

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 ( $0.5 < z < 1.0$ ) is reported. The  $\text{PO}_4$  tetrahedra of the  $\text{P}_3\text{O}_9$  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  $\text{P}_3\text{O}_9$  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  $0.5 < z < 1.0$ .

The stability of such an arrangement results from a net of strong hydrogen bonds ( $\text{N}-\text{H}\cdots\text{O}$ ) connecting the  $\text{NH}_3$  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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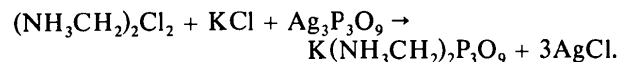
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**Abstract.**  $\text{C}_2\text{H}_{10}\text{N}_2^{2+}\cdot\text{K}^+\cdot\text{P}_3\text{O}_9^{3-}$ ,  $M_r = 338.13$ , orthorhombic,  $Ccca$  ( $D_{2h}^{22}$ ),  $a = 20.850$  (8),  $b = 9.044$  (4),  $c = 11.653$  (5) Å,  $V = 2197$  (3) Å<sup>3</sup>,  $Z = 8$ ,  $D_x = 2.044$  Mg m<sup>-3</sup>,  $\lambda(\text{Ag } K\alpha) = 0.5608$  Å,  $\mu = 0.496$  mm<sup>-1</sup>,  $F(000) = 1376$ ,  $T = 295$  K,  $R = 0.038$  for 1081 independent reflexions. Layers of  $\text{P}_3\text{O}_9$  ring anions ( $z \sim 0.25$  and  $0.75$ ) alternate with layers of ethylenediammonium groups and K atoms. The  $\text{P}_3\text{O}_9$  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  $\text{H}_3\text{P}_3\text{O}_9-\text{NH}_2(\text{CH}_2)_2\text{NH}_2-M_2\text{O}-\text{H}_2\text{O}$ .

**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



After about 1 h of mechanical stirring the insoluble silver chloride is eliminated by filtration. The resulting solution is then kept at room temperature and after some days of evaporation, colourless diamond-shaped thick plates of potassium ethylenediammonium *cyclo*-triphosphate appear. Prism fragment:  $0.19 \times 0.27 \times 0.24$  mm. Density not measured. Philips PW 1100 diffractometer, graphite monochromator. Systematic absences:  $hkl$ ,  $h + k = 2n$ ;  $Ok\bar{l}$ ,  $l = 2n$ ;  $hk0$ ,  $h = 2n$ . 20 reflexions ( $10 < \theta < 12^\circ$ ) for refining unit-cell dimensions.  $\omega$  scan, scan width  $1.20^\circ$ , scan speed  $0.03^\circ \text{ s}^{-1}$ , total background measuring time: 6 s, multiscan: 2. 1549 measured reflexions ( $3 < \theta < 30^\circ$ ),  $h$ ,  $k$ ,  $l$ ,  $h_{\max} = 33$ ,  $k_{\max} = 16$ ,  $l_{\max} = 18$ . Two intensity and orientation reference reflexions (004 and  $00\bar{4}$ ) every 2 h, no significant variation. Lorentz and polarization corrections, no absorption correction. Unit weights. Crystal structure solved by direct methods with *MULTAN77* (Main, Hull, Lessinger, Germain, De-

Table 2. Main interatomic bond distances ( $\text{\AA}$ ) and bond angles ( $^\circ$ ) with *e.s.d.*'s in parentheses

The $\text{P}_3\text{O}_9$ ring anion				
P(1) $\text{O}_4$ tetrahedron				
P(1)	O(L12)	O(E11)	O(E12)	O(L11)
O(L12)	1.605 (2)	2.527 (4)	2.496 (4)	2.488 (4)
O(E11)	109.5 (2)	1.489 (3)	2.545 (4)	2.509 (3)
O(E12)	108.6 (2)	118.8 (2)	1.467 (3)	2.498 (3)
O(L11)	101.6 (1)	108.3 (1)	108.7 (2)	1.606 (2)
P(2) $\text{O}_4$ tetrahedron				
P(2)	O(L12)	O(L12)	O(E2)	O(E2)
O(L12)	1.628 (2)	2.491 (3)	2.564 (4)	2.467 (3)
O(L12)	99.8 (1)	1.628 (2)	2.467 (3)	2.564 (4)
O(E2)	111.5 (1)	105.3 (1)	1.472 (3)	2.567 (4)
O(E2)	105.3 (1)	111.5 (1)	121.3 (2)	1.472 (3)
P(1)–P(1)	2.900 (1)	P(1)–O(L11)–P(1)	129.1 (2)	
2 $\times$ P(1)–P(2)	2.901 (1)	P(1)–O(L12)–P(2)	127.6 (2)	
		P(1)–P(2)–P(1)	59.97 (3)	
		P(1)–P(1)–P(2)	60.01 (3)	
$\text{K}\text{O}_8$ polyhedron				
2 $\times$ K–O(L12)	2.931 (3)	2 $\times$ K–O(E2)	2.726 (3)	
2 $\times$ K–O(E11)	2.894 (3)	2 $\times$ K–O(E2)	3.029 (3)	
$\text{NH}_3(\text{CH}_2)_2\text{NH}_3$ group				
C–C	1.502 (5)	N–C–C	110.4 (3)	
2 $\times$ C–N	1.482 (5)			

