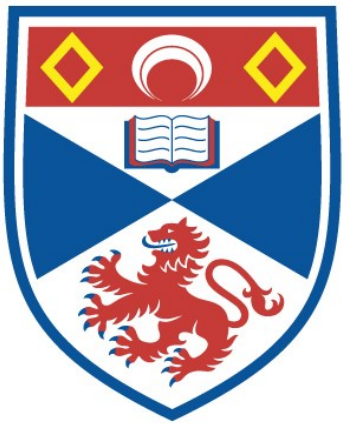
FERROCENYLKETONES AND THE STABILISATION OF THEIR THIO ANALOGUES

William Bell

A Thesis Submitted for the Degree of PhD
at the
University of St Andrews



1991

Ferrocenylketones and the Stabilisation of their Thio Analogues.

By William Bell.

A Thesis Presented to the

University of St. Andrews for the

Degree of

Doctor of Philosophy.

Dedication

To my Dad, Brethren and Jane.

Declaration for the Degree of Ph.D.

I William Bell hereby certify that this thesis has been composed by myself, that it is a record of my own work, and that it has not been accepted in partial or complete fulfillment of any other degree or professional qualification

Signed Date 23-10-90

I was admitted to the Faculty of Science of the University of St.Andrews under Ordinance General No 12 on the 1st of October 1987 and as a candidate for the degree of Ph.D. on the 22nd of October 1990

Signed Date 23-10-90

Certificate

I hereby certify that the candidate has fulfilled the conditions of the Resolution and Regulations appropriate to the Degree of Ph.D.

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Summary

Chapter One gives a brief overview of some chemistry of ferrocene and a few examples of the types of transformations possible using the technique of Flash Vacuum Pyrolysis. Chapter Two deals with the complexation of unstable thiones to $M(CO)_5$ (M= Cr,Mo, W) to give very stable products. The crystal structure has been determined for pentacarbonyl(thiobenzoylferrocene-S) chromium and also its photodegradation product benzoylferrocene. Chapter Three is involved with attempts to bridge dithioferrocenes through the thione sulphur atoms, also discussed is the crystal structure of a by-product of thionation, an unusual 1,2,4-trithiolane compound. Chapter Four describes the spectral characterisation of an unexpected tetra substituted ferrocene. Chapter Five deals with the Flash Vacuum Pyrolysis of acyl ferrocenes, and ferrocenecarboxaldoxime acetate. Chapter Six, finally, describes the investigation of the crystal structure of ferrocenecarboxaldoxime and its existence in α and β forms, within the one crystal.

Abstract.

An effective method for the stabilisation of otherwise unstable thiones is their complexation to $M(CO)_5$ (M= Cr, Mo, W). This complexation led to a very stable material of composition $C_{22}H_{14}CrFeO_5S$, whose crystals are triclinic, space group $P\bar{1}$ (No. 2) with a=9.058(7), b=10.040(7), c=12.568(8) Å, α=113.70(5), β=93.42(6), γ=95.25(6)° and Z=2. The structure was refined from diffractometer data to an R value of 0.0049. The structure was found to be that of pentacarbonyl(thiobenzoylferrocene-S) chromium, in which the $Cr(CO)_5$ fragment is bonded to the sulphur atom of the thioacyl ferrocene. The photodegradation product was found to have a composition of $C_{17}H_{14}FeO$, crystals were monoclinic, space group $P2_1/c$ (No. 14) with a=6.09(6), b=15.145(7), c=14.263(4)Å, β=105.91(1)° and Z=4. The structure was refined to an R value of 0.059, and was found to be benzoylferrocene.

1,1'-Dibenzoylferrocene reacts with tetraphosphorus decasulphide to yield, in addition to the expected 1,1'-bis(thiobenzoyl)ferrocene, a minor, yellow by-product of composition $C_{24}H_{18}FeS_3$, whose crystals are monoclinic, space group $P2_1/n$ with a=11.769(3), b=11.750(4), c=14.835(2)Å, β=98.63(1)°, and Z=4: the structure was found to be that of 1,4-diphenyl-1,4-epithio-2,3-dithia[4](1,1')-ferrocenophane in which the two rings of the ferrocene nucleus are spanned by a 1,2,4-trithiolane ring.

Whereas the reaction of ferrocene with a stoichiometric quantity of $Me_3CCOCl/AlCl_3$ provides the monoacylated product in 87% yield, the use of excess of the acylation reagent leads to simultaneous acylation and alkylation, two products of which have been isolated and characterised by ^1H and ^{13}C NMR. spectroscopy as 1,1,3-tri-t-butyl-3'-(2,2-dimethylpropionyl) ferrocene, $[C_5H_3(CMe_3)_2]$ Fe $[C_5H_3(CMe_3)COCMe_3)]$ and 1,1'-di-t-butyl-3,3'-bis(2,2-dimethylpropionyl)ferrocene, $[C_5H_3(CMe_3)(COCMe_3)]_2Fe$.

Flash Vacuum Pyrolysis of a number of ferrocenes have been carried out and the mono and diacyl ferrocenes were found to display a high degree of thermal stability, most of these being recovered in almost quantitative yields at furnace temperatures up to 700°C. Ferrocenecarboxaldoxime acetate was found to yield cyanoferrocene and acetic acid in a clean reaction in which the cyanoferrocene was uncontaminated with the acetic acid.

Crystals of the low melting form of ferrocenecarboxaldoxime, $(C_5H_5)Fe(C_5H_4CH = NOH)$ are monoclinic, space-group C2/c with a= 26.512(6), b = 12.798 (4), c = 12.855 (2)Å, β = 114.37 (1)°, and Z = 16. The structure was refined from diffractometer data to an R value of 0.054. There are two molecular sites in the asymmetric unit, and both sites contain a disordered mixture of E and Z geometrical isomers. There are no close contacts between the iron atom and the hydroxyl group of the oxime substituent, but rather there is extensive intermolecular hydrogen bonding.

Contents

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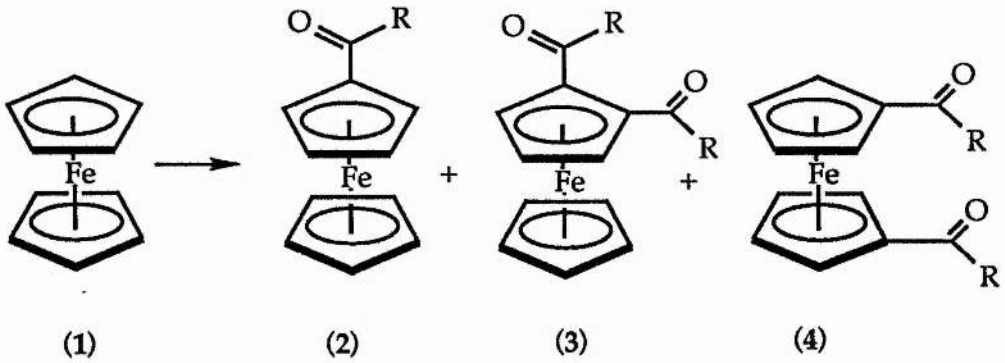
The Crystal Structures were accomplished with the expert guidance of John Barnes in Dundee University, Alan Howie in Aberdeen University and George Ferguson in Guelph University over in Canada.

Chapter One

1.1 Introduction

The chemistry of dicyclopentadienyl iron, given the trivial name ferrocene (**1**), began soon after its initial discovery[1] when Woodward[2] pointed out that the ferrocene system would undergo the Friedel Crafts acylation reaction to give monoacyl ferrocene (**2**) and both isomers (1,1'- and 1,2-) of diacylferrocenes (**3**) and (**4**) (figure 1.1). This was followed by many reports of other aromatic substitution reactions such as alkylation[3], formylation[4], metallation[5], sulphonation[6] and aminomethylation[7].

Since these early discoveries a plethora of new ferrocene derivatives have been prepared[8].



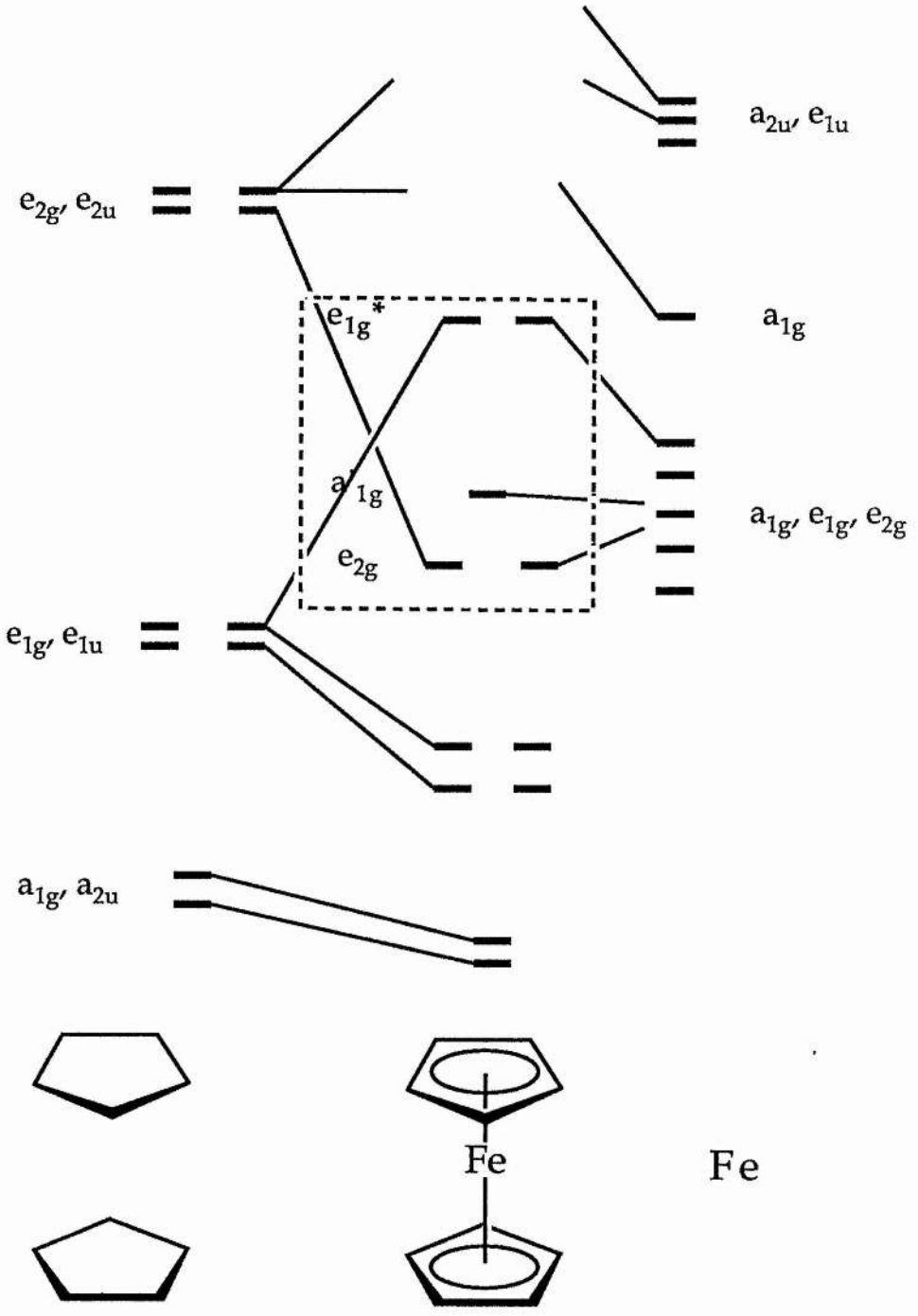
(1) (2) (3) (4)

Figure 1.1

1.1.1 The bonding in ferrocene.

Bonding considerations in the ferrocene molecule are best described using molecular orbital theory. The molecular orbitals of ferrocene can be considered as a combination of Fe(II) and two $C_5H_5^-$ ligand

orbitals. Each carbon in the cyclopentadiene ring, $C_5H_5^-$ of a regular pentagon has a p_z orbital perpendicular to the plane of the ring, and these p_z orbitals form five delocalized ligand group orbitals (LGO), so that the two cyclopentadiene ring form ten LGOs. Scheme 1.1. shows the energy level diagram for metallocenes, computed by Lauher and Hoffmann[11]. In the centre of this region is shown how the new metallocene MOs are formed by the interaction of the cyclopentadiene e_{2g} and a_{1g} orbitals with the metal d-orbitals. For ferrocene with iron in the Fe(II) oxidation state having six d-electrons, the low lying e_{2g} and a_{1g} orbitals are occupied by the six d-electrons. All six electrons will occupy the a_{1g} and e_{2g} orbitals due to the large energy gap between a_{1g} and e_{1g}^* orbitals leaving the antibonding e_{1g}^* orbital unoccupied and giving a diamagnetic complex.



Scheme 1.1
Energy Level diagram for ferrocene showing how the new metallocene orbitals are formed in the centre box.

1.1.2 Thermal Stability of ferrocene.

The bond-dissociation energy of a molecule is the energy required to dissociate it into known fragments in known states. The dissociation energy of the molecule XY, denoted D(XY) can be determined by electron impact studies. If, for example an ion X+ is known to arise by the process ;

$$XY + e \longrightarrow X^+ + Y$$

then the appearance potential is given by the equation,

$$V(X^+) = D(XY) + I(X) + K.E + E.E$$

where I(X) is the ionisation potential of X and K.E is the excess kinetic energy and E.E the excitation energy. Thus the measurement of appearance potential of the ion X leads to the dissociation of the X-Y bond provided that the kinetic energy[12] and excitation energies are known or can be measured.

The appearance potential for the process,

$$(C_5H_5)_2M^+ \longrightarrow C_5H_5M^+ + C_5H_5^{\cdot}$$

for various transition metals M lead to $D(M^+\text{-}C_5H_5)$, although these values should be taken as upper limits since the appearance potentials also include the excess energy due to excited states. The ferrocene system is known for its thermal stability and this is borne out in the bond dissociation values (Table 1.1) derived from measured appearance potentials of a range of metallocenes .

Table 1.1

Bond Dissociation Energies for the process,

$$(C_5H_5)_2M^+ \longrightarrow C_5H_5M^+ + C_5H_5^{\cdot}$$

M	$D(C_5H_5 - M^+)$ (kJmol^{-1})
Mg	311
V	513
Cr	632
Mn	364
Fe	640
Co	752
Ni	524

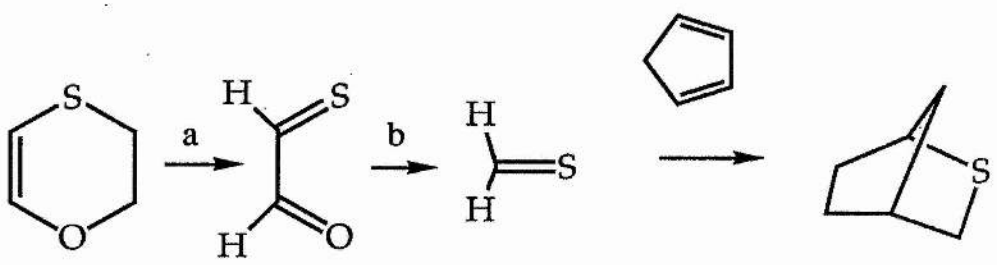
From: M.Cais and M.S.Lupin, *Advances in Organometallic Chemistry* , **8**, (1970), 211.

1.1.3 Flash Vacuum Pyrolysis.

Pyrolysis in the gas phase is achieved by one of two main techniques flow pyrolysis or flash vacuum pyrolysis (FVP). The difference between the two is not very great. In flow pyrolysis the compound is vaporised into a stream of carrier gas, usually nitrogen, which flows through a heated tube at atmospheric or reduced pressure, and the products are collected in a cold trap. The contact time can be regulated by changing the rate of flow of the carried gas. The excitations occurring in such a system are mostly molecule-molecule interactions which permit many unwanted secondary reactions leading to mixtures of products.

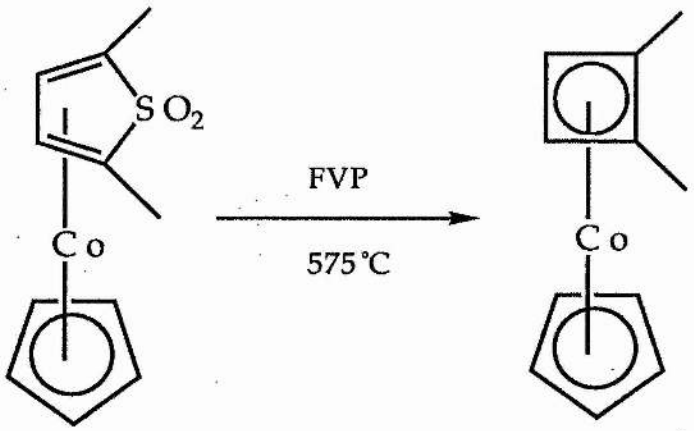
In the flash vacuum pyrolysis technique the sample is simply vaporised under a moderate to high vacuum (10^{-1} - 10^{-5} mmHg) and the vapour passed through a hot evacuated quartz tube before being collected in a cold trap. By using a high vacuum and short tubes the contact times can be very short, and these conditions favour clean unimolecular decompositions and enable the isolation of the primary thermal products uncontaminated with the products of bimolecular reactions. The excitations occurring in such a pyrolytic method are caused by molecule-wall interactions. Because of complications arising from bimolecular reactions the unimolecular thermal chemistry is best studied by taking advantage of the conditions of very high dilution found in the gas phase at low pressures. Bond fissions leading to non-stabilised species generally require much higher temperatures to effect the cleavage: for example methyl iodide requires temperatures in excess of 1100°C to effect the transformation of only 20% to methyl radicals and iodine atoms[14].

When the formation of stable thermal products is achieved in the pyrolysis, the required temperatures are lower. Examples of such pyrolyses are found with the extrusion of small stable molecules such as N_2[16], CS[17], CO(figure 1.2[20]) [18], SO_2(figure 1.3[19], 1.4[23], 1.5[23]), C_2H_4 (figure 1.2[20], 1.4[20])



a) 720°C, $-C_2H_4$
b) 900°C, -CO

Figure 1.2



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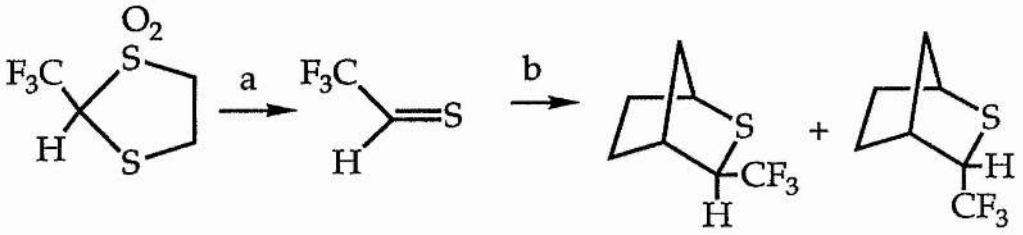


a) FVP, 750°C, $-SO_2$, $-C_2H_4$

b) Cyclopentadiene

Figure 1.4

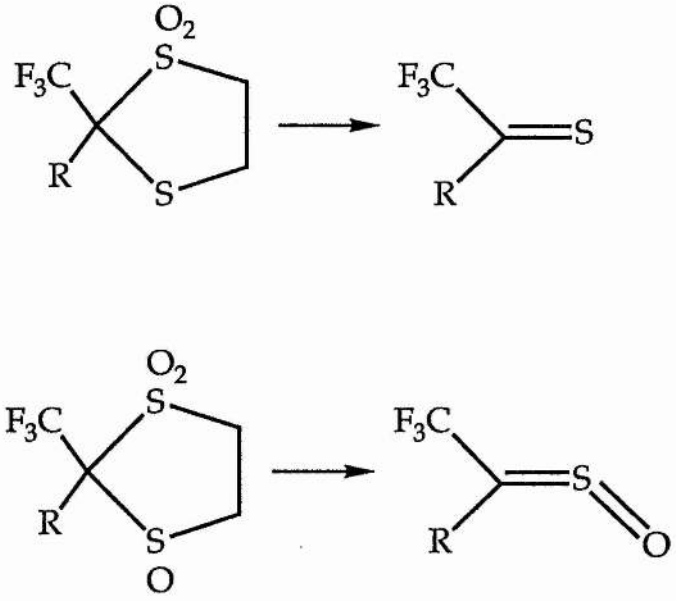


Figure 1.5

and in rearrangements such as the interconversion of diastereoisomers (figure 1.6[21]),

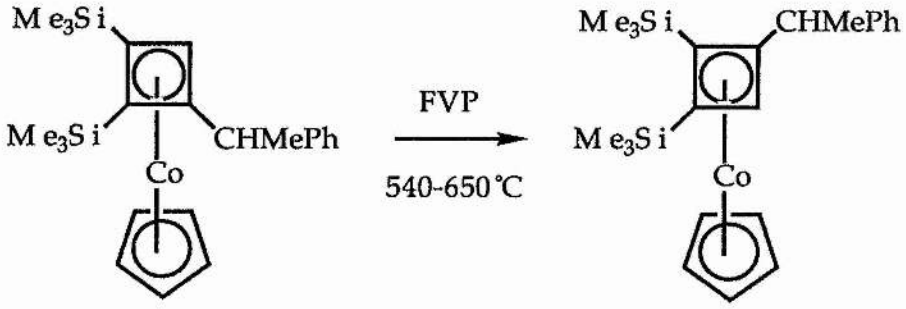


Figure 1.6

in retro-ene reactions (figure 1.7[15]) .Also the thermal elimination of HCl[24], HAc, COS, CH_3SH (figure 1.8[15]) proceed well under FVP conditions.

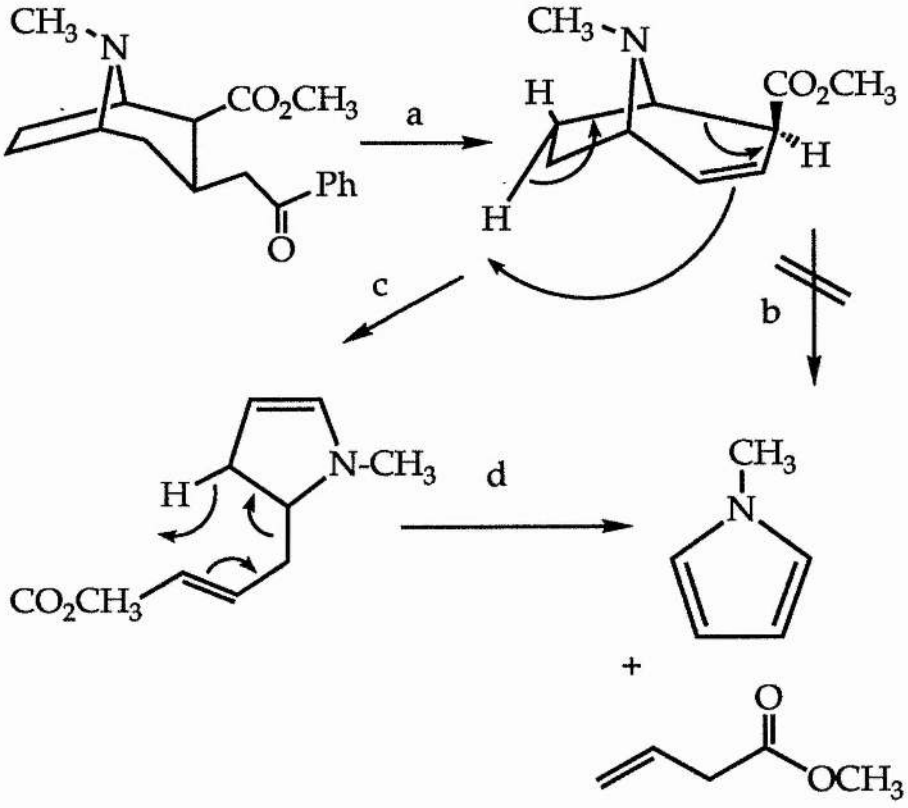


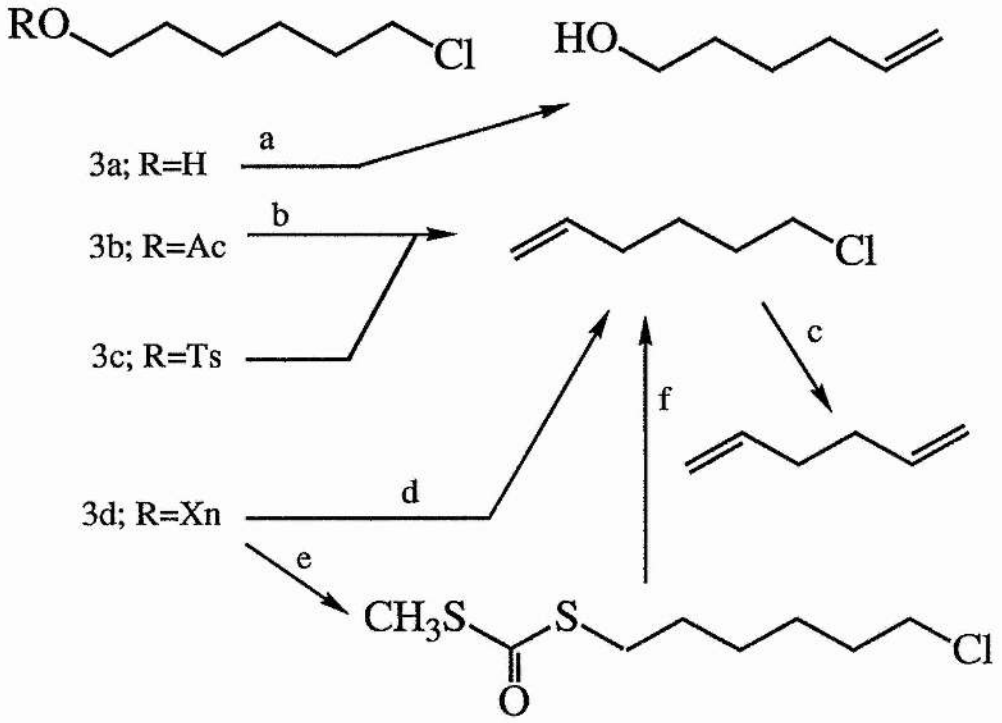
Figure 1.7

a) FVP 550°C, -$PhCO_2H$

b) No retro Diels Alder

c) Retro-ene

d) Retro-ene



Ac=$CH_3(CO)$

Ts = p-$CH_3C_6H_5SO_2$

Xn = $CH_3S(CS)$

a) 700°C, -HCl

b) 450-650°C, -HAc (3b)

" , -HTs (3c)

c) 500-750°C, -HCl

d) 400-525°C, -COS and -CH_3SH

e) 400-575°C

f) 525-650°C, -COS and -CH_3SH

Figure 1.8

The driving force in the process is fission of weak bonds and the formation of strong bonds, this is the common factor to all of the FVP reactions, with the formation of a more thermodynamically stable product with an even number of electrons.

A few energy surfaces[26] of substances have been determined, showing the energy wells and the formation of various intermediates at different temperatures. This is made possible by being able to trap the reactive intermediates and examine these spectroscopically before the onset of secondary reactions.

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Chapter Two
Synthesis and Complexation of monothioacylferrocenes

2.1 Introduction

An effective method for the stabilisation of otherwise unstable thiones is their complexation, via the thione sulphur atom, to a soft, electron-rich metal fragment[1,2,3] A particularly convenient method for the attachment of $M(CO)_5$ fragments (M=Cr, Mo, W) to such thiones has been developed by Pogorzelec and Reid[1]: the thione is treated with $Et_4N[M(CO)_5I]$ and silver nitrate in a two-phase solvent system, in reactions which, it has been suggested, proceed via the transient sixteen-electron intermediates[4,5] $[M(CO)_5]^-$. Here we have applied this complexation reaction to some ferrocenyl thioketones, which are in general photolabile and difficult to crystallise in a manner suitable for X-ray crystallography: complexation to $[M(CO)_5]^-$ provides excellent crystals, and we have determined the crystal and molecular structure of pentacarbonyl(thiobenzoylferrocene-S)chromium, $(C_5H_5)Fe[C_5H_4CPh\{SCr(CO)_5\}]$ (**1**), and of the photodegradation product, benzoylferrocene (**7**).

2.2 Experimental

2.2.1 Preparation of $C_5H_5Fe[C_5H_4CO(R)]$

To 9.30 g of ferrocene (0.05 mol) dissolved in 50 cm^3 dry methylene chloride was added $AlCl_3$ (6.67 g, 0.05 mol) and RCOCl (0.05 mol) in 20 cm^3 methylene chloride dropwise, over 20 minutes. After stirring 24 hours under N_2, the reaction mixture was poured onto ice, washed with water, dried over $CaCl_2$ and reduced to a small volume. Chromatography yielded 1% unreacted ferrocene on elution with light petroleum and typically 85% of the mono acyl ferrocene eluting with 5% ethanol in the eluant.

Crystallisation from light petroleum and methylene chloride yielded 80% red needles. (Table 2.1), The ^1H NMR recorded in (Table 2.2), the ^{13}C NMR in (Table 2.3)

An alternative work up procedure was employed on those preparations which required a large amount of material where chromatography was impractical .

The mixture was reduced to a small volume as before, then poured directly onto a large volume of light petroleum (2l). This was found to have one of two effects , either,

(1) Pure crystalline material was deposited which could be filtered off, or

(2) Black decomposition product precipitated from the solution,the solution was filtered through Hyflo/activated charcoal, and reduced to dryness to yield a red - orange powder.

2.2.2 Thionation of acylferrocenes

In a typical reaction, benzoylferrocene (10 g, 0.034 mol) was dissolved in a mixture of CH_2Cl_2 (20 cm^3) and diethyl ether

(250 cm^3). Sodium hydrogencarbonate (17 g, 0.207 mol) and tetraphosphorus decasulphide (72.3 g, 0.163 mol) were added, and the mixture was heated under reflux for 3 hours. The mixture was then cooled and filtered: the filtrate was reduced to small volume and chromatographed on alumina. Elution with CH_2Cl_2 gave a purple fraction, which on evaporation yielded thiobenzoylferrocene (8.28 g, 79%) as deep purple needles, m.p. 71-72 °C. Found: C, 66.3; H, 4.9: $C_{17}H_{14}FeS$ requires C, 66.7; H, 4.6%.NMR ($CDCl_3$): δ_H 4.18 (s, 5H, C_5H_5); 4.85 (t, 2H) and 5.10 (t, 2H), C_5H_4; 7.2 - 7.8 (m, 5H, C_6H_5). Infra-red: ν_{max}(/cm^{-1}), 1244 (C=S).

In a similar manner were prepared, as deep purple oils, the following:

(a) Thioacetylferrocene: δH 2.87 (s, 3H, CH_3); 4.19 (s, 5H); 4.72 (m, 2H) and 5.03 (m, 2H); ν (C=S), 1289 cm^{-1}.

(b) Thiopropionylferrocene: δH 1.35 (t, J = 7Hz, 3H, CH_3); 3.06 (q, J = 7 Hz, 2H, CH_2); 4.15 (s, 5H); 4.70 (m, 2H) and 5.04 (m, 2H); ν (C=S), 1266 cm^{-1}.

(c) Thio(2–methylpropionyl)ferrocene: δH 1.28 (d, J = 6.5Hz, 6H, CH_3); 3.60 (septet, J = 6.5Hz, 1H, CH); 4.11 (s, 5H); 4.67 (m, 2H) and 5.01 (m, 2H); ν (C=S), 1262 cm^{-1}.

(d) Thio(2,2-dimethylpropionyl)ferrocene: δ H 1.47 (s, 9H, CH_3); 4.12 (s, 5H); 4.65 (m, 2H) and 5.10 (m, 2H), ν (C=S), 1217 cm^{-1}.

2.2.3 Preparation of $(C_2H_5)_4N[Mo(CO)_5I]$

$Mo(CO)_6$ (13.25 g; 50 mmol), $(C_2H_5)_4NI$ (12.75 g; 50 mmol) and 250 cm^3 n-butanol were added together and refluxed under nitrogen for 2 hours. After cooling, the yellow solid was filtered and washed with a little cold hexane. The product was dissolved in 300 cm^3 acetone: addition of 600 cm^3 hexane precipitated out 18.3 g (74%) pure $(C_2H_5)_4N[Mo(CO)_5I]$ as a yellow powder.

2.2.4 Reactions of thiobenzoylferrocene with $[M(CO)_5I]^-$ (M=Cr,Mo, W)

In a typical reaction, a mixture of thiobenzoylferrocene (0.61 g, 2.00 mmol) and tetraethylammonium pentacarbonyliodochromate(0) (1.00 g, 2.20 mmol) was stirred with CH_2Cl_2 (80 cm^3) until a homogeneous solution was formed. Aqueous silver nitrate (8.8 cm^3 of a 0.25 mol dm^{-3} solution; 2.20 mmol) was added to the mixture, with exclusion of light, and the two-phase system was stirred for 24 hours. The organic phase was then separated, dried, and filtered. The CH_2Cl_2 solution was reduced to small volume and chromatographed on silica. Elution with CH_2Cl_2 gave a blue fraction which upon crystallisation yielded pentacarbonyl(thiobenzoylferrocene-S) chromium (**1**) (0.63 g, 66%) as black needles. Found: C, 53.5; H, 2.5: $C_{22}H_{14}CrFeO_5S$ requires C, 53.0; H, 2.8%. Infra-red: ν_{max} (/cm^{-1}), CCl_4 solution; 2061 s, 1988 m, 1950 vs, 1932 s.

In a similar manner were prepared:

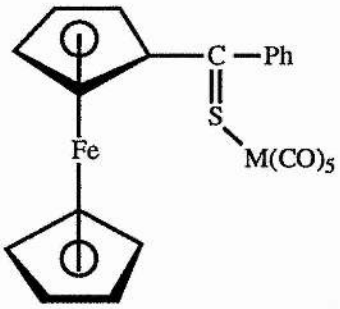
(a) pentacarbonyl(thiobenzoylferrocene-S) molybdenum (**2**) (58%), as black needles; found: C, 49.2; H, 2.5: $C_{22}H_{14}FeMoO_5S$ requires C, 48.6; H, 2.6%. Infra-red: ν_{max}(/cm^{-1}), CCl_4 solution; 2069 s, 1986 m, 1952 vs, 1927 s.

(b) pentacarbonyl(thiobenzoylferrocene-S) tungsten (**3**) (84%), also as black needles; found:C, 41.9; H, 2.4: $C_{22}H_{14}FeO_5SW$ requires C, 41.9; H, 2.2%. Infra-red: $\nu_{max}(/cm^{-1})$), CCl_4 solution; 2068 s, 1981 s, 1945 vs, 1925 s.

2.3 Results and discussion

Acylferrocenes $(C_5H_5)Fe(C_5H_4COR)$ for R = CH_3, C_2H_5, $CH(CH_3)_2$, $C(CH_3)_3$, or C_6H_5 were readily thionated to the thioacyl derivatives $(C_5H_5)Fe(C_5H_4CSR)$ by the use [6] of tetraphosphorus decasulphide in the presence of sodium hydrogen carbonate: after work-up, chromatography on alumina provided the thioacyl ferrocenes as deep purple oils (R = alkyl) or as low melting black-purple needles (R = C_6H_5). The choice of solvent employed is critical to the effectiveness of the thionation procedure for acylferrocenes. When dry benzene, toluene or CH_2Cl_2 was employed, good yields were obtained. However when one of the more polar solvents such as THF or diglyme which have been recommended [6] for such thionations was employed, the yields of thioacylferrocenes were poor: not only was unchanged starting material still present, but other unidentified impurities were also formed under these conditions. With dry solvents of low polarity, practical yields of pure thioacylferrocenes were readily achieved.

These compounds all have low melting points, and are readily photooxidised yielding as one product the parent acylferrocene. In order to form crystalline derivatives suitable for X-ray study, we employed the reaction of $(C_5H_5)Fe(C_5H_4CSPh)$ with $Et_4N[M(CO)_5I]$ and silver nitrate in a two-phase aqueous/CH_2Cl_2 system: work-up of the organic phase, followed by chromatography on silica gave good yields of compounds **(1)** – **(3)**: $(C_5H_5)Fe[C_5H_4CPh\{SM(CO)_5]$ M = Cr **(1)**, Mo**(2)**, and W**(3)**, all readily crystallised from CH_2Cl_2 as black needles.

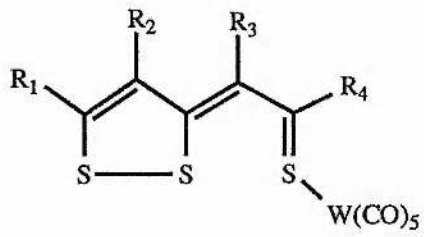


(1) M = Cr

(2) M = Mo

(3) M = W

The infra-red spectra of (1) – (3) in the carbonyl stretching region all exhibit four bands (see Experimental part) whose frequencies and relative intensities follow fairly closely those observed [1] for the tungsten thione complexes (4) – (6):



	R^1	R^2	R^3	R^4
(4)	H	$-(CH_2)_3-$		H
(5)	CH_3	H	H	CH_3
(6)	$C(CH_3)_3$	H	H	H

On the basis of their relative intensities these bands are readily assigned [7], in order of decreasing frequencies, as a_1, b_1, e, and a_1, where the lower frequency a_1 vibration is that primarily associated with the carbonyl ligand *trans* to the thione.

When attempts were made to recrystallise (1) – (3) in strong illumination, the black-purple products were found to be contaminated with small quantity of a second compound (7) crystallising as red needles. Because of the small quantity available, and the excellent crystal habit, X-ray methods were employed to identify (7) as benzoylferrocene $(C_5H_5)Fe(C_5H_4COPh)$.

2.4 X-Ray Crystallography of pentacarbonyl(thiobenzoylferrocene-S) chromium (1)

Crystals suitable for X-ray examination were grown from solutions in CH_2Cl_2/light petroleum. Prior to X-ray analysis, the crystals were re-examined (m.p., microanalysis) to ensure that they were of the same materials as obtained earlier from CH_2Cl_2 alone; they were found to be identical.

2.4.1 Crystal Data Compound (1). $C_{22}H_{14}CrFeO_5S$, M = 498.26, triclinic a = 9.058(7), b = 10.040(7), c = 12.568(8)Å, α = 113.70(5), β = 93.42(6), γ=95.25(6)°, V = 1036.4(12)Å^3, space group $P\bar{1}$ (No. 2), Z = 2, D_C = 1.59 gcm^3, μ(Mo-Kα) = 13.34 cm^{-1}, λ = 0.71069Å, F(000) = 500.

2.4.2 Data Collection Compound (1). A crystal of dimensions 0.20 x 0.32 x 0.76 mm was used. Cell dimensions were determined by least-squares refinement using the setting angles of 25 reflections in the range $12° \leq \theta \leq 16°$. Intensity data were collected at the University of Aberdeen by Dr. Alan Howie, at 22 °C using a Nicolet P3 diffractometer with graphite-monochromated Mo-Kα radiation, in the $\omega/2\theta$ scan mode; ω-scan rate 2.4 - 2.8° min^{-1}; ω-scan width 1.2 - 1.4°; the maximum value of 2θ was 60°.6084 reflections were measured, of which 6083 were unique, and 4945 had $F \geq 2\sigma(F)$. Lorentz and polarisation corrections were made: the data were also corrected for absorption.The internal R value, a guide to the quality of the data, was 0.16% showing the data to be self consistent.

The structure was solved by direct methods, followed by difference Fourier syntheses.

The first SHELX run on the absorption corrected data found the heavy atoms Fe, Cr and S. These were labelled, the structure factor information for Fe and Cr put in and refined isotropically on SHELX 76, The R factor began at 46.47% and fell to 18.90%, when we were able to locate all the non hydrogen atoms.in a difference map. These atoms were labelled and ordered,reflections in which 2s(F)>F were suppressed and all the non-hydrogen atoms refined isotropically. The R factor fell to 10.57%. When the non-hydrogen atoms were allowed to go anisotropic, R fell to 5.61%.

A difference map at this point now showed all the hydrogen atoms which were included in the final refinements with grouped isotropic temperature factors, one for the hydrogen atom on each ring with non unit weights the R factor fell to 4.91%.

For compound (**1**) the weighting scheme $w = 1.3944/[\sigma^2(F) + 0.000508(F^2)]$ gave final R and R_W values of 0.049 and 0.051, with 317 refined parameters. Scattering factor data were taken from refs. [8,9,10]. All calculations were performed on a Prime 6350 computer using SHELX-76 [11], SHELXS [12], and XANADU [13]. Molecular drawings were made with PLUTO [14].

Final refined coordinates for compound (**1**) are given in table 2.4, and selected bond lengths and angles in tables 2.5 and 2.6: a perspective view, showing the atom numbering scheme is in Figure 2.1

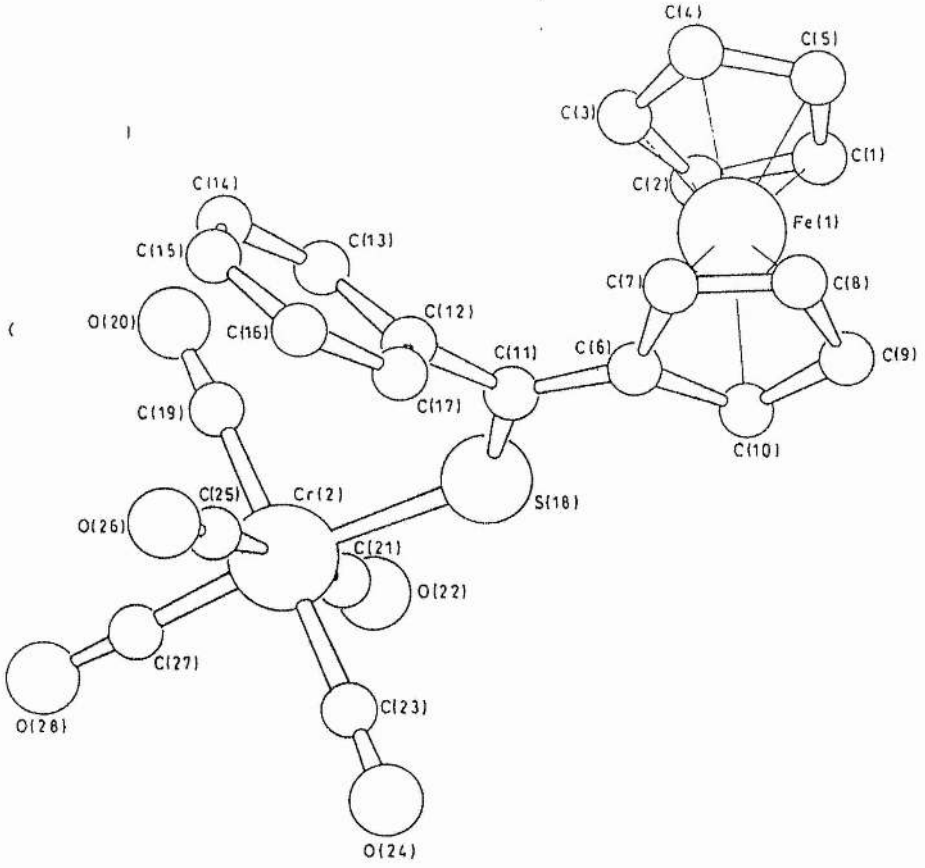


Figure2.1. Perspective view of the molecule of pentacarbonyl(thiobenzoylferrocene–S)chromium (1), showing the atom numbering scheme.

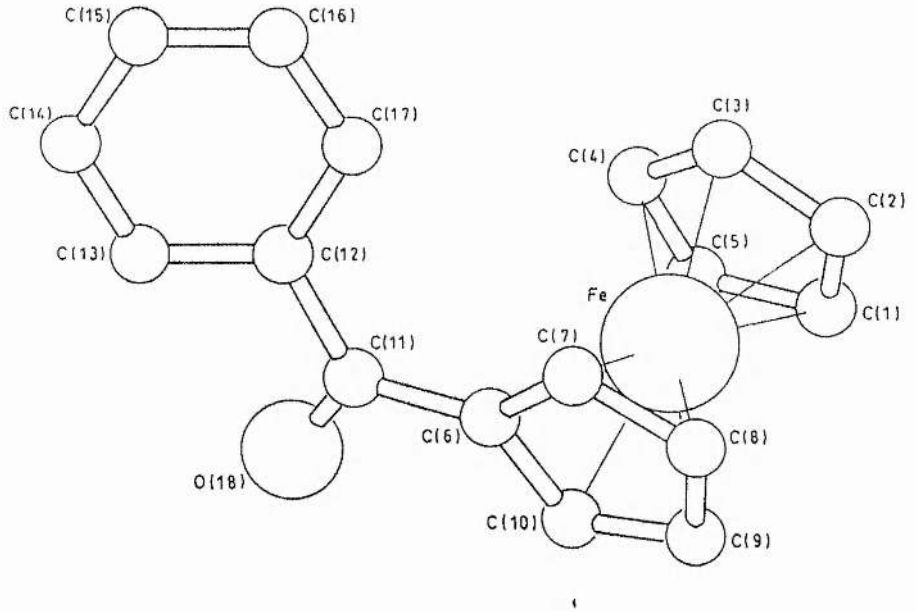


Figure 2.2. Perspective view of the molecule of benzoylferrocene, (7), showing the atom numbering scheme.

Tables of refined hydrogen coordinates are in appendix 2.1, anisotropic temperature factors for non-hydrogen atoms in appendix 2.2, and observed and calculated structure factors are in in appendix 2.3.

2.4.4 Crystal and Molecular Structure

The structure comprises isolated molecules in which a $Cr(CO)_5$ fragment of approximate C_{4v} local symmetry is bonded to the sulphur atom of the thioacylferrocene. The Cr(2)–S(18)–C(11) bond angle is 121.9(1)° and the Cr(2)–S(18) bond is almost eclipsed by the C(11)–C(12) bond (dihedral angle 10.2°) (see Figure 2.1 for atom-numbering scheme). In a similar fashion, the C(11)–S(18) bond is almost eclipsed by the Cr(2)–C(25) bond (dihedral angle 18.8°). This conformation of the $Cr(CO)_5$ fragment relative to the thioacylferrocene fragment leads to some close non-bonded contacts between O(26) and C(16), 3.31Å and between O(26) and C(17), 3.32Å: these are both only a little greater than the sum, 3.2Å of the van der Waals' radii [15]. These close contacts are presumably responsible for the small but significant bending of the Cr(2)–C(25)–O(26) fragment from linearity : the Cr–C–O bond angle here is 173.8(3)°, compared with a mean bond angle for the other three equatorial ligands of 177.6° and an axial bond angle of 177.5°. Within the $Cr(CO)_5$ fragment the axial Cr–C and C–O bond lengths are 1.850(4) and 1.150(5)Å respectively, compared with the mean values for the equatorial bonds of 1.908Å and 1.134Å respectively. These values are consistent with much stronger metal-ligand π-bonding at the axial carbonyl ligand than at the equatorial carbonyls, confirming that thione ligands compete very poorly with carbonyls for metal π-electron density [2,16,17].

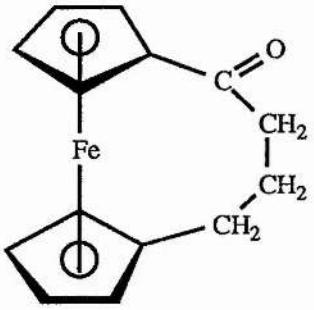
The Cr–S distance observed here, 2.412(1)Å, lies almost mid-way between the values observed in $Cr(CO)_5SCMe_2$, 2.377(4)Å [16], and in $Cr(CO)_5S(Et)CH_2Ph$, 2.458(2)Å [18], although it is very much shorter than the value, 2.510(2)Å found for $Cr(CO)_5SPMe_3$ [17]. The S=C bond length, S(18)–C(11) of 1.667(2)Å, while essentially identical to that found [2] in (**4**), 1.68(1)Å, is significantly longer that the corresponding bond length, 1.618(8)Å in $Cr(CO)_5SCMe_2$ [16]. These differences may be rationalised in terms of the electronic character of the substituents in the thione fragment. In both (**1**) and (**4**) the thione has one extremely electron rich substituent, the ferrocenyl group in (**1**) and the dihydro–4*H*–benzodithiole group in (**4**): such groups readily stabilise the polar form (**8b**) at the expense of the doubly-bonded form (**8a**) of the thione.

(**8a**) (**8b**)

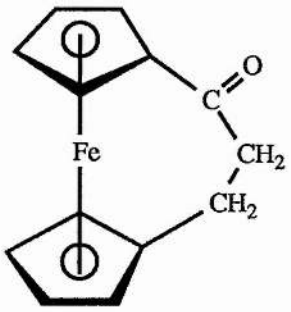
Consequently, in the presence of such electron donor substituents R^1,R^2 it may be expected that in complexes $R^1(R^2)CSM(CO)_5$ the unique C–S bond is longer than when $R^1 = R^2 = CH_3$: such thiones will also be poorer π-acceptors from $M(CO)_5$ than $(CH_3)_2CS$, so that the M–S bonds will likewise be longer when R^1, R^2 are electron donors.

In the ferrocenyl fragment, the mean Fe–C distances for the two independent rings are identical, within experimental uncertainty: however the mean C–C distances within the two cyclopentadienyl rings are significantly different. In the

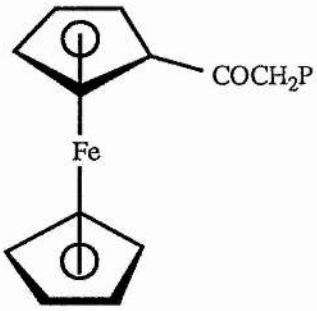
unsubstituted ring the mean C–C distance is 1.387Å, and in the substituted ring it is 1.423Å. Such a difference between rings carrying or not carrying an acyl substituent has been observed previously in (**9**)[19], although not in (**10**)[20] or (**11**)[21].



(**9**)



(**10**)



(**11**)

2.5 X-Ray Crystallography of Benzoyl Ferrocene

A crystal of dimensions 0.42 x 0.17 x 0.13 mm was used. When attempts were made to recrystallise (**1**) – (**3**) in strong illumination, the black-purple products were found to be contaminated with small quantity of a second compound crystallising as red needles. Because of the small quantity available, and the excellent crystal habit, X-ray methods were employed to identify this as benzoylferrocene $(C_5H_5)Fe(C_5H_4COPh)$ (**7**).

2.5.1 Crystal Data

$C_{17}H_{14}FeO$, M=290.15, monoclinic a = 6.09(6), b = 15.145(7), c = 14.263(4)Å, β = 105.9(1)°, V = 1265.2(5)Å^3, space group P $2_1/c$ (No. 14), Z = 4, D_c = 1.52 g cm^{-3}, μ(Mo - K_α) = 11.10 cm^{-1}, λ = 0.71069 Å, F(000) = 592.

2.5.2 Data Collection

Intensity data were collected at 22 °C using a STADI-2 diffractometer with graphite-monochromated Mo - K_α radiation in the $\omega/2\theta$ scan mode: the maximum value of 2θ was 50° The data was collected in layers 0,1, 2a, 2b, 3, 4 and 5, these were merged, the space group determined as $P2_1/c$ (No. 14), the internal R was equal to 30.40% at this stage and after filtering out some bad data on layers 0 and 2, R(int) was brought down to 2.72% for 1983 unique reflexions.
Of the 1983 reflections 1837 were unique, and 1652 had $F \geq 3\sigma$ (F). Lorentz and polarisation corrections, but no absorption corrections, were made..

2.5.3 Structure Solution and Refinement The structure was solved by direct methods, followed by difference Fourier syntheses.
The first SHELX run on the absorption corrected data found all the non hydrogen atoms. These were labelled, the structure factor information for Fe put in and the non hydrogen atoms refined isotropically on SHELX 76, The R factor began at 73.36% and fell to 50.33% after three cycles of least squares refinement. Reflections with $2\sigma(F)>F$ were suppressed and weight cards added, the R factor fell to 11.85 %. The 19 found non-hydrogen atoms were allowed to go anisotropic.The R factor fell to 8.38% and the difference map located all of the H atoms. Found hydrogens were put in and the R factor fell to 7.69%.
A weight card was inserted , bringing R to 6.76%. Finally two bad data were removed.

The weighting scheme $w = 2.7310/[\sigma^2(F) + 0.000608\ F^2]$ gave final R and R_w values of 0.059 and 0.069, with 179 refined parameters.

Scattering factor data were taken from refs.[8,9,10]. All calculations were performed on a Prime 6350 computer using SHELX-76[11], SHELXS[12], and XANADU[13]. Molecular drawings were made with PLUTO[14].

Final refined coordinates for benzoylferrocene are given in table 2.7, selected bond lengths and angles in tables 2.8 and 2.9 respectively,and a perspective view, showing the atom numbering scheme is in figure 2.2.

Tables of refined hydrogen coordinates in appendix 2.4, anisotropic temperature factors for non-hydrogen atoms in appendix 2.5, and observed and calculated structure factors are iin appendix 2.6.

2.5.4 Crystal and Molecular Structure

The structure of benzoylferrocene presents few unusual features. We note however that, as usual, the mean Fe–C distances for the two independent cyclopentadienyl rings are identical within experimental uncertainty: likewise there is no significant difference between the mean C–C distances in the substituted ring, 1.410Å, and the unsubstituted ring, 1.399Å. The structure thus resembles in this respect those of (**10**) and (**11**), rather than those of (**1**) and (**9**): no reason is obvious for this dichotomy of behaviour.

Table 2.1 Preparations of Mono acyl ferrocenes.

Derivative [a]	ν (C=O) [b] (cm^{-1})	m.pt. (°C)	analysis Theory	analysis Found
FcCOH	1687	130-132	61.73 %C 4.71 %H	62.18 %C 4.92 %H
$FcCOCH_3$	1676	82-83	63.20 %C 5.30 %H	63.59 %C 5.33 %H
$FcCOCH_2CH_3$	1677	37-38	64.50 %C 5.83 %H	64.87 %C 5.85 %H
$FcCO\ CH(CH_3)_2$	1669	15-16	65.65 %C 6.30 %H	65.54 %C 6.53 %H
$FcCOC(CH_3)_3$	1661	82.5	66.69 %C 6.72 %H	66.57 %C 6.78 %H
$FcCOC_6H_5$	1646	108-109	70.37 %C 4.86 %H	70.31 %C 4.89 %H
$FcCOCH_2C_6H_5$	1668	125-126	71.08 %C 5.30 %H	71.04 %C 5.32 %H

a/ Fc is $C_5H_5FeC_5H_4$-

b/
Infra red spectra were recorded in CCl_4 solutions.

Table 2.2 ^{1}H NMR Data for Mono acyl ferrocenes in ppm.[a]

Derivative[b]	Associated R group	Ferrocenyl group unsub.	sub.	
$FcCOCH_3$	2.38 (s,3H,CH_3)	4.2	4.5	4.75
$FcCOCH_2CH_3$	1.20 (t,3H,CH_3) 2.68 (q,2H,CH_2)	4.18	4.48	4.78
$FcCOCH(CH_3)_2$	1.20 (d,6H,$(CH_3)_2$) 3.13 (sept.,1H,CH)	4.20	4.50	4.80
$FcCOC(CH_3)_3$	1.33 (s,9H,$(CH_3)_3$)	4.20	4.45	4.85
$FcCOC_6H_5$	7.45, 7.85 (m,5H,C_6H_5)	4.15	4.55	4.90
$FcCOCH_2C_6H_5$	7.35 (m,5H,C_6H_5) 3.95 (s,2HCH$_2$)	4.10	4.50	4.80

a/ All spectra were recorded in $CDCl_3$, with TMS as internal standard, at 20 °C.

b/ Fc is $C_5H_5FeC_5H_4$-

Table 2.3 ^{13}C NMR Data for Mono acyl ferrocenes in ppm[a]

Derivative[b]	C=O	quaternary C on Cp ring	C-H on Cp ring subs. Ha	Hb	unsubs	R group
FcCOH	193.1	79.2	73.1	69.5	69.5	——
$FcCOCH_3$	201.6	79.0	72.2	69.4	69.6	27.3 ($\underline{C}H_3$)
$FcCOCH_2CH_3$	204.0	78.8	72.0	69.1	69.6	32.6 ($\underline{C}H_2CH_3$) 8.4 ($CH_2\underline{C}H_3$)
$FcCOCH(CH_3)_2$	208.3	78.0	72.1	69.3	69.5	37.1 ($\underline{C}H(CH_3)_2$) 19.5 ($CH(\underline{C}H_3)_2$)
$FcCOC(CH_3)_3$	210.1	76.7	71.1	70.9	69.7	44.1 ($\underline{C}(CH_3)_3$) 28.1 ($C(\underline{C}H_3)_3$)
$FcCOC_6H_5$	198.7	77.9	72.5	71.3	70.1	139.6 128.1 131.3 127.9 } $\underline{C}$-H, of C_6H_5
$FcCOCH_2C_6H_5$	201.7	78.7	72.3	69.7	69.8	135.2 128.4 129.3 126.7 } $\underline{C}$-H, of C_6H_5 46.8 ($\underline{C}H_2$)

a/ All spectra were recorded in $CDCl_3$ at 20° C, with TMS as internal standard.

b/ Fc is $C_5H_5FeC_5H_4$-

Table 2.4

Compound (1). Coordinates for non-hydrogen atoms x 10^4 with e.s.d.'s in parentheses. U_{eq} x 10^3

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* a_i . a_j$$

	x/a	y/b	z/c	U_{eq}
Fe1	2389 (1)	6469 (1)	2006 (1)	35 (1)
Cr2	8002 (1)	1038 (1)	2167 (1)	36 (1)
C1	2484 (5)	4862 (4)	385 (3)	69 (1)
C2	2063 (4)	6101 (5)	280 (3)	72 (1)
C3	3203 (5)	7246 (4)	860 (3)	76 (1)
C4	4318 (4)	6730 (5)	1309 (3)	77 (1)
C5	3891 (5)	5252 (5)	1026 (3)	78 (1)
C6	1178 (3)	7900 (2)	3159 (2)	33 (1)
C7	2598 (3)	7864 (3)	3724 (2)	39 (1)
C8	2655 (3)	6421 (3)	3622 (2)	48 (1)
C9	1319 (3)	5546 (3)	2990 (3)	49 (1)
C10	407 (3)	6430 (3)	2684 (2)	41 (1)
C11	712 (2)	9140 (2)	2985 (2)	31 (1)
C12	1663 (2)	10569 (3)	3583 (2)	35 (1)
C13	2276 (3)	11300 (3)	2950 (3)	47 (1)
C14	3147 (4)	12658 (4)	3532 (4)	64 (1)
C15	3367 (4)	13271 (4)	4720 (4)	76 (1)
C16	2783 (4)	12568 (4)	5362 (3)	68 (1)
C17	1940 (3)	11181 (3)	4805 (2)	49 (1)
S18	-888 (1)	8950 (1)	2180 (1)	41 (1)
C19	9431 (3)	1515 (3)	1292 (2)	43 (1)
O20	10245 (3)	1828 (3)	751 (2)	65 (1)
C21	6915 (3)	-280 (3)	726 (3)	49 (1)
O22	6279 (3)	-1022 (3)	-149 (2)	76 (1)
C23	6542 (3)	551 (3)	3033 (2)	44 (1)
O24	5664 (2)	287 (3)	3548 (2)	68 (1)
C25	9077 (3)	2458 (3)	3598 (2)	42 (1)
O26	9637 (3)	3385 (2)	4426 (2)	61 (1)
C27	6963 (3)	2534 (3)	2154 (3)	50 (1)
O28	6352 (3)	3500 (3)	2179 (2)	79 (1)

Table 2.5

Compound (**1**). Bond distances (Å)

Fe(1)–C(1)	2.041(3)	Fe(1)–C(6)	2.042(2)
Fe(1)–C(2)	2.047(4)	Fe(1)–C(7)	2.032(2)
Fe(1)–C(3)	2.037(5)	Fe(1)–C(8)	2.051(3)
Fe(1)–C(4)	2.042(4)	Fe(1)–C(9)	2.056(4)
Fe(1)–C(5)	2.038(4)	Fe(1)–C(10)	2.036(3)
C(1)–C(2)	1.386(7)	C(6)–C(7)	1.442(4)
C(2)–C(3)	1.393(5)	C(7)–C(8)	1.409(4)
C(3)–C(4)	1.367(7)	C(8)–C(9)	1.413(4)
C(4)–C(5)	1.390(7)	C(9)–C(10)	1.409(5)
C(1)–C(5)	1.400(6)	C(6)–C(10)	1.441(3)
C(6)–C(11)	1.442(4)	Cr(2)–C(19)	1.896(3)
C(11)–C(12)	1.482(3)	Cr(2)–C(21)	1.904(3)
C(12)–C(13)	1.389(5)	Cr(2)–C(23)	1.911(3)
C(13)–C(14)	1.395(4)	Cr(2)–C(25)	1.921(2)
C(14)–C(15)	1.360(7)	Cr(2)–C(27)	1.850(4)
C(15)–C(16)	1.367(7)	C(19)–O(20)	1.138(4)
C(16)–C(17)	1.404(4)	C(21)–O(22)	1.133(3)
C(12)–C(17)	1.403(4)	C(23)–O(24)	1.132(4)
C(11)–S(18)	1.667(2)	C(25)–O(26)	1.130(3)
S(18)–Cr(2)	2.412(1)	C(27)–O(28)	1.150(5)

Table 2.6

Compound (**1**). Selected bond angles (°)

C(1)–C(2)–C(3)	107.6(4)	C(12)–C(13)–C(14)	119.9(3)
C(2)–C(3)–C(4)	108.8(4)	C(13)–C(14)–C(15)	119.7(4)
C(3)–C(4)–C(5)	108.2(3)	C(14)–C(15)–C(16)	121.4(3)
C(4)–C(5)–C(1)	107.7(4)	C(15)–C(16)–C(17)	120.5(4)
C(5)–C(1)–C(2)	107.7(3)	C(16)–C(17)–C(12)	118.3(3)
C(6)–C(7)–C(8)	107.7(2)	C(17)–C(12)–C(13)	120.1(2)
C(7)–C(8)–C(9)	108.8(3)	C(11)–S(18)–Cr(2)	121.9(1)
C(8)–C(9)–C(10)	108.7(3)	S(18)–Cr(2)–C(19)	94.8(1)
C(9)–C(10)–C(6)	107.7(2)	S(18)–Cr(2)–C(21)	86.5(1)
C(10)–C(6)–C(7)	107.0(2)	S(18)–Cr(2)–C(23)	85.5(1)
C(7)–C(6)–C(11)	125.7(2)	S(18)–Cr(2)–C(25)	96.6(1)
C(10)–C(6)–C(11)	126.8(2)	S(18)–Cr(2)–C(27)	174.1(1)
C(6)–C(11)–C(12)	117.8(2)	Cr(2)–C(19)–O(20)	177.1(3)
C(6)–C(11)–S(18)	119.9(2)	Cr(2)–C(21)–O(22)	177.2(4)
C(12)–C(11)–S(18)	122.3(2)	Cr(2)–C(23)–O(24)	178.6(3)
C(11)–C(12)–C(13)	120.9(2)	Cr(2)–C(25)–O(26)	173.8(3)
C(11)–C(12)–C(17)	119.9(3)	Cr(2)–C(27)–O(28)	177.5(3)

Table 2.7

Compound (7). Coordinates for non-hydrogen atoms x 10^4 with e.s.d.'s in parentheses.U_{eq} x 10^3

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* a_i . a_j$$

	x/a	y/b	z/c	U_{eq}
Fe1	3557 (1)	4596 (1)	2330 (1)	37 (1)
C1	3614 (13)	3289 (4)	2015 (4)	64 (2)
C2	5629 (11)	3680 (4)	1989 (4)	58 (2)
C3	5196 (11)	4355 (4)	1294 (4)	55 (2)
C4	2866 (11)	4376 (4)	861 (4)	54 (2)
C5	1825 (11)	3725 (4)	1297 (5)	64 (2)
C6	2223 (9)	5766 (3)	2581 (3)	38 (1)
C7	4614 (9)	5785(3)	2931 (3)	40 (1)
C8	5318 (9)	5117 (3)	3642 (4)	41 (1)
C9	3385 (9)	4673 (3)	3735 (4)	46 (1)
C10	1467 (9)	5059 (4)	3095(4)	42 (1)
C11	643 (9)	6325 (3)	1873 (4)	47 (1)
C12	1392 (8)	6928 (3)	1194 (3)	37 (1)
C13	35 (9)	7656 (3)	848 (4)	50 (1)
C14	592 (11)	8223 (4)	192 (4)	60 (2)
C15	2446 (11)	8054 (4)	-145 (4)	56 (2)
C16	3784 (9)	7337 (3)	193 (4)	47 (1)
C17	3271 (8)	6772 (3)	868 (4)	41 (1)
O18	-1346 (7)	6318 (3)	1851 (4)	81 (1)

Table 2.8

Compound (7). Bond distances (Å)

Fe(1)–C(1)	2.032(7)	Fe(1)–C(6)	2.022(6)
Fe(1)–C(2)	2.022(8)	Fe(1)–C(7)	2.023(6)
Fe(1)–C(3)	2.031(9)	Fe(1)–C(8)	2.042(6)
Fe(1)–C(4)	2.049(7)	Fe(1)–C(9)	2.038(7)
Fe(1)–C(5)	2.043(8)	Fe(1)–C(10)	2.015(8)
C(1)–C(2)	1.372(10)	C(6)–C(7)	1.405(7)
C(2)–C(3)	1.398(8)	C(7)–C(8)	1.413(7)
C(3)–C(4)	1.384(9)	C(8)–C(9)	1.393(8)
C(4)–C(5)	1.406(9)	C(9)–C(10)	1.398(7)
C(5)–C(1)	1.436(8)	C(10)–C(6)	1.443(8)
C(12)–C(13)	1.384(7)	C(6)–C(11)	1.460(7)
C(13)–C(14)	1.380(9)	C(11)–C(12)	1.491(8)
C(14)–C(15)	1.366(10)	C(11)–O(18)	1.203(7)
C(15)–C(16)	1.364(8)		
C(16)–C(17)	1.387(8)		
C(17)–C(12)	1.369(8)		

Table 2.9

Compound (7). Selected bond angles (°)

C(1)–C(2)–C(3)	109.8(5)	C(6)–C(7)–C(8)	108.7(5)
C(2)–C(3)–C(4)	107.8(6)	C(7)–C(8)–C(9)	108.5(4)
C(3)–C(4)–C(5)	108.5(5)	C(8)–C(9)–C(10)	108.2(5)
C(4)–C(5)–C(1)	107.0(6)	C(9)–C(10)–C(6)	108.4(5)
C(5)–C(1)–C(2)	107.0(6)	C(10)–C(6)–C(7)	106.2(4)
C(7)–C(6)–C(11)	131.1(5)	C(12)–C(13)–C(14)	120.2(6)
C(10)–C(6)–C(11)	122.7(5)	C(13)–C(14)–C(15)	120.1(5)
C(6)–C(11)–C(12)	122.9(5)	C(14)–C(15)–C(16)	119.7(6)
C(6)–C(11)–O(18)	118.8(6)	C(15)–C(16)–C(17)	120.7(6)
C(12)–C(11)–O(18)	118.3(5)	C(16)–C(17)–C(12)	119.8(5)
C(11)–C(12)–C(13)	117.4(5)	C(17)–C(12)–C(13)	119.3(5)
C(11)–C(12)–C(17)	123.2(4)		

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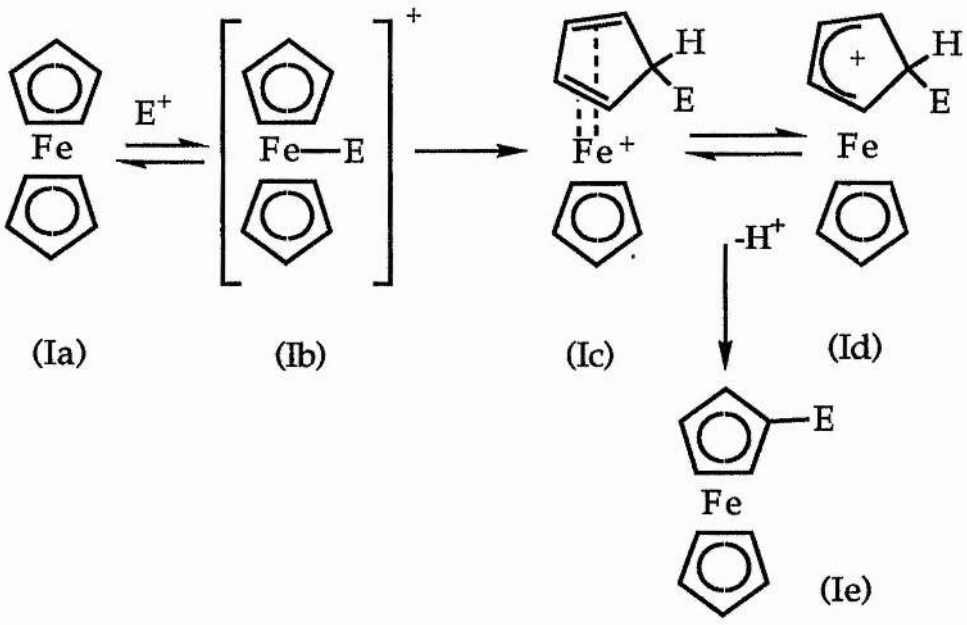
Chapter Three
Ferrocenyl-1,1′-diketones

3.1 Introduction

After the discovery of ferrocene in 1952 it was quickly pointed out that it would undergo the Friedel Crafts reaction[1,2]. The reaction of acetyl chloride, $AlCl_3$ and ferrocene was shown to yield the monoacetyl, 1,1′-diacetyl, and 1,2-diacetyl ferrocenes. Under appropriate conditions an almost quantitative yield of either the mono- or the 1,1′-diacetyl derivative can be achieved.

