shortest intermolecular contact is O(1)···O(6^{ii}) = 2·789 (3) Å, (ii) = $\bar{x}, \bar{y}, 1-z$.

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Structure of Isopropylammonium *cyclo*-Triphosphate

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Abstract. $[NH_3(C_3H_7)]_3P_3O_9$, $M_r = 417{\cdot}27$, monoclinic, $C2$, $a = 25{\cdot}22\ (2)$, $b = 12{\cdot}22\ (2)$, $c = 15{\cdot}45\ (2)$ Å, $\beta = 123{\cdot}90\ (5)°$, $V = 3953\ (17)$ Å^3, $Z = 8$, $D_x = 1{\cdot}402$ Mg m^{-3}, $\lambda(\text{Mo } K\bar{\alpha}) = 0{\cdot}7107$ Å, $\mu = 0{\cdot}350$ mm^{-1}, $F(000) = 1776$, $T = 293$ K, final $R = 0{\cdot}039$ for 5327 independent reflexions. The P_3O_9 ring anions are aligned in rows parallel to the c axis. These rows arranged in an almost hexagonal way form large hexagonal channels lined by the isopropylammonium groups.

Introduction. A systematic investigation of isopropylammonium phosphates, condensed or not, has shown the existence of a monophosphate: $[C_3H_{10}N][H_2PO_4]$ (Averbuch-Pouchot, Durif & Guitel, 1988*a*) and of a *cyclo*-tetraphosphate: $[C_3H_{10}N]_4P_4O_{12}{\cdot}\frac{3}{2}H_2O$ (Averbuch-Pouchot, Durif & Guitel, 1988*b*). In the present work we describe the chemical preparation and crystal structure of the *cyclo*-triphosphate.

Experimental. Crystalline samples of isopropylammonium *cyclo*-triphosphates have been prepared by using the metathesis reaction first described by Boullé (1938) and extensively used in organic chemistry for the preparation of water-soluble *cyclo*-triphosphates. To a slurry of silver *cyclo*-triphosphate and water, the stoichiometric amount of an aqueous solution of isopropylamine hydrochloride is slowly added:

$$Ag_3P_3O_9 + 3C_3H_{10}NCl \rightarrow 3AgCl + [C_3H_{10}N]_3P_3O_9.$$

After removing the silver chloride by filtration, the resulting solution is kept at room temperature until formation of the crystals, which appear as pseudo-hexagonal thick plates. Several recrystallizations are necessary to obtain good quality crystals.

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Density not measured. Plate fragment 0·25 × 0·30 × 0·30 mm. Enraf–Nonius CAD-4 diffractometer, graphite monochromator. Systematic absences: hkl, $h + k = 2n$. 19 reflexions ($12 < \theta < 18°$) for refining unit-cell dimensions. ω scan. 7386 reflexions measured ($3 < \theta < 32{\cdot}5°$), $\pm h,k,l$, $h_{max} = 39$, $k_{max} = 18$, $l_{max} = 21$. Scan width: 1·20°, scan speed between 0·02 and 0·04° s^{-1}, total background measuring time: between 17 and 30 s.

Two orientation (426 and $4\bar{2}6$) and two intensity (10,2,0 and $\bar{1}0,\bar{2},0$) reference reflexions, no variation. Lorentz and polarization corrections, no absorption correction. Crystal structure solved by direct methods (*MULTAN*; Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1977). Anisotropic full-matrix least-squares refinement (on F) for non-hydrogen atoms, isotropic for H atoms. Unit weights. Final refinement with 5327 reflexions ($I > 3\sigma_I$). Final $R = 0{\cdot}039$ ($wR = 0{\cdot}039$). For the complete set of unique reflexions (6473) $R = 0{\cdot}049$. Extinction not refined. Max. $\Delta/\sigma = 0{\cdot}26$ [β_{33} of C(22)]. Maximum peak height in final difference Fourier map: 0·267 e Å^{-3}. $S = 0{\cdot}702$. Scattering factors for neutral atoms and f', f'' from *International Tables for X-ray Crystallography* (1974). Enraf–Nonius (1977) *SDP* used for all calculations. Computer used: MicroVAX II.

