# A Survey of Dimeric mu-Bridged Compounds and Determination of Unit Cell Dimensions of Several Pt-Complexes

Thesis

Presented to the Graduate Council of

Southwest Texas State University

in Partial Fulfillment of the Requirements

For the Degree of

MASTER OF ARTS

By

John Charles Mayfield, B. S.
(Johnson City, Texas)

San Marcos, Texas

May, 1971

## Dedication

The thesis is dedicated to the writer's parents, Tom and Addie Mayfield. Their continuous encouragement, financial support throughout an undergraduate career, and emphasis of the importance of a proper education formed the background that made this thesis a reality.

## Acknowledgments

The writer wishes to express appreciation to Dr. Willis A. Cude, Jr., Associate Professor of Chemistry, Southwest Texas State University for his supervision and support throughout the course of this investigation.

Appreciation is expressed to Dr. John W. Fitch, III, Assistant Professor Chemistry and Dr. Henry N. McEwen, Associate Professor Mathematics for their review of this thesis.

Appreciation is also expressed to the Physics and Mathematics Departments of S. W. T. S. U. for use of the X-ray source and computer, respectively, without which this work would have been impossible.

Special acknowledgment goes to the writer's wife, Janice. Without her understanding and support this thesis would not have become a reality.

John Charles Mayfield

San Marcos, Texas

May, 1971

Table of Contents

List of Tables

List of Figures

# Introduction

The objective of the problem was to survey recent literature for all mu-bridged compounds and to determine the unit cell dimensions of several platinum complexes.

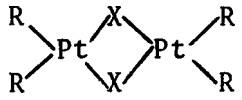
The literature survey was done using the subject indices of Chemical Abstracts for the last ten years. Each compound was recorded for future reference. A more detailed examination of the reported platinum complexes involved the structural determination and method of preparation of each compound.

The experimental part of the problem employed an X-ray source and Weissenberg camera to obtain photographs of single crystals. From these photographs, data were taken and the unit cell dimensions calculated using an University of Arkansas program which was modified for the IBM 1130 computer at Southwest Texas State University.

# Chapter I

## Introduction

Compounds of the structural type (I) were defined as dimeric mu-bridged complexes. The complexes consisted of two metal atoms, in this case platinum, and two bridging groups between the metal atoms. The molecule also contained various other groups to satisfy the coordination requirements of the platinum atoms.




(I)

The bridging groups, X, were found to be of various types. Detailed literature searching was restricted to X groups which consisted of only one atom, such as a halide, or groups in which there was only one atom actually in the bridge, such as $NH_2$. In the latter case the two hydrogens were attached directly to the nitrogen and were not attached to the metal atoms.

The subject indices of Chemical Abstracts were searched from volume 54 through 70 (January 1960 - June 1969). for all mu-bridged compounds.

Seventy-three element headings, see Table I, were searched for the mu-bridged compounds. Each compound

reported was recorded for future reference. Over 5400 compounds were found in the indices listed as being mu-bridged. Of the seventy-three element headings searched, sixty-four of these were reported to form mu-bridged compounds. The number of compounds reported for each element ranged from only one for five elements to over 600 for copper and iron.

Because the number of compounds reported was over 5400, only those compounds reported for the element platinum were studied further.

About 200 complexes were reported for platinum. Of these compounds, about 80 were rejected as not fitting the definition of dimeric mu-bridged compound as given above.

The references pertaining to the remaining platinum dimeric mu-bridged compounds were searched in detail for information concerning structural details and method of preparation. Although the search was concentrated on the complexes of platinum, special note was made of analogous palladium compounds.

The platinum compounds were indexed according to the nature of the dimeric mu-bridge and the ligands coordinated to the platinum atoms. Details of the indexing were recorded with the introduction to each dimeric type.

Table I

Elements Surveyed

| Element | Number of mu-bridged Compounds Reported |
|---|---|
| Aluminum | 200 |
| Antimony | 45 |
| Arsenic | 11 |
| Barium | 1 |
| Berylium | 70 |
| Bismuth | 8 |
| Boron | 115 |
| Cadmium | 80 |
| Calcium | 6 |
| Cerium | 2 |
| Cesium | 7 |
| Chromium | 180 |
| Cobalt | 375 |
| Copper | 615 |
| Erbium | 4 |
| Europium | 1 |
| Gadolinium | 2 |
| Gallium | 65 |
| Germanium | 10 |
| Gold | 60 |
| Hafnium | 7 |
| Holmium | 1 |
| Indium | 20 |
| Iridium | 55 |
| Iron | 650 |
| Lanthanum | 15 |
| Lead | 10 |
| Lithium | 25 |
| Magnesium | 40 |
| Manganese | 185 |
| Mercury | 275 |
| Molybdenum | 250 |
| Neodymium | 25 |
| Nickel | 225 |
| Niobium | 50 |
| Osmium | 25 |
| Palladium | 425 |
| Phosphorus | 30 |
| Platinum | 200 |
| Potassium | 6 |

Table I (continued)

| Element | Number of mu-bridged Compounds Reported |
|---|---|
| Praseodymium | 20 |
| Rhenium | 145 |
| Rhodium | 200 |
| Rubidium | 1 |
| Ruthenium | 100 |
| Samarium | 6 |
| Selenium | 1 |
| Silicon | 15 |
| Silver | 25 |
| Sodium | 10 |
| Strontium | 2 |
| Tantalum | 40 |
| Technetium | 15 |
| Thallium | 25 |
| Thorium | 10 |
| Tin | 45 |
| Titanium | 130 |
| Tungsten | 70 |
| Uranium | 50 |
| Vanadium | 25 |
| Ytterbium | 4 |
| Yttrium | 4 |
| Zinc | 110 |
| Zirconium | 45 |

Elements searched for which no mu-bridged compounds were reported.

Lutetium, Polonium, Promethium, Protactinium, Radium, Scandium, Tellurium, Terbium, Thulium

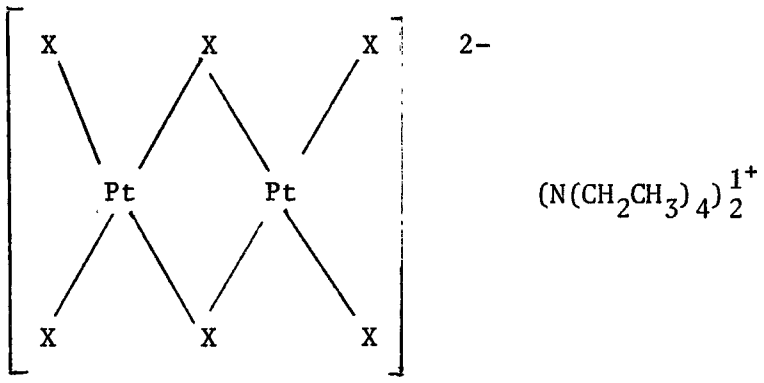
The formula was given for each compound according to the following style; the platinum atoms first, followed by the bridging groups and then the ligands attached to the platinum atoms. The following is given as an example of the style used $Pt_2Cl_2(C_2H_4)_2Cl_2$, di-mu-chlorodichlorobis (ethylene)diplatinum(II).

The name of the compound was given after the formula for each species except when a series of complexes was discussed and the changes in the naming was obvious.

A complete list of all compounds was collected in Appendix I. The existence of analogous palladium complexes also was noted in Appendix I.

The compounds were divided into seven classifications according to the type of bridging and the type of ligands present

Type A consisted of three compounds each with halide bridging and four terminal halides. The type had the following structural representation where X= Br, Cl, I.




$Pt_2X_2(X)_4$, di-mu-bromotetrabromodiplatinum(II)bis (tetraethylammonium)

Adams, et al.[1] and Day, et al.[2] assigned the structures of the halide complexes. Adams, et al. discussed the halide complexes with emphasis on determination of the differences of the ir spectra due to terminal and bridging halides. The authors showed that the two halide positions had different force constants. Day, et al. discussed only the bromo- and iodo- complexes. The assignment of the structures made by Day, et al. was based on X-ray powder patterns and polarised crystal spectra.

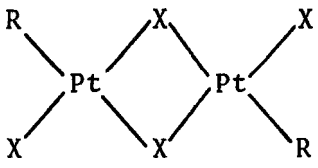
---

[1]D. M. Adams, P. J. Chandler and R. G. Churchill, J. Chem. Soc. Sect. A, 1272-4 (1967).

[2]P. Day, M. J. Smith and R. J. P. Williams, ibid., 668-72 (1968).

Harris, et al.[3] reported the preparation of the bromo-complex as the reaction of potassium tetrachloroplatinum(II) in 20 ml of water and 1 g NaBr. The mixture was added to tetraethylammonium bromide (1 g) in 8 ml of water. The resulting mixture was filtered and the product was recrystallized from acetone to yield 0.52 g of yellowish-brown crystals. Anal. Calcd for $C_{16}H_{20}N_2Br_6Pt_2$: C, 17.0; H, 3.6; N, 2.5; Br, 42.4 Pt, 34.6. Found: C, 17.5; H, 3.5; N, 2.5; Br, 41.5; Pt, 35.2. Similar preparations were reported for the analogous chloro- and iodo- complexes.

Fifty-two compounds were classified as Type B. The type consisted of two halides as bridging groups, two terminal halides and two other terminal groups of various types. Type B had the following general structure.

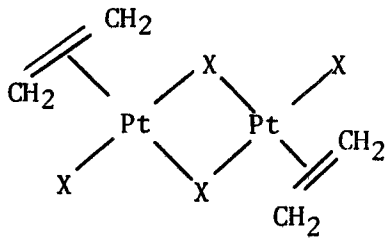



The various structures were shown in Figure 1 and indexed for reference to the discussion section.

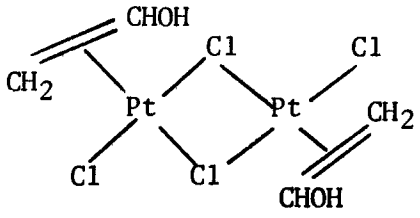
---

[3]C. M. Harris, S. E. Livingstone and N. C. Stephenson, ibid., 3697 (1958).

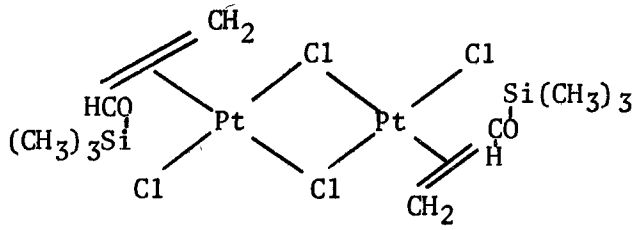
B-1




B-2




B-3




B-4

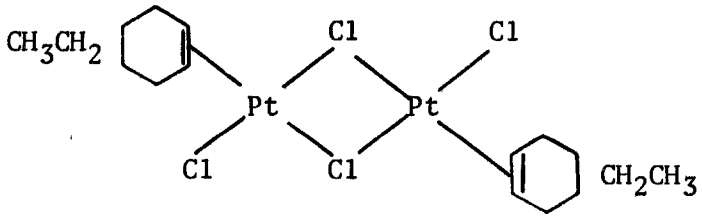
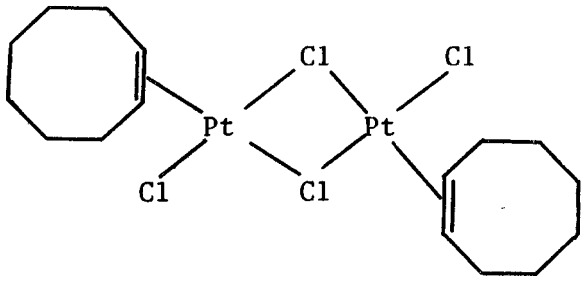


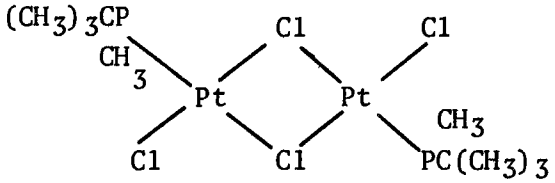

Figure 1

Structures of Complexes Type B

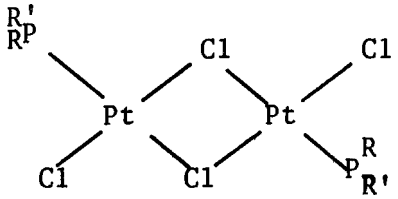
B-5


Cl
Cl
Pt
Pt
Cl
Cl

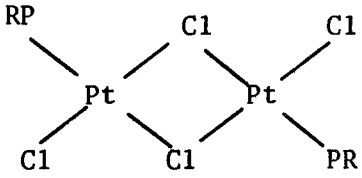
B-6


(CH3)3CP
CH3
Cl
Cl
Pt
Pt
Cl
Cl
CH3
PC(CH3)3

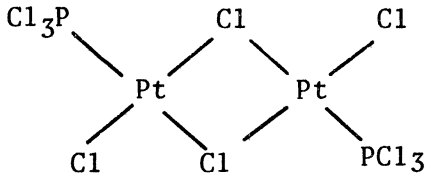
B-7


R'
RP
Cl
Cl
Pt
Pt
Cl
Cl
R
P
R'

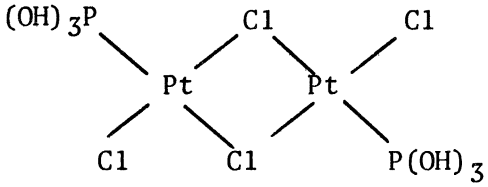
B-8


RP
Cl
Cl
Pt
Pt
Cl
Cl
PR

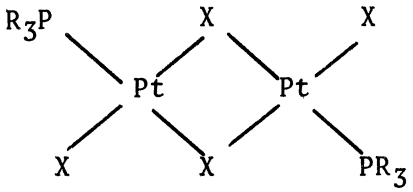
B-9



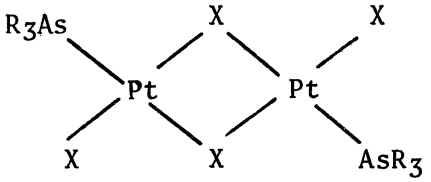

B-10



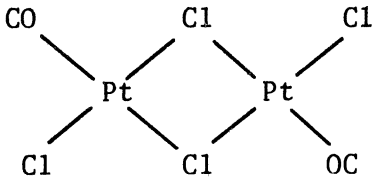

B-11



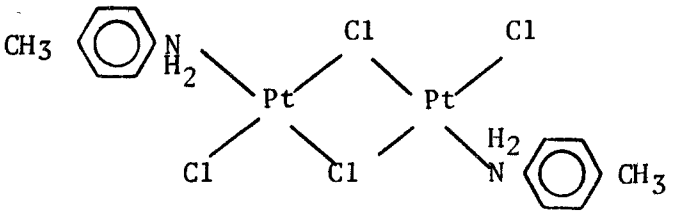

B-12



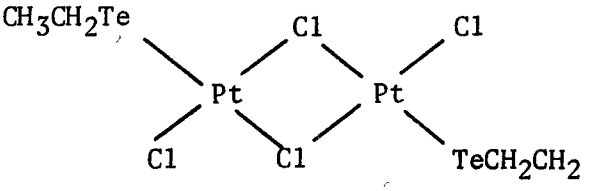

B-13



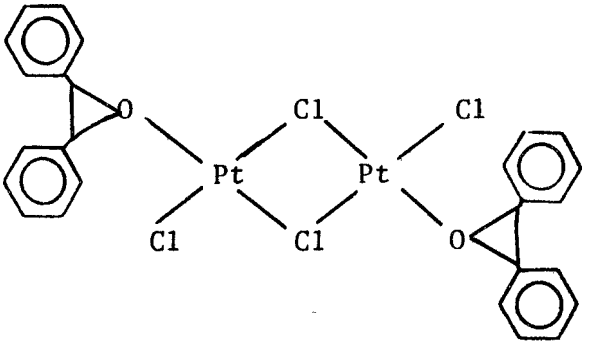
B-14



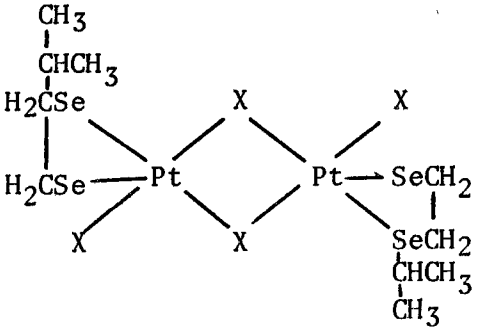
B-15



B-16



B-17



B-1

$Pt_2X_2R_2Cl_2$, X=Br when R= $C_2H_4$ and X= Cl when R= $C_2H_4$, $C_3H_6$, $C_4H_8$; di-mu-bromodibromobis(ethylene)diplatinum(II)

The structure of the complexes have been discussed in detail by Sorzano and Fackler[4], Goodfellow, et al.[5] and Grogan and Nakamoto[6]. All of the authors based the assigned structures on ir and Raman spectra of the complexes. Structural features of each of the compounds were correlated to the various stretching frequences of the spectra.

The preparation of the complex for R= $C_2H_4$ and X= Cl was carried out by the detailed procedure described by Chatt and Searle.[7] The other complexes were prepared by the same general procedure.

B-2

$Pt_2Cl_2R_2Cl_2$, R= $CH_2$:CHOH, $CH_3CH$:CHOH; di-mu -chlorodichlorbis(vinyl alcohol)diplatinum(II)

---

[4]J. P. Sorzano and J. P. Fackler, J. Mol. Spectrosc. 22, 80-98 (1967).

[5]R. J. Goodfellow, P. L. Goggin, J. Chem. Soc. Sect. A, 1897-1900 (1967).

[6]M. J. Grogan and K. Nakamoto, J. Amer. Chem. Soc. 90, 918-22 (1968).

[7]J. Chatt and M. L. Searle, ''Inorganic Syntheses'', V, McGraw-Hill, New York, 1957, p 210.

B-3

$Pt_2Cl_2(CH_2{:}CHOSi(CH_3)_3)Cl_2$; di-mu-chlorodichlorobis (trimethyl(vinyloxy)silane)diplatinum(II)

The complexes were characterized by Wakatsuki, et al.[8] on the basis of interpretation of ir spectra.

Details of the preparation were not given in the abstract for the complexes of Type B-2 but were given for the silane complex as the reaction of toluene and $(C_2H_4PtCl_2)_2$ with $H_2C{:}CHOSi(CH_3)_3$. The complex decomposed on melting, 120-2°.

B-4

$Pt_2Cl_2(C_6H_9CH_2CH_3)_2Cl_2$, di-mu-chlorodichlorobis (ethylcyclohexene)diplatinum(II)

Benkeser, et al.[9] reported the complex to be dimeric. Their determination was on the basis of molecular weight measurements.

The authors gave the following procedure for the preparation of the complex. One gram of freshly prepared platinum(II)chloride was partially dissolved

---

[8]Y. Wakatsuki, S. Nozajura and S. Murahashi, Bull. Chem. Soc. Jap. 42, 273 (1969), Chem. Abstr. 70, 87942a (1969).

[9]R. A. Benkeser, S. Dunny, G. S. Li, P. G. Nerlekar and S. D. Work, J. Amer. Chem. Soc. 90, 1871-5. (1968).

with 21 ml of glacial acetic acid. The solution was filtered after first adding 1.5 ml 1-ethylcyclohexene. The precipitate was recrystallized from chloroform-hexane. The yellowish orange crystal decomposed on melting, 130°. The complex was soluble in chloroform and insoluble in water. The product was stable in air and was not hygroscopic. Anal.Calcd: C, 25.55; H, 3.72; Cl, 18.90; molecular weight, 752. Found: C, 25.87; H, 3.72; Cl, 18.63; molecular weight, 748.

B-5

$Pt_2Cl_2(C_8H_{14})_2$, di-mu-chlorodichlorobis(cyclooctene) diplatinum(II)

Wrixon, et al.[10] assigned the structure of the complex and made a study of the complex using circular dichroism. The authors discussed how circular dichroism can be used to establish the absolute configuration of complexes and applied their methods to the platinum-olefin complex.

The authors did not discuss the method of preparation but noted that the complex was a gift from the collection of the late Professor A. C. Cope of MIT.

---

[10]A. D. Wrixon, E. Premuxic and A. I. Scott, Chem. Commun., 639-41 (1968).

B-6

$Pt_2Cl_2((CH_3)_3CCH_3C_6H_5P)_2Cl_2$, di-mu-chlorodichlorobis (tert-butylmethylphenylphosphine)diplatinum(II)

Chan[11] made the structural assignment of this tert-phosphine on the basis of nmr spectra and studies of the optical rotation of the complex.

The author reported that the complex was prepared by reacting methylphenyl-tert-butylphosphine with potassium tetrachloroplatinum(II). The reaction mixture was refluxed with platinum dichloride in a solution of xylene. The orange crystals had a melting point of 273-275°.

B-7

$Pt_2Cl_2(RR'P)_2Cl_2$, R= $(CH_3)_2$, $(CH_3CH_2)$, $(CH_3CH_2CH_2)_2$, $(CH_3CH_2CH_2CH_2)_2$ when R'= $C_6H_5$ and R= $CH_3$, $CH_3CH_2$, $CH_3CH_2CH_2$, $CH_3CH_2CH_2CH2$ when R'= $(C_6H_5)_2$; di-mu-chlorodichlorobis(dimethylphenylphosphine)diplatinum(II)

Grim, et al. [12] confirmed the structures of the complexes on the basis of phosphorus-31 nmr spectra.

---

[11]T. H. Chan, Chem. Commun., 895-6 (1968).

[12]S. O. Grim, R. L. Keiter and W. McFarlane, Inorg. Chem. 6, 1133-7 (1967).

The authors reported that the preparation of the complexes followed the general procedure given below except that the appropriate ligand was used in place of cis-$((CH_3)_2C_6H_5P)_2PtCl_2$).

Di-mu-chlorodichlorobis(dimethylphenylphosphine) diplatinum(II) was prepared by reacting a mixture of cis-$((CH_3)_2C_6H_5P)_2PtCl_2$) (3.0 g) and $PtCl_2$ (1.5 g) with naphthalene (10 g). The mixture was heated slowly to 200° in an oil bath and maintained at that temperature for 5 minutes. The mixture was cooled slowly to room temperature. The naphthalene was extracted with petroleum ether and methylene chloride was used to dissolve the residue. Unreacted $PtCl_2$ was removed by filtration. The resulting filtrate was concentrated to about 50 ml, and the petroleum ether was added slowly until the solution was just cloudy. The mixture was then cooled in an ice bath which caused precipitation of bright yellow crystals of the complex. The complex was recrystallized twice. The yield was 1.3 g of product. Anal. Calcd for $C_{16}H_{22}P_2Pt_2Cl_4$: C, 33.49; H, 2.81; cl, 15.21. Found: C, 33.53; H, 2.97; Cl, 15.25; mp, 263-66° decomposed. Calcd for $C_{20}H_{30}P_2Pt_2Cl_4$: C, 27.79; H, 3.50; Cl, 16.41. Found: C, 28.03; H, 3.39; Cl, 16.70; mp, 163-4°. Calcd for $C_{24}H_{38}P_2Pt_2Cl_4$: C, 31.31; H, 4.16; Cl, 15.40. Found: C, 31.18; H, 4.20; Cl, 15.55; mp, 199-201°.

Calcd for $C_{28}H_{46}P_2Pt_2Cl_4$: C, 34.44; H, 4.75. Found: C, 34.22; H, 4.75; mp, 176-7°. Calcd for $C_{28}H_{30}P_2Pt_2Cl_4$: C, 35.01; H, 3.15; Cl, 14.76. Found: C, 34.93; H, 3.39; Cl, 14.98; mp, 236-41° decomposed. Calcd for $C_{30}H_{34}P_2Pt_2Cl_4$: C, 36.45; H, 3.46; Cl, 14.35. Found: C, 36.55; H, 3.41; Cl, 14.57; mp, 240-45° decomposed. Calcd for $C_{32}H_{38}P_2Pt_2Cl_4$: C, 37.58; H, 4.33; Cl, 13.87. Found C, 37.46; H, 4.13; Cl, 13.80; mp, 205-20° decomposed.

B-8

$Pt_2Cl_2(RP)_2Cl_2$, R= $(C_6H_5)_2C_6H_{11}$, $C_6H_5(C_6H_{11})_2$; di-mu-chlorodichlorobis(cyclohexyldiphenylphosphine)diplatinum(II)

Smithies, et al.[13] discussed the structures in terms of observed stretching frequencies and dipole moments as a function of the R groups.

The tert-phosphines were prepared by the following general procedure. Platinum(II)chloride (1.1 mmole) was added to a solution of dichlorobis(tert-phosphine) platinum(II) (0.9 mmole) in tetrachlor-ethane (25 ml). The solution was refluxed for 1 hour under an atmosphere of nitrogen. After cooling, the solution was filtered and reduced to a small volume (25 ml) by evaporation under reduced pressure. The product was

---

[13]A. C. Smithies, M. Rycheck and M. Orchin, J. Organometal. Chem. 12, 199-202 (1968).

then precipitated by the addition of hexane. The product was purified by recrystallization from chloroform and hexane. The final yields for the various complexes were 50-80%.

B-9,10

$Pt_2Cl_2(PCl_3)_2Cl_2$, di-mu-chlorodichlorobis(phosphorous trichloride)diplatinum(II)

$Pt_2Cl_2(P(OH)_3)_2Cl_2$, di-mu-chlorodichlorobis(phosphorous acid)diplatinum(II)

Adams and Chandler[14] reported the structures based on interpretation of ir stretching frequencies of the platinum-phosphorous bonds.

The method of preparation was not discussed.

