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Key indicators

Powder X-ray study
 T = 295 K
 Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$
 R factor = 0.025
 wR factor = 0.029

For details of how these key indicators were
 automatically derived from the article, see
<http://journals.iucr.org/e>.

Powder study of (*R*)-1-phenylethylammonium (*R*)-2-phenylbutyrate form 2

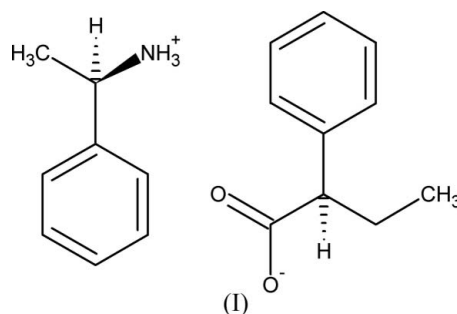
The crystal structure of a new polymorph of the title
 compound, $\text{C}_8\text{H}_{12}\text{N}^+\cdot\text{C}_{10}\text{H}_{11}\text{O}_2^-$, was solved by simulated
 annealing from laboratory X-ray powder diffraction data,
 collected at 295 K. Subsequent Rietveld refinement using data
 collected to 1.54 Å resolution yielded an R_{wp} of 0.029. The
 compound crystallized with one (*R*)-1-phenylethylammonium
 cation and one (*R*)-2-phenylbutyrate anion in the asymmetric
 unit.

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Comment

The structure of the title compound, (I), was first reported by
 Brianso (1978), hereafter referred to as form 1. Crystallization
 from ethanol yielded a second polymorph, which is reported
 here.



The crystal structure of the new form (form 2) was solved by
 simulated annealing using laboratory capillary X-ray powder
 diffraction data. The compound crystallizes in the ortho-
 rhombic space group $P2_12_12_1$ with one (*R*)-1-phenylethyl-
 ammonium cation and one (*R*)-2-phenylbutyrate anion in the
 asymmetric unit (Fig. 1).

The ion pairs in this new polymorph pack to form a
 hydrogen-bonded ladder parallel to the *a* axis (Fig. 2). Each
 ladder consists of $R_2^2(10)$ (Etter, 1990) hydrogen-bonded rings
 comprising four alternating ammonium and carboxylate
 groups linked by $\text{N}-\text{H}\cdots\text{O}=\text{C}$ contacts (Table 1). O1 forms
 a bifurcated hydrogen bond to H1NB and H1NC, while O2
 forms just one hydrogen bond to H1NA. All strong hydrogen-
 bond donors and acceptors are satisfied.

Form 1 shows the same hydrogen-bonded ring motif, albeit
 with different packing of the ladders in the crystal structure.
 This arises because the orientation of the 2-phenylbutyrate ion
 with respect to the 1-phenylethylammonium ion is different.
 Also, the conformation of the terminal methyl group differs
 between the two forms – adopting a *gauche* conformation with
 respect to the carboxylate group in form 2, while in form 1 the
 groups are in an *anti* conformation.

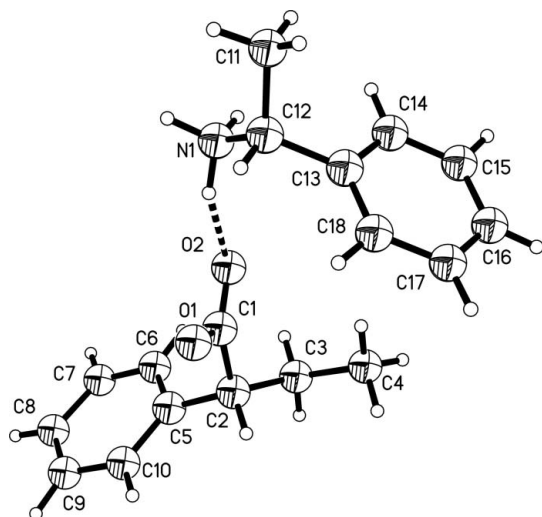


Figure 1
The asymmetric unit of (I), with the atom-numbering scheme. Displacement spheres are shown at the 50% probability level (Bruker, 2000). The dashed line indicates a hydrogen bond.