Ferrocene consists of two cyclopentadienyl rings rotating essentially freely about an axis of symmetry through the iron atom. The same rotation is found in mono-substituted and di-substituted ferrocenes, and this ease of rotation precludes the possibility of rotameric isomers[3].

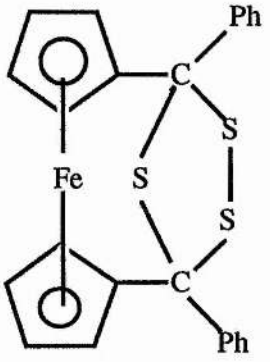
When ferrocene reacts with a stoichiometric quanty of the $AlCl_3$/RCOCl complex, a product which is exclusively mono-substituted[2,3,4] is recovered. Only when $AlCl_3$ is used in excess is the di-acyl ferrocene produced[4,5]. These observations have been accounted for by a general mechanism in which the iron atom is the primary site for electrophilic attack... The mechanism has been proposed[6,7,8] ,to follow the initial electrophilic attack of the iron atom by the $AlCl_3$/RCOCl complex (**Ib**), followed by a rate-limiting rearrangement to a σ-bonded complex (**Ic**) which loses a proton yielding the mono-substituted acylferrocene (**Ie**),



An explanation why only mono-substitution is observed with stoichiometric quantities of ferrocene and electrophile invokes the effective removal of ferrocene from the reaction by formation of a stable, non-acylable, σ-bonded complex. However, with the $AlCl_3$ in excess , the reaction proceeds further, leading to the symmetrical 1,1′-disubstituted ferrocene. This symmetrical derivative predominates over the two alternative homoannular 1,2- and 1,3-isomers, although significant amounts of 1,2-isomers have been reported[2,9,10]. The 1,3-isomer has not been reported as the result of a Friedel-Crafts reaction, but it has been synthesized indirectly [11].

Thionation of 1,1'-diacylferrocenes with tetraphosphorus decasulphide is generally straightforward, except that the deep purple-black bis(thioacyl) derivatives $Fe(C_5H_4CSR)_2$ are sometimes accompanied with very small quantities of a pale yellow by-product. For the case of R = Ph, this by-product has now been isolated and purified , albeit in yields of less than 1%, and has been

characterised analytically, spectroscopically, and crystallographically as 1,4-diphenyl-1,4-epithio-2,3-dithia[4](1,1')ferrocenophane, **(2)**, which contains the rather uncommon 1,2,4-trithiolane ring.



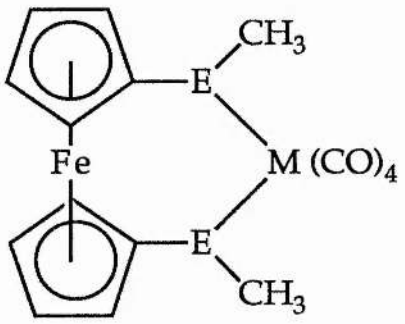
(2)

Tetraphosphorus decasulphide was found to facilitate complete reaction in 1 hour at room temperature using an aprotic solvent. Polar solvents have been reported[12] to give both enhanced rates and yields; however the use of these polar solvents produced mixtures of unidentified products with incomplete reaction of the starting material, and overall poor yields of the thiones.

The di-thiones were found to be photo-labile oils which decomposed over a short period, as has been observed with mono(thioacyl)ferrocenes[13].which were stabilised by complexation to the $[M(CO)_5]$ fragment.

In the hope that the dithione could be bridged by the use of $M(CO)_4$ by reaction with $Mo(CO)_4$(2,5-norbornadiene), the dithione was treated with the molybdenum complex but no reaction was apparent. Compounds of similar type have been observed recently by Abel and co-workers[14] who reported the crystal structure of $Fe(C_5H_4SeCH_3)_2W(CO)_4$ where the $W(CO)_4$ acted as a

bridge between the two seleniums of the ferrocenyl ligand(Figure 3.1), but here the sulphur atoms are of sulphide type rather than of the thione type .



M=Cr, Mo, W

E=S, Se

Figure 3.1

We attempted to use a dithione as the ligand and either $Mo(CO)_4$ or $Mo(CH_3CN)(CO)_3$, as the bridge(Figure 3.2). With neither $Mo(CO)_4$(norbornadiene) nor $Mo(CH_3CN)(CO)_3$,did any reaction take place.

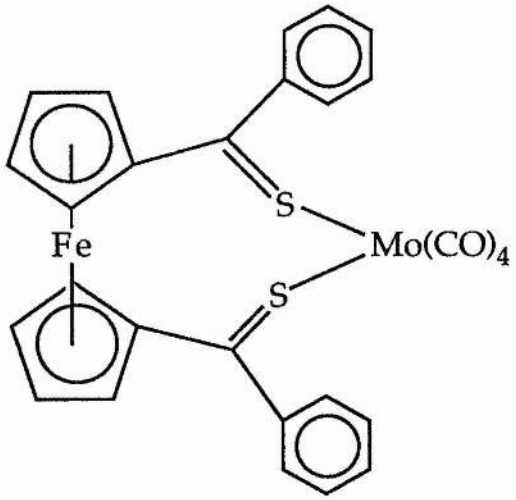
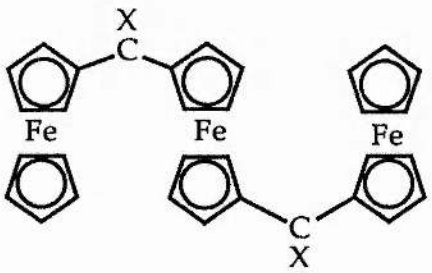


Figure 3.2

In the hope of observing fluxional behaviour (to be followed by VT-NMR) a dithione was reacted with one mole of $[Mo(CO)_5I]^-$ in order to prepare a mono-substituted dithione, which might exhibit exchange of the $M(CO)_5$ unit. However a pure sample could not be obtained for analysis.

Through handling these mono- and di- thioacyl ferrocenes it was noticed that the di-substituted derivatives were generally more stable than their mono-substituted analogues, and this stability became more pronounced as the R-group exerted a greater electron donating effect upon the adjacent thione group, thus $CH_3<C_6H_5<C_6H_4CH_3$. As an extention of this it was decided to prepare a dithioacylferrocene with an R-group capable of exerting a much greater electron donating effect than previously attempted. The use of larger aromatics naphthalene, anthracene and so on were rejected on the grounds that these may lead to unwanted polymerised products. The R-group of choice was ferrocene itself, and so the target molecule was $Fcd(CSFc)_2$ **(3b)**.



3a X=O

3b X=S

These preparations were achieved by literature methods. Ferrocene was di-lithiated and converted to the di-carboxylic acid[15]

then to the di-acid chloride[16] followed by a Friedel- Crafts reaction with ferrocene[17], to yield **(3a)**, which has been reported previously as a minor side-product which was separated only after extensive chromatography[18,19,20] from a reaction mixture containing many components, and finally conversion to the thioketone, Unfortunately the thioketone **(3b)** proved to be unstable, attempts to purify the material failed due to extensive decomposition in solution over a short period.

3.2 Experimental

3.2.1 Preparation of $Fe[C_5H_4CO(C_6H_4CH_3\text{-}p)]_2$.

Ferrocene (7g; 0.038 mol) was dissolved in 100 cm^3 methylene chloride and the solution was added dropwise to a stirred mixture of $AlCl_3$ (20.2g;0.152 mol) and p-toluyl chloride (23.6g;0.152 mol) in 500 cm^3 methylene chloride . After 12 hours a t.l.c analysis showed only one component. The mixture was hydrolysed with ice and the organic fraction was washed with water, dried and reduced in volume to 100 cm^3. The brown solution was poured slowly onto 2l swirling light petroleum; this precipitated out black decomposition product, which was filtered off using Hyflo. The filtrate was then reduced to dryness to give 15.8g (99%) 1,1'-di-p-toluoylferrocene as a red powder. This was recrystallised from methylene chloride/light petroleum to give red needles mpt. 170-171°C ν(CO) 1645cm^{-1} (CCl_4 solution).

A range of diacylferrocenes were prepared similarly (table 3.1): the ^1H NMR data are recorded in table 3.2, and the ^{13}C NMR data in table 3.3.

3.2.2 Preparation of $Fe[C_5H_4CS(C_6H_4CH_3\text{-}p)]_2$

To $Fe[C_5H_4CO(C_6H_4CH_3\text{-}p)]_2$ (2g; 4.74 mol) was added P_4S_{10} (50g; 0.11 mol) , 200 cm^3 methylene chloride and 50 cm^3 diethyl ether. The mixture was stirred for 2 hours , and then filtered through Hyflo. The filtrate was reduced to a small volume and chromatographed on neutral alumina, with methylene chloride as eluant. The first purple band was collected and reduced to dryness giving 0.85g (40%) of $Fe[C_5H_4CS(C_6H_4CH_3\text{-}p)]_2$ as a purple powder. NMR ($CDCl_3$):δ_H 7.41 (d,2H) and 7.04 (d,2H), C_6H_4; 4.78 (s,4H) and 4.62 (s,4H) C_5H_4; 2.33(s,3H,CH_3);δ_C 229.0 (C=S); 145.7

and 140.9 (quaternary carbons in C_6H_4);128.3 and 127.6 (C-H in C_6H_4); 91.5 (quaternary carbon in C_5H_4); 77.7 and 75.1 (C-H in C_5H_4);21.4 (CH_3).Infra-red:Vmax (C=S) 1217cm^{-1}.

3.2.3 Reaction of 1,1'-dibenzoylferrocene with tetraphosphorus decasulphide

1,1'-Dibenzoylferrocene (0.40 g, 1.0 mmol) was dissolved in a mixture of methylene chloride (50 cm^3) and diethyl ether (50 cm^3). Tetraphosphorus decasulphide (11.3 g, 25.5mmol) was added with vigorous stirring, and the mixture was refluxed during 1 h. The resulting mixture was filtered twice through Hyflo-supercel, and the deep-purple filtrate was reduced to small volume and chromatographed on silica, with toluene eluent, to remove the last traces of P_4S_{10}. The toluene eluate was reduced to small volume, and re-chromatographed on alumina. Elution with ether gave firstly **(2)**, in a yield of less than 1%: (Found: C, 62.7;H,3.9. $C_{24}H_{18}FeS_3$ requires: C,62.9; H, 4.0%). NMR. $\delta_H(CDCl_3)$ 4.26(m,2H), 4.30(m,2H), 4.32(m,2H), 4.33 (m,2H) (2 x C_5H_4), 7.2-7.3(m,6H) and 7.7-7.8(m,4H), C_6H_5; $\delta_c(CDCl_3)$ 69.0(d), 70.3(d), 71.7(d), 74.6(d), 83.0(s)(C_5H_4), 96.6(s, q,C·), 128.1(d,Ph,C_2 or C_3), 128.6(d,C_4), 129.0(d,C_3 or C_2), 137.1(s,C_1), (C_6H_5); the infra-red spectrum (CCl_4 solution) showed no absorption assignable to ν(C = O) or ν(C = S). This was followed by purple 1,1'-bis(thiobenzoyl)ferrocene, yield ca. 40%. Copies of the actual ^1H, ^{13}C and DEPT NMR spectra are in Spectra 3.1 to 3.4.

3.2.4 Preparation of $Mo(CO)_4$(norbornadiene)

$Mo(CO)_6$ (6.1 g;23mmol) and 2,5-norbornadiene (8 cm^3; 74 mmol) were refluxed in petroleum (40 cm^3; 100-120°C). After 12

hours the reaction mixture was allowed to cool and reduced to dryness. The yellow-brown solid was extracted with hot hexane and this crystallised in the cold yielding yellow crystals of $Mo(CO)_4$(norbornadiene) (4.2 g ; 60%).

3.2.5 Preparation of $(CH_3CN)_3M(CO)_3$

$Mo(CO)_6$ (10 g;38mmol) and 50 cm^3 CH_3CN were allowed to reflux for 6 hours; the mixture was then cooled and filtered and the filtrate was reduced to dryness . This gave a quantitative yield of $(CH_3CN)_3Mo(CO)_3$ as a yellow powder.

The preparations of the W and Cr analogues differed only in the reflux times. $W(CO)_6$ was refluxed 48 hours, and the $Cr(CO)_6$ 12 hours, but the latter could not be obtained free of the hexacarbonyl.

3.2.6 Reaction of $Fe[C_5H_4CS(C_6H_4CH_3\text{-}p)]_2$ with $(C_2H_5)_4N[Mo(CO)_5I]$

The $Fe[C_5H_4CS(C_6H_4CH_3\text{-}p)]_2$ (0.1g; 0.22 mmol) and $(C_2H_5)_4N[Mo(CO)_5I]$ were added together in a small conical flask wrapped in foil to exclude light, and 30 cm^3 dry methylene chloride added. After stirring for 15 minutes $AgNO_3$ (aq) , (0.88 cm^3 ;0.22 mmol;0.25mol dm^{-3}) was added. The flask ws stoppered and the mixture was stirred for 2 hours. After reduction to small volume chromatography on neutral alumina, with methylene chloride as eluant, gave a single blue band which was evaporated to give a blue-purple powder. This reaction was repeated using a (1:2) ratio of $Fe[C_5H_4CS(C_6H_4CH_3\text{-}p)]_2$ to $(C_2H_5)_4N[Mo(CO)_5I]$ and this yielded a similar blue-purple powder.

3.2.7 Reaction of $Fe[C_5H_4CS(C_6H_4CH_3\text{-}p)]_2$ with $(CH_3CN)_3Mo(CO)_3$

$Fe[C_5H_4CS(C_6H_4CH_3\text{-}p)]_2$ (0.15 g; 0.33 mmol) and $Mo(CH_3CN)_3(CO)_3$ (0.42 g;1.39 mmol) were dissolved in a mixture of dry hexane (10 cm^3) and dry methylene chloride (3 cm^3). After stirring for 5 days at room temperature, t.l.c examination showed the presence of a brown spot at the solvent front, an unknown blue spot (R_f 0.5) which was not the dithione, and decomposition products at the origin. The blue material could not be separated by chromatography on silica or alumina, because it quickly decomposed to a brown material which could not be removed from the column.

3.2.8 Reaction of $Fe[C_5H_4CS(C_6H_4CH_3\text{-}p)]_2$ with $Mo(CO)_4$(norbornadiene).

$Fe[C_5H_4CS(C_6H_4CH_3\text{-}p)]_2$ (0.4 g;0.88 mol) and $Mo(CO)_4$(nor) (0.26 g; 0.88 mol) were combined and dry hexane (50 cm^3) added. After stirring at room temperature for 15 minutes t.l.c examination showed the presence of a blue spot (R_f 0.5), which decomposed quickly to a brown material. Chromatography both on silica and on alumina failed.

3.2.9 Preparation of ferrocene dicarboxylic acid.

This reaction was carried out under nitrogen and with the exclusion of light.

To tetramethylethylenediamine (TMEDA) (5.6 cm^3; 37 mmol) and 5 cm^3 dry hexane was added n-butyllithium (23 cm^3;1.6M;37 mmol) and allowed to stir for 15 minutes. Ferrocene (2.8g ; 15 mmol) dissolved in hexane (125 cm^3) was added to the complex over 30 minutes with rapid stirring. After stirring 12 hours, the reaction mixture was poured onto a CO_2 (s)/diethyl ether bath with stirring and allowed to warm to room temperature, filtered at the pump, and washed with a little cold ether.The precipitate was a yellow-golden powder.

HCl (conc.,20 cm^3) was added to the precipitate whilst in the Büchner funnel, sucked dry, washed with a little water and dried in a vacuum dessicator.

Soxhlet extraction with ethyl acetate yielded 4.1g (99%) of insoluble ferrocene dicarboxylic acid, $Fe(C_5H_4CO_2H)_2$, mpt. dec>240°C, and also <0.1g ferrocene monocarboxylic acid, $FeC_5H_5(C_5H_4CO_2H)$, as a yellow powder, mpt 209-210°C.

3.2.10 Preparation of ferrocene diacid chloride

Ferrocene dicarboxylic acid (3 g; 11 mmol) methylene chloride (50 cm^3), oxalyl chloride (5 cm^3;55mmol) and 3 drops of pyridine were combined under nitrogen and refluxed for 4 hours. Reduced to dryness at 40°C, the brown-red solid was extracted with hot hexane and the combined extracts reduced to yield 2.02g (59%) deep red crystals of $Fe(C_5H_4COCl)_2$, mpt. 88.5-89°C Calc. 46.35%C 2.59%H found 46.85%C 2.55%H. NMR ($CDCl_3$):δ_H 5.03 (s,4H) and

4.77 (s,4H) C_5H_4. NMR ($CDCl_3$): δ_C 168.44 (C=O); 76.70 (quaternary C in ring); 76.11 and 74.18 (C-H in ring).

3.2.11 Reaction of ferrocene diacid chloride with ferrocene

Ferrocene diacid chloride (2.00g;6.4mmol), ferrocene (1.1g;5.8 mmol) and $AlCl_3$(4.3g;32mmol) were added together, and 20 cm^3 dry methylene chloride (20 cm^3) added. After stirring for 6 hours, a t.l.c examination showed no diacid chloride to be present. The mixture was hydrolysed in ice, washed with water, dried and evaporated to give 0.30g (8.50%) crude **(3a)**. Soxhlet extraction with benzene, gave a trace of red material, further extraction with ethyl acetate yielded a red-orange powder of pure diferrocenoyl ferrocene **(3a)** $C_{32}H_{26}O_2Fe_3$ (0.20g;5.6%).NMR ($CDCl_3$):δ_H 5.00 and 4.96 (s,24.3, C_5H_4); 4.54, and 4.50 (s,25.7,C_5H_4);4.17 (s,29.9,C_5H_5).NMR ($CDCl_3$):δ_C 198.60 (s,CO);81.59 and 79.94 (quaternary C on rings);73,74, 71.82, 71.82, and 70.59 (C-H on substituted ring);70.03 (C-H on unsubstituted ring). Infra-red ν(CO) 1628 cm^{-1}.(CCl_4 solution)

3.2.12 Preparation of the dithione **(3b)**

(3a) (0.3g;0.6mmol) and P_4S_{10} (7.0g;15mmol) were added together in 50 cm^3 methylene chloride. A purple colouration was apparent immediately. After stirring at room temperature 24 hours, a t.l.c examination showed a vivid purple band at the solvent front. The mixture was filtered through Hyflo, and chromatography on alumina eluting with ethyl acetate yielded a single purple band which reduced to a purple powder 0.20g (53% yield).of dithioferrocenoyl ferrocene **(3b)**.$C_{32}H_{26}S_2Fe_3$ The infra-red spectrum showed no absorbance in the 1600 cm^{-1} region. All attempts at crystallisation failed.

3.3 Results and Discussion

Diacylferrocenes $Fe(C_5H_4COR)_2$ were readily thionated to the thioacyl derivatives $Fe(C_5H_4CSR)_2$ by the use of tetraphosphorus decasulphide: after work-up, chromatography on alumina provided the dithioacyl ferrocenes as deep purple oils . The choice of solvent employed is critical to the effectiveness of the thionation procedure for diacylferrocenes. When dry benzene, toluene or CH_2Cl_2 was employed, good yields were obtained. However when one of the more polar solvents such as THF or diglyme which have been recommended[12] for such thionations was employed, the yields of dithioacylferrocenes were poor: not only was unchanged starting material still present, but other unidentified impurities were also formed under these conditions. With dry solvents of low polarity, practical yields of pure dithioacylferrocenes were readily achieved.

Thionation of 1,1'-dibenzoylferrocene with tetraphosphorus decasulphide produced 1,1'-bis(thiobenzoyl)ferrocene in yields around 40%, together with a chromatographically homogeneous by-product **(2)** in yields never more than *ca* 1%. The use of sodium hydrogencarbonate, as recommended earlier[12] for the thionation of organic ketones, effected no improvement in the yield of either product, but merely rendered the work-up more complex.

The yellow colour of **(2)** suggested that it contained no C = S bonds, and the infra-red spectrum showed no absorption assignable to either C = O or C = S stretches. The ^{1}H NMR. spectrum of **(2)** showed, in addition to the absorption characteristic of a mono-substituted phenyl group, four resonances in the cyclopentadienyl region, each of which had an intensity equivalent to one proton for each phenyl group present. Since mono-substituted cyclopentadienyl ligands normally provide just two proton resonances, the observation of four such signals initially suggested the presence of two non-equivalent cyclopentadienyl ligands. The ^{13}C NMR. spectrum, in addition to the usual signals from a mono-substituted phenyl ring of local C_{2v} symmetry, showed six other resonances, of which four were shown by DEPT to arise from C-H groups, while the remaining two were from carbons not attached to hydrogen. Again the observation of four C-H resonances in the cyclopentadienyl region suggested the presence of two distinct cyclopentadienyl ligands; together the ^{1}H and ^{13}C spectra indicated that the initial C_5H_4 - C - C_6H_5 fragment was retained.

Microanalysis supported a C:H atomic ratio of 12:9 as required by this fragment: on the assumption of two cyclopentadienyl ligands per iron atom, the analytical data indicated a relative molar mass per iron atom of 459, consistent with the formation $C_{24}H_{18}FeS_3$ (M_r, 458.4). This deduction was fully supported by the results of a single crystal X-ray structure determination.

The reaction of an organic compound containing a relatively acidic hydrogen and an organolithium reagent is referred to as a

metalation reaction[21]. This reaction is limited to those materials with a fairly acidic hydrogen, aromatic hydrocarbons do not generally undergo this metalation[22] also, the metalating reagents are genarally unstable in the working solvent, THF usually. It has been found[23] that certain diamines such as TMEDA and DABCO form stable coordination compounds with organolithium reagents and these complexes are considerably more reactive than the organolithium reagents alone. The di-lithiated derivative can then be carbonated and hydrolysed to the di-carboxylic acid by the method of Rausch[15]. The conversion of di-carboxylic acid to the di-acid chloride was achieved using oxalyl chloride and pyridine by the method of Adams[24].

The di-ketone **(3a)**, once formed, was then converted to the di-thioketone **(3b)** by the standard reaction with P_4S_{10} in an aprotic solvent yielding the characteristically purple di-thioketone. **(3b)** was however unstable and resisted all attempts at purification. Crystallisation was not possible since the di-thioketone degraded in solution. Sublimation resulted in complete decomposition to a black insoluble material. Repeated chromatography undoubtedly purified the material but, once pure, quickly began to decompose in the solvent. Since a pure analytical sample could not be obtained, no spectral data could be reliably recorded.

3.3.1 X-ray Crystallography.

Crystals suitable for X-ray examination were grown from CH_2Cl_2/light petroleum. The data was collected and the structure solved by Dr. G.Ferguson at the Department of Chemistry and Biochemistry, University of Guelph, Guelph, Ontario, Canada, N1G 2W1.

3.3.2 Crystal Data.

$C_{24}H_{18}FeS_3$, M_r = 458.45, monoclinic, a = 11.769(3), b = 11.750(4), c = 14.835(2)Å, β = 98.63(1)°, V = 2028(1)Å^3, Z = 4, D_c = 1.50 g cm^{-3}, μ(Mo-K$_\alpha$) = 10.5 cm^{-1}, λ=0.71073Å F(000) = 944, space group $P2_1/n$ (No. 14) (from systematic absences: hol, h + l = 2n + 1; oko, k = 2n + 1).

3.3.3 Data Collection.

Cell dimensions were determined by least-squares refinement using the setting angles for 22 reflections in the range $7° \leq \theta \leq 20°$. Intensity data were measured at 21 °C using a CAD4 diffractometer with graphite-monochromated Mo-K$_\alpha$ radiation, in the $\omega/2\theta$ scan mode; the ω-scan rate was 1-7° min^{-1}, the ω-scan width was (0.70 + 0.35 tanθ)°, and the maximum value of 2θ was 54°. A total of 4950 reflections were measured, of which 3176 were unique and 3146 had $I \geq 3\sigma(I)$. Lorentz and polarisation correction were applied, together with a numerical absorption correction: maximum and minimum transmission coefficients were 0.683 and 0.561.

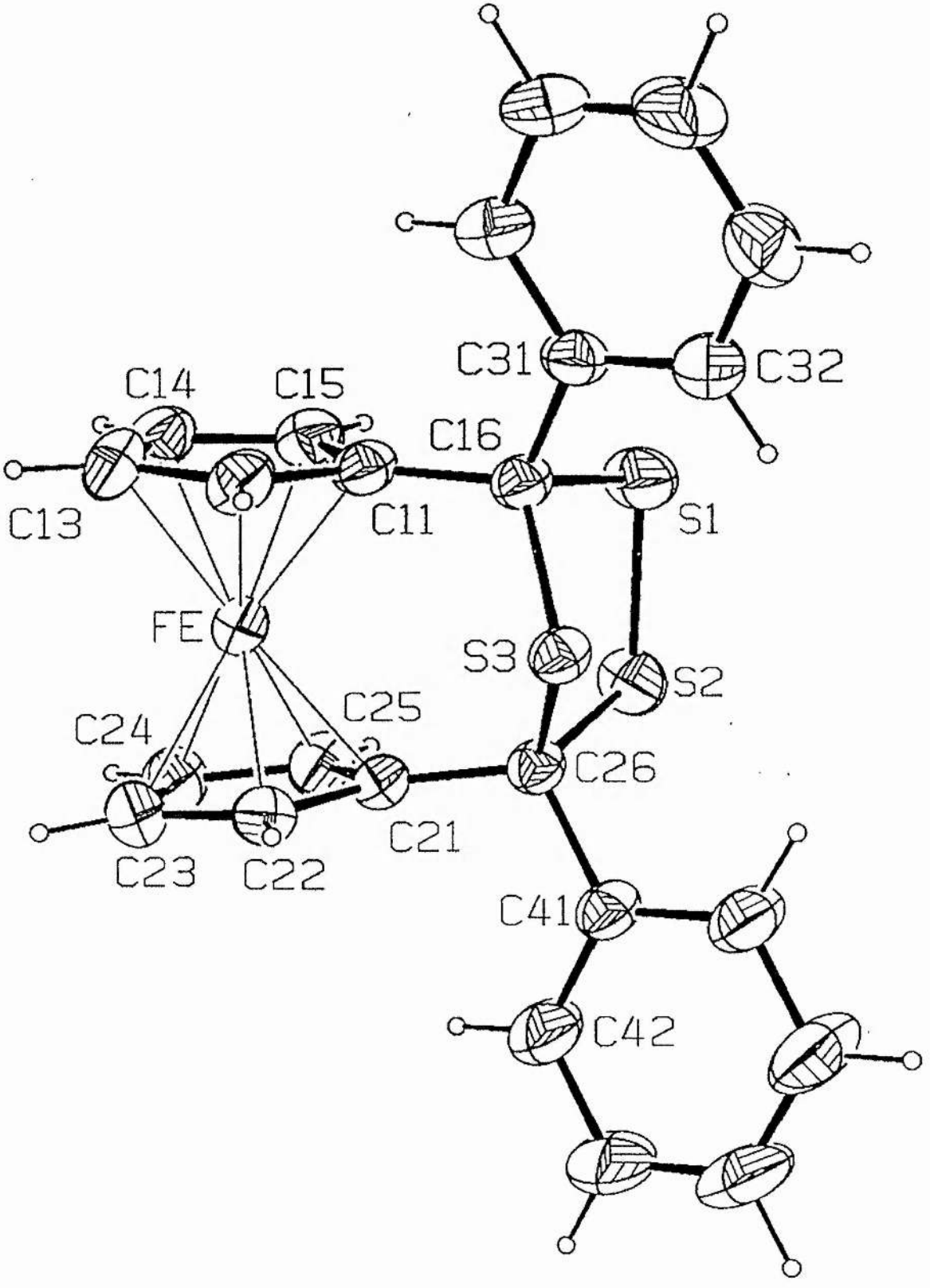
3.3.4 Structure Solution and Refinement.

The structure was solved using the Patterson heavy-atom method which revealed the position of the iron atom, followed by difference Fourier syntheses. All non-hydrogen atoms were refined anisotropically: hydrogen atoms were visible in difference maps and were included in the refinement as riding atoms with δ(C-H) = 0.95Å and B_{iso} fixed at 5Å^2. The final R values were R, 0.041 and R_w, 0.053.

Scattering factor data were taken from refs[25-27]. All calculations were performed on a PDP-11/73 computer using SDP-Plus[28]. Final refined atom coordinates are given in Table 3.4; bond lengths and selected bond angles are given in Table 3.5 A perspective view of the molecule, showing the atom-numbering scheme is in Figure 3.3.

Hydrogen-atom coordinates are in appendix 3.1, anisotropic temperature factors in appendix 3.2, least-squares planes in appendix 3.3, and torsional angles in appendix 3.4, as well as a complete table of bond angles in appendix 3.5. Observed and calculated structure factors are in Appendix 3.6.

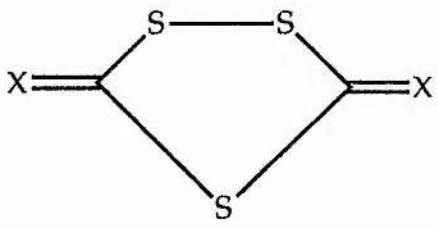
Figure 3.3. Perspective view of the 1,4-diphenyl-1,4-epithio-2,3-dithia[4](1,1')ferrocenophane, **(2)** molecule, showing the atom-numbering scheme.



3.3.5 Crystal and Molecular Structure.

The crystal structure revealed a molecular unit in which the two carbon atoms which originally formed the carbonyl groups in 1,1'-dibenzoylferrocene had been incorporated into a five-membered C_2S_3 ring; since the resulting 1,2,4-trithiolane ring is connected, at carbon, to the two cyclopentadienyl rings, the compound **(2)** is thus a bridged ferrocenophane.

Rather few derivatives containing 1,2,4-trithiolane rings have been structurally characterised: the known examples **(4a)** - **(4g)** all have two exocyclic double bonds.

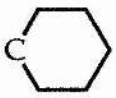


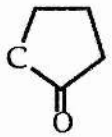
(4)

a X = CHC(O)CMe_3 (ref. 29)

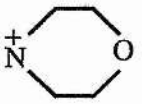
b X = NNHPh (ref. 30)

c X = N^+Et_2 (ref. 31)

d X =  (ref. 32)

e X =  (ref. 33)

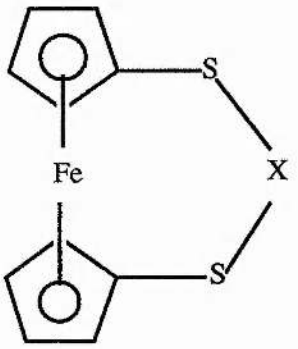
f X = CMe_2 (ref. 34)

g X =  (ref. 35)

In compound **(2)** on the other hand, there are no double bonds exocyclic to the trithiolane ring, whose carbon atoms are both essentially

tetrahedral: accordingly the C-S bond lengths in **(2)** (range 1.825(3)-1.868(3)Å) are significantly longer than those found in compounds **(4)**, in which the C-S bond lengths cluster around 1.74Å.

The overall molecular symmetry of **(2)** is approximately μ (C_s), although no symmetry is imposed crystallographically. The two cyclopentadiene rings are not parallel: the rings are inclined at an angle of 8.5°, while the ring centroids subtend an angle at iron of 174.9°. Thus as well as a bending at iron, there is also a simple tilting of the rings about their centroids, as shown by the variation of the Fe-C bond lengths (Table 2). The bending at iron is much more than observed in the two derivatives **(5)**[14] and **(6)**[15];



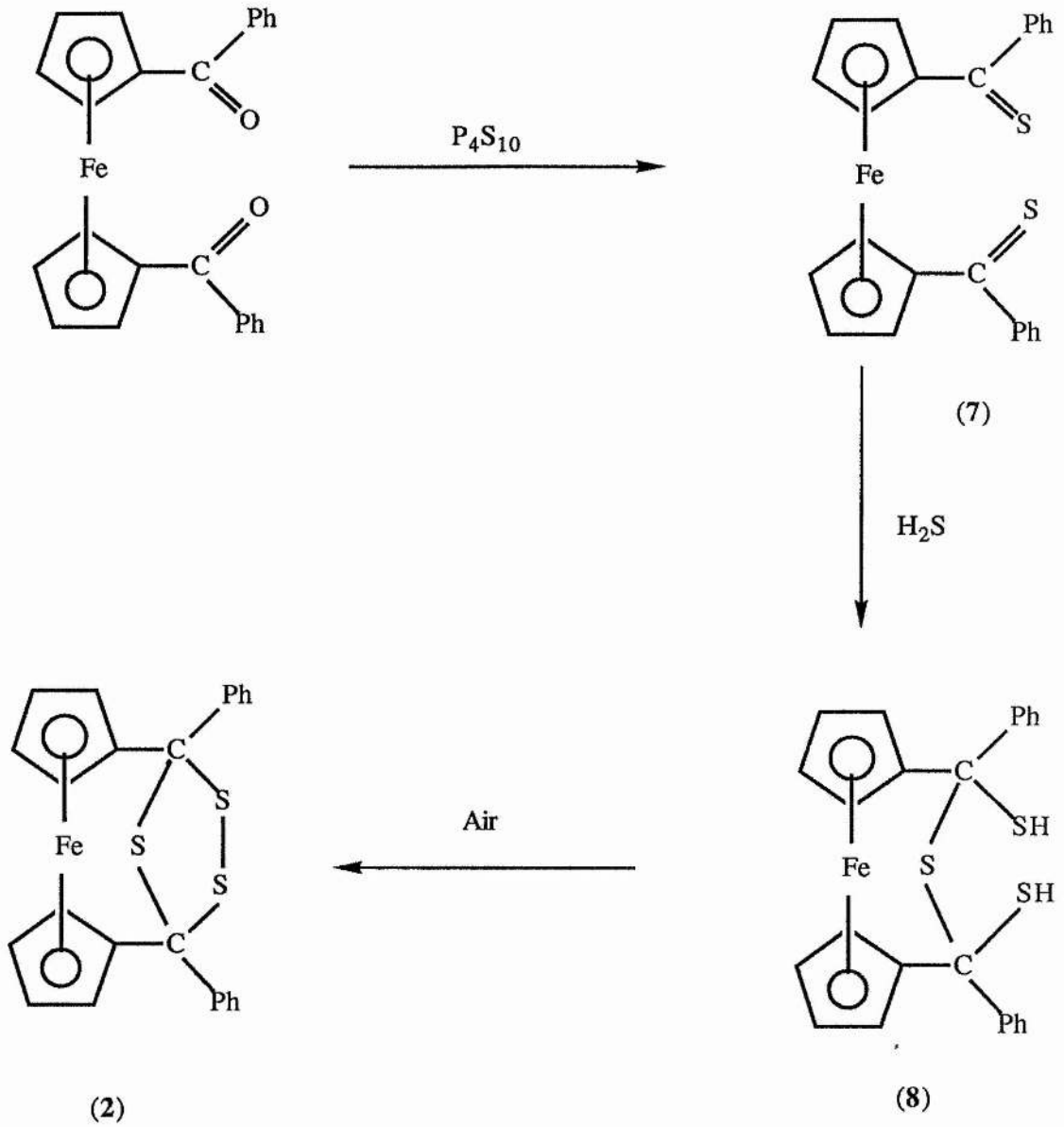
(5) X = S

(6) X = Se

in **(5)** the ring centroids subtend an angle of 177.1° at iron, while in **(6)** the angle is 177.6°. In compound **(2)** the rings are twisted by 2.6(2)° from the fully eclipsed conformation, as compared with 0.1° and 1.5° in **(5)** and **(6)** respectively.

The observed NMR. spectra in solution are readily interpreted in terms of the structure revealed by X-ray analysis, provided only that the rotation of the phenyl rings about bonds C16-C31 and C26-C41 is not restrained. The presence of the trithiolane ring renders distinct each of the four C-H fragments in the cyclopentadienyl rings, although the two cyclopentadienyl rings are equivalent; hence the observation of four signals due to cyclopentadienyl C-H fragments in the ^{1}H and ^{13}C spectra shown in spectra 3.1, 3.2 and clarified in spectra 3.3 and in the DEPT spectrum 3.4. The molecule contains two types of quaternary carbon atoms, other than in the phenyl rings, with δ_C 83.0 and 96.6: we tentatively assign the resonance at higher frequency to the carbon bound to two sulphur atoms and that at lower frequency to the carbon in the cyclopentadienyl ring.

While the mechanism of formation of **(2)** is not established, a reasonable route is that shown in figure 3.3: thionation gives 1,1'-bis(thiobenzoyl)ferrocene (**7**) as the major isolated product, but H_2S (produced by the action of traces of moisture with P_4S_{10}) could add across the two thioketone groups to give the dithiol (**8**), oxidation of which provides **(2)** as the isolable by-product.



(2) (8)

Figure 3.3. Possible route for formation of (2)

Table 3.1 Properties of Di-acyl ferrocene derivatives.[b]

Derivative[a]	ν(CO) cm⁻¹	mpt °C	Analysis calc. %C %H	found %C %H	yield %
Fcd($COCH_3$)$_2$	1679	124-125	62.25 5.22	62.20 5.11	70
Fcd($COCH_2CH_3$)$_2$	1680	54-55	64.45 6.08	64.36 6.20	45
Fcd(COC_6H_5)$_2$	1650	103	73.12 4.60	72.08 4.35	60
META Fcd($COC_6H_4CH_3$)$_2$	1650	93-94	73.95 5.25	73.81 5.18	85
PARA Fcd($COC_6H_4CH_3$)$_2$	1645	170-171	73.95 5.25	73.21 5.21	99

a/ All spectra were recorded in $CDCl_3$, with TMS as internal standard, at 20 °C.

b/ Fcd is -$C_5H_4FeC_5H_4$-

Table 3.2 ^{1}H NMR Data for Di- acyl ferrocenes in ppm.[a]

Derivative[b]	Ferrocenyl group	Associated R group
$Fcd(COCH_3)_2$	4.65 (s,2H) 4.40 (s,2H)	2.25 (s,3H)
$Fcd(COCH_2CH_3)_2$	4.76 (s,2H) 4.46 (s,2H)	2.66 (q,2H) 1.17 (t,3H)
$Fcd(COC_6H_5)_2$	4.80 (s,2H) 4.95 (s,2H)	7.70-7.30 (m,4H)
(META) $Fcd(COC_6H_4CH_3)_2$	4.90 (s,2H) 4.55 (s,2H)	7.55-7.30 (m,4H) 2.4 (s,3H)
(PARA) $Fcd(COC_6H_4CH_3)_2$	4.90 (s,2H) 4.57 (s,2H)	7.70-7.20 (m,4H) 2.4 (s,3H)

a/ All spectra were recorded in $CDCl_3$, with TMS as internal standard, at 20°C.

b/ Fcd is $-C_5H_4FeC_5H_4-$

TABLE 3.3 ^{13}C NMR Data for Di-acyl ferrocenes in ppm [a]

Derivative [b]	C=O	quaternary C on Cp ring	C-H on Cp ring	R group
Fcd($COCH_3$)$_2$	201.47	81.07	74.03 71.38	28.07 ($\underline{C}H_3$)
Fcd($COCH_2CH_3$)$_2$	203.90	80.20	73.16 70.44	32.95 ($\underline{C}H_2CH_3$) 8.07 ($CH_2\underline{C}H_3$)
Fcd(COC_6H_5)$_2$	198.03	79.80	74.91 73.42	139.33 132.23 138.39 128.64 (C-H,C_6H_5)
(META) Fcd($COC_6H_4CH_3$)$_2$	198.03	79.64	74.58 73.10	21.40 ($\underline{C}H_3$) 139.17 138.09 132.64 132.65 (C-H,C_6H_5)x 2 128.07 125.34
(PARA) Fcd($COC_6H_4CH_3$)$_2$	197.57	79.73	74.51 73.06	21.62 ($\underline{C}H_3$) 142.47 128.93 136.41 128.31 (C-H,C_6H_5)

a/ All spectra were recorded in $CDCl_3$ with TMS as internal standard, at 20 °C.

b/ Fcd is -$C_5H_4FeC_5H_4$-

Table 3.4

Positional and thermal parameters and their e.s.d.'s

Atom	x	y	z	B(Å^2)
Fe	0.03444(3)	0.23062(4)	0.03142(3)	2.628(8)
S1	-0.22537(7)	0.16293(7)	-0.17424(5)	3.65(2)
S2	-0.07424(7)	0.18521(7)	-0.22804(5)	3.38(1)
S3	-0.13760(6)	0.39300(6)	-0.13226(4)	2.45(1)
C11	-0.1371(2)	0.2305(2)	0.0043(2)	2.75(5)
C12	-0.1002(3)	0.3014(3)	0.0814(2)	3.13(6)
C13	-0.0362(3)	0.2339(3)	0.1496(2)	3.66(7)
C14	-0.0342(3)	0.1214(3)	0.1165(2)	3.69(7)
C15	-0.0976(3)	0.1184(3)	0.0277(2)	3.16(6)
C16	-0.2067(2)	0.2728(2)	-0.0835(2)	2.53(5)
C21	0.0729(2)	0.2877(2)	-0.0872(2)	2.53(5)
C22	0.1148(3)	0.3655(3)	-0.0153(2)	3.07(6)
C23	0.1934(3)	0.3056(3)	0.0499(2)	3.74(7)
C24	0.2013(3)	0.1926(3)	0.0196(2)	3.91(7)
C25	0.1290(3)	0.1808(3)	-0.0659(2)	3.17(6)
C26	-0.0164(2)	0.3174(2)	-0.1679(2)	2.36(5)
C31	-0.3287(2)	0.3088(3)	-0.0713(2)	2.97(6)
C32	-0.3959(3)	0.3749(3)	-0.1354(2)	4.01(7)
C33	-0.5059(3)	0.4055(4)	-0.1243(3)	4.97(9)
C34	-0.5518(3)	0.3678(4)	-0.0502(3)	5.02(9)
C35	-0.4877(3)	0.3004(4)	0.0136(2)	4.89(8)
C36	-0.3760(3)	0.2716(3)	0.0035(2)	3.89(7)
C41	0.0326(2)	0.3917(3)	-0.2373(2)	2.81(5)

Positional and thermal parameters and their e.s.d.'s (con.

Atom	x	y	z	B(Å^2)
C42	0.1476(3)	0.4035(4)	−0.2358(2)	5.25(9)
C43	0.1912(3)	0.4716(5)	−0.2984(3)	7.7(1)
C44	0.1190(3)	0.5291(4)	−0.3630(3)	6.3(1)
C45	0.0040(4)	0.5172(4)	−0.3670(2)	5.71(9)
C46	−0.0402(3)	0.4477(3)	−0.3053(2)	4.29(7)

Anisotropically refined atoms are given in the form of the isotropic equivalent thermal parameter defined as:

(4/3) * [a2*B(1,1) + b2*B(2,2) + c2*B(3,3) + ab(cos gamma)*B(1,2) + ac(cos beta)*B(1,3) + bc(cos alpha)*B(2,3)]

Table 3.5

Molecular dimensions

(a) Bond lengths (Å)

Fe	C11	1.998(3)
Fe	C12	2.026(3)
Fe	C13	2.051(3)
Fe	C14	2.050(3)
Fe	C15	2.032(3)
Fe	C21	1.997(3)
Fe	C22	2.021(3)
Fe	C23	2.048(3)
Fe	C24	2.046(3)
Fe	C25	2.037(3)
S1	S2	2.072(1)
S1	C16	1.854(3)
S2	C26	1.868(3)
S3	C16	1.831(3)
S3	C26	1.825(3)
C11	C12	1.429(4)
C11	C15	1.422(4)
C11	C16	1.515(4)
C12	C13	1.412(4)
C13	C14	1.411(5)
C14	C15	1.414(4)

C16	C31	1.534(4)
C21	C22	1.435(4)
C21	C25	1.431(4)
C21	C26	1.511(3)
C22	C23	1.421(4)
C23	C24	1.409(5)
C24	C25	1.424(4)
C26	C41	1.528(4)
C31	C32	1.381(4)
C31	C36	1.385(5)
C32	C33	1.378(5)
C33	C34	1.369(6)
C34	C35	1.370(5)
C35	C36	1.387(5)
C41	C42	1.356(5)
C41	C46	1.387(4)
C42	C43	1.382(6)
C43	C44	1.362(6)
C44	C45	1.353(6)
C45	C46	1.386(5)

C41	C42	C43	121.2(3)
C42	C43	C44	120.3(4)
C43	C44	C45	119.6(4)
C44	C45	C46	120.3(4)
C41	C46	C45	120.5(3)

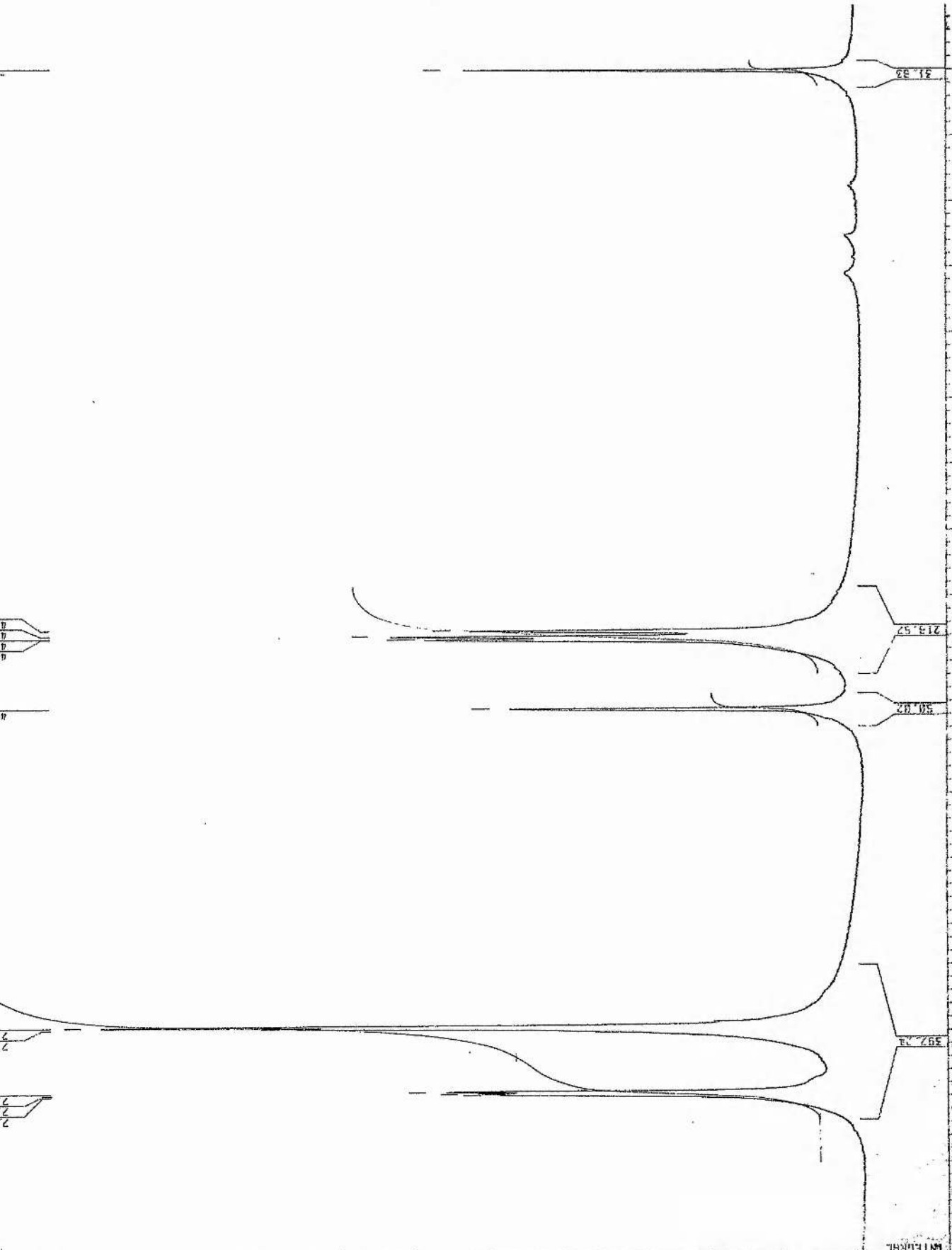
Selected

(b) Bond angles (°)

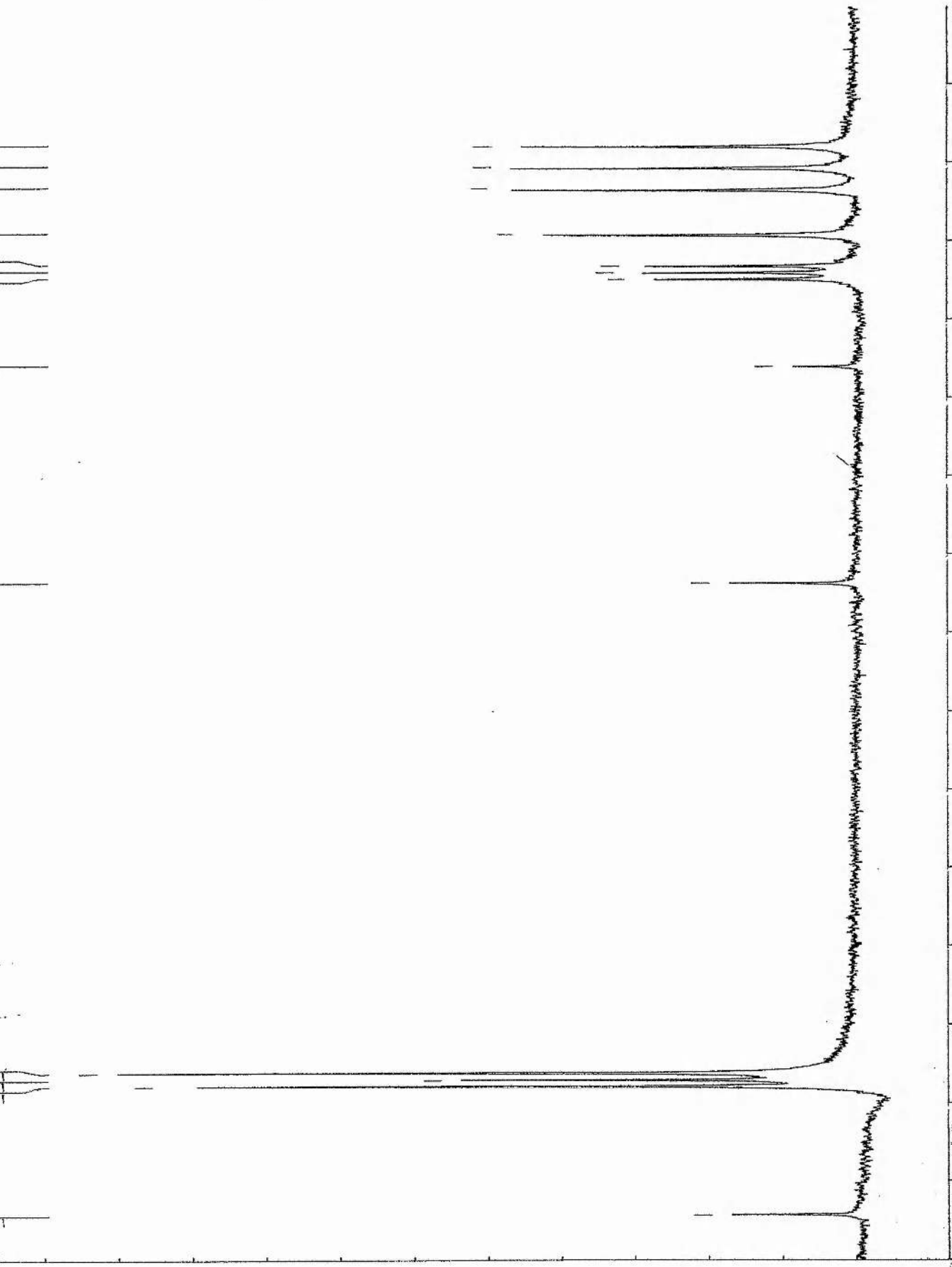
C11	Fe	C12	41.6(1)
C11	Fe	C15	41.3(1)
C12	Fe	C13	40.5(1)
C13	Fe	C14	40.3(1)
C14	Fe	C15	40.5(1)
C21	Fe	C22	41.8(1)
C21	Fe	C25	41.5(1)
C22	Fe	C23	40.9(1)
C23	Fe	C24	40.3(1)
C24	Fe	C25	40.8(1)
S2	S1	C16	100.1(1)
S1	S2	C26	101.2(1)
C16	S3	C26	98.7(1)
Fe	C11	C12	70.2(2)
Fe	C11	C15	70.6(2)
Fe	C11	C16	124.9(2)
C12	C11	C15	107.2(2)
C12	C11	C16	123.9(3)
C15	C11	C16	128.9(3)
C11	C12	C13	108.2(3)
C12	C13	C14	108.1(3)
C13	C14	C15	108.3(3)
C11	C15	C14	108.2(3)
S1	C16	S3	105.2(1)
S1	C16	C11	112.7(2)
S1	C16	C31	105.5(2)
S3	C16	C11	112.2(2)
S3	C16	C31	108.2(2)

C11	C16	C31	112.6(2)
Fe	C21	C22	70.0(2)
Fe	C21	C25	70.7(2)
Fe	C21	C26	123.6(2)
C22	C21	C25	107.4(2)
C22	C21	C26	124.2(3)
C25	C21	C26	128.3(2)
C21	C22	C23	107.8(3)
C22	C23	C24	108.4(3)
C23	C24	C25	108.5(3)
C21	C25	C24	107.8(3)
S2	C26	S3	107.4(1)
S2	C26	C21	110.3(2)
S2	C26	C41	107.5(2)
S3	C26	C21	111.3(2)
S3	C26	C41	107.9(2)
C21	C26	C41	112.2(2)
C16	C31	C32	121.5(3)
C16	C31	C36	120.2(3)
C32	C31	C36	118.2(3)
C31	C32	C33	121.0(3)
C32	C33	C34	120.2(3)
C33	C34	C35	119.9(3)
C34	C35	C36	120.0(4)
C31	C36	C35	120.6(3)
C26	C41	C42	121.5(3)
C26	C41	C46	120.4(3)
C42	C41	C46	118.1(3)

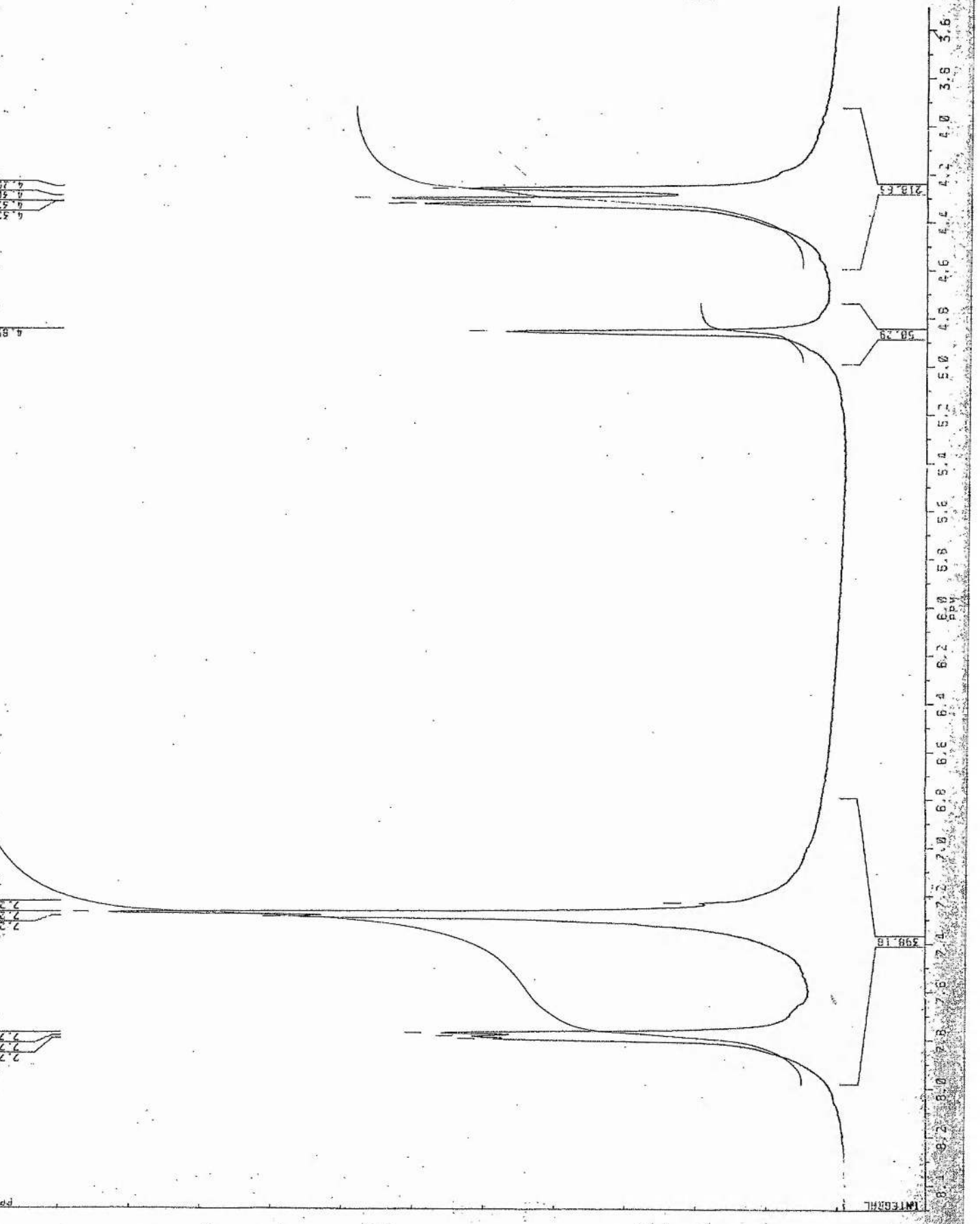
Spectrum 3.1 ^{1}H NMR spectrum of(2) in $CDCl_3$ at 20°C with TMS as internal standard.



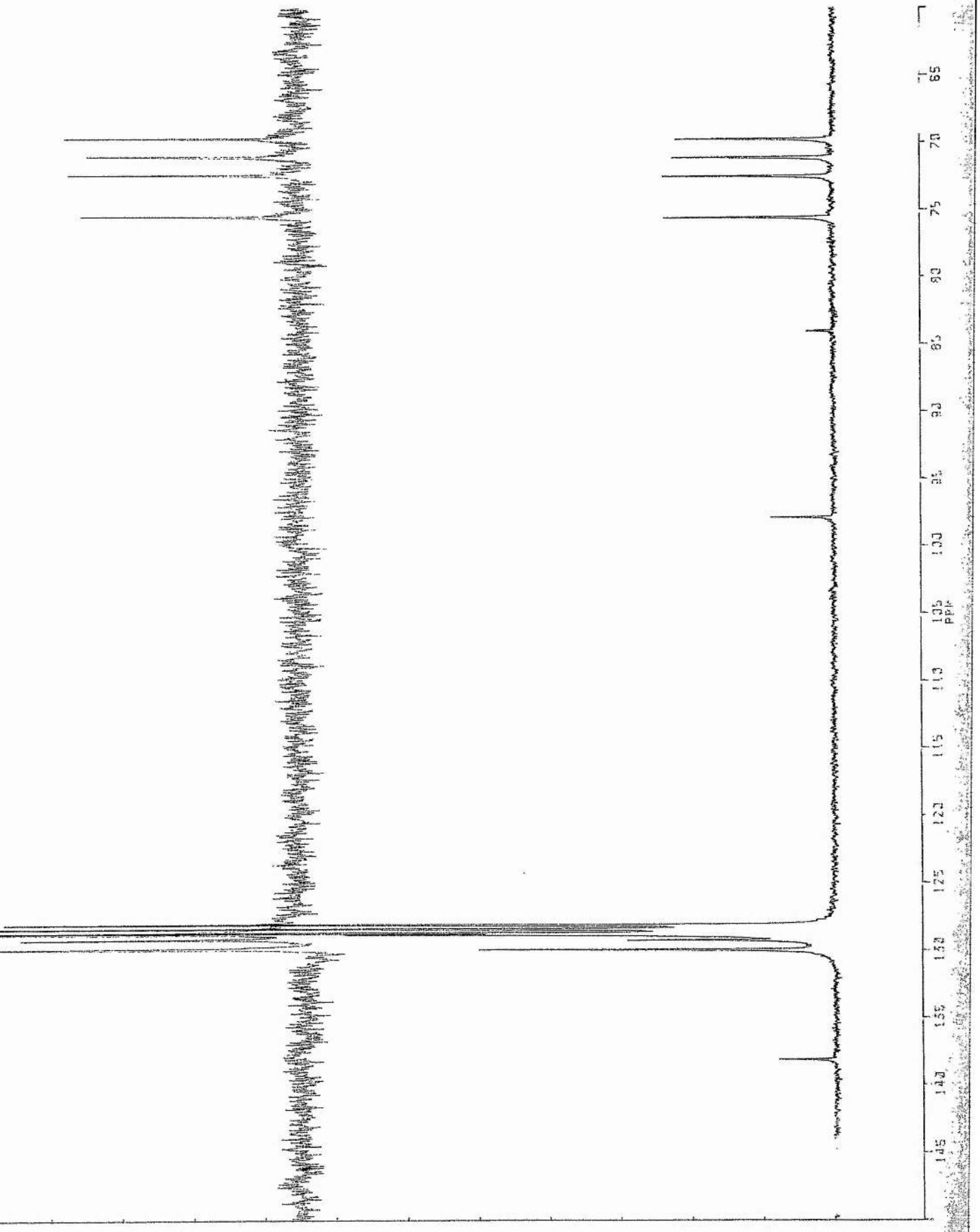
Spectrum 3.2 ^{13}C NMR spectrum of(**2**) in $CDCl_3$ at 20°C with TMS as internal standard.