B-11

$Pt_2X_2(PR_3)_2X_2$, X= Br, Cl, I and R= $CH_3$, $CH_3CH_2$, $CH_3CH_2CH_2$, $CH_2CH_2CH_2CH_2$, $C_6H_5$; di-mu-bromodibromobis(trimethylphosphine)diplatinum(II)

The structures for this series were reported by Goodfellow, et al.[5] for R= $CH_3$, Adams and Chandler[14] for R= $CH_3CH_2$, $CH_3CH_2CH_2$, $C_6H_5$ and Miller[15] for R=

---

[14]D. M. Adams and P. J. Chandler, J. Chem. Soc. Sect. A, 588-97,(1969).

[15]R. G. J. Miller, Colloq. Spectrosc. 12th, Exeter, 523-39 (1965); Chem. Abstr. 68, 17250h (1968).

$CH_3CH_2CH_2CH_2$. The authors stated that the structural determinations were based on interpretation of ir spectra and chemical analysis.

B-12

$Pt_2X_2(AsR_3)_2X_2$, X= Br, Cl, I, R= $CH_3$, $CH_3CH_2$, $CH_3CH_2CH_2$, and R= $C_6H_5$ only for X= Cl; di-mu-bromo-dibromobis(trimethylarsine)diplatinum(II)

Several authors[5,14,16-18] discussed the assignment of the structures for the complexes. Their structural determinations were based on the interpretation of ir spectra, chemical analyses and single crystal X-ray diffraction.

The methods of preparation were not discussed, however; the chemical analyses for the following complexes were recorded. Anal. Calcd for $C_6H_{18}As_2Br_4Pt_2$: C, 7.7; H, 1.9. Found: C, 7.6 ; H, 1.9; orange, mp, 230-39° decomposed. Calcd for $C_{12}H_{30}As_2Br_4Pt_2$: C, 14.2; H, 3.0. Found: C, 13.9; H, 2.9; orange-red, mp, 182-83° Calcd for $C_{18}H_{42}As_2Br_4Pt_2$: C, 19.5; H, 4.0. Found: C, 19.3; H, 3.8; red, mp, 144-5°. Calcd for $C_6H_{18}As_2$

---

[16]S. F. Watkins, Chem. Commun. 504-5 (1968).

[17]R. J. Goodfellow, J. G. Evans, P. L. Goggin and D. A. Duddell, J. Chem. Soc. Sect. A, 1604-9 (1968).

[18]M. J. Taylor, A. L. Odell and H. A. Raethel, Spectrochim. Acta, 1855-61 (1968).

$I_4Pt_2$: C, 6.4; H, 1.6. Found: C, 6.35; H, 1.6; red, mp, 275-78°. Calcd for $C_{12}H_{30}As_2I_4Pt_2$: C, 12.0; H, 2.6. Found: C, 11.8; H, 2.5; claret, mp, 193° decomposed.

B-13

$Pt_2Cl_2(CO)_2Cl_2$, dicarbonyldi-mu-chlorodichlorodiplatinum(II)

Goodfellow, et al.[5] determined the structure of the complex by interpretation of ir spectra. The authors discussed the various stretching frequencies in terms of the proposed structure.

Detailed method of preparation was reported by Chatt and Searle.[7]

B-14-15

$Pt_2Cl_2(C_6H_4CH_3NH_2)_2Cl_2$, di-mu-chlorodichlorobis(para-toluidine)diplatinum(II)

$Pt_2Cl_2(TeCH_2CH_3)_2Cl_2$, di-mu-chlorodichlorobis(ethyltelluride)diplatinum(II)

Adams and Chandler[14] discussed the ir spectra of the complex and assigned the structure on the basis of characteristic bands.

B-16

$Pt_2Cl_2((C_6H_4)_2C_3H_3O)_2Cl_2$, di-mu-chlorodichlorobis(diphenylcyclopropenone)diplatinum(II)

Bird and Briggs[19] determined the structure of the complex based on interpretation of ir spectra.

The method of preparation was reported as the reaction of diphenylcyclopropenone with benzonitril-platinumdichloride. The crystals produced were bright yellow.

B-17

$Pt_2X_2(Se_2C_2H_4(C_3H_7)_2)_2X_2$, X= Br, Cl; di-mu-bromodibromo-bis(1,2-bis(isopropylseleno)ethane))diplatinum(II)

Greenwood and Hunter[20] assigned the structure as one of two possible. Their structural determination was the result of interpretation of ir, nmr, uv and visible spectra. The authors noted that the structure shown was less favored than a structure which had the ligand containing selenium as bridging groups. The structure, B-17, implied penta-coordination. The alternate structure implied the more common tetra-coordination.

The complex for X= Cl was prepared by the addition of 1.88 g (40% Pt) hexachloroplatinum(IV) acid to 10 ml ethanol. The platinum(IV) was reduced to platinum

---

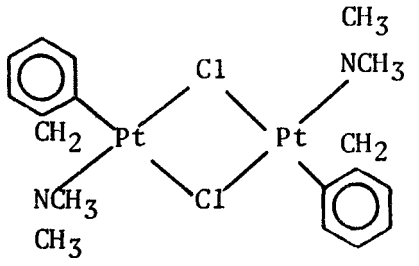
[19]C. W. Bird and E. M. Briggs, J. Chem. Soc. Sect. A, 1004-6 (1967).

[20]N. N. Greenwood and G. Hunter, ibid. 1520-3 (1967).

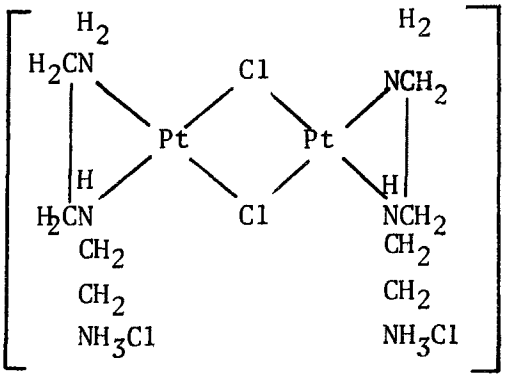
(II) by the addition of 0.2 g of hydrazine hydrochloride. The deep red solution was warmed until evolution of nitrogen was completed, the mixture was added to a solution of 1.05 g (3.85 mmole) of 1,2-bis(isopropylseleno)ethane. The resulting mixture was refluxed for 30 minutes, filtered, cooled and recrystallized from chloroform. The bromo-complex was prepared by a similar method.

Forty-one compounds were classified as Type C. This type consisted of two halides as bridging groups and various other groups to satisfy the coordination requirements of the platinum atoms. The structures of Type C were drawn in Figure 2.

C-1

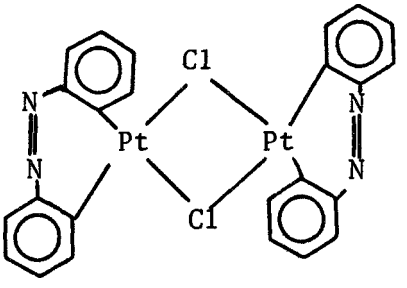



C-2




$PtCl_4^{2-}$

C-3




C-4

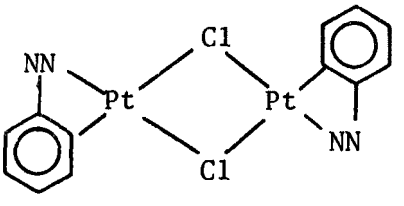
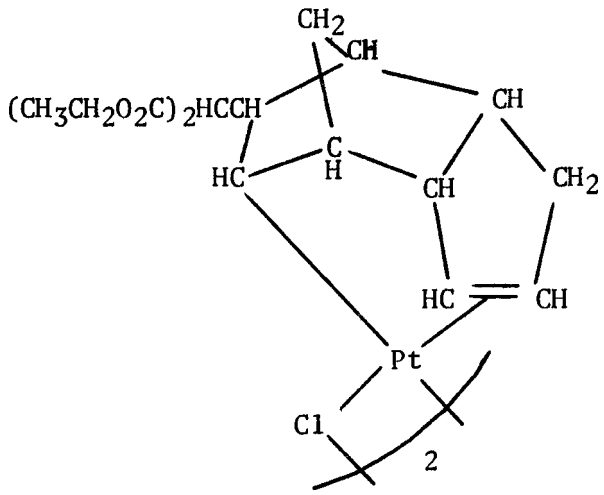


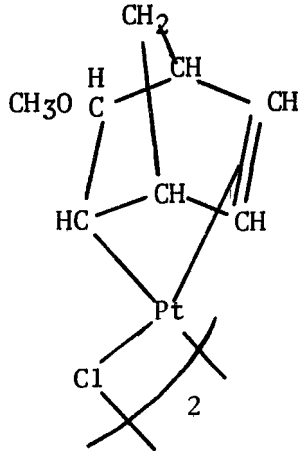

Figure 2

Structure of Complexes Type C

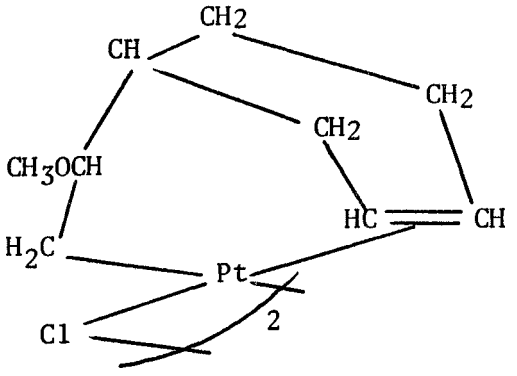
C-5


CH2
CH
(CH3CH2O2C)2HCCH
CH
C
H
HC
CH
CH2
HC
CH
Pt
Cl
2

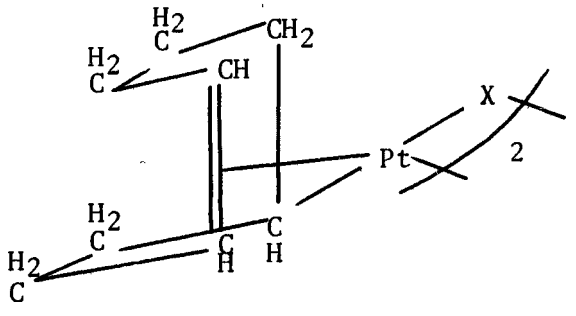
C-6


CH2
CH
H
C
CH3O
CH
CH
HC
CH
Pt
Cl
2

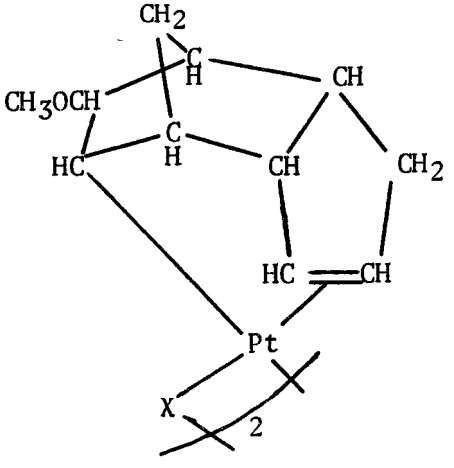
C-7


CH2
CH
CH2
CH2
CH3OCH
HC
CH
H2C
Pt
2
Cl

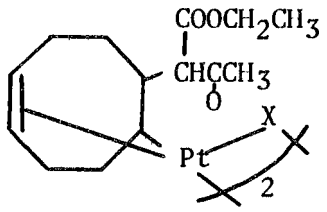
C-8


H2
C
CH2
H2
C
CH
X
Pt
2
H2
C
C
H
C
H
H2
C

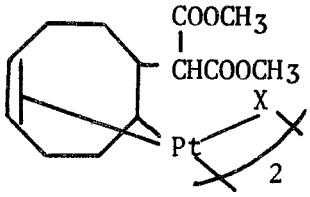
C-9


CH2
C
H
CH
CH3OCH
C
H
HC
CH
CH2
HC
CH
Pt
X
2

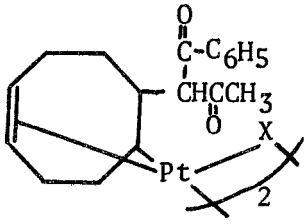
C-10


COOCH2CH3
CHCCH3
O
X
Pt
2

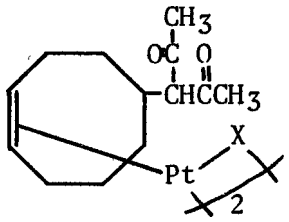
C-11


COOCH3
CHCOOCH3
X
Pt
2

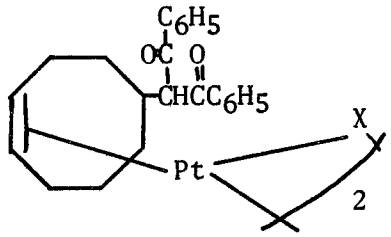
C-12


O
C-C6H5
CHCCH3
O
X
Pt
2

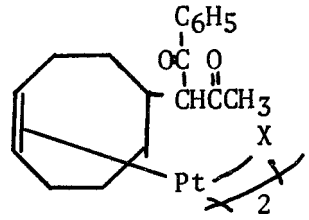
C-13



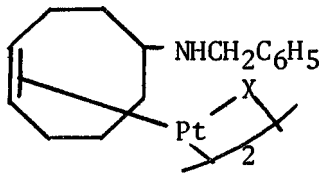

C-14



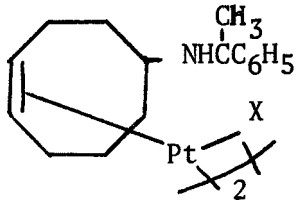

C-15



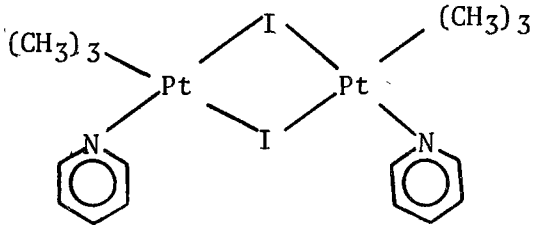

C-16



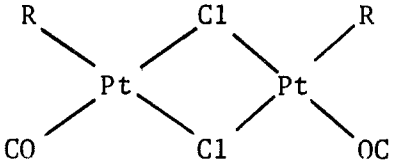

C-17



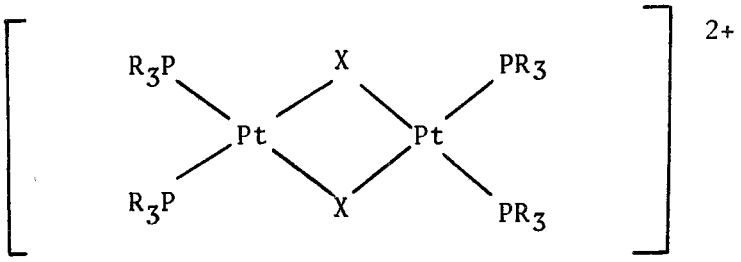
C-18




C-19




C-20



C-1

$Pt_2Cl_2(C_6H_4CH_2N(CH_3)_2)_2$, di-mu-chlorobis(N,N-dimethylbenzylamine-2-C,N) diplatinum(II)

Cope and Friedrich[21] determined the structure of the complex on the basis of interpretation of ir, nmr, uv, visible and mass spectra.

The authors reported their method of preparation of the white needle crystals as the reaction of potassium tetrachloroplatinum(II) (4.15 g) in 50 ml of water and 2.7 g of N,N-dimethylbenzylamine in 20 ml of methanol. After 45 hours at room temperature, the initially clear red solution became colorless and precipitated 3.15 g of brown solids. These solids were extracted with 200 ml of boiling benzene and filtered to remove 1.60 g of brown insoluble material. The benzene was removed on a rotary vacuum evaporator. Chromatography of the brown residue through a short silicic acid column with chloroform as the solvent, produced 1.0 g (28%) of pale yellow crystals. The crystals were purified by dissolving them in 100 ml of boiling benzene. The benzene solution was diluted with 150 ml of n-hexane and cooled to obtain 0.85 g of dull white needle crystals; mp 225-227° decomposed.

---

[21]A. C. Cope and E. C. Friedrich, J. Amer. Chem. Soc. 90, 909-13 (1968).

Anal. Calcd: C, 29.63; H, 3.29; N, 3.84; Pt, 53.5; Cl, 9.74. Found: C, 29.99; H, 3.28; N, 3.69; Pt, 53.3; Cl, 10.01.

C-2

$Pt_2Cl_2(C_4H_8NH_3ClNHNH_2)_2PtCl_4H_2O$, di-mu-chlorobis (diethylenetriamine)diplatinum(II)platinumtetrachloride monhydrate

Watt and Cude[22] postulated the structure on the basis of chemical evidence and interpretation of the ir spectrum. The position of the monohydrate was not assigned.

They reported the synthesis of the complex as the reaction of a suspension of 17.6 g of $K_2PtCl_6$ in 150 ml of water which was treated with 1.90 g of $N_2H_4 2HCl$ and stirred for one hour or until evolution of nitrogen subsided. This solution was stirred and heated slowly to 90° to form a clear, deep-red solution. After cooling to 5°, the solution was filtered and there was no evidence of the presence of either elemental platinum or unreacted $K_2PtCl_6$. The filtrate was adjusted to pH 7 with 20% KOH solution, a twofold excess of diethylenetriamine 3HCl (15.4 g) was added, the volume was increased to 300 ml with water, and this

---

[22]G. W. Watt and W. A. Cude, Inorg. Chem. 7, 335-8 (1968).

mixture was refluxed for 8 hours. The solution was cooled to 5° to provide orange crystals which were filtered and washed with cold (5°) water. The crystals were dried in vacuo over KOH for 2 hours. The yield was 6.0 g or 46% based on $K_2PtCl_6$. Anal. Calcd: C, 8.77; H, 2.76; Cl, 25.90; Pt, 53.44; Found: C, 8.85: H, 2.80; Cl, 25.6; Pt, 53.36.

Recrystallization of the complex from 1M HCl resulted in the formation of red crystals. The red and orange crystals yielded the same analytical data as well as the same ir spectrum and X-ray powder pattern which implied identical structures.

C-3

$Pt_2Cl_2(C_6H_4N_2C_6H_5)_2$, di-mu-chlorobis(N-phenylazobenzene) diplatinum(II)

Cope and Friedrich[21] reported the structure of the complex, however; they did not discuss their structural determination.

Cope and Siekman[23] reported the method of preparation as the reaction of azobenzene and potassium tetrachloroplatinum(II) in a mixture of dioxane and water. The mixture was kept at room temperature for two weeks.

---

[23]A. C. Cope and R. W. Siekman, J. Amer. Chem. Soc. 90, 3272 (1965).

A dark maroon crystalline product was formed when the solution was evaporated to dryness and the residue was extracted with benzene. The yield was 48% and decomposed at about 270°.

C-4

$Pt_2Cl_2(C_6H_4N_2)_2$, di-mu-chlorobis(azobenzene-2-C,N') diplatinum(II)

Cope and Siekman[24] disclosed in their patent the structure of the complex, however; the method of structural determination was not noted.

The complex was prepared by the reaction of 1.05 g azobenzene and 2.34 g of potassium tetrachloroplatinum(II) in 110 ml of dioxane and 110 ml of water. The reaction mixture was kept at room temperature for two months. The mixture was evaporated to dryness and the residue was repeatly extracted with benzene until the benzene was no longer colored. The combined extracts were concentracted by evaporation, the yield was reported as 1.28 g of product.

C-5

$Pt_2Cl_2(C_{10}H_{12}CH(CH_3CH_2O_2C)_2)_2$, di-mu-chlorobis(6-(dicarboxymethyl)-3a,4,5,6,7,7a-hexahydro-4,7-methan-

---

[24]A. C. Cope, and R. W. Siekman, U. S. Patent 3424739 (1969); Chem. Abstr., 70, P68529e (1969).

oinden-5-yl)diplatinum(II)

Stille and Fox[25] made the structural assignment on the basis of molecular weight determination and nmr spectra interpretation.

The complex was prepared by the reaction of dichloro(endo-dicyclopentadiene)platinum(II) and diethyl malonate in the presence of $Na_2CO_3$.

C-6,7,8,9

$Pt_2Cl_2(C_7H_8OCH_3)_2$, di-mu-chlorobis(exo-6-methoxy-2-norbornene-endo-5 simga, 2pi)diplatinum(II)

$Pt_2Cl_2(C_9H_{15}O)_2$, di-mu-chlorobis(8-methoxy-para-menth-1ene-9simga, 1pi) diplatinum(II)

$Pt_2X_2(C_9H_{15}O)_2$, X= Br, Cl, I; di-mu-bromobis(8-methoxy-4-cycloocten-1-yl)diplatinum(II)

$Pt_2X_2(C_{11}H_{15}O)_2$, X= Br, Cl, I; di-mu-bromobis(3a,4,5,6,7,7a-hexahydro-6-methoxy-4,7-methanoinden-5-yl)diplatinum(II)

The structure of the complexes were determined by Stille and Morgan[26] on the basis of chemical analyses,

---

[25]J. K. Stille and D. B. Fox, Inorg. Nucl. Chem. Lett. 5, 157-61 (1969); Chem. Abstr., 70, 115311q (1969).

[26]J. K. Stille and R. A. Morgan, J. Amer. Chem. Soc. 88, 5135-41 (1966).

nmr and ir spectra and the reduction of the complexes to known or independently synthesized ethers.

The complex, C-6, was prepared by refluxing a solution of dichloro(norborandiene)diplatinum(II) (0.86 g) and sodium carbonate (0.26 g) in 40 ml of methanol for 30 minutes. The resulting solution was filtered and evaporated to dryness. Recrystallization of the residue from methylene chloride yielded 0.42 g (59%) of product with mp, 134-7°. Anal. Calcd; C, 27.16; H, 3.14. Found: C, 27.45; H, 2.87.

The complex C-7 was prepared by the method reported by Chatt, et al.[27]

Complex C-8 was prepared by the method as reported by Stille, et al.[28]

C-10,11

$Pt_2X_2(C_{14}H_{17}O_3)_2$, X= Br, Cl; di-mu-bromobis(8-(1-carboxyacetonyl)pi-4-cycloocten-1-yl)diplatinum(II)

$Pt_2X_2(R)_2$, R= $C_{13}H_{19}O_4$, $C_{15}H_{21}O_4$, X= Br, Cl; di-mu-bromobis(8-(dicarboxymethyl)-pi-cycloocten-1-yl)diplatinum(II)

---

[27]J. Chatt, M. L. Vallarino and L. M. Venanzi, J. Chem. Soc. Sect. A, 2496, 3413 (1957).

[28]J. K. Stille, R. A. Morgan, D. D. Whitehurst and J. R. Doyle, J. Amer. Chem. Soc. 87, 3282 (1965).

Takahashi and Tsuji[29] described the complex on the basis of a molecular weight determination to show the dimeric character and nmr spectra to evaluate and assign structural features. Degradation of the complexes was an important part of their analyses.

The preparation of $Pt_2Cl_2(C_{15}H_{21}O_4)_2$ was reported as the reaction of $C_8H_{12}Pt_2Cl_2$ (0.7 g) with anhydrous sodium carbonate (1.5 g) and ethyl malonate (3ml) in an excess of ether. The mixture was allowed to stand at room temperature for 24 hours with stirring. The complex which formed was collected by filtration. The product was washed with water and ether, and dried in a desiccator. The chloroform insoluble complex ($C_8H_{12}Pt_2Cl_2$) was removed and the crystallization of the product was accomplished from a mixture of chloroform and ether. The resulting crystals were needle-like, mp 195-7°. The other complexes were prepared in a similar manner.

C-12,13,14,15

$Pt_2X_2(C_{18}H_{21}O_2)_2$, X= Br, Cl, I, SCN; di-mu-bromobis (8-(alpha-acetylphenacy)-pi-4-cycloocten-1-yl)diplatinum(II)

---

[29]H. Takahashi and J. Tsuji, ibid. 90, 2387-92 (1968).

$Pt_2X_2(C_{13}H_{19}O_2)_2$, X= Br, Cl, I, SCN; di-mu-bromobis (8-(1-acetylacetoyl)-pi-cycloocten-1-yl)diplatinum(II)

$Pt_2X_2(C_{23}H_{23}O_2)_2$, X= Br, Cl, I, SCN; di-mu-bromobis (8-(dibenzoylmenthyl)-pi-4-cycloocten-1-yl)diplatinum(II)

$Pt_2X_2(C_{15}H_{23}O_2)_2$, X= Br, Cl, I, SCN; di-mu-bromobis (8-(acetylacetoyl)-pi-cyclodecen-1-yl)diplatinum(II)

The structures of these complexes were assigned by Johnson, et al.[30] on the basis of ir, nmr and mass spectra and molecular weight determinations. The authors noted that much of the information leading to the structural determinations was the result of studies of the degradation products of the complexes.

All of the complexes were prepared by methods similar to that reported by Chatt, et al.[31]

C-16-17

$Pt_2Cl_2R_2$, R= $C_{15}H_{20}N$, $C_{16}H_{22}N$; di-mu-chlorobis(8-(benzylamino)-4-cycloocten-1-yl)diplatinum(II) and di-mu-chlorobis(8-((alpha-methylbenzl)amino)-4-cycloocten-1-yl)diplatinum(II)

Paiaro, et al.[32] reported the structures on the

---

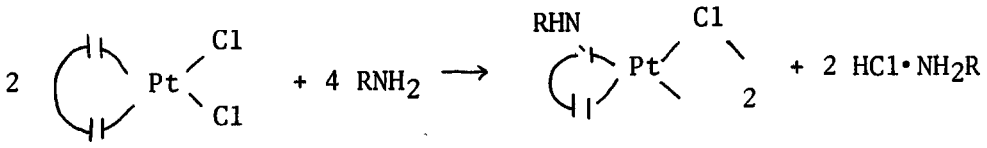
[30]B. F. G. Johnson, J. Lewis and M. S. Subramanian, J. Chem. Soc. Sect. A, 1993-2001 (1968).

