

1-[3-(4-Nitrophenyl)propanoyl]urea acetic acid monosolvate

Soraya Merzouki,^a Chabane Mouats,^a El-Eulmi Bendeif,^b Sebastien Pillet^b and Karim Bouchouit^{c*}

^aLaboratoire de Chimie Moléculaire, du Contrôle de l'Environnement et des Mesures Physico-Chimiques, Faculté des Sciences Exats, Département de Chimie, Université Mentouri de Constantine, 25000 Constantine, Algeria, ^bLaboratoire de Cristallographie, Résonance Magnétique et Modélisations, (CRM2, UMR CNRS 7036), Institut Jean Barriol, Nancy Université, BP 70239, Boulevard des Aiguillettes, 54506 Vandoeuvre-lès Nancy, France, and ^cDépartement de Chimie, Faculté des Sciences, Université de Jijel, 18000-Jijel, Algeria
Correspondence e-mail: karim.bouchouit@laposte.net

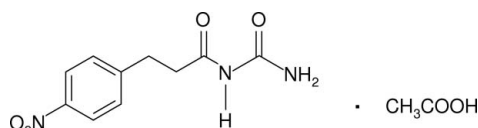
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.041; wR factor = 0.111; data-to-parameter ratio = 15.7.

The title compound, $\text{C}_{10}\text{H}_{11}\text{N}_3\text{O}_4 \cdot \text{C}_2\text{H}_4\text{O}_2$, was prepared by an electrochemical technique. In the crystal, acetic acid molecules are involved in hydrogen bonding to two separate propanoylurea molecules, acting as a donor in an $\text{O}-\text{H} \cdots \text{O}$ interaction and as an acceptor in two $\text{N}-\text{H} \cdots \text{O}$ interactions. The propanoylurea molecules interact with each other *via* $\text{N}-\text{H} \cdots \text{O}$ hydrogen bonds. $\text{C}-\text{H} \cdots \text{O}$ interactions also stabilize the crystal structure.

Related literature

For the preparation of heterocyclic compounds, see: Weinberg & Tilak (1982); Katritzky & Lagowski (1971); Sicker *et al.* (1995). For bond lengths and angles in similar compounds, see: Cai *et al.* (2011); Yakimanski *et al.* (1997).



Experimental

Crystal data

$\text{C}_{10}\text{H}_{11}\text{N}_3\text{O}_4 \cdot \text{C}_2\text{H}_4\text{O}_2$ $\gamma = 91.091$ (3)°
 $M_r = 297.27$ $V = 674.20$ (4) Å³
 Triclinic, $P\bar{1}$ $Z = 2$
 $a = 7.4252$ (3) Å $\text{Mo K}\alpha$ radiation
 $b = 7.9601$ (3) Å $\mu = 0.12$ mm⁻¹
 $c = 11.4375$ (4) Å $T = 100$ K
 $\alpha = 92.736$ (3)° $0.40 \times 0.20 \times 0.10$ mm
 $\beta = 92.939$ (3)°

Data collection

Oxford Diffraction SuperNova diffractometer 16680 measured reflections
 3913 independent reflections
 Absorption correction: integration (*ABSORB*; DeTitta, 1985) 3563 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.026$
 $T_{\text{min}} = 0.954$, $T_{\text{max}} = 0.988$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$ 250 parameters
 $wR(F^2) = 0.111$ All H-atom parameters refined
 $S = 1.05$ $\Delta\rho_{\text{max}} = 0.41$ e Å⁻³
 3913 reflections $\Delta\rho_{\text{min}} = -0.32$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
$\text{O6}-\text{H} \cdots \text{O4}^i$	0.90 (2)	1.76 (2)	2.6492 (12)	166 (2)
$\text{N2}-\text{H9} \cdots \text{O4}^{ii}$	0.863 (15)	1.998 (15)	2.8611 (12)	178.7 (15)
$\text{N3}-\text{H10} \cdots \text{O3}$	0.884 (15)	2.036 (16)	2.6892 (12)	129.8 (13)
$\text{N3}-\text{H10} \cdots \text{O5}^{iii}$	0.884 (15)	2.349 (15)	2.9434 (13)	124.7 (13)
$\text{N3}-\text{H11} \cdots \text{O5}^{iv}$	0.899 (16)	2.017 (16)	2.9050 (12)	169.1 (14)
$\text{C2}-\text{H2} \cdots \text{O3}^i$	0.980 (16)	2.550 (17)	3.3434 (14)	138.0 (13)
$\text{C3}-\text{H3} \cdots \text{O5}^v$	0.957 (16)	2.536 (15)	3.4793 (13)	168.6 (12)
$\text{C5}-\text{H5} \cdots \text{O2}^{iv}$	0.945 (16)	2.463 (16)	3.3724 (14)	161.6 (12)
$\text{C8}-\text{H81} \cdots \text{O1}^{vi}$	0.950 (15)	2.496 (15)	3.4266 (15)	166.3 (13)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2009); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *enCIFer* (Allen *et al.*, 2004).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FY2022).

