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Synthesis, characterization and crystal structure of $[(\eta^6\text{-}C_6Me_6)Ru(\mu\text{-}Cl)_3Ru(\eta^6\text{-}C_6Me_6)]PF_6$

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Abstract

Reaction of $[\{Ru(\eta^6\text{-}C_6Me_6)Cl_2\}_2]$ with silver trifluoroacetate in presence of ammonium hexafluorophosphate and organonitriles viz. 4-cyanopyridine, 1,4-piperazine dicarbonitrile or 1,4-dicyanobenzene in methanol gives trichloro bridged complex $[(\eta^6\text{-}C_6Me_6)\ Ru(\mu\text{-}Cl)_3Ru(\eta^6\text{-}C_6Me_6)]PF_6$. The complex has been characterized by various physico-chemical techniques (Elemental analyses, IR,UV–vis, ^1H-, ^{13}C-, ^{31}P-NMR and FAB mass spectroscopy) and single crystal X-ray diffraction studies. [Crystal data: monoclinic, space group $P2_1c$, $a = 10.7604(9)$, $b = 24.897(2)$, $c = 10.8833(11)$ Å, $\beta = 100.990(7)°$. © 1999 Elsevier Science S.A. All rights reserved.

Keywords: Ruthenium complexes; Trichloro bridged hexa methylbenzene complex

1. Introduction

Much attention has been paid towards the synthesis and characterization of transition metal complexes having good catalytic properties [1]. In this regard arene complexes of the general formula $[\{Ru(arene)Cl_2\}_2]$ (arene = benzene and its derivatives) have received special attention [2–15]. Corresponding hexamethylbenzene complexes have been studied only occasionally [11–17]. Arene di-μ-chloro complex $[\{Ru(arene)Cl_2\}]$ (arene = benzene, *p*-cymene) on reaction with water gives dinuclear complex $[Ru_2(\mu\text{-}Cl)_3(arene)_2]^+$ and its reaction with silver tetrafluoroborate in acetonitrile gives monomeric complex $[Ru(arene)(MeCN)_3]^{2+}$ [15–17]. Corresponding reactions of $[\{Ru(\eta^6\text{-}C_6Me_6)Cl_2\}_2]$ have not been studied so far. Because of our continuing interest in the reactivity of Ru(II) arene complexes [18,19], we thought it worthwhile, to make a detailed study of the reactivity of this molecule with silvertrifluroacetate in presence of an organonitrile viz. 4-cyanopyridine; 1,4-dicyanobenzene, 1,4-piperazine-dicarbonitrile or a simple alkyne like phenylacetylene, diphenylacetylene or propargyl alcohol. Such a reaction may give monomeric complexes with the general formula $[\{Ru(\eta^6\text{-}C_6Me_6)(L)_3\}]^{2+}$ or it may lead to the formation of trifluoroacetato bridged complexes. However, none of the above products could be isolated but, surprisingly we could isolate a complex with the formula $[(\eta^6\text{-}C_6Me_6)Ru(\mu\text{-}Cl)_3Ru(\eta^6\text{-}C_6Me_6)]PF_6$. In this short communication we report synthesis, characterization and single crystal X-ray structure of the complex $[(\eta^6\text{-}C_6Me_6)Ru(\mu\text{-}Cl)_3Ru(\eta^6\text{-}C_6Me_6)]PF_6$.

2. Experimental

2.1. Materials

Analar grade chemicals were used and solvents were purified prior to use by standard methods. $RuCl_3 \cdot xH_2O$ (Sigma), hexamethylbenzene, ammonium hexafluorophosphate and silvertrifluoroacetate (all Aldrich) were used as received. The complex $[\{Ru(\eta^6\text{-}C_6Me_6)Cl_2\}_2]$ was prepared by a literature method [20].

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Physicochemical measurements were made as described elsewhere [19]. FAB mass spectra was recorded on a JEOL SX-120A mass spectrometer. NBA was used as the matriz and matrix peaks appeared at m/z 136, 137 and 154.