[31]J. Chatt, M. L. Vallarino and L. M. Venanzi, ibid. 2496 (1954).

[32]G. Paiaro, A. D. Renzi and R. Palumbo, Chem. Commun. 1150-1 (1967).

basis of ir and nmr spectra and molecular weight determinations. The decomposition of the complexes was important in characterization of the structures.

The complexes were prepared according to the general reaction,



C-18

$Pt_2I_2((CH_3)_3C_5H_5N)_2$, di-mu-iodohexamethylbis(pyridine) diplatinum(II)

The patent abstract[33] did not give information concerning structural determination or method of preparation.

C-19

$Pt_2Cl_2(COR)_2$, R= $CH_3CH_2$, $C_8H_{17}$, $C_{12}H_{25}$; di-mu-chlorobis (carbonyl)diethyldiplatinum(II)

Lodewijk and Wright[34] assigned the structures on the interpretation of ir and nmr spectra and chemical evidence.

---

[33]German Patent 1259888 (1968); Chem. Abstr. 68 P695946d (1968).

[34]E. Lodewijk and D. Wright, J. Chem. Soc. Sect. A, 119-22 (1968).

The preparation of the complex when R= $CH_3CH_2$ was reported as the reaction of dichloroplatinum(II) (2.66g) and lithium chloride (0.85 g) in a mixture of dimethylformide (30 ml) and formic acid (10 ml). The solution was shaken for 2.5 hours at 100° in ethylene (40 atm). After filtration, the mixture was diluted with chloroform (2 x 25 ml) which was washed with water, dried and evaporated at less than 50°. The residue was dissolved in 2-methylbutane, washed with water, dried and evaporated. The yield was 1.4 g, 48%, mp 47-50°. Anal. Calcd for $C_6H_{10}Cl_2O_2Pt_2$: C, 12.6; H, 1.7. Found: C, 12.3; H, 1.5.

The preparation of the complex for R= $C_8H_{17}$ was the reaction of dichloroplatinum(II) (2.66 g) and lithium chloride (0.85 g) in a mixture of dimethylformide (30 ml), formic acid (5 ml) and oct-1-ene (7.5 ml) at 80°. The reaction mixture also included 0.09 g of potassium formate in 5 ml of formic acid. The mixture was kept at 100° for 2.75 hours and then the solution was filtered, diluted with water (100 ml) and extracted with 2-methylbutane ( 2 x 25 ml). The extract was washed with water, dried and concentrated. The excess octene was removed at less than 50°. A thick oil remained as residue (1.42 g). White crystals, mp 42°, slowly crystallized from the oil. Anal.

Calcd for $C_9H_{17}Cl_2O_2Pt_2$: C, 29.05; H, 4.6; Cl, 9.4.
Found C, 28.7; H, 4.9; Cl, 9.1.

A similar procedure was used for R= $C_{12}H_{25}$ except dodec-1-ene (7.4 g) was used instead of octene.
Anal. Calcd for $C_{12}H_{17}Cl_2O_2Pt_2$: C, 36.5; H, 5.85.
Found: C, 36.5; H, 5.9.

C-20

$Pt_2X_2(R_3P)_2$, X= Br, Cl, I and R= $CH_3CH_2$, $CH_3CH_2CH_2CH_2$, $C_6H_5$; di-mu-bromotetrakis(triethylphosphine)diplatinum(II) di(borontetrafluoride)

Clark, et al.[35,36] assigned the structures for the complexes for R= $CH_3CH_2$ on the basis of interpretation of ir and nmr spectra and molecular weight determinations. A structural determination of the chloro- complex by single crystal X-ray diffraction agreed with the ir and nmr studies.

The preparation of the three complexes for R= $CH_3CH_2$ was reported as being from the reaction of cis-($PtX_2((CH_3CH_2)_3P)_2$) (0.3 g) and boron trifluoride (0.3 g). The reaction mixture was heated for 12 hours at 50° in chloroform. The complexes were recrystallized from methanol and diethyl ether.

---

[35]H. C. Clark, K. R. Dixon and W. J. Jacobs, J. Amer. Chem. Soc., 91, 1346-50 (1969).

[36]H. C. Clark, K. R. Dixon and W. J. Jacobs, ibid., 90, 2259-66 (1968).

The structures of the complexes for R= $CH_3CH_2CH_2CH_2$ were assigned by Druce, et al.[37] Their interpretation was based on elemental analyses, conductivity measurements and spectra of ir, $^{11}B$ nmr and $^{31}P$ nmr.

The following reaction was given as the reaction route.

$$\underline{cis}\text{-}(PtX_2((C_3H_7)_3P)_2 + BX_3 = ((C_3H_7)_3P)_2Pt\langle^{X}_{X}\rangle Pt((C_3H_7)_3P)_2^{2+} + 2(BF_4)^-$$

Duddell, et al.[38] assigned the structure of the complexes for R= $C_6H_5$ on the basis of interpretation of ir and Raman spectra. The various stretching frequencies were correlated to the structural feactures.

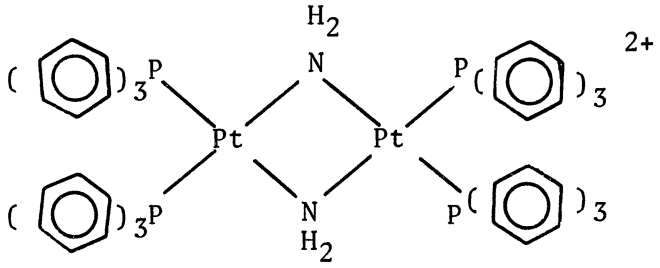
The method of preparation was reported to be similar to the procedure used for R= $CH_3CH_2$.[36]

---

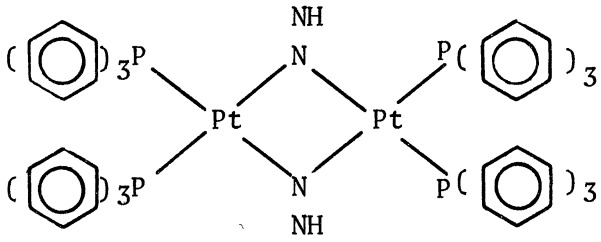
[37]P. M. Druce, M. F. Lappert, P. N. K. Riley, Chem. Commun., 486-7 (1967).

[38]D. A. Duddell, P. L. Goggin, R. J. Goodfellow and M. G. Norton, ibid., 879-81 (1968).

D-1



D-2



D-3

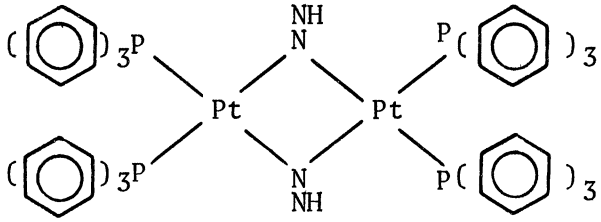
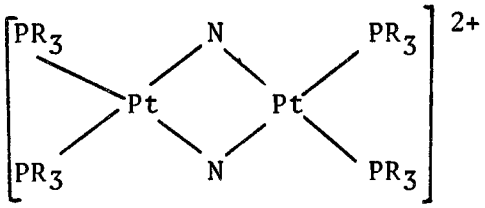


Figure 3

Structures of Complexes Type D

The Type D consisted of nitrogens in bridging positions and four tert-phosphines in the terminal positions. The type had the following general structure.




D-1,2,3,

$Pt_2(NH_2)_2(P(C_6H_5)_3)_4^{2+}$, di-mu-aminotetrakis(triphenylphosphine)diplatinum(II) ion

$Pt_2(N_2H)_2(P(C_6H_5)_3)_4^{2+}$, bis(mu-diazenotetrakis(triphenylphosphine)diplatinum(II) ion

$Pt_2(NH_2)(N_2H)(P(C_6H_5)_3)_4^{2+}$, mu-amido(mu-diazeno)tetrakis(triphenylphosphine)diplatinum(II) ion

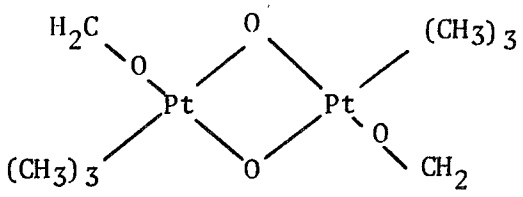
Dobinson, et al.[39] discussed the structural assignments of the complexes on the basis of interpretation of ir and nmr spectra and single crystal X-ray diffraction. The crystals were reported to be monoclinic.

Detailed preparation procedures were not given.

---

[39]G. C. Dobinson, R. Mason, G. B. Robertson, R. Ugo, F. Conti, D. Morelli, S. Cenini and F. Bonati, ibid., 739-41 (1967).

E-1




E-2

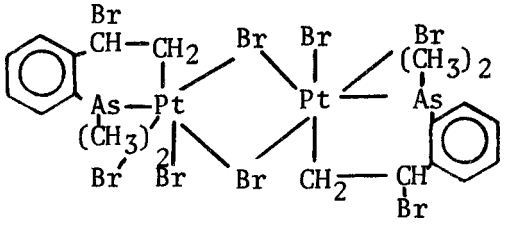



Figure 4

Structures of Complexes Type E

The structures for Type E were collected in Figure 4. The classification consisted of platinum(II) and platinum(IV) with halide and oxygen bridging.

E-1

$Pt_2O_2((CH_3)_3C_6H_4CH_2O)_2$, hexamethylbis(mu-salicylaldehydato)diplatinum(IV)

Truter and Watling[40] reported the structure of this platinum(IV) oxygen bridged complex on the basis of nmr spectra and single crystal X-ray diffraction. The crystals were found to be triclinic.

The authors did not prepare the complex, but noted that the crystals were a gift from the collection of R. C. Menziew. No reference was given to indicate how Menziew prepared the crystals.

Pidcock and Waterhouse[41] reported that the three complexes, bis(mu-dihydrogen phosphonate)bis(dihydrogen phosphonato)bis(triethylarsine)diplatinum(II) dichloride bis(mu-dihydrogen phosphonato)tetrakis(triethylphosphine)diplatinum(II)dichloride and bis(phosphinic acid)di-mu-phosphonbis(triethylarsine)diplatinum(II)dichloride, were platinum(II) oxygen bridged complexes. However

---

[40]M. R. Truter and R. C. Watling, J. Chem. Soc. Sect. A, 1955-63 (1967).

[41]A. Pidcock and C. R. Waterhouse, Inorg. Nucl. Chem. Lett. 3, 487-9 (1967); Chem. Abstr., 68, 45804k (1968).

the information contained in the abstract was not complete enough to determine the exact structures. The method of preparation was not explained either.

E-2

$Pt_2Br_2(C_6H_4As(CH_3)_2C_2H_3Br_2)_2$, di-mu-bromotetrabromobis (ortho-dimethylarsino)-beta-bromoethylbenzenediplatinum(IV)

The platinum(IV) halide bridged complex was reported by Bennett, et al.[42] Their structural interpretation was based on ir and nmr spectra as well as X-ray powder patterns.

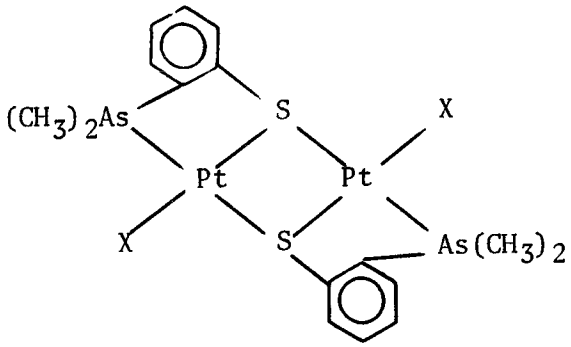
The authors did not discuss the method of preparation, but noted that the procedure had been given by Bennett, et al.[43]

---

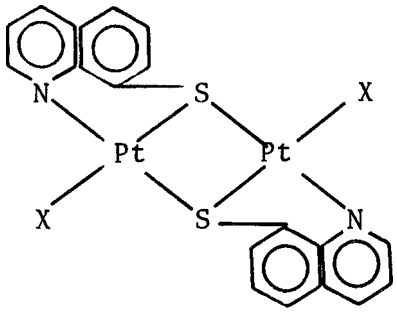
[42]M. A. Bennett, G. J. Erskine and R. S. Nyholm, J. Chem. Soc. Sect. A, 1260-3 (1967).

[43]M. A. Bennett, J. Chatt, G. J. Erskine, J. Lewis, R. F. Lomg and R. S. Nyholm, ibid., 501 (1967).

F-1




F-2




F-3

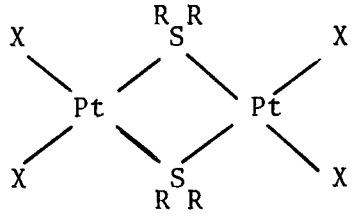
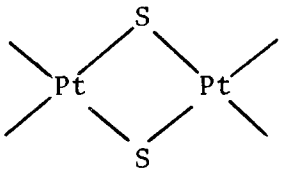



Figure 5

Structures of Complexes Type F

Type F consisted of those complexes with sulfide bridging. The type had the following general structure.




F-1

$Pt_2S_2(C_8H_{10}As)_2X_2$, X= Br, Cl, I, dibromobis(dimethyl-ortho-mu-thiolophenylarsine)diplatinum(II)

Lindoy, et al.[44] made the structural determination on the basis of chemical evidence and mass spectrum. The complex was decomposed to parts which could be identified by standard methods.

The three complexes were prepared by heating bis(dimethyl-ortho-methylthiophenylarsine)platinum(II) tetrabromoplatinum(II), tetrachloroplatinum(II) or diiodo(dimethyl-ortho-methylthiophenylarsine)platinum(II) in dimethylformide at the reflux temperature for eight hours. The bromo- complex deposited as yellow crystals (yield, 60%). Anal. Calcd: C, 19.7; H, 2.1. Found C, 20.0; H, 2.3. The chloro- complex yielded yellow crystals (yield, 40%) Calcd: C, 21.7; H, 2.3; Pt, 44.0. Found C, 21.55;

---

[44]L. F. Lindoy, S. E. Livingstone and T. N. Lockyer, Inorg. Chem. 6, 652-6 (1967).

H, 2.3; Pt, 43.9. A 70% yield of the iodo- complex was obtained as yellow crystals. Calcd: C, 18.0; H, 1.9; Pt, 36.5. Found: C, 18.1; H, 2.0; Pt, 36.5.

F-2

$Pt_2(S_2C_9H_6N)_2X_2$, X= Br, Cl; dibromodi(8-mu-thio quinoline)diplatinum(II)

Lindoy, et al.[44] reported the structure of these complexes on the basis of interpretation of chemical analyses and mass spectra.

The method of preparation was reported as the reaction of dibromo(8-methylthioquinoline)platinum(II) (0.5 g) in 50 ml of dimethylformide. The reaction mixture was heated at reflux temperature for two hours. The solution was cooled and filtered. Orange crystals (0.4 g, 95%) were deposited. Anal. Calcd for Pt $(S_2C_9H_6N)_2Br_2$: C, 24.8; H, 1.4; Pt, 44.8. Found: C, 25.1; H, 1.5; Pt, 44.1.

The same method was reported for the chloro-complex except dichloro(8-methylthioquinoline)platinum(II) was used in place of the dibromo- complex. Anal.Calcd for $Pt_2S_2(C_9H_6N)_2Cl_2$: C, 27.7; H, 1.55; Pt, 49.9. Found C, 27.6; H, 1.75; Pt, 49.8.

F-3

$Pt_2(SR_2)_2X_4$, R= $CH_3$, $CH_3CH_2$, $CH_3CH_2CH_2$, $CH_3CH_2CH_2CH_2$, X= Br, Cl; tetrabromobis(mu-dimethyl sulfide))diplatinum(II)

The structures of these complexes have been discussed by several authors, Goggin, et al.[45], Sales, et al.[46] and Adams and Chandler.[14] The assignments of the structures was on the basis of interpretation of ir spectra, chemical analyses and single crystal X-ray diffraction. X-Ray data showed that the crystals were monoclinic and the ir spectra proved that the complexes had sulfide bridging.

The compounds were prepared by methods analogous to those reported by Chatt and Venanzi.[47]

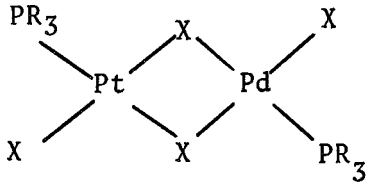
---

[45]P. L. Goggin, R. J. Goodfellow, D. L. Sales, J. Stokes and P. Woodward, Chem. Commun. 31-2 (1968).

[46]D. L. Sales, J. Stokes and P. Woodward, J. Chem. Soc. Sect. A, 1852-8 (1968).

[47]J. Chatt and L. M. Venanzi, ibid., 2787 (1955), 2351 (1957).

Only three compounds were classified as Type G. This type was very unusual when compared with the platinum complexes already discussed. Type G complexes had only one platinum atom. The bridging was between the one platinum and one palladium atom. The bridging groups were halides and two halides in the terminal positions. Two tert-phosphines were present in the other terminal positions. The complexes had the following general structure.




$PtPdX_2(PR_3)_2X_2$, X= Br, Cl for R= $C_4H_9$ and X= Cl for R= $C_3H_7$; di-mu-bromobromo(bromo(tributylphosphine)palladium) tributylphosphineplatinum(II)

The structure were reported in a Netherlands patent[48] and the abstract did not give information on how the structure was determined.

The method of preparation was given as the reaction of trans-$((C_4H_9)_3P)_2PtX_2$ (1.0 g) with 0.56 g of $Na_2PdX_4$ in 80 g ethanol. The reaction mixture was refluxed for one hour. The mixture was concentrated, hydrolyzed and

---

[48]Netherlands Patent 6611373 (1967); Chem. Abstr. 67, PC54266t (1967).

extracted with benzene. The bromo- complex had a mp 144-6°. The melting point of the chloro- complex was 145-6°. A similar procedure was used to make the choro- complex for R= $C_3H_7$, which had a melting point of 184-8°.

## Summary

The literature survey consisted of 122 different compounds which were reported to be dimeric mu-bridged complexes.

The methods of structural determination were mainly ir and nmr spectra together with chemical analyses. Only a few of the structures were determined by X-ray techniques.

Many of the authors noted that the structures were determined indirectly. Much of the data reported for ir and nmr spectra were of the degradation products of the complexes. The spectra of the complexes constituted the bulk of the structural determinations.

## Chapter II

### Introduction

In 1895, Wilhelm Roentgen accidentally observed that a screen coated with a fluoresent salt glowed everytime a cathode-ray tube was switched on. After much experimental work, Roentgen christened the unknown radiation as X-rays after the algebraic symbol for an unknown quanity.[1]

X-Rays can be produced by cathode-rays (fast moving electrons) being abruptly stopped. The more abruptly the electrons can be stopped, the more powerful will be the resulting X-rays.

The X-ray machine for this work employed a copper anode as the stop for the electrons. A thin nickel foil was used to filter the resulting beam of X-rays to obtain essentially monochromatic radiation.

The X-rays were directed through a collimator tube 0.5 mm in diameter. The beam was focused on a single crystal of the complex being studied. The crystal was mounted on the goniometer head of a Weissenberg camera. The photographs were developed using standard techniques.

---

[1]A. Beiser and K. Krauskopf, "Introduction to Physics and Chemistry", McGraw-Hill Book Company, New York, New York, 1964, p. 317-18.

## Experimental

A single crystal, approximately 0.01 mm in diameter, of the platinum complex was mounted in a glass capillary tube with inside diameter of approximately 0.1 mm diameter. Human hairs were used as tools to handle the crystals. A microscope was necessary to work with the crystals of such a small size. The single crystal was picked up with a human hair and inserted in the capillary tube. The crystal was then worked to the bottom of the tube using a long hair and by vibrating the sides of the tube. The bottom section of the tube which contained the crystal was broken off to a length of about one half of an inch. The length of capillary was then glued to a special mounting rod and secured with a set screw in the goniometer head.

The crystal was alined visually by use of crosshairs in a telescope mounted on the base of the Weissenberg camera.

Photographs, having a four hour exposure time, were taken of the crystal with the film cassette locked in the center position and the crystal oscillating 5-10°. The goniometer head was positioned so that the x-ray beam was alined at a 45° angle to the arc adjustments.

After developing, the film had rows of dots. One row

of these dots passed through the center of the film. This center row of dots was used to determine the adjustments necessary to obtain a properly alined crystal. The mark of proper alinement was when center row of dots passed through the center of the film and formed a straight line 45 mm on both sides of the film center. This straight line was perpendicular to the base line on the film. The base line was obtained by raising the beam stop and moving the film holder back and forth while the x-ray beam was striking the film.

After the crystal was properly alined, the photograph of the crystal consisted of several rows of dots similar to Figure 6. The center row was arbitrarily called the zero level. The equally spaced rows on each side of the center were numbered starting with one, two, etc.

The dots were the result of the X-ray beam being diffracted from the crystal. The diffraction obeyed Bragg's Law, n lambda = 2 d sin theta. The level number was denoted by n and the wavelength by lambda. The distance d indicated one cell dimension and theta was used to denote the angle of diffraction. The dots on the photograph were indicative of the reciprocal lattice, a reflection of the real lattice within the crystal. The direct and reciprocal lattice

of an orthorhombic crystal has been presented in Figure 7.

To obtain the zero level photograph, a screen was positioned to block all reflection other than those due to the zero level. The sled drive was engaged and the camera mechanism was set to cause a crystal oscillation of about 240°. The gear train of the camera was such that 1 mm of film translation produced two degrees of crystal rotation. When the film cassette would reach each end of the sled drive screw, an automatic switch would reverse both the sled movement and the direction of rotation of the crystal. A typical photograph has been represented in Figure 8.

Two calculations were made using data taken from the alinement photograph. The objective of one computation was to determine the necessary camera adjustments to obtain upper level photographs. The second calculation involved measurement of one cell dimension.

The first and second level photographs were obtained in a manner similar to the zero level. Two adjustments were necessary to obtain these upper level photographs. The tilt angle, mu, and the screen shift, s, were calculated from the data obtained from the oscillation photograph using the following relationships.

$s = r \tan mu$, r = 25 mm, radius of the screen, mu = tilt angle

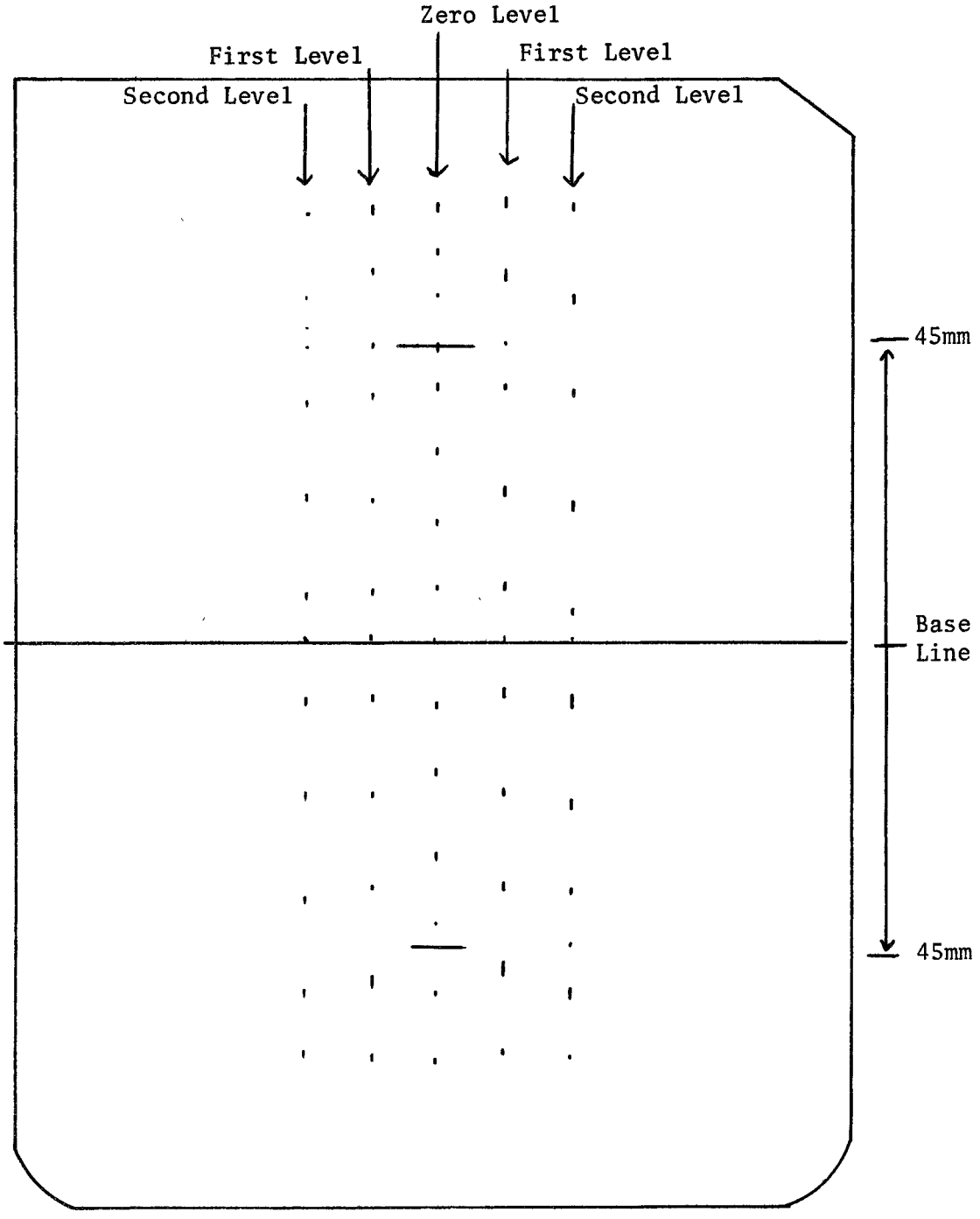



Figure 6

Alinement Photograph

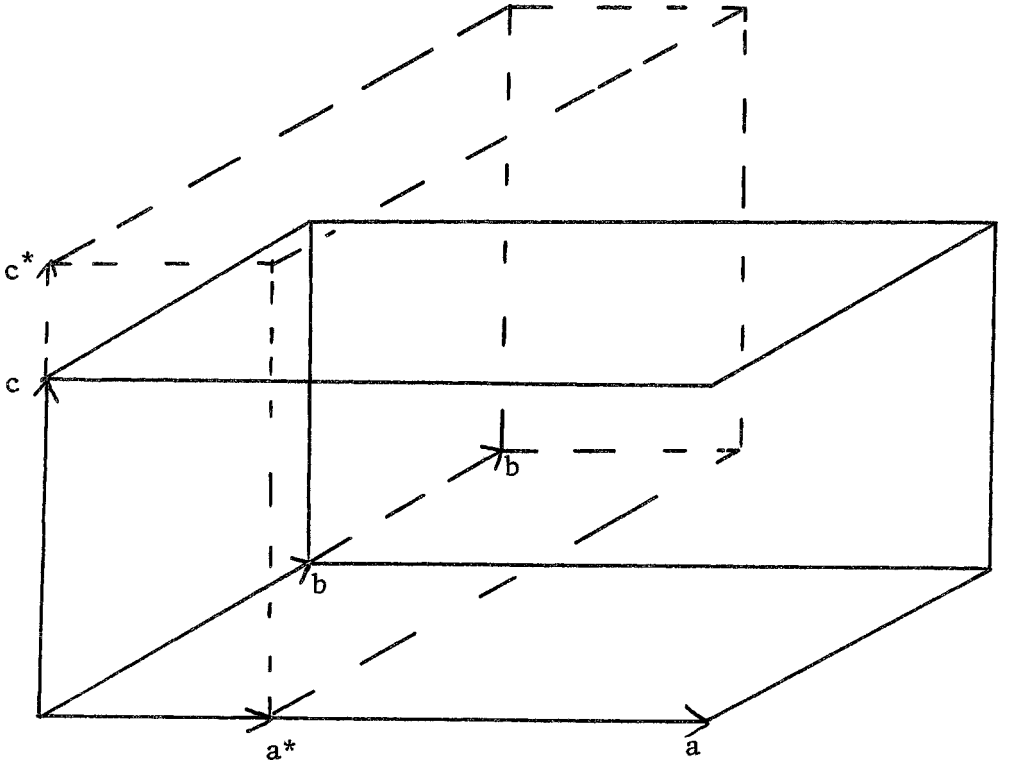



Figure 7

Orthorhombic direct and reciprocal unit cells.[2]

---

[2]G. H. Stout and L. H. Jensen, "X-ray Structure Determination", The Macmillan Company, New York, New York, 1968, p. 28.

sin mu =(sin tau)/ 2

tau° = 2L/D, 2L = distance in mm of corresponding dots on alinement photograph, D = 60 mm, film diameter

The calculation of one cell dimension was based upon data from the oscillation photograph. The values 2L and D were employed to compute the cell parameter according to the example given.

