STRUCTURE AND BONDING
IN SOME PLATINUM COMPLEXES

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To

I.A.R.B.

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Summary

In this thesis the technique of X-ray crystal structure analysis is applied to the study of molecular structure, conformation and bonding in some platinum(II) and platinum(I) complexes. The contents are divided into three parts.

In Part I some of the theoretical and practical aspects of the X-ray diffraction methods, pertinent to the work described in Parts II and III, are surveyed.

Part II, which is presented in six Chapters, is concerned with the structures of eight square-planar platinum(II) complexes. Current views on trans- and cis-influence of ligands and some aspects of metal-phosphorus bonding are first reviewed (Chapter 1). This is followed by a description of the crystal structure analyses of three platinum(II) complexes containing the novel ligand $PMe_2C_6F_5$ (Chapter 2). The interest is centred on the effect of electron-withdrawal of phosphine substituents on the metal-ligand bonding. Chapter 3 is devoted to the structure analyses of the complexes cis-$PtCl_2(PEt_3)L$, where L=PEt_3 and CO. This work completes a systematic study of such complexes and the results are discussed in terms of the cis and trans-influence of the ligands. The constancy of the observed triethylphosphine conformation in square-planar platinum complexes has led to molecular mechanics calculations on the triethylphosphine molecule, which are also described. In Chapter 4 the structure of a platinum(II) complex which provides the first known example of a metallated phosphine-carborane is described. The interest in the effect of

strongly electron-withdrawing substituents on sulphur-donor atom on metal-ligand bonding has led to determination of the crystal structure of cis-$PtCl_2(CF_3SCH_2CHMeSCF_3)$, presented in Chapter 5. Chapter 6 is devoted to the structure analysis of trans-$[PtCl(COEt)(PMe_2Ph)]_2$. This complex displays an unusually large $^1J(Pt-P)$ coupling constant. The X-ray study was carried out in order to examine the correlation between Pt-P bond lengths and coupling constants in bridged binuclear platinum(II) species.

Part III is concerned with the structure analyses of two closely similar and novel platinum(I) complexes, which contain direct metal-ligand bonds.

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II Tables of Observed and Calculated Structure Factors are presented separately from this thesis.

PART I

X-RAY CRYSTAL STRUCTURE ANALYSIS

1. Introduction

Crystals have been a subject of study and speculation for hundreds of years. The idea that they are built by three-dimensional geometrical repetition of identical units was established as early as 1665.[1] By the middle of the eighteenth century Haüy[2] was able to develop the law of rational indices, which states that the intercepts made on crystallographic axes a, b, c, by any crystal face can be expressed as ratios a/h, b/k, c/l, where h, k and l are small whole numbers. This notation was popularized in the next century by Miller,[3] and the three integer indices h, k, l, now bear his name. Subsequent work led Hessel[4] to deduce, from Haüy's law of rational indices, the 32 symmetry classes; these are the groups of self-consistent symmetry operations applicable to crystal morphology. In 1850 Bravais[5] described 14 space lattices geometrically compatible with the 32 crystal classes (crystallographic point groups). This purely group-theoretical investigation was extended in 1890, independently by Fedorov[6] and Schoenflies.[7] They considered all symmetry operations possible in space lattices, thus arriving at 230 space groups. However, this work aroused little interest until 1919, when Niggli[8] showed that the space group of a crystal can be determined by an analysis of the X-ray diffraction pattern.

Although X-rays were discovered in 1895, by Röntgen, it was not until 1912 that the first diffraction pattern (of crystalline copper sulphate) was observed by Friedrich, Knipping and von Laue.[9] This experiment simultaneously demonstrated the wave nature of X-rays and the periodic structure of crystals. Shortly afterwards the ionization spectrometer was developed by W.H. and W.L. Bragg.[10] They realised that the intensity-weighted X-ray diffraction pattern could be used for determination of the internal structure of crystals, and deduced a simple equation which treats diffraction as reflection from planes in the crystal lattice.[11] This led rapidly to improved understanding of the relationship between the diffraction pattern and the structure of crystals. During the 1920's the ionisation chamber was gradually superseded by the X-ray camera. General acceptance of photographic methods was promoted mainly by application of the theory of the reciprocal lattice, and of the Ewald sphere of reflection,[12] to the interpretation of single crystal rotation photographs,[13] and also by the invention of the Weissenberg[14] and precession[15] cameras. Since 1945, however, interest in counter methods has revived. The Geiger counter and later proportional and scintillation counters have been developed as reliable detectors. With the recent development of high-speed electronic computers and computer-controlled diffractometers, the determination of increasingly large and complex crystal structures, such as proteins, has become possible.

A crystal structure analysis normally proceeds through three distinct stages. (i) Measurement of the intensities of Bragg reflections, which are then corrected for various geometrical and physical factors to yield a set of structure amplitudes. (ii) The solution of the phase problem: the phases of the diffracted beams cannot be measured directly and yet they must be derived in some way before the structure can be solved. Because of the remaining uncertainties in the phases, this first structure is only approximately correct. (iii) The approximate atomic parameters must be refined to obtain the best agreement between the observed and calculated structure amplitudes.

The following sections summarise briefly the theoretical and experimental techniques employed in X-ray structure analysis, with the emphasis on those methods actually used in the determination of the crystal structures described in this thesis.

2. The Structure Factor

X-rays are scattered by electrons, and when the Bragg condition is obeyed the scattering is coherent and elastic. The amplitude and phase of the beam scattered by a single unit cell, when reflection occurs from the hkl Bragg planes, are defined by the structure factor:

$$F(hkl) = \iiint \rho(xyz)\exp\left[2\pi i(hx+ky+lz)\right] dxdydz \qquad (1.1)$$

The integration is over the volume of the unit cell. The number of electrons per unit volume at the point with fractional coordinates x,y,z is expressed by the electron density function, $\rho(xyz)$. The structure factor is a complex quantity and can be written as

$$F = A + iB$$

The quantities

$$|F| = (A^2 + B^2)^{\frac{1}{2}}$$

and

$$\alpha = \tan^{-1} B/A$$

define the amplitude and phase of the scattered beam, relative to those of the beam that would be scattered in the same direction by an electron placed at the origin of the unit cell.

The function r(xyz) reaches its maxima at the atomic centres in the unit cell and falls asymptotically to zero in the space between the atoms. It is therefore convenient to rewrite (1.1) as

$$F(hkl) = \sum_j f_j \exp\left[2\pi i(hx_j+ky_j+lz_j)\right] \tag{1.2}$$

The summation is over all atoms in the unit cell, the centroid of electron density of the jth atom being at x_j, y_j, z_j. The <u>atomic scattering factor</u>, f_j, is defined by

$$f_j = \iiint r_j(xyz)\exp\left[2\pi i(hx+ky+lz)\right]dxdydz \tag{1.3}$$

where the integration is now over the volume occupied by the jth atom and the origin of fractional coordinates has been shifted to x_j, y_j, z_j. It is a good approximation to assume that all atoms of the same chemical type have identical electron distributions. Integrals of the type (1.3) can be evaluated by quantum mechanical methods.[16] The finite volume occupied by electrons in an atom leads to differences in the phases of rays scattered from different points in the atomic volume. The resulting destructive interference becomes greater as the Bragg angle, and hence the phase differences increase . The scattering power of the atom, measured by the scattering factor f, therefore decreases as the Bragg angle increases.

The atomic scattering factors used in this work are those listed in refs. 16 and 17. They are based on spherical atomic electron density functions. This assumption is obviously not strictly valid for covalently bound atoms, but the resulting error is small and can be balanced out, at least partly, when anisotropic temperature factors are employed (see below).

Published atomic scattering factors refer to atoms at rest. In order to apply (1.2) quantitatively it is necessary to introduce temperature factors, which allow for the vibrations of atoms about their equilibrium positions. The frequencies of these vibrations are so much smaller than the frequency of X-rays that, to X-rays, the atoms appear to be stationary and displaced from their equilibrium positions. Thus, in producing a given X-ray reflection, atoms in neighbouring unit cells will scatter slightly out of phase, the total effect leading to a reduction of the atomic scattering factor by an amount which increases with the Bragg angle. In practice, the scattering factor should therefore be multiplied by

$$\exp\,(-B\sin^2\theta/\lambda^2) \qquad (1.4)$$

where B is related to the mean-square amplitude of the isotropic atomic vibration, $\bar{u}^2$, by

$$B = 8\pi^2\bar{u}^2.$$

An approximate estimate of the average value of B (for all atoms in the unit cell) can be obtained from statistical comparison of the observed structure amplitudes with those theoretically predicted for a crystal composed of a random assemblage of atoms.[18]

In general, however, thermal motion of atoms is not spherically symmetrical; rather, it is anisotropic and leads to an ellipsoidal distribution of electron density. The general form of the temperature factor expression contains six parameters, specifying the magnitude and orientation of the three principal axes of the ellipsoidal electron density of an atom. For any set of lattice planes hkl it can be written as

$$\exp\left[-2\pi^2(U_{11}h^2a^{*2}+\ldots+2U_{12}hka^*b^*+\ldots)\right] \qquad (1.5)$$

where the quantities U_{ij} are the thermal parameters of an atom, expressed in terms of mean-square amplitudes of vibration in Å^2 units, and a^*, b^*, c^* are the reciprocal lattice axes.

In the calculation of structure factors it is assumed that electrons in atoms behave like free classical electrons. Provided that the force exerted by the external electromagnetic field is much larger than the binding forces between the atomic nucleus and electrons, the above assumption is justifiable. Although this is generally the case for lighter atoms with the radiations commonly used in X-ray analysis, it is often not true for the heavier atoms. When the external force approaches in magnitude the binding forces in the atom (absorption edge) this assumption breaks down. The resulting <u>anomalous dispersion</u> causes both the magnitude and phase of the atomic scattering factor to differ from the classical value. Allowance for this effect is made by using scattering factors of the type

$$f_j^{anom} = f_j + \Delta f'_j + i\Delta f''_j$$

where $\Delta f'_j$ and $\Delta f''_j$ correct the external-force-independent scattering factor, f_j, for anomalous dispersion. $\Delta f''$ is always positive; that is, the phase of the scattered radiation is advanced relative to that which would be scattered from a hypothetical atom containing free classical electrons. It should be noted that the terms $\Delta f'$ and $\Delta f''$ are almost independent of the scattering angle. This occurs because the effect involves the inner electrons of the atom. Hence, for a given atom, the effects of anomalous scattering are greater at higher scattering angles. Anomalous scattering corrections for many atoms, based on quantum-mechanical calculations involving hydrogen-like atomic wave functions and probably fairly accurate for the innermost K electrons, have been calculated by Dauben and Templeton[19] for several wave lengths and are listed in ref. 16. Typical values (for Mo - K_{α} radiation) used in this work are:

Atom	$\Delta f'$	$\Delta f''$
Pt	-2.352	8.388
Cl	0.132	0.159
P	0.090	0.095
S	0.110	0.124

3. Experimental Study of the Diffraction Pattern

The preceding discussion has shown that the structure factors of Bragg reflections are related to the positions of atoms in the unit cell of a crystal. To utilise this relationship in the structure analysis it is first necessary to examine the characteristics of the diffraction pattern of the crystal. Such an examination permits the crystal system, unit cell dimensions, Laue group and the possible space groups to be established. The diffraction pattern can also provide values of the structure amplitudes, $|F(hkl)|$, for all symmetrically independent reflections. To obtain these the integrated intensities of the diffracted beams, I(hkl), must be measured.

In this work the usual procedure was to make a preliminary examination of the crystal by taking oscillation and Weissenberg photographs, using Cu - K_α radiation. From these photographs the crystal symmetry (system, Laue group and space group) and the approximate values of cell dimensions were established. The space group was determined from systematic absences of reflections.

The crystal was then transferred to a four-circle diffractometer (Hilger and Watts Y-290 or Enraf-Nonius CAD-4F) and, if necessary, reoriented on the gonimeter head so that none of the crystal axes coincided with the diffractometer ϕ-axis, in order to avoid multiple reflections.[20] The setting angles of <u>ca</u>. 10-12 reflections, well dispersed through reciprocal space, were measured accurately and used to determine the orientation of the crystal and the unit cell parameters by a least-squares technique. Integrated intensities were then measured automatically for each reflection, by rotating the crystal through the Bragg reflecting position and recording E, the total X-ray energy diffracted by the crystal. The quantity E is related to I(hkl) by

$$I(hkl) = Ew/I_o$$

where w is the angular velocity of the crystal rotation and I_o is the intensity of the incident X-ray beam. Usually w and I_o are constant, and I(hkl) is directly proportional to E.

In all experiments the intensity measurements were carried out with molybdenum radiation. A pulse-height analyser was used in conjunction with either a β-filter or crystal monochromator, to remove other than the K_α characteristic radiation. The $\theta/2\theta$ scan technique was employed, the counter moving at twice the angular velocity of the crystal. Care was taken to ensure that the scan width and number of steps were sufficient to cover the full profile of the reflection, both at low and high angles. Stationary crystal-stationary counter backgrounds were measured at each end of the scan range. The integrated intensities were calculated from the expression

$$I = C-(B_1+B_2)T_p/2T_b \qquad (1.6)$$

where C, B_1 and B_2 are the peak and background counts, and T_p and T_b the times spent measuring the peak and each background, respectively. The standard deviation of I was derived from the equation

$$\sigma(I) = \left[C+(B_1+B_2)T_p{}^2/4T_b{}^2+(qI)^2\right]^{\frac{1}{2}} \qquad (1.7)$$

where the empirical factor q was taken to be 0.04.[21] Two or three strong reflections (standards) were measured periodically throughout each experiment, to monitor the stability of the crystal, counting chain and intensity of the incident beam. Where necessary, the intensities of all reflections were scaled according to variations in the intensities of standards.

For an infinitesimally small crystal of volume δV, it can be shown that the integrated intensity is related to the structure amplitude by the equation

$$I(hkl) = (e^2/mc^2)^2 N_c{}^2 \lambda^3 Lp \, |F(hkl)|^2$$

where e and m are the electron charge and mass, respectively, c the velocity of light, N_c the number of unit cells per unit volume and λ the X-ray wavelength.[22] The Lorentz factor, L, and polarisation factor, p, are functions of Bragg angle and depend on the experimental conditions.

The intensity of a reflection is proportional to the time during which the corresponding reciprocal lattice point is close to the surface of the reflecting sphere. The Lorentz factor arises because this time varies with the position of the reciprocal lattice point and the direction in which it approaches the sphere. For normal-beam equatorial geometry diffractometers (such as the Y-290 and CAD-4) and $\theta/2\theta$ scans

$$L = 1/\sin 2\theta.$$

The polarisation factor arises because of the dependence of the scattered intensity on the orientation (with respect to the reflecting plane) of the electric vector, E, of the incident X-ray beam. The magnitude of p depends on the degree of polarisation of X-ray beam. Although characteristic X-ray radiation itself is not polarised, it becomes partially polarised when reflected from a monochromator crystal. For the normal-beam equatorial geometry, a perfect monochromator crystal and an ideally imperfect crystal specimen

$$p = (1+|\cos 2\theta_m|/\cos^2 2\theta)/(1+|\cos 2\theta_m|)$$

where θ_m is the Bragg angle appropriate to the monochromator crystal.

4. Systematic Errors in Measured Integrated Intensities

The integrated intensity of a reflection obtained from a macroscopic crystal must be related to that of an infinitesimal volume element, δV. To do this the observed intensities must be corrected for the effects of absorption, extinction and coincidence loss.

Absorption correction

The intensities of both the incident and diffracted X-ray beams are attenuated in the crystal by photo-electric absorption. If I is the observed integrated intensity and I_o the value it would have in the absence of absorption, then

$$I/I_o = V^{-1}\int \exp[-\mu(l_1+l_2)]dV \qquad (1.8)$$

where the integration is over the total volume of the crystal specimen, V, and l_1 and l_2 are the respective path lengths through the crystal of the beams incident on, and scattered from, the volume element dV. The linear absorption coefficient, μ, is a constant for the specimen. It depends mainly on the chemical nature of the crystal and can be estimated from the relationship

$$\mu=\rho\sum_i p_i(\mu/\rho)_i \qquad (1.9)$$

where ρ is the density of the crystalline compound, p_i the fraction by weight of the ith element present and $(\mu/\rho)_i$ the mass absorption coefficient of the ith element appropriate to the X-ray wavelength used. The mass absorption coefficients used in this work were those listed in ref. 16.

Although several procedures for calculation of the integral (1.8), suitable for high-speed computers, have been developed, no fully satisfactory method for the general case has so far been described. Busing and Levy[23] introduced accurate numerical evaluation of the absorption integral based on the Gaussian method.[24] In this procedure, which cannot be used if the crystal has re-entrant angles, a non-isometric grid is set up along the crystallographic axes a, b and c. If these axes are selected in the order a, b, c and if min and max abbreviations indicate the maximum and minimum coordinates, along the crystal axes, of a set of crystal vertices then the grid points are defined as

$$x_i = x_{min}+(x_{max}-x_{min})u_i$$

$$y_{ij} = y_{min}(x_i)+\left[y_{max}(x_i)-y_{min}(x_i)\right]u_j$$

$$z_{ijk}= z_{min}(x_i,y_j)+\left[z_{max}(x_i,y_j)-z_{min}(x_i,y_j)\right]u_k$$

where the Gaussian constants, u, depend only on the number of grid points chosen. The grid points are accumulated near the surface of the crystal where the change in absorption is largest. The tables of Gaussian constants[25] contain values for u and for the associated weights, R. The weight of a grid point $x_i y_j z_k$ is $R_i R_j R_k$ and is proportional to the volume element represented by the point. The absorption integral is then obtained as

$$A = 1/V \sum_i \sum_j \sum_k R_i R_j R_k \exp(-\mu L_{ijk}) \qquad (1.10)$$

where $L = \ell_1 + \ell_2$. The integral is evaluated separately for each reflection.

A different approach to correction has been proposed by de Meulenaer and Tompa.[26] The basis of their analytical method is the division of the crystal into polyhedra which the rays enter or leave through one face only. Hence, the path length within a polyhedron is a linear function of the coordinates of the point considered.

Choice between the numerical and analytical methods should be based on accuracy desired, complexity of the crystal shape, magnitude of correction and computing time. Analytical method has advantage over the numerical when absorption is severe. The results of the numerical method will always approach those of the analytical method for a suitably chosen grid.[27] The Gaussian integration remains competitive for accuracies better than 2%, and down to a transmission factor of 0.6.

The absorption coefficient, μ, derived on the basis of equation (1.9) has been criticised because it neglects any effect of the state of aggregation on the wave function.[28] However, for all but severe absorption, an accuracy of about 7% in μ is sufficient to achieve a 1% accuracy in the relative correction.[29] Errors in the crystal dimensions are more serious especially that for the smallest dimension of a strongly absorbing crystal.[30]

In this work the absorption integral was evaluated by the numerical method based on a Gaussian grid. To assess the validity of the results, a few test calculations have been carried out for each compound. The plot representations of the crystal projections, obtained from the computed coordinates of the crystal vertices, were compared with the actual view of the crystal on an optical gorniometer. The number of grid points chosen for each case was the one for which the value of the calculated crystal volume converged. Agreement was considered between intensities of symmetry equivalent reflections before and after absorption correction. An example of absorption effects is given below.

The crystal of $[PtCl(PMe_2Ph)\{C(O)Et\}]_2$ chosen for the structure analysis was a ~~plater~~-shaped with the ratio of its thickness to maximum separation between the side faces of 1:6. The linear absorption coefficient was calculated to be 107.2cm^{-1}. A grid of 860 sampling points was employed. The calculated transmission factors, on $|F|^2$, were in the range 0.17 - 0.60. The values of the R factor before and after absorption correction, for the same structural model, were 0.095 and 0.028, respectively. The most prominent features in difference Fourier syntheses, before and after absorption correction respectively, were peaks of 8.2 and 1.6 eÅ^{-3}. The changes in the positional parameters were small. The bond lengths and angles were practically the same (in terms of standard deviations) before and after absorption correction. The most dramatic change, however, was a large reduction in the standard deviations of atomic parameters.

Extinction

For a perfect crystal, absorption along the direction in which Bragg reflection occurs may be many times as large as that for an imperfect specimen. In such a case, the rays reflected from a crystal plane are at the correct angle to be reflected a second time, and the phase difference between the doubly-reflected and incident beams is π. This enhanced attenuation of the diffracted beam from an almost perfect crystal is called primary extinction.

However, as pointed out by Darwin,[31] most real crystals behave as mosaics of small blocks of perfect crystals not accurately fitted together; for such crystals the effects of primary extinction are small and can be neglected. In contrast, the effects of secondary extinction are often large. They arise from the losses of energy from the incident beam occurring on its reflection from each crystal plane in a given hkl set. Consequently, the intensities of the incident beam reaching a particular plane in the set is equal to that of the original incident beam less that which has been lost through reflection by the preceeding planes.

Both primary and secondary extinction are more pronounced for stronger reflections, bigger crystals and smaller wavelengths. Several methods for extinction correction have been suggested,[29,32] but none of these is entirely satisfactory. It is therefore common practice to exclude from the final stages of structure analysis those reflections which appear to be seriously affected by extinction. In this work no signs of serious extinction effects were detected and no reflections have been rejected for that reason.

Counting loss correction

When the intensity of a diffracted beam is measured, if two or more events occur within a period shorter than the resolving time of the detector, then only one count will be recorded. Thus, the proportion of the lost counts increases with the counting rate.

On the Y-290 diffractometer corrections for counting loss were made by remeasuring all the stronger reflections at reduced generator settings. The coefficient P_1 and P_2 in the empirical equation

$$J = P_1I+P_2I^2 \tag{1.11}$$

(where I and J are, respectively, the integrated intensities of a given reflection at normal and reduced generator settings) were then obtained by the method of least-squares. The corrected intensity was taken as J/P_1.

In the analysis of <u>cis</u>-$PtCl_2(CO)PEt_3$, for example, the coefficients P_1 and P_2 were derived from a set of 50 I and J values. It was found that only five integrated intensities suffered significant counting losses ($>1\%$), the largest correction being 7%.

On the CAD-4 diffractometer, calibrated attenuation foils were inserted automatically when high counting rates were encountered, so that all measurements were made under conditions of negligible counting loss.

5. Fourier Synthesis and The Phase Problem

A continuous, three-dimensional function, such as that describing the electron density distribution in a crystal, can be represented by a Fourier series:

$$r(xyz) = \sum_{pst}\sum\sum_{-\infty}^{+\infty} A_{pst} \exp\left[2\pi i(px+sy+tz)\right]$$

To evaluate the series the coefficients A_{pst} must be known.

By substituting the series for r(xyz) in the general expression for the structure factor (1.3) it is possible to show that the Fourier coefficients are directly related to the corresponding structure factors

$$A_{pst} = F(-p,-s,-t)/U$$

where U is the unit cell volume. Thus the Fourier series which represents the electron density distribution at every point in the unit cell may be written as

$$r(xyz) = U^{-1}\sum_{hkl}\sum\sum_{-\infty}^{+\infty} F(hkl)\exp\left[-2\pi i(hx+ky+lz)\right] \qquad (1.12)$$

The individual terms in this series correspond to the various reciprocal lattice points weighted according to the values of the structure amplitudes with which they are associated. In other words, the electron density is the Fourier transform of the weighted reciprocal lattice and vice verse.

For practical purposes (assuming that Friedel's law, $|F(hkl)| = |F(\bar{h}\bar{k}\bar{l})|$, holds), equation (1.12) can be written in the form

$$r(xyz) = U^{-1}\sum_{-\infty}^{+\infty}\sum_{hkl}\sum |F(hkl)|\cos\left[2\pi(hx+ky+lz)-\alpha(hkl)\right] \qquad (1.13)$$

In general, the structure amplitudes decrease on passing outwards from the origin of the reciprocal space, and for sufficiently large values of sinθ they become negligible. Thus, the Fourier series for the electron density effectively converges after a finite number of terms. To apply equation (1.13) to the solution of crystal structures we must know the phase associated with each structure amplitude. Unfortunately, knowledge of these phases is lost in the process of recording the intensities. The need to know, at least approximately, the phases of the Bragg reflections in order to calculate an electron density distribution constitutes the phase problem in crystal structure analysis.

6. The Patterson Function and The Heavy-Atom Method

The key to solution of the phase problem is that the number of structure amplitudes greatly exceeds the number of parameters to be determined. In modern structure analysis two strategies for overcoming the problem are in common use. Relationships between the phases of different reflections can be applied systematically - the direct methods approach.[33]

Alternatively, in the heavy-atom method advantage is taken of the presence in the unit cell of a few atoms of high atomic number. In the work described here the latter approach was used exclusively.

The heavy-atom method is based on the properties of the Patterson function.

The Patterson function

In 1934 A.L. Patterson discovered that direct evidence about atomic positions could be inferred from a Fourier synthesis of the form

$$P(uvw) = \sum_{-\infty}^{+\infty}\sum_{hkl}\sum |F(hkl)|^2 \cos 2\pi(hu+kv+lw) \qquad (1.14)$$

Peaks at (xyz) and (x+u,y+v,z+w) in the electron density distribution give rise to a maximum in the Patterson function at (uvw), the value of P(uvw) being approximately related to the product of r(xyz) and r(x+u,y+v,z+w).[34]

Resolution problems usually make it unprofitable to attempt to derive all the atomic positions from the Patterson function. For a crystal containing N atoms per unit cell there will be N(N-1) maxima in the Patterson function, compared with only N in the electron density distribution. Moreover, the maxima in P(uvw) are typically twice as broad as those in r(xyz). Correction for the decrease in atomic scattering with increasing Bragg angle gives a sharpened Patterson function in which these difficulties are alleviated rather than eliminated.[35]

The heavy-atom method

When the unit cell contains a relatively small number of heavy atoms, the Patterson peaks due to these atoms stand out strongly against the background of overlapping smaller peaks. Thus they can readily be identified and used to derive the coordinates of the heavy atoms. Since the heavy atom terms tend to dominate the structure factor expression (1.3), phases calculated from the heavy atom scattering alone are usually sufficiently close to the true phases for the electron density expression to be used to locate more atoms. The process can then be repeated with successively more atoms included in the structure factor calculation, until all atoms have been located. The more serious disadvantage of this approach is that the domination of the scattering by the heavy atoms diminishes the accuracy with which the positions of the other atoms can be determined.

7. Refinement Techniques

Once all or most of the atoms have been located the analysis is completed by the process of refinement. This involves elaborating the model by, for example, allowing for anisotropic vibration or hydrogen scattering, finding the best values for the parameters which describe the model, and detecting and removing systematic errors in the structure amplitudes.

Difference synthesis

A direct measure of the differences between the true electron density and that associated with the model is provided by the difference density function

$$\Delta r(xyz) = U^{-1}\sum_{h}\sum_{k}\sum_{l}(|F_o|-|F_c|)\exp[-2\pi i(hx+ky+lz)+i\alpha_c] \qquad (1.15)$$

where α_c is the calculated phase angle. Even in the early stages of an analysis this function tends to be more informative than the corresponding approximate electron density function because the coefficients required are numerically smaller and series termination effects thus less serious. At a first approximation, correctly placed atoms will not appear in the difference map, incorrect ones will be in holes, and missing ones will appear as peaks. However every peak which appears is not necessarily connected with a missing atom and it is necessary to select atomic peaks on the basis of their relative positions and shapes. Anisotropic vibrations will produce positive and negative regions near atomic positions. Absorption and extinction errors will give rise to peaks, usually near the heavy atoms, even after correction for anisotropic vibrations. Smoothening of the difference synthesis after correction for absorption, in the later stages of analysis, is to be expected. For location of hydrogen atoms from the difference synthesis a good data set is required and both the positional and vibrational parameters of non-hydrogen atoms should have been already refined. Peaks corresponding to hydrogen atoms are normally in the range 0.3-0.7eÅ^{-3}.

The least-squares method

In modern structure analysis the final values of the atomic positional and vibrational parameters are usually determined by the least-squares method, first introduced into crystallography by Hughes.[36] The mathematical basis of this method is the proposition that the best agreement between sets of experimental and calculated quantities is obtained when the sum of the squares of the discrepancies between them is a minimum. In X-ray analysis the function most commonly minimised is

$$D = \sum_{hkl} w_{hkl}(|F_o|-|F_c|)^2$$

where w_{hkl} is the weight of the observation. The sum is over all independent structure amplitudes. If the standard deviation for each $|F(hkl)|$ is $\sigma(hkl)$, derived from equation (1.7), the value of w which gives the lowest standard deviations in the derived parameters may be shown to be

$$w_{hkl} = 1/\sigma(hkl)^2 \qquad (1.16)$$

Let $p_j (j=1,\ldots,n)$ be the n parameters occuring in $|F_c|$ whose values are to be determined. For D to be at minimum we must have

$$\partial D/\partial p_j = 0 (j = 1,n)$$

The parameters have to be varied until these n conditions are satisfied. If the trial set of parameters are close to the correct values it is possible to approximate value of

$$\Delta = |F_o| - |F_c|$$

by means of a truncated Taylor series as

$$\Delta(p+\varepsilon) = \Delta(p) - \sum_{i=1}^{n} \varepsilon_i \, \partial|F_c|/\partial p_i$$

where ε_i is a small change in parameter p_i, and p and ε stand for the whole set of parameters and changes. This leads to the normal equations

$$\sum_{i=1}^{n} \Big\{ \sum_{hkl} w_{hkl} (\partial|F_c|/\partial p_i)(\partial|F_c|/\partial p_j) \Big\} \varepsilon_i = \sum_{hkl} w_{hkl} (\partial|F_c|/\partial p_j) \qquad (1.17)$$

This is a set of n linear equations with n unknowns. Its solution yields the vector ε and hence gives better, although still approximate values for the various parameters. These may be used to repeat the process until the parameters shifts are negligible.

It is common to express the normal equations in matrix form $\underline{\underline{a}}\underline{\underline{\varepsilon}} = \underline{b}$ where

$$a_{ij} = \sum_{hkl} w_{hkl} (\partial|F_c|/\partial p_i)(\partial|F_c|/\partial p_j) \qquad (1.18)$$

$$b_j = \sum_{hkl} w_{hkl} (\partial|F_c|/\partial p_j) \qquad (1.19)$$

To reduce the time and computer-storage requirements needed for large structures, it is necessary, even with modern computers to consider approximations in which many off-diagonal elements of the matrix, $a_{ij} (i \neq j)$, are neglected. The off-diagonal elements are sums of products which may be either positive or negative, and they are in general smaller than the diagonal elements. It is evident, equation (1.18), that the magnitude of a_{ij} depends on the joint variation of $|F_c|$ with p_i and p_j. If these parameters are correlated in any way, the contributions to a_{ij} will not cancel in a random way. This will be true, for example, for the scale and thermal parameters, occupation number and thermal parameters of the same atom, the six U_{ij} parameters of one atom, the coordinates of a given atom if the interaxial angles of the unit cell differ appreciably from 90°, vibrational parameters of one atom and the coordinates of its nearest neighbours, etc. Therefore it is preferable, if the structure is too large for the full-matrix least-squares method, to work with some approximation intermediate between a full and diagonal matrix. With three-dimensional data a block-diagonal approximation is useful. This involves a chain of 4 x 4 or 9 x 9 matrices for the coordinates and isotropic or anisotropic thermal parameters, and a 2 x 2 matrix for the scale and overall isotropic thermal parameter (the latter is usually a dummy parameter). Although much faster than the full-matrix approach, more cycles of refinement are usually required when such approximations are employed.

R - Factors

It is conventional to report the agreement between observed and calculated structure amplitudes in terms of the figures of merit

$$R = \sum(|F_o| - |F_c|)/\sum|F_o| \qquad (1.20)$$

$$R_w = \{\sum w(|F_o| - |F_c|)^2/\sum w|F_o|^2\}^{\frac{1}{2}} \qquad (1.21)$$

where the summations are over the reflections actually employed in the analysis. Final values of R and R_w for satisfactory analyses are typically 0.02-0.06 and 0.04-0.08 respectively. They depend on a great variety of factors and are thus not an especially reliable guide to the relative accuracy of a particular structure determination. The figures of merit are however more useful as a guide to the course of refinement since they drop sharply as the model is improved.

8. Interpretation of Results

Systematic errors

Systematic errors may occur in two ways: from incorrect data or an incorrect model. They may influence precision or accuracy, or both. The most common sources of errors in the data originate from absorption or extinction effects. Untreated absorption or extinction can often be detected from the appearance of the difference synthesis or from analysis of the discrepancies between $|F_o|$ and $|F_c|$. Uncorrected absorption, particularly with centrosymmetric crystal specimens, affects mainly scale and thermal parameters.[37] Extinction effects are usually concentrated on a small number of low angle data which can be given reduced or zero weights.

Errors in the model include the assumption that the electron density distribution of atoms at the rest are spherical. For hydrogen atoms this is a severe approximation and bond lengths involving hydrogen atoms determined by X-ray methods are typically 0.1Å shorter than determinations made spectroscopically or by neutron diffraction.

The atomic coordinates obtained from a crystal structure analysis are those of centroids of the scattering densities associated with vibrating atoms. Angular oscillation of a molecule has the effect of shifting the electron density maxima towards the axis of oscillation, leading to an apparent decrease in internal atomic distances. For small librations the displacement can be calculated to a good degree of approximation.[38]

Busing and Levy have suggested that bond lengths derived from X-ray analysis should be corrected for the motion of the bonded atoms relative to one another.[39] Unfortunately, the required knowledge of the dynamics of the atomic system is rarely available.

Random errors

Although the true value of an experimentally measured quantity can never be known it is possible to determine the probability that the true and experimental values differ from one another by a specified amount as a consequence of random error. The importance of applying proper significance tests before drawing conclusions from a comparison of measurements which are subject to error has been emphasized by Cruickshank.[40]

For example, the probability, P, that a difference between the quantities x_1 and x_2, with standard deviations σ_1 and σ_2, arises from random errors can be assessed from the statistics

$$t = |x_1 - x_2| / (\sigma_1^2 + \sigma_2^2)^{\frac{1}{2}}$$

Values of $t = 1.96$, 2.58 and 3.29 are associated with probabilities P of 5, 1 and 0.1%. These levels are often described as not significant, significant and highly significant.

The standard deviations of molecular parameters, such as bond lengths, can be obtained as follows. A bond length or angle is in general a function of several parameters

$$g = f(p_1,\ldots,p_n)$$

and its standard deviation is then given by

$$\sigma^2(g) = \sum_{i=1}^{n}\sum_{j=1}^{n}(\partial f/\partial p_i)(\partial f/\partial p_j)\sigma_i\sigma_j\nu_{ij}$$

where ν_{ij} is the correlation coefficient between parameters i and j. The geometry program, used here, first transformed fractional co-ordinates and their standard deviations to an orthogonal basis set. For oblique cell correlations between fractional co-ordinates of the same atom were included in the calculation of the standard deviations of the orthogonal co-ordinates.

For a bond length between independent atoms with isotropic errors σ_1 and σ_2 (in Å) the standard deviation is given by

$$\sigma^2 = \sigma_1^2+\sigma_2^2.$$[41]

With the same assumption, if θ is the bond angle formed at atom 2 by bonds l_{12} and l_{23}, then the standard deviation of the angle is

$$\sigma^2 = \sigma_1^2/l_{12}^2 + \sigma_2^2 l_{13}^2/l_{12}^2 l_{23}^2 + \sigma_3^2/l_{23}^2.$$[42]

For standard deviations of dihedral angles the expression of Stanford and Waser[43] was used.

PART II

THE CRYSTAL AND MOLECULAR STRUCTURES OF SOME PLATINUM(II) COMPLEXES

CHAPTER 1 The Mutual Influence of Ligands in Square-Planar Platinum(II) Complexes

1.1 Introduction

In the later Chapters of Part II of this thesis the structures of eight square-planar complexes of platinum(II) are described. Although each complex was chosen for study because of particular points of interest (which are outlined in the appropriate Chapters), the structures as a whole display some common characteristics which are introduced here and set in the context of current views of the nature of bonding in transition metal complexes.

trans-Influence of ligands

The *trans*-labilising properties of ligands in platinum(II) complexes were recognised as early as 1926 by Chernyaev.[44] More recently it has become customary to distinguish the *trans*-effect, a kinetic phenomenon, from *trans*-influence which refers to the equilibrium state of complexes.

The definition of *trans*-influence used here is that of Pidcock, Richards and Venanzi: the *trans*-influence of a ligand is its ability to weaken the bond *trans* to itself in the equilibrium state of the complex.[45] Any experimental technique which gives information on the nature of metal-ligand bonding can be used to study *trans*-influence. In platinum(II) chemistry the most common measures of *trans*-influence have been Pt-L bond lengths,[46-48] especially Pt-Cl bond lengths, ν(Pt-L) stretching frequencies, and n.m.r. coupling constants.[49]

A selection of Pt-Cl bond lengths is given in Table 1, from which it is evident that the *trans*-ligand can cause variations of *ca.* 0.2Å in the length of a Pt-Cl bond.

TABLE 1 Selected structural *trans*-influence data for platinum(II) complexes:Some Pt-Cl bond lengths (Å)

Compound	*trans*-ligand	Pt-Cl	Ref.
cis-$PtCl_2(CO)PEt_3$	CO	2.296(4)	50
$[PtCl_4]^{2-}$	Cl	2.308(2)	51
cis-$PtCl_2(CNEt)(PEt_2Ph)$	CNEt	2.314(10)	52
$[PtCl_3(C_4H_{12}N_2)]^+$	π-(C=C)	2.342(2)	53
cis-$PtCl_2[C(OEt)(NHPh)]PEt_3$	C(OEt)(NHPh)	2.361(5)	54
cis-$PtCl_2(CNEt)(PEt_2Ph)$	PEt_2Ph	2.390(8)	52
trans-$PtCl(CH{=}CH_2)(PEt_2Ph)_2$	δ-C_{sp^2}	2.398(4)	55
trans-$PtCl(C{\equiv}PPh)(PEt_2Ph)_2$	δ-C_{sp}	2.407(6)	55
trans-$PtCl(CH_2SiMe_3)(PMe_2Ph)_2$	δ-C_{sp^3}	2.415(5)	56
trans-$PtCl(SiMePh_2)(PMe_2Ph)_2$	$SiMePh_2$	2.45	46

The data in Table 1 lead to the *trans*-influence series $Cl \sim CO < CNR \sim \pi\text{-}(C{=}C) < PR_3 < \sigma\text{-}C < SiR_3$. The very extensive spectroscopic studies of *trans*-influence have recently been reviewed. For platinum(II) complexes vibrational and n.m.r. parameters lead to *trans*-influence series which are broadly similar to those derived from bond lengths.[49] Agreement between the various techniques is by no means perfect, and the differing sensitivity of Pt-P bond lengths and coupling constants to the nature of the *trans*-ligand is considered in detail in Chapters 3 and 6.

The directional nature of *trans*-influence was considered by Sirkin, who pointed out that the s and d acceptor orbitals of a metal atom with square-planar co-ordination can be combined to give two s±d hybrids which are nearly orthogonal to each other.[57] In a more recent theoretical treatment[46] high *trans*-influence of a ligand L is related to high covalence in the M-L bond and specifically to the parameter $S^2/\Delta E$, where S is the M-L σ-overlap integral and ΔE the energy separation of the metal σ-acceptor and L σ-donor orbitals. This approach is semi-quantitative, but it leads to the *trans*-influence series $Cl < \pi\text{-}(C{=}C) < PR_3 \leqslant \sigma\text{-}C < SiR_3$, which is in accord with experimental data (see Table 1). It does not, however, formally consider how the *trans*-influence is affected by π-acidity.

Recent work in this department on complexes containing linear C-Pt-Cl systems leads to the *trans*-influence series (Table 1) $CO < CNR <$ carbenoid $< \sigma\text{-}C_{sp^3} \sim \sigma\text{-}C_{sp^2} < \sigma\text{-}C_{sp}$, which is a reversal of the π-acidity series of the carbon-donor ligands, derived from the Pt-C bond lengths. This <u>trans</u>-influence series was explained in terms of a strengthening of electrostatic Pt-Cl bonding , which occurs when d_π electron density is removed from the metal by a π-acceptor ligand <u>trans</u> to chlorine.[47]

<u>cis</u>-Influence of ligands

By analogy with <u>trans</u>-influence, <u>cis</u>-influence may be defined as the ability of a ligand to weaken bonds <u>cis</u> to itself in the equilibrium state of a complex. An extensive review of spectroscopic evidence of <u>cis</u>-influence has recently appeared.[58] In platinum(II) chemistry the existance of <u>cis</u>-influence is well-established from spectroscopic parameters (e.g. see ref. 59), but is generally considered of less importance than <u>trans</u>-influence.[46] There is little information concerning <u>cis</u>-influence on Pt-L bond lengths, except for the observation that Pt-Cl distances appear to show essentially no sensitivity to the nature of <u>cis</u>-ligands in a wide variety of complexes.[60] Semi-empirical M.O. calculations for platinum(II) complexes suggest that <u>cis</u>- and <u>trans</u>-influence are of comparable magnitude.[61] This conclusion is not generally accepted[49] and is inconsistent with the variations of Pt-Cl bond lengths in platinum(II) complexes (see above).

The existance of bond length changes which can be ascribed to <u>cis</u>-influence is demonstrated in Chapter 3 for a series of <u>cis</u>-$PtCl_2(PEt_3)L$ complexes. The effect is discussed in terms of steric and electronic properties of ligands.

Nature of platinum-phosphine bonding

All the platinum(II) complexes described below contain tertiary phosphine ligands. Phosphines are normally regarded as strong σ-donor ligands, but the phosphorus atom also possesses vacant 3d orbitals which are capable of accepting d_π electrons from a transition metal. The importance of $M \rightarrow P$ backbonding has been extensively discussed, but it still remains controversial.[62,63]

N.m.r. evidence has been widely used to suggest that $Pt-PR_3$ bonds involve little or no backbonding. $^1J(Pt-P)$ coupling constants are believed to depend mainly on the Fermi contact term, which in turn is determined by the s-electron Pt-P bond order.[64] The ratio of $^1J(Pt-P)$ coupling constants in *cis*- and *trans*-$PtCl_2(PBu^n_3)_2$ complexes is nearly identical to that for *cis*- and *trans*-$PtCl_4(PBu^n_3)_2$ species.[45] This has lead to the suggestion that the weaker Pt-P bonds in the *trans*-complexes arise because of a weakening of the Pt-P σ-bonds compared with those in the *cis*- complexes. Backbonding, if it exists at all, would not be expected to be as important in platinum(IV) as in platinum(II) complexes.[45] On the other hand, Grim has argued that n.m.r. parameters are consistent with metal → phosphine backdonation.[65]

A further complication is introduced by the substituents of the phosphorus atom. These may display widely different steric and electronic properties which can influence metal-ligand bonding. Fortunately, Tolman has developed semi-empirical parameters which can be used as a measure of the steric and electronic properties of the phosphine substituents.[63,66] Thus the combined electron withdrawing ability of the substituents of a ligand $PR^1R^2R^3$ can be represented by the parameter $\sum_{i=1}^{3}\chi_i$, the summation being over the three substituents. The individual χ_i values are obtained by fitting the A_1 $\nu(CO)$ stretching frequencies in $Ni(CO)_3(PR^1R^2R^3)$ complexes to the relationship

$$A_1\ \nu(CO) = 2056.1 + \sum_{i=1}^{3}\chi_i\ (cm^{-1}).$$

The steric bulk of a phosphine ligand can be represented by the angle θ. This is the internal angle of a right-circular cone, with apex at the metal atom, which just encloses the van der Waals envelope of the phosphine ligand. The values of θ proposed by Tolman are based on a M-P distance of 2.28Å and are measured using space-filling models.[66] The phosphine ligand is assumed to adopt the conformation which gives the minimum value of θ. For unsymmetrical phosphines the relationship $\theta = \frac{2}{3}\sum_{i=1}^{3}\theta i/2$ is used, where θi is the cone angle appropriate to an individual substituent. In Chapter 3 the validity of this approach for solid-state structures is considered in the light of conformational energy calculations on triethylphosphine.

CHAPTER 2

The Crystal and Molecular Structures of Three Square - Planar Platinum(II) Complexes Containing the Ligand Dimethyl(pentafluorophenyl)phosphine, $PMe_2C_6F_5$

I $\underline{cis}$-$PtCl_2(PMe_2C_6F_5)_2$

II $\underline{cis}$-$Pt(CF_3)_2(PMe_2C_6F_5)_2$

III $[Pt(CH_3)(PMe_2C_6F_5)_3](PF_6)$

2.1 Introduction

It is now recognised that the chemical behaviour of transition metal ions depends sensitively on the nature of ligands in the primary sphere of co-ordination. For example, the Wilkinson complex $RhCl(PPh_3)_3$ is an effective catalyst for the homogeneous hydrogenation of alkenes, whereas the closely analogous complex $RhCl(PPhEt_2)_3$ is not an effective catalyst for these reactions.[67] Such effects may be related, in principle, either to the electronic or the steric properties of ligands and consequently there is much interest in quantifying these properties for common ligands. Tertiary phosphines have played a key role in studies of this type. They are widely used to stabilize the lower oxidation states of transition metals and they can be prepared with a great variety of substituents on the donor phosphorus atom. Recently, Tolman has proposed empirically-derived parameters which measure the electronic and steric properties of different phosphines and used them to rationalise the chemical and physical characteristics of transition metal-phosphine complexes.[63,66]

In this context a comparison of alkyl- or arylphosphine complexes with similar fluoroaryl- or fluoroalkylphosphine complexes would appear to be of interest. The van der Waals radii of hydrogen and fluorine (respectively 1.2 and 1.35 Å)[68] suggest that fluorocarbon and hydrocarbon substituents are of comparable size, but the phosphines with fluorinated substituents are believed to be much more strongly electron-withdrawing.[63] Although the structural literature on transition metal phosphine complexes is extensive, little attention has been paid to phosphines with fluorocarbon substituents. Previous studies in this laboratory have shown that in the complexes cis-$MCl_2[(F_3C)_2PCH_2CH_2PPh_2]$, (M=Pd,Pt), the trifluoromethyl substituents (which are the most strongly electron-withdrawing of those considered by Tolman)[63] contract the adjacent P-M and trans M-Cl bond lengths by ca. 0.07Å.[69] To extend this investigation the crystal structures of cis-PtX_2L_2,(I) X=Cl, (II) $X=CF_3$, and $[Pt(CH_3)L_3](PF_6)$ (III), where $L=PMe_2C_6F_5$, have been determined accurately. The results of this work permit the trans-influence, and bonding to platinum, of L to be compared with those of sterically similar but less electron-withdrawing phosphines containing hydrocarbon substituents. In addition a similar comparison between CH_3^- and CF_3^- can be made.

It was originally hoped that these structure analyses would also aid the interpretation of the ^1H, ^{19}F and ^{31}P n.m.r. chemical shifts and coupling constants observed for a wider range of platinum complexes containing the ligand $PMe_2C_6F_5$. The n.m.r. experiments were carried out by Professor D.W. Meek, who also first synthesised the complexes. This hope, however, has been only partially realised.

2.2 Experimental

The three analyses were carried out using similar methods. Details peculiar to each are presented in the accompanying Crystal Data and Data Collection and Refinement Tables.

Measurements

For (I) a transparent needle with dimensions 0.011 x 0.030 x 0.023 cm along a*, b*, and c*, respectively, was employed. The crystal faces belonged to the forms $\{100\}$, $\{010\}$ and $\{001\}$.

For (II) a transparent crystal of centrosymmetric habit was used. Its eight faces belonged to the forms $\{110\}$, $\{101\}$ and $\{001\}$. The distances of the faces from the crystal centroid were in the range 0.012 - 0.016 cm.

For (III) a transparent needle was used. Its ten faces were members of the forms $\{110\}$, $\{111\}$, $\{\bar{1}\bar{1}1\}$ and $\{001\}$. The distances from an origin, defined by the intersection of the $\bar{1}\bar{1}0$, 111 and 110 faces, were in the range 0.019 - 0.027 cm.

In each case the possible space groups and approximate cell dimensions were obtained from oscillation and Weissenberg photographs. The crystal was then transferred to a Hilger and Watts Y-290 diffractometer equipped with a graphite monochromator, scintillation counter and pulse-height analyser. Molybdenum K_α X-rays, $\lambda = 0.71069$Å, were employed. Ten or eleven high-angle reflections[with θ(Mo-K_α) in the ranges 15-20° for (I), 11-17° for (II) and 12-15° for (III)] were centred and the setting angles used to determine the final cell dimensions (T = 21°C) and the orientation matrix by a least-squares procedure. The integrated intensities were estimated by the $\theta/2\theta$ scan technique, in conjunction with stationary crystal-stationary counter background measurements. In order to monitor the crystal and the electronic stability, the intensities of three standard reflections were measured periodically throughout the experiments. The intensities of these reflections exhibited only small statistical fluctuations about the corresponding mean values [±5% for (I), ±2% for (II), and ±3% for (III)]. Lorentz-polarisation and absorption corrections were applied. Transmission factors on $|F_o|^2$ were found to range between 0.20-0.46 (I), 0.21-0.32 (II), and 0.39-0.46 (III). No extinction corrections appeared to be necessary.

Structure analyses

For (I) and (III) the systematic absences define the space groups uniquely. In the case of (II) the systematic absences were consistent with the non-centrosymmetric space group Cc and the centrosymmetric space group C2/c (C_2-symmetry imposed on each molecule). The space group C2/c led to a successful refinement of the structure.

In each case the platinum atom was found from the three-dimensional Patterson function and the other atoms from subsequent difference syntheses.

The structure was then refined by the least-squares method, using weights $w = 1/\sigma^2(|F_o|)$. For (I) and (III) a block-diagonal approximation was necessary, but for (II) a full-matrix was employed. Adjustment of the positional and isotropic thermal parameters of the non-hydrogen atoms led to R = 0.12 (I), 0.063 (II) and 0.091 (III). After the correction for absorption effects was introduced and an allowance was made for anisotropic thermal vibrations of atoms, R decreased to 0.029 (I), 0.025 (II) and 0.033 (III). The hydrogen atoms were then found from subsequent difference syntheses, in positions consistent with the stereochemistry of the adjacent carbon atoms. The positional and isotropic thermal parameters of the hydrogen atoms were refined.

The final values of R were 0.025 (I), 0.023 (II) and 0.029 (III). The final difference syntheses displayed no unexpected features: function values (in $e\text{Å}^{-3}$) were in the range ±0.4 (I), ±0.4 (II) and ±0.8 (III). The adequacy of the weighting scheme was verified by establishing that mean values of $(|F_o| - |F_c|)^2/\sigma^2(|F_o|)$ varied little with either $|F_o|$ or $\sin\theta$.

The final atomic parameters and a selection of functions derived from them are presented in Tables 2.1 - 2.7. These tables were generated by the local molecular geometry program GEOM, of P.R. Mallinson and D.N.J. White. Typing errors are thus prevented, but in some cases parameters are given to more significant figures than their accuracy warrants.

The computer programs used for all structures described in this thesis were J.M. Stewart's X-RAY '72 system,[70] C.K. Johnson's ORTEP2,[71] the local data-processing program HILGER of P.R. Mallinson and K.W. Muir and GEOM.

The observed and calculated structure amplitudes for (I)-(III), and also for all the other structures which are the subject of this work, are presented as supplementary material to this thesis.

Crystal Data

Compound	I	II	III
Molecular weight	722.20	789.27	1039.39
Crystal system	monoclinic	monoclinic	monoclinic
a(Å)	11.318(2)	12.460	13.972
b(Å)	14.056(5)	8.276	14.101
c(Å)	13.914(3)	22.343	16.827
β(°)	103.51(3)	103.68	93.39
Cell volume(Å^3)	2152.17	2238.68	3309.64
No. of mol. per unit cell	4	4	4
Calculated density(g cm^{-3})	2.226	2.341	2.086
μ(Mo-K$_\alpha$)(cm^{-1})	70.7	66.0	46.1
Space group	$P2_1/c$	C2/c	$P2_1/n(C^5_{2h}$,No.14)
Molecular symmetry	----	C_2	----
Equivalent positions	$\pm(xyz)\pm(x,\frac{1}{2}-y,\frac{1}{2}+z)$	$\pm(xyz)\pm(\bar{x},y,\frac{1}{2}-z)$	$\pm(xyz)\pm(\frac{1}{2}+x,\frac{1}{2}-y,\frac{1}{2}+z)$

Summary of Data Collection and Refinement

Compound	I	II	III
θ_{max} (°)	30	30	30
Scan width $\Delta\theta$(°)	0.6	0.6	0.5
Step size in θ(°)	0.02	0.02	0.02
T_p (s)	60	60	50
T_b (s)	20	10	15
q(see Part I, Ch.3)	0.04	0.04	0.04
No. of reflections with $I > 3\sigma(I)$, n	3859	2673	5374
No. of parameters, p	329	272	545
n/p	11.7	9.8	9.9
R (%)	2.5	2.3	2.9
R_w (%)	3.3	3.0	4.1
Standard deviation of observation of unit weight	1.1	1.2	1.3

TABLE 2.1 Final atomic fractional co-ordinates and thermal parameters. Anisotropic temperature factors are of the form $\exp(2\pi^2 \times 10^{-n} \sum_{i=1}^{3} \sum_{j=1}^{3} U_{ij} h_i h_j a_i^* a_j^*)$ where n=4 for Pt and 3 for other atoms. Isotropic **thermal** parameters U_{iso} are in $Å^2 \times 10^2$.

a) cis-$PtCl_2(PMe_2C_6F_5)_2$ (I)

Atom	x	y	z	$U_{11}(U_{iso})$	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pt	0.38301(2)	0.21176(1)	0.21820(1)	412(1)	331(1)	346(1)	25(1)	112(1)	34(1)
P(1)	0.5022(1)	0.3001(1)	0.1456(1)	43(1)	44(1)	43(1)	4(1)	16(1)	8(1)
P(2)	0.2061(1)	0.2655(1)	0.1280(1)	40(1)	40(1)	41(1)	1(1)	10(1)	2(1)
Cl(1)	0.2817(1)	0.1095(1)	0.3038(1)	67(1)	48(1)	48(1)	-7(1)	21(1)	8(1)
Cl(2)	0.5666(1)	0.1463(1)	0.3096(1)	53(1)	60(1)	72(1)	12(1)	6(1)	25(1)
C(1)	0.4499(5)	0.4023(4)	0.0694(4)	50(3)	58(3)	53(3)	-2(2)	11(2)	19(2)
C(2)	0.5686(6)	0.2248(5)	0.0669(5)	75(4)	80(4)	70(4)	3(3)	39(3)	-7(3)
C(3)	0.6217(4)	0.3558(3)	0.2401(4)	37(2)	42(2)	56(3)	2(2)	11(2)	15(2)
C(4)	0.5899(4)	0.4050(4)	0.3173(4)	42(3)	58(3)	63(3)	-2(2)	15(2)	6(2)
C(5)	0.6723(5)	0.4492(4)	0.3917(4)	59(3)	59(3)	64(3)	-8(3)	8(2)	5(3)
C(6)	0.7926(5)	0.4451(4)	0.3912(5)	49(3)	63(4)	81(4)	-18(3)	-6(3)	13(3)
C(7)	0.8288(5)	0.4001(4)	0.3173(5)	34(3)	70(4)	95(4)	-5(2)	6(3)	20(3)
C(8)	0.7453(5)	0.3567(4)	0.2427(4)	46(3)	57(3)	74(3)	5(2)	19(2)	18(3)
C(9)	0.0699(5)	0.2202(4)	0.1586(5)	45(3)	57(3)	86(4)	-5(2)	19(3)	11(3)
C(10)	0.1821(6)	0.2363(4)	-0.0017(4)	66(3)	63(3)	46(3)	4(3)	1(2)	-6(2)
C(11)	0.1841(4)	0.3943(3)	0.1389(3)	39(2)	47(2)	40(2)	8(2)	11(2)	3(2)

Table 2.1.a) (contd.)

C(12)	0.1561(4)	0.4596(4)	0.0623(3)	43(2)	55(3)	43(2)	6(2)	11(2)	7(2)
C(13)	0.1369(4)	0.5547(4)	0.0780(4)	42(3)	50(3)	60(3)	6(2)	9(2)	16(2)
C(14)	0.1394(5)	0.5861(4)	0.1711(4)	43(3)	43(3)	80(4)	4(2)	17(2)	-1(2)
C(15)	0.1657(5)	0.5254(4)	0.2491(4)	50(3)	60(3)	55(3)	-2(2)	15(2)	-9(2)
C(16)	0.1882(4)	0.4310(4)	0.2328(4)	44(2)	50(3)	43(2)	2(2)	12(2)	0(2)
F(1)	0.4715(3)	0.4114(2)	0.3194(2)	46(2)	80(2)	78(2)	-8(2)	23(2)	-24(2)
F(2)	0.6351(4)	0.4973(3)	0.4629(3)	89(3)	97(3)	74(2)	-19(2)	13(2)	-26(2)
F(3)	0.8741(4)	0.4899(3)	0.4616(3)	76(3)	113(3)	104(3)	-37(2)	-18(2)	-3(3)
F(4)	0.9468(3)	0.3983(3)	0.3150(4)	37(2)	124(4)	157(4)	-8(2)	14(2)	7(3)
F(5)	0.7857(3)	0.3156(3)	0.1698(3)	58(2)	108(3)	99(3)	7(2)	43(2)	3(2)
F(6)	0.1486(3)	0.4330(2)	-0.0311(2)	85(2)	73(2)	41(2)	25(2)	18(2)	11(1)
F(7)	0.1121(3)	0.6153(2)	0.0011(3)	81(2)	64(2)	85(2)	18(2)	18(2)	31(2)
F(8)	0.1155(4)	0.6778(2)	0.1844(3)	92(3)	47(2)	115(3)	3(2)	32(2)	-10(2)
F(9)	0.1694(4)	0.5565(3)	0.3405(2)	99(3)	79(2)	60(2)	7(2)	19(2)	-23(2)
F(10)	0.2121(3)	0.3724(2)	0.3107(2)	71(2)	66(2)	40(1)	9(2)	18(1)	6(1)
H(1)C(1)[+]	0.387(4)	0.383(4)	0.011(4)	6(2)					
H(2)C(1)	0.515(5)	0.427(4)	0.046(4)	6(2)					
H(3)C(1)	0.418(4)	0.454(3)	0.104(3)	5(1)					
H(4)C(2)	0.621(7)	0.172(6)	0.108(6)	11(3)					
H(5)C(2)	0.619(5)	0.259(4)	0.034(4)	7(2)					
H(6)C(2)	0.496(7)	0.191(5)	0.013(5)	11(2)					
H(7)C(9)	0.074(5)	0.154(4)	0.153(4)	7(2)					
H(8)C(9)	0.071(6)	0.237(5)	0.231(5)	8(2)					

[+] H(n)C(m) is hydrogen atom attached to the carbon atom C(m).

Table 2.1.a) (contd.)

H(9)C(9)	0.003(5)	0.253(4)	0.119(4)	0(2)
H(10)C(10)	0.204(5)	0.169(4)	-0.003(4)	8(2)
H(11)C(10)	0.242(6)	0.259(4)	-0.031(4)	7(2)
H(12)C(10)	0.097(6)	0.263(5)	-0.041(5)	9(2)

b) <u>cis</u>-$Pt(CF_3)_2(PMe_2C_6F_5)_2$ (II)

Atom	x	y	z	$U_{11}(U_{iso})$	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pt	0	0.07756(2)	$\frac{1}{4}$	411(1)	331(1)	407(1)	0	48(1)	0
P(1)	0.0552(1)	0.2682(1)	0.18690(4)	40(1)	42(1)	44(1)	2(1)	9(1)	4(1)
C(1)	0.1539(4)	0.1930(7)	0.1452(3)	62(2)	72(3)	75(3)	19(2)	33(2)	14(3)
C(2)	0.1331(4)	0.4335(5)	0.2313(2)	52(2)	56(2)	59(2)	-13(2)	0(2)	10(2)
C(3)	-0.0487(3)	0.3665(4)	0.1258(2)	43(2)	42(2)	42(2)	0(1)	9(1)	0(1)
C(4)	-0.1591(3)	0.3162(4)	0.1095(2)	44(2)	49(2)	44(2)	0(1)	12(1)	-4(1)
C(5)	-0.2355(3)	0.3841(5)	0.0616(2)	44(2)	59(2)	51(2)	2(2)	7(1)	-9(2)
C(6)	-0.2043(3)	0.5070(5)	0.0279(2)	55(2)	57(2)	44(2)	15(2)	5(1)	2(2)
C(7)	-0.0969(4)	0.5614(4)	0.0425(2)	58(2)	48(2)	45(2)	8(2)	12(2)	5(2)
C(8)	-0.0214(3)	0.4903(5)	0.0904(2)	46(2)	49(2)	45(2)	4(2)	11(1)	0(2)
F(1)	-0.1947(2)	0.1961(3)	0.1406(1)	49(1)	61(1)	64(1)	-11(1)	9(1)	7(1)
F(2)	-0.3396(2)	0.3315(4)	0.0473(1)	44(1)	85(2)	74(2)	-4(1)	-1(1)	-3(1)
F(3)	-0.2769(3)	0.5699(4)	-0.0199(1)	68(2)	86(2)	64(2)	17(1)	-6(1)	13(1)
F(4)	-0.0656(2)	0.6791(3)	0.0094(1)	74(2)	64(2)	63(1)	9(1)	20(1)	23(1)

Table 2.1.b) (contd.)

F(5)	0.0825(2)	0.5430(3)	0.1005(1)	50(1)	60(1)	61(1)	-7(1)	12(1)	14(1)
C(9)	0.0081(4)	-0.1012(5)	0.1872(2)	57(2)	43(2)	64(2)	0(2)	6(2)	13(2)
F(6)	-0.0579(3)	-0.3210(3)	0.1905(2)	86(2)	58(2)	101(2)	-20(1)	19(2)	-26(2)
F(7)	-0.0285(3)	-0.0526(4)	0.1269(1)	94(2)	82(2)	54(1)	6(2)	3(1)	-17(1)
F(8)	0.1078(2)	-0.1647(4)	0.1889(2)	68(2)	64(2)	97(2)	12(1)	19(1)	-20(2)
H(1)C(1)	0.105(6)	0.126(10)	0.108(4)	7(2)					
H(2)C(1)	0.208(6)	0.143(10)	0.176(4)	11(2)					
H(3)C(1)	0.190(4)	0.278(6)	0.129(2)	7(1)					
H(4)C(2)	0.169(4)	0.494(7)	0.205(2)	6(1)					
H(5)C(2)	0.192(5)	0.386(7)	0.260(3)	8(2)					
H(6)C(2)	0.086(4)	0.518(7)	0.253(2)	6(1)					

c) $[Pt(CH_3)(PMe_2C_6F_5)_3](PF_6)$ (III)

Atom	x	y	z	$U_{11}(U_{iso})$	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pt	0.19539(1)	0.20315(1)	0.00944(1)	492(8)	396(8)	312(7)	-11(9)	63(6)	-37(8)
P(1)	0.1064(1)	0.3179(1)	0.0713(7)	54(1)	40(1)	35(1)	5(1)	4(1)	-4(1)
P(2)	0.3406(1)	0.2536(1)	0.0700(1)	50(1)	51(1)	39(1)	-1(1)	5(1)	-4(1)
P(3)	0.2500(1)	0.0828(1)	-0.0681(1)	76(1)	38(1)	34(1)	6(1)	6(1)	-3(1)
C(1)	-0.0204(4)	0.3278(5)	0.0446(3)	64(3)	77(4)	55(3)	18(3)	0(3)	12(3)
C(2)	0.1473(4)	0.4377(4)	0.0551(3)	91(4)	45(3)	47(3)	7(3)	8(3)	4(2)
C(3)	0.0997(3)	0.3013(3)	0.1797(3)	49(2)	41(2)	39(2)	10(2)	7(2)	-6(2)

Table 2.1.c) (contd.)

C(4)	0.0648(4)	0.2160(3)	0.2063(3)	54(3)	44(3)	45(2)	-1(2)	5(2)	-4(2)
C(5)	0.0539(4)	0.1984(4)	0.2856(3)	60(3)	59(3)	56(3)	3(3)	18(2)	11(3)
C(6)	0.0764(4)	0.2671(4)	0.3409(3)	58(3)	77(4)	38(2)	9(3)	11(2)	1(2)
C(7)	0.1101(4)	0.3528(4)	0.3168(3)	56(3)	56(3)	41(2)	13(2)	5(2)	-9(2)
C(8)	0.1221(3)	0.3693(3)	0.2377(3)	50(3)	42(2)	47(2)	7(2)	4(2)	-6(2)
C(9)	0.4471(4)	0.1794(5)	0.0767(5)	66(4)	76(4)	61(3)	12(3)	9(3)	-4(3)
C(10)	0.3876(5)	0.3580(5)	0.0227(4)	80(4)	73(4)	65(4)	-16(3)	11(3)	3(3)
C(11)	0.3365(4)	0.2776(4)	0.1775(3)	48(3)	50(3)	44(2)	3(2)	1(2)	-7(2)
C(12)	0.3106(4)	0.2029(4)	0.2244(3)	55(3)	51(3)	42(2)	9(2)	-1(2)	-4(2)
C(13)	0.3090(4)	0.2089(4)	0.3064(3)	62(3)	64(3)	45(3)	19(3)	7(2)	4(2)
C(14)	0.3332(4)	0.2932(4)	0.3427(3)	59(3)	80(4)	46(3)	18(3)	-3(2)	-12(3)
C(15)	0.3582(4)	0.3695(4)	0.2991(3)	62(3)	70(4)	59(3)	-1(3)	-6(3)	-28(3)
C(16)	0.3604(4)	0.3609(4)	0.2170(3)	59(3)	52(3)	58(3)	-4(2)	5(2)	-10(2)
C(17)	0.3720(5)	0.0400(5)	-0.0656(3)	109(5)	94(5)	47(3)	51(4)	-8(3)	-18(3)
C(18)	0.1879(7)	-0.0285(4)	-0.0537(4)	170(8)	42(3)	59(4)	-18(4)	6(4)	-3(3)
C(19)	0.2320(4)	0.1165(3)	-0.1733(3)	58(3)	45(3)	37(2)	8(2)	6(2)	-4(2)
C(20)	0.1674(4)	0.0768(4)	-0.2292(3)	65(3)	58(3)	50(3)	-7(2)	7(2)	5(2)
C(21)	0.1616(4)	0.1072(5)	-0.3077(3)	70(4)	95(5)	43(3)	2(3)	-7(2)	-9(3)
C(22)	0.2192(5)	0.1771(5)	-0.3310(3)	77(4)	92(5)	41(3)	10(3)	6(3)	19(3)
C(23)	0.2830(4)	0.2201(4)	-0.2781(3)	60(3)	68(4)	57(3)	8(3)	16(2)	18(3)
C(24)	0.2881(4)	0.1892(4)	-0.2006(3)	49(3)	58(3)	46(3)	6(2)	6(2)	0(2)
C(25)	0.0651(4)	0.1660(4)	-0.0507(3)	53(3)	74(4)	57(4)	-15(3)	4(2)	-19(3)

Table 2.1.c) (contd.)

F(1)	0.0396(2)	0.1483(2)	0.1531(2)	85(2)	46(2)	58(2)	-12(2)	12(2)	-10(1)
F(2)	0.0187(3)	0.1150(2)	0.3085(2)	100(3)	66(2)	75(2)	-14(2)	25(2)	15(2)
F(3)	0.0670(3)	0.2501(3)	0.4179(3)	98(3)	107(3)	43(2)	10(2)	19(2)	10(2)
F(4)	0.1345(3)	0.4196(3)	0.3712(2)	95(2)	82(2)	45(2)	6(2)	1(2)	-23(2)
F(5)	0.1591(2)	0.4532(2)	0.2191(2)	84(2)	43(2)	55(2)	-6(2)	3(2)	-10(1)
F(6)	0.2854(2)	0.1200(2)	0.1905(2)	95(2)	44(2)	50(2)	4(2)	6(2)	1(1)
F(7)	0.2812(3)	0.1354(2)	0.3491(2)	104(3)	74(2)	53(2)	23(2)	15(2)	18(2)
F(8)	0.3266(3)	0.3005(3)	0.4220(2)	94(2)	111(3)	41(2)	28(2)	-2(2)	-17(2)
F(9)	0.3794(3)	0.4522(3)	0.3349(2)	103(3)	92(3)	74(2)	-17(2)	-2(2)	-42(2)
F(10)	0.3828(3)	0.4391(2)	0.1773(2)	104(3)	60(2)	79(2)	-33(2)	16(2)	-12(2)
F(11)	0.1086(3)	0.0071(3)	-0.2092(2)	104(3)	83(2)	61(2)	-39(2)	3(2)	-11(2)
F(12)	0.0981(3)	0.0653(4)	-0.3596(2)	112(3)	168(5)	59(2)	-31(3)	-28(2)	-8(3)
F(13)	0.2127(4)	0.2068(4)	-0.4073(2)	116(3)	166(5)	52(2)	2(3)	-4(2)	40(2)
F(14)	0.3405(3)	0.2885(3)	-0.3012(2)	84(2)	91(3)	82(2)	-7(2)	23(2)	32(2)
F(15)	0.3519(2)	0.2319(3)	-0.1499(2)	63(2)	76(2)	60(2)	-16(2)	7(2)	-6(2)
P(4)	0.5915(1)	0.0864(1)	0.2946(1)	67(1)	57(1)	48(1)	-8(1)	14(1)	-7(1)
F(16)	0.5135(4)	0.0479(4)	0.2318(3)	137(4)	198(6)	85(3)	-91(4)	-5(3)	-12(3)
F(17)	0.5144(3)	0.0854(4)	0.3600(2)	114(3)	135(4)	88(3)	-26(3)	58(2)	-18(3)
F(18)	0.5611(4)	0.1911(3)	0.2686(3)	139(4)	91(3)	131(4)	19(3)	17(3)	24(3)
F(19)	0.6685(3)	0.0879(3)	0.2295(2)	124(3)	106(3)	90(3)	-37(3)	61(2)	-33(2)
F(20)	0.6653(3)	0.1320(4)	0.3562(2)	116(4)	178(5)	76(3)	-48(4)	-11(2)	-20(3)
F(21)	0.6188(5)	-0.0151(3)	0.3181(4)	207(6)	73(3)	222(6)	47(4)	104(5)	51(4)
H(1)C(1)	-0.048(4)	0.371(4)	0.072(3)	6(2)					

Table 2.1.c) (contd.)

