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trans-2-(2-Nitro-1-phenylethyl)cyclohexanone

Ivo Zenz, Herbert Mayr and Peter Mayer*

Ludwig-Maximilians-Universität, Department, Butenandtstrasse 5–13, 81377 München, Germany
Correspondence e-mail: pemay@cup.uni-muenchen.de

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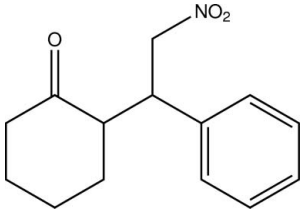
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.036; wR factor = 0.090; data-to-parameter ratio = 16.0.

In the title compound, $C_{14}H_{17}NO_3$, the plane of the phenyl ring and the least-squares plane of the cyclohexyl moiety enclose an angle of 89.14 (6)°. The cyclohexyl ring adopts a chair conformation. In the crystal, the molecules are linked by weak C—H···O bonds, with each of the nitro-O atoms accepting two such interactions.

Related literature

For the history and synthesis of nitroalkenes, see: Tsogoeva *et al.* (2007); Sulzer-Mosse & Alexakis (2007); Mukherjee *et al.* (2007); Kempf *et al.* (2003); Blarer *et al.* (1982); Juaristi *et al.* (1993). For related structures, see: Cobb *et al.* (2005), Xu *et al.* (2007*a*,*b*).



Experimental

Crystal data

$C_{14}H_{17}NO_3$
M_r = 247.29
Monoclinic, $P2_1/c$
a = 13.4567 (6) Å
b = 8.3618 (4) Å
c = 11.3668 (5) Å
β = 91.734 (4)°
V = 1278.43 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 173 K
0.38 × 0.27 × 0.18 mm

Data collection

Oxford Xcalibur diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2006)
T_{min} = 0.986, T_{max} = 1.000
9360 measured reflections
2605 independent reflections
1829 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.090
S = 0.98
2605 reflections
163 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 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
C1—H1B···O2^{i}	0.99	2.57	3.4403 (14)	146
C5—H5A···O2^{ii}	0.99	2.47	3.4312 (16)	165
C8—H8A···O1^{iii}	0.99	2.53	3.3536 (16)	140
C10—H10···O2^{i}	0.95	2.50	3.4289 (15)	165

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2006); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG5052).

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supporting information

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trans-2-(2-Nitro-1-phenylethyl)cyclohexanone

Ivo Zenz, Herbert Mayr and Peter Mayer

S1. Comment

Nitroalkenes are important reagents in organic chemistry and they are the most prominent Michael acceptors used in organocatalytic reactions [Tsogoeva *et al.* (2007), Sulzer-Mosse *et al.* (2007), Mukherjee *et al.* (2007)]. During our studies on the electrophilic reactivity of *trans*-*β*-nitrostyrenes, we employed enamines of known nucleophilic reactivities and, hence, obtained the title compound from a reaction of *trans*-*β*-nitrostyrene and 1-pyrrolidinocyclohexene [Kempf *et al.* (2003), Blarer *et al.* (1982), Juaristi *et al.* (1993)].

In the title compound, the 1′-Phenyl-2′-nitro-ethyl moiety occupies an equatorial binding site in 2-position of the cyclohexanone ring (see Fig. 1). The plane of the phenyl ring and the least-square plane of the cyclohexyl moiety enclose an angle of 89.14 (6)° which is close to the angles found in enantiopure crystals of the title compound (87.1 (3)° at 180 K (Cobb *et al.* (2005)), 87.40 (8)° at 296 K (Xu *et al.*, 2007a,b)). The plane through the nitro group and the adjacent C1 atom encloses an angle of 68.81 (7)° with the phenyl ring.

Taking into account merely interactions with hydrogen-acceptor distances at least 0.1 Å shorter than the sum of van-der-Waals radii, the molecules are linked by very weak contacts of the type C—H···O with nitro-O atoms as acceptors (see Fig. 2). The molecular structure of the title compound is stabilized by these contacts as well, as the involved hydrogen atoms are located in the cyclohexyl ring, the phenyl ring and the nitro-ethyl side chain. The keto group is not involved in hydrogen bonding. *π*-stacking and C—H···*π*-interactions are not observed.