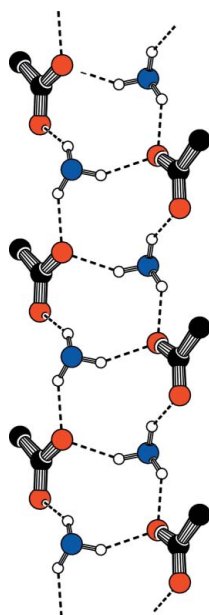


Figure 2
The hydrogen-bonded ladder motif observed in form 2. Atoms not directly involved in hydrogen-bond contacts have been omitted for clarity.

Experimental

(*R*)-2-phenylbutyric acid (Lancaster, 97% purity) and (*R*)-1-phenylethylamine (Alfa Aesar, 99+% purity) were used without further purification. The product was crystallized as a fine powder by evaporation of an ethanol solution with a starting ratio of 2:1 acid:base.

The sample was loaded into a 0.7 mm borosilicate glass capillary and rotated throughout the data collection to minimize preferred orientation effects. Data were collected using a variable count time (VCT) scheme in which the step time is increased with 2θ (Shankland *et al.*, 1997; Hill & Madsen, 2002).

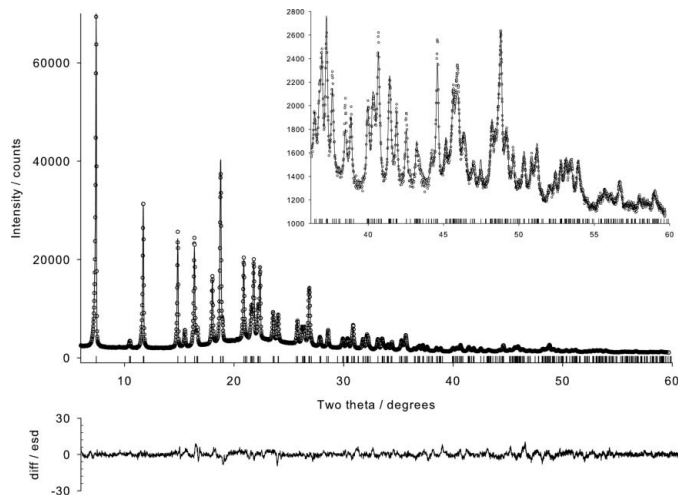


Figure 3
Final observed (points), calculated (line) and difference $[(y_{\text{obs}} - y_{\text{calc}}) / \sigma(y_{\text{obs}})]$ profiles for the Rietveld refinement of the title compound.

Crystal data

$\text{C}_8\text{H}_{12}\text{N}^+ \cdot \text{C}_{10}\text{H}_{11}\text{O}_2^-$
 $M_r = 285.37$
 Orthorhombic, $P2_12_12_1$
 $a = 6.0620$ (1) Å
 $b = 16.7794$ (3) Å
 $c = 16.8881$ (4) Å
 $V = 1717.80$ (6) Å³
 $Z = 4$
 $D_x = 1.104$ Mg m⁻³

Cu $K\alpha_1$ radiation
 Wavelength of incident radiation:
 1.54056 Å
 $\mu = 0.56$ mm⁻¹
 $T = 295$ K
 Specimen shape: cylinder
 12 × 0.7 × 0.7 mm
 Specimen prepared at 380 K
 Particle morphology: needle, white

Data collection

Bruker AXS D8 Advance diffractometer
 Specimen mounting: 0.7 mm borosilicate capillary
 Specimen mounted in transmission mode

Scan method: step
 Absorption correction: none
 $2\theta_{\text{min}} = 6.0$, $2\theta_{\text{max}} = 60.0^\circ$
 Increment in $2\theta = 0.017^\circ$

Refinement

$R_p = 0.025$
 $R_{\text{wp}} = 0.029$
 $R_{\text{exp}} = 0.015$
 $R_B = 0.022$
 $S = 2.03$
 Profile function: Fundamental parameters with axial divergence correction.
 161 parameters

Only H-atom coordinates refined
 $w = 1/\sigma(Y_{\text{obs}})^2$
 $(\Delta/\sigma)_{\text{max}} = 0.005$
 Preferred orientation correction: A spherical harmonics-based preferred orientation correction (Järvinen, 1993) was applied with *TOPAS* (Coelho, 2003) during the Rietveld refinement

