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Synthesis and Characterization of Imidazolo 3,1-Tetrakis (N-phenylacetamidato) Dirhodium (II) and a Crystallographic Study of a Copper and Two Molybdenum Model Cofactors

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by

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ABSTRACT

Synthesis and Characterization of Imidazolo 3,1- Tetrakis (N-phenylacetamidato) Dirhodium (II) and a Crystallographic Study of a Copper and Two Molybdenum Model Cofactors

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Imidazole was reacted with 3,1-tetrakis (N-phenlyacetamidato) dirhodium (II) to explore the chemistry of asymmetric dirhodium catalysts. The imidazolo 3,1-tetrakis (N-phenlyacetamidato) dirhodium (II) complex was synthesized and then characterized by Nuclear Magnetic Resonance and Ultraviolet-Visible spectroscopies as well as by single crystal X-ray Diffraction. Additionally, one copper and two molybdenum model cofactors were characterized by XRD to better understand their structure/function relationships. NMR results gave evidence of the formation of the 3,1-imidazole complex, and UV-Vis indicated that even in large excess imidazole was coordinated only to one axial site. The structure of the 3,1-imidazole complex was confirmed by XRD with the following refinement indicators: R_1: 3.97%, wR_2: 9.27%, GooF: 1.036. Model cofactors were also characterized by XRD and resulted in the following refinement indicators for Mo-1: R_1: 4.27%, wR_2: 9.15%, GooF: 1.074; for Cu-1: R_1: 10.10%, wR_2: 22.60%, GooF: 0.991, and for Mo-2: R_1: 17.75%, wR_2: 46.08%, GooF: 0.954.

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2,2-*cis*	2,2-*cis* Tetrakis(N-phenylacetamidato) Dirhodium(II) (2,2-*cis* $Rh_2(PhNCOCH_3)_4$)
2,2-*trans*	2,2-*trans* Tetrakis(N-phenylacetamidato) Dirhodium(II) (2,2-*trans* $Rh_2(PhNCOCH_3)_4$)
3,1-complex	3,1-Tetrakis(N-phenylacetamidato) Dirhodium(II) (3,1- $Rh_2(PhNCOCH_3)_4$)
3,1-Imidazole	Imidazolo 3,1-Tetrakis(N-phenylacetamidato) Dirhodium(II) (3,1- $Rh_2(PhNCOCH_3)_4N_2C_3H_4$)
$CDCl_3$	Deuterated Chloroform
CD_2Cl_2	Deuterated Dichloromethane
CH_2Cl_2	Dichloromethane
CCD	Charge Coupled Device
DMSO	Dimethylsulfoxide
NMR	Nuclear Magnetic Resonance
ORTEP	Oak Ridge Thermal Ellipsoid Plot
Ph	Phenyl
TLC	Thin Layer Chromatography
UV-Vis	Ultraviolet-Visible Spectroscopy
XRD	X-ray Diffraction

CHAPTER 1

INTRODUCTION

Chemistry involves various reactions; some occurring spontaneously, others only if a great amount of energy is introduced into the system. Catalysts are very useful for the second group mentioned. Catalysts generally function by forming a reaction intermediate with lower activation energy than would normally be required without the catalyst, which both lowers the energy needed to complete the reaction and increases the reaction rate.[1] By definition, catalysts are not consumed in the reaction, but they may degrade over time with use. They can be found in biological systems and are also used for industrial purposes and will often play crucial roles in both areas. Because of their importance and prevalence, research to better synthesize, characterize, and modify catalysts is imperative. This thesis examines rhodium, copper, and molybdenum catalysts and seeks to better understand their structure.

Catalysts

Catalysts come in a variety of forms including metallic, non-metal, organometallic, or enzymes.[1] They also can be categorized as either homogeneous – being in the same phase as the reactants – or heterogeneous – being in a different phase. Heterogeneous catalysts are most often solids that interact with either liquid or gas phase reactants, which gives the benefit of being easy to separate after the reaction is completed, but comes with the downside of catalysis only occurring on the surface of the catalyst. Homogeneous catalysts, on the other hand, have greater interaction with the reactants, but have to be separated out to be recovered for further use.

Metal-Metal Bonding

The majority of the work done for this thesis involved the use of dirhodium complexes, which have two rhodium atoms in the center of the complex bonded to each other. Bonds between metal atoms result from the overlap of the atomic d orbitals and can form three different types of bonds: σ, π, and δ bonds.[2] By convention, the axis of interaction between metal bonds is

the z-axis, and the five d orbitals that can participate in bonding are the d_{xy}, d_{xz}, d_{yz}, d_z^2, and d_x^2-$_y^2$. The five d orbitals interact between atoms to form three types of bonds.

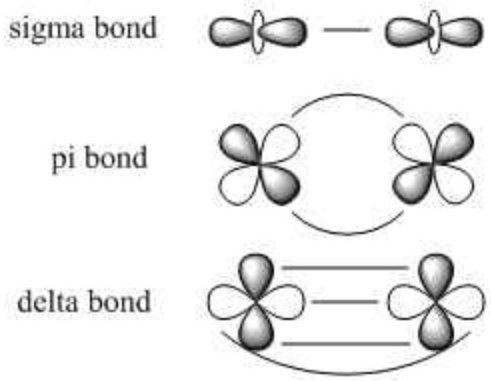


Figure 1.1: Diagram showing the possible metal bonding orbitals along the z-axis; σ bonds are formed by interaction of two d_z^2 orbitals, π bonds are formed by the interaction of two d_{xz} or two d_{yz} orbitals, and δ bonds are formed by the interaction of two d_{xy} orbitals

The strongest of the three types of bonds is the σ bond. The σ bonds are strongest because they have direct overlap with from the two d_z^2 orbitals on the z axis. The second strongest is the π bond. These are formed by the interaction of two d_{xz} and two d_{yz} orbitals. There is not as much direct overlap of these orbitals, so the bond is not as strong as with σ bonds. The third and weakest bond that can be found is the δ bond. It is the weakest because the two d_{xy} orbitals involved are parallel to each other, and as a result, are the farthest apart.

In simplest terms of this qualitative MO diagram[1] in an octahedral environment, the two metal atoms can only form four bonding molecular orbitals, as the d_x^2-$_y^2$ is utilized by ligand metal bonding along the x and y axes, and consequently, is unavailable for metal-metal bonding. The orbitals can be filled by the remaining valence electrons of the metal atoms and those donated by any ligands. Metal-metal bonds with a bond order of four are not too common among transition block metals but do occur much more often than bond orders of five. The first confirmed compound containing a quintuple bond was a dichromium complex isolated in 2005.[3]

The dirhodium(II) complexes studied are dimers with rhodium-rhodium cores. The bond order between the two rhodium atoms is only one, however. This is because between the two Rh(II) atoms, they have a total number of 14 electrons resulting in an electron configuration of

$\sigma^2\pi^4\delta^2\delta^{*2}\pi^{*4}$. The δ^* and π^* orbitals cancel out the bonding δ and π orbitals resulting in only a single σ bond.

Diazo Compounds and Their Reactions

Diazo compounds contain two nitrogen atoms linked by a double bond as a terminal functional group. In the presence of a dirhodium catalyst, the nitrogen atoms separate from the rest of the molecule as nitrogen. The remaining carbon atom that was bonded to the secondary nitrogen atom is left as a very labile carbene, which is a neutral carbon with two bonds and a lone pair of electrons. These carbenes that are generated can then be used in a variety of reactions including, cyclopropanation, C-H insertion, and carbene dimerization.[1]

In a cyclopropanation reaction, a carbene reacts with an alkene to form a three membered ring of carbon atoms. Depending on the orientation of the substituents on the alkene and carbene, either *cis* or *trans* cyclopropanes can be formed.[2] The *cis/trans* ratio is important for production of the compounds because one isomer may have a higher biological activity than the other. In the case of synthesizing pyrethroids, a class of synthetic pesticides based on the natural product pyrethrum, a *cis* configuration is preferred.[4]

Preparation of Dirhodium(II) Compounds

Dirhodium(II) compounds are useful for a number of different purposes, including antitumor studies,[5] but their catalytic activity is the most noteworthy. Once metal-metal bonds and compounds such as chromium (II) acetate were discovered,[6] this new class of bimetallic compounds was researched.[7] As part of a continued study in 1973, rhodium(II) acetate, ($Rh_2(CH_3CO_2)_4$), was synthesized and found to be able to catalyze the decomposition of diazo compounds,[8] making further reactions such as cyclopropanations easier to perform. These catalytic properties involving diazo compounds indicated that rhodium(II) acetate could be quite useful for other reactions, prompting additional study.[8]

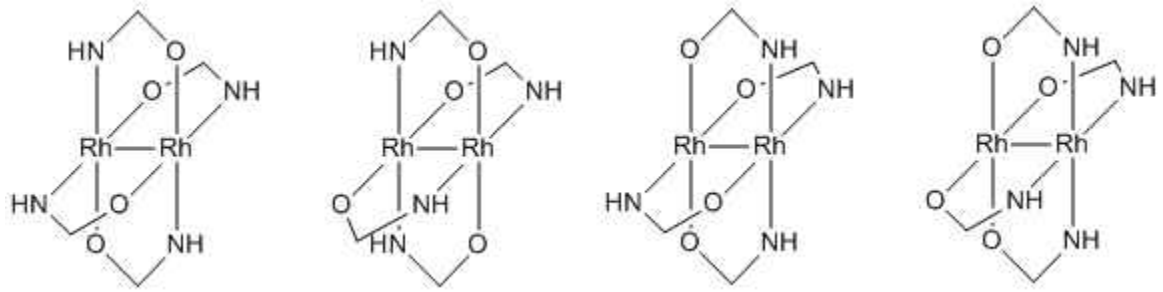
Rhodium(II) acetate has been synthesized by refluxing rhodium(III) chloride trihydrate with glacial acetic acid and sodium acetate for one hour.[2,9] The synthesized rhodium(II) acetate could then be further reacted if other bridging ligands were needed via substitution reactions.[9]

$$4\ RhCl_3{\cdot}3\ H_2O + 8\ CH_3COOH + CH_3CH_2OH \rightarrow$$
$$2\ Rh_2(OCOCH_3)_2 + 12\ HCl + CH_3COOH + 11\ H_2O \quad (1)$$

Because of the usefulness of the dirhodium catalysts, many derivatives were formed,[2] and dirhodium complexes with various axial and equatorial ligands have been synthesized.[2,7,8] The Rh-Rh bond has been proven to be quite stable, with its bond length remaining relatively constant with different equatorial ligands.[2]

With the modification of the equatorial chelating ligands comes a change in reactivity. More electron withdrawing groups or electron donating groups can be used, which have an impact on the electrophilicity of the rhodium atoms, and by consequence, the catalytic properties of the complex. In addition, equatorial ligands with large, bulky groups can change the reactivity of the rhodium complex by adding steric hindrance to the site of coordination.

In the 1970s, Bear, Kadish, and coworkers synthesized a new rhodium complex with acetamide bridging ligands. Using acetamide as a ligand introduces asymmetry, however, and as a result, four isomers of the tetrasubstituted complex can be formed. These are denoted as 2,2-*cis*, 2,2-*trans*, 3,1 and 4,0; they are named as such based on the positions of the nitrogen atoms on one of the rhodium atoms. See Figure 1.2 below.



2,2-*cis* 2,2-*trans* 3,1- 4,0-

Figure 1.2: Figure depicting a simplified form of the tetrakis acetamidato dirhodium (II) isomers

A similar derivative that was later produced used N-phenyl acetamide, which gave similar compounds as shown above, except for the phenyl groups replacing the hydrogen atoms on each nitrogen atom.[9] This introduced greater steric hindrance at the axial sites, yielding

greater selectivity in cyclopropanations.[10] This is especially applicable with reactions involving diazo compounds.

Cyclopropanation Using Dirhodium(II) Compounds

The dirhodium(II) compounds can react with diazo compounds to generate carbenoids, which are similar in their reactivities to carbenes but differ in that they are stabilized by a metal atom. The carbenoid is then available to perform carbon insertions, cyclopropanations, and cyclopropenations among other reactions.[2]

Cyclopropanation is particularly useful since cyclopropane is typically difficult to synthesize due to its having 60° bond angles instead of the preferred 109.5° for sp^3 hybridized carbon atoms. The method of cyclopropanation using the dirhodium catalyst involves the reaction of a carbenoid with an alkene to form the cyclopropane. This can lead to the formation of either a *cis* or *trans* cyclopropane depending on the orientation of the molecules upon reaction. Making the reaction more selective towards one product is useful, since one of the isomers is typically the preferred synthetic product, and any of the other isomers formed are not helpful. Studies have been performed to modify the *cis*/*trans* ratio produced when using rhodium phenylacetamide.[10] The data gathered in this study are summarized in Table 1.1 below.

Table 1.1: The reported *cis*/*trans* product ratios of cyclopropanation reactions of different alkenes with the three tetrakis(N-phenylacetamidato) dirhodium(II) isomers.[10]

Alkene	3,1	2,2-*trans*	2,2-*cis*
Cyclohexene	0.2	0.3	0.3
Ethyl vinyl ether	0.7	0.6	1.0
Styrene	0.8	1.2	1.1
Trimethyl styrene	1.0	1.2	1.8

These data show that greater steric hindrance in both the alkene and the dirhodium complex leads to the formation of a larger proportion of the *cis* isomer. The difference is most notable in the 2,2-*cis* and 2,2-*trans* rhodium isomers, but not as much with the 3,1-isomer, where the *cis/trans* ratio only increases from 0.2 to 1.0, compared to the 0.3 to 1.8 difference of the 2,2-*cis*. The suspected reason for this was that cyclopropanation would take place on the less sterically hindered "1" side of complex.[9] Because of the success of these results in generating a greater amount of *cis*-cyclopropane, it would be interesting to see what the *cis*/*trans* ratios would be like if the reaction were forced to be done on the more sterically hindered "3" side of the 3,1-isomer. See Figure 1.3 below for a depiction of the sides of the 3,1-isomer.

Axial Ligation of the 3,1-Isomer

In order to test the selectivity of the "3" side of the 3,1-isomer, it would be necessary to coordinate an axial ligand to the "1" side. The theory behind this would be that having a stable axial ligand on the "1" side would force catalysis to occur on the "3" side, thereby using the more sterically hindered side to generate a higher ratio of *cis* to *trans* cyclopropane.

Many structures of dirhodium compounds with axial ligands have been published in the past.[2, 6, 11-13] In these compounds, electron donors bond to the axial site of the electron deficient rhodium atom.

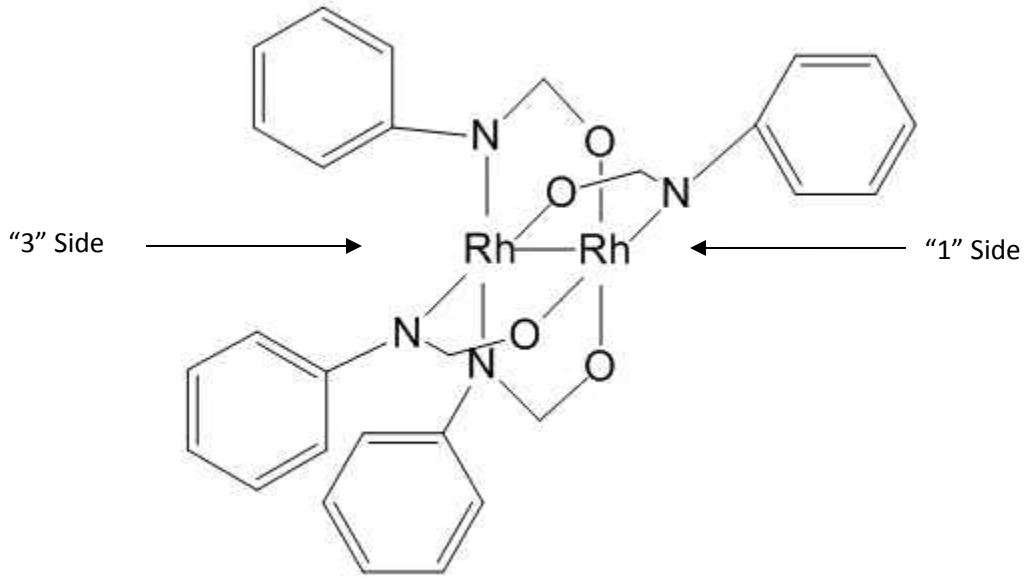


Figure 1.3: Figure showing the sides of the 3,1-isomer

Ligands with a lone pair of electrons make particularly good ligands in this case, and as such, nitrogen containing compounds were often chosen.[2, 6, 11-13]

The selection of a suitable axial ligand also includes the need for it to bond only to the axial site of the "1" side and not to both axial sites, otherwise the catalyst would not function at all. This means that the axial ligand needs to be somewhat bulky itself, so that it cannot fit in between the three phenyl rings of the "3" side. Previous research has also shown that some axial ligands can inhibit the reactivity of the catalyst, so those would also have to be avoided.[14] These aspects were taken into consideration, and previous research done in the Eagle research group has shown imidazole to be a good candidate for use as the axial ligand for this research.[15]

The main goal of this research was to synthesize a mono substituted 3,1-imidazole complex with the most sterically hindered axial site being open and active for catalytic activity. The three phenyls rings result in a significant barrier to interaction between the rhodium center and an approaching ligand. However, 3,1-isomer di-adducts made with nitriles that are comparatively long and narrow have been developed in previous studies in the Eagle research group, which has shown that it is possible to fit something into the sterically hindered axial site. Nitriles cannot be used for the purpose of occupying the axial site of the "1" side due to their reactivity with diazo compounds in addition to the fact that they would likely coordinate to the "3" side as well.

Molybdenum and Copper Cofactors

Cofactors are molecules or atoms that can modify the activity of an enzyme. They are essential to the workings of many different enzymes found in biological systems. Molybdenum was found to be cofactor in certain enzymes of different plants and animals.[16] Examples of enzymes that contain molybdenum cofactors are sulfite oxidase, nitrogenase, and nitrate reductase.[16] Copper is also commonly found as a cofactor in various biological systems.[17] It can be found in the active site of cytochrome c oxidase, superoxide reductase, and galactose oxidase among many others.[17] Without the molybdenum or copper cofactors, these enzymes could not function.

Because these cofactors are prevalent in biological systems, a significant amount of research is put into understanding how they function. Determining how these cofactors work can be done by both examining the naturally occurring cofactors in the enzymes of living organisms and synthesizing and testing model cofactors that can be modified to observe changes in reactivity.[16, 17] One of the first steps in understanding how these cofactors and model cofactors work is to know the molecular structure of these compounds, and one of the best methods of determining this is through X-ray Diffraction.

History of X-ray Diffraction

Knowing the arrangement of atoms in molecules is crucial to understanding how they work. This is especially true when trying to make effective catalysts by modifying the structure. With the discovery of X-rays in 1895 by Wilhelm Conrad Röntgen and the first use of X-rays in diffraction by Max Von Laue in 1912, our ability to discover the structure of crystalline compounds was greatly enhanced.[18, 19]

X-ray radiation, like other forms of light, can be separated into component wavelengths using a diffraction grating. The obstacle with diffracting X-rays, however, is their very short wavelength means that the spacing of the lines in a diffraction grating must be similarly short. Von Laue theorized that crystal lattices might be the about the right size to act as a diffraction grating since the distances between atoms in a crystalline solid would be comparable to the short X-ray wavelength.[18, 19] Through the work of Walter Friedrich and Paul Knipping, the large scale order of the crystal along with the right spacing for diffraction caused by inter-atomic distances led to the successful working of diffraction as von Laue predicted. Building upon the foundation of von Laue, Friedrich and Knipping, father and son duo W. H. Bragg and W. L. Bragg were able to mathematically describe the relationship between the wavelength of X-rays and the diffraction pattern generated when the rays are diffracted through a crystal.

Theory of X-ray Diffraction

A crystal is a solid where the atoms, molecules, or both are arranged in a three dimensional repeating pattern.[20] These repeating patterns can form when the solid forms slowly enough for the atoms and molecules to arrange themselves into the orientation that is most favorable energetically. Given enough time to move around and change orientation, the molecules will end up forming this repeating pattern minimizing any repulsive forces at work in the system.[20] It is this repeating pattern that makes X-ray crystallography possible.

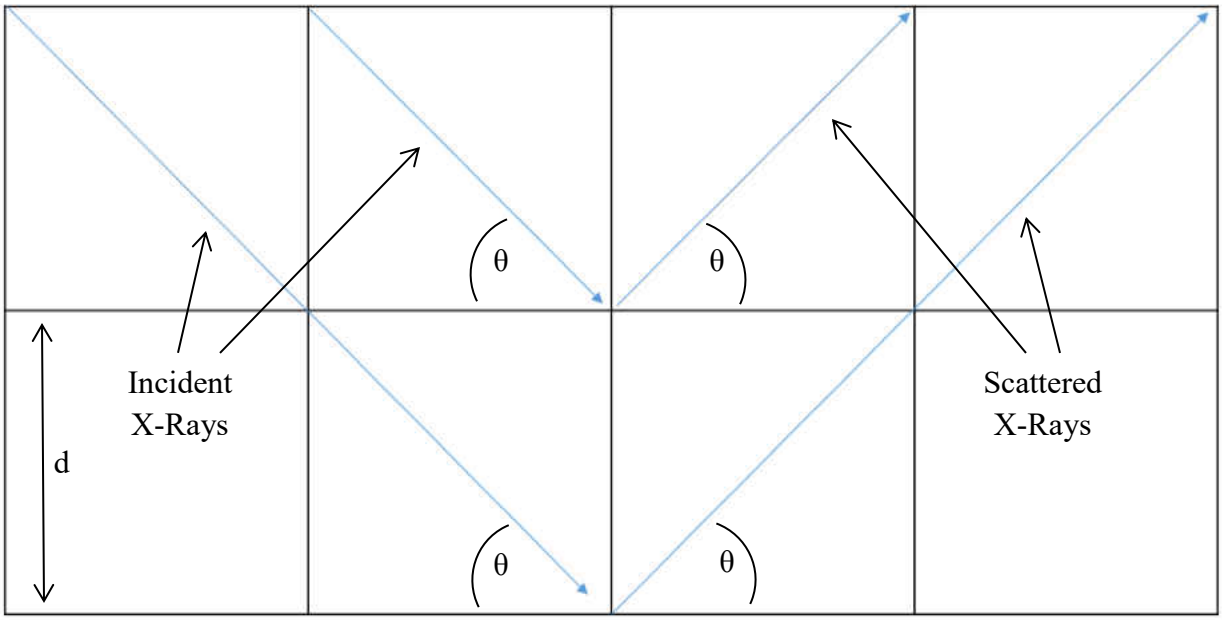


Figure 1.4: Representation of the diffraction pattern of parallel lines in a two-dimensional crystal

In the figure above, two parallel lines of incident radiation which then reflect back out of the crystal. Looking at this diagram, it can be shown that the bottom line travels twice the distance of top line. If this difference in distances is an integer value of the wavelength, the two rays will interact with constructive interference. Any other non-integer wavelength value, will lead to less constructive interference and even destructive interference if the beams are out of phase.[20]

Bragg was able to derive an equation for the conditions of constructive interference of diffraction patterns, which is known as Bragg's Law, shown in equation 1.2 below, where d is

the distance between the layers of a crystal, θ is the angle between the incident ray and plane at which it diffracts, n is an integer, and λ is the wavelength of the x-ray.[20]

$$n\lambda = 2d \sin\theta \quad (2)$$

The repeating pattern of a crystal creates identical areas known as lattice points. Each lattice point in a perfect crystal will be indistinguishable from another lattice point, and these points continue throughout the crystal in all three dimensions. These lattice points are then used to define a unit cell, which is the smallest repeating unit of a crystal. A unit cell is a parallelepiped designated with sides of lengths a, b, and c and angles of α, β, and γ. Unit cells are measured using these six parameters, and these become the coordinates in which the structure is oriented. Unit cells can be categorized into seven different crystal systems based on their symmetry as shown in Table 1.2 below.

Table 1.2: Crystal systems and their restrictions on lengths and angles

Crystal System	**Restrictions on Dimensions**
Triclinic	None
Monoclinic	α = γ = 90°
Orthorhombic	α = β = γ = 90°
Trigonal	a = b = c α = β = γ **or** a = b α = β = 90° γ = 120°
Tetragonal	a = b α = β = γ = 90°
Hexagonal	a = b α = β = 90° γ = 120°
Cubic	a = b = c α = β = γ = 90°

Using X-ray diffraction to construct a data set allows one to calculate the position of the atoms in a crystal structure. These positions are useful for determining the bond lengths and bond angles, which can lead to an understanding of many other properties of the crystal and the compound itself. Bond lengths can determine the strength of the bonding; the longer a bond is, the weaker the interaction between the two atoms. Bond angles can give insight on the inter- and intramolecular forces acting upon the molecules in the crystal. Additionally, comparing two or more crystal structures can lead to information regarding how the molecules interact with one another within the lattice.

X-Ray Diffraction Studies

X-ray diffraction works by directing a beam of x-ray radiation at a crystal and recording the diffraction pattern generated. X-rays diffract off of the electron clouds of the atoms in the repeating lattice of the crystal. The angle at which the beam is directed at the crystal is changed throughout a single data collection, enabling the detection of the diffraction pattern from multiple angles. All the data collected is then sent to a computer where it is processed to form a three-dimensional map of the electron density throughout the crystal, and from here it is reduced to a repeating unit cell.

This electron density map is then used to find the position of the atoms in the unit cell. The areas with the most electron density may be assumed to represent the locations of the atoms since the electrons have a higher probability of being near the nuclei than anywhere else. Atom identities are assigned in accordance with the measure of electron density recorded. For example, in a metal complex ligands containing only carbon, nitrogen, oxygen, and hydrogen, an area of very high electron density right in the center of map would likely correspond to the metal atom, and from there other peaks could be assigned to the atoms of the ligands. As peaks get assigned to atoms, other peaks of lower electron density will be assigned until there are none left with any significant electron density. This process generates a model of the collected data that can be used to investigate the structural factors of the molecules in the lattice, which is particularly useful for studying catalysts because its effectiveness can be tailored to better suit the needs of the reactants if the structure is thoroughly known.

CHAPTER 2

MATERIALS AND METHODS

Materials

Solvents and Reagents

1. 1,5-Naphthyridine ($C_8H_6N_2$)

   (96%) Used as purchased from ALDRICH.

2. Acetone [$(CH_3)_2CO$]

   (99.9%) Used as purchased from VWR International.

3. Chloroform-D ($CDCl_3$)

   (D, 99.8%) Used as purchased from Cambridge Isotope Laboratories, Inc.

4. Chlorobenzene [$(C_6H_5)Cl$]

   (99.8%) Used as purchased from Fischer Scientific.

5. Dichloromethane (CH_2Cl_2)

   (99.5%) Used as purchased from BDH.

6. Ethanol (CH_3CH_2OH)

   (99.9%) Used as purchased from VWR International.

7. Ethyl Acetate ($CH_3COOCH_2CH_3$)

   (99.5%) Used as purchased from VWR International.

8. Hexanes [$CH_3(CH_2)_4CH_3$]

   (98.5%) Used as purchased from Fisher Scientific.

9. Imidazole ($C_3H_4N_2$)

(99%) Used as purchased from Sigma Aldrich

10. Methanol (CH_3OH)

(99.8%) Used as purchased from VWR International.

11. N-phenylacetamide [$HN(C_6H_5)C_2H_3O$]

(99.9%) Used as purchased from Fischer Scientific.

12. Tetrakis(acetato) Dirhodium(II) [$Rh_2(O_2CCH_3)_4$]

Used as purchased from Pressure Chemical.

13. Tetrakis(N-phenylacetamidato) Dirhodium(II) [$Rh_2(N(C_6H_5)COCH_3)_4$]

Synthesized as described below.

14. Deionized Water (H_2O)

Produced at East Tennessee State University through mixed bed filtration.

15. Coarse Sand

Used as purchased from EMD Chemicals

16. SiliaFlash 230-400 mesh Silica gel

Used as purchased from SiliCycle.

17. Sodium Carbonate, Anhydrous (Na_2CO_3)

Used as purchased from Amresco

Methods

Tetrakis(N-phenylacetamidato) Dirhodium(II) Synthesis

Tetrakis(N-phenylacetamidato) dirhodium(II) was synthesized following the method previously published by the Eagle research group.[15] In preparation for the reaction, all glassware, a stir bar, seven thimbles, coarse sand, and sodium carbonate were dried in the oven at 150 ºC for 24 hours. The seven thimbles were packed with sand and sodium carbonate, each in alternating layers of 1 cm, until the thimbles were approximately three-quarters full with sand as the top layer. The soxhlet apparatus glassware was assembled and allowed to cool under nitrogen (N_2).

Once cooled, 9.92 g ($7.34x10^{-2}$ mols) of acetanilide [$C_6H_5NH(COCH_3)$] and 0.978 g ($2.21x10^{-3}$ mols) of rhodium acetate [$Rh_2(O_2CCH_3)_4$] were added to the round bottom flask. A packed thimble was removed from the oven, placed in the soxhlet chamber, and subsequently cooled under N_2. After cooling, chlorobenzene (C_6H_5Cl) was slowly added to the soxhlet chamber with a long stem funnel. This process was continued until the soxhlet chamber had emptied via the siphon three times resulting in 240 mL of chlorobenzene being added to the round bottom flask.

A tank of N_2 was connected to the soxhlet extractor and the gas outlet, which routed into the fume hood, using a three-way valve. The valve was clamped to the top of the condenser to secure it. The out gas line was connected to a test tube partially filled with mineral oil, which allowed for visual confirmation of gas flow and also provided a one-way valve preventing oxygen from entering the system. The system was flushed with N_2 for 10 minutes before heating, and then the N_2 was set on a low flow rate for the duration of the reaction. The chlorobenzene solution was brought to a low, steady boil and observed for 20 minutes. Once the solvent was cycling through the soxhlet extractor at a rate of at least once per hour, the round bottom flask and soxhlet chamber were wrapped in insulation to promote heat transfer.

Every 24 hours the thimble was changed after allowing 1 hour for the system to cool under N_2. When replacing the thimble, chlorobenzene was added by filtering through the new thimble to ensure that at least 150 mL of solution was in the round bottom flask. After the

addition of chlorobenzene, the system was flushed with N_2 for 10 minutes before heating was resumed. This precaution was necessary to remove oxygen from contact with the hot solution to lower the probability of the formation of decomposed products. The reaction was run continually in this manner for 7 days during which the solution turned dark green, and a green precipitate formed.

After 7 days, the solution was allowed to cool and was tested for completion using thin layer chromatography (TLC). Solutions of rhodium acetate and acetanilide in dichloromethane were spotted on a TLC plate along with the reaction solution and developed in a chamber containing a 50/50 mixture of ethyl acetate and hexanes. The TLC of the product showed acetanilide and two other components. The first component that remained on the baseline was most likely decomposed rhodium. The second component presented a different retention factor than the rhodium acetate. In conjunction with the color change, these results were indicative of product formation.

Rotary evaporation was used to remove chlorobenzene from the product mixture. The product mixture remaining was heated overnight in a vacuum oven at approximately 60.0 ºC to remove any residual chlorobenzene. After removal of chlorobenzene, sublimation was utilized to remove acetanilide over the course of a week, resulting in a green product.

Separation of Tetrakis(N-phenylacetamidato) Dirhodium(II) Isomers

Separation of the product mixture was achieved using flash column chromatography on silica gel. A 20% solution of ethyl acetate in hexanes was initially used after which the percentage of ethyl acetate was increased over time to promote movement down the column as colored bands did not elute. The increase in the polarity of the mobile phase promoted movement down the column by the products. After all colored bands had been collected in separate Erlenmeyer flasks, methanol was used to remove the remaining rhodium compounds from the column so that the remaining rhodium could be reclaimed. Multiple fractions were collected and then tested using TLC. Fractions with the same retention factor were combined. Some fractions however, presented multiple spots and needed additional purification. Fractions of similar composition were combined and chromatographed again.

Synthesis of Dirhodium(II) Complexes with Axial Ligands

Synthesis of the modified complexes with axial ligands was achieved through the following procedure. Tetrakis(N-phenylacetamidato) dirhodium(II) solid and ligand were measured in a 1 to 10 mol ratio. Reactants were dissolved separately in 10 mL of CH_2Cl_2. The ligand solution was then added to the dirhodium(II) solution. After addition of the ligand solution, a color change occurred in all samples. The resulting complexes were identified after X-ray quality crystals were grown.

Crystal Growth

The solutions of $Rh_2L_4X_n$ (where X = axial ligand; n = 1 or 2) were split equally into half dram vials. One drop of acetone was added to each half dram vial. The acetone additive has previously been noted to enhance crystal growth by members of the Eagle research group. The half dram vials were then placed inside labeled glass vials with screw tops and approximately 4 mL of an outer solvent; ethanol, methanol, hexanes, ethyl acetate, toluene, or acetone. The capped vials were left undisturbed at approximately 21° C for up to a month to allow crystals to grow through vapor diffusion/slow evaporation.

X-ray Diffraction

X-ray diffraction (XRD) was performed on three crystals received from Dr. Dinda in addition to the crystals grown as described in the previous procedure. Potential crystals were selected after observation with a microscope. If the material in the vapor diffusion chamber appeared crystalline in shape, it was considered. Identified crystals were removed from vapor diffusion chambers using a small drop of STP oil on the end of a needle. Samples were then placed on a slide and examined under a microscope to determine which would be utilized for data collection. Crystals were selected for further study on the basis of size and lack of obvious faults.

A potential crystal was selected using a microscope and then it was cut using a scalpel so that no dimension exceeded 0.3 mm as the limit for the crystal to remain entirely within the X-ray beam is 0.7mm.

The crystal was then mounted on a MiTeGen MicroMount™ using residual STP™ oil. The MicroMount™ was secured into a magnetic cup which allowed it to be mounted on the goniometer in the Rigaku XtaLab mini™. The crystal was cooled to -50 ºC by a sample cooler to reduce thermal motion and to freeze the mounting oil, thus preventing the sample from moving during data collection.

After centering the crystal and recording its dimensions and color, the crystal was tested for diffraction using 12 initial scans. The scans showed diffraction patterns from various angles. A crystal that diffracted well produced a number of distinct spots on the detector readout, generally in a geometric arrangement. Crystals with poor diffraction were still used to obtain results by increasing the exposure time. This allowed weaker diffraction patterns to be separated from background or random scatter.

If a sufficient number of spots were identified, the Crystal Clear™ software was then tasked with identifying diffraction spots and attempting to predict spots based on the 12 initial scans. Once a prediction had been generated, the prediction was compared to the diffraction pattern collected in the initial scans. If the prediction matched up well with the spots, there was a good chance of obtaining useable results.

The data collected by the diffractometer was initially processed by Crystal Clear™ software, which merged the various diffraction patterns and calculated the unit cell. This data was then fed to either Crystal Structure™ software that is provided with Rigaku instruments or Olex^2, an open source program for the solving of structures from X-ray diffraction. Data processing was done using SHELXS^{23} in both Crystal Structure™ and Olex^2™.

X-ray Solution

Crystal Structure™ and Olex^2™ were used to solve crystal structures. To begin either program, the data were first loaded into the program by opening the hkl.dat file generated by

Crystal Clear™. Once they were loaded, the data were processed using a crystal system and symmetry point group that best matched the calculated unit cell – there could be multiple possible crystal systems and point group suggestions. Often, the two most probable suggested point groups were very similar to each other, with one having more symmetry elements than the other. Because of this, both possibilities were typically examined.

Once a crystal system and symmetry point group were assigned, the structure was solved using either Patterson or direct methods in Crystal Structure™.

Once these programs solved the basic structure, a three-dimensional image with points indicating areas of high electron density appeared. These "peaks" were correlated to an atom manually. These peaks were then labeled and assigned as an atom to form the structure of the compound that best fit the data.

If the crystal was of high quality and diffracted well, the modeled structure would have been close to complete, and it was then refined using a least squares method. Upon refinement, the structure came together better, making bond lengths and angles closer to what they should be. In addition, new peaks appeared which may or may not be part of the structure. These peaks were numbered and given a value that measured the amount of electron density in that peak. Anything with a value under 1 unit did not need to be seriously considered as an atom; values greater than 1, however, were.

Once any high peaks were assigned to new atoms, the next step was to add hydrogen atoms. The electron cloud of a hydrogen atom was too small to noticeably diffract the x-rays, and because of this, did not always show up as a peak. Instead they were added manually. Each hydrogen atom was selected according to the correct type of hydrogen atom to be placed on each atom; for example, methyl hydrogen atoms were placed on terminal carbon atoms, and phenyl hydrogen atoms were selected for carbon atoms in an aromatic ring. The structure was then refined again using the recommended weights, which refined the structure to be more accurate than before.

The non-hydrogen atoms were then refined anisotropically. Previously, they were refined as isotropic, which caused them to move the same amount in the x, y, and z directions. When

anisotropic, the atom was allowed to move in different amount in each direction, as it would in reality. After that, the structure was refined once again.

If the crystal being examined was an excellent specimen and mounted perfectly, this was likely enough to get a structure that correlated very well with the data. The correlation between the data and the proposed structure is quantified by three values: R_1, wR_2, and Goodness of Fit (GooF). The R_1 value is determined by Equation 3 below:

$$R_1 = \frac{\sum\big||F_o| - |F_c|\big|}{\sum|F_o|} \tag{3}$$

where F is the structure factor, which refers to the mathematical description of how the crystal scatters incident x-rays. F_o is then the observed structure factor and F_c is the calculated structure factor. The R_1 then is essentially the measure of the difference between the model and the data, and it should decrease and approach zero as the structure is refined.

wR_2 is the confidence factor, or weighted R factor, and it too should approach zero. Its formula is given below in Equation 4:

$$wR_2 = \sqrt{\frac{\sum\left({F_o}^2 - {F_c}^2\right)^2}{\sum\left({F_o}^2\right)^2}} \tag{4}$$

The GooF is the measure of similarity between the electron density map calculated by the data and the map of the model, and it should approach 1. It is described in Equation 5 below:

$$GooF = \sqrt{\frac{\sum\left({F_o}^2 - {F_c}^2\right)^2}{N_{Ref} - N_{Par}}} \tag{5}$$

where N_{Ref} refers to the number of independent reflections and N_{Par} refers to the number of parameters.

Generally acceptable values of R_1 are under 5%, wR_2 should be under 12%, and GooF should be between 0.9 and 1.2. In a crystal with little disorder, these values can be achieved with

little additional work; this is not always the case, however, and some additional steps may need to be taken to deal with the disorder as described below.

Disorder in Crystal Structures

When a less than perfect crystal was used for diffraction, the crystal lattice was not uniform throughout with some unit cells not matching the others, and giving rise to disorder in the structure. This could have been due to vibrational motion in the molecules, solvent molecules intercalated within the crystal, or anything else that disrupted the otherwise regular pattern of the unit cells of the crystal.

Many of these forms of disorder could be modeled within the software, however, so that the proposed structure and data are made more alike. Commands within both Crystal Structure™ and Olex^2™ were used to correct for disordered crystals. The commands used most frequently in this project were FLAT, SADI, SIMU, and PART.

FLAT, SADI, and SIMU have completely different functions, but were executed in a similar manner. FLAT forced the selected atoms to stay in the same plane, with a certain amount of deviation allowed. If a phenyl ring was showing up irregularly in the model and needed to be flattened to have to correct geometry, this command was used. SADI forced the selected atoms to have similar atomic displacement parameters. If there were two methyl groups in equivalent places on a molecule, for example, but the atomic displacement parameters of one were significantly different than the other, this command was used to fix that. Thirdly, the SIMU command forced selected atoms to have similar bond lengths. This was helpful if parts of the molecule seemed to be unusually stretched out by bringing the bonds closer to the geometrical ideal.

The PART command was more complicated to implement than the previously mentioned commands. What it did was split selected atoms into two or more parts. Each part had a certain probabilistic weight associated with it that indicated how much of the crystal had the selected atoms in one position vs another. This was especially useful when solvent molecules were in the crystal; because they were not bonded to the rest of the molecule, they tended to vibrate and

move more giving rise to large atomic displacement parameters. Using the PART command often helped improve the model to fit the data in highly disordered structures.

Nuclear Magnetic Resonance (NMR) analysis of Compounds

In addition to XRD, samples were analyzed by NMR. NMR tubes were cleaned with acetone and then dried using N_2. After drying, they were placed in the oven for one hour to remove any trace solvent. An NMR tube was filled with approximately 1mL of analyte solution dissolved in either CD_2Cl_2 or $CDCl_3$. The tube was placed in a JOEL AS400 FT-NMR spectrophotometer for data collecion. Data were analyzed using JEOL Delta 5.0.4 NMR data processing software.

Ultraviolet-Visible Spectroscopy

Samples were also analyzed by ultraviolet-visible (UV-Vis) spectroscopy. Glass cuvettes were rinsed out with CH_2Cl_2 to clean them and then allowed to dry. Both cuvettes were then filled with CH_2Cl_2 and placed in the spectrometer to zero out the absorbance caused by the glass and solvent. The first cuvette was left in the spectrometer as a blank, and the analyte solution was pipetted into the second cuvette. The cuvette with the sample was then placed back into the spectrometer and the spectrum was taken from 300nm-800nm. The data were collected and compiled into a spreadsheet and graphed.

CHAPTER 3

RESULTS AND DISCUSSION

The goal of this research was to determine a method of making a substantial amount of imidazole 3,1-tetrakis (N-phenylacetamidato) dirhodium (II), fully characterize it, and probe how it would react with excess imidazole. The 3,1-Imidazole complex was synthesized, and analyzed by proton nuclear magnetic resonance spectroscopy (^{1}H NMR), ultraviolet-visible spectroscopy (UV-vis), and single crystal X-ray diffraction (XRD) to elucidate the structure of the complex. It was found that the imidazole ligand bound only to the "1" side of the 3,1- complex, even in the presence of excess imidazole.

Tetrakis(N-phenylacetamidato) Dirhodium(II) Synthesis

The synthesis of tetrakis(N-phenylacetamidato) dirhodium(II) was performed and generated the product as expected.[15] Rotary evaporation and sublimation were performed next to isolate the product, followed by flash column chromatography to separate the isomers. The recorded mass of the isomers are shown in table 3.1 below.

Table 3.1: Mass of tetrakis(N-phenylacetamidato) dirhodium(II) isomers

Isomer	Mass	% Yield
2,2-*cis*	196.1 mg	54.8%
2,2-*trans*	70.7 mg	19.8%
3,1-	90.7 mg	25.4%

These isomers were analyzed by ^{1}H NMR, UV-vis, and XRD. The Structure of the 3,1-isomer is show below in Figure 3.1.

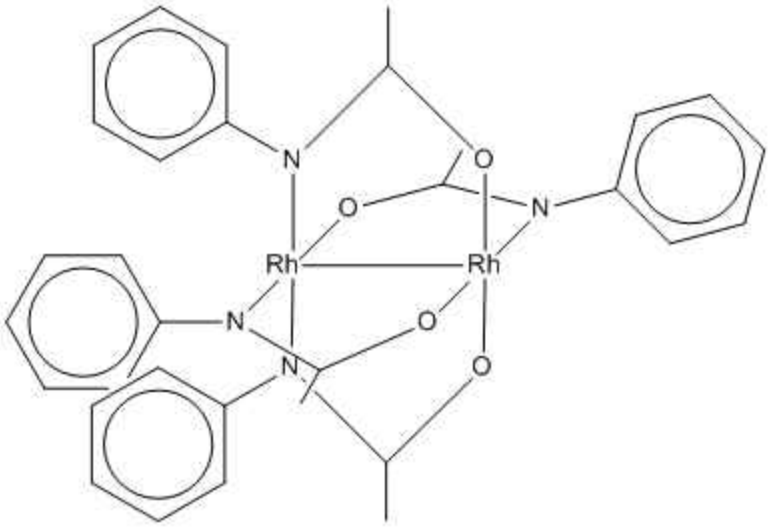


Figure 3.1: Structure of 3,1-tetrakis(N-phenyl)acetamidato dirhodium (II)

^{1}H NMR Study of tetrakis(N-phenylacetamidato) dirhodium(II) Isomers

^{1}H NMR spectra were taken for each isomer, and they can be seen below in Figures 3.2, 3.3, and 3.4. The spectrometer used was a 400 MHz model manufactured by Jeol. The compounds were dried in a vacuum oven at 50-60°C and 44 Torr to remove any residual solvents, and then dissolved in $CDCl_3$. The residual proton in $CHCl_3$ shows up as a peak at 7.260 ppm in the first two spectra, and a peak at 5.320 is present for the third due to the residual CH_2Cl_2 from the use of CD_2Cl_2 as a solvent. Both the 2,2-*cis* and 2,2-*trans* isomers have three groups of peaks in the phenyl region between 6.5-7.5 ppm, while the 3,1- isomer has seven. Similarly, the peaks corresponding to the methyl group protons in 2,2-*cis* and 2,2-*trans* can be seen as one singlet at 1.872 ppm (d) for 2,2-*cis* and 1.874 ppm (d) for 2,2-*trans*, but three singlets appear in the spectrum for the 3,1-complex at 1.942 ppm (j), 1.865 ppm (k), and 1.795 ppm (l).

In the spectrum of 2,2-*cis* (see Figure 3.2), the peaks corresponding to the phenyl protons appear between 7.00 and 7.32. These are not singlets due to the splitting from protons on the neighboring carbon atoms within the phenyl rings. The middle peak is seen with a shift of 7.153 ppm (b) and can be attributed to the proton on the carbon that is para- to the nitrogen atom of the phenylacetamide ligand since it has a relative integration of one. It is a triplet due to the splitting of two neighboring protons. Peak (a) at 7.299 ppm is also a triplet, making it best fit the protons

that are meta- to the nitrogen atom. Peak (c) at 7.017 ppm is a doublet since it corresponds to the protons para- to the nitrogen atom, which only have one neighboring proton.

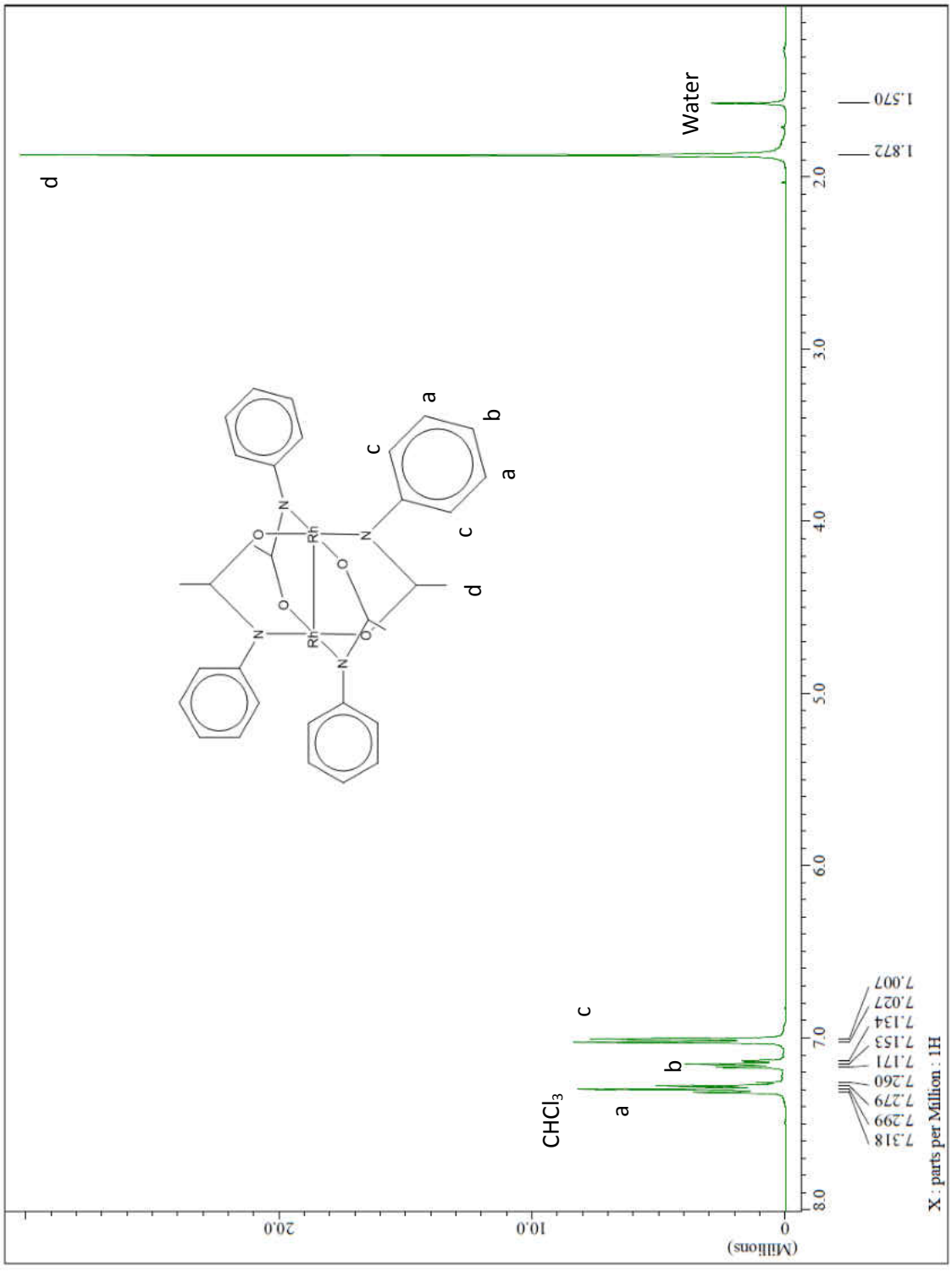


Figure 3.2: ^{1}H NMR spectrum of 2,2-*cis* isomer

2,2-*trans* (see Figure 3.3) follows the same splitting pattern as 2,2-*cis*, but the shifts are slightly different. Peak (a) occurs at 7.223 ppm, peak (b) occurs at 7.116 ppm, and peak (c) occurs at 6.950 ppm.

