

## 1-Chloro-1-[(4-methylphenyl)hydrazinylidene]propan-2-one

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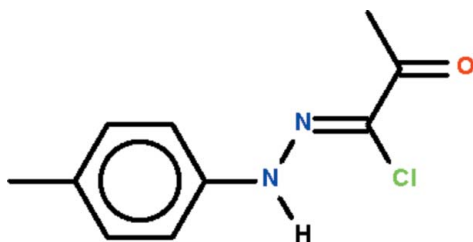
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Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(\text{C}-\text{C}) = 0.005$  Å;  $R$  factor = 0.045;  $wR$  factor = 0.125; data-to-parameter ratio = 10.0.

The asymmetric unit of the title compound,  $\text{C}_{10}\text{H}_{11}\text{ClN}_2\text{O}$ , contains two molecules. The non-H atoms of each molecule lie approximately on a plane (r.m.s. deviations = 0.062 and 0.110 Å), and the  $\text{C}=\text{N}$  double bond has a  $Z$ -configuration in both independent molecules. In the crystal, adjacent molecules are linked by  $\text{N}-\text{H}\cdots\text{O}_{\text{carbonyl}}$  hydrogen bonds, forming chains running along [100].

### Related literature

For the synthesis, see: Benincori *et al.* (1990); Sayed *et al.* (2002). For background to the title compound, see: Asiri *et al.* (2010).



### Experimental

#### Crystal data

$\text{C}_{10}\text{H}_{11}\text{ClN}_2\text{O}$   
 $M_r = 210.66$

Monoclinic,  $P2_1$   
 $a = 11.0572$  (3) Å

$b = 7.6570$  (2) Å  
 $c = 12.4613$  (3) Å  
 $\beta = 105.063$  (3)°  
 $V = 1018.79$  (5) Å<sup>3</sup>  
 $Z = 4$

Cu  $K\alpha$  radiation  
 $\mu = 3.06$  mm<sup>-1</sup>  
 $T = 100$  K  
 $0.20 \times 0.02 \times 0.02$  mm

#### Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector  
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)  
 $T_{\text{min}} = 0.580$ ,  $T_{\text{max}} = 0.941$

3467 measured reflections  
2667 independent reflections  
2516 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.021$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.045$   
 $wR(F^2) = 0.125$   
 $S = 1.06$   
2667 reflections  
266 parameters  
1 restraint

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\text{max}} = 0.31$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.42$  e Å<sup>-3</sup>  
Absolute structure: Flack (1983), 533 Friedel pairs  
Flack parameter: 0.17 (2)

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N2}-\text{H2}\cdots\text{O2}$	0.88 (5)	2.12 (5)	2.975 (4)	162 (4)
$\text{N4}-\text{H4}\cdots\text{O1}^i$	0.83 (5)	2.12 (5)	2.909 (4)	159 (4)

Symmetry code: (i)  $x + 1, y, z$ .

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: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5262).

### References

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**supplementary materials**

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## 1-Chloro-1-[(4-methylphenyl)hydrazinylidene]propan-2-one

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### Comment

We have previously reported the synthesis of ethyl (*Z*)-2-chloro-2-(2-phenylhydrazin-1-ylidene) acetate by the reaction of benzenediazonium chloride with ethyl 2-chloro-3-oxobutanoate (Asiri *et al.*, 2010). The compound is an ester. In the present study, the use of a substituted benzenediazonium chloride and the methyl ester (instead of the ethyl ester) afforded a 1-chloro-1-(arylhydrazono)-2-propanone. Such ketones are intermediates in the synthesis of pyrazoles (Sayed *et al.*, 2002) and other heterocycles (Benincori *et al.*, 1990). In the 4-methyl substituted compound (Scheme I, Fig. 1), the non-hydrogen atoms lie on a plane [r.m.s. deviation 0.062 and 0.110 Å in the two independent molecules]. (Scheme I, Fig. 1). The C<sub>aryl</sub>-N(H)-N=C(S)=O portion adopts an extended zigzag conformation. Adjacent molecules are linked by an *N*-H...O<sub>carbonyl</sub> hydrogen bond to form a chain running [1 0 0].

