Asiri, Abdulrahman O. Al-Youbi, Hassan M. Faidallah, Seik Weng Ng and Edward R. T. Tiekink

S1. Comment

In connection with a recent structural study (Asiri *et al.*, 2011), the title compound (I) was prepared as a part of on-going investigations of reactions between pyrazoles and aniline derivatives (Gelin *et al.*, 1983; Bendaas *et al.*, 1999).

The molecular structure of (I), Fig. 1, resembles closely that of the 4-chloroanilino derivative (Asiri *et al.*, 2011) and features a *Z* configuration about the C12—C13 [1.381 (2) Å] bond. The hydroxy and amino groups are *syn* to the central carbonyl group and each forms a hydrogen bond to close a *S*(6) ring (Table 1). A direct consequence of this is that the central residue is planar; the values of the C1—C2—C11—O2, C2—C11—C12—C13 and C11—C12—C13—N3 torsion angles are -3.6 (2), -171.74 (15) and -1.7 (2) °, respectively. The benzene and 4-methoxybenzene rings are each twisted out of the central plane as seen in the values of the C1—N1—C5—C6 and C13—N3—C15—C16 torsion angles of 159.97 (15) and -74.5 (2) °, respectively.

The most prominent feature of the crystal packing is the formation of supramolecular layers in the *ac* plane and mediated by C—H···π interactions, Fig. 2. and Table 1. Layers stack along the *b* axis as shown in Fig. 3.

S2. Experimental

A solution of 4-acetoacetyl-5-hydroxy-3-methyl-1-*p*-sulfamylphenypyrazole (1.7 g, 0.005 mole) and 4-methoxyaniline (0.63 g, 0.005 mole) in ethanol (25 ml) was refluxed for 2 h. The precipitate, obtained from the hot solution, was collected, washed with methanol and recrystallized from ethanol-benzene as yellow blocks; *M*.pt: 507–507 K.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions [C—H 0.95 to 0.98 Å, U_{iso}(H) 1.2 to 1.5U_{eq}(C)] and were included in the refinement in the riding model approximation. The hydroxyl- and amino- H-atoms were located in a difference Fourier map, and subsequently refined freely.

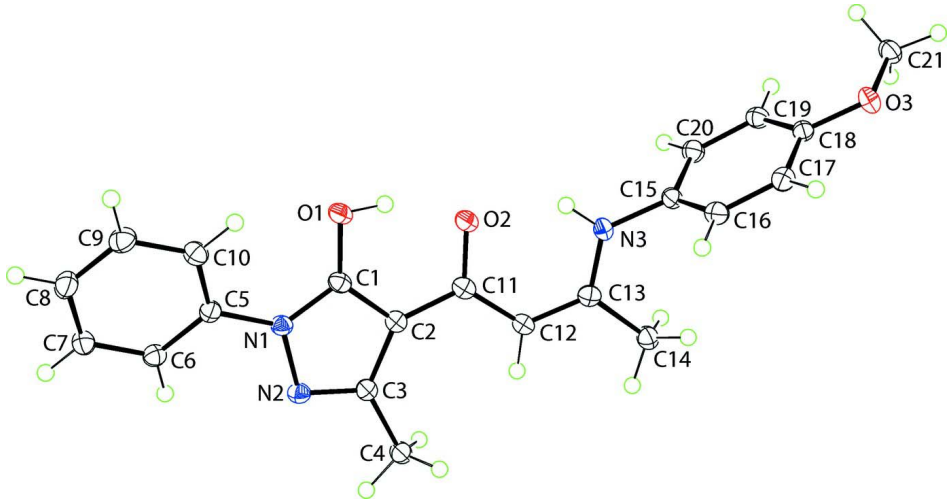


Figure 1

The molecular structure of (I) showing displacement ellipsoids at the 50% probability level.

