occupation maximale de son site. Par consequent des rapports $As_{oct}/Al$ encore plus faibles sont envisageables si le sodium pouvait être partiellement remplacé par un cation bivalent. On aurait alors dans ce nouveau diagramme d'ordre cinq, une série de solutions solides dont le présent composé serait l'une des limites. Des essais dans ce sens sont en cours.

L'arsenic révèle dans cette structure un comportement nouveau en adoptant la coordinence 6 en même temps qu'un autre élément (Al). En effet la comparaison du présent hétéropolyanion avec ceux connus renfermant l'arsenic(V) en présence d'un élément de coordinence 6: $Sb^V$, $Nb^V$, $V^V$ ou $Al^{III}$ dans les structures de $Na_3Sb_3As_2O_{14}$ (Haddad, Jouini & Ghedira, 1988), $K_2Nb_2As_2O_{11}$ (Zid, Jouini, Jouini & Omezzine, 1988), $(NH_4)_4H_6(As_6V_4O_{30})$ (Durif & Averbuch-Pouchot, 1969) et $NaAlAs_2O_7$ (Driss & Jouini, 1989*d*) montre que dans tous ces cas l'arsenic exerce exclusivement la coordinence 4 en laissant tous les sites octaédriques à l'hétéroélément.

Dans les structures des pyroarséniates et pyrophosphates de sodium $NaAlAs_2O_7$ (Driss & Jouini, 1989*d*) et $NaAlP_2O_7$ (Gamondes, d'Yvoire & Boullé, 1971) l'organisation des groupements $P_2O_7$ et des octaèdres $AlO_6$ diffère de celle dans la présente structure. En effet ces deux derniers sont formés, bien que n'étant pas isotypes entre eux, par l'alternance de couches contenant respectivement les groupements pyro et les octaèdres $AlO_6$, laissant libre des cages allongées pour loger les ions sodium.

La substitution partielle d'un élément par l'arsenic n'a été réalisée jusqu'ici, à notre connaissance que pour le phosphore (Thilo & Schulz, 1951). Signalons que des tentatives de substitution arsenic–phosphore dans le composé du titre ont réussi, comme le prouve le décalage des raies des clichés de poudre obtenus, conduisant à la solution solide $Na(Al_{1,5}As_{0,5})(As_{2-x}P_xO_7)_2$. Une étude complète est en cours.

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# Structure of Bis(hexamethylenetetramine)diiodozinc(II)*

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**Abstract.** $[ZnI_2(C_6H_{12}N_4)_2]$, $M_r = 599{\cdot}57$, orthorhombic, $P2_12_12$, $a = 18{\cdot}267\,(1)$, $b = 6{\cdot}978\,(3)$, $c = 7{\cdot}186\,(1)$ Å, $V = 916{\cdot}0\,(3)$ Å$^3$, $Z = 2$, $D_m = 2{\cdot}13$, $D_x = 2{\cdot}17$ Mg m$^{-3}$, $\lambda$(Mo $K\alpha$) $= 0{\cdot}71069$ Å, $\mu = 4{\cdot}464$ mm$^{-1}$, $F(000) = 576$, room temperature, final $R = 0{\cdot}0310$, $wR = 0{\cdot}0307$ for 2046 reflections with $I \geq 2\sigma(I)$ and 153 variables. The compound consists of monomeric molecules. The Zn atom lies on a crystallographic twofold axis and is approximately tetrahedrally coordinated to two I atoms and, *via* N atoms, to two molecules of hexamethylenetetramine.

* Hexamethylenetetramine is 1,3,5,7-tetraazatricyclo[3.3.1.1$^{3,7}$]-decane.

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**Introduction.** The present work constitutes part of our systematic investigation of the properties of polycyclic tertiary amines, *e.g.* quinuclidine, 1,4-diazabicyclo-[2.2.2]octane, and hexamethylenetetramine (HMTA), as ligands in metal complexes.

Since the discovery of HMTA well over a century ago (Butlerow, 1859), an enormous number of its salts, molecular adducts and coordination complexes have been reported in the literature [for a summary see Altpeter (1931, and references cited therein)], but many have not been investigated in detail, and relatively few have been fully characterized by X-ray crystallography.

