structure fall below these limits, although the Br(2)···H—O(2′) separation [3·42 (1) Å] is just above, and probably corresponds to some attraction. The short N(7)···O(3′) distance [2·67 (2) Å] is consistent with the presence of the extra proton at N(7) in the inosinium cation.

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References

AUTHIER-MARTIN, M. & BEAUCHAMP, A. L. (1977). *Can. J. Chem.* **55**, 1213–1217.

BÉLANGER-GARIÉPY, F. & BEAUCHAMP, A. L. (1980). *J. Am. Chem. Soc.* **102**, 3461–3464.

BÉLANGER-GARIÉPY, F. & BEAUCHAMP, A. L. (1981). *Cryst. Struct. Commun.* **10**, 1165–1171.

BÉLANGER-GARIÉPY, F. & BEAUCHAMP, A. L. (1982). *Cryst. Struct. Commun.* **11**, 991–998.

CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

JOHNSON, J. P., KRAUSE, H. B. & SHERRY, E. G. (1981). *Acta Cryst.* A**37**, C227.

MUNNS, A. R. I. & TOLLIN, P. (1970). *Acta Cryst.* B**26**, 1101–1113.

SADLER, P. J. (1976). *Struct. Bonding Berlin*, **29**, 171–211.

SADLER, P. J., NASR, M. & NARAYANAN, V. L. (1984). *Platinum Coordination Complexes in Cancer Chemotherapy*, pp. 290–304. Boston: Martinus Nijhoff Publishers.

SALAS, J. M., MORENO, M. N., LOPEZ, J. D. & ROMERO, M. A. (1986). *Spectrochim. Acta Part A*, **42**, 607–610.

SIMON, T. M., KUNISHIMA, D. H., VIBERT, G. J. & LORBER, A. (1981). *Cancer Res.* **41**, 94–97.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

STOUT, G. H. & JENSEN, L. H. (1968). *X-ray Structure Determination, A Practical Guide*. London: Macmillan.

STRÄHLE, J., GELINEK, J. & KÖLMEL, M. (1979). *Z. Anorg. Allg. Chem.* **456**, 241–260.

SUBRAMANIAN, E. (1979). *Cryst. Struct. Commun.* **8**, 777–785.

SUNDARALINGAM, M. (1969). *Biopolymers*, **7**, 821–860.

SUTTON, B. M. (1983). *Platinum, Gold and other Chemotherapeutic Agents. ACS Symp. Ser.* **209**, 355–369.

SWAMINATHAN, V. & SUNDARALINGAM, M. (1979). *CRC Crit. Rev. Biochem.* **6**, 245–336.

TAYLOR, R. & KENNARD, O. (1982). *J. Mol. Struct.* **78**, 1–28.

THEWALT, U., BUGG, C. E. & MARSH, R. E. (1970). *Acta Cryst.* B**26**, 1089–1101.

TOUGARD, P. & CHANTOT, J. F. (1974). *Acta Cryst.* B**30**, 214–220.

Acta Cryst. (1989). C**45**, 1877–1879

Structure of Ethylenediammonium Disodium *cyclo*-Tetraphosphate Dihydrate: $Na_2[(CH_2)_2(NH_3)_2]P_4O_{12}.2H_2O$

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Abstract. $C_2H_{10}N_2^{2+}.2Na^+.P_4O_{12}^{4-}.2H_2O$, M_r = 423·98, monoclinic, $C2/c$, $a = 7{\cdot}887\ (2)$, $b = 14{\cdot}869\ (3)$, $c = 13{\cdot}070\ (3)$ Å, $\beta = 91{\cdot}46\ (1)°$, $V = 1532\ (1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}838$, $D_m = 1{\cdot}896$ Mg m^{-3}, λ(Ag $K\alpha$) = 0·5608 Å, $\mu = 0{\cdot}305$ mm^{-1}, $F(000) = 856$, $T = 298$ K, final $R = 0{\cdot}042$ for 2262 unique reflexions. P_4O_{12} rings twist around 2_1 axes to form a channel with section (3 × 7 Å) perpendicular to the c axis. The two crystallographically independent sodium cations Na(1) and Na(2) are located on the channel axis. $Na(1)O_6$ and $Na(2)O_6$ polyhedra link together through the O(E12)—O(E21)—O(W) faces in linear arrays parallel to the c axis. The organic groups and P_4O_{12} rings both have twofold symmetry. Hydrogen atoms of the NH_3 groups act on external oxygen atoms of the $P_4O_{12}^{4-}$ anions to build a three-dimensional network. The potassium salt is isotypic with the title compound.