S2. Experimental

trans-2-[1′-Phenyl-2′-nitro-ethyl]-cyclohexanone has been obtained by dissolving *trans*-*β*-nitrostyrene (4.05 mmol, 604 mg) in dry diethylether (40 ml) and dropwise addition of 1-pyrrolidinocyclohexene (4.05 mmol, 612 mg) at -78 °C. After stirring the reaction at RT for 2 h, 60 ml water, 60 ml e thanol and 5 ml 1*M* HCl have been added, and the mixture was stirred for 30 min at 60 °C. After removing the solvent *in vacuo*, a white solid has been obtained (3.01 mmol, 745 mg, 74%).

Crystallization procedure: The title compound was dissolved in ethanol and heated to the boiling point. The solvent was allowed to cool slowly to room temperature. After 24 h, colourless crystals had formed that were suitable for X-ray analysis; mp 108 °C.

S3. Refinement

All H atoms were calculated in ideal geometry and refined in a riding model.

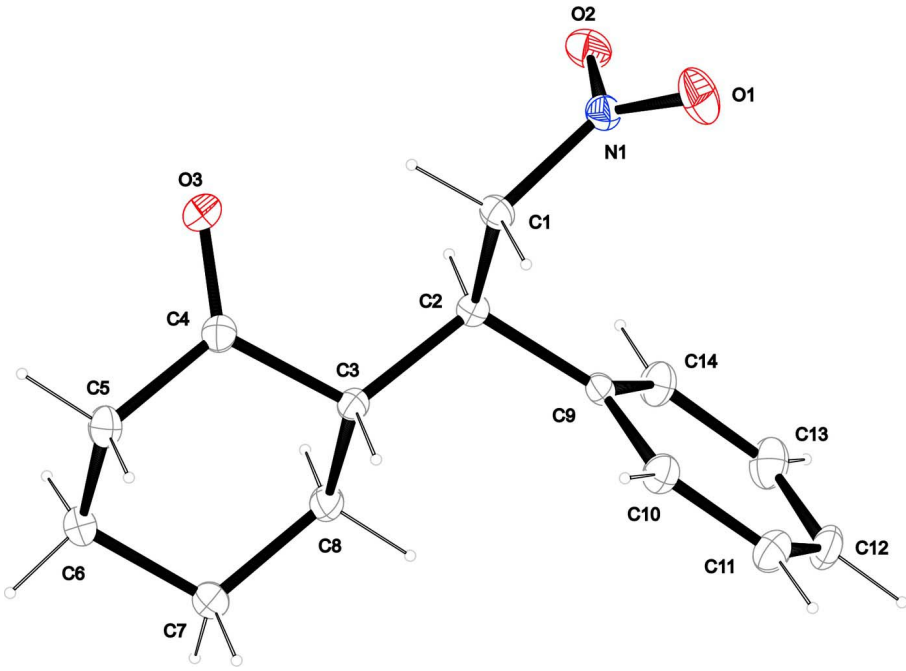

Figure 1

The molecular structure of the title compound, with atom labels and anisotropic displacement ellipsoids (drawn at 50% probability level) for non-H atoms.