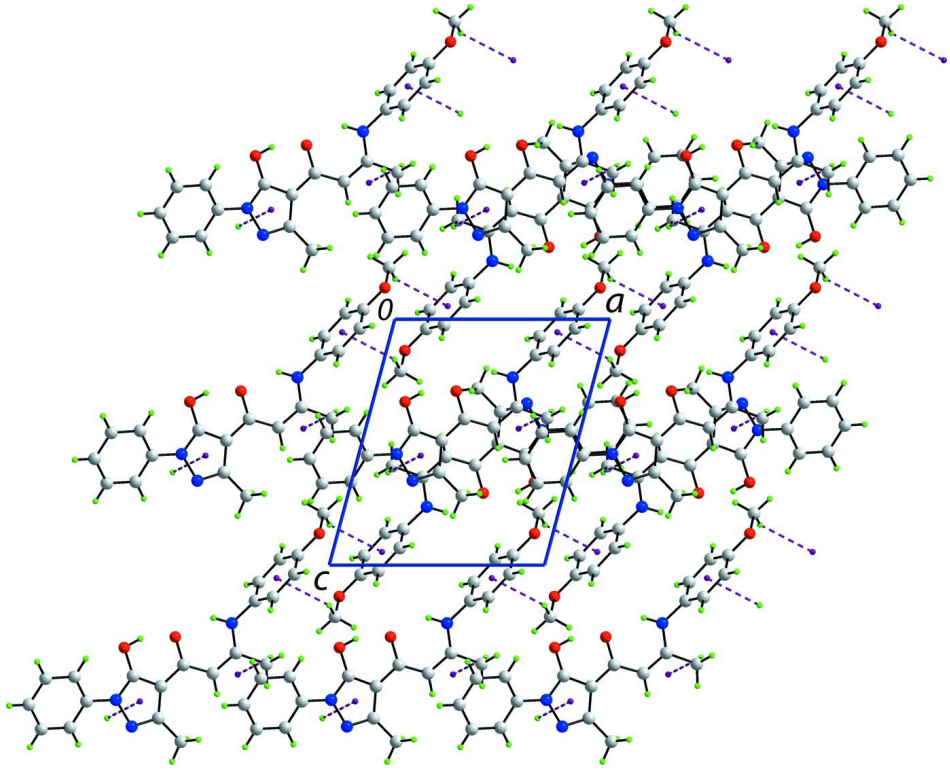


Figure 2

A view of a supramolecular layer in (I) mediated by C—H···π interactions, shown as purple dashed lines

