Inorganica Chimica Acta, 51 (1981) 177–173
©Elsevier Sequoia S.A., Lausanne – Printed in Switzerland

The Crystal and Molecular Structure of an Isomer of Chloro [1,9-bis(2-pyridyl)-2,5,8-triazanonane] cobalt(III), Tetrachlorozincate(II), [Co(*picdien*)Cl] $ZnCl_4$

G. BOMBIERI*, E. FORSELLINI
Istituto di Chimica e Tecnologia dei Radioelementi del C.N.R., Padua, Italy

A. DEL PRA
Istituto Chimico Farmaceutico, Universtià di Milano, Viale Abruzzi 42, 20131 Milan, Italy

and M. L. TOBE
University College, London, U.K.

Received February 19, 1981

The structure of the title compound has been determined by counter data. The crystals are orthorhombic, space group Pbca, *with* a = *16.363(7),* b = *18.382(8),* c = *14.940(6)* Å, Z = *8. The structure has been defined by full-matrix least-squares to a final conventional* R *value of 0.092 (*Rw = 0.081). *The coordination geometry of the complex cation is near regular octahedral, with the pentadentate* picdien *ligand present in the α, β configuration and with a Cl* trans *to an 'angular' secondary nitrogen. Only two,* i.e., *one 'angular' and the 'flat' of the three secondary nitrogen atoms of the* picdien *ligand exhibit chirality and it is opposite. The absolute configuration of the complex can be designed as Δ-α, β-R (or Λ-α, β-S). The* $ZnCl_4$ *anion assumes a distorted tetrahedral configuration.*

Introduction

Acido-pentamine complexes of cobalt(III) with the linear pentadentate ligands, 1,9-bis(2-pyridyl)-2,5,8-triazanonane(*picdien*) and 1,11-bis(2-pyridyl)-2,6,10-triazaundecane(*picditn*) are of considerable importance in our continuing study of the factors that determine the sensitivity of cobalt(III) amine complexes to base hydrolysis [1, 2]. In particular, they allow us to examine the role of coordinated pyridine or substituted pyridines. The only isomeric form of the [Co(*picdien*)Cl]$^{2+}$ cation so far characterised is extremely sensitive to base hydrolysis, indeed the acid independent rate constant has still to be measured with any reliability since plots of k_{obs} *vs.* $[H^+]^{-1}$ ($0.1 > [H^+] > 0.001$) are linear and pass through the origin (within experimental error) [3]. The only known isomer of [Co(*picditn*)Cl]$^{2+}$, whose structure has been reported elsewhere [4], follows a similar dependence on $[H^+]^{-1}$ but the intercept is quite large [3]. We have now been able to prepare a number of complexes, some in more than one isomeric form, belonging to the series [Co(*picdien*)X]$^{n+}$ and these are of considerable interest both with respect to their reactivity [3], and to their high resolution ^{1}H NMR spectra [5]. The results of these investigations will be published elsewhere.

Experimental

Preparation

[Co(*picdien*)Cl] $(ClO_4)_2$, prepared by the method of Henderson [2], was converted to the tetrachlorozincate by adding a filtered solution of $ZnCl_2$ (15 g) in HCl (10 cm^3 12 *M*) to a filtered hot (60–70 °C), saturated solution in 2 *M* HCl (50–100 cm^3). Well formed crystals separated after 30 minutes if the solution was set aside to crystallise undisturbed.

X-ray Structure Analysis

Accurate unit cell dimensions and crystal orientation matrices, together with their estimated standard errors, were obtained from least-squares refinement of the $2\theta, \omega, \chi$ and ϕ values of 20 carefully centered high angle reflexions. Intensity data were collected from a crystal of approximate dimensions 0.08 × 0.12 × 0.10 mm on a Philips PW 1100 four-circle diffractometer operating in the $\theta/2\theta$ scan mode (scan width = 1.2°, scan speed = 0.02° s^{-1}) and by using graphite monochromatized Mo$K\bar{\alpha}$ radiation. 1764 independent reflexions up to θ = 22° were measured, of which 1322 had $I \geqslant 2\sigma(I)$, $\sigma(I)$ being calculated from counting statistics. During the data collection two standard reflexions were measured every 150 min to check the stability of the crystal and the electronics. Intensities were corrected for Lorentz and polarization effects and put in an abso-