2.2. Synthesis

2.2.1. Preparation of $[(\eta^6\text{-}C_6Me_6)Ru(\mu\text{-}Cl)_3Ru(\eta^6\text{-}C_6Me_6)]PF_6$

Silver trifluoracetate (145 mg, 0.65 mmol.) was added to a suspension of $[\{Ru(\eta^6\text{-}C_6Me_6)Cl_2\}_2]$ (200 mg, 0.30 mmol.) in methanol (15 ml) and the solution was stirred for about ~1.0 h at room temperature (r.t.). The resulting white precipitate was removed by filtraton. The yellow–orange filtrate was treated with 4-cyanopyridine (95 mg, 0.95 mmol) and the solution was stirred for about 5 h at r.t. It was filtered to remove any solid residue and NH_4PF_6 (50 mg, 0.30 mmol) dissolved in methanol (10 ml) was added to the filtrate and left for slow crystallization. Golden shiny crystals separated out, these were filtered, washed twice with methanol and diethyl ether and dried in vacuo. Yield, 65%; m.p. 238°C; Anal. Calc. for $C_{24}Cl_3F_6H_{36}PRu_2$: C, 37.00; H, 4.60; Found: C, 36.91; H, 4.93; IR(KBr) 265 cm^{-1} ν(Ru–Cl); ^{1}H-NMR (acetone-d_6); δ 2.16 ppm (s, η^6-$C_6(CH_3)_6$); $^{13}C\{^1H\}$-NMR (acetone-d_6) δ 15.09 (η^6-$C_6(CH_3)_6$), δ 95.94 ppm ($C_6CH_3)_6$); $^{31}P\{^1H\}$-NMR (acetone-d_6) δ −143 ppm (PF_6^-); FAB mass (NBA); calc. (m/z) 633; found (m/z) 633 (molecular ion peak).

The above reaction was repeated in the presence of 1,4-dicyanobenzene, 1,4-piperazinedicarbonitrile and propargyl alcohol. However, in all the cases the product obtained was the same as above.

2.2.2. X-ray structure of $[(\eta^6\text{-}C_6Me_6)Ru(\mu\text{-}Cl)_3Ru(\eta^6\text{-}C_6Me_6)]PF_6$

Diffraction data was collected with a Siemens P4/PC diffractometer from plate like red crystal of dimentions 0.36 × 0.34 × 0.04 mm in the ω scan mode (2θ range from 2.0 to 60.0°). Intensities were measured by the 2θ ω scan method using Mo–K_α radiation ($\lambda = 0.71073$ Å). A variable scan speed between 4.00 and 60.00° min^{-1} in ω was used. Intensities of three standard reflections were measured every 197 reflections as a check of stability of the crystal. A total of 9341 reflections ($2\theta < 60°$) were measured and out of these 3104 reflections with $F > 4.0\sigma(F)$ were used in solution and refinement of the structure.

2.2.3. Structure and refinement

The structure was solved by direct methods (SIR-92) [21] and refined by full matrix least squares procedure using SHELXTL [22]. In the final cycles of refinement all non-H atoms were treated anisotropically. The H atoms attached to carbon atoms were included as fixed contributions, the function minimized was $\Sigma\omega(F_o - F_c)^2$ where $w^{-1} = \sigma^{-2}(F) + 0.0012F^2$, resulting in $R = 0.042$ and $\omega_r = 0.457$.

3. Results and discussion

The golden shiny crystals of the complex $[(\eta^6\text{-}C_6Me_6)Ru(\mu\text{-}Cl)_3Ru(\eta^6\text{-}C_6Me_6)]PF_6$ are soluble in dimethylsulfoxide, dimethylformamide and nitromethane; they have poor solubility in acetone, methanol and insoluble in diethylether and petroleum ether. The complex gave conducting solutions in nitromethane with characteristic values of 1:1 electrolyte. Infrared spectra of the complex exhibited a broad band at 256 cm^{-1}, along with the characteristic bands due to hexamethylbenzene and PF_6^- anion. The band at 256 cm^{-1} has been assigned to ν(Ru–Cl) and presence of this band suggested that only bridged chloro atoms are present in the complex [11,12,15]. The ^{1}H-NMR spectra of the complex in acetone-d_6 exhibited a singlet at δ 2.16 ppm (CH_3 protons of C_6Me_6). The singlet in the ^{1}H-NMR indicated that the two C_6Me_6 groups are chemically equivalent and they are bound to the metal in a η^6 mode. The ^{13}C-NMR spectrum shows two peaks, one at δ 15.09 ppm and other at δ 95.94 ppm; these have been assigned to methyl carbons of C_6Me_6 group and ring carbons of C_6Me_6 group, respectively. ^{31}P-NMR spectra exhibited a band at δ −143.53 ppm corresponding to ^{31}P nuclei of the anion PF_6^-. It was present in its characteristic septet pattern. The molecular ion peak in the FAB mass spectra appeared at m/z 633. The observed value matched well with the calculated value (633). All these data are in agreement with the formulation $[Ru_2(\eta^6\text{-}C_6Me_6)_2(\mu\text{-}Cl)_3]PF_6$. The structure of the complex was confirmed by single crystal X-ray study.

