

## Crystal structure of ammonium iminodiacetate, $\text{NH}_4\text{C}_4\text{H}_6\text{NO}_4$

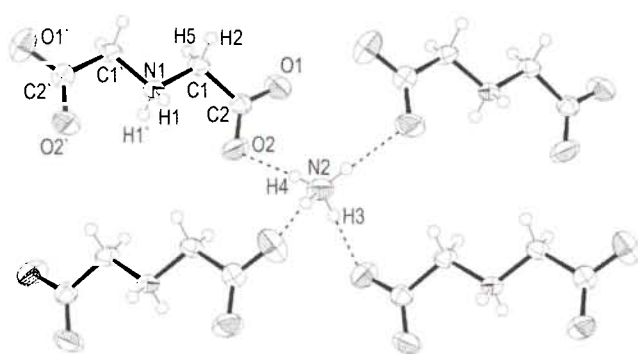
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### Abstract

$\text{C}_4\text{H}_{10}\text{N}_2\text{O}_4$ , orthorhombic, *Pccn* (No. 56),  $a = 7.782(2)$  Å,  $b = 8.642(2)$  Å,  $c = 10.230(3)$  Å,  $V = 688.0$  Å<sup>3</sup>,  $Z = 4$ ,  $R_{\text{gt}}(F) = 0.071$ ,  $wR_{\text{ref}}(F^2) = 0.215$ ,  $T = 293$  K.

### Source of material

Molecular structure of imino-diacetic acid (3-azapentane-1,5-dicarboxylic acid) as well as its hydrohalogenide derivatives, was determined crystallographically [1–4]. In this work, its monoammonium salt as supple colorless crystals, suitable for X-ray analysis, was prepared from mother water-methanol mixture of ammonia, imino-diacetic acid and carbondisulfide in a try to prepare the corresponding dithiocarboxy derivate of the iminodiacetic acid.

### Discussion

Most iminodiacetic derivates synthesized so far are in the form of positively charged iminodiacetic acid ions,  $[\text{C}_4\text{H}_8\text{NO}_4]^+$  [1–3], or in the form of neutral iminodiacetic acid,  $\text{C}_4\text{H}_7\text{NO}_4$  [4]. In this paper, we report the synthesis and crystal structure of ammonium iminodiacetate with negatively charged  $[\text{C}_4\text{H}_6\text{NO}_4]^-$ . Except for carboxylate groups, related interatomic distances and angles are not significantly different in comparison to  $[\text{C}_4\text{H}_8\text{NO}_4]^+$  and  $\text{C}_4\text{H}_7\text{NO}_4$ . The negative charge is most probably spread over the carboxylate groups  $\text{O1}–\text{C2}–\text{O2}$  and  $\text{O1}'–\text{C2}'–\text{O2}'$ . The  $\text{C2}–\text{O1}$  bond length (1.253(3) Å) is somewhat longer than the  $\text{C2}–\text{O2}$  bond (1.242(3) Å). This is probably due to the fact that O1 accepts two hydrogen bonds while O2 only one. All ammonium and

iminodiacetate ions are included in the three-dimensional network of hydrogen bonds. Every N2 atom is hydrogen bonded to four oxygen atoms, and all of them are related to four different symmetrical iminodiacetate groups:  $\text{N2}(i)\cdots\text{O2}(i) = 2.859(2)$  Å,  $\text{H3}(i)\cdots\text{O2}(i) = 1.98(4)$  Å,  $\angle\text{N2}(i)–\text{H3}(i)\cdots\text{O2}(i) = 175(3)^\circ$ ;  $\text{N2}(i)\cdots\text{O2}(ii) = 2.859(2)$  Å,  $\text{H3}(ii)\cdots\text{O2}(ii) = 1.98(4)$  Å,  $\angle\text{N2}(i)–\text{H3}(ii)\cdots\text{O2}(ii) = 175(3)^\circ$ ;  $\text{N2}(i)\cdots\text{O1}(iii) = 2.749(3)$  Å,  $\text{H3}(i)\cdots\text{O1}(iii) = 1.85(3)$  Å,  $\angle\text{N2}(i)–\text{H3}(i)\cdots\text{O1}(iii) = 166(3)^\circ$ ;  $\text{N2}(i)\cdots\text{O1}(iv) = 2.749(3)$  Å,  $\text{H3}(ii)\cdots\text{O1}(iv) = 1.85(3)$  Å,  $\angle\text{N2}(i)–\text{H3}(ii)\cdots\text{O1}(iv) = 166(3)^\circ$ ; (symmetry codes: (i)  $x, y, z$ ; (ii)  $-x+0.5, -y+0.5, z$ ; (iii)  $x, -y+0.5, z+0.5$ ; (iv)  $-x+0.5, y, z+0.5$ ). Atom O1 is also hydrogen bonded to N1 ( $x, y, z$ ):  $\text{N1}(i)\cdots\text{O1}(ii) = 2.783(2)$  Å,  $\text{H1}(i)\cdots\text{O1}(ii) = 1.93(3)$  Å,  $\angle\text{N1}(i)–\text{H1}(i)\cdots\text{O1}(ii) = 167(2)^\circ$ ;  $\text{N1}(i)\cdots\text{O1}(iii) = 2.783(2)$  Å,  $\text{H1}(iv)\cdots\text{O1}(iii) = 1.93(3)$  Å,  $\angle\text{N1}(i)–\text{H1}(iv)\cdots\text{O1}(iii) = 167(2)^\circ$ ; (symmetry codes: (i)  $x, y, z$ ; (ii)  $x+0.5, -y, -z+0.5$ ; (iii)  $-x, y+0.5, -z+0.5$ ; (iv)  $-x+0.5, -y+0.5, z$ ). The plan formed by O1, C2, N1, C2', O1' is planar, maximum displacement from the best plane doesn't exceed 0.0001 Å. But C1, C1', O2 and O2' deviate from this plane by  $-0.066(2)$  Å,  $+0.066(2)$  Å,  $+0.043(2)$  Å and  $-0.043(2)$  Å respectively. These positions of the C1 and C1' atoms decrease repulsion between H atoms bonded to C1 and C1'.