---

*Present address: Istituto di Chimica Generale, Università di Messina, Messina, Italy

TABLE I. Atomic Coordinates ($\times 10^4$, for hydrogen atoms $\times 10^3$) with e.s.d.s in Parentheses and Anisotropic Thermal Parameters ($\times 10^3$) with e.s.d.s in Parentheses.

	x	y	z		x	y	z
Zn	815(2)	7850(2)	3571(2)				
Cl(2)	894(5)	9233(4)	3438(5)				
Cl(3)	1778(4)	7557(4)	4518(5)				
Cl(4)	963(4)	7300(4)	2206(4)				
Cl(5)	−202(4)	7485(5)	4259(5)				
Co	2372(2)	−95(2)	5736(2)				
Cl(1)	2068(4)	730(4)	6874(5)				
N(1)	2032(10)	−1016(12)	6467(14)				
C(1)	2424(14)	−1525(13)	6949(15)	H(1)	293	−143	698
C(2)	2125(13)	−2177(15)	7397(18)	H(2)	243	−252	775
C(3)	1385(16)	−2312(19)	7352(20)	H(3)	116	−277	762
C(4)	990(15)	−1758(15)	6870(16)	H(4)	48	−181	684
C(5)	1316(14)	−1130(16)	6425(17)				
C(6)	931(14)	−493(16)	5947(17)	H(61)	53	−73	565
				H(62)	76	−10	636
N(2)	1393(9)	−84(12)	5278(13)	H(22)	133	−38	473
C(7)	1201(15)	780(17)	5059(20)	H(71)	77	80	471
				H(72)	114	109	559
C(8)	1834(14)	1100(18)	4540(19)	H(81)	182	168	452
				H(82)	184	88	395
N(3)	2524(11)	834(11)	4926(13)	H(33)	271	130	523
C(9)	3071(14)	594(16)	4254(18)	H(91)	306	96	376
				H(92)	354	61	452
C(10)	2896(14)	−292(14)	3944(17)	H(101)	331	−52	365
				H(102)	249	−29	355
N(4)	2732(10)	−793(11)	4757(12)	H(44)	237	−117	457
C(11)	3349(14)	−1235(16)	5082(18)	H(111)	319	−170	541
				H(112)	364	−141	460
N(5)	3396(10)	−97(11)	6079(12)				
C(12)	3782(13)	−681(15)	5698(16)				
C(13)	4526(16)	−810(18)	5819(19)	H(13)	479	−125	556
C(14)	4850(14)	−212(14)	6340(17)	H(14)	536	−24	642
C(15)	4492(14)	409(15)	6716(16)	H(15)	474	79	708
C(16)	3744(13)	466(14)	6566(15)	H(16)	348	91	681

Anisotropic and isotropic thermal parameters ($\times 10^3$) with e.s.d.s in parentheses

	U_{11} or U	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Zn	37(2)	31(2)	34(2)	−5(2)	−6(2)	1(2)
Cl(2)	95(6)	32(4)	47(5)	−13(5)	−18(5)	4(4)
Cl(3)	66(5)	32(4)	58(4)	2(4)	−32(4)	6(4)
Cl(4)	53(5)	49(5)	34(4)	−14(4)	2(4)	−2(4)
Cl(5)	51(6)	72(5)	73(6)	−5(5)	26(5)	16(5)
Co	36(2)	23(2)	27(2)	1(2)	−4(2)	−3(2)
Cl(1)	67(5)	32(4)	37(4)	9(4)	−3(4)	−8(4)
N(1)	34(5)					
C(1)	28(6)					
C(2)	42(7)					
C(3)	67(9)					
C(4)	44(8)					
C(5)	37(7)					
C(6)	53(8)					
N(2)	31(5)					
C(7)	61(9)					
C(8)	61(9)					
N(3)	34(5)					
C(9)	45(8)					
C(10)	40(8)					
N(4)	30(6)					
C(11)	41(8)					
N(5)	22(5)					

(continued on facing page)

TABLE I. *(continued)*

	U_{11} or U	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(12)	30(7)					
C(13)	55(9)					
C(14)	40(7)					
C(15)	35(7)					
C(16)	29(7)					

TABLE II. Interatomic Distances (Å) and Angles (°) with Their e.s.d.s in Parentheses.