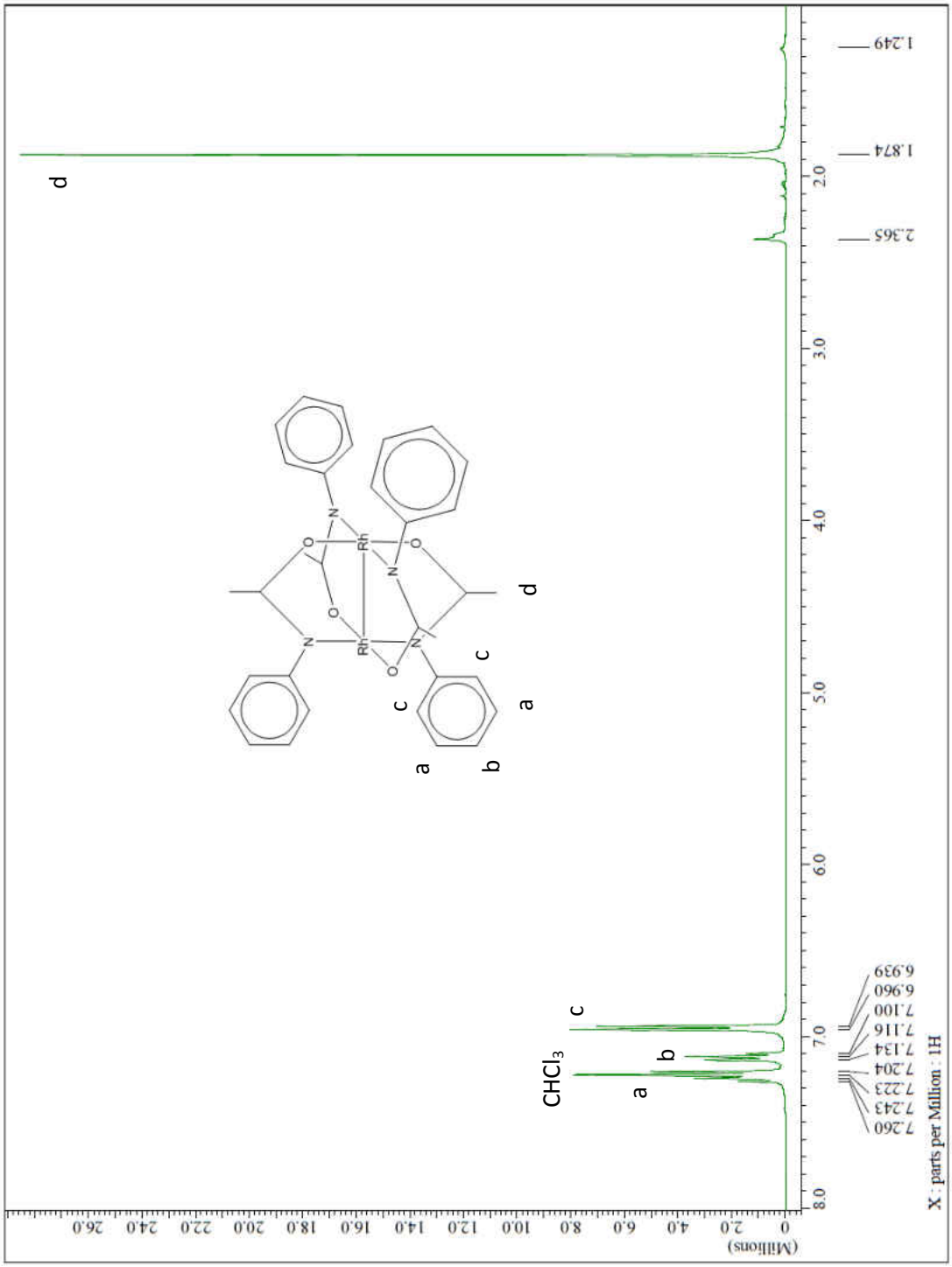


Figure 3.3: ^{1}H NMR spectrum of 2,2-*trans* isomer

For the 3,1-isomer (see Figure 3.4), the splitting is more complex due to its unsymmetrical nature. This also leads to an overlap of some of the peaks in the phenyl region with only seven distinct groups of peaks being visible instead of the expected nine.

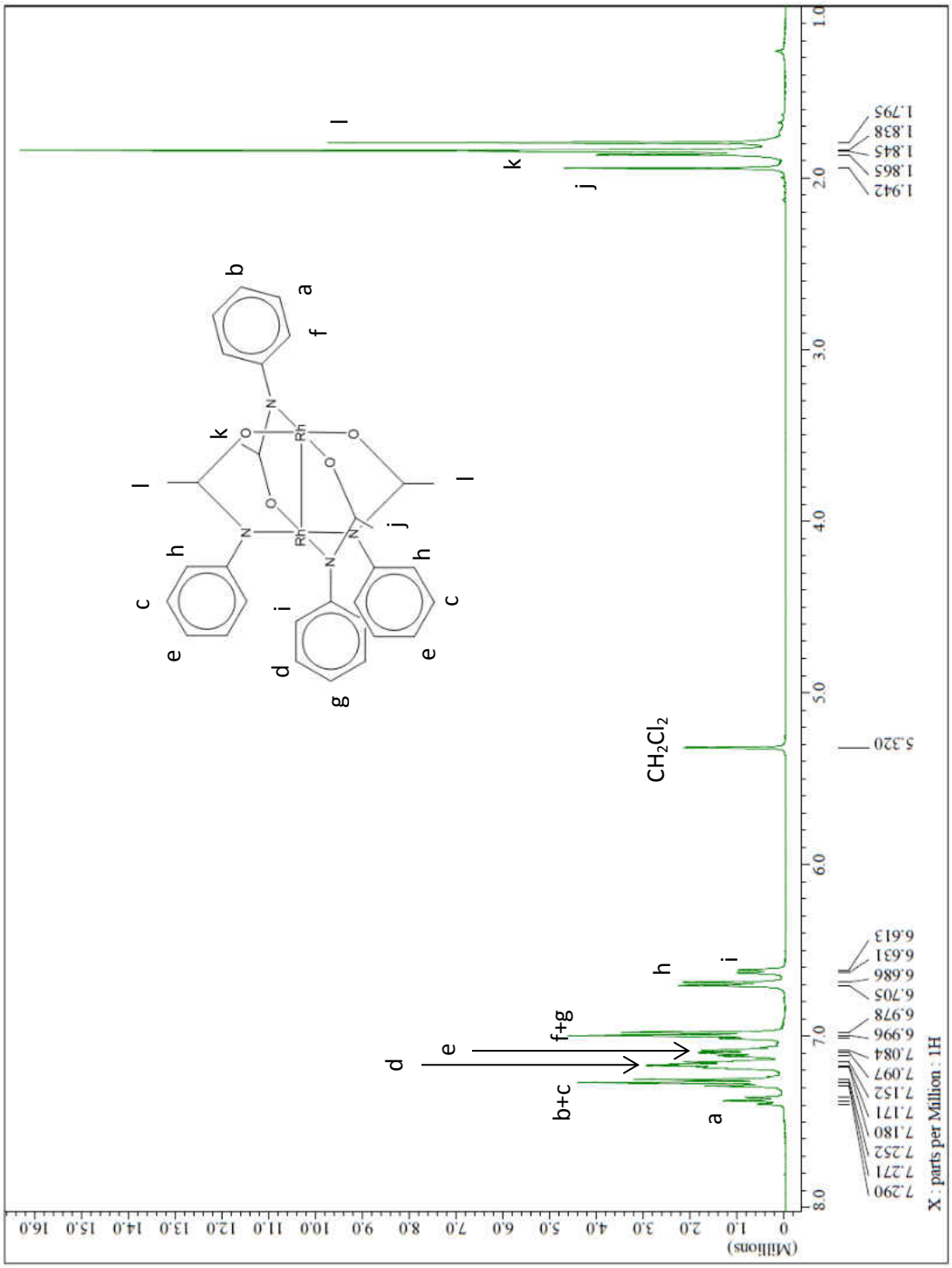


Figure 3.4: ^{1}H NMR spectrum of 3,1- isomer

3,1-[$Rh_2\{N(C_6H_5)COCH_3\}_4(N_2C_3H_4)$]

Imidazole was added to the green dichloromethane solution of 3,1- in a 1:1 mole ratio and produced a purple solution. This solution was analyzed by NMR and showed evidence that the imidazole group had coordinated with the 3,1- complex. The chemical shifts of the protons of uncoordinated imidazole were a singlet at 10.7 ppm for the proton attached to the nitrogen atom, a singlet at 7.7 ppm for the single proton on the carbon between the nitrogen atoms, and a doublet at 7.1 ppm for the two protons on the two chemically similar carbon atoms. After reaction with the 3,1- complex, the shift of the first imidazole proton became 10.3 ppm, the second 7.3 ppm, and the third 6.7 ppm. The relative integrations of the imidazole peaks compared to those corresponding to the 3,1-complex's peaks indicate that the imidazole was coordinated in a 1:1 ratio in solution. These spectra can be seen in Figures 3.5 and 3.6 below.

Crystals of the 3,1-Imidazole complex were grown through vapor diffusion from dichloromethane and ethanol. The crystals grown were red in color. One crystal was selected and used for data collection. Crystallographic data was collected at -50 °C using a Rigaku Xtalab Mini™ with sampler chiller.[21] The structure was solved in Crystal Structure™ and Olex[2] using direct method with SHELX97.[22,23] The crystal was solved with a triclinic crystal system and a P-1 space group. Further crystallographic data is shown in Table 3.2 below.

Table 3.2: Crystallographic data of 3,1-[$Rh_2\{N(C_6H_5)COCH_3\}_4(N_2C_3H_4)$]

Crystal System	Triclinic
Space Group	P-1
Unit cell Lengths	a: 11.086
	b: 13.310
	c: 13.958
Unit Cell Angles	α: 93.300
	β: 110.255
	γ: 90.488
R_1	3.97%
wR_2	9.27%
Goodness of Fit	1.036

The goal of crystallography is to find the structure of a compound that best fits the data gathered by the diffractometer. In the case of the 3,1-Imidazole complex, the results were an R_1 of 3.97%, a wR_2 of 9.27%, and a GooF of 1.036. From this, it was concluded that the proposed model accurately represented the collected data and that the 3,1- isomer with a mono-adduct of imidazole was successfully generated from the synthetic protocol described above.

A synthesis of the 3,1-complex with imidazole in both axial sites was also attempted. A solution with a 10:1 ratio of imidazole to 3,1-imidazole monoadduct was made and crystals were grown from this solution using vapor diffusion from dichloromethane and ethanol. The crystals that were grown from this solution were then sorted through to find a suitable crystal for single crystal XRD. None of the crystals were able to yield a successful diffraction however, so no crystal structure was generated from this product.

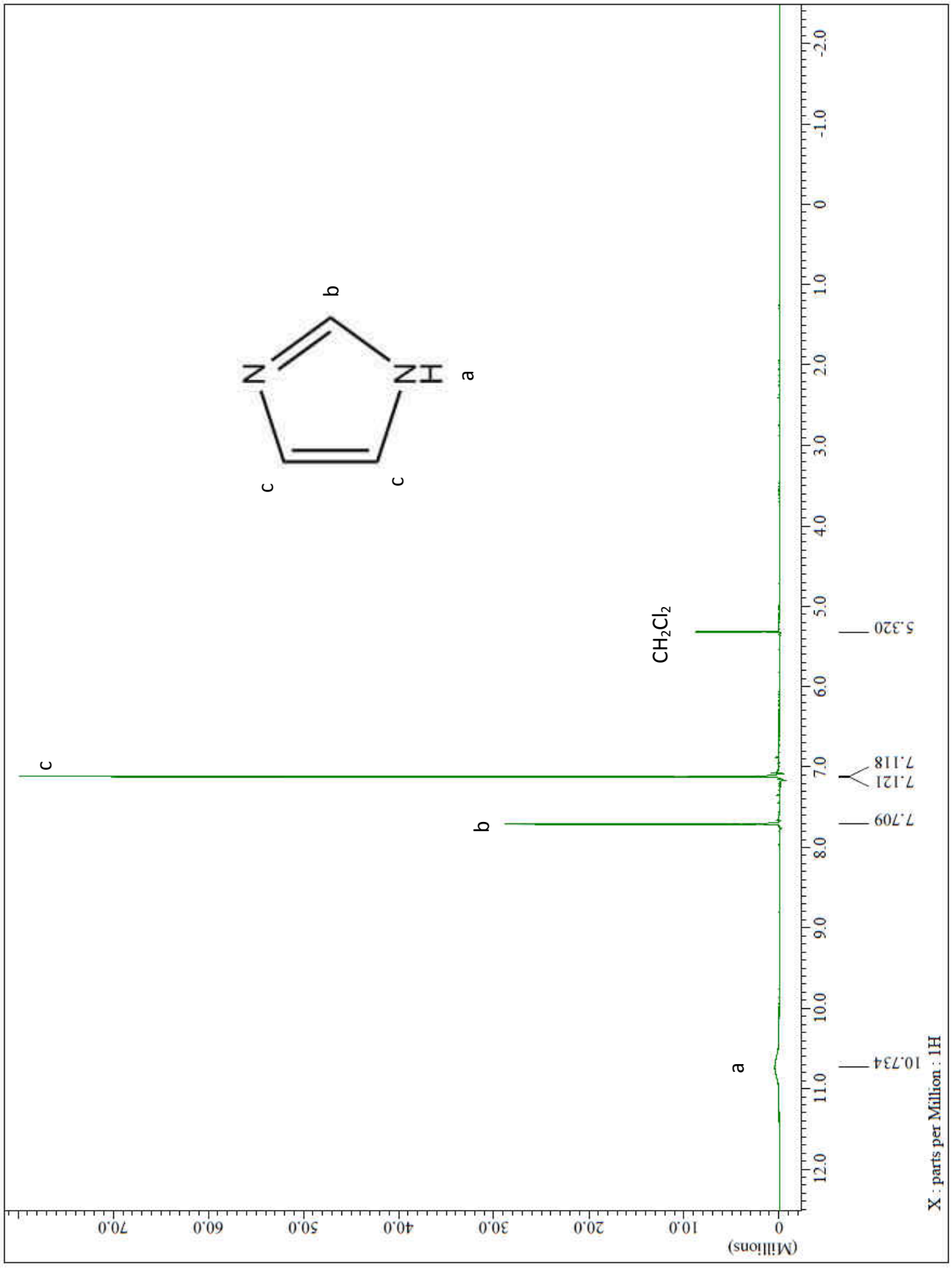


Figure 3.5: ^{1}H NMR spectrum of imidazole

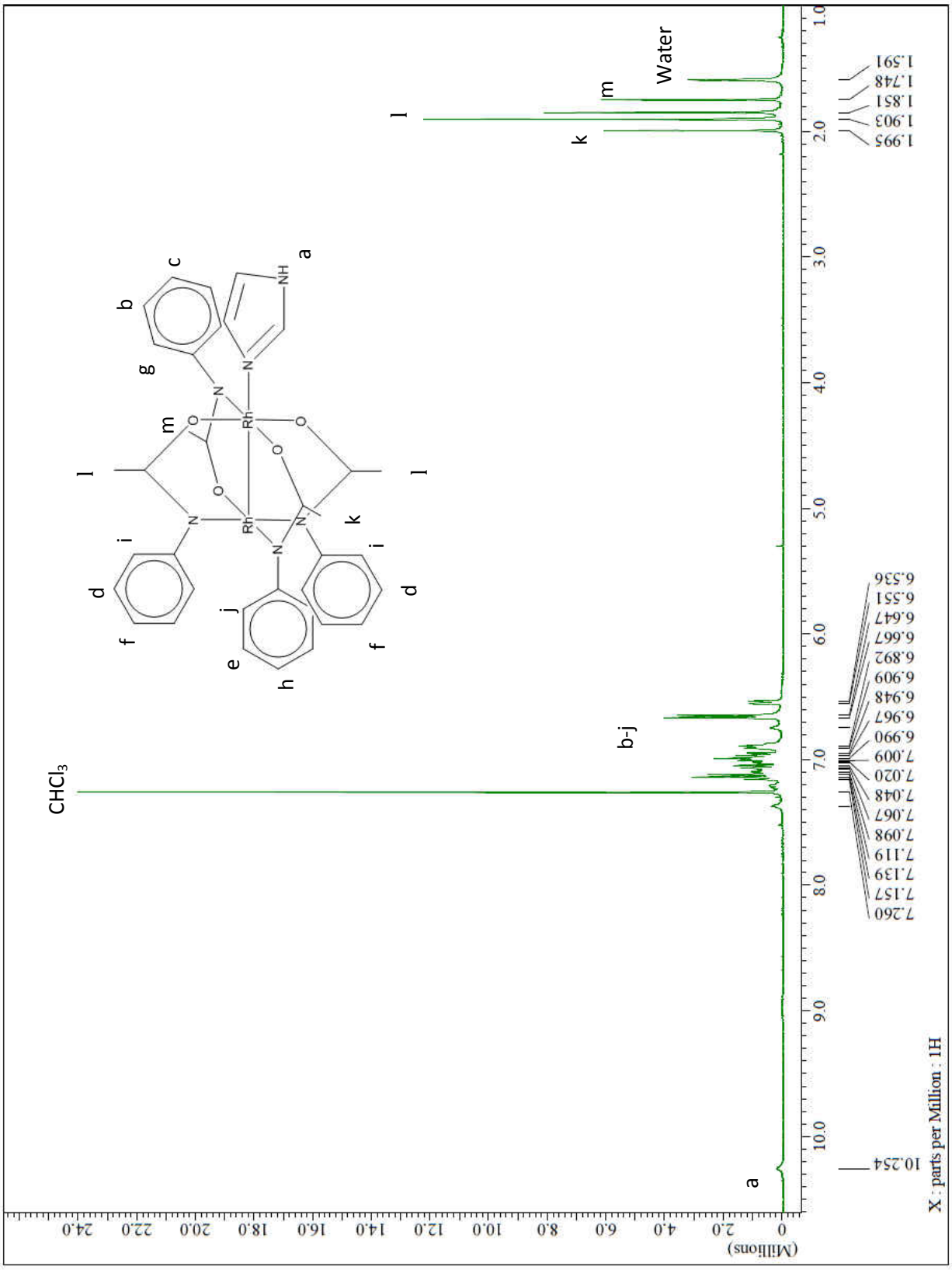


Figure 3.6: ^{1}H NMR spectrum of 3,1-imidazole complex

One of the ways that a solved crystal structure is visually represented is by using an Oak Ridge Thermal Ellipsoid Plot (ORTEP). The plot shows the anisotropic displacement of each atom in the structure due to thermal motion. Any data collected will have some movement inherent with the vibration of the electrons in the atoms of the compound. The greater the variation of the center of electron density, the larger these ellipsoids will be. The ORTEP is used to visually check for consistency of movement. The shape of a given atom provides a visual check of the structure. Adjacent atoms should have similar displacements and orientations. The exception to this rule is if the adjacent atom has more or less freedom than its neighbor. The largest displacement should be oriented perpendicular to a bond; and the more bonds an atom has, the smaller the ellipsoid should be. Figure 3.7 shows the ORTEP generated for the 3,1-Imidazole complex, and it can be seen to have little irregular thermal motion. The ellipsoids that are noticeably irregular are predominantly on the outer carbon atoms of the phenyl rings, where the structure allows greater freedom of motion.

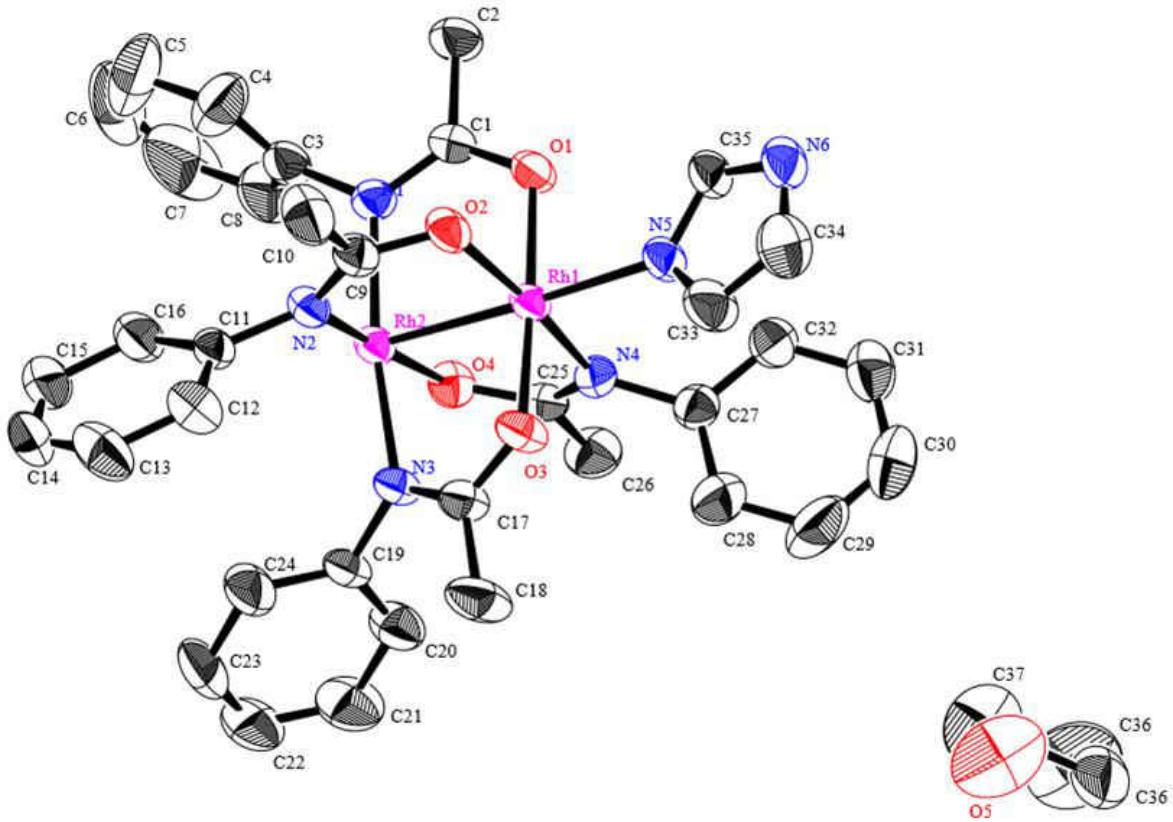


Figure 3.7: ORTEP of 3,1-[$Rh_2\{N(C_6H_5)COCH_3\}_4(N_2C_3H_4)$] with 50% probability ellipsoids

Addition of Excess Imidazole to 3,1-[$Rh_2\{N(C_6H_5)COCH_3\}_4(N_2C_3H_4)$]

The purpose of adding imidazole to the 3,1- complex was to have it coordinate to the "1" side, leaving the "3" side open and available for catalysis. If, however, imidazole formed a diadduct and coordinated to both axial sites of the complex, then the catalysis would not occur since at least one axial coordination site of the rhodium complex must be open in order for the carbenoid to be generated and stabilized. To probe the possible existence of a di-imidazole adduct, the 3,1-imidazole complex was reacted with excess imidazole and analyzed by UV-visible spectroscopy to observe any electronic changes.

A solution of a 1:1 ratio of the 3,1-complex and imidazole was analyzed by UV-vis with subsequent scans run after an addition of an imidazole solution. Each aliquot contained one mole equivalent of imidazole, which after only a few aliquots would result in changes to the UV-vis spectrum if it were to form a diadduct. Previous studies on rhodium carboxamidate complexes have shown that the UV-vis spectrum of one of these compounds changes with each axial ligand that coordinates to the complex.[24,25] An absorbance peak at a given wavelength will diminish and another will appear when the first axial ligand is coordinated and the same will occur again if a second ligand is added. The UV-vis spectrum obtained can be seen in Figure 3.8 below.

It shows that no new peaks appeared and no current peaks disappeared, indicating no changes to the structure of the 3,1-imidazole complex. The only change with each addition was that the absorbance went down slightly with each consecutive addition. This was likely due to the fact that by adding the imidazole solution each time, the concentration of the 3,1-Imidazole complex decreased, making the solution more dilute. Other than that, however, the spectrum remained constant throughout; there was no change in the λ_{max} peak position during the duration of the experiment. This confirmed that the desired outcome was achieved; the 3,1-imidazole complex only formed the mono-adduct. Even in the presence of excess imidazole, a di-imidazole adduct was not formed. Thus leaving the more sterically hindered axial site on the "3" side available for catalysis.

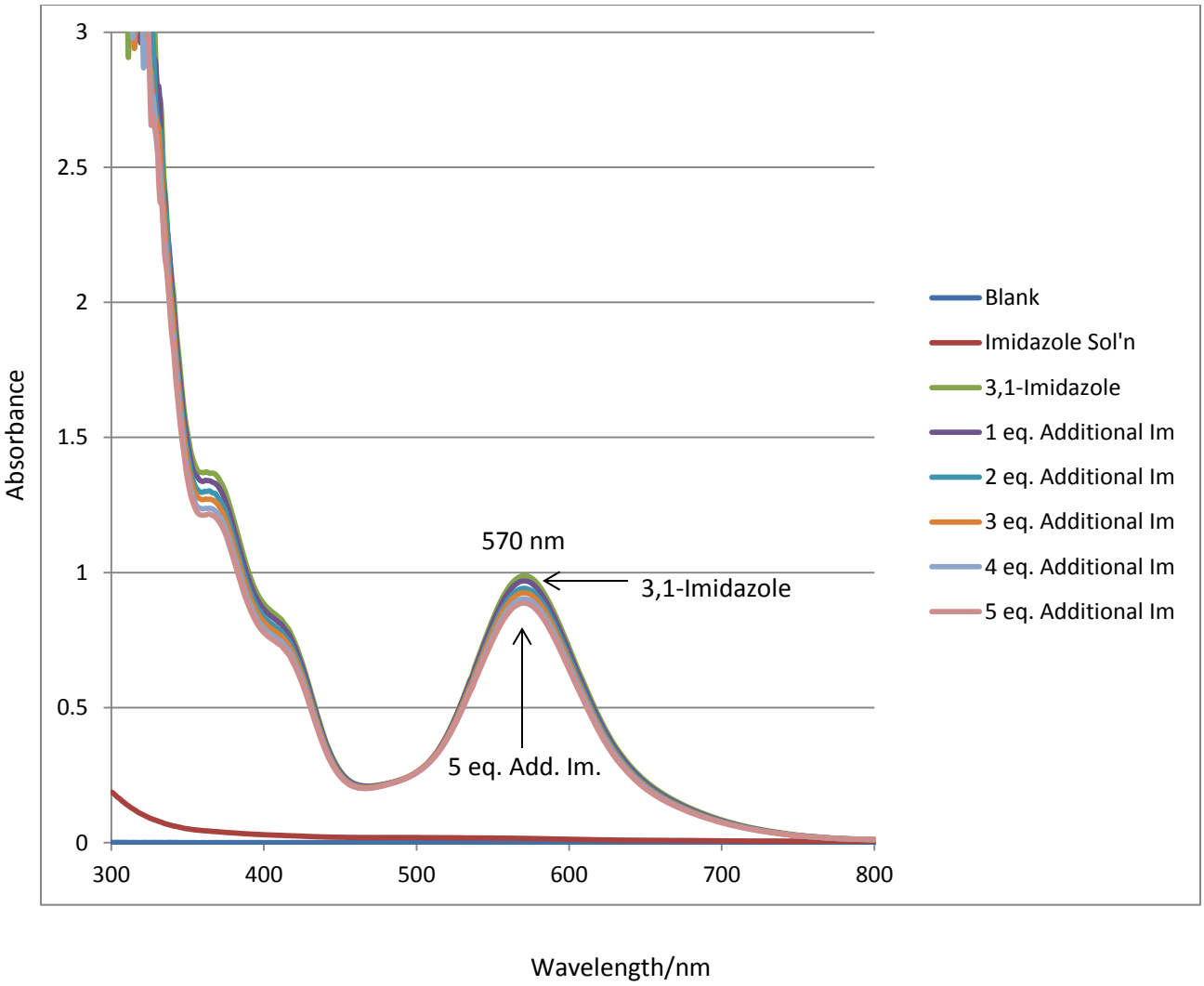


Figure 3.8: UV-Vis spectrum of 3,1-imidazole with consecutive additions of one molar equivalent of imidazole

<u>$MoSO_6N_2C_{21}H_{21}$ (Mo-1)</u>

Three complexes were sent to the Eagle research group to be analyzed by X-ray diffraction. These complexes, denoted as Mo-1, Mo-2, and Cu-1, were synthesized and crystallized by Dr. Rupam Dinda's research group.[26] The proposed structure of Mo-1 can be seen in Figure 3.9.

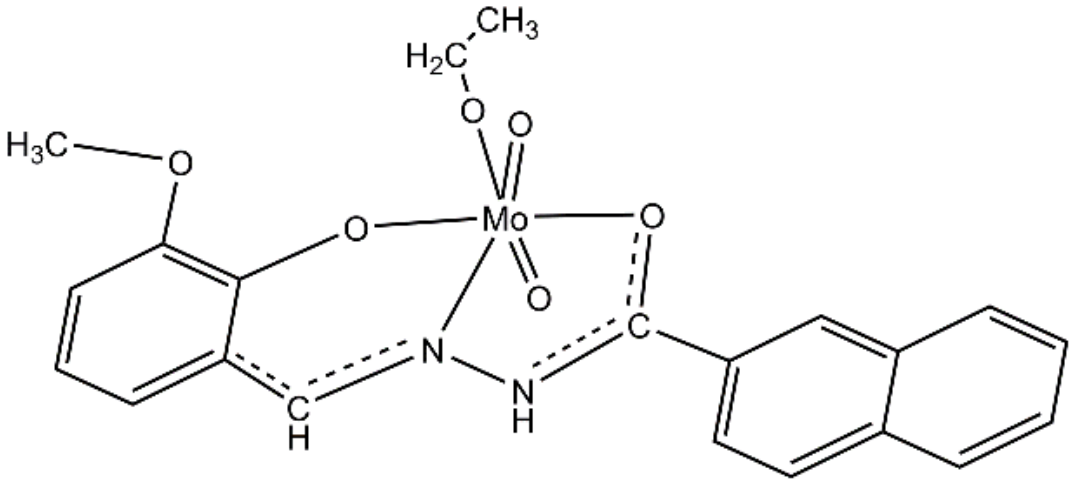


Figure 3.9: Proposed structure of Mo-1

The crystal structure was solved in a monoclinic crystal system with a P21/c space group using SHELX97. Shown below in Figure 3.10 is an Oak Ridge Thermal Ellipsoid Plot (ORTEP) of Mo-1.

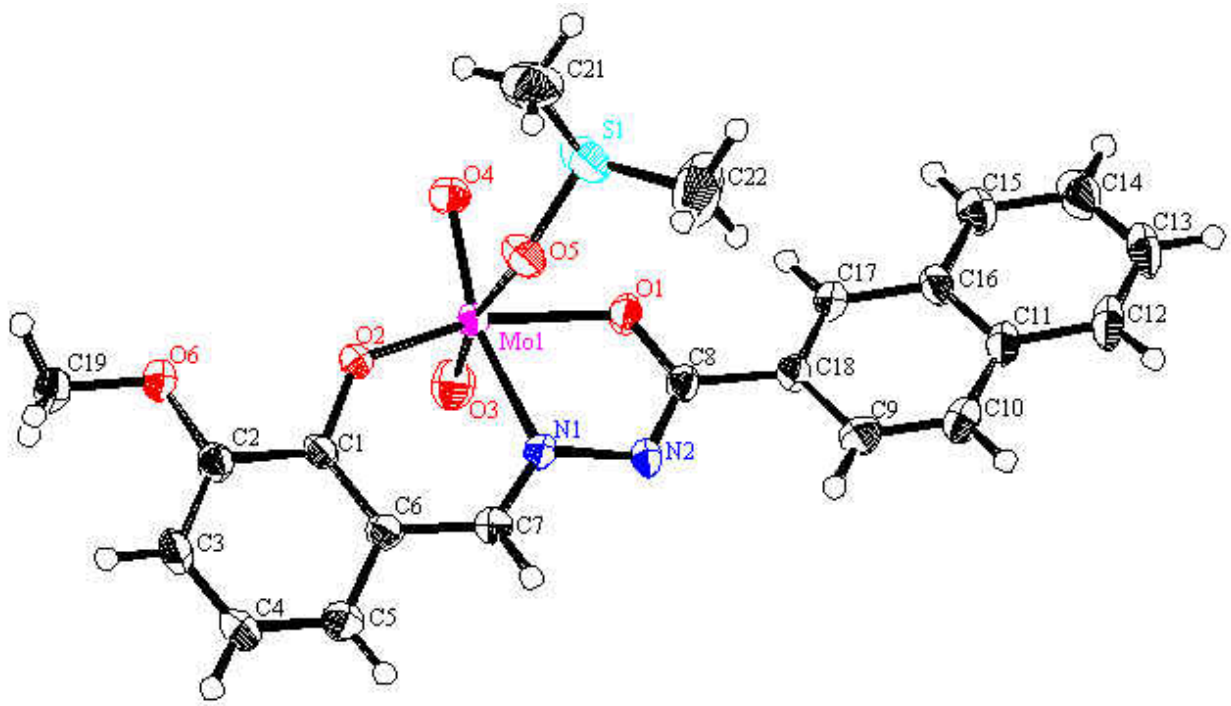


Figure 3.10: ORTEP of Mo-1 with 50% probability ellipsoids

The ORTEP of Mo-1 shows fairly consistent atomic displacement parameters (ADP) throughout all of the atoms within the conjugated pi system. The largest ellipsoids occur on methyl carbon atoms that are only attached through one bond, which is in accordance with what would be expected, since the methyl groups have much more freedom of motion being bonded to only one other atom. Consistent size of thermal ellipsoids is a good indicator that the model and data agree with each other.

One difference between the proposed structure in Figure 3.9 and the solved structure shown in the ORTEP in Figure 3.10 is that Dr. Dinda assumed that there would be an ethanol group coordinated to the molybdenum center, but the better solve was found to have a DMSO group there instead. When initially solving the structure, the model included coordinated ethanol, but there was a high peak of electron density attached to the carbon α to the hydroxyl group. In addition, the ellipsoids were stretched out to try to accommodate for this spread of electron density. Along with the crystals and proposed structures, Dr. Dinda also supplied some of the reagents and solvents used in the synthesis and crystallization of the compounds, and one of the solvents used was DMSO. DMSO looked to be a better fit for data, so it was placed there instead of the ethanol. An ORTEP of the structure as it was originally solved with ethanol is shown below in Figure 3.11.

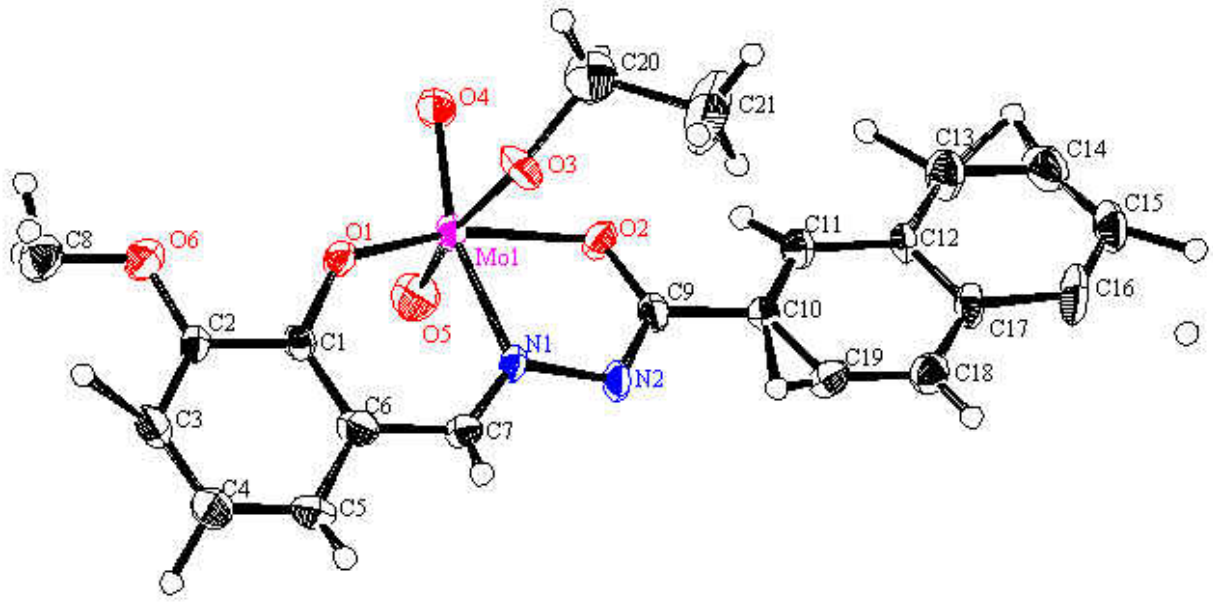


Figure 3.11: ORTEP of Mo-1 with ethanol with 50% probability ellipsoids

The refinement values, measures used to determine how well the proposed solution fit the data, can be seen in Table 3.3 below.

Table 3.3: Refinement Indicators of Mo-1

With Ethanol	
R_1	13.28%
wR_2	44.44%
GooF	1.903

With DMSO	
R_1	4.27%
wR_2	9.15%
GooF	1.074

In addition to the refinement indicators, another reason for concern with the Mo-1-Ethanol complex was the appearance of an electron density peak of 5.42, which was far too much electron density to leave unaccounted. The peak was located a short distance from the secondary carbon atom of the proposed ethanol ligand. Based on these values and the ORTEP generated, there is good evidence that the proposed structure was not quite the same as the structure determined experimentally by the diffractometer. The majority of the structure was indeed correct, but the coordination of DMSO instead of ethanol to the molybdenum center gave consistently lower refinement indicators.

$CuCl_2O_2N_2C_{26}H_{36}$ (Cu-1)

Cu-1 was provided by Dr. Rupam Dinda in the same manner as Mo-1 above. The proposed structure of the compound can be seen in Figure 3.12 below.

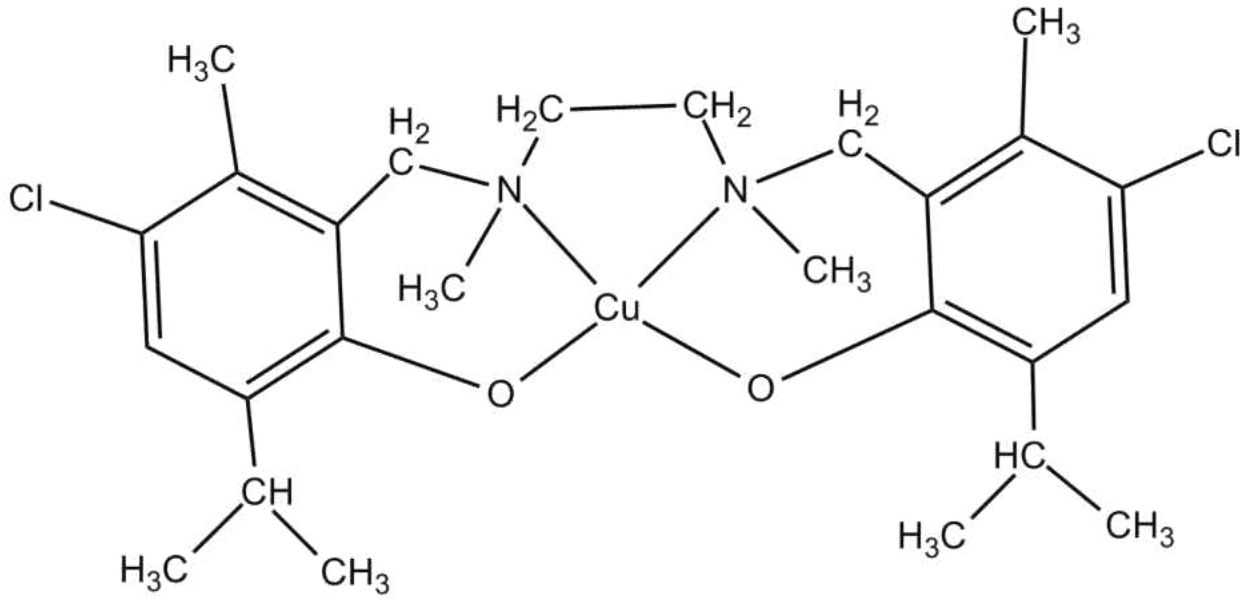


Figure 3.12: Proposed structure of Cu-1

This complex was solved in a monoclinic crystal system with a P21/c space group using SHELX97. Figure 3.13 shows the ORTEP generated for this complex.

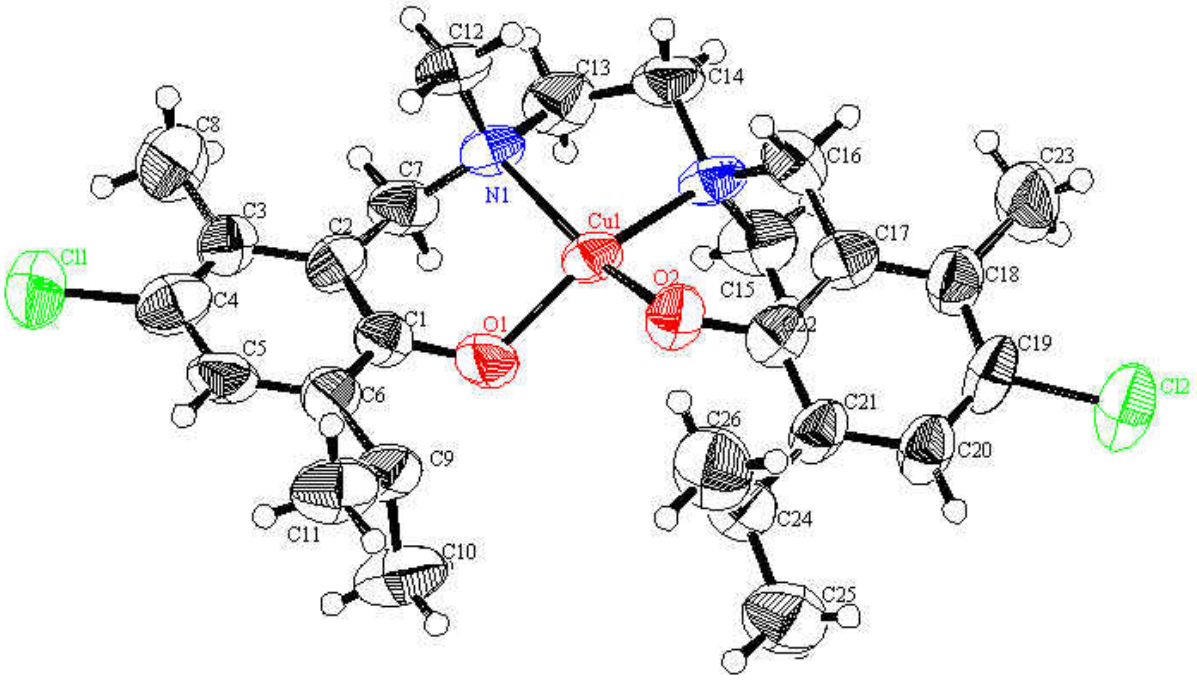


Figure 3.13: ORTEP of Cu-1 with 50% probability ellipsoids

The thermal ellipsoids in this complex were not as uniform as the ones in Mo-1. The isopropyl groups especially were significantly larger than the ellipsoids of the atoms in the majority of the compound. The larger number of groups only attached to one other atom in the molecule introduces more variation in the structure due to rotational motion in addition to the normal vibration of the atoms. This in turn led to refinement values that were not as good as the ones for Mo-1. To help minimize the additional motion, this crystal underwent another round of diffraction, but this time at -50°C. This moderately decreased the size of the ellipsoids and improved the refinement indicators, but the resulting values were still not within the range considered good and barely outside acceptable ranges. Table 3.4 below lists the refinement indicators of Cu-1.

Table 3.4: Refinement Indicators of Cu-1

R_1	10.10%
wR_2	22.60%
GooF	0.991

Additional refinement of the structure did not improve the values any further at this point, so the next step attempted was to model the disorder. Disorder can be minimized by using certain restraints and constraints. Restraints and constraints are similar in that they restrict certain parameters of the structure (such as bond lengths, bond angles, or ADP), but they differ in that constraints set the value equal to a set amount, whereas restraints keep the value within a certain range of the set amount. For example, a bond length can be constrained to be exactly 1.7 Å, but a restraint will keep it within ±0.1 Å of 1.7 Å.

After using the restraints SADI, SIMU, and FLAT, the refinement indicators did not change significantly. The PART command was also used to model the disorder in the large ADP of the isopropyl groups. Upon splitting up the isopropyl groups into two parts and refining the structure, however, the model fell apart and did not resemble reality. Because of this, no better values for the refinement indicators were able to be obtained for this complex. The .ins files showing the work done with the restraints can be found in Appendix F.

$\underline{MoClS_2O_4N_3C_{18}H_{18}}$ (Mo-2)

The third sample sent from Dr. Dinda to be characterized by XRD was labelled Mo-2. Its proposed structure can be seen in Figure 3.14 below.

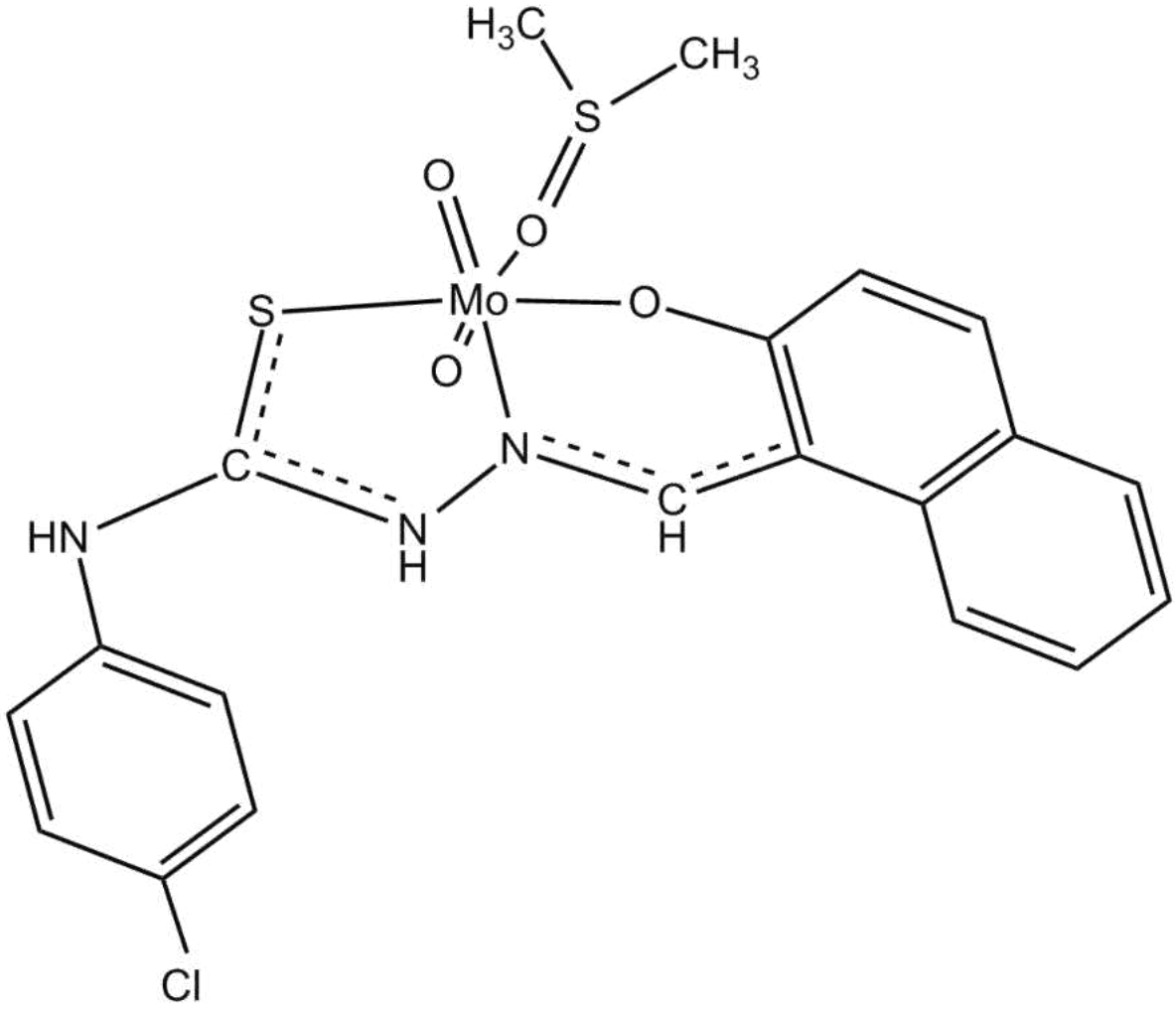


Figure 3.14: Proposed structure of Mo-2

The sample of this compound that was received had very small crystals; the largest one found was only about 0.05 mm in two dimensions and about 0.2 mm in the third. Because of this, getting a successful collection of data from the diffraction was difficult and took multiple attempts with the final one having an increased exposure time of 120 seconds, instead of the usual 10 seconds. When the data collection was finally successful, it solved with a monoclinic

crystal system with a C2/c space group using SHELXL97. The ORTEP generated can be seen in Figure 3.15 below.

