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Crystal structure of 5-(4-methoxyphenyl)-3-(4-methylphenyl)-4,5-dihydro-1*H*-pyrazole-1-carbaldehyde

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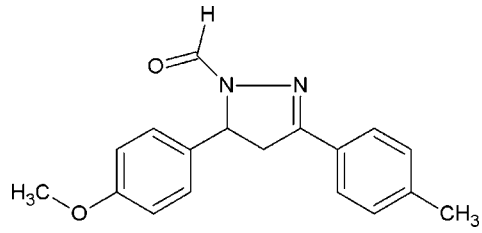
In the title compound, $C_{18}H_{18}N_2O_2$, the pyrazole ring has a twisted conformation on the CH—CH_2 bond. The tolyl ring and the 4-methoxyphenyl ring are inclined to the mean plane of the pyrazole ring by 4.40 (9) and 86.22 (9)°, respectively, while the two aromatic rings are inclined to one another by 88.75 (9)°. In the crystal, molecules are linked *via* bifurcated C—H···(O,O) hydrogen bonds and C—H···π interactions, forming sheets lying parallel to the *ab* plane.

Keywords: crystal structure; pyrazole; hydrogen bonding.

CCDC reference: 1441491

1. Related literature

For examples of the numerous pharmacological activities of pyrazoles, see: Samshuddin *et al.* (2012); Sarojini *et al.* (2010). For the use of 1,3,5-triaryl-2-pyrazolines as scintillation solutes, see: Wiley *et al.* (1958); and as fluorescent agents, see: Lu *et al.* (1999). For the crystal structures of pyrazoline-derived chalcones, see: Jasinski *et al.* (2012); Baktır *et al.* (2011).



2. Experimental

2.1. Crystal data

$C_{18}H_{18}N_2O_2$
M_r = 294.34
Monoclinic, *Cc*
a = 12.0839 (9) Å
b = 6.4197 (5) Å
c = 19.7427 (18) Å
β = 104.8264 (12)°
V = 1480.5 (2) Å^3
Z = 4
Mo *K*α radiation
μ = 0.09 mm^{-1}
T = 100 K
0.41 × 0.23 × 0.11 mm

2.2. Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2010)
T_{min} = 0.919, T_{max} = 0.965
14663 measured reflections
4301 independent reflections
4070 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.097
S = 1.05
4301 reflections
201 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

*Cg*2 is the centroid of the C1–C6 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*A*···O2^{i}	0.95	2.39	3.207 (2)	144
C14—H14*A*···O2^{ii}	0.95	2.57	3.477 (2)	160
C15—H15*A*···*Cg*2^{iii}	0.95	2.78	3.661 (2)	154

Symmetry codes: (i) $x, y+1, z$; (ii) $x+\frac{1}{2}, y+\frac{3}{2}, z$; (iii) $x+\frac{1}{2}, y+\frac{1}{2}, z$.

Data collection: *APEX2* (Bruker, 2010); cell refinement: *SAINT* (Bruker, 2010); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *SHELXTL* (Sheldrick 2008); software used to prepare material for publication: *SHELXTL*.

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: SU5259).

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supporting information

Acta Cryst. (2015). E**71**, o1093–o1094 [https://doi.org/10.1107/S2056989015023658]

Crystal structure of 5-(4-methoxyphenyl)-3-(4-methylphenyl)-4,5-dihydro-1*H*-pyrazole-1-carbaldehyde

Farook Adam, Seranthimata Samshuddin, Shruthi, Badiadka Narayana and Nadiah Ameram

S1. Comment

Pyrazoline derivatives exhibit numerous pharmacological activities including antioxidant, antiamoebic, anti-inflammatory, analgesic, antimicrobial, antidepressant and anticancer activities (Sarojini *et al.*, 2010; Samshuddin *et al.*, 2012). Many 1,3,5-triaryl-2-pyrazolines have also been used as scintillation solutes (Wiley *et al.*, 1958) and as fluorescent agents (Lu *et al.*, 1999).

