C51—O511—C511	117.2 (8)	O42—C41—N4	111.4 (8)
C53—O531—C531	117.0 (9)	O41—C41—N4	122.2 (10)
C54—O541—C541	115.6 (9)	O41—C41—O42	126.0 (9)
N2—N1—C6	118.9 (9)	O42—C42—C43	111.5 (10)
N1—N2—C21	120.5 (8)	O511—C51—C5	103.2 (7)
N1—N2—N3	118.6 (8)	C5—C51—C52	111.4 (8)
N3—N2—C21	120.3 (8)	O511—C51—C52	109.9 (7)
N2—N3—C31	120.3 (8)	O521—C52—C51	106.4 (7)
N2—N3—N4	111.3 (7)	C51—C52—C53	116.2 (8)
N4—N3—C31	116.0 (8)	O521—C52—C53	109.4 (7)
N3—N4—C41	120.1 (8)	O531—C53—C52	109.6 (8)
N3—N4—C5	114.2 (7)	C52—C53—C54	114.6 (9)
C5—N4—C41	120.1 (8)	O531—C53—C54	108.1 (9)
N4—C5—C51	112.2 (8)	O541—C54—C53	107.6 (9)
N4—C5—C6	108.8 (8)	O511—C511—O512	122.7 (11)
C6—C5—C51	111.8 (8)	O512—C511—C512	125.3 (12)
N1—C6—C5	125.2 (10)	O511—C511—C512	111.4 (10)
N2—C21—C26	122.2 (10)	O521—C521—O522	124.7 (10)
N2—C21—C22	116.7 (10)	O522—C521—C522	126.8 (12)
C22—C21—C26	121.0 (11)	O521—C521—C522	108.3 (11)
C21—C22—C23	118.2 (11)	O531—C531—O532	121.5 (13)
C22—C23—C24	117.6 (13)	O532—C531—C532	129.6 (14)
Cl—C24—C23	117.7 (12)	O531—C531—C532	108.6 (12)
C23—C24—C25	123.7 (4)	O541—C541—O542	122.5 (12)
Cl—C24—C25	118.4 (12)	O542—C541—C542	122.4 (13)
C24—C25—C26	118.0 (14)	O541—C541—C542	115.3 (11)
C21—C26—C25	121.1 (13)	O32—C31—N3	109.8 (8)

Preliminary Weissenberg photographs indicated that the crystal belonged to the orthorhombic system with systematic absences consistent with the $P2_12_12_1$ space group. Corrections were made for Lorentz and polarization effects. An empirical absorption correction following the *DIFABS* procedure (Walker & Stuart, 1983) was applied to anisotropically refined data. The structure was solved by direct methods using *SIR*88 (Burla, Camalli, Cascarano, Giacovazzo, Polidori, Spagna & Viterbo, 1989). After anisotropic refinement by full-matrix least squares of all of the 43 non-H atoms in the asymmetric unit, the H atoms were assigned the same isotropic displacement parameters as the atoms to which they were bonded and were included but not refined in the final stage of refinement. Refinement was over nine parameters per atom plus one for scale; the over-determination ratio was 4.8 reflections/parameter. All calculations were carried out with crystallographic programs of the *XRAY*70 System (Stewart, Kundell & Baldwin, 1970). Bond lengths and angles were calculated by the program *PARST* (Nardelli, 1983*a*).

We thank Dr Palacios for supplying the crystals and the Junta de Andalucía and DGICYT (PB89–0540) for financial support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: LI1072). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Aminoguanidinium Nitrate

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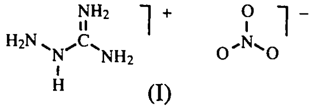
Abstract

The structure of aminoguanidinium nitrate, $CH_7N_4^+.NO_3^-$, has been determined by single-crystal X-ray methods. The structure is a salt complex containing discrete aminoguanidinium and nitrate ions. The O atoms of the nitrate group are involved in intermolecular bonds with the H atoms of the aminoguanidinium ion.

Comment

As part of a program aimed at synthesizing new optical materials having significant birefringence, we have prepared the compound aminoguanidinium nitrate, (I). Several guanidinium salts have been studied over the last two decades (Adams & Small, 1974) and the chloride (Bryden, 1957), dihydrogenphosphate (Adams, 1977) and sulfate (Mullen & Hellner, 1978) salts have been structurally characterized. In the oxoanion salts, multiple hydrogen bonds to O atoms are present, each O atom accepting three H atoms. Similar hydrogen-bond interactions are observed in the present structure.



(I)

A labeled drawing of the molecular units is given in Fig. 1. The aminoguandinium moiety is nearly planar with C—N distances and interatomic angles similar to those found in other guanidinium and substituted guanidinium salts [torsion angle N1—C1—N3—N4 is 4.0 (3)°]. A packing diagram is given in Fig. 2. The nitrate and guanidinium moieties are approximately coplanar. Within the same plane the nitrate groups are linked through hydrogen bonds to the N atoms of the guanidinium portion of the molecule. Above and below this plane, the groups are bonded intermolecularly through the H atoms of the amino group (N4, H6 and H7).