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

D—H...A	D—H	H...A	D...A	D—H...A
N1—H1N ^A ...O2	0.964 (5)	1.863 (6)	2.732 (3)	148.5 (5)
N1—H1NB...O1 ⁱ	0.976 (6)	1.907 (7)	2.744 (3)	142.2 (5)
N1—H1NC...O1 ⁱⁱ	0.956 (5)	1.932 (7)	2.797 (5)	149.4 (6)
C10—H10...O2 ⁱⁱⁱ	0.953 (5)	2.492 (6)	3.426 (3)	166.8 (4)

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

The diffraction pattern indexed to a monoclinic cell [$M(19) = 30.7$, $F(19) = 63.0$; *DICVOL91*; Boultif & Louër, 1991] and the space group $P2_12_12_1$ was assigned from volume considerations and a statistical consideration of the systematic absences (Markvardsen *et al.*, 2001). The data set was background subtracted and truncated to $59.5^\circ 2\theta$ for Pawley fitting (Pawley, 1981; $\chi^2_{\text{Pawley}} = 6.10$) and the structure solved using the simulated annealing (SA) global optimization procedure, described previously (David *et al.*, 1998), that is now implemented in the *DASH* computer program (David *et al.*, 2001). The SA structure solution used 311 reflections and involved the optimization of two fragments totaling 14 degrees of freedom (six positional and orientational for each fragment present in the asymmetric unit plus a torsion angle for each fragment). All degrees of freedom were assigned random values at the start of the simulated annealing. The best SA solution had a favourable $\chi^2_{\text{SA}}/\chi^2_{\text{Pawley}}$ ratio of 3.41 and a chemically reasonable packing arrangement, with no significant misfit to the diffraction data.

The solved structure was then refined against the data in the range $6\text{--}59.7^\circ 2\theta$ using a restrained Rietveld (1969) method as implemented in *TOPAS* (Coelho, 2003), with R_{wp} falling to 0.029 during the refinement. All atomic positions (including H atoms) for the structure of (I) were refined, subject to a series of restraints on bond lengths, bond angles and planarity. The refined C–H distances were 0.949 (5)–0.973 (5) Å. U_{iso} values for H atoms were constrained to equal 0.076 \AA^2 .

The restraints were set such that bonds and angles did not deviate more than 0.01 \AA and 0.8° , respectively, from their initial values during the refinement. Atoms C16, C15, C14, C13, C18, C17, H16, H15, H14, H18 and H17 (phenylethylammonium) and atoms C5, C6, C7, C8, C9, C10, H6, H7, H8, H9 and H10 (phenylbutyrate) were restrained to lie in respective planar groups. A spherical harmonics (fourth order) correction of intensities for preferred orientation was applied in the final refinement (Järvinen, 1993). The refined final spherical harmonics coefficients were consistent with mild preferred orientation effects in the sample. The observed and calculated diffraction patterns for the refined crystal structure are shown in Fig. 3.

Data collection: *DIFFRAC plus XRD Commander* (Kienle & Jacob, 2003); cell refinement: *TOPAS* (Coelho, 2003); data reduction: *DASH* (David *et al.*, 2001); program(s) used to solve structure: *DASH*; program(s) used to refine structure: *TOPAS*; molecular graphics: *Mercury* (Macrae *et al.*, 2006) and *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *enCIFer* (Version 1.1; Allen *et al.*, 2004).

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supporting information

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Powder study of (*R*)-1-phenylethylammonium (*R*)-2-phenylbutyrate form 2

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(R)-1-phenylethylammonium (*R*)-2-phenylbutyrate*Crystal data*