The crystal structures of some pyrazolines containing an *N*-alkyl chain, viz. 3,5-bis(4-fluorophenyl)-4,5-dihydro-1*H*-pyrazole-1 carbaldehyde (Baktır *et al.*, 2011), 3,5-bis(4-fluorophenyl)-4,5-dihydro- 1*H*-pyrazole-1-carboxamide and 3,5-bis(4-fluorophenyl)-4,5-dihydro- 1*H*-pyrazole-1-carbothioamide (Jasinski *et al.*, 2012) have been reported. In view of the importance of pyrazolines, the title compound was synthesized and we report herein on its crystal structure.

The molecular structure of the title compound is illustrated in Fig. 1. The pyrazole ring has a twisted conformation on bond C7—C8. The toluyl ring and the 4-methoxyphenyl ring are inclined to the mean plane of the pyrazole ring by 4.40 (9) and 86.22 (9) °, respectively. The two aromatic rings are inclined to one another by 88.75 (9) °.

In the crystal, molecules are linked via C—H···O hydrogen bonds and C—H···π interactions forming sheets lying parallel to the *ab* plane (Table 1 and Fig. 2).

S2. Synthesis and crystallization

A mixture of (2*E*)-3-(4-methoxyphenyl)-1-(4-methylphenyl)prop-2-en-1-one (2.52 g, 0.01 mol) and hydrazine hydrate (1 ml) in 30 ml formic acid was refluxed for 6 h. The reaction mixture was cooled and poured into 50 ml ice-cold water. The precipitate was collected by filtration and purified by recrystallization from ethanol. Single crystals were grown from toluene by slow evaporation of the solvent (yield: 75 %; m.p. 479-482 K).

S3. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The hydrogen atoms were fixed geometrically (C—H = 0.95–1.00 Å) and allowed to ride on their parent atoms with U_{iso}(H) = 1.5U_{eq}(*C*-methyl) and 1.2U_{eq}(C) for other H atoms.

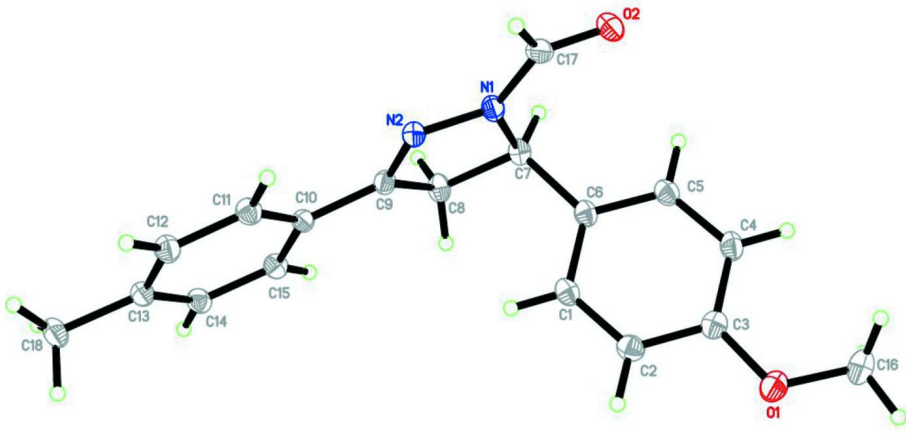

Figure 1

A view of the molecular structure of the title compound, with atom labelling. Displacement ellipsoids are drawn at the 50 % probability level.