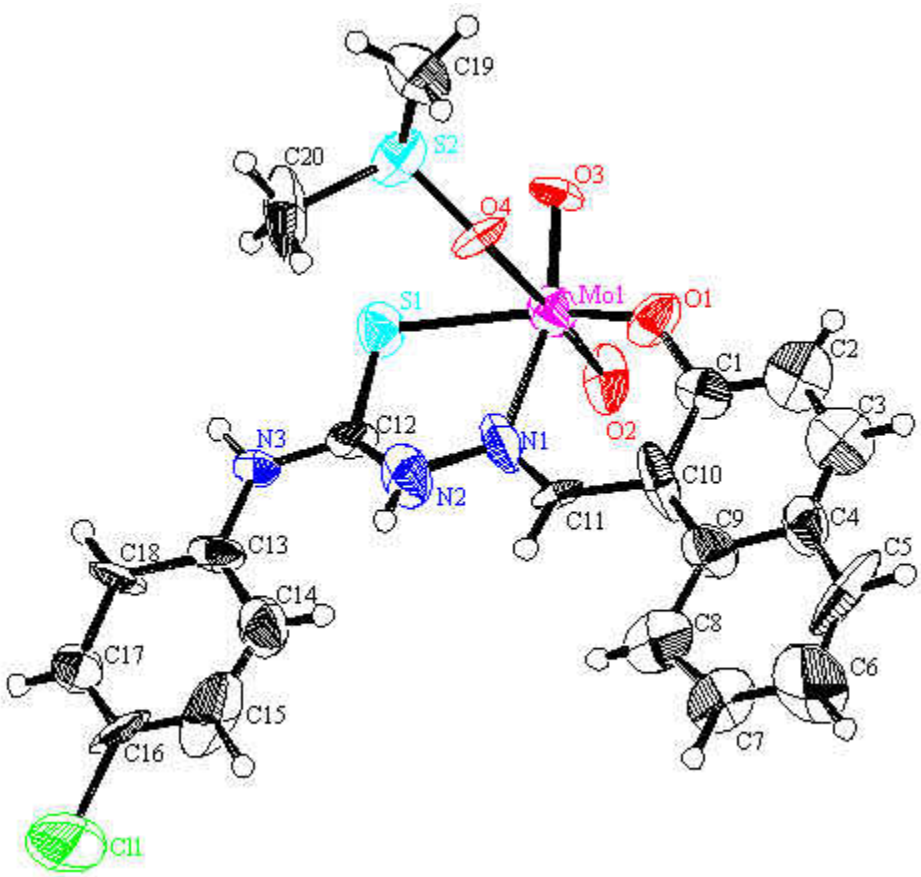


Figure 3.15: ORTEP of Mo-2 with 50% probability ellipsoids

From this ORTEP, it is apparent that the ellipsoids are significantly larger than the ones in other samples collected. In addition to their increased size, the ellipsoids are also not very regular in their shape. Typically, they should have similar shapes and sizes, but in this case they are not. The refinement indicators similarly do not indicate a very good match between the model and the data. The same restraints, constraints, and other commands that were used previously with Cu-1 were attempted with this complex as well, but like before, did not improve the fit of the model to the data. Table 3.5 below shows the best refinement indicators obtained for this complex.

Table 3.5: Refinement Indicators of Mo-2

R_1	17.75%
wR_2	46.08%
GooF	0.954

Ideally, the crystals could have been regrown in a different solvent system, but this would have required a larger sample than what was provided. The structure generated does match what was proposed and appears to be stable enough that refinement of the structure does not lead to errors appearing. With a crystal of higher quality, there could have been less disorder in the lattice, which would likely lead to a solved structure that had better ADPs and refinement indicators.

CHAPTER 4

CONCLUSION

This research had two goals; the first was to make a large batch of the 3,1-imidazole complex, characterize it and find out how it reacts with excess imidazole; the second was to elucidate the crystal structure of three model copper and molybdenum cofactors and either confirm or propose a new structure for the crystalline compounds.

A small amount of the 3,1-imidazole complex was generated – approximately 75 mg. A larger amount like this will be necessary for further studies to use the complex as a catalyst. Additionally, experiments were carried out that ensured the imidazole would only coordinate to one axial site of the complex, leaving the other site available for catalysis including a full characterization using UV-vis and NMR. Crystals of the 3,1-imidazole complex with excess imidazole were grown, but the crystals were not of high enough quality to successfully carry out a single crystal XRD experiment.

The results of NMR spectroscopy on the 3,1-imidazole complex showed a shift in peaks compared to both the pure 3,1- isomer and imidazole. This shift in peaks indicates that the imidazole is indeed coordinated to the 3,1-isomer in the axial site. It did not, however, discriminate between the two possibilities of imidazole coordination to one axial site or to both.

The intended result was to have the imidazole coordinate only to the less sterically hindered “1” side, which should be easier for the imidazole to access, thereby leaving only the “3” side of the complex available for catalytic activity. To find out if only the mono-adduct was formed, UV-vis spectroscopy was performed on the complex with spectra being taken after successive additions of imidazole. With a large excess of imidazole in solution, it is theoretically possible that the excess imidazole would begin to coordinate to the “3” side as well as the “1,” effectively neutralizing any catalytic capabilities of the complex. The UV-vis spectra showed no new peaks forming or peaks that were initially present disappearing, indicating that no new coordination was occurring. These results showed that the complex was indeed only a mono-adduct, even in the presence of a molar excess of 10:1 (imidazole to 3,1-complex). This was most likely due to the “3” side being too sterically hindering for the addition of an imidazole

ligand at that site. The fact that no ligands were found on the "3" side indicates that the three phenyls rings result in a significant barrier to interaction between the rhodium center and imidazole ligand. However, 3,1-isomer di-adducts made with nitriles that are comparatively long and narrow have been developed in previous studies in the Eagle research group, which has shown that it is possible to fit something into the sterically hindered axial site.

The complex was then also crystallized so that its crystal structure could be determined. The crystal structure matched what was anticipated with an imidazole molecule coordinated to the "1" side of the complex. The 3,1-imidazole complex had been previously analyzed by other members of the research group, but the results obtained with this crystal were slightly different than the previous one. Before, the crystal structure was modeled with a monoclinic crystal system, whereas the structure obtained from this crystal was best modeled with a triclinic crystal system. One difference with the crystal structure was that the previously studied crystal had only the complex in the crystal lattice, but the crystal analyzed in this study also contained a disordered ethanol molecule within the lattice. Ethanol was used as a solvent in the crystallization process in both cases, but depending on how long the crystal was allowed to sit in the vial the ethanol could have had time to evaporate more and be less likely to end up in the crystal itself. The addition of a solvent molecule like ethanol to the lattice apparently changed the interaction of the molecules in the lattice, thus the crystal system, so it was not unexpected to see a difference such as one crystal being monoclinic and the other triclinic.

Future research goals are to use the 3,1-imidazole complex as a catalyst for cyclopropanations to see if forcing the catalysis to occur at the sterically hindered "3" side will affect the *cis*/*trans* selectivity. In order for this this to work, however, the imidazole will have to remain coordinated to the "1" side in reaction conditions, and the carbene precursor will have to be small enough to reach the "3" axial site despite the steric hindrance. To monitor if the imidazole is staying coordinated to the complex, the color of the solution should be observed during the reaction. The 3,1-imidazole complex is purple, but the 3,1-complex without an axial ligand is green, so any noticeable color change from purple to green would indicate that the imidazole ligand is being displaced by the carbene precursor. Additionally, the carbene precursor is a diazo compound, and upon reaction with the catalyst, nitrogen gas is given off, so the solution should be monitored for gas evolution to ensure that the reaction is indeed taking place.

With regards to the crystallographic study of the model copper cofactor and model molybdenum cofactors, the structures of two were confirmed, and one was slightly different from the proposed structure. Mo-1 was proposed to have ethanol as one of its ligands, but DMSO was found to be a better fit to the crystal data. Cu-1 and, Mo-2, while not solved well enough to publish the crystal structure, matched the diffraction data well enough to conclude that the proposed structure was indeed correct. Additional steps taken to deal with the disorder in the crystal lattice helped in some cases, but still did not bring the suggested structure close enough to match the data perfectly. If more crystals could have been grown so that a higher quality sample could have been taken, better results may have been achieved, but since the sample size was very limited, this was precluded.

The results from these crystal studies will enable further research with these model cofactors. By having a better understanding of the structures of these compounds, those using them can get a better idea of how they may work in a biological system.

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APPENDICES

Appendix A: Supplementary Information for Imidazolo3,1-tetrakis(N-phenylacetamidato)dirhodium(II)

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{37}H_{37}N_6O_5Rh_2$
Formula Weight	851.55
Crystal Color, Habit	red, chunk
Crystal Dimensions	0.320 X 0.140 X 0.110 mm
Crystal System	triclinic
Lattice Type	Primitive
Lattice Parameters	a = 11.086(2) Å
	b = 13.310(3) Å
	c = 13.958(3) Å
	α = 93.300(7) ^{o}

	β = 110.255(8) ^{o}
	γ = 90.488(6) ^{o}
	V = 1928.1(7) Å^{3}
Space Group	P-1 (#2)
Z value	2
D_{calc}	1.467 g/cm^{3}
F_{000}	862.00
μ(MoKα)	9.014 cm^{-1}

B. Intensity Measurements

Diffractometer	XtaLAB mini
Radiation	MoKα (λ = 0.71075 Å) graphite monochromated
Voltage, Current	50kV, 12mA
Temperature	20.0^{o}C
Detector Aperture	75.0 mm (diameter)
Data Images	540 exposures
ω oscillation Range (χ=54.0, ϕ=0.0)	-60.0 - 120.0^{o}
Exposure Rate	10.0 sec./^{o}
Detector Swing Angle	29.50^{o}
ω oscillation Range (χ=54.0, ϕ=120.0)	-60.0 - 120.0^{o}

Exposure Rate	10.0 sec./^{o}
Detector Swing Angle	29.50^{o}
ω oscillation Range (χ=54.0, ϕ=240.0)	-60.0 - 120.0^{o}
Exposure Rate	10.0 sec./^{o}
Detector Swing Angle	29.50^{o}
Detector Position	50.00 mm
Pixel Size	0.073 mm
$2\theta_{max}$	55.0^{o}
No. of Reflections Measured	Total: 20432 Unique: 8829 (R_{int} = 0.0341)
Corrections	Lorentz-polarization Absorption (*trans*. factors: 0.720 - 0.906)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SHELXS97)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	$w = 1/ [\sigma^2(Fo^2) + (0.0332 \cdot P)^2 + 2.2204 \cdot P]$ where $P = (Max(Fo^2,0) + 2Fc^2)/3$
$2\theta_{max}$ cutoff	55.0^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	8829
No. Variables	600
Reflection/Parameter Ratio	14.72

Residuals: R1 (I>2.00σ(I))	0.0387
Residuals: R (All reflections)	0.0590
Residuals: wR2 (All reflections)	0.0917
Goodness of Fit Indicator	1.029
Max Shift/Error in Final Cycle	0.001
Maximum peak in Final Diff. Map	0.60 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.57 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq} and occupancy

atom	x	y	z	B_{eq}	occ
Rh1	0.67248(3)	0.816363(19)	0.38618(2)	2.507(6)	1
Rh2	0.76662(3)	0.792253(19)	0.25524(2)	2.529(6)	1
O1	0.7773(2)	0.94661(17)	0.43890(18)	3.17(5)	1
O2	0.5343(2)	0.90088(18)	0.28586(18)	3.26(5)	1
O3	0.5633(2)	0.68871(18)	0.3271(2)	3.35(5)	1
O4	0.9296(2)	0.74126(18)	0.36253(18)	3.07(4)	1
O5	0.7687(2)	0.29884(18)	0.7750(2)	10.00(7)	2.049500
N1	0.8377(3)	0.9378(2)	0.2980(2)	2.92(5)	1
N2	0.5993(3)	0.8407(2)	0.1563(2)	2.72(5)	1
N3	0.6880(3)	0.6483(2)	0.2309(2)	2.82(5)	1
N4	0.8181(3)	0.7320(2)	0.4723(2)	2.87(5)	1
N5	0.5827(3)	0.8437(2)	0.4985(2)	3.13(5)	1
N6	0.5209(4)	0.9258(3)	0.6135(3)	4.14(7)	1
C1	0.8335(3)	0.9848(3)	0.3816(3)	2.94(6)	1
C2	0.8936(5)	1.0881(3)	0.4188(4)	3.93(8)	1
C3	0.9007(4)	0.9844(3)	0.2382(3)	3.60(7)	1
C4	0.8420(6)	1.0533(4)	0.1697(4)	5.92(13)	1
C5	0.9062(12)	1.0936(7)	0.1105(5)	9.2(3)	1
C6	1.0259(12)	1.0660(8)	0.1194(7)	10.3(3)	1
C7	1.0837(10)	0.9976(8)	0.1861(9)	9.7(3)	1
C8	1.0236(6)	0.9570(5)	0.2470(5)	5.93(13)	1

C9	0.5165(4)	0.8878(3)	0.1891(3)	3.24(7)	1
C10	0.3980(5)	0.9355(5)	0.1202(4)	5.02(11)	1
C11	0.5693(4)	0.8112(3)	0.0510(3)	2.95(6)	1
C12	0.4591(4)	0.7556(3)	-0.0058(4)	4.30(8)	1
C13	0.4374(6)	0.7233(4)	-0.1063(4)	5.46(11)	1
C14	0.5264(6)	0.7441(4)	-0.1499(4)	5.69(13)	1
C15	0.6364(6)	0.7975(4)	-0.0962(4)	5.61(12)	1
C16	0.6578(5)	0.8313(3)	0.0045(4)	4.29(9)	1
C17	0.5997(4)	0.6260(3)	0.2692(3)	3.28(7)	1
C18	0.5318(6)	0.5251(4)	0.2483(6)	5.14(12)	1
C19	0.7405(3)	0.5715(3)	0.1826(3)	3.01(6)	1
C20	0.8275(4)	0.5064(3)	0.2418(4)	4.25(8)	1
C21	0.8830(5)	0.4334(4)	0.1973(5)	5.67(12)	1
C22	0.8535(6)	0.4254(4)	0.0936(5)	5.87(12)	1
C23	0.7687(7)	0.4884(4)	0.0349(4)	6.66(14)	1
C24	0.7106(6)	0.5628(4)	0.0785(4)	5.16(11)	1

Table 1. Atomic coordinates and B_{iso}/B_{eq} and occupancy (continued)

atom	x	y	z	B_{eq}	occ
C25	0.9179(3)	0.7118(3)	0.4448(3)	2.87(6)	1
C26	1.0292(5)	0.6537(4)	0.5104(4)	4.27(9)	1
C27	0.8048(4)	0.6889(3)	0.5595(3)	3.16(6)	1
C28	0.7516(5)	0.5939(4)	0.5506(4)	5.17(10)	1
C29	0.7327(6)	0.5533(5)	0.6345(5)	6.49(14)	1
C30	0.7672(5)	0.6083(5)	0.7260(4)	5.83(12)	1
C31	0.8217(5)	0.7023(4)	0.7356(4)	4.73(9)	1
C32	0.8405(4)	0.7430(3)	0.6533(3)	3.73(8)	1
C33	0.4816(5)	0.7922(4)	0.5083(4)	4.77(10)	1
C34	0.4442(6)	0.8433(4)	0.5792(4)	5.65(12)	1
C35	0.6032(4)	0.9230(3)	0.5625(3)	3.64(7)	1
C36	1.0256(10)	0.2778(7)	0.9108(7)	11.7(3)	2.049500
C36A	1.0054(16)	0.2626(9)	0.9284(10)	6.8(3)	1
C37	0.9283(6)	0.3300(6)	0.8066(6)	8.60(17)	1

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B_{iso} involving hydrogen atoms

atom	x	y	z	B_{iso}	occ
H2A	0.855(5)	1.131(4)	0.366(4)	6.3(13)	1
H2B	0.970(5)	1.087(4)	0.424(4)	6.2(14)	1
H2C	0.885(5)	1.104(4)	0.478(4)	6.6(14)	1
H4	0.761(5)	1.074(4)	0.161(4)	7.3(16)	1
H5	0.871(6)	1.125(5)	0.077(5)	8(2)	1
H6	1.067(6)	1.095(5)	0.070(5)	9.5(17)	1
H7	1.152(6)	0.975(5)	0.195(5)	8(2)	1
H8	1.052(5)	0.913(4)	0.286(4)	5.5(14)	1
H10A	0.380(5)	0.994(4)	0.148(4)	6.8(14)	1
H10B	0.320(5)	0.886(4)	0.113(4)	7.9(16)	1
H10C	0.396(5)	0.946(4)	0.054(4)	6.6(13)	1
H12	0.402(4)	0.745(3)	0.023(3)	5.1(11)	1
H13	0.367(5)	0.685(4)	-0.138(4)	5.4(12)	1
H14	0.513(4)	0.731(3)	-0.211(3)	3.7(9)	1
H15	0.699(5)	0.808(4)	-0.122(4)	6.7(14)	1
H16	0.722(4)	0.857(3)	0.039(3)	3.3(10)	1
H18A	0.461(6)	0.525(4)	0.254(4)	7.5(16)	1
H18B	0.567(6)	0.487(5)	0.305(5)	9(2)	1
H18C	0.524(5)	0.490(4)	0.187(4)	6.0(12)	1
H20	0.852(4)	0.513(3)	0.312(4)	5.4(11)	1
H21	0.938(5)	0.389(4)	0.239(4)	7.6(15)	1

H22	0.886(5)	0.370(4)	0.062(4)	7.0(13)	1
H23	0.743(5)	0.487(4)	-0.033(4)	6.0(12)	1
H24	0.651(4)	0.598(3)	0.043(3)	3.5(9)	1
H26A	1.033(7)	0.647(5)	0.571(6)	11(2)	1
H26B	1.104(6)	0.671(5)	0.507(5)	9.1(19)	1
H26C	1.028(6)	0.597(5)	0.485(5)	8.9(19)	1
H28	0.719(5)	0.562(4)	0.490(4)	6.4(13)	1
H29	0.693(5)	0.495(4)	0.625(4)	7.3(15)	1
H30	0.757(6)	0.586(5)	0.782(5)	9.0(17)	1
H31	0.847(4)	0.743(3)	0.796(3)	4.4(10)	1
H32	0.876(4)	0.803(3)	0.662(3)	4.5(10)	1
H33	0.449(5)	0.732(4)	0.467(4)	6.5(13)	1
H34	0.383(5)	0.829(4)	0.598(4)	5.7(12)	1
H35	0.666(5)	0.968(4)	0.572(4)	5.8(13)	1

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Rh1	0.03597(15)	0.02836(14)	0.03421(15)	0.00264(11)	0.01712(12)	-0.00313(11)
Rh2	0.03525(15)	0.02957(14)	0.03430(15)	0.00209(11)	0.01661(12)	-0.00255(11)
O1	0.0481(15)	0.0356(13)	0.0393(13)	-0.0045(11)	0.0206(12)	-0.0091(11)
O2	0.0464(15)	0.0412(14)	0.0382(14)	0.0149(11)	0.0176(12)	-0.0020(11)
O3	0.0415(14)	0.0364(14)	0.0541(15)	-0.0057(11)	0.0251(13)	-0.0131(12)
O4	0.0341(13)	0.0415(14)	0.0433(14)	0.0062(11)	0.0163(11)	0.0023(11)
O5	0.0815(15)	0.1205(19)	0.180(2)	0.0051(13)	0.0439(16)	0.0372(17)
N1	0.0403(17)	0.0318(15)	0.0421(16)	-0.0048(13)	0.0188(14)	-0.0009(13)
N2	0.0365(16)	0.0341(15)	0.0326(15)	0.0049(12)	0.0124(13)	-0.0034(12)
N3	0.0378(16)	0.0292(15)	0.0415(16)	0.0012(12)	0.0168(14)	-0.0059(12)
N4	0.0422(17)	0.0330(15)	0.0359(15)	0.0053(13)	0.0156(14)	0.0045(12)
N5	0.0459(18)	0.0394(17)	0.0385(16)	0.0093(14)	0.0211(14)	-0.0012(13)
N6	0.066(2)	0.054(2)	0.0450(19)	0.0209(18)	0.0286(18)	0.0054(16)
C1	0.0380(19)	0.0310(18)	0.0403(19)	0.0003(15)	0.0116(16)	-0.0029(15)
C2	0.057(3)	0.038(2)	0.054(3)	-0.012(2)	0.021(2)	-0.008(2)
C3	0.052(2)	0.042(2)	0.045(2)	-0.0172(18)	0.0216(19)	-0.0114(17)
C4	0.082(4)	0.072(4)	0.056(3)	-0.028(3)	0.004(3)	0.019(3)
C5	0.158(8)	0.122(6)	0.046(3)	-0.085(6)	0.004(4)	0.019(4)
C6	0.187(10)	0.144(8)	0.081(5)	-0.116(8)	0.084(6)	-0.045(5)
C7	0.133(7)	0.123(7)	0.158(8)	-0.040(6)	0.121(7)	-0.050(6)
C8	0.079(4)	0.074(4)	0.093(4)	-0.005(3)	0.059(4)	-0.008(3)

C9	0.047(2)	0.037(2)	0.041(2)	0.0083(16)	0.0178(18)	0.0003(16)
C10	0.069(3)	0.074(3)	0.046(3)	0.038(3)	0.016(2)	0.005(2)
C11	0.047(2)	0.0329(18)	0.0329(18)	0.0104(16)	0.0147(16)	0.0009(14)
C12	0.052(3)	0.057(3)	0.053(3)	0.001(2)	0.018(2)	-0.010(2)
C13	0.069(3)	0.074(3)	0.051(3)	0.003(3)	0.007(3)	-0.021(2)
C14	0.120(5)	0.061(3)	0.037(3)	0.014(3)	0.030(3)	-0.005(2)
C15	0.114(5)	0.055(3)	0.065(3)	-0.004(3)	0.060(3)	-0.007(2)
C16	0.066(3)	0.050(3)	0.055(3)	-0.007(2)	0.033(3)	-0.009(2)
C17	0.042(2)	0.0342(19)	0.049(2)	-0.0033(16)	0.0177(18)	-0.0064(16)
C18	0.068(3)	0.045(3)	0.093(4)	-0.023(2)	0.047(3)	-0.026(3)
C19	0.041(2)	0.0309(18)	0.044(2)	-0.0013(15)	0.0193(17)	-0.0091(15)
C20	0.056(3)	0.049(2)	0.052(3)	0.013(2)	0.015(2)	-0.006(2)
C21	0.071(3)	0.059(3)	0.087(4)	0.026(3)	0.030(3)	-0.002(3)
C22	0.095(4)	0.058(3)	0.089(4)	0.022(3)	0.058(3)	-0.009(3)
C23	0.136(5)	0.076(4)	0.054(3)	0.026(4)	0.051(4)	-0.010(3)
C24	0.094(4)	0.055(3)	0.051(3)	0.032(3)	0.030(3)	0.003(2)

Table 3. Anisotropic displacement parameters (continued)

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C25	0.0379(19)	0.0276(17)	0.041(2)	0.0009(14)	0.0104(16)	-0.0030(14)
C26	0.050(3)	0.049(3)	0.060(3)	0.016(2)	0.015(2)	0.008(2)
C27	0.042(2)	0.038(2)	0.044(2)	0.0058(16)	0.0182(17)	0.0059(16)
C28	0.085(4)	0.051(3)	0.060(3)	-0.014(2)	0.024(3)	0.006(2)
C29	0.093(4)	0.067(4)	0.087(4)	-0.021(3)	0.029(3)	0.029(3)
C30	0.071(3)	0.100(4)	0.062(3)	0.005(3)	0.034(3)	0.029(3)
C31	0.061(3)	0.075(3)	0.050(3)	0.012(2)	0.027(2)	0.007(2)
C32	0.049(2)	0.048(2)	0.051(2)	0.0059(19)	0.025(2)	0.0041(19)
C33	0.071(3)	0.056(3)	0.069(3)	-0.007(2)	0.046(3)	-0.010(2)
C34	0.077(4)	0.082(4)	0.080(4)	0.007(3)	0.058(3)	0.003(3)
C35	0.059(3)	0.045(2)	0.040(2)	0.012(2)	0.025(2)	0.0002(17)
C36	0.112(4)	0.141(5)	0.159(7)	-0.029(3)	0.007(5)	0.015(4)
C36A	0.123(9)	0.077(5)	0.071(4)	0.042(6)	0.046(6)	0.009(4)
C37	0.077(4)	0.128(6)	0.133(6)	0.018(4)	0.047(4)	0.028(5)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Fragment Analysis

fragment: 1

Rh(1)	Rh(2)	O(1)	O(2)	O(3)
O(4)	N(1)	N(2)	N(3)	N(4)
N(5)	N(6)	C(1)	C(2)	C(3)
C(4)	C(5)	C(6)	C(7)	C(8)
C(9)	C(10)	C(11)	C(12)	C(13)
C(14)	C(15)	C(16)	C(17)	C(18)
C(19)	C(20)	C(21)	C(22)	C(23)
C(24)	C(25)	C(26)	C(27)	C(28)
C(29)	C(30)	C(31)	C(32)	C(33)
C(34)	C(35)			

fragment: 2

O(5)	C(36)	C(36A)	C(37)

Table 5. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
Rh1	Rh2	2.4065(6)	Rh1	O1	2.031(2)
Rh1	O2	2.073(2)	Rh1	O3	2.036(2)
Rh1	N4	2.039(3)	Rh1	N5	2.145(4)
Rh2	O4	2.057(2)	Rh2	N1	2.060(3)
Rh2	N2	2.028(3)	Rh2	N3	2.058(3)
O1	C1	1.294(5)	O2	C9	1.297(5)
O3	C17	1.291(5)	O4	C25	1.282(5)
O5	C37	1.709(7)	N1	C1	1.307(5)
N1	C3	1.423(6)	N2	C9	1.308(5)
N2	C11	1.421(5)	N3	C17	1.307(6)
N3	C19	1.429(5)	N4	C25	1.316(5)
N4	C27	1.427(5)	N5	C33	1.360(6)
N5	C35	1.307(5)	N6	C34	1.341(7)
N6	C35	1.337(7)	C1	C2	1.506(5)
C3	C4	1.363(7)	C3	C8	1.379(8)
C4	C5	1.390(14)	C5	C6	1.345(19)
C6	C7	1.342(14)	C7	C8	1.378(16)
C9	C10	1.505(6)	C11	C12	1.384(5)
C11	C16	1.382(8)	C12	C13	1.380(7)
C13	C14	1.361(10)	C14	C15	1.358(8)
C15	C16	1.389(7)	C17	C18	1.497(6)

C19	C20	1.381(5)	C19	C24	1.371(6)
C20	C21	1.384(8)	C21	C22	1.367(9)
C22	C23	1.349(8)	C23	C24	1.407(9)
C25	C26	1.508(6)	C27	C28	1.371(6)
C27	C32	1.384(6)	C28	C29	1.396(10)
C29	C30	1.364(9)	C30	C31	1.363(8)
C31	C32	1.376(8)	C33	C34	1.351(9)
C36	C37	1.675(11)	C36A	C37	1.899(15)

Table 6. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C2	H2A	0.94(5)	C2	H2B	0.83(6)
C2	H2C	0.87(6)	C4	H4	0.91(6)
C5	H5	0.67(6)	C6	H6	1.03(8)
C7	H7	0.79(7)	C8	H8	0.82(5)
C10	H10A	0.90(6)	C10	H10B	1.05(6)
C10	H10C	0.94(6)	C12	H12	0.88(6)
C13	H13	0.89(4)	C14	H14	0.82(4)
C15	H15	0.91(6)	C16	H16	0.77(4)
C18	H18A	0.81(7)	C18	H18B	0.93(6)
C18	H18C	0.92(5)	C20	H20	0.92(5)
C21	H21	0.93(5)	C22	H22	0.97(6)
C23	H23	0.89(5)	C24	H24	0.84(4)
C26	H26A	0.84(8)	C26	H26B	0.88(7)
C26	H26C	0.81(7)	C28	H28	0.89(5)
C29	H29	0.87(6)	C30	H30	0.90(7)
C31	H31	0.93(4)	C32	H32	0.87(4)
C33	H33	0.94(5)	C34	H34	0.83(6)
C35	H35	0.89(5)			

Table 7. Bond angles (^{o})

atom	atom	atom	angle	atom	atom	atom	angle
Rh2	Rh1	O1	90.41(8)	Rh2	Rh1	O2	87.54(8)
Rh2	Rh1	O3	88.37(9)	Rh2	Rh1	N4	85.87(10)
Rh2	Rh1	N5	177.07(8)	O1	Rh1	O2	87.33(9)
O1	Rh1	O3	177.37(10)	O1	Rh1	N4	92.28(10)
O1	Rh1	N5	88.82(11)	O2	Rh1	O3	90.29(9)
O2	Rh1	N4	173.40(13)	O2	Rh1	N5	89.60(11)
O3	Rh1	N4	89.96(10)	O3	Rh1	N5	92.28(12)
N4	Rh1	N5	96.98(12)	Rh1	Rh2	O4	89.26(8)
Rh1	Rh2	N1	85.30(10)	Rh1	Rh2	N2	86.92(10)
Rh1	Rh2	N3	86.57(10)	O4	Rh2	N1	89.30(10)
O4	Rh2	N2	175.97(13)	O4	Rh2	N3	89.22(10)
N1	Rh2	N2	91.64(11)	N1	Rh2	N3	171.76(14)
N2	Rh2	N3	89.29(11)	Rh1	O1	C1	117.8(2)
Rh1	O2	C9	117.9(2)	Rh1	O3	C17	118.4(2)
Rh2	O4	C25	116.7(2)	Rh2	N1	C1	121.5(3)
Rh2	N1	C3	118.4(2)	C1	N1	C3	119.9(3)
Rh2	N2	C9	121.3(2)	Rh2	N2	C11	117.0(2)
C9	N2	C11	121.3(3)	Rh2	N3	C17	119.5(2)
Rh2	N3	C19	119.6(3)	C17	N3	C19	120.6(3)
Rh1	N4	C25	121.2(3)	Rh1	N4	C27	118.2(3)
C25	N4	C27	120.4(3)	Rh1	N5	C33	128.0(3)

Rh1	N5	C35	125.7(3)	C33	N5	C35	105.7(4)
C34	N6	C35	105.9(4)	O1	C1	N1	122.9(3)
O1	C1	C2	114.5(4)	N1	C1	C2	122.6(4)
N1	C3	C4	122.1(4)	N1	C3	C8	119.3(4)
C4	C3	C8	118.6(5)	C3	C4	C5	119.7(7)
C4	C5	C6	121.3(8)	C5	C6	C7	119.3(12)
C6	C7	C8	121.1(11)	C3	C8	C7	120.1(6)
O2	C9	N2	121.7(3)	O2	C9	C10	114.4(4)
N2	C9	C10	123.9(4)	N2	C11	C12	122.6(4)
N2	C11	C16	119.3(3)	C12	C11	C16	117.9(4)
C11	C12	C13	120.8(5)	C12	C13	C14	119.9(5)
C13	C14	C15	120.9(5)	C14	C15	C16	119.3(6)
C11	C16	C15	121.1(5)	O3	C17	N3	123.1(3)
O3	C17	C18	114.7(5)	N3	C17	C18	122.2(4)
N3	C19	C20	119.6(4)	N3	C19	C24	121.6(3)
C20	C19	C24	118.7(4)	C19	C20	C21	120.9(5)

Table 7. Bond angles (^{o}) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C20	C21	C22	120.2(5)	C21	C22	C23	119.5(6)
C22	C23	C24	121.2(5)	C19	C24	C23	119.5(4)
O4	C25	N4	123.2(3)	O4	C25	C26	115.1(4)
N4	C25	C26	121.6(4)	N4	C27	C28	120.2(4)
N4	C27	C32	120.9(3)	C28	C27	C32	118.8(4)
C27	C28	C29	120.4(5)	C28	C29	C30	119.9(6)
C29	C30	C31	119.9(6)	C30	C31	C32	120.6(5)
C27	C32	C31	120.4(4)	N5	C33	C34	108.3(4)
N6	C34	C33	108.1(5)	N5	C35	N6	112.1(4)
O5	C37	C36	114.7(6)	O5	C37	C36A	102.7(7)
C36	C37	C36A	12.2(7)				

Table 8. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
C1	C2	H2A	107(3)	C1	C2	H2B	108(3)
C1	C2	H2C	108(3)	H2A	C2	H2B	105(5)
H2A	C2	H2C	117(5)	H2B	C2	H2C	112(5)
C3	C4	H4	122(4)	H4	C4	C5	118(4)
C4	C5	H5	113(7)	H5	C5	C6	125(7)
C5	C6	H6	118(3)	H6	C6	C7	123(4)
C6	C7	H7	127(6)	H7	C7	C8	112(6)
C3	C8	H8	115(4)	C7	C8	H8	125(4)
C9	C10	H10A	113(3)	C9	C10	H10B	107(3)
C9	C10	H10C	116(3)	H10A	C10	H10B	105(5)
H10A	C10	H10C	108(5)	H10B	C10	H10C	107(4)
C11	C12	H12	118(3)	H12	C12	C13	122(3)
C12	C13	H13	117(4)	H13	C13	C14	123(4)
C13	C14	H14	122(3)	H14	C14	C15	117(3)
C14	C15	H15	122(3)	H15	C15	C16	118(3)
C11	C16	H16	116(4)	C15	C16	H16	122(4)
C17	C18	H18A	114(4)	C17	C18	H18B	109(4)
C17	C18	H18C	117(3)	H18A	C18	H18B	93(6)
H18A	C18	H18C	108(5)	H18B	C18	H18C	113(5)
C19	C20	H20	120(3)	H20	C20	C21	119(3)
C20	C21	H21	118(4)	H21	C21	C22	121(4)

C21	C22	H22	120(3)	H22	C22	C23	120(3)
C22	C23	H23	125(3)	H23	C23	C24	114(3)
C19	C24	H24	118(3)	C23	C24	H24	122(3)
C25	C26	H26A	116(5)	C25	C26	H26B	114(4)
C25	C26	H26C	110(4)	H26A	C26	H26B	112(6)
H26A	C26	H26C	106(7)	H26B	C26	H26C	95(7)
C27	C28	H28	120(4)	H28	C28	C29	119(4)
C28	C29	H29	118(4)	H29	C29	C30	122(4)
C29	C30	H30	123(4)	H30	C30	C31	117(4)
C30	C31	H31	123(3)	H31	C31	C32	116(3)
C27	C32	H32	121(3)	C31	C32	H32	119(3)
N5	C33	H33	120(4)	H33	C33	C34	132(4)
N6	C34	H34	124(3)	C33	C34	H34	127(3)
N5	C35	H35	123(4)	N6	C35	H35	124(3)

Table 9. Torsion Angles(°)

(Those having bond angles > 160 or < 20 degrees are excluded.)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
Rh2	Rh1	O1	C1	13.43(15)	O1	Rh1	Rh2	O4	78.66(6)
O1	Rh1	Rh2	N1	-10.70(6)	O1	Rh1	Rh2	N2	-102.60(6)
O1	Rh1	Rh2	N3	167.93(6)	Rh2	Rh1	O2	C9	20.70(16)
O2	Rh1	Rh2	O4	165.97(6)	O2	Rh1	Rh2	N1	76.61(7)
O2	Rh1	Rh2	N2	-15.29(6)	O2	Rh1	Rh2	N3	-104.77(7)
Rh2	Rh1	O3	C17	19.29(16)	O3	Rh1	Rh2	O4	-103.68(7)
O3	Rh1	Rh2	N1	166.96(7)	O3	Rh1	Rh2	N2	75.06(7)
O3	Rh1	Rh2	N3	-14.41(7)	Rh2	Rh1	N4	C25	12.91(17)
Rh2	Rh1	N4	C27	-162.46(16)	N4	Rh1	Rh2	O4	-13.60(8)
N4	Rh1	Rh2	N1	-102.96(8)	N4	Rh1	Rh2	N2	165.15(8)
N4	Rh1	Rh2	N3	75.67(8)	O1	Rh1	O2	C9	111.22(19)
O2	Rh1	O1	C1	-74.09(17)	O1	Rh1	N4	C25	-77.3(2)
O1	Rh1	N4	C27	107.30(18)	N4	Rh1	O1	C1	99.31(18)
O1	Rh1	N5	C33	172.0(2)	O1	Rh1	N5	C35	2.5(2)
N5	Rh1	O1	C1	-163.75(17)	O2	Rh1	O3	C17	106.82(18)
O3	Rh1	O2	C9	-67.66(19)	O2	Rh1	N5	C33	84.6(2)
O2	Rh1	N5	C35	-84.8(2)	N5	Rh1	O2	C9	-159.94(18)
O3	Rh1	N4	C25	101.3(2)	O3	Rh1	N4	C27	-74.09(18)
N4	Rh1	O3	C17	-66.58(19)	O3	Rh1	N5	C33	-5.6(2)
O3	Rh1	N5	C35	-175.1(2)	N5	Rh1	O3	C17	-163.57(17)

N4	Rh1	N5	C33	-95.9(2)	N4	Rh1	N5	C35	94.7(2)
N5	Rh1	N4	C25	-166.42(19)	N5	Rh1	N4	C27	18.21(18)
Rh1	Rh2	O4	C25	19.09(15)	Rh1	Rh2	N1	C1	11.87(18)
Rh1	Rh2	N1	C3	-172.71(17)	Rh1	Rh2	N2	C9	15.7(2)
Rh1	Rh2	N2	C11	-157.25(18)	Rh1	Rh2	N3	C17	14.69(16)
Rh1	Rh2	N3	C19	-158.27(16)	O4	Rh2	N1	C1	-77.4(2)
O4	Rh2	N1	C3	97.98(18)	N1	Rh2	O4	C25	104.40(18)
O4	Rh2	N3	C17	104.00(19)	O4	Rh2	N3	C19	-68.97(18)
N3	Rh2	O4	C25	-67.49(18)	N1	Rh2	N2	C9	-69.5(2)
N1	Rh2	N2	C11	117.5(2)	N2	Rh2	N1	C1	98.6(2)
N2	Rh2	N1	C3	-85.94(19)	N2	Rh2	N3	C17	-72.26(19)
N2	Rh2	N3	C19	114.78(19)	N3	Rh2	N2	C9	102.4(2)
N3	Rh2	N2	C11	-70.6(2)	Rh1	O1	C1	N1	-8.0(4)
Rh1	O1	C1	C2	171.85(15)	Rh1	O2	C9	N2	-14.7(4)
Rh1	O2	C9	C10	168.25(17)	Rh1	O3	C17	N3	-13.3(4)
Rh1	O3	C17	C18	167.09(16)	Rh2	O4	C25	N4	-14.7(4)
Rh2	O4	C25	C26	166.71(15)	Rh2	N1	C1	O1	-4.9(4)

Table 9. Torsion angles (^{o}) (continued)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
Rh2	N1	C1	C2	175.26(18)	Rh2	N1	C3	C4	102.6(3)
Rh2	N1	C3	C8	-75.2(3)	C1	N1	C3	C4	-81.9(4)
C1	N1	C3	C8	100.3(4)	C3	N1	C1	O1	179.7(3)
C3	N1	C1	C2	-0.1(5)	Rh2	N2	C9	O2	-3.8(5)
Rh2	N2	C9	C10	173.0(2)	Rh2	N2	C11	C12	120.5(3)
Rh2	N2	C11	C16	-54.1(3)	C9	N2	C11	C12	-52.5(5)
C9	N2	C11	C16	132.9(3)	C11	N2	C9	O2	168.9(3)
C11	N2	C9	C10	-14.3(5)	Rh2	N3	C17	O3	-4.0(4)
Rh2	N3	C17	C18	175.56(18)	Rh2	N3	C19	C20	97.8(3)
Rh2	N3	C19	C24	-79.7(3)	C17	N3	C19	C20	-75.1(4)
C17	N3	C19	C24	107.4(4)	C19	N3	C17	O3	168.9(3)
C19	N3	C17	C18	-11.5(4)	Rh1	N4	C25	O4	-1.4(4)
Rh1	N4	C25	C26	177.08(17)	Rh1	N4	C27	C28	91.6(3)
Rh1	N4	C27	C32	-86.0(3)	C25	N4	C27	C28	-83.8(4)
C25	N4	C27	C32	98.6(4)	C27	N4	C25	O4	173.8(3)
C27	N4	C25	C26	-7.7(4)	Rh1	N5	C33	C34	-171.09(19)
Rh1	N5	C35	N6	171.38(18)	C33	N5	C35	N6	-0.0(4)
C35	N5	C33	C34	0.0(4)	C34	N6	C35	N5	-0.0(5)
C35	N6	C34	C33	0.1(5)	N1	C3	C4	C5	-177.5(3)
N1	C3	C8	C7	176.7(4)	C4	C3	C8	C7	-1.2(7)
C8	C3	C4	C5	0.4(6)	C3	C4	C5	C6	-0.1(9)

C4	C5	C6	C7	0.7(12)	C5	C6	C7	C8	-1.5(14)
C6	C7	C8	C3	1.8(12)	N2	C11	C12	C13	-176.1(3)
N2	C11	C16	C15	175.4(3)	C12	C11	C16	C15	0.5(6)
C16	C11	C12	C13	-1.3(6)	C11	C12	C13	C14	1.5(7)
C12	C13	C14	C15	-0.8(8)	C13	C14	C15	C16	-0.1(8)
C14	C15	C16	C11	0.2(7)	N3	C19	C20	C21	-177.9(3)
N3	C19	C24	C23	177.5(3)	C20	C19	C24	C23	0.0(7)
C24	C19	C20	C21	-0.4(6)	C19	C20	C21	C22	0.8(7)
C20	C21	C22	C23	-0.8(8)	C21	C22	C23	C24	0.5(9)
C22	C23	C24	C19	-0.1(9)	N4	C27	C28	C29	-176.9(3)
N4	C27	C32	C31	177.0(3)	C28	C27	C32	C31	-0.6(6)
C32	C27	C28	C29	0.7(7)	C27	C28	C29	C30	0.0(8)
C28	C29	C30	C31	-0.9(9)	C29	C30	C31	C32	1.0(8)
C30	C31	C32	C27	-0.3(7)	N5	C33	C34	N6	-0.1(5)

Table 10. Intramolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
Rh2	C16	3.357(5)	O1	O4	3.528(4)
O1	C3	3.574(6)	O1	C25	3.497(4)
O1	C35	3.027(6)	O2	N1	3.340(4)
O2	C1	3.276(4)	O2	C11	3.573(5)
O3	N2	3.337(4)	O3	C9	3.305(5)
O3	C19	3.577(5)	O3	C27	3.417(4)
O3	C28	3.417(5)	O3	C33	3.213(6)
O4	C1	3.445(4)	O4	C19	3.381(4)
O4	C20	3.450(5)	O4	C27	3.578(5)
N1	C9	3.395(5)	N2	C17	3.341(5)
N3	N4	3.294(4)	N3	C11	3.335(4)
N3	C25	3.245(4)	N4	C17	3.260(4)
N5	C27	3.142(5)	N5	C32	3.280(5)
C1	C4	3.176(7)	C1	C8	3.283(9)
C2	C3	2.829(7)	C2	C4	3.326(8)
C2	C8	3.581(10)	C3	C6	2.763(13)
C4	C7	2.723(14)	C5	C8	2.711(11)
C9	C12	3.022(6)	C9	C16	3.499(8)
C10	C11	2.902(8)	C10	C12	3.107(8)
C11	C14	2.768(6)	C12	C15	2.747(9)
C13	C16	2.738(7)	C17	C20	3.118(6)

C17	C24	3.366(8)	C18	C19	2.844(9)
C18	C20	3.321(9)	C19	C22	2.781(7)
C20	C23	2.727(8)	C21	C24	2.750(7)
C25	C28	3.186(8)	C25	C32	3.312(7)
C26	C27	2.836(8)	C26	C28	3.409(9)
C26	C32	3.525(8)	C27	C30	2.769(8)
C28	C31	2.741(7)	C29	C32	2.744(8)
C32	C35	3.523(6)			

Table 11. Intramolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
Rh1	H33	3.27(6)	Rh1	H35	3.22(5)
Rh2	H8	3.42(5)	Rh2	H16	3.06(4)
O1	H2A	2.95(6)	O1	H2B	2.90(6)
O1	H2C	2.34(5)	O1	H32	3.59(4)
O1	H35	2.56(6)	O2	H10A	2.49(5)
O2	H10B	2.74(5)	O2	H10C	3.16(5)
O3	H18A	2.44(5)	O3	H18B	2.68(6)
O3	H18C	3.12(5)	O3	H28	2.96(5)
O3	H33	2.71(6)	O4	H8	3.07(6)
O4	H20	3.12(4)	O4	H26A	3.08(7)
O4	H26B	2.49(6)	O4	H26C	2.63(6)
N1	H2A	2.67(5)	N1	H2B	2.63(5)
N1	H2C	3.14(5)	N1	H4	2.64(6)
N1	H8	2.47(6)	N1	H16	3.49(4)
N2	H4	3.55(6)	N2	H10A	3.16(6)
N2	H10B	3.02(6)	N2	H10C	2.67(5)
N2	H12	2.59(4)	N2	H16	2.48(5)
N3	H18A	3.11(7)	N3	H18B	2.94(7)
N3	H18C	2.66(5)	N3	H20	2.58(4)
N3	H24	2.56(4)	N4	H26A	2.61(7)
N4	H26B	3.15(7)	N4	H26C	2.92(7)

N4	H28	2.58(5)	N4	H32	2.61(4)
N5	H32	3.34(4)	N5	H34	3.00(6)
N6	H33	3.11(5)	C1	H4	3.20(6)
C1	H8	3.27(6)	C2	H4	3.38(5)
H2A	C3	2.73(6)	H2A	C4	2.82(6)
H2A	H4	2.74(7)	H2B	C3	2.71(5)
H2B	C4	3.33(5)	H2B	H4	3.59(7)
H2B	C8	3.15(6)	H2B	H8	3.25(8)
C3	H5	2.95(7)	C3	H7	3.04(7)
C3	H16	3.18(4)	C4	H6	3.30(8)
C4	H7	3.50(7)	C4	H8	3.06(5)
C4	H16	3.10(4)	H4	H5	2.10(10)
H4	C6	3.19(7)	H4	C8	3.20(6)
H4	H16	3.21(7)	C5	H7	3.06(6)
C5	H8	3.52(5)	H5	H6	2.24(10)
H5	C7	2.94(6)	H5	C8	3.38(6)

Table 11. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C6	H8	3.12(5)	H6	H7	2.39(9)
H6	C8	3.32(7)	H7	H8	2.14(10)
C9	H12	2.83(4)	C10	H12	2.82(5)
H10B	C11	3.32(7)	H10B	C12	3.10(6)
H10B	H12	2.55(8)	H10C	C11	2.65(5)
H10C	C12	2.79(5)	H10C	H12	2.69(7)
H10C	C16	3.54(6)	C11	H13	3.18(4)
C11	H14	3.59(4)	C11	H15	3.22(6)
C11	H24	3.00(4)	C12	H14	3.13(5)
C12	H16	3.05(4)	C12	H24	2.94(4)
H12	H13	2.25(7)	H12	C14	3.17(6)
H12	C16	3.15(5)	H12	H24	3.34(6)
C13	H15	3.19(6)	C13	H16	3.51(4)
C13	H24	3.13(4)	H13	H14	2.29(8)
H13	C15	3.18(5)	H13	H24	3.55(6)
C14	H16	3.08(3)	C14	H24	3.32(4)
H14	H15	2.21(6)	H14	C16	3.09(4)
C15	H24	3.36(4)	H15	H16	2.24(7)
C16	H24	3.19(4)	H16	H24	3.54(6)
C17	H20	3.07(5)	C17	H24	3.40(5)
C17	H28	3.07(5)	C18	H20	3.35(5)

C18	H28	3.29(5)	H18A	H28	3.53(7)
H18B	C19	3.22(8)	H18B	C20	3.31(8)
H18B	H20	3.15(9)	H18B	C28	3.54(6)
H18B	H28	2.67(7)	H18C	C19	2.64(5)
H18C	C20	3.18(5)	H18C	H20	3.44(6)
H18C	C24	3.13(6)	H18C	H24	3.22(7)
C19	H21	3.23(6)	C19	H23	3.16(5)
C20	H22	3.27(6)	C20	H24	3.11(4)
C20	H26C	3.49(6)	H20	H21	2.27(8)
H20	C22	3.20(5)	H20	C24	3.20(4)
H20	C25	3.07(4)	H20	C26	3.26(4)
H20	H26C	2.70(7)	H20	H28	3.34(8)
C21	H23	3.18(5)	C21	H24	3.58(4)
H21	H22	2.34(8)	H21	C23	3.19(5)
C22	H24	3.15(4)	H22	H23	2.35(7)
H22	C24	3.28(5)	H23	H24	2.21(7)

Table 11. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C25	H28	3.21(6)	C25	H32	3.38(5)
C26	H28	3.56(6)	H26A	C27	2.55(8)
H26A	C28	3.11(8)	H26A	H28	3.43(9)
H26A	C32	3.02(8)	H26A	H32	3.19(9)
H26C	C27	3.23(7)	H26C	C28	3.49(8)
H26C	H28	3.49(9)	C27	H29	3.16(6)
C27	H31	3.21(5)	C28	H30	3.22(7)
C28	H32	3.16(4)	H28	H29	2.23(9)
H28	C30	3.18(5)	H28	C32	3.17(5)
C29	H31	3.22(4)	H29	H30	2.32(8)
H29	C31	3.15(5)	C30	H32	3.15(5)
H30	H31	2.28(7)	H30	C32	3.17(7)
H31	H32	2.20(7)	H32	C35	3.33(4)
H32	H35	3.20(6)	C33	H35	2.98(5)
H33	H34	2.49(8)	H33	C35	3.01(5)
C34	H35	2.99(5)	H34	C35	2.94(6)

