METAL-ORGANIC COMPOUNDS

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Bis(tetraethylammonium) Tetrachlorometallates, $[(C_2H_5)_4N]_2[MCl_4]$, where M = Hg, Cd, Zn

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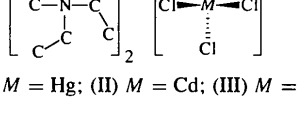
Abstract

Three compounds, $[(C_2H_5)_4N]_2[HgCl_4]$, $[(C_2H_5)_4N]_2[CdCl_4]$ and $[(C_2H_5)_4N]_2[ZnCl_4]$, have been investigated by means of single-crystal X-ray diffraction. They are isomorphous at room temperature. The structures of the three crystals are comprised of discrete $[(C_2H_5)_4N]^+$ cations and $[MCl_4]^{2-}$ anions, which both present dynamic orientational disorder. The major structural difference between these compounds is seen in the size of the slightly distorted $[MCl_4]^{2-}$ tetrahedra.

Comment

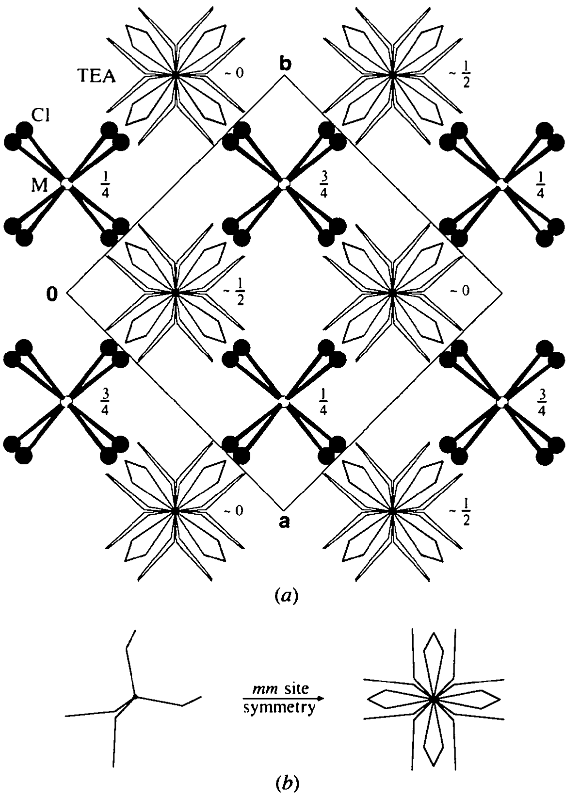
Extensive studies have been carried out on the crystal structures, phase transitions and physical properties of the extensive family of A_2BX_4-related materials. The title compounds belong to a subgroup of the A_2BX_4 series in which the A ion corresponds to the tetraethylammonium cation, $[(C_2H_5)_4N]^+$ (TEA), B to the divalent metal and X to the Cl atom. Several studies (Wolthuis, Huiskamp, De Jongh & Carlin, 1986; Iwata & Ishibashi, 1991; Czapla & Dacko, 1992; Kahrizi & Steinitz, 1990; Gesi, 1994) have revealed some interesting physical properties of these crystals connected with several phase transitions. As part of our attempt at understanding the mechanism of the phase transitions in these materials, we report the crystal structures of $(TEA)_2[HgCl_4]$, (I), $(TEA)_2[CdCl_4]$, (II), and $(TEA)_2[ZnCl_4]$, (III), at room temperature.



(I) M = Hg; (II) M = Cd; (III) M = Zn

The crystal structure analyses of the title compounds show them to have a similar organization to that of both $(TEA)_2[NiCl_4]$ (Stucky, Folker & Kistenmacher, 1967) and $(TEA)_2[CuCl_4]$ (Mahoui, Lapasset, Moret & Saint Grégoire, 1994). There is one $[MCl_4]^{2-}$ anion and one TEA moiety in the asymmetric unit of each of the title compounds and the mutual arrangement of the moieties is illustrated in Fig. 1(*a*). The structure can be described as alternating A and B layers stacked along [110]. Layer A is formed by $[MCl_4]^{2-}$ anions, while layer B is composed of TEA cations. This organization is very close to an antifluorite structure; each $[MCl_4]^{2-}$ ion is surrounded by eight TEA cations at the vertices of a pseudo-cube, while, reciprocally, each TEA cation is at the centre of a tetrahedron formed by four $[MCl_4]^{2-}$ ions. The particular geometry of the TEA and $[MCl_4]^2$ groups (less than a spherical symmetry) results in a lowering of the space-group symmetry ($P4_2/nmc$) which remains, however, a subgroup of the space group of the fluorite structure ($Fm\bar{3}m$).