3.1. Crystal structure

The structure of the compound has been determined by single crystal X-ray diffraction. The crystal data, intensity and refinement parameters are given in Table 1, final atomic coordinates and equivalent isotropic displacement parameters are listed in Table 2. Selected bond length and bond angles are given in Table 3. The geometry of the cation and atom numbering in it are shown in Fig. 1. The packing of cations and anions in the unit cell is ilustrated in Fig. 2.

In the cation $[(\eta^6\text{-}C_6Me_6)Ru(\mu\text{-}Cl)_3(\eta^6\text{-}C_6Me_6)]^+$, both the metal centres Ru(1) and Ru(2) have octahedral coordination with the two octahedral units sharing a common face defined by three chlorine atoms and approximately planer η^6-C_6Me_6 ligand. The Ru(1)···Ru(2) distance is 3.277(1)Å and the two hexa-

Table 1
Crystal data for the complex $[(\eta^6\text{-}C_6Me_6)Ru(\mu\text{-}Cl)_3Ru(\eta^6\text{-}C_6Me_6)]PF_6$

Formula	$C_{24}H_{36}Cl_3F_6PRu_2$
Molecular weight	778.0
Color, habit	Red, plates
Crystal system	Monoclinic
Space group	$P2_1/c$
Unit cell dimensions (Å)	
a	10.7604(9)
B	24.897(2)
c	10.8833(11)
β (°)	100.990(7)
Volume (Å^3)	2862.2(5)
Z	4
D_{calc} (mg m^{-3})	1.805
Absorption coefficient (mm^{-1})	1.444
F(000)	1552
Reflections collected	9341
Independent reflection	7786
Observed reflections	3104 [$F>4.0\sigma(F)$]
R	0.042
WR	0.0457
GoF	0.76

methylbenzene groups are present in an eclipsed position as indicated by a very small dihedral angle (2.37°) between the two (η^6-C_6Me_6) planes. The inter ruthenium distance of 3.277(1) Å in the complex is very close to the one having zero bond order [23,24]. Metal to chlorine bond lengths in the cation, average Ru–Cl 2.44(3) Å [range 2.432(3)–2.463(2) Å], are very close to one another (Table 3) and these are comparable with the one reported for an analogous trichloro bridged benzene complex $[(\eta^6\text{-}C_6H_6)Ru(\mu\text{-}Cl)_3(\eta^6\text{-}C_6H_6)]^+$ with average Ru–Cl bond lengths of 2.423(7) Å [25] and slightly shorter than the Ru–Cl bonds in the precursor complex $[\{Ru(\eta^6\text{-}C_6Me_6)Cl_2\}_2]$ [2.460(1) Å] [23]. The bridging Cl–Ru–Cl angles [79.85(1)° average, range 79.4(1)–80.8(1)°] are comparable to those in the complexes $[(\eta^6\text{-}C_6H_6)Ru(\mu\text{-}Cl)_3(\eta^6\text{-}C_6H_6)]^+$ (79.33°) and $[\{Ru(\eta^6\text{-}C_6Me_6)Cl_2\}_2]$ [80.90(2)°], while the bridging Ru(1)–Cl–Ru(2) angles in the the title cation are more acute [83.1(1), 84.9(1) and 84.4(1)°] than those in the precursor complex $[\{Ru(\eta^6\text{-}C_6Me_6)Cl_2\}_2]$ [99.02(2)°].

Both the ruthenium atoms Ru(1) and Ru(2) are bound approximately symmetrically to the aromatic carbon atoms of hexamethylbenzene ligand with the average Ru(1)–C bond distances of 2.176(13) Å [range 2.162(14)–2.194(16) Å] and Ru(2)–C bond distances of 2.179(14) Å [range 2.161(16)–2.190(12) Å]. These bond length with very small and insignificant variations are consistant with the planarity of the hexamethylbenzene rings. The ruthenium atoms Ru(1) and Ru(2) are displaced from their attendant hexamethylbenzene rings by 1.6458 and 1.6466 Å, respectively, which are comparable to Ru-ring plane distance in the precursor complex 1.654(1) Å [26]. However, these are slightly