Introduction. In the continuation of our studies on the *cyclo*-tetraphosphoric compounds (Jouini, Dabbabi & Durif, 1985; Jouini, Soua & Dabbabi, 1986) we are investigating the interaction between amines and tetraphosphoric acid. The present work deals with the crystal structure of disodium ethylenediammonium *cyclo*-tetraphosphate dihydrate: $2Na^+.C_2H_{10}N_2^{2+}.P_4O_{12}^{4-}.2H_2O$. The synthesis process used is the same as that employed for the preparation of inorganic phosphates (Jouini & Durif, 1983; Jouini & Dabbabi, 1985). The title compound is the first salt obtained in the $H_4P_4O_{12}$–M^I_2O–$C_2H_8N_2$ system. Its detailed preparation and main crystallographic features have been described (Bdiri & Jouini, 1989).

Experimental. Colourless and prismatic crystal 0·25 × 0·27 × 0·30 mm, density measured by flotation,

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Table 1. *Final atomic coordinates and* B_{eq} *for* $Na_2[(CH_2)_2(NH_3)_2]P_4O_{12}.2H_2O$, *with e.s.d.'s in parentheses*

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

	x	*y*	*z*	B_{eq} (Å²)
P(1)	0·29499 (8)	0·10914 (5)	0·34472 (5)	1·199 (8)
P(2)	0·33810 (8)	0·07788 (5)	0·12396 (5)	1·233 (8)
Na(1)	0·5000	0·4508 (1)	0·2500	2·01 (3)
Na(2)	0·0000	0·0000	0·0000	2·64 (3)
O(*L*12)	0·5358 (2)	0·0497 (1)	0·1341 (1)	1·39 (3)
O(*E*11)	0·6941 (3)	0·1957 (1)	0·0981 (2)	1·93 (3)
O(*L*21)	0·6888 (2)	0·1361 (2)	0·2745 (1)	1·40 (3)
O(*E*12)	0·1494 (2)	0·0480 (1)	0·3589 (1)	1·93 (3)
O(*E*21)	0·2417 (2)	−0·0083 (1)	0·1279 (2)	1·87 (3)
O(*E*22)	0·3097 (3)	0·1399 (2)	0·0358 (1)	2·20 (4)
O(*W*)	0·5376 (3)	0·3470 (2)	0·3926 (2)	3·07 (5)
N	0·0590 (3)	0·3321 (2)	0·3908 (2)	1·80 (4)
C	0·0522 (3)	0·3265 (2)	0·2022 (2)	1·95 (4)

Philips PW1100 diffractometer, graphite-monochromatized Ag *Kα* radiation, 25 reflexions with θ in the range $9 < \theta < 12°$ used for determining lattice constants and orientation matrix, 2665 reflexions measured ($3 < \theta < 30°$), $\pm hkl$, $h_{max} = \pm 14$, $k_{max} = 26$, $l_{max} = 23$, ω scan, scan width $(1{\cdot}2 + 0{\cdot}2\tan\theta)°$, scan speed $0{\cdot}03°\,s^{-1}$, total background measuring time 10 s, intensities of three reference reflexions (045, 0$\bar{4}$5, 04$\bar{5}$) measured every 2 h: no significant variation. Lorentz and polarization corrections, no absorption correction. The systematic absences (*hkl* absent if $h + k = 2n + 1$, *h*0*l* absent if $l = 2n + 1$) allow the space group to be *C*2/*c* or *Cc*. The choice of *C*2/*c* was confirmed by the *E* statistics and the successful solution of the structure and refinement. Structure solved by classical methods (Patterson and successive Fourier syntheses). Hydrogen atoms from difference-Fourier map. Anisotropic full-matrix least-squares refinement (on *F*), isotropic for hydrogen atoms. Unit weights. Final refinement cycles with 2262 reflexions corresponding to $F > 3\sigma_F$. Final $R = 0{\cdot}042$ ($wR = 0{\cdot}044$). $S = 0{\cdot}947$. Max. $\Delta/\sigma = 0{\cdot}00$, max. peak height in the final difference Fourier synthesis: $0{\cdot}254\ e\ Å^{-3}$. No extinction correction. Scattering factors for neutral atoms and *f′*, *f″* from *International Tables for X-ray Crystallography* (1974). Enraf–Nonius (1980) *SDP* employed for all calculations. Computer used: PDP 11/34.

Discussion. Final atomic coordinates are in Table 1.* Fig. 1 represents the projection of the atomic arrangement along the *b* axis. Sodium polyhedra, organic groups and detailed hydrogen bonds are depicted in Fig. 2. Main interatomic distances, bond angles in the $P_4O_{12}^{4-}$ anion and hydrogen bonds are reported in Table 2.