The $[MCl_4]^{2-}$ tetrahedra are disordered between two equivalent orientations relative to the mirror planes



(*a*)

(*b*)

Fig. 1. (*a*) The structure of $(TEA)_2[MCl_4]$ (M = Zn, Cd, Hg) viewed down the c axis. Note the z coordinates of the central ions (~0 means z = 0.0888 and ~$\frac{1}{2}$ means z = 0.5888) which emphasize the similarity of the organization with that of antifluorite. (*b*) The shaping of the TEA group with one orientation (left side) and four equivalent orientations (right side) generated by the *mm* symmetry operations.

intersecting along the $\bar{4}$ axis; the central divalent metal *M* atom is in a special position $(-\frac{1}{4},\frac{1}{4},\frac{1}{4})$ on a $\bar{4}$ axis, whereas the Cl atoms have been found to be displaced out of the mirror planes as for $[CuCl_4]^{2-}$ in $(TEA)_2[CuCl_4]$ (Mahoui *et al.*, 1994) and unlike $[NiCl_4]^{2-}$ in $(TEA)_2[NiCl_4]$ (Stucky *et al.*, 1967), where the Cl atoms lie on the mirror planes. The Cl—*M*—Cl angles are very close to the expected tetrahedral value, being in the range 108–110°. The TEA groups have a nordic-cross shape. Unlike $(TEA)_2[NiCl_4]$ (Stucky *et al.*, 1967) and $(TEA)_2[CuCl_4]$ (Mahoui *et al.*, 1994), where disorder of the TEA groups corresponds to two equivalent orientations, the disorder of TEA in the three title crystals is more complicated, with four equivalent orientations as shown in Fig. 1(*b*); the N atom is in a special position $(\frac{1}{4},\frac{1}{4},0.5888)$ on a 4_2 axis, whereas the C atoms of the four ethyl chains joined to the N atom are in general positions (except for C11 and C22 which are found on mirrors with $x = \frac{1}{4}$ and $y = \frac{1}{4}$, respectively). Such disorder could result in the twinning of the crystals. We have checked that ferroelastic twinning does not occur; the samples are optically uniaxial at room temperature, which shows that the tetragonal symmetry holds.

The main difference between these compounds is the size of the $[M\mathrm{Cl}_4]^2$ tetrahedron and therefore the compactness of the structure. In fact, the *a* parameters of the three tetragonal cells are close in value to each other, whereas the *c* parameters increase with the size of $[M\mathrm{Cl}_4]^{2-}$ [from $c = 14.964\,(2)$ Å for Zn—Cl = 2.265 (2) Å in (III) to $c = 15.439\,(3)$ Å for Hg—Cl = 2.458 (3) Å in (I)]. Two phase transitions have been observed for these crystals (Caëtano, Lopez, Mahoui, Lapasset, Moret, Assih & Saint Grégoire, 1995). They can be related, although probably through different steps, to the ordering of the TEA and $[M\mathrm{Cl}_4]^{2-}$ moieties at low temperature.

Experimental

The preparation of the three title compounds, $[(C_2H_5)_4N]_2[M\mathrm{Cl}_4]$ (*M* = Hg, Cd, Zn), was carried out according to the method described by Gill & Nyholm (1959). Colourless single crystals suitable for X-ray analysis were obtained by recrystallization from ethanol for all three compounds.