$C_8H_{12}N^+ \cdot C_{10}H_{11}O_2^-$

$M_r = 285.37$

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 6.0620$ (1) Å

$b = 16.7794$ (3) Å

$c = 16.8881$ (4) Å

$V = 1717.80$ (6) Å³

$Z = 4$

$F(000) = 616.0$

$D_x = 1.104$ Mg m⁻³

Cu $K\alpha_1$ radiation, $\lambda = 1.54056$ Å

$\mu = 0.56$ mm⁻¹

$T = 295$ K

Particle morphology: needles

white

cylinder, 12×0.7 mm

Specimen preparation: Prepared at 380 K

Data collection

Bruker AXS D8 Advance
diffractometer

Radiation source: sealed X-ray tube, Bruker-
AXS D8

Primary focussing, Ge 111 monochromator

Specimen mounting: 0.7 mm borosilicate
capillary

Data collection mode: transmission

Scan method: step

$2\theta_{\min} = 6.0^\circ$, $2\theta_{\max} = 60.0^\circ$, $2\theta_{\text{step}} = 0.017^\circ$

Refinement

Least-squares matrix: selected elements only

$R_p = 0.025$

$R_{wp} = 0.029$

$R_{\text{exp}} = 0.015$

$R_{\text{Bragg}} = 0.022$

3176 data points

Profile function: Fundamental parameters with
axial divergence correction.

161 parameters

101 restraints

1 constraint

Only H-atom coordinates refined

Weighting scheme based on measured s.u.'s

$1/\sigma(Y_{\text{obs}})^2$

$(\Delta/\sigma)_{\text{max}} = 0.005$

Background function: Chebyshev polynomial

Preferred orientation correction: A spherical
harmonics-based preferred orientation
correction (Järvinen, 1993) was applied with
TOPAS (Coelho, 2003) during the Rietveld
refinement.

Special details

Geometry. Bond distances, angles *etc.* have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.0508 (4)	0.71000 (16)	0.0862 (3)	0.0421 (7)*
O2	0.3485 (5)	0.64160 (14)	0.1193 (3)	0.0421 (7)*
C1	0.14750 (17)	0.65000 (8)	0.11781 (10)	0.0421 (7)*
C2	0.00199 (18)	0.58450 (6)	0.14941 (8)	0.0421 (7)*
C3	0.10549 (19)	0.54720 (8)	0.22291 (6)	0.0421 (7)*
C4	0.13261 (15)	0.60880 (5)	0.28920 (5)	0.0421 (7)*
C5	-0.0445 (2)	0.52320 (9)	0.08661 (10)	0.0421 (7)*
C6	0.1146 (2)	0.46661 (10)	0.06781 (8)	0.0421 (7)*
C7	0.07689 (19)	0.41050 (8)	0.00882 (15)	0.0421 (7)*
C8	-0.1161 (3)	0.41261 (12)	-0.03600 (16)	0.0421 (7)*
C9	-0.2746 (2)	0.46912 (13)	-0.01690 (18)	0.0421 (7)*
C10	-0.2409 (2)	0.52330 (8)	0.04430 (9)	0.0421 (7)*
N1	0.62344 (14)	0.76268 (5)	0.07509 (5)	0.0421 (7)*
C11	0.78013 (14)	0.89484 (5)	0.10073 (6)	0.0421 (7)*
C12	0.58697 (19)	0.83985 (7)	0.11586 (7)	0.0421 (7)*
C13	0.54484 (18)	0.82600 (11)	0.20304 (8)	0.0421 (7)*
C14	0.70957 (17)	0.79300 (11)	0.25178 (6)	0.0421 (7)*
C15	0.66760 (18)	0.78130 (10)	0.33136 (6)	0.0421 (7)*
C16	0.46837 (18)	0.80664 (12)	0.36380 (6)	0.0421 (7)*
C17	0.30689 (18)	0.83888 (11)	0.31627 (6)	0.0421 (7)*
C18	0.34622 (17)	0.84983 (10)	0.23623 (7)	0.0421 (7)*
H2	-0.1337 (9)	0.6075 (3)	0.1667 (3)	0.0760*
H3A	0.0084 (7)	0.5056 (3)	0.2389 (3)	0.0760*
H3B	0.2445 (10)	0.5257 (3)	0.2071 (3)	0.0760*
H4A	-0.0122 (8)	0.6269 (3)	0.3047 (3)	0.0760*
H4B	0.2255 (8)	0.6525 (3)	0.2712 (3)	0.0760*
H4C	0.1912 (9)	0.5830 (3)	0.3361 (3)	0.0760*
H6	0.2528 (8)	0.4653 (3)	0.0942 (3)	0.0760*
H7	0.1872 (7)	0.3724 (2)	-0.0051 (3)	0.0760*
H8	-0.1355 (9)	0.3764 (3)	-0.0787 (3)	0.0760*
H9	-0.4167 (8)	0.4686 (2)	-0.0417 (3)	0.0760*
H10	-0.3532 (8)	0.5609 (3)	0.0570 (3)	0.0760*
H1NA	0.4916 (9)	0.7307 (3)	0.0781 (3)	0.0760*
H1NB	0.7429 (10)	0.7305 (3)	0.0970 (3)	0.0760*
H1NC	0.6548 (12)	0.7730 (3)	0.0206 (3)	0.0760*
H11A	0.7480 (8)	0.9464 (3)	0.1241 (3)	0.0760*
H11B	0.9131 (9)	0.8714 (3)	0.1224 (3)	0.0760*
H11C	0.7980 (7)	0.9015 (3)	0.0449 (3)	0.0760*
H12	0.4565 (9)	0.8623 (3)	0.0938 (3)	0.0760*
H14	0.8497 (10)	0.7792 (3)	0.2291 (3)	0.0760*
H15	0.7892 (12)	0.7620 (3)	0.3634 (3)	0.0760*
H16	0.4371 (11)	0.7971 (3)	0.4182 (3)	0.0760*
H17	0.1700 (9)	0.8569 (3)	0.3396 (3)	0.0760*
H18	0.2294 (9)	0.8672 (3)	0.2010 (3)	0.0760*