Discussion. Final atomic coordinates are given in Table 1.* Two crystallographically independent P_3O_9 ring anions and six independent isopropylammonium groups

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and distances involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51213 (53 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Final atomic coordinates and* B_{eq} *for* $(C_3H_{10}N)_3P_3O_9$

$B_{eq} = \frac{4}{3}\sum_i\sum_j \mathbf{a}_i.\mathbf{a}_j\beta_{ij}$.

	x	*y*	*z*	B_{eq}(Å²)
P(1)	0·38382 (3)	0·5	0·32034 (5)	2·80 (2)
P(2)	0·34643 (3)	0·71725 (7)	0·23666 (5)	2·56 (1)
P(3)	0·28453 (4)	0·52857 (8)	0·10424 (6)	3·02 (2)
P(4)	0·09561 (3)	−0·02382 (7)	0·21506 (5)	2·63 (1)
P(5)	0·28539 (3)	0·51420 (7)	0·58868 (5)	2·68 (1)
P(6)	0·17150 (3)	0·16353 (8)	0·24098 (6)	3·16 (2)
O(*E*11)	0·34462 (9)	0·4916 (3)	0·3631 (2)	4·39 (6)
O(*E*12)	0·4445 (1)	0·4413 (3)	0·3722 (2)	4·92 (6)
O(*L*12)	0·3964 (1)	0·6273 (2)	0·3115 (2)	3·53 (5)
O(*L*13)	0·3426 (1)	0·4664 (2)	0·1997 (2)	3·90 (5)
O(*E*21)	0·1275 (1)	0·2742 (3)	0·8155 (2)	4·93 (7)
O(*E*22)	0·1732 (1)	0·2850 (2)	0·7066 (2)	4·10 (6)
O(*L*23)	0·28642 (9)	0·6439 (2)	0·1551 (2)	3·03 (5)
O(*E*31)	0·2247 (1)	0·4750 (3)	0·0771 (3)	7·7 (1)
O(*E*32)	0·3016 (1)	0·5430 (3)	0·0281 (2)	5·98 (8)
O(*E*41)	0·04371 (9)	−0·0371 (2)	0·2318 (2)	3·72 (5)
O(*E*42)	0·0901 (1)	0·9218 (3)	0·1262 (2)	4·61 (6)
O(*L*45)	0·1618 (1)	0·9395 (2)	0·3188 (2)	3·37 (5)
O(*E*51)	0·1874 (1)	0·0640 (3)	0·4650 (2)	4·93 (7)
O(*E*52)	0·2739 (1)	0·9511 (3)	0·4746 (2)	5·55 (9)
O(*L*56)	0·2243 (1)	0·1054 (3)	0·3478 (2)	4·40 (6)
O(*E*61)	0·3348 (1)	0·7793 (3)	0·7419 (3)	6·04 (9)
O(*E*62)	0·3153 (1)	0·6326 (3)	0·8368 (2)	5·52 (7)
O(*L*46)	0·10746 (9)	0·1054 (2)	0·2150 (2)	2·89 (5)
N(1)	0·2208 (1)	0·4809 (3)	0·3141 (2)	3·11 (5)
N(2)	0·7815 (1)	0·5016 (3)	0·1894 (2)	3·49 (6)
N(3)	0·4321 (1)	0·4107 (2)	0·5696 (2)	2·77 (5)
N(4)	0·3501 (1)	0·7911 (2)	−0·0069 (2)	3·08 (6)
N(5)	0·3445 (1)	0·7678 (3)	0·4929 (2)	3·38 (6)
N(6)	0·4157 (1)	0·4345 (2)	0·0563 (2)	3·00 (5)
C(11)	0·1713 (2)	0·5594 (3)	0·2421 (3)	3·53 (8)
C(12)	0·1933 (2)	0·6745 (4)	0·2776 (3)	4·6 (1)
C(13)	0·1098 (2)	0·5340 (4)	0·2313 (5)	7·7 (2)
C(21)	0·3403 (2)	0·0697 (4)	0·2453 (5)	7·3 (2)
C(22)	0·3810 (2)	0·0338 (5)	0·2096 (4)	6·7 (1)
C(23)	0·3242 (3)	0·1887 (4)	0·2120 (6)	8·5 (2)
C(31)	0·0804 (2)	0·7933 (3)	0·4567 (3)	3·63 (8)
C(32)	0·3539 (2)	0·2634 (4)	0·5128 (4)	5·2 (1)
C(33)	0·4702 (2)	0·2257 (5)	0·6292 (5)	7·7 (2)
C(41)	0·0854 (2)	0·3193 (4)	−0·0177 (3)	3·99 (8)
C(42)	0·0635 (2)	0·4239 (5)	0·9203 (5)	6·7 (2)
C(43)	0·4589 (2)	0·7232 (5)	0·0704 (4)	6·2 (1)
C(51)	0·0903 (2)	0·2923 (4)	0·4156 (3)	4·22 (9)
C(52)	0·4304 (2)	0·9002 (5)	0·5668 (6)	8·1 (2)
C(53)	0·0467 (2)	0·1979 (5)	0·3964 (4)	6·5 (1)
C(61)	0·0941 (1)	0·8172 (3)	0·9273 (2)	3·29 (7)
C(62)	0·1617 (2)	0·7844 (4)	0·0119 (3)	4·9 (1)
C(63)	0·4548 (2)	0·2483 (3)	0·0712 (3)	4·5 (1)

