shifts in parameters were less than their e.s.d.'s. Positional parameters are given in Table 1, bond distances and angles in Table 2.*

Discussion. The analysis confirms that the yellow isomer (*c*) of $[NiL](ClO_4)_2$ (Hay, Piplani & Jeragh, 1977) contains the cation (I) (Fig. 1), in which there is an approximate twofold axis through Ni perpendicular to the NiN_4 plane. Ni has one close $O[ClO_4]$ contact of 2·79 Å with O(111) which lies on the opposite side of the NiN_4 plane from the axial methyl groups. The five-membered rings have close to envelope conformations [C(3)—N(4)—Ni—N(1) and N(8)—Ni—N(11)—C(10) $\simeq$ 0°], and the six-membered rings are in approximate sofa conformations.

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34361 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

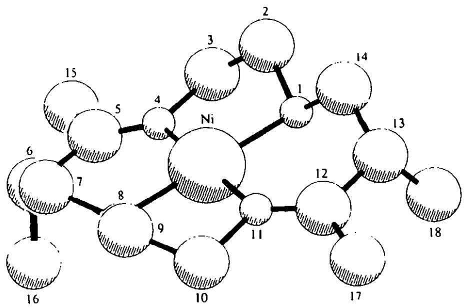

Fig. 1. General view of the cation.

The crystal structure of isomer (*b*) of this compound contains centrosymmetric *N-meso-C-meso* cations (Ferguson, Restivo & Hay, 1979), in which the geometries of the five- and six-membered rings are similar to those found in the present study.

We thank Dr D. P. Piplani for crystals.

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Acta Cryst. (1979). B35, 1706–1709

Dicadmium Dimethylammonium Pentachloride Dihydrate

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(*Received* 3 *January* 1979; *accepted* 10 *April* 1979)

Abstract. $(CH_3)_2NH_2Cd_2Cl_5.2H_2O$, $C_2H_8N^+.2Cd^{2+}.5Cl^-.2H_2O$, monoclinic, *Ic*, $Z = 4$, $a = 9{\cdot}047$ (2), $b = 21{\cdot}694$ (6), $c = 6{\cdot}529$ (1) Å, $\beta = 90{\cdot}57$ (2)°, $V = 1281{\cdot}4$ (5) Å^3 at 294 K, $D_c = 2{\cdot}51$, $D_m = 2{\cdot}49$ (1) Mg m^{-3}, $\mu = 4{\cdot}49$ mm^{-1}, 1885 diffractometer data up to $\sin\theta/\lambda = 0{\cdot}70$ Å^{-1}, final $R(F) = 0{\cdot}018$. The structure consists of corner-sharing $CdCl_6$ and $CdCl_5(H_2O)$ octahedra, forming infinite zigzag chains along **c**. The dimethylammonium ions are located in the free space between the chains. They and the hydrate molecules are involved in hydrogen bonding.

Introduction. Colourless plates of the title compound were obtained from an aqueous solution of equimolar quantities of $(CH_3)_2NH_2Cl$ and $CdCl_2$. Chemical analyses (wt%): theoretical: C 4·96, H 2·50, N 2·89, Cl 36·61, Cd 46·43, H_2O 7·44; experimental: C 4·95, H 2·31, N 2·94, Cl 36·74, Cd 46·27, H_2O 7·29.

A crystal 0·60 × 0·26 × 0·11 mm was selected for the experiments. Precession photographs showed the space group to be either *Cc* or *C*2/*c*; the former was found to be correct during the structure determination. In order to avoid an unfavourable β angle of 126°, a transformation was made according to $a_{new} = a_{old} + c_{old}$. The space group then is *Ic*.

Data were collected on a Syntex $P2_1$ diffractometer with Nb-filtered Mo $K\alpha$ radiation. Reflections were measured in two quadrants of reciprocal space ($h,k,\pm l$;

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$h,-k,\pm l$) up to $\sin\theta/\lambda = 0{\cdot}70$ Å^{-1}, yielding 4057 reflections, of which 1885 were unique. Background corrections were made (Blessing, Coppens & Becker, 1974). Three standard reflections observed after every 60 reflections showed long-range fluctuations with a maximum variation of 7%; this was due to changes in incident-beam intensity and counter response. This effect was corrected by rescaling the reflections with respect to the standard reflections. An absorption correction was applied; the transmission range was from 0·328 to 0·635. Weights were assigned according to $w(I) = [\sigma^2(I)_{\rm counting} + (0{\cdot}03I)^2]^{-1}$. The equivalent reflections were weight-averaged. 1876 of the resulting reflections had $I > 0$ and were used for the structure determination and refinement.