H(2)C(1)	-0.059(4)	0.266(4)	0.056(4)	8(2)
H(3)C(1)	-0.023(4)	0.344(4)	-0.008(3)	8(2)
H(4)C(2)	0.105(3)	0.483(3)	0.071(3)	5(1)
H(5)C(2)	0.213(4)	0.442(4)	0.073(3)	7(2)
H(6)C(2)	0.148(4)	0.447(4)	0.003(3)	5(1)
H(7)C(9)	0.430(3)	0.122(3)	0.098(2)	4(1)
H(8)C(9)	0.492(6)	0.218(5)	0.107(5)	13(3)
H(9)C(9)	0.462(4)	0.179(4)	0.024(4)	8(2)
H(10)C(10)	0.355(4)	0.392(5)	0.019(4)	8(2)
H(11)C(10)	0.454(4)	0.377(4)	0.057(3)	7(2)
H(12)C(10)	0.408(6)	0.353(6)	-0.023(4)	12(3)
H(13)C(17)	0.375(5)	-0.009(5)	-0.104(4)	10(2)
H(14)C(17)	0.393(4)	0.019(4)	-0.017(3)	8(2)
H(15)C(17)	0.413(6)	0.092(6)	-0.079(5)	14(3)
H(16)C(18)	0.203(4)	-0.046(4)	0.000(3)	7(2)
H(17)C(18)	0.196(6)	-0.071(5)	-0.080(4)	11(3)
H(18)C(18)	0.103(5)	-0.012(5)	-0.067(4)	12(2)
H(19)C(25)	0.021(5)	0.141(5)	-0.010(4)	10(2)
H(20)C(25)	0.033(4)	0.218(4)	-0.077(3)	7(2)
H(21)C(25)	0.064(4)	0.116(4)	-0.088(4)	9(2)

TABLE 2.2 Selected bond lengths (Å)

a) cis-$PtCl_2(PMe_2C_6F_5)_2$ (I)

PT -	P(1)	2.240(1)
PT -	P(2)	2.231(1)
PT -	CL(1)	2.332(1)
PT -	CL(2)	2.355(1)
P(1) -	C(1)	1.802(5)
P(1) -	C(2)	1.808(7)
P(1) -	C(3)	1.827(4)
P(2) -	C(9)	1.808(6)
P(2) -	C(10)	1.809(6)
P(2) -	C(11)	1.838(5)
C(3) -	C(4)	1.393(8)
C(3) -	C(8)	1.391(7)
C(4) -	C(5)	1.370(7)
C(4) -	F(1)	1.352(6)
C(5) -	C(6)	1.365(9)
C(5) -	F(2)	1.346(7)
C(6) -	C(7)	1.349(9)
C(6) -	F(3)	1.336(7)
C(7) -	C(8)	1.372(8)
C(7) -	F(4)	1.344(7)
C(8) -	F(5)	1.337(8)
C(11) -	C(12)	1.387(6)
C(11) -	C(16)	1.395(7)
C(12) -	C(13)	1.380(7)
C(12) -	F(6)	1.336(6)
C(13) -	C(14)	1.362(8)
C(13) -	F(7)	1.345(6)
C(14) -	C(15)	1.358(8)
C(14) -	F(8)	1.340(6)
C(15) -	C(16)	1.379(7)
C(15) -	F(9)	1.336(6)
C(16) -	F(10)	1.339(5)

Table 2.2 (contd.)

b) <u>cis</u>-$Pt(CF_3)_2(PMe_2C_6F_5)_2$ (II)

PT	- P(1)	2.326(1)	C(5)	- C(6)	1.375(6)
PT	- C(9)	2.058(4)	C(5)	- F(2)	1.333(5)
P(1)	- C(1)	1.819(7)	C(6)	- C(7)	1.377(6)
P(1)	- C(2)	1.828(5)	C(6)	- F(3)	1.330(5)
P(1)	- C(3)	1.834(3)	C(7)	- C(8)	1.378(5)
C(3)	- C(4)	1.400(5)	C(7)	- F(4)	1.336(5)
C(3)	- C(8)	1.385(6)	C(8)	- F(5)	1.334(4)
C(4)	- C(5)	1.374(5)	C(9)	- F(6)	1.365(5)
C(4)	- F(1)	1.345(5)	C(9)	- F(7)	1.377(5)
C(9)	- F(8)	1.341(5)			

Table 2.2 (contd.)

c) $[Pt(CH_3)(PMe_2C_6F_5)_3](PF_6)$ (III)

PT - P(1)	2.324(1)	C(6) - F(3)	1.333(6)
PT - P(2)	2.327(1)	C(7) - C(8)	1.372(7)
PT - P(3)	2.296(1)	C(7) - F(4)	1.344(6)
PT - C(25)	2.098(5)	C(8) - F(5)	1.337(6)
P(1) - C(1)	1.803(6)	C(11) - C(12)	1.378(7)
P(1) - C(2)	1.810(6)	C(11) - C(16)	1.381(7)
P(1) - C(3)	1.847(5)	C(12) - C(13)	1.384(7)
P(2) - C(9)	1.821(6)	C(12) - F(6)	1.338(6)
P(2) - C(10)	1.809(7)	C(13) - C(14)	1.368(8)
P(2) - C(11)	1.844(5)	C(13) - F(7)	1.333(7)
P(3) - C(17)	1.812(8)	C(14) - C(15)	1.360(9)
P(3) - C(18)	1.811(7)	C(14) - F(8)	1.347(6)
P(3) - C(19)	1.836(5)	C(15) - C(16)	1.390(8)
P(4) - F(16)	1.569(5)	C(15) - F(9)	1.339(7)
P(4) - F(17)	1.584(5)	C(16) - F(10)	1.336(7)
P(4) - F(18)	1.589(5)	C(19) - C(20)	1.383(7)
P(4) - F(19)	1.581(5)	C(19) - C(24)	1.385(7)
P(4) - F(20)	1.557(5)	C(20) - C(21)	1.386(8)
P(4) - F(21)	1.527(5)	C(20) - F(11)	1.336(7)
C(3) - C(4)	1.384(7)	C(21) - C(22)	1.344(10)
C(3) - C(8)	1.390(7)	C(21) - F(12)	1.346(8)
C(4) - C(5)	1.374(8)	C(22) - C(23)	1.365(8)
C(4) - F(1)	1.340(6)	C(22) - F(13)	1.348(7)
C(5) - C(6)	1.367(8)	C(23) - C(24)	1.372(8)
C(5) - F(2)	1.339(7)	C(23) - F(14)	1.329(7)
C(6) - C(7)	1.366(8)	C(24) - F(15)	1.341(6)

TABLE 2.3 Selected interbond angles (°)

a) cis-$PtCl_2(PMe_2C_6F_5)_2$ (I)

P(2) - PT - P(1)	96.58(4)
CL(2) - PT - P(1)	84.91(5)
C(2) - P(1) - PT	109.17(23)
CL(1) - PT - P(2)	90.71(5)
C(9) - P(2) - PT	116.76(20)
C(11) - P(2) - PT	114.09(15)
C(2) - P(1) - C(1)	103.63(27)
C(3) - P(1) - C(2)	110.15(27)
C(8) - C(3) - P(1)	126.37(39)
C(11) - P(2) - C(9)	100.83(24)
C(12) - C(11) - P(2)	126.87(35)
C(8) - C(3) - C(4)	114.51(46)
F(1) - C(4) - C(3)	119.21(45)
F(5) - C(8) - C(3)	119.80(49)
C(6) - C(5) - C(4)	118.75(53)
F(2) - C(5) - C(6)	120.61(52)
F(3) - C(6) - C(5)	119.63(54)
C(8) - C(7) - C(6)	120.46(53)
F(4) - C(7) - C(8)	119.10(55)
C(16) - C(11) - C(12)	115.21(43)
F(6) - C(12) - C(11)	120.84(44)
F(10) - C(16) - C(11)	119.16(42)
C(14) - C(13) - C(12)	119.69(47)
F(7) - C(13) - C(14)	120.42(46)
F(8) - C(14) - C(13)	118.88(49)
C(16) - C(15) - C(14)	119.16(47)
F(9) - C(15) - C(16)	120.36(46)

CL(1) - PT - P(1)	172.61(5)
C(1) - P(1) - PT	123.87(18)
C(3) - P(1) - PT	109.62(16)
CL(2) - PT - P(2)	176.73(4)
C(10) - P(2) - PT	112.98(21)
CL(2) - PT - CL(1)	87.75(5)
C(3) - P(1) - C(1)	99.67(23)
C(4) - C(3) - P(1)	119.10(36)
C(10) - P(2) - C(9)	102.54(29)
C(11) - P(2) - C(10)	108.32(24)
C(16) - C(11) - P(2)	117.89(34)
C(5) - C(4) - C(3)	123.69(49)
C(7) - C(8) - C(3)	122.27(52)
F(1) - C(4) - C(5)	117.10(48)
F(2) - C(5) - C(4)	120.63(53)
C(7) - C(6) - C(5)	120.28(56)
F(3) - C(6) - C(7)	120.00(53)
F(4) - C(7) - C(6)	120.44(57)
F(5) - C(8) - C(7)	117.93(50)
C(13) - C(12) - C(11)	122.42(43)
C(15) - C(16) - C(11)	122.89(44)
F(6) - C(12) - C(13)	116.73(42)
F(7) - C(13) - C(12)	119.86(44)
C(15) - C(14) - C(13)	120.56(49)
F(8) - C(14) - C(15)	120.56(51)
F(9) - C(15) - C(14)	120.47(50)
F(10) - C(16) - C(15)	117.93(41)

Table 2.3 (contd.)

b) <u>cis</u>-Pt$(CF_3)_2(PMe_2C_6F_5)_2$ (II)

P(12) - PT - P(1)+	94.57(3)	C(9) - PT - P(1)	90.31(12)
C(92) - PT - P(1)+	166.00(13)	C(1) - P(1) - PT	114.27(19)
C(2) - P(1) - PT	111.99(15)	C(3) - P(1) - PT	119.60(12)
C(9) - PT - P(12)+	165.99(13)	C(92) - PT - P(12)+	90.29(12)
C(92) - PT - C(9)+	88.04(17)	F(6) - C(9) - PT	113.71(31)
F(7) - C(9) - PT	113.49(26)	F(8) - C(9) - PT	117.21(30)
C(2) - P(1) - C(1)	101.39(24)	C(3) - P(1) - C(1)	102.38(22)
C(3) - P(1) - C(2)	105.19(19)	C(4) - C(3) - P(1)	122.59(28)
C(8) - C(3) - P(1)	121.86(28)	C(8) - C(3) - C(4)	115.47(33)
C(5) - C(4) - C(3)	122.57(35)	F(1) - C(4) - C(3)	120.59(32)
C(7) - C(8) - C(3)	123.01(36)	F(5) - C(8) - C(3)	120.31(33)
F(1) - C(4) - C(5)	116.84(33)	C(6) - C(5) - C(4)	119.71(38)
F(2) - C(5) - C(4)	120.60(37)	F(2) - C(5) - C(6)	119.69(37)
C(7) - C(6) - C(5)	119.77(38)	F(3) - C(6) - C(5)	120.10(39)
F(3) - C(6) - C(7)	120.10(38)	C(8) - C(7) - C(6)	119.47(37)
F(4) - C(7) - C(6)	120.09(36)	F(4) - C(7) - C(8)	120.42(38)
F(5) - C(8) - C(7)	116.65(35)	F(7) - C(9) - F(6)	102.59(36)
F(8) - C(9) - F(6)	104.84(32)	F(8) - C(9) - F(7)	103.43(37)

+ C(92) and P(12) are related to C(9) and P(1) by the molecular two-fold axis.

Table 2.3 (contd.)

c) $[Pt(CH_3)(PMe_2C_6F_5)_2](PF_6)$ (III)

P(2) -	PT -	P(1)	93.82(5)
C(25) -	PT -	P(1)	85.30(17)
C(2) -	P(1) -	PT	113.66(20)
P(3) -	PT -	P(2)	99.60(5)
C(9) -	P(2) -	PT	122.70(21)
C(11) -	P(2) -	PT	114.10(17)
C(17) -	P(3) -	PT	125.03(22)
C(19) -	P(3) -	PT	109.08(17)
C(3) -	P(1) -	C(1)	98.61(24)
C(10) -	P(2) -	C(9)	100.03(30)
C(11) -	P(2) -	C(10)	108.19(26)
C(19) -	P(3) -	C(17)	100.58(25)
F(17) -	P(4) -	F(16)	89.39(26)
F(19) -	P(4) -	F(16)	90.71(26)
F(21) -	P(4) -	F(16)	90.11(35)
F(19) -	P(4) -	F(17)	179.77(28)
F(21) -	P(4) -	F(17)	88.97(33)
F(20) -	P(4) -	F(18)	87.47(30)
F(20) -	P(4) -	F(19)	90.30(25)
F(21) -	P(4) -	F(20)	94.03(34)
P(3) -	PT -	P(1)	166.46(5)
C(1) -	P(1) -	PT	119.05(21)
C(3) -	P(1) -	PT	114.44(16)
C(25) -	PT -	P(2)	175.85(17)
C(10) -	P(2) -	PT	112.96(21)
C(25) -	PT -	P(3)	81.18(17)
C(18) -	P(3) -	PT	112.82(25)
C(2) -	P(1) -	C(1)	101.76(29)
C(3) -	P(1) -	C(2)	107.47(23)
C(11) -	P(2) -	C(9)	96.74(25)
C(18) -	P(3) -	C(17)	99.50(36)
C(19) -	P(3) -	C(18)	108.32(27)
F(18) -	P(4) -	F(16)	88.40(31)
F(20) -	P(4) -	F(16)	175.72(33)
F(18) -	P(4) -	F(17)	91.00(29)
F(20) -	P(4) -	F(17)	89.58(25)
F(19) -	P(4) -	F(18)	88.79(28)
F(21) -	P(4) -	F(18)	178.51(34)
F(21) -	P(4) -	F(19)	91.25(32)

Table 2.3 c) (contd.)

C(4) - C(3) - P(1)	117.99(36)	C(8) - C(3) - P(1)	125.70(36)
C(12) - C(11) - P(2)	116.55(38)	C(16) - C(11) - P(2)	127.39(40)
C(20) - C(19) - P(3)	126.56(40)	C(24) - C(19) - P(3)	117.76(37)
C(8) - C(3) - C(4)	116.21(43)	C(5) - C(4) - C(3)	122.14(47)
F(1) - C(4) - C(3)	119.10(43)	C(7) - C(8) - C(3)	121.98(46)
F(5) - C(8) - C(3)	121.34(42)	F(1) - C(4) - C(5)	118.75(44)
C(6) - C(5) - C(4)	119.92(51)	F(2) - C(5) - C(4)	120.08(49)
F(2) - C(5) - C(6)	119.98(51)	C(7) - C(6) - C(5)	119.68(49)
F(3) - C(6) - C(5)	120.10(55)	F(3) - C(6) - C(7)	120.21(51)
C(8) - C(7) - C(6)	120.05(48)	F(4) - C(7) - C(6)	119.78(44)
F(4) - C(7) - C(8)	120.13(46)	F(5) - C(8) - C(7)	116.65(43)
C(16) - C(11) - C(12)	115.99(47)	C(13) - C(12) - C(11)	123.01(49)
F(6) - C(12) - C(11)	119.57(43)	C(15) - C(16) - C(11)	122.28(52)
F(10) - C(16) - C(11)	121.28(48)	F(6) - C(12) - C(13)	117.42(45)
C(14) - C(13) - C(12)	118.70(51)	F(7) - C(13) - C(12)	120.76(49)
F(7) - C(13) - C(14)	120.50(48)	C(15) - C(14) - C(13)	120.74(53)
F(8) - C(14) - C(13)	118.65(54)	F(8) - C(14) - C(15)	120.51(56)
C(16) - C(15) - C(14)	119.27(56)	F(9) - C(15) - C(14)	120.36(53)
F(9) - C(15) - C(16)	120.36(53)	F(10) - C(16) - C(15)	116.37(50)
C(24) - C(19) - C(20)	115.68(45)	C(21) - C(20) - C(19)	121.38(53)
F(11) - C(20) - C(19)	120.82(47)	C(23) - C(24) - C(19)	123.51(49)
F(15) - C(24) - C(19)	119.30(44)	F(11) - C(20) - C(21)	117.80(51)
C(22) - C(21) - C(20)	120.24(56)	F(12) - C(21) - C(20)	118.71(58)
F(12) - C(21) - C(22)	121.05(52)	C(23) - C(22) - C(21)	120.91(54)
F(13) - C(22) - C(21)	119.89(57)	F(13) - C(22) - C(23)	119.19(60)
C(24) - C(23) - C(22)	118.25(54)	F(14) - C(23) - C(22)	120.95(54)
F(14) - C(23) - C(24)	120.77(51)	F(15) - C(24) - C(23)	117.19(47)

TABLE 2.4 Selected torsion angles (^{o})

a) cis-$PtCl_2(PMe_2C_6F_5)_2$ (I)

P(2) -	PT -	P(1) -	C(1)	15.9(2)	P(2) -	PT -	P(1) -	C(2)	-106.4(2)
P(2) -	PT -	P(1) -	C(3)	132.9(2)	CL(1) -	PT -	P(1) -	C(1)	-173.7(4)
CL(1) -	PT -	P(1) -	C(2)	64.0(4)	CL(1) -	PT -	P(1) -	C(3)	-56.7(4)
CL(2) -	PT -	P(1) -	C(1)	-167.0(2)	CL(2) -	PT -	P(1) -	C(2)	70.7(2)
CL(2) -	PT -	P(1) -	C(3)	-50.1(2)	P(1) -	PT -	P(2) -	C(9)	-177.6(2)
P(1) -	PT -	P(2) -	C(10)	63.9(2)	P(1) -	PT -	P(2) -	C(11)	-60.4(2)
CL(1) -	PT -	P(2) -	C(9)	3.7(2)	CL(1) -	PT -	P(2) -	C(10)	-114.9(2)
CL(1) -	PT -	P(2) -	C(11)	120.8(2)	CL(2) -	PT -	P(2) -	C(9)	65.6(9)
CL(2) -	PT -	P(2) -	C(10)	-53.0(9)	CL(2) -	PT -	P(2) -	C(11)	-177.3(8)
PT -	P(1) -	C(3) -	C(4)	-50.2(4)	PT -	P(1) -	C(3) -	C(8)	131.5(4)
C(1) -	P(1) -	C(3) -	C(4)	81.2(4)	C(1) -	P(1) -	C(3) -	C(8)	-97.1(5)
C(2) -	P(1) -	C(3) -	C(4)	-170.4(4)	C(2) -	P(1) -	C(3) -	C(8)	11.4(6)
PT -	P(2) -	C(11) -	C(12)	125.6(4)	PT -	P(2) -	C(11) -	C(16)	-58.6(4)
C(9) -	P(2) -	C(11) -	C(12)	-108.4(5)	C(9) -	P(2) -	C(11) -	C(16)	67.4(4)
C(10) -	P(2) -	C(11) -	C(12)	-1.1(5)	C(10) -	P(2) -	C(11) -	C(16)	174.6(4)

Table 2.4 (cont.)

b) cis-Pt$(CF_3)_2(PMe_2C_6F_5)_2$ (II)

P(12) -	PT -	P(1) -	C(1)	158.0(2)
P(12) -	PT -	P(1) -	C(3)	-80.2(1)
C(9) -	PT -	P(1) -	C(2)	-149.7(2)
C(92) -	PT -	P(1) -	C(1)	48.0(6)
C(92) -	PT -	P(1) -	C(3)	169.8(5)
P(1) -	PT -	C(9) -	F(7)	-38.6(3)
P(12) -	PT -	C(9) -	F(6)	-44.8(7)
P(12) -	PT -	C(9) -	F(8)	-167.5(3)
C(92) -	PT -	C(9) -	F(7)	155.3(3)
PT -	P(1) -	C(3) -	C(4)	-9.1(4)
C(1) -	P(1) -	C(3) -	C(4)	118.4(4)
C(2) -	P(1) -	C(3) -	C(4)	-136.0(3)

P(12) -	PT -	P(1) -	C(2)	43.4(2)
C(9) -	PT -	P(1) -	C(1)	-35.1(2)
C(9) -	PT -	P(1) -	C(3)	86.6(2)
C(92) -	PT -	P(1) -	C(2)	-66.6(5)
P(1) -	PT -	C(9) -	F(6)	-155.4(3)
P(1) -	PT -	C(9) -	F(8)	81.9(3)
P(12) -	PT -	C(9) -	F(7)	72.0(6)
C(92) -	PT -	C(9) -	F(6)	33.5(3)
C(92) -	PT -	C(9) -	F(8)	-84.2(3)
PT -	P(1) -	C(3) -	C(8)	174.5(3)
C(1) -	P(1) -	C(3) -	C(8)	-58.0(4)
C(2) -	P(1) -	C(3) -	C(8)	47.6(4)

Table 2.4 (contd.)

c) $[Pt(CH_3)(PMe_2C_6F_5)_3](PF_6)$ (III)

P(2) -	PT -	P(1) -	C(1)	175.0(2)
P(2) -	PT -	P(1) -	C(3)	-68.9(2)
P(3) -	PT -	P(1) -	C(2)	-117.1(3)
C(25) -	PT -	P(1) -	C(1)	-1.0(3)
C(25) -	PT -	P(1) -	C(3)	115.2(2)
P(1) -	PT -	P(2) -	C(10)	-79.0(2)
P(3) -	PT -	P(2) -	C(9)	-20.6(2)
P(3) -	PT -	P(2) -	C(11)	-136.7(2)
C(25) -	PT -	P(2) -	C(10)	-1.4(23)
P(1) -	PT -	P(3) -	C(17)	178.6(3)
P(1) -	PT -	P(3) -	C(19)	59.9(3)
P(2) -	PT -	P(3) -	C(18)	127.4(3)
C(25) -	PT -	P(3) -	C(17)	-177.6(3)
C(25) -	PT -	P(3) -	C(19)	63.7(2)
PT -	P(1) -	C(3) -	C(8)	127.4(4)
C(1) -	P(1) -	C(3) -	C(8)	-105.2(5)
C(2) -	P(1) -	C(3) -	C(8)	0.2(5)
PT -	P(2) -	C(11) -	C(16)	-123.8(5)
C(9) -	P(2) -	C(11) -	C(16)	105.8(5)
C(10) -	P(2) -	C(11) -	C(16)	2.9(6)
PT -	P(3) -	C(19) -	C(24)	69.1(4)
C(17) -	P(3) -	C(19) -	C(24)	-64.0(5)
C(18) -	P(3) -	C(19) -	C(24)	-167.8(4)

P(2) -	PT -	P(1) -	C(2)	55.1(2)
P(3) -	PT -	P(1) -	C(1)	2.8(3)
P(3) -	PT -	P(1) -	C(3)	118.9(2)
C(25) -	PT -	P(1) -	C(2)	-120.8(3)
P(1) -	PT -	P(2) -	C(9)	161.3(2)
P(1) -	PT -	P(2) -	C(11)	45.2(2)
P(3) -	PT -	P(2) -	C(10)	99.2(2)
C(25) -	PT -	P(2) -	C(9)	-121.1(23)
C(25) -	PT -	P(2) -	C(11)	122.8(23)
P(1) -	PT -	P(3) -	C(18)	-60.5(3)
P(2) -	PT -	P(3) -	C(17)	6.5(3)
P(2) -	PT -	P(3) -	C(19)	-112.2(2)
C(25) -	PT -	P(3) -	C(18)	-56.7(3)
PT -	P(1) -	C(3) -	C(4)	-56.2(4)
C(1) -	P(1) -	C(3) -	C(4)	71.3(4)
C(2) -	P(1) -	C(3) -	C(4)	176.5(4)
PT -	P(2) -	C(11) -	C(12)	59.3(4)
C(9) -	P(2) -	C(11) -	C(12)	-71.2(4)
C(10) -	P(2) -	C(11) -	C(12)	-174.1(4)
PT -	P(3) -	C(19) -	C(20)	-110.7(5)
C(17) -	P(3) -	C(19) -	C(20)	116.3(5)
C(18) -	P(3) -	C(19) -	C(20)	12.5(6)

TABLE 2.5 Selected intramolecular non-bonding distances (Å)

a) *cis*-$PtCl_2(PMe_2C_6F_5)_2$ (I)

Pt	···	F(1)	3.19	Cl(1)	···	C(9)	3.16
Cl(1)	···	H(7)C(9)	2.83	Cl(1)	···	H(8)C(9)	2.96
Cl(2)	···	C(3)	3.21				

b) *cis*-$Pt(CF_3)_2(PMe_2C_6F_5)_2$ (II)

Pt	···	F(1)	3.16	Pt	···	F(7)	2.90
Pt	···	F(6)	2.89	Pt	···	F(8)	2.92
P(1)	···	F(7)	3.04	F(7)	···	H(1)C(1)	2.34
F(6)	···	C(92)	2.86	C(1)	···	C(9)	3.30

c) $[Pt(CH_3)(PMe_2C_6F_5)_3](PF_6)$ (III)

C(1)	···	C(25)	3.07	C(18)	···	H(21)C(25)	2.75
C(25)	···	H(18)C(18)	2.58	P(1)	···	H(19)C(25)	3.05
C(17)	···	H(9)C(9)	2.73	P(1)	···	H(20)C(25)	3.01
P(3)	···	H(21)C(25)	2.67				

TABLE 2.6 Intermolecular distances less than sum of van der Waals radii (Å).

Symmetry operations: (i) $x,\frac{1}{2}-y,\frac{1}{2}+z$; (ii) $1-x,-\frac{1}{2}+y,\frac{1}{2}-z$; (iii) $-x,-1+y,\frac{1}{2}-z$; (iv) $-\frac{1}{2}+x,\frac{1}{2}-y,-\frac{1}{2}+z$; (v) $\frac{1}{2}+x,\frac{1}{2}-y,\frac{1}{2}+z$; (vi) $\frac{1}{2}-x,-\frac{1}{2}+y,\frac{1}{2}-z$; (vii) $1-x,-y,-z$.

a) *cis*-$PtCl_2(PMe_2C_6F_5)_2$ (I)

Cl(1)	... $F(6)^{i}$	3.08	Cl(1)	... $H(1)C(1)^{i}$	2.86
Cl(2)	... $H(3)C(1)^{ii}$	2.95			

b) *cis*-$Pt(CF_3)_2(PMe_2C_6F_5)_2$ (II)

F(6)	... $H(6)C(2)^{ii}$	2.50	F(8)	... $H(5)C(2)^{iii}$	2.53

c) $Pt(CH_3)(PMe_2C_6F_5)_3(PF_6)$ (III)

F(12)	... $H(11)C(10)^{iv}$	2.54	F(17)	... $H(3)C(1)^{v}$	2.52
F(17)	... $H(4)C(2)^{vi}$	2.53	F(19)	... $H(13)C(17)^{vii}$	2.45

TABLE 2.7 Deviations of the atoms (Å$\times 10^3$) from, and equations of, the weighted least-squares platinum co-ordination planes. X,Y,Z refer to an orthogonal basis set defined by a*, b and c.

a) cis-$PtCl_2(PMe_2C_6F_5)_2$ (I) [Plane defined by Pt,P(1),P(2),Cl(1) and Cl(2)]

Pt -2(1), P(1) 19(1), P(2) 48(1), Cl(1) 19(1), Cl(2) 48(1)

0.146X-0.712Y-0.687Z = -3.616

b) cis-$Pt(CF_3)_2(PMe_2C_6F_5)_2$ (II) [Plane defined by Pt,P(1),P(12),C(9) and C(92)]

Pt 0(1),P(1) -13(1),C(9) 458(1),P(12) 13(1),C(92) -458(5)

-0.806X+0.000Y-0.592Z=-2.145

c) $Pt(CH_3)(PMe_2C_6F_5)_3$ (PF_6) (III) [Plane defined by Pt,P(1),P(2),P(3) and C(25)]

Pt -2(1),P(1) 30(2),P(2) 15(2),P(3) 37(2),C(25) 132(5)

0.166X+0.637Y-0.753Z=2.160

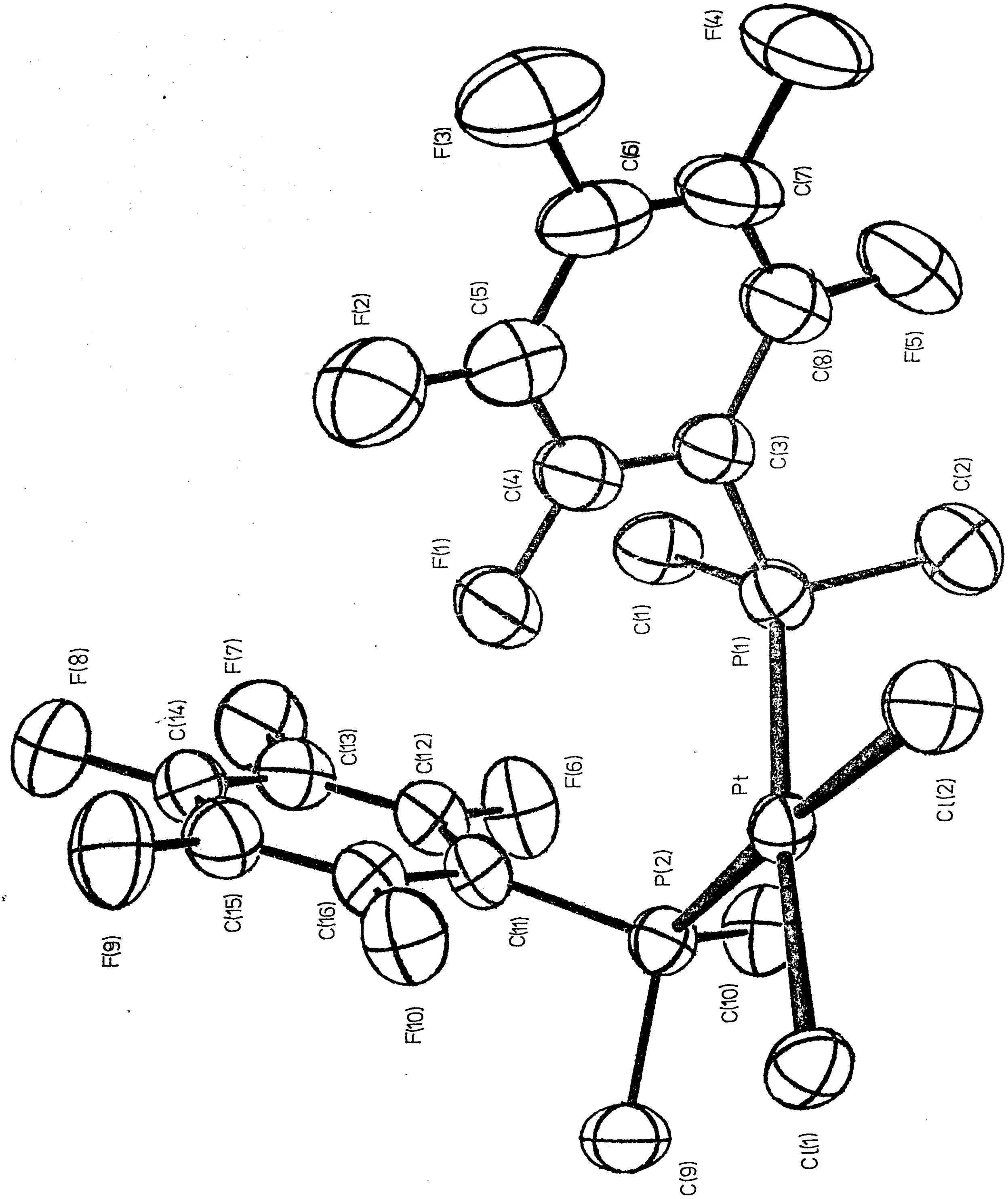
Legends

FIGURE 2.1 A perspective view of the molecular structure of cis-$PtCl_2(PMe_2C_6F_5)_2$, (I), showing the atomic numbering scheme. The vibrational ellipsoids display 50% probability. For clarity hydrogen atoms are omitted.

FIGURE 2.2 A perspective view of the molecular structure of cis-$Pt(CF_3)_2(PMe_2C_6F_5)_2$, (II), showing the atomic numbering scheme. The vibrational ellipsoids display 50% probability. For clarity hydrogen atoms are omitted.

FIGURE 2.3 A perspective view of the cation of $[Pt(CH_3)(PMe_2C_6F_5)_3]^+$, (III), showing the atomic numbering scheme. The vibrational ellipsoids of the Pt, P and C(25) atoms display 50% probability. For clarity atoms C(1)-C(24) are represented by spheres of arbitrary size. Hydrogen atoms are omitted.

FIGURE 2.1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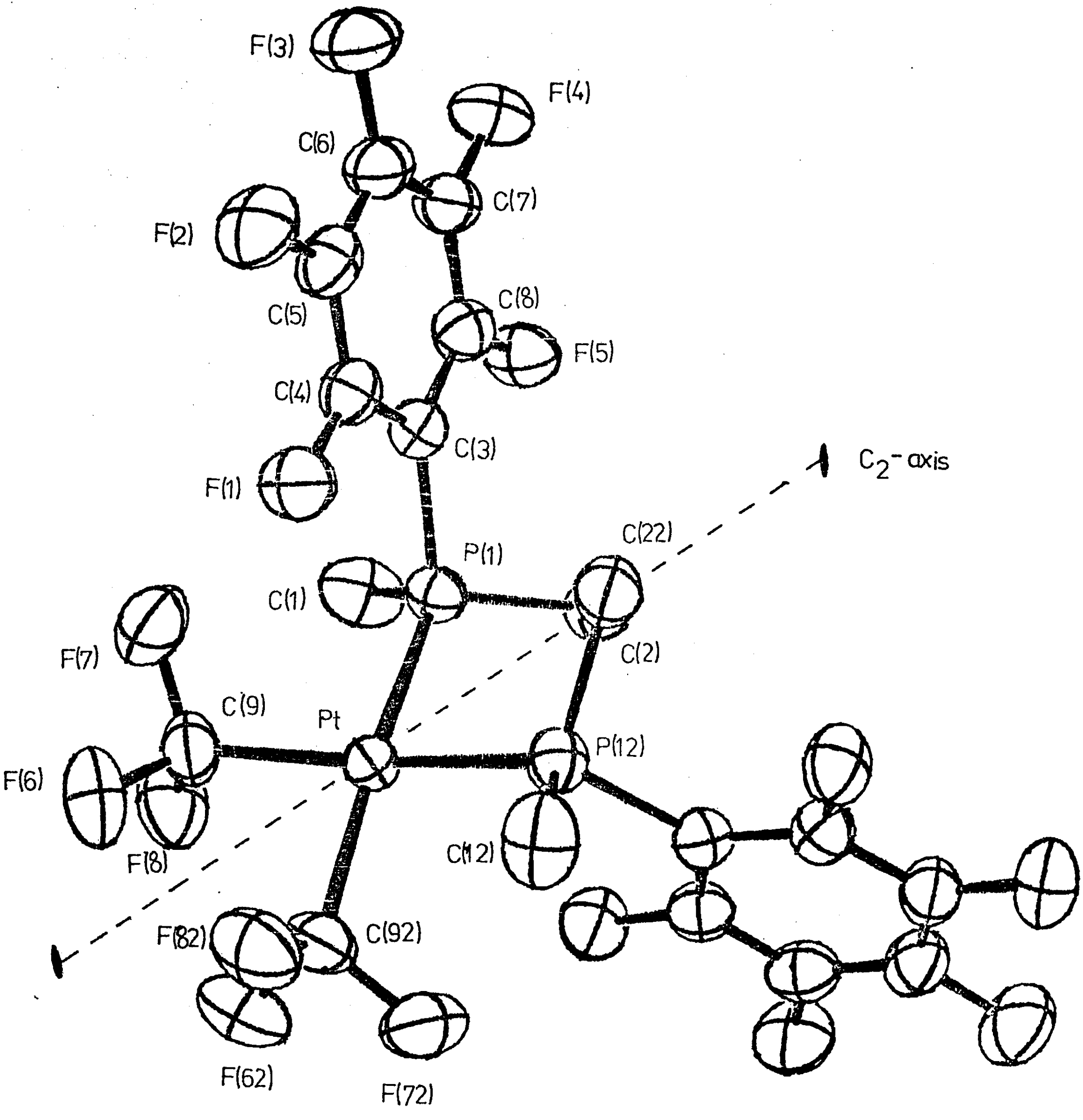
F(3)
C(6)
C(7)
C(5)
F(2)
F(5)
C(8)
C(3)
C(4)
C(2)
F(1)
C(1)
P(1)
F(8)
F(7)
C(14)
C(13)
C(12)
F(6)
Pt
Cl(2)
P(2)
C(15)
C(16)
C(11)
F(9)
F(10)
C(10)
Cl(1)
C(9)

FIGURE 2.2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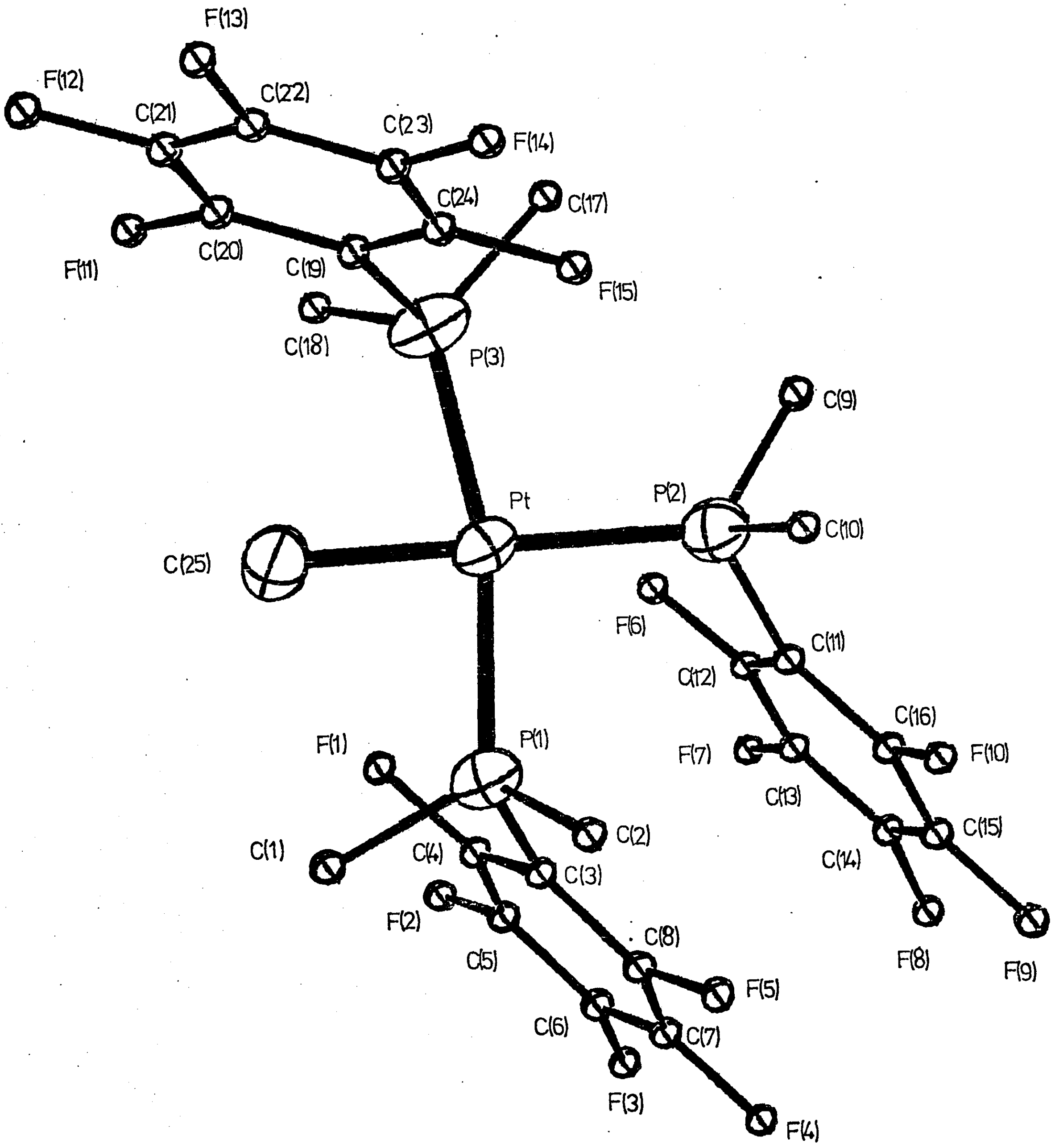
F(4)
C(6)
C(7)
F(2)
C(8)
C(5)
F(5)
C(4)
C(3)
C₂-axis
F(1)
C(22)
P(1)
C(1)
F(7)
C(2)
C(9)
Pt
P(12)
F(6)
C(12)
F(8)
F(82)
C(92)
F(62)
F(72)

FIGURE 2.3


F(13)
F(12)
C(22)
C(21)
C(23)
F(14)
C(24)
C(17)
C(20)
C(19)
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F(15)
C(18)
P(3)
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Pt
P(2)
C(10)
C(25)
F(6)
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C(12)
C(16)
F(1)
P(1)
F(7)
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F(5)
F(8)
F(9)
C(6)
C(7)
F(3)
F(4)

2.3 Discussion

Crystals of I, II, and III are built of discrete molecules. In each structure the shortest intermolecular distances are accounted for by the sum of the appropriate van der Waals radii (Table 2.6).

Metal atom co-ordination

In each molecule overcrowding induced by the sterically demanding $PMe_2C_6F_5$ ligands leads to minor distortions of the platinum co-ordination from an exactly square-planar arrangement. This is most prominent in III.

In I the phosphine ligands adopt similar conformations (see below) which result in the methyl carbon atoms C(1) and C(9) lying close to the metal co-ordination plane. The phosphine ligands interact through methyl carbon atoms C(1) and C(10). Opening of the P(1)-Pt-P(2) and Pt-P(1)-C(1) angles [respectively 96.58(4) and 123.9(2)°] leads to a minimum inter-ligand H•••H separation of 2.4Å for the hydrogen atoms attached to C(1) and C(10). The steric environments of the two chlorine atoms differ: Cl(1) is 3.16Å from the C(9) methyl carbon, whereas Cl(2) is 3.21Å from the C(3) phenyl carbon atom. This may account for slight differences in length between chemically equivalent metal-ligand bonds [Pt-P 2.231(1) and 2.240(1)Å; Pt-Cl 2.332(1) and 2.355(1)Å].

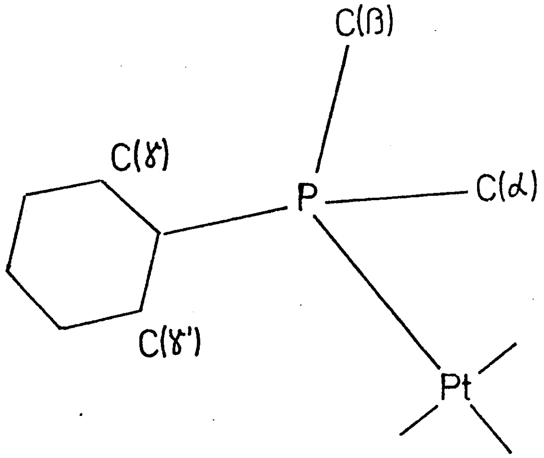
The platinum atom in II lies on a two-fold axis. The P-Pt-P angle is 4.57(3)$^{\circ}$ greater than 90°, thus helping to relieve the C(2)•••C(22) contact of 3.6Å. The C(1)•••C(9) intramolecular contact 3.30Å is also rather short. The Pt-P and Pt-Cl distances are 2.326(1) and 2.058(4)Å, respectively. The valency angles at the donor carbon atom C(9) are distorted from a regular tetrahedral arrangement. The mean Pt-C-F and F-C-F angles [114.8(7) and 103.6(4)$^{\circ}$] are consistent with more than 25% carbon s-character in the Pt-C bond. The C(9)-F distances [mean 1.361(6)Å] agree well with values of 1.35 and 1.36(2)Å found in $Hg(CF_3)_2$[72] and $[\mu-(CMe)_4(CF_3)-Pt(PMe_2Ph)_2](SbF_6)$.[73]

The geometry of the anion PF_6^-, in III, is as expected (Tables 2.2,2.3).The phosphine ligands in III, as in I, adopt conformations which bring methyl carbon atoms [C(1),C(9), and C(17)] close to the metal co-ordination plane. The phosphines containing the atoms P(1) and P(2) interact through their parallel phenyl groups, which approach each other to give a minimum C•••C separation of 3.23Å The closest inter-ligand approach between the phosphines containing P(2) and P(3) atoms involves the methyl carbon atoms [C(9)•••C(17) 3.22Å]. Steric strain is also evident in the valency angles of 81.2(2)-99.6(1)$^{\circ}$, subtended at platinum by <u>cis</u>-ligands (see Figure 2.5,p.79), and in the displacement of the donor carbon atom C(25) from the platinum co-ordination plane by 0.14Å. The Pt-P(<u>trans</u> to P) distances [2.296(1) and 2.324(1)Å] differ significantly, possibly (as in I) as a consequence of different steric environments. The Pt-P(trans to C) and Pt-C bond lengths are 2.327(1) and 2.098(5)Å.

Geometry of the $PMe_2C_6F_5$ ligands

The three structures contain altogether six $PMe_2C_6F_5$ ligands. The ligands in the compounds I and III adopt similar conformations: one methyl carbon ($C\alpha$) lies in the metal co-ordination plane, the other ($C\beta$) in the plane of the C_6F_5 group, adjacent to $C\gamma$. The weighted mean valency angles* for the five $PMe_2C_6F_5$ ligands of I and III are presented below:

Pt	-P	-C(Ph)	112(1)°
Pt	-P	-C(α)	122(2)°
Pt	-P	-C(β)	112(1)°
C(α)	-P	-C(Ph)	99(1)°
C(β)	-P	-C(Ph)	108.5(4)°
P	-C(Ph)	-C(γ)	126.6(4)°
P	-C(Ph)	-C(γ')	117.9(4)°



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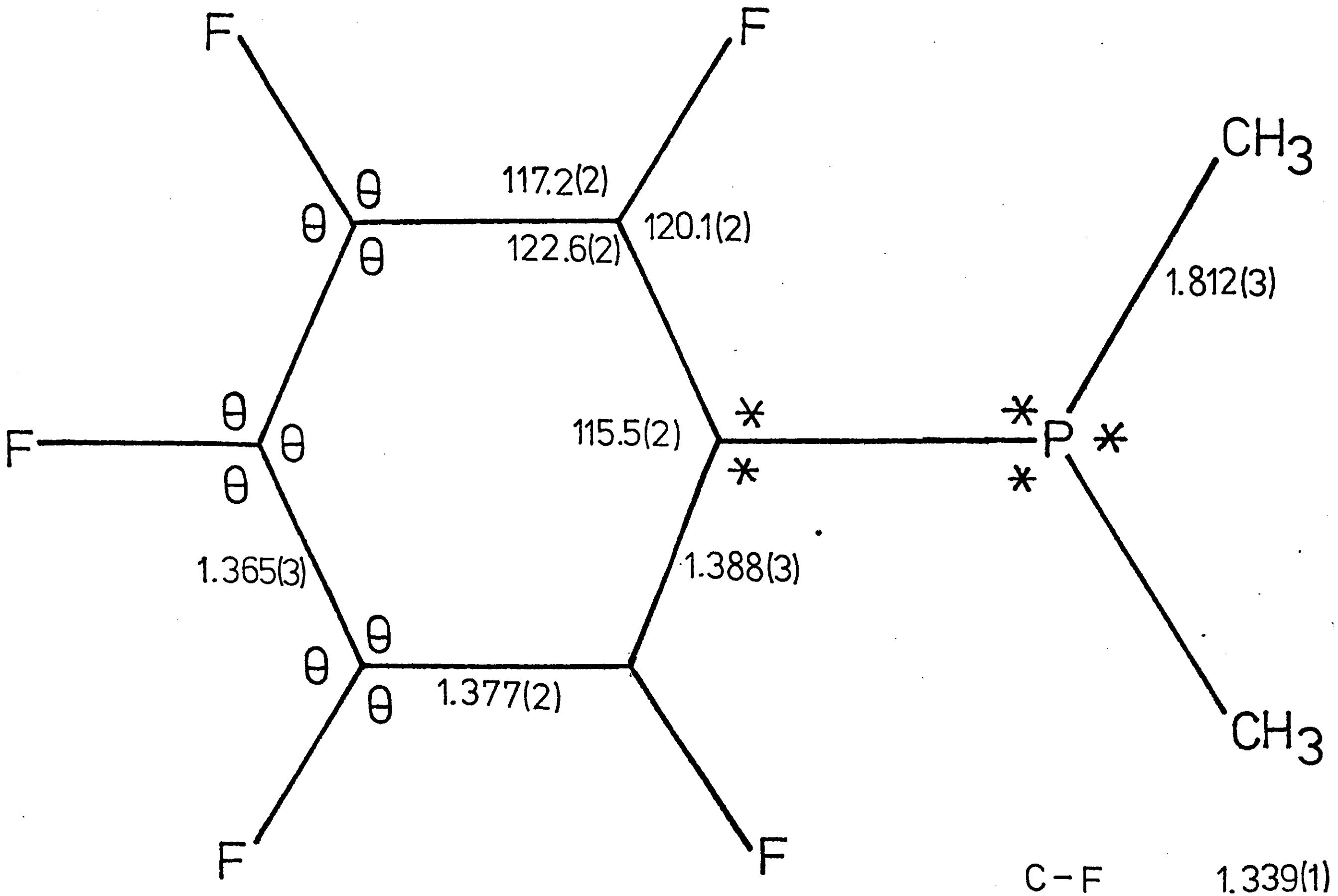
* Throughout this thesis the mean of a set of N parameters, x_i, is defined as $\sum_i w_i x_i / \sum_i w_i$ where $w_i=1$ for an unweighted mean and $w_i=\sigma_i^{-2}$ (σ_i being the standard deviation of x_i) for a weighted mean. The standard deviation of mean, $\bar{x}$, is $\{\sum_i w_i(x_i-\bar{x})^2/(N-1)\sum_i w_i\}^{\frac{1}{2}}$ or, for weighted means only, $\{\sum_i w_i\}^{-\frac{1}{2}}$ if larger than former value. All sums are over the N individual measurements.

In the compound II a more symmetrical conformation is adopted, with the platinum atom lying in the plane of the phenyl ring, which in turn is approximately normal to the metal co-ordination plane. The Pt-P-C(methyl) angles [112.0(2) and 114.3(2)°] are more nearly equal than the corresponding angles in I and III, as are the C(Ph)-P-C(methyl) [102.4(2) and 105.2(2)°] and P-C(Ph)-C(Ph) angles [122.6(3) and 121.9(3)°].

Valency angles at phosphorus and ipso-carbon atoms thus reflect the conformations of the $PMe_2C_6F_5$ ligands. Other bond lengths and valency angles in the six ligands agree well. Weighted means are presented in Figure 2.4. The deviations of the individual bond lengths and angles in I, II and III from the weighted means in Figure 2.4 lead to a value of χ^2 of 445 on 275 degrees of freedom. The differences are not significant at the 5% level. The standard deviation of an observation of unit weight is 1.27, satisfactorily close to its expectation value of unity, thus suggesting that the standard deviations derived from the least-squares refinement are realistic. On the most pessimistic assumption they should be increased by 30%.

The rather acute internal ring angle at the ipso-carbon atoms in I, II and III [weighted mean 115.5(2)°] is a common feature of structures containing C_6F_5X groups (Table 2.8).

TABLE 2.4 Weighted bond lengths (Å) and angles for six $PMe_2C_6F_5$ ligands in I,II, and III.



C-F	1.339(1)
C-H	0.96(1)
P-C-H	109.4(6)
H-C-H	109.5(10)
θ	120

*These angles are dependent upon conformation

TABLE 2.8 Comparison between mean bond lengths (Å) and angles (°) in crystal structures containing the group C_6F_5X.

	P-C	C-C	C-F	C-C-C(at X)	Ref.
$Hg(C_6F_5)_2$	-	1.38	1.39	116.4	87
$(PC_6F_5)_4$	1.838(3)	1.38(1)	1.34(1)	115.2(3)	74
$Pt(PBu_3)_2(SC_6F_5)_2$	-	1.37(5)	1.34(5)	118(3)	75
$[Pd(PPh_3)(SC_6F_5)_2]_2$ a)	-	1.37(7)	1.38(6)	111(4)	76
$[Pd(PPh_3)(SC_6F_5)_2]_2$ a)	-	1.39(5)	1.38(4)	114(3)	77
$Pt(PPh_3)_2(PC_6F_5)_2$	1.85(2)	1.37(3)	1.35(2)	113(2)	78
I, II, III b)	1.836(3)	1.38(1)	1.339(1)	115.5(2)	This work

a) This compound exists in two different crystalline forms.

b) Weighted means for the six $PMe_2C_6F_5$ ligands in I, II and III.

It is thought to reflect electron donation from X to the ring and has been explained either in terms of hybridization of the *ipso*-carbon atom or by the electron pair repulsion (VSEPR) theory.[79] The weighted mean P-C distance in I, II and III [1.836(3)Å] is similar to the corresponding values in other C_6F_5P systems (Table 2.8) and, perhaps surprisingly, also agrees well with a mean value of 1.828(3)Å for $P-C_6H_5$ distances, obtained from an extensive literature survey.[79]

Trans-influence on Pt-$PMe_2C_6F_5$ bonds

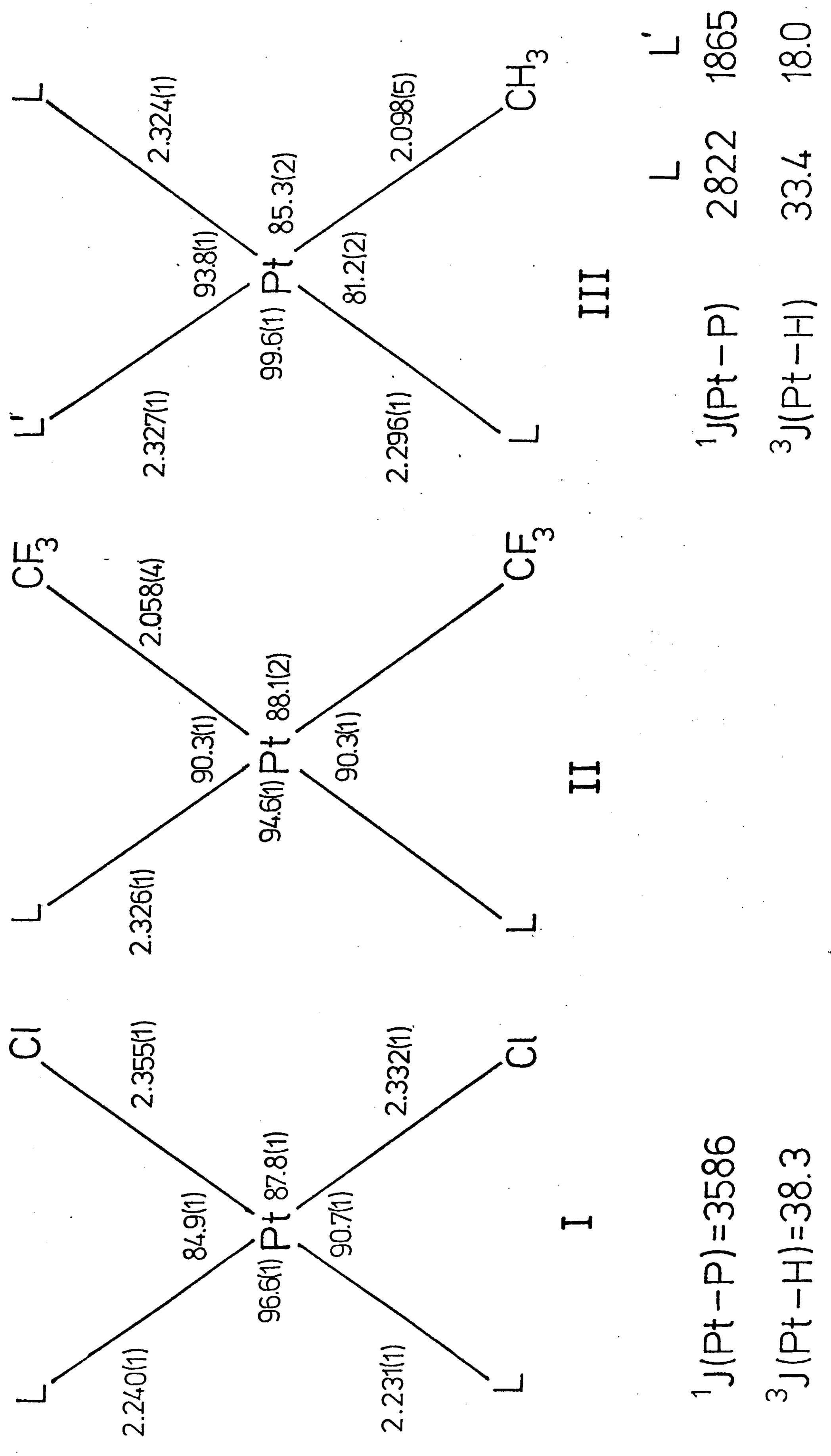
As already noted variations of up to 0.03Å in chemically equivalent Pt-$PMe_2C_6F_5$ bond lengths occur in I and III, possibly for steric reasons. Nevertheless, the mean Pt-P bond lengths lead to the *trans*-influence series $Cl < PMe_2C_6F_5 < CH_3 \sim CF_3$. The same ordering may be obtained from ^1J(Pt-P) and ^3J(Pt-H) coupling constants presented in Figure 2.5 (excluding CF_3 ligand in II for which n.m.r. parameters could not be obtained due to insolubility of the compound in common solvents). However, the coupling constants suggest a greater difference between the P- and C-donor ligands than is apparent from the bond lengths (Figure 2.5). In terms of the discussion in the Introduction to Part II (see above) this would seem to imply that the coupling constants are more sensitive to the platinum 6*s* components in the Pt-P bonds than are the bond lengths.

Effect of phosphine substituents on Pt-$PMe_2C_6F_5$ bonds

The trends observed in Pt-P bond lengths and coupling constants for I, II and III are closely comparable with those found in complexes containing phosphines with hydrocarbon substituents.

Thus the ^1J(Pt-P) coupling constants in I and III are typically only 3% larger than those for analogous PMe_2Ph complexes,[80] and the *trans*-influence series $Cl < PMe_2Ph < CF_3 < CH_3$

FIGURE 2.5 Bond lengths (Å) and angles (°) and coupling constants(Hz) in I,II,and III. L=L'=$PMe_2C_6F_5$.The insolubility of II in common solvents precluded n.m.r. experiments.



has been derived from ${}^3J$(Pt-P-C-H) couplings in the cationic species $[PtX(PMe_2Ph)_3]^+$.[81]

The mean Pt-Cl and Pt-P bond lengths in cis-$PtCl_2(PMe_2Ph)_2$ [2.362(3) and 2.260(2)Å][82] are each ca. 0.02Å longer than corresponding distances in I. This difference, though not dramatic, is probably a consequence of the difference in electronic properties of C_6F_5 and C_6H_5. Mean metal-ligand bond lengths in complexes with cis-$PtCl_2P_2$ donor sets are summarised in Table 2.9. Both the Pt-P and Pt-Cl distances show a roughly linear dependence on Tolman's $\sum_i \chi_i$ parameter, (see Introduction to Part II of this thesis) which measures the electron-withdrawing ability of the substituents on the phosphorus atom (Figure 2.6). The rate of variation of the Pt-Cl(trans to P) bond lengths is about half that of the Pt-P distances.

Rationalisation of these trends can be offered from two points of view. Electron-withdrawal at the phosphorus donor atom would be expected to contract the phosphorus σ-donor orbital, thus shortening the Pt-P bond. A less basic phosphine might also be expected to have a low trans-influence, thus leading to a shorter trans-Pt-Cl bond.

Alternatively, the observed changes in Pt-P and Pt-Cl (trans to P) bond lengths can be understood in terms of π-backbonding. Electron-withdrawal by substituents on phosphorus is expected to enhance $d_\pi - d_\pi$ backdonation from metal to phosphorus. Loss of charge from the metal would lead to a stronger electrostatic interaction between platinum and the trans chlorine atom. This would explain

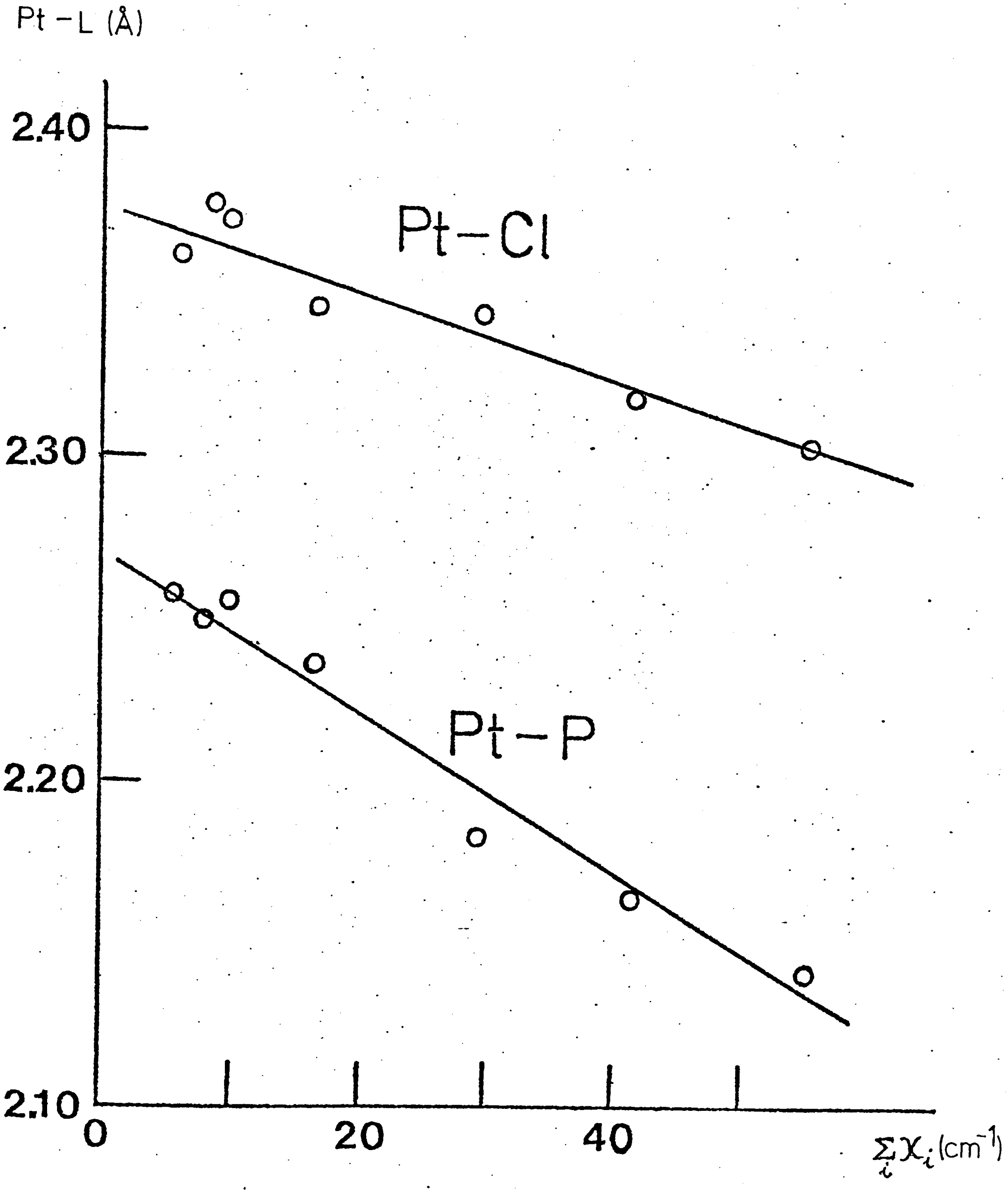
TABLE 2.9 Mean bond lengths (Å) in complexes with cis-$PtCl_2P_2$ donor sets

Compound	Pt-P	Pt-Cl	Cone angle(°)[a]	$\sum_i \chi_i$[b]	Ref.
cis-$PtCl_2(PEt_3)_2$	2.258(2)	2.361(8)	130	5.4	c
cis-$PtCl_2(PMe_2Ph)_2$	2.260(2)	2.362(3)	127	9.5	82
cis-$PtCl_2(PMe_3)_2$	2.248(9)	2.376(12)	118	7.8	83
cis-$PtCl_2(PMe_2C_6F_5)_2$	2.236(6)	2.344(12)	130	16.4	d
cis-$PtCl_2(PEt_3)[P'(OPh)_3]$[e]	2.182(2)	2.344(2)	128	29.1	84
cis-$PtCl_2[(F_3C)P'CH_2CH_2PPh_2]$[e]	2.168(3)	2.317(3)	119	41.0	69
cis-$PtCl_2(PEt_3)(P'F_3)$[e]	2.141(3)	2.305(3)	104	54.6	85

[a] Tolman's cone angle, see ref. 66 [b] Ref. 63 [c] Part II, Ch.3 of this thesis. [d] Compound I, this Chapter

[e] Pt-P and Pt-Cl (trans to P) bonds refer to primed phosphorus atom

FIGURE 2.6 Variation in the Pt-P and Pt-Cl(*trans* to P) bond lengths in complexes with *cis*-$PtCl_2P_2$ donor sets with $\sum_i \chi_i$, a measure of the electron-withdrawing ability of the phosphine substituents



shortening of both the Pt-P and Pt-Cl(trans to P) bonds.

However, the two rationalisations are not mutually exclusive. An explanation based on both σ- and π- effects seems most plausible.

A similar comparison of the Pt-P distances in II and III with these in analogous PMe_2Ph complexes is not possible. However, mean values of 2.30Å for Pt-P bonds involving phosphines with n-alkyl and phenyl substituents trans to C and P donor ligands[86] are in fair agreement with the Pt-P distances in II and III.

The Pt-C bond lengths in II and III

The $Pt-CF_3$ distance in II [2.058Å] is shorter than the sum of the appropriate van der Waals radii [ca. 2.09Å].[68] It is also significantly shorter than $Pt-CF_3$ distance [2.10(2)Å] reported for trans-[μ-$(CMe)_4(CF_3)Pt(PMe_2Ph)_2$]$SbF_3$.[73]

The two Pt-C bond lengths in II and III differ by 0.040(7)Å. Similar, though larger differences (ca. 0.1Å) have been found between fluorocarbyl- and hydrocarbyl-metal bond lengths for other transition metal ions.[88] As in other cases where it is possible to invoke π-backbonding (in this case between filled d-orbitals on the metal atom and σ^* orbitals of the trifluoromethyl group) two explanations have been put forward.

Thus, the relatively greater stability of fluoroalkyl- compared to the corresponding alkyl-metal complexes has been attributed, at least partly, to $Pt \rightarrow CF_3$ backbonding.[89]

The low C-F stretching frequencies in CF_3-transition metal complexes have also been explained on the basis of postulated π-acceptor properties of the fluoroalkyl group.[90]

Alternatively, it can be argued that differences in $Pt-CH_3$ and $Pt-CF_3$ σ-bonds explain the differences in length, with the $Pt-CF_3$ bond containing a greater proportion of carbon 2S component. This view is consistent with the linear variation of $^2J(Pt-CF_3)$ coupling constants for the complexes trans-$PtX(CF_3)(PMe_2Ph)$ with $^2J(Pt-CH_3)$ for the corresponding methyl-platinum complexes.[91] An M.O. calculation on $MnX(CO)_5$ ($X = CH_3, CF_3$) also suggests that the $M-CH_3$ and $M-CF_3$ bonds differ in their σ-component and that in either case back-donation is unimportant.[92]

Both theories predict the shortening of metal-carbon(fluoroalkyl) bonds compared with metal-carbon(alkyl) bonds in corresponding complexes.

The mean Pt-C-F and F-C-F angles [114.8(7) and 103.6(4)°] are consistent with the second view, as is the similarity in trans-influence on Pt-P bond lengths displayed by CF_3 and CH_3 groups. In this context it is worth noting that the trans-influence of σ-hydrocarbyl ligands on Pt-Cl bonds is insensitive to the hybridisation of the donor carbon atom.[93] Thus, the enhancement of the C(2S) component in the Pt-C bond in II, compared with III, seems to be the most plausible explanation for the differences in the corresponding Pt-C bond lengths in II and III.