### Experimental

To a stirred solution of methyl 2-chloro-3-oxobutanoate (1.64 g, 10 mmol) in ethanol (100 ml) was added sodium acetate trihydrate (1.30 g, 10 mmol). The mixture was chilled to 273 K and then treated with a cold solution of *p*-nitrobenzenediazonium chloride, prepared by diazotizing *p*-methylaniline (1.07 g, 10 mmol) dissolved in 6*M* hydrochloric acid (6 ml) with a solution of sodium nitrite (0.70 g, 10 mmol) in water (10 ml). The addition of the diazonium salt solution was carried out with rapid stirring over a period of 20 min. The reaction mixture was stirred for further 15 min. and left for 3 h in refrigerator. The resulting solid was collected by filtration and washed thoroughly with water. The crude product was crystallized from ethanol to give the corresponding hydrazoneyl chloride.

### Refinement

Carbon-bound H-atoms were placed in calculated positions [C—H 0.95 to 0.98 Å,  $U_{\text{iso}}(\text{H})$  1.2 to 1.5 $U_{\text{eq}}(\text{C})$ ] and were included in the refinement in the riding model approximation.

The amino H-atom was located in a difference Fourier map, and was freely refined.

The Flack parameter was refined from 533 Friedel pairs.

Omitted from the refinement was the (2 - 2 2) reflection.

## Figures

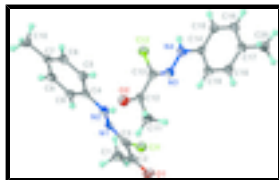


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of the two independent molecules of  $C_{10}H_{11}ClN_2O$  at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

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### Crystal data

$C_{10}H_{11}ClN_2O$	$F(000) = 440$
$M_r = 210.66$	$D_x = 1.373 \text{ Mg m}^{-3}$
Monoclinic, $P2_1$	Cu $K\alpha$ radiation, $\lambda = 1.54184 \text{ \AA}$
Hall symbol: P 2yb	Cell parameters from 1695 reflections
$a = 11.0572 (3) \text{ \AA}$	$\theta = 3.7\text{--}74.0^\circ$
$b = 7.6570 (2) \text{ \AA}$	$\mu = 3.06 \text{ mm}^{-1}$
$c = 12.4613 (3) \text{ \AA}$	$T = 100 \text{ K}$
$\beta = 105.063 (3)^\circ$	Prism, yellow
$V = 1018.79 (5) \text{ \AA}^3$	$0.20 \times 0.02 \times 0.02 \text{ mm}$
$Z = 4$	

### Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector	2667 independent reflections
Radiation source: SuperNova (Cu) X-ray Source	2516 reflections with $I > 2\sigma(I)$
Mirror	$R_{\text{int}} = 0.021$
Detector resolution: $10.4041 \text{ pixels mm}^{-1}$	$\theta_{\text{max}} = 74.2^\circ$ , $\theta_{\text{min}} = 3.7^\circ$
$\omega$ scan	$h = -13 \rightarrow 7$
Absorption correction: multi-scan (CrysAlis PRO; Agilent, 2010)	$k = -9 \rightarrow 9$
$T_{\text{min}} = 0.580$ , $T_{\text{max}} = 0.941$	$l = -13 \rightarrow 15$
3467 measured reflections	

### Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.045$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.125$	$w = 1/[\sigma^2(F_o^2) + (0.0867P)^2 + 0.3419P]$
$S = 1.06$	where $P = (F_o^2 + 2F_c^2)/3$
2667 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
	$\Delta\rho_{\text{max}} = 0.31 \text{ e \AA}^{-3}$

