nitro group. This elongation of the N—O bond is related to the strong hydrogen bonds involving the O(1) atom. A similar case of N—O bond elongation related to the hydrogen bonding has been found in the structure of ammonium nitrate phase IV (Choi, Mapes & Prince, 1972) and in the structure of 1,2,3-trinitroguanidinium nitrate (Choi & Prince, 1979). The molecular skeleton consisting of the central C atom and the four N atoms is approximately coplanar, with the largest deviation from the mean plane being 0·024 (9) Å (for the C atom). The four atoms in the nitro group are essentially coplanar (χ^2 = 1·07), and the mean plane is twisted out of the plane of the molecular skeleton by 3·2 (5)° [the torsional angle about the N(3)—N(4) axis (right-hand screw system)]. The N(2) nitramine group shows no significant distortion, with almost perfect planarity (χ = 0·02) and twofold symmetry about the N(2)–C axis, but the N(1) nitramine group is clearly non-planar and asymmetric. Both nitramine groups are essentially on the plane of the molecular skeleton, with torsional angles less than 3 standard deviations. The hydrogen-bonding scheme reported by BBHD was confirmed in this study. The bond parameters are given in Table 3. The intramolecular hydrogen bond N(1)—H(1)···O(1) has a very short H···O distance [1·93 (2) Å], which is caused by the molecular conformation. A part of the conformational strain is relieved by an increase of the N(1)—C—N(3) angle [128·5 (6)°] and by a distortion of the N(4) nitro group. Among the four intermolecular hydrogen bonds, N(2)—H(3)···O(2) is particularly strong with an O(2)···H(3) distance of 2·00 (2) Å, and N(2)—H(4)···O(1) is very weak, judging from the O···H distance of 2·37 (2) Å.

Table 3. *Parameters of the hydrogen bonds*

	N—H (Å)	H···O (Å)	N···O (Å)	∠N—H···O (°)
N(1)-H(1)···O(1)	0·96 (2)	1·93 (2)	2·59 (1)	124 (1)
N(1)-H(1)···O(1)′	0·96 (2)	2·25 (2)	3·022 (9)	137 (1)
N(1)–H(2)···N(3)′	0·98 (2)	2·19 (2)	3·146 (7)	164 (1)
N(2)—H(3)···O(2)′	1·00 (2)	2·00 (2)	3·00 (1)	171 (1)
N(2) H(4)···O(1)″	0·96 (2)	2·37 (2)	3·290 (9)	161 (1)

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Structure of Ammonium D-Gluconate

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Abstract. $C_6H_{11}O_7^-.NH_4^+$, orthorhombic, $P2_12_12_1$, a = 6·810 (4), b = 7·630 (4), c = 17·796 (9) Å, M_r = 213·2, V = 924·7 Å^3, Z = 4, D_m = 1·53, D_x = 1·53 Mg m^{-3}, μ(Mo $K\alpha$, λ = 0·71069 Å) = 0·15 mm^{-1}, final R = 0·061 and R_w = 0·055 for 1104 non-zero reflexions. Unlike other α-hydroxycarboxyl moieties the —C(OH)COO$^-$ group is not planar [the O—C—C—O torsion angles are 51·5 (6) and −130·4 (6)°]. All the hydroxy and ammonium H atoms are involved in intermolecular hydrogen bonds.

Introduction. Ammonium D-gluconate was prepared by combining an equimolar ratio of δ-D-gluconolactone (Fluka AG) and ammonium carbonate (POCh) in water. After slow evaporation of water orthorhombic platy crystals of ammonium D-gluconate appeared. Analysis: calculated for $C_6H_{15}NO_7$: C 33·8, H 7·1, N 6·6%; found: C 34·7, H 6·8, N 6·7%. A specimen 0·55 × 0·45 × 0·35 mm was cut from a large crystal and sealed in a capillary.