Distances and bond angles in the P_4O_{12} ring are not significantly different from what is commonly observed in inorganic *cyclo*-tetraphosphates. The

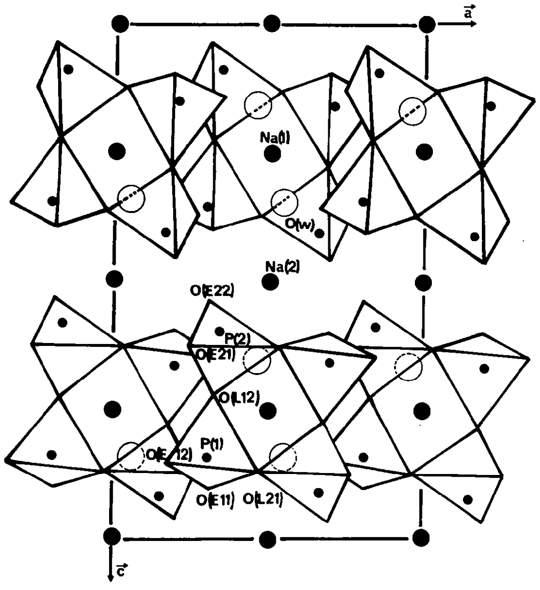

Fig. 1. Projection along the *b* axis of the atomic arrangement of $Na_2[(CH_2)_2(NH_3)_2]P_4O_{12}.2H_2O$. Ethylenediammonium groups are omitted.

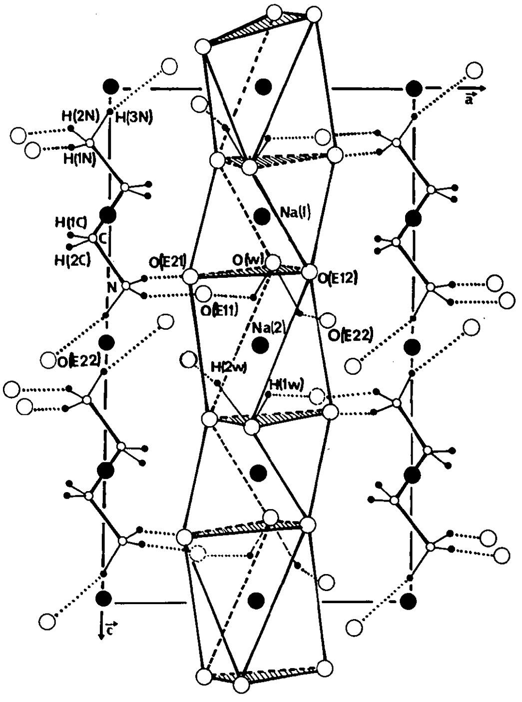

Fig. 2. Projection along the *b* axis of the linear array of NaO_6 polyhedra and ethylenediammonium groups, with hydrogen-bond scheme, in $Na_2[(CH_2)_2(NH_3)_2]P_4O_{12}.2H_2O$.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and main interatomic distances and bond angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52075 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Main interatomic distances* (Å), *bond angles* (°) *and hydrogen-bond scheme* (Å,°) *in* $Na_2[(CH_2)_2(NH_3)_2]P_4O_{12}.2H_2O$

$P(1)O_4$ tetrahedron

P(1)	O(*E*11)	O(*E*12)	O(*L*12)	O(*L*21)
O(*E*11)	1·490 (2)	2·575 (3)	2·555 (3)	2.472 (3)
O(*E*12)	120·2 (1)	1·480 (2)	2·482 (3)	2·549 (3)
O(*L*12)	110·4 (1)	106·4 (1)	1·619 (2)	2·515 (3)
O(*L*21)	105·3 (1)	110·7 (1)	102·4 (1)	1·617 (2)

$P(2)O_4$ tetrahedron

P(2)	O(*E*21)	O(*E*22)	O(*L*21)	O(*L*12)
O(*E*21)	1·492 (2)	2·574 (3)	2·550 (2)	2·474 (3)
O(*E*22)	119·5 (1)	1·488 (2)	2·480 (3)	2·552 (3)
O(*L*21)	110·9 (1)	106·6 (1)	1·603 (2)	2·515 (3)
O(*L*12)	105·4 (1)	110·5 (1)	102·7 (1)	1·617 (2)

2 × P(1)—P(2)	2·9503 (9)	P(1)—P(2)—P(1)	88·66 (3)
2 × P(2)—P(1)	2·9489 (9)	P(2)—P(1)—P(2)	88·50 (3)
		P(1)—O(*L*12)—P(2)	131·4 (1)
		P(2)—O(*L*21)—P(1)	132·8 (1)

