

. $[\text{Ni}(\text{H}_2\text{NNHCSSCH}_3)_3]_3\text{Cl} \cdot 3\text{H}_2\text{O}$ . A similar situation was found in the following compound II.  $[\text{NiC}_6\text{H}_{17.5}\text{N}_6\text{S}_6](\text{ClO}_4)_{1.5}$ , trigonal,  $P\bar{3}$ ,  $a = b = 9.670(4)$ ,  $c = 13.045(5)\text{\AA}$ ,  $Z = 2$ , with three  $[\text{ClO}_4]^-$  ions for the two Ni complexes.

One of the two  $[\text{ClO}_4]^-$  ions is statistically disordered across a center of inversion at  $001/2$ . Some bond parameters are: Ni-S = 2.386(2), Ni-N = 2.130(7), C-S = 1.672(7), C-N = 1.319(7), N-N = 1.401(9)\text{\AA}. The two crystals I and II have almost the same structure.

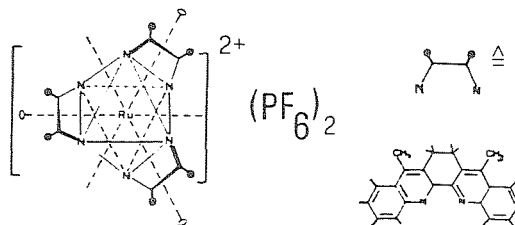
III.  $[\text{NiC}_6\text{H}_{18}\text{N}_6\text{S}_6](\text{ClO}_4)_2$ , monoclinic,  $P2_1/b$ ,  $a = 11.361(2)$ ,  $b = 22.323(5)$ ,  $c = 9.045(2)\text{\AA}$ ,  $\beta = 101^\circ 10(2)'$ ,  $Z = 4$ . Some lengths are: Ni-S = 2.370-2.393(2), Ni-N = 2.132-2.135(6), C-S = 1.650-1.667(7), C-N = 1.305-1.317(8), N-N = 1.402-1.409(8)\text{\AA}.

The  $\text{Ni}(\text{H}_2\text{NNHCSSCH}_3)_3^{2+}$  cation in this complex retains the pseudooctahedral NS coordination geometry. The difference between the complexes II and III exists in the molecular packing.

#### 09.4-40 CRYSTAL STRUCTURE OF RU-TRIS (4,7-DIMETHYL-2,3,8,9-DIBENZ-5,6-DIHYDRO-PHENANTHROLINE) DI-HEXAFLUOROPHOSPHATE.

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The title compound  $\text{C}_{66}\text{H}_{54}\text{F}_{12}\text{N}_6\text{P}_2\text{Ru}$  crystallizes in the trigonal space group  $P\bar{3}1c$  with  $a=b=14.180$ ,  $c=17.160$  and  $Z=2$ . The cation itself having exact  $D_3$  symmetry occupies a  $32$  special position of the space group. The structure was solved by direct methods by first eliminating the large structure factors of the metal ion. Spectroscopic data of the compound are given.



#### 09.4-39 CRYSTAL STRUCTURES OF TWO TRANS BIPYRIDINE COMPLEXES OF Ru(II), Ru(BIPYRIDINE)<sub>2</sub>(PPh<sub>3</sub>)<sub>2</sub>(PF<sub>6</sub>)<sub>2</sub> AND Ru(4,4'-DIMETHYLBIPYRIDINE)<sub>2</sub>(PYRIDINE)<sub>2</sub>(PF<sub>6</sub>)<sub>2</sub>;

VARIATIONS IN CONFORMATION FOR THE NECESSARILY DISTORTED TRANS BIS(BIPYRIDINE) CHELATION, By A. W. Cordes, P. N. Swepston, W. Pennington, S. M. Condren, and B. Durham, Department of Chemistry, University of Arkansas, Fayetteville, Arkansas 72701, USA.