Spectrum 3.3 Expanded view of Spectrum 3.1.



Spectrum 3.4 D E P T. spectrum of (2) showing the C-H on the top trace, with the quaternary and C-H on the lower trace.



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Chapter Four
Concurrent Acylation and Alkylation in the Friedel-Crafts Reaction of Ferrocene with Trimethylacetyl Chloride

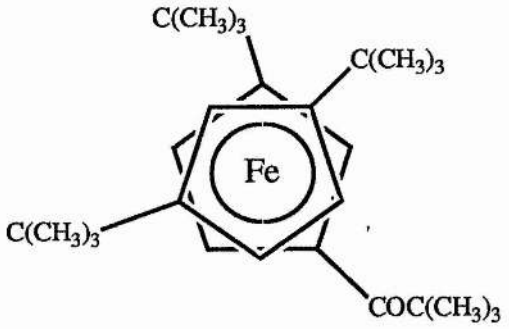
4.1 Introduction

Whereas the reaction of ferrocene with a stoichiometric quantity of $Me_3CCOCl/AlCl_3$ provides the monoacylated product in 87% yield, the use of excess of the acylation reagent leads to concurrent acylation and alkylation, two products of which have been isolated and characterised by NMR. spectroscopy.

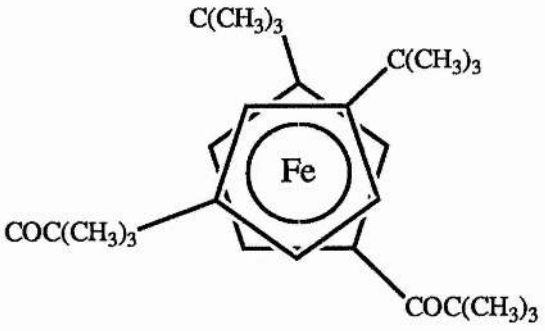
There are few reports in the literature of poly-acylated ferrocenes: acyl substituents deactivate the cyclopentadienyl rings towards electrophilic reagents, and the presence of a single acyl substituent is sufficient to direct virtually all subsequent acylation to the unsubstituted ring. The resulting 1, 1' - diacylferrocenes are generally inert to further acylation. However, it was reported some years ago[1] that reaction of ferrocene with a large excess of acetic anhydride, in the presence of trifluoracetic acid, yielded a purple tetraacetyl ferrocene. This result is surprising both in terms of the known deactivation of acyl-substituted cyclopentadienyl rings, and because of the unusual colour reported for the product.

Our attempts to repeat this work have been uniformly unsuccessful for a range of acyl derivatives. However, we have observed that reaction of ferrocene with an excess of the 1:1 complex formed between aluminium chloride and trimethylacetyl chloride does indeed yield tetrasubstituted derivatives: two of these have been isolated and characterised by ^1H and ^{13}C NMR. spectroscopy as 1,1,3-tri-t-butyl-3'-(2,2-dimethylpropionyl)

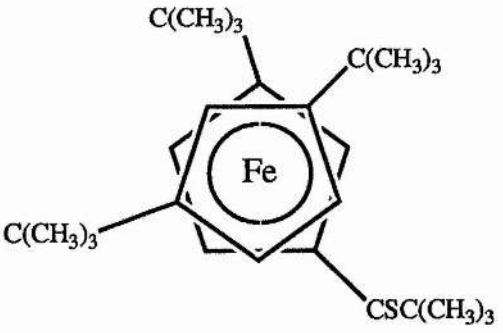
ferrocene, $[C_5H_3(CMe_3)_2]$ Fe $[C_5H_3(CMe_3)COCMe_3)]$ (1) and 1,1'-di-t-butyl-3,3'-bis(2,2-dimethylpropionyl)ferrocene, $[C_5H_3(CMe_3)(COCMe_3)]_2Fe$ (2). Compound (1) can readily be thionated, using P_4S_{10}, to the thioacyl analogue (3).



(1)



(2)



(3)

4.2 Experimental

Diethylether, and light petroleum (b.p. 40 - 60°C) toluene, were dried by reflux over sodium diphenylketyl; methylene chloride was dried by reflux over calcium hydride. NMR. spectra were recorded using a Bruker AM-300 spectrometer and mass spectra were recorded using an INCOS-50 GC-MS system. Thio(2,2-dimethylpropionyl)ferrocene, $(C_5H_5)Fe(C_5H_4CSCMe_3)$ was prepared as previously described[7] and had δ_C ($CDCl_3$): 32.7 (q, $C(\underline{C}H_3)_3$), 51.5 (s, $\underline{C}(CH_3)_3$), 72.0 (d, C_5H_5); 73.3(d), 73.8(d) and 87.9(s) (all $\underline{C}_5H_4CSCMe_3$); 258.1 (s, $\underline{C}$=S).

4.2.1 Preparation of (2,2-dimethylpropionyl)ferrocene, $(C_5H_5)Fe(C_5H_4COCMe_3)$.

Ferrocene (30g, 0.16 mol) was dissolved in dry CH_2Cl_2 (300 cm^3). Aluminium chloride (26 g, 0.20 mol) and Me_3CCOCl (24 g, 0.20 mol) were added, each in eight equivalent portions mixed in dry CH_2Cl_2 (20 cm^3). After the addition was complete, the whole mixture was stirred for 12 h. The mixture was hydrolysed with crushed ice, and the organic layer was washed with water, and then dried over $CaCl_2$. The volume was reduced to 50 cm^3, and this solution was then poured into 2 dm^3 of rapidly stirred light petroleum. After 12 h. red needles of the product were filtered off (37.5 g, 87%) m.p. 83°C. Found: C, 66.6; H, 6.8. $C_{15}H_{18}FeO$ requires: C, 66.7; H, 6.7%. NMR. δ_C ($CDCl_3$): 28.1 (q, $C(\underline{C}H_3)_3$), 44.1 (s, $\underline{C}(CH_3)_3$), 69.7 (d, C_5H_5): 70.9 (d), 71.1(d), and 76.7(s) (all $\underline{C}_5H_5COCMe_3$); 210.1 (s, CO).Infra-red (CCl_4 solution): 1661 cm^{-1}, ν (C=O).

4.2.2 Reaction of ferrocene with excess complex $Me_3CCOCl/AlCl_3$.

Aluminium chloride (43 g, 0.32 mol) and trimethylacetyl chloride (39 g, 0.32 mol) were mixed together in dry CH_2Cl_2 (500 cm^3). A solution of ferrocene (30 g, 0.16 mol) in dry CH_2Cl_2 (100 cm^3) was added dropwise with stirring, and the mixture was stirred for a further 24 h. At this point t.l.c. examination showed presence of five orange components. Further portions of $AlCl_3$ (86 g, 0.64 mol) and Me_3CCOCl (78 g, 0.64 mol) were then added directly to the mixture, and the whole stirred during 48 h. The mixture was hydrolysed with crushed ice, and the organic layer was washed with water, and then dried over $CaCl_2$. The organic phase was reduced to small volume and chromatographed on silica, with CH_2Cl_2 as eluant. The major orange product was recrystallised from hot cyclohexane to provide (1) (25.0 g, 36%), m.p. 182 - 183°C. NMR.; δ_H (C_6D_6 at 80°C) 1.41 (s, 9H, CMe_3), 1.48 (s, 9H, CMe_3), 1.51 (s, 9H, CMe_3), 1.54 (s, 9H, CMe_3), spectrum 4.2, 3.98 (dd, J_1 2.8, J_2 1.4 1H), 4.21 (t, J 1.4, 1H), 4.25 (dd, J_1 2.8, J_2 1.4, 1H), 4.48 (dd, J_1 3.2, J_2 1.6, 1H), 4.79 (dd, J_1 3.2, J_2 1.6, 1H), 5.29 (t, J 1.6, 1H), spectra 4.2 and 4.3: δ_C ($CDCl_3$) 26.9 (q), 28.3 (q), 31.4 (q) and 31.6 (q) (all $C(\underline{C}H_3)_3$; 30.8 (s, C-$\underline{C}(CH_3)_3$), 44.4 (s, C(O)-$\underline{C}(CH_3)_3$; 64.2 (2xd), 66.1 (d), 69.2(d), 69.7(d) and 70.1(d) (all ring C-H); 76.3 (s), 101.9 (s), 102.0 (s) and 103.5 (s) (all ring C); 211.3 (s, CO), spectrum4.4. Infra-red (CCl_4 solution): 1658 cm^{-1}, ν (C=O). Mass spectrum: m/z 438 (M^+), 338, 296.

Following the isolation of (1), the column was stripped with methanol, the solvent was removed, and the residue dissolved in dry CH_2Cl_2 (10 cm^3). This fraction was chromatographed on neutral alumina in 3 cm diameter dialysis tubing: of six bands

extracted with diethylether the first two were found to be homogeneous by t.l.c. The first band consisted of further (**1**), the second provided (**2**) (2.3 g, 2.9%) m.p. 220-221°C. NMR.; δ_H (C_6D_6) 1.23 (s, 9H, CMe_3), 1.27 (s, 9H, CMe_3), 1.33, (s, 9H, CMe_3), 1.34 (s, 9H, CMe_3), 4.16 (dd, J_1 2.6, $J_2$1.7, 1H), 4.39 (dd, J_1 2.6, J_2 1.7, 1H), 4.43 (dd, J_1 2.4, J_2 1.5, 1H), 4.73 (t, J 1.7, 1H), 4.75 (dd, J_1 2.4, J_2 1.5, 1H), 4.78 (t, J 1.5, 1H), spectra 4.5 and 4.6: δc ($CDCl_3$) 27.8(q), 28.2(q), 31.4(q) and 31.5(q) (all $C(\underline{C}H_3)_3$); 30.8(s) and 30.9(s) ($C\text{-}\underline{C}(CH_3)_3$); 44.4(s) and 44.6(s) ($C(O)\text{-}\underline{C}(CH_3)_3$); 69.5(d), 70.3(d), 70.4(2xd), 70.5(d), and 71.1(d) (all ring C-H);77.3(s), 105.5(2xs), 105.6(s) (all ring C); 208.9(s) and 210.6(s) (CO), spectrum 4.7. Infra-red (CCl_4 solution): 1661cm^{-1}, ν(C=O). Mass spectrum: m/z 466 (M^+), 409, 296.

4.2.3 Thionation of (1)

To a solution of (**1**) (2.0 g, 4.6 mmol, in dry CH_2Cl_2 (20 cm^3) and dry diethyl ether (200 cm^3) was added P_4S_{10} (45 g, 0.10 mol) with vigorous stirring. The mixture was then refluxed during 2 h., after which t.l.c. showed a single purple component. The mixture was filtered to small volume. Chromatography on alumina and elution with toluene gave purple (**3**) (0.82 g, 39%) m.p. 130-131°C. NMR.;δ_H (C_6D_6), 1.19 (s, 9H, CMe_3), 1.21 (s, 9H, CMe_3), 1.26 (s, 9H, CMe_3), 1.48 (s, 9H, CMe_3), 3.53 (s, 1H), 3.91 (s, 1H), 4.04 (s, 1H), 4.47 (s, 1H), 4.83 (s, 1H), 5.33 (s, 1H): δ_C ($CDCl_3$) 31.7 (s, $C\text{-}\underline{C}(CH_3)_3$), 31.9 (q), 32.2 (q), 32.3 (q) and 33.1 (q) (all $C(\underline{C}H_3)_3$); 51.8 (s, $C(S)\text{-}\underline{C}(CH_3)_3$); 64.9 (d), 66.8 (d), 67.5 (d), 71.4 (d), 72.7 (d) and 73.1 (d) (all ring C-H); 88.4 (s), 103.3 (s), 104.7 (s) and 106.8 (s) (all ring C); 258.7, (s, CS). Infra-red (CCl_4 solution): 1213 cm^{-1}, ν(C=S).

4.3 Results and Discussion

The ^{1}H NMR. spectrum of (1) comprised six signals, each of relative intensity one, in the cyclopentadienyl region, and four singlets, each of relative intensity nine, in the alkane region, spectrum 4.2. We assign these respectively to six distinguishable ring protons and to four distinguishable t-butyl groups, suggesting a total of four substituents each containing an Me_3C group. The ^{13}C NMR. spectrum shows four different CH_3 signals, but only one quaternary resonance assignable to $C(O)\underline{C}(CH_3)_3$: there are six CH resonances and four quaternary resonances in the cyclopentadienyl region, together with a single resonance assignable to $\underline{C}=O$, spectrum 4.4.

The ^{13}C spectrum is thus also consistent with the presence of four Me_3 groups per ferrocene nucleus. Of the four quaternary resonances in the cyclopentadienyl region, one has a chemical shift 76.3 close to the corresponding quaternary resonance (δ 76.7) in the monosubstituted trimethylacetyl ferrocene: the other three, in the range 101.9 - 103.5, are very similar to the quaternary ring resonance (δ 102.0[2]) in 1,1'-di-t-butylferrocene. These shifts point clearly to the presence of one Me_3CCO substituent and three Me_3C substituents per ferrocene nucleus.

Under very high resolution, spectrum 4.3, each of the six ^{1}H signals in the cyclopentadienyl region exhibits multiplet splitting, and this region of the spectrum was readily assigned as a pair of independent AMX systems, showing first-order behaviour at 300 MHz. The splitting pattern serves to establish the substituent pattern since it is well established that in poly-substituted ferrocenes $^{3}J(H\text{-}H)$ lies typically in the range 2.4 - 2.8 Hz, while

^{4}J(H-H) lies typically in the range 1.4 - 1.7 Hz.[3] Each of the six resonances in the range δ 3.8 - 5.3 showed the presence of two and only two coupling constants, so that each ring is disubstituted. Analysis of the spectrum showed that the resonances at δ 3.98, 4.21 and 4.25 constituted one AMX system, and that those at δ 4.48, 4.79, and 5.29 constiuted a second AMX system. Each AMX system was characterised by one large and two small coupling constants, pointing to a 1,3-substitution pattern in each ring. In the ring carrying two different substituents, the ring proton pattern follows straightforwardly. For the ring carrying two Me_3C substituents, the observation of an AMX system for the ring protons is due to the interaction with the other substituted ring, since at all orientations the three ring protons on the ring are non-equivalent. It follows that the molecule is chiral.

The evidence of the ^{1}H and ^{13}C spectral analyses thus demonstrates the constitution for (**1**) shown below. The second product isolated (**2**), shows features in the ^{1}H NMR. spectrum very similar to those of (**1**), namely four distinguishable $C(CH_3)_3$ signals, and six resonances each attributable to a single proton, assignable to a pair of independent AMX spin systems. The resonances at δ 4.16, 4.39 and 4.73 form one such system, characterised by one large and two small coupling constants while those at δ 4.43, 4.75 and 4.78 form the second, with a similar coupling pattern. As for (**1**) the pattern of coupling constants shows each ring to be 1,3-disubstituted. The ^{13}C NMR. spectrum of (**2**), shows the presence of two $C(CH_3)_3$ groups directly bound to the ferrocene nucleus[2] and two $C(O)$-$C(CH_3)_3$ groups, spectra 4.4 and 4.7. In this spectrum, all of the quaternary resonance of the four $\underline{C}(CH_3)_3$ were resolved: two of the shifts are characteristic of C_5H_4-$\underline{C}(CH_3)_3$ and two are

characteristic of C_5H_4-C(O)-$\underline{C}(CH_3)_3$. Hence in (**2**), the ferrocene nucleus carries two $C(CH_3)_3$ and two $C(O)C(CH_3)_3$ substituents. These observations suggest that (**2**) is not a single compound, but rather a mixture of two diastereomers in the ratio of 1:2.4 (taken from the ratio of the δC=O peak heights).

Direct alkylation of ferrocene generally produces a complex mixture of products[4], but for dialkyl products, the 1,1' isomer generally predominates over the 1,3 isomer[5,6]: thus direct alkylation with $(CH_3)_3CCl/AlCl_3$ gave a 1,1' to 1,3 isomer ratio of 4.3. The observed 1,3 pattern of disubstitution in each ring of (**2**) is thus consistent with alkylation followed by acylation so that the two rings have similar substitution patterns such that all the ring protons and ring carbons will be distinguishable by NMR., as will the two substituents of each type. Thionation[7] of (**1**) using P_4S_{10} provided the corresponding thioacyl derivative (**3**). The ^1H NMR. spectrum of (**3**) showed the same overall pattern as that of (**1**) and (**2**), with four distinguishable Me_3C resonances and six distinguishable ring CH resonances: however, even under high resolution, the higher line width prevented analysis of the multiplet structure of the cyclopentadienyl CH resonances. The ^{13}C NMR. spectrum of (**3**) again showed four $C(CH_3)_3$ signals, although as for (**1**), only a single resonance assignable to C(S)$\underline{C}$ $(CH_3)_3$ [δ 51.8, cf 51.5 in $(C_5H_5)Fe(C_5H_4CS\underline{C}Me_3)$]: there are six C-H resonances in the cyclopentadienyl region, together with four quaternary ring resonances, of which one (δ 88.4) is very close to that (δ 87.9) in $(C_5H_5)Fe(C_5H_4CSCMe_3)$ while the other three, in the range δ 103.3 - 106.8 are characteristic of ring carbon directly bound to a t-butyl substituent.[3] These spectra are fully in accord with the constitution for (**3**) shown above.

The occurence of both alkylation and acylation is undoubtedly due to the decarbonylation of the acylium ion formed in the initial reaction between Me_3CCOCl and $AlCl_3$, (Figure 4.1):

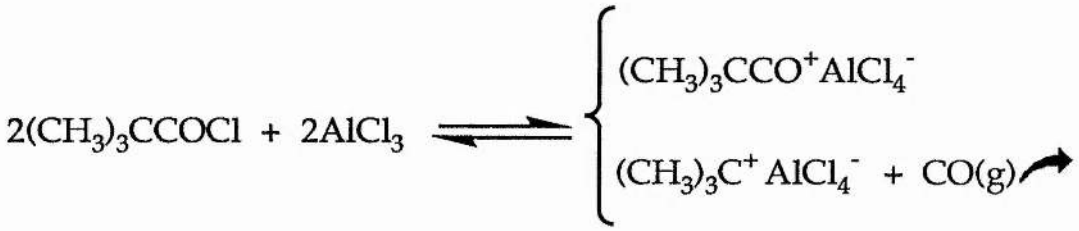
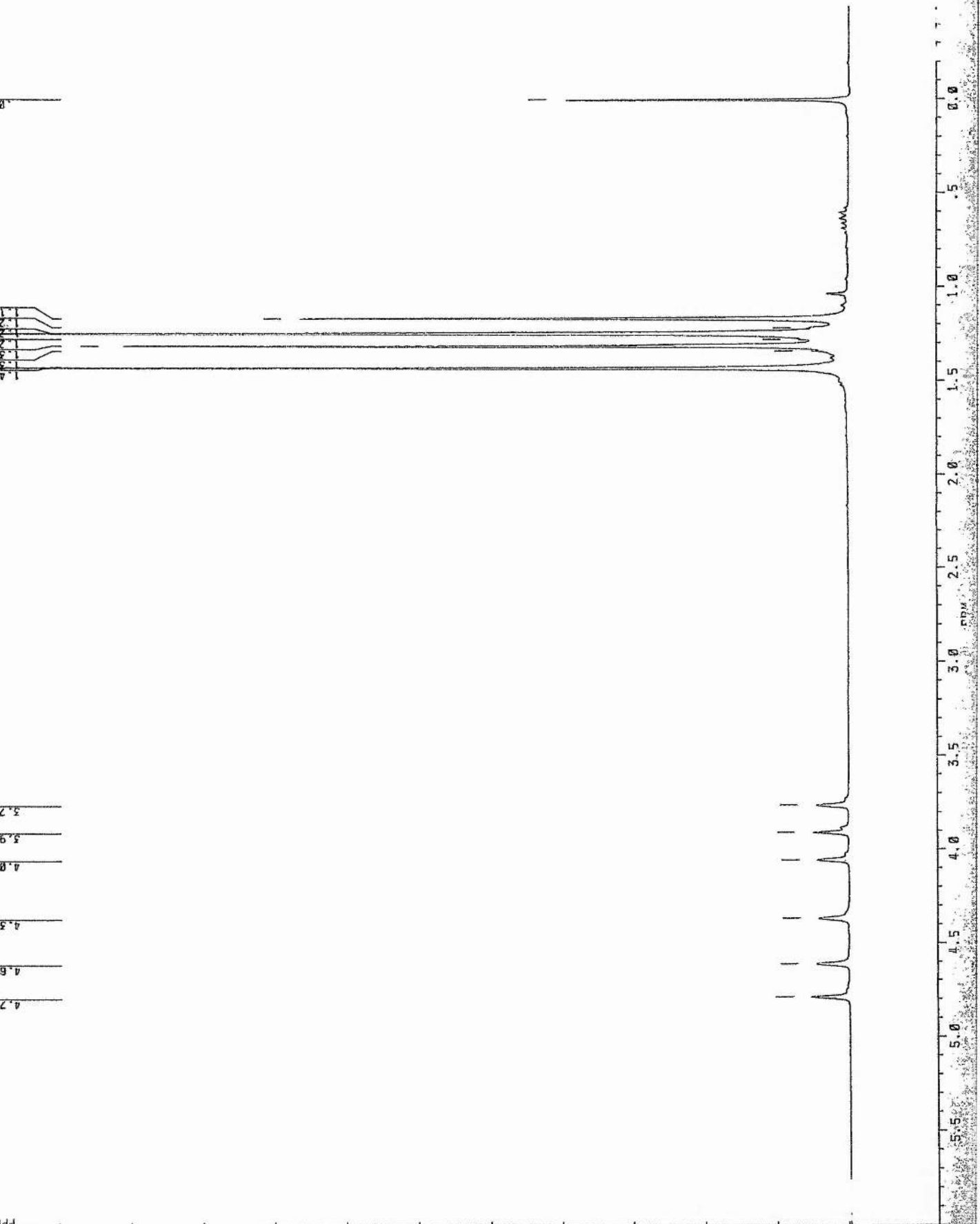


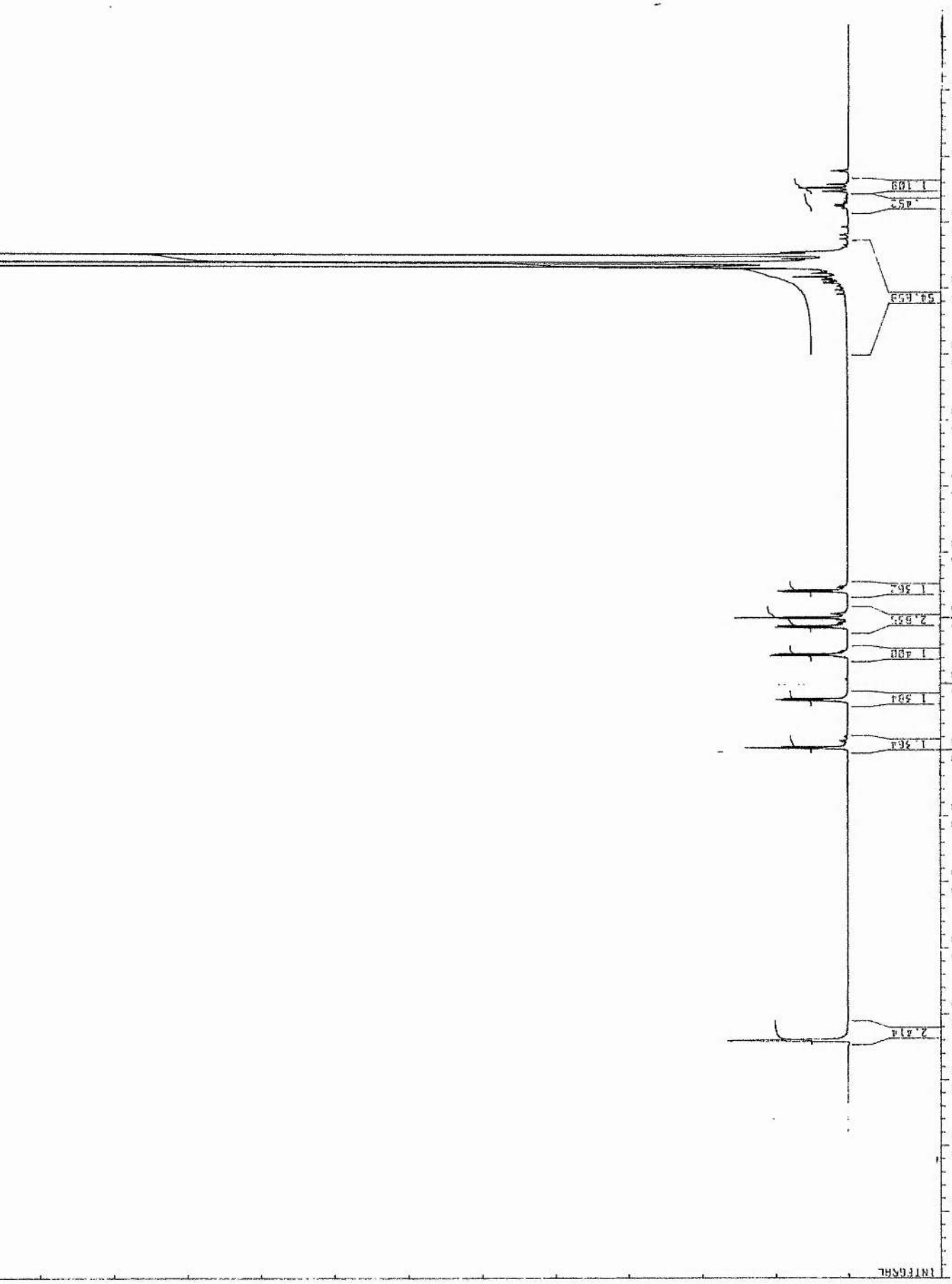
Figure 4.1

The 1,3-disubstitution pattern in the asymmetrically substituted rings of (1) and (2) is consistent with a mechanism of alkylation followed by acylation. The existence of two electrophiles, Me_3C^+ and Me_3CCO^+, in the reaction mixture is likely to lead to a mixture of products, as shown by t.l.c. examination (see Experimental part). Analysis by GC-MS has shown the presence of $[C_5H_3(CMe_3)_2]_2$ Fe and other unidentified products as well as (1) and (2) in the reaction mixture, but these compounds have not yet been isolated in pure homogeneous form. An example has been recorded in the literature[8] where use of Me_3CCOCl under Friedel-Crafts condition gave both alkylation and acylation in a single product; the reaction of benzene under certain conditions provides p-Me_3C $C(O)C_6H_4CMe_3$.

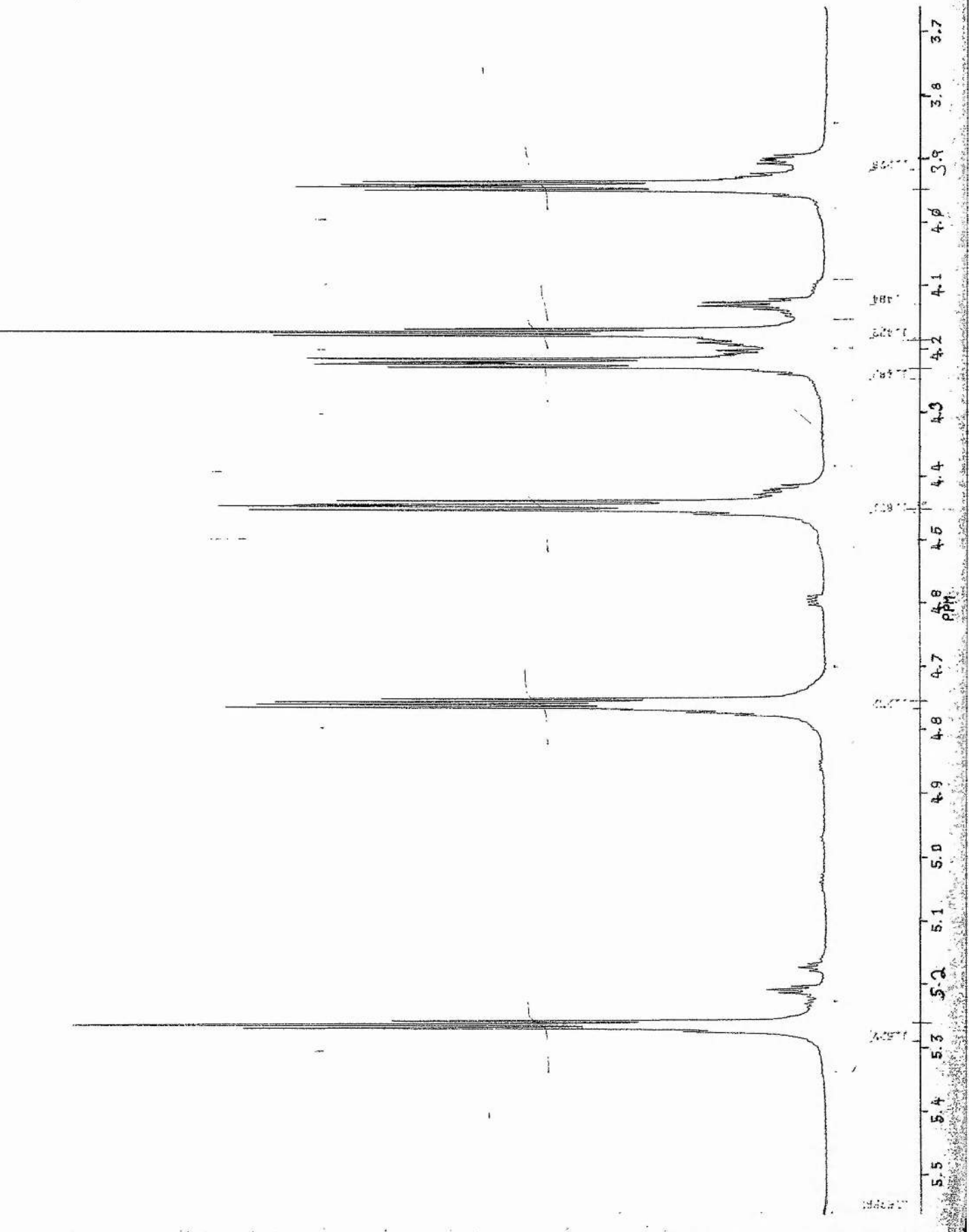
Spectrum 4.1 ^{1}H NMR spectrum of (1) in $CDCl_3$ solution at20°C with TMS at internal standard.



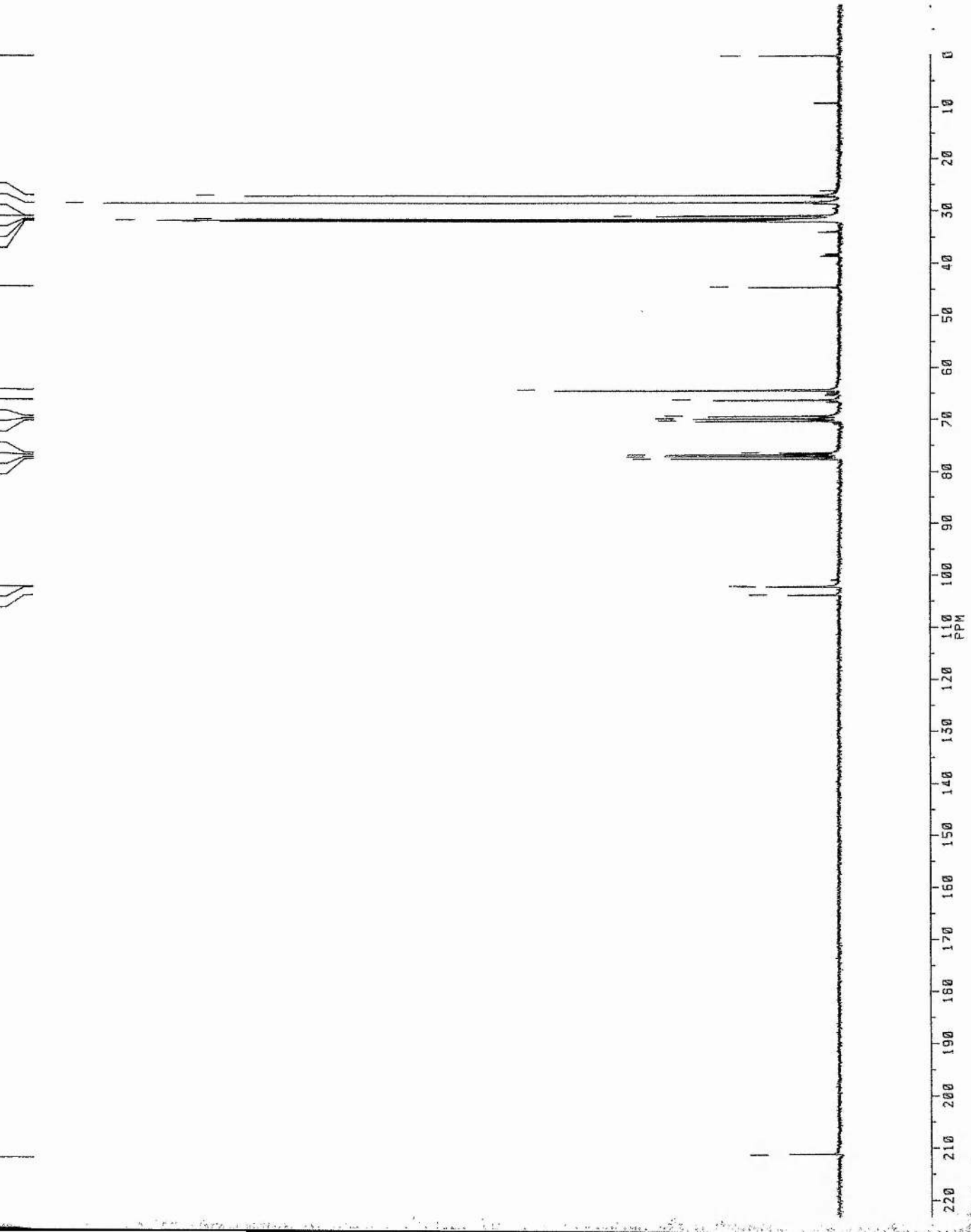
Spectrum 4.2 ^{1}H NMR spectrum of (1) in C_6D_6 solution at80°C with TMS at internal standard.



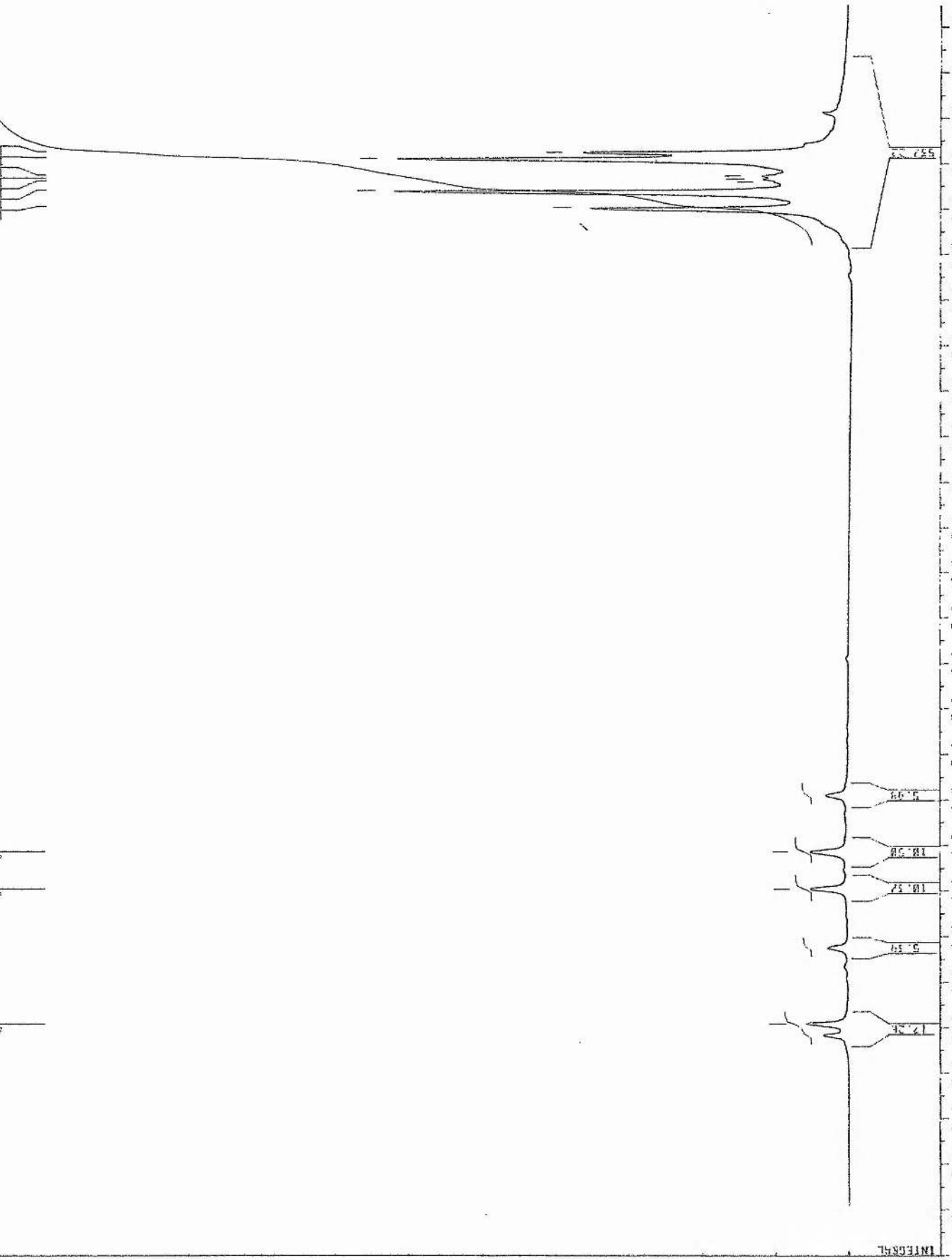
Spectrum 4.3 Expansion of Spectrum 4.2.



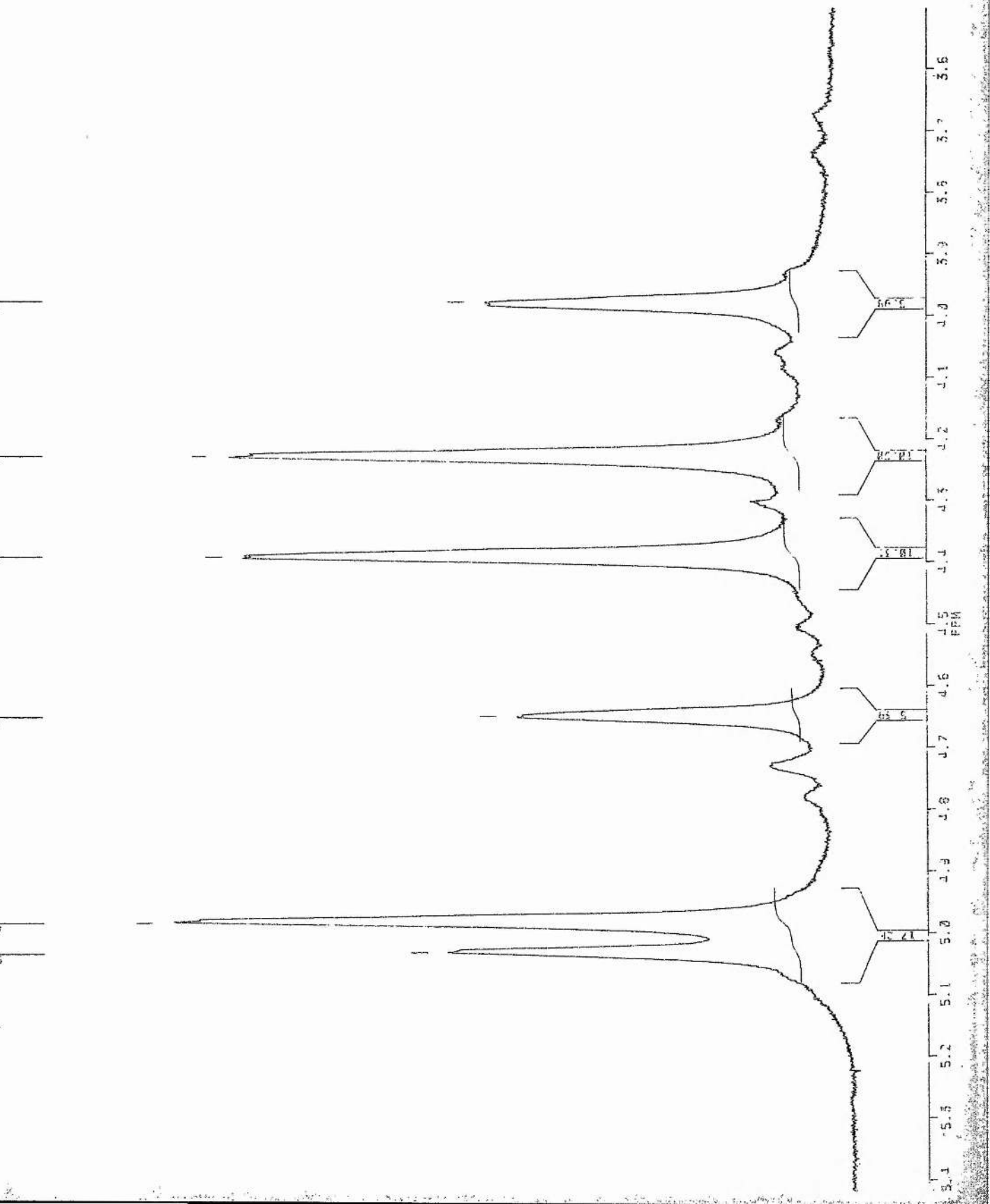
Spectrum 4.4 ^{13}C NMR spectrum of (1) in $CDCl_3$ solution at20°C with TMS at internal standard.



Spectrum 4.5 ^{1}H NMR spectrum of (2) in C_6D_6 solution at20°C with TMS at internal standard.

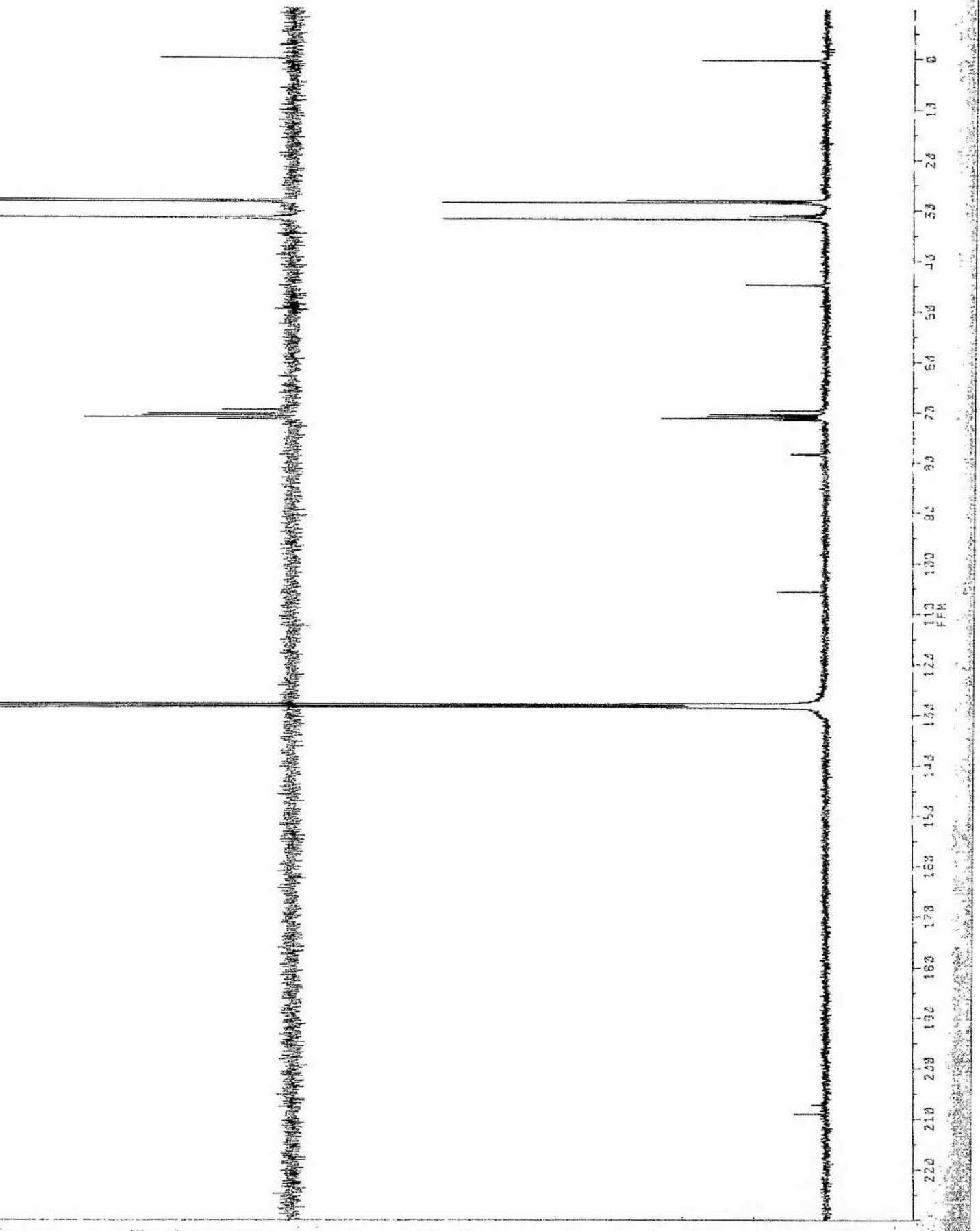


Spectrum 4.6 Expansion of Spectrum 4.5.



Spectrum 4.7 ^{13}C NMR spectrum of (2) in C_6D_6 solution at20°C with TMS at internal standard, with

D E P T spectrum of (2) showing the C-H on the top trace, with the quaternary and C-H on the lower trace.



4.4 References Chapter Four

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3. K. L. Rinehart, Jr., D. E. Bublitz, and D. H. Gustafson, *J. Am. Chem. Soc.*, 1963, **85**, 970.

4. D. E. Bublitz and K. L. Rinehart, Jr., *Org. React.*, 1969, **17**, 1.

5. E. W. Neuse and D. S. Trifan, *J. Am. Chem. Soc.*, 1962, **84**, 1850.

6. T. Leigh, *J. Chem. Soc.*, 1964, 3294.

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Chapter Five.
The Flash Vacuum Pyrolysis of various substituted ferrocenes.

5.1 Introduction.

Glidewell [1] has reported that (**1**) yielded a tetranuclear species (**2**) under FVP conditions at 500°C (Figure 5.1)

$$[(C_5H_5)Fe(CO)_2]_2 \xrightarrow{FVP} [(C_5H_5)Fe(CO)]_4$$

(**1**) (**2**)

Figure 5.1

whereas the molybdenum analogue (**3**) lost CO ligands to yield the triply bonded molybdenum complex (**4**) at 350°C. Under more harsh conditions the only product recovered was molybdenum hexacarbonyl (**5**) (Figure 5.2).

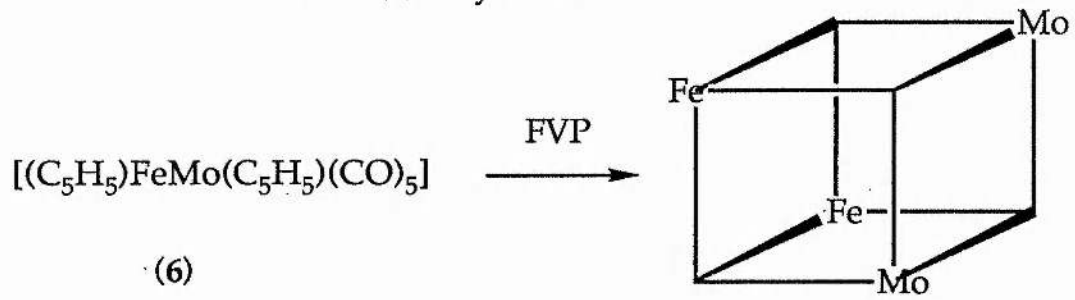
$$[(C_5H_5)Mo(CO)_3]_2 \xrightarrow{FVP} [(C_5H_5)Mo(CO)_2]_2 \xrightarrow{FVP} [Mo(CO)_6]$$

(**3**) (**4**) (**5**)

Figure 5.2

As an extention of this work the mixed metal dimer (**6**) (Figure 5.3) was synthesised in order to investigate its FVP behaviour. If (**6**)

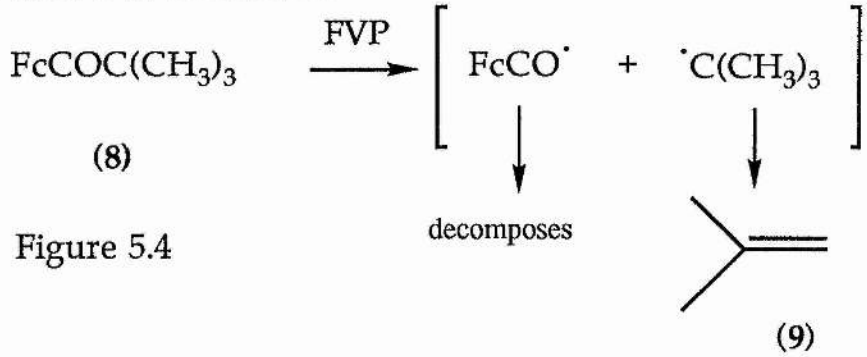
were to follow a similar reaction pathway as (1) (Figure 5.3) then the mixed metal tetramer (7) may be the result.



(6) (7)

Figure 5.3

Also reported[1] was the cleavage of pivaloyl ferrocene (8) ($FcCOC(CH_3)_3$), yielding the stabilised t-butyl radical which rearranges to isobutene (9), (Figure 5.4). As an extention of this, a range of acyl ferrocenes was prepared (see chapter 2) in order to investigate their FVP behaviour.



(8) (9)

Figure 5.4

5.2 Experimental

5.2.1 Preparation[3] of $C_5H_5Mo(CO)_3Na$

To sodium sand (1.27g;55 mmol) in THF (100 cm^3) was added, dropwise with stirring, some freshly cracked cyclopentadiene (11.1g;0.17 mol) which was dissolved in THF (100 cm^3). Then $Mo(CO)_6$ (10.6g;0,04 mol) was added and the mixture was refluxed for 12 hours. The solvent was evaporated to leave a yellow-gold powder of $C_5H_5Mo(CO)_3Na$ (10.5g;98%) .

5.2.2 Preparation[4] of $C_5H_5Fe(CO)_2I$

$[C_5H_5Fe(CO)_2]_2$ (50g;0.14 mol), iodine (50g;0.197 mol) and 250 cm^3 methylene chloride were refluxed together for 2 hours, the mixture was cooled , then washed with a solution of $Na_2S_2O_3.5H_2O$ (aq). The organic layer was separated, dried and evaporated to give black shiny crystals of $C_5H_5Fe(CO)_2I$ (81.4g;95%) .ν(C=O) 2043(s),2001(s) (CCl_4 solution).

5.2.3 Preparation[3] of $C_5H_5MoFeC_5H_5(CO)_5$. (6)

$C_5H_5Mo(CO)_3Na$ (3.81 g;14.2mmol), and ethanol (40 cm^3) were stirred until the mixture became homogeneous. Petroleum (100 cm^3 40/60°) was added and then $C_5H_5Fe(CO)_2I$ (2.0g;6.3mmol) together with a further portion of petroleum (100cm^3 , 40/60°). After stirring the whole for 45 minutes the solvent was evaporated and the red residue was extracted with toluene. The combined organic extracts were reduced to a small volume, and chromatography on alumina eluting with toluene gave a red band which was evaporated to yield a red powder of $C_5H_5MoFeC_5H_5(CO)_5$.(6) (1.14g;45.2%) Mpt. 209-210°C (lit[3]. mpt. 209°C) calculated for $C_{15}H_{10}O_5FeMo$ 42.69%C

2.39%H and found 42.89%C 2.42 %H. Infra-red (Cyclohexane solution)

observed 2039,2000,1958,1944,1919,1903,1888 cm^{-1}

(Lit [3] 2040,2002,1959,1944,1919,1902,1887 cm^{-1})

NMR (C_6D_6):δ_H 5.02 and 4.48 (s,5H,C_5H_5):δ_C 227.6 (C=O), 92.5 and 86.1 (C-H, C_5H_5).

5.2.4 Preparation of a mixture of the two dimers

$[C_5H_5Fe(CO)_2]_2$ (1.77g;5mmol) and $[C_5H_5Mo(CO)_3]_2$ (2.45g;5mmol) were dissolved in benzene and stirred for 20 minutes, then evaporated to dryness, the mixture being a brick-red crystalline solid.

5.3 Discussion

$[(C_5H_5)Fe(CO)_2Mo(C_5H_5)(CO)_3]$ (**6**) was readily prepared by the reaction of $[CpFe(CO)_2I]$ and $[CpMo(CO)_3Na]$ by the method of King[3]. The resulting red powder was stable to air, but when in solution it proved to be air sensitive and the red solution quickly decomposed.

When the mixed metal dimer (**6**) was subjected to FVP it was found to be wholly involatile under reduced pressure at 200°C, and this involatility made FVP experiments impossible. Heating (**6**) to 350°C in the solid phase caused it to decompose, giving a mixture of the two parent carbonyls $[CpFe(CO)_2]_2$ (**1**) and $[CpMo(CO)_3]_2$ (**3**) as noted previously by King [3].

An equimolar mixture of (**1**) and (**3**) was prepared and samples were subjected to FVP. At a furnace temperature of 350°C there was no change of the starting mixture recovered;at 400°C, (**4**) was noted by

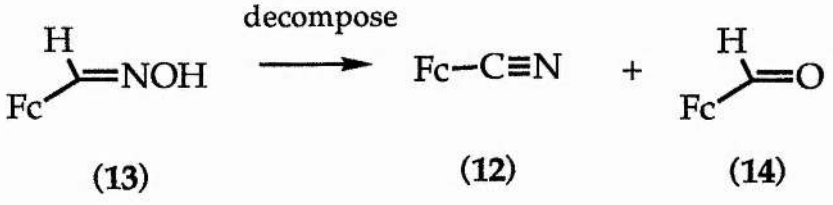
t.l.c examination, (1) was unchanged;450℃ there was a mixture of, (1),(2),(3),(4),(Figures 5.1 and5.2) a trace of ferrocene and a few white crystals of $Mo(CO)_6$; at 500℃, the amount of (2) had increased, as had the amounts of ferrocene and $Mo(CO)_6$, the amount of (4) had dropped; at 550℃, ferrocene and $Mo(CO)_6$ were the only products recovered. The materials were all identified by t.l.c comparison with authentic samples.

These observations indicate that neither of the individual dimers interacts with the other, but rather that each behaves as it would in the absence of the other.

Acyl ferrocenes were readily prepared by the Friedel Crafts reaction of ferrocene and the appropriate acid chloride using aluminium trichloride as the catalyst. These preparations have been explained in chapter 2.

A sample of (8) has been reported[1] to cleave the FcCO--R bond , the driving force of this reaction was possibly the formation of the stabilised t-butyl radical. In the cases of the other acyl ferrocenes (table 5.1) the radicals thus produced would possess no such stabilisation and their formation would be unfavorable.

A sample of the oxime (13) was found to be involatile under reduced pressure at 200℃, the sample decomposed, when melted at 200℃, to a mixture of cyanoferrocene (12) and formylferrocene (14).(Figure 5.5)

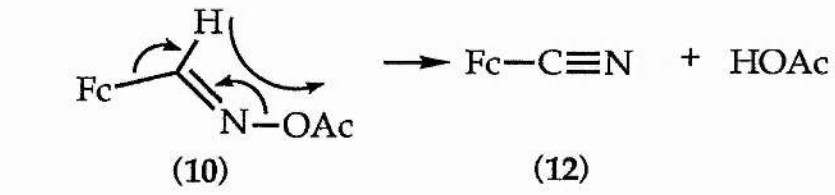


(13) (12) (14)

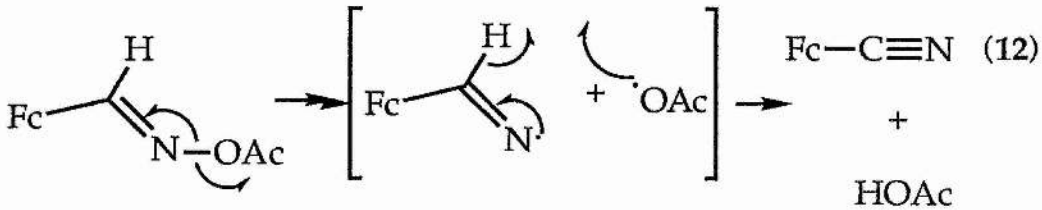
Figure 5.5

The formation of (14) can be exlained if the water, produced in the dehydration to cyanoferrocene (12), was to react with (13) under the conditions of the reaction and then undergo elimination of NH_2OH to give ferrocenecarboxaldehyde (14).

Ferrocenecarboxaldoxime acetate (**10**) was synthesised [2] with the intention of producing the reactive intermediate (**11**) (Figure5.6)



(**10**) (**12**)



(**10**) (**11**)

Figure 5.6

The formation of cyanoferrocene on FVP of ferrocenecarboxaldoxime acetate may proceed in two mechanistic pathways as shown above (Figure 5.6), proceeding by simple elimination of acetic acid or by the radical pathway.

Performing the FVP on the oxime acetate of acetyl ferrocene would remove the possibility of a simple elimination and this may lead to materials derived from radicals.

5.4 References Chapter 5

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2) J.K.Lindsay and C.R.Hauser, *Journal of Organic Chemistry*, **22**,(1957),355

3) R.B.King, P.M.Treichel, F.G.A.Stone, *Chemistry and Industry*, (1961), 747.

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Table 5.1 Flash Vacuum Pyrolyses on a variety of Substituted Ferrocenes.

Derivative	Temperatures (℃) and (%) recoveries.					
FcCOH	650 (98)	670 (82)	700 (69)	720 (27)	750 (0)	
$FcCOCH_3$	200 (92)	400 (94)	600 (90)	700 (65)	750 (0)	
$FcCOCH_2CH_3$	550 (87)	575 (81)	600 (75)	625 (75)	650 (65)	675 (6)
$FcCOCH(CH_3)_2$	650 (92)	680 (61)	700 (36)	750 (0)		
$FcCOC(CH_3)_3$	600 (91) [7.6]	650 (88) [10.5]	700 (40) [84.2]	800 (0) [0]		
$FcCOC_6H_5$	550 (92)	600 (84)	650 (86)	700 (73)	730 (16)	750 (5)
$FcCOCH_2C_6H_5$	600 (93)	650 (84)	700 (48)	750 (0)		
$Fcd(COCH_3)_2$	650 (86)	680 (64)	710 (52)	750 (21)	800 (0)	
$Fcd(COC_6H_5)_2$	Involatile					

Fc is $C_5H_5FeC_5H_4$- Fcd is $-C_5H_4FeC_5H_4$-

5.4 References Chapter 5

1) C.Glidewell, J.McKechnie. *Journal of Organometallic Chemistry*, **321**,(1987), C21-C23.

2) J.K.Lindsay and C.R.Hauser, *Journal of Organic Chemistry*, **22**,(1957),355

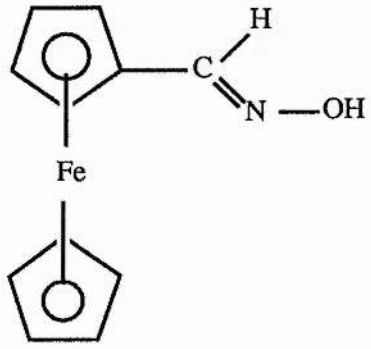
3) R.B.King, P.M.Treichel, F.G.A.Stone, *Chemistry and Industry*, (1961), 747.

4) J.Kleinberg, (Ed.) *Inorganic Syntheses* , **VII**, p110.

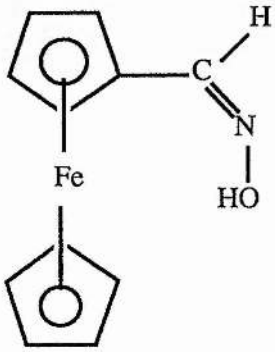
Chapter Six
Crystal and Molecular Structure of the Low-melting Form of Ferrocenecarboxaldoxime.

6.1 Introduction

Ferrocenecarboxaldoxime, $(C_5H_5)Fe(C_5H_4CH{=}NOH)$, has been reported[1] to exist in two crystalline modifications, denoted α, m.p. 96-99°C and β, m.p. 155-157°C. These two forms have been tentatively assigned the E and Z configurations (1) and (2) respectively, although there is little evidence to support this.



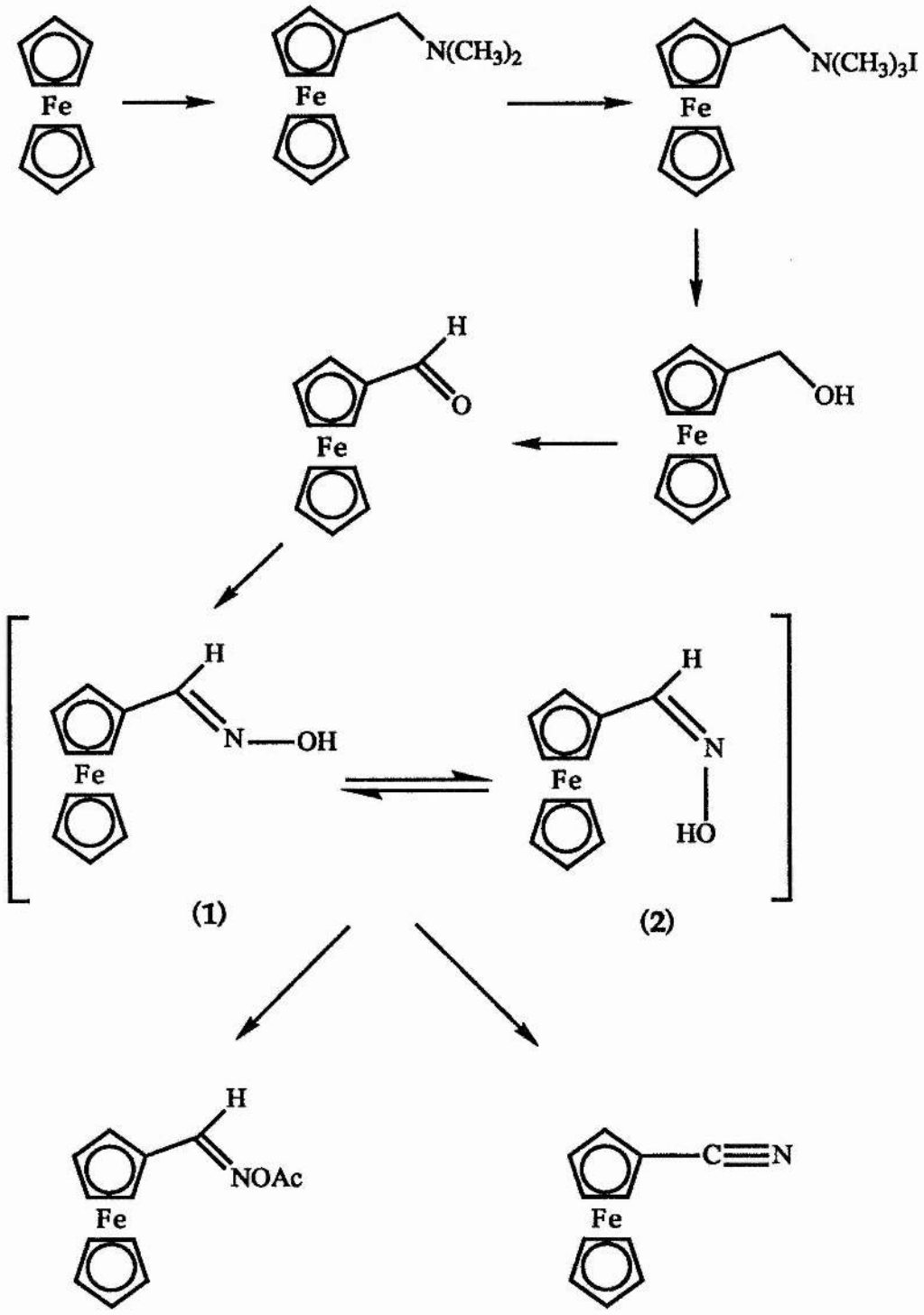
(1)



(2)

An X-ray structure analysis of the low-melting form shows it to contain both E and Z isomers, co-existing as disordered occupants of common molecular sites.

