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3-[(3,4-Dichlorophenyl)aminocarbonyl]-propionic acid monohydrate

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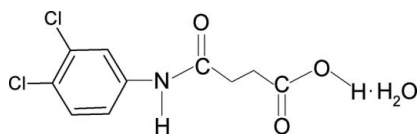
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Key indicators: single-crystal X-ray study; $T = 299$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.083; wR factor = 0.212; data-to-parameter ratio = 12.7.

In the crystal structure of the title compound, $\text{C}_{10}\text{H}_9\text{Cl}_2\text{NO}_3 \cdot \text{H}_2\text{O}$, the conformations of the amide O atom and the carbonyl O atom of the acid segment are *anti* to the H atoms of adjacent $-\text{CH}_2$ groups. In the crystal, the molecules are linked into a three-dimensional network through $\text{N}-\text{H} \cdots \text{O}$ and $\text{O}-\text{H} \cdots \text{O}$ intermolecular hydrogen bonds.

Related literature

For related structures, see: Gowda *et al.* (2009a,b,c). For hydrogen bonds involving carboxylic acids, see: Jagannathan *et al.* (1994); Leiserowitz (1976). For the modeling of water H atoms, see: Nardelli (1999).



Experimental

Crystal data

$\text{C}_{10}\text{H}_9\text{Cl}_2\text{NO}_3 \cdot \text{H}_2\text{O}$
 $M_r = 280.10$
Monoclinic, $P2_1/c$
 $a = 9.5634$ (9) Å
 $b = 7.4527$ (7) Å
 $c = 17.292$ (2) Å
 $\beta = 104.35$ (2)°

$V = 1194.0$ (2) Å³
 $Z = 4$
Cu $K\alpha$ radiation
 $\mu = 4.95$ mm⁻¹
 $T = 299$ K
 $0.55 \times 0.50 \times 0.40$ mm

Data collection

Enraf-Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.098$, $T_{\max} = 0.138$
2508 measured reflections

2129 independent reflections
2052 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.072$
3 standard reflections
frequency: 120 min
intensity decay: 1.0%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.083$
 $wR(F^2) = 0.212$
 $S = 1.08$
2129 reflections
167 parameters
5 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.92$ e Å⁻³
 $\Delta\rho_{\min} = -0.69$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{O2}-\text{H2O} \cdots \text{O4}^i$	0.88 (3)	1.79 (3)	2.672 (4)	177 (5)
$\text{N1}-\text{H1N} \cdots \text{O3}^{ii}$	0.84 (3)	2.11 (3)	2.941 (4)	168 (4)
$\text{O4}-\text{H41} \cdots \text{O1}^{iii}$	0.82 (3)	2.11 (4)	2.894 (4)	162 (5)
$\text{O4}-\text{H42} \cdots \text{O1}^{iv}$	0.84 (3)	2.09 (3)	2.881 (4)	156 (5)

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

Data collection: *CAD-4-PC* (Enraf-Nonius, 1996); cell refinement: *CAD-4-PC*; data reduction: *REDU4* (Stoe & Cie, 1987); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2838).

References

- Enraf-Nonius (1996). *CAD-4-PC*. Enraf-Nonius, Delft, The Netherlands.
Gowda, B. T., Foro, S., Saraswathi, B. S., Terao, H. & Fuess, H. (2009a). *Acta Cryst.* **E65**, o399.
Gowda, B. T., Foro, S., Saraswathi, B. S., Terao, H. & Fuess, H. (2009b). *Acta Cryst.* **E65**, o466.
Gowda, B. T., Foro, S., Saraswathi, B. S., Terao, H. & Fuess, H. (2009c). *Acta Cryst.* **E65**, o873.
Jagannathan, N. R., Rajan, S. S. & Subramanian, E. (1994). *J. Chem. Crystallogr.* **24**, 75–78.
Leiserowitz, L. (1976). *Acta Cryst.* **B32**, 775–802.
Nardelli, M. (1999). *J. Appl. Cryst.* **32**, 563–571.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
Stoe & Cie (1987). *REDU4*. Stoe & Cie GmbH, Darmstadt, Germany.

supporting information

Acta Cryst. (2009). E65, o1722 [doi:10.1107/S1600536809024519]