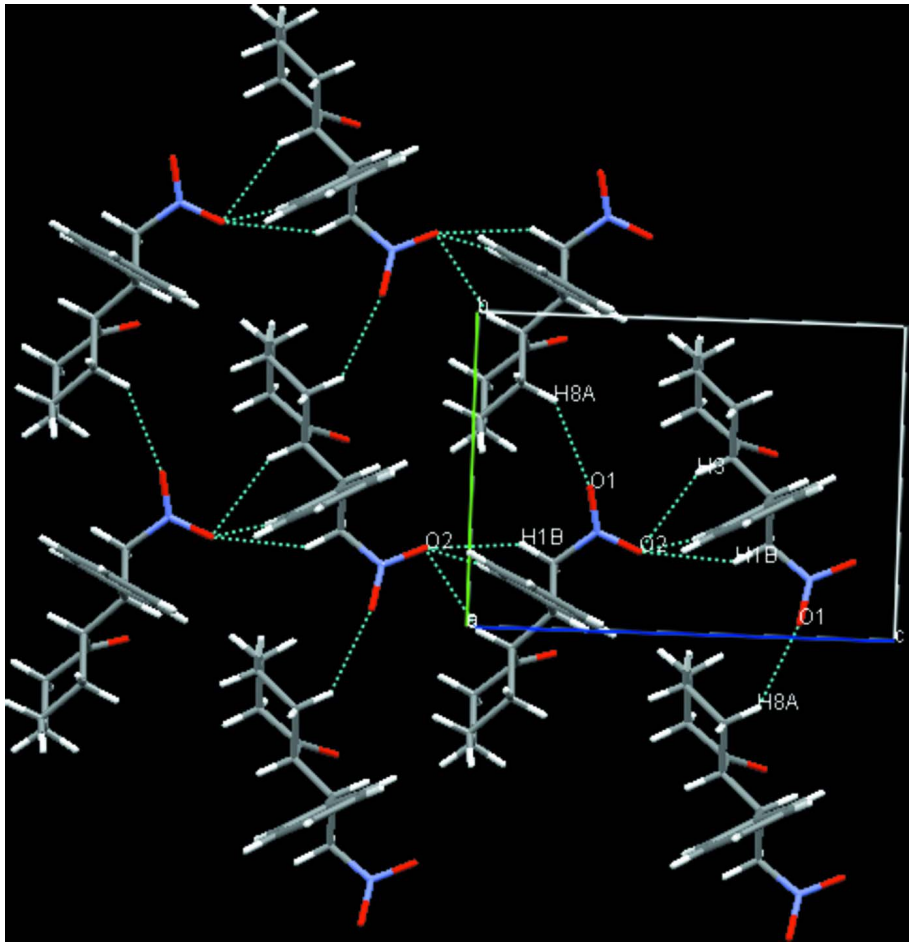

Figure 2

Weak intermolecular interactions in the crystal structure of the title compound viewed along [100].

trans-2-(2-Nitro-1-phenylethyl)cyclohexanone

Crystal data

$C_{14}H_{17}NO_3$
$M_r = 247.29$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 13.4567\ (6)$ Å
$b = 8.3618\ (4)$ Å
$c = 11.3668\ (5)$ Å
$\beta = 91.734\ (4)°$
$V = 1278.43\ (10)$ Å^3
$Z = 4$

$F(000) = 528$
$D_x = 1.285\ (1)$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3796 reflections
$\theta = 4.3–26.3°$
$\mu = 0.09$ mm^{-1}
$T = 173$ K
Block, colourless
0.38 × 0.27 × 0.18 mm

Data collection

Oxford Xcalibur diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans

Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2006)
$T_{min} = 0.986$, $T_{max} = 1.000$
9360 measured reflections
2605 independent reflections
1829 reflections with $I > 2\sigma(I)$

R_{int} = 0.026
θ_{max} = 26.4°, θ_{min} = 4.3°
h = −16→16
k = −10→10
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.090
S = 0.98
2605 reflections
163 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
$w = 1/[\sigma^2(F_o^2) + (0.0503P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Special details

Experimental. CrysAlisPro, Oxford Diffraction Ltd., Version 1.171.33.41 (release 06-05-2009 CrysAlis171 .NET) (compiled May 6 2009,17:20:42) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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

	x	y	z	U_{iso}*/U_{eq}
O1	0.27791 (8)	0.45264 (12)	0.27671 (8)	0.0497 (3)
O2	0.30532 (7)	0.25646 (11)	0.39600 (7)	0.0361 (3)
O3	0.46549 (7)	−0.07785 (11)	0.20442 (8)	0.0378 (3)
N1	0.30166 (8)	0.31433 (13)	0.29688 (9)	0.0272 (3)
C1	0.32921 (9)	0.20990 (14)	0.19718 (10)	0.0243 (3)
H1A	0.4012	0.1854	0.2029	0.029*
H1B	0.3155	0.2661	0.1218	0.029*
C2	0.26933 (9)	0.05426 (14)	0.20005 (10)	0.0214 (3)
H2	0.2817	0.0036	0.2788	0.026*
C3	0.30432 (9)	−0.06309 (14)	0.10615 (10)	0.0217 (3)
H3	0.2931	−0.0124	0.0272	0.026*
C4	0.41304 (9)	−0.11083 (15)	0.11829 (11)	0.0258 (3)
C5	0.44795 (10)	−0.21687 (16)	0.02103 (11)	0.0333 (3)
H5A	0.5197	−0.2403	0.0330	0.040*
H5B	0.4383	−0.1623	−0.0558	0.040*
C6	0.38803 (10)	−0.37313 (16)	0.02159 (11)	0.0333 (3)
H6A	0.4068	−0.4406	−0.0458	0.040*
H6B	0.4037	−0.4330	0.0950	0.040*
C7	0.27722 (9)	−0.33748 (16)	0.01310 (11)	0.0325 (3)
H7A	0.2397	−0.4386	0.0205	0.039*
H7B	0.2605	−0.2914	−0.0653	0.039*
C8	0.24528 (10)	−0.22137 (15)	0.10807 (11)	0.0279 (3)
H8A	0.2551	−0.2724	0.1862	0.033*
H8B	0.1735	−0.1978	0.0966	0.033*
C9	0.15860 (9)	0.08735 (14)	0.18614 (10)	0.0220 (3)
C10	0.11872 (9)	0.16173 (15)	0.08584 (10)	0.0298 (3)