CHAPTER 3

Conformations of the Triethylphosphine Ligand and *cis*- and *trans*-Influence of Ligands in *cis*-$PtCl_2(PEt_3)L$ Complexes

The Crystal and Molecular Structures of *cis*-Dichlorobis(triethylphosphine)platinum(II) and *cis*-Dichlorocarbonyl(triethylphosphine)platinum(II)

3.1 Introduction

While structural and spectroscopic studies of transition metal complexes have yielded substantial information concerning the trans-influence of ligands, investigations of cis-influence have been less rewarding.[94,95]

The main problem is due to the lack of appropriate data which clearly distinguish between the electronic effects of the cis-ligands and intramolecular steric effects. For example, it has been shown both structurally[96] and spectroscopically[97,98] that in cis-$PtCl_2(PR_3)L$ complexes (L = neutral ligand, PR_3 = PPh_3, PMe_3, PEt_3 or PEt_2Ph) the strongest Pt-Cl bonds and weakest Pt-P bonds occur when L = CO. Although an electronic cis-effect could explain this result, such interpretation could only be tentative since the complexes compared contain different phosphines.

Recently, in this laboratory and elsewhere, accurate structure analyses have been carried out on a number of cis-$PtCl_2(PEt_3)L$ complexes, including those with L = Cl^-,[60] C(OEt)NHPh,[54] $C(NPhCH_2)_2$,[99] CNPh,[97] PF_3,[85] and $P(OPh)_3$.[84] The structure analyses of the complexes with L = CO and PEt_3 are reported here. These studies allow the cis-influence of the ligands L on Pt-Cl and Pt-PEt_3 bonds to be examined in a closely related series of complexes. Additionally the trans-influence of L on Pt-Cl bonds can be compared.

An interesting structural feature of the series of cis-$PtCl_2(PEt_3)L$ complexes is the tendency of the triethylphosphine ligand to adopt one particular conformation. In an attempt to understand this result, molecular mechanics calculations on the triethylphosphine molecule have been carried out.

3.2 Measurements and Results for cis-$PtCl_2(PEt_3)_2$

Crystal Data

Formula	cis-$PtCl_2(PEt_3)_2$
Formula weight	502.3
Crystal system	monoclinic
a(Å)	7.941
b(Å)	17.442
c(Å)	12.960
β(°)	93.81
Cell volume (Å^3)	1791.0
No. of molecules per cell	4
Calculated density(g cm^{-3})	1.863
μ(Mo-Kα) (cm^{-1})	83.8
Space group	$P2_1/n(C_{2h}^5$, No.14)
Equivalent positions	±(x,y,z) and ±(1/2+x,1/2-y,1/2+z)

Data Collection Summary

Temperature (°C)	21
Diffractometer	Hilger and Watts Y-290
Radiation	graphite monochromatized Mo-K_{α}(λ=0.71069A)
Counter	scintillation with pulse-height analyser
Scan	$\theta/2\theta$
Background	stationary crystal- -stationary counter
θ_{max} (°)	30
Scan width $\Delta\theta$(°)	0.6
Step size in θ(°)	0.02
T_p (s)	60
T_b (s)	10
q(see Part I, Ch.3)	0.04
No. of reflections with $I>3\sigma(I)$, n	4004
No. of parameters, m	270
n/m	14.8

Measurements

The crystal of *cis*-$PtCl_2(PEt_3)_2$ was an air-stable transparent needle with nine faces belonging to the forms {100}, {010}, {001}, {011}, {101} and {101}. Its dimensions along the directions a*, b*, and c* were *ca*. 0.042x0.026x0.019 cm.

The preliminary cell dimensions were determined from oscillation and Weissenberg photographs. Systematically absent reflections were consistent with the space group $P2_1/n$. The crystal was then transferred to the diffractometer. The angle between the crystal a*-axis and the diffractometer ϕ-axis was a few degrees, in order to avoid multiple reflections. The unit cell dimensions were refined by a least-squares treatment of 11 reflections for which $\theta(Mo\text{-}K_\alpha) \geqslant 13$ and which were well dispersed through reciprocal space. The intensities of hk$\pm$l reflections were collected up to 30° in θ. In order to monitor the crystal and system stability, the intensities of three standard reflections were remeasured periodically throughout the experiment. They displayed only statistical fluctuations, with maximum deviations of ±5% from their corresponding mean values. The integrated intensities, I, and their standard deviations, were obtained as described in Part I, Ch.3. Data were corrected for Lorentz-polarisation factors, counting loss and absorption effects. The transmission factors on $|F|^2$, calculated by a Gaussian integration involving 1000 sampling points, varied between 0.20 and 0.35. Extinction corrections did not appear to be necessary.

Structure analysis

The platinum atom was located from a three-dimensional Patterson function. The positions of the remaining non-hydrogen atoms were determined from subsequent difference syntheses. Refinement of the positional and anisotropic thermal parameters of all non-hydrogen atoms converged at $R = 0.046$ and $R_w = 0.060$. Absorption correction was then carried out and subsequent refinement resulted in $R = 0.036$ and $R_w = 0.049$. From difference syntheses, based either on all the data or on low-angle data ($\sin\theta/\lambda \leqslant 0.4$), it was possible to locate all hydrogen atoms except H(30)C(12) (see Table 3.1). The positional and isotropic temperature parameters of the hydrogen atoms located from the difference syntheses were refined; the H(30)C(12) atom was also included in the structural model, but its calculated position and assigned temperature factor were not allowed to vary. The refinement converged at $R = 0.032$ and $R_w = 0.041$. The shifts in the parameters in the last cycle of refinement were smaller than 0.4σ. The standard deviation of an observation of unit weight was 1.59. The mean values of $(|F_o|-|F_c|)^2/\sigma^2(|F_o|)$ showed no systematic trends when analysed as a function of $|F_o|$ or $\sin\theta$. The extreme function values in the final difference synthesis (± 1.0 e$\AA^{-3}$) were associated with the position of the platinum atom.

Final atomic parameters and a selection of functions derived from them are presented in Tables 3.1 - 3.7. An ORTEP drawing of the molecular structure is presented in Figure 3.1.

TABLE 3.1 Final fractional atomic co-ordinates and isotropic thermal parameters (Åx10^2)

Atom	x	y	z	U(iso)
Pt	0.32951(2)	0.19372(1)	0.19303(1)	-
Cl(1)	-0.0182(2)	0.1334(1)	0.2266(2)	-
Cl(2)	0.0827(2)	0.3080(1)	0.1736(2)	-
P(1)	0.3484(2)	0.0760(1)	0.2255(1)	-
P(2)	0.4808(2)	0.2579(1)	0.1675(1)	-
C(1)	0.3148(8)	0.0454(3)	0.3576(5)	-
C(2)	0.4013(12)	0.0960(5)	0.4407(6)	-
C(3)	0.2406(7)	0.0003(3)	0.1495(5)	-
C(4)	0.2356(9)	0.0111(4)	0.0336(6)	-
C(5)	0.5716(6)	0.0590(3)	0.2095(5)	-
C(6)	0.6432(8)	-0.0194(4)	0.2415(6)	-
C(7)	0.4562(9)	0.3585(3)	0.1290(5)	-
C(8)	0.3946(11)	0.3743(4)	0.0192(7)	-
C(9)	0.6207(7)	0.2216(3)	0.0724(5)	-
C(10)	0.5332(11)	0.1907(4)	-0.0266(6)	-
C(11)	0.6164(7)	0.2667(3)	0.2871(5)	-
C(12)	0.5328(11)	0.3080(4)	0.3726(6)	-
H(1)C(1)$^{+}$	0.352(8)	-0.005(3)	0.372(5)	4(2)
H(2)C(1)	0.182(8)	0.040(3)	0.358(5)	4(2)
H(3)C(2)	0.377(7)	0.139(4)	0.445(5)	4(2)
H(4)C(2)	0.515(9)	0.098(4)	0.437(6)	6(2)
H(5)C(2)	0.361(11)	0.073(5)	0.512(7)	9(3)
H(6)C(3)	0.131(9)	-0.008(4)	0.183(6)	6(2)
H(7)C(3)	0.263(6)	-0.040(3)	0.177(4)	2(1)
H(8)C(4)	0.200(11)	0.068(5)	0.014(7)	8(3)
H(9)C(4)	0.161(10)	-0.034(5)	0.000(6)	7(2)
H(10)C(4)	0.341(11)	0.013(6)	0.018(7)	10(3)
H(11)C(5)	0.589(9)	0.072(4)	0.129(6)	6(2)
H(12)C(5)	0.634(7)	0.095(3)	0.244(5)	4(2)

$^{+}$H(n)C(m) is H(n) hydrogen atom attached to carbon atom C(m).

H(13)C(6)	0.594(9)	-0.058(4)	0.218(6)	6(2)
H(14)C(6)	0.768(8)	-0.025(4)	0.222(6)	6(2)
H(15)C(6)	0.623(10)	-0.028(5)	0.317(7)	8(3)
H(16)C(7)	0.400(8)	0.382(4)	0.185(5)	4(2)
H(17)C(7)	0.572(13)	0.397(6)	0.115(8)	12(4)
H(18)C(8)	0.510(11)	0.373(5)	-0.028(7)	8(3)
H(19)C(8)	0.295(9)	0.341(5)	0.003(6)	6(2)
H(20)C(8)	0.382(11)	0.437(5)	-0.007(7)	9(3)
H(21)C(9)	0.681(9)	0.183(4)	0.111(6)	5(2)
H(22)C(9)	0.701(9)	0.264(5)	0.061(6)	7(2)
H(23)C(10)	0.622(11)	0.180(4)	-0.091(7)	8(3)
H(24)C(10)	0.457(7)	0.235(3)	-0.072(5)	4(2)
H(25)C(10)	0.464(10)	0.148(5)	-0.009(6)	7(2)
H(26)C(11)	0.720(10)	0.296(4)	0.265(7)	6(2)
H(27)C(11)	0.651(8)	0.222(4)	0.305(5)	4(2)
H(28)C(12)	0.520(13)	0.377(6)	0.357(8)	12(4)
H(29)C(12)	0.433(8)	0.287(4)	0.382(5)	5(2)
H(30)C(12)	0.612[a]	0.304[a]	0.444[a]	8[a]

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[a] Not refined , see text.

TABLE 3.2 Anisotropic thermal parameters ($\mathring{A}^2$)of non-hydrogen atoms. The form of the temperature factor is $\exp(-2\pi^2\times10^{-n}\sum_{i=1}^{3}\sum_{j=1}^{3}h_ih_ja_i^*a_j^*U_{ij})$, where n=4 for Pt,P, and Cl atoms and 3 for carbon atoms.

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pt	261(1)	314(1)	294(1)	37(1)	-8(1)	-7(1)
Cl(1)	268(6)	608(9)	854(12)	-10(6)	52(7)	68(9)
Cl(2)	499(8)	471(8)	734(11)	201(6)	47(8)	77(7)
P(1)	297(5)	300(6)	331(6)	-1(5)	2(5)	11(5)
P(2)	354(6)	300(6)	363(7)	-1(5)	22(5)	-1(5)
C(1)	51(3)	40(3)	40(3)	-2(2)	3(2)	6(2)
C(2)	109(7)	72(5)	39(4)	-11(4)	-8(4)	-6(3)
C(3)	45(3)	38(3)	50(3)	-7(2)	-4(2)	-8(2)
C(4)	67(4)	63(4)	49(4)	-10(3)	-5(3)	-16(3)
C(5)	31(2)	36(3)	50(3)	0(2)	4(2)	6(2)
C(6)	50(3)	47(3)	85(5)	14(3)	11(3)	17(3)
C(7)	71(4)	34(3)	58(4)	3(3)	14(3)	6(3)
C(8)	87(6)	55(4)	68(5)	15(4)	3(4)	16(4)
C(9)	37(3)	42(3)	50(3)	0(2)	9(2)	4(2)
C(10)	76(5)	62(4)	45(4)	-0(3)	16(3)	-9(3)
C(11)	37(3)	48(3)	53(3)	-3(2)	-10(2)	-9(3)
C(12)	74(5)	69(5)	48(4)	5(3)	-10(3)	-19(3)

TABLE 3.3 Selected bond lengths (Å)

PT	-	CL(1)	2.366(1)	P(2)	-	C(9)	1.826(6)
PT	-	CL(2)	2.355(2)	P(2)	-	C(11)	1.834(6)
PT	-	P(1)	2.257(1)	C(1)	-	C(2)	1.521(10)
PT	-	P(2)	2.262(1)	C(3)	-	C(4)	1.511(10)
P(1)	-	C(1)	1.829(6)	C(5)	-	C(6)	1.529(8)
P(1)	-	C(3)	1.825(6)	C(7)	-	C(8)	1.499(11)
P(1)	-	C(5)	1.822(5)	C(9)	-	C(10)	1.517(10)
P(2)	-	C(7)	1.831(6)	C(11)	-	C(12)	1.512(10)

TABLE 3.4 Selected interbond angles (^{o})

CL(2) - PT - CL(1)	86.45(6)	P(1) - PT - CL(1)	83.50(5)
P(2) - PT - CL(1)	176.22(6)	P(1) - PT - CL(2)	169.56(5)
P(2) - PT - CL(2)	90.72(5)	P(2) - PT - P(1)	99.19(5)
C(1) - P(1) - PT	111.22(19)	C(3) - P(1) - PT	113.39(19)
C(5) - P(1) - PT	119.33(18)	C(7) - P(2) - PT	115.98(23)
C(9) - P(2) - PT	119.12(19)	C(11) - P(2) - PT	111.92(20)
C(3) - P(1) - C(1)	101.64(28)	C(5) - P(1) - C(1)	105.24(27)
C(2) - C(1) - P(1)	113.96(46)	C(5) - P(1) - C(3)	104.24(26)
C(4) - C(3) - P(1)	115.28(44)	C(6) - C(5) - P(1)	117.47(40)
C(9) - P(2) - C(7)	101.91(30)	C(11) - P(2) - C(7)	101.32(30)
C(8) - C(7) - P(2)	117.20(48)	C(11) - P(2) - C(9)	104.44(27)
C(10) - C(9) - P(2)	115.38(45)	C(12) - C(11) - P(2)	113.39(46)

TABLE 3.5 Selected torsion angles (°)

CL(1) - PT - P(1) - C(1)	-61.5(2)	CL(1) - PT - P(1) - C(3)	52.3(2)
CL(1) - PT - P(1) - C(5)	175.7(2)	CL(2) - PT - P(1) - C(1)	-45.0(4)
CL(2) - PT - P(1) - C(3)	68.2(4)	CL(2) - PT - P(1) - C(5)	-168.4(3)
P(2) - PT - P(1) - C(1)	115.8(2)	P(2) - PT - P(1) - C(3)	-130.3(2)
P(2) - PT - P(1) - C(5)	-7.0(2)	CL(1) - PT - P(2) - C(7)	-48.0(7)
CL(1) - PT - P(2) - C(9)	-171.0(8)	CL(1) - PT - P(2) - C(11)	66.9(7)
CL(2) - PT - P(2) - C(7)	-7.3(2)	CL(2) - PT - P(2) - C(9)	-129.5(2)
CL(2) - PT - P(2) - C(11)	108.3(2)	P(1) - PT - P(2) - C(7)	176.1(2)
P(1) - PT - P(2) - C(9)	53.8(2)	P(1) - PT - P(2) - C(11)	-68.4(2)
PT - P(1) - C(1) - C(2)	-62.5(5)	C(3) - P(1) - C(1) - C(2)	176.6(5)
C(5) - P(1) - C(1) - C(2)	68.1(5)	PT - P(1) - C(3) - C(4)	57.0(5)
C(1) - P(1) - C(3) - C(4)	176.4(5)	C(5) - P(1) - C(3) - C(4)	-74.3(5)
PT - P(1) - C(5) - C(6)	174.7(4)	C(1) - P(1) - C(5) - C(6)	49.0(5)
C(3) - P(1) - C(5) - C(6)	-57.6(5)	PT - P(2) - C(7) - C(8)	-75.7(5)
C(9) - P(2) - C(7) - C(8)	55.3(6)	C(11) - P(2) - C(7) - C(8)	162.8(5)
PT - P(2) - C(9) - C(10)	39.9(5)	C(7) - P(2) - C(9) - C(10)	-89.2(5)
C(11) - P(2) - C(9) - C(10)	165.7(5)	PT - P(2) - C(11) - C(12)	-59.1(5)
C(7) - P(2) - C(11) - C(12)	65.1(5)	C(9) - P(2) - C(11) - C(12)	178.7(5)

TABLE 3.6 Intramolecular non-bonding distances (Å) less than the sum of the appropriate van der Waals radii.

Cl(1) ... C(3)	3.30	Cl(2) .. C(7)	3.18
Cl(1) ... C(1)	3.41	Cl(2) ... H(19)C(8)	2.92

TABLE 3.7 The equation of, and atomic deviations (Å x 10^3) from, the weighted least-squares plane defined by the atoms Pt, Cl(1), Cl(2), P(1) and P(2). The equation refers to orthogonal axes defined by a^*, b and c.

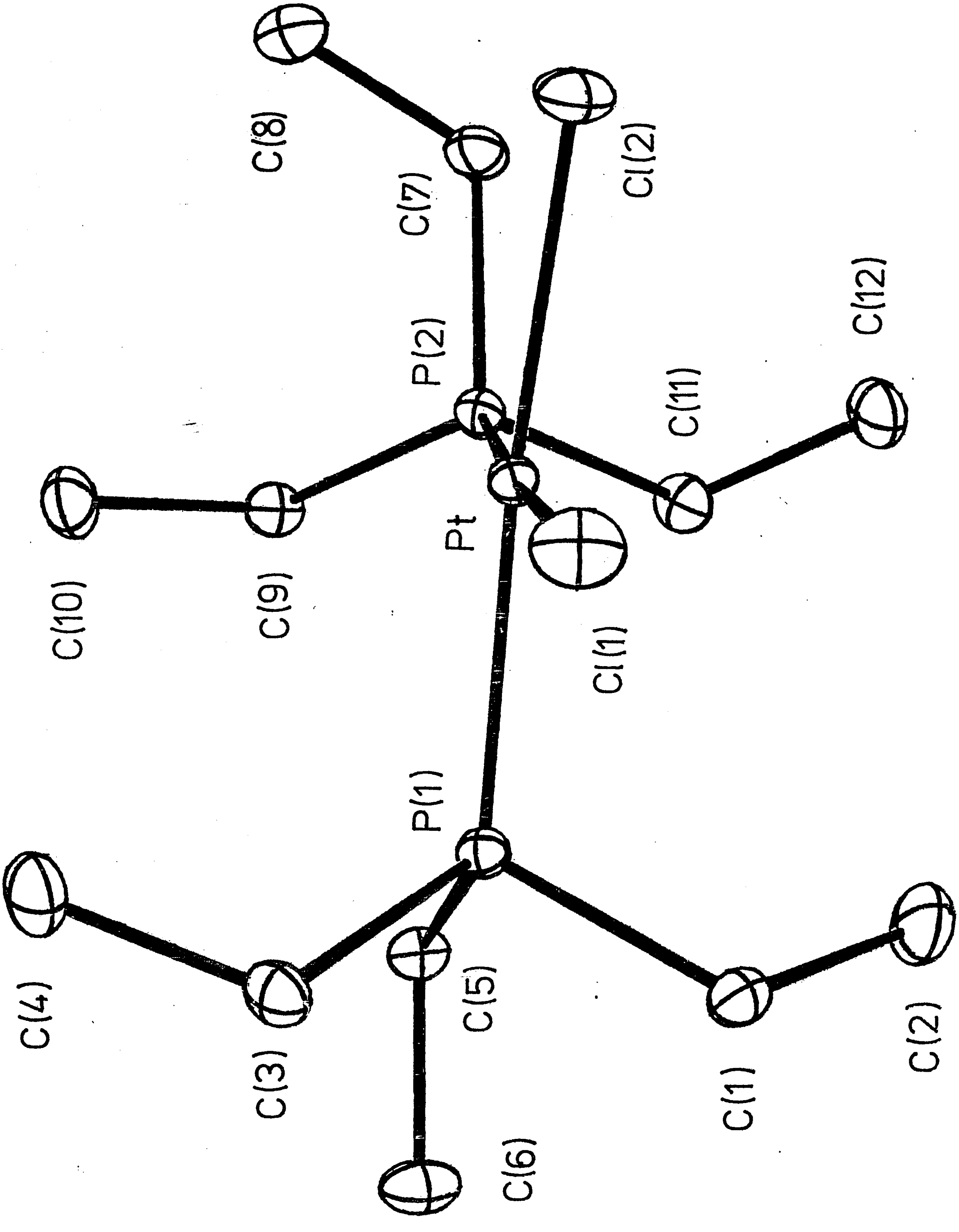
$$-0.071X-0.206Y-0.976Z=-3.260$$

Pt 3(1), Cl(1) -55(2), Cl(2) -76(2), P(1) -43(1), P(2) -44(1)

Legend

FIGURE 3.1 A perspective view of the molecular structure of cis-$PtCl_2(PEt_3)_2$. Hydrogen atoms are omitted for clarity. The vibrational ellipsoids represent 50% probability.

FIGURE 3.1



3.3 Measurements and Results for cis-$PtCl_2(CO)PEt_3$

A full account of the structure analysis of cis-$PtCl_2(CO)PEt_3$, together with atomic parameters and a selection of functions derived from them has been published (see inside back cover). Hence, only a brief summary of the experimental work and results will be given here.

The intensities of all independent reflections with $\theta(Mo\text{-}K_\alpha) \leqslant 35^\circ$ were measured. 1820 of the, for which $I > 3\sigma(I)$, were used in the structure analysis. Data were corrected for Lorentz-polarisation and absorption effects. The systematic absences were consistent with the space groups $Pca2_1$(No.29) and Pcam, the latter being an unconventional setting of the space group Pbcm(No.57). The structure was satisfactorily refined, by the full-matrix least-squares method, in the non-centrosymmetric space group $Pca2_1$. Solution of the structure was complicated by *pseudo*-symmetry, which gave rise to four possible arrangements for the atoms co-ordinated to platinum. The problem was resolved by refining each of these arrangements; that which produced the most acceptable stereochemistry and also gave the lowest value of R was used in further calculations. The final anisotropic structural model led to a value of R = 0.037. The correctness of the indexing of reflections was then verified by establishing that refinement of the structure, with hkl reflections reindexed as $hk\bar{l}$, produced a significantly greater value of R (0.039).

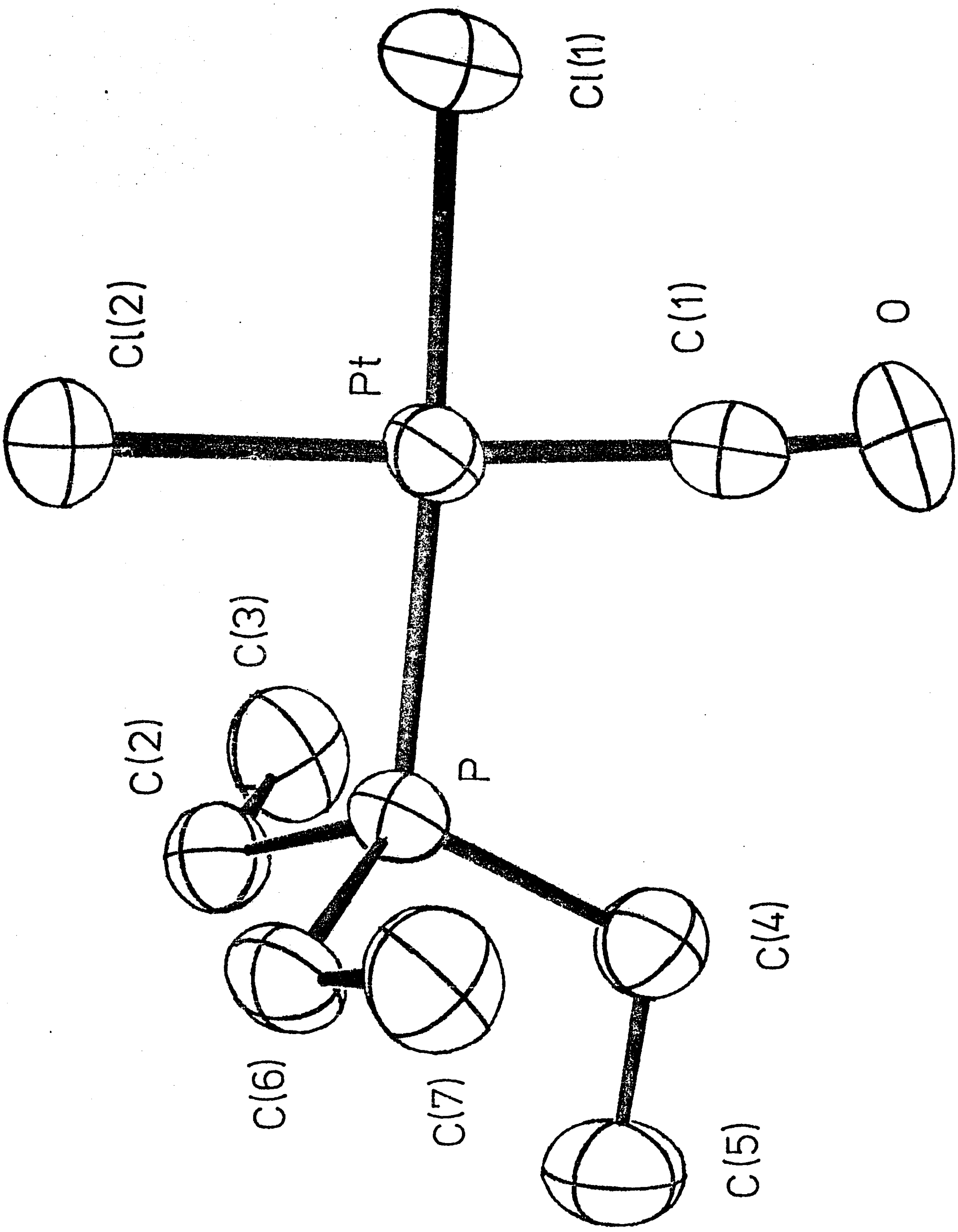
The adequacy of the weighting scheme was verified by establishing that the mean values of $(|Fo|-|Fc|)^2/\sigma^2(|Fo|)$ did not show systematic variations with either $|Fo|$ or $\sin\theta$. The extreme values in the final difference synthesis (+1.5 and -1.6 $e\text{Å}^{-3}$) were associated with the position of the platinum atom.

An ORTEP drawing of the molecular structure is presented in Figure 3.2 and a description of the structure is given in Section 3.5.

Legend

FIGURE 3.2 A perspective view of the molecular structure of cis-$PtCl_2(CO)PEt_3$. Hydrogen atoms are omitted. The vibrational ellipsoids display 50% probability.

FIGURE 3.2



3.4 Discussion

The crystal and molecular structure of cis-$PtCl_2(PEt_3)_2$

The crystals contain discrete, monomeric cis-$PtCl_2(PEt_3)_2$ units. The shortest distances between the atoms in different molecules are close to the sum of the corresponding van der Waals radii.

The platinum co-ordination is square-planar with a slight pyramidal distortion. However, the individual displacements of the platinum, phosphorus and chlorine atoms from the weighted $PtCl_2P_2$ least-squares plane do not exceed 0.08Å (Table 3.7). Intramolecular steric strain is relieved, at least partly, by distortions of the valency angles, subtended at platinum by cis-donor atoms, by up to 9° from the ideal value of 90° (Table 3.4). The two phosphine ligands interact somewhat differently with the adjacent cis-chlorine atoms [C(1)...Cl(1) 3.41, C(3)...Cl(1) 3.30, C(7)...Cl(2) 3.18Å]. This may explain the difference in length [0.011(2)Å] between the chemically equivalent Pt-Cl bonds [Pt-Cl(1) 2.366(1) and Pt-Cl(2) 2.355(2)Å]. The Pt-P bond lengths [2.257(1) and 2.262(1)Å] are more nearly equal. The mean Pt-P and Pt-Cl distances agree well with corresponding values in cis-$PtCl_2(PMe_3)_2$[83] (see below).

The chemically equivalent bond lengths and valency angles within the two triethylphosphine ligands agree to within the experimental error and their average values are in good agreement with those found in other cis-$PtCl_2(PEt_3)L$

complexes (Table 3.8). The opening of the Pt-P-C angles, which are typically 3-6° greater than the tetrahedral angle, and a comparable closure of the C-P-C angles, are general features of transition metal-phosphine complexes. They have given rise to the suggestion that the phosphorus 3S orbital is concentrated in the M-P bond. Recent e.p.r. studies of metallophosphine complexes lend support to this view.[100] The experimentally determined positions of the phosphine hydrogens are in accord with the stereochemistry of the tetrahedrally-hybridised carbon atoms to which they are attached. The C-H bond lengths range from 0.8(1) to 1.2(1)Å, with a mean value of 1.0(1)Å.

In both phosphines the conformations about the P-C and C-C bonds are approximately staggered and each ligand is oriented so that one α-carbon atom [C(5) and C(7)] lies close to the platinum co-ordination plane. However, a significant conformational difference is revealed by the Pt-P(1)-C(5)-C(6) and Pt-P(2)-C(7)-C(8) torsion angles of 174.7(4) and -75.7(6)°. The conformation of the phosphine containing P(1) atom is that usually found in platinum (II) complexes (see below), with the carbon atoms of one ethyl group lying approximately in the metal co-ordination plane, normal to which is a coplanar C-C-P-C-C unit formed by the carbon atoms of the two remaining ethyl groups. The conformation displayed by the phosphine containing P(2) atom differs by a 120° twist about the P-C bond lying in the co-ordination plane.

<u>TABLE 3.8</u> The mean bond lengths (Å) and angles (°) for the triethylphosphine ligand in <u>cis</u>-$PtCl_2(PEt_3)L$ complexes

L	P-C	C-C	Pt-P-C	P-C-C	C-P-C	Ref.
Cl^-	1.842	1.543	114.6	112.9	103.9	60
CO	1.806	1.527	112.0	112.7	106.9	This work
PF_3	1.823	1.518	113.1	114.1	105.5	85
PEt_3^a	1.828	1.515	115.2	115.5	103.1	This work
$P(OPh)_3$	1.827	1.511	113.7	114.7	104.8	84
$C(NPhCH_2)_2$	1.810	1.540	113.8	114.6	105.1	99

[a] The values presented are mean values for two phosphine ligands

The crystal and molecular structure of cis-$PtCl_2(CO)PEt_3$

The crystal contains discrete monomeric molecules. Intermolecular distances are close to the sum of the corresponding van der Waals radii.

The molecules exhibit the expected cis-square-planar co-ordination around the platinum atom and nearly ideal C_s symmetry. Thus, the individual displacements of the Pt, P, Cl(1) and Cl(2) atoms from their weighted least-squares plane do not exceed 0.002Å; the displacements of the C(1), O, C(4) and C(5) atoms from the same plane are less than 0.06Å and the differences in displacement of the two pairs of atoms approximately related by mirror symmetry do not exceed 0.04Å [C(2) and C(6), C(3) and C(7)].

Bond lengths and angles in the triethylphosphine ligand are normal (Table 3.8). Conformation of this ligand is the common one, also displayed by the phosphine containing P(1) atom in cis-$PtCl_2(PEt_3)_2$.

The molecule is subject to some steric strain. This is evident from intramolecular non-bonding distances [C(1)...C(4) 3.15, Cl(2)...C(2) 3.48 and Cl(2)...C(6) 3.49Å] and the platinum valency angles [P-Pt-C(1) 94.7(4) and P-Pt-Cl(2) 87.9(1)°].

The Pt-Cl(1) and Pt-Cl(2) distances [respectively 2.368(3) and 2.296(4)Å] show that the triethylphosphine ligand exerts a substantially larger trans-influence than the carbonyl group. The Pt-P distance [2.265(3)Å] reflects the cis-influence of the carbonyl group (see below).

The Pt-C distance [1.855(14)Å] is the same as that in the analogous compound cis-$PtCl_2(CO)PPh_3$ [1.858(7)Å]. Little can be concluded from a comparison of these two distances with the corresponding ones, mainly of low accuracy, found in other platinum(II) carbonyl complexes (Table 3.9).

TABLE 3.9 Bond lengths (Å) and angles (°) in some square-planar platinum(II) carbonyl complexes

Compound	Pt-C	C-O	Pt-C-O	Ref.
trans-$PtCl_2(CO)$-$(ON-C_6H_4-OMe)$	1.74(4)	1.16(4)	178(3)	101
$[$trans-$PtCl(CO)(PEt_3)_2]^+$	1.78	1.14	171	102
cis-$PtCl_2(CO)PEt_3$	1.855(14)	1.124(19)	176.5(12)	This work
cis-$PtCl_2(CO)PPh_3$	1.858(7)	1.114(8)	175.6(7)	96
$[$trans-$Pt(PEt_3)_2(CO)$-$C_6H_4Cl]^+$	1.97(5)	1.06(6)	171(5)	103

Triethylphosphine: molecular mechanics calculation for the free molecule and conformations in Pt^{II} - PEt_3 complexes

The tendency of triethylphosphine ligands to exhibit exclusively one particular conformation in square-planar complexes has already been mentioned. In order to understand this result, the relative energies of the minimum-energy conformations of the isolated triethylphosphine molecule have been calculated using the molecular mechanics method. Apart from a study of triphenylphosphine molecule,[104] in which the ring geometry was held invariant, this approach does not appear to have been used before to study phosphine conformations. The calculations will first be described and the results will then be compared with the available structural data on platinum (II)-triethylphosphine complexes.

The computational procedure used has been successfully applied to many organic systems.[105] The potential energy of the molecule is given by:

$$E = V(r) + V(\theta) + V(w) + V(nb).$$

The potential energy functions used to describe the deformations of bond lengths, Δr, and valency angles, $\Delta\theta$, (from hypothetical strain-free values r_0 and θ_0) are

$$V(r)=1/2k_r\Delta r^2$$

$$V(\theta)=1/2k_\theta(\Delta\theta-|k'_\theta\Delta\theta|)^2$$

where k_r and k_θ are harmonic stretching and bending force constants and k_θ' allows for anharmonicity. For torsional rotation about P-C and C-C bonds a three-fold potential energy of the form

$$V(w)=1/2k_w(1+\cos 3w)$$

was used; w is a torsion angle and k_w the contribution made by that torsion angle to the total height of the energy barrier. Non-bonding interactions were represented by a Lennard-Jones potential of the form:

$$V(nb)=\varepsilon\left[-2\alpha^{-6}+e^{12(1-\alpha)}\right],\ \alpha=r_{12}/r_{12}^*$$

where r_{12} is the internuclear distance and r_{12}^* depends only on the chemical nature of the atoms involved in the contact. The force-field parameters used are summarised in Table 3.10. They were taken from standard sources, supplemented by force constant data for trialkylphosphines obtained spectroscopically. The parameters r_0 and θ_0 for bonds and angles involving phosphorus atom are mean values for platinum (II) - triethylphosphine complexes. It should be noted that no allowance was made for coulombic interactions.

The calculations were performed on a PDP-11 computer using Dr. D.N.J. White's program PECALC. To minimise computing time the assumption was made that the lowest energy conformations of triethylphosphine will be those involving staggering about the P-C and C-C bonds. Seven such conformations, A-G, are possible (Table 3.11).

TABLE 3.10 Potential field parameters used for PEt_3

Bonds	k_r (kcalmol^{-1}Å^{-2})[a]	r_o (Å)
P-C	407.52	1.827
C-C	633.6	1.523
C-H	662.4	1.10

Angles	k_θ (kcalmol^{-1}deg^{-2})[b]	$k_{\theta'}$ (deg^{-1})[b]	θ (°)
C-P-C	0.0116	-	105.2
P-C-C	0.0136	-	114.3
P-C-H	0.0100	-	97.5
C-C-H	0.0160	0.0096	109.5
H-C-H	0.0142	0.0096	108.2

Torsion angles	k_w (kcalmol^{-1})
H-C-C-P	3.96[c]
C-C-P-C	3.84[d]
H-C-P-C	3.84[d]
H-C-C-H	3.96[c]

Non-bonding potentials

Type	ε (kcalmol^{-1})[e]	r_{12} (Å)[e]
P...C	0.076	4.025
P...H	0.033	3.65
C...C	0.120	3.85
C...H	0.0299	3.35
H...H	0.0160	3.10

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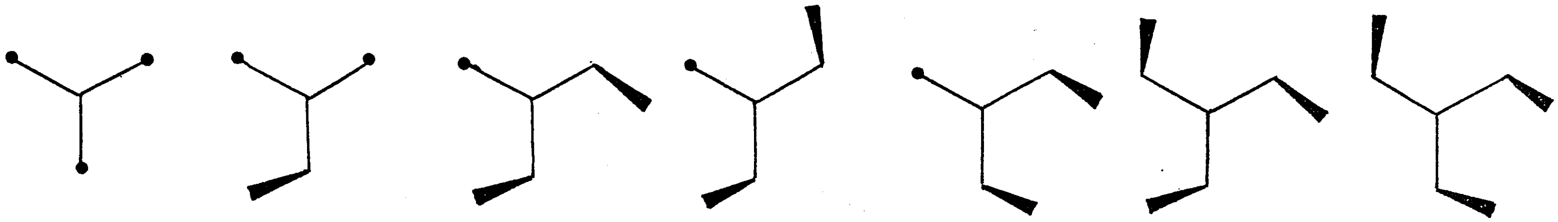
[a] Ref. 106; [b] Ref. 104; [c] Ref. 107; [d] Refs. 108 and 109; [e] Parameters supplied by Dr. D.N.J.White.

TABLE 3.11 The seven staggered conformations of triethylphosphine molecule. The conformations are represented schematically viewing down the lone-pair to phosphorus vector.



Conformation	A	B	C	D	E	F	G
E(kcalmol^{-1})	2.52	1.62	0.94	0.87	1.39	1.14	1.56
$\langle \partial E/\partial p \rangle$	0.15	0.16	0.11	0.11	0.09	0.11	0.12
$\theta(^{\circ})^{a}$	116	132	149	149	149	160	160

---

[a] θ is the Tolman's cone angle

For each possible conformation the geometry was adjusted by an iterative procedure so as to minimise the potential energy, E. It took about 100-150 cycles of refinement to bring the energy for a conformation close to a minimum, as measured by the quantity $\langle \partial E/\partial p \rangle$.

The results of such calculations indicate D as the minimum energy conformation of the isolated molecule, followed closely by C and F. Although the differences in energy between the conformations are not dramatic, it is remarkable that of 28 triethylphosphine ligands attached to platinum (II), for which structure analyses are available, 24 exhibit conformation D. Moreover, in every case the observed conformation differs only slightly, if at all, from the idealised fully-staggered D conformation. This is shown in Table 3.12A where the deviations of the Pt-P-C-C torsion angles w_1,w_2 and w_3 from the idealised values of 180, 60 and -60° are presented. The average deviation $\langle |\Delta| \rangle$ is 9°, or less, for the 24 ligands. The tendency for one L-Pt-P-C torsion angle to be close to zero is also obvious from the Table 3.12.A.

Of the four triethylphosphine ligands which do not display D conformation (Table 3.12B) two have the relatively low energy C arrangement. The complex cis-$PtCl_2(PEt_3)_2$ is especially interesting, since it displays both a high energy G and low energy D conformation. It is also apparent from Table 3.12B that the mean deviations of the Pt-P-C-C angles from

TABLE 3.12.A Discrepancy (°) between idealised and observed conformation D of PEt_3 ligands in square-planar Pt(II) complexes

Compound[a]	180-w_1	60-w_2	-60-w_3	$\langle\lvert\Delta\rvert\rangle$[b]	L-Pt-P-C[c]	Ref.
cis-$PtCl_2P(CO)$	-4	-2	1	2	0	This work
cis-$PtCl_2P(PF_3)$	-2	-7	-3	4	1	85
cis-$PtCl_2P[P(OPh)_3]$	9	-10	-6	8	-20	84
cis-$PtCl_2P_2$	5	3	2	3	7	This work
cis-$PtCl_2P[C(OEt)NHPh]$	2	-16	3	7	-14	54
cis-$PtCl_2P[C(NPhCH_2)_2]$	0	-4	4	3	0	99
trans-$PtCl_2P[C(NPhCH_2)_2]$	-8	-3	0	4	6	99
trans-$[Pt(NCS)P_2]_2$-p-	1	-10	9	7	6	
-C_6H_4-$(C_2)_2$	2	-3	6	4	7	110
cis-$PtPh(GePh_2OH)P_2$	0	2	-2	1	0	
	0	1	-1	1	0	111
trans-$PtBr_2P_2$	11	7	-3	7	0	112
trans-$Pt(C_3H_5)BrP_2$	0	-6	0	2	2	
	-4	0	1	5	-2	113
trans-$[PtP_2Cl(NH_2\text{-}NH\text{-}C_6H_4F)]^+$	-3	3	0	2	13	114
trans-$[PtClP_2(NH{=}NC_6H_4F)]^+$	1	5	0	2	1	
	-2	2	-2	2	-9	115

---

[a] P=triethylphosphine. [b] The Pt-P-C-C torsion angles w_1, w_2, w_3, have idealised values of 180,60,and -60,respectively.
[c] L-ligand *cis* to PEt_3

Table 3.12.A (contd.)

trans-$PtIP_2[C(Me)=N-C_6H_4Cl]$	4	5	-4	4	0	
	-2	6	-5	4	-10	116
trans-$[Pt(CO)P_2(C_6H_4Cl)]^+$	6	12	-9	9	-1	103
cis-$PtClP_2(1,10\text{-phenanthroline})]^+$	2	5	0	2	7	
	-6	14	-4	8	10	117
trans-$PtClP_2(N=N-C_6H_4F)$	-1	-2	3	2	0	118
$[PtCl_3P]^-$	0	-3	6	3	-29	60

B. Square-planar Pt(II) complexes exhibiting other than D conformation of PEt_3 ligand

Compound	Conformation[d]	$w_1(^\circ)$	$w_2(^\circ)$	$w_3(^\circ)$	$\langle\lvert\Delta\rvert\rangle(^\circ)$	Ref.
cis-$PtCl_2P[P(NMe_2)_3]$	C	173	-42	-58	9	119
trans-$PtHBrP_2$	C	-142	-85	-69	24	
	F	-66	-70	-88	11	120
cis-$PtCl_2P_2$	G	-76	40	-59	12	This work

---

[d] See Table 3.11

idealised values, $\langle|\Delta|\rangle$, are appreciably greater than corresponding values listed in Table 3.12A.

The calculations thus indicate that the conformation usually adopted by triethylphosphine ligand in a platinum(II) complex is that which gives the lowest energy for the isolated molecule. The incorporation of the ligand into a square-planar complex evidently does not normally involve a significant increase in conformational energy. Indeed, the marked tendency of the ligand to adopt conformation D rather than C or F might indicate that complex formation tends to increase the energy difference between D and other conformations.

The calculations are also relevant to Tolman's use of the minimum cone angle θ as a measure of the steric bulk of different phosphines.[66] His value of 132° for triethylphosphine would appear to be derived from conformation B (see Table 3.11). This is neither the conformation which minimises the cone angle, nor is it found in platinum (II) complexes. The experimentally determined molecular structures and the Tolman averaging formula $\theta=2/3\sum\theta_i/2$ [66] give cone angles of 145-150° for D conformations, and 157° for the G conformation found in cis-$PtCl_2(PEt_3)_2$. The cone angle concept has undoubtedly been helpful in rationalising properties of the complexes in solution, such as their n.m.r. parameters and heats of ligand replacement, but the above considerations suggest that it may not be useful in rationalising solid state structures.

cis- and trans-Influence of ligands in cis-$PtCl_2(PEt_3)L$ complexes

The structural work described earlier in this Chapter forms part of a more general study of cis-$PtCl_2(PEt_3)L$ complexes. Bond length data for eight such complexes are presented in Table 3.13, together with corresponding $^1J(Pt-PEt_3)$ coupling constants (see also Figure 3.3).

The trans-influence series derived from the Pt-Cl(trans to L) distances in Table 3.13 bears little relationship to the cis-influence series obtained from the Pt-PEt_3 bond lengths. This would appear to indicate that different electronic mechanisms are involved. The cis-influence series seems to reflect mainly the π-acidity of L: the shortest Pt-PEt_3 bond occurs cis to chloride, which has little π-acidity, and the longest Pt-PEt_3 bonds are those cis to phosphite, carbonyl and trifluorophosphine, which are usually considered to be π-acids. The lengthening of the Pt-PEt_3 bonds can then be ascribed to competition between PEt_3 and L for metal d_π electrons. The smaller variations in Pt-Cl(cis to L) distances may then arise from variations in trans-influence of PEt_3 induced by L. Alternatively, Pt→L backdonation may directly strengthen the Pt-Cl(cis to L) bonding through an electrostatic effect.

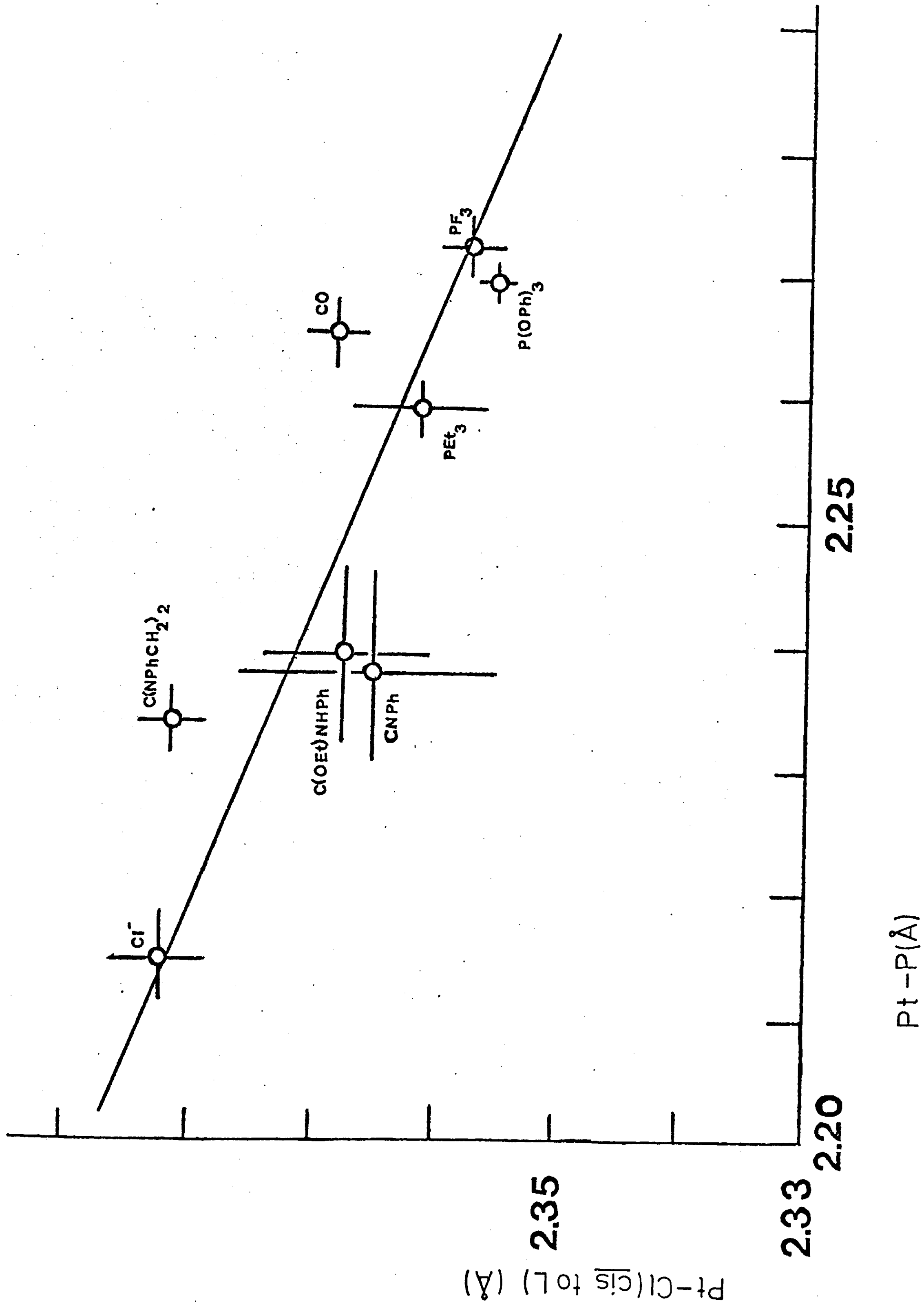
Finally, it should be noted that the Pt-PEt_3 bond

TABLE 3.13 Bond lengths (Å) and coupling constants (Hz) in *cis*-$PtCl_2(PEt_3)L$ complexes

L	Pt-PEt_3	Pt-Cl (*cis* to L)	Pt-Cl (*trans* to L)	$^1J(Pt-PEt_3)$	Ref.[a]
Cl^-	2.215(4)	2.382(4)	2.301(3)[b]	3704[c]	60
$C(NPhCH_2)_2$	2.234(3)	2.381(3)	2.362(3)	3720[d]	99
CNPh	2.238(8)	2.365(11)	2.333(12)	3049[e]	97
C(OEt)NHPh	2.239(8)	2.367(7)	2.361(5)	----	54
PEt_3	2.259(2)[b]	2.361(6)[b]	2.361(6)[b]	3515[f]	This work
CO	2.265(3)	2.368(3)	2.296(4)	2754[e]	This work
$P(OPh)_3$	2.269(1)	2.355(2)	2.344(2)	3210[f]	84
PF_3	2.272(3)	2.357(3)	2.305(3)	2760[g]	85

[a] Structural data; [b] Mean value; [c] Ref. 60; [d] Ref. 121; [e] Ref. 97; [f] Ref. 122; [g] Ref. 85

FIGURE 3.3 A plot of Pt-Cl(cis to L) versus Pt-P bond lengths in cis-$PtCl_2(PEt_3)L$ complexes (see Table 3.13). The ligands L and the unweighted least-squares trend line are shown. The errors indicated are standard deviations.



lengths, reflecting the overall bond order, show only a very poor correlation with $^{1}J(Pt-PEt_3)$ coupling constants, which depend mainly on the s-bond order. This result appears to contradict Pidcock's proposal that Pt-P bond lengths and coupling constants are inter-related.[45]

CHAPTER 4

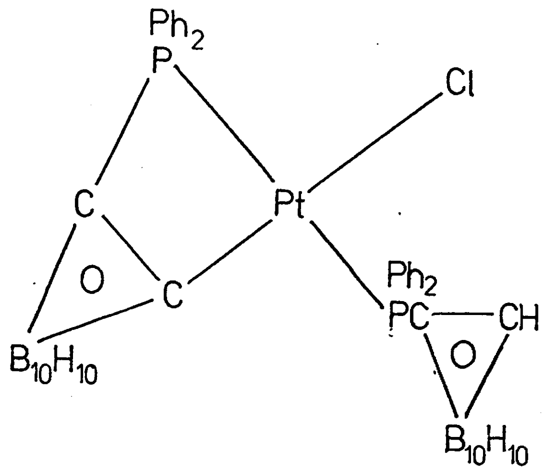
The Crystal and Molecular Structure of a Platinum(II) Complex Containing ortho-Carborane Phosphino Ligands

4.1 Introduction

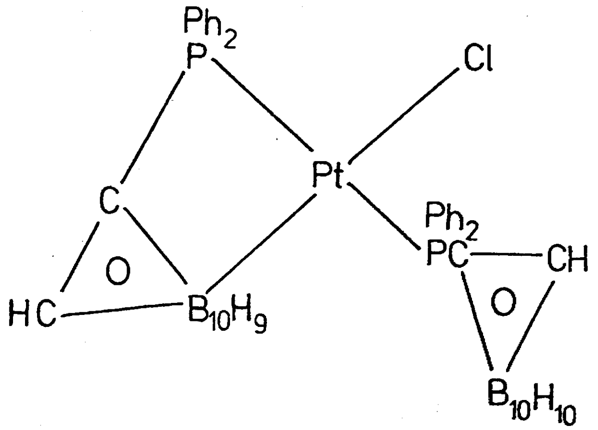
Hill and Silva-Trivino have prepared unsymmetrical bis-tertiaryphosphino-ortho-carborane derivatives of the type L = $Ph_2PCB_{10}H_{10}CPR_2$ (R = F,NMe_2,Ph) and reacted them with platinum(II) nitriles to obtain $PtCl_2L$ complexes in which L is thought to behave as a chelating ligand.[123]

The electron-withdrawing nature of the ortho-carborane cage is well established.[124] Accordingly, a structure analysis of the complex $PtCl_2L$(R=F) was undertaken, in order to study the effects of the electron-withdrawal by the fluoro- and carborane- substituents at phosphorus on the metal-ligand bonding. This appeared to be a natural extension both of the studies of $PMe_2C_6F_5$-platinum complexes (described in Part II, Ch. 2) and of a previous study in this department of the unsymmetrical chelate complex cis-$PtCl_2\{(CF_3)_2PCH_2CH_2PPh_2\}$.[69] In the mean time Miguel obtained an n.m.r. spectrum of $PtCl_2L$(R=F), which revealed that no fluorine was present.[125] The X-ray analysis was however continued in order to establish the identity of the compound.

It turned out that the compound contains two diphenylphosphino-ortho-carborane ligands. One of these is unidentate and co-ordinated to platinum through phosphorus, the other one is bidentate and bonded to platinum through phosphorus and through either a carbon or a boron atom, thus indicating an insertion of the metal atom into a C-H or B-H bond. Unfortunately, the X-ray analysis does not allow carbon and boron atom to be clearly distinguished. However, the structural and chemical evidence discussed below suggests that structure II is more likely than structure I.



(I)



(II)

4.2 Measurements and Results

Crystal Data

Formula	$PtCl(Ph_2PC_2B_{10}H_{10})(Ph_2PC_2B_{10}H_{11})$
Formula weight	886.33
Crystal system	monoclinic
a(Å)	26.122(3)
b(Å)	11.006(2)
c(Å)	29.775(3)
β(°)	106.34(1)
Cell volume ($Å^3$)	8214.4
No. of mol. per cell	8
Calculated density($g\ cm^{-3}$)	1.453
μ(Mo-K_α) (cm^{-1})	36.2
Space group	I2/a (C_{2h}^6, No.15)
Equivalent positions	(1/2 1/2 1/2); ± (xyz) and ± (1/2+x,$\bar{y}$,z)

Data Collection Summary

Temperature (^{o}C)	20
Diffractometer	Enraf-Nonius CAD-4F
Radiation	graphite monochromatised Mo-K_{α} (λ= 0.71069Å)
Counter	scintillation with pulse - height analyser
Scan	$\theta/2\theta$
Background measurement	moving crystal - moving counter
θ_{max} (^{o})	25
No. reflections with $I>4\sigma(I)$, (n)	4855
No. of parameters refined, (m)	233
n/m	20.8

Measurement and treatment of intensity data

A needle shaped crystal of dimensions 0.053 x 0.016 x 0.013cm, displaying the forms {101},{001} and {010}, was mounted in air. Preliminary oscillation and Weissenberg photographs revealed monoclinic symmetry and allowed approximate cell parameters to be determined. The crystal was then transferred to an Enraf-Nonius CAD-4F diffractometer, in an orientation such that the angle between the crystal b-axis and the diffractometer ϕ-axis was a few degrees. Final values of the unit cell parameters and the orientation matrix were determined from the setting angles of 25 reflections with $14 \leq \theta \leq 22^{\circ}$. The indexing of reflections corresponded to a body-centred unit cell; this gave a β-angle much closer to 90° than the alternative indexing based on a face-centred (C) cell. The systematic absences (hkl,h+k+l=2n+1; hol, h=2n+1) are consistent with the space groups Ia(C_s^4,No.9) and I2/a(C_{2h}^6,No.15). The latter space group led to a successful solution of the structure.

The intensities of all independent reflections in the range $2 \leq \theta \leq 25^{\circ}$ were measured, using the $\theta/2\theta$ scan method. The scan width (in degrees) of $0.60+0.35\tan\theta$ was increased by 25% at each extremity to allow for background measurement. The maximum counting time was 60s, but for stronger reflections it was adjusted so that $\sigma(I)/I$ was 0.03. In order to check the crystal and electronic stability the setting angles and intensities of two suitable reflections were remeasured periodically. No significant change either in intensity or orientation was observed during the experiment. Of the 7941 intensities measured, 4855, for which $I>4\sigma(I)$, were used in the subsequent analysis. The data were corrected for Lorentz-polarisation and absorption effects. The transmission factors (on $|F_o|^2$), based on Gaussian integration and a grid of 512 points, were in the range 0.56-0.72.

Structure determination and refinement

The position of the platinum atom was deduced from a three-dimensional Patterson synthesis. Structure factors containing contributions for the heavy-atom scattering only gave R=0.25 and a difference synthesis which revealed the positions of the phosphorus and chlorine atoms. Inclusion of the scattering contributions for these atoms in the structure factor calculations gave R=0.17, and the subsequent difference synthesis allowed the positions of all the remaining non-hydrogen atoms to be determined. This synthesis contained a peak, comparable in height with the peaks of phenyl and carborane carbon and boron atoms, at a distance of 0.68Å from a two-fold axis. This peak was thought to be associated with a solvent molecule. It was included in subsequent calculations and assigned a carbon scattering factor. The identity of the solvent was not established by the analysis.

The structure was then refined by the full-matrix least-squares method; the parameters of different groups of atoms were refined in successive cycles. Adjustment of the positional and isotropic thermal parameters of all non-hydrogen atoms gave $R=0.077$. Correction for absorption and the introduction of anisotropic temperature factors for the platinum, chlorine and phosphorus atoms gave $R=0.042$ and $R_w=0.062$. Before the final cycles of refinement, an attempt was made to distinguish between the carbon and boron atoms of the carborane cages. Inspection of the isotropic temperature factors of the cage atoms did not suggest that they could be used to deduce unambiguously the chemical nature of these atoms. However, a detailed examination of the interatomic distances within the carborane cages (see below) allowed atoms C(25) to C(28) to be tentatively identified as carbon atoms, and the refinement was completed on this assumption.

In the final cycle of least-squares refinement no parameter changed by more than 0.03 of its standard deviation. The final difference synthesis revealed two peaks of <u>ca</u>. 1.2eÅ^{-3} close to the platinum atom. All other peaks were lower than 0.8eÅ^{-3}. The adequacy of the weighting scheme was verified by establishing that mean values of $(|F_o|-|F_c|)^2/\sigma^2(|F_o|)$ showed little variation with either $|F_o|$ or $\sin\theta$. Extinction corrections did not appear to be necessary.

The final atomic parameters and a selection of functions derived from them are presented in Tables 4.1-4.5.

TABLE 4.1 Final positional parameters of the atoms (fractional co-ordinates) and isotropic thermal parameters ($\text{Å}\times10^2$) of carbon and boron atoms

Atom	x	y	z	U(iso)
Pt	0.11994(1)	0.30761(3)	0.09027(1)	----
Cl	0.1096(1)	0.1595(2)	0.0285(1)	----
P(1)	0.0871(1)	0.1729(2)	0.1333(1)	----
P(2)	0.1535(1)	0.4798(2)	0.0648(1)	----
C(1)	0.0186(4)	0.1296(9)	0.1153(3)	4.5(2)
C(2)	-0.0084(4)	0.134(1)	0.0677(4)	5.8(3)
C(3)	-0.0615(5)	0.094(1)	0.0518(4)	7.8(4)
C(4)	-0.0883(5)	0.057(1)	0.0839(5)	8.6(4)
C(5)	-0.0624(6)	0.060(1)	0.1328(5)	9.4(4)
C(6)	-0.0084(5)	0.099(1)	0.1471(5)	7.9(4)
C(7)	0.1276(3)	0.0413(8)	0.1568(3)	3.9(2)
C(8)	0.1747(4)	0.027(1)	0.1446(3)	5.2(2)
C(9)	0.2088(5)	-0.069(1)	0.1646(4)	6.7(3)
C(10)	0.1918(5)	-0.152(1)	0.1941(4)	6.7(3)
C(11)	0.1447(5)	-0.138(1)	0.2048(4)	7.1(3)
C(12)	0.1122(4)	-0.040(1)	0.1857(4)	5.7(3)
C(25)	0.0954(4)	0.2790(9)	0.1823(3)	4.5(2)
C(26)	0.1607(4)	0.306(1)	0.2040(4)	4.0(2)
B(1)	0.1235(4)	0.393(1)	0.1530(4)	5.9(3)
B(2)	0.1231(5)	0.263(1)	0.2406(4)	5.6(3)
B(3)	0.1702(6)	0.381(2)	0.2560(5)	7.4(4)
B(4)	0.1058(6)	0.397(2)	0.2655(6)	7.9(4)
B(5)	0.0614(5)	0.406(1)	0.1656(4)	5.2(3)
B(6)	0.0689(7)	0.491(1)	0.2177(6)	8.4(5)
B(7)	0.1714(6)	0.462(1)	0.2033(5)	6.6(4)
B(8)	0.1372(7)	0.523(2)	0.2415(6)	8.8(5)
B(9)	0.1078(6)	0.530(2)	0.1791(6)	7.8(4)
B(10)	0.0584(6)	0.327(1)	0.2168(6)	7.3(4)
C(13)	0.2199(3)	0.5086(8)	0.1043(3)	3.6(2)
C(14)	0.2505(4)	0.402(1)	0.1171(4)	5.4(3)
C(15)	0.3013(5)	0.409(1)	0.1499(4)	6.8(3)
C(16)	0.3205(4)	0.521(1)	0.1691(4)	6.0(3)
C(17)	0.2910(4)	0.625(1)	0.1553(4)	6.1(3)
C(18)	0.2396(4)	0.617(1)	0.1225(3)	5.0(2)
C(19)	0.1084(3)	0.6067(8)	0.0626(3)	3.6(2)
C(20)	0.0556(4)	0.577(1)	0.0584(4)	5.5(3)

Table 4.1 (contd.)

C(21)	0.0182(5)	0.667(1)	0.0562(5)	8.0(4)
C(22)	0.0332(5)	0.789(1)	0.0611(4)	7.2(3)
C(23)	0.0858(5)	0.822(1)	0.0646(4)	6.2(3)
C(24)	0.1238(4)	0.732(1)	0.0655(4)	5.2(3)
C(27)	0.1654(3)	0.4855(8)	0.0051(3)	3.4(2)
C(28)	0.1313(4)	0.3856(9)	-0.0336(3)	4.5(2)
B(11)	0.2268(5)	0.511(1)	-0.0047(5)	5.8(3)
B(12)	0.1742(5)	0.618(1)	-0.0246(4)	5.3(4)
B(13)	0.1420(6)	0.586(1)	-0.0845(5)	6.6(4)
B(14)	0.2121(6)	0.575(1)	-0.0616(5)	6.9(4)
B(15)	0.1127(5)	0.535(1)	-0.0410(4)	5.1(3)
B(16)	0.1136(5)	0.439(1)	-0.0888(4)	5.6(3)
B(17)	0.2276(5)	0.411(1)	-0.0517(5)	6.2(3)
B(18)	0.1754(5)	0.462(1)	-0.1022(4)	5.4(3)
B(19)	0.1980(5)	0.366(1)	-0.0096(4)	5.1(3)
B(20)	0.1674(5)	0.332(1)	-0.0687(5)	5.8(3)
C(29)	0.4757(9)	0.659(2)	0.2333(9)	18(1)

TABLE 4.2 Anisotropic thermal parameters ($\text{Å}^2 \times 10^3$) of the heavy atoms +

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pt	45.4(2)	24.2(2)	29.3(2)	-4.0(2)	15.2(1)	-0.8(2)
Cl	93(2)	34(1)	45(1)	-15(1)	32(1)	-12(1)
P(1)	46(1)	31(1)	34(1)	-5(1)	15(1)	2(1)
P(2)	37(1)	26(1)	29(1)	0(1)	10(1)	1(1)

+ The form of temperature factor is $\exp(-2\pi^2 \sum_{i=1}^{3} \sum_{j=1}^{3} h_i h_j a_i^* a_j^* U_{ij})$

TABLE 4.3 Bond lengths (Å)

PT	-	CL	2.415(3)	C(7)	-	C(12)	1.378(15)
PT	-	P(1)	2.279(3)	C(8)	-	C(9)	1.402(15)
PT	-	P(2)	2.305(2)	C(9)	-	C(10)	1.421(18)
PT	-	B(1)	2.073(9)	C(10)	-	C(11)	1.366(20)
P(1)	-	C(1)	1.783(10)	C(11)	-	C(12)	1.393(16)
P(1)	-	C(7)	1.814(8)	C(13)	-	C(14)	1.414(13)
P(1)	-	C(25)	1.834(10)	C(13)	-	C(18)	1.350(13)
P(2)	-	C(13)	1.829(7)	C(14)	-	C(15)	1.412(14)
P(2)	-	C(19)	1.816(9)	C(15)	-	C(16)	1.394(17)
P(2)	-	C(27)	1.888(10)	C(16)	-	C(17)	1.376(16)
C(1)	-	C(2)	1.392(13)	C(17)	-	C(18)	1.425(13)
C(1)	-	C(6)	1.375(19)	C(19)	-	C(20)	1.388(13)
C(2)	-	C(3)	1.408(16)	C(19)	-	C(24)	1.428(14)
C(3)	-	C(4)	1.392(21)	C(20)	-	C(21)	1.379(18)
C(4)	-	C(5)	1.423(19)	C(21)	-	C(22)	1.389(19)
C(5)	-	C(6)	1.420(20)	C(22)	-	C(23)	1.396(19)
C(7)	-	C(8)	1.385(14)	C(23)	-	C(24)	1.397(17)

Table 4.3 (contd.)

