1971

A structural investigation of the hydrogen bonding in copper (II), nickel (II), and palladium (II) complexes of some vic-dioximes

Raymond Harold Bowers
Iowa State University

Recommended Citation

Bowers, Raymond Harold, "A structural investigation of the hydrogen bonding in copper (II), nickel (II), and palladium (II) complexes of some vic-dioximes " (1971). *Retrospective Theses and Dissertations*. 4870.
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71-26,844

BOWERS, Raymond Harold, 1944-
A STRUCTURAL INVESTIGATION OF THE HYDROGEN
BONDING IN COPPER(II), NICKEL(II), AND
PALLADIUM(II) COMPLEXES OF SOME VIC-DIOXIMES.

Iowa State University, Ph.D., 1971
Chemistry, analytical

University Microfilms, A XEROX Company, Ann Arbor, Michigan

A structural investigation of the hydrogen bonding in copper(II), nickel(II), and palladium(II) complexes of some *vic*-dioximes

by

Raymond Harold Bowers

A Dissertation Submitted to the
Graduate Faculty in Partial Fulfillment of
The Requirements for the Degree of
DOCTOR OF PHILOSOPHY

Major Subject: Analytical Chemistry

Approved:

Signature was redacted for privacy.

In Charge of Major Work

Signature was redacted for privacy.

Head of Major Department

Signature was redacted for privacy.

Dean of Graduate College

Iowa State University
Of Science and Technology
Ames, Iowa

1971

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

I. INTRODUCTION

Early interest in the metal(II)-*vic*-dioximes arose as a result of the fact that dimethylglyoxime came as close, perhaps, as any compound to being a specific analytical reagent. As a result, a large number of *vic*-dioximes and their complexes with divalent transition metals have been prepared and studied. These investigations have included spectral, magnetic, and solubility studies, and application of the law of mass action to equilibrium studies of chemical reactions in solution.

More recently, structural investigations of several metal(II)-*vic*-dioxime complexes have been carried out using X-ray diffraction techniques. Many of these structural determinations, while not particularly precise, have provided valuable information which has been used to explain differences in the solubilities of several metal(II)-*vic*-dioximes in inert solvents. In addition, these investigations have confirmed the presence of short intramolecular hydrogen bonds in many of the complexes. Such bonds had been predicted to exist in the metal(II)-*vic*-dioximes on the basis of chemical information on the reactivities of the hydroxyl hydrogen atoms in the complexes. Of particular interest were some copper(II)- and nickel(II)-*vic*-dioximes which were thought to possess symmetric hydrogen bonds.

The major part of this investigation consisted of the precise determination of the crystal and molecular structures of copper dimethylglyoxime, deuterium substituted copper dimethylglyoxime, nickel ethylmethylglyoxime, and nickel dimethylglyoxime, in order to elucidate more

completely the nature of the hydrogen bonding in these metal(II)-*vic*-dioximes.

In addition, the hydrogen bonding in a series of palladium(II)-*vic*-dioximes was examined by infrared spectroscopic techniques and by chemical methods which involved the determination of the reactivity of the hydroxyl hydrogen atom in these complexes.

Based on this investigation, a number of conclusions are drawn regarding the hydrogen bonding in the metal(II)-*vic*-dioximes, and the unusual stability of these complexes, and some additional investigations are proposed.

II. ABBREVIATIONS ADOPTED FOR MANUSCRIPT

Throughout this manuscript the *vic*-dioximes and their complexes will generally be referred to by abbreviation. Structural formulas, nomenclature, and abbreviations for the *vic*-dioximes have been tabulated in Table 1.

Table 1. Structures, nomenclature, and abbreviations for some *vic*-dioximes

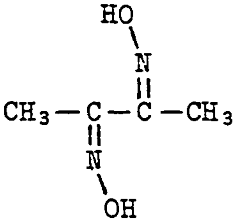
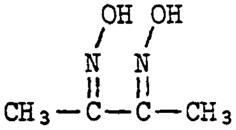
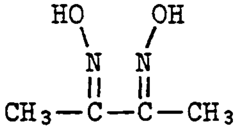
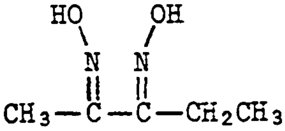
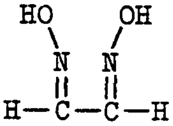
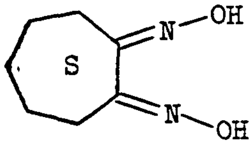
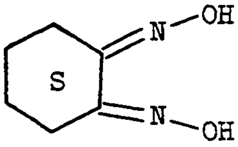
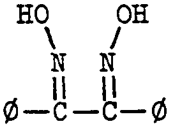
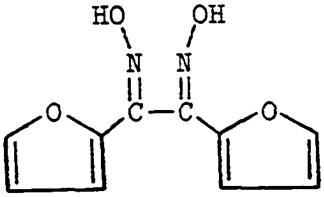
Structure	Systematic name Trivial name Abbreviation
	*syn*-2,3-butanedionedioxime *syn*-dimethylglyoxime *syn*-DMG
	*amphi*-2,3-butanedionedioxime *amphi*-dimethylglyoxime *amphi*-DMG
	*anti*-2,3-butanedionedioxime *anti*-dimethylglyoxime *anti*-DMG or DMG
	2,3-pentanedionedioxime ethylmethylglyoxime EMG

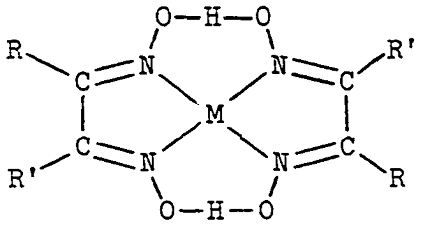
Table 1. (Continued)

Structure	Systematic name Trivial name Abbreviation
 $\mathrm{H-C(=N-OH)-C(=N-OH)-H}$	ethanedionedioxime glyoxime G
	1,2-cycloheptanedionedioxime heptoxime Heptox
	1,2-cyclohexanedionedioxime nioxime Niox
 $\mathrm{\emptyset-C(=N-OH)-C(=N-OH)-\emptyset}$	1,2-diphenylethanedionedioxime α-benzildioxime α-Benzil
	1,2-di(2-furyl)ethanedionedioxime α-furildioxime α-Furil

Examples of the use of the abbreviations given above to describe the composition of any metal(II)-vic-dioxime follow:

1. $Ni(G)_2$ for nickel glyoxime or bis(ethanedionedioximato-N,N') nickel(II).
2. $Cu(DMG)_2$ for copper dimethylglyoxime or bis(2,3-butanedionedioximato-N,N') copper(II).
3. $Pd(\alpha\text{-Benzil})_2$ for palladium α-benzildioxime or bis(1,2-diphenylethanedionedioximato-N,N') palladium(II).
4. -d indicates deuterium substitution of the oxime hydroxyl hydrogen atoms.

It should be pointed out that only the anti- isomer of a vic-dioxime forms the characteristic 2:1 complex with a divalent metal ion. On complex formation, each ligand loses a proton, and the resulting complexes are neutral in charge. The following structure is a generalized structural formula for a metal(II)-vic-dioxime complex:



The substituents R and R' may or may not be the same, depending on the vic-dioxime, and they may also be part of the cyclic ring in one of the alicyclic vic-dioximes. The substituents may be oriented so as to give either a cis- or trans- configuration to the complex; a trans- configuration has been shown.

All other abbreviations used in this manuscript will be defined in the text or will conform to the abbreviations set forth in the American

Chemical Society publication "Handbook for Authors of Papers in the Research Journals of the American Chemical Society".

III. PURPOSE OF RESEARCH

The purpose of this research was to elucidate as completely as possible the nature of the hydrogen bonding in the metal(II)-vic-dioximes. The hydrogen bonds in the vic-dioxime complexes of nickel(II) play an important role in stabilizing the complexes[1], thereby decreasing the extent of dissociation of the complexes in solution. Because the metal(II)-vic-dioximes are basically organic-type solutes (69) and as molecular species are quite insoluble in water, the formation of strong hydrogen bonds has the effect of decreasing the solubilities of these complexes relative to the solubilities they would have were no such bonds formed. The strengths of the hydrogen bonds in copper(II)- and palladium(II)-vic-dioximes have not been conclusively established. An understanding of the nature of the hydrogen bonds in these complexes could provide valuable information on the extent to which hydrogen bonds stabilize the copper(II)- and palladium(II)- complexes, and aid in the future design of specific reagents. If the complexes of copper(II) and palladium(II), like those of nickel(II), are significantly stabilized by hydrogen bonding, then reagents which sterically permit, or even favor, the formation of short, strong intramolecular hydrogen bonds on complexation ought, in principle, to form very stable complexes.

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[1] The lack of reactivity of the hydroxyl hydrogen atoms in the nickel complexes is well documented, confirming the fact that the hydrogen bonds are strong. The formation of strong hydrogen bonds must necessarily stabilize the complexes.

Hydrogen bonds also play major roles in a great many chemical systems, and particularly in biochemical systems. Further knowledge on the nature of the hydrogen bonding in the metal(II)-*vic*-dioximes would undoubtedly increase the understanding of this phenomenon in general, and should therefore be of more general interest.

IV. LITERATURE REVIEW

Although the hydrogen bond is undoubtedly the most thoroughly studied of all chemical interactions, relatively little is known about the true nature of this interaction. No attempt will be made to summarize all of the existing literature on hydrogen bonding as there are several thorough reviews available to the interested reader. A short summary of the literature directly pertinent to this study follows.

A. The Hydrogen Bond

The large majority of the literature on hydrogen bonding can be separated into three general categories, 1) theoretical studies, 2) spectroscopic studies, and 3) structural studies, with a considerable amount of cross correlation between categories.

1. Theory of hydrogen bonding

Since the early, and inadequate, electrostatic model of hydrogen bonding, there have been several different theoretical approaches to the study of the hydrogen bond, including the use of semi-empirical or empirical potential functions, and molecular orbital calculations. These studies have been extensively reviewed by Hadzi (38), Pimentel and McClellan (61), Bratoz (13), Hamilton and Ibers (41), and Murthy and Rao (53).

Perhaps of greatest utility have been the treatments of hydrogen bonding utilizing potential functions. Of these, the model of Lippincott and Schroeder (45) based on a potential function for diatomic molecules has been the most widely used. This model, for linear OHO hydrogen bonds, predicts that as the O---O distance decreases, the OH stretching frequency will decrease because the O-H bond is getting weaker as the H•••O hydrogen bond becomes stronger. This will occur up to the point where the bond becomes symmetrical, after which the frequency will increase as the O-H bond becomes stronger. The point of minimum frequency was calculated by Lippincott and Schroeder to occur in the region of O---O distances from 2.45-2.50 Å. This means that a hydrogen bond 2.53 Å in length might have nearly the same stretching frequency as one 2.45 Å in length. The Lippincott-Schroeder potential for OHO hydrogen bonds allows the successful prediction of the dependence of O-H distance, OH stretching frequency shift, and hydrogen bond energy on O---O distance.

Reid (65) used a slightly modified Lippincott-Schroeder potential to interpret the changes in infrared spectra and in lattice dimensions in hydrogen bonded crystals on deuteration. Reid concluded that the type of potential function proposed by Lippincott and Schroeder "provides an exceptionally good description of numerous hydroxyl compounds which form hydrogen bonds".

Rundle (68) used two Lippincott-Schroeder potentials to describe the hydrogen bonding in short, strong hydrogen bonds and to explain the effects encountered in such bonds. Briefly, it had been found that for many short hydrogen bonds, deuterium substitution caused an elongation of the hydrogen bond. This increase sometimes amounts to 0.06 Å or more.

Rundle described the OHO hydrogen bond as being represented by the potential wells of two normal O-H bonds being forced closer and closer together as the O---O distance decreases. Rundle pointed out that the zero point energy for deuterium is lower than that of hydrogen. As the potential wells are brought together, the barrier between the two wells becomes very small. At the same time, the number of vibrational levels below the barrier will decrease, and the separation of symmetric and asymmetric levels will increase. At sufficiently short O---O distances there will be at most one symmetric (and perhaps one asymmetric) vibrational level below the barrier. This level will lie deeper in the well for D than for H and as the O---O distance decreases the H will have a greater probability of being located near the center of the bond than the D. As the lowest H level approaches the top of the barrier the difference between the D and H distributions becomes marked. Thus, according to Rundle, the hydrogen density at the center will attract both oxygens and lead to a shortening of the bond, while deuterium will have a lower density in the center of the bond and less effect. Hence, it is in just that region where the difference in hydrogen and deuterium density differs most that the abnormal isotope effect should be largest, with the OHO distance shorter than the ODO distance.

Rundle predicted that this abnormal isotope effect should manifest itself most strongly in the range of O---O distances from 2.49-2.56 Å. Rundle also predicted that more direct physical evidence on the nature of short hydrogen bonds might be obtained by careful X-ray diffraction studies of the compounds involved. One should thus be able to distinguish between single and double well distributions in short hydrogen bonds by measuring the isotope effect upon deuterium substitution.

2. Spectroscopic studies of hydrogen bonding

Spectroscopic studies of hydrogen bonding have largely been restricted to infrared spectral studies of the asymmetrical X-H stretching and X-H bending vibrations in X-H•••Y bonds. Recently, far infrared, Raman, NMR, and electronic spectral studies have shed some light on the nature of the hydrogen bond. These studies have been thoroughly reviewed by Ratajczak and Orville-Thomas (63), Jakobsen, *et al*. (42), and Rao and Murthy (62).

Infrared spectroscopy provides the most straightforward technique for the detection and study of hydrogen bonding. Hydrogen bond formation results in a shift of the asymmetric X-H stretching mode (ν_s) to lower frequencies accompanied by an increase in integrated intensity and half-bandwidth. The shift in frequency ($\Delta\nu_s$) from the "free" X-H stretching frequency (3700 cm^{-1} for O-H bonds) can be correlated with various parameters and properties including X---Y distance and hydrogen bond energy. The bending frequency (ν_b) also shifts, generally to higher frequency, and this shift can also be correlated with various properties.

Hydrogen bond formation also results in additional vibrational modes, the most significant being the asymmetrical stretching (ν_σ) and bending (ν_β) modes of the H•••Y bond. The frequencies of the modes associated with the weaker H•••Y or hydrogen bond have been found to lie in the far infrared region. For a large number of compounds containing OHO hydrogen bonds, ν_σ has been assigned to absorptions found in the region from 100-200 cm^{-1} (42).

Recently several workers have attempted to correlate infrared spectral information on hydrogen and deuterium bonds with structural information in order to arrive at a useful description of the potential for the proton in short, strong hydrogen bonds. Snyder and Ibers (73) studied $HCrO_2$ and $DCrO_2$ and concluded that the 2.49 Å OHO bond is symmetrical whereas the 2.55 Å ODO bond is asymmetrical. Delaplane, _et al_. (26) studied the similar system $HCoO_2$-$DCoO_2$ and reached similar conclusions regarding the 2.50 Å OHO and 2.57 Å ODO bonds in these compounds. In both studies, the potential for the hydrogen atom was concluded to be a symmetric single-minimum well or double-minimum well with a low barrier. The potential for the deuterium atom was concluded to be a symmetric double-minimum well with a relatively high barrier extending to the third or fourth excited vibrational level.

3. _Structural studies of hydrogen bonding_

Structural studies of hydrogen bonding fall into two categories, 1) studies of the effect of deuterium substitution on the hydrogen bond length, and 2) studies which attempt to directly determine the nature of the potential for the hydrogen from structural features. This latter type of study has generally been restricted to investigations of compounds containing short, strong hydrogen bonds, in order to establish the point at which a particular class of hydrogen bond becomes symmetrical and the nature of the potential well at that point. Structural studies of hydrogen bonding in solids, with emphasis on neutron diffraction studies, have been thoroughly reviewed by Hamilton and Ibers (41). Ratajczak and Orville-Thomas (63) have published an extensive correlation of OHO bond lengths with vibrational frequencies.

A majority of the structural studies of hydrogen bonding have dealt with OHO hydrogen bonds. Pauling (57) has predicted that such bonds should become symmetrical at an O---O distance of about 2.32 Å. Experimental evidence in the form of neutron diffraction studies (2, 3, 22, 26, 30, 37, 40, 46, 47, 48, 51, 58, 68, 71, 72, 73, 74, 75) indicates that OHO bonds may become symmetrical at somewhat greater distances (2.40-2.50 Å), depending on the nature of the particular compound involved.