The positions of the Cd atoms were obtained by the Patterson method. Subsequent Fourier syntheses revealed the positions of the non-hydrogen atoms. They were refined with anisotropic thermal parameters. An isotropic extinction correction was made (Larson, 1969). At this stage anomalous-dispersion factors were applied to Cd and Cl (Cromer & Liberman, 1970). Refinement of the conformation presented in this work resulted in $R_w(F) = 0{\cdot}030$, while reversal of the polarity led to $R_w(F) = 0{\cdot}032$. Application of the R-factor ratio test (Hamilton, 1965) resulted in rejection of the second configuration with more than 99·5% probability. No bond lengths, however, changed more than 4σ by reversal of the polarity. A difference synthesis yielded the positions of 11 of the 12 H atoms. They were included in the refinement, but with their isotropic temperature factors fixed at $B = 4{\cdot}0$ Å^2. The final $R(F) = 0{\cdot}018$, $R_w(F) = 0{\cdot}022$ and $S = [\sum w(F_o - F_c)^2/(\mathrm{NO} - \mathrm{NV})]^{1/2} = 1{\cdot}67$. Scattering factors were from *International Tables for X-ray Crystallography* (1974), except for H (Stewart, Davidson & Simpson, 1965).

The calculations were carried out with the XRAY system (Stewart, Kruger, Ammon, Dickinson & Hall, 1972) on the Univac 1108 computer of the University of Frankfurt. The atomic coordinates are reported in Table 1, bond lengths and angles in Table 2.*

Table 1. *Positional parameters*

	x	y	z
Cd(1)	0·0	0·04993 (1)	0·0
Cd(2)	0·00496 (5)	0·13847 (1)	−0·50986 (6)
Cl(1)	−0·15938 (14)	0·04283 (5)	−0·66929 (20)
Cl(2)	0·15840 (14)	0·04287 (5)	0·66405 (19)
Cl(3)	−0·14266 (13)	0·13698 (5)	−0·17408 (18)
Cl(4)	0·17990 (13)	0·12554 (6)	0·18090 (16)
Cl(5)	−0·14628 (13)	0·22287 (6)	−0·66487 (16)
N	−0·0379 (4)	0·3769 (2)	−0·2059 (6)
C(1)	−0·0008 (6)	0·4326 (3)	−0·0887 (11)
C(2)	0·0033 (6)	0·3792 (3)	−0·4234 (8)
O(1)	0·0875 (3)	0·2723 (1)	−0·0121 (5)
O(2)	−0·3135 (3)	0·3021 (1)	−0·3375 (4)
H(1)	−0·058 (7)	0·435 (3)	0·040 (11)
H(2)	−0·042 (7)	0·462 (2)	−0·155 (10)
H(3)	0·073 (7)	0·445 (3)	−0·107 (9)
H(4)	−0·022 (7)	0·343 (3)	−0·508 (11)
H(5)	0·097 (7)	0·378 (2)	−0·445 (9)
H(6)	−0·071 (6)	0·405 (2)	−0·491 (8)
H(7)	0·015 (6)	0·360 (3)	−0·160 (9)
H(8)	−0·138 (7)	0·376 (2)	−0·204 (8)
H(9)	0·033 (6)	0·257 (3)	0·069 (8)
H(11)	−0·344 (6)	0·281 (3)	−0·260 (9)
H(12)	−0·286 (7)	0·283 (3)	−0·405 (9)

Discussion. The preparation of compounds with general formulae $(C_nH_{2n+1})_xNH_{4-x}M^{II}X_3$ and $[(C_nH_{2n+1})_xNH_{4-x}]_2M^{II}X_4$ with $n = 1, 2, 3, 4$; $x = 1, 2, 3, 4$; X = Cl, Br, I and M^{II} = Cd, Mn, Cu, Pd has been studied by Daoud (1976). Solvents used were methanol, ethanol, water or mixtures of alcohol and water. The relative stability of the tri- and tetrahalides was described. In the special case of salts between $(CH_3)_2NH_2Cl$ and $CdCl_2$ the trichlorides are more stable than the tetrachlorides. It seems that the trichloride is

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Table 2. *Bond lengths* (Å) *and angles* (°)