HMTA can act in different ways towards metal salts: it can form addition compounds without coordination to metal centers, *e.g.* in $NdCl_3.2C_6H_{12}N_4.13H_2O$ (Sommer, 1986) or $[Co(H_2O)_6](NO_3)_2.2C_6H_{12}N_4.4H_2O$ (Viossat, Khodadad & Rodier, 1981), it may form protonated species, as in $[C_6H_{13}N_4][HgCl_3]$ (Pickardt & Schendler, 1982), it may coordinate to one metal center, *e.g.* in $[Cu_4Cl_6O(C_6H_{12}N_4)_4]$ (Pickardt & Rautenberg, 1982) or $AgNO_3.C_6H_{12}N_4$ (Michelet, Viossat, Khodadad & Rodier, 1981), it may form bridges between two metal centers, *e.g.* in $2CdCl_2.C_6H_{12}N_4.5H_2O$ (Lai & Mak, 1983), or even occur in different coordination modes, *e.g.* in $3CdI_2.2C_6H_{12}N_4.4H_2O$ (Pickardt, 1981).

In order to investigate the influence of additional ligands on the coordination mode of HMTA we have determined the crystal structures of addition compounds of HMTA with zinc halides $ZnX_2$ with $X = Cl$ and Br (Pickardt & Droas, 1985), and $X = I$ (this work).

**Experimental.** The title compound was prepared by diffusion of a solution of HMTA in water/methanol into an aqueous solution of $ZnI_2$. Elemental analysis confirmed the identity of the complex {found: Zn 11·89, C 23·89, N 18·61, H 3·90%; calculated for $[ZnI_2(C_6H_{12}N_4)_2]$: Zn 10·90, C 24·04, N 18·69, H 4·04%}. A colorless crystal of approximate dimensions 0·25 × 0·5 × 0·8 mm was used. $D_m$ was determined by flotation in diiodomethane/acetone. Nonius CAD-4 diffractometer, graphite-crystal-monochromated Mo $K\alpha$ radiation, unit-cell parameters determined from the angular settings of 25 reflections with $7{\cdot}8 \le \theta \le 21{\cdot}9°$. The space group was inferred as $P2_12_12$ (No. 18) from systematic absences ($h00$ with $h = 2n + 1$; $0k0$ with $k = 2n + 1$) and from the structure determination. The intensity data of 3212 unique reflections up to $\theta = 40°$ in the range $0 \le h \le 32$, $0 \le k \le 12$, $0 \le l \le 12$ were measured, using the $\omega$–$2\theta$-scan technique with a scan angle of 1·00° and a variable scan rate with a max. scan time of 60 s per reflection; two reflections for orientation control monitored every 200 reflections; three intensity-control reflections every hour of data collection showed no significant variation during the period of measurement. An empirical absorption correction, using $\psi$ scans (North, Phillips & Mathews, 1968) (correction factors in the range 0·84 to 1·00), as well as Lorentz and polarization corrections (Frenz, 1981) were applied. 2046 unique observed $[I \ge 2\sigma(I)]$ reflections. Structure solved by Patterson method with *SHELXS*86 (Sheldrick, 1986). Isotropic least-squares refinement on $F$ with *SHELX*76 (Sheldrick, 1976) converged at $R = 0{\cdot}125$. At this stage an additional empirical absorption correction was applied (Walker & Stuart, 1983) leading to a further reduction of $R$ to 0·099 (relative correction factors were in the range 0·752 to 1·053 with an average of 0·944).

H atoms were introduced in calculated positions. During the final stages of refinement all non-H atoms were refined anisotropically, H atoms were refined with isotropic temperature factors, the final agreement factors were $R = 0{\cdot}0310$ and $wR = 0{\cdot}0307$, $w = 1{\cdot}418/[\sigma^2(F) + 0{\cdot}0002F^2]$, $S = 0{\cdot}78$, $(\Delta/\sigma)_{max} = 0{\cdot}1$ for the positional parameters of the non-H atoms, max. and min. residual electron densities 1·18 and $-1{\cdot}16$ e Å$^{-3}$ near the I atoms. A refinement of the inverse structure gave $R = 0{\cdot}0365$. This was rejected according to Hamilton's (1965) test. Scattering factors used for $Zn^{2+}$, $I^-$ ions and neutral C and N atoms were taken from Cromer & Mann (1968), for H atoms from Stewart, Davidson & Simpson (1965); anomalous-dispersion correction factors from Cromer & Liberman (1970).