2L = 18.9 mm, D = 60 mm

D/2L = 3.175 = tan z, z° = 72° 31', cos z = 0.30043

n lambda = 1.5418 Å for first level

n lambda/cos z = 5.126 Å

Special note was made that these calculations were used only as guides. The actual adjustments were made after checking for proper alinement and the actual cell dimensions reported later were calculated by the IBM computer.

To use the computer for calculation of the actual cell dimensions, indexing of the photographs was necessary. Twenty-five data points were taken from each of three photographs, zero, first and second levels. These data points consisted of h,k and l values and a distance theta in millimeters. The l value refered to the level number on the various photographs. Each photograph, such as Figure 8, had what was called two zero lines, actual rows

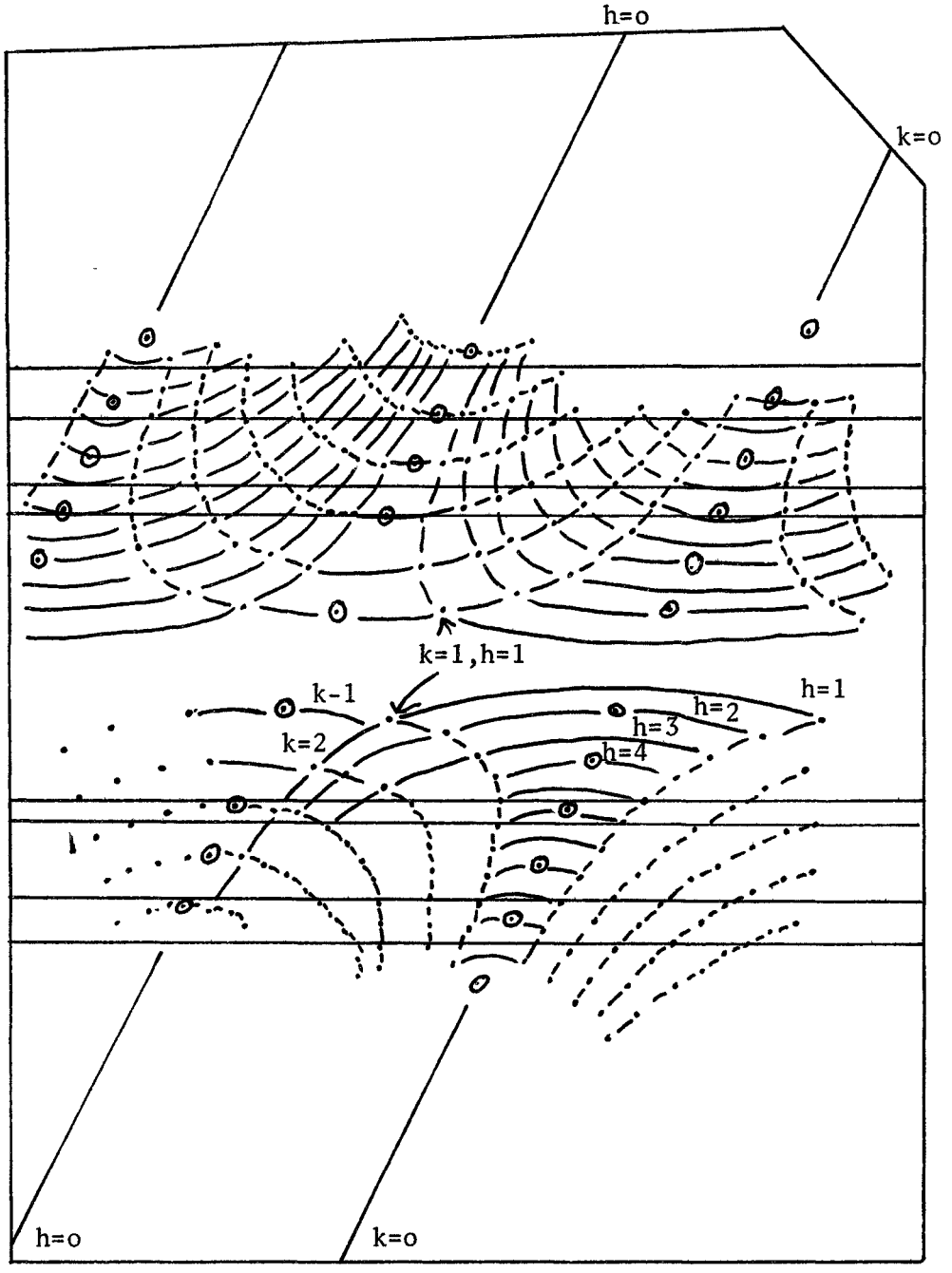



Figure 8

Zero Level Weissenberg Photograph

of dots that formed lines. These zero lines were labeled k for the right one and h for the one to the left of k. The two lines were parallel to one another and formed a 63.4° angle to the base of the film. Next to the zero line, was a series of lines, called festons, which started close to the zero line near the top and bottom of the film. As the lines approached the center of the film, they departed from the one zero line and formed "U" shaped festons approaching the other zero line near the top and bottom of the photograph. The feston lines were numbered starting with the straight line as zero and the first line 1, etc. At each point where h and k lines crossed, that point had three index numbers, l = level number, h = number of h line and k = the number of the k line. The distance theta = ½ the distance between corresponding points on the top and bottom of the photograph.

The film cassette was designed to accomodate several sheets of film but all the data obtained throughout this investigation was recorded on single sheets. An aluminum powder standard was superimposed over the zero level photograph of each crystal. The aluminum powder produced a series of horizontal lines. The distance between these lines was measured and the true diameter of the film was calculated. The

distance between the first set of aluminum lines was multiplied by 1.48796, the second by 0.73166, the third by 0.87934 and the fourth by 0.73166. The four values were averaged and the result was used an input data for the computer program.

The other cell dimension computation used data taken from the zero level photograph as follows,

n lambda/ 2 sin theta = 3.0836 Å / 0.22814 - 13.50 Å

theta - 6.55° = ½( 2L ) = ½(13.1 mm)

sin theta = 0.11407, 2 sin theta = 0.22814

These calculations were based on Bragg's Law and were explained in detail by Stout and Jensen.[1]

The data from the photographs of the complexes [Pt(dien)Cl] Cl, [Pt(dien)Br] Br and [Pt(dien)SCN] I, where dien = diethylenetriamine, were key-punched and analyzed by an IBM 1130 computer program designed to calculate the unit cell parameters. The computer program was adapted from a similar one obtained from the University of Arkansas.

All the data used to determine the unit cell parameters was recorded in Appendix III.

Attempts were made to mount and photograph other complexes of platinum, [Pt(dien)I] I, [Pt(dien)SCN] SCN [Pt(dien) $NO_2$] I and $Pt_3C_8H_{30}ON_6Cl_8$ (see C-2 red and orange forms. Each attempt failed because the crystals were too small, as was the case for [Pt(dien)I] I or,

the crystals were not single. When two or more crystals were stuck together, reflections were obtained from each crystal and could not be used.

Results and Discussion

The computer program was used to calculate the unit cell dimensions and the volume of the unit cell. The results of the calculations were recorded in Table III.

The unit cell dimensions of the three crystals followed the expected trend. The complexes were almost identical except for two chlorine ions in one, two bromine ions in another and thiocyanate-iodine in the third. The complexes were expected to increase in size in the order listed. The expected trend was based on the increasing size of the unlike atoms.

Twenty-five data points were measured for each of the three photographs of each crystal. The data was very consistent for the chloride complex. The bromide complex had two data points on the zero level which had deviations greater than 1.0 from the theoretical value. These data were rejected from the final analyses. The thiocyanate-iodide complex had seven data points rejected, four on the zero level, one on the first level and two on the second level.

In the final analyses all the data points had a deviation of less than 1.0 and a least squares deviation of less than 2.0.

Table III

Results of Computer Analyses

| Complex | Dimensions in Å | | | Volume |
|---|---|---|---|---|
| | A | B | C | cubic Å |
| [Pt(dien)Cl]Cl | 13.157 | 13.586 | 4.816 | 855 |
| [Pt(dien)Br]Br | 13.726 | 13.780 | 4.957 | 935 |
| [Pt(dien)SCN]I | 12.245 | 18.726 | 5.279 | 1210 |

The specific reason for each deviation was not determined. Errors of the measurements were minimized by the fact that the actual photographs were $10^7$ times larger than the corresponding distances within the crystal.

Summary

The three platinum complexes were successfully mounted, photographed, indexed and unit cell parameters calculated by use of the computer program. The resulting cell dimensions corresponded to expected trends.

## Appendix I

### Complexes Listed According to Type

Type A

di-mu-bromotetrabromodiplatinum(II)bis(tetraethylammonium)*

di-mu-chlorotetrachlorodiplatinum(II)bis(tetraethylammonium)*

di=mu-iodotetraiododiplatinum(II)bis(tetraethylammonium)*

Type B

di-mu-bromodibromobis(1,2-bis(isopropylseleno)ethane))diplatinum(II)*

di-mu-bromodibromobis(ethylene)diplatinum(II)*

di-mu-bromodibromobis(tributylphosphine)diplatinum(II)*

di-mu-bromodibromobis(triethylarsine)diplatinum(II)*

di-mu-bromodibromobis(triethylphosphine)diplatinum(II)*

di-mu-bromodibromobis(trimethylarsine)diplatinum(II)*

di-mu-bromodibromobis(trimethylphosphine)diplatinum(II)*

di-mu-bromodibromobis(triphenylphosphine)diplatinum(II)*

di-mu-bromodibromobis(tripropylarsine)diplatinum(II)*

di-mu-bromodibromobis(tripropylphosphine)diplatinum(II)*

---

*palladium analog reported

## Appendix I

### Complexes Listed According to Type

Type A

di-mu-bromotetrabromodiplatinum(II)bis(tetraethylammonium)*

di-mu-chlorotetrachlorodiplatinum(II)bis(tetraethylammonium)*

di=mu-iodotetraiododiplatinum(II)bis(tetraethylammonium)*

Type B

di-mu-bromodibromobis(1,2-bis(isopropylseleno)ethane))diplatinum(II)*

di-mu-bromodibromobis(ethylene)diplatinum(II)*

di-mu-bromodibromobis(tributylphosphine)diplatinum(II)*

di-mu-bromodibromobis(triethylarsine)diplatinum(II)*

di-mu-bromodibromobis(triethylphosphine)diplatinum(II)*

di-mu-bromodibromobis(trimethylarsine)diplatinum(II)*

di-mu-bromodibromobis(trimethylphosphine)diplatinum(II)*

di-mu-bromodibromobis(triphenylphosphine)diplatinum(II)*

di-mu-bromodibromobis(tripropylarsine)diplatinum(II)*

di-mu-bromodibromobis(tripropylphosphine)diplatinum(II)*

---

*palladium analog reported

di-mu-chlorodichlorobis(1,2-bis(isopropylseleno) ethane))diplatinum(II)*

di-mu-chlorodichlorobis(butylene)diplatinum(II)*

di-mu-chlorodichlorobis(tert-butylmethylphenyl-phosphine)diplatinum(II)

di-mu-chlorodichlorobis(carbonyl)diplatinum(II)*

di-mu-chlorodichlorobis(cyclohexydiphenylphosphine) diplatinum(II)

di-mu-chlorodichlorobis(cyclooctene)diplatinum(II)

di-mu-chlorodichlorobis(dibutyldiphenylphosphine) diplatinum(II)

di-mu-chlorodichlorobis(dibutylphenylphosphine) diplatinum(II)

di-mu-chlorodichlorobis(dicyclohexyphenylphospine) diplatinum(II)

di-mu-chlorodichlorobis(diethylphenylphosphine) diplatinum(II)

di-mu-chlorodichlorobis(dimethylphenylphosphine) diplatinum(II)

di-mu-chlorodichlorobis(diphenylcyclopropenone) diplatinum(II)*

di-mu-chlorodichlorobis(dipropylphenylphosphine) diplatinum(II)

di-mu-chlorodichlorobis(ethylcyclohexene)diplatinum(II)

---

*palladium analog reported

di-mu-chlorodichlorobis(ethyldiphenylphosphine) diplatinum(II)

di-mu-chlorodichlorobis(ethylene)diplatinum(II)*

di-mu-chlorodichlorobis(ethyltelluride)diplatinum(II)*

di-mu-chlorodichlorobis(methyldiphenylphosphine) diplatinum(II)

di-mu-chlorodichlorobis(phosphorous acid)di-platinum(II)*

di-mu-chlorodichlorobis(phosphorous trichloride) diplatinum(II)*

di-mu-chlorodichlorobis(1-propen-1-ol)diplatinum(II)

di-mu-chlorodichlorobis(propyldiphenylphosphine) diplatinum(II)

di-mu-chlorodichlorobis(propylene)diplatinum(II)*

di-mu-chlorodichlorobis(para-toluidine)diplatinum(II)*

di-muchlorodichlorobis(tributylphosphine)di-platinum(II)*

di-mu-chlorodichlorobis(triethylarsine)diplatinum(II)*

di-mu-chlorodichlorobis(triethylphosphine)diplatinum(II)*

di-mu-chlorodichlorobis(trimethylarsine)diplatinum(II)*

di-mu-chlorodichlorobis(trimethylphosphine)di-platinum(II)*

di-mu-chlorodichlorobis(trimethyl(vinyloxy)silane) diplatinum(II)

---

*Palladium analog reported

di-mu-chlorodichlorobis(triphenylarsine)diplatinum(II)*

di-mu-chlorodichlorobis(triphenylphosphine)di-platinum(II)*

di-mu-chlorodichlorobis(tripropylarsine)diplatinum(II)*

di-mu-chlorodichlorobis(tripropylphosphine)di-platinum(II)*

di-mu-chlorodichlorobis(vinyl alcohol)di-platinum(II)

di-mu-iododiiodbis(tributylphosphine)diplatinum(II)*

di-mu-iododiiodbis(triethylarsine)diplatinum(II)*

di-mu-iododiiodbis(triethylphosphine)diplatinum(II)*

di-mu-iododiiodbis(trimethylarsine)diplatinum(II)*

di-mu-iododiiodbis(trimethylphosphine)diplatinum(II)*

di-mu-iododiiodbis(triphenylphosphine)diplatinum(II)*

di-mu-iododiiodbis(tripropylarsine)diplatinum(II)*

di-mu-iododiiodbis(tripropylphosphine)diplatinum(II)*

Type C

di-mu-bromobis(8-(acetylacetoyl)-pi-cycloden-1-yl) diplatinum(II)*

di-mu-bromobis(8-(1-acetylacetoyl)-pi-cycloocten-1-yl)diplatinum(II)*

di-mu-bromobis(8-(alpha-acetylphenacyl)-pi-4-cycloocten-1-yl)diplatinum(II)*

di-mu-bromobis(8-(1-carboxyacetonyl)-pi-4-cycloocten-1-yl)diplatinum(II)*

---

*palladium analog reported

di-mu-bromobis(8-dibenzoylmethyl)-pi-4-cycloocten-1-yl)diplatinum(II)*

di-mu-bromobis(3a,4,5,6,7,7a-hexahydro-6-methoxy-4,7-methanoinden-5-yl)diplatinum(II)*

di-mu-bromobis(8-methoxy-4-cycloocten-1-yl)diplatinum(II)*

di-mu-bromobistetrakis(tributylphosphine)diplatinum(II)di(borontetrafluoride)

di-mu-bromotetrakis(triethylphosphine)diplatinum(II)di(borontetrafluoride)

di-mu-bromotetrakis(triphenylphosphine)diplatinum(II)di(borontetrafluoride)

di-mu-chlorobis(8-(acetylacetoyl)-pi-cycloden-1-yl)diplatinum(II)*

di-mu-chlorobis(8-(1-acetylacetyl)-pi-cycloocten-1-yldiplatinum(II)*

di-mu-chlorobis(8-(alpha-acetylphenacyl)-pi-cycloocten-1-yl)diplatinum(II)*

di-mu-chlorobis(azobenzene-2-C,N')diplatinum(II)*

di-mu-chlorobis(8-(benzylamino)-4-cycloocten-1-yl)diplatinum(II)*

di-mu-chlorobis(carbonyl)diethyldiplatinum(II)

di-mu-chlorobis(8-dibenzoylmethyl)-pi-4-cyclo-octen-1-yl)diplatinum(II)*

---

*palladium analog reported

di-mu-chlorobis(6-(dicarboxymethyl)3a,4,5,6,7,7a-hexahydro-4,7-methanionden-5-yl)diplatinum(II)

di-mu-chlorobis(diethylenetriamine)diplatinum(II) platinumtetrachloride monohydrate

di-mu-chlorobis(N,N-dimethylbenzyamine-2-C,N) diplatinum(II)*

di-muchlorobis(3a,4,5,6,7,7a-hexahydro-6-methoxy-4,7-methanoinden-5-yl)diplatinum(II)*

di-mu-chlorobis(8-((alpha-methylbenyl)amino) -4-cycloocten-1-yl)diplatinum(II)*

di-mu-chlorobis(8-methoxy-4-cycloocten-1-yl) diplatinum(II)*

di-mu-chlorobis(8-methoxy-para-menth-1-ene-9simga,1pi)diplatinum(II)*

di-mu-chlorobis(exo-6-methoxy-2-norbornene-endo-5simga,2pi)diplatinum(II)*

di-mu-chlorobis(N-phenylazobenzene)diplatinum(II)

di-mu-chlorotetrakis(tributylphosphine)diplatinum(II) di(borontetrafluoride)

di-mu-chlorotetrakis(triethylphosphine)diplatinum(II) di(borontetrafluoride)

di-mu-chlorotetrakis(triphenylphosphine)diplatinum(II) di(borontetrafluoride)

di-mu-iodobis(8-(acetylacetoyl)-pi-cycloden-1-yl diplatinum(II)*

---

*palladium analog reported

di-mu-iodobis(8-(1-acetylacetoyl)-pi-cycloocten-1-yl) diplatinum(II)*

di-mu-iodobis(8-(alpha-acetylphenacyl)-pi-4-cycloocten-1-yl)diplatinum(II)*

di-mu-iodobis(8-dibenzoylmethyl)-pi-4-cycloocten-1-yl)diplatinum(II)*

di-mu-iodobis(3a,4,5,6,7,7a-hexahydro-6-methoxy-4,7-methanoinden-5-yl)diplatinum(II)*

di-mu-iodobis(8-methoxy-4-cycloocten-1-yl)diplatinum(II)*

di-mu-iodohexamethylbis(pyridine)diplatinum(II)

di-mu-iodotetrakis(triethylphosphine)diplatinum(II) di(borontetrafluoride)

di-mu-iodotetrakis(triphenylphosphine)diplatinum(II) di(borontetrafluoride)

di-mu-thiocyanatobis(8-(acetylacetoyl)-pi-cycloden-1-yl)diplatinum(II)*

di-mu-thiocyanatobis(8-(1-acetylacetoyl)-pi-cycloocten-1-yl)diplatinum(II)*

di-mu-thiocyanatobis(8-(alpha-acetylphenacycl-pi-cycloocten-1-yl)diplatinum(II)*

di-mu-thiocyanatobis(8-dibenzoylmethyl)-pi-4-cycloocten-1-yl)diplatinum(II)*

---

*palladium analog reported

Type D

mu-amido(mu-diazeno)tetrakis(triphenylphosphine) diplatinum(II)*ion

di-mu-aminotetrakis(triphenylphosphine)diplatinum (II)*ion

bis-mu-diazenotetrakis(triphenylphosphine)di-platinum(II)*ion

Type E

bis(mu-dihydrogen phosphonato)bis(dihydrogen phosphonato)bis(triethylarsine)diplatinum(II) dichloride

bis(mu-dihydrogen phosphonato)tetrakis(triethyl-phosphine)diplatinum(II) dichloride

bis(phosphonic acid)di-mu-phosphonbis(triethyl-arsine)diplatinum(II) dichloride

di-mu-bromotetrabromobis(ortho-dimethylarsino)-beta-bromoethylbenzenediplatinum(IV)

hexamethylbis(mu-salicylaldehydato)diplatinum(II)

Type F

dibromobis(dimethyl-ortho-mu-thiolophenylarsine) diplatinum(II)*

dibromodi(8-mu-thiolo-quinoline)diplatinum(II)*

tetrabromobis(mu-(dibutyl sulfide))diplatinum(II)*

tetrabromobis(mu-(diethyl sulfide))diplatinum(II)*

tetrabromobis(mu-(dimethyl sulfide))diplatinum(II)*

---

*palladium analog reported

tetrabromobis(mu-(dipropyl sulfide))diplatinum(II)*

dichlorobis(dimethyl-ortho-mu-thiolophenylarsine) diplatinum(II)*

dichlorodi(8-mu-thiolo-quinoline)diplatinum(II)*

tetrachlorobis(mu-(dibutyl sulfide))diplatinum(II)*

tetrachlorobis(mu-(diethyl sulfide))diplatinum(II)*

tetrachlorobis(mu-(dimethyl sulfide))diplatinum(II)*

tetrachlorobis(mu-(dipropyl sulfide))diplatinum(II)*

diiodobis(dimethyl-ortho-mu-thiolophenylarsine) diplatinum(II)*

Type G

di-mu-bromobromo(bromo(tributylphosphine) palladium)tributylphosphineplatinum(II)

di-mu-bromobromo(bromo(tripropylphosphine) palladium)tripropylphosphineplatinum(II)

di-mu-chlorochloro(chloro(tributylphosphine) palladium)tributylphosphineplatinum(II)

di-mu-chlorochloro(chloro(tripropylphosphine) palladium)tripropylphosphineplatinum(II)

---

*palladium analog reported

## Appendix II

### List of Standard Abbreviations

| | |
|---|---|
| Å | angstrom |
| °C | degree Celsius |
| g | gram |
| ml | milliliter |
| mm | millimeter |
| ir | infrared |
| nmr | nuclear magnetic resonance |
| mp | melting point |
| bp | boiling point |
| calcd. | calculated |
| et al. | and others |
| mmole | millimole |
| uv | ultraviolet |
| Anal. | analytical |

# BIBLIOGRAPHY

## Abstracts

German Patent 1259888 (1968); Chem. Abstr., 68, PC 95946d (1968).

Miller, R. G. J., Colloq. Spectrosc. Int., 12th, Exeter, 523-39 (1965); Chem. Abstr., 68, 17250h (1968).

Netherlands Patent 6611373 (1967); Chem. Abstr., 67, PC 54266t (1967).