Several recent studies of the effect of deuterium substitution on O---O distance have appeared in the literature. Hamilton and Ibers (40) have found an expansion of 0.06 Å in the 2.49 Å OHO hydrogen bond of $HCrO_2$ on deuterium substitution. Delaplane, et al. (26) have found an expansion of 0.07 Å in the 2.50 Å OHO hydrogen bond of $HCoO_2$ on deuteration. Sabine, et al. (71) determined the structure of α-oxalic acid dihydrate using neutron diffraction techniques and found the crystalline solid to contain three different hydrogen bonds, their lengths being 2.506, 2.864, and 2.881 Å, respectively. Coppens and Sabine (22) determined the crystal structure of the corresponding deuterium substituted acid. All three hydrogen bonds were found to expand on deuteration; the amounts of these expansions were 0.018, 0.015, and 0.025 Å, respectively.

B. Hydrogen Bonding in the Metal(II)-*vic*-Dioximes

The intramolecular hydrogen bonds present in the metal(II)-*vic*-dioximes play a significant rôle in making the *vic*-dioximes the selective gravimetric reagents that they are. These bonds help to stabilize the molecular complexes, increasing the formation constants of the complexes and decreasing their solubilities in water. This latter effect is largely a result of the fact that the metal(II)-*vic*-dioxime complexes are organic-type solutes (69) which, in the absence of dissociation into ionic products, are insoluble in solvents like water. The following review summarizes the knowledge on the hydrogen bonding in the metal(II)-*vic*-dioximes.

1. Early chemistry of the metal(II)-*vic*-dioximes

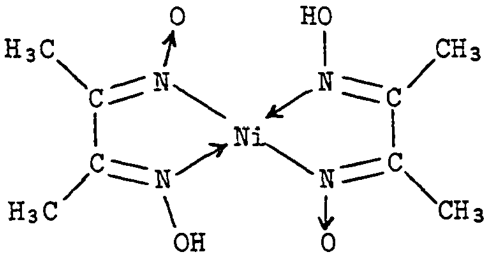
In 1905 a Russian chemist, Tschugaeff (79), discovered that nickel ions and DMG react quantitatively to form a bright red complex which is insoluble in water and many organic solvents. At the time it was known that symmetric *vic*-dioximes exist in three isomeric forms, *syn*(β), *amphi*(γ), and *anti*(α). Tschugaeff (79) was able to show that the α-isomers formed isolable compounds with nickel(II), palladium(II), platinum(II), and copper(II). These compounds had a ligand to metal stoichiometry of 2:1. He was also able to show that the reaction could be described by the equation

$$M^{2+} + 2H_2D = M(HD)_2 + 2H^+$$

where M^{2+} is a divalent metal ion, H_2D is the acid form of a vic-dioxime, and HD^- is the conjugate base. He noted that the nickel, palladium, and platinum complexes sublime in vacuo and are non-electrolytes, and that they are insoluble in water and common organic solvents.

Because of a misunderstanding about which group migrates in the Beckmann rearrangement (the acid catalyzed transformation of a ketoxime to an amide), Werner and Pfeiffer (87) assumed that the α-vic-dioximes had the amphi- configuration. On the basis of this erroneous assumption they proposed a structure for $Ni(DMG)_2$ in which the nickel atom was coordinated to one nitrogen atom and one oxygen atom on each ligand, giving two six-membered rings.

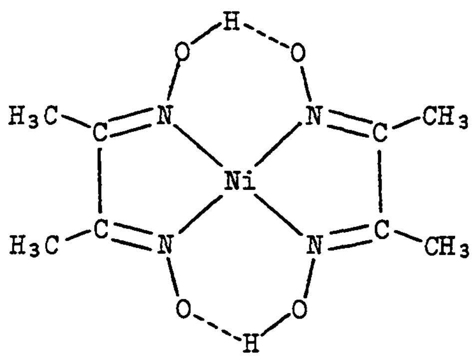
In 1921, Meisenheimer (50) showed that the α-vic-dioximes had the anti- configuration. In 1924, Brady and Mehta (11) presented evidence which indicated that oximes could exist in a zwitterion form as well as in the normal oxime configuration. On the basis of this evidence, Pfeiffer and Richarz (60) proposed the following structure:



Pfeiffer (59) provided additional evidence to support this structure when he found that the mono-O-ethers of the α-vic-dioximes react with nickel in much the same way as do the α-vic-dioximes. Pfeiffer also found that the reaction occurred if one of the oxime groups was replaced by an imino or methylimino group. These observations indicated that the hydroxyl group of the second oxime is not involved in the reaction and that the metal is not linked to the oxygen and, therefore, strongly supported the five-membered ring structure with the metal coordinated to the nitrogen atoms.

The lack of reactivity of the hydroxyl groups in the metal(II)-vic-dioximes proved to be remarkable. Tschugaeff (80) found that $Ni(DMG)_2$ did not react with phenyl isocyanate. Barker (5) found that $Ni(DMG)_2$ did not react with acetic anhydride but that it was methylated by methyl iodide. Thilo and Friedrich (77) found that a suspension of $Ni(DMG)_2$ in ethanol would react with lithium ethoxide or sodium ethoxide but that the addition of water rapidly reversed the reaction. Thilo and Friedrich also reported no reaction with either dimethyl sulfate or methyl iodide. Brady and Muers (12) found that $Ni(DMG)_2$ did not even liberate methane in the presence of methyl magnesium iodide. According to Krause, et al. (44), $Ni(DMG)_2$ does react with acetyl chloride to give, eventually, anhydrous nickel chloride ($NiCl_2$) and the diacylated vic-dioxime [$(CH_3CNOCOCH_3)_2$].

After Brady and Muers (12) found no reaction with methyl magnesium iodide in amyl ether they proposed a structure which attempted to account for the lack of reactivity of the hydroxyl groups. Their structure, a modified version of the structure of Pfeiffer and Richarz (60), contained a pair of intramolecular hydrogen bonds, and is shown below:



This structure, based entirely on chemical evidence, resembles very closely the structure later determined by X-ray diffraction methods.

2. Infrared spectral studies of the metal(II)-*vic*-dioximes

A number of infrared spectral studies of the metal(II)-*vic*-dioximes have been reported in the literature. These studies have been primarily concerned with the hydrogen bonding in these complexes. There were considerable discrepancies among the early studies in the assignment of the OH stretching frequency. Voter, *et al.* (86) published the first infrared spectral study of the hydrogen bonding in the metal(II)-*vic*-dioximes. These workers studied a series of nickel complexes in both the normal and deuterated forms. The OH stretching frequency was assigned to a band in the 1700 cm^{-1} region, and it was concluded that the hydrogen bonds in these complexes were probably symmetrical. Several studies which followed (35, 54, 55, 67, 70) led to similar assignments and conclusions.

In more recent studies, Blinc and Hadzi (8, 9, 10) have assigned the OH stretching frequency for the nickel complexes to a broad, weak band in the 2300 cm^{-1} region. The spectra of some Pd, Pt, and Cu complexes were also studied. Of particular interest was the assignment of two different OH stretching frequencies, at 2340 and 2650 cm^{-1}, for $Cu(DMG)_2$. The copper complex was later found to contain two hydrogen bonds of different lengths.

Blinc and Hadzi (8) made assignments for several other bands in the spectra of metal(II)-vic-dioximes. The assignments were as follows: OH stretch (ν_{OH}), 2300-2900 cm^{-1}; OH in-plane bend (δ_{OH}), 1650-1800 cm^{-1}; OH out-of-plane bend (γ_{OH}), 820-930 cm^{-1}; CN stretch (ν_{CN}), 1500-1600 cm^{-1} and NO stretch (ν_{NO}), about 1240 and 1000 cm^{-1}.

Rundle and Banks (69), in attempting to explain the relative solubilities and enthalpies of solution of $Cu(DMG)_2$, $Ni(DMG)_2$, and $Ni(EMG)_2$, predicted that the hydrogen bonds in $Cu(DMG)_2$ rearrange on dissolution to form much stronger hydrogen bonds such as those in the nickel chelates. This prediction was based on knowledge of the crystal and molecular structures of the three complexes, and the fact that in order for $Cu(DMG)_2$ to have an enthalpy of solution similar to that of $Ni(DMG)_2$ in an inert solvent, some source of energy was required. $Cu(DMG)_2$ was known to crystallize as a dimer held together by a pair of Cu-O bonds. Assuming monomeric specie in solution, energy has to be provided in order to pay for breaking these bonds. The predicted hydrogen bonding rearrangement did this. In order to test this prediction, Caton and Banks (21) undertook an infrared spectral study of the hydrogen bonding in the normal and deuterated forms of $Cu(DMG)_2$, $Ni(DMG)_2$, and $Ni(EMG)_2$. On the basis of the infrared spectra and the reactivities of the complexes with dimethyl sulfate, phenyl isocyanate and acetic anhydride, it was concluded that the hydrogen bonding in $Cu(DMG)_2$ rearranges upon dissolution so that it is quite similar to the hydrogen bonding in the two nickel complexes. In their study, however, Caton and Banks did not consider the evidence provided by the OH in-plane bending frequencies. The frequencies reported by Caton and Banks have been summarized in Table 2.

Table 2. Summary of data from Caton and Banks (21) on OH stretching and bending frequencies for some metal(II)-*vic*-dioximes

Chelate	State[a]	R[b]	ν_{OH}[c]	δ_{OH}[d]
$Cu(DMG)_2$	C	2.70	2650	1492
	C	2.53	2382	1640
	S	—	2375	1640
$Ni(EMG)_2$	C	2.33	2388	1784
	S	—	2350	1715
$Ni(DMG)_2$	C	2.40	2322	1790

[a] C is the solid or crystalline state; S indicates solution.

[b] R is the O---O distance in Å.

[c] ν is the stretching frequency in cm^{-1}.

[d] δ is the bending frequency in cm^{-1}.

Paying particular attention to the OH in-plane bending frequencies, these data suggest that only the longer bond in $Cu(DMG)_2$ rearranges on dissolution, becoming similar to the shorter bond. Caton and Banks felt that the lack of reactivity of $Cu(DMG)_2$ in solution supported a shortening of both bonds, but no reactivity data were available on bonds intermediate in length between the two in $Cu(DMG)_2$.

The most recent infrared study of metal(II)-*vic*-dioximes is that of Bigotto, *et al*. (7), who studied the infrared spectra of $Cu(DMG)_2$, $Ni(DMG)_2$, $Pd(DMG)_2$, and $Pt(DMG)_2$ in the region 4000-200 cm^{-1}. A normal coordinate analysis was also carried out for the complexes as 31-body structures with $\underline{D}_{2h}$ symmetry. Vibrational assignments were made for the in-plane infrared active vibrations in $Cu(DMG)_2$ and $Pd(DMG)_2$. The calculations indicated considerable mixing of modes for many of the observed frequencies. The assignments for frequencies in $Cu(DMG)_2$ and $Pd(DMG)_2$ which are relatively pure are as follows: ν_{OH}, 2300-2600 cm^{-1}; δ_{OH}, 1700-1800 cm^{-1}; ν_{CN}, 1540-1580 cm^{-1}, ~1320 cm^{-1}; ν_{CC}, 1500-1550 cm^{-1}; ν_{NO}, 1210-1260 cm^{-1}, ~1080 cm^{-1}. The calculations for $Cu(DMG)_2$ confirmed the assignments for the OH stretching and bending frequencies made by Blinc and Hadzi and later by Caton and Banks. This agreement is somewhat artificial, however, because of the model used in the normal coordinate treatment. The model assumed symmetrical hydrogen bonds and the calculation adjusted force constants to fit the observed spectrum, with the result that the force constant calculated for the OH stretching mode is not reasonable. In addition, the frequencies associated with the weaker H•••O bond were not calculated because force constants for the OH stretching and bending modes of this bond were not included in the calculation (they were not required because the molecular symmetry which was assumed did not distinguish between the O-H and H•••O bonds). In spite of these shortcomings, the conclusion that the ν_{OH} and δ_{OH} modes are all but free of mixing with other modes should still be valid.

3. Structural studies of the metal(II)-*vic*-dioximes

The crystal and molecular structures of a number of metal(II)-*vic*-dioximes have been determined using X-ray diffraction methods. These studies have, for the most part, depended on photographic methods of data collection. Many of the studies have relied on two-dimensional data. Such studies do not provide sufficiently precise information to allow conclusions to be reached regarding the nature of the hydrogen bonding in complexes whose structures are so determined.

The structure of $Ni(DMG)_2$ was first determined by Godycki and Rundle (36) and later refined by Williams, *et al*. (90). $Ni(DMG)_2$ was found to crystallize in the orthorhombic space group *Ibam*, with four molecules in the unit cell. The structural determination was based on intensities estimated from three-dimensional film data. Within the limits of experimental error the molecule was found to have symmetry D_{2h}. The final value of the conventional discrepancy factor was

$$R = \Sigma||F_o| - |F_c||/\Sigma|F_o| = 0.124,$$

where F_o and F_c are the observed and calculated structure amplitudes, respectively. The molecules were found to pack in parallel layers 3.25 Å apart with molecules in adjacent layers rotated 90° to accommodate the relatively bulky methyl groups. The metal atom was found, as expected, to be coordinated by four nitrogen atoms. An extremely short intramolecular hydrogen bond was found, the O---O distance being 2.40 ± 0.02 Å. The packing configuration, which resulted in chains of nickel atoms, led to the prediction of Ni-Ni bonding (36) in the solid.

The structure of $Cu(DMG)_2$ was first determined by Frasson, et al. (32). $Cu(DMG)_2$ was found to crystallize in the monoclinic space group $P2_1/n$ with four molecules in the unit cell. The structure was solved at liquid nitrogen temperature using photometrically integrated intensities taken from two-dimensional film data. The structure was solved in projection using difference maps. The final value of R was 0.10 for the (001) projection and 0.12 for the (100) projection. $Cu(DMG)_2$ was found to crystallize as dimers joined by a pair of copper-oxygen bonds. One of the more interesting features of the solid complex was the existence of two non-equivalent hydrogen bonds, a consequence of the involvement of one oxygen atom per molecule in dimer formation. The shorter of the hydrogen bonds was found to be 2.53 Å long and does not involve the oxygen atom which participates in dimer formation. The longer hydrogen bond was found to be 2.70 Å long. As a result of the increase in the coordination number of copper from four to five on dimer formation, the coordination is not planar. Rather, it is more correctly described as distorted square pyramidal.

The structure of $Cu(DMG)_2$ was very recently redetermined at room temperature by Vaciago and Zambonelli (81). Intensities were estimated visually from three-dimensional film data. Two crystals were used in the data collection. The structure was refined with anisotropic thermal parameters for the non-hydrogen atoms to a value for R of 0.063. The hydrogen bonds were found to be 2.526 and 2.694 Å long. The hydrogen atom in the longer bond was found to be located at a distance of 1.04 Å from the oxygen atom not involved in dimer formation. The longer hydrogen bond was found to be non-linear, the OHO angle being 167.5°. The NOH angle was found to be 101.0°. Although they were unable to locate the hydrogen atom in the other hydrogen bond, Vaciago and Zambonelli stated, indirectly, that the N-O distances indicated it was bound more strongly to one oxygen atom than to the other, suggesting that the shorter OHO bond is asymmetrical.

The crystal structure of $Pd(DMG)_2$ was first reported by Williams, et al. (90). $Pd(DMG)_2$ was found to crystallize, like $Ni(DMG)_2$, in the orthorhombic space group Ibam, with four molecules in the unit cell. The intensities were taken with a proportional counter. Three-dimensional data were collected and the structure was refined to a value for R of 0.065. The structure was found to be nearly isostructural with that of $Ni(DMG)_2$ but the $Pd(DMG)_2$ molecule appeared to be less symmetrical than that of $Ni(DMG)_2$. The hydrogen bond in $Pd(DMG)_2$ was found to be much longer than in $Ni(DMG)_2$, the O---O distance being 2.59 Å. Williams, et al. also found the hydrogen bond in

$Pd(DMG)_2$ to be asymmetrical, their conclusion being based on the non-equivalence of N-O and Pd-N distances.

The structure of $Pd(DMG)_2$ was also determined by Panattoni, et al. (56). The structure, based on intensities photometrically integrated from two-dimensional film data, was not significantly different from that of Williams, et al. The value for the O---O distance, 2.62 Å, was slightly longer than that of Williams, et al., but the difference was not significant (25).

Frasson, et al. (34) reported the structure of $Pt(DMG)_2$. This complex was found to crystallize in the orthorhombic space group Ibam also, with four molecules in the unit cell. Two-dimensional intensity data were collected by photometric integration of reflections recorded on a Weissenberg camera equipped with an attachment for linear integration. The structure was refined from difference maps, with anisotropic thermal motion included for Pt, to a final value for R of 0.07. The structure was found to be isostructural with those of $Ni(DMG)_2$ and $Pd(DMG)_2$. The O---O distance was found to be 3.03 Å. The N-O distances were not equivalent, suggesting an asymmetrical hydrogen bond.

