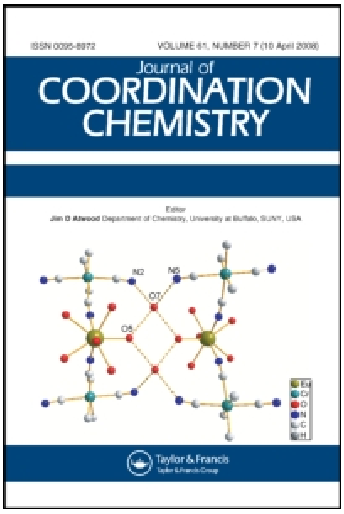

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NICKEL(II), COPPER(II), ZINC(II), CADMIUM(II) AND MERCURY(II) COMPLEXES OF *TRIS*(2-AMINOETHYL)PHOSPHINE OXIDE

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New coordination compounds Ni(TEAPO)$X_2 \cdot nH_2O$, Cu(TAEPO)$(NO_3)_2 \cdot 4H_2O$, Zn(TEAPO)$(NO_3)_2 \cdot 2H_2O$, Cd(TEAPO)$(NO_3)_2 \cdot 2H_2O$, Hg(TEAPO)$Br_2 \cdot 2H_2O$ (TAEPO = *tris*(2-aminoethyl)phosphine oxide, X=Cl or ClO_4, n = 4 or 2) have been synthesized and characterized. As revealed by a single-crystal X-ray analysis of Cu(TAEPO)$(NO_3)_2 \cdot 4H_2O$, the Cu(II) ion has a distorted octahedral arrangement of donor atoms with a CuN_3O_3 chromophore. *Tris*(2-aminoethyl)phosphine oxide acts as a tetradentate donor forming a bridge among three adjacent copper(II) atoms in the cyclic-chain, polymeric structure. Each central atom is coordinated by three nitrogens and one oxygen from three TAEPO ligands in a square-planar geometry. The coordination geometry of Cu(II) is completed by weaker interactions with two water molecules [Cu-O = 2.461(5), 2.601(5) Å]. The remaining water molecules and nitrate anions are not coordinated.

Keywords: transition metal complexes; *tris*(2-aminoethyl)phosphine oxide; X-ray structure

INTRODUCTION

α-Aminoalkylphosphine oxides as electroneutral ligands were considerably studied owing to their possibilities to act as N,O-donor chelating ligands forming five-membered rings.[1-3] An unsymmetric *P*-(*N*,*N*-dimethylaminomethyl)-*P*,*P*-diphenylphosphine oxide also forms coordination compounds in combination with transition metals and their Co(II), Cu(II) and Ni(II) complexes are well

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known[4,5]. On the other hand, *P*-aminoethyl-*P*,*P*-dimethylphosphine oxide affords complexes with Ni(II), Pd(II) or Zn(II) ions, in which it acts as a N,O-donor, bridging ligand.[6-8] In this paper we report the synthesis of the tetradentate ligand *tris*(2-aminoethyl)phosphine oxide (TAEPO) and the preparation of its Ni(II), Cu(II), Zn(II), Cd(II) and Hg(II) coordination compounds. The X-ray structure of the polymeric Cu(TAEPO)$(NO_3)_2 \cdot 4H_2O$ complex is also discussed.

EXPERIMENTAL

Materials

All chemicals were of reagent grade and used as purchased from commercial sources. $PO(CH_2CH_2CONH_2)_3$ was prepared according to the literature method.[9]

Physical Measurements

Carbon, hydrogen and nitrogen analyses were performed on an EA1108 instrument (Fisons). The phosphorus content was determined gravimetrically. Bromine and chlorine were quantified by the Schöniger method. Water molecules were determined gravimetrically by standing samples in a vacuum desiccator over P_4O_{10}. IR spectra were measured on a Specord M80 spectrophotometer (Carl Zeiss, Jena) using nujol mulls in the region 4000-400 cm^{-1}. Electronic absorption spectra (diffuse reflectance) of the Cu(II) and Ni(II) complexes were obtained on a Specord M40 (Carl Zeiss, Jena) instrument in the region 30000-11000 cm^{-1}. The magnetic susceptibility of the complexes was measured by the Faraday method with $Hg[Co(NCS_4)]$ as calibrant. Corrections for the diamagnetism were made using Pascal's constants.[10] ^1H NMR and ^{31}P NMR spectra of TAEPO·3HBr (in D_2O) were recorded with a Tesla BS-587 UG instrument with HMDS as internal standard at 80 MHz (^1H) or 85% H_3PO_4 as external standard at 32 MHz (^{31}P).

