CRYSTAL AND MOLECULAR STRUCTURES
OF SOME ORGANOMETALLIC COMPOUNDS

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by

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SUMMARY

In Part I of this thesis, I describe some aspects of crystal structure analysis; in particular those methods employed in this research.

In Part II, the structural investigation of three organometallic complexes by X-ray diffraction is reported, viz. (N-ethoxycarbonyl-3-formylazepine)tricarbonyliron, π-cyclopentadienyl-cis-dicarbonylbromo(triphenylphosphine)molybdenum - methylene chloride and π-cyclopentadienyl-trans-dicarbonyliodo-(phenyl isocyanide)molybdenum.

Previous X-ray analyses of azepine-tricarbonyliron complexes have shown that the geometry within the azepine ring is modified by substitution, therefore the structure elucidation of (N-ethoxycarbonyl-3-formylazepine)tricarbonyliron was undertaken in order to examine the geometry in a 1,3-substituted complex. Bond length variations consistent with those in the other complexes have been found.

The structure determinations of the molybdenum complexes were undertaken to augment the already considerable structural comparisons possible among complexes of the type $(\pi\text{-}C_5H_5)MoL_2XY$. In the analysis of cis-$(\pi\text{-}C_5H_5)Mo(CO)_2(PPh_3)Br$, halo/carbonyl disorder has prevented the determination of meaningful Mo - CO distances, but sensible values involving the other ligands have been obtained. The interest in the structure of trans-$(\pi\text{-}C_5H_5)Mo(CO)_2(CNPh)I$ was centred on the phenyl isocyanide ligand, for which an almost linear Mo - C - N - C geometry was found, with a Mo - CNPh bond length consistent with the π-accepting ability of the phenyl isocyanide ligand being nearly comparable with that of the carbonyl ligands.

In Part III, the characterization by X-ray analysis of a chromium and three iron organometallic complexes is reported.

The analysis of the chromium complex was undertaken to determine whether the monomeric formulation, $(\pi\text{-}C_5H_5)Cr(NO)(NPh_2)I$, or a formulation involving weak iodine or nitrogen bridges was correct. The complex was found to be monomeric and structural comparisons have been made with related chromium-cyclopentadienyl-nitrosyl complexes.

An X-ray analysis was carried out on the hexafluorophosphate salt of a stable intermediate isolated from the Friedel-Crafts acylation of (hexa-2,4-diene)tricarbonyliron, in order to assert whether the iron atom directs the entering electrophile endo or exo to the organic ligand. The configuration of the cation of the intermediate, tricarbonyl(2——4-η-hepten-5-methyl-6-one)iron, was found to be that resulting from endo-acylation.

A product of the photochemical reaction of benzyldicarbonyl-(π-cyclopentadienyl)iron with triphenylphosphine has been examined by X-ray diffraction and found to be (1-exo-benzylcyclopenta-2,4-diene)-dicarbonyl(triphenylphosphine)iron, in contradiction to the spectroscopic assignment. The tetracyanoethylene adduct of this product has been characterized by a further X-ray analysis as dicarbonyl(π-cyclopentadienyl)(triphenylphosphine)iron 1,1,2,3,3-pentacyanopropenide. As a consequence of disorder, accurate dimensions for the anion could not be obtained, but structural comparisons have been possible between the cation, formally a Fe^{II} complex, and its closely related precursor, formally a Fe^{0} complex. The most notable feature is a longer, ca. 0.03 Å, Fe - P bond length in the Fe^{II} complex.

CONTENTS

CHAPTER 1

CHAPTER 1: (N-ETHOXYCARBONYL-3-FORMYLAZEPINE)TRICARBONYLIRON

CHAPTER 2: TWO MOLYBDENUM COMPLEXES

π-CYCLOPENTADIENYL-CIS-DICARBONYLBROMO-(TRIPHENYLPHOSPHINE)MOLYBDENUM - METHYLENE CHLORIDE

Page

π-CYCLOPENTADIENYL-TRANS-DICARBONYLIODO-(PHENYL ISOCYANIDE)MOLYBDENUM

CHAPTER 1: π-CYCLOPENTADIENYL(DIPHENYLAMIDO)IODONITROSYLCHROMIUM

CHAPTER 2: TRICARBONYL(2—4-η-HEPTEN-5-METHYL-6-ONE)IRON HEXAFLUOROPHOSPHATE

Page

CHAPTER 3: TWO IRON COMPLEXES

PART I

SOME ASPECTS OF CRYSTAL STRUCTURE ANALYSIS

CHAPTER I

I.1.1 HISTORICAL

Steno's observation in 1669, of the constancy of angles between corresponding faces in quartz, stimulated thought on the geometrical theory of crystallography, and this was developed and completed some years before Röntgen's discovery of X-rays in 1895. The nature of this very penetrating radiation was unknown until in 1912, Freidrich and Knipping, following the suggestion of von Laue, showed that a crystal could act as a three-dimensional diffraction grating for X-rays. This not only demonstrated that X-rays could be considered as electromagnetic radiation of wavelength similar to the interatomic spacing in a crystal lattice, but also initiated the growth of a powerful physical technique for investigating the internal structure of crystals on an atomic scale.

Bragg's Law was published in the following year, and gradually other important advances, both in theory and technique, have resulted in a greater number of structures being determined, greater accuracy being achieved and greater complexity in structure being overcome.

I.1.2 DATA REDUCTION

The observed structure amplitude, $|F_{o(hk\ell)}|$, of the reflection (hkℓ) is related to the intensity, $I_{(hk\ell)}$, by the equation:

$$|F_{o(hk\ell)}| = \sqrt{\frac{I_{(hk\ell)}}{L.p}} \qquad (1)$$

where both L, the Lorentz factor, and p, the polarisation factor, are dependent on the individual reflection and the method of data collection. The Lorentz factor allows for the relative time each crystal plane is in a reflecting position and, for normal beam diffractometers, is given by:

$$L = \frac{1}{\sin 2\theta_{(hk\ell)}} \qquad (2)$$

where $\theta_{(hk\ell)}$ is the Bragg angle of the reflection (hkℓ).
The polarisation factor given by:

$$p = \frac{1 + \cos^2 2\theta_{(hk\ell)}}{2} \qquad (3)$$

allows for the partial polarisation of the reflected beam. However, if the X-ray beam has been reflected from a crystal monochromator, the incident beam is also partially polarised and the modified equation is:

$$p = \frac{|\cos 2\theta_M| + \cos^2 2\theta_{(hk\ell)}}{|\cos 2\theta_M| + 1} \qquad (4)$$

where θ_M is the Bragg angle of the reflecting family of planes of the crystal monochromator.

Two other factors, namely absorption and extinction, reduce the reflected beam intensity and failure to allow for either of these effects, if they are significantly present, results in a systematic lowering of the temperature factors and a raising of the standard deviations.

X-rays are absorbed to a considerable extent even in the small crystals used for X-ray diffraction, but this can be allowed for when the crystal dimensions are known. If the original beam has intensity I_o, then the intensity I after passing through a thickness t of the crystal is given by:

$$I = I_o e^{-\mu t} \tag{5}$$

where μ is the linear absorption coefficient and is dependent on the atoms in the crystal and the X-ray wavelength.

Extinction is caused by a transfer of energy between the incident and diffracted beams and depends partly on the size and alignment of the mosaic blocks in the crystal. These are difficult to calculate, therefore extinction is usually allowed for, either by removing the extinct reflections or by the more satisfactory method of including an extinction parameter in the least-squares refinement.

I.1.3 THE STRUCTURE FACTOR AND THE ELECTRON DENSITY DISTRIBUTION

The structure factor, $F_{(hk\ell)}$, is defined by the complex equation:

$$F_{(hk\ell)} = \sum_{j=1}^{N} f_j \exp\left[2\pi i(hx_j + ky_j + \ell z_j)\right] \qquad (6)$$

where the summation is over all the atoms in the unit cell and f_j is the atomic scattering factor of the j^{th} atom whose fractional coordinates are (x_j, y_j, z_j). Neglecting anomalous scattering, then the atomic scattering factors can be represented by real numbers and equation (6), when reduced into real and imaginary parts, becomes:

$$F_{(hk\ell)} = \sum_{j=1}^{N} f_j \cos 2\pi(hx_j + ky_j + \ell z_j) + i\sum_{j=1}^{N} f_j \sin 2\pi(hx_j + ky_j + \ell z_j) \qquad (7)$$

i.e.
$$F_{(hk\ell)} = A_{(hk\ell)} + iB_{(hk\ell)} \qquad (8)$$

Another equivalent expression is:

$$F_{(hk\ell)} = \left|F_{(hk\ell)}\right| \exp\left[i\alpha_{(hk\ell)}\right] \qquad (9)$$

where the structure amplitude $\left|F_{(hk\ell)}\right|$ and the phase $\alpha_{(hk\ell)}$ are related to $A_{(hk\ell)}$ and $B_{(hk\ell)}$ by the equations:

$$\left|F_{(hk\ell)}\right|^2 = A^2_{(hk\ell)} + B^2_{(hk\ell)} \qquad (10)$$

and
$$\alpha_{(hk\ell)} = \tan^{-1}\left(\frac{B_{(hk\ell)}}{A_{(hk\ell)}}\right) \qquad (11)$$

The structure of a crystal is periodic in three dimensions, therefore the electron density, ρ, at the point with fractional coordinates (x, y, z) can be represented by a three-dimensional Fourier series. It can be shown that the coefficients of this Fourier series are directly related to the set of structure factors $F_{(hk\ell)}$ resulting in the following expression:

$$\rho_{(x,\, y,\, z)} = \frac{1}{V}\sum_{h=-\infty}^{\infty}\sum_{k=-\infty}^{\infty}\sum_{\ell=-\infty}^{\infty} F_{(hk\ell)} \exp\left[-2\pi i(hx + ky + \ell z)\right] \qquad (12)$$

where V is the volume of the direct cell. This can be shown to reduce to:

$$\rho_{(x,\ y,\ z)} = \frac{1}{V}\sum_{-\infty}^{\infty}{}_{h}\sum_{-\infty}^{\infty}{}_{k}\sum_{-\infty}^{\infty}{}_{\ell}\left|F_{(hk\ell)}\right|\cos\left[2\pi(hx + ky + \ell z) - \alpha_{(hk\ell)}\right] \qquad (13)$$

I.1.4 THE PATTERSON FUNCTION AND THE HEAVY ATOM METHOD

Examination of equations (12) and (13) reveals that electron-density calculations would be routine if the relative phases and not merely the structure amplitudes could be determined directly from the intensity measurements. However, Patterson (1934/36) realised that knowledge of the structure amplitudes is sufficient to enable a Fourier series representing the interatomic vectors in a crystal to be calculated. The value, P, of this Fourier series at the point with fractional coordinates (u, v, w) is given by:

$$P_{(u,\ v,\ w)} = \frac{1}{V}\sum_{h=-\infty}^{\infty}\sum_{k=-\infty}^{\infty}\sum_{\ell=-\infty}^{\infty}\left|F_{(hk\ell)}\right|^{2}\exp\left[2\pi i(hu + kv + \ell w)\right] \qquad (14)$$

which is equivalent to:

$$P_{(u,\ v,\ w)} = \frac{1}{V}\sum_{h=-\infty}^{\infty}\sum_{k=-\infty}^{\infty}\sum_{\ell=-\infty}^{\infty}\left|F_{(hk\ell)}\right|^{2}\cos 2\pi(hu + kv + \ell w) \qquad (15)$$

A cell containing N atoms will generate $N^2 - N$ non-origin peaks, with height proportional to the product of the atomic numbers of the atoms involved and maximum width twice that of the corresponding electron density Fourier peaks. For those reasons, peaks tend to overlap and normally the only readily distinguishable features in a sea of vector density are the vectors between the heavy atoms. Hence only the position of the heavy atom/atoms can be found quickly and easily, but this is normally a sufficient foundation to enable the rest of the structure to be built up by the following iterative method.

The phases calculated using the dominant scatterers in the cell (i.e. the heavy atoms) approximate to the true phases. Therefore, an electron density distribution based on the calculated phases and the observed structure amplitudes should reveal the positions of some or all of the light atoms. This new set of atoms determines a more correct set of phases, and the procedure can be repeated.

An indication of both the number of iterations which will be required to build up the whole structure and the likely final accuracy of the light atom positions, can be obtained from an examination of the ratio $\dfrac{\sum_{\text{heavy atoms}} Z^2}{\sum_{\text{light atoms}} Z^2}$, where Z is the atomic number. As the ratio increases beyond 1 the heavy atom dominance increases, with the result that, although most of the observations will be phased correctly, comparison between the observed and calculated structure factors to obtain the position of the light atoms will be less meaningful. However, if the ratio is less than 1, more accurate final parameters for the light atoms will be obtained, but the interpretation of the Patterson function and the subsequent electron density distributions will be more difficult, and several iterations will be required to produce parameters good enough to begin least-squares refinement.

I.1.5 LEAST-SQUARES REFINEMENT

In order to improve the agreement between the observed and calculated structure factors, the n parameters (p_1, p_2, - - - - p_n), which occur in the structure factor calculation, are adjusted by the method of least-squares. The function most commonly minimised is:

$$R = \sum_{r=1}^{m} w_r(|F_o| - |F_c|)^2 = \sum_{r=1}^{m} w_r\Delta^2 \tag{16}$$

where the summation is over the set of m observations and w_r is a weight for each term. Minimisation is achieved by taking the derivative with respect to each of the parameters and equating to zero. This gives the following n equations:

$$\sum_{r=1}^{m} w_r\Delta\frac{\partial|F_c|}{\partial p_j} = 0 \quad (j = 1, 2, - - - - n) \tag{17}$$

Δ can be expanded as a function of the parameters by a Taylor series, and if the trial set of parameters is close to the correct values, the terms higher than first derivative of $|F_c|$ can be neglected, giving:

$$\Delta(\underset{\sim}{p} + \underset{\sim}{\epsilon}) = \Delta(\underset{\sim}{p}) - \sum_{i=1}^{n}\epsilon_i\frac{\partial|F_c|}{\partial p_i} \tag{18}$$

where $\underset{\sim}{p}$ and $\underset{\sim}{\epsilon}$ stand for the whole set of parameters and changes. Substituting (18) into (17) gives n equations in n unknowns. These are called the normal equations and can be expressed as:

$$\sum_{i=1}^{n}\sum_{r=1}^{m} w_r\frac{\partial|F_c|}{\partial p_i}\cdot\frac{\partial|F_c|}{\partial p_j}\epsilon_i = \sum_{r=1}^{m} w_r\Delta\frac{\partial|F_c|}{\partial p_j} \tag{19}$$

or in matrix form:

$$A.X = B \tag{20}$$

where,

A is a nxn matrix with $a_{ij} = \sum_{r=1}^{m} w_r\frac{\partial|F_c|}{\partial p_i}\cdot\frac{\partial|F_c|}{\partial p_j}$

X is a vector of order n with $x_j = \epsilon_i$

B is a vector of order n with $b_j = \sum_{r=1}^{m} w_r \Delta \frac{\partial |F_c|}{\partial p_j}$

For equation (20) to have a solution, A must possess an inverse A^{-1} such that:

$$X = A^{-1}.B \tag{21}$$

The normal equations can therefore be solved but, because they were derived using a truncated Taylor series, exact adjustments are not calculated and it is necessary to repeat the calculation, until it is clear that a further cycle of refinement would give small shifts when compared with their estimated standard deviations.

The estimated standard deviation, σ, of p_i, when using absolute weights, is given by:

$$\sigma_{p_i}^{2} = a_{ii}^{-1} \tag{22}$$

and, when using relative weights, by:

$$\sigma_{p_i}^{2} = \frac{a_{ii}^{-1} \sum_{r=1}^{m} w_r \Delta^2}{m - n} \tag{23}$$

where a_{ii}^{-1} is the i^{th} diagonal element of the inverse matrix A^{-1}.

Absolute weights reflect the precision of the measurements of $|F_o|$ and are calculated from:

$$w_r = \frac{1}{\sigma_r^{2}} \tag{24}$$

where σ_r^{2} is the estimated variance of the r^{th} observation due to random experimental errors. This type of weighting scheme can only be used when $\sum w\Delta^2/(m - n)$ is close to unity, indicating that the only significant errors are the random experimental errors. The presence of both random and systematic errors is allowed for by using relative weights which reflect the trends in the $|\Delta F|$ values. Calculation of relative weights is achieved by choosing a weighting function to give constant averages of $w\Delta^2$, when the set of $w\Delta^2$ values is analysed in ranges of $|F_o|$ and sinθ.

The estimated standard deviations derived in the above way are sometimes underestimated, particularly if block diagonal least-squares is employed, therefore it is desirable to have an independent assessment of their reliability from an examination of the bond lengths, valency angles and estimated standard deviations in any part of the molecule which has known geometry (e.g. a phenyl ring).

PART II

STRUCTURAL STUDIES OF SOME ORGANOMETALLIC COMPLEXES

CHAPTER 1

(N-ETHOXYCARBONYL-3-FORMYLAZEPINE)TRICARBONYLIRON

II.1.1 INTRODUCTION

Discussion(1 - 3) of the nature of the very stable bond formed between the iron atom and the butadiene group has been stimulated and aided by the structure elucidation of a wide variety of 1,3-diene $Fe(CO)_3$ complexes(4 - 27)(see Table 1 page 15). These reveal that the iron atom is bonded to a planar 'cis-butadiene fragment', producing substantial alterations in the molecular geometry of the olefinic ligand. For example, the characteristic 'tub' conformation of cyclo-octatetraene (COT)(28) is greatly altered on forming $(COT)Fe(CO)_3$ and $(COT)2Fe(CO)_3$(27), geometrical isomerisation occurs when vitamin-A aldehyde forms a tricarbonyliron complex(9) and in all 1,3-diene $Fe(CO)_3$ complexes, donation of electrons from the highest-occupied bonding molecular orbital of the diene to suitable metal orbitals, together with back-donation from filled metal d orbitals to the lowest antibonding orbital of the diene(2), results in the two outer bonds being significantly longer than normal double bonds and the central bond being significantly shorter than a single bond.

X-ray analyses of the seven-membered unsaturated azepine ring, both in the free state [N-p-bromobenzenesulphonylazepine(24), dibenz[b,f]azepine(22) and N-phenoxycarbonylazepine(29)] and when complexed with tricarbonyliron [1H-azepine $Fe(CO)_3$(22), N-methoxycarbonylazepine $Fe(CO)_3$(24) and 3-acetyl-1H-azepine $Fe(CO)_3$(23)] have enabled changes on complex formation and variations between complexes to be examined. Uncomplexed, the azepine ring exists in a boat conformation with localised double bonds, whereas when complexed, the ring is divided into two almost planar groups of atoms, hinged about the outer atoms of the delocalised 'butadiene fragment' and inclined at a dihedral angle of about 140°. Comparison of the complexes indicates that an electron-withdrawing substituent in the 3-position of the azepine ring (numbering as in Figure 2 page 24) increases the amount of back-donation from the iron, while substitution at the 1-position only significantly affects the bonds involving the nitrogen atom.

In order to investigate the molecular geometry of a 1,3-substituted complex, a crystal structure analysis of (1-ethoxycarbonyl-3-formylazepine)tricarbonyliron was undertaken on crystals prepared(30) by A. W. Johnson et al.

TABLE 1

(1,3-Diene)tricarbonyliron complexes investigated by X-ray analysis

a) Diene in an open chain

Butadiene $Fe(CO)_3$(4)

Sorbic acid $Fe(CO)_3$(5)

1,4-Diphenylbutadiene $Fe(CO)_3$(6)

N-Cinnamylideneaniline $Fe(CO)_3$(7)

Cinnamaldehyde $Fe(CO)_3$(8)

Vitamin-A aldehyde $Fe(CO)_3$(9)

(-)-2-(S-α-Methylbenzylammonium)-cis,trans-hepta-3,5-diene $Fe(CO)_3$(10)

b) Diene partly in an open chain and partly in a six-membered ring

m-Divinylbenzene $Fe_2(CO)_6$(11)

p-Divinylbenzene $Fe_2(CO)_6$(11)

1-Vinylnaphthalene $Fe(CO)_3$(11)

2-Vinylnaphthalene $Fe(CO)_3$(11)

c) Diene in a five-membered ring

Tetrakis(trifluoromethyl)cyclopentadienone $Fe(CO)_3$(12)

d) Diene in a six-membered ring

Octafluorocyclohexa-1,3-diene $Fe(CO)_3$ (13)

Symmetrical 12-Oxa[4.4.3]propella-2,4,7,9-tetraene $Fe_2(CO)_6$ (14)

Unsymmetrical 12-Oxa[4.4.3]propella-2,4,7,9-tetraene $Fe_2(CO)_6$ (15)

Tricyclo[$4.3.1.0^{1,6}$]deca-2,4-diene $Fe(CO)_3$ (16)

π-Tetracyclo[$8.6.0.0^{2,9}.0^{3,8}$]hexadecapenta-4,6,11,13,15-ene $Fe(CO)_3$ (17)

Thebaine $Fe(CO)_3$ (18)

4,5,6,7-Tetrahapto-pentacyclo[$8.3.3.0^{2,9}.0^{3,8}.0^{11,16}$]hexadeca-4,6,12,14-tetraene $Fe(CO)_3$ (19)

e) Diene in a seven-membered ring

Tropone $Fe(CO)_3$ (20)

2,4,6-Triphenyltropone $Fe(CO)_3$ (21)

1H-Azepine $Fe(CO)_3$ (22)

3-Acetyl-1H-azepine $Fe(CO)_3$ (23)

N-Methoxycarbonylazepine $Fe(CO)_3$ (24)

1-Isopropoxycarbonyl-1,2-diazepine $Fe(CO)_3$ (25)

Diazulene $Fe_4(CO)_{10}$ (26)

f) Diene in an eight-membered ring

Cyclo-octatetraene $Fe(CO)_3$ (27)

Cyclo-octatetraene $Fe_2(CO)_6$ (27)

II.1.2 EXPERIMENTAL

Crystal Data (N-Ethoxycarbonyl-3-formylazepine)tricarbonyliron; $C_{13}H_{11}O_6NFe$.

Crystal system	Triclinic
Unit cell dimensions	a = 7.022(3) Å
	b = 10.550(4) Å
	c = 9.500(3) Å
	α = 92°49′ (1)
	β = 94°32′ (2)
	γ = 96° 8′ (2)
Space group	$P\bar{1}$ (C_i^1)
Unit cell volume	U = 696.4 $Å^3$
Molecular weight	M = 333.0 a.m.u.
Observed density	D_o = 1.58 $gm.cm.^{-3}$ (flotation in aqueous zinc iodide solution)
Calculated density	D_c = 1.587 $gm.cm.^{-3}$
Number of molecules per unit cell	Z = 2
Number of electrons per unit cell	F(000) = 340
Linear absorption coefficient	μ(Mo-Kα) = 11.45 $cm.^{-1}$
Wavelength of Mo-Kα radiation	λ = 0.71069 Å

Crystallographic Measurements

Preliminary triclinic cell dimensions were obtained from precession photographs of a yellow crystal of dimensions ca. 0.10 x 0.35 x 0.50 mm^3.

The crystal was transfered to a Hilger and Watts' Y290 automatic diffractometer controlled by a PDP-8 computer and offset on χ by about 5° to prevent multiple reflections(31). Mo-$\underline{K}\alpha$ radiation was produced by a fully stabilized X-ray generator, operated at 46 kV and 16 mA, with a zirconium filter at the source.

The reciprocal lattice spacings and orientation determined from the precession photographs enabled the angular settings of two strong, low order, zonal reflections to be estimated. These settings were optimised by means of 2θ, ϕ and χ scans and incorporated with the cell dimensions in an orientation matrix, which was used to locate nine fairly strong reflections with a wide spread of ϕ and χ values and with θ values in the range 11° to 16°. The cell dimensions and the setting angles of two of these reflections were adjusted by a least-squares treatment(32), in order to minimise the disagreement between the optimised observed and the calculated setting angles of these nine reflections, thereby obtaining an orientation matrix with which to locate the reflections during the data collection. The adjusted cell dimensions, together with the standard deviations derived from the least-squares calculations, are given in the crystal data.

The intensity data were collected, in octants $hk\ell$, $hk\bar{\ell}$, $h\bar{k}\ell$ and $h\overline{k\ell}$ with $\theta(\text{Mo-}\underline{K}\alpha) \leqslant 25^\circ$, by the $\theta - 2\theta$ scan procedure, each reflection being scanned in 80 equal steps from $2\theta_{calc.}$ -0.80° to $2\theta_{calc.}$ $+0.80^\circ$. Each step of the scan was counted for 1 second and stationary-crystal, stationary-counter background counts were taken at each end of the scan range for 20 seconds. The intensities of two standard reflections were monitored every 40 intensity measurements and only small random fluctuations were observed. The scan counts (P) and the background counts (B_1, B_2) of each reflection were combined to yield the integrated intensity, $I = P - 2(B_1 + B_2)$, which was corrected for Lorentz-polarisation effects but not for absorption. 2,467 independent values of $|F_o|$ were obtained.

Structure Analysis

The initial coordinates of the iron atom were deduced from the three-dimensional Patterson synthesis and used to phase $\left[R = \Sigma(|F_o - F_c|)/\Sigma|F_o| = 46\%\right]$ an electron density distribution which, when contoured on to glass sheets, revealed the positions of the remaining non-hydrogen atoms. This map confirmed that the space group is the centrosymmetric $P\bar{1}$ and not the alternative noncentrosymmetric P1.

The approximate atomic parameters were adjusted by a series of least-squares calculations, using the program due to D. W. J. Cruickshank, J. G. F. Smith and J. G. Sime on Glasgow University's KDF9 computer. The function minimised was $\Sigma w(|F_o| - |F_c|)^2$, where w is a weight given by the expression:

$$w = \left[1 - \exp(-p_1\{\sin\theta/\lambda\}^2)\right]/\left[1 + p_2|F_o| + p_3|F_o|^2\right].$$

The values of p_1, p_2 and p_3 were adjusted at several stages of the refinement to ensure approximate constancy of $\langle w\Delta^2\rangle$ over various ranges of $|F_o|$ and $\sin\theta$. The final values were 50.0, 0.1 and 0.001 respectively. The atomic scattering factors, including dispersion corrections $\Delta f'$ and $\Delta f''$ for the iron atom, were taken from 'International Tables for X-ray Crystallography,' Vol. III.(33)

Two cycles of full-matrix refinement using isotropic thermal parameters reduced R to 10.6%, and after two cycles of block diagonal least-squares refinement with the atoms assigned anisotropic thermal parameters, the R-factor was 6.2%. At this stage a difference electron density distribution was calculated revealing the hydrogen atom positions (average peak height = $0.60e^-/\text{Å}^3$). The highest peak not attributable to a hydrogen atom (peak height $0.57e^-/\text{Å}^3$) was close to the iron atom. The positional and isotropic thermal parameters of the hydrogen atoms were then included in the least-squares adjustment and two further cycles of block diagonal refinement converged at $R = 5.0\%$. When those reflections with $P < 2(B_1 + B_2)$ were removed from the analysis, two rounds of block diagonal refinement, using the remaining 2,363 observations, converged at a conventional R-factor of 4.59% and a weighted R-factor $\left[R' = (\Sigma w\Delta^2/\Sigma w|F_o|^2)^{\frac{1}{2}}\right]$ of 6.43%. Each of the above increases in the number of refined parameters resulted in a significant(34) lowering of R'.

Results

The observed and final calculated structure factors are listed in Table 2, with the final positional and thermal parameters, including the estimated standard deviations derived from the least-squares refinement, in Table 3. The atomic numbering scheme of the non-hydrogen atoms (each hydrogen atom is numbered according to the atom to which it is bonded) is shown both in Figure 1; a perspective view of the molecule illustrating the thermal ellipsoids(35), and in Figure 2; the molecule projected on to the butadiene plane. Figure 2 also contains the bond lengths involving the non-hydrogen atoms, while those involving the hydrogen atoms are listed in Table 4. The valency angles, torsion angles and displacements of atoms from various planes are given in Tables 5, 6 and 7 respectively. The crystal packing is shown in Figure 3 and the intermolecular contacts < 3.60 Å are listed in Table 8.

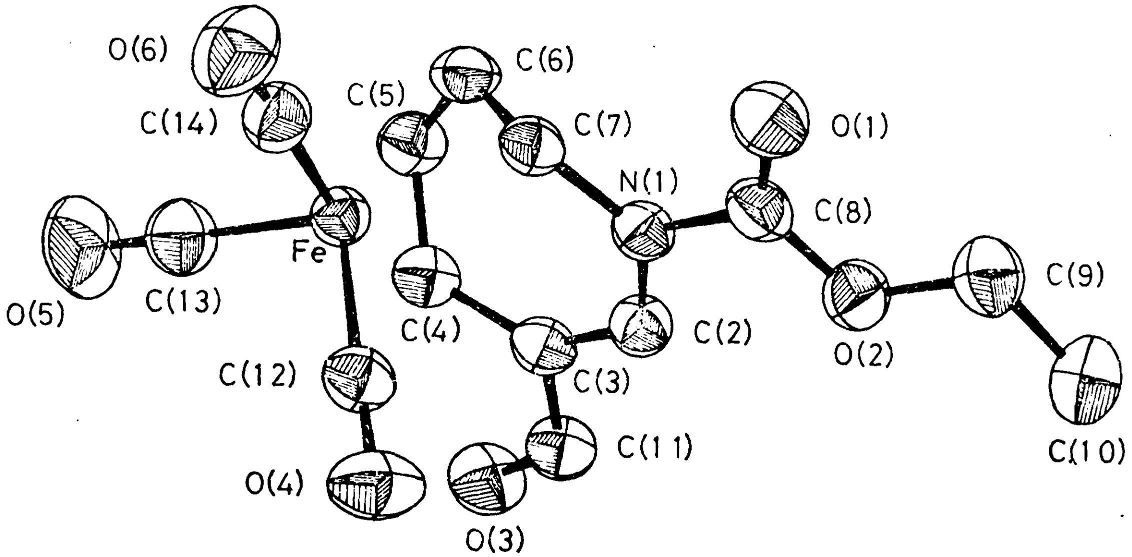

FIGURE 1

Perspective view of (N-ethoxycarbonyl-3-formylazepine)tricarbonyliron showing the 50% probability thermal ellipsoids.

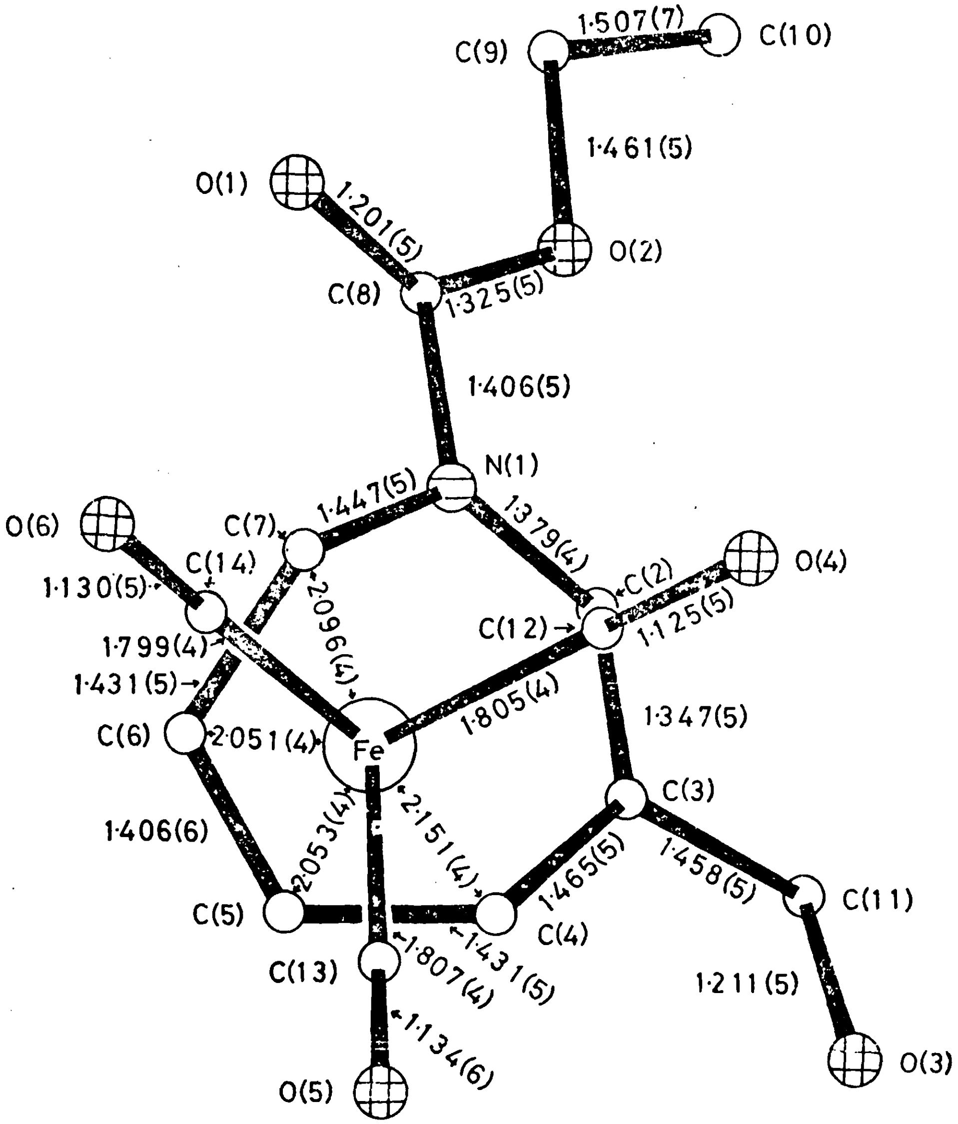

FIGURE 2

Projection of (N-ethoxycarbonyl-3-formylazepine)tricarbonyliron on to the butadiene plane, illustrating the bond lengths.

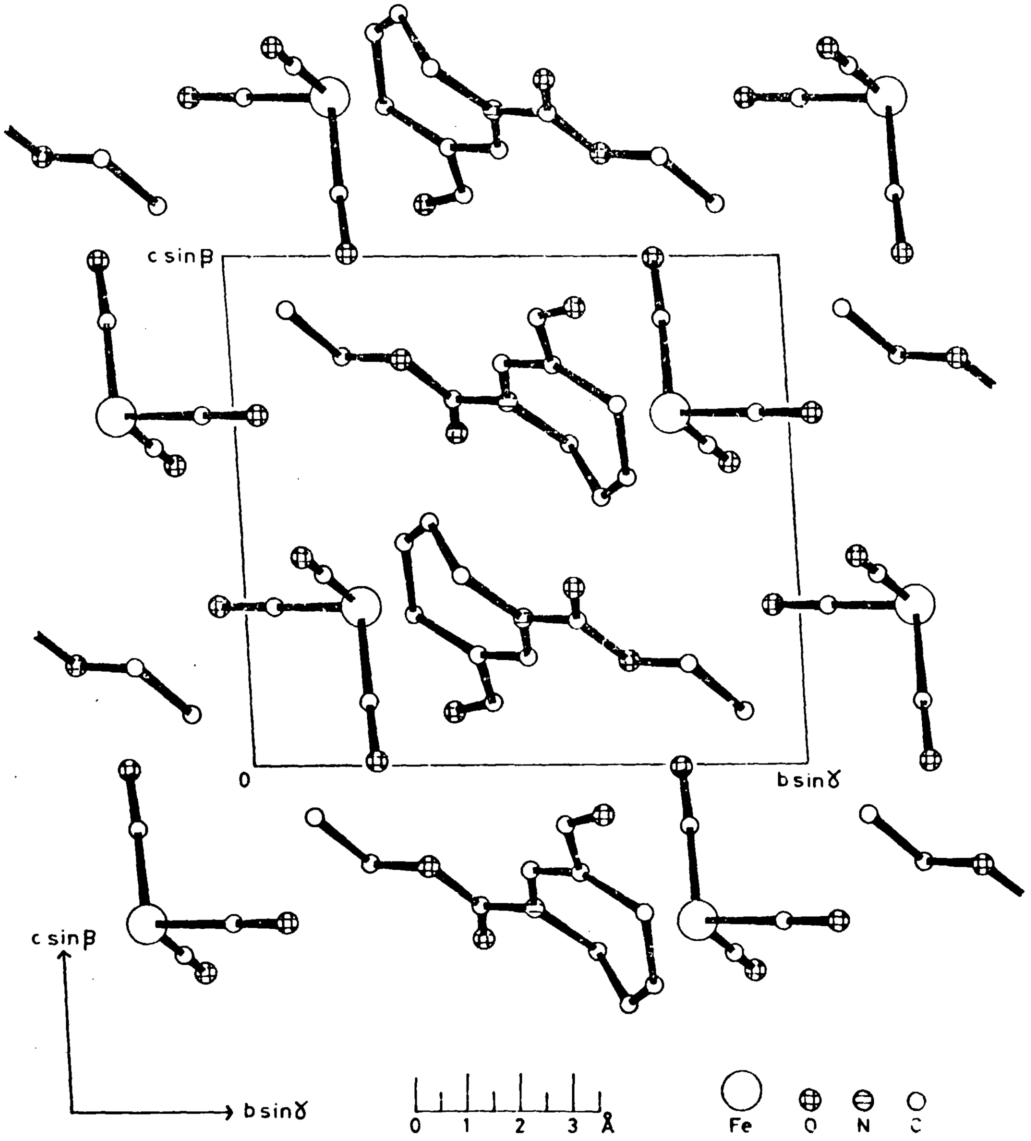

FIGURE 3

Packing arrangement of (N-ethoxycarbonyl-3-formylazepine)tricarbonyliron viewed along the a-axis.

TABLE 2

(N-Ethoxycarbonyl-3-formylazepine)tricarbonyliron; observed and final calculated structure amplitudes.

H	K	L	F OBS	F CALC
8	2	0	4.3	3.7
8	2	-1	1.3	1.4
8	2	-2	4.7	5.0
8	1	1	1.9	0.5
8	1	0	8.1	7.7
8	1	-1	5.9	5.6
8	1	-2	4.5	5.2
8	1	-3	5.7	5.6
8	0	2	8.9	8.7
8	0	1	11.5	11.6
8	0	3	3.8	3.3
8	0	-1	7.7	6.9
8	0	-2	13.2	12.4
8	-1	2	12.9	12.6
8	-1	1	5.4	4.7
8	-1	0	9.6	9.6
8	-1	-1	6.0	6.9
8	-1	-2	4.0	3.8
8	-1	-3	5.8	5.8
8	-2	2	7.3	6.7
8	-2	1	6.5	6.1
8	-2	0	3.5	3.7
8	-2	-1	3.9	4.3
8	-2	-2	7.9	8.0
8	-2	-3	7.5	6.7
8	-3	2	5.2	5.1
8	-3	1	6.6	6.4
8	-3	0	6.7	6.6
8	-3	-1	1.1	2.0
8	-3	-2	4.4	4.5
8	-3	-3	8.8	8.5
8	-4	1	2.0	1.0
8	-4	0	5.0	5.1
7	5	1	11.0	10.7
7	5	0	4.1	3.9
7	5	-1	4.3	4.1
7	5	-2	8.5	9.1
7	5	-3	3.6	3.7
7	4	3	4.0	3.4
7	4	2	3.6	3.9
7	4	1	4.9	4.5
7	4	0	5.1	5.4
7	4	-1	2.3	1.3
7	4	-2	5.3	5.2
7	4	-3	4.6	5.4
7	4	-4	2.0	2.7
7	4	-5	6.2	5.9
7	3	3	3.9	4.1
7	3	2	2.7	2.5
7	3	1	5.1	5.5
7	3	-1	7.8	7.7
7	3	-2	5.9	6.9
7	3	-3	3.8	3.5
7	3	-4	5.1	3.8
7	3	-5	4.9	5.4
7	2	3	5.4	4.6
7	2	2	2.8	3.0
7	2	1	9.4	9.6
7	2	0	1.1	0.9
7	2	-2	8.7	9.0
7	2	-3	4.6	5.4
7	2	-4	5.0	4.9
7	2	-5	6.9	6.0
7	1	4	3.4	2.7
7	1	2	12.6	12.0
7	1	1	5.7	5.6
7	1	0	11.9	11.8
7	1	-1	11.7	12.4
7	1	-2	6.7	7.0
7	1	-3	18.6	17.8
7	1	-4	11.1	10.1
7	1	-5	10.5	9.7
7	1	-6	10.0	8.8
7	0	5	6.3	5.9
7	0	4	10.5	9.8
7	0	3	11.6	11.7
7	0	2	4.8	4.3
7	0	1	15.6	15.4
7	0	0	1.7	1.8
7	0	-1	1.3	0.4
7	0	-2	1.3	1.7
7	0	-3	8.9	8.3
7	0	-4	3.3	3.1
7	0	-5	13.8	13.0
7	0	-6	2.8	3.1
7	-1	5	12.7	12.3
7	-1	4	2.8	2.1
7	-1	3	7.0	6.7
7	-1	2	10.1	10.2
7	-1	1	6.2	6.3
7	-1	0	14.6	14.7
7	-1	-1	1.8	2.4
7	-1	-2	11.6	11.8
7	-1	-3	2.4	2.4
7	-1	-4	5.1	4.8
7	-1	-5	9.9	9.7
7	-1	-6	4.7	5.4
7	-2	6	0.4	1.2
7	-2	4	13.9	13.7
7	-2	3	11.8	11.6
7	-2	2	4.7	4.5
7	-2	1	17.0	16.8
7	-2	0	9.1	8.5
7	-2	-1	7.9	8.5
7	-2	-2	9.9	9.8
7	-2	-3	10.1	9.9
7	-2	-4	12.4	12.1
7	-2	-5	3.3	4.6
7	-2	-6	8.6	8.4
7	-3	5	3.7	3.8
7	-3	4	2.6	1.1
7	-3	3	7.0	6.3
7	-3	2	7.9	7.3
7	-3	1	1.0	2.0
7	-3	0	1.9	1.2
7	-3	-1	8.4	7.1
7	-3	-2	4.3	4.3
7	-3	-3	11.3	10.9
7	-3	-4	3.1	2.6
7	-3	-5	9.4	9.2
7	-3	-6	4.4	4.2
7	-4	4	6.1	5.8
7	-4	2	12.1	12.4
7	-4	1	7.9	8.0
7	-4	0	7.9	9.6
7	-4	-1	3.4	3.9
7	-4	-2	5.4	5.5
7	-4	-3	2.4	2.6
7	-4	-4	10.6	10.4
7	-4	-5	4.5	4.2
7	-4	-6	4.8	4.4
7	-5	4	9.1	4.8
7	-5	3	7.9	7.8
7	-5	2	10.2	9.7
7	-5	1	9.3	10.1
7	-5	0	11.1	11.9
7	-5	-1	1.3	0.2
7	-5	-2	7.4	8.2
7	-5	-3	10.4	10.0
7	-5	-4	1.9	1.6
7	-5	-5	5.3	4.5
7	-6	2	5.3	5.2
7	-6	1	3.8	3.8
7	-6	0	6.6	6.7
7	-6	-1	8.2	8.7
7	-6	-2	3.8	3.9
7	-6	-3	4.9	5.0
7	-6	-4	5.8	5.8
7	-7	1	7.3	7.3
7	-7	0	4.2	3.1
7	-7	-1	4.9	5.2
7	-7	-2	7.6	7.3
7	-7	-3	3.0	3.3
6	7	2	6.6	6.9
6	7	1	10.0	9.9
6	7	0	12.0	12.6
6	7	-1	2.6	2.0
6	7	-2	8.2	8.1
6	7	-3	9.8	10.5
6	6	2	12.4	12.6
6	6	1	11.1	11.0
6	6	0	2.3	2.7
6	6	-1	12.5	12.3
6	6	-2	4.5	3.9
6	6	-3	9.7	10.0
6	6	-4	7.5	6.9
6	5	4	6.8	6.1
6	5	3	9.4	9.1
6	5	2	1.7	2.1
6	5	1	9.8	9.9
6	5	0	5.9	5.8
6	5	-2	12.9	13.9
6	5	-3	2.4	2.5
6	5	-4	9.4	9.7
6	5	-5	9.7	9.5
6	5	-6	1.8	1.5
6	4	5	6.4	6.6
6	4	4	6.9	7.7
6	4	3	3.0	3.4
6	4	2	18.5	17.8
6	4	1	8.7	9.0
6	4	0	7.1	7.2
6	4	-1	7.7	8.2
6	4	-2	8.8	9.6
6	4	-3	13.5	13.2
6	4	-4	14.9	14.8
6	4	-6	7.7	7.7
6	4	-7	2.8	3.6
6	3	6	8.0	7.5
6	3	5	2.7	1.9
6	3	4	5.0	4.5
6	3	3	3.8	3.6
6	3	2	2.4	2.6
6	3	1	12.2	12.1
6	3	0	2.4	2.3
6	3	-1	0.7	1.3
6	3	-2	11.5	12.1
6	3	-3	10.6	10.7
6	3	-4	9.3	8.9
6	3	-5	11.0	10.9
6	3	-6	3.8	2.2
6	3	-7	6.1	5.4
6	2	6	2.1	1.2
6	2	5	7.3	6.6
6	2	4	2.0	1.7
6	2	3	8.2	7.9
6	2	2	17.2	16.1
6	2	1	7.4	7.9
6	2	0	37.8	37.7
6	2	-1	3.7	4.1
6	2	-2	18.7	18.4
6	2	-3	12.0	11.9
6	2	-4	8.9	9.0
6	2	-5	8.2	7.1
6	2	-6	7.9	7.6
6	2	-7	2.6	1.0
6	2	-8	7.8	7.6
6	1	6	7.1	7.1
6	1	5	3.1	2.1
6	1	4	12.4	11.9
6	1	3	2.4	2.4
6	1	2	0.3	1.1
6	1	1	14.0	13.9
6	1	0	11.2	11.3
6	1	-1	17.2	17.7
6	1	-2	28.1	27.9
6	1	-4	15.1	13.9
6	1	-5	3.6	4.1
6	1	-6	12.6	12.5
6	1	-7	3.5	3.7
6	1	-8	3.2	2.7
6	0	7	2.7	3.2
6	0	6	6.8	6.6
6	0	5	3.1	3.1
6	0	4	1.9	0.4
6	0	3	7.3	7.1
6	0	2	5.2	4.9
6	0	1	14.5	14.8
6	0	0	3.5	2.8
6	0	-1	4.6	4.2
6	0	-2	8.5	7.6
6	0	-3	5.5	5.0
6	0	-4	1.4	0.4
6	0	-5	7.9	7.6
6	0	-6	5.8	5.8
6	0	-7	6.5	6.0
6	0	-8	7.9	8.3
6	-1	7	15.0	15.2
6	-1	6	3.9	4.1
6	-1	5	9.5	9.1
6	-1	4	12.9	12.7
6	-1	3	3.3	3.7
6	-1	2	22.7	22.3
6	-1	1	1.3	1.6
6	-1	0	17.2	17.6
6	-1	-1	5.5	6.7
6	-1	-2	4.5	4.9
6	-1	-3	13.4	13.1
6	-1	-4	8.0	7.6
6	-1	-5	6.7	6.3
6	-1	-6	13.4	12.8
6	-1	-7	3.3	3.4
6	-1	-8	1.8	2.2
6	-2	6	10.8	9.4
6	-2	5	5.8	5.6
6	-2	4	3.7	3.3
6	-2	3	4.8	5.3
6	-2	2	13.3	13.9
6	-2	1	17.5	17.6
6	-2	0	4.6	4.4
6	-2	-1	9.4	10.6
6	-2	-2	6.0	6.2
6	-2	-3	4.3	3.7
6	-2	-4	15.4	14.7
6	-2	-5	11.5	10.8
6	-2	-6	2.1	1.4
6	-2	-7	5.5	5.0
6	-2	-8	7.5	8.1
6	-3	7	6.3	7.6
6	-3	6	2.0	2.1
6	-3	5	9.8	9.5
6	-3	4	2.8	3.0
6	-3	3	6.4	5.8
6	-3	2	7.5	6.7
6	-3	1	4.1	4.0
6	-3	0	9.7	9.7
6	-3	-1	0.6	1.5
6	-3	-2	6.8	7.1
6	-3	-3	17.5	17.1
6	-3	-4	4.8	5.1
6	-3	-5	10.6	10.5
6	-3	-6	10.0	9.6
6	-3	-7	0.9	0.8
6	-3	-8	2.5	0.7
6	-4	6	7.5	7.1
6	-4	5	2.0	1.0
6	-4	4	18.1	18.3
6	-4	3	12.7	12.3
6	-4	1	14.8	15.2
6	-4	0	5.0	5.4
6	-4	-1	6.1	6.2
6	-4	-2	8.3	7.8
6	-4	-3	2.5	2.2
6	-4	-4	11.7	11.3
6	-4	-5	10.6	10.2
6	-4	-6	4.1	4.1
6	-4	-7	4.9	5.4
6	-5	6	9.1	8.9
6	-5	5	11.5	11.5
6	-5	3	11.6	11.7
6	-5	2	14.6	14.9
6	-5	1	4.5	3.3
6	-5	0	10.6	11.0
6	-5	-1	2.1	2.1
6	-5	-2	8.4	8.4
6	-5	-3	11.6	12.0
6	-5	-4	1.5	1.4
6	-5	-5	9.6	9.9
6	-5	-6	3.6	2.9
6	-5	-7	1.6	3.2
6	-6	5	7.4	7.0
6	-6	4	16.2	15.6
6	-6	3	8.2	7.8
6	-6	2	9.3	9.3
6	-6	1	13.3	13.5
6	-6	0	1.3	2.7
6	-6	-1	7.7	7.6
6	-6	-2	7.7	7.6
6	-6	-3	0.6	3.0
6	-6	-4	11.3	10.9
6	-6	-5	2.4	1.2
6	-6	-6	10.1	9.9
6	-7	5	5.2	5.1
6	-7	3	5.6	5.4
6	-7	2	4.6	4.8
6	-7	1	2.4	2.1
6	-7	0	7.3	6.8
6	-7	-2	4.3	4.5
6	-7	-3	3.0	3.4
6	-7	-4	2.4	0.4
6	-7	-5	5.6	4.8
6	-8	4	3.9	3.2
6	-8	3	2.1	2.1
6	-8	2	7.8	7.9
6	-8	0	10.4	10.5
6	-8	-1	6.5	6.5
6	-8	-3	3.9	3.9
6	-8	-4	2.4	1.3
6	-9	1	8.5	7.9
6	-9	0	8.7	8.1
6	-9	-1	1.5	0.4
6	-9	-2	11.1	10.3
6	-9	-3	5.3	4.8
5	9	0	4.3	3.9
5	9	-1	5.7	5.1
5	9	-2	2.0	2.2
5	8	3	6.3	6.5
5	8	2	3.2	3.2
5	8	1	10.7	11.0
5	8	0	6.7	7.9
5	8	-2	13.5	13.9
5	8	-3	4.3	4.4
5	8	-4	5.8	5.6
5	8	-5	9.0	8.6
5	7	4	3.3	2.6
5	7	3	7.4	7.1
5	7	2	13.4	13.7
5	7	1	0.9	1.7
5	7	0	10.4	10.5
5	7	-1	6.1	6.2
5	7	-2	0.5	2.0
5	7	-3	7.5	7.0
5	7	-4	2.7	2.0
5	7	-5	7.8	7.7
5	7	-6	4.7	5.0
5	6	5	6.6	6.5
5	6	4	6.1	6.4
5	6	3	3.0	3.2
5	6	2	6.8	6.6
5	6	1	17.1	17.3
5	6	0	8.8	9.4
5	6	-1	6.1	5.9
5	6	-2	2.6	0.9
5	6	-3	6.7	7.3
5	6	-4	9.0	9.2
5	6	-5	2.7	1.0
5	6	-6	4.4	4.0
5	6	-7	2.1	2.5
5	5	6	7.4	7.2
5	5	5	15.3	15.3
5	5	3	11.6	11.9
5	5	2	19.1	19.4
5	5	1	0.7	1.7
5	5	0	16.8	17.2
5	5	-1	3.2	3.3
5	5	-2	7.7	8.3
5	5	-3	10.9	11.4
5	5	-5	14.5	14.0
5	5	-6	2.3	3.0
5	5	-7	2.4	2.3
5	5	-8	1.7	1.7
5	4	7	9.1	9.0
5	4	6	5.8	6.1
5	4	5	5.1	4.9
5	4	4	9.6	9.6
5	4	3	4.3	3.8
5	4	2	7.1	7.9
5	4	1	18.5	19.6
5	4	0	6.7	7.3
5	4	-1	16.8	17.8
5	4	-2	4.8	5.5
5	4	-3	13.9	13.9
5	4	-4	18.9	18.3
5	4	-5	6.7	7.4
5	4	-6	11.4	10.8
5	4	-7	3.4	2.8
5	4	-8	1.3	0.8
5	3	7	1.4	1.5
5	3	6	10.4	10.0
5	3	5	11.4	11.3
5	3	4	19.5	19.1
5	3	3	28.9	27.9
5	3	2	9.3	8.7
5	3	1	24.7	24.6
5	3	-1	7.9	8.6
5	3	-2	22.1	23.7
5	3	-3	17.2	17.8
5	3	-4	4.8	4.8
5	3	-5	23.7	23.2
5	3	-6	4.5	4.7
5	3	-7	10.6	10.1
5	3	-8	4.3	4.6
5	3	-9	2.9	3.3
5	2	7	11.6	11.7
5	2	6	5.7	5.4
5	2	5	12.5	12.4
5	2	4	23.9	23.2
5	2	3	3.9	3.9
5	2	2	36.6	36.1
5	2	1	16.5	17.0
5	2	0	22.2	22.5
5	2	-1	25.9	25.6
5	2	-3	29.3	29.1
5	2	-4	7.6	7.4
5	2	-6	13.9	10.6
5	2	-7	2.9	3.5
5	2	-8	9.7	9.1
5	2	-9	10.2	10.3
5	1	7	4.7	5.0
5	1	6	7.2	7.0
5	1	4	5.1	5.0
5	1	3	13.4	13.1
5	1	2	9.1	9.2
5	1	1	28.6	28.3
5	1	0	4.1	4.1
5	1	-1	4.2	4.3
5	1	-2	21.4	21.9
5	1	-3	16.0	15.4
5	1	-4	10.8	10.6
5	1	-5	14.4	14.5
5	1	-6	6.2	6.1
5	1	-7	10.7	10.6
5	1	-8	9.4	9.7
5	1	-9	6.2	6.1
5	0	8	7.4	7.9
5	0	7	8.8	8.7
5	0	6	14.9	15.2
5	0	5	20.2	19.6
5	0	4	9.1	9.5
5	0	3	7.4	7.9
5	0	2	17.2	15.7
5	0	1	3.8	3.5
5	0	0	33.7	34.2
5	0	-1	17.3	15.9
5	0	-2	4.3	4.0
5	0	-3	21.5	21.3
5	0	-4	3.5	2.7
5	0	-5	14.5	14.1
5	0	-6	4.5	4.7
5	0	-8	4.1	3.9
5	0	-9	8.8	9.0
5	-1	8	3.9	4.6
5	-1	7	5.8	5.4
5	-1	6	14.9	15.3
5	-1	5	6.2	6.7
5	-1	4	14.3	14.7
5	-1	3	1.9	2.3
5	-1	2	11.9	12.5
5	-1	1	21.6	22.0
5	-1	0	4.6	5.9
5	-1	-1	10.0	10.6
5	-1	-2	12.2	11.5
5	-1	-4	12.5	11.6
5	-1	-5	7.7	7.3
5	-1	-6	18.6	18.6
5	-1	-7	10.3	10.1
5	-1	-8	1.8	1.3
5	-1	-9	7.4	7.5
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H	K	L	F OBS	F CALC
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1	-4	-10	9.5	10.2
1	-5	10	7.8	7.7
1	-5	9	1.0	1.9
1	-5	8	12.2	12.0
1	-5	7	4.4	4.5
1	-5	6	18.6	17.8
1	-5	5	11.1	11.1
1	-5	4	8.8	8.2
1	-5	3	29.3	30.3
1	-5	2	12.5	10.8
1	-5	1	8.0	7.4
1	-5	0	10.9	12.7
1	-5	-1	4.2	4.1
1	-5	-2	26.3	26.9
1	-5	-3	23.3	22.8
1	-5	-4	10.4	10.5
1	-5	-5	14.6	15.4
1	-5	-6	5.7	5.3
1	-5	-7	14.4	14.2
1	-5	-8	5.6	5.6
1	-5	-9	4.4	4.6
1	-5	-10	3.8	3.5
1	-6	10	8.3	8.5
1	-6	8	3.7	1.5
1	-6	7	14.0	13.7
1	-6	6	2.5	1.7
1	-6	5	22.3	22.3
1	-6	4	17.6	17.4
1	-6	3	8.0	7.6
1	-6	2	33.5	34.3
1	-6	1	11.0	11.7
1	-6	0	38.2	39.0
1	-6	-1	26.7	27.8
1	-6	-2	13.5	10.4
1	-6	-3	10.1	10.6
1	-6	-4	18.2	18.4
1	-6	-5	2.1	1.9
1	-6	-6	12.9	13.0
1	-6	-7	3.8	4.4
1	-6	-8	6.2	6.8
1	-6	-9	7.4	7.3
1	-7	9	8.6	9.9
1	-7	8	7.5	7.8
1	-7	7	4.8	5.0
1	-7	6	25.5	25.5
1	-7	5	8.1	8.1
1	-7	4	5.5	6.4
1	-7	3	18.9	19.6
1	-7	2	8.0	7.9
1	-7	1	23.2	24.2
1	-7	0	25.9	26.6
1	-7	-1	8.0	7.5
1	-7	-2	15.0	15.8
1	-7	-3	12.3	11.8
1	-7	-4	6.5	6.0
1	-7	-5	4.5	3.8
1	-7	-6	2.5	2.7
1	-7	-7	11.0	10.3
1	-7	-8	2.7	2.5
1	-7	-9	2.5	2.9
1	-8	8	7.5	7.3
1	-8	7	12.4	12.1
1	-8	6	4.6	5.4
1	-8	5	17.8	17.9
1	-8	4	15.3	14.9
1	-8	3	22.2	12.5
1	-8	2	12.9	13.6
1	-8	1	4.8	4.5
1	-8	0	22.3	22.7
1	-8	-1	18.4	19.7
1	-8	-2	1.6	0.9
1	-8	-3	12.1	12.2
1	-8	-4	8.7	9.7
1	-8	-5	10.5	10.6
1	-8	-6	14.1	13.5
1	-8	-7	3.9	4.2
1	-8	-8	4.5	4.7
1	-9	8	1.1	1.3
1	-9	7	9.2	9.3
1	-9	6	11.6	11.1
1	-9	4	6.3	6.7
1	-9	3	6.5	5.9
1	-9	2	3.0	1.7
1	-9	1	19.0	17.2
1	-9	0	10.7	10.5
1	-9	-1	16.1	16.0
1	-9	-2	8.7	9.0
1	-9	-4	10.0	9.7
1	-9	-5	5.2	5.3
1	-9	-6	3.0	2.2
1	-9	-7	7.2	7.6
1	-10	7	6.6	6.3
1	-10	6	4.3	4.5
1	-10	5	7.1	6.9
1	-10	4	1.1	0.7
1	-10	3	9.6	9.6
1	-10	2	4.5	4.0
1	-10	1	6.8	6.7
1	-10	0	8.3	8.6
1	-10	-1	2.3	1.1
1	-10	-2	6.3	6.7
1	-10	-3	7.3	7.5
1	-10	-4	1.7	1.9
1	-10	-5	7.7	7.0
1	-10	-6	3.2	2.1
1	-11	5	5.3	4.2
1	-11	4	6.4	6.1
1	-11	3	3.0	0.6
1	-11	2	5.0	4.2
1	-11	1	9.8	9.9
1	-11	0	3.8	4.1
1	-11	-1	13.0	12.4
1	-11	-2	6.8	6.4
1	-11	-3	4.4	3.8
1	-11	-4	9.4	9.2
1	-12	3	9.5	9.3
1	-12	2	4.1	3.6
1	-12	0	6.1	5.3
1	-12	-1	2.9	1.8
1	-12	-2	1.5	0.5
0	12	1	2.4	2.0
0	12	0	8.0	7.2
0	12	-1	2.2	1.4
0	12	-2	5.9	5.3
0	12	-3	7.2	7.4
0	11	4	8.6	8.1
0	11	3	5.6	5.6
0	11	2	15.9	15.4
0	11	1	1.4	0.6
0	11	0	13.5	12.6
0	11	-1	7.4	6.9
0	11	-2	0.4	0.7
0	11	-3	6.8	6.6
0	11	-4	5.8	5.5
0	11	-5	1.9	0.4
0	10	6	4.9	4.7
0	10	5	2.6	2.3
0	10	4	1.9	2.2
0	10	3	9.0	9.1
0	10	2	9.5	8.9
0	10	1	9.0	9.4
0	10	0	19.2	18.2
0	10	-1	3.9	3.3
0	10	-2	17.1	16.3
0	10	-3	3.8	4.2
0	10	-4	4.9	5.1
0	10	-5	4.7	4.4
0	10	-6	1.5	1.0
0	10	-7	5.0	5.7
0	9	7	6.6	6.5
0	9	6	2.5	0.5
0	9	5	6.9	6.7
0	9	4	6.4	6.6
0	9	3	4.3	4.6
0	9	2	5.4	5.8
0	9	1	9.7	9.7
0	9	0	6.8	6.2
0	9	-1	4.1	4.1
0	9	-2	10.2	10.3
0	9	-3	3.7	3.5
0	9	-4	10.2	10.1
0	9	-5	5.2	5.6
0	9	-6	11.2	11.2
0	9	-7	1.6	1.0
0	9	-8	4.5	4.7
0	8	8	7.2	7.3
0	8	7	5.7	6.3
0	8	6	17.5	17.5
0	8	5	0.9	0.1
0	8	4	14.6	14.5
0	8	3	7.4	7.4
0	8	2	0.6	1.1
0	8	1	16.8	16.8
0	8	0	7.5	7.6
0	8	-1	13.4	13.1
0	8	-2	21.5	22.0
0	8	-3	3.6	4.0
0	8	-4	20.1	20.7
0	8	-5	8.9	8.7
0	8	-6	6.3	6.3
0	8	-7	11.7	11.7
0	8	-8	6.4	5.9
0	8	-9	1.4	1.4
0	7	8	5.5	5.7
0	7	7	8.1	8.0
0	7	6	1.6	1.2
0	7	5	19.1	18.8
0	7	4	15.8	14.8
0	7	3	14.7	14.6
0	7	2	17.2	17.7
0	7	1	1.8	1.0
0	7	0	25.4	26.2
0	7	-1	0.4	0.9
0	7	-2	9.0	9.4
0	7	-3	16.8	17.3
0	7	-4	6.3	6.3
0	7	-5	18.9	18.7
0	7	-6	23.5	23.5
0	7	-7	3.7	3.9
0	7	-8	9.1	9.4
0	7	-9	3.2	2.8
0	6	9	3.5	3.3
0	6	8	1.9	0.4
0	6	7	10.7	10.7
0	6	6	7.1	7.7
0	6	5	8.0	8.2
0	6	4	11.4	11.8
0	6	3	2.4	2.7
0	6	2	4.3	3.6
0	6	1	25.9	21.9
0	6	0	5.8	6.4
0	6	-1	14.4	15.4
0	6	-2	0.8	1.2
0	6	-3	15.7	16.1
0	6	-4	31.4	31.3
0	6	-5	6.8	6.7
0	6	-6	12.1	11.5
0	6	-7	8.7	8.3
0	6	-8	5.8	6.1
0	6	-9	9.0	9.3
0	6	-10	5.6	5.9
0	5	9	7.2	7.2
0	5	8	5.2	5.2
0	5	7	1.0	1.2
0	5	6	22.0	21.1
0	5	5	14.7	15.3
0	5	4	14.3	13.6
0	5	3	40.4	40.0
0	5	2	23.1	22.5
0	5	1	5.6	6.6
0	5	0	29.7	31.3
0	5	-1	5.1	4.4
0	5	-2	13.9	12.0
0	5	-3	23.1	23.2
0	5	-4	3.5	1.8
0	5	-5	21.6	21.2
0	5	-6	12.8	15.6
0	5	-7	7.0	7.1
0	5	-8	2.3	2.3
0	5	-10	9.6	9.7
0	4	10	11.2	11.9
0	4	9	1.8	1.5
0	4	8	12.3	12.7
0	4	7	16.5	16.0
0	4	6	8.3	7.6
0	4	5	23.7	23.4
0	4	4	40.3	39.5
0	4	3	7.4	7.2
0	4	2	21.8	22.3
0	4	1	43.2	43.5
0	4	0	4.4	4.6
0	4	-1	21.0	21.5
0	4	-2	30.0	27.6
0	4	-3	21.4	21.4
0	4	-4	7.4	5.6
0	4	-5	3.6	3.7
0	4	-6	16.8	17.1
0	4	-7	3.3	5.5
0	4	-8	9.6	9.7
0	4	-9	10.1	10.1
0	4	-10	3.3	2.6
0	3	10	2.0	1.1
0	3	9	6.3	8.4
0	3	8	9.8	10.1
0	3	7	8.5	8.9
0	3	6	30.0	29.4
0	3	5	1.0	0.6
0	3	4	44.1	42.7
0	3	3	39.2	36.7
0	3	2	6.2	6.5
0	3	1	15.0	15.7
0	3	0	22.4	23.6
0	3	-1	36.1	35.8
0	3	-2	46.4	45.3
0	3	-3	5.0	7.9
0	3	-4	45.7	42.5
0	3	-5	11.0	11.0
0	3	-6	17.1	16.5
0	3	-7	23.8	24.0
0	3	-8	0.9	0.7
0	3	-9	3.8	3.9
0	3	-10	11.1	10.9
0	3	-11	4.5	1.1
0	2	10	6.8	6.7
0	2	9	2.2	0.7
0	2	8	4.3	3.9
0	2	7	20.5	20.8
0	2	6	19.3	19.0
0	2	5	16.5	16.9
0	2	4	25.5	23.7
0	2	3	4.6	4.0
0	2	2	43.6	41.9
0	2	1	13.5	13.1
0	2	0	36.0	39.2
0	2	-1	20.7	19.7
0	2	-2	61.1	59.9
0	2	-3	32.1	30.6
0	2	-4	6.3	5.9
0	2	-5	3.9	3.3
0	2	-6	17.0	16.5
0	2	-7	1.6	0.4
0	2	-8	19.0	19.0
0	2	-9	11.8	12.1
0	2	-10	2.7	2.9
0	2	-11	3.7	3.6
0	1	11	8.6	9.1
0	1	10	7.2	7.6
0	1	9	6.9	6.9
0	1	8	8.3	8.0
0	1	7	5.7	5.8
0	1	6	33.8	34.0
0	1	5	1.2	0.7
0	1	4	44.7	43.2
0	1	3	3.2	2.5
0	1	2	65.4	65.3
0	1	1	60.4	61.3
0	1	0	29.9	32.4
0	1	-1	48.3	46.7
0	1	-2	38.1	36.0
0	1	-3	50.7	45.7
0	1	-4	27.5	27.7
0	1	-5	10.8	9.4
0	1	-6	21.5	21.8
0	1	-7	20.7	20.2
0	1	-8	2.0	2.8
0	1	-9	6.3	6.4
0	1	-10	8.2	8.4
0	1	-11	2.4	2.0
0	0	11	4.5	4.5
0	0	10	15.3	16.3
0	0	9	10.5	10.5
0	0	8	13.7	14.2
0	0	7	12.0	11.9
0	0	6	21.9	21.2
0	0	5	32.5	32.2
0	0	4	9.5	9.5
0	0	3	52.3	49.6
0	0	2	73.8	74.8
0	0	1	31.4	29.6

TABLE 3

(N-Ethoxycarbonyl-3-formylazepine)tricarbonyliron; fractional atomic coordinates with esd in parentheses and thermal parameters.

Atom	x	y	z	U_{iso}($x10^3 Å^2$)
Fe	0.09012(7)	0.20994(4)	0.31160(5)	*
O(1)	0.4821(4)	0.5994(3)	0.3480(3)	*
O(2)	0.2767(4)	0.6911(3)	0.2021(3)	*
O(3)	-0.4677(4)	0.3699(3)	0.1045(4)	*
O(4)	0.1398(5)	0.2252(4)	0.0095(3)	*
O(5)	-0.1322(6)	-0.0425(4)	0.3124(5)	*
O(6)	0.4497(5)	0.1128(3)	0.4102(4)	*
N(1)	0.1772(4)	0.5013(3)	0.2865(3)	*
C(2)	0.0021(5)	0.5073(3)	0.2120(4)	*
C(3)	-0.1513(5)	0.4191(4)	0.2140(4)	*
C(4)	-0.1667(5)	0.3045(4)	0.2952(4)	*
C(5)	-0.0889(5)	0.2960(4)	0.4377(4)	*
C(6)	0.1035(6)	0.3445(4)	0.4764(4)	*
C(7)	0.2191(5)	0.3954(3)	0.3705(4)	*
C(8)	0.3282(5)	0.5996(3)	0.2831(4)	*
C(9)	0.4255(7)	0.7983(4)	0.1970(5)	*
C(10)	0.3369(9)	0.8929(5)	0.1055(6)	*
C(11)	-0.3188(5)	0.4402(4)	0.1210(4)	*
C(12)	0.1243(5)	0.2170(4)	0.1257(4)	*
C(13)	-0.0495(6)	0.0558(4)	0.3116(5)	*
C(14)	0.3105(6)	0.1500(4)	0.3728(4)	*
H(2)	-0.010(5)	0.583(3)	0.162(4)	10
H(4)	-0.290(5)	0.256(3)	0.274(3)	6
H(5)	-0.157(7)	0.239(4)	0.495(5)	37
H(6)	0.159(6)	0.340(4)	0.567(4)	22
H(7)	0.349(5)	0.406(3)	0.404(4)	8
H(9a)	0.548(8)	0.763(5)	0.150(6)	52
H(9b)	0.445(7)	0.837(5)	0.283(5)	45
H(10a)	0.312(10)	0.861(6)	0.018(7)	87
H(10b)	0.407(8)	0.965(6)	0.105(5)	63
H(10c)	0.214(10)	0.914(7)	0.157(7)	90
H(11)	-0.307(6)	0.528(4)	0.076(4)	20

* These atoms were assigned anisotropic temperature factors of the form:

$$T = \exp\left[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}\ell^2c^{*2} + 2U_{23}k\ell b^*c^* + 2U_{31}\ell hc^*a^* + 2U_{12}hka^*b^*)\right]$$

with final parameters (U_{ij}x10^4Å^2):

Atom	U_{11}	U_{22}	U_{33}	$2U_{23}$	$2U_{31}$	$2U_{12}$
Fe	378	443	371	58	15	-52
O(1)	390	668	706	129	-299	-244
O(2)	462	505	584	203	-70	-141
O(3)	394	867	709	42	-262	-97
O(4)	657	1040	412	74	138	-104
O(5)	985	633	1146	67	345	-692
O(6)	577	791	874	380	-140	371
N(1)	320	463	422	55	-121	-69
C(2)	364	472	432	-14	-59	153
C(3)	348	521	416	-75	-14	90
C(4)	312	589	479	36	101	-64
C(5)	467	592	434	90	230	16
C(6)	562	569	323	-6	-28	64
C(7)	364	458	440	-15	-185	-7
C(8)	428	484	422	-94	29	-132
C(9)	642	577	669	72	53	-424
C(10)	967	566	767	273	135	-204
C(11)	399	665	512	-2	-6	147
C(12)	403	556	458	-39	22	2
C(13)	550	589	616	118	113	-151
C(14)	512	487	550	205	-2	6

<u>Average estimated standard deviations</u>. (U_{ij}x10^4Å^2) <u>and</u> (U_{iso}x10^3Å^2)

Atom	U_{11} or U_{iso}	U_{22}	U_{33}	$2U_{23}$	$2U_{31}$	$2U_{12}$
Fe	2	3	2	4	4	4
O	18	20	20	31	30	30
N	14	15	15	24	23	23
C	21	21	21	33	33	33
H	13					

TABLE 4

(N-Ethoxycarbonyl-3-formylazepine)tricarbonyliron;

bond lengths (Å), with esd in parentheses, involving the hydrogen atoms.

C(2)	-	H(2)	0.95(3)	C(9)	-	H(9a)	1.10(5)
C(4)	-	H(4)	0.96(3)	C(9)	-	H(9b)	0.89(5)
C(5)	-	H(5)	0.95(5)	C(10)	-	H(10a)	0.88(7)
C(6)	-	H(6)	0.92(4)	C(10)	-	H(10b)	0.87(6)
C(7)	-	H(7)	0.93(3)	C(10)	-	H(10c)	1.06(7)
C(11)	-	H(11)	1.04(4)				

TABLE 5

(N-Ethoxycarbonyl-3-formylazepine)tricarbonyliron; valency angles (°) with esd in parentheses.

C(12) - Fe - C(13)	100.3(2)	N(1) - C(8) - O(1)	123.1(3)
C(12) - Fe - C(14)	99.8(2)	N(1) - C(8) - O(2)	113.3(3)
C(13) - Fe - C(14)	93.4(2)	O(1) - C(8) - O(2)	125.5(3)
Fe - C(12) - O(4)	176.9(4)	C(8) - O(2) - C(9)	114.2(3)
Fe - C(13) - O(5)	178.0(4)	O(2) - C(9) - C(10)	106.0(4)
Fe - C(14) - O(6)	179.3(4)	C(3) - C(11) - O(3)	125.2(4)
Fe - C(4) - C(5)	66.5(2)	C(3) - C(11) - H(11)	114(2)
Fe - C(4) - C(3)	114.2(2)	O(3) - C(11) - H(11)	121(2)
Fe - C(4) - H(4)	121(2)	N(1) - C(2) - H(2)	116(2)
Fe - C(7) - C(6)	68.1(2)	C(3) - C(2) - H(2)	119(2)
Fe - C(7) - N(1)	120.6(2)	C(3) - C(4) - H(4)	110(2)
Fe - C(7) - H(7)	118(2)	C(5) - C(4) - H(4)	114(2)
C(4) - Fe - C(5)	39.7(1)	C(4) - C(5) - H(5)	118(3)
C(4) - Fe - C(6)	71.2(2)	C(6) - C(5) - H(5)	121(3)
C(4) - Fe - C(7)	82.5(1)	C(5) - C(6) - H(6)	122(2)
C(5) - Fe - C(6)	40.1(2)	C(7) - C(6) - H(6)	119(3)
C(5) - Fe - C(7)	72.3(1)	C(6) - C(7) - H(7)	110(2)
C(6) - Fe - C(7)	40.4(1)	N(1) - C(7) - H(7)	109(2)
N(1) - C(2) - C(3)	124.3(3)	O(2) - C(9) - H(9a)	108(3)
C(2) - C(3) - C(4)	127.8(3)	O(2) - C(9) - H(9b)	107(3)
C(2) - C(3) - C(11)	115.2(3)	H(9a) - C(9) - H(9b)	118(4)
C(4) - C(3) - C(11)	117.0(3)	C(10) - C(9) - H(9a)	111(3)
C(3) - C(4) - C(5)	125.8(3)	C(10) - C(9) - H(9b)	105(3)
C(4) - C(5) - C(6)	119.1(3)	C(9) - C(10) - H(10a)	111(4)
C(5) - C(6) - C(7)	119.1(3)	C(9) - C(10) - H(10b)	114(4)
C(6) - C(7) - N(1)	125.0(3)	C(9) - C(10) - H(10c)	104(4)
C(7) - N(1) - C(2)	123.2(3)	H(10a)- C(10) - H(10b)	109(6)
C(7) - N(1) - C(8)	115.9(3)	H(10a)- C(10) - H(10c)	114(6)
C(8) - N(1) - C(2)	120.9(3)	H(10b)- C(10) - H(10c)	105(5)

TABLE 6

(N-Ethoxycarbonyl-3-formylazepine)tricarbonyliron; torsion angles (^{o}).

N(1) - C(2) - C(3) - C(4)	2	H(7) - C(7) - N(1) - C(2)	174
N(1) - C(2) - C(3) - C(11)	-177	C(6) - C(7) - N(1) - C(8)	129
H(2) - C(2) - C(3) - C(4)	-174	H(7) - C(7) - N(1) - C(8)	-6
H(2) - C(2) - C(3) - C(11)	8	C(7) - N(1) - C(2) - C(3)	3
C(2) - C(3) - C(4) - C(5)	40	C(7) - N(1) - C(2) - H(2)	179
C(2) - C(3) - C(4) - H(4)	-177	C(8) - N(1) - C(2) - C(3)	-178
C(11)- C(3) - C(4) - C(5)	-142	C(8) - N(1) - C(2) - H(2)	-2
C(11)- C(3) - C(4) - H(4)	1	C(2) - N(1) - C(8) - O(1)	179
C(2) - C(3) - C(11)- O(3)	178	C(2) - N(1) - C(8) - O(2)	0
C(2) - C(3) - C(11)- H(11)	-9	C(7) - N(1) - C(8) - O(1)	-2
C(4) - C(3) - C(11)- O(3)	1	C(7) - N(1) - C(8) - O(2)	179
C(4) - C(3) - C(11)- H(11)	172	N(1) - C(8) - O(2) - C(9)	178
C(3) - C(4) - C(5) - C(6)	-49	O(1) - C(8) - O(2) - C(9)	-1
C(3) - C(4) - C(5) - H(5)	148	C(8) - O(2) - C(9) - C(10)	-178
H(4) - C(4) - C(5) - C(6)	170	C(8) - O(2) - C(9) - H(9a)	63
H(4) - C(4) - C(5) - H(5)	6	C(8) - O(2) - C(9) - H(9b)	-66
C(4) - C(5) - C(6) - C(7)	-3	O(2) - C(9) - C(10)- H(10a)	-66
C(4) - C(5) - C(6) - H(6)	-180	O(2) - C(9) - C(10)- H(10b)	172
H(5) - C(5) - C(6) - C(7)	160	O(2) - C(9) - C(10)- H(10c)	58
H(5) - C(5) - C(6) - H(6)	-17	H(9a)- C(9) - C(10)- H(10a)	52
C(5) - C(6) - C(7) - N(1)	60	H(9a)- C(9) - C(10)- H(10b)	-71
C(5) - C(6) - C(7) - H(7)	-166	H(9a)- C(9) - C(10)- H(10c)	175
H(6) - C(6) - C(7) - N(1)	-124	H(9b)- C(9) - C(10)- H(10a)	-179
H(6) - C(6) - C(7) - H(7)	10	H(9b)- C(9) - C(10)- H(10b)	58
C(6) - C(7) - N(1) - C(2)	-52	H(9b)- C(9) - C(10)- H(10c)	-56

The sign of the torsion angle is negative if an anticlockwise rotation of A is required to eclipse D whilest looking down the B - C bond, the atom labelling being from left to right in the above table.

TABLE 7

(N-Ethoxycarbonyl-3-formylazepine)tricarbonyliron; displacements (Å) of atoms from various planes.

	Atoms in plane	Displacements	Atoms out of plane	Displacements
a)	C(4)	-0.007	H(4)	0.17
	C(5)	0.014	H(5)	0.28
	C(6)	-0.014	H(6)	0.00
	C(7)	0.007	H(7)	0.19
			Fe	1.588
b)	N(1)	0.003	C(7)	0.096
	C(2)	-0.006	C(8)	-0.062
	C(3)	0.006	O(1)	-0.078
	C(4)	-0.003	O(2)	-0.127
			C(9)	-0.235
			C(10)	-0.350
			H(2)	-0.09
			C(11)	0.072
			O(3)	0.135
			H(11)	-0.05
c)	C(7)	0.023	C(8)	-0.097
	N(1)	-0.029	C(11)	0.135
	C(2)	0.004		
	C(3)	0.022		
	C(4)	-0.021		
d)	C(3)		H(4)	-0.54
	C(4)			
	C(5)			
e)	C(6)		H(7)	-0.63
	C(7)			
	N(1)			
f)	C(7)		N(1)	-0.006
	C(8)			
	C(2)			

	Atoms in plane	Displacements	Atoms out of plane	Displacements
g)	C(8)	-0.007	N(1)	-0.051
	O(1)	-0.007	H(9a)	0.94
	O(2)	0.011	H(9b)	-0.76
	C(9)	0.019	H(10a)	0.72
	C(10)	-0.017	H(10b)	-0.13
			H(10c)	-0.90
h)	C(3)	-0.008	C(2)	0.103
	C(11)	0.031	C(4)	-0.127
	O(3)	-0.011		
	H(11)	-0.011		
i)	C(13)	-0.021	Fe	0.521
	C(14)	0.022		
	C(4,5)†	0.027		
	C(6,7)†	-0.027		

† C(X,Y) is the midpoint of the C(X) - C(Y) bond.

TABLE 8

(N-Ethoxycarbonyl-3-formylazepine)tricarbonyliron; intermolecular contacts (≤3.60Å).