Table 2. *Main interatomic distances* (Å) *and bond angles* (°) *in the arrangement of* $(C_3H_{10}N)_3P_3O_9$

The PO_4 tetrahedra are described by double-entry tables. P—O distances are underlined, O—P—O angles are given in the lower-left triangles and O—O distances in the corresponding upper-right triangles.

The first P_3O_9 ring anion

$P(1)O_4$ tetrahedron

P(1)	O(*E*11)	O(*E*12)	O(*L*12)	O(*L*13)
O(*E*11)	1·468 (3)	2·520 (4)	2·501 (4)	2·516 (4)
O(*E*12)	118·8 (2)	1·460 (3)	2·501 (4)	2·482 (3)
O(*L*12)	108·6 (2)	109·0 (1)	1·610 (3)	2·466 (3)
O(*L*13)	110·1 (1)	100·4 (2)	100·4 (1)	1·600 (2)

$P(2)O_4$ tetrahedron

P(2)	O(*L*12)	O(*E*21)	O(*E*22)	O(*L*23)
O(*L*12)	1·584 (2)	2·475 (4)	2·514 (4)	2·469 (2)
O(*E*21)	108·2 (2)	1·470 (4)	2·518 (5)	2·522 (4)
O(*E*22)	110·4 (1)	117·4 (2)	1·477 (3)	2·479 (3)
O(*L*23)	101·8 (1)	110·5 (1)	107·4 (1)	1·598 (2)

$P(3)O_4$ tetrahedron

P(3)	O(*L*13)	O(*L*23)	O(*E*31)	O(*E*32)
O(*L*13)	1·577 (2)	2·470 (3)	2·482 (3)	2·432 (4)
O(*L*23)	101·9 (1)	1·602 (3)	2·459 (4)	2·520 (4)
O(*E*31)	108·7 (2)	106·0 (2)	1·476 (4)	2·581 (6)
O(*E*32)	105·8 (2)	110·2 (2)	122·3 (2)	1·470 (4)

Table 2 (*cont.*)

P(1)–P(2)	2·8735 (8)	P(1)–P(2)–P(3)	59·58 (2)
P(1)–P(3)	2·8659 (6)	P(1)–P(3)–P(2)	59·84 (2)
P(2)–P(3)	2·8948 (11)	P(2)–P(1)–P(3)	60·58 (2)

P(1)–O(*L*12)–P(2) 128·3 (1)
P(1)–O(*L*13)–P(3) 128·8 (2)
P(2)–O(L23)–P(3) 129·5 (1)

The second P_3O_9 ring anion

$P(4)O_4$ tetrahedron

P(4)	O(*E*41)	O(*E*42)	O(*L*45)	O(*L*46)
O(*E*41)	1·478 (3)	2·533 (4)	2·509 (3)	2·481 (4)
O(*E*42)	119·0 (2)	1·461 (3)	2·484 (3)	2·539 (4)
O(*L*45)	108·9 (1)	108·2 (1)	1·604 (2)	2·472 (3)
O(*L*46)	106·9 (2)	111·5 (2)	100·6 (1)	1·608 (2)

$P(5)O_4$ tetrahedron

P(5)	O(*L*45)	O(*E*51)	O(*E*52)	O(*L*56)
O(*L*45)	1·589 (2)	2·492 (4)	2·492 (3)	2·451 (4)
O(*E*51)	109·0 (2)	1·471 (4)	2·515 (5)	2·507 (5)
O(*E*52)	109·1 (2)	117·7 (2)	1·469 (3)	2·496 (4)
O(*L*56)	100·8 (1)	109·8 (2)	109·2 (2)	1·592 (3)

$P(6)O_4$ tetrahedron

P(6)	O(*L*56)	O(*E*61)	O(*E*62)	O(*L*46)
O(*L*56)	1·597 (2)	2·522 (4)	2·464 (4)	2·480 (2)
O(*E*61)	110·8 (2)	1·465 (3)	2·533 (5)	2·450 (4)
O(*E*62)	107·0 (2)	119·7 (2)	1·465 (4)	2·508 (4)
O(*L*46)	101·9 (1)	106·2 (2)	109·9 (1)	1·597 (2)