Table 12. Intermolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
O1	N6[1]	3.594(5)	O1	C2[2]	3.548(5)
O2	N6[1]	2.813(5)	O3	O5[3]	3.466(3)
O4	C37[4]	3.361(9)	O5	O3[3]	3.466(3)
N5	N6[1]	3.541(5)	N6	O1[1]	3.594(5)
N6	O2[1]	2.813(5)	N6	N5[1]	3.541(5)
N6	C35[1]	3.203(5)	C1	C2[2]	3.518(6)
C2	O1[2]	3.548(5)	C2	C1[2]	3.518(6)
C2	C25[2]	3.413(5)	C2	C26[2]	3.535(7)
C6	C6[5]	3.536(13)	C10	C15[6]	3.599(8)
C10	C16[6]	3.599(8)	C15	C10[6]	3.599(8)
C16	C10[6]	3.599(8)	C24	C36[4]	3.553(12)
C25	C2[2]	3.413(5)	C26	C2[2]	3.535(7)
C35	N6[1]	3.203(5)	C35	C35[1]	3.196(6)
C36	C24[4]	3.553(12)	C37	O4[4]	3.361(9)

Symmetry Operators:

(1) -X+1,-Y+2,-Z+1

(2) -X+2,-Y+2,-Z+1

(3) -X+1,-Y+1,-Z+1

(4) -X+2,-Y+1,-Z+1

(5) -X+2,-Y+2,-Z

(6) -X+1,-Y+2,-Z

Table 13. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
O1	H2B[1]	2.85(5)	O1	H2C[1]	3.59(5)
O1	H34[2]	3.47(5)	O4	H2B[1]	3.50(5)
O4	H2C[1]	3.11(5)	O5	H10B[3]	3.31(6)
O5	H18A[3]	3.41(6)	O5	H29	3.38(6)
O5	H33[3]	3.41(4)	N4	H2B[1]	3.25(5)
N6	H10A[2]	3.24(5)	N6	H35[2]	3.12(4)
C1	H2B[1]	3.04(5)	C1	H2C[1]	3.32(5)
C1	H34[2]	3.54(5)	C2	H2B[1]	3.30(5)
C2	H2C[1]	3.56(5)	C2	H26A[1]	3.60(7)
C2	H26B[1]	3.31(6)	C2	H32[1]	3.46(5)
C2	H34[2]	3.21(5)	H2A	C25[1]	3.52(4)
H2A	C26[1]	3.27(5)	H2A	H26A[1]	3.15(9)
H2A	H26B[1]	3.03(8)	H2A	H32[1]	3.25(8)
H2A	H34[2]	2.91(8)	H2B	O1[1]	2.85(5)
H2B	O4[1]	3.50(5)	H2B	N4[1]	3.25(5)
H2B	C1[1]	3.04(5)	H2B	C2[1]	3.30(5)
H2B	H2B[1]	3.15(7)	H2B	H2C[1]	3.13(7)
H2B	C25[1]	3.15(5)	H2B	C26[1]	3.52(5)
H2B	H26A[1]	3.54(9)	H2B	H26B[1]	3.50(8)
H2B	C32[1]	3.53(6)	H2B	H32[1]	2.83(8)
H2C	O1[1]	3.59(5)	H2C	O4[1]	3.11(5)

H2C	C1[1]	3.32(5)	H2C	C2[1]	3.56(5)
H2C	H2B[1]	3.13(7)	H2C	H8[1]	3.14(8)
H2C	C25[1]	3.15(5)	H2C	C26[1]	3.34(5)
H2C	H26A[1]	3.60(9)	H2C	H26B[1]	2.99(8)
H2C	C34[2]	3.55(5)	H2C	H34[2]	2.96(7)
C4	H10C[4]	3.32(5)	H4	H10C[4]	2.89(7)
H4	C12[4]	3.58(5)	H4	C13[4]	3.45(6)
H4	H13[4]	3.50(7)	C5	H6[5]	3.54(7)
C5	H10B[4]	3.28(5)	C5	H10C[4]	3.36(5)
C5	H31[1]	3.32(4)	C5	H32[1]	3.46(4)
H5	C10[4]	3.33(6)	H5	H10B[4]	2.75(7)
H5	H10C[4]	3.00(7)	H5	H12[4]	3.40(8)
H5	H22[6]	3.28(8)	H5	H31[1]	3.43(7)
H5	C36A[7]	3.52(8)	C6	H6[5]	3.17(6)
C6	H15[5]	3.45(6)	C6	H31[1]	2.88(4)
C6	H32[1]	3.26(4)	H6	C5[5]	3.54(7)

Table 13. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H6	C6^{5}	3.17(6)	H6	H6^{5}	3.13(8)
H6	C7^{5}	3.53(6)	H6	H10A^{8}	3.57(8)
H6	C15^{5}	3.47(7)	H6	H15^{5}	2.73(8)
H6	H16^{5}	3.29(9)	H6	C31^{1}	3.60(6)
H6	H31^{1}	2.70(7)	H6	C36^{7}	3.32(7)
H6	C36A^{7}	3.00(6)	C7	H6^{5}	3.53(6)
C7	H10A^{8}	3.51(6)	C7	H10B^{8}	3.44(7)
C7	H31^{1}	3.51(4)	C7	H32^{1}	3.23(4)
C7	H35^{1}	3.55(4)	H7	C10^{8}	3.28(8)
H7	H10A^{8}	2.85(10)	H7	H10B^{8}	2.75(10)
H7	H32^{1}	3.55(8)	H7	H35^{1}	3.22(7)
C8	H32^{1}	3.40(4)	C8	H35^{1}	3.58(4)
H8	H2C^{1}	3.14(8)	H8	H35^{1}	3.38(6)
H8	C36^{9}	3.50(5)	H8	C36A^{9}	3.56(5)
H8	C37^{9}	3.45(5)	C10	H5^{4}	3.33(6)
C10	H7^{10}	3.28(8)	C10	H15^{4}	3.59(5)
H10A	N6^{2}	3.24(5)	H10A	H6^{10}	3.57(8)
H10A	C7^{10}	3.51(6)	H10A	H7^{10}	2.85(10)
H10A	C15^{4}	2.90(6)	H10A	H15^{4}	2.78(7)
H10A	C16^{4}	3.17(6)	H10A	H16^{4}	3.26(7)
H10B	O5^{3}	3.31(6)	H10B	C5^{4}	3.28(5)

H10B	H5[4]	2.75(7)	H10B	C7[10]	3.44(7)
H10B	H7[10]	2.75(10)	H10C	C4[4]	3.32(5)
H10C	H4[4]	2.89(7)	H10C	C5[4]	3.36(5)
H10C	H5[4]	3.00(7)	H10C	H10C[4]	3.49(9)
H10C	C15[4]	3.47(5)	H10C	C16[4]	3.13(5)
H10C	H16[4]	3.09(6)	C12	H4[4]	3.58(5)
H12	H5[4]	3.40(8)	H12	C22[11]	3.48(4)
H12	H22[11]	3.32(7)	H12	C23[11]	3.52(4)
H12	H23[11]	3.50(7)	C13	H4[4]	3.45(6)
C13	H18C[11]	3.09(5)	H13	H4[4]	3.50(7)
H13	C18[11]	3.49(5)	H13	H18B[11]	3.44(9)
H13	H18C[11]	2.80(7)	H13	C19[11]	3.56(5)
H13	C20[11]	3.25(4)	H13	C21[11]	3.01(5)
H13	H21[11]	3.30(7)	H13	C22[11]	3.10(5)
H13	H22[11]	3.42(8)	H13	C23[11]	3.39(5)
C14	H18B[11]	3.59(6)	C14	H18C[11]	3.15(5)

Table 13. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C14	H34[12]	3.57(5)	H14	C18[11]	3.42(4)
H14	H18A[11]	3.45(7)	H14	H18B[11]	3.10(7)
H14	H18C[11]	3.02(6)	H14	H30[12]	3.36(8)
H14	C34[12]	3.22(4)	H14	H34[12]	2.94(6)
C15	H6[5]	3.47(7)	C15	H10A[4]	2.90(6)
C15	H10C[4]	3.47(5)	C15	H31[12]	3.25(5)
H15	C6[5]	3.45(6)	H15	H6[5]	2.73(8)
H15	C10[4]	3.59(5)	H15	H10A[4]	2.78(7)
H15	C30[12]	3.55(6)	H15	H30[12]	3.34(8)
H15	C31[12]	3.06(6)	H15	H31[12]	2.43(8)
C16	H10A[4]	3.17(6)	C16	H10C[4]	3.13(5)
H16	H6[5]	3.29(9)	H16	H10A[4]	3.26(7)
H16	H10C[4]	3.09(6)	H16	C36[9]	3.22(4)
H16	C36A[9]	3.33(5)	C18	H13[11]	3.49(5)
C18	H14[11]	3.42(4)	C18	H23[11]	3.45(4)
C18	H29[3]	3.54(7)	C18	H30[3]	3.40(6)
H18A	O5[3]	3.41(6)	H18A	H14[11]	3.45(7)
H18A	H23[11]	3.12(7)	H18A	C29[3]	3.25(7)
H18A	H29[3]	2.81(10)	H18A	C30[3]	3.18(7)
H18A	H30[3]	2.70(9)	H18B	H13[11]	3.44(9)
H18B	C14[11]	3.59(6)	H18B	H14[11]	3.10(7)

H18B	H29[3]	3.35(10)	H18B	H30[3]	3.48(9)
H18C	C13[11]	3.09(5)	H18C	H13[11]	2.80(7)
H18C	C14[11]	3.15(5)	H18C	H14[11]	3.02(6)
H18C	H23[11]	3.03(6)	H18C	H24[11]	3.27(6)
H18C	H30[3]	3.45(9)	C19	H13[11]	3.56(5)
C20	H13[11]	3.25(4)	C20	H26A[9]	3.36(7)
H20	C26[9]	3.36(5)	H20	H26A[9]	2.80(8)
H20	H26B[9]	3.55(8)	H20	H26C[9]	3.15(8)
C21	H13[11]	3.01(5)	C21	H26A[9]	3.29(8)
H21	H13[11]	3.30(7)	H21	C26[9]	3.47(6)
H21	H26A[9]	2.63(10)	H21	C27[9]	3.45(5)
H21	C29[9]	3.51(5)	H21	C30[9]	3.13(6)
H21	H30[9]	3.50(9)	H21	C31[9]	2.85(6)
H21	H31[9]	3.13(8)	H21	C32[9]	3.03(5)
H21	H32[9]	3.35(7)	C22	H12[11]	3.48(4)
C22	H13[11]	3.10(5)	H22	H5[13]	3.28(8)

Table 13. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H22	H12[11]	3.32(7)	H22	H13[11]	3.42(8)
H22	H31[9]	3.37(6)	H22	C36[12]	3.21(6)
H22	C36A[12]	2.95(6)	C23	H12[11]	3.52(4)
C23	H13[11]	3.39(5)	H23	H12[11]	3.50(7)
H23	C18[11]	3.45(4)	H23	H18A[11]	3.12(7)
H23	H18C[11]	3.03(6)	H23	H30[12]	3.01(9)
H24	H18C[11]	3.27(6)	C25	H2A[1]	3.52(4)
C25	H2B[1]	3.15(5)	C25	H2C[1]	3.15(5)
C26	H2A[1]	3.27(5)	C26	H2B[1]	3.52(5)
C26	H2C[1]	3.34(5)	C26	H20[9]	3.36(5)
C26	H21[9]	3.47(6)	C26	H26C[9]	3.40(7)
H26A	C2[1]	3.60(7)	H26A	H2A[1]	3.15(9)
H26A	H2B[1]	3.54(9)	H26A	H2C[1]	3.60(9)
H26A	C20[9]	3.36(7)	H26A	H20[9]	2.80(8)
H26A	C21[9]	3.29(8)	H26A	H21[9]	2.63(10)
H26A	H26C[9]	3.31(10)	H26B	C2[1]	3.31(6)
H26B	H2A[1]	3.03(8)	H26B	H2B[1]	3.50(8)
H26B	H2C[1]	2.99(8)	H26B	H20[9]	3.55(8)
H26B	H34[8]	3.53(8)	H26C	H20[9]	3.15(8)
H26C	C26[9]	3.40(7)	H26C	H26A[9]	3.31(10)
H26C	H26C[9]	2.75(10)	H26C	H28[9]	3.46(9)

C27	H21[9]	3.45(5)	H28	H26C[9]	3.46(9)
C29	H18A[3]	3.25(7)	C29	H21[9]	3.51(5)
H29	O5	3.38(6)	H29	C18[3]	3.54(7)
H29	H18A[3]	2.81(10)	H29	H18B[3]	3.35(10)
H29	H33[3]	3.38(7)	C30	H15[14]	3.55(6)
C30	H18A[3]	3.18(7)	C30	H21[9]	3.13(6)
H30	H14[14]	3.36(8)	H30	H15[14]	3.34(8)
H30	C18[3]	3.40(6)	H30	H18A[3]	2.70(9)
H30	H18B[3]	3.48(9)	H30	H18C[3]	3.45(9)
H30	H21[9]	3.50(9)	H30	H23[14]	3.01(9)
C31	H6[1]	3.60(6)	C31	H15[14]	3.06(6)
C31	H21[9]	2.85(6)	H31	C5[1]	3.32(4)
H31	H5[1]	3.43(7)	H31	C6[1]	2.88(4)
H31	H6[1]	2.70(7)	H31	C7[1]	3.51(4)
H31	C15[14]	3.25(5)	H31	H15[14]	2.43(8)
H31	H21[9]	3.13(8)	H31	H22[9]	3.37(6)

Table 13. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C32	H2B[1]	3.53(6)	C32	H21[9]	3.03(5)
H32	C2[1]	3.46(5)	H32	H2A[1]	3.25(8)
H32	H2B[1]	2.83(8)	H32	C5[1]	3.46(4)
H32	C6[1]	3.26(4)	H32	C7[1]	3.23(4)
H32	H7[1]	3.55(8)	H32	C8[1]	3.40(4)
H32	H21[9]	3.35(7)	H33	O5[3]	3.41(4)
H33	H29[3]	3.38(7)	C34	H2C[2]	3.55(5)
C34	H14[14]	3.22(4)	C34	H35[2]	3.34(5)
H34	O1[2]	3.47(5)	H34	C1[2]	3.54(5)
H34	C2[2]	3.21(5)	H34	H2A[2]	2.91(8)
H34	H2C[2]	2.96(7)	H34	C14[14]	3.57(5)
H34	H14[14]	2.94(6)	H34	H26B[10]	3.53(8)
C35	H35[2]	3.33(4)	H35	N6[2]	3.12(4)
H35	C7[1]	3.55(4)	H35	H7[1]	3.22(7)
H35	C8[1]	3.58(4)	H35	H8[1]	3.38(6)
H35	C34[2]	3.34(5)	H35	C35[2]	3.33(4)
C36	H6[15]	3.32(7)	C36	H8[9]	3.50(5)
C36	H16[9]	3.22(4)	C36	H22[14]	3.21(6)
C36A	H5[15]	3.52(8)	C36A	H6[15]	3.00(6)
C36A	H8[9]	3.56(5)	C36A	H16[9]	3.33(5)
C36A	H22[14]	2.95(6)	C37	H8[9]	3.45(5)

Symmetry Operators:

(1) -X+2,-Y+2,-Z+1

(2) -X+1,-Y+2,-Z+1

(3) -X+1,-Y+1,-Z+1

(4) -X+1,-Y+2,-Z

(5) -X+2,-Y+2,-Z

(6) X,Y+1,Z

(7) X,Y+1,Z-1

(8) X+1,Y,Z

(9) -X+2,-Y+1,-Z+1

(10) X-1,Y,Z

(11) -X+1,-Y+1,-Z

(12) X,Y,Z-1

(13) X,Y-1,Z

(14) X,Y,Z+1

(15) X,Y-1,Z+1

Appendix B: Supplementary Information for Mo-1

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{21}H_{21}MoN_2O_6S$
Formula Weight	525.41
Crystal Color, Habit	orange, block
Crystal Dimensions	0.200 X 0.180 X 0.090 mm
Crystal System	monoclinic
Lattice Type	Primitive
Lattice Parameters	a = 10.9687(9) Å
	b = 12.3436(10) Å

	c = 15.6255(12) Å
	β = 99.434(7) ^{o}
	V = 2087.0(3) Å^{3}
Space Group	P2$_{1}$/c (#14)
Z value	4
D_{calc}	1.672 g/cm^{3}
F_{000}	1068.00
μ(MoKα)	7.695 cm^{-1}

B. Intensity Measurements

Diffractometer	XtaLAB mini
Radiation	MoKα (λ = 0.71075 Å) graphite monochromated
Voltage, Current	50kV, 12mA
Temperature	20.0^{o}C
Detector Aperture	75.0 mm (diameter)
Data Images	540 exposures
ω oscillation Range (χ=54.0, ϕ=0.0)	-60.0 - 120.0^{o}
Exposure Rate	10.0 sec./^{o}
Detector Swing Angle	29.50^{o}
ω oscillation Range (χ=54.0, ϕ=120.0)	-60.0 - 120.0^{o}

Exposure Rate	10.0 sec./^{o}
Detector Swing Angle	29.50^{o}
ω oscillation Range (χ=54.0, ϕ=240.0)	-60.0 - 120.0^{o}
Exposure Rate	10.0 sec./^{o}
Detector Swing Angle	29.50^{o}
ω oscillation Range (χ=0.0, ϕ=0.0)	-60.0 - 120.0^{o}
Exposure Rate	10.0 sec./^{o}
Detector Swing Angle	29.50^{o}
ω oscillation Range (χ=0.0, ϕ=120.0)	-60.0 - 120.0^{o}
Exposure Rate	10.0 sec./^{o}
Detector Swing Angle	29.50^{o}
ω oscillation Range (χ=0.0, ϕ=240.0)	-60.0 - 120.0^{o}

Exposure Rate	10.0 sec./^{o}
Detector Swing Angle	29.50^{o}
Detector Position	50.00 mm
Pixel Size	0.146 mm
$2\theta_{max}$	55.0^{o}
No. of Reflections Measured	Total: 21488 Unique: 4773 (R_{int} = 0.0674)
Corrections	Lorentz-polarization Absorption (*trans*. factors: 0.738 - 0.933)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	$w = 1/ [\sigma^2(Fo^2) + (0.0252 \cdot P)^2 + 2.8910 \cdot P]$ where $P = (Max(Fo^2,0) + 2Fc^2)/3$
$2\theta_{max}$ cutoff	55.0^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	4773
No. Variables	283
Reflection/Parameter Ratio	16.87

Residuals: R1 (I>2.00σ(I))	0.0419
Residuals: R (All reflections)	0.0641
Residuals: wR2 (All reflections)	0.0882
Goodness of Fit Indicator	1.068
Max Shift/Error in Final Cycle	0.001
Maximum peak in Final Diff. Map	0.58 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.57 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
Mo1	0.75920(3)	0.71332(2)	0.634074(18)	1.943(7)
S1	0.99263(9)	0.89172(9)	0.69155(7)	3.60(2)
O1	0.7030(2)	0.86868(18)	0.63133(14)	2.13(4)
O2	0.8429(2)	0.59757(18)	0.58587(14)	2.37(4)
O3	0.6168(2)	0.6589(2)	0.62428(17)	3.17(5)
O4	0.8193(3)	0.7080(2)	0.74186(15)	3.05(5)
O5	0.9461(2)	0.80863(19)	0.62273(16)	2.80(5)
O6	0.9053(2)	0.39277(19)	0.58258(16)	2.83(5)
N1	0.7367(2)	0.7717(2)	0.49741(17)	1.85(5)
N2	0.7014(3)	0.8801(2)	0.48353(17)	2.03(5)
C1	0.8269(3)	0.5492(3)	0.5078(2)	1.91(6)
C2	0.8626(3)	0.4397(3)	0.5043(2)	2.16(6)
C3	0.8550(3)	0.3886(3)	0.4251(2)	2.70(7)
C4	0.8126(4)	0.4442(3)	0.3494(2)	2.90(7)
C5	0.7774(3)	0.5506(3)	0.3508(2)	2.54(6)
C6	0.7833(3)	0.6045(3)	0.4305(2)	2.00(6)
C7	0.7463(3)	0.7166(3)	0.4290(2)	2.02(5)
C8	0.6850(3)	0.9223(3)	0.5571(2)	1.87(6)
C9	0.6400(3)	1.1041(3)	0.4865(2)	2.35(6)
C10	0.6130(3)	1.2114(3)	0.4917(2)	2.56(6)
C11	0.5871(3)	1.2571(3)	0.5703(2)	2.28(6)

C12	0.5627(3)	1.3691(3)	0.5795(3)	3.15(8)
C13	0.5351(3)	1.4083(3)	0.6556(3)	3.29(8)
C14	0.5294(4)	1.3397(3)	0.7259(3)	3.38(8)
C15	0.5542(3)	1.2322(3)	0.7200(3)	2.80(7)
C16	0.5854(3)	1.1886(3)	0.6425(2)	2.04(6)
C17	0.6167(3)	1.0778(3)	0.6360(2)	2.10(6)
C18	0.6455(3)	1.0368(3)	0.5603(2)	1.91(6)
C19	0.9383(4)	0.2814(3)	0.5817(3)	3.03(7)
C21	1.1537(4)	0.8703(4)	0.7185(3)	4.62(10)
C22	1.0010(6)	1.0140(4)	0.6357(5)	9.8(3)

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B_{iso} involving hydrogen atoms

atom	x	y	z	B_{iso}
H3	0.87845	0.31641	0.42268	3.236
H4	0.80793	0.40873	0.29640	3.476
H5	0.74963	0.58707	0.29915	3.045
H7	0.72846	0.75110	0.37544	2.427
H9	0.65496	1.07506	0.43434	2.815
H10	0.61151	1.25528	0.44316	3.068
H12	0.56556	1.41608	0.53337	3.777
H13	0.51974	1.48193	0.66074	3.950
H14	0.50866	1.36761	0.77694	4.057
H15	0.55059	1.18702	0.76719	3.363
H17	0.61758	1.03240	0.68360	2.523
H19A	0.99190	0.27003	0.53975	3.638
H19B	0.86500	0.23836	0.56660	3.638
H19C	0.98017	0.26064	0.63813	3.638
H21A	1.19046	0.87454	0.66694	5.548
H21B	1.16886	0.80000	0.74430	5.548
H21C	1.18910	0.92479	0.75892	5.548
H22A	0.91910	1.03726	0.61129	11.746
H22B	1.04866	1.00355	0.59003	11.746
H22C	1.03967	1.06817	0.67515	11.746

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Mo1	0.03308(16)	0.01956(14)	0.02114(14)	0.00419(14)	0.00432(11)	0.00001(12)
S1	0.0343(5)	0.0438(6)	0.0567(7)	0.0000(5)	0.0018(5)	-0.0235(5)
O1	0.0357(13)	0.0206(12)	0.0251(12)	0.0063(10)	0.0067(10)	0.0028(10)
O2	0.0414(15)	0.0233(12)	0.0229(12)	0.0098(11)	-0.0013(10)	-0.0055(10)
O3	0.0423(16)	0.0338(14)	0.0455(16)	-0.0019(12)	0.0104(13)	0.0054(12)
O4	0.0608(18)	0.0326(14)	0.0209(12)	0.0081(13)	0.0021(11)	-0.0024(11)
O5	0.0356(14)	0.0318(14)	0.0387(15)	-0.0029(11)	0.0048(11)	-0.0100(11)
O6	0.0481(16)	0.0250(13)	0.0318(14)	0.0120(12)	-0.0008(12)	-0.0010(11)
N1	0.0267(14)	0.0198(14)	0.0233(14)	0.0041(12)	0.0029(11)	0.0014(11)
N2	0.0333(16)	0.0179(14)	0.0253(15)	0.0040(12)	0.0035(12)	-0.0006(11)
C1	0.0260(17)	0.0213(17)	0.0250(17)	0.0000(14)	0.0034(14)	-0.0062(14)
C2	0.0252(18)	0.0243(17)	0.0318(19)	0.0022(14)	0.0026(14)	-0.0035(15)
C3	0.040(2)	0.0199(17)	0.041(2)	0.0027(16)	0.0043(17)	-0.0070(16)
C4	0.048(2)	0.030(2)	0.031(2)	0.0029(18)	0.0054(17)	-0.0105(16)
C5	0.038(2)	0.033(2)	0.0257(19)	0.0028(16)	0.0043(15)	-0.0022(15)
C6	0.0248(17)	0.0261(17)	0.0249(17)	0.0005(14)	0.0030(14)	-0.0033(14)
C7	0.0252(16)	0.0274(17)	0.0239(16)	-0.0004(15)	0.0031(13)	-0.0010(15)
C8	0.0201(16)	0.0211(16)	0.0293(18)	0.0024(13)	0.0017(14)	0.0014(14)
C9	0.0288(19)	0.034(2)	0.0268(18)	0.0013(15)	0.0048(15)	0.0031(15)
C10	0.0322(19)	0.0283(18)	0.037(2)	0.0027(16)	0.0057(15)	0.0094(17)
C11	0.0226(17)	0.0198(16)	0.043(2)	0.0003(14)	0.0015(15)	0.0023(15)

C12	0.038(2)	0.0194(18)	0.062(3)	0.0043(16)	0.0060(19)	0.0067(18)
C13	0.031(2)	0.0224(19)	0.071(3)	0.0055(16)	0.006(2)	-0.0087(19)
C14	0.036(2)	0.038(2)	0.054(3)	0.0070(18)	0.0067(19)	-0.013(2)
C15	0.035(2)	0.030(2)	0.041(2)	0.0029(16)	0.0078(17)	-0.0029(17)
C16	0.0222(17)	0.0195(17)	0.0353(19)	-0.0011(13)	0.0037(14)	-0.0027(14)
C17	0.0299(19)	0.0214(17)	0.0281(18)	0.0035(14)	0.0030(14)	0.0019(14)
C18	0.0236(17)	0.0215(16)	0.0266(18)	0.0021(14)	0.0009(13)	-0.0001(14)
C19	0.040(2)	0.0272(19)	0.045(2)	0.0078(17)	-0.0022(17)	0.0003(17)
C21	0.039(2)	0.075(3)	0.058(3)	0.002(2)	-0.005(2)	-0.014(3)
C22	0.110(5)	0.039(3)	0.189(8)	-0.016(3)	-0.077(5)	0.022(4)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
Mo1	O1	2.013(2)	Mo1	O2	1.918(2)
Mo1	O3	1.684(3)	Mo1	O4	1.706(2)
Mo1	O5	2.395(2)	Mo1	N1	2.229(3)
S1	O5	1.513(3)	S1	C21	1.767(4)
S1	C22	1.754(6)	O1	C8	1.322(4)
O2	C1	1.344(4)	O6	C2	1.365(4)
O6	C19	1.422(4)	N1	N2	1.400(4)
N1	C7	1.286(4)	N2	C8	1.301(4)
C1	C2	1.410(5)	C1	C6	1.400(4)
C2	C3	1.380(5)	C3	C4	1.381(5)
C4	C5	1.371(5)	C5	C6	1.404(5)
C6	C7	1.442(5)	C8	C18	1.481(5)
C9	C10	1.362(5)	C9	C18	1.415(5)
C10	C11	1.421(5)	C11	C12	1.420(5)
C11	C16	1.414(5)	C12	C13	1.362(6)
C13	C14	1.397(6)	C14	C15	1.361(5)
C15	C16	1.418(5)	C16	C17	1.418(4)
C17	C18	1.369(5)			

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C3	H3	0.930	C4	H4	0.930
C5	H5	0.930	C7	H7	0.930
C9	H9	0.930	C10	H10	0.930
C12	H12	0.930	C13	H13	0.930
C14	H14	0.930	C15	H15	0.930
C17	H17	0.930	C19	H19A	0.960
C19	H19B	0.960	C19	H19C	0.960
C21	H21A	0.960	C21	H21B	0.960
C21	H21C	0.960	C22	H22A	0.960
C22	H22B	0.960	C22	H22C	0.960

Table 6. Bond angles (^{o})

atom	atom	atom	angle	atom	atom	atom	angle
O1	Mo1	O2	150.00(10)	O1	Mo1	O3	95.87(11)
O1	Mo1	O4	97.20(10)	O1	Mo1	O5	78.06(9)
O1	Mo1	N1	71.70(9)	O2	Mo1	O3	99.42(12)
O2	Mo1	O4	103.08(11)	O2	Mo1	O5	82.31(9)
O2	Mo1	N1	81.35(10)	O3	Mo1	O4	106.08(13)
O3	Mo1	O5	168.97(10)	O3	Mo1	N1	94.98(11)
O4	Mo1	O5	83.97(11)	O4	Mo1	N1	157.25(11)
O5	Mo1	N1	74.44(9)	O5	S1	C21	106.0(2)
O5	S1	C22	105.6(3)	C21	S1	C22	96.6(3)
Mo1	O1	C8	119.8(2)	Mo1	O2	C1	133.45(19)
Mo1	O5	S1	118.28(15)	C2	O6	C19	116.9(3)
Mo1	N1	N2	116.23(19)	Mo1	N1	C7	127.9(2)
N2	N1	C7	115.8(3)	N1	N2	C8	108.7(3)
O2	C1	C2	117.7(3)	O2	C1	C6	122.8(3)
C2	C1	C6	119.5(3)	O6	C2	C1	115.4(3)
O6	C2	C3	124.8(3)	C1	C2	C3	119.7(3)
C2	C3	C4	120.3(3)	C3	C4	C5	121.2(3)
C4	C5	C6	119.7(3)	C1	C6	C5	119.6(3)
C1	C6	C7	122.6(3)	C5	C6	C7	117.8(3)
N1	C7	C6	123.7(3)	O1	C8	N2	123.5(3)
O1	C8	C18	116.8(3)	N2	C8	C18	119.7(3)

C10	C9	C18	120.2(3)	C9	C10	C11	120.9(3)
C10	C11	C12	122.9(3)	C10	C11	C16	118.9(3)
C12	C11	C16	118.3(3)	C11	C12	C13	120.5(4)
C12	C13	C14	121.1(4)	C13	C14	C15	120.2(4)
C14	C15	C16	120.5(4)	C11	C16	C15	119.4(3)
C11	C16	C17	119.0(3)	C15	C16	C17	121.6(3)
C16	C17	C18	120.8(3)	C8	C18	C9	120.4(3)
C8	C18	C17	119.5(3)	C9	C18	C17	120.1(3)

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
C2	C3	H3	119.9	C4	C3	H3	119.9
C3	C4	H4	119.4	C5	C4	H4	119.4
C4	C5	H5	120.2	C6	C5	H5	120.1
N1	C7	H7	118.2	C6	C7	H7	118.2
C10	C9	H9	119.9	C18	C9	H9	119.9
C9	C10	H10	119.5	C11	C10	H10	119.6
C11	C12	H12	119.8	C13	C12	H12	119.7
C12	C13	H13	119.5	C14	C13	H13	119.5
C13	C14	H14	119.9	C15	C14	H14	119.9
C14	C15	H15	119.8	C16	C15	H15	119.8
C16	C17	H17	119.6	C18	C17	H17	119.6
O6	C19	H19A	109.5	O6	C19	H19B	109.5
O6	C19	H19C	109.5	H19A	C19	H19B	109.5
H19A	C19	H19C	109.5	H19B	C19	H19C	109.5
S1	C21	H21A	109.5	S1	C21	H21B	109.5
S1	C21	H21C	109.5	H21A	C21	H21B	109.5
H21A	C21	H21C	109.5	H21B	C21	H21C	109.5
S1	C22	H22A	109.5	S1	C22	H22B	109.5
S1	C22	H22C	109.5	H22A	C22	H22B	109.5
H22A	C22	H22C	109.5	H22B	C22	H22C	109.5

Table 8. Torsion Angles(°)

(Those having bond angles > 160 or < 20 degrees are excluded.)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
O1	Mo1	O2	C1	-59.3(3)	O2	Mo1	O1	C8	26.0(3)
O3	Mo1	O1	C8	-94.43(18)	O4	Mo1	O1	C8	158.50(17)
O1	Mo1	O5	S1	57.24(14)	O5	Mo1	O1	C8	76.25(16)
O1	Mo1	N1	N2	1.86(15)	O1	Mo1	N1	C7	-174.4(2)
N1	Mo1	O1	C8	-1.11(15)	O3	Mo1	O2	C1	60.4(2)
O4	Mo1	O2	C1	169.5(2)	O2	Mo1	O5	S1	-145.57(15)
O5	Mo1	O2	C1	-108.6(2)	O2	Mo1	N1	N2	-164.79(18)
O2	Mo1	N1	C7	19.0(2)	N1	Mo1	O2	C1	-33.3(2)
O3	Mo1	N1	N2	96.41(18)	O3	Mo1	N1	C7	-79.8(2)
O4	Mo1	O5	S1	-41.45(15)	O4	Mo1	N1	N2	-61.5(4)
O4	Mo1	N1	C7	122.2(3)	O5	Mo1	N1	N2	-80.43(16)
O5	Mo1	N1	C7	103.3(2)	N1	Mo1	O5	S1	131.32(16)
C21	S1	O5	Mo1	136.3(2)	C22	S1	O5	Mo1	-121.8(3)
Mo1	O1	C8	N2	0.2(4)	Mo1	O1	C8	C18	-179.15(15)
Mo1	O2	C1	C2	-151.15(19)	Mo1	O2	C1	C6	32.5(4)
C19	O6	C2	C1	178.0(3)	C19	O6	C2	C3	-3.2(5)
Mo1	N1	N2	C8	-2.3(3)	Mo1	N1	C7	C6	-4.6(4)
N2	N1	C7	C6	179.1(2)	C7	N1	N2	C8	174.5(2)
N1	N2	C8	O1	1.4(4)	N1	N2	C8	C18	-179.2(2)
O2	C1	C2	O6	2.8(4)	O2	C1	C2	C3	-176.1(3)

O2	C1	C6	C5	175.4(3)	O2	C1	C6	C7	-3.1(5)
C2	C1	C6	C5	-0.8(5)	C2	C1	C6	C7	-179.4(3)
C6	C1	C2	O6	179.3(3)	C6	C1	C2	C3	0.4(5)
O6	C2	C3	C4	-178.7(3)	C1	C2	C3	C4	0.0(5)
C2	C3	C4	C5	0.0(6)	C3	C4	C5	C6	-0.5(6)
C4	C5	C6	C1	0.9(5)	C4	C5	C6	C7	179.5(3)
C1	C6	C7	N1	-8.1(5)	C5	C6	C7	N1	173.3(3)
O1	C8	C18	C9	171.6(2)	O1	C8	C18	C17	-7.0(4)
N2	C8	C18	C9	-7.8(4)	N2	C8	C18	C17	173.6(3)
C10	C9	C18	C8	-174.8(3)	C10	C9	C18	C17	3.7(5)
C18	C9	C10	C11	-1.4(5)	C9	C10	C11	C12	177.8(3)
C9	C10	C11	C16	-2.2(5)	C10	C11	C12	C13	178.2(3)
C10	C11	C16	C15	-177.1(3)	C10	C11	C16	C17	3.6(4)
C12	C11	C16	C15	2.9(4)	C12	C11	C16	C17	-176.4(3)
C16	C11	C12	C13	-1.8(5)	C11	C12	C13	C14	-0.3(5)
C12	C13	C14	C15	1.4(5)	C13	C14	C15	C16	-0.3(5)
C14	C15	C16	C11	-1.9(5)	C14	C15	C16	C17	177.3(3)

Table 8. Torsion angles (°) (continued)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C11	C16	C17	C18	-1.4(5)	C15	C16	C17	C18	179.4(3)
C16	C17	C18	C8	176.3(3)	C16	C17	C18	C9	-2.3(5)

Table 9. Intramolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
Mo1	C6	3.502(3)	S1	O1	3.175(2)
S1	O4	3.141(3)	O1	C17	2.754(4)
O2	O6	2.622(3)	O2	C7	2.906(4)
O3	C1	3.440(4)	O3	C8	3.535(4)
O5	N2	3.286(3)	O5	C8	3.202(4)
N1	C1	2.915(4)	N2	C9	2.848(4)
C1	C4	2.776(5)	C2	C5	2.788(5)
C3	C6	2.783(5)	C3	C19	2.800(5)
C7	C8	3.369(5)	C9	C16	2.804(5)
C10	C17	2.788(5)	C11	C14	2.804(6)
C11	C18	2.804(5)	C12	C15	2.784(6)
C13	C16	2.782(5)			

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
O1	H17	2.425	O1	H22A	3.208
O5	H21A	2.778	O5	H21B	2.841
O5	H21C	3.440	O5	H22A	2.840
O5	H22B	2.740	O5	H22C	3.422
O6	H3	2.641	N2	H7	2.375
N2	H9	2.552	N2	H22A	3.448
C1	H3	3.254	C1	H5	3.265
C1	H7	3.305	C2	H4	3.229
C2	H19A	2.539	C2	H19B	2.668
C2	H19C	3.168	C3	H5	3.235
C3	H19A	2.593	C3	H19B	2.874
C5	H3	3.232	C5	H7	2.574
C6	H4	3.239	C7	H5	2.588
C8	H9	2.671	C8	H17	2.604
C8	H22A	2.934	C9	H17	3.252
C9	H22A	3.449	C10	H12	2.682
C11	H9	3.261	C11	H13	3.254
C11	H15	3.284	C11	H17	3.279
C12	H10	2.679	C12	H14	3.235
C13	H15	3.230	C14	H12	3.239
C15	H13	3.223	C15	H17	2.649

C16	H10	3.280	C16	H12	3.275
C16	H14	3.253	C17	H9	3.246
C17	H15	2.652	C17	H22A	3.439
C18	H10	3.247	C18	H22A	2.976
C19	H3	2.501	C21	H22A	3.501
C21	H22B	2.702	C21	H22C	2.776
C22	H21A	2.681	C22	H21B	3.496
C22	H21C	2.807	H3	H4	2.300
H3	H19A	2.116	H3	H19B	2.473
H3	H19C	3.438	H4	H5	2.295
H5	H7	2.380	H9	H10	2.284
H10	H12	2.532	H12	H13	2.281
H13	H14	2.318	H14	H15	2.286
H15	H17	2.491	H21A	H22A	3.578
H21A	H22B	2.406	H21A	H22C	2.921
H21B	H22B	3.581	H21C	H22B	2.992

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H21C	H22C	2.613			

Table 11. Intermolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
S1	O6[1]	3.523(3)	O1	C5[2]	3.535(4)
O2	C3[3]	3.350(5)	O3	C10[4]	3.272(4)
O3	C12[4]	3.476(5)	O3	C13[5]	3.279(5)
O3	C15[6]	3.428(5)	O4	C5[2]	3.499(4)
O4	C7[2]	3.289(4)	O4	C22[7]	3.470(7)
O5	C3[3]	3.433(4)	O6	S1[7]	3.523(3)
O6	C1[3]	3.535(4)	O6	C5[3]	3.536(4)
O6	C6[3]	3.455(4)	O6	C21[7]	3.289(6)
N1	C11[4]	3.554(4)	N2	C11[4]	3.566(4)
N2	C16[4]	3.531(4)	C1	O6[3]	3.535(4)
C1	C2[3]	3.444(5)	C1	C3[3]	3.559(5)
C2	C1[3]	3.444(5)	C2	C2[3]	3.383(5)
C3	O2[3]	3.350(5)	C3	O5[3]	3.433(4)
C3	C1[3]	3.559(5)	C5	O1[8]	3.535(4)
C5	O4[8]	3.499(4)	C5	O6[3]	3.536(4)
C5	C13[4]	3.450(5)	C6	O6[3]	3.455(4)
C6	C13[4]	3.535(5)	C6	C19[3]	3.397(5)
C7	O4[8]	3.289(4)	C7	C12[4]	3.533(5)
C7	C13[4]	3.509(5)	C7	C19[3]	3.490(5)
C8	C9[4]	3.534(5)	C9	C8[4]	3.534(5)
C9	C18[4]	3.552(5)	C10	O3[4]	3.272(4)

C11	N1[4]	3.554(4)	C11	N2[4]	3.566(4)
C12	O3[4]	3.476(5)	C12	C7[4]	3.533(5)
C13	O3[9]	3.279(5)	C13	C5[4]	3.450(5)
C13	C6[4]	3.535(5)	C13	C7[4]	3.509(5)
C14	C21[1]	3.463(6)	C15	O3[10]	3.428(5)
C16	N2[4]	3.531(4)	C18	C9[4]	3.552(5)
C18	C18[4]	3.548(4)	C19	C6[3]	3.397(5)
C19	C7[3]	3.490(5)	C19	C22[5]	3.448(6)
C21	O6[1]	3.289(6)	C21	C14[7]	3.463(6)
C22	O4[1]	3.470(7)	C22	C19[9]	3.448(6)

Symmetry Operators:

(1) -X+2,Y+1/2,-Z+1/2+1 (2) X,-Y+2,Z+1

(3) -X+2,-Y+1,-Z+1 (4) -X+1,-Y+2,-Z+1

(5) X,Y-1,Z (6) -X+1,Y+1/2-1,-Z+1/2+1

(7) -X+2,Y+1/2-1,-Z+1/2+1 (8) X,-Y+2,Z

(9) X,Y+1,Z (10) -X+1,Y+1/2,-Z+1/2+1

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
Mo1	H5[1]	3.581	S1	H3[2]	3.551
S1	H5[1]	3.387	S1	H19C[3]	3.087
O1	H5[1]	2.645	O1	H14[4]	2.922
O2	H3[2]	3.259	O2	H19A[2]	3.312
O2	H21C[5]	3.291	O3	H10[6]	2.764
O3	H12[7]	3.325	O3	H12[6]	3.051
O3	H13[7]	2.535	O3	H14[4]	3.408
O3	H15[4]	2.718	O4	H5[1]	2.830
O4	H7[1]	2.507	O4	H19C[3]	2.725
O4	H21C[5]	3.497	O4	H22C[5]	2.529
O5	H3[2]	2.653	O5	H19A[2]	2.901
O6	H21B[5]	3.164	O6	H21C[5]	2.864
N1	H19A[2]	3.167	N2	H22B[8]	3.454
C1	H12[7]	3.384	C1	H19A[2]	3.157
C2	H10[7]	3.581	C2	H12[7]	3.376
C3	H10[7]	3.189	C3	H21A[2]	3.554
C3	H21B[2]	3.502	C4	H15[9]	3.365
C4	H17[9]	3.091	C4	H21B[2]	3.371
C5	H13[6]	3.259	C5	H14[6]	3.576
C5	H17[9]	3.073	C5	H19A[2]	3.579
C5	H19C[2]	3.518	C6	H13[6]	3.559

C6	H19A[2]	2.884	C6	H19C[2]	3.401
C7	H19A[2]	2.837	C7	H19C[2]	3.348
C9	H14[10]	3.373	C9	H19B[11]	3.064
C9	H21A[8]	3.277	C10	H3[11]	3.514
C10	H14[10]	3.502	C10	H19B[11]	2.839
C11	H19B[11]	3.067	C12	H12[12]	3.363
C12	H21C[3]	3.464	C13	H5[6]	3.314
C13	H7[6]	3.465	C13	H17[13]	3.584
C13	H21C[3]	3.107	C14	H5[6]	3.154
C14	H7[6]	3.209	C14	H9[14]	3.484
C14	H10[14]	3.564	C14	H17[13]	3.317
C14	H21A[3]	3.277	C14	H21B[3]	3.302
C14	H21C[3]	3.233	C15	H4[1]	3.333
C15	H7[6]	3.219	C15	H10[14]	3.443
C15	H21A[3]	3.525	C15	H21B[3]	3.112
C16	H4[1]	3.355	C16	H7[6]	3.489

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C16	H19B^{11}	3.519	C16	H21B^{3}	3.274
C17	H4^{1}	2.998	C17	H5^{1}	3.398
C17	H9^{6}	3.545	C17	H14^{4}	3.329
C18	H9^{6}	3.586	C18	H19B^{11}	3.453
C19	H21B^{5}	3.142	C19	H21C^{5}	3.525
C19	H22A^{7}	3.061	C19	H22C^{7}	3.125
C21	H3^{2}	3.170	C21	H4^{2}	3.483
C21	H9^{8}	3.494	C21	H19C^{3}	3.176
C22	H19A^{11}	3.492	C22	H19B^{11}	3.245
C22	H19C^{11}	3.054	C22	H22B^{8}	3.486
H3	S1^{2}	3.551	H3	O2^{2}	3.259
H3	O5^{2}	2.653	H3	C10^{7}	3.514
H3	C21^{2}	3.170	H3	H10^{7}	3.091
H3	H21A^{2}	2.782	H3	H21B^{2}	2.949
H4	C15^{9}	3.333	H4	C16^{9}	3.355
H4	C17^{9}	2.998	H4	C21^{2}	3.483
H4	H15^{9}	3.025	H4	H17^{9}	2.605
H4	H21A^{2}	3.543	H4	H21B^{2}	2.676
H4	H22A^{9}	3.384	H4	H22C^{9}	3.422
H5	Mo1^{9}	3.581	H5	S1^{9}	3.387
H5	O1^{9}	2.645	H5	O4^{9}	2.830

H5	C13[6]	3.314	H5	C14[6]	3.154
H5	C17[9]	3.398	H5	H13[6]	3.233
H5	H14[6]	2.941	H5	H17[9]	2.584
H5	H19C[2]	3.511	H7	O4[9]	2.507
H7	C13[6]	3.465	H7	C14[6]	3.209
H7	C15[6]	3.219	H7	C16[6]	3.489
H7	H14[6]	3.543	H7	H15[6]	3.561
H7	H19A[2]	3.146	H7	H19C[2]	3.241
H7	H22C[8]	3.568	H9	C14[10]	3.484
H9	C17[6]	3.545	H9	C18[6]	3.586
H9	C21[8]	3.494	H9	H14[10]	2.798
H9	H17[6]	3.505	H9	H19B[11]	3.476
H9	H21A[8]	2.578	H9	H22B[8]	3.473
H10	O3[6]	2.764	H10	C2[11]	3.581
H10	C3[11]	3.189	H10	C14[10]	3.564
H10	C15[10]	3.443	H10	H3[11]	3.091

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H10	H14[10]	3.059	H10	H15[10]	2.813
H10	H19B[11]	3.120	H10	H21A[8]	3.387
H12	O3[11]	3.325	H12	O3[6]	3.051
H12	C1[11]	3.384	H12	C2[11]	3.376
H12	C12[12]	3.363	H12	H12[12]	2.638
H12	H13[12]	3.276	H13	O3[11]	2.535
H13	C5[6]	3.259	H13	C6[6]	3.559
H13	H5[6]	3.233	H13	H12[12]	3.276
H13	H15[13]	2.925	H13	H17[13]	3.126
H13	H21C[3]	3.310	H14	O1[13]	2.922
H14	O3[13]	3.408	H14	C5[6]	3.576
H14	C9[14]	3.373	H14	C10[14]	3.502
H14	C17[13]	3.329	H14	H5[6]	2.941
H14	H7[6]	3.543	H14	H9[14]	2.798
H14	H10[14]	3.059	H14	H17[13]	2.591
H14	H21A[3]	3.274	H14	H21C[3]	3.523
H15	O3[13]	2.718	H15	C4[1]	3.365
H15	H4[1]	3.025	H15	H7[6]	3.561
H15	H10[14]	2.813	H15	H13[4]	2.925
H15	H21B[3]	3.410	H17	C4[1]	3.091
H17	C5[1]	3.073	H17	C13[4]	3.584

H17	C14[4]	3.317	H17	H4[1]	2.605
H17	H5[1]	2.584	H17	H9[6]	3.505
H17	H13[4]	3.126	H17	H14[4]	2.591
H19A	O2[2]	3.312	H19A	O5[2]	2.901
H19A	N1[2]	3.167	H19A	C1[2]	3.157
H19A	C5[2]	3.579	H19A	C6[2]	2.884
H19A	C7[2]	2.837	H19A	C22[7]	3.492
H19A	H7[2]	3.146	H19A	H22A[7]	3.230
H19A	H22B[7]	3.415	H19A	H22C[7]	3.255
H19B	C9[7]	3.064	H19B	C10[7]	2.839
H19B	C11[7]	3.067	H19B	C16[7]	3.519
H19B	C18[7]	3.453	H19B	C22[7]	3.245
H19B	H9[7]	3.476	H19B	H10[7]	3.120
H19B	H21B[5]	3.132	H19B	H22A[7]	2.620
H19B	H22B[7]	3.514	H19B	H22C[7]	3.146
H19C	S1[5]	3.087	H19C	O4[5]	2.725

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H19C	C5[2]	3.518	H19C	C6[2]	3.401
H19C	C7[2]	3.348	H19C	C21[5]	3.176
H19C	C22[7]	3.054	H19C	H5[2]	3.511
H19C	H7[2]	3.241	H19C	H21B[5]	2.696
H19C	H21C[5]	3.335	H19C	H22A[7]	2.852
H19C	H22B[7]	3.374	H19C	H22C[7]	2.506
H21A	C3[2]	3.554	H21A	C9[8]	3.277
H21A	C14[5]	3.277	H21A	C15[5]	3.525
H21A	H3[2]	2.782	H21A	H4[2]	3.543
H21A	H9[8]	2.578	H21A	H10[8]	3.387
H21A	H14[5]	3.274	H21B	O6[3]	3.164
H21B	C3[2]	3.502	H21B	C4[2]	3.371
H21B	C14[5]	3.302	H21B	C15[5]	3.112
H21B	C16[5]	3.274	H21B	C19[3]	3.142
H21B	H3[2]	2.949	H21B	H4[2]	2.676
H21B	H15[5]	3.410	H21B	H19B[3]	3.132
H21B	H19C[3]	2.696	H21C	O2[3]	3.291
H21C	O4[3]	3.497	H21C	O6[3]	2.864
H21C	C12[5]	3.464	H21C	C13[5]	3.107
H21C	C14[5]	3.233	H21C	C19[3]	3.525
H21C	H13[5]	3.310	H21C	H14[5]	3.523