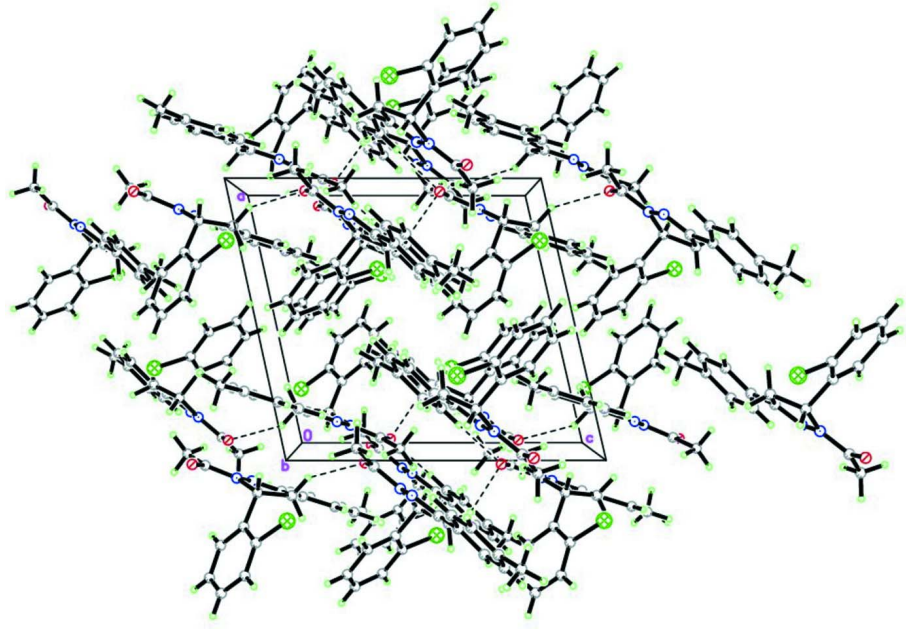

Figure 2

A view along the *ab* axis of the crystal packing of the title compound. Hydrogen bonds are shown as dashed lines (see Table 1).

5-(4-Methoxyphenyl)-3-(4-methylphenyl)-4,5-dihydro-1*H*-pyrazole-1-carbaldehyde

Crystal data

$C_{18}H_{18}N_2O_2$
M_r = 294.34
Monoclinic, *Cc*
a = 12.0839 (9) Å
b = 6.4197 (5) Å
c = 19.7427 (18) Å
β = 104.8264 (12)°
V = 1480.5 (2) Å^3
Z = 4
F(000) = 624
D_x = 1.321 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 6705 reflections
θ = 3.5–30.2°

μ = 0.09 mm^{-1}
T = 100 K

Block, colourless
0.41 × 0.23 × 0.11 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2010)
T_{min} = 0.919, T_{max} = 0.965
14663 measured reflections

4301 independent reflections
4070 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 30.2°, θ_{min} = 2.1°
h = −17→17
k = −9→9
l = −27→27

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.097
S = 1.05
4301 reflections
201 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0604P)^2 + 0.3156P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 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.

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

	x	y	z	U_{iso}*/U_{eq}
O1	0.20348 (12)	0.4331 (2)	0.35129 (8)	0.0248 (3)
O2	0.54286 (12)	−0.1313 (2)	0.56436 (8)	0.0229 (3)
N1	0.64240 (13)	0.1726 (2)	0.58103 (8)	0.0167 (3)
N2	0.68881 (12)	0.3318 (2)	0.62747 (8)	0.0164 (3)
C1	0.49214 (15)	0.4726 (3)	0.46209 (9)	0.0179 (3)
H1A	0.5393	0.5782	0.4884	0.022*
C2	0.38193 (16)	0.5207 (3)	0.42425 (9)	0.0189 (3)
H2A	0.3543	0.6591	0.4246	0.023*
C3	0.31110 (15)	0.3672 (3)	0.38556 (9)	0.0186 (3)
C4	0.35270 (16)	0.1650 (3)	0.38360 (9)	0.0199 (4)
H4A	0.3059	0.0603	0.3566	0.024*
C5	0.46443 (15)	0.1188 (3)	0.42203 (9)	0.0187 (3)
H5A	0.4930	−0.0187	0.4208	0.022*
C6	0.53452 (15)	0.2698 (3)	0.46190 (9)	0.0168 (3)
C7	0.65113 (15)	0.2100 (3)	0.50826 (9)	0.0171 (3)
H7A	0.6815	0.0838	0.4894	0.021*
C8	0.74108 (15)	0.3870 (3)	0.52205 (10)	0.0191 (3)
H8A	0.8174	0.3345	0.5204	0.023*