Geometric parameters (Å, °)

O1—C1	1.282 (3)	C6—H6	0.949 (5)
O2—C1	1.227 (3)	C7—H7	0.955 (4)
N1—C12	1.4831 (14)	C8—H8	0.950 (6)
N1—H1NC	0.956 (5)	C9—H9	0.958 (5)
N1—H1NA	0.964 (5)	C10—H10	0.953 (5)
N1—H1NB	0.976 (6)	C11—C12	1.5125 (14)
C1—C2	1.5069 (17)	C12—C13	1.5123 (18)
C2—C3	1.5252 (17)	C13—C18	1.3870 (17)
C2—C5	1.504 (2)	C13—C14	1.4076 (18)
C3—C4	1.5326 (14)	C14—C15	1.3818 (15)
C5—C6	1.390 (2)	C15—C16	1.3927 (17)
C5—C10	1.3885 (19)	C16—C17	1.3767 (17)
C6—C7	1.390 (3)	C17—C18	1.3848 (16)
C7—C8	1.394 (3)	C11—H11A	0.971 (5)
C8—C9	1.388 (3)	C11—H11B	0.969 (5)
C9—C10	1.392 (3)	C11—H11C	0.956 (5)
C2—H2	0.954 (5)	C12—H12	0.952 (5)
C3—H3B	0.955 (6)	C14—H14	0.960 (6)
C3—H3A	0.952 (5)	C15—H15	0.970 (7)
C4—H4A	0.965 (5)	C16—H16	0.952 (5)
C4—H4B	0.973 (5)	C17—H17	0.967 (5)
C4—H4C	0.970 (5)	C18—H18	0.970 (5)
H1NA—N1—H1NB	106.7 (5)	C8—C7—H7	118.1 (3)
H1NA—N1—H1NC	108.5 (5)	C9—C8—H8	121.8 (4)
H1NB—N1—H1NC	108.5 (5)	C7—C8—H8	120.0 (4)
C12—N1—H1NC	108.6 (3)	C10—C9—H9	117.6 (3)
C12—N1—H1NA	109.8 (3)	C8—C9—H9	121.0 (3)
C12—N1—H1NB	114.7 (3)	C5—C10—H10	119.9 (3)
O2—C1—C2	119.36 (18)	C9—C10—H10	119.7 (3)
O1—C1—O2	123.6 (2)	C11—C12—C13	112.89 (10)
O1—C1—C2	116.92 (14)	N1—C12—C13	110.06 (11)
C1—C2—C3	110.29 (10)	N1—C12—C11	109.80 (9)
C3—C2—C5	111.74 (10)	C12—C13—C14	120.66 (10)
C1—C2—C5	111.02 (12)	C12—C13—C18	119.72 (11)
C2—C3—C4	111.22 (10)	C14—C13—C18	119.53 (12)
C6—C5—C10	118.57 (14)	C13—C14—C15	119.61 (11)
C2—C5—C6	119.88 (12)	C14—C15—C16	119.93 (11)
C2—C5—C10	121.51 (12)	C15—C16—C17	120.48 (10)
C5—C6—C7	120.82 (12)	C16—C17—C18	119.92 (10)
C6—C7—C8	120.68 (14)	C13—C18—C17	120.38 (12)
C7—C8—C9	118.2 (2)	C12—C11—H11A	108.6 (3)
C8—C9—C10	121.15 (17)	C12—C11—H11B	109.4 (3)
C5—C10—C9	120.48 (13)	C12—C11—H11C	109.0 (3)
C5—C2—H2	109.3 (3)	H11A—C11—H11B	112.0 (4)
C1—C2—H2	108.5 (3)	H11A—C11—H11C	108.6 (4)

