Cdr(CN)₂ has Cu₂O-like structure, where two diamond-like 3D frameworks based on tetrahedral Cd²⁺ and bridging CN⁻ are interpenetrated each other. Cd(CN)₂·G clathrates were prepared on the idea that one framework in Cd(CN)₂ was replaced with bulky organic guest molecules such as cyclohexane, CCL₄, etc. X-ray study on Cd(CN)₂ cyclohexane clathrate revealed that the host lattice has the diamond-like 3D framework and guest cyclohexane molecule is trapped in empty space of the host lattice. However, two questions remained. One was about ordering of the bridging CN⁻· X-ray showed shortening of the CN⁻ in disordered state. However, it has been believed that CN⁻ in Cd(CN)₂ is ordered. ¹³Cd MAS NMR revealed that the CN⁻ was in disordered state in Cd(CN)₂·G and even in Cd(CN)₂. Another question about structure and motion behavior of the cyclohexane. X-ray showed only diffuse electron density around the center of the cavity. ¹H-NMR powder patterns indicated that the cyclohexane was in rapid random isotropic motion at room temperature and this isotropic motion and the inversion of the cyclohexane ring began to freeze in lower temperature region.

Crystallographic data are often directly used as a basis for conclusions of chemical significance. For example, observed differences in Pt-CI bond lengths in different structures may be used to evaluate cis- and trans-influence in square-planar complexes. However, crystallographic bond lengths are not determined by intramolecular effects alone but also packing effects. To establish a reasonable level of significance for an evaluation of an intramolecular "chemical" effect, it is necessary to consider the combined outcome of the experimental errors and packing effects. We have determined each of the crystal structures of [PtCl(dms)₃](PF₆)₃ and [PtCl₂(dms)CF₃SO₃] from two independent data sets using different crystals. Comparisons of the two data sets for one compound using half-normal probability plots revealed only small systematic errors and the e.s.d.'s are not underestimated more than 15-20 %. Cross-comparisons of the geometry in the Pt(II)-complex in the different compounds gave "noise" levels for differences in Pt-Cl and Pt-S distances of 0.014 and 0.024 Å for 95 and 99% confidence interval, respectively. Therefore we propose that observed differences of Pt-Cl or Cl bond lengths should be at least 0.02 Å to be claimed to be the result of intramolecular effects.

A variety of related Pt(II) compounds were synthesised and characterised with the aid of various physical techniques, such as X-ray crystallography and multi-nuclear NMR spectroscopy. In general the compounds contained one labile group, Cl⁻ in this case: one strong coordinating ligand like H₂O, CH₃OH or CH₃CN and two tertiary phosphine or arson ligands which could be either cis or trans to one another. Some lesser known mono- and bidentate ferrocene containing phosphine ligands were among those used in the study. The aim of the investigation was to manipulate the reactivity of the Pt(II) centre and to bring the observed kinetic results in correlation with the obtained physical parameters like bond distances, chemical shifts and coupling constants of the various ligands under investigation. One example of a compound investigated in this study is:


In another reaction, a by-product formed as green crystals insoluble in most solvents. The structure solved easily to a R factor of 2% and the molecules appeared to be the rhenium(VI) monomer trans-ReO₃Cl₂(PPh₃)₃. Bond lengths were consistent with this formulation and all thermal ellipsoids looked normal, but the presence of an unusual infrared band remained puzzling. Very careful analysis helped elucidate the identity of this intriguing structure, which turned out to contain a Re(V) centre.

Investigation structure of 8-oxyquinoline, 8-mercaptopquinoline and 8-selenoquinoline metal complexes is of great interest because it offers the possibility to elucidate some regularities of spatial structure of the mentioned compounds depending on the nature of ligand (O,S,Se) atoms. The object of the present work is the study of crystal and molecular structure of Sb(C₆H₅NSe)₃(I) in comparison with the structures of Sb(C₆H₅NS)₃(II) and Sb(C₆H₅NO)₃(III). By our X-ray method values of dihedral angles between coordination planes restricted by OSbN, SbSBn and SbSn (in Fig.) it is shown, that in molecules of octahedral complexes of transition metals the angles are close to 90°. But these angles diverge considerably more in octahedral complexes of non-transition elements. The dihedral angles between the coordination planes SbSBn,