3-[(3,4-Dichlorophenyl)aminocarbonyl]propionic acid monohydrate

B. Thimme Gowda, Sabine Foro, B. S. Saraswathi and Hartmut Fuess

S1. Comment

As a part of studying the effect of ring and side chain substitutions on the structures of aromatic amides (Gowda *et al.*, 2009*a,b,c*), the crystal structure of *N*-(3,4-dichlorophenyl)succinamic acid monohydrate (I), systematic name: 3-[(3,4-dichloro)-aminocarbonyl]propionic acid monohydrate has been determined. The conformation of the N—H bond is *anti* to both the 3-chloro substituent in the aromatic ring and the C=O bond in the amide segment of the structure. Further, the amide O atom and the carbonyl O atom of the acid segment are *anti* to each other and are also *anti* to H atoms attached to the adjacent C atoms (Fig.1). Further, C=O and O—H bonds of the acid group are *syn* to each other, contrary to the *anti* position observed in 3-[(3,5-dichloro)-aminocarbonyl]propionic acid (Gowda *et al.*, 2009*c*). The observed *anti* position with the latter may be due to the hydrogen bond donated to the amide carbonyl group by the acid segment, which is prevented in the present structure due to the H-bonding effect of hydration. The N—H \cdots O and O—H \cdots O intermolecular hydrogen bonds link the molecules into a three-dimensional network (Table 1 and Fig.2).

The modes of interlinking carboxylic acids by hydrogen bonds is described elsewhere (Leiserowitz, 1976). The packing of molecules involving dimeric hydrogen-bonded association of each carboxyl group with a centrosymmetrically related neighbor has also been observed (Jagannathan *et al.*, 1994).

S2. Experimental

The solution of succinic anhydride (0.02 mol) in toluene (25 ml) was treated dropwise with the solution of 3,4-dichloroaniline (0.02 mol) also in toluene (20 ml) with constant stirring. The resulting mixture was stirred for about 1 h and set aside for an additional hour at room temperature for the completion of reaction. The mixture was then treated with dilute hydrochloric acid to remove the unreacted 3,4-dichloroaniline. The resultant solid *N*-(3,4-dichlorophenyl)-succinamic acid was filtered under suction and washed thoroughly with water to remove the unreacted succinic anhydride and succinic acid. It was recrystallized to constant melting point from ethanol. The purity of the compound was checked by elemental analysis and characterized by its infrared spectra. Single crystals used in X-ray diffraction studies were grown in an ethanol solution by slow evaporation at room temperature.

S3. Refinement

The O-bound and N-bound H atoms were located in a difference map. The positional parameters of the N-bound H atom were refined with N-H = 0.86 (4) Å and those of the O-bound (hydroxyl) H atom were refined with O-H distance restrained to 0.82 (4) Å. The positions of water H atoms were refined with restrained geometry (Nardelli, 1999) *viz.* O-H = 0.85 (4) Å and H \cdots H = 1.365 (4) Å. The other H atoms were positioned with idealized geometry using a riding model [C-H = 0.93–0.97 Å]. The isotropic displacement parameters of all H atoms were set to 1.2 times of the U_{eq} of the parent atom.

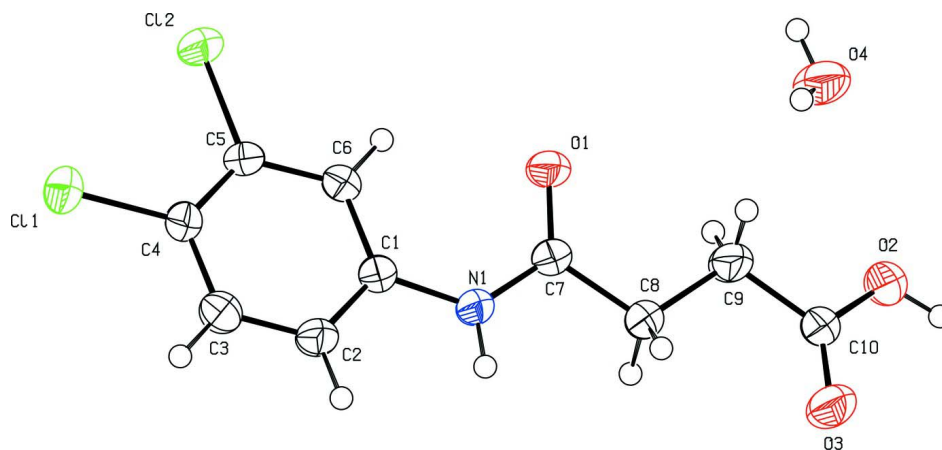


Figure 1

The molecular structure of the title compound, showing the atom labelling and the displacement ellipsoids are at the 50% probability level. H atoms are represented as small spheres of arbitrary radii.