The structure of $Ni(EMG)_2$ was reported by Frasson and Panattoni (33). $Ni(EMG)_2$ crystallizes in the monoclinic space group $P2_1/c$, with two molecules in the unit cell. Two-dimensional intensity data were collected by photometric integration of films. The atomic coordinates were refined with difference synthesis in two projections to final values for R in both projections of 0.11. The molecule was found to have a transconfiguration. The packing precludes the possibility of Ni-Ni bonding, the Ni atom in one molecule approximately overlying an oxygen atom of the molecule in the next layer. The ethyl group was found not to lie in the same plane as the remainder of the molecule. The Ni-N, N-O, and C-N distances did not appear to be equivalent but the large standard deviations in these distances resulted in a lack of significance in any of the differences. The O---O distance in this complex was found to be 2.33 Å, considerably shorter than any previously studied OHO hydrogen bond.

The structure of $Ni(G)_2$ was first reported by Calleri, et al. (18). $Ni(G)_2$ was found to crystallize in the monoclinic space group $P2_1/c$, with two molecules in the unit cell. The structural determination was based on three-dimensional counter data. The structure was refined with anisotropic thermal parameters for the heavy atoms by full matrix least-squares techniques to a final value for R of 0.039. The molecule was found to have C_2 symmetry, the C-N bond distances being non-equivalent. The lower symmetry was ascribed to intermolecular contacts. The N-O distances were found to be equivalent and this fact, coupled with an O---O distance of 2.453 Å led Calleri, et al. to propose the existence of a symmetrical hydrogen bond in $Ni(G)_2$.

Following the study by Calleri, et al., Murmann and Schlemper (52) published a determination based on visually estimated intensities taken from film data. The agreement between these structural studies was remarkably good.

The structure of $Pd(G)_2$ was reported by Calleri, *et al.* (19). $Pd(G)_2$ was found to crystallize in the triclinic space group *P1*, with two molecules in the unit cell. The structural determination was based on three-dimensional counter data. The structure was refined with anisotropic thermal parameters for the non-hydrogen atoms by full matrix least-squares techniques to a final value for *R* of 0.046. The two molecules in the unit cell are not crystallographically equivalent. The molecules were found to have symmetry C_{2h}. A significant difference was found in the lengths of the two hydrogen bonds, the O---O distances being 2.599 and 2.659 Å. The N-O distances are consistent with asymmetric hydrogen bonds in both molecules.

The structure of $Pt(G)_2$ was reported by Ferraris and Viterbo (31). $Pt(G)_2$ crystallizes in the monoclinic space group $P2_1/n$, with two molecules in the unit cell. The structural determination was based on three-dimensional counter data. The structure was refined with anisotropic thermal parameters by full matrix least-squares techniques to a final value for *R* of 0.049. The molecule was found to be planar with apparent symmetry C_{2h}. The N-O distances were not significantly different but the O---O distance of 2.655 Å led Ferraris and Viterbo to postulate an asymmetrical hydrogen bond.

Comparison of the well-determined structures of metal(II)-*vic*-dioximes [$Ni(G)_2$, $Pd(G)_2$, $Pt(G)_2$, and $Cu(DMG)_2$] reveals some interesting and consistent features. For these complexes there appears to be a significant delocalization of the C-N double bonds through the C-C bond of the chelate ring (18, 19, 31). Vaciago and Zambonelli (81) stated that the shortening of the C-C bond is the result of a smaller single-bond covalent radius for _sp_^{2} hybrids (27). In calculating the _sp_^{2} single-bond radius, however, Dewar and Schmeising (27) have used diffraction studies of molecules in which a significant degree of conjugation can occur. Vaciago and Zambonelli also concluded that the lengthening of the C-N bonds and the shift in ν_{CN} from 1645 cm^{-1} in DMG to 1565 cm^{-1} in $Cu(DMG)_2$ were not adequate evidence of delocalization over the five-membered rings. This option, however, seems to be a minority one.

V. X-RAY DIFFRACTION STUDIES

In order to provide definitive information on the nature of the hydrogen bonds in some metal(II)-vic-dioximes, precise structural studies of these complexes were undertaken using X-ray diffraction techniques.

A. Crystal and Molecular Structure of $Cu(DMG)_2$-d_2

Caton and Banks (21) concluded that both of the hydrogen bonds in deuterated $Cu(DMG)_2$ are asymmetric. The structure of $Cu(DMG)_2$-d_2 was determined in order to test this conclusion and to provide additional structural information on short hydrogen bonds.

1. Apparatus and materials

Much of the apparatus used in this investigation consisted of items found in most adequately equipped laboratories. No mention will be made of such equipment in this manuscript. The more sophisticated equipment will be referred to by make and model only.

Most of the chemicals used were from the laboratory stock of reagent-grade quality chemicals. Only in cases where chemicals were not obtained in reagent-grade quality or where they were treated in some manner before use will specific details be given.

a. Instrumentation and apparatus Film data were collected on a Charles Supper Company precession camera. Intensity data were collected on a General Electric SPG-2 diffractometer equipped with a General Electric quarter-circle orienter in conjunction with a General Electric

XRD-6 source unit and a General Electric SPG-4 detection unit equipped with a scintillation counter.

Infrared spectra in the 4000 to 600 cm^{-1} range were obtained on a Beckman IR-7 spectrophotometer.

Many of the computations were performed on an IBM System 360 Model 65 computer.

b. Chemicals Dimethylglyoxime was a 'Baker Analyzed' reagent. It was recrystallized twice from 1:1 ethanol-water solution.

2. Experimental procedures

a. Preparation of $Cu(DMG)_2$-d_2 $Cu(DMG)_2$-d_2 was prepared in a glove box under a dry nitrogen atmosphere as follows. The hydroxyl hydrogen atoms of DMG were replaced via two recrystallizations from hot D_2O. Stoichiometric amounts of DMG-d_2 and $CuCl_2$ in D_2O solution were then mixed and a stoichiometric amount of Na_2CO_3 was added. The resulting dark brown solution was allowed to evaporate to near-dryness producing a dark brown powdery solid which was then redissolved in a fresh portion of D_2O. Upon standing several needle-like crystals were obtained. A halocarbon oil mull infrared spectrum of this compound indicated complete replacement of the oxime hydroxyl hydrogen atoms by deuterium. Crystals suitable for X-ray examination were sealed in thin-walled Lindemann glass capillaries using glove-box techniques, to prevent deuterium-hydrogen exchange with atmospheric water. Microscopic examination of the crystal selected for X-ray study revealed it to be needle-like with approximate dimensions of 0.05 x 0.07 x 0.54 mm. The deuterated and undeuterated compounds were found to be similar in appearance.

b. Collection of data Preliminary precession photographs exhibited 2/m Laue symmetry indicating a monoclinic space group. Systematic absences of ($h0\ell$) for $h + \ell$ odd and of ($0k0$) for k odd indicated the space group C_{2h}^5-$P2_1/n$. The same space group has been reported (32) for Cu(DMG)$_2$. The unit cell parameters, calculated from zero, first, and second layer precession photographs corrected for film shrinkage are:

$$a = 9.82 \pm 0.01\ \text{Å},$$
$$b = 17.15 \pm 0.02\ \text{Å},$$
$$c = 7.16 \pm 0.01\ \text{Å};$$
$$\beta = 106.70 \pm 0.12°.$$

There are no significant differences between these parameters and those reported for Cu(DMG)$_2$ at room temperature (32). Other crystal data are: M = 295.79 g/mole, V = 1155 Å^3, Z = 4, D_c = 1.70 g/cm^3, $F(000)$ = 604 e$^-$.

Intensity data were collected at room temperature. The crystal was mounted with c along the spindle axis. Integrated intensities were collected by the moving-crystal moving-counter (θ-2θ) technique using Ni-filtered Cu K_α radiation (λ = 1.5418 Å) with an 83.3 second scan, centered about each peak, at a scanning speed of 2° per minute. Stationary-crystal stationary-counter background counts of a duration equal to one-half the total scan time were taken at the beginning and end of each scan. Within a 2θ sphere of 40° all accessible data in two unequivalent octants were recorded. Duplicate measurements of some of the more intense reflections were made in two additional octants.

A total of 1048 reflections were measured. The intensity data were corrected for Lorentz-polarization effects, and for absorption. The absorption coefficient, μ, is 28.51 cm^{-1}, and an absorption correction (15) was made using ABCOR. The maximum and minimum transmission factors were 0.897 and 0.674, respectively. The estimated error in each intensity was calculated using the formula $[\sigma(I)]^2 = [C_t + C_b + (0.05\ C_t)^2 + (0.05\ C_b)^2]$ (89) where C_t and C_b are the total count and background count, respectively. The equivalent values of $\underline{F}_{\underline{o}}^{\,2}$ were then averaged to give 916 independent $\underline{F}_{\underline{o}}^{\,2}$ values. The estimated standard deviation in each structure factor was calculated from the mean deviation of intensity by the method of finite differences (89). The reciprocals of the structure factor variances were used as weights in the least-squares refinement.

3. Solution and refinement of the structure

The atomic coordinates for the non-hydrogen atoms in $Cu(DMG)_2$ (32) were used as a starting point. This model was refined by full matrix least-squares techniques [using a local modification of ORFLS (16)] with isotopic thermal parameters to a conventional discrepancy factor of $\underline{R} = \Sigma||\underline{F}_{\underline{o}}|-|\underline{F}_{\underline{c}}||/\Sigma|\underline{F}_{\underline{o}}| = 0.095$. The relativistic Dirac-Slater X-ray scattering factors for neutral atoms as reported by Cromer and Waber (24) were used, with those of copper (23) and oxygen (76) modified for the real and imaginary parts of anomalous dispersion. Only 625 reflections which could be considered observed ($\underline{F}_{\underline{o}}^{\,2} \geq \sigma_{\underline{F}_{\underline{o}}^{\,2}}$) were used at this stage of the refinement. Two cycles of refinement with anisotropic thermal parameters for copper gave $\underline{R} = 0.090$. Anisotropic thermal paramaters were inserted in the refinement for nitrogen, oxygen, and carbon atoms, in that order, with two cycles at each stage. The corresponding values for $\underline{R}$ were 0.088, 0.079, and 0.078, respectively.

An electron density calculation revealed some diffuse regions of positive density which were attributed to the hydrogen atoms. It was not possible, however, to locate these atoms with any precision, and their positions were calculated assuming tetrahedral methyl groups. The orientation of the methyl group was adjusted to place the hydrogen atoms as close as possible to the positive regions in the map. The C-H bond distance was taken to be 0.92 Å (99, 78). The hydrogen atoms were assigned isotropic thermal parameters similar to those of the atoms to which they are bound. Hydrogen atom parameters were not varied, but the positional parameters were frequently recalculated to allow for shifts in the positions of the carbon atoms. Deuterium atoms were placed midway between the oxygen atoms and assigned isotropic thermal parameters similar to those of the oxygen atoms. Deuterium parameters also were not varied but positional parameters were recalculated from time to time.

Inclusion of the hydrogen and deuterium atoms with two cycles of refinement gave a value for $\underline{R}$ of 0.071.

At this point the weights used in the refinement were adjusted by the following procedure. The reflection data were sorted on $\underline{F}_o$. The sum $\Sigma\omega\Delta^2$, where ω is the weight used in the refinement and Δ is $||\underline{F}_o|-|\underline{F}_c||$, was calculated and divided by a number of reflections, giving a result we will arbitrarily call Q. The reflection data were then placed into 25 to 30 overlapping sets of equal size so that each reflection was a member of two sets, and the sum $\Sigma\omega\Delta^2$ was calculated for each set. These sums were then divided by a number of reflections in the set and by Q to give a correction factor for each set. The weight, ω, of each reflection was then divided by the average of the correction factors for the two sets in which it resided. Repetition of this procedure, varying the number of sets used, yielded a set of weights such that $\Sigma\omega\Delta^2$/No. of reflections = 1.0 and furthermore such that a plot of $\overline{\omega\Delta^2}$ vs. $\underline{F_o}$ gave approximately a straight line with $\overline{\omega\Delta^2} \approx 1.0$ over the entire range of values of $\underline{F_o}$.

Using the adjusted weights, two cycles of refinement gave a value for $\underline{R}$ of 0.063. Recalculation of hydrogen positions followed by two further cycles gave an $\underline{R}$ of 0.062. An electron density difference map calculated at this point revealed no peaks greater than $0.5e^-/\text{Å}^3$.

A study was next made of the effect of exclusion of hydrogen or deuterium atoms, or both, on the refined structure. Exclusion of both resulted in a structure in which the O---O distances were ~0.04Å shorter than when these atoms were included. Exclusion of hydrogen atoms resulted in a structure with slightly longer O---O distances as well as less reasonable values for other bond distances. The placement of the deuterium atoms was not critical. As long as their positions did not vary unreasonably far from the center of the O-H-O bond the positional and thermal parameters of other atoms were not significantly affected.

The final cycles of refinement were carried out using all reflections except those with $\underline{F_o} = 0$. This gave a set of 834 independent reflections. Weight adjustment followed by three cycles of refinement gave a value for R of 0.084 and a weighted R-factor of

$$\omega\underline{R} = [\Sigma\omega(|\underline{F_o}|-|\underline{F_c}|)^2/\Sigma\omega|\underline{F_o}|^2]^{1/2} = 0.046.$$

An electron density difference map was found to contain no peaks larger than 0.6e⁻/Å³. The final refined positional and anisotropic thermal parameters, along with their estimated standard deviations as derived from the inverse matrix of the final least-squares refinement cycle are found in Appendix A. A listing of the observed and calculated structure amplitudes for the 834 reflections used in the final cycles is found in Appendix B.

4. Description of the structure

Interatomic distances and angles with standard deviations are given in Tables 3 and 4, respectively. Distances and angles are also illustrated in Figure 1. The standard deviations in the distances and angles were calculated using the variance-covariance matrix and ORFFE (17), and include errors in the lattice constants. Bond distances corrected for thermal motion assuming a riding model are also given in Table 3.

The O---O distances are 2.569 and 2.716 Å. Statistical tests (25) indicate that the differences between the N(4)-O(3) bond and the other N-O bonds are highly significant, and suggest that oxygen atoms O(1) and O(3) do not participate equally in the formation of the hydrogen bond between them.

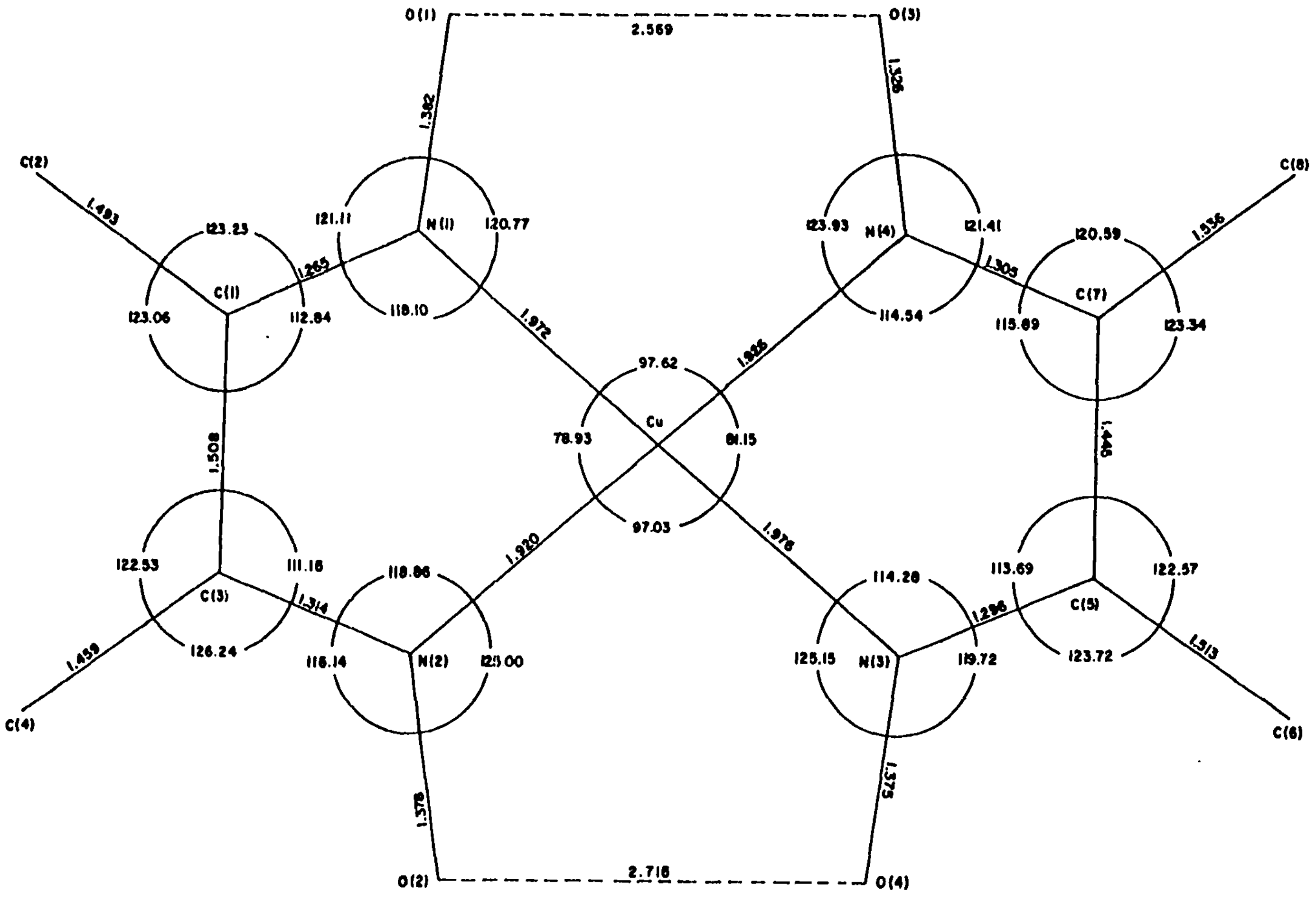


Figure 1. Interatomic distances and angles in $Cu(DMG)_2-d_2$

Table 3. Interatomic distances in $Cu(DMG)_2$-d_2

	Without thermal motion, Å	With thermal motion[a], Å
Cu-N(1)	1.972 (9)[b]	1.980 (9)
Cu-N(2)	1.920 (11)	1.927 (11)
Cu-N(3)	1.976 (9)	1.985 (9)
Cu-N(4)	1.926 (11)	1.935 (11)
N(1)-O(1)	1.382 (10)	1.400 (11)
N(2)-O(2)	1.378 (10)	1.383 (11)
N(3)-O(4)	1.375 (11)	1.401 (11)
N(4)-O(3)	1.326 (10)	1.336 (12)
N(1)-C(1)	1.265 (12)	1.285 (15)
C(3)-N(2)	1.314 (12)	1.331 (14)
N(3)-C(5)	1.296 (12)	1.299 (14)
N(4)-C(7)	1.305 (12)	1.324 (15)
C(1)-C(2)	1.493 (15)	1.522 (16)
C(3)-C(4)	1.459 (13)	1.465 (15)
C(5)-C(6)	1.513 (14)	1.539 (16)
C(7)-C(8)	1.536 (15)	1.556 (17)
C(1)-C(3)	1.508 (15)	
C(5)-C(7)	1.445 (14)	
O(1)-O(3)	2.569 (11)	
O(2)-O(4)	2.716 (11)	
Cu-O(2')	2.303 (7)	2.316 (8)

[a] Assuming a riding model with the second atom given riding on the first.

