AD-A080 167 TEXAS A AND M UNIV COLLEGE STATION DEPT OF CHEMISTRY F/6 7/3
SYNTHESIS AND STRUCTURE OF A NEW CLASS OF METALLOPHTHALOCYANATO--ETC(U)
JAN 80 M TSUTSUI, S OMIYA N00014-75-C-0417
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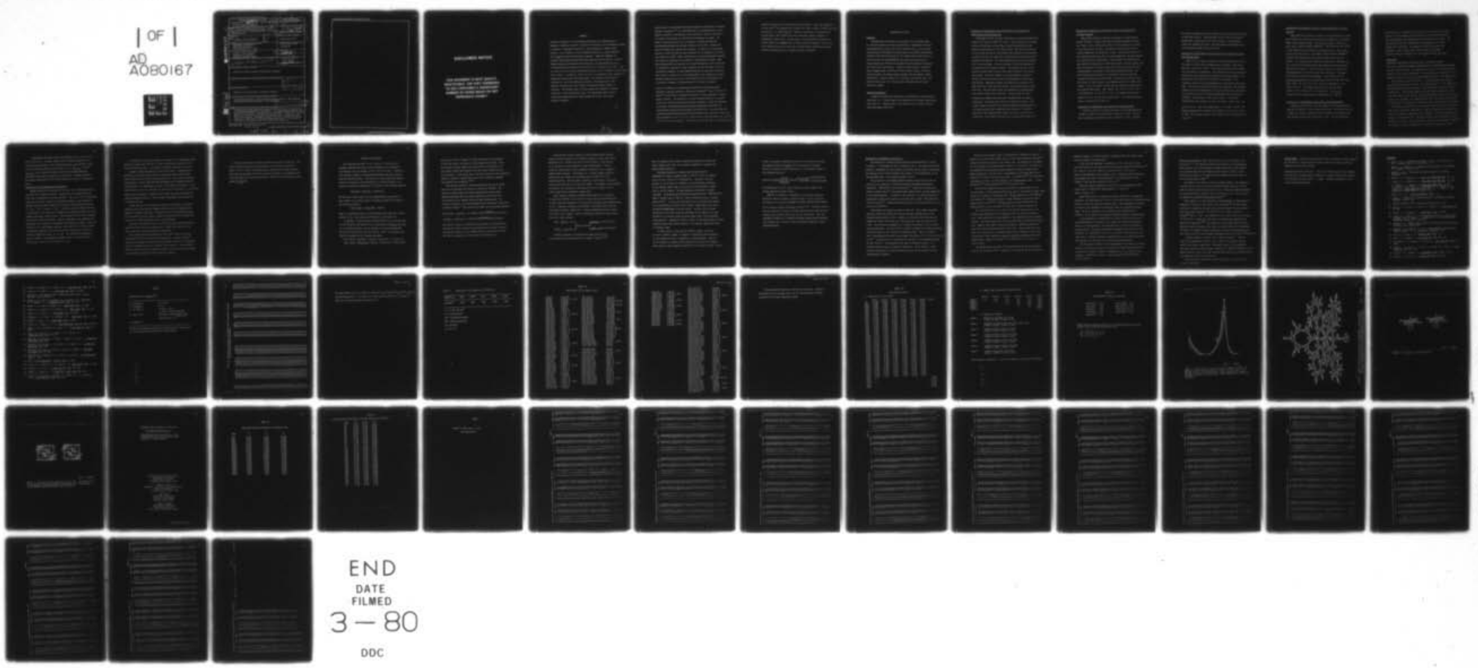

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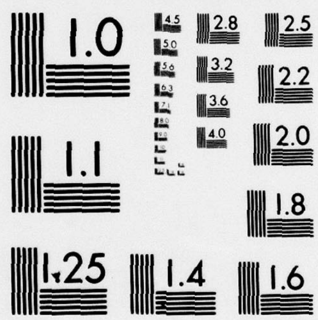

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SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE

READ INSTRUCTIONS BEFORE COMPLETING FORM

1. REPORT NUMBER: Technical Report No. 30 (14) TR-30	2. GOVT ACCESSION NO. / 3. RECIPIENT'S CATALOG NUMBER (9)
4. TITLE (and Subtitle): (6) Synthesis and Structure of a New Class of Metallophthalocyanato(pyridine or THF)-ruthenium(II) and carbonylphthalocyanato (pyridine or THF)osmium(II).	5. TYPE OF REPORT & PERIOD COVERED: Interim Rept.
	6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s): (10) M. Tsutsui, Shuichi Omiya	8. CONTRACT OR GRANT NUMBER(s): N00014-75-C-0417
9. PERFORMING ORGANIZATION NAME AND ADDRESS: Department of Chemistry, Texas A&M University, College Station, TX 77843	10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS: NR 053-559
11. CONTROLLING OFFICE NAME AND ADDRESS: Office of Naval Research, Department of the Navy, Arlington, Virginia 22217	12. REPORT DATE: (11) January 1980
	13. NUMBER OF PAGES: 54
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office): (15) N00014-75-C-0417	15. SECURITY CLASS. (of this report): (12) 56
	15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for Public Release; Distribution Unlimited

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

Submitted for publication in Inorganic Chemistry

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Metallophthalocyanines, Carbonylphthalocyanato(pyridine or THF)ruthenium, Carbonylphthalocyanato(pyridine or THF) osmium(II)

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Carbonylphthalocyanato(pyridine)ruthenium(II) PcRu(CO)(Py), carbonylphthalocyanatopyridineosmium(II) PcOs(CO)(Py) , and carbonylphthalocyanato (tetrahydrofuran)ruthenium(II) PcRu(CO)(THF) were prepared in pure form. They are among the first reported metallophthalocyanines with a carbonyl as one of their axial ligands. Furthermore, several new ways of synthesizing these complexes in quantitative yields have been established. The structure of PCOs(CO)(Py) elucidated by the X-ray diffraction analysis.

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S/N 0102-LF-014-6601

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ABSTRACT

During our synthesis of phthalocyanatoruthenium(II) and phthalocyanato-osmium(II) complexes reported by Berizin and Sennikova, and by Krueger and Kenny we observed a remarkable solubility of these complexes in common organic solvents. The solubility of these complexes enabled us to isolate several pure reported and new phthalocyanine complexes. Among the complexes we studied, carbonylphthalocyanato(pyridine)ruthenium(II)[PcRu(CO)(Py), carbonyl-phthalocyanatopyridineosmium(II)[PcOs(CO)(Py)], and carbonylphthalocyanato (tetrahydrofuran)ruthenium(II)[PcRu(CO)(THF)] were isolated in pure form. They are among the first reported metallophthalocyanines with a carbonyl as one of their axial ligands. Furthermore, several new ways of synthesizing these complexes in quantitative yields have been established. The structure of PcOs(CO)(Py) elucidated by the X-ray diffraction analysis. The osmium ion is octahedrally coordinated with the carbonyl and pyridine groups axially coordinated. The pyridine ring is tilted slightly with respect to the perpendicular to the phthalocyanine ring. The interplanar angle is 98.6°. Interesting comparisons may be made between PcOs(CO)(Py) and related porphyrin complexes;

Phthalocyanine and metallophthalocyanines demonstrate significant electrical and photo properties,[1,2] e.g. semiconductivity, photoconductiviety, photochemical reactivity, luminescence, and fluorescence, which are relevant to the current world problem of conversion and production of energy. The number of publications per year on chemistry of phthalocyanines and metallophthalocyanines has tripled during the past decade. During 1977, nearly 700 papers and patents were published. In spite of the increasing interest in this area and the near completion of the synthesis of all metal ions having the normal classic configuration, basic phthalocyanine chemistry has lacked the advantage of any dynamic or extraordinary progress. Many metalloporphyrins[3] have been made and studied extensively, and a large body of coordination chemistry of these complexes has been developed, while the coordination chemistry of metallophthalocyanines has been poorly developed. This contrast is mainly due to the significantly lower solubility of metallophthalocyanines and has led to the presumption that the development of coordination chemistry of phthalocyanines is difficult in general.

During our synthesis of pthalocyanatoruthenium(II) and phthalocyanatoosmium(II) complexes reported by Berizin and Sennikova[4,5] and by Krueger and Kenny,[6a] we have found a remarkable solubility of these complexes in common organic solvents. The solubility of these complexes enabled us to isolate several pure reported and new phthalocyanine complexes. Among the complexes we studied, carbonylphthalocyanato(pyridine)ruthenium(II)[PcRu(CO)(Py)], carbonylphthalocyanato(pyridine)osmium(II)[PcOs(CO)(Py)], and carbonylphthalocyanato(tetrahydrofuran)ruthenium(II)[PcRu(CO)(THF)] were isolated in pure form. These compounds are among the first reported metallophthalocyanines with carbonyl as one of their axial ligands. Furthermore, several new ways of synthesizing these complexes in quantitative yields have been established. Since the completion of this work,[6c] the preparation of PcRu(CO)(L)L (L=Py, 4-MePy; 4-t-BuPy) has been reported by N. P. Farrell _et al_.[6b] However, the methods of preparation described herein, which differ from their techniques, produce products in considerably higher yield. In this paper we also describe the structure of PcOs(CO)(Py)Py as determined from an x-ray diffraction analysis. This is the first reported structure of this type of phtalocyanine complex and provides interesting comparisons with analogous porphyrin complexes.

Experimental Section

Materials

Phthalonitrile (practical) was purchased from the Eastman Kodak Co. and used without any further purifications. Ruthenium-trichloride-(trihydrate)($RuCl_3.3(H_2O)$) and osmium tetraoxide(OsO_4) were purchased from the Ventron Corporation and used without any further purifications. Ruthenium dodecacarbonyl($Ru_3(CO)_{12}$) and osumium dodecrabonyl($Os_3(CO)_{12}$) were purchased from Strem Chemicals and used without purification. Carbon monoxide gas (99.9%) was purchased from the Matheson Gas Company and used without purification. Pyridine (reagent grade), tetrahydrofuran (reagent grade), methylene chloride (spectro grade), chloroform (spectro grade), and benzene (spectro grade) were used without further purification. Neutral alumina (60 - 100 mesh), acid alumina (80 - 200 mesh) and silica gel (60 - 200 mesh) were purchased from the Fisher Scientific Company.

Physical Measurements

Elemental analysis were performed by the Schwarzkopf Microanalytical Laboratory, N.Y. Visible spectra were measured with a Beckman Spectrophotometer Model 24. Infrared spectra were measured with a Beckman Infrared Spectrophotometer Model IR-8.

Isolation of PcRu(CO)(THF) from the Products of the Reaction of $RuCl_3 \cdot 3(H_2O)$ with Phthalonitrile

$RuCl_3 \cdot 3H_2O$ (1.00 g; 3.8 mmol) was heated with excess phthalonitrile (8.00 g; 62.5 mmol) at 250°C for 4 hours. After washing the resulting product with methanol several times, unreacted phthalonitrile was removed by sublimation at 150°C under vacuum. The blue-black residue (4.1 g), which was left at the bottom of the sublimator, was dissolved in aniline (10 mL), and metal-free phthalocyanine was removed from the solution by filtration. The aniline solution was poured into benzene (1.0 L) with stirring. A black precipitate (0.8 g) was separated by filtration from the benzene solution. The solution was then concentrated and dried under vacuum at 100°C until aniline in the solution was removed. The dried residue (3.5 g) was dissolved in benzene (30 mL), and then the solution was chromatographed over silica gel with benzene. Meanwhile, benzene was replaced by chloroform, and a light blue band, which was most intense, was separated from the dark blue band. The light blue eluate was condensed and chromatographed over neutral alumina with chloroform. Another bright blue band followed the original blue band when tetrahydrofuran was added to chloroform. The second blue eluate was condensed and dried. Fine needle crystals (120 mg) with a typical phthalocyanine appearance of red reflection and blue transmission were obtained. The visible spectrum of this compound shows maxima (in nm) at 642 and 581 (in chloroform). The infrared spectrum has an intense peak at 1960 cm^{-1}.

Preparation of PcRu(CO)(Py) from $RuCl_3 \cdot 3(H_2O)$ and Phthalonitrile in Carbon Monoxide

$RuCl_3 \cdot 3(H_2O)$ (100 mg; 0.38 mmol) was heated in molten phthalonitrile (500 mg; 1.9 mmol) under carbon monoxide atmosphere at 250°C for 4 hours. After cooling the product to room temperature, pyridine (5 mL) was added to the cake. The mixture was again heated to the boiling temperature of pyridine for 1 hour. The pyridine solution of the resulting product was cooled to room temperature, and then pyridine was removed first by distillation. Excess amounts of phthalonitrile were removed by sublimation at 150° under vacuum. The blue residue (265 mg) was dissolved in methylene chloride (15 mL), and the solution was chromatographed over neutral alumina with methylene chloride. The first blue band was followed by a second blue band when methylene chloride was replaced with chloroform. The second blue eluate, which was present in large quantity, was condensed and dried at 100°C under vacuum. A fine blue powder (230 mg) with red reflection (PcRu(CO)(Py)) was obtained (84% yield). Anal. Found: Ru, 13.97; N, 17.35. Calc. for PcRu(CO)(Py): Ru, 14.02; N, 17.49. The visible spectrum shows maxima (in nm) at 624 and 581 (in chloroform). The infrared spectrum shows an intense peak at 1965 cm^{-1}.

Preparation of PcRu(CO)(Py) from $Ru_3(CO)_{12}$ and Phthalonitrile

$Ru_3(CO)_{12}$ (100 mg; 0.16 mmol) was added to molten phthalonitrile (500 mg; 1.9 mmol) in air and heated at 250°C for 4 hours. The product was heated with pyridine (5 mL) at 150°C for 1 hour. Pyridine and unreacted phthalonitrile were removed, and the residue was dissolved in methylene chloride. Chromatography of the solution over neutral alumina gave a single blue band with chloroform. The chloroform eluate was condensed and dried. The blue powder (PcRu(CO)(Py)) with red reflection weighed 315 mg (91% yield).

<u>Isolation of PcOs(CO)(THF) from the Products of the Reaction of OsO_4 with Phthalonitrile</u>

OsO_4 (1.0 g; 3.9 mmol) was added to molten phthalonitrile (6.0 g; 46.8 mmol) in a 50 mL flask equipped with a condenser. The mixture was kept at 250°C for 4 hours. The excess phthalonitrile was removed from the product by sublimation. The residue was put in an extraction thimble, and some products were extracted with tetrahydrofuran. The THF solution was condensed and dissolved in chloroform. The chloroform solution was chromatographed over acid alumina with chloroform. The first blue band was collected and condensed. The condensed residue was then chromatographed over neutral alumina with chloroform. The first blue band of the second chromatography was collected and condensed. After drying at 100°C under vacuum, a blue powder (PcOs(CO)(THF)) was obtained (115 mg; 3.7% yield). Anal. Found: Os, 23.20; N, 14.15. Calc. for PcOs(CO)(THF): Os, 23.69; N, 13.96. The visible spectrum of the compound shows maxima (in nm) at 636, and 576 in THF. The infrared spectrum of the compound shows an intense peak at 1930 cm^{-1}.

Preparation of PcOs(CO)(Py) from OsO_4 and Phthalonitrile in Carbon Monoxide

OsO_4 (107 mg; 0.42 mmol) was added to molten phthalonitrile (500 mg; 3.9 mmol) in a 50 mL round bottom flask equipped with a condenser under carbon monoxide stream. The mixture was kept at 250°C for 4 hours under a carbon monoxide atmosphere. The mixture was then cooled to room temperature and dissolved in pyridine (5 mL). The pyridine solution was refluxed for 1 hour under a carbon monoxide atmosphere. Pyridine and phthalonitrile were removed under vacuum from the product. The resulting blue residue was dissolved in chloroform (50 mL), and metal-free phthalocyanine was removed from the chloroform solution by filtration. The chloroform solution was condensed and chromatographed over neutral alumina with chloroform. The single blue band was collected and condensed. A blue powder with red reflection(PcOs(CO)(Py)) was obtained after drying (280 mg; 83% yield). Anal. Found: Os, 22.96; N, 15.48. Calc. for PcOs(CO)(Py): Os, 23.49; N, 15.57. The visible spectrum of the compound shows maxima (in nm) at 632 and 575 (in chloroform). The infrared spectrum of the compound shows an intense peak at 1930 cm^{-1}.