P(4)–P(5)	2·8749 (8)	P(4)–P(5)–P(6)	59·83 (3)
P(4)–P(6)	2·8660 (13)	P(4)–P(6)–P(5)	60·13 (3)
P(5)–P(6)	2·8722 (12)	P(5)–P(4)–P(6)	60·04 (3)

P(4)–O(*L*45)–P(5) 128·4 (2)
P(4)–O(*L*46)–P(6) 126·8 (1)
P(5)–O(*L*56)–P(6) 128·5 (2)

The isopropylammonium groups

N(1)–C(11)	1·472 (4)	N(1)–C(11)–C(12)	110·6 (2)
C(11)–C(12)	1·500 (6)	N(1)–C(11)–C(13)	109·7 (4)
C(11)–C(13)	1·499 (7)	C(12)–C(11)–C(13)	112·5 (4)
N(2)–C(21)	1·487 (5)	N(2)–C(21)–C(22)	107·0 (5)
C(21)–C(22)	1·477 (11)	N(2)–C(21)–C(23)	110·3 (3)
C(21)–C(23)	1·522 (8)	C(23)–C(21)–C(22)	105·5 (6)
N(3)–C(31)	1·477 (5)	N(3)–C(31)–C(32)	110·8 (3)
C(31)–C(32)	1·492 (6)	N(3)–C(31)–C(33)	110·5 (3)
C(31)–C(33)	1·478 (6)	C(32)–C(31)–C(33)	113·4 (4)
N(4)–C(41)	1·491 (5)	N(4)–C(41)–C(42)	108·7 (4)
C(41)–C(42)	1·505 (7)	N(4)–C(41)–C(43)	109·4 (4)
C(41)–C(43)	1·508 (6)	C(42)–C(41)–C(43)	114·8 (3)
N(5)–C(51)	1·483 (4)	N(5)–C(51)–C(52)	108·6 (3)
C(51)–C(52)	1·498 (8)	N(5)–C(51)–C(53)	109·4 (3)
C(51)–C(53)	1·505 (7)	C(52)–C(51)–C(53)	114·9 (5)
N(6)–C(61)	1·500 (5)	N(6)–C(61)–C(62)	108·6 (3)
C(61)–C(62)	1·514 (4)	N(6)–C(61)–C(63)	108·6 (3)
C(61)–C(63)	1·505 (6)	C(62)–C(61)–C(63)	112·2 (3)

Hydrogen bonds

N—H···O	N—H	H···O	N—O	N—H···O
N(1)–H(1N1)···O(*E*61)	1·03 (4)	1·72 (4)	2·726 (4)	167 (3)
N(1)–H(2N1)···O(*E*51)	1·01 (4)	2·16 (4)	3·038 (3)	145 (5)
N(1)–H(3N1)···O(*E*11)	0·85 (4)	1·93 (4)	2·775 (4)	175 (4)
N(2)–H(1N2)···O(*E*62)	0·89 (5)	1·94 (4)	2·753 (5)	151 (4)
N(2)–H(2N2)···O(*E*32)	0·97 (3)	1·99 (3)	2·843 (3)	146 (3)
N(2)–H(3N2)···O(*E*22)	0·91 (5)	2·12 (5)	2·975 (4)	156 (5)
N(3)–H(1N3)···O(*E*12)	1·05 (4)	1·71 (4)	2·739 (4)	168 (4)
N(3)–H(2N3)···O(*E*41)	0·96 (6)	1·89 (5)	2·847 (4)	175 (4)
N(3)–H(3N3)···O(*E*11)	0·97 (3)	1·99 (3)	2·859 (3)	147 (4)
N(4)–H(1N4)···O(*E*62)	0·96 (3)	1·94 (4)	2·826 (4)	153 (3)
N(4)–H(2N4)···O(*E*31)	0·85 (3)	1·94 (4)	2·739 (4)	156 (3)
N(4)–H(3N4)···O(*E*21)	1·08 (6)	1·66 (6)	2·690 (4)	157 (5)
N(5)–H(1N5)···O(*E*51)	0·95 (5)	1·86 (5)	2·804 (5)	168 (5)
N(5)–H(2N5)···O(*E*52)	0·82 (5)	1·97 (5)	2·776 (5)	169 (3)
N(5)–H(3N5)···O(*E*22)	0·81 (7)	2·07 (7)	2·865 (4)	170 (7)
N(6)–H(1N6)···O(*E*32)	1·03 (5)	1·96 (5)	2·972 (5)	170 (5)
N(6)–H(2N6)···O(*E*42)	0·99 (5)	1·78 (5)	2·745 (4)	162 (4)
N(6)–H(3N6)···O(*E*41)	0·91 (3)	1·95 (3)	2·852 (2)	172 (4)

