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(*E*)-1-(3-Methoxyphenyl)ethanone 4-nitrophenylhydrazone

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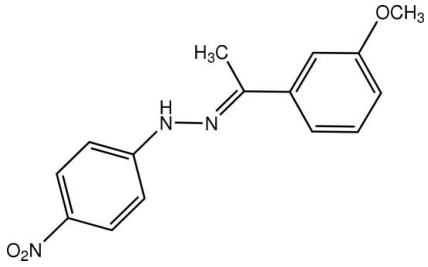
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.003 Å; R factor = 0.049; wR factor = 0.141; data-to-parameter ratio = 15.7.

Crystals of the title compound, $C_{15}H_{15}N_3O_3$, were obtained from a condensation reaction of 4-nitrophenylhydrazine and 3-methoxyacetophenone. In the crystal structure, the methoxyphenyl ring is twisted slightly with respect to the nitrophenylhydrazine plane, making a dihedral angle of 14.81 (8)°. The nitro and methoxy groups are each coplanar with the attached benzene rings. The nitrophenyl and methoxyphenyl groups are located on opposite sides of the C=N double bond, indicating an *E* configuration of the molecule. Adjacent molecules are linked together *via* N—H···O hydrogen bonding, forming chains along the [101] direction.

Related literature

For general background, see: Okabe *et al.* (1993); Shan *et al.* (2003*a*). For related structures, see: Shan *et al.* (2003*b*, 2004, 2008).



Experimental

Crystal data

$C_{15}H_{15}N_3O_3$ M_r = 285.30
Monoclinic, $P2_1/c$
a = 4.2977 (17) Å
b = 24.709 (9) Å
c = 13.132 (5) Å
β = 96.332 (11)°
V = 1386.0 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 295 (2) K
0.32 × 0.26 × 0.22 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
Absorption correction: none
16470 measured reflections
3014 independent reflections
1643 reflections with $I > 2\sigma(I)$
R_{int} = 0.045

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.141
S = 1.03
3014 reflections
192 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O3^{i}	0.86	2.45	3.279 (2)	161

Symmetry code: (i) $x-1, -y+\frac{3}{2}, z-\frac{1}{2}$.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: OM2241).

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supporting information

Acta Cryst. (2008). E**64**, o1341 [doi:10.1107/S1600536808018618]

(*E*)-1-(3-Methoxyphenyl)ethanone 4-nitrophenylhydrazone

Zheng Fan, Shang Shan, Shan-Heng Wang and Wen-Long Wang

S1. Comment

Since some phenylhydrazone derivatives have shown to be potential DNA damaging and mutagenic agents (Okabe *et al.*, 1993), a series of new phenylhydrazone derivatives have been prepared in our laboratory (Shan *et al.*, 2003*a*). As part of the ongoing investigation, the title compound has recently been prepared and its crystal structure is reported here.

The molecular structure of the title compound is shown in Fig. 1. The N1—C7 bond distance of 1.295 (2) Å indicates a typical C=N double bond. The molecule assumes an E configuration, with the nitrophenyl ring and methoxyphenyl rings located on the opposite sites of the C=N bond. The dihedral angle of 1.4 (3)° between nitro group and C10-benzene ring and the C1—C2—C3—C4 torsion angle of 0.9 (3)° suggest that nitro and methoxyl groups are co-planar with the individual benzene rings. The methoxyphenyl ring is slightly twisted with respect to the nitrophenylhydrazine mean plane by a small dihedral angle of 14.81 (8)°, indicating the molecule is approximately co-planar except for methyl H atoms.

In the crystal structure adjacent molecules are linked *via* N—H···O hydrogen bonding to form chains along the [1 0 1] direction (Table 1 and Fig. 2). Although π-π stacking was found between 4-nitrophenyl rings in several related structures previously reported, benzil 4-nitrophenylhydrazone (Shan *et al.*, 2003*b*), 2-chloro-3,4-dimethoxybenzaldehyde 4-nitrophenylhydrazone (Shan *et al.*, 2004) and acetylpyrazine 4-nitrophenylhydrazone (Shan *et al.*, 2008), no π-π stacking is observed in the crystal structure.