Pidcock, A. and Waterhouse, C. R., Inorg. Nucl. Chem. Lett.3, 487-9 (1967); Chem. Abstr., 68, 45804k (1968)

Sorzano, J. P., and Fackler, J. P., J. Mol. Spectrosc. 22, 80-98 (1967).; Chem. Abstr., 66, 41996k (1967).

Stille, J. K. and Fox, D. B., Inorg. Nucl. Chem. Lett.5, 157-61 (1969); Chem. Abstr. 70, 115311q (1969).

Taylor, M. J. and Odell, A. L., Spectrochim. Acta, Part A, 24, 1855-61 (1968); Chem. Abstr.,69, 111664p (1968).

U.S. Patent 3424739 (1969); Chem. Abstr., 70, P 68529e (1969).

Wakatsuki, Y., Nozakura, S. and Murahashi, S., Bull. Chem. Soc. Jap., 42, 273 (1969); Chem Abstr., 70, 87942a (1969).

JOURNALS

Adams, D. M. and Chandler, P. J., J. Chem. Soc. Sect. A, 588-97 (1969).

Adams, D. M., Chandler, P. J. and Churchill, R. G., J. Chem. Soc. Sect. A, 1272-4 (1967).

Benkexer, R. A., Dunny, S., Li, G. S., Nerlekar, P. G. and Work, S. D., J. Amer. Chem. Soc. 90, 1871-5 (1968).

Bennett, M. A., Chatt, J., Erskine, G. J., Lewis J., Long, R. F. and Nyholm, R. S., J. Chem. Soc. Sect. A, 501 (1967).

Bennett, M. A., Erskine, G. J. and Nyholm, R. S., J. Chem. Soc. Sect. A, 1260-3 (1967).

Bird, C. W. and Brigg, E. M., J. Chem. Soc. Sect. A, 1004-6 (1967).

Chan, T. H., Chem. Commun., 895-6 (1968).

Chatt, J., Vallarino, M. L. and Venznzi, L. M., J. Chem. Soc. Sect. A, 2496 (1954).

Chatt, J., Vallarino. M. L., and Venanzi, L. M., J. Chem. Soc. Sect. A, 2496 (1957).

Chatt, J. and Venanzi, L. M., J. Chem. Soc. Sect. A, 2787 (1955).

Clark, H. C., Dixon, K. R. and Jacobs, W. J., J. Amer. Soc. 90, 2259-66 (1968).

Clark, H. C., Dixon, K. R. and Jacobs, W. J., J. Amer. Soc. 91, 1346-50 (1969).

Cope, A. C. and Friedrich, E. C., J. Amer. Chem. Soc. 90, 909-13 (1968).

Cope, A. C. and Siekman, R. W., J. Amer. Chem. Soc. 87, 3272 (1965).

Day, P., Smith, M. J. and Williams, R. J. P., J. Chem. Soc. Sect. A, 668-72 (1968).

Dubinson, G. C., Mason, R., Robertson, G. B., Ugo, R., Conti, F., Morelli, D., Cenini, S. and Bonati, F., Chem. Commun., 739-41 (1967).

Druce, P. M., Lappert, M. F. and Riley, P. N. K., Chem. Commun., 486-7 (1967).

Duddell, D. A., Goggin, P. L., Goodfellow, R. J. and Norton, M. G., Chem. Commun., 879-81 (1968).

Goggin, P. L., Goodfellow, R. J., Sales, D. L., Stokes, J. and Woodward, P., Chem. Commun., 31-2 (1968).

Goodfellow, R. J., Evans, J. G., Goggin, P. L. and Duddell, D. A., J. Chem. Soc. Sect. A, 1604-9 (1968).

Goodfellow, R. J., Goggin, P. L. and Venanzi, L. M., J. Chem. Soc. Sect. A, 1897-900 (1967)

Greenwood, N. N. and Hunter, G., J. Chem. Soc. Sect. A, 1520-3 (1967).

Grim, S. O., Keiter, R. L. and McFarlane, W., Inorg. Chem., 1133-7 (1967).

Grogan, M. J. and Nakamoto, K., J. Amer. Chem. Soc. 90, 918-22 (1968).

Harris, C. M., Livingstone, S. E. and Stephenson, N. C., J. Chem. Soc. Sect. A, 3697 (1958).

Johnson, B. F. G., Lewis, J. and Subramanian, M. S., J. Chem. Soc. Sect. A, 1993-2001 (1968).

Lindoy, L. F., Livingstone, S. E. and Lockeyer, T. N., Inorg. Chem., 652-6 (1967).

Lodewijk, E. and Wright, D., J. Chem. Soc. Sect. A, 119-22 (1968).

Paiaro, G., Renzi, A. D. and Paulumbo, R., Chem. Commun. 1150-1 (1967).

Sales, D. L., Stokes, J. and Woodward, P., J. Chem. Soc. Sect. A, 1852-8 (1968).

Smithies, A. C., Rycheck, M. and Orchin, M., J. Organometal. Chem. 12, 199-202 (1968).

Stille, J. K., Morgan, R. A., Whitehurst, D. D., and Doyle, J. R., J. Amer. Chem. Soc. 87, 3282 (1965).

Stille, J. K. and Morgan, R. A., J. Amer. Chem. Soc. 88, 5135-41 (1966).

Takahashi, H. and Tsuji, J., J. Amer. Chem. Soc. 90, 2387-92 (1968).

Truter, M. R. and Watling, R. C., J. Chem. Soc. Sect. A, 1955-63 (1967).

Watkins, S. F., Chem. Commun. 504-5 (1968).

Watt, G. W. and Cude, W. A., Inorg. Chem. 335-8 (1968).

Wrixon, A. D., Premuzic, E. and Scott, A. I., Chem. Commun. 639-41 (1968).

## BOOKS

A. Beiser and K. Krauskopf, ''Introduction to Physics and Chemistry'', McGraw-Hill Book Company, New York, New York, 1964, pp. 317-8.

J. Chatt and M. L. Searle, ''Inorganic Syntheses'', V, McGraw-Hill Book Company, New York, New York, 1957, p. 210.

G. H. Stout and L. H. Jensen, ''X-Ray Structure Determination'' Macmillan Company, New York, New York, 1968.

```
PAGE   1

// JOB T

LOG DRIVE   CART SPEC   CART AVAIL  PHY DRIVE
  0000        3333        3333        0000

V2 M07   ACTUAL 16K  CONFIG 16K

// FOR
*IOCS(CARD,1132 PRINTER)
*ONE WORD INTEGERS

UNREFERENCED STATEMENTS
 478

FEATURES SUPPORTED
 ONE WORD INTEGERS
 IOCS

CORE REQUIREMENTS FOR
 COMMON       0  VARIABLES   4190  PROGRAM   2340

 END OF COMPILATION

// XEQ

IROTA =  3 1=A AXIS,2=B AXIS,3=C AXIS,    NOLA=    3
MU ANGLE INPUT
   0.0000    8.1700   16.7500


INPUT

  H  K  L      XMEAS      WAVLN          WT    DIAM
  0  4  0     9.1500    1.54180       1.000    55.692
  0  8  0    18.6000    1.54180       1.000    55.692
  0 10  0    23.5250    1.54180       1.000    55.692
  0 14  0    34.1000    1.54180       1.000    55.692
  2  0  0     6.9700    1.54180       1.000    55.692
  4  0  0    14.1200    1.54180       1.000    55.692
  6  0  0    21.6500    1.54180       1.000    55.692
  8  0  0    29.3500    1.54180       1.000    55.692
 10  0  0    37.8500    1.54180       1.000    55.692
  1  4  0     9.9750    1.54180       1.000    55.692
  1  6  0    14.3000    1.54180       1.000    55.692
  1  6  0    14.3250    1.54180       1.000    55.692
  1  8  0    18.9500    1.54180       1.000    55.692
  1  8  0    19.0000    1.54180       1.000    55.692
  2  1  0     7.3000    1.54180       1.000    55.692
  2  1  0     7.3250    1.54180       1.000    55.692
  3  2  0    11.5250    1.54180       1.000    55.692
  3  2  0    11.4250    1.54180       1.000    55.692
  5  1  0    17.9250    1.54180       1.000    55.692
```

| | | | | | | |
|---|---|---|---|---|---|---|
| 5 | 1 | 0 | 17.8500 | 1.54180 | 1.000 | 55.692 |
| 5 | 2 | 0 | 18.8000 | 1.54180 | 1.000 | 55.692 |
| 0 | 2 | 1 | 4.6500 | 1.54180 | 1.000 | 55.692 |
| 0 | 4 | 1 | 9.3000 | 1.54180 | 1.000 | 55.692 |
| 0 | 6 | 1 | 14.0500 | 1.54180 | 1.000 | 55.692 |
| 0 | 8 | 1 | 18.9500 | 1.54180 | 1.000 | 55.692 |
| 0 | 10 | 1 | 23.9750 | 1.54180 | 1.000 | 55.692 |
| 0 | 12 | 1 | 29.2500 | 1.54180 | 1.000 | 55.692 |
| 2 | 0 | 1 | 7.1000 | 1.54180 | 1.000 | 55.692 |
| 3 | 0 | 1 | 10.6700 | 1.54180 | 1.000 | 55.692 |
| 5 | 0 | 1 | 18.1000 | 1.54180 | 1.000 | 55.692 |
| 6 | 0 | 1 | 21.9200 | 1.54180 | 1.000 | 55.692 |
| 7 | 0 | 1 | 25.8200 | 1.54180 | 1.000 | 55.692 |
| 1 | 2 | 1 | 5.8250 | 1.54180 | 1.000 | 55.692 |
| 2 | 1 | 1 | 7.5000 | 1.54180 | 1.000 | 55.692 |
| 2 | 2 | 1 | 8.3250 | 1.54180 | 1.000 | 55.692 |
| 2 | 4 | 1 | 11.7250 | 1.54180 | 1.000 | 55.692 |
| 2 | 7 | 1 | 18.0500 | 1.54180 | 1.000 | 55.692 |
| 3 | 1 | 1 | 10.9200 | 1.54180 | 1.000 | 55.692 |
| 3 | 2 | 1 | 11.8500 | 1.54180 | 1.000 | 55.692 |
| 3 | 3 | 1 | 12.8500 | 1.54180 | 1.000 | 55.692 |
| 3 | 4 | 1 | 15.0200 | 1.54180 | 1.000 | 55.692 |
| 3 | 5 | 1 | 15.9250 | 1.54180 | 1.000 | 55.692 |
| 4 | 1 | 1 | 14.3700 | 1.54180 | 1.000 | 55.692 |
| 5 | 1 | 1 | 18.2200 | 1.54180 | 1.000 | 55.692 |
| 6 | 1 | 1 | 22.0000 | 1.54180 | 1.000 | 55.692 |
| 0 | 2 | 2 | 4.8250 | 1.54180 | 1.000 | 55.692 |
| 0 | 4 | 2 | 9.7250 | 1.54180 | 1.000 | 55.692 |
| 0 | 5 | 2 | 13.7500 | 1.54180 | 1.000 | 55.692 |
| 2 | 0 | 2 | 7.4250 | 1.54180 | 1.000 | 55.692 |
| 1 | 2 | 2 | 6.1000 | 1.54180 | 1.000 | 55.692 |
| 1 | 2 | 2 | 6.1000 | 1.54180 | 1.000 | 55.692 |
| 1 | 5 | 2 | 13.9750 | 1.54180 | 1.000 | 55.692 |
| 2 | 1 | 2 | 7.7250 | 1.54180 | 1.000 | 55.692 |
| 2 | 1 | 2 | 7.8000 | 1.54180 | 1.000 | 55.692 |
| 2 | 2 | 2 | 8.8500 | 1.54180 | 1.000 | 55.692 |
| 2 | 2 | 2 | 8.8000 | 1.54180 | 1.000 | 55.692 |
| 2 | 2 | 2 | 8.8250 | 1.54180 | 1.000 | 55.692 |
| 2 | 2 | 2 | 8.7750 | 1.54180 | 1.000 | 55.692 |
| 2 | 3 | 2 | 10.2500 | 1.54180 | 1.000 | 55.692 |
| 2 | 4 | 2 | 12.2250 | 1.54180 | 1.000 | 55.692 |
| 2 | 4 | 2 | 12.2750 | 1.54180 | 1.000 | 55.692 |
| 2 | 5 | 2 | 15.4750 | 1.54180 | 1.000 | 55.692 |
| 3 | 1 | 2 | 11.4000 | 1.54180 | 1.000 | 55.692 |
| 3 | 1 | 2 | 11.6000 | 1.54180 | 1.000 | 55.692 |
| 3 | 2 | 2 | 12.6250 | 1.54180 | 1.000 | 55.692 |
| 3 | 3 | 2 | 13.3000 | 1.54180 | 1.000 | 55.692 |
| 3 | 3 | 2 | 13.4250 | 1.54180 | 1.000 | 55.692 |
| 3 | 4 | 2 | 15.2000 | 1.54180 | 1.000 | 55.692 |

| H | K | L | WAVLN | TH OBS | TH CALC | X OBS | X CALC | DELTA X | DELT(SNTH2)/WT |
|---|---|---|---|---|---|---|---|---|---|
| 0 | 4 | 0 | 1.54180 | 9.413 | 9.447 | 9.15 | 9.18 | -0.03 | 0.0006 |
| 0 | 8 | 0 | 1.54180 | 19.135 | 19.165 | 18.60 | 18.62 | -0.02 | 0.0005 |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| 0 | 10 | 0 | 1.54180 | 24.202 | 24.228 | 23.52 | 23.55 | -0.02 | 0.0004 |
| 0 | 14 | 0 | 1.54180 | 35.082 | 35.066 | 34.10 | 34.08 | 0.01 | 0.0002 |
| 2 | 0 | 0 | 1.54180 | 7.170 | 7.226 | 6.97 | 7.02 | -0.05 | 0.0009 |
| 4 | 0 | 0 | 1.54180 | 14.526 | 14.572 | 14.12 | 14.16 | -0.04 | 0.0007 |
| 6 | 0 | 0 | 1.54180 | 22.273 | 22.172 | 21.65 | 21.55 | 0.09 | 0.0017 |
| 8 | 0 | 0 | 1.54180 | 30.195 | 30.211 | 29.35 | 29.36 | -0.01 | 0.0002 |
| 10 | 0 | 0 | 1.54180 | 38.940 | 38.976 | 37.85 | 37.88 | -0.03 | 0.0006 |
| 1 | 4 | 0 | 1.54180 | 10.262 | 10.124 | 9.97 | 9.84 | 0.13 | 0.0023 |
| 1 | 6 | 0 | 1.54180 | 14.711 | 14.722 | 14.30 | 14.31 | -0.01 | 0.0001 |
| 1 | 6 | 0 | 1.54180 | 14.737 | 14.722 | 14.32 | 14.31 | 0.01 | 0.0002 |
| 1 | 8 | 0 | 1.54180 | 19.495 | 19.528 | 18.95 | 18.98 | -0.03 | 0.0005 |
| 1 | 8 | 0 | 1.54180 | 19.547 | 19.528 | 19.00 | 18.98 | 0.01 | 0.0003 |
| 2 | 1 | 0 | 1.54180 | 7.510 | 7.603 | 7.30 | 7.39 | -0.09 | 0.0016 |
| 2 | 1 | 0 | 1.54180 | 7.536 | 7.603 | 7.32 | 7.39 | -0.06 | 0.0011 |
| 3 | 2 | 0 | 1.54180 | 11.856 | 11.874 | 11.52 | 11.54 | -0.01 | 0.0003 |
| 3 | 2 | 0 | 1.54180 | 11.754 | 11.874 | 11.42 | 11.54 | -0.11 | 0.0021 |
| 5 | 1 | 0 | 1.54180 | 18.441 | 18.491 | 17.92 | 17.97 | -0.04 | 0.0008 |
| 5 | 1 | 0 | 1.54180 | 18.364 | 18.491 | 17.85 | 17.97 | -0.12 | 0.0022 |
| 5 | 2 | 0 | 1.54180 | 19.341 | 18.967 | 18.80 | 18.43 | 0.36 | 0.0064 |
| 0 | 2 | 1 | 1.54180 | 9.459 | 9.400 | 4.65 | 4.53 | 0.11 | 0.0010 |
| 0 | 4 | 1 | 1.54180 | 12.556 | 12.553 | 9.30 | 9.29 | 0.00 | 0.0000 |
| 0 | 6 | 1 | 1.54180 | 16.560 | 16.589 | 14.05 | 14.08 | -0.03 | 0.0005 |
| 0 | 8 | 1 | 1.54180 | 21.076 | 21.092 | 18.95 | 18.96 | -0.01 | 0.0002 |
| 0 | 10 | 1 | 1.54180 | 25.902 | 25.929 | 23.97 | 24.00 | -0.02 | 0.0004 |
| 0 | 12 | 1 | 1.54180 | 31.081 | 31.087 | 29.25 | 29.25 | -0.00 | 0.0001 |
| 2 | 0 | 1 | 1.54180 | 10.942 | 10.953 | 7.10 | 7.11 | -0.01 | 0.0001 |
| 3 | 0 | 1 | 1.54180 | 13.653 | 13.708 | 10.67 | 10.73 | -0.06 | 0.0009 |
| 5 | 0 | 1 | 1.54180 | 20.276 | 20.275 | 18.10 | 18.09 | 0.00 | 0.0000 |
| 6 | 0 | 1 | 1.54180 | 23.912 | 23.886 | 21.92 | 21.89 | 0.02 | 0.0004 |
| 7 | 0 | 1 | 1.54180 | 27.704 | 27.679 | 25.82 | 25.79 | 0.02 | 0.0004 |
| 1 | 2 | 1 | 1.54180 | 10.120 | 10.115 | 5.82 | 5.81 | 0.00 | 0.0000 |
| 2 | 1 | 1 | 1.54180 | 11.219 | 11.213 | 7.50 | 7.49 | 0.00 | 0.0001 |
| 2 | 2 | 1 | 1.54180 | 11.815 | 11.962 | 8.32 | 8.52 | -0.19 | 0.0025 |
| 2 | 4 | 1 | 1.54180 | 14.535 | 14.598 | 11.72 | 11.79 | -0.07 | 0.0011 |
| 2 | 7 | 1 | 1.54180 | 20.229 | 20.275 | 18.05 | 18.09 | -0.04 | 0.0007 |
| 3 | 1 | 1 | 1.54180 | 13.860 | 13.919 | 10.92 | 10.99 | -0.07 | 0.0010 |
| 3 | 2 | 1 | 1.54180 | 14.641 | 14.538 | 11.85 | 11.72 | 0.12 | 0.0017 |
| 3 | 3 | 1 | 1.54180 | 15.502 | 15.519 | 12.85 | 12.86 | -0.01 | 0.0002 |
| 3 | 4 | 1 | 1.54180 | 17.432 | 16.804 | 15.02 | 14.32 | 0.69 | 0.0107 |
| 3 | 5 | 1 | 1.54180 | 18.257 | 18.339 | 15.92 | 16.01 | -0.08 | 0.0014 |
| 4 | 1 | 1 | 1.54180 | 16.847 | 17.039 | 14.37 | 14.58 | -0.21 | 0.0033 |
| 5 | 1 | 1 | 1.54180 | 20.389 | 20.426 | 18.22 | 18.25 | -0.03 | 0.0006 |
| 6 | 1 | 1 | 1.54180 | 23.989 | 24.018 | 22.00 | 22.02 | -0.02 | 0.0004 |
| 0 | 2 | 2 | 1.54180 | 17.449 | 17.499 | 4.82 | 4.99 | -0.17 | 0.0008 |
| 0 | 4 | 2 | 1.54180 | 19.436 | 19.811 | 9.72 | 10.43 | -0.70 | 0.0065 |
| 0 | 5 | 2 | 1.54180 | 21.792 | 21.405 | 13.75 | 13.14 | 0.60 | 0.0067 |
| 2 | 0 | 2 | 1.54180 | 18.364 | 18.219 | 7.42 | 7.06 | 0.35 | 0.0025 |
| 1 | 2 | 2 | 1.54180 | 17.855 | 17.843 | 6.10 | 6.06 | 0.03 | 0.0002 |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| 1 | 2 | 2 | 1.54180 | 17.855 | 17.843 | 6.10 | 6.06 | 0.03 | 0.0002 |
| 1 | 5 | 2 | 1.54180 | 21.939 | 21.696 | 13.97 | 13.60 | 0.37 | 0.0042 |
| 2 | 1 | 2 | 1.54180 | 18.490 | 18.414 | 7.72 | 7.54 | 0.17 | 0.0013 |
| 2 | 1 | 2 | 1.54180 | 18.523 | 18.414 | 7.80 | 7.54 | 0.25 | 0.0018 |
| 2 | 2 | 2 | 1.54180 | 19.002 | 18.991 | 8.85 | 8.82 | 0.02 | 0.0001 |
| 2 | 2 | 2 | 1.54180 | 18.978 | 18.991 | 8.80 | 8.82 | -0.02 | 0.0002 |
| 2 | 2 | 2 | 1.54180 | 18.990 | 18.991 | 8.82 | 8.82 | -0.00 | 0.0000 |
| 2 | 2 | 2 | 1.54180 | 18.966 | 18.991 | 8.77 | 8.82 | -0.05 | 0.0004 |
| 2 | 3 | 2 | 1.54180 | 19.712 | 19.920 | 10.25 | 10.63 | -0.38 | 0.0036 |
| 2 | 4 | 2 | 1.54180 | 20.837 | 21.163 | 12.22 | 12.75 | -0.53 | 0.0057 |
| 2 | 4 | 2 | 1.54180 | 20.867 | 21.163 | 12.27 | 12.75 | -0.48 | 0.0051 |
| 2 | 5 | 2 | 1.54180 | 22.950 | 22.680 | 15.47 | 15.08 | 0.39 | 0.0046 |
| 3 | 1 | 2 | 1.54180 | 20.351 | 20.298 | 11.40 | 11.30 | 0.09 | 0.0009 |
| 3 | 1 | 2 | 1.54180 | 20.466 | 20.298 | 11.60 | 11.30 | 0.29 | 0.0029 |
| 3 | 2 | 2 | 1.54180 | 21.081 | 20.830 | 12.62 | 12.21 | 0.41 | 0.0043 |
| 3 | 3 | 2 | 1.54180 | 21.503 | 21.693 | 13.30 | 13.59 | -0.29 | 0.0033 |
| 3 | 3 | 2 | 1.54180 | 21.583 | 21.693 | 13.42 | 13.59 | -0.17 | 0.0019 |
| 3 | 4 | 2 | 1.54180 | 22.760 | 22.858 | 15.20 | 15.34 | -0.14 | 0.0017 |
| 0 | 4 | 0 | 1.54180 | 9.413 | 9.477 | 9.15 | 9.21 | -0.06 | 0.0011 |
| 0 | 8 | 0 | 1.54180 | 19.135 | 19.227 | 18.60 | 18.68 | -0.08 | 0.0016 |
| 0 | 10 | 0 | 1.54180 | 24.202 | 24.308 | 23.52 | 23.62 | -0.10 | 0.0018 |
| 0 | 14 | 0 | 1.54180 | 35.082 | 35.191 | 34.10 | 34.20 | -0.10 | 0.0019 |
| 2 | 0 | 0 | 1.54180 | 7.170 | 7.233 | 6.97 | 7.03 | -0.06 | 0.0011 |
| 4 | 0 | 0 | 1.54180 | 14.526 | 14.585 | 14.12 | 14.17 | -0.05 | 0.0010 |
| 6 | 0 | 0 | 1.54180 | 22.273 | 22.194 | 21.65 | 21.57 | 0.07 | 0.0013 |
| 8 | 0 | 0 | 1.54180 | 30.195 | 30.242 | 29.35 | 29.39 | -0.04 | 0.0008 |
| 10 | 0 | 0 | 1.54180 | 38.940 | 39.019 | 37.85 | 37.92 | -0.07 | 0.0013 |
| 1 | 4 | 0 | 1.54180 | 10.262 | 10.153 | 9.97 | 9.86 | 0.10 | 0.0018 |
| 1 | 6 | 0 | 1.54180 | 14.711 | 14.767 | 14.30 | 14.35 | -0.05 | 0.0009 |
| 1 | 6 | 0 | 1.54180 | 14.737 | 14.767 | 14.32 | 14.35 | -0.02 | 0.0005 |
| 1 | 8 | 0 | 1.54180 | 19.495 | 19.589 | 18.95 | 19.04 | -0.09 | 0.0016 |
| 1 | 8 | 0 | 1.54180 | 19.547 | 19.589 | 19.00 | 19.04 | -0.04 | 0.0007 |
| 2 | 1 | 0 | 1.54180 | 7.510 | 7.612 | 7.30 | 7.39 | -0.09 | 0.0017 |
| 2 | 1 | 0 | 1.54180 | 7.536 | 7.612 | 7.32 | 7.39 | -0.07 | 0.0013 |
| 3 | 2 | 0 | 1.54180 | 11.856 | 11.890 | 11.52 | 11.55 | -0.03 | 0.0005 |
| 3 | 2 | 0 | 1.54180 | 11.754 | 11.890 | 11.42 | 11.55 | -0.13 | 0.0023 |
| 5 | 1 | 0 | 1.54180 | 18.441 | 18.510 | 17.92 | 17.99 | -0.06 | 0.0012 |
| 5 | 1 | 0 | 1.54180 | 18.364 | 18.510 | 17.85 | 17.99 | -0.14 | 0.0025 |
| 5 | 2 | 0 | 1.54180 | 19.341 | 18.988 | 18.80 | 18.45 | 0.34 | 0.0061 |
| 0 | 2 | 1 | 1.54180 | 9.459 | 9.650 | 4.65 | 5.00 | -0.35 | 0.0033 |
| 0 | 4 | 1 | 1.54180 | 12.556 | 12.713 | 9.30 | 9.50 | -0.20 | 0.0027 |
| 0 | 6 | 1 | 1.54180 | 16.560 | 16.674 | 14.05 | 14.17 | -0.12 | 0.0019 |
| 0 | 8 | 1 | 1.54180 | 21.076 | 21.115 | 18.95 | 18.99 | -0.04 | 0.0006 |
| 0 | 10 | 1 | 1.54180 | 25.902 | 25.898 | 23.97 | 23.97 | 0.00 | 0.0000 |
| 0 | 12 | 1 | 1.54180 | 31.081 | 31.005 | 29.25 | 29.17 | 0.07 | 0.0013 |
| 2 | 0 | 1 | 1.54180 | 10.942 | 11.065 | 7.10 | 7.27 | -0.17 | 0.0021 |
| 3 | 0 | 1 | 1.54180 | 13.653 | 13.748 | 10.67 | 10.78 | -0.11 | 0.0016 |
| 5 | 0 | 1 | 1.54180 | 20.276 | 20.229 | 18.10 | 18.05 | 0.04 | 0.0008 |
| 6 | 0 | 1 | 1.54180 | 23.912 | 23.811 | 21.92 | 21.81 | 0.10 | 0.0017 |
| 7 | 0 | 1 | 1.54180 | 27.704 | 27.579 | 25.82 | 25.69 | 0.12 | 0.0021 |
| 1 | 2 | 1 | 1.54180 | 10.120 | 10.287 | 5.82 | 6.09 | -0.27 | 0.0029 |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| 2 | 1 | 1 | 1.54180 | 11.219 | 11.320 | 7.50 | 7.64 | -0.14 | 0.0017 |
| 2 | 2 | 1 | 1.54180 | 11.815 | 12.054 | 8.32 | 8.64 | -0.32 | 0.0042 |
| 2 | 4 | 1 | 1.54180 | 14.535 | 14.648 | 11.72 | 11.85 | -0.13 | 0.0019 |
| 2 | 7 | 1 | 1.54180 | 20.229 | 20.256 | 18.05 | 18.07 | -0.02 | 0.0004 |
| 3 | 1 | 1 | 1.54180 | 13.860 | 13.957 | 10.92 | 11.03 | -0.11 | 0.0017 |
| 3 | 2 | 1 | 1.54180 | 14.641 | 14.567 | 11.85 | 11.76 | 0.08 | 0.0012 |
| 3 | 3 | 1 | 1.54180 | 15.502 | 15.535 | 12.85 | 12.88 | -0.03 | 0.0005 |
| 3 | 4 | 1 | 1.54180 | 17.432 | 16.806 | 15.02 | 14.32 | 0.69 | 0.0107 |
| 3 | 5 | 1 | 1.54180 | 18.257 | 18.324 | 15.92 | 15.99 | -0.07 | 0.0011 |
| 4 | 1 | 1 | 1.54180 | 16.847 | 17.028 | 14.37 | 14.57 | -0.20 | 0.0031 |
| 5 | 1 | 1 | 1.54180 | 20.389 | 20.378 | 18.22 | 18.20 | 0.01 | 0.0001 |
| 6 | 1 | 1 | 1.54180 | 23.989 | 23.942 | 22.00 | 21.95 | 0.04 | 0.0008 |
| 0 | 2 | 2 | 1.54180 | 17.449 | 17.663 | 4.82 | 5.53 | -0.70 | 0.0037 |
| 0 | 4 | 2 | 1.54180 | 19.436 | 19.589 | 9.72 | 10.01 | -0.29 | 0.0026 |
| 0 | 5 | 2 | 1.54180 | 21.792 | 20.934 | 13.75 | 12.38 | 1.36 | 0.0147 |
| 2 | 0 | 2 | 1.54180 | 18.364 | 18.479 | 7.42 | 7.69 | -0.27 | 0.0020 |
| 1 | 2 | 2 | 1.54180 | 17.855 | 18.019 | 6.10 | 6.55 | -0.45 | 0.0028 |
| 1 | 2 | 2 | 1.54180 | 17.855 | 18.019 | 6.10 | 6.55 | -0.45 | 0.0028 |
| 1 | 5 | 2 | 1.54180 | 21.939 | 21.243 | 13.97 | 12.88 | 1.08 | 0.0119 |
| 2 | 1 | 2 | 1.54180 | 18.490 | 18.640 | 7.72 | 8.06 | -0.34 | 0.0026 |
| 2 | 1 | 2 | 1.54180 | 18.523 | 18.640 | 7.80 | 8.06 | -0.26 | 0.0020 |
| 2 | 2 | 2 | 1.54180 | 19.002 | 19.116 | 8.85 | 9.08 | -0.23 | 0.0019 |
| 2 | 2 | 2 | 1.54180 | 18.978 | 19.116 | 8.80 | 9.08 | -0.28 | 0.0024 |
| 2 | 2 | 2 | 1.54180 | 18.990 | 19.116 | 8.82 | 9.08 | -0.26 | 0.0022 |
| 2 | 2 | 2 | 1.54180 | 18.966 | 19.116 | 8.77 | 9.08 | -0.31 | 0.0026 |
| 2 | 3 | 2 | 1.54180 | 19.712 | 19.887 | 10.25 | 10.57 | -0.32 | 0.0030 |
| 2 | 4 | 2 | 1.54180 | 20.837 | 20.927 | 12.22 | 12.37 | -0.14 | 0.0015 |
| 2 | 4 | 2 | 1.54180 | 20.867 | 20.927 | 12.27 | 12.37 | -0.09 | 0.0010 |
| 2 | 5 | 2 | 1.54180 | 22.950 | 22.205 | 15.47 | 14.37 | 1.09 | 0.0128 |
| 3 | 1 | 2 | 1.54180 | 20.351 | 20.412 | 11.40 | 11.50 | -0.10 | 0.0010 |
| 3 | 1 | 2 | 1.54180 | 20.466 | 20.412 | 11.60 | 11.50 | 0.09 | 0.0009 |
| 3 | 2 | 2 | 1.54180 | 21.081 | 20.853 | 12.62 | 12.25 | 0.37 | 0.0039 |
| 3 | 3 | 2 | 1.54180 | 21.503 | 21.573 | 13.30 | 13.40 | -0.10 | 0.0012 |
| 3 | 3 | 2 | 1.54180 | 21.583 | 21.573 | 13.42 | 13.40 | 0.01 | 0.0001 |
| 3 | 4 | 2 | 1.54180 | 22.760 | 22.549 | 15.20 | 14.89 | 0.30 | 0.0036 |