X-ray Crystallography

Single crystals of Cu(TAEPO)$(NO_3)_2 \cdot 4H_2O$ suitable for X-ray analysis were obtained by recrystallization from a water solution at 21°C. Data were measured on a Kuma KM-4 diffractometer with graphite-monochromatized Mo-K_α radiation (0.71073 Å) using an $\bar{\omega}/2\theta$ scan mode. Unit cell dimensions were determined from 24 reflections in the range $19.0° < 2\theta < 21.0°$. Three standard reflections (110, 0-10, 002) were monitored after every 300 measurements; no significant changes in their intensities were detected.

TABLE I Crystal Data and Structure Refinement Details for Cu(TAEPO)$(NO_3)_2$·$4H_2O$.

Empirical formula	$C_6H_{26}CuN_5O_{11}P$
Formula weight	438.83
Temperature	301(2) K
Wavelength	0.71073 Å
Crystal system	triclinic
Space group	$P\bar{1}$
Unit cell dimensions	$a = 11.655(2)$ Å $\alpha = 110.92(3)°$
	$b = 11.648(2)$ Å $\beta = 110.94(3)°$
	$c = 15.101(3)$ Å $\gamma = 93.58(3)°$
Volume	1745.5(5) $Å^3$
Z	4
Density (calcd.)	1.670 Mg m^{-3}
Absorption coefficient	1.405 mm^{-1}
$F(000)$	916
Crystal size	0.60 × 0.30 × 0.30 mm
Theta range for data collection	2.40 to 26.09°
Index ranges	$0 \leq h \leq 13, -13 \leq k \leq 13, -16 \leq l \leq 16$
Reflections collected	6549
Independent reflections	6230 [$R(int) = 0.0314$]
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	6230/0/636
Goodness-of-fit on F^2	1.050
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.058$, $wR_2 = 0.171$
R indices (all data)	$R_1 = 0.070$, $wR_2 = 0.180$

The structure was solved by direct methods (SHELXS-86).[11] All non-hydrogen atoms were refined anisotropically by full-matrix least-squares procedures (SHELXL-96)[12] with weighting scheme $w = 1/[\sigma^2(F_o^2) + (0.1110P)^2 + 4.4250P]$, where $P = (F_o^2 + 2F_c^2)/3$. Some hydrogen atoms of water molecules were not located. The other hydrogen atom positions were found from difference Fourier maps and all their parameters were refined. The calculations were performed on PC AT Pentium and IBM RS/6000 98F computers. Crystal data and structure refinement parameters are summarized in Table I. Atomic coordinates of non-hydrogen atoms and equivalent isotropic displacement parameters are given in Table II. Selected bond distances and angles are listed in Table III. The structure of Cu(TAEPO)$(NO_3)_2$·$4H_2O$ and a packing diagram are shown in Figures 1 and 2, respectively.

Syntheses

Preparation of TAEPO·3HBr

$PO(CH_2CH_2CONH_2)_3$ (0.3 mol) was added to a cooled water solution of sodium bromate formed by reaction of NaOH (6 mol) in H_2O (2000 cm^3) with Br_2 (1.2