266 parameters

$$\Delta\rho_{\min} = -0.42 \text{ e } \text{\AA}^{-3}$$

1 restraint

Absolute structure: Flack (1983), 533 Friedel pairs

Primary atom site location: structure-invariant direct methods

Flack parameter: 0.17 (2)

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.22799 (7)	0.50001 (13)	0.90675 (6)	0.0252 (2)
C12	0.75288 (7)	0.52179 (14)	1.00248 (6)	0.0252 (2)
N1	0.2864 (2)	0.4827 (4)	1.1276 (2)	0.0187 (6)
N2	0.3920 (3)	0.4043 (4)	1.1235 (2)	0.0195 (6)
H2	0.416 (4)	0.391 (7)	1.062 (4)	0.035 (13)*
N3	0.7037 (2)	0.4842 (4)	0.7819 (2)	0.0193 (6)
N4	0.8068 (3)	0.5762 (4)	0.7850 (2)	0.0201 (6)
H4	0.853 (5)	0.615 (7)	0.844 (4)	0.035 (13)*
O1	0.0213 (2)	0.6872 (4)	0.9612 (2)	0.0297 (7)
O2	0.5199 (3)	0.3196 (4)	0.9474 (2)	0.0281 (6)
C1	0.0693 (3)	0.6493 (6)	1.1582 (3)	0.0253 (8)
H1A	0.0074	0.7420	1.1556	0.038*
H1B	0.1477	0.6800	1.2125	0.038*
H1C	0.0373	0.5391	1.1800	0.038*
C2	0.0928 (3)	0.6290 (6)	1.0454 (3)	0.0225 (8)
C3	0.2071 (3)	0.5347 (5)	1.0390 (3)	0.0207 (7)
C4	0.4745 (3)	0.3415 (5)	1.2216 (3)	0.0177 (7)
C5	0.5915 (3)	0.2783 (5)	1.2169 (3)	0.0207 (7)
H5	0.6156	0.2824	1.1491	0.025*
C6	0.6723 (3)	0.2097 (5)	1.3117 (3)	0.0212 (7)
H6	0.7515	0.1664	1.3077	0.025*
C7	0.6406 (3)	0.2026 (5)	1.4124 (3)	0.0200 (7)
C8	0.5243 (3)	0.2683 (5)	1.4160 (3)	0.0205 (8)
H8	0.5011	0.2654	1.4842	0.025*
C9	0.4416 (3)	0.3377 (5)	1.3228 (3)	0.0190 (7)
H9	0.3630	0.3825	1.3274	0.023*
C10	0.7273 (3)	0.1199 (6)	1.5139 (3)	0.0255 (8)
H10A	0.7083	0.1659	1.5810	0.038*
H10B	0.8143	0.1473	1.5154	0.038*
H10C	0.7155	-0.0070	1.5108	0.038*
C11	0.4814 (4)	0.2920 (5)	0.7503 (3)	0.0240 (8)
H11A	0.4059	0.2297	0.7562	0.036*
H11B	0.5335	0.2139	0.7190	0.036*
H11C	0.4573	0.3938	0.7019	0.036*
C12	0.5538 (3)	0.3510 (5)	0.8636 (3)	0.0203 (8)
C13	0.6703 (3)	0.4509 (5)	0.8711 (3)	0.0194 (7)
C14	0.8352 (3)	0.6173 (5)	0.6841 (3)	0.0176 (7)
C15	0.9434 (3)	0.7145 (5)	0.6877 (3)	0.0206 (7)
H15	0.9979	0.7478	0.7569	0.025*
C16	0.9699 (3)	0.7618 (5)	0.5883 (3)	0.0220 (8)