Scheme 6.1 Showing the synthetic pathway from ferrocene to ferrocenecarboxaldoxime acetate and cyanoferrocene.



6.2 Experimental

The reaction pathway is given in scheme 6.1 after modifications to reference[2]. The ^{1}H NMR and ^{13}C NMR data are recorded in tables 6.1 and 6.2 respectively.

6.2.1 Preparation of N,N,N',N',-Tetramethyldiaminomethane

Formaldehyde (162g, 37/40 % aqueous; 2 mol) and dimethylamine (722g, 25% aqueous; 4 mol) were pre-cooled in an ice-bath. The dimethylamine was added dropwise to the stirred solution of formaldehyde. The mixture was stirred for a further hour in the ice-bath, and solid potassium hydroxide salted out the organic material, and the mixture was then warmed to room temperature. The organic material was separated, dried over potassium hydroxide, filtered, and distilled to give 174.06g of N,N,N',N',-tetramethyldiaminomethane (85% yield), bpt 82-83 $^{\circ}C$ (lit[2]. 82-84 $^{\circ}C$).

6.2.2 Preparation of N,N-dimethylaminomethylferrocene

N,N,N',N'-tetramethyldiaminomethane (25.5g : 0.25mol), paraformaldehyde (7.9g; 0.26 mol) and glacial acetic acid (190 cm^3) were heated together with stirring until homogeneous, then ferrocene (93g;0.5 mol) was added with stirring and the mixture refluxed. After 5 hours the reaction mixture was cooled and 500 cm^3 of water added, precipitating unreacted ferrocene which was filtered off and washed with dilute acetic acid, then water. To the combined filtrate and washings sodium hydroxide solution (300cm^3 0.5 mol dm^{-3}) was added and the solution extracted into ether, the organic extract washed with water, dried, filtered and evaporated giving of N,N-dimethylaminomethylferrocene (41%;51g) (bpt. 91-93°C, 0.45 mmHg).

6.2.3 Preparation of $FcCH_2N(CH_3)_3I$

To an ice-cooled solution of N,N-dimethylaminomethylferrocene (51.2g:0.21mol) in 50cm^3 absolute methanol was added dropwise a solution of methyl iodide (45.2g;0.32mol) in 50 cm^3 absolute methanol.The mixture was then refluxed for ten minutes, cooled and 500 cm^3 ether added with stirring, precipitating out the yellow product. This was filtered and washed with ether to give the methiodide, $FcCH_2N(CH_3)_3I$ (79.6g;78%) as yellow plates. Mpt.. 210-220°C(decomp) (lit .mpt 220°C (decomp))

6.2.4 Preparation of $FcCH_2OH$

$FcCH_2N(CH_3)_3I$ (20 g;0.052 mol) was added to aqueous sodium hydroxide (200cm^3,1mol dm^{-3}) and refluxed for 2 hours, cooled, then extracted with 200cm^3 diethyl ether. The organic fraction was then washed with water, dried, filtered and evaporated, to give a yellow crystalline solid of ferrocenylcarbinol (8.00g:71%) .Recrystallization from hexane gave an analytical sample mpt. 81-82°C. Calc. 61.1 C, 5.6 H,found. 61.8 C,5.9 H.

6.2.5 Preparation of "active MnO_2"

$MnSO_4.4H_2O$ (1110g;5mol) in water (1500 cm^3) and NaOH (40% aqueous,1170 cm^3:11.7 mol) were added simultaneously to a hot stirring solution of $KMnO_4$ (960g; 6.1 mol) in water (6000 cm^3). After stirring for a further hour the fine brown precipitate of "MnO_2" was filtered off and washed with water until the washings became colourless. The solid was dried in an oven at 110°C for 1 hour and then ground to a fine powder to give the MnO_2 (803g;92%).

6.2.6 Preparation of FcCHO.

To an ice cooled solution of ferrocenylcarbinol (8.00 g) in 50 cm^3 methylene chloride was added 'active ' MnO_2 (40 g). After 12 hours the mixture was filtered through Hyflo, and the filtrate evaporated to give ferrocene carboxaldehyde as a red powder (7.50 g:94%) . Mpt 130-132°C.Calc. 61.7 C, 4.7 H,found. 62.1 C 4.9 H

6.2.7 Preparation of FcCHNOH

To FcCHO (11.22g; 0.052 mol) in 75 cm^3 ethanol was added $NH_2OH.HCl$(5.2 g;0.075mol) dissolved in 7 cm^3 water and NaOH (9.6 g;0.23 mol) pellets. After refluxing for 2.5 hours the mixture

was cooled and 500 cm^3 water was added. The mixture was filtered through Hyflo and the filtrate cooled in ice; CO_2 gas was slowly bubbled through, precipitating the oxime as copper coloured powder.(8.48g;70.6%). Recrystallisation from hexane/ methylene chloride gave red-brown crystals. Mpt 97-98°C (lit: 96-99°C)[1] found C, 58.1; H, 5.0; N, 5.9: $C_{11}H_{11}FeNO$ requires C, 57.7; H,4.8; N, 6.1%. For purposes of identification, the oxime was also converted into the corresponding nitrile[1], m.p. 106-108°C (lit: 107-108°C [1]), found C, 62.8; H,4.3; N, 6.7: $C_{11}H_9FeN$ requires 62.6; H, 4.3; N, 6.6%; and into the oxime acetate [2], m.p. 80-81°C (lit: 80-81°C [2]), found C, 57.5; H, 4.8; N, 5.1: $C_{13}H_{13}FeNO_2$ requires C, 57.6; H, 4.8; N, 5.2%.

Spectroscopic data for Ferrocenecarboxaldoxime: NMR ($CDCl_3$) δ_H 4.19 (s,5H,C_5H_5), 4.33 (m,2H) and 4.55 (m, 2H) (C_5H_4), 8.01(s,1H, = C(H)), 9.5 (br,s,1H, NOH); δ_c 67.6(d), 70.0(d), 76.1(s) (C_5H_4), 69.2(d) (C_5H_5), 149.9(d), (=C·). Infrared(/cm^{-1}) (CH_2Cl_2), 3570 and 3271, υ(OH); 1656, υ(C = N). Mass spectrum m/z 229 $(M)^+$, 211 $(M-H_2O)^+$, 185 $(M-CH_2NO)^+$, 164 $(M-C_5H_5)^+$, 146 $(M-C_5H_5-H_2O)^+$, 138 $(C_5H_5Fe-OH)^+$, 121 $(C_5H_5Fe)^+$.

6.2.8 Preparation of β-ferrocenecarboxaldoxime (FcCHNOH)

A solution of FcCHO (3.0g;14 mmol) and $NH_2OH.HCl$ (1.40g;42mmol) in 20 cm^3 of ethanol was refluxed for 2 hours, cooled and the black hydrochloride filtered off, suspended in diethyl ether and shaken with $NaCO_3$ (aq) solution. The red solution was filtered and evaporated to give β-oxime as an orange solid (mpt 145/6°C),(2.03g;63.2%) after recrystallisation from benzene the mpt became depressed to 138/9°C. Repeated recrystallisations failed to give crystals suitable for X-ray crystallography

6.2.9 Preparation of FcCHNOAc

To FcCHNOH (0.50g;2.2mmol) was added acetic anhydride (0.5 cm^3) shaken briefly and cooled in an ice bath. Immediately cold ethanol (5cm^3) was added and cold water introduced dropwise until the cloudiness produced just disappeared on shaking. After 24 hours in a refrigerator copper-brown crystals of $FcCHNOCOCH_3$ (0.30g;51%) were filtered off, $\nu_{(C=O)}$ 1775, $\nu_{(C=N)}$ 1611 cm^{-1}, calculated for $C_{13}H_{13}NO_2Fe$ 57.60%C, 4.83%H, 5.17%N and found 57.45%C , 4.80%H ,5.13%N. Mpt. 80-80.5°C

6.2.10 Preparation of ferrocenyl cyanide FcCN

FcCHNOH (1.0g;4.4mmol) and 10cm^3 acetic anhydride were warmed for 30 minutes. The mixture was poured onto ice and then extracted with methylene chloride. The organic material was separated, washed, dried and reduced to a small volume. Chromatography on alumina, eluting with methylene chloride gave a single yellow band, which on evaporation gave FcCN as a yellow powder (0.91g;98%). Recrystallisation from methylene chloride/hexane yielded yellow plates, mpt. 106-108°C ν(C=N) 2224 cm^{-1} (CCl_4 solution). Calculated for $C_{11}H_9FeN$ 62.6 %C, 4.3%H, 6.6%N and found 62.8%C, 4.3%H, 6.7%N.

Table 6.1 ^{1}H NMR Data for some Mono substituted ferrocenes in ppm[a]

Derivative[b]	Ferrocenyl group C_5H_4 (2H x 2)		C_5H_5 (5H)	Substituents
$FcCH_2N(CH_3)_2$	4.20	4.18	4.18	3.30(s,2H,CH_2) 2.18(s,3H,CH_3)
$FcCH_2N(CH_3)_3I$	4.78	4.45	4.15	3.15(s,9H,CH_3)
$FcCH_2OH$	4.40	4.25	4.15	4.13 (s,2H,CH_2) 1.80(br,s,1H,OH)
FcCHO	4.75	4.55	4.25	10.5 (s,1H,O=CH)
FcCHNOH	4.33	4.55	4.19	8.00 (s,1H,=CH) 9.5 (br,s,1H,NOH)
$FcCHNOCOCH_3$	4.68	4.45	4.22	2.20(s,3H,CH_3) 8.22(s,1H,=CH)

a/ All spectra were recorded in $CDCl_3$, with TMS as internal standard, at 20 °C.

b/ Fc is $C_5H_5FeC_5H_4$-

Table 6.2 ^{13}C NMR Data for some Mono substituted ferrocenes in ppm.[a]

Derivative[b]	Ferrocenyl group			Substituents		
	(d, C_5H_4)	(s, C_5H_5)	(q, C_5H_4)	(d, CH_2)	(t, CH_3)	others
$FcCH_2N(CH_3)_2$	69.8, 67.8	68.3	83.1	59.0	44.6	
$FcCH_2N(CH_3)_3I$	72.3, 70.7	69.6		67.0	52.6	
$FcCH_2OH$	68.3, 67.9	68.3	88.3	60.7		
FcCHO	73.1, 68.3	69.5	79.1			193.2 (C=O)
FcCHNOH	70.0, 67.6	69.2	76.1			149.9 (=C)
$FcCHNOCOCH_3$	71.2, 68.7	69.5	73.3		19.6	157.0 (=C) 168.6 (C=O)
FcCN	71.7, 70.7	70.6				120.2 (CN)

a/ All spectra were recorded in $CDCl_3$ at 20 °C, with TMS as internal standard.

b/ Fc is $C_5H_5FeC_5H_4$-

6.3 X-ray crystallography

Crystals suitable for X-ray examination were grown from solutions in CH_2Cl_2/light petroleum. A crystal of dimensions 0.06 x 0.54 x 0.58 mm was used. The data was collected and the structure solved by Dr. G.Ferguson at the Department of Chemistry and Biochemistry, University of Guelph, Guelph, Ontario, Canada, N1G 2W1.

6.3.1 Crystal data

$C_{11}H_{11}FeNO$, M = 229.06, monoclinic, a = 26.512(6), b = 12.798 (4), c = 12.855(2)Å, β = 114.37 (1)°, V = 3973(3)Å^3, Z = 16, D_c = 1.53 g cm^{-3}, μ(Mo-K_α) = 14.8 cm^{-1}, λ = 0.71073Å, F(000) = 1888,. Systematic absences; h 0 l absent if l = 2n + 1, hkl absent if h + k = 2n+1 allow the space group to be either C2/c or Cc: the former was chosen and confirmed by the successful refinement.

6.3.2 Data collection

Cell dimensions were determined by least-squares refinement using the setting angles of 25 reflections in the range $9^\circ \leq \theta \leq 15^\circ$. Intensity data were collected at 21°C on an Enraf-Nonius CAD4 diffractometer with graphite-monochromated Mo-K_α radiation in the ω-2θ scan mode; ω-scan rate 1-7° min^{-1}; ω-scan width (0.70+ 0.335 tanθ)°; the maximum value of 2θ was 54°. 4671 reflections were measured, of which 1768 were unique and not systematically absent, and 1762 had $F \geq 3\sigma(F)$. Lorentz and polarisation corrections were applied, together with a numerical absorption correction for which the maximum and minimum transmission coefficents were 0.921 and 0.542, and an anisotropic decay

correction for which the correction factors on I ranged from 0.972 to 1.104 with mean 1.041.

6.3.3 Structure solution and refinement

The structure was solved using the Patterson heavy atom method, which allowed the location of the positions of the two non-equivalent iron atoms. The remaining atoms were located in succeeding difference Fourier syntheses. All non-hydrogen atoms were refined anisotropically. Hydrogen atoms bonded to carbon were included in the refinement, but were restrained to ride on the atom to which they are bonded, with δ(C-H) = 0.95Å and fixed isotropic thermal parameters. A secondary extinction coefficient[3] refined to 1.0×10^{-6}; the final R values were R, 0.054; R_w, 0.079.

Scattering factors were taken from Cromer and Waber[4]: anomalous dispersion effects were included in F_c [5], and values of f' and f" were those of Cromer[5]. All calculations were performed on a PDP-11/73 computer using SDP-Plus[7].

Final refined atom coordinates are given in table 6.3, and selected bond lengths and angles in table 6.4. Perspective views of molecules A and B of the asymmetric unit, showing also the atom-numbering scheme, are in Figures 6.1 and 6.2.

Tables of hydrogen-atom coordinates are in appendix 6.1, anisotropic temperature factors are in appendix 6.2, and torsional angles are in appendix 6.3, and tables of observed and calculated structure factors are in appendix 6.4.

Figure 6.1 Perspective view of the ferrocenecarboxaldoxime molecule as found in sites A, showing the atom-numbering scheme and the E,Z disorder.

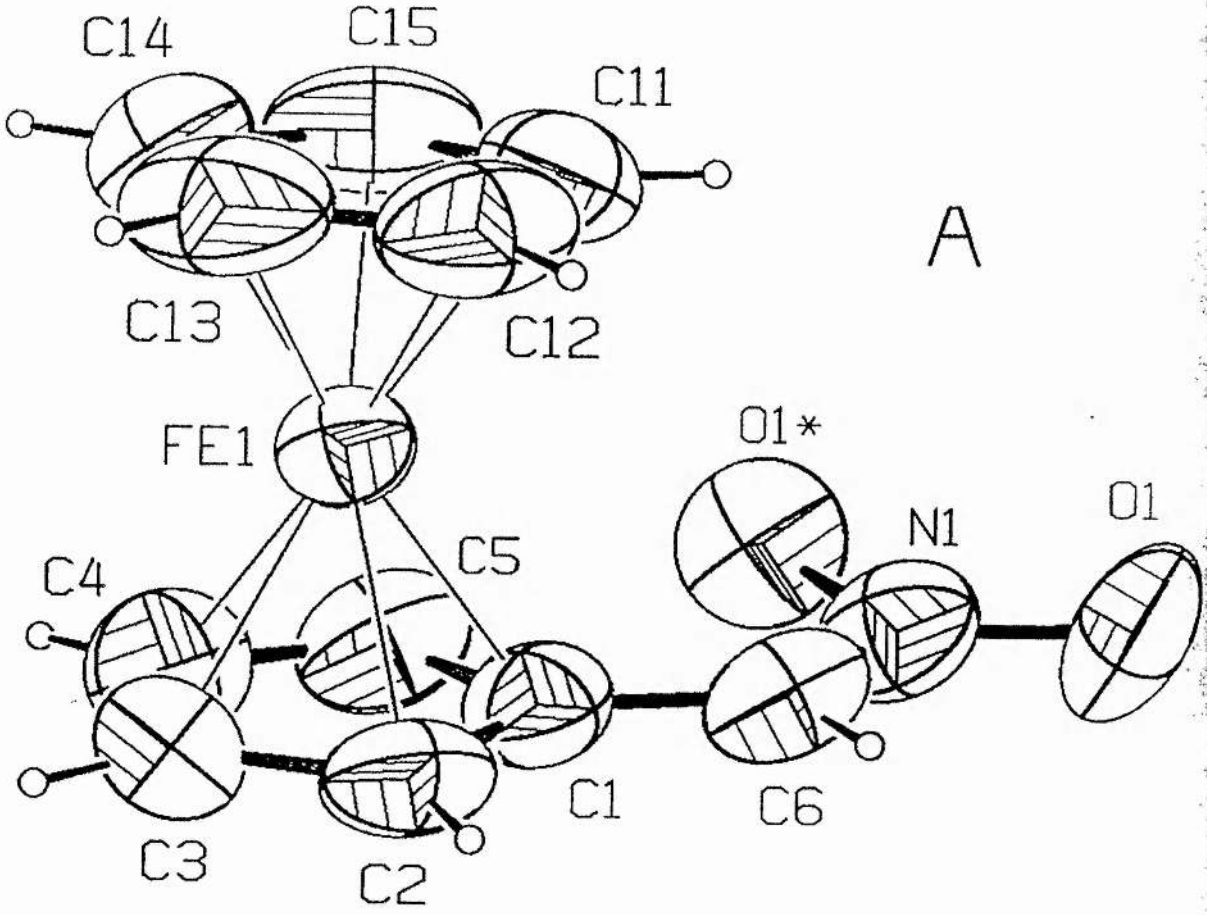
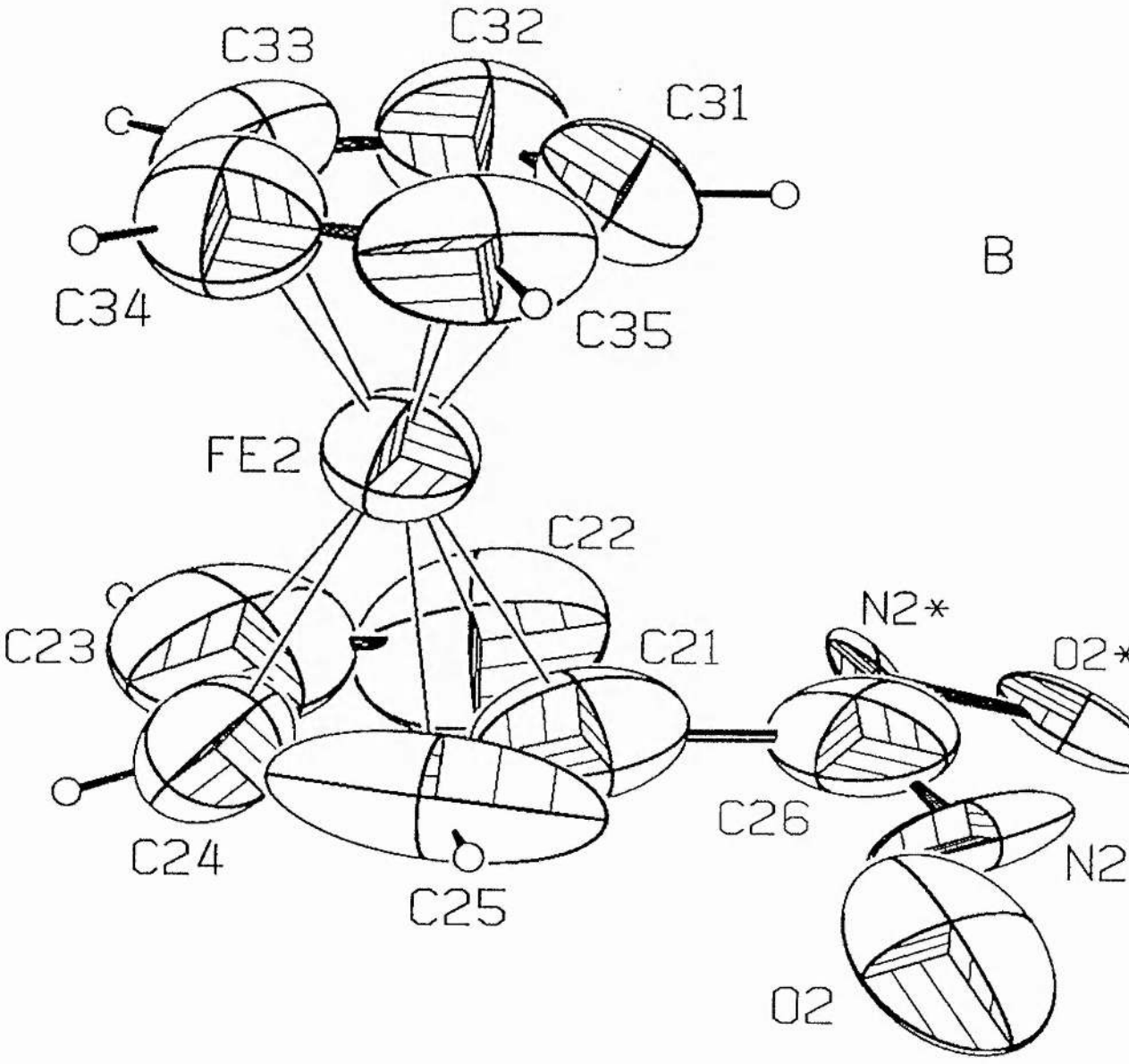


Figure 6.2 Perspective view of the ferrocenecarboxaldoxime molecule as found in sites B, showing the atom-numbering scheme the rotational and E,Z disorder.

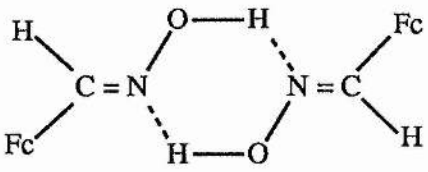


6.4 Results and Discussion

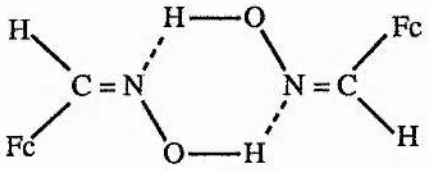
The unit-cell dimensions and the space-group demand that the asymmetric unit contains two molecules, in sites hereafter designated as A and B.

In sites A, the molecules exhibit disorder resulting from the presence of a mixture of E and Z isomers. Refinement of the occupancy factors for the oxygen atoms in the two alternative sites showed that 59% of the molecules in sites A have the E configuration while 41% have the Z configuration. The whole, disordered, C(H) = NOH group is effectively coplanar with the adjacent cyclopentadiene ring, the two C-C-N-O dihedral angles being 6.6(10)° and 177.9(6)°. Within the ferrocenyl fragment, the mean Fe-C distances for the two independent rings are identical within experimental uncertainty, but the mean C-C distance in the unsubstituted ring is marginally shorter than that in the substituted ring: this phenomenon has been noted previously[8]. The cyclopentadiene rings are twisted by 11.3° from the fully eclipsed conformation.

Pairs of site A molecules are related by a centre of inversion and are joined by weak hydrogen bonds: because of the E, Z disorder the net effect is the superimposition of 59% site occupancy by the E molecules, in pairs (**3**) upon 41% site occupancy by pairs of Z molecules, (**4**) [Fc = $(C_5H_5)Fe(C_5H_4)$].

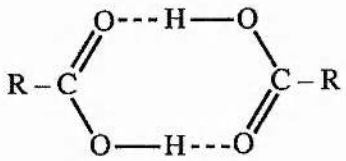


(3)



(4)

This pattern of hydrogen bonding resembles that widely found in carboxylic acid dimers $(RCOOH)_2$ (**5**), but having a six-membered ring rather than the eight-membered ring in (**5**).



(5)

The hydrogen bond distances N...O are 2.78(1)Å in (**3**), and 2.76(2)Å in (**4**), both significantly shorter than the sum 3.05Å of the van der Waals' radii[9]. The structural data and the infra-red spectrum both show that the hydrogen bonds are weak.

While the isolated monomers (**1**) and (**2**) of the E and Z isomers respectively are significantly different from one another, the dimers (**3**) and (**4**), based upon a common ring as the molecular core, are extremely similar in overall molecular shape and polarity: this, no doubt, contributes significantly to the existence of both isomers in the disordered form revealed by the X-ray analysis.

The molecules in sites B exhibit a more complex form of disorder. Firstly, refinement of the site occupancies for the C(H) =

NOH fragment showed that only 47% of the site B molecules are in fact Fc-C(H) = NOH, the other 53% being unsubsituted ferrocene; secondly, the molecules exhibit E, Z disorder as for the site A molecules, but now with 30% of the oxime molecules having the E configuration and 70% the Z configuration; thirdly, there is disorder caused by rotation about the exocyclic carbon-carbon bond, C21-C26; the best fit of the X-ray data was found with one rotamer having all E configuration, and the other rotamer all Z. The hydrogen bonding involving the oxime molecules in site B is also more complex than that for the A site molecules. One set of hydrogen bonds connects molecules related by the c glide-plane, with N...O distances of 3.11(6)Å, and another set of hydrogen bonds connects molecules related by the two-fold rotation axis, with N...O distances of 3.07(2)Å: all of these hydrogen bonds are very weak.

For the oxime substituents in site A, the C = N bond length, 1.269(10)Å is typical of that found in oximes[10], but the N-O distances, 1.438(10)Å in the E isomer and 1.478(14)Å in the Z isomer, are both significantly in excess of the upper quartile values, 1.401Å for a sample of twenty carboxaldoximes and 1.408Å for a sample of 67 oximes of all types[10]. The multiple disorder found for site B precludes meaningful comparisons of structural data.

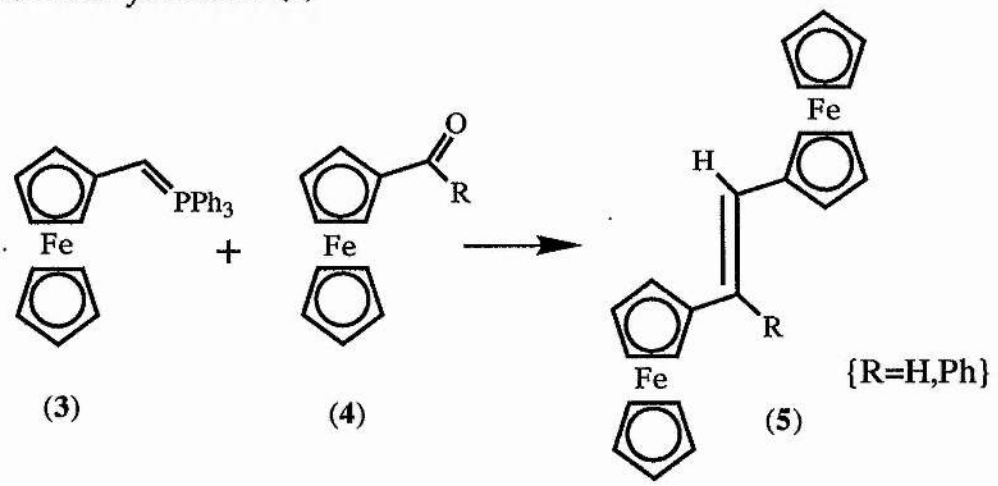
The structural results above were obtained from a sample of ferrocenecarboxaldoxime which was chromatographically homogeneous and apparently analytically pure, and which moreover provided two analytically pure derivatives, the nitrile and the oxime acetate. They show that the characterisation of the

isomeric forms of ferrocenecarboxaldoxime by the use of simple physical properties such as melting point is not secure, and that both E and Z isomers can co-exist in a single crystal.

Neither site A nor site B molecules of the oxime show any close contacts between the iron atom in the ferrocene nucleus, and the hydroxyl group of the oxime fragment: there are no structural features which could explain the very ready dehydration of the oxime to the nitrile.

Attempted formation of Potentially Electroactive Polymers

The Wittig reaction of a phosphorane (3) with an acyl ferrocene (4) has been reported [11] to yield the trans-1,2-diferrocenylethene: (5)



(3) (4) (5)

this work has been repeated in order that this may be extended to alternative diphosphoranes or diacyl ferrocenes with the aim of producing electroactive polymers. The use of n-butyllithium, in place of the suggested phenyllithium, appears to be detrimental to the success of the reaction, since no coupled materials could be separated from the insoluble polymeric residues.

6.4.1 Preparation of $FcCH_2PPh_3I$

$FcCH_2N(CH_3)_3I$ (20.5g;53mmol) and PPh_3 (26.2g;0.1 mol) were combined with ethanol (600cm^3) and refluxed for 24 hours. After cooling, yellow crystals of $FcCH_2PPh_3I$ (30.2g;96%) were filtered off.

6.4.2 Reaction of $FcCH_2PPh_3I$ with FcCHO

$FcCH_2PPh_3I$ (1g;1.7 mmol) was dissolved in 15 cm^3 diethyl ether and n-butyllithium (1.2cm^3;1.7mmol) was added. The mixture was stirred for 1 hour. A solution of FcCHO (0.36g;1.7 mmol) in 10 cm^3 diethyl ether was added dropwise with stirring. The whole was stirred for 24 hours and then evaporated to dryness. THF (50 cm^3) was added and the solution was refluxed for 2.5 hours, cooled, and filtered. Evaporation of the filtrate gave an insoluble red polymeric material.

6.4.3 Reaction of $FcCH_2PPh_3I$ with $FcC(CH_3)O$

$FcCH_2PPh_3I$ (1g;1.7 mmol) was dissolved in 15 cm^3 diethyl ether and n-butyllithium (1.2cm^3;1.7mmol) was added. The mixture was stirred for 1 hour. A solution of $FcC(CH_3)O$ (0.4g;1.7mmol) in 10 cm^3 diethyl ether was added dropwise with stirring. The whole was stirred for 24 hours and then evaporated to dryness. THF (50 cm^3) was added and the solution was refluxed for 2.5 hours, cooled, and filtered. Evaporation of the filtrate gave an insoluble red polymeric material.

Table 6.3 Final refined atom coordinates.

Positional and thermal parameters and their e.s.d.'s

Atom	x	y	z	B(Å²)
Fe1	0.07592(4)	0.17487(7)	0.55583(7)	4.81(2)
Fe2	0.17273(4)	0.68270(8)	0.61094(9)	6.51(3)
C1	0.1056(3)	0.1463(6)	0.4370(5)	5.9(2)
C2	0.0634(3)	0.0715(5)	0.4280(5)	6.3(2)
C3	0.0142(3)	0.1296(6)	0.4047(6)	7.3(2)
C4	0.0256(3)	0.2321(6)	0.3987(6)	7.9(2)
C5	0.0805(3)	0.2433(6)	0.4183(5)	6.9(2)
C6	0.1633(3)	0.1143(6)	0.4689(5)	6.8(2)
C11	0.1419(3)	0.1855(7)	0.7078(6)	7.2(2)
C12	0.1101(3)	0.0998(6)	0.7071(5)	7.6(2)
C13	0.0580(3)	0.1385(7)	0.6897(6)	8.7(2)
C14	0.0571(3)	0.2412(7)	0.6786(6)	8.3(2)
C15	0.1090(3)	0.2727(6)	0.6894(6)	7.8(2)
C21	0.1340(4)	0.5465(6)	0.5508(7)	9.4(3)
C22	0.1486(5)	0.5558(7)	0.670(1)	14.2(4)
C23	0.2113(5)	0.5659(8)	0.7203(9)	14.0(5)
C24	0.2226(4)	0.5571(7)	0.627(1)	15.7(4)
C25	0.1754(5)	0.5534(7)	0.5146(9)	15.6(3)
C26	0.0666(6)	0.5406(10)	0.4710(13)	7.4(4)
C31	0.1193(3)	0.8047(6)	0.5515(7)	7.8(2)
C32	0.1456(4)	0.8100(6)	0.6659(7)	8.3(2)
C33	0.2024(4)	0.8164(6)	0.6998(6)	7.6(2)
C34	0.2126(3)	0.8157(6)	0.6033(7)	7.7(2)
C35	0.1601(4)	0.8070(6)	0.5083(6)	8.5(3)
O1	0.2558(3)	0.1244(7)	0.5172(8)	8.6(2)

Positional and thermal parameters and their e.s.d.'s (cont.

Atom	x	y	z	B(Å^2)
O1*	0.1876(5)	0.2874(10)	0.4747(10)	8.2(4)
N1	0.2030(2)	0.1758(5)	0.4819(4)	6.9(2)
O2	0.0684(6)	0.5050(10)	0.3070(16)	10.5(5)
N2	0.0507(8)	0.5212(10)	0.3762(11)	6.6(5)
O2*	0.0	0.5	0.5	4.8(8)
N2*	0.0525(15)	0.5302(24)	0.5557(42)	B(1)

Occupancy factors for C26, O1, O1*, O2, N2, O2* and N2* were 0.47, 0.59, 0.41, 0.33, 0.33, 0.14, 0.14 respectively. This allowed for the disorder of the OH group in molecule A, and the partial occupancy and disorder of the CH=NOH group in molecule B

Anisotropically refined atoms are given in the form of the isotropic equivalent thermal parameter defined as:

(4/3) * [a2*B(1,1) + b2*B(2,2) + c2*B(3,3) + ab(cos gamma)*B(1,2) + ac(cos beta)*B(1,3) + bc(cos alpha)*B(2,3)]

Table 6.4 Selected bond lengths and angles.

Molecular dimensions

(a) Interatomic distances (Å)

Fe1	C1	2.020(8)
Fe1	C2	2.028(7)
Fe1	C3	2.040(6)
Fe1	C4	2.043(7)
Fe1	C5	2.020(8)
Fe1	C11	2.019(6)
Fe1	C12	2.018(6)
Fe1	C13	2.020(9)
Fe1	C14	2.028(9)
Fe1	C15	2.010(7)
Fe2	C21	2.009(8)
Fe2	C22	2.006(12)
Fe2	C23	2.018(10)
Fe2	C24	2.036(10)
Fe2	C25	2.086(11)
Fe2	C31	2.033(8)
Fe2	C32	2.022(9)
Fe2	C33	2.027(7)
Fe2	C34	2.028(8)
Fe2	C35	2.004(7)
C1	C2	1.441(10)
C1	C5	1.382(10)
C1	C6	1.468(10)
C2	C3	1.422(11)
C3	C4	1.354(12)

C4	C5	1.378(12)
C6	N1	1.269(10)
C11	C12	1.381(12)
C11	C15	1.376(11)
C12	C13	1.396(12)
C13	C14	1.322(12)
C14	C15	1.384(13)
C21	C22	1.422(15)
C21	C25	1.362(19)
C21	C26	1.655(16)
C22	C23	1.521(18)
C23	C24	1.351(20)
C24	C25	1.473(14)
C26	N2	1.141(21)
C26	N2*	1.30(6)
C31	C32	1.344(11)
C31	C35	1.405(15)
C32	C33	1.386(13)
C33	C34	1.375(13)
C34	C35	1.427(10)
O1	N1	1.438(10)
O1*	N1	1.478(14)
O2	N2	1.18(3)
O2*	N2*	1.33(3)
O1 ...	N1(I)	2.779(11)

O1*...	N1(I)	2.759(15)
O2 ...	N2*(II)	3.11(6)
O2 ...	N2(III)	3.070(21)

The roman numerals refer to the following equivalent

(I)	1/2 − x,	1/2 − y,	1 − z
(II)	x,	1 − y,	−1/2 + z
(III)	− x,	y,	1/2 − z

positions

(b) Bond angles (°)

C1	Fe1	C2	41.7(3)
C1	Fe1	C3	68.8(3)
C1	Fe1	C4	67.5(3)
C1	Fe1	C5	40.0(3)
C2	Fe1	C3	40.9(3)
C2	Fe1	C4	67.1(3)
C2	Fe1	C5	67.8(3)
C3	Fe1	C4	38.7(3)
C3	Fe1	C5	66.6(3)
C4	Fe1	C5	39.7(3)
C11	Fe1	C12	40.0(3)
C11	Fe1	C13	66.8(3)
C11	Fe1	C14	67.2(3)
C11	Fe1	C15	39.9(3)
C12	Fe1	C13	40.5(4)
C12	Fe1	C14	66.8(3)
C12	Fe1	C15	67.0(3)
C13	Fe1	C14	38.1(4)
C13	Fe1	C15	65.7(3)
C14	Fe1	C15	40.1(4)
C21	Fe2	C22	41.5(4)
C21	Fe2	C23	70.0(4)
C21	Fe2	C24	64.0(4)
C21	Fe2	C25	38.8(5)
C22	Fe2	C23	44.4(5)
C22	Fe2	C24	67.4(5)
C22	Fe2	C25	71.1(5)

C23	Fe2	C24	38.9(6)
C23	Fe2	C25	72.2(4)
C24	Fe2	C25	41.9(4)
C31	Fe2	C32	38.7(3)
C31	Fe2	C33	67.2(3)
C31	Fe2	C34	68.5(3)
C31	Fe2	C35	40.7(4)
C32	Fe2	C33	40.0(4)
C32	Fe2	C34	66.9(4)
C32	Fe2	C35	66.7(4)
C33	Fe2	C34	39.6(4)
C33	Fe2	C35	67.8(3)
C34	Fe2	C35	41.5(3)
Fe1	C1	C2	69.4(4)
Fe1	C1	C5	70.0(5)
Fe1	C1	C6	121.7(4)
C2	C1	C5	106.2(6)
C2	C1	C6	121.4(6)
C5	C1	C6	132.2(7)
Fe1	C2	C1	68.9(4)
Fe1	C2	C3	70.0(4)
C1	C2	C3	106.5(6)
Fe1	C3	C2	69.1(3)
Fe1	C3	C4	70.7(4)
C2	C3	C4	108.2(7)
Fe1	C4	C3	70.5(4)
Fe1	C4	C5	69.3(4)

C3	C4	C5	109.4(7)
Fe1	C5	C1	70.0(4)
Fe1	C5	C4	71.1(5)
C1	C5	C4	109.7(7)
C1	C6	N1	125.1(7)
Fe1	C11	C12	69.9(4)
Fe1	C11	C15	69.7(4)
C12	C11	C15	107.4(7)
Fe1	C12	C11	70.1(4)
Fe1	C12	C13	69.9(4)
C11	C12	C13	106.3(7)
Fe1	C13	C12	69.7(5)
Fe1	C13	C14	71.3(5)
C12	C13	C14	110.1(8)
Fe1	C14	C13	70.6(5)
Fe1	C14	C15	69.3(5)
C13	C14	C15	107.7(8)
Fe1	C15	C11	70.4(4)
Fe1	C15	C14	70.7(4)
C11	C15	C14	108.5(7)
Fe2	C21	C22	69.1(5)
Fe2	C21	C25	73.7(6)
Fe2	C21	C26	121.9(6)
C22	C21	C25	117.6(9)
C22	C21	C26	115.(1)
C25	C21	C26	128.(1)
Fe2	C22	C21	69.4(6)

Fe2	C22	C23	68.2(6)
C21	C22	C23	103.(1)
Fe2	C23	C22	67.4(6)
Fe2	C23	C24	71.3(6)
C22	C23	C24	102.6(9)
Fe2	C24	C23	69.8(7)
Fe2	C24	C25	70.9(6)
C23	C24	C25	118.(1)
Fe2	C25	C21	67.6(6)
Fe2	C25	C24	67.3(6)
C21	C25	C24	98.(1)
C21	C26	N2	120.(2)
C21	C26	N2*	96.(2)
N2	C26	N2*	140.(2)
Fe2	C31	C32	70.2(5)
Fe2	C31	C35	68.6(4)
C32	C31	C35	107.1(7)
Fe2	C32	C31	71.1(5)
Fe2	C32	C33	70.2(5)
C31	C32	C33	110.6(9)
Fe2	C33	C32	69.8(5)
Fe2	C33	C34	70.2(4)
C32	C33	C34	108.0(6)
Fe2	C34	C33	70.2(5)
Fe2	C34	C35	68.4(5)
C33	C34	C35	106.6(8)
Fe2	C35	C31	70.7(5)

Fe2	C35	C34	70.2(4)
C31	C35	C34	107.6(7)
C6	N1	O1	113.8(6)
C6	N1	O1*	113.3(8)
O1	N1	O1*	132.1(7)
C26	N2	O2	139.(2)
C26	N2*	O2*	100.(3)

6.5 References Chapter Six.

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Appendix 1

Publications.

The following list of publications contains the results of my research for the period 1987-90.

1) Metal Complexation of Thioacylferrocenes: Crystal structures of pentacarbonyl(thiobenzoylferrocene-S)chromium and benzoylferrocene.

J.C.Barnes, W.Bell, C.Glidewell and R.A.Howie. *Journal of Organometallic Chemistry* , (1990), 385, 369-378.

2) Crystal and Molecular Structure of the Low-Melting form of ferrocenecarboxaldoxime

"in press",*Journal of Organometallic Chemistry* .G.Ferguson, W.Bell and C.Glidewell.

3) Simultaneous Acylation and Alkylation in the Friedel Crafts Reaction of Ferrocene with Trimethylacetyl Chloride.

"submitted", *Journal of Chemical Research* ,W.Bell and C.Glidewell.

4) Crystal and Molecular Structure of 1,4-diphenyl-1,4-epithio-2,3-dithia[4](1,1')ferrocenophane.

"in press". *Journal of The Chemical Society. Dalton Transactions* , G.Ferguson,W.Bell and C.Glidewell.

Appendix 2

Crystallographic data for pentacarbonyl(thiobenzoylferrocene-S) chromium and benzoylferrocene.

Appendix 2.1 Compound (1)

Coordinates x 10^4 for hydrogen atoms :

	x/a	y/b	z/c
H11	2154	4018	178
H21	1092	6221	58
H31	3089	8280	976
H41	3415	6223	3942
H51	4251	4823	1170
H71	3327	8636	4109
H81	4996	7288	1773
H91	1113	4546	2752
H101	-513	6170	2234
H131	2070	10876	2133
H141	3560	13088	3101
H151	3929	14123	5047
H161	2882	12960	6082
H171	1536	10758	5361

Appendix 2.2 Compound (1)

Anisotropic temperature factors X 10^3
with e.s.d's in parentheses :

	U11	U22	U33	U23	U13	U12
FE1	40(1)	33(1)	35(1)	13(1)	7(1)	12(1)
CR2	36(1)	35(1)	36(1)	15(1)	3(1)	11(1)
C1	89(3)	51(2)	48(2)	1(1)	23(2)	1(2)
C2	72(2)	106(3)	46(2)	34(2)	15(2)	33(2)
C3	114(3)	60(2)	72(2)	35(2)	54(2)	27(2)
C4	56(2)	88(3)	61(2)	5(2)	21(2)	-9(2)
C5	90(3)	100(3)	68(2)	43(2)	42(2)	65(2)
C6	37(1)	33(1)	30(1)	13(1)	6(1)	8(1)
C7	43(1)	41(1)	33(1)	14(1)	0(1)	10(1)
C8	59(2)	49(2)	46(1)	27(1)	4(1)	20(1)
C9	64(2)	34(1)	57(2)	26(1)	16(1)	10(1)
C10	42(1)	35(1)	45(1)	16(1)	10(1)	5(1)
C11	34(1)	33(1)	26(1)	10(1)	7(1)	8(1)
C12	30(1)	34(1)	40(1)	14(1)	3(1)	8(1)
C13	37(1)	52(2)	68(2)	38(1)	10(1)	12(1)
C14	44(2)	55(2)	110(3)	50(2)	10(2)	6(1)
C15	52(2)	42(2)	125(4)	30(2)	-15(2)	-7(1)
C16	67(2)	48(2)	66(2)	4(2)	-19(2)	1(2)
C17	51(2)	41(1)	44(1)	9(1)	-3(1)	5(1)
S18	43(1)	33(1)	43(1)	12(1)	-7(1)	8(1)
C19	50(2)	40(1)	39(1)	15(1)	4(1)	12(1)
O20	72(2)	68(2)	63(1)	34(1)	21(1)	8(1)
C21	43(1)	56(2)	51(2)	23(1)	0(1)	14(1)
O22	69(2)	85(2)	53(1)	11(1)	-16(1)	4(1)
C23	40(1)	45(1)	46(1)	18(1)	0(1)	8(1)
O24	54(1)	83(2)	69(2)	35(1)	14(1)	1(1)
C25	44(1)	37(1)	46(1)	17(1)	11(1)	12(1)
O26	67(1)	48(1)	51(1)	3(1)	5(1)	4(1)
C27	53(2)	52(2)	52(2)	26(1)	9(1)	20(1)
O28	85(2)	73(2)	95(2)	45(2)	12(1)	45(1)

Appendix 2.3

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Pentacarbonyl(thiobenzoylferrocene–S)chromium (1),

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
1	0	0	559	552	-9	2	0	147	150	3	3	0	97	-104	-4	5	0	48	55	-11	7	0	62	63
2	0	0	1040	-1036	-8	2	0	90	-95	4	3	0	40	-52	-3	5	0	226	-222	-10	7	0	77	81
3	0	0	182	183	-7	2	0	338	-340	5	3	0	119	117	-2	5	0	220	219	-9	7	0	109	-108
4	0	0	225	245	-6	2	0	90	83	6	3	0	131	128	-1	5	0	238	227	-8	7	0	129	-133
5	0	0	195	199	-5	2	0	534	524	7	3	0	153	153	0	5	0	501	-492	-7	7	0	261	262
6	0	0	276	-280	-4	2	0	102	-91	8	3	0	29	34	1	5	0	408	-396	-6	7	0	245	248
7	0	0	304	-308	-3	2	0	395	-368	9	3	0	41	40	2	5	0	128	132	-5	7	0	161	-152
8	0	0	67	-66	-2	2	0	170	152	10	3	0	52	-51	3	5	0	388	400	-4	7	0	305	-300
9	0	0	118	120	-1	2	0	541	495	-10	4	0	47	-51	4	5	0	105	106	-3	7	0	205	-204
10	0	0	34	24	0	2	0	253	-237	-9	4	0	113	-109	5	5	0	285	-282	-2	7	0	257	253
-11	1	0	91	88	1	2	0	781	-720	-8	4	0	52	51	6	5	0	68	-79	-1	7	0	33	-34
-10	1	0	74	76	2	2	0	29	22	-7	4	0	341	348	7	5	0	194	195	0	7	0	360	-360
-9	1	0	32	-51	3	2	0	357	-350	-6	4	0	45	-46	8	5	0	62	67	1	7	0	125	-121
-8	1	0	197	-201	4	2	0	366	-363	-5	4	0	676	-668	9	5	0	73	-70	2	7	0	129	121
-7	1	0	64	-61	5	2	0	131	-127	-4	4	0	115	-108	10	5	0	79	-81	3	7	0	180	177
-6	1	0	48	-54	6	2	0	30	17	-3	4	0	469	472	11	5	0	31	-18	7	7	0	53	-41
-5	1	0	286	-277	7	2	0	192	192	-2	4	0	369	366	-10	6	0	185	-190	8	7	0	54	-53
-4	1	0	94	-86	8	2	0	62	-60	-1	4	0	632	-622	-9	6	0	105	-107	-10	8	0	92	98
-2	1	0	103	-96	9	2	0	142	-141	0	4	0	790	-780	-8	6	0	269	286	-9	8	0	86	90
-1	1	0	565	-552	11	2	0	83	83	1	4	0	295	-261	-7	6	0	295	294	-8	8	0	35	-37
0	1	0	435	413	12	2	0	35	35	2	4	0	170	191	-6	6	0	67	-74	-7	8	0	153	-150
1	1	0	1095	1090	-12	3	0	57	46	3	4	0	64	59	-5	6	0	107	-104	-6	8	0	46	19
2	1	0	337	322	-11	3	0	84	-89	4	4	0	334	-339	-4	6	0	109	101	-5	8	0	152	150
3	1	0	471	-442	-10	3	0	190	-188	5	4	0	69	-59	-3	6	0	242	234	-4	8	0	24	-24
4	1	0	404	-414	-9	3	0	45	39	6	4	0	308	315	-2	6	0	75	69	-3	8	0	306	-307
5	1	0	350	348	-8	3	0	100	102	7	4	0	293	297	-1	6	0	364	-352	-2	8	0	108	-119
6	1	0	257	264	-7	3	0	105	-108	9	4	0	31	-25	0	6	0	39	-28	-1	8	0	325	328
7	1	0	203	-199	-6	3	0	353	-350	10	4	0	42	31	1	6	0	127	134	0	8	0	233	229
8	1	0	266	-269	-5	3	0	101	-103	-12	5	0	36	-29	3	6	0	112	123	1	8	0	200	-205
9	1	0	55	54	-4	3	0	543	557	-11	5	0	104	-103	4	6	0	300	296	2	8	0	217	-215
10	1	0	168	170	-3	3	0	618	616	-10	5	0	88	-86	5	6	0	210	215	3	8	0	166	158
11	1	0	57	71	-2	3	0	205	-221	-9	5	0	83	89	6	6	0	74	-70	4	8	0	189	187
12	1	0	39	-12	-1	3	0	651	-651	-8	5	0	176	176	7	6	0	81	-85	5	8	0	74	-73
-12	2	0	55	-53	0	3	0	404	379	-7	5	0	187	193	8	6	0	37	27	6	8	0	147	-143
-11	2	0	37	-47	1	3	0	152	-133	-6	5	0	163	159	9	6	0	54	68	7	8	0	54	-56
-10	2	0	66	67	2	3	0	278	-247	-5	5	0	144	151	10	6	0	53	-63	-6	9	0	134	-145

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
-5	9	0	48	-54	-5	12	0	59	-56	0	-10	1	71	67	2	-8	1	61	61	0	-6	1	151	-157
-4	9	0	41	-32	-4	12	0	110	120	1	-10	1	235	233	3	-8	1	109	111	1	-6	1	312	-316
-3	9	0	123	-127	-3	12	0	174	170	2	-10	1	116	119	4	-8	1	44	-55	2	-6	1	120	127
-2	9	0	85	-84	-1	12	0	96	-98	3	-10	1	132	-135	5	-8	1	231	-235	3	-6	1	385	374
-1	9	0	29	35	1	12	0	81	90	4	-10	1	69	-68	6	-8	1	241	-238	4	-6	1	150	-153
0	9	0	253	260	2	12	0	33	29	5	-10	1	169	169	7	-8	1	42	19	5	-6	1	408	-400
1	9	0	171	181	3	12	0	44	-64	6	-10	1	82	72	8	-8	1	71	85	6	-6	1	117	-116
2	9	0	65	-70	0	-13	1	42	40	7	-10	1	102	-93	9	-8	1	89	-86	7	-6	1	232	246
3	9	0	75	-80	1	-13	1	30	17	8	-10	1	88	-92	10	-8	1	122	-129	8	-6	1	155	164
4	9	0	47	44	2	-13	1	65	-59	9	-10	1	33	-30	-10	-7	1	58	-50	9	-6	1	31	-55
5	9	0	95	89	3	-13	1	72	-74	-7	-9	1	46	-37	-9	-7	1	79	-76	10	-6	1	55	-44
6	9	0	29	-39	4	-13	1	46	52	-5	-9	1	32	-32	-7	-7	1	38	46	11	-6	1	53	51
7	9	0	100	-93	-3	-12	1	83	-76	-4	-9	1	85	-92	-6	-7	1	36	-60	12	-6	1	41	52
-8	10	0	124	-131	-2	-12	1	47	-35	-3	-9	1	56	-57	-5	-7	1	350	-354	-11	-5	1	46	33
-7	10	0	77	-69	-1	-12	1	49	-39	-2	-9	1	48	40	-4	-7	1	100	-103	-10	-5	1	72	-76
-6	10	0	54	57	0	-12	1	88	-79	-1	-9	1	38	-29	-3	-7	1	376	364	-6	-5	1	28	31
-4	10	0	50	-46	1	-12	1	60	-49	0	-9	1	88	-92	-2	-7	1	231	224	-5	-5	1	32	-6
-3	10	0	87	-95	3	-12	1	35	4	2	-9	1	233	232	-1	-7	1	144	-149	-4	-5	1	225	225
-2	10	0	35	47	4	-12	1	42	7	3	-9	1	92	94	0	-7	1	101	-107	-2	-5	1	116	112
-1	10	0	79	78	5	-12	1	80	75	4	-9	1	230	-235	1	-7	1	371	364	-1	-5	1	260	254
0	10	0	31	-37	6	-12	1	76	77	5	-9	1	202	-197	2	-7	1	516	508	0	-5	1	246	241
2	10	0	57	54	-5	-11	1	119	110	6	-9	1	64	61	3	-7	1	79	-71	1	-5	1	85	-88
3	10	0	61	-63	-4	-11	1	87	88	7	-9	1	113	119	4	-7	1	215	-216	2	-5	1	590	-601
4	10	0	95	-95	-3	-11	1	50	-58	8	-9	1	106	-106	5	-7	1	33	23	3	-5	1	33	41
-8	11	0	46	-30	-2	-11	1	120	-117	9	-9	1	175	-173	7	-7	1	25	20	4	-5	1	361	360
-7	11	0	129	-128	-1	-11	1	30	-33	-9	-8	1	59	69	8	-7	1	50	40	5	-5	1	30	-14
-6	11	0	79	-72	0	-11	1	70	75	-8	-8	1	79	80	9	-7	1	35	-16	6	-5	1	179	-174
-5	11	0	103	104	2	-11	1	125	-122	-7	-8	1	42	-50	11	-7	1	40	27	7	-5	1	46	-53
-4	11	0	170	165	3	-11	1	60	-67	-6	-8	1	119	-121	-9	-6	1	34	-28	8	-5	1	48	44
-2	11	0	46	-39	4	-11	1	106	109	-5	-8	1	36	-48	-8	-6	1	89	-96	9	-5	1	180	168
-1	11	0	93	104	5	-11	1	102	108	-4	-8	1	86	84	-6	-6	1	67	71	10	-5	1	35	-23
0	11	0	135	129	7	-11	1	33	-32	-3	-8	1	28	-25	-5	-6	1	49	-47	11	-5	1	142	-137
2	11	0	64	-68	-4	-10	1	63	67	-2	-8	1	103	-107	-4	-6	1	298	-293	-11	-4	1	66	57
3	11	0	56	-52	-3	-10	1	66	68	-1	-8	1	33	36	-3	-6	1	83	-88	-9	-4	1	125	-121
5	11	0	57	-40	-2	-10	1	121	-125	0	-8	1	251	254	-2	-6	1	466	470	-8	-4	1	98	-95
-6	12	0	76	-87	-1	-10	1	170	-171	1	-8	1	142	142	-1	-6	1	228	239	-7	-4	1	145	152

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
-6	-4	1	296	298	7	-3	1	32	34	-3	-1	1	154	142	9	0	1	227	-240	3	2	1	274	259
-5	-4	1	137	-135	8	-3	1	77	74	-2	-1	1	122	-132	10	0	1	65	-68	4	2	1	598	-615
-4	-4	1	230	-237	9	-3	1	58	58	-1	-1	1	442	-411	11	0	1	82	77	5	2	1	457	-450
-3	-4	1	68	70	12	-3	1	63	-54	0	-1	1	312	-258	12	0	1	64	65	6	2	1	28	41
-2	-4	1	186	178	-12	-2	1	42	-41	1	-1	1	510	510	-12	1	1	38	31	7	2	1	161	153
-1	-4	1	32	-35	-11	-2	1	34	-30	2	-1	1	638	616	-10	1	1	125	-127	9	2	1	94	-86
0	-4	1	139	-138	-10	-2	1	82	-84	3	-1	1	696	647	-9	1	1	112	113	10	2	1	78	83
1	-4	1	353	-338	-9	-2	1	36	33	4	-1	1	258	-274	-8	1	1	355	355	11	2	1	76	85
2	-4	1	370	-348	-8	-2	1	133	139	5	-1	1	355	356	-7	1	1	45	40	12	2	1	43	43
3	-4	1	179	-172	-6	-2	1	55	-49	6	-1	1	539	551	-6	1	1	383	-389	-12	3	1	57	39
4	-4	1	136	-125	-5	-2	1	287	291	7	-1	1	140	-143	-5	1	1	494	-491	-11	3	1	81	-82
5	-4	1	450	427	-4	-2	1	557	551	8	-1	1	309	-311	-4	1	1	456	458	-9	3	1	43	39
6	-4	1	292	284	-3	-2	1	214	-225	9	-1	1	72	-71	-3	1	1	660	643	-8	3	1	41	38
7	-4	1	40	-42	-2	-2	1	544	-524	10	-1	1	140	138	-2	1	1	219	214	-7	3	1	29	-34
9	-4	1	119	115	-1	-2	1	506	-498	12	-1	1	34	-34	-1	1	1	912	-909	-6	3	1	287	-291
10	-4	1	115	111	0	-2	1	1192	1225	-12	0	1	98	-96	1	1	1	1422	1433	-5	3	1	201	-202
11	-4	1	77	-68	1	-2	1	575	564	-11	0	1	123	-119	2	1	1	162	145	-4	3	1	136	129
12	-4	1	136	-132	2	-2	1	1642	-1617	-10	0	1	111	119	3	1	1	197	-195	-3	3	1	212	-214
-12	-3	1	50	-50	3	-2	1	320	-322	-9	0	1	290	295	4	1	1	211	-211	-2	3	1	52	-45
-11	-3	1	30	-17	4	-2	1	672	651	-8	0	1	134	137	5	1	1	125	-119	-1	3	1	451	453
-10	-3	1	27	40	5	-2	1	548	549	-7	0	1	320	-323	6	1	1	124	-126	0	3	1	227	-222
-8	-3	1	110	-109	6	-2	1	67	-62	-6	0	1	140	-147	8	1	1	42	-33	1	3	1	226	-230
-7	-3	1	83	-77	7	-2	1	247	-249	-5	0	1	443	444	9	1	1	95	-87	2	3	1	90	74
-6	-3	1	353	353	8	-2	1	112	-110	-4	0	1	56	56	11	1	1	35	-7	3	3	1	240	236
-5	-3	1	602	588	9	-2	1	100	103	-3	0	1	400	-412	-11	2	1	42	56	4	3	1	165	-149
-4	-3	1	226	-221	10	-2	1	58	65	-2	0	1	70	-71	-10	2	1	51	48	5	3	1	527	-527
-3	-3	1	909	-903	11	-2	1	87	-96	-1	0	1	159	145	-9	2	1	40	-57	6	3	1	266	-278
-2	-3	1	73	69	12	-2	1	33	-39	0	0	1	202	193	-8	2	1	42	23	7	3	1	85	76
-1	-3	1	105	-102	-12	-1	1	102	-109	1	0	1	444	-408	-6	2	1	62	-58	8	3	1	122	122
0	-3	1	311	-294	-11	-1	1	31	-9	2	0	1	30	-18	-5	2	1	754	-760	10	3	1	34	-14
1	-3	1	699	-664	-10	-1	1	107	103	3	0	1	539	513	-4	2	1	432	-434	-12	4	1	109	101
2	-3	1	1387	-1336	-8	-1	1	99	-95	4	0	1	90	79	-3	2	1	759	761	-11	4	1	76	63
3	-3	1	88	-90	-7	-1	1	43	46	5	0	1	84	-90	-2	2	1	248	241	-10	4	1	140	-145
4	-3	1	472	462	-6	-1	1	330	323	6	0	1	20	23	-1	2	1	86	-72	-9	4	1	218	-221
5	-3	1	31	25	-5	-1	1	158	-148	7	0	1	116	113	0	2	1	611	-558	-7	4	1	154	153
6	-3	1	56	-48	-4	-1	1	233	-221	8	0	1	84	-84	2	2	1	708	656	-6	4	1	36	-33

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR CR3B

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
-5	4	1	419	-420	-7	6	1	88	-91	8	7	1	39	-45	-7	10	1	64	68	-4	-11	2	42	50
-4	4	1	256	237	-6	6	1	26	-34	9	7	1	58	45	-6	10	1	62	-58	0	-11	2	99	-98
-3	4	1	677	660	-5	6	1	336	334	-10	8	1	137	138	-5	10	1	149	-147	1	-11	2	87	-101
-2	4	1	291	291	-4	6	1	310	313	-9	8	1	74	79	-4	10	1	43	54	3	-11	2	31	23
-1	4	1	81	-70	-3	6	1	341	-331	-8	8	1	104	-109	-3	10	1	127	125	4	-11	2	71	-81
0	4	1	229	-222	-2	6	1	318	-297	-7	8	1	74	-71	-2	10	1	96	-93	5	-11	2	56	-68
1	4	1	275	248	-1	6	1	470	472	-6	8	1	280	282	-1	10	1	147	-142	6	-11	2	77	75
3	4	1	173	-181	0	6	1	286	273	-5	8	1	204	198	1	10	1	151	161	7	-11	2	161	165
4	4	1	123	122	1	6	1	313	-314	-4	8	1	157	-164	2	10	1	123	119	8	-11	2	44	47
5	4	1	53	64	2	6	1	443	-436	-3	8	1	241	-240	3	10	1	68	-59	-7	-10	2	35	39
6	4	1	151	-150	3	6	1	49	45	-2	8	1	215	-210	4	10	1	125	-113	-4	-10	2	91	96
7	4	1	90	-88	4	6	1	311	311	-1	8	1	111	-120	6	10	1	64	73	-3	-10	2	67	71
8	4	1	34	49	5	6	1	25	-11	0	8	1	125	-128	-1	11	1	68	66	-2	-10	2	113	-122
9	4	1	122	113	6	6	1	139	-138	1	8	1	60	-45	2	11	1	109	95	-1	-10	2	169	-178
10	4	1	36	36	8	6	1	98	96	2	8	1	93	91	3	11	1	57	59	1	-10	2	27	6
11	4	1	87	-79	9	6	1	58	48	3	8	1	104	103	4	11	1	97	-101	2	-10	2	82	-86
-11	5	1	46	49	10	6	1	33	-32	5	8	1	72	71	-5	12	1	60	-59	5	-10	2	39	35
-10	5	1	75	70	-11	7	1	68	66	6	8	1	97	99	-4	12	1	37	48	8	-10	2	55	60
-9	5	1	149	-142	-10	7	1	66	66	7	8	1	35	-8	-3	12	1	43	49	9	-10	2	30	-31
-8	5	1	282	-293	-9	7	1	84	-91	8	8	1	38	-26	0	12	1	65	74	-8	-9	2	33	15
-6	5	1	371	373	-8	7	1	66	-70	-9	9	1	122	117	1	12	1	60	70	-7	-9	2	109	105
-5	5	1	107	102	-7	7	1	186	190	-8	9	1	98	94	-2	-13	2	49	-45	-6	-9	2	103	105
-4	5	1	322	-321	-6	7	1	93	99	-7	9	1	69	-71	0	-13	2	68	76	-5	-9	2	76	-80
-2	5	1	632	643	-5	7	1	84	-82	-6	9	1	125	-122	1	-13	2	38	23	-4	-9	2	94	-101
-1	5	1	526	529	-4	7	1	50	57	-5	9	1	142	146	2	-13	2	117	-112	-3	-9	2	182	183
0	5	1	334	-337	-3	7	1	46	-47	-4	9	1	166	167	3	-13	2	108	-103	-2	-9	2	129	129
1	5	1	251	-259	-2	7	1	231	-236	-3	9	1	112	-113	4	-13	2	47	28	-1	-9	2	282	-284
2	5	1	73	-65	-1	7	1	118	-113	-2	9	1	265	-266	-3	-12	2	67	68	0	-9	2	366	-363
3	5	1	119	123	0	7	1	64	66	-1	9	1	114	-114	-1	-12	2	60	-61	1	-9	2	39	26
5	5	1	65	-69	1	7	1	98	94	0	9	1	40	47	1	-12	2	81	82	2	-9	2	199	196
6	5	1	74	72	2	7	1	103	-96	1	9	1	42	35	3	-12	2	143	-143	3	-9	2	114	104
7	5	1	81	73	3	7	1	100	-89	2	9	1	47	-39	4	-12	2	146	-142	4	-9	2	53	-36
8	5	1	78	78	4	7	1	59	71	3	9	1	38	-36	5	-12	2	63	71	6	-9	2	144	147
10	5	1	32	22	5	7	1	194	191	4	9	1	69	67	6	-12	2	174	167	7	-9	2	54	55
-11	6	1	33	-18	6	7	1	92	86	5	9	1	78	80	7	-12	2	45	51	8	-9	2	73	-72
-8	6	1	43	-45	7	7	1	81	-82	-8	10	1	65	58	-5	-11	2	34	55	9	-9	2	49	-70