Co–Cl(1)	2.242(8)	N(3)–C(8)	1.46(3)
Co–N(1)	1.96(2)	N(3)–C(9)	1.47(3)
Co–N(2)	1.92(2)	C(9)–C(10)	1.56(4)
Co–N(3)	1.96(2)	N(4)–C(10)	1.50(3)
Co–N(4)	1.97(2)	N(4)–C(11)	1.43(3)
Co–N(5)	1.95(2)	C(11)–C(12)	1.52(4)
N(1)–C(1)	1.32(3)	C(12)–C(13)	1.40(4)
N(1)–C(5)	1.33(3)	C(13)–C(14)	1.38(4)
C(1)–C(2)	1.37(3)	C(14)–C(15)	1.33(3)
C(2)–C(3)	1.38(4)	C(15)–C(16)	1.40(4)
C(3)–C(4)	1.37(4)	N(5)–C(12)	1.32(3)
C(4)–C(5)	1.36(4)	N(5)–C(16)	1.34(3)
C(5)–C(6)	1.45(4)	Zn–Cl(2)	2.276(7)
N(2)–C(6)	1.47(3)	Zn–Cl(3)	2.316(8)
N(2)–C(7)	1.49(3)	Zn–Cl(4)	2.246(7)
C(7)–C(8)	1.49(4)	Zn–Cl(5)	2.215(8)
N(1)–Co–N(2)	84.7(8)	C(7)–N(2)–Co	108(1)
N(2)–Co–N(3)	84.6(8)	C(6)–N(2)–C(7)	116(2)
N(3)–Co–N(5)	91.5(8)	N(2)–C(7)–C(8)	105(2)
N(5)–Co–N(1)	99.2(8)	C(7)–C(8)–N(3)	112(2)
Cl(1)–Co–N(1)	87.8(6)	C(8)–N(3)–Co	111(2)
Cl(1)–Co–N(2)	91.8(6)	C(9)–N(3)–Co	108(1)
Cl(1)–Co–N(3)	92.1(6)	C(8)–N(3)–C(9)	114(2)
Cl(1)–Co–N(5)	92.4(6)	N(3)–C(9)–C(10)	108(2)
N(4)–Co–N(1)	94.3(8)	C(9)–C(10)–N(4)	108(2)
N(4)–Co–N(2)	93.2(8)	C(10)–N(4)–Co	111(1)
N(4)–Co–N(3)	86.8(8)	C(11)–N(4)–Co	108(1)
N(4)–Co–N(5)	82.5(8)	C(10)–N(4)–C(11)	113(2)
Co–N(1)–C(1)	128(2)	N(4)–C(11)–C(12)	109(2)
Co–N(1)–C(5)	113(2)	C(11)–C(12)–N(5)	114(2)
C(1)–N(1)–C(5)	119(2)	C(13)–C(12)–N(5)	126(2)
N(1)–C(1)–C(2)	123(2)	C(11)–C(12)–C(13)	120(2)
C(1)–C(2)–C(3)	120(2)	C(12)–C(13)–C(14)	113(2)
C(2)–C(3)–C(4)	116(3)	C(13)–C(14)–C(15)	124(2)
C(3)–C(4)–C(5)	122(3)	C(14)–C(15)–C(16)	118(2)
C(4)–C(5)–N(1)	121(2)	C(15)–C(16)–N(5)	121(2)
N(1)–C(5)–C(6)	114(2)	C(12)–N(5)–Co	114(2)
C(4)–C(5)–C(6)	125(2)	C(16)–N(5)–Co	127(2)
C(5)–C(6)–N(2)	112(2)	C(12)–N(5)–C(16)	118(2)
C(6)–N(2)–Co	107(1)		
Cl(2)–Zn–Cl(3)	102.1(3)	Cl(3)–Zn–Cl(4)	112.3(3)
Cl(2)–Zn–Cl(4)	108.1(3)	Cl(3)–Zn–Cl(5)	107.8(3)
Cl(2)–Zn–Cl(5)	111.2(3)	Cl(4)–Zn–Cl(5)	114.6(3)

