09.5-7 THERMAL REACTION OF H$_3$Re$_3$(CO)$_{12}$ WITH P(C$_6$H$_5$)$_3$ AND P(OCC$_6$H$_5$)$_3$, By L.K. Liu, Institute of Chemistry, Academia Sinica, Nanjing, Taiwan 115; S.C. Lin and C.P. Cheng, Department of Chemistry, National Tsing Hua University, Hsin-Chu, Taiwan 300, Republic of China.

The thermal reaction of $H_3Re_3(CO)_{12}$ with excess $P(C_6H_5)_3$, results in triphenylphosphine substituted rhodium cluster, $H_3Re_3(CO)(P(C_6H_5)_3)_h$, as the major product with a 71% yield. An x-ray structure analysis of $H_3Re_3(CO)(P(C_6H_5)_3)_h$, has been performed with the following results: Mr 1597.57, monoclinic P2$_1$/n, $a$=15.598(1), $b$=9.530(2), $c$=41.685(3) Å; $\alpha$=90, $\beta$=112.50(1)°. CuKa radiation, room temperature data, R=0.048 for 7361 reflections with I>2.5σ(I). The $H_3Re_3(CO)(P(C_6H_5)_3)_h$ molecule has a triangular Re$_3$ core coordinated propeller-wise by 3 $P(C_6H_5)_3$ ligands in the molecular plane with a virtual C$_3$ symmetry, not counting the orientation of phenyl groups. The bulky $P(C_6H_5)_3$ ligands introduce crowding such that all of the $s$i$n$-CO ligands are tilted away from it. The mono-substituted $H_3Re_2(CO)(P(C_6H_5)_3)$ exists in two interconvertible isomeric forms with $P(C_6H_5)_3$, axial or equatorial (C.Y. Niel et al., J. Organomet. Chem. 1981, 273, 63-78). Nonetheless, the tri-substituted $H_3Re_3(CO)(P(C_6H_5)_3)_h$, has a structure consistent with a minimal steric interaction. The thermal reaction of $H_3Re_3(CO)_{12}$ with excess $P(OCC_6H_5)_3$, similarly results in triphenylphosphate substituted $H_3Re_3(CO)(P(OCC_6H_5)_3)_h$, as a major product but with only a 37.5% yield because of further ligand substitutions.

09.5-8 THE CRYSTAL AND MOLECULAR STRUCTURE OF A NOVEL GOLD-SULPHUR CLUSTER COMPOUND, P.A. Bates, Chemistry Department, University of Auckland, New Zealand and J.M. Waters, Department of Chemistry and Biotechnology, Massey University, Palmerston North, New Zealand.

The three-dimensional structure of a novel cluster compound of gold has been determined by the x-ray method. The complex $[Au_3S_3(Ph_2PCH_2CH_2PPh_2)_6]H_2O$, crystallises from chloroform solution in the monoclinic system, space group P2$_1$/n, with $a$=17.746(1), $b$=34.344(3), $c$=29.272(6) Å, $\beta$=122.12(3), $U$=17932 Å$^3$, $Z$=4. A total of 17363 data were collected with MoK$_\alpha$ radiation ($\mu$(Na-K$_2$O) = 153.8 cm$^{-1}$) on a CAD4 diffractometer using the w/2θ scan technique. A crystal of size 0.03x0.02x0.004 cm was employed in the data collection. The structure was solved by a combination of direct methods, Patterson and Fourier techniques and refined by a large-block least-squares process to a final R of 0.066 on the $F^2$ value for which $F^2=3(F^2)^2$. In the cluster two structurally distinct types of gold atom can be identified. Type I gold is bound to two sulphide groups to form an inner core of six gold and two sulphur atoms: type II gold forms an outer ring of metal atoms where the donors are one sulphur and one phosphorus atom. Each bidentate phosphine ligand spans two adjacent gold atoms around the outside of the cluster. The inner core is distinguished by the approximately octahedral geometry of its six metal atoms with the two sulphurs occupying trans coordination sites. The remaining sites involve Au...Au...Au interactions. The bonding geometry of the type II gold atoms is less well defined although in each instance a trans arrangement of sulphur and phosphorus donors is observed. Further Au...Au interactions are noted with at least one of these being with a type I metal.

09.5-9 STRUCTURES OF HIGH NUCLEARIY CLUSTERS; SPECT AND FICTION, By Mary McPartlin, School of Chemistry The Polytechnic of North London, U.K.

Much frontier work in high nuclearity cluster chemistry depends, like protein crystallography, on an ability to extract an extraordinary amount of information from very limited x-ray data. Metal core geometry alone, judiciously combined with spectroscopic and analytical data, often proves to be the only means of assigning a correct molecular formula to a new cluster. For example the first high nuclearity cluster ever reported was shown from poor data to have a highly improbable metal centred $Au_3(S_2)_{10}$ (X = P or S) core (1).