Table 1. Data collection and handling.

Crystal:	colourless, prismatic, size $0.28 \times 0.31 \times 0.38$ mm
Wavelength:	Mo $K_\alpha$ radiation (0.71073 Å)
$\mu$ :	$1.29 \text{ cm}^{-1}$
Diffractometer, scan mode:	Enraf-Nonius CAD-4, $\omega/2\theta$
$2\theta_{\text{max}}$ :	$55.88^\circ$
$N(hkl)_{\text{measured}}, N(hkl)_{\text{unique}}$ :	2854, 998
Criterion for $I_{\text{obs}}, N(hkl)_{\text{gt}}$ :	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$ , 599
$N(\text{param})_{\text{refined}}$ :	67
Programs:	PARST [5], SHELXS-97 [6], SHELXL-97 [7], ORTEP [8]

Table 2. Atomic coordinates and displacement parameters (in Å<sup>2</sup>).

Atom	Site	$x$	$y$	$z$	$U_{\text{iso}}$
H(1)	8e	0.166(3)	-0.214(3)	0.270(2)	0.031(6)
H(2)	8e	0.217(4)	-0.080(4)	0.094(3)	0.052(8)
H(3)	8e	0.334(4)	0.291(4)	0.532(3)	0.055(8)
H(4)	8e	0.299(4)	0.174(4)	0.437(3)	0.056(8)
H(5)	8e	0.398(4)	-0.161(4)	0.083(3)	0.048(6)

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**Table 3.** Atomic coordinates and displacement parameters (in Å<sup>2</sup>).

Atom	Site	x	y	z	<i>U</i> <sub>11</sub>	<i>U</i> <sub>22</sub>	<i>U</i> <sub>33</sub>	<i>U</i> <sub>12</sub>	<i>U</i> <sub>13</sub>	<i>U</i> <sub>23</sub>
N(1)	4 <i>d</i>	1/4	3/4	0.2232(2)	0.023(1)	0.033(1)	0.0215(9)	-0.0004(9)	0	0
C(1)	8 <i>e</i>	0.3151(2)	-0.1207(2)	0.1432(2)	0.0303(9)	0.038(1)	0.0258(8)	-0.0063(8)	0.0014(7)	0.0033(7)
C(2)	8 <i>e</i>	0.3970(2)	0.0048(2)	0.2256(2)	0.028(1)	0.036(1)	0.035(1)	-0.0016(8)	-0.0059(7)	0.0021(9)
O(1)	8 <i>e</i>	0.4610(2)	0.1157(2)	0.1636(2)	0.051(1)	0.054(1)	0.057(1)	-0.0222(8)	-0.0251(8)	0.0170(9)
O(2)	8 <i>e</i>	0.3952(3)	-0.0092(2)	0.3465(2)	0.080(1)	0.056(1)	0.0324(9)	-0.017(1)	-0.0014(7)	-0.0097(8)
N(2)	4 <i>c</i>	1/4	1/4	0.4804(3)	0.048(2)	0.051(2)	0.030(1)	-0.008(2)	0	0

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