TABLE III. Least-squares Planes with Deviations (Å) of the Relevant Atoms in Square Brackets. The equation of a plane in the direct space is given by PX + QY + RZ = S and angles (°) between the planes and between line and planes.

	P	Q	R	S
Plane 1 N(1), C(1), C(2), C(3), C(4), C(5) [N(1) −0.007, C(1) 0.008, C(2) 0.002, C(3) −0.013, C(4) 0.015, C(5) −0.005]	−2.24220	9.41750	12.08091	6.40733
Plane 2 N(5), C(12), C(13), C(14), C(15), C(16) [N(5) 0.022, C(12) −0.025, C(13) 0.013, C(14) −0.000, C(15) −0.002, C(16) −0.008]	−3.05343	−9.14242	12.13946	6.40948
Plane 3 Co, N(1), N(2), N(3), N(5) [Co 0.027, N(1) 0.027, N(2) −0.044, N(3) 0.029, N(5) −0.039]	−4.31453	10.34829	11.02885	5.17750
Plane 4 Co, N(2), Cl(1), N(5), N(4) [Co −0.035, N(2) 0.026, Cl(1) −0.007, N(5) 0.028, N(4) −0.010]	3.44433	13.18601	−8.39181	−4.08637
Plane 5 Co, Cl(1), N(1), N(4), N(3) [Co 0.037, Cl(1) 0.119, N(1) −0.144, N(4) 0.135, N(3) −0.147]	17.14899	−0.10102	5.37875	7.11701
Plane 6 Co, N(1), N(2) [C(5) 0.190, C(6) 0.526]	−4.94020	10.40364	10.80987	4.92989
Plane 7 Co, N(2), N(3) [C(7) 0.747, C(8) 0.197]	−4.97463	10.30111	10.88105	4.96353
Plane 8 Co, N(3), N(4) [C(9) 0.554, C(10) −0.052]	17.16452	1.66181	5.12692	6.99644
Plane 9 Co, N(4), N(5) [C(11) −0.685, C(12) −0.345]	2.84621	13.27738	−8.41975	−4.28059
Line (a) Co, Cl(1)				

Angles (°) between the planes and between line and planes

1–2	69.2	1–a	8.0
3–1	8.3	2–a	71.3
3–2	73.4		

lute scale by Wilson's method. An experimental absorption correction was applied [6].

Crystal Data

$C_{16}H_{23}N_5Cl_5CoZn$, M = 588.9. Orthorhombic, space group *Pbca*, a = 16.363(7), b = 18.382(8), c = 14.940(6) Å, U = 4493.7 Å^3, $F(000)$ = 2368 e, D_o = 1.73, D_c = 1.74 g cm^{-3} for Z = 8, MoK$\bar{\alpha}$ radiation, λ = 0.71069 Å, μ(MoK$\bar{\alpha}$) = 23.5 cm^{-1}.

Structure Solution and Refinement

The molecular structure of the complex was solved by conventional Patterson and Fourier syntheses. The refinement of the structural model, which was by the method of full-matrix least-squares was car-