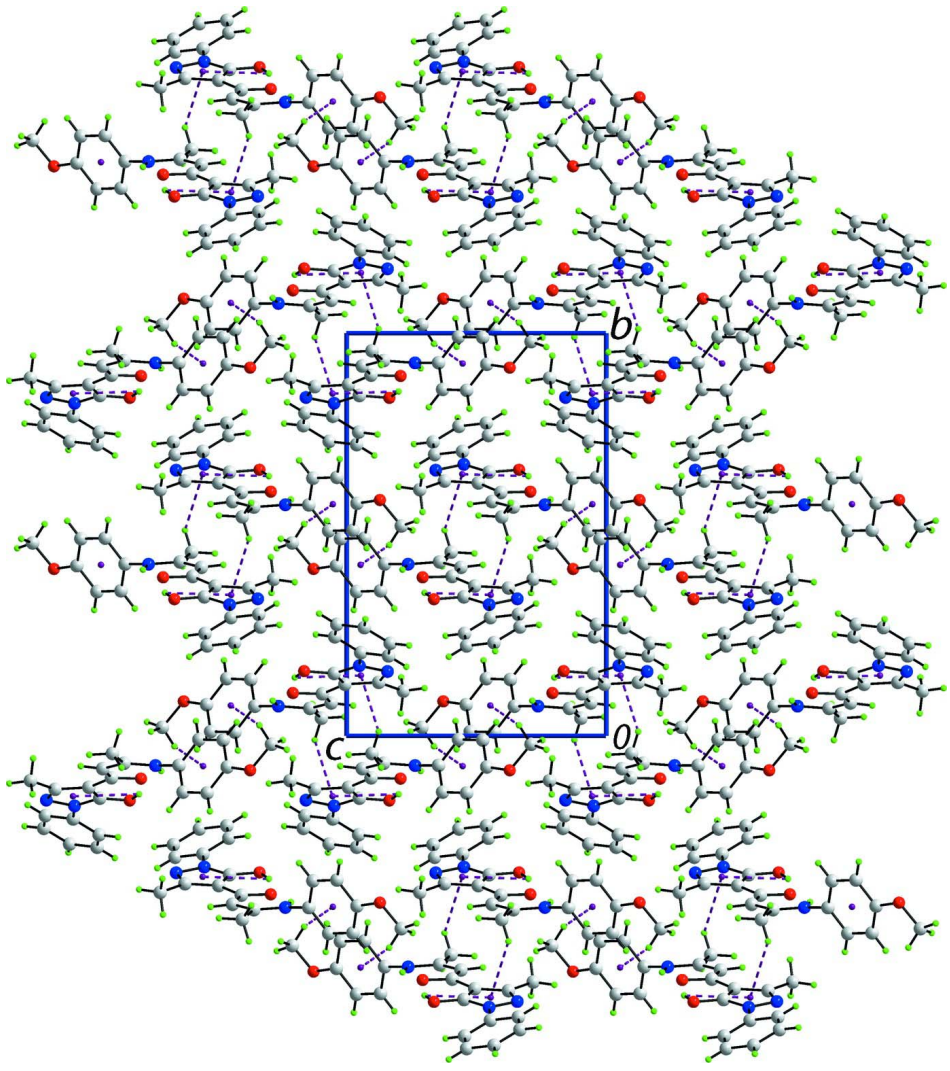


Figure 3

Stacking of supramolecular layers along the *b* axis in (I). The C—H···π interactions are shown as purple dashed lines.

(2*Z*)-1-(5-Hydroxy-3-methyl-1-phenyl-1*H*-pyrazol-4-yl)- 3-(4-methoxyanilino)but-2-en-1-one

Crystal data

$C_{21}H_{21}N_3O_3$
M_r = 363.41
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 9.5717 (3) Å
b = 16.9516 (6) Å
c = 11.3143 (4) Å
β = 104.946 (4)°
V = 1773.70 (10) Å^3
Z = 4
$F(000)$ = 768
D_x = 1.361 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3917 reflections
θ = 2.4–29.3°
μ = 0.09 mm^{-1}
T = 100 K
Block, yellow
0.30 × 0.25 × 0.20 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Radiation source: SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.4041 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
T_{min} = 0.837, T_{max} = 1.000
8486 measured reflections
3939 independent reflections
3145 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 27.5°, θ_{min} = 2.4°
h = −12→9
k = −17→21
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.104
S = 1.05
3939 reflections
255 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0364P)^2 + 0.8331P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 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.

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

	x	y	z	U_{iso}*/U_{eq}
O1	0.14980 (12)	0.66071 (7)	0.33592 (9)	0.0222 (3)
O2	0.37382 (11)	0.60631 (7)	0.29255 (9)	0.0213 (3)
O3	0.91460 (11)	0.58562 (7)	−0.12793 (9)	0.0219 (3)
N1	0.17619 (13)	0.67412 (8)	0.54868 (11)	0.0166 (3)
N2	0.28248 (13)	0.65978 (8)	0.65708 (11)	0.0175 (3)
N3	0.61937 (14)	0.57378 (8)	0.23667 (11)	0.0195 (3)
C1	0.22616 (16)	0.65491 (9)	0.45138 (13)	0.0170 (3)
C2	0.36749 (16)	0.62804 (9)	0.49321 (13)	0.0169 (3)
C3	0.39577 (15)	0.63279 (9)	0.62323 (13)	0.0162 (3)
C4	0.52961 (16)	0.61127 (10)	0.71833 (13)	0.0196 (3)
H4A	0.5134	0.6184	0.7997	0.029*
H4B	0.6094	0.6452	0.7101	0.029*
H4C	0.5540	0.5560	0.7078	0.029*
C5	0.04095 (15)	0.70640 (9)	0.55398 (13)	0.0166 (3)
C6	−0.00329 (16)	0.69838 (9)	0.66119 (13)	0.0187 (3)