S2. Experimental

4-Nitrophenylhydrazine (0.31 g, 2 mmol) was dissolved in ethanol (10 ml), then H_2SO_4 solution (98%, 0.5 ml) was added slowly to the ethanol solution with stirring. The solution was heated at about 333 K for several minutes until the solution cleared. An ethanol solution (5 ml) of 3-methoxyacetophenone (0.30 g, 2 mmol) was dropped slowly into the above solution with continuous stirring, and the mixture solution was kept at about 333 K for 0.5 h. When the solution had cooled to room temperature, red microcrystals appeared. They were separated and washed with cold water three times to get the product 0.45 g. Single crystals of the title compound were obtained by recrystallization from an absolute ethanol solution.

S3. Refinement

Methyl H atoms were placed in calculated positions with C—H = 0.96 Å and the torsion angle was refined to fit the electron density, U_{iso}(H) = 1.5U_{eq}(C). Other H atoms were placed in calculated positions with C—H = 0.93 and N—H = 0.86 Å, and refined in riding mode with U_{iso}(H) = 1.2U_{eq}(C,*N*).

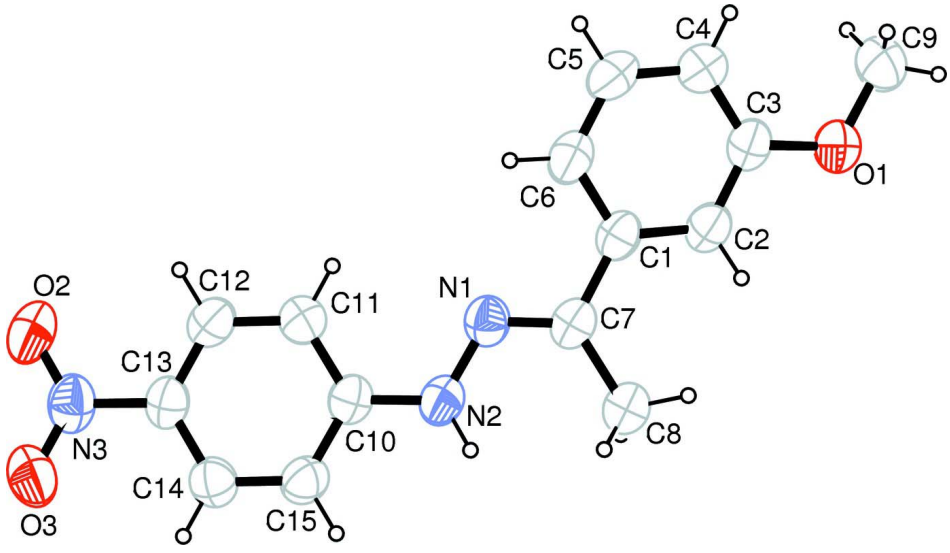

Figure 1

The molecular structure of the title compound with 30% probability displacement ellipsoids for non-H atoms.