O(3)	- - -	$O(4^{I})$	3.05
O(6)	- - -	$O(6^{II})$	3.11
O(3)	- - -	$C(12^{I})$	3.16
O(1)	- - -	$C(11^{III})$	3.17
O(1)	- - -	$C(6^{IV})$	3.22
O(1)	- - -	$C(7^{IV})$	3.26
O(1)	- - -	$O(3^{III})$	3.33
O(3)	- - -	$C(9^{V})$	3.34
O(2)	- - -	$O(3^{V})$	3.36
O(2)	- - -	$C(11^{V})$	3.36
O(3)	- - -	$C(11^{VI})$	3.38
O(3)	- - -	$C(8^{I})$	3.39
O(5)	- - -	$C(9^{VII})$	3.44
N(1)	- - -	$C(5^{VIII})$	3.44
C(2)	- - -	$C(6^{VIII})$	3.45
C(5)	- - -	$C(8^{VIII})$	3.45
O(6)	- - -	$C(4^{III})$	3.47
O(3)	- - -	$C(7^{I})$	3.50
O(3)	- - -	$O(3^{VI})$	3.51
O(3)	- - -	$N(1^{I})$	3.51
O(4)	- - -	$C(10^{V})$	3.51
O(4)	- - -	$O(5^{IX})$	3.52
O(5)	- - -	$C(14^{X})$	3.52
N(1)	- - -	$C(6^{VIII})$	3.55
C(8)	- - -	$C(11^{III})$	3.55
O(6)	- - -	$C(5^{III})$	3.57
O(3)	- - -	$C(10^{V})$	3.58

The superscripts refer to the following transformations of the atomic coordinates:

I	-1 + x,	y,	z;
II	1 - x,	- y,	1 - z;
III	1 + x,	y,	z;
IV	1 - x,	1 - y,	1 - z;
V	- x,	1 - y,	- z;
VI	-1 - x,	1 - y,	- z;
VII	-1 + x,	-1 + y,	z;
VIII	- x,	1 - y,	1 - z;
IX	- x,	- y,	- z;
X	- x,	- y,	1 - z.

II.1.3 DISCUSSION

For comparison purposes, selected bond lengths in azepine-Fe$(CO)_3$ complexes investigated by X-ray analysis are given in Table 9.

TABLE 9

Selected bond lengths (Å) in the Fe$(CO)_3$ complexes of 1H-azepine(22), A, 3-acetyl-1H-azepine(23), B, N-methoxycarbonylazepine(24), C, and N-ethoxycarbonyl-3-formylazepine, D.

Bond†	A††	B	C	D
Fe - C(4)	2.206(5)	2.182(7)	2.145(10)	2.151(4)
Fe - C(5)	2.033(5)	2.063(8)	2.059(10)	2.053(4)
Fe - C(6)	2.039(5)	2.055(9)	2.041(9)	2.051(4)
Fe - C(7)	2.174(4)	2.168(9)	2.091(8)	2.096(4)
N(1) - C(2)	1.352(7)	1.343(8)	1.382(11)	1.379(4)
N(1) - C(7)	1.402(6)	1.391(10)	1.436(11)	1.447(5)
C(2) - C(3)	1.322(7)	1.364(12)	1.334(14)	1.343(5)
C(3) - C(4)	1.451(7)	1.458(11)	1.439(14)	1.465(5)
C(4) - C(5)	1.414(7)	1.457(10)	1.398(14)	1.431(5)
C(5) - C(6)	1.407(7)	1.392(13)	1.409(13)	1.406(6)
C(6) - C(7)	1.409(7)	1.464(13)	1.440(12)	1.431(5)
C(3) - C(11)		1.467(9)		1.458(5)
N(1) - C(8)			1.372(11)	1.406(5)
C(8) - O(1)			1.192(12)	1.201(5)
C(8) - O(2)			1.334(12)	1.325(5)
O(2) - C(9)			1.465(15)	1.461(5)
C(9) - C(10)				1.507(7)
Fe - C(12)	1.765(4)	1.791(8)	1.713(11)	1.805(4)
Fe - C(13)	1.779(4)	1.799(10)	1.784(10)	1.807(4)
Fe - C(14)	1.782(4)	1.802(6)	1.779(10)	1.799(4)
C(12)- O(4)	1.142(6)	1.138(10)	1.153(14)	1.125(5)
C(13)- O(5)	1.138(5)	1.148(14)	1.123(12)	1.134(6)
C(14)- O(6)	1.144(6)	1.137(9)	1.142(13)	1.130(5)

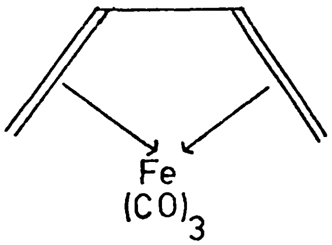
† The numbering scheme conforms to that in D.

†† All bond lengths in A are an average of values in two crystallographically independent molecules.

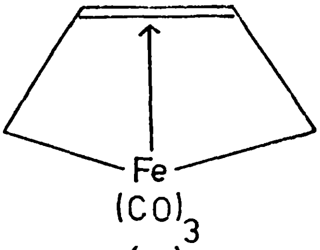
In this analysis, as in the 3-acetyl-1H-azepine(23) complex, the $Fe(CO)_3$ group is complexed to atoms C(4) - - - C(7) rather than C(2) - - - C(5), in agreement with Nicholson's argument(36) that during complex formation it is energetically more favourable for the $Fe(CO)_3$ group to interact with the more localised butadiene group, thereby retaining maximum conjugation in the complex.

The iron atom is significantly closer to the central atoms of the butadiene group, with mean Fe - C(central) and Fe - C(terminal) distances of 2.052 and 2.124 Å respectively. These compare favourably with corresponding mean values of 2.048 and 2.161 Å in A, B and C, and with values in a variety of 1,3-diene $Fe(CO)_3$ complexes, e.g. 2.050 and 2.131 Å in sorbic acid $Fe(CO)_3$(5), 2.038 and 2.114 Å in unsymmetrical 12-oxa[4.4.3]propella-2,4,7,9-tetraene bis-$Fe(CO)_3$(15) and 2.05 and 2.18 Å in (COT)$Fe(CO)_3$(27). The Fe - C(terminal) bonds are nonequivalent, cf. Fe - C(4) and Fe - C(7) bond lengths of 2.151(4) and 2.096(4) Å respectively; an asymmetry which probably occurs in A, B and C (see Table 9).

The terminal C - C bond lengths, both 1.431 Å, and the central C - C bond, 1.406 Å, differ significantly from those in an uncomplexed 1,3-diene system, e.g. 1.337 and 1.483 Å respectively in trans-butadiene(37). This apparent flow of electron density from the terminal bonds to the central bond can be explained either by a valence bond approach, in which the complex is considered as a hybrid of π (I) and σ-π (II) canonical forms, or by a molecular orbital

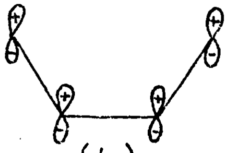


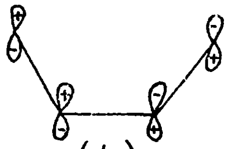
(I)

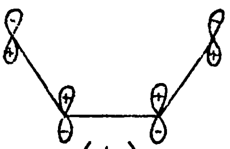


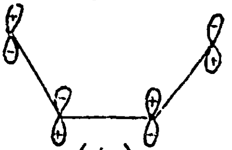
(II)

approach, in which a smooth transition from (I) to (II) occurs with increasing back-donation into the lowest antibonding orbital (ψ_3) of the butadiene group$^{(2)}$. In the Hückel molecular orbital representation of cis-butadiene$^{(38)}$, shown above, the doubly occupied orbitals (ψ_1) and (ψ_2) are of appropriate symmetry for σ-donation, while the vacant (ψ_3) and (ψ_4) orbitals are appropriate for π-acceptance from filled metal d orbitals. The back-donation/contribution of (II) is increased by the presence of an electron-withdrawing substituent in the 3-position of the azepine ring. For example, in 1H-azepine $Fe(CO)_3$$^{(22)}$ the C – C bonds within the butadiene group are essentially the same [1.414(7), 1.407(7) and 1.409(7) Å], whereas in 3-acetyl-1H-azepine-$Fe(CO)_3$$^{(23)}$ [1.457(10), 1.392(13) and 1.467(9) Å] and to a lesser and not necessarily significant extent in this analysis [1.431(5), 1.406(6) and 1.431(5) Å] the terminal bonds are longer than the central bond.


(ψ_1)


(ψ_2)


(ψ_3)


(ψ_4)

Other evidence of the contribution of (II) in this analysis is the partial sp^3 hybridisation of the terminal atoms, as indicated by displacements of H(4) from the C(3) - C(4) - C(5) plane and H(7) from the C(6) - C(7) - N(1) plane by 0.54 and 0.63 Å respectively. The resulting displacements of these hydrogen atoms from the essentially planar butadiene group, 0.17 and 0.19 Å respectively, are towards the iron atom, as predicted$^{(39)}$ by Clinton and Lillya from a conformational analysis of 1,3-diene $Fe(CO)_3$ complexes. However, the hybridisation of the terminal atoms is not ideally sp^3. The C - C(4) - H(4) angles, 110(2) and 114(2)°, the C(6) - C(7) - H(7) angle, 110(2)°, and the N(1) - C(7) - H(7) angle, 109(2)°, do not differ significantly from the tetrahedral angle of 109°28', but the C(3) - C(4) - C(5) and

N(1) - C(7) - C(6) angles, 125.8(3) and 125.0(3)° respectively, are larger than a normal sp^2 angle.

Torsion angles in complexed and uncomplexed azepine rings are given in Table 10.

TABLE 10

Selected torsion angles (°) in N-methoxycarbonylazepine $Fe(CO)_3$ (24), C, N-ethoxycarbonyl-3-formylazepine $Fe(CO)_3$, D, N-p-bromobenzenesulphonyl-azepine (24), E, and N-phenoxycarbonylazepine (29), F.

Torsion angle†	Complexed azepines		Uncomplexed azepines	
	C	D	E	F
C(7) - N(1) - C(2) - C(3)	-1	3	64	54
N(1) - C(2) - C(3) - C(4)	5	2	-3	-1
C(2) - C(3) - C(4) - C(5)	40	40	-34	-28
C(3) - C(4) - C(5) - C(6)	-50	-49	1	-1
C(4) - C(5) - C(6) - C(7)	-4	-3	35	28
C(5) - C(6) - C(7) - N(1)	60	60	-2	4
C(6) - C(7) - N(1) - C(2)	-49	-52	-61	-55
C(6) - C(7) - N(1) - X††	134	129	147	143
X - N(1) - C(2) - C(3)	176	182	-143	-144

† The numbering scheme and sign convention conform to that in D.

†† X is the exocyclic atom bonded to N(1).

Inspection of the above table illustrates that complex formation involves substantial conformational changes to the azepine ring. In the free state a boat conformation is adopted with C(2), C(3), C(6) and C(7) being effectively coplanar, whereas when complexed with $Fe(CO)_3$ the ring is divided between two nearly planar groups of atoms [C(4), C(5), C(6), C(7) and C(4), C(3), C(2), N(1), C(7)]. The dihedral angle between these planes in this analysis, 138°, compares favourably with the corresponding angles of 137° in N-methoxycarbonylazepine-$Fe(CO)_3$ (24), 140° in 3-acetyl-1H-azepine $Fe(CO)_3$ (23) and 143° in

1H-azepine $Fe(CO)_3$(22). Another conformational difference is the greater planarity about the nitrogen atom in the complexes. For example, in N-p-bromobenzenesulphonylazepine(22) and N-phenoxycarbonylazepine(29) the nitrogen atom deviates considerably, 0.22 and 0.13 Å respectively, from the plane defined by its three bonded atoms, but in N-methoxycarbonylazepine $Fe(CO)_3$(24) and N-ethoxycarbonyl-3-formylazepine $Fe(CO)_3$ the corresponding deviations are only 0.020 and 0.006 Å respectively, suggesting that in these complexes the nitrogen is effectively sp^2 hybridised.

The planes of the formyl and ethoxycarbonyl substituents are inclined at angles of 6.8 and 4.9° respectively to the N(1), C(2), C(3), C(4) plane. This near planarity may reflect conjugative interactions of the nitrogen lone-pair with the formyl substituent via the C(2) - C(3) bond and with the ethoxycarbonyl group.

The former type of interaction has been suggested(23) by Sim to account for the probably significant lengthening of the C(2) - C(3) bond in 3-acetyl-1H-azepine $Fe(CO)_3$(23), 1.364(12) Å, compared both with the corresponding bond in 1H-azepine $Fe(CO)_3$(22), 1.322(7) Å, and with the bond in ethylene(40), 1.334(3) Å. In this analysis the lone-pair also interacts with the ethoxycarbonyl group, as demonstrated by the double-bond character of the N(1) - C(8) bond, 1.406 Å, therefore the C(2) - C(3) bond length should be intermediate between the above values. Although the small Δ/σ ratios prevent any definite assertions, it is interesting to note that an intermediate value, 1.343(5) Å, is indeed found. Similarly the apparent shortening of the C(3) - C(substituent) bond in 3-acetyl-1H-azepine $Fe(CO)_3$(23), 1.467(9) Å, and N-ethoxycarbonyl-3-formylazepine $Fe(CO)_3$, 1.458(5) Å, compared with an ideal single bond, 1.48 Å, between sp^2 carbon atoms, although not statistically significant, is nevertheless consistent with conjugation. Better evidence of interaction of the lone-pair with the C(2) - C(3) bond is the significant shortening in each complex of the N(1) - C(2) bond compared both with the N(1) - C(7) bond, cf. N(1) - C(2) = 1.379(4) Å and N(1) - C(7) = 1.447(5) Å in this analysis, and with the N - C bonds in uncomplexed azepines, e.g. 1.41(1) and 1.42(1) Å in N-phenoxycarbonylazepine(29).

Interaction of the lone-pair with an N-alkoxycarbonyl group results in the significant lengthening of both the N(1) - C(2) and N(1) - C(7) bonds, cf. 1.352(7) and 1.402(6) Å respectively in 1H-azepine $Fe(CO)_3$(22) and 1.379(4) and 1.447(5) Å in this analysis. These competing conjugative interactions should be reflected in the N - C(exocyclic) bond in N-ethoxycarbonyl-3-formylazepine $Fe(CO)_3$, 1.406(5) Å, being longer than in N-methoxycarbonylazepine $Fe(CO)_3$(24), 1.372(11) Å, with both lengthened compared to the N - C bond in ethyl carbamate(41), 1.349(4) Å, in which the only possible lone-pair interaction is with the ester group. The observed trend is not unequivocal, but is in the sense expected.

The geometry of the ethoxycarbonyl group is in good agreement with that of ethyl carbamate(41) and the methoxycarbonyl group in N-methoxycarbonylazepine $Fe(CO)_3$(24). The non-hydrogen atoms are nearly planar, the root-mean-square deviation from planarity being 0.013 Å, with the hydrogen atoms, as demonstrated by the torsion angles, in a staggered conformation. The mean C - H bond length in the ethyl group, 0.96 Å, is the same as that found in the rest of the molecule. Similar contractions from the spectroscopic value of 1.07 Å have been observed in other X-ray analyses(42) and can be attributed to the displacement of the hydrogen electron density towards the carbon atom during bond formation(43).

The carbonyl of the ester group is cis to the complexed butadiene group in agreement with the preferred conformation of N-ethoxycarbonylazepine $Fe(CO)_3$, as indicated by its temperature dependent n.m.r. spectrum(44), and with the conformations found in the X-ray analyses of N-methoxycarbonylazepine $Fe(CO)_3$(24) and 1-isopropoxycarbonyl-1,2-diazepine $Fe(CO)_3$(25).

When the molecule is viewed in projection on to the butadiene plane (see Figure 2), two of the carbonyl ligands eclipse the two formal double bonds of the diene; such an arrangement is normal(38) in 1,3-diene $Fe(CO)_3$ complexes. The coordination about the iron atom can be described either, by considering canonical form (II), as a distorted octahedron or, by considering form (I), as a distorted square pyramid with the iron atom 0.52 Å above the plane through atoms C(13) and C(14) and the midpoints of the C(4) - C(5) and C(6) - C(7) bonds, and with atom C(12) occupying the apical position. In both descriptions the trans environment of carbonyl C(12) - O(3) is different from that of the other carbonyls, allowing the possibility of different Fe - CO bond lengths. Apical lengthening has been observed in other types of d^8 square pyramidal complexes(45), but in the present analysis, as seems typical(23) of 1,3,-diene $Fe(CO)_3$ complexes, the Fe - CO bond lengths are essentially the same [1.805(4), 1.807(4) and 1.799(4) Å]. [N-methoxycarbonylazepine $Fe(CO)_3$(24) is a curious exception with an apical Fe - CO bond length of 1.713(11) Å and basal Fe - CO bond lengths of 1.784(10) and 1.779(10) Å.] The average Fe - CO and C - O bond lengths, 1.804 and 1.130 Å respectively, can be compared with mean values of 1.779 and 1.147 Å respectively, calculated by Birnbaum(14) from 20 bonds in related structures. The nonequivalence of the carbonyl groups is however reflected in the angle between the basal Fe - CO bonds, 93.4°, being significantly smaller than the angles between apical and basal Fe - CO bonds, 100.3 and 99.8°. This angular disposition is common to other 1,3-diene $Fe(CO)_3$ complexes, cf. mean angles of 90.2, 102.5 and 98.4° in sorbic acid $Fe(CO)_3$(5). The Fe - C - O angles [176.9(4), 178.0(4) and 179.3(4)°] depart only slightly from linearity. Kettle has pointed out(46) that small deviations from linearity may arise both from nonequivalence of the two distinct sets of π^* orbitals on the carbonyl groups and from crystal packing forces. In this analysis the crystal packing does not involve any abnormal intermolecular contacts.

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CHAPTER 2

TWO MOLYBDENUM COMPLEXES

II.2.1 INTRODUCTION

Investigation into the bonding in organometallic complexes is often undertaken by the structural comparison of a series of closely related complexes. Seven-coordinate molybdenum complexes of the type $(\pi\text{-}C_5H_5)MoL_2XY$ or $(\pi\text{-}C_5H_5)MoL_3X$ represent one series for which numerous structure analyses have been reported, e.g. trans-$(\pi\text{-}C_5H_5)Mo(CO)(PPh_3)_2(NCO)$(1), $(\pi\text{-}C_5H_5)Mo(CO)(PPh_2{\cdot}CH_2{\cdot}CH_2{\cdot}PPh_2)C\ell$(2), trans-$(\pi\text{-}C_5H_5)Mo(CO)_2(PPh_3)(COMe)$(3), trans-$(\pi\text{-}C_5H_5)Mo(CO)_2(PPh_3)I$(2), trans-$(\pi\text{-}C_5H_5)Mo(CO)_2[P(OMe)_3]I$(4), trans-$(\pi\text{-}CH_3C_5H_4)Mo(CO)_2[P(OMe)_3]I$(4), $(\pi\text{-}C_5H_5)Mo(CO)_3C\ell$(5) and $(\pi\text{-}C_5H_5)Mo(CO)_3(C_3F_7)$(6). Therefore, in order to augment the number of possible comparisons, crystal structure analyses were undertaken on compounds with different ligands, i.e. $(\pi\text{-}C_5H_5)Mo(CO)_2(PPh_3)Br$, A, and $(\pi\text{-}C_5H_5)Mo(CO)_2(CNPh)I$, B.

Both A and B were supplied by Dr. G. R. Knox of Strathclyde University. The former was prepared(7) by refluxing $(\pi\text{-}C_5H_5)Mo(CO)_3Br$ with triphenylphosphine and recrystallising from 60-80 petrol/methylene chloride, while the latter was prepared(8) by the treatment of $(\pi\text{-}C_5H_5)Mo(CO)_3I$ with phenyl isocyanide. Interconversion of isomers of $(\pi\text{-}C_5H_5)Mo(CO)_2XY$ occurs in solution(7), therefore for both complexes the recrystallisation solution contained an equilibrium mixture of isomers. In the case of A the crystals isolated were spectroscopically assigned(9) as the cis-isomer, while for B there was difficulty in obtaining only crystals of one isomer, and the crystal used in the X-ray analysis was selected from a mixture (predominantly trans) of isomers.

$(\pi\text{-}C_5H_5)Mo(CO)_2(CNPh)I$ is of particular interest in view of the growth, in recent years, of interest in transition-metal isocyanide chemistry(10 - 13). Although isocyanides are isoelectronic with carbon monoxide, the marked decrease in ν(CO) on coordination in not mirrored by a similar decrease in ν(CN) and indeed increases are often observed(14). These increases in ν(CN) result from the greater contribution of canonical form (II) on coordination(15) and have been

$$\underset{\text{(I)}}{R{-}\ddot{N}{=}C:} \longleftrightarrow \underset{\text{(II)}}{R{-}\overset{+}{N}{\equiv}\overset{-}{C}}$$

generally interpreted(10, 15 - 17) as indicating little back-donation by the metal. However in cis-$[PtC\ell_2(CNEt)(PEt_2Ph)]$ (18) and cis-$[PtC\ell_2(CNPh)_2]$(19) both large increases in ν(CN) and appreciable double-bond character in the Pt - C bonds have been found.

This analysis of $(\pi\text{-}C_5H_5)Mo(CO)_2(CNPh)I$ should not only yield information on the relationship between ν(CN) and the extent of M$\rightarrow$C back-donation, but also enable a comparison to be made of the π-accepting abilities of carbonyl and isocyanide ligands.

II.2.2 EXPERIMENTAL

Crystal Data π-Cyclopentadienyl-cis-dicarbonylbromo(triphenylphosphine)-molybdenum - methylene chloride; $C_{25}H_{20}O_2PBrMo$, $0.25CH_2C\ell_2$.

Crystal system	Triclinic
Unit cell dimensions	a = 18.927(4) Å
	b = 12.335(2) Å
	c = 11.003(2) Å
	α = 103°37'(1)
	β = 105°35'(1)
	γ = 99°42'(1)
Space group	$P\bar{1}$ (C_i^1)

U = 2330.1 $Å^3$

M = 580.3 a.m.u.

D_o = 1.66 $gm.cm.^{-3}$ (flotation in $MeI/CC\ell_4$ solution)

D_c = 1.654 $gm.cm.^{-3}$

Z = 4

F(000) = 1154

μ(Mo-$K\alpha$) = 25.19 $cm.^{-1}$

Crystallographic Measurements

Preliminary triclinic cell dimensions were determined from precession photographs of a red polyhedron-shaped crystal of dimensions ca. 0.20 x 0.20 x 0.25 mm^3.

The crystal, having been transfered to a Hilger and Watts' Y290 automatic diffractometer controlled by a PDP-8 computer, was offset on χ by about 5° to prevent multiple reflections(20). A fully stabilized X-ray generator was operated at 46 kV and 16 mA. The cell dimensions were then adjusted by a least-squares treatment(21) of the θ, χ and ϕ setting angles of eleven reflections measured with zirconium-filtered Mo-$\underline{K}\alpha$ radiation.

The intensity data were collected, in octants $hk\ell$, $hk\bar{\ell}$, $h\bar{k}\ell$ and $h\bar{k}\bar{\ell}$ with θ(Mo-$\underline{K}\alpha$) $\leqslant 25^{\circ}$, by the θ - 2θ scan procedure, each reflection being scanned in 36 equal steps from $2\theta_{calc.}$ -0.72° to $2\theta_{calc.}$ $+0.72^{\circ}$. Each step was counted for 2 seconds and stationary-crystal, stationary-counter background counts were measured at each end of the scan range for 18 seconds. Monitoring of the intensities of two standard reflections every 40 intensity measurements revealed drifts of about 10%, therefore the orientation matrix was redetermined after about every 2,000 intensity measurements. The data were scaled according to the intensities of these standards and the scan counts (P) and the background counts (B_1,B_2) of each reflection combined to yield the integrated intensity, $I = P - 2(B_1 + B_2)$, which was corrected for Lorentz-polarisation effects but not for absorption. 6,944 independent values of $|F_o|$ were obtained, of which the 5,212 observations with $I/\sigma_I^{\dagger} > 3.0$ were used in the structure analysis.

† σ_I, the standard deviation in the measured intensity, I, is derived from counting statistics by the formula:

$$\sigma_I = \surd(P + B_1 + B_2)$$

Structure Analysis

The three-dimensional Patterson synthesis was readily interpreted in terms of space group $P\bar{1}$ with two crystallographically independent molecules per asymmetric unit, giving the positions of the two unique molybdenum atoms, which were then used to phase an electron density distribution (R = 53%), revealing coordinates for both bromine and both phosphorus atoms. The electron density distribution (R = 35%) phased by these six heavy atoms was contoured on to glass sheets, yielding for both molecules the positions of all the remaining non-hydrogen atoms except the atoms of the carbonyl group trans to the bromine atom. The newly found atoms were included with the heavy atoms in a further structure factor calculation (R = 21%), but the subsequent electron density distribution still contained a region of electron density in both molecules trans to the bromine atom, which was unresolvable into distinct peaks. Another unexplained and prominent feature of this map was a peak (peak height = $13.0e^-/\AA^3$ or 1.5 times the height of the highest carbon atom) making no bonding contacts with either of the molecules and about 1.45 Å from a centre of symmetry. These maps despite the unexplained features, confirmed that the space group is $P\bar{1}$ and not the alternative P1.

The three unexplained peaks were each assigned the scattering curve of oxygen in the initial least-squares calculations. After three cycles of full-matrix refinement on the positional and isotropic thermal parameters of all the atoms convergence was reached at R = 13.8%. Low thermal parameters (average $B_{iso} = 1.7\AA^2$) resulted for each of the three unexplained peaks indicating that the scattering assigned to them was underestimated. A difference electron density distribution was calculated revealing peaks (average peak height = $4.6e^-/\AA^3$) at either side of the 'oxygen' atom in both molecules (see Table 1).

TABLE 1

Geometry involving the molybdenum atoms, the 'oxygen' atoms and the peaks (P) found in the difference electron density distribution (R = 13.8%)

	A1†	A101†
Mo - 'O' bond length (Å)	2.77	2.73
Mo - P_1 bond length (Å)	2.05	2.15
P_1 - P_2 bond length (Å)	1.16	0.98
Mo - P_1 - P_2 valency angles (°)	168.6	163.4

† A1 and A101 refer to the molécules containing Mo(1) and Mo(101) respectively.

Comparison of the above values with expected$^{(1 - 6)}$ Mo - Br, Mo - C(carbonyl) and C - O bond lengths of ca. 2.65, 1.96 and 1.14 Å respectively strongly suggested disorder in both molecules between the bromine atom and the carbonyl group trans to it (illustrated in Figure 1 page 61). This disorder is a consequence of the similar steric requirements of a bromine atom and a carbonyl group enabling both enantiomorphs of cis-$(\pi\text{-}C_5H_5)Mo(CO)_2(PPh_3)Br$ to be almost mutually replaceable during the crystallisation. Halo/carbonyl disorder has also been found in the crystal structures of $IrO_2C\ell(CO)(PPh_3)^{(22)}$, $IrC\ell(CO)_2(PPh_3)_2^{(23)}$, $[(n\text{-}C_4H_9)_4N]^+[RhC\ell_2(CO)_2]^{-(24)}$, $IrC\ell(CN)(NCS)(CO)(PPh_3)_2^{(25)}$, $IrBr(CO)(TCNE)(PPh_3)_2^{(26)}$ and $IrIO_2(CO)(PPh_3)_2^{(27)}$. The treatment of the disorder in this analysis is almost identical to that employed satisfactorily in $IrBr(CO)(TCNE)(PPh_3)_2^{(26)}$ in which a similar degree of disorder was found.

An initial model for the disorder was derived as follows: structure factors were calculated without any contribution from the disordered atoms (R = 39%), and from a comparison of the bromine peak heights in the subsequent difference electron density distribution, estimates of 0.79 and 0.74 were obtained for the extent of disorder in A1 and A101 respectively. Coordinates for the bromine atoms were taken from this map, while those for the disordered carbonyl groups were calculated corresponding to Mo - C and C - O bond lengths of 1.96 and 1.14 Å respectively with a linear Mo - C - Br - O geometry. Isotropic thermal parameters, B_{iso} = 5.0, 4.0 and 3.6Å^2, were assigned to these bromine, carbon and oxygen atoms respectively.

All the parameters of the disordered carbonyl groups plus the population parameters of the bromine atoms were held fixed and contributions from the remaining unexplained peak omitted, during two cycles of full-matrix least-squares refinement on the remaining isotropic thermal and positional parameters. A difference electron density distribution (R = 14.6%) was calculated showing firstly, that a reasonable model for the disorder was being employed, and secondly that the previously unexplained peak and the peak related by the adjacent centre of symmetry could be assigned to the chlorine atoms of a solvent methylene chloride molecule. The carbon atom of this molecule is necessarily disordered and appeared as two peaks (peak height = 1.4e^-/Å^3) related by the centre of symmetry. With the disordered solvent molecule included in the model the structure refinement proceeded as shown in Table 2.

TABLE 2

Progress of the least-squares refinement of cis-$(\pi\text{-}C_5H_5)Mo(CO)_2(PPh_3)Br$, $0.25CH_2C\ell_2$

Step	Treatment of parameters	Final R-factor
1)	3 cycles; 9 blocks; refinement of overall scale factor; x, y, z, U_{iso} for all the atoms except the disordered carbonyl groups.	10.0%
2)	1 cycle; 8 blocks; refinement of overall scale factor; x, y, z, U_{ij} for all the atoms except the disordered carbonyl groups, the bromine atoms and the carbon atom of the solvent.	8.9%
3)	2 cycles; 1 block; refinement of overall scale factor; x, y, z, U_{ij}, population parameter (PP.) for the bromine atoms; the sum of the PP.s of the bromine atoms in the same molecule constrained to unity.	6.9%
4)	PP. of each of the disordered carbonyl groups made equal to that of the appropriate bromine atom.	
5)	2 cycles; 11 blocks; refinement of overall scale factor; bromine atoms as in step 3; x, y, z, U_{iso} for the carbon atom of the solvent; x, y, z, U_{ij} for the remaining atoms except the disordered carbonyl groups.	6.7%

The results of this refinement were not entirely satisfactory as demonstrated both by the range of Mo - Br bond lengths obtained (0.07 Å), and by the occurrence of peaks and holes (0.82 to $-1.46e^-/Å^3$) in the difference synthesis in the region of the disordered groups. It was likely that these features were partly dependent on the thermal motion assigned to the fixed carbonyl groups, therefore this was investigated by fixing the B_{iso} of these carbon and oxygen atoms at various values and refining the bromine atoms as in step 3. The resulting bond length variations are given in Table 3.

TABLE 3

The effect of changing the B_{iso} (Å^2) of the carbon and oxygen atoms of the disordered carbonyl groups on the Mo - Br bond lengths (Å)

Part I: B_{iso} of the oxygen atoms varied while that of the carbon atoms fixed at 4.0Å^2

B_{iso} of oxygen atoms A1	 A101	Bond lengths of Mo - Br(1)	 Mo - Br(1)'	 Mo - Br(101)	 Mo - Br(101)'	R-factor (%)
3.6		2.673	2.631	2.664	2.606	6.664
5.0		2.676	2.655	2.666	2.622	6.564
6.0		2.676	2.686	2.669	2.638	6.516
7.0		2.677	2.705	2.670	2.648	6.485
8.0		2.677	2.720	2.671	2.658	6.464
6.0	9.0	2.676	2.685	2.672	2.666	6.460
6.0	11.0	2.676	2.686	2.674	2.676	6.445
6.0	13.0	2.676	2.685	2.675	2.689	6.439

Part II: B_{iso} of the carbon atoms varied while that of the oxygen atoms fixed at 6.0Å^2

B_{iso} of carbon atoms	Bond lengths of Mo - Br(1)	 Mo - Br(1)'	 Mo - Br(101)	 Mo - Br(101)'	R-factor (%)
3.0	2.676	2.691	2.669	2.643	6.532
4.0	2.676	2.686	2.669	2.638	6.516
5.0	2.675	2.679	2.668	2.632	6.528
6.0	2.675	2.670	2.668	2.628	6.545
7.0	2.675	2.660	2.667	2.622	6.571

The above table and the difference syntheses calculated at each step were examined with a view to obtaining both consistent Mo - Br bond lengths and minimum deviations in the difference map, while retaining physically reasonable thermal parameters for the disordered carbonyl groups. These carbon and oxygen atoms were therefore assigned B_{iso} = 5.0 and 7.5Å^2 respectively and, after least-squares refinement as in step 5, convergence was reached at R = 6.41%.

A difference synthesis was calculated revealing stereochemically acceptable positions for 33 of the 40 hydrogen atoms of A, at an average peak height of 0.52e^-/Å^3. All of the hydrogen atoms of the cyclopentadienyl rings were found, suggesting that disorder of these rings, if any, was minimal. In further least-squares calculations, the hydrogen atoms were not refined but were included in the structure factor calculations, at positions calculated assuming an idealized geometry with a C - H bond length of 0.98 Å and with an isotropic temperature factor equal to that of the atom to which it was bonded. Three cycles of least-squares refinement, treating the other parameters as in step 5, converged at R = 5.71%, R' = 4.95%. The peaks and holes in a final difference electron density distribution all lay between 0.76 and -0.86e^-/Å^3, the major deviations being near the disordered groups.

Examination of the final bond lengths revealed that the ordered carbonyl group in both molecules had a Mo - C bond length ca. 0.13 Å (11σ) longer than expected and a C - O bond length ca. 0.20 Å (13σ) shorter than expected. Abnormal bond lengths of this type have been found by Ibers$^{(23)}$ to be symptomatic of halo/carbonyl disorder therefore, in view of the reasonable estimated standard deviations in an ordered part of both molecules e.g. the triphenylphosphine groups, these anomalous values very probably reflect disorder between cis-(π-C_5H_5)Mo(CO)$_2$(PPh$_3$)Br and a small proportion of the trans-isomer rather than an underestimation of the estimated standard deviations in an ordered model. Since no evidence of this further disorder was found in any of the difference syntheses, the scattering due to the partial bromine is probably less than $2.5e^-$ in each molecule, i.e. less than 7% of the trans-isomer, and therefore no further calculations were undertaken.

Notes:

1) The atomic scattering factors, including dispersion corrections $\Delta f'$ and $\Delta f''$ for the molybdenum, bromine, phosphorus and chlorine atoms, were taken from 'International Tables for X-ray Crystallography,' Vol. III.(28)

2) The final weights employed in the least-squares refinement, in order to ensure approximate constancy of $\langle w\Delta^2 \rangle$ over various ranges of $|F_o|$ and $\sin\theta$, were given by the expression:

$$w^{-1} = 17 + 12\left[(|F_o| - 70)/35\right]^2 - 30\sin\theta$$

3) Most of the calculations were carried out, using the 'X-ray System' programs(29), on the Univac 1108 computer at the National Engineering Laboratory, East Kilbride, and on the IBM 370/155 computer of the Edinburgh Regional Computer Centre.

Results

The observed and final calculated structure factors are listed in Table 4, with the final positional and thermal parameters, including the estimated standard deviations derived from the least-squares refinement, in Table 5. The atomic numbering scheme of the non-hydrogen atoms (each hydrogen atom is numbered according to the atom to which it is bonded) is shown in Figure 1; A1 and A101, each projected on to the plane of its π-cyclopentadienyl ring illustrating the halo/carbonyl disorder. The bond lengths, valency angles and mean plane calculations are given in Tables 6, 7 and 8 respectively. The crystal packing is shown in Figure 2 and the intermolecular contacts $\leqslant 3.75$ Å are listed in Table 9.

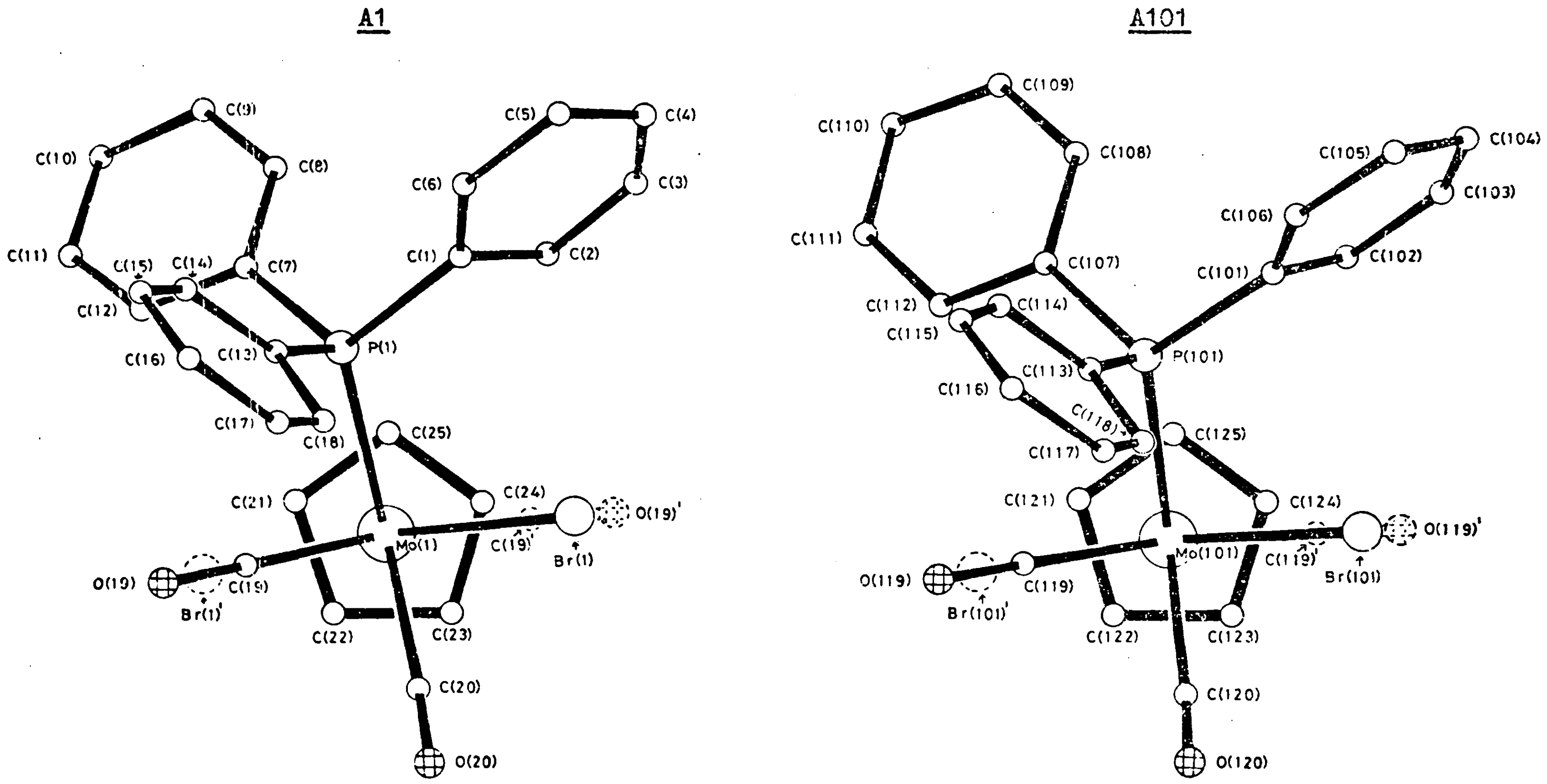

FIGURE 1

The two crystallographically independent molecules of π-cyclopentadienyl-cis-dicarbonylbromo(triphenylphosphine)molybdenum; each projected on to the plane of its π-cyclopentadienyl ring illustrating the halo/carbonyl disorder.

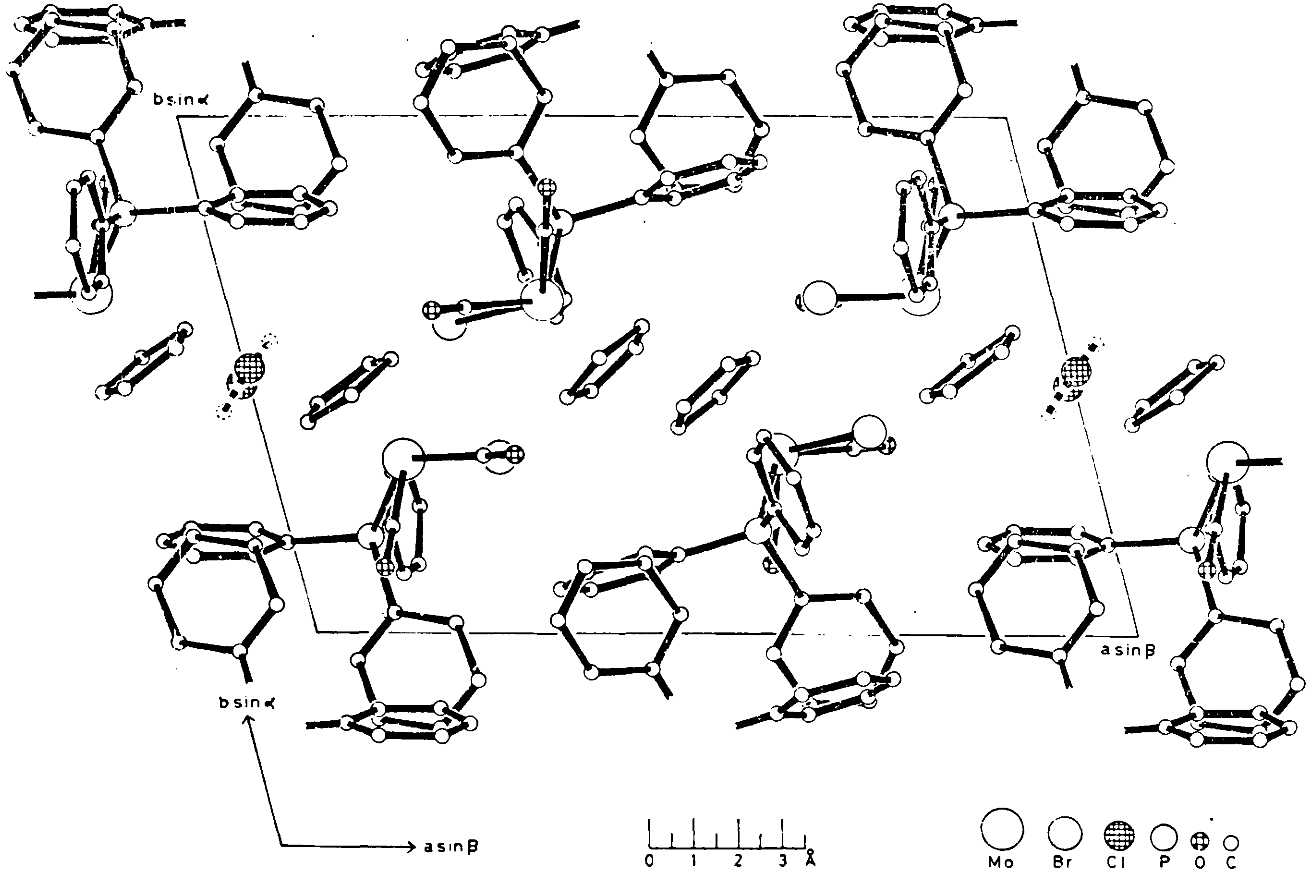

FIGURE 2

Packing arrangement of π-cyclopentadienyl-cis-dicarbonylbromo(triphenylphosphine)molybdenum - methylene chloride viewed along the c-axis. For clarity, only the preferred orientation of A1 and A101 is shown.