## supplementary materials

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H16	1.0423	0.8302	0.5907	0.026*
C17	0.8940 (3)	0.7123 (5)	0.4861 (3)	0.0204 (7)
C18	0.7876 (3)	0.6112 (5)	0.4846 (3)	0.0209 (7)
H18	0.7349	0.5739	0.4154	0.025*
C19	0.7581 (3)	0.5651 (5)	0.5822 (3)	0.0195 (7)
H19	0.6853	0.4977	0.5796	0.023*
C20	0.9210 (4)	0.7668 (6)	0.3778 (3)	0.0263 (8)
H20A	0.8425	0.7988	0.3241	0.040*
H20B	0.9602	0.6695	0.3484	0.040*
H20C	0.9778	0.8673	0.3910	0.040*

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C11	0.0246 (4)	0.0350 (5)	0.0168 (4)	-0.0019 (4)	0.0070 (3)	0.0016 (4)
C12	0.0246 (4)	0.0320 (5)	0.0193 (4)	-0.0022 (4)	0.0065 (3)	-0.0005 (4)
N1	0.0137 (12)	0.0207 (17)	0.0231 (13)	-0.0041 (12)	0.0074 (11)	-0.0008 (13)
N2	0.0191 (14)	0.0255 (17)	0.0158 (13)	-0.0014 (13)	0.0082 (12)	0.0000 (12)
N3	0.0170 (13)	0.0199 (17)	0.0230 (13)	0.0019 (12)	0.0089 (11)	0.0016 (13)
N4	0.0201 (14)	0.0242 (16)	0.0176 (14)	-0.0045 (12)	0.0079 (12)	-0.0019 (13)
O1	0.0162 (12)	0.046 (2)	0.0253 (14)	-0.0022 (12)	0.0019 (11)	0.0054 (13)
O2	0.0251 (13)	0.0375 (17)	0.0263 (14)	-0.0004 (12)	0.0146 (11)	0.0041 (12)
C1	0.0190 (16)	0.034 (2)	0.0246 (18)	0.0011 (16)	0.0089 (15)	0.0000 (17)
C2	0.0163 (15)	0.030 (2)	0.0212 (17)	-0.0053 (16)	0.0053 (14)	-0.0001 (16)
C3	0.0189 (15)	0.025 (2)	0.0194 (15)	-0.0068 (16)	0.0079 (13)	-0.0007 (15)
C4	0.0165 (16)	0.0152 (17)	0.0218 (17)	-0.0030 (14)	0.0057 (14)	-0.0003 (14)
C5	0.0194 (17)	0.0233 (19)	0.0217 (17)	0.0007 (15)	0.0096 (14)	-0.0010 (15)
C6	0.0189 (16)	0.0219 (17)	0.0245 (18)	-0.0011 (15)	0.0088 (14)	-0.0003 (15)
C7	0.0195 (16)	0.0182 (17)	0.0206 (17)	-0.0037 (14)	0.0024 (14)	0.0001 (14)
C8	0.0214 (17)	0.0245 (19)	0.0181 (16)	-0.0041 (15)	0.0099 (14)	-0.0036 (15)
C9	0.0172 (16)	0.0243 (18)	0.0172 (16)	-0.0021 (14)	0.0072 (14)	-0.0012 (14)
C10	0.0229 (17)	0.027 (2)	0.0252 (18)	0.0014 (16)	0.0043 (15)	0.0060 (16)
C11	0.0217 (18)	0.028 (2)	0.0217 (18)	-0.0005 (16)	0.0042 (15)	-0.0003 (16)
C12	0.0199 (17)	0.0193 (18)	0.0255 (18)	0.0056 (15)	0.0129 (15)	0.0032 (15)
C13	0.0219 (17)	0.0207 (18)	0.0167 (15)	0.0034 (14)	0.0068 (14)	0.0020 (13)
C14	0.0174 (15)	0.0181 (18)	0.0185 (16)	0.0023 (14)	0.0067 (13)	0.0013 (14)
C15	0.0196 (16)	0.0199 (17)	0.0223 (17)	0.0014 (14)	0.0057 (14)	0.0016 (15)
C16	0.0149 (15)	0.0212 (19)	0.032 (2)	0.0006 (15)	0.0104 (15)	0.0019 (16)
C17	0.0218 (17)	0.0204 (17)	0.0220 (17)	0.0023 (15)	0.0108 (14)	0.0014 (15)
C18	0.0198 (16)	0.0244 (19)	0.0186 (16)	0.0049 (15)	0.0050 (14)	0.0002 (14)
C19	0.0171 (15)	0.0189 (18)	0.0245 (16)	0.0000 (13)	0.0087 (14)	-0.0015 (14)
C20	0.0234 (18)	0.035 (2)	0.0237 (18)	0.0013 (17)	0.0109 (15)	0.0076 (17)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