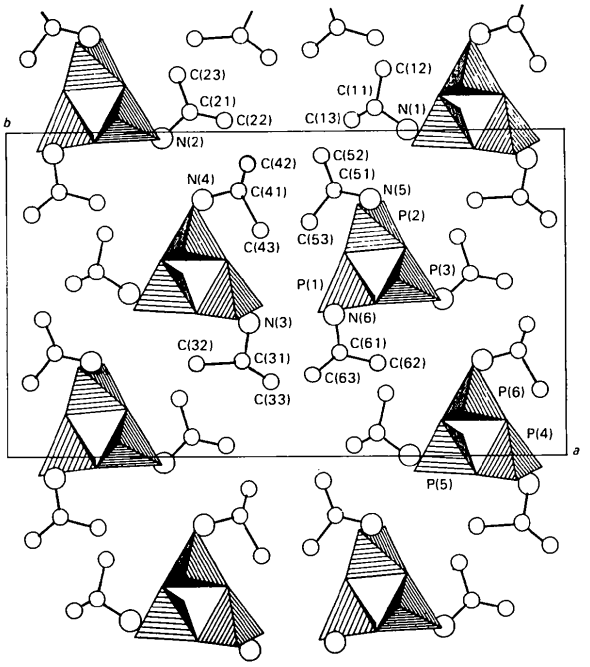

Fig. 1. Projection along the *c* axis of the atomic arrangement of isopropylammonium *cyclo*-triphosphate. As anionic groups and organic groups are almost superimposed in projection only one half of the arrangement ($0{\cdot}5 < z < 1{\cdot}0$) is reported. The PO_4 tetrahedra of the P_3O_9 ring anions are simply denoted by the name of the central phosphorus atom.

are observed in the atomic arrangement. Main geometrical features (interatomic distances and bond angles) are reported in Table 2.

The P_3O_9 ring anions are arranged in rows parallel to the *c* axis, these rows being themselves organized to build large hexagonal channels whose internal face is lined by the organic groups. Fig. 1 gives a projection of the atomic arrangement along the *c* axis restricted to $0{\cdot}5 < z < 1{\cdot}0$.

The stability of such an arrangement results from a net of strong hydrogen bonds (N–H···O) connecting the NH_3 radicals to the external oxygen atoms of the ring anions (Table 2).

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Structure of Potassium Ethylenediammonium *cyclo*-Triphosphate

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Abstract. $C_2H_{10}N_2^{2+}.K^+.P_3O_9^{3-}$, $M_r = 338{\cdot}13$, orthorhombic, *Ccca* (D_{2h}^{22}), $a = 20{\cdot}850\ (8)$, $b = 9{\cdot}044\ (4)$, $c = 11{\cdot}653\ (5)$ Å, $V = 2197\ (3)$ Å^3, $Z = 8$, $D_x = 2{\cdot}044$ Mg m^{-3}, $\lambda(\text{Ag } K\alpha) = 0{\cdot}5608$ Å, $\mu = 0{\cdot}496$ mm^{-1}, $F(000) = 1376$, $T = 295$ K, $R = 0{\cdot}038$ for 1081 independent reflexions. Layers of P_3O_9 ring anions ($z \sim 0{\cdot}25$ and $0{\cdot}75$) alternate with layers of ethylenediammonium groups and K atoms. The P_3O_9 anion has a twofold symmetry not previously observed in the *cyclo*-triphosphate crystal chemistry.

Introduction. The title compound is the first salt obtained and characterized during the investigation of systems of the type $H_3P_3O_9$–$NH_2(CH_2)_2NH_2$–M_2^IO–H_2O.

Experimental. The title compound was prepared by a metathesis reaction deriving from that described by Boullé (1938) for the preparation of purely inorganic water-soluble *cyclo*-triphosphates. An aqueous solution of ethylenediammonium chloride and potassium chloride is added to a slurry in water of the sparingly soluble silver *cyclo*-triphosphate monohydrate with the stoichiometric ratio 1 : 1 : 1. The reaction is

$$(NH_3CH_2)_2Cl_2 + KCl + Ag_3P_3O_9 \rightarrow K(NH_3CH_2)_2P_3O_9 + 3AgCl.$$

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