TABLE II Atomic coordinates (× 10^4) and equivalent isotropic displacement parameters (Å^2 × 10^3) for Cu(TAEPO) $(NO_3)_2 \cdot 4H_2O$. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	*x/a*	*y/b*	*z/c*	U_{eq}
Cu(1)	4124(1)	1919(1)	3307(1)	31(1)
Cu(2)	8080(1)	−4124(1)	1692(1)	32(1)
P(1)	2272(1)	3806(1)	3408(1)	27(1)
P(2)	6195(1)	−2271(1)	1590(1)	27(1)
C(1)	1208(4)	2470(4)	3207(4)	37(1)
C(2)	1221(4)	1293(4)	2348(4)	41(1)
C(3)	2610(4)	5006(4)	4667(3)	33(1)
C(4)	1477(4)	4363(4)	2435(3)	31(1)
C(5)	6329(4)	3909(4)	5012(3)	36(1)
C(6)	6095(4)	−6325(4)	−9(3)	37(1)
C(7)	5125(4)	−323(4)	2475(3)	35(1)
C(8)	5640(4)	−1475(4)	2565(3)	31(1)
C(9)	7525(4)	−1206(4)	1789(4)	37(1)
C(10)	8707(4)	−1219(4)	2654(4)	41(1)
C(11)	4996(4)	−2613(4)	333(3)	33(1)
C(12)	10322(4)	−5129(4)	2521(3)	34(1)
O(1)	3464(3)	3450(3)	3323(2)	34(1)
O(2)	6549(3)	−3462(3)	1682(2)	34(1)
O(3)	6492(9)	1698(9)	1749(8)	165(3)
O(4)	1819(6)	698(8)	4696(6)	126(2)
O(5)	8304(9)	−6524(10)	3265(9)	181(4)
O(6)	9290(7)	−1809(6)	304(6)	122(2)
O(7)	4023(5)	1673(4)	1591(3)	64(1)
O(8)	4086(5)	2155(5)	5075(4)	81(2)
O(9)	7837(6)	−4088(6)	−66(4)	83(2)
O(10)	8324(4)	−4026(5)	3408(3)	63(1)
N(1)	2383(4)	824(4)	2707(4)	43(1)
N(2)	4895(4)	428(4)	3379(3)	36(1)
N(3)	5839(3)	3056(4)	3895(3)	37(1)
N(4)	9178(4)	−2382(4)	2294(4)	44(1)
N(5)	9564(4)	−4905(4)	1621(3)	36(1)
N(6)	6942(4)	−5840(3)	1101(3)	36(1)
N(41)	8850(5)	1853(5)	4704(4)	69(2)
O(41)	7799(5)	1299(6)	4064(5)	97(2)
O(42)	9495(9)	1377(9)	5240(7)	186(5)
O(43)	9349(7)	2904(7)	4809(6)	126(3)
N(31)	8153(5)	1157(5)	306(4)	71(2)
O(31)	7124(6)	640(8)	192(6)	127(3)
O(32)	8637(8)	500(9)	−243(7)	177(4)
O(33)	8701(5)	2206(5)	935(5)	96(2)
N(21)	5217(4)	4775(4)	7501(3)	50(1)
O(21)	5486(8)	5731(5)	8271(5)	126(2)
O(22)	5917(5)	4072(5)	7498(5)	85(2)
O(23)	4272(5)	4507(8)	6721(5)	126(2)
N(11a)[i]	8237(8)	2744(9)	8468(8)	56(2)
O(11a)[i]	7433(8)	1952(8)	8450(9)	78(3)
O(12a)[i]	8919(6)	3680(6)	9221(4)	38(2)
O(13a)[i]	8301(9)	2560(11)	7616(9)	90(4)
N(11b)[i]	7260(9)	1781(8)	6548(8)	52(2)
O(11b)[i]	8042(8)	2574(9)	6557(8)	71(3)
O(12b)[i]	6308(6)	1087(6)	5776(4)	36(2)
O(13b)[i]	7450(11)	1706(9)	7399(8)	79(3)

[i] Atoms having non-unit occupancy factors.

TABLE III Selected bond lengths [Å] and angles [°] for Cu(TAEPO) $(NO_3)_2$·$4H_2O$.