H8B	0.7182	0.5012	0.4876	0.023*
C9	0.74102 (14)	0.4580 (3)	0.59498 (9)	0.0153 (3)
C10	0.79896 (14)	0.6448 (2)	0.62925 (9)	0.0151 (3)
C11	0.79596 (15)	0.6957 (3)	0.69768 (9)	0.0178 (3)
H11A	0.7553	0.6091	0.7220	0.021*
C12	0.85202 (15)	0.8719 (3)	0.73026 (10)	0.0191 (3)
H12A	0.8487	0.9049	0.7766	0.023*
C13	0.91324 (14)	1.0015 (3)	0.69604 (10)	0.0181 (3)
C14	0.91505 (14)	0.9520 (3)	0.62759 (9)	0.0180 (3)
H14A	0.9554	1.0394	0.6033	0.022*
C15	0.85852 (14)	0.7760 (3)	0.59414 (9)	0.0171 (3)
H15A	0.8604	0.7451	0.5474	0.020*
C16	0.12863 (18)	0.2852 (4)	0.30873 (12)	0.0300 (4)
H16A	0.0551	0.3519	0.2871	0.045*
H16B	0.1163	0.1682	0.3378	0.045*
H16C	0.1631	0.2340	0.2720	0.045*
C17	0.58798 (15)	0.0120 (3)	0.60245 (10)	0.0191 (3)
H17A	0.5844	0.0099	0.6500	0.023*
C18	0.97566 (17)	1.1908 (3)	0.73207 (11)	0.0236 (4)
H18A	1.0484	1.2064	0.7192	0.035*
H18B	0.9906	1.1740	0.7829	0.035*
H18C	0.9285	1.3151	0.7174	0.035*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0198 (6)	0.0266 (7)	0.0257 (7)	0.0009 (5)	0.0014 (5)	−0.0011 (5)
O2	0.0226 (6)	0.0158 (6)	0.0292 (7)	−0.0031 (5)	0.0044 (5)	−0.0006 (5)
N1	0.0171 (7)	0.0156 (6)	0.0168 (7)	−0.0029 (5)	0.0032 (5)	−0.0016 (5)
N2	0.0152 (6)	0.0153 (6)	0.0171 (7)	−0.0010 (5)	0.0015 (5)	−0.0010 (5)
C1	0.0205 (8)	0.0170 (7)	0.0171 (8)	−0.0034 (6)	0.0063 (6)	−0.0027 (6)
C2	0.0227 (8)	0.0171 (7)	0.0177 (8)	0.0006 (6)	0.0068 (6)	−0.0008 (6)
C3	0.0182 (8)	0.0226 (8)	0.0154 (7)	−0.0009 (6)	0.0050 (6)	0.0007 (6)
C4	0.0205 (9)	0.0202 (8)	0.0177 (8)	−0.0050 (6)	0.0028 (7)	−0.0028 (6)
C5	0.0213 (8)	0.0164 (7)	0.0185 (8)	−0.0016 (6)	0.0051 (7)	−0.0024 (6)
C6	0.0183 (8)	0.0171 (7)	0.0154 (8)	−0.0030 (6)	0.0051 (6)	−0.0022 (6)
C7	0.0158 (7)	0.0180 (8)	0.0176 (8)	−0.0022 (6)	0.0045 (6)	−0.0028 (6)
C8	0.0166 (7)	0.0212 (8)	0.0202 (8)	−0.0049 (6)	0.0063 (6)	−0.0034 (6)
C9	0.0131 (7)	0.0156 (7)	0.0163 (8)	−0.0002 (6)	0.0022 (6)	−0.0016 (6)
C10	0.0120 (7)	0.0139 (7)	0.0182 (8)	0.0003 (5)	0.0019 (6)	−0.0005 (6)
C11	0.0187 (8)	0.0173 (7)	0.0180 (8)	−0.0017 (6)	0.0058 (6)	0.0006 (6)
C12	0.0193 (8)	0.0186 (8)	0.0189 (8)	−0.0005 (6)	0.0040 (7)	−0.0018 (6)
C13	0.0163 (8)	0.0148 (7)	0.0216 (8)	−0.0001 (6)	0.0017 (6)	−0.0016 (6)
C14	0.0164 (8)	0.0163 (7)	0.0217 (8)	−0.0016 (6)	0.0055 (7)	0.0005 (6)
C15	0.0165 (7)	0.0173 (7)	0.0180 (8)	−0.0009 (6)	0.0057 (6)	−0.0001 (6)
C16	0.0212 (9)	0.0341 (10)	0.0294 (11)	−0.0026 (8)	−0.0031 (8)	−0.0018 (8)
C17	0.0172 (8)	0.0163 (7)	0.0233 (9)	0.0007 (6)	0.0043 (7)	0.0036 (6)
C18	0.0242 (9)	0.0168 (8)	0.0280 (10)	−0.0041 (6)	0.0035 (7)	−0.0046 (7)