C(25) - C(26)	1.672(14)	C(27) - C(28)	1.658(12)
C(25) - B(1)	1.802(15)	C(27) - B(11)	1.732(16)
C(25) - B(2)	1.690(14)	C(27) - B(12)	1.751(16)
C(25) - B(5)	1.657(16)	C(27) - B(15)	1.733(13)
C(25) - B(10)	1.680(22)	C(27) - B(19)	1.692(16)
C(26) - B(1)	1.828(14)	C(28) - B(15)	1.706(16)
C(26) - B(2)	1.726(19)	C(28) - B(16)	1.685(15)
C(26) - B(3)	1.712(19)	C(28) - B(19)	1.702(15)
C(26) - B(7)	1.747(19)	C(28) - B(20)	1.702(19)
B(1) - B(5)	1.769(18)	B(11) - B(12)	1.776(18)
B(1) - B(7)	1.825(16)	B(11) - B(14)	1.771(19)
B(1) - B(9)	1.789(20)	B(11) - B(17)	1.789(20)
B(2) - B(3)	1.756(20)	B(11) - B(19)	1.756(19)
B(2) - B(4)	1.766(22)	B(12) - B(13)	1.779(17)
B(2) - B(10)	1.782(19)	B(12) - B(14)	1.740(22)
B(3) - B(4)	1.790(24)	B(12) - B(15)	1.792(18)
B(3) - B(7)	1.816(22)	B(13) - B(14)	1.771(19)
B(3) - B(8)	1.781(24)	B(13) - B(15)	1.774(21)
B(4) - B(6)	1.802(23)	B(13) - B(16)	1.770(20)
B(4) - B(8)	1.853(26)	B(13) - B(18)	1.777(21)
B(4) - B(10)	1.798(21)	B(14) - B(17)	1.857(21)
B(5) - B(6)	1.773(22)	B(14) - B(18)	1.805(18)
B(5) - B(9)	1.790(20)	B(15) - B(16)	1.772(18)
B(5) - B(10)	1.779(22)	B(16) - B(18)	1.787(20)
B(6) - B(8)	1.762(24)	B(16) - B(20)	1.807(18)
B(6) - B(9)	1.785(28)	B(17) - B(18)	1.817(16)
B(6) - B(10)	1.828(23)	B(17) - B(19)	1.721(21)
B(7) - B(8)	1.761(26)	B(17) - B(20)	1.744(18)
B(7) - B(9)	1.775(21)	B(18) - B(20)	1.798(19)
B(8) - B(9)	1.800(23)	B(19) - B(20)	1.758(17)

TABLE 4.4 Selected interbond angles (^{o})

P(1) - PT - CL	90.58(9)	P(2) - PT - CL	106.00(8)
B(1) - PT - CL	163.98(28)	P(2) - PT - P(1)	163.40(8)
B(1) - PT - P(1)	73.59(28)	C(1) - P(1) - PT	120.50(31)
C(7) - P(1) - PT	117.68(29)	C(25) - P(1) - PT	93.16(32)
B(1) - PT - P(2)	89.88(28)	C(13) - P(2) - PT	108.36(29)
C(19) - P(2) - PT	109.90(29)	C(27) - P(2) - PT	121.03(28)
C(7) - P(1) - C(1)	109.30(43)	C(25) - P(1) - C(1)	107.16(47)
C(2) - C(1) - P(1)	118.48(78)	C(6) - C(1) - P(1)	121.41(82)
C(25) - P(1) - C(7)	106.10(42)	C(8) - C(7) - P(1)	117.16(69)
C(12) - C(7) - P(1)	121.04(70)	C(19) - P(2) - C(13)	112.20(40)
C(27) - P(2) - C(13)	102.89(38)	C(14) - C(13) - P(2)	113.01(70)
C(18) - C(13) - P(2)	126.55(72)	C(27) - P(2) - C(19)	102.60(39)
C(20) - C(19) - P(2)	116.24(71)	C(24) - C(19) - P(2)	124.72(69)
C(6) - C(1) - C(2)	119.98(106)	C(3) - C(2) - C(1)	120.01(103)
C(5) - C(6) - C(1)	121.63(124)	C(4) - C(3) - C(2)	119.85(110)
C(5) - C(4) - C(3)	120.65(128)	C(6) - C(5) - C(4)	117.45(132)
C(12) - C(7) - C(8)	121.80(88)	C(9) - C(8) - C(7)	118.75(92)
C(11) - C(12) - C(7)	120.40(102)	C(10) - C(9) - C(8)	118.30(102)
C(11) - C(10) - C(9)	122.05(113)	C(12) - C(11) - C(10)	118.57(113)
C(18) - C(13) - C(14)	120.37(87)	C(15) - C(14) - C(13)	119.21(100)
C(17) - C(18) - C(13)	120.74(98)	C(16) - C(15) - C(14)	119.60(113)
C(17) - C(16) - C(15)	120.62(107)	C(18) - C(17) - C(16)	119.41(107)
C(24) - C(19) - C(20)	119.04(87)	C(21) - C(20) - C(19)	120.65(105)
C(23) - C(24) - C(19)	119.66(99)	C(22) - C(21) - C(20)	120.73(122)
C(23) - C(22) - C(21)	119.98(122)	C(24) - C(23) - C(22)	119.79(112)

TABLE 4.5 a) Deviations of atoms (Å$\times 10^3$) from, and the equation of, the weighted least-squares plane defined by the atoms Pt, Cl, P(1), P(2), and B(1)

Pt 0, Cl 0, P(1) -17, P(2) -13, B(1) 83,

C(25) 27, C(27) -73, C(28) -657

$$0.844X - 0.330Y + 0.424Z = 1.981^*$$

b) Selected intramolecular distances[+] (Å)

Pt	··· C(25)	3.00	Pt	··· B(5)	3.24
Pt	··· C(26)	3.25	Cl	··· C(28)	3.24
P(1)	··· B(1)	2.61	P(2)	··· C(28)	3.00
P(1)	··· C(26)	2.83	P(2)	··· B(15)	3.09
P(1)	··· B(5)	2.88	P(2)	··· B(19)	3.04

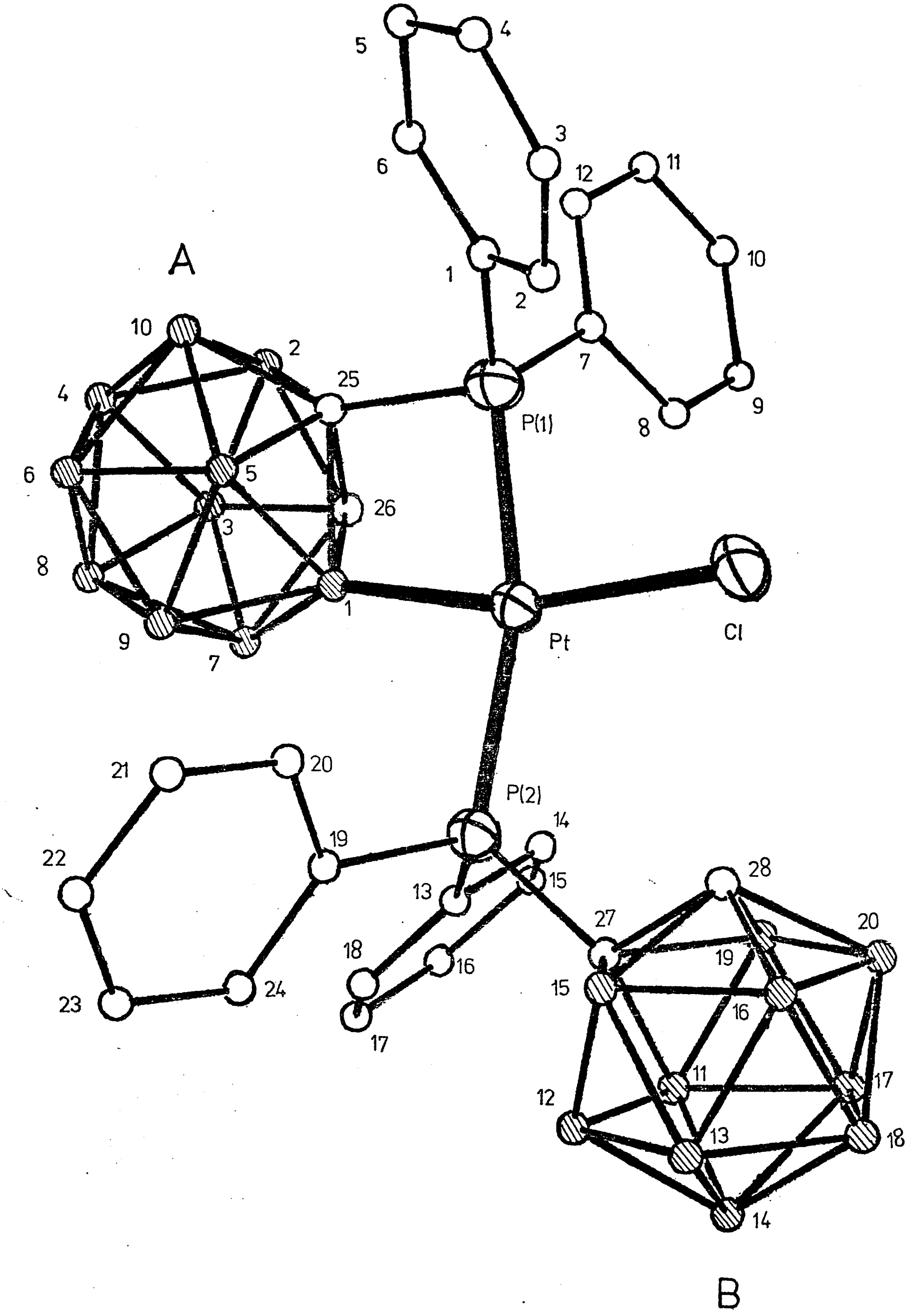
X,Y,Z, refer to an orthogonal co-ordinate system defined by a, b, and c.

[+] There are no intermolecular distances shorter than the sum of the corresponding van der Waals radii.

Legend

FIGURE 4.1 A perspective view of the molecular structure of $PtCl(Ph_2PC_2B_{10}H_{10})(Ph_2PC_2B_{10}H_{11})$. The vibrational ellipsoids of the Pt, Cl, and P atoms display 50% probability. For clarity , carbon and boron atoms are represented, respectively, by open and diagonally-shaded circles of arbitrary size; they are labelled by numbers only, corresponding to those listed in Table 4.1.

FIGURE 4.1



5
4
3
6
11
12
10
A
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2
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2
7
25
4
P(1)
9
8
6
5
26
3
8
Cl
1
Pt
9
7
21
20
P(2)
14
19
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28
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16
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24
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18
14
B

4.3 Discussion

The crystals are composed of discrete, monomeric $PtCl(Ph_2PC_2B_{10}H_{10})(Ph_2PC_2B_{10}H_{11})$ molecules and of disordered solvent molecules, separated by normal van der Waals contacts. The solvent was not identified in the course of the analysis. The chemical history of the sample suggests that it might be acetone. A view of the molecular structure of $PtCl(Ph_2PC_2B_{10}H_{10})(Ph_2PC_2B_{10}H_{11})$ is shown in Figure 4.1. The platinum atom is in a distorted square-planar environment with two phosphine ligands trans to one another. The ortho-carborane substituent of one phosphine is metallated to form a four-membered Pt-P-C-(cage atom) ring. The geometry of each carborane group is icosahedral.

Carborane Icosahedra

a) Identification of boron and carbon atoms

The nearly ideal icosahedral C_{2v} geometry of the ortho-carborane cage is well established from X-ray structural studies.[126-134] However, the relative insensitivity of the X-ray diffraction method to the chemical character of the atoms in the carborane units makes it difficult to distinguish between carbon and boron atoms. In many derivatives of icosahedral carboranes the situation is further complicated by the presence of disorder.[126,127] Nevertheless, it is well established that the bond distances in ordered ortho-carboranes follow the trend $C-C<C-B<B-B$, although the ranges of the different types of bond lengths overlap with one another.

Accordingly, the two atoms in each icosahedron which form the shortest set of polyhedral bonds were identified as carbon atoms. This criterion is based on the assumptions (i) that there is no disorder between carbon and boron sites and (ii) that bonding to the phosphorus and platinum atoms does not effect the bond lengths within the icosahedra.

The mean polyhedral bond lengths (Å) in the two icosahedra are:

Icosahedron A [B(1)-B(10),C(25),C(26)]

C(25)	C(26)	B(5)	other atoms
1.70	1.74	1.75	$\geq$1.77

Icosahedron B [B(11)-B(20),C(27),C(28)]

C(27)	C(28)	B(19)	other atoms
1.71	1.69	1.73	$\geq$1.76

For icosahedron B the geometrical criterion strongly suggests that C(27) and C(28) are indeed carbon atoms: C(27)-C(28) [1.658(12)Å] is the shortest cage bond. The resulting assignment is that of a 1-P-1,2-dicarbadodecaborane, as expected. In the case of icosahedron A, the mean cage bond lengths for C(26) and B(5) are rather similar and the geometrical criterion does not give a clear-cut result. It should however be noted that switching the chemical identities of C(26) and B(5) does not alter the formulation of A as a 1-P-3-Pt-1,2-dicarbadodecaborane. The final isotropic temperature factors (Table 4.1) support the chemical identification shown in Figure 4.1. The lowest temperature factors in the polyhedra are those of C(25)-C(28), a result not expected if these are truly boron atoms. The analysis thus suggests that structure II is more likely than structure I. At the time of writing an attempt is being made by Hill and Silva-Trivino to obtain further n.m.r. evidence, which might settle this point unambiguously.

b) The geometry of the carborane cages

The C-C, C-B and B-B distances in icosahedron B agree well with corresponding distances in other <u>ortho</u>-carboranes (Table 4.6). Some rather untypical bond lengths are found in icosahedron A: thus the bonds C(25)-B(1) and C(26)-B(1) [respectively 1.802(15) and 1.828(14)Å] are unusually long for <u>ortho</u>-carborane C-B bonds (see Table 4.6), while C(25)-C(26) [1.672(14)Å] is slightly longer than C(27)-C(28) [1.658(12)Å]. These bond lengths may reflect a distortion of icosahedron A, arising from a radial displacement of the B(1) atom towards the platinum atom. Moreover, although the polyhedral angles approximate regular icosahedral values

TABLE 4.6 Mean bond lengths (Å) in *ortho*-carboranes[a]

Compound	C-C	C-B	B-B	Ref
$(o\text{-}B_{10}H_{10}(CCH_2Br)_2$	1.64	1.72	1.77	128
$(o\text{-}C_2B_{10}H_{11})_2$	1.70[b]	1.72	1.77	126
$o\text{-}B_{10}Cl_8H_2C_2H_2$	1.67	1.71	1.79	129
$o\text{-}B_{10}Br_2H_8C_2H_2$	1.63	1.72	1.76	130
$o\text{-}B_{10}Br_3H_7C_2H_2$	1.66[b]	1.71[b]	1.78	127
$o\text{-}B_{10}Br_4H_2C_2(CH_3)_2$	1.65	1.72	1.77	131
Carborane cage A				
$PtCl(Ph_2PC_2B_{10}H_{10})$-$(Ph_2PC_2B_{10}H_{11})$	1.67(1)	1.73(2)	1.80(1)	This work
Carborane cage B				
$PtCl(Ph_2PC_2B_{10}H_{10})$-$(Ph_2PC_2B_{10}H_{11})$	1.66(1)	1.71(1)	1.78(1)	This work

[a] The estimated standard deviation, presented in parentheses, of the mean of N independent observations is given by the expression $\sigma^2 = \sum_{i=1}^{N}(x_i-\bar{x})^2/(N-1)N$, where x_i is the i^{th} and $\bar{x}$ the mean value.

[b] Includes atomic sites for which disorder is likely.

of 60 and 108° [icosahedron A: 55(1)-64(1)°, mean 60°; 100(1)-117(1)°, mean 106°; icosahedron B: 57(1)-64(1)°, mean 60°; 103(1)-117(1)°, mean 108°], the mean angles at B(1) [57 and 102°], are the smallest for any of the twenty four atoms in the two icosahedra.

The constraint imposed by the Pt,P(1),C(25),B(1) chelate ring would appear to be a major factor in the distortion of A from a normal ortho-carborane geometry.

Phosphine ligands

Each of the four phenyl rings is planar to within ±0.03Å and the mean phenyl C-C bond length of 1.40(2)Å is normal. The P-C(phenyl) distances [1.783(10)-1.829(7)Å] are on average slightly shorter than the mean value of 1.828(1)Å obtained for such bonds by Domenicano, Vaciago and Coulson;[79] they are also shorter than the P-C(cage) distances [1.834(10) and 1.888(10)Å]. As expected, the bond angles at the phosphorus atoms deviate somewhat from the ideal tetrahedral angle of 109°. In the case of P(1), its incorporation in the four-numbered chelate ring leads to an unusually small Pt-P(1)-C(25) angle of 93.2(3)°.

The platinum co-ordination

The platinum atom displays significant deviations from ideal square-planar co-ordination. Thus the atom B(1) lies 0.083Å from the metal co-ordination plane [defined by Pt,P(1),P(2),Cl, and B(1)] (Table 4.5), whereas the other donor atoms are within 0.02Å of this plane. The displacement of C(25) from the co-ordination plane is only 0.027Å. The constraint imposed by the four-membered chelate ring leads to an unusually acute P(1)-Pt-B(1) angle of 73.6(3)°. Simultaneously the P(2)-Pt-Cl angle opens to 106.0(1)°.

The disposition of icosahedron B with respect to the metal co-ordination plane is such that C(27) and C(28) are displaced from it by 0.07 and 0.66Å, respectively. This leads to an intramolecular C(28)•••Cl contact of 3.24Å. If the position of the hydrogen atom attached to C(28) is deduced on the assumption that C(28) has the stereochemistry expected for a carbon incorporated in regular carborane icosahedron, then the Cl•••H contact is estimated to be no greater than 2.4Å. This is substantially less than the sum of the appropriate van der Waals radii(<u>ca</u>. 3.0Å)[68] and may be compared with Cl•••H contacts of <u>ca</u>. 2.5Å, established by neutron diffraction analysis, in structures containing Cl•••H-N hydrogen bonds.[135] There is thus a strong possibility that Cl and C(28) are linked by a weak intramolecular hydrogen bond.

The two Pt-P bond lengths differ by 0.026(4)Å, the shorter bond being that involved in the chelate ring. Both bonds are in the range 2.28-2.32Å found for Pt-P(<u>trans</u> to P) distances in mono-tertiary phosphine platinum(II) complexes.[86]

The Pt-B(1) distance is 2.073(9)Å. This appears to be the first determination of a Pt^{II}-B(carborane) σ-bond length. It is slightly shorter than the Pt-C(<u>ortho</u>-carborane) distance of 2.13(1)Å found in $1-[(P\text{-}n\text{-}Pr_3)\ Pt^{II}(P\text{-}n\text{-}Pr_2CHCH_2CH_3)]-2-C_6H_5-1,2-(\sigma\text{-}B_{10}C_2H_{10})$,[136] and it is similar to the mean value of 2.08Å recently proposed for Pt^{II}-C_{sp^3} bond lengths subject to low *trans*-influence.[137] The Pt-Cl(*trans* to B) bond length of 2.415(3)Å lies at the upper end of the range of values observed for Pt-Cl distances (2.28-2.45Å),[48] and it indicates that the *trans*-influence of a σ-bonded boron atom is high.(At this point it is worth noting that high *trans*-influence , on Pt-H bond, of carborane cage σ-bonded to platinum atom through a carbon atom has been suggested on the basis of J(Pt-H) n.m.r. data.[138]) Indeed, the Pt-Cl(*trans* to C) distances in *trans*-$PtCl(CH_2SiMe_3)(PMe_2Ph)_2$ is also 2.415(5)Å,[56] indicating that σ-bonded boron and sp^3-hybridised carbon atoms have comparable *trans*-influence on Pt-Cl bonds. For alkyl, and now by extrapolation for σ-bonded boron, high *trans*-influence is thought to arise from the strongly covalent nature of the bond formed with platinum.[46]

This work provides the first structural example of a 1-phosphino-*ortho*-carborane ligand forming a four-membered metal-P-C-(cage atom) chelate ring.The details of the molecular geometry suggest, but do not conclusively prove, that the cage atom incorporated in the ring is boron rather than carbon. In most complexes containing *ortho*-carboranes, the metal-carborane linkage involves a M-C σ-bond,e.g. ML_2X(σ-carborane) [M=Pt,Pd ;L=PEt_3,PPh_3,PPh_2Me, $PPhMe_2$;X=Cl,H;σ-carborane=2-R-1,2- or 7-R-1,7-$B_{10}C_2H_{10}$

$(R=CH_3, Ph, H)]$,[139-142] although M-carborane π-interactions[143] and M•••H-B co-ordination have also been observed.[143] The bulk of such species have been synthesised by reaction of 1-Li-ortho-carborane derivatives with metal halides, resulting in elimination of lithium halide.

More pertinent to the present work is the study by Hoel and Hawthorne of oxidative addition reactions of iridium(I) species with $1\text{-}(1,2\text{-}C_2B_{10}H_{11})PMe_2$, where extensive spectroscopic evidence indicates that iridium(I) forms an Ir-P-C-B chelate ring, by insertion of the metal into a B-H bond.[144] From these and other experiments[145] it has been concluded that 'terminal B-H groups are much more reactive with low-valent transition metal complexes than ordinary C-H groups'.[146] The molecular geometry of the platinum(II) complex described here is compatible with this observation.

CHAPTER 5

The Crystal and Molecular Structure of *cis*-Dichloro[1,2-bis(trifluoromethylthio)propane]platinum(II)

5.1 Introduction

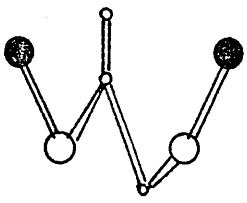
Many fluorinated bis(alkylthio)ethanes, containing electron-withdrawing groups attached to sulphur, have been prepared and characterised.[147] At present, little is known about their properties as ligands. Accordingly, Sharp and co-workers have recently prepared and spectroscopically characterized the following complexes:

I $PtCl_2(MeSCF_2CH_2SMe)$

II $PtCl_2(CF_3SC_2H_4SCF_3)$

III $PtCl_2(CF_3SCHMeCH_2SCF_3)$.[148]

Compound III contains three asymmetric atoms and the chelate ring introduces a further asymmetric centre. Hence, the compound can exist in eight diastereoisomeric forms:



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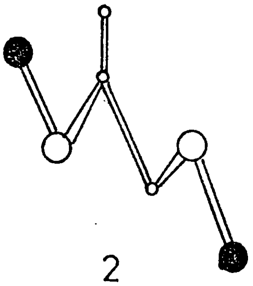

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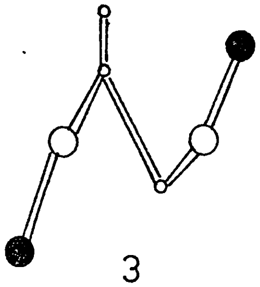

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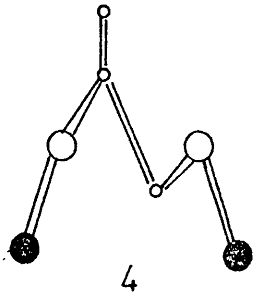

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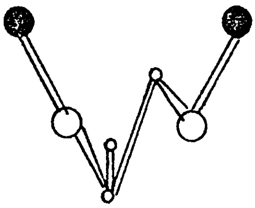

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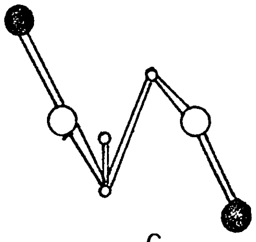

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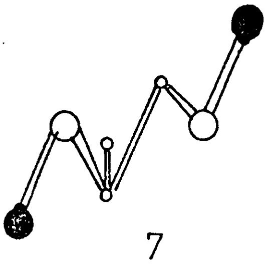

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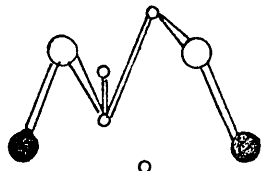

8

○ - S, ∘ - C, ● - CF_3

^{19}F n.m.r. analysis of III in acetone solution, over a large temperature range (173 - 323°K), established the presence of only four isomers.[148] These have been associated with four diastereoisomeric forms and rapid interconversion of the isomers does not appear to occur. Furthermore, long range F-F coupling has been observed for two of the isomers, presumably those with a syn arrangement of the trifluoromethyl groups.[148] It was felt that structure analysis of the complex in the solid-state might contribute to the interpretation of these results.

More relevant to the work described in this thesis is the effect of electronegative substituents attached to the sulphur atom on the metal-sulphur bonding. Strong electron-withdrawing groups linked to the sulphur atom may cause a contraction of sulphur lone pair orbitals,[149] thus reducing their availability for co-ordination; they can also promote back-bonding from the metal d orbitals of appropriate symmetry into the vacant sulphur 3d orbitals, thus strengthening the metal-sulphur bond. In this context it is worth noting that trifluoromethyl comes highest on Tolman's scale of the electron-withdrawing ability of substituent groups in phosphines.[63]

Evidence for and against the existance of $M \rightarrow S$ back-bonding is still scarce and contradictory.[150-151] Thus, Mössbauer and i.r. spectra of the complexes $C_6F_5SFe_2(CO)_6SR$ ($R = C_6H_5$ or C_6F_5) have been explained by increased metal→sulphur backbonding when R = C_6F_5.[152] Somewhat surprisingly, it has also been suggested, on the basis of ionization potential data for complexes $L_1L_2Fe_2(CO)_6$ (L_1,L_2 = SR or PR_2), that phosphorous donor ligands are less effective π-acceptors than sulphur donor ligands.[153] In contrast, Mössbauer and i.r. data for some mercapto iron derivatives suggest that the π-acidity of sulphur is smaller than that of phosphorous,[154] and π-bonding is thought to be totally absent in some metal-sulphur bonds.[155]

At present, there are very few X-ray structural studies relevant to this problem. The synthesis of the complex cis-$PtCl_2(F_3C\ SCHMeCH_2SCF_3)$ provided an opportunity to investigate the effect of trifluoromethyl substituents on the trans-influence and bonding to platinum of thioether ligands.

5.2 Results and Discussion

A full account of the structure analysis of cis-$PtCl_2(F_3CSCHMeCH_2SCF_3)$ has been published (see inside back cover), and accordingly only brief details of the experiment and salient results will be presented here.

2301 Diffractometric data ,with $I>3\sigma(I)$, corrected for Lorentz-polarisation and absorption effects, were used in the analysis. The structure was solved by the heavy-atom method and refined by the full-matrix least-squares procedure. The final structural model, involving anisotropic non-hydrogen atoms and isotropic hydrogens, gave R = 0.054.

The analysis revealed the expected cis-square-planar co-ordination at the platinum atom (Figure5.1). The methyl group attached to the C(2) atom is pseudo-equatorial relative to the almost symmetrically-puckered chelate ring. The CF_3 groups are in a syn arrangement. The relevant information for platinum (II) complexes is not available; in bis(thioether) and bis(selenoether) chelate complexes of palladium (II), however, the terminal substituents at the Group VIA donor atoms are usually in syn positions.[156,157]

Although the intermolecular distances are predominantly of the van der Waals type, there are strong interactions between pairs of centrosymmetrically related molecules (Figure 5.2). The arrangement is such that the platinum co-ordination planes are antiparallel to each other and the metal-ligand bonds are eclipsed. The Pt...Pt separation (3.42Å) is too long to reflect normal covalent bonding, and it is longer than the corresponding distances in Magnus' Green Salt (3.25Å) and related complexes.[158] In this context it is worth mentioning that a semi-empirical M.O. calculation on Magnus' Green Salt indicates that the covalent Pt - Pt bond order is about 0.04.[159] The Cl...S separations (3.35 and 3.38Å) are slightly shorter than the Pt...Pt contact, thus introducing a small pyramidal distortion of the co-ordination geometry of platinum. The pairing of the centrosymmetrically related molecules may therefore be explained by a weak electrostatic interaction between sulphur and chloro ligands.

The Pt-Cl distances are at the lower end of the range of values observed for terminal Pt^{II}-Cl distances (2.26 - 2.45Å).[48] Thus, the trans-influence of the thioether ligand is relatively weak in this compound. The Pt-S distances [2.239(3) and 2.260(3)Å] are shorter than the sum of the appropriate covalent radii (ca. 2.34Å)[68] and the longer bond is adjacent to the chelate-ring methyl substituent. The effect of trifluoromethyl groups on the metal-ligand bonding is difficult to establish, for very little comparative structural information is available (Table 5.1). Furthermore, the influence of the strong S...Cl intermolecular interactions on the bonding within the molecule is difficult to assess.

Of the compounds containing mutually trans sulphur and chlorine atoms (Table 5.1), cis-$PtCl_2[S(p-C_6H_4Cl)_2]_2$, where the Pt-S and Pt-Cl bond lengths are 2.285(7) and 2.300(5)Å, is perhaps most closely related to cis-$PtCl_2$-($CF_3SCHMeCH_2SCF_3$). The comparison of bond lengths in the two compounds suggests that the electron-withdrawing trifluoromethyl groups may be responsible for a slight shortening of the Pt-S bonds in the latter. It should however be noted that a Pt-S distance of 2.26Å has been reported for cis-$PtCl_2[MeSCH_2CH_2(COOH)CNH_2]$, although the accuracy of this result is low. From Table 5.1 it is also apparent that

TABLE 5.1 Selected bond lengths (Å) in some platinum(II) complexes containing mutually trans sulphur and chlorine ligands

Compound	Pt-S	Pt-Cl(trans to S)	Ref.
cis-$PtCl_2[MeSCH_2CH_2(COOH)CNH_2]$	2.26	2.32	160
cis-$PtCl_2[Me_2(O)SCH_2CH_2(COOH)CNH_2]$	2.198(2)	2.323(2)	161
$PtCl_3(DMSO)^a$	2.193(5)	2.318(5)	162
cis-$PtCl_2(DMSO)_2{}^a$	2.229(2) 2.244(2)	2.312(2) 2.306(3)	163
cis-$PtCl_2(NH_3)(DMSO)^a$	2.186(4)	2.321(5)	164
cis-$PtCl_2$(2-picoline)$(DMSO)^a$	2.200(3)	2.307(4)	165
cis-$PtCl_2[S(p\text{-}C_6H_4Cl)_2]_2$	2.292(6) 2.278(7)	2.298(7) 2.301(6)	166
cis-$PtCl_2[CF_3SCHMeCH_2SCF_3]$	2.260(3) 2.239(3)	2.290(4) 2.295(3)	This work

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[a] Dimethylsulphoxide, Me_2SO

Pt-S(DMSO) bonds are substantially shorter than Pt-S(thioether) bonds, but that the trans-influence of both types of sulphur-donor ligands on Pt-Cl distances is comparable. The conformations of the two trifluoromethyl groups are similar (corresponding torsion angles about the S-C bonds agree to within 8°), and such that the C-F bonds involving the F(3) and F(6) atoms are pointed towards each other so that the F(3)...F(6) non-bonding contact is 2.82Å. This is somewhat greater than the sum of the van der Waals radii (ca. 2.70Å),[68] but nevertheless may be responsible for the F—F coupling observed in acetone solution. The C-F bond lengths and F-C-F angles, with respective means of 1.305(7)Å and 107.7(6)°, are normal. The S-C-F angles involving F(3) and F(6) atoms are some 5° larger than the other S-C-F angles.

The co-ordination of the sulphur atoms is approximately tetrahedral. The four S-C bond lengths agree to within experimental error, the mean value of 1.835(Å) being typical for a bond of unit order.

The chelate ring has a symmetrical-puckered gauche conformation, as evident from the C-C-S-Pt and C-S-Pt-S angles [C(2)C(3)S(1)Pt -37(1),C(3)C(2)S(2)Pt -33(1)°; C(3)S(1)PtS(2) 11(0.5),C(2)S(2)PtS(1) 10(0.5)°].

The molecules of cis-$PtCl_2(CF_3SCHMeCH_2SCF_3)$ contain four chiral centres: the asymmetric atoms S(1), S(2) and C(2), and the chelate ring. Thus eight enantiomeric pairs of diastereoisomers may exist. The crystalline form studied here contains equal numbers of enantiomeric molecules. In those in which the chelate configuration is δ [S(1)C(3)C(2)S(2) torsion angle +47°, as in Figure 5.1], the absolute configurations at S(1), S(2) and C(2) are respectively (R),(S) and (S).

Legends

FIGURE 5.1 A perspective view of the molecular structure of *cis*-$PtCl_2(F_3CSCHMeCH_2SCF_3)$. The vibrational ellipsoids display 50% probability. Hydrogen atoms are omitted for clarity.

FIGURE 5.2 Molecular packing in a crystal of *cis*-$PtCl_2(F_3CSCHMeCH_2SCF_3)$. For clarity, atoms are represented by circles of arbitrary size. Hydrogen atoms are omitted.

FIGURE 5.1

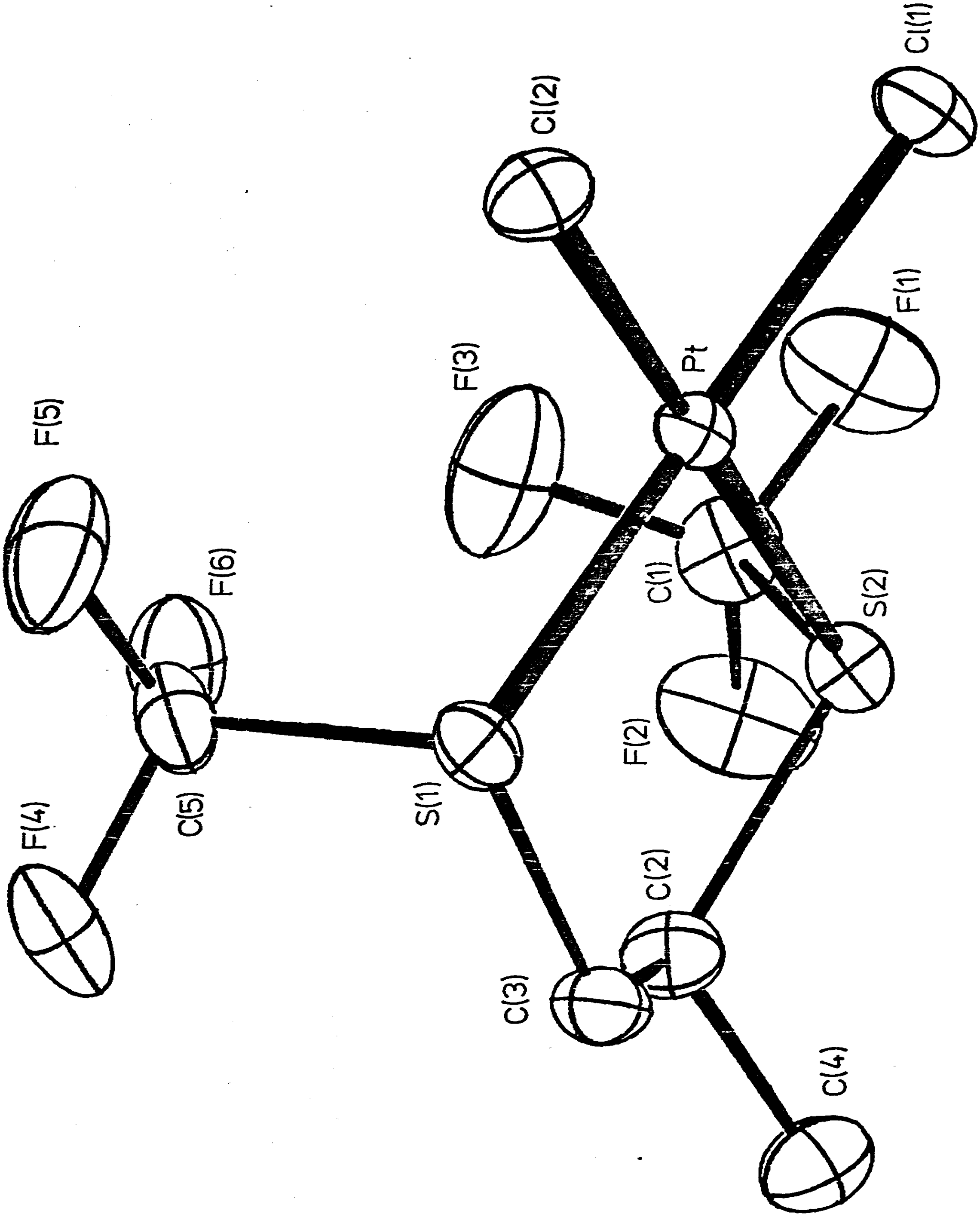
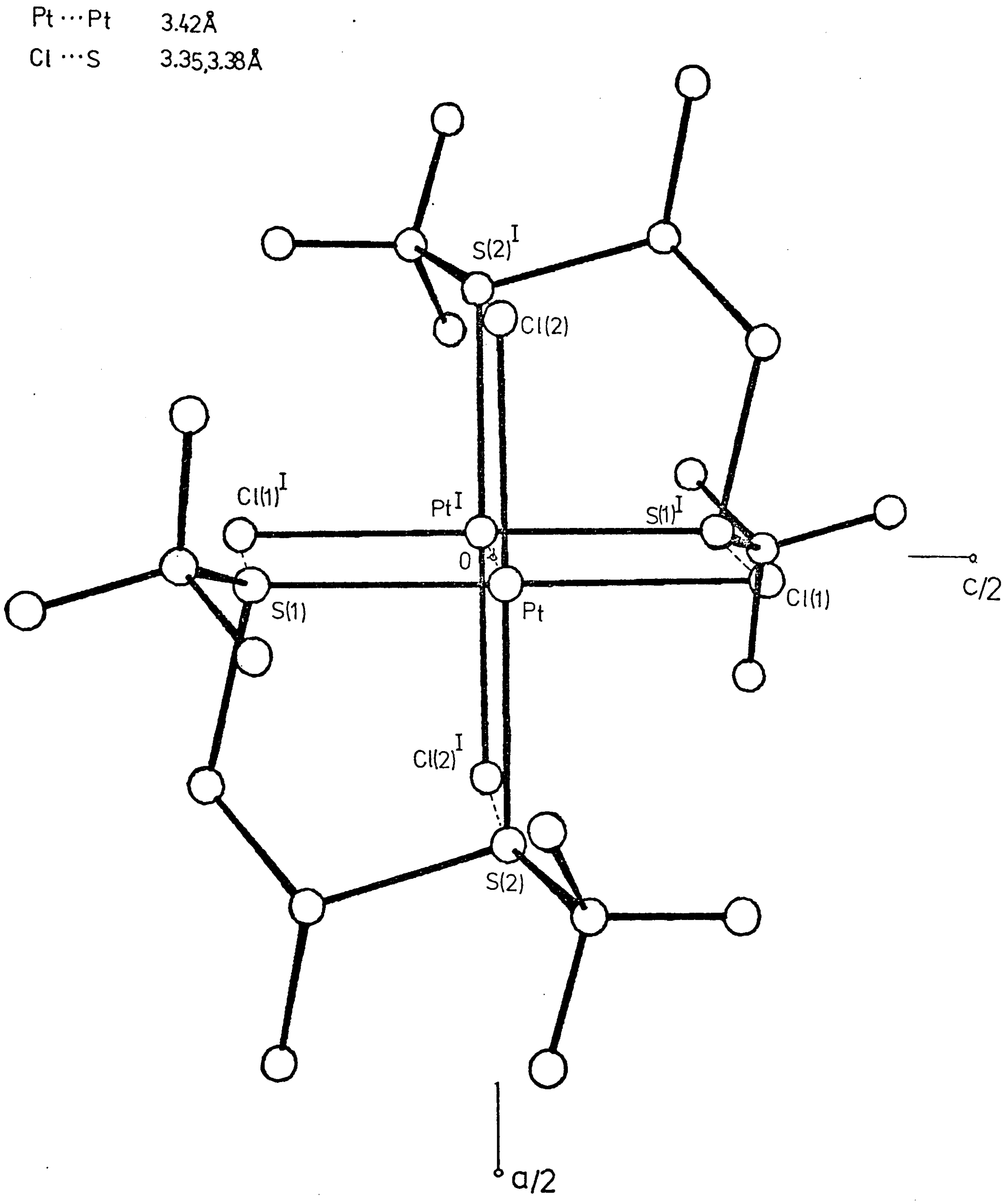


FIGURE 5.2



I -X, -Y, -Z

CHAPTER 6

The Crystal and Molecular Structure of Di-μ-chlorodi(propionyl)bis(dimethylphenylphosphine)diplatinum(II)

6.1 Introduction

The compound $[Pt_2(\mu\text{-}Cl)_2(COEt)_2(PMe_2Ph)_2]$ was prepared in this department by Dr. R.J. Cross and Mr. G.K. Anderson who also examined its ^{31}P n.m.r. spectra in various solvents. These spectra indicate that the compound exists in solution as a mixture of readily-interconvertable cis- and trans-isomers. The most remarkable features of the spectra are the $^1J(Pt\text{-}P)$ coupling constants, which are ca. 5400Hz for both isomers. The values are at the upper end of the range for $^1J(Pt\text{-}P)$ coupling constants for halogen-bridged binuclear platinum(II) complexes containing tertiary phosphines.[167-169] They are almost twice as large as the $^1J(Pt\text{-}P)$ values for mononuclear cis-dichloro(monotertiary-phosphine)platinum(II) species.[170]

In binuclear complexes the coupling constants do not depend only on the nature of the halide bridge, being much lower in tetrahalide species than in di-organodihalide complexes $\{$e.g. trans-$[Pt_2(\mu\text{-}Cl)_2Cl_2(PMe_2Ph)_2]$, $^1J(Pt\text{-}P) = 3931Hz$[169]$\}$. It has therefore been suggested that organic ligands indirectly influence the Pt-P bonding by weakening the halogen bridge.[168] A cis-influence mechanism seems equally plausible in view of the similar coupling constants displayed by the cis- and trans-isomers of $[Pt_2(\mu\text{-}Cl)_2(COEt)_2(PMe_2Ph)_2]$. In mononuclear complexes variations of ca. 1000Hz in $^1J(Pt\text{-}P)$ values due to change of cis-ligand have been observed.[170]

To examine the bridge-weakening effect of organic ligands, and also because of the proposed correlation between Pt-P coupling constants and bond lengths,[45] a structure analysis of <u>trans</u>-$[Pt_2(\mu\text{-}Cl)_2(COEt)_2(PMe_2Ph)_2]$ has been carried out. Only this isomer is obtained on recrystallisation from methylene chloride/ether.

Up till recently the discussion of the mutual influence of ligands has been concentrated on mononuclear species[46,47,49] and the results of this analysis are pertinent to any consideration of such effects in halogen-bridged binuclear complexes.

6.2 Experimental

Crystal Data

Formula	trans-$[Pt_2(\mu\text{-}Cl)_2(COEt)_2(PMe_2Ph)_2]$
Formula weight	851.3
Crystal system	triclinic
a(Å)	7.839(1)
b(Å)	8.531(1)
c(Å)	11.892(1)
α(°)	73.88(1)
β(°)	73.19(1)
γ(°)	65.05(1)
Cell volume (Å^3)	679.06
No. of mol. per cell	1
Calculated density (g cm^{-3})	2.082
μ(Mo-K$_\alpha$) (cm^{-1})	107.2
Space group	$P\bar{1}$
Equivalent positions	xyz, $\bar{x}\bar{y}\bar{z}$
Molecular symmetry	C_i

Data Collection Summary

Temperature (^{o}C)	20
Diffractometer	Enraf-Nonius CAD-4F
Radiation	graphite-monochromatised Mo-K_{α} (λ=0.71069Å)
Counter	scintillation with pulse-height analyser
Scan	$\theta/2\theta$
Background measurement	moving crystal-moving counter
θ_{max} (^{o})	30
q (see Part I, Ch.3)	0.04
No. of reflections with $I>3\sigma(I)$, n	3194
No. of parameters, m	136
n/m	23.5

Measurements

The photographic measurements were in accord with triclinic symmetry. The preliminary cell dimensions obtained from oscillation and Weissenberg photographs were later refined on an Enraf-Nonius CAD-4 diffractometer from the setting angles of 25 reflections [with $14<\theta<19^{\circ}$] from diverse regions of reciprocal space. The structure was satisfactorily refined in the centrosymmetric space group $P\bar{1}$.

The intensities of all independent reflections [in the range $2\leq\theta\leq30^{\circ}$] were measured by a symmetrical $\theta/2\theta$ scan. The θ scan width (in degrees) of $0.06 + 0.35\tan\theta$ was increased by 25% at each of the scan ends to allow for background measurements. The maximum counting time was 60 s, but for stronger reflections it was adjusted so that $\sigma(I)/I$ was 0.03. In order to check the crystal and system stability two standard reflections were remeasured periodically throughout the experiment. No significant change either in intensity or orientation was observed during the experiment. Of the 3762 intensity measurements, 3194, for which $I>3\sigma(I)$, were used in the analysis.

Data were corrected for Lorentz, polarisation and absorption effects. The transmission factors on $|F_o|^2$, derived by Gaussian integration and a grid of 640 points, were in the range 0.17 - 0.60. The crystal used was plate-shaped, the largest faces being (001) and $(00\bar{1})$.

Structure analysis

The position of the platinum atom was derived from the three-dimensional Patterson synthesis. The positions of the other non-hydrogen atoms were obtained from a subsequent difference synthesis. Full-matrix least-squares adjustment of the positional and isotropic thermal parameters converged with R = 0.139. When atomic thermal vibrations were treated anisotropically R fell to 0.095. Data were then corrected for absorption effects, giving R = 0.028. Hydrogen atoms were found from the subsequent difference synthesis and were included in the further calculations but their parameters were not refined. The final values of R and R_w were 0.026 and 0.033. A final difference synthesis contained regions of ± 1.6 $e\text{Å}^{-3}$ close to platinum; elsewhere function values were within ± 0.8 $e\text{Å}^{-3}$. Mean values of $(|F_o| - |F_c|)^2/\sigma^2(|F_o|)$ showed no significant variation with $|F_o|$ or sin θ. Extinction correction did not appear to be necessary.

TABLE 6.1 Final atomic fractional co-ordinates and thermal parameters (Å^2) of non-hydrogen atoms. The form of the temperature factor is $\exp(2\pi^2 \times 10^{-3} \sum_{i=1}^{3} \sum_{j=1}^{3} h_i h_j a_i^* a_j^* U_{ij})$

Atom	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pt(1)	0.05445(2)	-0.16072(2)	0.13492(1)	35.0(1)	36.2(1)	32.0(1)	-10.5(1)	-4.0(1)	-2.1(1)
Cl(1)	0.1561(2)	0.0800(2)	-0.0001(1)	55.0(6)	57.6(7)	46.4(6)	-29.8(6)	-18.8(5)	10.4(5)
P(1)	0.2468(2)	-0.2397(2)	0.2613(1)	42.4(6)	39.9(5)	39.5(6)	-10.7(5)	-9.7(5)	-4.8(5)
O(1)	-0.1465(7)	-0.3321(6)	0.3303(4)	76(3)	78(3)	46(2)	-41(2)	8(2)	-10(2)
C(1)	-0.0431(7)	-0.3446(6)	0.2330(4)	44(2)	47(2)	40(2)	-17(2)	-8(2)	1(2)
C(2)	0.0073(10)	-0.4998(8)	0.1735(6)	81(4)	58(3)	52(3)	-34(3)	-8(3)	-9(2)
C(3)	-0.0103(14)	-0.6608(9)	0.2598(8)	135(7)	60(4)	95(6)	-49(4)	-35(5)	2(4)
C(4)	0.1231(10)	-0.1829(8)	0.4060(5)	75(4)	63(3)	45(3)	-13(3)	-9(3)	-22(2)
C(5)	0.4232(10)	-0.1395(9)	0.2131(7)	67(4)	66(4)	91(5)	-35(3)	-32(3)	4(3)
C(6)	0.3846(7)	-0.4749(6)	0.2852(5)	45(2)	38(2)	45(2)	-6(2)	-13(2)	-5(2)
C(7)	0.4995(10)	-0.5464(9)	0.1847(6)	67(4)	66(4)	55(3)	0(3)	-8(3)	-15(3)
C(8)	0.6027(10)	-0.7277(10)	0.1999(8)	69(4)	72(4)	96(6)	11(3)	-9(4)	-44(4)
C(9)	0.5946(11)	-0.8345(8)	0.3074(8)	71(4)	42(3)	107(6)	-3(3)	-30(4)	-15(3)
C(10)	0.4777(11)	-0.7604(8)	0.4055(7)	84(4)	46(3)	89(5)	-23(3)	-30(4)	11(3)
C(11)	0.3738(9)	-0.5815(7)	0.3953(5)	65(3)	49(3)	54(3)	-18(2)	-9(3)	-2(2)

TABLE 6.2 Fractional co-ordinates of hydrogen atoms. The isotropic temperature parameter assigned to each hydrogen atom was 0.08Å^2.

Atom[+]	x	y	z
H(1)C(2)	0.142	-0.512	0.120
H(2)C(2)	-0.056	-0.455	0.103
H(3)C(3)	0.081	-0.703	0.322
H(4)C(3)	-0.160	-0.628	0.309
H(5)C(3)	0.021	-0.772	0.218
H(6)C(4)	0.028	-0.235	0.437
H(7)C(4)	0.219	-0.234	0.462
H(8)C(4)	0.061	-0.052	0.399
H(9)C(5)	0.358	-0.005	0.199
H(10)C(5)	0.510	-0.180	0.272
H(11)C(5)	0.500	-0.180	0.132
H(12)C(7)	0.526	-0.474	0.091
H(13)C(8)	0.689	-0.784	0.122
H(14)C(9)	0.660	-0.963	0.316
H(15)C(10)	0.492	-0.839	0.479
H(16)C(11)	0.294	-0.547	0.468

[+] H(n)C(m) is hydrogen atom attached to carbon atom C(m)

TABLE 6.3 Selected bond lengths (Å) +

PT(1) - CL(1)	2.498(1)	C(1) - C(2)	1.526(7)
PT(1) - CL(1')	2.393(1)	C(2) - C(3)	1.501(10)
PT(1) - P(1)	2.209(1)	C(6) - C(7)	1.389(8)
PT(1) - C(1)	1.972(5)	C(6) - C(11)	1.375(8)
P(1) - C(4)	1.799(6)	C(7) - C(8)	1.398(10)
P(1) - C(5)	1.800(6)	C(8) - C(9)	1.351(12)
P(1) - C(6)	1.818(5)	C(9) - C(10)	1.379(11)
O(1) - C(1)	1.208(7)	C(10) - C(11)	1.383(8)

+ Here, and elsewhere, primed atoms are related to the corresponding unprimed atoms by the symmetry operation $\overline{x}\overline{y}\overline{z}$ applied to the co-ordinates in Table 6.1

TABLE 6.4 Selected interbond angles (^{o})

CL(1') - PT(1) - CL(1)	83.74(4)
C(1) - PT(1) - CL(1)	174.94(14)
P(1) - PT(1) - CL(1')	178.84(4)
C(1) - PT(1) - P(1)	87.62(14)
C(5) - P(1) - PT(1)	113.94(25)
O(1) - C(1) - PT(1)	123.23(39)
C(5) - P(1) - C(4)	104.34(32)
C(6) - P(1) - C(5)	104.95(25)
C(11) - C(6) - P(1)	123.07(38)
C(3) - C(2) - C(1)	113.80(53)
C(8) - C(7) - C(6)	117.96(62)
C(9) - C(8) - C(7)	122.73(73)
C(11) - C(10) - C(9)	121.38(68)
P(1) - PT(1) - CL(1)	97.42(4)
PT(1') - CL(1) - PT(1)	96.26(4)
C(1) - PT(1) - CL(1')	91.22(14)
C(4) - P(1) - PT(1)	113.58(20)
C(6) - P(1) - PT(1)	112.33(16)
C(2) - C(1) - PT(1)	115.27(35)
C(6) - P(1) - C(4)	106.94(26)
C(7) - C(6) - P(1)	116.74(41)
C(2) - C(1) - O(1)	121.32(47)
C(11) - C(6) - C(7)	120.16(47)
C(10) - C(11) - C(6)	119.64(57)
C(10) - C(9) - C(8)	118.12(61)

TABLE 6.5 Selected torsion angles (°)

CL(1') - PT(1) - CL(1) - PT(1')	0.0(0)	P(1) - PT(1) - CL(1) - PT(1')	179.9(7)
C(1) - PT(1) - CL(1) - PT(1')	-5.0(16)	CL(1) - PT(1) - P(1) - C(4)	109.7(2)
CL(1) - PT(1) - P(1) - C(5)	-9.6(3)	CL(1) - PT(1) - P(1) - C(6)	-128.8(2)
CL(1') - PT(1) - P(1) - C(4)	-74.8(22)	CL(1') - PT(1) - P(1) - C(5)	165.9(21)
CL(1') - PT(1) - P(1) - C(6)	46.7(22)	C(1) - PT(1) - P(1) - C(4)	-69.9(3)
C(1) - PT(1) - P(1) - C(5)	170.8(3)	C(1) - PT(1) - P(1) - C(6)	51.6(2)
CL(1) - PT(1) - C(1) - O(1)	-102.4(16)	CL(1) - PT(1) - C(1) - C(2)	72.8(17)
CL(1') - PT(1) - C(1) - O(1)	-107.4(4)	CL(1') - PT(1) - C(1) - C(2)	67.8(4)
P(1) - PT(1) - C(1) - O(1)	72.7(4)	P(1) - PT(1) - C(1) - C(2)	-112.2(4)
PT(1) - P(1) - C(6) - C(7)	57.7(4)	PT(1) - P(1) - C(6) - C(11)	-120.1(4)
C(4) - P(1) - C(6) - C(7)	-177.1(4)	C(4) - P(1) - C(6) - C(11)	5.2(5)
C(5) - P(1) - C(6) - C(7)	-66.6(5)	C(5) - P(1) - C(6) - C(11)	115.6(5)
PT(1) - C(1) - C(2) - C(3)	161.1(5)	O(1) - C(1) - C(2) - C(3)	-23.7(8)

TABLE 6.6 The equation of, and individual displacements of atoms (10^3x Å), from the weighted mean plane [defined by the atoms Pt(1), Pt(1'), Cl(1), Cl(1'), P(1), P(1'), C(1) and C(1')].

$$-0.313X + 0.769Y + 0.557Z = 0.000^{*}$$

Pt(1)	0.0(1),	Cl(1)	2(1),	P(1)	2(1),	C(1)	13(5),
Pt(1')	0.0(1),	Cl(1')	-2(1),	P(1')	-2(1),	C(1')	-13(5)

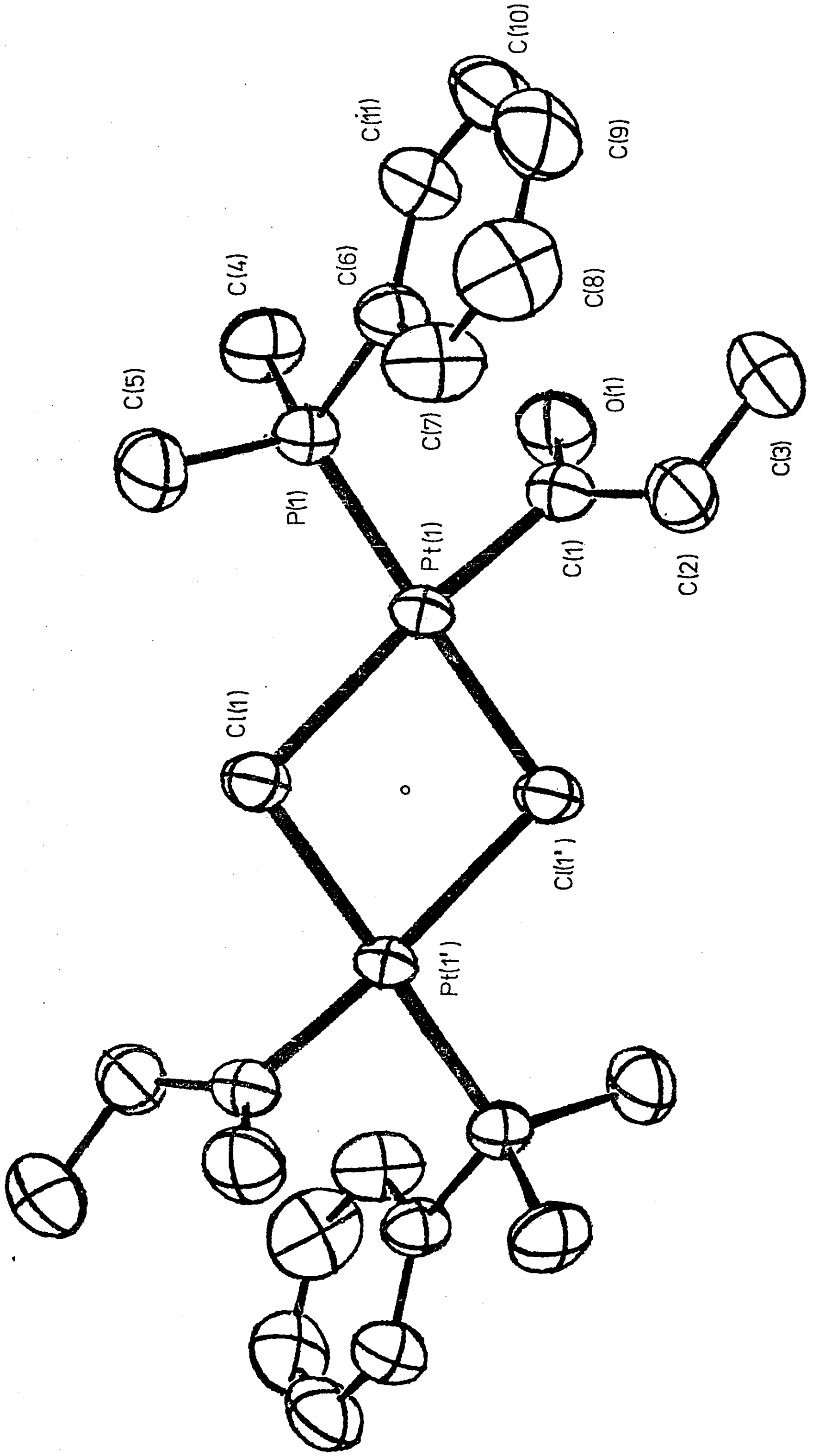
---

* X,Y,Z, refer to an orthogonal co-ordinate system defined by a*,b, and c.

Legend

FIGURE 6.1 A perspective view of the molecular structure of trans-$[Pt_2(\mu\text{-}Cl)_2(COEt)_2(PMe_2Ph)_2]$. The thermal ellipsoids display 50% probability. Hydrogen atoms are omitted for clarity. The small circle represents a crystallographic centre of symmetry.

FIGURE 6.1



6.3 Discussion

The crystals are built from discrete binuclear molecules separated by normal van der Waals contacts. Final atomic parameters and a selection of functions derived from them are presented in Tables 6.1 - 6.6. An ORTEP drawing of the molecule is shown in the Figure 6.1.

Each molecule is constrained to exact C_i symmetry. This implies both that the crystals contain the trans-isomer and also that the Pt_2Cl_2 moiety is exactly planar. Each metal centre has a slightly distorted square-planar co-ordination, with the two planes sharing a common edge so that the two platinum and six ligand donor atoms are all within 0.013Å from their mean plane (Table 6.6). Bond lengths and angles within the PMe_2Ph ligand are unexceptional; the mean values of P-C and C-C bonds are 1.806(6) and 1.379(7)Å, respectively. The Pt-C-C and C-P-C angles are as expected, respectively greater and smaller than the tetrahedral value [mean Pt-P-C 113.7(9), C-P-C 105.4(8)°]. The conformation adopted by the ligand is such that methyl carbon C(5) lies close to the metal co-ordination plane and the phenyl ring plane passes near the atom C(4), as shown by Cl(1) - Pt(1) - P(1) - C(5) and C(4) - P(1) - C(6) - C(11) torsion angles of -9.6(3) and 5.2(5)°, respectively. Bond lengths and valency angles within the propionyl ligand are also normal. The P(1) - Pt(1) - C(1) - O(1) and Pt(1) - C(1) - C(2) - C(3) torsion angles are

72.4(4) and 161.1(5)°. This conformation leads to a somewhat short intramolecular Pt...H contact, involving a hydrogen atom attached to C(2), of 2.8Å.

Examination of the metal-ligand bond lengths provides two interesting points. First, despite the large ^1J(Pt-P) coupling constant (ca.5400Hz), the Pt(1)-P(1) distance [2.209(1)Å] is only slightly shorter than the range of comparable Pt-P(trans to Cl) bond lengths [2.21 -2.28Å] in mononuclear complexes containing alkyl- or aryl-substituted tertiary phosphines.[170] Indeed, it is equal, to within experimental error, to the shortest such value, 2.214(1)Å in the anion $[PtCl_3(PEt_3)]^-$, which displays a $^1J(Pt\text{-}PEt_3)$ coupling constant of 3704Hz,[60] some 30% smaller than that for trans-$[Pt_2(\mu\text{-}Cl)_2(COEt)_2(PMe_2Ph)_2]$. This suggests that the proposed correlation between Pt-P coupling constants and bond lengths[45] breaks down if the complexes compared differ greatly. Second, the substantial difference between the two Pt-(μ-Cl) bond lengths [0.105(2)Å] indicates that σ-propionyl exerts a much greater trans-influence than phosphine on the bridging chlorine ligands. This is a somewhat surprising result, since Pt-Cl distances in mononuclear complexes [2.36 - 2.39Å] trans to tertiary phosphine are only slightly shorter than the corresponding distances [2.40 - 2.42Å] trans to σ-carbon donors,[47] suggesting that the trans-influence of phosphines is only slightly less than that of σ-carbon donor ligands.

The generality of these observations appears to be confirmed by an examination of metal-ligand bond lengths in trans-[$Pt_2(\mu-Cl)_2X_2Y_2$] complexes (Table 6.7).[171-179] In these molecules each bridging chlorine atom participates in two Pt-Cl bonds which are trans to different terminal ligands. The data in Table 6.7 do not indicate any significant relationship between the lengths of the two Pt-(μ-Cl) bonds formed by a given chlorine atom. Instead, each Pt-(μ-Cl) distance appears to reflect the influence of the trans-terminal ligand, according to the series $Cl < \pi-(C=C) < AsMe_3 \leq PR_3 < \sigma-C$. Apart from the previously noted disparity between PR_3 and σ-carbon donors, this series appears to be identical with that derived from mononuclear complexes.[46,49] The terminal platinum-ligand bonds in Table 6.7, including those to tertiary phosphine, appear on average to be ca. 0.03Å shorter than corresponding bonds trans to chlorine in mononuclear complexes, while the Pt-(μ-Cl) bonds are typically ca. 0.03Å longer than Pt-Cl bonds in mononuclear complexes trans to similar ligands. Thus bridging chlorine atoms form weaker bonds to platinum and have a lower trans-influence than terminal chlorine atoms. The participation of bridging chlorine atoms in two Pt-Cl bonds, compared with only one for a terminal chlorine atom, seems the obvious reason for this. In terms of the σ-trans-influence theory[46-49] bridging chlorine atoms are weaker donors, being effectively more electronegative, than terminal chlorine atoms. The features apparent in Table 6.7 therefore seem to be accounted for by current views of trans-influence.

TABLE 6.7 Bond lengths (Å) in trans-[$Pt_2(\mu\text{-}Cl)_2X_2Y_2$] complexes

Compound	X	Y	Pt-X	Pt-Y	Pt-Cl (trans to X)	Pt-Cl (trans to Y)	Ref.
$[Pt_2Cl_6]^{2-}$	Cl Cl	Cl Cl	2.265 2.280	2.279 2.268	2.318 2.337	2.330 2.327	171
$Pt_2Cl_4[CH_2C(OMe)_2]$	δ-C	Cl	2.09(3)	2.264(8)	-	2.324(7)	172
$Pt_2Cl_4(C_5H_8)_2$	π-(C=C)	Cl	2.20(2)	2.264(6)	2.349(5)	2.320(5)	173
$Pt_2Cl_4(C_7H_{12})_2$	π-(C=C)	Cl	2.12(2)	2.257(6)	2.362(6)	2.328(6)	173
$Pt_2Cl_4(C_7H_{12})_2$	π-(C=C)	Cl	2.07-2.25(2)	2.273(5)	2.383(5)	2.342(5)	174
$Pt_2Cl_4(PPr^n_3)_2$	PPr^n_3	Cl	2.230(9)	2.279(9)	2.425(8)	2.315(8)	175
$Pt_2Cl_4(AsMe_3)_2$	$AsMe_3$	Cl	2.308(2)	2.268(6)	2.394(6)	2.312(5)	176
$Pt_2Cl_2(C_{11}H_{15}O)_2$	σ-C	π-(C=C)	2.07	2.11-2.23	2.51	2.34	177
$Pt_2Cl_2(C_{12}H_9N_2)_2$	σ-C	N	1.94(2)	1.98(2)	2.460(5)	2.326(6)	178
$Pt_2Cl_2(Bu^t_2PCH_2CMe_2CH_2)_2$	σ-C	Bu^t_2P-	2.06	2.200	2.460	2.402	179
$Pt_2Cl_2(COEt)_2(PMe_2Ph)_2$	σ-C	PMe_2Ph	1.972(5)	2.209(1)	2.498(1)	2.393(1)	This work

PART III

The Crystal and Molecular Structures of Two Binuclear Platinum(I) Complexes Containing Bridged Metal-Metal Bonds

I $[PtCl(\mu\text{-dppm})]_2$

II $[Pt_2Cl(CO)(\mu\text{-dppm})_2](PF_6)$

dppm=Bis(diphenylphosphino)methane

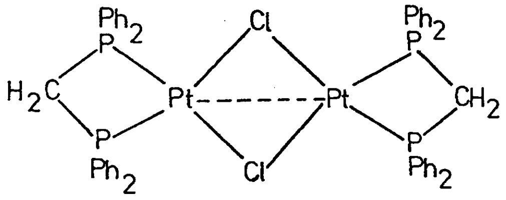
7.1 Introduction

In the past 15 years it has been abundantly demonstrated that the transition metals can form direct metal-to-metal bonds. Recent suggestions that metal clusters[180] and binuclear complexes[181] may be capable of new modes of catalytic activity have stimulated interest in their chemical behaviour and structural properties. It is also hoped that further experimental and theoretical studies of these compounds may help bridge the gap between current understanding of co-ordination and surface chemistry.

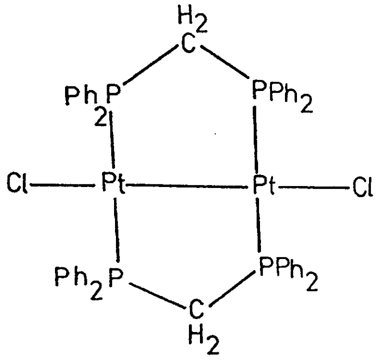
This part of the thesis describes the X-ray structure analysis of two dimeric platinum(I) complexes containing metal-metal bonds. Platinum(I) is an uncommon oxidation state of platinum. Its well-characterised complexes are still relatively rare and only a few of these have been examined crystallographically. They involve metal centres co-ordinated by a variety of ligands, displaying different electronic and steric properties, and contain metal-metal bonds with[182-186] and without[187,188] bridging groups.

The compound $[PtCl(\mu\text{-dppm})]_2$, I, where dppm = $(C_6H_5)_2PCH_2P(C_6H_5)_2$, was first obtained by Glockling and Pollock,[189] and later by Brown _et al._,[190] using different synthetic routes. Glockling and Pollock assigned it a chloride-bridged structure, (A). This, however, was subsequently questioned by Schmidbaur _et al._[191] who proposed a dppm-bridged structure, (B), by analogy with a related gold(II) complex. The latter structure was strongly supported by detailed spectroscopic evidence (n.m.r., i.r.

and Raman) of Brown *et al*.[190]

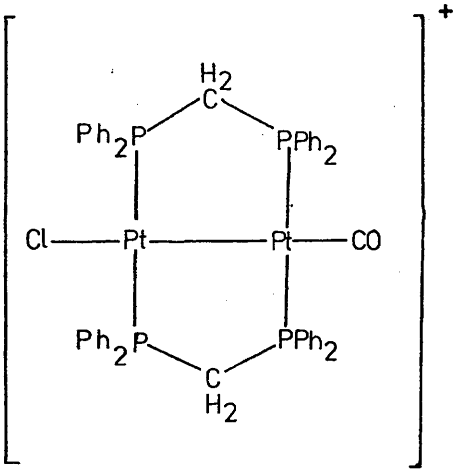


(A)



(B)

Brown *et al*. also showed that I reacts with carbon monoxide in methanol to form an ionic complex, $[Pt_2Cl(CO)(\mu\text{-dppm})_2]Cl$, which can readily be isolated as the hexafluorophosphate salt $[Pt_2Cl(CO)(\mu\text{-dppm})_2](PF_6)$, II, on addition of hexafluorophosphate.[181] On the basis of spectroscopic data (n.m.r., i.r. and Raman) they suggested structure (C) for the cationic complex.



(C)

An interesting feature revealed by formation of these two salts is the ability of carbon monoxide to compete with chloride for the platinum(I) centre; displacement of chloride by CO is not known in square-planar platinum(II) complexes.

The results of the work described here established (B) and (C) as the correct structures for the complexes I and II.

7.2 Experimental

The X-ray analyses of I and II were carried out by similar methods. Details peculiar to each are presented in the accompanying Crystal Data and Data Collection and Refinement Tables.

Measurement and structure analyses

The space group and preliminary cell dimensions for each compound were determined from rotation and Weissenberg photographs. The crystals were then transferred onto a Hilger and Watts Y-290 diffractometer, equipped with a graphite monochromator and a scintillation counter with a pulse-height analyser. Final values of cell parameters ($T = 21^{\circ}C$) were obtained by a least-squares refinement of the setting angles of 11 reflections [with $12<\theta<17^{\circ}$ for I and $11<\theta<18^{\circ}$ for II], which have been chosen from diverse regions of reciprocal space. Intensities of all independent reflections with $\theta(\text{Mo-K}_{\alpha}) \leqslant 30^{\circ}$ for I, and $\theta(\text{Mo-K}_{\alpha}) \leqslant 25^{\circ}$ for II, were measured. Symmetrical $\theta/2\theta$ scan technique and stationary crystal-stationary counter background counting were employed.

The intensities were corrected for Lorentz-polarisation effects and for the variations in intensity of three periodically-remeasured standard reflections, but not for extinction nor absorption. A satisfactory allowance for absorption was not possible because of difficulties in defining adequately the shape of the specimens. The transmission factors on $|F|^2$ were estimated to be ca. 0.3 - 0.4 for I and 0.2 - 0.4 for II.

For each compound the positions of the platinum atoms were obtained from the three-dimensional Patterson synthesis. Refinement of the positional and isotropic thermal parameters of the platinum atoms gave R = 0.25 for I and R = 0.24 for II. The positions of the other atoms, apart from those of hydrogens, were determined from subsequent difference syntheses. The structures were refined by the method of block-diagonal least-squares. The refinement converged with R and R_w values of 0.086 and 0.108 for I, and 0.082 and 0.098 for II. Anisotropic temperature factors were used only for Pt, Cl and P atoms in the case of I and for Pt, Cl, P, O, F and the carbonyl carbon atoms in the case of II. No allowance was made for the scattering of the hydrogen atoms. Final difference syntheses showed a number of peaks in the vicinity of the platinum atoms [up to 3.5 $e\text{Å}^{-3}$ for I and 3.4 $e\text{Å}^{-3}$ for II]. The mean values of $(|F_o|-|F_c|)^2/\sigma^2(|F_o|)$ showed no systematic trends when analysed as a function of $|F_o|$ or $\sin\theta$.

The final atomic parameters, and a selection of functions derived from them, are presented in Tables 7.1-7.7. Views of the molecular structures of I and II are shown in Figures 7.1 and 7.2, respectively.