[b] Numbers in parentheses here and in succeeding tables are the estimated standard deviations in the least significant digits.

Table 4. Interatomic angles (°) in $Cu(DMG)_2$-d_2

N(1)-Cu-N(3)	158.1 (3)	N(2)-Cu-N(4)	166.2 (3)
N(1)-Cu-N(2)	78.9 (5)	N(1)-Cu-N(4)	97.6 (5)
N(3)-Cu-N(4)	81.2 (5)	N(2)-Cu-N(3)	97.0 (5)
Cu-N(1)-O(1)	120.8 (9)	Cu-N(3)-O(4)	125.2 (8)
Cu-N(2)-O(2)	125.0 (7)	Cu-N(4)-O(3)	123.9 (9)
Cu-N(1)-C(1)	118.1 (9)	Cu-N(3)-C(5)	114.3 (10)
Cu-N(2)-C(3)	118.9 (8)	Cu-N(4)-C(7)	114.5 (9)

Table 4. (Continued)

C(1)-N(1)-O(1)	121.1 (11)	C(5)-N(3)-O(4)	119.7 (10)
C(3)-N(2)-O(2)	116.1 (11)	C(7)-N(4)-O(3)	121.4 (11)
N(1)-C(1)-C(2)	123.2 (13)	N(3)-C(5)-C(6)	123.7 (12)
N(2)-C(3)-C(4)	126.2 (11)	N(4)-C(7)-C(8)	120.6 (11)
N(1)-C(1)-C(3)	112.8 (12)	N(3)-C(5)-C(7)	113.7 (12)
N(2)-C(3)-C(1)	111.2 (11)	N(4)-C(7)-C(5)	115.9 (12)
C(2)-C(1)-C(3)	123.1 (13)	C(6)-C(5)-C(7)	122.6 (13)
C(4)-C(3)-C(1)	122.5 (12)	C(8)-C(7)-C(5)	123.3 (13)

The two ligands coordinated to copper are not coplanar. Figure 2 illustrates the configuration of the two ligands. This illustration, and many others in this manuscript, were computer drawn using ORTEP (43). Calculation of the best mean planes through the chelate rings reveals a lack of planarity for both rings. The equations of the least-squares planes and the deviations of the atoms from them are presented in Table 5.

Table 5. Equations of the least-squares planes through the rings in $Cu(DMG)_2$-d_2 and deviations of the atoms from them

Plane I[a]

$$2.283x - 4.276y + 5.969z - 1.804 = 0.0$$

	d(Å)	t_o	
Cu	-0.0002	0.20	
N(1)	0.0189	2.10	
N(2)	0.0174	1.94	$\chi^2 = 13.2$
C(1)	-0.0239	1.84	
C(3)	-0.0138	1.26	

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[a] Equations are in fractional coordinates, for the molecule nearest the origin, referred to the non-orthogonal crystallographic axes.

Table 5. (Continued)

Plane II

$$-1.019x - 0.423y + 7.032z - 2.500 = 0.0$$

	d(Å)	t_o	
Cu	0.0003	0.31	
N(3)	-0.0401	4.46	
N(4)	-0.0230	2.56	$\chi^2 = 49.6$
C(5)	0.0574	4.78	
C(7)	0.0046	0.46	

Significant differences (25) in bond length occur between adjacent Cu-N bonds but not between non-adjacent Cu-N bonds. The differences between the C(1)-N(1) bond and the other C-N bonds are also significant, possibly the result of distortions caused by dimerization. The configuration of the molecule, especially with regard to the coordination about copper, is distorted upon dimer formation as illustrated in Figure 2. The difference between the bond angles N(1)-Cu-N(3) (158°) and N(2)-Cu-N(4) (166°) is highly significant, indicating that the chelate rings are twisted with respect to one another.

A comparison of the bond distances in $Cu(DMG)_2-d_2$ with those found in similar metal(II)-*vic*-dioxime compounds suggests that all of the values are reasonable. The carbon-carbon bonds to the four methyl carbon atoms are somewhat short, but a correction for thermal motion assuming a riding model gives quite reasonable values (the average C-C distance is 1.52 Å) considering the estimated errors in these distances.

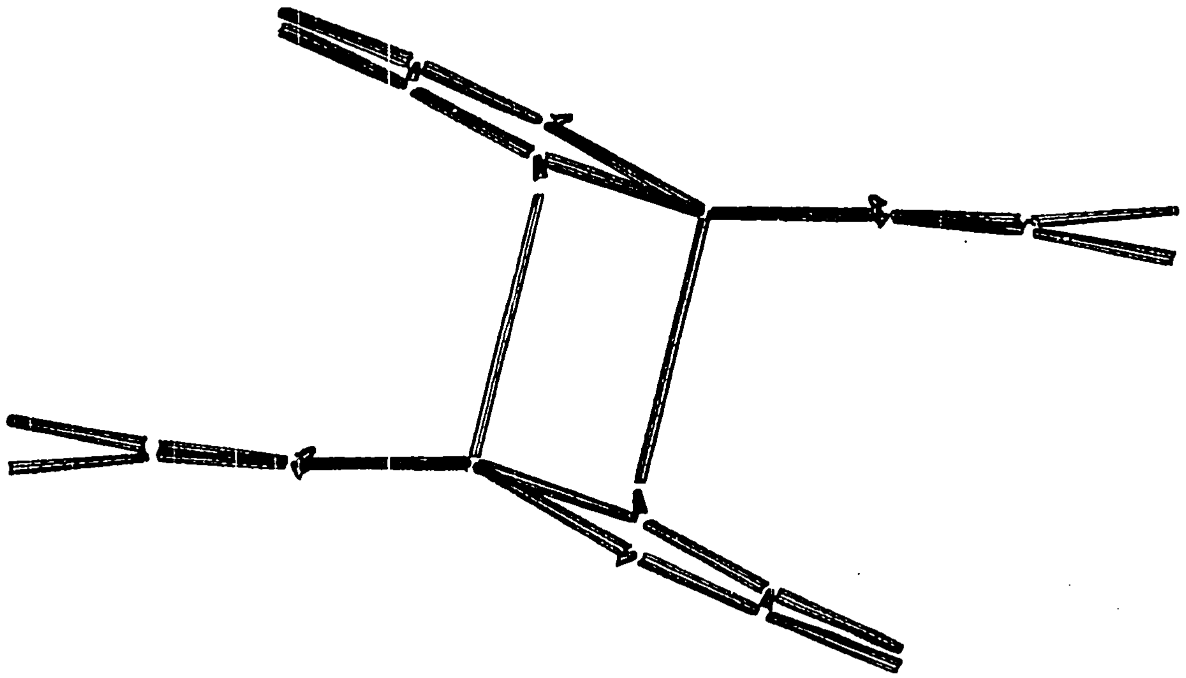

Figure 2. Stick bond illustration of a dimer of $Cu(DMG)_2$-d_2

The nature of the thermal motion in this molecule is illustrated in Figure 3. The ellipsoides are scaled to enclose 50% probability. The thermal motion of all atoms is reasonable.

Figures 4 and 5 illustrate the packing of the dimers projected along the a and c directions, respectively.

B. Crystal and Molecular Structure of $Cu(DMG)_2$

At the time the determination of the structure of $Cu(DMG)_2$-d_2 was completed, the only structural study of $Cu(DMG)_2$ in the literature was that of Frasson, et al. (32). This structural determination was not sufficiently precise to allow meaningful comparisons of the structures of $Cu(DMG)_2$ and $Cu(DMG)_2$-d_2 to be made. The structure of $Cu(DMG)_2$ was therefore redetermined. There has since appeared in the literature another redetermination (81). Comparisons of the two redeterminations and of the structures of $Cu(DMG)_2$ and its deuterated analog will be made later in this manuscript.

1. Apparatus and materials

a. Instrumentation and apparatus Film data were recorded with a Nonius precession camera. Intensity data were collected with a fully automated Hilger-Watts four circle diffractometer equipped with a scintillation counter and interfaced to an SDS-910 computer in a real-time mode. Computations were performed on an IBM 360/65 computer.

b. Chemicals Copper dimethylglyoxime had been prepared by J. E. Caton (20) of this laboratory using the method of Basu, Cook, and

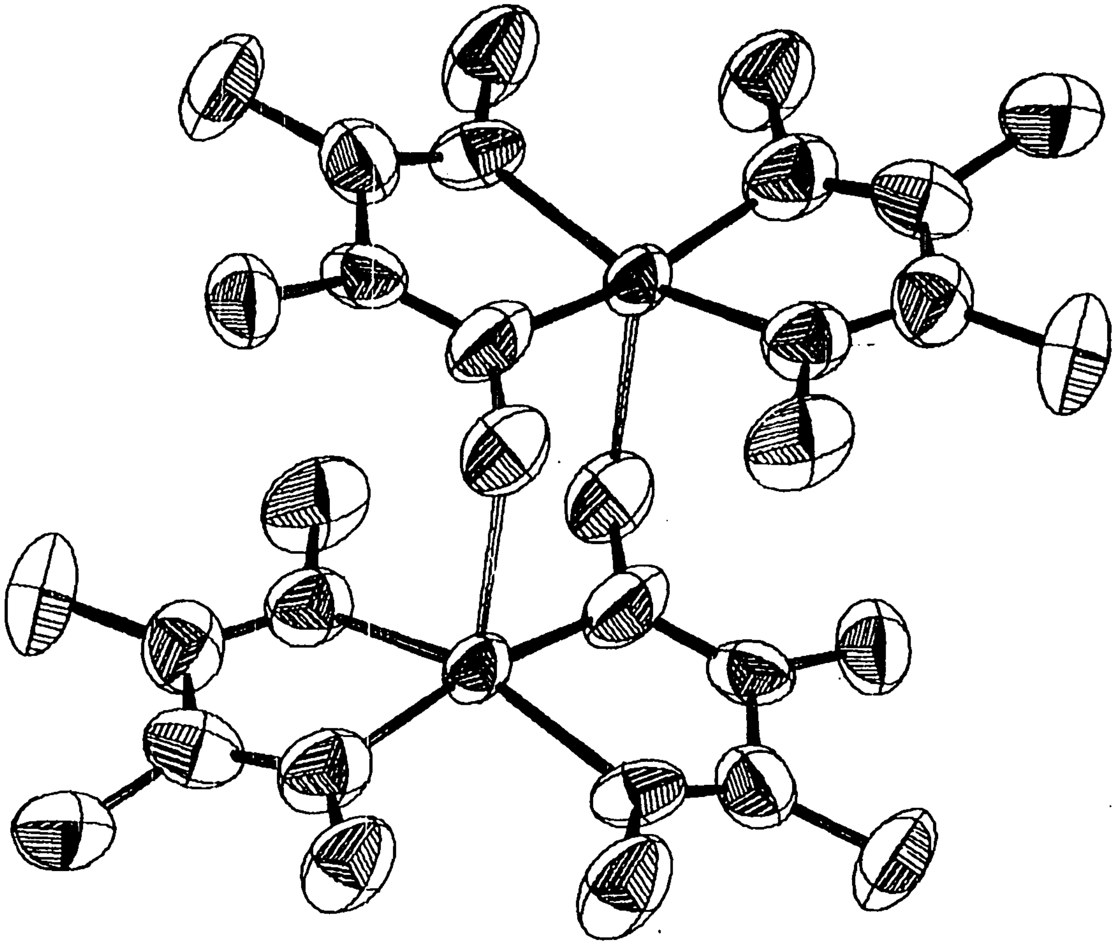

Figure 3. Illustration of the thermal motion in $Cu(DMG)_2$-d_2 with ellipsoids scaled to enclose 50% probability.

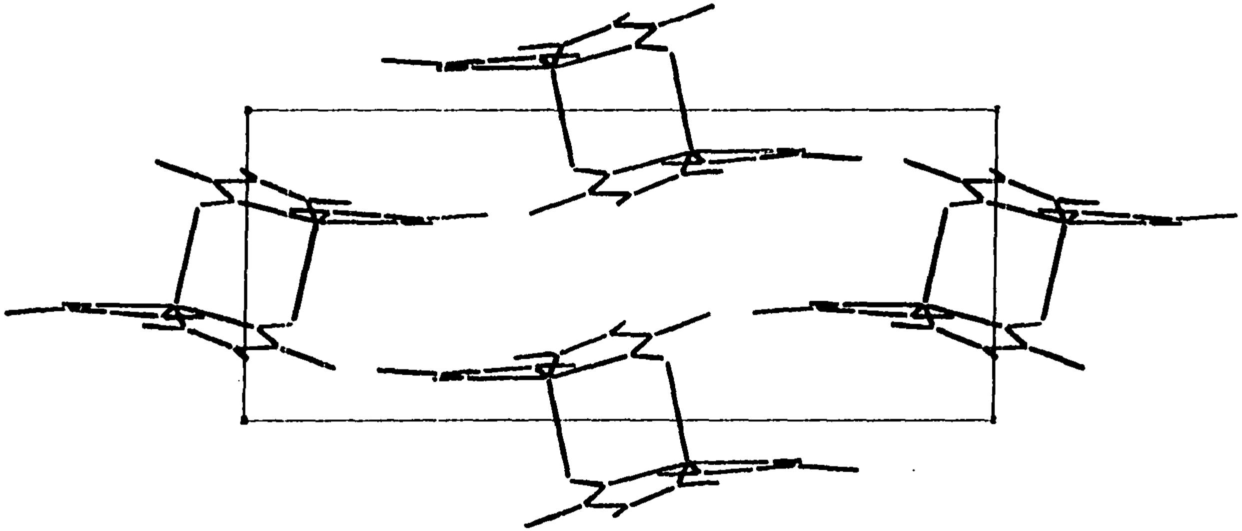

Figure 4. Clinographic projection, along a, of the contents of a unit cell of crystalline $Cu(DMG)_2$-d_2

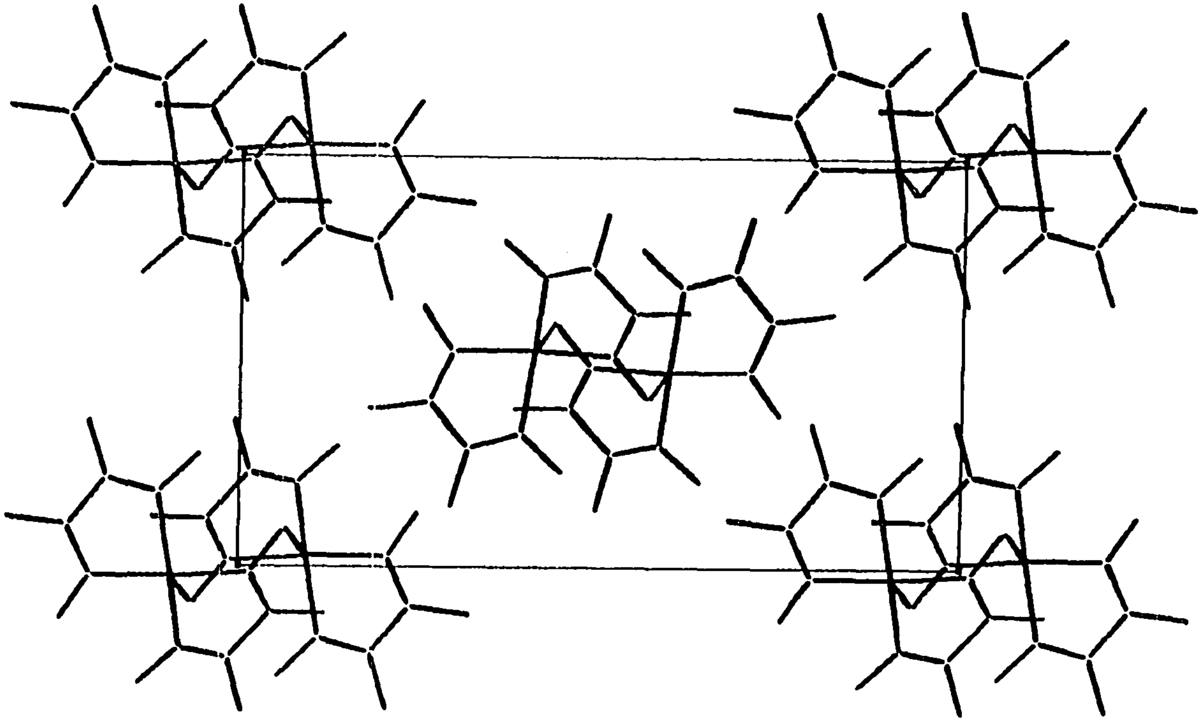

Figure 5. Clinographic projection, along c, of the contents of a unit cell of crystalline $Cu(DMG)_2$-d_2

Belfort (6) and consisted of crystals of form and purity suitable for X-ray studies.