H6	0.0562	0.6716	0.7296	0.022*
C7	−0.13461 (16)	0.72972 (10)	0.66734 (14)	0.0217 (3)
H7	−0.1651	0.7245	0.7405	0.026*
C8	−0.22213 (17)	0.76870 (10)	0.56770 (15)	0.0247 (4)
H8	−0.3126	0.7898	0.5722	0.030*
C9	−0.17707 (17)	0.77671 (10)	0.46184 (14)	0.0235 (4)
H9	−0.2367	0.8036	0.3936	0.028*
C10	−0.04527 (17)	0.74579 (10)	0.45410 (14)	0.0205 (3)
H10	−0.0146	0.7516	0.3811	0.025*
C11	0.44652 (16)	0.60431 (9)	0.40619 (13)	0.0176 (3)
C12	0.59321 (16)	0.58162 (9)	0.44007 (13)	0.0175 (3)
H12	0.6385	0.5752	0.5247	0.021*
C13	0.67514 (16)	0.56811 (9)	0.35792 (13)	0.0176 (3)
C14	0.83048 (16)	0.54384 (10)	0.40247 (14)	0.0211 (3)
H14A	0.8907	0.5790	0.3677	0.032*
H14B	0.8417	0.4895	0.3769	0.032*
H14C	0.8606	0.5471	0.4919	0.032*
C15	0.70003 (15)	0.57341 (10)	0.14592 (13)	0.0179 (3)
C16	0.77859 (16)	0.63978 (10)	0.13037 (13)	0.0193 (3)
H16	0.7840	0.6838	0.1834	0.023*
C17	0.84893 (16)	0.64190 (10)	0.03781 (13)	0.0190 (3)
H17	0.9022	0.6874	0.0271	0.023*
C18	0.84160 (15)	0.57738 (9)	−0.03966 (13)	0.0172 (3)
C19	0.76391 (16)	0.51053 (10)	−0.02410 (13)	0.0193 (3)
H19	0.7589	0.4663	−0.0766	0.023*
C20	0.69349 (16)	0.50912 (10)	0.06938 (13)	0.0196 (3)
H20	0.6405	0.4636	0.0807	0.023*
C21	0.90919 (17)	0.52107 (10)	−0.21023 (13)	0.0223 (3)
H21A	0.9647	0.5343	−0.2692	0.033*
H21B	0.8084	0.5106	−0.2540	0.033*
H21C	0.9507	0.4740	−0.1640	0.033*
H1	0.214 (2)	0.6449 (15)	0.300 (2)	0.067 (8)*
H3	0.5266 (11)	0.5872 (11)	0.2149 (16)	0.030 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0225 (6)	0.0292 (7)	0.0142 (5)	0.0037 (5)	0.0039 (4)	−0.0013 (5)
O2	0.0214 (5)	0.0273 (6)	0.0154 (5)	0.0025 (5)	0.0049 (4)	−0.0012 (4)
O3	0.0259 (6)	0.0232 (6)	0.0198 (5)	−0.0003 (5)	0.0119 (5)	0.0004 (4)
N1	0.0172 (6)	0.0178 (7)	0.0141 (6)	0.0020 (5)	0.0027 (5)	0.0006 (5)
N2	0.0187 (6)	0.0179 (7)	0.0149 (6)	0.0010 (5)	0.0024 (5)	0.0007 (5)
N3	0.0178 (6)	0.0250 (8)	0.0171 (6)	0.0018 (6)	0.0070 (5)	−0.0006 (5)
C1	0.0207 (7)	0.0155 (8)	0.0150 (7)	−0.0020 (6)	0.0048 (6)	−0.0008 (6)
C2	0.0185 (7)	0.0158 (8)	0.0166 (7)	−0.0001 (6)	0.0050 (6)	0.0000 (6)
C3	0.0181 (7)	0.0138 (7)	0.0172 (7)	−0.0021 (6)	0.0055 (6)	−0.0004 (6)
C4	0.0204 (7)	0.0215 (8)	0.0172 (7)	0.0024 (6)	0.0053 (6)	−0.0002 (6)
C5	0.0159 (7)	0.0145 (8)	0.0193 (7)	−0.0009 (6)	0.0042 (6)	−0.0029 (6)

C6	0.0183 (7)	0.0189 (8)	0.0184 (7)	−0.0013 (6)	0.0036 (6)	−0.0002 (6)
C7	0.0201 (7)	0.0254 (9)	0.0208 (7)	−0.0020 (7)	0.0075 (6)	−0.0022 (6)
C8	0.0181 (7)	0.0273 (9)	0.0288 (8)	0.0025 (7)	0.0060 (7)	−0.0032 (7)
C9	0.0217 (8)	0.0232 (9)	0.0234 (8)	0.0040 (7)	0.0018 (6)	0.0020 (6)
C10	0.0230 (8)	0.0205 (8)	0.0183 (7)	0.0006 (6)	0.0060 (6)	0.0010 (6)
C11	0.0225 (7)	0.0137 (8)	0.0169 (7)	−0.0013 (6)	0.0056 (6)	−0.0003 (6)
C12	0.0205 (7)	0.0178 (8)	0.0145 (7)	0.0001 (6)	0.0051 (6)	−0.0005 (6)
C13	0.0204 (7)	0.0142 (8)	0.0178 (7)	−0.0013 (6)	0.0040 (6)	−0.0009 (6)
C14	0.0210 (7)	0.0231 (9)	0.0202 (7)	0.0018 (6)	0.0072 (6)	−0.0007 (6)
C15	0.0168 (7)	0.0225 (9)	0.0149 (7)	0.0027 (6)	0.0046 (6)	0.0013 (6)
C16	0.0205 (7)	0.0180 (8)	0.0188 (7)	0.0039 (6)	0.0039 (6)	−0.0011 (6)
C17	0.0189 (7)	0.0174 (8)	0.0205 (7)	0.0011 (6)	0.0045 (6)	0.0034 (6)
C18	0.0151 (7)	0.0215 (8)	0.0145 (7)	0.0041 (6)	0.0030 (6)	0.0041 (6)
C19	0.0210 (7)	0.0194 (8)	0.0170 (7)	0.0009 (6)	0.0039 (6)	−0.0022 (6)
C20	0.0202 (7)	0.0197 (8)	0.0191 (7)	−0.0020 (6)	0.0056 (6)	0.0001 (6)
C21	0.0235 (8)	0.0273 (9)	0.0178 (7)	0.0040 (7)	0.0083 (6)	−0.0011 (6)