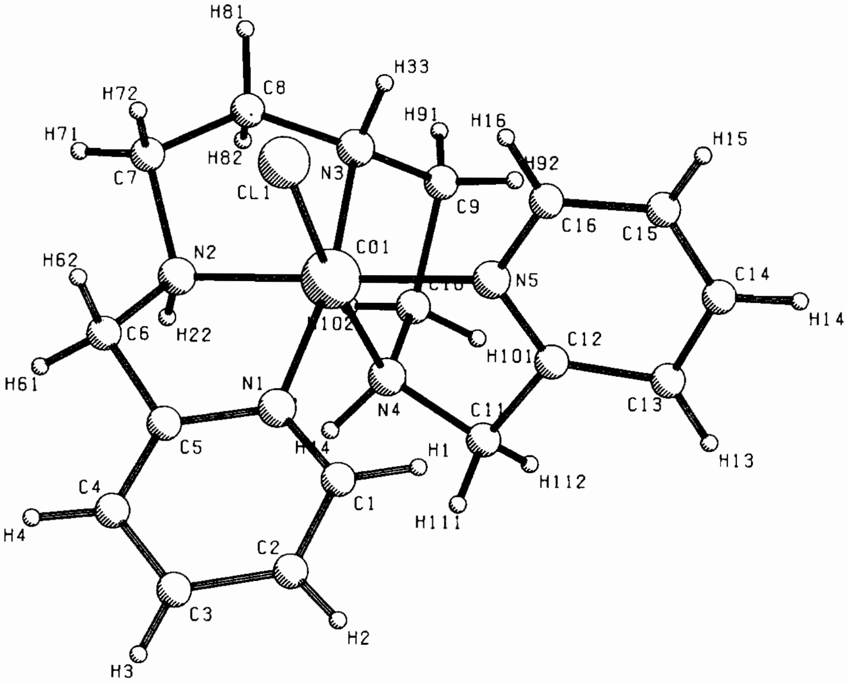

Fig. 1. View of the complex cation down *c*.

ried out allowing all non-hydrogen atoms to vibrate anisotropically. Hydrogen atoms could not be located from electron density difference maps and therefore were included in the scattering model in calculated idealized positions (C–H = 0.95 Å), but not varied.

The function minimized was $\Sigma w\Delta^2$, in which $w = 3.56[\sigma^2(Fo) + 0.005\ Fo^2]^{-1}$ and $\Delta = |Fo| - |Fc|$. Weighting scheme analyses showed no serious dependence of the mean $w\Delta^2$ on either $|Fo|$ and $\lambda^{-1} \sin\theta$. The final electron density difference map showed no unusual features. Atomic scattering factors were taken from reference 7. Allowance was made for the anomalous scattering of zinc, cobalt and chlorine atoms, using values of $\Delta f'$ and $\Delta f''$ from reference 7. The final conventional *R* value for the 1322 observed reflexions with $I > 2\sigma(I)$ was 0.092 (*R*w = 0.081). Final atomic parameters and a selection of functions derived from them are presented in Tables I–VI. All calculations were carried out on the CYBER 76 computer of the 'C.I.N.E.C.A.' with the SHELX-76 program for crystal structure determination [8].

Results and Discussion

The stereochemical arrangement of the complex cation is shown in Fig. 1, along with the atom nomenclature.

The coordination geometry of the cation is near regular octahedral with the pentadentate *picdien* ligand present in the α,β configuration [9]. Two secondary nitrogens and the two pyridine nitrogen atoms of the *picdien* ligand are found to occupy one plane. The chlorine and the remaining secondary nitrogen of *picdien* complete the coordination about the cobalt atom. Only two, *i.e.*, N(2) and N(4) of the three secondary nitrogen atoms of the *picdien* ligand exhibit chirality; whereas the configuration of the 'flat' nitrogen N(2) is R that of the 'angular' N(4) is S [owing to the space group *Pbca* requirements N(2) can be also S, in which case N(4) is R]. Therefore the absolute configuration of the complex can be designated as Δ-α, β-R or Λ-α, β-S [10].

In this isomer the N(2)–H bond and the Co–Cl bond lie on opposite sides of the Co, N(1), N(2), N(3), N(5) plane and we propose to label this the *'anti'* isomer, as distinct from the, as yet, missing *'syn'* isomer (Δ-α, β-S or Λ-α, β-R). An *'anti'* isomer has already been found in $[\alpha\beta\text{-Co}(\textit{picditn})\text{Cl}\text{CoCl}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$ [4]. *'Syn'* isomers, on the contrary, have been found in the dioxygen bridged dimeric cobalt complexes with *picdien* and *picditn* $[\text{LCoO}_2\text{CoL}]^{++}$ which also have the $\alpha\beta$ configuration [11, 12] and also the less soluble isomer of $\alpha\beta[\text{Co}(\textit{picdien})\text{Br}](\text{ClO}_4)_2$ [13].