Preparation of PcOs(CO)(Py) from $Os_3(CO)_{12}$ and Phthalonitrile

$Os_3(CO)_{12}$ (100 mg; 0.11 mmol) was added to molten phthalonitrile (500 mg; 3.9 mmol) in a 50 mL round bottom flask. The mixture was kept at 250°C for 4 hours. Pyridine (5 mL) was added to the mixture, and the pyridine solution was refluxed for 1 hour. Pyridine and excess phthalonitrile were removed from the product by distillation and sublimation. The blue residue was then dissolved in chloroform, and metal-free phthalocyanine was separated from the solution by filtration. The chloroform solution was condensed and chromatographed over neutral alumina with chloroform. The single blue band was collected and condensed. After drying at 100°C under vacuum, a blue powder with red reflection (PcOs(CO)(Py)) was obtained (220 mg; 82% yield).

X-ray Study

Crystals of PcOs(CO)(Py) were grown from a chloroform solution. The crystal chosen for intensity measurements was a parallellopiped bounded by {100}, {010} and {001}. The dimensions were 0.20 x 0.20 x 0.09 mm in the direction of $\underline{a}$, $\underline{b}$ and $\underline{c}$ respectively. It was mounted in a capillary [7] at an arbitrary orientation, but with $\underline{a}$ approximately parallel to the spindle axis.

Crystal Data are listed in Table 1. An Enraf-Nonius CAD-4 computer controlled diffractometer was used. The radiation (Mo Kα, λ=0.71069Å) was monochromatized by pyrolitic graphite. The instrument centered the crystal automatically. The setting angles for 25 reflections, measured at + and - 2Θ, were used to index the cell and then were refined to give an orientation matrix, cell constants, and a Niggli matrix[8] which indicated that the system was monoclinic.[9] The systematic absences uniquely determined the space group.

The diffracted intensities were collected using the Θ-2Θ scan technique. Scan speeds, which were determined by a rapid preliminary scan, ranged from 0.28 to 3.35 deg/min. depending on the intensity. Very weak reflections were measured at the maximum rate. The scan range for each reflection was equal to 0.90 + 0.35 tan Θ. Other experimental conditions are described elsewhere.[10] No evidence of crystal decomposition or machine instability was noted.

Independent reflections (4596) were measured out to a sinΘ value of 0.54 or 22.5° in Θ. Of these, 2820 had a net intensity greater than $2\sigma_I$ and were used in analysis. The standard deviation σ_I was defined in terms of the statistical variances of the counts as $\sigma_I^2 = \sigma_{I(count)}^2 + (0.02I)^2$ $\sigma_{I(count)}$ is the variance determined solely from counting statistics. Structure factors were calculated in the usual way, including correction for partial polarization of the incident beam due to the use of a monochromator.

Determination and Refinement of the Structure

Because there are four molecules in the unit cell of space group $P2_1/n$, all atoms lie in general positions. The position of the osmium atom was found from an unsharpened Patterson synthesis. The rest of the 49 nonhydrogen atoms were found from a series of difference syntheses. Least-squares refinement using full matrix methods was carried out minimizing the function $\Sigma w(|F_o| - |F_c|)^2$, where $w = 1/\sigma_F^2$. Initially isotropic temperature factors were used, but in the final refinements all nonhydrogen atoms were varied assuming anisotropic thermal motion. The positions of 18 of the 21 hydrogen atoms could be found from ΔF maps. However, refinement of the hydrogen atom parameters led to chemically unreasonable bond lengths and angles. Hence the positions of all the hydrogen atoms were calculated (C-H = 0.95A) and their contributions included in the structure factor calculations, assuming an isotropic temperature factor, B, of $4.0A^2$. The hydrogen atom parameters were not refined. The refinement converged with $R = \Sigma||Fo|-|Fc||/\Sigma|Fo| = 0.031$ and $R_w = (\Sigma w(|F_o|-|F_c|)^2/\Sigma w|Fo|^2)^{½} = 0.035$.

A correction for anomalous dispersion was made for all nonhydrogen atoms. Scattering factors were from Ref. 12. The osmium atom was assumed to be in the zero ionization state. No evidence of secondary extinction was found.

Attempts to apply absorption corrections were made. Transmission coefficients, calculated using a Gaussian integration method (6x4x6 grid), varied from 0.43 to 0.68 with most being about 0.6. The R factors increased substantially ($R=0.049$, $R_w=0.056$) when refinements were attempted using the corrected data. No improvement was noted in the standard deviations. It was concluded that the errors introduced in applying the corrections to a relatively small crystal enclosed in a capillary were larger than those introduced by ignoring absorption effects. Thus, the final refinements were carried out on uncorrected data.

In the last cycle of refinement all shifts on positional and thermal parameters were less than one standard deviation, with the largest shift being 0.37 standard deviations. The final value of the standard deviation of an observation of unit weight, defined as $[\Sigma w(|Fo|-|Fc|)^2/(N_o-N_v)]^{1/2}$ was 0.944 for $N_o = 2820$ reflections and $N_v = 442$ parameters.

There were two peaks of about 1.5 e/A^3 in the final difference Fourier. These were quite close to the osmium atom. Neither they nor any of the other peaks were considered physically significant.

Most calculations were performed on a PDP 11/40 computer using the Enraf-Nonius structure determination package (SDP). Johnson's ORTEP,[13] some molecular geometry calculations (using XANADU by Roberts and Sheldrick) and local programs were run on an Amdahl 470v/6 computer. Use was made of the PDP 11/40-Vector General graphics system.[14] Data reduction was performed on the Honeywell computer at the University of Houston.

The final positional and thermal parameters are given in Table II. The final calculated positions of the hydrogen atoms are given in Table III. Tables IV and V contain the root-mean-square components of thermal displacement along the principal axis of the thermal ellipsoids and the observed and calculated structure factors respectively. Tables III-V are available as supplementary material.

Results and Discussion

The following reactions for the synthesis of phthalocyanatoruthenium complexes have been carefully examined, and considerable discrepancies from the reported results[4] have been found. Furthermore, a new carbonyl complex of ruthenium phthalocyanine has been isolated from the reaction products. The reaction of $RuCl_3 \cdot 3(H_2O)$ with phthalonitrile was reported to give PcRu(III)Cl as the major product of the reaction by Berizin and Sennikova,[4,5] as shown in the following equation:

$$RuCl_3(H_2O) + C_6H_4(CN)_2 \rightarrow PcRu(III)Cl$$

The reaction of $RuCl_3 \cdot 3(H_2O)$ with o-cyanobenzoamide was reported to give PcRu(II) as its major product by Krueger and Kenny,[6a] as shown in the following equation:

$$RuCl_3 \cdot 3(H_2O) + NCC_6H_4CONH_2 \rightarrow PcRu(II)$$

From our investigations both of these reactions have been found to give PcRu(II) as the major product and PcRu(III)Cl as the minor one.

In addition, a small quantity of PcRu(II)(CO)L (L is a solvent used for the isolation) was detected in the crude $PcRu(II) \cdot 6C_6H_5NH_2$ according to its infrared spectrum, and was isolated by column chromatography. The yield of PcRu(II)(CO)L was less than 5%. Thus, the reactions above should be expressed by the following equations:

$$RuCl_3 \cdot 3(H_2O) + C_6H_4(CN)_2 \rightarrow PcRu(II) + PcRu(III)Cl + PcRu(II)(CO)L$$

$$RuCl_3 \cdot 3(H_2O) + NCC_6H_4CONH_2 \rightarrow PcRu(II) + PcRu(III)Cl + PcRu(II)(CO)L$$

The yield of 5% for CO complex is rather surprising in two respects. In the first place, the source of carbon monoxide is unknown. Since the reaction of $RuCl_3 \cdot 3(H_3O)$ with phthalonitrile was carried out in air without solvent there should not be any direct carbonyl sources in the system. In the second place, the major product was four-coordinate PcRu(II), whereas it appears that stable Ru(II) porphyrin complexes are generally six coordinate.[15-20]

This carbonyl complex of ruthenium phthalocyanine can be synthesized in tow different ways directly in high yield (80-90%). The reaction of $RuCl_3 \cdot 3(H_2O)$ with excess phthalonitrile under carbon monoxide atmosphere gives an almost quantitative yield even after the isolation of the compound through column chromatography. The reaction of $Ru_3(CO)_{12}$ with excess phthalonitrile also gives a high yield of PcRu(II)(CO)(Py). This reactions are shown in the following equations:

$$RuCl_3 \cdot 3(H_2O) + C_6H_4(CN)_2 + CO \rightarrow PCRu(II)(CO)L \xrightarrow{pyridine} PcRu(II)(CO)(Py)$$

$$Ru_3(CO)_{12} + C_6H_4(CN)_2 \longrightarrow RcRu(II)(CO)L \xrightarrow{pyridine} PcRu(II)(CO)(Py)$$

The carbonyl complex of ruthenium phthalocyanine was characterized by infrared and visible absorption spectroscopy and elemental analysis. The infrared spectrum of the compound shows an intense band at 1965 cm^{-1} which is assigned to $\nu C\equiv O$ attached to ruthenium ion.

A well-defined carbonyl complex of osmium phthalocyanine was also isolated from the products of the reported reactions of OsO_4 with molten phthalonitrile.[5] Although the yield of PcOs(II)(CO)L is less than 5%, it it isolated as a pure complex by column chromatography. The isolation was made possible by the remarkable solubility of the complex in common organic solvents. The presence of a carbonyl ligand attached to osmium metal was evidenced by an intense band at 1930 cm^{-1} in the infrared spectrum. This new type of compound, PcOs(II)(CO)L, coordinates another donor molecule as its last axial ligand. For example, tetrahydrofuran and pyridine can coordinate to the open sixth site of osmium to form a stable compound with octahedral configuration.

These carbonyl complexes of osmium phthalocyanine can be prepared in high yield (80-90%) by the reaction of OsO_4 with molten phthalonitrile in carbon monoxide atmosphere or the reaction of $Os_3(CO)_{12}$ with phthalonitrile in air. Subsequent treatment with pyridine or THF yield PcOs(II)(CO)(Py) and PcOs(II)(CO)(THF) respectively. Column chromatography gives a single blue band of each complex. An x-ray diffraction analysis has been carried out on the former complex.

$$\left.\begin{array}{l} OsO_4 + C_6H_4(CN) + CO \\ Os(CO)_{12} + C_6H_4(CN)_2 \end{array}\right] \text{PcOs(II)(CO)L} \left[\begin{array}{l} \xrightarrow{\text{pyridine}} \text{PcOs(II)(CO)(Py)} \\ \xrightarrow{\text{THF}} \text{PcOs(II)(CO)(THF)} \end{array}\right.$$

Carbonyl complexes of ruthenium and osmium porphines have been synthesized and characterized by a number of workers.[15-20]

Table VI summarized the carbonyl stretching frequencies of these complexes together with those of the carbonyl complexes of ruthenium and osmium phthalocyanocines.

(Tetraphenylporphinato)(carbonyl)(pyridine)ruthenium(II) (TPPRu(CO)(Py)) shows a carbonyl peak at 1939 cm^{-1}, which is slightly lower than that (1965 cm^{-1}P of PcRu(CO)(Py). Therefore, the back donation of 4d electrons of ruthenium to the anti-bonding π-orbitals of the carbonyl ligand seems to be less in the phthalocyanine complex than in the TPP complex. The same phenomenon is observed in osmium complexes. (Octaethylporphinato)(Carbonyl)(pyridine)Os(II) (OEPOs(CO)(Py)) shows a carbonyl peak at 1902 cm^{-1}, while PcOs(CO)(Py) shows a carbonyl peak at 1930^{-1}. In this case back donation of 5d electrons from osmium to the anti-bonding π-orbital of the carbonyl ligand seems to be less in the phthalocyanine than in the OEP complex. Probably the structural difference of the rings causes the difference in the degree of the back donoation of d-electrons from the central metal ions to the coordinated carbon monoxide. Apparently, Ru(II) or Os(II) in the phthalocyanine carbonyl complexes donate less electron density to the carbonyl ligand than those in porphyrin rings.

It should also be noted that the PcRu(II) complex, which does not have a carbonyl ligand, is capable of coordinating carbon monoxide at room temperature under 1 atmosphere of carbon monoxide. PcRu(II) can be prepared by removing aniline molecules from PcRu(II)·6($C_6H_5NH_2$) under vacuum at high temperature (200°C) and it can form a carbonyl complex in THF or pyridine by bubbling CO gas into the solution for one day. The original PcRu(II)·6($C_6H_5NH_2$) can be recovered by refluxing PcRu(II) in freshly distilled aniline for a few minutes as shown in the following equations:

$$\text{PcRu(II)}\cdot 6(C_6H_5NH_2) \underset{\Delta,\, C_6H_5NH_2}{\overset{\Delta,\,\text{vacuum}}{\rightleftarrows}} \text{PcRu(II)} \xrightarrow[\text{CO in THF}]{\text{CO in pyridine}} \begin{matrix}\text{PcRu(II)(CO)(Py)} \\ \text{PcRu(II)(CO)(THF)}\end{matrix}$$

The carbonylation process can be traced by noting changes in its visible spectrum as shown in Fig. I.

PcRu(II) is expected to demonstrate an interesting chemistry, because it does not possess a carbonyl ligand. On the other hand, the chemistry of ruthenium porphyrin is rather limited becasue removal of the carbonyl ligand is extremely difficult.[16,21] PcRu(II) appears to have the potential to form new ruthenium phthalocyanine complexes with various other molecules such as O_2, N_2, NO, and olefins. Also, the remarkable solubility of ruthenium and osmium phthalocyanine complexes will possibly open a new era in the coordination chemistry of metallophthalocyanines.

DESCRIPTION OF STRUCTURE OF PcOs(CO)(Py)

The structure of (carbonyl)(pyridine)phthalocyanatoosmium(II) is shown in Figure 2. Stereoviews are shown in Figures 3 and 4. The osmium ion is octahederally coordinated with the carbonyl and pyridine groups axially coordinated. The pyridine ring is tilted slightly with respect to the perpendicular to the phthalocyanine ring. The interplanar angle is 98.6°.

The structure of an analogous osmium porphyrin complex has not been reported, but the structure of (carbonyl)(pyridine)tetraphenylporphinatoruthenium(II), TPPRu(CO)(Py) has been published.[22] Osmium and ruthenium have similar covalent radii.[23] The phthalocyanine complex bears a strong resemblance to this metalloporphine. In addition, the structure of a carbonyl osmium porphodimethene complex with pyridine as the other axial ligand, $OEPMe_2Os(CO)(Py)$ has been reported.[24] Interesting comparisons may be made between these two complexes and PcOs(CO)(Py).

Bond lengths and angles are given in Table VII. The average interatomic distances between the osmium atom and the isoindole nitrogen atoms of the phthalocyanine molecule is 2.01A. However, it should be noted that the bond lengths for Os-N(2) and Os-N(6) are 1.98A, while the other two distances are 2.03A. The difference corresponds to ~5 standard deviations, so it is statistically significant. The C-N-C and N-C-C angles involving the isoindole nitrogen atoms also differ in the two pairs of isoindole groups. Thus, for groups 1 and 3 (containing N(2) and N(6), the average C-N-C angle is 112°, while the N-C-C an is 107°. In groups 2 and 4 (containing N(4) and N(8)) the corresponding angles are 107° and 111°. The differences once again correspond to about 4 or 5 standard deviations and are therefore significant. We are unable to offer a plausible explanation for these differences, which are not observed in other phthalocyanine complexes.

The size of the central "hole" in phthalocyanine complexes has been estimated to be 0.046 -0.050A smaller than in corresponding porphyrin complexes.[25] A similar difference is noted in the present case. The osmium atom is slightly out of the plane of the phthalocyanine molecule, so the size of the hole has to be estimated from the distances between opposite isoindole nitrogen atoms. These distances are 4.06 and 3.96A, corresponding to an average radius (Ct-N distance) of 2.01A for the macrocyclic hole. In TPPRu(CO)(Py)[22] the corresponding distance is 2.05A. The M-N distances are correspondingly longer. In both TPPRu(CO)(Py)[22] and $OEPMe_2Os(CO)(Py)$[24] these average 2.06A.

The Os-N bond lengths involving the isoindole nitrogen atoms are short for such bonds. On the other hand the $Os-N_{Py}$ (N_{Py} is the pyridine nitrogen atom) distance of 2.209(9)A is relatively long. $Ru-N_{Py}$ distances of 2.06-2.09Å have been reported.[26] $M-NH_3$ bond lengths in amine complexes of Os(II) and Ru(II) range from 2.12-2.14A.[26-29] The $M-N_{Py}$ distance in both TPPRu(CO)(Py) and $OEPMe_2Os(CO)(Py)$ is also elongated (2.193(4) and 2.230(4)A respectively.)