Crystal Data

Compound	I	II
Formula	$[PtCl(\mu\text{-dppm})]_2$	$[Pt_2Cl(CO)(\mu\text{-dppm})_2](PF_6)$
Formula weight	1229.9	1367.4
Crystal system	monoclinic	monoclinic
a(Å)	13.592	12.919
b(Å)	16.577	15.576
c(Å)	21.438	25.151
β(°)	105.63	94.82
Cell volume (Å^3)	4651.6	5043.3
No. of mol. per cell	4	4
Calculated density (g cm^{-3})	1.756	1.801
μ(Mo-K$_\alpha$)(cm^{-1})	63.4	59.2
Space group	$P2_1/c$	$P2_1/n$(C_{2h}^5 , No. 14)
Equivalent positions	$\pm(xyz)$ $\pm(x,\frac{1}{2}-y,\frac{1}{2}+z)$	$\pm(xyz)$ $\pm(\frac{1}{2}+x,\frac{1}{2}-y,\frac{1}{2}+z)$

Summary of Data Collection and Refinement

Compound	I	II
θ_{max} (°)	30	25
Scan width $\Delta\theta$(°)	0.6	0.5
Step size in θ(°)	0.02	0.02
T_p(s)	75	50
T_b(s)	15	15
q(see Part I, Ch.3)	0.04	0.04
No. of reflections with $I > 3\sigma(I)$,n	4307	5646
No. of parameters p	274	345
n/p	15.7	16.4
R (%)	8.6	8.2
R_w (%)	10.8	9.8

TABLE 7.1 Final fractional co-ordinates of atoms and isotropic thermal parameters (Å×10^3) of carbon atoms

a) $[PtCl(\mu\text{-dppm})]_2$ (I)

Atom	x	y	z	U(iso)
Pt(1)	0.28976(6)	0.06308(6)	0.23640(4)	-
Pt(2)	0.09531(6)	0.07584(5)	0.17005(4)	-
Cl(1)	0.4674(4)	0.0546(6)	0.2953(3)	-
Cl(2)	-0.0773(4)	0.0833(5)	0.1031(3)	-
P(1)	0.3294(4)	0.0256(4)	0.1431(3)	-
P(2)	0.2498(4)	0.0899(4)	0.3302(3)	-
P(3)	0.0513(4)	0.0219(4)	0.2555(3)	-
P(4)	0.1568(4)	0.1352(4)	0.0939(3)	-
C(1)	0.237(2)	0.063(2)	0.066(1)	47(6)
C(2)	0.113(2)	0.090(2)	0.324(1)	43(6)
C(3)	0.342(2)	-0.083(2)	0.135(1)	53(7)
C(4)	0.446(2)	0.065(2)	0.131(1)	44(6)
C(5)	0.305(2)	0.014(2)	0.397(1)	49(6)
C(6)	0.285(2)	0.190(2)	0.369(1)	43(6)
C(7)	-0.085(2)	0.025(2)	0.256(1)	41(6)
C(8)	0.088(2)	-0.085(2)	0.279(1)	48(6)
C(9)	0.240(2)	0.226(2)	0.118(1)	47(6)
C(10)	0.066(2)	0.171(2)	0.017(1)	44(6)
C(11)	0.296(2)	-0.127(2)	0.079(1)	64(8)
C(12)	0.304(2)	-0.206(2)	0.077(1)	68(8)
C(13)	0.358(3)	-0.251(2)	0.128(2)	82(10)
C(14)	0.404(2)	-0.205(2)	0.187(2)	80(9)
C(15)	0.398(2)	-0.127(2)	0.191(1)	65(8)
C(16)	0.498(2)	0.027(2)	0.090(1)	55(7)
C(17)	0.582(2)	0.063(2)	0.077(2)	69(8)
C(18)	0.617(2)	0.141(2)	0.102(1)	66(8)
C(19)	0.569(2)	0.178(2)	0.142(1)	60(7)
C(20)	0.485(2)	0.143(2)	0.157(1)	50(6)
C(21)	0.346(2)	-0.048(2)	0.385(1)	57(7)
C(22)	0.382(2)	-0.110(2)	0.435(2)	72(8)
C(23)	0.383(3)	-0.087(3)	0.501(2)	121(14)
C(24)	0.332(3)	-0.022(3)	0.510(2)	115(14)
C(25)	0.296(3)	0.031(2)	0.459(2)	91(11)
C(26)	0.395(3)	0.199(3)	0.395(2)	101(12)

Table 7.1.a) (contd.)

C(27)	0.421(3)	0.280(3)	0.426(2)	96(11)
C(28)	0.358(2)	0.333(2)	0.430(2)	79(9)
C(29)	0.253(2)	0.323(2)	0.406(1)	69(8)
C(30)	0.216(2)	0.247(2)	0.373(1)	63(8)
C(31)	-0.120(3)	0.082(2)	0.296(2)	92(11)
C(32)	-0.227(3)	0.070(3)	0.295(2)	101(12)
C(33)	-0.289(2)	0.034(2)	0.252(2)	72(9)
C(34)	-0.254(2)	-0.019(2)	0.216(2)	75(9)
C(35)	-0.149(2)	-0.022(2)	0.214(1)	54(7)
C(36)	0.039(3)	-0.123(2)	0.320(2)	84(10)
C(37)	0.067(3)	-0.206(2)	0.337(2)	86(10)
C(38)	0.138(2)	-0.240(2)	0.314(2)	73(9)
C(39)	0.184(4)	-0.205(2)	0.273(1)	62(7)
C(40)	0.158(2)	-0.121(2)	0.254(1)	45(6)
C(41)	0.254(2)	0.258(2)	0.181(1)	67(8)
C(42)	0.325(3)	0.323(2)	0.199(2)	89(10)
C(43)	0.372(2)	0.354(2)	0.156(2)	78(8)
C(44)	0.360(2)	0.324(2)	0.096(1)	65(8)
C(45)	0.291(2)	0.256(2)	0.075(1)	66(8)
C(46)	0.039(2)	0.249(2)	0.011(1)	54(7)
C(47)	-0.029(2)	0.278(2)	-0.052(2)	77(9)
C(48)	-0.059(2)	0.219(2)	-0.101(1)	69(8)
C(49)	-0.028(2)	0.139(2)	-0.092(1)	60(7)
C(50)	0.035(2)	0.111(2)	-0.030(1)	47(6)

b) $[Pt_2Cl(CO)(\mu\text{-dppm})_2](PF_6)$ (II)

Atom	x	y	z	U(iso)
Pt(1)	0.31035(6)	0.37598(5)	0.18583(3)	-
Pt(2)	0.31310(6)	0.22834(5)	0.13607(3)	-
Cl(1)	0.3057(4)	0.5082(3)	0.2333(2)	-
P(1)	0.4270(4)	0.4350(3)	0.1326(2)	-
P(2)	0.2056(4)	0.3154(3)	0.2451(2)	-
P(3)	0.1376(4)	0.2308(3)	0.1460(2)	-
P(4)	0.4866(4)	0.2544(3)	0.1270(2)	-
O	0.304(2)	0.044(1)	0.0967(9)	-
C(51)	0.310(2)	0.114(2)	0.110(1)	-
P(5)	0.7342(6)	0.2592(5)	0.4540(2)	-
F(1)	0.823(2)	0.255(2)	0.424(1)	-

Table 7.1.b) (contd.)

F(2)	0.692(2)	0.178(2)	0.428(1)	-
F(3)	0.771(2)	0.346(1)	0.480(1)	-
F(4)	0.636(2)	0.262(3)	0.480(1)	-
F(5)	0.672(3)	0.307(2)	0.411(1)	-
F(6)	0.797(2)	0.216(2)	0.502(1)	-
C(1)	0.485(2)	0.358(1)	0.0918(8)	54(5)
C(2)	0.130(1)	0.225(1)	0.2170(8)	50(5)
C(3)	0.372(2)	0.516(1)	0.0870(8)	60(5)
C(4)	0.538(1)	0.490(1)	0.1677(7)	49(5)
C(5)	0.104(2)	0.378(1)	0.2712(8)	54(5)
C(6)	0.284(1)	0.279(1)	0.3037(8)	51(5)
C(7)	0.057(1)	0.144(1)	0.1174(7)	47(4)
C(8)	0.068(1)	0.325(1)	0.1229(7)	44(4)
C(9)	0.577(2)	0.267(1)	0.1872(8)	57(5)
C(10)	0.551(2)	0.178(1)	0.0858(8)	56(5)
C(11)	0.333(2)	0.488(2)	0.031(1)	74(6)
C(12)	0.292(2)	0.558(2)	0.000(1)	71(6)
C(13)	0.292(2)	0.638(2)	0.014(1)	73(6)
C(14)	0.330(2)	0.666(2)	0.065(1)	91(8)
C(15)	0.368(2)	0.601(2)	0.1011(9)	65(6)
C(16)	0.607(2)	0.536(2)	0.1400(9)	68(6)
C(17)	0.688(2)	0.580(2)	0.168(1)	82(7)
C(18)	0.703(2)	0.571(2)	0.223(1)	88(8)
C(19)	0.638(2)	0.523(2)	0.2505(9)	70(6)
C(20)	0.553(2)	0.482(1)	0.2241(9)	62(6)
C(21)	0.064(2)	0.451(1)	0.2431(9)	65(6)
C(22)	-0.027(2)	0.491(2)	0.258(1)	92(8)
C(23)	-0.072(2)	0.459(2)	0.306(1)	106(10)
C(24)	-0.032(2)	0.387(2)	0.331(1)	93(8)
C(25)	0.054(2)	0.349(2)	0.315(1)	74(6)
C(26)	0.311(2)	0.341(2)	0.343(1)	77(7)
C(27)	0.378(2)	0.308(2)	0.389(1)	84(7)
C(28)	0.413(2)	0.229(2)	0.392(1)	82(7)
C(29)	0.385(2)	0.167(2)	0.352(1)	88(8)
C(30)	0.322(2)	0.195(1)	0.3058(9)	64(6)
C(31)	0.044(2)	0.069(1)	0.1446(9)	63(6)
C(32)	-0.017(2)	0.001(1)	0.1214(9)	66(6)
C(33)	-0.062(2)	0.009(2)	0.070(1)	82(7)
C(34)	-0.054(2)	0.085(2)	0.043(1)	104(9)

Table 7.1.b) (contd.)

C(35)	0.012(2)	0.153(2)	0.066(1)	90(8)
C(36)	-0.033(2)	0.337(1)	0.1364(1)	62(6)
C(37)	-0.087(2)	0.414(2)	0.1214(9)	72(6)
C(38)	-0.042(2)	0.474(2)	0.088(1)	76(7)
C(39)	0.051(2)	0.460(2)	0.072(1)	74(6)
C(40)	0.110(2)	0.384(1)	0.0897(8)	56(5)
C(41)	0.536(2)	0.248(1)	0.2372(8)	58(5)
C(42)	0.607(2)	0.264(2)	0.2832(9)	70(6)
C(43)	0.705(2)	0.294(2)	0.2799(9)	72(6)
C(44)	0.741(2)	0.309(2)	0.229(1)	85(7)
C(45)	0.676(2)	0.297(1)	0.1830(9)	62(6)
C(46)	0.619(2)	0.122(2)	0.108(1)	99(9)
C(47)	0.671(3)	0.057(2)	0.078(2)	130(10)
C(48)	0.640(2)	0.055(2)	0.023(1)	103(9)
C(49)	0.569(3)	0.115(2)	0.001(1)	120(10)
C(50)	0.520(2)	0.177(2)	0.032(1)	94(8)

TABLE 7.2 Thermal parameters of atoms(Åx10^3). Anisotropic temperature factors used are of the form $\exp(-2\pi^2\sum_{i=1}^{3}\sum_{j=1}^{3}h_ih_ja_i^*a_j^*U_{ij})$

a) $[PtCl(\mu\text{-dppm})]_2$ (I)

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pt(1)	27.0(4)	43.3(5)	33.9(5)	3.3(4)	-1.8(3)	-1.6(4)
Pt(2)	28.0(4)	34.5(5)	40.1(5)	3.0(4)	-3.1(4)	-1.1(4)
Cl(1)	24(3)	127(7)	44(4)	2(4)	0(2)	-2(4)
Cl(2)	27(3)	87(6)	58(4)	4(3)	-7(3)	2(4)
P(1)	29(3)	45(4)	37(3)	2(3)	-2(2)	-1(3)
P(2)	33(3)	44(4)	34(3)	-1(3)	2(2)	-2(3)
P(3)	30(3)	51(4)	42(4)	1(3)	6(3)	-5(3)
P(4)	32(3)	43(4)	33(3)	2(3)	-7(2)	-5(3)

Table 7.2 (contd.)

b) $[Pt_2Cl(CO)(\mu\text{-dppm})_2](PF_6)$ (II)

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pt(1)	45.3(4)	40.5(4)	33.0(4)	-2.3(3)	-3.3(3)	-0.8(3)
Pt(2)	46.6(4)	45.7(4)	37.7(4)	-1.4(4)	-1.6(3)	-4.4(3)
Cl(1)	65(3)	47(3)	-72(4)	-2(2)	-2(3)	-19(3)
P(1)	51(3)	47(3)	36(3)	-3(2)	-3(2)	2(2)
P(2)	46(3)	45(3)	32(2)	-5(2)	-4(2)	-2(2)
P(3)	47(3)	45(3)	32(2)	-6(2)	-1(2)	-4(2)
P(4)	46(3)	50(3)	42(3)	2(2)	4(2)	6(2)
O	149(19)	58(12)	122(17)	3(12)	22(14)	-36(11)
C(51)	66(15)	90(19)	72(16)	-30(14)	-6(12)	33(15)
P(5)	78(4)	94(5)	46(3)	3(4)	-11(3)	-1(3)
F(1)	204(13)	384(46)	224(31)	-133(31)	121(26)	-166(33)
F(2)	149(19)	178(23)	277(31)	-52(17)	10(20)	-144(23)
F(3)	213(25)	96(14)	222(27)	5(16)	-66(21)	-4(16)
F(4)	142(22)	410(50)	207(30)	-27(26)	64(21)	-159(33)
F(5)	278(40)	264(38)	209(32)	-38(30)	-145(30)	76(29)
F(6)	217(26)	160(21)	212(26)	-5(18)	-88(21)	113(19)

TABLE 7.3 Bond lengths (Å)

a) [PtCl(μ-dppm)]$_2$ (I)

PT(1) - PT(2)	2.651(1)	C(10) - C(46)	1.342(38)
PT(1) - CL(1)	2.408(5)	C(10) - C(50)	1.404(35)
PT(1) - P(1)	2.294(7)	C(11) - C(12)	1.312(45)
PT(1) - P(2)	2.264(7)	C(12) - C(13)	1.361(44)
PT(2) - CL(2)	2.401(5)	C(13) - C(14)	1.460(47)
PT(2) - P(3)	2.259(7)	C(14) - C(15)	1.298(49)
PT(2) - P(4)	2.250(7)	C(16) - C(17)	1.381(44)
P(1) - C(1)	1.887(23)	C(17) - C(18)	1.439(45)
P(1) - C(3)	1.820(29)	C(18) - C(19)	1.355(46)
P(1) - C(4)	1.793(26)	C(19) - C(20)	1.396(41)
P(2) - C(2)	1.832(25)	C(21) - C(22)	1.469(42)
P(2) - C(5)	1.907(25)	C(22) - C(23)	1.458(59)
P(2) - C(6)	1.858(25)	C(23) - C(24)	1.325(69)
P(3) - C(2)	1.851(24)	C(24) - C(25)	1.389(56)
P(3) - C(7)	1.850(24)	C(26) - C(27)	1.501(58)
P(3) - C(8)	1.877(27)	C(27) - C(28)	1.249(55)
P(4) - C(1)	1.820(27)	C(28) - C(29)	1.391(42)
P(4) - C(9)	1.868(25)	C(29) - C(30)	1.474(44)
P(4) - C(10)	1.867(22)	C(31) - C(32)	1.454(59)
C(3) - C(11)	1.410(38)	C(32) - C(33)	1.227(48)
C(3) - C(15)	1.439(37)	C(33) - C(34)	1.334(51)
C(4) - C(16)	1.427(40)	C(34) - C(35)	1.450(44)
C(4) - C(20)	1.453(36)	C(36) - C(37)	1.455(53)
C(5) - C(21)	1.228(41)	C(37) - C(38)	1.320(53)
C(5) - C(25)	1.384(47)	C(38) - C(39)	1.344(48)
C(6) - C(26)	1.460(43)	C(39) - C(40)	1.457(40)
C(6) - C(30)	1.343(40)	C(41) - C(42)	1.419(47)
C(7) - C(31)	1.449(49)	C(42) - C(43)	1.359(55)
C(7) - C(35)	1.321(33)	C(43) - C(44)	1.356(46)
C(8) - C(36)	1.372(48)	C(44) - C(45)	1.460(42)
C(8) - C(40)	1.354(38)	C(46) - C(47)	1.497(38)
C(9) - C(41)	1.410(40)	C(47) - C(48)	1.405(46)
C(9) - C(45)	1.390(43)	C(48) - C(49)	1.388(45)
C(49) - C(50)	1.454(34)		

Table 7.3 (contd.)

b) $[Pt_2Cl(CO)(\mu\text{-dppm})_2](PF_6)$ (II)

PT(1) - PT(2)	2.620(1)	C(14) - C(15)	1.428(36)
PT(1) - CL(1)	2.384(5)	C(6) - C(26)	1.404(32)
PT(1) - P(1)	2.291(5)	C(6) - C(30)	1.385(30)
PT(1) - P(2)	2.298(5)	C(21) - C(22)	1.410(37)
PT(2) - P(3)	2.303(5)	C(21) - C(5)	1.413(30)
PT(2) - P(4)	2.308(5)	C(22) - C(23)	1.465(45)
PT(2) - C(51)	1.890(28)	C(23) - C(24)	1.355(43)
P(1) - C(1)	1.785(21)	C(24) - C(25)	1.351(39)
P(1) - C(4)	1.825(19)	C(25) - C(5)	1.406(33)
P(1) - C(3)	1.816(22)	C(26) - C(27)	1.484(35)
P(2) - C(2)	1.824(20)	C(27) - C(28)	1.312(39)
P(2) - C(6)	1.811(19)	C(28) - C(29)	1.416(39)
P(2) - C(5)	1.807(21)	C(29) - C(30)	1.432(34)
P(3) - C(2)	1.800(20)	C(8) - C(36)	1.392(29)
P(3) - C(8)	1.795(18)	C(8) - C(40)	1.382(28)
P(3) - C(7)	1.812(19)	C(36) - C(37)	1.419(33)
P(4) - C(1)	1.843(21)	C(37) - C(38)	1.420(36)
P(4) - C(10)	1.826(22)	C(38) - C(39)	1.311(36)
P(4) - C(9)	1.839(21)	C(39) - C(40)	1.450(32)
O - C(51)	1.153(34)	C(7) - C(31)	1.379(29)
P(5) - F(1)	1.433(35)	C(7) - C(35)	1.381(33)
P(5) - F(2)	1.485(28)	C(31) - C(32)	1.413(31)
P(5) - F(3)	1.567(24)	C(32) - C(33)	1.365(34)
P(5) - F(4)	1.469(31)	C(33) - C(34)	1.372(42)
P(5) - F(5)	1.497(36)	C(34) - C(35)	1.457(42)
P(5) - F(6)	1.546(27)	C(10) - C(46)	1.319(36)
C(4) - C(20)	1.422(28)	C(10) - C(50)	1.386(35)
C(4) - C(16)	1.381(31)	C(46) - C(47)	1.455(51)
C(20) - C(19)	1.393(31)	C(47) - C(48)	1.403(51)
C(19) - C(18)	1.358(38)	C(48) - C(49)	1.397(47)
C(18) - C(17)	1.372(39)	C(49) - C(50)	1.415(47)
C(17) - C(16)	1.397(34)	C(9) - C(45)	1.383(30)

Table 7.3.b) (contd.)

C(3) - C(11)	1.509(32)	C(9) - C(41)	1.432(31)
C(3) - C(15)	1.362(32)	C(45) - C(44)	1.382(33)
C(11) - C(12)	1.418(34)	C(44) - C(43)	1.423(37)
C(12) - C(13)	1.297(34)	C(43) - C(42)	1.362(34)
C(13) - C(14)	1.399(37)	C(42) - C(41)	1.433(31)

TABLE 7.4 Interbond angles (°)

a) [PtCl(μ-dppm)]$_2$ (I)

CL(1) - PT(1) - PT(2)	178.50(20)	P(1) - PT(1) - PT(2)	89.31(15)
P(2) - PT(1) - PT(2)	91.02(16)	CL(2) - PT(2) - PT(1)	175.72(16)
P(3) - PT(2) - PT(1)	88.67(16)	P(4) - PT(2) - PT(1)	85.00(16)
P(1) - PT(1) - CL(1)	90.20(22)	P(2) - PT(1) - CL(1)	89.58(22)
P(2) - PT(1) - P(1)	175.60(24)	C(1) - P(1) - PT(1)	114.44(81)
C(3) - P(1) - PT(1)	113.60(88)	C(4) - P(1) - PT(1)	116.71(81)
C(2) - P(2) - PT(1)	114.42(78)	C(5) - P(2) - PT(1)	113.37(82)
C(6) - P(2) - PT(1)	118.23(78)	P(3) - PT(2) - CL(2)	94.33(22)
P(4) - PT(2) - CL(2)	92.16(22)	P(4) - PT(2) - P(3)	172.86(22)
C(2) - P(3) - PT(2)	103.76(81)	C(7) - P(3) - PT(2)	118.22(78)
C(8) - P(3) - PT(2)	119.08(81)	C(1) - P(4) - PT(2)	108.74(84)
C(9) - P(4) - PT(2)	117.75(79)	C(10) - P(4) - PT(2)	119.35(81)
C(3) - P(1) - C(1)	107.47(118)	C(4) - P(1) - C(1)	99.34(111)
P(4) - C(1) - P(1)	104.45(121)	C(4) - P(1) - C(3)	103.77(121)
C(11) - C(3) - P(1)	124.53(213)	C(15) - C(3) - P(1)	117.39(206)
C(16) - C(4) - P(1)	122.25(192)	C(20) - C(4) - P(1)	120.83(183)
C(5) - P(2) - C(2)	104.24(113)	C(6) - P(2) - C(2)	99.92(112)
P(3) - C(2) - P(2)	107.15(125)	C(6) - P(2) - C(5)	104.89(110)
C(21) - C(5) - P(2)	120.13(208)	C(25) - C(5) - P(2)	117.20(232)
C(26) - C(6) - P(2)	112.10(220)	C(30) - C(6) - P(2)	123.50(203)
C(7) - P(3) - C(2)	102.88(110)	C(8) - P(3) - C(2)	109.25(111)
C(8) - P(3) - C(7)	102.42(113)	C(31) - C(7) - P(3)	120.36(215)
C(35) - C(7) - P(3)	117.39(194)	C(36) - C(8) - P(3)	117.45(222)
C(40) - C(8) - P(3)	118.16(191)	C(9) - P(4) - C(1)	104.24(115)
C(10) - P(4) - C(1)	103.67(111)	C(10) - P(4) - C(9)	101.30(112)
C(41) - C(9) - P(4)	119.04(200)	C(45) - C(9) - P(4)	118.66(199)
C(46) - C(10) - P(4)	119.56(191)	C(50) - C(10) - P(4)	113.99(185)
C(15) - C(3) - C(11)	117.99(261)	C(12) - C(11) - C(3)	121.41(275)
C(14) - C(15) - C(3)	117.85(282)	C(20) - C(4) - C(16)	116.44(224)
C(17) - C(16) - C(4)	120.40(258)	C(19) - C(20) - C(4)	120.85(238)
C(25) - C(5) - C(21)	122.66(285)	C(22) - C(21) - C(5)	119.95(264)
C(24) - C(25) - C(5)	121.32(357)	C(30) - C(6) - C(26)	124.37(274)

Table 7.4.a) (contd.)

C(27) - C(26) - C(6)	110.88(320)	C(29) - C(30) - C(6)	118.77(263)
C(35) - C(7) - C(31)	122.04(261)	C(32) - C(31) - C(7)	112.87(319)
C(34) - C(35) - C(7)	115.19(253)	C(40) - C(8) - C(36)	124.36(271)
C(37) - C(36) - C(8)	116.85(306)	C(39) - C(40) - C(8)	116.70(232)
C(45) - C(9) - C(41)	122.18(256)	C(42) - C(41) - C(9)	117.25(276)
C(44) - C(45) - C(9)	118.35(258)	C(50) - C(10) - C(46)	126.40(234)
C(47) - C(46) - C(10)	118.62(253)	C(49) - C(50) - C(10)	114.89(238)
C(13) - C(12) - C(11)	123.31(305)	C(14) - C(13) - C(12)	115.03(326)
C(15) - C(14) - C(13)	124.35(319)	C(18) - C(17) - C(16)	122.07(283)
C(19) - C(18) - C(17)	117.83(284)	C(20) - C(19) - C(18)	122.33(276)
C(23) - C(22) - C(21)	116.89(317)	C(24) - C(23) - C(22)	118.40(399)
C(25) - C(24) - C(23)	119.36(396)	C(28) - C(27) - C(26)	125.21(368)
C(29) - C(28) - C(27)	123.25(358)	C(30) - C(29) - C(28)	117.50(292)
C(33) - C(32) - C(31)	124.71(380)	C(34) - C(33) - C(32)	117.63(351)
C(35) - C(34) - C(33)	123.91(303)	C(38) - C(37) - C(36)	118.74(329)
C(39) - C(38) - C(37)	124.60(338)	C(40) - C(39) - C(38)	118.69(273)
C(43) - C(42) - C(41)	120.31(318)	C(44) - C(43) - C(42)	123.98(334)
C(45) - C(44) - C(43)	117.82(281)	C(48) - C(47) - C(46)	116.26(292)
C(49) - C(48) - C(47)	122.71(283)	C(50) - C(49) - C(48)	121.04(260)

Table 7.4 (contd.)

b) $[Pt_2Cl(CO)(\mu\text{-dppm})_2](PF_6)$ (II)

CL(1) - PT(1) - PT(2)	178.27(14)	P(1) - PT(1) - PT(2)	92.12(13)
P(2) - PT(1) - PT(2)	88.95(13)	P(3) - PT(2) - PT(1)	83.12(13)
P(4) - PT(2) - PT(1)	86.85(13)	C(51) - PT(2) - PT(1)	171.25(79)
P(1) - PT(1) - CL(1)	89.52(19)	P(2) - PT(1) - CL(1)	89.36(18)
P(2) - PT(1) - P(1)	174.83(18)	C(1) - P(1) - PT(1)	113.41(70)
C(4) - P(1) - PT(1)	115.53(63)	C(3) - P(1) - PT(1)	113.90(71)
C(2) - P(2) - PT(1)	113.13(64)	C(6) - P(2) - PT(1)	109.71(66)
C(5) - P(2) - PT(1)	120.07(68)	P(4) - PT(2) - P(3)	168.90(18)
C(51) - PT(2) - P(3)	93.41(75)	C(2) - P(3) - PT(2)	104.08(65)
C(8) - P(3) - PT(2)	116.80(62)	C(7) - P(3) - PT(2)	118.67(63)
C(51) - PT(2) - P(4)	97.18(76)	C(1) - P(4) - PT(2)	103.42(68)
C(10) - P(4) - PT(2)	115.62(69)	C(9) - P(4) - PT(2)	119.37(69)
O - C(51) - PT(2)	175.74(232)	C(4) - P(1) - C(1)	103.85(92)
C(3) - P(1) - C(1)	105.58(97)	P(4) - C(1) - P(1)	107.41(107)
C(3) - P(1) - C(4)	103.38(94)	C(20) - C(4) - P(1)	118.65(146)
C(16) - C(4) - P(1)	120.89(152)	C(11) - C(3) - P(1)	117.83(161)
C(15) - C(3) - P(1)	121.79(169)	C(6) - P(2) - C(2)	108.38(90)
C(5) - P(2) - C(2)	100.15(91)	P(3) - C(2) - P(2)	105.72(100)
C(5) - P(2) - C(6)	104.45(91)	C(26) - C(6) - P(2)	116.14(160)
C(30) - C(6) - P(2)	119.93(154)	C(21) - C(5) - P(2)	119.99(155)
C(25) - C(5) - P(2)	120.52(162)	C(8) - P(3) - C(2)	107.24(87)
C(7) - P(3) - C(2)	106.16(89)	C(7) - P(3) - C(8)	103.05(85)
C(36) - C(8) - P(3)	119.27(147)	C(40) - C(8) - P(3)	122.01(144)
C(31) - C(7) - P(3)	122.01(150)	C(35) - C(7) - P(3)	118.56(167)
C(10) - P(4) - C(1)	106.61(94)	C(9) - P(4) - C(1)	106.39(94)
C(9) - P(4) - C(10)	104.48(95)	C(46) - C(10) - P(4)	120.70(189)
C(50) - C(10) - P(4)	117.50(179)	C(45) - C(9) - P(4)	120.16(160)
C(41) - C(9) - P(4)	116.67(153)	F(2) - P(5) - F(1)	88.64(188)
F(3) - P(5) - F(1)	91.59(189)	F(4) - P(5) - F(1)	174.51(193)
F(5) - P(5) - F(1)	92.52(207)	F(6) - P(5) - F(1)	89.64(180)
F(3) - P(5) - F(2)	178.41(154)	F(4) - P(5) - F(2)	87.88(187)
F(5) - P(5) - F(2)	68.58(182)	F(6) - P(5) - F(2)	95.46(151)

Table 7.4.b) (contd.)

F(4) - P(5) - F(3)	91.78(189)	F(5) - P(5) - F(3)	89.83(175)
F(6) - P(5) - F(3)	86.12(138)	F(5) - P(5) - F(4)	83.16(203)
F(6) - P(5) - F(4)	94.91(174)	F(6) - P(5) - F(5)	175.47(176)
C(16) - C(4) - C(20)	120.23(185)	C(19) - C(20) - C(4)	118.46(195)
C(17) - C(16) - C(4)	119.41(211)	C(18) - C(19) - C(20)	120.18(224)
C(17) - C(18) - C(19)	121.95(252)	C(16) - C(17) - C(18)	119.60(243)
C(15) - C(3) - C(11)	120.32(201)	C(12) - C(11) - C(3)	112.41(205)
C(14) - C(15) - C(3)	122.08(221)	C(13) - C(12) - C(11)	125.98(232)
C(14) - C(13) - C(12)	122.54(244)	C(15) - C(14) - C(13)	116.55(245)
C(30) - C(6) - C(26)	123.66(197)	C(27) - C(26) - C(6)	113.86(216)
C(29) - C(30) - C(6)	119.70(209)	C(5) - C(21) - C(22)	119.93(212)
C(23) - C(22) - C(21)	117.64(251)	C(25) - C(5) - C(21)	118.89(195)
C(24) - C(23) - C(22)	120.03(283)	C(25) - C(24) - C(23)	121.35(278)
C(5) - C(25) - C(24)	121.81(234)	C(28) - C(27) - C(26)	122.85(248)
C(29) - C(28) - C(27)	122.36(257)	C(30) - C(29) - C(28)	117.29(242)
C(40) - C(8) - C(36)	118.64(179)	C(37) - C(36) - C(8)	120.00(198)
C(39) - C(40) - C(8)	120.43(189)	C(38) - C(37) - C(36)	119.55(213)
C(39) - C(38) - C(37)	120.35(235)	C(40) - C(39) - C(38)	120.64(224)
C(35) - C(7) - C(31)	119.38(200)	C(32) - C(31) - C(7)	121.48(197)
C(34) - C(35) - C(7)	118.52(247)	C(33) - C(32) - C(31)	119.93(217)
C(34) - C(33) - C(32)	119.82(252)	C(35) - C(34) - C(33)	120.38(271)
C(50) - C(10) - C(46)	121.67(238)	C(47) - C(46) - C(10)	124.38(292)
C(49) - C(50) - C(10)	116.24(267)	C(48) - C(47) - C(46)	114.63(321)
C(49) - C(48) - C(47)	119.88(315)	C(50) - C(49) - C(48)	123.03(317)
C(41) - C(9) - C(45)	123.07(193)	C(44) - C(45) - C(9)	119.22(210)
C(42) - C(41) - C(9)	114.92(190)	C(43) - C(44) - C(45)	120.38(235)
C(42) - C(43) - C(44)	119.54(226)	C(41) - C(42) - C(43)	122.79(216)

TABLE 7.5 Selected torsion angles (°)

a) $[PtCl(\mu\text{-dppm})]_2$ (I)

CL(1) - PT(1) - PT(2) - CL(2)	77.2(72)	CL(1) - PT(1) - PT(2) - P(3)	-148.2(68)
CL(1) - PT(1) - PT(2) - P(4)	28.5(68)	P(1) - PT(1) - PT(2) - CL(2)	6.3(23)
P(1) - PT(1) - PT(2) - P(3)	140.9(2)	P(1) - PT(1) - PT(2) - P(4)	-42.4(2)
P(2) - PT(1) - PT(2) - CL(2)	-169.3(23)	P(2) - PT(1) - PT(2) - P(3)	-34.7(2)
P(2) - PT(1) - PT(2) - P(4)	142.0(2)	PT(2) - PT(1) - P(1) - C(1)	25.4(9)
PT(2) - PT(1) - P(1) - C(3)	-98.5(10)	PT(2) - PT(1) - P(1) - C(4)	140.8(9)
CL(1) - PT(1) - P(1) - C(1)	-153.2(9)	CL(1) - PT(1) - P(1) - C(3)	82.9(10)
CL(1) - PT(1) - P(1) - C(4)	-37.8(9)	P(2) - PT(1) - P(1) - C(1)	119.7(20)
P(2) - PT(1) - P(1) - C(3)	-4.2(32)	P(2) - PT(1) - P(1) - C(4)	-124.9(20)
PT(2) - PT(1) - P(2) - C(2)	10.2(9)	PT(2) - PT(1) - P(2) - C(5)	129.6(9)
PT(2) - PT(1) - P(2) - C(6)	-107.0(9)	CL(1) - PT(1) - P(2) - C(2)	-171.1(9)
CL(1) - PT(1) - P(2) - C(5)	-51.8(9)	CL(1) - PT(1) - P(2) - C(6)	71.6(9)
P(1) - PT(1) - P(2) - C(2)	-84.0(31)	P(1) - PT(1) - P(2) - C(5)	35.4(32)
P(1) - PT(1) - P(2) - C(6)	158.7(28)	PT(1) - PT(2) - P(3) - C(2)	56.2(8)
PT(1) - PT(2) - P(3) - C(7)	169.3(9)	PT(1) - PT(2) - P(3) - C(8)	-65.5(9)
CL(2) - PT(2) - P(3) - C(2)	-126.9(8)	CL(2) - PT(2) - P(3) - C(7)	-13.8(9)
CL(2) - PT(2) - P(3) - C(8)	111.5(10)	P(4) - PT(2) - P(3) - C(2)	28.7(21)
P(4) - PT(2) - P(3) - C(7)	141.7(19)	P(4) - PT(2) - P(3) - C(8)	-93.0(21)
PT(1) - PT(2) - P(4) - C(1)	61.6(9)	PT(1) - PT(2) - P(4) - C(9)	-56.5(9)
PT(1) - PT(2) - P(4) - C(10)	-179.9(9)	CL(2) - PT(2) - P(4) - C(1)	-115.1(9)
CL(2) - PT(2) - P(4) - C(9)	126.7(9)	CL(2) - PT(2) - P(4) - C(10)	3.3(9)
P(3) - PT(2) - P(4) - C(1)	89.3(20)	P(3) - PT(2) - P(4) - C(9)	-28.9(22)
P(3) - PT(2) - P(4) - C(10)	-152.3(19)	PT(1) - P(1) - C(1) - P(4)	10.5(14)
C(3) - P(1) - C(1) - P(4)	137.7(13)	C(4) - P(1) - C(1) - P(4)	-114.6(13)
PT(1) - P(1) - C(3) - C(11)	132.6(23)	PT(1) - P(1) - C(3) - C(15)	-44.1(24)
C(1) - P(1) - C(3) - C(11)	4.9(28)	C(1) - P(1) - C(3) - C(15)	-171.8(21)
C(4) - P(1) - C(3) - C(11)	-99.7(25)	C(4) - P(1) - C(3) - C(15)	83.6(23)
PT(1) - P(1) - C(4) - C(16)	157.5(16)	PT(1) - P(1) - C(4) - C(20)	-30.8(23)
C(1) - P(1) - C(4) - C(16)	-79.0(23)	C(1) - P(1) - C(4) - C(20)	92.7(21)
C(3) - P(1) - C(4) - C(16)	31.7(24)	C(3) - P(1) - C(4) - C(20)	-156.6(20)
PT(1) - P(2) - C(2) - P(3)	26.8(14)	C(5) - P(2) - C(2) - P(3)	-97.5(13)
C(6) - P(2) - C(2) - P(3)	154.2(12)	PT(1) - P(2) - C(5) - C(21)	-9.2(27)

Table 7.5.a) (contd.)

PT(1) - P(2) - C(5) - C(25)	171.7(22)
C(2) - P(2) - C(5) - C(25)	-63.3(26)
C(6) - P(2) - C(5) - C(25)	41.3(26)
PT(1) - P(2) - C(6) - C(30)	112.2(22)
C(2) - P(2) - C(6) - C(30)	-12.6(25)
C(5) - P(2) - C(6) - C(30)	-120.3(24)
C(7) - P(3) - C(2) - P(2)	178.5(12)
PT(2) - P(3) - C(7) - C(31)	-104.8(23)
C(2) - P(3) - C(7) - C(31)	8.8(26)
C(8) - P(3) - C(7) - C(31)	122.1(24)
PT(2) - P(3) - C(8) - C(36)	-163.8(21)
C(2) - P(3) - C(8) - C(36)	77.4(25)
C(7) - P(3) - C(8) - C(36)	-31.2(26)
PT(2) - P(4) - C(1) - P(1)	-51.8(12)
C(10) - P(4) - C(1) - P(1)	-179.7(11)
PT(2) - P(4) - C(9) - C(45)	170.3(19)
C(1) - P(4) - C(9) - C(45)	49.8(24)
C(10) - P(4) - C(9) - C(45)	-57.6(24)
PT(2) - P(4) - C(10) - C(50)	-84.0(19)
C(1) - P(4) - C(10) - C(50)	37.1(21)
C(9) - P(4) - C(10) - C(50)	145.0(19)
C(2) - P(2) - C(5) - C(21)	115.9(25)
C(6) - P(2) - C(5) - C(21)	-139.6(24)
PT(1) - P(2) - C(6) - C(26)	-69.9(23)
C(2) - P(2) - C(6) - C(26)	165.3(22)
C(5) - P(2) - C(6) - C(26)	57.6(24)
PT(2) - P(3) - C(2) - P(2)	-57.8(12)
C(8) - P(3) - C(2) - P(2)	70.2(15)
PT(2) - P(3) - C(7) - C(35)	70.0(22)
C(2) - P(3) - C(7) - C(35)	-176.5(21)
C(8) - P(3) - C(7) - C(35)	-63.1(23)
PT(2) - P(3) - C(8) - C(40)	14.2(24)
C(2) - P(3) - C(8) - C(40)	-104.7(21)
C(7) - P(3) - C(8) - C(40)	146.7(21)
C(9) - P(4) - C(1) - P(1)	74.6(14)
PT(2) - P(4) - C(9) - C(41)	-5.8(25)
C(1) - P(4) - C(9) - C(41)	-126.3(22)
C(10) - P(4) - C(9) - C(41)	126.3(22)
PT(2) - P(4) - C(10) - C(46)	98.5(21)
C(1) - P(4) - C(10) - C(46)	-140.5(22)
C(9) - P(4) - C(10) - C(46)	-32.6(24)

Table 7.5 (contd.)

b) $[Pt_2Cl(CO)(\mu\text{-dppm})_2](PF_6)$ (II)

CL(1) -	PT(1) -	PT(2) -	P(3)	58.0(46)	CL(1) -	PT(1) -	PT(2) -	P(4)	-126.8(46)
P(1) -	PT(1) -	PT(2) -	P(3)	-140.3(2)	P(1) -	PT(1) -	PT(2) -	P(4)	34.9(2)
P(2) -	PT(1) -	PT(2) -	P(3)	44.8(2)	P(2) -	PT(1) -	PT(2) -	P(4)	-140.0(2)
PT(2) -	PT(1) -	P(1) -	C(1)	-10.5(8)	PT(2) -	PT(1) -	P(1) -	C(4)	-130.3(7)
PT(2) -	PT(1) -	P(1) -	C(3)	110.2(8)	CL(1) -	PT(1) -	P(1) -	C(1)	168.9(8)
CL(1) -	PT(1) -	P(1) -	C(4)	49.2(7)	CL(1) -	PT(1) -	P(1) -	C(3)	-70.3(8)
P(2) -	PT(1) -	P(1) -	C(1)	91.4(21)	P(2) -	PT(1) -	P(1) -	C(4)	-28.3(22)
P(2) -	PT(1) -	P(1) -	C(3)	-147.8(20)	PT(2) -	PT(1) -	P(2) -	C(2)	-25.7(7)
PT(2) -	PT(1) -	P(2) -	C(6)	95.4(7)	PT(2) -	PT(1) -	P(2) -	C(5)	-143.7(8)
CL(1) -	PT(1) -	P(2) -	C(2)	154.7(7)	CL(1) -	PT(1) -	P(2) -	C(6)	-84.2(7)
CL(1) -	PT(1) -	P(2) -	C(5)	36.7(8)	P(1) -	PT(1) -	P(2) -	C(2)	-127.8(20)
P(1) -	PT(1) -	P(2) -	C(6)	-6.7(22)	P(1) -	PT(1) -	P(2) -	C(5)	114.2(21)
PT(1) -	PT(2) -	P(3) -	C(2)	-65.2(7)	PT(1) -	PT(2) -	P(3) -	C(8)	52.7(7)
PT(1) -	PT(2) -	P(3) -	C(7)	177.1(7)	P(4) -	PT(2) -	P(3) -	C(2)	-90.8(11)
P(4) -	PT(2) -	P(3) -	C(8)	27.1(13)	P(4) -	PT(2) -	P(3) -	C(7)	151.5(10)
PT(1) -	PT(2) -	P(4) -	C(1)	-57.1(7)	PT(1) -	PT(2) -	P(4) -	C(10)	-173.2(8)
PT(1) -	PT(2) -	P(4) -	C(9)	60.7(8)	P(3) -	PT(2) -	P(4) -	C(1)	-31.7(12)
P(3) -	PT(2) -	P(4) -	C(10)	-147.8(11)	P(3) -	PT(2) -	P(4) -	C(9)	86.2(12)
PT(1) -	P(1) -	C(1) -	P(4)	-28.1(12)	C(4) -	P(1) -	C(1) -	P(4)	98.1(11)
C(3) -	P(1) -	C(1) -	P(4)	-153.5(10)	PT(1) -	P(1) -	C(4) -	C(20)	9.1(18)
PT(1) -	P(1) -	C(4) -	C(16)	-172.7(15)	C(1) -	P(1) -	C(4) -	C(20)	-115.7(17)
C(1) -	P(1) -	C(4) -	C(16)	62.5(19)	C(3) -	P(1) -	C(4) -	C(20)	134.2(16)
C(3) -	P(1) -	C(4) -	C(16)	-47.6(19)	PT(1) -	P(1) -	C(3) -	C(11)	-94.0(17)
PT(1) -	P(1) -	C(3) -	C(15)	88.8(19)	C(1) -	P(1) -	C(3) -	C(11)	31.1(19)
C(1) -	P(1) -	C(3) -	C(15)	-146.1(19)	C(4) -	P(1) -	C(3) -	C(11)	139.8(17)
C(4) -	P(1) -	C(3) -	C(15)	-37.3(21)	PT(1) -	P(2) -	C(2) -	P(3)	-14.9(11)
C(6) -	P(2) -	C(2) -	P(3)	-136.8(10)	C(5) -	P(2) -	C(2) -	P(3)	114.2(10)
PT(1) -	P(2) -	C(6) -	C(26)	83.0(17)	PT(1) -	P(2) -	C(6) -	C(30)	-91.2(17)
C(2) -	P(2) -	C(6) -	C(26)	-153.0(17)	C(2) -	P(2) -	C(6) -	C(30)	32.7(19)
C(5) -	P(2) -	C(6) -	C(26)	-46.9(19)	C(5) -	P(2) -	C(6) -	C(30)	138.8(17)
PT(1) -	P(2) -	C(5) -	C(21)	22.2(20)	PT(1) -	P(2) -	C(5) -	C(25)	-166.7(15)
C(2) -	P(2) -	C(5) -	C(21)	-102.2(18)	C(2) -	P(2) -	C(5) -	C(25)	68.9(19)

Table 7.5.b) (contd.)

C(6) -	P(2) -	C(5) -	C(21)	145.7(17)
PT(2) -	P(3) -	C(2) -	P(2)	56.8(9)
C(7) -	P(3) -	C(2) -	P(2)	-177.2(9)
PT(2) -	P(3) -	C(8) -	C(40)	15.0(10)
C(2) -	P(3) -	C(8) -	C(40)	131.2(10)
C(7) -	P(3) -	C(8) -	C(40)	-117.0(17)
PT(2) -	P(3) -	C(7) -	C(35)	-90.9(10)
C(2) -	P(3) -	C(7) -	C(35)	152.6(10)
C(8) -	P(3) -	C(7) -	C(35)	40.0(20)
C(10) -	P(4) -	C(1) -	P(1)	-178.0(10)
PT(2) -	P(4) -	C(10) -	C(40)	-108.3(21)
C(1) -	P(4) -	C(10) -	C(40)	137.4(21)
C(9) -	P(4) -	C(10) -	C(40)	25.0(23)
PT(2) -	P(4) -	C(9) -	C(45)	-108.8(15)
C(1) -	P(4) -	C(9) -	C(45)	-52.4(20)
C(10) -	P(4) -	C(9) -	C(45)	60.1(19)

C(6) -	P(2) -	C(5) -	C(25)	-43.2(20)
C(8) -	P(3) -	C(2) -	P(2)	-67.0(11)
PT(2) -	P(3) -	C(8) -	C(36)	-168.5(14)
C(2) -	P(3) -	C(8) -	C(36)	-52.2(18)
C(7) -	P(3) -	C(8) -	C(36)	59.5(17)
PT(2) -	P(3) -	C(7) -	C(31)	86.4(17)
C(2) -	P(3) -	C(7) -	C(31)	-30.2(19)
C(8) -	P(3) -	C(7) -	C(31)	-142.0(17)
PT(2) -	P(4) -	C(1) -	P(1)	59.0(10)
C(9) -	P(4) -	C(1) -	P(1)	-60.9(10)
PT(2) -	P(4) -	C(10) -	C(50)	67.0(20)
C(1) -	P(4) -	C(10) -	C(50)	-46.7(21)
C(9) -	P(4) -	C(10) -	C(50)	-159.1(10)
PT(2) -	P(4) -	C(9) -	C(41)	7.9(10)
C(1) -	P(4) -	C(9) -	C(41)	124.2(16)
C(10) -	P(4) -	C(9) -	C(41)	-123.2(17)

TABLE 7.6 Deviations of atoms (Å x 10^3) from, and equations of, their weighted mean planes

a) $[PtCl(\mu\text{-dppm})]_2$ (I)

(i) Pt(1) -2, Pt(2) -1, Cl(1) -61, P(1) 86, P(2) 84.

$-0.189X - 0.969Y + 0.161Z = -0.710^+$

(ii) Pt(1) 2, Pt(2) -1, Cl(2) 127, P(3) -66, P(4) -66.

$0.158X - 0.904Y - 0.397Z = -2.480^+$

b) $[Pt_2Cl(CO)(\mu\text{-dppm})_2](PF_6)$ (II)

(i) Pt(1) -4, Pt(2) 0, Cl(1) 11, P(1) 102, P(2) 92.

$0.735X - 0.289Y + 0.613Z = 3.823^+$

(ii) Pt(1) 0, Pt(2) -3, C(51) -265, P(3) 87, P(4) 99.

$-0.126X + 0.468Y - 0.875Z = -1.791^+$

$^+$ X,Y,Z refer to an orthogonal basis set defined by a*, b and c.

TABLE 7.7 Selected intramolecular non-bonding distances (Å) less than the sum of the appropriate van der Waals radii

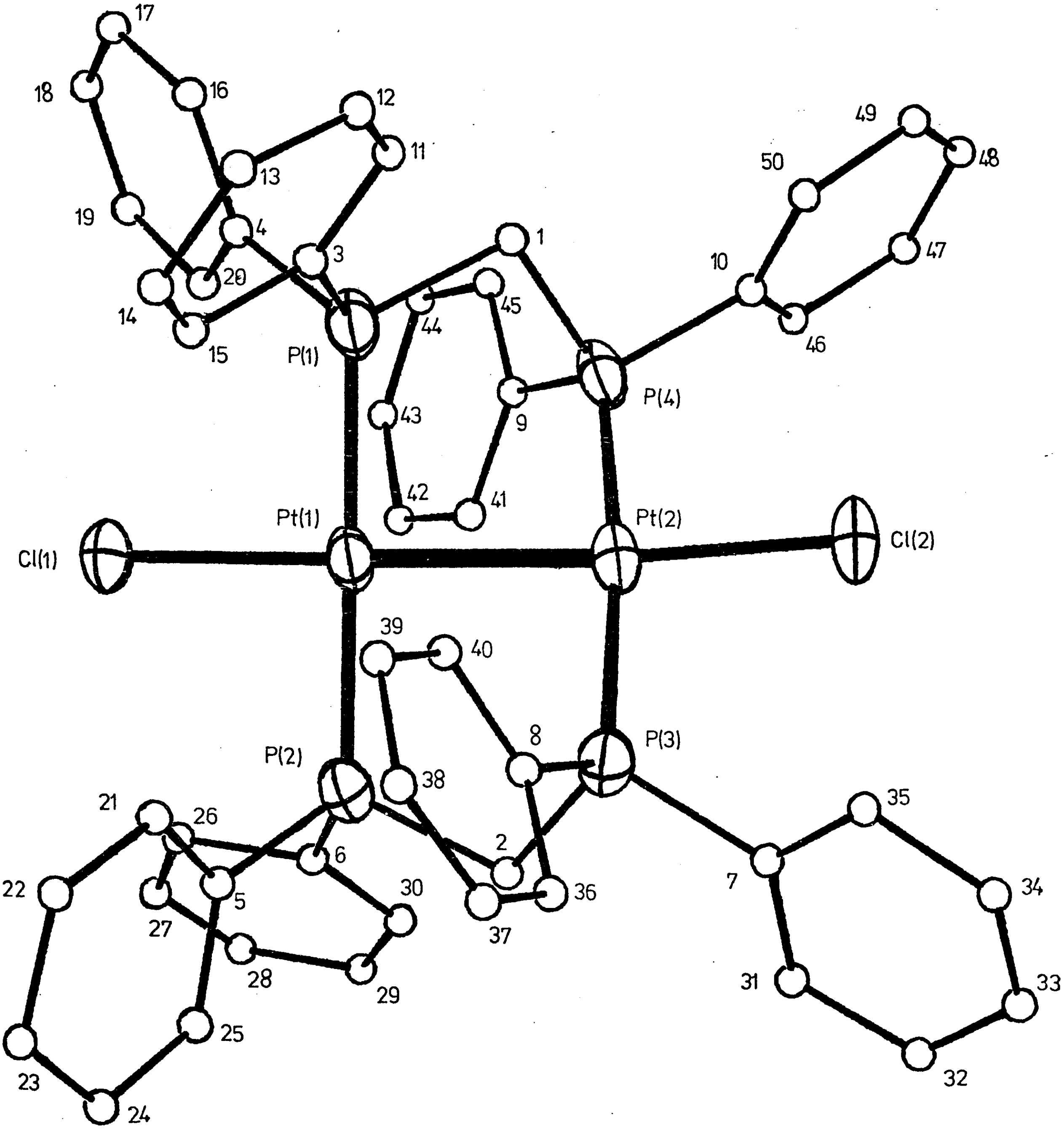
Compound	$[PtCl(\mu\text{-dppm})]_2$ (I)	$[Pt_2Cl(CO)(\mu\text{-dppm})_2](PF_6)$ (II)
Cl(1)...C(4)	3.45	3.55
Cl(1)...C(20)	3.37	3.28
Cl(1)...C(21)	3.32	3.28
Cl(2)...C(10)	3.35	-
Cl(2)...C(7)	3.43	-
Cl(2)...C(35)	3.29	-
C(51)...C(10)	-	3.30
C(51)...C(7)	-	3.40
Pt(1)...C(1)	3.52	3.43
Pt(1)...C(2)	3.45	3.43
Pt(2)...C(1)	3.32	3.29
Pt(2)...C(2)	3.24	3.23
C(1) ...C(11)	3.25	3.12
C(3) ...C(16)	3.14	3.20
C(1) ...C(50)	3.06	3.24
C(2) ...C(30)	3.01	3.24
C(2) ...C(31)	3.05	3.17

Legends

FIGURE 7.1 A perspective view of the molecular structure of $[PtCl(\mu\text{-dppm})]_2$. The vibrational ellipsoids of the Pt, Cl and P atoms display 50% probability. For clarity, all carbon atoms are represented by circles of arbitrary size; they are labelled by numbers only, corresponding to those listed in Table 7.1 a.

FIGURE 7.2 A perspective view of the cation $[Pt_2Cl(CO)(\mu\text{-dppm})_2]^+$. The vibrational ellipsoids of Pt, Cl, P, O and C(51) atoms display 50% probability. For clarity, all other atoms are represented by circles of arbitrary size and labelled by numbers only, corresponding to those listed in Table 7.1 b.

FIGURE 7.1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Pt(1)
Pt(2)
Cl(1)
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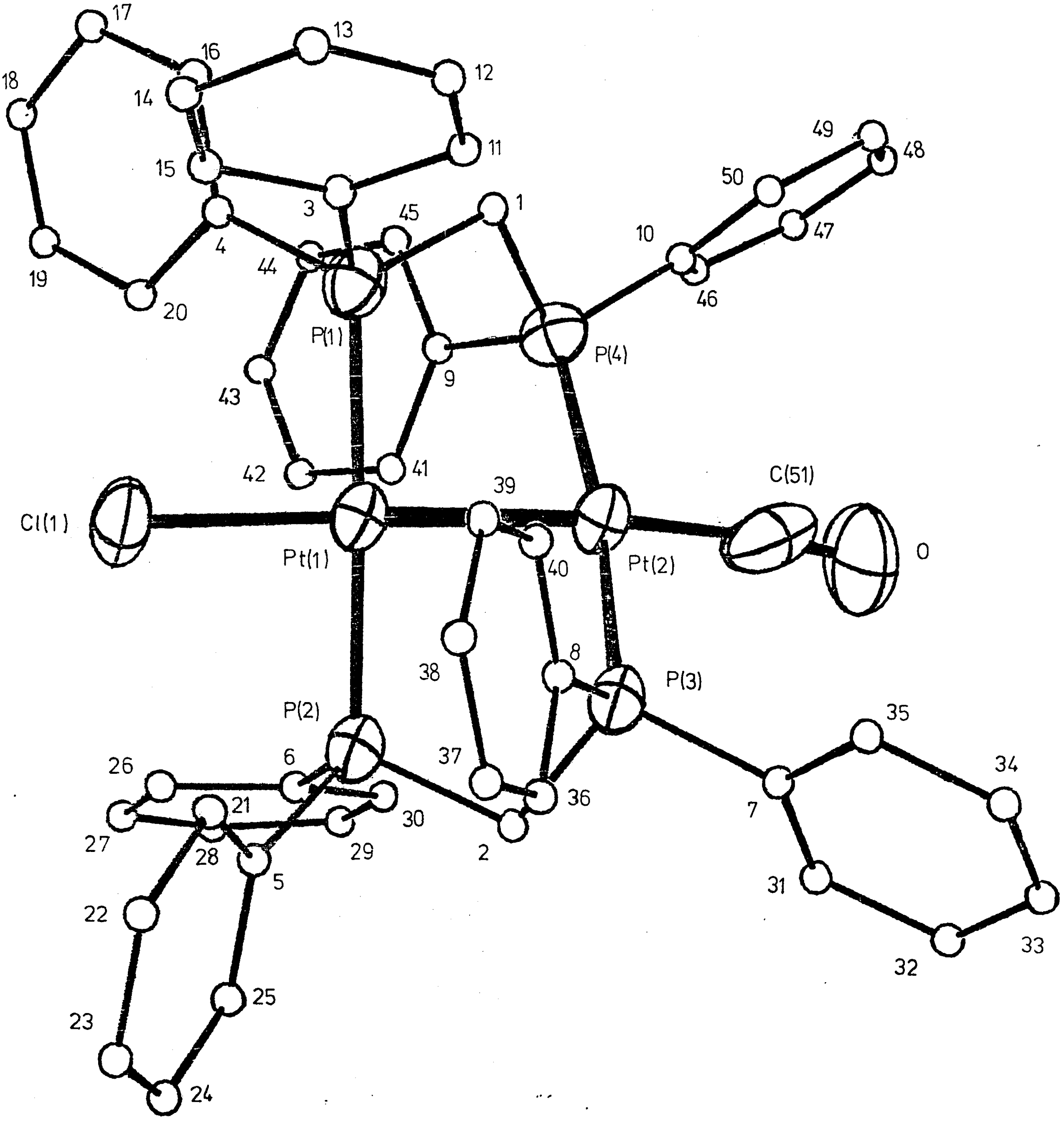
FIGURE 7.2



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P(4)
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C(51)
Cl(1)
Pt(1)
40
Pt(2)
O
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P(3)
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7.3 Results and Discussion

The crystal structure of I is built of discrete binuclear molecules, and that of II of discrete cations and anions, separated by normal van der Waals contacts. Thus, in I the shortest distance between non-hydrogen atoms in different molecules is $C(12)\ldots C(48)^{i} = 3.46Å$, and in II the shortest distance between non-hydrogen atoms in different cations and anions is $C(1)\ldots C(30)^{ii} = 3.44Å$; the superscripts i and ii represent the $\bar{x}\bar{y}\bar{z}$ and $\frac{1}{2}-x,\frac{1}{2}+y,\frac{1}{2}-z$ transformations of the co-ordinates listed in Table 7.1.

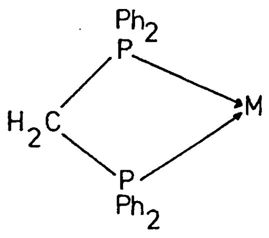
The molecular structure of I, shown in the Figure 7.1, comprises two Pt-Cl fragments linked directly through a Pt-Pt bond and bridged by two dppm ligands. The structure of the cationic complex II, shown in the Figure 7.2, is closely similar and can be considered as derived from that of I by substituting one chloride ligand by a carbonyl group. Hence, the results of this work prove the structures (B) and (C), deduced from spectroscopic data, to be correct for I and II, respectively.

The octahedral hexafluorophosphate anion in II displays no unexpected structural features (Tables 7.3.a and 7.4.a). The mean value of P-F bond lengths is 1.50(8)Å, and the F-P-F angles are in the ranges $83(2)-95(2)^{\circ}$ and $175(2)-178(2)^{\circ}$.

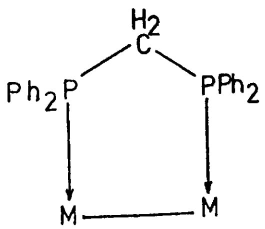
The non-bonding intramolecular distances in I and II, shorter than the sum of the appropriate van der Waals radii are presented in the Table 7.7. They do not reveal the presence of any unusually strong repulsive intramolecular interactions.

Geometry of the dppm ligands

Bis(diphenylphosphino)methane, dppm, can act as a unidentate[192] or bidentate ligand in metal complexes.[193] Flexibility of this ligand, arising from its ability to rotate internally about each of the two methylene carbon--phosphorus bonds, allows it to function as a bidentate ligand in two different ways.[194] The ligand can make two P-M bonds involving one metal centre only (D), and thus form a four-membered metallocyclic ring, such as, for example, in $PtPh_2(dppm)$.[193] It can also act as a bridging ligand,forming P-M bonds with two metal atoms (E). In the latter mode of co-ordination the conformations of the two PPh_2 groups about CH_2-P bonds are such as to make the phosphorus donor orbitals nearly pallarel to each other; the bite of the ligand is such as to make it suitable for bridging metal atoms which are about 2.5 to 3.0 Å apart. Since the transition metals in low oxidation states display a tendency to form metal-to-metal bonds this mode of dppm co-ordination usually results in formation of the five-membered metallocyclic rings.



(D)



(E)

In the complexes I and II the dppm ligands adopt a bridging mode of co-ordination. Their preference for (E), rather than (D), mode is likely to arise from the steric strain, which is smaller in a five-membered than in a four-membered ring.

In each complex the two dppm ligands display similar conformations and the molecular structures of I and II therefore approximate to C_2 symmetry, the two-fold axis coinciding with essentially linear Cl-Pt-Pt-Cl or Cl-Pt-Pt-CO unit. Furthermore, the conformations of the dppm ligands are essentially the same in both complexes as is evident from a comparison of the relevant torsion angles listed in Table 7.5. Thus conformations about C(1)-P(4) and C(2)-P(3) bonds are practically staggered [the P-C-P-Pt(2) torsion angles being -52(1), -58(1)° for I and 60(1), 57(1)° for II*], while those about the C(1)- P(1) and C(2)-P(2) bonds are closer to eclipsed [the P-C-P-Pt(1) torsion angles being 11(1), 27(1)° for I and -28(1), -15(1)° for II]. The P(1)-C(1) and P(2)-C(2) bonds are nearer to eclipsing the Pt(1)-Pt(2) vector than are the P(3)-C(2) and P(4)-C(1) bonds, as is apparent from the respective Pt-Pt-P-C torsion angles of 25.4(9), 10.2(9), 56.2(8) and 61.6(9)° for I and -10.5(8), -25.7(7), -65.2(7) and -57.1(7)° for II.

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* The opposite signs of the corresponding torsion angles in the two complexes arise from different enantiomers, chosen from the pairs of centrosymmetrically related molecules, to represent the asymmetric units in I and II.

The bond lengths and angles within the dppm ligands are unexceptional. The mean values of P-C and C-C bonds are 1.85(1) and 1.39(1)Å for I and 1.82(1) and 1.40(1)Å for II. In both compounds P-CH_2-P angles are close to the tetrahedral value [104(1) and 107(1)° for I and 107(1) and 106(1)° for II].

Co-ordination of the metal atoms

In both compounds the co-ordination around the platinum atoms is square-planar with small tetrahedral distortions. These distortions are evident from the valency angles subtended at the metal centres (Table 7.4) and from the displacement of atoms from their mean planes (Table 7.6). On average, the distortions are slightly higher in II than in I and this feature of the molecular geometry may reflect different properties of the carbonyl and chloride ligands.

Perhaps the most interesting structural feature is the twisted configuration of the molecules as a whole. In each compounds the two metal co-ordination planes are mutually rotated about the Pt-Pt bond, to afford a dihedral angle of 38.6° in I and 40.1° in II. This, however, leads to only slight angular distortions in the Pt_2P_2C rings (see above). Thus the flexibility of the dppm ligands allows minimisation of antibonding interactions between filled interaxial d orbitals of the metal atoms. In the platinum(I) anion $[PtCl_2(CO)]_2^{2-}$, where the constraint of the bridging groups is not present, the corresponding dihedral angle is ca. 60°;[187] this has been interpreted as a compromise between the tendency of the filled interaxial metal d orbitals to be positioned as far from one another as possible (dihedral angle 45°) and the tendency of the ligands attached to the adjacent metal atoms to minimise their steric repulsions (dihedral angle 90°).[187]

The Pt-Pt bond in I [2.651(1)Å] is somewhat shorter than the Pd-Pd bond [2.699(5)Å] in the isomorphous and isostructural palladium analogue, $[PdBr(\mu\text{-}dppm)]_2$.[195] In II the Pt-Pt bond is 2.620(1)Å. The metal-metal bonding is discussed below.

The Pt-Cl bond lengths in I [2.401(5) and 2.408(5)Å] and II [2.383(5)Å] are similar to the corresponding ones in the $[PtCl_2(CO)]_2^{2-}$ anion [2.382(10) and 2.426(9)Å].[187] They are close to the upper end of the range of Pt-Cl distances observed in square-planar Pt(II) complexes,[48] and may suggest a relatively high trans-influence of the Pt-Pt bond.

The Pt-P distances in I [2.250(7) - 2.294(4)Å] and II [2.291(5) - 2.308(5)Å] are within the range of Pd-P distances [2.26(1) - 2.32(1)Å] in $[PdBr(\mu\text{-}dppm)]_2$.

Metal-metal bonding in platinum(I) complexes

Although complexes containing metal-to-metal bonding have been intensively studied in recent years, theoretical treatment of their structural properties is still at an early stage of its development.[196] It has, however, been suggested that the strength and length of the metal-metal bonds may depend on the formal oxidation state of the metal atoms and also on electron-donating, or electron-withdrawing, properties of the ligands bonded to the metal.

The metal-metal distances in platinum(I) complexes examined crystallographically, including I and II, are listed in Table 7.8. They display a range of values, 2.58 - 2.65Å, and are usually shorter than Pt(0)-Pt(0) distances in binuclear complexes and clusters [2.65 - 2.79Å].[197] The range of the observed Pt(I) - Pt(I) distances is likely to reflect different electronic and steric properties of the variety of ligands represented in Table 7.8. The only complexes in this Table which are closely similar to one another are I and II.

The square-planar co-ordination at the metal centres in I and II suggests that dsp^2 hybrid orbitals are used to form platinum-ligand bonds. These hybrid orbitals are not entirely empty, as in platinum(II) d^8 complexes, since one of them contains an electron. Coupling of the unpaired electrons of the platinum atoms results in formation of a covalent metal-metal bond. In II the Pt-Pt distance [2.620(1)Å] is shorter than in I [2.651(1)Å], and this is compatible with Raman ν(Pt-Pt) frequencies of 157 cm^{-1} in II and 150 cm^{-1} in I.[181] Contraction of the Pt(I) - Pt(I) bond by _ca_. 0.03Å, which occurs on substitution of one Cl-ligand in I by the strongly π-acidic CO group, can be attributed to some depopulation of the filled antibonding d orbitals of the metal atoms. Such an

TABLE 7.8 Pt-Pt bond lengths in platinum(I) complexes

Compound	Pt-Pt(Å)	Bridging group	Ref.
$[Pt_2(\eta^5-C_5H_5)_2(\eta^4-C_{10}H_{10})]$	2.581(4)	$\eta^4-C_{10}H_{10}$	182
$[PtCl_2(CO)]_2^{2-}$	2.584(2)	-----	187
$[Pt_2\{(CF_3)_2CO\}(1,5-C_8H_{12})_2]$	2.585(1)	$(CF_3)_2CO$	183
$[Pt_2(PPh_3)_2(PPh_2)_2]$	2.604(1)	$2\times PPh_2$	184
$[Pt_2Cl(CO)(\mu-dppm)_2]^+$	2.620(1)	2×dppm	This work
$[Pt\{SP(Et)_2\}P(OPh)_3]_2$	2.628(1)	$2\times SP(Et)_2$	185
$[Pt_2(CO)(PPh_3)_3S]$	2.647(2)	S	186
$[PtCl(\mu-dppm)]_2$	2.651(1)	2×dppm	This work

electronic effect may lower the repulsive interactions between antibonding orbitals of the platinum atoms and thus contribute to further stabilization of the Pt-Pt bond. A similar mechanism is likely to operate in binuclear platinum(0) complexes, as shown recently by Extended Hückel Molecular Orbital calculations on complexes Pt_2L_4, where L = PH_3,CO.[197]

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cis- AND trans-INFLUENCE OF LIGANDS IN PLATINUM(II) COMPLEXES. THE CRYSTAL AND MOLECULAR STRUCTURE OF cis-[PtCl$_2$(PEt$_3$)(CO)]

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Summary

In the complexes cis-[PtCl$_2$(PEt$_3$)L], where L = Cl$^-$, C(NPhCH$_2$)$_2$, C(OEt)NHPh, CNPh, CO, PEt$_3$, P(OPh)$_3$ or PF$_3$, ligands L exert cis-influence on the Pt-P bond lengths (ca. 0.06Å), which is almost as large as their trans-influence on the Pt-Cl(trans to L) bond lengths (ca. 0.07Å). The two effects are independent of each other and lead to different cis- and trans-influence series of L. The trend in Pt-Cl(cis to L) distances, displaying a variation of about 0.03Å, reflects the change in the length, and presumably strength, of the Pt-P bonds.

The X-ray analysis of cis-[PtCl$_2$(PEt$_3$)(CO)] was based on diffractometric intensities of 1820 independent reflections. The crystal structure was solved by the heavy atom method and refined by full-matrix least-squares to R = 0.037. The crystals are orthorhombic, space group Pca2$_1$, a = 12.777, b = 8.587, c = 11.424Å, Z = 4. They are built of discrete monomeric molecules with cis-square planar geometry. Selected bond lengths are: Pt-C 1.855(14), Pt-P 2.265(3), Pt-Cl(trans to C) 2.296(4) and Pt-Cl(trans to P) 2.368(3)Å.

Introduction

In square planar transition metal complexes the effects of ligands on the strength of cis-metal-ligand bonds are of interest, not only intrinsically, but also because they are germane to the much studied phenomenon of trans-influence. The existance of cis-influence in platinum(II) complexes has been inferred from spectroscopic results [1].

We, however, first noted in 1974 that the bond lengths in cis-[$PtCl_2(PPh_3)(CO)$], compared with those in other cis-[$PtCl_2(PR_3)L$] complexes (PR_3 = PMe_3, PEt_3 or PEt_2Ph and L = PMe_3, carbenoid or isocyanide), indicate that the carbonyl group weakens the Pt-P and strengthens the Pt-Cl bonds cis to itself; this was in conformity with trends displayed by ^1J(Pt-P) coupling constants and ν(Pt-Cl) stretching frequencies in analagous complexes [2]. While we considered it likely that these observations reflect a cis-influence of the carbonyl group, we pointed out that since the complexes compared contain different PR_3 ligands the effects of the phosphine substituents may also be involved [2-4]. Variations in the Pt-P bond lengths in the complexes cis-[$PtCl_2(PR_3)L$] have also been noticed by Russell et al. [5].

To investigate variations in the lengths of platinum-ligand bonds cis and trans to L, originating from change in the nature of L only, we have chosen to examine a series of triethylphosphine complexes cis-[$PtCl_2(PEt_3)L$]. Crystallographic studies of such complexes with L = Cl^-, C(OEt)NHPh, $C(NPhCH_2)_2$, CNPh, PEt_3, PF_3 or $P(OPh)_3$ have already been carried out in this laboratory and elsewhere [6-12], and we report here the results of an accurate X- ray analysis of the compound with L = CO. The crystal structure of this compound was first determined by E.M. Badley, using photographic diffraction data, but the results obtained are of low accuracy [9].