Octahedral complexes containing two 2,2'-bipyridine (bpy) ligands generally are found to have the *cis* geometry, consistent with the expected instability of the *trans* configuration due to steric interaction between opposing  $\alpha$ -hydrogen atoms of the bpy ligands. The title cations represent the second and third structures reported of octahedral complexes with *trans* bpy groups and these, along with four square-planar di-bpy complexes, allow generalizations on the modes of accommodating the steric strain. Three of the complexes have bpy units which are in a "twist" arrangement whereby the two  $\alpha$  hydrogen atoms of each bpy move to opposite sides of the idealized  $\text{MN}_4$  plane. The other complexes display a "bowed" configuration in which the  $\alpha$  hydrogen atoms of each bpy are displaced on the same side of the plane, opposite the  $\alpha$  hydrogens of the *trans* bpy. These distortions are readily analyzed and contrasted by use of the bend and twist coordinates developed for the discussion of non-planar peptide linkages. The two title compounds are found to be in the bowed configuration, in contrast to the complex

$\text{Ru}(\text{bpy})_2(\text{H}_2\text{O})(\text{OH})^{+2}$  which has a twist conformation.

#### 09.4-41 TRIBUTYLPHOSPHINE SUBSTITUTED POLYNUCLEAR Ru-CARBONYL CARBOXYLATES. By M. Bianchi & P. Frediani, *Istituto di Chimica Organica dell'Università di Firenze*, Via G. Capponi 6, Firenze, and M. Nardelli & G. Pelizzi, *Istituto di Chimica Generale e Inorganica dell'Università di Parma*, Centro di Studio per la Strutturistica Diffrattometrica del C.N.R., Via M. D'Azeglio 85, Parma (Italy).

Two new polynuclear Ru-carbonyl tributylphosphine substituted carboxylates have been synthesized by reaction of  $\text{Ru}_3(\text{CO})_{12}$  with acetic and glutaric acids respectively and subsequent treatment with tributylphosphine. Their crystal data are:

(I)  $[\text{Ru}_3(\text{CO})_4\{\text{OOC}(\text{CH}_2)_3\text{COO}\}(\text{Pbut}_3)_2]_2$ , monoclinic  $P2_1/n$ ,  $a = 16.544(10)$ ,  $b = 23.819(15)$ ,  $c = 11.046(8)\text{\AA}$ ,  $\beta = 103.72(6)^\circ$ ,  $V = 4229(5)\text{\AA}^3$ ,  $Z = 2$ ,  $D_c = 1.333\text{ g cm}^{-3}$ ,  $\mu = 8.14\text{ cm}^{-1}$  (Mo K $\alpha$ )

(II)  $[\text{Ru}_3(\text{CO})_4(\text{CH}_2\text{COO})_2(\text{Pbut}_3)]_2$ , monoclinic  $P2_1/n$ ,  $a = 14.411(8)$ ,  $b = 19.198(10)$ ,  $c = 9.665(8)\text{\AA}$ ,  $\beta = 93.45(6)^\circ$ ,  $V = 2669(3)\text{\AA}^3$ ,  $Z = 2$ ,  $D_c = 1.579\text{ g cm}^{-3}$ ,  $\mu = 12.06\text{ cm}^{-1}$  (Mo K $\alpha$ ).

Both structures were solved by single crystal diffractometer data (Mo K $\alpha$ ), and refined by full-matrix least-squares to  $R = 5.18\%$  for (I) and  $R = 3.83\%$  for (II).

The structure of (I) consists of couples of dimers  $(\text{Pbut}_3)(\text{CO})_2\text{Ru}-\text{Ru}(\text{CO})_2(\text{Pbut}_3)$  joined together by glutarate bridges coordinating to the metal atoms through their oxygens, each carboxylate group bridging two Ru atoms. Coordination around each Ru is distorted octahedral and involves two *cis* carbonyl ligands and two oxygen atoms from two carboxylate groups in a coordination plane and one phosphine and a Ru-Ru bond, 2.734(3)\text{\AA} long, *trans* with respect to that plane.