Table 2
Atomic coordinates (×10) and equivalent isotropic displacement coefficients (A^2 × 10^3) [a]

	x	*y*	*z*	U_{eq}
Ru(1)	1217(1)	6215(1)	6751(1)	29(1)
Ru(2)	−1389(1)	6270(1)	7890(1)	31(1)
Cl(1)	−1012(2)	6203(1)	5732(2)	56(1)
Cl(2)	364(3)	6891(1)	7976(3)	59(1)
Cl(3)	352(3)	5629(1)	8180(2)	61(1)
C(1)	2570(15)	5624(4)	6302(10)	41(3)
C(2)	3167(16)	5931(5)	7363(10)	37(4)
C(3)	3138(13)	6509(5)	7238(9)	37(3)
C(4)	2513(12)	6769(4)	6109(9)	39(9)
C(5)	1949(12)	6441(4)	5085(8)	39(3)
C(6)	1946(13)	5871(4)	5199(9)	39(3)
C(7)	2525(16)	5018(4)	6464(10)	61(4)
C(8)	3752(16)	5677(5)	8558(10)	57(4)
C(9)	3722(15)	6847(5)	8361(10)	61(5)
C(10)	2489(13)	7376(4)	6027(9)	59(4)
C(11)	1255(12)	6698(4)	3878(8)	64(4)
C(12)	1260(13)	5541(4)	4077(8)	66(4)
C(21)	−3313(16)	6555(6)	7355(10)	49(4)
C(22)	−3341(15)	5983(6)	7438(10)	44(4)
C(23)	−2711(14)	5739(5)	8591(11)	46(4)
C(24)	−2053(15)	6041(5)	9585(10)	49(4)
C(25)	−2046(14)	6606(5)	9513(9)	47(4)
C(26)	−2668(14)	6875(5)	8385(9)	45(4)
C(27)	−3938(16)	6828(6)	6146(9)	72(5)
C(28)	−3955(16)	5646(5)	6331(11)	69(5)
C(29)	−2651(17)	5128(4)	8665(11)	72(5)
C(30)	−1349(15)	5778(5)	10 760(9)	82(5)
C(31)	−1313(15)	6940(5)	10 536(9)	80(5)
C(32)	−2652(15)	7491(4)	8271(10)	71(5)
P(1)	4859(3)	6254(3)	2348(2)	65(1)
F(1)	5257(14)	6670(4)	1361(8)	119(5)
F(2)	3489(6)	6243(5)	1524(5)	103(3)
F(3)	6250(6)	6264(4)	3124(5)	97(2)
F(4)	4490(12)	6745(4)	3166(7)	114(4)
F(5)	5189(12)	5767(4)	1538(8)	103(4)
F(6)	4521(14)	5840(4)	3300(8)	125(5)

[a] Equivalent isotropic *U* defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 3
Selected bond lengths (Å) and angles (°) for the complex

Ru(1)–Ru(2)	3.277(1)		
Ru(1)–Cl(1)	2.443(2)	Ru(2)–Cl(1)	2.463(2)
Ru(1)–Cl(2)	2.432(3)	Ru(2)–Cl(2)	2.426(3)
Ru(1)–Cl(3)	2.443(3)	Ru(2)–Cl(3)	2.434(3)
Ru(1)–C(1)	2.189(14)	Ru(2)–C(21)	2.161(16)
Ru(1)–C(2)	2.194(16)	Ru(2)–C(22)	2.184(16)
Ru(1)–C(3)	2.162(14)	Ru(2)–C(23)	2.183(14)
Ru(1)–C(4)	2.168(12)	Ru(2)–C(24)	2.178(13)
Ru(1)–C(5)	2.184(10)	Ru(2)–C(25)	2.190(12)
Ru(1)–C(6)	2.169(12)	Ru(2)–C(26)	2.177(14)
Cl(1)–Ru(1)–Cl(2)	79.4(1)	Cl(1)–Ru(2)–Cl(2)	79.2(1)
Cl(1)–Ru(1)–Cl(3)	79.7(1)	Cl(2)–Ru(2)–Cl(3)	80.8(1)
Cl(2)–Ru(1)–Cl(3)	80.5(1)	Cl(1)–Ru(2)–Cl(3)	79.5(1)