LAYER 0, 21 REFL A= 12.256( 0.006) B= 18.785( 0.011) C= 1.000( 0.001) BETA= 90.00( 0.06)
V= 230.A*B*C* 0.0815 0.0532 0.0000

LAYER 1, 24 REFL A= 12.235( 0.037) B= 18.621( 0.022) C= 5.475( 0.028) BETA= 90.64( 0.46)
V= 1247.A*B*C* 0.0817 0.0537 0.1826

LAYER 2, 23 REFL A= 11.797( 0.274) B= 17.079( 0.108) C= 5.376( 0.019) BETA= 88.77( 0.99)
V= 1083.A*B*C* 0.0847 0.0585 0.1860

TOTALS , 68 REFL A= 12.245( 0.013) B= 18.726( 0.019) C= 5.279( 0.010) BETA= 89.46( 0.21)
V= 1210.A*B*C* 0.0816 0.0533 0.1894

```
// JOB T

LOG DRIVE   CART SPEC   CART AVAIL  PHY DRIVE
  0000        3333        3333        0000

V2 M07   ACTUAL 16K  CONFIG 16K

// FOR
*IOCS(CARD,1132 PRINTER)
*ONE WORD INTEGERS

UNREFERENCED STATEMENTS
 478

FEATURES SUPPORTED
 ONE WORD INTEGERS
 IOCS

CORE REQUIREMENTS FOR
 COMMON       0  VARIABLES   4190  PROGRAM   2340

 END OF COMPILATION

// XEQ

IROTA =  3 1=A AXIS,2=B AXIS,3=C AXIS,     NOLA=     3
MU ANGLE INPUT
   0.0000    8.7500   17.7500


INPUT

  H  K  L      XMEAS      WAVLN          WT    DIAM
  2  0  0     6.5750    1.54180       1.000    57.865
  4  0  0    13.2750    1.54180       1.000    57.865
  6  0  0    20.1500    1.54180       1.000    57.865
  5  4  0    21.1000    1.54180       1.000    57.865
  7  2  0    24.6000    1.54180       1.000    57.865
  7  2  0    24.6000    1.54180       1.000    57.865
  8  2  0    28.1700    1.54180       1.000    57.865
  9  2  0    31.9000    1.54180       1.000    57.865
  1  2  0     7.1000    1.54180       1.000    57.865
  1  4  0    13.0000    1.54180       1.000    57.865
  2  2  0     9.1500    1.54180       1.000    57.865
  2  2  0     9.0750    1.54180       1.000    57.865
  2  4  0    14.2000    1.54180       1.000    57.865
  3  2  0    11.7500    1.54180       1.000    57.865
  3  2  0    11.7500    1.54180       1.000    57.865
  3  4  0    16.1000    1.54180       1.000    57.865
  3  6  0    21.6500    1.54180       1.000    57.865
  4  2  0    14.7250    1.54180       1.000    57.865
  4  2  0    14.7250    1.54180       1.000    57.865
```

| | | | | | | |
|---|---|---|---|---|---|---|
| 5 | 2 | 0 | 17.8250 | 1.54180 | 1.000 | 57.865 |
| 5 | 2 | 0 | 17.8500 | 1.54180 | 1.000 | 57.865 |
| 6 | 2 | 0 | 21.1750 | 1.54180 | 1.000 | 57.865 |
| 6 | 2 | 0 | 21.1500 | 1.54180 | 1.000 | 57.865 |
| 0 | 2 | 1 | 6.6500 | 1.54180 | 1.000 | 57.865 |
| 0 | 4 | 1 | 13.4250 | 1.54180 | 1.000 | 57.865 |
| 0 | 6 | 1 | 20.4000 | 1.54180 | 1.000 | 57.865 |
| 0 | 8 | 1 | 27.7750 | 1.54180 | 1.000 | 57.865 |
| 0 | 10 | 1 | 34.4500 | 1.54180 | 1.000 | 57.865 |
| 3 | 0 | 1 | 9.5500 | 1.54180 | 1.000 | 57.865 |
| 5 | 0 | 1 | 16.0000 | 1.54180 | 1.000 | 57.865 |
| 6 | 0 | 1 | 19.6000 | 1.54180 | 1.000 | 57.865 |
| 7 | 0 | 1 | 22.7500 | 1.54180 | 1.000 | 57.865 |
| 9 | 0 | 1 | 29.9000 | 1.54180 | 1.000 | 57.865 |
| 1 | 1 | 1 | 4.6000 | 1.54180 | 1.000 | 57.865 |
| 1 | 2 | 1 | 7.3750 | 1.54180 | 1.000 | 57.865 |
| 1 | 5 | 1 | 17.1500 | 1.54180 | 1.000 | 57.865 |
| 1 | 6 | 1 | 20.7500 | 1.54180 | 1.000 | 57.865 |
| 2 | 1 | 1 | 7.1500 | 1.54180 | 1.000 | 57.865 |
| 2 | 2 | 1 | 9.2000 | 1.54180 | 1.000 | 57.865 |
| 3 | 1 | 1 | 10.1250 | 1.54180 | 1.000 | 57.865 |
| 3 | 2 | 1 | 11.7000 | 1.54180 | 1.000 | 57.865 |
| 3 | 3 | 1 | 13.9250 | 1.54180 | 1.000 | 57.865 |
| 4 | 1 | 1 | 13.2750 | 1.54180 | 1.000 | 57.865 |
| 4 | 2 | 1 | 14.5500 | 1.54180 | 1.000 | 57.865 |
| 5 | 1 | 1 | 16.4000 | 1.54180 | 1.000 | 57.865 |
| 5 | 2 | 1 | 17.5500 | 1.54180 | 1.000 | 57.865 |
| 6 | 1 | 1 | 19.7000 | 1.54180 | 1.000 | 57.865 |
| 7 | 1 | 1 | 23.0250 | 1.54180 | 1.000 | 57.865 |
| 0 | 2 | 2 | 6.8750 | 1.54180 | 1.000 | 57.865 |
| 0 | 4 | 2 | 13.8750 | 1.54180 | 1.000 | 57.865 |
| 0 | 6 | 2 | 21.1750 | 1.54180 | 1.000 | 57.865 |
| 0 | 8 | 2 | 28.7500 | 1.54180 | 1.000 | 57.865 |
| 0 | 10 | 2 | 37.1000 | 1.54180 | 1.000 | 57.865 |
| 1 | 0 | 2 | 2.7250 | 1.54180 | 1.000 | 57.865 |
| 4 | 0 | 2 | 13.1500 | 1.54180 | 1.000 | 57.865 |
| 6 | 0 | 2 | 20.1250 | 1.54180 | 1.000 | 57.865 |
| 1 | 1 | 2 | 4.8500 | 1.54180 | 1.000 | 57.865 |
| 1 | 2 | 2 | 7.6250 | 1.54180 | 1.000 | 57.865 |
| 1 | 3 | 2 | 10.9750 | 1.54180 | 1.000 | 57.865 |
| 1 | 4 | 2 | 14.3750 | 1.54180 | 1.000 | 57.865 |
| 2 | 1 | 2 | 7.4000 | 1.54180 | 1.000 | 57.865 |
| 2 | 2 | 2 | 9.5500 | 1.54180 | 1.000 | 57.865 |
| 2 | 3 | 2 | 12.8000 | 1.54180 | 1.000 | 57.865 |
| 2 | 4 | 2 | 15.3500 | 1.54180 | 1.000 | 57.865 |
| 2 | 5 | 2 | 18.7500 | 1.54180 | 1.000 | 57.865 |
| 3 | 1 | 2 | 10.4250 | 1.54180 | 1.000 | 57.865 |
| 3 | 2 | 2 | 12.0500 | 1.54180 | 1.000 | 57.865 |
| 4 | 1 | 2 | 13.6500 | 1.54180 | 1.000 | 57.865 |
| 5 | 1 | 2 | 16.9500 | 1.54180 | 1.000 | 57.865 |
| 6 | 1 | 2 | 20.4500 | 1.54180 | 1.000 | 57.865 |
| 6 | 3 | 2 | 22.7250 | 1.54180 | 1.000 | 57.865 |
| 6 | 4 | 2 | 24.6750 | 1.54180 | 1.000 | 57.865 |
| 7 | 2 | 2 | 24.6500 | 1.54180 | 1.000 | 57.865 |

| H | K | L | WAVLN | TH OBS | TH CALC | X OBS | X CALC | DELTA X | DELT(SNTH2)/WT |
|---|---|---|---|---|---|---|---|---|---|
| 2 | 0 | 0 | 1.54180 | 6.510 | 6.531 | 6.57 | 6.59 | -0.02 | 0.0003 |
| 4 | 0 | 0 | 1.54180 | 13.144 | 13.150 | 13.27 | 13.28 | -0.00 | 0.0001 |
| 6 | 0 | 0 | 1.54180 | 19.951 | 19.953 | 20.15 | 20.15 | -0.00 | 0.0000 |
| 5 | 4 | 0 | 1.54180 | 20.892 | 20.897 | 21.10 | 21.10 | -0.00 | 0.0000 |
| 7 | 2 | 0 | 1.54180 | 24.358 | 24.357 | 24.60 | 24.59 | 0.00 | 0.0000 |
| 7 | 2 | 0 | 1.54180 | 24.358 | 24.357 | 24.60 | 24.59 | 0.00 | 0.0000 |
| 8 | 2 | 0 | 1.54180 | 27.892 | 27.876 | 28.17 | 28.15 | 0.01 | 0.0002 |
| 9 | 2 | 0 | 1.54180 | 31.586 | 31.539 | 31.90 | 31.85 | 0.04 | 0.0008 |
| 1 | 2 | 0 | 1.54180 | 7.030 | 6.993 | 7.10 | 7.06 | 0.03 | 0.0006 |
| 1 | 4 | 0 | 1.54180 | 12.872 | 12.867 | 13.00 | 12.99 | 0.00 | 0.0000 |
| 2 | 2 | 0 | 1.54180 | 9.060 | 9.010 | 9.15 | 9.10 | 0.04 | 0.0008 |
| 2 | 2 | 0 | 1.54180 | 8.985 | 9.010 | 9.07 | 9.10 | -0.02 | 0.0004 |
| 2 | 4 | 0 | 1.54180 | 14.060 | 14.093 | 14.20 | 14.23 | -0.03 | 0.0005 |
| 3 | 2 | 0 | 1.54180 | 11.634 | 11.639 | 11.75 | 11.75 | -0.00 | 0.0000 |
| 3 | 2 | 0 | 1.54180 | 11.634 | 11.639 | 11.75 | 11.75 | -0.00 | 0.0000 |
| 3 | 4 | 0 | 1.54180 | 15.941 | 15.945 | 16.10 | 16.10 | -0.00 | 0.0000 |
| 3 | 6 | 0 | 1.54180 | 21.437 | 21.423 | 21.65 | 21.63 | 0.01 | 0.0002 |
| 4 | 2 | 0 | 1.54180 | 14.580 | 14.577 | 14.72 | 14.72 | 0.00 | 0.0000 |
| 4 | 2 | 0 | 1.54180 | 14.580 | 14.577 | 14.72 | 14.72 | 0.00 | 0.0000 |
| 5 | 2 | 0 | 1.54180 | 17.649 | 17.702 | 17.82 | 17.87 | -0.05 | 0.0009 |
| 5 | 2 | 0 | 1.54180 | 17.674 | 17.702 | 17.85 | 17.87 | -0.02 | 0.0004 |
| 6 | 2 | 0 | 1.54180 | 20.966 | 20.967 | 21.17 | 21.17 | -0.00 | 0.0000 |
| 6 | 2 | 0 | 1.54180 | 20.942 | 20.967 | 21.15 | 21.17 | -0.02 | 0.0004 |
| 0 | 2 | 1 | 1.54180 | 10.935 | 11.017 | 6.65 | 6.78 | -0.13 | 0.0014 |
| 0 | 4 | 1 | 1.54180 | 15.870 | 15.824 | 13.42 | 13.36 | 0.05 | 0.0008 |
| 0 | 6 | 1 | 1.54180 | 21.940 | 21.759 | 20.40 | 20.19 | 0.20 | 0.0031 |
| 0 | 8 | 1 | 1.54180 | 28.756 | 28.365 | 27.77 | 27.35 | 0.41 | 0.0067 |
| 0 | 10 | 1 | 1.54180 | 35.083 | 35.621 | 34.45 | 35.01 | -0.56 | 0.0094 |
| 3 | 0 | 1 | 1.54180 | 12.856 | 12.901 | 9.55 | 9.61 | -0.06 | 0.0007 |
| 5 | 0 | 1 | 1.54180 | 18.044 | 18.121 | 16.00 | 16.09 | -0.09 | 0.0013 |
| 6 | 0 | 1 | 1.54180 | 21.219 | 21.055 | 19.60 | 19.41 | 0.18 | 0.0028 |
| 7 | 0 | 1 | 1.54180 | 24.083 | 24.144 | 22.75 | 22.81 | -0.06 | 0.0010 |
| 9 | 0 | 1 | 1.54180 | 30.759 | 30.759 | 29.90 | 29.90 | -0.00 | 0.0000 |
| 1 | 1 | 1 | 1.54180 | 9.856 | 9.939 | 4.60 | 4.77 | -0.17 | 0.0014 |
| 1 | 2 | 1 | 1.54180 | 11.378 | 11.427 | 7.37 | 7.45 | -0.07 | 0.0008 |
| 1 | 5 | 1 | 1.54180 | 19.043 | 18.950 | 17.15 | 17.04 | 0.10 | 0.0016 |
| 1 | 6 | 1 | 1.54180 | 22.257 | 21.985 | 20.75 | 20.44 | 0.30 | 0.0047 |
| 2 | 1 | 1 | 1.54180 | 11.238 | 11.306 | 7.15 | 7.26 | -0.11 | 0.0011 |
| 2 | 2 | 1 | 1.54180 | 12.605 | 12.641 | 9.20 | 9.25 | -0.05 | 0.0006 |
| 3 | 1 | 1 | 1.54180 | 13.277 | 13.310 | 10.12 | 10.16 | -0.04 | 0.0005 |
| 3 | 2 | 1 | 1.54180 | 14.481 | 14.473 | 11.70 | 11.68 | 0.01 | 0.0001 |
| 3 | 3 | 1 | 1.54180 | 16.284 | 16.242 | 13.92 | 13.87 | 0.05 | 0.0007 |
| 4 | 1 | 1 | 1.54180 | 15.747 | 15.729 | 13.27 | 13.25 | 0.02 | 0.0003 |
| 4 | 2 | 1 | 1.54180 | 16.807 | 16.739 | 14.55 | 14.46 | 0.08 | 0.0011 |
| 5 | 1 | 1 | 1.54180 | 18.390 | 18.425 | 16.40 | 16.44 | -0.04 | 0.0006 |
| 5 | 2 | 1 | 1.54180 | 19.395 | 19.310 | 17.55 | 17.45 | 0.09 | 0.0014 |
| 6 | 1 | 1 | 1.54180 | 21.308 | 21.323 | 19.70 | 19.71 | -0.01 | 0.0002 |
| 7 | 1 | 1 | 1.54180 | 24.336 | 24.386 | 23.02 | 23.07 | -0.05 | 0.0008 |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| 0 | 2 | 2 | 1.54180 | 18.971 | 18.964 | 6.87 | 6.85 | 0.02 | 0.0001 |
| 0 | 4 | 2 | 1.54180 | 22.309 | 22.338 | 13.87 | 13.92 | -0.04 | 0.0005 |
| 0 | 6 | 2 | 1.54180 | 27.210 | 27.219 | 21.17 | 21.18 | -0.01 | 0.0001 |
| 0 | 8 | 2 | 1.54180 | 33.150 | 33.203 | 28.75 | 28.81 | -0.06 | 0.0009 |
| 0 | 10 | 2 | 1.54180 | 40.247 | 40.190 | 37.10 | 37.03 | 0.06 | 0.0010 |
| 1 | 0 | 2 | 1.54180 | 17.947 | 18.021 | 2.72 | 3.20 | -0.47 | 0.0012 |
| 4 | 0 | 2 | 1.54180 | 21.888 | 21.924 | 13.15 | 13.21 | -0.06 | 0.0006 |
| 6 | 0 | 2 | 1.54180 | 26.443 | 26.373 | 20.12 | 20.02 | 0.09 | 0.0012 |
| 1 | 1 | 2 | 1.54180 | 18.367 | 18.334 | 4.85 | 4.71 | 0.13 | 0.0005 |
| 1 | 2 | 2 | 1.54180 | 19.241 | 19.247 | 7.62 | 7.64 | -0.01 | 0.0001 |
| 1 | 3 | 2 | 1.54180 | 20.720 | 20.692 | 10.97 | 10.91 | 0.05 | 0.0004 |
| 1 | 4 | 2 | 1.54180 | 22.607 | 22.586 | 14.37 | 14.34 | 0.03 | 0.0003 |
| 2 | 1 | 2 | 1.54180 | 19.157 | 19.164 | 7.40 | 7.41 | -0.01 | 0.0001 |
| 2 | 2 | 2 | 1.54180 | 20.039 | 20.044 | 9.55 | 9.56 | -0.01 | 0.0000 |
| 2 | 3 | 2 | 1.54180 | 21.691 | 21.444 | 12.80 | 12.35 | 0.44 | 0.0042 |
| 2 | 4 | 2 | 1.54180 | 23.207 | 23.290 | 15.35 | 15.48 | -0.13 | 0.0014 |
| 2 | 5 | 2 | 1.54180 | 25.467 | 25.509 | 18.75 | 18.81 | -0.06 | 0.0007 |
| 3 | 1 | 2 | 1.54180 | 20.449 | 20.472 | 10.42 | 10.47 | -0.04 | 0.0004 |
| 3 | 2 | 2 | 1.54180 | 21.279 | 21.307 | 12.05 | 12.10 | -0.05 | 0.0004 |
| 4 | 1 | 2 | 1.54180 | 22.177 | 22.190 | 13.65 | 13.67 | -0.02 | 0.0002 |
| 5 | 1 | 2 | 1.54180 | 24.240 | 24.253 | 16.95 | 16.96 | -0.01 | 0.0002 |
| 6 | 1 | 2 | 1.54180 | 26.679 | 26.605 | 20.45 | 20.34 | 0.10 | 0.0012 |
| 6 | 3 | 2 | 1.54180 | 28.370 | 28.416 | 22.72 | 22.78 | -0.06 | 0.0008 |
| 6 | 4 | 2 | 1.54180 | 29.875 | 29.942 | 24.67 | 24.76 | -0.08 | 0.0011 |
| 7 | 2 | 2 | 1.54180 | 29.855 | 29.858 | 24.65 | 24.65 | -0.00 | 0.0000 |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| 2 | 0 | 0 | 1.54180 | 6.510 | 6.465 | 6.57 | 6.52 | 0.04 | 0.0007 |
| 4 | 0 | 0 | 1.54180 | 13.144 | 13.014 | 13.27 | 13.14 | 0.13 | 0.0022 |
| 6 | 0 | 0 | 1.54180 | 19.951 | 19.742 | 20.15 | 19.93 | 0.21 | 0.0036 |
| 5 | 4 | 0 | 1.54180 | 20.892 | 21.075 | 21.10 | 21.28 | -0.18 | 0.0032 |
| 7 | 2 | 0 | 1.54180 | 24.358 | 24.183 | 24.60 | 24.42 | 0.17 | 0.0030 |
| 7 | 2 | 0 | 1.54180 | 24.358 | 24.183 | 24.60 | 24.42 | 0.17 | 0.0030 |
| 8 | 2 | 0 | 1.54180 | 27.892 | 27.650 | 28.17 | 27.92 | 0.24 | 0.0042 |
| 9 | 2 | 0 | 1.54180 | 31.586 | 31.258 | 31.90 | 31.56 | 0.33 | 0.0057 |
| 1 | 2 | 0 | 1.54180 | 7.030 | 7.194 | 7.10 | 7.26 | -0.16 | 0.0029 |
| 1 | 4 | 0 | 1.54180 | 12.872 | 13.340 | 13.00 | 13.47 | -0.47 | 0.0083 |
| 2 | 2 | 0 | 1.54180 | 9.060 | 9.133 | 9.15 | 9.22 | -0.07 | 0.0012 |
| 2 | 2 | 0 | 1.54180 | 8.985 | 9.133 | 9.07 | 9.22 | -0.14 | 0.0025 |
| 2 | 4 | 0 | 1.54180 | 14.060 | 14.506 | 14.20 | 14.65 | -0.45 | 0.0079 |
| 3 | 2 | 0 | 1.54180 | 11.634 | 11.688 | 11.75 | 11.80 | -0.05 | 0.0009 |
| 3 | 2 | 0 | 1.54180 | 11.634 | 11.688 | 11.75 | 11.80 | -0.05 | 0.0009 |
| 3 | 4 | 0 | 1.54180 | 15.941 | 16.280 | 16.10 | 16.44 | -0.34 | 0.0059 |
| 3 | 6 | 0 | 1.54180 | 21.437 | 22.070 | 21.65 | 22.28 | -0.63 | 0.0111 |
| 4 | 2 | 0 | 1.54180 | 14.580 | 14.563 | 14.72 | 14.70 | 0.01 | 0.0002 |
| 4 | 2 | 0 | 1.54180 | 14.580 | 14.563 | 14.72 | 14.70 | 0.01 | 0.0002 |
| 5 | 2 | 0 | 1.54180 | 17.649 | 17.632 | 17.82 | 17.80 | 0.01 | 0.0002 |
| 5 | 2 | 0 | 1.54180 | 17.674 | 17.632 | 17.85 | 17.80 | 0.04 | 0.0007 |
| 6 | 2 | 0 | 1.54180 | 20.966 | 20.844 | 21.17 | 21.05 | 0.12 | 0.0021 |
| 6 | 2 | 0 | 1.54180 | 20.942 | 20.844 | 21.15 | 21.05 | 0.09 | 0.0016 |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| 0 | 2 | 1 | 1.54180 | 10.935 | 11.062 | 6.65 | 6.86 | -0.21 | 0.0022 |
| 0 | 4 | 1 | 1.54180 | 15.870 | 15.825 | 13.42 | 13.36 | 0.05 | 0.0007 |
| 0 | 6 | 1 | 1.54180 | 21.940 | 21.720 | 20.40 | 20.15 | 0.24 | 0.0038 |
| 0 | 8 | 1 | 1.54180 | 28.756 | 28.287 | 27.77 | 27.27 | 0.49 | 0.0081 |
| 0 | 10 | 1 | 1.54180 | 35.083 | 35.500 | 34.45 | 34.88 | -0.43 | 0.0072 |
| 3 | 0 | 1 | 1.54180 | 12.856 | 12.807 | 9.55 | 9.48 | 0.06 | 0.0008 |
| 5 | 0 | 1 | 1.54180 | 18.044 | 18.185 | 16.00 | 16.16 | -0.16 | 0.0024 |
| 6 | 0 | 1 | 1.54180 | 21.219 | 21.222 | 19.60 | 19.60 | -0.00 | 0.0000 |
| 7 | 0 | 1 | 1.54180 | 24.083 | 24.427 | 22.75 | 23.12 | -0.37 | 0.0060 |
| 9 | 0 | 1 | 1.54180 | 30.759 | 31.305 | 29.90 | 30.47 | -0.57 | 0.0095 |
| 1 | 1 | 1 | 1.54180 | 9.856 | 9.867 | 4.60 | 4.62 | -0.02 | 0.0002 |
| 1 | 2 | 1 | 1.54180 | 11.378 | 11.354 | 7.37 | 7.33 | 0.03 | 0.0004 |
| 1 | 5 | 1 | 1.54180 | 19.043 | 18.857 | 17.15 | 16.93 | 0.21 | 0.0032 |
| 1 | 6 | 1 | 1.54180 | 22.257 | 21.881 | 20.75 | 20.33 | 0.41 | 0.0065 |
| 2 | 1 | 1 | 1.54180 | 11.238 | 11.188 | 7.15 | 7.06 | 0.08 | 0.0008 |
| 2 | 2 | 1 | 1.54180 | 12.605 | 12.526 | 9.20 | 9.08 | 0.11 | 0.0013 |
| 3 | 1 | 1 | 1.54180 | 13.277 | 13.216 | 10.12 | 10.04 | 0.08 | 0.0010 |
| 3 | 2 | 1 | 1.54180 | 14.481 | 14.377 | 11.70 | 11.56 | 0.13 | 0.0018 |
| 3 | 3 | 1 | 1.54180 | 16.284 | 16.143 | 13.92 | 13.75 | 0.17 | 0.0024 |
| 4 | 1 | 1 | 1.54180 | 15.747 | 15.700 | 13.27 | 13.21 | 0.05 | 0.0008 |
| 4 | 2 | 1 | 1.54180 | 16.807 | 16.704 | 14.55 | 14.42 | 0.12 | 0.0017 |
| 5 | 1 | 1 | 1.54180 | 18.390 | 18.485 | 16.40 | 16.51 | -0.11 | 0.0016 |
| 5 | 2 | 1 | 1.54180 | 19.395 | 19.362 | 17.55 | 17.51 | 0.03 | 0.0005 |
| 6 | 1 | 1 | 1.54180 | 21.308 | 21.487 | 19.70 | 19.89 | -0.19 | 0.0031 |
| 7 | 1 | 1 | 1.54180 | 24.336 | 24.664 | 23.02 | 23.38 | -0.35 | 0.0057 |
| 0 | 2 | 2 | 1.54180 | 18.971 | 19.343 | 6.87 | 7.89 | -1.01 | 0.0065 |
| 0 | 4 | 2 | 1.54180 | 22.309 | 22.566 | 13.87 | 14.30 | -0.43 | 0.0045 |
| 0 | 6 | 2 | 1.54180 | 27.210 | 27.264 | 21.17 | 21.24 | -0.07 | 0.0009 |
| 0 | 8 | 2 | 1.54180 | 33.150 | 33.052 | 28.75 | 28.63 | 0.11 | 0.0017 |
| 0 | 10 | 2 | 1.54180 | 40.247 | 39.822 | 37.10 | 36.61 | 0.48 | 0.0074 |
| 1 | 0 | 2 | 1.54180 | 17.947 | 18.234 | 2.72 | 4.28 | -1.56 | 0.0050 |
| 4 | 0 | 2 | 1.54180 | 21.888 | 21.814 | 13.15 | 13.01 | 0.13 | 0.0012 |
| 6 | 0 | 2 | 1.54180 | 26.443 | 26.318 | 20.12 | 19.95 | 0.17 | 0.0021 |
| 1 | 1 | 2 | 1.54180 | 18.367 | 18.533 | 4.85 | 5.47 | -0.62 | 0.0029 |
| 1 | 2 | 2 | 1.54180 | 19.241 | 19.408 | 7.62 | 8.05 | -0.43 | 0.0029 |
| 1 | 3 | 2 | 1.54180 | 20.720 | 20.797 | 10.97 | 11.12 | -0.15 | 0.0013 |
| 1 | 4 | 2 | 1.54180 | 22.607 | 22.623 | 14.37 | 14.40 | -0.02 | 0.0002 |
| 2 | 1 | 2 | 1.54180 | 19.157 | 19.194 | 7.40 | 7.49 | -0.09 | 0.0006 |
| 2 | 2 | 2 | 1.54180 | 20.039 | 20.044 | 9.55 | 9.56 | -0.01 | 0.0000 |
| 2 | 3 | 2 | 1.54180 | 21.691 | 21.399 | 12.80 | 12.27 | 0.52 | 0.0050 |
| 2 | 4 | 2 | 1.54180 | 23.207 | 23.187 | 15.35 | 15.31 | 0.03 | 0.0003 |
| 2 | 5 | 2 | 1.54180 | 25.467 | 25.341 | 18.75 | 18.56 | 0.18 | 0.0021 |
| 3 | 1 | 2 | 1.54180 | 20.449 | 20.398 | 10.42 | 10.31 | 0.10 | 0.0008 |
| 3 | 2 | 2 | 1.54180 | 21.279 | 21.208 | 12.05 | 11.91 | 0.13 | 0.0012 |
| 4 | 1 | 2 | 1.54180 | 22.177 | 22.073 | 13.65 | 13.47 | 0.17 | 0.0018 |
| 5 | 1 | 2 | 1.54180 | 24.240 | 24.142 | 16.95 | 16.80 | 0.14 | 0.0017 |
| 6 | 1 | 2 | 1.54180 | 26.679 | 26.543 | 20.45 | 20.26 | 0.18 | 0.0023 |
| 6 | 3 | 2 | 1.54180 | 28.370 | 28.298 | 22.72 | 22.62 | 0.09 | 0.0012 |
| 6 | 4 | 2 | 1.54180 | 29.875 | 29.777 | 24.67 | 24.54 | 0.12 | 0.0017 |
| 7 | 2 | 2 | 1.54180 | 29.855 | 29.856 | 24.65 | 24.65 | -0.00 | 0.0000 |

```
LAYER 0, 23 REFL A= 13.553( 0.001) B= 14.322( 0.004) C=  1.000( 0.000) BETA=  90.00( 0.01)
         V=  194.A*B*C* 0.0737 0.0698 0.0000

LAYER 1, 25 REFL A= 14.170( 0.073) B= 13.728( 0.025) C=  4.985( 0.039) BETA=  89.20( 0.78)
         V=  969.A*B*C* 0.0705 0.0728 0.2005

LAYER 2, 25 REFL A= 14.370( 0.051) B= 13.547( 0.010) C=  5.065( 0.005) BETA=  90.31( 0.24)
         V=  986.A*B*C* 0.0695 0.0738 0.1974

TOTALS , 73 REFL A= 13.726( 0.016) B= 13.780( 0.017) C=  4.957( 0.009) BETA=  85.99( 0.19)
         V=  935.A*B*C* 0.0730 0.0725 0.2022
```