Cd(1)–Cl(1)^b	2·613 (1)	C(1)–H(1)	1·00 (7)
Cd(1)–Cl(1)^d	2·705 (1)	C(1)–H(2)	0·85 (6)
Cd(1)–Cl(2)^c	2·636 (1)	C(1)–H(3)	0·73 (6)
Cd(1)–Cl(2)^e	2·688 (1)	C(2)–H(4)	0·98 (6)
Cd(1)–Cl(3)^a	2·548 (1)	C(2)–H(5)	0·86 (6)
Cd(1)–Cl(4)^a	2·588 (1)	C(2)–H(6)	0·97 (5)
Cd(2)–Cl(1)^a	2·751 (1)	N–H(7)	0·68 (6)
Cd(2)–Cl(2)^c	2·736 (1)	N–H(8)	0·91 (6)
Cd(2)–Cl(3)^a	2·579 (1)	O(1)–H(9)	0·80 (5)
Cd(2)–Cl(4)^c	2·593 (1)	O(2)–H(11)	0·74 (6)
Cd(2)–Cl(5)^a	2·494 (1)	O(2)–H(12)	0·66 (6)
Cd(2)–O(2)^f	2·364 (3)		
N–C(1)	1·467 (7)		
N–C(2)	1·472 (7)		

Cl(1)^b–Cd(1)–Cl(1)^d	89·86 (4)	Cl(1)^a–Cd(2)–Cl(2)^c	81·69 (4)
Cl(1)^b–Cd(1)–Cl(2)^c	173·27 (3)	Cl(1)^a–Cd(2)–Cl(3)^a	91·71 (4)
Cl(1)^b–Cd(1)–Cl(2)^e	85·58 (4)	Cl(1)^a–Cd(2)–Cl(4)^c	87·45 (4)
Cl(1)^b–Cd(1)–Cl(3)^a	97·53 (4)	Cl(1)^a–Cd(2)–Cl(5)^a	96·20 (4)
Cl(1)^b–Cd(1)–Cl(4)^a	90·57 (4)	Cl(1)^a–Cd(2)–O(2)^f	164·07 (8)
Cl(1)^d–Cd(1)–Cl(2)^c	84·76 (4)	Cl(2)^c–Cd(2)–Cl(3)^a	84·41 (4)
Cl(1)^d–Cd(1)–Cl(2)^e	83·42 (4)	Cl(2)^c–Cd(2)–Cl(4)^c	85·99 (4)
Cl(1)^d–Cd(1)–Cl(3)^a	95·94 (4)	Cl(2)^c–Cd(2)–Cl(5)^a	177·22 (4)
Cl(1)^d–Cd(1)–Cl(4)^a	171·21 (4)	Cl(2)^c–Cd(2)–O(2)^f	82·43 (8)
Cl(2)^c–Cd(1)–Cl(2)^e	89·77 (4)	Cl(3)^a–Cd(2)–Cl(4)^c	170·38 (4)
Cl(2)^c–Cd(1)–Cl(3)^a	87·08 (4)	Cl(3)^a–Cd(2)–Cl(5)^a	93·87 (4)
Cl(2)^c–Cd(1)–Cl(4)^a	94·13 (4)	Cl(3)^a–Cd(2)–O(2)^f	88·01 (8)
Cl(2)^e–Cd(1)–Cl(3)^a	176·83 (4)	Cl(4)^c–Cd(2)–Cl(5)^a	95·74 (4)
Cl(2)^e–Cd(1)–Cl(4)^a	87·85 (4)	Cl(4)^c–Cd(2)–O(2)^f	90·18 (8)
Cl(3)^a–Cd(1)–Cl(4)^a	92·71 (4)	Cl(5)^a–Cd(2)–O(2)^f	99·72 (8)
C(1)–N–C(2)	114·6 (4)	C(2)–N–H(8)	106 (3)
C(1)–N–H(7)	94 (5)	H(7)–N–H(8)	133 (6)
C(1)–N–H(8)	104 (3)	H(11)–O(2)–H(12)	102 (7)
C(2)–N–H(7)	105 (5)		

Symmetry code

(*a*)	x,y,z	(*d*)	$x, -y, z + \frac{1}{2}$
(*b*)	$x, y, z + 1$	(*e*)	$x, -y, z - \frac{1}{2}$
(*c*)	$x, y, z - 1$	(*f*)	$x + \frac{1}{2}, -y + \frac{1}{2}, z$

obtained in solutions containing an excess (2:1) of $(CH_3)_2NH_2Cl$ over $CdCl_2$. Stoichiometric quantities and aqueous solution give the title compound.

A whole range of ammonium cadmium salts exists. The structure of the unmethylated salt NH_4CdCl_3 consists of infinite chains of nearly regular $CdCl_6$ octahedra, with two edges shared by two adjacent octahedra (Rolies & de Ranter, 1978). The chains are linked by Cl bridges in pairs.