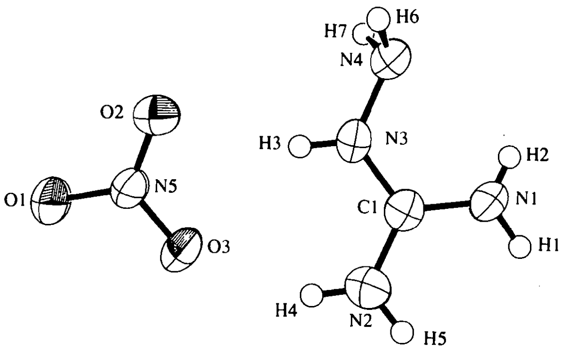


Fig. 1. *ORTEP* (Johnson, 1976) plot of the title structure. Displacement ellipsoids are plotted at the 50% probability level.

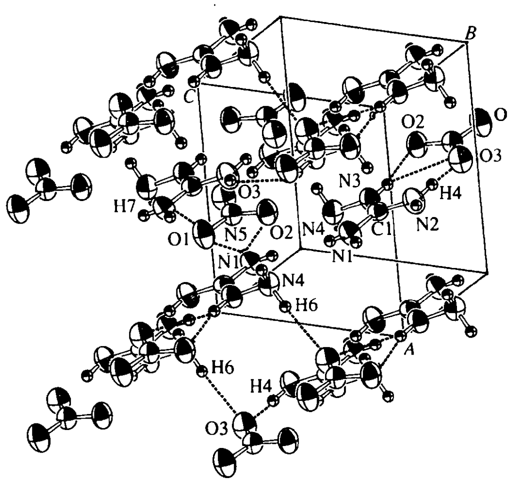


Fig. 2. Packing diagram for aminoguandinium nitrate. Hydrogen-bond interactions are indicated by dashed lines.

Experimental

The crystals were formed by slow evaporation of a solution that was prepared by the stoichiometric addition of aminoguanidinium hydrochloride (Aldrich, 99.9%) to an aqueous solution of sodium nitrate (Aldrich, 99.9%).

Crystal data

$CH_7N_4^+.NO_3^-$
$M_r = 137.10$
Triclinic
$P\bar{1}$
$a = 7.341$ (1) Å
$b = 7.722$ (2) Å
$c = 6.024$ (1) Å
$\alpha = 110.47$ (2)°
$\beta = 102.64$ (2)°
$\gamma = 104.87$ (2)°
$V = 290.7$ (1) Å^3
$Z = 2$
$D_x = 1.566$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 20 reflections
$\theta = 10$–14°
$\mu = 0.136$ mm^{-1}
$T = 296$ K
Block
0.25 × 0.15 × 0.15 mm
Colorless

Data collection

Rigaku AFC-6*R* diffractometer
ω–2θ scans
Absorption correction: none
3384 measured reflections
1692 independent reflections
1126 observed reflections [$I > 3\sigma(I)$]

$R_{\rm int} = 0.076$
$\theta_{\rm max} = 30°$
$h = -10 \rightarrow 10$
$k = -10 \rightarrow 10$
$l = -8 \rightarrow 8$
3 standard reflections monitored every 200 reflections intensity variation: 1.9%

Refinement

Refinement on F
$R = 0.044$
$wR = 0.054$
$S = 1.44$
1126 reflections
110 parameters
All H-atom parameters refined
Weighting scheme based on measured e.s.d.'s (*TEXSAN*; Molecular Structure Corporation, 1989)

$(\Delta/\sigma)_{\rm max} = 0.01$
$\Delta\rho_{\rm max} = 0.27$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.40$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.4.6B)

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

$$B_{\rm eq} = (8\pi^2/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
O1	0.8178 (2)	0.4752 (2)	0.2681 (2)	4.82 (4)
O2	0.6710 (2)	0.4385 (2)	−0.1057 (2)	4.36 (4)
O3	0.5573 (2)	0.2153 (2)	0.0154 (3)	4.74 (4)
N1	0.0380 (2)	−0.1698 (2)	−0.8063 (3)	3.88 (4)
N2	0.2316 (2)	−0.1301 (2)	−0.4248 (3)	4.61 (5)
N3	0.2980 (2)	0.1237 (2)	−0.5409 (3)	3.81 (4)
N4	0.2462 (2)	0.2045 (2)	−0.7091 (3)	4.00 (4)
N5	0.6820 (2)	0.3772 (2)	0.0583 (2)	3.35 (3)
C1	0.1875 (2)	−0.0603 (2)	−0.5925 (3)	3.21 (4)
H1	−0.036 (3)	−0.286 (3)	−0.834 (4)	4.6 (4)
H2	0.007 (3)	−0.128 (3)	−0.901 (4)	4.1 (4)
H3	0.396 (3)	0.192 (3)	−0.406 (5)	5.3 (5)
H4	0.338 (3)	−0.062 (3)	−0.286 (5)	6.5 (6)
H5	0.172 (3)	−0.254 (3)	−0.474 (4)	4.7 (4)
H6	0.352 (3)	0.240 (3)	−0.768 (5)	6.4 (5)
H7	0.230 (3)	0.328 (3)	−0.617 (5)	6.3 (5)