H21C	H19C[3]	3.335	H22A	C19[11]	3.061
H22A	H4[1]	3.384	H22A	H19A[11]	3.230
H22A	H19B[11]	2.620	H22A	H19C[11]	2.852
H22A	H22B[8]	3.262	H22B	N2[8]	3.454
H22B	C22[8]	3.486	H22B	H9[8]	3.473
H22B	H19A[11]	3.415	H22B	H19B[11]	3.514
H22B	H19C[11]	3.374	H22B	H22A[8]	3.262
H22B	H22B[8]	2.842	H22C	O4[3]	2.529
H22C	C19[11]	3.125	H22C	H4[1]	3.422
H22C	H7[8]	3.568	H22C	H19A[11]	3.255
H22C	H19B[11]	3.146	H22C	H19C[11]	2.506

Symmetry Operators:

(1) X,-Y+2,Z+1 (2) -X+2,-Y+1,-Z+1

(3) -X+2,Y+1/2,-Z+1/2+1 (4) -X+1,Y+1/2-1,-Z+1/2+1

(5) -X+2,Y+1/2-1,-Z+1/2+1 (6) -X+1,-Y+2,-Z+1

(7) X,Y-1,Z (8) -X+2,-Y+2,-Z+1

(9) X,-Y+2,Z (10) X,-Y+3,Z

(11) X,Y+1,Z (12) -X+1,-Y+3,-Z+1

(13) -X+1,Y+1/2,-Z+1/2+1 (14) X,-Y+3,Z+1

Appendix C: Supplementary Information for Cu-1

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{26}H_{36}Cl_2CuN_2O_2$
Formula Weight	543.03
Crystal Color, Habit	black, chunk
Crystal Dimensions	0.200 X 0.160 X 0.120 mm
Crystal System	triclinic
Lattice Type	Primitive
Lattice Parameters	a = 14.680(9) Å b = 21.224(13) Å c = 18.956(11) Å

	α = 90.00000 ^{o}
	β = 110.277(13) ^{o}
	γ = 90.00000 ^{o}
	V = 5540(6) Å^{3}
Space Group	P-1 (#2)
Z value	8
D_{calc}	1.302 g/cm^{3}
F_{000}	2280.00
μ(MoKα)	10.050 cm^{-1}

B. Intensity Measurements

Diffractometer	XtaLAB mini
Radiation	MoKα (λ = 0.71075 Å) graphite monochromated
Voltage, Current	50kV, 12mA
Temperature	20.0^{o}C
Detector Aperture	75.0 mm (diameter)
Data Images	540 exposures
ω oscillation Range (χ=54.0, ϕ=0.0)	-60.0 - 120.0^{o}
Exposure Rate	10.0 sec./^{o}
Detector Swing Angle	29.50^{o}
ω oscillation Range (χ=54.0, ϕ=120.0)	-60.0 - 120.0^{o}

Exposure Rate	10.0 sec./^{o}
Detector Swing Angle	29.50^{o}
ω oscillation Range (χ=54.0, ϕ=240.0)	-60.0 - 120.0^{o}
Exposure Rate	10.0 sec./^{o}
Detector Swing Angle	29.50^{o}
Detector Position	50.00 mm
Pixel Size	0.073 mm
$2\theta_{max}$	55.0^{o}
No. of Reflections Measured	Total: 51897 Unique: 12593 (R_{int} = 0.2753)
Corrections	Lorentz-polarization Absorption (*trans*. factors: 0.510 - 0.886)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SHELXS97)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	w = 1/ [$\sigma^2(Fo^2)$ + $(0.0922 \cdot P)^2$ + $0.0000 \cdot$ P] where P = $(Max(Fo^2,0) + 2Fc^2)/3$
$2\theta_{max}$ cutoff	55.0^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	12593
No. Variables	611
Reflection/Parameter Ratio	20.61

Residuals: R1 (I>2.00σ(I))	0.0977
Residuals: R (All reflections)	0.3114
Residuals: wR2 (All reflections)	0.2621
Goodness of Fit Indicator	0.973
Max Shift/Error in Final Cycle	0.001
Maximum peak in Final Diff. Map	0.62 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.34 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
Cu1	0.09583(7)	0.52146(4)	0.65734(5)	4.30(3)
Cu2	0.39381(7)	0.01870(4)	0.35286(5)	4.08(3)
Cl1	0.3886(2)	0.42436(12)	0.44794(19)	8.96(9)
Cl2	-0.0069(2)	0.72822(13)	0.92389(17)	9.34(9)
Cl3	0.1995(2)	-0.06503(13)	0.63790(17)	8.81(8)
Cl4	0.5127(2)	0.22116(11)	0.09714(14)	7.56(7)
O1	0.1236(4)	0.5510(2)	0.5735(3)	4.85(12)
O2	0.1350(4)	0.5968(2)	0.7116(3)	5.50(13)
O3	0.2893(4)	0.0413(2)	0.3842(3)	5.49(13)
O4	0.4304(4)	0.1028(2)	0.3489(3)	5.19(13)
N1	0.1011(5)	0.4287(3)	0.6311(4)	4.75(15)
N2	0.0085(5)	0.4966(3)	0.7156(4)	4.82(15)
N3	0.4011(5)	-0.0723(3)	0.3926(4)	4.31(14)
N4	0.4595(5)	-0.0123(3)	0.2816(4)	4.67(15)
C1	0.1825(6)	0.5209(4)	0.5437(5)	4.42(17)
C2	0.2494(6)	0.5571(4)	0.5208(4)	4.49(18)
C3	0.3079(6)	0.5245(4)	0.4912(4)	5.20(19)
C4	0.3095(7)	0.4598(4)	0.4872(5)	5.6(2)
C5	0.2441(7)	0.4218(4)	0.5085(5)	5.2(2)
C6	0.1789(6)	0.4547(4)	0.5368(5)	4.71(18)
C7	0.2416(7)	0.3499(4)	0.5003(6)	7.6(3)

C8	0.2434(8)	0.6291(4)	0.5245(5)	6.2(2)
C9	0.2593(10)	0.6615(5)	0.4610(7)	11.5(4)
C10	0.3034(11)	0.6544(5)	0.5959(6)	14.1(6)
C11	0.0952(7)	0.4199(4)	0.5530(5)	5.9(2)
C12	0.1886(6)	0.3966(4)	0.6843(5)	6.1(2)
C13	0.0108(7)	0.4028(4)	0.6410(6)	6.4(2)
C14	0.0021(7)	0.4266(4)	0.7128(5)	6.2(2)
C15	-0.0911(6)	0.5257(4)	0.6784(5)	6.8(2)
C16	0.0518(6)	0.5199(4)	0.7950(5)	5.7(2)
C17	0.0551(6)	0.5907(4)	0.8022(5)	5.5(2)
C18	0.0974(6)	0.6264(4)	0.7579(5)	4.92(19)
C19	0.1029(6)	0.6925(4)	0.7632(5)	5.2(2)
C20	0.0703(6)	0.7224(4)	0.8142(5)	5.5(2)
C21	0.0304(7)	0.6878(5)	0.8576(5)	6.2(2)
C22	0.0237(6)	0.6214(5)	0.8556(5)	5.5(2)
C23	-0.0165(7)	0.5844(5)	0.9080(6)	8.1(3)

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
C24	0.1505(8)	0.7268(4)	0.7134(5)	6.5(2)
C25	0.0787(9)	0.7376(7)	0.6375(7)	13.5(5)
C26	0.1998(13)	0.7848(6)	0.7462(7)	15.9(6)
C27	0.2731(6)	0.0153(4)	0.4437(5)	4.38(17)
C28	0.2500(6)	0.0566(4)	0.4924(5)	4.85(19)
C29	0.2278(6)	0.0294(4)	0.5509(6)	5.9(2)
C30	0.2323(6)	-0.0355(4)	0.5621(5)	5.5(2)
C31	0.2588(6)	-0.0767(4)	0.5152(5)	4.91(19)
C32	0.2800(6)	-0.0498(4)	0.4550(5)	4.58(18)
C33	0.2659(7)	-0.1475(4)	0.5301(6)	8.1(3)
C34	0.2470(7)	0.1274(4)	0.4804(5)	5.1(2)
C35	0.2665(7)	0.1661(4)	0.5515(6)	7.3(3)
C36	0.1536(8)	0.1476(4)	0.4221(5)	8.6(3)
C37	0.3059(6)	-0.0897(4)	0.3985(5)	5.4(2)
C38	0.4798(6)	-0.0814(4)	0.4668(4)	5.5(2)
C39	0.4192(7)	-0.1099(4)	0.3327(5)	5.9(2)
C40	0.4955(7)	-0.0768(4)	0.3082(5)	5.7(2)
C41	0.3892(7)	-0.0159(4)	0.2022(5)	6.4(2)
C42	0.5407(6)	0.0321(4)	0.2868(5)	5.7(2)
C43	0.5074(6)	0.0959(4)	0.2570(5)	5.2(2)
C44	0.4506(6)	0.1298(4)	0.2927(5)	4.86(19)

C45	0.4172(6)	0.1911(4)	0.2690(5)	5.02(19)
C46	0.4385(7)	0.2179(4)	0.2093(5)	5.3(2)
C47	0.4925(7)	0.1838(4)	0.1742(5)	5.8(2)
C48	0.5266(7)	0.1236(4)	0.1942(5)	5.8(2)
C49	0.5837(8)	0.0873(5)	0.1538(6)	8.5(3)
C50	0.3598(7)	0.2257(4)	0.3096(5)	5.6(2)
C51	0.4278(8)	0.2482(5)	0.3868(6)	8.7(3)
C52	0.2997(8)	0.2804(5)	0.2668(6)	9.2(3)

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B_{iso} involving hydrogen atoms

atom	x	y	z	B_{iso}
H3	0.34894	0.54736	0.47273	6.238
H7A	0.20747	0.33878	0.44881	9.119
H7B	0.20894	0.33193	0.53154	9.119
H7C	0.30677	0.33401	0.51556	9.119
H8	0.17626	0.63854	0.52002	7.457
H9A	0.32780	0.66505	0.47059	13.761
H9B	0.23104	0.70279	0.45523	13.761
H9C	0.22959	0.63775	0.41566	13.761
H10A	0.37038	0.65174	0.60027	16.936
H10B	0.29330	0.63080	0.63575	16.936
H10C	0.28641	0.69774	0.59931	16.936
H11A	0.03335	0.43567	0.51951	7.075
H11B	0.09847	0.37523	0.54310	7.075
H12A	0.24573	0.41165	0.67612	7.375
H12B	0.18286	0.35190	0.67593	7.375
H12C	0.19332	0.40548	0.73514	7.375
H13A	-0.04574	0.41556	0.59882	7.694
H13B	0.01348	0.35715	0.64190	7.694
H14A	-0.05963	0.41339	0.71603	7.406
H14B	0.05359	0.40888	0.75529	7.406
H15A	-0.13189	0.51526	0.70676	8.218

H15B	-0.11945	0.50959	0.62814	8.218
H15C	-0.08494	0.57066	0.67659	8.218
H16A	0.11735	0.50356	0.81666	6.889
H16B	0.01430	0.50305	0.82393	6.889
H20	0.07527	0.76594	0.81943	6.658
H23A	-0.08361	0.57451	0.88179	9.748
H23B	-0.01082	0.60947	0.95143	9.748
H23C	0.01971	0.54609	0.92341	9.748
H24	0.19994	0.69835	0.70753	7.792
H25A	0.11127	0.75283	0.60487	16.198
H25B	0.03191	0.76822	0.64031	16.198
H25C	0.04615	0.69876	0.61798	16.198
H26A	0.15301	0.81489	0.75034	19.029
H26B	0.23276	0.80169	0.71460	19.029
H26C	0.24610	0.77627	0.79523	19.029
H29	0.20937	0.05507	0.58341	7.061

Table 2. Atomic coordinates and B_{iso} involving hydrogens/B_{eq} (continued)

atom	x	y	z	B_{eq}
H33A	0.22008	-0.16907	0.48826	9.663
H33B	0.25164	-0.15627	0.57489	9.663
H33C	0.33038	-0.16168	0.53660	9.663
H34	0.29875	0.13755	0.46061	6.158
H35A	0.32303	0.15004	0.59023	8.793
H35B	0.21154	0.16339	0.56784	8.793
H35C	0.27710	0.20929	0.54150	8.793
H36A	0.10081	0.13842	0.43937	10.369
H36B	0.14448	0.12519	0.37616	10.369
H36C	0.15563	0.19202	0.41344	10.369
H37A	0.30763	-0.13360	0.41294	6.502
H37B	0.25567	-0.08496	0.34953	6.502
H38A	0.54081	-0.06868	0.46315	6.572
H38B	0.48310	-0.12510	0.48071	6.572
H38C	0.46629	-0.05644	0.50415	6.572
H39A	0.35934	-0.11428	0.29004	7.082
H39B	0.44171	-0.15169	0.35144	7.082
H40A	0.55635	-0.07426	0.35010	6.872
H40B	0.50645	-0.10033	0.26798	6.872
H41A	0.33066	-0.03640	0.20175	7.672
H41B	0.41775	-0.03957	0.17198	7.672

H41C	0.37421	0.02584	0.18209	7.672
H42A	0.58237	0.03572	0.33902	6.869
H42B	0.57928	0.01442	0.25910	6.869
H46	0.41685	0.25831	0.19288	6.411
H49A	0.60403	0.11564	0.12265	10.192
H49B	0.54314	0.05503	0.12299	10.192
H49C	0.63967	0.06828	0.19016	10.192
H50	0.31470	0.19523	0.31846	6.678
H51A	0.47346	0.27793	0.38025	10.459
H51B	0.46209	0.21279	0.41529	10.459
H51C	0.39027	0.26799	0.41327	10.459
H52A	0.25897	0.26643	0.21784	11.086
H52B	0.34203	0.31315	0.26163	11.086
H52C	0.25998	0.29629	0.29375	11.086

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cu1	0.0577(7)	0.0410(6)	0.0619(7)	-0.0058(5)	0.0173(6)	0.0012(5)
Cu2	0.0612(7)	0.0377(6)	0.0529(6)	0.0063(5)	0.0156(5)	-0.0009(5)
Cl1	0.134(3)	0.0752(19)	0.162(3)	0.0237(17)	0.091(2)	-0.0045(18)
Cl2	0.168(3)	0.086(2)	0.143(3)	0.0057(19)	0.107(3)	-0.0139(18)
Cl3	0.129(3)	0.100(2)	0.135(2)	0.0113(18)	0.082(2)	0.0376(19)
Cl4	0.158(3)	0.0668(17)	0.0812(17)	-0.0099(16)	0.0650(18)	0.0032(13)
O1	0.078(4)	0.040(3)	0.070(4)	0.004(3)	0.030(3)	0.011(3)
O2	0.091(5)	0.049(4)	0.080(4)	-0.021(3)	0.043(4)	-0.014(3)
O3	0.075(4)	0.047(4)	0.101(5)	0.018(3)	0.048(4)	0.009(3)
O4	0.101(5)	0.040(3)	0.069(4)	0.002(3)	0.045(4)	-0.003(3)
N1	0.056(5)	0.059(5)	0.055(5)	-0.008(4)	0.007(4)	0.002(4)
N2	0.064(5)	0.051(5)	0.065(5)	-0.016(4)	0.018(4)	0.004(4)
N3	0.069(5)	0.025(4)	0.067(5)	0.008(3)	0.021(4)	-0.002(3)
N4	0.074(5)	0.043(4)	0.052(4)	0.019(4)	0.011(4)	-0.002(3)
C1	0.073(6)	0.036(5)	0.058(5)	0.007(5)	0.022(5)	-0.002(4)
C2	0.078(7)	0.047(5)	0.054(5)	0.003(5)	0.033(5)	0.003(4)
C3	0.076(7)	0.066(6)	0.056(5)	0.003(5)	0.024(5)	-0.000(5)
C4	0.085(7)	0.053(6)	0.083(7)	0.009(5)	0.038(6)	-0.008(5)
C5	0.081(7)	0.041(6)	0.068(6)	-0.004(5)	0.016(6)	0.004(5)
C6	0.063(6)	0.049(6)	0.064(6)	0.002(5)	0.019(5)	0.001(5)
C7	0.117(9)	0.042(6)	0.148(10)	0.015(6)	0.069(8)	-0.003(6)

C8	0.122(9)	0.051(6)	0.088(7)	-0.026(6)	0.068(7)	-0.016(5)
C9	0.233(16)	0.076(9)	0.146(11)	0.001(9)	0.090(12)	0.012(8)
C10	0.281(19)	0.048(7)	0.117(10)	-0.004(9)	-0.045(11)	0.003(7)
C11	0.093(8)	0.037(5)	0.084(7)	-0.011(5)	0.019(6)	-0.011(5)
C12	0.078(7)	0.062(6)	0.074(6)	-0.005(5)	0.002(6)	0.005(5)
C13	0.100(8)	0.051(6)	0.099(8)	-0.031(5)	0.043(7)	0.005(5)
C14	0.094(8)	0.052(6)	0.096(7)	-0.031(5)	0.043(7)	-0.009(5)
C15	0.061(7)	0.111(8)	0.081(7)	-0.005(6)	0.015(6)	0.002(6)
C16	0.069(6)	0.070(7)	0.079(7)	-0.016(5)	0.025(6)	0.020(6)
C17	0.065(7)	0.060(6)	0.083(7)	0.001(5)	0.025(6)	0.011(5)
C18	0.057(6)	0.065(7)	0.055(6)	-0.002(5)	0.007(5)	-0.002(5)
C19	0.080(7)	0.044(6)	0.076(6)	-0.011(5)	0.033(6)	0.001(5)
C20	0.083(7)	0.060(6)	0.080(7)	0.011(5)	0.043(6)	0.001(5)
C21	0.083(7)	0.060(7)	0.091(7)	0.005(5)	0.030(6)	-0.011(6)
C22	0.060(6)	0.075(7)	0.085(7)	-0.000(5)	0.037(6)	0.003(6)
C23	0.118(9)	0.104(9)	0.118(9)	-0.017(7)	0.081(8)	-0.005(7)

Table 3. Anisotropic displacement parameters (continued)

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C24	0.133(10)	0.043(6)	0.072(7)	-0.007(6)	0.038(7)	0.001(5)
C25	0.146(13)	0.225(17)	0.141(12)	0.014(11)	0.049(11)	0.093(12)
C26	0.37(2)	0.146(13)	0.152(13)	-0.093(15)	0.169(16)	-0.012(11)
C27	0.055(6)	0.046(6)	0.067(6)	-0.001(5)	0.024(5)	-0.000(5)
C28	0.066(7)	0.042(5)	0.081(6)	-0.001(4)	0.031(6)	0.004(5)
C29	0.070(7)	0.047(6)	0.114(8)	0.015(5)	0.041(6)	0.004(6)
C30	0.069(7)	0.069(7)	0.083(7)	-0.010(5)	0.040(6)	0.008(5)
C31	0.056(6)	0.049(6)	0.081(7)	-0.010(4)	0.023(5)	0.004(5)
C32	0.066(6)	0.041(5)	0.067(6)	-0.002(4)	0.023(5)	0.002(5)
C33	0.116(9)	0.063(7)	0.138(10)	-0.014(6)	0.059(8)	0.011(6)
C34	0.082(7)	0.045(6)	0.080(7)	0.009(5)	0.044(6)	-0.006(5)
C35	0.100(8)	0.047(6)	0.121(9)	0.008(5)	0.025(7)	-0.014(6)
C36	0.120(10)	0.085(8)	0.094(8)	0.045(7)	0.000(8)	0.003(6)
C37	0.063(7)	0.046(5)	0.092(7)	0.001(5)	0.019(6)	-0.004(5)
C38	0.057(6)	0.064(6)	0.067(6)	0.010(5)	-0.005(5)	0.004(5)
C39	0.099(8)	0.040(5)	0.086(7)	0.008(5)	0.032(6)	-0.010(5)
C40	0.082(7)	0.058(6)	0.079(6)	0.028(5)	0.029(6)	0.002(5)
C41	0.095(8)	0.070(7)	0.066(6)	0.011(6)	0.013(6)	-0.017(5)
C42	0.067(7)	0.072(7)	0.086(7)	-0.001(5)	0.037(6)	-0.016(5)
C43	0.078(7)	0.042(5)	0.078(6)	0.005(5)	0.026(6)	-0.003(5)
C44	0.071(7)	0.051(6)	0.060(6)	-0.001(5)	0.019(5)	-0.002(5)

C45	0.090(7)	0.038(5)	0.069(6)	0.001(5)	0.035(6)	0.004(5)
C46	0.101(8)	0.043(5)	0.070(6)	-0.004(5)	0.042(6)	0.003(5)
C47	0.113(8)	0.054(6)	0.056(6)	-0.019(6)	0.033(6)	0.008(5)
C48	0.091(8)	0.061(7)	0.079(7)	-0.004(5)	0.041(6)	0.010(5)
C49	0.169(12)	0.081(8)	0.111(8)	0.023(7)	0.098(9)	0.005(6)
C50	0.102(8)	0.046(6)	0.078(6)	0.006(5)	0.048(6)	-0.012(5)
C51	0.148(11)	0.091(8)	0.106(9)	0.005(7)	0.061(9)	-0.025(7)
C52	0.147(11)	0.086(8)	0.128(9)	0.040(8)	0.061(9)	-0.002(7)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Fragment Analysis

fragment: 1

Cu(1)	Cl(1)	Cl(2)	O(1)	O(2)
N(1)	N(2)	C(1)	C(2)	C(3)
C(4)	C(5)	C(6)	C(7)	C(8)
C(9)	C(10)	C(11)	C(12)	C(13)
C(14)	C(15)	C(16)	C(17)	C(18)
C(19)	C(20)	C(21)	C(22)	C(23)
C(24)	C(25)	C(26)		

fragment: 2

Cu(2)	Cl(3)	Cl(4)	O(3)	O(4)
N(3)	N(4)	C(27)	C(28)	C(29)
C(30)	C(31)	C(32)	C(33)	C(34)
C(35)	C(36)	C(37)	C(38)	C(39)
C(40)	C(41)	C(42)	C(43)	C(44)
C(45)	C(46)	C(47)	C(48)	C(49)
C(50)	C(51)	C(52)		

Table 5. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
Cu1	O1	1.880(6)	Cu1	O2	1.879(5)
Cu1	N1	2.038(7)	Cu1	N2	2.030(9)
Cu2	O3	1.889(7)	Cu2	O4	1.873(5)
Cu2	N3	2.063(6)	Cu2	N4	2.022(8)
Cl1	C4	1.751(12)	Cl2	C21	1.758(12)
Cl3	C30	1.780(11)	Cl4	C47	1.774(10)
O1	C1	1.346(12)	O2	C18	1.344(12)
O3	C27	1.348(12)	O4	C44	1.330(12)
N1	C11	1.466(13)	N1	C12	1.495(10)
N1	C13	1.504(13)	N2	C14	1.489(10)
N2	C15	1.517(11)	N2	C16	1.499(11)
N3	C37	1.487(13)	N3	C38	1.491(9)
N3	C39	1.484(12)	N4	C40	1.490(10)
N4	C41	1.504(9)	N4	C42	1.496(12)
C1	C2	1.428(14)	C1	C6	1.411(11)
C2	C3	1.364(14)	C2	C8	1.534(12)
C3	C4	1.376(12)	C4	C5	1.416(15)
C5	C6	1.431(15)	C5	C7	1.533(11)
C6	C11	1.552(14)	C8	C9	1.475(18)
C8	C10	1.440(14)	C13	C14	1.499(15)
C16	C17	1.508(13)	C17	C18	1.426(15)

C17	C22	1.408(15)	C18	C19	1.406(12)
C19	C20	1.374(15)	C19	C24	1.537(16)
C20	C21	1.375(15)	C21	C22	1.414(14)
C22	C23	1.535(16)	C24	C25	1.478(14)
C24	C26	1.455(16)	C27	C28	1.398(14)
C27	C32	1.396(12)	C28	C29	1.385(15)
C28	C34	1.519(11)	C29	C30	1.391(13)
C30	C31	1.396(14)	C31	C32	1.404(14)
C31	C33	1.525(12)	C32	C37	1.514(14)
C34	C35	1.519(13)	C34	C36	1.494(12)
C39	C40	1.524(15)	C42	C43	1.485(12)
C43	C44	1.438(15)	C43	C48	1.440(15)
C44	C45	1.407(11)	C45	C46	1.395(14)
C45	C50	1.514(15)	C46	C47	1.400(15)
C47	C48	1.377(13)	C48	C49	1.526(16)
C50	C51	1.535(12)	C50	C52	1.512(13)

Table 6. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C3	H3	0.930	C7	H7A	0.960
C7	H7B	0.960	C7	H7C	0.960
C8	H8	0.980	C9	H9A	0.960
C9	H9B	0.960	C9	H9C	0.960
C10	H10A	0.960	C10	H10B	0.960
C10	H10C	0.960	C11	H11A	0.970
C11	H11B	0.970	C12	H12A	0.960
C12	H12B	0.960	C12	H12C	0.960
C13	H13A	0.970	C13	H13B	0.970
C14	H14A	0.970	C14	H14B	0.970
C15	H15A	0.960	C15	H15B	0.960
C15	H15C	0.960	C16	H16A	0.970
C16	H16B	0.970	C20	H20	0.930
C23	H23A	0.960	C23	H23B	0.960
C23	H23C	0.960	C24	H24	0.980
C25	H25A	0.960	C25	H25B	0.960
C25	H25C	0.960	C26	H26A	0.960
C26	H26B	0.960	C26	H26C	0.960
C29	H29	0.930	C33	H33A	0.960
C33	H33B	0.960	C33	H33C	0.960
C34	H34	0.980	C35	H35A	0.960

C35	H35B	0.960	C35	H35C	0.960
C36	H36A	0.960	C36	H36B	0.960
C36	H36C	0.960	C37	H37A	0.970
C37	H37B	0.970	C38	H38A	0.960
C38	H38B	0.960	C38	H38C	0.960
C39	H39A	0.970	C39	H39B	0.970
C40	H40A	0.970	C40	H40B	0.970
C41	H41A	0.960	C41	H41B	0.960
C41	H41C	0.960	C42	H42A	0.970
C42	H42B	0.970	C46	H46	0.930
C49	H49A	0.960	C49	H49B	0.960
C49	H49C	0.960	C50	H50	0.980
C51	H51A	0.960	C51	H51B	0.960
C51	H51C	0.960	C52	H52A	0.960
C52	H52B	0.960	C52	H52C	0.960

Table 7. Bond angles (^{o})

atom	atom	atom	angle	atom	atom	atom	angle
O1	Cu1	O2	93.7(3)	O1	Cu1	N1	94.5(3)
O1	Cu1	N2	155.4(2)	O2	Cu1	N1	157.6(2)
O2	Cu1	N2	93.1(3)	N1	Cu1	N2	88.1(3)
O3	Cu2	O4	92.7(3)	O3	Cu2	N3	93.9(3)
O3	Cu2	N4	156.9(2)	O4	Cu2	N3	158.1(2)
O4	Cu2	N4	94.2(3)	N3	Cu2	N4	87.8(3)
Cu1	O1	C1	123.8(5)	Cu1	O2	C18	129.7(6)
Cu2	O3	C27	123.9(5)	Cu2	O4	C44	126.1(5)
Cu1	N1	C11	112.1(5)	Cu1	N1	C12	111.9(5)
Cu1	N1	C13	102.3(5)	C11	N1	C12	111.0(7)
C11	N1	C13	109.4(6)	C12	N1	C13	109.7(7)
Cu1	N2	C14	106.5(6)	Cu1	N2	C15	109.3(6)
Cu1	N2	C16	109.4(5)	C14	N2	C15	110.5(6)
C14	N2	C16	111.3(6)	C15	N2	C16	109.8(7)
Cu2	N3	C37	109.1(5)	Cu2	N3	C38	113.0(4)
Cu2	N3	C39	103.0(5)	C37	N3	C38	109.8(7)
C37	N3	C39	110.3(6)	C38	N3	C39	111.5(6)
Cu2	N4	C40	105.6(6)	Cu2	N4	C41	111.3(6)
Cu2	N4	C42	107.8(5)	C40	N4	C41	109.3(6)
C40	N4	C42	112.1(7)	C41	N4	C42	110.6(7)
O1	C1	C2	118.9(7)	O1	C1	C6	120.1(8)

C2	C1	C6	121.0(9)	C1	C2	C3	116.8(8)
C1	C2	C8	117.6(9)	C3	C2	C8	125.3(9)
C2	C3	C4	123.5(9)	Cl1	C4	C3	118.6(8)
Cl1	C4	C5	119.4(7)	C3	C4	C5	121.8(10)
C4	C5	C6	116.0(8)	C4	C5	C7	122.2(9)
C6	C5	C7	121.8(9)	C1	C6	C5	120.8(9)
C1	C6	C11	117.6(8)	C5	C6	C11	121.4(7)
C2	C8	C9	113.5(9)	C2	C8	C10	112.9(7)
C9	C8	C10	112.0(9)	N1	C11	C6	111.4(6)
N1	C13	C14	110.2(7)	N2	C14	C13	110.1(8)
N2	C16	C17	114.1(7)	C16	C17	C18	118.9(9)
C16	C17	C22	121.2(9)	C18	C17	C22	119.7(8)
O2	C18	C17	119.8(8)	O2	C18	C19	119.2(9)
C17	C18	C19	120.9(9)	C18	C19	C20	119.1(9)
C18	C19	C24	117.1(9)	C20	C19	C24	123.8(8)
C19	C20	C21	120.0(9)	Cl2	C21	C20	118.1(7)

Table 7. Bond angles (^{o}) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
Cl2	C21	C22	118.0(9)	C20	C21	C22	123.8(10)
C17	C22	C21	116.4(10)	C17	C22	C23	121.5(9)
C21	C22	C23	122.1(10)	C19	C24	C25	110.4(10)
C19	C24	C26	113.8(10)	C25	C24	C26	111.4(9)
O3	C27	C28	116.7(7)	O3	C27	C32	120.6(8)
C28	C27	C32	122.7(9)	C27	C28	C29	116.4(8)
C27	C28	C34	121.5(9)	C29	C28	C34	122.1(9)
C28	C29	C30	121.5(10)	Cl3	C30	C29	117.4(8)
Cl3	C30	C31	120.4(7)	C29	C30	C31	122.2(10)
C30	C31	C32	116.8(8)	C30	C31	C33	121.0(9)
C32	C31	C33	122.1(9)	C27	C32	C31	120.2(9)
C27	C32	C37	117.8(8)	C31	C32	C37	121.9(7)
C28	C34	C35	114.5(8)	C28	C34	C36	111.4(7)
C35	C34	C36	110.1(8)	N3	C37	C32	112.9(6)
N3	C39	C40	109.3(7)	N4	C40	C39	108.3(8)
N4	C42	C43	113.6(7)	C42	C43	C44	116.5(9)
C42	C43	C48	123.7(9)	C44	C43	C48	119.8(8)
O4	C44	C43	119.2(7)	O4	C44	C45	120.4(9)
C43	C44	C45	120.4(9)	C44	C45	C46	119.1(9)
C44	C45	C50	118.6(9)	C46	C45	C50	122.3(7)
C45	C46	C47	119.8(8)	Cl4	C47	C46	116.4(7)

Cl4	C47	C48	119.5(8)	C46	C47	C48	124.1(9)
C43	C48	C47	116.8(10)	C43	C48	C49	120.5(8)
C47	C48	C49	122.8(10)	C45	C50	C51	110.1(8)
C45	C50	C52	115.1(9)	C51	C50	C52	109.7(7)

Table 8. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
C2	C3	H3	118.2	C4	C3	H3	118.2
C5	C7	H7A	109.5	C5	C7	H7B	109.5
C5	C7	H7C	109.5	H7A	C7	H7B	109.5
H7A	C7	H7C	109.5	H7B	C7	H7C	109.5
C2	C8	H8	105.9	C9	C8	H8	105.9
C10	C8	H8	105.9	C8	C9	H9A	109.5
C8	C9	H9B	109.5	C8	C9	H9C	109.5
H9A	C9	H9B	109.5	H9A	C9	H9C	109.5
H9B	C9	H9C	109.5	C8	C10	H10A	109.5
C8	C10	H10B	109.5	C8	C10	H10C	109.5
H10A	C10	H10B	109.5	H10A	C10	H10C	109.5
H10B	C10	H10C	109.5	N1	C11	H11A	109.3
N1	C11	H11B	109.3	C6	C11	H11A	109.3
C6	C11	H11B	109.3	H11A	C11	H11B	108.0
N1	C12	H12A	109.5	N1	C12	H12B	109.5
N1	C12	H12C	109.5	H12A	C12	H12B	109.5
H12A	C12	H12C	109.5	H12B	C12	H12C	109.5
N1	C13	H13A	109.6	N1	C13	H13B	109.6
C14	C13	H13A	109.6	C14	C13	H13B	109.6
H13A	C13	H13B	108.1	N2	C14	H14A	109.6
N2	C14	H14B	109.6	C13	C14	H14A	109.6

C13	C14	H14B	109.6	H14A	C14	H14B	108.2
N2	C15	H15A	109.5	N2	C15	H15B	109.5
N2	C15	H15C	109.5	H15A	C15	H15B	109.5
H15A	C15	H15C	109.5	H15B	C15	H15C	109.5
N2	C16	H16A	108.7	N2	C16	H16B	108.7
C17	C16	H16A	108.7	C17	C16	H16B	108.7
H16A	C16	H16B	107.6	C19	C20	H20	120.0
C21	C20	H20	120.0	C22	C23	H23A	109.5
C22	C23	H23B	109.5	C22	C23	H23C	109.5
H23A	C23	H23B	109.5	H23A	C23	H23C	109.5
H23B	C23	H23C	109.5	C19	C24	H24	106.9
C25	C24	H24	106.9	C26	C24	H24	106.9
C24	C25	H25A	109.5	C24	C25	H25B	109.5
C24	C25	H25C	109.5	H25A	C25	H25B	109.5
H25A	C25	H25C	109.5	H25B	C25	H25C	109.5
C24	C26	H26A	109.5	C24	C26	H26B	109.5

Table 8. Bond angles involving hydrogens (^{o}) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C24	C26	H26C	109.5	H26A	C26	H26B	109.5
H26A	C26	H26C	109.5	H26B	C26	H26C	109.5
C28	C29	H29	119.2	C30	C29	H29	119.2
C31	C33	H33A	109.5	C31	C33	H33B	109.5
C31	C33	H33C	109.5	H33A	C33	H33B	109.5
H33A	C33	H33C	109.5	H33B	C33	H33C	109.5
C28	C34	H34	106.8	C35	C34	H34	106.8
C36	C34	H34	106.8	C34	C35	H35A	109.5
C34	C35	H35B	109.5	C34	C35	H35C	109.5
H35A	C35	H35B	109.5	H35A	C35	H35C	109.5
H35B	C35	H35C	109.5	C34	C36	H36A	109.5
C34	C36	H36B	109.5	C34	C36	H36C	109.5
H36A	C36	H36B	109.5	H36A	C36	H36C	109.5
H36B	C36	H36C	109.5	N3	C37	H37A	109.0
N3	C37	H37B	109.0	C32	C37	H37A	109.0
C32	C37	H37B	109.0	H37A	C37	H37B	107.8
N3	C38	H38A	109.5	N3	C38	H38B	109.5
N3	C38	H38C	109.5	H38A	C38	H38B	109.5
H38A	C38	H38C	109.5	H38B	C38	H38C	109.5
N3	C39	H39A	109.8	N3	C39	H39B	109.8
C40	C39	H39A	109.8	C40	C39	H39B	109.8

H39A	C39	H39B	108.3	N4	C40	H40A	110.0
N4	C40	H40B	110.0	C39	C40	H40A	110.0
C39	C40	H40B	110.0	H40A	C40	H40B	108.4
N4	C41	H41A	109.5	N4	C41	H41B	109.5
N4	C41	H41C	109.5	H41A	C41	H41B	109.5
H41A	C41	H41C	109.5	H41B	C41	H41C	109.5
N4	C42	H42A	108.8	N4	C42	H42B	108.8
C43	C42	H42A	108.9	C43	C42	H42B	108.9
H42A	C42	H42B	107.7	C45	C46	H46	120.1
C47	C46	H46	120.1	C48	C49	H49A	109.5
C48	C49	H49B	109.5	C48	C49	H49C	109.5
H49A	C49	H49B	109.5	H49A	C49	H49C	109.5
H49B	C49	H49C	109.5	C45	C50	H50	107.2
C51	C50	H50	107.2	C52	C50	H50	107.2
C50	C51	H51A	109.5	C50	C51	H51B	109.5
C50	C51	H51C	109.5	H51A	C51	H51B	109.5

Table 8. Bond angles involving hydrogens (°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
H51A	C51	H51C	109.5	H51B	C51	H51C	109.5
C50	C52	H52A	109.5	C50	C52	H52B	109.5
C50	C52	H52C	109.5	H52A	C52	H52B	109.5
H52A	C52	H52C	109.5	H52B	C52	H52C	109.5

Table 9. Torsion Angles(°)

(Those having bond angles > 160 or < 20 degrees are excluded.)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
O1	Cu1	O2	C18	143.0(4)	O2	Cu1	O1	C1	125.1(4)
O1	Cu1	N1	C11	-16.2(4)	O1	Cu1	N1	C12	109.3(5)
O1	Cu1	N1	C13	-133.3(3)	N1	Cu1	O1	C1	-34.0(4)
O1	Cu1	N2	C14	100.6(6)	O1	Cu1	N2	C15	-18.7(8)
O1	Cu1	N2	C16	-138.9(5)	N2	Cu1	O1	C1	-129.1(6)
O2	Cu1	N1	C11	-127.2(7)	O2	Cu1	N1	C12	-1.8(12)
O2	Cu1	N1	C13	115.6(8)	N1	Cu1	O2	C18	-105.8(8)
O2	Cu1	N2	C14	-153.5(3)	O2	Cu1	N2	C15	87.2(4)
O2	Cu1	N2	C16	-33.0(4)	N2	Cu1	O2	C18	-13.3(5)
N1	Cu1	N2	C14	4.1(3)	N1	Cu1	N2	C15	-115.2(4)
N1	Cu1	N2	C16	124.6(4)	N2	Cu1	N1	C11	139.3(4)
N2	Cu1	N1	C12	-95.2(5)	N2	Cu1	N1	C13	22.2(3)
O3	Cu2	O4	C44	-132.5(4)	O4	Cu2	O3	C27	-130.0(4)
O3	Cu2	N3	C37	22.5(4)	O3	Cu2	N3	C38	-99.9(5)
O3	Cu2	N3	C39	139.7(3)	N3	Cu2	O3	C27	29.0(4)
O3	Cu2	N4	C40	-107.0(6)	O3	Cu2	N4	C41	11.6(9)
O3	Cu2	N4	C42	133.1(5)	N4	Cu2	O3	C27	122.6(6)
O4	Cu2	N3	C37	129.6(7)	O4	Cu2	N3	C38	7.2(12)
O4	Cu2	N3	C39	-113.2(7)	N3	Cu2	O4	C44	120.2(8)
O4	Cu2	N4	C40	146.0(3)	O4	Cu2	N4	C41	-95.5(4)

O4	Cu2	N4	C42	26.0(3)	N4	Cu2	O4	C44	25.5(5)
N3	Cu2	N4	C40	-12.2(3)	N3	Cu2	N4	C41	106.4(4)
N3	Cu2	N4	C42	-132.1(3)	N4	Cu2	N3	C37	-134.4(4)
N4	Cu2	N3	C38	103.1(5)	N4	Cu2	N3	C39	-17.3(3)
Cu1	O1	C1	C2	-138.1(5)	Cu1	O1	C1	C6	40.8(8)
Cu1	O2	C18	C17	32.8(9)	Cu1	O2	C18	C19	-148.8(4)
Cu2	O3	C27	C28	135.2(5)	Cu2	O3	C27	C32	-44.5(8)
Cu2	O4	C44	C43	-41.9(9)	Cu2	O4	C44	C45	138.5(5)
Cu1	N1	C11	C6	58.6(7)	Cu1	N1	C13	C14	-45.5(6)
C12	N1	C11	C6	-67.4(7)	C11	N1	C13	C14	-164.5(6)
C13	N1	C11	C6	171.3(5)	C12	N1	C13	C14	73.4(7)
Cu1	N2	C14	C13	-30.8(7)	Cu1	N2	C16	C17	65.9(7)
C15	N2	C14	C13	87.8(9)	C14	N2	C16	C17	-176.6(7)
C16	N2	C14	C13	-150.0(7)	C15	N2	C16	C17	-54.0(9)
Cu2	N3	C37	C32	-63.7(6)	Cu2	N3	C39	C40	43.6(6)
C38	N3	C37	C32	60.7(7)	C37	N3	C39	C40	159.9(5)
C39	N3	C37	C32	-176.0(5)	C38	N3	C39	C40	-77.8(7)

Table 9. Torsion angles (^{o}) (continued)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
Cu2	N4	C40	C39	39.2(6)	Cu2	N4	C42	C43	-67.4(7)
C41	N4	C40	C39	-80.7(8)	C40	N4	C42	C43	176.9(7)
C42	N4	C40	C39	156.3(6)	C41	N4	C42	C43	54.6(9)
O1	C1	C2	C3	179.8(5)	O1	C1	C2	C8	-5.7(9)
O1	C1	C6	C5	-177.2(5)	O1	C1	C6	C11	9.1(10)
C2	C1	C6	C5	1.7(10)	C2	C1	C6	C11	-172.0(6)
C6	C1	C2	C3	0.9(10)	C6	C1	C2	C8	175.4(6)
C1	C2	C3	C4	-4.3(10)	C1	C2	C8	C9	-141.5(7)
C1	C2	C8	C10	89.6(9)	C3	C2	C8	C9	32.5(11)
C3	C2	C8	C10	-96.4(10)	C8	C2	C3	C4	-178.3(6)
C2	C3	C4	Cl1	-179.8(6)	C2	C3	C4	C5	5.0(12)
Cl1	C4	C5	C6	-177.2(5)	Cl1	C4	C5	C7	1.6(10)
C3	C4	C5	C6	-2.1(11)	C3	C4	C5	C7	176.7(6)
C4	C5	C6	C1	-1.1(10)	C4	C5	C6	C11	172.4(6)
C7	C5	C6	C1	-179.9(6)	C7	C5	C6	C11	-6.4(11)
C1	C6	C11	N1	-62.8(8)	C5	C6	C11	N1	123.5(7)
N1	C13	C14	N2	53.2(8)	N2	C16	C17	C18	-51.8(10)
N2	C16	C17	C22	132.9(8)	C16	C17	C18	O2	-1.1(10)
C16	C17	C18	C19	-179.5(6)	C16	C17	C22	C21	179.8(6)
C16	C17	C22	C23	-0.9(11)	C18	C17	C22	C21	4.5(10)
C18	C17	C22	C23	-176.2(6)	C22	C17	C18	O2	174.3(6)

C22	C17	C18	C19	-4.1(11)	O2	C18	C19	C20	-175.7(6)
O2	C18	C19	C24	1.2(10)	C17	C18	C19	C20	2.7(11)
C17	C18	C19	C24	179.6(6)	C18	C19	C20	C21	-2.0(11)
C18	C19	C24	C25	84.7(9)	C18	C19	C24	C26	-149.1(7)
C20	C19	C24	C25	-98.5(9)	C20	C19	C24	C26	27.7(11)
C24	C19	C20	C21	-178.7(7)	C19	C20	C21	Cl2	178.2(6)
C19	C20	C21	C22	2.7(12)	Cl2	C21	C22	C17	-179.4(5)
Cl2	C21	C22	C23	1.2(10)	C20	C21	C22	C17	-4.0(12)
C20	C21	C22	C23	176.7(7)	O3	C27	C28	C29	176.3(5)
O3	C27	C28	C34	-2.2(10)	O3	C27	C32	C31	-177.2(6)
O3	C27	C32	C37	-0.1(10)	C28	C27	C32	C31	3.2(11)
C28	C27	C32	C37	-179.8(6)	C32	C27	C28	C29	-4.0(11)
C32	C27	C28	C34	177.4(6)	C27	C28	C29	C30	2.6(11)
C27	C28	C34	C35	-153.6(7)	C27	C28	C34	C36	80.7(10)
C29	C28	C34	C35	28.0(11)	C29	C28	C34	C36	-97.8(9)
C34	C28	C29	C30	-178.9(6)	C28	C29	C30	Cl3	-178.6(6)

Table 9. Torsion angles (^{o}) (continued)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C28	C29	C30	C31	-0.4(12)	Cl3	C30	C31	C32	177.6(5)
Cl3	C30	C31	C33	-3.5(10)	C29	C30	C31	C32	-0.6(11)
C29	C30	C31	C33	178.3(6)	C30	C31	C32	C27	-0.7(10)
C30	C31	C32	C37	-177.6(6)	C33	C31	C32	C27	-179.7(6)
C33	C31	C32	C37	3.5(11)	C27	C32	C37	N3	59.2(9)
C31	C32	C37	N3	-123.8(8)	N3	C39	C40	N4	-58.0(7)
N4	C42	C43	C44	59.8(9)	N4	C42	C43	C48	-118.3(9)
C42	C43	C44	O4	-0.5(10)	C42	C43	C44	C45	179.0(6)
C42	C43	C48	C47	-178.8(7)	C42	C43	C48	C49	0.3(12)
C44	C43	C48	C47	3.2(11)	C44	C43	C48	C49	-177.7(6)
C48	C43	C44	O4	177.6(7)	C48	C43	C44	C45	-2.8(11)
O4	C44	C45	C46	-179.2(6)	O4	C44	C45	C50	1.1(10)
C43	C44	C45	C46	1.2(11)	C43	C44	C45	C50	-178.5(6)
C44	C45	C46	C47	-0.1(11)	C44	C45	C50	C51	74.5(9)
C44	C45	C50	C52	-160.9(6)	C46	C45	C50	C51	-105.2(8)
C46	C45	C50	C52	19.4(11)	C50	C45	C46	C47	179.6(6)
C45	C46	C47	Cl4	178.3(6)	C45	C46	C47	C48	0.6(12)
Cl4	C47	C48	C43	-179.7(5)	Cl4	C47	C48	C49	1.2(11)
C46	C47	C48	C43	-2.1(12)	C46	C47	C48	C49	178.8(7)

Table 10. Intramolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
Cu1	C6	3.263(10)	Cu1	C17	3.346(11)
Cu2	C32	3.301(10)	Cu2	C43	3.298(10)
Cl1	C7	3.102(12)	Cl2	C23	3.066(10)
Cl3	C33	3.093(12)	Cl4	C49	3.088(10)
O1	C8	2.799(13)	O1	C10	3.346(15)
O1	C11	2.820(9)	O2	C15	3.498(11)
O2	C16	2.827(12)	O2	C24	2.768(10)
O2	C25	3.284(15)	O3	C29	3.583(14)
O3	C34	2.799(11)	O3	C36	3.250(13)
O3	C37	2.794(10)	O4	C42	2.755(12)
O4	C50	2.810(10)	O4	C51	3.172(11)
N1	C1	3.063(12)	N2	C12	3.596(12)
N2	C18	3.034(11)	N3	C27	3.032(12)
N4	C44	3.031(11)	C1	C4	2.773(15)
C1	C10	3.307(13)	C2	C5	2.878(12)
C3	C6	2.770(14)	C3	C9	3.000(14)
C3	C10	3.412(14)	C6	C12	3.014(13)
C7	C11	3.050(16)	C12	C14	3.036(15)
C13	C15	3.206(14)	C15	C17	2.920(12)
C15	C18	3.407(12)	C16	C23	2.993(16)
C17	C20	2.807(13)	C18	C21	2.744(15)

C18	C25	3.227(17)	C19	C22	2.848(15)
C20	C25	3.409(18)	C20	C26	2.95(2)
C27	C30	2.736(14)	C27	C36	3.261(13)
C27	C38	3.563(12)	C28	C31	2.858(12)
C29	C32	2.772(14)	C29	C35	2.956(12)
C29	C36	3.405(13)	C32	C38	2.942(13)
C33	C37	3.016(16)	C38	C40	3.094(14)
C39	C41	3.088(13)	C41	C43	2.912(12)
C41	C44	3.502(12)	C42	C49	3.038(16)
C43	C46	2.812(12)	C44	C47	2.775(14)
C44	C51	3.165(14)	C45	C48	2.866(15)
C46	C51	3.483(15)	C46	C52	2.939(17)

Table 11. Intramolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
Cu1	H11A	3.053	Cu1	H12A	3.140
Cu1	H12C	2.970	Cu1	H13A	3.001
Cu1	H14B	3.215	Cu1	H15B	3.024
Cu1	H15C	2.989	Cu1	H16A	2.950
Cu2	H37B	2.978	Cu2	H38A	3.056
Cu2	H38C	3.128	Cu2	H39A	3.037
Cu2	H40A	3.111	Cu2	H41A	2.931
Cu2	H41C	3.153	Cu2	H42A	2.892
Cl1	H3	2.750	Cl1	H7A	3.225
Cl1	H7C	2.798	Cl2	H20	2.770
Cl2	H23A	3.453	Cl2	H23B	2.579
Cl3	H29	2.773	Cl3	H33B	2.529
Cl4	H46	2.768	Cl4	H49A	2.569
Cl4	H49B	3.567	O1	H8	2.372
O1	H10B	2.904	O1	H11A	2.801
O1	H25C	3.537	O2	H10B	3.210
O2	H15C	3.112	O2	H16A	2.882
O2	H24	2.369	O2	H25C	2.816
O3	H34	2.481	O3	H36B	2.737
O3	H37B	2.762	O3	H38C	3.482
O3	H50	3.563	O4	H34	3.406

O4	H41C	3.397	O4	H42A	2.707
O4	H50	2.528	O4	H51B	2.616
N1	H7B	3.516	N1	H14A	3.300
N1	H14B	2.709	N2	H12C	3.247
N2	H13A	2.697	N2	H13B	3.285
N3	H40A	2.667	N3	H40B	3.295
N3	H41A	3.485	N4	H38A	3.442
N4	H39A	2.652	N4	H39B	3.288
C1	H3	3.221	C1	H8	2.532
C1	H10B	3.026	C1	H11A	2.754
C1	H11B	3.328	C1	H12A	3.305
C2	H9A	2.871	C2	H9B	3.309
C2	H9C	2.566	C2	H10A	2.758
C2	H10B	2.577	C2	H10C	3.297
C3	H8	3.259	C3	H9A	3.035
C3	H9C	2.829	C3	H10A	3.330

