

(2*R*,3*S*)-2-Ammonio-3-hydroxy-3-(4-nitrophenyl)propanoic acid chloride monohydrateVincent Gaumet,^{a*} Valérie Weber,^b Valery P. Zaitsev^c and Michel Madesclaire^b^aLaboratoire de Chimie Physique, EA 4231, Faculté de Pharmacie, Université d'Auvergne, 63001 Clermont-Ferrand, France, ^bLaboratoire de Chimie Thérapeutique, EA 4231, Faculté de Pharmacie, Université d'Auvergne, 63001 Clermont-Ferrand, France, and ^cChimie Organique, Faculté de Chimie, 443011 Samara, Russian Federation

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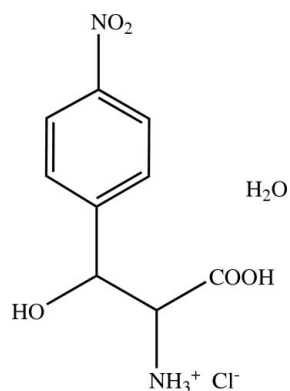
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.043; wR factor = 0.115; data-to-parameter ratio = 25.5.

The title compound, $\text{C}_9\text{H}_{11}\text{N}_2\text{O}_5^+\cdot\text{Cl}^-\cdot\text{H}_2\text{O}$, was synthesized from (1*S*,2*S*)-2-amino-1-(4-nitrophenyl)propane-1,3-diol in four steps. As demonstrated by this work, no racemization occurs during this synthetic procedure. The crystal structure displays many intermolecular hydrogen bonds between the acidic cation, chloride anions and the water molecules, forming a three-dimensional network. An intramolecular bond between the ammonium group and a hydroxyl O atom is also present.

Related literature

For related compounds see: Crich *et al.* (2007); Di Giovanni *et al.* (1996); Easton *et al.* (1996); Madesclaire *et al.* (2006, 2007); Steinreiber *et al.* (2007); Zaitsev *et al.* (1998).

**Experimental***Crystal data*

$\text{C}_9\text{H}_{11}\text{N}_2\text{O}_5^+\cdot\text{Cl}^-\cdot\text{H}_2\text{O}$
 $M_r = 280.66$
 Monoclinic, $P2_1$
 $a = 8.1286$ (17) Å
 $b = 5.056$ (3) Å
 $c = 15.848$ (3) Å
 $\beta = 104.626$ (17)°

$V = 630.2$ (4) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.33$ mm⁻¹
 $T = 293$ (2) K
 $0.49 \times 0.25 \times 0.20$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.872$, $T_{\max} = 0.931$
 6026 measured reflections

5517 independent reflections
 5058 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$
 3 standard reflections every 63 reflections
 intensity decay: 3%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.115$
 $S = 1.09$
 5517 reflections
 216 parameters
 1 restraint

All H-atom parameters refined
 $\Delta\rho_{\text{max}} = 0.60$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.63$ e Å⁻³
 Absolute structure: Flack (1983),
 2752 Friedel pairs
 Flack parameter: 0.00 (4)

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N10}-\text{H101}\cdots\text{O13}^{\text{i}}$	0.810 (17)	2.420 (16)	2.9259 (13)	121.5 (14)
$\text{N10}-\text{H101}\cdots\text{Cl}^{\text{ii}}$	0.810 (17)	2.489 (16)	3.2298 (11)	152.7 (15)
$\text{N10}-\text{H102}\cdots\text{O14}$	0.83 (2)	2.265 (19)	2.6759 (14)	110.8 (16)
$\text{N10}-\text{H102}\cdots\text{Cl}^{\text{iii}}$	0.83 (2)	2.62 (2)	3.3409 (14)	145.5 (16)
$\text{N10}-\text{H103}\cdots\text{Cl}^{\text{iv}}$	0.97 (3)	2.28 (3)	3.2435 (14)	176 (3)
$\text{O12}-\text{H12}\cdots\text{O17}^{\text{v}}$	0.78 (5)	1.84 (5)	2.6168 (19)	171 (4)
$\text{O14}-\text{H14}\cdots\text{Cl}$	0.82 (3)	2.25 (3)	3.0539 (13)	166 (3)
$\text{O17}-\text{H171}\cdots\text{O16}^{\text{vi}}$	0.78 (3)	2.33 (3)	3.069 (2)	159 (4)
$\text{O17}-\text{H172}\cdots\text{Cl}^{\text{vii}}$	0.76 (3)	2.45 (3)	3.2170 (13)	178 (3)

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms, 1996); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CS2076).