Cu(1)-N(1)	2.025(4)	C(2)-N(1)	1.485(6)
Cu(1)-N(2)	2.024(4)	C(3)-C(5a)[a]	1.517(7)
Cu(1)-N(3)	2.025(4)	C(4)-C(12a)[b]	1.533(6)
Cu(1)-O(1)	1.980(3)	C(5)-N(3)	1.484(6)
Cu(1)-O(7)	2.461(5)	C(5)-C(3a)[a]	1.517(7)
Cu(1)-O(8)	2.601(5)	C(6)-N(6)	1.474(6)
Cu(2)-N(4)	2.027(4)	C(6)-C(11a)[c]	1.521(6)
Cu(2)-N(5)	2.022(4)	C(7)-N(2)	1.462(6)
Cu(2)-N(6)	2.025(4)	C(7)-C(8)	1.533(6)
Cu(2)-O(2)	1.985(3)	C(9)-C(10)	1.528(7)
Cu(2)-O(9)	2.582(5)	C(10)-N(4)	1.488(6)
Cu(2)-O(10)	2.463(5)	C(11)-C(6a)[c]	1.521(6)
P(1)-O(1)	1.510(3)	C(12)-N(5)	1.456(6)
P(1)-C(1)	1.789(5)	C(12)-C(4a)[d]	1.533(6)
P(1)-C(3)	1.795(4)	O(1)-Cu(1)-N(2)	176.23(14)
P(1)-C(4)	1.796(4)	N(3)-Cu(1)-N(1)	178.2(2)
P(2)-O(2)	1.510(3)	O(7)-Cu(1)-O(8)	176.6(2)
P(2)-C(9)	1.787(5)	O(2)-Cu(2)-N(5)	176.14(14)
P(2)-C(11)	1.791(4)	N(6)-Cu(2)-N(4)	178.3(2)
P(2)-C(8)	1.796(4)	O(10)-Cu(2)-O(9)	176.7(2)
C(1)-C(2)	1.520(7)		

Symmetry transformations used to generate equivalent atoms: [a]$-x+1$, $-y+1$, $-z+1$; [b]$x-1$, $y+1$, z; [c]$-x+1$, $-y-1$, $-z$; [d]$x+1$, $y-1$, z.

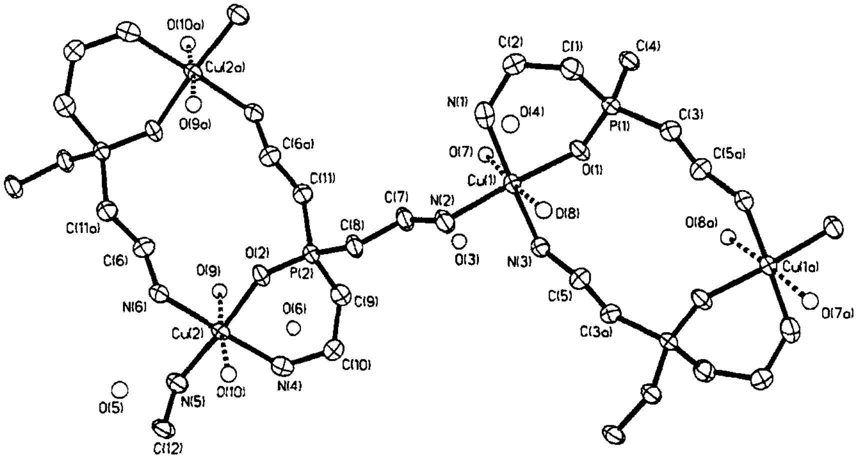

FIGURE 1 The structure of Cu(TAEPO)$(NO_3)_2$·$4H_2O$ (50% ellipsoids except for the water molecules); nitrate anions and hydrogen atoms are omitted for clarity.