2. Experimental procedures

a. Preparation of $Cu(DMG)_2$ A suitable crystal of $Cu(DMG)_2$ was selected and sealed in a thin-walled Lindemann glass capillary. Microscopic examination of the crystal revealed it to be needle-like with approximate dimensions 0.1 x 0.2 x 0.5 mm.

b. Collection of data Preliminary precession photographs exhibited $2/m$ Laue symmetry indicating a monoclinic space group. Systematic absences of ($h0\ell$) for $h + \ell$ odd and ($0k0$) for k odd indicated the space group C_{2h}^5-$P2_1/n$, in agreement with the previous determination (32). The lattice constants and their standard deviations were obtained by a least-squares fit (88) to 12 independent reflection angles whose centers were determined by left-right, top-bottom beam splitting on a previously aligned Hilger-Watts four circle diffractometer (Mo K_α radiation, $\lambda = 0.71069$ Å). Any error in the instrumental zero was eliminated by centering the reflection at both $+2\theta$ and -2θ. The lattice constants are:

$$\begin{aligned} a &= 9.7969 \pm 0.0019 \text{ Å}, \\ b &= 17.1194 \pm 0.0022 \text{ Å}, \\ c &= 7.1451 \pm 0.0022 \text{ Å}; \\ \beta &= 106.93 \pm 0.02°. \end{aligned}$$

There are no statistically significant differences between these parameters and those previously reported (32). Other crystal data are:

$\underline{M} = 293.77$ g/mole, $\underline{V} = 1146.4$ Å^3, $\underline{Z} = 4$, $\underline{D}_{\underline{c}} = 1.702$ g/cm^3, $\underline{F}(000) = 604e^-$. For data collection the selected crystal was mounted with $\underline{c}^*$ along the spindle axis. Data were collected at room temperature using the automated diffractometer. Two octants of data were taken using Zr-filtered Mo K_α radiation within a θ sphere of 25° ($\sin\theta/\lambda = 0.5947$). Some additional intensities were measured within a θ shell of 25-28° but the percentage of reflections for which the intensity could be considered observed was so low that data collection in this region of θ values was not completed. The θ-2θ step-scan technique, 0.01°/step counting for 0.4096 sec./step, was employed with a take-off angle of 4.5°. Variable step symmetric scan ranges were used with the number of steps used for a particular reflection determined as follows: $\underline{N} = 50 + 2$ per $^\circ\theta$. Individual backgrounds were obtained from stationary-crystal stationary-counter measurements for one-half the total scan time at each end of the scan. A total of 2653 reflections were measured in this way.

As a general check on electronic and crystal stability, the intensities of three standard reflections were measured periodically during the data collection. Monitoring options based on these standard counts were employed to maintain crystal alignment and to stop the data collection process if the standard counts fell below statistically allowed levels. No decrease resulting from decomposition was observed for any of the standards during data collection.

The intensity data were corrected for Lorentz-polarization effects, and for effects due to absorption. The absorption coefficient, μ, is 19.92 cm^{-1}, and an absorption correction (15) was made using ABCOR. The maximum and minimum transmission factors were 0.915 and 0.793, respectively. The estimated error in each intensity was calculated by $[\sigma(I)]^2 = [C_t + C_b + (0.05\ C_t)^2 + (0.05\ C_b)^2]$ where C_t and C_b are the total count and background count, respectively. Equivalent values of $\underline{F_o}^2$ were then averaged to yield 2455 independent $\underline{F_o}^2$ values. The estimated standard deviation in each structure factor was calculated from the mean deviation of intensity by the method of finite differences (89). The reciprocals of the structure factor variances were used as weights in the least-squares refinement.

3. Solution and refinement of the structure

The atomic coordinates for the non-hydrogen atoms in $Cu(DMG)_2$-d_2 were used as a starting point. This model was refined by full matrix least-squares techniques using a local modification of ORFLS (16) with isotropic thermal parameters to a conventional discrepancy factor of

$$\underline{R} = \Sigma||\underline{F_o}|-|\underline{F_c}||/\Sigma|\underline{F_o}| = 0.126$$

and a weighted $\underline{R}$-factor of

$$\omega\underline{R} = [\Sigma\omega(|\underline{F_o}|-|\underline{F_c}|)^2/\Sigma\omega|\underline{F_o}|^2]^{1/2} = 0.139.$$

The relativistic Dirac-Slater scattering factors for neutral atoms of Cromer and Waber (24) were used with those of copper modified for the real and imaginary parts of anomalous dispersion (23). Unless otherwise indicated, all unique reflections were used throughout the refinement.

At this stage anisotropic thermal parameters were included for copper and nitrogen and two cycles of refinement gave values for R and ωR of 0.084 and 0.095, respectively. Two further cycles with anisotropic thermal parameters included for all atoms except the methyl carbon atoms gave values of R and ωR of 0.074 and 0.083, respectively. An electron density difference map did not reveal the hydrogen atoms, but did reveal regions of positive density near the methyl carbon atoms which had the general shape of toroids and were interpreted as indicating more or less free rotation of the methyl groups. As an approximation to free rotation, six half-hydrogen atoms were located symmetrically about the methyl carbon atoms with the H-C-C angle taken as 109.5° and the C-H distance taken as 1.01 Å. The hydroxyl hydrogen atoms were placed midway between the oxygen atoms. All hydrogen atoms were given isotropic thermal parameters similar to those of the atoms to which they are bound. Two cycles of refinement with hydrogen parameters unvaried gave values for R and ωR of 0.059 and 0.060, respectively.

The positional and isotropic thermal parameters for the hydroxyl hydrogen atoms were next allowed to vary and two cycles of refinement gave values for R and ωR of 0.059 and 0.060, respectively. The isotropic B for one of the hydrogen atoms was negative, however. A statistical analysis of $\overline{\omega\Delta^2}$ [where $\Delta^2 = (|F_o|-|F_c|)^2$] as a function of scattering angle and magnitude of F_o revealed systematic fluctuations, and the weights were adjusted using the procedure described in the section on the refinement of the structure of Cu(DMG)$_2$-d$_2$. One cycle of refinement gave values for R and ωR of 0.060 and 0.057, respectively. Hydrogen positions were recalculated and two cycles of refinement with hydroxyl hydrogen parameters allowed to vary gave values for $\underline{R}$ and $\omega\underline{R}$ of 0.059 and 0.057, respectively. Recalculation of methyl hydrogen positions followed by two final cycles gave values for $\underline{R}$ and $\omega\underline{R}$ of 0.058 and 0.056, respectively. Repetition of the final two cycles using 1753 "observed" reflections (those with $\underline{F}_{\underline{o}}{}^2 > 3\sigma_{\underline{F}_{\underline{o}}{}^2}$) gave values for $\underline{R}$ and $\omega\underline{R}$ of 0.038 and 0.047, respectively, with no significant differences in the final refined parameters. A final electron density difference map revealed no peaks greater than 0.3e$^-$/Å^3. The final standard deviation for an observation of unit weight ($[\Sigma\omega\Delta^2/(NO-NV)]^{1/2}$ where NO is the number of observations [2455] and NV is the number of variables [162]) was 0.997 electrons. During the final cycle, the largest shift in any parameter was less than 0.01 times its own sigma.

The final positional and thermal parameters for non-hydrogen atoms, along with their standard deviations, are listed in Appendix A. Table 6 gives the final positional and isotropic thermal parameters, with standard deviations, for the hydroxyl hydrogen atoms. Standard deviations

Table 6. Final refined positional and isotropic thermal parameters for the hydroxyl hydrogen atoms in Cu(DMG)$_2$

	X	Y	Z	B
H(1)	0.2363(67)	0.1946(41)	0.3374(115)	5.95(170)
H(2)	-0.2184(57)	0.0085(29)	0.3221(78)	2.49(104)

were obtained from the inverse matrix of the final least-squares refinement cycle. Root-mean-square components of thermal displacement along the principal axes are given in Table 7 for all refined atoms. A listing of all 2455 unique recorded and calculated structure amplitudes is found in Appendix B. An indication of the directions and root-mean-square amplitudes of vibration for the refined atoms is provided by Figure 6.

Table 7. Root-mean-square components of thermal displacement (Å) along principal axes for refined atoms in $Cu(DMG)_2$

	Axis		
	1	2	3
Cu	0.155	0.178	0.230
N(1)	0.168	0.211	0.236
N(2)	0.173	0.189	0.223
N(3)	0.164	0.207	0.232
N(4)	0.176	0.202	0.230
O(1)	0.160	0.246	0.295
O(2)	0.162	0.219	0.230
O(3)	0.170	0.229	0.293
O(4)	0.171	0.229	0.263
C(1)	0.172	0.207	0.224
C(2)	0.181	0.260	0.307
C(3)	0.177	0.191	0.206
C(4)	0.190	0.240	0.258
C(5)	0.180	0.201	0.217
C(6)	0.183	0.253	0.267
C(7)	0.196	0.211	0.219
C(8)	0.180	0.273	0.282
H(1)	0.275		
H(2)	0.178		

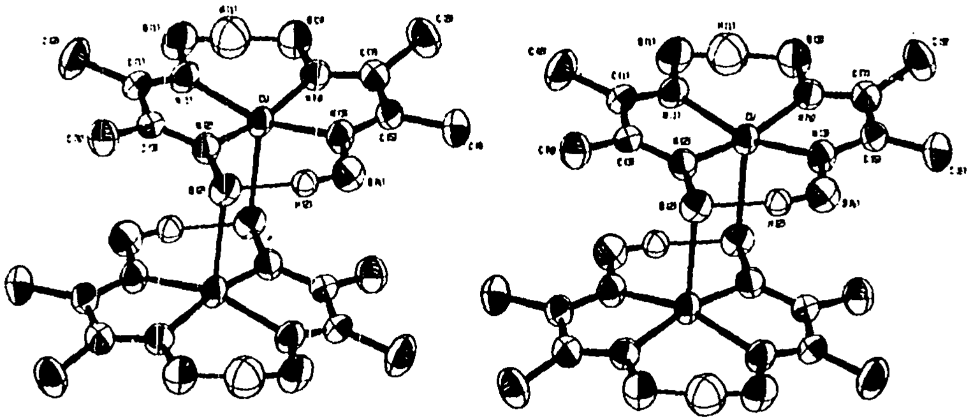

Figure 6. Stereo illustration of the thermal motion in $Cu(DMG)_2$ with ellipsoids scaled to enclose 50% probability.

4. Description of the structure

Interatomic distances and angles with standard deviations are given in Tables 8 and 9, respectively, and illustrated in Figure 7. The

Table 8. Interatomic distances in Cu(DMG)$_2$

	Without thermal motion, Å	With thermal motion[a], Å
Cu-N(1)	1.949 (3)	1.952 (3)
Cu-N(2)	1.947 (3)	1.948 (3)
Cu-N(3)	1.968 (3)	1.972 (3)
Cu-N(4)	1.943 (3)	1.946 (3)
N(1)-O(1)	1.367 (4)	1.385 (4)
N(2)-O(2)	1.353 (4)	1.358 (4)
N(3)-O(4)	1.389 (4)	1.399 (4)
N(4)-O(3)	1.333 (4)	1.350 (4)
C(1)-N(1)	1.289 (5)	1.292 (5)
C(3)-N(2)	1.294 (5)	1.295 (5)
C(5)-N(3)	1.283 (5)	1.284 (5)
N(4)-C(7)	1.299 (5)	1.302 (5)
C(3)-C(1)	1.481 (5)	1.484 (5)
C(5)-C(7)	1.486 (5)	1.489 (6)
C(1)-C(2)	1.483 (6)	1.507 (6)
C(3)-C(4)	1.488 (6)	1.505 (6)
C(5)-C(6)	1.494 (6)	1.510 (6)
C(7)-C(8)	1.494 (5)	1.515 (6)
Cu-O(2')	2.294 (3)	2.298 (3)
O(1)-O(3)	2.547 (4)	2.547 (4)
O(2)-O(4)	2.699 (4)	2.703 (4)
O(1)-H(1)	1.142 (71)	
O(3)-H(1)	1.414 (70)	
O(2)-H(2)	1.731 (53)	
O(4)-H(2)	0.976 (52)	

[a] Assuming a riding model with the second atom given riding on the first.