Weissenberg photographs showed the space group to be $P2_12_12_1$. Data were collected on a Syntex $P2_1$ diffractometer with monochromatized Mo $K\alpha$ radiation. The intensities were measured by the 2θ–ω scan technique. After each group of 100 reflexions two standards were measured; no significant change in intensity was observed. Of 1480 reflexions accessible below 2θ = 60°, 1104 with $I > 1\cdot96\sigma(I)$ were used for

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the structure determination. All calculations were performed on a NOVA 1200 computer with the Syntex *XTL/XTLE Structure Determination System* (1976). Neutral-atom scattering factors were those listed in *International Tables for X-ray Crystallography* (1974). The anomalous dispersion was included for O atoms. The structure was solved by direct methods using the *MULTAN* programs (Syntex version; Germain, Main & Woolfson, 1971) and 197 reflexions with $|E| > 1{\cdot}40$. Full-matrix least-squares refinement first with isotropic, then anisotropic, thermal parameters gave $R = 0{\cdot}096$ and 0·083 respectively. A difference synthesis at this stage showed all H atoms. They were included with isotropic temperature factors, and two cycles of full-matrix refinement yielded a final $R = 0{\cdot}061$ and $R_w = 0{\cdot}055$. A final difference synthesis was featureless.

The final atomic coordinates are listed in Table 1, interatomic distances and angles in Table 2.* A view of the crystal structure down **c** and the atom-numbering scheme are shown in Fig. 1.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36076 (29 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Final positional parameters and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

$B_{eq} = \frac{1}{3}(B_{11} + B_{22} + B_{33})$.

	x	y	z	B_{eq} (Å^2)
C(1)	0·6798 (9)	0·4330 (7)	0·4073 (3)	1·86 (33)
C(2)	0·7133 (8)	0·6283 (7)	0·3901 (3)	1·68 (32)
C(3)	0·6816 (8)	0·6625 (7)	0·3063 (3)	1·67 (33)
C(4)	0·8054 (8)	0·5459 (7)	0·2553 (3)	1·59 (32)
C(5)	0·7624 (8)	0·5821 (7)	0·1721 (3)	1·79 (33)
C(6)	0·8635 (9)	0·4533 (7)	0·1183 (4)	2·11 (37)
O(0)	0·8251 (6)	0·3377 (5)	0·4227 (3)	2·50 (26)
O(1)	0·5047 (6)	0·3803 (5)	0·4025 (2)	2·45 (27)
O(2)	0·5812 (6)	0·7329 (5)	0·4338 (3)	2·10 (25)
O(3)	0·7235 (6)	0·8423 (5)	0·2890 (3)	2·11 (27)
O(4)	1·0096 (6)	0·5774 (5)	0·2704 (3)	2·29 (27)
O(5)	0·5540 (5)	0·5724 (5)	0·1602 (3)	1·95 (25)
O(6)	0·7902 (6)	0·2780 (5)	0·1270 (3)	2·33 (26)
N	1·1773 (9)	0·4501 (7)	0·4907 (3)	2·15 (33)
H(2)	0·861 (7)	0·664 (6)	0·407 (3)	1·5 (11)
H(3)	0·516 (8)	0·623 (6)	0·298 (3)	1·6 (10)
H(4)	0·767 (7)	0·407 (6)	0·268 (3)	1·4 (10)
H(5)	0·815 (9)	0·714 (7)	0·161 (3)	3·3 (13)
H(61)	1·035 (8)	0·469 (7)	0·123 (3)	1·8 (11)
H(62)	0·848 (10)	0·489 (8)	0·065 (4)	3·6 (14)
H(6)	1·075 (10)	0·424 (10)	0·472 (4)	4·0 (18)
H(7)	1·187 (11)	0·575 (8)	0·513 (4)	4·4 (16)
H(8)	1·201 (13)	0·347 (9)	0·532 (4)	6·6 (20)
H(9)	1·309 (13)	0·485 (11)	0·471 (5)	11·5 (20)
H(22)	0·471 (12)	0·720 (11)	0·417 (4)	6·1 (22)
H(33)	0·641 (9)	0·900 (8)	0·304 (4)	2·3 (16)
H(44)	1·093 (9)	0·486 (8)	0·259 (4)	3·5 (16)
H(55)	0·513 (9)	0·679 (7)	0·138 (3)	3·1 (13)
H(66)	0·834 (12)	0·201 (9)	0·165 (5)	9·9 (22)