Compound (I) (*M* = Hg)

Crystal data

$(C_8H_{20}N)_2[HgCl_4]$
$M_r = 602.89$
Tetragonal
$P4_2/nmc$
$a = 8.962\,(2)$ Å
$c = 15.439\,(3)$ Å
$V = 1240.1\,(5)$ Å^3
$Z = 2$
$D_x = 1.615$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 7–14°$
$\mu = 6.638$ mm^{-1}
$T = 293\,(2)$ K
Transparent prism
0.37 × 0.33 × 0.13 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: *SHELX*76 Gaussian (Sheldrick, 1976)
$T_{\min} = 0.1741$, $T_{\max} = 0.4342$
2101 measured reflections
906 independent reflections

444 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int} = 0.009$
$\theta_{\max} = 29.97°$
$h = 0 \rightarrow 8$
$k = 0 \rightarrow 12$
$l = 0 \rightarrow 21$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0299$
$wR(F^2) = 0.0862$
$S = 0.886$
900 reflections
38 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.0424P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = -0.001$
$\Delta\rho_{\max} = 0.358$ e Å^{-3}
$\Delta\rho_{\min} = -0.370$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (I)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	$U_{\rm eq}$
Hg	−1/4	1/4	1/4	0.0672 (2)
Cl†	−0.0294 (3)	0.2231 (4)	0.3433 (2)	0.0980 (8)
N	1/4	1/4	0.5888 (6)	0.067 (2)
C1‡	0.219 (3)	0.0859 (12)	0.5823 (11)	0.106 (5)
C11†	1/4	0.022 (2)	0.4922 (11)	0.106 (4)
C2‡	0.4168 (12)	0.283 (3)	0.5926 (12)	0.106 (5)
C22†	0.482 (3)	1/4	0.6836 (11)	0.106 (4)
C3‡	0.177 (2)	0.318 (3)	0.6669 (11)	0.106 (5)
C33‡	0.004 (2)	0.325 (3)	0.6596 (16)	0.106 (4)
C4‡	0.199 (3)	0.320 (2)	0.5059 (10)	0.106 (5)
C44‡	0.181 (3)	0.491 (2)	0.5113 (16)	0.106 (4)

† Site occupancy = 0.50. ‡ Site occupancy = 0.25.

Table 2. *Selected geometric parameters (Å, °) for (I)*

Hg—Cl	2.458 (3)	C1—C11	1.530 (10)
N—C4	1.500 (9)	C2—C22	1.550 (10)
N—C1	1.501 (9)	C3—C33	1.552 (10)
N—C3	1.501 (9)	C4—C44	1.541 (10)
N—C2	1.525 (9)		
Cl—Hg—Cl$^{\rm i}$	110.09 (7)	C1—N—C2	112.1 (15)
Cl—Hg—Cl$^{\rm ii}$	108.24 (15)	C3—N—C2	108.7 (12)
C4—N—C1	107.2 (12)	N—C1—C11	113.1 (14)
C4—N—C3	112.3 (11)	N—C2—C22	111.5 (14)
C1—N—C3	111.6 (13)	N—C3—C33	113.2 (15)
C4—N—C2	104.7 (13)	N—C4—C44	113.8 (14)

Symmetry codes: (i) $-y, \frac{1}{2}+x, \frac{1}{2}-z$; (ii) $-\frac{1}{2}-x, \frac{1}{2}-y, z$.

Compound (II) (*M* = Cd)

Crystal data

$(C_8H_{20}N)_2[CdCl_4]$
$M_r = 514.70$

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å

Tetragonal
$P4_2/nmc$
$a = 9.034$ (2) Å
$c = 15.297$ (3) Å
$V = 1248.5$ (5) Å^3
$Z = 2$
$D_x = 1.369$ Mg m^{-3}
D_m not measured

Cell parameters from 25 reflections
$\theta = 7–16°$
$\mu = 1.304$ mm^{-1}
$T = 293$ (2) K
Transparent prism
0.21 × 0.20 × 0.12 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: *SHELX*76 Gaussian (Sheldrick, 1976)
$T_{\min} = 0.8454$, $T_{\max} = 0.8912$
2098 measured reflections
885 independent reflections

376 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int} = 0.0093$
$\theta_{\max} = 29.95°$
$h = 0 \rightarrow 8$
$k = 0 \rightarrow 12$
$l = 0 \rightarrow 21$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0389$
$wR(F^2) = 0.1028$
$S = 0.755$
881 reflections
38 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.0493P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.397$ e Å^{-3}
$\Delta\rho_{\min} = -0.272$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (II)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Cd	−1/4	1/4	1/4	0.0652 (3)
Cl†	−0.0325 (3)	0.2271 (5)	0.34247 (15)	0.1113 (6)
N	1/4	1/4	0.5896 (5)	0.071 (2)
C1‡	0.211 (2)	0.0880 (11)	0.5799 (9)	0.107 (4)
C11†	1/4	0.0300 (18)	0.4887 (8)	0.103 (3)
C2‡	0.4166 (10)	0.284 (3)	0.5946 (9)	0.107 (4)
C22†	0.4726 (19)	1/4	0.6882 (8)	0.103 (3)
C3‡	0.1729 (15)	0.322 (2)	0.6652 (9)	0.107 (4)
C33‡	0.0010 (16)	0.318 (2)	0.6587 (11)	0.103 (3)
C4‡	0.198 (2)	0.3217 (15)	0.5063 (8)	0.107 (4)
C44‡	0.188 (2)	0.4918 (16)	0.5084 (12)	0.103 (3)