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supplementary materials

Acta Cryst. (2011). E67, o2892 [doi:10.1107/S1600536811039729]

1-[3-(4-Nitrophenyl)propanoyl]urea acetic acid monosolvate

S. Merzouki, C. Mouats, E.-E. Bendeif, S. Pillet and K. Bouchouit

Comment

Over the past few years, significant research has been directed toward the development of new technologies for environment-friendly processes, such as electrochemical synthesis and green chemistry, which are both economically and technologically feasible. Electrochemistry seems to be a method of choice to prepare various heterocyclic compounds, because the anodic oxidation and the cathodic reduction allow the selective preparation of the active intermediates (Weinberg & Tilak, 1982). However, obtaining aniline products is generally not easy. The chemical reductions are not always selective and often lead to mixtures (Katritzky & Lagowski, 1971). In particular, the reduction of substituted nitrobenzenes with controlled potential can produce a number of heterocycles (Sicker *et al.*, 1995).

The present paper reports the crystal structure determination and analysis of a new organic compound. The asymmetric unit contains one 1-(3-(4-nitrophenyl)propanoyl)urea and one acetic acid molecule (Fig. 1). The cohesion and stability of the crystal is provided by N—H \cdots O, O—H \cdots O and C—H \cdots O hydrogen bonds (Table 1).

These interactions form molecular tapes composed of alternating acetic acid and 1-(3-(4-nitrophenyl)propanoyl)urea molecules (Fig. 2).

All bond lengths and angles are within usual values and are comparable to those observed in similar compounds (Cai *et al.*, 2011; Yakimanski *et al.*, 1997).

Experimental

The electrochemical studies were carried out in ethanol using sodium acetate buffer (1 N) as electrolyte support. We worked on a potential of -1 V/SCE with mercury electrode. The electroreduction of ethyl 3-(4-nitrophenyl)propanoate gave a mixture of the title compound and 6-hydroxy-2,3-dihydroinden-1-one. Only the title compound is soluble in ethanol, which allowed easy separation of the two reaction products. The title compound was recrystallized from ethanol/acetic acid (1:1), and gave crystals that melt at 110°C. 6-Hydroxy-2,3-dihydroinden-1-one, after recrystallization from ethanol/water (2:1), was found to melt at 156°C.

Refinement

The electron density of the H atoms was clearly identified in the Fourier difference map, and the atomic coordinates and isotropic displacements parameters of the H atoms were refined freely.

Figures

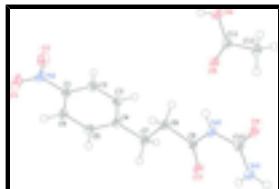


Fig. 1. ORTEP-3 drawing of the title compound with the atom-numbering scheme. Ellipsoids are drawn at the 50 % probability level.

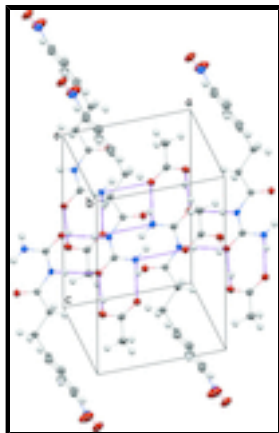


Fig. 2. Molecular packing and hydrogen bond pattern.