Geometric parameters (Å, º)

O1—C3	1.370 (2)	C8—H8A	0.9900
O1—C16	1.427 (2)	C8—H8B	0.9900
O2—C17	1.225 (2)	C9—C10	1.464 (2)
N1—C17	1.348 (2)	C10—C11	1.399 (2)
N1—N2	1.3916 (19)	C10—C15	1.401 (2)
N1—C7	1.487 (2)	C11—C12	1.389 (2)
N2—C9	1.294 (2)	C11—H11A	0.9500
C1—C2	1.385 (3)	C12—C13	1.397 (2)
C1—C6	1.400 (2)	C12—H12A	0.9500
C1—H1A	0.9500	C13—C14	1.394 (3)
C2—C3	1.397 (2)	C13—C18	1.509 (2)
C2—H2A	0.9500	C14—C15	1.396 (2)
C3—C4	1.396 (2)	C14—H14A	0.9500
C4—C5	1.401 (3)	C15—H15A	0.9500
C4—H4A	0.9500	C16—H16A	0.9800
C5—C6	1.391 (2)	C16—H16B	0.9800
C5—H5A	0.9500	C16—H16C	0.9800
C6—C7	1.520 (2)	C17—H17A	0.9500
C7—C8	1.547 (2)	C18—H18A	0.9800
C7—H7A	1.0000	C18—H18B	0.9800
C8—C9	1.510 (2)	C18—H18C	0.9800
C3—O1—C16	117.61 (16)	N2—C9—C8	113.76 (14)
C17—N1—N2	120.11 (15)	C10—C9—C8	124.81 (15)
C17—N1—C7	126.02 (15)	C11—C10—C15	118.75 (15)
N2—N1—C7	113.67 (13)	C11—C10—C9	120.65 (15)
C9—N2—N1	107.38 (14)	C15—C10—C9	120.60 (16)
C2—C1—C6	120.53 (16)	C12—C11—C10	120.46 (16)
C2—C1—H1A	119.7	C12—C11—H11A	119.8
C6—C1—H1A	119.7	C10—C11—H11A	119.8
C1—C2—C3	120.51 (16)	C11—C12—C13	121.13 (16)
C1—C2—H2A	119.7	C11—C12—H12A	119.4
C3—C2—H2A	119.7	C13—C12—H12A	119.4
O1—C3—C4	125.21 (16)	C14—C13—C12	118.34 (16)
O1—C3—C2	115.02 (16)	C14—C13—C18	120.72 (16)
C4—C3—C2	119.76 (16)	C12—C13—C18	120.95 (17)
C3—C4—C5	119.09 (16)	C13—C14—C15	121.08 (16)
C3—C4—H4A	120.5	C13—C14—H14A	119.5
C5—C4—H4A	120.5	C15—C14—H14A	119.5
C6—C5—C4	121.44 (16)	C14—C15—C10	120.23 (16)
C6—C5—H5A	119.3	C14—C15—H15A	119.9
C4—C5—H5A	119.3	C10—C15—H15A	119.9
C5—C6—C1	118.65 (16)	O1—C16—H16A	109.5
C5—C6—C7	120.08 (15)	O1—C16—H16B	109.5
C1—C6—C7	121.11 (15)	H16A—C16—H16B	109.5
N1—C7—C6	109.79 (14)	O1—C16—H16C	109.5