Table 11. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C4	H7A	2.935	C4	H7B	3.334
C4	H7C	2.727	C5	H3	3.263
C5	H11A	3.185	C5	H11B	2.636
C5	H12A	3.177	C6	H7A	3.082
C6	H7B	2.650	C6	H7C	3.282
C6	H12A	2.641	C6	H12B	3.407
C7	H11B	2.557	C7	H12A	3.561
C8	H3	2.725	C9	H3	2.728
C9	H10A	2.587	C9	H10B	3.241
C9	H10C	2.628	C10	H3	3.485
C10	H9A	2.532	C10	H9B	2.707
C10	H9C	3.226	C10	H24	3.143
C10	H25A	3.560	C11	H7A	3.440
C11	H7B	2.628	C11	H12A	2.605
C11	H12B	2.662	C11	H12C	3.262
C11	H13A	2.508	C11	H13B	2.730
C12	H7B	3.311	C12	H11A	3.272
C12	H11B	2.582	C12	H13A	3.280
C12	H13B	2.553	C12	H14B	2.766
C13	H11A	2.534	C13	H11B	2.663
C13	H12A	3.283	C13	H12B	2.613

C13	H12C	2.656	C13	H15B	2.920
C14	H12B	3.366	C14	H12C	2.728
C14	H15A	2.695	C14	H15B	2.622
C14	H15C	3.293	C14	H16A	2.665
C14	H16B	2.616	C15	H13A	2.979
C15	H14A	2.485	C15	H14B	3.262
C15	H16A	3.301	C15	H16B	2.698
C16	H14A	2.885	C16	H14B	2.476
C16	H15A	2.636	C16	H15B	3.295
C16	H15C	2.665	C16	H23A	3.205
C16	H23C	2.693	C17	H15A	3.150
C17	H15C	2.585	C17	H23A	2.943
C17	H23B	3.315	C17	H23C	2.696
C18	H15C	2.843	C18	H16A	2.811
C18	H16B	3.313	C18	H20	3.240
C18	H24	2.550	C18	H25C	2.928

Table 11. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C19	H25A	3.304	C19	H25B	2.722
C19	H25C	2.588	C19	H26A	2.734
C19	H26B	3.327	C19	H26C	2.658
C20	H24	3.261	C20	H25B	3.293
C20	H26A	2.795	C20	H26C	2.956
C21	H23A	3.054	C21	H23B	2.654
C21	H23C	3.282	C22	H15C	3.386
C22	H16A	3.064	C22	H16B	2.574
C22	H20	3.289	C23	H16A	3.487
C23	H16B	2.494	C24	H10B	3.585
C24	H10C	3.469	C24	H20	2.735
C25	H10C	3.474	C25	H20	3.518
C25	H26A	2.615	C25	H26B	2.610
C25	H26C	3.250	C26	H20	2.681
C26	H25A	2.629	C26	H25B	2.607
C26	H25C	3.245	C27	H29	3.212
C27	H34	2.627	C27	H36B	2.995
C27	H37A	3.284	C27	H37B	2.731
C27	H38C	3.069	C28	H35A	2.673
C28	H35B	2.839	C28	H35C	3.357
C28	H36A	2.701	C28	H36B	2.648

C28	H36C	3.314	C29	H34	3.244
C29	H35A	2.889	C29	H35B	2.881
C29	H36A	3.251	C30	H33A	3.140
C30	H33B	2.582	C30	H33C	3.157
C31	H29	3.267	C31	H37A	2.587
C31	H37B	3.130	C31	H38C	3.154
C32	H33A	2.822	C32	H33B	3.331
C32	H33C	2.790	C32	H38B	3.267
C32	H38C	2.569	C33	H37A	2.518
C34	H29	2.688	C35	H29	2.640
C35	H36A	2.682	C35	H36B	3.299
C35	H36C	2.617	C36	H29	3.484
C36	H35A	3.294	C36	H35B	2.615
C36	H35C	2.698	C37	H33A	2.968
C37	H33C	2.944	C37	H38A	3.265
C37	H38B	2.636	C37	H38C	2.604

Table 11. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C37	H39A	2.496	C37	H39B	2.781
C38	H33C	3.388	C38	H37A	2.621
C38	H37B	3.272	C38	H39A	3.275
C38	H39B	2.543	C38	H40A	2.810
C39	H37A	2.641	C39	H37B	2.583
C39	H38A	2.648	C39	H38B	2.652
C39	H38C	3.282	C39	H41A	2.837
C39	H41B	3.386	C40	H38A	2.783
C40	H38B	3.491	C40	H41A	2.698
C40	H41B	2.560	C40	H41C	3.268
C40	H42A	2.676	C40	H42B	2.631
C41	H39A	2.798	C41	H40A	3.263
C41	H40B	2.498	C41	H42A	3.298
C41	H42B	2.698	C41	H49B	3.462
C42	H40A	2.529	C42	H40B	2.855
C42	H41A	3.282	C42	H41B	2.754
C42	H41C	2.563	C42	H49B	3.155
C42	H49C	2.810	C43	H41B	3.336
C43	H41C	2.475	C43	H49A	3.342
C43	H49B	2.898	C43	H49C	2.724
C44	H41C	2.983	C44	H42A	2.707

C44	H42B	3.289	C44	H46	3.258
C44	H50	2.611	C44	H51A	3.515
C44	H51B	2.873	C45	H51A	2.706
C45	H51B	2.660	C45	H51C	3.325
C45	H52A	2.708	C45	H52B	2.801
C45	H52C	3.360	C46	H50	3.228
C46	H51A	3.352	C46	H52A	2.886
C46	H52B	2.837	C47	H49A	2.615
C47	H49B	3.076	C47	H49C	3.212
C48	H41C	3.002	C48	H42A	3.183
C48	H42B	2.612	C48	H46	3.277
C49	H41C	3.548	C49	H42B	2.544
C50	H46	2.713	C51	H34	3.591
C51	H52A	3.324	C51	H52B	2.655
C51	H52C	2.685	C52	H46	2.610
C52	H51A	2.708	C52	H51B	3.319

Table 11. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C52	H51C	2.643	H3	H8	3.543
H3	H9A	2.516	H3	H9C	2.570
H3	H10A	3.213	H7A	H11B	2.887
H7B	H11A	3.337	H7B	H11B	1.941
H7B	H12A	3.105	H7B	H12B	2.923
H7C	H11B	3.390	H8	H9A	2.755
H8	H9B	2.168	H8	H9C	2.366
H8	H10A	2.740	H8	H10B	2.276
H8	H10C	2.184	H8	H25A	3.232
H8	H25C	3.345	H9A	H10A	2.334
H9A	H10B	3.418	H9A	H10C	2.799
H9B	H10A	3.003	H9B	H10B	3.565
H9B	H10C	2.568	H9C	H10A	3.405
H9C	H10C	3.525	H10B	H24	2.660
H10C	H24	2.761	H10C	H25A	2.859
H10C	H26B	3.385	H11A	H12A	3.518
H11A	H12B	3.501	H11A	H13A	2.236
H11A	H13B	2.952	H11B	H12A	2.802
H11B	H12B	2.441	H11B	H12C	3.477
H11B	H13A	2.807	H11B	H13B	2.614
H12A	H13B	3.442	H12B	H13A	3.439

H12B	H13B	2.345	H12B	H14B	3.050
H12C	H13A	3.571	H12C	H13B	2.813
H12C	H14B	2.214	H12C	H16A	3.026
H13A	H14A	2.301	H13A	H14B	2.819
H13A	H15A	3.472	H13A	H15B	2.425
H13B	H14A	2.366	H13B	H14B	2.301
H14A	H15A	2.388	H14A	H15B	2.589
H14A	H15C	3.413	H14A	H16A	3.258
H14A	H16B	2.728	H14B	H15A	3.409
H14B	H15B	3.548	H14B	H16A	2.348
H14B	H16B	2.557	H15A	H16A	3.536
H15A	H16B	2.510	H15A	H23A	3.386
H15B	H16B	3.549	H15C	H16A	3.526
H15C	H16B	3.027	H16A	H23C	2.996
H16B	H23A	2.581	H16B	H23B	3.420
H16B	H23C	2.072	H20	H24	3.549

Table 11. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H20	H25B	3.231	H20	H26A	2.266
H20	H26C	2.710	H24	H25A	2.250
H24	H25B	2.780	H24	H25C	2.307
H24	H26A	2.764	H24	H26B	2.240
H24	H26C	2.275	H25A	H26A	2.923
H25A	H26B	2.452	H25A	H26C	3.498
H25B	H26A	2.433	H25B	H26B	2.882
H25B	H26C	3.484	H25C	H26A	3.479
H25C	H26B	3.486	H29	H34	3.510
H29	H35A	2.591	H29	H35B	2.319
H29	H35C	3.589	H29	H36A	3.179
H33A	H37A	2.351	H33A	H37B	3.366
H33B	H37A	3.476	H33C	H37A	2.328
H33C	H38B	2.898	H33C	H38C	3.194
H34	H35A	2.372	H34	H35B	2.806
H34	H35C	2.260	H34	H36A	2.792
H34	H36B	2.291	H34	H36C	2.291
H34	H50	3.042	H34	H51B	3.235
H34	H51C	3.333	H35A	H36A	3.523
H35A	H36C	3.515	H35B	H36A	2.472
H35B	H36B	3.514	H35B	H36C	2.819

H35C	H36A	3.039	H35C	H36B	3.545
H35C	H36C	2.491	H36B	H50	3.398
H36C	H50	3.408	H37A	H38A	3.502
H37A	H38B	2.454	H37A	H38C	2.882
H37A	H39A	2.720	H37A	H39B	2.640
H37B	H38B	3.507	H37B	H38C	3.498
H37B	H39A	2.273	H37B	H39B	3.066
H37B	H41A	3.501	H38A	H39A	3.570
H38A	H39B	2.751	H38A	H40A	2.235
H38A	H42A	3.437	H38B	H39A	3.445
H38B	H39B	2.379	H38B	H40A	3.209
H38C	H39B	3.446	H39A	H40A	2.846
H39A	H40B	2.356	H39A	H41A	2.285
H39A	H41B	3.096	H39B	H40A	2.359
H39B	H40B	2.374	H40A	H41B	3.365
H40A	H42A	2.386	H40A	H42B	2.652

Table 11. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H40B	H41A	2.798	H40B	H41B	2.242
H40B	H41C	3.376	H40B	H42A	3.218
H40B	H42B	2.688	H41A	H42B	3.594
H41B	H42B	2.636	H41B	H49B	3.077
H41C	H42A	3.454	H41C	H42B	2.865
H41C	H49B	3.117	H42A	H49C	3.285
H42B	H49A	3.477	H42B	H49B	2.595
H42B	H49C	2.146	H46	H50	3.490
H46	H51A	3.381	H46	H52A	2.527
H46	H52B	2.292	H46	H52C	3.558
H50	H51A	2.827	H50	H51B	2.332
H50	H51C	2.337	H50	H52A	2.347
H50	H52B	2.808	H50	H52C	2.281
H51A	H52A	3.568	H51A	H52B	2.516
H51A	H52C	3.017	H51B	H52B	3.545
H51B	H52C	3.537	H51C	H52A	3.534
H51C	H52B	2.876	H51C	H52C	2.480

Table 12. Intermolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
Cu2	$C38^{1}$	3.537(8)	O1	$C11^{2}$	3.333(10)
O2	$C37^{3}$	3.566(10)	O3	$C16^{4}$	3.551(10)
O4	$C38^{1}$	3.310(10)	C1	$C41^{3}$	3.452(10)
C2	$C41^{3}$	3.450(10)	C10	$C41^{3}$	3.540(13)
C11	$O1^{2}$	3.333(10)	C16	$O3^{3}$	3.551(10)
C16	$C27^{3}$	3.565(10)	C17	$C37^{3}$	3.498(12)
C18	$C37^{3}$	3.381(11)	C27	$C16^{4}$	3.565(10)
C30	$C42^{1}$	3.563(11)	C37	$O2^{4}$	3.566(10)
C37	$C17^{4}$	3.498(12)	C37	$C18^{4}$	3.381(11)
C38	$Cu2^{1}$	3.537(8)	C38	$O4^{1}$	3.310(10)
C41	$C1^{4}$	3.452(10)	C41	$C2^{4}$	3.450(10)
C41	$C10^{4}$	3.540(13)	C42	$C30^{1}$	3.563(11)

Symmetry Operators:

(1) -X+1,-Y,-Z+1

(2) -X,-Y+1,-Z+1

(3) X,-Y+1,Z+1

(4) X,-Y+1,Z

Table 13. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
Cu1	H11A[1]	3.355	Cu1	H41A[2]	3.268
Cu2	H12C[3]	3.417	Cu2	H38A[4]	3.448
Cu2	H38C[4]	2.889	Cl1	H49B[5]	3.382
Cl1	H49B[2]	3.336	Cl1	H51C	3.385
Cl2	H11B[6]	3.538	Cl2	H13B[6]	2.995
Cl2	H25A[7]	3.289	Cl2	H33A[2]	3.369
Cl2	H35B[6]	3.357	Cl3	H23A[8]	3.371
Cl3	H26A[9]	3.537	Cl3	H26B[9]	3.140
Cl3	H42A[4]	3.140	Cl3	H42B[4]	3.334
Cl3	H49C[4]	3.297	Cl4	H7C[3]	3.108
Cl4	H9A[10]	3.261	Cl4	H26C[11]	3.421
Cl4	H35C[3]	3.567	Cl4	H38B[5]	3.591
Cl4	H39B[5]	2.869	Cl4	H51B[3]	3.553
Cl4	H51C[3]	3.330	O1	H11A[1]	2.383
O1	H13A[1]	3.145	O1	H41A[2]	3.182
O2	H37B[2]	2.614	O2	H39A[2]	3.131
O2	H41A[2]	3.209	O3	H12C[3]	2.914
O3	H16A[3]	2.583	O3	H38A[4]	3.151
O3	H38C[4]	3.492	O4	H12A[3]	3.467
O4	H12C[3]	3.405	O4	H38A[4]	3.514
O4	H38B[4]	3.070	O4	H38C[4]	2.845

N3	H38C[4]	3.528	C1	H11A[1]	3.114
C1	H13A[1]	3.064	C1	H15B[1]	3.131
C1	H41A[2]	3.049	C1	H41B[2]	3.489
C1	H41C[2]	3.266	C2	H13A[1]	3.118
C2	H15B[1]	3.143	C2	H41A[2]	3.248
C2	H41B[2]	3.090	C2	H41C[2]	3.452
C3	H15B[1]	2.993	C3	H41B[2]	3.259
C3	H41C[2]	3.570	C3	H49A[5]	3.464
C4	H15B[1]	2.959	C4	H41C[2]	3.495
C4	H49B[2]	3.518	C5	H15B[1]	2.981
C5	H15C[1]	3.480	C5	H41C[2]	3.370
C6	H15B[1]	3.037	C6	H41A[2]	3.594
C6	H41C[2]	3.241	C7	H35C	3.083
C8	H13A[1]	3.174	C9	H13A[1]	3.366
C9	H20[12]	3.442	C9	H26C[12]	3.351
C9	H49A[5]	3.112	C10	H39A[2]	3.585

Table 13. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C10	H41A^{2}	3.148	C10	H41B^{2}	3.028
C10	H51A^{11}	3.460	C12	H41A^{2}	3.577
C12	H41C^{2}	3.197	C12	H50^{2}	3.229
C13	H8^{1}	3.437	C13	H9C^{1}	3.424
C13	H20^{8}	3.355	C14	H20^{8}	3.579
C14	H26A^{8}	3.521	C14	H36B^{2}	3.269
C16	H36B^{2}	3.500	C16	H37B^{2}	3.129
C17	H37B^{2}	2.768	C18	H37A^{2}	3.450
C18	H37B^{2}	2.528	C19	H37A^{2}	3.570
C19	H37B^{2}	3.221	C20	H9B^{7}	3.296
C20	H13B^{6}	3.331	C20	H33A^{2}	3.460
C21	H33A^{2}	3.045	C22	H33A^{2}	3.261
C22	H37B^{2}	3.532	C23	H29^{6}	2.958
C23	H35B^{6}	3.483	C25	H36C^{1}	3.563
C26	H9C^{7}	3.498	C27	H16A^{3}	2.715
C27	H38A^{4}	2.920	C28	H16A^{3}	3.468
C28	H38A^{4}	2.901	C28	H40A^{4}	3.355
C29	H23A^{8}	2.982	C29	H23B^{8}	3.597
C29	H23C^{3}	3.552	C29	H38A^{4}	3.597
C29	H40A^{4}	3.217	C29	H42A^{4}	3.163
C30	H23A^{8}	3.599	C30	H23C^{3}	3.319

C30	H42A[4]	2.717	C30	H42B[4]	3.583
C31	H23C[3]	3.397	C31	H42A[4]	3.060
C32	H16A[3]	3.034	C32	H38A[4]	3.580
C33	H9B[9]	3.445	C33	H10C[9]	3.511
C33	H42A[4]	3.593	C34	H38A[4]	3.177
C35	H38A[4]	3.592	C35	H40A[4]	3.268
C35	H46[2]	3.245	C35	H52A[2]	3.499
C36	H14B[3]	3.221	C36	H25B[1]	3.127
C37	H16A[3]	3.230	C38	H34[4]	3.283
C38	H38A[4]	3.506	C38	H38C[4]	3.032
C38	H51B[4]	3.489	C39	H10B[3]	3.573
C40	H35A[4]	3.109	C41	H10A[3]	3.427
C41	H10B[3]	2.878	C41	H12A[3]	2.976
C44	H12A[3]	3.178	C45	H12A[3]	3.334
C45	H12B[3]	3.402	C46	H35C[3]	3.591
C47	H7C[3]	3.306	C49	H3[10]	3.015

Table 13. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C49	H9A[10]	3.478	C49	H9C[10]	3.591
C50	H12B[3]	3.363	C51	H10A[11]	3.585
C51	H38B[4]	3.545	C52	H12B[3]	3.427
H3	C49[5]	3.015	H3	H15B[1]	3.464
H3	H41B[2]	3.559	H3	H49A[5]	2.591
H3	H49B[5]	2.797	H3	H49C[5]	3.181
H7A	H15B[1]	3.584	H7A	H15C[1]	3.101
H7A	H25C[1]	3.582	H7A	H35C	3.231
H7A	H36C	3.221	H7A	H51C	3.341
H7A	H52C	3.408	H7B	H35C	2.771
H7C	Cl4[2]	3.108	H7C	C47[2]	3.306
H7C	H35C	2.754	H7C	H51C	2.979
H8	C13[1]	3.437	H8	H11A[1]	3.303
H8	H13A[1]	2.659	H8	H13B[1]	3.356
H9A	Cl4[5]	3.261	H9A	C49[5]	3.478
H9A	H26C[12]	3.358	H9A	H49A[5]	2.540
H9A	H51A[11]	3.500	H9B	C20[12]	3.296
H9B	C33[13]	3.445	H9B	H13A[1]	3.581
H9B	H20[12]	2.866	H9B	H26C[12]	3.148
H9B	H33A[13]	2.808	H9B	H33C[13]	3.350
H9C	C13[1]	3.424	H9C	C26[12]	3.498

H9C	C49[5]	3.591	H9C	H13A[1]	2.851
H9C	H13B[1]	3.352	H9C	H14A[1]	3.053
H9C	H15B[1]	3.486	H9C	H20[12]	3.126
H9C	H26A[12]	3.107	H9C	H26C[12]	2.997
H9C	H49A[5]	2.818	H9C	H49C[5]	3.543
H10A	C41[2]	3.427	H10A	C51[11]	3.585
H10A	H40B[2]	3.299	H10A	H41A[2]	3.288
H10A	H41B[2]	2.710	H10A	H51A[11]	2.652
H10B	C39[2]	3.573	H10B	C41[2]	2.878
H10B	H39A[2]	2.766	H10B	H40B[2]	3.325
H10B	H41A[2]	2.325	H10B	H41B[2]	2.586
H10B	H41C[2]	3.537	H10C	C33[13]	3.511
H10C	H33A[13]	3.457	H10C	H33B[13]	3.148
H10C	H33C[13]	3.356	H10C	H51A[11]	3.449
H11A	Cu1[1]	3.355	H11A	O1[1]	2.383
H11A	C1[1]	3.114	H11A	H8[1]	3.303

Table 13. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H11A	H11A[1]	2.909	H11B	Cl2[8]	3.538
H11B	H25C[1]	3.436	H12A	O4[2]	3.467
H12A	C41[2]	2.976	H12A	C44[2]	3.178
H12A	C45[2]	3.334	H12A	H41A[2]	2.895
H12A	H41C[2]	2.276	H12A	H50[2]	3.400
H12B	C45[2]	3.402	H12B	C50[2]	3.363
H12B	C52[2]	3.427	H12B	H50[2]	2.902
H12B	H52A[2]	2.752	H12C	Cu2[2]	3.417
H12C	O3[2]	2.914	H12C	O4[2]	3.405
H12C	H36B[2]	3.068	H12C	H41C[2]	3.470
H12C	H50[2]	2.879	H13A	O1[1]	3.145
H13A	C1[1]	3.064	H13A	C2[1]	3.118
H13A	C8[1]	3.174	H13A	C9[1]	3.366
H13A	H8[1]	2.659	H13A	H9B[1]	3.581
H13A	H9C[1]	2.851	H13B	Cl2[8]	2.995
H13B	C20[8]	3.331	H13B	H8[1]	3.356
H13B	H9C[1]	3.352	H13B	H20[8]	2.578
H14A	H9C[1]	3.053	H14A	H20[8]	3.192
H14A	H26A[8]	2.695	H14A	H36B[2]	3.544
H14B	C36[2]	3.221	H14B	H20[8]	3.596
H14B	H36A[2]	3.462	H14B	H36B[2]	2.331

H14B	H36C^{2}	3.564	H15B	C1^{1}	3.131
H15B	C2^{1}	3.143	H15B	C3^{1}	2.993
H15B	C4^{1}	2.959	H15B	C5^{1}	2.981
H15B	C6^{1}	3.037	H15B	H3^{1}	3.464
H15B	H7A^{1}	3.584	H15B	H9C^{1}	3.486
H15C	C5^{1}	3.480	H15C	H7A^{1}	3.101
H16A	O3^{2}	2.583	H16A	C27^{2}	2.715
H16A	C28^{2}	3.468	H16A	C32^{2}	3.034
H16A	C37^{2}	3.230	H16A	H36B^{2}	2.930
H16A	H37B^{2}	2.573	H16B	H36B^{2}	3.276
H20	C9^{7}	3.442	H20	C13^{6}	3.355
H20	C14^{6}	3.579	H20	H9B^{7}	2.866
H20	H9C^{7}	3.126	H20	H13B^{6}	2.578
H20	H14A^{6}	3.192	H20	H14B^{6}	3.596
H23A	Cl3^{6}	3.371	H23A	C29^{6}	2.982
H23A	C30^{6}	3.599	H23A	H29^{6}	2.204

Table 13. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H23A	H35B^{6}	3.040	H23B	C29^{6}	3.597
H23B	H29^{6}	2.988	H23B	H33A^{2}	3.455
H23B	H35B^{6}	3.063	H23B	H36A^{6}	2.881
H23C	C29^{2}	3.552	H23C	C30^{2}	3.319
H23C	C31^{2}	3.397	H23C	H29^{6}	3.326
H24	H37B^{2}	3.490	H24	H39A^{2}	2.927
H25A	Cl2^{12}	3.289	H25A	H33A^{13}	3.554
H25A	H33B^{13}	3.017	H25B	C36^{1}	3.127
H25B	H36A^{1}	2.822	H25B	H36B^{1}	3.370
H25B	H36C^{1}	2.718	H25C	H7A^{1}	3.582
H25C	H11B^{1}	3.436	H26A	Cl3^{13}	3.537
H26A	C14^{6}	3.521	H26A	H9C^{7}	3.107
H26A	H14A^{6}	2.695	H26B	Cl3^{13}	3.140
H26B	H33B^{13}	2.897	H26B	H49C^{11}	3.470
H26C	Cl4^{11}	3.421	H26C	C9^{7}	3.351
H26C	H9A^{7}	3.358	H26C	H9B^{7}	3.148
H26C	H9C^{7}	2.997	H26C	H49A^{11}	3.186
H29	C23^{8}	2.958	H29	H23A^{8}	2.204
H29	H23B^{8}	2.988	H29	H23C^{8}	3.326
H29	H40A^{4}	3.252	H29	H42A^{4}	3.479
H33A	Cl2^{3}	3.369	H33A	C20^{3}	3.460

H33A	C21[3]	3.045	H33A	C22[3]	3.261
H33A	H9B[9]	2.808	H33A	H10C[9]	3.457
H33A	H23B[3]	3.455	H33A	H25A[9]	3.554
H33B	H10C[9]	3.148	H33B	H25A[9]	3.017
H33B	H26B[9]	2.897	H33B	H42A[4]	3.520
H33C	H9B[9]	3.350	H33C	H10C[9]	3.356
H33C	H42A[4]	3.500	H33C	H51B[4]	3.060
H34	C38[4]	3.283	H34	H38A[4]	2.723
H34	H38B[4]	3.016	H35A	C40[4]	3.109
H35A	H38A[4]	3.066	H35A	H38B[4]	3.583
H35A	H39B[4]	3.241	H35A	H40A[4]	2.369
H35A	H40B[4]	3.153	H35A	H46[2]	2.759
H35A	H52A[2]	3.385	H35A	H52B[2]	3.258
H35B	Cl2[8]	3.357	H35B	C23[8]	3.483
H35B	H23A[8]	3.040	H35B	H23B[8]	3.063
H35B	H46[2]	3.537	H35B	H52A[2]	3.069

Table 13. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H35B	H52B^{2}	3.540	H35C	Cl4^{2}	3.567
H35C	C7	3.083	H35C	C46^{2}	3.591
H35C	H7A	3.231	H35C	H7B	2.771
H35C	H7C	2.754	H35C	H46^{2}	2.972
H35C	H52A^{2}	3.482	H36A	H14B^{3}	3.462
H36A	H23B^{8}	2.881	H36A	H25B^{1}	2.822
H36B	C14^{3}	3.269	H36B	C16^{3}	3.500
H36B	H12C^{3}	3.068	H36B	H14A^{3}	3.544
H36B	H14B^{3}	2.331	H36B	H16A^{3}	2.930
H36B	H16B^{3}	3.276	H36B	H25B^{1}	3.370
H36C	C25^{1}	3.563	H36C	H7A	3.221
H36C	H14B^{3}	3.564	H36C	H25B^{1}	2.718
H37A	C18^{3}	3.450	H37A	C19^{3}	3.570
H37B	O2^{3}	2.614	H37B	C16^{3}	3.129
H37B	C17^{3}	2.768	H37B	C18^{3}	2.528
H37B	C19^{3}	3.221	H37B	C22^{3}	3.532
H37B	H16A^{3}	2.573	H37B	H24^{3}	3.490
H38A	Cu2^{4}	3.448	H38A	O3^{4}	3.151
H38A	O4^{4}	3.514	H38A	C27^{4}	2.920
H38A	C28^{4}	2.901	H38A	C29^{4}	3.597
H38A	C32^{4}	3.580	H38A	C34^{4}	3.177

H38A	C35[4]	3.592	H38A	C38[4]	3.506
H38A	H34[4]	2.723	H38A	H35A[4]	3.066
H38A	H38C[4]	2.737	H38B	Cl4[10]	3.591
H38B	O4[4]	3.070	H38B	C51[4]	3.545
H38B	H34[4]	3.016	H38B	H35A[4]	3.583
H38B	H51B[4]	2.626	H38C	Cu2[4]	2.889
H38C	O3[4]	3.492	H38C	O4[4]	2.845
H38C	N3[4]	3.528	H38C	C38[4]	3.032
H38C	H38A[4]	2.737	H38C	H38C[4]	2.618
H38C	H42A[4]	3.319	H39A	O2[3]	3.131
H39A	C10[3]	3.585	H39A	H10B[3]	2.766
H39A	H24[3]	2.927	H39B	Cl4[10]	2.869
H39B	H35A[4]	3.241	H39B	H46[10]	3.142
H40A	C28[4]	3.355	H40A	C29[4]	3.217
H40A	C35[4]	3.268	H40A	H29[4]	3.252
H40A	H35A[4]	2.369	H40B	H10A[3]	3.299

Table 13. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H40B	H10B^{3}	3.325	H40B	H35A^{4}	3.153
H40B	H46^{10}	3.200	H40B	H52B^{10}	3.082
H41A	Cu1^{3}	3.268	H41A	O1^{3}	3.182
H41A	O2^{3}	3.209	H41A	C1^{3}	3.049
H41A	C2^{3}	3.248	H41A	C6^{3}	3.594
H41A	C10^{3}	3.148	H41A	C12^{3}	3.577
H41A	H10A^{3}	3.288	H41A	H10B^{3}	2.325
H41A	H12A^{3}	2.895	H41B	C1^{3}	3.489
H41B	C2^{3}	3.090	H41B	C3^{3}	3.259
H41B	C10^{3}	3.028	H41B	H3^{3}	3.559
H41B	H10A^{3}	2.710	H41B	H10B^{3}	2.586
H41C	C1^{3}	3.266	H41C	C2^{3}	3.452
H41C	C3^{3}	3.570	H41C	C4^{3}	3.495
H41C	C5^{3}	3.370	H41C	C6^{3}	3.241
H41C	C12^{3}	3.197	H41C	H10B^{3}	3.537
H41C	H12A^{3}	2.276	H41C	H12C^{3}	3.470
H42A	Cl3^{4}	3.140	H42A	C29^{4}	3.163
H42A	C30^{4}	2.717	H42A	C31^{4}	3.060
H42A	C33^{4}	3.593	H42A	H29^{4}	3.479
H42A	H33B^{4}	3.520	H42A	H33C^{4}	3.500
H42A	H38C^{4}	3.319	H42B	Cl3^{4}	3.334

H42B	C30[4]	3.583	H46	C35[3]	3.245
H46	H35A[3]	2.759	H46	H35B[3]	3.537
H46	H35C[3]	2.972	H46	H39B[5]	3.142
H46	H40B[5]	3.200	H49A	C3[10]	3.464
H49A	C9[10]	3.112	H49A	H3[10]	2.591
H49A	H9A[10]	2.540	H49A	H9C[10]	2.818
H49A	H26C[11]	3.186	H49B	Cl1[10]	3.382
H49B	Cl1[3]	3.336	H49B	C4[3]	3.518
H49B	H3[10]	2.797	H49C	Cl3[4]	3.297
H49C	H3[10]	3.181	H49C	H9C[10]	3.543
H49C	H26B[11]	3.470	H50	C12[3]	3.229
H50	H12A[3]	3.400	H50	H12B[3]	2.902
H50	H12C[3]	2.879	H51A	C10[11]	3.460
H51A	H9A[11]	3.500	H51A	H10A[11]	2.652
H51A	H10C[11]	3.449	H51B	Cl4[2]	3.553
H51B	C38[4]	3.489	H51B	H33C[4]	3.060

Table 13. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H51B	H38B[4]	2.626	H51C	Cl1	3.385
H51C	Cl4[2]	3.330	H51C	H7A	3.341
H51C	H7C	2.979	H52A	C35[3]	3.499
H52A	H12B[3]	2.752	H52A	H35A[3]	3.385
H52A	H35B[3]	3.069	H52A	H35C[3]	3.482
H52B	H35A[3]	3.258	H52B	H35B[3]	3.540
H52B	H40B[5]	3.082	H52C	H7A	3.408

Symmetry Operators:

(1) -X,-Y+1,-Z+1 (2) X,-Y+1,Z+1

(3) X,-Y+1,Z (4) -X+1,-Y,-Z+1

(5) -X+1,Y+1/2,-Z+1/2 (6) -X,Y+1/2,-Z+1/2+1

(7) X,-Y+2,Z+1 (8) -X,Y+1/2-1,-Z+1/2+1

(9) X,Y-1,Z (10) -X+1,Y+1/2-1,-Z+1/2

(11) -X+1,-Y+1,-Z+1 (12) X,-Y+2,Z

(13) X,Y+1,Z

Appendix D: Supplementary Information for Mo-2

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{20}H_{19}ClMoN_3O_4S_2$
Formula Weight	560.90
Crystal Color, Habit	red, needle
Crystal Dimensions	0.120 X 0.040 X 0.040 mm
Crystal System	triclinic
Lattice Type	Primitive
Lattice Parameters	a = 42.376(13) Å
	b = 6.750(2) Å
	c = 15.491(5) Å
	α = 90.00000 ^{o}

	β = 92.810(7) ^{o}
	γ = 90.00000 ^{o}
	V = 4426(2) Å^{3}
Space Group	P-1 (#2)
Z value	8
D_{calc}	1.683 g/cm^{3}
F_{000}	2264.00
μ(MoKα)	9.337 cm^{-1}

B. Intensity Measurements

Diffractometer	XtaLAB mini
Radiation	MoKα (λ = 0.71075 Å) graphite monochromated
Voltage, Current	50kV, 12mA
Temperature	20.0^{o}C
Detector Aperture	75.0 mm (diameter)
Data Images	540 exposures
ω oscillation Range (χ=54.0, ϕ=0.0)	-60.0 - 120.0^{o}
Exposure Rate	120.0 sec./^{o}
Detector Swing Angle	29.50^{o}
ω oscillation Range (χ=54.0, ϕ=120.0)	-60.0 - 120.0^{o}

Exposure Rate	120.0 sec./^{o}
Detector Swing Angle	29.50^{o}
ω oscillation Range (χ=54.0, ϕ=240.0)	-60.0 - 120.0^{o}
Exposure Rate	120.0 sec./^{o}
Detector Swing Angle	29.50^{o}
Detector Position	50.00 mm
Pixel Size	0.073 mm
$2\theta_{max}$	55.0^{o}
No. of Reflections Measured	Total: 13396 Unique: 4861 (R_{int} = 0.4355)
Corrections	Lorentz-polarization Absorption (*trans*. factors: 0.294 - 0.963)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SHELXS86)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	$w = 1/ [\sigma^2(Fo^2) + (0.1717 \cdot P)^2 + 0.0000 \cdot P]$ where $P = (Max(Fo^2,0) + 2Fc^2)/3$
$2\theta_{max}$ cutoff	55.0^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	4861
No. Variables	282
Reflection/Parameter Ratio	17.24

Residuals: R1 (I>2.00σ(I))	0.1776
Residuals: R (All reflections)	0.3939
Residuals: wR2 (All reflections)	0.4634
Goodness of Fit Indicator	0.951
Max Shift/Error in Final Cycle	0.003
Maximum peak in Final Diff. Map	0.39 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.64 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
Mo1	0.18698(6)	0.3729(3)	0.61329(14)	3.83(6)
Cl1	0.0371(2)	-0.522(2)	0.2961(7)	11.0(4)
S1	0.19407(16)	0.0661(9)	0.5326(5)	4.13(14)
S2	0.22307(17)	0.4541(11)	0.4239(5)	4.65(15)
O1	0.1687(4)	0.634(3)	0.6238(10)	4.4(4)
O2	0.1752(4)	0.275(2)	0.7062(9)	4.7(4)
O3	0.2262(3)	0.410(2)	0.6269(10)	4.1(4)
O4	0.1945(3)	0.525(2)	0.4749(10)	3.4(3)
N1	0.1399(6)	0.325(3)	0.5454(13)	4.9(5)
N2	0.1345(6)	0.169(3)	0.4898(12)	4.9(5)
N3	0.1523(4)	-0.133(3)	0.4380(13)	4.5(5)
C1	0.1396(7)	0.709(4)	0.6374(14)	4.1(6)
C2	0.1376(8)	0.882(5)	0.6819(19)	5.8(7)
C3	0.1098(8)	0.972(4)	0.6818(18)	5.4(7)
C4	0.0823(7)	0.907(4)	0.6498(16)	4.3(6)
C5	0.0534(9)	0.995(5)	0.646(2)	7.7(11)
C6	0.0247(9)	0.920(7)	0.617(3)	10.3(14)
C7	0.0272(7)	0.747(5)	0.580(3)	7.9(10)
C8	0.0551(7)	0.615(5)	0.5716(15)	6.1(7)
C9	0.0818(6)	0.726(4)	0.6055(17)	4.4(6)
C10	0.1120(8)	0.619(4)	0.6041(19)	6.0(7)

C11	0.1154(5)	0.440(4)	0.5532(17)	4.0(6)
C12	0.1550(5)	0.037(3)	0.4879(16)	3.2(5)
C13	0.1247(6)	-0.203(4)	0.4025(14)	3.8(5)
C14	0.0935(7)	-0.107(4)	0.4028(17)	5.1(6)
C15	0.0683(9)	-0.225(6)	0.371(2)	8.3(11)
C16	0.0704(6)	-0.399(7)	0.3312(19)	7.4(10)
C17	0.0981(7)	-0.491(4)	0.3322(17)	4.6(6)
C18	0.1260(5)	-0.402(3)	0.3617(18)	4.5(6)
C19	0.2352(6)	0.673(3)	0.3713(17)	5.1(7)
C20	0.2060(9)	0.326(3)	0.3301(17)	7.9(11)

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B_{iso} involving hydrogen atoms

atom	x	y	z	B_{iso}
H2	0.15521	0.93599	0.71135	6.921
H2A	0.11752	0.16275	0.45717	5.931
H3	0.10959	1.09601	0.70771	6.475
H3A	0.16926	-0.19878	0.42900	5.401
H5	0.05302	1.12515	0.66663	9.270
H6	0.00561	0.98500	0.62353	12.388
H7	0.00841	0.69873	0.55462	9.515
H8	0.05525	0.48757	0.54902	7.262
H11	0.09727	0.40111	0.52126	4.823
H14	0.09087	0.02249	0.42223	6.111
H15	0.04804	-0.17716	0.37953	9.927
H17	0.09882	-0.62083	0.31231	5.547
H18	0.14532	-0.46503	0.35593	5.409
H19A	0.24980	0.74464	0.40892	6.171
H19B	0.21711	0.75408	0.35715	6.171
H19C	0.24533	0.63797	0.31927	6.171
H20A	0.18511	0.37496	0.31687	9.491
H20B	0.20508	0.18630	0.34206	9.491
H20C	0.21890	0.34770	0.28172	9.491

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Mo1	0.0538(15)	0.0489(13)	0.0425(13)	-0.0058(12)	0.0009(10)	0.0003(12)
Cl1	0.056(6)	0.230(13)	0.131(9)	-0.035(7)	-0.009(6)	-0.058(9)
S1	0.056(4)	0.038(4)	0.062(5)	0.008(3)	0.001(4)	-0.008(3)
S2	0.056(5)	0.076(5)	0.046(4)	0.013(4)	0.011(3)	0.003(4)
O1	0.036(10)	0.091(13)	0.042(10)	0.019(10)	0.000(8)	-0.003(10)
O2	0.117(16)	0.043(10)	0.018(9)	-0.006(10)	0.016(9)	0.009(7)
O3	0.031(9)	0.066(12)	0.060(11)	-0.024(8)	-0.003(8)	-0.012(9)
O4	0.026(9)	0.059(11)	0.046(10)	0.012(8)	0.020(8)	0.010(8)
N1	0.10(2)	0.050(14)	0.040(13)	-0.033(13)	0.011(13)	-0.017(11)
N2	0.092(19)	0.070(17)	0.026(12)	-0.017(14)	-0.003(12)	-0.002(11)
N3	0.019(11)	0.076(15)	0.076(16)	-0.009(10)	0.002(10)	-0.037(13)
C1	0.059(19)	0.08(2)	0.018(13)	-0.017(15)	-0.011(12)	0.010(12)
C2	0.08(2)	0.07(2)	0.07(2)	0.025(18)	-0.010(16)	0.023(18)
C3	0.09(2)	0.055(18)	0.06(2)	0.015(17)	-0.009(18)	0.033(15)
C4	0.08(2)	0.057(19)	0.032(15)	-0.008(15)	-0.001(14)	-0.005(13)
C5	0.10(3)	0.12(3)	0.07(2)	0.08(2)	0.06(2)	0.03(2)
C6	0.06(3)	0.17(5)	0.15(4)	0.03(3)	-0.06(3)	-0.06(3)
C7	0.029(17)	0.11(3)	0.16(4)	0.048(18)	-0.01(2)	-0.04(3)
C8	0.07(2)	0.14(3)	0.022(14)	0.00(2)	-0.008(13)	0.021(17)
C9	0.044(17)	0.065(18)	0.056(18)	0.000(14)	-0.019(14)	-0.003(14)
C10	0.12(3)	0.036(15)	0.07(2)	0.007(17)	-0.026(18)	-0.029(16)

C11	0.006(11)	0.071(18)	0.076(19)	0.006(11)	0.012(12)	-0.020(14)
C12	0.013(11)	0.041(14)	0.066(18)	0.011(10)	-0.004(11)	0.001(12)
C13	0.032(15)	0.08(2)	0.030(14)	-0.021(13)	-0.010(11)	0.007(12)
C14	0.07(2)	0.08(2)	0.050(18)	0.001(17)	-0.009(15)	-0.016(16)
C15	0.08(3)	0.12(3)	0.11(3)	0.08(2)	0.00(2)	-0.02(2)
C16	0.018(15)	0.21(4)	0.055(19)	-0.01(2)	0.019(13)	-0.05(2)
C17	0.07(2)	0.039(15)	0.07(2)	-0.021(14)	0.025(16)	-0.005(13)
C18	0.021(13)	0.040(15)	0.11(2)	-0.014(11)	-0.008(14)	-0.019(15)
C19	0.08(2)	0.06(2)	0.052(17)	-0.009(15)	-0.026(15)	0.012(14)
C20	0.23(4)	0.024(16)	0.053(19)	-0.020(18)	0.05(2)	-0.004(13)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
Mo1	S1	2.445(7)	Mo1	O1	1.935(18)
Mo1	O2	1.681(15)	Mo1	O3	1.684(15)
Mo1	O4	2.411(15)	Mo1	N1	2.23(2)
Cl1	C16	1.70(3)	S1	C12	1.77(2)
S2	O4	1.553(16)	S2	C19	1.78(3)
S2	C20	1.81(3)	O1	C1	1.36(3)
N1	N2	1.37(3)	N1	C11	1.31(3)
N2	C12	1.24(3)	N3	C12	1.39(3)
N3	C13	1.35(3)	C1	C2	1.36(4)
C1	C10	1.39(4)	C2	C3	1.33(4)
C3	C4	1.32(4)	C4	C5	1.36(5)
C4	C9	1.40(4)	C5	C6	1.37(5)
C6	C7	1.31(6)	C7	C8	1.49(4)
C8	C9	1.43(4)	C9	C10	1.47(4)
C10	C11	1.46(4)	C13	C14	1.47(4)
C13	C18	1.48(4)	C14	C15	1.40(5)
C15	C16	1.34(6)	C16	C17	1.33(4)
C17	C18	1.38(3)			

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
N2	H2A	0.860	N3	H3A	0.860
C2	H2	0.930	C3	H3	0.930
C5	H5	0.930	C6	H6	0.930
C7	H7	0.930	C8	H8	0.930
C11	H11	0.930	C14	H14	0.930
C15	H15	0.930	C17	H17	0.930
C18	H18	0.930	C19	H19A	0.960
C19	H19B	0.960	C19	H19C	0.960
C20	H20A	0.960	C20	H20B	0.960
C20	H20C	0.960			

Table 6. Bond angles (^{o})

atom	atom	atom	angle	atom	atom	atom	angle
S1	Mo1	O1	150.9(5)	S1	Mo1	O2	98.9(5)
S1	Mo1	O3	92.5(6)	S1	Mo1	O4	83.2(4)
S1	Mo1	N1	76.4(6)	O1	Mo1	O2	98.5(7)
O1	Mo1	O3	104.6(7)	O1	Mo1	O4	76.0(6)
O1	Mo1	N1	79.6(7)	O2	Mo1	O3	106.5(8)
O2	Mo1	O4	170.3(7)	O2	Mo1	N1	92.9(8)
O3	Mo1	O4	82.9(6)	O3	Mo1	N1	159.1(8)
O4	Mo1	N1	78.3(6)	Mo1	S1	C12	99.1(8)
O4	S2	C19	103.2(11)	O4	S2	C20	105.4(14)
C19	S2	C20	98.2(12)	Mo1	O1	C1	136.4(17)
Mo1	O4	S2	117.5(8)	Mo1	N1	N2	121.7(17)
Mo1	N1	C11	124.4(16)	N2	N1	C11	114(2)
N1	N2	C12	118(2)	C12	N3	C13	124(2)
O1	C1	C2	119(2)	O1	C1	C10	122(2)
C2	C1	C10	119(3)	C1	C2	C3	118(3)
C2	C3	C4	128(3)	C3	C4	C5	130(3)
C3	C4	C9	118(3)	C5	C4	C9	112(3)
C4	C5	C6	129(3)	C5	C6	C7	113(3)
C6	C7	C8	131(3)	C7	C8	C9	106(3)
C4	C9	C8	129(3)	C4	C9	C10	116(2)
C8	C9	C10	114(2)	C1	C10	C9	120(2)

C1	C10	C11	117(3)	C9	C10	C11	121(2)
N1	C11	C10	130(2)	S1	C12	N2	123.3(19)
S1	C12	N3	111.0(15)	N2	C12	N3	125(2)
N3	C13	C14	127(2)	N3	C13	C18	116(2)
C14	C13	C18	117(2)	C13	C14	C15	114(3)
C14	C15	C16	126(3)	Cl1	C16	C15	120(3)
Cl1	C16	C17	120(3)	C15	C16	C17	119(3)
C16	C17	C18	123(3)	C13	C18	C17	119(2)

Table 7. Bond angles involving hydrogens (^{o})

atom	atom	atom	angle	atom	atom	atom	angle
N1	N2	H2A	121.0	C12	N2	H2A	121.0
C12	N3	H3A	117.9	C13	N3	H3A	117.9
C1	C2	H2	121.1	C3	C2	H2	121.0
C2	C3	H3	115.9	C4	C3	H3	115.9
C4	C5	H5	115.5	C6	C5	H5	115.5
C5	C6	H6	123.6	C7	C6	H6	123.6
C6	C7	H7	114.7	C8	C7	H7	114.7
C7	C8	H8	127.2	C9	C8	H8	127.2
N1	C11	H11	115.0	C10	C11	H11	115.0
C13	C14	H14	122.8	C15	C14	H14	122.8
C14	C15	H15	116.8	C16	C15	H15	116.8
C16	C17	H17	118.6	C18	C17	H17	118.6
C13	C18	H18	120.6	C17	C18	H18	120.6
S2	C19	H19A	109.5	S2	C19	H19B	109.5
S2	C19	H19C	109.5	H19A	C19	H19B	109.5
H19A	C19	H19C	109.5	H19B	C19	H19C	109.5
S2	C20	H20A	109.5	S2	C20	H20B	109.5
S2	C20	H20C	109.5	H20A	C20	H20B	109.5
H20A	C20	H20C	109.5	H20B	C20	H20C	109.5

Table 8. Torsion Angles(O)

(Those having bond angles > 160 or < 20 degrees are excluded.)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
S1	Mo1	O1	C1	72.3(19)	O1	Mo1	S1	C12	-38.7(12)
O2	Mo1	S1	C12	87.4(7)	O3	Mo1	S1	C12	-165.5(6)
S1	Mo1	O4	S2	-48.5(8)	O4	Mo1	S1	C12	-83.0(4)
S1	Mo1	N1	N2	-2.7(12)	S1	Mo1	N1	C11	178.3(16)
N1	Mo1	S1	C12	-3.4(6)	O2	Mo1	O1	C1	-53.9(16)
O3	Mo1	O1	C1	-163.4(14)	O1	Mo1	O4	S2	152.0(10)
O4	Mo1	O1	C1	117.9(15)	O1	Mo1	N1	N2	160.8(15)
O1	Mo1	N1	C11	-18.2(15)	N1	Mo1	O1	C1	37.5(14)
O2	Mo1	N1	N2	-101.1(14)	O2	Mo1	N1	C11	79.9(16)
O3	Mo1	O4	S2	45.0(9)	O3	Mo1	N1	N2	57(3)
O3	Mo1	N1	C11	-122(2)	O4	Mo1	N1	N2	83.0(14)
O4	Mo1	N1	C11	-96.0(15)	N1	Mo1	O4	S2	-126.0(10)
Mo1	S1	C12	N2	12(2)	Mo1	S1	C12	N3	-179.4(13)
C19	S2	O4	Mo1	-145.4(10)	C20	S2	O4	Mo1	112.1(10)
Mo1	O1	C1	C2	147.8(16)	Mo1	O1	C1	C10	-35(3)
Mo1	N1	N2	C12	11(3)	Mo1	N1	C11	C10	0(4)
N2	N1	C11	C10	-179(2)	C11	N1	N2	C12	-170(2)
N1	N2	C12	S1	-15(3)	N1	N2	C12	N3	177.0(19)
C12	N3	C13	C14	6(4)	C12	N3	C13	C18	-171.9(19)
C13	N3	C12	S1	173.6(19)	C13	N3	C12	N2	-18(4)