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supplementary materials

Acta Cryst. (2008). E64, o1003-o1004 [doi:10.1107/S1600536808012750]

(2*R*,3*S*)-2-Ammonio-3-hydroxy-3-(4-nitrophenyl)propanoic acid chloride monohydrate

V. Gaumet, V. Weber, V. P. Zaitsev and M. Madesclaire

Comment

Preparation of new α -amino acids are of constant interest. (1*S*,2*S*) and (1*R*,2*R*)-2-amino-1-(4-nitrophenyl)-1,3-propanediols, byproducts in the manufacture of the antibiotic Chloramphenicol (Zaitsev *et al.*, 1998), can be used as starting materials in the preparation of corresponding α -amino acid isomers. The title compound (V) was synthesized from (1*S*,2*S*)-2-amino-1-(4-nitrophenyl)-1,3-propanediol (I) in four steps according to Figure 1. The intermediate formation of the 2-oxazolidinone derivative (III) allowed to protect both the adjacent amino and hydroxyl groups (Di Giovanni *et al.*, 1996; Crich *et al.*, 2007). The absolute configuration, determined using anomalous dispersion by chlorine, confirms the *R* and *S* configurations respectively for C2 and C3 atoms, confirming that no racemization occurs during this synthetic route (note that the Cahn-Ingold-Prelog designation at the α -carbon of the hydroxyl group is reversed by comparison with that of the starting material due to the change in priority of the substituents). As expected, the phenyl ring is planar, r.m.s. deviation from the best plane is ca. 0.007Å. The nitro group is coplanar with the adjacent phenyl ring (O15—N11—C7—C8 = 0.2 (3)° and O16—N11—C7—C8 = 179.9 (2)°). However, the σ bond C7—N11 (1.4704 (17) Å) shows that there is no appreciable π delocalization in the bond between the sp^2 hybridized N11 and the phenyl ring.

Experimental

Figure 1 summarizes the synthetic route used (Madesclaire *et al.*, 2006 and 2007). Crystals suitable for an X-ray diffraction study were obtained by slow evaporation of a water solution containing compound (V).

Refinement

The structure was solved by direct methods and refined with anisotropic temperature factors for non-H atoms. All H atoms were found from difference Fourier maps. The H atoms were all refined isotropically with no constraints.

Figures

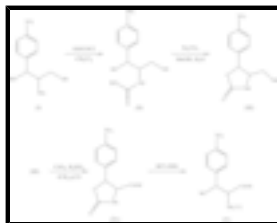


Fig. 1. Synthesis of the title compound (V).

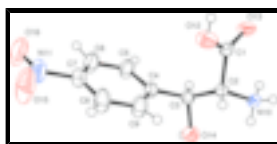


Fig. 2. The molecular structure of the title compound (V), with atom labels and 50% probability displacement ellipsoids for non-H atoms.

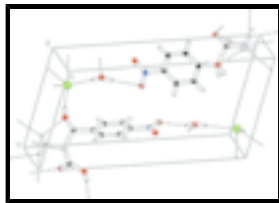


Fig. 3. Crystal packing of the title compound. Intra and intermolecular hydrogen bonds are shown as dashed lines. H atoms not involved in the interactions have been omitted.