Table 9. Interatomic angles (°) in Cu(DMG)$_2$

N(1)-Cu-N(3)	158.5 (1)	N(2)-Cu-N(4)	165.5 (1)
N(1)-Cu-N(2)	80.6 (1)	N(1)-Cu-N(4)	96.1 (1)
N(3)-Cu-N(4)	80.2 (1)	N(2)-Cu-N(3)	97.7 (1)
N(1)-Cu-O(2')	103.8 (1)	N(3)-Cu-O(2')	97.7 (1)
N(2)-Cu-O(2')	91.7 (1)	N(4)-Cu-O(2')	102.8 (1)
Cu-N(1)-O(1)	123.8 (2)	Cu-N(3)-O(4)	124.9 (2)
Cu-N(2)-O(2)	122.6 (2)	Cu-N(4)-O(3)	122.9 (2)
Cu-N(1)-C(1)	116.1 (3)	Cu-N(3)-C(5)	116.3 (3)
Cu-N(2)-C(3)	115.8 (2)	Cu-N(4)-C(7)	116.2 (3)
C(1)-N(1)-O(1)	120.0 (3)	C(5)-N(3)-O(4)	118.6 (3)
C(3)-N(2)-O(2)	121.6 (3)	C(7)-N(4)-O(3)	120.8 (3)
N(1)-C(1)-C(2)	123.6 (4)	N(3)-C(5)-C(6)	124.9 (4)
N(2)-C(3)-C(4)	123.3 (3)	N(4)-C(7)-C(8)	123.5 (4)
N(1)-C(1)-C(3)	113.5 (3)	N(3)-C(5)-C(7)	113.2 (3)
N(2)-C(3)-C(1)	113.8 (3)	N(4)-C(7)-C(5)	113.8 (3)
C(2)-C(1)-C(3)	122.9 (4)	C(6)-C(5)-C(7)	121.9 (3)
C(4)-C(3)-C(1)	122.9 (3)	C(8)-C(7)-C(5)	122.6 (4)
N(1)-O(1)-O(3)	96.0 (2)	N(2)-O(2)-O(4)	96.7 (2)
N(4)-O(3)-O(1)	98.8 (2)	N(3)-O(4)-O(2)	93.6 (2)
N(1)-O(1)-H(1)	101.1 (33)	N(2)-O(2)-H(2)	100.0 (16)
N(4)-O(3)-H(1)	103.0 (27)	N(3)-O(4)-H(2)	99.5 (29)
O(1)-H(1)-O(3)	170.7 (61)	O(2)-H(2)-O(4)	170.7 (45)
N(2)-O(2)-Cu'	102.6 (2)		

Dihedral angle between two planes, each defined by three atoms

Cu, N(1), N(2) Cu, N(3), N(4)	155.8 (1)

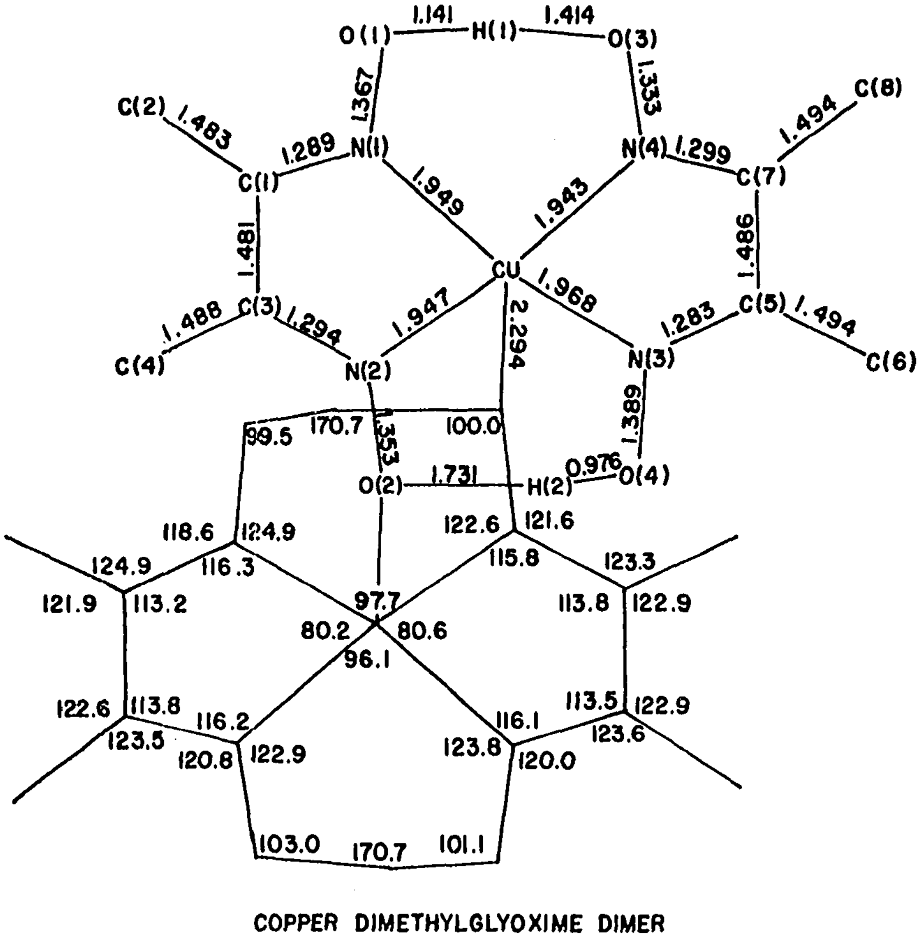

Figure 7. Interatomic distances and angles in $Cu(DMG)_2$

standard deviations in bond distances and angles were calculated using the variance-covariance matrix and ORFFE (17) and include errors in the lattice constants. The crystal packing of $Cu(DMG)_2$ is illustrated in Figure 8.

It seems reasonable to conclude that the shorter hydrogen bond in $Cu(DMG)_2$, as well as the longer bond, is asymmetrical. This conclusion is based on several structural features. The difference in the N(1)-O(1) and N(4)-O(3) bond distances is highly significant. The hydroxyl hydrogen atom in the shorter bond did not refine to the center of the bond, but rather is much closer to O(1), in agreement with the N-O distances. Finally, the refined O---O distance of 2.547 Å is longer than would be expected were this hydrogen bond symmetrical.

It also seems reasonable to conclude that the hydrogen bonds are not linear. This conclusion is suggested by the refined positions of the hydroxyl hydrogen atoms, and by the thermal motion of the oxygen atoms. Examination of the N-O-O and N-O-H angles also supports this conclusion. The N-O-O angles are, on the average, 8.2° smaller than the H-O-H angle of 104.5° in H_2O. The N-O-H angles, in contrast, are, on the average, only 3.6° smaller than the H-O-H angle in H_2O. This decrease can be explained as resulting from hydrogen bond formation. Hamilton (39) has noted in a neutron diffraction study of DMG that the tendency of the covalent bond angles to remain undistorted can outweigh any inherent tendency of the hydrogen bond to be linear.

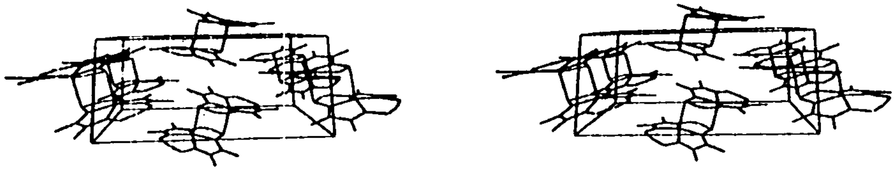

Figure 8. Stereo illustration of the crystal packing of $Cu(DMG)_2$

An examination of other bond distances in the molecule reveals no significant differences between bonds expected to be chemically equivalent with the exception of bonds Cu-N(3) (1.968 Å) and Cu-N(4) (1.943 Å). Some difference would be expected here though because of the distortions which arise as a result of dimer formation. The nitrogen atoms in question are a part of the ligand not involved in dimer formation. This ligand is bent away from the other half of the dimer and twisted with respect to the other ligand in the same half of the dimer. This is illustrated in Figure 9. This distorted square pyramidal configuration around the copper atom might well be expected to give rise to differences in bond lengths for bonds which, in the absence of the distortion, would normally be equivalent. The intraligand bond distances in $Cu(DMG)_2$ agree quite well with those in other oximes, and especially in other metal(II)-vic-dioximes.

An examination of the thermal parameters for the non-hydrogen atoms or their root-mean-square displacements along principal axes reveals several interesting features. For eleven of the non-hydrogen atoms at least one of the two shorter principal axes is within 14° of one of the bonds to that atom, the exceptions being the carbon atoms in the chelate rings and atoms N(4) and O(4). The carbon atoms though are nearly isotropic. In the case of the remaining two atoms, N(4) and O(4), at least one axis is within 26° of a bond and for N(4) an additional axis is within 22° of a bond.

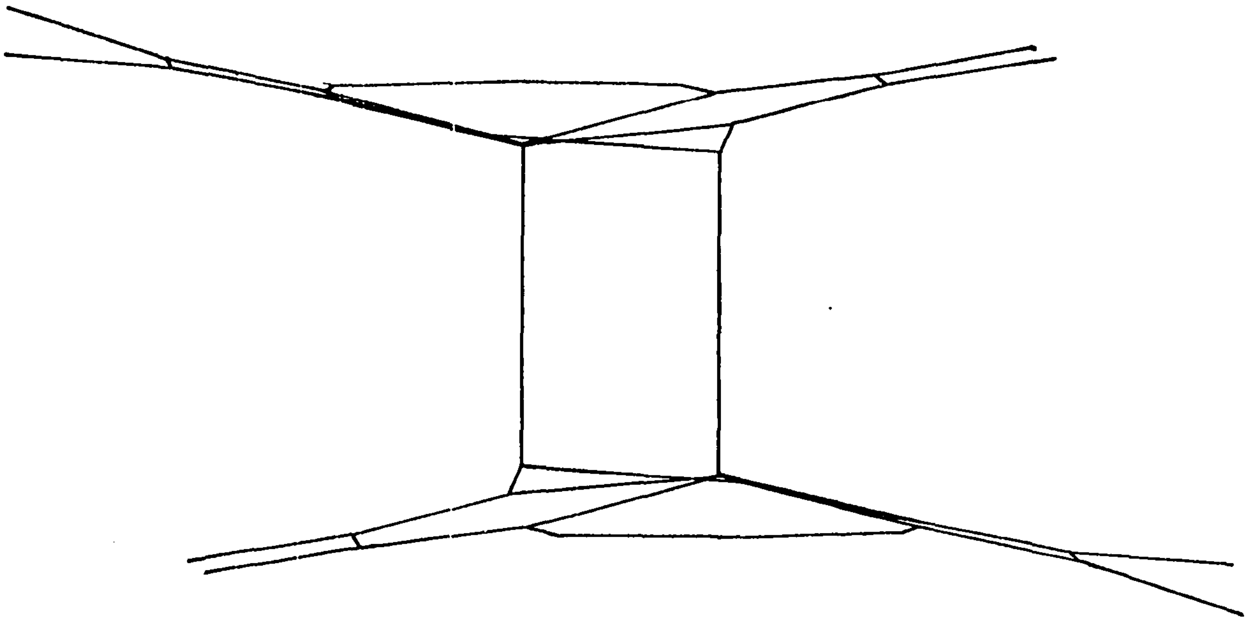

Figure 9. Stick bond illustration of a dimer of $Cu(DMG)_2$

A comparison of root-mean-square components of thermal displacement along principal axes reveals fairly good agreement between like atoms. The magnitude of the root-mean-square thermal displacement is found to increase with distance from the center of the molecule, a consequence of greater freedom of motion for atoms nearer to the extremeties of the molecule.

An examination of bond distances corrected for thermal motion assuming a riding model reveals some small, insignificant increases in the bond lengths in the chelate ring, and larger, significant increases in the N-O and C-C bonds to terminal atoms. The average increase of these distances amount to 0.013 and 0.019 Å, respectively.

Calculation of the best mean planes through the five-membered chelate rings reveals a lack of planarity for both rings by as much as 0.05 Å. Table 10 gives the equations of these planes and the deviations of the atoms from them.

Table 10. Equations of the least-squares planes through the rings in $Cu(DMG)_2$ and deviations of the atoms from them

Plane I[a]

$$2.219x - 4.481y + 5.942z - 1.823 = 0.0$$

	d(Å)	t_o	
Cu	-0.00005	0.13	
N(1)	-0.00716	1.84	
N(2)	0.00902	3.27	$\chi^2 = 82.8$
C(1)	0.01854	4.75	
C(3)	-0.02791	6.79	

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[a] Equations are in fractional coordinates, for the molecule nearest the origin, referred to the non-orthogonal crystallographic axes.

Table 10. (Continued)

Plane II

$$-0.947x - 0.369y + 7.003z - 2.511 = 0.0$$

	d(Å)	t_o	
Cu	0.00029	0.71	
N(3)	-0.02444	9.05	
N(4)	-0.00827	2.95	$\chi^2 = 263.4$
C(5)	0.05294	12.91	
C(7)	-0.00889	2.36	

A comparison of the structures of $Cu(DMG)_2$ and $Cu(DMG)_2$-d_2 determined in this study reveals only one significant difference, namely the C(5)-C(7) bond lengths. Careful examination of the structures suggests that this difference is very likely the result of inaccuracy in the position of C(7) in $Cu(DMG)_2$-d_2.

The present study does not indicate a significant expansion of the hydrogen bonds in $Cu(DMG)_2$ on deuteration. Both hydrogen bonds do increase in length however, and the lack of significance does not necessarily exclude an isotope effect.

A comparison of the results of this study, and another recently reported (81) redetermination of the structure of $Cu(DMG)_2$ reveals an amazing agreement in all refined parameters. This is particularly remarkable in view of the fact that thermal parameters tend to be sensitive to systematic errors which can occur in any structural determination, and the fact that the other redetermination was based on intensities visually estimated from films. This agreement lends credence to the refined thermal parameters and the conclusions drawn from them. A comparison of bond distances from the two studies reveals only one significant difference. Unfortunately, that difference is in the O(1)-O(3) distance, the value reported by Vaciago and Zambonelly being 2.526 Å. If the value of 2.526 Å (81) is used in comparing the normal and deuterated structures, a significant expansion of the shorter hydrogen bond is indicated. In view of the fact that both studies result in an apparent expansion of both bonds a result which was anticipated from other structural studies of hydrogen bonds (22, 71), and the fact that the differences in the O---O distances determined in the present study are close to being significant, it seems reasonable to conclude that more precise structural information for $Cu(DMG)_2$-d_2 would reveal a significant increase in both O---O distances.

C. Crystal and Molecular Structure of $Ni(EMG)_2$

The crystal and molecular structure of $Ni(EMG)_2$ was first reported by Frasson and Ponattoni (33). The O---O distance reported in that study was totally inconsistent with the observed O-H stretching frequency (21), the reported value (2.33 Å) being much shorter than the expected value. The structure was therefore redetermined.

1. Apparatus and materials

a. Instrumentation and apparatus A Nonius precession camera was used to obtain preliminary photographs.

Intensity data were collected on a fully automated Hilger-Watts four circle diffractometer equipped with a scintillation counter and interfaced with an SDS-910 computer in a real time mode. Subsequent calculations were performed on an IBM 360/65 computer.

b. Chemicals Ethylmethylglyoxime was prepared by oximation of the Eastman product 2,3-pentanedione. The preparation was by N. Wilcox of this Laboratory following the procedure of Bryant and Smith (14).

2. Experimental procedures

a. Preparation of $Ni(EMG)_2$ $Ni(EMG)_2$ was prepared by the following procedure. Stoichiometric amounts of $NiCl_2$ and EMG in aqueous solutions were mixed and dilute aqueous NH_3 was added to precipitate the bright orange complex. The precipitate was filtered and washed. Two different modifications of $Ni(EMG)_2$ had been reported in the literature (1). One of these crystallized in a monoclinic space group and was designated the α-form. The other crystallized in an orthorhombic space group and was designated the β-form. The previous structural study was of the α-form. A recent study by Egneus (29) indicated that the α-form is the more stable of the two and that crystallization from chloroform produces this modification alone. The precipitated $Ni(EMG)_2$ was therefore recrystallized from chloroform, giving dark orange needle-like crystals of relatively large cross-section. A suitable crystal was selected and sealed in a Lindemann thin-walled glass capillary.

b. Collection of data Preliminary precession photographs exhibited $2/m$ Laue symmetry, indicating a monoclinic space group. Systematic absences of $h0\ell$ for ℓ odd and $0k0$ for k odd indicated the space group C_{2h}^5-$P2_1/c$ in agreement with the previously reported determination. The unit cell parameters and their standard deviations were obtained by a least-squared fit (88) to 14 independent reflection angles whose centers were determined by left-right top-bottom beam splitting on a previously aligned Hilger-Watts four circle diffractometer (Mo K_α radiation, $\lambda = 0.71069$ Å). Any error in the instrumental zero was eliminated by centering the reflection at both $+2\theta$ and -2θ. The refined lattice constants are:

$$\begin{aligned} \underline{a} &= 4.7471 \pm 0.0005\ \text{Å}, \\ \underline{b} &= 11.7409 \pm 0.0030\ \text{Å}, \\ \underline{c} &= 11.9895 \pm 0.0020\ \text{Å}; \\ \beta &= 91.611 \pm 0.016^\circ. \end{aligned}$$

Other crystal data are: $C_{10}H_{18}N_4O_4Ni$, $\underline{M} = 316.99$ g/mole, $\underline{Z} = 2$, $\underline{V} = 668.0$ Å^3, $\underline{D}_{\underline{c}} = 1.576$ g/cm^3, $\underline{F}(000) = 332e^-$. For data collection the selected crystal, with approximate dimensions 0.2 x 0.2 x 0.5 mm, was mounted with a along the spindle axis. Data were collected at room temperature using the fully automated Hilger-Watts diffractometer. Two octants of data were recorded using Zr-filtered Mo K_α radiation within a 2θ sphere of 50° ($\sin \theta/\lambda = 0.5947$). The θ-2θ step scan technique, 0.01°/step counting for 0.4096 sec./step, was employed with a take-off angle of 4.5°. Variable step symmetric scan ranges were used with the number of steps for a given reflection determined as follows: $\underline{N} = 50 + 2$ per $^\circ\theta$. Stationary-crystal stationary-counter background measurements were made by counting for one-half the total scan time at each end of the scan. A total of 1608 reflections were measured this way.

As a general check on electronic and crystal stability, the intensities of three standard reflections were measured periodically during the data collection. Monitoring options based on these standard counts were used to maintain crystal alignment and to stop the collection of data if standards counts fell below statistically allowed fluctuations. No decrease due to decomposition was observed in any of the standards during data collection.

The intensity data were corrected for Lorentz-polarization effects. The absorption coefficient, μ, is 14.68 cm^{-1}, and an absorption correction (15) was made using ABCOR. The maximum and minimum transmission factors were 0.839 and 0.755, respectively. The estimated error in each intensity was calculated by $[\sigma(I)]^2 = [C_t + C_b + (0.05\ C_t)^2 + (0.05\ C_b)^2]$ where C_t and C_b are the total count and background count, respectively. The individual values of $\underline{F_o}^2$ from equivalent sets were then averaged to give 1397 independent $\underline{F_o}^2$ values. The estimated standard deviation in each structure factor was calculated from the mean deviation of intensity by the method of finite differences (89). The reciprocals of the structure factor variances were used as weights in the least-squares refinement.