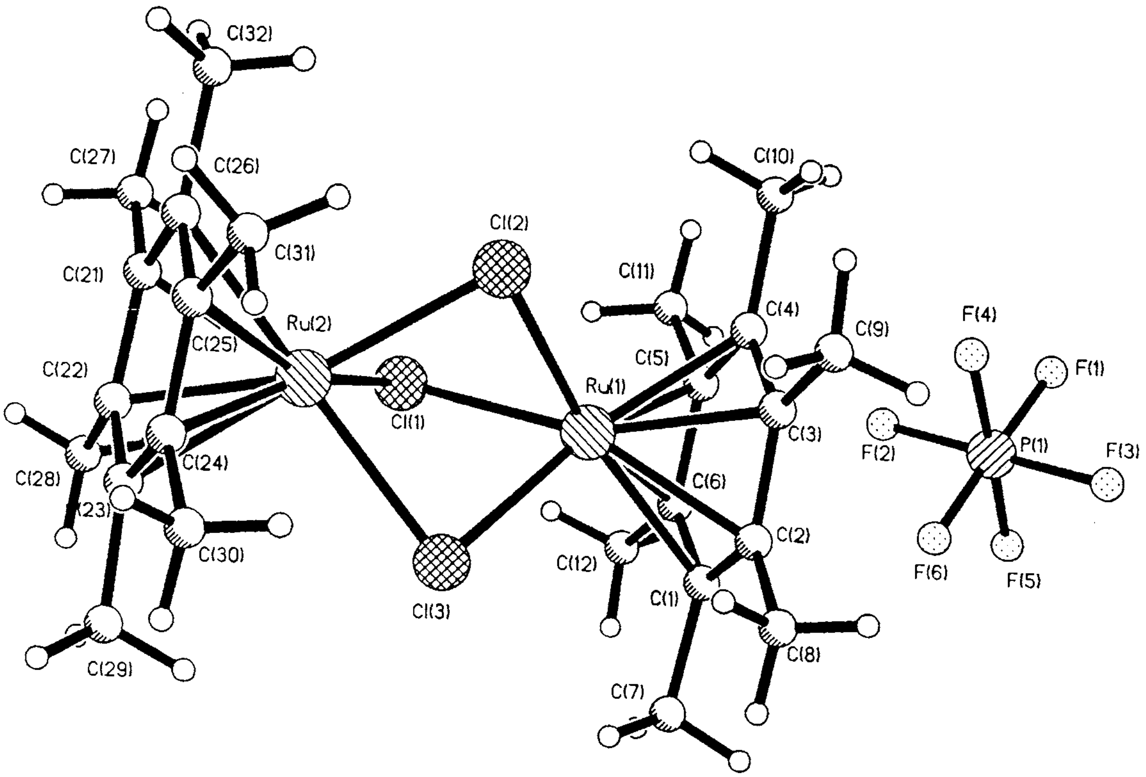

Fig. 1. The geometry of $Ru_2(\eta^6\text{-}C_6Me_6)_2(\mu\text{-}Cl)_3PF_6$.

smaller as compared to the average distance in the bis arene complex $[Ru(\eta^6\text{-}C_6Me_6)(\eta^4\text{-}C_6Me_6)]$ (1.76 Å) [27].

The average C–C bond lengths in the hexamethylbenzene ring attached to Ru(1) is 1.426(15) Å [range 1.400(15)–1.445(18) Å]. There are no alternate short and long bond lengths as well as there are only insignificant differences bettwen C–C $(\eta^6\text{-}C_6Me_6)$ bonds, which clearly indicates that there is no π localization and the bond lengths are in between C–C single and double bonds. A similar trend is observed for hexamethylbenzene ring attached to Ru(2) with average C=C distances of 1.423(17) Å [range 1.394(16)–1.446(14) Å]. The bond angles in the hexamethylbenzene rings are also unexceptional and show no significant trends associated with the non-planarity of the ring. The relative orientations of the methyl groups on the hexamethylbenzene rings and C(ring)–C(methyl) distances are also unexceptional. The counter ion PF_6^- exhibited virtually octahedral geometry with average P–F distances of 1.582(10) Å.