TABLE 4

π-Cyclopentadienyl-cis-dicarbonylbromo(triphenylphosphine)molybdenum - methylene chloride; observed and final calculated structure amplitudes (x10)

```
21,-1,L

-4  144   91

21,-2,L

-3  373  356
-4  145  162
-5  219  192

21,-3,L

-2  171  138
-3  196  188
-4  142  183

21,-4,L

-3  151  168

20,1,L

-5  195  214
-6  124   27

20,0,L

-1  228  263
-2  124  101
-3  173  178
-4  279  241
-5  342  339
-6  353  337
-7  181  149

20,-1,L

 0  222  257
-1  240  200
-2  290  262
-5  328  295
-6  228  275

20,-2,L

-1  289  300
-2  183  196
-3  137  124
-4  226  249
-5  217  205
-6  138  151

20,-3,L

-4  147  159
-5  428  433
-8  145   97

20,-4,L

 0  318  299
-1  130  110
-2  157  151
-4  212  206
-5  174  144
-6  351  354

20,-5,L

 0  179  135
-1  131   69
-3  211  181
-4  133  168

20,-6,L

-1  337  283
-2  164  119
-4  201  177

19,3,L

-3  133  187
-4  186  197
-7  177  188

19,2,L

-2  133  103
-3  160  152
-4  162  158
-5  126   18
-7  508  516

19,1,L

 0  192   25
-2  503  508
-4  157  143
-5  377  402
-7  272  227
-8  341  326

19,0,L

 0  378  297
-1  210  221
-2  310  371
-3  130  110
-5  132   90
-6  243  247

19,-1,L

 1  177  166
 0  157   80
-1  307  315
-2  221  233
-3  331  332
-4  214  247
-6  326  335
-7  263  258
-8  150  163

19,-2,L

 2  232  231
 1  201  239
 0  181  228
-2  283  289
-4  373  379
-5  126   18

-7  449  434
-8  301  266

19,-3,L

 2  317  292
 1  316  276
-1  132  113
-3  173  156
-8  158  151

19,-4,L

 2  237  218
-1  262  240
-4  193  284
-5  175  243
-6  157  203
-7  308  318

19,-5,L

 2  448  425
 1  356  329
 0  150  153
-1  293  289
-2  137  163
-4  164  207
-6  183  185

19,-6,L

 1  273  242
-1  138  130
-2  362  336
-3  205  163
-4  212  221
-5  161   94

19,-7,L

-1  344  308
-3  220  212

19,-8,L

-1  224  190
-2  133  159

18,5,L

-3  198  138
-5  232  228

18,4,L

-1  244  252
-2  169  180
-3  164  114
-4  132  170
-6  405  399

18,3,L

 0  172  168
-1  289  299
-3  252  204
-4  193  180
-5  191  163
-6  481  485
-7  270  283
-8  140  131

18,2,L

 1  124   68
-1  319  313
-3  241  258
-5  214  283
-7  145  139

18,1,L

 0  339  350
-2  122   24
-3  314  317
-4  149  131
-5  316  331
-6  374  400
-9  302  350

18,0,L

 2  212  164
 0  384  364
-1  576  557
-2  131   32
-3  154   34
-6  318  310
-9  286  254

18,-1,L

 1  154  150
-1  456  486
-3  151  200
-4  250  229
-5  385  372
-6  323  376
-7  164  121
-8   93  181

18,-2,L

 0  364  340
-3  132  114
-4  170  182
-5  341  345

18,-3,L

 3  302  281
 2  207  283
 1  139  166
 0  545  543
-1  220  219
-4  379  406
-6  432  425
-7  150  159
-8  201  173

18,-4,L

 3  358  343
 2  315  319
 0  286  275
-1  324  327
-3  152  121
-4  315  306
-6  188  181
-7  184  185

18,-5,L

 1  163  172
 0  132   90
-1  302  296
-2  125   71
-3  165  176
-5  351  334

18,-6,L

 3  316  280
 2  508  481
-1  182  167
-3  156  139
-5  311  297
-6  206  205

18,-7,L

 2  195  201
 1  149   64
 0  310  293
-1  319  323
-2  127   91
-4  251  251

18,-8,L

 2  203  126
-3  143  170
-4  211  211

18,-9,L

 0  205  193
-1  172  161

17,6,L

-4  249  277

17,5,L

-2  205  191
-3  209  288
-4  165  156
-5  256  254
-6  193  217
-7  168  166
-8  299  304

17,4,L

 0  238  233
-2  220  205
-3  159  142
-4  320  326
-5  283  269

17,3,L

 1  220  160
-2  232  208
-3  120  101
-5  139   61
-7  200  195
-8  295  292

17,2,L

 2  131  174
 1  428  407
 0  335  327
-2  515  484
-3  255  238
-4  184  125
-5  156  167
-8  347  341
-9  272  259

17,1,L

 0  683  579
-1  162  162
-2  374  369
-3  172  153
-4  233  223
-5  245  201
-6  227  222
-7  124  120
-8  429  431

17,0,L

 3  201  179
 1  255  250
 0  131   80
-1  212  230
-2  344  [illegible]
-4  603  595
-5  181  163
-6  132   44
-8  186  210

17,-1,L

 2  187  134
 1  545  536
 0  237  215
-1  177  150
-2  458  452
-4  273  134
-5  120   90
-6  150  152
-7  253  257
-8  390  391
-9  392  308

17,-2,L

 2  200  202
 1  364  352
 0  338  332
-3  436  430
-6  168  220
-8  329  351
-9  223  231

17,-3,L

 4  188  121
 3  187   26
 2  271  299
 0  228  227
-1  182  136
-4  227  214
-5  240  208
-7  261  288

17,-4,L

 3  124  184
 1  649  637
-1  322  280
-2  331  299
-3  375  375
-4  156  135
-7  136   72
-8  422  443

17,-5,L

 4  151  145
 1  521  534
 0  212  233
-1  248  213
-2  469  469
-3  566  577
-6  152  187
-8  336  318

17,-6,L

 2  244  229
 1  133  146
 0  202  196
-1  122  110
-3  183  202
-4  215  185
-7  170  166

17,-7,L

 2  177  184
 1  243  226
-1  190  207
-2  482  468
-4  148  169
-5  232  219

17,-8,L

 2  127  185
 1  392  397
 0  224  218
-2  266  279
-3  428  421
-5  122   91

17,-9,L

 1  286  197
-3  345  333

16,7,L

-6  300  300

16,6,L

-2  307  297
-3  181  203
-5  200  327
-7  225  239
-8  138   75

16,5,L

-1  212  177
-2  184  172
-3  255  248
-5  148  160
-6  435  430
-8  149   42
-9  203  184

16,4,L

 0  140  160
-1  451  441
-2  193  159
-3  364  350
-5  283  285
-6  224  217
-7  200  204

16,3,L

 2  303  313
 1  231  237
-1  437  443
-2  250  257
-3  277  279
-5  504  504
-8  154  117
-9  145  168
-10 372  339

16,2,L

 3  191  195
 2  183  175
 1  275  304
 0  222  213
-2  170  187
-4  369  372
-5  187  139
-6  187  220
-7  153  160
-8  208  240
-9  231  285
-10 189  160

16,1,L

 3  284  175
 2  209  307
 1  141  125
 0  230  212
-1  598  590
-2  423  411
-4  311  297
-5  219  263
-8  191  156
-10 519  515

16,0,L

 4  198  206
 2  345  336
 1  287  238
-1  543  535
-2  550  556
-3  195  238
-4  321  322
-5  314  351
-8  138  139
-10 216  267

16,-1,L

 4  337  301
 1  202  213
 0  280  276
-2  208  230
-3  331  310
-4  152  147
-5  491  485
-6  131  107
-9  244  303

16,-2,L

 5  287  270
 3  165  173
 1  237  118
 0  586  478
-1  417  412
-2  346  313
-4  625  639
-6  199  210
-7  150   98
-9  341  341

16,-3,L

 4  260  297
 3  201  217
 2  399  388
 0  354  356
-1  398  379
-2  544  524
-3  203  168
-4  250  265
-5  504  583
-7  315  314
-8  221  255

16,-4,L

 3  304  287
 0  324  317
-1  262  269
-2  410  397
-3  193  170
-6  283  238
-8  166   95
-9  198  147

16,-5,L

 5  137   78
 4  151  116
 3  441  454
 2  195  136
 1  287  241
 0  496  486
-1  [illegible]  490
-3  188  177
-4  383  378
-5  202  168
-6  320  204
-8  289  240

16,-6,L

 5  199  222
 2  323  324
 0  341  312
-1  684  674
-2  439  432
-4  306  366
-5  151  124
-6  177  123

16,-7,L

 4  275  280
 2  307  324
 1  118   25
-1  140  165
-2  147  154
-3  143  175
-4  333  330
-5  418  421

16,-8,L

 4  187  145
 3  200  209
 1  126   77
 0  182  187
-1  303  306
-4  208  205

16,-9,L

 3  314  308
 0  323  285
-1  271  282
-2  223  211
-4  283  244
-5  323  333

16,-10,L

 1  277  260
 0  126   44
-2  287  267
-1  178  193
-3  221  197

15,7,L

-1  149  121
-4  168  193
-5  229  275
-8  142  130

15,6,L

 0  414  420
-2  127  159
-3  334  318
-4  400  391
-5  196  220
-6  268  234
-8  164  140
-9  273  297

15,5,L

 0  433  403
-1  284  289
-2  210  258
-4  771  768
-7  182  216
-8  127  166
-9  290  307

15,4,L

 1  251  262
 0  210  203
-1  124  174
-4  270  284
-6  306  338
-9  155   19
-10 223  198

15,3,L

 3  126  122
 1  371  356
 0  360  353
-2  328  300
-3  252  259
-4  248  232
-8  144  174
-9  242  297

15,2,L

 3  130   96
 0  643  619
-1  318  318
-2  396  359
-3  509  506
-4  716  723
-7  214  210
-8  231  194
-9  397  406
-10 165  153

15,1,L

 3  173  145
 1  223  214
 0  474  475
-2  186  165
-4  576  544
-5  342  308
-6  176  194
-7  144  165
-8  361  394

15,0,L

 5  318  313
 3  142  167
 2  226  293
 1  512  491
 0  367  421
-1  207  235
-2  168  132
-3  588  591
-4  152   91
-5  115   78
-6  346  363
-7  290  290
-8  278  286
-9  273  281

15,-1,L

 5  196  192
 4  118   66
 1  207  177
 0  394  374
-1  401  375
-3  758  759
-4  583  592
-5  140  156
-6  311  308
-7  360  357
-8  181  112
-9  181  189
-10 124   89

15,-2,L

 5  433  442
 3  220  236
 2  197  155
 0  184  189
-1  371  382
-2  153  128
-4  743  753
-6  221  181
-7  470  485
-8  235  221

15,-3,L

 5  135   97
 2  164  109
 1  692  671
-3  467  481
-4  121  115
-7  261  223
-8  585  593

15,-4,L

 6  382  351
 5  235  215
 3  183  185
 2  505  494
 1  477  464
 0  442  411
-1  395  349
-2  479  467
-3  647  650
-4  329  333
-5  205  232
-7  462  471
-9  187  150

15,-5,L

 6  260  275
 4  143  187
 2  451  458
 1  214  219
 0  523  526
-1  204  205
-2  320  287
-3  598  603
-4  397  388
-5  154  206
-6  297  269
-7  281  282
-9  169  191

15,-6,L

 5  282  289
 2  132   58
 3  235  229
 1  241  236
 0  256  270
-1  113   22
-2  520  498
-3  332  311
-4  303  309
-5  191  213
-8  228  200

15,-7,L

 1  503  487
 0  322  326
-2  149  120
-3  611  612
-4  225  249
-5  249  231
-6  344  326
-7  137   29

15,-8,L

 5  330  337
 4  165   69
 3  179   90
 2  230  218
 1  211  114
 0  183   97
-1  357  344
-3  370  385
-4  344  374

15,-9,L

 4  177   24
 2  309  369
 1  247  210
-1  157   56
-2  131  162
-3  114    5

15,-10,L

 3  184  177
 1  311  315
-2  380  387
-3  351  334
-4  209  243

15,-11,L

 2  231  242
 0  296  295

14,8,L

-2  282  285
-5  320  367

14,7,L

-1  161  163
-2  291  306
-3  359  372
-5  342  371
-6  217  217
-7  181  184
-8  143  140

14,6,L

-1  166  150
-3  569  558
-4  207  206
-6  245  247

14,5,L

 2  201  186
 1  167   84
 0  172   80
-1  339  319
-2  393  350
-5  459  479
-7  252  255
-10 189  203

14,4,L

 1  161   97
 0  166  115
-1  434  421
-2  359  367
-3  735  736
-4  117  122
-5  380  381
-6  371  372
-7  215  248
-8  158  119
-9  316  339
-10 242  211

14,3,L

 3  197  203
 2  385  391
 1  445  431
 0  145  189
-2  455  466
-3  429  424
-4  316  313
-6  387  376
-7  593  571
-8  216  225
-9  195  185
-11 133   84

14,2,L

 4  152   65
 3  122   76
 2  467  487
-1  131  150
-2  536  512
-3  131  121
-5  144   35
-6  238  233
-8  232  227
-10 307  324
-11 241  210

14,1,L

 4  311  329
 3  238  225
 2  120   35
 1  325  299
 0  232  197
-1  362  364
-2  900  885
-4  222  218
-5  633  620
-6  484  459
-8  141  120
-9  178  148
-11 181  222

14,0,L

 4  285  278
 1  271  270
 0  148  183
-2  417  430
-3  768  795
-4  151  192
-5  643  651
-6  314  305
-8  137   66
-9  192  190
-10 183  182

14,-1,L

 6  242  278
 5  186  229
 3  176  140
 2  379  354
 1  245  267
 0  191  192
-1  133  166
-2  478  474
-3  643  655
-4  510  512
-5  484  482
-7  329  365
-9  167  162

14,-2,L

 5  173  179
 4  266  274
 3  203  211
 2  418  408
 1  201  260
-1  115  107
-2  773  742
-3  528  501
-4  373  373
-5  384  441
-6  481  492
-8  381  401
-9  179  154
-10 198  203

14,-3,L

 5  121  112
 4  120  130
 3  557  552
 1  161  137
 0  174  150
-1  147  150
-2  551  551
-3  384  385
-4  158  120
-5  708  806
-7  327  353
-8  235  230
-10 125   70

14,-4,L

 3  389  400
 2  284  252
 1  341  346
 0  120   92
-1  475  450
-3  278  185
-5  167  190
-6  129   77
-7  186  172
-4  274  277

14,-5,L

 6  147  187
 5  129   74
 4  257  261
 3  234  288
 2  253  214
 1  137   45
-1  652  651
-2  790  786
-3  129  115
-4  384  273
-5  511  514
-6  394  388
-8  153  182
-9  170   13

14,-6,L

 5  177  220
 4  311  361
 3  355  352
 2  134   92
 0  145  135
-1  332  350
-2  824  811
-3  287  277
-4  292  282
-5  677  657
-6  457  458

14,-7,L

 3  458  448
 2  118   17
 1  130   89
 0  233  273
-1  424  409
-2  300  318
-3  246  220
-4  203  187
-5  218  196
-6  298  262
-8  282  183

14,-8,L

 5  201  167
 4  341  337
 3  257  222
 2  237  226
-1  362  355
-2  392  400
-4  235  228
-5  397  403

14,-9,L

 4  364  347
 3  239  183
 2  191  142
-2  346  346
-3  216  227
-4  192  173
-5  212  210

14,-10,L

 3  479  463
 0  262  268
-2  149  136

14,-11,L

 1  222  231
 0  155  168
-1  482  484

13,9,L

-5  293  217

13,8,L

-1  224  173
-3  158  118
-4  182  189
-5  376  306
-6  223  202

13,7,L

 0  308  413
-1  185  172
-3  127   87
-4  420  407
-5  152  184
-9  311  354

13,6,L

 1  293  258
 0  147  188
-1  130  130
-2  255  242
-4  914  926
-5  120   71
-6  190  217
-7  182  233
-8  410  445

13,5,L

 1  432  437
-2  597  604
-4  320  322
-5  513  508
-6  170  164
-7  145   68
-8  640  682

13,4,L

 3  284  275
 2  169  190
 1  287  215
 0  154  114
-2  111  102
-3  220  [illegible]
-4  482  387
-5  316  308
-6  317  306
-7  218  233
-8  168  140
-9  205  249
-10 172  120

13,3,L

 4  296  274
 2  246  263
 1  291  272
 0  753  750
-1  383  370
-3  295  310
-4  526  536
-5  361  347
-6  423  430
-7  319  292
-8  398  308
-9  309  295
-10 211  236

13,2,L

 5  332  331
 4  300  299
 3  452  448
 1  400  449
 0  166   87
-1  465  457
-2  147   39
-3  211  137
-4  961  965
-5  493  500
-6  527  554
-8  408  435

13,1,L

 5  319  346
 4  203  193
 2  521  521
 1  336  327
 0  461  454
-1  332  326
-2  283  292
-3  552  560
-4  242  263
-7  231  214
-8  239  217
-9  214  220
-10 161   99
-11 188  204

13,0,L

 6  331  324
 5  581  482
 4  131   65
 3  252  266
 2  168  109
-1  218  160
-2  414  408
-3  734  737
-4 1090 1117
-5  439  436
-7  421  403
-8  196  160
-9  127  110
-11 102  154

13,-1,L

 6  188  217
 5  419  453
 4  433  446
 3  108  222
 2  297  286
 1  296  283
 0  283  214
-1  460  451
-2  271  274
-3  303  327
-4  627  620
-5  458  458
-6  177  142
-7  514  541
-8  593  603
-9  184  182

13,-2,L

 6  186  155
 4  424  424
 2  144  192
 1  510  486
-1  210  248
-2  238  243
-3  160  155
-5  394  306
-6  210  186
-7  396  397
-8  507  523
-9   81   60
-10 140  115

13,-3,L

 6  466  440
 5  201  250
 4  134  141
 3  172  120
 2  649  632
 0  434  402
-1  471  442
-3  537  554
-4  341  318
-5  243  240
-7  772  777

13,-4,L

 7  155  189
 6  344  340
 5  340  176
 4  470  400
 3  133  145
 2  658  682
 1  547  529
 0  113  106
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13,-4,L

-1  460  449
-2  299  302
-3  496  511
-4  535  537
-5  688  674
-6  419  411
-7  259  245
-8  417  432
-9  148  114

13,-5,L

 7  170  146
 5  211  227
 4  483  438
 2  141  122
 1  276  246
 0  571  864
-1  389  398
-2  252  270
-3  332  342
-4  437  439
-5  421  425
-6  236  216
-7  147  118
-8  328  324
-9  240  231

13,-6,L

 6  218  248
 5  126  163
 2  234  199
 0  446  441
-2  224  216
-3  376  373
-4  538  533
-5  124   97
-6  329  285
-7  213  183

13,-7,L

 6  383  378
 5  545  534
 2  425  407
-1  115  112
-3  446  456
-4  586  614
-7  515  520

13,-8,L

 6  134   20
 5  318  289
 4  433  436
 2  257  271
 1  484  518
-1  386  360
-2  285  215
-3  178  136
-4  125   39
-6  338  324
-7  170  203

13,-9,L

 5  245  224
 4  277  305
 3  219  223
 1  290  288
 0  231  217
-1  131   32
-2  249  229
-3  301  304
-4  272  243
-5  288  181

13,-10,L

 5  372  375
 4  140   45
 2  444  440
 1  394  397
 0  347  362
-3  302  287
-4  284  248

13,-11,L

 2  101  201
 1  359  347
-2  232  189

12,[illegible],L

-2  261  221
-3  165   44
-4  207  290
-5  164  154
-6  137  149
-7  177  190

12,8,L

-3  432  418
-5  318  321
-6  170  120
-7  400  377
-8  167  160

12,7,L

 1  128   20
-1  145   32
-2  225  229
-3  652  659
-4  425  418
-6  100  100
-7  399  422
-8  211  193
-9  192  188

12,6,L

 2  377  383
 1  204  145
-2  316  305
-3  167  200
-4  240  197

-5  242  275
-6  126   39
-7  371  376
-8  252  225

12,5,L

 3  263  264
 2  223  238
 0  258  221
-1  144   20
-2  381  388
-3  917  944
-6  736  741
-7  539  548
-8  143  112
-9  386  381
-10  161   32

12,4,L

 4  223  186
 3  257  261
 2  784  691
 1  192  207
-3  328  361
-4  167  150
-5  355  369
-6  292  301
-7 1055 1069
-9  203  176
-10  267  201
-11  181  184

12,3,L

 3  157  185
 2  591  596
 0  136  128
-2  637  635
-3  578  598
-4  170  200
-5  186  225
-6  304  279
-7  149  159
-8  178  188
-11  459  417

12,2,L

 5  138  138
 3  414  431
 2  243  243
 1  224  186
-1  282  270
-2  823  920
-3  136  138
-5  335  343
-6  687  687
-7  274  268
-8  173  178
-9  137   89
-10  315  329
-11  173  180

12,1,L

 6  400  470
 5  219  239
 4  219  224
 3  649  608
 2  347  331
 0  264  273
-1  366  357
-2  151  184
-3 1199 1185
-4  579  575
-6  772  778
-7  533  545
-8  137   96
-9  308  328
-10  304  333

12,0,L

 6  357  383
 5  209  308
 4  178  121
 3  112   86
 2  762  752
 1  210  156
 0  181  134
-2  681  702
-3  822  532
-4  219  211
-5  163  119
-6  117  160
-7  837  884
-9  228  211
-11  271  249

12,-1,L

 7  454  469
 6  162  159
 4  128   78
 3  415  415
 2  437  414
 1  313  312
 0  100  127
-1  215  234
-2  887  880
-3  466  420
-5  161  137
-6  837  840
-8  313  274
-9  224  128
-11  222  207

12,-2,L

 7  384  368
 6  157  171
 5  373  300
 4  176  190
 3  710  707
 2  448  437
 0  232  159
-1  279  253
-2  567  548
-3  231  210
-6 1002  998

-7  662  679
-9  197  233
-10  275  264

12,-3,L

 6  224  214
 5  395  378
 4  285  291
 3  492  513
 2  585  579
 1  139   12
-1  530  525
-2  252  253
-3  391  371
-4  370  372
-5  119   36
-6  163  158
-7  354  368
-9  168  121
-10  321  321

12,-4,L

 7  349  341
 5  127  163
 3  418  417
 1  239  226
 0  362  316
-1  317  332
-2  669  655
-5  184  289
-6  457  459
-7  122  162
-8  159  125
-10  170  151

12,-5,L

 7  317  330
 6  283  275
 5  227  276
 3  790  858
 2  262  263
 1  133  142
 0  322  323
-1  666  664
-2  744  748
-3  324  312
-4  211  225
-5  401  440
-6  823  829
-7  301  308
-8  129   76
-9  279  238

12,-6,L

 7  287  250
 6  370  377
 4  186   73
 3  388  473
 2  157  184
 1  391  404
-1  518  527
-2  167  149
-3  382  401
-4  309  324
-5  328  362
-6  492  486
-7  404  414
-8  215  239

12,-7,L

 5  215  165
 4  759  752
 2  345  343
 1  150  147
 0  171  170
-2  211  160
-3  156   92
-5  618  627

12,-8,L

 7  277  254
 6  348  328
 5  189  210
 4  343  330
 3  500  503
 2  303  312
 1  123   87
 0  188  174
-1  158   62
-2  227  222
-3  182  202
-5  170  165
-6  494  480
-7  203  203

12,-9,L

 5  193  170
 3  408  517
 2  453  438
 1  191  180
 0  268  270
-1  456  502
-2  147   94
-3  148  156
-6  365  303

12,-10,L

 5  132  110
 4  227  182
 3  214  245
 2  377  368
-1  460  478
-2  126  120

12,-11,L

 4  207  206
 3  366  341
 1  201  310
 0  307  321
-1  184   49
-2  246  274

12,-12,L

 0  234  257
-1  294  264

11,10,L

-4  239  225
-5  249  254
-6  338  347

11,9,L

-2  195  227
-5  278  294
-6  300  327
-7  166  115

11,8,L

 0  236  228
-1  279  303
-2  218  184
-3  162  152
-4  354  357
-9  210  189

11,7,L

 1  255  262
 0  137   85
-2  241  243
-4  530  546
-6  251  255
-8  487  433
-9  152  181
-10  198  168

11,6,L

 3  256  261
 1  535  525
 0  228  225
-2  476  474
-3  211  204
-5  329  357
-6  684  712
-8  496  523
-9  187  207
-10  257  234

11,5,L

 3  590  606
 2  157  122
 1  120  133
 0  206  203
-1  332  339
-2  208  170
-6  742  742
-7  125  117
-9  403  436
-10  212  188

11,4,L

 5  184  150
 4  156  223
 3  123    7
 2  325  307
 1  310  304
 0  280  251
-1  455  452
-4  176  140
-5  456  457
-6  240  216
-8  483  504
-9  122   77
-10  258  274

11,3,L

 5  228  251
 4  513  542
 3  477  484
 2  140   97
 1  654  676
 0  606  599
-1  114   87
-3  309  274
-4  504  592
-5  736  703
-6  718  719
-8  538  549
-9  511  523
-10  164  138

11,2,L

 6  212  174
 5  183  184
 4  323  302
 3  404  477
 2  116   50
 1  289  246
 0  523  519
-1  748  767
-2  770  771
-3  342  356
-4  213  232
-6  382  389
-8  920  943
-9  272  262
-10  486  481

11,1,L

 5  140  131
 4  307  392
 3  101  205
 2  117  135
 0  156  140
-1  325  350
-2  715  710
-4  610  606
-5  501  572
-6  450  442
-7  231  240
-8  143  130
-9  218  243
-11  250  237

11,0,L

 7  248  220
 6  146  173
 5  373  374
 4  690  692
 3  176  193
 2  392  387
 1  646  638
 0  473  503
-1  148  189
-4  267  273
-5  883  871
-7  523  495
-8  537  556
-9  483  463

11,-1,L

 6  147   53
 4  882  818
 3  458  448
 2  455  460
 1  455  434
 0  855  886
-1  279  264
-2  347  335
-3  336  307
-4  179  174
-5  635  628
-6  729  745
-8  392  483
-9  439  420
-10  159  174

11,-2,L

 6  168  170
 5  541  534
 4  167  125
 3  646  663
 2  331  303
 1  115   68
 0  813  805
-1  584  481
-2  101   45
-3  178  172
-5  286  210
-6  281  284
-7  358  368
-8  164  188
-9  137   39
-10  269  258

11,-3,L

 8  238  266
 7  139  125
 6  151  164
 5  181  136
 4  715  744
 2  816  814
 1  664  683
 0  452  409
-1  347  363
-2  394  375
-4  252  220
-5  672  671
-6  298  292
-7  146   81
-8  545  550
-9  428  432

11,-4,L

 6  249  241
 4  627  541
 3  278  314
 1  266  263
 0  935  945
-1   95   62
-2  396  373
-5  639  654
-6  151  141
-7  148  140
-8  278  285
-9  429  415

11,-5,L

 6  142  167
 4  388  376
 3  131   73
 1  389  387
 0  604  594
-1  211  285
-2  431  448
-4  710  708
-9  174  188

11,-6,L

 8  147  118
 7   87    6
 6  184  144
 5  749  760
 4  288  299
 1  207  178
 0  379  347
-2  188  187
-3  218  211
-4  931  935
-5  494  451
-7  463  454
-8  495  468
-9  284  254

11,-7,L

 7  182  123
 6  198  211
 5  207  386
 4  587  562
 2  699  715
 1  770  786
 0  344  334
-1  317  287
-2  186  164
-3  212  185
-4  198  172
-5  456  403
-6  111  113

11,-7,L

-7  271  274
-8  411  418

11,-8,L

 7  141  122
 5  404  446
 4  280  281
 3  345  350
 2  122  141
 0  385  396
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-3  226  286
-4  286  274
-5  275  273

11,-9,L

 6  145  184
 5  411  486
 4  143  113
 2  199  198
 1  884  884
 0  213  183
-2  144  159
-4  247  263
-5  218  195

11,-10,L

 6  147   48
 5  258  235
 4  392  385
 3  262  273
 2   99   18
 1  518  507
 0  261  267
-2  281  248
-3  222  228
-8  279  295

11,-11,L

 4  284  271
 2  241  184
 1  199  221
 0  537  568
-3  266  216

10,10,L

-2  237  218
-4  213  220

10,9,L

-2  257  234
-3  400  489
-5  181  190
-7  269  284
-8  127  122

10,8,L

 2  493  491
 1  183  219
 0  163  192
-1  151   55
-2  384  380
-3  379  374
-4  276  267
-7  539  568
-8  269  267
-9  297  286
-10  159   53

10,7,L

 2  418  429
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 0  223  255
-2  295  282
-3  261  247
-5  178  173
-7  298  320
-8  472  474

10,6,L

 4  249  264
 2  220  223
 0  192  136
-1  137  181
-2  166  166
-3  493  489
-4  325  284
-5  365  361
-6  485  411
-7  610  649
-8  238  248
-9  129  151
-11  183   79

10,5,L

 6  175  173
 3  276  285
 2  760  718
 1  161  165
-1  276  285
-2  528  530
-3  143  148
-4  127  188
-5  122   88
-6  210  193
-7 1020 1018
-8  294  298
-9  144  160
-10  254  235
-11  424  433

10,4,L

 5  221  149
 4  287  281
 3  142   53
 2  747  758
 1  337  358
 0  279  298
-1  188  138

-2  881  784
-3  245  232
-4  151  136
-5  218  234
-6  367  372
-7  407  417
-8  418  421
-9  242  248
-11  514  557

10,3,L

 4  198  187
 3  513  507
 2  184  189
 1  272  269
 0  539  540
-1  497  508
-2  654  674
-3  232  206
-4  315  388
-6  613  597
-7  148  114
-8  181  178
-9  229  268
-10  330  351

10,2,L

 6  237  179
 5  339  337
 4  238  167
 3  458  431
 2  784  787
 1  580  597
-1  735  727
-2  415  488
-3  638  604
-4  497  498
-5   97   61
-6  714  782
-7  343  438
-8  325  332
-9  191  184
-10  341  365
-11  397  393

10,1,L

 7  186  192
 6  339  352
 5  681  596
 3  211  214
 2  789  797
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 0  318  319
-1  427  444
-2 1367 1307
-4  283  273
-5  142  186
-6  121   76
-7  977 1018
-8  641  654
-9  197  183
-11  430  462

10,0,L

 7  261  230
 6  225  228
 4  165  169
 3  375  384
 2  621  593
 1  215  181
-1  348  352
-2  464  487
-3  351  389
-4  444  431
-5  438  414
-6  769  788
-8  185  206
-10  191  149
-11  276  314

10,-1,L

 7  185  142
 6  253  269
 5  262  289
 4  458  460
 3 1080 1072
 2  788  779
 1  175  158
 0  146  119
-1  648  655
-2  227  185
-4  182   69
-5  552  563
-6  860  889
-7  785  792
-8  146  133
-9  270  268
-10  462  457

10,-2,L

 8  284  243
 7  131  135
 5  817  547
 3  726  722
 2  935  986
 1  482  480
-1  823  763
-2  379  370
-3   97  184
-4  651  682
-5  159  171
-6  284  231
-7  414  434
-8  426  449
-10  485  493
-11  386  374

10,-3,L

 8  245  211
 5  265  324
 3  141  133
 2  169  147
 1 1184 1292
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-2  709  623
-4  621  591
-5   99   92
-6  227  199
-8  392  402

10,-4,L

 7  163  138
 4  252  342
 3  494  651
 2  551  537
 1  111   75
 0  157  155
-1  978 1011
-2  164  171
-3  298  324
-4  138  128
-5  180  178
-6  634  632
-7  144  171
-10  486  420

10,-5,L

 8  464  469
 6  432  548
 5  317  411
 2  482  529
 1  843  860
 0  149  118
-1 1061 1074
-2  482  508
-3  127   98
-4  248  243
-5  364  385
-6  366  362
-7  412  415
-8  496  511
-9  181  156

10,-6,L

 7  282  230
 5  341  476
 4  383  555
 2  419  418
 1  442  484
-2  273  255
-3  284  292
-4  214  194
-5  393  387
-6  114  133
-7  147  113
-8  275  271

10,-7,L

 7  149  128
 6  495  569
 5  249  261
 3  358  387
 2  273  292
 0  655  676
-2  154  181
-3  255  299
-4  387  311
-5  165  172
-6  467  481
-7  318  388

10,-8,L

 6  142  161
 5  132  138
 3  688  679
 2  809  828
 1  125  142
 0  175  197
-1  376  374
-2  334  343
-6  258  288
-7  399  398

10,-9,L

 7  195  192
 5  227  184
 2  281  268
 1  493  885
-1  323  358
-2  464  474
-4  235  188
-5  183  121

10,-10,L

 7  257  252
 6  153  175
 5  160  120
 4  173  193
 3  438  412
 2  334  314
 1  330  337
 0  186  218
-1  219  235
-3  120  148
-5  141  157

10,-11,L

 6  292  277
 4  181  170
 3  226  256
 2  329  334
 0  397  377
-1  516  473
-3  243  230
-4  190  227

10,-12,L

 4  241  232
 2  362  391
 1  272  277
 0  127   63
-1  178  158
-2  274  290

9,10,L

-1  327  344

-3  163  133
-5  144  127
-6  429  418

9,9,L

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-1  466  485
-3  264  247
-4  355  368
-5  252  254
-6  718  726
-8  157  135
-9  259  212

9,8,L

 2  146  157
-2  277  253
-3  218  204
-5  336  343
-7  264  257
-8  233  228
-9  131  135
-10  218  181

9,7,L

 4  141  178
 3  274  288
 2  121   29
 1  341  348
 0  427  415
-1  369  434
-2  322  311
-3  242  262
-4  315  293
-6  696  699
-7  382  251
-8  267  261
-9  181  176
-10  381  488

9,6,L

 3  793  812
 2  197  228
 1  281  212
 0  228  220
-1  777  785
-2  172  108
-3  379  369
-5  153  128
-6  811  823
-7  150   98
-8  127  108
-9  166  179
-10  434  466

9,5,L

 4  267  247
 3  366  383
 2  247  228
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 0  187  135
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-2  288  214
-3  121   64
-5  815  859
-7  330  320
-9  283  226
-10  316  389
-11  178  181

9,4,L

 6  136   98
 4  543  535
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 0  718  715
-1  158  188
-3  381  355
-4  134   46
-5  438  451
-6  533  554
-8  188  160
-9  796  792
-10  284  381

9,3,L

 3  913  814
 2  277  298
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 0 1158 1141
-1 1317 1309
-2  317  277
-3  248  267
-4  268  259
-5  487  489
-6  425  434
-8  223  218
-9  425  425
-10  841  887

9,2,L

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 4  285  287
 3  260  245
 2  123   78
 1  281  183
 0  258  262
-1  128  222
-2  259  257
-3  115   78
-4  482  428
-5  703  808
-8  563  586
-9  239  215
-10  314  325
-11  141   94

9,1,L

 5  386  286
 4  769  767
 3  423  428
 1  133  141

 0 1498 1454
-1  443  431
-2  489  487
-3  283  185
-4 1034  968
-5  989  949
-6  151  140
-8  414  429
-9  623  615
-10  141   56

9,0,L

 8  125  160
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 3  980  894
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-2  250  233
-4 1121 1180
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-6  850  868
-7  286  283
-8  136  132
-9  625  648
-10  428  411
-11  188  246

9,-1,L

 8  188  225
 7  232  217
 5  316  323
 4  256  213
 3  822  836
 2  502  515
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-1 1218 1191
-2  528  478
-4  848  588
-5  214  243
-6  578  576
-9  157  168
-10  488  465
-11  134  132

9,-2,L

 8  152  152
 7  128   25
 6  245  238
 4  673  718
 3  389  372
 2  428  458
 1  488  482
 0  818  801
-1  733  737
-2  442  429
-3  415  397
-5  738  729
-6  145   95
-7  154   81
-8  272  261
-9  855  584

9,-3,L

 8  465  488
 6   86  140
 4  471  550
 3  311  342
 2  181  167
 1  114   66
 0 2118 2171
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-2  275  291
-3  458  431
-4  858  558
-5  381  384
-6  138  132
-7  279  270
-9  828  527
-10  348  361

9,-4,L

 9  481  447
 8  299  297
 6  130  172
 5  184  231
 4  231  248
 3  341  455
 2  392  418
 1  588  588
 0 1079 1079
-1  748  743
-3  182  138
-4  755  761
-7  426  444
-8  281  187
-9  288  268
-10  333  318

9,-5,L

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 7  327  132
 6  238  225
 5  558  688
 4  143  227
 3  228  317
 2  189  225
 1  543  490
 0  174  142
-2  385  487
-3  286  275
-4  249  224
-5  382  373
-6  133  111
-7  134   82
-8  378  378
-9  234  272

9,-6,L

 9  382  353
 7  431  416
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9,-6,L

6  151  168
4  526  551
3  267  286
2  306  283
1 1002 1035
0  814  822
-1  122   55
-2  332  308
-3  529  493
-4  675  677
-5  311  321
-6  201  194
-7  124  128
-8  301  324
-9  371  365

9,-7,L

8  207  223
6  145  122
5  710  712
4  336  311
3  595  587
2  122   89
1  168  202
0  563  590
-1  442  461
-2  295  290
-3  178  136
-4  652  657
-6  296  298
-7  145   88

9,-8,L

8  147  180
6  261  247
5  198  165
4  314  326
3  358  370
2   85   65
1  567  571
0  359  382
-1  257  244
-3  417  435
-4  151  133
-5  291  280

9,-9,L

8  189  173
5  291  180
4  263  225
3  373  352
1  452  424
0  483  476
-1  271  274
-2  169  192
-3  561  592
-5  265  257
-6  224  186

9,-10,L

7  152  144
6  272  241
5  176   54
4  131  128
3  336  353
1  147  127
0  721  705
-1  316  319
-2  158  130
-3  410  393
-4  324  288

9,-11,L

5  383  384
4  199  195
3  299  270
1  226  257
0  173  130
-1  191  195
-4  311  325

9,-12,L

4  158  187
1  364  369
0  363  387

9,-13,L

1  327  294

8,11,L

-2  172  154
-3  250  266
-7  235  177

8,10,L

0  292  292
-1   89  139
-2  226  228
-3  380  366
-4  148   78
-7  290  303

8,9,L

2  252  242
1  341  346
0  246  276
-1  181  192
-2  417  429
-4  284  281
-6  285  257
-7  436  418
-8  155  131
-9  314  303

8,8,L

2  419  350
1  373  330
0  500  570
-2  356  346

-3  474  498
-4  322  325
-5  214  198
-7  261  261
-8  339  346
-9  301  284

8,7,L

4  231  238
0  331  314
-2  169  153
-3  395  390
-4  518  507
-5  285  252
-6  112  108
-7  382  385
-9  124  118
-11  155   62

8,6,L

5  158  114
2  713  725
1  359  366
0  218  225
-1  181  188
-2  537  529
-3  343  382
-5  192  184
-6  133  129
-7  411  411
-9  463  508
-11  514  515

8,5,L

5  182  174
4  498  505
3  154  143
2  374  373
1  483  524
0  589  605
-1  378  368
-2  728  739
-3  177  153
-4  531  523
-5  291  318
-6  538  547
-8  427  434
-9  385  375
-11  588  597

8,4,L

4  321  291
3  137  153
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-1  156  141
-2  688  675
-3  166  187
-4  333  307
-5  585  573
-6  529  524
-7  189  168
-9  518  516
-10  144  140

8,3,L

7  142  115
5  312  279
4  115   80
3  291  290
2  147  154
1  512  522
0  362  338
-1  211  182
-2  572  540
-3   94   68
-4  819  823
-5  485  445
-6  284  266
-8  345  338
-11  341  332

8,2,L

8  170  153
7  285  179
5  263  285
4  385  386
3  219  215
2  236  265
1 1358 1426
0  923  924
-2 1145 1152
-3 1097 1104
-4  173  121
-5  251  233
-7  442  404
-8  640  657
-9  338  332
-11  334  380

8,1,L

7  154  144
6  568  552
4  351  368
3  181  167
2  241  204
1  177  166
0  525  541
-1  272  221
-2  177  220
-3 1155 1138
-4 1062 1046
-5  137   69
-6  717  715
-7  141   89
-8  234  213
-9  252  240
-10  218  150
-11  242  225

8,0,L

7  162  144
5  388  396
4  574  584

3  306  294
2  728  787
1  348  340
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-1  731  745
-2  562  573
-3  533  529
-4  297  210
-5  184  218
-6  343  349
-7  509  536
-8  381  370
-9  131  162
-10  269  238
-11  251  248

8,-1,L

9  169  129
8  276  243
7  285  215
6  569  555
5  371  392
4  243  218
3  282  174
2 1303 1326
1 1025 1011
0  912  874
-1 1138 1044
-2  959  918
-3  842  840
-4  342  314
-5  389  371
-6  219  224
-7  183   61
-8  654  644
-10  433  493
-11  543  534

8,-2,L

9  167  123
8  236  247
6  174  172
4  215  182
2  421  402
1 1949 1925
0 1476 1338
-1  734  725
-2  342  345
-3  922  895
-5  191  167
-6  133   86
-7  406  412
-8  736  723
-9  182  185
-11  316  315

8,-3,L

9  227  215
7  243  268
6  402  430
5  118  131
4  158  145
3  258  277
2  476  573
1  253  262
-1  473  465
-2  152  115
-3  441  487
-4  372  370
-5  148  108
-6  206  277
-9  225  199
-10  198  287

8,-4,L

8  286  282
7  568  557
6  221  216
5  288  312
3  194  245
2  238  380
1 1133 1155
0  583  582
-1  708  740
-2 1331 1375
-3  735  758
-4  297  273
-5  445  452
-8  534  522
-10  188  184

8,-5,L

8  147  128
7  174  178
6  565  585
5  192  188
4  247  311
2  648  646
1 1237 1046
0  117   99
-1  113  115
-2  837  841
-3 1187 1236
-4  168  121
-5  495  506
-6  331  324
-8  368  367

8,-6,L

6  742  742
5  185  179
2  311  353
1  665  651
0  122   98
-1  234  221
-2  109   97
-3  555  591
-4  146   96
-5  214  208
-6  278  289
-7  331  317

8,-7,L

7  257  250
3  186  120

2  971  984
-1  119  132
-2  648  635
-3  117   96
-4  128   62
-5  192  157
-6  123  117
-7  389  333
-8  152   78

8,-8,L

8  298  257
7  493  484
6  249  214
5  240  243
4  164  176
3  223  222
2  194  178
1  538  528
0  185  170
-1  489  512
-2  533  548
-3  349  355
-4  169  181
-6  214  221

8,-9,L

7  232  202
6  278  294
5  246  268
4  164  155
3  264  223
2  229  269
1  495  511
0  114    5
-1  365  380
-3  362  362
-5  182  139

8,-10,L

7  281  273
6  329  327
3  125   58
2  353  361
1  289  213
0  286  277
-1  408  368
-2  469  447
-3  184  185

8,-11,L

7  337  344
6  168  166
4  181  147
3  176  179
2  373  386
1  373  335
0  180  161
-1  136   60
-2  585  578
-3  188  183
-4   84   32

8,-12,L

6  219  255
3  244  252
-2  197  165

8,-13,L

3  256  258
2  217  238

7,11,L

-1  451  441
-5  194  149
-6  294  294

7,10,L

1  291  259
0  135  136
-1  577  571
-2  190  238
-4  270  267
-5  390  378
-7  152   31

7,9,L

2  182  145
1  210  238
-2  440  453
-5  567  516
-6  180  175
-7  221  197
-8  130   67
-9  222  227
-10   85  183
-11  324  292

7,8,L

2  122  180
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0  296  198
-1  242  249
-2  248  281
-4  322  324
-5  318  311
-6  556  559
-8  133   10
-9  183  169
-10  361  357
-11  174  186

7,7,L

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3  735  730
2  264  324
1  173  118
0  182  127
-1  749  748
-2  437  431
-3  422  420

-5  619  610
-6  587  559
-7  147   63
-8  138   72
-10  395  378
-12  256  227

7,6,L

7  269  233
5  175  141
4  376  358
3  176  172
2  288  264
1  381  298
-1  986  925
-2  458  462
-3  587  558
-5  811  806
-6  251  277
-7  291  274
-8  270  242
-9  261  262
-10  333  313
-11  262  269

7,5,L

4  199  236
3  482  491
1  411  429
0  719  738
-2  175  173
-4  233  228
-5  512  507
-6  381  363
-8  174  164
-9  564  568
-10  387  278
-11  162   82

7,4,L

8  354  373
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2  368  376
1  181   28
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-1 1405 1429
-2  195  218
-3  335  320
-4  896  865
-5  459  475
-6  123  123
-9  282  183
-10  677  689
-11  234  261

7,3,L

8  380  348
7  168  175
6  124   11
5  249  226
4  408  421
3  178  181
2  418  423
1  508  685
0  283  225
-1  717  724
-2  740  738
-3  181  168
-4  455  417
-5 1019 1017
-6   95   51
-7  120  118
-8  120  141
-9  386  367
-10  359  371
-11  239  236

7,2,L

9  186  227
8  183  171
7  358  350
5  278  196
4  489  521
3  470  459
2  175  161
1  122  140
0  916  914
-1  241  210
-2  648  612
-3  149  143
-4 [illegible]  424
-5  329  343
-6  193  163
-8  241  248
-9  538  530
-10  167  155
-11  178  193
-12  182  138

7,1,L

8  244  237
7  133  127
6  283  276
5  578  596
3  876  884
2  676  682
1  263  280
0 1243 1188
-1  283  280
-2  884  778
-3  123   98
-4 1484 1419
-5  484  472
-6  884  893
-7  421  441
-8  471  465
-9  370  357
-10  281  281

7,0,L

9  179  150
8  480  376
7  782  772
5  488  484

7,0,L

4  528  521
3  451  472
2  787  757
1  624  551
0 1694 1597
-1  895  845
-2 1446 1429
-3  497  469
-4  754  711
-5  953  986
-6  185  211
-8  427  410
-9  114   94
-10  402  478
-11  312  330

7,-1,L

8  218  188
7  198  156
6  195  194
5  132  110
4  714  727
3  562  606
2  165  135
0  795  726
-1  834  783
-2  728  734
-3  147  124
-4  556  568
-5  734  738
-6  584  602
-7  231  217
-8  183  193
-9  686  592
-10  217  200

7,-2,L

9  369  371
8  260  190
7  160   80
6  194  177
5  396  423
3   94   86
2  244  247
1  466  415
0 1790 1722
-1 1181 1159
-2  484  446
-3  171  170
-4 1115 1111
-5  179  149
-6  226  195
-7  168  156
-8  296  247
-9  486  473
-10  432  442
-11  142  132

7,-3,L

9  376  377
8  505  518
7  181  167
6  115  116
5  736  736
3  191  170
2  363  421
1   97  122
0  317  287
-1  914  924
-2  619  636
-3  453  423
-4 1441 1458
-5  266  249
-6  261  242
-7  338  322
-9  135   55
-10  323  337

7,-4,L

9  143  130
8  207  187
7  708  716
5  464  450
4  170  156
3  187  141
2  887  891
1  167   94
0  663  636
-1  118   77
-2 1257 1256
-3  776  748
-4  712  702
-5  776  779
-7  217  208

7,-5,L

9  429  423
8  303  360
7  413  406
5  169  176
4  274  295
3  761  749
2  108   78
1  428  408
0 1430 1424
-1  572  553
-2  377  350
-3  525  522
-4  767  767
-5  313  343
-6  111  103
-7  148  121
-9  240  252

7,-6,L

9  189  221
8  584  488
7  394  389
5  639  637
4  385  429
3  713  665
1  130  124
0 1428 1449
-1  997 1004

-2  721  725
-3  348  352
-4  727  716
-5  485  483
-6  141  198
-7  196  201
-8  462  437
-9  234  189

7,-7,L

8  347  369
7  501  517
6  223  229
4  367  368
3  141  179
1  689  708
0  528  631
-1  576  562
-2  550  546
-3  147   44
-4  178  182
-5  362  365
-6  326  356
-7  199  127
-8  250  285

7,-8,L

7  123  127
6  365  346
4  224  197
3  356  353
2  353  395
1  354  345
0  173  193
-1  183  190
-2  125   99
-3  497  522
-4  131  126
-5  117   97
-6  329  299
-7  274  257

7,-9,L

8  508  524
7  311  288
6  179  198
5  338  317
4  305  326
3  315  324
1  157   30
0  583  490
-1  592  594
-3  312  314
-4  416  437
-5  334  288

7,-10,L

6  349  365
5  319  341
3  507  513
1  131   51
-1  332  332
-2  191  191
-4  463  468
-5  328  333

7,-11,L

7  208  274
4  261  273
3  165  186
1  164  167
0  398  400
-3  332  306

7,-12,L

6  247  281
5  324  301
3  319  305
1  153  174
0  223  221
-1  285  260

7,-13,L

4  234  213
3  198  173
2  175  173
0  133   57

6,12,L

-3  249  286
-4  264  263

6,11,L

0  327  286
-1  281  218
-3  277  295
-4  344  352
-5  281  275
-7  129  163
-8  222  241

6,10,L

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0  188  155
-1  340  373
-2 [illegible]
-3  208  199
-4  224  182
-5  370  392
-6  239  228
-7  221  275
-8  208  286

6,9,L

3  184  144
2  290  311
0  652  655
-3  455  472
-4  649  607
-5  242  285
-6  191  221

-8  246  265
-9  355  356

6,8,L

3  148   72
0  401  435
-1  337  363
-2  224  232
-3  313  256
-4 1108 1115
-7  393  376
-8  133  158
-10  167  138

6,7,L

5  241  237
4  197  181
3  148  138
2  288  275
1  331  318
0  339  351
-2  328  341
-3  382  307
-4  312  272
-5  245  214
-6  203  191
-7  152  118
-8  445  454
-10  137  147
-11  289  301

6,6,L

8  344  356
5  170  149
4  471  479
3  138  134
2  348  332
1  415  412
0  524  533
-2  507  614
-3  988  967
-4  625  620
-5  510  499
-6  401  379
-7  520  508
-9  286  310
-11  304  343

6,5,L

7  144  114
6  228  196
5  377  393
4  158  163
3  128   88
2  150   96
1  410  380
0 1580 1629
-1  331  310
-2  565  544
-3  833  844
-4  814  797
-5  168  125
-6  251  273
-7  178  163
-8  582  560
-9  587  583

6,4,L

5  490  444
4  131   40
2  128   78
1  726  754
0  847  851
-1  525  515
-2   91   51
-3  183   92
-4 1294 1273
-5  262  232
-6  128   85
-7  284  284
-8  585  597
-9  217  199
-11  224  232

6,3,L

6  582  567
5  267  274
4  297  310
3  228  212
2  143  142
1  791  793
0  731  710
-1  668  674
-2  422  419
-3 1088 1068
-5  180  160
-6  289  288
-7  273  283
-8  548  545
-9  266  271
-12  389  377

6,2,L

8  264  214
7  149   99
6  931  930
5  509  496
4  188  195
2  238  232
1  186  185
0  185   74
-1  676  659
-2  245  190
-3 1567 1557
-4 1922 1920
-5  219  222
-6  877  863
-7  453  471
-9  184  222
-12  315  325

6,1,L

9  185  181
8  239  271

6  222  284
5  759  774
4  231  226
3   92   22
2  345  331
1  192  200
0 1034 1014
-1  188   43
-2  923  942
-3  252  276
-4 1240 1262
-5  355  336
-7  654  681
-8  833  833
-9  198  166

6,0,L

9  172  123
7  228  192
6  782  888
5  173  189
4  268  288
3  116   96
1  892  645
0  342  371
-1  152   87
-2  186  132
-3 1744 1712
-4  195  182
-5  292  265
-6  669  691
-7  642  647
-8  375  393
-9  217  193
-11  353  368

6,-1,L

9  288  296
8  333  345
7  217  226
6  220  203
5  280  271
4  157  152
3  126   21
2 1225 1197
1 1314 1278
0 1713 1672
-1  389  385
-2  346  349
-3 1770 1743
-4  230  207
-5  416  391
-6  336  347
-7  995 1011
-8  337  335
-9  129   75

6,-2,L

10  178  184
9  386  401
8  185  182
7  381  383
6  545  562
5  427  448
4  874  905
3  156  142
2  753  815
1  367  308
0  470  502
-1  381  383
-2  379  348
-3 1294 1320
-4  961  971
-5  744  717
-6  136   13
-7  220  289
-8  350  362
-9  263  247
-10  127   26
-11  334  348

6,-3,L

10  383  288
7  556  547
5  127  113
4  156  148
3  820  841
2  270  308
1 1223 1246
0  163  200
-1  255  294
-2 1544 1532
-3  684  594
-4  533  525
-5  185  223
-6  177  172
-7  194  214
-8  582  582
-9  159  158

6,-4,L

10  381  374
9  411  433
8  120   75
6 1040 1075
5  220  266
3  452  447
2  524  554
1  680  633
0  391  364
-2  455  452
-3 1892 1943
-4  610  624
-5  454  448
-6  672  678
-7  225  321
-8  232  246

6,-5,L

10  176  136
9  242  274
8  157  132
7  226  229
6  491  475
5  487  467
3  279  287

2  840  832
1  325  317
0   92   66
-1  527  526
-2  281  289
-3  756  748
-4  674  646
-5  284  166
-6  180   77
-7  780  755
-8  178  129
-9  188  184

6,-6,L

8  282  286
7  393  378
5  374  351
4  111   83
3  456  447
2 1307 1285
1  131   87
0  286  278
-1  222  258
-2  818  798
-3  138  110
-4  132  120
-6  546  523
-7  281  279
-8  224  216
-9  172  172

6,-7,L

9  171  182
7  652  643
6  577  552
5  175  169
4  169  215
3  410  455
1  742  752
0  612  623
-1  126  151
-2  678  698
-3  626  646
-4  286  291
-6  577  575
-7  366  352

6,-8,L

9  353  392
8  446  436
5  578  576
4  225  234
3  475  468
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 -2 1090 1920
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 -6 1402 1422
 -7  477  486
 -8  383  271
 -9  188  174
-11  300  251

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  3   92   69
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 -3  433  424
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 -5  535  546
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  3  211  210
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 -7  171  160

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 -4  198  187
 -5  230  225
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-11  480  476
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 -6  145  152
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 -3  253  242
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  4  246  243
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  4  754  732
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  2 1427 1365

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```

TABLE 5

π-Cyclopentadienyl-cis-dicarbonylbromo(triphenylphosphine)molybdenum - methylene chloride; fractional atomic coordinates and population parameters (PP.), if other than 1.0, with esd in parentheses and thermal parameters.

Atom	x	y	z	PP.	$U_{iso}(x10^3Å^2)$
Mo(1)	0.83611(4)	0.66078(6)	-0.04501(6)		*
Br(1)	0.71875(6)	0.65433(11)	0.04731(11)	0.792(4)	*
Br(1)'	0.88706(29)	0.84269(48)	-0.12185(46)	0.208	*
P(1)	0.90319(11)	0.81259(16)	0.17641(18)		*
C(1)	0.8760(4)	0.7906(7)	0.3173(7)		*
C(2)	0.8547(6)	0.6802(8)	0.3256(8)		*
C(3)	0.8343(6)	0.6625(9)	0.4312(9)		*
C(4)	0.8320(6)	0.7509(10)	0.5280(9)		*
C(5)	0.8538(5)	0.8611(10)	0.5254(8)		*
C(6)	0.8755(5)	0.8819(8)	0.4194(8)		*
C(7)	1.0053(4)	0.8231(7)	0.2288(8)		*
C(8)	1.0418(5)	0.7990(8)	0.3438(10)		*
C(9)	1.1172(6)	0.7980(9)	0.3751(12)		*
C(10)	1.1568(6)	0.8245(10)	0.2946(14)		*
C(11)	1.1235(5)	0.8501(9)	0.1819(12)		*
C(12)	1.0479(5)	0.8496(8)	0.1508(9)		*
C(13)	0.8972(4)	0.9610(6)	0.1836(7)		*
C(14)	0.9586(5)	1.0525(8)	0.2393(11)		*
C(15)	0.9501(6)	1.1633(8)	0.2462(15)		*
C(16)	0.8808(7)	1.1818(8)	0.1971(12)		*
C(17)	0.8193(5)	1.0913(8)	0.1410(10)		*
C(18)	0.8264(5)	0.9797(7)	0.1329(10)		*
C(19)	0.8733	0.7929	-0.1007	0.80	63
O(19)	0.8949	0.8697	-0.1331	0.80	95
C(19)'	0.7502	0.6562	0.0228	0.20	63
O(19)'	0.7002	0.6535	0.0622	0.20	95
C(20)	0.7429(6)	0.6531(7)	-0.2028(13)		*
O(20)	0.6994(4)	0.6468(6)	-0.2782(7)		*
C(21)	0.9387(5)	0.5893(8)	-0.0640(10)		*
C(22)	0.8765(5)	0.5338(8)	-0.1828(8)		*
C(23)	0.8202(5)	0.4686(7)	-0.1504(8)		*
C(24)	0.8478(6)	0.4779(8)	-0.0180(10)		*
C(25)	0.9191(6)	0.5526(8)	0.0377(9)		*

Atom	x	y	z	PP.	$U_{iso}(\times 10^{3}\,\text{Å}^{2})$
H(2)	0.855	0.616	0.256		55
H(3)	0.823	0.584	0.438		62
H(4)	0.813	0.737	0.598		64
H(5)	0.854	0.925	0.596		54
H(6)	0.891	0.959	0.417		50
H(8)	1.013	0.781	0.399		57
H(9)	1.143	0.782	0.456		74
H(10)	1.209	0.823	0.314		81
H(11)	1.151	0.868	0.125		63
H(12)	1.023	0.870	0.073		56
H(14)	1.008	1.040	0.274		63
H(15)	0.994	1.228	0.286		86
H(16)	0.875	1.259	0.201		76
H(17)	0.770	1.104	0.108		59
H(18)	0.783	0.915	0.092		58
H(21)	0.985	0.643	-0.054		62
H(22)	0.874	0.540	-0.269		52
H(23)	0.771	0.424	-0.211		51
H(24)	0.822	0.439	0.030		65
H(25)	0.951	0.574	0.129		64
Mo(101)	0.61945(4)	0.35353(6)	0.27555(7)		*
Br(101)	0.73785(7)	0.39556(12)	0.49201(12)	0.689(4)	*
Br(101)'	0.58646(26)	0.16768(35)	0.07178(38)	0.311	*
P(101)	0.57168(10)	0.20250(16)	0.37428(18)		*
C(101)	0.5977(4)	0.2428(6)	0.5538(7)		*
C(102)	0.5915(5)	0.3493(7)	0.6221(8)		*
C(103)	0.6094(5)	0.3821(8)	0.7582(8)		*
C(104)	0.6329(5)	0.3061(9)	0.8278(8)		*
C(105)	0.6388(5)	0.2018(8)	0.7616(8)		*
C(106)	0.6202(5)	0.1686(7)	0.6255(8)		*
C(107)	0.4677(4)	0.1549(7)	0.3181(8)		*
C(108)	0.4280(5)	0.1562(8)	0.4076(9)		*
C(109)	0.3494(6)	0.1212(9)	0.3617(13)		*
C(110)	0.3110(5)	0.0880(9)	0.2276(16)		*
C(111)	0.3509(6)	0.0867(9)	0.1413(12)		*
C(112)	0.4294(5)	0.1196(8)	0.1844(9)		*
C(113)	0.6022(4)	0.0685(6)	0.3329(7)		*
C(114)	0.5517(5)	-0.0383(7)	0.2848(9)		*

Atom	x	y	z	PP.	$U_{iso}(x10^{3}\AA^{2})$
C(115)	0.5773(7)	-0.1367(8)	0.2517(11)		*
C(116)	0.6519(8)	-0.1298(9)	0.2751(12)		*
C(117)	0.7021(6)	-0.0229(10)	0.3237(13)		*
C(118)	0.6782(5)	0.0761(8)	0.3527(11)		*
C(119)	0.5929	0.2160	0.1242	0.69	63
O(119)	0.5775	0.1360	0.0362	0.69	95
C(119)'	0.7064	0.3844	0.4346	0.31	63
O(119)'	0.7571	0.4024	0.5270	0.31	95
C(120)	0.7153(7)	0.3639(8)	0.2136(9)		*
O(120)	0.7561(4)	0.3677(7)	0.1854(7)		*
C(121)	0.5084(5)	0.4004(9)	0.1809(12)		*
C(122)	0.5687(7)	0.4634(10)	0.1504(10)		*
C(123)	0.6187(6)	0.5401(8)	0.2715(11)		*
C(124)	0.5886(7)	0.5252(8)	0.3716(10)		*
C(125)	0.5209(6)	0.4395(9)	0.3145(12)		*
H(102)	0.575	0.401	0.574		47
H(103)	0.605	0.456	0.802		62
H(104)	0.644	0.328	0.921		61
H(105)	0.658	0.151	0.810		55
H(106)	0.621	0.093	0.579		47
H(108)	0.455	0.181	0.502		59
H(109)	0.322	0.120	0.424		76
H(110)	0.257	0.066	0.196		83
H(111)	0.323	0.063	0.048		73
H(112)	0.458	0.118	0.122		52
H(114)	0.498	-0.045	0.273		53
H(115)	0.542	-0.210	0.212		73
H(116)	0.670	-0.199	0.258		85
H(117)	0.755	-0.018	0.339		85
H(118)	0.714	0.150	0.387		67
H(121)	0.466	0.341	0.119		69
H(122)	0.574	0.455	0.065		81
H(123)	0.665	0.595	0.282		73
H(124)	0.611	0.567	0.465		71
H(125)	0.489	0.412	0.361		81
Cℓ	0.99289(27)	0.48825(34)	0.36332(38)		*
C(50)	0.9596(19)	0.4381(28)	0.4816(34)	0.50	123
H(50a)	0.967	0.354	0.471	0.50	122
H(50b)	0.906	0.431	0.464	0.50	122

* These atoms were assigned anisotropic temperature factors of the form: $T = \exp\left[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}\ell^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}h\ell a^*c^* + 2U_{23}k\ell b^*c^*)\right]$ with final parameters ($U_{ij} \times 10^4 Å^2$):

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Mo(1)	372	374	317	85	128	80
Br(1)	431	676	486	25	197	-7
Br(1)'	637	830	481	48	133	55
P(1)	323	354	316	74	100	94
C(1)	303	454	285	34	45	94
C(2)	758	488	392	24	220	113
C(3)	755	640	523	-179	131	188
C(4)	715	896	356	-11	127	196
C(5)	550	896	333	208	142	32
C(6)	508	566	397	137	66	130
C(7)	397	383	546	110	174	136
C(8)	392	583	720	144	124	288
C(9)	446	661	1023	172	-47	358
C(10)	389	834	1119	141	264	87
C(11)	423	652	867	-62	347	-7
C(12)	501	584	572	43	252	67
C(13)	436	338	354	71	121	42
C(14)	318	406	1095	623	-5	166
C(15)	660	331	1595	77	285	175
C(16)	750	403	1065	211	176	206
C(17)	493	501	819	216	5	158
C(18)	502	411	713	145	100	130
C(20)	638	263	907	96	490	168
O(20)	686	470	507	80	9	82
C(21)	470	620	767	284	302	119
C(22)	540	530	418	182	193	31
C(23)	541	362	501	120	199	37
C(24)	846	401	682	267	383	203
C(25)	732	642	451	490	110	152

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Mo(101)	375	380	331	105	111	149
Br(101)	431	608	458	-41	42	237
Br(101)'	1117	791	685	36	274	305
P(101)	292	310	330	78	80	92
C(101)	318	336	435	63	141	134
C(102)	546	461	456	181	231	221
C(103)	699	594	423	236	299	161
C(104)	524	704	382	34	193	184
C(105)	460	674	438	139	106	256
C(106)	506	446	444	86	248	196
C(107)	304	369	525	106	77	122
C(108)	396	512	719	112	216	188
C(109)	493	672	1080	156	367	430
C(110)	314	654	1448	159	126	488
C(111)	392	673	992	57	-132	223
C(112)	461	531	610	-96	-9	148
C(113)	421	370	341	140	120	100
C(114)	607	364	677	102	260	60
C(115)	894	436	806	245	266	101
C(116)	1127	596	969	535	573	279
C(117)	682	833	1284	489	538	540
C(118)	453	455	1034	239	314	266
C(120)	980	459	317	363	82	118
O(120)	618	797	581	51	323	390
C(121)	430	598	922	259	31	141
C(122)	896	889	636	594	347	517
C(123)	760	471	928	265	329	436
C(124)	943	419	661	395	264	161
C(125)	788	606	912	394	499	336
Cℓ	1667	1069	991	438	652	441

<u>Average estimated standard deviations.</u> (U_{ij}x10^{4}Å^2) and (U_{iso}x10^{3}Å^2)

Atom	U_{11} or U_{iso}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Mo	4	4	3	3	3	3
Br	8	9	8	6	6	6
Br^{1}	31	31	24	24	21	22
P	10	10	10	8	8	8
C	59	58	66	48	51	50
O	47	45	43	37	36	35
Cℓ	38	27	25	26	26	21
C(50)	10					

TABLE 6

π-Cyclopentadienyl-cis-dicarbonylbromo(triphenylphosphine)molybdenum - methylene chloride; bond lengths (Å) with esd in parentheses.

Bond[†]	Molecule A1	Molecule A101
Mo(1) - Br(1)	2.676(2)	2.666(1)
Mo(1) - Br(1)'	2.698(6)	2.648(4)
Mo(1) - P(1)	2.538(2)	2.525(2)
Mo(1) - C(20)	2.087(11)	2.095(14)
C(20) - O(20)	0.977(13)	0.904(17)
Mo(1) - C(21)	2.303(11)	2.313(10)
Mo(1) - C(22)	2.290(10)	2.294(13)
Mo(1) - C(23)	2.310(8)	2.315(11)
Mo(1) - C(24)	2.383(10)	2.374(11)
Mo(1) - C(25)	2.363(11)	2.379(13)
P(1) - C(1)	1.823(9)	1.822(8)
P(1) - C(7)	1.834(8)	1.837(8)
P(1) - C(13)	1.837(8)	1.838(9)
C(1) - C(2)	1.385(13)	1.392(11)
C(2) - C(3)	1.371(16)	1.384(12)
C(3) - C(4)	1.349(15)	1.400(15)
C(4) - C(5)	1.361(17)	1.360(14)
C(5) - C(6)	1.400(15)	1.383(12)
C(6) - C(1)	1.394(11)	1.390(13)
C(7) - C(8)	1.395(14)	1.389(15)
C(8) - C(9)	1.378(14)	1.387(13)
C(9) - C(10)	1.364(21)	1.381(19)
C(10) - C(11)	1.366(19)	1.360(21)
C(11) - C(12)	1.376(14)	1.386(13)
C(12) - C(7)	1.381(15)	1.376(12)
C(13) - C(14)	1.360(10)	1.379(11)
C(14) - C(15)	1.389(15)	1.387(15)
C(15) - C(16)	1.358(17)	1.347(19)
C(16) - C(17)	1.354(12)	1.377(15)
C(17) - C(18)	1.390(14)	1.370(17)
C(18) - C(13)	1.387(13)	1.377(13)

Bond†			Molecule A1	Molecule A101
C(21)	-	C(22)	1.430(11)	1.430(17)
C(22)	-	C(23)	1.399(14)	1.410(13)
C(23)	-	C(24)	1.379(14)	1.403(19)
C(24)	-	C(25)	1.383(13)	1.394(14)
C(25)	-	C(21)	1.414(16)	1.375(17)

'Fixed' bond lengths.

Bond			Molecule A1	Molecule A101
Mo(1)	-	C(19)	1.96	1.96
Mo(1)	-	C(19)'	1.96	1.96
C(19)	-	O(19)	1.14	1.14
C(19)'	-	O(19)'	1.14	1.14
C	-	H	0.98	0.98

† The atomic numbering refers to A1. The numbering of A101 is 100 plus that in A1.

Methylene chloride

C(50) - Cℓ	1.78(4)
C(50) - Cℓ††	1.65(4)
C(50) - H(50a)	0.98(fixed)
C(50) - H(50b)	0.98(fixed)

†† Cℓ is transformed to the coordinates 2 - x, 1 - y, 1 - z.

TABLE 7

π-Cyclopentadienyl-cis-dicarbonylbromo(triphenylphosphine)molybdenum - methylene chloride; valency angles (°) with esd in parentheses.

	Molecule A1	Molecule A101
Br(1) - Mo(1) - Br(1)'	120.3(1)	121.6(1)
Br(1) - Mo(1) - P(1)	82.1(1)	79.6(1)
Br(1)' - Mo(1) - P(1)	78.8(1)	80.6(1)
Br(1) - Mo(1) - C(20)	74.0(4)	73.9(3)
Br(1)' - Mo(1) - C(20)	77.9(3)	74.4(3)
P(1) - Mo(1) - C(20)	131.4(3)	125.2(3)
Br(1) - Mo(1) - C(21)	147.7(3)	146.5(3)
Br(1) - Mo(1) - C(22)	136.6(2)	134.7(2)
Br(1) - Mo(1) - C(23)	101.9(3)	99.9(2)
Br(1) - Mo(1) - C(24)	91.7(3)	90.2(2)
Br(1) - Mo(1) - C(25)	113.1(3)	113.7(3)
Br(1)' - Mo(1) - C(21)	90.8(3)	91.2(3)
Br(1)' - Mo(1) - C(22)	94.4(3)	94.6(3)
Br(1)' - Mo(1) - C(23)	127.1(3)	127.8(3)
Br(1)' - Mo(1) - C(24)	148.0(4)	148.3(3)
Br(1)' - Mo(1) - C(25)	120.7(3)	119.5(3)
P(1) - Mo(1) - C(21)	97.1(2)	101.1(3)
P(1) - Mo(1) - C(22)	133.2(2)	137.2(3)
P(1) - Mo(1) - C(23)	141.4(2)	141.8(3)
P(1) - Mo(1) - C(24)	108.2(2)	107.3(3)
P(1) - Mo(1) - C(25)	85.0(2)	87.0(3)
C(20) - Mo(1) - C(21)	125.2(4)	126.9(5)
C(20) - Mo(1) - C(22)	90.7(4)	93.2(5)
C(20) - Mo(1) - C(23)	85.5(3)	90.2(4)
C(20) - Mo(1) - C(24)	114.1(3)	119.6(4)
C(20) - Mo(1) - C(25)	143.3(3)	147.6(4)
Mo(1) - P(1) - C(1)	117.2(2)	116.9(2)
Mo(1) - P(1) - C(7)	111.1(3)	112.7(3)
Mo(1) - P(1) - C(13)	116.5(2)	114.6(3)
C(1) - P(1) - C(7)	103.4(4)	103.7(4)
C(1) - P(1) - C(13)	103.2(4)	103.4(4)
C(7) - P(1) - C(13)	103.8(4)	104.0(3)
P(1) - C(1) - C(2)	120.1(6)	119.3(7)
P(1) - C(1) - C(6)	122.3(7)	122.3(6)

	Molecule A1	Molecule A101
C(6) - C(1) - C(2)	117.6(8)	118.3(7)
C(1) - C(2) - C(3)	120.6(8)	121.0(9)
C(2) - C(3) - C(4)	121.5(11)	119.4(9)
C(3) - C(4) - C(5)	120.0(11)	119.9(8)
C(4) - C(5) - C(6)	119.8(9)	120.8(10)
C(5) - C(6) - C(1)	120.4(9)	120.7(8)
P(1) - C(7) - C(8)	121.7(7)	121.2(6)
P(1) - C(7) - C(12)	120.5(7)	118.5(7)
C(12) - C(7) - C(8)	117.7(8)	120.3(8)
C(7) - C(8) - C(9)	120.5(11)	119.7(9)
C(8) - C(9) - C(10)	119.5(11)	119.9(13)
C(9) - C(10) - C(11)	121.8(10)	119.6(10)
C(10) - C(11) - C(12)	118.2(12)	121.7(11)
C(11) - C(12) - C(7)	122.2(10)	118.8(10)
P(1) - C(13) - C(14)	122.9(7)	122.1(7)
P(1) - C(13) - C(18)	118.0(6)	118.4(6)
C(18) - C(13) - C(14)	119.1(8)	119.5(8)
C(13) - C(14) - C(15)	120.3(9)	119.9(9)
C(14) - C(15) - C(16)	120.7(8)	120.7(9)
C(15) - C(16) - C(17)	119.6(9)	119.1(12)
C(16) - C(17) - C(18)	120.8(9)	121.5(11)
C(17) - C(18) - C(13)	119.6(7)	119.3(8)
Mo(1) - C(20) - O(20)	178.0(10)	179.0(8)
C(25) - C(21) - C(22)	106.7(8)	108.8(8)
C(21) - C(22) - C(23)	107.6(9)	106.4(10)
C(22) - C(23) - C(24)	108.0(7)	108.0(10)
C(23) - C(24) - C(25)	109.9(10)	108.5(9)
C(24) - C(25) - C(21)	107.7(8)	108.4(11)

The disordered carbonyl groups were 'fixed' to give a linear Mo - C - Br - O geometry.

The C(sp^2) hydrogen atoms were 'fixed' with the C - H vector bisecting the C - C - C angle.

<u>Methylene chloride</u>

Cℓ - C - Cℓ†† 115(2)

†† Cℓ is transformed to the coordinates 2 - x, 1 - y, 1 - z.

The C(sp^3) hydrogen atoms were 'fixed' to give a H - C - H angle of 109°, with the vector bisecting the H - C - H angle also bisecting the Cℓ - C - Cℓ angle.

TABLE 8

π-Cyclopentadienyl-cis-dicarbonylbromo(triphenylphosphine)molybdenum - methylene chloride; mean plane calculations.

	Atoms in plane	Displacements (Å)	Atoms out of plane	Displacements (Å)
a)	C(1)	0.005	P(1)	0.021
	C(2)	0.003		
	C(3)	-0.013		
	C(4)	0.015		
	C(5)	-0.007		
	C(6)	-0.003		
b)	C(7)	0.008	P(1)	0.138
	C(8)	-0.010		
	C(9)	0.007		
	C(10)	-0.001		
	C(11)	-0.001		
	C(12)	0.003		
c)	C(13)	-0.003	P(1)	-0.064
	C(14)	0.002		
	C(15)	0.000		
	C(16)	-0.001		
	C(17)	0.000		
	C(18)	0.002		
d)	C(21)	-0.006	Mo(1)	-1.999
	C(22)	0.014		
	C(23)	-0.018		
	C(24)	0.015		
	C(25)	-0.006		

	Atoms in plane	Displacements (Å)	Atoms out of plane	Displacements (Å)
e)	C(101)	-0.010	P(101)	0.030
	C(102)	0.005		
	C(103)	0.000		
	C(104)	0.002		
	C(105)	-0.008		
	C(106)	0.012		
f)	C(107)	-0.003	P(101)	-0.048
	C(108)	-0.005		
	C(109)	0.010		
	C(110)	-0.008		
	C(111)	0.000		
	C(112)	0.006		
g)	C(113)	0.002	P(101)	-0.005
	C(114)	-0.018		
	C(115)	0.025		
	C(116)	-0.015		
	C(117)	-0.001		
	C(118)	0.007		
h)	C(121)	-0.006	Mo(101)	-2.005
	C(122)	0.007		
	C(123)	-0.005		
	C(124)	0.001		
	C(125)	0.003		

The dihedral angles (°) between selected planes are:

a) - b)	83.1	e) - f)	79.8
a) - c)	84.5	e) - g)	99.3
b) - c)	70.3	f) - g)	105.5

TABLE 9

π-Cyclopentadienyl-cis-dicarbonylbromo(triphenylphosphine)molybdenum - methylene chloride; intermolecular contacts ($\leqslant$3.75Å).

O(119)$'$	---	C(22^{I})	3.22	C(16)	---	C(50^{VII})	3.64
O(20)	---	C(110^{II})	3.24	C(6)	---	C(117^{VII})	3.64
O(119)$'$	---	C(23^{I})	3.27	O(119)	---	C(111^{V})	3.65
O(19)	---	C(14^{III})	3.37	Br(1)$'$	---	C(14^{III})	3.66
O(20)	---	C(109^{II})	3.40	O(20)	---	C(124^{VI})	3.66
C(21)	---	C(25^{IV})	3.43	C(105)	---	C(114^{XI})	3.66
O(119)	---	C(112^{V})	3.45	C(18)	---	C(111^{II})	3.67
O(19)	---	C(4^{VI})	3.45	O(20)	---	C(111^{II})	3.67
O(119)	---	C(114^{V})	3.50	O(19)	---	C(11^{III})	3.68
O(19)$'$	---	C(123)	3.51	C(23)	---	C(104^{VI})	3.68
O(19)	---	C(12^{III})	3.52	O(119)	---	C(104^{VI})	3.68
O(19)	---	C(15^{III})	3.53	C(15)	---	C(50^{VII})	3.69
O(19)$'$	---	C(116^{VII})	3.54	C(17)	---	C(117^{VII})	3.69
Br(1)$'$	---	C(4^{VI})	3.55	C(21)	---	C(21^{IV})	3.69
C(18)	---	C(117^{VII})	3.55	O(119)	---	C(115^{V})	3.69
O(120)	---	C(16^{VIII})	3.55	C(102)	---	C(125^{X})	3.69
O(120)	---	C(24)	3.55	C(20)	---	C(103^{VI})	3.69
C(20)	---	C(110^{II})	3.56	Br(1)$'$	---	C(15^{III})	3.70
Cℓ	---	C(22^{IV})	3.57	Br(101)$'$	---	C(104^{VI})	3.72
O(20)	---	C(103^{VI})	3.58	O(119)	---	C(105^{VI})	3.72
C(5)	---	C(14^{IX})	3.60	C(17)	---	C(111^{II})	3.72
O(19)	---	C(5^{VI})	3.60	O(120)	---	C(2)	3.73
O(19)$'$	---	C(122)	3.61	C(25)	---	C(25^{IV})	3.73
C(103)	---	C(125^{X})	3.63	C(106)	---	C(109^{XI})	3.74
Br(101)	---	C(22^{I})	3.63	C(10)	---	C(23^{IV})	3.74
Br(101)	---	C(23^{I})	3.64	C(23)	---	C(103^{VII})	3.74
O(20)	---	C(102^{VI})	3.64				

The superscripts refer to the following transformations of the atomic coordinates:

I	x,	y,	$1 + z$;	VII	x,	$1 + y$,	z;
II	$1 - x$,	$1 - y$,	$- z$;	VIII	x,	$-1 + y$,	z;
III	$2 - x$,	$2 - y$,	$- z$;	IX	$2 - x$,	$2 - y$,	$1 - z$;
IV	$2 - x$,	$1 - y$,	$- z$;	X	$1 - x$,	$1 - y$,	$1 - z$;
V	$1 - x$,	$- y$,	$- z$;	XI	$1 - x$,	$- y$,	$1 - z$.
VI	x,	y,	$-1 + z$;				

II.2.3 EXPERIMENTAL

Crystal Data π-Cyclopentadienyl-trans-dicarbonyl(phenyl isocyanide)-molybdenum; $C_{14}H_{10}O_2NIMo$.

Crystal system	Orthorhombic
Unit cell dimensions	a = 12.035(4) Å
	b = 17.248(5) Å
	c = 14.510(5) Å
Space group	Pbca (D_{2h}^{15})

U = 3011.9 $Å^3$

M = 447.1 a.m.u.

D_o = 1.95 $gm.cm.^{-3}$ (flotation in aqueous zinc iodide solution)

D_c = 1.972 $gm.cm.^{-3}$

Z = 8

F(000) = 1696

μ(Mo-$K\alpha$) = 29.25 $cm.^{-1}$

Crystallographic Measurements

Preliminary cell dimensions, and systematically absent reflections ($h0\ell$ absent if $\ell = 2n + 1$, $0k\ell$ absent if $k = 2n + 1$ and $hk0$ absent if $h = 2n + 1$) consistent with space group Pbca were found from precession and Wiessenburg photographs (using Mo-$\underline{K}\alpha$ and Cu-$\underline{K}\alpha$ radiation respectively) of a red diamond-shaped plate with dimensions ca. 0.01 x 0.45 x 0.50 mm^3.

An initial set of diffractometer data, collected using zirconium-filtered Mo-$\underline{K}\alpha$ radiation, refined unsatisfactorily due to the large number of weakly observed reflections, therefore a second set was collected and the counting statistics improved, both by using graphite-crystal monochromatised Mo-$\underline{K}\alpha$ radiation and by increasing the counting times. A fully stabilized X-ray generator operated at 46 kV and 16 mA and a Hilger and Watts' Y290 automatic diffractometer were used in both data collections. In collection I, the diffractometer was controlled by a PDP-8 computer (a DF32 DEC disc file backed PDP-8 computer in collection II) and the cell dimensions were adjusted by a least-squares treatment of the θ, χ and ϕ setting angles of 11 (14) reflections.

In both collections the intensity measurements were made, in octant $h\bar{k}\ell$ with $\theta(\text{Mo-}\underline{K}\alpha) \leqslant 27^\circ$, by the $\theta - 2\theta$ scan procedure, each reflection being scanned in 36 equal steps from $2\theta_{calc.} -0.72^\circ$ to $2\theta_{calc.} +0.72^\circ$. In collection I, each step was counted for 2 seconds and stationary-crystal, stationary-counter background counts were measured at each end of the scan range for 18 seconds. (In collection II, three θ-ranges $\theta(\text{Mo-}\underline{K}\alpha) \leqslant 16^\circ$, $16^\circ \leqslant \theta(\text{Mo-}\underline{K}\alpha) \leqslant 23^\circ$ and $23^\circ \leqslant \theta(\text{Mo-}\underline{K}\alpha) \leqslant 27^\circ$ were collected, the corresponding counting times being 4 and 36, 6 and 54, and 8 and 72 seconds respectively.) The intensities of 2 (3) standard reflections were monitored every 40 (30) intensity measurements and the results used to ensure that all the observations were on a common scale. The scan counts (P) and the background counts (B_1,B_2) of each reflection were combined to yield the integrated intensity, $I = P - 2(B_1 + B_2)$, which was corrected for Lorentz-polarisation effects but not for absorption. In collection I, 3,284 independent measurements were obtained and only the 1,742 with $I/\sigma_I > 2.0$ used in the structure analysis. (In collection II, only the 1,564 reflections were measured, which a quick scan had shown to be significantly above background, and, of these, the 1,471 with $I/\sigma_I > 3.0$ were used in the final refinement.)

Structure Analysis

Using the first set of data, the coordinates of both the molybdenum and iodine atoms were derived from the three-dimensional Patterson synthesis, and the subsequent electron density distribution ($R = 36\%$) revealed the positions of the remaining non-hydrogen atoms.

The approximate atomic parameters were adjusted by a series of least-squares calculations, using the program due to D. W. J. Cruickshank, J. G. F. Smith and J. G. Sime on Glasgow University's KDF9 computer. The weighting scheme:

$$w = \left[1 - \exp(-p_1\{\sin\theta/\lambda\}^2)\right] / \left[1 + p_2|F_o| + p_3|F_o|^2\right]$$

was employed, and the values of p_1, p_2 and p_3 adjusted at several stages of the refinement to ensure approximate constancy of $\langle w\Delta^2\rangle$ over various ranges of $|F_o|$ and $\sin\theta$. The final values were 90.0, 0.54 and 0.01 respectively. The atomic scattering factors, including dispersion corrections $\Delta f'$ and $\Delta f''$ for the molybdenum and iodine atoms, were taken from 'International Tables for X-ray Crystallography,' Vol. III.(28)

Two cycles of full-matrix refinement on the positional and isotropic thermal parameters lowered the R-factor to 11.1%. With all the atoms assigned anisotropic thermal parameters, four cycles of block diagonal least-squares refinement converged at $R = 9.3\%$.

The final set of parameters were used as the starting set for refinement with the second set of data and, after four cycles of block diagonal least-squares, convergence was reached at $R = 4.33\%$. A difference electron density distribution was calculated revealing the positions of all the hydrogen atoms at an average peak height of $0.40e^-/\text{Å}^3$. The main deviations in this map were peaks of height ca. $0.7e^-/\text{Å}^3$ and holes of depth ca. $0.5e^-/\text{Å}^3$ in the region of the iodine atom. Each hydrogen atom was included in further structure factor calculations with an isotropic temperature factor equal to that of the carbon atom to which it was bonded and, after three cycles of block diagonal refinement of the parameters of the other atoms, the R-factor was lowered to 3.99%. The refinement was completed with four cycles of full-matrix least-squares; firstly, two cycles refining only the phenyl isocyanide, molybdenum and iodine atomic parameters, then two cycles refining only the cyclopentadienyl, carbonyl, molybdenum and iodine atomic parameters. Convergence was reached at R = 3.99%, R' = 5.58%.

Results

The observed and final calculated structure factors are listed in Table 10, with the final positional and thermal parameters, including the estimated standard deviations derived from the least-squares refinement, in Table 11. The atomic numbering scheme of the non-hydrogen atoms (each hydrogen atom is numbered according to the atom to which it is bonded) is shown in Figure 3; the projection of the molecule on to the plane of the π-cyclopentadienyl ring showing the thermal ellipsoids(30). The bond lengths, valency angles and displacements of atoms from various planes are given in Tables 12, 13 and 14 respectively. The crystal packing is shown in Figure 4 and the intermolecular contacts $\leqslant 3.75$ Å are listed in Table 15.

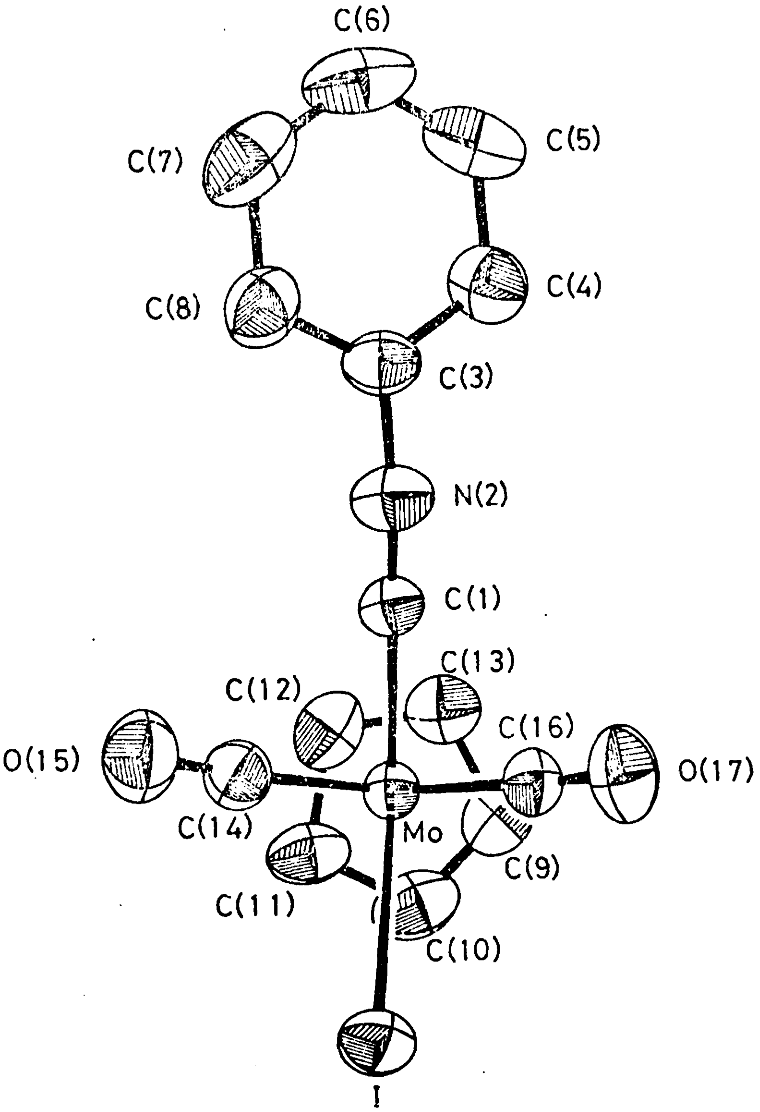

FIGURE 3

Perspective view of π-cyclopentadienyl-trans-dicarbonyliodo-(phenyl isocyanide)molybdenum projected on to the plane of the π-cyclopentadienyl ring, showing the 50% probability thermal ellipsoids.

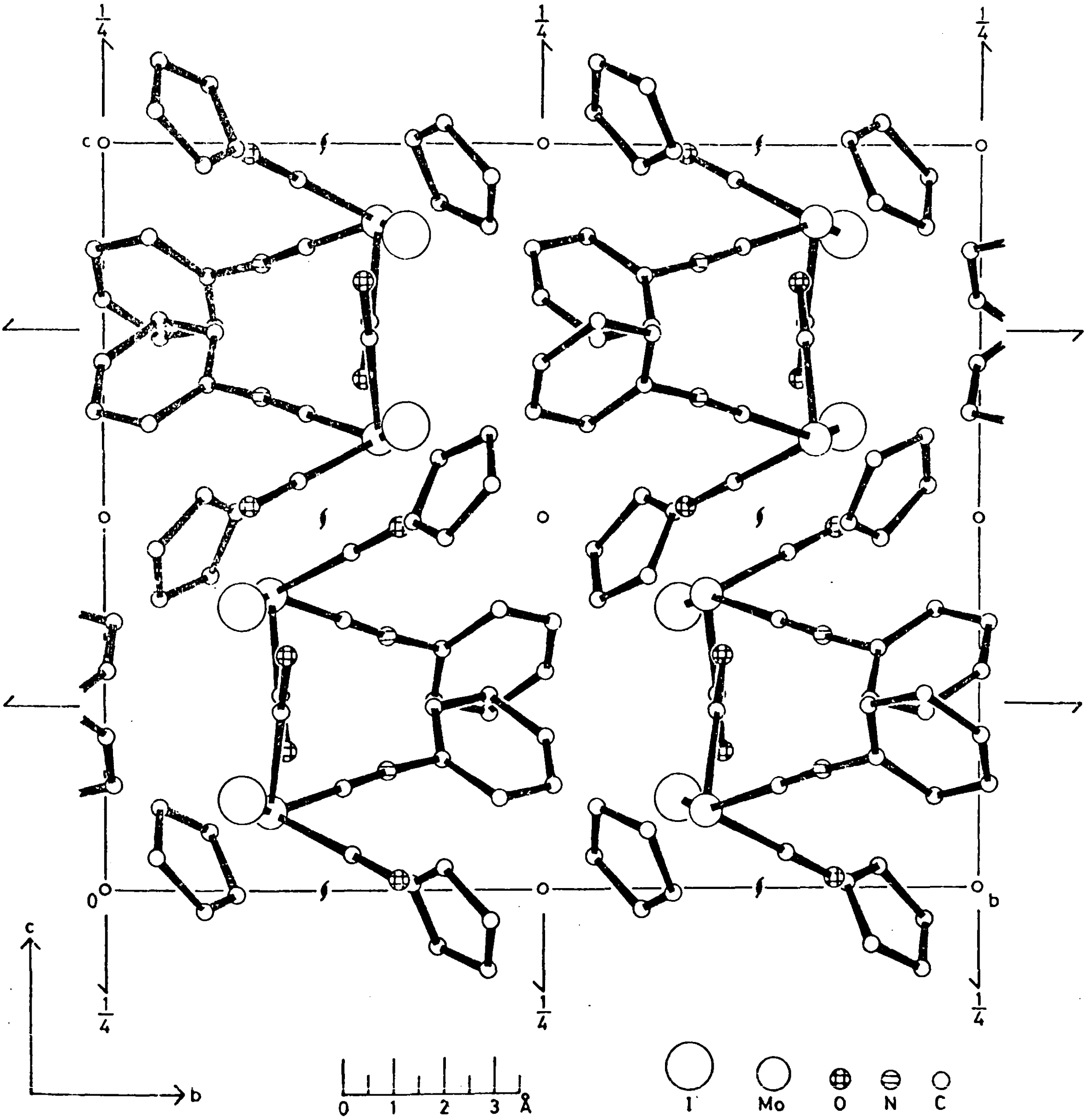

FIGURE 4

Crystal packing of π-cyclopentadienyl-trans-dicarbonyliodo-(phenyl isocyanide)molybdenum viewed along the a-axis.

TABLE 10

π-Cyclopentadienyl-trans-dicarbonyliodo(phenyl isocyanide)molybdenum: observed and final calculated structure amplitudes.

H	K	L	F OBS	F CALC
14	0	4	47.3	51.2
14	0	0	44.4	44.1
14	1	4	35.9	35.0
14	1	1	23.4	26.7
14	1	0	39.7	39.9
14	2	7	27.5	33.3
14	2	1	37.3	36.9
14	2	0	30.8	35.2
14	3	3	31.0	32.2
14	3	1	41.6	39.1
14	4	3	40.5	43.3
14	5	4	44.9	42.9
14	5	0	53.8	51.5
14	6	4	35.3	33.4
14	6	3	24.5	25.3
14	6	0	41.4	39.3
14	7	3	29.9	29.6
13	0	8	36.8	37.6
13	0	6	73.4	77.6
13	0	4	25.0	30.0
13	0	2	173.6	134.4
13	1	6	39.6	40.4
13	1	2	54.4	57.5
13	2	9	47.2	50.9
13	2	6	35.8	35.7
13	2	5	74.8	79.7
13	2	3	46.7	45.6
13	2	2	54.3	55.9
13	2	1	75.5	75.8
13	3	5	49.5	50.4
13	3	3	43.5	45.3
13	3	1	53.4	54.0
13	4	9	44.9	43.5
13	4	6	37.9	38.0
13	4	5	66.4	73.3
13	4	3	45.9	46.9
13	4	2	52.6	50.7
13	4	1	66.9	66.1
13	5	6	34.8	33.7
13	5	2	54.3	51.7
13	6	6	59.8	51.1
13	6	2	84.4	82.7
13	7	2	43.6	42.2
13	8	3	25.7	25.9
13	8	2	39.1	37.2
13	8	1	36.9	37.4
13	10	5	28.5	28.3
12	0	8	54.3	59.1
12	0	4	111.4	123.4
12	0	0	116.0	121.9
12	1	10	34.1	33.4
12	1	6	34.4	35.4
12	1	4	38.7	40.7
12	1	3	21.9	26.0
12	1	0	59.6	60.3
12	2	11	37.3	36.0
12	2	7	67.1	71.5
12	2	5	46.0	43.1
12	2	4	48.9	53.2
12	2	3	74.6	79.2
12	2	1	79.1	79.3
12	2	0	67.4	66.3
12	3	7	43.9	44.4
12	3	3	51.6	51.0
12	4	11	29.6	33.2
12	4	8	35.7	36.7
12	4	7	50.5	50.9
12	4	5	37.6	38.3
12	4	4	57.6	57.5
12	4	3	66.9	63.6
12	4	1	44.7	43.2
12	4	0	58.0	62.9
12	5	7	42.8	43.1
12	5	3	42.2	43.9
12	5	1	37.3	36.9
12	6	8	43.6	46.9
12	6	4	82.7	83.1
12	6	0	82.0	84.9
12	7	10	27.2	28.9
12	7	4	47.5	42.1
12	7	3	20.1	24.4
12	7	0	49.8	51.7
12	8	9	25.1	21.6
12	8	7	39.6	39.9
12	8	5	28.0	29.7
12	8	3	56.0	55.6
12	8	1	57.0	56.3
12	9	4	45.4	44.6
12	9	3	43.7	49.2
12	10	7	24.8	20.9
12	10	4	39.1	36.1
12	10	1	25.5	25.9
12	10	0	39.4	43.2
12	11	3	35.1	34.7
12	11	0	21.0	22.0
12	12	0	35.7	32.7
12	13	3	33.3	32.9
11	0	10	27.9	25.5
11	0	6	42.4	43.4
11	0	2	35.6	42.5
11	1	12	32.0	29.7
11	1	8	32.9	34.6
11	2	7	37.2	36.0
11	2	3	33.7	33.0
11	2	1	36.1	35.3
11	3	11	43.1	41.5
11	3	7	41.7	45.9
11	3	6	24.8	30.8
11	3	2	52.5	57.2
11	4	6	37.3	42.2
11	4	2	23.5	24.9
11	5	9	35.1	35.7
11	5	5	57.4	55.6
11	5	2	53.3	51.1
11	5	1	37.2	37.3
11	6	6	29.6	29.2
11	6	2	34.7	36.2
11	6	1	36.3	35.5
11	7	9	41.2	42.4
11	7	8	35.9	37.1
11	7	7	21.4	21.4
11	7	5	55.8	55.1
11	7	4	26.6	27.2
11	7	3	41.9	41.7
11	7	2	30.9	31.4
11	7	1	69.3	66.5
11	8	7	21.0	21.2
11	8	5	33.2	33.6
11	8	3	33.9	28.8
11	8	1	44.0	47.0
11	9	6	55.5	56.4
11	9	2	91.4	90.6
11	10	6	37.3	35.8
11	10	2	48.5	47.1
11	11	7	40.0	35.8
11	11	6	48.7	45.1
11	11	5	53.2	54.0
11	11	2	49.6	50.7
11	11	1	42.3	45.3
11	12	5	26.5	24.9
11	12	2	24.5	26.3
11	13	5	53.1	49.4
11	13	3	36.5	36.7
11	13	1	61.0	58.7
11	15	2	53.7	50.1
10	0	10	44.9	46.8
10	0	8	50.2	54.6
10	0	6	54.0	61.1
10	0	4	83.5	80.2
10	0	0	110.5	110.4
10	1	12	26.9	24.5
10	1	8	31.4	32.7
10	1	4	61.8	65.3
10	1	3	35.9	37.9
10	1	1	45.2	49.6
10	1	0	56.9	56.9
10	2	11	31.7	31.0
10	2	9	32.7	30.0
10	2	7	50.2	50.5
10	2	4	55.2	58.8
10	2	3	72.4	70.2
10	2	1	59.4	57.7
10	2	0	56.9	53.2
10	3	13	27.1	24.6
10	3	9	49.7	49.8
10	3	7	22.7	26.0
10	3	5	38.8	36.6
10	3	4	54.1	54.5
10	3	3	41.6	40.5
10	3	1	72.4	69.9
10	3	0	34.5	34.5
10	4	11	32.1	28.4
10	4	9	21.0	16.8
10	4	7	72.3	67.3
10	4	3	63.5	62.7
10	4	1	57.0	53.5
10	5	12	39.3	37.0
10	5	5	51.5	51.6
10	5	4	102.7	102.5
10	5	3	32.0	32.7
10	5	0	102.0	99.4
10	6	10	34.2	29.2
10	6	7	35.4	35.4
10	6	6	26.3	27.3
10	6	4	53.7	49.5
10	6	3	51.4	51.3
10	6	0	68.0	73.1
10	7	11	35.4	33.4
10	7	10	33.6	30.8
10	7	7	54.2	55.7
10	7	5	37.1	34.5
10	7	3	79.0	76.9
10	7	2	22.9	23.1
10	7	1	61.2	62.6
10	8	10	34.8	33.3
10	8	8	21.0	22.6
10	8	6	35.7	35.3
10	8	4	41.9	41.5
10	8	0	65.2	75.4
10	9	7	35.1	36.1
10	9	6	32.0	31.1
10	9	5	38.3	35.4
10	9	4	81.1	78.1
10	9	3	26.6	26.0
10	9	0	63.7	67.2
10	10	7	39.4	37.3
10	10	4	21.8	22.5
10	10	3	38.6	40.4
10	11	8	39.0	39.3
10	11	7	36.3	35.6
10	11	4	64.2	61.9
10	11	3	50.3	49.0
10	11	1	38.4	41.4
10	11	0	66.9	67.4
10	12	7	35.9	31.8
10	12	1	27.2	26.2
10	13	7	46.6	41.4
10	13	5	36.3	34.5
10	13	3	55.4	59.8
10	13	1	48.2	45.6
10	15	4	42.	41.2
10	15	0	54.7	51.2
9	0	12	61.7	63.1
9	0	10	80.6	78.4
9	0	8	78.6	80.5
9	0	6	173.5	176.4
9	0	4	49.6	49.2
9	0	2	275.4	248.2
9	1	10	46.9	45.1
9	1	9	26.9	27.2
9	1	6	78.1	75.5
9	1	5	43.3	41.5
9	1	3	45.1	46.0
9	1	2	91.4	84.5
9	1	1	42.2	46.2
9	2	13	49.8	48.4
9	2	12	41.0	39.6
9	2	10	33.5	33.5
9	2	9	93.6	91.4
9	2	8	58.5	57.6
9	2	7	40.6	42.1
9	2	6	87.4	86.7
9	2	5	141.0	141.7
9	2	3	100.0	99.4
9	2	2	116.2	124.4
9	2	1	147.9	153.1
9	3	9	41.4	43.0
9	3	7	32.2	32.7
9	3	5	69.3	66.9
9	3	3	63.7	61.1
9	3	1	75.5	77.6
9	4	13	43.7	43.5
9	4	10	36.4	35.2
9	4	9	96.2	95.9
9	4	7	22.0	21.1
9	4	6	50.5	51.8
9	4	5	151.6	146.3
9	4	3	116.2	111.5
9	4	2	85.1	85.3
9	4	1	156.7	153.4
9	5	10	37.3	33.7
9	5	6	65.5	62.1
9	5	2	80.9	77.6
9	5	1	25.2	24.2
9	6	12	44.0	44.4