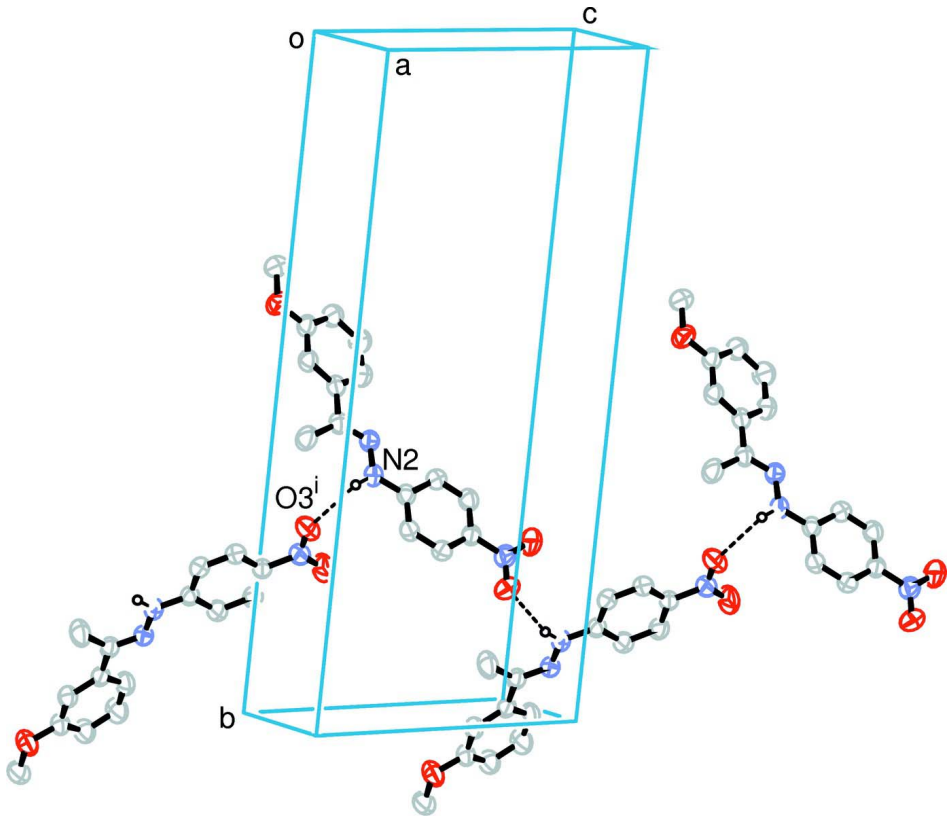

Figure 2

A diagram showing the N—H···O hydrogen bond chain (dashed lines) [symmetry code: (i) -1 + *x*, 3/2 - *y*, -1/2 + *z*].

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Crystal data

$C_{15}H_{15}N_3O_3$
$M_r = 285.30$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 4.2977\ (17)$ Å
$b = 24.709\ (9)$ Å
$c = 13.132\ (5)$ Å
$\beta = 96.332\ (11)°$
$V = 1386.0\ (9)$ Å^3
$Z = 4$
$F(000) = 600$
$D_x = 1.367$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4665 reflections
$\theta = 2.0–25.0°$
$\mu = 0.10$ mm^{-1}
$T = 295$ K
Prism, red
0.32 × 0.26 × 0.22 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 10.00 pixels mm^{-1}
ω scans
16470 measured reflections
3014 independent reflections
1643 reflections with $I > 2\sigma(I)$
$R_{int} = 0.045$
$\theta_{max} = 27.0°$, $\theta_{min} = 1.7°$
$h = -5\rightarrow5$
$k = -30\rightarrow31$
$l = -16\rightarrow15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.141$
$S = 1.03$
3014 reflections
192 parameters
0 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
$w = 1/[\sigma^2(F_o^2) + (0.0685P)^2 + 0.02P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.15$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.2858 (3)	0.59466 (6)	0.29114 (11)	0.0540 (4)
N2	0.4535 (3)	0.63991 (6)	0.27160 (11)	0.0573 (4)
H2	0.4405	0.6533	0.2109	0.069*
N3	1.2264 (4)	0.73954 (7)	0.58666 (13)	0.0656 (5)
O1	−0.4958 (3)	0.41329 (6)	0.10899 (10)	0.0775 (5)

