Crystals of suitable dimensions were grown by slow evapora-
tion at room temperature from a saturated aqueous solution ob-
tained by dissolving $[(C_2H_5)_2NH_2]Cl$ and $BiCl_3$ (molar ratio
3:1) in 36% HCl. Absolute configuration was determined by
least-squares comparison of the two different forms. The R val-
ues obtained for the inverse structure are $R = 0.05$ and $wR =
0.06$. H atoms were located from a difference Fourier map; their
positions were refined with a common displacement parameter.
$\Delta\rho_{max}$ was exactly at the position of Bi; all other maxima in
the difference map were smaller than 0.36 e Å^{-3}. All compu-
tations were performed on a PC'386 with an adapted version of
the program system *ATARI CRYSTAN*88 (Burzlaff & Rotham-
mel, 1989).

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port.

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Structure of [NH3(C2H5)]2[Hg(CN)2Cl2]

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Abstract

The structure of bis(ethylammonium) dichlorodi-
cyanomercurate consists of slightly deformed tetrag-
onal nets of Cl atoms parallel to (100) at $x = 0.25$
and 0.75. Half of the chlorine squares are nearly
centred by Hg atoms resulting in a chess-board

arrangement. The environment of the Hg atom is
augmented to a distorted octahedron by two CN
groups nearly perpendicular to the chlorine net. The
$C_2H_5NH_3$ groups are located above and below the
non-centred chlorine squares. Two H atoms of the
NH_3 group bridge to Cl atoms of the neighbouring
net with distances of 2.233 (2) and 2.442 (2) Å. The
shortest distance from the third H atom to the N
atom of the CN group located in the next slice is
2.078 (7) Å. Thus the two slices may be regarded as
being tied up by hydrogen bridges. The nets are
shifted against each other in the **b** direction accord-
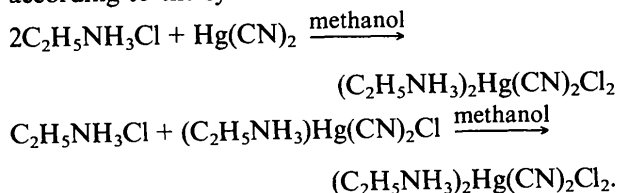ing to the glide operation **a**.

Comment

The reaction of substituted ammonium halogenides
with metal halogenides leads to a series of com-
pounds of the general formula $(R_xNH_{4-x})_2MX_4$,
with R being an aliphatic or aromatic hydrocarbon,
M a divalent metal and X a halogen Cl, Br, I. Most
of these compounds show interesting structural and
physical properties such as ferroelectricity or ferro-
elasticity. Many of them show successive phase tran-
sitions and quite often incommensurate phases {*e.g.*
$[N(CH_3)_4]_2ZnCl_4$, *cf.* Mashiyama (1980)}. The phase
transitions are mainly a result of order–disorder
effects for the substituted ammonium group. An
example for interesting physical properties is the
compound $[N(CH_3)_4]_2MnCl_4$ that was used by
Dupas & Renard (1973) and Birgeneau, Shirane &
Kitchens (1974) for studying low-dimensional mag-
netism.

Another point of interest is the investigation of the
coordinative environment of Hg^{II}; because of the
closed d shell and relatively large coordination dis-
tances it will be difficult to distinguish bonded and
non-bonded neighbours. Preferred occurance of
nearly linear $HgCl_2$ units is reported (*e.g.* Subra-
manian & Seff, 1980) with Hg—Cl distances from
2.28 (1) to 2.30 (1) Å. Additional weaker interactions
are reported that range from 3.36 (1) to 3.48 (1) Å
and augment the environment to a distorted hexago-
nal scalenohedron (coordination No. 8). Regular
octahedral coordination is rare and has been
observed for complexes with O atoms (Sandström,
1978) or ternary F atoms (Hoppe, Wilhelm &
Müller, 1972) as ligands. Other coordination
numbers can be three, four (tetrahedral) and five
(trigonal bipyramidal) as reported by Sandström
(1978) and Grdenic (1965). In the special case of
$(RNH_3)_2HgX_4$, perovskite-like structures with MX_6
octahedra are found (Ben Salah, Daoud, Constant,
Jaud & Galy, 1983; Ben Salah, Daoud, Bats & Fuess,
1986).

The present investigation intends to study the
effects of the substitution of Cl atoms by CN groups.