9	6	11	32.5	28.8
9	6	10	45.4	46.0
9	6	8	60.4	55.9
9	6	7	34.8	33.6
9	6	6	113.4	110.6
9	6	5	22.2	23.6
9	6	4	55.4	57.0
9	6	2	153.5	147.8
9	6	1	41.1	41.6
9	7	8	26.7	21.3
9	7	6	37.1	33.5
9	7	5	30.8	27.1
9	7	3	43.6	43.7
9	7	2	53.4	52.5
9	7	1	35.9	35.2
9	4	12	39.5	39.7
9	8	9	42.7	41.4
9	8	8	57.3	57.1
9	8	7	35.6	36.0
9	8	6	43.2	40.8
9	8	5	62.7	61.6
9	8	4	54.4	52.6
9	8	3	66.4	65.9
9	8	2	87.8	87.2
9	8	1	66.5	67.0
9	9	9	25.8	27.1
9	9	7	30.1	29.5
9	9	5	40.4	37.9
9	9	3	33.1	32.8
9	9	2	20.3	20.6
9	9	1	47.7	48.2
9	10	9	41.6	40.2
9	10	5	74.3	71.6
9	10	3	55.2	56.0
9	10	2	29.6	27.1
9	10	1	65.5	68.4
9	11	6	39.3	36.3
9	11	2	48.0	45.1
9	12	9	22.3	21.3
9	12	6	32.1	29.4
9	12	5	31.1	30.9
9	12	3	25.5	19.9
9	12	2	37.7	37.6
9	13	1	76.0	26.3
8	0	14	34.2	31.8
8	0	12	54.1	52.8
8	0	10	75.7	78.3
8	0	8	145.2	144.6
8	0	6	62.9	62.0
8	0	4	193.1	221.5
8	0	2	61.8	65.9
8	0	0	310.0	315.4
8	1	14	32.2	27.7
8	1	10	50.6	45.3
8	1	8	37.5	34.1
8	1	6	44.0	43.7
8	1	4	63.9	70.3
8	1	0	92.1	87.4
8	2	11	71.8	72.4
8	2	10	45.7	48.9
8	2	9	24.8	21.8
8	2	8	74.4	75.3
8	2	7	149.2	153.4
8	2	6	36.8	35.5
8	2	5	57.8	54.9
8	2	4	133.7	133.0
8	2	3	192.6	188.5
8	2	2	39.9	41.0
8	2	1	159.5	154.2
8	2	0	120.6	116.3
8	3	13	25.9	25.7
8	3	11	28.1	28.7
8	3	7	55.1	56.1
8	3	6	30.7	30.7
8	3	4	26.0	26.4
8	3	3	53.8	52.6
8	3	1	60.8	57.5
8	3	0	38.1	35.9
8	4	12	27.7	27.9
8	4	11	72.0	72.8
8	4	10	21.6	19.2
8	4	8	65.3	65.1
8	4	7	149.4	144.7
8	4	5	73.1	69.8
8	4	4	109.4	110.6
8	4	3	164.9	169.2
8	4	2	41.3	39.4
8	4	1	147.8	139.4
8	4	0	160.0	152.4
8	5	11	25.3	24.4
8	5	10	24.0	24.2
8	5	7	54.0	51.3
8	5	3	77.6	74.7
8	5	1	76.3	74.8
8	6	14	28.9	26.5
8	6	12	36.5	39.1
8	6	10	56.4	57.0
8	6	8	100.7	104.1
8	6	6	39.6	42.0
8	6	4	176.7	171.0
8	6	3	29.3	29.1
8	6	2	34.2	32.4
8	6	0	214.4	222.2
8	7	10	41.9	39.1
8	7	8	32.9	32.6
8	7	6	63.8	64.3
8	7	4	49.6	47.1
8	7	3	33.4	34.7
8	7	2	24.4	25.1
8	7	1	33.7	32.7
8	7	0	62.3	59.2
8	8	11	41.5	41.4
8	8	9	23.3	26.7
8	8	8	37.3	33.2
8	8	7	76.7	74.3
8	8	6	43.0	40.4
8	8	5	67.3	67.3
8	8	4	51.4	45.2
8	8	3	96.1	91.2
8	8	1	94.6	95.3
8	8	0	60.3	64.7
8	9	10	35.6	34.2
8	9	8	25.9	27.2
8	9	6	52.2	51.4
8	9	4	46.9	47.1
8	9	3	27.3	28.6
8	9	1	20.7	23.3
8	9	0	69.7	71.5
8	10	11	34.0	32.4
8	10	9	41.6	40.7
8	10	7	50.7	47.1
8	10	5	39.2	35.9
8	10	4	43.9	41.3
8	10	3	72.5	69.1
8	10	1	37.5	35.5
8	10	0	66.7	67.2
8	11	7	51.0	47.7
8	11	5	22.8	24.4
8	11	3	58.4	59.5
8	11	1	46.1	50.9
8	12	9	24.5	27.2
8	12	5	38.4	35.8
8	12	4	47.4	46.3
8	12	0	79.6	78.8
8	13	7	39.5	37.7
8	13	3	29.3	32.2
8	13	1	31.3	30.4
8	15	6	22.3	20.7
8	15	4	28.5	30.3
8	15	0	43.6	41.7
8	17	0	29.6	25.6
7	0	10	40.4	40.8
7	0	6	99.7	99.5
7	0	2	135.1	151.5
7	1	14	34.6	34.7
7	1	12	43.1	45.0
7	1	11	33.8	33.1
7	1	9	78.7	77.6
7	1	7	37.6	40.2
7	1	4	42.8	39.6
7	1	3	53.3	53.4
7	2	13	35.9	36.0
7	2	9	42.1	41.0
7	2	7	35.4	35.0
7	2	6	36.2	34.5
7	2	5	96.8	100.4
7	2	3	95.0	98.7
7	2	2	78.0	77.2
7	2	1	115.6	110.9
7	3	15	43.5	42.6
7	3	12	28.0	31.0
7	3	11	67.2	65.2
7	3	10	26.9	27.8
7	3	9	27.7	28.6
7	3	8	41.5	41.0
7	3	7	62.5	65.5
7	3	6	76.5	76.6
7	3	3	64.1	63.5
7	3	2	99.7	96.3
7	3	1	33.7	33.9
7	4	10	42.8	47.1
7	4	9	41.9	42.7
7	4	8	27.3	21.9
7	4	6	63.4	64.8
7	4	5	82.8	81.1
7	4	4	33.3	25.2
7	4	3	81.3	77.1
7	4	2	106.2	101.9
7	4	1	64.9	69.4
7	5	15	32.1	33.2
7	5	13	31.9	28.7
7	5	11	36.1	39.4
7	5	10	45.1	47.0
7	5	9	73.3	69.4
7	5	8	29.5	27.6
7	5	7	27.7	25.7
7	5	6	53.4	49.1
7	5	5	92.3	93.9
7	5	3	27.3	25.3
7	5	2	67.8	65.9
7	5	1	84.5	83.5
7	6	10	49.4	47.2
7	6	6	98.6	96.5
7	6	2	145.2	141.9
7	7	13	34.0	35.7
7	7	12	51.8	45.9
7	7	9	54.8	57.8
7	7	8	62.5	59.8
7	7	7	35.5	31.1
7	7	5	117.2	108.9
7	7	4	43.0	42.9
7	7	3	82.5	80.7
7	7	2	45.8	45.4
7	7	1	113.6	109.3
7	8	9	47.4	46.6
7	8	7	19.9	20.2
7	8	5	82.5	79.6
7	8	3	60.0	57.9
7	8	1	39.7	43.6
7	9	12	49.7	41.5
7	9	11	32.4	30.3
7	9	10	47.1	45.1
7	9	8	66.1	61.9
7	9	6	103.9	99.9
7	9	4	49.1	47.8
7	9	3	41.9	39.7
7	9	2	156.4	153.9
7	10	10	35.5	32.3
7	10	6	29.7	26.0
7	10	5	31.2	32.5
7	10	2	54.4	55.3
7	11	12	21.5	19.0
7	11	11	28.7	28.4
7	11	10	38.4	35.8
7	11	9	71.1	67.1
7	11	8	35.7	32.0
7	11	6	75.3	73.5
7	11	5	95.9	89.6
7	11	3	47.7	49.3
7	11	2	91.4	94.6
7	11	1	66.5	71.2
7	12	10	23.1	21.5
7	12	6	35.6	34.7
7	12	5	23.9	23.5
7	12	3	26.5	29.5
7	12	2	42.7	47.4
7	13	12	19.5	20.0
7	13	9	61.6	62.0
7	13	5	103.4	96.6
7	13	3	65.9	70.0
7	13	2	31.2	36.1
7	13	1	92.6	93.2
7	14	5	28.6	26.1
7	15	10	27.7	24.0
7	15	8	35.0	36.4
7	15	6	52.7	56.6
7	15	4	33.1	36.2
7	15	3	21.9	20.7
7	15	2	93.2	91.7
7	15	1	25.4	23.0
7	17	6	29.4	34.1
7	17	5	31.7	33.0
7	17	2	62.9	59.2
7	17	1	39.2	34.6
7	19	3	29.2	24.5
6	0	14	32.7	28.7
6	0	10	32.3	33.1
6	0	8	39.9	39.8
6	0	6	60.8	60.7
6	0	4	101.6	99.1
6	0	0	158.1	156.8
6	1	16	35.6	36.7
6	1	14	31.7	29.3
6	1	12	54.1	53.7
6	1	10	53.6	51.1
6	1	9	48.7	44.7
6	1	8	63.2	63.7
6	1	7	37.5	33.3
6	1	6	74.6	72.4
6	1	5	71.7	69.7
6	1	4	65.5	66.0
6	1	3	31.0	30.5
6	1	1	68.0	65.6
6	1	0	98.7	91.5
6	2	11	35.5	36.7
6	2	10	36.7	34.2
6	2	8	28.8	29.0
6	2	7	42.8	44.1
6	2	6	47.4	46.1
6	2	4	53.7	53.0
6	2	3	85.2	81.2
6	2	2	40.3	38.6
6	2	1	91.5	85.9
6	2	0	153.0	143.9
6	3	13	59.2	55.8
6	3	10	48.6	45.7
6	3	9	90.2	89.0
6	3	8	52.3	49.9
6	3	6	27.2	29.9
6	3	5	106.2	105.1
6	3	4	100.4	97.9
6	3	1	83.1	79.6
6	3	0	64.0	59.3
6	4	13	31.5	30.9
6	4	11	39.9	40.1
6	4	9	27.9	28.1
6	4	7	67.8	66.2
6	4	6	34.2	33.9
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6	4	1	57.1	56.8
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6	5	11	46.6	46.1
6	5	9	91.4	89.6
6	5	7	84.2	81.1
6	5	4	132.5	99.4
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6	5	1	39.1	36.3
6	5	0	124.6	119.9
6	6	10	26.7	26.9
6	6	8	37.9	37.0
6	6	7	63.5	58.0
6	6	6	34.2	34.5
6	6	4	74.5	72.1
6	6	1	66.5	64.7
6	6	0	94.4	87.2
6	7	14	31.2	30.2
6	7	12	22.5	21.8
6	7	11	54.4	53.2
6	7	10	39.8	43.3
6	7	9	41.4	39.9
6	7	7	100.9	98.5
6	7	6	45.6	49.3
6	7	5	67.1	68.6
6	7	4	29.6	29.2
6	7	3	145.0	142.9
6	7	1	178.2	178.1
6	8	14	24.8	26.2
6	8	10	39.5	40.1
6	8	6	22.0	20.4
6	8	4	50.3	49.1
6	8	3	23.7	24.9
6	8	0	55.1	62.2
6	9	14	32.7	33.3
6	9	13	32.8	31.7
6	9	12	24.4	23.7
6	9	10	63.3	59.7
6	9	9	39.1	40.4
6	9	8	81.3	80.7
6	9	7	31.7	32.4
6	9	6	47.6	45.6
6	9	5	75.2	77.9
6	9	4	144.5	141.6
6	9	3	54.8	51.6
6	9	2	29.4	28.5
6	9	1	69.5	66.9
6	9	0	180.5	181.4
6	10	14	21.4	18.8
6	10	11	19.6	21.8
6	10	8	19.4	19.7
6	10	7	44.6	44.5
6	10	[illegible]	38.5	36.5
6	10	[illegible]	33.2	31.2
6	10	3	25.4	27.8
6	10	1	24.1	23.7
6	10	[illegible]	55.8	59.9
6	11	12	29.5	31.3
6	11	11	47.8	46.2
6	11	[illegible]	[illegible]	85.4
6	11	7	92.9	92.3
6	11	[illegible]	[illegible]	27.5
6	11	[illegible]	115.1	111.9
6	11	3	[illegible]	73.9
6	11	1	47.8	51.7
6	11	[illegible]	147.7	142.1
6	12	[illegible]	37.3	34.7
6	12	4	27.2	29.5
6	12	[illegible]	46.4	41.2
6	12	1	[illegible]	33.2
6	13	11	49.2	49.0
6	13	10	23.6	22.9
6	13	7	97.8	95.4
6	13	5	52.6	53.4
6	13	3	97.1	107.6
6	13	1	84.4	86.5
6	13	0	35.1	33.4
6	14	1	24.2	24.0
6	15	8	46.7	44.9
6	15	6	37.1	37.7
6	15	4	70.9	79.8
6	15	3	29.2	27.3
6	15	2	23.2	24.7
6	15	1	25.4	23.0
6	15	0	130.9	126.7
6	16	0	25.5	26.7
6	17	[illegible]	34.5	31.8
6	17	7	31.6	31.7
6	17	4	55.8	58.5
6	17	3	44.4	41.0
6	17	1	34.2	31.1
6	17	0	90.4	71.1
6	18	1	25.2	25.5
6	19	3	50.6	43.6
6	19	1	41.4	36.1
5	0	16	44.1	45.1
5	0	12	53.3	55.1
5	0	10	91.8	97.4
5	0	8	137.5	145.7
5	0	6	220.3	239.4
5	0	4	104.9	104.3
5	0	2	352.6	379.5
5	1	10	62.5	63.5
5	1	9	33.5	37.6
5	1	8	23.8	24.9
5	1	7	34.9	36.4
5	1	6	62.0	67.9
5	1	5	55.8	59.1
5	1	4	31.4	32.3
5	1	3	62.7	66.1
5	1	2	113.7	110.9
5	1	1	50.2	54.6
5	2	14	29.8	30.4
5	2	13	59.9	60.1
5	2	12	51.7	57.4
5	2	11	54.0	57.1
5	2	9	149.9	154.0
5	2	8	55.1	59.1
5	2	7	41.9	44.5
5	2	6	156.4	156.7
5	2	5	215.9	221.1
5	2	4	64.9	65.4
5	2	3	117.2	113.6
5	2	2	202.5	198.1
5	2	1	237.5	267.6
5	3	11	31.5	31.1
5	3	9	45.7	42.7
5	3	8	23.6	25.6
5	3	7	52.1	55.0
5	3	5	60.8	62.1
5	3	3	72.7	69.6
5	3	2	27.6	25.3
5	3	1	67.3	64.9
5	4	11	58.6	62.2
5	4	10	41.0	41.4
5	4	9	171.6	173.6
5	4	7	39.7	43.2
5	4	6	111.4	124.7
5	4	5	264.9	257.3
5	4	3	146.8	143.0
5	4	2	149.5	168.7
5	4	1	271.1	255.2
5	5	14	27.1	23.4
5	5	11	30.2	29.7
5	5	10	57.8	57.8
5	5	[illegible]	[illegible]	96.3
5	5	[illegible]	[illegible]	[illegible]
5	5	2	149.9	141.9
5	5	1	57.0	55.2
5	6	15	24.6	25.5
5	6	12	53.4	55.1
5	6	11	25.0	23.9
5	6	10	76.6	79.0
5	6	9	35.5	34.2
5	6	8	97.1	96.6
5	6	7	31.3	27.8
5	6	6	172.2	174.8
5	6	5	33.5	35.7
5	6	4	38.8	40.1
5	6	2	294.6	289.0
5	7	9	40.5	38.8
5	7	8	23.3	23.9
5	7	7	34.2	31.0
5	7	6	27.5	28.0

H	K	L	F OBS	F CALC
5	7	5	65.4	67.5
5	7	4	45.1	43.1
5	7	3	66.9	65.1
5	7	1	117.3	113.0
5	8	13	33.4	32.5
5	8	12	74.4	45.4
5	8	10	24.1	27.5
5	8	9	55.6	54.7
5	8	8	49.2	51.1
5	8	7	43.7	43.1
5	8	6	81.2	81.5
5	8	5	83.9	82.0
5	8	4	57.6	59.5
5	8	3	73.2	69.1
5	8	2	136.6	133.9
5	8	1	96.1	102.1
5	9	12	24.9	23.9
5	9	11	37.4	33.5
5	9	10	76.1	26.3
5	9	9	29.0	29.9
5	9	8	33.4	29.5
5	9	7	44.4	43.7
5	9	6	57.7	57.4
5	9	5	25.7	30.5
5	9	4	34.3	30.1
5	9	3	41.6	40.6
5	9	2	89.1	89.2
5	9	1	49.9	59.1
5	10	11	22.0	25.1
5	10	9	71.7	70.3
5	10	8	43.7	42.2
5	10	6	29.4	23.7
5	10	5	79.7	77.6
5	10	3	37.8	42.3
5	10	2	25.9	25.7
5	10	1	84.3	90.7
5	11	9	32.1	31.3
5	11	6	67.4	64.7
5	11	5	35.9	35.3
5	11	3	30.0	29.5
5	11	2	82.2	90.3
5	11	1	39.4	41.5
5	12	11	45.7	44.2
5	12	9	43.2	40.9
5	12	7	31.1	33.8
5	12	6	42.0	39.4
5	12	5	44.9	44.6
5	12	2	52.1	59.2
5	12	1	24.5	26.7
5	13	9	44.4	44.2
5	13	7	23.6	22.6
5	13	5	54.5	51.9
5	13	3	27.0	30.1
5	13	1	52.5	52.4
5	14	12	21.0	19.4
5	14	8	32.4	31.1
5	14	7	32.7	32.7
5	14	6	29.4	27.5
5	14	4	20.5	18.2
5	14	2	45.4	44.7
5	15	6	44.3	39.6
5	15	3	21.0	19.7
5	15	2	63.0	58.3
5	16	8	30.3	26.9
5	16	4	19.5	20.1
5	17	2	41.3	36.0
5	19	1	26.8	21.6
5	20	1	22.0	16.1
4	0	14	89.8	89.2
4	0	12	87.5	90.2
4	0	10	117.3	117.3
4	0	8	216.4	215.7
4	0	6	162.0	159.7
4	0	4	464.9	472.8
4	0	2	52.2	52.0
4	0	0	475.5	472.7
4	1	14	35.6	32.5
4	1	10	35.0	33.2
4	1	4	53.1	43.1
4	1	0	183.4	168.8
4	2	15	45.3	44.0
4	2	14	51.2	49.6
4	2	12	24.6	24.8
4	2	11	127.4	127.9
4	2	10	88.3	89.2
4	2	9	57.4	55.4
4	2	8	93.4	95.0
4	2	7	236.6	239.1
4	2	6	132.0	129.4
4	2	5	143.7	142.3
4	2	4	183.6	176.7
4	2	3	321.7	313.0
4	2	2	53.5	49.8
4	2	1	334.2	306.4
4	2	0	237.6	215.5
4	3	11	28.8	27.9
4	3	7	83.7	81.0
4	3	6	34.2	32.0
4	3	4	19.2	18.5
4	3	1	86.8	81.5
4	3	0	71.4	70.9
4	4	17	21.6	21.1
4	4	15	41.8	39.7
4	4	13	35.2	34.1
4	4	12	55.9	56.1
4	4	11	130.4	128.7
4	4	8	89.6	90.5
4	4	7	209.8	211.0
4	4	6	27.5	26.9
4	4	5	158.4	151.9
4	4	4	173.8	172.7
4	4	3	344.1	332.0
4	4	2	35.8	32.9
4	4	1	308.8	294.0
4	4	0	219.9	225.0
4	5	11	26.3	26.4
4	5	7	46.5	44.5
4	5	5	48.9	48.4
4	5	4	46.8	42.2
4	5	3	40.7	42.3
4	5	1	92.9	93.0
4	6	14	53.3	48.9
4	6	13	40.7	40.1
4	6	12	61.2	62.5
4	6	10	69.8	69.4
4	6	9	34.0	32.2
4	6	8	162.9	162.0
4	6	6	139.5	134.8
4	6	5	59.2	57.8
4	6	4	343.8	329.9
4	6	3	39.3	39.6
4	6	1	75.7	72.4
4	6	0	268.7	295.7
4	7	8	41.2	39.8
4	7	4	39.9	40.3
4	7	0	97.8	104.4
4	8	14	32.3	34.0
4	8	11	63.6	64.4
4	8	10	71.7	73.7
4	8	9	52.2	50.5
4	8	8	41.3	41.7
4	8	7	101.1	99.5
4	8	6	97.2	92.9
4	8	5	90.0	87.1
4	8	4	123.3	121.0
4	8	3	169.0	162.4
4	8	2	49.2	46.5
4	8	1	137.4	144.3
4	8	0	132.5	140.0
4	9	14	19.3	19.5
4	9	6	27.9	27.2
4	9	5	21.6	23.2
4	9	4	23.2	24.7
4	9	1	30.1	33.4
4	9	0	51.4	51.5
4	10	13	28.0	27.8
4	10	12	43.1	44.7
4	10	11	48.1	50.2
4	10	10	24.4	23.7
4	10	8	29.8	30.0
4	10	7	81.7	81.1
4	10	5	25.4	27.0
4	10	4	81.5	82.6
4	10	3	120.3	119.1
4	10	1	111.2	118.2
4	10	0	48.6	46.8
4	11	11	25.1	25.5
4	11	7	36.5	34.7
4	11	3	46.7	50.7
4	11	1	24.4	27.7
4	11	0	52.2	53.7
4	12	9	45.6	45.2
4	12	8	46.2	45.9
4	12	7	38.3	40.2
4	12	6	27.0	27.9
4	12	4	92.5	93.0
4	12	3	23.5	20.0
4	12	0	67.9	58.1
4	13	7	33.5	35.6
4	13	4	22.1	20.8
4	14	13	24.6	22.9
4	14	10	30.4	30.1
4	14	9	35.7	35.4
4	14	6	29.4	26.9
4	14	5	42.2	41.0
4	14	1	35.2	32.9
4	14	0	41.9	39.6
4	15	4	31.1	34.1
4	15	0	31.7	29.1
4	17	0	21.0	21.5
3	0	16	27.5	25.6
3	0	14	35.4	33.6
3	0	12	48.5	50.8
3	0	10	90.7	86.0
3	0	8	123.2	124.7
3	0	6	248.6	244.3
3	0	4	34.4	40.4
3	0	2	292.3	297.9
3	1	16	33.9	32.6
3	1	15	30.1	30.4
3	1	14	42.1	42.5
3	1	12	53.0	51.4
3	1	8	97.3	98.6
3	1	7	67.4	68.0
3	1	6	59.2	57.8
3	1	5	21.4	19.2
3	1	4	133.7	131.7
3	1	3	94.9	90.6
3	1	2	94.2	90.4
3	1	1	78.3	69.6
3	2	13	61.5	60.0
3	2	12	30.6	30.2
3	2	10	47.6	44.6
3	2	9	124.5	125.9
3	2	8	51.7	50.8
3	2	7	92.7	90.7
3	2	6	148.1	149.2
3	2	5	136.9	140.7
3	2	4	28.5	25.2
3	2	3	155.5	153.5
3	2	2	162.8	158.4
3	2	1	377.2	351.7
3	3	15	53.6	55.9
3	3	12	27.2	29.3
3	3	11	58.1	58.3
3	3	10	37.8	36.4
3	3	8	65.4	67.6
3	3	7	67.1	69.7
3	3	6	76.1	76.2
3	3	5	55.9	52.6
3	3	4	105.0	103.5
3	3	2	120.0	115.3
3	3	1	47.1	41.6
3	4	14	27.6	27.8
3	4	13	41.4	41.6
3	4	10	51.1	49.6
3	4	9	98.3	94.8
3	4	8	33.7	33.7
3	4	7	54.1	51.7
3	4	6	133.0	133.4
3	4	5	134.7	132.4
3	4	4	49.4	45.9
3	4	3	84.6	84.4
3	4	2	88.7	89.1
3	4	1	192.7	188.0
3	5	15	42.2	42.6
3	5	14	36.6	37.4
3	5	11	42.9	44.6
3	5	10	54.6	51.9
3	5	9	96.7	95.8
3	5	7	34.2	34.1
3	5	6	67.8	66.6
3	5	5	153.2	153.4
3	5	3	72.3	62.8
3	5	2	121.7	115.2
3	5	1	99.1	96.4
3	6	12	32.4	34.5
3	6	10	69.9	65.1
3	6	8	74.4	73.2
3	6	7	39.9	39.0
3	6	6	135.4	135.3
3	6	5	40.4	37.8
3	6	2	226.0	219.0
3	6	1	28.1	25.2
3	7	13	24.9	24.0
3	7	12	56.6	54.6
3	7	9	103.8	101.5
3	7	8	91.1	87.6
3	7	7	69.1	67.7
3	7	5	129.8	131.1
3	7	4	111.9	111.3
3	7	3	72.3	90.2
3	7	2	52.9	55.1
3	7	1	168.5	173.5
3	8	13	31.1	27.0
3	8	12	36.2	39.4
3	8	9	73.6	71.3
3	8	8	33.3	37.1
3	8	7	69.2	66.0
3	8	6	35.1	39.4
3	8	5	103.6	94.3
3	8	4	61.4	59.7
3	8	3	86.5	86.7
3	8	2	57.9	54.7
3	8	1	114.7	125.5
3	9	12	49.6	47.1
3	9	11	35.6	37.4
3	9	10	56.5	57.6
3	9	9	29.1	30.0
3	9	8	85.4	86.4
3	9	7	69.0	68.2
3	9	6	136.3	130.1
3	9	4	80.8	77.9
3	9	2	185.7	198.3
3	10	10	24.7	23.1
3	10	9	60.9	61.6
3	10	7	20.1	23.0
3	10	6	53.8	52.1
3	10	5	81.6	75.1
3	10	3	48.8	49.9
3	10	2	58.5	61.3
3	10	1	92.3	87.4
3	11	15	25.7	29.6
3	11	13	32.6	29.7
3	11	11	36.1	35.4
3	11	10	52.0	50.1
3	11	9	74.6	74.8
3	11	8	37.9	39.1
3	11	6	100.9	101.3
3	11	5	101.0	99.4
3	11	3	49.1	49.9
3	11	2	102.6	113.8
3	11	1	110.6	115.2
3	12	11	30.8	29.2
3	12	6	44.5	45.7
3	12	2	70.8	77.4
3	13	13	42.9	34.7
3	13	9	91.6	82.1
3	13	8	40.1	39.9
3	13	7	29.2	27.3
3	13	6	23.0	21.1
3	13	5	107.4	105.4
3	13	3	54.6	64.1
3	13	1	127.4	125.5
3	15	12	33.0	32.7
3	15	10	31.9	31.5
3	15	8	51.5	48.3
3	15	6	74.9	77.6
3	15	5	21.9	24.0
3	15	4	20.2	22.7
3	15	2	113.9	111.8
3	15	1	26.4	24.9
3	17	6	44.1	44.1
3	17	5	29.9	31.9
3	17	2	72.0	63.8
3	17	1	47.8	46.3
3	19	5	42.4	46.1
3	19	3	36.4	34.4
3	19	1	53.1	49.4
3	21	2	31.5	29.8
3	21	1	24.4	22.1
2	0	14	24.7	26.6
2	0	6	39.5	34.8
2	0	4	134.3	127.0
2	0	2	79.5	81.2
2	0	0	115.2	115.6
2	1	16	40.2	41.2
2	1	14	55.7	52.7
2	1	13	38.5	38.3
2	1	12	55.3	56.9
2	1	10	51.1	60.9
2	1	9	52.0	54.8
2	1	8	75.7	74.9
2	1	6	82.5	85.3
2	1	5	40.6	44.7
2	1	4	98.2	99.4
2	1	3	95.2	92.5
2	1	1	64.4	59.7
2	1	0	44.6	42.3
2	2	10	30.8	29.7
2	2	8	13.2	30.4
2	2	5	96.1	91.3
2	2	4	19.6	20.8
2	2	2	90.5	85.0
2	2	1	24.1	21.7
2	3	17	40.9	42.6
2	3	14	33.0	32.4
2	3	13	96.1	96.8
2	3	11	18.7	20.6
2	3	10	41.2	42.2
2	3	9	143.0	141.3
2	3	8	42.4	40.5
2	3	6	53.5	57.3
2	3	5	133.1	125.3
2	3	4	149.0	141.7
2	3	3	157.2	144.7
2	3	2	87.2	89.7
2	3	1	63.0	61.4
2	3	0	209.6	216.2
2	4	8	36.7	36.3
2	4	7	52.4	51.4
2	4	5	41.9	39.6
2	4	1	142.9	134.3
2	4	0	69.8	76.6
2	5	17	24.7	30.7
2	5	16	33.1	34.2
2	5	13	63.6	61.3
2	5	12	57.6	62.5
2	5	11	61.7	61.9
2	5	10	26.7	30.2
2	5	9	53.7	52.7
2	5	8	98.0	99.4
2	5	7	107.7	107.5
2	5	4	190.0	179.3
2	5	3	146.4	140.2
2	5	2	30.4	30.6
2	5	1	135.7	130.3
2	5	0	255.8	250.9
2	6	10	25.7	26.6
2	6	4	42.6	39.0
2	6	3	31.7	30.0
2	6	1	75.3	73.2
2	6	0	72.9	75.6
2	7	15	33.6	36.5
2	7	14	48.4	50.9
2	7	12	40.3	40.2
2	7	11	87.2	91.3
2	7	10	80.0	79.1
2	7	9	32.7	33.1
2	7	7	149.1	147.5
2	7	6	102.6	102.3
2	7	5	89.1	92.6
2	7	3	219.2	218.4
2	7	1	183.9	199.6
2	7	0	56.0	66.5
2	8	6	23.2	21.7
2	8	3	24.7	23.3
2	8	2	29.5	27.5
2	8	0	34.6	38.9
2	9	14	56.9	59.6
2	9	13	33.2	29.7
2	9	10	85.0	89.4
2	9	9	48.0	48.0
2	9	8	111.1	112.5
2	9	6	107.7	109.0
2	9	5	91.7	91.1
2	9	4	194.4	194.8
2	9	1	65.6	73.1
2	9	0	262.2	493.5
2	10	0	48.7	48.4
2	11	13	35.3	32.1
2	11	12	44.4	43.8
2	11	11	56.0	58.7
2	11	10	25.8	26.6
2	11	8	93.2	96.5
2	11	7	108.0	109.2
2	11	6	32.9	29.6
2	11	5	36.3	34.8
2	11	4	153.8	152.1
2	11	3	132.0	141.9
2	11	1	98.5	101.6
2	11	0	184.0	190.3
2	12	1	31.4	32.7
2	13	11	61.8	62.9
2	13	10	35.7	35.9
2	13	8	21.0	19.9
2	13	7	125.2	125.5
2	13	6	35.4	33.3
2	13	5	61.4	65.5
2	13	4	29.6	32.6
2	13	3	121.9	142.0
2	13	1	152.4	150.4
2	13	0	25.0	31.0
2	14	2	16.7	15.7
2	15	13	52.7	51.8
2	15	9	63.7	62.5
2	15	7	32.1	34.4
2	15	6	61.7	60.9
2	15	4	96.3	109.8
2	15	3	39.9	41.2
2	15	2	29.4	38.7
2	15	1	59.9	56.4
2	15	0	130.1	124.4
2	17	8	42.7	44.4
2	17	7	43.1	43.2
2	17	6	24.3	30.5
2	17	4	86.2	95.1
2	17	3	59.9	57.7
2	17	1	53.8	48.3
2	17	0	105.2	94.4
2	19	7	43.9	50.4
2	19	3	61.1	57.4
2	19	1	48.9	46.3
2	21	4	37.4	36.5
2	21	3	29.7	25.2
2	21	0	41.1	31.2
1	0	16	50.7	47.9
1	0	12	83.0	78.6
1	0	10	156.2	148.7
1	0	8	135.9	133.7
1	0	6	274.5	264.2
1	0	4	257.8	252.9
1	0	2	411.0	419.1
1	1	15	22.2	21.7
1	1	14	37.0	37.6
1	1	12	25.0	24.9
1	1	11	43.3	41.7
1	1	10	51.3	50.2
1	1	8	54.0	56.0
1	1	7	82.0	80.1
1	1	4	37.7	37.8
1	1	3	192.2	184.3
1	1	2	79.8	78.5
1	2	16	24.2	23.6
1	2	15	24.9	24.2
1	2	13	70.0	70.8
1	2	12	42.4	42.2
1	2	10	99.1	85.7
1	2	9	164.9	161.7
1	2	8	81.6	80.1
1	2	7	68.7	65.5
1	2	6	156.5	147.5
1	2	5	319.9	309.3
1	2	4	85.4	82.9
1	2	3	230.7	220.2
1	2	2	304.0	293.9
1	2	1	327.7	312.3
1	3	15	34.0	36.7
1	3	11	65.5	68.5
1	3	10	43.1	41.1
1	3	9	29.2	28.1
1	3	7	94.4	91.7
1	3	6	65.6	64.1
1	3	4	21.1	22.7
1	3	3	181.4	170.2
1	3	2	42.6	42.9
1	3	1	19.2	17.7
1	4	15	32.9	33.8
1	4	14	19.9	23.2
1	4	13	50.4	50.1
1	4	12	21.6	21.1
1	4	10	64.5	62.8
1	4	9	174.2	171.1
1	4	8	34.5	31.8
1	4	7	26.7	27.6
1	4	6	129.9	125.6
1	4	5	296.9	284.7
1	4	4	39.1	40.1
1	4	3	227.4	216.2
1	4	2	198.0	188.9
1	4	1	164.6	166.6
1	5	15	33.0	34.2
1	5	14	40.9	41.4
1	5	13	21.1	22.0
1	5	11	41.8	41.3
1	5	10	93.5	93.7
1	5	9	66.3	66.7
1	5	7	46.7	45.3
1	5	6	123.2	124.3
1	5	5	78.1	75.2
1	5	4	35.8	29.6
1	5	2	159.9	182.7
1	6	16	26.5	27.1
1	6	12	69.1	69.9
1	6	10	53.6	86.1
1	6	9	46.1	45.4
1	6	8	77.7	79.3
1	6	7	31.8	30.3
1	6	6	200.2	198.4
1	6	5	91.7	86.8
1	6	4	97.6	95.7
1	6	3	34.9	33.2
1	6	2	264.5	271.3
1	7	16	26.4	23.6
1	7	13	41.2	40.9
1	7	12	44.4	44.8
1	7	9	53.4	81.6
1	7	8	48.9	53.6
1	7	7	33.9	35.9
1	7	5	137.1	138.3
1	7	4	55.7	54.8
1	7	3	112.3	110.0
1	7	2	34.3	37.1
1	7	1	99.8	113.6
1	8	13	37.9	39.3
1	8	12	44.2	46.6
1	8	10	36.2	37.2
1	8	9	48.9	52.0
1	8	8	77.4	77.3
1	8	7	34.2	35.9
1	8	6	52.3	50.4
1	8	5	127.9	124.0
1	8	4	56.6	56.1
1	8	3	126.6	127.4
1	8	2	161.6	167.7
1	8	1	106.7	125.2
1	9	17	41.1	45.6
1	9	11	33.0	32.7
1	9	10	48.6	52.1
1	9	8	61.4	61.6
1	9	7	42.5	41.7
1	9	6	111.4	112.9
1	9	5	39.2	37.5
1	9	4	49.2	48.4
1	9	3	38.3	36.6
1	9	2	156.3	168.1
1	9	1	26.9	28.0
1	10	13	31.1	33.4
1	10	10	31.5	30.6
1	10	9	50.6	52.4
1	10	8	37.7	40.1
1	10	5	121.9	120.0
1	10	4	36.3	34.7
1	10	3	79.6	80.4
1	10	2	66.0	74.9
1	10	1	102.2	111.5
1	11	10	33.5	34.4
1	11	9	63.8	66.6
1	11	8	32.0	30.5
1	11	6	81.9	79.7
1	11	5	95.4	95.3
1	11	3	48.0	50.8
1	11	2	93.3	111.3
1	11	1	73.9	76.8
1	12	15	26.2	30.6
1	12	7	35.3	37.7
1	12	6	53.4	47.7
1	12	5	21.5	22.4
1	12	2	75.4	77.2
1	12	1	29.7	28.0
1	13	13	26.1	28.8
1	13	9	65.0	64.0
1	13	8	20.0	19.8
1	13	5	107.0	110.5
1	13	3	55.7	64.4
1	13	1	157.6	107.0
1	14	12	25.7	22.3
1	14	8	32.2	33.6
1	14	7	21.2	21.7
1	14	2	24.3	25.9
1	15	9	23.1	19.7
1	15	8	37.5	38.3
1	15	6	63.7	68.1
1	15	5	30.7	31.7
1	15	4	27.0	28.9
1	15	2	118.9	119.1
1	15	1	42.4	41.6
1	16	8	32.8	32.6
1	17	8	27.0	25.1
1	17	6	32.1	36.7
1	17	5	37.1	42.7
1	17	2	55.9	56.0
1	17	1	35.5	34.2
1	19	5	43.8	42.7
1	19	1	39.5	36.2
0	0	16	31.8	32.5
0	0	14	106.0	102.5
0	0	12	81.7	79.1
0	0	10	143.2	140.6
0	0	8	333.5	314.2
0	0	6	134.1	127.6
0	0	4	520.0	508.2
0	0	2	62.8	69.5
0	2	16	20.3	19.2
0	2	15	58.3	58.6
0	2	14	59.5	56.6
0	2	12	20.8	18.6
0	2	11	153.8	147.2
0	2	10	87.7	83.9
0	2	8	111.0	109.4
0	2	7	345.7	337.7
0	2	6	48.0	48.9
0	2	5	208.9	198.1
0	2	4	231.8	225.3
0	2	3	492.2	471.8
0	2	2	82.4	80.1
0	2	1	300.6	301.0
0	2	0	263.7	349.9
0	4	15	57.9	60.1
0	4	14	19.9	20.7
0	4	13	45.5	45.9
0	4	12	29.8	27.3
0	4	11	138.4	137.9
0	4	10	49.3	47.8
0	4	9	138.7	134.9
0	4	7	333.7	327.5
0	4	6	33.8	32.0
0	4	5	139.4	141.5
0	4	4	126.4	127.1
0	4	3	256.6	256.8
0	4	2	106.0	102.1
0	4	1	361.6	361.6
0	4	0	263.2	296.0
0	6	14	44.1	46.8
0	6	13	65.6	66.1
0	6	12	50.0	52.7
0	6	11	24.4	25.9
0	6	10	113.1	111.5
0	6	9	52.6	50.4
0	6	8	198.9	195.9
0	6	6	59.6	61.2
0	6	5	54.6	52.5
0	6	4	322.7	316.9
0	6	2	51.4	49.9
0	6	1	78.6	79.3
0	6	0	446.5	455.3
0	8	15	25.2	25.9
0	8	14	51.8	57.9
0	8	13	35.6	33.4
0	8	11	61.5	62.3
0	8	10	83.8	85.8
0	8	9	55.2	56.4
0	8	8	78.2	77.0
0	8	7	123.0	121.5
0	8	6	97.5	97.9
0	8	5	113.3	115.2
0	8	4	146.2	149.6
0	8	3	198.7	204.5
0	8	2	36.3	37.9
0	8	1	135.8	140.3
0	8	0	202.4	205.4
0	10	15	21.3	26.0
0	10	12	45.1	45.3
0	10	11	62.8	65.9
0	10	9	78.3	83.5
0	10	7	119.2	120.6
0	10	5	28.4	29.5
0	10	4	70.1	65.8
0	10	3	150.1	154.8
0	10	2	39.0	37.5
0	10	1	115.6	121.7
0	10	0	63.0	62.9
0	12	13	33.0	33.4
0	12	10	28.8	26.6
0	12	9	34.1	35.3
0	12	8	67.2	65.0
0	12	7	44.4	49.7
0	12	5	31.0	32.0
0	12	4	90.9	92.2
0	12	3	27.5	30.3
0	12	2	79.4	75.0
0	14	13	33.1	33.0
0	14	8	26.9	27.5
0	14	5	45.0	45.6
0	14	4	33.7	33.9
0	14	1	25.0	25.9
0	14	0	24.5	22.3
0	16	13	27.5	30.7
0	16	5	41.1	47.1
0	16	1	23.0	21.9
0	16	0	34.2	34.8
0	19	3	27.0	27.3
0	22	1	20.1	20.6

TABLE 11

π-Cyclopentadienyl-trans-dicarbonyliodo(phenyl isocyanide)molybdenum; fractional atomic coordinates with esd in parentheses and thermal parameters.

Atom	x	y	z	U_{iso}($x10^3 Å^2$)
Mo	0.64036(6)	0.18578(4)	0.39509(5)	*
I	0.87306(5)	0.15570(4)	0.37895(5)	*
C(1)	0.5325(8)	0.2712(5)	0.3608(6)	*
N(2)	0.4737(8)	0.3209(5)	0.3396(7)	*
C(3)	0.4022(8)	0.3832(5)	0.3234(7)	*
C(4)	0.3230(9)	0.3760(6)	0.2541(8)	*
C(5)	0.2518(10)	0.4388(8)	0.2399(10)	*
C(6)	0.2561(11)	0.5023(8)	0.2931(10)	*
C(7)	0.3385(14)	0.5115(6)	0.3588(9)	*
C(8)	0.4110(10)	0.4493(7)	0.3768(7)	*
C(9)	0.5530(10)	0.0631(6)	0.3907(8)	*
C(10)	0.6376(12)	0.0575(7)	0.4557(9)	*
C(11)	0.6191(10)	0.1120(7)	0.5267(8)	*
C(12)	0.5148(11)	0.1492(6)	0.5056(8)	*
C(13)	0.4774(9)	0.1209(6)	0.4209(9)	*
C(14)	0.7052(8)	0.2794(6)	0.4518(6)	*
O(15)	0.7367(7)	0.3360(5)	0.4847(6)	*
C(16)	0.6678(8)	0.1989(5)	0.2612(7)	*
O(17)	0.6788(7)	0.2077(5)	0.1827(5)	*
H(4)	0.312	0.332	0.202	66
H(5)	0.186	0.433	0.184	77
H(6)	0.200	0.546	0.276	85
H(7)	0.360	0.574	0.387	87
H(8)	0.483	0.463	0.400	65
H(9)	0.530	0.035	0.325	69
H(10)	0.705	0.015	0.439	77
H(11)	0.662	0.110	0.588	68
H(12)	0.482	0.187	0.541	70
H(13)	0.420	0.143	0.373	68

* These atoms were assigned anisotropic temperature factors of the form:

$$T = \exp\left[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}\ell^2c^{*2} + 2U_{23}k\ell b^*c^* + 2U_{31}\ell hc^*a^* + 2U_{12}hka^*b^*)\right]$$

with final parameters (U_{ij}x10^4Å^2):

Atom	U_{11}	U_{22}	U_{33}	$2U_{23}$	$2U_{31}$	$2U_{12}$
Mo	383	473	447	-28	1	56
I	409	748	778	-121	12	59
C(1)	460	543	580	-33	27	194
N(2)	579	653	871	81	15	250
C(3)	416	527	695	215	95	50
C(4)	547	597	812	138	-201	-191
C(5)	632	856	967	752	-176	239
C(6)	741	694	1075	539	709	671
C(7)	1240	472	852	7	632	166
C(8)	771	639	590	-64	243	127
C(9)	760	530	760	82	305	-199
C(10)	893	559	955	473	483	301
C(11)	675	661	775	483	363	303
C(12)	825	670	606	77	650	-181
C(13)	503	622	862	178	169	-175
C(14)	470	562	593	-156	45	-75
O(15)	756	837	788	-410	-5	-200
C(16)	557	581	540	-282	-115	-23
O(17)	956	979	453	-63	-66	-115

<u>Average estimated standard deviations</u> (U_{ij}x10^4Å^2)

Atom	U_{11}	U_{22}	U_{33}	$2U_{23}$	$2U_{31}$	$2U_{12}$
Mo	4	4	4	6	6	6
I	3	5	5	6	6	5
C	67	62	70	109	111	103
N	52	51	63	98	92	90
O	59	64	48	81	79	88

TABLE 12

π-Cyclopentadienyl-trans-dicarbonyliodo(phenyl isocyanide)molybdenum; interatomic bond lengths (Å) with esd in parentheses.

Mo	- I	2.858(1)	N(2)	- C(3)	1.397(13)
Mo	- C(9)	2.363(11)	C(3)	- C(4)	1.392(15)
Mo	- C(10)	2.382(12)	C(4)	- C(5)	1.395(17)
Mo	- C(11)	2.309(11)	C(5)	- C(6)	1.342(20)
Mo	- C(12)	2.293(12)	C(6)	- C(7)	1.385(20)
Mo	- C(13)	2.289(11)	C(7)	- C(8)	1.409(17)
Mo	- C(14)	1.973(10)	C(8)	- C(3)	1.381(15)
C(14)	- O(15)	1.150(12)	C(9)	- C(10)	1.390(18)
Mo	- C(16)	1.983(10)	C(10)	- C(11)	1.412(17)
C(16)	- O(17)	1.158(11)	C(11)	- C(12)	1.443(17)
Mo	- C(1)	2.025(10)	C(12)	- C(13)	1.396(17)
C(1)	- N(2)	1.153(13)	C(13)	- C(9)	1.418(15)

The C - H distances range from 0.92 - 1.17, mean 1.06 Å.

TABLE 13

π-Cyclopentadienyl-trans-dicarbonyliodo(phenyl isocyanide)molybdenum; valency angles (°) with esd in parentheses.

I - Mo - C(1)	137.7(3)	C(16) - Mo - C(10)	118.0(4)
I - Mo - C(14)	78.2(3)	C(16) - Mo - C(11)	153.0(4)
I - Mo - C(16)	77.1(3)	C(16) - Mo - C(12)	145.7(4)
C(1) - Mo - C(14)	76.2(4)	C(16) - Mo - C(13)	111.0(4)
C(1) - Mo - C(16)	77.5(4)	Mo - C(14) - O(15)	175.9(9)
C(14) - Mo - C(16)	104.4(4)	Mo - C(16) - O(17)	176.8(9)
I - Mo - C(9)	105.7(3)	Mo - C(1) - N(2)	177.8(9)
I - Mo - C(10)	82.8(3)	C(1) - N(2) - C(3)	174.2(11)
I - Mo - C(11)	94.4(3)	N(2) - C(3) - C(4)	118.3(9)
I - Mo - C(12)	130.8(3)	N(2) - C(3) - C(8)	119.6(9)
I - Mo - C(13)	139.9(3)	C(8) - C(3) - C(4)	122.1(9)
C(1) - Mo - C(9)	111.0(4)	C(3) - C(4) - C(5)	117.2(10)
C(1) - Mo - C(10)	139.2(4)	C(4) - C(5) - C(6)	121.7(12)
C(1) - Mo - C(11)	122.2(4)	C(5) - C(6) - C(7)	121.2(12)
C(1) - Mo - C(12)	87.1(4)	C(6) - C(7) - C(8)	118.9(11)
C(1) - Mo - C(13)	81.2(4)	C(7) - C(8) - C(3)	118.5(10)
C(14) - Mo - C(9)	156.9(4)	C(13) - C(9) - C(10)	108.0(10)
C(14) - Mo - C(10)	127.8(4)	C(9) - C(10) - C(11)	109.4(11)
C(14) - Mo - C(11)	98.7(4)	C(10) - C(11) - C(12)	106.2(10)
C(14) - Mo - C(12)	101.2(4)	C(11) - C(12) - C(13)	108.1(10)
C(14) - Mo - C(13)	132.1(4)	C(12) - C(13) - C(9)	108.1(10)
C(16) - Mo - C(9)	98.6(4)		

The phenyl C - C - H angles range from 111 - 131, mean 119°.

The cyclopentadienyl C - C - H angles range from 115 - 137, mean 125°.

TABLE 14

π-Cyclopentadienyl-trans-dicarbonyliodo(phenyl isocyanide)molybdenum; displacements (Å) of atoms from various planes.

	Atoms in plane	Displacements	Atoms out of plane	Displacements
a)	C(3)	-0.008	Mo	-0.201
	C(4)	0.004	C(1)	-0.123
	C(5)	0.017	N(2)	-0.035
	C(6)	-0.033	H(4)	0.12
	C(7)	0.029	H(5)	0.01
	C(8)	-0.008	H(6)	-0.01
			H(7)	0.34
			H(8)	0.41
b)	C(9)	0.000	H(9)	-0.16
	C(10)	-0.014	H(10)	0.01
	C(11)	0.022	H(11)	-0.18
	C(12)	-0.022	H(12)	-0.02
	C(13)	0.014	H(13)	0.27
			Mo	1.991

TABLE 15

π-Cyclopentadienyl-trans-dicarbonyliodo(phenyl isocyanide)molybdenum; intermolecular contacts ($\leqslant 3.75$Å).

C(13)	---	$O(15^{I})$	3.29	C(6)	---	$C(11^{I})$	3.67
C(12)	---	$O(15^{I})$	3.36	C(7)	---	$C(16^{III})$	3.67
C(14)	---	$O(17^{II})$	3.37	C(6)	---	$C(9^{III})$	3.67
C(7)	---	$O(17^{III})$	3.44	C(4)	---	$O(15^{IV})$	3.68
C(4)	---	$O(17^{IV})$	3.51	C(4)	---	$C(14^{IV})$	3.70
C(4)	---	$C(16^{IV})$	3.59	C(5)	---	$C(9^{III})$	3.70
C(7)	---	$O(15^{V})$	3.59	C(5)	---	$O(15^{IV})$	3.72
C(6)	---	$C(16^{III})$	3.60	C(5)	---	$C(10^{III})$	3.74
C(6)	---	$O(17^{III})$	3.65				

The superscripts refer to the following transformations of the atomic coordinates:

I	$-\frac{1}{2} + x$,	$\frac{1}{2} - y$,	$1 - z$;	IV	$-\frac{1}{2} + x$,	y,	$\frac{1}{2} - z$;
II	x,	$\frac{1}{2} - y$,	$\frac{1}{2} + z$;	V	$1 - x$,	$1 - y$,	$1 - z$.
III	$-\frac{1}{2} + x$,	y,	$\frac{1}{2} - z$;				

II.2.4 DISCUSSION

These analyses have confirmed the spectroscopic assignment of $(\pi\text{-}C_5H_5)Mo(CO)_2(PPh_3)Br$, A, as the cis-isomer and demonstrated that the crystal selected from a mixture of isomers of $(\pi\text{-}C_5H_5)Mo(CO)_2(CNPh)I$, B, was that of the trans-isomer.

Regarding the cyclopentadienyl ligand as a formally tridentate six-electron donor, $C_5H_5^-$, then both A and B can be considered as seven-coordinate d^4 Mo^{II} complexes with distorted square pyramidal shape; the $\pi\text{-}C_5H_5$ group is at the apex of the pyramid, the four monodentate ligands are at the corners of the base and the molybdenum atom is above the base. An examination of the X-ray analyses of related compounds(1 - 6) suggests that the preferred conformation in their crystals of complexes of this type has a molybdenum - carbonyl bond passing over the midpoint of a carbon - carbon bond of the cyclopentadienyl ring, and this tendency is indeed observed in both A and B (see Figures 1 and 3). The slight difference between the orientation of the monodentate ligands with respect to the cyclopentadienyl ring in A1 and A101 could be a manifestation of the different extent of disorder in these molecules or merely an artifact of crystal packing.

The valency angles about the molybdenum atom in B and any meaningful angles obtainable from the disordered structure of A are similar to those found in related complexes. The I - Mo - CNPh and OC - Mo - CO angles of 137.7 and 104.4° in B compare favourably with the I - Mo - P and OC - Mo - CO angles of 141 and 105° respectively in trans-$(\pi\text{-}C_5H_5)Mo(CO)_2(PPh_3)I$(2), 138 and 107° in trans-$(\pi\text{-}CH_3C_5H_4)Mo(CO)_2[P(OMe)_3]I$(4) and 136 and 109° in trans-$(\pi\text{-}C_5H_5)Mo(CO)_2[P(OMe)_3]I$(4). Other OC - Mo - CO (trans) angles are 108° in trans-$(\pi\text{-}C_5H_5)Mo(CO)_2$-

$(PPh_3)(COMe)$ (3) and 117° in $(\pi\text{-}C_5H_5)Mo(CO)_3(C_3F_7)$ (6)

$(PPh_3)(COMe)$ (3) and 117° in $(\pi\text{-}C_5H_5)Mo(CO)_3(C_3F_7)$ (6). In A the mean Br - Mo - Br angle of 121.0° is of limited chemical significance, but the mean P - Mo - CO (trans) angle of 128.3° is probably a fair reflection of this angle in an ordered model and is reasonably close to the P - Mo - COMe angle of 133° in trans-$(\pi\text{-}C_5H_5)Mo(CO)_2(PPh_3)(COMe)$ (3). Similar values include the P - Mo - P angle of 135° in trans-$(\pi\text{-}C_5H_5)Mo(CO)(PPh_3)_2(NCO)$ (1) and the OC - Mo - C_3F_7 (trans) angle of 132° in $(\pi\text{-}C_5H_5)Mo(CO)_3(C_3F_7)$ (6). The cis-valency angles, on the other hand, are less sensitive to changes in the ligands. The values in A and B range from 73.9 - 82.1 and 76.2 - 78.2° respectively, while those in the other complexes all lie within the range 73 - 82°.

The molybdenum - carbonyl Mo - C bond lengths in B, 1.973(10) and 1.983(10) Å, do not differ significantly from each other and may be compared with the Mo - CO separation of 1.934 in $(\pi\text{-}C_5H_5)Mo(CO)$-$(PPh_3)_2(NCO)$ (1), 1.938 Å in $(\pi\text{-}C_5H_5)Mo(CO)(PPh_2{\cdot}CH_2{\cdot}CH_2{\cdot}PPh_2)C\ell$ (2) and mean separations of 1.955 in $(\pi\text{-}C_5H_5)Mo(CO)_2(PPh_3)(COMe)$ (3), 1.983 in $(\pi\text{-}C_5H_5)Mo(CO)_2(PPh_3)I$ (2), 1.99 in $(\pi\text{-}C_5H_5)Mo(CO)_3C\ell$ (5), 2.00 in $(\pi\text{-}C_5H_5)Mo(CO)_3(C_3F_7)$ (6), 2.00 in $(\pi\text{-}CH_3C_5H_4)Mo(CO)_2[P(OMe)_3]I$ (4), 2.02 in $(\pi\text{-}C_5H_5)Mo(CO)_2[P(OMe)_3]I$ (4) and 2.06 Å in $Mo(CO)_6$ (31). These values are all considerably shorter than the distance of 2.38 Å in a purely σ-bonded Mo - C linkage (32), illustrating the double-bond character of the Mo - C(carbonyl) bonds produced by a σ-π synergic interaction, in which σ-donation of an electron pair from the carbonyl to the metal is accompanied by back-donation from filled metal d orbitals to doubly degenerate π^*(CO) levels. Though the individual values in the above series are subject to some uncertainty, there is a trend which reflects, at least partially, the greater back-donation to the π^* orbitals of an individual carbonyl group as the number of these groups on the metal atom is reduced. This trend is analogous to differences between the Cr - C(carbonyl) distances in various arenechromium tricarbonyls, ca. 1.82 Å(33), and the distance in chromium hexacarbonyl, 1.909 Å(34). The C - O bond lengths in B, 1.150(12) and 1.158(11) Å, are in good agreement with Sutton's(35) value of 1.14(1) Å, while the corresponding Mo - C - O valency angles, 175.9(9) and 176.8(9)°, depart only slightly from linearity.

Comparisons with the above values demonstrate that, as a result of the cis/trans disorder, the Mo - C and C - O bond lengths of 2.087(11) and 0.977(13) Å respectively in A1 and 2.095(14) and 0.904(17) Å in A101 are unrealistic.

The molybdenum - (phenyl isocyanide) Mo - C bond length, 2.025(10) Å, is probably longer than the mean molybdenum - carbonyl bond length in B, 1.978(7) Å, and can be compared with the mean molybdenum - (tert-butyl isocyanide) bond lengths of 2.055(15) and 2.115(15) Å for the two sets of nonequivalent Mo - C distances in $[Mo(CNBu^t)_6I]^+I^-$(36) and the mean molybdenum - (methyl isocyanide) bond length of 2.148(5) Å in $Mo(CNCH_3)_4(CN)_4$(37). In this analysis the π-accepting ability of the phenyl isocyanide ligand is nearly comparable with that of the carbonyl ligands, but the decrease in ν(CO) from 2155cm^{-1} in carbon monoxide to 1974† cm^{-1} (symmetric stretching mode) and 1936† cm^{-1} (asymmetric stretching mode) in $(\pi\text{-}C_5H_5)Mo(CO)_2(CNPh)I$ is not paralleled by a similar decrease in ν(CN) on coordination. A small decrease in ν(CN) from 2132cm^{-1}(14) in the free ligand to 2108† cm^{-1} in B is observed, and can be compared with an increase of ca. 50cm^{-1} on forming $Mo(CNCH_3)_4(CN)_4$(37), in which only weak π-bonding is suspected. These

† Does not correspond exactly with the spectra of trans-$(\pi\text{-}C_5H_5)Mo(CO)_2(CNPh)I$ because the chloroform solution, in which the i.r. was measured, contained a mixture (ca. 50/50) of isomers.

frequency changes are a balance between the decrease due to π back-donation and the increase due to the increased contribution of canonical form (II) on coordination to the positively charged metal ion,

$$\underset{(I)}{R\text{—}\ddot{N}\text{=}C:} \longleftrightarrow \underset{(II)}{R\text{—}\overset{+}{N}\equiv\bar{C}}$$

and more structural and spectroscopic information is obviously required before ν(CN) values and bond lengths can be correlated.

The C(sp) - N bond length, 1.153(13) Å, in the phenyl isocyanide ligand compares favourably with mean values of 1.139(6) in $Mo(CNCH_3)_4(CN)_4$(37), 1.143(5) in $Ni(CNBu^t)_2(TCNE)$(38), 1.16(3) in cis-$[PtC\ell_2(CNPh)_2]$(19), 1.17(1) Å in $[Mo(CNBu^t)_6I]^+I^-$(36) and the value of 1.166 Å in methyl isocyanide(39). These distances in isocyanides are very similar to the mean distance of 1.158(2) Å in simple organic cyanides(40). The N - C(sp^2) bond length, 1.397(13) Å, in B is equal within experimental error to the mean value of 1.37(2) Å in cis-$[PtC\ell_2(CNPh)_2]$(19). The Mo - C - N - C fragment is nearly linear with Mo - C - N and C - N - C angles of 177.8(9) and 174.2(11)° respectively; an arrangement which is typical of metal-isocyanide linkages, cf. M - C - N angles of 176.9(4) and 172.4(4)° in $Ni(CNBu^t)_2$-(TCNE)(38) and 177.8(17) and 170.0(18)° in cis-$[PtC\ell_2(CNPh)_2]$(19) with corresponding C - N - C angles of 176.6(4), 171.5(4), 174.5(18) and 177.9(20)°. In both these complexes chemically equivalent angles are statistically different, suggesting a steric rather than electronic reason for the deviations from linearity in these and presumably in this analysis. These near-linearities are consistent with a large contribution of canonical form (II) in isocyanide complexes. The phenyl ring of the isocyanide ligand is planar within the limits of experimental error, the root-mean-square deviation from planarity being 0.020 Å, with the nitrogen atom deviating 0.035 Å from the plane. The C - C distances range from 1.342 - 1.409, mean 1.384 Å, and deviations of the individual values from the mean should be considered as an independent assessment of the reliability of the estimated standard deviations in B rather than as an indication of genuine variations.

The molybdenum - iodine separation of 2.858(1) Å can be compared with 2.836(4), 2.850(3), 2.858(3) and 2.862(3) Å in $(\pi\text{-}C_5H_5)Mo(CO)_2[P(OMe)_3]I^{(4)}$, $(\pi\text{-}CH_3C_5H_4)Mo(CO)_2[P(OMe)_3]I^{(4)}$, $(\pi\text{-}C_5H_5)Mo(CO)_2(PPh_3)I^{(2)}$ and $[Mo(CNBu^t)_6I]^+I^{-(36)}$ respectively. The individual values of the molybdenum - bromine separation in the analysis of $(\pi\text{-}C_5H_5)Mo(CO)_2(PPh_3)Br$ are Mo - Br(1) = 2.676(2), Mo - Br(1)' = 2.698(6), Mo - Br(101) = 2.666(1) and Mo - Br(101)' = 2.648(4) Å, while the average value, weighting each individual value according to the population parameter of the corresponding bromine atom, is 2.671 Å. The estimated standard deviations of the individual values are underestimated, but this analysis has the advantage of having two crystallographically independent molecules per asymmetric unit, enabling standard deviations to be derived from a comparison of the two independent sets of bond lengths. Allowing for the dependence of the Mo - Br bond lengths on the vibrational treatment of the disordered carbonyl groups, a reasonable standard deviation of the weighted mean distance is 0.003 Å. The weighted mean molybdenum - bromine separation, 2.671(3) Å, compares favourably with Mo - Br bond lengths found in other types of seven-coordinate Mo^{II} complexes, e.g. mean values of 2.662(2) and 2.662(3) Å in $MoBr_2(CO)_3(PPh_2\cdot CH_2\cdot CH_2\cdot PPh_2)^{(41)}$ and $MoBr_2(CO)_2(AsPh_2\cdot CH_2\cdot AsPh_2)_2^{(42)}$ respectively. [The latter complex is seven-coordinate because one of the arsine ligands is bi- and the other unidentate.] The molybdenum - bromine separation in A is 0.19 Å shorter than the molybdenum - iodine separation in B, and this is in excellent agreement with the difference in the covalent radii$^{(43)}$ of bromine, 1.14 Å, and iodine, 1.33 Å, atoms. The covalent radii$^{(43)}$ of chlorine atoms is 0.99 Å suggesting probable molybdenum - chlorine separations in related complexes of ca. 2.52 Å, compared with observed values of 2.541(5) and 2.542(9) Å in $(\pi\text{-}C_5H_5)Mo(CO)(PPh_2\cdot CH_2\cdot CH_2\cdot PPh_2)C\ell^{(2)}$ and $(\pi\text{-}C_5H_5)Mo(CO)_3C\ell^{(5)}$ respectively.

The standard deviations of the molybdenum - phosphorus bond lengths, 2.538(2) and 2.525(2) Å in A1 and A101 respectively, appear to be underestimated in the analysis of cis-$(\pi\text{-}C_5H_5)Mo(CO)_2(PPh_3)Br$, but nevertheless this bond is undoubtedly longer than that, 2.481(5) Å, in trans-$(\pi\text{-}C_5H_5)Mo(CO)_2(PPh_3)I^{(2)}$, in agreement with the difference found in $(\pi\text{-}C_5H_5)Mo(CO)(PPh_2\cdot CH_2\cdot CH_2\cdot PPh_2)C\ell^{(2)}$ in which the Mo - P bond, 2.496(4) Å, trans to the carbonyl group is appreciably longer than the bond, 2.439(5) Å, trans to the halogen atom. Although these ligands are not strictly trans to one another [The P - Mo - L angles lie between 110 and 145°.], this trans influence is reminiscent of bond-lengthening effects in square-planar and octahedral complexes$^{(44)}$. For comparison purposes the molybdenum - phosphorus separations in other complexes are 2.388 in trans-$(\pi\text{-}CH_3C_5H_4)Mo(CO)_2[P(OMe)_3]I^{(4)}$, 2.406 in trans-$(\pi\text{-}C_5H_5)Mo(CO)_2[P(OMe)_3]I^{(4)}$, 2.473 in trans-$(\pi\text{-}C_5H_5)Mo(CO)_2(PPh_3)(COMe)^{(3)}$, 2.499 in trans-$(\pi\text{-}C_5H_5)Mo(CO)$-$(PPh_3)_2(NCO)^{(1)}$, 2.505 in $(PPh_2\cdot NEt\cdot PPh_2)Mo(CO)_4^{(45)}$ and 2.517 Å in $(PPh_2C_6H_4\cdot CH{:}CHMe)Mo(CO)_4^{(46)}$. Subtraction of the covalent radius$^{(43)}$ of the phosphorus atom, 1.10 Å, from the molybdenum - phosphorus separation in the above complexes yields values between 1.29 and 1.43 Å for the contribution of the molybdenum atom to the molybdenum - phosphorus bond, whereas the single bond covalent radius of molybdenum in $(\pi\text{-}C_5H_5)MoL_2XY$ and $(\pi\text{-}C_5H_5)MoL_3X$ complexes is 1.61 Å(32). This contraction may be evidence for double-bond character in the metal - phosphorus bonds arising from $d\pi\text{-}d\pi$ Mo⟶P back-donation, and can be compared (see Table 16) with contractions involving other ligands, some of which have been attributed to multiple-bond character in the metal - ligand bonds.

TABLE 16

Bond lengths (B) and ligand single-bond covalent radii (R) in complexes of the type $(\pi\text{-}C_5H_5)MoL_2XY$ and $(\pi\text{-}C_5H_5)MoL_3X$

Bond	B (Å)	R (Å)	B - R	References
Mo - CO	1.93 - 2.02	0.70	1.23 - 1.32	This work, 1 - 6.
Mo - CNPh	2.03	0.70	1.33	This work.
Mo - P	2.39 - 2.53	1.10	1.29 - 1.43	This work, 1 - 4.
Mo - NCO	2.13	0.65	1.48	1.
Mo - COMe	2.26	0.74	1.52	3.
Mo - C_3F_7	2.29	0.77	1.52	6.
Mo - I	2.84 - 2.86	1.33	1.51 - 1.53	This work, 2, 4.
Mo - Br	2.67	1.14	1.53	This work.
Mo - Cl	2.54	0.99	1.55	2, 5.
Mo - C_2H_5	2.40	0.77	1.63	47.

The phosphorus - carbon bond lengths in the triphenylphosphine groups are normal, the mean distances of 1.831 and 1.832 Å in A1 and A101 respectively being close to the sum of the covalent radii, 1.84 Å, to mean distances in other triphenylphosphine complexes, e.g. 1.833, 1.826 and 1.828 Å in $(\pi\text{-}C_5H_5)Mo(CO)_2(PPh_3)I$(2), $(\pi\text{-}C_5H_5)Ni(PPh_3)Ph$(48) and $Cr(CO)_5(PPh_3)$(49) respectively and to the mean distance, 1.828 Å, in the uncomplexed triphenylphosphine molecule(50). Each of the C - P - C angles [103.4, 103.2 and 103.8° in A1 and 103.7, 103.4 and 104.0° in A101] is appreciably smaller than the regular tetrahedral value of 109°28' and this is typical(51) of triphenylphosphine transition-metal complexes that have been investigated by X-ray analysis, e.g. mean values of 103.4, 104.3 and 102.6° in $(\pi\text{-}C_5H_5)Mo(CO)_2(PPh_3)I$(2), $(\pi\text{-}C_5H_5)Ni(PPh_3)Ph$(48) and $Cr(CO)_5(PPh_3)$(49) respectively, and also of the uncomplexed triphenylphosphine molecule(50), 103.0°. Such deviations from ideal sp^3 hybridisation have been suggested(2) to be indicative of the phosphorus orbital directed towards the metal atom having more s-character than the orbitals directed towards the carbon atoms.

The carbon - carbon bond lengths in the triphenylphosphine ligands are equal within experimental error and range from 1.349 - 1.400, mean 1.375 Å, in A1 and from 1.347 - 1.400, mean 1.379 Å, in A101. Similar contractions from the spectroscopic value of 1.397 Å appropriate to benzene have been noted in other X-ray analyses(e.g. 1,2), and have been attributed to thermal motion. While the phenyl rings are accurately planar [The root-mean-square deviations from planarity are 0.009, 0.006 and 0.002 Å in A1 and 0.007, 0.006 and 0.014 Å in A101.], the phosphorus atoms are appreciably displaced from some of these planes. The displacements [0.02, 0.14 and 0.06 Å in A1 and 0.03, 0.05 and 0.01 Å in A101] are however considerably less than the corresponding displacements of 0.23 and 0.36 Å found in $(\pi\text{-}C_5H_5)Mo(CO)(PPh_3)_2(NCO)$(1) and (1-exo-benzylcyclopenta-2,4-diene)$Fe(CO)_2(PPh_3)$(52) respectively, and are probably due to van der Waals' repulsions between the phenyl groups. The different displacements in the two crystallographically independent molecules of A, therefore indicate that dissimilar repulsions arise from the different rotational orientation of the phenyl rings in A1 and A101, cf. dihedral angles between the phenyl rings of 83.1, 84.5 70.5° in A1 and 79.8, 99.3 and 105.5° in A101. Similar differences between two crystallographically independent molecules have been observed in the crystal structures of $(\pi\text{-}C_5H_5)Ni(PPh_3)(CF_3)^{(53)}$ and (1-exo-benzylcyclopenta-2,4-diene)$Fe(CO)_2(PPh_3)^{(52)}$ demonstrating the importance of intermolecular repulsions in determining the rotational orientation of the phenyl rings in triphenylphosphine transition-metal complexes.

The π-cyclopentadienyl rings in A and B are accurately planar, the root-mean-square deviations from planarity being 0.013, 0.005 and 0.016 Å in A1, A101 and B respectively. The cyclopentadienyl carbon - carbon bond lengths range from 1.379 - 1.430, mean 1.401 Å, in A1, 1.375 - 1.430, mean 1.402 Å, in A101 and 1.390 - 1.443, mean 1.412 Å, in B and can be compared with a value of 1.419 Å found from an average of 23 different X-ray analyses of π-cyclopentadienyl derivatives$^{(54)}$ and with values of 1.431(5) and 1.427(7) Å found in the electron-diffraction studies of $(\pi\text{-}C_5H_5)_2Fe^{(55)}$ and $(\pi\text{-}C_5H_5)In^{(56)}$ respectively. The shorter values found in X-ray analyses have again been attributed$^{(54)}$ to thermal motion. The molybdenum - carbon(cyclopentadienyl) distances range significantly from 2.290 - 2.383, mean 2.330 Å, in A1, in an equivalent trend from 2.294 - 2.374, mean 2.335 Å, in A101 and from 2.289 - 2.382, mean 2.327 Å, in B. These mean separations are in good agreement with values in related complexes, e.g. 2.333 in $(\pi\text{-}C_5H_5)Mo(CO)_2(PPh_3)I^{(2)}$, 2.333 in $(\pi\text{-}C_5H_5)Mo(CO)(PPh_3)_2(NCO)^{(1)}$ and 2.347 Å in $(\pi\text{-}C_5H_5)Mo(CO)_2(PPh_3)(COMe)^{(3)}$, and are only a little shorter than the molybdenum - ethyl bond length of 2.397(19) Å in $(\pi\text{-}C_5H_5)Mo(CO)_3(C_2H_5)^{(47)}$.

Significant variations in metal - carbon and carbon - carbon bond lengths can be rationalized$^{(57)}$ in terms of the lack of cylindrical symmetry around the metal atom removing the degeneracy of the e_1 molecular orbitals of the π-cyclopentadienyl ring. In both A1 and A101 significant variations in the molybdenum - carbon bond lengths are observed and, with respect to their major orientation, are in the sense expected. Both the molybdenum - carbon bonds in A situated approximately trans to a carbonyl group are ca. 0.07 Å longer than those associated with the atoms of the cyclopentadienyl bonds which pass under either of the molybdenum - carbonyl bonds, analogous to trends in for example $(\pi\text{-}C_5H_5)Mo(CO)(PPh_3)_2(NCO)^{(1)}$, $(\pi\text{-}C_5H_5)Mo(CO)_2(PPh_3)(COCH_3)^{(3)}$ and $(\pi\text{-}C_5H_5)Mo(CO)_3(C_3F_7)^{(6)}$. Sim has suggested(1) that these variations may reflect partly the appreciable π-character of the metal - carbonyl bond, therefore it is interesting to observe that in $(\pi\text{-}C_5H_5)Mo(CO)_2(CNPh)I$, of the two molybdenum - carbon bonds which are ca. 0.08 Å longer than the others, one is situated approximately trans to a carbonyl group while the other is approximately trans to the π-accepting phenyl isocyanide ligand. Although significant variations in the C - C bond lengths and C - C - C valency angles have been observed in other π-cyclopentadienyl complexes, e.g. $(\pi\text{-}C_5H_5)Cr(NO)_2(NCO)^{(58)}$, in A and B none of the deviations of these dimensions from their mean value is statistically significant.

The intermolecular contacts in both analyses appear to be purely van der Waals' interactions. In the methylene chloride molecule the carbon - chlorine bond lengths, 1.78(4) and 1.65(4) Å, are in reasonable agreement with each other and with the distance of 1.77 Å found both in a microwave study(59) of the gaseous molecule and in an X-ray analysis(60) at -120°C, while the most accurately determined dimension, i.e. the Cℓ - - - Cℓ separation of 2.885(6) Å, is slightly shorter, presumably as a result of the fairly high thermal motion in this analysis, than the values of 2.935 and 2.932(4) Å in these analyses respectively. The Cℓ - C - Cℓ angle, 115(2)°, is in good agreement with the angles of 111.8 and 112(1)° found in the microwave(59) and X-ray(60) analyses respectively.

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PART III

CHARACTERIZATION OF SOME ORGANOMETALLIC COMPLEXES

CHAPTER 1

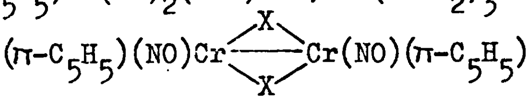
π-CYCLOPENTADIENYL(DIPHENYLAMIDO)IODONITROSYLCHROMIUM

III.1.1 INTRODUCTION

Dr. G. R. Knox and co-workers at Strathclyde University prepared the title complex according to the reactions:

$$\left.\begin{array}{l} \mathrm{MeI} + 2\mathrm{Li} \longrightarrow \mathrm{MeLi} + \mathrm{LiI} \\ \mathrm{MeLi} + \mathrm{HNPh_2} \longrightarrow \mathrm{LiNPh_2} \end{array}\right\} + (\pi\text{-}C_5H_5)Cr(NO)_2C\ell \longrightarrow (\pi\text{-}C_5H_5)Cr(NO)(NPh_2)I$$

Characterization by elemental analysis, i.r. and n.m.r. spectroscopy, mass spectrometry and osmetric molecular weight measurements indicated the above monomeric formulation, but, because this implied only 16 metal valence electrons, very weak iodine or nitrogen bridges were not excluded.

An X-ray analysis was undertaken in the knowledge that, whatever formulation was correct, interesting structural comparisons were likely with some of the following materials: $(\pi\text{-}C_5H_5)Cr(NO)_2C\ell^{(1)}$, $(\pi\text{-}C_5H_5)Cr(NO)_2(NCO)^{(2)}$, $Cr(NPr^i_2)_3^{(3)}$, the anion $(CO)_5Cr - I - Cr(CO)_5^{(4)}$ and  $(\pi\text{-}C_5H_5)(NO)Cr\overset{X}{\underset{X}{=\!=}}Cr(NO)(\pi\text{-}C_5H_5)$ where X = $SPh^{(5)}$, $OMe^{(6)}$ and $NMe_2^{(7)}$.

III.1.2 EXPERIMENTAL

Crystal Data π-Cyclopentadienyl(diphenylamido)iodonitrosylchromium; $C_{17}H_{15}N_2CrI$.

Crystal system	Monoclinic
Unit cell dimensions	a = 10.247(2) Å
	b = 8.888(2) Å
	c = 9.236(2) Å
	β = 91°30'(1)
Space group	$P2_1$ (C_2^2)

U = 840.9 $Å^3$

M = 442.2 a.m.u.

D_o = 1.74 $gm.cm.^{-3}$ (flotation in aqueous zinc iodide solution)

D_c = 1.746 $gm.cm.^{-3}$

Z = 2

F(000) = 432

μ(Mo-$K\alpha$) = 25.66 $cm.^{-1}$

Crystallographic Measurements

Preliminary cell dimensions and systematically absent reflections (0k0 absent if $k = 2n + 1$) consistent with space groups $P2_1$ and $P2_1/m$ were obtained from precession photographs of a red crystal mounted about b* with dimensions 0.22 x 0.40 x 0.50 mm^3 in the $[001]$, $[1\bar{1}0]$ and $[110]$ directions respectively.

The crystal, having been transfered to a Hilger and Watts' Y290 automatic diffractometer controlled by a PDP-8 computer, was offset on χ by about 3° both to prevent multiple reflections$^{(8)}$ and to enable a data set to be collected without any observations being made at χ values of about 90°. The cell dimensions were then adjusted by a least-squares treatment$^{(9)}$ of the θ, χ and ϕ setting angles of twelve reflections measured with Mo-$\underline{K}\alpha$ radiation from a graphite crystal monochromator. The fully stabilized X-ray generator was operated at 42 kV and 17 mA, when collecting all the reflections in octants $hk\ell$ and $hk\bar{\ell}$ with $\theta(\text{Mo-}\underline{K}\alpha) \leqslant 30^\circ$, then at 28 kV and 10 mA, in order to minimise dead time losses, within the range $\theta(\text{Mo-}\underline{K}\alpha) \leqslant 10^\circ$.

The intensity data were collected by the $\theta - 2\theta$ scan procedure, each reflection being scanned in 36 equal steps from $2\theta_{calc.} -0.72^\circ$ to $2\theta_{calc.} +0.72^\circ$. Each step was counted for 2 seconds and stationary-crystal, stationary-counter background counts were taken at each end of the scan range for 18 seconds. The intensities of two standard reflections were monitored every 40 intensity measurements and only small random fluctuations were observed. The scan counts (P) and the background counts (B_1,B_2) of each reflection were combined to yield the integrated intensity, $I = P - 2(B_1 + B_2)$, which was corrected for Lorentz-polarisation effects. 2,590 independent measurements (including 28 corrected for dead time losses) were obtained, of which 192 had $I/\sigma_I < 5.0$ and were omitted from the structure determination. The remaining 2,398 independent values of

$|F_o|$ were corrected for absorption using the DATAP program of P. Coppens modified for use on Glasgow University's KDF9 computer by I. R. Mackay(10). A Gaussian 8 x 8 x 8 grid was employed and the transmission factors ranged from 0.37 to 0.58.

Structure Analysis

The initial coordinates of the chromium and iodine atoms were obtained from the three-dimensional Patterson synthesis, and the subsequent electron density distribution (R = 20%) revealed the positions of the remaining non-hydrogen atoms, indicating that the space group is almost certainly $P2_1$ and not the alternative $P2_1/m$.

The approximate atomic parameters were adjusted by a series of least-squares calculations, using the program due to D. W. J. Cruickshank, J. G. F. Smith and J. G. Sime on Glasgow University's KDF9 computer. The weighting scheme used throughout the refinement was:

$$w = \left[1 - \exp(-p_1\{\sin\theta/\lambda\}^2)\right]/\left[1 + p_2|F_o| + p_3|F_o|^2\right],$$

where the values of p_1, p_2 and p_3 were adjusted at several stages of the refinement to ensure approximate constancy of $\langle w\Delta^2\rangle$ over various ranges of $|F_o|$ and $\sin\theta$. The final values were 5.0, 0.1 and 0.0005 respectively. The atomic scattering factors, including dispersion corrections $\Delta f'$ and $\Delta f''$ for the chromium and iodine atoms, were taken from 'International Tables for X-ray Crystallography,' Vol. III.(11)

The y-coordinate of the chromium atom was arbitrarily fixed at 1.0 to define the origin. Two cycles of full-matrix refinement of the remaining positional and the isotropic thermal parameters reduced R to 5.25%, R' = 7.10%, and with all the atoms assigned anisotropic thermal parameters, three cycles of block diagonal least-squares reduced R to 2.84%, R' = 3.71%. Comparison between the calculated and observed structure factors revealed that, for the 0k0 reflections, $|F_o|$ was about 15% lower than $|F_c|$. These reflections had been collected at a high χ setting (ca. 87°) where the orientation matrix is sometimes unreliable, therefore they were recollected, as were six general reflections for scaling purposes, with the crystal further offset by about 10°. Three further cycles of refinement using the corrected data converged at R = 2.62%, R' = 3.25%.

$P2_1$ is a polar space group, therefore two polarities for the crystal structure are possible. If anomalous scattering occurs (as in this analysis) it should be possible to distinguish between the two possibilities either by statistical tests on the crystallographic R-factor(12) or by an assessment of the relative stereochemical plausibilities of the two structures.

The approximate parameters of the other enantiomorph were calculated by reflecting through the plane y = 1.0, and then refined by five cycles of block diagonal least-squares to convergence at R = 2.64%, R' = 3.23%. For both enantiomorphs an electron density difference map was calculated restricting the summation to those reflections with $\sin\theta/\lambda < 0.4$. All of the hydrogen atoms, except H(15), were found in both maps at average peak heights of 0.26 and $0.27e^-/\text{Å}^3$ in the maps corresponding to enantiomorphs I and II respectively. The highest peaks not attributable to hydrogen atoms had peak heights 0.27 and $0.21e^-/\text{Å}^3$ in maps I and II respectively. The coordinates of H(15) were calculated and both enantiomorphs refined identically as follows:

5 cycles of a) block diagonal least-squares refining the non-hydrogen atoms anisotropically and the hydrogen atoms isotropically.

2 cycles of b) full-matrix least-squares refining the hydrogen atoms isotropically.

1 cycle of type a).

Refinement converged for enantiomorph I at R = 2.16%, R' = 2.857%, and for enantiomorph II at R = 2.16%, R' = 2.841%. Neglecting anomalous dispersion, and starting with the set of parameters obtained by averaging the final parameters of enantiomorph II and the final parameters of enantiomorph I reflected through the plane $y = 1.0$, five cycles of least-squares refinement [type a)] converged at $R = 2.11\%$, $R' = 2.825\%$. Including anomalous dispersion, structure factors were calculated on the final parameters obtained without the dispersion corrections giving $R = 2.66\%$, $R' = 3.539\%$, for enantiomorph I and $R = 2.67\%$, $R' = 3.477\%$, for enantiomorph II. The difference between these weighted R-factors is unexpectedly small, considering the presence of both chromium and iodine atoms, and is insufficient to allow the absolute stereochemistry to be determined.

It has been pointed out(13) that unless the $\Delta f''$ contribution is included in the structure factor calculation, the anomalous scatterer will appear to be closer to the X-ray source than it actually is. Consequently, if the origin is fixed by an anomalous scatterer in a polar space group, on allowing for anomalous dispersion the other atoms undergo shifts in the polar direction. For different enantiomorphs the shifts have the same magnitude but opposite sense with respect to the anomalous scatterer. The coordinate error, Δy, introduced by including $\Delta f''$ in the calculation and choosing the incorrect enantiomorph can be estimated from the following formula derived by Cruickshank and McDonald(14):

$$\Delta y = \frac{2}{\pi S_{max}}\left(\frac{\Delta f''}{|f|}\right)_{\frac{1}{2}S_{max}}$$

where $S = (2\sin\theta)/\lambda$, $|f|$ is the modulus of the complex atomic scattering factor and the quantity $(\Delta f''/|f|)$ is taken as the phase shift due to anomalous scattering evaluated at $S = \frac{1}{2}S_{max}$.

In this analysis, the above formula predicts an insignificant error in the y-coordinate of the iodine atom and an error of 0.043 Å in the y-coordinate of the light atoms with respect to the chromium atom, compared with observed differences between the two enantiomorphs of 0.003 and 0.038 Å (average) respectively, i.e. 6σ and 8σ respectively. Comparison of the bond lengths obtained from the two refinements including anomalous dispersion reveals essentially no differences in the bond lengths not involving the chromium, while differences in the chromium - light atom bond lengths reflect the weight of the y component in the bond length calculation and range from 0.003 to 0.029 Å, σ to 6σ; for the Cr - N(19) and Cr - C(15) bonds respectively. The corresponding Cr - N distances [1.892 and 1.675 Å in enantiomorph I and 1.896 and 1.678 Å in enantiomorph II] are very similar and the Cr - C(cyclopentadienyl) distances [2.224 - 2.297, mean 2.260 Å, in enantiomorph I and 2.195 - 2.286, mean 2.239 Å, in enantiomorph II] are in reasonable agreement with those found in other chromium-cyclopentadienyl complexes (see Table 8, page 138), therefore, unlike some other analyses(e.g. 13, 15, 16) in which one refinement clearly gave more chemically reasonable bond lengths, an unequivocal assignment of the absolute stereochemistry from bond length comparisons is impossible.

Results

All the tables and figures refer to the refinement carried out neglecting anomalous dispersion.

The observed and final calculated structure factors are listed in Table 1, and the final positional and thermal parameters, including the estimated standard deviations derived from the least-squares refinement, are given in Table 2. The atomic numbering scheme of the non-hydrogen atoms (each hydrogen atom is numbered according to the atom to which it is bonded) is shown in Figure 1; a perspective view of the molecule projected on to the π-cyclopentadienyl plane illustrating the thermal ellipsoids(17). The bond lengths, valency angles and selected mean plane calculations are given in Tables 3, 4 and 5 respectively. The crystal packing is shown in Figure 2 and the intermolecular contacts $\leqslant 3.80$ Å are listed in Table 6.

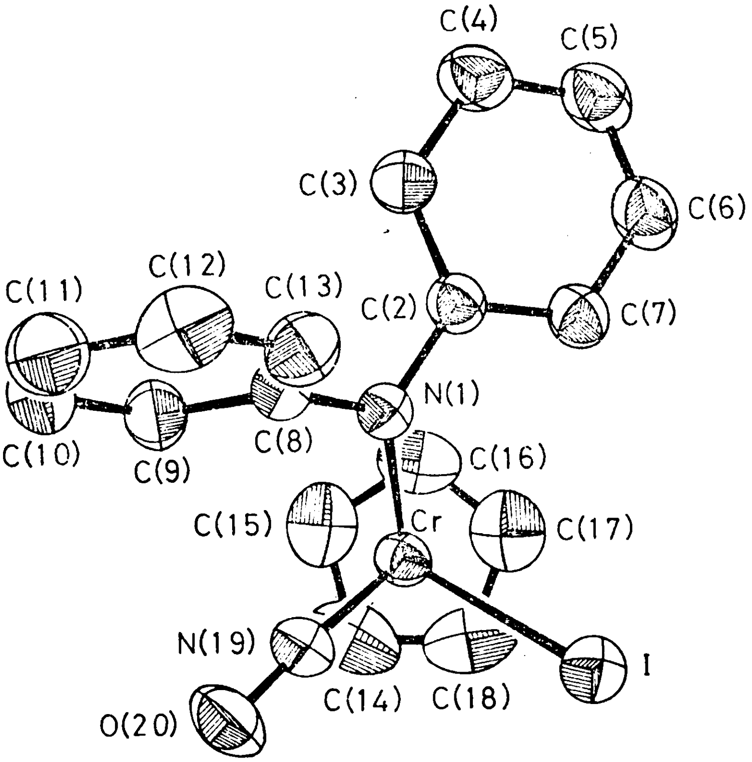


FIGURE 1

Perspective view of π-cyclopentadienyl(diphenylamido)iodonitrosylchromium projected on to the π-cyclopentadienyl plane, showing the 50% probability thermal ellipsoids.

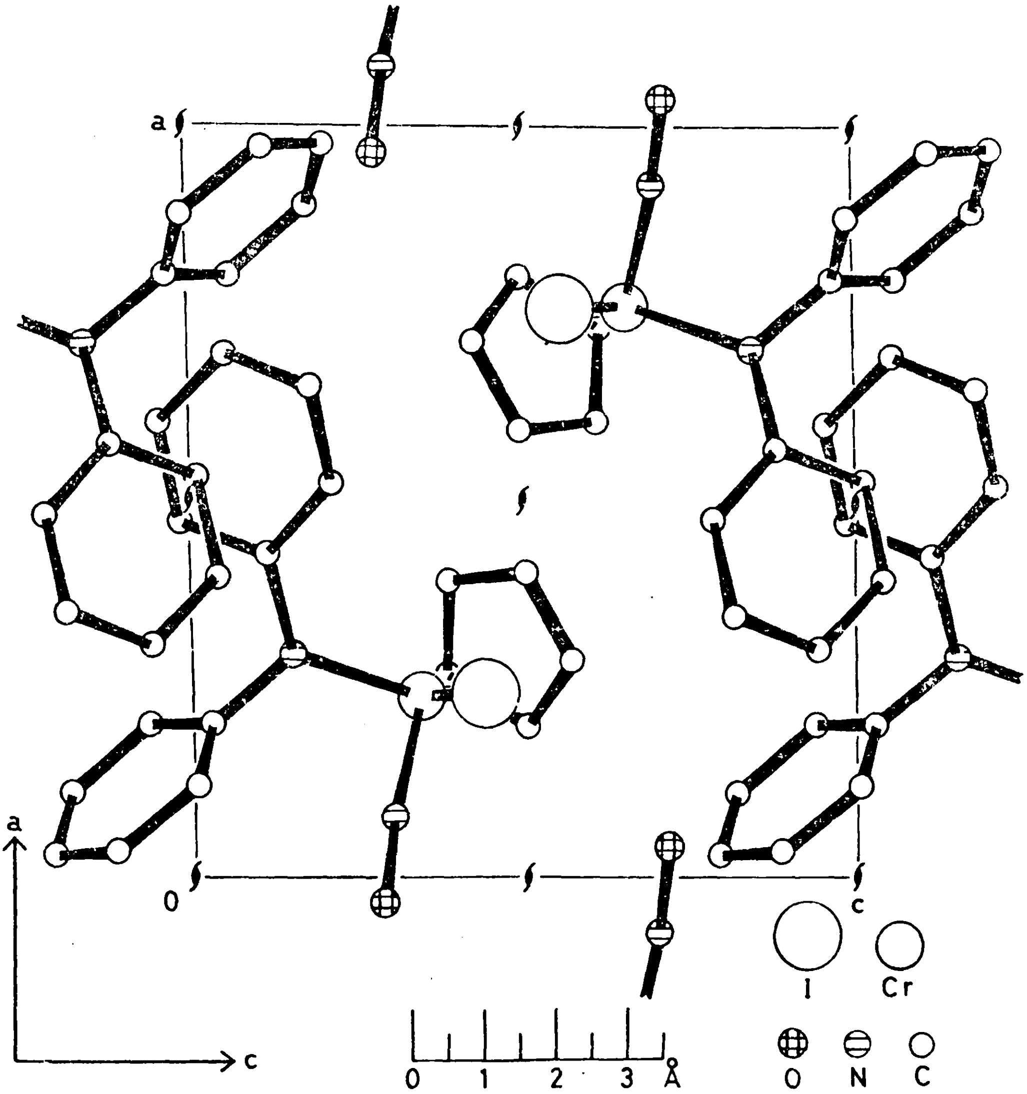


FIGURE 2

Crystal packing of π-cyclopentadienyl(diphenylamido)iodonitrosylchromium viewed along the b-axis.

TABLE 1

π-Cyclopentadienyl(diphenylamido)iodonitrosylchromium; observed and final calculated structure amplitudes.

H	K	L	F OBS	F CALC
14	2	1	6.2	5.7
14	2	0	4.6	4.6
14	2	-2	4.2	4.4
14	1	2	5.1	5.6
14	1	1	4.4	5.0
14	1	0	5.2	5.4
14	1	-1	2.7	2.9
14	1	-2	9.1	9.3
14	0	0	7.7	8.0
14	0	-1	11.8	12.4
14	0	-2	8.6	8.5
13	5	1	6.1	5.5
13	5	0	3.6	3.5
13	5	-1	3.6	3.9
13	5	-2	3.9	3.9
13	4	3	4.3	3.5
13	4	2	7.5	7.9
13	4	1	6.1	6.5
13	4	0	7.9	8.0
13	4	-2	5.2	5.2
13	4	-3	6.5	6.3
13	4	-4	4.9	5.2
13	3	2	4.6	4.8
13	3	1	8.5	8.9
13	3	-1	11.9	11.5
13	3	-2	8.2	8.2
13	3	-3	5.6	5.4
13	3	-4	3.4	2.9
13	2	4	5.8	9.2
13	2	3	10.8	11.0
13	2	2	4.4	4.5
13	2	1	4.8	4.6
13	2	-2	3.0	3.4
13	2	-4	7.4	7.7
13	2	-5	8.3	8.6
13	1	5	7.4	7.5
13	1	4	3.5	3.8
13	1	3	2.6	2.7
13	1	2	6.1	6.4
13	1	1	6.4	6.2
13	1	0	6.7	6.7
13	1	-1	9.6	9.9
13	1	-2	7.9	8.1
13	1	-3	6.2	6.7
13	1	-4	5.4	5.6
13	1	-5	2.8	3.1
13	0	5	3.6	3.0
13	0	3	2.8	2.7
13	0	2	10.6	11.1
13	0	1	10.5	10.9
13	0	0	6.1	6.4
13	0	-1	4.3	3.9
13	0	-2	9.3	9.3
13	0	-3	12.1	11.9
13	0	-4	5.0	5.3
13	0	-5	4.4	4.1
12	6	2	5.6	5.4
12	6	1	4.2	4.2
12	6	0	6.6	6.5
12	6	-1	6.1	5.9
12	6	-2	4.9	4.9
12	6	-3	7.4	7.5
12	5	4	8.7	8.6
12	5	3	10.7	10.6
12	5	2	6.3	6.6
12	5	1	4.2	3.9
12	5	0	2.4	2.0
12	5	-2	4.5	4.4
12	5	-4	7.1	7.1
12	5	-5	7.4	7.6
12	4	4	4.6	4.4
12	4	2	4.2	3.6
12	4	1	4.9	4.9
12	4	0	9.9	9.8
12	4	-1	13.5	13.6
12	4	-2	11.7	11.7
12	4	-4	4.1	3.5
12	4	-6	2.8	2.1
12	3	6	4.7	4.1
12	3	5	3.8	3.7
12	3	4	6.3	6.6
12	3	3	7.7	7.8
12	3	2	10.0	10.1
12	3	1	11.5	11.8
12	3	0	5.6	8.8
12	3	-2	9.9	10.3
12	3	-3	10.1	10.0
12	3	-4	6.0	6.1
12	3	-5	7.3	7.1
12	3	-6	4.6	5.0
12	2	6	7.3	7.3
12	2	5	11.6	12.1
12	2	4	3.9	4.1
12	2	2	9.9	9.9
12	2	1	8.2	8.0
12	2	0	8.6	8.3
12	2	-1	5.2	4.9
12	2	-2	7.6	7.9
12	2	-3	10.8	11.0
12	2	-4	9.3	9.7
12	2	-6	5.1	5.0
12	2	-7	9.0	9.2
12	1	6	4.6	4.2
12	1	5	4.7	4.9
12	1	4	9.4	9.4
12	1	3	11.4	11.5
12	1	2	9.9	10.5
12	1	1	11.6	12.1
12	1	0	4.8	4.9
12	1	-1	3.0	2.9
12	1	-2	7.6	7.6
12	1	-3	8.2	8.1
12	1	-4	8.5	9.0
12	1	-5	10.2	10.2
12	1	-6	9.1	9.4
12	1	-7	4.7	4.9
12	0	6	5.7	5.2
12	0	4	4.6	4.9
12	0	3	3.1	3.3
12	0	2	5.0	4.9
12	0	1	6.3	6.1
12	0	0	10.3	10.3
12	0	-1	24.0	24.7
12	0	-2	11.4	11.6
12	0	-3	5.7	5.4
12	0	-4	3.1	3.2
12	0	-5	2.4	2.5
11	8	0	2.5	2.7
11	7	3	3.7	3.7
11	7	1	4.1	4.0
11	7	0	8.7	9.0
11	7	-1	12.0	12.3
11	7	-2	7.1	7.3
11	7	-3	2.7	2.2
11	6	5	4.7	5.1
11	6	4	9.1	9.5
11	6	3	9.8	9.6
11	6	2	6.0	6.1
11	6	1	7.2	6.9
11	6	0	3.1	2.7
11	6	-2	5.6	5.5
11	6	-3	5.4	5.2
11	6	-4	8.0	7.8
11	6	-5	7.6	7.7
11	5	6	7.4	7.6
11	5	5	11.1	11.0
11	5	4	4.7	4.9
11	5	3	3.4	4.1
11	5	2	7.4	7.4
11	5	1	8.7	8.1
11	5	0	7.9	7.7
11	5	-1	5.4	5.5
11	5	-2	8.4	8.4
11	5	-3	10.7	10.4
11	5	-4	7.6	7.8
11	5	-6	4.7	4.7
11	4	6	4.2	4.3
11	4	5	4.0	3.8
11	4	4	5.0	5.0
11	4	3	5.7	5.6
11	4	2	12.7	13.1
11	4	1	13.0	13.4
11	4	0	9.5	9.7
11	4	-1	2.9	2.5
11	4	-2	11.7	11.9
11	4	-3	10.2	9.5
11	4	-4	7.3	7.0
11	4	-5	8.1	7.7
11	4	-6	6.1	6.0
11		-7	3.6	3.5
11	3	7	6.0	6.2
11	3	6	5.7	5.9
11	3	5	6.9	7.1
11	3	4	5.5	5.6
11	3	3	5.6	5.7
11	3	2	4.7	4.7
11	3	1	9.1	8.9
11	3	0	16.3	16.3
11	3	-1	20.7	20.9
11	3	-2	14.2	13.9
11	3	-3	8.6	8.5
11	3	-4	6.6	6.7
11	3	-7	4.6	4.5
11	3	-8	5.3	5.0
11	2	7	4.9	4.8
11	2	6	3.9	3.6
11	2	5	5.6	5.9
11	2	4	16.2	16.4
11	2	3	18.3	18.6
11	2	2	9.9	9.8
11	2	0	5.1	4.5
11	2	-1	4.6	4.9
11	2	-2	3.5	3.7
11	2	-3	5.2	5.1
11	2	-4	14.5	14.4
11	2	-5	15.4	15.7
11	2	-6	11.2	11.2
11	2	-7	5.7	5.9
11	1	8	5.1	5.2
11	1	7	6.5	6.2
11	1	6	8.0	8.1
11	1	5	11.7	11.9
11	1	4	4.9	4.9
11	1	3	6.9	6.9
11	1	2	14.0	14.0
11	1	1	11.1	10.9
11	1	0	15.7	15.8
11	1	-1	18.8	19.0
11	1	-2	14.1	14.7
11	1	-3	12.6	12.9
11	1	-4	9.7	10.0
11	1	-5	3.1	2.6
11	1	-6	3.9	4.0
11	1	-7	9.4	9.6
11	1	-8	4.6	4.9
11	0	7	2.9	3.2
11	0	6	5.5	5.4
11	0	5	7.3	7.4
11	0	4	3.7	3.8
11	0	3	6.6	6.8
11	0	2	15.5	16.0
11	0	1	22.0	22.7
11	0	0	15.2	15.7
11	0	-1	6.6	6.2
11	0	-2	12.6	12.1
11	0	-3	16.6	16.4
11	0	-4	9.4	8.6
11	0	-5	6.3	6.5
11	0	-6	5.5	5.4
11	0	-7	4.4	4.7
11	0	-8	7.3	7.1
10	8	4	3.0	3.3
10	8	3	3.6	3.5
10	8	2	3.0	2.7
10	8	1	8.8	8.8
10	8	0	5.2	5.7
10	8	-1	8.3	8.1
10	8	-2	5.0	4.7
10	8	-3	4.8	5.0
10	8	-4	3.0	2.6
10	7	5	3.1	2.7
10	7	3	5.9	5.6
10	7	2	9.2	9.1
10	7	1	12.4	12.7
10	7	0	5.4	5.4
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7	5	1	14.5	15.9
7	5	0	21.3	21.5
7	5	-1	12.8	12.9
7	5	-2	25.1	24.7
7	5	-3	23.3	23.1
7	5	-4	20.7	19.9
7	5	-5	7.9	8.1
7	5	-6	13.0	13.4
7	5	-7	15.1	15.4
7	5	-8	10.7	10.9
7	5	-9	5.1	4.6
7	5	-10	2.5	3.1
7	4	10	6.7	6.9
7	4	9	8.0	9.0
7	4	8	2.6	2.5
7	4	7	5.5	6.0
7	4	6	7.7	8.0
7	4	5	9.7	9.7
7	4	4	22.4	22.0
7	4	3	15.7	16.1
7	4	2	37.9	37.5
7	4	1	39.9	39.2
7	4	0	12.4	12.1
7	4	-1	9.4	9.1
7	4	-2	28.3	27.3
7	4	-3	32.7	31.9
7	4	-4	14.5	14.6
7	4	-5	17.6	17.1
7	4	-6	6.7	6.6
7	4	-7	13.0	13.1
7	4	-8	6.5	6.5
7	4	-9	3.3	4.0
7	4	-10	5.4	5.0
7	3	9	7.9	6.3

H	K	L	F OBS	F CALC
7	3	8	12.9	13.3
7	3	7	13.3	13.4
7	3	6	13.5	14.0
7	3	5	11.7	11.9
7	3	3	12.3	12.2
7	3	2	21.6	21.2
7	3	1	32.7	31.7
7	3	0	37.8	37.1
7	3	-1	51.3	50.4
7	3	-2	33.5	33.3
7	3	-3	23.3	22.9
7	3	-4	14.2	13.9
7	3	-5	8.3	8.1
7	3	-6	6.5	6.4
7	3	-7	13.3	13.9
7	3	-8	10.8	10.9
7	3	-9	13.5	13.4
7	3	-10	8.4	9.2
7	3	-11	3.7	3.4
7	2	11	3.0	2.7
7	2	10	4.2	3.4
7	2	9	4.5	4.2
7	2	8	8.0	7.4
7	2	7	9.5	9.1
7	2	6	4.3	4.8
7	2	5	22.7	22.9
7	2	4	38.2	38.0
7	2	3	53.3	50.8
7	2	2	30.3	30.3
7	2	1	12.9	12.5
7	2	0	5.0	4.9
7	2	-1	10.3	9.2
7	2	-2	9.4	8.7
7	2	-3	17.0	17.2
7	2	-4	44.1	44.1
7	2	-5	28.6	29.5
7	2	-6	26.8	26.9
7	2	-7	2.5	2.1
7	2	-8	5.9	6.2
7	2	-9	4.6	4.7
7	2	-11	2.9	2.9
7	1	9	7.2	7.3
7	1	8	12.8	12.8
7	1	7	17.9	17.4
7	1	6	23.9	24.4
7	1	5	19.1	19.2
7	1	4	11.5	12.0
7	1	3	15.8	15.1
7	1	2	32.5	32.3
7	1	1	27.4	25.3
7	1	0	41.8	41.8
7	1	-1	44.2	44.4
7	1	-2	31.2	31.1
7	1	-3	35.5	35.1
7	1	-4	15.8	15.5
7	1	-5	6.0	6.0
7	1	-6	16.9	17.3
7	1	-7	19.4	20.0
7	1	-8	15.7	16.0
7	1	-9	11.0	11.1
7	1	-10	6.6	6.6
7	0	11	6.1	6.3
7	0	10	10.7	10.7
7	0	9	7.7	7.7
7	0	8	3.1	2.5
7	0	7	9.8	9.7
7	0	6	12.7	12.6
7	0	5	15.3	15.0
7	0	4	15.9	15.8
7	0	3	26.1	26.4
7	0	2	39.9	39.7
7	0	1	61.2	61.1
7	0	0	22.2	21.9
7	0	-1	28.2	27.0
7	0	-2	42.7	42.2
7	0	-3	47.7	45.2
7	0	-4	10.9	11.0
7	0	-5	16.5	19.1
7	0	-6	4.9	4.7
7	0	-7	12.9	13.1
7	0	-8	12.1	12.1
7	0	-10	7.5	7.5
7	0	-11	7.2	7.9
6	11	2	6.0	6.4
6	11	1	6.5	6.8
6	11	0	2.2	2.3
6	11	-1	2.4	2.2
6	11	-2	6.3	6.5
6	11	-3	4.6	4.6
6	10	5	2.5	2.1
6	10	3	5.1	5.3
6	10	2	4.5	4.6
6	10	1	4.5	4.4
6	10	0	8.3	9.6
6	10	-1	10.5	10.6
6	10	-2	5.6	5.5
6	10	-3	5.1	4.3
6	9	5	7.8	7.6
6	9	4	13.5	13.8
6	9	3	13.5	10.6
6	9	2	6.5	6.4
6	9	1	2.2	1.6
6	9	-2	5.6	6.3
6	9	-3	7.6	7.0
6	9	-4	11.4	11.6
6	9	-5	13.8	10.7
6	9	-6	6.5	6.9
6	8	8	5.1	5.4
6	8	7	9.1	8.0
6	8	6	7.6	7.3
6	8	5	8.9	8.5
6	8	4	4.6	4.7
6	8	3	6.4	6.8
6	8	2	6.4	6.2
6	8	1	15.4	15.3
6	8	0	17.6	17.7
6	8	-1	13.2	13.0
6	8	-2	12.8	12.9
6	8	-3	10.8	10.7
6	8	-4	5.4	5.6
6	8	-6	6.3	6.4
6	8	-7	7.2	6.9
6	8	-8	6.0	5.5
6	7	9	5.1	5.0
6	7	7	4.2	4.7
6	7	6	5.4	5.9
6	7	5	6.5	6.5
6	7	4	6.1	6.1
6	7	2	23.5	23.7
6	7	1	21.8	21.3
6	7	0	7.2	7.2
6	7	-1	11.6	11.2
6	7	-2	23.2	20.3
6	7	-3	13.9	13.7

H	K	L	F OBS	F CALC
6	7	-4	11.1	10.9
6	7	-5	8.4	7.9
6	7	-6	7.2	6.7
6	7	-7	7.6	7.5
6	7	-8	5.0	5.0
6	6	9	3.7	4.0
6	6	8	8.2	8.1
6	6	7	9.7	10.1
6	6	6	16.6	16.3
6	6	5	14.2	13.9
6	6	4	5.5	5.3
6	6	3	11.9	11.4
6	6	2	20.2	20.2
6	6	1	15.7	15.6
6	6	0	20.6	20.9
6	6	-1	25.2	27.8
6	6	-2	23.0	22.5
6	6	-3	15.7	15.3
6	6	-4	14.7	14.5
6	6	-5	7.9	7.8
6	6	-6	8.5	8.4
6	6	-7	12.9	12.6
6	6	-8	8.3	9.1
6	6	-10	4.4	4.5
6	5	10	2.6	1.9
6	5	9	4.1	3.9
6	5	8	7.2	6.8
6	5	7	7.1	6.7
6	5	6	6.4	6.8
6	5	5	23.5	23.1
6	5	4	30.6	30.0
6	5	3	33.4	33.5
6	5	2	19.1	18.8
6	5	1	14.6	14.2
6	5	0	7.7	7.7
6	5	-1	8.5	8.1
6	5	-2	4.9	4.5
6	5	-3	19.2	19.0
6	5	-4	26.8	26.9
6	5	-5	29.8	29.7
6	5	-6	19.0	19.1
6	5	-7	3.9	3.9
6	5	-8	4.2	4.7
6	5	-9	4.7	4.9
6	5	-10	2.8	2.5
6	4	9	11.5	11.6
6	4	8	13.9	13.9
6	4	7	14.8	14.5
6	4	6	14.0	13.0
6	4	5	11.0	10.8
6	4	4	9.3	8.6
6	4	3	13.0	17.6
6	4	2	10.6	10.3
6	4	1	35.5	34.9
6	4	0	51.3	51.8
6	4	-1	43.2	39.8
6	4	-2	24.7	24.5
6	4	-3	14.5	14.0
6	4	-4	18.3	18.0
6	4	-5	5.9	5.5
6	4	-6	14.8	15.0
6	4	-7	12.2	12.3
6	4	-8	12.5	12.5
6	4	-9	11.4	11.4
6	4	-10	8.5	8.5
6	4	-11	2.9	2.5
6	3	11	6.6	6.2
6	3	10	9.9	9.8
6	3	9	9.6	9.1
6	3	8	2.9	3.1
6	3	7	10.3	10.6
6	3	6	14.9	14.7
6	3	5	25.0	24.7
6	3	4	21.5	21.7
6	3	3	34.5	34.6
6	3	2	31.6	31.4
6	3	1	46.9	46.2
6	3	0	6.1	6.1
6	3	-1	23.3	22.7
6	3	-2	39.1	37.9
6	3	-3	26.9	27.1
6	3	-4	39.0	40.4
6	3	-5	15.5	15.7
6	3	-6	16.1	16.1
6	3	-7	15.3	15.3
6	3	-8	9.5	9.3
6	3	-9	3.1	3.3
6	3	-10	7.4	7.4
6	3	-11	6.7	6.6
6	2	11	3.1	2.8
6	2	10	3.3	3.6
6	2	8	10.0	10.0
6	2	7	17.2	17.2
6	2	6	36.1	36.2
6	2	5	30.6	30.5
6	2	4	12.6	12.8
6	2	3	28.1	27.8
6	2	2	39.5	37.8
6	2	1	26.2	26.2
6	2	0	36.0	35.0
6	2	-1	40.1	33.6
6	2	-2	38.0	37.1
6	2	-3	34.1	32.9
6	2	-4	23.7	29.1
6	2	-5	24.1	24.0
6	2	-6	15.5	15.6
6	2	-7	29.4	29.6
6	2	-8	14.4	14.8
6	2	-9	7.3	7.3
6	2	-10	3.1	2.7
6	2	-11	3.3	3.1
6	1	11	6.5	6.6
6	1	10	9.3	8.6
6	1	9	9.1	8.9
6	1	8	9.9	9.6
6	1	7	14.5	14.1
6	1	6	11.9	11.8
6	1	5	26.4	26.4
6	1	4	42.5	42.6
6	1	3	36.1	36.2
6	1	2	44.3	44.6
6	1	1	27.0	26.8
6	1	0	10.5	10.6
6	1	-1	13.3	13.2
6	1	-2	28.6	27.5
6	1	-3	43.1	42.9
6	1	-4	37.5	38.0
6	1	-5	42.7	43.4
6	1	-6	26.3	26.7
6	1	-7	12.4	12.4
6	1	-8	10.7	11.0
6	1	-9	9.3	9.4
6	1	-10	6.7	6.5
6	1	-11	6.1	6.4

H	K	L	F OBS	F CALC
6	0	10	3.1	3.0
6	0	9	14.0	14.7
6	0	8	17.6	17.6
6	0	7	23.0	23.1
6	0	6	5.1	4.5
6	0	5	9.6	10.0
6	0	4	17.1	16.6
6	0	3	21.5	21.4
6	0	1	61.3	59.8
6	0	0	76.6	76.9
6	0	-1	70.3	72.3
6	0	-2	50.0	50.3
6	0	-3	4.4	4.7
6	0	-4	12.7	13.1
6	0	-5	6.7	6.5
6	0	-6	17.3	17.6
6	0	-7	3.1	3.7
6	0	-8	18.2	19.0
6	0	-9	16.2	16.7
6	0	-10	12.4	12.9
5	11	1	6.4	6.9
5	11	0	8.2	9.6
5	11	-1	7.5	8.2
5	11	-2	4.7	4.5
5	11	-3	2.9	3.1
5	10	6	2.7	3.0
5	10	5	4.9	4.9
5	10	4	5.7	6.1
5	10	3	6.1	5.5
5	10	2	7.9	7.8
5	10	1	7.4	7.7
5	10	0	2.8	2.2
5	10	-1	2.6	2.7
5	10	-2	9.3	9.2
5	10	-3	5.9	5.9
5	10	-4	8.5	9.4
5	10	-5	5.1	4.9
5	10	-6	3.6	3.9
5	9	7	6.3	5.7
5	9	6	9.7	9.3
5	9	5	6.6	6.7
5	9	3	6.3	6.7
5	9	2	10.9	10.8
5	9	1	7.3	7.7
5	9	0	7.0	7.1
5	9	-1	9.9	9.5
5	9	-2	11.0	11.1
5	9	-3	9.7	9.9
5	9	-4	7.4	7.5
5	9	-5	4.7	5.0
5	9	-6	7.4	7.7
5	9	-7	8.3	8.3
5	8	8	3.0	2.4
5	8	7	3.9	4.0
5	8	6	11.2	11.0
5	8	4	14.4	14.2
5	8	3	16.3	16.2
5	8	2	15.8	15.9
5	8	1	9.6	9.7
5	8	0	3.5	3.1
5	8	-1	8.9	8.8
5	8	-2	9.8	9.5
5	8	-3	14.1	14.4
5	8	-4	11.9	11.9
5	8	-5	13.1	13.2
5	8	-6	9.1	8.6
5	8	-7	3.7	3.4
5	8	-8	5.0	5.2
5	8	-9	2.9	2.7
5	7	9	7.8	8.0
5	7	8	9.5	9.2
5	7	7	8.4	9.0
5	7	6	2.4	2.2
5	7	5	2.7	3.1
5	7	4	7.6	7.6
5	7	3	5.6	5.4
5	7	2	5.2	4.9
5	7	1	20.4	20.2
5	7	0	31.6	31.7
5	7	-1	28.6	29.7
5	7	-2	17.8	17.5
5	7	-3	3.5	2.9
5	7	-4	4.7	4.7
5	7	-5	6.5	6.3
5	7	-6	4.2	4.2
5	7	-7	5.4	5.7
5	7	-8	9.2	9.2
5	7	-9	8.0	8.1
5	6	10	6.3	6.1
5	6	9	3.8	4.1
5	6	8	5.8	5.4
5	6	7	6.3	6.2
5	6	6	8.7	5.7
5	6	5	23.6	23.3
5	6	4	25.2	24.8
5	6	3	25.5	25.5
5	6	2	23.8	23.6
5	6	1	16.4	16.4
5	6	0	9.9	9.8
5	6	-1	10.0	10.0
5	6	-2	14.4	14.4
5	6	-3	19.9	19.6
5	6	-4	28.5	28.3
5	6	-5	20.0	19.6
5	6	-6	13.0	12.7
5	6	-7	6.5	6.2
5	6	-8	5.2	5.1
5	6	-9	5.4	5.4
5	6	-10	2.9	2.2
5	5	11	3.0	3.1
5	5	9	5.2	5.1
5	5	8	4.7	4.5
5	5	7	10.4	10.4
5	5	6	27.7	27.3
5	5	5	19.6	19.8
5	5	4	7.4	6.9
5	5	3	21.3	20.7
5	5	2	25.9	25.1
5	5	1	28.9	29.0
5	5	0	17.9	23.9
5	5	-1	24.9	24.9
5	5	-2	27.0	26.4
5	5	-3	33.9	33.3
5	5	-4	9.9	9.6
5	5	-6	19.2	19.3
5	5	-7	20.8	21.0
5	5	-8	13.1	13.0
5	5	-9	7.3	7.0
5	5	-11	3.6	3.2
5	4	11	9.1	4.9
5	4	10	8.9	8.9
5	4	9	9.9	9.5
5	4	8	4.6	4.3
5	4	7	9.6	9.2

H	K	L	F OBS	F CALC
5	4	6	15.5	15.9
5	4	5	9.0	9.3
5	4	4	25.7	25.9
5	4	3	31.8	34.9
5	4	2	41.4	41.5
5	4	1	37.8	37.5
5	4	0	16.1	15.7
5	4	-1	23.7	22.9
5	4	-2	39.5	38.9
5	4	-3	45.5	45.0
5	4	-4	21.9	21.8
5	4	-5	25.9	25.7
5	4	-6	16.5	16.4
5	4	-7	9.9	9.7
5	4	-8	12.4	12.1
5	4	-10	9.6	9.6
5	4	-11	6.4	6.1
5	3	11	3.2	2.4
5	3	10	3.7	3.7
5	3	9	13.9	13.9
5	3	8	18.9	19.0
5	3	7	19.6	19.7
5	3	6	17.0	15.4
5	3	5	16.0	16.0
5	3	4	13.1	10.1
5	3	3	18.3	19.3
5	3	2	19.4	19.0
5	3	1	41.6	41.3
5	3	0	69.9	69.1
5	3	-1	58.4	58.3
5	3	-2	42.4	42.0
5	3	-3	19.6	19.1
5	3	-4	19.9	19.2
5	3	-5	3.7	3.5
5	3	-6	17.2	17.0
5	3	-7	13.7	13.9