Table 2. *Interatomic distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

C(1)–O(0)	1·258 (6)	C(1)–O(1)	1·262 (7)
C(1)–C(2)	1·537 (7)	C(2)–O(2)	1·432 (6)
C(2)–C(3)	1·530 (7)	C(3)–O(3)	1·434 (6)
C(3)–C(4)	1·526 (7)	C(4)–O(4)	1·437 (6)
C(4)–C(5)	1·534 (7)	C(5)–O(5)	1·437 (6)
C(5)–C(6)	1·535 (7)	C(6)–O(6)	1·436 (6)
O(0)–C(1)–O(1)	125·0 (5)	O(0)–C(1)–C(2)	119·1 (5)
O(1)–C(1)–C(2)	115·8 (5)	C(1)–C(2)–C(3)	109·8 (4)
C(1)–C(2)–O(2)	109·9 (4)	C(3)–C(2)–O(2)	110·2 (4)
C(2)–C(3)–C(4)	113·8 (4)	C(2)–C(3)–O(3)	110·2 (4)
C(4)–C(3)–O(3)	108·7 (4)	C(3)–C(4)–C(5)	111·3 (4)
C(3)–C(4)–O(4)	109·0 (4)	C(5)–C(4)–O(4)	109·6 (4)
C(4)–C(5)–C(6)	113·6 (5)	C(4)–C(5)–O(5)	108·7 (4)
C(6)–C(5)–O(5)	108·6 (4)	C(5)–C(6)–O(6)	112·0 (5)

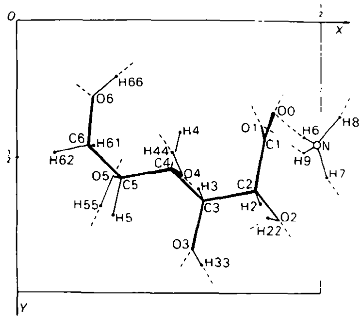

Fig. 1. The crystal structure viewed down **c** showing atom numbering.

Table 3. *Geometry of the hydrogen bonds*

X–H···O	X···O	X–H	H···O	X–H···O
N–H(6)···O(0)	2·819 (6) Å	0·80 (7) Å	2·02 (7) Å	175 (7)°
N–H(7)···O(2^{i})	2·843 (6)	1·03 (6)	1·89 (7)	152 (6)
N–H(8)···O(0^{ii})	2·865 (6)	1·09 (7)	1·83 (8)	157 (6)
N–H(9)···O(1^{iii})	2·779 (6)	1·00 (9)	1·97 (9)	136 (7)
O(2)–H(22)···O(6^{iv})	2·772 (5)	0·81 (8)	2·00 (8)	160 (8)
O(3)–H(33)···O(5^{iv})	2·734 (5)	0·77 (6)	1·97 (6)	173 (6)
O(4)–H(44)···O(3^{v})	2·763 (5)	0·92 (6)	1·87 (6)	163 (6)
O(5)–H(55)···O(1^{iv})	2·631 (5)	0·95 (6)	1·70 (6)	167 (5)
O(6)–H(66)···O(4^{v})	2·745 (5)	0·95 (8)	1·83 (8)	163 (7)

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

Discussion. The structure is built up from ammonium cations and gluconate anions held together by hydrogen bonding involving all hydroxy and ammonium H atoms. The data on these hydrogen bonds are summarized in Table 3.

All bond distances and angles in the gluconate anion are normal and comparable with those found in other D-gluconate salts. The gluconate anion has a conformation similar to one of the two most probable bent-chain conformations predicted by Jeffrey & Kim (1970). This conformation can be derived from the planar conformation found earlier in potassium D-gluconate (Littleton, 1953) and in the orthorhombic