UNREFERENCED STATEMENTS
478

FEATURES SUPPORTED
ONE WORD INTEGERS
IOCS

CORE REQUIREMENTS FOR
COMMON 0 VARIABLES 4190 PROGRAM 2340

END OF COMPILATION

// XEQ

IROTA = 3 1=A AXIS,2=B AXIS,3=C AXIS, NOLA= 3
MU ANGLE INPUT
0.0000 8.6600 17.9160

INPUT

| H | K | L | XMEAS | WAVLN | WT | DIAM |
|---|---|---|---|---|---|---|
| 0 | 2 | 0 | 6.2200 | 1.54180 | 1.000 | 55.181 |
| 0 | 6 | 0 | 18.9500 | 1.54180 | 1.000 | 55.181 |
| 0 | 8 | 0 | 25.6500 | 1.54180 | 1.000 | 55.181 |
| 0 | 10 | 0 | 32.8700 | 1.54180 | 1.000 | 55.181 |
| 0 | 12 | 0 | 40.7500 | 1.54180 | 1.000 | 55.181 |
| 0 | 14 | 0 | 49.7500 | 1.54180 | 1.000 | 55.181 |
| 2 | 0 | 0 | 6.5500 | 1.54180 | 1.000 | 55.181 |
| 4 | 0 | 0 | 13.1250 | 1.54180 | 1.000 | 55.181 |
| 6 | 0 | 0 | 19.9250 | 1.54180 | 1.000 | 55.181 |
| 8 | 0 | 0 | 27.0750 | 1.54180 | 1.000 | 55.181 |
| 10 | 0 | 0 | 34.7500 | 1.54180 | 1.000 | 55.181 |
| 12 | 0 | 0 | 43.4000 | 1.54180 | 1.000 | 55.181 |
| 14 | 0 | 0 | 54.0500 | 1.54180 | 1.000 | 55.181 |
| 1 | 2 | 0 | 7.0000 | 1.54180 | 1.000 | 55.181 |
| 1 | 6 | 0 | 19.1750 | 1.54180 | 1.000 | 55.181 |
| 2 | 2 | 0 | 9.0000 | 1.54180 | 1.000 | 55.181 |
| 3 | 2 | 0 | 11.6700 | 1.54180 | 1.000 | 55.181 |
| 3 | 6 | 0 | 21.5500 | 1.54180 | 1.000 | 55.181 |
| 4 | 2 | 0 | 14.5700 | 1.54180 | 1.000 | 55.181 |
| 5 | 2 | 0 | 17.6500 | 1.54180 | 1.000 | 55.181 |
| 5 | 6 | 0 | 25.5500 | 1.54180 | 1.000 | 55.181 |
| 6 | 2 | 0 | 20.9700 | 1.54180 | 1.000 | 55.181 |
| 7 | 2 | 0 | 24.3500 | 1.54180 | 1.000 | 55.181 |
| 7 | 3 | 0 | 25.7000 | 1.54180 | 1.000 | 55.181 |
| 8 | 2 | 0 | 27.8700 | 1.54180 | 1.000 | 55.181 |
| 0 | 2 | 1 | 6.6500 | 1.54180 | 1.000 | 55.181 |
| 0 | 4 | 1 | 13.3750 | 1.54180 | 1.000 | 55.181 |
| 0 | 6 | 1 | 20.3000 | 1.54180 | 1.000 | 55.181 |
| 0 | 8 | 1 | 27.1500 | 1.54180 | 1.000 | 55.181 |
| 0 | 10 | 1 | 35.6500 | 1.54180 | 1.000 | 55.181 |

| | | | | | | |
|---|---|---|---|---|---|---|
| 3 | 0 | 1 | 9.4500 | 1.54180 | 1.000 | 55.181 |
| 5 | 0 | 1 | 15.9000 | 1.54180 | 1.000 | 55.181 |
| 6 | 0 | 1 | 19.0000 | 1.54180 | 1.000 | 55.181 |
| 7 | 0 | 1 | 22.5500 | 1.54180 | 1.000 | 55.181 |
| 9 | 0 | 1 | 29.6500 | 1.54180 | 1.000 | 55.181 |
| 1 | 1 | 1 | 4.5500 | 1.54180 | 1.000 | 55.181 |
| 2 | 1 | 1 | 7.1250 | 1.54180 | 1.000 | 55.181 |
| 2 | 2 | 1 | 9.6250 | 1.54180 | 1.000 | 55.181 |
| 2 | 3 | 1 | 11.9000 | 1.54180 | 1.000 | 55.181 |
| 3 | 1 | 1 | 10.0250 | 1.54180 | 1.000 | 55.181 |
| 3 | 2 | 1 | 11.6000 | 1.54180 | 1.000 | 55.181 |
| 3 | 3 | 1 | 14.4000 | 1.54180 | 1.000 | 55.181 |
| 4 | 1 | 1 | 13.1000 | 1.54180 | 1.000 | 55.181 |
| 5 | 1 | 1 | 16.3250 | 1.54180 | 1.000 | 55.181 |
| 5 | 2 | 1 | 17.3250 | 1.54180 | 1.000 | 55.181 |
| 5 | 3 | 1 | 18.9250 | 1.54180 | 1.000 | 55.181 |
| 5 | 3 | 1 | 18.9750 | 1.54180 | 1.000 | 55.181 |
| 6 | 1 | 1 | 19.3000 | 1.54180 | 1.000 | 55.181 |
| 7 | 1 | 1 | 22.7750 | 1.54180 | 1.000 | 55.181 |
| 7 | 2 | 1 | 23.6500 | 1.54180 | 1.000 | 55.181 |
| 0 | 2 | 2 | 6.8000 | 1.54180 | 1.000 | 55.181 |
| 0 | 4 | 2 | 13.7500 | 1.54180 | 1.000 | 55.181 |
| 0 | 6 | 2 | 20.9500 | 1.54180 | 1.000 | 55.181 |
| 0 | 8 | 2 | 28.5750 | 1.54180 | 1.000 | 55.181 |
| 0 | 10 | 2 | 36.7750 | 1.54180 | 1.000 | 55.181 |
| 2 | 0 | 2 | 6.4500 | 1.54180 | 1.000 | 55.181 |
| 4 | 0 | 2 | 13.0250 | 1.54180 | 1.000 | 55.181 |
| 5 | 0 | 2 | 16.4750 | 1.54180 | 1.000 | 55.181 |
| 6 | 0 | 2 | 19.5250 | 1.54180 | 1.000 | 55.181 |
| 1 | 1 | 2 | 4.6250 | 1.54180 | 1.000 | 55.181 |
| 1 | 2 | 2 | 7.5500 | 1.54180 | 1.000 | 55.181 |
| 1 | 3 | 2 | 10.7250 | 1.54180 | 1.000 | 55.181 |
| 1 | 4 | 2 | 14.1750 | 1.54180 | 1.000 | 55.181 |
| 1 | 6 | 2 | 21.2500 | 1.54180 | 1.000 | 55.181 |
| 2 | 1 | 2 | 7.3000 | 1.54180 | 1.000 | 55.181 |
| 2 | 2 | 2 | 9.4000 | 1.54180 | 1.000 | 55.181 |
| 2 | 5 | 2 | 18.6000 | 1.54180 | 1.000 | 55.181 |
| 2 | 6 | 2 | 21.9750 | 1.54180 | 1.000 | 55.181 |
| 3 | 1 | 2 | 10.3750 | 1.54180 | 1.000 | 55.181 |
| 3 | 2 | 2 | 11.9250 | 1.54180 | 1.000 | 55.181 |
| 3 | 3 | 2 | 14.2500 | 1.54180 | 1.000 | 55.181 |
| 4 | 1 | 2 | 13.5000 | 1.54180 | 1.000 | 55.181 |
| 4 | 2 | 2 | 14.4250 | 1.54180 | 1.000 | 55.181 |
| 5 | 1 | 2 | 16.7250 | 1.54180 | 1.000 | 55.181 |
| 5 | 2 | 2 | 17.9000 | 1.54180 | 1.000 | 55.181 |