H10	0.1618	0.1982	0.0268	0.036*
C11	0.01755 (10)	0.18327 (17)	0.07092 (12)	0.0358 (3)
H11	−0.0082	0.2349	0.0021	0.043*
C12	−0.04660 (10)	0.13068 (16)	0.15475 (12)	0.0368 (4)
H12	−0.1163	0.1439	0.1434	0.044*
C13	−0.00848 (11)	0.05889 (17)	0.25497 (13)	0.0393 (4)
H13	−0.0521	0.0231	0.3136	0.047*
C14	0.09322 (10)	0.03820 (16)	0.27118 (11)	0.0316 (3)
H14	0.1186	−0.0103	0.3415	0.038*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0667 (8)	0.0264 (6)	0.0566 (7)	0.0132 (5)	0.0092 (5)	−0.0020 (5)
O2	0.0429 (6)	0.0418 (6)	0.0236 (5)	−0.0013 (5)	0.0033 (4)	−0.0039 (4)
O3	0.0260 (5)	0.0419 (6)	0.0449 (6)	0.0040 (4)	−0.0083 (4)	−0.0096 (5)
N1	0.0240 (6)	0.0270 (6)	0.0306 (6)	−0.0007 (5)	0.0020 (4)	−0.0046 (5)
C1	0.0260 (7)	0.0258 (7)	0.0212 (6)	−0.0004 (6)	0.0048 (5)	−0.0022 (5)
C2	0.0215 (6)	0.0223 (7)	0.0204 (6)	0.0001 (5)	0.0014 (5)	0.0015 (5)
C3	0.0202 (6)	0.0225 (7)	0.0224 (6)	0.0013 (5)	−0.0008 (5)	0.0007 (5)
C4	0.0233 (7)	0.0234 (7)	0.0308 (7)	−0.0014 (6)	0.0026 (5)	0.0010 (5)
C5	0.0247 (7)	0.0381 (8)	0.0373 (7)	0.0055 (6)	0.0055 (6)	−0.0061 (6)
C6	0.0321 (8)	0.0299 (8)	0.0377 (7)	0.0080 (6)	−0.0019 (6)	−0.0082 (6)
C7	0.0309 (8)	0.0269 (7)	0.0395 (8)	0.0019 (6)	−0.0040 (6)	−0.0070 (6)
C8	0.0215 (7)	0.0253 (7)	0.0368 (7)	−0.0004 (6)	0.0004 (5)	−0.0035 (6)
C9	0.0222 (7)	0.0183 (6)	0.0255 (6)	0.0010 (5)	0.0011 (5)	−0.0044 (5)
C10	0.0270 (7)	0.0326 (8)	0.0299 (7)	0.0017 (6)	0.0018 (5)	0.0017 (6)
C11	0.0315 (8)	0.0384 (8)	0.0371 (8)	0.0066 (7)	−0.0052 (6)	0.0032 (6)
C12	0.0202 (7)	0.0360 (8)	0.0540 (9)	0.0061 (6)	−0.0015 (6)	−0.0032 (7)
C13	0.0274 (8)	0.0412 (9)	0.0500 (8)	0.0009 (7)	0.0126 (6)	0.0048 (7)
C14	0.0277 (7)	0.0332 (8)	0.0340 (7)	0.0031 (6)	0.0053 (6)	0.0060 (6)

Geometric parameters (Å, º)

O1—N1	1.2198 (13)	C6—H6A	0.9900
O2—N1	1.2258 (12)	C6—H6B	0.9900
O3—C4	1.2210 (14)	C7—C8	1.5234 (17)
N1—C1	1.4865 (15)	C7—H7A	0.9900
C1—C2	1.5316 (16)	C7—H7B	0.9900
C1—H1A	0.9900	C8—H8A	0.9900
C1—H1B	0.9900	C8—H8B	0.9900
C2—C9	1.5192 (17)	C9—C14	1.3888 (17)
C2—C3	1.5343 (16)	C9—C10	1.3919 (16)
C2—H2	1.0000	C10—C11	1.3786 (18)
C3—C4	1.5187 (17)	C10—H10	0.9500
C3—C8	1.5442 (16)	C11—C12	1.3770 (19)
C3—H3	1.0000	C11—H11	0.9500
C4—C5	1.5035 (18)	C12—C13	1.3732 (19)