(2*R*,3*S*)-2-Ammonio-3-hydroxy-3-(4-nitrophenyl)propanoic acid chloride monohydrate

Crystal data

$C_9H_{11}N_2O_5^+ \cdot Cl^- \cdot H_2O$

$M_r = 280.66$

Monoclinic, $P2_1$

Hall symbol: P 2yb

$a = 8.1286$ (17) Å

$b = 5.056$ (3) Å

$c = 15.848$ (3) Å

$\beta = 104.626$ (17)°

$V = 630.2$ (4) Å³

$Z = 2$

$F_{000} = 292$

$D_x = 1.479$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 25 reflections

$\theta = 8$ – 18°

$\mu = 0.33$ mm⁻¹

$T = 293$ (2) K

Elongated prism, colourless

$0.49 \times 0.25 \times 0.20$ mm

Data collection

Enraf–Nonius CAD-4
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293$ (2) K

$\omega/2\theta$ scans

Absorption correction: ψ scan
(North et al., 1968)

$T_{\min} = 0.872$, $T_{\max} = 0.931$

6026 measured reflections

5517 independent reflections

5058 reflections with $I > 2\sigma(I)$

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

$\theta_{\max} = 35.0^\circ$

$\theta_{\min} = 1.3^\circ$

$h = -13 \rightarrow 13$

$k = -8 \rightarrow 8$

$l = -25 \rightarrow 25$

3 standard reflections

every 63 reflections

intensity decay: 3%

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.043$

$wR(F^2) = 0.115$

$S = 1.09$

Hydrogen site location: inferred from neighbouring sites

All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0825P)^2 + 0.0202P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.60$ e Å⁻³

5517 reflections $\Delta\rho_{\min} = -0.63 \text{ e } \text{\AA}^{-3}$
 216 parameters Extinction correction: SHELXL97 (Sheldrick, 2008),
 $F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
 1 restraint Extinction coefficient: 0.089 (10)
 Primary atom site location: structure-invariant direct methods Absolute structure: Flack (1983), 2752 Friedel pairs
 Secondary atom site location: difference Fourier map Flack parameter: 0.00 (4)

Special details

Experimental. North A.C.T., Phillips D.C. & Mathews F.S. (1968) Acta. Cryst. A24, 351. Number of psi-scan sets used was 4. Theta correction was applied. Weighted transmission curves were used. No Fourier smoothing was applied.

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl	0.71520 (3)	0.93514 (6)	0.922526 (17)	0.03591 (8)
C1	-0.00336 (13)	1.2561 (2)	0.83505 (7)	0.02674 (16)
C2	0.09722 (12)	1.00092 (18)	0.85613 (6)	0.02405 (15)
C3	0.27337 (13)	1.0067 (2)	0.83713 (6)	0.02743 (16)
C4	0.26848 (14)	1.0458 (2)	0.74189 (7)	0.02986 (18)
C5	0.3717 (2)	1.2362 (3)	0.71889 (9)	0.0427 (3)
C6	0.3767 (3)	1.2672 (4)	0.63235 (10)	0.0499 (4)
C7	0.2774 (2)	1.1044 (3)	0.57084 (8)	0.0415 (3)
C8	0.1743 (2)	0.9107 (4)	0.59137 (9)	0.0506 (4)
C9	0.1720 (2)	0.8796 (3)	0.67820 (9)	0.0459 (3)
N10	0.12271 (11)	0.9477 (2)	0.95052 (5)	0.02702 (14)
N11	0.2786 (2)	1.1354 (4)	0.47874 (9)	0.0569 (4)
O12	-0.08726 (19)	1.2705 (3)	0.75327 (6)	0.0491 (3)
O13	-0.00519 (12)	1.42165 (18)	0.88974 (5)	0.03491 (16)
O14	0.34476 (13)	0.7557 (2)	0.86696 (7)	0.03708 (19)
O15	0.1882 (3)	0.9890 (7)	0.42545 (9)	0.0966 (9)
O16	0.3691 (3)	1.3062 (5)	0.45945 (10)	0.0847 (6)
H2	0.034 (3)	0.872 (5)	0.8304 (13)	0.037 (5)*
H3	0.343 (3)	1.127 (5)	0.8692 (13)	0.032 (5)*
H5	0.445 (3)	1.340 (6)	0.7648 (17)	0.052 (6)*
H6	0.453 (4)	1.424 (10)	0.620 (2)	0.091 (10)*
H8	0.112 (5)	0.807 (10)	0.548 (2)	0.087 (10)*
H9	0.090 (4)	0.766 (8)	0.6899 (19)	0.073 (9)*
H101	0.032 (2)	0.927 (4)	0.9615 (10)	0.025 (3)*
H102	0.186 (2)	0.816 (4)	0.9639 (12)	0.030 (4)*
H103	0.174 (4)	1.087 (7)	0.9905 (16)	0.058 (7)*
H12	-0.147 (5)	1.395 (10)	0.743 (2)	0.087 (10)*
H14	0.448 (3)	0.778 (6)	0.8856 (15)	0.049 (6)*
O17	0.29100 (18)	0.1787 (3)	0.26402 (8)	0.0491 (3)
H171	0.307 (4)	0.250 (8)	0.309 (2)	0.073 (9)*
H172	0.290 (3)	0.237 (6)	0.2194 (17)	0.053 (7)*