The structures of $(CH_3)_4NCdCl_3$ (Morosin, 1972) and $(CH_3)_3NHCdCl_3$ (Walter, Brinkmann, Chapuis & Arend, 1979) are described as infinite chains of face-sharing $CdCl_6$ octahedra. The methylammonium groups are located in the free space between the chains and link the chains by hydrogen bonds to form two-dimensional sheets.

To investigate whether the title compound contains the familiar one-dimensional chain structure, the crystal structure determination was undertaken. A view down **a** of the structure is shown in Fig. 1.

The two independent Cd atoms both have a distorted octahedral coordination. Cd(1) is bonded to six Cl atoms, Cd(2) to five Cl atoms and one hydrate O atom. The Cl—Cd—Cl and Cl—Cd—O angles range from 81·69 (4) to 99·72 (8)°. The octahedra form two infinite zigzag chains in the direction of **c**, and they are linked by a common edge between adjacent octahedra. O(2) and Cl(5) belong only to one octahedron.

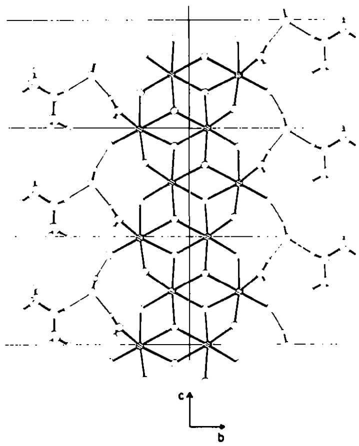

Fig. 1. View of the structure down **a**.

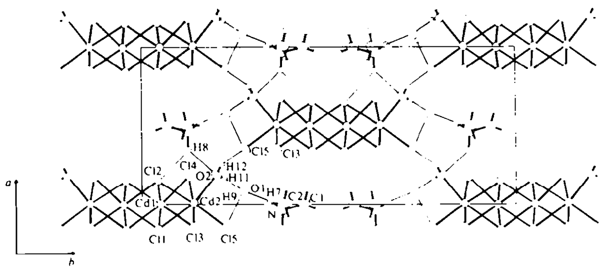

Fig. 2. View of the structure down **c**.

The dimethylammonium ion and water molecules are involved in hydrogen bonding. The hydrogen-bond system consists of medium to weak bonds. It is reported in Table 3 and shown in Fig. 2. This system forms two-dimensional sheets in the *ab* plane by linking the $CdCl_6$ chains together.

Only one H atom belonging to O(1) could be located in the difference syntheses. Investigation of the molecular packing, however, revealed two additional contacts between O(1) and Cl atoms, which may correspond to hydrogen bonds. They are represented in Fig. 2 as broken lines and are: O(1)···Cl(5) $(x+\frac{1}{2}, -y+\frac{1}{2}, z+1)$ 3·293 (3) and O(1)···Cl(3) $(x+\frac{1}{2}, -y+\frac{1}{2}, z)$ 3·318 (3) Å. Consequently, the H atoms of O(1) may be poorly located.

The dimethylammonium ion has a geometry similar to that in dimethylammonium manganese chloride (Caputo & Willett, 1976).

The Cd···Cd separations of adjacent octahedra are 3·732 (1), 3·844 (1) and 3·918 (1) Å; these are considerably longer than the values of about 3·36 Å in the linear chain structures $(CH_3)_4NCdCl_3$ (Morosin, 1972) and $(CH_3)_3NHCdCl_3$ (Walter, Brinkmann, Chapuis & Arend, 1979). Cl(1) and Cl(2) are both bonded to three Cd atoms. They are involved in Cd—Cl bonds (mean 2·688 Å) which are considerably longer than those involving Cl(3) and Cl(4), both of which are bonded to two Cd atoms (mean 2·577 Å). The terminal Cl(5) is involved in the shortest Cd—Cl bond of 2·494 (1) Å.

Table 3. *Hydrogen bonds*

D—H···*A*	*D*···*A* (Å)	H···*A* (Å)	∠*D*—H···*A* (°)
N—H(7)···O(1)[a]	2·831 (5)	2·22 (6)	150 (6)
N—H(8)···O(2)[a]	3·089 (5)	2·40 (5)	132 (4)
N—H(8)···Cl(2)[b]	3·354 (4)	2·68 (5)	131 (4)
O(1)—H(9)···Cl(5)[c]	3·295 (3)	2·50 (5)	173 (5)
O(2)—H(11)···O(1)[d]	2·821 (4)	2·09 (6)	172 (6)
O(2)—H(12)···Cl(5)[a]	3·143 (3)	2·49 (6)	170 (7)

Symmetry code

(*a*) x,y,z (*c*) $x,y,z+1$
(*b*) $x-\frac{1}{2},-y+\frac{1}{2},z-1$ (*d*) $x-\frac{1}{2},-y+\frac{1}{2},z$

This work was supported by the Bundesministerium für Forschung und Technologie. Thanks are due to Dr M. Müllner for kind permission to use his equipment.