This long bond may be due to steric interactions between atoms in the pyridine ring, particularly the α-hydrogen atoms, and atoms in the macrocycle. This would block a closer approach to the osmium atom by the pyridine molecule. The osmium atom lies 0.15A on the other side of phthalocyanine molecule. However, the closest contacts involving the α-hydrogen atoms are between H(33) and C(32) and between H(37) and C(16). These contacts are both about 2.73A, which is not an unusually short contact distance in such cases. A tabulation of such contacts in porphyrin complexes containing planar axial ligands shows a range of 2.45-2.9A.[30] In TPPRu(CO)(Py) the closest contact is 2.51A.

The angle between the plane of the pyridine ring and the plane defined by N(2), Os, and N(6) is 48°. An angle of ~45° minimizes steric interaction, whereas an angle of ~0° would bring the α-hydrogen atoms into close contact with the isoindole nitrogen atoms.[31]

It has also been suggested a lengthening of ~0.1A will occur in the axial M-N bond due to the trans effect of the carbonyl group.[22] In $OEPRu(Py)_2$ [32] the observed axial bond lenghts are indeed shorter, 2.09-2.10A, in spite of some relatively close contacts of the α-hydrogen atoms of the pyridine ligands with atoms in the macrocycle.[30] An even shorter M-N bond length of 2.00A has been reported in $PcFe(Py)_2$.[33]

The metal carbonyl distance of 1.83(1) is in good agreement with the values found in TPPRu(CO)(Py) $OEPMe_2Os(CO)(Py)$. The Os-C-O angle is linear (177°).

Least squares plane of interest are given in Table VIII. As mentioned previously, the osmium atom is 0.15A out of the plane defined by the phthalocyanine group in a direction toward the carbonyl group. The displacement decreases to 0.099A if one considers only the plane of the four isoindole nitrogen atoms. By way of comparison, the ruthenium atom in TPPRu(CO)(Py) is 0.079A out of the plane of the porphyrin skeleton toward the carbonyl group.

In the discussion on TPPRu(CO)(Py) the authors attributed the out-of-plane displacement of the metal ion to either very strong metal-carbonyl bonds or to the inability of the large metal ion to fit into the plane of the macrocycle.[22] The former possibility now appears more likely, since in $OEPRu(Py)_2$ the ruthenium(II) ion lies in the plane of the macrocycle. The same factor is probably primarily responsible for the out-of-plane displacement observed for the metal ion in this phtalocyanine complex.

The difference of 0.065A in the displacements of the metal ion from the planes of the macrocycle and of the isoindole nitrogen atoms indicate that the macrocycle itself is "domed", corresponding to a C_{4v} deviation from planarity. Deviations of equal or larger magnitude have been observed in other metallophthalocyanine complexes, where the metal ion is out of the plane of the macrocycle.[34-37] The difference between the deviation of the osmium atom from the plane of the four isoindole nitrogen atoms and the plane defined by the pyrrole rings and the bridging nitrogen atoms is 0.027A, a value falling in the middle of the range found for porphyrins with the metal ion out of the macrocyclic plane.[38]

The doming is not equal for the four isoindole groups. The maximum deviations from the plane of the four isoindole nitrogen atoms are 0.23 and 0.35A for phenyl carbon atoms in groups 2 and 3, while the maximum deviations in the same direction in groups 1 and 4 are 0.04 and 0.12A. Such a pattern was observed in aquophthalocyanatomagensium(II).[36]

There appears to be some variation in bond parameters of phthalocyanine complexes as the size of the central "hole" increases.[39] The C-N-C angle involving the azamethine nitrogen atom is the most sensitive bond parameter. This angle ranges from 121.7° in Fe(Pc)[25] (Ct-N: 1.93A) to 126.2° in $Cl_2Sn(Pc)$[40] (Ct-N: 2.05A). The average value of 125(1)° found for this angle in PcOs(CO)(Py) agrees with that found in phthalocyanine complexes with similar Ct-N distances.[35,39] The presence of a very heavy metal atom like osmium decreases the accuracy with which the lighter atoms can be determined, but within the observed standard deviations, the other bond parameters agree with those reported in the accurately determined structure of Zn(Pc)[39] and Sn(Pc).[34]

A packing diagram of the unit cell is shown in Figure 3. Table IX lists the intermolecular contacts $\leq$ 3.5A. Most of the shortest contacts involve the carbonyl oxygen atom. Non-bonded contacts of this magnitude have been observed in other carbonyl complexes (e.g. $[H_3OEP]^+$ $[Re_2(CO)_6Cl_3]^-$).[41] Neither these nor any of the other intermolecular contacts are believed to have any significant effect on the structure.

The structure of PcRu(CO)(Py) probably possesses a structure similar to that of OsPc(CO)Py.

Acknowledgment. This work was supported in part by the Office of Naval Research and the Robert A. Welch Foundation (Grant A-328) and the Texas Agricultural Experiment Station (Grant H-1668).

Supplementary Material Available: Table III-V containing calculated hydrogen atom positions, root-mean-square components of thermal ellipsoids and observed and calculated structure factors (pages). Ordering information is given on any current masthead page.

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TABLE I

Crystal Data for $(C_{38}H_{21}N_8)Os$[a]

a = 11.966(9) A	Fw = 809.9
b = 15.705(3) A	Z = 4
c = 17.749(5) A	d calcd = 1.721
	μ = 43.9 cm^{-1} (MoK α radiation)
B = 107.92 (4)°	Systematic absences h0l (h+l odd) 0k0 (k odd)
	Space group P 2_1/n
V = 3125(4) A^3	

[a]In this and subsequent tables the estimated standard deviation of the least significant figures shown in parentheses.

Table II Fractional Coordinates and Thermal Motion Parameters Derived from the Least-Squares Refinement

ATOM	X	Y	Z	U(11)	U(22)	U(33)	U(12)	U(13)	U(23)
OS	0.22473(4)	0.11171(3)	0.11752(3)	352(2)	303(2)	276(2)	-1(7)	274(3)	61(6)
N(1)	0.4585(7)	0.1150(6)	0.0454(4)	33(4)	41(5)	37(4)	0(12)	31(7)	1(11)
N(2)	0.2651(7)	0.1753(6)	0.0286(4)	31(4)	34(5)	31(4)	1(11)	27(7)	31(11)
N(3)	0.0855(7)	0.2557(5)	-0.0241(5)	42(5)	34(6)	26(5)	-5(10)	16(8)	17(9)
N(4)	0.0711(7)	0.1710(6)	0.0896(4)	32(5)	47(6)	23(4)	-5(10)	21(7)	33(9)
N(5)	-0.0051(6)	0.1119(6)	0.1914(4)	33(4)	32(5)	28(4)	8(11)	22(7)	-9(10)
N(6)	0.1925(7)	0.0575(5)	0.2143(4)	38(5)	29(5)	28(4)	-6(9)	33(7)	15(8)
N(7)	0.3804(7)	-0.0112(5)	0.2739(5)	33(5)	28(5)	43(5)	13(9)	33(8)	16(9)
N(8)	0.3844(7)	0.0620(6)	0.1521(4)	37(5)	47(6)	24(4)	-10(10)	25(8)	32(9)
C(1)	0.3667(9)	0.1631(7)	0.0084(6)	48(7)	38(7)	30(6)	-31(13)	35(10)	-21(11)
C(2)	0.3569(10)	0.2151(7)	-0.0625(6)	55(7)	27(6)	26(5)	-27(12)	37(10)	-8(10)
C(3)	0.4347(10)	0.2275(8)	-0.1073(6)	52(7)	48(8)	46(6)	-14(13)	58(10)	-4(13)
C(4)	0.3991(11)	0.2840(8)	-0.1704(6)	81(8)	56(8)	45(6)	-20(15)	86(10)	11(13)
C(5)	0.2923(11)	0.3273(8)	-0.1889(6)	96(9)	47(8)	39(6)	-50(15)	51(12)	21(13)
C(6)	0.2155(10)	0.3135(7)	-0.1460(6)	62(8)	39(7)	33(6)	-14(13)	40(11)	4(12)
C(7)	0.2483(9)	0.2568(7)	-0.0826(5)	50(7)	34(7)	22(5)	-13(12)	29(10)	1(11)
C(8)	0.1908(9)	0.2290(7)	-0.0237(6)	50(7)	30(7)	29(6)	-14(12)	36(10)	5(11)
C(9)	0.0300(9)	0.2300(7)	0.0282(6)	40(7)	30(7)	34(6)	-4(12)	18(10)	-5(11)
C(10)	-0.0855(10)	0.2601(7)	0.0268(6)	51(7)	34(7)	27(6)	10(13)	20(10)	-1(11)
C(11)	-0.1664(10)	0.3177(7)	-0.0210(6)	62(8)	41(7)	39(7)	33(14)	18(12)	24(12)
C(12)	-0.2715(10)	0.3290(8)	-0.0091(7)	46(7)	56(8)	65(8)	61(14)	20(13)	17(15)
C(13)	-0.3023(10)	0.2830(8)	0.0492(7)	51(7)	52(8)	66(8)	44(14)	40(12)	7(14)
C(14)	-0.2244(9)	0.2267(7)	0.0985(6)	34(6)	49(8)	35(6)	16(12)	25(10)	-2(12)
C(15)	-0.1160(10)	0.2150(7)	0.0874(6)	46(7)	25(6)	36(6)	10(12)	19(11)	-9(11)
C(16)	-0.0135(9)	0.1622(7)	0.1269(5)	38(6)	23(6)	23(5)	15(11)	6(10)	15(10)
C(17)	0.0883(9)	0.0669(7)	0.2311(6)	28(6)	36(7)	29(5)	-9(11)	29(9)	-5(11)
C(18)	0.1005(9)	0.0206(6)	0.3057(6)	37(6)	22(6)	28(6)	1(11)	22(9)	12(10)
C(19)	0.0226(9)	0.0057(7)	0.3493(6)	47(6)	38(7)	39(6)	-2(12)	55(9)	-5(11)
C(20)	0.0643(10)	-0.0424(7)	0.4189(6)	76(7)	39(7)	45(6)	0(13)	90(10)	15(12)
C(21)	0.1783(11)	-0.0752(7)	0.4443(6)	77(8)	46(8)	46(6)	9(14)	70(11)	29(12)
C(22)	0.2542(9)	-0.0603(7)	0.4014(6)	40(7)	50(8)	36(7)	13(13)	22(11)	4(12)
C(23)	0.2153(10)	-0.0139(7)	0.3308(5)	58(7)	26(6)	23(5)	5(12)	32(10)	14(10)
C(24)	0.2726(10)	0.0111(6)	0.2707(6)	52(7)	26(6)	24(6)	13(12)	21(10)	15(10)
C(25)	0.4314(9)	0.0105(7)	0.2180(6)	33(6)	32(7)	41(6)	-12(12)	28(10)	2(12)
C(26)	0.5508(9)	-0.0171(7)	0.2209(6)	34(6)	29(7)	40(6)	8(12)	19(10)	2(11)
C(27)	0.6338(9)	-0.0660(8)	0.2741(6)	34(6)	55(8)	50(7)	18(13)	35(10)	13(13)
C(28)	0.7382(10)	-0.0813(7)	0.2582(7)	38(7)	40(7)	72(8)	23(12)	32(12)	12(13)
C(29)	0.7596(10)	-0.0452(7)	0.1915(7)	38(7)	41(8)	65(7)	17(13)	43(11)	-12(13)
C(30)	0.6756(10)	0.0044(7)	0.1377(6)	48(7)	33(7)	52(7)	0(12)	46(10)	1(12)
C(31)	0.5704(9)	0.0193(7)	0.1533(6)	29(6)	33(7)	44(6)	-6(11)	29(10)	-2(11)
C(32)	0.4680(9)	0.0661(7)	0.1120(6)	43(7)	31(6)	29(6)	11(12)	14(10)	19(11)
N(9)	0.3026(7)	0.2178(5)	0.1988(5)	37(5)	37(5)	27(5)	-15(10)	23(8)	-1(9)
C(33)	0.4083(9)	0.2481(7)	0.2054(6)	31(6)	37(7)	40(6)	-15(12)	14(10)	-14(12)
C(34)	0.4642(11)	0.3067(8)	0.2613(7)	52(8)	46(8)	65(8)	-6(14)	14(14)	3(14)
C(35)	0.4107(11)	0.3387(7)	0.3133(7)	61(8)	32(7)	48(7)	-14(14)	0(13)	-14(13)
C(36)	0.2990(11)	0.3112(7)	0.3062(6)	84(9)	36(7)	41(6)	-13(14)	58(12)	-24(12)
C(37)	0.2474(10)	0.2520(7)	0.2489(6)	44(6)	32(6)	55(7)	-19(13)	49(10)	8(12)
C(38)	0.1629(9)	0.0225(8)	0.0509(6)	50(6)	97(9)	27(5)	-80(14)	66(9)	-33(13)
O(1)	0.1273(8)	-0.0374(5)	0.0109(5)	95(7)	64(6)	67(5)	-35(11)	56(10)	-59(10)

[a]The Debye-Waller Factor is defined as $T=\exp[-2\pi^2(U_{11}a^{*2}h^2+U_{22}b^{*2}k^2+U_{33}c^{*2}l^2+U_{12}a^*b^*hk+U_{13}a^*c^*hl+U_{33}b^*c^*kl)]$. The values for U have been multiplied by 10^3, except for those of Os, which have been multiplied by 10^4.

Table VI. Comparison of CO Frequencies of MP(CO)(Py)

PcM(CO)(Py)	RuPc	RuTPP	OsPc	OsTPP	OsOEP
$\nu C \equiv O (cm^{-1})$	1965	1939	1930	1920	1902

P = Pc, TPP, and OEP

Pc = phthalocyanine

TPP = tetraphenylporphine

OEP = octaethylporphyrin

Py = pyridine

M = Os or Ru

TABLE VII

Bond Lengths (Å) and Angles (deg)[a]

Os-N(2)	2.027(9)		N(2)-Os-N(4)	90.1(4)	
Os-N(4)	1.983(9)	2.01(3)	N(2)-Os-N(8)	89.6(4)	
Os-N(6)	2.034(9)		N(4)-Os-N(6)	90.1(4)	89.9(3)
Os-N(8)	1.978(9)		N(6)-Os-N(8)	89.7(4)	
Os-N(9)	2.202(9)		N(2)-Os-N(6)	174.2(4)	174.3(2)
Os-C(38)	1.83(1)		N(4)-Os-N(8)	174.5(4)	
N(2)-C(1)	1.38(1)		N(2)-Os-N(9)	88.2(4)	
N(2)-C(8)	1.36(1)		N(4)-Os-N(9)	88.8(4)	
N(4)-C(9)	1.39(1)		N(6)-Os-N(9)	86.0(4)	87(1)
N(4)-C(16)	1.37(1)	1.37(1)	N(8)-Os-N(9)	85.7(4)	
N(6)-C(17)	1.37(1)		N(2)-Os-C(38)	91.9(5)	
N(6)-C(24)	1.36(1)		N(4)-Os-C(38)	92.3(5)	
N(8)-C(25)	1.38(1)		N(6)-Os-C(38)	93.8(5)	93(1)
N(8)-C(32)	1.39(1)		N(8)-Os-C(38)	93.2(5)	
N(1)-C(1)	1.33(1)		N(9)-Os-C(38)	178.9(6)	
N(1)-C(32)	1.33(1)		Os-N(2)-C(1)	124.3(8)	
N(3)-C(8)	1.35(1)		Os-N(2)-C(8)	124.3(8)	
N(3)-C(9)	1.36(1)		Os-N(4)-C(9)	125.8(8)	125(2)
N(5)-C(16)	1.32(1)	1.34(2)	Os-N(4)-C(16)	126.9(7)	
N(5)-C(17)	1.32(1)		Os-N(6)-C(17)	123.3(8)	
N(7)-C(24)	1.35(1)		Os-N(6)-C(24)	123.8(9)	
N(5)-C(25)	1.37(1)		Os-N(8)-C(25)	126.4(8)	
C(1)-C(2)	1.46(1)		Os-N(8)-C(32)	127.3(8)	
C(7)-C(8)	1.47(1)		C(1)-N(2)-C(8)	111(1)	
C(9)-C(10)	1.45(2)		C(9)-N(4)-C(16)	107(1)	109(3)
C(15)-C(16)	1.47(1)	1.46(2)	C(17)-N(6)-C(24)	113(1)	
C(17)-C(18)	1.46(1)		C(25)-N(8)-C(32)	106(1)	
C(23)-C(24)	1.47(2)				
C(25)-C(26)	1.48(2)		C(1)-N(1)-C(32)	125(1)	
C(31)-C(32)	1.42(2)		C(8)-N(3)-C(9)	126(1)	125(1)
C(2)-C(7)	1.40(2)		C(16)-N(5)-C(17)	124(1)	
C(10)-C(15)	1.42(1)		C(24)-N(7)-C(25)	125(1)	
C(18)-C(23)	1.41(2)	1.41(1)	N(1)-C(1)-N(2)	128(1)	
C(26)-C(31)	1.40(2)		N(2)-C(8)-N(3)	125(1)	
C(2)-C(3)	1.40(2)		N(3)-C(9)-N(4)	126(1)	
C(6)-C(7)	1.38(1)		N(4)-C(16)-N(5)	125(1)	
C(10)-C(11)	1.40(2)		N(5)-C(17)-N(6)	128(1)	127(2)
C(14)-C(15)	1.38(2)	1.39(1)	N(6)-C(24)-N(7)	129(1)	
C(18)-C(19)	1.39(1)		N(7)-C(25)-N(8)	127(1)	
C(22)-C(23)	1.39(1)		N(8)-C(32)-N(1)	125(1)	
C(26)-C(27)	1.37(2)		N(1)-C(1)-C(2)	125(1)	
C(30)-C(31)	1.39(2)		N(3)-C(8)-C(7)	123(1)	
C(3)-C(4)	1.38(2)		N(3)-C(9)-C(10)	124(1)	
C(5)-C(6)	1.37(2)		N(5)-C(16)-C(15)	124(1)	
C(1)-C(12)	1.35(2)		N(5)-C(17)-C(18)	125(1)	124(1)
C(13)-C(14)	1.38(2)		N(7)-C(24)-C(23)	124(1)	
C(19)-C(20)	1.39(2)	1.38(1)	N(7)-C(25)-C(26)	123(1)	
C(21)-C(22)	1.36(2)		N(1)-C(32)-C(31)	124(1)	
C(27)-C(128)	1.38(2)				
C(29)-C(130)	1.39(2)				

