

Hui-Fen Qian,^{a*} Jian-Lan Liu^a
and Wei Huang^{b*}^aCollege of Sciences, Nanjing University of
Technology, Nanjing 210009, People's
Republic of China, and ^bState Key Laboratory of
Coordination Chemistry, Coordination
Chemistry Institute, Department of Chemistry
and Chemical Engineering, Nanjing University,
Nanjing 210093, People's Republic of China

Correspondence e-mail: whuang@nju.edu.cn

Key indicators

Single-crystal X-ray study
 $T = 291$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.059
 wR factor = 0.130
Data-to-parameter ratio = 11.4For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.(1*S*,3*R*)-1-Carboxy-2,2,3-trimethylcyclopentane-
1-ammonium nitrate

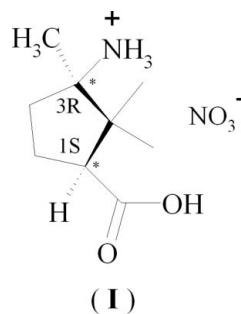
The title compound, $\text{C}_9\text{H}_{18}\text{NO}_2^+\cdot\text{NO}_3^-$, was prepared by an anion exchange from Cl^- to NO_3^- by adding excess NaNO_3 to an aqueous solution of (1*S*,3*R*)-3-amino-2,2,3-trimethylcyclopentane-1-carboxylic acid hydrochloride. $\text{O}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{O}$ hydrogen-bond interactions are observed, through which each nitrate anion links three adjacent cations, forming a three-dimensional network.

Received 28 March 2007

Accepted 27 May 2007

Comment

Recently, we have been engaging in the transformation of functionalized groups of a series of camphor derivatives, having two chiral atoms in their molecular structures. Starting from (1*R*,5*S*)-1,8,8-trimethyl-3-azabicyclo[3.2.1]octane-2,4-dione (Huang, Qian *et al.* 2004), we have obtained the crystal structures of (1*S*,3*R*)-3-carbamoyl-2,2,3-trimethylcyclopentane-1-carboxylic acid (Huang *et al.*, 2003) and its sodium(I) and copper(II) complexes (Huang *et al.* 2005), and of (1*S*,3*R*)-3-amino-2,2,3-trimethylcyclopentane-1-carboxylic acid hydrochloride (Qian *et al.*, 2006). In this paper, we report the crystal structure of the title compound, (I).



The atom-numbering scheme of (I) is shown in Fig. 1, while selected bond distances and bond angles are given in Table 1. The title compound crystallizes in the orthorhombic space group $P2_12_12_1$ and the amine group is protonated. The absolute configurations of the chiral atoms C1 and C3 are assumed to be the same as those in the starting material. The conformation of the five-membered ring in (I) is similar to those of the previously reported (1*S*,3*R*)-3-amino-2,2,3-trimethylcyclopentane-1-carboxylic acid hydrochloride, (1*S*,3*R*)-3-carbamoyl-2,2,3-trimethylcyclopentane-1-carboxylic acid and D-(+)- or racemic camphoric diamine complexes (Huang, Zhou *et al.* 2004; Qian *et al.*, 2003, 2007). However, it is noted that the flap atom (C2) of the five-membered ring points in a different direction with respect to the basal plane, owing to the existence of different H-atom contacts discussed below. Consequently, the distance between atom N1 and atom C6 of