1-[3-(4-Nitrophenyl)propanoyl]urea acetic acid monosolvate

Crystal data

$C_{10}H_{11}N_3O_4 \cdot C_2H_4O_2$

$M_r = 297.27$

Triclinic, $P\bar{1}$

$a = 7.4252$ (3) Å

$b = 7.9601$ (3) Å

$c = 11.4375$ (4) Å

$\alpha = 92.736$ (3)°

$\beta = 92.939$ (3)°

$\gamma = 91.091$ (3)°

$V = 674.20$ (4) Å³

$Z = 2$

$F(000) = 312$

$D_x = 1.464$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 4625 reflections

$\theta = 3.2\text{--}30.0^\circ$

$\mu = 0.12$ mm⁻¹

$T = 100$ K

Prism, yellow

$0.40 \times 0.20 \times 0.10$ mm

Data collection

Oxford Diffraction SuperNova
diffractometer

Radiation source: fine-focus sealed tube
graphite

ω scans

Absorption correction: integration
(*ABSORB*; DeTitta, 1985)

$T_{\min} = 0.954$, $T_{\max} = 0.988$

16680 measured reflections

3913 independent reflections

3563 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.026$

$\theta_{\max} = 30.0^\circ$, $\theta_{\min} = 3.2^\circ$

$h = -10 \rightarrow 10$

$k = -11 \rightarrow 11$

$l = 0 \rightarrow 16$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.041$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.111$	All H-atom parameters refined
$S = 1.05$	$w = 1/[\sigma^2(F_o^2) + (0.0556P)^2 + 0.2344P]$
3913 reflections	where $P = (F_o^2 + 2F_c^2)/3$
250 parameters	$(\Delta/\sigma)_{\max} < 0.001$
0 restraints	$\Delta\rho_{\max} = 0.41 \text{ e } \text{\AA}^{-3}$
	$\Delta\rho_{\min} = -0.32 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
N2	0.77505 (12)	0.49422 (11)	0.56957 (8)	0.01868 (18)
O3	0.51190 (10)	0.47679 (9)	0.66343 (7)	0.02065 (17)
C9	0.64971 (13)	0.55423 (12)	0.64495 (8)	0.01614 (18)
C10	0.77604 (14)	0.33654 (13)	0.51251 (9)	0.01820 (19)
O4	0.91036 (11)	0.30011 (10)	0.45541 (7)	0.02409 (18)
N3	0.63746 (13)	0.23181 (11)	0.52143 (8)	0.01982 (18)
O5	0.71569 (11)	0.90282 (10)	0.41754 (7)	0.02333 (17)
O6	0.95760 (12)	1.01068 (11)	0.33799 (8)	0.0291 (2)
C11	0.84078 (14)	0.88799 (13)	0.35312 (9)	0.0189 (2)
C12	0.87300 (17)	0.73163 (14)	0.28067 (10)	0.0242 (2)
H71	0.450 (2)	0.7953 (19)	0.7578 (13)	0.025 (4)*
H81	0.820 (2)	0.714 (2)	0.7386 (14)	0.029 (4)*
H6	0.865 (2)	1.0680 (19)	1.1039 (14)	0.028 (4)*
H2	0.619 (2)	1.350 (2)	0.8551 (15)	0.038 (4)*
H82	0.710 (2)	0.8064 (19)	0.6429 (13)	0.026 (4)*
H5	0.763 (2)	0.822 (2)	0.9966 (13)	0.029 (4)*
H72	0.556 (2)	0.687 (2)	0.8510 (14)	0.029 (4)*
H3	0.524 (2)	1.101 (2)	0.7488 (14)	0.029 (4)*