C5—C6	1.5355 (19)	C12—H12	0.9500
C5—H5A	0.9900	C13—C14	1.3862 (19)
C5—H5B	0.9900	C13—H13	0.9500
C6—C7	1.5209 (18)	C14—H14	0.9500

O1—N1—O2	123.37 (10)	C7—C6—H6B	109.6
O1—N1—C1	118.92 (10)	C5—C6—H6B	109.6
O2—N1—C1	117.70 (10)	H6A—C6—H6B	108.1
N1—C1—C2	109.84 (9)	C6—C7—C8	112.15 (10)
N1—C1—H1A	109.7	C6—C7—H7A	109.2
C2—C1—H1A	109.7	C8—C7—H7A	109.2
N1—C1—H1B	109.7	C6—C7—H7B	109.2
C2—C1—H1B	109.7	C8—C7—H7B	109.2
H1A—C1—H1B	108.2	H7A—C7—H7B	107.9
C9—C2—C1	110.98 (10)	C7—C8—C3	112.33 (10)
C9—C2—C3	111.39 (9)	C7—C8—H8A	109.1
C1—C2—C3	110.85 (9)	C3—C8—H8A	109.1
C9—C2—H2	107.8	C7—C8—H8B	109.1
C1—C2—H2	107.8	C3—C8—H8B	109.1
C3—C2—H2	107.8	H8A—C8—H8B	107.9
C4—C3—C2	114.83 (9)	C14—C9—C10	117.75 (12)
C4—C3—C8	105.55 (10)	C14—C9—C2	120.91 (10)
C2—C3—C8	111.68 (10)	C10—C9—C2	121.29 (11)
C4—C3—H3	108.2	C11—C10—C9	120.93 (12)
C2—C3—H3	108.2	C11—C10—H10	119.5
C8—C3—H3	108.2	C9—C10—H10	119.5
O3—C4—C5	122.47 (12)	C12—C11—C10	120.70 (12)
O3—C4—C3	123.05 (11)	C12—C11—H11	119.7
C5—C4—C3	114.19 (11)	C10—C11—H11	119.7
C4—C5—C6	108.84 (11)	C13—C12—C11	119.17 (13)
C4—C5—H5A	109.9	C13—C12—H12	120.4
C6—C5—H5A	109.9	C11—C12—H12	120.4
C4—C5—H5B	109.9	C12—C13—C14	120.48 (13)
C6—C5—H5B	109.9	C12—C13—H13	119.8
H5A—C5—H5B	108.3	C14—C13—H13	119.8
C7—C6—C5	110.30 (11)	C13—C14—C9	120.94 (12)
C7—C6—H6A	109.6	C13—C14—H14	119.5
C5—C6—H6A	109.6	C9—C14—H14	119.5

O1—N1—C1—C2	−128.32 (12)	C6—C7—C8—C3	56.07 (15)
O2—N1—C1—C2	52.56 (14)	C4—C3—C8—C7	−56.14 (13)
N1—C1—C2—C9	61.75 (12)	C2—C3—C8—C7	178.44 (10)
N1—C1—C2—C3	−173.91 (9)	C1—C2—C9—C14	−122.22 (12)
C9—C2—C3—C4	−176.37 (10)	C3—C2—C9—C14	113.75 (13)
C1—C2—C3—C4	59.53 (13)	C1—C2—C9—C10	60.49 (14)
C9—C2—C3—C8	−56.26 (13)	C3—C2—C9—C10	−63.53 (15)
C1—C2—C3—C8	179.64 (9)	C14—C9—C10—C11	−1.04 (19)
C2—C3—C4—O3	10.01 (17)	C2—C9—C10—C11	176.33 (11)

C8—C3—C4—O3	−113.44 (13)	C9—C10—C11—C12	−0.4 (2)
C2—C3—C4—C5	−176.03 (10)	C10—C11—C12—C13	1.2 (2)
C8—C3—C4—C5	60.52 (13)	C11—C12—C13—C14	−0.6 (2)
O3—C4—C5—C6	112.56 (13)	C12—C13—C14—C9	−0.9 (2)
C3—C4—C5—C6	−61.43 (14)	C10—C9—C14—C13	1.68 (19)
C4—C5—C6—C7	55.04 (14)	C2—C9—C14—C13	−175.70 (12)
C5—C6—C7—C8	−53.99 (14)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*B*···O2^{i}	0.99	2.57	3.4403 (14)	146
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C8—H8*A*···O1^{iii}	0.99	2.53	3.3536 (16)	140
C10—H10···O2^{i}	0.95	2.50	3.4289 (15)	165

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