Table 1. Final atomic coordinates and  $B_{\text{eq}}$  values with *e.s.d.*'s in parentheses

$$B_{\text{eq}} = \frac{4}{3} \sum_i \sum_j a_i \cdot a_j \beta_{ij}$$

	x	y	z	$B_{\text{eq}} (\text{\AA}^2)$
K	$\frac{1}{2}$	0	–0.0082 (1)	1.97 (2)
P(1)	0.87599 (4)	0.4076 (1)	0.22701 (7)	1.40 (1)
P(2)	0.75547 (5)	$\frac{1}{2}$	$\frac{1}{2}$	1.31 (2)
O(L12)	0.8057 (1)	0.3823 (3)	0.2798 (2)	1.60 (4)
O(E11)	0.1291 (1)	0.4295 (3)	0.3993 (2)	1.94 (4)
O(E12)	0.4091 (1)	0.4803 (3)	0.2041 (2)	2.64 (5)
O(L11)	0.0909 (2)	$\frac{1}{2}$	$\frac{1}{2}$	2.35 (7)
O(E12)	0.7209 (1)	0.2790 (3)	0.1422 (2)	1.97 (5)
N	0.4103 (1)	0.2648 (4)	0.4332 (3)	1.74 (5)
C	0.4776 (2)	0.3150 (4)	0.4459 (4)	1.98 (6)

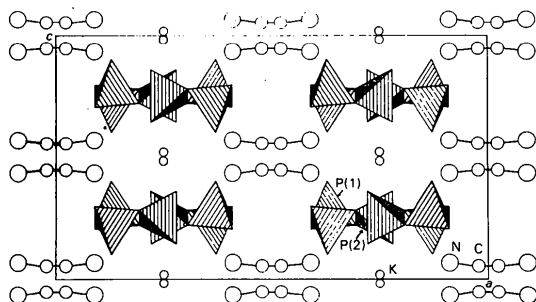


Fig. 1. Projection of the atomic arrangement along the  $b$  axis.

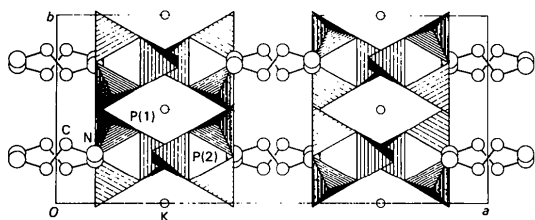


Fig. 2. Projection of the atomic arrangement along the  $c$  axis.

clercq & Woolfson, 1977). H atoms located by difference Fourier syntheses. Anisotropic full-matrix least-squares refinement (on  $F$ ) for non-H atoms, isotropic for H atoms. Final refinement with 1081 reflexions ( $I > 3\sigma_I$ ). Final  $R = 0.038$  ( $wR = 0.039$ ),  $S = 1.361$ . Max.  $\Delta/\sigma = 0.01$ . Max. peak height in the final difference Fourier synthesis:  $0.247 \text{ e \AA}^{-3}$ . Final  $R$  value = 0.058 for the complete set of 1549 independent reflexions. Scattering factors for neutral atoms and  $f'$ ,  $f''$  from *International Tables for X-ray Crystallography* (1974). Enraf–Nonius (1977) *SDP* employed for all calculations. Computer used: MicroVAX II.

**Discussion.** Table 1 reports the final atomic coordinates\* while Fig. 1 gives a projection of the atomic arrangement along the  $b$  axis. The atomic arrangements can be described as a succession of layers perpendicular to the  $c$  axis. All the  $\text{P}_3\text{O}_9$  ring anions are located in planes  $z \sim 0.25$  and  $0.75$ , while the ethylenediammonium groups and the K atoms alternate in  $z \sim 0.0$  and  $0.50$ . Fig. 2, a projection along the  $c$  axis, shows the distribution inside these two kinds of

\* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51210 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

layers. The  $P_3O_9$  ring anion has a twofold symmetry through the P(2) atom and the O(L11) bonding oxygen atom. This kind of internal symmetry has up to now never been observed for this type of anion. The K atom located halfway between  $P_3O_9$  layers (Fig. 1) has an eightfold coordination made by six external O atoms of the ring anion and two bonding O atoms of this ring. The participation of bonding O atoms of a phosphoric ring to a metal coordination is rather rare in condensed phosphate chemistry.