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
10	-9	2	34	-52	7	-7	2	256	-261	2	-5	2	404	-410	-4	-3	2	141	-128	10	-2	2	69	-75
-9	-8	2	30	18	9	-7	2	139	140	3	-5	2	175	-174	-3	-3	2	95	95	11	-2	2	51	32
-8	-8	2	28	15	11	-7	2	88	-83	4	-5	2	491	483	-2	-3	2	461	450	-11	-1	2	48	-45
-6	-8	2	53	53	-10	-6	2	67	61	5	-5	2	43	-31	-1	-3	2	112	113	-10	-1	2	106	-99
-4	-8	2	178	-172	-8	-6	2	55	-53	6	-5	2	462	-459	0	-3	2	308	-286	-9	-1	2	97	-97
-3	-8	2	174	-179	-7	-6	2	54	69	7	-5	2	181	-186	1	-3	2	362	349	-8	-1	2	239	244
-2	-8	2	154	155	-6	-6	2	133	139	8	-5	2	78	82	2	-3	2	140	-137	-7	-1	2	223	216
-1	-8	2	266	264	-5	-6	2	139	-151	9	-5	2	94	97	3	-3	2	311	-313	-6	-1	2	215	-218
0	-8	2	191	-189	-4	-6	2	394	-402	10	-5	2	58	62	4	-3	2	461	-451	-5	-1	2	210	-218
1	-8	2	315	-312	-3	-6	2	41	-42	11	-5	2	34	32	5	-3	2	452	-434	-4	-1	2	248	268
2	-8	2	32	48	-2	-6	2	293	301	-11	-4	2	32	36	6	-3	2	438	442	-3	-1	2	775	781
3	-8	2	280	275	-1	-6	2	330	323	-9	-4	2	55	-53	7	-3	2	153	148	-2	-1	2	125	-110
4	-8	2	175	174	0	-6	2	87	73	-7	-4	2	205	-203	8	-3	2	174	-171	-1	-1	2	1311	-1390
5	-8	2	91	-83	1	-6	2	104	93	-6	-4	2	258	-255	10	-3	2	128	128	0	-1	2	554	-584
6	-8	2	180	-184	2	-6	2	59	60	-5	-4	2	75	77	11	-3	2	89	90	1	-1	2	1261	1253
7	-8	2	106	109	3	-6	2	228	221	-4	-4	2	309	313	12	-3	2	55	-46	2	-1	2	318	303
8	-8	2	128	128	4	-6	2	174	165	-3	-4	2	249	238	-12	-2	2	44	-53	3	-1	2	221	-216
9	-8	2	103	-107	5	-6	2	144	-146	-2	-4	2	118	-128	-11	-2	2	154	-149	4	-1	2	125	-134
10	-8	2	99	-95	6	-6	2	61	-64	-1	-4	2	124	-116	-9	-2	2	179	179	5	-1	2	180	183
-10	-7	2	65	-50	7	-6	2	165	-165	0	-4	2	110	114	-8	-2	2	30	25	6	-1	2	366	373
-9	-7	2	37	-36	8	-6	2	155	-155	1	-4	2	421	420	-7	-2	2	339	-345	7	-1	2	161	164
-8	-7	2	101	103	9	-6	2	73	86	2	-4	2	727	-709	-6	-2	2	326	-325	8	-1	2	52	68
-7	-7	2	40	33	10	-6	2	139	144	3	-4	2	765	-773	-5	-2	2	353	353	9	-1	2	106	-109
-6	-7	2	56	-49	12	-6	2	41	-41	4	-4	2	61	66	-4	-2	2	525	539	10	-1	2	51	-53
-5	-7	2	143	-142	-10	-5	2	57	63	5	-4	2	219	221	-3	-2	2	26	8	11	-1	2	41	-23
-4	-7	2	122	-118	-9	-5	2	44	45	6	-4	2	92	-92	-2	-2	2	685	-648	12	-1	2	62	-56
-3	-7	2	59	-44	-8	-5	2	86	-92	7	-4	2	289	-283	-1	-2	2	53	41	-11	0	2	46	-37
-2	-7	2	39	-35	-7	-5	2	135	-130	8	-4	2	60	-63	0	-2	2	430	399	-10	0	2	38	43
-1	-7	2	133	125	-6	-5	2	59	50	9	-4	2	120	125	1	-2	2	63	-69	-7	0	2	142	145
0	-7	2	183	189	-5	-5	2	215	212	10	-4	2	163	160	2	-2	2	216	217	-6	0	2	320	310
1	-7	2	167	164	-4	-5	2	25	21	12	-4	2	62	-66	3	-2	2	50	46	-5	0	2	213	205
2	-7	2	72	-82	-3	-5	2	419	-436	-12	-3	2	51	-49	4	-2	2	72	79	-4	0	2	250	-241
3	-7	2	118	118	-2	-5	2	89	-92	-10	-3	2	137	139	6	-2	2	76	82	-3	0	2	543	561
4	-7	2	271	260	-1	-5	2	536	555	-8	-3	2	205	-205	7	-2	2	262	257	-2	0	2	110	-108
5	-7	2	43	40	0	-5	2	241	251	-7	-3	2	219	-219	8	-2	2	103	111	-1	0	2	649	-684
6	-7	2	199	-199	1	-5	2	86	75	-5	-3	2	145	144	9	-2	2	113	-119	0	0	2	985	-964

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
1	0	2	259	-246	-9	2	2	165	-160	5	3	2	263	-271	2	5	2	46	-45	-2	7	2	231	-236
2	0	2	773	754	-8	2	2	128	125	6	3	2	213	-212	3	5	2	255	-252	-1	7	2	163	-156
3	0	2	356	360	-7	2	2	385	385	7	3	2	48	23	4	5	2	155	-160	0	7	2	265	264
4	0	2	64	60	-6	2	2	146	149	8	3	2	44	35	5	5	2	25	50	1	7	2	65	58
5	0	2	253	-265	-5	2	2	457	-467	11	3	2	35	27	6	5	2	63	70	2	7	2	176	-175
6	0	2	222	217	-4	2	2	530	-517	-12	4	2	60	71	7	5	2	148	-135	3	7	2	196	-189
7	0	2	355	345	-3	2	2	111	108	-10	4	2	87	-95	8	5	2	115	-116	5	7	2	113	120
9	0	2	208	-206	-2	2	2	347	-333	-8	4	2	72	-70	9	5	2	70	68	6	7	2	80	83
10	0	2	91	-92	-1	2	2	412	-404	-7	4	2	110	-108	10	5	2	96	102	-10	8	2	45	-31
-12	1	2	90	78	0	2	2	455	-444	-5	4	2	61	69	-10	6	2	131	135	-9	8	2	39	-35
-11	1	2	75	-76	1	2	2	245	-231	-4	4	2	162	152	-9	6	2	100	100	-8	8	2	32	21
-10	1	2	75	-70	2	2	2	150	163	-3	4	2	106	-112	-8	6	2	231	-239	-7	8	2	60	54
-9	1	2	142	140	3	2	2	38	-22	-2	4	2	436	-427	-7	6	2	239	-243	-6	8	2	38	-22
-8	1	2	184	189	4	2	2	85	84	-1	4	2	636	621	-6	6	2	101	95	-5	8	2	98	-95
-7	1	2	119	116	5	2	2	35	31	0	4	2	940	916	-5	6	2	263	261	-3	8	2	169	169
-6	1	2	50	-57	6	2	2	97	-96	1	4	2	69	-69	-4	6	2	64	57	-1	8	2	282	-285
-5	1	2	130	-128	7	2	2	116	-115	2	4	2	230	-228	-3	6	2	138	-140	0	8	2	112	-108
-4	1	2	47	-31	8	2	2	24	-7	3	4	2	50	-55	-2	6	2	36	38	1	8	2	72	77
-3	1	2	69	72	9	2	2	54	50	4	4	2	327	320	-1	6	2	376	372	3	8	2	123	-132
-2	1	2	422	-424	11	2	2	74	-71	6	4	2	340	-339	0	6	2	206	208	4	8	2	157	-151
-1	1	2	142	-149	-12	3	2	32	-5	7	4	2	145	-142	1	6	2	52	-50	6	8	2	159	154
0	1	2	173	-183	-10	3	2	147	141	8	4	2	90	83	2	6	2	68	-70	7	8	2	84	81
1	1	2	726	-706	-9	3	2	26	26	9	4	2	109	112	3	6	2	78	-98	-9	9	2	68	69
2	1	2	368	376	-8	3	2	190	-198	10	4	2	34	7	4	6	2	106	-113	-8	9	2	66	58
3	1	2	311	328	-7	3	2	111	104	-11	5	2	115	119	5	6	2	112	-116	-7	9	2	26	-11
4	1	2	126	131	-6	3	2	355	352	-10	5	2	108	103	6	6	2	63	69	-5	9	2	100	112
5	1	2	35	14	-5	3	2	70	73	-9	5	2	173	-177	7	6	2	42	21	-4	9	2	48	45
6	1	2	109	-109	-4	3	2	543	-567	-8	5	2	258	-261	8	6	2	44	-52	-1	9	2	36	-41
7	1	2	69	65	-3	3	2	396	-403	-6	5	2	41	43	-11	7	2	59	-68	0	9	2	63	-77
8	1	2	133	129	-2	3	2	236	231	-5	5	2	48	-56	-10	7	2	64	-66	1	9	2	101	-102
9	1	2	40	-42	-1	3	2	736	717	-4	5	2	109	-112	-9	7	2	39	44	3	9	2	106	107
10	1	2	145	-143	0	3	2	96	-93	-3	5	2	123	116	-7	7	2	143	-145	5	9	2	48	-50
11	1	2	80	-83	1	3	2	414	-411	-2	5	2	262	256	-6	7	2	226	-222	6	9	2	34	42
-12	2	2	70	72	2	3	2	47	41	-1	5	2	76	79	-5	7	2	84	83	7	9	2	38	37
-11	2	2	110	114	3	3	2	140	159	0	5	2	212	215	-4	7	2	300	298	-8	10	2	142	143
-10	2	2	29	-25	4	3	2	228	218	1	5	2	240	237	-3	7	2	148	146	-7	10	2	113	112

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
-6	10	2	65	-71	-4	-11	3	85	-81	9	-9	3	136	140	11	-7	3	90	-95	6	-5	3	43	34
-5	10	2	107	-112	-3	-11	3	82	93	-9	-8	3	29	-29	-10	-6	3	41	49	7	-5	3	155	152
-4	10	2	59	50	-2	-11	3	180	179	-8	-8	3	77	-72	-9	-6	3	33	9	8	-5	3	129	-126
-3	10	2	88	91	0	-11	3	162	-164	-6	-8	3	134	141	-7	-6	3	27	23	9	-5	3	193	-195
-2	10	2	66	-76	1	-11	3	85	-86	-5	-8	3	98	101	-6	-6	3	40	-28	10	-5	3	52	53
-1	10	2	129	-127	2	-11	3	135	134	-4	-8	3	191	-184	-5	-6	3	92	96	11	-5	3	172	177
1	10	2	33	25	3	-11	3	74	72	-3	-8	3	142	-136	-4	-6	3	154	161	-11	-4	3	70	-63
3	10	2	45	38	4	-11	3	155	-157	-2	-8	3	67	57	-3	-6	3	34	-35	-10	-4	3	40	-8
4	10	2	76	64	5	-11	3	163	-163	0	-8	3	263	-272	-2	-6	3	317	-333	-9	-4	3	161	159
5	10	2	85	77	6	-11	3	38	40	1	-8	3	346	-335	-1	-6	3	176	-187	-8	-4	3	74	74
-6	11	2	72	82	7	-11	3	104	99	2	-8	3	86	90	1	-6	3	297	301	-7	-4	3	123	-127
-5	11	2	72	-76	-5	-10	3	48	37	3	-8	3	159	154	2	-6	3	173	168	-6	-4	3	270	-263
-4	11	2	156	-151	-2	-10	3	106	113	4	-8	3	179	183	3	-6	3	407	-415	-5	-4	3	140	142
-2	11	2	96	95	-1	-10	3	153	156	5	-8	3	130	135	4	-6	3	179	184	-4	-4	3	414	412
-1	11	2	63	-61	0	-10	3	25	-27	6	-8	3	31	-21	5	-6	3	394	399	-3	-4	3	150	-154
0	11	2	149	-149	1	-10	3	285	-289	7	-8	3	47	40	6	-6	3	147	141	-2	-4	3	424	-442
1	11	2	44	-42	2	-10	3	177	-176	8	-8	3	29	22	7	-6	3	33	-34	-1	-4	3	144	-145
2	11	2	81	80	3	-10	3	128	125	9	-8	3	25	-5	8	-6	3	184	-186	0	-4	3	609	609
3	11	2	87	85	4	-10	3	76	77	-10	-7	3	44	41	10	-6	3	118	115	1	-4	3	667	656
-4	12	2	53	-56	5	-10	3	154	-146	-9	-7	3	89	68	11	-6	3	35	-28	2	-4	3	133	118
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3	3	4	323	-312	-1	5	4	266	-256	6	7	4	64	-54	-1	11	4	42	38	-2	-10	5	68	-79
4	3	4	77	-79	0	5	4	146	-149	7	7	4	47	44	0	11	4	87	95	-1	-10	5	65	70
5	3	4	223	222	1	5	4	93	100	8	7	4	59	42	1	11	4	34	3	0	-10	5	59	66
6	3	4	179	173	4	5	4	99	99	-7	8	4	60	61	1	-14	5	40	-46	1	-10	5	58	62
7	3	4	69	-70	6	5	4	36	-20	-4	8	4	48	62	2	-14	5	52	-60	2	-10	5	170	165
8	3	4	109	-109	7	5	4	90	94	-3	8	4	75	-77	-3	-13	5	37	32	4	-10	5	90	-93
10	3	4	51	36	9	5	4	54	-57	-2	8	4	123	-127	-2	-13	5	40	49	5	-10	5	86	87
-12	4	4	71	-69	-11	6	4	45	32	-1	8	4	62	60	0	-13	5	97	-89	6	-10	5	86	91
-11	4	4	72	-59	-10	6	4	44	-44	0	8	4	112	111	1	-13	5	77	-74	7	-10	5	106	-113
-10	4	4	69	62	-8	6	4	165	161	1	8	4	40	-32	5	-13	5	97	103	8	-10	5	186	-186
-9	4	4	29	30	-7	6	4	155	151	2	8	4	94	-89	6	-13	5	114	106	-9	-9	5	38	30
-8	4	4	38	-46	-6	6	4	54	-54	4	8	4	78	84	-5	-12	5	75	69	-8	-9	5	38	-10
-6	4	4	40	-27	-5	6	4	278	-270	6	8	4	121	-116	-4	-12	5	49	-54	-7	-9	5	126	-125
-4	4	4	122	125	-4	6	4	96	-100	7	3	4	62	-50	-3	-12	5	154	-156	-6	-9	5	87	-83
-3	4	4	67	69	-3	6	4	179	179	-9	9	4	114	-109	-2	-12	5	35	-35	-5	-9	5	56	54
-2	4	4	27	-14	-2	6	4	53	44	-6	9	4	125	121	-1	-12	5	120	124	-4	-9	5	69	76
-1	4	4	116	-111	-1	6	4	239	-296	-5	9	4	61	-43	0	-12	5	40	38	-3	-9	5	101	-107
0	4	4	479	-473	0	6	4	218	-214	-4	9	4	111	-121	1	-12	5	122	-122	-2	-9	5	151	-152
2	4	4	385	383	1	6	4	242	254	-3	9	4	100	102	2	-12	5	72	-70	-1	-9	5	124	120
3	4	4	251	257	2	6	4	214	217	-2	9	4	164	170	3	-12	5	80	86	0	-9	5	138	135
4	4	4	41	41	3	6	4	74	72	-1	9	4	71	81	4	-12	5	93	102	1	-9	5	75	-78
5	4	4	44	-36	4	6	4	51	-32	0	9	4	50	-56	6	-12	5	36	-46	2	-9	5	68	-58
6	4	4	238	236	5	6	4	67	-66	4	9	4	48	-53	-3	-11	5	77	-68	3	-9	5	101	98
7	4	4	192	191	-10	7	4	74	74	-7	10	4	80	-89	-2	-11	5	149	-144	4	-9	5	28	-3
8	4	4	60	-56	-8	7	4	34	22	-6	10	4	53	68	0	-11	5	185	177	7	-9	5	36	-34
9	4	4	129	-130	-7	7	4	87	86	-5	10	4	100	106	1	-11	5	110	109	9	-9	5	46	-59
10	4	4	43	-42	-6	7	4	47	42	-4	10	4	47	-39	2	-11	5	198	-190	10	-9	5	29	-16
-11	5	4	104	-104	-5	7	4	70	-76	-3	10	4	95	-93	3	-11	5	84	-89	-8	-8	5	81	77
-10	5	4	49	-31	-4	7	4	255	-271	-2	10	4	47	46	4	-11	5	174	172	-7	-8	5	60	-58

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR CR39

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
-6	-8	5	148	-147	-6	-6	5	158	-157	-9	-4	5	79	-78	11	-3	5	115	-119	2	-1	5	392	361
-5	-3	5	95	-105	-4	-6	5	212	212	-8	-4	5	146	-146	12	-3	5	36	-34	3	-1	5	280	247
-4	-8	5	50	57	-3	-6	5	113	112	-6	-4	5	172	174	-11	-2	5	92	89	4	-1	5	40	36
-3	-8	5	290	288	-2	-6	5	45	-44	-5	-4	5	31	44	-10	-2	5	94	104	8	-1	5	77	-76
-2	-8	5	94	95	-1	-6	5	233	230	-4	-4	5	325	-337	-9	-2	5	51	-52	9	-1	5	92	-93
-1	-8	5	142	-146	0	-6	5	132	-134	-2	-4	5	528	545	-8	-2	5	57	54	10	-1	5	31	31
0	-8	5	75	68	1	-6	5	329	-324	-1	-4	5	204	218	-7	-2	5	148	153	11	-1	5	70	66
1	-8	5	407	404	2	-6	5	49	-53	0	-4	5	703	-717	-6	-2	5	111	106	-10	0	5	65	61
3	-8	5	403	-405	3	-6	5	198	200	1	-4	5	397	-398	-5	-2	5	179	173	-9	0	5	121	125
4	-8	5	181	-182	4	-6	5	56	-59	2	-4	5	169	172	-4	-2	5	239	-249	-8	0	5	53	44
6	-8	5	44	54	5	-6	5	364	-356	3	-4	5	273	271	-3	-2	5	353	-364	-7	0	5	209	-218
7	-8	5	58	-57	6	-6	5	149	-147	4	-4	5	276	265	-2	-2	5	208	-212	-6	0	5	165	-172
8	-8	5	33	-33	7	-6	5	189	185	5	-4	5	118	-126	0	-2	5	127	-121	-5	0	5	249	249
9	-8	5	81	83	8	-6	5	244	245	6	-4	5	48	59	1	-2	5	305	313	-4	0	5	315	326
10	-8	5	41	51	10	-6	5	121	-128	7	-4	5	186	187	3	-2	5	51	-55	-3	0	5	168	-168
-10	-7	5	30	-5	11	-6	5	46	25	9	-4	5	44	-42	4	-2	5	436	416	-2	0	5	652	-670
-5	-7	5	128	-124	-10	-5	5	115	-119	10	-4	5	70	-63	5	-2	5	326	322	-1	0	5	217	-198
-4	-7	5	45	-36	-9	-5	5	166	-174	11	-4	5	61	-66	6	-2	5	138	142	0	0	5	496	512
-3	-7	5	343	349	-8	-5	5	113	125	-9	-3	5	31	11	7	-2	5	290	-272	1	0	5	290	299
-2	-7	5	388	394	-7	-5	5	170	167	-7	-3	5	37	-50	8	-2	5	171	-175	2	0	5	430	-447
-1	-7	5	40	35	-6	-5	5	102	-97	-5	-3	5	173	176	9	-2	5	94	91	3	0	5	364	-372
0	-7	5	278	-289	-5	-5	5	78	-73	-4	-3	5	212	-220	10	-2	5	31	44	4	0	5	253	253
1	-7	5	121	128	-4	-5	5	98	97	-3	-3	5	473	-494	11	-2	5	97	-90	5	0	5	151	154
2	-7	5	457	463	-3	-5	5	420	416	-2	-3	5	338	-330	-12	-1	5	73	-69	6	0	5	238	-237
4	-7	5	487	-483	-2	-5	5	171	171	-1	-3	5	167	180	-11	-1	5	32	40	7	0	5	163	-169
5	-7	5	324	-322	-1	-5	5	515	-503	0	-3	5	403	424	-10	-1	5	169	165	8	0	5	40	34
6	-7	5	148	149	0	-5	5	550	-553	1	-3	5	128	-126	-9	-1	5	75	76	9	0	5	30	-14
7	-7	5	200	200	1	-5	5	251	235	2	-3	5	516	-510	-8	-1	5	82	-78	11	0	5	63	52
8	-7	5	45	-45	2	-5	5	83	-80	3	-3	5	404	403	-6	-1	5	277	285	-10	1	5	77	74
9	-7	5	86	-89	3	-5	5	254	-257	4	-3	5	772	798	-5	-1	5	500	498	-9	1	5	38	-33
10	-7	5	89	84	4	-5	5	42	41	5	-3	5	196	194	-4	-1	5	399	-403	-6	1	5	178	-181
11	-7	5	105	119	7	-5	5	137	-126	6	-3	5	263	-260	-3	-1	5	566	-585	-5	1	5	253	-249
-11	-6	5	51	-58	8	-5	5	47	46	7	-3	5	109	-112	-2	-1	5	142	-151	-4	1	5	92	-93
-10	-6	5	60	-66	9	-5	5	147	153	8	-3	5	230	235	-1	-1	5	259	264	-3	1	5	580	566
-8	-6	5	134	136	10	-5	5	51	-52	9	-3	5	82	81	0	-1	5	141	154	-2	1	5	23	-28
-7	-6	5	40	-45	11	-5	5	133	-134	10	-3	5	125	-123	1	-1	5	366	-347	-1	1	5	533	-538

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
0	1	5	125	-112	-3	3	5	335	348	-2	5	5	114	118	0	7	5	169	-174	0-13	6	78	73
1	1	5	665	661	-2	3	5	33	-31	-1	5	5	158	167	1	7	5	129	-129	2-13	6	117	-114
2	1	5	415	404	-1	3	5	38	-93	0	5	5	48	-47	2	7	5	37	43	3-13	6	100	-99
3	1	5	737	-727	0	3	5	191	178	1	5	5	394	-397	3	7	5	63	63	4-13	6	59	59
4	1	5	489	-439	1	3	5	268	262	2	5	5	134	-129	5	7	5	53	51	5-13	6	101	97
6	1	5	147	146	2	3	5	26	26	3	5	5	153	159	6	7	5	41	55	6-13	6	49	-32
8	1	5	168	-164	3	3	5	314	-315	4	5	5	131	134	-9	8	5	79	83	-2-12	6	50	43
9	1	5	34	-45	4	3	5	114	-113	5	5	5	77	-78	-8	8	5	34	-38	1-12	6	46	-37
10	1	5	115	105	7	3	5	94	38	6	5	5	66	-62	-7	8	5	58	-47	4-12	6	44	-24
11	1	5	62	66	8	3	5	114	117	7	5	5	95	94	-6	8	5	75	72	6-12	6	142	136
-12	2	5	31	-23	9	3	5	32	19	8	5	5	70	71	-5	8	5	104	104	7-12	6	34	29
-11	2	5	81	-36	10	3	5	37	-23	-10	6	5	90	-84	-4	8	5	72	-80	-6-11	6	54	-48
-9	2	5	133	134	-10	4	5	104	-133	-9	6	5	61	-61	-3	8	5	210	-204	-5-11	6	85	84
-8	2	5	95	-96	-9	4	5	140	-147	-8	6	5	59	53	-2	8	5	112	-106	-4-11	6	114	105
-7	2	5	230	-231	-8	4	5	71	-75	-7	6	5	144	147	-1	8	5	121	131	-3-11	6	68	-69
-6	2	5	127	-128	-7	4	5	200	202	-6	6	5	38	36	0	8	5	210	213	-2-11	6	170	-172
-5	2	5	109	105	-6	4	5	66	65	-5	6	5	36	-8	1	8	5	72	-71	-1-11	6	54	-58
-4	2	5	126	124	-5	4	5	180	-182	-3	6	5	91	-80	2	8	5	95	-91	1-11	6	42	-39
-3	2	5	50	59	-3	4	5	344	348	-2	6	5	49	-55	3	8	5	101	109	2-11	6	49	-43
-2	2	5	46	59	-2	4	5	303	306	-1	6	5	51	-57	4	8	5	110	116	3-11	6	31	20
-1	2	5	325	333	-1	4	5	127	-133	0	6	5	29	6	5	8	5	30	-7	4-11	6	86	86
1	2	5	175	-183	0	4	5	381	-382	1	6	5	78	-87	6	8	5	56	-63	5-11	6	67	67
2	2	5	411	406	1	4	5	63	63	2	6	5	152	-157	-6	9	5	65	-51	6-11	6	32	53
4	2	5	336	-331	2	4	5	186	180	4	6	5	160	153	-5	9	5	60	-69	7-11	6	33	33
5	2	5	277	-282	3	4	5	123	-129	5	6	5	128	131	-2	9	5	95	-103	-8-10	6	73	64
6	2	5	36	37	4	4	5	122	-115	7	6	5	76	-70	-1	9	5	75	-79	-7-10	6	42	40
7	2	5	233	236	5	4	5	114	-114	-10	7	5	49	47	0	9	5	131	142	-6-10	6	84	-88
9	2	5	90	-95	6	4	5	67	65	-9	7	5	82	-81	1	9	5	144	145	-5-10	6	60	-58
-12	3	5	65	46	7	4	5	126	129	-8	7	5	85	-89	4	9	5	47	44	-4-10	6	43	39
-11	3	5	100	-102	-11	5	5	42	-26	-7	7	5	103	100	-4	10	5	38	-62	-3-10	6	86	82
-10	3	5	163	-171	-9	5	5	28	2	-6	7	5	213	214	1	10	5	48	47	-2-10	6	82	-80
-8	3	5	181	181	-8	5	5	77	-82	-5	7	5	33	-18	2	10	5	72	63	-1-10	6	244	-247
-7	3	5	49	-58	-7	5	5	31	13	-4	7	5	287	-279	0-14	6	73	68	0-10	6	126	-129
-6	3	5	335	-337	-6	5	5	182	181	-3	7	5	110	-113	1-14	6	108	-96	1-10	6	171	168
-5	3	5	42	-50	-5	5	5	82	85	-2	7	5	141	145	2-14	6	89	-100	2-10	6	222	219
-4	3	5	366	373	-4	5	5	115	-120	-1	7	5	42	49	-3-13	6	54	53	3-10	6	150	-143

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
4	-10	6	111	-114	10	-8	6	110	-103	7	-6	6	73	77	5	-4	6	165	167	2	-2	6	292	-289
5	-10	6	182	178	-9	-7	6	86	-91	8	-6	6	104	98	6	-4	6	64	57	3	-2	6	516	-516
6	-10	6	182	181	-8	-7	6	40	32	9	-6	6	78	75	7	-4	6	78	-71	4	-2	6	82	85
8	-10	6	81	-83	-7	-7	6	194	194	-9	-5	6	50	-45	8	-4	6	60	-61	5	-2	6	524	531
-4	-9	6	116	-121	-6	-7	6	44	-42	-8	-5	6	53	-49	9	-4	6	113	105	6	-2	6	136	140
-2	-9	6	26	-14	-5	-7	6	149	-150	-6	-5	6	63	-71	10	-4	6	129	125	7	-2	6	116	-117
-1	-9	6	207	-203	-4	-7	6	37	-58	-5	-5	6	25	21	-10	-3	6	50	44	8	-2	6	31	-6
0	-9	6	161	-155	-3	-7	6	198	200	-4	-5	6	66	68	-9	-3	6	29	-7	10	-2	6	51	-43
1	-9	6	59	52	-2	-7	6	234	240	-3	-5	6	132	-130	-8	-3	6	172	-176	-12	-1	6	59	-43
2	-9	6	328	327	-1	-7	6	116	-109	-2	-5	6	106	-90	-7	-3	6	137	-139	-10	-1	6	62	53
3	-9	6	315	309	0	-7	6	138	-191	-1	-5	6	469	484	-6	-3	6	93	89	-9	-1	6	54	53
4	-9	6	160	-153	1	-7	6	135	179	0	-5	6	636	635	-5	-3	6	467	478	-7	-1	6	50	57
5	-9	6	239	-234	2	-7	6	427	410	1	-5	6	102	-112	-4	-3	6	65	67	-6	-1	6	38	41
6	-9	6	135	140	3	-7	6	204	-208	2	-5	6	614	-616	-3	-3	6	579	-598	-5	-1	6	118	-114
7	-9	6	186	188	4	-7	6	213	-204	3	-5	6	285	-277	-2	-3	6	149	147	-4	-1	6	236	-225
8	-9	6	56	-59	5	-7	6	98	93	4	-5	6	280	285	-1	-3	6	312	303	-3	-1	6	391	394
9	-9	6	142	-145	6	-7	6	39	-86	5	-5	6	89	-83	0	-3	6	59	63	-2	-1	6	194	174
-9	-8	6	66	56	7	-7	6	135	-134	6	-5	6	442	-446	1	-3	6	496	-498	-1	-1	6	691	-717
-8	-8	6	117	114	9	-7	6	78	77	7	-5	6	110	-107	2	-3	6	307	-311	0	-1	6	86	-95
-7	-8	6	44	32	11	-7	6	56	-58	8	-5	6	204	198	3	-3	6	84	81	1	-1	6	282	278
-6	-8	6	115	-115	-10	-6	6	34	28	9	-5	6	201	204	4	-3	6	55	63	2	-1	6	433	446
-5	-8	6	37	-36	-9	-6	6	132	-132	10	-5	6	28	25	5	-3	6	157	152	3	-1	6	278	-270
-4	-8	6	118	116	-7	-6	6	53	-52	11	-5	6	67	-66	6	-3	6	115	114	4	-1	6	489	-475
-3	-8	6	41	34	-6	-6	6	80	72	-11	-4	6	50	49	7	-3	6	117	108	5	-1	6	263	255
-2	-8	6	169	-166	-5	-6	6	86	89	-9	-4	6	155	-162	8	-3	6	50	64	6	-1	6	380	391
-1	-8	6	66	-55	-4	-6	6	176	-133	-8	-4	6	226	-235	11	-3	6	46	44	7	-1	6	109	115
0	-8	6	43	44	-3	-6	6	230	-236	-7	-4	6	37	48	-10	-2	6	33	-28	8	-1	6	205	-202
1	-8	6	61	49	-2	-6	6	487	494	-6	-4	6	94	97	-9	-2	6	37	36	9	-1	6	173	-172
2	-8	6	66	61	-1	-6	6	516	531	-5	-4	6	55	49	-6	-2	6	63	-67	10	-1	6	43	42
3	-8	6	121	127	0	-6	6	240	-250	-4	-4	6	152	-153	-5	-2	6	226	229	11	-1	6	53	60
4	-8	6	237	238	1	-6	6	489	-491	-3	-4	6	129	-125	-4	-2	6	652	679	-11	0	6	105	-101
5	-8	6	144	-142	2	-6	6	31	-22	-2	-4	6	264	263	-3	-2	6	161	165	-9	0	6	263	271
6	-8	6	219	-219	3	-6	6	178	182	-1	-4	6	52	48	-2	-2	6	460	-494	-8	0	6	153	154
7	-8	6	45	51	4	-6	6	108	-108	1	-4	6	75	69	-1	-2	6	229	-236	-7	0	6	114	-115
8	-8	6	69	70	5	-6	6	410	-400	2	-4	6	120	114	0	-2	6	399	405	-6	0	6	86	-93
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3	0	6	315	308	-4	2	6	460	-464	-7	4	6	153	157	0	6	6	117	118	-1	10	6	100	-100
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-9	1	6	27	36	8	2	6	26	-9	7	4	6	92	-87	1	7	6	137	145	-2	-12	7	44	58
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-5	1	6	383	-377	-7	3	6	105	-102	-8	5	6	149	-147	5	7	6	100	92	2	-12	7	98	95
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-3	1	6	336	340	-4	3	6	151	-147	-5	5	6	104	95	-8	8	6	63	-69	4	-12	7	172	-172
-2	1	6	404	-411	-3	3	6	115	-110	-4	5	6	263	-257	-7	8	6	114	-116	6	-12	7	108	105
-1	1	6	360	-377	-2	3	6	226	226	-3	5	6	123	-123	-5	8	6	123	116	-7	-11	7	41	-43
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6	-11	7	48	51	-4	-8	7	121	-123	-4	-6	7	170	-178	-6	-4	7	158	-159	-10	-2	7	73	-69
7	-11	7	192	193	-3	-8	7	304	-323	-3	-6	7	215	-226	-4	-4	7	214	209	-9	-2	7	126	124
8	-11	7	68	67	-1	-8	7	268	273	-2	-6	7	97	102	-3	-4	7	72	69	-8	-2	7	135	137
-8	-10	7	43	38	0	-8	7	176	-185	-1	-6	7	194	194	-2	-4	7	155	-165	-7	-2	7	211	-206
-7	-10	7	30	27	1	-8	7	365	-361	0	-6	7	55	-49	-1	-4	7	229	-236	-6	-2	7	160	-162
-6	-10	7	69	-64	2	-8	7	40	57	1	-6	7	112	115	0	-4	7	278	305	-5	-2	7	122	120
-5	-10	7	55	-57	3	-8	7	350	356	2	-6	7	202	204	1	-4	7	613	621	-4	-2	7	421	430
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-1	-10	7	134	-126	7	-8	7	61	61	7	-6	7	146	-152	5	-4	7	247	239	1	-2	7	210	-194
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-2	-9	7	167	173	0	-7	7	252	260	-1	-5	7	310	324	-1	-3	7	142	140	-9	-1	7	91	-97
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7	-9	7	61	55	-10	-6	7	68	70	10	-5	7	27	32	8	-3	7	75	-66	0	-1	7	288	-295
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10	-9	7	32	-34	-7	-6	7	77	77	-9	-4	7	36	28	10	-3	7	128	124	2	-1	7	85	-76
-6	-8	7	145	141	-6	-6	7	184	186	-8	-4	7	94	97	11	-3	7	145	138	3	-1	7	229	-235

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5	-1	7	48	44	1	1	7	317	-321	-2	3	7	27	25	4	5	7	111	-112	-1	8	7	93	-90
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-11	0	7	50	-41	7	1	7	47	42	4	3	7	203	213	-7	6	7	185	-193	-1	9	7	31	28
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1	-11	8	98	-93	-7	-8	8	42	-29	-7	-6	8	91	81	-9	-4	8	116	110	-10	-2	8	69	58
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5	-11	8	133	-129	-3	-8	8	36	26	-2	-6	8	332	-331	-5	-4	8	28	-33	-4	-2	8	213	-218
8	-11	8	29	26	-2	-8	8	202	204	-1	-6	8	338	-350	-4	-4	8	124	134	-3	-2	8	43	-38
-9	-10	8	34	-20	-1	-8	8	262	275	0	-6	8	52	-58	-3	-4	8	27	-27	-2	-2	8	436	462
-7	-10	8	45	-26	1	-8	8	178	-182	1	-6	8	292	288	-2	-4	8	249	-260	-1	-2	8	254	258
-5	-10	8	52	51	2	-8	8	48	-61	2	-6	8	105	95	-1	-4	8	184	-191	0	-2	8	139	-142
-3	-10	8	71	-83	4	-8	8	86	-91	3	-6	8	218	-222	0	-4	8	90	88	1	-2	8	246	-249
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2	-10	8	164	-167	9	-8	8	32	30	7	-6	8	118	-116	4	-4	8	140	138	5	-2	8	366	-380
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5	-10	8	106	-108	-9	-7	8	44	51	-8	-5	8	36	-22	9	-4	8	28	-12	8	-2	8	115	123
6	-10	8	133	-132	-8	-7	8	39	-14	-7	-5	8	99	-102	10	-4	8	98	-87	9	-2	8	37	-29
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H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
8	-6	13	64	-58	-5	-3	13	244	-245	-6	2	13	75	-73	0	3	13	76	-80	-1	-8	14	113	-112
-9	-5	13	41	43	-4	-3	13	110	-104	-7	0	13	63	62	1	3	13	36	14	1	-8	14	147	147
-8	-5	13	44	30	-3	-3	13	179	171	-6	0	13	103	110	3	3	13	32	-29	2	-8	14	71	59
-7	-5	13	30	5	-2	-3	13	73	32	-5	0	13	129	-105	-4	4	13	70	61	3	-8	14	127	-130
-6	-5	13	31	29	-1	-3	13	147	-149	-4	0	13	84	-81	-3	4	13	48	-35	4	-8	14	122	-127
-5	-5	13	57	47	0	-3	13	106	-109	-3	0	13	109	100	-2	4	13	74	-78	5	-8	14	31	-22
-4	-5	13	26	21	1	-3	13	95	98	-2	0	13	96	95	-2	-12	14	30	-10	6	-8	14	57	45
-3	-5	13	25	19	2	-3	13	175	166	-1	0	13	72	77	-1	-12	14	64	60	-5	-7	14	33	35
-2	-5	13	94	96	3	-3	13	69	73	1	0	13	33	-24	0	-12	14	49	52	-3	-7	14	37	24
-1	-5	13	24	17	4	-3	13	105	-107	2	0	13	34	-27	2	-12	14	43	-38	-2	-7	14	28	25
0	-5	13	146	-148	5	-3	13	71	-72	3	0	13	60	-66	-4	-11	14	65	-42	2	-7	14	34	47
1	-5	13	29	-3	7	-3	13	43	-41	4	0	13	49	-64	-2	-11	14	28	-17	4	-7	14	70	-79
2	-5	13	119	116	-8	-2	13	60	-46	-8	1	13	79	-73	1	-11	14	88	81	5	-7	14	91	-100
3	-5	13	79	80	-7	-2	13	50	-53	-7	1	13	108	-98	4	-11	14	45	44	7	-7	14	69	75
4	-5	13	65	-66	-6	-2	13	96	92	-6	1	13	71	68	-5	-10	14	56	61	-8	-6	14	46	45
5	-5	13	57	-53	-4	-2	13	203	-198	-5	1	13	139	134	-4	-10	14	37	-20	-6	-6	14	47	-51
6	-5	13	75	80	-3	-2	13	73	-67	-3	1	13	63	-66	-3	-10	14	51	-50	-4	-6	14	92	95
7	-5	13	83	80	-2	-2	13	123	129	-1	1	13	173	175	-1	-10	14	67	64	-3	-6	14	120	118
8	-5	13	29	-18	-1	-2	13	166	167	0	1	13	107	109	1	-10	14	39	-21	-2	-6	14	48	-41
-9	-4	13	43	33	0	-2	13	72	-77	1	1	13	78	-81	2	-10	14	45	45	-1	-6	14	91	-98
-8	-4	13	75	72	1	-2	13	125	-125	2	1	13	82	-74	-6	-9	14	118	-112	1	-6	14	47	57
-6	-4	13	106	-103	2	-2	13	112	122	4	1	13	42	18	-4	-9	14	93	93	2	-6	14	28	38
-5	-4	13	33	-43	3	-2	13	216	216	5	1	13	41	-33	-3	-9	14	29	-19	3	-6	14	28	-17
-4	-4	13	113	119	5	-2	13	155	-149	-6	2	13	29	-34	-2	-9	14	105	-98	7	-6	14	61	49
-3	-4	13	48	47	6	-2	13	72	-77	-4	2	13	75	69	-1	-9	14	46	44	-8	-5	14	31	-2
-2	-4	13	134	-129	-6	-1	13	129	-133	-2	2	13	137	-137	0	-9	14	152	162	-7	-5	14	67	66
-1	-4	13	47	-50	-5	-1	13	47	-45	-1	2	13	43	-56	1	-9	14	55	66	-6	-5	14	38	32
0	-4	13	48	51	-4	-1	13	56	47	0	2	13	66	63	2	-9	14	71	-77	-5	-5	14	41	-44
1	-4	13	59	65	-2	-1	13	68	-52	1	2	13	42	43	3	-9	14	76	-82	-4	-5	14	53	-54
2	-4	13	53	45	-1	-1	13	83	86	2	2	13	48	-55	5	-9	14	44	38	-3	-5	14	117	122
3	-4	13	80	80	0	-1	13	139	137	3	2	13	77	-75	6	-9	14	44	-28	-2	-5	14	144	147
4	-4	13	35	42	1	-1	13	59	-59	4	2	13	54	34	-7	-8	14	30	-13	-1	-5	14	97	-101
5	-4	13	61	-52	2	-1	13	120	-120	-6	3	13	53	40	-6	-8	14	64	-62	0	-5	14	212	-219
6	-4	13	50	-51	4	-1	13	90	93	-5	3	13	39	44	-5	-8	14	79	-90	1	-5	14	86	-84
-9	-3	13	49	-33	6	-1	13	106	-110	-4	3	13	33	32	-3	-8	14	81	91	2	-5	14	75	72
-7	-3	13	93	93	7	-1	13	50	-55	-1	3	13	100	-103	-2	-8	14	52	-45	3	-5	14	62	72

H	K	L	10FO	10FC
4	-5	14	56	-52
5	-5	14	42	-46
6	-5	14	86	88
7	-5	14	74	76
-7	-4	14	34	25
-6	-4	14	62	69
-5	-4	14	48	50
-4	-4	14	64	-63
-3	-4	14	37	-46
-2	-4	14	47	31
-1	-4	14	82	86
1	-4	14	191	-190
2	-4	14	48	-33
3	-4	14	141	142
4	-4	14	85	77
5	-4	14	65	-70
6	-4	14	28	-17
7	-4	14	76	69
-6	-3	14	40	41
-7	-3	14	77	80
-5	-3	14	52	-58
-4	-3	14	31	-36
-2	-3	14	85	-86
-1	-3	14	132	-129
0	-3	14	42	53
2	-3	14	91	-91
3	-3	14	59	51
4	-3	14	141	136
5	-3	14	74	73
6	-3	14	32	-31
-8	-2	14	58	-46
-7	-2	14	72	66
-6	-2	14	151	162
-4	-2	14	183	-135
-3	-2	14	77	-69
-2	-2	14	41	38

H	K	L	10FO	10FC
-1	-2	14	36	-38
0	-2	14	88	-78
1	-2	14	32	-34
-7	-1	14	73	-70
-5	-1	14	189	191
-3	-1	14	133	-126
-2	-1	14	63	-65
-1	-1	14	116	118
0	-1	14	147	143
1	-1	14	60	-54
2	-1	14	50	-45
3	-1	14	38	42
4	-1	14	48	41
5	-1	14	33	-15
-6	0	14	62	-55
-5	0	14	33	-38
-4	0	14	74	67
-3	0	14	41	32
-2	0	14	67	-59
2	0	14	163	162
1	0	14	98	113
2	0	14	44	-32
3	0	14	83	-82
5	0	14	49	45
-1	1	14	29	15
0	1	14	39	51
1	1	14	71	69
3	1	14	65	-73
4	1	14	88	-75
-4	2	14	78	71
-1	2	14	53	52
0	2	14	79	84
1	2	14	50	45
3	2	14	39	-51
-4	3	14	34	20
-3	3	14	67	62

H	K	L	10FO	10FC
-1	3	14	74	-68
1	3	14	68	65
-2	-11	15	87	-82
-1	-11	15	53	-49
0	-11	15	61	58
1	-11	15	73	63
2	-11	15	42	-44
3	-11	15	76	-82
-4	-10	15	32	-22
-2	-10	15	45	-41
-1	-10	15	93	-88
0	-10	15	26	-31
1	-10	15	55	62
2	-10	15	78	84
3	-10	15	48	-37
4	-10	15	75	-75
-5	-9	15	67	59
-3	-9	15	40	-31
-1	-9	15	46	-51
0	-9	15	22	-1
2	-9	15	34	24
3	-9	15	57	57
5	-9	15	62	-57
-6	-8	15	55	-53
-5	-8	15	69	-61
-3	-8	15	73	69
-1	-8	15	65	-63
1	-8	15	84	85
2	-8	15	53	59
4	-8	15	27	-26
5	-8	15	34	-37
-6	-7	15	28	5
-5	-7	15	58	-61
-4	-7	15	89	-80
-3	-7	15	29	44
-2	-7	15	155	157

H	K	L	10FO	10FC
-1	-7	15	47	40
0	-7	15	65	-70
2	-7	15	88	88
3	-7	15	64	62
4	-7	15	64	-73
5	-7	15	125	-116
-7	-6	15	28	-10
-5	-6	15	49	-26
-2	-6	15	109	108
-1	-6	15	138	130
0	-6	15	73	85
2	-6	15	38	-44
3	-6	15	41	37
5	-6	15	106	-103
6	-6	15	105	-103
-7	-5	15	54	50
-5	-5	15	63	-67
-4	-5	15	48	-46
-3	-5	15	30	27
-2	-5	15	66	64
0	-5	15	34	49
1	-5	15	71	72
4	-5	15	35	36
6	-5	15	37	-38
-7	-4	15	28	-1
-6	-4	15	98	93
-5	-4	15	33	34
-4	-4	15	93	-97
-3	-4	15	77	-79
-2	-4	15	55	65
0	-4	15	60	-66
1	-4	15	102	-90
2	-4	15	77	-66
4	-4	15	42	34
-7	-3	15	64	-60
-5	-3	15	131	127

H	K	L	10FO	10FC
-4	-3	15	69	66
-3	-3	15	111	-112
-2	-3	15	83	-80
0	-3	15	80	76
1	-3	15	93	-89
2	-3	15	173	-173
3	-3	15	41	-25
4	-3	15	86	84
5	-3	15	65	70
-5	-2	15	31	-28
-4	-2	15	32	39
-2	-2	15	65	-55
-1	-2	15	52	-61
1	-2	15	52	54
2	-2	15	67	-69
3	-2	15	90	-88
5	-2	15	117	120
-6	-1	15	58	46
-5	-1	15	46	52
-4	-1	15	30	-29
-3	-1	15	54	-57
-2	-1	15	52	-57
-1	-1	15	50	-31
2	-1	15	38	17
-6	0	15	42	-25
-5	0	15	34	21
-4	0	15	96	86
-2	0	15	84	-78
-1	0	15	46	-63
1	0	15	54	46
-3	1	15	83	75
-1	1	15	59	-67
0	1	15	43	-64
1	1	15	50	53
2	1	15	97	91
-2	2	15	66	60

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
-1	2	15	31	34	-2	-7	16	41	45	2	-5	16	147	141	1	-2	16	39	45	-1	-6	17	71	-72
-2	-10	16	34	10	2	-7	16	57	44	1	-5	16	30	34	3	-2	16	41	-37	0	-6	17	48	-53
-1	-10	16	75	-70	4	-7	16	43	35	2	-5	16	123	-119	-3	-1	16	114	110	1	-6	17	35	36
0	-10	16	63	-71	-5	-6	16	32	36	3	-5	16	69	-62	-1	-1	16	64	-59	2	-6	17	53	62
2	-10	16	29	21	-4	-6	16	44	-33	4	-5	16	56	66	0	-1	16	86	-80	-3	-5	17	52	-42
-4	-9	16	73	-64	-3	-6	16	80	-72	1	-4	16	87	91	1	-1	16	30	5	-2	-5	17	66	-56
-2	-9	16	42	47	-1	-6	16	112	116	2	-4	16	29	-5	2	-1	16	62	69	-4	-4	17	43	47
-1	-9	16	40	-30	1	-6	16	63	-59	3	-4	16	113	-108	0	-9	17	39	-45	-3	-4	17	44	36
0	-9	16	108	-113	2	-6	16	36	-36	4	-4	16	59	-60	-1	-8	17	74	79	-2	-4	17	69	-52
1	-9	16	67	-65	3	-6	16	64	64	-5	-3	16	47	53	1	-8	17	87	-74	-1	-4	17	94	-86
2	-9	16	31	29	4	-6	16	78	70	-1	-3	16	49	42	2	-8	17	38	-48	0	-4	17	45	31
3	-9	16	67	74	-6	-5	16	35	-26	0	-3	16	39	23	-2	-7	17	87	-90	1	-4	17	114	104
0	-8	16	46	-47	-5	-5	16	37	36	4	-3	16	36	-53	0	-7	17	69	72	-3	-3	17	47	41
3	-8	16	30	23	-4	-5	16	39	32	-4	-2	16	101	101	1	-7	17	40	41	-1	-3	17	41	-30
4	-8	16	96	97	-3	-5	16	48	-45	-3	-2	16	82	83	2	-7	17	50	-43	0	-3	17	32	-41
-4	-7	16	38	-51	-2	-5	16	57	-55	-2	-2	16	55	-48	-4	-6	17	32	-21	-2	-2	17	47	29
-3	-7	16	29	-6	-1	-5	16	63	67	-1	-2	16	70	-63	-2	-6	17	48	-41	0	-2	17	67	47

Appendix 2.4 Compound (7)

Coordinates x 10^4 for hydrogen atoms

	x/a	y/b	z/c
H11	3417	2754	2486
H21	7300	3497	2439
H31	6454	4783	1126
H41	1997	4818	283
H51	23	3579	1127
H71	5732	6238	2699
H81	7058	4975	4044
H91	3373	4126	4219
H101	-282	4858	3001
H131	-1463	7777	1095
H141	-451	8799	-58
H151	2851	8489	-674
H161	5263	7210	-65
H171	4346	6208	1136

Appendix 2.5 Compound (7)

Anisotropic temperature factors X 10^3
with e.s.d's in parentheses:

	U11	U22	U33	U23	U13	U12
FE	48(1)	29(1)	31(1)	-4(1)	5(1)	-2(1)
C1	110(6)	29(3)	53(4)	-9(3)	22(4)	3(3)
C2	67(4)	47(4)	57(4)	-8(3)	9(3)	20(3)
C3	66(4)	52(4)	49(3)	-12(3)	20(3)	5(3)
C4	75(4)	50(4)	30(3)	-8(2)	4(3)	7(3)
C5	59(4)	57(4)	71(4)	-32(3)	10(3)	-6(3)
C6	44(3)	28(2)	37(3)	-2(2)	7(2)	0(2)
C7	48(3)	32(3)	39(3)	-5(2)	10(2)	-6(2)
C8	50(4)	37(3)	34(3)	-3(2)	9(2)	-4(2)
C9	65(4)	37(3)	35(3)	3(2)	14(3)	-4(3)
C10	36(3)	46(3)	47(3)	-2(3)	18(3)	-2(2)
C11	47(3)	39(3)	54(3)	-6(3)	13(3)	-2(3)
C12	41(3)	32(3)	36(3)	-1(2)	7(2)	-3(2)
C13	45(3)	40(3)	59(4)	0(3)	7(3)	9(3)
C14	70(4)	40(3)	60(4)	6(3)	3(3)	6(3)
C15	80(4)	44(3)	37(3)	5(3)	6(3)	-12(3)
C16	53(3)	45(3)	39(3)	-5(2)	6(3)	-7(3)
C17	43(3)	34(3)	41(3)	-3(2)	5(2)	0(2)
O18	45(2)	90(4)	114(4)	41(3)	30(2)	13(2)

Appendix 2.6

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR Benzoylferrocene, (7),

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
1	1	0	222	215	3	8	0	244	-237	-2	1	1	801	772	2	5	1	82	-91	-2	9	1	473	-479
2	1	0	61	76	4	8	0	48	20	-1	1	1	1223	-1234	3	5	1	70	55	-1	9	1	287	292
5	1	0	160	174	5	8	0	69	50	0	1	1	245	-260	4	5	1	207	193	0	9	1	123	129
0	2	0	816	820	1	9	0	607	-608	1	1	1	1322	1331	5	5	1	228	-224	1	9	1	453	-460
1	2	0	1040	-1015	2	9	0	206	220	2	1	1	504	-509	-5	6	1	114	-105	2	9	1	440	445
2	2	0	195	-188	3	9	0	87	-61	5	1	1	267	-278	-4	6	1	444	-415	4	9	1	47	-30
5	2	0	45	-43	5	9	0	179	160	-5	2	1	52	63	-3	6	1	638	619	-5	10	1	153	146
1	3	0	901	-862	0	10	0	401	-422	-2	2	1	341	-306	-2	6	1	38	-33	-4	10	1	45	-47
2	3	0	449	440	1	10	0	286	296	-1	2	1	987	-969	-1	6	1	231	-242	-3	10	1	237	236
3	3	0	112	-104	3	10	0	388	-382	0	2	1	284	251	0	6	1	839	870	-2	10	1	211	-200
4	3	0	242	-229	4	10	0	170	161	1	2	1	489	-468	1	6	1	317	-316	-1	10	1	173	-185
5	3	0	340	341	1	11	0	124	-134	2	2	1	124	111	2	6	1	119	-141	0	10	1	327	343
0	4	0	261	239	2	11	0	161	167	5	2	1	104	98	3	6	1	522	493	1	10	1	249	-240
1	4	0	449	-434	5	11	0	47	34	-5	3	1	194	205	4	6	1	256	-234	2	10	1	65	-65
2	4	0	46	-58	0	12	0	286	-306	-3	3	1	289	-289	5	6	1	78	84	3	10	1	116	113
3	4	0	455	430	1	12	0	152	147	-2	3	1	342	351	-4	7	1	127	-112	4	10	1	95	-85
4	4	0	142	-132	2	12	0	97	110	-1	3	1	701	-678	-3	7	1	45	26	5	10	1	62	-5
5	4	0	57	43	3	12	0	253	-232	0	3	1	402	394	-2	7	1	193	-190	-5	11	1	123	-108
1	5	0	574	-587	4	12	0	247	231	2	3	1	362	-347	0	7	1	44	47	-4	11	1	181	161
2	5	0	736	730	1	13	0	96	89	5	3	1	263	-252	1	7	1	488	-484	-3	11	1	74	30
3	5	0	182	-172	3	13	0	63	-33	-5	4	1	76	66	2	7	1	210	205	-2	11	1	311	-317
4	5	0	371	-357	4	13	0	59	29	-3	4	1	488	488	3	7	1	68	-72	-1	11	1	153	152
5	5	0	186	168	0	14	0	219	-227	-2	4	1	121	-117	5	7	1	71	-60	0	11	1	185	187
0	6	0	349	341	1	14	0	133	136	-1	4	1	444	-432	-5	8	1	104	102	1	11	1	240	-240
1	6	0	181	172	2	14	0	95	108	0	4	1	679	683	-4	8	1	238	-223	2	11	1	366	363
2	6	0	90	-93	3	14	0	345	-340	1	4	1	228	-221	-3	8	1	289	275	3	11	1	150	-135
4	6	0	189	-179	4	14	0	228	205	2	4	1	301	-290	-2	8	1	212	-204	4	11	1	163	-176
5	6	0	82	-94	1	15	0	116	129	3	4	1	491	460	-1	8	1	542	-532	5	11	1	142	119
1	7	0	405	-410	2	15	0	123	-135	5	4	1	130	-137	0	8	1	671	695	-2	12	1	75	-63
2	7	0	924	901	1	16	0	133	122	-5	5	1	236	247	1	8	1	448	-445	0	12	1	40	-31
3	7	0	230	-229	3	16	0	154	-161	-4	5	1	54	62	2	8	1	176	-156	3	12	1	110	104
4	7	0	166	-145	1	17	0	215	231	-3	5	1	226	-228	3	8	1	402	383	-5	13	1	162	-154
5	7	0	167	164	2	17	0	142	-150	-2	5	1	208	206	4	8	1	229	-215	-4	13	1	137	117
0	8	0	276	-273	0	18	0	71	-78	-1	5	1	182	-174	-5	9	1	155	-145	-2	13	1	243	-244
1	8	0	353	354	-5	1	1	283	306	0	5	1	388	370	-4	9	1	148	146	-1	13	1	274	277
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2	13	1	301	317	3	1	2	205	198	0	5	2	318	-312	-2	9	2	439	431	2	13	2	66	-60
3	13	1	132	-145	5	1	2	142	-161	1	5	2	419	432	-1	9	2	243	-252	3	13	2	68	71
4	13	1	160	-157	-5	2	2	33	-18	2	5	2	712	-689	1	9	2	348	374	4	13	2	55	59
-4	14	1	63	28	-4	2	2	274	289	3	5	2	337	299	2	9	2	265	-270	-4	14	2	164	-144
-3	14	1	73	-54	-3	2	2	523	-501	4	5	2	226	200	3	9	2	140	129	-3	14	2	188	193
-2	14	1	51	-45	-2	2	2	64	-52	5	5	2	83	-89	4	9	2	82	99	-1	14	2	273	-273
-1	14	1	231	252	-1	2	2	155	130	-4	6	2	145	127	5	9	2	173	-157	0	14	2	113	109
0	14	1	153	-145	0	2	2	1415	-1552	-3	6	2	38	9	-4	10	2	196	-204	1	14	2	129	-131
1	14	1	125	119	1	2	2	580	548	-2	6	2	175	-160	-3	10	2	290	273	2	14	2	112	-122
2	14	1	61	79	2	2	2	200	192	-1	6	2	127	124	-2	10	2	53	-36	3	14	2	253	252
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-2	15	1	336	-354	5	2	2	130	-140	1	6	2	73	-65	0	10	2	405	430	-4	15	2	68	7
-1	15	1	77	59	-5	3	2	245	244	3	6	2	197	-172	1	10	2	157	-160	-3	15	2	112	105
1	15	1	223	-235	-4	3	2	251	-238	4	6	2	135	140	2	10	2	135	-145	-2	15	2	167	-169
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-1	16	1	64	86	1	3	2	193	187	-2	7	2	398	409	-1	11	2	179	-186	-3	16	2	111	105
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-1	17	1	123	133	5	3	2	268	-263	2	7	2	616	-602	3	11	2	50	-84	2	16	2	80	-79
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0	1	2	290	290	-3	5	2	195	-187	-5	9	2	172	154	-2	13	2	47	-38	2	1	3	464	466
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5	1	3	157	148	0	5	3	167	-174	0	9	3	303	-294	3	13	3	79	82	-1	1	4	587	-569
-5	2	3	169	-185	1	5	3	53	-55	1	9	3	324	330	4	13	3	200	181	0	1	4	508	472
-4	2	3	191	196	3	5	3	46	53	2	9	3	272	-282	-3	14	3	126	105	1	1	4	232	-233
-3	2	3	437	-411	-4	6	3	524	483	3	9	3	71	68	-1	14	3	48	45	2	1	4	259	262
-2	2	3	84	87	-3	6	3	167	-170	4	9	3	47	52	0	14	3	139	141	3	1	4	78	73
-1	2	3	627	603	-2	6	3	182	-181	5	9	3	111	-83	1	14	3	52	-62	4	1	4	91	-78
1	2	3	291	285	-1	6	3	146	154	-5	10	3	127	-114	3	14	3	69	27	5	1	4	162	164
2	2	3	136	129	0	6	3	600	-622	-3	10	3	91	-47	-3	15	3	107	-102	-5	2	4	158	181
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4	2	3	304	296	2	6	3	504	492	-1	10	3	396	394	-1	15	3	112	-106	-2	2	4	128	135
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-5	3	3	151	-178	4	6	3	269	261	1	10	3	181	172	1	15	3	185	185	0	2	4	169	170
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2	3	3	309	296	4	7	3	83	-95	0	11	3	173	-177	-2	17	3	85	100	-2	3	4	511	-505
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-5	4	3	79	-96	-5	8	3	165	-162	2	11	3	303	-313	1	17	3	85	81	0	3	4	36	-10
-4	4	3	332	317	-4	8	3	171	151	3	11	3	122	134	-5	0	4	151	171	1	3	4	572	-589
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-2	4	3	65	-63	-2	8	3	47	23	5	11	3	143	-139	-3	0	4	606	600	3	3	4	228	-221
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2	4	3	252	-222	2	8	3	96	89	2	12	3	46	21	1	0	4	525	-498	-4	4	4	175	-190
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4	4	3	194	198	4	8	3	66	74	-4	13	3	95	-61	3	0	4	458	445	-2	4	4	78	-74
5	4	3	78	-78	5	8	3	59	39	-3	13	3	80	-81	4	0	4	106	-103	-1	4	4	222	-224
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-5	5	4	129	-140	2	8	4	114	90	4	12	4	77	53	4	1	5	222	240	2	5	5	310	-305
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-3	5	4	244	245	4	8	4	94	65	0	13	4	163	165	-5	2	5	121	132	5	5	5	49	-61
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1	5	4	135	-153	-1	9	4	62	61	-4	14	4	56	73	-1	2	5	382	-375	-2	6	5	162	182
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-2	6	4	312	298	-3	10	4	94	-81	-2	15	4	196	186	-3	3	5	493	-480	-4	7	5	86	-83
-1	6	4	148	142	-1	10	4	330	341	-1	15	4	115	-131	-2	3	5	468	472	-3	7	5	125	107
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1	6	4	221	213	1	10	4	92	90	2	15	4	112	-124	0	3	5	170	-166	-1	7	5	405	412
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3	6	4	73	54	4	10	4	189	184	-3	16	4	58	-7	4	3	5	114	94	1	7	5	131	-127
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-5	8	4	205	-211	-1	12	4	380	384	-1	1	5	325	-327	-3	5	5	173	-173	3	8	5	248	250
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-2	9	5	340	-341	-4	14	5	123	109	-1	2	6	667	691	3	5	6	76	-77	3	9	6	129	-123
-1	9	5	184	187	-3	14	5	95	-106	0	2	6	527	-537	4	5	6	231	246	4	9	6	136	135
0	9	5	53	-48	-2	14	5	79	60	1	2	6	95	98	5	5	6	201	-204	-5	10	6	134	121
1	9	5	242	-261	0	14	5	88	-105	2	2	6	466	459	-5	6	6	86	90	-4	10	6	133	-108
2	9	5	84	89	2	14	5	65	55	3	2	6	265	-269	-4	6	6	197	192	-3	10	6	80	76
4	9	5	170	-166	3	14	5	58	-60	4	2	6	69	49	-3	6	6	102	-95	-2	10	6	81	97
5	9	5	82	109	-3	15	5	128	151	5	2	6	93	97	-2	6	6	141	-140	-1	10	6	375	-397
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-3	10	5	147	142	1	15	5	171	-182	-3	3	6	454	-443	1	6	6	188	189	2	10	6	184	-205
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-5	11	5	168	-148	0	17	5	79	62	2	3	6	291	-292	-3	7	6	335	-339	-2	11	6	125	127
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-2	11	5	213	-209	-2	0	6	350	-336	5	3	6	96	-131	0	7	6	468	-484	1	11	6	93	111
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4	3	8	89	-76	-5	8	8	81	-84	0	12	8	212	-205	0	3	9	218	-233	-4	8	9	252	-234
5	3	8	90	86	-4	8	8	158	148	2	12	8	184	191	1	3	9	175	186	-3	8	9	81	88
-4	4	8	229	-210	-3	8	8	48	43	3	12	8	180	-177	2	3	9	182	-181	-2	8	9	61	57
-3	4	8	149	144	-2	8	8	214	-217	-4	13	8	57	-32	3	3	9	111	-106	-1	8	9	136	-160
-2	4	8	214	213	-1	8	8	136	139	-3	13	8	71	-71	4	3	9	178	228	0	8	9	301	304
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-3	1	12	127	123	0	6	12	91	-92	-4	2	13	57	-48	0	9	13	150	152	0	5	14	243	-238
-2	1	12	49	51	1	6	12	74	32	-2	2	13	148	148	1	9	13	96	-104	1	5	14	135	128
0	1	12	169	169	2	6	12	55	11	-1	2	13	138	-144	-3	10	13	99	-89	-1	6	14	46	-24
-5	2	12	169	204	-4	7	12	172	-157	1	2	13	124	134	-2	10	13	158	165	1	6	14	52	-24
-4	2	12	191	-168	-3	7	12	193	177	-4	3	13	217	225	-1	10	13	101	-104	-4	7	14	148	120
-3	2	12	162	-169	-1	7	12	133	-135	-3	3	13	168	-173	0	10	13	62	-21	-3	7	14	159	-135
-2	2	12	140	136	0	7	12	226	224	-1	3	13	67	64	-2	11	13	137	-141	-2	7	14	91	92
-1	2	12	235	-226	1	7	12	138	-138	0	3	13	343	-340	-1	11	13	115	-103	-1	7	14	133	128
1	2	12	235	236	-4	8	12	100	84	1	3	13	100	78	-3	0	14	135	145	0	7	14	238	-235
2	2	12	299	-294	-1	8	12	76	72	-4	4	13	124	-143	-2	0	14	299	-285	1	7	14	80	102
3	2	12	151	154	0	8	12	55	-83	-2	4	13	253	253	1	0	14	293	-290	-4	8	14	72	-65
-5	3	12	60	16	2	8	12	177	187	-1	4	13	317	-327	2	0	14	320	310	-3	8	14	69	-35
-4	3	12	70	-52	-5	9	12	58	9	0	4	13	104	90	-4	1	14	51	38	-2	8	14	120	102
-3	3	12	180	179	-4	9	12	102	-76	1	4	13	115	96	-3	1	14	55	18	-1	8	14	48	-51
-2	3	12	200	-198	-3	9	12	128	118	2	4	13	261	-256	-2	1	14	103	122	-3	9	14	157	-133
0	3	12	213	210	-2	9	12	76	-86	-4	5	13	104	114	1	1	14	48	-34	-2	9	14	53	-23
1	3	12	130	-133	-1	9	12	94	-93	-3	5	13	79	-72	-3	2	14	45	20	-1	9	14	121	104
-5	4	12	94	97	0	9	12	197	207	-2	5	13	68	61	-2	2	14	116	-122	0	9	14	103	-94
-4	4	12	63	-73	1	9	12	98	-111	-1	5	13	50	33	1	2	14	241	-234	-2	10	14	176	174
-3	4	12	40	-23	-4	10	12	115	91	0	5	13	108	-107	2	2	14	176	169	-1	10	14	201	-196
-2	4	12	121	125	-2	10	12	147	-126	-4	6	13	110	-97	-4	3	14	147	138	-3	1	15	195	195

OBSERVED AND CALCULATED STRUCTURE FACTORS FOR MOX

H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC	H	K	L	10FO	10FC
-1	1	15	148	-155	-2	3	15	48	-54	0	4	15	47	13	-2	8	15	172	-159	-2	3	16	49	-69
0	1	15	226	228	-1	3	15	91	-87	1	4	15	181	-193	-1	8	15	95	99	-1	3	16	83	-56
1	1	15	142	-142	0	3	15	251	238	-1	5	15	83	-73	-2	0	16	143	145	0	3	16	57	66
-3	2	15	51	28	1	3	15	128	-128	0	5	15	134	112	0	0	16	73	54	-1	4	16	99	-92
-2	2	15	91	-90	-3	4	15	92	87	-2	6	15	182	-184	-2	1	16	44	-33	-2	5	16	59	-45
-1	2	15	176	165	-2	4	15	309	-308	-1	6	15	203	202	-2	2	16	213	201	-1	5	16	130	-117
-3	3	15	84	63	-1	4	15	175	174															

Appendix 3

Crystallographic data for 1,4-diphenyl-1,4-epithio-2,3-dithia[4](1,1')ferrocenophane.