5	3	-8	17.3	17.2
5	3	-9	16.9	16.9
5	3	-10	10.9	11.0
5	3	-11	2.9	3.1
5	2	11	2.8	2.7
5	2	10	5.0	4.9
5	2	9	5.9	5.9
5	2	8	12.4	11.9
5	2	7	12.2	12.0
5	2	6	10.6	10.3
5	2	5	39.7	39.8
5	2	4	55.2	54.5
5	2	3	53.5	50.2
5	2	2	33.8	33.3
5	2	1	21.5	20.7
5	2	0	15.8	14.7
5	2	-1	8.2	7.2
5	2	-2	26.8	25.5
5	2	-3	26.0	25.9
5	2	-4	55.0	54.8
5	2	-5	52.7	52.8
5	2	-6	21.9	22.0
5	2	-7	10.1	9.7
5	2	-8	8.5	8.6
5	2	-9	9.5	9.9
5	2	-10	4.5	4.6
5	2	-11	4.1	4.1
5	2	-12	3.3	3.3
5	1	12	5.4	4.9
5	1	10	3.8	4.0
5	1	9	12.7	12.6
5	1	8	15.9	15.9
5	1	7	23.7	23.8
5	1	6	32.0	31.3
5	1	5	23.8	24.2
5	1	4	13.8	13.9
5	1	3	23.9	20.8
5	1	2	48.3	47.2
5	1	1	47.3	46.7
5	1	0	64.2	62.9
5	1	-1	53.6	50.2
5	1	-2	44.8	44.5
5	1	-3	29.7	30.5
5	1	-4	21.9	21.7
5	1	-5	14.1	13.9
5	1	-6	23.5	24.0
5	1	-7	25.5	25.9
5	1	-8	18.7	19.3
5	1	-9	13.5	13.5
5	1	-10	9.5	9.3
5	1	-11	3.7	3.2
5	0	12	4.2	4.1
5	0	11	10.3	10.3
5	0	10	12.0	12.0
5	0	9	9.7	9.5
5	0	8	3.4	2.9
5	0	7	23.8	20.9
5	0	6	15.5	15.1
5	0	5	21.5	21.5
5	0	4	23.3	23.6
5	0	3	40.1	47.9
5	0	2	78.2	77.2
5	0	1	63.7	63.5
5	0	0	24.9	24.9
5	0	-1	24.2	23.5
5	0	-2	44.8	46.7
5	0	-3	55.1	57.7
5	0	-4	33.5	34.7
5	0	-5	2.6	2.9
5	0	-6	25.4	25.6
5	0	-7	8.6	9.4
5	0	-8	5.7	4.5
5	0	-10	9.3	9.3
5	0	-11	12.3	12.5
5	0	-12	6.7	7.0
4	12	0	4.2	4.3
4	11	4	3.3	3.3
4	11	3	6.3	6.2
4	11	2	6.7	6.8
4	11	1	7.4	7.7
4	11	-2	6.7	7.6
4	11	-3	5.2	5.2
4	11	-4	4.1	3.9
4	11	-5	3.7	3.5
4	10	6	5.1	5.2
4	10	4	2.6	2.5
4	10	3	2.6	3.1
4	10	2	5.7	5.7
4	10	1	9.5	9.9
4	10	0	11.3	11.7
4	10	-1	11.9	12.0
4	10	-2	7.9	8.1
4	10	-4	3.7	3.5
4	10	-7	3.7	3.8
4	9	8	4.1	3.5
4	9	6	4.7	4.4
4	9	5	11.6	11.7
4	9	4	15.2	15.5

H	K	L	F OBS	F CALC
4	9	3	12.4	12.7
4	9	2	7.6	7.1
4	9	1	3.5	3.7
4	9	0	4.1	3.7
4	9	-2	6.1	6.5
4	9	-3	8.7	9.0
4	9	-4	14.1	14.5
4	9	-5	12.2	12.1
4	9	-6	9.1	9.0
4	9	-8	3.2	3.1
4	8	9	5.4	5.1
4	8	8	6.9	7.4
4	8	7	9.3	9.3
4	8	6	12.4	12.5
4	8	5	10.1	10.1
4	8	4	7.0	7.0
4	8	3	7.1	7.1
4	8	2	12.0	12.0
4	8	1	17.2	17.2
4	8	0	21.5	21.7
4	8	-1	19.4	19.5
4	8	-2	13.5	12.9
4	8	-3	13.0	12.6
4	8	-4	4.5	4.6
4	8	-5	11.5	11.4
4	8	-7	9.3	8.9
4	8	-8	9.4	6.5
4	8	-9	4.5	4.4
4	7	10	7.4	7.5
4	7	9	5.6	5.4
4	7	8	3.6	3.5
4	7	7	6.1	6.7
4	7	6	9.7	9.0
4	7	5	7.6	7.8
4	7	4	10.3	10.6
4	7	3	17.3	17.7
4	7	2	27.3	27.5
4	7	1	25.3	24.9
4	7	0	8.1	9.0
4	7	-1	16.3	16.9
4	7	-2	22.5	22.2
4	7	-3	25.2	24.9
4	7	-4	15.6	15.8
4	7	-5	10.2	10.1
4	7	-6	9.7	9.8
4	7	-7	8.2	8.5
4	7	-8	5.6	5.5
4	7	-10	4.3	4.6
4	6	9	6.8	6.5
4	6	8	9.4	9.3
4	6	7	16.4	16.1
4	6	6	26.3	25.9
4	6	5	13.9	13.8
4	6	4	8.3	8.4
4	6	3	13.8	13.6
4	6	2	21.3	20.9
4	6	1	27.2	27.3
4	6	0	26.6	26.7
4	6	-1	30.2	30.2
4	6	-2	28.3	28.4
4	6	-3	22.5	22.0
4	6	-4	10.3	9.8
4	6	-5	11.1	11.2
4	6	-6	14.0	13.7
4	6	-7	14.3	14.1
4	6	-8	12.7	12.6
4	6	-9	8.7	8.6
4	5	11	4.2	4.2
4	5	10	4.5	4.1
4	5	9	4.5	4.6
4	5	8	9.4	8.9
4	5	7	7.6	7.0
4	5	6	8.3	8.4
4	5	5	28.7	28.6
4	5	4	37.1	37.4
4	5	3	34.9	34.8
4	5	2	23.9	23.1
4	5	1	7.6	7.4
4	5	0	9.6	9.0
4	5	-1	5.9	5.6
4	5	-2	12.5	12.4
4	5	-3	25.0	24.8
4	5	-4	40.6	40.3
4	5	-5	36.0	36.3
4	5	-6	19.8	18.5
4	5	-7	5.5	5.6
4	5	-8	4.8	4.9
4	5	-9	8.5	8.6
4	5	-10	4.7	4.9
4	5	-11	2.7	2.6
4	4	10	5.1	5.1
4	4	9	12.6	12.6
4	4	8	17.9	18.0
4	4	7	17.4	17.5
4	4	6	9.1	9.2
4	4	5	24.6	25.0
4	4	4	8.0	7.5
4	4	3	16.5	17.2
4	4	2	23.9	21.5
4	4	1	42.8	43.0
4	4	0	77.5	77.5
4	4	-1	55.6	56.2
4	4	-2	31.2	30.6
4	4	-3	20.7	20.7
4	4	-4	14.5	13.6
4	4	-5	6.5	6.7
4	4	-6	14.3	14.2
4	4	-7	14.5	14.6
4	4	-8	13.5	13.3
4	4	-9	15.6	15.6
4	4	-10	9.5	9.5
4	3	11	11.2	10.9
4	3	10	10.3	13.4
4	3	9	8.3	8.5
4	3	8	5.7	5.5
4	3	7	10.7	10.3
4	3	6	20.2	20.2
4	3	5	21.3	20.6
4	3	4	33.4	33.3
4	3	3	44.5	44.5
4	3	2	53.1	53.9
4	3	1	46.5	45.9
4	3	0	9.3	9.0
4	3	-1	35.0	34.3
4	3	-2	52.7	52.8
4	3	-3	56.5	55.9
4	3	-4	33.2	33.2
4	3	-5	31.3	30.4
4	3	-6	18.0	17.8
4	3	-7	13.7	13.7
4	3	-8	6.7	6.3
4	3	-9	7.4	7.2
4	3	-10	8.9	9.0
4	3	-11	10.7	10.7

H	K	L	F OBS	F CALC
4	3	-12	4.9	4.9
4	2	12	2.9	2.6
4	2	10	3.5	3.7
4	2	9	4.3	4.2
4	2	8	13.7	13.9
4	2	7	29.2	28.9
4	2	6	46.5	46.4
4	2	5	22.3	21.9
4	2	4	15.0	13.7
4	2	3	40.6	39.4
4	2	2	54.5	54.3
4	2	1	57.1	55.5
4	2	0	29.2	29.0
4	2	-1	45.5	45.0
4	2	-2	65.4	64.1
4	2	-3	45.1	44.8
4	2	-4	27.0	26.1
4	2	-5	15.2	15.2
4	2	-6	31.3	32.0
4	2	-7	31.4	31.8
4	2	-8	21.6	21.9
4	2	-9	6.2	5.0
4	2	-10	3.4	3.5
4	2	-11	4.2	4.0
4	1	12	5.7	5.9
4	1	11	8.1	9.3
4	1	10	10.9	10.5
4	1	9	13.4	13.2
4	1	8	12.2	11.5
4	1	7	15.7	15.7
4	1	6	12.4	12.7
4	1	5	37.7	38.3
4	1	4	52.9	53.3
4	1	3	55.5	55.7
4	1	2	59.5	58.4
4	1	1	34.8	34.3
4	1	0	24.3	22.8
4	1	-1	47.1	44.3
4	1	-2	34.4	34.2
4	1	-3	56.1	57.0
4	1	-4	48.7	49.5
4	1	-5	18.7	19.8
4	1	-6	32.7	33.3
4	1	-7	14.9	14.6
4	1	-8	7.3	6.9
4	1	-9	7.3	7.1
4	1	-10	7.2	7.3
4	1	-11	7.4	7.5
4	1	-12	6.6	6.6
4	0	12	6.1	5.9
4	0	10	6.7	7.3
4	0	9	21.9	22.0
4	0	8	27.9	29.0
4	0	7	21.7	21.7
4	0	6	16.3	17.2
4	0	5	15.3	15.6
4	0	4	19.1	19.9
4	0	3	21.7	21.4
4	0	2	11.7	11.2
4	0	1	57.2	57.3
4	0	0	110.4	115.5
4	0	-1	102.0	106.4
4	0	-2	35.7	37.5
4	0	-3	10.7	10.4
4	0	-4	11.6	12.0
4	0	-5	4.4	5.1
4	0	-6	12.0	12.5
4	0	-7	14.6	15.7
4	0	-8	21.6	21.4
4	0	-9	24.3	24.3
4	0	-10	16.1	16.2
3	12	1	2.5	2.2
3	11	5	5.2	5.0
3	11	2	3.4	3.4
3	11	1	6.4	6.7
3	11	0	10.1	10.5
3	11	-1	8.5	8.9
3	11	-2	3.5	3.7
3	10	6	4.7	4.8
3	10	5	5.6	6.2
3	10	4	6.3	6.2
3	10	3	9.7	10.0
3	10	2	9.7	9.8
3	10	1	8.4	8.4
3	10	0	3.3	3.4
3	10	-1	5.2	5.4
3	10	-2	10.6	10.7
3	10	-3	9.9	9.7
3	10	-4	9.0	8.6
3	10	-5	6.2	6.3
3	10	-6	4.9	4.9
3	9	8	3.9	3.2
3	9	7	8.5	8.4
3	9	6	11.5	11.9
3	9	5	6.2	6.3
3	9	3	9.4	9.4
3	9	2	11.3	11.5
3	9	1	11.2	11.6
3	9	0	8.5	9.1
3	9	-1	10.6	11.1
3	9	-2	14.1	13.8
3	9	-3	10.0	10.1
3	9	-4	7.2	7.1
3	9	-5	4.7	4.5
3	9	-6	8.5	8.1
3	9	-7	10.6	10.5
3	9	-8	6.3	6.0
3	8	8	4.9	4.7
3	8	7	4.7	4.6
3	8	6	4.5	4.2
3	8	5	12.2	11.9
3	8	4	18.7	19.2
3	8	3	16.1	16.2
3	8	2	19.6	19.7
3	8	1	12.5	12.6
3	8	0	2.7	2.9
3	8	-1	12.0	11.9
3	8	-2	11.9	12.0
3	8	-3	19.2	18.7
3	8	-4	18.7	19.1
3	8	-5	14.3	14.3
3	8	-6	8.0	7.9
3	8	-7	5.1	4.9
3	8	-8	4.9	5.1
3	8	-9	3.9	3.4
3	7	10	4.4	4.6
3	7	9	9.6	9.6
3	7	8	10.2	10.3
3	7	7	7.9	7.7
3	7	6	3.6	3.6
3	7	5	3.4	3.7
3	7	4	6.2	6.2
3	7	3	6.7	6.5
3	7	2	10.6	11.2

H	K	L	F OBS	F CALC
3	7	1	26.7	26.5
3	7	0	41.8	42.7
3	7	-1	28.3	28.7
3	7	-2	23.3	23.3
3	7	-3	5.1	4.9
3	7	-4	6.1	6.4
3	7	-5	4.5	4.8
3	7	-6	5.4	5.6
3	7	-7	5.9	5.7
3	7	-8	9.9	9.6
3	7	-9	10.9	10.8
3	7	-10	7.0	7.3
3	6	11	3.3	3.3
3	6	10	5.9	6.0
3	6	9	7.6	7.0
3	6	8	5.2	4.9
3	6	7	9.5	9.3
3	6	6	12.2	12.4
3	6	5	23.6	23.5
3	6	4	32.4	32.1
3	6	3	32.2	32.7
3	6	2	23.5	23.7
3	6	1	15.3	15.9
3	6	0	12.3	12.0
3	6	-1	14.5	14.4
3	6	-2	21.9	22.3
3	6	-3	27.7	27.1
3	6	-4	26.4	27.6
3	6	-5	27.8	27.5
3	6	-6	16.9	16.9
3	6	-7	5.3	5.4
3	6	-8	4.5	4.7
3	6	-9	6.3	6.2
3	6	-10	4.8	5.2
3	6	-11	4.6	4.5
3	5	11	2.9	3.1
3	5	10	3.7	3.5
3	5	9	4.4	4.5
3	5	8	12.1	12.3
3	5	7	22.0	21.9
3	5	6	30.3	30.5
3	5	5	23.3	23.4
3	5	4	5.8	5.4
3	5	3	25.6	25.6
3	5	2	30.1	30.6
3	5	1	33.7	33.9
3	5	0	26.6	26.7
3	5	-1	40.8	40.4
3	5	-2	29.3	29.5
3	5	-3	34.7	34.2
3	5	-4	12.0	11.5
3	5	-5	14.4	13.7
3	5	-6	25.5	26.0
3	5	-7	26.2	26.3
3	5	-8	15.7	15.6
3	5	-9	7.6	7.3
3	5	-10	4.5	4.1
3	4	11	10.0	10.0
3	4	10	12.1	12.2
3	4	9	6.7	6.5
3	4	8	6.6	6.6
3	4	7	11.6	11.6
3	4	6	13.7	13.7
3	4	5	29.8	29.0
3	4	4	36.5	35.6
3	4	3	49.1	49.2
3	4	2	71.4	70.3
3	4	1	49.4	49.1
3	4	0	4.7	4.3
3	4	-1	26.5	26.2
3	4	-2	49.6	45.4
3	4	-3	44.7	44.7
3	4	-4	39.5	38.9
3	4	-5	25.7	25.5
3	4	-6	17.5	17.5
3	4	-7	17.2	17.5
3	4	-8	6.9	6.5
3	4	-9	4.9	5.1
3	4	-10	9.9	9.5
3	4	-11	10.8	11.0
3	4	-12	5.8	5.7
3	3	12	5.2	5.1
3	3	11	2.7	1.9
3	3	10	9.2	9.4
3	3	9	15.5	15.5
3	3	8	25.2	25.0
3	3	7	22.0	21.6
3	3	6	31.5	31.8
3	3	5	15.2	15.5
3	3	4	21.6	21.5
3	3	3	24.2	23.7
3	3	2	28.1	28.0
3	3	1	55.2	55.7
3	3	0	81.5	82.3
3	3	-1	78.9	79.9
3	3	-2	34.2	34.7
3	3	-3	32.1	31.2
3	3	-4	15.5	15.5
3	3	-5	15.6	16.2
3	3	-6	18.9	19.3
3	3	-7	20.7	20.8
3	3	-8	25.2	25.4
3	3	-9	19.1	19.0
3	3	-10	11.6	12.3
3	3	-11	3.4	4.2
3	3	-12	3.3	3.6
3	2	12	5.2	5.1
3	2	11	3.6	3.6
3	2	10	6.1	5.9
3	2	9	10.9	10.6
3	2	8	14.3	13.6
3	2	7	10.5	10.2
3	2	6	23.6	23.6
3	2	5	59.7	59.7
3	2	4	63.5	65.2
3	2	3	79.5	80.7
3	2	2	26.8	27.6
3	2	1	8.5	8.7
3	2	0	24.5	23.8
3	2	-1	5.3	4.2
3	2	-2	32.7	31.5
3	2	-3	45.2	46.3
3	2	-4	61.9	62.8
3	2	-5	55.2	56.5
3	2	-6	34.2	34.4
3	2	-7	4.0	3.8
3	2	-8	5.7	5.5
3	2	-9	14.6	15.0
3	2	-10	5.6	4.9
3	2	-11	5.2	4.9
3	2	-12	4.3	4.3
3	1	12	5.2	5.3
3	1	11	2.7	2.7
3	1	10	7.1	7.0
3	1	9	13.6	13.7
3	1	8	22.1	22.2
3	1	7	29.5	30.1
3	1	6	35.0	35.0
3	1	5	25.3	25.7
3	1	4	21.2	21.3
3	1	3	33.9	34.3
3	1	2	50.1	50.0
3	1	1	64.9	64.9
3	1	0	80.5	81.8
3	1	-1	57.4	59.8
3	1	-2	79.5	79.2
3	1	-3	36.3	36.5
3	1	-4	15.1	15.1
3	1	-5	16.7	17.7
3	1	-6	32.7	33.4
3	1	-7	31.3	32.0
3	1	-8	21.0	21.3
3	1	-9	15.9	16.2
3	1	-10	13.7	14.0
3	1	-11	3.5	3.6
3	0	12	5.5	5.9
3	0	11	13.2	13.5
3	0	10	15.7	15.6
3	0	9	8.6	8.3
3	0	7	23.7	23.7
3	0	6	20.2	20.3
3	0	5	6.6	7.1
3	0	4	65.0	63.3
3	0	3	31.9	33.6
3	0	2	96.1	98.1
3	0	1	73.7	74.1
3	0	0	2.2	1.2
3	0	-1	65.5	65.3
3	0	-2	45.3	47.4
3	0	-3	61.0	62.5
3	0	-4	37.4	38.5
3	0	-5	19.0	19.2
3	0	-6	14.9	15.5
3	0	-7	19.0	18.9
3	0	-8	8.6	8.5
3	0	-10	16.2	16.5
3	0	-11	11.3	11.9
3	0	-12	9.7	9.9
2	12	3	2.8	2.8
2	12	2	4.7	5.0
2	12	1	2.9	2.8
2	12	0	4.1	4.0
2	12	-1	4.1	4.0
2	12	-2	4.9	5.0
2	12	-3	3.5	4.2
2	11	4	5.0	4.8
2	11	3	7.0	7.1
2	11	2	7.0	7.2
2	11	1	5.2	5.4
2	11	-1	5.1	5.2
2	11	-2	7.9	7.4
2	11	-3	8.4	8.4
2	11	-4	3.5	4.1
2	10	7	3.4	3.8
2	10	6	5.6	5.9
2	10	4	3.8	4.1
2	10	3	2.5	3.2
2	10	2	4.2	4.0
2	10	1	11.5	12.1
2	10	0	12.1	12.8
2	10	-1	16.0	16.2
2	10	-2	5.3	5.1
2	10	-3	5.1	5.1
2	10	-4	3.2	2.8
2	10	-6	4.7	4.7
2	10	-7	4.7	5.0
2	9	8	4.3	4.1
2	9	6	4.9	5.2
2	9	5	13.7	13.9
2	9	4	15.0	18.4
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2	8	1	19.6	19.9
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0	4	6	16.7	16.7
0	4	5	13.6	13.5
0	4	4	12.5	13.6
0	4	3	32.5	32.0
0	4	2	23.8	23.4
0	4	1	67.8	69.0
0	4	0	105.2	110.2
0	3	12	5.1	5.5
0	3	11	12.5	12.3
0	3	10	11.9	11.9
0	3	9	9.7	9.6
0	3	8	5.7	5.8
0	3	7	18.0	19.2
0	3	6	28.7	25.4
0	3	5	35.0	35.0
0	3	4	45.8	45.8
0	3	3	45.2	46.2
0	3	2	68.3	69.0
0	3	1	63.5	63.1
0	2	11	5.4	5.1
0	2	10	4.9	4.6
0	2	9	9.0	9.3
0	2	8	17.0	16.9
0	2	7	31.9	32.1
0	2	6	53.5	52.6
0	2	5	34.8	34.7
0	2	4	5.0	1.7
0	2	3	63.9	62.6
0	2	2	85.9	87.2
0	2	1	116.5	124.6
0	2	0	45.6	43.8
0	1	12	7.6	7.5
0	1	11	9.3	9.1
0	1	10	14.3	14.3
0	1	9	10.4	10.6
0	1	8	8.2	8.4
0	1	7	16.1	16.2
0	1	6	36.3	35.9
0	1	5	42.6	43.1
0	1	4	80.5	82.9
0	1	3	68.8	71.3
0	1	2	49.9	50.4
0	1	1	67.8	67.3
0	0	12	4.2	4.0
0	0	10	10.0	10.3
0	0	9	25.0	25.0
0	0	8	31.1	31.1
0	0	7	25.0	25.7
0	0	5	15.4	15.5
0	0	4	12.6	12.5
0	0	3	7.8	7.7
0	0	2	67.3	69.4
0	0	1	60.9	65.6

TABLE 2

π-Cyclopentadienyl(diphenylamido)iodonitrosylchromium; fractional atomic coordinates with esd in parentheses and thermal parameters.

Atom	x	y	z	U_{iso}($\times 10^3 Å^2$)
Cr	0.23779(4)	1.00000	0.33700(5)	*
I	0.24376(2)	1.28244(6)	0.43524(2)	*
N(1)	0.2948(2)	1.0281(3)	0.1455(3)	*
C(2)	0.4278(3)	1.0380(4)	0.1066(4)	*
C(3)	0.4701(3)	0.9780(5)	-0.0253(4)	*
C(4)	0.6024(4)	0.9856(5)	-0.0590(5)	*
C(5)	0.6927(4)	1.0496(5)	0.0398(6)	*
C(6)	0.6508(3)	1.1081(5)	0.1666(6)	*
C(7)	0.5194(3)	1.1055(5)	0.2014(4)	*
C(8)	0.2037(3)	1.0288(4)	0.0246(3)	*
C(9)	0.1189(4)	0.9076(5)	0.0004(4)	*
C(10)	0.0302(4)	0.9103(6)	-0.1172(5)	*
C(11)	0.0243(4)	1.0324(7)	-0.2075(5)	*
C(12)	0.1075(5)	1.1533(6)	-0.1836(5)	*
C(13)	0.1990(4)	1.1521(5)	-0.0686(4)	*
C(14)	0.2002(6)	0.8204(7)	0.4991(7)	*
C(15)	0.2654(6)	0.7571(5)	0.3782(7)	*
C(16)	0.3912(5)	0.8206(6)	0.3786(6)	*
C(17)	0.4031(5)	0.9215(7)	0.4910(6)	*
C(18)	0.2854(7)	0.9234(8)	0.5650(5)	*
N(19)	0.0773(3)	1.0155(4)	0.3003(3)	*
O(20)	-0.0379(3)	1.0203(5)	0.2895(4)	*
H(3)	0.415(5)	0.950(7)	-0.104(6)	36
H(4)	0.633(6)	0.936(8)	-0.150(7)	51
H(5)	0.778(5)	1.059(7)	0.025(6)	38
H(6)	0.708(6)	1.173(8)	0.259(7)	50
H(7)	0.491(5)	1.165(6)	0.288(5)	26
H(9)	0.126(5)	0.836(6)	0.057(5)	26
H(10)	-0.027(7)	0.841(9)	-0.135(8)	61
H(11)	-0.032(5)	1.034(7)	-0.298(6)	36
H(12)	0.110(6)	1.239(7)	-0.242(6)	48
H(13)	0.260(6)	1.228(8)	-0.050(6)	47

Atom	x	y	z	$U_{iso}(\times 10^3 Å^2)$
H(14)	0.097(7)	0.786(15)	0.514(8)	75
H(15)	0.222(10)	0.702(13)	0.294(11)	95
H(16)	0.449(6)	0.785(13)	0.302(7)	62
H(17)	0.472(11)	0.971(13)	0.470(11)	101
H(18)	0.270(5)	0.990(7)	0.620(5)	35

* These atoms were assigned anisotropic temperature factors of the form:

$$T = \exp\left[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}\ell^2c^{*2} + 2U_{23}k\ell b^*c^* + 2U_{31}\ell hc^*a^* + 2U_{12}hka^*b^*)\right]$$

with final parameters ($U_{ij} \times 10^4 Å^2$):

Atom	U_{11}	U_{22}	U_{33}	$2U_{23}$	$2U_{31}$	$2U_{12}$
Cr	347	489	406	78	32	81
I	532	581	579	-74	40	21
N(1)	362	467	434	-32	41	-8
C(2)	367	473	519	-18	69	18
C(3)	455	560	579	-173	151	69
C(4)	524	618	758	-162	402	148
C(5)	430	573	980	-125	426	36
C(6)	379	633	898	-217	45	-29
C(7)	389	598	637	-155	14	-31
C(8)	362	464	436	-60	52	23
C(9)	494	556	541	-30	-75	-153
C(10)	472	833	653	-258	-64	-147
C(11)	540	947	557	-163	-101	245
C(12)	790	641	538	94	-30	459
C(13)	622	473	526	-32	60	87
C(14)	746	944	937	1009	375	257
C(15)	954	515	888	294	-185	51
C(16)	688	683	833	311	210	548
C(17)	673	751	865	292	-463	340
C(18)	1057	955	509	466	159	835
N(19)	396	527	538	34	108	6
O(20)	343	816	1028	-35	123	-14

<u>Average estimated standard deviations</u>. (U_{ij}x10^4Å^2) <u>and</u> (U_{iso}x10^3Å^2)

Atom	U_{11} or U_{iso}	U_{22}	U_{33}	$2U_{23}$	$2U_{31}$	$2U_{12}$
Cr	2	2	2	4	3	4
I	1	1	1	2	1	2
N	10	13	11	19	16	18
O	10	22	23	39	23	25
C	19	25	22	38	32	36
H	16					

TABLE 3

π-Cyclopentadienyl(diphenylamido)iodonitrosylchromium; bond lengths (Å) with esd in parentheses.

Cr	-	I	2.6694(5)	C(13)	-	C(8)	1.394(5)
Cr	-	N(1)	1.894(3)	C(14)	-	C(15)	1.432(9)
Cr	-	N(19)	1.676(3)	C(15)	-	C(16)	1.407(8)
Cr	-	C(14)	2.229(7)	C(16)	-	C(17)	1.375(8)
Cr	-	C(15)	2.210(5)	C(17)	-	C(18)	1.401(8)
Cr	-	C(16)	2.265(5)	C(18)	-	C(14)	1.394(9)
Cr	-	C(17)	2.292(6)	C(3)	-	H(3)	0.94(6)
Cr	-	C(18)	2.254(5)	C(4)	-	H(4)	1.00(6)
N(19)	-	O(20)	1.183(4)	C(5)	-	H(5)	0.89(6)
N(1)	-	C(2)	1.421(4)	C(6)	-	H(6)	1.17(7)
N(1)	-	C(8)	1.436(4)	C(7)	-	H(7)	1.01(5)
C(2)	-	C(3)	1.409(5)	C(9)	-	H(9)	0.83(5)
C(3)	-	C(4)	1.400(5)	C(10)	-	H(10)	0.87(8)
C(4)	-	C(5)	1.403(6)	C(11)	-	H(11)	1.00(5)
C(5)	-	C(6)	1.361(7)	C(12)	-	H(12)	0.93(6)
C(6)	-	C(7)	1.393(5)	C(13)	-	H(13)	0.93(7)
C(7)	-	C(2)	1.402(5)	C(14)	-	H(14)	1.11(8)
C(8)	-	C(9)	1.399(5)	C(15)	-	H(15)	1.01(11)
C(9)	-	C(10)	1.398(6)	C(16)	-	H(16)	0.98(7)
C(10)	-	C(11)	1.369(8)	C(17)	-	H(17)	0.86(11)
C(11)	-	C(12)	1.386(8)	C(18)	-	H(18)	0.80(7)
C(12)	-	C(13)	1.398(6)				

TABLE 4

π-Cyclopentadienyl(diphenylamido)iodonitrosylchromium; valency angles (°) with esd in parentheses.

N(1) - Cr - N(19)	97.2(1)	C(12) - C(13) - C(8)	119.2(4)
I - Cr - N(1)	100.9(1)	C(18) - C(14) - C(15)	107.4(5)
I - Cr - N(19)	90.3(1)	C(14) - C(15) - C(16)	106.7(5)
I - Cr - C(14)	116.6(2)	C(15) - C(16) - C(17)	109.1(5)
I - Cr - C(15)	149.2(2)	C(16) - C(17) - C(18)	108.4(5)
I - Cr - C(16)	126.5(1)	C(17) - C(18) - C(14)	108.5(5)
I - Cr - C(17)	93.8(2)	C(2) - C(3) - H(3)	125(3)
I - Cr - C(18)	87.9(2)	C(4) - C(3) - H(3)	114(3)
N(1) - Cr - C(14)	141.7(2)	C(3) - C(4) - H(4)	119(4)
N(1) - Cr - C(15)	104.4(2)	C(5) - C(4) - H(4)	120(4)
N(1) - Cr - C(16)	91.2(2)	C(4) - C(5) - H(5)	125(4)
N(1) - Cr - C(17)	112.3(2)	C(6) - C(5) - H(5)	115(4)
N(1) - Cr - C(18)	148.0(2)	C(5) - C(6) - H(6)	130(3)
N(19) - Cr - C(14)	90.5(2)	C(7) - C(6) - H(6)	108(3)
N(19) - Cr - C(15)	103.6(2)	C(6) - C(7) - H(7)	119(3)
N(19) - Cr - C(16)	139.9(2)	C(2) - C(7) - H(7)	121(3)
N(19) - Cr - C(17)	148.9(2)	C(8) - C(9) - H(9)	116(4)
N(19) - Cr - C(18)	113.7(2)	C(10) - C(9) - H(9)	124(3)
Cr - N(19) - O(20)	172.7(3)	C(9) - C(10) - H(10)	124(5)
Cr - N(1) - C(2)	124.4(2)	C(11) - C(10) - H(10)	115(5)
Cr - N(1) - C(8)	121.1(2)	C(10) - C(11) - H(11)	122(4)
C(2) - N(1) - C(8)	114.3(3)	C(12) - C(11) - H(11)	117(4)
N(1) - C(2) - C(3)	121.0(3)	C(11) - C(12) - H(12)	124(4)
N(1) - C(2) - C(7)	120.0(3)	C(13) - C(12) - H(12)	115(4)
C(7) - C(2) - C(3)	119.0(3)	C(12) - C(13) - H(13)	125(4)
C(2) - C(3) - C(4)	119.7(4)	C(8) - C(13) - H(13)	116(4)
C(3) - C(4) - C(5)	120.1(4)	C(18) - C(14) - H(14)	135(5)
C(4) - C(5) - C(6)	119.9(4)	C(15) - C(14) - H(14)	117(5)
C(5) - C(6) - C(7)	121.3(4)	C(14) - C(15) - H(15)	126(6)
C(6) - C(7) - C(2)	119.9(4)	C(16) - C(15) - H(15)	125(6)
N(1) - C(8) - C(9)	120.6(3)	C(15) - C(16) - H(16)	116(5)
N(1) - C(8) - C(13)	119.7(3)	C(17) - C(16) - H(16)	135(5)
C(13) - C(8) - C(9)	119.7(3)	C(16) - C(17) - H(17)	103(7)
C(8) - C(9) - C(10)	120.0(4)	C(18) - C(17) - H(17)	146(7)
C(9) - C(10) - C(11)	120.3(5)	C(17) - C(18) - H(18)	121(4)
C(10) - C(11) - C(12)	120.0(4)	C(14) - C(18) - H(18)	129(4)
C(11) - C(12) - C(13)	120.8(4)		

TABLE 5

π-Cyclopentadienyl(diphenylamido)iodonitrosylchromium; mean plane calculations.

	Atoms in plane	Displacements (Å)	Atoms out of plane	Displacements (Å)
a)	Cr		N(1)	-0.039
	C(2)			
	C(8)			
b)	N(1)		Cr	0.134
	C(2)			
	C(8)			
c)	C(2)	-0.009	N(1)	-0.063
	C(3)	-0.004	H(3)	0.17
	C(4)	0.012	H(4)	-0.05
	C(5)	-0.008	H(5)	0.01
	C(6)	-0.005	H(6)	0.05
	C(7)	0.013	H(7)	0.17
			Cr	-0.984
d)	C(8)	-0.003	N(1)	-0.024
	C(9)	-0.006	H(9)	0.00
	C(10)	0.008	H(10)	-0.04
	C(11)	-0.001	H(11)	0.09
	C(12)	-0.007	H(12)	0.00
	C(13)	0.009	H(13)	0.05
			Cr	-1.380
e)	C(14)	-0.012	H(14)	0.04
	C(15)	0.011	H(15)	0.26
	C(16)	-0.006	H(16)	-0.02
	C(17)	-0.002	H(17)	0.20
	C(18)	0.009	H(18)	0.19
			Cr	1.906
f)	Cr		O(20)	-0.151
	I			
	N(19)			

The dihedral angles (°) between selected planes are:

c) - d) 77.5   b) - c) 29.5   b) - d) 60.6

TABLE 6

π-Cyclopentadienyl(diphenylamido)iodonitrosylchromium; intermolecular contacts ($\leqslant$3.80Å).

O(20)	---	C(6^{I})	3.45	O(20)	---	C(14^{V})	3.72
O(20)	---	C(12^{II})	3.47	C(10)	---	C(13^{II})	3.73
O(20)	---	C(5^{I})	3.56	C(3)	---	C(6^{IV})	3.74
C(11)	---	C(18^{III})	3.58	C(5)	---	C(9^{VI})	3.75
C(12)	---	C(18^{III})	3.62	C(4)	---	C(13^{IV})	3.77
C(9)	---	C(12^{II})	3.68	C(6)	---	C(18^{VII})	3.79
C(3)	---	C(7^{IV})	3.69	C(11)	---	C(14^{III})	3.79
C(5)	---	C(13^{IV})	3.71				

The superscripts refer to the following transformations of the atomic coordinates:

I	$-1 + x$,	y,	z;	V	$- x$,	$\frac{1}{2} + y$,	$1 - z$;
II	$- x$,	$-\frac{1}{2} + y$,	$- z$;	VI	$1 - x$,	$\frac{1}{2} + y$,	$- z$;
III	x,	y,	$-1 + z$;	VII	$1 - x$,	$\frac{1}{2} + y$,	$1 - z$.
IV	$1 - x$,	$-\frac{1}{2} + y$,	$- z$;				

III.1.3 DISCUSSION

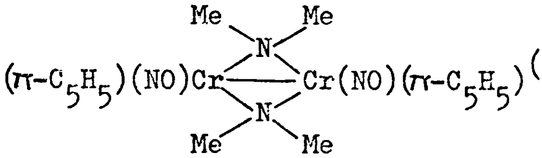
The discussion refers to the molecular geometry obtained neglecting anomalous dispersion in the calculations, and although the standard deviations derived from the least-squares refinement are underestimated by a factor of about three in some cases, because they allow only for the random experimental errors and not for the systematic error of neglecting the anomalous dispersion, none of the comparisons or conclusions would be invalid if the correct absolute configuration was known.

The intermolecular contacts are normal van der Waals' interactions consistent with the monomeric formulation, $(\pi\text{-}C_5H_5)Cr(NO)(NPh_2)I$. The formal oxidation state and the number of valence electrons assigned to the chromium atom in the above formula is dependent on the nature of the ligands. For instance, nitrosyl ligands are regarded as NO^+ or NO^- ligands depending whether the metal - N - 0 valency angle is about 180°, linear type, or about 120°, bent type.(18) The bonding in linearly coordinated nitrosyls is analogous to that in carbonyl ligands, with σ-donation of an electron pair from NO^+, isoelectronic with CO, to the metal together with back-donation from filled metal d orbitals to empty doubly degenerate $\pi^*(NO)$ levels, whereas the bonding in bent nitrosyls is considered primarily as σ-donation of an electron pair from NO^- to the metal. In this analysis, the Cr - N - 0 angle is of the linear type, 172.7(3)°, therefore the ligand is regarded as the 2-electron donor NO^+. Customarily, cyclopentadienyl and iodo ligands are considered to be 6-electron, $C_5H_5^-$, and 2-electron, I^-, donors respectively, therefore with the arbitrary and somewhat extreme assignment of the diphenylamido ligand as $^-NPh_2$ or $^+NPh_2$, $(\pi\text{-}C_5H_5)Cr(NO)(NPh_2)I$ can be considered as a Cr^{II} complex with 16 metal valence electrons or a Cr^{0}

complex with 18 metal valence electrons.

The Cr - NPh_2 bond, 1.894(3) Å, although considerably longer than tha Cr - NO bond, 1.676(3) Å, still has some double-bond character compared with the Cr - NCO bond, 1.982 Å, in $(\pi\text{-}C_5H_5)Cr(NO)_2(NCO)$(2) and the Cr - N bonds, average 2.185 Å, in diethylenetriamine $Cr(CO)_3$(19), both of which have been regarded as essentially single bonds. It seems more reasonable to explain this double-bond character by overlap of suitable filled metal orbitals with the vacant lone pair orbital of a $^{+}NPh_2$ ligand, than by the interaction of the lone pair of a $^{-}NPh_2$ ligand with vacant metal orbitals, because the depletion of electron density on the metal in the former formulation is consistent with that due to metal$\longrightarrow$ligand back-donation in the Cr - NO linkage. The metal $d\pi \longrightarrow$ ligand $p\pi$ bonding in the Cr - NPh_2 linkage is also reflected in the approximate sp^2 hybridisation of the nitrogen atom, which is displaced only 0.039 Å from the plane defined by its three bonded atoms. Corresponding planarities with similar Cr - N bond lengths, average 1.87 Å, are found in $Cr(NPr^i{}_2)_3$(3), in which the other extreme formulation of the ligand, i.e. $^{-}NPr^i{}_2$, has been considered the more appropriate, with the double-bond character therefore attributed to ligand$\longrightarrow$metal π-bonding.

Although, as a result of the planarity, the sum of the valency angles about the nitrogen atom of the diphenylamido ligand, 358.8°, is nearly 360°, the individual angles deviate significantly from 120°. The difference between the Cr - N - C(2) angle, 124.4(2)°, and the Cr - N - C(8) angle, 121.1(2)°, is presumably due to packing requirements, but differences between these and the C(2) - N - C(8) angle, 114.3(3)°, probably reflect electronegativity differences between the chromium and carbon atoms resulting in the bond to the chromium atom having more s-character than those to the carbon atoms. This argument has been used previously to explain similar deviations from ideal hybridisation about the bridging nitrogen atoms in



$(\pi\text{-}C_5H_5)(NO)Cr\overset{\overset{Me\quad Me}{N}}{\underset{\underset{Me\quad Me}{N}}{=\!=\!=}}Cr(NO)(\pi\text{-}C_5H_5)$ (7).

Phenyl rings C(2) - - - C(7) and C(8) - - - C(13) are effectively planar, the root-mean-square deviations from planarity being 0.009 and 0.006 Å respectively, with mean C - C bond lengths, 1.395 and 1.391 Å respectively, which are comparable with the spectroscopic value of 1.397 Å appropriate to benzene. The mean C - H bond length, 0.96 Å, [0.95 Å in the cyclopentadienyl ring] is contracted from the spectroscopic value of 1.07 Å. This is usual in X-ray analysis (20) and can be attributed to the displacement of the hydrogen electron density towards the carbon atom during bond formation (21).

The variations in the geometry of chromium - nitrosyl groupings are demonstrated in Table 7.

TABLE 7

Geometry of various chromium - nitrosyl groupings

Complex	Bond lengths (Å)		Angle (°)
	Cr - NO	N - O	Cr - N - O
A: $(\pi\text{-}C_5H_5)Cr(NO)_2(NCO)$ (2)	1.716(3)	1.157(3)	171.0
B: $(\pi\text{-}C_5H_5)Cr(NO)_2C\ell$ (1)	1.711(9)†	1.140(13)†	168.6†
C: cis-$[(\pi\text{-}C_5H_5)Cr(NO)OMe]_2$ (6)	1.689(8)	1.199(10)	166.3
D: $(\pi\text{-}C_5H_5)Cr(NO)(NPh_2)I$	1.676(3)	1.183(4)	172.7
E: trans-$[(\pi\text{-}C_5H_5)Cr(NO)SPh]_2$ (5)	1.662(7)	1.19(1)	169.9
F: trans-$[(\pi\text{-}C_5H_5)Cr(NO)NMe_2]_2$ (7)	1.63(2)	1.23(2)	169.1
G: cis-$[(\pi\text{-}C_5H_5)Cr(NO)NMe_2]_2$ (7)	1.63(1)†	1.22(2)†	169.1†

† These values are an average of two independent values, with the estimated standard deviation of the mean $[\sigma_m]$ related to the individual standard deviations $[\sigma_1, \sigma_2]$ by $\sigma_m = \frac{1}{2}(\sigma_1^2 + \sigma_2^2)^{\frac{1}{2}}$.

Examination of Table 7 reveals that the values found in this analysis are physically reasonable. The trend in the Cr - NO distances suggests that there is a stronger Cr - NO bond with greater back-donation of charge to the π^* orbitals of an individual nitrosyl ligand as the number of these ligands on the metal atom is reduced, and is analogous to differences between the Cr - CO distances in chromium hexacarbonyl, mean 1.909 Å(22), and in various arenechromium tricarbonyls, ca. 1.82 Å(23). After allowing for the difference of ca. 0.06 Å in the radii of nitrogen and carbon atoms, the above values demonstrate that the σ-π synergic interaction of nitrosyl ligands is comparable with, if not greater than, that of carbonyl ligands with chromium. Increased back-donation should weaken the N - O bond and although N - O bond lengths are presumably relatively insensitive to bond order changes, analogous to the situation in carbonyl complexes(24), the N - O bond lengths appear to lengthen as the Cr - NO bond lengths decrease.

The metal - nitrosyl groupings in complexes A to G depart from linearity by highly significant amounts. The good agreement suggests that the non-linearity is not due purely to lattice forces, which are known to affect molecular conformations(25), but has electronic origins. An appropriate mechanism has been described by Kettle(26) who pointed out that small deviations from linearity in metal - carbonyl or metal - nitrosyl groupings are not necessarily attributable to packing effects in crystals, but may arise from a difference in occupation of the two distinct sets of π^* orbitals on the CO or NO ligands. As the Cr - NO bond lengths clearly indicate appreciable dπ back-donation into the π^* orbitals of the NO ligands, it is highly probable that Kettle's π-bonding mechanism is the primary cause of the Cr - N - O valency angles of ca. 170° in these complexes.

Although there are no reports of any suitable Cr - I bonds [The bridging Cr - I bond length in $[(CO)_5Cr - I - Cr(CO)_5]^{-}$(4) is 2.79 Å.] with which to compare the bond length of 2.669(1) Å found in this analysis, it does however seem reasonable considering the Cr - Cℓ bond length of 2.311 Å in $(\pi\text{-}C_5H_5)Cr(NO)_2C\ell$(1) and the respective covalent radii of iodine and chlorine atoms, 1.33 and 0.99 Å(27).

Comparison of the bond lengths in similar complexes of molybdenum and chromium reveals that the metal - chlorine bonds in $(\pi\text{-}C_5H_5)Mo(CO)_3C\ell$(28) and $(\pi\text{-}C_5H_5)Cr(NO)_2C\ell$(1) differ by 0.23 Å; the metal - iodine bonds in $(\pi\text{-}C_5H_5)Mo(CO)_2(PPh_3)I$(29) and $(\pi\text{-}C_5H_5)Cr(NO)(NPh_2)I$ differ by 0.19 Å; the metal - nitrogen bonds in $(NH\{[CH_2]_2\cdot NH_2\}_2)M(CO)_3$ where M is Mo(24) and Cr(19) differ by 0.14 Å; the metal - isocyanate bonds in $(\pi\text{-}C_5H_5)Mo(CO)(PPh_3)_2(NCO)$(30) and $(\pi\text{-}C_5H_5)Cr(NO)_2(NCO)$(2) differ by 0.15 Å and the mean metal - cyclopentadienyl distances in these molecules differ by 0.14 Å. It has been suggested(29) that the greater ionic character of the metal - halogen bonds may account for the disagreement between the values obtained by considering the metal - halogen bonds and by considering the metal - carbon and metal - nitrogen bonds.

The cyclopentadienyl ring is accurately planar, the root-mean-square deviation from planarity being 0.009 Å. The variations in the Cr - C and C - C bond lengths can be compared with those found in other chromium-cyclopentadienyl complexes (see Table 8) and, as in these examples, are probably significant.

TABLE 8

Cr - C and C - C bond lengths (Å) in chromium-cyclopentadienyl complexes

Complex†	Cr - C bond lengths		C - C bond lengths	
	Range	Mean	Range	Mean
A	2.173 - 2.227	2.199	1.369 - 1.417	1.394
B††		2.20		1.41
C	2.206 - 2.248	2.232	1.36 - 1.43	1.39
D	2.210 - 2.292	2.250	1.375 - 1.432	1.402
E	2.167 - 2.253	2.207	1.35 - 1.43	1.38
F	2.23 - 2.28	2.26	1.33 - 1.49	1.42
G†††	2.21 - 2.32	2.27	1.30 - 1.47	1.40

† A: $(\pi\text{-}C_5H_5)Cr(NO)_2(NCO)$(2)

B: $(\pi\text{-}C_5H_5)Cr(NO)_2C\ell$(1)

C: cis-$[(\pi\text{-}C_5H_5)Cr(NO)OMe]_2$(6)

D: $(\pi\text{-}C_5H_5)Cr(NO)(NPh_2)I$

E: trans-$[(\pi\text{-}C_5H_5)Cr(NO)SPh]_2$(5)

F: trans-$[(\pi\text{-}C_5H_5)Cr(NO)NMe_2]_2$(7)

G: cis-$[(\pi\text{-}C_5H_5)Cr(NO)NMe_2]_2$(7)

†† The cyclopentadienyl ring is disordered.

††† Values are given for the ordered cyclopentadienyl ring.

Significant variations in metal - carbon and carbon - carbon bond lengths have also been found in other types of π-cyclopentadienyl complexes, e.g. $(\pi\text{-}C_5H_5)Mo(CO)(PPh_3)_2(NCO)$(30) and $(\pi\text{-}C_5H_5)Rh(\pi\text{-}2,3\text{-dichlorobutadiene})$(31), and can be rationalized(32) as arising from the lack of cylindrical symmetry about the metal atom removing the degeneracy of the e_1 molecular orbitals of the cyclopentadienyl ring. The variations in this analysis typify those found in the other chromium-cyclopentadienyl complexes; the longest Cr - C bond, Cr - C(17), is situated approximately trans to the nitrosyl ligand, the longest C - C bond, C(14) - C(15), is associated with the shortest Cr - C bonds and the deviations, although not necessarily significant, of the C - C - C angles from the mean of 108.0° reflect the π-character of the C - C bonds with the smallest angles, 106.7(5) and 107.4(5)° at C(15) and C(14) respectively, being associated with the longest C - C bond.

The mean cyclopentadienyl carbon - carbon bond lengths in Table 8 are consistently shorter than the C - C bond lengths of 1.431(5) and 1.427(7) Å found in the electron-diffraction studies of $(\pi\text{-}C_5H_5)_2Fe$(33) and $(\pi\text{-}C_5H_5)In$(34) respectively. Such contractions in other π-cyclopentadienyl complexes are well documented(35) and have been attributed(35) to librational motion of the cyclopentadienyl ring.

Regarding the cyclopentadienyl ligand as tridentate, the chromium atom has a distorted octahedral coordination with angles between monodentate ligands [I - Cr - NPh_2 = 100.9, I - Cr - NO = 90.3 and ON - Cr - NPh_2 = 97.2°] fairly similar to those found in $(\pi\text{-}C_5H_5)Cr(NO)_2Cl$(1) [mean Cl - Cr - NO = 99.1 and ON - Cr - NO = 94.3°] and in $(\pi\text{-}C_5H_5)Cr(NO)_2(NCO)$(2) [ON - Cr - NO = 94.9 and ON - Cr - NCO = 99.7°]. The significant variations in this analysis seem to arise from the greater steric requirements of the diphenylamido and iodo ligands than by electron-electron repulsions between bonding electrons, which would have resulted in the greater double-bond character of the Cr - NO bond being reflected in the larger angles associated with the nitrosyl ligand.

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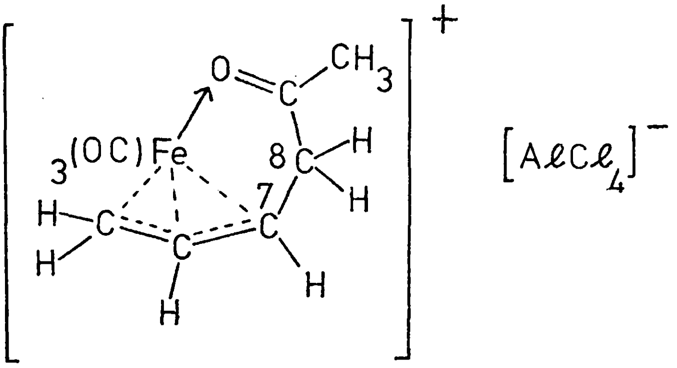
CHAPTER 2

TRICARBONYL(2—4-η-HEPTEN-5-METHYL-6-ONE)IRON HEXAFLUOROPHOSPHATE

III.2.1 INTRODUCTION

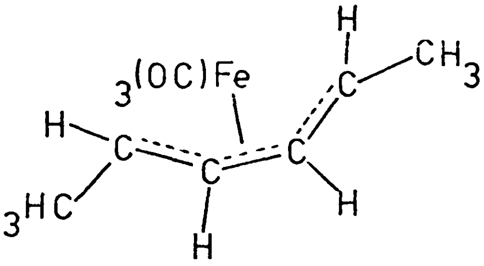
Although numerous electrophilic substitution reactions of transition metal-complexed dienes and polyenes have been reported$^{(e.g.\ 1 - 13)}$, the role of the metal atom in these reactions is uncertain.

Butadiene $Fe(CO)_3$ undergoes Friedel-Crafts acylation on reaction with a $CH_3COC\ell$-$A\ell C\ell_3$ Perrier complex under homogeneous conditions ($CH_2C\ell_2$ solution) 3,800 times faster than benzene$^{(10)}$. A stable intermediate, I, has been isolated$^{(2)}$ and the cation characterized$^{(14)}$



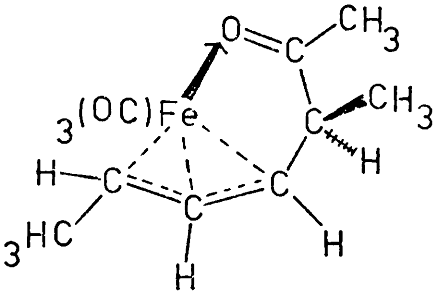
I

by an X-ray analysis of the PF_6^- salt, but this does not reveal whether acylation occurs by attack on the side of the ligand near to or away from the iron atom (endo or exo), the latter being followed by rotation of the C(7) - C(8) bond through 180°. In order to determine which mechanism operates, Dr. G. R. Knox repeated the acylation on hexa-2,4-diene $Fe(CO)_3$, II. However, n.m.r. investigations, even when



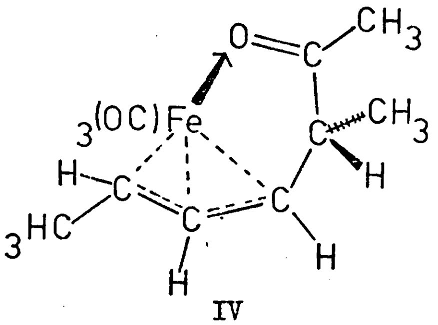
II

isotopically labelled substrates were used, did not demonstrate whether the cation of the isolable intermediate was III or IV, therefore an X-ray analysis of the PF_6^- salt was undertaken.



III

endo-attack



IV

exo-attack

III.2.2 EXPERIMENTAL

Crystal Data Tricarbonyl(2—4-η-hepten-5-methyl-6-one)iron hexafluorophosphate; $[C_{11}H_{13}O_4Fe]^+[PF_6]^-$.

Crystal system	Monoclinic
Unit cell dimensions	$a = 9.363(6)$ Å
	$b = 12.174(9)$ Å
	$c = 16.547(10)$ Å
	$\beta = 121^\circ 36'(3)$
Space group	$P2_1/c$ (C_{2h}^5)

U = 1606.8 $Å^3$

M = 409.9 a.m.u.

D_o = 1.68 $gm.cm.^{-3}$ (flotation in MeI/CCl_4 solution)

D_c = 1.694 $gm.cm.^{-3}$

Z = 4

F(000) = 824

μ(Mo-$K\alpha$) = 11.46 $cm.^{-1}$

Crystallographic Measurements

Preliminary cell dimensions, and systematically absent reflections ($h0\ell$ absent if $\ell = 2n + 1$, $0k0$ absent if $k = 2n + 1$) consistent with space group $P2_1/c$ were found from precession and Wiessenburg photographs (using Mo-$\underline{K}\alpha$ and Cu-$\underline{K}\alpha$ radiation respectively) of a yellow crystal, mounted about b*, with dimensions ca. 0.02 x 0.12 x 1.20 mm^3.

The crystal, having been transfered to a Hilger and Watts' Y290 automatic diffractometer controlled by a PDP-8 computer, was offset on χ by about 4° both to prevent multiple reflections(15) and to enable a data set to be collected without any observations being made at χ values of about 90°. A fully stabilized X-ray generator, operated at 46 kV and 16 mA, and a graphite crystal monochromator were used to produce Mo-$\underline{K}\alpha$ radiation. The cell dimensions were then adjusted by a least-squares treatment(16) of the θ, χ and ϕ setting angles of twelve reflections from octants $hk\bar{\ell}$ and $\bar{h}k\bar{\ell}$.

The intensity data were collected by the $\theta - 2\theta$ scan procedure, each reflection being scanned in 36 equal steps from $2\theta_{calc.} -0.72°$ to $2\theta_{calc.} +0.72°$. Each step was counted for 3 seconds and stationary-crystal, stationary-counter background counts were measured at each end of the scan range for 27 seconds. The intensities of two standard reflections were monitored every 40 intensity measurements and only small random fluctuations were observed. The scan counts (P) and the background counts (B_1, B_2) of each reflection were combined to yield the integrated intensity, $I = P - 2(B_1 + B_2)$, which was corrected for Lorentz-polarisation effects but not for absorption.

Initially, observations were collected in octant $\bar{h}k\bar{\ell}$ with θ(Mo-$\underline{K}\alpha$) $\leqslant 27°$. Next, the collection of octant $hk\bar{\ell}$ was started, but examination of equivalent reflections ($\bar{h}k0$ and $hk0$) revealed intensity differences of up to 25%. The data were therefore recollected with each octant being defined by a separate orientation matrix calculated using the setting angles of 9 reflections in that octant. This resulted in a set of $\bar{h}k\bar{\ell}$ observations which were essentially the same as before, and an improved set of $hk\bar{\ell}$ observations, in which the intensity differences had been reduced by about a third. The two sets of $\bar{h}k\bar{\ell}$ observations were averaged to give 1,287 observations and then combined with the 2,401 $hk\bar{\ell}$ observations using the common $0k\bar{\ell}$ reflections for scaling purposes. All these 3,688 observations were used in the initial structure elucidation, but only the 1,538 independent values of $|F_o|$ which had $I/\sigma_I > 3.0$ were used in the least-squares refinement.

Structure Analysis

The initial coordinates of the iron and phosphorus atoms were obtained from the three-dimensional Patterson synthesis and used to phase an electron density distribution. From this and subsequent electron density maps (see Table 1), the positions of the remaining non-hydrogen atoms were found from an examination of the bond lengths and angles calculated on the highest peaks in each map.

TABLE 1

Steps in the structure elucidation of $[C_{11}H_{13}O_4Fe]^+[PF_6]^-$

Step	Atoms used to phase the electron density distribution	R-factor	Atoms unambiguously found
1)	Fe,P.	47%	F(1),F(2),F(3),F(4), C(1),O(1),C(3),O(3).
2)	Fe,P plus atoms found in step 1.	44%	Rest of the non-hydrogen atoms except F(5), F(6),C(4),C(11).
3)	All the non-hydrogen atoms except F(5),F(6),C(4),C(11).	37%	F(5),F(6),C(4),C(11).

The atomic numbering scheme is that employed in Figure 4, page 156.

The approximate atomic parameters were then adjusted by a series of least-squares calculations (see Table 2).

TABLE 2

Progress of the least-squares refinement of $[C_{11}H_{13}O_4Fe]^+[PF_6]^-$

Step	Treatment of parameters	Final R-factor
1)	4 cycles; full-matrix; refinement of overall scale factor; x, y, z, U_{iso} for all the atoms.	14.0%
2)	2 cycles; full-matrix; refinement of overall scale factor; x, y, z, U_{iso} for C, O, F; x, y, z, U_{ij} for Fe, P.	12.6%
3)	3 cycles; full-matrix; refinement of overall scale factor; x, y, z, U_{ij} for Fe, P, F.	10.6%
4)	5 cycles; block diagonal; refinement of overall scale factor; x, y, z, U_{ij} for all the atoms.	9.2%

At this stage a difference electron density distribution was calculated revealing:

1) Peaks and holes (1.50 to $-0.90e^-/Å^3$) around the iron atom.
2) Four well-resolved peaks plus two elongated regions of electron density (average peak height $0.75e^-/Å^3$) at bonding distances from the phosphorus atom.
3) Peaks (average height $0.46e^-/Å^3$) assignable to the four non-methyl hydrogen atoms.
4) Poorly-resolved peaks (ca. $0.45e^-/Å^3$) in the region of the methyl hydrogen atoms.

The positions of the hydrogen atoms attached to C(11) were calculated assuming a staggered conformation about the C(8) - C(11) bond and a C - H bond length of 0.98 Å. These methyl hydrogen atoms were assigned an isotropic temperature factor equal to that of C(11) and included with fixed parameters during five cycles of block diagonal least-squares refinement of the overall scale factor; x, y, z, U_{iso} for the non-methyl hydrogen atoms and x, y, z, U_{ij} for the non-hydrogen atoms. Convergence was reached at R = 8.72%, R' = 11.70%.

The peaks in the difference synthesis at bonding distances from the phosphorus atom, the diffuseness of the fluorine peaks in the original F_o maps and their high thermal parameters (average B_{iso} 15.4Å^2) were indicative of disorder in the hexafluorophosphate anion, therefore the residual electron density around the phosphorus atom was contoured on to glass sheets and more closely examined. This suggested a model for the anion consisting of three distinct octahedra (see Figure 1).

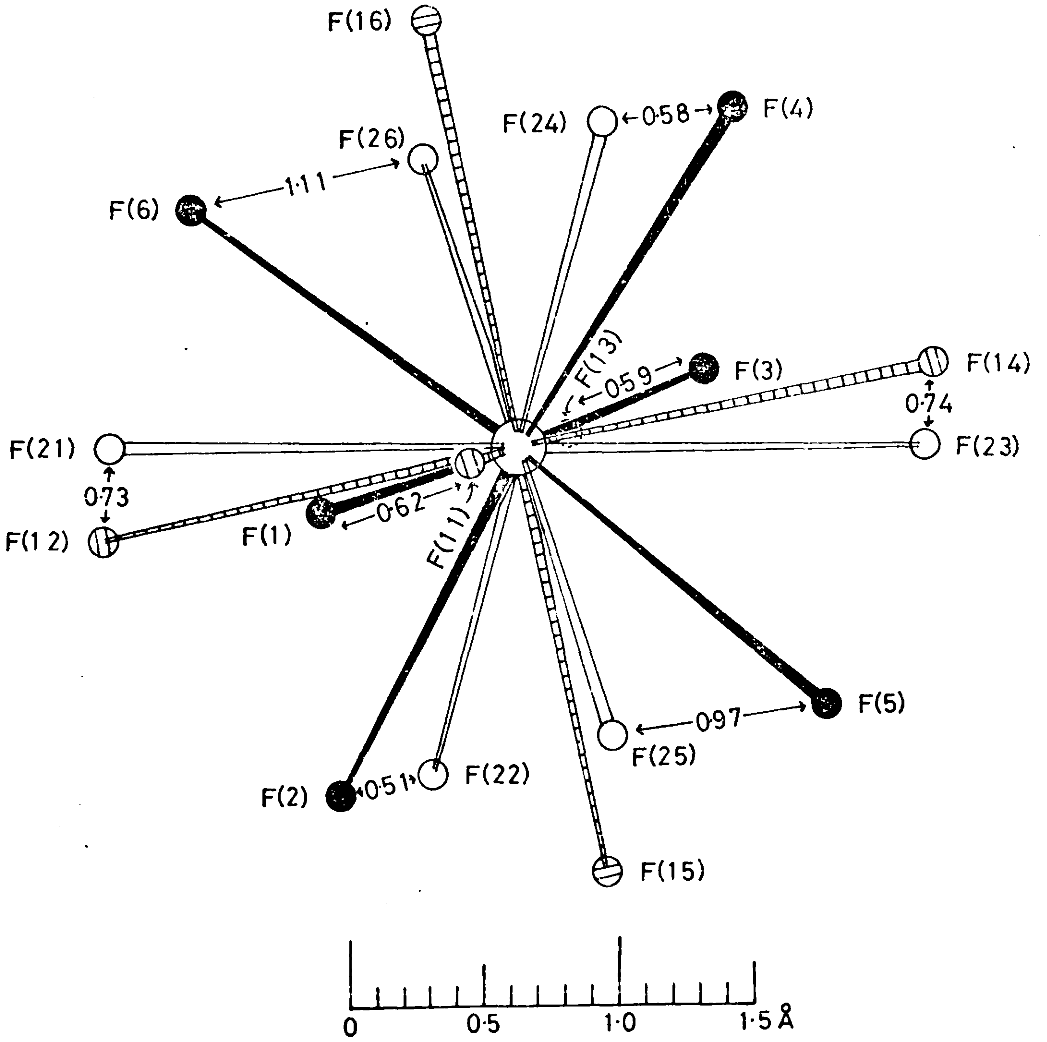


FIGURE 1

The three orientations of the hexafluorophosphate anion viewed along the a-axis, illustrating the interatomic distances <1.14 Å.

Atoms F(15), F(25), F(16) and F(26) correspond to the four well-defined residual peaks and the overlap of F(12) with F(21) and that of F(14) with F(23) correspond to the two elongated regions of electron density. Using these eight positions as the starting basis and assuming the same phosphorus position in each orientation of the anion, coordinates for the two minor orientations were calculated; each corresponding to an ideal octahedron with P - F bond lengths equal to that found in $NaPF_6$ and KPF_6(17), i.e. 1.58 Å. The proximity of the calculated positions of F(11), F(22), F(13) and F(24) to the refined positions of F(1), F(2), F(3) and F(4) respectively explains both the absense of the former set of peaks in the difference synthesis and the higher peak heights, compared with F(5) and F(6), of the latter set of peaks in the original F_o maps.

From a comparison of the peak heights in the final difference synthesis and in the difference synthesis calculated omitting all the fluorine atoms, the population parameters of the three orientations were estimated to be 0.68, 0.16 and 0.16. The fluorine atoms of the minor orientations were each assigned $B_{iso} = 9.0Å^2$. In the subsequent least-squares refinement the cation was treated as previously, while in the anion the refined parameters were x, y, z, U_{ij} for the fluorine atoms of the major orientation and also for the single phosphorus atom which was used to account for the three partial phosphorus atoms. Five cycles of least-squares refinement, with 1 block per ion, reached convergence at R = 8.01%, R' = 10.91%.

The above treatment of the disorder resulted in a considerable improvement in the parameters of the anion with the P - F distances, 1.503 - 1.573, mean 1.532(29[†]) Å, being lengthened to 1.539 - 1.596, mean 1.569(18[†]) Å, the average deviation of the F - P - F angles from

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[†] Root-mean-square deviation from the mean.

ideal octahedral geometry being reduced from 3.1 to 2.3° and the average B_{iso} of the refined fluorine atoms being lowered from 15.4Å^2 to the more realistic value of 11.2Å^2. A difference electron density distribution still contained residual electron density in the region of the anion [The average peak height of the previously noted peaks had been reduced from 0.75 to 0.49e$^-$/Å^3.] but, because the dimensions of the anion were of little interest and the dimensions of the cation not significantly dependent on the model employed for the anion, no further calculations were undertaken.

Notes:

1) The scattering factors employed for Fe^+, P, F, O, C and H were taken from 'International Tables for X-ray Crystallography,' Vol. III$^{(18)}$, as were the dispersion corrections $\Delta f'$ and $\Delta f''$ for the iron and phosphorus atoms.

2) The final weights employed in the least-squares refinement, in order to ensure approximate constancy of $\langle w\Delta^2\rangle$ over various ranges of $|F_o|$ and $\sin\theta$, were given by the expression:

$$w^{-1} = 5 + 0.07|F_o| + 0.01|F_o|^2 - 6.66\sin\theta$$

3) The calculations were carried out on Glasgow University's KDF9 computer [The least-squares program due to D. W. J. Cruickshank, J. G. F. Smith and J. G. Sime was used], and latterly on the IBM 370/155 computer of the Edinburgh Regional Computer Centre using the 'X-ray System' programs$^{(19)}$.

Results

The observed and final calculated structure factors are listed in Table 3, with the final positional and thermal parameters, including the estimated standard deviations derived from the least-squares refinement, in Tables 4 and 5 respectively. The thermal parameters of the cation and anion are also illustrated by 50% and 20% probability thermal ellipsoids(20) respectively in Figures 2 and 3 respectively. The bond lengths, valency angles, torsion angles and mean plane calculations are given in Tables 6, 7, 8 and 9 respectively. The crystal packing is shown in Figure 4 and the interionic contacts $\leqslant$ 3.50 Å are listed in Table 10.

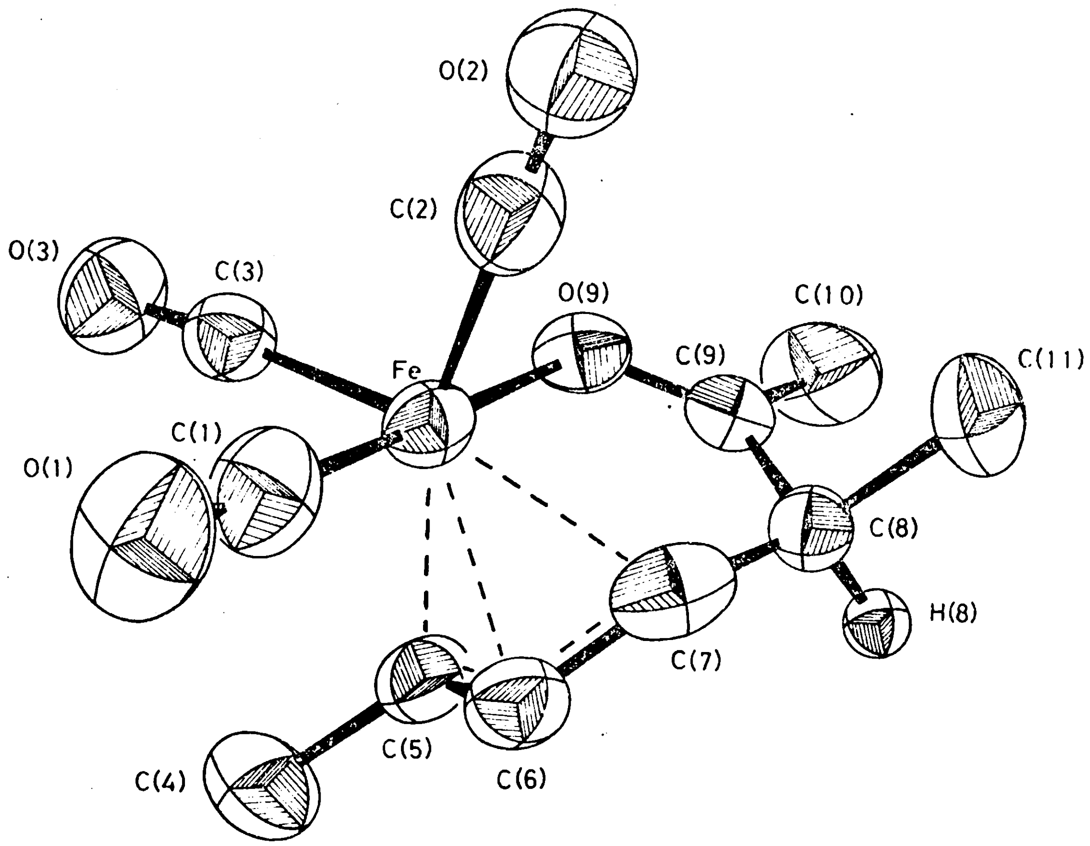

FIGURE 2

Perspective view of the tricarbonyl(2——4-η-hepten-5-methyl-6-one)iron cation illustrating the 50% probability thermal ellipsoids.

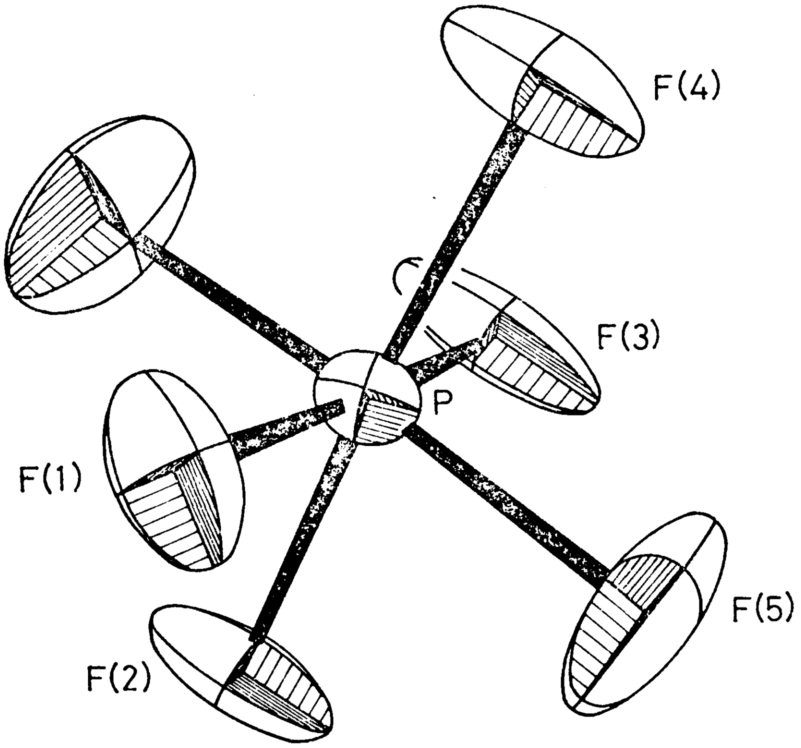

FIGURE 3

Perspective view of the major orientation of the hexafluorophosphate anion viewed along the a-axis, illustrating the 20% probability thermal ellipsoids.

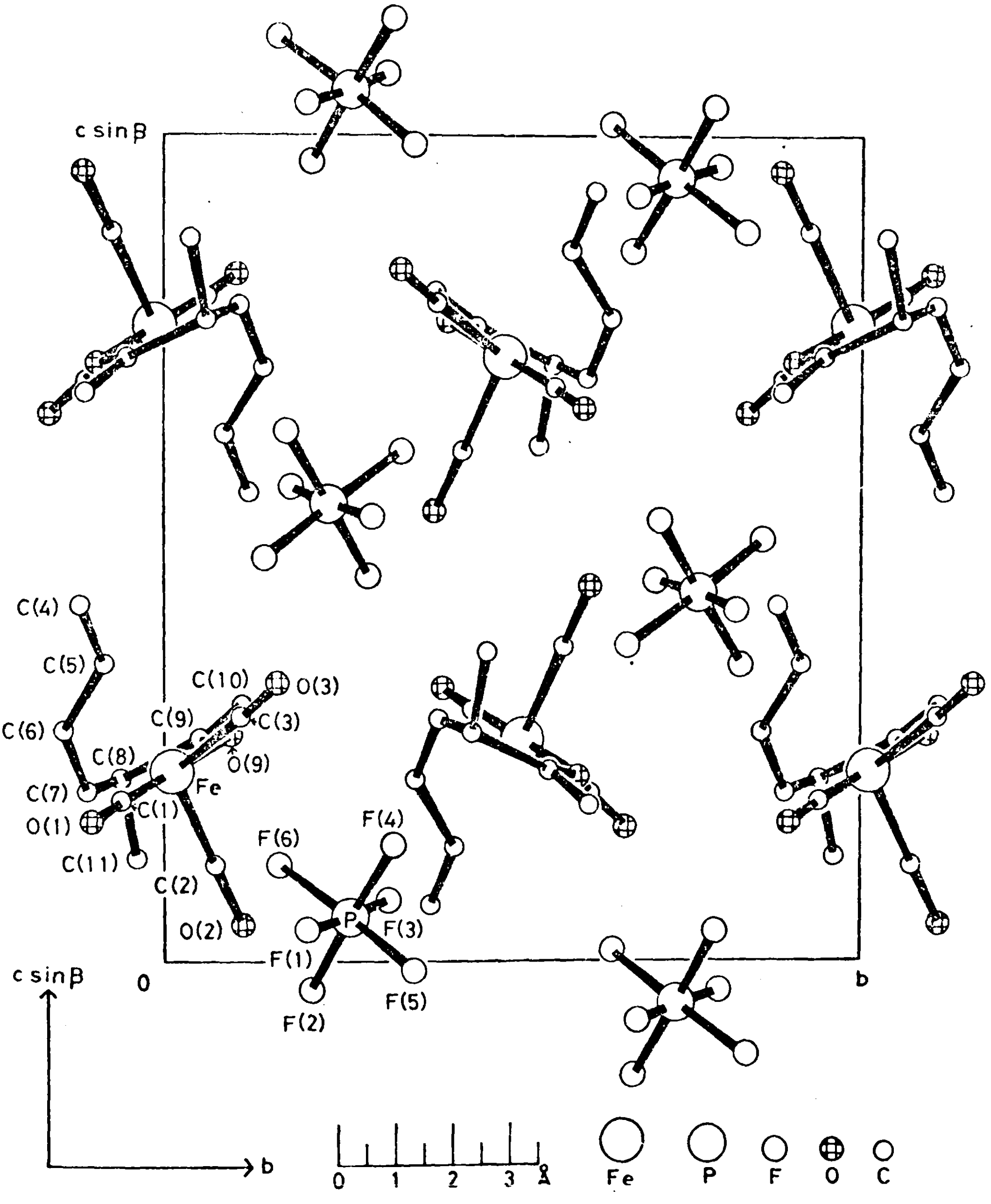

FIGURE 4

Packing arrangement of tricarbonyl(2——4-η-hepten-5-methyl-6-one)iron hexafluorophosphate viewed along the a-axis. For clarity, only the major orientation of the anion is shown.

TABLE 3

Tricarbonyl(2——4-η-hepten-5-methyl-6-one)iron hexafluorophosphate; observed and final calculated structure amplitudes (x10).

10,3,L		
-7	98	109
-9	124	135
-10	115	125
-11	165	125
-13	137	121
-15	121	96
10,2,L		
-4	100	92
10,1,L		
-7	125	132
-11	160	163
-13	107	166
-15	168	162
-17	141	122
10,0,L		
0	106	105
-10	101	92
9,8,L		
-7	134	139
9,7,L		
-13	137	132
-16	124	108
9,6,L		
0	100	83
-2	88	78
-4	156	161
-5	98	80
-6	129	136
-8	112	120
-10	153	140
9,5,L		
-6	111	88
-7	156	141
-11	126	136
-13	129	123
-15	125	128
-17	108	86
9,4,L		
-2	174	195
-4	150	143
-6	166	182
-8	103	131
-12	107	112
9,3,L		
-4	132	118
-9	271	192
-10	181	158
-11	132	126
-13	153	154
-15	102	102
9,2,L		
0	139	149
-4	166	176
9,1,L		
-5	135	140
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-19	121	108
9,0,L		
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8,10,L		
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-6	124	121
8,9,L		
-13	116	96
8,8,L		
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-5	137	130
-6	101	105
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-11	114	84
-13	140	110
8,7,L		
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-13	161	140
-15	178	159
8,6,L		
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-10	178	166
-11	136	122
-12	187	132
8,5,L		
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-11	141	139
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-15	116	117
-16	101	77
-17	151	118
8,4,L		
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-12	159	159
-14	120	110
8,3,L		
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-9	132	129
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-13	107	75
-15	117	126
-16	106	77
-17	117	107
8,2,L		
0	199	237
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-3	106	147
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8,1,L		
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-5	129	124
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-11	129	134
-12	105	102
-17	152	132
-19	103	141
8,0,L		
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7,12,L		
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7,10,L		
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-7	151	157
-8	129	129
-11	115	105
7,9,L		
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7,8,L		
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-13	128	132
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-3	126	112
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-12	131	125
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-13	125	110
-14	126	120
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7,5,L		
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-15	110	136
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-14	284	293
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7,3,L		
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-12	165	226
-15	112	120
-17	101	116
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7,2,L		
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-10	142	160
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7,1,L		
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-13	170	154
-14	129	181
-15	127	140
-17	148	144
-19	158	151
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6,12,L		
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6,11,L		
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-6	115	108
6,10,L		
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-4	148	126
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-11	150	124
-12	112	132
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6,8,L		
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-11	191	173
-12	169	145
-13	135	117
-16	170	126
6,7,L		
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-15	111	78
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6,6,L		
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-10	133	124
-12	123	110
-13	190	165
-14	193	185
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6,5,L		
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6,4,L		
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-13	112	106
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-15	161	160
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-12	130	143
-13	157	138
-14	162	166
-15	161	177
-17	174	164
6,0,L		
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5,13,L		
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5,12,L		
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5,11,L		
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-5	175	162
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5,10,L		
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-10	193	185
-11	132	131
-13	130	88
5,9,L		
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-6	174	174
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-3	140	160
-6	315	319
-7	117	126
-8	172	169
-10	125	123
-11	147	164
-12	209	195
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5,7,L		
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-7	103	177
-8	162	184
-10	122	100
-13	132	133
-16	126	107
5,6,L		
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-14	235	226
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-16	130	139
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5,5,L		
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-6	213	234
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5,4,L		
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-12	434	415
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-14	116	119
-15	129	127
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-18	149	139
5,3,L		
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-17	119	110
-19	115	81
5,2,L		
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-5	288	174
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-7	261	271
-8	292	304
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-10	554	565
-11	125	133
-12	152	147
-13	111	87
-14	408	404
-15	132	162
-16	112	122
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5,1,L		
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-5	359	425
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-7	187	287
-8	273	281
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-16	118	141
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5,0,L		
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4,13,L		
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4,12,L		
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-5	137	15
-8	116	147
-9	139	149
-13	163	168
4,11,L		
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-11	122	118
4,10,L		
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-5	120	107
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-10	184	193
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4,9,L		
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-5	197	187
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-9	144	150
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4,8,L		
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-6	159	153
-7	111	93
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-13	120	110
-14	141	144
4,7,L		
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-4	578	561
-5	225	227
-7	313	296
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-12	143	103
-13	107	85
4,6,L		
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-10	369	386
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-15	130	112
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4,5,L		
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-3	772	771
-4	127	134
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-6	378	378
-7	585	486
-9	412	382
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-12	145	125
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4,4,L		
0	224	220
-1	94	104
-2	87	67
-3	91	74
-4	114	144
-5	179	189
-6	249	244
-7	356	355
-8	485	400
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-13	166	173
-14	348	345
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4,3,L		
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-2	189	182
-3	241	287
-4	264	262
-5	910	903
-6	108	68
-7	762	804
-8	253	250
-9	395	420
-10	273	264
-11	229	289
-12	133	117
-14	184	169
4,2,L		
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-3	92	21
-5	536	568
-6	397	436
-7	258	243
-9	142	126
-10	498	498
-11	328	358
-12	240	239
-13	109	104
-14	194	216
-15	93	58
-16	314	323
-17	107	88
-20	116	135
4,1,L		
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-3	655	725
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4,0,L		
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-18	216	204
3,15,L		
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3,13,L		
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3,12,L		
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3,11,L		
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-3	120	124
-4	170	162
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-7	168	181
-9	122	97
-13	110	116
3,10,L		
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-7	114	97
-8	153	157
-11	140	169
-12	112	134
3,9,L		
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-2	423	377
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-5	291	257
-6	153	172
-8	369	368
-9	170	180
-10	141	125
-11	189	122
-15	139	120
3,8,L		
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-6	223	223
-8	103	84
-9	166	159
-10	262	272
-14	107	158
3,7,L		
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-2	236	198
-3	488	449
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-5	198	285
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-7	365	376
-9	261	237
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-13	170	152
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3,6,L		
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-5	418	404
-6	154	175
-8	142	114
-9	146	160
-11	171	175
-12	300	312
-16	172	174
3,5,L		
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-4	470	442
-5	870	868
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-7	604	645
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-10	105	117
-11	270	250
-12	99	97
-14	112	111
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3,4,L		
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-4	195	175
-6	135	101
-7	214	220
-8	182	167
-9	137	126
-10	149	152
-11	115	107
-13	212	211
-14	334	332
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3,3,L		
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-3	444	411
-5	854	817
-7	591	580
-8	142	129
-9	514	522
-10	700	704
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3,2,L		
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-2	190	245
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-4	228	227
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-6	308	364
-7	321	307
-8	243	224
-9	181	205
-10	233	254
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-12	440	411
-14	104	122
-16	235	240
-18	143	141
3,1,L		
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-5	600	693
-6	221	186
-7	479	511
-8	182	209
-9	560	585
-11	483	523
-12	170	169
-13	261	279
-15	95	94
-16	104	100
3,0,L		
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-6	346	341
-8	457	483
-10	348	370
-14	454	436
-16	115	117
-18	147	150
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2,13,L		
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2,12,L		
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-3	114	144
2,11,L		
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-3	187	149
-4	118	123
-5	132	142
-6	139	175
-7	122	115
-9	188	218
2,10,L		
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-2	198	199
-4	227	194
-5	114	140
-6	160	201
-7	118	96
2,9,L		
0	276	243
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-3	201	227
-4	366	387
-5	160	157
-6	179	219
-7	389	406
-8	277	270
-9	126	132
-10	282	290
-11	280	271
-13	111	100
-14	140	132
2,8,L		
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-3	317	314
-4	384	347
-6	168	171
-8	247	265
-15	132	126
-16	120	137
2,7,L		
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-2	247	190
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-5	704	702
-6	560	597
-7	259	278
-8	289	293
-9	388	397
-10	211	201
-12	380	310
-13	227	241
2,6,L		
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-4	267	276
-5	287	288
-6	186	205
-8	176	139
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-11	117	130
-12	120	110
-13	108	159
-14	129	131
-17	112	96
-18	114	105
2,5,L		
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-3	719	681
-4	252	277
-5	711	698
-6	86	36
-7	689	647
-8	327	327
-9	249	287
-10	344	353
-11	370	384
-13	138	131
-14	190	186
-15	101	109
2,4,L		
0	930	834
-1	704	806
-2	231	215
-3	408	400

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  -8   134   105
 -10   375   352
 -11   193   191
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 -15   100   108
 -16   126   132

     2,3,L

   0   528   504
  -1   851   746
  -2   752   659
  -3   513   553
  -4   752   679
  -5   661   688
  -6   347   348
  -7   233   236
  -8   111   117
  -9   562   570
 -11   427   439
 -12   310   295
 -13   283   261
 -14   120   113
 -16   150   103

     2,2,L

   0   918   849
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  -2   198   183
  -3   680   639
  -5   339   384
  -7   297   265
  -9   307   321
 -12   207   208
 -13   172   205
 -14   193   178
 -16   181   190

     2,1,L

   0   310   381
  -1   703   671
  -2   516   526
  -3   640   576
  -4  1485  1425
  -6   209   219
  -7   651   735
  -9   494   551
 -11   312   323
 -12   175   147
 -13   181   203
 -14   170   170
 -15   116   112
 -17   166   161

     2,0,L

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  -6   458   484
  -8   150   193
 -10    86    93
 -12   159   170
 -14   225   246
 -16   240   267
 -18   113   104

     1,14,L

  -1   176   199

     1,12,L

   0   142   151
  -1   105   149
  -2   100    83
  -3   198   219
  -5   132   144

     1,11,L

  -1   143   148
  -4   144   149
  -5   184   200
  -6   112   109
  -8   167   201
  -9   166   175
 -11   121   130

     1,10,L

   0   265   257
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  -2   263   274
  -6   163   171
  -7   107   116
  -8   103   113

     1,9,L

   0   326   202
  -3   275   286
  -4   175   194
  -6   302   317
  -7   362   400
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  -9   173   166
 -10   268   267
 -11   159   167
 -12   113   117
 -13   120   111

     1,8,L

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  -4   446   445
  -5   163   192
  -6   135   118
  -8   109   112

     1,7,L

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  -3   265   256
  -4   187   110
  -5   463   499
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  -8   420   454
  -9   238   252
 -10   100   104
 -11   235   230
 -12   186   273
 -13   168   180
 -14   187   185
 -15   118   124

     1,6,L

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  -5   236   214
  -6   485   547
  -7   299   333
  -9    97   109
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 -12   133   118
 -13   115   124
 -15   112   105

     1,5,L

  -1   186   166
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 -13   333   319
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  -8   234   217
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 -11   227   225
 -13   101   113
 -14   113   112

     1,3,L

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  -7   782   776
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 -11   346   358
 -12   108   128
 -13   186   202
 -15   219   214
 -17   110   104

     1,2,L

   0  1192   967
  -1   356   366
  -2  1901  1739
  -3   265   280
  -5   239   236
  -6   190   223
  -8   149   130
  -9   219   230
 -11   102   113
 -12   141   135
 -13   114   107
 -14   227   251

     1,1,L

   0  1580  1452
  -1   196   255
  -2   833   764
  -3   174   287
  -4   148   137
  -5   120   115
  -6   197   183
  -7   217   243
  -9   237   265
 -10   172   180
 -11   387   325
 -12    92   105
 -13   226   234
 -15   167   167
 -17   139   149

     1,0,L

   0  1687  1889
  -2   463   573
  -4  1416  1550
  -6   615   644
  -8   175   211
 -10   161   171
 -12   381   352
 -16   197   235
 -18   121   136

     0,14,L

  -1   159   163
  -3   149   190
  -7   126   104

     0,12,L

   0   124   144
  -1   305   263
  -3   180   190
  -5   223   264

     0,11,L

  -7   111   116

     0,10,L

   0   227   238
  -1   171   174
  -2   272   284
  -3   302   316
  -4   101   207
  -7   126   152

     0,9,L

  -2   113   112
  -3   127   113
  -5   124   139
  -6   374   352
  -8   199   231
  -9   111   117
 -12   126   150
 -13   109   123

     0,8,L

   0   632   552
  -1   280   289
  -2   158   182
  -4   420   412
  -5   183   180
  -6   307   298
  -9   102   113
 -10   132   135
 -11   116   114

     0,7,L

  -4   508   560
  -6   104    87
  -7   305   316
  -8   340   335
 -10   123   128
 -11   241   243
 -13   150   161

     0,6,L

   0    93    83
  -1   175   164
  -2   350   407
  -3   346   387
  -4   296   277
  -5   140   145
  -6   201   192
  -7   261   265
  -8   401   400
 -12   199   208

     0,5,L

  -3   198   168
  -5   424   422
  -7   320   308
  -8   149   140
  -9   482   500
 -11   259   271
 -13   243   237
 -15   117   122

     0,4,L

   0   422   231
  -1   125   117
  -2   120   100
  -3   349   353
  -4   454   473
  -5   598   567
  -6   593   624
  -7   281   251
  -8   217   222
 -10   364   371

     0,3,L

  -1   662   564
  -2   671   605
  -3   846   783
  -4   671   769
  -5   483   456
  -6   436   460
  -7   445   456
  -8   127   100
  -9   334   337
 -10   103   111
 -11   323   325
 -13   246   265
 -15   123   126
 -17   148   117

     0,2,L

   0   973   458
  -2  2375  2215
  -3   370   261
  -4   646   530
  -5   273   285
  -6   521   531
  -8   614   679
  -9    95    83
 -10   175   170

     0,1,L

  -2   799   744
  -3   318   320
  -4  1394  1360
  -5   203   207
  -7   264   281
  -8   125   140
  -9   324   367
 -11   233   258
 -12   196   224
 -14   115   132
 -16   159   164

     0,0,L

  -2   989   971
  -4   923   988
  -6   475   561
 -10   452   459
 -12   256   285
 -18   123   125

    -1,14,L

  -1   113    94
  -3   158   179
  -5   114   133

    -1,12,L

  -1   322   306
  -2   112   115
  -3   169   160
  -5   145   180
  -6   134   143
  -7   123   122

    -1,11,L

  -1   105    97
  -2   133   122

    -1,10,L

  -1   137   135
  -2   214   222
  -3   263   262
  -4   268   281
  -5   105   131
  -6   137   136

    -1,9,L

  -3   134   176
  -4   129   134
  -5   135    88
  -6   132   184
  -8    99   117
 -11   102   102
 -12   120   140

    -1,8,L

  -1   330   264
  -2   487   430
  -4   245   264
  -5   124   101
  -6   283   263
  -7   201   220
 -10   163   144

    -1,7,L

  -1   221   224
  -4   289   287
  -5   161   150
  -6   273   257
  -7   118   123
  -9   112   115
 -10   108    92
 -11   134   131
 -13   150   156

    -1,6,L

  -1   479   396
  -2   249   256
  -3   471   392
  -4   597   598
  -5   144   144
  -6   163   169
  -7   133   130
  -8   326   311
  -9   199   214
 -10   289   208
 -12   183   181

    -1,5,L

  -1   430   334
  -2   380   278
  -4   122   124
  -5   157   146
  -6   422   401
  -8   311   307
  -9   198   215
 -11   277   264
 -13   150   158
 -15   138   147

    -1,4,L

  -1   835   645
  -2   885   772
  -3   297   292
  -4   140   150
  -5   543   585
  -6   742   751
  -7   270   256
  -8   152   172
  -9   139   126
 -10   197   207
 -11   139   120
 -12   254   287
 -13   108   100

    -1,3,L

  -3   151   135
  -4   348   387
  -5   113   107
  -7   461   487
  -8   411   395
  -9   274   274
 -10   391   399
 -11   172   191
 -13   217   223

    -1,2,L

  -1   241   257
  -2   865   819
  -4  1183  1179
  -6   364   358
  -7   434   420
  -8   476   481
  -9   325   316
 -10   193   196
 -14   137   168

    -1,1,L

  -2   862   835
  -3   169   158
  -5   587   591
  -6   103   131
  -7   320   322
  -8   236   248
  -9   201   229
 -10   186   182
 -11   104   100
 -15   137   129

    -1,0,L

  -2  1451  1443
  -4  1190  1116
  -6   921   973
  -8   269   276
 -12   104   116

    -2,14,L

  -1   148   170
  -5   121   107

    -2,12,L

  -1   153   143
  -2   155   132
  -3   226   226
  -4   123   143
  -5   137   143
  -6   145   151
  -7   168   178
  -9   120   118

    -2,11,L

  -1   132   143
  -2   129   135

    -2,10,L

  -4   164   170
  -5   233   224
  -6   179   178
  -7   111   114
  -8   136   158
  -9   118   132
 -10   146   128

    -2,9,L

  -2   216   181
  -3   192   170
  -4   113    89

    -2,8,L

  -1   163   126
  -2   661   586
  -3   252   238
  -4   196   220
  -6   123   135
  -7   133   136
  -8   199   195
 -12   118   101

    -2,7,L

  -1   403   314
  -2   146   111
  -4   358   320
  -5   149   136
  -6   172   167
 -12   124   139

    -2,6,L

  -1   240   241
  -2   667   579
  -3   120   119
  -4   520   478
  -5   293   296
  -6   302   290
 -12   195   191

    -2,5,L

  -1   352   335
  -2   131   133
  -3   418   342
  -4   206   189
  -6   111    78
  -8   264   237
 -11   190   185
 -13   118   111

    -2,4,L

  -1   552   456
  -2   713   668
  -4   459   429
  -6   166   178
  -7   286   276
  -8   398   374
  -9   122   109
 -10   146   158
 -12   136   155

    -2,3,L

  -1   909   780
  -2   478   365
  -3   309   237
  -5   318   341
  -6   472   445
  -7   178   198
  -8   118   127
  -9   179   193
 -13   127   130

    -2,2,L

  -1   317   239
  -2   274   276
  -3   636   680
  -4   428   438
  -5   296   277
  -6   673   674
  -8   217   237
  -9   115   127
 -10   430   421
 -11   109   118
 -12   111   100
 -14   101   100

    -2,1,L

  -1   129   140
  -2   223   181
  -3   366   355
  -4   538   549
  -5   215   186
  -6   318   295
  -8   279   266
  -9    90   111
 -10    99   100
 -11   126   117
 -13   101   101

    -2,0,L

  -2   695   678
  -4   583   533
  -6   554   544
  -8   844   870
 -12   242   241
 -14   147   130

    -3,14,L

  -1   171   157

    -3,13,L

  -2   135   120

    -3,12,L

  -1   138   125
  -3   249   247
  -4   123   112
  -5   148   166
  -7   120   135

    -3,10,L

  -1   197   184
  -2   168   139
  -3   147   137
  -5   172   168
  -6   197   190
  -7   156   159

    -3,9,L

  -2   261   237

    -3,8,L

  -2   241   193
  -3   133   171
  -4   279   256
  -8   181   176
  -9   109   120
 -10   121   128

    -3,7,L

  -1   545   452
  -3   330   311
  -4   191   171
  -7   124   128

    -3,6,L

  -1   410   325
  -2   677   575
  -3   223   184
  -5   106   114
  -6   230   205
 -10   124   125
 -12   121   111

    -3,5,L

  -1   446   369
  -2   199   146
  -3   527   477
  -5   283   244
  -7    99    94
  -8   135   107

    -3,4,L

  -1   160   143
  -2   164   151
  -3   524   456
  -4   762   695
  -8   283   274
 -10   143   155

    -3,3,L

  -1   619   575
  -2   139   130
  -3   137   136
  -4   105   110
  -5   162   163
  -8   269   269

    -3,2,L

  -1   324   295
  -2   164   178
  -3   142   128
  -4   228   223
  -5   182   111
  -6   827   792
  -8   233   239
 -10   188   195
 -12   200   205

    -3,1,L

  -1   535   467
  -3   671   602
  -4   241   235
  -5   364   367
  -6   172   153

    -3,0,L

  -2    84   110
  -4   557   563
  -6   292   325
  -8   476   485
 -10   280   220
 -12   184   195
 -14   165   169

    -4,12,L

  -1   130   124

    -4,11,L

  -2   110    99

    -4,10,L

  -1   160   152
  -2   146   137
  -3   184   174
  -7   114   130

    -4,9,L

  -1   241   236
  -4   148   130
  -5   133   182
  -7   126    96

    -4,8,L

  -4   195   187
  -5   149   153
  -6   126   119
  -9   126   127
 -10   133   136

    -4,7,L

  -1   348   288
  -2   299   282
  -3   384   294
  -5   183   166
  -6   185   159
  -7   116   107
  -9   185   182

    -4,6,L

  -2   239   198
  -6   144   138
  -7    96    84
  -8   214   215

    -4,5,L

  -1   742   621
  -2   218   203
  -3   302   285
  -4   451   483
  -6    98    97
  -7   111   111

    -4,4,L

  -3   132   130
  -4   259   226
  -5   198   166
  -6   111   115
  -8   130   141
 -10   287   216

    -4,3,L

  -1   368   368
  -2   287   244
  -3   634   553
  -5   123   127
  -6   137   138

    -4,2,L

  -1   286   192
  -2   540   497
  -4   193   172
  -6   282   215
  -7   195   191
  -8   289   287
 -10   109   110
 -12   188   171

    -4,1,L

  -1   533   519
  -3   117   116
  -4   144   162
  -5   359   356
  -6   200   195
  -7   199   218
  -9   110   113

    -4,0,L

  -2   175   144
  -4   530   514
  -6   332   328
  -8   130   126
 -10   245   210

    -5,11,L

  -1   107    92
  -2   126   110

    -5,9,L

  -1   257   237
  -3   219   261
  -4   126   117
  -7   104    82

    -5,8,L

  -1   165   172
  -3   184   156
  -5   111   126
  -8   184   184

    -5,7,L

  -1   354   311
  -2   330   293
  -3   215   287
  -4   132    92
  -5   274   241
  -6   120    94
  -8   126   120
  -9   110   103

    -5,6,L

  -3   236   224
  -4   121   117
  -5   155   134
  -8   143   151
  -9   101   113

    -5,5,L

  -1   347   293
  -2   142   186
  -3   293   268
  -4   217   213
  -5   153   151
  -7   213   204

    -5,4,L

  -6   200   103
  -8   117    93

    -5,3,L

  -1   197   201
  -2   314   231
  -3   220   214
  -6   216   150
  -7   148   147

    -5,2,L

  -4   100   102
  -5   144   158
  -8   120   139

    -5,1,L

  -1   247   232
  -2   295   277
  -3   365   338
  -5   317   307
  -7   114   111
  -9    99    98
 -11    99    51

    -5,0,L

  -2   270   188
  -6   188   189
 -10   182    79

    -6,10,L

  -2   104    89

    -6,9,L

  -1   189    89
  -3   114   118

    -6,7,L

  -1   273   239
  -2   154   150
  -3   160   156
  -4   135   142
  -5   144   137

    -6,6,L

  -1   130   113
  -2   103    81

    -6,5,L

  -1   161   182
  -3   164   148
  -5   210   195
  -6   118   115
  -7   149   145

    -6,4,L

  -1   149   119
  -2   124   102

    -6,3,L

  -1   270   256
  -3   150   151
  -5   102   120
  -6   163    93
  -7   129   144
  -9   114   105

    -6,2,L

  -1   105    93
  -2   235   211
  -3   178   147
  -4   114   115

    -6,1,L

  -1   360   338
  -3   370   334
  -5   142   127
  -7    95    64

    -7,7,L

  -3   100    84
  -5   134    83

    -7,6,L

  -2   142   115
  -4   100    98

    -7,5,L

  -1   170   158
  -2   145   119
  -3   107    86
  -5   139   143

    -7,4,L

  -1   177   156

    -7,3,L

  -1   115   114
  -3   194   108
  -5   135   124

    -7,2,L

  -1   161   125

    -7,1,L

  -1   233   201
  -2   101    88
  -3   148   121
  -5    96   100
  -7    96    78

    -7,0,L

  -2   105   178

    -8,4,L

  -2   103   104

    -8,3,L

  -1   103    92

    -8,2,L

  -4   118    86

    -8,1,L

  -1   117   118
  -3   109   125

    -8,0,L

  -2   164   147

    -9,1,L

  -1   107    93
```