C(4)-C(5)	1.40(2)	
C(12)-C(13)	1.39(2)	1.39(1)
C(20)-C(21)	1.40(2)	
C(28)-C(29)	1.39(2)	
N(9)-C(33)	1.32(1)	1.34(3)
N(9)-C(37)	1.36(2)	
C(33)-C(34)	1.36(2)	
C(34)-C(35)	1.36(2)	1.36(1)
C(35)-C(36)	1.37(2)	
C(36)-C(37)	1.37(2)	
C(38)-O(1)	1.17(1)	
N(2)-N(4)	2.84(1)	
N(2)-N(8)	2.82(1)	2.83(1)
N(4)-N(6)	2.84(1)	
N(6)-N(8)	2.83(1)	
N(2)-N(6)	4.06(1)	4.01(7)
N(4)-N(8)	3.96(1)	
N(1)-N(5)	6.78(1)	6.78(1)
N(3)-N(7)	6.77(1)	

N(2)-C(1)-C(2)	107(1)	
N(2)-C(8)-C(7)	108(1)	
N(4)-C(9)-C(10)	110(1)	
N(4)-C(16)-C(15)	111(1)	
N(6)-C(17)-C(18)	106(1)	109(2)
N(6)-C(24)-C(23)	106(1)	
N(8)-C(25)-C(26)	110(1)	
N(8)-C(32)-C(31)	111(1)	
C(1)-C(2)-C(7)	108(1)	
C(2)-C(7)-C(8)	106(1)	
C(9)-C(10)-C(15)	107(1)	
C(10)-C(15)-C(16)	105(1)	
C(17)-C(18)-C(23)	107(1)	107(1)
C(18)-C(23)-C(24)	107(1)	
C(25)-C(26)-C(31)	105(1)	
C(26)-C(31)-C(32)	107(1)	
C(1)-C(2)-C(3)	131(1)	
C(6)-C(7)-C(8)	132(1)	
C(9)-C(10)-C(11)	134(1)	
C(14)-C(15)-C(16)	134(1)	132(1)
C(17)-C(18)-C(19)	132(1)	
C(22)-C(23)-C(24)	132(1)	
C(25)-C(26)-C(27)	132(1)	
C(30)-C(31)-C(32)	133(1)	
C(7)-C(2)-C(3)	121(1)	
C(2)-C(7)-C(6)	121(1)	
C(15)-C(10)-C(11)	119(1)	121(1)
C(10)-C(15)-C(14)	121(1)	
C(23)-C(18)-C(19)	121(1)	
C(18)-C(23)-C(22)	120(1)	
C(31)-C(26)-C(27)	122(1)	
C(26)-C(31)-C(30)	120(1)	
C(2)-C(3)-C(4)	116(1)	
C(5)-C(6)-C(7)	118(1)	
C(10)-C(11)-C(12)	120(1)	118(1)
C(13)-C(14)-C(15)	119(1)	
C(18)-C(19)-C(20)	117(1)	
C(21)-C(22)-C(23)	119(1)	
C(26)-C(27)-C(28)	118(1)	
C(29)-C(30)-C(31)	118(1)	
C(3)-C(4)-C(5)	122(1)	
C(4)-C(5)-C(6)	121(1)	
C(11)-C(12)-C(13)	121(1)	
C(12)-C(13)-C(14)	121(1)	121(1)
C(19)-C(20)-C(21)	122(1)	
C(20)-C(21)-C(22)	121(1)	
C(27)-C(28)-C(29)	121(1)	
C(28)-C(29)-C(30)	121(1)	
Os-N(9)-C(33)	122.2(9)	121.8(5)
Os-N(9)-C(37)	121.5(8)	
C(33)-N(9)-C(38)	116(1)	
N(9)-C(33)-C(34)	124(1)	123(1)
N(9)-C(37)-C(36)	123(1)	
C(33)-C(34)-C(35)	120(1)	
C(34)-C(35)-C(36)	118(1)	119(1)
C(35)-C(36)-C(37)	119(1)	
Os-C(38)-O(1)	177(1)	

[a] Some nonbonded distances of interest are also given. Figures in parenthesis for the averaged values are the root-mean-square standard deviations of the least significant figure

TABLE VIII

Least-Squares Planes

A. Deviations (a) from planes

	Plane 1	Plane 2	Plane 3	Plane 4	Plane 5	Plane 6	Plane 7
Os	-0.154	-0.099	-0.083	-0.056	0.017	-0.070	
N(1)	-0.082	-0.102	0.044	-0.147	-0.001	0.088	
N(2)	-0.006	0.003	0.025	-0.086	0.248	0.181	
N(3)	0.053	0.074	-0.026	-0.111	0.511	0.297	
N(4)	-0.075	-0.003	-0.090	-0.018	0.251	0.039	
N(5)	-0.157	-0.029	-0.143	0.102	0.102	-0.158	
N(6)	-0.003	0.014	0.180	-0.013	-0.013	-0.117	
N(7)	0.067	0.156	0.288	0.424	-0.053	-0.009	
N(8)	-0.041	-0.004	0.113	0.096	-0.028	0.011	
C(1)	-0.049	-0.068	0.014	-0.176	0.148	0.160	
C(2)	-0.029	-0.075	-0.011	-0.287	0.261	0.258	
C(3)	-0.042	-0.121	-0.016	-0.390	0.240	0.295	
C(4)	0.030	-0.069	0.000	-0.437	0.418	0.437	
C(5)	0.117	0.030	0.028	-0.379	0.615	0.546	
C(6)	0.097	0.043	0.001	-0.309	0.603	0.478	
C(7)	0.020	-0.013	-0.022	-0.267	0.420	0.329	
C(8)	0.014	0.016	-0.018	-0.157	0.389	0.258	
C(9)	0.025	0.078	-0.047	-0.034	0.462	0.209	
C(10)	0.070	0.144	-0.054	0.018	0.600	0.255	
C(11)	0.159	0.228	-0.031	0.021	0.813	0.397	
C(12)	0.111	0.205	-0.115	0.009	0.830	0.333	
C(13)	-0.051	0.075	-0.250	-0.031	0.612	0.102	
C(14)	-0.118	0.013	-0.253	-0.011	0.422	-0.021	
C(15)	-0.062	0.044	-0.159	0.010	0.412	0.052	
C(16)	-0.102	0.001	-0.130	0.035	0.244	-0.031	
C(17)	-0.124	0.002	-0.048	0.196	0.018	-0.166	
C(18)	-0.037	0.118	0.086	0.416	0.014	-0.157	
C(19)	-0.066	0.122	0.049	0.478	-0.009	-0.236	
C(20)	0.039	0.246	0.208	0.699	-0.012	-0.203	
C(21)	0.157	0.353	0.386	0.845	-0.004	-0.106	
C(22)	0.187	0.351	0.423	0.787	0.021	-0.026	
C(23)	0.062	0.205	0.244	0.542	0.001	-0.080	
C(24)	0.021	0.128	0.195	0.387	-0.016	-0.055	
C(25)	0.005	0.060	0.216	0.256	-0.093	-0.012	
C(26)	0.014	0.049	0.279	0.259	-0.178	-0.003	
C(27)	0.083	0.122	0.411	0.412	-0.232	0.011	
C(28)	0.046	0.059	0.411	0.337	-0.333	-0.008	
C(29)	-0.010	-0.027	0.328	0.159	-0.329	0.008	
C(30)	-0.079	-0.102	0.194	0.003	-0.275	-0.007	
C(31)	-0.052	-0.049	0.184	0.069	-0.183	0.003	
C(32)	-0.099	-0.094	0.070	-0.041	-0.106	-0.003	
N(9)							0.020
C(33)							-0.013
C(34)							-0.004
C(35)							0.013
C(36)							-0.005
C(37)							-0.011

B. Angles (deg) between Least-Squares Planes

	Plane 2	Plane 3	Plane 4	Plane 5	Plane 6	Plane 7
Plane 1	1.4	2.7	5.2	5.1	3.1	98.6
Plane 2		3.0	4.1	5.5	4.2	98.0
Plane 3			4.1	7.9	5.3	96.1
Plane 4				9.4	8.3	94.1
Plane 5					3.7	103.4
Plane 6						101.4

C. Equations of Planes[a]

Plane 1 Marcocycle; N(1)N(8), C(1)-C(32)
$2.721x + 12.314y + 8.382z = 3.126$

Plane 2 Isoindole nitrogen atoms; N(2), N(4), N(6), N(8)
$2.509x + 12.216y + 8.726z = 3.053$

Plane 3 Isoindole Group I; N(2), C(1)-C(8)
$3.051x + 11.869y + 8.660z = 3.113$

Plane 4 Isoindole Group 2; N(4), C(9)-C(16)
$2.438x + 11.516y + 9.693z = 3.029$

Plane 5 Isoindole Group 3; N(6), C(17)-C(24)
$2.125x + 13.099y + 7.731z = 2.833$

Plane 6 Isoindole Group 4; N(8), C(25)-C(32)
$2.836x + 12.756y + 7.589z = 3.024$

Plane 7 Pyridine Ring; N(9), C(33)-C(37)
$2.361x - 11.469y + 9.817z = 0.148$

[a]All planes are unweighted. x,y,z are in monoclinic fractional coordinates.

TABLE IX

Intermolecular contacts (A) $\leq$3.5A[a]

C(16)-O(1)^{I}	3.08	N(3)-C(35)^{II}	3.32
O(1)-O(1)^{I}	3.18	N(5)-C(4)^{III}	3.40
N(4)-O(1)^{I}	3.25	C(23)-C(35)^{IV}	3.41
C(15)-O(1)^{I}	3.26	C(24)-C(36)^{IV}	3.42
C(38)-O(1)^{I}	3.31		

[a]Roman numeral superscripts denote the following equivalent positions relative to the reference molecule at x,y,z:

I - x, - y, - z
II - ½ +x, ½ - y, - ½ + z
III - ½ + x, ½ - y, ½ - z
IV 1 - x, - y, - z

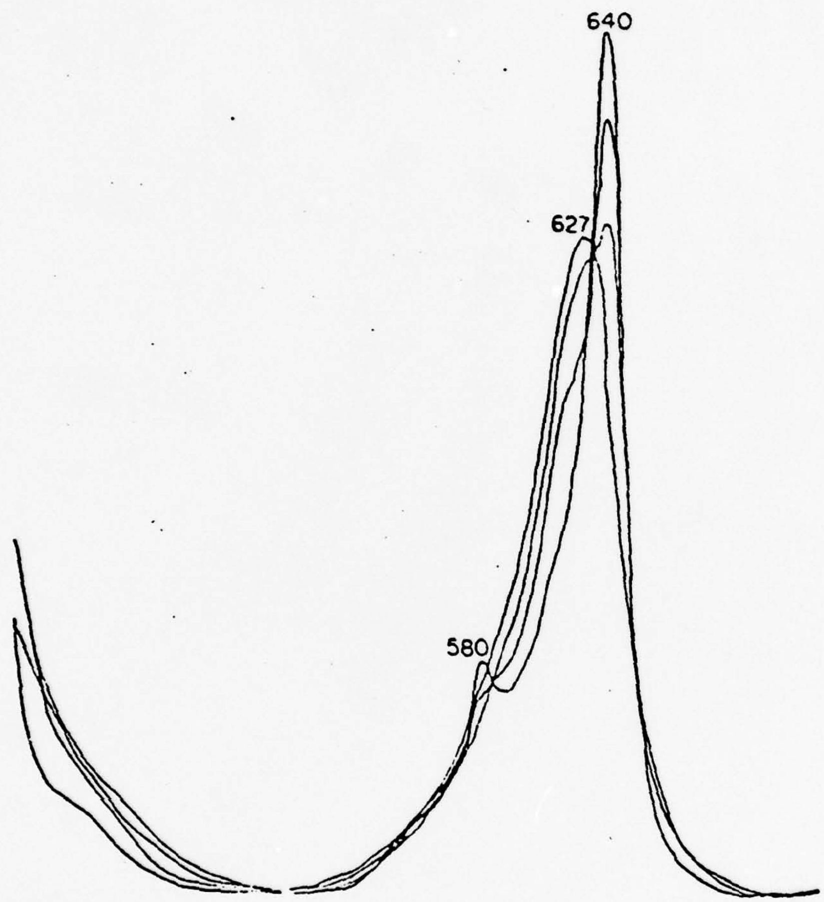

Fig. 1 Tsutsui

Figure 1. Coordination of carbon monoxide by PcRu(II) in THF. The peak at 627 nm is characteristic for PcRu(II) and the peaks at 640 and 580 nm are due to PcRu(CO)(THF). Upon coordination of carbon monoxide, the peak at 627 disappears, while new peaks (at 540 and 580 nm) appear.