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H9	0.871 (2)	0.555 (2)	0.5624 (14)	0.030 (4)*
H10	0.545 (2)	0.263 (2)	0.5625 (14)	0.029 (4)*
H11	0.646 (2)	0.127 (2)	0.4896 (14)	0.033 (4)*
H121	0.832 (2)	0.635 (2)	0.3199 (16)	0.041 (5)*
H	0.927 (3)	1.101 (3)	0.383 (2)	0.064 (6)*
H122	0.999 (3)	0.723 (2)	0.2602 (16)	0.047 (5)*
H123	0.802 (3)	0.738 (3)	0.207 (2)	0.067 (6)*
C4	0.63442 (13)	0.93684 (12)	0.86240 (9)	0.01726 (19)
C1	0.75280 (14)	1.22675 (12)	0.98721 (9)	0.0184 (2)
C8	0.70192 (14)	0.72292 (12)	0.70362 (9)	0.01779 (19)
N1	0.81408 (14)	1.38040 (12)	1.05424 (9)	0.0242 (2)
C5	0.73579 (15)	0.92833 (13)	0.96774 (9)	0.0193 (2)
C7	0.56967 (15)	0.77809 (13)	0.79493 (9)	0.0203 (2)
C3	0.59529 (15)	1.09439 (14)	0.82046 (9)	0.0218 (2)
O1	0.90310 (14)	1.36682 (12)	1.14622 (9)	0.0386 (2)
C2	0.65298 (16)	1.24075 (13)	0.88280 (10)	0.0221 (2)
C6	0.79598 (15)	1.07320 (13)	1.03125 (9)	0.0195 (2)
O2	0.77247 (18)	1.51546 (11)	1.01607 (10)	0.0464 (3)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N2	0.0173 (4)	0.0162 (4)	0.0221 (4)	-0.0037 (3)	0.0049 (3)	-0.0059 (3)
O3	0.0193 (4)	0.0183 (3)	0.0243 (4)	-0.0037 (3)	0.0052 (3)	-0.0026 (3)
C9	0.0170 (4)	0.0159 (4)	0.0154 (4)	0.0010 (3)	0.0007 (3)	-0.0014 (3)
C10	0.0188 (5)	0.0172 (4)	0.0182 (4)	-0.0014 (3)	0.0017 (3)	-0.0035 (3)
O4	0.0215 (4)	0.0205 (4)	0.0298 (4)	-0.0053 (3)	0.0099 (3)	-0.0104 (3)
N3	0.0193 (4)	0.0173 (4)	0.0227 (4)	-0.0028 (3)	0.0052 (3)	-0.0036 (3)
O5	0.0230 (4)	0.0203 (4)	0.0266 (4)	-0.0034 (3)	0.0065 (3)	-0.0053 (3)
O6	0.0293 (4)	0.0218 (4)	0.0359 (5)	-0.0090 (3)	0.0145 (4)	-0.0114 (3)
C11	0.0194 (5)	0.0183 (4)	0.0186 (4)	-0.0016 (4)	0.0001 (4)	-0.0027 (3)
C12	0.0280 (6)	0.0189 (5)	0.0252 (5)	-0.0010 (4)	0.0050 (4)	-0.0064 (4)
C4	0.0173 (4)	0.0162 (4)	0.0183 (4)	-0.0010 (3)	0.0063 (3)	-0.0033 (3)
C1	0.0206 (5)	0.0138 (4)	0.0209 (5)	-0.0014 (3)	0.0050 (4)	-0.0031 (3)
C8	0.0171 (4)	0.0164 (4)	0.0195 (4)	-0.0024 (3)	0.0036 (3)	-0.0045 (3)
N1	0.0284 (5)	0.0159 (4)	0.0279 (5)	-0.0018 (3)	0.0036 (4)	-0.0043 (3)
C5	0.0236 (5)	0.0135 (4)	0.0208 (5)	0.0018 (4)	0.0032 (4)	-0.0012 (3)
C7	0.0197 (5)	0.0189 (5)	0.0219 (5)	-0.0034 (4)	0.0062 (4)	-0.0068 (4)
C3	0.0247 (5)	0.0215 (5)	0.0192 (5)	-0.0001 (4)	0.0003 (4)	0.0012 (4)
O1	0.0432 (6)	0.0270 (5)	0.0426 (5)	0.0031 (4)	-0.0148 (4)	-0.0129 (4)
C2	0.0282 (5)	0.0160 (4)	0.0225 (5)	0.0010 (4)	0.0028 (4)	0.0029 (4)
C6	0.0228 (5)	0.0174 (4)	0.0180 (4)	0.0016 (4)	0.0015 (4)	-0.0020 (3)
O2	0.0810 (8)	0.0129 (4)	0.0435 (6)	-0.0018 (4)	-0.0115 (5)	-0.0004 (4)