Single crystals of the compound bis(ethylammonium) dichlorodicyanomercurate are obtained by mixing, in stoichiometric ratio and using methanol as solvent, the salts ethylammonium chloride and cyanide mercurate or ethylammonium chloride and monochlorine dicyanide mercurate(II) of ethylammonium(3,4) according to the synthesis reactions:

$$2C_2H_5NH_3Cl + Hg(CN)_2 \xrightarrow{\text{methanol}}$$

$$(C_2H_5NH_3)_2Hg(CN)_2Cl_2$$

$$C_2H_5NH_3Cl + (C_2H_5NH_3)Hg(CN)_2Cl \xrightarrow{\text{methanol}}$$

$$(C_2H_5NH_3)_2Hg(CN)_2Cl_2.$$

Slow evaporation at room temperature of limpid solution leads to transparent colourless slender square-shaped crystals. Chemical analysis confirms the formula above. The structural arrangement is described in the *Abstract*. Further results are given in the *Experimental* and in Table 1 (coordinates) and Table 2 (distances and angles). The substitution of half of the Cl atoms (in HgCl$_4$) by CN groups changes not only the dimension of the 'pseudo-tetragonal' (HgCl$_2$) layers significantly (*cf.* Ben Salah, Daoud, Constant, Jaud & Galy, 1983; Ben Salah, Daoud, Bats & Fuess, 1986) but gives rise to strong changes in the coordination environment of the Hg atom: the Hg—Cl connections increase from 2.767 (1)–2.842 (1) or 2.81 Å to 2.901 (2)–3.432 (2) Å; the angles of 90 or 180° within the net are changed to 92.64 (5), 97.69 (6) and 167.65 (5)°. The local symmetry *mmm* for the Hg atom is lowered to 2/*m*.
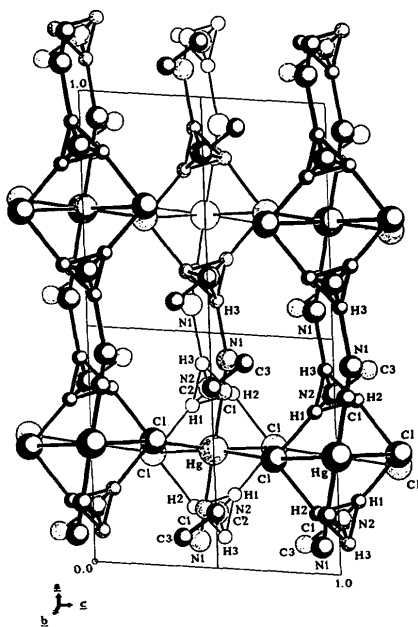

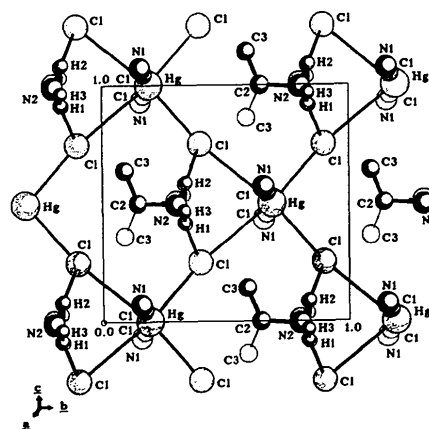Fig. 1. Unit cell (projection along *b*).

Fig. 2. Unit cell (projection along *a*; 0 < *x* < 0.5).

Contrary to the earlier investigations no disorder effects could be recognized for the cation in this structure. The angle N2—C2—C3 and the distances N2—C2 and C2—C3 are in good agreement with the values adopted normally (*cf.* Table 2).

Experimental

Crystal data

[NH$_3$(C$_2$H$_5$)]$_2$[Hg(CN)$_2$Cl$_2$]	D_x = 2.146 Mg m^{-3}
M_r = 415.72	Mo $K\alpha_1$ radiation
Monoclinic	λ = 0.70926 Å
$A2/a$	Cell parameters from 46 reflections
a = 16.320 (1) Å	θ = 8.4–11.7°
b = 9.153 (1) Å	μ = 12.35 mm^{-1}
c = 8.649 (1) Å	T = 293 K
β = 95.21 (1)°	Tetragonal columns
V = 1287 Å^3	0.33 × 0.20 × 0.20 mm
Z = 4	Colourless

Data collection

PW1100 (modified) diffractometer	R_{int} = 0.031
ω–2θ scans	θ_{max} = 24.5°
Absorption correction:	h = −19 → 19
analytical by integration	k = −10 → 10
from crystal shape	l = −10 → 10
T_{min} = 6.32, T_{max} = 14.81	6 standard reflections
4325 measured reflections	monitored every 100
1079 independent reflections	reflections
1079 observed reflections	intensity variation: 1.00–
[all reflections observed]	0.93, mean 0.95%