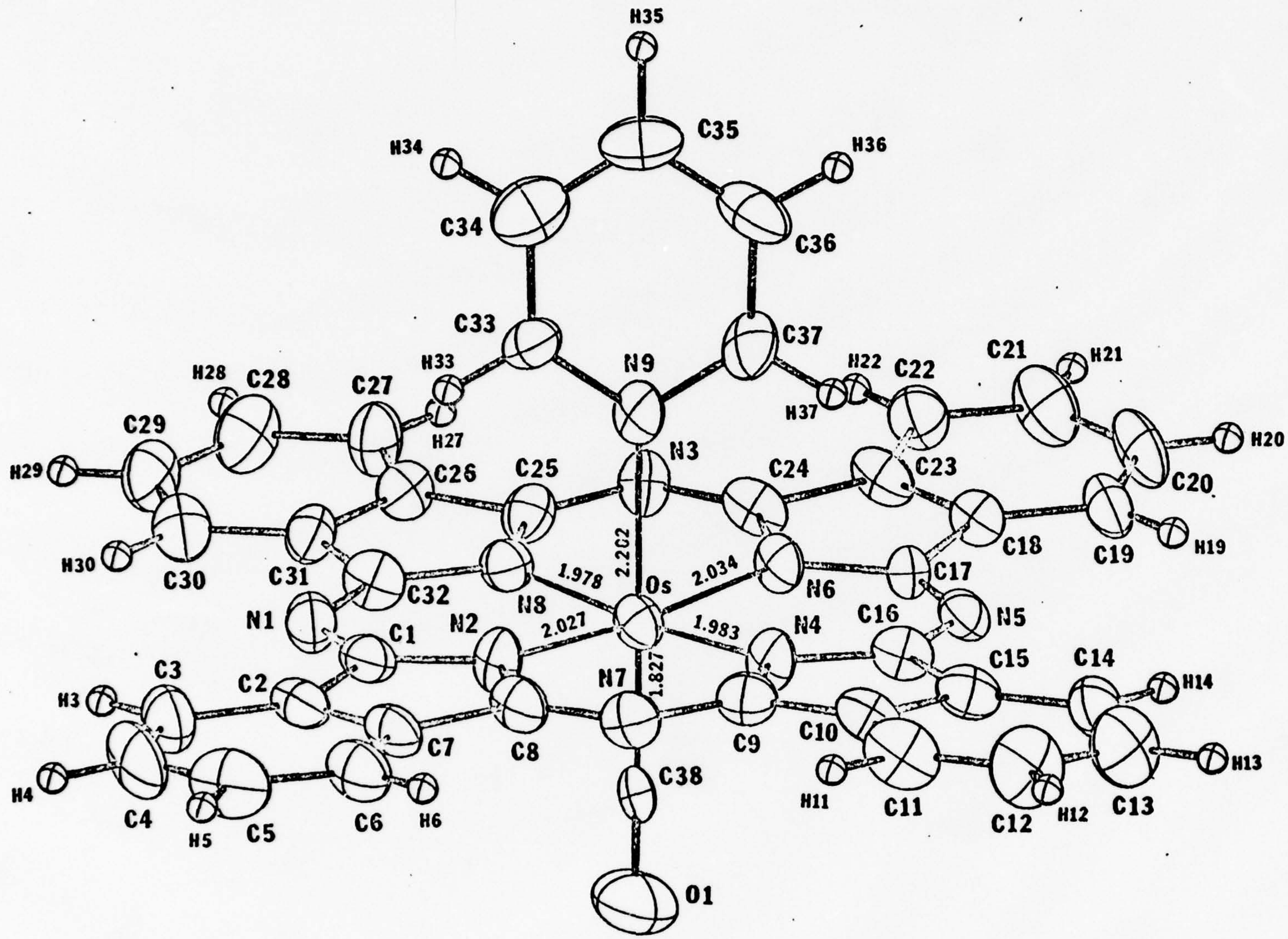

Figure 2. ORTEP[13] drawing of the structure of PcOs(CO)(Py). Numbering scheme is shown. The bond lengths involving the osmium ion are also shown. The thermal ellipsoids are drawn for 50% probability, except those of

Fig. 2 Tsutsui

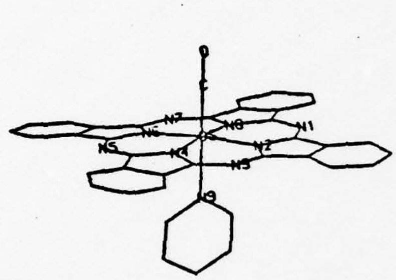

Fig. 3 Tsutsui

Figure 3. Stereoview of PcOs(II)(CO)(Py)

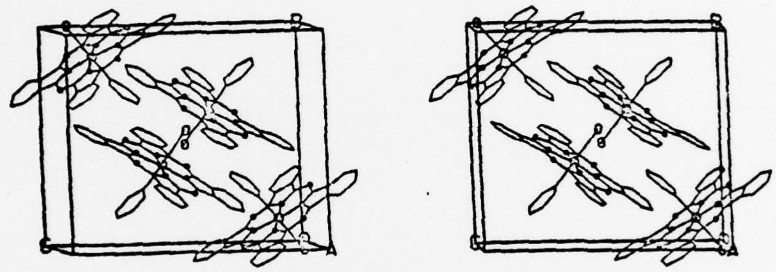

Fig. 4 Tsutsui

Figure 4. Stereoview of the packing in the unit cell. The osmium atoms are indicated by the larger circles, the nitrogen atom. Oxygen atoms of the carbonyl groups are labeled with the letter O.

SYNTHESIS AND STRUCTURE OF A NEW CLASS OF METALLOPHTHALOCYANINES:

Carbonylphthalocyanato(pyridine or THF)-ruthenium(II) and carbonylphthalocyanato (pyridine or THF)osmium(II)

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SUPPLEMENTARY MATERIAL

TABLE III

Calculated Postional Parameters for Hydrogen Atoms

Atom	X	Y	Z
H(3)	0.512	0.197	-0.094
H(4)	0.444	0.292	-0.205
H(5)	0.274	0.368	-0.232
H(6)	0.142	0.342	-0.159
H(11)	-0.145	0.351	-0.064
H(12)	-0.324	0.369	-0.040
H(13)	-0.378	0.292	0.055
H(14)	-0.244	0.198	0.140
H(19)	-0.054	0.028	0.333
H(20)	0.015	-0.053	0.450
H(21)	0.203	-0.108	0.492
H(22)	0.331	-0.083	0.419
H(27)	0.618	-0.090	0.319
H(28)	0.796	-0.117	0.293
H(29)	0.832	-0.055	0.181
H(30)	0.691	0.028	0.093
H(33)	0.449	0.227	0.168
H(34)	0.544	0.327	0.264
H(35)	0.451	0.379	0.356
H(36)	0.256	0.334	0.342
H(37)	0.167	0.233	0.244

TABLE IV

ROOT-MEAN-SQUARE AMPLITUDES OF THERMAL VIBRATION IN ANGSTROMS.

ATOM	MIN.	INT'MED.	MAX.
O5	0.143	0.177	0.190
N1	0.167	0.197	0.203
N2	0.113	0.178	0.219
N3	0.141	0.192	0.217
N4	0.110	0.180	0.239
N5	0.147	0.179	0.197
N6	0.133	0.191	0.200
N7	0.155	0.173	0.218
N8	0.107	0.192	0.241
C1	0.151	0.173	0.247
C2	0.145	0.150	0.248
C3	0.167	0.215	0.251
C4	0.136	0.243	0.299
C5	0.136	0.225	0.330
C6	0.165	0.199	0.254
C7	0.136	0.181	0.230
C8	0.144	0.179	0.229
C9	0.170	0.188	0.205
C10	0.161	0.182	0.232
C11	0.157	0.222	0.271
C12	0.144	0.261	0.289
C13	0.170	0.252	0.272
C14	0.168	0.183	0.232
C15	0.149	0.190	0.225
C16	0.120	0.176	0.211
C17	0.142	0.173	0.201
C18	0.135	0.177	0.195
C19	0.148	0.196	0.234
C20	0.125	0.205	0.292
C21	0.136	0.227	0.287
C22	0.187	0.196	0.232
C23	0.127	0.177	0.242
C24	0.131	0.176	0.235
C25	0.158	0.193	0.202
C26	0.165	0.191	0.201
C27	0.171	0.213	0.248
C28	0.160	0.224	0.268
C29	0.159	0.221	0.256
C30	0.182	0.195	0.236
C31	0.162	0.184	0.208
C32	0.141	0.200	0.215
N9	0.159	0.173	0.212
C33	0.158	0.203	0.213
C34	0.212	0.222	0.274
C35	0.164	0.213	0.283
C36	0.157	0.211	0.293
C37	0.145	0.214	0.239
O1	0.190	0.286	0.326

TABLE V

Values of 10|Fo| and 10 |Fc|

For PcOs(CO)(Py)

(PYRIDINE)(CARBONYL)PHTHALOCYANINATOOSMIUM(II) 10 FO AND 10 FC

H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC
0	0	2	329	347	0	3	17	549	523	0	7	5	467	506	0	12	9	357	415	1	1	-3	2345	2221
0	0	4	1302	1299	0	4	0	558	437	0	7	6	1085	1204	0	12	11	840	890	1	1	-2	2201	2109
0	0	6	460	447	0	4	1	283	308	0	7	7	360	373	0	12	12	465	492	1	1	-1	1872	1794
0	0	8	1881	1850	0	4	2	607	620	0	7	8	495	536	0	13	1	875	872	1	1	0	587	643
0	0	10	1160	1140	0	4	4	2448	2629	0	7	10	1258	1317	0	13	3	708	715	1	1	1	2298	2413
0	0	12	1451	1442	0	4	5	586	617	0	7	12	862	902	0	13	5	785	784	1	1	2	1517	1535
0	0	14	914	872	0	4	6	400	452	0	7	14	1092	1139	0	13	6	368	410	1	1	3	670	681
0	0	16	598	566	0	4	7	685	723	0	7	16	615	620	0	13	7	386	372	1	1	4	642	612
0	0	18	529	541	0	4	8	1696	1747	0	8	0	1529	1533	0	13	9	760	788	1	1	5	1391	1385
0	1	2	2293	2362	0	4	10	758	767	0	8	1	886	865	0	14	0	1104	1096	1	1	6	1448	1466
0	1	3	1545	1569	0	4	11	346	348	0	8	3	658	704	0	14	2	378	326	1	1	8	347	369
0	1	4	956	947	0	4	12	1231	1241	0	8	4	1264	1339	0	14	4	835	848	1	1	9	831	897
0	1	5	1290	1319	0	4	13	401	409	0	8	5	736	801	0	14	8	820	817	1	1	10	1107	1101
0	1	6	1297	1322	0	4	14	785	822	0	8	7	871	930	0	14	10	513	545	1	1	12	627	612
0	1	7	953	971	0	4	15	582	592	0	8	8	1076	1131	0	15	1	528	508	1	1	13	847	831
0	1	8	896	884	0	4	16	1043	1070	0	8	11	846	862	0	15	2	882	886	1	1	14	655	628
0	1	9	1678	1670	0	5	1	1291	1295	0	8	12	892	929	0	15	5	388	371	1	1	16	782	768
0	1	10	1357	1339	0	5	3	1573	1652	0	8	14	487	491	0	15	6	790	785	1	1	17	472	492
0	1	11	433	492	0	5	4	618	629	0	8	15	594	603	0	16	0	424	403	1	2	-18	636	612
0	1	12	543	540	0	5	5	1268	1387	0	9	1	1549	1535	0	16	1	709	687	1	2	-15	825	803
0	1	13	1286	1295	0	5	6	952	1023	0	9	3	857	906	0	16	3	760	755	1	2	-14	617	600
0	1	14	761	756	0	5	7	1057	1130	0	9	5	1644	1754	0	17	1	534	555	1	2	-12	1599	1623
0	1	16	537	540	0	5	9	1205	1268	0	9	7	470	496	1	0	-15	1430	1432	1	2	-10	636	625
0	1	17	721	713	0	5	10	509	512	0	9	9	1228	1301	1	0	-13	559	595	1	2	-8	2798	2719
0	2	1	2456	2512	0	5	11	423	431	0	9	13	1177	1231	1	0	-11	2034	2011	1	2	-7	688	656
0	2	2	1895	1979	0	5	12	487	503	0	10	0	1204	1199	1	0	-9	1602	1596	1	2	-5	422	400
0	2	3	2693	2819	0	5	13	1223	1223	0	10	1	992	965	1	0	-7	1680	1697	1	2	-4	2078	2024
0	2	4	702	717	0	5	14	370	396	0	10	3	663	679	1	0	-5	466	479	1	2	-3	2070	1934
0	2	5	1628	1677	0	5	17	930	956	0	10	4	1272	1329	1	0	-3	2196	2168	1	2	0	3424	3521
0	2	7	1627	1660	0	6	0	889	884	0	10	5	566	587	1	0	1	2428	2390	1	2	1	1830	1885
0	2	8	415	439	0	6	1	1215	1219	0	10	6	341	334	1	0	3	690	724	1	2	2	1013	1041
0	2	9	905	916	0	6	2	484	489	0	10	7	767	820	1	0	5	746	728	1	2	3	1372	1398
0	2	11	1924	1929	0	6	3	1651	1740	0	10	8	769	809	1	0	7	1749	1750	1	2	4	951	1024
0	2	15	1080	1067	0	6	4	807	855	0	10	11	607	661	1	0	11	1861	1832	1	2	6	1082	1100
0	3	1	1517	1535	0	6	5	326	372	0	10	12	689	724	1	0	15	1045	996	1	2	8	1606	1621
0	3	2	3474	3617	0	6	6	979	1014	0	10	14	415	411	1	1	-18	384	354	1	2	10	914	909
0	3	3	1157	1232	0	6	7	1365	1418	0	11	2	1455	1461	1	1	-17	616	621	1	2	12	1246	1230
0	3	4	587	626	0	6	8	668	708	0	11	4	361	365	1	1	-14	1087	1084	1	2	14	890	878
0	3	5	1301	1336	0	6	9	578	524	0	11	5	410	404	1	1	-13	1003	1016	1	2	16	633	634
0	3	6	1717	1816	0	6	10	371	372	0	11	6	1256	1332	1	1	-12	658	636	1	2	17	481	487
0	3	8	753	769	0	6	11	1185	1247	0	11	10	742	750	1	1	-11	477	488	1	3	-18	886	887
0	3	9	999	1074	0	6	12	570	612	0	11	12	618	659	1	1	-10	1892	1853	1	3	-17	837	847
0	3	10	1447	1408	0	6	14	590	636	0	12	1	756	735	1	1	-9	1352	1339	1	3	-16	304	292
0	3	12	467	408	0	6	15	1128	1172	0	12	3	1222	1205	1	1	-8	712	673	1	3	-14	698	654
0	3	13	839	844	0	7	1	288	275	0	12	4	923	940	1	1	-7	1169	1159	1	3	-13	1129	1115
0	3	14	742	725	0	7	2	2099	2178	0	12	5	481	520	1	1	-6	1362	1370	1	3	-12	344	337
0	3	16	892	806	0	7	4	440	412	0	12	7	694	723	1	1	-5	829	815	1	3	-11	831	812

(PYRIDINE)(CARBONYL)PHTALOCYANATOOSMIUM(II) 10 FO AND 10 FC

H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC
1	3	-10	904	877	1	4	15	1100	1090	1	6	8	1549	1599	1	8	9	340	322	1	11	4	371	388
1	3	-9	1640	1578	1	5	-16	697	686	1	6	10	787	834	1	8	10	365	363	1	11	5	1293	1371
1	3	-7	936	906	1	5	-14	637	607	1	6	11	566	583	1	8	11	616	603	1	11	7	566	595
1	3	-6	1291	1229	1	5	-13	425	414	1	6	12	529	502	1	8	12	477	499	1	11	9	613	864
1	3	-5	2323	2230	1	5	-10	1936	1898	1	6	14	775	775	1	8	14	502	533	1	11	13	821	899
1	3	-4	1371	1292	1	5	-9	700	682	1	6	16	562	564	1	8	15	881	903	1	12	-12	828	814
1	3	-3	320	336	1	5	-8	545	530	1	7	-14	306	377	1	9	-14	1219	1204	1	12	11	599	595
1	3	-2	1360	1308	1	5	-7	976	941	1	7	-13	1372	1366	1	9	-12	712	719	1	12	-10	437	428
1	3	-1	1577	1532	1	5	-6	2329	2308	1	7	-11	496	471	1	9	-10	1145	1137	1	12	-9	415	419
1	3	0	1026	989	1	5	-5	422	420	1	7	-9	1531	1517	1	9	-6	1368	1374	1	12	-8	956	963
1	3	1	2311	2381	1	5	-3	727	714	1	7	-7	1072	1078	1	9	-2	1603	1622	1	12	-7	516	507
1	3	2	3427	3603	1	5	-2	2084	2052	1	7	-6	415	433	1	9	2	1300	1348	1	12	-6	341	298
1	3	3	2418	2524	1	5	2	2609	2670	1	7	-5	1252	1246	1	9	4	903	917	1	12	-5	557	539
1	3	4	414	393	1	5	3	521	503	1	7	-4	301	250	1	9	6	1715	1815	1	12	-4	1196	1154
1	3	5	2048	2118	1	5	4	317	307	1	7	-3	1181	1172	1	9	8	663	709	1	12	-3	435	444
1	3	6	361	421	1	5	5	1241	1294	1	7	-2	337	327	1	9	10	861	894	1	12	-1	358	361
1	3	7	379	413	1	5	6	1802	1905	1	7	-1	1355	1344	1	9	12	694	714	1	12	0	1011	1006
1	3	8	538	563	1	5	7	487	498	1	7	1	1870	1924	1	9	14	773	781	1	12	1	440	439
1	3	9	1351	1379	1	5	8	1075	1128	1	7	3	620	647	1	10	-15	686	683	1	12	2	544	484
1	3	10	536	544	1	5	9	549	581	1	7	4	1445	1457	1	10	-12	1143	1157	1	12	3	752	757
1	3	12	570	584	1	5	10	1251	1307	1	7	5	1655	1762	1	10	-11	1088	1097	1	12	4	1126	1171
1	3	13	1322	1312	1	5	12	816	836	1	7	6	747	772	1	10	-9	408	421	1	12	5	504	525
1	3	14	440	436	1	5	13	485	460	1	7	7	618	655	1	10	-8	685	677	1	12	6	711	733
1	3	17	938	917	1	5	14	650	655	1	7	9	1582	1655	1	10	-7	941	893	1	12	7	677	720
1	4	-15	790	766	1	5	16	772	753	1	7	12	367	400	1	10	-5	895	879	1	12	8	788	843
1	4	-12	504	484	1	6	-16	729	718	1	7	13	972	965	1	10	-4	1320	1314	1	12	10	511	559
1	4	-11	1736	1695	1	6	-15	779	768	1	8	-16	495	478	1	10	-3	724	722	1	12	11	484	530
1	4	-10	324	274	1	6	-14	537	528	1	8	-15	995	986	1	10	-1	704	709	1	12	12	436	446
1	4	-9	995	966	1	6	-13	381	404	1	8	-14	437	441	1	10	0	1068	1085	1	13	-10	1144	1149
1	4	-8	624	611	1	6	-12	992	939	1	8	-13	625	633	1	10	1	853	869	1	13	-6	1346	1268
1	4	-7	1776	1747	1	6	-11	433	418	1	8	-12	890	895	1	10	3	1036	1067	1	13	-5	352	352
1	4	-6	822	792	1	6	-10	498	493	1	8	-11	900	883	1	10	4	981	1020	1	13	-2	1258	1231
1	4	-5	1559	1547	1	6	-9	395	388	1	8	-9	463	473	1	10	6	360	410	1	13	0	340	347
1	4	-4	712	686	1	6	-8	1706	1693	1	8	-8	521	918	1	10	7	757	789	1	13	1	407	373
1	4	-3	1410	1388	1	6	-7	935	904	1	8	-7	1012	1037	1	10	8	357	366	1	13	2	1435	1436
1	4	-2	1673	1565	1	6	-5	677	678	1	8	-5	1200	1209	1	10	11	753	772	1	13	4	338	368
1	4	-1	1423	1436	1	6	-4	1535	1578	1	8	-4	1081	1089	1	10	12	689	753	1	13	5	467	406
1	4	0	1059	1026	1	6	-3	811	826	1	8	-3	810	822	1	10	14	439	444	1	13	6	1017	1055
1	4	1	[illegible]	977	1	6	-1	719	717	1	8	-2	426	361	1	11	-13	1012	1018	1	13	8	479	513
1	4	2	[illegible]	2400	1	6	0	1741	1751	1	8	-1	1456	1403	1	11	-11	633	648	1	13	10	604	630
1	4	3	[illegible]	5281	1	6	1	346	368	1	8	0	1367	1360	1	11	-9	1108	1163	1	14	-9	475	472
1	4	4	[illegible]	1574	1	6	2	757	750	1	8	1	876	922	1	11	-7	671	670	1	14	-7	712	662
1	4	5	771	[illegible]	1	6	3	572	594	1	8	3	280	347	1	11	-5	1057	995	1	14	-5	525	498
1	4	7	1381	1110	1	6	4	3005	3181	1	8	4	1104	1146	1	11	-3	1137	1112	1	14	-4	434	413
1	4	9	471	470	1	6	5	1481	1502	1	8	5	519	559	1	11	-1	636	627	1	14	-3	790	801
1	4	11	1170	1165	1	6	6	791	843	1	8	7	1560	1556	1	11	1	1174	1128	1	14	-1	1001	1005
1	4	12	400	416	1	6	7	1185	1243	1	8	8	773	867	1	11	3	686	733	1	14	0	415	420