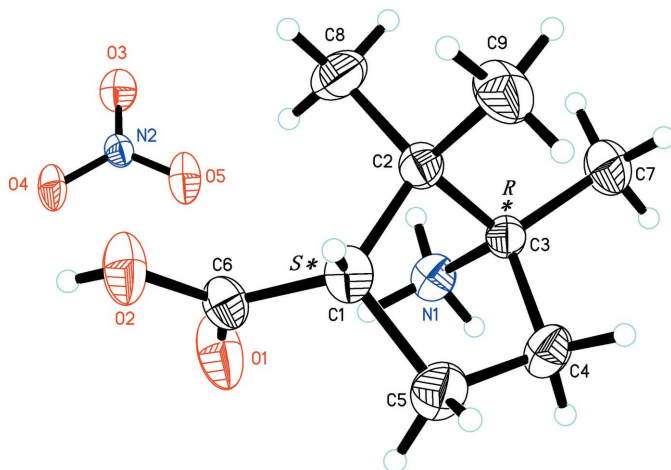


Figure 1
The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

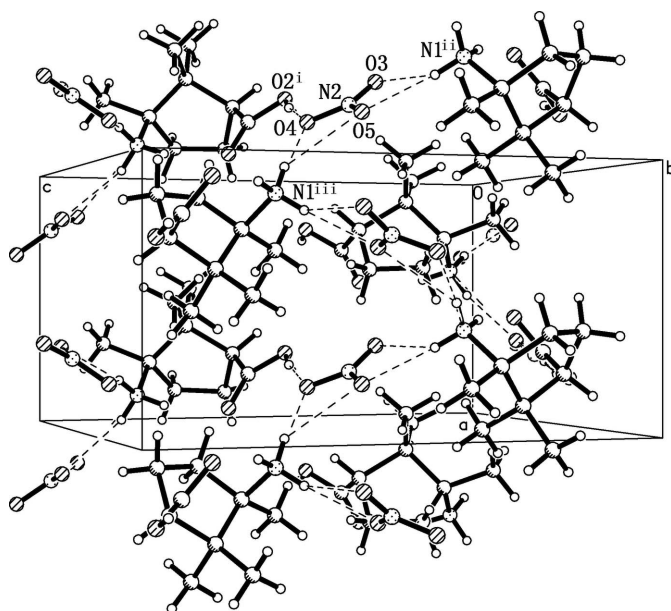


Figure 2
A perspective view of the intermolecular hydrogen-bond contacts in (I) together with the unit cell. Hydrogen bonds are indicated as dashed lines. [Symmetry codes: (i) $\frac{1}{2} + x, \frac{1}{2} - y, 1 - z$; (ii) $1 - x, \frac{1}{2} + y, \frac{3}{2} - z$; (iii) $\frac{1}{2} - x, 1 - y, -\frac{1}{2} + z$.]

the carboxylic acid group is 3.220 (4) Å in (I), while the measured values are 4.943 (5) and 4.960 (3) Å in (1*S*,3*R*)-3-amino-2,2,3-trimethylcyclopentane-1-carboxylic acid hydrochloride and (1*S*,3*R*)-3-carbamoyl-2,2,3-trimethylcyclopentane-1-carboxylic acid, respectively.

In the crystal packing of (I), each nitrate anion links to three adjacent molecules by means of one O—H...O and four N—H...O hydrogen-bonding interactions (Table 2). The donors are the H atoms of the carboxylic acid and amine groups, while the acceptors are the three O atoms of the nitrate anion. Thus, a three-dimensional hydrogen-bonded network is constructed, as illustrated in Fig. 2.

Experimental

Compound (I) was obtained by an anion exchange from Cl[−] to NO₃[−] by adding excess NaNO₃ to an aqueous solution of (1*S*,3*R*)-3-amino-2,2,3-trimethylcyclopentane-1-carboxylic acid hydrochloride (yield 65%). Analysis, calculated for C₉H₁₈N₂O₅: C 46.15, H 7.75, N 11.96%; found: C 46.21, H 7.81, N 11.99%. ESI-MS: *m/z* 172.1 [C₉H₁₇NO₂H]⁺ (100%). Single crystals of (I) suitable for X-ray diffraction measurements were grown from a solution in a mixture of water and methanol (2:1 *v/v*) by slow evaporation in air at room temperature.

