09.4—43 THE CRYSTAL STRUCTURE OF 5,6-DIHYDROPYRIMI-DINO [5,4-c] CARBAZOLE-METHANOL – BIS (5,6-DIHYDRO PYRI-MIDINO [5,4-c] CARBAZOLE-N[3]) TETRAKIS (METHANOL) COBALT (II) DINITRATE (2,2,1), $2C_{14}H_{11}N_3 \cdot 2CH_4O$. Co$(C_{14}H_{11}N_3)_2$ $(CH_4O)_4 \cdot 2NO_3$. By B. Viossat*, Nguyen-Huy**, J.C.Lancelot** M. Robba**, U.E.R. des Sciences Pharmaceutiques *(Poitiers, 34,rue du Jardin des Plantes, ** **1, rue Vaubénard, 14032 Caen Cédex).

We have reported the crystal structure of the crystal structure of the monoclinic variety I of {5,6-dihydropy-rimidino [5,4-c] carbazole}, called hereafter DPC (Acta Cryst.(1984) in press). Moreover, the crystal structure of a second variety (monoclinic II) of this tetrahetero-cycle was solved but not published. The molecules exhibit the same conformation for the two polymorphs and they form ribbons linked by hydrogen bonds. However, the arran-gement of the ribbons in the form (II), gives rise to voids in which the distances between two homologous nitro-gen atoms related by a binary axis appear to permit complexation with the 3d elements such as Ni, Co or Zn. This is why we looked for a synthesis of new compounds by interacting the ions of these oligoelements with the tetraheterocycle, in order to find out the possi-ble modification of the pharmacological properties of the organic compound when it plays a role of ligand.
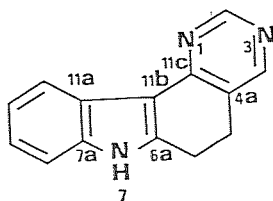
Numbering of atoms of the DPC molecule

$M_r = 1260.3$, P$\bar{1}$, a=14.43(2), b=10.70(1), c=13.966(7)Å, α=111.94(5), β=92.77(6), γ =124.9(1)°, V=1541(3)Å^3, Z=1. D_x=1.36Mg m^{-3}, R is 0.053 for 2929 independent reflec-tions.

The structure contains [Co$(C_{14}H_{11}N_3)_2$$(CH_3OH)_4$]$^{2+}$, NO$_3^-$ ions, two $(C_{14}H_{11}N_3)$ {5,6-dihydropyrimidino-[5,4-c] car-bazole (DPC)}and two (CH_3OH) molecules.

The cobalt atom is surrounded by two nitrogen atoms which belongto two DPC molecules and four oxygen atoms of four methanol molecules. The octahedral arrangement is almost regular. The 1.3-cyclohexadiene rings of the two different molecules of DPC remain in a skew chair confor-mation but with a large tendency towards planarity and with an inversion of relative positions of atoms C(5) and C(6) about the mean plane compared with the homologous ring of the monoclinic form (1) of this molecule.
So the polymorphism of the various DPC molecules might be explained by the flexibility of this 1.3-cyclohexadiene ring. Many hydrogen bonds take part in the cohesion of the structure of the title compound.