3. Solution and refinement of the structure

Due to the presence of only two molecules per unit cell, the metal atoms are restricted to the centers of symmetry. The positions of the remaining atoms except hydrogen were obtained from three-dimensional electron density difference maps and were refined by full matrix least-squares techniques (16) with isotropic thermal parameters to a conventional discrepancy factor of

$$\underline{R} = \Sigma||\underline{F_o}|-|\underline{F_c}||/\Sigma|\underline{F_o}| = 0.135,$$

and a weighted $\underline{F}$-factor of

$$\omega\underline{R} = [\Sigma\omega(|\underline{F_o}|-|\underline{F_c}|)^2/\Sigma\omega|\underline{F_o}|^2]^{1/2} = 0.122.$$

The relativistic Dirac-Slater X-ray scattering factors for neutral atoms as computed by Cromer and Waber (24) were used, with those of nickel modified for the real and imaginary parts of anomalous dispersion (23). All unique data were used in the refinement.

At this point anisotropic thermal parameters were included for nickel and nitrogen atoms and two cycles of refinement gave a value for $\underline{R}$ of 0.115. The remaining atoms, with the exception of the methyl carbon atoms, were then given anisotropic thermal parameters and two cycles of refinement gave values for $\underline{R}$ and $\omega\underline{R}$ of 0.104 and 0.094, respectively.

An electron density difference map was calculated and revealed some rather diffuse positive regions which could be interpreted as hydrogen atoms. Due to the uncertainty in the positions of these atoms, however, their positions were calculated assuming tetrahedral methyl groups and a C-H distance of 1.08 Å. The methyl hydrogen atoms were placed as near as possible to the observed peaks in the difference map. The methylene hydrogen atoms were placed in the plane which bisects the C-C-C angle of the ethyl substituent, at an angle of 109.5° from one another. The four H-C-C angles were assumed to be equal. The hydroxyl hydrogen atom was placed midway between the oxygen atoms. All hydrogen atoms were assigned isotropic thermal parameters similar to those of the atoms to which they are bound.

Two cycles of refinement with hydrogen atom parameters unvaried and anisotropic thermal parameters included for all non-hydrogen atoms gave values for $\underline{R}$ of $\omega\underline{R}$ of 0.089 and 0.074, respectively. Hydrogen atom positions were then recalculated and two further cycles gave values for $\underline{R}$ and $\omega\underline{R}$ of 0.088 and 0.073, respectively.

A statistical analysis of $\overline{\omega\Delta^2}$, where $\Delta^2 = (|\underline{F}_{\underline{o}}|-|\underline{F}_{\underline{c}}|)^2$, as a function of scattering angle and magnitude of $\underline{F}_{\underline{o}}$ indicated that the relative weighting scheme used was reasonable. An attempt to refine the positional and thermal parameters of the methyl hydrogen atoms was not entirely successful. The average refined C-H distance of 1.01 Å was used in subsequent calculations of the positions of the methyl hydrogen atoms, however. During the final refinement the positional parameters of all hydrogen atoms were calculated and not varied because the agreement between observed and calculated structure amplitudes seemed insensitive to these parameters. Two final cycles of refinement gave values for $\underline{R}$ and $\omega\underline{R}$ of 0.088 and 0.073, respectively. Two final cycles of refinement using 802 "observed" reflections (those such that $\underline{F}_{\underline{o}}^{\,2} > 3\sigma_{\underline{F}_{\underline{o}}^{\,2}}$) gave values for $\underline{R}$ and $\omega\underline{R}$ of 0.048 and 0.060, respectively, with no significant shifts in the final refined parameters. A final electron density difference map revealed no peaks greater than $0.4e^-/\text{Å}^3$. The final standard deviation for an observation of unit weight $([\Sigma\omega\Delta^2/(\text{NO-NV})]^{1/2}$ where NO is the number of observations [1397] and NV is the number of variables [88]) was 1.26e$^-$. During the final cycle the largest shift in any parameter was less than 0.01 times its own sigma.

A listing of the final refined positional and thermal parameters for the non-hydrogen atoms along with their standard deviations as derived from the inverse matrix of the final least-squares refinement cycle is provided in Appendix A. Root-mean-square components of thermal displacement along the principal axes are given in Table 11. A listing of all 1397 unique recorded and calculated structure amplitudes is found in Appendix B.

Table 11. Root-mean-square components of thermal displacement (Å) along principal axes for refined atoms in Ni(EMG)$_2$

	Axis		
	1	2	3
Ni	0.185	0.213	0.240
N(1)	0.176	0.232	0.238
N(2)	0.201	0.231	0.253
O(1)	0.206	0.245	0.315
O(2)	0.200	0.249	0.306
C(1)	0.219	0.222	0.248
C(2)	0.204	0.211	0.245
C(3)	0.219	0.271	0.311
C(4)	0.206	0.261	0.274
C(5)	0.230	0.304	0.352

4. Description of the structure

Interatomic distances and angles with standard deviations are given in Table 12 and illustrated in Figure 10. The standard deviations in the

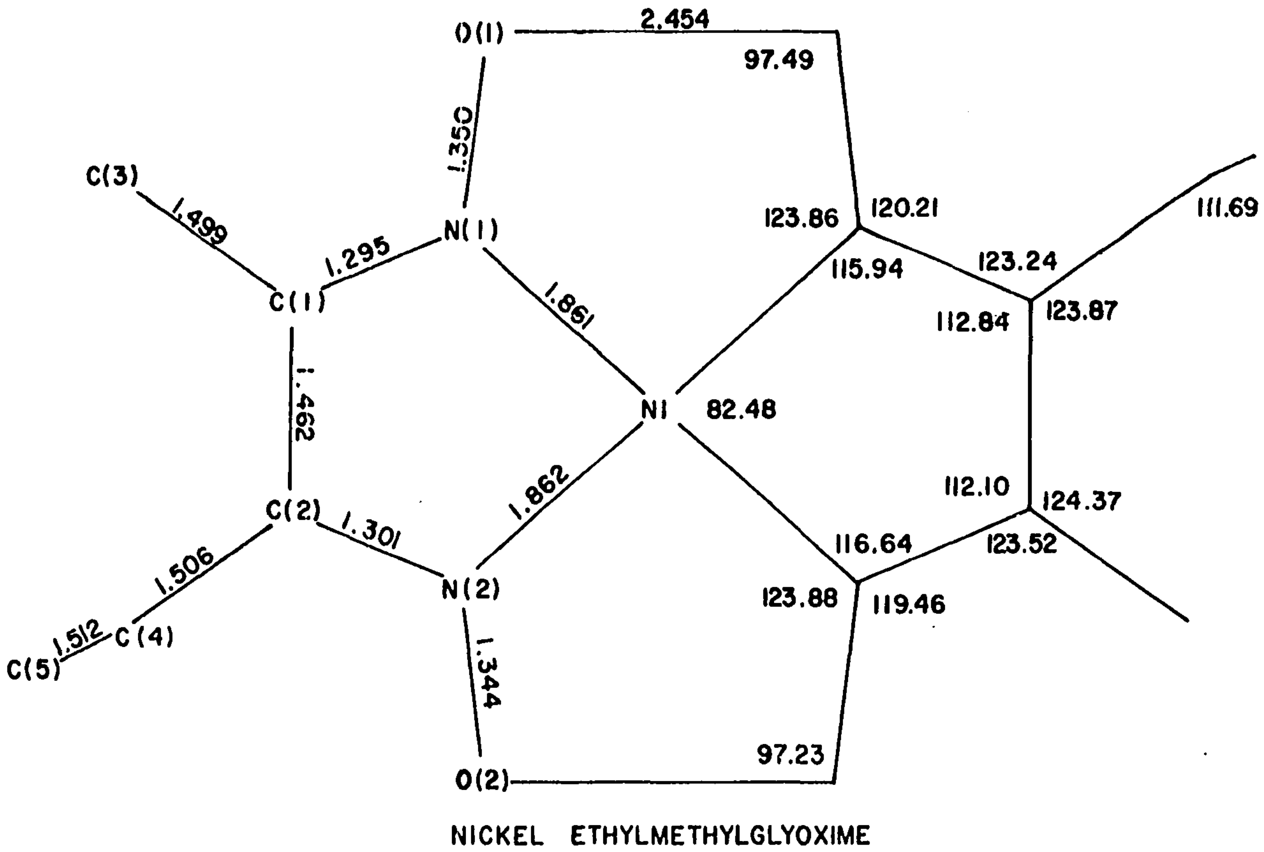


Figure 10. Interatomic distances and angles in $Ni(EMG)_2$

Table 12. Interatomic distances and angles in Ni(EMG)$_2$

Distances (Å)

	Without thermal motion	With thermal motion*
Ni-N(1)	1.861 (4)	1.860 (4)
Ni-N(2)	1.862 (4)	1.865 (4)
N(1)-O(1)	1.350 (5)	1.369 (5)
N(2)-O(2)	1.344 (5)	1.358 (5)
N(1)-C(1)	1.295 (6)	1.297 (6)
C(2)-N(2)	1.301 (5)	1.304 (6)
C(2)-C(1)	1.462 (7)	1.466 (7)
C(1)-C(3)	1.499 (7)	1.519 (7)
C(2)-C(4)	1.506 (7)	1.520 (7)
C(4)-C(5)	1.512 (8)	1.539 (8)
O(2)-O(1')	2.454 (5)	2.454 (5)

Angles (°)

Ni-N(1)-O(1)	123.9 (3)	Ni-N(2)-O(2)	123.9 (3)
Ni-N(1)-C(1)	116.6 (3)	Ni-N(2)-C(2)	115.9 (3)
N(1)-O(1)-O(2')	97.2 (3)	N(2)-O(2)-O(1')	97.5 (3)
C(1)-N(1)-O(1)	119.5 (4)	C(2)-N(2)-O(2)	120.2 (4)
N(1)-C(1)-C(2)	112.1 (4)	N(2)-C(2)-C(1)	112.8 (4)
N(1)-C(1)-C(3)	123.5 (5)	N(2)-C(2)-C(4)	123.2 (4)
C(3)-C(1)-C(2)	124.4 (5)	C(4)-C(2)-C(1)	123.9 (4)
C(2)-C(4)-C(5)	111.7 (5)	N(1)-Ni-N(2)	82.5 (2)

*Assuming a riding model with the second atom given riding on the first.

distances and angles were calculated using the variance-covariance matrix and OR FFE (17), and include errors in the lattice constants. An indication of the directions and root-mean-square amplitudes of vibration of the refined atoms is provided by Figure 11. The packing of the molecules in the crystal is illustrated in Figure 12.

The present study reveals a much longer O---O distance than was previously reported (33). The value of 2.454 Å found in this study agrees well with the distance expected from the infrared spectral study of Caton and Banks (21), and with the 2.453 Å O---O distance (18) in $Ni(G)_2$. There is no significant difference in the N-O distances, indicating a symmetrical hydrogen bond. A symmetrical hydrogen bond is also suggested by the O---O distance. The thermal motion of the oxygen atoms suggests that the hydrogen bond is not linear. A non-linear hydrogen bond seems reasonable on considering the fact that the N-O-O angles in $Ni(EMG)_2$ average 7.2° smaller than the 104.5° H-O-H angle in H_2O.

A critical examination of chemically equivalent bonds reveals no significant differences, and good agreement with the corresponding distances in other oximes, vic-dioximes, and metal(II)-vic-dioxime complexes.

A significant feature of the complex is the delocalization of the two C=N bonds over the C-C bond in the chelate ring, which is evidenced in the shortening of the C-C bond from the normal single bond distance and a lengthening of the C=N bonds from the normal double bond length. Similar effects have been observed in the glyoxime complexes of nickel (18, 52), palladium (19) and platinum (31), and in dimethylglyoxime (39).

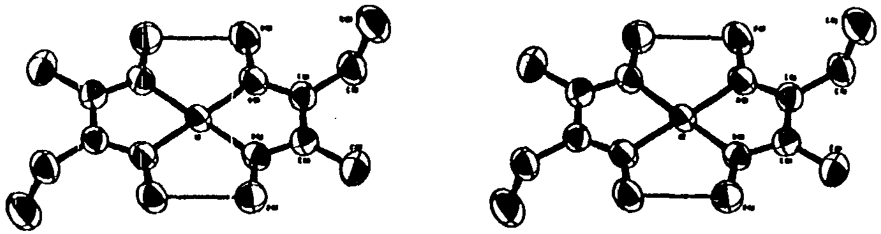


Figure 11. Stereo illustration of the thermal motion in $Ni(EMG)_2$ with ellipsoids scaled to enclose 50% probability.

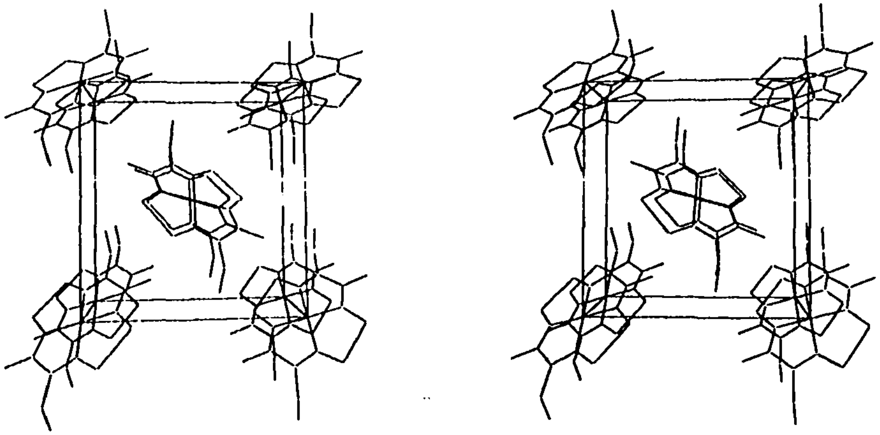


Figure 12. Stereo illustration of the crystal packing of $Ni(EMG)_2$

A careful examination of the thermal parameters reveals nothing unusual or unexpected in the thermal motion of the heavy atoms. The motion of the atoms in the chelate ring is more nearly isotropic than that of the atoms bound to the ring. The motion of the oxygen atoms and the carbon atoms bound to the ring is considerably more restricted in the direction of the bonds joining them to the ring. The motion of the terminal carbon atom of the ethyl group reflects a significant contribution from a wagging of the entire ethyl group. For the nickel atom, the largest amplitude of motion is normal to the molecular plane.

Calculation of interatomic distances corrected for thermal motion assuming a riding model does not significantly change bond distances in the chelate ring, but results in increases of about 0.02 Å in the N-O bonds and the C-C bonds external to the ring.

Calculation of the best mean plane through the molecule indicates that the molecule is planar with the exception of the ethyl group and oxygen atom O(1), which lies only 0.02 Å out of the plane. The equation of the best plane through the planar portion of the molecule and the distances of the atoms from this plane are given in Table 13.

Table 13. Equation of the least-squares plane through the planar portion of the $Ni(EMG)_2$ molecule and deviations of the atoms from the plane

$$3.286x - 2.742y + 7.950z = 0.0^{a}$$

---

[a] Equation is in fractional coordinates, for the molecule at the origin, referred to the non-orthogonal crystallographic axes.

Table 13. (Continued)

	d(Å)	t_0[b]	
Ni	0.0000	0.00	
N(1)	0.0016	0.40	
N(2)	-0.0009	0.23	
C(1)	-0.0041	0.81	$\chi^2 = 5.71$
C(2)	-0.0078	1.55	
C(3)	0.0071	1.26	
C(4)	-0.0757		
C(5)	-1.5017		
O(1)	-0.0242		
O(2)	0.0033	0.92	

[b] Atoms for which t_o values are not given were not used in the calculation of the best mean plane.

D. The Crystal and Molecular Structure of $Ni(DMG)_2$

In view of the similarity in O-H stretching frequencies for $Ni(DMG)_2$ and $Ni(EMG)_2$ and the similarity in O---O distances in $Ni(EMG)_2$ (2.453 Å) and $Ni(G)_2$ (2.454 Å) it seemed likely that the O---O distance in $Ni(DMG)_2$ was not as short as the 2.40 Å reported (36, 90). The structure of this complex was therefore redetermined.

1. Apparatus and materials

a. Instrumentation and apparatus A Nonius precession camera was used to obtain preliminary photographs. Intensity data were collected on a fully automated Hilger-Watts four circle diffractometer equipped with a scintillation counter and interfaced with an SDS-910 computer in a real time mode. Calculations were performed on an IBM 360/65 computer.

b. Chemicals The preparation of $Ni(DMG)_2$ was by R. W. Vander Haar (82) following the standard analytical procedure (28). The product had been recrystallized from 1,2-dichlorobenzene and existed as needle-like crystals of small cross section.