O1	C1	C2	C3	169(2)	O1	C1	C10	C9	-168(2)
O1	C1	C10	C11	-0(4)	C2	C1	C10	C9	9(4)
C2	C1	C10	C11	177(2)	C10	C1	C2	C3	-9(4)
C1	C2	C3	C4	6(5)	C2	C3	C4	C5	-178(3)
C2	C3	C4	C9	-4(4)	C3	C4	C5	C6	-176(3)
C3	C4	C9	C8	174(2)	C3	C4	C9	C10	4(4)
C5	C4	C9	C8	-11(4)	C5	C4	C9	C10	179(2)
C9	C4	C5	C6	10(4)	C4	C5	C6	C7	-8(6)
C5	C6	C7	C8	6(6)	C6	C7	C8	C9	-7(5)
C7	C8	C9	C4	9(4)	C7	C8	C9	C10	179(2)
C4	C9	C10	C1	-7(4)	C4	C9	C10	C11	-174(2)
C8	C9	C10	C1	-178(2)	C8	C9	C10	C11	15(4)
C1	C10	C11	N1	14(4)	C9	C10	C11	N1	-179(2)
N3	C13	C14	C15	-172(2)	N3	C13	C18	C17	173(2)
C14	C13	C18	C17	-5(3)	C18	C13	C14	C15	6(3)
C13	C14	C15	C16	-10(5)	C14	C15	C16	Cl1	-179(3)
C14	C15	C16	C17	12(5)	Cl1	C16	C17	C18	-178.6(18)

Table 8. Torsion angles (^{o}) (continued)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C15	C16	C17	C18	-10(5)	C16	C17	C18	C13	7(4)

Table 9. Possible hydrogen bonds

Donor	H	Acceptor	D...A	D-H	H...A	D-H...A
N2	H2A	N1	1.37(3)	0.86	1.96	36.86 intramol.
N3	H3A	O4[1]	2.96(2)	0.86	2.25	139.67

Symmetry Operators:

(1) X,Y-1,Z

Table 10. Intramolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
Mo1	C10	3.58(3)	S1	S2	3.377(10)
S2	O3	3.155(18)	O1	C3	3.53(3)
O1	C11	2.79(3)	O2	C1	3.44(3)
O2	C11	3.56(3)	O4	N2	3.51(3)
N1	N3	3.56(3)	N1	C1	2.96(3)
N2	C13	2.88(3)	N2	C14	2.84(4)
C1	C4	2.78(4)	C2	C9	2.80(4)
C3	C10	2.67(4)	C4	C7	2.75(4)
C5	C8	2.82(5)	C6	C9	2.76(5)
C8	C11	2.84(4)	C11	C12	3.38(3)
C12	C14	3.02(4)	C13	C16	2.83(4)
C14	C17	2.83(4)	C15	C18	2.73(4)

Table 11. Intramolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
Cl1	H15	2.690	Cl1	H17	2.699
S1	H2A	3.456	S1	H3A	2.591
S1	H20B	3.118	O1	H2	2.530
O4	H19A	2.996	O4	H19B	2.610
O4	H19C	3.398	O4	H20A	2.661
O4	H20B	3.123	O4	H20C	3.430
N1	H14	3.424	N2	H3A	3.059
N2	H11	2.293	N2	H14	2.303
N3	H2A	2.510	N3	H14	2.807
N3	H18	2.583	N3	H20B	3.492
C1	H3	3.122	C1	H11	3.235
C3	H5	2.618	C4	H2	3.193
C4	H6	3.299	C4	H8	3.404
C5	H3	2.610	C5	H7	3.066
C6	H8	3.384	C7	H5	3.059
C8	H6	3.384	C8	H11	2.453
C9	H3	3.155	C9	H5	3.123
C9	H7	3.174	C9	H11	2.651
C10	H2	3.221	C10	H3	3.599
C10	H8	2.667	C11	H2A	2.394
C11	H8	2.566	C11	H14	3.595

C12	H11	3.523	C12	H14	2.855
C12	H20B	3.331	C13	H2A	2.636
C13	H15	3.253	C13	H17	3.307
C14	H2A	2.227	C14	H3A	3.274
C14	H18	3.369	C15	H2A	3.561
C15	H17	3.127	C16	H14	3.273
C16	H18	3.209	C17	H15	3.112
C18	H3A	2.476	C18	H14	3.382
C19	H20A	3.014	C19	H20B	3.544
C19	H20C	2.669	C20	H19A	3.564
C20	H19B	2.956	C20	H19C	2.697
H2	H3	2.213	H2A	H3A	3.324
H2A	H11	2.096	H2A	H14	1.551
H3	H5	2.458	H3A	H18	2.330
H3A	H20B	3.328	H5	H6	2.290
H6	H7	2.214	H7	H8	2.449

Table 11. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H8	H11	1.942	H11	H14	2.986
H14	H15	2.330	H17	H18	2.305
H19A	H20C	3.539	H19B	H20A	2.949
H19B	H20C	2.984	H19C	H20A	3.107
H19C	H20B	3.520	H19C	H20C	2.317

Table 12. Intermolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
Cl1	$Cl1^{1}$	3.390(13)	S1	$O1^{2}$	3.438(18)
O1	$S1^{3}$	3.438(18)	O1	$N3^{3}$	3.32(3)
O1	$C12^{3}$	3.47(3)	O1	$C20^{4}$	3.51(3)
O2	$C2^{2}$	3.11(4)	O2	$C3^{2}$	3.45(4)
O2	$C18^{5}$	3.37(3)	O2	$C19^{4}$	3.53(3)
O2	$C20^{4}$	3.52(3)	O4	$N3^{3}$	2.96(2)
O4	$C18^{3}$	3.35(3)	N1	$C3^{2}$	3.47(4)
N1	$C18^{3}$	3.42(3)	N2	$C2^{2}$	3.55(4)
N2	$C3^{2}$	3.47(4)	N2	$C18^{3}$	3.52(3)
N3	$O1^{2}$	3.32(3)	N3	$O4^{2}$	2.96(2)
N3	$C1^{2}$	3.34(3)	N3	$C10^{2}$	3.57(4)
C1	$N3^{3}$	3.34(3)	C1	$C12^{3}$	3.29(4)
C2	$O2^{3}$	3.11(4)	C2	$N2^{3}$	3.55(4)
C2	$C12^{3}$	3.30(4)	C3	$O2^{3}$	3.45(4)
C3	$N1^{3}$	3.47(4)	C3	$N2^{3}$	3.47(4)
C8	$C15^{3}$	3.36(4)	C9	$C14^{3}$	3.40(4)
C10	$N3^{3}$	3.57(4)	C10	$C13^{3}$	3.41(4)
C11	$C13^{3}$	3.39(4)	C11	$C17^{3}$	3.50(4)
C11	$C18^{3}$	3.21(4)	C12	$O1^{2}$	3.47(3)
C12	$C1^{2}$	3.29(4)	C12	$C2^{2}$	3.30(4)
C13	$C10^{2}$	3.41(4)	C13	$C11^{2}$	3.39(4)

C14	$C9^{2}$	3.40(4)	C15	$C8^{2}$	3.36(4)
C17	$C11^{2}$	3.50(4)	C18	$O2^{6}$	3.37(3)
C18	$O4^{2}$	3.35(3)	C18	$N1^{2}$	3.42(3)
C18	$N2^{2}$	3.52(3)	C18	$C11^{2}$	3.21(4)
C19	$O2^{7}$	3.53(3)	C20	$O1^{7}$	3.51(3)
C20	$O2^{7}$	3.52(3)			

Symmetry Operators:

(1) -X,Y,-Z+1/2 (2) X,Y-1,Z

(3) X,Y+1,Z (4) X,-Y+1,Z+1

(5) X,-Y,Z+1 (6) X,-Y,Z

(7) X,-Y+1,Z

Table 13. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
Mo1	H20A[1]	3.588	Mo1	H20C[1]	3.442
Cl1	H5[2]	3.431	Cl1	H7[3]	3.307
S1	H2[4]	3.406	S2	H3A[5]	3.273
S2	H18[5]	3.453	O1	H3A[5]	3.224
O1	H20A[1]	3.037	O1	H20C[1]	3.165
O2	H2[4]	2.444	O2	H3[4]	3.034
O2	H3A[6]	3.511	O2	H18[6]	2.986
O2	H19B[1]	2.873	O2	H19C[1]	3.425
O2	H20A[1]	2.936	O2	H20C[1]	3.326
O3	H19C[1]	3.066	O3	H20C[1]	2.932
O4	H3A[5]	2.248	O4	H18[5]	2.714
N1	H3[4]	3.267	N1	H18[5]	3.278
N2	H17[5]	3.383	N2	H18[5]	3.271
N3	H19B[4]	3.166	C1	H3A[5]	3.577
C1	H17[6]	3.337	C1	H20A[1]	3.354
C2	H17[6]	3.197	C2	H20A[1]	3.321
C3	H17[6]	3.163	C4	H17[6]	3.222
C6	H15[7]	3.541	C7	H15[5]	3.311
C8	H15[5]	3.289	C9	H14[5]	3.510
C9	H17[6]	3.326	C10	H17[6]	3.300
C11	H3[4]	3.352	C11	H18[5]	3.426

C12	H2[4]	3.528	C13	H3[2]	3.138
C13	H11[4]	3.474	C14	H3[2]	3.131
C14	H17[5]	3.577	C15	H3[2]	3.270
C15	H5[2]	3.277	C15	H6[7]	3.529
C15	H8[4]	3.433	C16	H3[2]	3.302
C16	H5[2]	3.206	C16	H8[4]	3.550
C16	H11[4]	3.384	C17	H2A[4]	3.119
C17	H3[2]	3.340	C17	H11[4]	3.020
C17	H14[4]	3.587	C18	H2A[4]	3.319
C18	H3[2]	3.204	C18	H11[4]	3.109
C18	H20A[4]	3.031	C19	H3A[5]	3.100
C20	H2[2]	3.280	C20	H18[5]	2.979
H2	S1[5]	3.406	H2	O2[5]	2.444
H2	C12[5]	3.528	H2	C20[1]	3.280
H2	H20A[1]	2.912	H2	H20B[1]	2.971
H2	H20C[1]	3.443	H2A	C17[5]	3.119

Table 13. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H2A	C18[5]	3.319	H2A	H17[5]	2.761
H2A	H18[5]	3.216	H3	O2[5]	3.034
H3	N1[5]	3.267	H3	C11[5]	3.352
H3	C13[1]	3.138	H3	C14[1]	3.131
H3	C15[1]	3.270	H3	C16[1]	3.302
H3	C17[1]	3.340	H3	C18[1]	3.204
H3	H11[5]	3.565	H3	H14[1]	3.546
H3A	S2[4]	3.273	H3A	O1[4]	3.224
H3A	O2[8]	3.511	H3A	O4[4]	2.248
H3A	C1[4]	3.577	H3A	C19[4]	3.100
H3A	H19A[4]	3.463	H3A	H19B[4]	2.382
H3A	H20A[4]	3.444	H5	Cl1[1]	3.431
H5	C15[1]	3.277	H5	C16[1]	3.206
H5	H8[5]	3.054	H5	H11[5]	3.531
H5	H15[1]	3.334	H6	C15[7]	3.529
H6	H7[9]	3.516	H6	H15[7]	2.616
H7	Cl1[3]	3.307	H7	H6[9]	3.516
H7	H7[7]	3.233	H7	H8[7]	3.318
H7	H15[5]	3.365	H8	C15[5]	3.433
H8	C16[5]	3.550	H8	H5[4]	3.054
H8	H7[7]	3.318	H8	H15[5]	3.468

H11	C13[5]	3.474	H11	C16[5]	3.384
H11	C17[5]	3.020	H11	C18[5]	3.109
H11	H3[4]	3.565	H11	H5[4]	3.531
H11	H17[5]	3.244	H11	H18[5]	3.469
H14	C9[4]	3.510	H14	C17[5]	3.587
H14	H3[2]	3.546	H14	H17[5]	2.978
H15	C6[7]	3.541	H15	C7[4]	3.311
H15	C8[4]	3.289	H15	H5[2]	3.334
H15	H6[7]	2.616	H15	H7[4]	3.365
H15	H8[4]	3.468	H17	N2[4]	3.383
H17	C1[8]	3.337	H17	C2[8]	3.197
H17	C3[8]	3.163	H17	C4[8]	3.222
H17	C9[8]	3.326	H17	C10[8]	3.300
H17	C14[4]	3.577	H17	H2A[4]	2.761
H17	H11[4]	3.244	H17	H14[4]	2.978
H18	S2[4]	3.453	H18	O2[8]	2.986

Table 13. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H18	O4[4]	2.714	H18	N1[4]	3.278
H18	N2[4]	3.271	H18	C11[4]	3.426
H18	C20[4]	2.979	H18	H2A[4]	3.216
H18	H11[4]	3.469	H18	H19B[4]	3.382
H18	H20A[4]	2.116	H18	H20B[4]	3.472
H19A	H3A[5]	3.463	H19B	O2[2]	2.873
H19B	N3[5]	3.166	H19B	H3A[5]	2.382
H19B	H18[5]	3.382	H19B	H20B[5]	2.969
H19C	O2[2]	3.425	H19C	O3[2]	3.066
H20A	Mo1[2]	3.588	H20A	O1[2]	3.037
H20A	O2[2]	2.936	H20A	C1[2]	3.354
H20A	C2[2]	3.321	H20A	C18[5]	3.031
H20A	H2[2]	2.912	H20A	H3A[5]	3.444
H20A	H18[5]	2.116	H20B	H2[2]	2.971
H20B	H18[5]	3.472	H20B	H19B[4]	2.969
H20C	Mo1[2]	3.442	H20C	O1[2]	3.165
H20C	O2[2]	3.326	H20C	O3[2]	2.932
H20C	H2[2]	3.443			

Symmetry Operators:

(1) X,-Y+1,Z+1

(2) X,-Y+1,Z

(3) -X,-Y,-Z+1

(4) X,Y-1,Z

(5) X,Y+1,Z

(6) X,-Y,Z+1

(7) -X,-Y+1,-Z+1

(8) X,-Y,Z

(9) -X,-Y+2,-Z+1

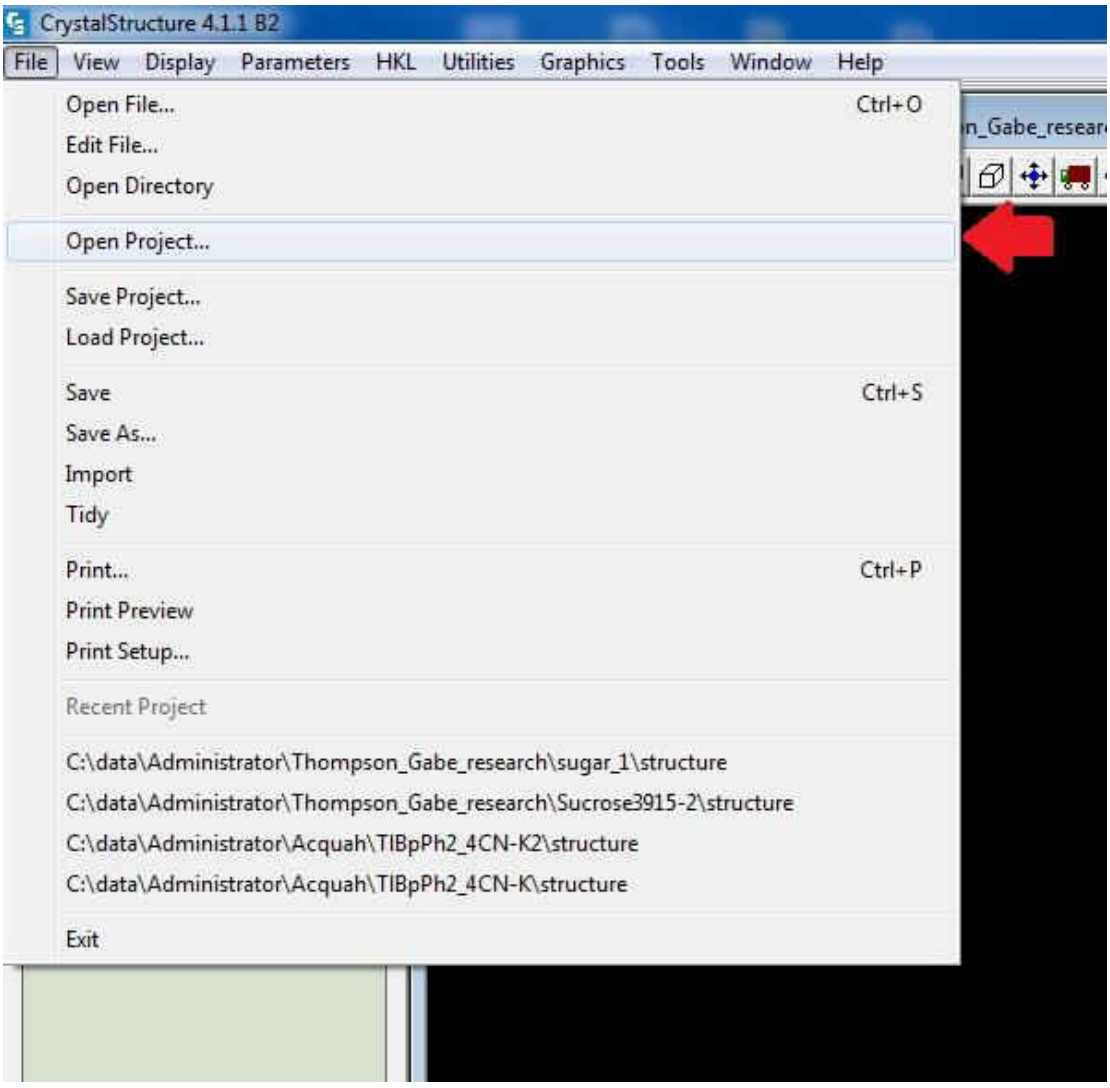
Appendix E: Crystal Structure and Olex Tutorial

Once a crystal has been analyzed using Rigaku's Crystal Clear program, the data needs to be analyzed using another program. This was done using two different programs; the first being Rigaku's Crystal Structure (CS), the second being an open source program called Olex2. The first section will walk through with the procedure in Crystal Structure, and the second will do the same with Olex2.

Crystal Structure:

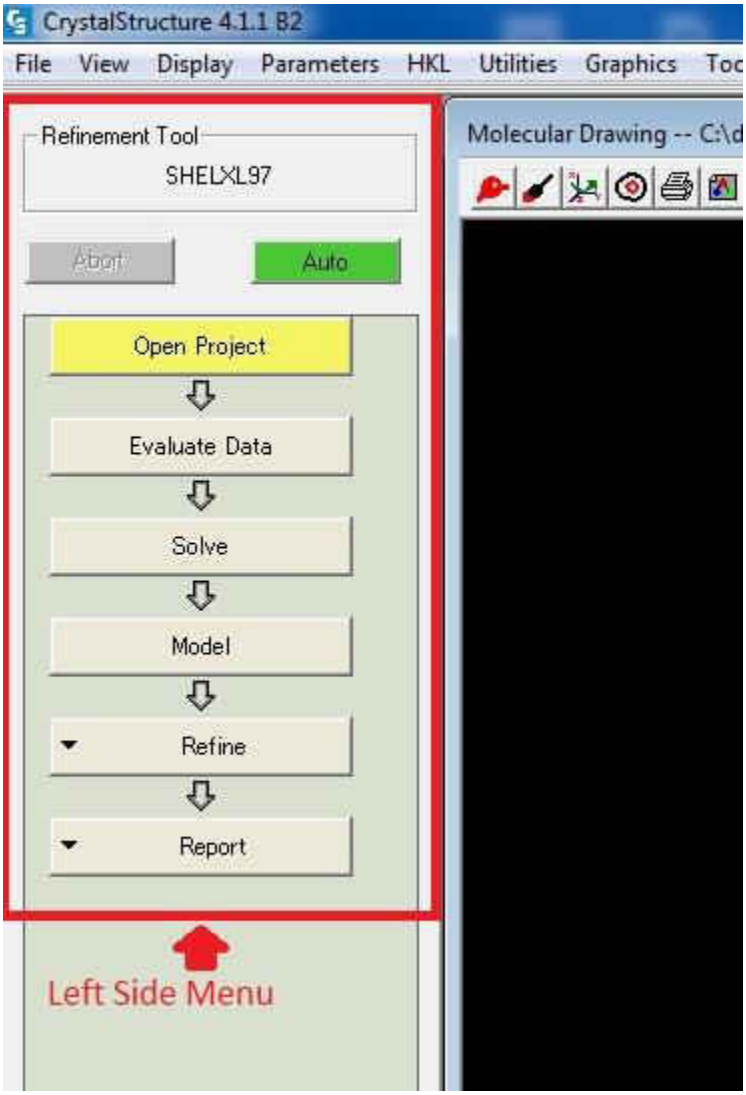
1. Open a file

Click on file from the toolbar and then select the hkl.dat file associated with the project of choice.



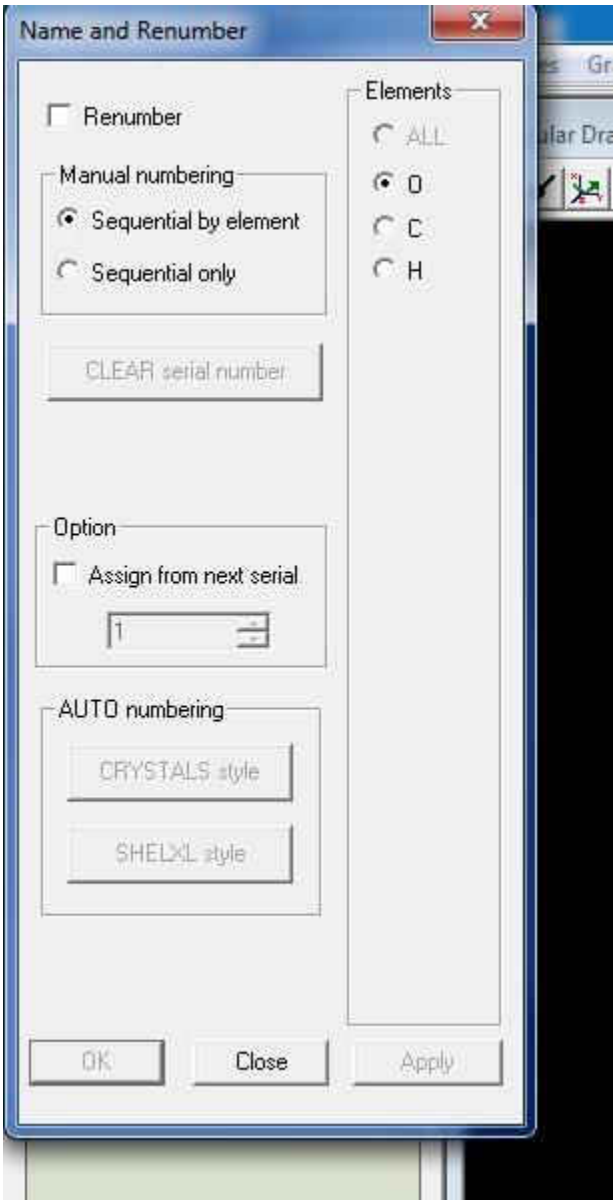
2. Solve for symmetry/space group

Click the “Evaluate Data” icon from the menu on the left side menu, then click “Yes” when the next window pops up. This opens a dialog box in which you can choose parameters such as symmetry, lattice, and space group. Click through each of the categories on the left. The software will pick what it thinks is best for each parameter, and for the first solve, the default options are typically best. After going through all of them, click the “Exit” button on the bottom-left. Next, click “Solve” from the left side menu and select the desired program to solve the structure; this includes both direct and Patterson methods. For the first try, SHELXS97 usually works the best. The program will run and then display an electron density map with the highest peaks and bonds between those peaks if they are within a certain distance from each other.



3. Assign atoms to points of electron density

Click the “Model” icon and then select “Name and Renumber” from the drop-down menu. This opens up a dialog box with all of the possible elements for the structure. Select an element, then select any peaks that you want to become that element, then click “OK” to accept the changes and close the dialog box.

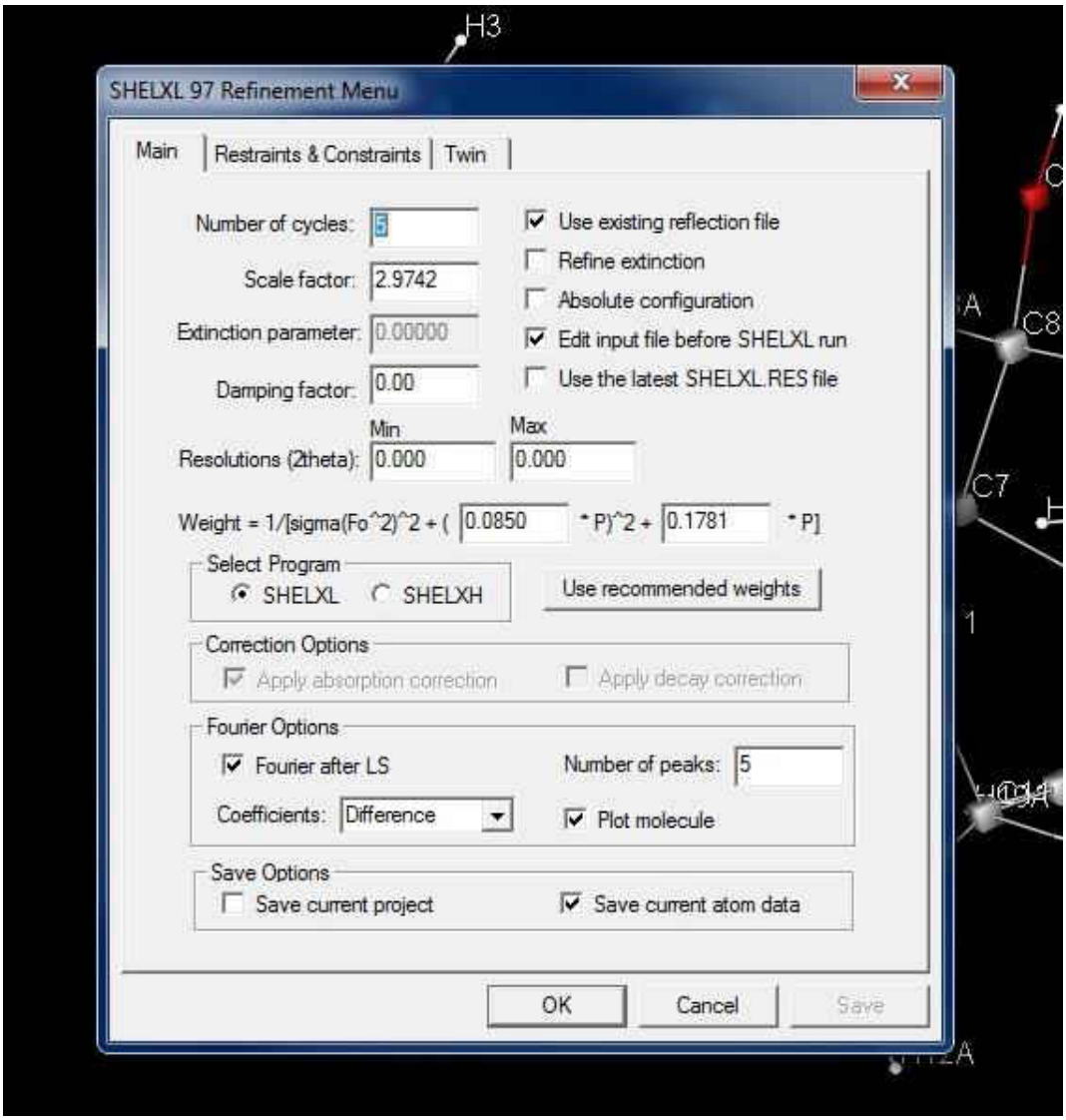


4. Deleting atoms/peaks - The software may have too many peaks, making things cluttered, or you may have assigned a wrong atom that you now want to get rid of.

To delete an atom or peak, right-click on the desired atom/peak and then select “Delete” from the drop-down menu. To delete multiple atoms/peaks at once, right-click and drag the cursor. This will form a box that you can drag over the atoms you wish to be deleted. Upon releasing the right mouse button, a drop-down menu will appear; select “Delete.”

5. Refine - Once the atoms have been placed onto the electron density map, the structure should be refined.

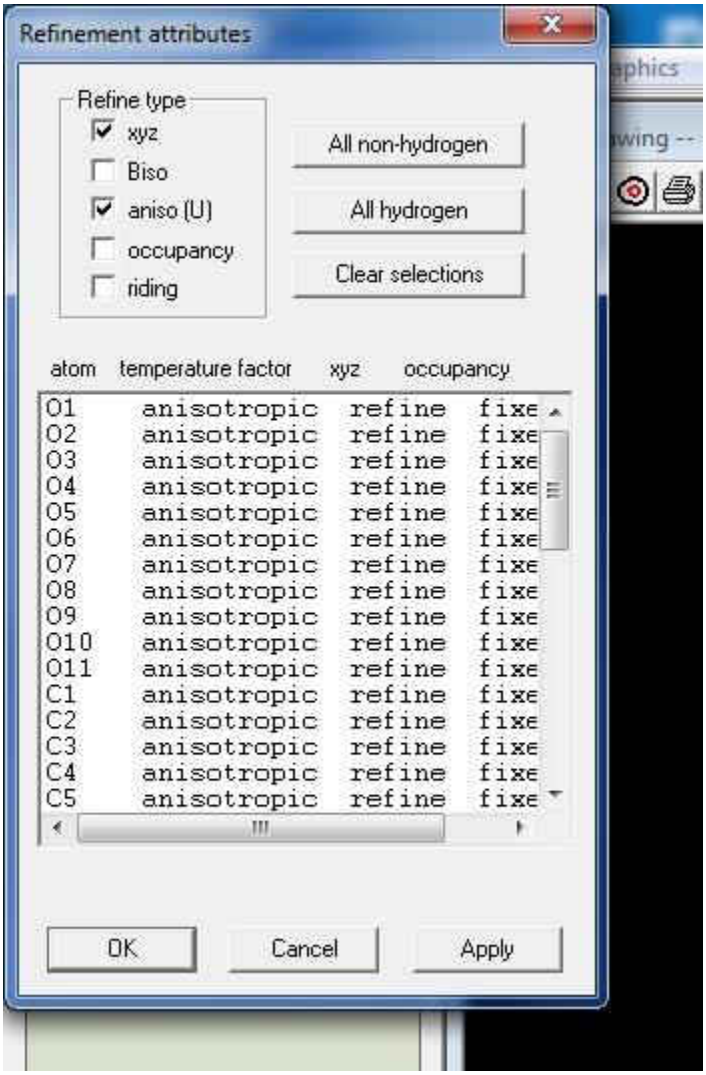
Click the “Refine” icon from the menu on the left, followed by “Least Squares,” and a new dialog box will open. In this dialog box, a number of parameters can be set including the number of refinements and the number of peaks to be displayed upon completion. For the first run, running 5 refinements should be adequate. Click “Run” to begin refinement.



6. Solve anisotropically - Depending on how well the structure solved, you may have to go back and repeat steps 3-5 a few times. Once the structure is looking close to what you think it should, the next step is to solve the atoms anisotropically.

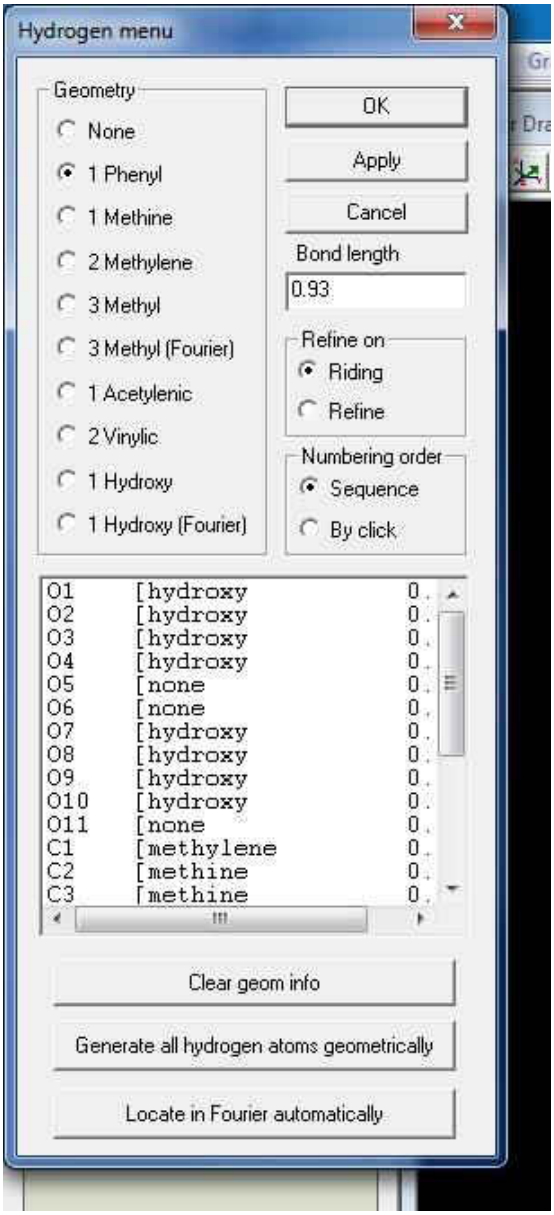
This can be done by clicking the “Model” icon from the menu on the left, then selecting “Refinement Attributes” from the drop down menu. This opens a dialog box with a list of all of the assigned atoms. Check the box next to “aniso” if it was not done automatically, and then

select “All non-hydrogen atoms.” This will then change all of the spheres on the electron density map to squares. Refine the structure again.



7. Add hydrogen atoms

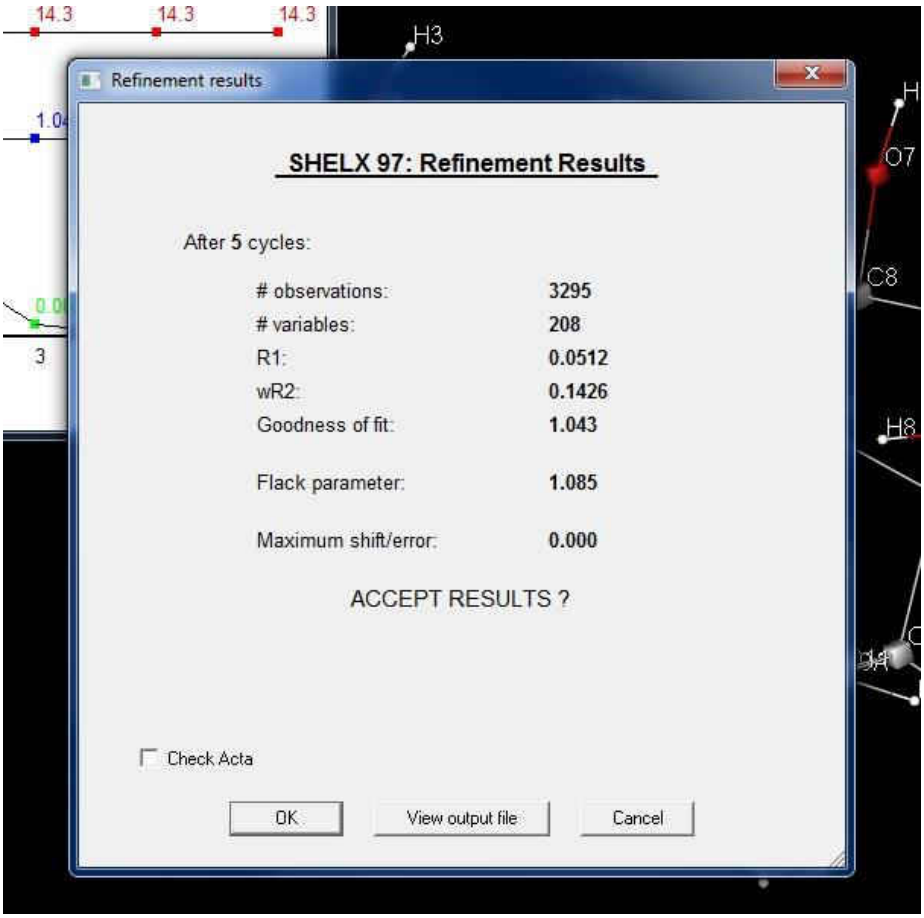
Click the “Model” icon from the left-side menu, and select “Add Hydrogen” from the drop-down menu. This will open up a dialog box listing all of the atoms. Select the type of hydrogen atom to add to a selection of atoms (the program will treat methyl hydrogens differently than phenyl ones for example), then select any atoms to which you want to apply that kind of hydrogen atom, then select “Apply.” Do the same for any other types of hydrogen atoms needed, then click “OK” to close the dialog box.



8. Refinement results – At this point, if the data collected from the diffraction was good, the structure should be close to finished. The way to find out if your structure matches the data well is to look at the refinement results. The values to look at are the R1, wR2, Goodness of Fit (GooF), and Shift/Error (S/E). To have a "good" model, the values should be close to the following:

    - R1: The lower the percentage the better. Less than 10% is generally acceptable.
    - wR2: The lower the percentage the better. Less than 15% is generally acceptable.
    - GooF: The closer to 1 the better. Between 0.8 and 1.2 is generally acceptable.
    - S/E: Should be approaching 0. If not less than 0.1, run more refinements.

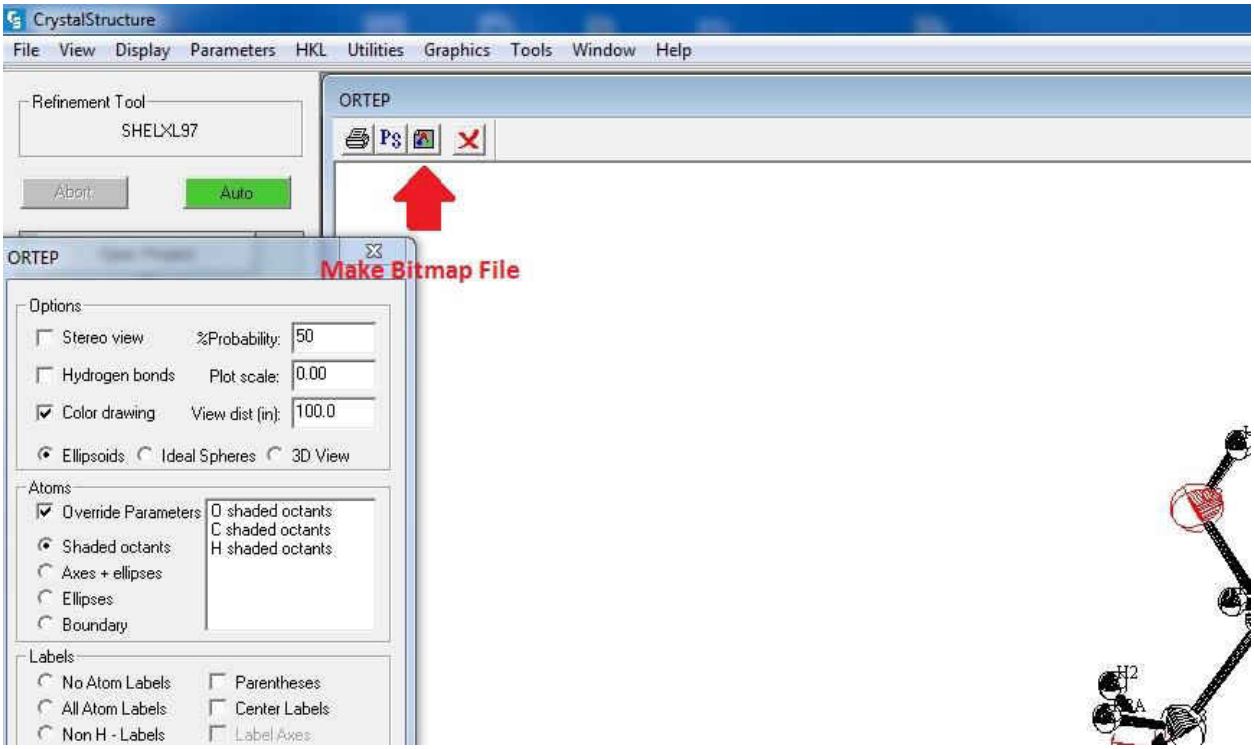
Run a refinement as described in step 5. Upon completion of refinement, a dialog box will appear listing the R1, wR2, GooF, and S/E.



9. Use restraints and constraints – If after all of steps 1-7 have been done, and the refinement attributes are not where they should be, restraints or constraints may need to be used. Further information regarding the use of these can be found in a book in the lab.

10. Generating an ORTEP – It is necessary to generate this so you can show the structure of the model.

To generate an ORTEP, click on “Graphics” on the toolbar, then select “ORTEP” from the drop-down menu. This opens a dialog box with many different options. The main thing that should be changed is that all non-hydrogen atoms should be in the form of a “shaded octant,” which can be selected from the menu. To do this, check the “Override Parameters” box, then click on the atoms to apply the shaded octant view. Next, click “Run” to generate the ORTEP. Move atom labels to make them easily visible, and make any other needed adjustments. Click the “Make Bitmap File” icon to save the ORTEP as a bitmap file that can be used later.



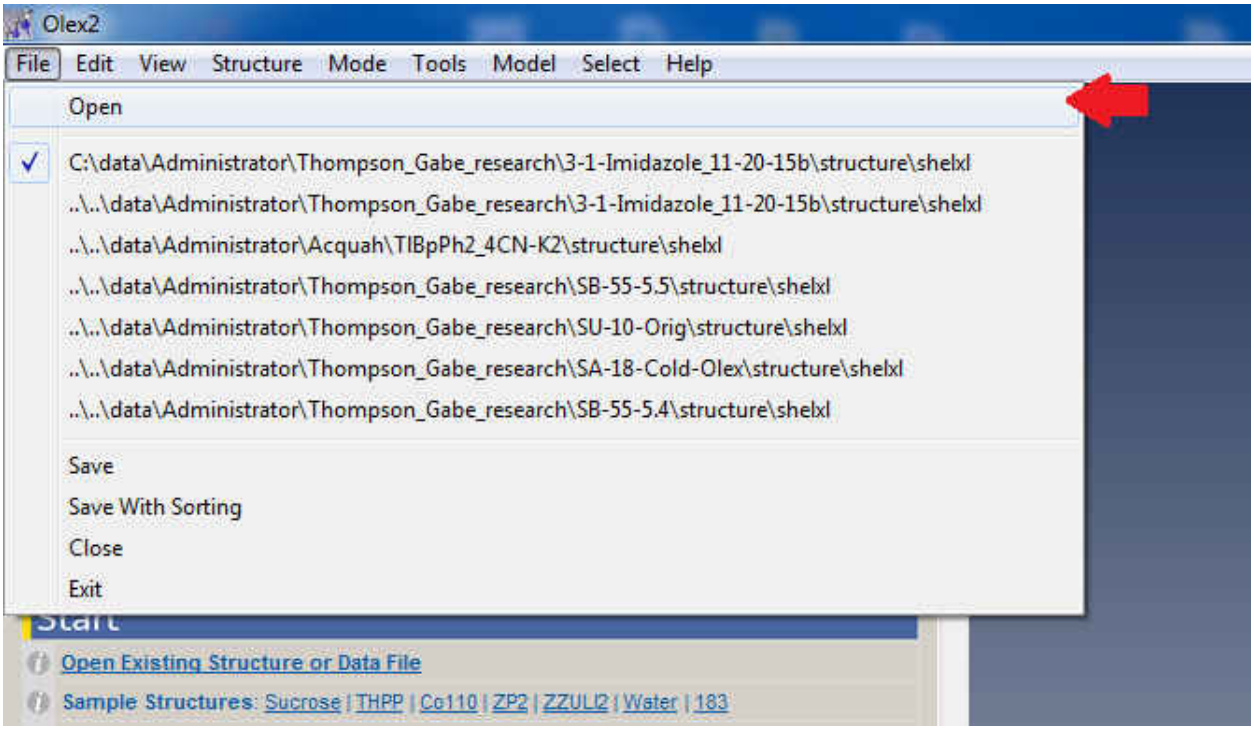
11. Once all of the above steps have been completed and the model sufficiently matches the data, all of the information can be compiled into a report.

Click “Report” from the left side menu, bringing down more options. From here click the second “Report” to open up a dialog box where options can be changed. When ready, click “Generate report.” Also, A CIF file should be generated, which can be done by selecting “CIF” from the left side menu. Select “Yes” when the window prompts you, and the CIF will be made. It can be found in the project folder.

Olex2 Procedure:

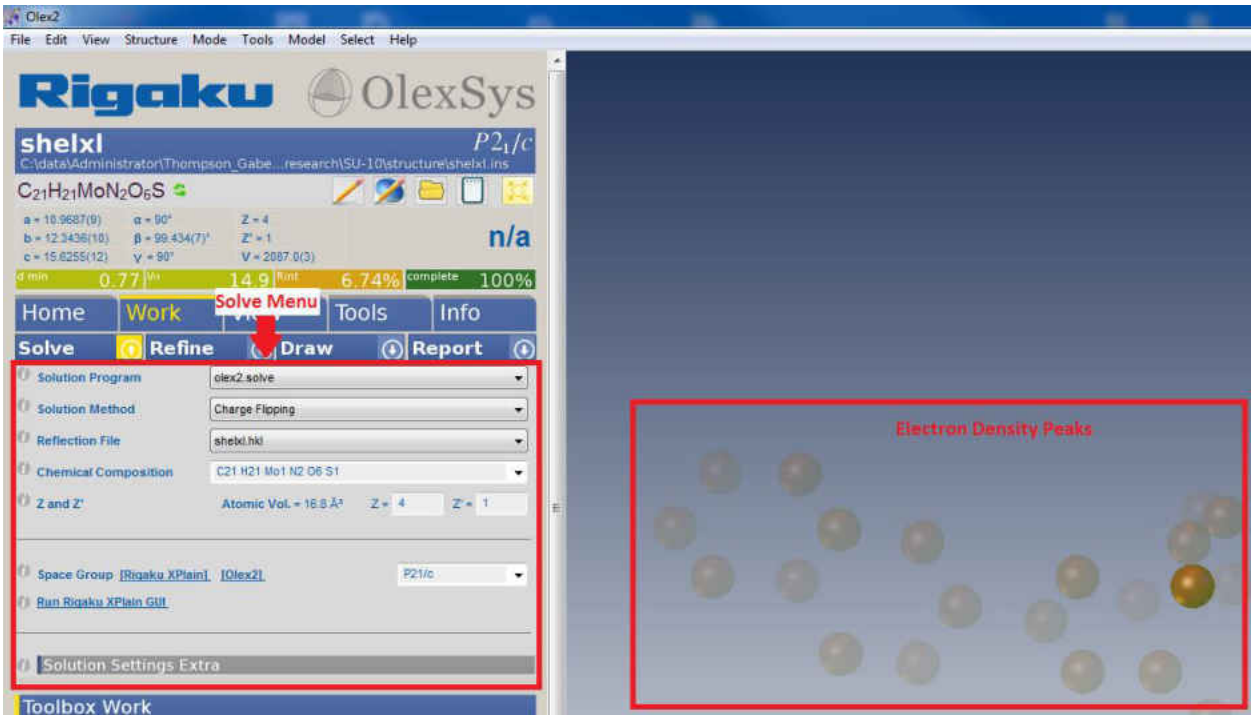
1. Open a file

Click on file from the toolbar, then open, and then select the shelxl.ins file associated with the project of choice.



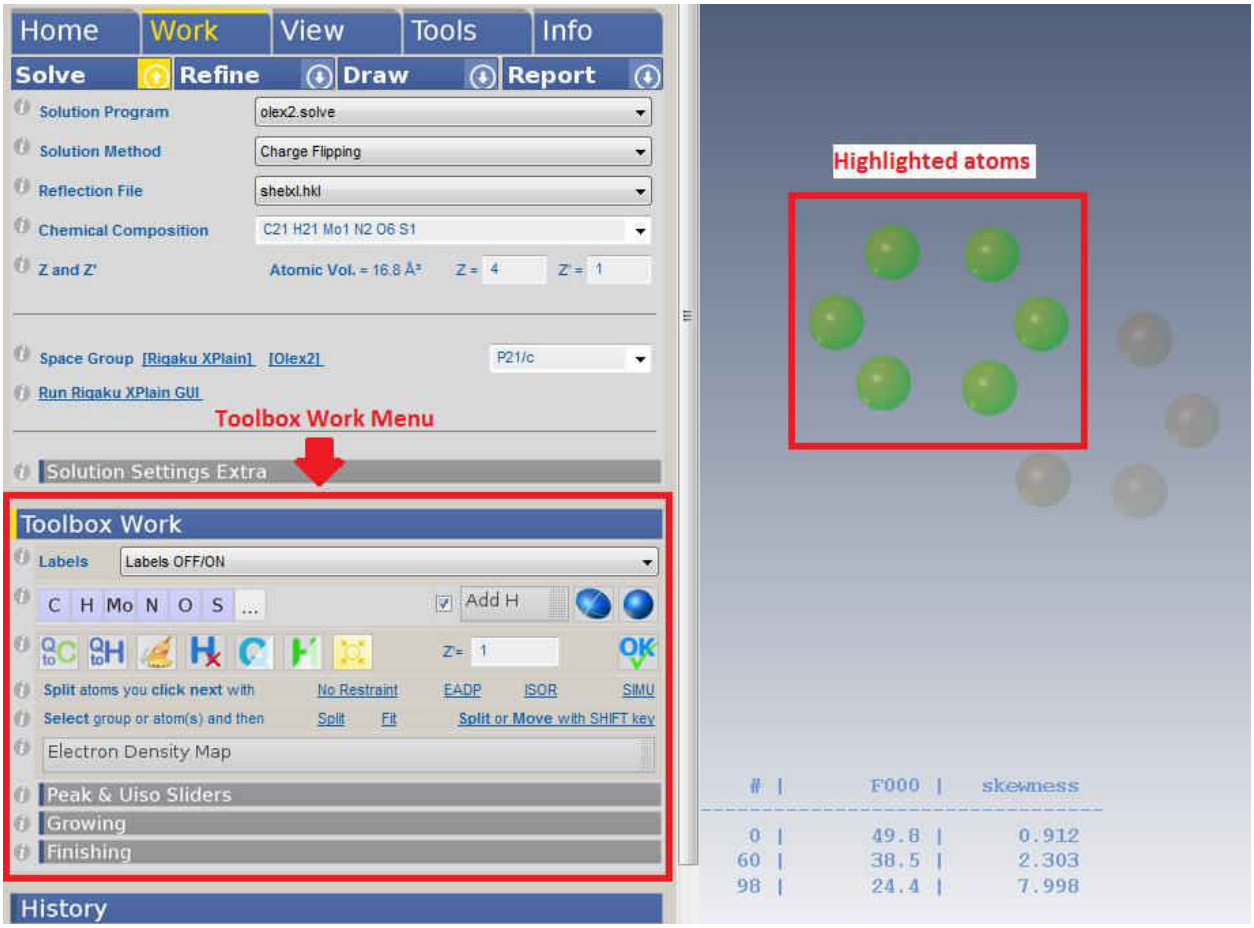
2. Solve for symmetry/space group

Click the “Work” icon from the left side menu, then click the down arrow next to “Solve” to bring up the solve menu. Options like solution program and solution method can be changed from this menu. For the first run, the default settings are usually not a bad option. To solve, click the “Solve” button, and it will solve the structure, bringing up areas of high electron density and picking a space group which will be displayed on the left in the Solve menu.