**Discussion.** Final positional and equivalent isotropic thermal parameters are given in Table 1,* bond distances and angles in Table 2. Fig. 1 shows an *ORTEP* (Johnson, 1976) drawing of the molecule, Fig. 2 a packing diagram, drawn with *PLUTO* (Motherwell & Clegg, 1978). The complex is monomeric in the crystalline state and consists of a Zn atom coordinated to two I atoms and, *via* N atoms, to two HMTA molecules in a distorted tetrahedral arrangement. In contrast, the zinc halide–HMTA adducts $[ZnX_2(C_6H_{12}N_4)]$, with $X = Cl$ and Br, prepared under similar conditions (Pickardt & Droas, 1985), contain only one HMTA molecule per Zn atom and in the crystal form infinite chains *via* HMTA molecules.

The Zn atom occupies a special position [2(*a*)] on a twofold axis. The point symmetry of the complex is thus $C_2$. The Zn—I distance is 2·543 (1) Å. We may compare this with Zn—I distances of 2·543 (3) and 2·562 (2) Å in [2,2-dimethyl-1,3-bis(methylamino)propane]diiodozinc (Richard, Boulanger, Guedon,

* 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 51497 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

$$B_{eq} = \frac{8}{3}\pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j .$$

| | $x$ | $y$ | $z$ | $B_{eq}$(Å$^2$) |
|---|---|---|---|---|
| Zn | 0 | 0 | 0·00414 (9) | 2·21 (2) |
| I | 0·05916 (1) | 0·25844 (4) | 0·20317 (4) | 3·00 (1) |
| N(1) | 0·0839 (2) | −0·1213 (4) | −0·1565 (3) | 2·21 (4) |
| N(2) | 0·2059 (2) | −0·2661 (4) | −0·1176 (4) | 3·29 (4) |
| N(3) | 0·1803 (2) | −0·0710 (4) | −0·3947 (4) | 3·99 (4) |
| N(4) | 0·1227 (2) | −0·3771 (4) | −0·3658 (4) | 4·31 (4) |
| C(1) | 0·1431 (2) | −0·1837 (4) | −0·0208 (4) | 2·81 (4) |
| C(2) | 0·1183 (2) | 0·0116 (4) | −0·2971 (4) | 3·50 (4) |
| C(3) | 0·0609 (2) | −0·2918 (4) | −0·2647 (4) | 3·95 (4) |
| C(4) | 0·1542 (2) | −0·2333 (5) | −0·4955 (4) | 5·14 (4) |
| C(5) | 0·1787 (3) | −0·4281 (4) | −0·2243 (5) | 4·52 (4) |
| C(6) | 0·2354 (2) | −0·1319 (5) | −0·2545 (5) | 4·15 (4) |

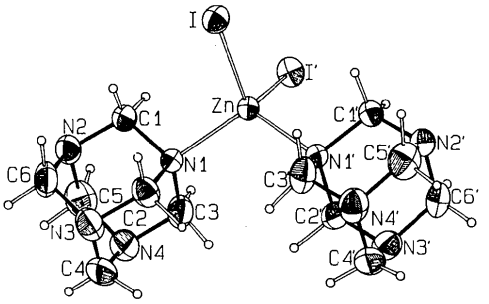


Fig. 1. An *ORTEP* drawing of the title compound indicating the atom-numbering scheme. Thermal ellipsoids are drawn at the 50% probability level (for clarity the H atoms are not labeled).