Table 2 reports the main interatomic distances and bond angles in the various entities of the arrangement and gives the numerical data corresponding to the three-dimensional network of hydrogen bonds connect-

ing the  $NH_3$  radicals to the external oxygen atoms of the  $P_3O_9$  ring anions.

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### Structure of a Permethyl Zirconocene Ylide Complex:



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**Abstract.** (Dimethylphosphinodimethylidene-*C,C'*-hydridobis(pentamethylcyclopentadienyl)zirconium,  $C_{24}H_{41}PZr$ ,  $M_r = 451.78$ , monoclinic,  $P2_1/c$ ,  $a = 13.7765$  (14),  $b = 10.9262$  (12),  $c = 15.9459$  (19) Å,  $\beta = 93.358$  (9)°,  $V = 2396.1$  (8) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.25$  g cm<sup>-3</sup>, Mo  $K\alpha$  radiation,  $\lambda = 0.7107$  Å,  $\mu = 5.2$  cm<sup>-1</sup>,  $T \approx 290$  K,  $F(000) = 960$ ;  $S$ (goodness-of-fit) = 2.20 (5540 reflections),  $R = 0.077$  (4990 reflections,  $I > 0$ ),  $R = 0.052$  (3456 reflections,  $I > 3\sigma$ ). Important structural features: Zr–C(1), 2.486 (5), Zr–C(2), 2.467 (6), Zr–H, 1.88 (4) Å, C(1)–Zr–C(2), 65.9 (2)°.

**Introduction.** Phosphorus ylides are used commonly as methylene transfer reagents in organic synthesis. Recently, phosphoranes have found application as reagents in the synthesis of organometallic compounds. Permethylzirconocene dihydride,  $Cp_2^*ZrH_2$  (Me  $\equiv$  CH<sub>3</sub>,  $Cp^* \equiv$  C<sub>5</sub>Me<sub>5</sub>; Wolczanski, Threlkel & Bercau, 1979), reacts with a threefold excess of CH<sub>2</sub>PMe<sub>3</sub> at room temperature to generate two permethylzirconocene complexes:  $Cp_2^*Zr(H)Me$ , 71%,

and  $Cp_2^*Zr(H)CH_2PMe_2CH_2$ , 29%; the latter can also be cleanly generated by the reaction of two equivalents of CH<sub>2</sub>PMe<sub>3</sub> with  $Cp_2^*Zr(H)Cl$  (Hillhouse & Bercau, 1984) at room temperature for 12 h (Moore, 1984). We report herein on the structure of  $Cp_2^*Zr(H)CH_2PMe_2CH_2$ .

**Experimental.** Orange crystals from toluene, air-sensitive, sealed in glass capillary under N<sub>2</sub>. Crystal size 0.20 × 0.30 × 0.40 mm. Space group  $P2_1/c$  by oscillation and Weissenberg photographs (systematic absences:  $h0l$  for  $l$  odd,  $0k0$  for  $k$  odd). Diffractometry: Syntex  $P2_1$ , graphite monochromator, Mo  $K\alpha$  radiation. Lattice constants by least-squares refinement of 15 average  $2\theta$  values (four settings for each reflection:  $\pm\theta$ ,  $\pm\omega$ ,  $\varphi$ ,  $\chi$  and  $\pm\theta$ ,  $\pm\omega$ ,  $\varphi+\pi$ ,  $-\chi$ ;  $25 \leq 2\theta \leq 40^\circ$ ). Intensity measurements: 12020 reflections ( $+h\pm k\pm l$ ),  $2\theta \leq 55^\circ$ ,  $\theta$ – $2\theta$  scans at  $2.02^\circ$  min<sup>-1</sup> with  $2\theta$  width of  $2.0^\circ$  plus angular dispersion; scan-time-to-background-time ratio  $r = t_s/t_b = 2.0$ ; stationary-counter-stationary-crystal background counts fore and aft. Four check reflections every 96 reflections; no decomposition. No absorption correction, averaging over  $2/m$  symmetry, preliminary scaling by Wilson (1942) plot. Structure derivation: Patterson and electron density maps. Refinement: full-matrix least squares on  $F^2$ , all reflections, weights  $w$ ,  $w^{-1} = [s + r^2b + (0.02s)^2]k^4 / (Lp)^2$  ( $s$  = scan counts,  $b$  = total background counts,

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