Inspection of the Co–N bond distances in the *picdien*-cobalt moiety shows that the bonds to the terminal trigonal nitrogen atoms [Co–N(1) = 1.96(2), Co–N(5) = 1.95(2) Å] are slightly longer than

TABLE IV. Helicity of the Conformation for all Chelate Rings.

Ring	Conformation	Helicity
N(1)–Co–N(2)	envelope	δ
N(2)–Co–N(3)	envelope	λ
N(3)–Co–N(4)	envelope	λ
N(4)–Co–N(5)	envelope	δ

(predicted from radii-sum rules, 1.92 Å, [14], although similar values have been already found in crystal structures of other Co(III) complexes with linear pentadentate *picdien* and *picditn* ligands [4, 11]. The bonds to the 'flat' and 'angular' secondary nitrogens [Co–N(2) = 1.92(2), Co–N(3) = 1.96(2), Co–N(4) = 1.97(2) Å] lie in the middle of the range of observed Co–N (tetrahedral) lengths (1.89 to 2.06 Å) [4, 11, 12, 15], the bond to the 'flat' secondary nitrogen N[2] being slightly shorter than the other Co–N bonds, as has been found in the structures of a number of cobalt(III) complexes containing *trien,* coordinated in the *β* configuration [16]. Another geometrical features of the 'flat' secondary nitrogen N(2) atom, common to all the structures examined [16], is the expansion of the C(6)–N(2)–C(7) bond angle [116(2)°]. The Co–N(2) bond length is reduced relative to the other Co–N bond distances in order to reduce strain at this centre. By comparison there is less distortion in the angles about the angular secondary N(3) and N(4) atoms. Likewise there are no other significant intra-ligand angular distortions in the polyamine skeleton. In fact, within the *picdien* chelate all internal angles at C and N, are close to the regular tetrahedral value. The angles subtended by the chelate rings at the metal ion are similar and compare well with the values found in analogous compounds [4, 16]. All C–C and C–N distances are reasonably close to their usual values [14].

The conformations and the helicities [10] adopted by the chelate rings are reported in Table IV. The torsion angles about the non-rigid bonds of the *picdien* ligand are presented in Table V.

The two pyridine rings in this complex are planar within experimental error and make an angle of 69.2° each other.

TABLE V. Torsion Angles (°).[a]

N(1)–C(5)–C(6)–N(2)	27
C(7)–N(2)–C(6)–C(5)	−153
C(6)–N(2)–C(7)–C(8)	168
N(2)–C(7)–C(8)–N(3)	−42
C(9)–N(3)–C(8)–C(7)	139
C(8)–N(3)–C(9)–C(10)	−80
N(3)–C(9)–C(10)–N(4)	−45
C(11)–N(4)–C(10)–C(9)	−96
C(10)–N(4)–C(11)–C(12)	86
N(4)–C(11)–C(12)–N(5)	26

[a]The torsion angles w(IJKL) is defined as the angle between the vector KL when viewed down JK. It is positive if clockwise and negative if counter-clockwise.

TABLE VI. Possible Hydrogen-bonding Interactions and Key for Symmetry.

Cl(2)···N(2)^{I}	3.11	Cl(2)···H(22)^{I}	2.19
Cl(3)···N(4)^{I}	3.24	Cl(3)···H(44)^{I}	2.35
C(7)···Cl(5)^{II}	3.53	H(72)···Cl(5)^{II}	2.90
Cl(3)···N(3)^{III}	3.21	Cl(3)···H(33)^{III}	2.30
Cl(4)···C(15)^{III}	3.59	Cl(4)···H(15)^{III}	2.98
Cl(4)···C(16)^{III}	3.56	Cl(4)···H(16)^{III}	2.89
Cl(5)···C(13)^{IV}	3.63	Cl(5)···H(13)^{IV}	2.81
Cl(1)···C(9)^{V}	3.57	Cl(1)···H(91)^{V}	2.85
Cl(1)···C(10)^{V}	3.52	Cl(1)···H(102)^{V}	3.11
C(1)···Cl(4)^{VI}	3.55	H(1)···Cl(4)^{VI}	2.92
Cl(1)···C(2)^{VII}	3.60	Cl(1)···H(2)^{VII}	2.99