Table 2. *Selected bond lengths* (Å) *and angles* (°)

| | | | |
|---|---|---|---|
| Zn—I | 2·543 (1) | N(2)—C(6) | 1·462 (4) |
| Zn—N(1) | 2·097 (3) | N(3)—C(2) | 1·451 (5) |
| N(1)—C(1) | 1·521 (4) | N(3)—C(4) | 1·426 (5) |
| N(1)—C(2) | 1·509 (4) | N(3)—C(6) | 1·487 (5) |
| N(1)—C(3) | 1·482 (4) | N(4)—C(3) | 1·469 (5) |
| N(2)—C(1) | 1·459 (4) | N(4)—C(4) | 1·486 (5) |
| N(2)—C(5) | 1·453 (5) | N(4)—C(5) | 1·487 (5) |
| I—Zn—I$^{i}$ | 111·5 (1) | C(2)—N(3)—C(4) | 107·5 (3) |
| I—Zn—N(1) | 106·6 (1) | C(2)—N(3)—C(6) | 108·3 (3) |
| I$^{i}$—Zn—N(1) | 109·5 (1) | C(4)—N(3)—C(6) | 110·1 (3) |
| N(1)—Zn—N(1$^{i}$) | 113·2 (2) | C(3)—N(4)—C(4) | 109·5 (3) |
| Zn—N(1)—C(1) | 106·4 (2) | C(3)—N(4)—C(5) | 106·7 (3) |
| Zn—N(1)—C(2) | 115·2 (2) | C(4)—N(4)—C(5) | 108·9 (3) |
| Zn—N(1)—C(3) | 113·9 (2) | N(1)—C(1)—N(2) | 111·5 (2) |
| C(1)—N(1)—C(2) | 108·0 (3) | N(1)—C(2)—N(3) | 113·9 (3) |
| C(1)—N(1)—C(3) | 108·0 (2) | N(1)—C(3)—N(4) | 111·5 (3) |
| C(2)—N(1)—C(3) | 105·1 (2) | N(3)—C(4)—N(4) | 110·3 (3) |
| C(1)—N(2)—C(5) | 106·9 (3) | N(2)—C(5)—N(4) | 114·2 (3) |
| C(1)—N(2)—C(6) | 111·0 (3) | N(2)—C(6)—N(3) | 112·9 (3) |
| C(5)—N(2)—C(6) | 105·6 (3) | | |

Symmetry code: (i) $-x, -y, z$.

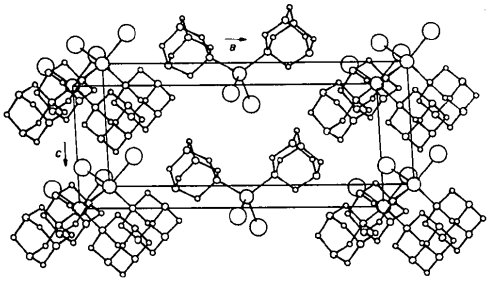


Fig. 2. A perspective *PLUTO* drawing of the contents of one unit cell (H atoms omitted).

Kasowski & Bordeleau, 1977), 2·557 (5) and 2·573 (6) Å in diiodobis(2-isopropylaminopent-2-en-4-one)zinc (Bresciani Pahor, Randaccio & Libertini, 1980), as well as 2·551 (2) and 2·553 (2) Å in diiodobis(pyridine)zinc (Le Querler, Borel & Leclaire, 1977); it is shorter than the Zn—I distance of 2·605 (1) Å in the nearly tetrahedral anion $[ZnI_4]^{2-}$ (Orioli & Lip, 1974). The Zn—N distance is 2·097 (3) Å which is slightly longer than the Zn—N distances of 2·05 (1) and 2·06 (1) Å in diiodobis(pyridine)zinc or 2·035 and 2·045 Å in [2,2-dimethyl-1,3-bis(methylamino)propane]diiodozinc, in which the ligands are stronger bases than HMTA; it is shorter than the distance of 2·111 (8) Å found in $[ZnCl_2$(HMTA)] and 2·129 (4) Å in $[ZnBr_2$(HMTA)], in which HMTA acts as a bidentate bridging ligand which results in an increased electron transfer to the metal atom and accordingly to a decreased basicity of the coordinating N atoms. Comparison of the structures of the three zinc halide–HMTA adducts provides an interesting example of the fact that the coordination mode of HMTA is dependent not only on the central metal but also on the additional ligands. For the $ZnX_2$–HMTA complexes this might be explained by a simple model: the order of Lewis acidity towards nitrogen-containing bases is $ZnCl_2 \simeq ZnBr_2 > ZnI_2$ (Satchell & Wardell, 1964), which means that the $ZnX_2$ moiety is a weaker Lewis acid for $X$=I than for Cl or Br and therefore tends to form an adduct with the HMTA ligand in its more basic monodentate non-bridging state.