Geometric parameters (\AA , $^\circ$)

N2—C9	1.3776 (12)	C4—C7	1.5064 (14)
N2—C10	1.3871 (12)	C1—C6	1.3811 (14)
N2—H9	0.866 (17)	C1—C2	1.3831 (15)

O3—C9	1.2175 (13)	C1—N1	1.4643 (13)
C9—C8	1.5070 (13)	C8—C7	1.5250 (14)
C10—O4	1.2510 (12)	C8—H81	0.950 (16)
C10—N3	1.3230 (14)	C8—H82	0.987 (15)
N3—H10	0.884 (17)	N1—O2	1.2187 (13)
N3—H11	0.898 (18)	N1—O1	1.2237 (14)
O5—C11	1.2189 (13)	C5—C6	1.3872 (14)
O6—C11	1.3174 (13)	C5—H5	0.942 (16)
O6—H	0.90 (2)	C7—H71	0.981 (15)
C11—C12	1.4920 (14)	C7—H72	1.000 (16)
C12—H121	0.960 (18)	C3—C2	1.3854 (15)
C12—H122	0.98 (2)	C3—H3	0.956 (15)
C12—H123	0.97 (2)	C2—H2	0.977 (17)
C4—C5	1.3926 (15)	C6—H6	0.958 (16)
C4—C3	1.3944 (14)		
C9—N2—C10	127.24 (9)	C9—C8—C7	112.08 (8)
C9—N2—H9	117.7 (11)	C9—C8—H81	107.3 (10)
C10—N2—H9	114.6 (11)	C7—C8—H81	110.8 (10)
O3—C9—N2	123.04 (9)	C9—C8—H82	108.7 (9)
O3—C9—C8	123.57 (9)	C7—C8—H82	110.8 (9)
N2—C9—C8	113.38 (8)	H81—C8—H82	106.8 (13)
O4—C10—N3	123.15 (9)	O2—N1—O1	123.29 (10)
O4—C10—N2	117.71 (9)	O2—N1—C1	118.30 (10)
N3—C10—N2	119.13 (9)	O1—N1—C1	118.41 (9)
C10—N3—H10	120.3 (11)	C6—C5—C4	121.10 (9)
C10—N3—H11	117.3 (11)	C6—C5—H5	119.4 (10)
H10—N3—H11	122.2 (15)	C4—C5—H5	119.4 (10)
C11—O6—H	107.8 (14)	C4—C7—C8	111.51 (8)
O5—C11—O6	123.10 (10)	C4—C7—H71	109.6 (9)
O5—C11—C12	123.45 (10)	C8—C7—H71	110.7 (9)
O6—C11—C12	113.42 (9)	C4—C7—H72	108.9 (9)
C11—C12—H121	109.7 (11)	C8—C7—H72	109.1 (9)
C11—C12—H122	112.0 (11)	H71—C7—H72	106.8 (13)
H121—C12—H122	112.5 (15)	C2—C3—C4	121.03 (10)
C11—C12—H123	107.4 (13)	C2—C3—H3	119.5 (10)
H121—C12—H123	108.7 (17)	C4—C3—H3	119.4 (10)
H122—C12—H123	106.3 (17)	C1—C2—C3	118.28 (10)
C5—C4—C3	118.86 (9)	C1—C2—H2	121.2 (10)
C5—C4—C7	120.31 (9)	C3—C2—H2	120.5 (10)
C3—C4—C7	120.83 (10)	C1—C6—C5	118.21 (10)
C6—C1—C2	122.52 (9)	C1—C6—H6	120.4 (9)
C6—C1—N1	118.62 (9)	C5—C6—H6	121.4 (9)
C2—C1—N1	118.86 (9)		
C10—N2—C9—O3	-6.33 (17)	C5—C4—C7—C8	-92.93 (12)
C10—N2—C9—C8	172.56 (9)	C3—C4—C7—C8	86.63 (12)
C9—N2—C10—O4	-173.81 (10)	C9—C8—C7—C4	173.53 (9)
C9—N2—C10—N3	5.63 (16)	C5—C4—C3—C2	-1.10 (16)
O3—C9—C8—C7	4.31 (14)	C7—C4—C3—C2	179.33 (10)