† Site occupancy = 0.50. ‡ Site occupancy = 0.25.

Table 4. *Selected geometric parameters (Å, °) for (II)*

Cd—Cl	2.430 (2)	C1—C11	1.531 (9)
N—C3	1.499 (9)	C2—C22	1.549 (9)
N—C4	1.504 (8)	C3—C33	1.556 (9)
N—C1	1.513 (9)	C4—C44	1.540 (10)
N—C2	1.538 (9)		
Cl—Cd—Cl$^{\rm i}$	109.81 (6)	C2—N—C4	105.1 (10)
Cl—Cd—Cl$^{\rm ii}$	108.80 (12)	C3—N—C4	108.8 (8)
C1—N—C2	115.3 (11)	N—C1—C11	111.5 (11)
C1—N—C3	112.7 (10)	N—C2—C22	109.0 (11)
C1—N—C4	105.1 (9)	N—C3—C33	113.8 (11)
C2—N—C3	109.2 (9)	N—C4—C44	115.6 (11)

Symmetry codes: (i) $y - \frac{1}{2}, -x, \frac{1}{2} - z$; (ii) $-\frac{1}{2} - x, \frac{1}{2} - y, z$.

Compound (III) (M = Zn)

Crystal data

$(C_8H_{20}N)_2[ZnCl_4]$
$M_r = 467.67$
Tetragonal
$P4_2/nmc$
$a = 9.0147$ (10) Å
$c = 14.964$ (2) Å
$V = 1216.1$ (3) Å^3
$Z = 2$
$D_x = 1.277$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 10–43°$
$\mu = 5.437$ mm^{-1}
$T = 293$ (2) K
Transparent prism
0.31 × 0.30 × 0.17 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: *SHELX*76 Gaussian (Sheldrick, 1976)
$T_{\min} = 0.2934$, $T_{\max} = 0.4717$
1072 measured reflections
511 independent reflections

416 observed reflections [$I > 2\sigma(I)$]
$R_{\rm int} = 0.0547$
$\theta_{\max} = 59.92°$
$h = 0 \rightarrow 7$
$k = 0 \rightarrow 10$
$l = 0 \rightarrow 16$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0599$
$wR(F^2) = 0.1924$
$S = 1.120$
511 reflections
39 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.1205P)^2 + 0.8609P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.450$ e Å^{-3}
$\Delta\rho_{\min} = -0.366$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0064 (18)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 5. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (III)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Zn	−1/4	1/4	1/4	0.0636 (8)
Cl†	−0.0474 (3)	0.2265 (5)	0.3383 (2)	0.1013 (8)
N	1/4	1/4	0.5866 (5)	0.067 (2)
C1‡	0.214 (3)	0.0889 (13)	0.5722 (12)	0.112 (4)
C11†	1/4	0.035 (2)	0.4770 (11)	0.099 (4)
C2‡	0.4121 (13)	0.289 (3)	0.6021 (12)	0.112 (4)
C22†	0.462 (2)	1/4	0.6988 (10)	0.099 (4)
C3‡	0.1696 (18)	0.325 (3)	0.6612 (12)	0.112 (4)
C33‡	−0.0007 (18)	0.307 (3)	0.6601 (13)	0.099 (4)
C4‡	0.191 (3)	0.3254 (18)	0.5046 (11)	0.112 (4)
C44‡	0.197 (3)	0.4961 (18)	0.5008 (15)	0.099 (4)

† Site occupancy = 0.50. ‡ Site occupancy = 0.25.