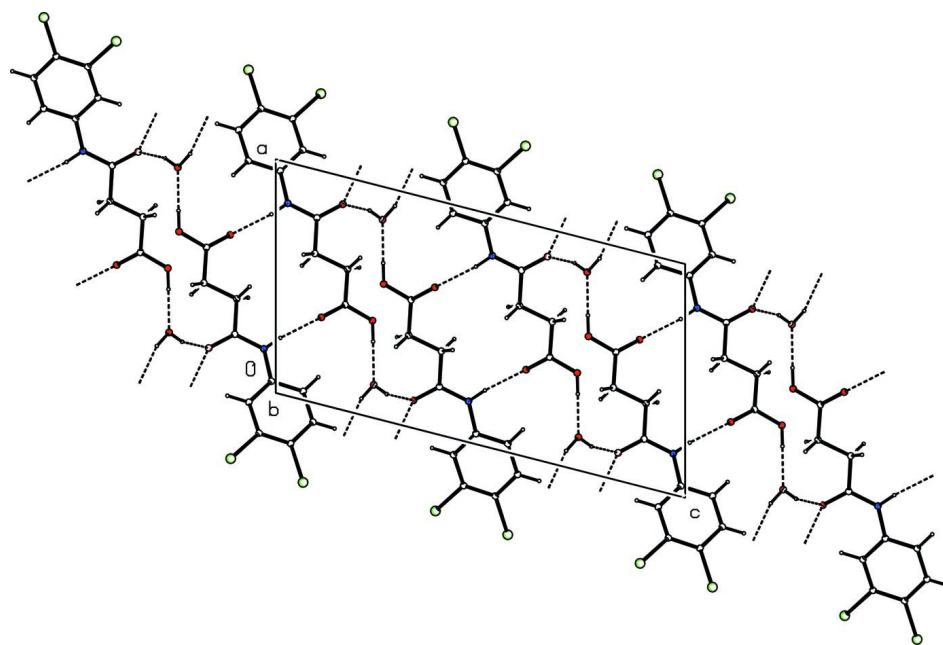


Figure 2

The crystal packing of the title compound, with hydrogen bonds shown as dashed lines.

3-[(3,4-Dichlorophenyl)aminocarbonyl]propionic acid monohydrate

Crystal data

$C_{10}H_9Cl_2NO_3 \cdot H_2O$

$M_r = 280.10$

Monoclinic, $P2_1/c$

Hall symbol: $-P 2_1/c$

$a = 9.5634 (9) \text{ \AA}$

$b = 7.4527 (7) \text{ \AA}$

$c = 17.292 (2) \text{ \AA}$

$\beta = 104.35 (2)^\circ$

$V = 1194.0 (2) \text{ \AA}^3$

$Z = 4$

$F(000) = 576$

$D_x = 1.558 \text{ Mg m}^{-3}$

Cu $K\alpha$ radiation, $\lambda = 1.54180 \text{ \AA}$

Cell parameters from 25 reflections

$\theta = 4.8\text{--}20.7^\circ$

$\mu = 4.95 \text{ mm}^{-1}$

$T = 299$ K $0.55 \times 0.50 \times 0.40$ mm
 Prism, colourless

Data collection

Enraf–Nonius CAD-4 diffractometer	2129 independent reflections 2052 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{\text{int}} = 0.072$
Graphite monochromator	$\theta_{\text{max}} = 67.0^\circ$, $\theta_{\text{min}} = 4.8^\circ$
$\omega/2\theta$ scans	$h = -11 \rightarrow 1$
Absorption correction: ψ scan (North <i>et al.</i> , 1968)	$k = -8 \rightarrow 0$
$T_{\text{min}} = 0.098$, $T_{\text{max}} = 0.138$	$l = -19 \rightarrow 20$
2508 measured reflections	3 standard reflections every 120 min intensity decay: 1.0%

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.083$	$w = 1/[\sigma^2(F_o^2) + (0.1684P)^2 + 0.6399P]$
$wR(F^2) = 0.212$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.08$	$(\Delta/\sigma)_{\text{max}} = 0.003$
2129 reflections	$\Delta\rho_{\text{max}} = 0.92 \text{ e } \text{\AA}^{-3}$
167 parameters	$\Delta\rho_{\text{min}} = -0.69 \text{ e } \text{\AA}^{-3}$
5 restraints	Extinction correction: <i>SHELXL97</i> (Sheldrick, 2008), $F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.014 (2)
Secondary atom site location: difference Fourier map	