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Acta Cryst. (1979). B**35**, 1709–1711

Structure of Bis[bis(η-cyclopentadienyl)tantalum(V)bis(μ-methanethiolato)]-platinum(0) Hexafluorophosphate

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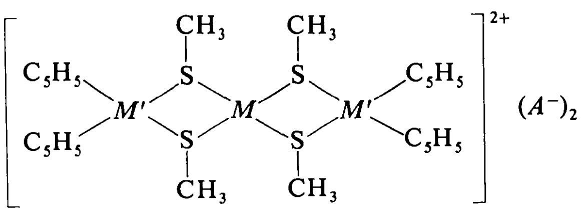
(*Received* 10 *February* 1979; *accepted* 19 *March* 1979)

Abstract. $[(\eta\text{-}C_5H_5)_2Ta(\mu\text{-}SCH_3)_2Pt(\mu\text{-}SCH_3)_2Ta(\eta\text{-}C_5H_5)_2](PF_6)_2$, $C_{24}H_{32}PtS_4Ta_2^{2+}.2PF_6^-$, $M_r = 1295$, monoclinic, $P2_1/c$, $a = 16.44$ (1), $b = 12.65$ (1), $c = 17.94$ (1) Å, $\beta = 97.8$ (1)°, $U = 3699$ Å^3, $Z = 4$, $D_c = 2.32$ Mg m^{-3}, Mo $K\alpha$ ($\lambda = 0.71069$ Å), $\mu = 4.47$ mm^{-1}. The complex formally contains Ta^{V} and Pt^{0}. The Pt atom has a tetrahedral coordination and the short Pt–Ta contacts (2·788 and 2·809 Å) correspond to metal–metal bonds.

Introduction. The complex $Ta(\eta\text{-}C_5H_5)_2(SCH_3)_2$ reacts with $Pt(C_6H_5CN)_2Cl_2$ to give a diamagnetic trinuclear complex (I) isolated as the hexafluorophosphate salt (Siganporia, 1977).



(I*a*) M = Pt, M' = Ta, A = PF_6

(I*b*) M = Ni, M' = Nb, A = BF_4

(I*c*) M = Ni, M' = Mo, A = BF_4

The electronic structure and the formal valence of the metals in these trinuclear complexes depend on the nature of the metals (Douglas & Green, 1972; Prout, Critchley & Rees, 1974). For (I*b*) the short Ni–Nb distance (2·78 Å) corresponds to a metal–metal bond, in contrast to the Ni–Mo non-bonded contact in (I*c*) which is much longer (3·39 Å). Moreover, the coordination of the Ni atom is tetrahedral in (I*b*) and square-planar in (I*c*). Complex (I*c*) has been described as a d^2–d^8–d^2 system, but for (I*b*) the favoured system was considered to be d^0–d^{10}–d^0. A d^1–d^8–d^1 system could also be proposed for (I*b*) with Ni^{II} in tetrahedral coordination (for a complete discussion, see Prout, Critchley & Rees, 1974). The determination of the structure of (I*a*) could clarify the situation, because tetrahedral coordination for Pt^{II} is unknown, and all attempts to make it have resulted in the stepped square-planar configuration found in (I*c*).

The crystals were supplied by Dr M. L. H. Green and Mr N. Siganporia. A small red-orange crystal (approximately 0·2 × 0·2 × 0·4 mm) was mounted on a Nonius CAD-4F diffractometer, and Mo $K\alpha$ radiation from a graphite monochromator was used. Cell dimensions and the orientation matrix were obtained by least squares from the setting angles of 25 reflexions. The intensities of reflexions, $\theta < 20°$, were measured by an $\omega/2\theta$ scan, with a variable scan rate and an ω-scan angle of (1·80 + 0·35 tan θ)°. Lorentz and polarization corrections were applied to the 1228 reflexions with $I > 3\sigma(I)$ which were used in subsequent calculations. No corrections were made for absorption. The structure was solved by Patterson and Fourier techniques and refined by least squares with a large-block approximation. Because the locations of the

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