Crystal data

C ₉ H ₁₈ NO ₂ ⁺ ·NO ₃ [−]	<i>V</i> = 1195.3 (6) Å ³
<i>M_r</i> = 234.25	<i>Z</i> = 4
Orthorhombic, <i>P</i> 2 ₁ 2 ₁ 2 ₁	Mo <i>K</i> α radiation
<i>a</i> = 7.0696 (19) Å	<i>μ</i> = 0.11 mm ^{−1}
<i>b</i> = 12.473 (3) Å	<i>T</i> = 291 (2) K
<i>c</i> = 13.555 (4) Å	0.50 × 0.40 × 0.30 mm

Data collection

Bruker SMART CCD area-detector diffractometer	7286 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 2000)	1657 independent reflections
<i>T</i> _{min} = 0.951, <i>T</i> _{max} = 0.966	1256 reflections with <i>I</i> > 2σ(<i>I</i>)
	<i>R</i> _{int} = 0.069

Refinement

<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)] = 0.059	145 parameters
<i>wR</i> (<i>F</i> ²) = 0.130	H-atom parameters constrained
<i>S</i> = 1.06	Δ <i>ρ</i> _{max} = 0.25 e Å ^{−3}
1657 reflections	Δ <i>ρ</i> _{min} = −0.16 e Å ^{−3}

Table 1

Selected geometric parameters (Å, °).

C1—C6	1.495 (4)	C6—O1	1.206 (4)
C3—N1	1.504 (4)	C6—O2	1.291 (3)
C3—C7	1.515 (4)		
N1—C3—C7	105.7 (2)	O1—C6—C1	123.4 (3)
O1—C6—O2	122.7 (3)	O2—C6—C1	113.8 (3)

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1 <i>B</i> ...O4 ⁱⁱⁱ	0.89	2.02	2.897 (3)	166
N1—H1 <i>B</i> ...O5 ⁱⁱⁱ	0.89	2.47	3.109 (4)	130
N1—H1 <i>C</i> ...O3 ⁱⁱ	0.89	2.42	3.113 (3)	135
N1—H1 <i>C</i> ...O5 ⁱⁱ	0.89	2.12	2.992 (4)	166
N1—H1 <i>D</i> ...O1	0.89	1.94	2.802 (3)	163
O2—H2 <i>A</i> ...O4 ⁱ	0.81	1.87	2.671 (3)	171
C5—H5 <i>B</i> ...O1	0.97	2.55	2.914 (5)	102

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

H atoms were placed in geometrically idealized positions (C—H = 0.96–0.97 Å, N—H = 0.89 Å and O—H = 0.81 Å) and refined as riding atoms, with *U*_{iso}(H) = 1.5*U*_{eq}(N, O and methyl C) or *U*_{iso}(H) = 1.2*U*_{eq}(C) for the other C atoms. In the absence of significant anomalous scattering, Friedel equivalents were merged before the final refinement.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The authors are indebted to the Major State Basic Research Development Programme (No. 2006CB806104) and the National Natural Science Foundation of China (project No. 20301009) for financial support.

References

Bruker (2000). *SMART* (Version 5.622), *SAINT* (Version 6.02a), *SADABS* (Version 2.03) and *SHELXTL* (Version 6.10). Bruker AXS Inc., Madison, Wisconsin, USA.

- Huang, W., Qian, H.-F., Chen, Y.-H., Gou, S.-H. & Li, Y.-Z. (2003). *Acta Cryst. C* **59**, o479–o480.
- Huang, W., Qian, H. F., Gou, S. H. & Yao, C. (2005). *J. Mol. Struct.* **743**, 183–190.
- Huang, W., Qian, H. F., Li, Y. Z., Chu, Z. L. & Gou, S. H. (2004). *Anal. Sci.* **20**, X75–X76.
- Huang, W., Zhou, Y. S., Li, H. H., Qian, H. F. & Gou, S. H. (2004). *Chin. J. Inorg. Chem.* **20**, 205–209.
- Qian, H. F., Chu, Z. L., Huang, W. & Liu, J. L. (2007). *J. Mol. Struct.* In the press. (DOI: 10.1016/j.molstruc.2006.11.038)
- Qian, H. F., Huang, W. & Gou, S. H. (2003). *J. Mol. Struct.* **658**, 65–70.
- Qian, H. F., Huang, W., Li, H. H. & Chen, Y. (2006). *Chin. J. Struct. Chem.* **25**, 1243–1249.