| H | K | L | WAVLN | TH OBS | TH CALC | X OBS | X CALC | DELTA X | DELT(SNTH2)/WT |
|---|---|---|---|---|---|---|---|---|---|
| 0 | 2 | 0 | 1.54180 | 6.458 | 6.436 | 6.22 | 6.19 | 0.02 | 0.0003 |
| 0 | 6 | 0 | 1.54180 | 19.676 | 19.650 | 18.95 | 18.92 | 0.02 | 0.0004 |
| 0 | 8 | 0 | 1.54180 | 26.633 | 26.640 | 25.65 | 25.65 | -0.00 | 0.0001 |
| 0 | 10 | 0 | 1.54180 | 34.129 | 34.088 | 32.87 | 32.83 | 0.03 | 0.0007 |
| 0 | 12 | 0 | 1.54180 | 42.311 | 42.266 | 40.75 | 40.70 | 0.04 | 0.0007 |
| 0 | 14 | 0 | 1.54180 | 51.656 | 51.690 | 49.75 | 49.78 | -0.03 | 0.0005 |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| 2 | 0 | 0 | 1.54180 | 6.801 | 6.788 | 6.55 | 6.53 | 0.01 | 0.0002 |
| 4 | 0 | 0 | 1.54180 | 13.628 | 13.674 | 13.12 | 13.16 | -0.04 | 0.0008 |
| 6 | 0 | 0 | 1.54180 | 20.688 | 20.768 | 19.92 | 20.00 | -0.07 | 0.0014 |
| 8 | 0 | 0 | 1.54180 | 28.112 | 28.216 | 27.07 | 27.17 | -0.09 | 0.0018 |
| 10 | 0 | 0 | 1.54180 | 36.081 | 36.228 | 34.75 | 34.89 | -0.14 | 0.0025 |
| 12 | 0 | 0 | 1.54180 | 45.063 | 45.169 | 43.40 | 43.50 | -0.10 | 0.0018 |
| 14 | 0 | 0 | 1.54180 | 56.121 | 55.832 | 54.05 | 53.77 | 0.27 | 0.0050 |
| 1 | 2 | 0 | 1.54180 | 7.268 | 7.280 | 7.00 | 7.01 | -0.01 | 0.0002 |
| 1 | 6 | 0 | 1.54180 | 19.909 | 19.964 | 19.17 | 19.22 | -0.05 | 0.0009 |
| 2 | 2 | 0 | 1.54180 | 9.345 | 9.375 | 9.00 | 9.02 | -0.02 | 0.0005 |
| 3 | 2 | 0 | 1.54180 | 12.117 | 12.108 | 11.67 | 11.66 | 0.00 | 0.0001 |
| 3 | 6 | 0 | 1.54180 | 22.375 | 22.343 | 21.55 | 21.51 | 0.03 | 0.0005 |
| 4 | 2 | 0 | 1.54180 | 15.128 | 15.166 | 14.57 | 14.60 | -0.03 | 0.0006 |
| 5 | 2 | 0 | 1.54180 | 18.326 | 18.424 | 17.65 | 17.74 | -0.09 | 0.0017 |
| 5 | 6 | 0 | 1.54180 | 26.529 | 26.594 | 25.55 | 25.61 | -0.06 | 0.0011 |
| 6 | 2 | 0 | 1.54180 | 21.773 | 21.832 | 20.97 | 21.02 | -0.05 | 0.0010 |
| 7 | 2 | 0 | 1.54180 | 25.283 | 25.379 | 24.35 | 24.44 | -0.09 | 0.0016 |
| 7 | 3 | 0 | 1.54180 | 26.684 | 26.523 | 25.70 | 25.54 | 0.15 | 0.0028 |
| 8 | 2 | 0 | 1.54180 | 28.938 | 29.071 | 27.87 | 27.99 | -0.12 | 0.0023 |
| 0 | 2 | 1 | 1.54180 | 11.059 | 11.045 | 6.65 | 6.62 | 0.02 | 0.0002 |
| 0 | 4 | 1 | 1.54180 | 16.321 | 16.301 | 13.37 | 13.35 | 0.02 | 0.0003 |
| 0 | 6 | 1 | 1.54180 | 22.712 | 22.700 | 20.30 | 20.28 | 0.01 | 0.0002 |
| 0 | 8 | 1 | 1.54180 | 29.386 | 29.808 | 27.15 | 27.57 | -0.42 | 0.0074 |
| 0 | 10 | 1 | 1.54180 | 37.874 | 37.664 | 35.65 | 35.44 | 0.20 | 0.0036 |
| 3 | 0 | 1 | 1.54180 | 13.059 | 13.141 | 9.45 | 9.55 | -0.10 | 0.0014 |
| 5 | 0 | 1 | 1.54180 | 18.586 | 18.614 | 15.90 | 15.92 | -0.02 | 0.0004 |
| 6 | 0 | 1 | 1.54180 | 21.475 | 21.660 | 19.00 | 19.19 | -0.19 | 0.0032 |
| 7 | 0 | 1 | 1.54180 | 24.879 | 24.861 | 22.55 | 22.53 | 0.01 | 0.0003 |
| 9 | 0 | 1 | 1.54180 | 31.865 | 31.712 | 29.65 | 29.49 | 0.15 | 0.0026 |
| 1 | 1 | 1 | 1.54180 | 9.856 | 9.921 | 4.55 | 4.68 | -0.13 | 0.0011 |
| 2 | 1 | 1 | 1.54180 | 11.371 | 11.442 | 7.12 | 7.23 | -0.10 | 0.0012 |
| 2 | 2 | 1 | 1.54180 | 13.195 | 12.904 | 9.62 | 9.24 | 0.37 | 0.0050 |
| 2 | 3 | 1 | 1.54180 | 15.049 | 15.048 | 11.90 | 11.89 | 0.00 | 0.0000 |
| 3 | 1 | 1 | 1.54180 | 13.510 | 13.588 | 10.02 | 10.12 | -0.09 | 0.0013 |
| 3 | 2 | 1 | 1.54180 | 14.797 | 14.853 | 11.60 | 11.66 | -0.06 | 0.0009 |
| 3 | 3 | 1 | 1.54180 | 17.229 | 16.770 | 14.40 | 13.88 | 0.51 | 0.0079 |
| 4 | 1 | 1 | 1.54180 | 16.080 | 16.132 | 13.10 | 13.15 | -0.05 | 0.0009 |
| 5 | 1 | 1 | 1.54180 | 18.976 | 18.943 | 16.32 | 16.28 | 0.03 | 0.0005 |
| 5 | 2 | 1 | 1.54180 | 19.902 | 19.904 | 17.32 | 17.32 | -0.00 | 0.0000 |
| 5 | 3 | 1 | 1.54180 | 21.404 | 21.426 | 18.92 | 18.94 | -0.02 | 0.0003 |
| 5 | 3 | 1 | 1.54180 | 21.452 | 21.426 | 18.97 | 18.94 | 0.02 | 0.0004 |
| 6 | 1 | 1 | 1.54180 | 21.760 | 21.951 | 19.30 | 19.50 | -0.20 | 0.0033 |
| 7 | 1 | 1 | 1.54180 | 25.097 | 25.123 | 22.77 | 22.80 | -0.02 | 0.0004 |
| 7 | 2 | 1 | 1.54180 | 25.949 | 25.899 | 23.65 | 23.59 | 0.05 | 0.0008 |
| 0 | 2 | 2 | 1.54180 | 19.214 | 19.202 | 6.80 | 6.76 | 0.03 | 0.0002 |
| 0 | 4 | 2 | 1.54180 | 22.761 | 22.764 | 13.75 | 13.75 | -0.00 | 0.0000 |
| 0 | 6 | 2 | 1.54180 | 27.901 | 27.902 | 20.95 | 20.95 | -0.00 | 0.0000 |
| 0 | 8 | 2 | 1.54180 | 34.233 | 34.200 | 28.57 | 28.53 | 0.03 | 0.0005 |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| 0 | 10 | 2 | 1.54180 | 41.590 | 41.589 | 36.77 | 36.77 | 0.00 | 0.0000 |
| 2 | 0 | 2 | 1.54180 | 19.088 | 19.087 | 6.45 | 6.44 | 0.00 | 0.0000 |
| 4 | 0 | 2 | 1.54180 | 22.312 | 22.288 | 13.02 | 12.98 | 0.04 | 0.0004 |
| 5 | 0 | 2 | 1.54180 | 24.575 | 24.456 | 16.47 | 16.30 | 0.17 | 0.0020 |
| 6 | 0 | 2 | 1.54180 | 26.802 | 26.923 | 19.52 | 19.68 | -0.15 | 0.0021 |
| 1 | 1 | 2 | 1.54180 | 18.528 | 18.535 | 4.62 | 4.65 | -0.03 | 0.0001 |
| 1 | 2 | 2 | 1.54180 | 19.504 | 19.501 | 7.55 | 7.54 | 0.00 | 0.0000 |
| 1 | 3 | 2 | 1.54180 | 20.998 | 21.028 | 10.72 | 10.78 | -0.05 | 0.0005 |
| 1 | 4 | 2 | 1.54180 | 23.032 | 23.026 | 14.17 | 14.16 | 0.00 | 0.0000 |
| 1 | 6 | 2 | 1.54180 | 28.137 | 28.128 | 21.25 | 21.23 | 0.01 | 0.0001 |
| 2 | 1 | 2 | 1.54180 | 19.404 | 19.406 | 7.30 | 7.30 | -0.00 | 0.0000 |
| 2 | 2 | 2 | 1.54180 | 20.325 | 20.337 | 9.40 | 9.42 | -0.02 | 0.0002 |
| 2 | 5 | 2 | 1.54180 | 26.108 | 26.097 | 18.60 | 18.58 | 0.01 | 0.0001 |
| 2 | 6 | 2 | 1.54180 | 28.711 | 28.767 | 21.97 | 22.04 | -0.07 | 0.0009 |
| 3 | 1 | 2 | 1.54180 | 20.813 | 20.775 | 10.37 | 10.30 | 0.07 | 0.0006 |
| 3 | 2 | 2 | 1.54180 | 21.662 | 21.657 | 11.92 | 11.91 | 0.00 | 0.0000 |
| 3 | 3 | 2 | 1.54180 | 23.080 | 23.067 | 14.25 | 14.23 | 0.01 | 0.0002 |
| 4 | 1 | 2 | 1.54180 | 22.605 | 22.569 | 13.50 | 13.44 | 0.05 | 0.0006 |
| 4 | 2 | 2 | 1.54180 | 23.193 | 23.397 | 14.42 | 14.73 | -0.31 | 0.0035 |
| 5 | 1 | 2 | 1.54180 | 24.750 | 24.718 | 16.72 | 16.67 | 0.04 | 0.0005 |
| 5 | 2 | 2 | 1.54180 | 25.593 | 25.493 | 17.90 | 17.76 | 0.13 | 0.0017 |
| 0 | 2 | 0 | 1.54180 | 6.458 | 6.516 | 6.22 | 6.27 | -0.05 | 0.0010 |
| 0 | 6 | 0 | 1.54180 | 19.676 | 19.904 | 18.95 | 19.16 | -0.21 | 0.0039 |
| 0 | 8 | 0 | 1.54180 | 26.633 | 26.995 | 25.65 | 25.99 | -0.34 | 0.0063 |
| 0 | 10 | 0 | 1.54180 | 34.129 | 34.569 | 32.87 | 33.29 | -0.42 | 0.0077 |
| 0 | 12 | 0 | 1.54180 | 42.311 | 42.913 | 40.75 | 41.32 | -0.57 | 0.0105 |
| 0 | 14 | 0 | 1.54180 | 51.656 | 52.596 | 49.75 | 50.65 | -0.90 | 0.0163 |
| 2 | 0 | 0 | 1.54180 | 6.801 | 6.770 | 6.55 | 6.52 | 0.02 | 0.0005 |
| 4 | 0 | 0 | 1.54180 | 13.628 | 13.638 | 13.12 | 13.13 | -0.00 | 0.0001 |
| 6 | 0 | 0 | 1.54180 | 20.688 | 20.712 | 19.92 | 19.94 | -0.02 | 0.0004 |
| 8 | 0 | 0 | 1.54180 | 28.112 | 28.136 | 27.07 | 27.09 | -0.02 | 0.0004 |
| 10 | 0 | 0 | 1.54180 | 36.081 | 36.119 | 34.75 | 34.78 | -0.03 | 0.0006 |
| 12 | 0 | 0 | 1.54180 | 45.063 | 45.021 | 43.40 | 43.35 | 0.04 | 0.0007 |
| 14 | 0 | 0 | 1.54180 | 56.121 | 55.615 | 54.05 | 53.56 | 0.48 | 0.0088 |
| 1 | 2 | 0 | 1.54180 | 7.268 | 7.347 | 7.00 | 7.07 | -0.07 | 0.0013 |
| 1 | 6 | 0 | 1.54180 | 19.909 | 20.213 | 19.17 | 19.46 | -0.29 | 0.0053 |
| 2 | 2 | 0 | 1.54180 | 9.345 | 9.418 | 9.00 | 9.07 | -0.07 | 0.0012 |
| 3 | 2 | 0 | 1.54180 | 12.117 | 12.129 | 11.67 | 11.68 | -0.01 | 0.0002 |
| 3 | 6 | 0 | 1.54180 | 22.375 | 22.559 | 21.55 | 21.72 | -0.17 | 0.0032 |
| 4 | 2 | 0 | 1.54180 | 15.128 | 15.169 | 14.57 | 14.60 | -0.03 | 0.0007 |
| 5 | 2 | 0 | 1.54180 | 18.326 | 18.410 | 17.65 | 17.73 | -0.08 | 0.0014 |
| 5 | 6 | 0 | 1.54180 | 26.529 | 26.763 | 25.55 | 25.77 | -0.22 | 0.0040 |
| 6 | 2 | 0 | 1.54180 | 21.773 | 21.804 | 20.97 | 20.99 | -0.02 | 0.0005 |
| 7 | 2 | 0 | 1.54180 | 25.283 | 25.337 | 24.35 | 24.40 | -0.05 | 0.0009 |
| 7 | 3 | 0 | 1.54180 | 26.684 | 26.510 | 25.70 | 25.53 | 0.16 | 0.0030 |
| 8 | 2 | 0 | 1.54180 | 28.938 | 29.015 | 27.87 | 27.94 | -0.07 | 0.0013 |
| 0 | 2 | 1 | 1.54180 | 11.059 | 11.361 | 6.65 | 7.11 | -0.46 | 0.0053 |
| 0 | 4 | 1 | 1.54180 | 16.321 | 16.157 | 13.37 | 13.18 | 0.18 | 0.0028 |
| 0 | 6 | 1 | 1.54180 | 22.712 | 22.123 | 20.30 | 19.68 | 0.61 | 0.0101 |
| 0 | 8 | 1 | 1.54180 | 29.386 | 28.792 | 27.15 | 26.54 | 0.60 | 0.0102 |
| 0 | 10 | 1 | 1.54180 | 37.874 | 36.144 | 35.65 | 33.92 | 1.72 | 0.0299 |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| 3 | 0 | 1 | 1.54180 | 13.059 | 13.045 | 9.45 | 9.43 | 0.01 | 0.0002 |
| 5 | 0 | 1 | 1.54180 | 18.586 | 18.661 | 15.90 | 15.98 | -0.08 | 0.0013 |
| 6 | 0 | 1 | 1.54180 | 21.475 | 21.850 | 19.00 | 19.39 | -0.39 | 0.0065 |
| 7 | 0 | 1 | 1.54180 | 24.879 | 25.223 | 22.55 | 22.90 | -0.35 | 0.0060 |
| 9 | 0 | 1 | 1.54180 | 31.865 | 32.496 | 29.65 | 30.28 | -0.63 | 0.0110 |
| 1 | 1 | 1 | 1.54180 | 9.856 | 10.064 | 4.55 | 4.95 | -0.40 | 0.0036 |
| 2 | 1 | 1 | 1.54180 | 11.371 | 11.372 | 7.12 | 7.12 | -0.00 | 0.0000 |
| 2 | 2 | 1 | 1.54180 | 13.195 | 12.727 | 9.62 | 9.01 | 0.60 | 0.0080 |
| 2 | 3 | 1 | 1.54180 | 15.049 | 14.728 | 11.90 | 11.51 | 0.38 | 0.0055 |
| 3 | 1 | 1 | 1.54180 | 13.510 | 13.458 | 10.02 | 9.96 | 0.06 | 0.0008 |
| 3 | 2 | 1 | 1.54180 | 14.797 | 14.634 | 11.60 | 11.40 | 0.19 | 0.0028 |
| 3 | 3 | 1 | 1.54180 | 17.229 | 16.422 | 14.40 | 13.49 | 0.90 | 0.0137 |
| 4 | 1 | 1 | 1.54180 | 16.080 | 16.047 | 13.10 | 13.06 | 0.03 | 0.0005 |
| 5 | 1 | 1 | 1.54180 | 18.976 | 18.963 | 16.32 | 16.31 | 0.01 | 0.0002 |
| 5 | 2 | 1 | 1.54180 | 19.902 | 19.846 | 17.32 | 17.26 | 0.06 | 0.0009 |
| 5 | 3 | 1 | 1.54180 | 21.404 | 21.249 | 18.92 | 18.76 | 0.16 | 0.0026 |
| 5 | 3 | 1 | 1.54180 | 21.452 | 21.249 | 18.97 | 18.76 | 0.21 | 0.0035 |
| 6 | 1 | 1 | 1.54180 | 21.760 | 22.116 | 19.30 | 19.67 | -0.37 | 0.0062 |
| 7 | 1 | 1 | 1.54180 | 25.097 | 25.462 | 22.77 | 23.15 | -0.37 | 0.0063 |
| 7 | 2 | 1 | 1.54180 | 25.949 | 26.168 | 23.65 | 23.87 | -0.22 | 0.0038 |
| 0 | 2 | 2 | 1.54180 | 19.214 | 19.966 | 6.80 | 8.63 | -1.83 | 0.0133 |
| 0 | 4 | 2 | 1.54180 | 22.761 | 23.203 | 13.75 | 14.44 | -0.69 | 0.0077 |
| 0 | 6 | 2 | 1.54180 | 27.901 | 27.946 | 20.95 | 21.00 | -0.05 | 0.0007 |
| 0 | 8 | 2 | 1.54180 | 34.233 | 33.819 | 28.57 | 28.09 | 0.47 | 0.0072 |
| 0 | 10 | 2 | 1.54180 | 41.590 | 40.725 | 36.77 | 35.83 | 0.94 | 0.0150 |
| 2 | 0 | 2 | 1.54180 | 19.088 | 19.304 | 6.45 | 7.03 | -0.58 | 0.0037 |
| 4 | 0 | 2 | 1.54180 | 22.312 | 22.190 | 13.02 | 12.82 | 0.20 | 0.0021 |
| 5 | 0 | 2 | 1.54180 | 24.575 | 24.331 | 16.47 | 16.12 | 0.35 | 0.0042 |
| 6 | 0 | 2 | 1.54180 | 26.802 | 26.837 | 19.52 | 19.56 | -0.04 | 0.0005 |
| 1 | 1 | 2 | 1.54180 | 18.528 | 19.023 | 4.62 | 6.26 | -1.63 | 0.0087 |
| 1 | 2 | 2 | 1.54180 | 19.504 | 19.904 | 7.55 | 8.49 | -0.94 | 0.0070 |
| 1 | 3 | 2 | 1.54180 | 20.998 | 21.304 | 10.72 | 11.29 | -0.56 | 0.0053 |
| 1 | 4 | 2 | 1.54180 | 23.032 | 23.148 | 14.17 | 14.35 | -0.18 | 0.0020 |
| 1 | 6 | 2 | 1.54180 | 28.137 | 27.898 | 21.25 | 20.94 | 0.30 | 0.0041 |
| 2 | 1 | 2 | 1.54180 | 19.404 | 19.598 | 7.30 | 7.77 | -0.47 | 0.0033 |
| 2 | 2 | 2 | 1.54180 | 20.325 | 20.458 | 9.40 | 9.67 | -0.27 | 0.0023 |
| 2 | 5 | 2 | 1.54180 | 26.108 | 25.825 | 18.60 | 18.21 | 0.38 | 0.0049 |
| 2 | 6 | 2 | 1.54180 | 28.711 | 28.329 | 21.97 | 21.49 | 0.48 | 0.0066 |
| 3 | 1 | 2 | 1.54180 | 20.813 | 20.765 | 10.37 | 10.28 | 0.09 | 0.0008 |
| 3 | 2 | 2 | 1.54180 | 21.662 | 21.587 | 11.92 | 11.79 | 0.12 | 0.0012 |
| 3 | 3 | 2 | 1.54180 | 23.080 | 22.904 | 14.25 | 13.97 | 0.27 | 0.0030 |
| 4 | 1 | 2 | 1.54180 | 22.605 | 22.453 | 13.50 | 13.25 | 0.24 | 0.0026 |
| 4 | 2 | 2 | 1.54180 | 23.193 | 23.226 | 14.42 | 14.47 | -0.05 | 0.0005 |
| 5 | 1 | 2 | 1.54180 | 24.750 | 24.576 | 16.72 | 16.47 | 0.24 | 0.0030 |
| 5 | 2 | 2 | 1.54180 | 25.593 | 25.299 | 17.90 | 17.49 | 0.40 | 0.0050 |

LAYER 0, 25 REFL A= 13.043( 0.005) B= 13.754( 0.006) C= 1.000( 0.001) BETA= 90.00( 0.08)
V= 179.A*B*C* 0.0766 0.0727 0.0000

LAYER 1, 25 REFL A= 13.883( 0.058) B= 13.017( 0.019) C= 5.120( 0.038) BETA= 91.42( 0.67)
V= 925.A*B*C* 0.0720 0.0768 0.1953

LAYER 2, 25 REFL A= 13.992( 0.060) B= 13.100( 0.008) C=  5.020( 0.005) BETA=  90.40( 0.25)
V=  920.A*B*C* 0.0714 0.0763 0.1991

TOTALS , 75 REFL A= 13.157( 0.018) B= 13.586( 0.020) C=  4.816( 0.017) BETA=  83.69( 0.33)
V=  855.A*B*C* 0.0764 0.0736 0.2088

```
// JOB T

LOG DRIVE   CART SPEC   CART AVAIL  PHY DRIVE
  0000        3333        3333        0000

V2 M07   ACTUAL 16K  CONFIG 16K

// FOR
*IOCS(CARD,1132 PRINTER)
*ONE WORD INTEGERS
*LIST SOURCE PROGRAM
C  MONOCLINIC UNIT CELLS    APRIL 1969 .
      DIMENSION  UU(16),IH(200),IK(200),IL(200),XMEAS(200),WAVLN(200),
     1WT(200),THETA(200),SNTH2(200),ILAP1(200), X(45),XX(45),Y(45),Z(45),
     2,Q(6,6),NREF(16),FLMDA(200), XOUT(12,16)
      DO 490 I = 1,12
      DO 490 J = 1,16
  490 XOUT(I,J) = 0.0
      READ(2,1)IROTA, NOLA
      WRITE(3,470) IROTA, NOLA
  470 FORMAT (8H IROTA =,I3,37H 1=A AXIS,2=B AXIS,3=C AXIS,    NOLA=,I5)
      WRITE(3,401)
  401 FORMAT (15H MU ANGLE INPUT)
      READ(2,15) (UU(N), N=1, NOLA)
      WRITE (3,15) (UU(N), N=1, NOLA)
      DO 475 N = 1,NOLA
  475 NREF(N) = 0
      ILAYM  = 1
      N = 1
      WRITE(3,402)
  402 FORMAT(/,/,/,6H INPUT,/)
      WRITE (3,403)
  403 FORMAT ( 47H   H  K  L     XMEAS    WAVLN          WT   DIAM)
   10 READ(2,1) IH(N),IK(N),IL(N),XMEAS(N),WAVLN(N),EXTWT,FLMDA(N)
      WAVLN(N) = 1.5418
      IF(XMEAS(N)) 11,11,404
  404 WRITE(3,1)IH(N),IK(N),IL(N),XMEAS(N),WAVLN(N),EXTWT,FLMDA(N)
      IF(IROTA -2) 441,442,443
  441 ILAP1(N)  = IH(N)+1
      GO TO 444
  442 ILAP1(N)  = IK(N) +1
      GO TO 444
  443 ILAP1(N)  = IL(N) + 1
  444 IF(ILAP1(N) -ILAYM)  9,9,65
   65 ILAYM = ILAP1(N)
    9 IIII = ILAP1(N)
      UM = UU(IIII)
      COSMU = COS(UM*3.14159/180.)
      XRAD = XMEAS(N)/FLMDA (N)
      COTHR  = COS(XRAD)*COSMU
      THRAD = ACOS(COTHR)
      THETA(N) = THRAD*180./3.14159
      SNTH2(N) = 1.-COTHR *COTHR
      SN2TH  = SIN(THETA(N)*3.14159/90.)
```

```
      WT(N) = EXTWT*EXTWT/(SN2TH *SN2TH)
      NREF(IIII) = NREF(IIII) + 1
      N = N+1
      GO TO 10
   11 NOREF  = N-1
      WRITE (3,52)
      IPASS = 1
      ILAYM  = ILAYM +1
      NREF(ILAYM)  = NOREF
  476 CONTINUE
      IF(NREF(IPASS)) 451,461,451
451   DO 12 I = 1,4
      DO 12 J = I,5
      N = I*10+J
   12 XX(N) = 0.0
      DO 14 N = 1,NOREF
      IF(IPASS-ILAYM) 511,501,511
511   IF(ILAP1(N) -IPASS) 14,501,14
  501 X(1)=IH(N)*IH(N)
      X(2) = IK(N)*IK(N)
      X(3) = IL(N)*IL(N)
      X(4) = IH(N)*IL(N)
      X(5) = 4.*SNTH2(N)/(WAVLN (N)**2)
      DO 13 I = 1,4
      DO 13 J = I,5
      NN = I*10+J
   13 XX(NN) = XX(NN) + X(I)*X(J)*WT(N)
   14 CONTINUE
      DO 70 I = 11,15
   70 Y(I) = XX(I)/XX(11)
      DO 71 I = 22,25
      Z(I) = XX(I)-XX(I-10)*Y(12)
   71 Y(I) = Z(I)/Z(22)
      DO 72 I = 33,35
      Z(I) = XX(I)-Y(13)* XX(I-20)-Y(23)*Z(I-10)
   72 Y(I) = Z(I)/Z(33)
      DO 73 I = 44,45
      Z(I) = XX(I)-Y(14)*XX(I-30)-Y(24)*Z(I-20)-Y(34)*Z(I-10)
   73 Y(I) = Z(I)/Z(44)
      EE = Y(45)
      CC =Y(35)-Y(34)*EE
      BB = Y(25)-Y(24)*EE-Y(23)*CC
      AA = Y(15)-Y(14)*EE-Y(13)*CC-Y(12)*BB
      ASTAR = SQRT(AA)
      BSTAR = SQRT(BB)
      CSTAR = SQRT(CC)
      COSBS=EE/(2.*ASTAR*CSTAR)
      BETST=ACOS(COSBS)*180./3.14159
      SINBS =SQRT(1.-COSBS*COSBS)
      A=1./(ASTAR*SINBS)
      B = 1./BSTAR
      C=1./(CSTAR*SINBS)
      COSB=-COSBS
      SINB = SQRT(1.-COSB*COSB)
```

```
      BETA = ACOS(COSB)*180./3.14159
      VOL = A*B*C*SINB
      SUMSI  = 0.0
      DO 50 N = 1,NOREF
      IF(IPASS-ILAYM)  522,502,522
  522 IF(ILAP1(N) -IPASS) 50,502,50
  502 AHH = IH(N)
      AKK = IK(N)
      ALL = IL(N)
      SSCAL  = AHH*AHH*AA+AKK*AKK*BB+ALL*ALL*CC+AHH*ALL*EE
      SSCAL  = SSCAL *WAVLN(N) *WAVLN(N)/4.
      THCAL = ASIN(SQRT(SSCAL))
      IF(IROTA -2) 457,458,459
  457 IIII=IH(N)+1
      GO TO 456
  458 IIII = IK(N)+1
      GO TO 456
  459 IIII = IL(N)+1
  456 UM = UU(IIII)
      COSMU = COS(UM*3.14159/180.)
      XCALC = FLMDA (N)*ACOS(COS(THCAL)/COSMU)
      DELTX  = XMEAS(N) - XCALC
      CRUD = THETA(N)*3.14159/180.
      CRUDY  = SIN(CRUD)
      SNTHQ  = CRUDY *CRUDY
      SUMSI  = SUMSI  + (SNTHQ -SSCAL )**2
      RESID  =(ABS(SNTHQ -SSCAL ))*SQRT(WT(N))
      THCAL  = THCAL *180./3.14159
      WRITE (3,51) IH(N),IK(N),IL(N),WAVLN(N),THETA(N),THCAL,XMEAS(N),
     1 XCALC,DELTX ,RESID
   50 CONTINUE
      DO 60 J = 1,4
      DO 60 I = J,4
      NN = 10*J + I
      Q(I,J) = XX(NN)
   60 Q(J,I) = XX(NN)
      DO 61 I = 1,4
      QQ = Q(I,I)
      Q(I,I) = 1.0
      DO 62 K = 1,4
   62 Q(I,K) = Q(I,K)/QQ
      DO 61 J = 1,4
      IF(I-J) 59,61,59
   59 QQ = Q(J,I)
      Q(J,I) = 0.0
      DO 63 K = 1,4
   63 Q(J,K) = Q(J,K)-QQ*Q(I,K)
   61 CONTINUE
      XNREF  = NREF(IPASS)
      IF(XNREF -4.)461,471,471
  471 CONST = SUMSI /(XNREF -3.)
      SIGAA = SQRT(CONST*Q(1,1))
      SIGBB = SQRT(CONST*Q(2,2))
      SIGCC = SQRT(CONST*Q(3,3))
```

```
      SIGEE = SQRT(CONST*Q(4,4))
      SIGA = SIGAA*A/(2.*ASTAR*ASTAR)
      SIGB = SIGBB*B/(2.*BSTAR*BSTAR)
      SIGC = SIGCC*C/(2.*CSTAR*CSTAR)
      SGBET =SIGEE/(2.*ASTAR*CSTAR*SINB)
      SGBET  = SGBET *57.2958
      IF(BETA -0.)21,22,21
22    A = 0.0
21    XOUT(1,IPASS) = A
      XOUT(2,IPASS) = SIGA
      XOUT(3,IPASS) = B
      XOUT(4,IPASS) = SIGB
      XOUT(5,IPASS) = C
      XOUT(6,IPASS) = SIGC
      XOUT(7,IPASS) = BETA
      XOUT(8,IPASS) = SGBET
      XOUT(9,IPASS) = VOL
      XOUT(10,IPASS) = ASTAR
      XOUT(11,IPASS) = BSTAR
      XOUT(12,IPASS) = CSTAR
      WRITE (3,491)
  491 FORMAT (/,/)
      IF(IPASS-ILAYM ) 484,480,484
484   IPASS = IPASS +1
      GO TO 476
  461 IPAS = IPASS-1
      WRITE (3,462) IPAS
  462 FORMAT (25H LESS THAN 5 REFLECTIONS ,3H ON,I5,25H CANT CALCULATE
     1SEPARATE)
      IPASS = IPASS +1
      GO TO 476
  480 IIEND = ILAYM  -1
      DO 488 IIII = 1,IIEND
      IF(NREF(IIII)-5) 488,498,498
498   IIIM1  = IIII-1
  478 WRITE (3,53) IIIM1,NREF(IIII),(XOUT(N,IIII),N=1,12)
  488 CONTINUE
      WRITE (3,54) NREF(ILAYM),(XOUT(N,ILAYM), N=1,12)
   53 FORMAT (/,6H LAYER,I2,1H,,I3,5H REFL,
     1                       3H A=,F7.3,1H(,F6.3,1H),3H B=,F7.3,1H(,F6.3,1H),
     21H),3H C=,F7.3,1H(,F6.3,1H),6H BETA=,F7.2,1H(,F5.2,1H),/9X,
     33H V=,F6.0,6HA*B*C*,3F7.4)
   54 FORMAT (/,8H TOTALS , 1H,,I3,5H REFL,
     1                       3H A=,F7.3,1H(,F6.3,1H),3H B=,F7.3,1H(,F6.3,1H),
     21H),3H C=,F7.3,1H(,F6.3,1H),6H BETA=,F7.2,1H(,F5.2,1H),/9X,
     33H V=,F6.0,6HA*B*C*,3F7.4)
    1 FORMAT (I4,2I3,F10.4,F10.5,2F10.3)
   15 FORMAT (8F10.4)
   52 FORMAT(/,84H   H  K  L  WAVLN      TH OBS  TH CALC      X OBS   X C
     1ALC     DELTA X DELT(SNTH2)/WT,/)
   51 FORMAT (I4,2I3,F11.5,2F8.3,4X,2F8.2,F11.2,F16.4)
      CALL EXIT
      END
```