C11—C3	1.743 (3)	C8—C9	1.384 (5)
C12—C13	1.742 (4)	C8—H8	0.9500
N1—C3	1.281 (4)	C9—H9	0.9500
N1—N2	1.326 (4)	C10—H10A	0.9800

N2—C4	1.407 (5)	C10—H10B	0.9800
N2—H2	0.88 (5)	C10—H10C	0.9800
N3—C13	1.283 (4)	C11—C12	1.500 (5)
N3—N4	1.332 (4)	C11—H11A	0.9800
N4—C14	1.409 (4)	C11—H11B	0.9800
N4—H4	0.83 (5)	C11—H11C	0.9800
O1—C2	1.221 (5)	C12—C13	1.480 (5)
O2—C12	1.222 (4)	C14—C19	1.391 (5)
C1—C2	1.502 (5)	C14—C15	1.399 (5)
C1—H1A	0.9800	C15—C16	1.393 (5)
C1—H1B	0.9800	C15—H15	0.9500
C1—H1C	0.9800	C16—C17	1.383 (5)
C2—C3	1.477 (5)	C16—H16	0.9500
C4—C5	1.397 (5)	C17—C18	1.404 (5)
C4—C9	1.399 (5)	C17—C20	1.515 (5)
C5—C6	1.386 (5)	C18—C19	1.385 (5)
C5—H5	0.9500	C18—H18	0.9500
C6—C7	1.389 (5)	C19—H19	0.9500
C6—H6	0.9500	C20—H20A	0.9800
C7—C8	1.391 (5)	C20—H20B	0.9800
C7—C10	1.513 (5)	C20—H20C	0.9800
C3—N1—N2	121.3 (3)	H10A—C10—H10B	109.5
N1—N2—C4	120.0 (3)	C7—C10—H10C	109.5
N1—N2—H2	123 (3)	H10A—C10—H10C	109.5
C4—N2—H2	117 (3)	H10B—C10—H10C	109.5
C13—N3—N4	121.1 (3)	C12—C11—H11A	109.5
N3—N4—C14	118.7 (3)	C12—C11—H11B	109.5
N3—N4—H4	123 (3)	H11A—C11—H11B	109.5
C14—N4—H4	118 (3)	C12—C11—H11C	109.5
C2—C1—H1A	109.5	H11A—C11—H11C	109.5
C2—C1—H1B	109.5	H11B—C11—H11C	109.5
H1A—C1—H1B	109.5	O2—C12—C13	120.2 (3)
C2—C1—H1C	109.5	O2—C12—C11	122.5 (3)
H1A—C1—H1C	109.5	C13—C12—C11	117.3 (3)
H1B—C1—H1C	109.5	N3—C13—C12	119.3 (3)
O1—C2—C3	120.3 (3)	N3—C13—C12	123.6 (3)
O1—C2—C1	122.3 (3)	C12—C13—C12	117.1 (3)
C3—C2—C1	117.4 (3)	C19—C14—C15	119.9 (3)
N1—C3—C2	120.6 (3)	C19—C14—N4	121.6 (3)
N1—C3—C11	122.6 (3)	C15—C14—N4	118.5 (3)
C2—C3—C11	116.8 (3)	C16—C15—C14	119.0 (3)
C5—C4—C9	119.4 (3)	C16—C15—H15	120.5
C5—C4—N2	118.7 (3)	C14—C15—H15	120.5
C9—C4—N2	121.9 (3)	C17—C16—C15	122.0 (3)
C6—C5—C4	119.6 (3)	C17—C16—H16	119.0
C6—C5—H5	120.2	C15—C16—H16	119.0
C4—C5—H5	120.2	C16—C17—C18	118.0 (3)
C5—C6—C7	121.8 (3)	C16—C17—C20	122.1 (3)
C5—C6—H6	119.1	C18—C17—C20	119.9 (3)