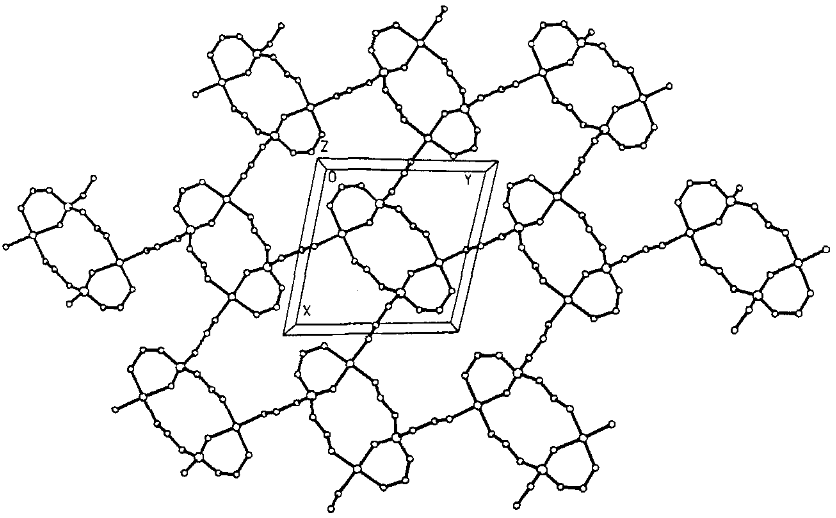

FIGURE 2 Packing diagram for the copper complex showing the polymeric structure (nitrate anions and water molecules are omitted for clarity).

mol). The reaction mixture was stirred until the solution was clear and then it was heated for 20 minutes on a water bath at 60°C. The solution was cooled, acidified with HBr and then evaporated to crystallization. The first fractions of crystals were of NaBr. The rest of the solution was evaporated to dryness. The solid was washed with EtOH, dissolved in H_2O and precipitated by EtOH (55% yield).

Anal. Calcd. for $C_6H_{21}N_3Br_3OP$ (%) ($M_r = 421.9$): C, 17.1; H, 5.0; N, 10.0; Br, 56.8; P, 7.3. Found: C, 16.8; H, 4.9; N, 9.9; Br, 56.9; P, 7.3. ^{31}P NMR (D_2O) = 49.9 ppm; ^1H NMR 2.05-2.40 ppm (2m), 2.40-2.60 ppm (m) and 2.90-3.40 ppm (m, exchange of hydrogens between water molecules and NH_2 groups). IR data (cm^{-1}): 430 w, 484 vs, 612 w, 632 w, 680 w, 722 vs, 734 s, 782 vs, 850 w, 894 m, 918 m, 936 m, 1010 s, 1040 m, 1050 m, 1124 s, 1168 s, 1238 s, 1268 m, 1320 w, 1350 m, 1404 m, 1500 m, 1562 s, 1602 m, 1648 w, 2680 w, 2728 w, 3000-3200 w, 3400-3700 w.

Preparation of TAEPO·3HCl and TAEPO·3HNO$_3$

A water solution of TAEPO·3HBr was filtered through a column of DOWEX 50W in the H^+ cycle until the reaction of the filtrate to Br^- was negative. TAEPO was eluted by 4M HCl (4M HNO_3) and the filtrate was evaporated to dryness on a water bath. The product was triturated with EtOH and dried (nearly quantitative yield). *Anal.* Calcd. for $C_6H_{21}N_3Cl_3OP$(%) ($M_r = 288.6$): C, 25.0; H, 7.3; N, 14.6;

Cl, 36.9; P, 10.7. Found: C, 25.0; H, 7.5; N, 14.5; Cl, 36.8; P, 10.7. *Anal.* Calcd. for $C_6H_{21}N_6O_{10}P$(%) (M_r = 368.2): C, 19.6; H, 5.7; N, 22.8; P, 8.4. Found: C, 19.6; H, 5.9; N, 22.0; P, 8.4.

Preparation of TAEPO·3HClO$_4$

TAEPO·3HBr was reacted with 3.5 equivalents of 70% $HClO_4$ in evaporating dish. The reaction mixture was fumed to a paste-like consistence on a water bath (until HBr formed). The solid after cooling was extracted three-times by EtOH (20 cm^3). Crystals were filtered off and dried in air (nearly quantitative yield). *Anal.* Calcd. for $C_6H_{21}N_3Cl_3O_{13}P$(%) (M_r = 480.5): C, 15.0; H, 4.4; N, 8.7; Cl, 22.1; P, 6.5. Found: C, 15.0; H, 4.5; N, 8.2; Cl, 22.6; P, 6.4.