O2	1.2533 (4)	0.71902 (6)	0.67268 (11)	0.0944 (6)
O3	1.3617 (4)	0.78225 (6)	0.56823 (11)	0.0865 (5)
C1	−0.0634 (4)	0.52365 (7)	0.24122 (12)	0.0507 (5)
C2	−0.2090 (4)	0.48991 (7)	0.16601 (13)	0.0563 (5)
H2A	−0.2011	0.4985	0.0974	0.068*
C3	−0.3663 (4)	0.44357 (8)	0.19125 (13)	0.0574 (5)
C4	−0.3852 (5)	0.43034 (8)	0.29148 (15)	0.0680 (6)
H4	−0.4900	0.3993	0.3087	0.082*
C5	−0.2442 (5)	0.46436 (9)	0.36672 (14)	0.0775 (7)
H5	−0.2576	0.4560	0.4351	0.093*
C6	−0.0856 (5)	0.51000 (8)	0.34328 (14)	0.0669 (6)
H6	0.0075	0.5319	0.3955	0.080*
C7	0.1106 (4)	0.57271 (7)	0.21575 (13)	0.0528 (5)
C8	0.0787 (5)	0.59459 (9)	0.10844 (15)	0.0818 (7)
H8A	0.0396	0.6328	0.1099	0.123*
H8B	−0.0926	0.5769	0.0686	0.123*
H8C	0.2686	0.5880	0.0782	0.123*
C9	−0.6460 (5)	0.36382 (8)	0.13053 (16)	0.0790 (7)
H9A	−0.5045	0.3419	0.1746	0.119*
H9B	−0.7058	0.3448	0.0677	0.119*
H9C	−0.8290	0.3715	0.1638	0.119*
C10	0.6430 (4)	0.66358 (7)	0.35048 (13)	0.0488 (4)
C11	0.6824 (4)	0.64122 (7)	0.44883 (14)	0.0574 (5)
H11	0.5794	0.6094	0.4625	0.069*
C12	0.8742 (4)	0.66645 (8)	0.52547 (14)	0.0582 (5)
H12	0.8993	0.6518	0.5911	0.070*
C13	1.0288 (4)	0.71338 (7)	0.50517 (13)	0.0520 (5)
C14	0.9974 (4)	0.73569 (7)	0.40765 (14)	0.0566 (5)
H14	1.1054	0.7670	0.3942	0.068*
C15	0.8043 (4)	0.71083 (7)	0.33116 (14)	0.0564 (5)
H15	0.7809	0.7257	0.2657	0.068*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0518 (9)	0.0552 (9)	0.0533 (9)	0.0029 (7)	−0.0015 (7)	−0.0024 (7)
N2	0.0604 (10)	0.0626 (10)	0.0466 (9)	−0.0004 (8)	−0.0039 (7)	0.0005 (7)
N3	0.0698 (11)	0.0610 (11)	0.0636 (11)	0.0079 (9)	−0.0042 (8)	−0.0123 (9)
O1	0.1001 (11)	0.0751 (9)	0.0548 (8)	−0.0272 (8)	−0.0025 (7)	−0.0045 (7)
O2	0.1219 (14)	0.1013 (12)	0.0540 (9)	−0.0124 (10)	−0.0175 (9)	−0.0037 (8)
O3	0.1030 (12)	0.0664 (10)	0.0860 (11)	−0.0165 (9)	−0.0083 (9)	−0.0133 (8)
C1	0.0511 (11)	0.0553 (11)	0.0446 (10)	0.0082 (9)	0.0000 (8)	−0.0006 (8)
C2	0.0628 (12)	0.0616 (12)	0.0435 (10)	0.0016 (9)	0.0011 (9)	−0.0009 (8)
C3	0.0604 (12)	0.0628 (12)	0.0472 (11)	0.0002 (10)	−0.0021 (9)	−0.0039 (9)
C4	0.0795 (15)	0.0695 (13)	0.0539 (12)	−0.0123 (11)	0.0031 (10)	0.0044 (10)
C5	0.1069 (18)	0.0822 (16)	0.0425 (11)	−0.0145 (13)	0.0039 (11)	0.0070 (10)
C6	0.0812 (15)	0.0711 (13)	0.0454 (11)	−0.0056 (11)	−0.0058 (10)	−0.0020 (9)
C7	0.0540 (11)	0.0578 (11)	0.0456 (11)	0.0077 (9)	0.0014 (9)	−0.0020 (8)

C8	0.0992 (17)	0.0880 (16)	0.0542 (12)	−0.0295 (13)	−0.0098 (11)	0.0072 (10)
C9	0.0948 (16)	0.0627 (13)	0.0760 (15)	−0.0158 (12)	−0.0063 (12)	−0.0003 (11)
C10	0.0459 (10)	0.0516 (11)	0.0481 (10)	0.0075 (8)	0.0014 (8)	−0.0032 (8)
C11	0.0610 (12)	0.0545 (11)	0.0557 (12)	−0.0037 (9)	0.0027 (9)	0.0008 (9)
C12	0.0644 (12)	0.0630 (12)	0.0465 (11)	0.0042 (10)	0.0019 (9)	0.0022 (9)
C13	0.0518 (11)	0.0514 (11)	0.0511 (11)	0.0084 (9)	−0.0020 (8)	−0.0077 (8)
C14	0.0564 (12)	0.0496 (11)	0.0625 (12)	0.0029 (9)	0.0001 (9)	−0.0003 (9)
C15	0.0601 (12)	0.0566 (11)	0.0509 (11)	0.0044 (9)	−0.0013 (9)	0.0055 (9)

Geometric parameters (Å, º)