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	−0.35278 (8)	0.10598 (12)	0.56662 (5)	0.0441 (4)
C12	−0.33021 (8)	0.03602 (12)	0.38996 (4)	0.0449 (4)
O1	0.1190 (2)	0.2952 (3)	0.33626 (13)	0.0417 (6)
O2	0.5845 (3)	0.4233 (4)	0.26373 (15)	0.0534 (7)
H2O	0.674 (4)	0.448 (6)	0.262 (3)	0.064*
O3	0.6264 (3)	0.5188 (4)	0.38871 (15)	0.0543 (7)
N1	0.1755 (3)	0.3197 (3)	0.47092 (15)	0.0331 (6)
H1N	0.240 (4)	0.353 (5)	0.511 (2)	0.040*
C1	0.0452 (3)	0.2667 (4)	0.48923 (17)	0.0301 (7)
C2	0.0368 (3)	0.2923 (4)	0.56697 (18)	0.0345 (7)
H2	0.1138	0.3446	0.6036	0.041*
C3	−0.0840 (3)	0.2416 (4)	0.59077 (18)	0.0367 (7)

H3	-0.0879	0.2582	0.6435	0.044*
C4	-0.2005 (3)	0.1652 (4)	0.53626 (17)	0.0319 (7)
C5	-0.1905 (3)	0.1364 (4)	0.45895 (17)	0.0315 (7)
C6	-0.0693 (3)	0.1877 (4)	0.43433 (17)	0.0325 (7)
H6	-0.0646	0.1696	0.3818	0.039*
C7	0.2060 (3)	0.3302 (4)	0.39971 (17)	0.0311 (7)
C8	0.3601 (3)	0.3889 (4)	0.40446 (18)	0.0360 (7)
H8A	0.3773	0.5034	0.4319	0.043*
H8B	0.4260	0.3022	0.4359	0.043*
C9	0.3923 (4)	0.4068 (7)	0.3247 (2)	0.0566 (11)
H9A	0.3294	0.4973	0.2939	0.068*
H9B	0.3717	0.2938	0.2963	0.068*
C10	0.5465 (3)	0.4577 (5)	0.33053 (19)	0.0400 (8)
O4	0.1421 (3)	0.9875 (5)	0.23932 (16)	0.0570 (8)
H41	0.070 (4)	0.945 (6)	0.209 (3)	0.068*
H42	0.116 (5)	1.059 (6)	0.271 (3)	0.068*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0340 (5)	0.0587 (6)	0.0425 (6)	-0.0077 (3)	0.0149 (4)	0.0020 (3)
C12	0.0329 (5)	0.0645 (7)	0.0332 (6)	-0.0134 (3)	0.0003 (4)	-0.0034 (3)
O1	0.0306 (11)	0.0668 (15)	0.0259 (11)	-0.0049 (10)	0.0035 (9)	-0.0020 (9)
O2	0.0391 (13)	0.0936 (19)	0.0297 (13)	-0.0109 (13)	0.0123 (10)	0.0014 (12)
O3	0.0375 (13)	0.0839 (19)	0.0395 (14)	-0.0159 (13)	0.0058 (11)	-0.0119 (13)
N1	0.0270 (12)	0.0455 (14)	0.0256 (13)	-0.0077 (10)	0.0045 (10)	-0.0018 (10)
C1	0.0263 (13)	0.0343 (14)	0.0291 (15)	0.0006 (10)	0.0057 (11)	0.0034 (11)
C2	0.0326 (15)	0.0411 (15)	0.0279 (15)	-0.0056 (12)	0.0039 (12)	-0.0048 (11)
C3	0.0392 (16)	0.0438 (16)	0.0281 (15)	-0.0024 (12)	0.0103 (12)	-0.0045 (12)
C4	0.0273 (14)	0.0380 (14)	0.0310 (14)	-0.0010 (11)	0.0085 (11)	0.0037 (11)
C5	0.0265 (14)	0.0372 (14)	0.0272 (14)	-0.0027 (11)	-0.0001 (11)	0.0022 (11)
C6	0.0313 (14)	0.0417 (16)	0.0237 (14)	-0.0022 (11)	0.0055 (11)	0.0017 (11)
C7	0.0255 (14)	0.0359 (14)	0.0301 (14)	0.0002 (11)	0.0039 (11)	0.0017 (11)
C8	0.0299 (15)	0.0489 (17)	0.0288 (16)	-0.0071 (12)	0.0063 (12)	0.0003 (12)
C9	0.0322 (18)	0.110 (3)	0.0274 (17)	-0.0167 (18)	0.0062 (13)	-0.0016 (17)
C10	0.0315 (16)	0.060 (2)	0.0281 (15)	-0.0061 (13)	0.0071 (12)	0.0040 (13)
O4	0.0364 (13)	0.094 (2)	0.0387 (14)	-0.0004 (13)	0.0064 (11)	-0.0163 (14)