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

O1—N5	1.248 (2)	N2—H4	0.88 (2)
O2—N5	1.232 (2)	N2—H5	0.85 (2)
O3—N5	1.246 (2)	N3—N4	1.399 (3)
N1—C1	1.312 (2)	N3—C1	1.328 (2)
N1—H1	0.86 (2)	N3—H3	0.84 (2)
N1—H2	0.77 (2)	N4—H6	0.94 (2)
N2—C1	1.320 (3)	N4—H7	0.97 (2)
O1···N1$^{\rm i}$	3.038 (2)	O2···N3	3.043 (2)
O1···N2$^{\rm ii}$	3.088 (2)	O3···N2$^{\rm ii}$	2.983 (2)
O1···N4$^{\rm iii}$	3.153 (2)	O3···N2	2.977 (2)
O2···N1$^{\rm i}$	3.087 (2)	O3···N4$^{\rm iv}$	3.104 (2)
O1—N5—O3	119.2 (1)	N1—C1—N3	120.4 (1)
O1—N5—O2	120.8 (1)	N1—C1—N2	121.0 (1)
O3—N5—O2	120.1 (1)	N3—C1—N2	118.6 (1)

Symmetry codes: (i) $1 + x, 1 + y, 1 + z$; (ii) $1 - x, -y, -z$; (iii) $1 - x, 1 - y, -z$; (iv) $x, y, 1 + z$.

All H atoms were located from difference electron density maps and refined with isotropic displacement coefficients.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Data reduction: *TEXSAN* (Molecular Structure Corporation, 1989). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEP*II (Johnson, 1976).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, including H-atom geometry, have been deposited with the IUCr (Reference: BK1062). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1994). C50, 1976–1978

2,2,4,4-Tetrakis(3,5-dimethylpyrazol-1-yl)-$2\lambda^5,4\lambda^5,6\lambda^5$-cyclotriphosphaza-1,3,5-triene-6-spiro-2′-1′,3′-diaza-2′-phosphacyclohexane, C$_{23}$H$_{36}$N$_{13}$P$_3$

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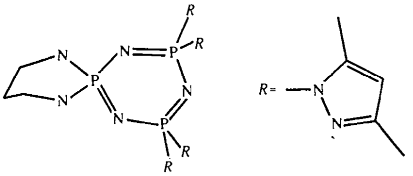
Abstract

The title phosphazene, P$_3$N$_3R_4XX$, where R is 3,5-dimethylpyrazole and XX is a spiro-bonded —NH—CH$_2$CH$_2$CH$_2$NH— ring, has P—N bonds of 1.554 (5)–1.614 (5) Å in the phosphazene ring with exocylic bonds of 1.609 (5) and 1.635 (5) Å to the spiro ring and 1.683 (4)–1.708 (5) Å to the pyrazole groups. The N—P—N angle within the phosphazene ring at the unique P atom is 6.9 (3)° smaller than those at the other P atoms. One of the pyrazole-substituted P atoms is displaced by 0.252 (5) Å from the best plane of the other five ring atoms, which are coplanar to within 0.087 (6) Å. The —PNCCCN— ring conformation is intermediate between a boat and a twist-boat.

Comment

The determination of the structure of the title compound, (I), was undertaken to obtain structural parameters for comparison with proposed metal complexes which utilize this molecule as a ligand (Thomas, Chandrasekhar, Scott, Hallford & Cordes, 1993).



(I)

The alternation of the P—N bond lengths in the phosphazene ring found here has been noted previously for unsymmetrically substituted rings (Cordes, Swepston, Oakley, Paddock & Ranganathan, 1981), and the long ring bonds and small ring angle in the phosphazene at P1

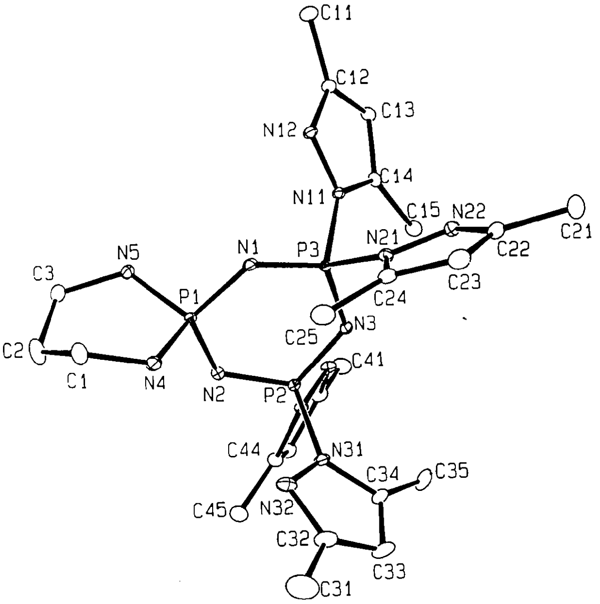


Fig. 1. An *ORTEP*II (Johnson, 1976) drawing of the molecule showing the atom-numbering scheme. Displacement ellipsoids are shown at the 5% probability level and H atoms have been omitted for clarity.