(PYRIDINE)(CARBONYL)PHTHALOCYANATOOSMIUM(II) 10 FO AND 10 FC PAGE 3

H	K	L	FOBS	FCALC
1	14	1	372	411
1	14	3	1072	1091
1	14	4	768	767
1	14	7	984	1043
1	15	-7	471	445
1	15	-6	432	419
1	15	-5	668	646
1	15	-3	738	700
1	15	-2	530	515
1	15	-1	753	773
1	15	1	736	749
1	15	2	409	498
1	15	3	538	611
1	15	4	373	358
1	15	5	970	1020
1	16	-4	836	806
1	16	0	983	1001
1	16	2	476	503
1	16	4	889	902
2	0	-18	382	367
2	0	-16	1458	1455
2	0	-14	450	461
2	0	-12	1238	1271
2	0	-10	380	391
2	0	-8	2437	2420
2	0	-4	1877	1883
2	0	-2	608	595
2	0	0	3578	3601
2	0	2	1167	1166
2	0	4	1590	1677
2	0	6	1253	1235
2	0	8	1178	1148
2	0	10	1225	1201
2	0	12	994	955
2	0	14	1057	1000
2	1	-18	479	456
2	1	-17	605	606
2	1	-16	670	682
2	1	-15	608	620
2	1	-14	[illegible]	378
2	1	-13	1053	1061
2	1	-11	700	610
2	1	-10	1042	1059
2	1	-9	1063	1071
2	1	-7	1350	1321
2	1	-6	1297	1290
2	1	-5	1631	1579

H	K	L	FOBS	FCALC
2	1	-3	1652	1595
2	1	-2	1652	1605
2	1	-1	263	245
2	1	0	912	938
2	1	1	1984	2043
2	1	2	879	897
2	1	4	710	747
2	1	5	1560	1638
2	1	6	1074	1081
2	1	7	504	492
2	1	8	1273	1244
2	1	9	1090	1084
2	1	10	1042	1017
2	1	12	673	664
2	1	13	1134	1099
2	1	14	401	417
2	1	16	632	608
2	2	-17	381	357
2	2	-15	1134	1138
2	2	-14	454	464
2	2	-13	619	623
2	2	-12	338	341
2	2	-11	1453	1439
2	2	-10	603	610
2	2	-9	1497	1492
2	2	-8	449	426
2	2	-7	2049	1998
2	2	-6	323	338
2	2	-5	2089	2050
2	2	-4	520	501
2	2	-3	1293	1250
2	2	-1	1606	1605
2	2	1	983	1032
2	2	3	1971	2078
2	2	5	371	384
2	2	7	1647	1652
2	2	8	311	323
2	2	10	705	672
2	2	11	1533	1588
2	2	15	1004	753
2	3	-18	745	741
2	3	-17	638	634
2	3	-14	947	935
2	3	-13	623	597
2	3	-11	463	466
2	3	-10	2011	[illegible]
2	3	-9	1112	1100

H	K	L	FOBS	FCALC
2	3	-8	370	358
2	3	-7	1217	1161
2	3	-6	2321	2251
2	3	-5	544	548
2	3	-4	315	294
2	3	-3	557	561
2	3	-2	1269	1240
2	3	-1	241	238
2	3	0	813	810
2	3	1	332	369
2	3	2	2401	2502
2	3	3	623	622
2	3	4	1274	1350
2	3	5	915	993
2	3	6	1149	1214
2	3	7	400	371
2	3	8	712	734
2	3	9	906	892
2	3	10	767	763
2	3	12	897	884
2	3	13	623	601
2	3	14	445	409
2	3	16	660	644
2	4	-18	529	530
2	4	-16	611	600
2	4	-14	380	363
2	4	-13	359	374
2	4	-12	1958	1916
2	4	-10	376	362
2	4	-8	2456	2407
2	4	-7	1229	1192
2	4	-6	296	272
2	4	-5	513	512
2	4	-4	1855	1838
2	4	-3	377	352
2	4	-2	319	323
2	4	0	1897	1922
2	4	1	772	761
2	4	2	995	1033
2	4	3	929	948
2	4	4	1790	1850
2	4	6	598	637
2	4	8	1389	1430
2	4	10	969	962
2	4	11	527	524
2	4	12	516	513
2	4	14	759	745

H	K	L	FOBS	FCALC
2	4	15	361	326
2	4	16	471	475
2	5	-17	1023	1031
2	5	-13	983	963
2	5	-11	1105	1065
2	5	-10	708	706
2	5	-9	1675	1653
2	5	-7	1606	1550
2	5	-6	999	986
2	5	-5	1934	1870
2	5	-4	486	464
2	5	-3	1354	1333
2	5	-2	517	499
2	5	-1	839	836
2	5	0	444	452
2	5	1	1745	1780
2	5	2	400	427
2	5	3	1109	1112
2	5	4	979	956
2	5	5	1514	1571
2	5	6	275	302
2	5	9	1626	1639
2	5	10	394	371
2	5	13	822	812
2	6	-16	525	504
2	6	-15	807	840
2	6	-13	441	423
2	6	-12	494	487
2	6	-11	1197	1210
2	6	-9	1259	1252
2	6	-8	1058	1028
2	6	-7	1119	1117
2	6	-6	447	403
2	6	-5	1860	1816
2	6	-4	1268	1232
2	6	-3	1219	1163
2	6	-1	1752	1723
2	6	0	1082	1072
2	6	1	449	406
2	6	2	605	606
2	6	3	1761	1874
2	6	4	1020	1041
2	6	5	489	527
2	6	6	1444	1459
2	6	7	1346	1402
2	6	8	584	604
2	6	10	390	405

H	K	L	FOBS	FCALC
2	6	11	995	1016
2	6	14	368	392
2	6	15	944	972
2	7	-14	1147	1133
2	7	-12	425	441
2	7	-10	1611	1627
2	7	-6	2076	2023
2	7	-3	491	479
2	7	-2	2566	2403
2	7	0	735	762
2	7	1	492	484
2	7	2	1558	1647
2	7	3	502	451
2	7	4	385	465
2	7	5	1459	1444
2	7	6	2019	2059
2	7	7	468	470
2	7	8	1264	1263
2	7	10	838	862
2	7	12	750	774
2	7	14	536	542
2	8	-16	925	892
2	8	-15	653	644
2	8	-12	434	391
2	8	-11	713	716
2	8	-9	628	619
2	8	-8	1640	1628
2	8	-7	819	803
2	8	-5	834	812
2	8	-4	1817	1767
2	8	-3	873	905
2	8	-2	461	483
2	8	-1	941	936
2	8	0	961	955
2	8	1	576	572
2	8	2	507	519
2	8	3	1037	1086
2	8	4	1457	1518
2	8	5	1216	1219
2	8	6	1590	1608
2	8	7	1423	1471
2	8	8	1093	1125
2	8	10	538	546
2	8	11	721	732
2	8	12	614	605
2	8	14	643	639
2	9	15	512	515

(PYRIDINE)(CARBONYL)PHTALOCYANATOOSMIUM(II) 10 FO AND 10 FC

H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC
2	9	-13	1051	1027	2	12	-12	735	765	2	15	4	518	547	3	1	12	964	903	3	3	6	578	617
2	9	-11	600	587	2	12	-11	951	941	2	15	5	435	457	3	1	13	839	794	3	3	8	556	562
2	9	-9	1026	1012	2	12	-9	753	764	2	15	6	692	684	3	1	14	399	378	3	3	9	1544	1556
2	9	-7	1431	1378	2	12	-8	462	400	2	16	-5	727	675	3	1	16	566	525	3	3	10	609	619
2	9	-5	1157	1105	2	12	-7	717	686	2	16	-3	579	571	3	2	-18	390	409	3	3	11	708	692
2	9	-3	1974	1891	2	12	-5	1007	969	2	16	-1	810	831	3	2	-16	1402	1436	3	3	12	475	461
2	9	-1	1306	1296	2	12	-4	983	959	2	16	1	416	383	3	2	-12	1352	1362	3	3	13	1183	1172
2	9	1	1435	1456	2	12	-3	756	748	2	16	3	889	908	3	2	-8	2104	2102	3	4	-17	529	537
2	9	3	399	435	2	12	-1	1187	1115	3	0	-15	1566	1578	3	2	-7	1115	1075	3	4	-16	454	481
2	9	5	2145	2216	2	12	0	548	633	3	0	-13	548	588	3	2	-6	796	769	3	4	-15	690	883
2	9	7	533	533	2	12	1	362	403	3	0	-11	1276	1294	3	2	-4	2680	2608	3	4	-13	1266	1246
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2	10	-11	697	696	2	12	11	679	683	3	0	7	1597	1588	3	2	3	421	458	3	4	-4	790	780
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2	10	-3	751	676	2	13	-5	832	794	3	1	-17	673	723	3	2	9	453	455	3	4	1	575	582
2	10	-1	929	898	2	13	-3	1054	1014	3	1	-15	649	677	3	2	10	1283	1249	3	4	2	537	571
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2	11	8	770	807	2	15	-2	712	705	3	1	8	753	774	3	3	3	494	507	3	5	-2	1826	1786
2	11	10	766	761	2	15	1	448	410	3	1	9	910	911	3	3	4	829	916	3	5	-1	540	520
2	11	12	769	774	2	15	2	653	645	3	1	10	551	525	3	3	5	1190	1261	3	5	0	1306	1301

H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC
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3	5	2	1114	1198	3	7	-3	1940	1886	3	10	-12	522	505	3	12	7	759	798	4	1	-17	841	889
3	5	3	443	440	3	7	-2	392	377	3	10	-11	556	549	3	12	8	980	1005	4	1	-15	898	951
3	5	4	1064	1104	3	7	-1	578	570	3	10	-9	399	372	3	12	10	645	635	4	1	-14	1143	1154
3	5	5	609	654	3	7	1	1662	1663	3	10	-8	956	933	3	13	-10	1047	994	4	1	-13	973	985
3	5	6	1132	1200	3	7	2	618	621	3	10	-7	413	417	3	13	-7	373	386	4	1	-11	1145	1167
3	5	7	307	293	3	7	5	2118	2164	3	10	-5	1291	1239	3	13	-6	977	939	4	1	-10	1128	1111
3	5	8	1381	1409	3	7	6	326	329	3	10	-4	1392	1326	3	13	-4	373	355	4	1	-9	798	788
3	5	9	678	676	3	7	9	1551	1641	3	10	-3	691	702	3	13	-3	384	341	4	1	-8	956	922
3	5	10	946	977	3	7	10	401	431	3	10	-2	596	593	3	13	-2	1231	1195	4	1	-7	746	778
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3	6	-4	2164	2083	3	8	-2	945	874	3	10	11	723	704	3	14	-1	1003	1009	4	1	7	523	521
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H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC
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(PYRIDINE)(...)(...)PHTHALOCYANATOOSMIUM(II) 10 FO AND 10 FC