Appendix 3.1

Deposition data

Calculated hydrogen coordinates (C–H 0.95 Å, Biso 5 Å^2)

Atom	x	y	z
H12	−0.1159	0.3802	0.0859
H13	−0.0005	0.2596	0.2077
H14	0.0032	0.0584	0.1484
H15	−0.1114	0.0528	−0.0098
H22	0.0937	0.4432	−0.0119
H23	0.2337	0.3364	0.1047
H24	0.2470	0.1341	0.0508
H25	0.1196	0.1140	−0.1023
H32	−0.3656	0.3996	−0.1880
H33	−0.5501	0.4527	−0.1682
H34	−0.6279	0.3883	−0.0430
H35	−0.5196	0.2735	0.0646
H36	−0.3314	0.2258	0.0484
H42	0.1989	0.3642	−0.1907
H43	0.2720	0.4782	−0.2964
H44	0.1491	0.5773	−0.4050
H45	−0.0466	0.5567	−0.4123
H46	−0.1210	0.4382	−0.3095

Appendix 3.2

Deposition data

General Temperature Factor Expressions - U's

Name	U(1,1)	U(2,2)	U(3,3)	U(1,2)	U(1,3)	U(2,3)
Fe	0.0349(2)	0.0377(2)	0.0270(2)	-0.0002(2)	0.0035(2)	0.0034(2
S1	0.0474(4)	0.0512(4)	0.0419(4)	-0.0167(3)	0.0121(3)	-0.0152(3
S2	0.0415(4)	0.0487(4)	0.0394(4)	-0.0024(3)	0.0095(3)	-0.0158(3
S3	0.0315(3)	0.0321(3)	0.0306(3)	0.0020(3)	0.0086(2)	0.0015(3
C11	0.035(1)	0.040(1)	0.032(1)	-0.002(1)	0.012(1)	0.001(1)
C12	0.046(2)	0.042(2)	0.032(1)	-0.002(1)	0.011(1)	0.001(1)
C13	0.056(2)	0.057(2)	0.027(1)	-0.004(2)	0.007(1)	0.004(1)
C14	0.051(2)	0.049(2)	0.041(1)	0.000(1)	0.010(1)	0.013(1)
C15	0.043(1)	0.037(1)	0.041(1)	-0.004(1)	0.011(1)	0.003(1)
C16	0.033(1)	0.036(1)	0.029(1)	-0.004(1)	0.009(1)	-0.002(1)
C21	0.031(1)	0.039(1)	0.027(1)	-0.001(1)	0.007(1)	0.001(1)
C22	0.038(1)	0.045(2)	0.034(1)	-0.009(1)	0.006(1)	-0.001(1)
C23	0.037(1)	0.068(2)	0.036(1)	-0.008(2)	0.000(1)	0.001(2)
C24	0.034(1)	0.069(2)	0.045(2)	0.011(1)	0.003(1)	0.013(2)
C25	0.039(1)	0.044(2)	0.038(1)	0.010(1)	0.010(1)	0.004(1)
C26	0.031(1)	0.032(1)	0.027(1)	-0.000(1)	0.008(1)	-0.001(1)
C31	0.030(1)	0.049(2)	0.035(1)	-0.007(1)	0.010(1)	-0.006(1)
C32	0.035(1)	0.077(2)	0.041(2)	-0.000(2)	0.009(1)	0.006(2)
C33	0.036(2)	0.095(3)	0.056(2)	0.011(2)	0.002(2)	0.004(2)
C34	0.035(2)	0.093(3)	0.067(2)	0.003(2)	0.020(1)	-0.008(2)
C35	0.045(2)	0.093(3)	0.053(2)	-0.005(2)	0.026(1)	0.001(2)
C36	0.042(2)	0.062(2)	0.047(2)	-0.002(2)	0.016(1)	0.005(2)
C41	0.038(1)	0.041(1)	0.030(1)	0.002(1)	0.013(1)	0.002(1)

General Temperature Factor Expressions - U's (Continued)

Name	U(1,1)	U(2,2)	U(3,3)	U(1,2)	U(1,3)	U(2,3)
C42	0.041(2)	0.111(3)	0.051(2)	0.000(2)	0.014(1)	0.031(2)
C43	0.041(2)	0.179(4)	0.075(2)	-0.018(2)	0.022(2)	0.050(2)
C44	0.072(2)	0.108(3)	0.068(2)	-0.013(2)	0.033(2)	0.037(2)
C45	0.071(2)	0.087(2)	0.064(2)	0.021(2)	0.029(2)	0.044(2)
C46	0.044(2)	0.075(2)	0.048(2)	0.008(2)	0.016(1)	0.025(2)

The form of the anisotropic thermal parameter is:

exp[-2PI2{h2a2U(1,1) + k2b2U(2,2) + l2c2U(3,3) + 2hkabU(1,2) + 2hlacU(1,3) + 2klbcU(2,3)}] where a, b, and c are reciprocal lattice constants.

Appendix 3.3 Tables of Least Squares Planes.

The equation of the plane is of the form: A*x + B*y + C*z - D = 0
Where A, B, C & D are constants and x, y & z are orthogonalized coordinates.

Plane No.	A	B	C	D	Atom	x	y	z	Distance	Esd
1	0.9050	0.2014	-0.3746	-0.9573	----------Atoms in Plane----------					
					C11	-1.6224	2.7084	0.0628	0.011	0.003
					C12	-1.3601	3.5413	1.1938	-0.008	0.003
					C13	-0.7586	2.7479	2.1950	0.002	0.003
					C14	-0.6620	1.4264	1.7092	0.005	0.003
					C15	-1.2102	1.3915	0.4060	-0.010	0.003
					Chi Squared = 34.					
					----------Other Atoms----------					
					FE	0.3354	2.7098	0.4609	1.634	0.001
					C16	-2.2462	3.2057	-1.2251	0.029	0.003
2	0.8328	0.2700	-0.4833	2.4165	----------Atoms in Plane----------					
					C21	1.0520	3.3802	-1.2788	-0.010	0.003
					C22	1.3852	4.2948	-0.2244	0.005	0.003
					C23	2.1648	3.5908	0.7325	0.002	0.003
					C24	2.3250	2.2632	0.2872	-0.008	0.003
					C25	1.6648	2.1250	-0.9670	0.011	0.003
					Chi Squared = 36.					
					----------Other Atoms----------					
					FE	0.3354	2.7098	0.4609	-1.628	0.001
					C26	0.1807	3.7293	-2.4632	-0.069	0.003
3	0.9012	0.2040	-0.3825	-0.9611	----------Atoms in Plane----------					
					C12	-1.3601	3.5413	1.1938	0.001	0.003
					C13	-0.7586	2.7479	2.1950	-0.002	0.003
					C14	-0.6620	1.4264	1.7092	0.002	0.003
					C15	-1.2102	1.3915	0.4060	-0.001	0.003
					Chi Squared = 1.					
					----------Other Atoms----------					
					FE	0.3354	2.7098	0.4609	1.640	0.001
					C11	-1.6224	2.7084	0.0628		

Table of Least-Squares Planes (continued)

Plane No	A	B	C	D	Atom	x	y	z	Distance	Esd
4	0.9117	0.1959	-0.3611	-0.9758	----------Atoms in Plane----------					
					C11	-1.6224	2.7084	0.0628	0.005	0.003
					C12	-1.3601	3.5413	1.1938	-0.001	0.003
					C15	-1.2102	1.3915	0.4060	-0.002	0.003
					C16	-2.2462	3.2057	-1.2251	-0.002	0.003
					Chi Squared = 4.					
5	0.8372	0.2682	-0.4765	2.4217	----------Atoms in Plane----------					
					C22	1.3852	4.2948	-0.2244	-0.003	0.003
					C23	2.1648	3.5908	0.7325	0.005	0.003
					C24	2.3250	2.2632	0.2872	-0.005	0.003
					C25	1.6648	2.1250	-0.9670	0.003	0.003
					Chi Squared = 7.					
					----------Other Atoms----------					
					FE	0.3354	2.7098	0.4609	-1.634	0.001
					C21	1.0520	3.3802	-1.2788	-0.025	0.003
6	0.8145	0.2791	-0.5086	2.4432	----------Atoms in Plane----------					
					C21	1.0520	3.3802	-1.2788	0.007	0.003
					C22	1.3852	4.2948	-0.2244	-0.002	0.003
					C25	1.6648	2.1250	-0.9670	-0.002	0.003
					C26	0.1807	3.7293	-2.4632	-0.003	0.003
					Chi Squared = 10.					
7	-0.4312	0.6057	-0.6687	3.7789	----------Atoms in Plane----------					
					C16	-2.2462	3.2057	-1.2251	-0.049	0.003
					S1	-2.2646	1.9145	-2.5556	0.066	0.001
					S2	-0.3663	2.1763	-3.3447	-0.066	0.001
					C26	0.1807	3.7293	-2.4632	0.049	0.003
					Chi Squared = 14157.					
					----------Other Atoms----------					
					S3	-1.3250	4.6179	-1.9399	0.887	0.001
					C11	-1.6224	2.7084	0.0628	-1.481	0.003
					C31	-3.7101	3.6286	-1.0456	0.718	0.003

Table of Least-Squares Planes (continued)

Plane No.	A	B	C	D	Atom	x	y	z	Distance	Esd
					C41	0.9123	4.6025	-3.4814	0.944	0.003
8	-0.4159	-0.1799	-0.8914	1.4497	----------Atoms in Plane----------					
					C16	-2.2462	3.2057	-1.2251	0.000	0.003
					S3	-1.3250	4.6179	-1.9399	0.000	0.001
					C26	0.1807	3.7293	-2.4632	0.000	0.003
9	-0.2721	-0.8192	-0.5049	-1.4301	----------Atoms in Plane----------					
					C31	-3.7101	3.6286	-1.0456	-0.005	0.003
					C32	-4.3573	4.4048	-1.9863	0.010	0.004
					C33	-5.6776	4.7643	-1.8236	-0.007	0.004
					C34	-6.3821	4.3220	-0.7364	-0.002	0.004
					C35	-5.7693	3.5304	0.1992	0.007	0.004
					C36	-4.4323	3.1910	0.0518	-0.004	0.003
					Chi Squared =	18.				
10	0.1024	-0.7818	-0.6151	-1.3504	----------Atoms in Plane----------					
					C41	0.9123	4.6025	-3.4814	-0.013	0.003
					C42	2.2613	4.7410	-3.4582	0.002	0.004
					C43	2.9144	5.5410	-4.3770	0.009	0.005
					C44	2.2077	6.2172	-5.3246	-0.009	0.004
					C45	0.8634	6.0776	-5.3829	-0.002	0.004
					C46	0.2058	5.2604	-4.4777	0.013	0.004
					Chi Squared =	42.				

Dihedral Angles Between Planes:

Plane No.	Plane No.	Dihedral Angle	
1	2	8.5	Angle between two C5 rings
3	4	1.4	fold in ring C11-C15
3	5	7.5	Angle between C12-C15 and C22-C25
5	6	2.3	fold in ring C21-C25
7	8	48.2	fold in S3C2 ring

Appendix 3.4 Torsion Angles.

Atom 1	Atom 2	Atom 3	Atom 4	Angle	
C16	S1	S2	C26	-8.6	(0.1)
S2	S1	C16	S3	37.6	(0.1)
S2	S1	C16	C11	-85.0	(0.2)
S2	S1	C16	C31	151.9	(0.2)
S1	S2	C26	S3	-23.1	(0.1)
S1	S2	C26	C21	98.3	(0.2)
S1	S2	C26	C41	-139.0	(0.2)
C26	S3	C16	S1	-52.8	(0.1)
C26	S3	C16	C11	70.0	(0.2)
C26	S3	C16	C31	-165.2	(0.2)
C16	S3	C26	S2	46.5	(0.1)
C16	S3	C26	C21	-74.3	(0.2)
C16	S3	C26	C41	162.1	(0.2)
C15	C11	C12	C13	1.8	(0.3)
C16	C11	C12	C13	-179.0	(0.3)
C12	C11	C15	C14	-1.9	(0.3)
C16	C11	C15	C14	178.9	(0.3)
C12	C11	C16	S1	173.3	(0.2)
C12	C11	C16	S3	54.8	(0.3)
C12	C11	C16	C31	-67.6	(0.4)
C15	C11	C16	S1	-7.7	(0.4)
C15	C11	C16	S3	-126.2	(0.3)
C15	C11	C16	C31	111.4	(0.3)
C11	C12	C13	C14	-0.9	(0.4)
C12	C13	C14	C15	-0.3	(0.4)
C13	C14	C15	C11	1.4	(0.4)
S1	C16	C31	C32	-72.9	(0.3)
S1	C16	C31	C36	104.6	(0.3)
S3	C16	C31	C32	39.2	(0.3)
S3	C16	C31	C36	-143.3	(0.3)
C11	C16	C31	C32	163.8	(0.3)
C11	C16	C31	C36	-18.7	(0.4)
C25	C21	C22	C23	-1.4	(0.3)
C26	C21	C22	C23	177.4	(0.3)
C22	C21	C25	C24	1.9	(0.3)
C26	C21	C25	C24	-176.7	(0.3)
C22	C21	C26	S2	-165.2	(0.2)
C22	C21	C26	S3	-46.1	(0.3)
C22	C21	C26	C41	75.0	(0.3)
C25	C21	C26	S2	13.3	(0.4)
C25	C21	C26	S3	132.4	(0.3)
C25	C21	C26	C41	-106.5	(0.3)
C21	C22	C23	C24	0.3	(0.3)
C22	C23	C24	C25	0.9	(0.4)
C23	C24	C25	C21	-1.8	(0.4)
S2	C26	C41	C42	-106.2	(0.3)
S2	C26	C41	C46	72.6	(0.3)
S3	C26	C41	C42	138.3	(0.3)
S3	C26	C41	C46	-42.9	(0.3)
C21	C26	C41	C42	15.2	(0.4)
C21	C26	C41	C46	-166.0	(0.3)
C16	C31	C32	C33	179.1	(0.3)
C36	C31	C32	C33	1.6	(0.5)

Torsion Angles (continued)

Atom 1	Atom 2	Atom 3	Atom 4	Angle
C16	C31	C36	C35	-177.8 (0.3)
C32	C31	C36	C35	-0.2 (0.5)
C31	C32	C33	C34	-1.8 (0.6)
C32	C33	C34	C35	0.6 (0.6)
C33	C34	C35	C36	0.7 (0.6)
C34	C35	C36	C31	-1.0 (0.6)
C26	C41	C42	C43	-179.5 (0.4)
C46	C41	C42	C43	1.7 (0.6)
C26	C41	C46	C45	178.5 (0.3)
C42	C41	C46	C45	-2.7 (0.5)
C41	C42	C43	C44	0.4 (0.7)
C42	C43	C44	C45	-1.6 (0.7)
C43	C44	C45	C46	0.5 (0.6)
C44	C45	C46	C41	1.6 (0.6)
C11	Cp1	Cp2	C21	-2.6 (0.2)
C12	Cp1	Cp2	C22	-2.6 (0.2)
C13	Cp1	Cp2	C23	-2.5 (0.2)
C14	Cp1	Cp2	C24	-2.8 (0.2)
C15	Cp1	Cp2	C25	-2.6 (0.2)

Cp1 and Cp2 are the centroids of C11-C15 and C21-C25 respectively.

Appendix 3.5 Bond Angles.

C11	Fe	C12	41.6(1)
C11	Fe	C13	69.3(1)
C11	Fe	C14	69.1(1)
C11	Fe	C15	41.3(1)
C11	Fe	C21	100.3(1)
C11	Fe	C22	116.2(1)
C11	Fe	C23	154.4(1)
C11	Fe	C24	159.3(1)
C11	Fe	C25	120.0(1)
C12	Fe	C13	40.5(1)
C12	Fe	C14	68.2(1)
C12	Fe	C15	68.9(1)
C12	Fe	C21	118.5(1)
C12	Fe	C22	103.6(1)
C12	Fe	C23	121.5(1)
C12	Fe	C24	159.1(1)
C12	Fe	C25	156.6(1)
C13	Fe	C14	40.3(1)
C13	Fe	C15	68.2(1)
C13	Fe	C21	157.2(1)
C13	Fe	C22	123.3(1)
C13	Fe	C23	110.9(1)
C13	Fe	C24	126.5(1)
C13	Fe	C25	161.2(1)
C14	Fe	C15	40.5(1)
C14	Fe	C21	156.2(1)
C14	Fe	C22	161.9(1)
C14	Fe	C23	128.5(1)

C14	Fe	C24	112.8(1)
C14	Fe	C25	124.4(1)
C15	Fe	C21	117.8(1)
C15	Fe	C22	153.5(1)
C15	Fe	C23	164.1(1)
C15	Fe	C24	126.6(1)
C15	Fe	C25	106.9(1)
C21	Fe	C22	41.8(1)
C21	Fe	C23	69.6(1)
C21	Fe	C24	69.5(1)
C21	Fe	C25	41.5(1)
C22	Fe	C23	40.9(1)
C22	Fe	C24	68.7(1)
C22	Fe	C25	69.4(1)
C23	Fe	C24	40.3(1)
C23	Fe	C25	68.5(1)
C24	Fe	C25	40.8(1)
S2	S1	C16	100.1(1)
S1	S2	C26	101.2(1)
C16	S3	C26	98.7(1)
Fe	C11	C12	70.2(2)
Fe	C11	C15	70.6(2)
Fe	C11	C16	124.9(2)
C12	C11	C15	107.2(2)
C12	C11	C16	123.9(3)
C15	C11	C16	128.9(3)
Fe	C12	C11	68.2(2)
Fe	C12	C13	70.7(2)
C11	C12	C13	108.2(3)

Fe	C24	C25	69.2(2)
C23	C24	C25	108.5(3)
Fe	C25	C21	67.8(2)
Fe	C25	C24	70.0(2)
C21	C25	C24	107.8(3)
S2	C26	S3	107.4(1)
S2	C26	C21	110.3(2)
S2	C26	C41	107.5(2)
S3	C26	C21	111.3(2)
S3	C26	C41	107.9(2)
C21	C26	C41	112.2(2)
C16	C31	C32	121.5(3)
C16	C31	C36	120.2(3)
C32	C31	C36	118.2(3)
C31	C32	C33	121.0(3)
C32	C33	C34	120.2(3)
C33	C34	C35	119.9(3)
C34	C35	C36	120.0(4)
C31	C36	C35	120.6(3)
C26	C41	C42	121.5(3)
C26	C41	C46	120.4(3)
C42	C41	C46	118.1(3)
C41	C42	C43	121.2(3)
C42	C43	C44	120.3(4)
C43	C44	C45	119.6(4)
C44	C45	C46	120.3(4)
C41	C46	C45	120.5(3)

Fe	C13	C12	68.8(2)
Fe	C13	C14	69.8(2)
C12	C13	C14	108.1(3)
Fe	C14	C13	69.9(2)
Fe	C14	C15	69.1(2)
C13	C14	C15	108.3(3)
Fe	C15	C11	68.1(2)
Fe	C15	C14	70.4(2)
C11	C15	C14	108.2(3)
S1	C16	S3	105.2(1)
S1	C16	C11	112.7(2)
S1	C16	C31	105.5(2)
S3	C16	C11	112.2(2)
S3	C16	C31	108.2(2)
C11	C16	C31	112.6(2)
Fe	C21	C22	70.0(2)
Fe	C21	C25	70.7(2)
Fe	C21	C26	123.6(2)
C22	C21	C25	107.4(2)
C22	C21	C26	124.2(3)
C25	C21	C26	128.3(2)
Fe	C22	C21	68.2(2)
Fe	C22	C23	70.6(2)
C21	C22	C23	107.8(3)
Fe	C23	C22	68.5(2)
Fe	C23	C24	69.8(2)
C22	C23	C24	108.4(3)
Fe	C24	C23	69.9(2)

Appendix 3.6 Observed and Calculated Structure Factors.

10Fo, 10Fc, 10sig(Fo) for C24,H18,Fe,S3 File: 90-25

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
0	0	2	1270	1246	6	3	0	-5	400	374	2
0	0	4	481	446	2	3	0	-3	905	774	3
0	0	6	637	622	3	3	0	-1	533	491	3
0	0	8	418	413	2	3	0	1	276	263	1
0	0	10	340	328	2	3	0	3	1093	1088	3
0	0	12	171	161	3	3	0	5	222	202	2
0	0	14	86	84	6	3	0	7	92	89	3
0	0	16	103	103	6	3	0	9	554	566	3
0	0	18	205	191	5	3	0	11	438	435	2
1	0	-17	119	118	6	3	0	13	152	146	4
1	0	-15	566	572	3	3	0	15	343	328	3
1	0	-11	463	479	2	3	0	17	95	106	8
1	0	-9	312	310	2	4	0	-16	394	401	3
1	0	-7	208	210	2	4	0	-14	402	404	3
1	0	-5	140	153	2	4	0	-12	112	110	4
1	0	-3	869	797	2	4	0	-10	412	424	2
1	0	-1	2740	2374	7	4	0	-8	393	388	2
1	0	1	901	879	4	4	0	-6	1249	1114	4
1	0	3	165	154	1	4	0	-4	427	392	2
1	0	5	895	889	3	4	0	-2	932	829	3
1	0	7	422	406	2	4	0	0	104	104	2
1	0	9	54	39	5	4	0	2	389	379	2
1	0	11	372	382	2	4	0	4	197	187	2
1	0	13	66	50	7	4	0	6	201	202	2
1	0	15	364	362	3	4	0	8	544	562	3
1	0	17	144	141	5	4	0	10	507	505	2
2	0	-18	118	136	6	4	0	14	567	540	3
2	0	-16	392	397	3	4	0	16	84	95	8
2	0	-14	245	242	3	5	0	-17	199	197	4
2	0	-12	486	483	2	5	0	-15	191	195	4
2	0	-10	307	307	2	5	0	-13	224	226	3
2	0	-8	1052	1004	4	5	0	-11	310	307	2
2	0	-6	130	117	2	5	0	-9	459	440	2
2	0	-2	1009	860	2	5	0	-7	1277	1114	4
2	0	0	1854	1806	9	5	0	-5	1135	997	4
2	0	2	151	146	1	5	0	-3	557	467	3
2	0	4	830	830	3	5	0	-1	432	431	2
2	0	6	69	75	3	5	0	1	248	240	2
2	0	8	159	153	2	5	0	3	308	308	2
2	0	10	717	739	4	5	0	5	427	450	2
2	0	12	291	284	3	5	0	7	411	436	2
2	0	14	67	72	8	5	0	9	487	498	3
2	0	16	153	158	4	5	0	11	109	111	5
2	0	18	148	139	6	5	0	13	526	504	3
3	0	-17	261	256	4	5	0	15	225	211	4
3	0	-15	376	371	3	6	0	-18	74	85	11
3	0	-13	432	427	2	6	0	-14	388	393	3
3	0	-11	240	237	2	6	0	-12	172	165	4
3	0	-9	314	320	2	6	0	-10	77	61	6
3	0	-7	394	361	2	6	0	-8	834	739	4

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
6	0	-6	1583	1442	4	9	0	7	402	411	3
6	0	-4	474	422	2	9	0	9	343	356	3
6	0	-2	757	744	4	10	0	-14	88	69	8
6	0	0	243	205	2	10	0	-12	437	408	3
6	0	4	656	660	3	10	0	-10	115	92	5
6	0	6	791	821	4	10	0	-8	453	441	3
6	0	8	302	312	2	10	0	-6	67	54	7
6	0	10	165	170	3	10	0	-4	102	95	5
6	0	12	437	422	3	10	0	-2	353	378	2
6	0	14	254	242	4	10	0	0	121	129	4
6	0	16	160	144	5	10	0	2	293	308	3
7	0	-15	427	427	3	10	0	4	205	206	3
7	0	-11	64	60	8	10	0	8	374	392	3
7	0	-9	142	127	4	10	0	10	105	103	7
7	0	-7	947	888	5	10	0	12	95	72	8
7	0	-5	576	549	3	11	0	-13	231	207	4
7	0	-3	656	622	3	11	0	-11	181	177	4
7	0	-1	400	373	2	11	0	-3	371	403	3
7	0	1	74	54	5	11	0	-1	443	482	3
7	0	3	464	470	2	11	0	1	290	304	3
7	0	5	798	830	4	11	0	3	75	77	7
7	0	7	237	242	3	11	0	7	428	429	3
7	0	9	178	187	4	12	0	-12	261	263	4
7	0	11	346	343	3	12	0	-4	131	119	5
7	0	13	264	264	4	12	0	-2	444	463	3
7	0	15	96	67	7	12	0	0	338	365	3
8	0	-16	440	444	3	12	0	2	118	129	5
8	0	-14	75	50	9	12	0	4	219	230	4
8	0	-12	280	259	3	12	0	6	306	318	4
8	0	-10	468	432	3	13	0	-5	202	213	4
8	0	-8	1036	992	5	13	0	-3	339	366	3
8	0	-6	570	556	3	13	0	-1	254	272	4
8	0	-4	194	196	3	13	0	3	284	298	4
8	0	-2	241	229	2	14	0	-8	165	170	5
8	0	0	211	193	2	14	0	-4	249	264	4
8	0	4	867	884	4	14	0	-2	225	245	4
8	0	6	298	299	3	14	0	2	167	172	5
8	0	8	170	154	4	15	0	-3	122	128	7
8	0	10	296	307	3	0	1	1	50	44	1
8	0	12	156	158	5	0	1	2	932	939	2
8	0	14	91	91	8	0	1	3	361	361	2
9	0	-11	702	644	3	0	1	4	1199	1207	3
9	0	-9	269	261	3	0	1	5	598	590	3
9	0	-7	253	241	3	0	1	6	708	695	3
9	0	-5	153	152	3	0	1	7	192	206	2
9	0	-3	268	262	3	0	1	8	940	923	4
9	0	-1	55	48	7	0	1	9	89	84	4
9	0	1	60	59	7	0	1	10	530	521	3
9	0	3	385	412	2	0	1	11	172	179	3
9	0	5	177	181	4	0	1	12	602	595	3

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
0	1	13	110	99	5	2	1	-4	278	259	1
0	1	15	166	160	4	2	1	-3	511	458	3
0	1	16	139	131	5	2	1	-2	433	375	2
0	1	18	273	262	4	2	1	-1	399	341	2
1	1	-18	95	87	8	2	1	0	43	40	2
1	1	-16	246	242	3	2	1	1	378	358	2
1	1	-15	110	86	5	2	1	2	1603	1609	9
1	1	-14	151	149	4	2	1	3	547	557	2
1	1	-13	303	299	3	2	1	4	804	812	3
1	1	-12	94	97	5	2	1	5	448	463	2
1	1	-11	575	571	3	2	1	6	358	386	2
1	1	-10	184	188	3	2	1	8	455	472	2
1	1	-9	589	582	3	2	1	9	230	233	2
1	1	-8	95	92	3	2	1	10	83	47	5
1	1	-7	960	954	4	2	1	11	105	104	4
1	1	-6	447	434	2	2	1	12	346	347	3
1	1	-5	341	338	2	2	1	13	201	201	3
1	1	-4	109	120	2	2	1	14	79	73	7
1	1	-3	336	326	2	2	1	18	126	111	6
1	1	-2	225	207	1	3	1	-18	104	97	7
1	1	-1	39	40	2	3	1	-17	136	144	6
1	1	0	178	179	1	3	1	-15	172	178	4
1	1	1	855	855	2	3	1	-14	196	194	3
1	1	2	1037	1067	5	3	1	-13	337	335	3
1	1	3	2171	2223	10	3	1	-12	289	286	2
1	1	4	432	448	2	3	1	-9	1090	1058	4
1	1	5	243	242	1	3	1	-7	528	487	3
1	1	6	451	450	2	3	1	-6	606	565	3
1	1	7	614	616	3	3	1	-5	149	140	2
1	1	8	64	58	4	3	1	-4	103	89	2
1	1	9	448	437	2	3	1	-3	343	292	2
1	1	10	172	171	3	3	1	-2	311	258	2
1	1	11	213	214	3	3	1	-1	254	241	1
1	1	12	144	149	3	3	1	0	523	519	3
1	1	13	127	117	4	3	1	1	674	667	3
1	1	14	276	279	3	3	1	2	404	399	2
1	1	15	76	70	8	3	1	3	747	748	3
1	1	17	144	146	6	3	1	4	325	325	2
2	1	-17	140	144	5	3	1	5	42	50	5
2	1	-16	138	135	5	3	1	6	566	551	3
2	1	-15	148	158	4	3	1	7	143	177	2
2	1	-14	217	217	3	3	1	8	602	602	3
2	1	-13	104	109	5	3	1	9	65	39	6
2	1	-12	448	448	2	3	1	10	100	97	4
2	1	-11	262	272	2	3	1	11	507	496	3
2	1	-10	71	70	5	3	1	12	197	203	3
2	1	-9	110	111	3	3	1	13	234	234	3
2	1	-8	915	921	4	3	1	15	182	173	4
2	1	-7	68	71	4	3	1	17	88	86	8
2	1	-6	369	346	2	4	1	-18	190	191	5

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
4	1	-16	256	264	3	5	1	10	89	72	5
4	1	-15	190	192	4	5	1	12	93	106	6
4	1	-14	189	197	4	5	1	13	349	337	3
4	1	-13	160	162	4	6	1	-18	244	244	4
4	1	-11	105	100	4	6	1	-14	331	334	3
4	1	-10	653	643	3	6	1	-12	476	474	3
4	1	-9	276	270	2	6	1	-11	110	111	5
4	1	-8	274	282	2	6	1	-10	343	343	2
4	1	-7	68	58	4	6	1	-9	93	87	4
4	1	-6	227	220	2	6	1	-8	198	186	3
4	1	-5	112	107	2	6	1	-7	78	68	5
4	1	-4	764	654	3	6	1	-6	372	340	2
4	1	-3	523	439	3	6	1	-5	183	172	2
4	1	-2	212	183	1	6	1	-4	684	593	3
4	1	-1	209	208	1	6	1	-3	103	91	3
4	1	0	64	51	3	6	1	-2	719	700	3
4	1	1	486	493	3	6	1	0	260	266	2
4	1	2	951	939	3	6	1	1	250	264	2
4	1	3	385	376	2	6	1	2	884	878	4
4	1	4	478	475	2	6	1	3	200	202	2
4	1	5	129	121	2	6	1	4	351	327	2
4	1	6	565	561	3	6	1	5	49	42	7
4	1	7	471	471	2	6	1	6	432	430	2
4	1	8	254	254	2	6	1	8	214	211	3
4	1	9	155	157	3	6	1	9	142	148	4
4	1	10	613	625	3	6	1	11	84	90	6
4	1	12	80	84	6	6	1	12	280	268	3
4	1	14	275	255	3	7	1	-15	272	286	3
5	1	-17	201	201	4	7	1	-13	434	422	3
5	1	-14	112	114	5	7	1	-11	145	132	4
5	1	-13	141	137	4	7	1	-10	451	408	2
5	1	-11	438	430	2	7	1	-9	290	265	2
5	1	-10	136	136	3	7	1	-8	145	128	3
5	1	-9	416	403	2	7	1	-7	173	160	3
5	1	-8	224	201	2	7	1	-6	157	144	3
5	1	-7	328	315	2	7	1	-5	410	383	2
5	1	-6	273	247	2	7	1	-4	189	173	2
5	1	-5	639	573	3	7	1	-3	757	733	3
5	1	-4	466	410	2	7	1	-2	362	349	2
5	1	-3	876	726	3	7	1	-1	358	360	2
5	1	-2	1000	880	3	7	1	0	473	474	2
5	1	-1	704	665	3	7	1	1	835	865	4
5	1	1	831	824	3	7	1	2	170	164	3
5	1	2	348	361	2	7	1	3	136	127	3
5	1	3	997	963	4	7	1	5	435	432	2
5	1	4	129	129	3	7	1	6	110	117	4
5	1	5	520	506	3	7	1	8	213	215	3
5	1	6	79	94	4	7	1	9	247	240	3
5	1	7	245	243	2	7	1	11	237	232	3
5	1	9	392	394	2	7	1	12	71	64	8

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
7	1	13	95	93	7	10	1	-4	635	640	3
7	1	15	139	134	6	10	1	-3	124	118	4
8	1	-16	201	210	5	10	1	-2	211	210	3
8	1	-14	485	476	3	10	1	-1	68	70	7
8	1	-13	80	85	8	10	1	0	110	110	4
8	1	-12	123	108	5	10	1	1	127	132	4
8	1	-10	84	79	6	10	1	2	185	182	3
8	1	-9	224	221	3	10	1	4	494	512	3
8	1	-8	190	174	3	10	1	6	105	100	6
8	1	-7	154	139	3	10	1	7	91	88	7
8	1	-6	262	250	2	10	1	12	218	201	5
8	1	-5	277	232	2	11	1	-14	107	107	7
8	1	-4	530	486	3	11	1	-11	94	98	7
8	1	-2	399	403	2	11	1	-9	212	204	4
8	1	-1	366	382	2	11	1	-8	302	305	3
8	1	0	534	560	3	11	1	-7	65	43	8
8	1	1	166	166	3	11	1	-5	491	479	2
8	1	3	249	253	3	11	1	-3	159	154	4
8	1	4	524	524	3	11	1	-2	107	124	5
8	1	5	329	340	2	11	1	0	89	93	6
8	1	6	64	66	7	11	1	1	259	261	3
8	1	8	469	482	3	11	1	3	212	215	3
8	1	9	158	171	4	11	1	4	92	106	6
8	1	10	123	126	5	11	1	7	135	134	5
8	1	11	106	107	6	11	1	8	110	106	6
8	1	12	81	70	7	12	1	-13	99	97	8
9	1	-15	352	334	3	12	1	-10	123	115	6
9	1	-13	108	88	6	12	1	-9	150	155	5
9	1	-12	199	186	4	12	1	-8	178	171	4
9	1	-11	282	272	3	12	1	-6	413	402	3
9	1	-8	70	74	7	12	1	-5	74	80	9
9	1	-6	443	427	2	12	1	-4	228	231	3
9	1	-5	205	190	3	12	1	-3	99	109	6
9	1	-3	492	501	2	12	1	-2	128	139	5
9	1	-2	252	257	2	12	1	0	333	356	3
9	1	-1	206	214	3	12	1	3	81	91	8
9	1	1	72	68	6	12	1	6	92	63	8
9	1	3	83	79	6	13	1	-10	130	135	6
9	1	5	164	166	4	13	1	-9	300	298	4
9	1	6	129	146	5	13	1	-8	80	71	9
9	1	7	557	573	3	13	1	-7	305	289	3
9	1	10	66	64	9	13	1	-6	92	98	7
9	1	11	145	138	5	13	1	-5	188	192	4
9	1	13	136	138	6	13	1	-1	248	254	4
10	1	-13	153	153	5	13	1	1	175	182	4
10	1	-12	80	74	9	13	1	2	131	130	6
10	1	-11	211	210	4	13	1	6	92	91	8
10	1	-8	320	300	3	14	1	-8	126	120	6
10	1	-7	199	184	3	14	1	-7	74	84	10
10	1	-6	86	87	6	14	1	-2	239	258	4

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
14	1	1	105	103	8	1	2	16	149	160	5
14	1	2	164	174	6	1	2	17	126	146	6
0	2	0	2385	2491	8	1	2	18	137	117	6
0	2	1	769	801	2	2	2	-18	121	128	6
0	2	2	1749	1817	8	2	2	-16	492	492	3
0	2	3	806	824	2	2	2	-15	403	410	3
0	2	4	1179	1193	3	2	2	-13	379	389	3
0	2	5	70	77	3	2	2	-12	82	86	6
0	2	6	140	150	2	2	2	-11	67	66	6
0	2	7	89	89	3	2	2	-10	130	131	3
0	2	8	207	211	2	2	2	-9	305	288	2
0	2	9	699	683	4	2	2	-8	312	318	2
0	2	10	148	154	3	2	2	-7	254	252	2
0	2	11	370	367	2	2	2	-6	818	828	3
0	2	12	195	199	3	2	2	-5	383	378	2
0	2	13	127	118	4	2	2	-4	696	696	3
0	2	14	195	189	3	2	2	-3	392	400	2
0	2	16	298	291	3	2	2	-2	109	118	2
0	2	17	246	238	4	2	2	-1	877	826	2
0	2	18	73	76	10	2	2	0	607	580	2
1	2	-18	193	191	5	2	2	1	518	518	2
1	2	-17	244	255	4	2	2	2	652	644	3
1	2	-15	322	322	3	2	2	3	304	300	2
1	2	-14	175	175	4	2	2	4	739	758	3
1	2	-12	417	418	2	2	2	5	928	908	3
1	2	-11	274	267	2	2	2	6	439	456	2
1	2	-10	386	379	2	2	2	7	544	577	3
1	2	-8	194	187	2	2	2	9	90	93	4
1	2	-7	171	180	2	2	2	10	504	498	2
1	2	-6	45	58	4	2	2	11	89	92	5
1	2	-5	638	657	3	2	2	12	318	311	3
1	2	-4	819	845	3	2	2	14	260	266	3
1	2	-3	268	271	1	2	2	16	132	129	5
1	2	-2	360	369	2	2	2	17	134	119	5
1	2	-1	2242	2266	9	2	2	18	95	92	8
1	2	0	275	272	1	3	2	-17	289	287	4
1	2	1	827	832	2	3	2	-16	268	276	3
1	2	2	666	675	2	3	2	-15	191	198	4
1	2	3	152	164	1	3	2	-14	177	184	4
1	2	4	562	558	3	3	2	-13	199	198	3
1	2	5	71	78	3	3	2	-12	98	92	5
1	2	6	452	430	2	3	2	-11	124	127	4
1	2	7	344	355	2	3	2	-10	292	300	2
1	2	8	694	695	4	3	2	-9	503	521	2
1	2	9	135	124	3	3	2	-8	367	368	2
1	2	10	270	267	2	3	2	-7	253	256	2
1	2	11	318	309	2	3	2	-6	183	181	2
1	2	13	224	209	3	3	2	-5	527	537	3
1	2	14	304	300	3	3	2	-4	1130	1050	3
1	2	15	277	268	3	3	2	-3	523	446	2

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
3	2	-2	323	297	2	5	2	-15	97	101	6
3	2	-1	304	289	2	5	2	-14	260	259	3
3	2	0	126	130	2	5	2	-13	79	81	6
3	2	1	570	560	3	5	2	-12	217	221	3
3	2	2	36	39	4	5	2	-11	224	223	3
3	2	3	370	372	2	5	2	-9	279	272	2
3	2	4	704	713	3	5	2	-8	109	109	3
3	2	5	232	248	2	5	2	-7	771	720	4
3	2	6	783	796	4	5	2	-6	81	45	4
3	2	7	126	128	3	5	2	-5	876	778	4
3	2	8	157	152	3	5	2	-4	580	481	3
3	2	9	560	549	3	5	2	-3	154	161	2
3	2	10	391	392	2	5	2	-2	459	415	2
3	2	11	496	485	2	5	2	-1	750	725	3
3	2	12	78	71	6	5	2	0	852	847	3
3	2	13	171	172	4	5	2	1	203	194	2
3	2	15	155	149	4	5	2	2	213	201	2
4	2	-18	108	105	7	5	2	3	64	74	5
4	2	-17	151	160	5	5	2	4	528	538	3
4	2	-16	266	263	3	5	2	5	147	167	3
4	2	-14	234	245	3	5	2	6	219	213	2
4	2	-13	146	151	4	5	2	7	157	163	3
4	2	-12	98	109	5	5	2	8	309	310	2
4	2	-11	256	263	2	5	2	9	542	547	3
4	2	-10	274	272	2	5	2	10	203	204	3
4	2	-9	292	297	2	5	2	11	248	247	3
4	2	-8	73	88	5	5	2	12	69	58	7
4	2	-7	81	86	4	5	2	13	360	350	3
4	2	-6	1144	1076	4	5	2	16	124	105	6
4	2	-5	558	508	3	6	2	-17	145	141	6
4	2	-3	267	245	1	6	2	-12	194	194	3
4	2	-2	177	181	2	6	2	-11	173	180	3
4	2	-1	210	203	1	6	2	-10	278	260	2
4	2	0	464	462	2	6	2	-8	397	369	2
4	2	1	190	197	2	6	2	-7	244	223	2
4	2	2	172	171	2	6	2	-6	935	846	4
4	2	3	143	142	2	6	2	-5	425	378	2
4	2	5	363	379	2	6	2	-4	776	657	4
4	2	6	199	196	2	6	2	-3	1028	928	4
4	2	7	149	145	3	6	2	-2	1225	1142	4
4	2	8	251	248	2	6	2	-1	653	640	3
4	2	9	617	622	3	6	2	0	130	137	2
4	2	10	649	644	3	6	2	1	583	578	3
4	2	11	138	140	4	6	2	3	471	474	2
4	2	12	235	222	3	6	2	4	254	259	2
4	2	14	278	272	3	6	2	5	129	118	3
4	2	16	110	97	7	6	2	6	302	299	2
4	2	17	105	105	7	6	2	7	66	63	6
5	2	-17	141	144	5	6	2	8	429	422	2
5	2	-16	98	113	7	6	2	10	297	304	3

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
6	2	12	227	219	3	8	2	13	153	161	5
6	2	14	115	120	6	9	2	-13	231	225	4
6	2	15	91	89	8	9	2	-12	154	154	5
7	2	-15	160	156	5	9	2	-11	206	192	4
7	2	-14	155	154	5	9	2	-10	235	229	3
7	2	-12	279	277	3	9	2	-8	70	57	7
7	2	-11	190	183	3	9	2	-7	245	223	3
7	2	-10	96	84	5	9	2	-6	67	58	7
7	2	-9	121	125	4	9	2	-5	94	101	5
7	2	-8	303	288	2	9	2	-4	216	205	3
7	2	-7	655	619	3	9	2	-2	156	149	3
7	2	-6	375	315	2	9	2	-1	130	134	4
7	2	-5	580	499	3	9	2	0	332	353	2
7	2	-4	894	802	4	9	2	1	79	80	5
7	2	-3	572	530	3	9	2	2	103	104	4
7	2	-2	278	291	2	9	2	3	354	370	2
7	2	-1	113	121	3	9	2	5	236	245	3
7	2	1	293	288	2	9	2	6	182	185	4
7	2	2	591	600	3	9	2	7	241	237	3
7	2	3	314	317	2	9	2	9	190	194	4
7	2	4	81	98	5	9	2	12	137	140	6
7	2	5	585	592	3	9	2	13	104	90	8
7	2	6	269	268	3	10	2	-14	158	138	5
7	2	7	119	119	4	10	2	-12	442	421	3
7	2	9	304	313	3	10	2	-11	303	296	3
7	2	10	218	216	3	10	2	-9	202	186	4
7	2	11	164	163	4	10	2	-8	184	177	4
7	2	13	173	177	5	10	2	-7	179	178	4
7	2	14	102	72	7	10	2	-6	155	142	4
8	2	-16	119	114	7	10	2	-5	129	125	4
8	2	-12	431	404	3	10	2	-4	83	92	6
8	2	-11	225	224	3	10	2	-3	108	92	5
8	2	-10	491	462	2	10	2	-1	238	243	3
8	2	-9	489	467	2	10	2	0	95	96	5
8	2	-8	174	162	3	10	2	2	221	226	3
8	2	-7	526	488	3	10	2	4	246	236	3
8	2	-6	686	619	4	10	2	5	147	143	4
8	2	-5	370	336	2	10	2	6	290	297	3
8	2	-4	509	461	3	10	2	8	203	206	4
8	2	-3	90	84	4	10	2	11	130	129	6
8	2	-2	62	55	6	11	2	-13	176	166	5
8	2	-1	109	109	4	11	2	-12	154	150	5
8	2	0	223	223	3	11	2	-10	305	297	3
8	2	1	517	529	3	11	2	-9	241	233	3
8	2	3	233	248	3	11	2	-8	75	82	8
8	2	4	206	209	3	11	2	-7	145	119	4
8	2	8	297	295	3	11	2	-6	285	271	3
8	2	9	140	135	5	11	2	-5	111	100	5
8	2	10	64	69	9	11	2	-4	154	145	4
8	2	12	97	87	7	11	2	-3	61	51	9

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
11	2	-2	98	105	6	0	3	6	141	137	2
11	2	-1	190	197	4	0	3	7	111	123	3
11	2	0	135	145	5	0	3	8	980	985	4
11	2	1	179	190	4	0	3	9	50	47	7
11	2	2	162	173	4	0	3	10	817	811	4
11	2	3	356	372	3	0	3	13	66	45	7
11	2	5	221	215	4	0	3	15	366	357	3
11	2	6	112	115	6	0	3	16	97	98	7
11	2	7	208	200	4	0	3	17	77	82	9
11	2	10	212	201	5	1	3	-17	117	120	6
12	2	-12	132	148	6	1	3	-16	195	190	4
12	2	-11	304	308	4	1	3	-14	226	230	3
12	2	-10	132	132	6	1	3	-13	329	337	3
12	2	-9	100	102	7	1	3	-11	518	530	3
12	2	-8	145	146	5	1	3	-10	102	98	4
12	2	-7	213	207	4	1	3	-9	591	609	3
12	2	-6	122	118	5	1	3	-8	232	230	2
12	2	-5	227	225	3	1	3	-7	314	309	2
12	2	-4	66	71	9	1	3	-6	475	487	2
12	2	-3	91	99	7	1	3	-5	562	585	3
12	2	-2	283	303	3	1	3	-4	181	190	1
12	2	0	147	158	5	1	3	-3	205	200	1
12	2	1	172	187	4	1	3	-2	358	374	2
12	2	2	300	318	3	1	3	-1	155	149	1
12	2	4	249	258	4	1	3	0	1290	1309	7
12	2	5	73	80	9	1	3	1	315	314	2
12	2	6	153	166	5	1	3	2	44	36	3
12	2	7	81	79	9	1	3	3	246	236	1
12	2	8	130	130	6	1	3	4	1047	1076	3
13	2	-11	106	100	7	1	3	5	258	262	2
13	2	-10	82	89	9	1	3	6	248	271	2
13	2	-9	143	141	6	1	3	7	826	843	4
13	2	-8	154	139	5	1	3	8	71	89	5
13	2	-6	101	85	7	1	3	9	368	377	2
13	2	-3	116	115	6	1	3	12	421	407	3
13	2	-1	141	144	5	1	3	13	155	166	4
13	2	1	185	191	4	1	3	14	224	217	3
13	2	3	202	214	5	2	3	-17	167	166	5
13	2	4	110	123	7	2	3	-15	116	107	5
14	2	-7	129	119	6	2	3	-14	159	166	4
14	2	-5	112	118	7	2	3	-13	187	193	4
14	2	-2	118	126	6	2	3	-12	468	482	2
14	2	1	123	125	7	2	3	-11	397	420	2
14	2	2	159	176	6	2	3	-10	450	451	2
14	2	3	99	114	8	2	3	-9	187	203	2
0	3	1	505	496	2	2	3	-8	438	445	2
0	3	2	974	997	3	2	3	-7	376	358	2
0	3	3	209	208	1	2	3	-6	138	134	2
0	3	4	389	425	2	2	3	-5	198	208	2
0	3	5	721	734	3	2	3	-4	844	867	3

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
2	3	-3	40	29	4	4	3	-11	177	194	3
2	3	-2	322	317	2	4	3	-10	66	64	6
2	3	-1	131	136	2	4	3	-8	458	471	2
2	3	0	710	692	3	4	3	-6	470	473	2
2	3	1	546	525	2	4	3	-5	472	474	2
2	3	2	1185	1144	3	4	3	-4	76	65	3
2	3	3	836	858	3	4	3	-3	451	425	2
2	3	4	222	239	2	4	3	-2	890	821	3
2	3	5	502	489	3	4	3	-1	701	662	3
2	3	6	668	692	3	4	3	0	447	424	2
2	3	7	252	258	2	4	3	2	392	383	2
2	3	8	430	463	2	4	3	4	631	640	3
2	3	9	339	341	2	4	3	5	447	426	2
2	3	10	145	154	4	4	3	6	663	658	3
2	3	11	344	342	2	4	3	7	468	480	2
2	3	12	190	191	3	4	3	8	87	105	5
2	3	13	223	222	3	4	3	9	174	182	3
2	3	15	125	129	5	4	3	10	195	203	3
3	3	-18	142	145	6	4	3	11	66	62	7
3	3	-15	133	123	5	4	3	15	104	95	6
3	3	-14	189	189	4	4	3	16	236	225	4
3	3	-13	536	550	3	5	3	-15	254	260	3
3	3	-12	489	505	2	5	3	-13	236	234	3
3	3	-11	263	270	3	5	3	-11	78	84	6
3	3	-10	274	296	2	5	3	-10	280	285	2
3	3	-9	165	170	3	5	3	-9	209	217	3
3	3	-7	398	399	2	5	3	-7	396	384	2
3	3	-6	201	207	2	5	3	-6	747	712	3
3	3	-5	954	968	3	5	3	-5	103	86	3
3	3	-4	634	607	3	5	3	-3	1125	1014	3
3	3	-3	271	268	1	5	3	-2	803	750	3
3	3	-2	165	155	2	5	3	-1	295	283	2
3	3	-1	652	622	3	5	3	0	254	237	2
3	3	0	203	199	1	5	3	1	566	557	3
3	3	1	83	80	2	5	3	2	184	189	2
3	3	2	501	506	2	5	3	3	744	750	3
3	3	3	133	146	2	5	3	4	222	208	2
3	3	4	462	454	2	5	3	5	657	652	3
3	3	5	190	226	2	5	3	7	299	292	2
3	3	6	372	381	2	5	3	8	125	119	4
3	3	7	536	572	3	5	3	9	154	158	4
3	3	8	603	610	3	5	3	11	127	147	5
3	3	10	365	375	2	5	3	12	115	125	5
3	3	12	125	126	4	5	3	14	143	127	5
3	3	14	176	180	4	5	3	15	205	198	4
3	3	17	276	256	4	6	3	-17	106	116	7
4	3	-15	128	139	5	6	3	-16	169	177	5
4	3	-14	410	419	3	6	3	-15	71	81	9
4	3	-13	211	229	3	6	3	-14	376	386	3
4	3	-12	298	312	3	6	3	-12	114	130	5

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
6	3	-11	189	186	3	7	3	13	219	216	4
6	3	-10	307	316	2	8	3	-16	192	185	5
6	3	-9	122	123	4	8	3	-14	88	86	7
6	3	-8	282	271	2	8	3	-13	125	121	5
6	3	-7	335	320	2	8	3	-12	225	219	3
6	3	-6	72	70	5	8	3	-11	346	330	3
6	3	-5	366	324	2	8	3	-10	220	210	3
6	3	-4	565	523	3	8	3	-9	281	280	3
6	3	-3	394	369	2	8	3	-8	139	130	4
6	3	-2	386	363	2	8	3	-7	294	289	2
6	3	-1	146	153	2	8	3	-6	326	309	2
6	3	0	337	349	2	8	3	-5	222	214	3
6	3	1	483	489	2	8	3	-4	95	95	4
6	3	2	829	817	4	8	3	-3	148	152	3
6	3	3	405	392	2	8	3	-2	72	73	5
6	3	4	550	560	3	8	3	-1	415	423	2
6	3	5	297	301	2	8	3	0	514	534	3
6	3	6	246	242	2	8	3	1	250	258	2
6	3	7	106	107	4	8	3	2	630	639	3
6	3	8	66	73	7	8	3	4	72	57	6
6	3	9	135	127	4	8	3	5	379	382	2
6	3	10	240	255	3	8	3	6	140	154	4
6	3	12	116	119	5	8	3	7	209	209	3
6	3	13	134	136	5	8	3	8	73	70	8
6	3	14	201	195	4	8	3	9	264	258	3
7	3	-17	195	205	5	8	3	12	268	263	4
7	3	-15	207	214	4	9	3	-16	98	68	8
7	3	-14	75	97	9	9	3	-13	194	192	4
7	3	-13	143	144	4	9	3	-12	230	221	4
7	3	-10	436	420	2	9	3	-11	222	220	4
7	3	-9	160	156	3	9	3	-9	133	118	4
7	3	-8	122	124	4	9	3	-8	311	289	3
7	3	-7	337	327	2	9	3	-7	173	164	3
7	3	-6	73	64	5	9	3	-6	371	333	2
7	3	-5	182	181	3	9	3	-5	151	141	3
7	3	-4	67	49	5	9	3	-3	170	168	3
7	3	-3	533	498	3	9	3	-2	204	202	3
7	3	-2	380	371	2	9	3	-1	337	349	2
7	3	-1	120	126	3	9	3	1	163	149	3
7	3	0	371	384	2	9	3	2	74	79	6
7	3	1	597	607	3	9	3	4	315	317	3
7	3	2	578	591	3	9	3	5	127	123	5
7	3	3	559	558	3	9	3	6	99	97	6
7	3	4	271	273	2	9	3	8	190	188	4
7	3	5	281	287	2	9	3	9	88	94	7
7	3	6	252	257	3	9	3	11	227	231	4
7	3	7	136	143	4	10	3	-15	113	100	7
7	3	8	153	162	4	10	3	-13	162	147	5
7	3	9	92	99	6	10	3	-12	258	262	4
7	3	10	154	150	4	10	3	-10	111	86	5

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
10	3	-8	299	277	3	13	3	3	93	100	8
10	3	-7	345	322	3	14	3	-7	108	113	7
10	3	-6	297	274	3	14	3	-6	180	188	5
10	3	-5	109	94	5	14	3	-5	127	130	6
10	3	-4	178	169	3	14	3	-4	146	163	6
10	3	-2	265	267	3	14	3	1	117	110	7
10	3	-1	209	218	3	0	4	0	39	41	4
10	3	0	327	342	3	0	4	1	933	972	3
10	3	7	215	223	4	0	4	2	889	910	3
10	3	10	200	191	4	0	4	3	680	710	3
11	3	-14	124	119	6	0	4	4	707	719	3
11	3	-13	158	157	5	0	4	5	42	37	5
11	3	-9	345	321	3	0	4	6	77	88	3
11	3	-8	431	408	3	0	4	7	236	245	2
11	3	-6	144	139	4	0	4	8	231	234	2
11	3	-5	134	111	4	0	4	9	186	186	3
11	3	-4	75	89	8	0	4	10	92	112	5
11	3	-3	225	221	3	0	4	11	105	101	4
11	3	-2	141	135	4	0	4	13	162	162	4
11	3	-1	327	350	3	0	4	14	95	91	6
11	3	0	96	94	6	0	4	15	161	169	4
11	3	1	115	127	5	0	4	16	277	270	3
11	3	4	108	93	6	0	4	18	134	128	6
11	3	5	138	132	5	1	4	-17	271	266	4
11	3	6	93	95	7	1	4	-16	135	134	5
11	3	9	274	278	4	1	4	-15	216	217	4
12	3	-10	245	237	4	1	4	-12	414	435	2
12	3	-9	236	217	4	1	4	-10	102	112	4
12	3	-8	232	221	4	1	4	-9	349	364	2
12	3	-7	139	132	5	1	4	-8	524	545	3
12	3	-6	173	171	4	1	4	-6	382	411	2
12	3	-5	69	87	9	1	4	-5	889	930	3
12	3	-4	356	380	3	1	4	-4	109	115	2
12	3	-2	249	266	3	1	4	-3	651	683	3
12	3	-1	218	235	4	1	4	-2	634	634	3
12	3	3	194	191	4	1	4	-1	83	78	2
12	3	4	124	121	6	1	4	0	233	246	1
12	3	6	108	109	7	1	4	1	729	744	3
12	3	8	190	189	5	1	4	2	552	545	3
13	3	-11	169	158	5	1	4	3	616	635	3
13	3	-10	73	51	11	1	4	4	343	364	2
13	3	-9	119	104	6	1	4	5	593	568	3
13	3	-7	115	104	6	1	4	6	145	138	2
13	3	-6	90	80	8	1	4	7	144	142	3
13	3	-5	273	284	4	1	4	8	447	463	2
13	3	-4	115	110	6	1	4	9	311	309	2
13	3	-3	216	224	4	1	4	11	106	115	5
13	3	-2	155	164	5	1	4	12	477	476	3
13	3	-1	89	90	7	1	4	13	307	297	3
13	3	2	115	121	7	1	4	14	84	94	7

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
1	4	15	264	249	3	3	4	5	307	291	2
1	4	17	174	166	5	3	4	6	442	453	2
2	4	-18	126	139	6	3	4	7	209	206	2
2	4	-16	215	219	4	3	4	9	305	313	2
2	4	-13	275	278	3	3	4	11	405	401	3
2	4	-12	103	115	5	3	4	12	74	84	7
2	4	-11	132	139	4	3	4	13	434	422	3
2	4	-9	306	302	2	3	4	14	238	232	4
2	4	-8	63	64	5	4	4	-18	177	174	5
2	4	-7	597	605	3	4	4	-16	178	184	4
2	4	-6	942	974	4	4	4	-13	176	184	4
2	4	-5	246	222	2	4	4	-12	177	198	3
2	4	-4	445	447	2	4	4	-11	103	108	4
2	4	-2	267	276	1	4	4	-10	119	126	4
2	4	-1	142	136	2	4	4	-9	631	664	3
2	4	0	547	567	3	4	4	-8	679	705	3
2	4	1	108	107	2	4	4	-7	202	224	2
2	4	2	391	403	2	4	4	-6	216	226	2
2	4	3	515	539	3	4	4	-5	191	192	2
2	4	4	155	150	2	4	4	-4	113	120	2
2	4	5	356	353	2	4	4	-3	162	158	2
2	4	7	544	543	3	4	4	-2	581	571	3
2	4	8	201	211	2	4	4	-1	275	263	2
2	4	9	103	103	4	4	4	0	655	642	3
2	4	10	356	359	2	4	4	1	424	429	2
2	4	11	78	84	7	4	4	2	143	145	2
2	4	12	271	267	3	4	4	3	215	218	2
2	4	14	349	320	3	4	4	4	115	119	3
2	4	15	129	125	5	4	4	5	319	316	2
2	4	16	135	125	5	4	4	8	253	260	2
2	4	17	171	156	5	4	4	9	128	130	4
3	4	-17	232	238	4	4	4	10	508	503	3
3	4	-15	178	183	4	4	4	11	123	111	5
3	4	-14	186	193	4	4	4	12	484	476	3
3	4	-13	107	108	5	4	4	13	301	300	3
3	4	-12	96	97	5	4	4	15	123	116	6
3	4	-11	134	149	4	5	4	-16	101	113	7
3	4	-9	60	65	6	5	4	-15	80	80	7
3	4	-8	629	654	3	5	4	-14	203	207	4
3	4	-7	833	863	4	5	4	-13	194	211	4
3	4	-6	159	167	2	5	4	-12	127	151	4
3	4	-5	872	885	4	5	4	-11	195	204	3
3	4	-4	235	241	2	5	4	-10	383	391	2
3	4	-3	50	58	4	5	4	-9	590	607	3
3	4	-2	266	239	1	5	4	-8	323	323	2
3	4	-1	836	822	3	5	4	-7	276	277	2
3	4	1	655	654	3	5	4	-6	110	101	3
3	4	2	1061	1126	3	5	4	-5	341	333	2
3	4	3	122	118	2	5	4	-4	91	89	4
3	4	4	139	118	2	5	4	-3	964	933	4

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
5	4	-2	302	284	2	7	4	4	141	140	3
5	4	-1	660	617	3	7	4	6	360	368	2
5	4	0	179	164	2	7	4	7	214	215	3
5	4	1	127	128	3	7	4	9	338	329	3
5	4	2	708	707	3	8	4	-16	146	158	6
5	4	5	166	169	3	8	4	-15	226	239	4
5	4	6	327	325	2	8	4	-14	156	166	5
5	4	8	184	188	3	8	4	-12	272	273	3
5	4	9	391	393	2	8	4	-10	140	135	4
5	4	10	197	192	3	8	4	-9	106	106	5
5	4	11	425	418	3	8	4	-8	88	87	5
5	4	12	199	195	4	8	4	-7	107	94	4
6	4	-13	247	254	3	8	4	-6	444	419	2
6	4	-10	418	440	2	8	4	-5	331	322	2
6	4	-9	198	197	3	8	4	-4	302	288	2
6	4	-7	191	183	3	8	4	-3	243	232	2
6	4	-6	403	385	2	8	4	-1	85	92	5
6	4	-4	634	615	3	8	4	0	283	279	2
6	4	-3	568	544	3	8	4	1	143	145	4
6	4	-2	439	414	2	8	4	3	428	436	2
6	4	-1	144	149	2	8	4	4	78	75	6
6	4	0	228	223	2	8	4	5	363	366	3
6	4	1	504	503	2	8	4	6	313	311	3
6	4	2	49	46	7	8	4	7	116	108	5
6	4	3	134	123	3	8	4	8	189	190	4
6	4	4	126	133	3	8	4	13	94	78	8
6	4	5	206	207	3	9	4	-14	139	144	6
6	4	6	76	82	5	9	4	-13	222	209	4
6	4	7	362	363	2	9	4	-11	107	114	6
6	4	8	253	251	3	9	4	-10	272	276	3
6	4	9	91	96	6	9	4	-9	151	146	4
6	4	10	354	347	3	9	4	-8	165	156	4
6	4	11	116	125	5	9	4	-6	223	205	3
6	4	12	113	110	5	9	4	-5	241	231	3
7	4	-16	142	141	6	9	4	-4	269	255	3
7	4	-15	204	222	4	9	4	-3	166	160	3
7	4	-14	286	288	3	9	4	-2	124	115	4
7	4	-11	366	360	3	9	4	-1	226	233	3
7	4	-10	101	95	5	9	4	0	181	190	3
7	4	-9	188	188	3	9	4	1	258	261	3
7	4	-7	70	48	5	9	4	3	98	89	5
7	4	-6	88	81	5	9	4	4	313	329	3
7	4	-5	580	562	3	9	4	5	481	480	3
7	4	-4	447	413	2	9	4	6	135	134	5
7	4	-3	396	378	2	9	4	7	242	250	3
7	4	-1	66	67	5	9	4	8	114	116	6
7	4	0	388	396	2	9	4	11	122	126	7
7	4	1	472	466	2	9	4	12	98	94	8
7	4	2	88	79	5	10	4	-14	197	205	5
7	4	3	205	218	3	10	4	-13	143	126	6