N1—C7	1.295 (2)	C6—H6	0.9300
N1—N2	1.3699 (19)	C7—C8	1.501 (3)
N2—C10	1.376 (2)	C8—H8A	0.9600
N2—H2	0.8600	C8—H8B	0.9600
N3—O2	1.232 (2)	C8—H8C	0.9600
N3—O3	1.242 (2)	C9—H9A	0.9600
N3—C13	1.443 (2)	C9—H9B	0.9600
O1—C3	1.380 (2)	C9—H9C	0.9600
O1—C9	1.425 (2)	C10—C15	1.395 (2)
C1—C2	1.388 (2)	C10—C11	1.398 (3)
C1—C6	1.395 (2)	C11—C12	1.378 (2)
C1—C7	1.482 (2)	C11—H11	0.9300
C2—C3	1.388 (2)	C12—C13	1.377 (3)
C2—H2A	0.9300	C12—H12	0.9300
C3—C4	1.367 (3)	C13—C14	1.387 (2)
C4—C5	1.385 (3)	C14—C15	1.375 (2)
C4—H4	0.9300	C14—H14	0.9300
C5—C6	1.370 (3)	C15—H15	0.9300
C5—H5	0.9300		
C7—N1—N2	118.18 (15)	C7—C8—H8B	109.5
N1—N2—C10	119.09 (14)	H8A—C8—H8B	109.5
N1—N2—H2	120.5	C7—C8—H8C	109.5
C10—N2—H2	120.5	H8A—C8—H8C	109.5
O2—N3—O3	121.99 (17)	H8B—C8—H8C	109.5
O2—N3—C13	118.92 (18)	O1—C9—H9A	109.5
O3—N3—C13	119.08 (17)	O1—C9—H9B	109.5
C3—O1—C9	117.53 (15)	H9A—C9—H9B	109.5
C2—C1—C6	117.71 (18)	O1—C9—H9C	109.5
C2—C1—C7	122.00 (16)	H9A—C9—H9C	109.5
C6—C1—C7	120.29 (16)	H9B—C9—H9C	109.5
C1—C2—C3	121.26 (17)	N2—C10—C15	118.92 (16)
C1—C2—H2A	119.4	N2—C10—C11	121.93 (16)
C3—C2—H2A	119.4	C15—C10—C11	119.14 (16)
C4—C3—O1	124.23 (18)	C12—C11—C10	119.85 (17)
C4—C3—C2	120.60 (17)	C12—C11—H11	120.1
O1—C3—C2	115.17 (16)	C10—C11—H11	120.1

C3—C4—C5	118.30 (19)	C13—C12—C11	120.15 (17)
C3—C4—H4	120.8	C13—C12—H12	119.9
C5—C4—H4	120.8	C11—C12—H12	119.9
C6—C5—C4	121.90 (18)	C12—C13—C14	120.89 (16)
C6—C5—H5	119.0	C12—C13—N3	119.38 (17)
C4—C5—H5	119.0	C14—C13—N3	119.73 (18)
C5—C6—C1	120.22 (18)	C15—C14—C13	119.09 (18)
C5—C6—H6	119.9	C15—C14—H14	120.5
C1—C6—H6	119.9	C13—C14—H14	120.5
N1—C7—C1	115.80 (16)	C14—C15—C10	120.86 (17)
N1—C7—C8	123.50 (18)	C14—C15—H15	119.6
C1—C7—C8	120.69 (16)	C10—C15—H15	119.6
C7—C8—H8A	109.5		

C7—N1—N2—C10	−179.48 (14)	C6—C1—C7—C8	−166.53 (19)
C6—C1—C2—C3	−1.2 (3)	N1—N2—C10—C15	−177.60 (15)
C7—C1—C2—C3	178.84 (16)	N1—N2—C10—C11	3.4 (2)
C9—O1—C3—C4	−3.0 (3)	N2—C10—C11—C12	−179.86 (15)
C9—O1—C3—C2	176.82 (17)	C15—C10—C11—C12	1.2 (3)
C1—C2—C3—C4	0.9 (3)	C10—C11—C12—C13	−0.5 (3)
C1—C2—C3—O1	−178.98 (16)	C11—C12—C13—C14	−0.7 (3)
O1—C3—C4—C5	179.96 (18)	C11—C12—C13—N3	179.40 (16)
C2—C3—C4—C5	0.1 (3)	O2—N3—C13—C12	0.4 (3)
C3—C4—C5—C6	−0.7 (3)	O3—N3—C13—C12	−179.01 (17)
C4—C5—C6—C1	0.3 (3)	O2—N3—C13—C14	−179.54 (17)
C2—C1—C6—C5	0.7 (3)	O3—N3—C13—C14	1.1 (3)
C7—C1—C6—C5	−179.42 (18)	C12—C13—C14—C15	1.2 (3)
N2—N1—C7—C1	179.54 (13)	N3—C13—C14—C15	−178.92 (16)
N2—N1—C7—C8	−0.8 (3)	C13—C14—C15—C10	−0.5 (3)
C2—C1—C7—N1	−166.90 (16)	N2—C10—C15—C14	−179.69 (15)
C6—C1—C7—N1	13.2 (2)	C11—C10—C15—C14	−0.7 (3)
C2—C1—C7—C8	13.4 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O3^{i}	0.86	2.45	3.279 (2)	161

Symmetry code: (i) $x-1, -y+3/2, z-1/2$.