Refinement

Refinement on F	$\Delta\rho_{max}$ = 3.19 e Å^{-3} near Hg
Final R = 0.0390	$\Delta\rho_{min}$ = −1.54 e Å^{-3} near Hg
wR = 0.0392	Extinction correction: equation (22) of Ahmed, Hall & Huber (1970)
S = 2.54	Extinction coefficient:
1079 reflections	2365 (270)
62 parameters	

H-atom parameters not refined	Atomic scattering factors from *International Tables for X-ray Crystallography* (1962, Vol. III)	

$w = 1/\sigma^2(F)$

$(\Delta/\sigma)_{max} = 0.00$

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

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

	x	y	z	U_{eq}
Hg	0.25000	0.18949 (5)	0.00000	0.0417
Cl	0.2374 (1)	0.3981 (2)	0.2493 (2)	0.0476
C1	0.3757 (5)	0.1674 (8)	0.046 (1)	0.0443
N1	0.4435 (4)	0.155 (1)	0.0699 (9)	0.0588
N2	0.3824 (4)	0.3005 (8)	0.492 (1)	0.0571
C2	0.3828 (6)	0.143 (1)	0.501 (1)	0.0674
C3	0.4381 (6)	0.089 (1)	0.640 (1)	0.0629

Table 2. *Geometric parameters* (Å, °)

HgCl$_4$(CN)$_2$ octahedron

Hg—Cl	2.901 (2)	Cl—Hg—Cl^i	92.64 (5)
Hg—Cl^i	3.432 (2)	Cl—Hg—Cl^{ii}	97.69 (6)
Hg—Cl	2.064 (9)	Cl—Hg—Cl^{iii}	167.65 (5)
Hg—N1	3.176 (7)	Cl^i—Hg—Cl^{iii}	77.99 (5)
		Cl—Hg—Cl	93.3 (2)
		Cl—Hg—Cl^i	78.5 (2)
		Cl—Hg—Cl^{iii}	92.7 (2)
		Cl—Hg—Cl^{ii}	94.1 (2)
		Cl—Hg—Cl^{ii}	168.8 (3)

Hg(CN)

Cl—N1	1.11 (1)	Hg—Cl—N1	179.6 (5)

C$_2$H$_5$NH$_3$

C2—N2	1.45 (1)	N2—C2—C3	111.5 (8)
C2—C3	1.52 (1)		

Hydrogen bridges

H1···N2	0.92 (1)	H2···Cl^{iv}	2.442 (2)
H1···Cl	2.233 (2)	H3···N2	0.92 (1)
H2···N2	0.92 (1)	H3···N1^v	2.078 (7)

Symmetry codes: (i) $2,[\tfrac{1}{4}y\tfrac{1}{4}]$; (ii) $2[\tfrac{1}{4}y0]$; (iii) $4[0\tfrac{1}{2}\tfrac{1}{2}]$; (iv) $2[\tfrac{1}{4}y\tfrac{1}{4}]$; (v) $\overline{1}[\tfrac{1}{4}\tfrac{1}{4}\tfrac{1}{4}]$.

The six standard reflections first showed a small increase then a small decrease in intensity indicating changing extinction behaviour. The positions of Hg and Cl atoms were determined by direct methods; a subsequent difference Fourier map revealed all N and C atoms. All H atoms could be found in correct positions on a difference Fourier map. Refinement, however, resulted in slightly deformed arrangements. Thus for the final refinement, H atoms were set at calculated positions and kept fixed. All computations were performed on a PC'386 with an adapted version of the program system *ATARI CRYSTAN*88 (Burzlaff & Rothammel, 1989).

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A Monodentate 1,3-Diphenyl-2-triazeno Terpyridineplatinum(II) Complex

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Abstract

The compound (1,3-diphenyl-2-triazeno)(2,2′,2″-terpyridine)platinum(II) perchlorate dimethylformamide solvate (1) is formed by reaction of dpt (dptH = 1,3-diphenyltriazene) and [Pt(tpy)Cl]Cl (tpy = terpyridine) in the presence of excess base (NEt$_3$). There are two independent cations in the asymmetric unit. The platinum centers have a monodentate triazeno ligand with the remaining coordination sites occupied by the terpyridine group. The crystal packing is dominated by π–π stacking interactions, and metal–metal interactions are conspicuously absent.

Comment

Both metal–metal (Smith, Miskowski, Mason & Gray, 1990; Rice, Miskowski & Gray, 1987; Stiegman, Rice, Gray & Miskowski, 1987; Rice & Gray, 1983) and ligand(π)–ligand(π) (Miskowski &

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† Contribution No. 8734.