Table 4. *Torsion angles* (°) *for the gluconate ion*

O(0) C(1) C(2) O(2)	130·4 (6)		O(1) C(1)–C(2) O(2)	51·5 (6)
C(1) C(2) C(3) C(4)	54·4 (7)		C(2) C(3)–C(4) C(5)	178·2 (5)
C(3) C(4) C(5) C(6)	−172·8 (6)		C(4) C(5)–C(6) O(6)	65·3 (6)
O(2) C(2) C(3) C(4)	175·5 (6)		O(2) C(2) C(3)–O(3)	62·2 (7)

modification of potassium D-gluconate monohydrate (Panagiotopoulos, Jeffrey, La Placa & Hamilton, 1974) and in one of two different gluconate ions in manganese(II) D-gluconate dihydrate (Lis, 1979), by rotating 125° about C(2)—C(3) (see Fig. 1 and Table 4). Other types of bent-chain conformations were found earlier in the monoclinic modification of $KC_6H_{11}O_7.H_2O$ (Panagiotopoulos *et al.*, 1974), in trisodium 6-phospho-D-gluconate dihydrate (Smith, Fitzgerald, Caughlan, Kerr & Ashmore, 1974) and in one of the gluconate ions of $Mn(C_6H_{11}O_7)_2.2H_2O$ (Lis, 1979). The most interesting fact is that contrary to all other α-hydroxycarboxylic moieties (Newton & Jeffrey, 1977) the $-C(OH)COO^-$ group is not planar (Table 4). It may be assumed that the deviation of the O(2) atom from the plane of the carboxylate group is brought about by the hydrogen bonds in this salt. It is noteworthy that each O atom is involved in two H bonds (Table 3).

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Structures of Tris(2-cyanoethyl)phosphine Sulphide, $P(CH_2CH_2CN)_3S$ and Tris(2-cyanoethyl)phosphine Selenide, $P(CH_2CH_2CN)_3Se$

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Abstract. $C_9H_{12}N_3PS$ and $C_9H_{12}N_3PSe$, triclinic, $P\bar{1}$, $Z = 2$. Cell dimensions, $(PCH_2CH_2CN)_3S$: $a = 8{\cdot}363\ (8)$, $b = 9{\cdot}026\ (4)$, $c = 9{\cdot}777\ (6)$ Å, $\alpha = 98{\cdot}75\ (4)$, $\beta = 107{\cdot}31\ (5)$, $\gamma = 115{\cdot}85\ (5)$°, $V = 599{\cdot}1\ (3)$ Å^3, $D_c = 1{\cdot}252\ (3)$, $D_o = 1{\cdot}247\ (5)$ Mg m^{-3}, final $R = 0{\cdot}095$ for a total of 738 independent observed reflections; $P(CH_2CH_2CN)_3Se$: $a = 8{\cdot}497\ (5)$, $b = 9{\cdot}148\ (2)$, $c = 9{\cdot}972\ (8)$ Å, $\alpha = 98{\cdot}85\ (2)$, $\beta = 107{\cdot}20\ (6)$, $\gamma = 116{\cdot}96\ (3)$°, $V = 621{\cdot}1\ (3)$ Å^3, $D_c = 1{\cdot}467\ (3)$, $D_o = 1{\cdot}477\ (3)$ Mg m^{-3}, final $R = 0{\cdot}081$ for a total of 701 independent observed reflections. The two compounds are isostructural and the individual molecules have C_1 internal symmetry: two of the cyanoethyl groups are related by an imaginary mirror plane which includes the P—S(Se) bond but the third cyanoethyl group does not lie in this plane.

Introduction. Tris(2-cyanoethyl)phosphine sulphide and tris(2-cyanoethyl)phosphine selenide, $P(CH_2CH_2CN)_3X$ (X = S,Se), are triclinic, with almost identical unit-cell dimensions and closely related X-ray powder diffraction patterns (Blake, Howie & McQuillan, 1979). The corresponding oxide is trigonal, with C_3 internal molecular symmetry (Blake, Howie & McQuillan, 1981). The vibrational spectrum of the oxide is much simpler than those of the sulphide or selenide (Blake, 1980) suggesting a lower molecular symmetry for the latter compounds. We now report structure determinations for $P(CH_2CH_2CN)_3S$ and $P(CH_2CH_2CN)_3Se$.

The compounds were prepared and unit-cell parameters determined as described previously (Blake *et al.*, 1979). Single-crystal diffraction data were obtained using the equi-inclination multiple-film Weissenberg technique, and intensities were measured by the SRC

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