## supplementary materials

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C7—C6—H6	119.1	C19—C18—C17	121.1 (3)
C6—C7—C8	117.8 (3)	C19—C18—H18	119.5
C6—C7—C10	121.1 (3)	C17—C18—H18	119.5
C8—C7—C10	121.0 (3)	C18—C19—C14	120.0 (3)
C9—C8—C7	121.8 (3)	C18—C19—H19	120.0
C9—C8—H8	119.1	C14—C19—H19	120.0
C7—C8—H8	119.1	C17—C20—H20A	109.5
C8—C9—C4	119.6 (3)	C17—C20—H20B	109.5
C8—C9—H9	120.2	H20A—C20—H20B	109.5
C4—C9—H9	120.2	C17—C20—H20C	109.5
C7—C10—H10A	109.5	H20A—C20—H20C	109.5
C7—C10—H10B	109.5	H20B—C20—H20C	109.5
C3—N1—N2—C4	-177.1 (3)	N2—C4—C9—C8	177.5 (4)
C13—N3—N4—C14	-176.5 (3)	N4—N3—C13—C12	178.9 (3)
N2—N1—C3—C2	-177.1 (3)	N4—N3—C13—C12	0.6 (5)
N2—N1—C3—C11	2.5 (5)	O2—C12—C13—N3	-179.3 (3)
O1—C2—C3—N1	175.7 (4)	C11—C12—C13—N3	0.7 (5)
C1—C2—C3—N1	-4.4 (5)	O2—C12—C13—C12	-0.9 (5)
O1—C2—C3—C11	-3.9 (5)	C11—C12—C13—C12	179.1 (3)
C1—C2—C3—C11	176.0 (3)	N3—N4—C14—C19	0.4 (5)
N1—N2—C4—C5	-172.5 (3)	N3—N4—C14—C15	179.8 (3)
N1—N2—C4—C9	8.7 (5)	C19—C14—C15—C16	1.9 (5)
C9—C4—C5—C6	1.3 (6)	N4—C14—C15—C16	-177.5 (3)
N2—C4—C5—C6	-177.6 (3)	C14—C15—C16—C17	-1.5 (6)
C4—C5—C6—C7	-0.4 (6)	C15—C16—C17—C18	0.0 (6)
C5—C6—C7—C8	-0.5 (6)	C15—C16—C17—C20	178.7 (4)
C5—C6—C7—C10	177.2 (4)	C16—C17—C18—C19	1.1 (5)
C6—C7—C8—C9	0.5 (6)	C20—C17—C18—C19	-177.6 (4)
C10—C7—C8—C9	-177.2 (4)	C17—C18—C19—C14	-0.7 (5)
C7—C8—C9—C4	0.5 (6)	C15—C14—C19—C18	-0.9 (5)
C5—C4—C9—C8	-1.3 (6)	N4—C14—C19—C18	178.5 (3)

### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H2 $\cdots$ O2	0.88 (5)	2.12 (5)	2.975 (4)	162 (4)
N4—H4 $\cdots$ O1 <sup>i</sup>	0.83 (5)	2.12 (5)	2.909 (4)	159 (4)

Symmetry codes: (i)  $x+1, y, z$ .



Fig. 1