Geometric parameters (Å, º)

O1—C1	1.3259 (17)	C8—C9	1.380 (2)
O1—H1	0.864 (10)	C8—H8	0.9500
O2—C11	1.2950 (17)	C9—C10	1.390 (2)
O3—C18	1.3657 (17)	C9—H9	0.9500
O3—C21	1.4291 (19)	C10—H10	0.9500
N1—C1	1.3484 (19)	C11—C12	1.410 (2)
N1—N2	1.3981 (16)	C12—C13	1.381 (2)
N1—C5	1.4208 (19)	C12—H12	0.9500
N2—C3	1.3213 (19)	C13—C14	1.499 (2)
N3—C13	1.3410 (19)	C14—H14A	0.9800
N3—C15	1.4350 (19)	C14—H14B	0.9800
N3—H3	0.888 (9)	C14—H14C	0.9800
C1—C2	1.390 (2)	C15—C20	1.383 (2)
C2—C3	1.4277 (19)	C15—C16	1.389 (2)
C2—C11	1.445 (2)	C16—C17	1.384 (2)
C3—C4	1.490 (2)	C16—H16	0.9500
C4—H4A	0.9800	C17—C18	1.392 (2)
C4—H4B	0.9800	C17—H17	0.9500
C4—H4C	0.9800	C18—C19	1.391 (2)
C5—C10	1.387 (2)	C19—C20	1.393 (2)
C5—C6	1.391 (2)	C19—H19	0.9500
C6—C7	1.383 (2)	C20—H20	0.9500
C6—H6	0.9500	C21—H21A	0.9800
C7—C8	1.386 (2)	C21—H21B	0.9800
C7—H7	0.9500	C21—H21C	0.9800
C1—O1—H1	99.0 (16)	C9—C10—H10	120.3
C18—O3—C21	117.30 (12)	O2—C11—C12	121.30 (13)
C1—N1—N2	110.07 (12)	O2—C11—C2	115.32 (13)

C1—N1—C5	130.27 (12)	C12—C11—C2	123.38 (13)
N2—N1—C5	119.65 (11)	C13—C12—C11	124.10 (13)
C3—N2—N1	105.77 (11)	C13—C12—H12	118.0
C13—N3—C15	125.88 (13)	C11—C12—H12	118.0
C13—N3—H3	114.1 (12)	N3—C13—C12	122.10 (13)
C15—N3—H3	119.1 (12)	N3—C13—C14	117.52 (13)
O1—C1—N1	124.40 (13)	C12—C13—C14	120.35 (13)
O1—C1—C2	126.92 (14)	C13—C14—H14A	109.5
N1—C1—C2	108.68 (12)	C13—C14—H14B	109.5
C1—C2—C3	103.98 (13)	H14A—C14—H14B	109.5
C1—C2—C11	119.59 (13)	C13—C14—H14C	109.5
C3—C2—C11	136.42 (14)	H14A—C14—H14C	109.5
N2—C3—C2	111.50 (13)	H14B—C14—H14C	109.5
N2—C3—C4	119.49 (12)	C20—C15—C16	119.82 (14)
C2—C3—C4	129.00 (13)	C20—C15—N3	120.37 (14)
C3—C4—H4A	109.5	C16—C15—N3	119.71 (14)
C3—C4—H4B	109.5	C17—C16—C15	120.12 (15)
H4A—C4—H4B	109.5	C17—C16—H16	119.9
C3—C4—H4C	109.5	C15—C16—H16	119.9
H4A—C4—H4C	109.5	C16—C17—C18	120.05 (15)
H4B—C4—H4C	109.5	C16—C17—H17	120.0
C10—C5—C6	120.45 (14)	C18—C17—H17	120.0
C10—C5—N1	120.55 (13)	O3—C18—C19	124.54 (14)
C6—C5—N1	119.00 (13)	O3—C18—C17	115.33 (14)
C7—C6—C5	119.44 (14)	C19—C18—C17	120.13 (14)
C7—C6—H6	120.3	C18—C19—C20	119.28 (14)
C5—C6—H6	120.3	C18—C19—H19	120.4
C6—C7—C8	120.57 (14)	C20—C19—H19	120.4
C6—C7—H7	119.7	C15—C20—C19	120.60 (15)
C8—C7—H7	119.7	C15—C20—H20	119.7
C9—C8—C7	119.60 (15)	C19—C20—H20	119.7
C9—C8—H8	120.2	O3—C21—H21A	109.5
C7—C8—H8	120.2	O3—C21—H21B	109.5
C8—C9—C10	120.63 (15)	H21A—C21—H21B	109.5
C8—C9—H9	119.7	O3—C21—H21C	109.5
C10—C9—H9	119.7	H21A—C21—H21C	109.5
C5—C10—C9	119.30 (14)	H21B—C21—H21C	109.5
C5—C10—H10	120.3		