The reaction of $[\{Ru(\eta^6\text{-}C_6Me_6)Cl_2\}_2]$ with silver trifluoroacetate leads to the formation of $[Ru(\eta^6\text{-}C_6Me_6)Cl(O_2CCF_3)]$ in benzene [28]. It has also been shown that in solvents like nitromethane and methanol, ionic dissociation of trifluoroacetate complex takes place to give solvated species like $[Ru(\eta^6\text{-}C_6Me_6)Cl(Solvent)_2]^{2+}$ [28]. In our hands under similar reaction conditions in methanol and in the presence of NH_4PF_6 the chloro bridged complex $[(\eta^6\text{-}C_6Me_6)Ru(\mu\text{-}Cl)_3Ru(\eta^6\text{-}C_6Me_6)]PF_6$ was isolated. In the present case, it seems that instead of the formation of a trifluoroacetato complex, monomeric solvated species are formed, which in turn, by intramolecular coupling, gets converted into the chloro bridged complex. It is also expected that in the absence of NH_4PF_6 and under given reaction conditions, one may get the complex $[(\eta^6\text{-}C_6Me_6)Ru(\mu\text{-}Cl)_3Ru(\eta^6\text{-}C_6Me_6)]CF_3COO^-$. However, all our attempts failed to isolate the above complex, instead, we could only get a highly hygroscopic yellow product, which could not be properly characterized.

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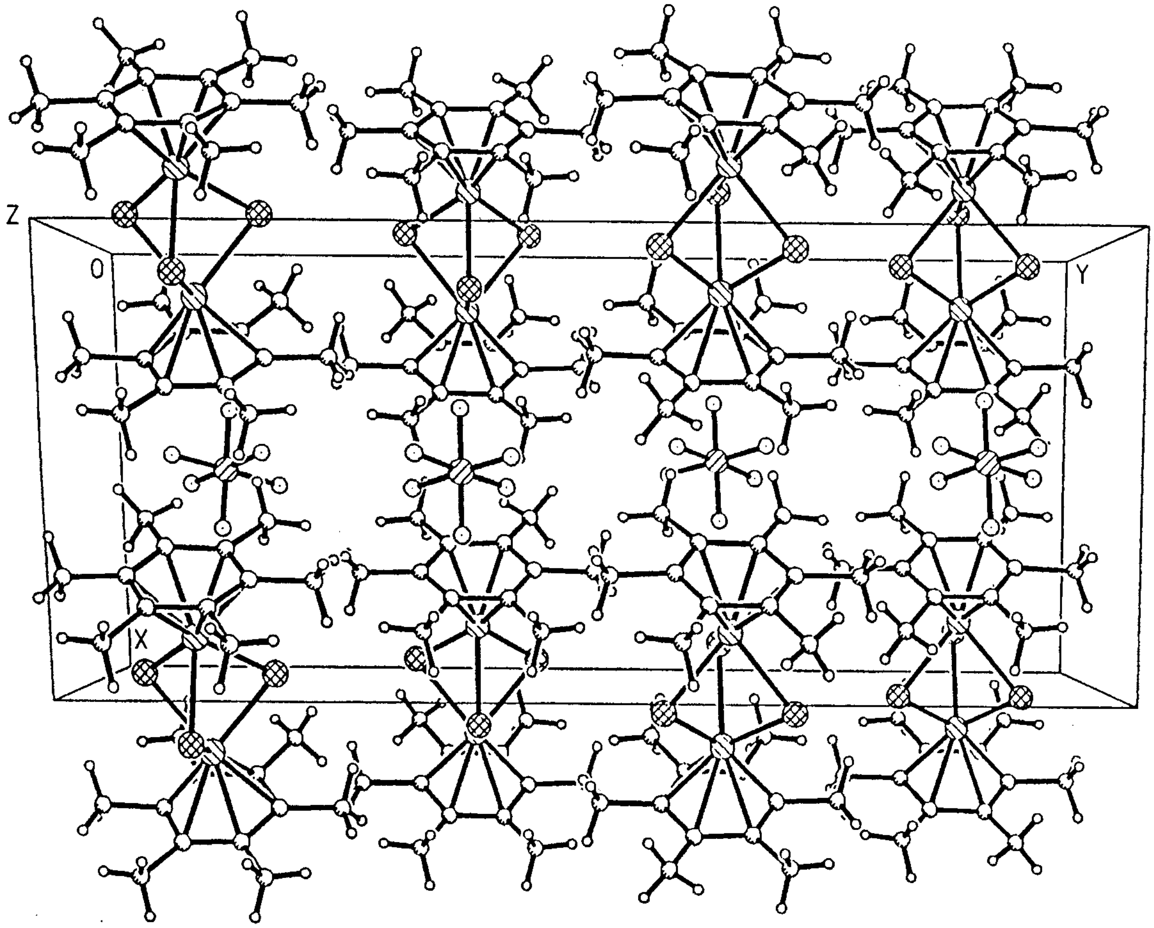

Fig. 2. The packing of cations and anions in the unit cell.

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