supplementary materials

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.03069 (11)	0.03544 (12)	0.03887 (13)	0.00116 (10)	0.00372 (8)	-0.00215 (10)
C1	0.0319 (4)	0.0221 (3)	0.0267 (4)	0.0005 (3)	0.0084 (3)	0.0022 (3)
C2	0.0281 (3)	0.0200 (3)	0.0249 (3)	-0.0009 (3)	0.0083 (3)	0.0000 (2)
C3	0.0288 (4)	0.0274 (4)	0.0276 (4)	-0.0040 (3)	0.0099 (3)	-0.0011 (3)
C4	0.0339 (4)	0.0297 (4)	0.0287 (4)	-0.0028 (3)	0.0130 (3)	-0.0025 (3)
C5	0.0576 (7)	0.0393 (6)	0.0359 (5)	-0.0182 (6)	0.0206 (5)	-0.0051 (4)
C6	0.0705 (10)	0.0481 (7)	0.0377 (6)	-0.0171 (7)	0.0260 (6)	-0.0004 (5)
C7	0.0531 (7)	0.0457 (7)	0.0295 (5)	0.0025 (5)	0.0176 (5)	0.0005 (4)
C8	0.0595 (8)	0.0634 (10)	0.0304 (5)	-0.0190 (8)	0.0141 (5)	-0.0110 (6)
C9	0.0582 (8)	0.0504 (8)	0.0323 (5)	-0.0227 (6)	0.0172 (5)	-0.0090 (5)
N10	0.0337 (3)	0.0226 (3)	0.0266 (3)	0.0022 (3)	0.0110 (2)	0.0036 (3)
N11	0.0739 (10)	0.0692 (11)	0.0324 (5)	-0.0018 (8)	0.0221 (6)	0.0009 (6)
O12	0.0684 (7)	0.0451 (5)	0.0274 (4)	0.0240 (5)	0.0005 (4)	0.0003 (4)
O13	0.0471 (4)	0.0216 (3)	0.0339 (3)	0.0039 (3)	0.0062 (3)	-0.0026 (3)
O14	0.0310 (4)	0.0371 (4)	0.0441 (5)	0.0061 (3)	0.0112 (3)	0.0058 (4)
O15	0.1309 (16)	0.127 (2)	0.0336 (5)	-0.0504 (17)	0.0250 (8)	-0.0151 (8)
O16	0.1330 (17)	0.0846 (12)	0.0465 (7)	-0.0305 (13)	0.0414 (9)	0.0055 (8)
O17	0.0652 (7)	0.0431 (5)	0.0357 (5)	-0.0145 (5)	0.0070 (4)	0.0004 (4)

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

C1—O13	1.2078 (14)	C7—C8	1.380 (2)
C1—O12	1.3053 (14)	C7—N11	1.4704 (17)
C1—C2	1.5188 (15)	C8—C9	1.3898 (18)
C2—N10	1.4821 (12)	C8—H8	0.91 (4)
C2—C3	1.5360 (14)	C9—H9	0.94 (4)
C2—H2	0.87 (2)	N10—H101	0.810 (17)
C3—O14	1.4253 (16)	N10—H102	0.83 (2)
C3—C4	1.5128 (14)	N10—H103	0.97 (3)
C3—H3	0.90 (2)	N11—O15	1.219 (3)
C4—C5	1.3851 (17)	N11—O16	1.222 (3)
C4—C9	1.3931 (18)	O12—H12	0.78 (5)
C5—C6	1.3913 (19)	O14—H14	0.82 (3)
C5—H5	0.97 (3)	O17—H171	0.78 (3)
C6—C7	1.372 (2)	O17—H172	0.76 (3)
C6—H6	1.05 (4)		
O13—C1—O12	125.15 (11)	C5—C6—H6	115.3 (17)
O13—C1—C2	122.31 (10)	C6—C7—C8	122.79 (12)
O12—C1—C2	112.53 (9)	C6—C7—N11	119.42 (14)
N10—C2—C1	107.85 (8)	C8—C7—N11	117.79 (14)
N10—C2—C3	107.55 (8)	C7—C8—C9	118.33 (14)
C1—C2—C3	114.68 (8)	C7—C8—H8	119 (3)
N10—C2—H2	104.6 (14)	C9—C8—H8	123 (3)
C1—C2—H2	108.4 (15)	C8—C9—C4	120.22 (13)