09.5—1 RESEARCH ON THE CRYSTAL STRUCTURE OF TRINU-CLEAR MOLYBDENUM CLUSTERS. By Huang Jinling,Shang Maoyu, Huang Jianquan, Lu Shaofang,Zhuang Honghei,Wu Diengming, Lin Xianti and Lu Jiaxi, Fujian Institute of Research on the Structure of Matter, Academia Sinica, China.
In a systematic investigation of the behavior of middle-valenced Mo atom in an EtOH-HCl medium, a variety of tri-nuclear Mo clusters has been synthesized and structurally characterized by X-ray crystallography. The results show that the Mo clusters so obtained may be divided into three categories(B1,M1,M2) according to the configuration of the cluster skeleton. 1) The structure of B1 type is a bi-capped trinuclear species. Each Mo atom has a distort-ed octahedral coordination as shown in Fig.1. Two com-pounds $(C_5H_7S_2)_3${$Mo_3(\mu_3-S)_2Cl_9$}(I), $(Et_4N)_2${$Mo_3(\mu_3-O)(\mu_3-Cl)Cl_9$}(II) are found to belong to this cat-egory. 2) M1 type structure is a monocapped species with a dis-torted octahedral coordination for each Mo atom as shown in Fig. 2, including $(Et_4N)_2${$Mo_3(\mu_3-O)$-$(OAc)_2Cl_8$}(III), $(C_5H_7S_2)${Mo_3-$(\mu_3-O)(OAc)_3Cl_6$}(IV), $(C_5H_7S_2)$-{$Mo_3(\mu_3-O)(OAc)_3Br_3Cl_3$}(V), (Me_4N){$Mo_3(\mu_3-O)(O_2CH)_3Br_3Cl_3$}(VI).
3) M2 type structure is a monocap-ped species with a distorted pentagonal bipyramidal coordina-tion for each Mo atom as shown in Fig.3. This type of cluster includes $(C_5H_7S_2)${Mo_3-$(\mu_3-S)(S_2)_3Cl_7$}(VII),(PyH)$_3${$Mo_3(\mu_3-S)(S_2)_3Cl_7$}$\cdot$EtOH (VIII) $Mo_3(\mu_3-S)(S_2)_3${$S_2P(OEt)_2$}$_3Cl$ (IX), $Mo_3(\mu_3-O)(S_2)_3$-{$S_2P(OEt)_2$}$_3I$ (X).
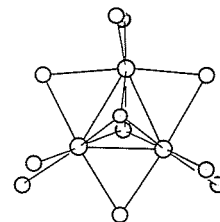The crystal data and the average Mo-Mo bond distances for all these compounds are summarized in Table 1.
The metal-metal bonding character has been discussed in terms of EHMO method.
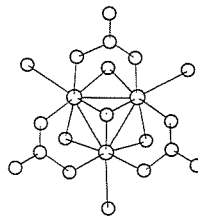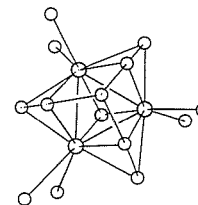

Fig.1

Fig.2

Fig.3

Table 1

Compd	S.G.	Parameters		Z	Mo—Mo (Å)	
I	I4$_1$/a	a=22.882(9)	c=25.407(7)	16	2.641(1)	2.653(1)
					2.556(1)	
II	P2$_1$/c	a=11.379(3)	c=14.897(3)	4	2.601(1)	2.642(1)
		b=20.766(2)	β=91.49 (2)		2.608(1)	
III	Pna2$_1$	a=17.815(1)	c=12.003(1)	4	2.584(2)	2.594(2)
		b=16.629(2)			2.618(1)	
IV	P2$_1$/c	a=10.423(1)	c=14.062(7)	4	2.568(2)	2.578(2)
		b=17.095(4)	β=97.55 (3)		2.585(2)	
V	P2$_1$/c	a=10.517(3)	c=14.053(3)	4	2.584(1)	2.594(2)
		b=17.213(2)	β=96.72 (2)		2.603(2)	
VI	P$\bar{1}$	a= 6.848(1)	α=100.20(1)		2.593(1)	
		b=11.933(1)	β= 93.12(1)	2	2.598(1)	
		c=13.164(1)	γ=101.14(1)		2.596(1)	
VII	Pnam	a=17.226(4)	c=15.074(4)	4	2.755(1)	2.755(1)
		b=14.306(7)			2.743(1)	
VIII	Pa3	a=19.605(2)		8	2.750(2)	
IX	P$\bar{1}$	a=11.570(2)	α=102.62(1)		2.727(1)	
		b=13.093(1)	β=102.15(1)	2	2.722(1)	
		c=14.399(2)	γ=111.87(1)		2.725(1)	
X	P2$_1$/n	a=16.570(2)	c=20.007(2)	4	2.627(1)	2.620(1)
		b=12.370(1)	β=99.97 (1)		2.630(1)	