Table 6. *Selected geometric parameters (Å, °) for (III)*

Zn—Cl	2.265 (2)	C1—C11	1.540 (10)
N—C3	1.492 (9)	C2—C22	1.554 (10)
N—C4	1.499 (9)	C3—C33	1.543 (10)
N—C1	1.504 (9)	C4—C44	1.540 (10)
N—C2	1.519 (10)		

Cl—Zn—Cl^i	109.91 (7)	C2—N—C4	111.2 (12)
Cl—Zn—Cl^{ii}	108.60 (14)	C3—N—C4	103.6 (12)
C1—N—C2	116.8 (14)	N—C1—C11	112.9 (13)
C1—N—C3	116.0 (13)	N—C2—C22	111.6 (13)
C1—N—C4	104.1 (12)	N—C3—C33	115.3 (14)
C2—N—C3	104.5 (12)	N—C4—C44	118.2 (15)

Symmetry codes: (i) $-y, \frac{1}{2}+x, \frac{1}{2}-z$; (ii) $-\frac{1}{2}-x, \frac{1}{2}-y, z$.

The structures were solved by the Patterson method using *SHELXS*86 (Sheldrick, 1990) and from subsequent difference Fourier calculations using *SHELXL*93 (Sheldrick, 1993), and refined by full-matrix least-squares techniques, with isotropic displacement parameters for non-H atoms, except for Hg and Cl which were refined anisotropically. Because of disorder of the tetraethylammonium group, the bond lengths were constrained [N—C = 1.50 (1) and C—C = 1.54 (1) Å]. The H atoms were idealized with the standard methods in *SHELXL*93 and included in the least-squares process as fixed contributors, with a common freely refining isotropic displacement parameter. The figure was prepared using *ORTEP*II (Johnson, 1976) adapted for the Macintosh by André & Michalowicz (1991).

For all compounds, data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: local program; program(s) used to refine structures: *SHELXL*93; software used to prepare material for publication: *SHELXL*93.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MD1004). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tetraethylammonium Tetramethylammonium Tetrachlorocuprate(II), $[(C_2H_5)_4N][(CH_3)_4N][CuCl_4]$

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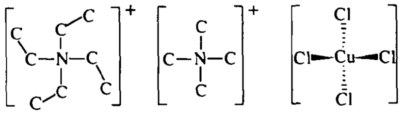
Abstract

The title compound contains four isolated and discrete moieties, *i.e.* two independent $[(CH_3)_4N]^+$ cations, with one ordered on a $\bar{4}$ axis and the other disordered with the central N atom on a 2_z axis, one $[(C_2H_5)_4N]^+$ cation, on a mirror plane and in a *trans* conformation, and one distorted tetrahedral $[CuCl_4]^{2-}$ anion on another mirror. The crystal structure is isomorphous with that of $[(C_2H_5)_4N][(CH_3)_4N][ZnCl_4]$ [Caëtano, Lapasset & Saint Grégoire (1995). *Acta Cryst.* C**51**, 220–222].

Comment

As part of our studies of structural instabilities in the $[(C_2H_5)_4N]_2[MX_4]$ family (Mahoui, Lapasset, Moret & Saint Grégoire, 1994; Mahoui, Lapasset, Sannikov, Moret & Saint Grégoire, 1996; Mahoui, Lapasset, Moret, Lopez & Saint Grégoire, 1995; Caëtano, Lopez, Mahoui, Lapasset, Moret, Assih & Saint Grégoire, 1995), we have investigated the single-crystal structure of the title compound, (I), which belongs to a sub-family of $[(C_2H_5)_4N]_2[MX_4]$, where one $[(C_2H_5)_4N]^+$ (TEA) cation has been substituted by a $[(CH_3)_4N]^+$ (TMA) cation and MX_4 corresponds to $CuCl_4$.



(I)

The crystal structure analysis of (I) shows a structure isomorphous with (TEA)(TMA)[ZnCl$_4$] (Caëtano, Lapasset & Saint Grégoire, 1995). In this compound, the substitution of zinc by copper does not modify the structure, unlike in (TEA)$_2$[ZnCl$_4$] (Mahoui, Lapasset, Moret & Saint Grégoire, 1996) and (TEA)$_2$[CuCl$_4$] (Mahoui, Lapasset, Sannikov, Moret & Saint Grégoire, 1996), where the TEA cations possess different disorders.

The crystal structure of (I) consists of discrete $[CuCl_4]^{2-}$, TEA and TMA moieties. The mutual arrangement of the different moieties is illustrated in

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