3. Assign atoms to points of electron density

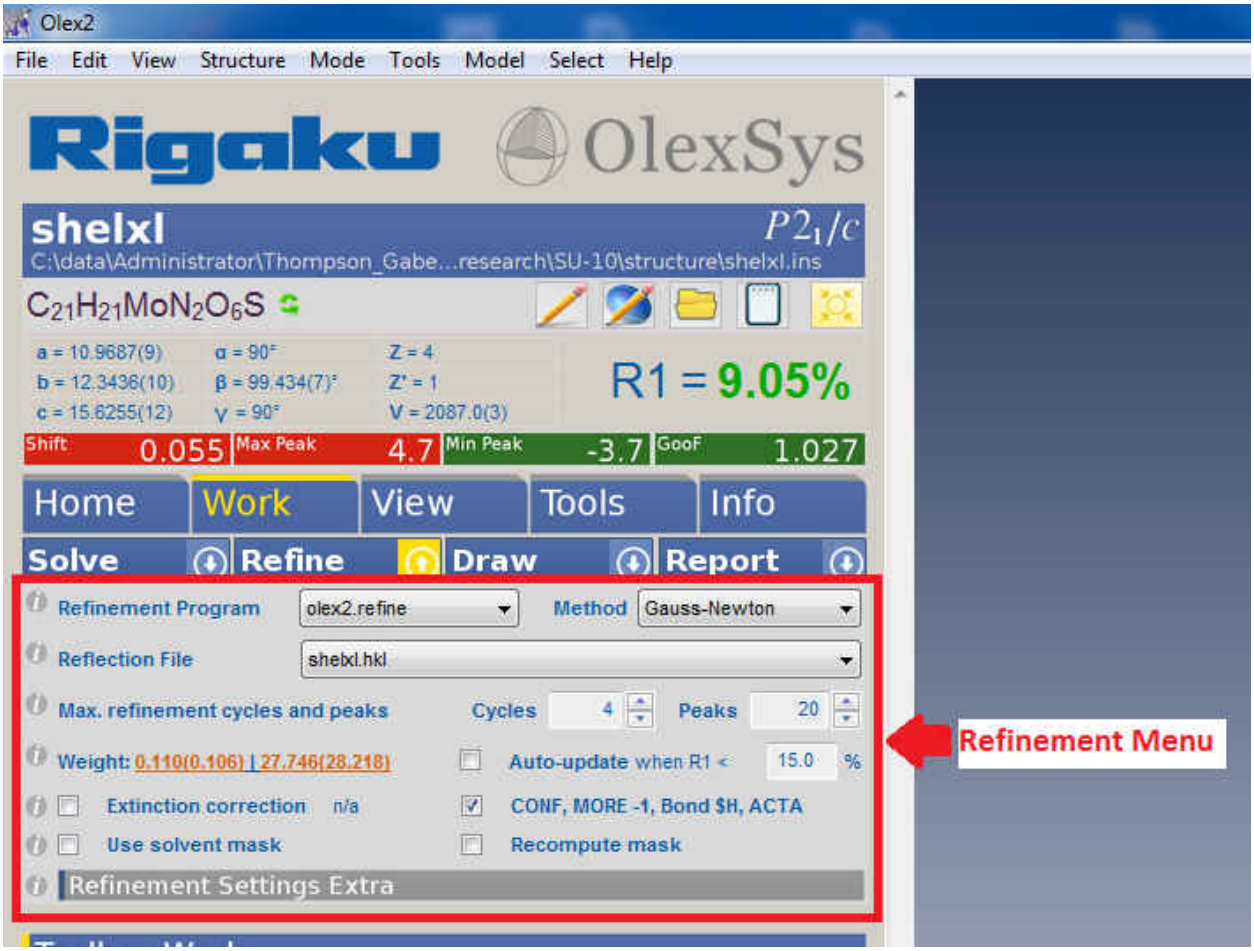
Click on any of the electron density spheres on the map to highlight them, then select the desired element listed in the "Toolbox Work" menu on the left to change the selected peaks to that element. If the desired element is not listed, click the "…" icon next to the listed elements. Spheres within a certain distance from each other will automatically have bonds drawn between them.



4. Deleting atoms/peaks - The software may have too many peaks, making things cluttered, or you may have assigned a wrong atom that you now want to get rid of.

Left click any atoms or peaks you want deleted to select them, then press the “Delete” key on the keyboard.

5. Refine - Once the atoms have been placed onto the electron density map, the structure should be refined.



Click the down arrow next to the "Refine" tab to bring up the refinement options. Refinements can be made using either olex2.refine or SHELXL; these can be selected from a drop-down menu below the "Refine" tab. Once one is selected, and the other parameters are set accordingly, click the "Refine" button.

6. Solve anisotropically - Depending on how well the structure solved, you may have to go back and repeat steps 3-5 a few times. Once the structure is looking close to what you think it should, the next step is to solve the atoms anisotropically.

There is an ellipsoid icon in the "Toolbox Work" menu on the left, which if clicked will solve all non-hydrogen atoms anisotropically and automatically refine the structure.

7. Add hydrogen atoms

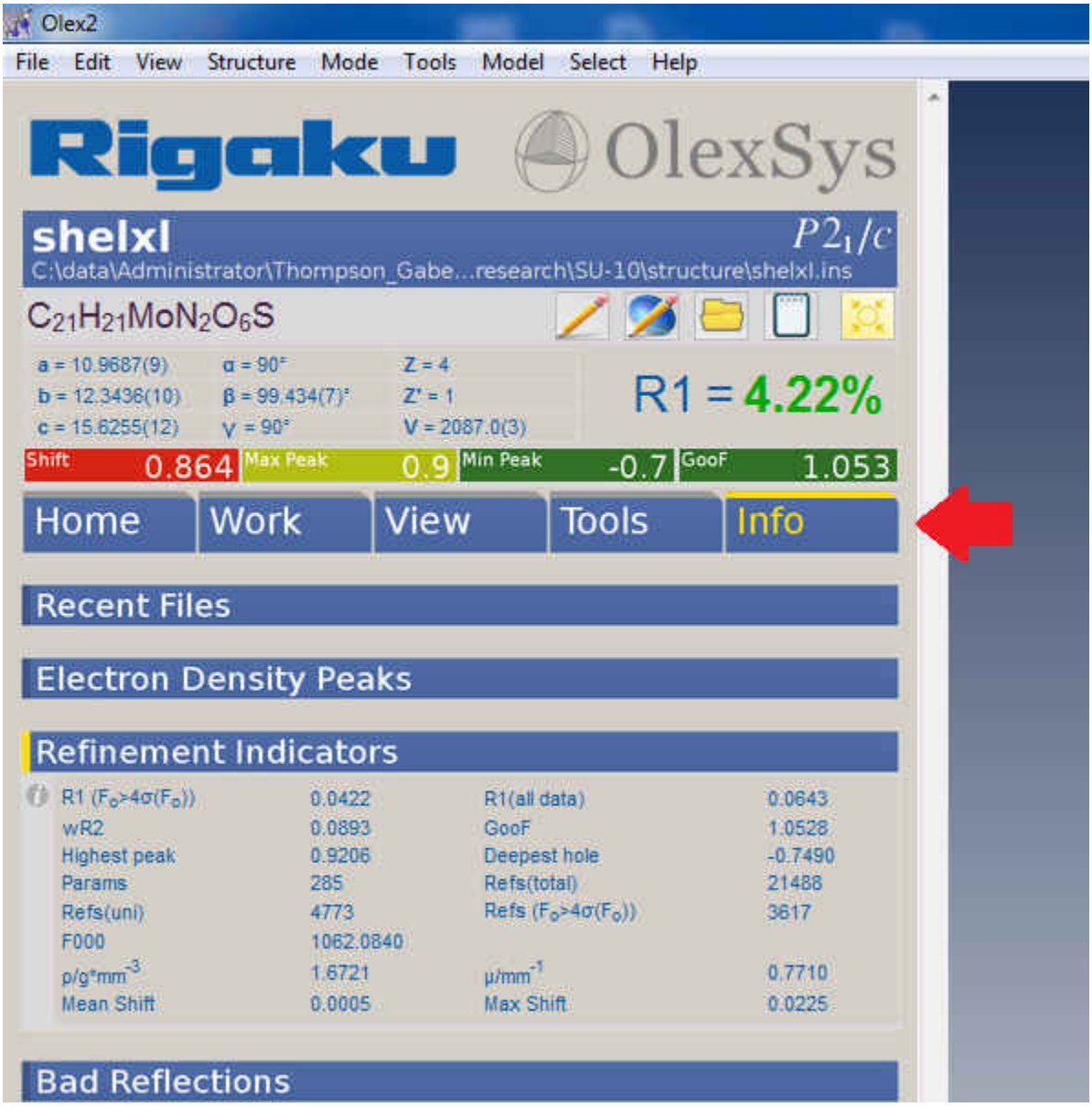
Click the "Add H" icon from the "Toolbox Work" menu on the left, and it will automatically add hydrogen atoms to all the atoms on the map that are electron deficient. Olex may add too many hydrogen atoms in cases where two atoms are bonded with a bond order greater than one, but in general, it does a fairly good job automatically. It is a good idea to double-check the structure and delete any hydrogen atoms that did not add correctly.

8. Refinement results – At this point, if the data collected from the diffraction was good, the structure should be close to finished. The way to find out if your structure matches the data well is to look at the refinement results. The values to look at are the R1, wR2, Goodness of Fit (GooF), and Shift/Error (S/E). To have a "good" model, the values should be close to the following:

    - R1: The lower the percentage the better. Less than 10% is generally acceptable; less than 5% preferred.
    - wR2: The lower the percentage the better. Less than 15% is generally acceptable; less than 10% preferred.
    - GooF: The closer to 1 the better. Between 0.8 and 1.2 is generally acceptable; 0.95-1.1 preferred.

- S/E: Should be approaching 0. If not less than 0.1, run more refinements.

Most of these are displayed on the left-side menu at all times. To find the wR2, click on the "Info" tab and select "Refinement Indicators" to get a full list of all the relevant information.

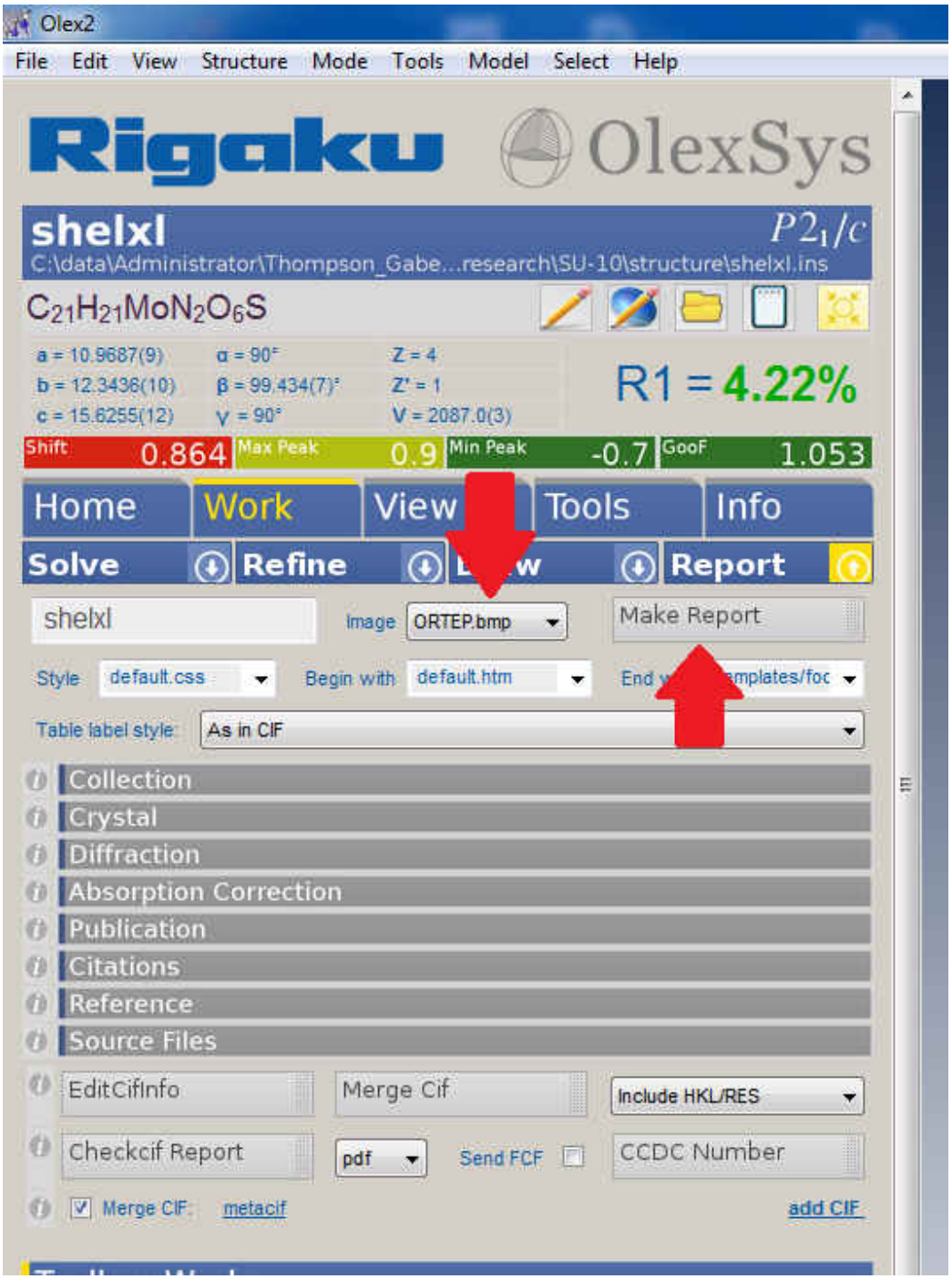


9. Use restraints and constraints – If after all of steps 1-7 have been done, and the refinement attributes are not where they should be, restraints or constraints may need to be used. Further information regarding the use of these can be found in a book in the lab.

To see the list of restraints and constraints in Olex, select the “Tools” tab and then click on either “Olex2 Constraints Restraints,” Shelx Compatible Constraints,” or “Shelx Compatible Restraints.” After clicking one of those, the desired restraint or constraint can be selected from the drop down menu, and further steps can be listed by clicking the “*i*” icon on the far left.

10. Generating an ORTEP – It is necessary to generate this so you can show the structure of the model.

To generate an ORTEP in Olex, click the “Work” tab, followed by the “Report” tab. This will bring up everything necessary for generating an ORTEP as well as a full report. For the ORTEP to be included when a report is generated, click the dropdown menu next to “Image” and select ORTEP.bmp.



11. Once all of the above steps have been completed and the model sufficiently matches the data, all of the information can be compiled into a report.

To generate the report, do everything in Step 10, then click the “Make Report” button.

Appendix F: .ins and .res files Showing Modeling of Disorder

1. .ins File of Cu-1 (SA-18) Before Restraints

```
TITL SA-18-Crystal4
CELL  0.71075 14.52400 21.07100 18.87500 90.00000 110.69000 90.00000
ZERR  8 0.00300 0.00400 0.00300 0.00000 0.00800 0.00000
LATT 1
SYMM    -X, .50+Y, .50-Z
SFAC C H CL CU N O
UNIT 208 288 16 8 16 16
L.S. 5
FMAP 2
PLAN -8
WPDB -2
HTAB
BOND $H
CONF
LIST 4
TEMP 20.0
ACTA
SIZE 0.140 0.240 0.080
WGHT 0.0699 0.0000
FVAR 0.35913
Cu1   4   0.39411   0.51898   0.85264  11.00000   0.05188   0.02968 =
    0.04481  -0.00038   0.01637  0.00581
Cu2   4   0.09521   0.47819   0.65731  11.00000   0.04827   0.03253 =
    0.05137  -0.00068   0.01731  0.00547
```

Cl1 3 0.51329 0.72112 0.59776 11.00000 0.14123 0.05303 =
0.07358 0.00065 0.06391 -0.00795
Cl2 3 0.39102 0.57569 0.45060 11.00000 0.11467 0.05944 =
0.13466 0.00120 0.08078 -0.02283
Cl3 3 -0.00678 0.27005 0.92290 11.00000 0.14700 0.07076 =
0.12135 0.01193 0.09391 -0.00298
Cl4 3 0.19764 0.43489 1.13571 11.00000 0.10612 0.08649 =
0.11427 0.02883 0.07274 0.00662
O1 6 0.28850 0.54190 0.88224 11.00000 0.06486 0.04055 =
0.06662 0.01022 0.04000 0.01024
O45 6 0.43160 0.60349 0.84957 11.00000 0.08676 0.03472 =
0.05082 -0.00145 0.04307 -0.00398
O65 6 0.12330 0.44940 0.57357 11.00000 0.06524 0.03240 =
0.05679 -0.00475 0.01992 0.00305
O66 6 0.13569 0.40258 0.71014 11.00000 0.06566 0.04268 =
0.07306 0.00836 0.03863 0.01601
N3 5 0.46029 0.48856 0.78238 11.00000 0.05679 0.03377 =
0.04966 -0.00273 0.01197 0.01600
N4 5 0.00757 0.50161 0.71559 11.00000 0.04419 0.03338 =
0.05288 0.00035 0.01105 0.01037
N60 5 0.40084 0.42872 0.89185 11.00000 0.04377 0.02889 =
0.03936 0.00158 0.00728 0.00545
N64 5 0.10038 0.57083 0.63161 11.00000 0.05189 0.04721 =
0.05044 -0.00066 0.01736 0.00704
C4 1 0.18334 0.48008 0.54471 11.00000 0.05826 0.03447 =

```
    0.04497  -0.00187   0.00688  -0.00726
C5    1   0.24786   0.55558   0.99120  11.00000   0.04008   0.04346 =
    0.04846   0.00246   0.01767  -0.00501
C6    1   0.18056   0.54537   0.53702  11.00000   0.05486   0.03655 =
    0.04758  -0.00295   0.00945  0.00549
C7    1   0.49605   0.42381   0.80867  11.00000   0.06618   0.03965 =
    0.05500  -0.00235   0.01834  0.02172
AFIX  23
H7A   2   0.55809   0.42630   0.85081  11.00000  -1.2
H7B   2   0.50682   0.40039   0.76804  11.00000  -1.2
AFIX  0
C8    1   0.30442   0.41072   0.89841  11.00000   0.05094   0.03343 =
    0.06900  -0.00825   0.01074  -0.00495
AFIX  23
H8A   2   0.30724   0.36665   0.91389  11.00000  -1.2
H8B   2   0.25291   0.41460   0.84909  11.00000  -1.2
AFIX  0
C9    1   0.24168   0.37089   0.52360  11.00000   0.09700   0.04564 =
    0.05528   0.02384   0.05071  0.03032
AFIX  13
H9    2   0.17305   0.36065   0.51608  11.00000  -1.2
AFIX  0
C10   1   0.24526   0.57711   0.50873  11.00000   0.05938   0.03507 =
    0.06138   0.01402   0.01303  -0.01146
C11   1   0.26582   0.35222   1.03085  11.00000   0.08453   0.03942 =
```

```
    0.10585   0.01147   0.05133  -0.00394
AFIX  137
H11A  2   0.33287   0.33905   1.04265  11.00000  -1.2
H11B  2   0.22416   0.32908   0.98759  11.00000  -1.2
H11C  2   0.24596   0.34392   1.07337  11.00000  -1.2
AFIX  0
C12   1   0.41856   0.69124   0.76939  11.00000   0.06278   0.03177 =
    0.04878  -0.00543   0.02416  0.00667
C13   1   0.09729   0.37405   0.75661  11.00000   0.03436   0.05890 =
    0.04564  -0.00269   0.01182  0.00205
C14   1   0.30925   0.47521   0.49253  11.00000   0.06054   0.04827 =
    0.04672   0.00098   0.01981  0.00532
AFIX  43
H14   2   0.35285   0.45230   0.47630  11.00000  -1.2
AFIX  0
C15   1   0.09637   0.57941   0.55335  11.00000   0.07253   0.03381 =
    0.06116   0.00519   0.01317  0.00641
AFIX  23
H15A  2   0.09927   0.62440   0.54345  11.00000  -1.2
H15B  2   0.03394   0.56340   0.51900  11.00000  -1.2
AFIX  0
C16   1   0.27855   0.45118   0.95425  11.00000   0.03683   0.04403 =
    0.05079  -0.00589   0.01287  -0.00316
C17   1   0.10412   0.30789   0.76219  11.00000   0.05882   0.04189 =
    0.06312  -0.00733   0.03033  0.00630
```

```
C18   1   0.22668   0.52986   1.04991  11.00000   0.05894   0.05246 =
    0.06373  -0.00438   0.03275  0.00717
AFIX  43
H18   2   0.20977   0.55586   1.08323  11.00000  -1.2
AFIX  0
C19   1   0.43985   0.71773   0.70977  11.00000   0.08819   0.02931 =
    0.05630  -0.00318   0.02798  0.00219
AFIX  43
H19   2   0.41797   0.75841   0.69299  11.00000  -1.2
AFIX  0
C20   1   0.15272   0.27213   0.71495  11.00000   0.09659   0.03847 =
    0.06433  -0.01000   0.03695  0.00221
AFIX  13
H20   2   0.20505   0.29985   0.71130  11.00000  -1.2
AFIX  0
C21   1  -0.00078   0.57242   0.71185  11.00000   0.06401   0.03023 =
    0.08505  -0.00559   0.02952  0.01177
AFIX  23
H21A  2   0.05036   0.59103   0.75497  11.00000  -1.2
H21B  2  -0.06408   0.58525   0.71359  11.00000  -1.2
AFIX  0
C22   1   0.24783   0.44326   0.52020  11.00000   0.06086   0.02603 =
    0.04238   0.00383   0.02291  -0.00251
C23   1   0.25713   0.42249   1.01344  11.00000   0.05508   0.04189 =
    0.06647   0.00364   0.02175  -0.00561
```

```
C24   1   0.45243   0.62958   0.79283  11.00000   0.05943   0.04950 =
    0.04666  -0.00508   0.03225  -0.00520
C25   1   0.00935   0.59554   0.63984  11.00000   0.06589   0.03925 =
    0.07594   0.00035   0.02826  0.01349
AFIX  23
H25A  2  -0.04717   0.58185   0.59706  11.00000  -1.2
H25B  2   0.01076   0.64157   0.63989  11.00000  -1.2
AFIX  0
C26   1   0.05343   0.40896   0.80135  11.00000   0.03598   0.04730 =
    0.06530  -0.00088   0.02214  -0.00044
C27   1   0.36077   0.72607   0.81064  11.00000   0.08037   0.04069 =
    0.06861  -0.00868   0.03555  0.00424
AFIX  13
H27   2   0.31507   0.69558   0.81954  11.00000  -1.2
AFIX  0
C28   1   0.30884   0.54016   0.48784  11.00000   0.06106   0.03832 =
    0.08873  -0.00288   0.04489  -0.00377
C29   1   0.26488   0.33845   0.46021  11.00000   0.15852   0.05876 =
    0.10691  -0.01406   0.07097  -0.00478
AFIX  137
H29A  2   0.24231   0.29530   0.45569  11.00000  -1.2
H29B  2   0.33468   0.33906   0.47134  11.00000  -1.2
H29C  2   0.23234   0.36042   0.41347  11.00000  -1.2
AFIX  0
C30   1   0.52805   0.62287   0.69540  11.00000   0.08119   0.04440 =
```

```
    0.07105  -0.00434   0.03960  -0.00275
C31   1   0.24716   0.62712   0.97903  11.00000   0.07827   0.03492 =
    0.06985  -0.00706   0.04523  -0.00250
AFIX  13
H31   2   0.29982   0.63661   0.95950  11.00000  -1.2
AFIX  0
C32   1   0.05023   0.48015   0.79476  11.00000   0.05620   0.05721 =
    0.05353   0.00646   0.02010  0.01741
AFIX  23
H32A  2   0.01149   0.49706   0.82304  11.00000  -1.2
H32B  2   0.11651   0.49680   0.81713  11.00000  -1.2
AFIX  0
C33   1   0.41976   0.38961   0.83343  11.00000   0.08630   0.02726 =
    0.06569  -0.00232   0.03199  0.00923
AFIX  23
H33A  2   0.35934   0.38379   0.79041  11.00000  -1.2
H33B  2   0.44425   0.34817   0.85391  11.00000  -1.2
AFIX  0
C34   1   0.54266   0.53097   0.78736  11.00000   0.06466   0.05197 =
    0.06928  -0.00184   0.03087  0.01032
AFIX  23
H34A  2   0.58017   0.51271   0.75888  11.00000  -1.2
H34B  2   0.58590   0.53425   0.83985  11.00000  -1.2
AFIX  0
C35   1   0.15097   0.64846   0.91912  11.00000   0.11862   0.05682 =
```

```
   0.08879  -0.01115   0.00122  0.02696
AFIX  137
H35A  2   0.09747   0.64000   0.93642  11.00000  -1.2
H35B  2   0.14083   0.62570   0.87292  11.00000  -1.2
H35C  2   0.15395   0.69315   0.91024  11.00000  -1.2
AFIX  0
C36   1   0.49367   0.68360   0.67524  11.00000   0.09478   0.03510 =
   0.05381  -0.00184   0.04635  -0.01132
C37   1   0.07152   0.27811   0.81431  11.00000   0.08097   0.05073 =
   0.07771   0.00447   0.04241  -0.00750
AFIX  43
H37   2   0.07773   0.23430   0.81994  11.00000  -1.2
AFIX  0
C38   1   0.50857   0.59668   0.75717  11.00000   0.05834   0.05342 =
   0.03938  -0.00270   0.02274  -0.00642
C39   1   0.02981   0.31199   0.85839  11.00000   0.09816   0.05746 =
   0.06468  -0.00111   0.04550  -0.00332
C40   1   0.26684   0.66660   1.05036  11.00000   0.10881   0.04568 =
   0.09857  -0.00539   0.02696  0.01537
AFIX  137
H40A  2   0.32402   0.65050   1.08978  11.00000  -1.2
H40B  2   0.21107   0.66422   1.06631  11.00000  -1.2
H40C  2   0.27787   0.70998   1.04001  11.00000  -1.2
AFIX  0
C41   1  -0.01746   0.41508   0.90587  11.00000   0.10820   0.07661 =
```

```
    0.10067   0.00837   0.08388  0.01585
AFIX  137
H41A  2  -0.01307   0.38976   0.94922  11.00000  -1.2
H41B  2  -0.08485   0.42665   0.87939  11.00000  -1.2
H41C  2   0.02148   0.45276   0.92210  11.00000  -1.2
AFIX  0
C42   1   0.24431   0.64821   0.50274  11.00000   0.10681   0.04712 =
    0.10177  -0.00330   0.06474  -0.01223
AFIX  137
H42A  2   0.29506   0.66164   0.48448  11.00000  -1.2
H42B  2   0.25579   0.66647   0.55172  11.00000  -1.2
H42C  2   0.18147   0.66195   0.46818  11.00000  -1.2
AFIX  0
C43   1   0.02040   0.37741   0.85375  11.00000   0.05539   0.07101 =
    0.06699   0.00410   0.04043  0.00194
C44   1   0.30095   0.78070   0.76703  11.00000   0.13454   0.08899 =
    0.10177   0.01716   0.07202  0.04374
AFIX  137
H44A  2   0.25848   0.76618   0.71821  11.00000  -1.2
H44B  2   0.34416   0.81293   0.76075  11.00000  -1.2
H44C  2   0.26196   0.79793   0.79422  11.00000  -1.2
AFIX  0
C46   1   0.30135   0.34502   0.59763  11.00000   0.17350   0.05135 =
    0.11460  -0.01403   0.04637  -0.00638
AFIX  137
```

```
H46A  2   0.28749   0.36782   0.63675  11.00000  -1.2
H46B  2   0.36986   0.34934   0.60477  11.00000  -1.2
H46C  2   0.28583   0.30096   0.59989  11.00000  -1.2
AFIX  0
C47   1   0.39137   0.48460   0.70364  11.00000   0.08436   0.05499 =
     0.06055  -0.01890   0.01366  0.00086
AFIX  137
H47A  2   0.33160   0.46447   0.70259  11.00000  -1.2
H47B  2   0.42061   0.46018   0.67416  11.00000  -1.2
H47C  2   0.37694   0.52655   0.68282  11.00000  -1.2
AFIX  0
C48   1   0.23062   0.46387   1.05959  11.00000   0.07014   0.06394 =
     0.07764   0.01844   0.04794  -0.00115
C49   1   0.08092   0.26447   0.63590  11.00000   0.11137   0.17430 =
     0.11249  -0.05650   0.04570  -0.00786
AFIX  137
H49A  2   0.03024   0.23522   0.63590  11.00000  -1.2
H49B  2   0.11451   0.24842   0.60403  11.00000  -1.2
H49C  2   0.05196   0.30485   0.61706  11.00000  -1.2
AFIX  0
C50   1   0.47950   0.41873   0.96506  11.00000   0.05673   0.04827 =
     0.05948   0.01065   0.00904  0.00940
AFIX  137
H50A  2   0.46526   0.44237   1.00344  11.00000  -1.2
H50B  2   0.54093   0.43288   0.96222  11.00000  -1.2
```

```
H50C  2   0.48390   0.37439   0.97752  11.00000  -1.2
AFIX  0
C51   1   0.42971   0.74893   0.88759  11.00000   0.14533   0.07535 =
    0.06373  -0.01210   0.04315  0.00308
AFIX  137
H51A  2   0.39171   0.76489   0.91626  11.00000  -1.2
H51B  2   0.47109   0.78210   0.88070  11.00000  -1.2
H51C  2   0.46983   0.71422   0.91435  11.00000  -1.2
AFIX  0
C53   1   0.27275   0.51662   0.94116  11.00000   0.04269   0.03391 =
    0.06544   0.00152   0.02792  0.00163
C54   1  -0.09283   0.47415   0.67721  11.00000   0.05308   0.07091 =
    0.06231   0.00322   0.01316  0.01270
AFIX  137
H54A  2  -0.13424   0.48507   0.70532  11.00000  -1.2
H54B  2  -0.08808   0.42881   0.67486  11.00000  -1.2
H54C  2  -0.12059   0.49096   0.62682  11.00000  -1.2
AFIX  0
C55   1   0.18839   0.60346   0.68588  11.00000   0.06244   0.05885 =
    0.07828  -0.00441   0.01074  0.00679
AFIX  137
H55A  2   0.18054   0.64856   0.67931  11.00000  -1.2
H55B  2   0.24629   0.59032   0.67643  11.00000  -1.2
H55C  2   0.19491   0.59247   0.73679  11.00000  -1.2
AFIX  0
```

```
C57   1   0.58492   0.58778   0.65401  11.00000   0.12411   0.05648 =
    0.09807  -0.00538   0.07698  0.00005
AFIX  137
H57A  2   0.64764   0.57490   0.68955  11.00000  -1.2
H57B  2   0.59469   0.61523   0.61669  11.00000  -1.2
H57C  2   0.54847   0.55099   0.62958  11.00000  -1.2
AFIX  0
C58   1   0.20020   0.21269   0.74749  11.00000   0.23198   0.11134 =
    0.13446   0.00543   0.12394  0.06369
AFIX  137
H58A  2   0.23656   0.21853   0.80049  11.00000  -1.2
H58B  2   0.24425   0.19983   0.72239  11.00000  -1.2
H58C  2   0.15107   0.18054   0.74104  11.00000  -1.2
AFIX  0
HKLF 4
END
```

2. .ins File of Cu-1 (SA-18) with FLAT, SADI, and SIMU

```
TITL SB-55-4

CELL  0.71075 42.37600 6.75000 15.49100 90.00000 92.81000 90.00000

ZERR  8 0.01300 0.00200 0.00500 0.00000 0.00700 0.00000

LATT 1

SYMM    -X,    +Y, .50-Z

SFAC C MO S CL O N H

UNIT 160 8 16 8 32 24 152

L.S. 10

FMAP 2

PLAN -10

WPDB -2

HTAB

BOND $H

CONF

LIST 4

TEMP 20.0

ACTA

SIZE 0.120 0.040 0.040

FLAT 0.1000 C17 C29 C28 C20 C19 C18

SADI 0.0200 C17 C29 C28 C20 C19 C18

SIMU 0.0400 0.0800 C17 C29 C28 C20 C19 C18

WGHT 0.1760 0.0000

FVAR 0.46150

Mo1   2   0.18698   0.37281   0.11326  11.00000   0.05386   0.04948 =

    0.04262   0.00075   0.00090  -0.00585
```

Cl30 4 0.03706 -0.52163 -0.20411 11.00000 0.05629 0.22674 =

0.13489 -0.06314 -0.00921 -0.03450

S4 3 0.22308 0.45380 -0.07619 11.00000 0.05542 0.07807 =

0.04651 0.00323 0.01129 0.01323

S27 3 0.19407 0.06627 0.03293 11.00000 0.05668 0.03806 =

0.06283 -0.00789 0.00099 0.00724

O2 5 0.22621 0.40988 0.12665 11.00000 0.03212 0.06678 =

0.05909 -0.01275 -0.00120 -0.02543

O3 5 0.19456 0.52527 -0.02498 11.00000 0.02774 0.06071 =

0.04902 0.00854 0.02391 0.01260

O5 5 0.16876 0.63356 0.12440 11.00000 0.03647 0.09031 =

0.04146 -0.00044 0.00031 0.01858

O9 5 0.17506 0.27540 0.20629 11.00000 0.11578 0.04170 =

0.02053 0.00782 0.01703 -0.00535

N10 6 0.13376 0.16981 -0.01054 11.00000 0.09348 0.05697 =

0.03366 0.00261 0.00770 -0.00757

N12 6 0.13966 0.32502 0.04589 11.00000 0.11086 0.05495 =

0.03499 -0.01917 0.01305 -0.04340

N31 6 0.15270 -0.13517 -0.06176 11.00000 0.02462 0.07418 =

0.06988 -0.03533 0.00138 -0.01390

C6 1 0.15466 0.03740 -0.01175 11.00000 0.01317 0.03782 =

0.06446 0.00093 -0.00307 0.00743

C7 1 0.23553 0.67813 -0.13095 11.00000 0.07793 0.05383 =

0.05010 0.01488 -0.02415 -0.01125

C8 1 0.20522 0.32493 -0.17121 11.00000 0.20404 0.02578 =

```
    0.04735  -0.00755   0.03398  -0.02013
C11   1   0.11199   0.61732   0.10416  11.00000   0.11903   0.03566 =
    0.07095  -0.02168  -0.03011  0.00425
C13   1   0.02740   0.74833   0.08308  11.00000   0.02623   0.10841 =
    0.19920  -0.05180  -0.00719  0.04762
AFIX  43
H13   7   0.00856   0.69868   0.05850  11.00000  -1.2
AFIX  0
C14   1   0.02432   0.91958   0.11919  11.00000   0.06226   0.15503 =
    0.16283  -0.06580  -0.06115  0.03693
AFIX  43
H14   7   0.00517   0.98389   0.12562  11.00000  -1.2
AFIX  0
C15   1   0.05512   0.61287   0.07259  11.00000   0.06655   0.13714 =
    0.02094   0.02236  -0.01038  0.00360
AFIX  43
H15   7   0.05507   0.48565   0.04954  11.00000  -1.2
AFIX  0
C16   1   0.13967   0.70851   0.13768  11.00000   0.05712   0.07479 =
    0.01852   0.01071  -0.01096  -0.01474
C17   1   0.12468  -0.20313  -0.09657  11.00000   0.03312   0.08529 =
    0.03165   0.00771  -0.01165  -0.01949
C18   1   0.12590  -0.40223  -0.13862  11.00000   0.02204   0.03614 =
    0.10629  -0.01733  -0.01002  -0.01404
AFIX  43
```

```
H18   7   0.14515  -0.46585  -0.14422  11.00000  -1.2
AFIX  0
C19   1   0.09835  -0.49007  -0.16885  11.00000   0.06491   0.03654 =
    0.07500  -0.00817   0.02494  -0.01880
AFIX  43
H19   7   0.09931  -0.61850  -0.19031  11.00000  -1.2
AFIX  0
C20   1   0.07056  -0.40192  -0.16910  11.00000   0.02345   0.20093 =
    0.05098  -0.04113   0.01595  -0.00384
C21   1   0.11487   0.43862   0.05240  11.00000   0.01373   0.06510 =
    0.07508  -0.02071   0.01770  0.00982
C22   1   0.08149   0.72449   0.10614  11.00000   0.04990   0.06435 =
    0.05588  -0.00238  -0.01976  0.00043
C23   1   0.08196   0.90755   0.14969  11.00000   0.07036   0.05998 =
    0.03074  -0.00448  -0.00125  -0.00883
C24   1   0.05318   0.99268   0.14657  11.00000   0.09566   0.12782 =
    0.08263   0.03353   0.05503  0.08876
AFIX  43
H24   7   0.05290   1.12275   0.16632  11.00000  -1.2
AFIX  0
C25   1   0.10958   0.97199   0.18240  11.00000   0.09039   0.05764 =
    0.06558   0.03902   0.00060  0.01851
AFIX  43
H25   7   0.10939   1.09554   0.20893  11.00000  -1.2
AFIX  0
```

```
C26   1   0.13774   0.88168   0.18208  11.00000   0.08344   0.07479 =
    0.06284   0.02829  -0.00901  0.02718
AFIX  43
H26   7   0.15541   0.93567   0.21130  11.00000  -1.2
AFIX  0
C28   1   0.06847  -0.22307  -0.12846  11.00000   0.07584   0.13515 =
    0.11928  -0.02067   0.00460  0.08283
AFIX  43
H28   7   0.04821  -0.17461  -0.12153  11.00000  -1.2
AFIX  0
C29   1   0.09336  -0.10525  -0.09624  11.00000   0.06387   0.07611 =
    0.05945  -0.01802  -0.01060  -0.00046
AFIX  43
H29   7   0.09058   0.02323  -0.07632  11.00000  -1.2
AFIX  0
HKLF 4
END
```

3. .ins File of Mo-2 (SB-55)

```
TITL SB-55-4
CELL  0.71075 42.37600 6.75000 15.49100 90.00000 92.81000 90.00000
ZERR  8 0.01300 0.00200 0.00500 0.00000 0.00700 0.00000
LATT 7
SYMM   -X,   +Y, .50-Z
SFAC C MO CL S O N H
UNIT 160 8 8 16 32 24 152
L.S. 5
FMAP 2
PLAN -10
WPDB -2
HTAB
BOND $H
CONF
LIST 4
TEMP 20.0
ACTA
SIZE 0.120 0.040 0.040
WGHT 0.1682 0.0000
FVAR 0.22944
Mo1   2   0.18699   0.37286   0.11330  11.00000   0.05353   0.04883 =
    0.04239   0.00038   0.00096  -0.00587
Cl30  3   0.03709  -0.52150  -0.20376  11.00000   0.05535   0.22866 =
    0.13160  -0.05846  -0.00911  -0.03498
S4    4   0.22308   0.45413  -0.07606  11.00000   0.05594   0.07569 =
```

0.04575 0.00308 0.01160 0.01272

S27 4 0.19407 0.06607 0.03263 11.00000 0.05636 0.03792 =

0.06184 -0.00807 0.00108 0.00769

O2 5 0.22623 0.40964 0.12691 11.00000 0.03008 0.06522 =

0.05993 -0.01259 -0.00256 -0.02394

O3 5 0.19451 0.52503 -0.02510 11.00000 0.02691 0.05919 =

0.04644 0.00890 0.02155 0.01224

O5 5 0.16871 0.63447 0.12398 11.00000 0.03632 0.09044 =

0.04063 -0.00415 -0.00010 0.01942

O9 5 0.17528 0.27550 0.20633 11.00000 0.11698 0.04294 =

0.01780 0.00909 0.01643 -0.00545

N10 6 0.13368 0.16932 -0.01096 11.00000 0.09573 0.05732 =

0.02860 -0.00201 0.00616 -0.01112

N12 6 0.13973 0.32451 0.04515 11.00000 0.09530 0.05085 =

0.03951 -0.01625 0.00899 -0.03357

N31 6 0.15283 -0.13588 -0.06238 11.00000 0.02410 0.07188 =

0.06816 -0.03286 0.00104 -0.01421

C6 1 0.15477 0.03714 -0.01247 11.00000 0.01345 0.04075 =

0.06216 0.00260 -0.00272 0.00767

C7 1 0.23511 0.67327 -0.12911 11.00000 0.07717 0.06513 =

0.05148 0.01161 -0.02730 -0.01060

AFIX 137

H7A 7 0.24967 0.74559 -0.09159 11.00000 -1.2

H7B 7 0.21695 0.75411 -0.14325 11.00000 -1.2

H7C 7 0.24521 0.63837 -0.18113 11.00000 -1.2

```
AFIX  0
C8    1   0.20619   0.32638  -0.16986  11.00000   0.22438   0.02419 =
     0.05054  -0.00289   0.04161  -0.01765
AFIX  137
H8A   7   0.18524   0.37534  -0.18311  11.00000  -1.2
H8B   7   0.20530   0.18691  -0.15811  11.00000  -1.2
H8C   7   0.21907   0.34904  -0.21819  11.00000  -1.2
AFIX  0
C11   1   0.11198   0.61913   0.10424  11.00000   0.11654   0.03707 =
     0.07179  -0.02922  -0.02595  0.00701
C13   1   0.02712   0.74832   0.08039  11.00000   0.02963   0.10887 =
     0.17154  -0.04396  -0.00516  0.04819
AFIX  43
H13   7   0.00835   0.70018   0.05474  11.00000  -1.2
AFIX  0
C14   1   0.02460   0.92056   0.11769  11.00000   0.06537   0.16382 =
     0.14680  -0.05977  -0.05544  0.03391
AFIX  43
H14   7   0.00548   0.98540   0.12393  11.00000  -1.2
AFIX  0
C15   1   0.05513   0.61420   0.07172  11.00000   0.07021   0.13782 =
     0.02034   0.01970  -0.00823  0.00386
AFIX  43
H15   7   0.05520   0.48639   0.04932  11.00000  -1.2
AFIX  0
```

```
C16   1   0.13960   0.70935   0.13759  11.00000   0.05859   0.07528 =
    0.01793   0.00963  -0.01233  -0.01664
C17   1   0.12481  -0.20336  -0.09732  11.00000   0.03432   0.08271 =
    0.02937   0.00700  -0.00921  -0.02030
C18   1   0.12603  -0.40204  -0.13837  11.00000   0.02095   0.03910 =
    0.10797  -0.01868  -0.00778  -0.01391
AFIX  43
H18   7   0.14527  -0.46574  -0.14408  11.00000  -1.2
AFIX  0
C19   1   0.09810  -0.49101  -0.16800  11.00000   0.07034   0.03958 =
    0.07015  -0.00581   0.02450  -0.02319
AFIX  43
H19   7   0.09878  -0.62075  -0.18803  11.00000  -1.2
AFIX  0
C20   1   0.07034  -0.39864  -0.16900  11.00000   0.01844   0.20795 =
    0.05472  -0.04658   0.01887  -0.00567
C21   1   0.11535   0.43986   0.05329  11.00000   0.00590   0.07123 =
    0.07548  -0.01999   0.01121  0.00566
AFIX  43
H21   7   0.09722   0.40135   0.02142  11.00000  -1.2
AFIX  0
C22   1   0.08168   0.72622   0.10549  11.00000   0.04445   0.06408 =
    0.05628  -0.00401  -0.01991  0.00048
C23   1   0.08225   0.90695   0.14968  11.00000   0.07341   0.05599 =
    0.03149  -0.00447  -0.00015  -0.00754
```

C24 1 0.05341 0.99554 0.14676 11.00000 0.09952 0.12169 =

0.07390 0.02777 0.05495 0.08212

AFIX 43

H24 7 0.05310 1.12496 0.16729 11.00000 -1.2

AFIX 0

C25 1 0.10970 0.97140 0.18178 11.00000 0.08556 0.05502 =

0.06277 0.03392 -0.00716 0.01496

AFIX 43

H25 7 0.10951 1.09553 0.20780 11.00000 -1.2

AFIX 0

C26 1 0.13759 0.88227 0.18182 11.00000 0.07866 0.07182 =

0.06488 0.02318 -0.01093 0.02333

AFIX 43

H26 7 0.15521 0.93643 0.21120 11.00000 -1.2

AFIX 0

C28 1 0.06816 -0.22449 -0.12918 11.00000 0.08000 0.12474 =

0.10825 -0.01812 0.00351 0.08160

AFIX 43

H28 7 0.04790 -0.17663 -0.12170 11.00000 -1.2

AFIX 0

C29 1 0.09349 -0.10531 -0.09722 11.00000 0.06757 0.07539 =

0.05120 -0.01637 -0.00824 0.00161

AFIX 43

H29 7 0.09078 0.02362 -0.07776 11.00000 -1.2

AFIX 0

HKLF 4

END

4. .res File of Mo-2 (SB-55)

```
TITL SB-55-4
CELL  0.71075 42.37600 6.75000 15.49100 90.00000 92.81000 90.00000
ZERR  8 0.01300 0.00200 0.00500 0.00000 0.00700 0.00000
LATT 1
SYMM    -X,    +Y, .50-Z
SFAC C MO S CL O N H
UNIT 160 8 16 8 32 24 152
L.S. 5
FMAP 2
PLAN -10
WPDB -2
HTAB
BOND $H
CONF
LIST 4
TEMP 20.0
ACTA
SIZE 0.120 0.040 0.040
FLAT 0.1000 C16 C26 C25 C23 C24 C14 C13 C15 C22 N32
FLAT 0.1000 C17 C29 C28 C20 C19 C18 Cl30
SADI 0.0200 C16 C26 C25 C23 C24 C14 C13 C15 C22 N32
SADI 0.0200 C17 C29 C28 C20 C19 C18
SIMU 0.0400 0.0800 C17 C29 C20 C19 C18
SIMU 0.0400 0.0800 C16 C26 C25 C23 C24 C14 C13 C15 C22 N32
WGHT    0.168100
```

FVAR 0.45798

.

.

.

REM SB-55-4

REM R1 = 0.1780 for 1262 Fo > 4sig(Fo) and 0.3948 for all 4861 data

REM 282 parameters refined using 114 restraints

END

WGHT 0.1681 0.0000

REM Highest difference peak 0.380, deepest hole -0.645, 1-sigma level 0.103

Q1 1 0.3280 0.3740 0.1377 11.00000 0.05 0.38

Q2 1 0.1723 0.1250 -0.1348 11.00000 0.05 0.38

Q3 1 0.0812 -0.3476 -0.0631 11.00000 0.05 0.36

Q4 1 0.3542 0.2920 0.1305 11.00000 0.05 0.36

Q5 1 0.1463 0.2073 -0.1283 11.00000 0.05 0.36

Q6 1 0.4192 0.8446 0.0658 11.00000 0.05 0.36

Q7 1 0.2368 0.0259 0.0101 11.00000 0.05 0.35

Q8 1 0.2404 0.0398 -0.0696 11.00000 0.05 0.34

Q9 1 0.2600 0.4592 0.0726 11.00000 0.05 0.34

Q10 1 0.0084 -0.6740 -0.2215 11.00000 0.05 0.34

5. .res of Cu-1 (SU-10)

```
TITL SU-10
CELL  0.71075 10.96870 12.34360 15.62550 90.00000 99.43400 90.00000
ZERR  4 0.00090 0.00100 0.00120 0.00000 0.00700 0.00000
LATT 1
SYMM    -X, .50+Y, .50-Z
SFAC C H MO N O S
UNIT 84 84 4 8 24 4
L.S. 20
FMAP 2
PLAN -5
WPDB -2
HTAB
BOND $H
CONF
LIST 4
TEMP 20.0
ACTA
SIZE 0.180 0.200 0.090
WGHT    0.026100    3.157000
FVAR       0.87877
.
.
.
REM  SU-10
REM R1 =  0.0427 for   3617 Fo > 4sig(Fo)  and  0.0650 for all   4773 data
```

REM 291 parameters refined using 0 restraints

END

WGHT 0.0261 3.1625

REM Highest difference peak 0.599, deepest hole -0.746, 1-sigma level 0.092

Q1 1 0.0299 0.9340 0.3545 11.00000 0.05 0.60

Q2 1 0.2194 0.5610 0.5378 11.00000 0.05 0.46

Q3 1 0.2413 0.8841 0.3339 11.00000 0.05 0.42

Q4 1 0.2698 0.8192 0.3635 11.00000 0.05 0.37

Q5 1 0.1100 0.5981 0.3695 11.00000 0.05 0.36

VITA

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	Gabriel Thompson, "Single Crystal X-Ray Diffraction Modeling of Disorder in Molybdenum and Copper Enzyme Like Complexes" Appalachian Student Research Forum, Johnson City, Tennessee, 2016
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