H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC
5	0	7	1706	1624	5	2	5	386	347	5	4	7	1445	1447	5	6	10	702	686	5	9	4	1101	1131
5	0	9	634	607	5	2	6	1512	1569	5	4	9	585	546	5	7	-17	738	789	5	9	6	443	413
5	0	11	1063	987	5	2	8	712	692	5	4	11	1014	978	5	7	-15	926	935	5	9	8	966	941
5	1	-18	941	1016	5	2	10	1223	1165	5	4	13	517	498	5	7	-13	794	805	5	10	-15	505	551
5	1	-15	450	486	5	2	14	1006	971	5	5	-18	724	774	5	7	-11	1504	1494	5	10	-13	777	787
5	1	-14	1076	1136	5	3	-17	745	759	5	5	-15	630	668	5	7	-9	660	639	5	10	-12	946	962
5	1	-13	611	627	5	3	-16	370	394	5	5	-14	1228	1266	5	7	-7	1691	1651	5	10	-11	524	552
5	1	-12	362	380	5	3	-15	711	743	5	5	-13	353	365	5	7	-6	421	388	5	10	-9	896	875
5	1	-11	1018	1040	5	3	-14	607	623	5	5	-12	387	355	5	7	-5	454	480	5	10	-8	1070	1035
5	1	-10	1267	1312	5	3	-13	849	850	5	5	-11	885	883	5	7	-4	534	499	5	10	-6	434	430
5	1	-9	595	617	5	3	-12	416	397	5	5	-10	1660	1634	5	7	-3	1543	1533	5	10	-5	965	942
5	1	-8	375	391	5	3	-11	1238	1237	5	5	-8	520	548	5	7	1	1843	1862	5	10	-4	537	523
5	1	-7	1190	1211	5	3	-10	1078	1080	5	5	-7	589	569	5	7	2	386	419	5	10	-3	411	408
5	1	-6	1067	1112	5	3	-9	890	898	5	5	-6	1635	1602	5	7	5	1264	1291	5	10	-2	890	875
5	1	-4	621	653	5	3	-8	861	839	5	5	-4	1333	1275	5	7	7	369	356	5	10	-1	1102	1102
5	1	-3	1116	1096	5	3	-7	1235	1234	5	5	-3	505	500	5	7	8	357	375	5	10	0	726	724
5	1	-2	718	772	5	3	-6	408	431	5	5	-2	1364	1356	5	7	9	877	834	5	10	2	615	621
5	1	-1	615	619	5	3	-3	1973	1942	5	5	-1	430	400	5	7	11	445	423	5	10	3	900	917
5	1	0	1615	1635	5	3	-2	1716	1685	5	5	0	1221	1270	5	8	-16	716	750	5	10	4	404	450
5	1	1	1616	1652	5	3	0	699	716	5	5	1	612	638	5	8	-15	579	579	5	10	6	460	425
5	1	2	1105	1106	5	3	1	1937	2041	5	5	2	1139	1192	5	8	-13	1146	1159	5	10	7	619	609
5	1	4	1458	1409	5	3	2	882	890	5	5	4	1363	1411	5	8	-12	817	820	5	10	8	363	396
5	1	5	1463	1406	5	3	3	337	328	5	5	5	644	637	5	8	-11	564	535	5	10	10	583	604
5	1	6	902	780	5	3	4	719	746	5	5	6	705	613	5	8	-9	1515	1502	5	11	-13	576	582
5	1	7	490	473	5	3	5	1517	1571	5	5	8	960	980	5	8	-8	785	784	5	11	-11	905	919
5	1	8	963	922	5	3	7	352	371	5	5	12	841	799	5	8	-7	446	427	5	11	-9	382	377
5	1	9	832	810	5	3	8	678	689	5	6	-16	833	871	5	8	-6	609	578	5	11	-7	1505	1514
5	1	10	444	439	5	3	9	1038	1081	5	6	-15	602	615	5	8	-5	1461	1416	5	11	-3	1348	1330
5	1	11	517	474	5	3	11	492	476	5	6	-14	356	375	5	8	-4	1300	1266	5	11	1	1012	972
5	1	12	903	851	5	3	12	596	547	5	6	-13	572	568	5	8	-2	426	449	5	11	2	415	411
5	1	13	650	599	5	3	13	715	678	5	6	-12	1410	1435	5	8	-1	1289	1289	5	11	5	866	903
5	2	-16	1132	1172	5	4	-17	547	588	5	6	-11	696	714	5	8	0	725	696	5	11	9	843	811
5	2	-12	1513	1571	5	4	-16	491	520	5	6	-10	649	634	5	8	2	729	743	5	12	-13	492	477
5	2	-11	453	430	5	4	-15	698	722	5	6	-9	775	777	5	8	3	890	932	5	12	-12	831	843
5	2	-10	641	642	5	4	-13	1055	1085	5	6	-8	1542	1502	5	8	4	427	428	5	12	-9	391	376
5	2	-8	1560	1567	5	4	-12	686	678	5	6	-6	712	667	5	8	6	701	709	5	12	-8	999	958
5	2	-7	903	961	5	4	-11	896	899	5	6	-5	799	760	5	8	7	529	487	5	12	-5	450	419
5	2	-6	866	875	5	4	-9	1471	1472	5	6	-4	1576	1507	5	8	10	468	427	5	12	-4	712	670
5	2	-5	644	630	5	4	-8	909	981	5	6	-3	339	331	5	8	11	552	471	5	12	-2	761	753
5	2	-4	1264	1230	5	4	-5	1913	1251	5	6	-2	901	908	5	9	-14	1243	1271	5	12	-1	688	690
5	2	-3	607	570	5	4	-3	697	660	5	6	-1	861	854	5	9	-10	1327	1308	5	12	0	817	823
5	2	-2	1455	1425	5	4	-2	912	872	5	6	0	1219	1259	5	9	-8	711	665	5	12	2	719	741
5	2	-1	574	578	5	4	-1	1846	1947	5	6	2	1216	1279	5	9	-6	1245	1195	5	12	3	765	786
5	2	0	1470	1552	5	4	0	657	686	5	6	3	896	919	5	9	-4	737	698	5	12	4	484	528
5	2	2	1832	1958	5	4	2	496	463	5	6	4	905	892	5	9	-2	1501	1494	5	12	6	805	817
5	2	3	519	533	5	4	3	1856	1911	5	6	6	1190	1217	5	9	0	1219	1243	5	12	7	475	522
5	2	4	1127	1173	5	4	5	434	404	5	6	7	609	624	5	9	2	755	785	5	13	-10	1006	1054

(PYRIDINE)(CARBONYL)PHTHALOCYANATOOSMIUM(II) 10 FO AND 10 FC

H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCAL
5	13	-6	1891	1087	6	1	3	386	385	6	3	6	733	712	6	5	9	737	706	6	8	-8	893	88
5	13	-4	675	657	6	1	4	1140	1107	6	3	7	614	596	6	5	11	562	517	6	8	-6	890	89
5	13	-2	536	509	6	1	5	1262	1217	6	3	8	1103	1078	6	6	-17	744	797	6	8	-5	1170	114
5	13	0	632	612	6	1	6	408	390	6	3	9	495	462	6	6	-16	535	546	6	8	-4	830	81
5	13	1	370	391	6	1	7	534	497	6	3	11	430	426	6	6	-15	543	545	6	8	-3	486	48
5	13	2	846	866	6	1	8	915	887	6	3	12	708	671	6	6	-13	1138	1166	6	8	-2	897	89
5	13	4	772	792	6	1	9	805	752	6	4	-16	1048	1108	6	6	-11	558	534	6	8	-1	817	87
5	13	6	445	441	6	1	12	520	470	6	4	-13	518	503	6	6	-10	563	558	6	8	0	801	83
5	14	-9	780	803	6	1	13	399	355	6	4	-12	1398	1403	6	6	-9	1499	1448	6	8	2	810	84
5	14	-7	438	460	6	2	-17	737	771	6	4	-10	944	839	6	6	-8	838	834	6	8	3	754	75
5	14	-6	414	443	6	2	-15	459	466	6	4	-9	717	695	6	6	-7	360	349	6	8	5	449	46
5	14	-5	872	837	6	2	-13	1101	1106	6	4	-8	1531	1501	6	6	-6	440	431	6	8	6	1011	97
5	14	-4	475	491	6	2	-11	602	601	6	4	-6	1262	1242	6	6	-5	1577	1555	6	8	7	613	61
5	14	-1	808	893	6	2	-9	1856	1863	6	4	-5	500	489	6	6	-4	620	608	6	8	10	768	78
5	14	3	1007	990	6	2	-7	292	293	6	4	-4	1302	1172	6	6	-2	585	612	6	9	-15	924	93
5	15	-7	639	631	6	2	-5	1757	1754	6	4	-3	568	546	6	6	-1	1085	1126	6	9	-13	557	56
5	15	-3	655	703	6	2	-4	291	218	6	4	-2	1048	1076	6	6	0	608	585	6	9	-11	1172	116
5	15	-2	437	417	6	2	-2	688	664	6	4	-1	654	677	6	6	2	723	729	6	9	-9	473	45
5	15	1	1112	1134	6	2	-1	1620	1729	6	4	0	405	467	6	6	3	1395	1429	6	9	-7	1641	160
6	0	-16	747	770	6	2	1	477	493	6	4	1	404	395	6	6	4	362	410	6	9	-3	1452	143
6	0	-12	977	987	6	2	3	1673	1703	6	4	2	1601	1645	6	6	5	531	533	6	9	-1	346	32
6	0	-10	556	576	6	2	5	713	692	6	4	3	510	529	6	6	6	675	704	6	9	1	1173	121
6	0	-8	2115	2159	6	2	7	1391	1252	6	4	4	831	856	6	6	7	977	976	6	9	3	365	37
6	0	-6	921	943	6	2	9	593	550	6	4	6	1246	1260	6	6	8	427	443	6	9	5	1409	142
6	0	-4	958	996	6	2	11	619	532	6	4	7	404	384	6	6	9	606	596	6	9	7	520	50
6	0	-2	1826	1846	6	2	13	547	482	6	4	10	776	724	6	6	10	452	447	6	9	9	930	90
6	0	0	2006	1960	6	3	-18	857	903	6	5	-17	598	642	6	6	11	677	666	6	10	-13	557	56
6	0	2	1166	1163	6	3	-15	419	434	6	5	-15	1138	1193	6	7	-14	1441	1504	6	10	-12	1000	99
6	0	4	801	786	6	3	-14	695	691	6	5	-14	501	533	6	7	-12	663	663	6	10	-10	415	43
6	0	6	1593	1550	6	3	-13	588	591	6	5	-13	390	416	6	7	-10	1397	1386	6	10	-9	872	88
6	0	10	1359	1265	6	3	-12	575	555	6	5	-11	1628	1618	6	7	-8	672	646	6	10	-8	1045	102
6	1	-18	583	638	6	3	-11	788	810	6	5	-10	679	653	6	7	-7	337	313	6	10	-6	696	66
6	1	-17	513	570	6	3	-10	1566	1540	6	5	-9	416	384	6	7	-6	1356	1346	6	10	-5	961	93
6	1	-15	419	456	6	3	-8	1038	998	6	5	-8	475	461	6	7	-4	1272	1278	6	10	-4	908	89
6	1	-14	480	452	6	3	-7	993	965	6	5	-7	1544	1517	6	7	-3	351	359	6	10	-2	761	74
6	1	-13	367	317	6	3	-6	1555	1452	6	5	-6	560	543	6	7	-2	599	647	6	10	-1	1045	104
6	1	-11	977	991	6	3	-5	413	369	6	5	-4	385	364	6	7	0	1202	1247	6	10	2	664	68
6	1	-10	956	979	6	3	-4	1172	1136	6	5	-3	1530	1555	6	7	1	386	410	6	10	3	657	67
6	1	-9	713	717	6	3	-3	806	795	6	5	-2	312	303	6	7	2	855	860	6	10	6	734	82
6	1	-7	[illegible]	[illegible]	6	3	-2	629	368	6	5	0	326	368	6	7	4	935	917	6	10	7	706	70
6	1	-6	773	782	6	3	-1	[illegible]	[illegible]	6	5	1	1267	1345	6	7	8	974	976	6	11	-12	412	41
6	1	-4	495	519	6	3	0	1075	1175	6	5	2	511	509	6	8	-16	736	719	6	11	-10	677	91
6	1	-3	[illegible]	[illegible]	6	3	1	834	888	6	5	3	474	497	6	8	-15	416	436	6	11	-8	758	[illegible]
6	1	-2	1130	1101	6	3	2	1023	1079	6	5	4	739	772	6	8	-13	670	608	6	11	-6	1120	109
6	1	-1	494	493	6	3	3	252	265	6	5	5	1036	1124	6	8	-12	1056	1051	6	11	-4	1072	101
6	1	0	1097	1119	6	3	4	1410	1430	6	5	7	401	485	6	8	-10	533	514	6	11	-2	1044	107
6	1	1	1506	1541	6	3	5	859	850	6	5	8	467	413	6	8	-9	768	765	6	11	0	734	73

(PYRIDINE)(CARBONYL)PHTHALOCYANATOOSMIUM(II) 10 FO AND 10 FC

H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC
6	11	1	459	459	7	0	9	697	661	7	3	-8	564	547	7	[illegible]	[illegible]	[illegible]	411	7	8	-1	1291	1325
6	11	2	352	367	7	0	11	626	570	7	3	-7	1668	1626	7	5	4	[illegible]	1083	7	8	2	581	573
6	11	4	1174	1274	7	1	-18	707	829	7	3	-6	369	358	7	5	8	1296	1298	7	8	5	534	567
6	11	8	834	769	7	1	-17	437	473	7	3	-4	550	588	7	6	-16	368	682	7	8	6	805	833
6	12	-11	561	576	7	1	-16	433	466	7	3	-3	1503	1521	7	6	-15	372	418	7	8	7	890	864
6	12	-10	451	450	7	1	-15	702	719	7	3	-2	636	646	7	6	-14	582	621	7	8	9	522	487
6	12	-9	929	938	7	1	-14	732	724	7	3	-1	463	508	7	6	-13	534	555	7	9	-14	978	1009
6	12	-8	481	527	7	1	-13	402	407	7	3	0	980	1022	7	6	-12	1034	985	7	9	-12	511	536
6	12	-6	619	614	7	1	-12	628	625	7	3	1	1179	1253	7	6	-10	506	472	7	9	-10	645	643
6	12	-5	1198	1164	7	1	-11	952	982	7	3	3	471	459	7	6	-9	736	721	7	9	-8	857	845
6	12	-4	742	739	7	1	-10	1202	1217	7	3	4	483	517	7	6	-8	923	912	7	9	-6	1149	1128
6	12	-2	402	374	7	1	-8	1182	1156	7	3	5	915	928	7	6	-6	935	918	7	9	-4	1391	1367
6	12	-1	914	920	7	1	-7	1722	1672	7	3	7	406	408	7	6	-5	785	774	7	9	-2	859	890
6	12	2	557	573	7	1	-6	1329	1303	7	3	8	603	575	7	6	-4	890	913	7	9	0	727	750
6	12	3	843	896	7	1	-4	1176	1143	7	3	9	712	690	7	6	-2	843	906	7	9	4	1089	1120
6	12	4	445	515	7	1	-3	1291	1342	7	3	11	676	633	7	6	-1	630	644	7	9	8	959	922
6	12	5	371	344	7	1	-2	641	690	7	4	-17	948	968	7	6	0	750	772	7	10	-13	732	735
6	12	6	514	465	7	1	0	1211	1230	7	4	-13	920	897	7	6	1	614	624	7	10	-12	672	716
6	12	7	623	623	7	1	1	746	780	7	4	-11	344	293	7	6	2	1169	1225	7	10	-9	714	712
6	13	-10	394	376	7	1	2	471	471	7	4	-10	608	592	7	6	3	382	417	7	10	-8	598	594
6	13	-7	1347	1257	7	1	3	422	431	7	4	-9	1455	1421	7	6	6	976	959	7	10	-6	778	757
6	13	-4	407	414	7	1	4	1084	1043	7	4	-5	1803	1781	7	6	7	488	507	7	10	-5	1097	1093
6	13	-3	628	817	7	1	5	769	762	7	4	-2	702	727	7	6	10	843	836	7	10	-4	769	770
6	13	-2	353	346	7	1	7	696	675	7	4	-1	1296	1358	7	7	-15	1108	1144	7	10	-2	762	758
6	13	1	1068	1030	7	1	8	642	506	7	4	0	372	411	7	7	-14	405	415	7	10	-1	734	718
6	13	5	757	761	7	1	9	534	487	7	4	1	624	653	7	7	-11	919	912	7	10	2	527	534
6	14	-8	730	742	7	1	11	431	364	7	4	2	460	452	7	7	-10	380	369	7	10	3	608	678
6	14	-6	526	509	7	1	12	499	431	7	4	3	1633	1677	7	7	-7	1435	1403	7	10	5	535	526
6	14	-5	552	563	7	2	-16	1156	1216	7	4	5	512	496	7	7	-3	1648	1649	7	10	6	652	610
6	14	-4	549	531	7	2	-14	386	365	7	4	7	678	663	7	7	-2	397	417	7	10	7	551	532
6	14	-2	584	555	7	2	-12	949	952	7	4	9	741	703	7	7	-1	576	607	7	11	-11	1104	1162
6	14	0	617	620	7	2	-10	1153	1157	7	4	10	390	351	7	7	1	1208	1243	7	11	-7	1292	1291
6	14	2	849	821	7	2	-8	1594	1585	7	4	11	595	545	7	7	3	496	486	7	11	-3	1107	1089
6	15	-4	466	478	7	2	-6	1627	1611	7	5	-14	917	968	7	7	5	1024	1003	7	11	1	687	662
6	15	-3	501	532	7	2	-5	410	447	7	5	-12	865	855	7	7	7	795	807	7	11	3	407	404
7	0	-17	971	992	7	2	-4	1129	1117	7	5	-11	529	495	7	7	9	753	745	7	11	5	799	793
7	0	-15	430	448	7	2	-2	1511	1617	7	5	-10	883	854	7	8	-15	399	405	7	12	-9	549	590
7	0	-13	1179	1209	7	2	0	734	775	7	5	-9	349	345	7	8	-14	451	415	7	12	-8	532	501
7	0	-11	363	388	7	2	1	405	356	7	5	-8	932	943	7	8	-13	997	1030	7	12	-6	685	670
7	0	[illegible]	[illegible]	[illegible]	7	2	[illegible]	1221	1256	7	5	-7	419	417	7	8	-12	645	685	7	12	-5	536	[illegible]
7	0	-5	[illegible]	2519	7	2	[illegible]	1309	1323	7	5	-6	815	801	7	8	-10	675	661	7	12	-4	584	[illegible]
7	0	-3	451	494	7	2	10	964	884	7	5	[illegible]	1412	1408	7	8	-9	958	924	7	12	-2	694	[illegible]
7	0	-1	1595	1607	7	3	-18	627	689	7	5	-3	402	416	7	8	-8	633	615	7	12	0	497	517
7	0	1	479	477	7	3	-15	795	827	7	5	-2	623	649	7	8	-6	688	684	7	12	2	609	[illegible]
7	0	3	509	537	7	3	-14	470	508	7	5	0	1245	1322	7	8	-5	1113	1117	7	12	3	602	590
7	0	5	718	799	7	3	-11	1197	1172	7	5	1	543	660	7	8	-4	412	437	7	13	-8	525	575
7	0	7	1121	1099	7	3	-10	834	802	7	5	2	619	637	7	8	-2	944	970	7	13	-6	752	716