TABLE 4

Tricarbonyl(2---4-η-hepten-5-methyl-6-one)iron hexafluorophosphate; fractional atomic coordinates with esd in parentheses.

Part I: The cation

Atom	x	y	z
Fe	0.05319(18)	0.01305(11)	0.23181(10)
O(1)	0.2515(15)	-0.1012(9)	0.1673(8)
O(2)	-0.1593(13)	0.1131(10)	0.0434(7)
O(3)	0.3445(11)	0.1615(7)	0.3354(7)
O(9)	-0.0767(9)	0.0952(5)	0.2762(5)
C(1)	0.1730(17)	-0.0576(11)	0.1927(9)
C(2)	-0.0745(16)	0.0751(12)	0.1155(9)
C(3)	0.2327(13)	0.1087(9)	0.2962(7)
C(4)	0.3101(18)	-0.1203(14)	0.4314(12)
C(5)	0.1332(14)	-0.0841(9)	0.3620(8)
C(6)	0.0303(16)	-0.1415(9)	0.2786(9)
C(7)	-0.1306(17)	-0.1081(9)	0.2060(9)
C(8)	-0.2569(13)	-0.0576(9)	0.2217(7)
C(9)	-0.2020(13)	0.0512(8)	0.2699(7)
C(10)	-0.2910(19)	0.1088(11)	0.3104(11)
C(11)	-0.4252(15)	-0.0384(13)	0.1248(9)
H(5)†	0.043(21)	-0.044(14)	0.391(11)
H(6)	0.091(16)	-0.182(11)	0.267(9)
H(7)	-0.165(12)	-0.111(8)	0.159(7)
H(8)	-0.294(13)	-0.098(9)	0.263(7)
H(11a)	-0.405	0.009	0.085
H(11b)	-0.461	-0.107	0.094
H(11c)	-0.507	-0.007	0.135

† Each hydrogen atom is numbered according to the atom to which it is bonded.

Part II: The anion

Atom	x	y	z	PP.[††]
P	0.23566(41)	0.26390(27)	0.05379(19)	1.00
F(1)	0.3679(16)	0.2032(10)	0.0377(11)	0.68
F(2)	0.0854(17)	0.2092(15)	-0.0351(10)	0.68
F(3)	0.1100(18)	0.3199(19)	0.0739(14)	0.68
F(4)	0.3863(15)	0.3275(14)	0.1414(10)	0.68
F(5)	0.2338(25)	0.3571(16)	-0.0121(14)	0.68
F(6)	0.2455(29)	0.1637(19)	0.1171(16)	0.68
F(11)	0.399	0.250	0.050	0.16
F(12)	0.194	0.138	0.030	0.16
F(13)	0.073	0.279	0.058	0.16
F(14)	0.278	0.389	0.076	0.16
F(15)	0.122	0.292	-0.056	0.16
F(16)	0.339	0.237	0.164	0.16
F(21)	0.285	0.141	0.054	0.16
F(22)	0.052	0.239	-0.030	0.16
F(23)	0.186	0.387	0.055	0.16
F(24)	0.420	0.289	0.138	0.16
F(25)	0.287	0.293	-0.020	0.16
F(26)	0.184	0.235	0.128	0.16

[††] PP. is the population parameter.

TABLE 5

Tricarbonyl(2——4-η-hepten-5-methyl-6-one)iron hexafluorophosphate; anisotropic temperature factor* (U_{ij}x$10^4$$\text{Å}^2$) or isotropic temperature factor (U_{iso}x$10^3$$\text{Å}^2$) for each atom.

* The form of the anisotropic temperature factor is:

$$T = \exp\left[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}\ell^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}h\ell a^*c^* + 2U_{23}k\ell b^*c^*)\right].$$

Part I: The cation

Atom	U_{11} or U_{iso}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Fe	504	359	427	-78	222	-45
O(1)	1088	948	1191	-64	862	-298
O(2)	818	1199	600	-140	228	268
O(3)	644	625	864	-218	321	-167
O(9)	627	369	552	22	329	11
C(1)	622	751	594	-142	286	-92
C(2)	599	860	530	-132	266	5
C(3)	504	431	515	23	247	3
C(4)	595	828	976	118	132	263
C(5)	579	529	534	-63	264	97
C(6)	661	385	700	-18	381	-24
C(7)	828	418	401	-115	304	-94
C(8)	456	546	443	-32	198	111
C(9)	478	435	501	84	222	109
C(10)	1006	610	1134	212	764	-15
C(11)	437	923	681	-91	101	98
H(5)	80					
H(6)	39					
H(7)	1					
H(8)	22					
H(11a)	71(fixed)					
H(11b)	71(fixed)					
H(11c)	71(fixed)					

Part II: The anion

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
P	696	601	433	-38	260	-74
F(1)	922	673	1365	-29	715	-309
F(2)	850	1925	1017	-441	359	-1040
F(3)	648	2682	1769	-277	534	-1715
F(4)	712	1634	1060	-240	338	-888
F(5)	1666	1384	1518	79	595	992
F(6)	1998	1751	1898	-41	1198	1067

The fluorine atoms of the two minor orientations of the anion were each assigned a 'fixed' $U_{iso}(x10^3Å^2) = 114$.

Average estimated standard deviations. $(U_{ij}x10^4Å^2)$ and $(U_{iso}x10^3Å^2)$

Part I: The cation

Atom	U_{11} or U_{iso}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Fe	7	6	6	7	5	6
O	61	63	59	50	52	49
C	72	72	71	56	60	57
H	35					

Part II: The anion

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
P	18	17	14	14	14	12
F	113	157	131	105	104	123

TABLE 6

Tricarbonyl(2——4-η-hepten-5-methyl-6-one)iron hexafluorophosphate; bond lengths (Å) with esd in parentheses.

Part I: The cation

Fe	-	C(1)	1.782(18)	C(7)	-	C(8)	1.470(22)
Fe	-	C(2)	1.809(12)	C(8)	-	C(9)	1.490(15)
Fe	-	C(3)	1.852(10)	C(8)	-	C(11)	1.563(13)
Fe	-	C(5)	2.210(12)	C(9)	-	C(10)	1.488(26)
Fe	-	C(6)	2.087(13)	C(9)	-	O(9)	1.244(16)
Fe	-	C(7)	2.132(14)	C(5)	-	H(5)	1.26(23)
Fe	-	O(9)	1.987(10)	C(6)	-	H(6)	0.84(17)
C(1)	-	O(1)	1.149(24)	C(7)	-	H(7)	0.67(11)
C(2)	-	O(2)	1.127(15)	C(8)	-	H(8)	1.04(14)
C(3)	-	O(3)	1.102(13)	C(11)	-	H(11a)	0.98(fixed)
C(4)	-	C(5)	1.501(17)	C(11)	-	H(11b)	0.98(fixed)
C(5)	-	C(6)	1.384(15)	C(11)	-	H(11c)	0.98(fixed)
C(6)	-	C(7)	1.404(16)				

Part II: The anion

P	-	F(1)	1.580(18)	P	-	F(4)	1.596(13)
P	-	F(2)	1.555(13)	P	-	F(5)	1.568(23)
P	-	F(3)	1.539(23)	P	-	F(6)	1.578(26)

The fluorine atoms of the two minor orientations of the anion were 'fixed' at positions corresponding to P - F distances of 1.58 Å.

TABLE 7

Tricarbonyl(2——4-η-hepten-5-methyl-6-one)iron hexafluorophosphate; valency angles ($^{\circ}$) with esd in parentheses.

Part I: The cation

C(1) - Fe - C(2)	90.4(7)	Fe - O(9) - C(9)	119.0(7)
C(1) - Fe - C(3)	87.6(6)	Fe - C(5) - C(6)	66.5(7)
C(2) - Fe - C(3)	104.0(6)	Fe - C(6) - C(5)	76.1(7)
C(1) - Fe - C(5)	97.9(6)	Fe - C(7) - C(6)	68.8(8)
C(1) - Fe - C(6)	84.6(7)	Fe - C(6) - C(7)	72.3(7)
C(1) - Fe - C(7)	100.9(6)	C(4) - C(5) - C(6)	121.5(13)
C(2) - Fe - C(5)	162.2(6)	C(5) - C(6) - C(7)	125.1(12)
C(2) - Fe - C(6)	129.0(5)	C(6) - C(7) - C(8)	124.8(13)
C(2) - Fe - C(7)	93.6(6)	C(7) - C(8) - C(9)	112.5(10)
C(3) - Fe - C(5)	92.1(4)	C(7) - C(8) - C(11)	110.7(11)
C(3) - Fe - C(6)	126.3(4)	C(9) - C(8) - C(11)	107.0(9)
C(3) - Fe - C(7)	160.5(6)	C(8) - C(9) - C(10)	122.7(12)
C(5) - Fe - C(6)	37.5(4)	C(8) - C(9) - O(9)	118.2(12)
C(5) - Fe - C(7)	69.5(4)	C(10) - C(9) - O(9)	119.0(10)
C(6) - Fe - C(7)	38.9(4)	C(4) - C(5) - H(5)	121(6)
O(9) - Fe - C(1)	178.5(6)	C(6) - C(5) - H(5)	108(6)
O(9) - Fe - C(2)	89.4(6)	C(5) - C(6) - H(6)	108(7)
O(9) - Fe - C(3)	91.0(5)	C(7) - C(6) - H(6)	122(8)
O(9) - Fe - C(5)	82.6(4)	C(6) - C(7) - H(7)	128(10)
O(9) - Fe - C(6)	96.6(5)	C(8) - C(7) - H(7)	107(10)
O(9) - Fe - C(7)	80.6(5)	C(7) - C(8) - H(8)	120(6)
Fe - C(1) - O(1)	178.6(14)	C(9) - C(8) - H(8)	102(6)
Fe - C(2) - O(2)	177.3(15)	C(11) - C(8) - H(8)	104(5)
Fe - C(3) - O(3)	176.7(11)		

The hydrogen atoms attached to C(11) were 'fixed' at positions giving a staggered conformation about the C(8) - C(11) bond.

Part II: The anion

F(1) - P - F(2)	92.4(9)	F(2) - P - F(6)	90.3(10)
F(1) - P - F(3)	177.3(10)	F(3) - P - F(4)	90.0(9)
F(1) - P - F(4)	89.0(8)	F(3) - P - F(5)	95.7(13)
F(1) - P - F(5)	86.8(11)	F(3) - P - F(6)	89.1(14)
F(1) - P - F(6)	88.4(12)	F(4) - P - F(5)	87.9(9)
F(2) - P - F(3)	88.8(10)	F(4) - P - F(6)	93.2(9)
F(2) - P - F(4)	176.2(9)	F(5) - P - F(6)	175.1(15)
F(2) - P - F(5)	88.7(9)		

The fluorine atoms of the two minor orientations of the anion were 'fixed' at ideally octahedral positions about the phosphorus atom.

TABLE 8

Tricarbonyl(2——4-η-hepten-5-methyl-6-one)iron hexafluorophosphate; torsion angles ($^{\circ}$).

Fe - O(9) - C(9) - C(8)	-5	H(6) - C(6) - C(7) - C(8)	166
Fe - O(9) - C(9) - C(10)	175	H(6) - C(6) - C(7) - H(7)	-13
Fe - C(5) - C(6) - C(7)	-56	C(6) - C(7) - C(8) - C(9)	64
Fe - C(5) - C(6) - H(6)	100	C(6) - C(7) - C(8) - C(11)	-176
Fe - C(6) - C(7) - C(8)	-98	C(6) - C(7) - C(8) - H(8)	-55
Fe - C(6) - C(7) - H(7)	96	H(7) - C(7) - C(8) - C(9)	-115
Fe - C(7) - C(6) - C(5)	57	H(7) - C(7) - C(8) - C(11)	4
Fe - C(7) - C(6) - H(6)	-95	H(7) - C(7) - C(8) - C(8)	125
C(4) - C(5) - C(6) - C(7)	-174	C(7) - C(8) - C(9) - O(9)	12
C(4) - C(5) - C(6) - H(6)	-18	C(7) - C(8) - C(9) - C(10)	-168
H(5) - C(5) - C(6) - C(7)	39	C(11)- C(8) - C(9) - O(9)	-109
H(5) - C(5) - C(6) - H(6)	-164	C(11)- C(8) - C(9) - C(10)	70
C(5) - C(6) - C(7) - C(8)	-40	H(8) - C(8) - C(9) - O(9)	141
C(5) - C(6) - C(7) - H(7)	139	H(8) - C(8) - C(9) - C(10)	-38

The sign of the torsion angle is negative if an anticlockwise rotation of A is required to eclipse D whilest looking down the B - C bond, the atom labelling being from left to right in the above table.

TABLE 9

Tricarbonyl(2——4-7-hepten-5-methyl-6-one)iron hexafluorophosphate; mean plane calculations.

Part I: The cation

	Atoms in plane	Displacements (Å)	Atoms out of plane	Displacements (Å)
a)	C(5)		H(5)	-0.76
	C(6)		H(6)	0.33
	C(7)		H(7)	0.34
			C(4)	0.136
			C(8)	-0.783
			H(8)	-1.82
			C(11)	-0.609
			C(9)	-0.363
			O(9)	-0.609
			Fe	1.683
b)	C(5)	0.069	H(5)	-0.21
	C(6)	-0.154	H(6)	-0.18
	C(7)	0.149	H(7)	0.41
	C(8)	-0.064	H(8)	-1.01
			C(4)	-0.161
			C(11)	0.298
			C(9)	0.778
			O(9)	1.697
			Fe	1.887
c)	O(1)		C(1)	-0.623
	O(2)		C(2)	-0.569
	O(3)		C(3)	-0.578
			Fe	-1.552
d)	C(2)	0.007	Fe	0.119
	C(3)	-0.007	C(1)	1.885
	C(5)	0.008	O(9)	-1.856
	C(7)	-0.008	C(6)	0.591
			C(8)	-1.423

	Atoms in plane	Displacements (Å)	Atoms out of plane	Displacements (Å)
e)	C(2)		Fe	-0.089
	C(3)		C(1)	1.690
	C(6)		O(9)	-2.073
			C(5)	-0.526
			C(7)	-0.543
f)	C(1)		Fe	0.014
	C(6)		O(1)	-0.015
	O(9)		C(2)	1.426
			C(3)	-1.465
			C(5)	-1.257
			C(7)	1.224

The dihedral angles (°) between selected planes are:

a) - c)	1.2	a) - f)	89.4
b) - c)	24.7	b) - f)	89.7
a) - d)	66.8	d) - f)	88.4
b) - d)	89.1	e) - f)	88.7

<u>Part II: The anion</u>

	Atoms in plane	Displacements (Å)	Atoms out of plane	Displacements (Å)
g)	F(1)	-0.040	F(5)	1.556
	F(2)	0.042	F(6)	-1.585
	F(3)	-0.042	P	0.007
	F(4)	0.041		
h)	F(1)	-0.016	F(2)	1.553
	F(3)	-0.015	F(4)	-1.595
	F(5)	0.015	P	0.000
	F(6)	0.015		
i)	F(2)	-0.044	F(1)	1.556
	F(4)	-0.042	F(3)	-1.560
	F(5)	0.044	P	-0.023
	F(6)	0.041		

The dihedral angles (°) between the planes are:

g) - h)	89.1	h) - i)	88.9
g) - i)	88.1		

TABLE 10

Tricarbonyl(2——4-η-hepten-5-methyl-6-one)iron hexafluorophosphate; interionic contacts (3.50Å).

F(25)	- - -	$O(3^{I})$	2.77
F(15)	- - -	$O(9^{I})$	2.77
F(13)	- - -	O(2)	2.88
F(16)	- - -	O(3)	2.96
F(26)	- - -	C(3)	2.99
F(11)	- - -	$C(4^{II})$	3.02
F(26)	- - -	C(2)	3.03
F(2)	- - -	$O(1^{III})$	3.04
F(25)	- - -	$C(3^{I})$	3.05
F(22)	- - -	$O(1^{III})$	3.05
F(26)	- - -	O(3)	3.06
F(2)	- - -	$C(1^{III})$	3.07
F(6)	- - -	C(3)	3.10
O(1)	- - -	$O(2^{III})$	3.12
F(26)	- - -	O(2)	3.13
F(6)	- - -	C(2)	3.17
F(5)	- - -	$C(3^{I})$	3.19
F(21)	- - -	$C(11^{IV})$	3.19
F(6)	- - -	C(1)	3.19
F(5)	- - -	$O(3^{I})$	3.20
F(22)	- - -	O(2)	3.20
F(3)	- - -	$C(5^{V})$	3.20
F(13)	- - -	C(2)	3.21
F(5)	- - -	$O(9^{I})$	3.22
F(6)	- - -	O(3)	3.22
F(14)	- - -	$C(10^{V})$	3.23
F(12)	- - -	$O(2^{III})$	3.24
F(16)	- - -	C(3)	3.24
F(22)	- - -	$C(1^{III})$	3.26
F(3)	- - -	$C(8^{V})$	3.27
F(5)	- - -	$C(5^{I})$	3.29
O(3)	- - -	$C(7^{V})$	3.30
F(23)	- - -	$C(10^{V})$	3.30
F(2)	- - -	$C(7^{III})$	3.31
F(12)	- - -	$C(2^{III})$	3.31
F(13)	- - -	$C(5^{V})$	3.31
F(4)	- - -	$O(1^{II})$	3.32
F(6)	- - -	O(1)	3.32
F(15)	- - -	$C(3^{I})$	3.35
F(24)	- - -	$O(1^{II})$	3.35
F(26)	- - -	$C(8^{V})$	3.35
F(3)	- - -	$C(6^{V})$	3.36
F(6)	- - -	O(2)	3.37
F(2)	- - -	O(2)	3.38
F(15)	- - -	$C(7^{III})$	3.38
F(4)	- - -	$C(8^{V})$	3.38
F(16)	- - -	$C(10^{IV})$	3.39
F(1)	- - -	$C(11^{IV})$	3.40
F(21)	- - -	$O(2^{III})$	3.40
F(3)	- - -	O(2)	3.41
O(9)	- - -	$C(6^{V})$	3.42
F(25)	- - -	$C(4^{II})$	3.42
F(22)	- - -	$O(9^{I})$	3.43
F(12)	- - -	O(2)	3.44
F(15)	- - -	$O(3^{I})$	3.44
F(22)	- - -	$C(10^{I})$	3.44
F(13)	- - -	$C(6^{V})$	3.45
F(26)	- - -	$C(6^{V})$	3.45
F(24)	- - -	$C(4^{II})$	3.46
F(25)	- - -	$C(7^{III})$	3.46
F(23)	- - -	$C(9^{V})$	3.46
F(23)	- - -	$C(8^{V})$	3.46
F(16)	- - -	$C(8^{V})$	3.46
F(15)	- - -	$C(9^{I})$	3.47
F(11)	- - -	$O(3^{I})$	3.48
F(24)	- - -	$C(10^{IV})$	3.49

The superscripts refer to the following transformations of the atomic coordinates:

I	x,	$\frac{1}{2} - y$,	$-\frac{1}{2} + z$;	IV	$1 + x$,	y,	z;
II	$1 - x$,	$\frac{1}{2} + y$,	$\frac{1}{2} - z$;	V	$-x$,	$\frac{1}{2} + y$,	$\frac{1}{2} - z$.
III	$-x$,	$-y$,	$-z$;				

III.2.1 DISCUSSION

The view of the cation in Figure 2 and the displacements from the π-allyl plane of Fe, C(8), H(8) and C(11) [1.68, -0.78, -1.82 and -0.61 Å respectively] both indicate that endo-acylation has occurred. Although endo-acylation is also thought likely in the Friedel-Crafts reaction involving cycloheptatriene $Fe(CO)_3$(5) and endo-protonation of butadiene $Fe(CO)_3$ has been demonstrated(3), electrophiles do not always attack the π-system of coordinated olefins endo to the metal, e.g. protonation of cycloheptatriene $Fe(CO)_3$(5) and tropone $Fe(CO)_3$(9).

Figure 2 also illustrates that the organic ligand is bound to the iron atom by the π-allyl residue C(5) - C(6) - C(7) [The Fe - C bond lengths are 2.210(12), 2.087(13) and 2.132(14) Å respectively.] and by the acyl oxygen O(9) [1.987(10) Å], but not by any of the other atoms. [The Fe - C(4), C(8) and C(9) distances are 3.31, 2.95 and 2.81 Å respectively.] Considering the π-allyl residue as the 4-electron donor, $\pi\text{-}C_3H_3^-$, and with the donation of a lone-pair of electrons from each of the carbonyl ligands and from the acyl oxygen, tricarbonyl(2——4-η-hepten-5-methyl-6-one)iron hexafluorophosphate can be considered as a d^6 Fe^{II} complex with 18 metal valence electrons.

The dimensions of the iron—π-allyl linkage are in good agreement with those found in other π-allyl $Fe(CO)_3$ complexes (see Table 11).

TABLE 11

Geometry in iron—π-allyl linkages

Complex†	Fe - C bonds (Å)	C - C bonds (Å)	C - C - C angle (°)
This work	2.210(12) 2.087(13) 2.132(14)	1.384(15) 1.404(16)	121.5(13)
A	2.141(10) 2.063(9) 2.144(8)	1.380(15) 1.415(15)	123.7(8)
B	2.194(18) 2.091(16) 2.202(17)	1.440(24) 1.420(23)	124.3(16)
C	2.17(1) 2.08(2) 2.11(1)	1.37(2) 1.44(3)	120.8(15)
D	2.200(8) 2.050(8) 2.142(8)	1.432(12) 1.391(12)	122.8

† A: Tricarbonyl(1——3-7-hexen-5-one)iron hexafluorophosphate; $[C_9H_9FeO_4]^+[PF_6]^-$(14)

B: Butadieneiron tricarbonyl-sulphur dioxide-boron trifluoride adduct; $C_4H_6Fe(CO)_3 \cdot SO_2 \cdot BF_4$(21)

C: Tricarbonyl-cycloheptatrieneiron-tetracyanoethylene adduct; $(C_{13}H_8N_4)Fe(CO)_3$(22)

D: Azulenepentacarbonyldi-iron; $C_{10}H_8Fe_2(CO)_5$(23)

In these π-allyl complexes the Fe - C(terminal) distances, mean 2.165 Å, are longer than the Fe - C(central) distance, mean 2.074 Å, reminiscent of the situation in 1,3-diene $Fe(CO)_3$ complexes, in which typical Fe - C(terminal) and Fe - C(central) distances are 2.16 and 2.05 Å respectively(24). This asymmetry strengthens the σ-π bonding in the 1,3-diene $Fe(CO)_3$ complexes and although a similar interaction probably occurs in the π-allyl complexes, a contributing factor to the variation in the Fe - C bond lengths in most of these complexes is the distortion of the ligand to allow metal - ligand bonds other than to the π-allyl group. The C - C distances in these groups, e.g. 1.384(15) and 1.404(16) Å in this analysis, compare favourably with those in other metal — π-allyl complexes, e.g. 1.419(18) and 1.428(19) Å in π-methylallyl[bis-1,2-(diphenylphosphino)-ethane]nickel bromide(25) and 1.352(11) and 1.421(12) Å in π-allyl-dihydrobis(3,5-dimethyl-1-pyrazolyl)-boratodicarbonylmolybdenum(26), and as in 1,3-diene $Fe(CO)_3$ complexes, e.g. 1.414(7), 1.407(7) and 1.409(7) Å in 1H-azepine $Fe(CO)_3$(27), are intermediate between single- and double-bond values for carbon - carbon bonds, indicating delocalisation of the π-electrons over the bonded atoms. The approximate sp^2 hybridisation of these atoms is also demonstrated by the C - C - C valency angles in this analysis at C(5), C(6) and C(7) of 121.5(13), 125.1(12) and 124.8(13)° respectively.

The other C - C bond lengths in this analysis are consistent with single bonds, the $C(sp^3) - C(sp^3)$ bond [C(8) - C(11) = 1.563(13) Å] and the $C(sp^3) - C(sp^2)$ bonds [C(4) - C(5) = 1.501(17), C(8) - C(7) = 1.470(22), C(8) - C(9) = 1.490(15) and C(10) - C(9) = 1.488(26) Å] being close to the commonly accepted values of 1.54 and 1.51 Å respectively. The refined C - H distances range from 0.67 - 1.26, mean 0.95 Å; a contraction from the spectroscopic value of 1.07 Å similar to that noted in other X-ray analyses(28) and attributed to the displacement of the hydrogen electron density towards the carbon atom during bond formation(29).

The Fe - O(9) bond length, 1.987(10) Å, is identical within experimental error to the Fe - O bond lengths of 1.999(5) and 2.004(10) Å in the corresponding butadiene $Fe(CO)_3$ acylation intermediate(14), A, and in $C_4H_6Fe(CO)_3 \cdot SO_2 \cdot BF_3$(21), B, respectively, and considering the covalent radii of O and Fe^{II} to be 0.66(30) and 1.34 Å(31) respectively indicates that the iron - acyl oxygen bond is essentially single. The C(9) - O(9) bond length, 1.244(16) Å, [1.207(8) Å in A] is in good agreement with Sutton's(32) value of 1.215(5) Å for ketones and aldehydes, demonstrating that the acyl CO bond order is only marginally affected, if at all, by the formation of the iron – acyl oxygen linkage.

In A the Fe - C(carbonyl) bond trans to the acyl oxygen, 1.787(8) Å, is significantly shorter than those trans to the π-allyl system, 1.840(9) and 1.828(8) Å, reflecting the negligable back-donation of charge from the iron to the acyl oxygen compared with that to the π-allyl system. Although the variations in these bond lengths in this analysis [1.782(18), 1.809(12) and 1.852(10) Å] and in B [1.767(20), 1.818(17) and 1.830(16) Å] are not necessarily significant, the shortest bond in both complexes is indeed trans to the oxygen. The C - O(carbonyl) bond lengths in this analysis [1.149(24), 1.127(15) and 1.102(13) Å] agree very well with each other and with Sutton's(32) value of 1.14(1) Å, while the Fe - C - O angles [178.6(14), 177.3(15) and 176.7(11)°] are reasonably close to 180°.

The lack of cylindrical symmetry trans to the $Fe(CO)_3$ group results in the C(1) - Fe - C(2) and C(1) - Fe - C(3) angles, 90.4(7) and 87.6(6)° respectively, being considerably smaller than the C(2) - Fe - C(3) angle, 104.0(6)°; a distortion identical to that found in A, in which the corresponding angles are 90.4(4), 88.7(4) and 103.8(4)° respectively, and of the same magnitude as that found in other metal$(CO)_3$ complexes(33).

The hexafluorophosphate anion is disordered, but this is neither uncommon, e.g. $[RuC\ell(NO)_2(PPh_3)_2]^+[PF_6]^-\cdot C_6H_6$(34) and $[M(O_2)(Ph_2P\cdot CH_2\cdot CH_2\cdot PPh_2)]^+[PF_6]^-$(35) where M is Rh or Ir, nor unexpected in view of the almost spherical appearance of the ion to an external observer. The dimensions of the major orientation, the only one refined, are in reasonable agreement despite the disorder with the expected values; the P - F bond lengths 1.539 - 1.596, mean 1.569 Å are close to the value of 1.58 Å in $NaPF_6$ and KPF_6(17), while the F - P - F angles have a maximum deviation of 5.7° and a mean deviation of only 2.3° from ideal octahedral geometry.

In agreement with the relative population of the three orientations of the anion, the close interionic contacts involving the minor orientations are more numerous and generally shorter than those involving the major orientation. For example, the shortest contact involving both the minor orientations is an O - - - F contact of 2.77 Å, while that involving the major orientation is a similar contact of 3.04 Å. The shortest O - - - F, C - - - F, O - - - O and O - - - C interionic contacts [2.77, 2.99, 3.12 and 3.30 Å respectively] appear to be purely van der Waals' interactions.

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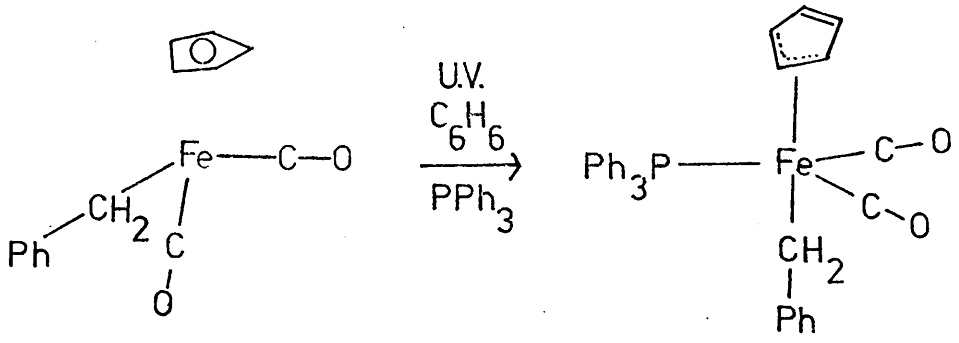
CHAPTER 3

TWO IRON COMPLEXES

III.3.1 INTRODUCTION

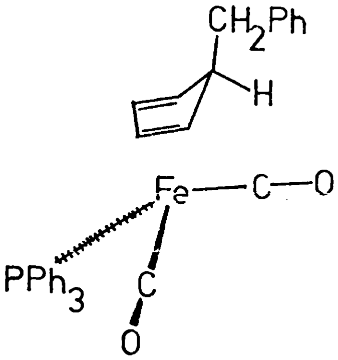
In a continuation of previous(1) studies of photochemical reactions involving organometallic compounds, Dr. G. R. Knox investigated the reaction of triphenylphosphine with I and isolated a compound, which was characterized as II by elemental analysis, an osmetric



I II

molecular weight determination and i.r. and n.m.r. spectroscopy. Confirmation of the free double bond by chemical methods proved difficult, but a tetracyanoethylene (TCNE) adduct was eventually isolated.

In order to determine unequivocally the structure of II an X-ray analysis was undertaken. However, this demonstrated the compound to be (1-exo-benzylcyclopenta-2,4-diene)dicarbonyl(triphenylphosphine)iron, III,



III

and a further X-ray analysis was therefore carried out to elucidate the structure of the TCNE adduct.

III.3.2 EXPERIMENTAL

Crystal Data (1-Exo-benzylcyclopenta-2,4-diene)dicarbonyl-(triphenylphosphine)iron; $C_{32}H_{27}O_2PFe$.

Crystal system	Monoclinic
Unit cell dimensions	a = 17.318(6) Å
	b = 11.687(4) Å
	c = 26.811(6) Å
	β = 102°18'(1)
Space group	$P2_1/c$ (C^5_{2h})

$U = 5301.6$ Å^3

$M = 530.1$ a.m.u.

$D_o = 1.33$ gm.cm.$^{-3}$ (flotation in aqueous zinc iodide solution)

$D_c = 1.328$ gm.cm.$^{-3}$

$Z = 8$

$F(000) = 2208$

$\mu(\text{Mo-}K\alpha) = 6.74$ cm.$^{-1}$

Crystallographic Measurements

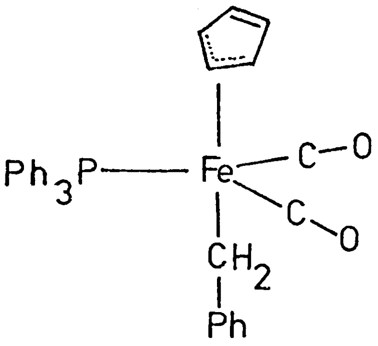
Preliminary cell dimensions, and systematically absent reflections ($h0\ell$ absent if $\ell = 2n + 1$, $0k0$ absent if $k = 2n + 1$) consistent with space group $P2_1/c$ were found from precession photographs of a yellow crystal, mounted about b*, with dimensions ca. 0.30 x 0.38 x 0.50 mm^3. $P2_1/c$ is a four-fold space group and $Z = 8$, therefore there are two crystallographically independent molecules per asymmetric unit.

The crystal, having been transfered to a Hilger and Watts' Y290 automatic diffractometer controlled by a PDP-8 computer, was offset on χ by about 4° both to prevent multiple reflections$^{(2)}$ and to enable a data set to be collected without any observations being made at χ values of about 90°. A fully stabilized X-ray generator was operated at 46 kV and 16 mA. The cell dimensions were then adjusted by a least-squares treatment$^{(3)}$ of the θ, χ and ϕ setting angles of eleven reflections measured with zirconium-filtered Mo-$\underline{K}\alpha$ radiation.

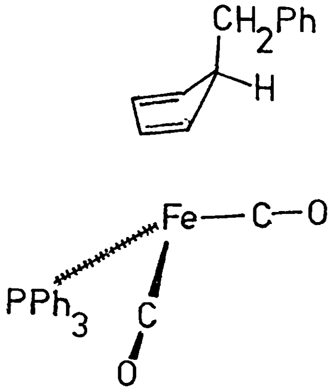
The intensity data were collected, in octants $hk\ell$ and $hk\bar{\ell}$ with $\theta(\text{Mo-}\underline{K}\alpha) \leqslant 26^\circ$, by the $\theta - 2\theta$ scan procedure, each reflection being scanned in 30 equal steps from $2\theta_{\text{calc.}} -0.60^\circ$ to $2\theta_{\text{calc.}} +0.60^\circ$. Each step was counted for 2 seconds and stationary-crystal, stationary-counter background counts were measured at each end of the scan range for 15 seconds. The intensities of two standard reflections were monitored every 40 intensity measurements and, although only small random fluctuations were observed, the orientation matrix was checked and slightly adjusted after about 6,000 measurements. The scan counts (P) and the background counts (B_1,B_2) of each reflection were combined to yield the integrated intensity, $I = P - 2(B_1 + B_2)$, which was corrected for Lorentz-polarisation effects but not for absorption. 10,421 independent values of $|F_o|$ were obtained, of which the 6,517 observations with $I/\sigma_I > 3.0$ were used in the structure analysis.

Struture Analysis

The initial coordinates of both iron and both phosphorus atoms were obtained from the three-dimensional Patterson synthesis and used to phase an electron density distribution (R = 40%), which revealed that both independent molecules in the asymmetric unit were not as expected II, but were III; a complex with an identical molecular formula to II and more consistent with the spectroscopic data.



II



III

Coordinates for all the non-hydrogen atoms were found from this electron density distribution, and after three cycles of block diagonal least-squares refinement of the positional and isotropic thermal parameters, the R-factor was 10.2%. With all the atoms assigned anisotropic thermal parameters, three cycles of refinement reached convergence at R = 7.5%. Comparison between the observed and calculated structure factors revealed six reflections with high F_o and low sinθ values which appeared to be suffering from appreciable secondary extinction and/or dead time losses, and these were removed from further refinement. A difference electron density distribution was calculated revealing the positions of all the hydrogen atoms at an average peak height of $0.47e^-/Å^3$, and no other regions of electron density higher than $0.36e^-/Å^3$. Each hydrogen atom was included in further structure factor calculations with an isotropic temperature factor equal to that of the carbon atom to which it was bonded and, after three cycles of block diagonal least-squares refinement of the parameters of the other atoms, convergence was reached at R = 5.72%, R' = 6.45%. A structure factor calculation, including the six previously removed reflections, gave R = 5.80%.

Notes:

1) The atomic scattering factors, including dispersion corrections $\Delta f'$ and $\Delta f''$ for the iron and phosphorus atoms, were taken from 'International Tables for X-ray Crystallography,' Vol. III.(4)

2) The final weights employed in the least-squares refinement, in order to ensure approximate constancy of $\langle w\Delta^2\rangle$ over various ranges of $|F_o|$ and $\sin\theta$, were given by the expression:

$$w = X \times Y$$

where:

if $0.1 \times |F_o| > |F_c|$, then $w = 0.000000001$;

if $\sin\theta > 0.3$, then $X = 1$ else $X = \sin\theta/0.3$;

if $|F_o| < 110.0$, then $Y = 1$ else $Y = 110.0/|F_o|$.

3) Nearly all the calculations were carried out on the Univac 1108 computer at the National Engineering Laboratory, East Kilbride, using the 'X-ray System' programs(5).

Results

The observed and final calculated structure factors are listed in Table 1, with the final positional and thermal parameters, including the estimated standard deviations derived from the least-squares refinement, in Table 2. The atomic numbering scheme of the non-hydrogen atoms (each hydrogen atom is numbered according to the atom to which it is bonded) is shown in Figure 1; A1 and A101 (the molecules containing Fe1 and Fe101 respectively), each projected on to its butadiene plane. The bond lengths, valency angles and mean plane calculations are given in Tables 3, 4 and 5 respectively. The crystal packing is shown in Figure 2 and the intermolecular contacts $\leqslant 3.60$ Å are listed in Table 6.

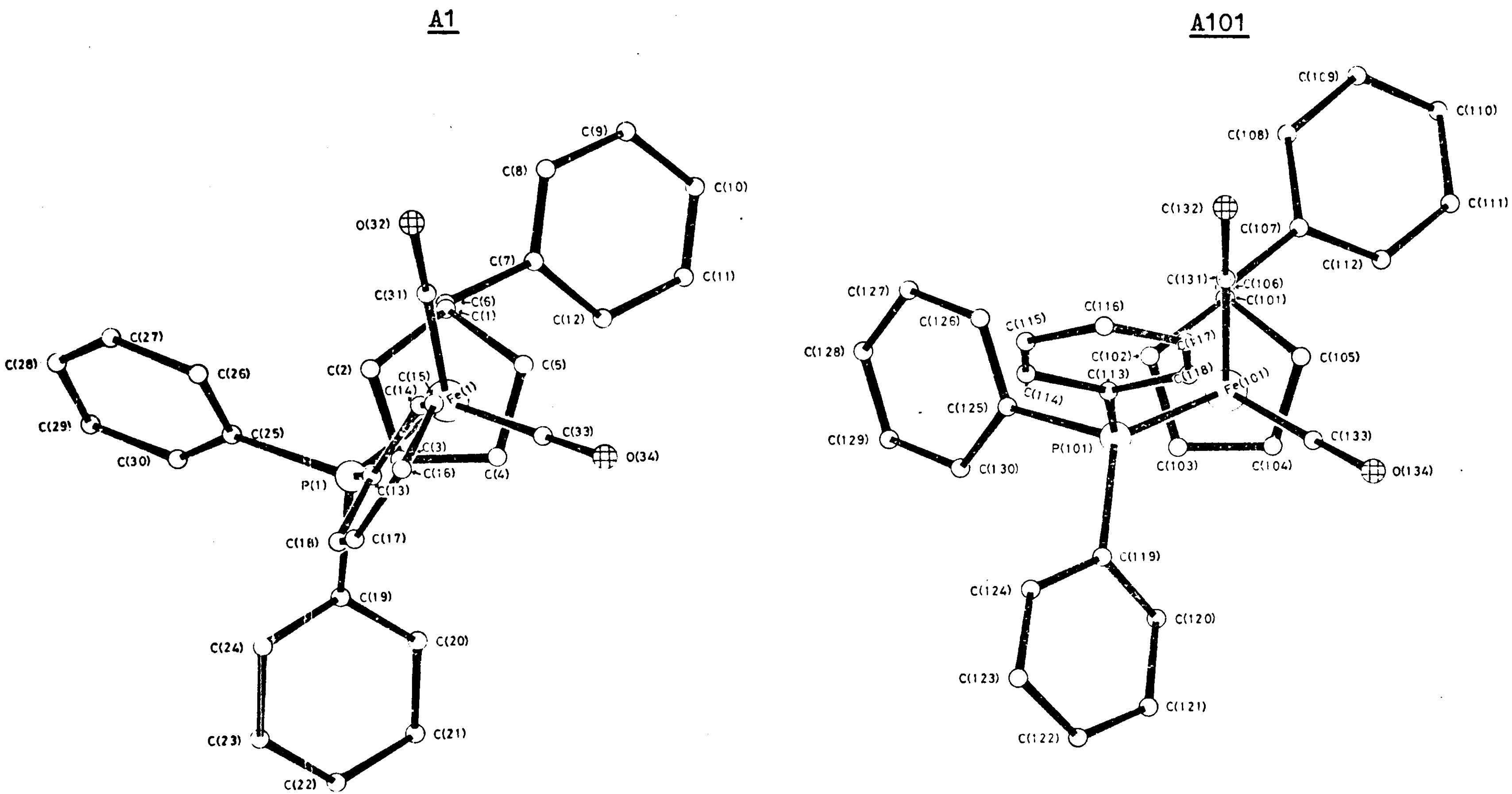


FIGURE 1

The two crystallographically independent molecules of (1-exo-benzylcyclopenta-2,4-diene)dicarbonyl(triphenylphosphine)iron; each projected on to the butadiene plane.

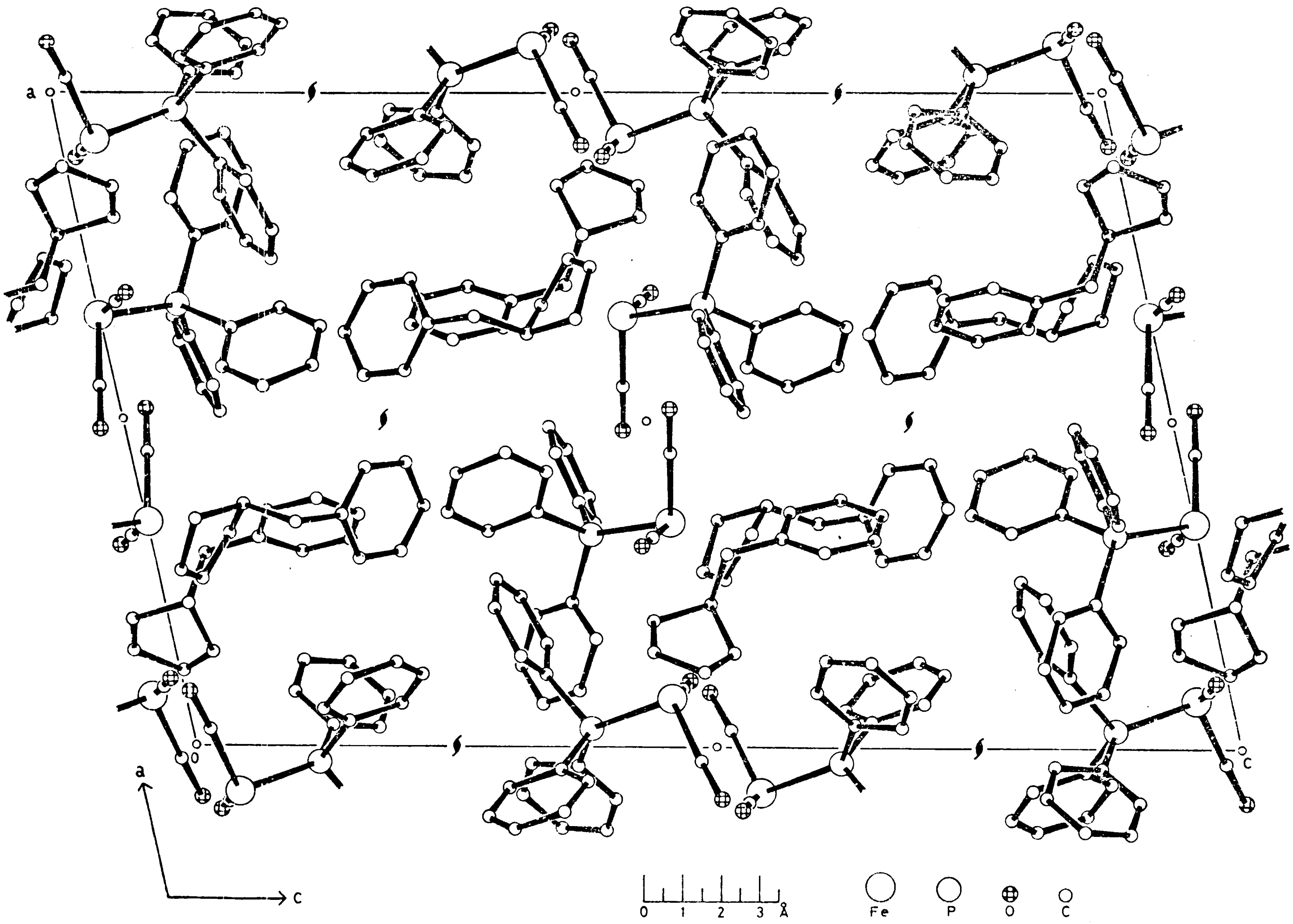


FIGURE 2 Crystal packing of (1-exo-benzylcyclopenta-2,4-diene)dicarbonyl(triphenylphosphine)iron viewed along the b-axis.

TABLE 1

(1-Exo-benzylcyclopenta-2,4-diene)dicarbonyl(triphenylphosphine)iron; observed and final calculated structure amplitudes (x10).