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
10	4	-12	98	94	7	13	4	0	154	162	5
10	4	-11	127	123	5	13	4	1	210	210	4
10	4	-10	149	146	4	14	4	-6	91	103	8
10	4	-8	88	85	7	14	4	-1	88	92	8
10	4	-7	254	247	3	14	4	0	128	143	6
10	4	-6	76	60	7	14	4	1	121	115	7
10	4	-5	177	157	4	0	5	1	495	529	3
10	4	-4	115	115	5	0	5	2	687	709	3
10	4	-3	191	187	3	0	5	3	407	427	2
10	4	-2	211	213	3	0	5	4	234	230	2
10	4	1	184	193	4	0	5	5	472	489	2
10	4	2	137	143	4	0	5	6	727	760	3
10	4	4	419	424	3	0	5	7	339	330	2
10	4	5	78	90	8	0	5	8	551	558	3
10	4	6	375	376	3	0	5	9	320	335	2
10	4	7	264	266	4	0	5	10	211	206	3
10	4	10	104	115	8	0	5	11	642	645	3
10	4	11	123	115	7	0	5	12	132	141	4
11	4	-13	152	160	6	0	5	13	71	83	7
11	4	-11	120	115	6	0	5	14	121	116	5
11	4	-10	131	121	5	1	5	-15	254	258	3
11	4	-9	155	150	5	1	5	-13	169	182	4
11	4	-7	185	184	4	1	5	-11	201	208	3
11	4	-6	142	129	4	1	5	-10	78	68	5
11	4	-4	315	327	3	1	5	-9	637	671	3
11	4	-3	222	230	3	1	5	-8	440	460	2
11	4	-2	95	82	6	1	5	-7	323	327	2
11	4	0	290	296	3	1	5	-6	54	62	5
11	4	1	94	104	7	1	5	-5	97	104	3
11	4	2	98	98	6	1	5	-4	584	613	3
11	4	3	444	454	3	1	5	-3	664	672	3
11	4	5	321	324	3	1	5	-2	295	311	1
11	4	6	159	150	5	1	5	-1	316	320	1
12	4	-11	117	110	7	1	5	0	627	669	3
12	4	-10	156	155	5	1	5	1	378	415	2
12	4	-9	176	164	5	1	5	2	238	248	2
12	4	-8	203	196	4	1	5	3	413	411	2
12	4	-7	70	56	9	1	5	4	1086	1089	4
12	4	-5	208	202	4	1	5	5	310	328	2
12	4	-4	144	151	5	1	5	6	276	273	2
12	4	-3	182	194	4	1	5	7	440	447	2
12	4	-1	142	148	5	1	5	8	160	159	3
12	4	1	106	112	6	1	5	9	297	294	2
12	4	2	363	386	3	1	5	10	436	432	2
12	4	4	184	188	5	1	5	12	166	172	4
13	4	-9	272	267	4	1	5	16	69	68	10
13	4	-6	222	218	4	2	5	-16	171	172	5
13	4	-5	124	126	6	2	5	-14	222	233	3
13	4	-4	156	156	5	2	5	-13	84	86	6
13	4	-2	87	86	8	2	5	-12	169	182	3

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
2	5	-11	228	239	3	3	5	10	383	384	2
2	5	-10	439	450	2	3	5	11	148	151	4
2	5	-9	284	311	2	3	5	14	242	230	4
2	5	-8	242	239	2	3	5	16	261	241	4
2	5	-7	255	260	2	4	5	-17	222	224	4
2	5	-6	465	476	2	4	5	-15	137	145	5
2	5	-4	287	293	2	4	5	-14	150	164	4
2	5	-3	599	590	3	4	5	-12	327	343	3
2	5	-2	100	79	2	4	5	-11	265	268	3
2	5	-1	777	750	3	4	5	-9	84	82	5
2	5	0	223	237	2	4	5	-8	271	281	2
2	5	1	289	283	1	4	5	-7	198	193	2
2	5	2	101	102	2	4	5	-6	590	613	3
2	5	3	53	48	4	4	5	-5	735	747	3
2	5	4	598	652	3	4	5	-4	550	552	3
2	5	5	59	47	5	4	5	-3	466	454	2
2	5	6	482	509	2	4	5	-2	412	394	2
2	5	8	134	141	3	4	5	-1	204	206	2
2	5	9	144	145	3	4	5	0	389	390	2
2	5	10	322	318	2	4	5	1	206	208	2
2	5	11	516	518	3	4	5	2	747	739	3
2	5	12	112	113	5	4	5	3	176	195	2
2	5	13	63	70	9	4	5	4	270	280	2
2	5	15	157	158	5	4	5	5	339	331	2
2	5	17	138	128	6	4	5	6	132	135	3
3	5	-17	113	117	7	4	5	7	152	159	3
3	5	-16	182	182	5	4	5	8	361	357	2
3	5	-15	162	171	5	4	5	9	214	209	3
3	5	-14	71	58	8	4	5	10	68	66	7
3	5	-13	186	200	4	4	5	13	175	179	4
3	5	-12	103	109	5	4	5	15	190	188	5
3	5	-11	436	449	2	4	5	16	264	245	4
3	5	-10	335	354	2	5	5	-16	168	172	5
3	5	-8	323	333	2	5	5	-15	115	120	6
3	5	-7	390	396	2	5	5	-13	281	308	3
3	5	-5	387	401	2	5	5	-12	223	222	3
3	5	-4	899	895	4	5	5	-11	81	96	6
3	5	-3	127	118	2	5	5	-10	107	101	4
3	5	-2	580	589	3	5	5	-9	180	198	3
3	5	-1	166	179	2	5	5	-8	191	202	3
3	5	0	267	255	2	5	5	-7	290	311	2
3	5	1	329	331	2	5	5	-6	703	723	3
3	5	2	251	232	2	5	5	-5	632	634	3
3	5	3	246	277	2	5	5	-4	414	390	2
3	5	4	257	289	2	5	5	-3	161	146	2
3	5	5	305	322	2	5	5	-2	427	403	2
3	5	6	139	142	3	5	5	-1	428	408	2
3	5	7	78	72	4	5	5	0	70	61	4
3	5	8	73	76	6	5	5	1	654	647	3
3	5	9	405	414	2	5	5	2	277	294	2

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
5	5	3	311	314	2	7	5	1	223	214	2
5	5	4	72	73	5	7	5	2	529	535	3
5	5	5	352	353	2	7	5	3	602	607	3
5	5	6	424	432	2	7	5	4	349	365	2
5	5	7	304	304	2	7	5	5	211	216	3
5	5	8	114	101	4	7	5	6	152	143	4
5	5	9	110	126	5	7	5	8	98	86	5
5	5	14	252	242	4	7	5	9	95	98	6
5	5	15	215	196	4	7	5	10	197	184	4
6	5	-17	209	215	4	7	5	12	191	189	4
6	5	-16	168	165	5	7	5	13	148	151	6
6	5	-15	113	109	6	8	5	-16	124	120	7
6	5	-14	205	209	4	8	5	-15	188	183	5
6	5	-13	95	92	6	8	5	-14	122	113	6
6	5	-12	170	172	4	8	5	-13	217	228	4
6	5	-11	144	131	4	8	5	-12	293	296	3
6	5	-10	137	146	4	8	5	-11	148	146	4
6	5	-9	60	65	7	8	5	-9	107	93	5
6	5	-8	181	192	3	8	5	-8	87	90	6
6	5	-7	497	504	2	8	5	-7	266	257	3
6	5	-6	247	258	2	8	5	-5	447	441	2
6	5	-5	177	169	3	8	5	-3	170	164	3
6	5	-2	347	337	2	8	5	-2	396	391	2
6	5	-1	128	115	3	8	5	-1	108	112	4
6	5	0	614	597	3	8	5	0	105	110	4
6	5	1	158	159	3	8	5	1	285	287	2
6	5	2	279	265	2	8	5	2	419	427	2
6	5	3	96	92	4	8	5	3	504	529	3
6	5	4	420	428	2	8	5	4	138	146	4
6	5	5	558	575	3	8	5	5	217	221	3
6	5	6	225	230	3	8	5	6	116	115	5
6	5	7	96	94	5	8	5	8	136	135	5
6	5	10	62	68	9	8	5	9	213	209	4
6	5	11	166	164	4	8	5	10	145	143	5
6	5	13	247	240	4	8	5	11	163	153	5
6	5	14	178	180	5	8	5	12	144	130	6
7	5	-16	111	107	7	9	5	-14	214	222	4
7	5	-15	130	130	6	9	5	-13	271	273	4
7	5	-14	139	165	5	9	5	-12	196	195	4
7	5	-13	89	105	7	9	5	-10	238	237	3
7	5	-12	197	195	4	9	5	-8	224	210	3
7	5	-10	210	204	3	9	5	-7	88	82	6
7	5	-8	271	270	3	9	5	-5	217	215	3
7	5	-7	93	96	5	9	5	-4	137	140	4
7	5	-6	77	66	5	9	5	-2	215	211	3
7	5	-5	125	127	4	9	5	-1	79	88	6
7	5	-3	147	154	3	9	5	0	172	169	3
7	5	-2	182	177	3	9	5	1	287	308	3
7	5	-1	362	372	2	9	5	2	89	102	6
7	5	0	192	189	3	9	5	4	93	86	6

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
9	5	5	96	93	6	12	5	6	111	109	7
9	5	6	183	183	4	12	5	7	103	102	8
9	5	7	118	109	5	13	5	-9	133	135	6
9	5	8	259	252	3	13	5	-5	148	149	5
9	5	9	180	175	4	13	5	-3	217	226	4
9	5	11	124	120	6	13	5	-2	252	256	4
10	5	-14	181	188	5	13	5	1	168	177	5
10	5	-13	90	92	8	13	5	4	103	97	7
10	5	-11	265	263	4	14	5	-4	121	117	6
10	5	-9	216	222	4	14	5	-3	100	97	8
10	5	-7	128	131	5	0	6	0	199	194	2
10	5	-6	334	327	3	0	6	1	404	404	2
10	5	-5	282	284	3	0	6	2	611	651	3
10	5	-4	169	158	4	0	6	3	75	79	4
10	5	-3	172	173	4	0	6	4	107	106	3
10	5	-2	253	258	3	0	6	5	117	133	3
10	5	-1	303	310	3	0	6	6	92	88	4
10	5	0	147	161	4	0	6	7	522	543	3
10	5	1	156	152	4	0	6	8	376	399	2
10	5	3	183	189	4	0	6	9	299	297	2
10	5	4	122	116	5	0	6	10	93	96	5
10	5	5	134	129	5	0	6	11	81	88	6
10	5	6	79	57	8	0	6	12	261	262	3
10	5	7	123	123	5	0	6	13	276	278	3
10	5	8	131	134	6	0	6	14	75	70	8
10	5	10	139	133	6	0	6	15	106	108	6
11	5	-12	229	236	4	0	6	17	124	122	7
11	5	-10	128	127	6	1	6	-16	146	153	5
11	5	-7	315	312	3	1	6	-14	163	161	4
11	5	-6	117	108	6	1	6	-13	378	386	3
11	5	-4	257	260	3	1	6	-11	88	87	5
11	5	-3	164	154	4	1	6	-9	111	105	4
11	5	-2	131	132	5	1	6	-8	593	637	3
11	5	-1	167	185	4	1	6	-7	109	115	4
11	5	0	376	382	3	1	6	-6	102	110	3
11	5	6	126	133	6	1	6	-3	640	676	3
11	5	8	183	180	5	1	6	-2	257	258	2
11	5	9	178	179	5	1	6	-1	236	223	2
12	5	-11	75	95	10	1	6	0	150	134	2
12	5	-10	111	109	7	1	6	1	695	696	3
12	5	-8	223	213	4	1	6	2	419	425	2
12	5	-6	136	141	5	1	6	4	491	509	2
12	5	-5	145	145	5	1	6	6	136	152	3
12	5	-4	165	169	4	1	6	7	451	472	2
12	5	-3	101	91	7	1	6	8	306	312	2
12	5	-2	220	241	4	1	6	9	163	155	3
12	5	-1	362	359	3	1	6	11	320	322	3
12	5	0	91	73	7	1	6	12	349	347	3
12	5	3	119	124	6	2	6	-15	151	150	5
12	5	5	116	121	7	2	6	-14	389	399	3

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
2	6	-13	160	169	4	3	6	13	230	231	4
2	6	-10	99	101	4	3	6	14	193	181	4
2	6	-9	237	243	3	4	6	-17	123	127	7
2	6	-7	407	441	2	4	6	-16	179	195	5
2	6	-6	191	188	2	4	6	-15	93	98	7
2	6	-5	83	86	4	4	6	-13	182	190	4
2	6	-4	555	572	3	4	6	-12	119	127	5
2	6	-3	190	189	2	4	6	-11	199	215	3
2	6	-2	412	432	2	4	6	-10	84	75	6
2	6	-1	161	155	2	4	6	-9	415	428	2
2	6	0	613	624	3	4	6	-8	299	313	2
2	6	1	146	140	2	4	6	-6	150	150	3
2	6	2	152	155	2	4	6	-5	49	37	7
2	6	3	431	443	2	4	6	-4	418	431	2
2	6	4	283	292	2	4	6	-2	258	261	2
2	6	5	162	164	3	4	6	-1	122	114	3
2	6	6	462	493	2	4	6	0	86	87	4
2	6	7	308	317	2	4	6	1	590	605	3
2	6	8	357	365	2	4	6	2	283	290	2
2	6	10	135	136	4	4	6	3	528	534	3
2	6	11	279	275	3	4	6	4	85	81	4
2	6	12	83	93	6	4	6	6	481	489	2
2	6	13	72	78	8	4	6	7	345	356	2
2	6	14	138	129	5	4	6	8	449	432	2
2	6	15	154	148	5	4	6	9	244	235	3
2	6	16	214	189	5	4	6	10	71	80	7
3	6	-16	113	123	7	4	6	12	337	336	3
3	6	-15	336	335	3	4	6	13	158	160	5
3	6	-14	124	112	5	4	6	15	87	82	9
3	6	-12	120	127	5	5	6	-14	299	318	3
3	6	-11	62	74	8	5	6	-13	195	199	4
3	6	-10	167	170	3	5	6	-12	307	331	3
3	6	-9	97	100	4	5	6	-10	256	258	3
3	6	-8	557	569	3	5	6	-9	256	284	3
3	6	-7	458	477	2	5	6	-8	76	71	5
3	6	-5	162	165	2	5	6	-6	108	110	4
3	6	-3	803	837	4	5	6	-5	485	492	2
3	6	-2	64	65	4	5	6	-4	149	147	3
3	6	-1	394	389	2	5	6	-3	502	503	3
3	6	0	172	170	2	5	6	-2	97	95	4
3	6	1	227	231	2	5	6	-1	285	284	2
3	6	2	825	828	4	5	6	0	435	431	2
3	6	4	627	612	3	5	6	1	474	470	2
3	6	5	377	394	2	5	6	2	445	457	2
3	6	6	315	313	2	5	6	3	93	88	4
3	6	7	410	410	2	5	6	5	369	383	2
3	6	8	190	182	3	5	6	6	387	395	2
3	6	9	271	269	3	5	6	7	400	410	2
3	6	10	214	208	3	5	6	8	301	307	3
3	6	12	100	104	6	5	6	10	105	88	5

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
5	6	11	295	277	3	8	6	-10	170	176	4
5	6	12	164	167	4	8	6	-9	98	98	6
6	6	-15	85	98	8	8	6	-8	453	450	3
6	6	-13	169	164	4	8	6	-7	70	86	8
6	6	-12	115	119	5	8	6	-6	154	149	4
6	6	-11	86	96	6	8	6	-4	139	147	4
6	6	-10	128	150	4	8	6	-3	462	454	2
6	6	-9	66	67	7	8	6	-2	167	161	3
6	6	-8	155	163	3	8	6	-1	113	107	4
6	6	-7	162	174	3	8	6	0	119	119	4
6	6	-6	216	221	3	8	6	1	166	167	3
6	6	-5	147	155	3	8	6	2	268	284	3
6	6	-4	492	491	2	8	6	3	67	73	8
6	6	-3	102	98	4	8	6	5	220	217	3
6	6	-2	458	455	2	8	6	6	226	237	3
6	6	-1	217	230	2	8	6	7	131	131	5
6	6	0	378	385	2	8	6	10	137	125	6
6	6	1	419	430	2	8	6	11	140	127	6
6	6	2	68	68	6	8	6	12	166	159	5
6	6	4	275	280	2	9	6	-14	156	155	6
6	6	5	265	277	3	9	6	-13	83	74	8
6	6	6	162	167	4	9	6	-12	83	83	8
6	6	7	366	364	2	9	6	-11	90	78	7
6	6	9	87	82	6	9	6	-9	437	437	3
6	6	10	183	172	4	9	6	-7	81	69	6
6	6	12	168	166	5	9	6	-5	72	74	7
7	6	-14	201	212	4	9	6	-4	516	491	3
7	6	-13	169	168	4	9	6	-3	186	195	4
7	6	-12	138	140	5	9	6	-1	65	78	8
7	6	-10	137	142	4	9	6	0	217	221	3
7	6	-9	225	232	3	9	6	1	295	290	3
7	6	-8	103	97	5	9	6	2	84	94	6
7	6	-7	388	391	2	9	6	3	95	106	6
7	6	-6	188	199	3	9	6	4	140	129	4
7	6	-5	375	377	2	9	6	5	250	246	3
7	6	-4	213	207	3	9	6	6	162	157	4
7	6	-3	253	244	2	9	6	7	136	141	5
7	6	-2	270	263	2	9	6	8	86	81	7
7	6	-1	190	187	3	9	6	10	74	68	10
7	6	0	399	399	2	9	6	11	202	190	5
7	6	1	141	144	3	10	6	-13	99	98	8
7	6	2	101	104	4	10	6	-11	71	51	10
7	6	3	276	287	3	10	6	-10	380	376	3
7	6	6	218	213	3	10	6	-8	122	120	5
7	6	7	225	219	3	10	6	-7	67	77	9
7	6	11	244	234	4	10	6	-5	191	180	4
7	6	13	90	75	8	10	6	-4	147	155	4
8	6	-15	134	134	6	10	6	-3	181	176	4
8	6	-14	130	123	6	10	6	-1	90	85	6
8	6	-13	126	116	6	10	6	0	224	226	3

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
10	6	1	204	206	4	1	7	-10	120	122	4
10	6	2	111	116	5	1	7	-9	221	222	3
10	6	3	77	69	7	1	7	-8	177	189	3
10	6	4	229	237	3	1	7	-7	687	727	4
10	6	6	189	192	4	1	7	-6	232	257	2
10	6	7	238	230	4	1	7	-5	80	84	4
11	6	-12	120	122	7	1	7	-4	520	545	3
11	6	-11	295	295	4	1	7	-3	122	108	3
11	6	-9	83	85	9	1	7	-2	264	273	2
11	6	-8	170	159	4	1	7	-1	459	451	2
11	6	-6	312	306	3	1	7	0	379	380	2
11	6	-5	247	238	4	1	7	1	66	72	4
11	6	-4	268	274	3	1	7	2	456	474	2
11	6	0	320	342	3	1	7	3	86	97	4
11	6	1	201	206	4	1	7	4	372	372	2
11	6	2	123	123	5	1	7	5	634	661	3
11	6	3	97	108	7	1	7	6	219	219	3
11	6	5	135	147	6	1	7	7	83	70	5
11	6	6	160	169	5	1	7	8	310	313	2
12	6	-9	119	116	7	1	7	10	326	310	3
12	6	-7	187	178	5	1	7	11	325	320	3
12	6	-6	143	141	5	1	7	12	126	119	5
12	6	-5	134	127	5	1	7	13	138	139	5
12	6	-4	99	104	7	1	7	14	98	93	6
12	6	-3	82	85	8	1	7	15	173	162	5
12	6	-1	220	220	4	1	7	16	107	101	7
12	6	0	222	221	4	2	7	-16	165	162	5
12	6	1	135	134	6	2	7	-15	172	169	5
12	6	6	124	124	7	2	7	-14	218	222	4
13	6	-8	197	190	5	2	7	-12	121	123	5
13	6	-1	130	128	6	2	7	-11	66	83	8
13	6	0	121	126	7	2	7	-10	328	331	3
13	6	3	113	108	7	2	7	-9	165	168	3
0	7	1	114	135	3	2	7	-8	221	235	3
0	7	2	165	162	2	2	7	-6	238	238	2
0	7	3	452	470	2	2	7	-5	574	605	3
0	7	5	390	387	2	2	7	-4	181	202	2
0	7	6	581	587	3	2	7	-3	198	213	2
0	7	7	168	171	3	2	7	-2	544	543	3
0	7	8	214	226	3	2	7	-1	394	396	2
0	7	11	232	240	3	2	7	1	300	326	2
0	7	12	95	97	6	2	7	2	125	123	3
0	7	13	148	147	5	2	7	4	663	666	3
0	7	14	244	235	4	2	7	6	113	128	4
0	7	15	94	104	8	2	7	8	127	122	4
0	7	16	194	176	5	2	7	9	283	269	3
1	7	-15	261	248	4	2	7	10	367	358	3
1	7	-14	103	97	6	2	7	11	113	105	5
1	7	-13	110	123	6	2	7	13	91	97	7
1	7	-12	99	96	5	2	7	15	197	186	5

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
3	7	-16	177	181	5	5	7	-10	179	186	4
3	7	-15	272	282	4	5	7	-8	141	143	4
3	7	-13	162	163	4	5	7	-7	53	55	8
3	7	-11	322	337	3	5	7	-6	164	174	3
3	7	-10	128	138	4	5	7	-5	562	568	3
3	7	-9	180	190	3	5	7	-4	275	281	2
3	7	-7	420	437	2	5	7	-3	196	210	3
3	7	-6	230	245	2	5	7	-2	302	301	2
3	7	-5	88	95	4	5	7	-1	441	441	2
3	7	-4	144	145	3	5	7	0	98	88	4
3	7	-2	498	514	2	5	7	1	555	552	3
3	7	-1	396	385	2	5	7	2	121	122	4
3	7	0	337	355	2	5	7	3	75	75	5
3	7	1	302	313	2	5	7	4	266	268	2
3	7	2	247	231	2	5	7	6	246	258	3
3	7	3	756	765	4	5	7	7	264	268	3
3	7	4	58	72	7	5	7	8	69	66	7
3	7	5	180	184	3	5	7	9	58	57	9
3	7	6	79	92	5	5	7	10	212	200	3
3	7	7	226	227	3	5	7	11	141	130	5
3	7	8	108	99	5	5	7	12	252	252	4
3	7	9	464	452	2	6	7	-14	79	86	9
3	7	10	160	151	4	6	7	-12	210	221	4
3	7	12	150	139	4	6	7	-11	354	374	3
3	7	14	239	234	4	6	7	-9	227	228	3
3	7	15	169	154	5	6	7	-8	77	81	6
4	7	-14	129	124	5	6	7	-7	105	117	5
4	7	-12	197	212	4	6	7	-6	218	223	3
4	7	-10	159	169	4	6	7	-5	307	311	2
4	7	-9	98	103	5	6	7	-4	139	147	4
4	7	-8	244	252	3	6	7	-3	249	241	2
4	7	-6	51	37	7	6	7	-2	107	110	4
4	7	-5	327	337	2	6	7	0	481	474	2
4	7	-4	457	460	2	6	7	1	138	138	3
4	7	-3	472	481	2	6	7	2	147	145	3
4	7	-2	356	361	2	6	7	3	115	104	4
4	7	-1	311	302	2	6	7	4	288	289	3
4	7	0	622	615	3	6	7	5	250	253	3
4	7	1	145	130	3	6	7	6	93	85	5
4	7	2	469	474	2	6	7	7	98	105	5
4	7	4	140	145	3	6	7	8	95	101	6
4	7	5	229	228	3	6	7	9	328	328	3
4	7	6	247	262	3	6	7	10	255	250	3
4	7	7	223	227	3	6	7	11	130	129	5
4	7	8	382	381	3	7	7	-13	184	192	5
4	7	9	140	128	4	7	7	-12	280	292	3
4	7	11	126	113	5	7	7	-11	159	177	4
4	7	13	235	218	4	7	7	-10	271	279	3
5	7	-13	96	102	7	7	7	-9	157	163	4
5	7	-11	334	342	3	7	7	-8	145	148	4

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
7	7	-6	313	321	3	9	7	-3	289	283	3
7	7	-4	54	41	7	9	7	-2	106	109	5
7	7	-3	81	81	5	9	7	0	108	105	5
7	7	-2	195	191	3	9	7	3	253	260	3
7	7	-1	621	618	3	9	7	4	74	56	7
7	7	0	320	313	2	9	7	6	135	143	5
7	7	1	124	116	4	9	7	9	222	212	4
7	7	2	124	128	4	10	7	-12	182	179	5
7	7	3	333	335	3	10	7	-11	128	137	6
7	7	4	148	150	4	10	7	-9	158	153	5
7	7	5	145	149	4	10	7	-8	176	183	4
7	7	6	76	82	7	10	7	-7	82	95	8
7	7	7	139	150	4	10	7	-6	307	314	3
7	7	8	242	245	3	10	7	-5	66	55	9
7	7	9	124	128	5	10	7	-4	226	225	3
7	7	10	117	114	6	10	7	-2	136	129	5
7	7	11	156	148	5	10	7	-1	306	310	3
7	7	12	88	85	9	10	7	1	73	77	8
8	7	-14	149	171	6	10	7	2	114	97	5
8	7	-13	122	109	6	10	7	3	97	94	6
8	7	-12	157	160	5	10	7	5	111	114	6
8	7	-11	167	169	4	10	7	8	184	176	5
8	7	-10	229	234	4	11	7	-10	128	119	6
8	7	-9	114	121	6	11	7	-9	218	211	4
8	7	-8	165	171	4	11	7	-7	326	321	3
8	7	-7	170	172	4	11	7	-5	153	155	5
8	7	-6	121	123	5	11	7	-3	135	148	5
8	7	-4	306	297	3	11	7	-2	199	201	4
8	7	-3	161	161	4	11	7	0	68	43	9
8	7	-2	293	298	3	11	7	2	139	152	5
8	7	-1	81	75	6	11	7	4	166	167	5
8	7	0	85	78	6	11	7	5	97	101	8
8	7	1	107	100	5	11	7	6	83	86	9
8	7	2	121	119	4	11	7	7	115	106	7
8	7	3	122	124	5	12	7	-8	222	228	4
8	7	4	293	296	3	12	7	-3	103	97	7
8	7	5	100	112	6	12	7	-1	137	131	6
8	7	6	96	108	6	12	7	1	131	138	6
8	7	7	232	239	4	12	7	2	132	137	6
8	7	9	132	127	5	12	7	3	133	151	6
8	7	10	252	240	4	12	7	4	165	163	5
9	7	-13	184	190	5	13	7	-3	83	90	9
9	7	-11	192	191	4	13	7	0	75	91	11
9	7	-10	193	197	4	0	8	0	169	160	3
9	7	-9	113	126	6	0	8	1	569	597	3
9	7	-8	252	256	3	0	8	2	445	456	2
9	7	-7	104	117	6	0	8	3	388	397	2
9	7	-6	116	120	5	0	8	4	119	121	3
9	7	-5	240	244	3	0	8	6	226	239	3
9	7	-4	150	138	4	0	8	7	220	222	3

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
0	8	8	379	381	2	2	8	12	138	136	5
0	8	9	340	356	3	2	8	13	134	136	5
0	8	10	123	121	5	2	8	15	84	77	9
0	8	11	104	113	6	3	8	-15	132	138	6
0	8	12	260	265	3	3	8	-13	140	148	5
0	8	13	72	66	8	3	8	-12	364	386	3
1	8	-14	124	129	6	3	8	-10	167	163	4
1	8	-13	310	310	3	3	8	-9	66	60	7
1	8	-12	115	119	5	3	8	-7	99	106	5
1	8	-10	295	294	3	3	8	-6	327	338	2
1	8	-8	269	270	3	3	8	-4	112	110	4
1	8	-7	167	172	3	3	8	-3	566	585	3
1	8	-6	91	100	5	3	8	-2	448	448	2
1	8	-5	115	116	4	3	8	1	219	220	2
1	8	-4	335	359	2	3	8	2	149	143	3
1	8	-3	414	432	2	3	8	3	59	63	7
1	8	-2	300	340	2	3	8	4	425	423	2
1	8	-1	183	182	2	3	8	5	256	258	3
1	8	0	437	442	2	3	8	6	373	382	2
1	8	1	383	387	2	3	8	7	248	239	3
1	8	2	308	314	2	3	8	8	236	240	3
1	8	5	250	247	2	3	8	10	99	90	5
1	8	6	509	508	3	3	8	12	146	134	5
1	8	7	367	383	2	3	8	13	101	100	7
1	8	8	146	152	4	3	8	14	103	94	7
1	8	9	96	85	5	4	8	-14	122	125	6
1	8	10	259	269	3	4	8	-13	368	376	3
1	8	12	90	91	7	4	8	-12	193	208	4
1	8	13	114	102	5	4	8	-11	132	148	5
2	8	-16	97	98	8	4	8	-10	174	188	4
2	8	-14	302	299	3	4	8	-8	88	99	5
2	8	-13	87	90	7	4	8	-7	305	311	3
2	8	-12	144	167	5	4	8	-6	177	191	3
2	8	-11	401	399	3	4	8	-4	339	353	2
2	8	-10	217	226	3	4	8	-3	261	278	2
2	8	-9	314	338	3	4	8	1	144	132	3
2	8	-5	349	363	2	4	8	2	146	139	3
2	8	-4	267	283	2	4	8	3	337	351	2
2	8	-3	335	346	2	4	8	4	74	98	6
2	8	-2	502	515	2	4	8	5	329	322	2
2	8	-1	257	253	2	4	8	6	428	439	2
2	8	0	258	248	2	4	8	7	223	223	3
2	8	1	177	183	3	4	8	8	306	307	3
2	8	2	184	193	3	4	8	11	122	114	5
2	8	4	145	151	3	4	8	12	242	229	4
2	8	5	274	266	2	5	8	-14	269	281	4
2	8	6	227	227	3	5	8	-13	224	229	4
2	8	7	196	198	3	5	8	-12	119	116	6
2	8	8	95	85	5	5	8	-9	88	103	6
2	8	9	254	251	3	5	8	-8	293	307	3

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
5	8	-5	195	186	3	7	8	7	176	177	4
5	8	-4	322	326	2	7	8	10	105	105	7
5	8	-3	163	165	3	7	8	11	247	228	4
5	8	-2	132	134	3	8	8	-11	164	177	5
5	8	-1	269	274	2	8	8	-10	209	207	4
5	8	0	179	172	3	8	8	-8	341	356	3
5	8	1	417	401	2	8	8	-7	108	130	6
5	8	2	286	277	2	8	8	-6	150	162	4
5	8	3	153	157	3	8	8	-5	176	164	4
5	8	4	314	316	3	8	8	-4	126	130	5
5	8	5	254	251	3	8	8	-3	205	213	3
5	8	6	318	326	3	8	8	-2	110	114	5
5	8	7	348	343	3	8	8	0	178	174	3
5	8	11	158	141	5	8	8	1	225	230	3
5	8	12	103	99	7	8	8	2	150	162	4
6	8	-15	156	163	5	8	8	3	232	231	3
6	8	-12	111	122	6	8	8	5	151	159	4
6	8	-11	100	114	6	8	8	6	215	222	4
6	8	-9	291	303	3	8	8	9	110	120	7
6	8	-8	253	263	3	8	8	10	162	160	5
6	8	-6	95	99	5	9	8	-12	146	159	6
6	8	-5	278	285	3	9	8	-9	302	313	3
6	8	-4	236	237	3	9	8	-6	143	141	5
6	8	-3	176	184	3	9	8	-5	254	262	3
6	8	-2	360	360	2	9	8	-4	108	103	5
6	8	-1	234	233	3	9	8	-3	133	132	5
6	8	0	214	209	3	9	8	-2	195	199	4
6	8	1	244	246	3	9	8	0	180	184	4
6	8	2	106	98	5	9	8	1	137	139	5
6	8	3	317	314	3	9	8	2	209	204	3
6	8	4	126	126	4	9	8	5	147	147	5
6	8	5	330	326	3	9	8	6	81	66	9
6	8	6	254	254	3	9	8	8	86	80	8
6	8	7	102	106	5	9	8	9	100	95	8
6	8	11	116	112	6	10	8	-10	253	257	4
6	8	12	159	150	5	10	8	-8	76	74	9
7	8	-13	191	198	5	10	8	-7	115	115	6
7	8	-10	231	248	4	10	8	-1	288	302	3
7	8	-9	279	289	3	10	8	0	107	113	6
7	8	-7	182	191	4	10	8	1	142	151	5
7	8	-6	149	148	4	10	8	2	118	118	5
7	8	-5	216	223	3	10	8	6	84	90	9
7	8	-4	113	114	5	11	8	-10	145	132	6
7	8	-3	83	88	6	11	8	-9	82	82	9
7	8	-2	374	366	2	11	8	-8	145	136	6
7	8	0	94	87	6	11	8	-5	108	115	7
7	8	1	231	222	3	11	8	-2	350	370	3
7	8	2	195	202	3	11	8	0	119	124	6
7	8	4	256	255	3	11	8	1	174	171	5
7	8	6	80	79	7	11	8	2	73	84	10

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
11	8	5	87	89	9	2	9	10	140	122	5
12	8	-6	111	106	7	2	9	13	286	273	4
12	8	-3	243	245	4	3	9	-15	146	146	6
12	8	-1	90	87	8	3	9	-11	123	123	5
12	8	0	217	217	4	3	9	-10	326	334	3
0	9	2	167	160	3	3	9	-8	159	155	4
0	9	3	645	651	3	3	9	-7	270	273	3
0	9	5	124	130	4	3	9	-6	357	378	3
0	9	6	191	191	3	3	9	-3	98	104	5
0	9	7	111	118	5	3	9	-2	228	238	3
0	9	8	74	89	7	3	9	-1	70	47	6
0	9	9	66	65	8	3	9	0	534	545	3
0	9	10	182	180	4	3	9	1	320	320	2
0	9	13	201	205	4	3	9	2	95	91	5
1	9	-14	158	151	5	3	9	3	164	156	3
1	9	-9	176	196	4	3	9	4	280	287	3
1	9	-8	92	99	6	3	9	5	262	265	3
1	9	-7	124	123	4	3	9	7	150	152	4
1	9	-6	417	445	2	3	9	8	146	147	4
1	9	-5	138	148	4	3	9	9	127	119	5
1	9	-4	504	532	3	3	9	11	119	120	6
1	9	-2	143	141	3	3	9	12	275	255	4
1	9	-1	303	309	2	3	9	13	142	124	6
1	9	0	188	190	3	4	9	-13	77	89	9
1	9	1	284	287	2	4	9	-11	233	237	4
1	9	2	745	764	4	4	9	-10	107	114	6
1	9	5	99	102	5	4	9	-9	160	162	4
1	9	6	192	187	3	4	9	-8	97	98	6
1	9	9	329	320	3	4	9	-7	366	384	3
1	9	14	230	224	4	4	9	-4	174	182	3
2	9	-15	179	171	5	4	9	-3	248	261	3
2	9	-14	120	133	6	4	9	-1	415	434	2
2	9	-10	113	118	6	4	9	0	348	340	2
2	9	-9	266	278	3	4	9	1	135	129	4
2	9	-7	285	306	3	4	9	2	79	66	5
2	9	-6	157	163	4	4	9	3	160	160	3
2	9	-5	389	414	2	4	9	4	230	214	3
2	9	-4	91	96	5	4	9	5	172	169	4
2	9	-2	274	276	2	4	9	8	68	61	8
2	9	-1	228	227	2	4	9	11	294	267	3
2	9	0	96	92	4	4	9	12	114	110	7
2	9	1	581	581	3	4	9	13	205	193	5
2	9	2	68	62	6	5	9	-13	85	87	8
2	9	3	65	64	6	5	9	-12	110	118	7
2	9	4	108	112	4	5	9	-11	119	143	6
2	9	5	126	126	4	5	9	-10	241	261	3
2	9	6	136	126	4	5	9	-8	264	272	3
2	9	7	98	96	5	5	9	-7	182	201	4
2	9	8	238	241	3	5	9	-6	73	75	7
2	9	9	133	119	5	5	9	-5	239	239	3

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
5	9	-4	263	263	3	8	9	2	139	136	5
5	9	-3	108	93	4	8	9	3	100	110	6
5	9	-2	192	191	3	8	9	5	164	163	5
5	9	-1	255	259	3	8	9	7	390	381	3
5	9	0	116	107	4	9	9	-10	96	95	7
5	9	3	172	165	3	9	9	-9	82	77	8
5	9	4	247	243	3	9	9	-8	152	156	5
5	9	5	146	143	4	9	9	-7	113	107	6
5	9	8	158	160	4	9	9	-6	247	256	4
5	9	10	309	306	3	9	9	-5	108	109	6
5	9	12	176	158	5	9	9	-4	179	188	4
6	9	-12	100	100	7	9	9	-2	96	97	7
6	9	-11	270	278	3	9	9	-1	179	188	4
6	9	-9	306	315	3	9	9	0	108	108	6
6	9	-8	120	128	5	9	9	1	79	79	8
6	9	-5	262	264	3	9	9	4	84	94	8
6	9	-4	69	73	7	9	9	6	327	324	4
6	9	-3	77	90	6	10	9	-9	83	85	9
6	9	-1	143	144	4	10	9	-7	99	108	7
6	9	1	85	78	6	10	9	-6	84	88	9
6	9	2	70	71	7	10	9	-5	212	226	4
6	9	3	175	170	4	10	9	-3	220	215	4
6	9	4	203	209	3	10	9	-2	267	274	4
6	9	6	96	92	6	10	9	-1	105	114	7
6	9	7	180	182	4	10	9	3	104	87	6
6	9	9	319	313	3	10	9	5	269	247	4
6	9	11	120	106	6	11	9	-8	108	108	7
7	9	-12	270	290	4	11	9	-7	86	96	9
7	9	-10	302	320	3	11	9	-6	145	138	6
7	9	-8	71	69	8	11	9	-4	198	206	5
7	9	-7	125	129	5	11	9	-3	184	181	5
7	9	-6	239	248	3	11	9	-2	121	135	7
7	9	-5	182	178	4	11	9	2	155	159	5
7	9	-4	236	233	3	12	9	-3	91	85	9
7	9	-2	119	110	5	0	10	0	246	254	3
7	9	-1	78	80	7	0	10	1	411	422	2
7	9	0	93	96	6	0	10	2	95	84	5
7	9	2	165	160	4	0	10	3	210	220	3
7	9	3	228	225	3	0	10	4	120	111	4
7	9	6	127	124	5	0	10	5	308	307	3
7	9	7	99	104	7	0	10	6	307	305	3
7	9	8	352	343	3	0	10	7	217	215	3
8	9	-11	127	137	6	0	10	8	234	239	3
8	9	-9	90	88	7	0	10	9	228	233	3
8	9	-8	125	137	5	0	10	11	181	183	4
8	9	-7	110	137	6	0	10	13	91	68	8
8	9	-6	168	170	4	1	10	-14	134	130	6
8	9	-5	291	281	3	1	10	-12	91	95	8
8	9	-2	94	88	6	1	10	-10	349	348	3
8	9	1	207	204	4	1	10	-9	232	237	3

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
1	10	-8	159	163	4	3	10	6	107	112	5
1	10	-7	180	178	4	3	10	7	75	81	7
1	10	-6	186	190	3	3	10	8	245	244	3
1	10	-5	98	89	5	3	10	9	133	137	5
1	10	-4	263	270	3	3	10	10	138	134	5
1	10	-3	167	160	3	3	10	11	244	236	4
1	10	-2	455	443	2	3	10	12	121	110	7
1	10	-1	68	40	6	4	10	-13	165	163	5
1	10	0	213	208	3	4	10	-11	218	213	4
1	10	2	193	186	3	4	10	-9	85	65	6
1	10	3	254	252	3	4	10	-7	185	193	4
1	10	4	134	130	4	4	10	-5	112	114	5
1	10	5	63	66	8	4	10	-3	205	212	3
1	10	6	312	304	3	4	10	-1	62	48	8
1	10	8	104	124	6	4	10	0	267	274	3
1	10	10	304	282	3	4	10	1	73	71	7
1	10	11	114	113	6	4	10	2	69	54	7
1	10	13	229	219	4	4	10	3	237	231	3
2	10	-14	85	93	9	4	10	4	342	334	3
2	10	-12	73	100	9	4	10	5	319	311	3
2	10	-11	346	340	3	4	10	7	122	116	5
2	10	-10	242	242	4	4	10	9	64	52	9
2	10	-9	273	286	3	4	10	10	200	190	4
2	10	-7	183	190	4	4	10	11	74	71	10
2	10	-6	163	178	4	4	10	12	117	104	7
2	10	-5	307	316	3	5	10	-12	87	83	8
2	10	-4	182	178	3	5	10	-9	79	87	7
2	10	-3	207	206	3	5	10	-8	190	200	4
2	10	-2	140	137	4	5	10	-7	66	76	8
2	10	2	251	258	3	5	10	-6	67	74	8
2	10	4	66	69	8	5	10	-5	119	127	5
2	10	5	382	368	3	5	10	-4	235	241	3
2	10	6	97	117	6	5	10	-3	88	77	6
2	10	9	248	255	3	5	10	-2	96	93	5
2	10	10	160	167	4	5	10	-1	308	305	3
2	10	12	218	213	4	5	10	0	245	247	3
2	10	13	73	45	10	5	10	2	186	194	4
3	10	-12	285	268	4	5	10	3	299	290	3
3	10	-10	268	289	3	5	10	4	364	349	3
3	10	-7	191	202	4	5	10	9	68	84	10
3	10	-6	154	166	4	5	10	11	105	95	7
3	10	-5	119	111	4	6	10	-10	119	127	6
3	10	-4	60	69	8	6	10	-9	115	120	6
3	10	-3	150	159	4	6	10	-8	92	101	7
3	10	-2	62	36	7	6	10	-6	169	177	4
3	10	0	60	44	8	6	10	-5	227	226	3
3	10	1	295	303	3	6	10	-4	118	119	5
3	10	3	77	80	7	6	10	-3	199	202	4
3	10	4	261	257	3	6	10	-2	173	164	4
3	10	5	273	273	3	6	10	-1	469	472	3

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
6	10	0	82	51	7	10	10	-2	126	133	6
6	10	1	146	150	4	10	10	-1	137	134	6
6	10	2	184	183	4	11	10	-3	88	85	9
6	10	3	333	331	3	11	10	-2	264	263	4
6	10	4	133	143	5	11	10	-1	86	69	9
6	10	5	215	212	4	0	11	1	264	257	3
6	10	8	106	109	7	0	11	2	115	107	5
7	10	-12	87	76	9	0	11	3	118	112	4
7	10	-11	120	125	7	0	11	6	141	149	5
7	10	-10	79	99	9	0	11	7	245	238	3
7	10	-9	190	199	4	0	11	9	134	123	5
7	10	-8	117	127	6	0	11	10	182	184	4
7	10	-7	151	154	5	0	11	11	112	117	7
7	10	-6	133	141	5	0	11	12	143	144	6
7	10	-5	100	96	6	1	11	-12	80	101	10
7	10	-4	119	120	5	1	11	-11	106	106	7
7	10	-3	164	166	4	1	11	-10	72	81	9
7	10	-2	320	317	3	1	11	-8	175	176	4
7	10	0	65	71	9	1	11	-7	89	99	7
7	10	2	260	251	3	1	11	-6	188	195	4
7	10	3	218	228	4	1	11	-5	176	178	4
7	10	4	302	299	3	1	11	-4	140	138	4
7	10	6	106	110	7	1	11	-2	332	334	3
7	10	7	116	112	6	1	11	0	236	247	3
7	10	8	94	90	8	1	11	1	132	124	4
8	10	-11	90	104	8	1	11	2	267	258	3
8	10	-10	118	109	6	1	11	4	105	97	5
8	10	-8	90	92	8	1	11	5	86	88	6
8	10	-5	111	105	6	1	11	6	261	253	3
8	10	-4	285	290	3	1	11	9	247	233	4
8	10	-3	206	200	4	1	11	10	144	142	5
8	10	-2	117	113	5	1	11	11	160	144	5
8	10	-1	213	206	4	1	11	12	81	94	10
8	10	1	127	130	5	2	11	-12	107	104	7
8	10	2	131	125	5	2	11	-7	216	217	3
8	10	3	264	262	3	2	11	-6	101	115	6
8	10	6	188	182	5	2	11	-3	303	296	3
8	10	7	117	105	7	2	11	-1	224	223	3
9	10	-7	70	57	10	2	11	0	132	134	4
9	10	-6	150	144	5	2	11	1	310	297	3
9	10	-5	266	283	4	2	11	2	155	142	4
9	10	-3	174	172	4	2	11	3	93	95	6
9	10	-2	161	158	5	2	11	4	170	174	4
9	10	0	91	96	7	2	11	5	177	191	4
9	10	2	169	163	5	2	11	6	85	86	7
9	10	5	162	153	5	2	11	8	256	244	3
10	10	-7	90	104	9	2	11	9	233	215	4
10	10	-6	119	111	6	2	11	10	107	105	7
10	10	-4	130	127	6	2	11	12	92	89	9
10	10	-3	80	82	9	3	11	-12	168	173	5

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
3	11	-10	95	103	7	6	11	-1	80	75	8
3	11	-8	63	63	9	6	11	0	203	209	4
3	11	-6	100	108	6	6	11	1	241	239	4
3	11	-5	113	125	5	6	11	2	182	175	4
3	11	-4	266	259	3	6	11	3	123	119	5
3	11	-3	63	55	9	6	11	4	77	72	8
3	11	-2	289	297	3	6	11	6	84	79	8
3	11	-1	179	173	4	6	11	7	195	200	4
3	11	0	199	192	3	6	11	8	74	62	10
3	11	1	172	169	4	6	11	9	81	72	9
3	11	2	93	86	6	7	11	-10	105	97	7
3	11	3	128	127	4	7	11	-9	113	109	7
3	11	5	213	207	3	7	11	-8	227	237	4
3	11	7	215	210	4	7	11	-7	81	82	9
3	11	8	279	265	3	7	11	-6	200	200	4
3	11	9	125	109	6	7	11	-5	168	164	5
4	11	-7	164	181	4	7	11	-4	115	111	6
4	11	-6	145	141	4	7	11	-1	120	120	6
4	11	-5	218	214	3	7	11	0	212	206	4
4	11	-3	348	349	3	7	11	1	132	128	5
4	11	-2	235	223	3	7	11	2	162	160	5
4	11	0	130	122	4	7	11	6	180	182	5
4	11	1	218	213	3	7	11	7	79	80	9
4	11	2	108	114	5	8	11	-9	200	189	5
4	11	4	192	186	4	8	11	-7	107	120	7
4	11	6	162	148	4	8	11	-6	207	217	4
4	11	7	161	148	4	8	11	-5	128	111	6
4	11	8	111	107	6	8	11	-4	85	97	8
4	11	9	74	70	10	8	11	-1	83	80	8
4	11	10	73	63	10	8	11	0	122	120	6
5	11	-12	100	111	8	8	11	1	79	71	9
5	11	-8	178	181	4	8	11	3	130	131	6
5	11	-7	82	73	7	8	11	4	137	133	6
5	11	-6	153	162	4	8	11	5	146	131	6
5	11	-4	279	282	3	9	11	-7	110	122	7
5	11	-3	208	205	4	9	11	-6	101	94	7
5	11	-2	84	82	7	9	11	-5	71	76	10
5	11	0	125	120	5	9	11	-1	176	177	5
5	11	1	167	172	4	9	11	2	182	177	5
5	11	3	186	184	4	9	11	4	104	111	8
5	11	5	141	132	5	10	11	-2	188	178	5
5	11	6	122	118	5	0	12	2	129	131	4
5	11	8	166	169	5	0	12	5	387	373	3
5	11	10	123	125	6	0	12	6	101	95	7
6	11	-9	144	144	5	0	12	7	165	162	4
6	11	-8	98	100	7	0	12	8	131	124	5
6	11	-7	163	166	4	0	12	11	94	100	8
6	11	-6	67	69	9	1	12	-9	98	117	7
6	11	-5	240	245	3	1	12	-8	106	105	7
6	11	-4	149	145	5	1	12	-6	389	383	3

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
1	12	-5	76	68	8	6	12	-7	75	81	10
1	12	0	86	78	6	6	12	-3	108	97	6
1	12	1	111	108	5	6	12	-2	170	161	5
1	12	4	372	379	3	6	12	-1	433	434	3
1	12	5	142	125	5	6	12	1	138	138	6
1	12	6	211	194	4	6	12	3	204	196	4
1	12	7	123	125	6	6	12	4	75	79	9
1	12	10	146	133	6	6	12	7	112	109	7
2	12	-11	166	154	5	7	12	-6	79	88	9
2	12	-9	197	200	4	7	12	-3	117	110	6
2	12	-7	364	356	3	7	12	-2	261	257	4
2	12	-2	106	103	5	7	12	0	169	163	5
2	12	-1	172	166	4	7	12	3	97	88	8
2	12	0	86	94	7	7	12	4	129	126	6
2	12	3	203	196	3	8	12	-5	67	45	10
2	12	5	216	199	4	8	12	-3	156	161	5
2	12	6	116	103	6	8	12	-2	76	66	10
2	12	9	197	181	4	8	12	-1	190	187	5
3	12	-10	269	273	4	8	12	3	143	141	6
3	12	-8	178	167	4	9	12	-2	180	153	5
3	12	-5	166	158	4	0	13	1	266	258	3
3	12	-4	244	247	3	0	13	2	171	168	4
3	12	-3	120	130	5	0	13	4	138	128	5
3	12	-2	143	139	4	0	13	7	175	168	5
3	12	0	108	98	5	0	13	9	130	120	6
3	12	2	94	87	6	1	13	-8	163	152	5
3	12	4	274	261	3	1	13	-6	127	131	6
3	12	5	101	100	7	1	13	-3	88	75	7
3	12	8	189	189	5	1	13	-2	289	284	3
3	12	10	168	151	5	1	13	-1	80	36	7
4	12	-9	90	77	8	1	13	0	219	209	4
4	12	-6	115	118	6	1	13	1	159	159	4
4	12	-5	200	196	4	1	13	3	125	119	5
4	12	-1	110	104	6	1	13	6	195	187	4
4	12	0	129	144	5	1	13	8	130	119	6
4	12	1	205	198	4	2	13	-9	82	84	9
4	12	3	221	211	4	2	13	-8	120	114	6
4	12	4	113	108	6	2	13	-7	137	132	6
4	12	5	154	141	5	2	13	-3	310	297	3
4	12	7	119	104	6	2	13	-1	172	156	4
4	12	9	239	214	4	2	13	0	128	119	5
5	12	-10	116	109	6	2	13	1	112	105	5
5	12	-9	74	70	9	2	13	5	123	120	6
5	12	-2	91	73	7	2	13	7	81	83	9
5	12	-1	199	192	4	3	13	-9	114	102	7
5	12	0	386	377	3	3	13	-5	77	57	9
5	12	1	79	50	8	3	13	-4	287	283	4
5	12	2	130	125	5	3	13	-2	172	172	4
5	12	4	221	217	4	3	13	-1	79	72	8
5	12	8	175	155	5	4	13	-7	109	104	7

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
4	13	-6	75	82	10						
4	13	-5	284	272	4						
4	13	-4	106	96	7						
4	13	-3	242	242	4						
4	13	1	145	146	5						
4	13	7	154	134	6						
5	13	-7	132	137	6						
5	13	-6	194	185	5						
5	13	-5	101	91	7						
5	13	-4	201	183	4						
5	13	-2	75	81	9						
6	13	-5	142	124	5						
6	13	1	95	98	8						
6	13	4	78	88	10						
7	13	-4	122	122	7						
7	13	0	127	122	6						
0	14	0	200	191	4						
0	14	2	135	133	6						
0	14	3	77	55	9						
0	14	5	199	192	5						
1	14	-6	197	182	5						
1	14	-3	86	92	8						
1	14	-1	148	130	5						
1	14	2	133	122	6						
1	14	3	119	121	7						
1	14	4	155	150	5						
1	14	5	79	68	9						
2	14	-6	101	99	7						
2	14	-5	80	64	9						
2	14	1	132	123	6						
2	14	2	183	172	5						
2	14	3	77	76	9						
2	14	4	86	82	9						
2	14	5	82	76	9						
3	14	-4	109	94	7						
3	14	0	165	155	5						
3	14	1	144	119	5						
3	14	4	105	103	7						
3	14	5	80	62	9						
4	14	-1	162	147	5						
4	14	0	88	72	8						
4	14	1	76	63	9						
4	14	3	127	113	6						
5	14	-3	72	51	10						
5	14	-2	130	129	6						
5	14	0	119	111	7						

Appendix 4

The Following is a List of Lecture Courses which I have attended during the period 1987-1990.

Dr J.A.Crayston	Cyclic Voltammetry.
Dr.C.Thompson	Carcinogenesis.
Dr.D.Lloyd	Aromaticity.
Dr.G.Harris	Vacuum Techniques.

Appendix 5

List of Abbreviations.

Ferrocene	Dicyclopentadienyliron(II)
Fc	$C_5H_5FeC_5H_4$-
Fcd	-$C_5H_4FeC_5H_4$-
OAc	Acetate, (-$OCOCH_3$)
Me	Methyl, (CH_3)
Ph	Phenyl, (C_6H_5)
FVP	Flash Vacuum Pyrolysis

Appendix 6

Crystallographic data for the low- melting form of ferrocenecarboxaldoxime.

Appendix 6.1

Calculated hydrogen coordinates (C–H 0.95 Å)

Atom	x	y	z	B(Å²)
H2	0.0676	−0.0022	0.4360	8
H3	−0.0206	0.1014	0.3949	8
H4	−0.0003	0.2876	0.3832	8
H5	0.0983	0.3078	0.4189	8
H11	0.1797	0.1845	0.7190	8
H12	0.1215	0.0286	0.7165	8
H13	0.0275	0.0969	0.6863	8
H14	0.0261	0.2856	0.6655	8
H15	0.1199	0.3426	0.6849	8
H22	0.1248	0.5557	0.7087	16
H23	0.2364	0.5759	0.7979	16
H24	0.2597	0.5533	0.6346	16
H25	0.1743	0.5553	0.4397	16
H31	0.0803	0.8001	0.5078	8
H32	0.1276	0.8094	0.7165	8
H33	0.2296	0.8204	0.7764	8
H34	0.2479	0.8202	0.6004	8
H35	0.1539	0.8033	0.4300	8
H6	0.1715	0.0417	0.4807	8

Appendix 6.2

General Temperature Factor Expressions - U's

Name	U(1,1)	U(2,2)	U(3,3)	U(1,2)	U(1,3)	U(2,3)
Fe1	0.0677(4)	0.0577(5)	0.0590(4)	-0.0039(5)	0.0280(3)	-0.0069(4
Fe2	0.0903(7)	0.0550(5)	0.0908(6)	0.0051(5)	0.0261(5)	0.0088(5
C1	0.083(4)	0.085(5)	0.061(3)	-0.003(4)	0.036(2)	-0.006(3)
C2	0.106(4)	0.064(4)	0.065(3)	-0.003(4)	0.032(3)	-0.020(3)
C3	0.071(4)	0.114(6)	0.078(4)	-0.002(4)	0.018(3)	-0.018(4)
C4	0.114(5)	0.094(5)	0.083(4)	0.031(5)	0.032(4)	0.013(4)
C5	0.127(5)	0.071(4)	0.082(3)	0.002(4)	0.059(3)	0.008(4)
C6	0.112(4)	0.078(4)	0.082(4)	0.004(4)	0.054(3)	-0.013(4)
C11	0.077(4)	0.124(6)	0.064(4)	-0.019(4)	0.022(3)	-0.016(4)
C12	0.146(6)	0.077(5)	0.061(4)	0.021(5)	0.038(3)	0.005(3)
C13	0.126(4)	0.149(7)	0.075(3)	-0.061(5)	0.060(3)	-0.027(4)
C14	0.106(5)	0.121(6)	0.092(4)	0.017(5)	0.045(3)	-0.020(5)
C15	0.157(6)	0.062(4)	0.075(4)	-0.032(4)	0.045(4)	-0.019(4)
C21	0.178(7)	0.052(4)	0.130(6)	-0.008(5)	0.067(5)	0.001(4
C22	0.249(9)	0.074(5)	0.241(8)	-0.048(5)	0.123(6)	0.036(5
C23	0.25(1)	0.074(6)	0.164(9)	0.030(7)	0.043(9)	0.038(6
C24	0.114(7)	0.079(5)	0.25(1)	0.032(5)	-0.073(8)	-0.054(7
C25	0.358(9)	0.072(6)	0.266(7)	0.043(6)	0.232(5)	0.003(5
C26	0.117(9)	0.037(7)	0.15(1)	-0.011(7)	0.076(7)	-0.000(8
C31	0.096(5)	0.072(5)	0.116(5)	0.027(4)	0.030(4)	0.019(4
C32	0.135(6)	0.097(6)	0.093(4)	0.006(5)	0.055(3)	-0.005(4
C33	0.136(6)	0.068(4)	0.071(4)	-0.017(4)	0.029(4)	-0.011(3
C34	0.116(5)	0.078(5)	0.108(5)	-0.003(4)	0.055(3)	0.014(4
C35	0.195(7)	0.070(5)	0.057(3)	0.016(5)	0.049(4)	0.016(3
O1	0.100(4)	0.102(6)	0.152(5)	0.018(5)	0.082(3)	-0.017(5

Appendix 6.3 Torsion Angles

Atom 1	Atom 2	Atom 3	Atom 4	Angle
C5	C1	C2	C3	-0.4 (0.7)
C6	C1	C2	C3	175.6 (0.6)
C2	C1	C5	C4	0.1 (0.7)
C6	C1	C5	C4	-175.3 (0.7)
C2	C1	C6	N1	-177.8 (0.6)
C5	C1	C6	N1	-3.0 (1.1)
C1	C2	C3	C4	0.6 (0.8)
C2	C3	C4	C5	-0.6 (0.8)
C3	C4	C5	C1	0.3 (0.8)
C1	C6	N1	O1	177.9 (0.6)
C1	C6	N1	O1*	6.6 (1.0)
C15	C11	C12	C13	-0.9 (0.7)
C12	C11	C15	C14	0.7 (0.8)
C11	C12	C13	C14	0.9 (0.8)
C12	C13	C14	C15	-0.5 (0.8)
C13	C14	C15	C11	-0.1 (0.8)
C25	C21	C22	C23	-2.9 (1.0)
C26	C21	C22	C23	-176.8 (0.8)
C22	C21	C25	C24	6.1 (1.0)
C26	C21	C25	C24	179.1 (0.9)
C22	C21	C26	N2	-171.0 (1.2)
C22	C21	C26	N2*	-9.8 (1.7)
C25	C21	C26	N2	15.8 (1.8)
C25	C21	C26	N2*	177.0 (1.6)
C21	C22	C23	C24	-2.2 (1.0)
C22	C23	C24	C25	6.5 (1.1)
C23	C24	C25	C21	-7.9 (1.1)
C21	C26	N2	O2	-3.4 (2.8)
N2*	C26	N2	O2	-153.4 (3.1)
C21	C26	N2*	O2*	-165.9 (1.6)
N2	C26	N2*	O2*	-11.7 (4.2)
C35	C31	C32	C33	0.4 (0.9)
C32	C31	C35	C34	-0.9 (0.9)
C31	C32	C33	C34	0.4 (0.9)
C32	C33	C34	C35	-0.9 (0.9)
C33	C34	C35	C31	1.1 (0.8)

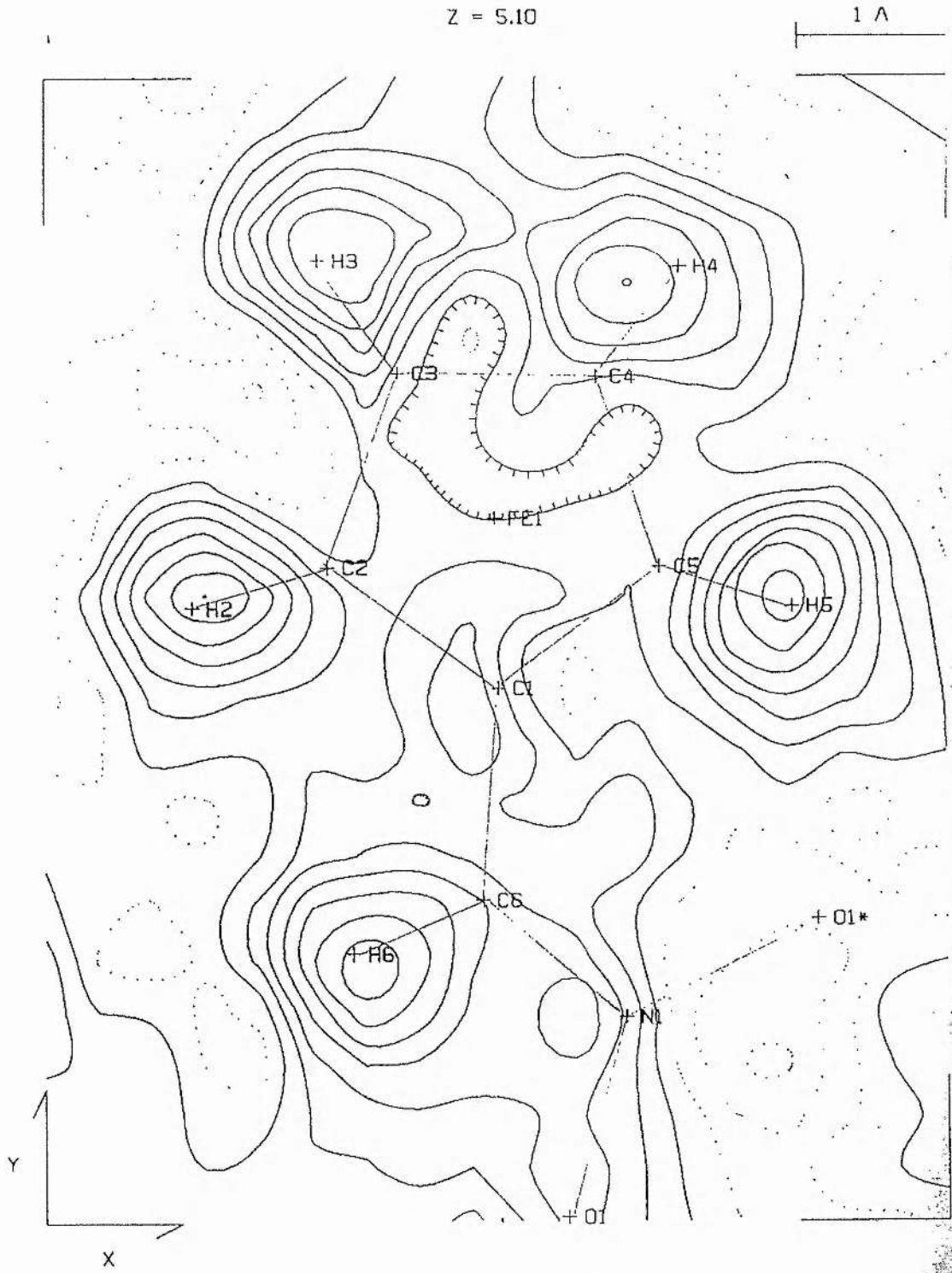
General Temperature Factor Expressions - U's (Continued)

Name	U(1,1)	U(2,2)	U(3,3)	U(1,2)	U(1,3)	U(2,3)
O1*	0.112(7)	0.090(8)	0.123(8)	-0.023(7)	0.062(5)	0.007(7)
N1	0.100(3)	0.092(4)	0.091(3)	-0.016(3)	0.061(2)	-0.011(3)
O2	0.12(1)	0.052(7)	0.23(1)	0.008(8)	0.076(9)	0.069(8)
N2	0.17(1)	0.021(6)	0.042(7)	-0.007(8)	0.031(8)	-0.008(6)
O2*	0.08(2)	0.05(2)	0.08(2)	0.04(2)	0.06(1)	0.01(2)
N2*	0.04(2)	0.03(2)	0.17(4)	0.03(2)	-0.03(2)	-0.01(2)

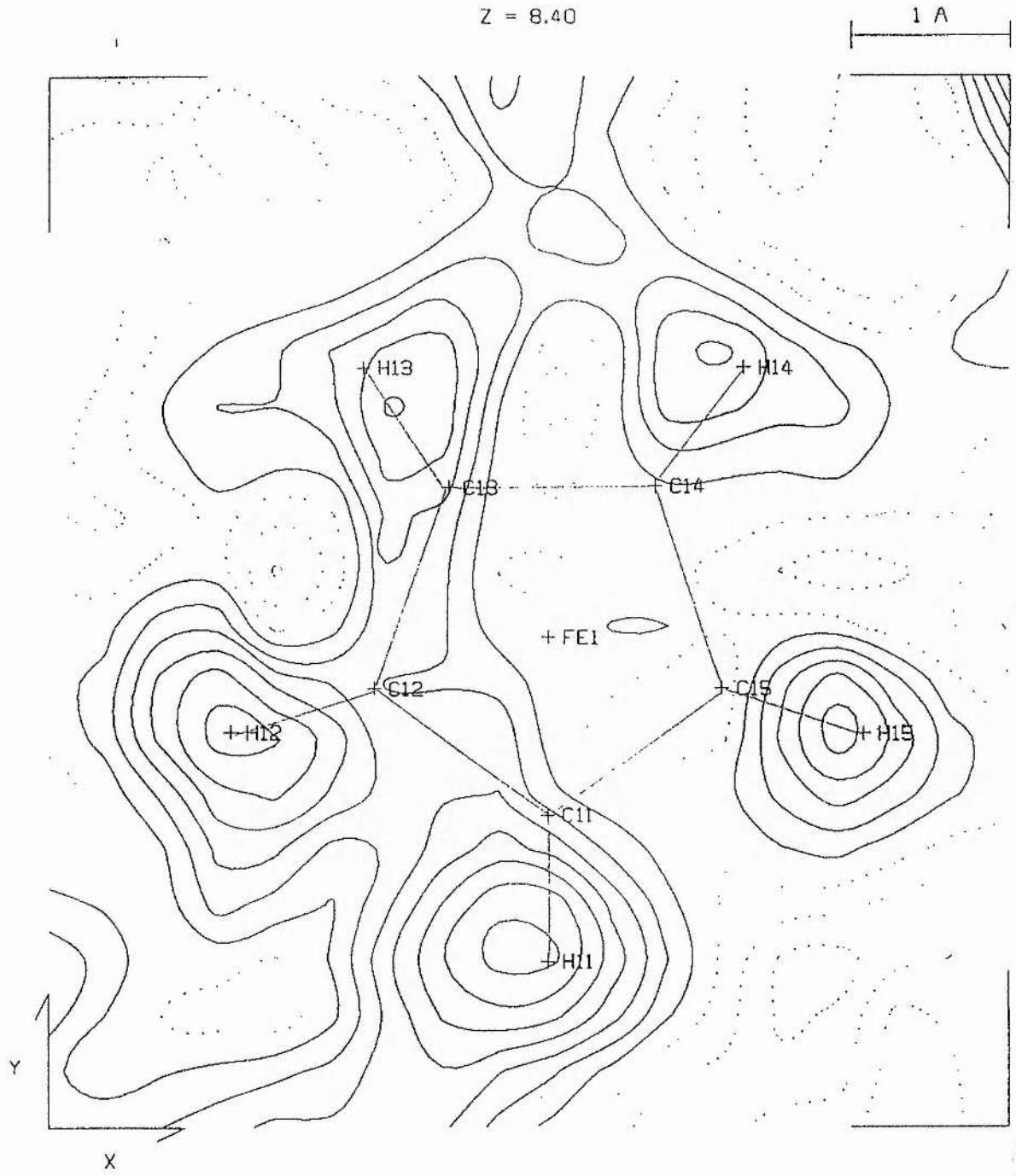
The form of the anisotropic thermal parameter is:

exp[-2PI2{h2a2U(1,1) + k2b2U(2,2) + l2c2U(3,3) + 2hkabU(1,2) + 2hlacU(1,3 + 2klbcU(2,3)}] where a, b, and c are reciprocal lattice constants.

