Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71817 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1075]

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Ammonium Salt of Nitroacetamide

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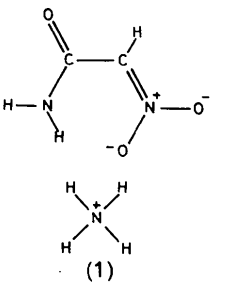
Abstract

The title compound, $NH_4^+.C_2H_3N_2O_3^-$ (1), has a nearly planar anion with an intramolecular hydrogen bond and delocalization of the negative charge. All the H atoms in the ammonium ion are involved in hydrogen bonds.

Comment

The present structure determination is part of a study of aliphatic nitro compounds and their salts, and has been undertaken to study structural changes connected with nitroacetamide salt formation. Compound (1) was prepared as described by Steinkopf (1904) and suitable single crystals were obtained from an ammonia–water solution (25% NH_3) by evaporation at room temperature.



(1)

A least-squares plane defined by all non-H atoms in the anion reveals a nearly planar system with a maximum deviation of 0.071 (7) Å (N1) from the least-squares plane. The angle C1—C2—N2 is 108.6 (2)° in nitroacetamide (Thorup, Dreier & Simonsen, 1981) and changes to 126.9 (4)° by the release of a proton from C2; hence the atom C2 becomes sp^2 hybridized by salt formation.

Apart from C1—N1, all bond lengths involving non-H atoms are changed significantly by the release of a proton from C2 (Table 3). The bond lengths in (1) are very much like the lengths of the corresponding bonds in the anion of nitromalonamide (Table 3) and indicate delocalization of the negative charge over the system O1—C1—C2—N2—(O21,O22).

All H atoms except H2 participate in hydrogen bonds. The stacking of the anions and NH_4^+ ions is stabilized by an infinite network of hydrogen bonds (Fig. 2). The four strongest N3···O hydrogen bonds are in the range 2.798 (5)–2.921 (6) Å. The only intramolecular hydrogen bond, N1—H11···O21, has the dimensions N1—H11 0.98 (3), H11···O21 2.08 (3), N1···O21 2.736 (7) Å.

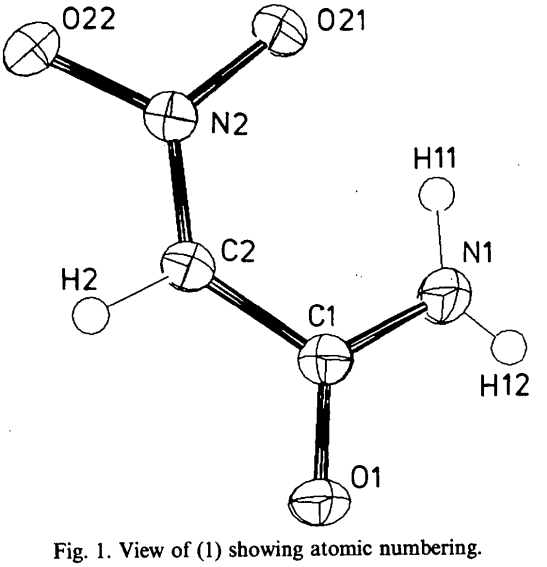


Fig. 1. View of (1) showing atomic numbering.

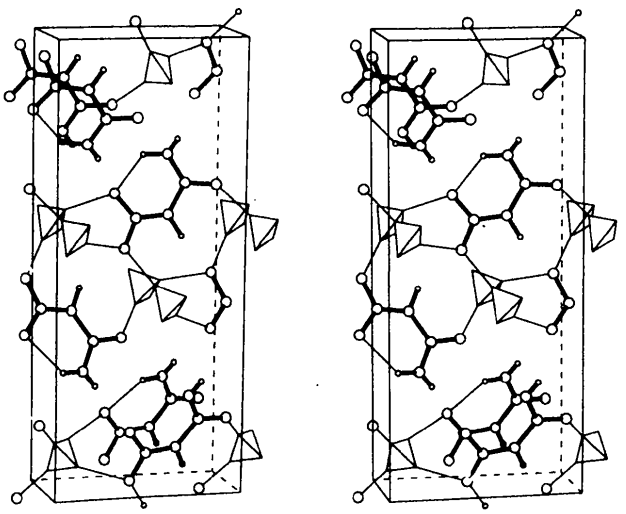


Fig. 2. Stereo drawing (*ATOMS*2.0; Dowty, 1991) of the unit cell of (1). The thin lines represent the intramolecular hydrogen bond and the four strongest N3—H···O hydrogen bonds. Ammonium ions are symbolized by tetrahedra.