C3—C2—H2	113.1 (14)	C8—C9—H9	117.0 (19)
O14—C3—C4	110.69 (9)	C4—C9—H9	121.9 (19)
O14—C3—C2	103.87 (8)	C2—N10—H101	109.8 (11)
C4—C3—C2	114.02 (9)	C2—N10—H102	108.8 (13)
O14—C3—H3	105.5 (14)	H101—N10—H102	112 (2)
C4—C3—H3	109.3 (13)	C2—N10—H103	117.1 (17)
C2—C3—H3	113.1 (13)	H101—N10—H103	102 (2)
C5—C4—C9	119.76 (11)	H102—N10—H103	106 (2)
C5—C4—C3	119.24 (10)	O15—N11—O16	123.43 (16)
C9—C4—C3	120.81 (10)	O15—N11—C7	117.97 (18)
C4—C5—C6	120.54 (13)	O16—N11—C7	118.60 (17)
C4—C5—H5	118.4 (17)	C1—O12—H12	113 (2)
C6—C5—H5	120.9 (17)	C3—O14—H14	107 (2)
C7—C6—C5	118.32 (13)	H171—O17—H172	129 (4)
C7—C6—H6	126.2 (17)		
O15—N11—C7—C6	-179.5 (2)	O14—C3—C4—C9	62.57 (15)
O16—N11—C7—C6	0.2 (3)	O14—C3—C4—C5	-112.37 (13)
O15—N11—C7—C8	0.2 (3)	C2—C3—C4—C9	-54.08 (14)
O16—N11—C7—C8	179.9 (2)	C5—C4—C9—C8	-2.4 (2)
O12—C1—C2—C3	84.78 (12)	C3—C4—C9—C8	-177.27 (13)
O12—C1—C2—N10	-155.44 (11)	C9—C4—C5—C6	1.6 (2)
O13—C1—C2—N10	23.39 (14)	C3—C4—C5—C6	176.57 (15)
O13—C1—C2—C3	-96.39 (12)	C4—C5—C6—C7	-0.2 (3)
C1—C2—C3—O14	176.50 (9)	C5—C6—C7—N11	179.19 (18)
C1—C2—C3—C4	-62.95 (11)	C5—C6—C7—C8	-0.5 (3)
N10—C2—C3—C4	177.11 (8)	C6—C7—C8—C9	-0.2 (3)
N10—C2—C3—O14	56.56 (10)	N11—C7—C8—C9	-179.96 (17)
C2—C3—C4—C5	130.98 (12)	C7—C8—C9—C4	1.7 (2)

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

<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>
N10—H101 \cdots O13 ⁱ	0.810 (17)	2.420 (16)	2.9259 (13)	121.5 (14)
N10—H101 \cdots Cl ⁱⁱ	0.810 (17)	2.489 (16)	3.2298 (11)	152.7 (15)
N10—H102 \cdots O14	0.83 (2)	2.265 (19)	2.6759 (14)	110.8 (16)
N10—H102 \cdots Cl ⁱⁱⁱ	0.83 (2)	2.62 (2)	3.3409 (14)	145.5 (16)
N10—H103 \cdots Cl ^{iv}	0.97 (3)	2.28 (3)	3.2435 (14)	176 (3)
O12—H12 \cdots O17 ^v	0.78 (5)	1.84 (5)	2.6168 (19)	171 (4)
O14—H14 \cdots Cl	0.82 (3)	2.25 (3)	3.0539 (13)	166 (3)
O17—H171 \cdots O16 ^{vi}	0.78 (3)	2.33 (3)	3.069 (2)	159 (4)
O17—H172 \cdots Cl ^{vii}	0.76 (3)	2.45 (3)	3.2170 (13)	178 (3)