Geometric parameters (Å, °)

C11—C4	1.723 (3)	C3—H3	0.93
C12—C5	1.726 (3)	C4—C5	1.381 (4)
O1—C7	1.229 (4)	C5—C6	1.384 (4)
O2—C10	1.319 (4)	C6—H6	0.93
O2—H2O	0.88 (3)	C7—C8	1.520 (4)
O3—C10	1.193 (4)	C8—C9	1.492 (5)
N1—C7	1.337 (4)	C8—H8A	0.97
N1—C1	1.415 (4)	C8—H8B	0.97

N1—H1N	0.84 (3)	C9—C10	1.501 (4)
C1—C2	1.380 (4)	C9—H9A	0.97
C1—C6	1.390 (4)	C9—H9B	0.97
C2—C3	1.372 (4)	O4—H41	0.82 (3)
C2—H2	0.93	O4—H42	0.84 (3)
C3—C4	1.390 (4)		
C10—O2—H2O	118 (3)	C1—C6—H6	120.5
C7—N1—C1	128.9 (2)	O1—C7—N1	123.8 (3)
C7—N1—H1N	117 (3)	O1—C7—C8	122.8 (3)
C1—N1—H1N	114 (3)	N1—C7—C8	113.4 (3)
C2—C1—C6	119.8 (3)	C9—C8—C7	113.3 (3)
C2—C1—N1	116.5 (3)	C9—C8—H8A	108.9
C6—C1—N1	123.6 (3)	C7—C8—H8A	108.9
C3—C2—C1	120.7 (3)	C9—C8—H8B	108.9
C3—C2—H2	119.6	C7—C8—H8B	108.9
C1—C2—H2	119.6	H8A—C8—H8B	107.7
C2—C3—C4	120.2 (3)	C8—C9—C10	112.6 (3)
C2—C3—H3	119.9	C8—C9—H9A	109.1
C4—C3—H3	119.9	C10—C9—H9A	109.1
C5—C4—C3	118.9 (3)	C8—C9—H9B	109.1
C5—C4—C11	121.4 (2)	C10—C9—H9B	109.1
C3—C4—C11	119.6 (2)	H9A—C9—H9B	107.8
C4—C5—C6	121.3 (3)	O3—C10—O2	123.7 (3)
C4—C5—C12	120.6 (2)	O3—C10—C9	124.5 (3)
C6—C5—C12	118.1 (2)	O2—C10—C9	111.8 (3)
C5—C6—C1	119.0 (3)	H41—O4—H42	109 (4)
C5—C6—H6	120.5		
C7—N1—C1—C2	-171.2 (3)	C4—C5—C6—C1	1.2 (4)
C7—N1—C1—C6	10.6 (5)	C12—C5—C6—C1	-179.2 (2)
C6—C1—C2—C3	-0.3 (5)	C2—C1—C6—C5	0.1 (4)
N1—C1—C2—C3	-178.6 (3)	N1—C1—C6—C5	178.3 (3)
C1—C2—C3—C4	-0.8 (5)	C1—N1—C7—O1	1.1 (5)
C2—C3—C4—C5	2.1 (5)	C1—N1—C7—C8	-178.6 (3)
C2—C3—C4—C11	-178.8 (3)	O1—C7—C8—C9	2.4 (5)
C3—C4—C5—C6	-2.3 (4)	N1—C7—C8—C9	-177.9 (3)
C11—C4—C5—C6	178.6 (2)	C7—C8—C9—C10	-177.7 (3)
C3—C4—C5—C12	178.1 (2)	C8—C9—C10—O3	-16.9 (6)
C11—C4—C5—C12	-1.0 (4)	C8—C9—C10—O2	161.8 (3)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O2—H2O \cdots O4 ⁱ	0.88 (3)	1.79 (3)	2.672 (4)	177 (5)
N1—H1N \cdots O3 ⁱⁱ	0.84 (3)	2.11 (3)	2.941 (4)	168 (4)

O4—H41···O1 ⁱⁱⁱ	0.82 (3)	2.11 (4)	2.894 (4)	162 (5)
O4—H42···O1 ^{iv}	0.84 (3)	2.09 (3)	2.881 (4)	156 (5)

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