$Na(1)O_6$ polyhedron		$Na(2)O_6$ polyhedron	
2 × Na(1)—O(*E*12)	2·327 (2)	2 × Na(2)—O(*E*12)	2·326 (2)
2 × Na(1)—O(*E*21)	2·589 (2)	2 × Na(2)—O(*E*21)	2·506 (2)
2 × Na(1)—O(*W*)	2·432 (3)	2 × Na(2)—O(*W*)	2·694 (3)

NH_3—$(CH_2)_2$—NH_3 group

N—C	1·484 (4)	C—C	1·513 (6)	N—C—C	110·7 (3)

Hydrogen bonds

	H—H		N—O	N—H···O
	O—H	H···O	O—O	O—H···O
N—H(1N)···O(*R*21)	0·92 (5)	1·94 (4)	2·860 (4)	178 (4)
N—H(2N)···O(*E*11)	0·81 (5)	2·02 (5)	2·281 (3)	169 (4)
N—H(3N)···O(*E*22)	0·85 (4)	1·98 (4)	2·797 (3)	159 (3)
O(*W*)—H(1*w*)···O(*E*11)	0·70 (5)	2·25 (5)	2·902 (4)	167 (5)
O(*W*)—H(2*w*)···O(*E*22)	0·93 (5)	1·91 (5)	2·818 (4)	164 (5)

P_4O_{12} groups are located around the twofold axes. They twist around the 2_1 axes, situated at $x = \frac{1}{2}$; $z = \frac{1}{4},\frac{3}{4}$, to form a channel with section (3 × 7 Å) perpendicular to the *c* axis.

The two crystallographically independent sodium atoms Na(1) and Na(2) both have sixfold coordination, formed by two water molecules and four external oxygen atoms belonging to four ring anions $P_4O_{12}^{4-}$. The $Na(1)O_6$ and $Na(2)O_6$ distorted octahedra link together through the O(*E*12)—O(*E*21)—O(*W*) faces to form an infinite one-dimensional chain parallel to the *c* axis, and are located at the centre of the channel (Fig. 2).

The organic group, present as the dication $(CH_2)_2(NH_3)_2^{2+}$, has twofold symmetry. The groups alternate with P_4O_{12} rings and NaO_6 polyhedra to build, by hydrogen bonds, a three-dimensional network. It should be noticed that only the external oxygen atoms of the ring anions and sodium polyhedra are involved in hydrogen bonds; the oxygen atoms O(*E*11) and O(*E*22) taking part in this scheme act twice as acceptors. The potassium salt is isotypic with the following non-refined unit-cell dimensions: *a* = 7·92, *b* = 14·89, *c* = 13·11 Å, *β* = 92·03°.

References

Bdiri, M. & Jouini, A. (1989). *C.R. Acad. Sci.* **308**, 1353–1358.

Enraf-Nonius (1980). *Structure Determination Package*. Enraf-Nonius, Delft, The Netherlands.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Jouini, A. & Dabbabi, M. (1985). *C. R. Acad. Sci.* **301**, 1347–1350.

Jouini, A., Dabbabi, M. & Durif, A. (1985). *J. Solid State Chem.* **60**, 6–12.

Jouini, A. & Durif, A. (1983). *C. R. Acad. Sci.* **297**, 573–575.

Jouini, A., Soua, M. & Dabbabi, M. (1986). *J. Solid State Chem.* **69**, 135–144.

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Structure of [*o*-Bis(diisopropylphosphino)benzene]tetracarbonyltungsten(0)

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Abstract. $[W(CO)_4(C_{18}H_{32}P_2)]$, M_r = 606·30, monoclinic, $P2_1/c$, *a* = 17·599 (4), *b* = 14·672 (2), *c* = 19·844 (3) Å, *β* = 102·25 (2)°, *V* = 4996 (2) Å^3, *Z* = 8, D_m = 1·60 (2), D_x = 1·61 Mg m^{-3}, λ(Mo *Kα*) = 0·7107 Å, *μ* = 4·87 mm^{-1}, *F*(000) = 1152, *T* = 300 K, *R* = 0·034 for 6268 observed reflections. The structure of the title compound shows that the two phosphorus donors coordinate to the metal centre with *cis* geometry. The average W—P distance is 2·510 (2) Å. All the methyl groups point toward the $W(CO)_4$ moiety in order to minimize steric interaction.

Introduction. Unlike the bidentate behavior of bis(diphenylphosphino)ethane (dppe), the *o*-phenylenebisphosphines have the interesting feature of

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