N1—C7—C8	99.73 (14)	H16A—C16—H16C	109.5
C6—C7—C8	114.96 (15)	H16B—C16—H16C	109.5
N1—C7—H7A	110.6	O2—C17—N1	123.94 (18)
C6—C7—H7A	110.6	O2—C17—H17A	118.0
C8—C7—H7A	110.6	N1—C17—H17A	118.0
C9—C8—C7	102.57 (14)	C13—C18—H18A	109.5
C9—C8—H8A	111.3	C13—C18—H18B	109.5
C7—C8—H8A	111.3	H18A—C18—H18B	109.5
C9—C8—H8B	111.3	C13—C18—H18C	109.5
C7—C8—H8B	111.3	H18A—C18—H18C	109.5
H8A—C8—H8B	109.2	H18B—C18—H18C	109.5
N2—C9—C10	121.34 (15)		

C17—N1—N2—C9	176.47 (16)	N1—C7—C8—C9	−15.61 (17)
C7—N1—N2—C9	−8.36 (18)	C6—C7—C8—C9	101.70 (17)
C6—C1—C2—C3	0.3 (3)	N1—N2—C9—C10	179.55 (14)
C16—O1—C3—C4	−1.7 (3)	N1—N2—C9—C8	−3.61 (19)
C16—O1—C3—C2	177.67 (17)	C7—C8—C9—N2	13.1 (2)
C1—C2—C3—O1	179.00 (16)	C7—C8—C9—C10	−170.18 (15)
C1—C2—C3—C4	−1.6 (3)	N2—C9—C10—C11	−2.2 (2)
O1—C3—C4—C5	−179.20 (17)	C8—C9—C10—C11	−178.69 (17)
C2—C3—C4—C5	1.5 (3)	N2—C9—C10—C15	177.74 (16)
C3—C4—C5—C6	−0.1 (3)	C8—C9—C10—C15	1.3 (2)
C4—C5—C6—C1	−1.2 (3)	C15—C10—C11—C12	−0.6 (2)
C4—C5—C6—C7	174.34 (16)	C9—C10—C11—C12	179.34 (16)
C2—C1—C6—C5	1.1 (3)	C10—C11—C12—C13	−0.5 (3)
C2—C1—C6—C7	−174.42 (16)	C11—C12—C13—C14	1.2 (3)
C17—N1—C7—C6	69.3 (2)	C11—C12—C13—C18	−178.97 (17)
N2—N1—C7—C6	−105.52 (16)	C12—C13—C14—C15	−0.8 (3)
C17—N1—C7—C8	−169.58 (16)	C18—C13—C14—C15	179.34 (16)
N2—N1—C7—C8	15.59 (18)	C13—C14—C15—C10	−0.3 (3)
C5—C6—C7—N1	−95.70 (18)	C11—C10—C15—C14	1.0 (2)
C1—C6—C7—N1	79.7 (2)	C9—C10—C15—C14	−178.97 (15)
C5—C6—C7—C8	152.86 (16)	N2—N1—C17—O2	178.37 (16)
C1—C6—C7—C8	−31.7 (2)	C7—N1—C17—O2	3.8 (3)

Hydrogen-bond geometry (Å, º)

Cg2 is the centroid of the C1–C6 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*A*···O2^{i}	0.95	2.39	3.207 (2)	144
C14—H14*A*···O2^{ii}	0.95	2.57	3.477 (2)	160
C15—H15*A*···*Cg*2^{iii}	0.95	2.78	3.661 (2)	154

Symmetry codes: (i) *x*, *y*+1, *z*; (ii) *x*+1/2, *y*+3/2, *z*; (iii) *x*+1/2, *y*+1/2, *z*.