Experimental

Crystal data

$NH_4^+.C_2H_3N_2O_3^-$
M_r = 121.10
Orthorhombic
$Pna2_1$
a = 17.887 (2) Å
b = 3.719 (1) Å
c = 7.675 (2) Å
V = 510.6 (3) Å^3
Z = 4
D_x = 1.575 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 8.34–15.60°
μ = 0.135 mm^{-1}
T = 295 K
Needle (cut)
0.33 × 0.20 × 0.10 mm
Pale yellow

Data collection

Enraf–Nonius CAD-4F diffractometer
$\omega/2\theta$ scans
Absorption correction: none
794 measured reflections
794 independent reflections
703 observed reflections [$I > 2.5\sigma(I)$]
θ_{max} = 29.98°

$h = 0 \rightarrow 24$
$k = 0 \rightarrow 5$
$l = 0 \rightarrow 10$
1 intensity and 3 orientation standard reflections
monitored every 100 reflections (orientation) and every 180 min (intensity)
intensity variation: 3%

Refinement

Refinement on F
R = 0.036
wR = 0.027
S = 3.271
703 reflections
93 parameters
Only coordinates of H atoms refined
Weighting scheme based on measured e.s.d.'s

$(\Delta/\sigma)_{max}$ = 0.022
$\Delta\rho_{max}$ = 0.215 e Å^{-3}
$\Delta\rho_{min}$ = −0.206 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2B, 2.3.1)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
O1	0.6683 (1)	0.5168 (6)	0.9156 (8)	0.032 (1)
O21	0.64946 (9)	0.3997 (6)	0.3826 (7)	0.031 (1)
O22	0.53275 (9)	0.5990 (6)	0.4122 (8)	0.033 (1)
N1	0.7298 (1)	0.2980 (8)	0.6820 (8)	0.031 (1)
N2	0.5973 (1)	0.5138 (7)	0.4837 (8)	0.024 (1)
N3	0.5773 (1)	−0.0165 (7)	0.1072 (8)	0.028 (1)
C1	0.6715 (1)	0.4531 (8)	0.7558 (8)	0.024 (1)
C2	0.6056 (2)	0.5505 (8)	0.6522 (8)	0.026 (1)

Table 2. *Selected geometric parameters* (Å, °)

O1^i—C1^i	1.250 (8)	N1^i—C1^i	1.319 (5)
O21^i—N2^i	1.286 (6)	N2^i—C2^i	1.309 (9)
O22^i—N2^i	1.317 (4)	C1^i—C2^i	1.468 (6)
O21^i—N2^i—O22^i	117.7 (6)	O1^i—C1^i—C2^i	116.6 (3)
O21^i—N2^i—C2^i	123.3 (3)	N1^i—C1^i—C2^i	120.6 (5)
O22^i—N2^i—C2^i	119.1 (4)	N2^i—C2^i—C1^i	126.9 (4)
O1^i—C1^i—N1^i	122.7 (4)		

Symmetry code: (i) $\frac{1}{2} + x, \frac{1}{2} - y, z$.

Table 3. *Bond lengths* (Å) *in* (1) *compared with corresponding bond lengths* (Å) *in related compounds*

	(1) [a]	$O_2NCH_2CONH_2$ [b]	$NH_4^+.O_2NC(CONH_2)_2^-$ [c]
C1—O1	1.250 (8)	1.221 (2)	1.253 (4)
C1—N1	1.319 (5)	1.315 (3)	1.336 (4)
C1—C2	1.468 (6)	1.524 (3)	1.468 (4)
C2—N2	1.309 (9)	1.481 (3)	1.327 (4)
N2—O21	1.286 (6)	1.206 (3)	1.286 (4)
N2—O22	1.317 (4)	1.210 (3)	1.300 (4)
N1···O21*	2.736 (7)		2.672 (4)

References: (*a*) this work; (*b*) Thorup, Dreier & Simonsen (1981); (*c*) Simonsen (1981).

* Intramolecular hydrogen bond N1—H11···O21.

Data reduction: *Xtal*3.2 *ADDREF* and *SORTRF* (Hall, Flack & Stewart, 1992; Norrestam & Nielsen, 1982). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *Xtal*3.2 *CRYLSQ*. Software used to prepare material for publication: *Xtal*3.2 *BONDLA*, *LSQPL*, *CIFIO* and *ATABLE*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1110). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1110]

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