C3—C2—H2	105.8 (3)	H11B—C11—H11C	109.0 (4)
C2—C3—H3A	106.1 (3)	N1—C12—H12	106.7 (3)
C2—C3—H3B	106.9 (3)	C11—C12—H12	109.6 (3)
C4—C3—H3A	110.7 (3)	C13—C12—H12	107.6 (3)
C4—C3—H3B	111.4 (3)	C13—C14—H14	119.3 (3)
H3A—C3—H3B	110.4 (4)	C15—C14—H14	121.1 (3)
H4A—C4—H4B	112.0 (4)	C14—C15—H15	116.8 (4)
H4A—C4—H4C	104.6 (4)	C16—C15—H15	122.8 (4)
C3—C4—H4C	109.6 (3)	C15—C16—H16	120.1 (4)
C3—C4—H4A	108.2 (3)	C17—C16—H16	119.2 (4)
C3—C4—H4B	110.0 (3)	C16—C17—H17	119.7 (3)
H4B—C4—H4C	112.3 (4)	C18—C17—H17	120.3 (3)
C7—C6—H6	117.8 (3)	C13—C18—H18	118.2 (3)
C5—C6—H6	121.4 (3)	C17—C18—H18	120.9 (3)
C6—C7—H7	121.0 (3)		
O1—C1—C2—C5	89.7 (3)	C7—C8—C9—C10	1.2 (3)
O2—C1—C2—C3	38.2 (3)	C8—C9—C10—C5	1.5 (3)
O2—C1—C2—C5	-86.2 (3)	N1—C12—C13—C14	64.12 (18)
C1—C2—C3—C4	61.18 (12)	N1—C12—C13—C18	-119.26 (15)
C5—C2—C3—C4	-174.82 (9)	C11—C12—C13—C14	-58.95 (19)
C1—C2—C5—C6	78.06 (16)	C11—C12—C13—C18	117.68 (14)
C1—C2—C5—C10	-99.50 (15)	C12—C13—C14—C15	179.65 (15)
C3—C2—C5—C6	-45.52 (17)	C18—C13—C14—C15	3.0 (3)
C3—C2—C5—C10	136.93 (14)	C12—C13—C18—C17	-178.80 (14)
C2—C5—C6—C7	-178.62 (15)	C14—C13—C18—C17	-2.1 (3)
C10—C5—C6—C7	-1.0 (2)	C13—C14—C15—C16	-4.0 (3)
C2—C5—C10—C9	175.95 (16)	C14—C15—C16—C17	4.0 (3)
C6—C5—C10—C9	-1.6 (2)	C15—C16—C17—C18	-3.1 (3)
C5—C6—C7—C8	3.8 (3)	C16—C17—C18—C13	2.2 (3)
C6—C7—C8—C9	-3.8 (3)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1NA \cdots O2	0.964 (5)	1.863 (6)	2.732 (3)	148.5 (5)
N1—H1NB \cdots O1 ⁱ	0.976 (6)	1.907 (7)	2.744 (3)	142.2 (5)
N1—H1NC \cdots O1 ⁱⁱ	0.956 (5)	1.932 (7)	2.797 (5)	149.4 (6)
C10—H10 \cdots O2 ⁱⁱⁱ	0.953 (5)	2.492 (6)	3.426 (3)	166.8 (4)

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