Preparation of the Complexes with TAEPO

All the complexes were synthesized by a similar procedure. To an EtOH (50 cm^3) suspension of TAEPO·3HX (X = Br, Cl, NO_3, ClO_4) (0.01 mol) was added NaOH (0.03 mol) as 0.1M EtOH solution. The reaction mixture was intensively stirred (ultrasonic stirring) for 30 minutes. The alcoholic solution of TAEPO was filtered and an EtOH solution of M(II) salts (M = Ni, Cu, Zn, Cd and Hg) was added to it. Microcrystals of M(TAEPO)X_2·$n$$H_2O$, which were formed immediately, were filtered off, washed with EtOH and dried in a desiccator over silica gel. The Hg(II) complex is stable in water suspension while the Zn(II), Cd(II) and Ni(II) salts decompose owing to hydrolysis. Only the Cu(II) complex dissolves in water without hydrolysis and it can be recrystallized from water to obtain monocrystals.

IR data for Cu(TEAPO)$(NO_3)_2$·$4H_2O$ (cm^{-1}): 432m, 488 m, 512 m, 562 w, 602 w, 646 m, 700 m, 720 m sh, 740 m, 764 s, 782 w, 808 w, 824 s, 836 m, 880 m, 920 m, 968 s, 992 s, 1024 s, 1042 w, 1060 w, 1088 m, 1120 m, 1140 w, 1166 w, 1232 m, 1250 m, 1274 w, 1320 s, 1390 s, 1472 m, 1496 s, 1520 w, 1560 m, 1586 s, 1612 m, 1642 m, 1680 s, 1754 m, 1842 w, 2640-2780 w, 3100-3500 w, 3600-3800 w.

RESULTS AND DISCUSSION

As can be seen from Figure 1, the TAEPO ligand acts as a tetradentate bridging ligand which is coordinated to the Cu(II) ion as a bidentate N,O-donor forming a six-membered ring and to two adjacent Cu(II) centres, bonded through nitrogen atoms of two remaining 2-aminoethyl groups. The basic coordination geometry of donor TAEPO atoms around each copper atom approaches square-planar. Deviations from the least-squares planes fitted through the Cu(1)-O(1)-N(1)-

TABLE IV Analytical data for the complexes.

		Found (Calcd.) (%)					
Compound	M_r	*C*	*H*	*N*	*P*	X^a	H_2O
$Zn(TAEPO)(NO_3)_2\cdot 2H_2O$	404.6	16.9(17.8)	5.0(5.5)	17.1(17.3)	7.6(7.7)		7.7(8.8)
$Cd(TAEPO)(NO_3)_2\cdot 2H_2O$	451.6	16.1(16.0)	4.6(4.9)	15.2(15.5)	6.6(6.9)		7.3(8.0)
$Hg(TAEPO)Br_2\cdot 2H_2O$	575.6	12.8(12.5)	3.4(3.9)	7.2(7.3)	5.5(5.4)	27.6(27.8)	6.3(6.3)
$Ni(TAEPO)Cl_2\cdot 4H_2O$	380.9	19.0(18.9)	6.9(6.9)	11.2(11.0)	8.0(8.1)	18.1(18.6)	17.1(18.9)
$Ni(TAEPO)(ClO_4)_2\cdot 2H_2O$	472.8	15.2(15.2)	4.6(4.7)	8.6(8.9)	6.4(6.6)	14.6(15.0)	7.0(7.6)
$Cu(TAEPO)(NO_3)_2\cdot 4H_2O$	438.8	16.5(16.4)	5.8(6.0)	15.5(16.0)	7.0(7.1)		17.5(16.4)

[a] X = Cl or Br.

N(2)-N(3) and Cu(2)-O(2)-N(4)-N(5)-N(6) atoms are −0.013(2), 0.027(2), −0.019(2), 0.026(2) and −0.021(2) Å (Rms deviations of fitted atoms = 0.022 Å), and 0.011(2), −0.022(2), 0.016(2), −0.021(2) and 0.017(2) Å (Rms deviations of fitted atoms = 0.018 Å), respectively. The dihedral angle between the above planes is 16.19(2)°. However, the coordination number Cu(II) is increased to six by weak interactions with two oxygens of two water molecules *trans* to each other. An average value of the Cu-N bond lengths is equal to 2.025 Å and is somewhat longer than the Cu-O(TAEPO) distances [1.980(3) and 1.985(3) Å]. The Cu-O(H_2O) distances are significantly longer than the above [Cu(1)-O(H_2O) = 2.461(5), 2.601(5) Å; Cu(2)-O(H_2O) = 2.463(5), 2.582(5) Å]. The Cu-O distances are due to Jahn-Teller distorsion.