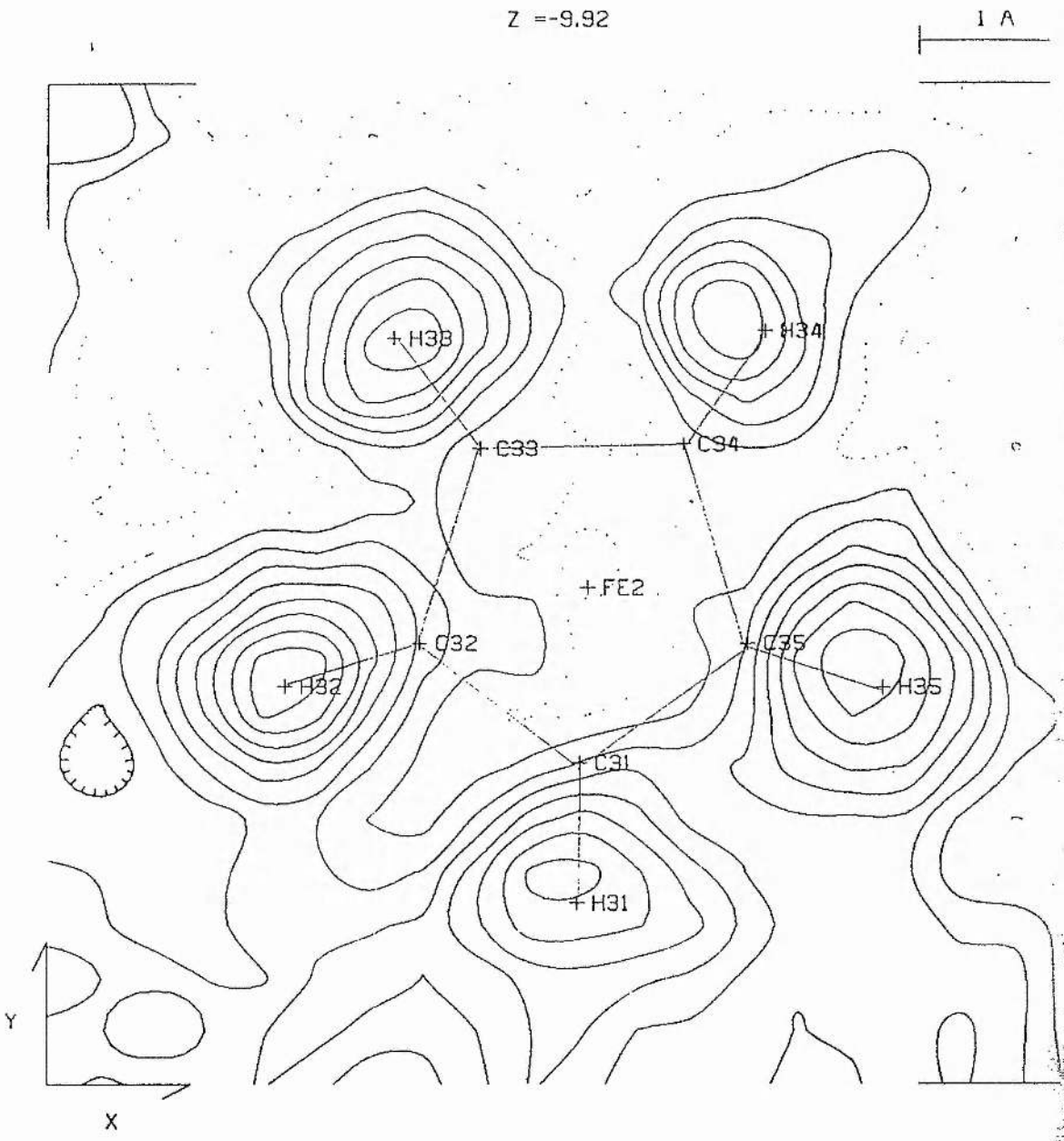
Appendix 6.4.1 Difference Map in C1-C5 Plane



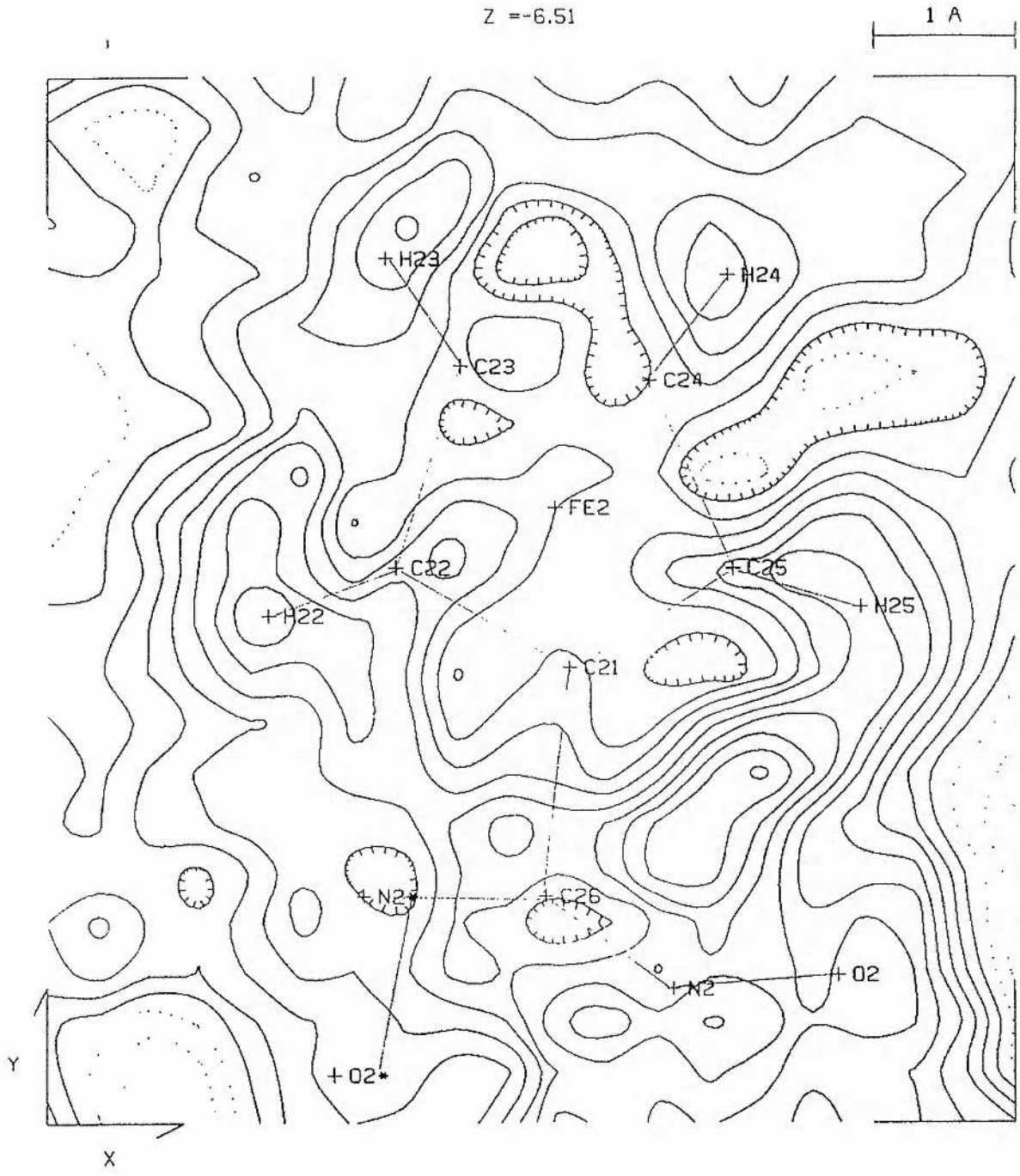
Appendix 6.4.2 Difference Map in C11-C15 Plane



Appendix 6.4.3 Difference Map in C31-C35 Plane



Appendix 6.4.4 Difference Map in C21-C25 Plane



Appendix 6.5 Observed and Calculated Structure Factors.

10Fo, 10Fc, 10sig(Fo) for (Fc)CH=NOH 90-21

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
4	0	-14	220	225	16	18	0	-4	224	228	9
6	0	-14	247	242	14	20	0	-4	173	190	12
8	0	-14	207	217	15	22	0	-4	879	909	6
10	0	-14	238	273	14	24	0	-4	223	257	14
16	0	-14	220	248	15	30	0	-4	181	141	19
18	0	-14	253	283	13	2	0	-2	3932	4038	17
4	0	-12	174	159	15	4	0	-2	2703	2525	13
8	0	-12	331	334	9	6	0	-2	466	414	3
16	0	-12	604	609	7	8	0	-2	1288	1344	6
20	0	-12	276	316	12	10	0	-2	1021	1058	5
22	0	-12	229	218	14	12	0	-2	997	1050	5
24	0	-12	237	215	14	14	0	-2	471	474	5
28	0	-12	177	171	20	18	0	-2	135	147	16
2	0	-10	348	367	7	20	0	-2	473	431	7
6	0	-10	600	653	6	22	0	-2	769	774	7
8	0	-10	260	294	9	24	0	-2	245	220	13
14	0	-10	409	404	7	4	0	0	2752	2660	6
16	0	-10	390	375	7	8	0	0	2512	2320	8
18	0	-10	372	397	8	10	0	0	239	234	6
20	0	-10	254	291	11	12	0	0	1416	1388	7
26	0	-10	360	374	10	14	0	0	503	499	6
2	0	-8	1145	1197	6	16	0	0	355	350	7
4	0	-8	1829	1826	9	20	0	0	684	658	6
6	0	-8	1617	1601	8	22	0	0	226	245	13
8	0	-8	159	170	11	0	0	2	3165	3014	16
12	0	-8	340	368	6	2	0	2	3199	3301	16
14	0	-8	819	814	5	4	0	2	1359	1306	7
16	0	-8	263	257	8	6	0	2	632	581	3
18	0	-8	511	529	6	8	0	2	1112	1116	6
22	0	-8	213	245	13	10	0	2	1192	1195	6
26	0	-8	321	362	11	12	0	2	1351	1391	7
4	0	-6	2179	2193	9	16	0	2	304	304	9
6	0	-6	244	233	5	18	0	2	533	464	7
8	0	-6	362	355	4	20	0	2	375	343	9
10	0	-6	200	195	6	22	0	2	188	187	16
12	0	-6	1415	1391	7	26	0	2	165	116	22
14	0	-6	465	453	5	0	0	4	387	388	4
16	0	-6	1146	1176	6	2	0	4	320	311	4
18	0	-6	308	342	7	4	0	4	692	735	4
20	0	-6	499	504	6	6	0	4	896	953	5
24	0	-6	373	412	9	8	0	4	678	721	5
26	0	-6	154	137	19	10	0	4	1067	1052	5
2	0	-4	1438	1372	7	16	0	4	141	124	18
4	0	-4	222	230	4	18	0	4	472	534	9
6	0	-4	1030	1044	5	20	0	4	243	246	14
8	0	-4	324	336	4	22	0	4	196	205	19
10	0	-4	1396	1298	7	0	0	6	774	756	4
12	0	-4	858	903	4	2	0	6	1019	1116	5
14	0	-4	1083	1086	5	4	0	6	1374	1443	7
16	0	-4	733	745	5	6	0	6	378	389	6

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
8	0	6	812	813	5	13	1	-9	377	400	7
10	0	6	226	223	10	15	1	-9	115	141	18
16	0	6	418	468	10	19	1	-9	342	339	8
20	0	6	224	206	17	21	1	-9	343	349	9
0	0	8	722	760	5	23	1	-9	216	245	14
2	0	8	631	636	5	1	1	-8	600	560	5
4	0	8	325	347	7	5	1	-8	482	513	6
6	0	8	898	908	6	7	1	-8	615	643	5
8	0	8	206	225	13	9	1	-8	280	292	7
14	0	8	335	302	11	11	1	-8	559	573	5
16	0	8	222	208	17	13	1	-8	143	114	13
0	0	10	152	153	14	19	1	-8	280	278	9
2	0	10	362	345	8	1	1	-7	1658	1676	8
4	0	10	317	311	10	3	1	-7	1365	1437	7
6	0	10	515	521	8	5	1	-7	110	145	13
0	0	12	208	222	14	7	1	-7	801	802	4
4	0	12	346	303	11	9	1	-7	1233	1262	6
3	1	-15	182	150	22	11	1	-7	275	274	7
7	1	-15	201	247	20	13	1	-7	694	726	5
11	1	-14	145	105	20	15	1	-7	395	406	6
1	1	-13	307	262	11	17	1	-7	683	655	5
9	1	-13	225	241	13	21	1	-7	620	661	6
11	1	-13	201	183	14	25	1	-7	194	200	15
13	1	-13	412	408	9	29	1	-7	230	218	15
19	1	-13	222	209	15	5	1	-6	643	716	4
21	1	-13	260	248	13	7	1	-6	301	330	5
25	1	-13	185	193	18	9	1	-6	602	572	4
3	1	-11	293	296	10	11	1	-6	693	656	4
5	1	-11	317	327	9	15	1	-6	164	161	10
7	1	-11	294	276	9	17	1	-6	357	345	7
11	1	-11	685	704	6	19	1	-6	374	385	7
13	1	-11	159	178	16	1	1	-5	814	756	4
15	1	-11	237	249	11	3	1	-5	577	590	4
19	1	-11	293	269	10	5	1	-5	336	358	4
23	1	-11	284	281	12	7	1	-5	1109	1134	5
25	1	-11	235	249	15	9	1	-5	690	724	4
1	1	-10	212	202	11	11	1	-5	831	808	4
3	1	-10	139	114	15	13	1	-5	934	910	5
5	1	-10	257	235	9	15	1	-5	1138	1171	6
9	1	-10	373	384	7	19	1	-5	782	829	5
11	1	-10	181	158	12	21	1	-5	345	338	8
13	1	-10	324	326	8	27	1	-5	348	347	11
19	1	-10	134	119	18	1	1	-4	124	128	7
21	1	-10	240	236	11	3	1	-4	1259	1266	6
1	1	-9	564	532	6	5	1	-4	553	575	3
3	1	-9	799	805	5	7	1	-4	469	430	3
5	1	-9	259	271	9	9	1	-4	962	1012	5
7	1	-9	165	193	12	15	1	-4	140	125	11
9	1	-9	809	829	5	17	1	-4	413	425	6
11	1	-9	723	734	5	21	1	-4	356	364	8

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
1	1	-3	3015	2995	6	21	1	1	133	133	21
3	1	-3	2899	2929	6	23	1	1	275	263	12
5	1	-3	1239	1185	6	1	1	2	1325	1372	6
7	1	-3	2147	2090	7	3	1	2	600	576	3
9	1	-3	1355	1291	7	5	1	2	948	967	5
11	1	-3	317	301	5	9	1	2	199	170	7
13	1	-3	485	510	4	11	1	2	343	359	6
15	1	-3	582	621	5	13	1	2	680	651	6
17	1	-3	861	873	5	17	1	2	323	331	9
19	1	-3	436	456	7	19	1	2	147	90	18
25	1	-3	286	261	12	1	1	3	89	115	9
27	1	-3	246	225	13	3	1	3	556	607	3
1	1	-2	348	397	2	5	1	3	1201	1282	6
3	1	-2	1017	907	5	7	1	3	646	670	4
5	1	-2	1063	943	5	9	1	3	768	775	4
7	1	-2	1662	1588	7	11	1	3	534	574	6
9	1	-2	501	451	4	13	1	3	746	755	6
11	1	-2	195	204	6	15	1	3	717	730	6
15	1	-2	368	388	6	17	1	3	203	205	13
17	1	-2	117	129	16	21	1	3	174	149	17
19	1	-2	247	277	10	23	1	3	179	186	18
21	1	-2	277	253	10	1	1	4	733	730	4
23	1	-2	211	153	13	3	1	4	581	625	4
1	1	-1	63	90	6	5	1	4	293	301	5
3	1	-1	703	808	3	7	1	4	315	314	5
5	1	-1	5761	5647	24	11	1	4	245	294	9
7	1	-1	520	480	3	15	1	4	359	350	9
9	1	-1	1165	1100	6	23	1	4	154	153	23
11	1	-1	212	222	7	1	1	5	1514	1598	7
13	1	-1	455	451	5	3	1	5	357	406	5
15	1	-1	244	238	8	5	1	5	898	961	5
17	1	-1	957	986	5	9	1	5	516	545	6
25	1	-1	312	277	11	13	1	5	635	669	7
3	1	0	1026	1308	5	15	1	5	134	138	20
5	1	0	1479	1395	7	17	1	5	230	229	14
7	1	0	596	599	4	21	1	5	172	187	21
9	1	0	323	272	5	1	1	6	781	774	4
11	1	0	226	224	6	3	1	6	730	737	4
13	1	0	249	256	7	5	1	6	179	219	10
15	1	0	465	481	6	7	1	6	172	174	11
17	1	0	300	309	8	11	1	6	134	162	18
1	1	1	231	243	2	13	1	6	226	193	12
3	1	1	3953	4077	20	15	1	6	163	184	18
5	1	1	737	717	4	1	1	7	450	480	5
7	1	1	1553	1426	7	3	1	7	988	1026	5
9	1	1	895	920	4	5	1	7	149	188	12
11	1	1	669	669	5	7	1	7	196	203	11
13	1	1	124	147	14	11	1	7	585	593	7
15	1	1	1108	1094	6	13	1	7	149	154	20
17	1	1	535	525	7	1	1	8	585	602	6

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
7	1	8	121	150	19	4	2	-8	866	885	5
9	1	8	227	190	13	6	2	-8	789	828	5
13	1	8	149	171	21	12	2	-8	268	271	8
1	1	9	589	616	6	14	2	-8	322	272	7
3	1	9	229	229	10	18	2	-8	281	289	9
7	1	9	202	205	14	26	2	-8	220	216	15
9	1	9	419	423	9	2	2	-7	1692	1697	9
11	1	9	337	335	11	4	2	-7	1310	1357	7
7	1	10	179	156	16	6	2	-7	1208	1266	6
1	1	11	346	325	9	8	2	-7	240	238	7
9	1	11	215	243	17	10	2	-7	448	491	5
1	1	13	188	165	17	12	2	-7	491	484	5
8	2	-15	295	269	14	14	2	-7	1037	969	5
4	2	-14	154	135	21	16	2	-7	573	568	6
8	2	-14	165	135	19	18	2	-7	125	128	16
10	2	-14	162	141	19	28	2	-7	165	138	19
16	2	-14	200	167	16	4	2	-6	1359	1419	7
18	2	-14	155	180	21	8	2	-6	297	241	5
6	2	-13	212	151	14	10	2	-6	284	262	6
8	2	-13	230	255	13	12	2	-6	1283	1314	7
12	2	-13	158	211	19	16	2	-6	740	755	5
14	2	-13	254	237	13	18	2	-6	269	258	8
18	2	-13	230	230	15	20	2	-6	428	399	7
20	2	-13	192	186	16	24	2	-6	368	382	9
4	2	-12	195	218	15	32	2	-6	156	65	24
8	2	-12	227	223	12	2	2	-5	1092	1057	6
16	2	-12	339	364	10	4	2	-5	427	410	4
6	2	-11	159	183	16	6	2	-5	1038	1009	5
10	2	-11	293	322	10	8	2	-5	141	126	8
12	2	-11	227	206	12	10	2	-5	821	816	4
14	2	-11	291	269	10	12	2	-5	898	892	4
16	2	-11	297	285	10	14	2	-5	1388	1404	7
18	2	-11	297	315	11	18	2	-5	185	193	12
24	2	-11	334	299	11	20	2	-5	377	355	7
26	2	-11	192	188	18	22	2	-5	252	238	10
2	2	-10	273	247	9	26	2	-5	230	253	13
4	2	-10	177	178	13	28	2	-5	148	100	23
6	2	-10	453	416	7	2	2	-4	143	145	6
8	2	-10	254	255	9	4	2	-4	358	352	4
10	2	-10	135	148	15	6	2	-4	648	644	3
16	2	-10	249	255	9	8	2	-4	83	101	12
26	2	-10	169	182	18	10	2	-4	812	875	4
4	2	-9	1007	1041	5	12	2	-4	874	874	4
8	2	-9	459	487	6	14	2	-4	863	855	4
10	2	-9	171	175	11	16	2	-4	186	189	9
12	2	-9	431	445	6	18	2	-4	119	115	16
16	2	-9	574	591	6	20	2	-4	348	338	8
24	2	-9	329	317	10	22	2	-4	452	419	8
28	2	-9	183	152	17	24	2	-4	203	230	15
2	2	-8	516	487	6	2	2	-3	570	597	3

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
4	2	-3	900	858	5	0	2	2	1048	940	5
6	2	-3	439	405	3	2	2	2	1850	1802	7
8	2	-3	553	542	3	6	2	2	742	837	4
12	2	-3	1530	1486	7	8	2	2	772	804	4
14	2	-3	156	150	10	10	2	2	1506	1506	8
16	2	-3	513	530	6	12	2	2	607	577	6
18	2	-3	119	120	17	14	2	2	129	108	17
20	2	-3	619	633	7	18	2	2	361	379	9
24	2	-3	445	435	9	20	2	2	253	286	12
2	2	-2	1859	1766	6	0	2	3	374	373	3
6	2	-2	183	173	5	2	2	3	411	384	4
8	2	-2	463	401	4	4	2	3	80	60	12
10	2	-2	817	763	4	8	2	3	1528	1671	8
12	2	-2	234	217	6	10	2	3	317	333	7
14	2	-2	186	193	8	12	2	3	696	699	6
16	2	-2	279	242	8	16	2	3	434	430	8
18	2	-2	158	166	14	20	2	3	337	343	11
20	2	-2	186	169	14	0	2	4	678	767	3
22	2	-2	396	378	9	4	2	4	168	162	7
2	2	-1	3970	3626	19	6	2	4	767	830	4
4	2	-1	1793	1432	6	8	2	4	511	519	5
6	2	-1	949	973	5	10	2	4	586	641	6
8	2	-1	765	787	4	12	2	4	154	157	14
10	2	-1	1273	1288	6	18	2	4	355	303	11
12	2	-1	717	735	5	0	2	5	369	383	4
14	2	-1	562	590	5	4	2	5	218	216	7
16	2	-1	160	171	12	6	2	5	930	1006	5
18	2	-1	364	401	8	8	2	5	512	542	6
20	2	-1	428	453	8	10	2	5	402	426	7
22	2	-1	369	352	9	18	2	5	383	399	11
0	2	0	3162	3897	16	20	2	5	172	151	21
2	2	0	552	193	3	0	2	6	372	381	5
4	2	0	1305	1121	6	2	2	6	331	293	6
6	2	0	344	323	5	4	2	6	875	883	5
8	2	0	1068	1131	5	8	2	6	486	499	6
10	2	0	413	418	5	16	2	6	259	232	13
12	2	0	818	794	5	4	2	7	382	401	7
18	2	0	167	142	13	6	2	7	621	625	6
20	2	0	363	380	8	8	2	7	244	219	10
0	2	1	1777	1681	5	10	2	7	225	239	12
2	2	1	2920	2684	14	14	2	7	194	177	16
4	2	1	120	125	8	0	2	8	270	261	8
6	2	1	911	901	5	2	2	8	369	404	7
8	2	1	705	697	4	4	2	8	341	337	8
10	2	1	1885	1864	9	6	2	8	470	527	7
12	2	1	422	406	6	8	2	8	176	118	14
14	2	1	388	405	7	14	2	8	185	193	18
16	2	1	268	280	9	4	2	9	459	434	7
18	2	1	499	480	7	8	2	9	319	321	10
22	2	1	188	183	15	10	2	9	198	208	16

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
2	2	10	197	221	13	17	3	-7	177	174	12
6	2	10	316	302	11	21	3	-7	298	248	9
2	2	11	256	268	12	1	3	-6	1027	1023	5
4	2	11	179	187	18	3	3	-6	277	272	6
4	2	12	199	184	16	5	3	-6	890	861	5
0	2	13	187	199	18	7	3	-6	1294	1310	7
2	2	13	198	157	18	9	3	-6	1376	1403	7
3	3	-14	253	254	17	11	3	-6	1198	1185	6
5	3	-14	178	188	20	15	3	-6	155	131	12
9	3	-14	153	165	21	17	3	-6	746	760	5
11	3	-14	287	229	13	19	3	-6	758	754	6
13	3	-14	154	134	20	21	3	-6	291	290	9
17	3	-14	196	216	18	25	3	-6	143	130	20
19	3	-14	148	89	23	1	3	-5	96	82	13
27	3	-13	154	50	23	5	3	-5	443	459	4
1	3	-12	334	286	10	7	3	-5	409	418	4
3	3	-12	280	299	12	13	3	-5	383	383	6
9	3	-12	304	283	10	15	3	-5	163	176	11
11	3	-12	307	316	11	19	3	-5	248	220	10
13	3	-12	223	193	13	1	3	-4	624	624	4
15	3	-12	373	386	10	3	3	-4	449	456	4
17	3	-12	268	237	11	5	3	-4	2001	1943	8
21	3	-12	260	253	13	7	3	-4	998	987	5
23	3	-12	277	280	13	9	3	-4	2310	2172	9
1	3	-10	568	556	7	13	3	-4	131	119	12
5	3	-10	316	314	8	15	3	-4	291	280	7
9	3	-10	629	612	6	17	3	-4	1116	1140	5
13	3	-10	358	359	8	21	3	-4	562	599	7
17	3	-10	234	216	12	23	3	-4	192	224	15
19	3	-10	264	271	11	29	3	-4	261	239	15
21	3	-10	358	370	10	1	3	-3	208	247	5
27	3	-10	207	179	16	3	3	-3	410	393	4
11	3	-9	139	113	14	5	3	-3	220	195	5
13	3	-9	185	173	11	7	3	-3	377	289	4
1	3	-8	520	556	6	9	3	-3	750	747	4
3	3	-8	713	695	5	11	3	-3	345	350	5
5	3	-8	350	348	7	13	3	-3	167	175	9
7	3	-8	791	830	5	15	3	-3	218	206	8
9	3	-8	493	511	6	17	3	-3	160	131	13
11	3	-8	1048	1038	5	1	3	-2	1542	1516	7
15	3	-8	166	175	12	3	3	-2	691	816	3
19	3	-8	734	716	6	5	3	-2	229	260	5
21	3	-8	127	114	19	7	3	-2	2222	2211	8
31	3	-8	170	136	21	9	3	-2	89	88	12
1	3	-7	454	485	6	11	3	-2	455	409	5
3	3	-7	286	287	7	13	3	-2	808	803	5
5	3	-7	189	195	9	15	3	-2	1133	1136	6
9	3	-7	301	298	6	17	3	-2	364	345	8
11	3	-7	134	162	13	19	3	-2	550	562	7
13	3	-7	268	258	8	21	3	-2	332	359	9

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
27	3	-2	233	233	14	3	3	4	2570	2521	9
1	3	-1	88	130	9	5	3	4	712	711	4
3	3	-1	811	559	4	7	3	4	766	785	5
5	3	-1	700	588	3	11	3	4	961	971	6
7	3	-1	117	123	8	13	3	4	255	230	10
9	3	-1	465	458	4	15	3	4	402	427	9
11	3	-1	593	603	5	23	3	4	249	232	17
13	3	-1	545	542	6	1	3	5	421	430	5
17	3	-1	202	217	11	3	3	5	186	189	8
19	3	-1	178	175	14	5	3	5	141	157	11
21	3	-1	160	145	17	7	3	5	238	229	9
1	3	0	4433	3553	23	9	3	5	355	339	7
3	3	0	148	170	7	1	3	6	1249	1331	6
5	3	0	1336	1316	7	3	3	6	1410	1440	7
7	3	0	1751	1660	8	5	3	6	584	559	5
9	3	0	650	639	4	9	3	6	117	112	18
11	3	0	624	647	5	11	3	6	480	463	7
13	3	0	867	886	5	13	3	6	254	259	12
15	3	0	1213	1225	6	15	3	6	214	207	15
17	3	0	695	688	6	1	3	7	246	244	8
19	3	0	202	177	12	9	3	7	171	179	14
21	3	0	172	161	16	1	3	8	1021	1011	5
23	3	0	219	205	14	5	3	8	357	310	8
25	3	0	227	176	15	9	3	8	341	353	9
1	3	1	264	187	4	13	3	8	336	320	12
3	3	1	402	353	4	3	3	10	219	213	13
5	3	1	591	574	4	5	3	10	249	272	12
7	3	1	202	208	7	7	3	10	318	311	11
11	3	1	175	196	11	11	3	10	275	253	14
13	3	1	152	166	13	8	4	-15	194	212	22
15	3	1	325	337	8	16	4	-15	167	118	23
1	3	2	1104	1083	6	6	4	-13	287	239	12
3	3	2	544	573	4	8	4	-13	310	311	12
5	3	2	2205	2197	8	12	4	-13	250	239	14
7	3	2	431	478	5	14	4	-13	299	252	12
9	3	2	289	312	6	18	4	-13	287	302	13
11	3	2	544	534	6	20	4	-13	148	156	22
13	3	2	1344	1406	7	6	4	-11	558	499	7
15	3	2	277	253	10	10	4	-11	467	473	8
17	3	2	522	505	7	12	4	-11	203	161	13
21	3	2	225	201	14	14	4	-11	190	200	15
25	3	2	289	250	14	16	4	-11	202	224	14
1	3	3	166	164	6	18	4	-11	299	338	11
3	3	3	320	296	4	24	4	-11	219	219	15
5	3	3	545	542	4	26	4	-11	225	189	15
7	3	3	108	128	13	8	4	-10	159	104	15
9	3	3	191	200	10	14	4	-10	216	218	12
11	3	3	191	174	10	20	4	-10	149	141	19
13	3	3	224	230	11	22	4	-10	139	133	21
1	3	4	970	967	5	26	4	-10	151	158	21

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
4	4	-9	811	805	6	6	4	-3	501	429	4
6	4	-9	118	125	17	8	4	-3	514	530	4
8	4	-9	687	680	6	10	4	-3	527	510	4
12	4	-9	337	306	8	12	4	-3	1916	1845	9
16	4	-9	453	452	7	14	4	-3	179	183	10
18	4	-9	157	145	16	16	4	-3	539	539	6
22	4	-9	150	172	19	18	4	-3	186	178	12
24	4	-9	338	302	10	20	4	-3	643	626	7
2	4	-8	498	464	6	24	4	-3	430	399	9
4	4	-8	119	143	16	2	4	-2	225	177	5
6	4	-8	188	179	11	4	4	-2	772	620	4
14	4	-8	231	214	10	8	4	-2	159	136	7
2	4	-7	1098	1120	6	10	4	-2	660	644	4
4	4	-7	1012	1009	5	12	4	-2	181	188	9
6	4	-7	1282	1304	6	14	4	-2	120	133	16
8	4	-7	131	165	13	16	4	-2	149	185	15
10	4	-7	631	638	5	22	4	-2	259	272	12
12	4	-7	470	440	6	2	4	-1	736	476	4
14	4	-7	1209	1241	6	4	4	-1	1028	1148	5
16	4	-7	452	437	7	6	4	-1	205	249	6
20	4	-7	210	256	13	8	4	-1	994	1000	5
22	4	-7	290	278	11	10	4	-1	1152	1117	6
24	4	-7	220	195	13	12	4	-1	812	803	5
26	4	-7	154	141	19	14	4	-1	755	736	6
2	4	-6	221	199	8	18	4	-1	426	375	8
4	4	-6	567	568	5	20	4	-1	277	261	10
8	4	-6	198	180	9	22	4	-1	399	422	9
10	4	-6	493	462	5	2	4	0	878	536	4
12	4	-6	330	294	7	8	4	0	182	168	8
14	4	-6	163	147	12	12	4	0	499	492	6
16	4	-6	296	293	8	0	4	1	207	216	5
2	4	-5	2127	2168	9	2	4	1	992	1061	5
4	4	-5	1082	1062	6	4	4	1	222	231	6
6	4	-5	1330	1258	7	6	4	1	1065	1087	5
10	4	-5	1321	1311	7	8	4	1	1304	1302	6
12	4	-5	1389	1329	7	10	4	1	2002	1997	10
14	4	-5	1637	1629	9	12	4	1	1040	1028	5
16	4	-5	385	398	7	14	4	1	316	323	8
20	4	-5	270	274	10	16	4	1	428	384	7
22	4	-5	463	428	8	18	4	1	400	402	8
26	4	-5	224	237	15	20	4	1	259	265	12
4	4	-4	527	528	4	22	4	1	170	252	19
6	4	-4	92	90	13	0	4	2	540	543	4
8	4	-4	224	234	7	2	4	2	446	442	4
10	4	-4	712	733	4	4	4	2	431	387	4
12	4	-4	169	170	10	6	4	2	610	591	4
16	4	-4	225	233	10	10	4	2	408	387	7
22	4	-4	261	267	12	12	4	2	386	370	8
2	4	-3	295	273	5	0	4	3	1996	1935	8
4	4	-3	1670	1553	8	2	4	3	280	254	5

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
4	4	3	228	212	6	11	5	-11	358	349	9
6	4	3	144	137	10	13	5	-11	205	225	14
8	4	3	2427	2491	11	17	5	-11	171	129	15
10	4	3	122	147	17	19	5	-11	234	214	13
12	4	3	738	773	6	25	5	-11	172	185	20
16	4	3	588	555	8	1	5	-10	450	441	8
20	4	3	355	342	11	5	5	-10	243	244	11
0	4	4	337	310	5	9	5	-10	332	308	9
2	4	4	229	225	7	11	5	-10	239	223	11
4	4	4	214	201	7	13	5	-10	298	288	9
6	4	4	366	429	6	17	5	-10	239	268	12
10	4	4	185	206	12	19	5	-10	175	170	16
16	4	4	171	166	19	21	5	-10	242	231	13
0	4	5	625	604	4	1	5	-9	216	207	11
2	4	5	362	364	6	3	5	-9	289	294	9
4	4	5	548	530	5	7	5	-9	212	191	10
6	4	5	911	874	5	9	5	-9	183	196	12
8	4	5	746	755	6	11	5	-9	229	237	10
10	4	5	517	509	7	17	5	-9	218	203	12
14	4	5	291	268	11	19	5	-9	204	203	13
16	4	5	209	181	16	23	5	-9	193	194	15
18	4	5	335	308	13	3	5	-8	506	556	6
20	4	5	154	180	23	5	5	-8	247	224	9
0	4	6	340	340	6	7	5	-8	415	433	6
8	4	6	206	185	12	9	5	-8	331	316	7
0	4	7	321	314	7	11	5	-8	691	699	6
2	4	7	261	279	8	15	5	-8	220	144	10
4	4	7	142	175	14	19	5	-8	530	525	7
6	4	7	711	691	6	23	5	-8	204	223	14
10	4	7	274	227	11	27	5	-8	142	99	22
0	4	8	148	195	15	1	5	-7	422	432	6
4	4	9	475	538	8	3	5	-7	183	188	10
8	4	9	356	349	10	7	5	-7	233	210	8
2	4	11	319	331	11	9	5	-7	658	643	5
4	4	11	214	206	15	13	5	-7	330	302	7
6	4	11	203	222	17	15	5	-7	292	285	8
0	4	13	158	175	23	17	5	-7	265	263	9
2	4	13	208	179	18	21	5	-7	297	264	10
23	5	-14	172	130	22	27	5	-7	144	80	20
1	5	-13	203	161	18	1	5	-6	785	812	5
7	5	-13	152	139	21	5	5	-6	111	122	15
13	5	-13	277	289	13	7	5	-6	1173	1179	6
19	5	-13	169	123	18	9	5	-6	892	901	5
1	5	-12	192	150	16	11	5	-6	804	823	5
3	5	-12	304	296	11	13	5	-6	131	123	14
9	5	-12	154	141	19	17	5	-6	392	390	7
11	5	-12	179	145	16	19	5	-6	508	488	7
13	5	-12	138	171	21	1	5	-5	230	214	7
3	5	-11	324	314	10	3	5	-5	339	317	6
7	5	-11	185	215	15	5	5	-5	218	223	7

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
7	5	-5	1359	1386	7	11	5	0	671	701	6
9	5	-5	521	515	5	13	5	0	541	518	6
11	5	-5	662	669	5	15	5	0	755	765	6
13	5	-5	422	385	6	17	5	0	575	583	7
15	5	-5	493	505	6	19	5	0	204	233	13
19	5	-5	484	482	7	25	5	0	170	156	19
21	5	-5	146	168	18	1	5	1	1019	854	5
27	5	-5	211	194	16	3	5	1	1021	933	5
1	5	-4	853	782	4	5	5	1	392	371	5
3	5	-4	248	241	6	7	5	1	1100	1076	6
5	5	-4	1399	1357	7	9	5	1	246	237	8
7	5	-4	636	664	4	11	5	1	416	431	7
9	5	-4	1470	1432	8	15	5	1	568	603	7
11	5	-4	315	293	6	1	5	2	739	747	4
17	5	-4	586	590	6	3	5	2	361	319	5
21	5	-4	194	198	14	5	5	2	1837	1813	8
1	5	-3	248	212	5	9	5	2	195	162	10
3	5	-3	343	319	5	11	5	2	196	213	12
5	5	-3	1175	1184	6	13	5	2	887	883	6
7	5	-3	1017	1029	5	15	5	2	227	222	12
9	5	-3	444	471	5	17	5	2	288	312	11
11	5	-3	239	232	7	21	5	2	227	207	14
13	5	-3	295	294	7	1	5	3	1180	1223	6
15	5	-3	519	537	6	3	5	3	1119	1124	6
17	5	-3	437	459	7	5	5	3	822	842	4
19	5	-3	451	504	8	7	5	3	737	750	5
25	5	-3	150	156	20	9	5	3	322	298	7
27	5	-3	154	151	21	11	5	3	225	235	11
1	5	-2	596	550	4	13	5	3	479	470	8
3	5	-2	1065	1017	5	15	5	3	314	320	10
7	5	-2	1816	1752	9	1	5	4	456	472	5
9	5	-2	105	100	12	3	5	4	1650	1714	9
11	5	-2	224	222	8	5	5	4	165	206	11
13	5	-2	633	646	5	7	5	4	497	476	6
15	5	-2	816	807	6	11	5	4	490	490	7
17	5	-2	292	318	9	15	5	4	273	256	12
19	5	-2	380	374	9	19	5	4	180	172	19
23	5	-2	178	160	17	1	5	5	1209	1274	6
27	5	-2	175	169	20	5	5	5	592	604	5
1	5	-1	681	518	3	9	5	5	365	339	8
3	5	-1	609	420	4	13	5	5	368	362	9
5	5	-1	1078	988	5	1	5	6	738	820	5
7	5	-1	278	292	6	3	5	6	687	708	5
9	5	-1	371	361	5	5	5	6	509	532	6
13	5	-1	595	617	6	11	5	6	363	371	9
17	5	-1	568	579	7	13	5	6	213	204	14
1	5	0	168	167	6	1	5	7	155	150	12
5	5	0	788	727	4	3	5	7	474	464	6
7	5	0	922	969	5	11	5	7	254	291	12
9	5	0	733	779	5	1	5	8	565	605	6

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
5	5	8	156	164	15	12	6	-6	1005	1008	5
9	5	8	316	319	11	16	6	-6	564	608	6
13	5	8	224	222	15	18	6	-6	131	119	18
1	5	9	336	334	9	24	6	-6	323	280	11
3	5	9	340	324	9	2	6	-5	887	864	5
9	5	9	200	260	16	4	6	-5	438	460	6
11	5	9	189	148	17	6	6	-5	192	183	9
1	5	11	355	335	10	8	6	-5	225	193	8
9	5	11	190	148	20	10	6	-5	424	427	6
6	6	-14	164	120	22	12	6	-5	245	249	9
4	6	-12	314	307	11	14	6	-5	339	319	7
8	6	-12	446	434	9	16	6	-5	279	286	9
10	6	-12	158	187	19	2	6	-4	1802	1826	9
16	6	-12	354	372	11	4	6	-4	1410	1369	7
20	6	-12	168	183	19	6	6	-4	450	410	5
6	6	-11	208	258	14	8	6	-4	185	168	9
10	6	-11	166	161	17	10	6	-4	1060	1040	6
16	6	-11	137	122	21	12	6	-4	766	761	5
2	6	-10	417	412	8	14	6	-4	848	868	5
4	6	-10	209	163	13	16	6	-4	365	382	7
6	6	-10	489	445	8	18	6	-4	214	193	11
8	6	-10	311	308	9	22	6	-4	423	447	9
14	6	-10	238	235	12	24	6	-4	215	245	16
16	6	-10	324	323	10	4	6	-3	1192	1129	6
20	6	-10	263	264	12	6	6	-3	408	380	5
22	6	-10	178	171	18	12	6	-3	730	691	5
26	6	-10	200	216	19	16	6	-3	236	236	10
4	6	-9	134	174	18	2	6	-2	1542	1615	8
8	6	-9	282	321	9	4	6	-2	733	680	4
12	6	-9	204	183	12	6	6	-2	114	144	12
16	6	-9	232	278	12	8	6	-2	135	147	11
2	6	-8	445	435	7	10	6	-2	911	923	5
4	6	-8	460	422	7	12	6	-2	257	290	8
6	6	-8	446	453	7	14	6	-2	661	676	6
12	6	-8	339	331	8	16	6	-2	212	178	11
14	6	-8	393	404	8	18	6	-2	254	259	11
18	6	-8	365	375	9	20	6	-2	256	267	12
20	6	-8	166	175	16	22	6	-2	423	468	10
22	6	-8	130	131	21	2	6	-1	1121	997	6
26	6	-8	239	231	14	4	6	-1	173	130	8
2	6	-7	174	173	12	8	6	-1	409	365	6
4	6	-7	117	136	17	10	6	-1	564	523	5
6	6	-7	278	279	8	12	6	-1	607	620	6
10	6	-7	162	185	12	14	6	-1	138	140	17
12	6	-7	340	362	8	18	6	-1	215	203	12
14	6	-7	292	291	9	22	6	-1	252	194	13
16	6	-7	142	119	16	0	6	0	2236	2109	9
4	6	-6	1316	1304	6	2	6	0	254	330	6
6	6	-6	175	186	11	4	6	0	638	734	4
10	6	-6	301	316	8	6	6	0	225	211	7

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
8	6	0	1239	1251	6	14	6	8	172	173	23
12	6	0	859	880	6	4	6	9	233	231	13
14	6	0	280	270	9	0	6	10	136	147	21
20	6	0	341	358	10	2	6	10	256	253	13
0	6	1	429	443	5	4	6	10	277	294	12
2	6	1	186	146	8	6	6	10	409	388	10
6	6	1	1072	1058	5	1	7	-13	196	180	21
10	6	1	659	627	6	9	7	-13	186	148	19
18	6	1	238	245	12	13	7	-13	240	249	16
0	6	2	1157	1163	6	3	7	-11	390	381	10
2	6	2	1134	1120	6	5	7	-11	183	153	17
4	6	2	129	110	12	7	7	-11	306	268	11
6	6	2	1037	1012	5	11	7	-11	516	533	8
8	6	2	660	631	6	17	7	-11	164	156	18
10	6	2	1260	1240	7	19	7	-11	225	231	15
12	6	2	396	396	8	23	7	-11	178	188	19
18	6	2	305	283	11	1	7	-9	400	411	9
20	6	2	212	190	15	3	7	-9	262	288	11
0	6	3	840	869	4	7	7	-9	160	156	16
2	6	3	239	241	7	9	7	-9	544	498	7
4	6	3	316	310	6	11	7	-9	464	465	8
6	6	3	291	294	7	13	7	-9	261	243	11
8	6	3	632	601	6	17	7	-9	278	286	12
10	6	3	138	125	16	19	7	-9	356	356	10
12	6	3	233	211	12	21	7	-9	226	249	15
14	6	3	170	168	18	23	7	-9	236	283	15
16	6	3	209	195	15	1	7	-8	171	157	13
0	6	4	397	399	5	3	7	-8	134	114	17
2	6	4	643	654	5	5	7	-8	153	173	15
4	6	4	628	638	5	1	7	-7	438	463	7
6	6	4	867	845	5	5	7	-7	197	187	11
8	6	4	487	468	7	7	7	-7	187	184	11
10	6	4	767	736	6	9	7	-7	773	761	6
18	6	4	396	383	11	11	7	-7	210	179	11
0	6	5	239	219	8	13	7	-7	463	506	7
2	6	5	187	184	10	17	7	-7	384	389	8
6	6	5	209	240	11	21	7	-7	379	404	9
8	6	5	193	204	13	29	7	-7	179	176	22
10	6	5	213	230	12	5	7	-6	247	228	9
0	6	6	396	425	6	7	7	-6	178	193	11
4	6	6	436	435	7	9	7	-6	110	122	18
8	6	6	488	455	7	11	7	-6	205	180	11
16	6	6	292	299	15	13	7	-6	269	271	9
20	6	6	181	123	24	15	7	-6	189	168	12
0	6	7	174	172	12	1	7	-5	400	385	6
6	6	7	131	193	20	3	7	-5	163	175	11
2	6	8	178	200	14	7	7	-5	1328	1319	7
4	6	8	176	184	15	9	7	-5	553	522	6
6	6	8	485	500	8	11	7	-5	458	480	6
8	6	8	238	214	13	13	7	-5	493	505	6

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
15	7	-5	241	257	10	5	7	2	241	264	9
19	7	-5	443	461	8	11	7	2	320	325	10
27	7	-5	202	198	19	1	7	3	1296	1292	7
3	7	-4	118	103	14	3	7	3	1359	1382	7
5	7	-4	166	163	11	5	7	3	745	700	5
11	7	-4	333	318	7	7	7	3	516	541	6
13	7	-4	168	173	12	9	7	3	316	377	9
15	7	-4	255	213	9	13	7	3	365	382	10
1	7	-3	201	210	9	15	7	3	400	408	10
3	7	-3	857	828	5	17	7	4	195	146	18
5	7	-3	1396	1354	7	1	7	5	1032	1042	5
7	7	-3	1427	1387	7	3	7	5	346	324	7
9	7	-3	970	919	5	5	7	5	548	525	6
11	7	-3	220	211	10	9	7	5	336	319	9
13	7	-3	553	567	6	11	7	5	187	178	16
15	7	-3	379	395	7	13	7	5	560	574	9
17	7	-3	480	466	7	3	7	7	530	571	7
19	7	-3	424	470	9	7	7	7	163	171	18
25	7	-3	209	202	17	11	7	7	523	506	9
27	7	-3	173	168	22	13	7	7	165	164	21
1	7	-2	424	421	5	15	7	7	211	157	17
3	7	-2	156	115	10	1	7	9	360	351	9
5	7	-2	210	215	8	3	7	9	301	308	11
7	7	-2	143	137	12	9	7	9	235	237	15
9	7	-2	235	231	8	1	7	11	327	316	12
1	7	-1	909	863	5	4	8	-12	214	201	17
3	7	-1	702	584	5	8	8	-12	255	289	14
5	7	-1	1847	1845	9	16	8	-12	229	232	16
7	7	-1	649	583	5	6	8	-11	208	202	16
9	7	-1	836	835	5	18	8	-11	210	188	16
13	7	-1	716	721	6	24	8	-11	150	90	24
15	7	-1	233	215	12	2	8	-10	285	256	12
17	7	-1	708	719	7	4	8	-10	144	140	20
25	7	-1	268	236	14	6	8	-10	483	490	8
1	7	0	158	222	10	8	8	-10	198	213	15
3	7	0	605	526	5	14	8	-10	286	285	12
5	7	0	191	226	9	16	8	-10	370	367	10
11	7	0	319	287	8	20	8	-10	203	192	16
1	7	1	1327	1276	7	4	8	-9	289	275	11
3	7	1	1713	1676	9	8	8	-9	140	103	19
5	7	1	464	472	7	2	8	-8	327	337	9
7	7	1	997	1026	6	4	8	-8	269	283	11
9	7	1	492	520	7	6	8	-8	293	288	9
11	7	1	207	215	11	12	8	-8	221	204	12
13	7	1	128	119	18	14	8	-8	458	426	8
15	7	1	602	637	7	18	8	-8	310	333	11
17	7	1	145	158	19	2	8	-7	298	279	9
19	7	1	227	269	14	6	8	-7	281	254	9
1	7	2	324	325	6	8	8	-7	131	138	18
3	7	2	223	223	9	10	8	-7	225	201	11

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
14	8	-7	241	264	12	2	8	1	1014	946	6
22	8	-7	147	95	20	8	8	1	374	353	8
4	8	-6	615	614	6	10	8	1	443	444	7
12	8	-6	570	574	6	14	8	1	153	134	17
16	8	-6	319	308	10	0	8	2	728	736	5
24	8	-6	288	276	12	2	8	2	978	999	5
2	8	-5	421	402	7	4	8	2	162	148	13
6	8	-5	368	373	7	6	8	2	610	652	6
8	8	-5	233	242	10	8	8	2	657	649	7
10	8	-5	245	237	9	10	8	2	521	492	8
12	8	-5	262	259	9	12	8	2	183	174	15
14	8	-5	372	399	8	18	8	2	227	204	15
20	8	-5	196	203	15	20	8	2	241	194	14
2	8	-4	1241	1228	6	0	8	3	665	670	5
4	8	-4	717	659	5	2	8	3	377	411	7
6	8	-4	382	415	7	6	8	3	133	131	15
10	8	-4	397	395	7	8	8	3	461	432	7
12	8	-4	406	383	7	12	8	3	274	240	12
14	8	-4	620	627	6	0	8	4	318	306	8
22	8	-4	307	315	12	2	8	4	176	192	12
4	8	-3	413	422	6	4	8	4	338	333	8
6	8	-3	202	211	10	6	8	4	425	444	7
12	8	-3	359	358	8	8	8	4	272	273	10
14	8	-3	280	258	9	10	8	4	433	426	9
18	8	-3	180	175	15	12	8	4	183	173	17
20	8	-3	187	216	17	18	8	4	316	333	14
2	8	-2	1813	1809	9	0	8	5	313	320	8
4	8	-2	1019	1019	5	2	8	5	158	144	13
6	8	-2	110	83	16	6	8	5	176	177	14
8	8	-2	456	461	6	8	8	5	219	222	13
10	8	-2	623	626	6	0	8	6	276	299	9
12	8	-2	272	269	9	4	8	6	382	391	8
14	8	-2	641	644	7	6	8	6	223	208	12
16	8	-2	203	223	14	8	8	6	488	485	8
18	8	-2	248	252	13	16	8	6	238	276	20
20	8	-2	240	230	14	4	8	7	218	172	12
22	8	-2	336	359	12	2	8	8	205	241	15
2	8	-1	931	834	5	4	8	8	225	225	14
4	8	-1	387	374	6	6	8	8	435	436	9
10	8	-1	324	363	8	0	8	9	135	115	21
12	8	-1	241	276	10	2	8	9	202	185	15
0	8	0	2069	2181	9	4	8	9	271	230	13
2	8	0	967	893	5	2	8	10	344	328	11
4	8	0	754	712	5	6	8	10	216	197	17
6	8	0	455	460	6	9	9	-12	165	174	22
8	8	0	943	953	6	11	9	-11	220	242	16
12	8	0	466	478	8	1	9	-10	345	308	11
14	8	0	216	224	13	7	9	-10	211	203	15
20	8	0	367	342	11	9	9	-10	356	330	10
0	8	1	345	334	7	13	9	-10	291	267	12

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
19	9	-10	159	169	21	17	9	-2	189	163	16
21	9	-10	191	212	20	19	9	-2	218	237	15
1	9	-9	175	185	18	1	9	-1	217	270	10
9	9	-9	205	204	14	5	9	-1	926	916	5
11	9	-9	280	286	12	7	9	-1	237	246	10
13	9	-9	135	163	22	9	9	-1	439	427	7
1	9	-8	183	196	16	13	9	-1	442	436	8
3	9	-8	177	170	16	15	9	-1	135	118	21
5	9	-8	129	145	21	17	9	-1	384	387	10
7	9	-8	396	401	9	1	9	0	206	206	10
11	9	-8	516	504	8	3	9	0	639	613	6
19	9	-8	324	351	11	5	9	0	675	696	6
1	9	-7	218	230	13	7	9	0	822	842	6
3	9	-7	166	161	16	9	9	0	149	147	16
9	9	-7	188	245	14	15	9	0	405	421	10
13	9	-7	161	157	17	17	9	0	212	216	15
17	9	-7	191	191	16	19	9	0	190	154	16
21	9	-7	162	169	21	1	9	1	653	622	7
1	9	-6	195	168	12	3	9	1	774	784	7
3	9	-6	149	148	15	5	9	1	147	167	17
5	9	-6	332	337	8	7	9	1	249	202	11
7	9	-6	389	385	8	13	9	1	225	253	14
9	9	-6	235	247	10	15	9	1	246	223	13
11	9	-6	413	416	8	1	9	2	548	534	6
17	9	-6	150	161	19	3	9	2	228	230	10
19	9	-6	217	231	15	5	9	2	764	724	6
7	9	-5	398	418	7	7	9	2	262	202	11
11	9	-5	181	154	13	13	9	2	261	282	12
19	9	-5	248	223	13	17	9	2	285	269	13
1	9	-4	273	265	9	1	9	3	360	379	8
3	9	-4	325	331	8	3	9	3	532	530	6
5	9	-4	749	752	6	5	9	3	302	337	9
7	9	-4	156	157	14	11	9	3	188	219	16
9	9	-4	541	531	7	15	9	3	187	135	18
11	9	-4	190	205	12	3	9	4	698	673	6
15	9	-4	340	328	9	5	9	4	174	197	15
17	9	-4	313	353	11	11	9	4	339	345	11
1	9	-3	247	261	9	15	9	4	263	274	15
3	9	-3	341	348	7	1	9	5	569	607	7
5	9	-3	414	410	7	5	9	5	301	312	10
7	9	-3	592	558	6	9	9	5	249	262	13
9	9	-3	477	510	6	13	9	5	274	300	14
13	9	-3	223	213	12	1	9	6	344	330	9
15	9	-3	194	204	15	3	9	6	345	373	9
17	9	-3	283	326	12	5	9	6	245	247	12
3	9	-2	583	624	6	9	9	6	198	211	17
5	9	-2	282	271	8	11	9	6	266	253	14
7	9	-2	934	919	5	3	9	7	279	276	11
9	9	-2	164	162	14	9	9	7	159	110	20
15	9	-2	570	559	8	11	9	7	226	243	17

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
1	9	8	427	440	9	12	10	-1	415	416	9
5	9	8	144	186	22	14	10	-1	230	244	14
9	9	8	180	168	20	18	10	-1	202	199	18
13	9	8	178	142	23	0	10	0	188	177	12
1	9	9	213	204	16	2	10	0	204	219	11
4	10	-11	170	112	22	6	10	0	348	331	9
6	10	-11	276	250	15	12	10	0	155	135	20
18	10	-11	249	217	16	0	10	1	584	608	6
4	10	-9	485	473	9	2	10	1	660	653	7
8	10	-9	361	349	10	6	10	1	322	295	10
12	10	-9	223	204	15	8	10	1	356	381	10
16	10	-9	368	358	11	10	10	1	652	627	8
8	10	-8	165	164	19	12	10	1	148	208	21
2	10	-7	461	491	8	18	10	1	220	209	17
4	10	-7	248	241	12	2	10	2	304	318	9
6	10	-7	467	454	8	4	10	2	206	201	13
10	10	-7	222	220	13	12	10	2	150	143	22
12	10	-7	227	212	14	0	10	3	809	806	6
14	10	-7	384	421	10	2	10	3	166	174	15
16	10	-7	165	172	18	4	10	3	170	169	15
22	10	-7	199	163	18	6	10	3	224	209	13
6	10	-6	160	159	17	8	10	3	648	682	8
10	10	-6	208	211	14	12	10	3	304	312	13
18	10	-6	169	138	18	16	10	3	223	215	18
2	10	-5	549	514	7	20	10	3	203	198	21
4	10	-5	358	341	9	6	10	4	229	243	13
6	10	-5	339	353	9	8	10	4	163	141	18
10	10	-5	453	441	8	0	10	5	262	250	11
12	10	-5	215	259	13	6	10	5	405	407	9
14	10	-5	471	462	8	8	10	5	277	274	12
16	10	-5	196	197	15	10	10	5	358	349	11
22	10	-5	206	213	18	4	10	6	272	256	12
2	10	-4	240	224	10	8	10	6	164	147	20
4	10	-4	152	158	15	0	10	7	161	107	18
2	10	-3	152	153	15	4	10	7	276	310	13
4	10	-3	646	623	6	6	10	7	371	379	11
6	10	-3	317	310	9	10	10	7	184	179	21
8	10	-3	219	226	11	4	10	9	310	294	14
12	10	-3	750	758	7	1	11	-10	229	234	18
14	10	-3	138	128	20	9	11	-10	267	266	15
16	10	-3	251	262	13	13	11	-10	213	240	18
20	10	-3	291	289	13	9	11	-9	162	161	20
24	10	-3	197	182	21	3	11	-8	242	207	13
2	10	-2	143	128	15	7	11	-8	195	214	17
10	10	-2	209	185	13	11	11	-8	401	413	10
12	10	-2	204	221	14	19	11	-8	229	240	18
2	10	-1	620	638	6	3	11	-7	167	161	18
4	10	-1	301	285	9	7	11	-7	165	155	18
6	10	-1	128	132	18	9	11	-7	215	224	14
10	10	-1	692	669	7	1	11	-6	440	448	9

H	K	L	Fobs	Fcalc	SigF	H	K	L	Fobs	Fcalc	SigF
7	11	-6	282	294	11	11	11	6	180	197	22
9	11	-6	352	354	10	3	11	7	192	145	17
11	11	-6	138	200	22	1	11	8	339	337	11
15	11	-6	174	191	18	3	11	9	170	151	21
19	11	-6	232	216	15	4	12	-9	204	197	18
1	11	-5	174	205	16	16	12	-9	185	178	21
3	11	-5	138	160	19	2	12	-8	175	131	19
1	11	-4	253	277	12	6	12	-8	266	253	13
5	11	-4	557	529	7	12	12	-8	199	227	18
9	11	-4	466	498	8	14	12	-8	149	150	23
11	11	-4	133	113	21	2	12	-7	178	167	18
17	11	-4	339	362	12	6	12	-7	408	428	10
3	11	-3	202	186	13	14	12	-7	172	190	21
5	11	-3	146	68	16	4	12	-6	333	294	10
1	11	-2	241	260	11	12	12	-6	316	297	11
3	11	-2	453	456	7	2	12	-5	374	372	10
5	11	-2	208	180	12	4	12	-5	295	294	11
7	11	-2	793	801	7	6	12	-5	211	218	14
9	11	-2	240	210	11	10	12	-5	297	338	11
15	11	-2	501	528	9	14	12	-5	225	242	15
17	11	-2	194	164	16	2	12	-4	283	266	11
19	11	-2	176	212	22	4	12	-4	198	215	15
1	11	-1	146	140	17	10	12	-4	165	209	19
3	11	-1	413	412	8	12	12	-4	187	178	17
5	11	-1	185	232	15	14	12	-4	238	251	14
11	11	-1	142	182	21	4	12	-3	231	224	13
1	11	0	299	324	9	8	12	-3	225	213	13
5	11	0	486	477	8	12	12	-3	430	470	10
7	11	0	460	447	8	2	12	-2	290	291	11
9	11	0	261	257	12	8	12	-2	237	191	13
11	11	0	199	201	16	10	12	-2	168	139	18
13	11	0	217	203	16	12	12	-2	139	132	22
15	11	0	386	372	11	14	12	-2	292	238	12
17	11	0	254	210	14	2	12	-1	226	196	12
3	11	1	396	427	9	4	12	-1	169	170	15
5	11	1	175	217	19	8	12	-1	194	172	15
9	11	1	271	252	12	10	12	-1	416	422	9
1	11	2	249	266	12	12	12	-1	223	196	15
3	11	2	225	255	13	14	12	-1	231	231	15
5	11	2	560	572	8	0	12	0	293	326	10
13	11	2	407	406	11	2	12	0	190	152	14
7	11	3	199	161	15	6	12	0	261	258	11
1	11	4	319	338	10	8	12	0	329	331	11
3	11	4	674	670	7	12	12	0	170	217	22
7	11	4	146	175	21	0	12	1	200	200	13
11	11	4	332	330	13	2	12	1	285	279	11
5	11	5	171	165	18	4	12	1	220	195	14
1	11	6	330	350	10	6	12	1	195	210	17
3	11	6	437	459	9	10	12	1	346	347	12
5	11	6	286	267	12	12	12	1	224	202	16

H	K	L	Fobs	Fcalc	SigF
8	12	2	218	172	17
10	12	2	234	236	15
0	12	3	384	385	9
8	12	3	423	418	11
10	12	4	287	263	14
4	12	5	202	221	16
6	12	5	324	342	11
0	12	6	251	271	13
1	13	-7	232	217	16
9	13	-7	262	264	15
1	13	-6	204	245	17
9	13	-6	173	139	19
7	13	-5	346	353	11
3	13	-4	177	125	16
5	13	-4	251	307	13
9	13	-4	211	227	16
5	13	-3	293	296	11
7	13	-3	311	340	11
5	13	-2	216	248	15
7	13	-2	187	210	17
5	13	-1	395	374	10
9	13	-1	254	248	14
13	13	-1	162	191	21
9	13	0	198	219	16
11	13	0	150	120	22
3	13	1	372	365	10
7	13	1	260	289	15
15	13	1	212	224	21
7	13	2	214	220	18
1	13	3	272	277	12
3	13	3	329	342	11
5	13	3	233	198	15
1	13	4	188	195	17
3	13	4	258	244	13
1	13	5	355	337	11
5	13	5	301	274	14
3	13	7	228	232	17
4	14	-6	312	321	14
12	14	-6	229	198	17
2	14	-4	333	328	11
4	14	-4	147	145	23
2	14	-2	385	386	10
4	14	-2	155	175	21
10	14	-2	291	298	14
14	14	-2	156	173	24
4	14	-1	154	144	20
0	14	0	521	514	9
8	14	0	288	298	13
12	14	0	260	210	16
4	14	1	158	185	21
0	14	2	232	250	15
2	14	2	257	243	15
10	14	2	215	179	19
6	14	4	172	175	24
5	15	-3	171	131	22
7	15	-3	179	186	22
5	15	-1	361	346	12
1	15	1	195	207	20
3	15	1	310	280	14
4	16	-3	175	158	23
0	16	0	211	242	21
2	16	1	189	206	22