Key for symmetry

I	x, 1 + y, z	V	½ – x, y, ½ + z
II	–x, 1 – y, 1 – z	VI	½ – x, –1 + y, ½ + z
III	½ – x, ½ + y, 1 – z	VII	x, ½ + y, 3/2 – z
IV	–½ + x, ½– y, z		

The Co–Cl(1) bond distance, 2.242(8) Å, is slightly longer than the sum of appropriate covalent radii, 2.21 Å [14] but agrees with the Co–Cl bonds in several Co(III) complexes [4, 16]. In the tetrachlorozincate(II) anion a distorted tetrahedral arrangement of chlorine atoms about the central zinc atom is observed (Table II) with mean Zn–Cl bond distance of 2.263(7) Å. Comparable values in related compounds are 2.260(3) Å in [Co(*tren*)pyCl]$ZnCl_4$ [17], 2.26(3) and 2.27(1) Å in π and χ isomers of [Co(*en*)(*dien*)Cl]$ZnCl_4$, [18] and 2.264(17) Å in [β-Co(*en*)(*dpt*)Cl]$ZnCl_4$, [19].

The crystal cohesion is obtained mainly by N–H···Cl hydrogen bonds linking cations and anions as shown in Table VI where selected intermolecular contacts are reported.

Acknowledgement

We thank Mrs. M. Magnabosco for technical assistance.

References

1 R. A. Henderson and M. L. Tobe, *Inorg. Chem., 16,* 2576 (1977).

2 R. A. Henderson, *Ph.D. Thesis*, London (1976).
3 M. Humanes and M. L. Tobe, to be published.
4 G. Bombieri, E. Forsellini, A. Del Pra and M. L. Tobe, *Inorg. Chim. Acta, 40,* 71 (1980).
5 C. D. Cooksey and M. L. Tobe, to be published.
6 A. C. T. North, D. C. Phillips and F. S. Mathews, *Acta Cryst., A24,* 351 (1968).
7 International Tables for X-Ray Crystallography, Vol. IV, 3rd Ed., Kynoch Press, Birmingham (1974).
8 G. M. Sheldrick, 'SHELX-76 – Program for Crystal Structure Determination', Univ. of Cambridge (1976).
9 M. R. Snow, *J. Chem. Soc. Dalton,* 1627 (1972).
10 IUPAC, 'Nomenclature of Inorganica Chemistry', 2nd ed., pp. 75–83, Butterworths, London (1970).
11 J. H. Timmons, A. Clearfield, A. E. Martell and R. H. Niswander, *Inorg. Chem., 18,* 1042 (1979).
12 J. H. Timmons, R. H. Niswander, A. Clearfield and A. E. Martell, *Inorg. Chem., 18,* 2977 (1979).
13 G. Bombieri, E. Forsellini, A. Del Pra and M. L. Tobe, *Inorg. Chim. Acta,* submitted.
14 L. Pauling, 'The Nature of the Chemical Bond', 3rd Ed., Cornell University Press, Ithaca, New York (1966).
15 P. A. Tucker, *Acta Cryst., B35,* 67 (1979).
16 G. Bombieri, E. Forsellini, A. Del Pra, M. L. Tobe and R. Henderson, *Gazz. Chim. It., 109,* 207 (1979).
17 G. Bombieri, F. Benetollo, A. Del Pra, M. L. Tobe and D. A. House, *Inorg. Chim. Acta,* submitted.
18 A. R. Gainsford, D. A. House and W. T. Robinson, *Inorg. Chim. Acta, 5:4,* 595 (1971).
19 P. R. Ireland, D. A. House and W. T. Robinson, *Inorg. Chim. Acta, 4,* 137 (1970).