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

Fig. 1

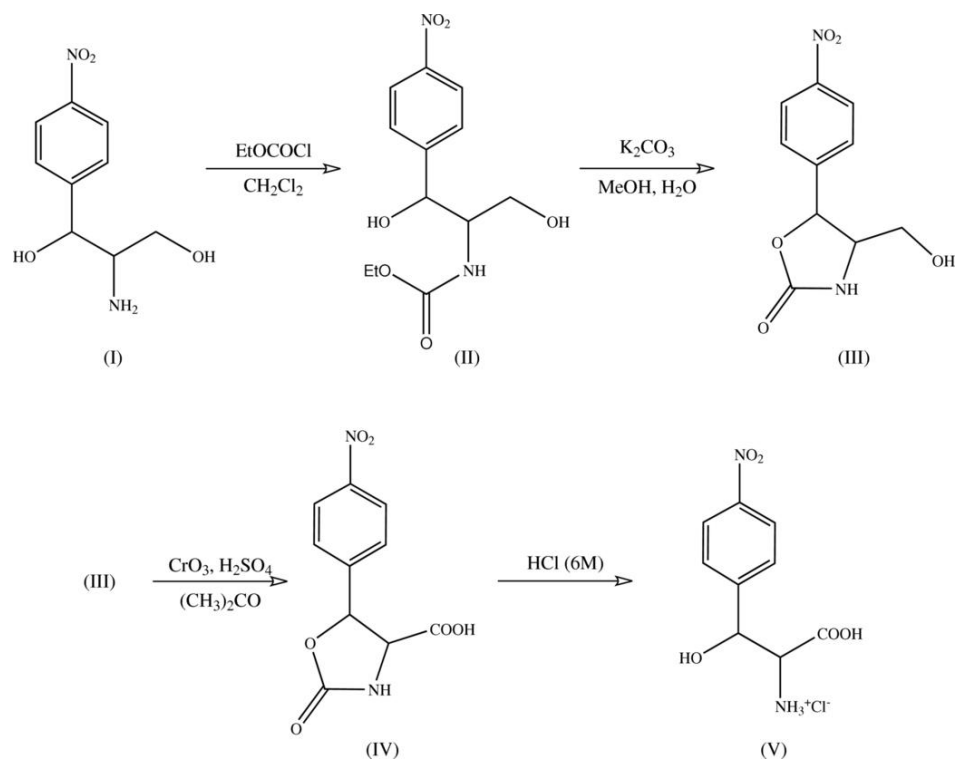


Fig. 2

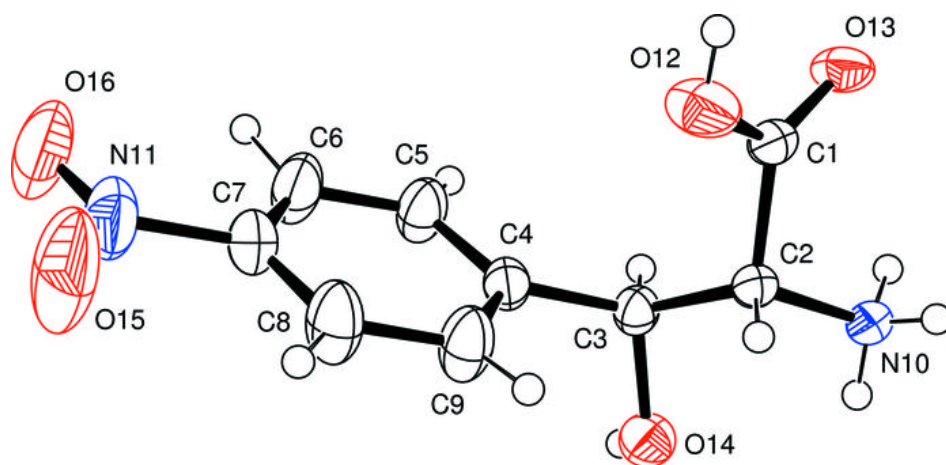


Fig. 3