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*Acta Cryst.* (1989). C**45**, 363–365

# Structure Cristalline des *cyclo*-Tétraphosphates Pentahydrates: $M^{II}Ag_2P_4O_{12}.5H_2O$ ($M^{II}$ = Co,Ni)

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(*Reçu le* 15 *février* 1988, *accepté le* 11 *octobre* 1988)

**Abstract.** $M_r = 680{\cdot}84$, monoclinic, $P2_1/n$, $a = 15{\cdot}712$ (3), $b = 7{\cdot}263$ (1), $c = 12{\cdot}619$ (3) Å, $\beta = 91{\cdot}85$ (1)°, $V = 1439{\cdot}3$ Å$^3$, $Z = 4$, $D_x = 3{\cdot}141$, $D_m = 3{\cdot}162$ Mg m$^{-3}$, $\lambda$(Mo $K\alpha$) = 0·7107 Å, $\mu = 2{\cdot}237$ mm$^{-1}$, $F(000) = 1308$, room temperature, $R = 0{\cdot}042$ for 3551 independent reflexions. The Co atoms are octahedrally surrounded by two water molecules and four O atoms, forming infinite linear chains parallel to the $c$ axis with a period $[Co(H_2O)_2P_4O_{12}]^{2-}$. $Co(2)O_6$ shares the O($E$31), O($E$41), O($W$2) face with $Ag(2)O_6$ which is linked to $Ag(1)O_6$ by the corner O($E$42). $Co(1)O_6$ is linked to $Ag(1)O_6$ by the edge O($E$11)–O($E$21). Indeed, polyhedra of associated cations form another infinite chain parallel with the $a$ axis: $CoO_6$ octahedra are at the intersection of these two perpendicular infinite chains.

**Introduction.** Les *cyclo*-tétraphosphates correspondant à la formule générale $M^{II}M^{I}_2P_4O_{12}.xH_2O$ ($M^{II}$ = Ni, Co et $M^{I}$ = monovalent) sont peu connus. On ne peut guère citer que: $NiM^{I}_2P_4O_{12}.7H_2O$ ($M^{I}$ = K,$NH_4$) (Jouini, Durif & Dabbabi, 1985) et $M^{II}K_2P_4O_{12}.5H_2O$ ($M^{II}$ = Ni, Co) (Jouini, Soua & Dabbabi, 1987). Dans la présente étude nous décrivons la structure cristalline des pentahydrates $M^{II}Ag_2P_4O_{12}.5H_2O$ ($M^{II}$ = Co, Ni). Des cristaux de $CoAg_2P_4O_{12}.5H_2O$ sont obtenus par la méthode des résines échangeuses d'ions (Jouini & Durif, 1983).

**Partie expérimentale.** Prisme de section 0,18 × 0,20 × 0,23 mm; appareillage Philips PW 1100; monochromateur: graphite; paramètres cristallins obtenus avec 25 réflexions ($10 < \theta < 12°$); domaine des mesures: 3 à 30° ($\theta$); type de balayage: $\omega$; domaine de balayage: (1,2 + 0,2tg$\theta$)°; vitesse de balayage 0,03° s$^{-1}$; réflexions de références: 611, 312 et 352, variation négligeable; temps de mesure du fond contenu: 10 s; nombre de réflexions mesurées: 6779 ($h$: −28 →28, $k$: −13 →13, $l$: 0→22); nombre de réflexions indépendantes: 3551; $R_{int}$ = 0,039; correction de Lorentz–polarisation;

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