Experimental

Crystals of cis-$[PtCl_2(PEt_3)(CO)]$ are air-stable transparent needles elongated along **a**.

Crystal data

$C_7H_{15}Cl_2OPPt$, M.W. = 412.2. Orthorhombic, space group Pca2_1, a = 12.777, b = 8.587, c = 11.424Å, U = 1253.4Å^3, Z = 4, D_c = 2.184gcm^{-3}, $F(000)$ = 768. Mo-K_α radiation, λ = 0.71069Å, μ(Mo-K_α) = 118.3 cm^{-1}.

Measurements

A crystal of approximate dimensions 0.50 x 0.21 x 0.24 mm was selected for the analysis and its principal faces, belonging to the forms {100}, {010} and {001}, were indentified by optical goniometry and X-ray measurements.

The crystal symmetry and preliminary unit cell dimensions were determined from oscillation and Weissenberg photographs. Systematically absent reflections are consistent with space groups Pca2_1 (No. 29) and Pcam, the latter being an unconventional setting of the space group Pbcm (No. 57). The non-centrosymmetric space group Pca2_1 was later proved correct by a successful structure analysis. The preliminary unit cell dimensions were adjusted by a least-squares treatment of the setting angles for 22 reflections, centred on a Hilger and Watts' Y290 four-circle diffractometer controlled by a PDP8 computer.

The intensities of reflections were measured on the Y290 diffractometer, using molybdenum radiation, a graphite monochromator and a pulse-height analyser. The θ-2θ scan technique was employed. Each reflection was scanned through a θ range of 0.6°, with a scan step of 0.02° and a counting time of 2s per step. The local background was counted for 15s at each end of the scan range. The intensities of two strong reflections, periodically remeasured throughout the experiment, varied by less than ± 5% of their mean values.

The integrated intensities, *I*, and their standard deviations, $\sigma(\underline{I})$, were obtained using relationships described earlier ($\underline{q}$ = 0.04) [13]. They were corrected for Lorentz, polarisation, counting-loss and absorption effects. The transmission factors on $\underline{F}^2$, calculated by Gaussian integration, varied between 0.07 and 0.16.

The intensities of all $\underline{h\bar{k}\bar{l}}$ reflections with $\theta(\text{Mo-}\underline{K}_\alpha) \leq 35°$ were measured. Of these, only 1820 (*ca*. 66%), for which $\underline{I} \geqslant 3\sigma(\underline{I})$, were used in the subsequent calculations.

Structure analysis

The position of the platinum atom, at $\underline{z}$ = 1/4, was found from a Patterson function. With $\underline{Z}$ = 4, the space group *Pcam* would then require all molecules to lie in mirror planes normal to $\underset{\sim}{c}$. This proved incompatible with the difference synthesis phased by the platinum atom, and the space group $\underline{Pca}2_1$ was therefore adopted in the subsequent analysis. Interpretation of this synthesis was complicated by pseudo-symmetry, giving rise to four possible arrangements for atoms co-ordinated to platinum. Each of these arrangements was refined and the one which gave an acceptable set of platinum-ligand bond lengths and angles, and also the lowest value of *R*, was used in further calculations. The positions of the remaining non-hydrogen atoms were determined from subsequent difference syntheses.

The structure was refined by a least-squares minimisation of the function $\Sigma\{(|\underline{F}_o|-|\underline{F}_c|)/\sigma(\underline{F}_o)\}^2$. The atomic scattering factors were taken from ref. 14, and the anomalous scattering of platinum, chlorine and phosphorus atoms was accounted for [14]. Hydrogen atoms were not located. Refinement of the ositional and anisotropic vibrational parameters of all non-ydrogen atoms converged at $\underline{R} = 0.037$ and $\underline{R}_w = 0.047$.

The correctness of the indexing of reflections was then rified, by refining the structure with $\underline{hk\bar{l}}$ reflections -indexed as $\underline{h\bar{k}l}$. This refinement converged at $\underline{R} = 0.039$ nd $\underline{R}_w = 0.049$, both significantly greater than the values tained with the original indexing.

In the last cycle of refinement all parameters shifted $\leq 0.05\sigma$. The standard deviation of an observation of unit eight was 1.71. The mean values of $(|\underline{F}_o| - |\underline{F}_c|)^2/\sigma^2(\underline{F}_o)$ howed no systematic trends when analysed as a function of

BLE 1

ACTIONAL ATOMIC COORDINATES

om	$\underline{x}$	$\underline{y}$	$\underline{z}$
Si	0.04379(3)	0.03769(3)	1/4
(1)	0.1001(3)	0.2962(3)	0.2146(3)
(2)	0.1884(3)	-0.0513(4)	0.1511(4)
	-0.0024(2)	-0.2141(3)	0.2789(3)
	-0.1411(8)	0.1743(13)	0.3710(14)
1)	-0.0731(11)	0.1187(14)	0.3245(14)
2)	0.1003(9)	-0.3213(15)	0.3563(12)
3)	0.1231(13)	-0.2580(19)	0.4789(15)
)	-0.1270(11)	-0.2363(15)	0.3580(13)
3)	-0.1503(13)	-0.4099(17)	0.3829(15)
3)	-0.0189(10)	-0.3149(13)	0.1414(11)
)	-0.1042(12)	-0.2477(17)	0.0656(15)

TABLE 2

THERMAL PARAMETERS OF ATOMS[a]

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pt	45.1(2)	33.5(1)	45.2(2)	1.5(1)	-0.4(3)	-4.1(3)
Cl(1)	77(2)	38(1)	75(2)	-8(1)	-6(2)	3(1)
Cl(2)	64(2)	58(2)	92(3)	0(2)	32(2)	-10(2)
P	45(1)	34(1)	45(1)	-1(1)	-1(1)	-5(1)
O	67(7)	68(6)	147(12)	25(6)	25(8)	-18(8)
C(1)	57(7)	37(5)	83(9)	-6(5)	4(7)	-2(6)
C(2)	45(6)	48(6)	63(7)	5(5)	-6(5)	1(5)
C(3)	73(9)	79(9)	69(9)	12(8)	-13(7)	-3(8)
C(4)	57(7)	53(7)	58(7)	-5(6)	8(6)	-4(6)
C(5)	87(10)	51(6)	80(10)	-15(7)	6(9)	2(7)
C(6)	61(7)	41(5)	50(6)	-1(5)	0(5)	-13(5)
C(7)	62(8)	69(9)	69(9)	6(7)	-17(7)	-6(8)

[a] Each atom was assigned an anisotropic temperature factor of the form $\exp(-2 \times 10^{-3}\pi^2 \sum_{i=1}^{3} \sum_{j=1}^{3} h_i h_j a_i^* a_j^* U_{ij})$.

$|F_o|$ or sinθ.* The extreme function values in the final difference synthesis (1.5 and -1.6 eÅ^{-3}), were associated with the position of the platinum atom. The final positional and vibrational parameters of atoms are presented in Tables 1 and 2, and a view of the molecular structure is shown in Figure 1.

The computer programs used are listed in ref. 12.

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* A list of the final values at $|F_o|$ and $|F_c|$ can be obtained from the authors on request.

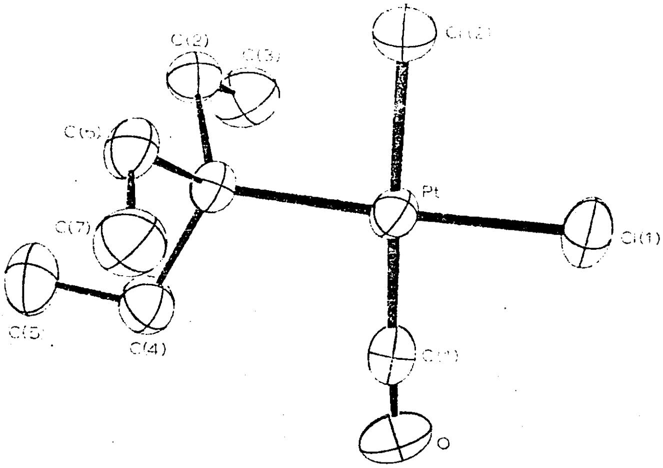

Figure 1. A perspective view of the molecule, with thermal ellipsoids displaying 50% probability. Hydrogen atoms are omitted.

Results and discussion

Crystal and molecular structure of *cis*-[$PtCl_2(PEt_3)(CO)$]

The crystal structure is built of discrete monomeric molecules. The shortest distances between atoms in different molecules are close to the sums of the appropriate van der Waals radii.

The molecules display a *cis*-square planar coordination around the platinum atom and almost ideal C_s symmetry.

The orientation of the phosphine ligand, evident from the Cl(2)-Pt-P-C torsion angles (Table 3), is such as to bring the ethyl group involving the atoms C(4) and C(5) into the coordination plane of platinum. The arrangement of the other two ethyl groups is such as to make the planes through the atoms P, C(2) and C(3) and P, C(6) and C(7) nearly coincident (dihedral angle 3°), and both normal to the plane defined by the atoms P, C(4) and C(5) (dihedral angles 93 and 90°). The conformations about the P-C bonds are staggered, as shown by the Pt-P-C-C torsion angles (Table 3). From an inspection of models it appears that such a conformation of the PEt_3 ligand and its orientation, with respect to the coordination plane of the metal atom, are favourable for the minimisation of steric repulsions in a square planar molecule. It is therefore not surprising that similar PEt_3 conformations and orientations have been observed in several other cis-$[PtCl_2(PEt_3)L]$ molecules [12]. The bond lengths and angles in the triethylphosphine ligand are normal (Table 3), the Pt-P-C and C-P-C angles showing the expected deviations from the ideal tetrahedral value [15].

The non-bonding intramolecular contacts and the angular distortions in the coordination plane of platinum indicate that the molecule is subject to some steric strain. Thus the C(1)...C(4), Cl(2)...C(2) and Cl(2)...C(6) distances are 3.15, 3.48 and 3.49Å, respectively, and the P-Pt-C(1) and P-Pt-Cl(2) angles deviate from 90° by 4.7 and -2.1° (Table 3). The individual displacements of the Pt, P, Cl(1) and Cl(2) atoms from their least-squares plane* do not exceed 0.002Å; the displacements of the C(1), O, C(4) and C(5) atoms from the same plane are less than 0.06Å.

The Pt-Cl(1) and Pt-Cl(2) distances, 2.368(3) and 2.296(4)Å respectively, show that the triethylphosphine ligand exerts a substantially larger trans-influence than the carbonyl group, while the Pt-P distance [2.265(3)Å] reflects the cis-influence of the carbonyl group (see below). The Pt-C distance [1.855(14)Å] is the same as that [1.858(7)Å] in the analagous compound cis-$[PtCl_2(PPh_3)(CO)]$, and indicates that

---

* Defined by the equation $-0.518\underline{X} + 0.011\underline{Y} - 0.855\underline{Z} = -2.729$; $\underline{X}$, $\underline{Y}$, and $\underline{Z}$ are co-ordinates referred to orthonormal axes along $\utilde{a}$, $\utilde{b}$, and $\utilde{c}$.*

the co-ordinated carbon monoxide possesses appreciable π-acceptor properties [4].

TABLE 3

SELECTED INTERATOMIC DISTANCES AND ANGLES

Bond lengths (Å)			
Pt-Cl(1)	2.368(3)	P-C(6)	1.806(13)
Pt-Cl(2)	2.296(4)	C(2)-C(3)	1.529(21)
Pt-P	2.265(3)	C(4)-C(5)	1.546(20)
Pt-C(1)	1.855(14)	C(6)-C(7)	1.507(20)
P-C(2)	1.830(13)	O-C(1)	1.124(19)
P-C(4)	1.840(14)		
Bond angles (°)			
Cl(1)-Pt-Cl(2)	89.0(1)	Pt-P-C(2)	111.4(4)
Cl(1)-Pt-C(1)	88.4(4)	Pt-P-C(4)	113.3(4)
P-Pt-Cl(2)	87.9(1)	Pt-P-C(6)	111.2(4)
P-Pt-C(1)	94.7(4)	C(2)-P-C(4)	109.3(6)
Cl(1)-Pt-P	177.0(1)	C(2)-P-C(6)	105.3(6)
Cl(2)-Pt-C(1)	176.9(4)	C(4)-P-C(6)	106.0(6)
P-C(2)-C(3)	113.6(9)	P-C(6)-C(7)	113.6(9)
P-C(4)-C(5)	110.9(10)	Pt-C(1)-O	176.5(12)
Torsion angles (°)			
Cl(2)-Pt-P-C(2)	58(1)	Pt-P-C(2)-C(3)	62(1)
Cl(2)-Pt-P-C(4)	-178(1)	Pt-P-C(4)-C(5)	-176(1)
Cl(2)-Pt-P-C(6)	-59(1)	Pt-P-C(6)-C(7)	-62(1)

cis- And *trans*-influence of ligands in *cis*-[$PtCl_2(PEt_3)L$] complexes

Accurate crystallographic studies are now available for eight complexes of the type *cis*-[$PtCl_2(PEt_3)L$], where the ligands L display a wide range of electronic properties. Both strong and weak σ-donors, such as PEt_3 and Cl^-, and also strong and weak π-acceptors, such as CO and carbenoid, are represented. The bond lengths in these complexes are listed in Table 4, together with the $^1\underline{J}(Pt\text{-}PEt_3)$ coupling constants. They enable us to examine in some detail the dependence of metal-ligand bonding upon the nature of the ligands L.

TABLE 4

BOND LENGTHS (Å) AND COUPLING CONSTANTS (Hz) in *cis*-[$PtCl_2(PEt_3)L$] COMPLEXES

L	Pt-P	Pt-Cl (*cis* to L)	Pt-Cl (*trans* to L)	^1J ($Pt\text{-}PEt_3$)	Ref.
Cl^-	2.215(4)	2.382(4)	2.301(3)[a]	3704	6
$C(NPhCH_2)_2$	2.234(3)	2.381(3)	2.362(3)	3720[b]	8
CNPh	2.238(8)	2.365(11)	2.333(12)	3049[c]	9
C(OEt)NHPh	2.239(8)	2.367(7)	2.361(5)	-	7
PEt_3	2.259(2)[a]	2.361(6)[a]	2.361(6)[a]	3515[d]	10
CO	2.265(3)	2.368(3)	2.296(4)	2754[c]	This work
$P(OPh)_3$	2.269(1)	2.355(2)	2.344(2)	3210[d]	12
PF_3	2.272(3)	2.357(3)	2.305(3)	2760	11

[a] Mean value. [b] Ref. 16. [c] Ref. 9. [d] Ref. 17.

The Pt-Cl(trans to L) bond lengths in Table 4 display a variation of about 0.07Å and increase along the series $CO \simeq Cl^- \simeq PF_3 < CNPh \leq P(OPh)_3 < PEt_3 \simeq C(OEt)NHPh \simeq C(NPhCH_2)_2$. This series reflects the increasing σ-basicity and decreasing π-acidity of the ligands and it is therefore compatible with current views on the trans-influence of ligands in transition metal complexes [1,18,19]

The platinum-ligand bonds cis to L are also affected by the nature of L. The Pt-P distances vary by about 0.06Å, almost as much as the Pt-Cl(trans to L) distances. The variation in Pt-Cl(cis to L) distances is smaller, ca. 0.03Å, but still statistically significant. In addition, we note that the

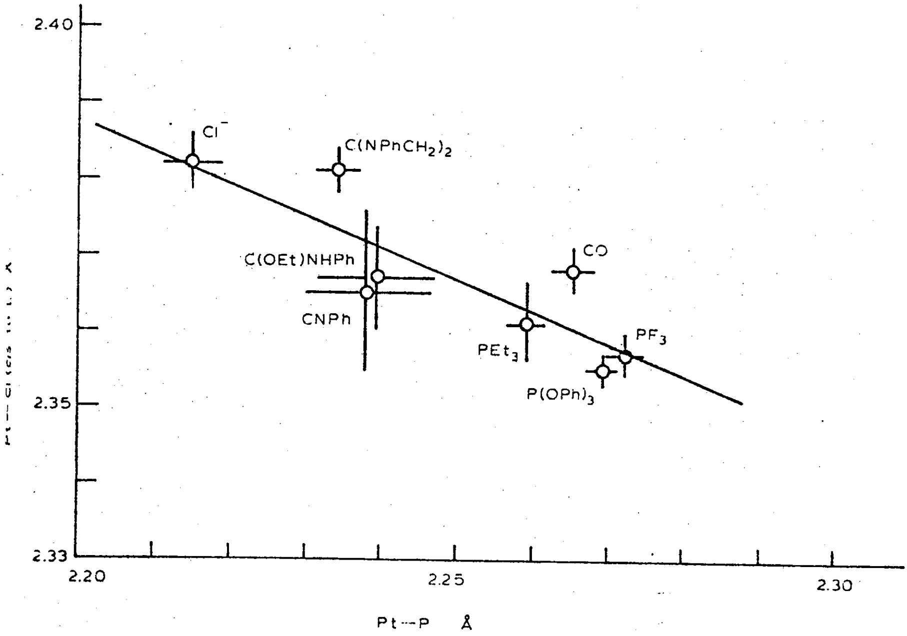

Figure 2. A plot of Pt-Cl(cis to L) versus Pt-P bond lengths in cis-$[PtCl_2(PEt_3)L]$ complexes (see Table 4). The ligands L and the unweighted least-squares trend line are shown. The errors indicated are standard deviations.

Pt-Cl(cis to L) distances display a consistent trend, illustrated in Figure 2: they decrease as the Pt-P distances increase (linear correlation coefficient -0.9).

Considering the cis-influence of ligands as their ability to weaken the cis-metal-ligand bonds, it is obvious that in cis-[$PtCl_2(PEt_3)L$] complexes the ligands L can be arranged in a cis-influence series on the basis of either the Pt-P or Pt-Cl(cis to L) bond lengths. The Pt-P distances, which display greater variability, increase along the series $Cl^- < C(NPhCH_2)_2 \simeq CNPh \simeq C(OEt)NHPh < PEt_3 \simeq CO \simeq P(OPh)_3 \simeq PF_3$. This, of course, is approximately a reversal of the series of increasing Pt-Cl(cis to L) distances.

The cis-influence of ligands L may arise either from their steric or electronic properties, or perhaps from a combination of both. It is now recognised that in severely overcrowded platinum(II) complexes the steric repulsions between ligands can lead to considerable lengthening of Pt-P bonds. Thus in trans-[$PtI_2\{P(C_6H_{11})_3\}_2$] [20] the Pt-P bonds are about 0.06Å longer than in trans-[$PtBr_2(PEt_3)_2$] [21], and this is attributed mainly to the change in steric demands of the ligands in the two complexes. In the less crowded cis-[$PtCl_2(PEt_3)L$] molecules discussed here the steric interactions of ligands are expected to be considerably weaker. To what extent, if at all, they affect the length of the Pt-P bonds is difficult to establish, since the force constants required for molecular mechanics calculations are not known. In this predicament we note that the observed cis-influence series bears little relationship to the size of ligands L, as measured, in the absence of a less crude estimate, by Tolman's cone angle (95,102,104,130 and 132° for CO, Cl^-, PF_3, $P(OPh)_3$ and PEt_3, respectively) [22]. Ligands of similar size, such as Cl^-, CO and PF_3, occur at opposite ends of the series, while ligands of different size, such as CO, PF_3, $P(OPh)_3$ and PEt_3, exert similar cis-influences. Furthermore, on steric grounds one ight expect both bonds cis to L to lengthen as L becomes larger, thus leading to a positive correlation between the Pt-P and Pt-Cl(cis to L) distances. The observed correlation is, however, negative. We therefore consider that the steric properties of L are at most a minor factor in determining their position in the cis-influence series and, consequently, that the is-influence of L is predominantly an electronic effect.

Another important observation emerges from the bond length ata in Table 4: cis- and trans-influence of L are not related o each other, for the Pt-P and Pt-Cl(cis to L) distances how no correlation with the Pt-Cl(trans to L) distances. his indicates that cis- and trans-influence are transmitted hrough different electronic mechanisms in the molecular ramework. Current theories emphasize that only those ligands hich are strong σ-bases exert high trans-influence [1,18,19]. rom the observed trans-influence series of ligands L, PEt_3 s expected to be a stronger base than Cl^-, $P(OPh)_3$, or PF_3. he same relative basicities of the three phosphorus-donor igands are evident from i.r. and u.v. spectroscopic data [22]. he ordering of Cl^-, PEt_3, $P(OPh)_3$, and PF_3 in the cis-influence eries is then obviously not related to their σ-basicities. is is consistent with Syrkin's theory [23], which considers that teractions between mutually cis σ-bonds are of minor importance, d which has been followed in most subsequent discussions of ans-influence of ligands. Zumdahl and Drago however have edicted, on the basis of extended Hückel molecular orbital lculations, that cis- and trans-influence transmitted through bonds are of comparable magnitude [24].

The $^1J(Pt-PEt_3)$ coupling constants, which are thought measure the s-component of the Pt-P σ-bond [25], display a range of ca. 1000 Hz in cis-$[PtCl_2(PEt_3)L]$ complexes (Table 4). However, they show only an indifferent correlation with the Pt-P bond lengths, which reflect the overall Pt-P bond order. In the complexes with L = PEt_3 and CO, the Pt-P bond lengths are equal to within experimental error despite a difference in the coupling constants of 761 Hz. It therefore appears that, although both the overall and s-electron Pt-P bond orders are sensitive to the nature of the cis-ligand, there is no simple correspondence between the two quantities.

In the cis-influence series of L the ordering of ligands shows an obvious tendency: ligands which are considered to be strong π-acids, notably CO and PF_3, occur at the upper end of the series, while weak π-acids, such as Cl^- and carbenoid, occur at the lower end of the series. We therefore suggest that the cis-influence of L may reflect its π-acceptor properties. The lengthening of the Pt-P bonds can then be rationalized on the basis of an increasing competition between the L and PEt_3 ligands for the metal atom d_π-electrons. A necessary assumption here is that the PEt_3 ligand is a π-acid, albeit a weak one. The Pt-Cl(cis to L) bond lengths may also be directly affected by the ligands L, increased Pt→L backdonation leading to enhanced electrostatic attraction between platinum and the chloride ligand. Alternatively, it may be considered that the ligand L influences the cis-Pt-Cl bond only indirectly, by modifying the trans-influence of the phosphine.

In conclusion we note that the Pt-P bond is more sensitive to the nature of the cis-ligands than the Pt-Cl bond. It then follows that platinum-phosphorus bond lengths, coupling constants or stretching frequencies will provide a valid measure of trans-influence of ligands only if in the complexes compared the ligands cis to phosphorus are always the same. This precaution is less important if Pt-Cl bond parameters are used as a measure of trans-influence.

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Crystal and Molecular Structure of *cis*-Dichloro[1,2-bis(trifluoromethylthio)pane] platinum(II)

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he crystal structure of cis-$PtCl_2(F_3CSCHMeCH_2$-$_3)$ *has been determined by X-ray methods. The pound crystallises in the monoclinic system, group P2$_1$/n, with four molecules in a unit cell imensions* a = *7.557(2),* b = *12.942(2),* c = *40(2) Å, β = 91.42(2)°. The structure has been ed by full-matrix least-squares to R 0.054 (on) for 2301 diffractometric intensity data. The als contain discrete monomeric molecules in h the platinum atom displays the expected* cis-*e-planar co-ordination. Selected bond lengths Pt–S 2.239(3) and 2.260(3), and Pt–Cl 2.290(4) 2.295(3) Å. The metal–ligand bonding does not ar to be strongly influenced by the electron-drawing properties of the trifluoromethyl sub-ents of the sulphur atoms. The chelate ring has mmetrically-puckered* gauche *conformation. The yl substituent is* pseudo-*equatorial and the tri-omethyl groups are mutually* syn. *Pairs of osymmetrically-related molecules are arranged at there are short S···Cl and Pt···Pt contacts 3.4 Å.*

duction

is by now well established that the presence of on-withdrawing substituents on a ligand donor tends to shorten, and hence presumably to gthen, transition-metal–ligand bonds. Churchill hown, for example, that the metal–carbon bonds horter in fluoroalkyl complexes than in alkyl lexes of similar formulation and has discussed me length the electronic factors which may be nsible for this phenomenon [1]. The contro- over the extent of backdonation in transition- -phosphine complexes has led to much interest relationship between M–P bond lengths and the on-withdrawing ability of the substituents of hosphorus atom. At present one of the strongest ents in favour of the significant M→P back-ion is based upon the shortening of the Cr–P length in $X_3PCr(CO)_5$ complexes by 0.11 Å X is changed from C_6H_5 to C_6H_5O [2, 3]. have recently shown that the metal–ligand ing in *cis*-$M^{II}Cl_2[Ph_2PCH_2CH_2P(CF_3)_2]$ complexes, where M = Pd or Pt, is sensitive to the electron-withdrawing properties of the substituents on phosphorus [4]. The M–P bond lengths differ by *ca.* 0.07 Å, the shorter bond being adjacent to the trifluoromethyl groups. The M–Cl distances indicate that the *trans*-influence of the $P(CF_3)_2$ group is much weaker than that of the PPh_2 group. In this context it is worth noting that trifluoromethyl comes highest in Tolman's ranking of substituent electron-withdrawing ability [5].

The recent synthesis of the complex *cis*-$PtCl_2$-$(F_3CSCHMeCH_2SCF_3)$ has given us the opportunity to investigate the effect of trifluoromethyl substituents on the *trans*-influence and bonding to platinum of a thioether ligand [6]. A further motive for the structure analysis was provided by the ^{19}F n.m.r. spectrum of the complex in acetone at ambient temperature. This spectrum is consistent with the presence in solution of four diastereoisomeric forms of the complex. Over the temperature range 173–323 °K rapid interconversion of isomers does not appear to occur. Two of the isomers display long range F–F coupling which may be associated with a *syn* arrangement of the trifluoromethyl groups [6]. We felt that the determination of the molecular structure of the complex in the solid-state might contribute to the interpretation of these results.

Experimental

Crystal Data

$PtCl_2(F_3CSCHMeCH_2SCF_3)$, M = 510.2, monoclinic, a = 7.557(2), b = 12.942(2), c = 12.340(2) Å, β = 91.42(2)°, U = 1206.5 Å^3, Z = 4, D_c = 2.808 g cm^{-3}, F(000) = 936, Mo-K$_\alpha$ radiation, λ = 0.71069 Å, μ(Mo-K$_\alpha$) = 125.7 cm^{-1}, space group C_{2h}^5 (No. 14)–P2$_1$/n with equivalent positions ± (x, y, z); ± (½ + x, ½ – y, ½ + z).

Measurements

The crystals are needles elongated along *a*. The dimensions of the specimen used in the analysis were 0.66 × 0.14 × 0.16 mm. The space group and approximate cell dimensions were determined from Weissenberg and rotation photographs. Final values

TABLE I. Final Fractional Co-ordinates and Thermal Parameters

Atom	x^a	y	z	U_{iso} or U_{11} ^{b}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pt	09767(6)	09798(3)	07246(3)	401(3)	334(2)	380(2)	−33(2)	65(2)	−34(2)
S(1)	−0400(4)	0337(2)	2163(2)	55(2)	40(2)	42(2)	−2(1)	8(1)	4(1)
S(2)	−1189(4)	2167(2)	0477(2)	55(2)	35(1)	43(2)	0(1)	4(1)	1(1)
Cl(1)	2361(5)	1594(3)	−0779(3)	69(2)	65(2)	49(2)	−10(2)	21(2)	2(2)
Cl(2)	3134(4)	−0246(3)	0978(3)	48(2)	54(2)	79(2)	11(2)	2(2)	−4(2)
F(1)	0582(17)	3776(7)	−0075(10)	128(10)	58(6)	126(9)	−28(6)	41(7)	7(6)
F(2)	−1388(16)	4107(6)	1021(10)	124(9)	40(5)	136(9)	2(5)	50(7)	−8(5)
F(3)	1017(17)	3406(7)	1584(10)	140(10)	59(6)	129(9)	−30(6)	−60(8)	5(6)
F(4)	−0438(17)	0836(8)	4188(6)	133(8)	122(9)	40(5)	−1(7)	18(5)	−2(5)
F(5)	2087(15)	0503(10)	3583(9)	106(8)	127(9)	99(8)	54(8)	−48(7)	−31(7)
F(6)	0874(13)	1939(7)	3266(7)	106(7)	68(6)	66(5)	−11(5)	−17(5)	−15(5)
C(1)	−0182(21)	3418(10)	0776(13)	71(10)	34(7)	74(10)	5(7)	1(8)	5(7)
C(2)	−2550(17)	2070(10)	1701(11)	48(7)	47(7)	65(8)	6(6)	7(6)	1(7)
C(3)	−2561(17)	0969(11)	2084(11)	44(7)	63(8)	57(7)	1(7)	12(6)	8(7)
C(4)	−4430(19)	2454(12)	1455(13)	51(9)	66(9)	87(11)	7(7)	−1(8)	−5(8)
C(5)	0610(21)	0959(12)	3367(10)	77(10)	67(10)	38(7)	12(9)	−1(6)	−2(7)
H(1)	−309	093	282	7(4)					
H(2)	−333	055	156	11(6)					
H(3)	−201	251	230	9(5)					
H(4)	−499	201	088	11(6)					
H(5)	−516	239	213	7(4)					
H(6)	−443	319	122	15(7)					

[a] Fractional co-ordinates have been multiplied by 10^5 for Pt, by 10^3 for H, and by 10^4 for other atoms. [b] The form of the anisotropic temperature factor is $\exp(-2\pi^2 \times 10^{-n} \sum_{i=1}^{3} \sum_{j=1}^{3} U_{ij}h_ih_ja_i^*a_j^*)$, where $n = 4$ for Pt and 3 for other atoms; U_{iso} values for hydrogen atoms are multiplied by 10^2.

of the unit cell dimensions and the intensities of all unique reflexions with $\theta(\text{Mo-K}_\alpha) \leqslant 30°$ were measured using standard techniques [7] on a Hilger and Watts Y290 diffractometer. Each reflexion was scanned symmetrically over 35 steps of 0.02° in θ/ω. At each step counting continued for 2.5 s. The background was measured at each extreme of the scan for 15 s, with crystal and counter stationary. The intensities of three standard reflexions, which were periodically remeasured during the experiment, displayed only random fluctuations of less than 5% of the corresponding mean values.

Structure amplitudes and their standard deviations were derived as described previously, the empirical factor q being taken as 0.04 [7]. Corrections were made for absorption using a Gaussian integration method. The transmission factors on F_0^2 ranged between 0.14 and 0.23. A total of 2301 unique reflexions with $I \geqslant 3\sigma(I)$ were used in the subsequent analysis.

Structure Analysis

The platinum atom was located from the three-dimensional Patterson function and the other atoms, except for hydrogen, from subsequent difference syntheses.

The structure was refined by full-matrix least-squares minimisation of $\Sigma\{(|F_0| - |F_c|)/\sigma(F_0)\}^2$. Atomic scattering factors, apart from that for hydrogen [8], and also the anomalous dispersion corrections for Pt, Cl, and S atoms were taken from International Tables [9]. Adjustments of the positional and isotropic vibrational parameters of the non-hydrogen atoms led to R = 0.08. When anisotropic temperature factors were introduced R fell to 0.064. The positions of the hydrogen atoms were then calculated so as to be consistent with the stereochemistry of adjacent carbon and sulphur atoms; they were compatible with appropriate peaks in a low-angle difference synthesis. Allowance was then made for the scattering of the hydrogen atoms; the calculated positional parameters were kept fixed but the isotropic thermal parameters were allowed to vary. Values of R and R′ were thus reduced to 0.058 and 0.065. The absorption correction was then applied and the refinement converged with R 0.054 and R′ 0.060. In the final cycle of refinement no parameter of a non-hydrogen atom shifted by more than 0.02σ. The final difference synthesis was featureless, apart from extreme function values of + 3.0 and − 4.8 eÅ^{-3} close to the position of the platinum atom. The adequacy of the weighting scheme was

LE II. Interatomic Distances (Å) and Angles (°)

ond Lengths

S(1)	2.239(3)
S(2)	2.260(3)
Cl(1)	2.295(3)
Cl(2)	2.290(4)
-C(3)	1.83(1)
-C(5)	1.84(1)
-C(1)	1.82(1)
-C(2)	1.85(1)
-C(3)	1.50(2)
-C(4)	1.53(2)
-F(1)	1.30(2)
-F(2)	1.32(2)
-F(3)	1.33(2)
-F(4)	1.31(2)
-F(5)	1.28(2)
-F(6)	1.29(2)

nterbond Angles

-Pt–S(2)	90.6(1)
-Pt–Cl(1)	178.2(1)
-Pt–Cl(2)	88.7(1)
-Pt–Cl(1)	89.9(1)
-Pt–Cl(2)	179.0(1)
-Pt–Cl(2)	90.8(1)
(1)–C(3)	102.9(4)
(1)–C(5)	106.6(5)
-S(1)–C(5)	101.5(7)
(2)–C(1)	106.2(5)
(2)–C(2)	105.0(4)
-S(2)–C(2)	97.6(7)
C(2)–C(3)	109.2(9)
C(2)–C(4)	110.4(10)
C(2)–C(4)	111.0(11)
C(3)–C(2)	115.5(9)
C(1)–F(1)	110.2(10)
C(1)–F(2)	111.1(11)
C(1)–F(3)	114.5(10)
C(1)–F(2)	105.6(12)
C(1)–F(3)	107.6(13)
C(1)–F(3)	107.4(12)
C(5)–F(4)	108.9(11)
C(5)–F(5)	108.0(11)
C(5)–F(6)	114.4(10)
C(5)–F(5)	108.8(12)
C(5)–F(6)	107.0(12)
C(5)–F(6)	109.6(14)

(c) Intramolecular Non-Bonded Distances

F(3)···F(6)	2.817(14)
S(1)···Cl(2)	3.167(5)
S(2)···Cl(1)	3.218(5)
S(1)···S(2)	3.199(4)
Cl(1)···Cl(2)	3.264(5)

(d) Intermolecular Contacts Less than the Sum of the van der Waals Radii

$S(1)\cdots Cl(1^{I})$	3.352(5)
$S(2)\cdots Cl(2^{I})$	3.382(5)
$Pt\cdots Pt^{I}$	3.417(1)
$Cl(1)\cdots C(2^{II})$	3.560(14)
$Cl(1)\cdots C(3^{I})$	3.693(14)
$Cl(1)\cdots C(4^{III})$	3.793(15)

Roman numerals refer to the folllwing transformations of the fractional co-ordinates in Table I:

I	$\bar{x}$	$\bar{y}$,	$\bar{z}$;
II	½+x,	½–y,	–½+z;
III	1 +x,	y,	z.

(e) Torsion Angles

PtS(1)C(5)F(4)	163(0.9)
PtS(1)C(5)F(5)	–79(1.1)
PtS(1)C(5)F(6)	43(1.2)
C(3)S(1)C(5)F(4)	55(1.1)
C(3)S(1)C(5)F(5)	173(1.0)
C(3)S(1)C(5)F(6)	–64(1.2)
PtS(2)C(1)F(1)	85(1.1)
PtS(2)C(1)F(2)	–158(0.9)
PtS(2)C(1)F(3)	–37(1.2)
C(2)S(2)C(1)F(1)	–167(1.1)
C(2)S(2)C(1)F(2)	–50(1.1)
C(2)S(2)C(1)F(3)	72(1.2)
PtS(1)C(3)C(2)	–37(1.0)
C(5)S(1)C(3)C(2)	73(1.1)
S(1)C(3)C(2)S(2)	47(1 2)
S(1)C(3)C(2)C(4)	169(0.9)
C(3)C(2)S(2)Pt	–33(1.0)
C(3)C(2)S(2)C(1)	–142(1.0)
C(4)C(2)S(2)Pt	–156(0.9)
C(4)C(2)S(2)C(1)	95(1.0)
C(2)S(2)PtS(1)	10(0.5)
C(1)S(2)PtS(1)	112(0.5)
S(2)PtS(1)C(3)	11(0.5)
S(2)PtS(1)C(5)	–96(0.5)

d by establishing that mean values of $||F_o| -$ (F_o) showed little variation with either $|F_o|$ or . The standard deviation of an observation of eight was 2.0. Extinction corrections were not d.

e final atomic parameters and a selection of ons derived from them are presented in Tables A perspective view of the molecule is displayed in Figure 1. Final observed and calculated structure amplitudes may be obtained by application to the Editor.

The programs used in this work were the HILGER data processing program of P. R. Mallinson, K. W. Muir and D. N. J. White, the Hilger and Watts software system for the Y290 diffractometer, and J. M. Stewart's X-RAY72 system.

TABLE III. Equations[a] of, and Atomic Displacements (Å × 10^3) from, Weighted Least-squares Planes.

Plane 1, defined by Pt, S(1), S(2), Cl(1) and Cl(2):
$-0.499X - 0.637\,Y - 0.587\,Z = -1.689$;
Pt −2(1), S(1) 28(3), S(2) 12(3), Cl(1) 35(4), Cl(2) 16(4).

Plane 2, defined by Pt, S(1), S(2), C(2), C(3):
$-0.488\,X - 0.633\,Y - 0.600\,Z = -1.689$;
Pt 0(1), S(1) −9(3), S(2) 6(3), C(1) −1608(15), C(2) −300(13), C(3) 328(13), C(4) 257(15), C(5) −1765(15).

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[a] In terms of orthogonal co-ordinates X, Y, Z along a, b and c^*.

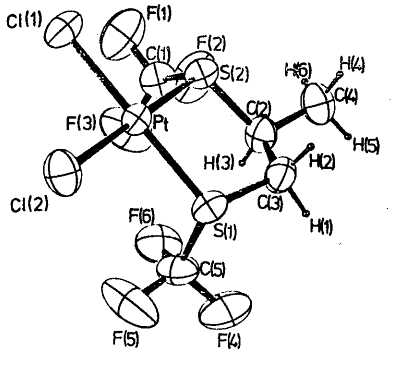

Fig. 1. A perspective view of the molecule.

Discussion

The crystals contain $PtCl_2(F_3CSCHMeCH_2SCF_3)$ molecules which exhibit the expected *cis*-square-planar co-ordination at the platinum atom (Figure 1). The methyl group is *pseudo*-equatorial with respect to the chelate ring and the trifluoromethyl substituents of the sulphur atoms are mutually *syn*. Comparisons with other bis(thioether) chelate complexes of platinum(II) cannot be made since there is no structural information available. However, in bis(thioether) and bis(selenoether) chelate complexes of palladium(II) the terminal substituents of the group VIA donor atoms are usually found to be *syn* [10, 11]. The only exception involves an unusual macrobicyclic ligand where the *anti* configuration may well be a consequence of the constrained geometry of the ligand [12].

The crystal packing is predominantly of the van der Waals type (Table IId), but it also involves strong interactions between pairs of centrosymmetrically-related molecules (Figure 2). The constituent molecules of each pair are arranged so that the platinum co-ordination planes are antiparallel and the metal–ligand bonds are eclipsed. The resulting Pt···Pt separation of 3.42 Å is too long to be indicative of normal covalent bonding; the lengths of Pt–Pt single bonds in platinum(II) complexes are typically 2.77–2.87 Å [13, 14]. The Pt···Pt contact is also longer than the corresponding distance in Magnus' Green Salt (3.25 Å) [15] and related compounds. However, it agrees well with the value of 3.39 Å found in *cis*-dichloro(ethylenediamine)platinum(II), crystals of which contain infinite stacks of molecules arranged so that adjacent metal co-ordination planes are antiparallel [16]. In *cis*-$PtCl_2(F_3CSCHMeCH_2SCF_3)$ the formation of infinite stacks of molecules appears to be prevented by the *syn* trifluoromethyl groups, which leave only one side of the metal co-ordination plane open for close approach to a neighbouring molecule. The eclipsing of the metal–ligand bonds leads to intermolecular Cl···S separations of 3.35 and 3.38 Å which are slightly shorter than the Pt···Pt contact. This is brought about by a small but significant pyramidal distortion of the platinum co-ordination. The displacement of the platinum atom from the weighted PtS_2Cl_2 plane is −0.002(1) Å, whereas the displacements of the atoms S(1), S(2), Cl(1), and Cl(2) are respectively 0.028(3), 0.012(3), 0.035(4), and 0.016(4) Å (a positive displacement is in the direction of the centrosymmetrically-related molecule). This contrasts with the situation in complexes containing covalent Pt–Pt bonds, such as bis(ethylene-1,2-dithiolato)platinum (II) dimer, for example [13], where the metal atoms are displaced by *ca.* 0.1 Å from their co-ordination planes towards rather than away from each other. Accordingly, we consider that the pairing of centrosymmetrically-related *cis*-$PtCl_2(F_3CSCHMeCH_2SCF_3)$ molecules is best explained by electrostatic interactions between electron-deficient sulphur atoms and negatively charged chloro ligands and that there is little, if any, direct bonding between the platinum atoms. This view is in conformity with a semi-empirical M.O. calculation on Magnus' Green Salt which indicated that the covalent Pt–Pt bond order is about 0.04 [17].

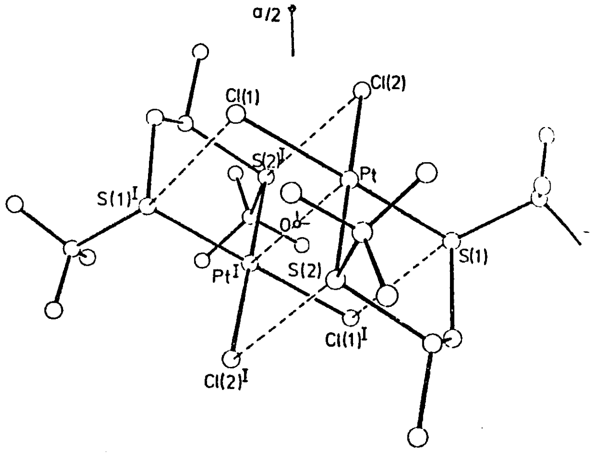

Fig. 2. The crystal packing.

Despite the pyramidal distortion of its co-ordination, valency angles subtended at the platinum atom are all within 2° of the ideal values of 90 or 180°. The Pt–Cl bond lengths are equal. Their mean of 2.293(3) Å lies at the lower end of the range of terminal Pt^{II}–Cl distances (2.26–2.45 Å) [18], indicating that in this compound the *trans*-influence of the thioether ligands is relatively weak. The Pt–S distances of 2.239(3) and 2.260(3) Å differ slightly, the longer bond being adjacent to the chelate-ring methyl substituent. Assessment of the influence of the electron-withdrawing trifluoromethyl groups on the metal–ligand bonding is rendered difficult by the lack of structural data on related compounds. The problem is further complicated by the participation of the sulphur and chlorine donor atoms in strong intermolecular interactions. Perhaps the most directly comparable structure is that of *cis*-$PtCl_2[S(p\text{-}C_6H_4Cl)_2]_2$ where the mean Pt–S and Pt–Cl distances are 2.285(7) and 2.300(5) Å [19]. Shorter, but less accurately determined Pt–S distances of 2.25 Å *trans* to amine in chloro(glycyl-*L*-methionine)platinum(II) and of 2.26 Å *trans* to chlorine in dichloro(*L*-methionine)platinum(II) have also been reported [20]. In the latter compound the Pt–Cl (*trans* to S) bond length is 2.32 Å. The only other Pt–S (thioether) distance which has been determined is that for the bridging bonds in μ-$(SEt_2)_2(PtBr_2)_2$; the mean value of 2.22(1) Å is remarkably short, for reasons which at present are uncertain [21]. We can suggest only tentatively that, by comparison with *cis*-$PtCl_2[S(p\text{-}C_6H_4Cl)_2]_2$, the trifluoromethyl groups in *cis*-$PtCl_2(F_3CSCHMeCH_2SCF_3)$ have little effect on the *trans*-influence of the thioether ligand, but that they may be responsible for a contraction of the Pt–S bonds by 0.03–0.05 Å.

The two trifluoromethyl groups adopt similar conformations relative to the chelate ring, so that corresponding torsion angles about the S–C bonds agree to within 8°. The conformations are such that the C–F bonds involving the atoms F(3) and F(6) point inwards and almost towards each other. The resulting F(3)···F(6) non-bonded contact of 2.82 Å, though greater than the van der Waals diameter of fluorine (2.70 Å), nevertheless suggests that the isomer present in the solid may be one of those which display F–F coupling in acetone solution. Interestingly, the S–C–F angles involving F(3) and F(6) are both some 5° larger than the other S–C–F angles, which average 109.6(7)°. However, there are no significant differences between the C–F bond lengths or between F–C–F bond angles, the respective means being 1.305(7) Å and 107.7(6)°.*

Both sulphur atoms adopt similar *quasi*-tetrahedral co-ordinations. Corresponding interbond angles at sulphur agree to within 4°, and all are less than the tetrahedral angle (109.5°). The four S–C bond lengths agree to within experimental error, the mean value of 1.835(7) Å being typical for a bond of unit order.

The chelate ring has a symmetrically-puckered *gauche* conformation. This is apparent from the internal torsion angles: the CCSPt angles differ by only 4° and the CSPtS angles by 1°. It is also evident from the displacements of the atoms from the weighted PtS_2C_2 mean plane (Table III); the platinum and sulphur atoms lie within 0.01 Å of the plane whereas the atoms C(2) and C(3) are displaced by nearly equal amounts (0.30 and 0.33 Å) in opposite directions.

Molecules of *cis*-$PtCl_2(F_3CSCHMeCH_2SCF_3)$ contain four chiral centres, namely the asymmetric atoms S(1), S(2), and C(2), and the chelate ring, so that in principle eight enantiomeric pairs of diastereoisomers may exist. The crystalline form which we have studied is, of course, racemic. In those molecules in which the chelate ring configuration is δ [S(1)C(3)C(2)S(2) torsion angle +47°, as in Figure 1], the absolute configurations at the atoms S(1), S(2), and C(2) are respectively (*R*), (*S*), and (*S*).

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*Limits of error on means are standard deviations and are the larger of the estimates: $(\sum_i \sigma_i^{-2})^{-1/2}$ and $[\sum_i (x_i - \bar{x})^2/(n - 1)]^{1/2}$, where the n individual bond lengths, or angles, have standard deviations σ_i and mean $\bar{x}$.

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APPENDIX II

to the thesis
submitted to the University of Glasgow
for the degree of Doctor of Philosophy
in the Faculty of Science
by
Tihomir Solomun

Tables of Observed and Calculated Structure Factors

Chemistry Department February 1979

Compound:

$\underline{\textbf{cis}}$-$PtCl_2(PMe_2C_6F_5)_2$

3

```
 -15,-6,L     10 238 240     1 527 546    -13,-1,L      1 318 325     9 226 239    -2 501 501    12 280 286
  5 175 193   11 202 237     3 373 406    -4 269 271    7 280 296    10 210 237    12 607 606    -11,-9,L
 -15,-5,L     -14,-2,L       5 265 272     4 422 442   11 360 377    11 402 401    14 358 364    -5 236 243
  5 216 205    2 268 273     7 192 185     6 602 612   13 277 324    12 358 382    -11,-14,L     -3 324 351
 -15,-4,L      4 522 542    13 255 283     8 498 521    -12,-7,L     13 407 402     4 179 152    -2 180 181
  6 162 178    5 224 216    -13,-5,L      10 371 395   -1 199 217    15 351 345    -11,-13,L     -1 383 363
  7 294 297    6 470 483    -5 280 291    12 285 319    1 322 331    -12,-3,L      -2 303 307     0 219 207
  9 268 358    8 244 236    -3 311 310    -13,0,L       3 495 482    -8 178 219     0 339 336     1 362 351
 -15,-2,L      -14,-1,L      3 281 275    -4 302 309    5 566 580    -7 185 189     2 358 357     2 237 245
  6 284 338   -2 244 250     5 241 285     0 337 364    6 168 156    -1 257 254     4 333 342     3 461 461
  8 308 344    0 247 262     6 179 182     2 574 596    7 488 503     0 289 311     6 221 264     4 277 323
  9 232 241    2 243 250     7 377 393     4 508 527    9 393 411     1 366 365    -11,-12,L      6 275 291
 -15,-1,L      6 266 280     9 372 430     6 291 301   11 209 212     2 592 593    -4 288 315    12 200 168
  2 232 279    8 437 443    11 363 356    14 236 267    -12,-6,L      3 435 452    -2 246 224    13 168 192
 -15,0,L      10 328 343    -13,-4,L      -12,-12,L    -7 230 221     4 505 519     4 177 192    -11,-8,L
  4 257 289    -14,0,L      -1 327 377     0 181 229   -5 375 386     5 378 384     6 362 384    -7 382 358
  6 396 413    2 280 323     0 272 295    -12,-11,L    -3 453 461     6 536 536     8 456 437    -6 250 244
  8 350 364    4 558 602     1 399 397     0 166 153   -1 478 484     7 289 277    10 387 373    -5 423 425
 -14,-8,L      6 596 621     2 387 406     3 191 211    0 163 154     8 380 387    -11,-11,L     -4 249 253
  3 298 325    8 378 401     3 325 335     4 286 320    1 332 360     9 233 258    -4 235 220    -3 357 358
  5 323 307    -13,-11,L     4 301 302     6 436 435    7 254 247    10 286 268    -3 229 237    -1 234 231
  7 309 317    6 307 318     5 262 280     8 333 366    9 376 384    -12,-2,L      -2 357 361     3 179 170
 -14,-7,L      -13,-10,L    13 332 345    -12,-10,L    11 380 400    -8 205 200    -1 190 190     5 541 541
 -1 317 346    0 184 221    -13,-3,L      -4 342 357   13 495 488    -6 348 342     0 351 350     7 609 604
 -14,-6,L      1 204 219    -5 216 231    -3 255 246    -12,-5,L     -4 375 394     1 246 229     8 232 228
  1 366 388    4 173 202    -3 220 261    -2 268 268    1 478 505    -3 269 271     2 372 370     9 259 485
  3 359 360    -13,-9,L      1 151 157    -1 216 208    2 190 171    -2 478 475     3 231 208    11 415 390
  9 306 302    5 227 228     4 373 391     8 300 305    3 573 592    -1 254 290     4 380 355    13 335 336
 -14,-5,L      7 308 307     5 317 332     9 240 240    4 171 234     0 417 456     5 219 188    -11,-7,L
 -3 293 291    8 243 298     6 381 409    10 291 321    5 554 569     2 371 391     6 262 263    -5 183 178
 -1 300 326    9 321 358     7 273 298    11 208 240    6 206 139     8 279 273    -11,-10,L     -3 258 262
  9 323 348   10 197 214     8 280 308    -12,-9,L      7 449 460    10 508 504    -6 354 337    -1 315 336
 11 423 449    -13,-8,L      9 301 328     1 203 228    9 273 265    12 508 515    -5 226 220     0 163 138
 -14,-4,L     -1 441 462    10 276 294     3 236 278   10 247 238    13 316 296    -4 267 270     1 619 617
  1 296 349    1 383 407    11 272 288     5 380 394   11 209 182    -12,-1,L      -2 163 136     3 711 727
  2 217 197    3 293 311    -13,-2,L       6 359 365    -12,-4,L      0 342 345    -1 166 146     4 165 173
  3 286 281    -13,-7,L      0 325 342     7 366 386   -4 179 216     2 553 549     5 222 252     5 594 590
  4 311 315    5 294 317     1 248 254     8 258 296   -3 348 365     4 667 657     6 307 291     6 218 206
  5 237 249    7 388 405     2 509 531     9 253 258   -2 265 308     8 505 509     7 414 413     7 205 198
  7 262 288   11 422 436     3 212 240    -12,-8,L     -1 383 408    10 343 350     8 446 454     8 204 206
  9 187 222    -13,-6,L      4 478 511    -4 162 155    0 257 255    -12,0,L        9 391 361    13 328 310
 -14,-3,L     -3 295 321     6 208 191    -3 540 555    1 353 359    -6 382 373    10 377 384    15 416 412
  2 315 340   -1 491 508    13 157 156    -1 460 500    3 256 256    -4 446 424    11 253 287
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4

```
-11,-6,L      9  484  489  -4  287  293  -6  252  252  -3  413  418  -7  390  417   6  736  735   2  708  726
 -7  503  497 10  395  389  -2  744  731  -4  176  179  -2  454  437  -5  293  291   7  741  753   3  252  254
 -5  447  435 11  385  375  -1  185  208   0  238  228  -1  410  420   1  487  487   8  595  598   4  651  649
 -3  330  351 12  131  164   0  932  953   2  411  405   0  365  349   3  995 1000   9  495  501   5  415  372
 -1  302  293 13  385  369   1  244  244   3  245  211   1  612  629   5 1081 1064  10  222  205   6  768  776
  3  359  358 15  189  210   2  828  848   4  493  500   2  316  318   6  213  199  11  292  296   7  362  375
  5  694  675  -11,-3,L      3  174  123   6  445  439   3  323  317   7  713  716  13  206  214   8  812  806
  7  673  668 -4  150  165   4  514  524   8  386  390   7  272  283   8  245  244  15  139  148   9  206  222
  9  380  378 -3  302  301   5  175  139  10  204  256   9  355  349   9  427  430  16  144  121  10  467  458
 11  380  379 -2  379  382   6  532  524   -10,-11,L    10  180  203  15  183  210  17  272  278  11  220  241
 13  369  367 -1  438  426   8  250  232  -6  180  198  11  388  394   -10,-5,L      -10,-3,L     16  169  193
 -11,-5,L      0  499  499  10  208  225  -5  260  269  12  320  326  -9  166  179  -7  267  255   -10,-1,L
 -3  202  238  1  581  573  12  506  486  -4  375  380  13  455  437  -7  392  384  -6  256  242  -6  232  242
 -2  158  151  2  172  186  14  375  398  -3  232  247  14  290  266  -6  213  203  -5  458  452  -4  817  831
 -1  455  463  3  626  621  16  228  251  -2  431  422   -10,-8,L     -5  501  497  -4  566  570  -3  204  208
  0  263  260  4  430  434   -11,0,L      -1  290  308  -9  339  356  -4  264  275  -3  515  505  -2 1162 1147
  1  822  834  5  325  307 -10  323  335   0  449  453  -8  152  166  -3  482  478  -2  710  719  -1  312  310
  2  312  314  6  585  588  -8  409  394   1  211  235  -7  325  334  -2  254  253  -1  693  704   0 1022 1043
  3  905  912  8  416  418  -6  396  391   2  272  304  -6  213  194  -1  562  560   0  416  407   1  276  251
  5  617  626 12  445  439  -4  473  479   7  178  172   1  435  450   0  203  237   1  890  897   2  388  371
  6  195  198 13  162  172  -2  395  386   8  207  189   2  260  269   1  748  748   2  210  212   3  325  313
  8  262  234 14  453  443   6  300  310  10  367  369   3  776  777   3  457  443   3  557  557   4  152  120
 13  252  264 15  282  276   8  777  792  12  447  457   4  320  310   6  177  188   4  274  277   7  324  325
 14  150  144 16  274  273  10 1043 1051  13  161  185   5  812  826   7  360  368   7  406  396   8  494  510
 15  459  453  -11,-2,L     12  667  654   -10,-10,L     6  302  291   9  445  456   8  265  276   9  253  242
 -11,-4,L     -8  346  341  14  277  264  -8  272  263   7  541  541  10  318  311   9  501  499  10  893  910
 -9  300  316 -6  423  419   -10,-15,L    -6  254  254   8  255  242  11  433  432  10  444  427  11  200  203
 -8  225  225 -4  502  494   0  279  254   0  269  281   9  214  223  12  164  167  11  390  393  12  715  736
 -7  356  352 -3  200  203   2  226  191   1  262  268  10  239  244  13  518  522  12  446  449  14  550  561
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 -3  252  276  4  282  279   4  381  392   5  427  440  -3  344  354  -9  349  360  16  266  278  -8  254  247
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  5  560  550 11  301  309   2  210  245   -10,-9,L     13  556  557   2  544  554  -6  417  423   6  683  680
  6  455  446 12  459  454   8  206  213  -7  310  291  15  529  514   3  756  757  -4  307  302   8  983  992
  7  642  646 13  243  259  10  353  314  -5  383  386   -10,-6,L      4  536  542  -3  183  175  10  536  534
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  -9,-16,L       -9,-11,L      -3  206  207    0  193  192    7 1024 1012    4  422  430   -2  703  705    5  392  352
  0  311  276   -8  319  274   -2  269  266    2  298  286    8  370  370    5  570  578    0  206  197    6  497  486
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 -3,-10,L      4  623  618   -2  246  257  -10  429  430    5 1229 1230   14  268  285  -11  359  357    -2,-19,L
-10  173  153    5  601  609   -1 1160 1196   -9  691  701    6  520  508   15  449  448  -10  594  577   -3  320  344
 -9  484  466    6  460  480    0  724  705   -8  511  525    7  990 1009   16  387  380   -8  888  879   -1  372  373
 -8  441  413    7  421  418    1 2223 2236   -7  490  493    8  867  868   17  431  433   -7  187  168    1  402  418
 -7  470  468    8  310  313    2  359  345   -6  496  490    9  628  624   18  327  331   -6  633  627    3  362  334
 -5  626  622    9  505  527    3 2187 2229   -4  489  487   10  980  990   19  297  303   -5  374  358    -2,-18,L
 -4 1048 1030   11  242  246    5 1144 1193   -3  386  387   11  941  932     -3,-2,L      -4  286  304   -5  334  332
 -3  686  679   12  269  283    7  864  885   -2 1152 1146   12  753  742  -16  261  270   -3  429  418    3  361  335
 -2  948  920   14  170  268    8  282  290   -1 1205 1220   13  665  664  -15  200  202   -2 1404 1409    5  482  476
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  0  646  643   17  303  327   12  258  266    1 2071 2108   15  518  534  -12  149  157    0  697  666    7  359  369
  1  428  427     -3,-8,L     13  314  278    2  822  816   16  347  377  -10  431  433    1  863  827    -2,-17,L
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  5  483  485  -11  325  314   15  425  438    4  600  619     -3,-3,L      -8  776  776    4 2153 2177   -6  243  245
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  -2,-17,L     -6  769  792   4  885  892  -2  334  320 -13  359  373   6  627  639  -3 1124 1128  12  380  389
  -5  307  296  -4  643  664   5  328  322   0  391  393 -12  192  196   8  217  214  -2 1373 1371  13  374  379
  -4  347  319  -2  247  250   6  845  859   1  371  382 -11  763  765   9  575  572  -1 1884 1897  14  230  234
  -3  271  302   0  273  285   7  228  225   2  217  218 -10  358  352  10  226  221   0  747  752    -2,-3,L
  -2  316  334   2  626  634   8 1064 1081   3  683  683  -9  821  822  11  613  610   1 2115 2128 -18  277  282
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   1  464  474   6  734  734  12  266  284   6  836  852  -6  479  482  15  572  594   4  555  555 -15  370  379
   2  356  334   7  184  197    -2,-11,L     7  804  819  -5  787  790  17  479  483   5 1461 1460 -14  255  246
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  -8  409  409  -9  157  178  -6  369  375  12  450  458   2  546  555  -8  255  268  13  786  794  -8  153  168
  -7  464  489  -6  229  213  -5  208  195    -2,-9,L     3  863  863  -7 1209 1204  14  335  327  -7  370  368
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  -2,-14,L     -1  313  309  -6  678  695  13  414  404   1 1895 1905  -7  498  495   8  662  686    -2,-2,L
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  -9  558  552   5  245  242  -2  300  281  -6  385  377   5  370  380  -1  921  944  -8  394  379   -1,-6,L
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  -6 1668 1680  10  751  739   1  358  355   2  894  921   8  680  688   2  714  740  -5  205  201 -13  735  733
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  -3  700  679  14  888  882   6  191  163   5  210  208   -1,-11,L     5  942  948  -2  192  188 -10  283  287
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   4 1695 1680  -6 2091 2077  -8  243  220  -8  409  417  -4 1064 1092   -1,-9,L      5 1455 1489  -1  979 1002
   5 1008 1001  -4 1583 1543   0  468  461  -6  620  629  -3  489  501 -10  184  196   6  484  502   0  242  229
   6 2096 2097  -2  402  391   1  411  430  -4  973  992  -2 1486 1516  -9  245  258   7 1152 1180   1  807  775
   7 1261 1236   0  667  640   2  555  545  -2  952  968  -1  312  313  -8  364  383   8  835  848   2  495  483
   8 1747 1750   2  275  267   3  701  680  -1  171  153   0  895  923  -7  447  439   9  574  589   3 2083 2131
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 -12  489  482   -1,-18,L    -4  526  521 -12  597  630   -1,-10,L     7  862  868  -3 1978 2008   -1,-5,L
 -10  545  542  -1  240  246  -2  495  487 -10  594  606 -14  505  521   8  354  347  -1 1519 1543 -17  362  373
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  -2 2476 2515   -1,-17,L     8  373  368  -2  674  687  -8  316  314  14  251  241   6  202  208  -8  715  707
  -1 1332 1292  -8  234  277  10  453  463  -1  433  427  -7  523  536  15  366  385   7 1498 1507  -7 1678 1711
   0 2206 2204  -7  394  405  12  398  399   0  919  937  -6  391  383   -1,-8,L      9 1308 1311  -6  821  825
   1 1495 1509  -6  285  309   -1,-14,L     1  354  368  -5  221  236 -15  486  498  11  994 1016  -5 1697 1770
   2 2352 2426  -5  386  393 -12  371  391   2 1003 1015  -4  126  123 -13  727  734  13  561  563  -4  951  941
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  -1,-5,L         5 2506 2592     -1,-2,L        -2 2160 2206     7  392  379    10  280  288    10  419  420     7  179  183
-3 1527 1588      6 1153 1177  -16  346  347     -1  780  773     8  266  250    12  423  424    11  486  496       0,-3,L
-2  741  748      7 1053 1069  -14  600  611      0 1898 1992     9  342  355    14  465  483    12  247  260     1  857  870
-1 1268 1289      8  886  887  -13  533  542      2  308  291       0,-16,L        0,-11,L       13  343  372     2 2858 2962
 0 1028 1032      9  227  221  -12  855  842      3  520  503     0  547  544     2  555  570       0,-6,L        3  122  110
 1 1129 1093     10  497  495  -11  597  587      4  208  197     1  385  399     3  430  417     0 1082 1112     4 2445 2492
 2  252  241     14  443  438  -10  981  992      5  270  278     2  548  545     4  643  638     1 1948 2023     5  674  667
 3  131  135     15  214  227   -9  827  831      6 1048 1009     3  210  244     5  452  467     2  193  196     6 1745 1766
 4  502  480     16  353  359   -8  894  911      7  491  478     4  350  352     6  729  740     3 1866 1971     7 1524 1521
 5  449  444     17  351  376   -7  449  454      8  992  983     6  212  197     7  424  428     4  170  164     8 1139 1158
 6  515  538       -1,-3,L      -6  188  197      9  338  340    11  220  232     8  628  642     5 1274 1358     9 1056 1056
 7 1699 1687    -18  329  346   -5  402  394     10 1539 1540       0,-15,L       9  344  354     7  223  223    10  961  970
 8  150  141    -15  226  234   -4  562  550     11  650  647     2  285  291    10  574  584     8  143  142    11  546  534
 9 1080 1089    -14  209  235   -3  881  860     12 1617 1637     3  267  263    11  242  239     9  273  281    12  262  249
10  256  254    -12  164  150   -2 1102 1079     14  931  933     4  446  455    12  426  436    10  133  162    13  356  361
11  826  820    -11  325  323   -1  980  961        -1,0,L        6  567  588    13  266  273    11  365  378       0,-2,L
12  362  373    -10  400  399    0 2417 2489    -16  438  435     7  308  318       0,-10,L       12  167  148     0 2086 2157
13  598  607     -9  594  605    1  610  593    -14  717  722     8  625  628     0 1247 1280    13  497  517     1  584  579
14  270  270     -8 1400 1409    2 2011 2083    -12 1258 1256     9  265  254     1  941  966    15  584  597     2 2726 2838
15  512  510     -7 1178 1183    3 1408 1446    -10 1496 1485    10  434  448     2 1045 1064    17  463  471     3 1202 1201
17  316  322     -6 1300 1315    4 2844 2913     -8 1526 1489    12  239  264     3 1032 1064       0,-5,L        4  746  731
  -1,-4,L        -5 1232 1219    5 1396 1402     -6  132  118       0,-14,L       4  634  629     1  933  918     5  760  762
-16  277  285    -4 1754 1751    6 1318 1323     -4  481  479     0  856  875     5  626  630     2 1002 1021     6  221  233
-15  375  388    -3 1693 1682    7  624  627     -2  691  644     1  195  173     7  138  148     3  329  328     7  327  308
-14  280  284    -2 1736 1754    8  936  950      0 1974 2066     2  830  847     8  191  212     4 1339 1369     8  379  383
-13  514  525    -1 1355 1363   10  814  816      2 3789 4277     4  630  641     9  272  270     5 1706 1753     9  538  551
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-11  930  935     1 1551 1535   14  552  548      6 1509 1528    12  312  314    11  233  249     7 1916 1966    11  605  621
-10  707  718     2  230  232   16  541  540      8  974  936       0,-13,L      12  421  420     8  542  547    12  756  764
 -9 1136 1139     3 1525 1544   18  307  344     10  742  727     2  456  470    13  304  310     9 1486 1500    13  342  351
 -8  465  459     4 1108 1131      -1,-1,L       14  499  520     4  922  921    14  477  474    10  325  317    14  858  876
 -7  990  982     5  148  170  -18  372  365     16  468  464     6  834  846    16  325  341    11  805  828    15  301  318
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 -5  650  644     7  467  488  -14  263  282       0,-18,L       10  524  542     1  311  301    13  321  338    17  193  229
 -3  406  394     8  331  334  -11  271  260      0  239  223    12  363  398     2  452  445       0,-4,L       18  303  336
 -2  463  468     9 1048 1055  -10  522  501      1  478  489       0,-12,L       3  537  533     0  498  527       0,-1,L
 -1  365  379    10  844  854   -9  313  296      3  383  395     0 1206 1258     4  740  744     1 2126 2190     2 1410 1465
  0  650  659    11  512  515   -8 1270 1255       0,-17,L        1  273  272     5 1244 1286     2  520  496     3  457  463
  1 2621 2726    12  764  763   -6 1773 1792      3  271  278     2 1038 1077     6  786  792     3 1547 1616     4  823  847
  2 1466 1486    13  569  571   -5  891  872      4  144  147     4  658  687     7 1278 1301     4  409  428     5  217  227
  3 2455 2547    14  564  555   -4 1724 1715      5  338  369     6  341  358     8  569  580     5 1174 1187
  4 1279 1335    15  396  415   -3  185  191      6  268  274     7  224  229     9  607  617     6 1034 1057
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Compound:

$\underline{\text{cis}}$-$Pt(CF_3)_2(PMe_2C_6F_5)_2$

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  2  545  536   -15,3,L      9  361  370   9  173  166  19  277  275  14  530  530   0  350  343  13  896  893
  4  506  499  1  331  312  10  401  390  10  684  696  20  282  269  15  396  407   1  257  239  15  742  740
  6  368  341  3  356  344  11  476  449  12  465  475   -13,7,L      16  439  437   2  231  261  17  672  686
 10  192  218  5  348  338  12  515  508  14  459  466   0  332  340  17  438  431   6  215  189  19  405  409
 12  291  284  7  461  442  13  328  313  16  361  361   2  284  291  18  242  245   9  245  239  21  289  296
 14  284  296  9  536  520  14  351  343  17  251  256   3  156  133  19  382  381  10  239  254  23  301  281
 16  357  360 11  429  427  15  347  337  18  441  461   4  321  308  21  258  270  12  297  301  25  300  292
 18  292  319 13  471  446  17  342  330  19  288  270   5  238  227  23  300  297   -11,1,L      27  266  265
 -16,2,L      15  339  326  19  247  269  20  278  217   6  269  255  25  245  221   0  845  844   -11,5,L
  1  312  304 17  162  167  21  219  227  21  331  339   7  145  166  26  228  229   1  259  230   0  484  471
  3  225  239   -15,5,L      -14,4,L      22  240  242   8  322  326   -12,4,L       2 1026 1029   1  352  349
  5  287  292  0  310  288   1  455  426  23  203  231  10  303  292   0  425  396   3  795  791   2  542  514
  6  253  222  1  314  317   3  535  518  24  269  283  12  406  392   1  749  743   4  824  815   3  495  497
  7  294  296  2  349  331   5  442  435   -13,3,L      14  336  337   3  304  298   5  883  908   4  549  532
  8  274  276  3  376  362   7  430  422   1  522  531   -12,0,L       4  243  254   6  700  703   5  453  453
  9  292  343  4  363  334   9  332  336   2  182  139   0  747  764   5  873  862   7  509  512   6  825  815
 10  218  313  5  260  267  10  240  226   3  501  491   2  783  804   7  789  782   8  610  608   7  610  594
 13  272  278  7  275  222  11  367  358   4  151  166   4  870  892   9  602  591   9  611  597   8  627  614
 -16,4,L       9  209  196  12  284  284   5  448  457   6 1019 1040  10  331  335  10  973  979   9  414  417
  1  265  273 10  235  247  13  339  325   6  146  132   8  733  724  11  404  389  11  204  189  10  376  370
  3  349  337   -14,0,L     14  227  194   7  494  471  10  722  715  12  369  354  12  923  917  11  367  374
  5  336  334  0  641  632  15  257  289   9  662  654  12  764  772  13  451  436  13  312  310  12  342  342
 11  255  259  2  604  589  17  227  265  11  770  740  14  618  624  14  325  338  14  468  460  13  178  176
 13  226  234  4  755  743  21  288  275  13  657  653  16  428  445  15  514  501  16  733  732  14  341  356
 -15,1,L       6  692  685   -14,6,L      15  546  541  18  282  258  16  253  244  18  589  588  16  319  329
  0  387  379  8  711  712   0  370  374  17  378  395  20  869  882  17  350  339  19  440  423  17  354  346
  1  292  331 10  295  280   2  460  463  19  251  255  22  422  439  19  355  357  20  267  265  18  301  285
  2  400  392 12  387  381   4  382  381  21  213  229  24  287  286  21  346  340  21  461  455  19  236  224
  3  323  325 14  423  420   6  476  453  23  272  260  26  239  232   -12,6,L      22  351  386  20  329  341
  4  344  329 16  472  470  10  288  292   -13,5,L      -12,2,L       0  429  416  23  281  291  21  282  277
  5  232  309 18  467  465  12  298  292   0  406  394   0  503  504   2  499  483  24  286  293  22  220  228
  6  401  397 20  376  383  13  143  125   1  302  294   1  820  813   4  628  628  28  183  179  24  218  221
  7  221  225 22  350  323  14  255  248   2  514  493   2  510  521   6  593  574   -11,3,L      -11,7,L
  8  372  374   -14,2,L      -13,1,L       3  452  456   3  783  780   8  553  544   0  236  224   0  527  517
 10  419  423  0  306  304   0  568  572   4  429  420   5  527  528  10  468  460   1  644  635   2  629  616
 12  339  333  1  405  389   1  436  418   5  443  434   6  310  310  11  191  205   3  810  800   4  429  460
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 -11,7,L           14   937   945   14   391   386   18   825   819    7   881   869    9   377   366   20   510   515   14   501   500
  6   365   371    15   924   939   15   252   268   19   155   151    8   819   802   11   300   294   21   436   435   15   389   378
  7   278   267    16   449   457   16   351   351   20   528   507    9   469   450   12   199   179   22   191   194   16   411   427
  8   360   360    17   490   484   18   351   349   21   334   325   10   822   805   13   278   311   23   602   588   17   265   227
 10   476   463    18   405   402   20   357   359   22   397   404   11   572   537   15   333   370   25   314   308   20   343   368
 12   528   522    19   444   458   22   373   394   23   319   326   12   679   669    -8,0,L          27   274   286   22   386   370
 14   493   479    20   275   270    -10,8,L          24   441   435   13   404   385    0   705   715    -8,4,L          24   393   380
 16   312   311    21   406   392    0   458   441   25   410   402   14   576   545    2  1655  1656    0  1004   992    -8,8,L
 18   276   302    23   400   401    1   427   423   26   346   334   15   338   328    4  1917  1942    1  1022  1020    0   519   502
 -11,9,L           25   274   295    2   383   376   27   178   184   16   287   309    6  1133  1180    2   738   732    1   602   570
  3   202   273     -10,4,L           3   416   399   28   236   229   17   245   229    8  2200  2263    3  1262  1246    2   609   594
  5   281   290     0   411   399    4   325   325    -9,3,L          18   345   348   10  1533  1589    4   833   813    3   565   548
  7   304   277     1   776   773    5   269   274    0   220   194   19   349   330   12   953   950    5  1021   999    4   548   549
 -10,0,L            2   565   545    6   257   257    1  1387  1379   20   381   413   14   955   955    7   921   948    5   497   474
  0   767   779     3   903   898    7   200   190    2   162   174   21   295   302   16   775   784    8   346   361    6   385   383
  2  1301  1317     4   279   277    9   212   235    3  1231  1234   22   293   318   18   606   613    9  1564  1538    7   353   338
  4   765   788     5   998  1000   10   323   310    4   234   239   23   284   293   20   557   569   10   228   223    8   410   400
  6  1222  1258     7   897   904   11   314   319    5  1144  1134   24   234   264   22   647   642   11   840   820    9   256   243
  8  1286  1311     8   184   167   12   240   245    6   275   254   25   262   241   24   600   607   12   271   275   10   338   335
 10  1104  1121     9   911   898   13   283   312    7  1058  1049   26   238   244   26   540   527   13   816   810   11   250   236
 12   857   872    11   725   718   14   266   273    8   265   256    -9,7,L          28   350   352   14   464   458   12   250   237
 14   757   769    12   531   538   15   275   281    9   515   494    0   784   760   30   251   202   15   581   575   13   356   367
 16   396   394    13   733   742   16   344   343   10   756   754    2   666   653    -8,2,L          16   472   464   14   377   372
 18   649   650    14   396   374   17   305   275   11  1126  1137    3   249   236    0  1629  1644   17   671   671   15   338   347
 20   663   657    15   508   506    -9,1,L          12   318   313    4   664   643    1  1074  1082   18   535   528   16   405   381
 22   548   538    16   442   446    0  1256  1295   13   912   905    6   633   622    2  1567  1577   19   538   520   17   345   384
 24   474   473    17   496   499    1   183   178   14   301   293    7   266   280    3   844   839   20   263   268   18   355   343
 26   396   392    18   321   323    2  1015  1027   15   865   866    8   568   566    4   793   813   21   551   549   19   299   320
 28   350   327    19   458   458    3   134   123   17   724   719    9   217   218    5  1238  1254   23   566   585    -8,10,L
 -10,2,L           21   509   497    4  1255  1284   19   656   667   10   477   476    6   173   160   25   334   347    1   268   273
  0  1186  1197    23   374   361    5  1194  1221   21   547   545   11   238   214    7  1534  1544   27   238   262    3   293   312
  1   821   831    25   289   309    6  1069  1078   23   304   307   12   597   605    8   626   612    -8,6,L           5   390   417
  2   810   811    27   249   210    7   946   973   24   142   129   14   576   561    9   870   899    0   824   823    7   406   413
  3   878   871     -10,6,L           8   943   955   25   267   255   16   485   481   10   330   331    1   234   237    9   393   426
  4   468   479     0   537   525    9   834   849   27   291   294   18   432   454   11   906   920    2   525   515    -7,1,L
  5   919   920     1   319   307   10   887   913    -9,5,L          20   354   354   12   723   746    3   322   322    0  1989  2006
  7  1072  1102     2   566   549   11   596   588    0   692   669   22   264   261   13   887   890    4   929   910    1   606   579
  8   329   351     4   741   730   12  1064  1088    1   444   445    -9,9,L          14   629   636    5   258   250    2  1254  1330
  9  1074  1064     6   765   747   13   207   196    2   831   813    1   436   433   15  1042  1053    6   883   872    3   275   271
 10   387   384     8   759   762   14   858   855    3   543   559    3   413   421   16   833   832    8  1020  1002    4  1605  1642
 11   588   590    10   775   759   15   142   151    4   696   699    5   374   392   17   657   675   10  1060  1038    5   266   266
 12   301   298    12   466   471   16   944   935    5   565   559    7   424   400   18   589   582   12   858   857    6  1484  1490
 13   512   501    13   262   257   17   260   275    6   575   561    8   219   223   19   588   589   13   150   151    7   800   806
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   -7,1,L       27  237  279   14  560  576    7  952  955   21  582  609   14  335  366   25  283  318   14 1263 1287
  8  997 1046   29  203  236   16  525  542    8  656  641   22  187  193   15  330  334   26  351  351   15  880  860
  9 1139 1158      -7,5,L      18  485  487    9 1250 1270   23  536  546   17  308  320   27  395  373   16  424  429
 10 1152 1170    0  827  823   20  539  558   10  347  351   25  454  474   18  384  379   28  360  343   17  247  215
 11 1144 1144    1  599  608   22  295  278   11 1259 1276   27  326  331   19  345  400   29  271  265   18  551  558
 12 1224 1231    2 1011 1000   23  156  162   12  520  510   29  264  280   20  259  286   30  298  300   19  281  279
 13 1068 1071    3  252  273      -7,9,L      13 1090 1109      -6,6,L      21  244  315      -5,3,L      20  318  338
 14  822  840    4  208  185    1  640  650   14  737  747    0  807  812   22  293  296    0  250  243   21  419  409
 15  318  319    5 1083 1110    3  605  596   15  751  755    1  445  454      -6,10,L      1 1992 2005   22  337  340
 16 1021 1021    6  998 1004    5  577  572   16 1097 1117    2  949  935    0  204  187    2  272  278   23  302  313
 17  172  168    7  964  962    7  497  491   17  961  980    3  791  788    1  393  408    3 2403 2484   24  298  295
 18  850  867    8  821  826    8  251  234   18  967  962    4 1063 1058    3  411  431    4  192  136   25  323  318
 20  753  775    9  716  729    9  453  452   19  599  608    5  130  110    5  310  329    5 2582 2662   26  330  329
 21  404  403   10  822  836   10  248  236   20  443  450    6  834  849    7  468  454    7 2452 2499   27  241  235
 22  515  519   11  640  641   11  350  352   21  651  664    7  190  183    9  456  474    9 1137 1144   28  277  253
 23  250  258   12  744  746   13  340  360   22  274  286    8 1205 1199   11  445  461   10  222  223      -5,7,L
 24  487  474   13  770  782   15  382  420   23  551  559   10  991 1014   13  380  408   11 1042 1040    0 1358 1360
 25  334  354   14  763  789   17  360  376   24  305  314   11  199  197      -5,1,L      12  208  200    1  156  149
 26  361  379   15  546  531      -6,0,L      25  307  337   12 1225 1244    0 2736 2807   13  948  951    2 1255 1245
 27  253  252   16  662  689    0 1540 1565   26  144  141   14  726  732    1  619  593   14  533  534    4 1097 1104
 30  226  257   17  237  239    2 3168 3283   27  300  304   16  724  741    2 2065 2099   15 1259 1267    5  194  190
   -7,3,L       18  321  312    4  955 1012   29  229  239   17  317  318    3  523  516   17  978  990    6  844  836
  0  106  104   19  289  316    6 1997 2082      -6,4,L      18  481  507    4 1750 1738   19 1094 1115    8  881  880
  1 1835 1872   20  356  360    8 1794 1821    0  715  726   19  280  284    5  854  852   21  945  949    9  491  481
  2  122  125   21  324  335   10 3331 3479    1  355  359   20  283  309    6 1492 1536   23  687  698   10  779  773
  3 1657 1670   22  345  352   12 1401 1410    2 1078 1084   22  395  414    7  731  731   25  417  421   11  507  521
  4  540  545   23  324  327   14 1239 1256    3 1263 1266   24  398  424    8 1931 1989   27  296  300   12  503  505
  5 2167 2205   24  290  325   16  476  496    4  909  904   26  324  334    9 1086 1076   29  256  259   13  199  182
  6  262  248   25  288  303   18 1107 1111    5 1805 1806      -6,8,L      10 1712 1746      -5,5,L      14  599  598
  7 1573 1557   27  210  209   20  712  733    6  552  542    0  615  607   11 1593 1610    0 1196 1202   16  513  503
  8  114  135      -7,7,L      22  642  654    7 1619 1651    1  698  707   12  871  885    1  710  711   18  692  698
  9 1233 1218    0  764  765   24  604  608    8  204  185    2  675  684   13 1318 1331    2  651  699   20  499  508
 10  463  472    2 1055 1062   26  583  598    9 1240 1236    3  636  634   14 1157 1167    3  203  205   22  373  362
 11  830  836    3  212  212   28  508  523   11  935  926    4  633  626   15  663  677    4 1004 1001   24  268  282
 12  203  195    4  747  749   30  270  271   12  273  268    5  599  602   16 1165 1183    5  729  730      -5,9,L
 13 1055 1087    5  235  228      -6,2,L      13  980  975    6  567  568   17  290  348    6 1871 1871    0  284  302
 14  185  183    6  780  791    0  990 1032   14  348  337    7  504  513   18  762  770    7 1063 1062    1  566  574
 15 1017 1021    7  193  187    1 1145 1179   15  843  861    8  440  460   19  353  338    8  695  700    3  749  738
 17  880  885    8  561  566    2 1826 1852   16  486  492    9  340  347   20  864  855    9  935  941    5  737  738
 19 1001 1007    9  442  442    3 1820 1849   17  729  720   10  542  528   21  201  183   10  882  869    7  633  631
 21  698  696   10  594  613    4 1299 1313   18  632  647   11  254  314   22  569  575   11  916  947    8  319  295
 23  392  400   11  256  272    5 1074 1077   19  369  368   12  410  392   23  179  193   12  838  833    9  536  539
 25  380  378   12  619  632    6  709  717   20  350  362   13  311  306   24  545  552   13  992 1001   10  311  320
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   -5,9,L        11   812   840   25   443   423   16   394   395   26   330   305   13  1478  1452    7   758   729    8  1343  1371
11   439   438   12   953   913   27   419   388   17   297   298   27   306   273   14   770   735    8   363   352    9  1381  1359
12   226   231   13  1424  1416   29   260   281   18   438   409   28   447   463   15   756   730    9   520   522   10  1432  1439
13   375   387   14   249   253      -4,6,L        19   342   368   29   193   212   16   674   682   10   305   274   11  1649  1616
14   207   190   15  1149  1140    0  1653  1643   20   302   305      -3,3,L        17   463   462   11   487   496   12   719   720
15   412   406   16   773   772    1   290   286   21   270   282    0  1428  1453   18   723   726   12   357   337   13  1651  1630
17   376   400   17   827   824    2  1282  1271   22   264   287    1  2500  2542   19   262   253   13   404   424   14   166   162
19   367   361   18   861   859    3   356   359      -4,10,L        2   790   805   20   469   477   14   320   290   15   828   817
  -5,11,L        19   767   766    4  1194  1176    1   656   671    3  2427  2492   21   318   322   15   384   396   16   433   415
 0   198   200   20   788   766    5   690   669    3   437   445    4   241   230   22   317   285   17   378   394   17   759   760
 1   244   261   21   694   695    6   757   746    4   165   154    5  2813  2837   23   320   304   19   350   360   18   498   494
 3   297   310   22   505   502    7   306   282    5   471   460    6   946   904   24   256   258      -3,11,L       19   720   726
 5   261   300   23   671   681    8  1032  1006    7   475   468    7  1785  1804   25   210   171    1   361   366   20   910   903
 7   256   246   24   350   343    9   172   165    9   492   490    8   310   310   26   307   324    2   213   225   21  1148  1164
   -4,0,L        25   319   394   10  1117  1135   11   440   450    9  2392  2359   27   343   316    3   337   342   22   332   320
 0  2394  2495   26   227   251   11   249   219   13   443   442   10  1331  1281   28   250   244    7   246   257   23   485   476
 2  1528  1529   27   249   260   12  1077  1084   15   427   414   11   697   683      -3,7,L         9   270   287   24   562   556
 4  1581  1631   29   321   294   14   934   939      -3,1,L        12   513   489    0  1151  1139      -2,0,L        25   509   510
 6  1828  1892      -4,4,L        16   941   938    0  2269  2354   13  1041  1014    1   323   306    0  3737  4320   26   325   322
 8  1905  1991    0   473   463   17   273   266    1   300   305   15   876   860    2  1361  1366    2  3719  4165   27   352   354
10  2150  2218    1  1073  1087   18   540   549    2  3004  3143   16   224   198    4  1397  1408    4   612   596   28   176   146
12  2542  2551    2   953   931   20   566   551    3   976   926   17   913   903    6  1220  1208    6  1357  1342   29   271   286
14  1144  1140    3  1984  1982   21   194   166    4  2301  2273   18   311   301    7   146   125    8  1047  1055      -2,4,L
16  1532  1525    4   793   775   22   404   388    5  1148  1123   19  1220  1221    8  1252  1233   10  1445  1440    0   156   120
18  1307  1310    5  1509  1548   24   378   392    6  1793  1791   21  1047  1030    9   510   522   12  2066  2098    1  1557  1582
20   457   451    6   818   821   26   308   323    7   271   266   23   852   846   10   717   706   14  2284  2279    2   628   595
22   576   567    7  1805  1808      -4,8,L         8  2837  2906   25   445   445   11   471   454   16  1703  1696    3  1914  1923
24   546   538    8   429   434    0   359   352    9   765   717   27   369   374   12   632   636   18  1454  1409    4   847   831
26   963   965    9   925   896    1   677   660   10  1595  1596   29   239   267   13   429   419   20   706   684    5  1375  1419
28   323   334   10   704   690    2   589   586   11  1141  1132      -3,5,L        14   587   591   22   261   254    6   196   233
30   378   369   11  2282  2272    3   645   634   12  1394  1408    0  1867  1860   15   182   189   24   760   726    7  1496  1496
   -4,2,L        12   256   245    4   687   676   13   995  1002    1   916   925   16   733   735   26   538   528    8  1478  1461
 0  1852  1873   13  1200  1173    5   633   606   14  1502  1500    2  1453  1407   18   632   611   28   414   397    9  1355  1388
 1  1814  1819   14   204   180    6   787   787   15  1451  1448    3   277   265   19   137    51   30   447   430   10   817   815
 2  1151  1166   15   793   799    7   843   845   16  1539  1532    4   908   902   20   582   555      -2,2,L        11  1706  1742
 3  1890  1885   16   483   497    8   522   508   17   374   375    5   502   460   22   372   388    0  1231  1212   12   285   290
 4  1867  1895   17   756   756    9   573   565   18   879   879    6   797   778   24   399   396    1  1734  1722   13  1466  1472
 5  2897  2929   18   405   389   10   507   478   19   304   305    7   599   575      -3,9,L         2  1371  1372   14   205   217
 6   569   560   19   722   725   11   424   399   20   652   658    8   936   910    0   269   276    3  1173  1188   15  1293  1293
 7  1797  1753   20   474   484   12   506   480   22   836   822    9  1139  1112    1   478   484    4   408   383   16   247   235
 8  1422  1411   21   576   570   13   246   271   23   318   288   10  1310  1282    2   214   179    5  4783  5025   17  1013  1003
 9  1281  1286   22   334   326   14   408   390   24   586   576   11   788   773    3   816   799    6  3462  3465   18   379   397
10   511   544   23   435   432   15   306   314   25   368   371   12  1317  1331    5   931   896    7  2094  2073   19   874   890
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   -2,4,L       8   606   586  19   357   349   8  1013   992   1   672   669   0   834   804  12   431   423   9   799   810
20   249   258   9   816   812  20   705   683   9   708   710   2   285   289   1   683   643  13  1395  1410  10   721   717
21   569   571  10   641   641  21   266   247  10   887   870   3   815   792   2   229   194  15  1743  1752  11   591   605
22   417   407  11   492   486  22   828   813  11   933   923   5   733   737   3  2221  2187  16   508   506  12   466   459
23   456   456  12   431   423  23   259   245  12   961   966   7   800   780   4  1780  1719  17   604   585  13   342   339
24   289   271  13   342   349  24   528   526  13  1272  1246   9   649   652   5  1689  1639  19   991   982  14   387   401
25   436   429  14   387   383  26   446   469  14  1185  1202  10   243   237   6  3131  3117  20   279   273  15   299   302
29   307   310  15   315   324  27   276   250  15   774   742  11   586   594   7  2559  2607  21   668   665  16   309   303
   -2,6,L      16   363   367  28   364   372  16   786   785  12   260   223   8  2273  2364  22   300   318  17   251   258
 0  1769  1744  17   275   281  29   236   240  17   651   653  13   414   418   9  2098  2101  23   548   557  18   245   259
 1   122    67  18   298   303  30   315   302  18   678   717  14   283   264  10  1315  1315  24   294   313  19   216   282
 2  1759  1766  19   275   276     -1,3,L      19   361   365  15   393   384  11  1868  1859  25   329   331  20   322   352
 3   222   220  20   300   303   0   400   388  20   436   453  16   224   199  12   969   986  27   339   336  21   241   276
 4  1127  1116  22   322   324   1  1395  1364  21   345   360  17   311   320  13  1429  1414     0,6,L      22   203   263
 5   814   801     -2,10,L      2   551   538  22   371   365  18   232   227  14   911   937   0  1482  1462     0,10,L
 6  1274  1267   1   751   759   3  3409  3468  23   159   156  19   357   374  15   550   543   1   261   242   1   727   736
 7   468   460   3   548   539   5  2648  2615  24   326   322     -1,11,L     16   141   118   2  1969  1955   3   589   605
 8   776   745   5   533   519   7  2902  2864  25   187   189   0   389   386  17   682   670   3   158   125   5   498   503
 9   278   279   7   456   465   8  1313  1283  26   264   288   1   351   351  18   505   452   4  1575  1536   7   413   427
10  1076  1062   9   447   476   9  1750  1740  27   171   207   2   307   316  19   987   999   5   367   353   9   392   418
11   278   256  11   443   431  11  1446  1475     -1,7,L       3   406   416  20   330   334   6  1154  1137  11   402   443
12   933   933  13   565   524  13  1155  1163   0  1122  1116   4   218   228  21   772   771   7   432   424  13   370   412
13   317   310  15   423   439  14   155   160   1   413   413   5   358   352  22   568   550   8   863   848  15   448   463
14   991  1006     -1,1,L      15   726   701   2  1321  1316   7   306   311  23   586   578   9   611   598     1,1,L
16   933   929   0  1778  1785  16   801   724   4  1039  1026   9   251   287  24   567   545  10  1049  1057   1  1161  1093
18   669   671   1  2075  2016  17  1051  1073   5   347   344  10   197   219  25   510   503  11   376   366   2  3536  3642
19   214   212   2  2978  3073  19   763   751   6  1266  1266     0,0,L      26   416   400  12   988   975   3  1149  1088
20   555   560   3  1632  1559  20   232   235   8  1218  1204   2  2462  2508  27   418   400  14  1060  1063   4  1529  1475
21   231   205   4  2742  2664  21   959   962   9   176   195   4  2154  2052  28   155   141  16   993   992   5  1513  1468
22   381   395   5   907   833  22   290   274  10  1062  1055   6  1085  1006  29   339   347  18   722   738   6  1753  1734
23   152   144   6   617   600  23   829   823  11   228   221   8  2346  2227     0,4,L      20   488   506   7  1539  1512
24   297   305   7  2233  2173  25   593   579  12   793   789  10  1268  1209   0   242   221  22   376   409   8  1336  1343
25   180   147   8  2182  2108  27   375   388  13   465   459  12  1129  1118   1  2129  2102  26   246   276   9  1311  1308
26   336   334   9   841   798  29   234   244  14   589   591  14  2348  2299   2   320   295     0,8,L      10  1865  1838
   -2,8,L      10  2618  2584     -1,5,L      15   301   302  16  2121  2056   3  1784  1817   0   816   815  11   588   596
 0   515   509  11  1825  1784   0  1691  1670  16   558   571  18  1637  1589   4   957   933   1   503   482  12  1308  1297
 1   465   467  12  1097  1069   1   627   628  17   275   243  20   973   970   5  2192  2167   2   536   535  13   843   800
 2   564   554  13   555   560   2  1088  1083  18   679   686  22   443   438   6   883   864   3   548   536  14   834   844
 3   664   670  14  1592  1589   3  1657  1643  20   500   500  24   346   349   7  1387  1351   4   702   697  15   876   854
 4   814   815  15   821   802   4  1186  1184  22   439   441  26   458   453   8  1051  1003   5   703   666  16  1701  1661
 5   687   662  16  1439  1419   5   814   789  24   437   427  28   381   356   9   477   465   6   706   688  17   636   646
 6   751   743  17   720   718   6  1356  1347     -1,9,L      30   403   364  10  1070  1064   7   821   822  18   988   949
 7   844   842  18  1244  1208   7   275   250   0   354   340     0,2,L      11  1239  1220   8   982   958  19   673   679
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   1.1.L        12 1336 1341   13  529  518   15  905  895    3  311  306    7  411  434   13 1661 1666    7  746  754
 20  678  659   13  696  666   15  389  398   16  436  413    4 1290 1278    9  426  415   15  859  852    8  786  779
 21  262  257   14  700  733   16  214  216   17  636  612    5  279  218   11  290  309   16  337  339    9  216  226
 22  673  668   15  699  699   17  322  314   18  320  314    6  877  868   13  387  407   17  878  874   10  997 1012
 23  238  241   16  731  731      1,11,L      19  594  576    7  416  403      3,1,L       18  168  169   12  875  882
 24  572  569   17  758  758    1  399  420   20  379  370    8  946  953    1 1362 1362   19  599  600   14  839  832
 25  152  117   18  714  723    2  399  413   21  681  671    9  498  474    2 2550 2628   21  650  661   16  543  541
 26  453  426   19  632  632    3  387  418   22  405  387   10 1139 1123    3 1803 1793   23  550  527   17  198  203
 28  333  347   20  556  592    4  253  266   23  557  550   12  645  643    4 2248 2275   25  576  582   18  322  335
   1.3.L        21  373  389    5  341  344   24  507  479   13  360  346    5 2791 2886   27  417  418   20  308  310
  1 1830 1816   22  357  381    6  240  236   25  406  384   14 1179 1161    6 2442 2523      3,5,L       22  275  272
  2  698  688   23  226  211    7  312  324   26  322  329   15  180  185    7  275  265    1 1938 1921      3,9,L
  3 2275 2247   24  234  226    9  297  291   27  402  389   16  916  902    8 1260 1305    2 1473 1497    1  595  583
  4  798  801   26  281  269      2,0,L       28  256  247   18  881  868    9  232  236    3 1821 1846    2  381  417
  5 2843 2861      1,7,L        2 4299 4791   29  274  291   20  510  512   10 1760 1757    4 1267 1270    3  717  696
  6  477  464    1  597  585    4 2645 2669      2,4,L       22  372  420   11  476  467    5 1457 1477    4  298  263
  7 1867 1878    2  794  772    6 2852 2873    1 2864 2901   23  155  149   12 1018 1059    6 1157 1157    5  610  620
  8  753  746    3  461  454    8 2312 2324    2  490  489   24  297  300   13  735  734    7 1023 1023    6  522  509
  9 2301 2287    4  842  832   10  471  451    3 1877 1892      2,8,L       14 1336 1351    8  780  782    7  551  557
 11 2213 2252    5  296  298   12 1698 1685    4  646  654    1  467  441   15  591  605    9  403  397    9  588  590
 12  215  210    6  989  984   14 1719 1747    5 1894 1838    2  467  466   16  942  955   10  950  930   11  627  623
 13 1459 1440    7  146  198   16 2182 2193    6  784  769    3  437  406   17  624  634   11  741  710   13  474  484
 15  946  960    8 1330 1323   18 1165 1161    7 1154 1135    4  769  767   18  811  817   12  539  545   15  414  415
 16  312  294    9  273  275   20 1110 1103    8 1037 1008    5  817  820   19  623  611   13  483  483   17  300  308
 17  754  767   10  920  910   22  575  589    9 1447 1433    6  716  700   20  619  605   14  545  531      3,11,L
 19  761  768   12  936  954   24  611  621   10  994  972    7  746  733   21  489  480   15  404  397    1  424  397
 21  768  776   14  565  564   26  282  291   11 1005  996    8  798  777   22  605  600   16  750  755    2  399  386
 23  724  725   15  293  317   28  281  264   12  444  430    9  677  662   23  315  306   17  685  666    3  368  350
 25  617  615   16  635  637      2,2,L       13 1133 1114   10  711  688   24  430  414   18  710  709    4  337  340
 27  392  405   17  172  225    1 1015  972   14  343  331   11  575  562   25  220  187   19  694  678    5  329  321
 29  253  261   18  440  462    2 1097 1057   15  757  744   12  598  598   26  408  410   20  633  637    6  286  264
   1.5.L        20  370  374    3 2045 1973   16  247  191   13  416  417   28  252  267   21  496  487      4,0,L
  1 1703 1691   22  378  375    4  425  415   17  877  882   14  422  417      3.3.L       22  212  207    2 2326 2393
  2 1297 1300      1,9,L        5 2132 2145   18  180  176   15  260  281    1 1190 1190   23  226  206    4 2564 2586
  3 1850 1838    1  677  681    6 1600 1596   19  867  857   16  343  324    2  552  515   24  280  291    6 1732 1679
  4 1640 1654    2  388  383    7 1209 1244   21  675  678   17  285  278    3  975 1000   25  220  212    8 2140 2181
  5 1609 1608    3  643  655    8 2626 2655   22  256  240   18  174  229    4  235  224      3,7,L       10 1157 1146
  6  929  923    4  284  279    9 2201 2220   23  410  414   20  280  268    5 1242 1261    1  499  484   12  927  903
  7  563  555    5  726  727   10 1803 1771   24  295  300   21  219  235    7 1959 1953    2  884  868   14 1213 1232
  8 1123 1100    6  233  218   11 1318 1302   25  276  277      2,10,L       8  320  310    3  542  528   16 1235 1242
  9  343  338    7  787  786   12  927  900   27  367  367    1  670  684    9 1854 1892    4  607  587   18 1233 1234
 10  305  309    9  659  661   13 1539 1515      2,6,L        3  635  639   10  241  188    5  138  116   20  719  704
 11 1124 1142   11  590  583   14  831  831    2 1836 1800    5  547  539   11 2181 2185    6 1218 1186   22  895  877
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    4,0,L       12  681  655   12  528  515    3  869  903   24  292  288   22  572  578   16  309  264    3  491  508
 24  550  542   13  888  881   13  490  502    4  242  263      5,7,L       24  493  504   17  579  575    5  496  526
 26  227  248   14  401  394   14  335  346    5 1485 1476    2  573  576   26  283  259   19  500  496    7  466  519
 28  256  260   15  715  712   15  287  277    6  763  784    3  547  540      6,2,L       20  149  165    9  317  362
    4,2,L       16  344  348   16  306  312    7 1081 1094    4  738  726    1 1781 1794   21  583  573      7,1,L
  1 1781 1775   17  844  836   18  254  245    9 1223 1218    5  555  539    2 1283 1328   23  425  415    1  458  479
  2  435  443   19  622  608   19  209  198   10  286  282    6  843  812    3 1693 1726      6,6,L        2 1225 1246
  3 2890 2910   21  565  532      4,10,L      11 1745 1755    7  375  364    4 1244 1248    1  190  205    3 1184 1227
  4  189  198   22  183  225    1  567  566   13 1407 1410    8  818  817    5 1069 1075    2 1442 1447    4  921  954
  5  877  870   23  488  489    3  619  602   14  234  203    9  265  252    6  266  284    3  319  327    5 1395 1429
  6 1234 1242   25  298  308    5  602  595   15 1130 1135   10  834  833    7 1234 1235    4 1245 1243    6 1293 1298
  7 1486 1497      4,6,L        7  552  557   16  310  311   12  799  801    8  402  406    6 1011 1003    7 1238 1249
  8  913  934    1  285  276    9  295  312   17  430  437   14  735  751    9 1135 1148    8  889  891    8 1147 1188
  9 1144 1157    2 1400 1391   11  356  361   19  612  601   15  188  150   10  371  374    9  183  180    9  663  662
 10 1180 1167    3  431  443      5,1,L       20  198  180   16  522  516   11 1279 1317   10  643  628   10 1087 1109
 11 1823 1849    4 1223 1235    1 2482 2551   21  419  421   18  403  409   12 1114 1135   11  243  232   11  119  110
 12  596  618    5  135   46    2 1289 1275   22  166  161   20  285  284   13  725  732   12  647  644   12  648  656
 13  983  994    6 1020 1024    3  994 1014   23  394  402      5,9,L       14  522  530   14  454  460   13  477  496
 14 1072 1067    7  264  259    4 1708 1761   25  429  428    1  696  695   15  668  649   15  239  242   14 1136 1151
 15  983  975    8  878  854    5 1660 1644      5,5,L        3  466  481   16  627  648   16  569  555   16  794  803
 16  525  521    9  214  198    6 1632 1672    1  809  821    4  453  458   17  911  921   17  179  186   17  263  268
 17  588  572   10  698  706    7 1001 1006    2 1497 1499    5  426  414   18  337  349   18  498  481   18  536  542
 18  383  394   11  359  338    8 2022 2068    3 1212 1204    6  369  364   19  561  558   20  447  453   19  279  306
 19  719  730   12  658  659    9  661  670    4 1441 1467    7  438  450   21  398  385      6,8,L       20  515  533
 21  508  510   13  268  237   10 1148 1199    5 1240 1241    8  256  234   22  262  210    1  520  528   21  389  388
 22  353  349   14  772  773   11  150  164    6 1060 1064    9  621  638   23  348  350    2  579  577   22  398  397
 23  373  361   15  281  219   12 1270 1312    7  914  917   11  468  488   25  311  296    3  262  249   23  294  300
 24  315  312   16  818  804   14 1137 1161    8  789  788   13  374  386      6,4,L        4  279  257   24  321  339
 25  348  347   18  698  690   15  330  339    9  512  518   15  355  366    1 1375 1392    5  437  409      7,3,L
 26  309  301   20  508  518   16  899  906   10  478  453      5,11,L       2  679  655    6  428  438    1 1571 1588
 27  308  321   22  452  451   17  554  553   11  565  560    1  331  360    3 1286 1311    7  363  391    2  463  482
    4,4,L          4,8,L       18  729  734   12  666  678    2  241  302    4  283  256    8  485  493    3 1231 1246
  1 1401 1406    1  393  406   19  547  529   13  307  288      6,0,L        5 1721 1737    9  399  400    4  119  118
  2  295  261    2  640  637   20  636  648   14  498  508    2 1017 1059    6  289  277   10  467  484    5  875  900
  3 2437 2434    3  421  415   21  520  516   15  289  293    4 1621 1629    7  840  862   11  410  410    6  355  381
  4  544  568    4  568  577   22  436  421   16  624  641    6 2101 2150    8  225  211   12  343  356    7 1003 1011
  5 1398 1411    5  525  534   23  258  265   17  325  314    8 2073 2127    9 1452 1449   13  380  452    8  379  392
  6  556  559    6  313  286   24  434  394   18  541  554   10 1122 1142   10  456  439   14  278  302    9 1184 1209
  7 1677 1680    7  620  608   25  209  162   19  669  666   12  769  768   11 1044 1037   15  347  339   11 1089 1104
  8  783  765    8  761  748   26  330  333   20  422  408   14  724  721   12  408  374   16  362  365   13 1203 1223
  9 1701 1693    9  580  581      5,3,L       21  323  317   16  968  986   13  881  898   17  284  241   14  250  262
 10  541  543   10  590  594    1 1212 1230   22  344  374   18  647  649   14  712  714      6,10,L      15  740  746
 11  982  984   11  507  501    2  817  798   23  268  264   20  695  704   15  611  615    1  369  411   17  649  653
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    7.3.L       3  429  458   2  732  720  12  309  323     9.5.L      18  506  496  12  428  451   6  446  446
 19  453  472   5  401  414   3  858  853  13  320  344   1  640  661  20  394  372  14  385  364   7  369  346
 21  328  334   7  370  397   4  255  236     8,10,L      2  444  442     10.2.L       10.8.L       8  382  381
 22  172  156   8  249  238   5  895  927   1  284  308   3  657  660   1 1268 1280   1  406  408   9  505  500
 23  303  290   9  429  461   7 1068 1085   3  278  353   4  634  623   2  376  387   2  428  449  10  490  472
    7.5,L      11  352  385   8  137   87     9,1,L      5  349  358   3  349  350   3  376  397  11  339  335
  1  961  978     8.0.L       9  809  810   1  155  127   6  728  728   4  798  819   4  250  294  12  397  404
  2 1018 1036   2 1346 1343  11  921  935   2 1462 1477   7  782  794   5  904  921   5  253  263  13  232  257
  3  814  839   4 1398 1438  12  355  349   3  696  700   8  617  593   6  481  492   6  257  269  14  270  272
  4  892  921   6 2515 2613  13  505  506   4  936  934   9  613  607   7  669  660   8  196  230     11,7,L
  5  924  925   8  552  550  14  445  433   5  861  875  10  572  562   8  161  166     11,1,L      2  565  576
  6  921  932  10 1310 1335  15  403  399   6  581  584  11  416  407   9  700  698   1  138  145   4  509  529
  7  668  665  12 1124 1135  16  372  359   7  491  505  12  389  391  11  631  635   2 1007 1026   6  469  455
  8  631  639  14  701  698  17  504  504   8 1142 1168  13  262  292  12  454  450   4  792  796   8  355  345
  9  749  779  16  516  514  19  414  418   9  749  743  14  327  338  13  420  426   6  765  773     12.0,L
 10  655  669  18  477  487  21  413  408  10  389  378  15  239  242  14  483  464   7  563  571   2  398  401
 11  409  438  20  531  538     8.6.L      11  659  669  16  305  276  15  480  456   8  677  668   4  565  576
 12  447  482  22  508  520   1  326  319  12  685  693  17  291  292  16  422  415   9  540  546   6  866  847
 13  267  270     8.2.L       2  742  763  13  290  287  18  351  350  17  359  348  10  554  545   8  609  598
 14  401  412   1  850  874   3  306  332  14  640  643     9,7,L      18  350  325  11  518  503  10  651  643
 16  391  408   2  774  782   4  959  983  15  143  136   2  578  599  19  297  322  12  409  383  12  493  499
 17  280  301   3 1506 1534   6  855  852  16  583  596   4  694  705  20  243  231  13  252  244  14  511  492
 18  409  418   4  693  697   8 1011 1025  18  418  418   5  237  253     10.4.L      14  483  471  16  310  301
 19  347  354   5  795  816  10  690  700  19  248  235   6  465  475   1  711  703  16  470  463     12,2,L
 20  369  392   6  581  600  11  215  224  20  314  324   7  247  265   2  816  819  18  276  278   1  572  550
 21  264  277   7  802  803  12  429  449  21  237  256   8  391  381   3  818  795     11.3,L      2  634  630
    7.7,L       8  242  259  14  420  439  22  377  381  10  377  388   4  453  427   1 1004 1014   3  600  568
  2  700  714   9  961  963  15  206  198     9.3,L     12  379  407   5  842  839   2  250  280   4  487  474
  3  349  339  10  555  585  16  396  394   1 1224 1265  14  408  417   7  687  684   3 1062 1065   5  542  539
  4  636  654  11  795  802  17  148  140   2  216  213     9,9,L      9  691  681   5  761  759   6  397  386
  5  283  286  12  465  464  18  343  352   3 1182 1216   1  437  467  11  571  588   7  525  530   7  638  632
  6  704  707  13  605  625     8.8.L       5  819  829   3  392  423  13  473  471   9  500  512   9  533  521
  7  326  334  14  551  576   1  487  504   6  249  242   5  330  353  14  314  295  10  232  203  10  134  132
  8  534  541  15  602  620   2  545  556   7  903  921   7  291  305  15  349  343  11  478  484  11  342  351
  9  175  171  16  577  593   3  415  414   8  243  250     10.0,L     16  274  270  13  516  513  12  269  257
 10  502  525  17  463  480   4  406  414   9  807  807   2  674  675  17  291  290  15  396  405  13  416  414
 11  195  225  18  425  431   5  291  284  10  199  210   4 1327 1342     10,6.L      17  462  460  14  167  185
 12  627  621  19  455  469   6  349  359  11  822  816   6  985 1001   1  314  307     11,5.L     15  324  317
 14  596  610  20  156  190   7  271  261  13  677  703   8 1070 1097   2  443  450   1  315  318     12,4,L
 16  430  451  21  296  306   8  252  272  15  793  779  10  904  910   4  540  529   2  536  508   1  598  605
 18  366  364  23  290  290   9  179  202  17  548  564  12  864  852   6  701  708   3  319  295   2  454  428
    7.9.L        8.4,L       10  329  345  19  438  449  14  587  562   8  721  699   4  416  420   3  613  611
  1  504  526   1  761  769  11  298  308  21  333  319  16  320  333  10  564  554   5  357  356   4  403  386
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   12,4,L        6  385  385    8  468  465    9  376  369        13,7,L      3  425  412    4  264  272    9  179  195
 5  470  482     8  391  393    9  245  239   11  381  365     2  428  432    4  419  420    5  300  307       15,3,L
 6  187  185    10  399  406   10  394  397       13,5,L          14,0,L      5  357  353    7  436  421    1  406  400
 7  549  528        12,8,L     11  375  363    1  246  239     2  497  490    6  313  309    9  263  290    3  452  452
 9  582  575     1  222  240   12  343  345    2  299  296     4  486  478    7  386  379       14,6,L      5  486  477
11  423  435     2  238  237   13  265  279    5  208  207     6  324  317    8  283  279    2  403  325    7  381  380
13  423  413        13,1,L     14  352  354    6  318  299     8  364  360    9  355  353       15,1,L         16,0,L
   12,6,L        2  600  599       13,3,L      7  206  206    10  423  443   11  263  251    1  237  234    2  458  462
 1  170  178     3  151  168    1  640  648    8  334  324    12  440  453       14,4,L      2  393  378    4  298  278
 2  416  410     4  631  631    3  706  706    9  288  280        14,2,L      1  413  397    4  305  306       16,2,L
 3  162  170     6  547  545    5  639  639   10  208  272     1  490  485    2  237  247    6  403  391    2  241  227
 4  358  351     7  220  211    7  464  461                    2  287  312    3  279  272    8  275  263    3  228  222
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Compound:

$[Pt(CH_3)(PMe_2C_6F_5)_3](PF_6)$

X 26

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  0,-19,L        13  297  306     0,-10,L        11 1061 1047     6  453  420    11 1181 1151    13  448  442     5  284  272
  3  222  231    15  289  283     0 1609 1613    12  529  525     7 2085 2068    12  459  462    14  956  945     6  500  513
  0,-18,L         0,-13,L         2 1001 1012    13  364  369     8  171  147    13  557  534    15  205  187     8  434  448
  0  442  417     1  498  491     3  610  605    14  610  588     9 1648 1647    14  220  247    16  719  686    10  402  421
  2  276  295     3  660  646     4 1547 1524    15  435  427    10  171  159    15  522  521    17  360  353    12  440  404
  4  234  250     4  138  132     6 1062 1065    16  333  322    11  950  929    16  764  735    18  975  952     1,-15,L
  0,-17,L         5  668  667     7  222  227    17  344  336    12  187  165    17  284  267    20  617  609   -14  424  420
  1  718  680     6  345  343     8  842  826    18  265  238    13  694  691    18  669  656    22  457  446   -12  375  369
  3  647  592     7  554  556    10 1256 1254    19  280  273    15  728  699    19  299  281     0,0,L        -10  454  441
  5  360  351     9  555  466    12  522  490    21  306  301    17  180  137    20  551  535     2  860  826    -8  450  462
  7  361  327    10  492  495    14  563  550     0,-7,L        19  487  476     0,-2,L         4 2264 2260    -7  205  193
  9  249  252    11  373  373    16  397  386    13 1155 1122    21  290  265     0 3400 3876     6 2300 2256    -6  426  427
 11  318  275    12  375  364    17  190  195    14  437  443     0,-4,L         1  359  381     8  716  643    -5  280  283
  0,-16,L        14  307  295    18  228  206    15  522  513     0  548  547     2 2738 2816    10 1380 1335    -4  468  465
  7  255  255    16  227  219     0,-9,L        16  426  431     1  529  547     3 1512 1520    12 1438 1398    -3  248  239
  9  296  281     0,-12,L         1  938  938    17  369  392     2 1502 1501     4 1524 1522    14 1971 1942    -2  284  302
 11  277  263     0 1025  989     3  685  682    18  281  284     4  836  844     5  534  526    16  807  783     0  287  280
 13  199  241     1  209  212     4  240  238    19  231  213     5  304  277     6 1377 1337    18  316  327     3  293  294
  0,-15,L         2  582  559     5  783  791    20  312  311     7  704  721     7  143  155     1,-19,L         7  270  246
  1  717  684     4 1145 1129     7  484  460     0,-6,L         8  370  371     8  798  844    -5  274  247     9  264  257
  2  215  216     5  232  254     8  837  828     0  437  437     9 1180 1191     9 1004  964    -1  317  300    13  177  182
  3  722  701     6 1277 1279     9  485  507     1  960  958    10  615  628    10 1413 1370     1  270  308     1,-14,L
  4  222  232     7  210  212    10  449  446     2  699  717    12  703  686    11 1189 1175     1,-18,L       -15  199  191
  5  680  661     8  891  871    11  410  395     3  621  595    13  625  607    12 1580 1551    -6  201  172   -13  325  302
  7  496  490     9  337  339    12  817  805     4  773  766    14  476  465    13  459  450    -4  283  286   -11  356  366
  8  199  180    10  833  829    13  387  404     5 1467 1465    15  798  793    14  881  886    -2  351  349   -10  338  351
  9  552  546    11  324  294    14  693  710     6  584  575    16  203  175    15  645  643     0  354  371    -9  443  420
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 11  412  391    13  199  179    16  390  384     8  353  358    18  256  251    17  295  278     4  386  392    -7  335  335
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 15  316  272    18  239  231    19  173  173    10  145  184    21  492  495    21  405  402     1,-17,L        -4  490  474
  0,-14,L         0,-11,L        20  300  299    11 1391 1380    23  451  450    23  258  277   -12  309  335    -2  724  739
  0  545  537     2  136  162     0,-8,L        12  205  211     0,-3,L         0,-1,L        -8  331  316    -1  186  188
  2  260  240     3  327  326     0 1714 1701    13  828  808     1 1513 1529     1  829  940    -2  348  328     0  589  588
  3  251  245     4  169  190     1  493  488    15  585  555     2 1175 1161     3 1549 1503     1,-16,L         2  439  432
  4  497  482     6  433  444     2 1344 1347    17  649  637     3 1541 1565     4 1413 1401   -10  246  234     3  144  157
  5  248  254     8  394  379     3  793  774    19  519  504     4  679  689     5  196  190    -8  328  328     4  799  787
  6  635  637     9  237  240     4 1338 1370    21  449  458     5 1790 1819     6 1848 1823    -4  530  519     5  332  299
  7  255  279    10  580  579     5  645  614     0,-5,L         6  609  601     8  338  345    -2  583  583     6  707  693
  8  543  516    12  571  590     6  529  519     1 2249 2350     7 1874 1841     9  395  387     0  717  696     8  634  625
  9  356  333    14  450  453     7  645  665     3 1839 1828     8  336  331    10  858  819     1  148  134    10  629  630
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   1,-14,L       3  459  443   -4  377  360   13  664  669     1,-7,L     -11  169  173   -1  762  774   10 1905 1919
  16  218  232    4  564  589   -3  641  640   15  619  609  -22  360  361  -10 1107 1057    0  718  753   11  429  452
   1,-13,L       6  474  465   -1  308  302   17  536  531  -20  469  465   -9  210  188    2  333  329   12  907  888
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 -11  248  235   12  272  263    4  660  649  -19  378  392  -14  869  832   -5  225  203    5  476  477   15  242  239
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  -9  579  561   17  204  194    6  503  479  -15  726  707  -12 1098 1049   -3  242  212    9  137   41   17  246  229
  -8  455  432     1,-11,L      7  279  261  -14  501  498  -11  177  121   -2 2009 2065   11  373  376   18  488  483
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  -3  769  754   -8  416  435   17  306  332   -9  710  697   -6  620  616    3  184  172   16  426  415  -22  389  396
  -2  263  266   -7 1055 1031   19  358  342   -8  769  715   -5  390  409    4 2166 2093   18  542  538  -20  571  577
  -1  493  477   -5  987  962     1,-9,L      -7  345  340   -4  888  850    5  552  538   20  408  405  -18  591  589
   0  143  125   -3 1075 1049  -18  230  231   -6  448  402   -3  785  769    6 1345 1421   22  320  337  -16  600  596
   1  656  652   -1  757  745  -16  474  447   -5  358  349   -2  704  716    8 2180 2153     1,-4,L     -14  940  913
   3  595  562    0  390  393  -15  325  329   -4 1423 1381   -1  460  466    9  470  474  -20  220  165  -13  515  492
   4  146  130    1 1073 1069  -14  382  370   -3  740  748    0  194  140   10 1551 1560  -19  413  392  -12  916  856
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  11  509  521    9 1009  976  -10  659  650    1  948  932    5 1234 1188   18  484  470  -15  425  441   -8  880  847
  13  281  286   10  282  293   -9  563  564    2 1598 1631    6  406  409   19  233  233  -12  201  194   -7 1696 1653
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  16  177  216   13  552  541   -7  658  649    4 1151 1150    9  791  765   22  343  320  -10  835  788   -5 1480 1459
  17  312  314   15  392  392   -6  367  333    5  262  237   10  731  708     1,-5,L      -9  500  494   -4 1506 1455
   1,-12,L      17  446  438   -5  956  933    6 1126 1128   11  422  416  -22  403  420   -8 1778 1711   -3 2080 2035
 -17  428  435   19  284  288   -4  789  789    7  570  579   14  677  650  -20  758  728   -7  177  168   -2 1110 1143
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 -13  579  594  -19  374  392   -2  406  435    9  624  592   16  374  369  -16  634  609   -4 2977 3000    0  584  579
 -11  666  666  -17  536  516   -1 1728 1761   10  947  915   17  325  305  -15  215  180   -3  809  811    1 2520 2565
  -9  550  531  -15  716  701    0  489  484   11  166  184   18  233  195  -14 1686 1620   -2 1968 1958    3 1043 1037
  -8  146  147  -13  958  927    1 1090 1118   12  820  809   19  269  257  -13  277  280   -1  348  351    4  127  155
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  -6  495  515  -11 1009  997    3 1648 1673   14  525  492     1,-6,L     -10 1349 1317    2  841  839    7 2393 2440
  -5  515  497  -10  443  455    4  168  203   15  211  210  -17  303  304   -9  147  123    3  469  460    8  278  261
  -4  329  304   -9 1001  962    5 1515 1503   16  581  571  -16  168  148   -8 1521 1471    4 1406 1413    9 1114 1095
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  14  565  540  18  207  200 -21  513  511  -5  400  419 -16  373  390  12  528  519 -19  349  318  -3 1046 1054
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   1,-2,L      -16  290  290  -7 1616 1608  -9  178  197  -5  526  518   2,-10,L     -10  835  825   4  241  257
 -21  544  568 -15  699  679  -5 2020 1981  -5  428  425  -4  688  676  20  308  288  -9  981  981   5 1329 1359
 -19  723  711 -13  791  792  -3 1522 1513  -3  383  380  -3  251  231   2,-9,L       -8  447  443   7 1566 1542
 -17 1148 1144 -12 1015 1001  -1 1456 1633  -1  573  580  -2  602  608 -20  294  296  -7  839  795   8  254  239
 -15 1052 1037 -11  790  779   1 1044 1069   1  508  522  -1  625  629 -18  336  338  -5  365  355   9  844  848
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 -13  698  675  -9 1096 1062   5 1065 1040   4  183  165   1  358  353 -14 1083 1071  -2  277  314  11  805  799
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 -11  540  530  -7  794  769   9  222  217   6  217  188   3  368  344 -12  881  856   0  843  855  13 1185 1182
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  -9  709  694  -5 2522 2497  13  778  755   9  566  553   5  631  633 -10 1392 1370   2 1737 1790  17  543  553
  -8  243  233  -4  882  870  15  530  509  11  504  509   6  173  136  -8  929  906   4 1595 1610  20  265  288
  -7 1656 1592  -3 2583 2570  17  801  802  13  415  427   7  504  493  -7  374  346   5  414  419  21  300  346
  -6 2140 2101  -2  380  355  19  457  441  15  340  362   8  309  274  -6  690  654   6 1127 1148   2,-6,L
  -5 2203 2157  -1 2947 3305  21  353  387   2,-14,L       9  602  580  -5  344  354   7  385  386 -21  534  550
  -4 1808 1763   0  436  501  23  361  375 -15  418  428  11  487  476  -4  577  574   8  834  828 -19  647  633
  -3 2294 2291   1 3044 3272   2,-19,L     -13  304  317  13  335  334  -2  881  877  10  480  475 -18  316  320
  -2 1078 1077   2  483  522   3  207  260 -11  439  443  15  217  212  -1  535  522  12  598  587 -17  848  820
  -1 1420 1435   3 3044 3216   2,-18,L      -9  506  492   2,-12,L       0 1677 1693  14  630  644 -16  451  452
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2	871	863	-6	1251	1233	11	255	266	-11	313	307	11,-7,L			-8	861	859	-6	635	637	-16	165	183
3	325	308	-4	1402	1443	11,-12,L			-9	154	134	-18	380	425	-7	364	353	-5	320	338	-11	404	391
4	1006	995	-2	1192	1187	-13	375	392	-8	261	235	-16	415	431	-6	731	734	-4	577	560	-9	533	562
5	171	147	0	1081	1060	-11	437	441	-7	275	252	-14	538	506	-5	159	172	-3	458	454	-8	358	372
6	759	741	2	825	836	-9	347	330	-6	263	240	-12	456	443	-4	649	645	-2	498	498	-7	456	467
7	265	240	4	1073	1051	-7	463	452	-5	374	390	-11	176	185	-2	449	451	-1	476	463	-5	669	666
8	812	781	6	1239	1194	-6	183	194	-4	330	330	-10	556	551	0	617	604	0	309	295	-4	197	202
10	788	767	8	1030	1013	-5	333	312	-3	190	188	-8	601	593	1	359	372	1	673	658	-3	562	553
12	546	549	10	1055	1050	-4	277	250	-2	277	273	-6	706	694	2	542	531	2	539	530	-2	492	474
14	332	342	12	779	762	-3	404	369	-1	740	747	-4	525	532	4	516	491	3	556	562	-1	858	843
16	289	314	14	317	313	-1	313	311	0	251	253	-3	215	229	6	280	295	4	324	317	1	595	588
10,-1,L			11,-16,L			1	236	272	1	486	484	-2	710	726	7	255	237	5	568	549	2	231	234
-20	499	517	-2	263	251	3	249	265	3	603	576	-1	460	418	9	173	142	7	432	441	3	989	999
-18	581	607	11,-15,L			6	225	202	5	542	552	0	408	398	11,-4,L			9	485	482	5	931	927
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-12	418	425	-4	266	275	11,-11,L			11	289	306	3	437	448	-9	390	396	11,-2,L			11	595	597
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-9	379	385	11,-14,L			-5	362	358	11,-8,L			7	206	165	-7	290	301	-15	497	487	15	324	343
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-6	645	654	-6	189	183	1	687	615	-13	377	362	-10	197	201	0	923	941	-10	246	251	-17	537	548
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3	495	466	4	331	323	13	346	369	-6	162	203	0	815	794	6	817	779	-4	552	544	-5	831	826
5	423	399	6	232	222	11,-10,L			-5	686	663	2	621	619	7	439	226	-3	445	442	-3	604	627
6	200	167	8	227	254	-15	453	478	-4	560	543	4	863	847	8	662	641	-2	257	261	-1	1040	1027
7	271	269	11,-13,L			-13	370	385	-3	482	482	5	142	125	9	208	185	-1	622	631	1	681	680
9	363	349	-10	378	364	-11	419	449	-2	310	298	6	629	641	10	662	653	0	400	397	3	400	410
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12	345	346	-5	218	224	-5	742	723	1	348	350	12	493	484	11,-3,L			3	553	560	12,-15,L		
14	345	362	-3	326	321	-3	493	490	2	481	497	14	353	356	-20	211	314	4	383	378	-6	188	177
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  12,-15,L     -1  179  179 -10  204  197  11  447  456   9  519  514  -4  746  751   3  337  343  -8  495  486
   3  226  248  0  282  281  -8  333  316  13  429  473  11  425  434  -2  896  877   7  258  276  -6  577  577
  12,-14,L      2  374  409  -7  152  182    12,-4,L       12,-2,L      0  943  920    13,-10,L    -4  648  647
  -9  289  298  4  370  377  -6  246  227 -17  251  279 -19  207  234   1  208  213 -11  439  423  -2  575  562
  -7  250  259  6  330  306  -4  418  414 -15  500  500 -18  208  212   2  578  587  -9  373  393  -1  197  216
  -5  270  269  8  370  393  -3  315  295 -13  481  488 -17  266  252   4  438  441  -7  457  455   0  529  527
  -3  246  248 10  272  309  -2  374  360 -11  608  629 -16  313  282   5  278  274  -5  541  554   2  436  430
   1  209  214 12  293  329  -1  263  272 -10  188  173 -15  207  227   6  418  424  -3  529  518   3  259  229
   3  252  258   12,-9,L     0  241  249  -9  791  810 -13  297  312   7  303  282  -1  439  445   4  387  385
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  -8  368  368-12  343  362   3  422  416  -3  612  606  -9  458  461  12  226  252   5  319  324   9  189  181
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  -2  242  238 -6  579  604   7  447  440   2  223  248  -6  149  139 -16  256  241 -13  220  198 -14  181  183
   0  221  189 -4  663  667   9  404  392   3  432  422  -5  525  536 -14  367  372 -12  265  261 -10  220  210
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   2  245  220 -1  196  214    12,-6,L     5  622  607  -3  730  714  -8  420  418  -8  221  233  -8  196  210
   6  233  240  0  402  415 -17  331  352   6  278  266  -1  301  282  -6  382  357  -6  246  250  -5  165  133
  12,-12,L      1  144  121 -15  430  449   7  462  455   0  383  359  -2  259  262  -2  373  346   0  384  378
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   13,-5,L         5   324   327 -13   515   507 -11   179   204 -11   406   417 -10   225   217  -5   230   222  -4   418   404
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 -11   313   323 -12   229   234   3   820   810   3   266   262  -9   323   324  -1   449   426     15,-8,L         15,-4,L
 -10   278   278 -11   626   638   5   447   422   5   280   293  -7   537   533   1   374   369  -9   247   254 -14   245   247
  -8   437   406  -9   457   460   7   448   461   7   226   219  -5   516   528   5   314   288  -8   180   193 -12   389   370
  -5   271   212  -8   263   261     14,-13,L      8   176   200  -4   275   245   7   245   257  -7   275   272 -10   334   320
  -3   187   215  -7   503   481  -4   238   256   9   255   251  -3   646   649     14,-1,L      -5   225   212  -8   395   390
  -2   284   273  -5   504   492     14,-12,L        14,-7,L      -1   541   536 -14   237   285  -4   190   192  -6   219   431
  -1   253   253  -3   552   555  -8   214   223 -15   204   230   1   537   557 -13   201   231   0   226   216  -5   151   171
   0   358   482  -1   595   601  -3   162   177 -13   349   325   3   499   493 -12   437   437   3   258   282  -4   338   307
   1   633   615   0   289   265  -1   159   167 -10   268   270   5   438   420 -10   420   400   7   274   344  -3   193   187
   2   196   192   1  1054  1039     14,-11,L     -9   265   252   7   476   455  -8   369   390     15,-7,L      -2   226   268
   3   229   230   3   647   623 -10   567   285  -8   302   284   9   340   336  -6   490   501 -13   209   200  -1   242   229
   4   137   136   5   414   413  -8   361   377  -6   208   209     14,-3,L      -4   559   554 -11   233   241   1   202   206
   5   236   228   6   166   240  -6   325   315  -4   183   194 -17   255   237  -2   666   655 -10   196   190   3   167   129
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   7   207   172  10   271   265  -2   320   327  -2   141   126 -14   303   297   2   839   788  -6   284   278     15,-3,L
   8   261   263     13,-1,L       0   308   315   2   283   272 -13   292   299   4   578   572  -4   303   317 -13   195   203
  10   347   319 -17   366   373   2   340   329   4   331   325 -12   240   231   6   404   413  -2   379   373 -10   183   187
  12   455   450 -15   321   319   4   366   354   6   342   320 -11   343   306   8   430   437   0   308   320  -9   345   320
   13,-3,L       -14   199   209   6   326   338   7   160   132 -10   423   415  12   258   281   2   403   328  -7   349   281
 -18   252   258 -13   332   349     14,-10,L      9   170   152  -9   304   304     14,0,L        4   343   355  -6   237   256
 -17   266   290  -8   229   217 -12   339   320     14,-6,L      -8   180   175 -16   502   500   8   268   288  -5   331   316
 -16   201   218  -6   309   309 -10   280   258 -13   227   256  -7   361   347 -14   293   317     15,-6,L      -4   399   391
 -14   305   327  -4   322   312     14,-9,L     -11   326   326  -6   274   279 -12   487   477 -14   285   289  -3   252   243
 -12   389   413  -2   235   244 -10   293   273  -9   349   356  -4   365   368 -10   277   269 -12   295   317  -2   445   440
 -10   298   309   0   246   226  -8   412   403  -8   192   148  -2   244   248  -8   207   202 -10   322   327   0   404   405
  -8   682   676   1   249   231  -6   376   387  -7   504   495   0   719   713  -6   346   309  -9   196   160   2   417   410
  -6   650   643   3   476   440  -4   383   407  -5   545   551   2   411   405  -2   350   347  -8   323   317   4   329   327
  -4   575   601   5   502   499  -2   420   412  -3   569   570   4   412   399   6   339   314  -6   412   377     15,-2,L
  -3   190   208   6   162   181   0   314   292  -1   523   535   6   350   356   8   264   229  -4   328   329 -14   351   324
  -2   696   695   7   493   493   2   389   392   1   526   518  10   205   185     15,-12,L     -2   289   291 -10   250   216
  -1   256   259   9   298   292   4   335   325   3   456   458     14,-2,L       1   215   223     15,-5,L      -9   290   278
   0   355   341  11   175   195   6   381   372   5   447   440 -16   339   335   3   233   239 -12   226   208  -7   387   239
   2   564   555     13,0,L       10   182   208   7   406   424 -15   225   217     15,-10,L    -10   173   190  -5   338   324
   3   288   289 -17   328   330     14,-8,L         14,-5,L     -14   234   239  -9   287   261  -8   299   304  -4   215   240
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  15,-2,L     -5  436  428    5  356  323   -7  320  323    2  296  276   -8  282  261   -6  208  208   -5  216  211
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  1  504  501  1  603  586  -11  285  309   -3  346  331    7  463   33     17,-5,L      -2  310  301  -10  192  202
  3  413  403  3  533  497   -9  341  335   -2  225  208    8  280  321    2  250  246    0  248  240   -6  285  282
  5  450  444  5  439  437   -7  376  377   -1  352  339   10  212  348    4  453  192     17,-1,L      -4  252  239
  7  296  291  7  474  460   -5  406  388    1  180  204     16,0,L       6  194  241  -13  262  273    0  168  164
  9  254  301  9  350  357   -3  459  434    4  211  206  -14  295  286     17,-4,L      -9  496  473    2  179  197
  15,-1,L       16,-10,L     -1  307  298     16,-2,L     -12  445  445  -12  196  252   -7  444  434    4  242  243
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-13  430  423   16,-8,L       5  170  121  -10  443  415   -8  539  506   -8  313  290   -3  376  377  -10  182  177
-11  392  364-10  231  239     16,-4,L      -8  424  405   -6  406  388   -6  335  330   -1  277  241     18,0,L
 -9  403  380 -8  171  184  -10  221  217   -6  386  354   -4  397  377   -4  357  351    1  247  226  -10  234  233
 -7  361  380 -6  172  184   -6  277  261   -4  351  354   -2  395  393   -2  359  344    3  264  246   -8  410  400
 -5  329  311   16,-7,L      -4  169  169   -2  240  229    0  211  197    0  353  357    5  286  293   -6  287  301
 -4  229  194-11  253  269   -3  225  240    1  251  221    2  341  335     17,-3,L        17,0,L      -4  334  312
 -3  359  365 -9  326  317    1  465  476    3  205  216    4  325  302  -11  187  196   -3  320  303   -2  348  336
 -2  231  200 -5  287  321    3  349  314    4  273  267     17,-9,L      -9  314  304     18,-6,L       2  238  250
 -1  331  333 -3  251  260    5  308  295     16,-1,L      -3  178  237   -7  314  290   -6  196  110    4  239  271
  0  163  157 -1  254  261    7  257  253   -6  452  261     17,-7,L      -5  288  286     18,-5,L        19,-1,L
 10  295  141   16,-6,L        16,-3,L      -4  333  309   -5  179  176   -1  201  175   -5  210  281   -5  240  213
   15,0,L     -3  218  228  -13  261  264   -2  439  428    2  206  203     17,-2,L      -3  287  251    3  211  230
 -9  273  263  1  445  437  -11  326  280   -1  213  237     17,-6,L     -12  162  141     18,-3,L
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Compound:

cis-$PtCl_2(PEt_3)_2$

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  0,-24,L      2  914  890  13  370  375   1  545  552   2 1515 1565  13  171  142   5 1713 1596  14  571  578
 0  269  264   3  136  142  14  255  251   2  682  640   3  304  305  14  224  189   6  413  418     0,0,L
 1  298  281   4  188  168    0,-14,L      3  973  966   4  249  228  15  305  296   7 1274 1191   2 2828 3598
 2  167  191   5  167  158   0  196  160   4  975  947   5  192  183  16  316  330   8  454  450   4  875  871
 3  216  210   6  508  484   1 1284 1297   5  925  895   6  893  845  17  443  454   9  185  172   6 1559 1562
  0,-23,L      7  146  108   3  815  776   6  822  780   7  211  207    0,-5,L     10 1021  982   8 1741 1745
 1  119  125   8  660  616   4  237  213   7  247  196   8 1369 1305   1 1454 1542  11  667  644  10 1082 1069
 3  317  308  10  503  474   5  293  287   8  128  130   9  475  443   2  297  288  12  620  602  12  407  412
 5  396  392  12  133  140   6  478  455  10  416  402  10 1175 1128   3 1222 1214  13  846  821  14  187  201
  0,-22,L       0,-17,L      7  781  737  11  343  336  11  215  211   4  519  468  15  560  555  16  439  440
 1  320  322   1  101   95   8  417  384  12  676  646  12  511  475   5 1593 1554    0,-2,L     18  370  371
 3  208  195   2  906  906   9  913  880  13  473  444  14  162  153   6  252  219   0 1924 2594    1,-24,L
 7  344  334   3  286  258  10  174  163  14  513  513  16  445  443   7  914  862   1 1950 2289  -2  337  329
  0,-21,L      4 1245 1216  11  477  457  15  228  251    0,-7,L      8  223  215   2 1641 1596  -1  216  221
 1  138  119   5  299  289    0,-13,L     16  250  246   1  526  536   9  189  193   3 1646 1617   0  404  397
 2  215  208   6  877  831   1  417  394    0,-10,L     2  894  903  11  831  792   4  338  325   1  165  180
 3  339  319   8  223  204   2  104  101   0 1636 1726   3  956  953  12  205  199   5  573  536   2  323  292
 4  300  284  10  221  201   3 1026 1027   1  911  906   4 1522 1480  13 1046 1027   6 1343 1318    1,-23,L
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  0,-20,L      1  839  827   7  514  481   5  102   68   8  467  437  17  162  156  10 1007 1005   1  141  126
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 1  354  364   3  544  539   9  167  156   7  374  345  10  525  500   0  750  786  12  152  126   3  162  170
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 9  332  324  11  253  237   2  403  396  16  406  415    0,-6,L      7 1317 1241    0,-1,L      0  495  500
10  264  242  12  190  159   3  397  375    0,-9,L      0 1122 1126   8  555  529   1  386  413   2  262  273
  0,-19,L       0,-15,L      4  101   70   2 1161 1181   1 1240 1257   9 1670 1626   2 2466 2712   3  147  130
 2  553  536   1  444  442   5  441  427   4 2010 2059   2  580  585  11  847  800   3 1113 1100   6  288  280
 3  213  214   2  520  500   6  314  284   5  234  213   3  475  480  12  118  117   4 3085 3389    1,-21,L
 4  852  813   3 1015 1000   7  988  923   6 1511 1454   5  470  443  14  127  125   5 1421 1388  -6  377  378
 5  347  321   4  824  799   8  453  427   8  648  606   6  751  704  15  518  491   6 1917 1991  -5  160  164
 6  482  456   5 1023  994   9  947  906  10  469  453   7 1109 1069  17  576  572   7  932  919  -4  459  449
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10  312  290  10  408  377  12  201  181    0,-8,L     10  630  609   2 1228 1219  10  794  778  -1  132  139
  0,-18,L     11  321  282  15  277  299   0 1799 1917  11  794  772   3 1712 1715  12  921  908   0  161  155
 0 1232 1225  12  418  399    0,-11,L      1  573  580  12  279  274   4 1099 1038  13  228  213   1  120   74
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  1,-21,L          8   139   112  -9   548   543  10   251   246   2   746   735  -6   798   801   6   100    53  -5   276   271
   2   470   449   9   146   132  -8   343   341  11   444   429   3   203   188  -5  1128  1141   7  1064  1029  -4   138   153
   4   473   468  11   394   380  -7   531   531  12   452   437   4  1305  1312  -4  1241  1254   8   384   371  -3  1630  1729
   5   201   211    1,-18,L       -6   202   190  13   356   365   6  1182  1154  -3   697   689   9   868   837  -2   425   478
   6   382   374 -11   399   396  -5   163   169  14   331   358   8   370   342  -2  1167  1195  10   270   250  -1  2234  2528
  1,-20,L         -9   652   639  -4   149   124    1,-14,L       10   556   540  -1   190   187  11   442   451   0   653   685
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  -9   311   304  -4   170   173  -2   467   465 -10   550   521  14   621   623   1  1082  1080  16   161   193   2   727   729
  -8   371   372  -3   528   521  -1   952   942  -9   166   157    1,-12,L        2   627   609    1,-9,L        3   778   768
  -7   348   319  -2   176   182   0   748   757  -8   696   691 -15   241   259   3  1293  1281 -15   465   481   4   346   334
  -6   135   149  -1   952   948   1   962   962  -7   190   169 -12   274   275   4   912   901 -13   858   868   5   292   285
  -5   188   171   0   132   119   2   686   676  -6   447   442 -11   251   249   5   898   857 -11   673   684   6   208   156
  -4   168   155   1   918   915   3   310   304  -5   138   154 -10   620   622   6   969   916  -8   183   191   7   700   664
  -3   121   133   3   429   418   4   334   337  -3    92    45  -9   454   461   7   227   211  -7  1529  1572   8   224   215
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  -1   494   488   7   679   651   6   238   220  -1   231   224  -7   431   433   9   171   218  -4   145   124  10   180   188
   0   476   477   9   652   623   7   726   685   0  1063  1058  -6   363   347  10   238   219  -3  2052  2241  11   407   389
   1   456   434  11   322   306   8   535   513   1   192   172  -3   612   628  11   499   491  -2   311   289  15   333   341
   2   280   275    1,-17,L        9   631   607   2   954   964  -2   698   694  12   469   459  -1   528   541  17   355   375
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   8   352   345  -6   150   134 -13   296   287   7   394   365   2  1049  1065  16   143   137   3  1603  1653 -14   444   453
   9   404   370  -5   858   856 -12   297   306   8  1081  1053   3   233   245    1,-10,L        4   114   116 -13   856   861
  10   281   282  -4   273   276 -11   307   290   9   398   368   4   352   353 -15   320   326   5  1177  1149 -12   525   535
  1,-19,L         -3   863   878 -10   166   166  10   861   841   5   552   519 -12   294   261   6   375   343 -11   758   764
 -11   320   305  -2   219   229  -8   124   154  11   143   152   6   797   780 -11   762   764   7   419   391  -8   574   579
  -8   219   207  -1   368   368  -7   255   251  12   255   239   7   553   520 -10   481   482   8   191   189  -7  1286  1325
  -7   368   360   0   143   129  -6   590   584  14   149   161   8  1151  1111  -9  1321  1329   9   172   172  -6  1190  1230
  -6   375   371   1   543   556  -5   432   425    1,-13,L        9   468   451  -8   461   473  11   595   584  -5  1600  1653
  -5   532   534   2   131   117  -4   858   873 -14   513   503  10   836   812  -7  1262  1267  13   519   519  -4  1525  1638
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  -3   579   567   4   310   299  -2   773   791 -12   585   594  12   257   260  -5   123   103    1,-8,L       -2   955  1019
  -2   140   126   5   991   975  -1   159   158 -11   227   197  14   181   201  -4   180   174 -17   426   445   0   912   890
  -1   196   184   6   349   337   1   290   290 -10   363   359    1,-11,L       -3  1313  1355 -15   320   331   1  1253  1306
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   1,-7,L     -13  276  288   2 1866 1864  10  563  550 -15  543  529  11  828  828  -8  283  269  10  436  440
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   1,-6,L      -5  134  139   9  365  344    1,-2,L      -6  899  888   1  461  446   0  417  407  -4  820  838
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-11  484  495   1 1379 1483  15  157  158 -11  579  557   0  254  268   1  270  252   8  386  375   4  521  532
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 -8 1237 1255   4 1757 1761    1,-3,L      -8 1032  998   3 3036 3165   4  159  172  -8  265  245   7  216  195
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  2 2080 2148  13  184  184  -8  333  308   1 1846 1882  13  856  852   7  539  516   3  272  267  -7  464  458
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  2,-15,L        10  303  304     9  947  920     0 1809 1890    -5  143  137     9  249  242    -6  221  220     9 1786 1750
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  2  559  566     3 1323 1328    -3  876  883   -14  635  647    10  702  698    -7 1007 1033     9  437  413    -9  161  156
  3  722  719     4  188  184    -2  623  638   -12  720  726    14  286  295    -6  425  431    10  307  298    -8  533  536
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7	215	214	-13	395	388	-6	249	240	-6	797	807	-4	922	942	-6	227	223	-5	387	401	-4	402	385
8	477	492	-11	255	271	-5	760	774	-5	399	408	-3	297	294	-5	1254	1283	-4	320	308	-3	280	261
9	186	202	-8	130	81	-4	312	298	-4	819	846	-2	394	410	-4	306	301	-3	261	262	-2	176	166
7,-11,L			-7	479	480	-3	594	590	-3	368	365	-1	265	270	-3	617	635	1	481	491	1	371	393
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-9	148	147	-4	174	146	1	714	713	-1	254	273	1	493	490	-1	327	332	3	603	601	3	445	459
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1	378	396	11	593	634	7,-6,L			12	527	572	7,-2,L			11	522	538	5	381	377	-1	252	263
2	617	629	7,-8,L			-13	204	201	7,-4,L			-13	289	268	12	210	215	6	144	126	0	571	577
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   9,0,L     -2  614  626    3  459  446   -3  239  237   -6  137  127    1  246  248    4  524  496     11,-2,L
 9  551  562  -1  298  294    4  373  344   -1  282  267   -5  522  527    4  176  175    5  138  128   -4  335  339
  10,-10,L     0  480  496    5  309  299    1  634  640   -3  258  263    5  265  260    6  282  253   -3  366  362
-4  350  355    1  208  164     10,-6,L     3  643  633    0  217  190    6  404  394      10,0,L     -2  455  440
-3  283  287    2  166  151   -5  272  269    5  335  333    1  448  448     10,-1,L    -8  245  245   -1  369  345
-2  537  538    4  257  234   -4  210  212     10,-4,L     2  278  277   -8  391  398   -4  328  319    0  369  365
-1  255  274     10,-7,L    -3  581  586   -7  176  193    3  504  486   -7  255  238   -2  556  558    1  201  182
 0  450  440   -7  345  338   -2  271  280   -5  185  202    4  303  309   -6  465  466    0  556  549     11,-1,L
  10,-9,L     -6  402  416   -1  634  631   -3  570  573    5  219  257   -5  304  296    2  319  285   -4  210  207
-4  361  359   -5  339  352    1  309  312   -1  616  612     10,-2,L    -4  396  405    6  458  432    1  413  394
 0  413  414   -4  293  292    4  284  287    1  247  233   -9  324  326   -3  193  201     11,-4,L      11,0,L
 2  607  609   -3  182  179    5  405  386    3  245  231   -7  149  162    0  250  244   -2  507  497   -3  548  545
  10,-8,L      0  360  355     10,-5,L     5  547  544   -4  187  195    1  197  195     11,-3,L     -1  607  594
-4  491  496    1  406  402   -7  455  462     10,-3,L    -3  401  409    2  486  464    0  306  313    1  400  385
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Compound:

<u>cis</u>-$PtCl_2(CO)(PEt_3)$

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   0,-13,L       -8  159  150 -14  426  449  -9  219  228  -7  811  818 -15  184  185  -2  120   96  -1  236  245
 -6  219  216    -6  180  172 -12  677  671  -7  293  284  -6  291  290 -13  298  292  -1 1131 1095   0  425  411
 -4  253  247    -4  150  145 -10  955  911  -6   97   90  -5 1103 1090 -12  119   88   0  466  413    2,-10,L
 -2  268  266    -2  184  164  -8 1155 1136  -5  370  355  -4  482  476 -11  427  413    1,-1,L     -10  166  173
  0  276  276     0  161  166  -6 1305 1341  -4   95   96  -3 1218 1143 -10  139  127 -13  177  188  -9  136  145
   0,-12,L        0,-6,L      -4 1461 1548  -3  381  379  -2  353  342  -9  622  614 -11  234  244  -8  228  233
 -8  218  223   -10  130  120  -2 1863 1950  -2   96  103  -1 1235 1207  -8  283  269  -9  384  384  -7  165  153
 -6  299  296    -8  165  169   0 1165 1155  -1  331  338   0  212  204  -7 1003 1054  -8  138  116  -6  305  306
 -4  386  393    -6  195  185    0,-1,L      0  116  124    1,-6,L      -6  517  522  -7  520  503  -5  127  136
 -2  486  476    -4  197  180 -16  216  233    1,-9,L     -15  222  242  -5 1583 1637  -6  414  399  -4  327  321
  0  488  484    -2  273  264 -14  378  393 -13  169  170 -13  385  375  -4  644  662  -5  777  768  -3  155  162
   0,-11,L        0  136  121 -12  640  656 -11  265  260 -12  130  124  -3 1929 1976  -4  316  339  -2  278  277
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 -8  301  275   -14  119  123  -8 1297 1298  -8  117  102 -10  145  153  -1 2062 1914  -2  454  483   0  263  256
 -6  331  309   -12  199  199  -6 1577 1560  -7  479  470  -9  728  737   0  287  321  -1  253  311    2,-9,L
 -4  372  361   -10  327  326  -4 1959 2038  -5  553  520  -8  213  226    1,-3,L       0  310  237 -12  119  132
 -2  517  497    -8  498  505  -2 2560 2945  -4  154  143  -7  895  911 -15  168  154    2,-13,L    -11  105   97
  0  585  576    -6  623  649   0 3193 3373  -3  614  611  -6  328  329 -13  240  256  -6  187  190 -10  177  182
   0,-10,L       -4  626  642    0,0,L      -2  140  131  -5 1009 1019 -12  124   97  -4  206  219  -9  118  126
-12  193  180    -2  683  633 -18  117  130  -1  655  663  -4  401  391 -11  445  429  -3  118  124  -8  261  262
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     2,-7,L      -2 1128 1114  -14  319  337   -6  242  250  -10  363  358   -2 1262 1203   -8  427  429   -4  165  184
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  -4  151  133  -10  581  572   -5  854  848  -11  158  166   -1  810  791  -10  427  431      3,-1,L      -6  160  172
  -3  157  169   -9  431  443   -4 1953 2102  -10  243  238    0  947  958   -9  519  518  -13  132  159   -5  321  316
  -2  224  222   -8  809  808   -3 1088 1140   -9  258  255      3,-6,L      -8  593  586  -12   81   79   -4  186  197
  -1  170  170   -7  556  568   -2 1934 2137   -8  301  293  -15  149  153   -7  649  646  -11  210  205   -3  319  335
   0  243  251   -6  999 1022   -1 1406 1471   -7  317  314  -14  211  203   -6  824  836  -10  161  153   -2  218  204
     2,-5,L      -5  578  604    0 2192 1994   -6  345  344  -13  238  239   -5  753  768   -9  268  262   -1  291  300
 -13  114  110   -4 1118 1180      2,0,L       -5  389  385  -12  315  325   -4 1143 1159   -8  178  182    0  188  179
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 -11  156  155   -2 1301 1298  -16  163  174   -3  466  473  -10  478  465   -2 1486 1469   -6  529  518  -11  119  125
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  -8  331  340      2,-2,L     -12  450  448    0  413  409   -7  588  584      3,-3,L      -3  206  224   -7  265  262
  -7  243  267  -18  124  117  -11  374  377      3,-8,L      -6  625  628  -14  188  196   -2  974 1027   -6  116  120
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  -4  686  699  -14  357  363   -8 1112 1133  -12  192  192   -3  838  802  -11  272  259      4,-13,L     -1  353  353
  -3  237  224  -13  215  221   -7  711  714  -11  227  210   -2  926  877  -10  406  403   -5  178  200      4,-8,L
  -2  693  666  -12  520  518   -6 1691 1740  -10  292  283   -1 1016  998   -9  430  431   -3  204  215   -9  146  154
  -1  144  123  -11  453  444   -5 1163 1191   -9  321  329    0 1015  992   -8  597  597   -2  119  125   -7  242  243
   0  601  588  -10  704  685   -4 2374 2413   -8  390  389      3,-5,L      -7  620  639   -1  221  214   -5  307  291
     2,-4,L      -9  584  567   -3 2269 2244   -7  433  447  -16  125  124   -6  587  611    0  133  124   -4  115  101
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  -8  522  540   -1 1257 1261    0  217  220      3,-7,L      -8  582  599  -14  127  129   -1  291  301   -4  201  207
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  -4 1019 1031  -16  204  201   -8  210  205  -12  263  260   -4  847  841  -10  361  341   -7  258  265    0  327  337
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   4,-5,L      -1 1462 1422 -10  411  387  -5  251  240  -6  896  914  -7  214  228 -11  105  147   -9  444  443
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 -3  386  371  -9  767  722  -1 1880 2032 -10  440  455 -14  218  224 -12  112  123    6,-8,L         6,-3,L
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 -7  544  544    4,-1,L     -7  104   93    5,-6,L     -3  541  520  -1  678  650  -2  207  210   -2  395  396
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   0   186   140  -8   375   381    -9   199   204 -14   128   128    -4   163   180  -1   458   461    -9   565   540   0   704   631
    6,0,L         -7   181   188    -8   564   586 -12   173   171    -3   233   253   0   104    79    -8   472   464    9,-11,L
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  -4   329   327 -12   242   250   -16   127   127  -2   719   743    -4   163   155  -7   393   407   -15   152   161   0   210   218
  -3  1497  1518 -10   336   365   -14   189   200  -1   266   245    -3   174   187  -6   285   289   -14   164   152    9,-9,L
  -1  1970  2083  -9   139   154   -13   108   108   0   647   597    -2   243   241  -5   443   463   -13   233   243 -10    98   117
   0   139   120  -8   457   473   -12   304   303    7,-1,L         -1   187   197  -4   299   297   -12   263   265  -9   149   153
    7,-11,L       -7   220   229   -11   173   177 -12   104   114     0   258   272  -3   428   425   -11   387   381  -8   153   156
  -8   104   123  -6   627   626   -10   432   436 -10   163   158      8,-8,L        -2   335   333   -10   417   405  -7   220   211
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  -1   117   121  -3   342   349    -7   319   312  -6   238   233    -2   143   142    8,-3,L          -7   808   800  -4   242   242
   0   136   168  -2   760   752    -6   735   743  -5   312   308     0   133   139 -15   107   130    -6   468   468  -3   288   289
    7,-10,L       -1   281   276    -5   303   314  -4   250   259      8,-7,L       -14   108    90    -5   915   929  -2   289   289
 -10   130   148   0   752   789    -4   847   861  -3   384   385    -7    98    98 -13   208   200    -4   614   613  -1   304   320
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  -4   285   279 -12   267   262    -1   292   279   0   190   187    -4   183   173 -10   261   255    -1  1290  1293 -11   187   186
  -3   135   136 -11    94    99     0   792   805    8,-12,L        -1   120   114  -9   395   396     0  1201  1108 -10   178   161
  -2   336   341 -10   351   349      7,-3,L       -5   159   142      8,-6,L        -8   371   382      8,0,L        -9   232   234
  -1   166   192  -9   175   176   -14   153   162  -3   140   158    -7   100    98  -7   541   561   -15   190   193  -8   212   209
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  0  446  449  -1  514  488    10,-11,L     -6  337  333  -7  261  262  -5  270  301  -1  600  574     12,-3,L
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-12  144  151      9,-3,L    -4  185  193  -4  401  397  -5  378  390  -1  343  355 -11  148  141 -10  237  233
-11  195  201 -13  202  178  -2  176  182  -3  234  245  -4  894  918     11,-7,L     -9  216  214  -8  292  296
-10  235  223 -12  166  156   0  155  176  -2  383  388  -3  426  442 -11  199  206  -7  288  297  -6  411  405
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  18,-4,L
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  18,-1,L
 -5  184  200
 -3  206  221
 -1  231  216
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  19,-4,L
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  20,0,L
 -2  139  126
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Compound:

$PtCl(Ph_2PC_2B_{10}H_{10})(Ph_2PC_2B_{10}H_{11})$

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1  819  894    5,K,-33      3 1178 1199   2  682  678   5  810  763   6 1210 1115    15,K,-29     0 1940 1960
 8,K,-35      4  802  807   5 1360 1376    8,K,-31       16,K,-30     2 1135 1122   6 1011 1068    14,K,-28
1 1483 1435   2  826  831    10,K,-32     1 1612 1542   2  609  644   0 2313 2254   4 1704 1681   2 1020  999
 12,K,-35      6,K,-33      0 1131 1067    9,K,-31       15,K,-30      1,K,-30      2 1610 1626   0  887  884
1 1708 1630   1 1753 1714    9,K,-32      6 1446 1397   3 1623 1593   3 1827 1783    16,K,-29      13,K,-28
 16,K,-34     3  688  613   3  926  918   4 1684 1744   5 1117 1119   5 1330 1324   1 1574 1534   1 1253 1195
0  729  865    7,K,-33       8,K,-32      2 1317 1327    14,K,-30      2,K,-29      3  944  966   3 1915 1896
 14,K,-34     4 1202 1223   2  992 1011    10,K,-31     6  885  909   1 1072 1061    17,K,-29     5 2164 2219
2 1208 1190   2  891  826   0 2012 1946   1  942  987   2 1214 1202   3  673  725   4  923  975    12,K,-28
0 1568 1469    9,K,-33       7,K,-32      3  578  500   0 2144 2197   7  496  543   2  632  551   6  797  791
 12,K,-34     4  679  613   1 1050 1014    12,K,-31      13,K,-30      3,K,-29       19,K,-29     4  752  749
2  915  943   2  627  570   3 1265 1284   1 1613 1619   1  667  677   6 1534 1473   4 1346 1306   2  759  782
0 1689 1501    10,K,-33     5 1684 1620    13,K,-31     3  837  817   4 1955 1934   2 1035  986   0 2336 2211
 10,K,-34     1 2025 1912    6,K,-32      4 1840 1713   5 1303 1239   2 1634 1581    20,K,-29      10,K,-28
2 1043 1025    11,K,-33     0 1081  951   2 1196 1203    11,K,-30      4,K,-29      1 1621 1597   6  653  639
0 1230 1041   4 1224 1251    4,K,-32       14,K,-31     3 1528 1527   1 1805 1786    21,K,-29     2  589  640
 9,K,-34      2  975  853   2  796  759   1 1201 1244   5 1515 1551   3  522  570   4  925  863   0 1221 1151
1  812  665    14,K,-33     0 2002 2006   3  698  581    10,K,-30     7 1432 1394   2  617  626    9,K,-28
3 1282 1191   1 2093 1931    3,K,-32       16,K,-31     6 1064  986    6,K,-29       23,K,-29     1 1525 1360
 8,K,-34      3  577  640   1  916  832   1 1351 1315   4  647  668   1  897  909   2  693  729   3 2135 2149
2  714  785    15,K,-33     3 1280 1271    17,K,-31     2 1182 1147    7,K,-29       24,K,-29     5 2174 2178
0 2080 1914   2  727  676   5 1840 1725   4 1640 1635   0 2243 2302   6 1731 1711   1 1393 1320    8,K,-28
 6,K,-34       16,K,-33      2,K,-32      2 1078 1013    9,K,-30      4 2276 2133    24,K,-28     6  588  679
2  634  559   1  604  640   2  755  737    18,K,-31     1  798  889   2 1998 1908   2  752  709   4  937  932
0 1102 1073    18,K,-33     0  986  884   1 1240 1223   3 1277 1328    8,K,-29       22,K,-28     2 1134 1160
 5,K,-34      1 1418 1435    1,K,-31      3  808  801   5 1454 1426   1 1646 1644   2 1343 1310   0 1724 1596
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3 1068 1067   0 1549 1527   2  896  858   1  880  860   1  754  710   7 1502 1456    21,K,-28     1  610  646
 4,K,-34       17,K,-32      2,K,-31       21,K,-31     3 1089 1119    10,K,-29     3 1485 1468   3 1389 1356
2  750  777   1  946  926   1  722  696   2  927  946   5 1497 1469   1 1011 1013   5 1353 1318   5  958  984
0 1700 1567    16,K,-32      3,K,-31       22,K,-30      6,K,-30      7  780  784    20,K,-28      6,K,-28
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0 1032  972   0 2051 1968   2  636  659   0 1050 1098   4  662  632   6 1667 1642   0 1251 1167   0 1548 1491
 1,K,-34       15,K,-32      4,K,-31       20,K,-30     2  978  979   4 2041 2042    18,K,-28      5,K,-28
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8  894  914  8  843  900  5  614  556    20,K,18   0 2255 2240     0,K,19    3 1123 1128  2  872  850
6  523  543  6 1466 1480  7 1450 1548  2 1005  978     7,K,18   1 3529 3599  7  745  746  0 1494 1423
2  706  734  2  832  760  9 1456 1439  0 1107 1184  1 1228 1269  3 1581 1590  9 1315 1338   13,K,20
0 1399 1334  0 1988 2128     7,K,17      19,K,18   3 2215 2160  5  777  758     9,K,19    3  996  936
                1,K,16    6 1401 1469  1  564  617  5 2347 2414  7 1753 1829  6 1627 1655  5  958  972
```

```
  11,K,20         0,K,20        3  519  599    8 1440 1372    9  895  856    4 1213 1180    3 2117 2165    6  861  753
 3 1146 1098   10  593  704      11,K,21       2 1335 1424      1,K,23       2  939  967    5 1596 1575    4 1426 1406
 5 1409 1486    4  551  502     6 1417 1425    0 1162 1100    8  495  513      14,K,23      7 1045 1013    2  840  823
  10,K,20       0 1024  920     4 1878 1880      7,K,22       6 1300 1349    1 1010  996      2,K,24         11,K,25
 8 1101 1134      0,K,21        2 1672 1619    3 1415 1462    4 2384 2349    3  666  649    8 1420 1443    4  853  840
 6  712  713    1 2450 2512      12,K,21       5  715  772    2 1504 1514      15,K,23      6  780  804      12,K,25
 2 1191 1142    3 1131 1123     1 1267 1264      6,K,22         2,K,23       4  868  922    2 1876 1892    1 1040 1027
 0 1582 1646    5 1163 1142     3  567  581    8  549  539    1 2385 2350    2  887  877    0 1909 1881    3  674  682
   9,K,20       7 1153 1241     7  833  872    6 1056 1012    3 1167 1300      15,K,24        1,K,24         13,K,25
 1  553  646    9 1332 1277      15,K,21       2  531  583    5  881  838    1  661  622    3  490  377    2  926  856
 3 1857 1916      1,K,21        4 1274 1274    0 1021 1096    7 1160 1151      14,K,24        0,K,24         10,K,26
 5 1581 1581    4  752  794     2 1233 1145      5,K,22       9 1798 1795    2  616  570    8  980  995    2 1200 1167
   8,K,20       2  707  703      16,K,21       3 1585 1538      3,K,23         12,K,24      2 1021 1064    0 1274 1228
 0  999  953      2,K,21        1  869  876    5 1942 1885    4  520  600    2  667  603    0  742  690      9,K,26
   7,K,20       1 1548 1560      17,K,21       7 1029  964    2  748  716    0 1196 1186      0,K,25       3 1435 1383
 1  646  620    3 1311 1310     2  490  527      4,K,22         4,K,23         11,K,24      1 2293 2273    5  996  997
 5 1722 1711    7  603  642      18,K,21       8 1830 1808    3  515  442    3 1496 1543    3 1428 1473      8,K,26
   6,K,20       9 1354 1362     1  547  498    6  443  446      5,K,23       5 1399 1319    5  484  522    2  736  747
 8 1568 1595      3,K,21         17,K,22       2 1880 1995    6 1148 1209      10,K,24      7 1610 1556      7,K,26
 6 1023  982    8  740  675     1  676  649    0 1921 2040    4 2042 2093    2  965  992      1,K,25       3  769  701
 4  478  402    6 1855 1922      16,K,22         3,K,22       2 1512 1531    0  901  794    6  663  757      6,K,26
 2 2014 2028    4 2599 2451     0 1071 1033    1  655  606      6,K,23         8,K,24       4 1699 1695    6  975  910
 0 1523 1671    2 1264 1328      14,K,22       3 1752 1763    1 1926 1922    6  705  679    2 1299 1312    2 1581 1524
   5,K,20         4,K,21        0  746  784    5  915  868    3 1223 1249    2  915  919      3,K,25       0 1567 1564
 3 2964 2961    1 2106 2073      13,K,22       7  683  651    5  561  647    0 1348 1374    6 1238 1252      5,K,26
 5 1569 1531    3 1175 1177     1  761  703      2,K,22       7 1068 1063      7,K,24       4 1241 1262    3 1190 1259
 7  903  993    5  938  930     3 1312 1378    4  554  516      7,K,23       1  618  578    2  869  836    5 1633 1594
 9  634  626    7 1217 1190     5 1627 1612    2  691  651    6  933  876    3 1719 1680      4,K,25       7  781  847
   3,K,20       9 1197 1190      12,K,22       0  635  571    4  904  978    5 1627 1597    1 2152 2170      4,K,26
 3 1119 1086      6,K,21        6  518  502      1,K,22       2  471  462    7  920  932    3 1139 1171    6  540  484
 5 1081 1030    1  857  949     2  879  955    3 1772 1915      9,K,23         6,K,24       5  592  657    2 1019 1050
 7  634  670    3 1071 1059     0 1272 1262    5 1537 1564    6 1093 1043    8 1168 1094    7 1175 1167    0 1390 1342
   2,K,20       9 1219 1169      11,K,22       7  933  985    4 1719 1716    2 1425 1403      5,K,25         3,K,26
10 1079 1108      7,K,21        3  880  898      0,K,22       2 1343 1360    0 1041 1000    4 1838 1830    3  804  765
 8 2009 2056    6 1538 1633      10,K,22       8 2173 2205      10,K,23        5,K,24       2  961  965      2,K,26
 6 1319 1313    4 2488 2458     6  722  629    6 1127 1162    1 1401 1400    3  752  647      7,K,25       8 1383 1442
 2 3092 3073    2 1789 1802     0 1062 1068    4  619  607    3  820  879      4,K,24       6  634  616    2 1773 1756
 0 1606 1763      8,K,21          9,K,22       2 2107 2220    7  952  925    8 1204 1150    4 1144 1103    0 1436 1504
   1,K,20       1 1632 1643     1  955  951    0 1902 1996      11,K,23      6  798  797    2  964  942      1,K,26
 3 2651 2817    3  807  865     3 1681 1689      0,K,23       6  700  723    2 1042 1135      8,K,25       1  664  734
 5 2418 2309    7 1025 1021     5 1601 1613    1  857  896    4 1015  961    0 1606 1483    1 1502 1531    3 1866 1946
 7 1294 1332     10,K,21        7  728  743    3  810  808    2  771  834      3,K,24       3 1101 1080    5 1613 1581
 9  673  648    1  670  651       8,K,22       7  520  645      13,K,23      1  857  853      9,K,25       7  869  945
```

```
  0,K,26       2 1173 1178    11,K,27      5  626  702    0,K,28       5  461  486    2,K,30       3  728  671
8 1194 1174      4,K,27     2  781  723     4,K,28     2 1634 1637     5,K,29     2 1404 1371     4,K,32
6  870  878    1  938  898   12,K,27      6  880  850  0 1748 1824   4 1338 1356   0 1835 1834   0 1267 1247
2 1119 1171      6,K,27     1  917  883  2 1467 1526     0,K,29        6,K,29        0,K,31        2,K,32
0 1730 1740    1 1530 1530    9,K,28      0 1776 1732  1 1273 1347   1 1264 1259   1 1632 1535   2  872  913
  1,K,27       3  885  881  3  935  944     3,K,28     3  592  615     8,K,30     3  756  695   0  948  917
6  763  765      7,K,27       8,K,28      3  728  810    1,K,29      0  808  756     1,K,31        1,K,32
  2,K,27       4 1403 1400  2 1205 1224  5  719  735  6 1070 1138     6,K,30     4 1070 1069   1  637  644
1 1649 1726    2 1020  929  0 1643 1607     2,K,28     4 1472 1521   2 1160 1148   2  640  631   3 1337 1386
3 1144 1144      8,K,27       7,K,28      2  455  448  2 1103 1059   0 1247 1300     3,K,31        0,K,32
7 1548 1523    1 1098 1112  3  665  666  0  997 1006    2,K,29        3,K,30     4  903  933   2  701  755
  3,K,27        10,K,27     5  695  747     1,K,28     1 1059 1072   3 1402 1412   2  708  704   0 1808 1831
6 1038 1044    1 1086 1034    5,K,28      3 1828 1792    4,K,29      5  845  880     4,K,31
4 1728 1728                 3 1194 1141  7  617  515  1 1157 1158                 1 1456 1468
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Compound:

<u>cis</u>-$PtCl_2(F_3CSCHMeCH_2SCF_3)$

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 -10,-6,L        2  220  256    -1  676  714    -4  448  382    -7  465  444     0  436  427    -9  364  358    10  572  591
  0  239  256    5  300  300     3  277  314    -2  444  411    -6  202  150     3  275  269    -7  273  278      -7,-4,L
 -10,-5,L          -9,-5,L       5  596  657    -1  299  298    -5  427  387     6  313  374    -4  328  277   -10  217  123
 -1  342  288   -2  339  325     7  287  320     1  245  262    -4  201  179      -7,-11,L      -3  459  407    -8  307  339
  1  260  274    0  281  270      -8,-11,L       2  333  317    -2  219  186    -6  220  216    -2  262  233    -6  272  269
 -10,-4,L        4  286  345    -3  305  298     4  453  506    -1  589  517    -4  493  441    -1  533  483    -5  585  529
 -3  279  196    6  341  364    -1  203  183     5  207  240     1  576  659    -2  235  180     1  327  330    -3  695  617
  0  206  246      -9,-4,L       3  357  372     6  252  268     2  221  281    -1  229  228     2  246  244    -2  568  505
 -10,-3,L       -4  231  188      -8,-10,L       9  210  202     3  207  219     0  205  206     4  219  265     0  701  682
 -4  307  277   -3  272  249    -4  402  346    10  284  363     5  201  306     2  492  470     5  546  579     1  570  586
 -2  244  231   -1  325  266    -2  362  344      -8,-5,L       7  524  581     3  221  155     7  340  372     3  772  844
  2  241  271    2  208  262     2  306  323    -9  227  216     9  240  291     4  398  395     8  206  208     4  302  357
  4  259  312    3  253  220     4  373  415    -5  335  284      -8,-1,L       8  230  227    11  270  297     5  218  232
 -10,-2,L        5  309  341      -8,-9,L       -3  615  534    -9  288  309      -7,-10,L        -7,-6,L       6  506  538
 -5  221  223    7  252  249    -6  302  253    -1  329  331    -8  222  220    -5  440  424   -11  255  257     8  222  218
 -3  306  291      -9,-3,L      -5  248  221     3  599  678    -6  326  338    -3  476  411   -10  235  197     9  398  440
  3  356  390   -7  215  180    -3  339  339     5  481  553    -5  330  327     1  383  349    -9  256  218    11  264  293
  5  216  248   -5  312  289     0  248  263     9  375  391    -3  492  464     3  535  562    -8  206  217      -7,-3,L
 -10,-1,L       -3  273  242     2  293  292      -8,-4,L       -1  460  383     5  238  185    -5  530  463    -9  336  345
 -1  328  291    1  301  366     3  332  359    -7  278  215     0  487  463     7  233  211    -3  644  575    -7  575  550
  5  262  295    3  325  379     5  270  229    -5  205  190     1  205  249     9  345  391    -2  538  481    -5  227  203
 -10,0,L         7  287  344      -8,-8,L       -4  378  336     2  283  339      -7,-9,L        0  325  305    -4  344  299
 -2  261  308    9  286  330    -7  359  314    -2  396  372     3  315  414    -7  210  188     1  451  433    -3  548  498
  0  297  342      -9,-2,L      -5  435  378    -1  325  317     5  391  436    -6  232  183     2  197  213    -1 1140 1028
  6  359  410   -6  330  279    -1  346  305     1  397  415     6  264  288    -4  370  358     3  761  796     0  158  162
  -9,-9,L       -4  303  320     1  470  488     2  284  357     8  250  278    -2  205  172     4  285  289     1  584  626
 -2  249  268   -1  206  171     3  230  215     4  501  597    11  278  299    -1  267  248     5  312  311     3  214  235
  0  209  206    0  314  336     7  259  285     5  194  122      -8,0,L         2  346  377     6  306  343     4  175  119
  1  264  276    2  330  415      -8,-7,L        6  320  320   -10  344  321     4  331  315     7  276  280     5  685  737
  -9,-8,L        4  233  304    -8  207  212     7  398  443    -8  395  370     5  308  311     8  265  300     6  256  281
 -4  403  334    5  191  205    -6  458  388     9  216  241    -4  493  527     7  227  229     9  474  490     7  722  754
  0  320  317    8  306  330    -4  214  141    10  342  391    -2  611  646     8  245  264    11  316  342    11  267  295
  2  471  493      -9,-1,L      -3  224  189      -8,-3,L        2  345  393    10  288  314      -7,-5,L       13  367  385
  4  231  198   -6  189  112    -2  336  300    -8  293  246     4  498  532      -7,-8,L      -10  332  311      -7,-2,L
  -9,-7,L       -2  557  524    -1  265  229    -6  414  387     6  394  429   -10  219  166    -6  472  419    -8  488  510
 -5  395  344    0  296  320     0  418  399    -2  258  224    10  286  307    -8  337  319    -4  909  804    -6  490  486
 -1  195  120    1  202  295     2  367  376    -1  212  162      -7,-13,L      -6  333  272    -2  495  435    -5  295  286
  0  200  145    3  282  277     5  190  168     0  708  727    -3  210  191    -2  510  475    -1  162   72    -3  192  126
  1  422  442    4  358  405     6  232  275     2  409  506    -1  478  444     0  334  341     0  380  371    -2  769  675
  3  307  337    6  360  437     8  314  375     3  186  167     1  327  338     4  395  400     2  924  954     0  893  863
  -9,-6,L          -9,0,L         -8,-6,L        6  502  598      -7,-12,L       6  405  435     4  697  754     1  264  285
 -3  300  223   -7  239  257    -8  217  190     8  503  551    -6  253  194     8  203  208     8  401  449     2  268  275
 -1  292  304   -3  453  467    -7  213  154      -8,-2,L       -2  383  367      -7,-7,L        9  198  175     3  248  227
```

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   -7,-2,L       -3   270   258   2   525   500   4   916   915 -10   196   197  13   211   182   0  1181  1225  -8   329   362
  4   488   549  -1   251   231   4   186   175   6   724   738  -9   444   425  14   195   205   2  1199  1251  -6   440   395
  5   338   357   0   243   236   5   187   145   7   285   294  -8   400   394    -6,-2,L        4   230   247  -5   340   359
  6   801   843   2   321   321   6   356   353   9   190   214  -7   443   395 -11   309   287   6   537   543  -4   179   203
  8   368   396   3   238   240   8   460   476  10   403   417  -6   722   695  -9   498   492   8  1262  1286  -3   200   207
  9   247   240   5   236   253  10   270   309  12   503   508  -5   279   266  -7   319   298  10   489   504  -2   319   295
 12   416   433    -6,-13,L        -6,-9,L         -6,-6,L       -4   723   660  -6   339   344  14   503   510   0   655   620
   -7,-1,L       -4   329   329 -10   224   190 -12   248   237  -3   715   610  -5   326   346    -5,-16,L       1   232   193
-12   305   259  -2   475   476  -8   257   255 -10   195   169  -1   871   792  -3   889   842  -1   285   310   2   317   333
-10   233   199   0   309   285  -7   286   252  -9   346   322   0   983   934  -2   337   274   1   437   391   3   401   402
 -9   220   180   4   400   387  -5   233   237  -8   291   244   1   208   209  -1   969   872    -5,-15,L       5   203   191
 -7   286   325   6   290   306  -2   563   512  -6   590   558   2   818   822   2   540   555  -2   275   283   6   494   498
 -6   418   433    -6,-12,L      -1   389   353  -4   537   461   3   614   626   3   755   818   0   426   434   8   318   328
 -5   243   249  -6   220   201   0   280   251  -3   517   477   4   321   293   4   247   238   2   290   296  11   224   214
 -4   463   481  -5   199   182   1   465   455  -2   227   195   5   544   580   5   746   794    -5,-14,L         -5,-10,L
 -2   241   240  -4   276   278   3   266   280  -1   516   483   6   507   520   7   375   386  -7   226   234  -9   327   292
 -1   529   472  -3   526   482   4   562   574   0   799   757   7   269   307   8   314   284  -4   341   341  -8   197   206
  0   356   338  -1   498   463   6   443   457   1   256   257   8   474   493   9   344   340  -2   265   221  -7   612   614
  1   335   364   2   222   233   7   407   417   2   574   578   9   240   242  11   470   488  -1   201   226  -5   380   345
  2   520   578   3   400   400   9   343   356   4   157   114  10   282   325  13   348   353   1   339   318  -3   340   327
  4   363   373   4   213   168  10   287   269   5   572   588  11   296   296    -6,-1,L        2   319   327  -2   303   291
  5   464   474   5   404   390    -6,-8,L        6   421   426  13   199   241 -13   301   260   4   306   339  -1   932   887
  7   390   421    -6,-11,L     -11   186   139   7   170   144  14   279   287  -8   252   237   5   215   225   1   715   697
  8   467   470  -7   344   319  -9   426   407   8   482   494    -6,-3,L       -7   427   445   7   242   242   4   188   163
 10   340   370  -5   483   432  -7   197   168  10   291   295 -10   452   466  -5   732   785    -5,-13,L       5   652   643
 11   215   230  -4   323   322  -3   718   655  11   295   265  -8   652   668  -4   326   365  -5   386   365   7   726   731
 13   296   292  -3   324   271  -2   156   116    -6,-5,L       -7   298   291  -3   260   248  -3   454   455   9   275   286
   -7,0,L        -2   254   257  -1   806   749 -13   247   285  -6   172   148  -2   494   455  -1   207   195    -5,-9,L
-11   359   343  -1   303   286   3   632   615 -11   336   339  -5   271   266  -1   339   306   1   233   237 -10   192   145
 -9   285   260   1   538   497   5   971   970  -7   702   683  -4   812   750   1  1058  1159   3   500   487  -9   386   365
 -7   223   253   2   341   352   7   288   301  -5   924   849  -2  1149  1022   2   380   401   9   323   335  -8   457   442
 -5   550   591   3   497   482   9   252   279  -4   322   282  -1   621   564   3   667   725    -5,-12,L      -6   384   351
 -3   318   353   4   335   343  10   177    81  -3   494   433   0   691   680   4   472   499  -7   228   191  -5   355   331
 -1   153    99   6   226   202  11   598   616  -1   679   638   1   322   343   6   438   461  -4   525   499  -3   716   660
  1   545   583   7   346   349    -6,-7,L        1  1004  1004   2   340   361   7   887   921  -3   220   152  -2   592   562
  3   615   667   9   248   304 -10   366   344   2   306   318   3   167   190   9   759   776  -2   308   290  -1   486   461
  5   337   330  10   213   210  -8   391   385   3   730   762   4   810   861  10   329   315  -1   222   180   0   917   881
  7   332   395    -6,-10,L      -4   459   383   7   669   706   5   325   350  12   320   313   2   382   401   1   431   417
  9   525   554  -8   271   247  -2   994   898   9   490   491   6   772   827    -6,0,L         4   452   416   2   411   406
 11   296   308  -6   415   402  -1   278   254  13   266   244   7   423   428 -12   413   388   7   203   198   3   716   714
   -6,-15,L      -4   371   347   0   362   349    -6,-4,L        9   246   214  -8   263   261   8   198   181   4   349   332
  1   449   421  -1   170   136   1   307   310 -12   290   294  10   384   409  -6   628   657  10   390   374   5   554   525
   -6,-14,L       0   617   579   2   209   175 -11   206   231  12   396   419  -4   501   560    -5,-11,L       6   698   704
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  -5,-9,L    -10  344  328  -7  655  660  -5  628  673  -9  316  343  -3  195  192 -10  346  327  -3  772  698
  8  480  480  -9  379  393  -5  480  448  -4 1465 1475  -7  716  760   0  337  337  -9  207  227  -2  367  341
  9  311  332  -8  229  270  -4  602  553  -2  920  828  -5  863  966   2  667  633  -8  716  722  -1  428  401
 11  353  367  -7  760  764  -2  585  534  -1  439  400  -3  360  381   4  368  345  -6  388  390   0  220  191
 12  254  261  -6  176  209  -1  956  898   0  148  121  -1 1249 1330   6  206  188  -4  409  382   1 1269 1213
  -5,-8,L     -5  438  419   0  347  327   1  664  693   1 1905 2039   8  478  473  -2 1021  955   3  834  810
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  4  109  106  -8 1267 1381   10  436  437   -2  619  672   -5  332  388   10  193  230  -11  271  318   12  655  624
  5  863  852  -7  245  259   11  815  785    0  894  884   -4  272  287   12  380  345   -9  360  374   14  293  256
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  7  499  485  -5  620  659   13  747  705    4 2126 2068   -2  515  553  -12  433  499   -7  336  381  -15  247  245
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   -2,-4,L      2 1065  985  -11  545  559   16  508  450    6  405  423   -4  860  916    1  195  194   -5  350  365
-15  227  250   3  753  713  -10  179  231     -1,-18,L      7  359  337   -3  510  540    2 1284 1300   -4  564  593
-13  201  232   4  808  756   -9  525  578   -2  348  395   10  248  221   -1  306  326    3  881  888   -3 1541 1583
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  4 1857 1899
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  3 1037 1062
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  8  861  851
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 12  380  357
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  6  555  543
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  8  170  196
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  3 1214 1230
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 7  272  265      0,-3,L       9  558  564    4  353  356   13  347  315    5 1127 1129   15  449  367
 8 1042 1017    1  458  444   10  974  933    5 1476 1505   14  338  297    6  736  733      0,0,L
 9  883  859    2 1280 1266   12  578  561    6 1002  998   15  239  227    7 2095 2146    2 1856 2049
11  652  616    3  790  779   16  464  377    7  357  363   17  343  253    8  218  205    4 1424 1471
12  683  640    4 1545 1518      0,-2,L       8  151  135      0,-1,L       9  377  359    6 2216 2287
13  196  205    5  726  701    0 1069 1074    9  898  884    1 1925 2047   10  471  429    8 1874 1883
14  543  499    6  817  813    1 1533 1579   10  340  340    2 1532 1572   11  197  172   12  722  668
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Compound:

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  H,-2,-15
1  129  115
  H,-3,-15
1  192  183
  H,-4,-15
1   89   80
  H,-5,-14
1  205  193
  H,-3,-14
1  143  135
  H,-2,-14
1  218  212
2  236  237
  H,-1,-14
3  220  210
2  149  143
1  104  100
  H,1,-14
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1  277  261
0  352  328
  H,2,-14
0  131  111
1  151  143
2  175  153
  H,3,-14
0  199  184
  H,4,-13
1  149  137
0  266  248
  H,3,-13
1   96   98
2  140  127
3  212  190
  H,2,-13
3  222  210
2  231  219
1  282  270
0  351  318
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1  166  160
2  110  109
  H,0,-13
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3  248  245
2  173  166
1   83   58
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2  302  306
3  277  274
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1  236  232
  H,-3,-13
2  151  144
3  199  194
  H,-4,-13
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2  288  274
1  256  260
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  H,-6,-12
1  230  221
2  190  181
  H,-4,-12
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2  217  206
  H,-3,-12
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2  171  164
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1  117  102
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1  463  475
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3  303  301
2  364  360
1  399  399
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 H,-6,-9
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 H,-7,-9
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2  267  257
3  238  230
 H,-8,-9
2  104  108
1  143  139
 H,-9,-8
1  201  197
 H,-8,-8
2  115  115
 H,-7,-8
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4  325  336
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   H,-7,8
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 2  115  107
   H,-6,8
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   H,-5,8
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 2  189  189
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 3  579  594
 4  553  552
 5  198  194
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 3  454  472
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 4  157  163
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 6  363  367
 7  270  273
 8  253  240
 9  229  221
   H,1,8
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 7  140  139
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 3  424  420
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 3  456  463

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 6  500  516
 7  235  238
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 8  226  222
 7  397  379
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 5  387  407
 4  326  344
 3  124  129
 2  159  162
 1  566  576
 0  598  611
   H,4,8
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 4  178  196
 5  149  164
 7  140  137
 8  192  187
 9  206  198
10  188  181
   H,5,8
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 4  428  435
 3  518  507
 2  516  517
 1  277  278
   H,6,8
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 1  261  270
 2   82   79
 4  229  231
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 6  367  379

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 8  316  315
 9  134  128
   H,7,8
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 9  167  167
 8  169  171
 7  151  147
 4  141  137
 3  398  400
 2  470  471
 1  363  366
 0  288  283
   H,8,8
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 2  301  308
 3  357  362
 4  380  381
 5  173  187
 6  169  159
 7  126  136
   H,9,8
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 7  199  193
 6  186  184
 5  234  230
 4  176  173
 3   92  103
 1   85   79
 0  110  112
   H,10,8
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 2  171  166
 3  180  174
 4  116  104
 9  146  142
   H,11,8
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 4  190  186
 3  148  147
 2  111  111

   H,11,9
 3  142  131
   H,10,9
 8  174  164
 7  133  137
 6  134  140
 5   90  105
 4   96   92
   H,9,9
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 3  259  261
 4  241  240
 5  192  180
 6  157  153
 7  164  158
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 9  141  126
 4  123  113
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 2  327  331
 1  265  267
 0  204  202
   H,7,9
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 1  215  218
 2  185  184
 4  111  117
 5  186  183
 6  292  306
 7  308  307
 8  250  248
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 5  375  373
 4  318  335
 3  370  384
 2  328  339
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 5  224  216
 6  106   93
 8  162  161
 9  188  187
10  186  172
   H,4,9
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 9  187  180
 8  197  196
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 6  338  350
 5  236  240
 2  286  297
 1  509  526
 0  602  612
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 3  397  387
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 5  595  637
 6  511  529
 7  201  208
   H,2,9
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 9  169  158
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 7   91   93
 6  127  124
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 3  484  476
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 0  510  517
   H,1,9
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 3  116  121
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7	274	275
8	249	247
9	260	252
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3	431	454
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0	163	167
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2	423	447
3	595	627
4	559	556
5	231	233
H,-2,9		
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7	190	192
6	198	198
5	77	86
3	284	296
2	353	372
1	331	346
H,-3,9		
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3	304	311
4	334	336
5	270	280
6	222	222
7	208	206
H,-4,9		
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5	111	113
4	266	260
3	439	444
2	469	467
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2	292	307
3	186	175
H,-6,9		
3	133	137
H,-7,9		
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2	232	233
H,-6,10		
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2	240	239
3	185	178
H,-5,10		
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1	146	142
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3	191	187
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3	429	451
2	443	470
1	347	366
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4	297	298
5	262	261
6	270	262
7	242	234
H,-1,10		
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7	218	215
6	148	148
5	102	99
4	86	80
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H,0,10		
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3	619	650
4	547	571
5	251	254
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7	204	203
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5	418	435
4	471	488
3	346	362
2	147	145
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H,2,10		
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3	256	257
5	114	115
6	229	233
7	262	262
8	253	247
9	245	235
H,3,10		
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9	120	119
8	102	102
6	96	109
5	394	407
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3	471	466
2	460	472
1	449	470
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H,4,10		
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3	300	312
4	424	439
5	560	547
6	423	431
7	179	188
H,5,10		
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9	178	182
8	183	194
7	240	238
6	272	274
5	131	133
2	240	249
1	456	467
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H,6,10		
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1	385	400
2	403	400
3	283	285
4	253	260
5	239	245
6	157	157
8	111	120
9	142	141
10	126	141
H,7,10		
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5	311	322
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3	250	245
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H,8,10		
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5	122	108
6	217	209
7	229	230
8	178	176
9	115	104
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2	243	251
1	199	201
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H,10,10		
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4	160	156
5	152	150
6	144	133
7	148	142
H,11,10		
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H,10,11		
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H,9,11		
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6	135	130
7	156	156
H,8,11		
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6	261	262
5	253	255
4	184	183
3	171	174
2	96	102
H,7,11		
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1	278	281
2	288	289
3	238	234
4	225	233
5	242	252
6	130	133
H,6,11		
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9	163	174
8	182	174
7	189	189
6	182	186
5	90	82
2	197	195
1	356	364
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H,5,11		
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5	382	383
6	317	304
7	164	153
8	113	115
9	97	99
H,4,11		
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6	121	115
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4	499	500
3	455	479
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H,3,11		
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3	265	273
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7	210	207
8	229	230
9	199	206
H,2,11		
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8	246	239
7	217	224
6	277	275
5	323	342
4	366	368
3	298	298
0	291	309
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3	559	568
4	475	477
5	242	250
7	88	72
H,0,11		
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3	381	400
2	467	472
1	439	450
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3	143	147
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5	232	224
6	268	266
7	247	244
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6	189	187
5	187	200
4	273	280
3	387	411
2	461	479
1	307	311
H,-3,11		
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2	383	385
3	193	207
H,-4,11		
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1	205	211
H,-5,11		
1	164	170
2	192	194
3	195	190
H,-6,11		
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H,-5,12		
2	188	196
1	275	269
H,-4,12		
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2	149	145
3	172	172
4	189	190
H,-3,12		
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4	103	118
1	256	251
H,-2,12		
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2	371	380
3	212	216
H,-1,12		
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6	229	228
5	234	236
4	228	236
3	346	355
2	341	344
1	238	235
H,0,12		
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1	165	162
2	113	111
4	184	190
5	192	196
6	265	263
7	263	258
H,1,12		
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3	389	392
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H,2,12		
0	140	133

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 7  247  249
 6  247  248
 5  240  245
 4  299  296
 0  218  227
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 3  271  270
 4  239  224
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 7  137  141
 8  181  186
 9  168  166
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 3  356  363
 2  261  273
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 6  236  238
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 2  172  181
 3  170  164
 4  183  180
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 6  194  191
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 4  134  129
 3  113  111
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 5  172  167
 6  101  107
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 6  225  222
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 2  243  243
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 3  216  212
 4  151  145
 5  114  117
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 2  103  110
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   H,-4,13

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 3  239  229
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 4  115   90
   H,7,15
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 3  189  195
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   H,6,15

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 5  114  101
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 6  174  163
 7  176  171
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 5   93   97
 6  155  132
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 3  168  158
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 4  186  184

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 1  205  199
   H,2,16
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 3  181  164
 4  194  186
 5  167  177
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 4  146  127
 3   94  102
   H,4,16
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 2  151  152
   H,5,16
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 3  163  164
 2  184  182
   H,6,16
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 4  123  125
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Compound:

$[PtCl(\mu\text{-}Ph_2PCH_2PPh_2)]_2$

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  H,-4,3
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  H,-4,7
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  H,-3,-18
13  370  390
  H,-3,-14
13  343  415
  H,-3,-12
13  430  339
  H,-3,-8
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  H,-3,-7
13  766  685
  H,-3,-6
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  H,-3,-4
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13  598  590
  H,-3,4
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  H,-3,6
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14  521  441
  H,-1,-14
14  448  457
  H,-1,-13
14  978  905
  H,-1,-12
14  421  375
  H,-1,-11
14  958  946
  H,-1,-9
14  657  713
  H,-1,-1
14  659  704
  H,-1,0
14  491  474
  H,-1,1
14  613  655
  H,-1,5
14  443  419
  H,0,-16
14  579  506
  H,0,-14
14  591  547
  H,0,-10
14  786  819
  H,0,-8
14 1197 1245
  H,0,-6
14 1203 1174
  H,0,-2
14  356  417
  H,0,2
14 1005 1043
  H,0,4
14 1421 1536
  H,0,6
14 1535 1790
  H,-7,-9
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  H,-6,-11
15  489  496
  H,-6,-4
15  689  640
  H,-6,-2
15  833  860
  H,-5,-11
15  387  343
  H,-5,-10
15  457  406
  H,-5,-9
15  559  543
  H,-5,0
15  503  492
  H,-4,-13
15 1187 1147
  H,-4,-11
15  917  938
  H,-4,-8
15  361  273
  H,-4,-5
15  462  562
  H,-4,-3
15  875  922
  H,-4,-2
15  468  476
  H,-4,-1
15  902  938
  H,-4,1
15  651  667
  H,-3,-10
15  621  604
  H,-3,-8
15  329  275
  H,-3,0
15  574  630
  H,-2,-15
15  857  826
  H,-2,-14
15  885  852
  H,-2,-13
15  995  908
  H,-2,-12
15  850  786
  H,-2,-11
15  673  619
  H,-2,-4
15  687  621
  H,-2,-3
15  737  787
  H,-2,-2
15  667  646
  H,-2,-1
15  927  915
  H,-2,0
15  445  486
  H,-2,1
15  551  570
  H,-1,-15
15  580  519
  H,-1,1
15  937  959
```

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  H,-6,-6
12 1149 1110
  H,-6,-5
12 1038 1036
  H,-6,-4
12 1825 1774
  H,-6,-3
12 1041 1027
  H,-6,-2
12 1199 1156
  H,-6,-1
12  966  942
   H,-6,1
12  584  506
   H,-6,2
12  501  521
   H,-6,3
12  318  415
   H,-6,5
12  738  768
   H,-6,6
12  730  985
   H,-6,7
12  459  534
   H,-6,8
12 1145 1267
  H,-5,-20
12  898  763
  H,-5,-19
12  684  549
  H,-5,-18
12  855  766
  H,-5,-16
12  546  498
  H,-5,-10
12  724  724
  H,-5,-9
12  543  516
  H,-5,-8
12 1075 1008
  H,-5,-7
```

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12  846  885
  H,-5,-5
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  H,-5,-1
12  852  873
    H,-5,0
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    H,-5,1
12  700  614
    H,-5,4
12  557  469
    H,-5,5
12  394  419
    H,-5,8
12  513  561
    H,-5,9
12  389  459
   H,-5,10
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  H,-4,-17
12  805  680
  H,-4,-15
12  716  659
  H,-4,-11
12  407  307
  H,-4,-7
12 1221 1192
  H,-4,-5
12 2321 2218
  H,-4,-3
12 2374 2306
  H,-4,-1
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12  396  405
    H,-4,5
12 1030 1126
    H,-4,7
12 1383 1509
```

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12 1107  948
  H,-3,-18
12 1186 1037
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12  386  337
  H,-3,-16
12  491  456
  H,-3,-10
12  874  723
  H,-3,-9
12  449  414
  H,-3,-8
12  950  946
  H,-3,-6
12 1038 1049
  H,-3,-2
12 1144 1121
    H,-3,0
12  896  895
    H,-3,8
12  410  366
    H,-3,9
12  500  275
   H,-3,10
12 1218 1339
  H,-2,-18
12  365  379
  H,-2,-17
12  835  754
  H,-2,-16
12  676  556
  H,-2,-15
12  618  559
  H,-2,-14
12  539  522
  H,-2,-13
12  344  387
  H,-2,-11
12  389  370
  H,-2,-8
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12 1096 1085
  H,-2,-6
12 1477 1410
  H,-2,-5
12 1919 1894
  H,-2,-4
12 1904 1875
  H,-2,-3
12 2040 2054
  H,-2,-2
12 1272 1354
  H,-2,-1
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    H,-2,4
12  694  729
    H,-2,5
12  659  721
    H,-2,6
12  893  928
    H,-2,7
12 1057 1102
    H,-2,8
12  484  767
    H,-2,9
12  960 1138
   H,-2,10
12  581  467
  H,-1,-21
12  868  718
  H,-1,-20
12  627  376
  H,-1,-19
12 1250 1107
  H,-1,-18
12  512  425
  H,-1,-17
12 1030  932
  H,-1,-11
12  686  585
  H,-1,-9
12  988  980
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  H,-1,-7
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  H,-1,-6
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  H,-1,-5
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12  784  758
  H,-1,-2
12  752  697
    H,-1,0
12  449  387
    H,-1,3
12  417  515
    H,-1,9
12  688  834
   H,-1,10
12  460  554
   H,-1,11
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12  775  642
  H,0,-16
12 1296 1107
   H,0,-8
12  822  796
   H,0,-6
12 2060 1954
   H,0,-4
12 3039 2935
   H,0,-2
12 1686 1597
    H,0,4
12  877  937
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12 1262 1356
    H,0,8
12 1290 1453
   H,0,10
12  841  942
  H,-11,-8
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13 1047  970
  H,-11,-4
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  H,-10,-13
13  361  338
  H,-10,-12
13  374  347
  H,-10,-11
13  886  912
  H,-10,-9
13 1031  998
  H,-10,-7
13  608  478
  H,-9,-14
13  519  458
  H,-9,-12
13  581  581
  H,-9,-8
13  738  690
  H,-9,-7
13  877  855
  H,-9,-6
13  786  815
  H,-9,-5
13  717  765
  H,-9,-3
13  617  642
    H,-9,0
13  461  431
    H,-9,1
13  424  428
    H,-9,3
13  542  570
  H,-8,-12
13  598  607
  H,-8,-11
13  411  317
  H,-8,-10
13 1060 1076
  H,-8,-9
13  473  445
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13  399  442
    H,-8,4
13  337  399
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13  382  375
  H,-7,-8
13  427  322
  H,-7,-7
13 1441 1436
  H,-7,-5
13 1259 1287
  H,-7,-2
13  617  670
    H,-7,0
13  670  572
    H,-7,3
13  528  595
    H,-7,5
13 1104 1210
  H,-6,-12
13  731  744
  H,-6,-11
13  395  397
  H,-6,-10
13 1109 1096
  H,-6,-9
13  742  589
  H,-6,-8
13 1040  978
  H,-6,-6
13  317  382
  H,-6,-4
13  440  358
    H,-6,0
13  344  295
    H,-6,2
13  521  596
    H,-6,3
13  322  377
    H,-6,4
```

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13  358  329
  H,-5,-13
13  520  538
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13  334  312
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13 1220 1246
  H,-5,-7
13  901  867
  H,-5,-6
13 1582 1602
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13 1083  962
  H,-5,-4
13 1128 1108
  H,-5,-2
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  H,-5,-1
13  517  583
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13  399  275
    H,-5,2
13  355  394
    H,-5,4
13 1090 1205
    H,-5,5
13  574  588
    H,-5,6
13 1276 1417
    H,-5,7
13  812  912
  H,-4,-19
13  554  490
  H,-4,-13
13  339  394
  H,-4,-12
13  336  333
  H,-4,-11
13  853  948
```

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  H,-5,-6     11  702  679     H,-3,8      11  927  945     H,-1,9      12  838  878    H,-10,3     12  654  693
11 1184 1179     H,-4,-1    11  976 1066     H,-2,4     11  698  832    H,-12,-4    12  360  337     H,-8,3
  H,-5,-5     11 1112 1023    H,-3,10      11  463  437    H,-1,10      12  577  557    H,-9,-11    12  516  529
11  673  587     H,-4,0     11  897  970     H,-2,7     11  502  638    H,-12,-3    12  417  468     H,-8,6
  H,-5,-4     11  786  689    H,-3,12      11  446  495    H,-1,11      12  917  921    H,-9,-9     12  888 1025
11 1489 1479     H,-4,1     11  372  474    H,-2,10     11  674  739    H,-12,-2    12  601  612    H,-7,-19
  H,-5,-3     11 1978 1926    H,-2,-21     11  378  393    H,0,-20      12  460  317    H,-9,-8     12  856  748
11 1563 1465     H,-4,3     11  408  334    H,-2,11     11  941  808    H,-12,-1    12  625  630    H,-7,-17
  H,-5,-2     11 1039 1063    H,-2,-20     11  843  890    H,0,-18      12  469  550    H,-9,-7     12  624  507
11 1664 1582     H,-4,6     11  617  541    H,-2,12     11 1652 1479    H,-12,1     12  463  442    H,-7,-11
  H,-5,-1     11  329  366    H,-2,-19     11  637  789    H,0,-16      12  356  307    H,-9,-6     12  342  394
11 1168 1165    H,-4,11     11  912  731    H,-2,13     11 1376 1358    H,-11,-10   12  447  450    H,-7,-9
   H,-5,0     11  813  896    H,-2,-18     11 1065 1291    H,0,-14      12  473  630    H,-9,-2     12  856  835
11 1487 1501    H,-3,-16    11 1136  978    H,-1,-17    11  478  598    H,-11,-8    12  515  525    H,-7,-7
   H,-5,3     11 1277 1120    H,-2,-17     11  873  797    H,0,-8       12  653  684    H,-9,-1     12  932  926
11  575  593    H,-3,-15    11 1163 1042    H,-1,-16    11  832  814    H,-11,-6    12  511  500    H,-7,-5
   H,-5,6     11  411  453    H,-2,-16     11  699  597    H,0,-6       12  456  471     H,-9,0     12  579  587
11  639  669    H,-3,-14    11  920  880    H,-1,-15    11 1597 1379    H,-11,-2    12  919  948    H,-7,-3
   H,-5,7     11 1404 1327    H,-2,-15     11 1444 1269    H,0,-4       12  815  595     H,-9,4     12  459  369
11  456  527    H,-3,-13    11  472  477    H,-1,-14    11  893  890    H,-11,0     12  566  606    H,-7,-1
   H,-5,8     11  452  486    H,-2,-10     11  566  582    H,0,-2       12  660  651     H,-9,6     12 1163 1137
11  633  663    H,-3,-7     11  333  418    H,-1,-13    11  711  751    H,-10,-11   12  485  429     H,-7,1
   H,-5,9     11  360  310    H,-2,-8      11  888  795    H,0,0        12  388  425    H,-8,-18    12  649  686
11  729  804    H,-3,-6     11  835  832    H,-1,-7     11 1651 1548    H,-10,-10   12  378  316     H,-7,5
  H,-5,11     11  999  926    H,-2,-7      11  486  365    H,0,2        12  476  423    H,-8,-16    12  423  441
11  507  609    H,-3,-5     11  762  734    H,-1,-5     11 1686 1633    H,-10,-7    12  515  470     H,-7,8
  H,-4,-21    11  618  683    H,-2,-6      11 1680 1596    H,0,4        12  635  675    H,-8,-14    12  376  398
11  590  481    H,-3,-4     11  865  805    H,-1,-4     11  729  645    H,-10,-6    12  458  266    H,-6,-18
  H,-4,-19    11 2268 2153    H,-2,-5      11  970  862    H,0,10       12  474  464    H,-8,-11    12  636  582
11 1214 1085    H,-3,-3     11 1277 1209    H,-1,-3     11  518  528    H,-10,-5    12  524  438    H,-6,-17
  H,-4,-17    11  604  577    H,-2,-4      11 2299 2182    H,0,12       12 1083 1057    H,-8,-8     12  321  225
11 1401 1318    H,-3,-2     11  459  425    H,-1,-2     11 1327 1511    H,-10,-4    12  599  643    H,-6,-16
  H,-4,-15    11 2503 2420    H,-2,-2      11 1283 1244    H,-13,-7     12  645  590    H,-8,-6     12  541  453
11  800  855    H,-3,-1     11  370  408    H,-1,-1     12  648  650    H,-10,-3    12 1258 1238    H,-6,-15
  H,-4,-9     11  538  541    H,-2,-1      11 1845 1809    H,-13,-5     12 1117 1110    H,-8,-5     12  490  424
11  484  368     H,-3,0     11 1184 1166     H,-1,0     12  475  346    H,-10,-2    12  359  329    H,-6,-12
  H,-4,-8     11 1485 1446     H,-2,0      11  559  524    H,-12,-10    12  556  577    H,-8,-4     12  413  384
11  301  283     H,-3,1     11 1045 1019     H,-1,1     12  382  460    H,-10,-1    12 1640 1592    H,-6,-9
  H,-4,-7     11  370  340     H,-2,1      11  946  922    H,-12,-7     12  600  486    H,-8,-2     12  411  377
11  818  983     H,-3,5     11 1398 1372     H,-1,7     12  437  484    H,-10,0     12 1363 1297    H,-6,-8
  H,-4,-5     11  391  379     H,-2,2      11  622  686    H,-12,-6     12  648  646     H,-8,0     12  339  275
```

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10  559  503    H,-1,-10    10 1598 1655    H,-13,2     11  776  600    H,-9,2      11  450  476    H,-6,-12
  H,-2,-3     10  391  406    H,0,-10     11  601  564    H,-10,-5    11  454  401    H,-8,10     11  572  547
10 1257 1174    H,-1,-9     10  818  862    H,-13,3     11  754  790    H,-9,3      11  564  649    H,-6,-10
  H,-2,-2     10  714  725    H,0,-4      11  720  453    H,-10,-3    11  397  344    H,-7,-17    11  334  288
10  765  717    H,-1,-6     10  988 1027    H,-12,-12   11  427  465    H,-9,4      11  486  506    H,-6,-9
  H,-2,-1     10  423  396    H,0,-2      11  508  498    H,-10,-2    11  791  774    H,-7,-15    11  397  478
10 1847 1672    H,-1,-5     10 1727 1650    H,-12,-6    11  512  454    H,-9,7      11 1072 1002    H,-6,-8
  H,-2,0      10  600  625    H,0,-1      11  535  565    H,-10,-1    11  549  521    H,-7,-13    11  431  436
10 1396 1352    H,-1,-4     10  342    0    H,-12,-4    11  657  734    H,-9,8      11  988  966    H,-6,-7
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10 1606 1514    H,-1,-3     10 1905 1766    H,-12,-1    11 1599 1577    H,-8,-18    11  424  305    H,-6,-6
  H,-2,2      10  513  466    H,0,2       11  828  762    H,-10,3     11  815  741    H,-7,-7     11  983  935
10  987  934    H,-1,-1     10 1507 1542    H,-12,0     11  788  856    H,-8,-16    11  434  354    H,-6,-4
  H,-2,3      10  723  528    H,0,6       11  672  705    H,-10,5     11  876  852    H,-7,-6     11  600  658
10  674  580    H,-1,0      10  732  746    H,-12,1     11  410  375    H,-8,-13    11  353  318    H,-6,-3
  H,-2,5      10  571  523    H,0,10      11  799  816    H,-10,8     11  386  466    H,-7,-5     11  449  360
10  569  592    H,-1,1      10  887  953    H,-12,3     11  381  408    H,-8,-11    11 1350 1323    H,-6,-2
  H,-2,6      10 1776 1704    H,0,12      11  498  582    H,-9,-16    11  576  628    H,-7,-3     11  346  410
10  519  459    H,-1,2      10  704  784    H,-11,-16   11  387  395    H,-8,-8     11 2016 2013    H,-6,-1
  H,-2,7      10  971  923    H,-14,-6    11  592  577    H,-9,-15    11  463  442    H,-7,-1     11  518  542
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  H,-2,9      10 2809 2846    H,-14,-4    11  645  593    H,-9,-14    11  387  341    H,-7,1      11 1595 1605
10  431  573    H,-1,4      11  394  436    H,-11,-9    11  657  689    H,-8,-6     11  759  734    H,-6,1
  H,-2,10     10 1075 1062    H,-14,-3    11  565  570    H,-9,-13    11  676  616    H,-7,2      11  504  545
10  464  324    H,-1,5      11  666  367    H,-11,-6    11  664  597    H,-8,-5     11  615  602    H,-6,2
  H,-2,11     10 1968 2086    H,-13,-10   11  451  436    H,-9,-7     11  692  648    H,-7,7      11 1684 1653
10  612  724    H,-1,6      11  449  531    H,-11,-4    11  501  535    H,-8,-3     11  914  975    H,-6,3
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  H,-1,-19    10  391  301    H,-13,-8    11 1378 1627    H,-9,-5     11  489  424    H,-7,11     11  446  390
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  H,-1,-17    10  450  536    H,-13,-5    11 1158 1082    H,-9,-4     11 1553 1548    H,-6,-20    11  431  503
10 1423 1261    H,-1,13     11  423  474    H,-11,3     11  863  829    H,-8,1      11  561  469    H,-5,-16
  H,-1,-16    10 1056 1202    H,-13,-4    11  868  890    H,-9,-3     11 1129 1154    H,-6,-19    11  942  807
10  675  712    H,-1,14     11  835  822    H,-11,5     11 1279 1282    H,-8,2      11  603  553    H,-5,-15
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10 1453 1374    H,-1,15     11 1065 1059    H,-11,6     11 1617 1484    H,-8,3      11  876  832    H,-5,-14
  H,-1,-14    10 1247 1481    H,-13,-2    11  538  387    H,-9,-1     11  337  309    H,-6,-17    11 1134 1059
10  570  548    H,0,-16     11 1009  855    H,-10,-15   11 1313 1238    H,-8,4      11  594  536    H,-5,-13
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```

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  H,-11,0     10  520  460    H,-8,-11    10  394  396    H,-6,-5     10  445  370    H,-4,-4     10 1162 1191
10 1016  971    H,-9,-16    10  605  484    H,-7,-5     10  680  645    H,-5,-5     10  402  421     H,-3,0
  H,-11,2     10  782  699    H,-8,-10    10  532  680    H,-6,-4     10  631  666    H,-4,-3     10 1112 1089
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Compound:

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 -5  555  503  11 1369 1296   8 1576 1510   4  640  810  26  494  519  17  735  771  25  730  798   7  653  653
 -4  384  291  12  654  552   9 1192 1142   5  230  149     0,7,L     18  632  747     0,11,L     9  366  443
 -1  285  262  13  661  609  10 3106 2892   6 2691 3270   1 1611 1671  19  620  655   1  700  746  10  486  495
```

```
   0,13,L      21  728  789  15  483  477   5  404  384  16  319  377   5  687  641     0,17,L       0,18,L
12  343  330      0,14,L     17 1071 1104   6  713  705     0,16,L      8  698  686   5  676  651   0  575  533
13  356  410    1 1892 1894  19  425  428   7  533  524   0  754  702   9  565  536   6  499  487   1  364  320
14  290  325    5 1105 1129     0,15,L      8  342  326   1  328  310  10  507  505   7  538  518   3  370  360
15  930  955    6  313  329   1  826  824  10  516  533   2  667  628  13  470  490  10  494  500   4  451  398
19  857  955    7  407  371   3 1268 1238  12  776  779   3  498  497  14  806  837  11  792  745   7  590  552
20  554  558   13  378  322   4  578  578  15  588  571   4  408  433
```