supplementary materials

N2—C9—C8—C7	-174.57 (9)	C6—C1—C2—C3	-0.24 (17)
C6—C1—N1—O2	-178.56 (11)	N1—C1—C2—C3	-179.77 (10)
C2—C1—N1—O2	1.00 (16)	C4—C3—C2—C1	0.88 (17)
C6—C1—N1—O1	0.81 (15)	C2—C1—C6—C5	-0.18 (16)
C2—C1—N1—O1	-179.63 (11)	N1—C1—C6—C5	179.36 (9)
C3—C4—C5—C6	0.67 (15)	C4—C5—C6—C1	-0.04 (16)
C7—C4—C5—C6	-179.76 (9)		

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>
O6—H \cdots O4 ⁱ	0.90 (2)	1.76 (2)	2.6492 (12)	166 (2)
N2—H9 \cdots O4 ⁱⁱ	0.863 (15)	1.998 (15)	2.8611 (12)	178.7 (15)
N3—H10 \cdots O3	0.884 (15)	2.036 (16)	2.6892 (12)	129.8 (13)
N3—H10 \cdots O5 ⁱⁱⁱ	0.884 (15)	2.349 (15)	2.9434 (13)	124.7 (13)
N3—H11 \cdots O5 ^{iv}	0.899 (16)	2.017 (16)	2.9050 (12)	169.1 (14)
C2—H2 \cdots O3 ⁱ	0.980 (16)	2.550 (17)	3.3434 (14)	138.0 (13)
C3—H3 \cdots O5 ^v	0.957 (16)	2.536 (15)	3.4793 (13)	168.6 (12)
C5—H5 \cdots O2 ^{iv}	0.945 (16)	2.463 (16)	3.3724 (14)	161.6 (12)
C8—H81 \cdots O1 ^{vi}	0.950 (15)	2.496 (15)	3.4266 (15)	166.3 (13)

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

Fig. 1

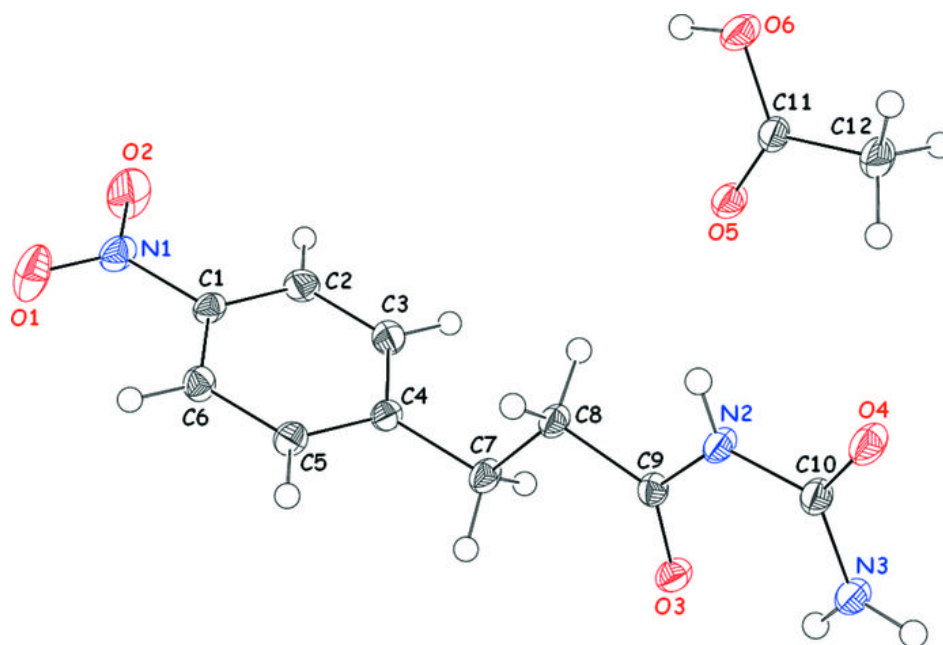


Fig. 2