21,2,L		
-3	191	197
-4	147	84
-5	198	156
-7	183	161
-8	188	174
21,1,L		
-2	151	141
-4	271	271
-6	287	258
-8	275	294
-10	176	288
21,0,L		
-12	183	229
20,5,L		
-8	274	285
20,4,L		
0	356	349
-2	281	267
-4	165	86
-7	211	197
-8	186	285
-10	138	137
20,3,L		
-3	226	195
-5	212	201
-7	213	211
-8	172	156
-9	145	165
-10	400	404
-12	256	274
20,2,L		
0	207	243
-2	216	212
-3	146	79
-5	177	125
-7	176	165
-8	163	137
-12	158	183
-13	153	152
-14	147	51
-15	147	165
-16	146	123
20,1,L		
3	193	158
0	137	116
-6	175	158
-10	237	232
-14	141	164
-16	225	209
20,0,L		
4	141	88
0	248	263
-4	191	193
-6	244	229
-12	143	126
-14	192	231
-16	158	136
-18	174	186
19,6,L		
-7	182	164
-8	134	120
-10	158	132
-12	200	264
19,5,L		
-10	163	170
-12	225	242
-15	187	181
19,4,L		
4	174	200
2	145	145
-4	164	181
-5	150	128
-9	142	184
-10	153	93
-11	131	33
-12	264	284
-14	233	281
19,3,L		
4	232	245
3	156	131
2	279	243
1	148	88
0	125	12
-3	205	220
-5	225	253
-9	226	232
-10	236	241
-11	288	325
-12	203	231
-13	158	154
-15	253	259
-16	171	132
-17	212	222
-18	187	130
19,2,L		
0	161	129
-2	371	371
-4	352	352
-6	375	380
-7	176	135
-8	388	355
-10	148	87
-13	140	133
19,1,L		
8	203	163
7	161	112
4	273	264
2	325	309
-2	160	130
-5	168	120
-6	168	182
-7	163	182
-9	157	186
-10	204	304
-12	401	403
-14	195	167
-16	135	187
19,0,L		
6	140	153
0	337	331
-2	504	471
-4	420	406
-6	414	402
-8	389	380
-10	189	151
18,7,L		
1	172	160
0	285	265
-6	158	153
-7	164	97
-8	133	32
-9	192	184
18,6,L		
-4	195	158
-6	232	242
-8	334	389
-10	412	429
-12	185	171
-13	177	133
-15	186	188
18,5,L		
4	338	317
2	441	433
0	385	481
-1	148	165
-2	195	168
-3	242	243
-4	132	146
-5	158	157
-7	130	107
-8	171	146
-10	130	84
-12	188	159
-13	237	267
-18	195	194
18,4,L		
7	218	206
3	162	138
1	134	114
-1	163	111
-3	226	218
-4	200	198
-5	271	269
-6	217	207
-7	250	228
-8	336	337
-10	512	524
-14	162	103
18,3,L		
6	142	187
4	201	183
2	432	420
1	184	128
0	217	215
-1	203	175
-3	158	189
-10	195	172
-11	238	265
-12	222	231
-13	330	330
-16	237	259
-20	173	211
-22	149	84
18,2,L		
10	285	264
9	185	134
7	204	212
6	132	96
5	163	152
4	152	103
3	286	220
1	158	126
-1	164	158
-3	228	217
-5	266	262
-6	155	166
-7	219	244
-8	311	324
-9	184	177
-10	312	282
-11	136	73
-13	147	155
-16	151	142
-20	283	283
18,1,L		
10	287	176
8	151	179
6	213	215
4	136	24
2	238	246
0	157	113
-2	163	146
-4	385	385
-6	384	391
-7	161	153
-8	237	234
-10	325	317
-12	440	439
-13	274	307
-14	291	302
-16	264	234
-18	329	343
-19	137	163
-20	290	285
18,0,L		
4	154	131
0	173	144
-2	153	142
-8	264	275
-12	433	451
-14	461	471
-16	275	270
-18	240	262
-20	251	264
-22	302	342
17,8,L		
0	178	117
-2	146	122
-4	170	91
-8	163	145
-9	142	166
-10	152	124
-11	142	126
17,7,L		
5	170	155
-1	139	60
-5	247	288
-8	152	156
-10	360	345
-12	313	314
-14	208	225
-16	283	214
-17	137	139
17,6,L		
6	176	210
4	137	153
2	191	163
-1	146	75
-2	188	180
-3	153	82
-4	180	88
-8	168	203
-10	333	365
-12	133	93
-15	278	294
-17	256	257
17,5,L		
10	160	127
8	229	229
6	178	211
4	188	181
-2	143	66
-3	145	179
-5	238	251
-7	227	220
-9	195	234
-10	387	402
-12	546	573
-14	220	230
-17	161	148
-18	234	235
-20	423	395
17,4,L		
11	239	230
9	215	184
6	159	137
5	158	182
3	142	122
0	167	170
-1	193	192
-2	155	142
-3	182	155
-4	135	53
-6	196	131
-7	186	208
-8	443	462
-9	260	256
-10	299	292
-11	271	248
-12	216	213
-13	278	296
-15	348	356
-17	343	331
-19	148	173
-20	312	294
-22	318	383
17,3,L		
12	145	78
11	267	250
9	166	122
6	130	157
4	168	148
0	255	251
-1	136	162
-2	307	315
-4	183	161
-5	260	262
-7	286	336
-9	194	281
-10	181	201
-12	311	317
-14	292	277
-17	167	152
-20	175	203
17,2,L		
11	303	312
9	197	171
8	279	268
6	383	408
4	262	278
1	177	243
1	172	197
-2	129	139
-3	159	133
-4	135	84
-5	231	257
-7	345	334
-8	390	403
-9	202	211
-10	358	380
-11	184	221
-12	133	145
-13	292	298
-15	278	258
-19	133	119
-20	337	345
-21	139	144
-22	393	415
-23	177	140
-24	186	126
17,1,L		
11	176	148
10	201	182
2	378	408
0	401	409
-2	378	387
-4	337	360
-6	378	403
-8	156	129
-10	187	156
-12	207	215
-16	137	88
-17	146	93
-18	128	98
-20	181	282
17,0,L		
10	217	200
8	407	400
6	408	440
4	439	445
2	121	84
0	254	260
-2	208	215
-4	125	189
-6	413	488
-10	365	377
-12	311	348
-18	152	137
-20	533	540
-22	416	425
-24	259	295
16,9,L		
1	165	178
-1	183	189
-6	148	147
-7	140	142
-8	286	289
-9	215	200
-11	224	216
-12	215	157
16,8,L		
4	236	253
3	132	138
2	218	218
-1	142	97
-5	186	163
-7	190	219
-8	172	159
16,7,L		
5	149	152
3	281	245
2	185	179
1	170	135
-2	140	165
-3	124	10
-4	226	183
-5	202	198
-6	290	276
-8	211	217
-11	190	197
-13	241	230
-14	160	138
-16	129	80
-17	169	101
-19	133	121
16,6,L		
10	154	173
6	348	342
5	195	195
4	489	513
2	385	405
1	129	127
0	239	227
-1	162	151
-2	173	204
-3	142	134
-20	293	305
16,5,L		
12	187	169
9	191	160
8	136	135
7	140	165
0	192	247
-1	142	148
-2	201	195
-3	182	201
-5	153	176
-6	191	185
-7	155	123
-8	295	297
-9	173	99
-11	210	239
-12	233	208
-17	171	190
-18	155	184
-19	251	274
-20	184	126
16,4,L		
6	280	266
4	412	409
2	388	359
1	287	206
0	162	152
-6	162	179
-9	134	109
-10	245	224
-11	183	141
-13	152	28
-14	187	159
-16	160	155
-20	194	201
-22	494	516
-23	148	87
-24	321	328
16,3,L		
15	165	174
14	332	316
13	181	118
12	278	242
10	144	107
9	350	368
8	151	119
7	311	269
3	187	183
1	288	297
-1	274	297
-2	141	175
-5	172	156
-6	349	394
-7	135	143
-8	230	245
-9	282	183
-11	329	344
-13	281	218
-14	219	227
-15	159	129
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15,9,L		
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12,7,L

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-6 218 211
-7 436 432
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-19 210 174
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12,6,L

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12,5,L

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12,4,L

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-28 117 127

12,3,L

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-23 423 441
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-27 237 243
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12,2,L

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12,1,L

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12,0,L

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11,12,L

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11,11,L

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-14 197 172
-18 221 197

11,10,L

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-18 153 167
-19 222 184

11,9,L

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-10 228 206
-11 155 215
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-14 258 211
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-17 153 163
-18 201 235
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11,8,L

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6 281 284
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3 252 277
2 160 164
-1 178 176
-2 227 214
-3 351 384
-4 242 219
-5 282 286
-6 491 513
-7 502 515
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-9 533 514
-10 215 228
-11 635 627
-13 456 466
-15 231 241
-18 257 217
-19 211 231
-20 287 281
-21 369 370
-23 254 240

11,7,L

13 163 169
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-14 183 169
-16 135 120
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-20 172 167
-21 166 145
-22 381 375
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-26 146 172

11,6,L

20 185 148
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14 339 351
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-5 533 557
-6 687 689
-8 228 214
-9 175 222
-10 306 331
-11 542 551
-12 135 138
-13 278 285
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-15 134 141
-18 185 184
-19 128 71
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-23 144 118
-26 150 107

11,5,L

20 175 151
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-28 191 192

11,4,L

22 248 236
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11,3,L

19 142 163
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16 122 94
14 152 186
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-24 265 288
-27 236 233
-28 193 218
-29 170 154
-30 235 272

11,2,L

22 230 247
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15 168 163
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-25 310 335

-26 195 215
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11,1,L

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11,0,L

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10,12,L

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10,11,L

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10,10,L

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10,9,L

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10,8,L

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10,8,L

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-20 137 131
-22 159 142

10,7,L

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-24 145 108
-25 245 248
-26 205 158

10,6,L

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10,5,L

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-4 340 374
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-19 183 187
-20 421 419
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10,4,L

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-27 153 116
-28 297 290
-29 248 183

10,3,L

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16 174 175
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13 218 191
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-15 349 362
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-17 346 338
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-26 209 198
-27 231 245
-28 230 210
-29 165 129
-30 158 224

10,2,L

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-4 145 190
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-11 447 458
-12 163 137
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-24 195 193
-26 314 308
-27 152 152
-28 244 238
-29 170 222

10,1,L

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-23 145 179

-24 383 374
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-27 184 185
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-30 415 423

10,0,L

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-6 480 460
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9,13,L

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9,12,L

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-13 136 74
-14 236 247

9,11,L

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-4 312 307
-6 284 217
-7 185 178
-9 365 375
-11 309 273
-16 143 131

9,10,L

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9,9,L

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-21 150 137

9,8,L

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-23 134 146
-26 150 172

9,7,L

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6 183 194
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9,6,L

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9,5,L

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9,4,L

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9,3,L

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9,2,L

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9,1,L

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-28  235  187
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-18  678  696
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-26  321  312
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-18 1044 1051
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 -6 1989 1947
 -7 1231 1171
 -8  311  354
 -9  719  717
-10  186  166
-11  297  287
-12  607  592
-13  556  558
-14  929  940
-15  571  566
-17  438  436
-18  150  194
-20  171  160
-22  141  127
-23  491  487
-24  370  369
-26  428  417
-29  225  222
-30  134   64

   6,0,L

 28  199  222
 24  139  159
 18  291  269
 14  481  427
 12  553  587
 10 1143 1157
  8  282  228
  6  213  213
  4  326  270
  2  763  789
  0  636  652
 -2  580  561
 -4  740  698
 -6  796  697
 -8  432  398
-12 1397 1399
-14 1610 1628
-16 1439 1452
-18  278  299
-22  540  553
-24  810  833
-28  134   58

   5,14,L

 -1  235  214
 -2  155  128

   5,13,L

  9  140  112
  8  263  266
  6  151  193
  4  184  154
  2  189  221
  0  178  175
 -2  243  224
 -5  186  198
-11  160  178
-13  240  224

   5,12,L

 12  166  137
 10  186  188
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    5,12,L

  9   141   134
  5   316   295
  4   137   121
  3   296   301
 -1   143   196
 -2   302   284
 -4   410   417
 -6   146   160
-11   187   151
-13   149   153
-15   249   215
-17   192   236

    5,11,L

 16   133    24
 13   235   231
 11   128    23
 10   329   313
  8   267   303
  7   132   126
  6   287   283
  4   322   345
  2   480   501
  0   400   470
 -2   225   184
 -4   179   193
 -7   268   264
-11   234   212
-12   135   110
-18   191   202

    5,10,L

 19   158   143
 13   193   188
 12   267   265
 11   135   164
 10   344   343
  8   264   269
  7   196   211
  5   555   583
  3   544   565
  1   130   133
 -1   146   139
 -4   489   476
 -5   185   175
 -6   159   167
 -7   180   185
 -8   168   189
 -9   122   122
-10   132   126
-11   149   141
-15   241   266
-16   215   185
-18   279   285
-19   141   104
-20   126   132
-21   211   242
-23   298   303

    5,9,L

 20   273   294
 15   233   241
 14   202   217
  9   118   104
  8   147   139
  6   377   387
  4   293   315
  3   142   143
  2   365   384
  0   845   613
 -1   133   136
 -2   595   594
 -4   127   125
 -7   140   119
 -9   242   242
-10   146   118
-11   386   373
-12   212   207
-13   289   255
-14   204   219
-15   336   347
-18   137    96
-22   129    36
-24   208   253

    5,8,L

 21   154   128
 20   138    87
 19   293   289
 13   247   249
 12   195   211
 10   416   437
  9   289   284
  8   245   235
  7   413   396
  6   146   154
  5   503   612
  4   251   271
  3   504   510
  1   422   426
 -2   458   454
 -4   253   272
 -5   357   349
 -6   281   295
 -7   405   412
 -8   224   219
 -9   327   357
-12   305   332
-13   286   287
-15   138   115
-16   434   421
-18   401   372
-19   151   122
-20   234   258
-21   354   365
-23   338   335
-24   139   149
-25   199   138

    5,7,L

 22   289   291
 20   565   576
 19   265   262
 18   342   338
 17   165   157
 16   437   452
 15   375   331
 14   546   554
 12   299   302
 11   354   352
  9   450   479
  6   263   193
  4   170   170
  1   165    75
  0   377   370
 -1   180   154
 -2   207   148
 -3   497   471
 -4   373   373
 -5   334   342

 -6   812   828
 -7   421   436
 -8   842   849
 -9   215   207
-10   378   393
-11   284   271
-12   777   762
-13   131   108
-14   757   772
-15   269   326
-16   184   187
-18   124   127
-19   157   150
-20   183   213
-22   265   253
-24   154   166
-26   237   233

    5,6,L

 26   160   166
 24   215   199
 22   263   237
 19   277   318
 18   121    85
 17   292   298
 16   178   151
 15   130   115
 13   166   155
 12   267   304
 10   525   552
  8   424   448
  5   168   168
  3   133   192
  2   258   266
 -2   249   268
 -3   178   142
 -4   285   294
 -5   137   119
 -9   130    94
-10   408   408
-11   441   429
-12   484   465
-14   179   167
-15   138   149
-16   794   798
-17   184   175
-18   664   665
-20   194   175
-21   224   219
-22   178   187
-23   173   177
-24   202   187

    5,5,L

 23   167    71
 22   254   214
 20   462   465
 19   284   302
 18   716   733
 17   309   317
 16   775   789
 15   400   383
 14   442   449
 13   282   268
 12   448   461
 11   249   267
  7   597   618
  6   234   242
  5   552   519
  4   131   131
  3   487   456
  2   446   408
  1   226   249
  0   343   310
 -1   153   121
 -2   114    78
 -3    96    86
 -5   489   490
 -6   828   858
 -7   156   124
 -8  1460  1437
-10  1122  1094
-11   392   411
-12   824   826
-13   232   242
-14   721   678
-15   241   255
-16   273   240
-17   363   353
-18   217   195
-19   250   237
-21   272   285
-22   379   361
-26   331   319
-28   163   127

    5,4,L

 27   195   187
 26   185   188
 25   131   153
 24   241   210
 23   256   235
 22   241   213
 20   163   163
 17   347   361
 14   787   728
 13   490   516
 11   716   741
 10  1039  1052
  9   133   360
  8   559   602
  7   786   791
  4   156   143
  3   238   202
  2   863   621
  1  1496  1875
  0   159   189
 -1   852   822
 -2  1473  1441
 -3   814   797
 -4   384   352
 -5   487   510
 -6   120    84
 -8   252   236
 -9   585   611
-10   232   213
-11  1210  1168
-12   441   423
-13   829   844
-14   414   388
-15   294   298
-16   922   931
-17   487   471
-18   758   760
-19   124   304
-20   361   360
-22   326   302
-23   187   158
-25   121   132
-29   176   186

    5,3,L

 24   176   154

 22   128    99
 21   247   243
 20   335   386
 19   458   478
 18   561   568
 17   184   147
 14   511   498
 12   257   286
 10   421   422
  8   868   876
  7   628   618
  6   249   246
  5   283   195
  4  1237   956
  3   442   432
  2   836   836
  1   154    87
  0   841   853
 -1   194   159
 -2   931   946
 -3   796   734
 -5   228   174
 -6   349   322
 -8  1245  1177
 -9   557   498
-10   303   251
-11   632   626
-12   309   354
-13   713   720
-14   611   617
-15   337   350
-16   191   172
-19   302   308
-21   421   413
-22   173   166
-23   167   173
-25   272   249
-26   329   356
-27   274   297
-28   378   164
-30   151   144

    5,2,L

 28   154   143
 27   143    58
 26   197   210
 25   231   234
 24   262   273
 23   191   185
 22   289   214
 21   249   214
 20   157   147
 19   263   230
 18   202   196
 17   203   182
 16   302   297
 15   304   321
 14   496   500
 13   687   624
 12   530   552
 11   984   990
 10   931   949
  9   634   846
  8  1165  1173
  7   864   911
  6   171   172
  5  1194  1153
  3  1294  1269
  2   718   692
  1  1260  1208
  0   219   174
 -1   773   785
 -2  1048  1085
 -4   391   362
 -5   689   682
 -6   373   367
 -8   526   480
 -9   737   730
-10   281   264
-11   464   478
-12   668   657
-13   258   248
-14   436   398
-15   369   409
-16   572   593
-17   269   293
-18   487   521
-19   454   459
-20   166   162
-21   157    85
-23   197   169
-24   121   122
-25   370   364
-26   172   156
-27   138   113
-28   352   352
-30   248   242

    5,1,L

 26   251   271
 24   231   214
 23   122    43
 22   278   265
 21   192   188
 20   315   318
 19   412   411
 18   265   252
 17   128    93
 16   413   399
 14   459   458
 12   566   555
 11   279   268
 10   984   994
  8   566   613
  6   689   685
  5   575   531
  4   838   838
  3   527   535
  2   861   833
  1   361   372
  0   736   787
 -1   275   308
 -2  1858  1787
 -3  1507  1467
 -5   110    94
 -6   758   783
 -7   313   329
 -8   845   842
 -9   445   480
-10   446   421
-11   254   249
-12   120   138
-14   487   487
-16   261   236
-18   182   228
-22   277   270
-23   233   230
-24   908   580
-26   574   572
-28   528   510
-32   166   190

    5,0,L

 24   301   374

 16   377   351
 14   117   116
 12   535   522
 10   641   628
  8   224   229
  6  1130  1092
  4  1187  1119
  2   780   775
  0  2582  2620
 -2  2249  2281
 -4  1720  1742
 -6   521   478
 -8   318   348
-10   571   527
-12  1223  1154
-16   921   934
-18   716   737
-22   134   145
-26   169   138
-28   359   350
-30   250   256

    4,14,L

  3   315   266
  1   261   258
 -1   163   169
 -4   228   236

    4,13,L

 11   162   195
  7   216   188
  5   226   268
  2   283   159
  0   147   119
 -1   235   225
 -3   156   171
 -9   193   221
-12   224   236

    4,12,L

 11   197   181
  9   178   135
  7   143   208
  4   157   127
  3   194   221
  1   210   222
 -1   192   178
 -2   197   180
 -4   439   439
 -6   342   336
 -7   179   155
 -8   227   209
 -9   181   151
-10   345   319
-12   338   343
-17   185   120

    4,11,L

 18   198   180
 17   176   216
 16   163   152
 14   202   234
 12   343   319
 10   285   288
  7   289   309
  5   172   157
  2   211   189
 -1   152   158
 -2   129   119
 -5   186   112
 -7   357   353
 -9   180   148
-10   351   353
-11   243   255
-12   421   429
-14   367   367
-16   272   253
-18   228   247
-19   158   183
-20   165   142

    4,10,L

 20   139   157
 19   177   138
 17   216   226
 16   131   171
 15   245   281
 14   145   143
  9   216   219
  8   238   254
  7   485   504
  6   152   182
  5   305   303
  4   184   199
  1   152   137
 -1   219   195
 -2   151   168
 -3   242   276
 -4   347   333
 -5   186   184
 -6   288   285
 -8   281   269
 -9   344   327
-10   254   262
-11   260   290
-12   349   312
-17   297   282
-19   411   429
-20   191   181

    4,9,L

 21   179   203
 19   446   472
 18   268   240
 17   361   368
 16   129   158
 15   195   154
 14   289   213
 13   223   227
 12   193   228
 11   198   191
  6   138   148
  5   188   146
  4   225   244
  3   256   252
  0   362   341
 -1   169   189
 -2   279   280
 -3   163   168
 -6   238   222
 -7   688   652
 -8   141   131
 -9   613   620
-10   301   300
-12   530   563
-13   124    60
-14   499   488
-15   132   117
-16   235   283
-17   139   136
-18   147    98

-20   183   151
-23   216   240
-28   223   215

    4,8,L

 22   177   212
 21   167   129
 18   258   254
 17   178   164
 13   134   131
 12   330   342
 11   149   193
 10   368   366
  9   319   312
  8   586   510
  7   602   622
  6   321   348
  5   378   368
  4   279   254
  2   459   438
  1   186   141
  0   152   158
 -1   493   492
 -2   559   531
 -3   226   232
 -4   829   898
 -5   435   431
 -6   418   418
 -7   486   486
 -8   285   273
-10   195   198
-12   454   437
-13   142   180
-16   183   191
-17   382   283
-18   293   324
-19   332   333
-20   155   142
-21   210   178
-24   211   187

    4,7,L

 20   204   243
 19   169   169
 17   572   569
 15   291   295
 14   302   311
 13   169   128
 12   129   120
 10   130   126
  8   428   458
  7   177   170
  6   782   753
  5   242   247
  4   540   581
  3   554   542
  2   818   844
  1   265   302
  0   803   821
 -1   168   173
 -2   475   491
 -4   419   422
 -6   259   264
 -7   568   564
 -8   153   155
 -9   437   431
-11   149    80
-12   511   520
-13   344   348
-14   357   342
-17   186   160
-18   331   324
-20   183   192
-21   257   271
-23   274   292
-24   315   304
-25   178   168
-26   366   361
-28   331   327

    4,6,L

 25   134    56
 22   227   286
 20   221   250
 16   516   536
 15   268   253
 12   255   276
 11   239   250
 10   667   707
  9   454   455
  8  1027  1080
  7   506   511
  6  1056  1082
  5   158   191
  4   220   287
  3   624   594
  2  1048  1361
  1   264   317
  0   271   317
 -1   677   638
 -2   523   508
 -4   410   414
 -5   113   108
 -6   282   220
 -8   114    27
-12   157   132
-13   235   246
-14   180   151
-15   114    30
-16   311   243
-17   176   163
-18   632   640
-19   163   147
-20   229   189
-22   313   284
-24   528   530
-28   167   127
-29   122   114

    4,5,L

 28   180   190
 25   133    99
 24   257   284
 19   200   197
 18   258   262
 15   144   155
 14   149   121
 13   430   433
 12   337   336
 11   270   283
  9   442   437
  8   430   473
  7   303   371
  6   872   910
  5   259   284
  4   749   775
  3   153   161
  2   586   635
  0   772   794
 -1   797   720
 -2   482   810
 -3   684   644

    4,5,L

 -4   378   351
 -6   911   837
 -7   126   154
 -9   545   514
-10   403   426
-11   174   187
-12   669   685
-13   364   355
-14   790   782
-15   265   257
-17   140   162
-18   531   542
-19   178   162
-20   438   439
-21   312   292
-24   421   432
-26   460   472
-28   369   389
-30   342   354

    4,4,L

 28   151   127
 27   169   171
 25   211   213
 24   153   164
 22   273   249
 21   156   154
 20   395   403
 18   287   284
 16   531   534
 15   139   154
 14   127   111
 11   397   388
 10   711   743
  9   166   157
  8   837   850
  7   285   254
  6   531   582
  5   256   198
  4   886   903
  3   843   836
  2  1508  1451
  1   692   678
  0   420   385
 -1   495   513
 -2   867   819
 -3   242   214
 -4   973   956
 -6   377   366
 -7   184    74
-10   150   169
-11   279   280
-12   678   618
-13   194   218
-14   509   470
-15   604   581
-16   273   264
-17   314   298
-18   478   478
-19   192   198
-21   255   250
-22   446   428
-23   228   228
-24   570   589
-25   212   210
-27   163   162
-29   164   181
-30   241   219
-31   156   148

    4,3,L

 26   156    59
 25   194   137
 24   227   242
 22   161   146
 20   128   124
 19   328   329
 18   201   182
 17   242   263
 16   205   199
 15   636   640
 14   343   331
 13   777   775
 12   768   788
 11   339   348
 10   379   404
  9   623   666
  8   476   474
  7   279   281
  6   220   184
  5   522   521
  4  1196  1112
  3   371   308
  2   125    92
  1  1249  1205
  0   780   742
 -1  1466  1426
 -2   706   679
 -3    86    74
 -4   401   363
 -6   537   585
 -7   490   423
 -8   134   125
 -9   271   288
-10   148   162
-11   344   336
-12   723   713
-13   507   494
-14   876   859
-15   213   248
-16   288   285
-17   193   204
-18   355   351
-19   168   191
-20   189   148
-21   121   103
-23   165   121
-25   128   117
-26   261   230
-28   288   302
-32   150   168

    4,2,L

 28   156   111
 27   140   141
 26   201   170
 25   147   166
 24   520   531
 22   891   929
 21   242   250
 20   551   555
 18   177   163
 14   249   272
 13   285   293
 12   497   495
 11   399   394
 10   252   272
  9   429   457
  8   418   394
  7   570   565
  5   276   222
  4   630   601

  2   855   830
  1  1881  1670
  0   454   460
 -1   741   699
 -2  1132  1097
 -3   962   922
 -4   152   172
 -5   186   174
 -6   355   354
 -7    91    81
 -8   134    94
-11   371   370
-12   278   256
-13   744   761
-14   129   115
-15   932   932
-16   385   375
-17   716   656
-18   250   251
-19   185   181
-21   534   527
-22   243   222
-23   403   384
-24   355   372
-25   146   124
-27   240   262
-28   389   291
-29   249   260
-30   234   237

    4,1,L

 22   271   267
 21   133    81
 20   193   186
 19   177   118
 18   437   431
 16   521   544
 14   801   938
 13   286   266
 12  1420  1473
 11   331   333
 10   350   390
  9   140   119
  8   337   337
  7   261   247
  6   802   730
  5   677   637
  4   238   209
  3   442   402
  2  1185  1195
  1   119    97
  0   846   842
 -1   481   484
 -2   962   954
 -3   341   351
 -4   732   707
 -6   883   851
 -7  1272  1250
 -9  1673  1595
-10   273   315
-11   987   955
-12  1037  1054
-13   196   168
-14  1546  1559
-15   834   639
-16  1038  1056
-18   245   216
-19   370   371
-20   187   190
-22   621   610
-24   493   493
-25   128   125
-26   148   132

    4,0,L

 30   298   305
 28   282   189
 26   365   343
 24   751   766
 22  1114  1137
 16   284   312
 14   345   352
 12   445   441
 10   127    82
  8   856   827
  6   528   459
  4   824   889
  2  1977  1943
  0  2972  3158
 -2  1752  1697
 -4  2209  2242
 -6  1284  1234
 -8  1197  1182
-10  1194  1168
-12  1180  1152
-14  1143  1153
-18   780   788
-20   452   435
-22   792   815
-28   288   284
-30   237   241

    3,14,L

  4   148   130
 -3   142   161
 -5   174   202

    3,13,L

 10   165   122
  7   158   172
  6   165   168
  5   196   265
  1   324   315
  0   312   331
 -1   242   236
 -2   299   312
 -5   142   127
 -7   199   192
 -9   245   246
-11   189   172
-12   148    92
-13   213   235
-14   179   166

    3,12,L

 15   190   195
 13   194   157
 11   136   142
 10   230   198
  8   351   324
  6   335   352
  4   265   279
  3   152   153
  2   287   252
  0   285   288
 -1   189   208
 -2   196   155
 -3   269   271
 -4   142   141
 -5   176   155
 -8   153    98
-11   187   186

-13   148   134
-14   174   168

    3,11,L

 14   187   194
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TABLE 2

(1-Exo-benzylcyclopenta-2,4-diene)dicarbonyl(triphenylphosphine)iron; fractional atomic coordinates with esd in parentheses and thermal parameters.

Atom	x	y	z	U_{iso}(x10^3Å^2)
Fe(1)	0.34386(4)	0.33664(6)	0.50311(3)	*
P(1)	0.32455(7)	0.27151(11)	0.42389(5)	*
C(1)	0.3701(3)	0.2512(5)	0.5973(2)	*
C(2)	0.3467(3)	0.1853(5)	0.5472(2)	*
C(3)	0.2683(3)	0.2162(5)	0.5227(2)	*
C(4)	0.2549(3)	0.3251(5)	0.5423(2)	*
C(5)	0.3252(3)	0.3586(5)	0.5775(2)	*
C(6)	0.3430(4)	0.1924(6)	0.6430(2)	*
C(7)	0.3525(4)	0.2734(5)	0.6884(2)	*
C(8)	0.4246(4)	0.2887(6)	0.7214(2)	*
C(9)	0.4329(5)	0.3640(8)	0.7623(3)	*
C(10)	0.3679(5)	0.4235(7)	0.7703(3)	*
C(11)	0.2968(5)	0.4089(8)	0.7380(3)	*
C(12)	0.2868(4)	0.3333(7)	0.6963(3)	*
C(13)	0.3595(3)	0.3623(4)	0.3769(2)	*
C(14)	0.4197(3)	0.4398(5)	0.3939(2)	*
C(15)	0.4477(4)	0.5095(6)	0.3588(3)	*
C(16)	0.4158(4)	0.5001(6)	0.3076(3)	*
C(17)	0.3563(4)	0.4229(5)	0.2910(2)	*
C(18)	0.3283(4)	0.3533(5)	0.3250(2)	*
C(19)	0.2208(3)	0.2437(7)	0.3915(2)	*
C(20)	0.1628(3)	0.3101(5)	0.4060(2)	*
C(21)	0.0842(4)	0.2970(6)	0.3809(3)	*
C(22)	0.0640(4)	0.2171(6)	0.3418(3)	*
C(23)	0.1212(4)	0.1503(6)	0.3285(3)	*
C(24)	0.1995(4)	0.1632(5)	0.3530(2)	*
C(25)	0.3741(3)	0.1348(4)	0.4185(2)	*
C(26)	0.4484(3)	0.1344(5)	0.4067(2)	*
C(27)	0.4875(4)	0.0313(6)	0.4059(3)	*
C(28)	0.4543(4)	-0.0706(6)	0.4173(3)	*
C(29)	0.3808(4)	-0.0694(5)	0.4291(3)	*
C(30)	0.3413(3)	0.0330(5)	0.4304(2)	*

Atom	x	y	z	$U_{iso}(x10^3 Å^2)$
C(31)	0.4482(3)	0.3404(5)	0.5156(2)	*
O(32)	0.5159(2)	0.3417(4)	0.5243(2)	*
C(33)	0.3206(3)	0.4781(5)	0.4823(2)	*
O(34)	0.3061(3)	0.5709(4)	0.4703(2)	*
H(1)	0.430	0.267	0.608	46
H(2)	0.372	0.114	0.540	46
H(3)	0.228	0.147	0.498	46
H(4)	0.205	0.378	0.536	47
H(5)	0.327	0.439	0.595	48
H(6a)	0.384	0.104	0.659	56
H(6b)	0.283	0.153	0.625	56
H(8)	0.482	0.244	0.722	65
H(9)	0.487	0.355	0.793	80
H(10)	0.372	0.496	0.800	85
H(11)	0.242	0.449	0.740	90
H(12)	0.236	0.328	0.836	75
H(14)	0.441	0.455	0.435	54
H(15)	0.502	0.546	0.374	63
H(16)	0.441	0.547	0.282	65
H(17)	0.324	0.418	0.252	62
H(18)	0.283	0.289	0.310	50
H(20)	0.168	0.354	0.437	48
H(21)	0.047	0.335	0.399	63
H(22)	0.000	0.220	0.327	70
H(23)	0.131	0.086	0.298	56
H(24)	0.238	0.089	0.355	51
H(26)	0.484	0.221	0.405	52
H(27)	0.553	0.026	0.405	68
H(28)	0.482	-0.140	0.412	68
H(29)	0.357	-0.141	0.432	58
H(30)	0.278	0.029	0.432	51
Fe(101)	0.92891(4)	0.24183(6)	0.53441(3)	*
P(101)	0.97405(7)	0.26554(11)	0.61756(5)	*
C(101)	0.7828(3)	0.2315(5)	0.4783(2)	*

Atom	x	y	z	U_{iso}(x10^{3}Å^{2})
C(102)	0.8089(3)	0.2055(5)	0.5352(2)	*
C(103)	0.8566(3)	0.1055(5)	0.5418(2)	*
C(104)	0.8870(3)	0.0943(5)	0.4970(2)	*
C(105)	0.8579(3)	0.1871(5)	0.4647(2)	*
C(106)	0.7060(3)	0.1702(6)	0.4520(2)	*
C(107)	0.6816(3)	0.2036(5)	0.3968(2)	*
C(108)	0.6404(4)	0.3057(6)	0.3829(2)	*
C(109)	0.6229(4)	0.3421(8)	0.3324(3)	*
C(110)	0.6459(4)	0.2772(7)	0.2953(2)	*
C(111)	0.6849(4)	0.1765(7)	0.3082(2)	*
C(112)	0.7027(4)	0.1394(5)	0.3580(2)	*
C(113)	1.0313(3)	0.3959(5)	0.6375(2)	*
C(114)	1.0280(3)	0.4545(5)	0.6826(2)	*
C(115)	1.0754(4)	0.5492(5)	0.6971(2)	*
C(116)	1.1265(4)	0.5887(6)	0.6676(3)	*
C(117)	1.1299(4)	0.5317(6)	0.6229(3)	*
C(118)	1.0828(3)	0.4364(5)	0.6078(2)	*
C(119)	1.0366(3)	0.1557(5)	0.6550(2)	*
C(120)	1.0555(4)	0.0567(5)	0.6319(2)	*
C(121)	1.1014(5)	-0.0272(6)	0.6607(3)	*
C(122)	1.1290(4)	-0.0134(7)	0.7121(3)	*
C(123)	1.1109(4)	0.0850(6)	0.7358(2)	*
C(124)	1.0651(4)	0.1684(5)	0.7076(2)	*
C(125)	0.8903(3)	0.2759(4)	0.6490(2)	*
C(126)	0.8437(3)	0.3740(5)	0.6420(2)	*
C(127)	0.7718(3)	0.3751(6)	0.6570(2)	*
C(128)	0.7455(4)	0.2793(6)	0.6779(2)	*
C(129)	0.7912(4)	0.1823(6)	0.6851(3)	*
C(130)	0.8639(3)	0.1791(5)	0.6707(2)	*
C(131)	0.9127(3)	0.3899(5)	0.5214(2)	*
O(132)	0.9015(3)	0.4852(4)	0.5129(2)	*
C(133)	1.0226(3)	0.2210(5)	0.5195(2)	*
O(134)	1.0806(3)	0.2063(5)	0.5072(2)	*
H(101)	0.775	0.322	0.472	42

Atom	x	y	z	$U_{iso}(x10^3Å^2)$
H(102)	0.783	0.261	0.560	43
H(103)	0.875	0.052	0.578	50
H(104)	0.922	0.026	0.492	47
H(105)	0.857	0.197	0.428	44
H(106a)	0.671	0.185	0.473	50
H(106b)	0.708	0.085	0.458	50
H(108)	0.625	0.341	0.418	65
H(109)	0.590	0.420	0.317	81
H(110)	0.637	0.287	0.253	74
H(111)	0.704	0.128	0.278	70
H(112)	0.734	0.067	0.370	56
H(114)	0.992	0.418	0.706	48
H(115)	1.068	0.589	0.734	55
H(116)	1.173	0.647	0.674	63
H(117)	1.162	0.574	0.598	64
H(118)	1.067	0.391	0.571	52
H(120)	1.032	0.057	0.593	53
H(121)	1.119	-0.097	0.640	72
H(122)	1.172	-0.059	0.739	71
H(123)	1.129	0.118	0.775	63
H(124)	1.045	0.232	0.726	52
H(126)	0.858	0.452	0.628	48
H(127)	0.736	0.431	0.640	55
H(128)	0.695	0.290	0.688	63
H(129)	0.775	0.104	0.701	65
H(130)	0.891	0.093	0.680	47

* These atoms were assigned anisotropic temperature factors of the form:

$$T = \exp\left[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}\ell^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}h\ell a^*c^* + 2U_{23}k\ell b^*c^*)\right]$$

with final parameters ($U_{ij}x10^4Å^2$):

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Fe(1)	376	391	374	11	116	-18
P(1)	369	361	383	12	108	-19
C(1)	529	465	381	7	127	54
C(2)	488	429	446	-31	161	31
C(3)	394	544	454	-35	167	53
C(4)	434	597	448	133	254	107
C(5)	609	447	387	57	249	25
C(6)	674	638	432	-90	127	85
C(7)	714	558	391	-7	188	158
C(8)	582	866	522	-87	158	52
C(9)	819	1050	545	-142	185	56
C(10)	1262	785	500	51	291	73
C(11)	1107	1031	648	434	360	249
C(12)	871	926	567	275	327	151
C(13)	430	346	481	22	170	12
C(14)	528	564	518	-136	183	-18
C(15)	570	669	680	-137	210	16
C(16)	747	600	650	6	385	108
C(17)	897	528	440	146	296	45
C(18)	711	409	377	26	169	-3
C(19)	385	356	421	-7	68	25
C(20)	428	604	431	61	106	72
C(21)	453	796	691	104	94	118
C(22)	502	741	863	-114	-74	158
C(23)	704	548	669	-101	-105	57
C(24)	623	420	505	8	13	-30
C(25)	422	412	303	50	76	-33
C(26)	390	478	737	84	181	2
C(27)	522	665	982	137	294	109
C(28)	661	555	986	281	225	138
C(29)	594	423	794	72	151	41
C(30)	486	431	642	42	170	22
C(31)	456	473	393	-53	67	-34
O(32)	414	889	821	-125	58	53
C(33)	487	499	415	90	182	-33
O(34)	914	482	827	190	341	88

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Fe(101)	376	385	320	17	111	-9
P(101)	392	356	327	38	97	14
C(101)	402	523	349	7	29	-36
C(102)	382	510	407	-31	96	-15
C(103)	471	405	545	-84	47	34
C(104)	529	378	530	-16	105	-126
C(105)	508	478	330	0	73	-92
C(106)	548	698	403	-169	68	32
C(107)	389	620	487	-150	52	-90
C(108)	676	787	531	155	117	-53
C(109)	784	988	642	255	85	121
C(110)	728	1063	481	8	54	-8
C(111)	731	853	513	-12	82	-201
C(112)	580	552	593	-21	28	-60
C(113)	428	408	367	-6	28	25
C(114)	581	462	430	9	69	-36
C(115)	607	503	561	-1	-63	-86
C(116)	609	514	825	-122	-65	22
C(117)	642	573	753	-112	145	1
C(118)	496	516	541	-59	104	18
C(119)	361	471	385	30	91	62
C(120)	578	504	519	178	142	34
C(121)	855	697	659	288	200	45
C(122)	579	782	707	199	80	206
C(123)	697	689	528	156	14	65
C(124)	643	533	445	142	26	65
C(125)	422	431	270	29	94	-13
C(126)	544	384	428	102	161	35
C(127)	530	667	495	155	152	-32
C(128)	520	857	598	109	311	28
C(129)	607	790	640	0	291	178
C(130)	502	504	454	27	154	145
C(131)	454	489	373	32	104	-7
O(132)	752	430	760	116	150	141
C(133)	510	537	482	28	179	-62
O(134)	552	959	1046	22	444	-151

Average estimated standard deviations (U_{ij}x10^4Å^2)

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Fe	3	4	3	3	3	3
P	6	7	6	6	5	6
O	28	30	32	24	24	25
C	36	38	35	31	28	30

TABLE 3

(1-Exo-benzylcyclopenta-2,4-diene)dicarbonyl(triphenylphosphine)iron; bond lengths (Å) with esd in parentheses.

Bond†	Molecule A1	Molecule A101
Fe(1) - P(1)	2.213(2)	2.216(2)
Fe(1) - C(31)	1.767(5)	1.775(6)
Fe(1) - C(33)	1.763(6)	1.768(6)
C(31) - O(32)	1.148(7)	1.145(7)
C(33) - O(34)	1.145(7)	1.137(8)
Fe(1) - C(2)	2.123(6)	2.126(5)
Fe(1) - C(3)	2.065(6)	2.062(6)
Fe(1) - C(4)	2.046(6)	2.049(6)
Fe(1) - C(5)	2.102(6)	2.106(5)
C(1) - C(2)	1.527(7)	1.526(7)
C(2) - C(3)	1.425(7)	1.419(8)
C(3) - C(4)	1.414(8)	1.417(9)
C(4) - C(5)	1.427(7)	1.412(8)
C(5) - C(1)	1.513(8)	1.516(8)
C(1) - C(6)	1.562(9)	1.543(8)
C(6) - C(7)	1.521(8)	1.504(8)
C(7) - C(8)	1.381(8)	1.399(9)
C(8) - C(9)	1.388(10)	1.390(10)
C(9) - C(10)	1.379(12)	1.374(11)
C(10) - C(11)	1.355(11)	1.364(11)
C(11) - C(12)	1.406(11)	1.374(9)
C(12) - C(7)	1.390(10)	1.393(9)
P(1) - C(13)	1.844(6)	1.835(5)
P(1) - C(19)	1.850(5)	1.834(5)
P(1) - C(25)	1.833(5)	1.831(6)
C(13) - C(14)	1.384(8)	1.401(8)
C(14) - C(15)	1.409(10)	1.383(8)
C(15) - C(16)	1.368(9)	1.384(10)
C(16) - C(17)	1.370(9)	1.385(10)
C(17) - C(18)	1.386(9)	1.388(9)
C(18) - C(13)	1.386(7)	1.398(9)

Bond†			Molecule A1	Molecule A101
C(19)	-	C(20)	1.388(8)	1.384(8)
C(20)	-	C(21)	1.392(8)	1.389(9)
C(21)	-	C(22)	1.392(10)	1.369(10)
C(22)	-	C(23)	1.368(10)	1.381(10)
C(23)	-	C(24)	1.384(9)	1.376(9)
C(24)	-	C(19)	1.388(8)	1.406(7)
C(25)	-	C(26)	1.390(8)	1.391(7)
C(26)	-	C(27)	1.385(9)	1.389(9)
C(27)	-	C(28)	1.385(10)	1.371(10)
C(28)	-	C(29)	1.376(10	1.373(10)
C(29)	-	C(30)	1.382(9)	1.393(9)
C(30)	-	C(25)	1.385(8)	1.393(8)
Range	$C(sp^2)$ - H		0.95 - 1.17	0.95 - 1.15
Mean	$C(sp^2)$ - H		1.07	1.07
Range	$C(sp^3)$ - H		1.02 - 1.28	0.93 - 1.07
Mean	$C(sp^3)$ - H		1.15	1.00

† The atomic numbering refers to A1. The numbering of A101 is 100 plus that in A1.

TABLE 4

(1-Exo-benzylcyclopenta-2,4-diene)dicarbonyl(triphenylphosphine)iron; valency angles (°) with esd in parentheses.

	Molecule A1	Molecule A101
P(1) - Fe(1) - C(31)	97.5(2)	95.8(2)
P(1) - Fe(1) - C(33)	92.4(2)	94.7(2)
C(31) - Fe(1) - C(33)	101.1(3)	101.8(3)
Fe(1) - C(31) - O(32)	179.0(5)	179.4(5)
Fe(1) - C(33) - O(34)	178.0(5)	176.1(5)
C(2) - Fe(1) - C(3)	39.8(2)	39.6(2)
C(2) - Fe(1) - C(4)	65.8(2)	65.9(2)
C(2) - Fe(1) - C(5)	64.1(2)	64.0(2)
C(3) - Fe(1) - C(4)	40.2(2)	40.3(2)
C(3) - Fe(1) - C(5)	66.9(2)	66.3(2)
C(4) - Fe(1) - C(5)	40.2(2)	39.7(2)
Fe(1) - C(2) - C(1)	92.3(3)	91.8(3)
Fe(1) - C(2) - C(3)	67.9(3)	67.8(3)
Fe(1) - C(3) - C(2)	72.3(3)	72.6(3)
Fe(1) - C(3) - C(4)	69.2(3)	69.3(3)
Fe(1) - C(4) - C(3)	70.6(3)	70.3(3)
Fe(1) - C(4) - C(5)	72.0(3)	72.3(3)
Fe(1) - C(5) - C(4)	67.8(3)	68.0(3)
Fe(1) - C(5) - C(1)	93.5(3)	92.9(3)
C(1) - C(2) - C(3)	109.0(5)	109.2(5)
C(2) - C(3) - C(4)	105.8(4)	106.4(5)
C(3) - C(4) - C(5)	108.0(5)	107.5(5)
C(4) - C(5) - C(1)	107.9(5)	109.2(5)
C(5) - C(1) - C(2)	95.1(4)	94.9(4)
C(5) - C(1) - C(6)	115.0(5)	115.6(5)
C(2) - C(1) - C(6)	113.6(5)	114.0(5)
C(1) - C(6) - C(7)	110.7(5)	111.3(5)
C(6) - C(7) - C(8)	121.5(6)	120.2(6)
C(6) - C(7) - C(2)	118.9(5)	122.2(5)
C(12) - C(7) - C(8)	119.6(6)	117.5(5)
C(7) - C(8) - C(9)	121.0(7)	120.9(7)
C(8) - C(9) - C(10)	119.5(6)	119.9(7)
C(9) - C(10) - C(11)	119.8(7)	119.8(7)
C(10) - C(11) - C(12)	121.9(8)	121.0(7)
C(11) - C(12) - C(7)	118.1(6)	121.0(6)

	Molecule A1	Molecule A101
Fe(1) - P(1) - C(13)	116.9(2)	116.7(2)
Fe(1) - P(1) - C(19)	116.3(2)	120.2(2)
Fe(1) - P(1) - C(25)	113.5(2)	109.1(2)
C(13) - P(1) - C(19)	102.0(2)	101.7(2)
C(13) - P(1) - C(25)	102.7(2)	104.1(3)
C(19) - P(1) - C(25)	103.5(2)	103.2(2)
P(1) - C(13) - C(14)	119.1(4)	122.5(4)
P(1) - C(13) - C(18)	121.8(4)	119.1(4)
C(18) - C(13) - C(14)	119.1(5)	118.4(5)
C(13) - C(14) - C(15)	120.1(5)	120.1(6)
C(14) - C(15) - C(16)	120.1(6)	121.4(6)
C(15) - C(16) - C(17)	119.6(7)	119.0(6)
C(16) - C(17) - C(18)	121.1(5)	120.4(7)
C(17) - C(18) - C(13)	120.0(5)	120.8(6)
P(1) - C(19) - C(20)	117.3(4)	120.6(4)
P(1) - C(19) - C(24)	123.1(4)	121.2(4)
C(24) - C(19) - C(20)	119.6(5)	118.2(5)
C(19) - C(20) - C(21)	119.7(5)	120.2(5)
C(20) - C(21) - C(22)	120.1(6)	120.8(7)
C(21) - C(22) - C(23)	120.0(6)	119.8(7)
C(22) - C(23) - C(24)	120.3(6)	119.9(6)
C(23) - C(24) - C(19)	120.4(6)	121.1(6)
P(1) - C(25) - C(26)	119.5(4)	119.0(4)
P(1) - C(25) - C(30)	120.6(4)	120.1(4)
C(30) - C(25) - C(26)	119.6(5)	119.5(5)
C(25) - C(26) - C(27)	119.2(6)	119.9(5)
C(26) - C(27) - C(28)	121.1(7)	120.5(6)
C(27) - C(28) - C(29)	119.4(6)	120.0(6)
C(28) - C(29) - C(30)	120.1(6)	120.7(7)
C(29) - C(30) - C(25)	120.6(6)	119.4(6)
Range 'butadiene' H - C - C	119 - 135	112 - 136
Mean 'butadiene' H - C - C	126	125
Range phenyl H - C - C	98 - 142	107 - 144
Mean phenyl H - C - C	119	121
Range H - C(sp^3) - C	104 - 122	104 - 118
Mean H - C(sp^3) - C	111	112
H - C(sp^3) - H	102	95

TABLE 5

(1-Exo-benzylcyclopenta-2,4-diene)dicarbonyl(triphenylphosphine)iron; mean plane calculations.

	Atoms in plane	Displacements (Å)	Atoms out of plane	Displacements (Å)
a)	C(2)	-0.003	H(2)	0.02
	C(3)	0.005	H(3)	-0.15
	C(4)	-0.005	H(4)	-0.05
	C(5)	0.003	H(5)	0.03
			C(1)	-0.584
			Fe(1)	1.721
b)	C(1)		C(3)	-0.774
	C(2)		C(4)	-0.779
	C(5)			
c)	C(1)		H(2)	-0.30
	C(2)			
	C(3)			
d)	C(1)		H(5)	-0.36
	C(5)			
	C(4)			
e)	C(7)	0.003	C(6)	0.017
	C(8)	-0.003	H(8)	-0.06
	C(9)	0.003	H(9)	-0.27
	C(10)	-0.003	H(10)	0.14
	C(11)	0.002	H(11)	0.00
	C(12)	-0.002	H(12)	0.20
f)	C(13)	-0.005	P(1)	0.011
	C(14)	0.002	H(14)	-0.11
	C(15)	0.000	H(15)	0.28
	C(16)	0.000	H(16)	0.09
	C(17)	-0.003	H(17)	-0.11
	C(18)	0.006	H(18)	0.07

	Atoms in plane	Displacements (Å)	Atoms out of plane	Displacements (Å)
g)	C(19)	0.009	P(1)	0.101
	C(20)	-0.006	H(20)	-0.22
	C(21)	-0.002	H(21)	-0.21
	C(22)	0.008	H(22)	0.01
	C(23)	-0.005	H(23)	0.10
	C(24)	-0.003	H(24)	-0.47
h)	C(25)	0.008	P(1)	-0.112
	C(26)	-0.004	H(26)	-0.22
	C(27)	0.001	H(27)	-0.23
	C(28)	-0.002	H(28)	0.11
	C(29)	0.006	H(29)	0.12
	C(30)	-0.009	H(30)	0.20
i)	P(1)	0.061	Fe(1)	-0.547
	C(33)	-0.071		
	C(4,5)[†]	0.108		
	C(2,3)[†]	-0.099		
j)	C(102)	-0.001	H(102)	0.20
	C(103)	0.002	H(103)	0.14
	C(104)	-0.002	H(104)	-0.04
	C(105)	0.001	H(105)	-0.20
			C(101)	-0.559
			Fe(101)	1.726
k)	C(101)		C(103)	-0.736
	C(102)		C(104)	-0.736
	C(105)			
l)	C(101)		H(102)	-0.09
	C(102)			
	C(103)			
m)	C(101)		H(105)	-0.47
	C(105)			
	C(104)			

	Atoms in plane	Displacements (Å)	Atoms out of plane	Displacements (Å)
n)	C(107)	0.008	C(106)	0.120
	C(108)	-0.005	H(108)	-0.07
	C(109)	-0.003	H(109)	-0.03
	C(110)	0.007	H(110)	-0.02
	C(111)	-0.003	H(111)	0.04
	C(112)	-0.004	H(112)	0.04
o)	C(113)	0.003	P(101)	0.086
	C(114)	-0.002	H(114)	0.08
	C(115)	0.001	H(115)	0.00
	C(116)	0.001	H(116)	0.16
	C(117)	0.000	H(117)	-0.18
	C(118)	-0.002	H(118)	-0.21
p)	C(119)	0.001	P(101)	0.037
	C(120)	-0.002	H(120)	-0.02
	C(121)	0.002	H(121)	-0.11
	C(122)	0.000	H(122)	-0.17
	C(123)	-0.002	H(123)	-0.07
	C(124)	0.002	H(124)	0.15
q)	C(125)	0.000	P(101)	-0.358
	C(126)	-0.004	H(126)	0.04
	C(127)	0.006	H(127)	-0.32
	C(128)	-0.005	H(128)	0.03
	C(129)	0.002	H(129)	0.00
	C(130)	0.001	H(130)	0.01
r)	P(101)	-0.016	Fe(101)	-0.511
	C(133)	0.018		
	C(104,105)†	-0.028		
	C(102,103)†	0.026		

† C(X,Y) is the midpoint of the C(X) - C(Y) bond.

The dihedral angles (°) between selected planes are:

a) - b)	34.7	j) - k)	32.9
f) - g)	103.4	o) - p)	102.1
f) - h)	90.2	o) - q)	64.1
g) - h)	59.6	p) - q)	102.5

TABLE 6

(1-Exo-benzylcyclopenta-2,4-diene)dicarbonyl(triphenylphosphine)iron; intermolecular contacts ($\leqslant 3.60$Å).

O(134)	---	C(3^{I})	3.19	O(134)	---	C(20^{I})	3.54
O(32)	---	O(34^{II})	3.22	O(134)	---	C(21^{I})	3.56
O(34)	---	C(101^{II})	3.24	O(134)	---	C(104^{VI})	3.56
O(34)	---	C(102^{II})	3.27	O(32)	---	C(33^{II})	3.56
O(134)	---	C(4^{I})	3.27	O(132)	---	C(117^{III})	3.57
O(32)	---	C(14^{II})	3.40	O(132)	---	C(4^{II})	3.57
O(132)	---	C(118^{III})	3.43	O(132)	---	C(20^{II})	3.57
O(34)	---	C(127^{II})	3.45	C(7)	---	C(17^{VII})	3.57
C(17)	---	C(8^{IV})	3.45	C(21)	---	C(115^{II})	3.58
O(32)	---	C(28^{V})	3.52	O(34)	---	C(126^{II})	3.59
O(32)	---	C(15^{II})	3.52				

The superscripts refer to the following transformations of the atomic coordinates:

I	$1 + x$,	y,	z;	V	$1 - x$,	$- y$,	$1 - z$;
II	$1 - x$,	$1 - y$,	$1 - z$;	VI	$2 - x$,	$- y$,	$1 - z$;
III	$2 - x$,	$1 - y$,	$1 - z$;	VII	x,	$\frac{1}{2} - y$,	$\frac{1}{2} + z$.
IV	x,	$\frac{1}{2} - y$,	$-\frac{1}{2} + z$;				

III.3.3 EXPERIMENTAL

Crystal Data Dicarbonyl(π-cyclopentadienyl)(triphenylphosphine)iron 1,1,2,3,3-pentacyanopropenide; $[C_{25}H_{20}O_2PFe]^+[C_8N_5]^-$.

Crystal system	Triclinic
Unit cell dimensions	$a = 11.812(3)$ Å
	$b = 15.913(5)$ Å
	$c = 8.873(2)$ Å
	$\alpha = 95^\circ 5'(1)$
	$\beta = 104^\circ 57'(1)$
	$\gamma = 110^\circ 24'(1)$
Space group	$P\bar{1}$ (C_i^1)

$U = 1480.7$ Å^3

$M = 605.2$ a.m.u.

$D_o = 1.345$ gm.cm.$^{-3}$ (flotation in aqueous potassium iodide solution)

$D_c = 1.357$ gm.cm.$^{-3}$

$Z = 2$

$F(000) = 620$

μ(Mo-*K*α) = 6.84 cm.$^{-1}$

Crystallographic Measurements

Preliminary triclinic cell dimensions were determined from precession photographs of a yellow crystal with dimensions ca. 0.02 x 0.20 x 1.40 mm^3.

The crystal was transfered to a Hilger and Watts' Y290 automatic diffractometer and offset on χ by about 5° to prevent multiple reflections$^{(2)}$. The intensity data were collected in two parts; firstly those reflections in octants $hk\ell$, $hk\bar{\ell}$, $h\bar{k}\ell$ and $h\bar{k}\bar{\ell}$ with $\theta(\text{Mo-}\underline{K}\alpha) \leqslant 19^\circ$ with the diffractometer controlled by a PDP-8 computer, and then, several months later, with $19^\circ \leqslant \theta(\text{Mo-}\underline{K}\alpha) \leqslant 27^\circ$ using a DF32 DEC disc file backed PDP-8 computer. Recollection of about 50 observations revealed that no serious crystal decomposition or counting chain variations occurred between the two collections. In both collections the cell dimensions and orientation matrix were obtained from a least-squares treatment of the θ, χ and ϕ setting angles of twelve reflections, measured with Mo-$\underline{K}\alpha$ radiation produced by a fully stabilized X-ray generator, operated at 46 kV and 16 mA, and a graphite crystal monochromator.

The intensity data were collected by the $\theta - 2\theta$ scan procedure, each reflection being scanned in 26 equal steps from $2\theta_{\text{calc.}} -0.52^\circ$ to $2\theta_{\text{calc.}} +0.52^\circ$. With $\theta(\text{Mo-}\underline{K}\alpha) \leqslant 15^\circ$, each step was counted for 4 seconds and stationary-crystal, stationary-counter background counts were measured at each end of the scan range for 26 seconds, while with $15^\circ \leqslant \theta(\text{Mo-}\underline{K}\alpha) \leqslant 27^\circ$ the corresponding times were 6 and 39 seconds and only those reflections were measured, which a quick scan had shown to be significantly above background. The intensities of two standard reflections were monitored every 40 intensity measurements and the results used to ensure that all the observations were on a common scale. The scan counts (P) and the background counts (B_1, B_2) of each reflection were combined to yield the integrated intensity, $I = P - 2(B_1 + B_2)$, which was corrected for Lorentz-polarisation effects but not for absorption. 3,567 independent values of $|F_o|$ were obtained, of which the 3,156 observations with $I/\sigma_I > 3.0$ were considered to be observed and were used in the final refinement.

Structure Analysis

Initially, the structure analysis was carried out using only the 2,114 independent values of $|F_o|$ obtained in the first part of the data collection, i.e. reflections with θ(Mo-Kα) $\leqslant$ 19°.

Coordinates for the iron and phosphorus atoms were deduced from the three-dimensional Patterson synthesis and used to phase an electron density distribution (R = 51%). From this and subsequent electron density syntheses peaks were found consistent with the dicarbonyl(π-cyclopentadienyl)-(triphenylphosphine)iron cation, but a group of peaks (see Figure 3) could not be sensibly interpreted in terms of an ordered or a disordered anion.

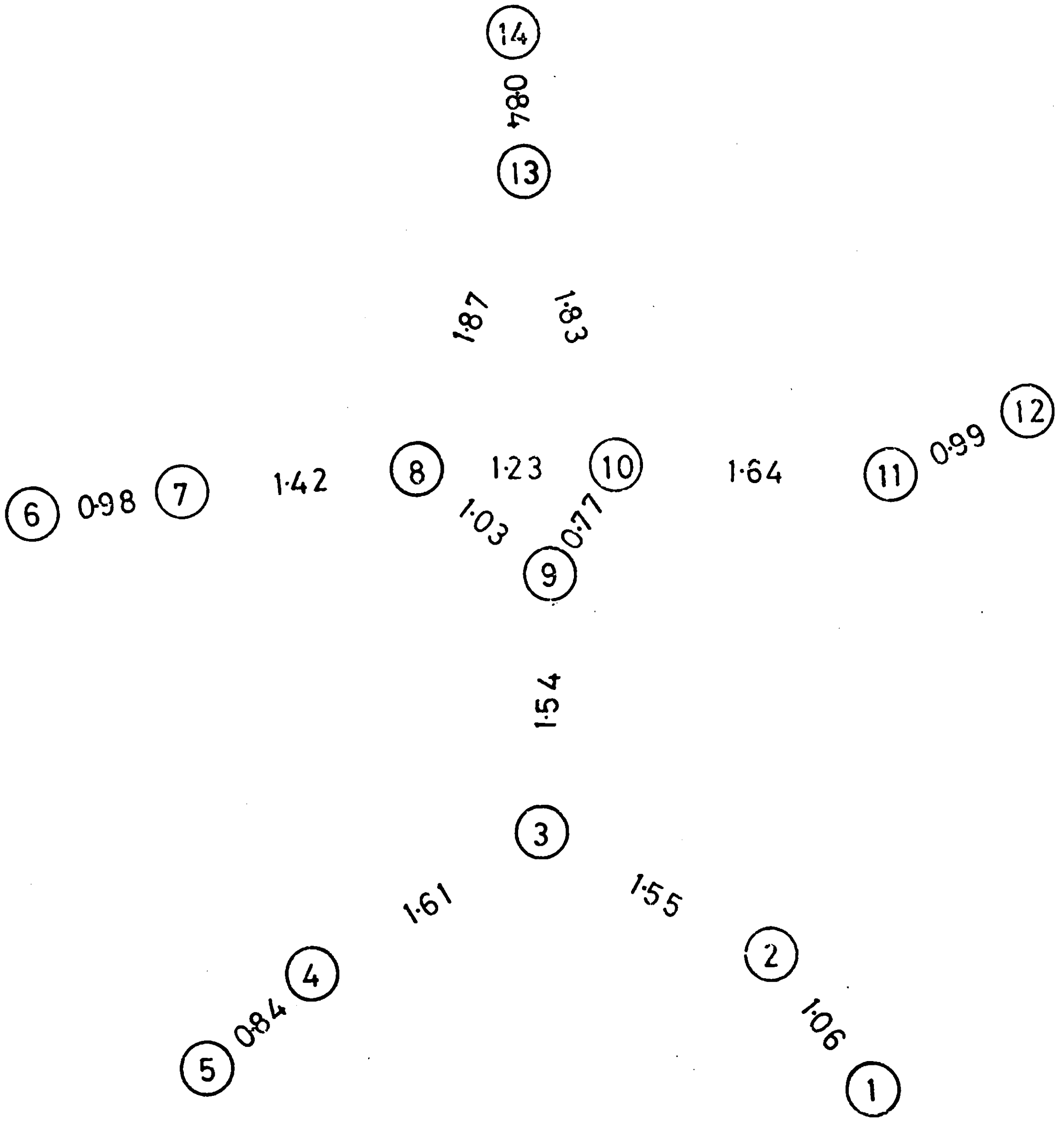

FIGURE 3 'Anion' peaks illustrating the separations $\leqslant$1.90 Å.

These maps, despite the unexplained 'anion' peaks, confirmed that the space group is $P\bar{1}$ and not the alternative P1.

All the 'anion' peaks, together with those of the cation, were included in the initial least-squares refinement, the five peripheral peaks being assigned the scattering curve of nitrogen and the others that of carbon. The thermal vibration of all the atoms was described by isotropic temperature factors during four cycles of full-matrix least-squares refinement [Convergence was reached at R = 12.9%.], which resulted in a similar uninterpretable geometry for the 'anion' peaks and a set of thermal parameters for these atoms, none of which was exceptionally high. U_{iso} of peaks 1, 8, 9 and 10 ranged from 0.14 - 0.16Å^2, compared with averages of 0.10 and 0.07Å^2 in the rest of the 'anion' and the cation respectively.

Using the complete set of observed data, the 'anion' peaks were omitted from three cycles of block diagonal least-squares refinement of the positional and anisotropic thermal parameters of the cation, convergence being reached at 21.1%. At this stage a difference electron density synthesis was calculated and the residual electron density in the region of the 'anion' contoured on to glass sheets, revealing peaks in essentially the same positions as previously noted. Peaks 8 and 10 appeared to be half-weight peaks and, although this was consistent with the observed density which indicated that 2 - 4 of the peaks should be half-weighted, no model involving disorder of an anion, in agreement with this, could at this stage be proposed.

The 'anion' peaks, all with a population parameter of 1.0, were then included stepwise in a series of least-squares refinements (see Table 7), and at each stage the positions of the omitted peaks obtained from a difference electron density synthesis.

TABLE 7

Progress of the least-squares refinement, on the stepwise addition of the 'anion' peaks

Step	Atoms included in the refinement	Treatment of parameters	Final R-factor
1)	All the atoms of the cation plus peaks 1, 2, 3, 4 and 5.	3 cycles; block diagonal; refinement of overall scale factor; x, y, z, U_{ij} for all the atoms.	17.6%
2)	As step 1, plus peaks 6, 12 and 14.	As step 1.	13.1%
3)	As step 2, plus peaks 7, 11 and 13.	As step 1.	9.9%
4)	As step 3, plus H of cation in calculated positions.	As step 1, but with 'fixed' H.	9.5%
5)	As step 4, plus remaining peaks i.e. 8, 9 and 10.	2 cycles; 1 block for anion, 3 blocks for cation; refinement as step 4.	5.9%

Each of the above additions of the 'anion' peaks resulted in a significant(6) lowering of R and R'.

From an examination of a plot(7) of the 50% probability thermal ellipsoids of the 'anion' peaks, a model for this ion explaining the smaller peak heights of 8 and 10 was derived, which consisted of the 1,1,2,3,3-pentacyanopropenide anion disordered as shown in Figure 4.

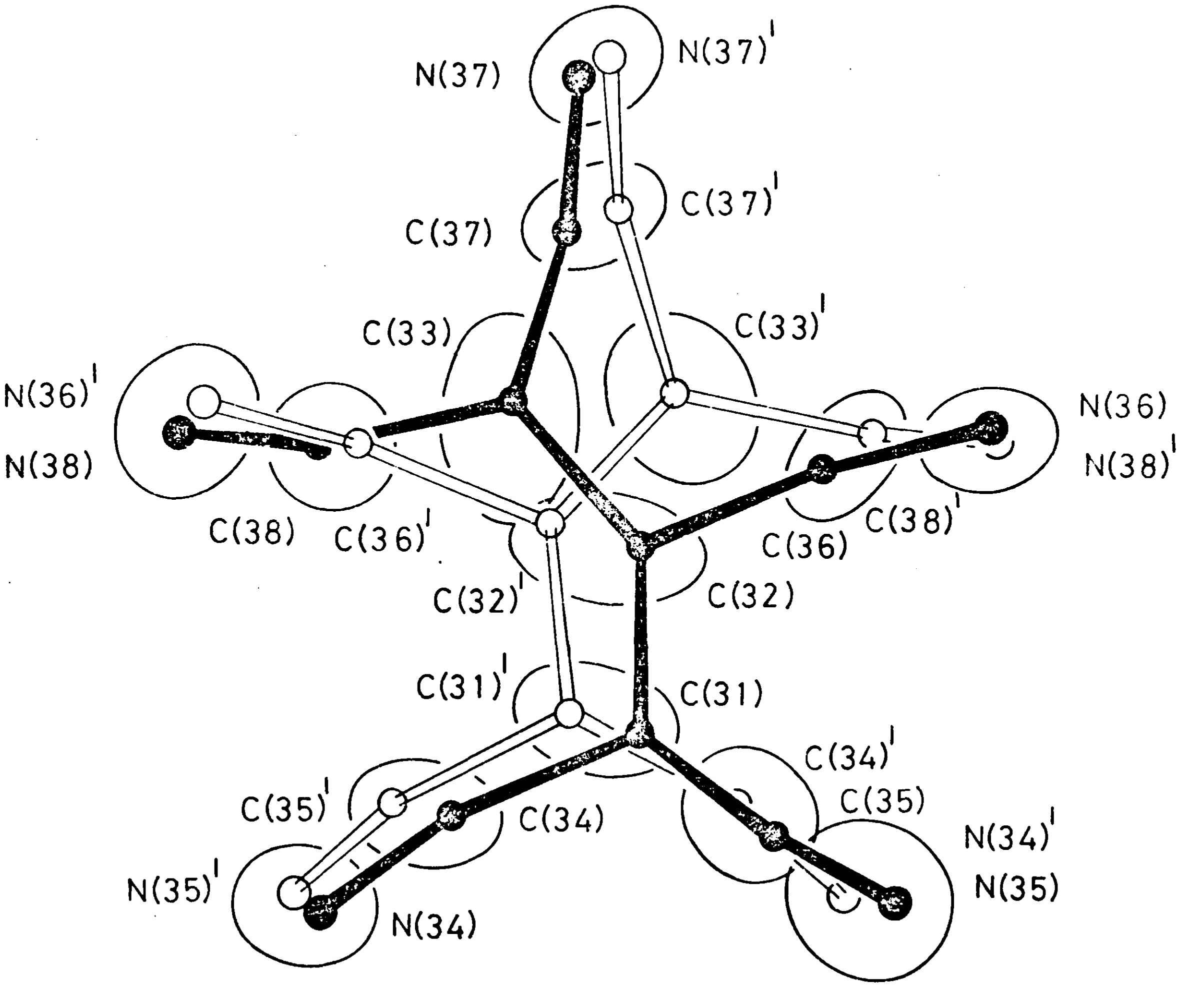

FIGURE 4

The 50% thermal ellipsoids of the 'anion' peaks before the model for disorder was employed, and the two orientations of the 1,1,2,3,3-pentacyanopropenide anion after refinement.

The probable geometry of this anion was estimated from an examination of the geometry in the similar ion, 2-cyanomethyl-1,1,3,3-tetracyanopropenide(8), and in other organic cyanides(9 - 13), and coordinates for both orientations of the anion obtained by matching two models of the anion with the observed peaks, in order to best explain the direction and extent of the thermal vibrations.

The similarity of the peak heights of 8 and 10 suggested that both orientations were equally populated, therefore each atom of the two orientations was assigned a population parameter of 0.5, which was held 'fixed' during the subsequent least-squares refinement. Both the hydrogen and non-hydrogen atoms of the cation were included in the structure factor calculations during four cycles of full-matrix refinement of the positional and isotropic thermal parameters of the anion, which reached convergence at R = 5.6%. The positional and anisotropic thermal parameters of the non-hydrogen atoms of the cation were then included in the parameters refined, and after two cycles of least-squares refinement, with 1 and 4 blocks for the anion and cation respectively, convergence was reached at R = 5.5%. Omitting the hydrogen atoms from the structure factor calculation, a difference electron density distribution was calculated revealing positions for these atoms at an average peak height of $0.38e^-/\text{Å}^3$. The hydrogen atoms were included, at these positions with each assigned an isotropic temperature factor equal to that of the atom to which it was bonded, in further structure factor calculations, but were not refined during two cycles of refinement [The other parameters were treated as previously.] which reached convergence at R = 5.53%, R' = 7.04%.

A consequence of the above treatment of the disorder was the large number of high correlations in the least-squares refinement between parameters in the different orientations of the anion. There were 35 such correlation coefficients whose modulus was > 0.5; the highest, 0.80, relating the shifts of the isotropic temperature factors of the closest atoms, i.e. C(38) and C(36)' which were 0.32 Å apart.

In the final difference electron density synthesis the residual electron density lay between 0.53 and $-0.31e^-/\text{Å}^3$ in the region of the anion and between 0.84 and $-0.79e^-/\text{Å}^3$ in the region of the cation, the major deviations being around the iron atom.

The satisfactory refinement, resulting in the geometry of the anion being reasonable in view of the disorder, indicated that the anion is indeed 1,1,2,3,3-pentacyanopropenide, and this has been confirmed by Dr. G. R. Knox in an independent chemical synthesis of dicarbonyl-(π-cyclopentadienyl)(triphenylphosphine)iron 1,1,2,3,3-pentacyanopropenide using the previously reported preparations of both the cation(14) and the anion(15).

Notes:

1) The scattering factors employed for Fe^+, P, O, N, C and H were taken from 'International Tables for X-ray Crystallography,' Vol. III(4), as were the dispersion corrections Δf' and Δf" for the iron and phosphorus atoms.

2) The final weights employed in the least-squares refinement, in order to ensure approximate constancy of $\langle w\Delta^2 \rangle$ over various ranges of $|F_o|$ and sinθ, were given by the expression:

$$w = X \times Y$$

where:

if $0.1 \times |F_o| > |F_c|$, then $w = 0.000000001$;

if $\sin\theta > 1.0$, then $X = 1$ else $X = \sin\theta/1.0$;

if $|F_o| < 10.0$, then $Y = 1$ else $Y = 10.0/|F_o|$.

3) The calculations were carried out on Glasgow University's KDF9 computer [The least-squares program due to D. W. J. Cruickshank,

J. G. F. Smith and J. G. Sime was used.], and latterly on the IBM 370/155 computer of the Edinburgh Regional Computer Centre using the 'X-ray System' programs(5).

Results

The observed and final calculated structure factors are listed in Table 8, with the final positional and thermal parameters, including the estimated standard deviations derived from the least-squares refinement, in Table 9. The atomic numbering scheme in the anion has been illustrated previously in Figure 4, while in the cation, that of the non-hydrogen atoms is shown in Figure 5; the cation projected on to the plane of the π-cyclopentadienyl ring. Each hydrogen atom is numbered according to the atom to which it is bonded. The bond lengths, valency angles and mean plane calculations are given in Tables 10, 11 and 12 respectively. The distances $\leqslant 1.25$ Å between atoms of the different orientations of the anion and the interionic contacts $\leqslant 3.60$ Å are listed in Tables 13 and 14 respectively. The crystal packing is shown in Figure 6.

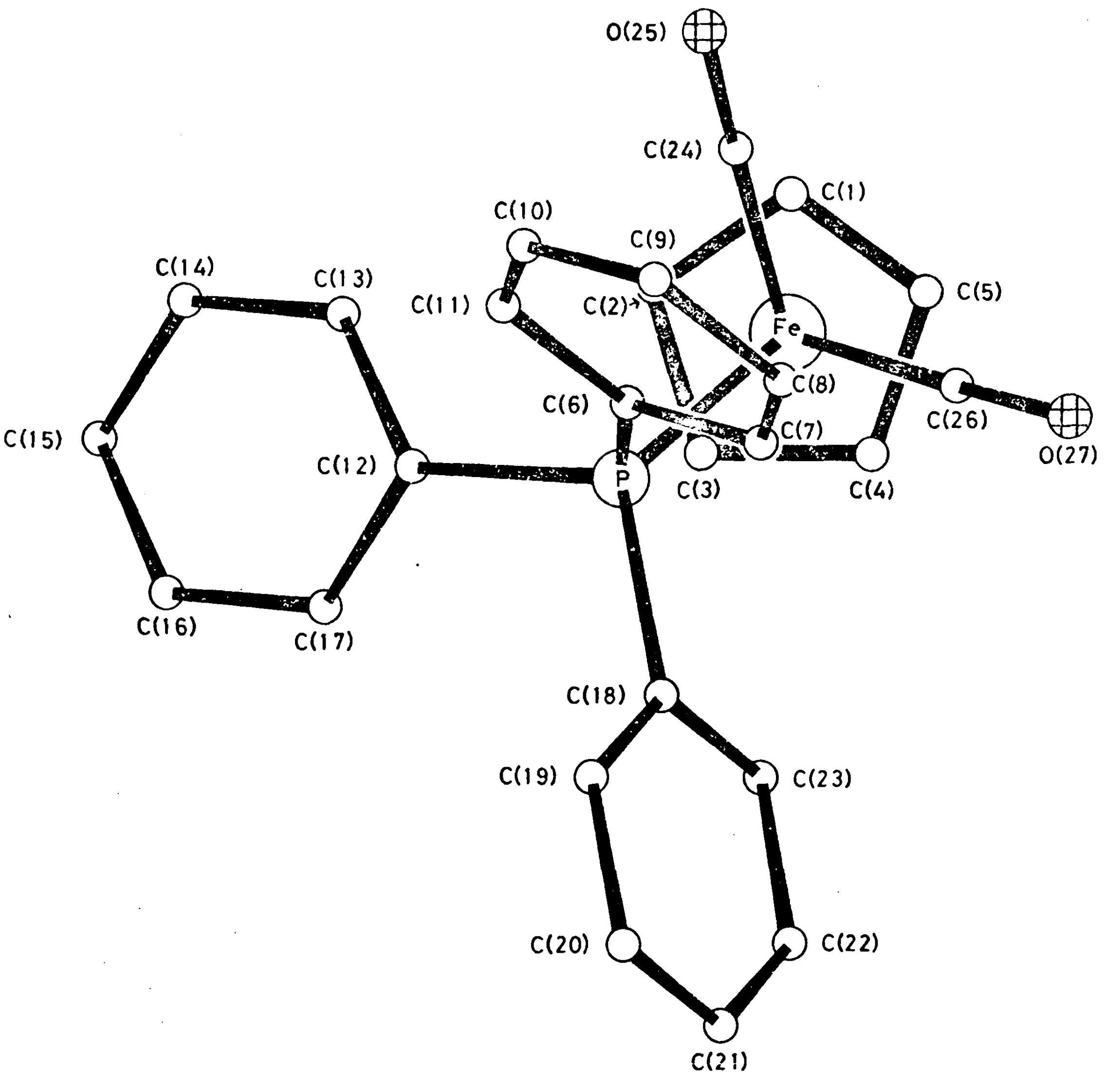

FIGURE 5

The dicarbonyl(π-cyclopentadienyl)(triphenylphosphine)iron cation projected on to the plane of the π-cyclopentadienyl ring.

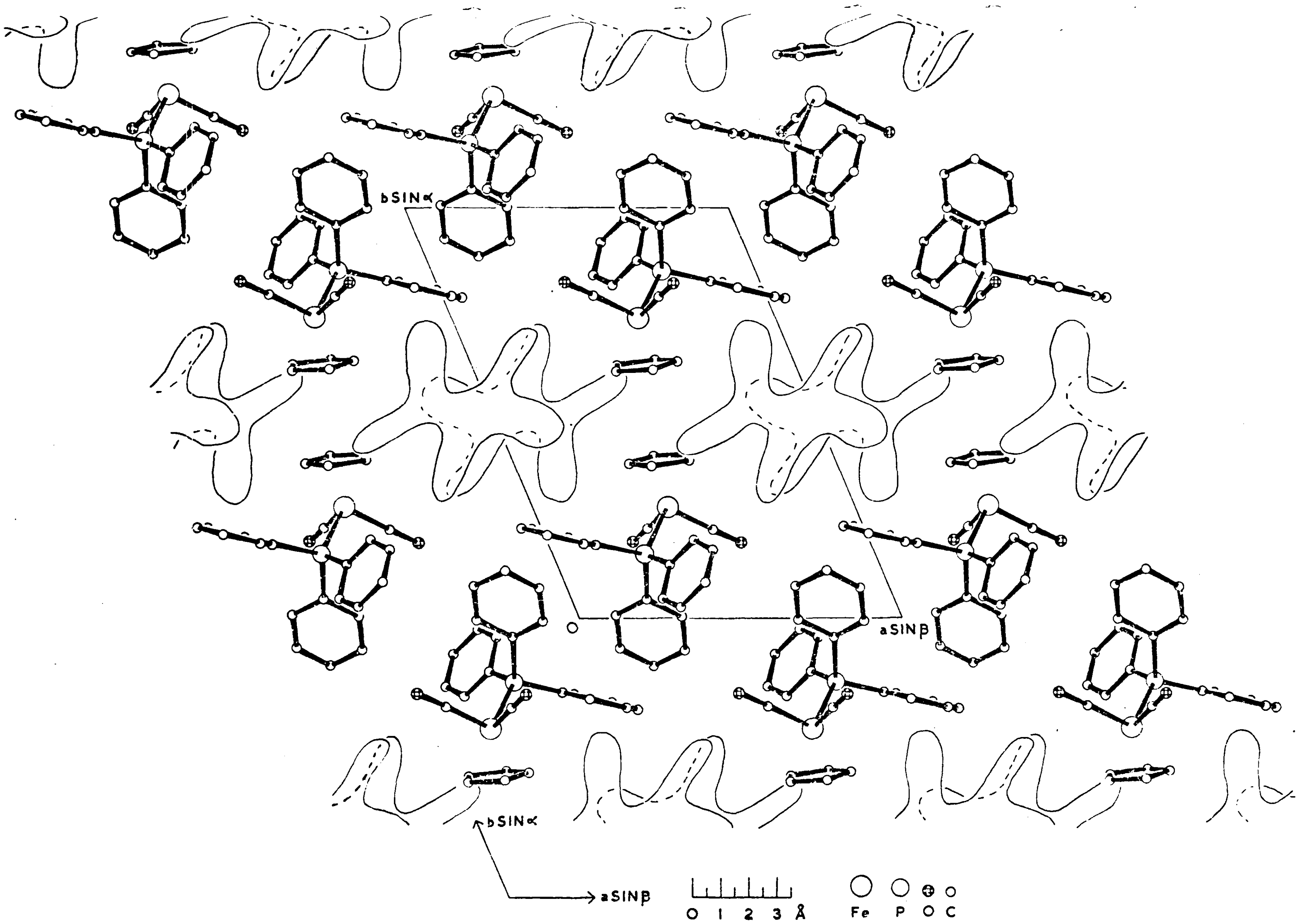

FIGURE 6 Crystal packing of dicarbonyl(π-cyclopentadienyl)(triphenylphosphine)iron 1,1,2,3,3-pentacyanopropenide viewed along the c-axis. For clarity, only the outline of each disordered pair of anions is shown.

TABLE 8

Dicarbonyl(π-cyclopentadienyl)(triphenylphosphine)iron 1,1,2,3,3-pentacyanopropenide; observed and final calculated structure amplitudes (x10).