(PYRIDINE)(CARBONYL)PHTALOCYANATOOSMIUM(II) 10 FO AND 10 FC

PAGE 10

H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC
7	13	-4	723	667	8	2	-2	377	411	8	5	-15	1061	1111	8	8	-12	397	390	8	13	-3	965	922
7	13	-2	393	336	8	2	-1	1226	1310	8	5	-14	439	453	8	8	-10	531	527	9	0	-17	925	943
7	13	0	769	771	8	2	1	727	755	8	5	-11	1245	1254	8	8	-9	581	587	9	0	-13	777	793
7	14	-5	914	843	8	2	3	1247	1238	8	5	-8	525	519	8	8	-6	812	804	9	0	-9	2373	2472
7	14	-2	379	365	8	2	5	758	735	8	5	-7	1356	1332	8	8	-5	732	718	9	0	-5	1504	1490
7	14	-1	812	770	8	2	7	590	537	8	5	-4	679	677	8	8	-4	449	442	9	0	-3	516	552
8	0	-16	[illegible]	1390	8	2	9	773	694	8	5	-3	1648	1675	8	8	-2	970	980	9	0	-1	1037	1006
8	0	-14	746	746	8	2	10	395	419	8	5	-2	453	441	8	8	-1	700	743	9	0	1	1124	1087
8	0	-12	543	538	8	3	-16	384	371	8	5	-1	776	784	8	8	2	635	623	9	0	3	1410	1352
8	0	-10	936	971	8	3	-15	417	447	8	5	0	559	564	8	8	3	592	583	9	0	5	997	922
8	0	-8	1520	1536	8	3	-14	966	1007	8	5	1	1266	1308	8	8	6	916	863	9	0	9	521	432
8	0	-6	1679	1660	8	3	-12	992	1010	8	5	3	400	358	8	9	-11	925	931	9	1	-16	528	548
8	0	-4	478	535	8	3	-11	1088	1101	8	5	4	370	357	8	9	-7	857	842	9	1	-15	750	810
8	0	-2	1362	[illegible]	8	3	-10	1016	1015	8	5	5	435	416	8	9	-3	1032	1017	9	1	-14	680	706
8	0	0	510	504	8	3	-8	907	[illegible]	8	5	7	644	605	8	9	-1	449	418	9	1	-12	761	785
8	0	2	1279	1261	8	3	-7	780	784	8	5	8	369	323	8	9	1	647	635	9	1	-11	843	850
8	0	6	1445	1342	8	3	-6	900	862	8	5	9	565	558	8	9	3	613	608	9	1	-10	751	793
8	0	10	826	760	8	3	-5	405	405	8	6	-16	566	561	8	9	5	831	813	9	1	-9	388	402
8	1	-16	418	451	8	3	-4	1391	1370	8	6	-13	1275	1287	8	9	7	634	584	9	1	-8	1153	1172
8	1	-15	1087	1162	8	3	-3	957	974	8	6	-10	488	501	8	10	-13	398	400	9	1	-7	984	981
8	1	-14	439	429	8	3	-2	411	439	8	6	-9	1050	1044	8	10	-12	623	621	9	1	-4	854	899
8	1	-13	390	448	8	3	0	1415	1518	8	6	-8	371	331	8	10	-10	529	529	9	1	-3	550	569
8	1	-12	354	361	8	3	1	801	861	8	6	-6	396	378	8	10	-9	396	380	9	1	-1	505	501
8	1	-11	843	849	8	3	4	957	950	8	6	-5	1203	1271	8	10	-6	504	487	9	1	0	1147	1135
8	1	-10	715	750	8	3	5	386	[illegible]	8	6	-4	603	550	8	10	-5	534	517	9	1	1	772	762
8	1	-8	1128	1134	8	3	7	423	412	8	6	-3	605	602	8	10	-4	426	458	9	1	3	753	718
8	1	-7	1585	1559	8	3	8	846	811	8	6	-2	850	845	8	10	-2	736	735	9	1	4	1107	1005
8	1	-6	802	783	8	3	9	405	457	8	6	-1	1395	1437	8	10	-1	747	735	9	1	5	476	443
8	1	-5	325	302	8	4	-16	748	758	8	6	1	570	582	8	10	2	673	670	9	1	7	550	479
8	1	-4	923	956	8	4	-14	793	817	8	6	3	583	557	8	10	3	521	501	9	1	8	510	414
8	1	-3	776	804	8	4	-13	543	541	8	6	5	594	577	8	11	-10	564	610	9	2	-16	898	942
8	1	-2	599	634	8	4	-12	1350	1375	8	6	6	630	644	8	11	-8	452	435	9	2	-14	860	890
8	1	-1	486	511	8	4	-10	956	950	8	6	7	583	547	8	11	-6	441	439	9	2	-12	757	760
8	1	0	951	972	8	4	-9	487	484	8	6	9	530	564	8	11	-4	890	852	9	2	-10	1163	1220
8	1	1	978	981	8	4	-8	900	888	8	7	-14	877	912	8	11	0	934	923	9	2	-8	709	660
8	1	3	831	837	8	4	-6	1161	1152	8	7	-12	682	709	8	11	4	807	786	9	2	-6	1593	1572
8	1	4	718	721	8	4	-5	556	521	8	7	-10	620	584	8	12	-10	411	421	9	2	-4	355	331
8	1	5	861	815	8	4	-4	724	755	8	7	-8	758	754	8	12	-9	633	622	9	2	-2	1131	1203
8	1	7	413	361	8	4	-2	1582	1542	8	7	-6	714	713	8	12	-6	410	408	9	2	2	1276	[illegible]
8	1	9	756	[illegible]	8	4	-1	391	410	8	7	-4	1290	1300	8	12	-5	786	774	9	2	5	[illegible]	[illegible]
8	2	-17	[illegible]	[illegible]	8	4	0	579	610	8	7	-3	382	411	8	12	-4	400	398	9	2	6	[illegible]	[illegible]
8	2	-13	[illegible]	[illegible]	8	4	1	583	596	8	7	-2	622	636	8	12	-3	543	515	9	3	-15	992	[illegible]
8	2	-10	361	[illegible]	8	4	2	1472	[illegible]	8	7	[illegible]	[illegible]	1017	8	12	-2	435	390	9	3	-12	[illegible]	[illegible]
8	2	-9	1640	1647	8	4	3	393	380	8	7	4	1042	1000	8	12	-1	741	736	9	3	-11	979	1000
8	2	-7	[illegible]	418	8	4	6	608	[illegible]	8	7	8	772	714	8	12	2	412	424	9	3	-10	[illegible]	[illegible]
8	2	-5	[illegible]	[illegible]	8	4	9	406	392	8	8	-14	457	455	8	13	-7	668	[illegible]	9	3	-8	543	540
8	2	-3	404	[illegible]	8	4	10	860	884	8	8	-13	621	616	8	13	-4	[illegible]	355	9	3	-7	1346	1310

(PYRIDINE)(CARBONYL)PHTHALOCYANATOOSMIUM(II) 10 FO AND 10 FC

H	K	L	FOBS	FCALC
9	3	-4	635	609
9	3	-3	1121	1145
9	3	-1	676	706
9	3	0	665	579
9	3	1	904	932
9	3	3	555	528
9	3	4	530	549
9	3	5	555	526
9	3	7	608	537
9	3	8	463	406
9	4	-13	1500	1566
9	4	-12	464	501
9	4	-10	448	483
9	4	-9	523	471
9	4	-7	469	457
9	4	-5	1142	1112
9	4	-4	341	335
9	4	-3	694	668
9	4	-2	392	372
9	4	-1	830	863
9	4	1	718	782
9	4	3	673	692
9	4	5	651	635
9	4	6	377	335
9	4	7	538	477
9	5	-15	434	451
9	5	-14	836	831
9	5	-12	1010	1047
9	5	-11	529	585
9	5	-10	593	549
9	5	-8	925	932
9	5	-6	538	528
9	5	-5	661	643
9	5	-4	1058	1055
9	5	-2	441	460
9	5	0	957	953
9	5	4	853	831
9	6	-14	757	757
9	6	-13	492	498
9	6	-12	725	764
9	6	-11	[illegible]	757
9	6	-10	757	750
9	6	-9	516	512
9	6	-8	510	558
9	6	-6	757	724
9	6	-5	659	[illegible]
9	6	-2	1141	1136

H	K	L	FOBS	FCALC
9	6	-1	515	553
9	6	1	582	582
9	6	2	1019	1010
9	6	5	394	363
9	6	6	711	683
9	7	-11	1134	1134
9	7	-7	985	950
9	7	-3	1009	1003
9	7	-1	675	688
9	7	1	869	860
9	7	3	778	751
9	7	5	641	617
9	8	-13	727	732
9	8	-12	579	581
9	8	-10	564	580
9	8	-9	1113	1141
9	8	-8	490	501
9	8	-6	377	344
9	8	-5	603	568
9	8	-4	400	366
9	8	-3	507	516
9	8	-2	746	768
9	8	-1	1021	991
9	8	1	472	494
9	8	2	676	668
9	8	3	478	419
9	8	5	549	515
9	9	-12	605	586
9	9	-10	608	597
9	9	-8	816	777
9	9	-6	554	511
9	9	-4	1251	1226
9	9	0	1186	1187
9	9	4	985	934
9	10	-10	675	661
9	10	-9	747	719
9	10	-6	546	519
9	10	-5	684	681
9	10	-4	377	378
9	10	-3	527	521
9	10	-2	754	700
9	10	-1	721	699
9	10	2	659	609
9	10	3	451	412
9	11	-7	712	697
9	11	-5	522	523
9	11	-3	870	807

H	K	L	FOBS	FCALC
9	11	-1	530	517
9	11	1	699	719
9	12	-6	742	726
9	12	-5	425	385
9	12	-2	734	724
10	0	-16	553	603
10	0	-14	782	892
10	0	-12	430	458
10	0	-10	1311	1327
10	0	-8	761	765
10	0	-6	611	579
10	0	-2	637	607
10	0	2	961	871
10	0	6	920	825
10	1	-16	414	458
10	1	-15	736	732
10	1	-12	610	619
10	1	-11	887	937
10	1	-10	377	370
10	1	-9	453	508
10	1	-8	938	951
10	1	-7	868	837
10	1	-5	617	640
10	1	-4	686	743
10	1	-3	531	548
10	1	-1	385	362
10	1	0	555	545
10	1	1	481	425
10	1	3	597	538
10	1	4	642	606
10	1	7	570	501
10	2	-13	1043	1127
10	2	-9	1224	1224
10	2	-7	655	639
10	2	-5	921	923
10	2	-3	590	627
10	2	-1	359	336
10	2	1	748	723
10	2	3	779	748
10	2	5	821	751
10	3	-15	399	463
10	3	-14	586	638
10	3	-12	757	779
10	3	-11	520	515
10	3	-10	452	450
10	3	-9	404	397
10	3	-8	765	720

H	K	L	FOBS	FCALC
10	3	-7	696	653
10	3	-6	403	386
10	3	-4	860	883
10	3	-1	405	423
10	3	0	772	762
10	3	1	427	415
10	3	3	465	445
10	3	4	1022	959
10	4	-14	785	849
10	4	-12	514	534
10	4	-10	713	710
10	4	-9	459	494
10	4	-6	1618	1618
10	4	-5	473	453
10	4	-2	1002	1016
10	4	1	467	475
10	4	2	1087	1076
10	4	6	881	809
10	5	-11	942	950
10	5	-9	402	367
10	5	-8	426	403
10	5	-7	1233	1240
10	5	-5	447	404
10	5	-4	478	506
10	5	-3	766	761
10	5	-1	811	832
10	5	0	424	406
10	5	1	582	576
10	5	3	803	808
10	5	5	469	446
10	6	-13	754	745
10	6	-10	471	477
10	6	-9	825	787
10	6	-7	450	424
10	6	-6	458	422
10	6	-5	927	896
10	6	-4	440	444
10	6	-3	653	680
10	6	-2	769	700
10	6	-1	970	939
10	6	1	604	587
10	6	2	475	474
10	6	3	410	388
10	6	5	706	606
10	7	-12	920	938
10	7	-10	580	584
10	7	-8	1133	1118

H	K	L	FOBS	FCALC
10	7	-4	1074	1048
10	7	0	1019	991
10	7	4	828	822
10	8	-12	422	414
10	8	-10	1120	1135
10	8	-9	690	695
10	8	-7	439	411
10	8	-6	549	523
10	8	-5	518	511
10	8	-2	875	860
10	8	-1	473	488
10	8	1	545	525
10	8	2	646	630
10	9	-11	1055	1056
10	9	-7	726	702
10	9	-5	573	545
10	9	-3	808	807
10	9	-1	582	570
10	9	1	655	654
10	10	-9	818	819
10	10	-8	490	507
10	10	-6	612	603
10	10	-5	418	377
10	10	-2	637	598
10	10	-1	477	477
11	0	-13	783	786
11	0	-11	495	504
11	0	-9	931	903
11	0	-7	522	472
11	0	-5	733	778
11	0	-3	828	856
11	0	-1	365	312
11	0	1	831	765
11	0	3	733	658
11	1	-12	648	658
11	1	-11	581	588
11	1	-9	856	827
11	1	-7	446	450
11	1	-4	952	[illegible]
11	1	-3	468	481
11	1	-1	457	445
11	1	0	527	453
11	1	1	441	430
11	1	2	400	373
11	1	3	543	481
11	1	4	790	685
11	2	-14	774	802

(PYRIDINE)(CARBONYL)PHTHALOCYANATOOSMIUM(II) 10 FO AND 10 FC

H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC	H	K	L	FOBS	FCALC
11	2	-10	849	806	11	8	-9	721	713															
11	2	-6	1245	1228	11	8	-7	386	389															
11	2	-2	928	939	11	8	-6	555	550															
11	2	1	392	387	11	8	-5	517	506															
11	2	2	812	744	11	8	-3	714	725															
11	3	-12	463	447	11	8	-2	600	612															
11	3	-11	605	574	11	8	-1	402	371															
11	3	-9	565	569	11	9	-4	978	995															
11	3	-8	553	549	12	0	-10	690	849															
11	3	-7	893	845	12	0	-6	974	1041															
11	3	-6	451	428	12	0	-2	996	955															
11	3	5	822	819	12	1	-12	555	575															
11	3	-3	519	535	12	1	-11	534	571															
11	3	-1	579	579	12	1	-9	539	520															
11	3	0	438	450	12	1	-8	576	518															
11	3	3	660	596	12	1	-7	549	526															
11	4	-13	777	780	12	1	-5	631	600															
11	4	-11	391	394	12	1	-4	781	781															
11	4	-10	400	382	12	1	-3	624	598															
11	4	-9	806	771	12	1	-1	588	576															
11	4	-7	769	736	12	1	0	540	487															
11	4	-6	498	471	12	2	-11	455	471															
11	4	-5	984	962	12	2	-9	723	690															
11	4	-3	526	515	12	2	-7	675	589															
11	4	-1	589	608	12	2	-5	572	590															
11	4	1	510	477	12	2	-3	899	953															
11	5	-12	789	761	12	2	-1	415	406															
11	5	-8	1021	982	12	2	1	719	668															
11	5	-7	439	430	12	3	-8	815	736															
11	5	-5	380	[illegible]	12	3	-7	411	370															
11	5	-4	[illegible]	[illegible]	12	3	-4	[illegible]	804															
11	5	0	742	729	12	3	-1	534	512															
11	6	-10	902	906	12	3	0	669	661															
11	6	-9	415	436	12	4	-10	754	685															
11	6	-6	982	952	12	4	-6	[illegible]	882															
11	6	-5	435	403	12	4	-2	875	841															
11	6	-3	451	421	12	5	-9	525	476															
11	6	-2	696	602	12	5	-7	[illegible]	557															
11	6	1	399	408	12	5	-5	747	703															
11	6	2	620	588	12	5	-1	751	735															
11	7	-11	960	876	12	6	-7	549	517															
11	7	-9	562	531	12	6	-5	631	605															
11	7	-7	844	845	12	6	-3	519	437															
11	7	-5	565	546																				
11	7	-3	631	678																				
11	7	-1	790	777																				
11	7	1	445	427																				