C1—N1—N2—C3	0.61 (16)	N1—C5—C10—C9	179.96 (14)
C5—N1—N2—C3	−177.93 (13)	C8—C9—C10—C5	0.2 (2)
N2—N1—C1—O1	178.89 (14)	C1—C2—C11—O2	−3.6 (2)
C5—N1—C1—O1	−2.8 (3)	C3—C2—C11—O2	176.96 (17)
N2—N1—C1—C2	−0.54 (17)	C1—C2—C11—C12	175.40 (15)
C5—N1—C1—C2	177.79 (15)	C3—C2—C11—C12	−4.1 (3)
O1—C1—C2—C3	−179.15 (15)	O2—C11—C12—C13	7.2 (2)
N1—C1—C2—C3	0.26 (17)	C2—C11—C12—C13	−171.74 (15)
O1—C1—C2—C11	1.2 (2)	C15—N3—C13—C12	169.33 (15)

N1—C1—C2—C11	−179.38 (13)	C15—N3—C13—C14	−12.5 (2)
N1—N2—C3—C2	−0.44 (17)	C11—C12—C13—N3	−1.7 (2)
N1—N2—C3—C4	−179.84 (13)	C11—C12—C13—C14	−179.77 (15)
C1—C2—C3—N2	0.12 (18)	C13—N3—C15—C20	109.16 (18)
C11—C2—C3—N2	179.67 (17)	C13—N3—C15—C16	−74.5 (2)
C1—C2—C3—C4	179.46 (15)	C20—C15—C16—C17	0.7 (2)
C11—C2—C3—C4	−1.0 (3)	N3—C15—C16—C17	−175.75 (13)
C1—N1—C5—C10	−20.5 (2)	C15—C16—C17—C18	−0.3 (2)
N2—N1—C5—C10	157.74 (14)	C21—O3—C18—C19	0.5 (2)
C1—N1—C5—C6	159.97 (15)	C21—O3—C18—C17	−179.53 (13)
N2—N1—C5—C6	−21.8 (2)	C16—C17—C18—O3	179.91 (13)
C10—C5—C6—C7	0.3 (2)	C16—C17—C18—C19	−0.2 (2)
N1—C5—C6—C7	179.88 (14)	O3—C18—C19—C20	−179.88 (13)
C5—C6—C7—C8	0.2 (2)	C17—C18—C19—C20	0.2 (2)
C6—C7—C8—C9	−0.5 (3)	C16—C15—C20—C19	−0.6 (2)
C7—C8—C9—C10	0.3 (3)	N3—C15—C20—C19	175.76 (13)
C6—C5—C10—C9	−0.5 (2)	C18—C19—C20—C15	0.2 (2)

Hydrogen-bond geometry (Å, º)

Cg1 and Cg2 are the centroids of the N1,N2,C1–C3 and C15–C20 rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2	0.86 (1)	1.68 (1)	2.4963 (15)	156 (2)
N3—H3···O2	0.89 (1)	1.92 (1)	2.6447 (16)	138 (2)
C14—H14b···*Cg*1^{i}	0.98	2.88	3.5542 (18)	127
C21—H21c···*Cg*2^{ii}	0.98	2.76	3.5195 (17)	134

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