14,-6,L		
-3	58	57
13,0,L		
-3	57	59
-4	97	95
13,-1,L		
-2	63	67
13,-2,L		
-4	71	78
-5	63	83
13,-3,L		
0	63	71
-6	66	67
13,-5,L		
1	136	146
0	146	150
-1	92	94
13,-6,L		
-1	128	138
-2	126	131
-3	118	123
13,-7,L		
1	152	155
0	135	146
-1	62	83
-3	84	82
-4	106	107
-5	55	48
13,-8,L		
0	128	125
-1	159	157
-2	132	140
-3	121	116
13,-9,L		
-3	119	120
-4	152	148
-5	85	81
13,-10,L		
0	83	88
-1	114	110
-2	85	98
-5	73	67
13,-11,L		
-2	77	71
-3	92	94
-4	96	92
13,-12,L		
-2	57	60
12,3,L		
-2	97	105
-3	70	73
12,2,L		
-2	55	53
-3	111	110
-4	100	100
-5	84	89
12,1,L		
-1	92	97
-2	83	88
-5	67	71
-6	112	108
12,0,L		
-3	98	98
-4	159	154
-5	107	108
-7	53	51
12,-1,L		
1	68	83
-1	86	94
-2	92	96
-4	82	89
-5	115	110
-6	117	114
12,-2,L		
-3	56	58
12,-3,L		
2	79	88
1	123	127
-2	62	65
-5	78	82
-8	73	74
12,-4,L		
0	117	115
-1	122	129
-2	86	88
-4	63	57
12,-5,L		
2	108	109
1	138	144
0	71	91
-2	100	96
-3	119	120
-4	76	82
12,-6,L		
1	67	85
0	113	125
-1	127	110
12,-7,L		
-1	63	54
-2	61	65
-3	155	157
-4	103	108
-5	80	73
12,-8,L		
0	85	85
-1	95	93
-4	97	95
-5	98	94
-6	81	81
12,-9,L		
-2	98	94
-3	135	137
-4	57	56
12,-10,L		
2	103	115
1	98	93
-2	82	74
-4	91	87
-5	103	98
-6	107	102
12,-11,L		
2	78	88
1	113	120
0	62	67
-3	77	63
12,-12,L		
2	102	104
12,-13,L		
1	144	140
0	140	147
-1	142	146
12,-14,L		
-1	73	73
-2	107	96
-3	67	69
11,5,L		
-1	114	110
-2	76	74
-4	67	59
-5	65	72
11,4,L		
-2	135	131
-3	125	131
-4	55	59
11,3,L		
0	97	120
-1	131	134
-4	65	45
-5	101	89
11,2,L		
-2	104	101
-3	124	116
-4	105	95
-6	70	59
-7	68	63
11,1,L		
0	71	66
-1	104	99
-3	73	58
-4	55	64
-5	121	106
-6	88	81
11,0,L		
2	71	76
1	101	91
-2	115	113
-3	129	117
-5	77	81
-6	106	98
-7	82	75
11,-1,L		
3	120	113
2	114	110
-1	122	122
-4	101	94
-5	118	108
11,-2,L		
2	128	124
1	106	107
0	92	93
-4	84	86
-6	72	81
-7	63	67
-8	56	63
11,-3,L		
-1	75	80
-2	115	112
-3	104	99
11,-4,L		
2	124	123
1	215	203
0	288	283
-1	114	113
-3	64	64
-4	130	128
11,-5,L		
0	87	93
-1	118	99
-2	143	132
-3	130	122
11,-6,L		
1	93	84
0	94	78
-1	52	46
-3	69	60
-4	120	110
-5	95	88
-6	67	69
11,-7,L		
-3	121	129
-7	60	63
11,-8,L		
3	82	86
2	131	133
1	65	55
-2	73	73
-3	86	82
-4	104	103
-5	113	108
11,-9,L		
2	133	139
1	74	76
-1	51	39
-3	72	76
-4	55	60
-5	83	84
-6	74	63
11,-10,L		
4	94	105
3	125	128
2	76	85
0	149	132
-1	90	81
-2	93	94
-5	71	56
11,-11,L		
2	165	169
1	190	193
0	155	150
-1	82	88
-2	99	87
-3	83	81
-4	69	64
-6	64	70
11,-12,L		
3	87	94
0	129	127
-1	202	200
-2	172	163
11,-13,L		
2	113	115
1	173	179
0	90	90
-2	74	73
-3	90	85
-4	104	100
-5	78	80
11,-14,L		
1	61	50
0	105	109
-1	169	174
-2	105	98
11,-15,L		
-2	108	107
-3	111	109
11,-16,L		
-1	88	76
10,6,L		
-1	118	112
-2	125	128
10,5,L		
1	96	102
0	131	139
-1	84	84
-3	77	80
10,4,L		
2	53	49
-1	122	110
-2	155	152
-3	143	145
-4	71	77
10,3,L		
0	72	72
-3	98	100
-5	78	71
10,2,L		
-3	113	119
-4	99	102
-6	76	77
10,1,L		
3	94	71
2	77	80
-3	51	53
-4	140	139
-5	157	147
-8	63	66
10,0,L		
2	100	101
1	97	86
-1	61	54
-2	69	61
-6	73	71
10,-1,L		
4	102	106
3	80	73
1	104	103
0	191	188
-1	150	142
-2	64	60
-4	105	100
-5	114	107
-6	79	71
-8	67	68
-9	64	80
10,-2,L		
2	174	168
1	206	190
0	154	140
-2	55	59
-3	147	142
-4	166	156
10,-3,L		
1	60	57
0	164	159
-1	327	321
-2	297	283
-3	64	61
-4	187	189
-5	165	160
-6	108	103
-9	61	70
10,-4,L		
2	119	122
1	211	203
0	172	169
-1	73	80
-2	174	177
-3	256	255
-4	200	202
-5	68	69
10,-5,L		
3	91	108
1	98	92
0	172	162
-1	136	134
-2	122	126
-3	93	94
-4	136	138
-5	135	124
-6	122	105
10,-6,L		
0	60	57
-2	159	146
-3	193	197
-4	142	141
10,-7,L		
3	138	139
2	184	171
1	106	100
0	69	63
-1	79	89
-2	79	83
-4	140	140
-5	138	133
-6	142	123
-7	55	51
10,-8,L		
5	75	95
4	119	136
3	87	83
2	118	109
0	48	47
-2	72	77
-3	125	125
-6	127	111
-7	85	83
10,-9,L		
3	105	121
2	191	184
1	134	126
0	75	66
-1	47	40
-2	67	63
-4	60	55
-5	110	107
10,-10,L		
5	72	92
4	107	140
2	76	85
1	133	120
0	142	126
-1	110	99
-2	56	56
-3	72	61
-4	78	67
10,-11,L		
3	99	97
2	153	169
1	129	137
0	57	62
-2	137	134
-4	96	85
10,-12,L		
4	85	100
2	59	59
1	136	128
0	263	258
-1	260	252
-4	78	74
10,-13,L		
3	63	68
2	124	120
1	91	91
-2	104	101
-3	113	112
-4	115	101
10,-14,L		
-1	123	121
-4	91	84
-5	105	91
10,-15,L		
-1	75	74
-2	90	80
-3	77	69
9,8,L		
0	93	91
-1	93	86
-2	64	59
9,7,L		
1	82	79
0	64	65
-3	83	91
-4	95	85
9,6,L		
1	59	47
0	111	110
-1	140	141
-2	146	144
9,5,L		
1	89	101
0	83	82
-2	181	174
-3	191	186
-4	187	192
-5	121	133
9,4,L		
0	75	67
-2	98	93
-3	97	101
-5	167	170
-6	181	186
9,3,L		
3	87	90
2	97	94
-2	56	59
-3	137	130
-4	207	201
-5	129	132
-8	81	76
9,2,L		
3	64	67
2	150	144
1	132	122
-1	96	88
-4	59	63
-5	173	174
-6	155	160
-7	76	82
9,1,L		
4	142	145
3	140	130
2	111	98
1	76	72
0	135	132
-1	121	112
-2	44	33
-4	72	76
-6	57	71
-7	121	96
-8	98	85
-9	72	70
9,0,L		
3	144	141
2	255	250
1	201	191
-1	166	166
-2	88	87
-4	112	110
-5	228	219
-6	113	107
-7	88	84
-9	56	49
9,-1,L		
4	98	96
2	95	82
1	231	225
0	222	218
-1	225	227
-2	144	152
-4	94	89
-9	57	42
9,-2,L		
3	154	159
2	288	274
1	248	244
-1	158	191
-2	280	275
-3	242	248
-4	82	88
-5	69	71
-6	118	186
-7	58	48
9,-3,L		
2	135	129
1	252	233
0	481	464
-1	223	224
-2	121	119
-3	104	101
-4	171	172
9,-4,L		
3	83	75
2	337	326
1	230	219
-1	245	248
-2	310	314
-3	307	311
-4	69	71
-7	62	59
9,-5,L		
4	75	85
3	156	158
2	72	76
1	119	120
0	153	150
-3	140	142
-4	144	144
-5	79	82
-6	60	55
9,-6,L		
6	76	113
5	99	116
3	99	132
2	64	60
1	71	65
-1	102	95
-2	206	210
-3	216	229
-4	85	86
-5	76	87
-6	65	75
-7	124	126
-8	124	120
9,-7,L		
4	60	70
3	147	144
0	66	81
-1	129	126
-3	79	87
-4	139	141
-5	74	75
9,-8,L		
5	92	95
4	105	134
3	97	112
2	206	191
1	113	99
-2	125	114
-3	68	64
-4	89	95
-5	55	52
-7	51	45
9,-9,L		
4	138	142
3	60	59
1	61	63
0	48	49
-1	87	75
-2	75	68
-8	50	56
9,-10,L		
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-3	79	78
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9,-12,L		
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9,-13,L		
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-2	107	112
-3	92	86
9,-14,L		
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2	66	66
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-4	141	135
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9,-15,L		
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1	121	126
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-1	118	117
-2	83	86
9,-16,L		
3	122	128
9,-17,L		
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1	149	150
-2	83	84
8,10,L		
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8,9,L		
-2	72	53
8,8,L		
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-5	99	94
8,7,L		
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-2	280	186
-3	141	132
8,6,L		
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-1	81	80
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-6	101	97
8,5,L		
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-1	121	123
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-3	128	132
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-6	93	98
-7	81	70
8,4,L		
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-3	121	118
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-6	134	122
-7	142	131
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8,2,L		
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8,1,L		
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2	124	108
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-5	261	259
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-2	313	310
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-6	224	213
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8,-7,L		
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-5	205	210
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-3	98	110
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8,-13,L		
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0	47	37

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-6 77 78

8,-14,L
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4 74 76
2 73 66
1 64 56
-2 64 68
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8,-15,L
4 66 72
3 134 131
2 191 186
1 105 101

8,-16,L
4 112 117
2 69 80
1 100 97
0 131 126

8,-17,L
1 97 91

8,-18,L
1 141 143
-1 82 81

7,11,L
0 88 82
-1 103 98

7,10,L
1 103 100
0 69 67

7,9,L
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-1 177 177
-2 131 121
-5 70 71
-6 51 42

7,8,L
2 63 63
1 164 166
0 68 70
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-2 93 110
-3 151 153
-4 134 120
-6 106 103

7,7,L
0 101 94
-1 177 168
-2 113 124
-5 94 97
-6 94 87
-7 67 53

7,6,L
-1 71 72
-2 214 202
-3 162 171
-4 164 163
-5 111 115

7,5,L
5 87 81
3 97 89
2 109 93
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-1 91 86
-2 147 140
-3 154 155
-5 156 160
-6 112 108
-7 110 103
-8 92 85

7,4,L
5 127 138
4 133 130
3 138 148
2 105 109
0 77 81
-1 78 87
-3 76 76
-4 144 155
-6 84 67
-7 119 110
-8 68 90

7,3,L
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2 241 231
1 164 163
0 89 102
-3 56 66
-5 84 88
-6 106 114
-7 100 100

7,2,L
5 113 114
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3 101 143
1 192 193
0 250 269
-1 236 232
-2 208 221

-3 120 109
-4 156 159
-5 94 88
-7 67 74
-8 125 123
-9 104 96

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2 225 228
1 197 191
0 134 137
-2 145 151
-3 314 329
-4 73 69
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7,0,L
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1 179 169
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7,-2,L
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3 86 86
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-5 213 236
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7,-3,L
6 83 88
4 330 307
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-1 153 142
-2 305 309
-3 312 339
-4 98 101
-5 59 60
-6 124 125
-7 109 108

7,-4,L
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4 219 213
3 286 278
2 259 266
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-2 142 142
-3 102 105
-4 182 242
-5 270 282
-7 109 107
-9 51 47

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-3 58 67
-4 201 215
-6 106 111
-7 108 104
-8 120 110

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-6 182 184
-7 86 82

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-6 67 59
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-2 205 215
-3 285 286

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-2 211 208
-3 208 209
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-4 63 67

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-2 119 110
-3 59 62

7,-18,L
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-8 66 72

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-6 61 62

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-6 108 107
-7 147 147
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-7 81 75
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-6 99 107
-7 79 72
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-8 78 75

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-9 81 84

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-6 141 143

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-4 212 201
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-6 129 130

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3 173 157
2 84 82
0 66 72
-1 93 108
-2 187 198
-3 135 131
-4 198 187
-5 123 130

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3 126 124
2 87 74
1 131 129
0 148 145
-4 82 84
-5 192 199
-6 131 133

6,-13,L
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5 176 168
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3 75 88
2 66 59
0 81 77
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-3 172 175
-4 56 87

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-2 55 60
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-1 86 99

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-3 224 240
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-8 58 62

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-9 64 59

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-3 347 364
-4 237 238
-5 170 188

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-8 83 85

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-4 241 238
-5 240 266
-6 200 202
-7 201 194

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4 440 384
2 230 212
1 308 287
0 215 205
-1 403 465
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-4 100 107
-5 126 134
-6 151 152
-7 133 141
-8 150 142
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-8 128 127
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5 117 111
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3 162 166
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-2 422 430
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-5 188 183

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5 67 65
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1 230 239
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-2 334 320
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-4 219 217
-5 163 147
-6 156 150

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7 113 115
6 83 70
5 114 118
4 81 83
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1 101 106
0 404 399
-1 444 452
-2 76 75
-3 69 73
-4 183 190
-5 113 110
-6 204 185

5,-9,L
6 169 175
5 219 224
4 175 153
2 80 88
1 310 303
-1 268 270
-2 226 235
-3 206 202
-4 49 51
-6 76 72

5,-10,L
8 76 74
7 171 181
6 78 75
5 127 124
4 175 180
3 240 220
2 220 222
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5,-10,L

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 0  262  266
-1   92  102
-2  121  110
-3  155  160
-4  131  133
-5  173  175

5,-11,L

 7  132  143
 6  151  151
 5  140  139
 4  146  144
 3   71   66
 2   56   61
 1  167  164
-1   62   62
-2   61   71
-3  121  122
-5   71   79
-6   88   86
-7   88   90

5,-12,L

 5  133  127
 4  108  113
 3  269  253
 2  153  156
 1   63   71
 0   74   69
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-2  103  112
-4   82   62
-5  121  114
-6   61   64

5,-13,L

 6  124  127
 5  137  129
 3  150  174
 2  239  234
 1  248  246
 0  283  285
-2   49   51
-3   57   54

5,-14,L

 5   89   97
 4  158  159
 3   86   70
 2  199  200
 1   47   54
 0  190  192
-1  112  112
-3   81   74

5,-15,L

 3  106   98
 2  250  254
 1  215  214
 0   87   85

5,-16,L

-1  121  125
-2  123  114
-3   94   86
-4   52   61

5,-17,L

 1   74   76
 0   55   50
-3  104   95

5,-18,L

 1   50   46
 0   53   50
-2   63   66

4,14,L

 0   80   65

4,13,L

 1   96   90
-3  100   89
-4   87   78

4,12,L

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 0  103  102
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-6   90   88

4,11,L

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 0  132  134
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-3  159  155
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-8   56   46

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 0  193  199
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-5  145  141
-6  122  115
-7   85   84

4,9,L

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 4  100  108

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-9   64   65

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 1  115  147
 0  121  129
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-7  127  120

4,7,L

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 4  111  122
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-3  262  266
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-7  150  156
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-9   74   71

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 4   55   61
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-5  231  231
-6  127  121
-9   96   95

4,5,L

 6   89   83
 4  138  132
 3   55   50
 2  181  188
 1  294  311
 0  432  460
-1  139  149
-2  183  195
-3  236  247
-4  239  240
-5   75   73
-7  115  121
-8   84   79
-9   87   57

4,4,L

 6   88   75
 5   72   75
 4  188  183
 3  333  326
 2  124  124
 0  191  184
-1  194  218
-2  365  399
-3  181  130
-4   90   83
-6  118  115
-9   50   41

4,3,L

 7   85   74
 5   51   43
 2  199  210
 1  247  228
 0  382  388
-1  185  209
-2  134  136
-4  193  207
-5  241  264
-6   92   99
-8   87   74

4,2,L

 7   80   79
 6  187  110
 5  154  159
 4   55   49
 3  207  199
 2  271  282
 1  112  109
 0  170  170
-1  194  194
-2  557  590
-3  472  512
-4   55   60
-5   49   44
-6   96   97
-7  131  120

4,1,L

 7  120  110
 4  186  181
 3  243  239
 1  149  147
 0  252  262
-1  169  166
-2  250  206
-3  178  195
-5  179  197
-6   90   97
-7   53   56

4,0,L

 7   39   63
 5  367  343
 4  187  184
 3   68   56
 2  146  297
 1  100   95
 0  359  338
-1  111   99
-2   55   78
-3   75  106
-4  138  162
-5   73   84
-6   76   87
-7  104   99
-8  138  144

4,-1,L

 8   86   95
 7   81   72
 5  142  139
 4  254  233
 3  191  180
 2  357  316
 0  389  342
-1  333  324
-2  113  113
-6  143  150
-8   92   94

4,-2,L

 7  148  144
 6   71   63
 5   65   64
 4  236  210
 3  186  168
 2  322  271
 1  587  529
 0   56   76
-1  298  290
-2  141  158
-3  175  201
-4  178  186
-5   64   81
-6  270  275
-7  165  157

4,-3,L

 6   74   73
 4  296  290
 3  321  301
 2  306  235
 1  360  311
 0   86   57
-1 1090 1101
-2   92   83
-3  143  150
-4  270  280
-5  102  126
-8  104  104
-10  57   57

4,-4,L

 5   84   84
 4   48   39
 3  191  160
 2  534  465
 1  641  571
 0  381  345
-1   56   32
-2   53   47
-3  101  114
-4  537  556
-5  132  131
-7  102   99

4,-5,L

 7   68   76
 6   70   75
 5   98   86
 4  173  172
 2  369  326
 1  212  195
 0  714  676
-1  789  784
-2  462  490
-3  104  108
-4  355  373
-5  165  163
-6  149  153
-8   84   73

4,-6,L

 8   93  101
 6   53   60
 5  123  116
 4  107  100
 2  574  513
 1  177  153
 0  333  362
-1  223  215
-2  307  305
-3  290  309
-4  141  146
-5  160  167
-6  138  146

4,-7,L

 7  100  107
 6  245  244
 5  137  141
 4   55   30
 3   82   91
 1  147  143
 0  184  170
-1  339  361
-2  207  225
-3  249  227
-4   69   71
-5   70   70
-6  109  117
-7  122  104
-8   82   86

4,-8,L

 7  102  100
 5  223  217
 4  228  223
 3  180  163
 2  188  371
 1  171  158
 0  213  207
-1   96   97
-2  320  341
-3  268  285
-4  303  300
-5  177  179

4,-9,L

 7  148  142
 6  157  164
 5  180  177
 4   94  100
 3  196  183
 2  121  114
 1  188  185
-2  277  284
-3   43   35
-4   71   74
-5   93  105
-6  156  156
-7   69   66
-8  107  105

4,-10,L

 6  174  172
 5  174  178
 4  306  289
 3  220  209
 2  119  125
 0  255  256
-1   81   68
-2   55   58
-4   63   60
-5   94   96
-8   73   68

4,-11,L

 7   76   78
 6  104  102
 5   67   64
 4  152  151
 3  109  112
 2  196  200
 1  233  250
 0  150  153
-1  249  248
-3   83   79
-5   91   85
-6   84   83
-7   81   68

4,-12,L

 6   90   90
 5  148  147
 4  139  130
 3  135  145
 2  153  148
 1  113  117
 0  151  156
-1  327  339
-2  120  123
-4   80   69
-5   65   65

4,-13,L

 3  263  258
 2  197  201
 1  350  343
 0   78   78
-2   68   75
-3   83   79
-4   50   50

4,-14,L

 5   92   92
 4   79   77
 2   72   80
 1  238  232
 0  150  159
-1  144  142
-2  142  153

4,-15,L

 2  171  174
 0   55  [illegible]
-2  127  124
-3  167  166
-4  110  112

4,-16,L

 0  174  164
-1  130  119
-4   51   50

4,-17,L

 5   80   63
 4   55   46
 3   92   94
-2   95  107
-3  104  107

4,-18,L

 3  109  109
-1   62   66

3,15,L

-1   59   63
-2   81   70

3,14,L

 1  119  114
 0  164  150
-1   96  105
-4   51   50

3,13,L

-1  110  100
-2  141  139
-3  139  130
-4   78   74

3,12,L

 1   70   77
 0  181  173
-1  172  163
-4  156  160
-5  149  130

3,11,L

 2   66   67
-1  124  120
-2  183  189
-3  161  153
-4   71   69

3,10,L

 4   83   85
 3  195  196
 1   51   63
 0   64   57
-1  184  187
-2   63   63
-4  197  205
-5  133  129
-6   92   91

3,9,L

 6   85   93
 5  130  128
 4  101   99
 2  165  161
 1  128  136
-1   75   74
-4   49   49
-5  111  112

3,8,L

 5  109  115
 4  164  150
 3  146  143
 2  224  222
 1   89   92
-2  146  158
-3   65   63
-4   54   59
-6  153  151
-7   99  100
-8   74   77
-9   74   67

3,7,L

 6   86   96
 4   52   49
 2  208  216
 1  324  329
 0  166  176
-3  239  223
-4   65   77
-6  246  241
-7  109   99

3,6,L

 4  189  198
 3  292  290
 1   82   80
 0  298  302
-1  357  374
-2  286  309
-3   81  107
-4  158  173
-5  130  142
-6   70   78
-9   70   81

3,5,L

 5   76   67
 4   99   94
 3  294  295
 2  317  324
 1  462  486
 0  133  130
-2  170  193
-3  437  462
-4  174  185
-5  261  266
-6   73   70
-7  123  131
-8   86   88

3,4,L

 4   45   42
 3  154  157
 1  166  171
 0  203  227
-1  290  308
-2  784  824
-3  324  330
-4  247  253
-5  173  169
-6  264  272
-9   95   87

3,3,L

 6   83   79
 5   65   70
 4  173  170
 3  159  160
 1  348  355
 0  198  170
-1   57   48
-2  132  126
-3  494  542
-4  612  642
-5  223  230
-7   80   85
-8  112  120

3,2,L

 8   94   85
 7   56   47
 6  139  137
 5  231  230
 3   93  123
 2  292  285
 0  572  482
-1   53   70
-2  448  455
-3  120  141
-5  219  255
-6  172  172
-7  152  150

3,1,L

 8   85   83
 7   88   82
 6  154  150
 5  221  201
 4  350  319
 3  266  264
 2  174  157
 0   55   61
-1  186  182
-2  644  670
-3  426  457
-4  142  143
-6  273  270
-7   63   63
-8  145  139
-10  50   41

3,0,L

 7  154  144
 5  149  140
 4  133  125
 3  111   98
 2  109  140
 1  570  584
 0   54   61
-2  216  229
-3  116  134
-4   44   51
-5  240  237
-6  330  335
-7  142  143

3,-1,L

 5  365  318
 4  227  229
 3  613  512
 2  456  334
 1   48   42
 0  844  778
-1  716  717
-2  297  321
-4  266  285
-5   71   72
-7  164  168
-8  172  123
-9   87   83

3,-2,L

 6   56   44
 4   47   51
 3  220  230
 2  480  422
 1  543  438
 0  453  387
-1  174  192
-2  420  416
-3  117  107
-5  197  193
-6  138  145
-7   63   69

3,-3,L

 6   68   71
 5  294  270
 4  293  264
 3  151  121
 2  234  203
 1  494  481
 0  841  882
-1 1094 1085
-2  127  123
-3  276  284
-4   46   67
-5  120  125
-6   67   62
-7   88   82
-8   82   85

3,-4,L

 9   99   93
 8  183  170
 6  103  105
 4  145   94
 3  464  395
 2  590  587
 1  155  143
 0  931  897
-1  718  734
-2  129  147
-3  319  368
-4  146  134
-5  213  280

3,-5,L

 7  135  124
 6  163  144
 4  236  201
 3  226  218
 1  396  346
 0  270  262
-1  182  231
-2  810  553
-3  288  282
-4  452  470
-5  158  170
-6  100  121
-9   71   75

3,-6,L

 6  130  120
 5  184  182
 3  236  220
 2  352  378
 1  200  276
 0  394  370
-1  169  166
-2  420  436
-3  360  400
-4  314  334
-7   64   67

3,-7,L

 8  120   97
 7   70   74
 6  150  126
 4  313  284
 3  366  355
 2  137   95
 1   71   76
 0  231  199
-1  237  229
-2  147  147
-3   73   78
-5  137  117
-8  135  130
-9   53   55

3,-8,L

 6  227  228
 5  211  282
 4  263  240
 3  126  127
 2   76   74
 1  488  485
-1  155  155
-2  124  135
-3  134  144
-4  182  178
-6  136  151
-7  105   96
-8   89   88

3,-9,L

 8   65   62
 7  104  111
 6   74   61
 5   73   60
 4  210  209
 3  341  321
 2  150  192
 1   42   46
 0  186  186
-1  122  126
-2  251  250
-4  172  183
-5  149  156
-7   54   51

3,-10,L

 7   62   66
 6   53   53
 5  210  192
 3  130  136
 2   56   61
 1  235  243
 0  324  342
-1  239  240
-2   88   62
-7   84   83
-8   92   91

3,-11,L

 7   65   63
 5   54   50
 4  180  173
 3  135  141
 2  209  208
 1   94   85
-1  142  151
-2  170  200
-3  123  129
-4   54   67
-5   96   92

3,-12,L

 8   77   73
 5   60   77
 4   86   56
 3   54   53
 2   90  101
 1  292  295
 0  210  220
-1  282  297
-2   90  100
-4   77   75

3,-13,L

 6   61   71
 3  142  137
 2  254  259
 0  122  123
-1  110  109
-2  144  146
-3  122  112

3,-14,L

 7   74   70
 4   76   72
 2  135  132
 1  157  159
 0   50   45
-2   87   95
-3   70   77
-4   82   94
-5   61   75

3,-15,L

 7   62   68
 6   70   70
 5   50   60
 1   62   53
 0   49   47
-1  197  150
-2  138  137
-3  142  140

3,-16,L

 4   77   75
 3  141  149
 2   49   51
 1   62   69
 0   58   63
-3  145  142
-4   95   91

3,-17,L

 4   88   79
 3   94   83
 2  119  113
-1   75   67

3,-18,L

 4  121  115
 1   64   57

2,16,L

 0   67   60

2,15,L

 0  112  112
-1  112  109
-2  105   99

2,14,L

 1   80   76
-2  121  122
-3  147  146
-4  114  108

2,13,L

 1   77   82
 0  103  103
-1  182  143
-2  128  126
-3   70   84
-5  117  116
-6  117  105

2,12,L

-1   73   73
-3  174  179
-4  252  242
-5   91   90

2,11,L

 2  181  178
 1   61   62
 0   55   57
-1   57   54
-3   83   41
-4   70   60
-5  131  126
-6  174  175
-7  133  126

2,10,L

 6   87   87
 5  153  143
 4  130  139
 3  127  125
 1   84  100
 0   65   69
-1   80   51
-2   53   62
-4   52   58
-6   66   66
-7   60   66
-8  101  104

2,9,L

 4   87   42
 3   70   77
 2  144  154
 1  331  331
 0  148  151
-1   93   79
-2   52   45
-3  163  184
-4   60   46
-5  105  102
-6  206  206
-7  122  123

2,8,L

 5   92   94
 4   75   82
 3  196  199
 2   81   81
 1  147  157
 0  288  302
-1  308  326
-2  177  196
-6   68   75
-7  106  107
-8  101   95
-9   84   87

2,7,L

 4   91   90
 3  285  286
 2  308  302
 1  472  492
 0  481  500
-1  136  132
-2   84  100
-3  330  337
-4  131  145
-6   78   77
-7  100  112

2,6,L

 6   50   53
 4  124  121
 3   87   73
 2   52   40
 1  211  231
 0  406  431
-1  510  532
-2  403  420
-3  255  250
-4  123  110
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   2,6,L
-5    71    76
-7    84    57
-8    99    97

   2,5,L
 6    60    60
 4    78    79
 2   210   212
 1   396   420
 0   240   249
-2   438   476
-3   283   276
-4   463   473
-5   243   245
-7    52    56

   2,4,L
 8   111   109
 7   111    92
 6    62    61
 5   143   145
 4    99   101
 3   226   225
 2   149   131
 1   400   396
 0   691   735
-1   593   643
-2   422   419
-3    82    77
-4   446   464
-5   528   508
-6   161   163
-7    84    51
-8   107   102

   2,3,L
 6    95    92
 5   300   286
 4   160   160
 3    64    58
 2   332   339
 1   269   260
 0   343   342
-1    92    99
-2  1134  1225
-3   728   764
-4   159   168
-5   102   105
-6    59    67
-7   141   149
-8    66    49

   2,2,L
 8    93    90
 7   160   156
 6    57    51
 4   430   406
 3   284   276
 2   753   684
 1   485   466
 0   469   443
-1  1161  1221
-2   115   100
-3   343   373
-4   120   128
-5   434   453
-6   100   109
-7    66    66

   2,1,L
 6   213   192
 5   445   415
 4   278   256
 3   107   104
 1   186   104
 0    48    62
-1   552   578
-2   251   266
-3   140   128
-5   229   227
-7   218   217
-8   142   135

   2,0,L
 8    98   100
 7    94    86
 5   141   131
 4   388   358
 3   410   369
 2   316   298
 1   419   379
 0   355   312
-1   356   381
-2   432   439
-3   165   159
-4   323   339
-5   288   287
-6   264   272
-9    81    49
-10   93    93

   2,-1,L
 5   263   247
 4   208   172
 3   131   119
 2   366   289
 1   439   384
 0  1165  1067
-1   928   976
-2  1194  1277
-3   160   156
-4   198   210
-5   211   218
-6   184   144
-7   134   142

   2,-2,L
 6   159   168
 4   335   382
 3  1019   867
 2   236   217
 1   549   470
 0   784   789
-1   184   193

-2   686   745
-3    48    58
-4   165   177
-5    58    41
-10   61    58

   2,-3,L
 5    85    82
 4   233   173
 3   280   144
 2   781   591
 1   328   246
 0    95    64
-1   128   138
-2  1141  1216
-3   168   189
-4    77   120
-5   216   234
-7    62    84

   2,-4,L
 7   128   108
 6   135   134
 4   174   137
 3   156   129
 2   349   335
 1   355   311
 0   397   388
-1   533   546
-2   381   389
-3   563   575
-4   345   323
-6   106   118

   2,-5,L
 9   132   137
 8    95    89
 7    84    73
 6    97    91
 5    73    59
 4   379   364
 3   282   167
 2   293   234
 1   164   145
 0   228   213
-1   330   331
-3   394   479
-4   143   170
-5   216   234
-6    48    51
-9    50    47

   2,-6,L
 7   127   117
 6   297   296
 5   115   105
 4   137   129
 1   515   486
 0   556   553
-1   357   366
-2   431   456
-3   255   275
-4   193   234
-6   164   169
-7    98   120

   2,-7,L
 7   122   113
 6    86    85
 5   145   145
 4   205   198
 3   387   369
 2   169   175
 1   120   131
 0   424   454
-1    68    67
-2    54    74
-3   169   188
-4   240   247
-5   181   169
-6    44    56

   2,-8,L
 6   149   138
 5   174   176
 4   170   170
 2   335   305
 1   257   292
 0   108   118
-1   115   119
-2   187   187
-3   207   205
-4    94    90
-5    76    77
-6   128   112
-7   101   122
-8    97   110

   2,-9,L
 6    60    64
 5   194   194
 4   414   412
 3   237   221
 2   321   336
 1   129   119
 0   292   299
-1   336   356
-2   145   140
-4   118   110
-5    86    79

   2,-10,L
 7    76    72
 6   154   150
 5    49    57
 3    75    79
 2   217   225
 1   258   290
 0   228   230
-1   167   163
-2    66    75
-3    56    62
-7    63    67

   2,-11,L
 3   280   282
 2   158   159
 0   216   240
-1   234   250
-2   221   223
-3    99   107
-4   105   110
-5    89    92

   2,-12,L
 6    85    80
 5   181   190
 4    46    46
 3    53    55
 2   211   211
 1   221   221
 0   145   156
-1    68    59
-2    66    68
-3   102   107
-4    96    86

   2,-13,L
 7    89    97
 6    95    95
 5    62    53
 3    53    55
-1    85    82
-2    47    44
-3    50    60
-5    47    37
-6    54    58

   2,-14,L
 5    54    53
 4   119   114
 2    77    81
-2   110   102
-3   152   153
-4    79    84

   2,-15,L
 7    68    67
 6    75    72
 3    92    82
 2   159   156
 0    49    36
-1    78    75

   2,-16,L
 5    96    92
 4   146   136
 3    92    87
 0    73    61
-2    94    88
-3    89    94

   2,-17,L
 4    89    85
 3   124   127
 2    90    76
 1    67    73
-2    75    71

   2,-18,L
 0    78    71

   1,16,L
-2    95    88
-3    89    93
-4    66    70

   1,15,L
 0    69    58
-1   114   110
-5   106   101
-6    51    50

   1,14,L
-1    86    82
-2   150   150
-3   197   190
-4   152   154
-7    65    73

   1,13,L
 3    83    65
 2   110   111
 0    97    96
-4   144   143
-5   177   173
-6    84    90

   1,12,L
 5   117   104
 4    63    59
 2    57    46
 0    70    75
-2   140   137
-3   184   188
-6    84    82
-7   145   147
-8   114   111

   1,11,L
 4   110   110
 3   159   155
 2   177   183
 1   126   133
 0    97    93
-1    96    99
-2    78    81
-3    73    74
-4   114   108
-5   155   161
-8    81    49
-9    68    75

   1,10,L
 6    73    88
 5   113   110
 4   135   127
 2   140   145
 1   275   273
 0   224   237
-2   166   188
-5    96    89
-6   109   100
-7   123   125
-8   112   100

   1,9,L
 5    73    66
 4    48    48
 3   174   174
 2   111   121
 0   118   113
-1   102   122
-2   251   256
-4    77    72
-6   118   119
-8    98    98
-9    94    97

   1,8,L
 4   129   136
 2   253   265
 1   483   494
 0   528   542
-1   387   400
-2    73    74
-3   265   273
-4    89    94
-5    55    48
-6    95    87
-7    57    62

   1,7,L
 7    81    89
 6    85    86
 5    79    81
 4   112   118
 3   184   190
 2   135   143
 1    55    73
 0   485   511
-1   442   484
-2   366   412
-3   183   208
-4   118   124
-5   140   139
-6    71    80

   1,6,L
 8   101   104
 7   132   131
 4    50    55
 3    64    52
 2   102   114
 1   346   351
 0   221   263
-1   413   427
-2    75    71
-3   168   180
-4   198   194
-5   371   359
-6   211   210
-7    56    65
-8    59    64

   1,5,L
 7   183   189
 6   126   122
 5   121   114
 4    89   101
 3   220   241
 2   103    94
 0   161   175
-1   250   271
-2   558   587
-3   219   220
-4   356   354
-5   123   134
-7   151   149

   1,4,L
 8   118   114
 6   134   136
 5   131   128
 4   228   220
 3   309   313
 2   337   334
 1   279   264
 0   433   449
-1   259   266
-2   295   287
-3   420   469
-4   368   378
-5   312   318
-6   106   105
-7    73    80

   1,3,L
 7   112   111
 6   394   390
 5   303   293
 4 [illegible]  92
 3   337   339
 2   443   425
 1   517   505
 0   132   129
-1  1182  1140
-2   731   774
-3    72   114
-4   218   223
-5    48    58
-6   239   245
-7   257   264
-8   145   147

   1,2,L
 8   140   142
 7    46    47
 6   210   224
 5   319   304
 4   524   511
 3   424   414
 2   120   117
 1   130   121
 0    81    25
-1   108   191
-2   316   330
-3   447   457
-4   322   344
-5   173   189
-6   172   172
-8    55    47
-9    74    72
-10  185   181

   1,1,L
 8   155   168
 7    77    70
 6    86    80
 5   308   287
 4   185   181
 3   212   204
 2  1016   946
 1   159   193
 0   865   837
-2   417   445
-3   590   596
-4   264   268
-5   309   335
-6   278   278
-7   292   320
-8   120   128
-9    83    80
-10   81    81

   1,0,L
 8    84    88
 7    55    62
 6   125   119
 5   247   242
 4   387   386
 3   650   596
 2   177   176
 1    62    29
 0   333   347
-1   845   924
-2   528   556
-4   168   189
-5   118   125
-6   109   106
-7    66    58
-8    99    91
-9   113   115

   1,-1,L
 7    89    87
 6    54    48
 5    79    78
 3    77    68
 2   995   915
 1  1460  1161
 0   480   464
-1   251   263
-2   432   432
-3   614   626
-4   428   445
-5    66    61
-6   168   174
-7   124   114

   1,-2,L
 7    49    46
 6    80    81
 5   140   141
 4   165   152
 3   154   119
 2    98   113
 0  1034  1020
-1   689   709
-2   557   570
-3   214   204
-4   121   119
-5   180   193
-6   180   191
-7   170   173

   1,-3,L
 9   121   113
 8    75    74
 7    52    52
 4    97   117
 3   361   328
 2   149   129
 1   521   472
 0   540   557
-1   423   414
-2   398   402
-3   332   361
-4   512   517
-5   276   275
-8    84    89

   1,-4,L
 7   161   164
 6   122   115
 5    99    96
 4   135   118
 3   397   331
 2   479   437
 1   401   375
-1    77    61
-2   329   337
-3   146   149
-4   125   113
-5   258   259
-6   173   172
-8   100   106

   1,-5,L
 8    86    88
 7   122   120
 6    97    91
 5   180   176
 4   415   377
 3   128   124
 2   499   473
 1   719   241
 0    72    81
-2   462   474
-3   428   423
-4   132   131
-5   132   127
-6    99   103

   1,-6,L
 9    61    59
 8    52    50
 7   155   144
 6   169   165
 5    51    44
 4   133   129
 3   299   286
 2   187   194
 1   329   349
 0   496   534
-1    93    92
-2    63    49
-3   264   280
-5   262   262
-7   133   127

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 8    89    91
 7    49    44
 6    69    81
 5   376   360
 4   395   393
 3   421   428
 2   118   117
 1   377   379
 0    66    82
-1    93    75
-3   215   223
-4   144   145
-6   120   113
-7    84    76
-8    54    62

   1,-8,L
 7   127   116
 6    54    56
 4   328   314
 3   320   320
 2   508   520
 1    84    99
 0   129   123
-1   337   353
-2   478   502
-4   121   121
-5   124   116
-6   102    99
-7    71    80

   1,-9,L
 6   156   152
 5    91    94
 4   293   291
 3   189   189
 2   101   174
 1   483   499
 0   616   646.
-1   449   469
-2   206   224
-3    81    80
-4    75    68

   1,-10,L
 9    71    77
 6   140   142
 4   140   158
 3   260   267
 2   404   405
 1   239   256
 0    78    88
-2    97   106
-3   146   160
-4    51    55

   1,-11,L
 8    88    71
 7    74    69
 5    53    93
 2   102   100
 1   125   138
 0   185   183
-1   263   262
-2   179   175
-4    72    73
-6    50    56

   1,-12,L
 6    78    88
 5   111   116
 4    49    51
 3   158   157
 2   140   135
 1    87    85
-1    61    74
-2   193   199
-3   122    93
-5    57    62

   1,-13,L
 8   107   110
 7   163   183
 6    59    57
 2    98   102
 1    74    62
 0    83    87
-4    87    78
-5    80    80

   1,-14,L
 6    78    78
 5    71    73
 4    61    60
-2   121   118
-3   184   186

   1,-15,L
 7    55    55
 4    97    99
 3   187   184
 2    72    63
-1    92    81
-3    93    93

   1,-16,L
 5   136   133
 4   120   101
 2    51    69

   1,-17,L
 5    57    63
 4    90    90
 3    79    71
 2   105    94
-1    82    81

   1,-18,L
 1    76    73

   0,17,L
 0    75    72
-4    93    82

   0,16,L
-1   102    99
-2   127   125
-3    88    87

   0,15,L
 3    52    45
 0   125   119
-2   112   107
-3   138   139
-4   150   149
-5    98    91

   0,14,L
 2    81    65
-1    75    74
-2    89    61
-3   100   106
-4    49    51
-5    76    84
-6    94    91

   0,13,L
 4   125   116
 3   140   138
 2   106   105
-3   123   124
-4   203   205
-8    65    65

   0,12,L
 6    76    66
 3   141    94
 2   117   115
-1    59    65
-2    57    64
-4    93   100
-5    75    84
-6    70    75
-7   119   110

   0,11,L
 6    95   100
 5   120   119
 4   144   139
 3   105   102
 1   127   127
 0   219   218
-1   103   121
-2   125   132
-5    96    96
-6    74    60
-7    66    79
-8   105    99
-9    98    92

   0,10,L
 3   163   161
 2   316   321
 1   207   215
-1    58    67
-2   146   156
-3   157   161
-5   184   181
-6    46    41

   0,9,L
 4   103   108
 3    83    92
 2    65    49
 1   229   243
 0   342   357
-1   389   444
-2   296   327
-4    45    44
-6    68    72
-7    40    42
-9    63    54

   0,8,L
 5    46    48
 3   132   138
 2   157   167
 1   383   370
 0    61    56
-1    48    29
-2   244   265
-3   225   237
-4   250   260
-5   178   185
-6    64    64

   0,7,L
 8    78    78
 7   135   148
 6    99    98
 5   108    99
 4    56    55
 3    43    37
 1    98    98
 0   126   112
-1   242   259
-2   227   237
-3    65    70
-4    43    55
-5   224   228
-6   143   143
-7   132   132

   0,6,L
 7    62    67
 4    47    54
 3    91    98
 2    41    59
 1    51    24
 0   225   226
-1    40    42
-2   254   280
-3   303   320
-4   446   450
-5   177   179
-7    66    52
-8   116   109

   0,5,L
 8    57    60
 7    76    84
 6   138   144
 5   234   236
 4   104   113
 3   159   164
 2   402   410
 1   670   677
 0   679   673
-1   239   240
-2   423   431
-3   130   141
-4    60    61
-5   214   214
-6   266   260
-7   165   174

   0,4,L
 7    60    72
 6   139   128
 5   249   280
 4   378   383
 3   313   321
 2   282   280
 1    96    86
 0    74    92
-1   374   380
-2   248   207
-3   180   177
-4   186   199
-5    97    95
-6   252   254
-7   260   257
-8   118   109
-9   125   124

   0,3,L
 6    91    90
 5    60    58
 3   481   459
 2   528   520
 1   720   737
 0   564   861
-1   413   423
-2   692   676
-3   365   339
-4    72    75
-5   247   243
-6   171   185
-7    97    97
-10   89    96

   0,2,L
 7    94   102
 6   196   199
 5   323   322
 4   454   454
 3   193   170
 2   252   225
 1  1288  1249
 0   368   369
-1   391   381
-2   427   403
-5    41    42
-7   380   381
-8   155   153
-9    97   101

   0,1,L
 7    92    93
 5   232   221
 3   539   553
 2   675   649
 1   561   551
-1   336   341
-2  1145  1094
-3   437   406
-4   318   313
-5   246   245
-6   177   170
-9    90    89
-10   80    81

   0,0,L
 7   170   170
 5   304   316
 4   207   189
 3   374   371
 2   224   221
 1   879   830
```

TABLE 9

Dicarbonyl(π-cyclopentadienyl)(triphenylphosphine)iron 1,1,2,3,3-pentacyanopropenide; fractional atomic coordinates with esd in parentheses and thermal parameters.

Part I: The cation

Atom	x	y	z	U_{iso}(x10^3Å^2)
Fe	0.58020(6)	0.73185(4)	0.11753(9)	*
P	0.71148(10)	0.84372(7)	0.03412(14)	*
C(1)	0.5563(7)	0.6054(4)	0.1851(9)	*
C(2)	0.6421(6)	0.6248(4)	0.0983(9)	*
C(3)	0.5772(6)	0.6318(4)	-0.0552(8)	*
C(4)	0.4518(6)	0.6175(4)	-0.0600(9)	*
C(5)	0.4395(5)	0.6022(4)	0.0882(10)	*
C(6)	0.7655(4)	0.9536(3)	0.1665(6)	*
C(7)	0.6793(5)	0.9962(4)	0.1577(8)	*
C(8)	0.7126(8)	1.0772(4)	0.2630(10)	*
C(9)	0.8312(9)	1.1164(4)	0.3769(8)	*
C(10)	0.9138(8)	1.0744(4)	0.3826(7)	*
C(11)	0.8848(5)	0.9944(4)	0.2806(6)	*
C(12)	0.8517(4)	0.8221(3)	0.0262(6)	*
C(13)	0.9232(4)	0.8040(3)	0.1614(6)	*
C(14)	1.0280(5)	0.7839(4)	0.1582(7)	*
C(15)	1.0594(5)	0.7791(5)	0.0229(9)	*
C(16)	0.9872(7)	0.7935(5)	-0.1146(9)	*
C(17)	0.8836(6)	0.8166(4)	-0.1118(7)	*
C(18)	0.6508(4)	0.8700(3)	-0.1573(6)	*
C(19)	0.7162(6)	0.9525(4)	-0.1930(7)	*
C(20)	0.6692(8)	0.9743(5)	-0.3400(8)	*
C(21)	0.5608(7)	0.9133(5)	-0.4505(7)	*
C(22)	0.4969(6)	0.8332(5)	-0.4177(7)	*
C(23)	0.5395(5)	0.8089(4)	-0.2702(6)	*
C(24)	0.6710(5)	0.7837(3)	0.3194(7)	*
O(25)	0.7292(4)	0.8141(3)	0.4482(5)	*
C(26)	0.4713(4)	0.7867(4)	0.1025(7)	*
O(27)	0.3997(4)	0.8203(3)	0.0942(7)	*

Atom	x	y	z	$U_{iso}(x10^3Å^2)$
H(1)	0.548	0.593	0.296	85
H(2)	0.730	0.628	0.110	78
H(3)	0.613	0.643	-0.143	74
H(4)	0.366	0.617	-0.157	72
H(5)	0.367	0.583	0.134	75
H(7)	0.584	0.961	0.069	71
H(8)	0.656	1.114	0.277	93
H(9)	0.840	1.176	0.454	88
H(10)	0.996	1.108	0.467	79
H(11)	0.945	0.963	0.264	60
H(13)	0.892	0.803	0.252	56
H(14)	1.075	0.760	0.262	65
H(15)	1.125	0.757	-0.001	104
H(16)	1.017	0.792	-0.207	100
H(17)	0.834	0.824	-0.210	74
H(19)	0.782	1.003	-0.110	74
H(20)	0.719	1.038	-0.362	91
H(21)	0.541	0.936	-0.571	92
H(22)	0.418	0.785	-0.480	81
H(23)	0.491	0.748	-0.245	65

* These atoms were assigned anisotropic temperature factors of the form: $T = \exp\left[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}\ell^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}h\ell a^*c^* + 2U_{23}k\ell b^*c^*)\right]$ with final parameters $(U_{ij}x10^4Å^2)$:

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Fe	350	474	672	170	83	132
P	386	439	482	174	50	79
C(1)	929	481	1058	166	158	255
C(2)	635	497	1160	280	107	107
C(3)	779	465	956	184	280	19
C(4)	609	513	969	132	-28	-42
C(5)	469	599	1139	46	179	132
C(6)	540	456	484	179	103	105
C(7)	593	608	871	258	234	70
C(8)	1139	626	1056	426	524	161
C(9)	1397	553	642	292	291	85
C(10)	1144	556	602	154	-165	55
C(11)	669	540	581	200	-85	57
C(12)	388	486	526	187	80	119
C(13)	467	631	570	271	85	111
C(14)	475	737	730	302	79	138
C(15)	566	899	998	400	290	277
C(16)	936	1155	921	625	569	410
C(17)	701	878	622	435	234	245
C(18)	509	570	514	290	-8	66
C(19)	875	623	639	319	99	184
C(20)	1196	805	679	544	223	314
C(21)	1084	1032	567	743	148	195
C(22)	678	1087	564	514	-17	43
C(23)	590	759	544	326	1	59
C(24)	529	593	635	285	164	168
O(25)	829	1010	615	457	162	127
C(26)	385	669	907	242	151	237
O(27)	557	960	1464	465	300	361

<u>Average estimated standard deviations</u> ($U_{ij} \times 10^4 Å^2$)

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Fe	3	3	4	3	3	3
P	5	6	6	5	5	5
O	25	31	36	24	24	26
C	36	33	39	29	30	28

Part II: The anion†

Atom	x	y	z	U_{iso} ($\times 10^4$ Å^2)††
C(31)	1.1996(25)	0.4838(15)	0.4356(23)	939
C(32)	1.1076(13)	0.5056(9)	0.3336(14)	653
C(33)	0.9727(12)	0.4676(8)	0.2577(15)	701
C(34)	1.1412(16)	0.3900(10)	0.4883(18)	678
N(34)	1.1230(18)	0.3176(13)	0.5339(22)	1021
C(35)	1.3206(31)	0.5255(19)	0.4758(35)	1113
N(35)	1.4151(24)	0.5694(17)	0.5119(31)	1304
C(36)	1.1645(14)	0.5937(12)	0.2832(19)	717
N(36)	1.2350(14)	0.6633(11)	0.2374(18)	833
C(37)	0.9177(15)	0.5116(12)	0.1613(20)	753
N(37)	0.8507(16)	0.5368(11)	0.0792(21)	932
C(38)	0.8922(25)	0.3822(18)	0.2981(29)	805
N(38)	0.8130(19)	0.3305(14)	0.3164(26)	1133
C(31)'	1.1501(14)	0.4575(10)	0.4285(15)	644
C(32)'	1.0514(15)	0.4697(10)	0.3251(16)	732
C(33)'	1.0517(11)	0.5406(8)	0.2485(14)	675
C(34)'	1.2832(23)	0.5229(13)	0.4852(23)	802
N(34)'	1.3815(31)	0.5615(20)	0.5433(25)	1429
C(35)'	1.1032(21)	0.3724(15)	0.4948(25)	948
N(35)'	1.0950(17)	0.3216(12)	0.5589(21)	931
C(36)'	0.9131(22)	0.3959(16)	0.2848(29)	845
N(36)'	0.8168(18)	0.3314(13)	0.2619(23)	983
C(37)'	0.9343(15)	0.5327(11)	0.1295(19)	695
N(37)'	0.8613(12)	0.5396(8)	0.0290(17)	742
C(38)'	1.1730(16)	0.6220(14)	0.2645(19)	724
N(38)'	1.2328(14)	0.6825(10)	0.2849(19)	844

† The population parameter of every atom of the anion = 0.50.

†† The average estimated standard deviations of U_{iso} ($\times 10^4$ Å^2) of the carbon and nitrogen atoms are 49 and 57 respectively.

TABLE 10

Dicarbonyl(π-cyclopentadienyl)(triphenylphosphine)iron 1,1,2,3,3-pentacyanopropenide; bond lengths (Å) with esd in parentheses.

Part I: The cation

Fe - P	2.240(1)	C(6) - C(7)	1.397(9)
Fe - C(24)	1.778(5)	C(7) - C(8)	1.389(9)
Fe - C(26)	1.774(7)	C(8) - C(9)	1.385(11)
C(24) - O(25)	1.129(7)	C(9) - C(10)	1.355(14)
C(26) - O(27)	1.138(9)	C(10) - C(11)	1.374(9)
Fe - C(1)	2.096(7)	C(11) - C(6)	1.392(6)
Fe - C(2)	2.085(7)	C(12) - C(13)	1.392(7)
Fe - C(3)	2.093(7)	C(13) - C(14)	1.389(9)
Fe - C(4)	2.102(5)	C(14) - C(15)	1.347(11)
Fe - C(5)	2.092(5)	C(15) - C(16)	1.383(11)
C(1) - C(2)	1.394(12)	C(16) - C(17)	1.400(12)
C(2) - C(3)	1.413(10)	C(17) - C(12)	1.374(9)
C(3) - C(4)	1.406(10)	C(18) - C(19)	1.382(8)
C(4) - C(5)	1.392(12)	C(19) - C(20)	1.402(10)
C(5) - C(1)	1.405(10)	C(20) - C(21)	1.362(8)
P - C(6)	1.820(5)	C(21) - C(22)	1.337(11)
P - C(12)	1.821(5)	C(22) - C(23)	1.413(9)
P - C(18)	1.810(5)	C(23) - C(24)	1.388(6)

The C - H distances range from 0.95 - 1.15, mean 1.03 Å.

Part II: The anion

C(31) - C(32)	1.390(30)	C(31)' - C(32)'	1.369(23)
C(32) - C(33)	1.436(18)	C(32)' - C(33)'	1.368(21)
C(31) - C(34)	1.580(28)	C(31)' - C(34)'	1.471(25)
C(34) - N(34)	1.221(27)	C(34)' - N(34)'	1.062(36)
C(31) - C(35)	1.282(40)	C(31)' - C(35)'	1.507(28)
C(35) - N(35)	1.034(37)	C(35)' - N(35)'	1.021(31)
C(32) - C(36)	1.491(22)	C(32)' - C(36)'	1.569(26)
C(36) - N(36)	1.303(23)	C(36)' - N(36)'	1.190(27)
C(33) - C(37)	1.333(24)	C(33)' - C(37)'	1.466(20)
C(37) - N(37)	1.138(27)	C(37)' - N(37)'	1.112(22)
C(33) - C(38)	1.497(29)	C(33)' - C(38)'	1.523(20)
C(38) - N(38)	1.070(34)	C(38)' - N(38)'	0.942(22)

TABLE 11

Dicarbonyl(π-cyclopentadienyl)(triphenylphosphine)iron 1,1,2,3,3-pentacyanopropenide; valency angles (°) with esd in parentheses.

Part I: The cation

P - Fe - C(24)	90.7(2)	C(6) - P - C(12)	107.6(2)
P - Fe - C(26)	91.9(2)	C(6) - P - C(18)	101.5(2)
C(24) - Fe - C(26)	96.4(3)	C(12) - P - C(18)	105.4(3)
Fe - C(24) - O(25)	177.9(6)	P - C(6) - C(7)	117.6(3)
Fe - C(26) - O(27)	178.4(7)	P - C(6) - C(11)	123.5(5)
P - Fe - C(1)	142.4(2)	C(11) - C(6) - C(7)	118.8(5)
P - Fe - C(2)	104.7(2)	C(6) - C(7) - C(8)	120.0(5)
P - Fe - C(3)	91.6(2)	C(7) - C(8) - C(9)	120.4(8)
P - Fe - C(4)	115.5(2)	C(8) - C(9) - C(10)	118.6(6)
P - Fe - C(5)	154.1(2)	C(9) - C(10) - C(11)	122.8(6)
C(24) - Fe - C(1)	87.1(3)	C(10) - C(11) - C(6)	119.3(6)
C(24) - Fe - C(2)	97.8(3)	P - C(12) - C(13)	118.7(4)
C(24) - Fe - C(3)	136.0(3)	P - C(12) - C(17)	122.0(4)
C(24) - Fe - C(4)	151.5(3)	C(17) - C(12) - C(13)	119.1(5)
C(24) - Fe - C(5)	114.0(3)	C(12) - C(13) - C(14)	120.4(5)
C(26) - Fe - C(1)	125.7(3)	C(13) - C(14) - C(15)	120.3(6)
C(26) - Fe - C(2)	158.0(2)	C(14) - C(15) - C(16)	120.5(7)
C(26) - Fe - C(3)	127.4(3)	C(15) - C(16) - C(17)	119.8(7)
C(26) - Fe - C(4)	94.0(3)	C(16) - C(17) - C(12)	119.9(6)
C(26) - Fe - C(5)	93.0(3)	P - C(18) - C(19)	119.6(3)
C(1) - C(2) - C(3)	107.6(6)	P - C(18) - C(23)	121.4(4)
C(2) - C(3) - C(4)	107.8(7)	C(23) - C(18) - C(19)	119.0(5)
C(3) - C(4) - C(5)	108.0(6)	C(18) - C(19) - C(20)	120.2(5)
C(4) - C(5) - C(1)	108.2(7)	C(19) - C(20) - C(21)	120.3(6)
C(5) - C(1) - C(2)	108.3(7)	C(20) - C(21) - C(22)	120.0(7)
Fe - P - C(6)	111.2(2)	C(21) - C(22) - C(23)	121.7(5)
Fe - P - C(12)	111.5(2)	C(22) - C(23) - C(18)	118.8(5)
Fe - P - C(18)	118.7(2)		

The cyclopentadienyl C - C - H angles range from 109 - 142, mean 126°.

The phenyl C - C - H angles range from 108 - 130, mean 120°.

Part II: The anion

C(32) - C(31) - C(34)	112.4(17)	C(32)' - C(31)' - C(34)'	125.8(15)
C(32) - C(31) - C(35)	127.8(24)	C(32)' - C(31)' - C(35)'	111.1(14)
C(34) - C(31) - C(35)	119.4(25)	C(34)' - C(31)' - C(35)'	122.8(16)
C(31) - C(34) - N(34)	165.4(22)	C(31)' - C(34)' - N(34)'	168.3(28)
C(31) - C(35) - N(35)	169.8(41)	C(31)' - C(35)' - N(35)'	165.2(21)
C(31) - C(32) - C(33)	139.0(15)	C(31)' - C(32)' - C(33)'	129.7(12)
C(31) - C(32) - C(36)	111.7(14)	C(31)' - C(32)' - C(36)'	119.4(16)
C(33) - C(32) - C(36)	109.2(12)	C(33)' - C(32)' - C(36)'	110.9(15)
C(32) - C(36) - N(36)	166.4(19)	C(32)' - C(36)' - N(36)'	170.3(28)
C(37) - C(33) - C(38)	119.8(16)	C(37)' - C(33)' - C(38)'	117.9(13)
C(32) - C(33) - C(37)	120.5(13)	C(32)' - C(33)' - C(37)'	119.0(11)
C(32) - C(33) - C(38)	119.5(16)	C(32)' - C(33)' - C(38)'	122.5(13)
C(33) - C(37) - N(37)	167.4(18)	C(33)' - C(37)' - N(37)'	166.2(17)
C(33) - C(38) - N(38)	161.9(33)	C(33)' - C(38)' - N(38)'	161.4(26)

TABLE 12

Dicarbonyl(π-cyclopentadienyl)(triphenylphosphine)iron 1,1,2,3,3-pentacyanopropenide; mean plane calculations.

Part I: The cation

	Atoms in plane	Displacements (Å)	Atoms out of plane	Displacements (Å)
a)	C(1)	0.007	H(1)	0.02
	C(2)	-0.006	H(2)	0.08
	C(3)	0.002	H(3)	0.02
	C(4)	0.003	H(4)	0.01
	C(5)	-0.006	H(5)	0.09
b)	C(6)	-0.006	P	-0.122
	C(7)	0.003	H(7)	-0.05
	C(8)	0.002	H(8)	-0.07
	C(9)	-0.004	H(9)	-0.08
	C(10)	0.001	H(10)	0.01
	C(11)	0.004	H(11)	0.15
c)	C(12)	-0.009	P	0.085
	C(13)	0.015	H(13)	0.10
	C(14)	-0.005	H(14)	0.16
	C(15)	-0.010	H(15)	0.10
	C(16)	0.015	H(16)	-0.04
	C(17)	-0.006	H(17)	0.05
d)	C(18)	-0.001	P	-0.005
	C(19)	0.007	H(19)	-0.21
	C(20)	-0.009	H(20)	-0.04
	C(21)	0.005	H(21)	0.13
	C(22)	0.001	H(22)	-0.04
	C(23)	-0.003	H(23)	-0.01

The dihedral angles (°) between selected planes are:

b) - c) 122.4 c) - d) 76.5

b) - d) 69.9

Part II: The anion

	Atoms in plane	Displacements (Å)	Atoms out of plane	Displacements (Å)
e)	C(31)	-0.058		
	C(32)	-0.038		
	C(33)	0.044		
	C(34)	0.006		
	N(34)	0.173		
	C(35)	0.006		
	N(35)	-0.073		
	C(36)	-0.078		
	N(36)	0.078		
	C(37)	0.054		
	N(37)	0.047		
	C(38)	-0.001		
	N(38)	-0.159		
f)	C(31)	0.005	C(32)	0.136
	C(34)	-0.045		
	N(34)	0.022		
	C(35)	0.050		
	N(35)	-0.032		
g)	C(33)	-0.019	C(32)	-0.205
	C(37)	-0.008		
	N(37)	0.008		
	C(38)	0.045		
	N(38)	-0.026		

	Atoms in plane	Displacements (Å)	Atoms out of plane	Displacements (Å)
h)	C(31)'	0.091		
	C(32)'	0.012		
	C(33)'	-0.047		
	C(34)'	0.089		
	N(34)'	0.057		
	C(35)'	0.009		
	N(35)'	-0.069		
	C(36)'	-0.067		
	N(36)'	-0.049		
	C(37)'	0.018		
	N(37)'	0.144		
	C(38)'	0.018		
	N(38)'	-0.206		
i)	C(31)'	-0.005	C(32)'	-0.175
	C(34)'	0.005		
	N(34)'	-0.003		
	C(35)'	0.005		
	N(35)'	-0.002		
j)	C(33)'	-0.026	C(32)'	0.188
	C(37)'	-0.044		
	N(37)'	0.028		
	C(38)'	0.113		
	N(38)'	-0.072		

The dihedral angles (°) between selected planes are:

e) - f) 5.2 h) - i) 4.4
e) - g) 4.8 h) - j) 3.5
e) - h) 7.5

TABLE 13

Dicarbonyl(π-cyclopentadienyl)(triphenylphosphine)iron 1,1,2,3,3-pentacyanopropenide; distances (≤1.25Å) between atoms of the different orientations of the anion.

C(31)	-	C(31)'	0.57	N(35)	-	N(34)'	0.53
C(31)	-	C(34)'	0.93	C(36)	-	C(33)'	1.24
C(32)	-	C(32)'	0.69	C(36)	-	C(38)'	0.48
C(33)	-	C(32)'	0.96	N(36)	-	C(38)'	0.90
C(33)	-	C(33)'	1.24	N(36)	-	N(38)'	0.51
C(33)	-	C(36)'	1.20	C(37)	-	C(37)'	0.48
C(34)	-	C(31)'	1.23	N(37)	-	C(37)'	1.00
C(34)	-	C(35)'	0.46	N(37)	-	N(37)'	0.49
N(34)	-	C(35)'	1.05	C(38)	-	C(36)'	0.32
N(34)	-	N(35)'	0.46	C(38)	-	N(36)'	0.93
C(35)	-	C(34)'	0.46	N(38)	-	N(36)'	0.50
C(35)	-	N(34)'	0.80				

TABLE 14

Dicarbonyl(π-cyclopentadienyl)(triphenylphosphine)iron 1,1,2,3,3-pentacyanopropenide; interionic contacts (≤3.60Å).

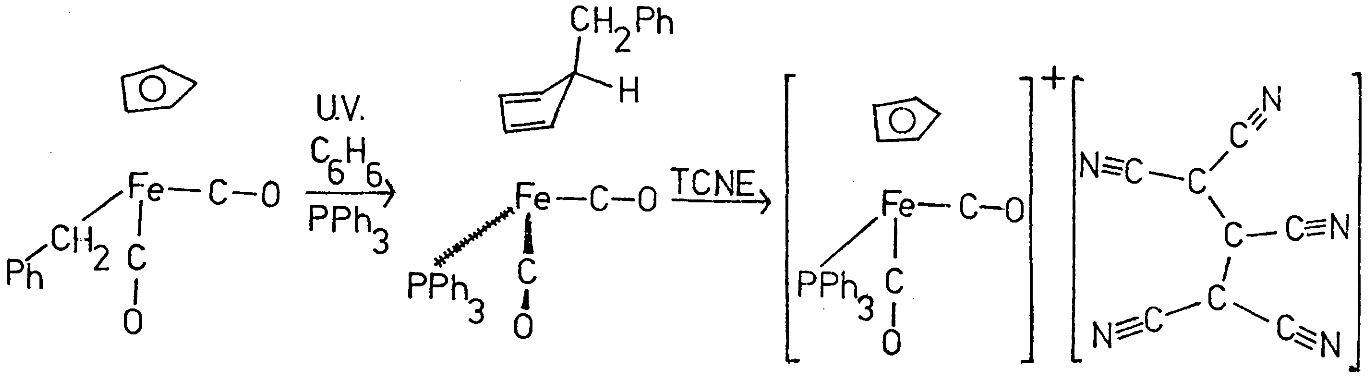
$N(34)$	---	$O(25^{I})$	3.15	$N(35)'$	---	$O(25^{I})$	3.49
$N(36)$	---	$O(27^{II})$	3.18	$N(35)'$	---	$C(9^{VIII})$	3.51
$O(25)$	---	$C(21^{III})$	3.18	$N(37)$	---	$C(4^{V})$	3.51
$C(36)$	---	$N(37^{IV})'$	3.21	$N(34)$	---	$C(13^{I})$	3.51
$N(37)$	---	$C(5^{V})$	3.22	$C(33)'$	---	$C(37^{IV})'$	3.52
$C(33)'$	---	$N(37^{IV})'$	3.23	$C(2)$	---	$C(5^{V})$	3.52
$N(38)'$	---	$O(27^{II})$	3.30	$N(35)'$	---	$C(2^{I})$	3.53
$N(37)'$	---	$C(5^{V})$	3.30	$C(37)$	---	$C(35^{I})'$	3.53
$C(35)'$	---	$C(13^{I})$	3.32	$N(37)$	---	$N(35^{I})'$	3.53
$N(37)'$	---	$C(38^{IV})'$	3.33	$C(37)$	---	$N(35^{I})'$	3.54
$O(25)$	---	$C(22^{III})$	3.35	$N(34)'$	---	$C(3^{VI})$	3.54
$C(37)'$	---	$C(37^{IV})'$	3.36	$N(36)'$	---	$O(27^{V})$	3.54
$N(34)'$	---	$C(4^{VI})$	3.36	$C(37)'$	---	$N(37^{IV})'$	3.54
$N(36)$	---	$C(26^{II})$	3.38	$C(24)$	---	$C(21^{III})$	3.55
$C(32)$	---	$N(37^{IV})'$	3.38	$C(32)'$	---	$N(37^{IV})'$	3.56
$N(36)$	---	$C(5^{II})$	3.40	$C(32)$	---	$N(38^{I})$	3.56
$O(25)$	---	$C(20^{III})$	3.41	$C(36)$	---	$N(38^{I})$	3.56
$N(34)$	---	$C(24^{I})$	3.42	$N(38)$	---	$C(9^{VIII})$	3.57
$N(38)'$	---	$C(22^{VI})$	3.44	$C(2)$	---	$C(4^{V})$	3.58
$N(38)$	---	$C(34^{I})'$	3.46	$C(1)$	---	$C(4^{V})$	3.58
$N(36)$	---	$N(37^{IV})'$	3.47	$C(32)$	---	$C(38^{I})$	3.58
$C(10)$	---	$C(16^{VII})$	3.47	$N(38)'$	---	$C(26^{II})$	3.59
$N(34)$	---	$C(2^{I})$	3.49	$C(31)$	---	$N(38^{I})$	3.59
$C(1)$	---	$C(3^{V})$	3.49	$C(36)$	---	$N(37^{IV})$	3.60
$N(35)'$	---	$C(14^{I})$	3.49				

The superscripts refer to the following transformations of the atomic coordinates:

I	2 - x,	1 - y,	1 - z;	V	1 - x,	1 - y,	- z;
II	1 + x,	y,	z;	VI	1 + x,	y,	1 + z;
III	x,	y,	1 + z;	VII	2 - x,	2 - y,	- z;
IV	2 - x,	1 - y,	- z;	VIII	x,	1 - y,	z.

III.3.4 DISCUSSION

These analyses, together with the independent chemical synthesis of B, have demonstrated that the reaction sequence under investigation was:

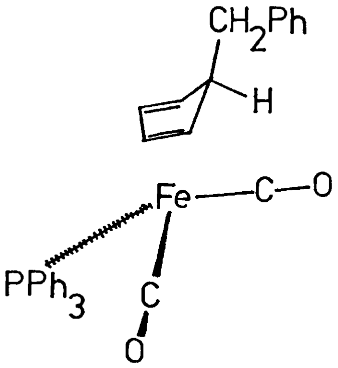


A

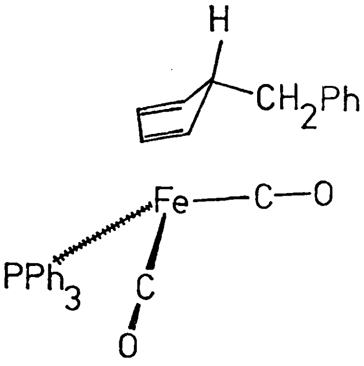
B

In the reaction A ⟶ B, the conversion of the 'butadiene' ligand, normally considered a neutral 4-electron donor, into a cyclopentadienyl ligand, customarily a 6-electron $C_5H_5^-$ donor, is accompanied by a change in the formal oxidation state of the iron atom from zero to two.

Both crystallographically independent molecules of A are the exo- rather than the endo-isomer suggesting that an intermolecular



exo-isomer



endo-isomer

migration of the benzyl group has occurred. The pathway from A to B is uncertain, but reactions involving cyclo-diene $Fe(CO)_3$ complexes often generate stable π-pentadienyl groups(16), and this, together with the formation of another stable ion [Numerous complexes of the resonance-stabilized anion, 1,1,2,3,3-pentacyanopropenide, are known(15).]

is probably part of the driving force behind the reaction.

The dimensions of the butadiene — $Fe(CO)_2(PPh_3)$ linkage in A are in good agreement with those in butadiene — $Fe(CO)_3$ linkages and, as is typical of the latter linkages, the Fe - C(terminal) distances [2.123(6) and 2.102(6) Å in A1 and 2.126(5) and 2.106(5) Å in A101] are significantly longer than the Fe - C(central) distances [2.065(6) and 2.046(6) Å in A1 and 2.062(6) and 2.049(6) Å in A101], the mean values of 2.114 and 2.056 Å respectively being comparable with the corresponding mean values of 2.124 and 2.052 Å in N-ethoxycarbonyl-3-formylazepine-$Fe(CO)_3$ (17) and 2.131 and 2.050 Å in sorbic acid $Fe(CO)_3$ (18). The C - C bond lengths within the butadiene group are essentially the same [1.425(7), 1.414(8) and 1.427(7) Å in A1 and 1.419(8), 1.417(9) and 1.412(8) Å in A101] and are intermediate between single- and double-bond values for carbon - carbon bonds, e.g. 1.337 and 1.483 Å respectively in trans-butadiene(19), reflecting delocalisation of the π-electrons over the bonded atoms as a result of back-donation from filled iron d orbitals into the lowest antibonding orbital of the butadiene group(20). This back-donation is more pronounced, demonstrated by the central C - C bond being shorter than the outer C - C bonds, if instead of strongly π-accepting carbonyl ligands being trans to the butadiene group, there is a more weakly π-accepting ligand. For example, compare the distances of 1.414(7), 1.407(7) and 1.409(7) Å in 1H-azepine $Fe(CO)_3$ (21) with 1.49(3), 1.36(3) and 1.54(3) in 1-exo-benzoylcyclopenta-2,4-diene-$Co(\pi\text{-}C_5H_5)$ (22).

The conformation of the cyclopentadiene ligand, as is the case with other organic ligands, e.g. 1H-azepine(21) and cyclo-octatetraene(23), is greatly altered on complex formation, with the planarity(24,25) being destroyed by the displacement of C(1) by 0.58 Å in A1 and 0.56 Å in A101 out of the butadiene plane and away from the iron atom. The resultant non-bonded Fe - C(1) distance is 2.664(6) Å in A1 and 2.655(6) Å in A101. The dihedral angle between the butadiene plane and the plane defined by atoms C(2), C(1) and C(5) is 34.7 and 32.9° in A1 and A101 respectively, compared with corresponding values of 37, 37 and 41° in 1-exo-benzoylcyclopenta-2,4-diene $Co(\pi-C_5H_5)$(22), 1-exo-phenylcyclopenta-2,4-diene $Co(\pi-C_5H_5)$(26) and 1-exo-methylcyclopenta-2,4-diene $Re(Me)_2(\pi-C_5H_5)$(27) respectively.

The dimensions of the 1-exo-benzyl susbstituent in A are normal and of little interest.

The Fe - C(cyclopentadienyl) distances in B are similar to the Fe - C(butadiene) distances in A and range from 2.085 - 2.102, mean 2.094(6†) Å, in good agreement with values of 2.074 - 2.097, mean 2.089(8†) Å, in trans-1,4-bis-$[(\pi-C_5H_5)Fe(CO)_2]$buta-1,3-diene(28) and 2.082 - 2.121, mean 2.106(15†) Å, in trans-di-μ-carbonyl-dicarbonyldi-π-cyclopentadienyldi-iron(29). The lengthening of these values compared with the Fe - C distance of 2.058(5) Å found in an electron-diffraction study of $(\pi-C_5H_5)_2Fe$(30) is expected(31), because one of the cyclopentadienyl rings in $(\pi-C_5H_5)_2Fe$ has been replaced in the above complexes by strongly π-accepting ligands. The C - C bond lengths in the cyclopentadienyl ring in B range from 1.392 - 1.413, mean 1.402(8†) Å, compared with a value of 1.419 Å found from an average of 23 different X-ray analyses of π-cyclopentadienyl derivatives(31) and with values of 1.431(5) and 1.427(7) Å found in the electron-diffraction studies of $(\pi-C_5H_5)_2Fe$(30) and $(\pi-C_5H_5)In$(32) respectively. The shorter values found in the X-ray analyses have been attributed to thermal motion(31). The C - C - C angles range from 107.6 - 108.3° with an average value

† Root-mean-square deviation from the mean.

of 108.0° consistent with that of a planar five-membered ring, the root-mean-square deviation from planarity being 0.005 Å. None of the deviations of the Fe - C, C - C or C - C - C dimensions from their mean value is significant, unlike some other complexes, e.g. $(\pi\text{-}C_5H_5)Cr(NO)_2(NCO)$(33) and $(\pi\text{-}C_5H_5)Mo(CO)(PPh_3)_2(NCO)$(1), in which deviations occur due to the lack of cylindrical symmetry around the metal atom removing the degeneracy of the e_1 molecular orbitals of the cyclopentadienyl ring(34).

The Fe - P bond length in A [2.213(2) and 2.216(2) Å in A1 and A101 respectively; mean 2.215(1) Å] is significantly shorter than in B, 2.240(1) Å, both being similar to the distances of 2.24(1) and 2.25(1) Å in different isomers of $Fe_3(CO)_{11}(PPh_3)$(35) and mean distances of 2.237(5) and 2.244(4) Å in $Fe_3(CO)_9(PMe_2Ph)_3$(36) and [1,2-bis(diphenylphosphino)hexafluorocyclopentene] $Fe(NO)_2$(37) respectively. Assuming the 'trans influence' of the butadiene and cyclopentadienyl ligands is similar, the Fe - P bond in B, formally a Fe^{II} complex, would be expected to be shorter than in A, formally a Fe^{0} complex, due to the shorter covalent radii of Fe^{II}. On the other hand, this bond would be expected to be longer in B due to both the π-bonded effect of the decreased dπ-pπ Fe ⟶ P back-donation as a result of the contraction, compared with A, of the metal d orbitals, and the σ-bonded effect of the phosphorus orbital directed towards the more electronegative metal, i.e. Fe^{II}, having the greater p-character(38). [Greater p-character indicates a longer bond, cf. the covalent radii of single-bonded $C(sp^3)$, $C(sp^2)$ and C(sp) atoms of 0.77, 0.74 and 0.71 Å respectively.] The latter considerations appear to be the more important in determining the difference in the Fe - P bond lengths in the present analyses, and although the Fe - CO bond lengths in A [1.767(5) and

1.763(6) Å in A1 and 1.775(6) and 1.768(6) Å in A101; mean 1.768(3) Å] and in B [1.778(5) and 1.774(7), mean 1.776(4) Å] are not significantly different, the variation is in the same sense. These Fe - CO bond lengths compare favourably with those in similar complexes, e.g. mean values of 1.759 and 1.768 Å in sorbic acid $Fe(CO)_3$(18) and $[(\pi-C_5H_5)Fe(CO)_2]_2SO_2$(39) respectively. Also consistent with greater back-donation in A, but again not statistically significant, the C - O bond lengths in A [1.148(7) and 1.145(7) Å in A1 and 1.145(7) and 1.137(8) Å in A101; mean 1.144(4) Å] are slightly longer than in B [1.129(7) and 1.138(9), mean 1.134(6) Å], both being in excellent agreement with the value of 1.14(1) Å given by Sutton(40). The Fe - C - O angles in A [179.0(5) and 178.0(5)° in A1 and 179.4(5) and 176.1(5)° in A101] and in B [177.9(6) and 178.4(7)°] depart only slightly from linearity.

Since there is more p-character in the phosphorus orbital directed towards the more electronegative iron atom, i.e. Fe^{II}, the phosphorus orbitals directed towards the carbon atoms in that complex should have more s-character than those in A. This approach suggests that the P - C bond lengths in B should be the shorter and this is indeed found, cf. 1.844(5), 1.850(5) and 1.833(5) Å in A1 and 1.835(5), 1.834(5) and 1.831(6) Å in A101, mean 1.838 Å; and 1.820(5), 1.821(5) and 1.810(5), mean 1.817 Å in B. These values are comparable with the sum of the covalent radii, 1.84 Å, with mean P - C(phenyl) distances of 1.810, 1.828 and 1.833 Å in trans-$(PPh_2Me)_2Ni(\sigma-C_6F_5)Br$(41), $Cr(CO)_5(PPh_3)$(42) and $(\pi-C_5H_5)Mo(CO)_2(PPh_3)I$(43) respectively and with the mean distance, 1.828 Å, in the uncomplexed triphenylphosphine molecule(44). The deviations of the C - P - C angles in triphenylphosphine ligands from ideal sp^3 hybridisation [The mean values of 102.6 and 103.4° found in $Cr(CO)_5(PPh_3)$(42) and $(\pi$-$C_5H_5)Mo(CO)_2(PPh_3)I$(43) respectively are typical of those in triphenylphosphine transition-metal complexes.] have been suggested(43) to be indicative of the phosphorus orbital directed towards the metal atom having more s-character than the orbitals directed towards the carbon atoms. Therefore in view of the suggested greater s-character in the P - C bonds in B compared with A, the C - P - C angles in A should be smaller than those in B with both significantly less than the tetrahedral value of 109°28'. The observed values [102.0(2), 102.7(2) and 103.5(2)° in A1 and 101.7(2), 104.1(3) and 103.2(2)° in A101, mean 102.9°; and 107.6(2), 101.5(2) and 105.4(3), mean 104.8° in B], although consistent with this argument, may merely be a reflection of different van der Waals' repulsions between the phenyl groups in A and B.

The C - C bond lengths in the triphenylphosphine ligands range from 1.368 - 1.409, mean 1.384 Å, in A1, 1.369 - 1.406, mean 1.386 Å, in A101 and 1.337 - 1.413, mean 1.381 Å, in B; contractions from the spectroscopic value of 1.397 Å appropriate to benzene similar to that noted in other X-ray analyses(e.g. 1,41,43) and attributed to thermal motion. While the phenyl rings are accurately planar [The root-mean-square deviations from planarity are 0.004, 0.006 and 0.006 Å in A1, 0.002, 0.002 and 0.004 Å in A101 and 0.003, 0.011 and 0.005 Å in B.] the phosphorus atoms are appreciably displaced from some of these planes. [The displacements are 0.01, 0.10 and 0.11 Å in A1, 0.09, 0.04 and 0.36 Å in A101 and 0.12, 0.09 and 0.01 Å in B.] Considerable phosphorus displacements of this type have been noted in other X-ray analyses, e.g. 0.23 and 0.21 Å in $(\pi$-$C_5H_5)Mo(CO)(PPh_3)_2(NCO)$(1) and $(Ph_2PC_6H_4 \cdot CH{:}CH \cdot Me)Mo(CO)_4$(45) respectively. In the two crystallographically independent molecules of A the different phosphorus displacements and the different rotational orientation of the phenyl rings [cf. dihedral angles between the phenyl rings of 103.4, 90.2 and 59.6° in A1 and 102.1, 64.1 and 102.5° in A101] demonstrate, as does the crystal structure of $(\pi\text{-}C_5H_5)Mo(CO)_2(PPh_3)Br$(17) which also contains two crystallographically independent molecules per asymmetric unit, the importance of intramolecular and intermolecular repulsions in determining the conformation of triphenylphosphine ligands in transition-metal complexes.

Different intermolecular repulsions also probably account for the slightly different orientation in A1 and A101 of the $Fe(CO)_2(PPh_3)$ group with respect to the butadiene ligand. Both conformations resemble the preferred conformation(46) in 1,3-diene $Fe(CO)_3$ complexes in which, when viewed on to the butadiene plane, the two formal double bonds of the diene are eclipsed by Fe - CO bonds. [Illustrated in the case of N-ethoxycarbonyl-3-formylazepine $Fe(CO)_3$ on page 24.] The coordination about the iron atom can be described as a distorted square pyramid with the iron atom above the plane [0.55 Å in A1 and 0.51 Å in A101] through atoms P(1) and C(33) and the midpoints of the formal double bonds of the diene, and with atom C(31) occupying the apical position. In 1,3-diene $Fe(CO)_3$ complexes the angle between the basal Fe - CO bonds is usually smaller than the angles between apical and basal Fe - CO bonds, e.g. angles of 90.2, 102.5 and 98.4° in sorbic acid $Fe(CO)_3$(18), and this feature is also evident in A, cf. P(1) - Fe(1) - C(33) angles of 92.4(2) and 94.7(2)° in A1 and A101 respectively and P(1) - Fe(1) - C(31) and C(31) - Fe(1) - C(33) angles of 97.5(2) and 101.1(3)° respectively in A1 and 95.8(2) and 101.8(3)° respectively in A101.

Regarding the cyclopentadienyl ligand in B as tridentate, the iron atom has a distorted octahedral coordination in which the angles between the monodentate ligands [The P - Fe - C(24), P - Fe - C(26) and C(24) - Fe - C(26) angles are 90.7(2), 91.9(2) and 96.4(3)° respectively.] are close to the ideal octahedral value of 90.0°. The OC - Fe - CO angle is significantly greater than the OC - Fe - PPh_3 angles, a disposition also found in A, suggesting that these differences are not determined by the greater size of the triphenylphosphine ligand, but reflect the better π-accepting ability of the carbonyl ligands when considering the non-bonded repulsions between the ligand orbitals which receive electron density by back-donation from the metal. In these analyses the greater π-accepting ability of the carbonyl ligands, reflected in the greater double-bond character of the Fe - CO bonds, is apparent from a comparison of the Fe - CO and Fe - PPh_3 bond lengths, ca. 1.77 and 2.23 Å respectively, and the covalent radii of $C(sp^3)$ and P atoms, 0.71 and 1.10 Å respectively. Non-bonded repulsion considerations of this type have been used previously$^{(47 - 50)}$ to explain similar angular dispositions, e.g. the larger OC/ON - Co - CO/NO angles, mean 113.4°, compared with the OC/ON - Co - PPh_3 angles, mean 105.2°, in $Co(CO)_2(NO)(PPh_3)$$^{(49)}$.

Although the disorder has resulted in the individual geometrical values in the 1,1,2,3,3-pentacyanopropenide anion being of limited meaning, the gross structure indicates that the anion is non-planar with the $C(CN)_2$ groups twisted out of the completely planar conformation by 5.2 and 4.8° in anion C(31) - - and by 4.4 and 3.5° in anion C(31)' - -. In 2-cyanomethyl-1,1,3,3-tetracyanopropenide$^{(8)}$ a corresponding twisting of 5.5° is observed which, together with the opening to about 125° of the angles corresponding to C(34) - C(31) - C(32), C(31) - C(32) - C(33) and C(32) - C(33) - C(38), avoids short intraionic contacts between the arms corresponding to C(34) - N(34) and C(38) - N(38). In this analysis the C(31) - C(32) - C(33) and C(31)' - C(32)' - C(33)' angles of 139.0 and 129.7° respectively may indicate a similar valency angle distortion. In the $C(CN)_2$ groups the C - CN distances range from 1.282 - 1.580, mean 1.457 Å, and the C - C - C angles from 117.9 - 122.8, mean 120.0°, compared with corresponding mean values of 1.425 Å and 114.2° in 2-cyanomethyl-1,1,3,3-tetracyanopropenide(8), and 1.439 Å and 115.6° for the one independent measurement of these values in TCNE(12). The ten C - N distances range from 0.942 - 1.303, mean 1.109 Å, and the C - C - N angles from 161.4 - 170.3, mean 166.2°, whereas in 2-cyanomethyl-1,1,3,3-tetracyanopropenide(8) and TCNE(12) the geometry is almost linear with C - N distances of 1.135 (mean) and 1.143 Å respectively. There is least overlap between the two orientations involving the central part of the anion, resulting in reasonable agreement among the four measurements equivalent to C(31) - C(32), which range from 1.368 - 1.436, mean 1.391 Å, in good accord with the corresponding value of 1.391 Å in 2-cyanomethyl-1,1,3,3-tetracyanopropenide(8).

The intermolecular contacts in the crystal structure of A and the interionic contacts in that of B appear to be purely van der Waals' interactions.

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