Analytical data for the prepared compounds and some physico-chemical properties are given in Tables IV and V, respectively. The magnetic moments of the Ni(II) and Cu(II) complexes indicate two and one unpaired electrons, respectively, even if in the case of $Ni(TEAPO)Cl_2\cdot 4H_2O$ the magnetic moment has a rather high orbital contribution. Thus, the μ_{eff}/μ_B values confirm an octahedral arrangement of donor atoms around the central ions. The absorption maxima displayed at 15600 cm^{-1} and 15900 cm^{-1} in the case of Ni(II) complexes can be attributed to the $^3A_{2g} \rightarrow {}^3T_{1g}$ (F) transition, while the maxima observed at 26000 cm^{-1} and 25000 cm^{-1} are assignable to the $^3A_{2g} \rightarrow {}^3T_{1g}$ (P) transition.[13] The absorption peak in the visible region of the diffuse reflectance spectrum of $Cu(TEAPO)(NO_3)_2\cdot 4H_2O$ (15400 cm^{-1}) is typical of octahedrally coordinated Cu(II) ions with ^2E ground state; however, the band assignment is not unambiguous because of the Jahn-Teller effect.[13] As expected, the Zn(II), Cd(II) and Hg(II) complexes are diamagnetic with the d^{10} configuration and for this reason *d-d* transitions are absent in the visible region.

IR spectra of the TAEPO·3HX (X = Br, Cl, NO_3, ClO_4) ligands, like the complexes, are practically identical and were not studied in detail. The occurrence of bands due to NO_3^- and ClO_4^- (non-coordinated) is clearly

TABLE V Magnetic, diffuse reflectance and IR data for the complexes.

Compound	μ_{eff}/μ_B	*diff.-reflect.spectra* *[×10³; cm⁻¹]*	*IR data [cm⁻¹]* *ν(P=O)*	*X*[a]
$Zn(TAEPO)(NO_3)_2 \cdot 2H_2O$	dia		1252m	ν_1 1048s ν_2 830s ν_3 1396w ν_4 720w sh
$Cd(TAEPO)(NO_3)_2 \cdot 2H_2O$	dia		1240m	ν_1 1054s ν_2 822s ν_3 1386m ν_4 706m
$Hg(TAEPO)Br_2 \cdot 2H_2O$	dia		1264m	-
$Ni(TAEPO)Cl_2 \cdot 4H_2O$	3.40	15.6 26.0	1250s	-
$Ni(TAEPO)(ClO_4)_2 \cdot 2H_2O$	3.10	15.9 25.0	1252s	ν_1 924m ν_2 470m ν_4 624vs
$Cu(TAEPO)(NO_3)_2 \cdot 4H_2O$	1.88	15.4	1250m	ν_1 1042w ν_2 824s ν_3 1390s ν_4 720 m sh

[a]Bands due to anions.

observed in the IR spectra of the complexes (see Table V).[14] The P=O stretching vibration was observed in the 1240-1264 cm^{-1} region. The presence of 2-aminoethyl groups is evident from the NH_2 stretch observed around 3335 cm^{-1} and between 910-770 cm^{-1}. The carbon-nitrogen stretch is found around 1060 cm^{-1} in the IR spectra of all the ligands and complexes.[15] Bands appeared in the 1670-1640 cm^{-1} and 3320-3542 cm^{-1} regions may be attributed to bending and stretching vibrations of non-coordinated water molecules, respectively.[14]

In conclusion, we assume on the basis of the abovementioned study that the complexes have octahedral geometry. The coordination of the TAEPO ligand is identical in all compounds, *i.e.*, it forms bridges, and the complex cations thus have cyclic-chain polymeric structures.

Supplementary Material

A complete lists of bond distances and angles, anisotropic displacement parameters, hydrogen coordinates, and observed and calculated structure factors are available from Zdeněk Trávníček upon request.

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