2. Experimental procedures

a. Preparation of $Ni(DMG)_2$ A suitable crystal was selected and mounted on the end of a thin quartz fiber with DuPont Duco brand cement. The complex was first tested for a reaction with the cement and none was found.

b. Collection of data Preliminary precession photographs exhibited mmm Laue symmetry, indicating an orthorhombic space group. Systematic absences of (hkℓ) for h + k + ℓ odd, (0kℓ) for k + ℓ odd and (h0ℓ) for h + ℓ odd indicated two possible space groups D_{2h}^{26} - Ibam and C_{2v}^{21} - Iba2, in agreement with the previous determination (36). The higher space group Ibam was chosen initially and the successful refinement of the structure in this space group confirmed the choice. The unit cell parameters are:

$$a = 16.5835 \pm 0.0024 \text{ Å},$$
$$b = 10.4412 \pm 0.0046 \text{ Å},$$
$$c = 6.4753 \pm 0.0016 \text{ Å}.$$

These parameters and their standard deviations were obtained by a least-squares fit (88) to 12 independent reflection angles whose centers were determined by left-right top-bottom beam splitting on a previously aligned Hilger-Watts four circle diffractometer (Mo K_α radiation, $\lambda = 0.71069$ Å).

Any error in the instrumental zero was eliminated by centering the reflection at both $+2\theta$ and -2θ. Other crystal data are: $C_8H_{14}N_4O_4Ni$, $\underline{M}$ = 288.92 g/mole, $\underline{Z}$ = 4, $\underline{V}$ = 1121.2 Å^3, $\underline{D_c}$ = 1.711 g/cm^3,,F(000) = 600 e$^-$.

For data collection the selected crystal, with approximate dimensions 0.05 x 0.07 x 0.17 mm, was mounted with $\underline{a}$ along the spindle axis. Data were collected at room temperature using the fully automated Hilger-Watts diffractometer. One octant of data was recorded using Zr-filtered Mo K_α radiation within a 2θ sphere of 50° ($\sin\theta/\lambda = 0.5947$). The θ-2θ step-scan technique, 0.01°/step counting for 0.4096 sec./step, was employed with a take-off angle of 4.5°. Variable step symmetric scan ranges were used with the number of steps determined as follows: $\underline{N}$ = 50 + 2 per °θ. Stationary-crystal stationary-counter background measurements were made by counting for one-half the total scan time at each end of the scan. As a check on the reliability of the data 80 reflections from another octant were also measured; the reproducibility was excellent. A total of 639 reflections were measured.

As a general check on electronic and crystal stability, the intensities of three standard reflections were measured periodically during the data collection. Monitoring options based on these standard counts were used to maintain crystal alignment and to stop the data collection if standard counts fell below statistically allowed fluctuations. No decrease in any of the standards due to decomposition was observed during data collection.

The intensity data were corrected for Lorentz-polarization effects. The absorption coefficient, μ, is 17.39 cm^{-1}, and an absorption correction (15) was made using ABCOR. The maximum and minimum transmission factors were 0.906 and 0.754, respectively. The estimated error in each intensity was calculated by $[\sigma(I)]^2 = [C_t + C_b + (0.05\ C_t)^2 + (0.05\ C_b)^2]$ where C_t and C_b are the total count and background count respectively. The individual values of $\underline{F}_o{}^2$ from equivalent sets were then averaged to give 598 independent $\underline{F}_o{}^2$ values. The estimated standard deviation in each structure factor was calculated from the mean deviation of intensity by the method of finite differences (89). The reciprocals of the structure factor variances were used as weights in the least-squares refinement.

3. Solution and refinement of the structure

The requirements of symmetry restrict the nickel atoms to special positions with symmetry $\underline{2}/\underline{m}$ and the remaining atoms except hydrogen to the mirror planes, the minimum molecular symmetry being $\underline{2}/\underline{m}$. The positions of all atoms except hydrogen were obtained from a three-dimensional difference map, and were refined by full matrix least-squares techniques (16) with isotropic thermal parameters to a conventional discrepancy factor of

$$\underline{R} = \Sigma\big||\underline{F}_o|-|\underline{F}_c|\big|/\Sigma|\underline{F}_o| = 0.143,$$

and a weighted $\underline{R}$-factor of

$$\omega\underline{R} = [\Sigma\omega(|\underline{F}_o|-|\underline{F}_c|)^2/\Sigma\omega|\underline{F}_o|^2]^{1/2} = 0.098.$$

The relativistic Dirac-Slater X-ray scattering factors for neutral atoms computed by Cromer and Waber (24) were used with those of nickel modified for the real and imaginary parts of anomalous dispersion (23). An option in the refinement program was employed which allowed the exclusion from the refinement of any reflection not meeting a certain criterion; the criterion used was $||\underline{F}_o|-|\underline{F}_c|| < 6\sigma_{\underline{F}_o}$ and resulted in the exclusion of two reflections, (200) and (321). Because of the proximity of a to the spindle axis during data collection, and the fact the orientation matrix for the diffractometer becomes indeterminate in ϕ for low angle reflections very near to $\chi = 90°$, the value of ϕ for (200) was not correct and the reflection was not centered during measurement. This reflection was discarded from the refinement. The other reflection, (321), was not excluded and, in the later stages of refinement, met the criterion for retaining a reflection.

Inclusion of anisotropic thermal parameters (symmetry requires that $\beta(13)$ and $\beta(23)$ for all atoms be zero) for nickel and nitrogen and two cycles of refinement gave values for R and ωR of 0.135 and 0.093, respectively. At this point the hydroxyl hydrogen atom was included. It was placed midway between the oxygen atoms and given an isotropic B similar to those of the oxygens. Inclusion of anisotropic thermal parameters for the oxygen and ring carbon atoms and two cycles of refinement gave values for R and ωR of 0.135 and 0.092, respectively.

An electron density difference map failed to reveal the locations of hydrogen atoms. Their positions were therefore calculated. A free-rotation model was used for the methyl groups. Six half-hydrogen atoms were placed symmetrically about the methyl carbon atoms. The H-C-C angle was taken as 109.5° and the C-H distance was taken as 1.01 Å. These atoms were assigned isotropic thermal parameters similar to those of the atoms to which they are bound. The calculated positions of the methyl hydrogen atoms were such that the requirements of symmetry (the mirror plane) were adhered to, with two of the half-hydrogens being located in the mirror at $\underline{Z} = 0.0$. Two cycles of refinement with anisotropic thermal parameters for all non-hydrogen atoms gave values for $\underline{R}$ and $\omega\underline{R}$ of 0.122 and 0.082, respectively. Recalculation of hydrogen atom positions followed by two further cycles of refinement gave values for $\underline{R}$ and $\omega\underline{R}$ of 0.122 and 0.082, respectively.

A statistical analysis of $\overline{\omega\Delta^2}$, where $\Delta^2 = (|\underline{F}_o|-|\underline{F}_c|)^2$, as a function of scattering angle and magnitude of $\underline{F}_o$ revealed some systematic fluctuations for low values of $|\underline{F}_o|$. The refinement weights were therefore adjusted using the procedure described in the section on the refinement of the structure of $Cu(DMG)_2$-d_2.

Two cycles of refinement using the adjusted weights gave values for $\underline{R}$ and $\omega\underline{R}$ of 0.121 and 0.068, respectively. Hydrogen atom positions were recalculated and two cycles of refinement with the hydroxyl hydrogen atom parameters allowed to vary gave values for $\underline{R}$ and $\omega\underline{R}$ of 0.121 and 0.068, respectively. The values for the hydroxyl hydrogen atom parameters were reasonable and these parameters were varied through the final cycle. The final cycle gave values for $\underline{R}$ and $\omega\underline{R}$ of 0.121 and 0.068, respectively. Discarding reflections with $\underline{F}_o = 0.0$ from the calculation of the agreement factors gave values for $\underline{R}$ and $\omega\underline{R}$ of 0.104 and 0.063, respectively.

Two cycles of refinement using 320 "observed" reflections (those such that $\underline{F_o}^2 > 2\sigma_{\underline{F_o}^2}$) and the original weights gave values for $\underline{R}$ and $\omega\underline{R}$ of 0.052 and 0.056, respectively, with no significant shifts in any of the parameters. This suggests that the structure is better determined than the final value of $\underline{R}$ indicates, with the reflections with low intensities contributing considerably to the value of $\underline{R}$ but little to the refinement. A final electron density difference map revealed no peaks greater than $0.8e^-/\text{Å}^3$. The final standard deviation for an observation of unit weight ($[\Sigma\omega\Delta^2/(NO-NV)]^{1/2}$ where NO is the number of observations [597] and NV is the number of variables [56]) was $0.983e^-$. During the final cycle the largest shift in any parameter was less than 0.01 times its own sigma.

A listing of the final refined positional and thermal parameters for the non-hydrogen atoms along with their standard deviations as derived from the inverse matrix of the final least-squares refinement cycle is provided in Appendix A. The final refined parameters for the hydroxyl hydrogen atom are:

$\underline{x}$	$\underline{y}$	$\underline{z}$	$\underline{B}$
0.1140 (84)	0.1872 (110)	0	6.268 (3753)

The numbers in parentheses are the estimated standard deviations in the least significant digits. Root-mean-square components of thermal displacement along the principal axes are given in Table 14. A listing of all 1397 unique recorded and calculated structure amplitudes is provided in Appendix B.

Table 14. Root-mean-square components of thermal displacement (Å) along principal axes for refined atoms in $Ni(DMG)_2$

	Axis		
	1	2	3
Ni	0.182	0.200	0.213
N(1)	0.198	0.213	0.228
N(2)	0.190	0.207	0.231
O(1)	0.203	0.247	0.251
O(2)	0.219	0.255	0.263
C(1)	0.176	0.224	0.233
C(2)	0.188	0.198	0.243
C(3)	0.245	0.246	0.272
C(4)	0.214	0.278	0.281
H(1)	0.282		

4. Description of the structure

Interatomic distances and angles with standard deviations are given in Table 15 and illustrated in Figure 13. The standard deviations in the

Table 15. Interatomic distances and angles in $Ni(DMG)_2$

	Distances (Å)	
	Without thermal motion	With thermal motion[a]
Ni-N(1)	1.855 (6)	1.858 (6)
Ni-N(2)	1.865 (7)	1.868 (7)
N(1)-O(1)	1.328 (9)	1.342 (9)

---

[a] Assuming a riding model with the second atom riding on the first.

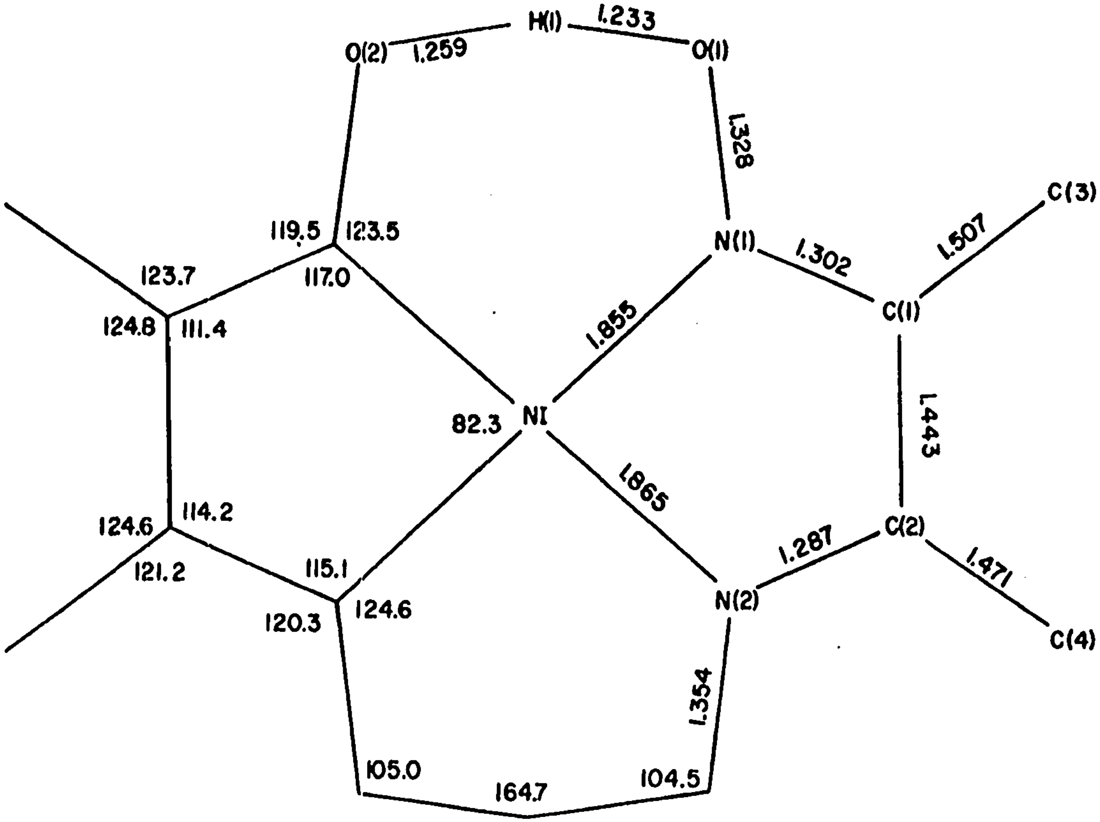
Table 15. (Continued)

	Without thermal motion	With thermal motion[a]
N(2)-O(2)	1.354 (9)	1.374 (9)
C(1)-N(1)	1.302 (11)	1.306 (12)
N(2)-C(2)	1.287 (11)	1.288 (12)
C(1)-C(3)	1.507 (14)	1.523 (14)
C(2)-C(4)	1.471 (13)	1.493 (13)
C(2)-C(1)	1.443 (12)	1.446 (13)
O(1)-O(2')	2.471 (9)	2.473 (9)
O(1)-H(1)	1.233 (124)	
O(2)-H(1')	1.259 (129)	

Angles (°)

Ni-N(1)-O(1)	124.6 (6)	Ni-N(2)-O(2)	123.5 (6)
Ni-N(1)-C(1)	115.1 (7)	Ni-N(2)-C(2)	117.0 (7)
C(1)-N(1)-O(1)	120.3 (7)	C(2)-N(2)-O(2)	119.5 (8)
N(1)-C(1)-C(2)	114.2 (9)	N(2)-C(2)-C(1)	111.4 (9)
N(1)-C(1)-C(3)	121.2 (9)	N(2)-C(2)-C(4)	123.7 (9)
C(3)-C(1)-C(2)	124.6 (10)	C(4)-C(2)-C(1)	124.8 (9)
N(1)-O(1)-O(2')	97.3 (5)	N(2)-O(2)-O(1')	96.9 (5)
N(1)-O(1)-H(1)	105.0 (55)	N(2)-O(2)-H(1')	104.5 (51)
O(1)-H(1)-O(2')	164.7 (104)	N(1)-Ni-N(2)	82.3 (4)

distances and angles were calculated using the variance-covariance matrix and OR FFE (17), and include errors in the lattice constants. An indication of the directions and root-mean-square amplitudes of vibration



NICKEL DIMETHYLGLYOXIME

Figure 13. Interatomic distances and angles in $Ni(DMG)_2$

of the refined atoms is provided by Figures 14 and 15. The stacking of molecules in the crystal is also illustrated in Figure 15. The crystal packing is illustrated in Figure 16.

This study reveals, as expected, a significantly longer O---O distance than previously reported (36, 90). The value of 2.471 Å found in this study agrees well with the O---O distances in $Ni(EMG)_2$ and $Ni(G)_2$. The difference in the N-O distances may be significant but in the absence of evidence to the contrary, the hydrogen bond must be considered to be symmetrical. The final refined position of the hydroxyl hydrogen atom indicates a symmetrical hydrogen bond, and the O---O distance is sufficiently short so as to be compatible with such a conclusion. The thermal motion of oxygen atom O(2) and the position of the hydroxyl hydrogen are suggestive of a non-linear hydrogen bond. The N-O-H angles average 104.8°, a value very close to the 104.5° H-O-H angle in H_2O, lending credence to the refined position of this hydrogen atom and supporting the conclusion of a non-linear bond. The O-H-O angle is 164.7°.

A critical examination of chemically equivalent bonds reveals only the one possibly significant difference, that in the N-O distances. Assuming equivalent N-O distances in the isolated molecule, the molecule must be considered to have $\underline{D_{2h}}$ symmetry. The bond distances agree well with their equivalents in other oximes, *vic*-dioximes, and metal(II)-*vic*-dioxime complexes.

As in other metal(II)-*vic*-dioximes, the C-C bond in the ring in $Ni(DMG)_2$ is significantly shorter than the C-C single bond distance. This was also found to be the case for $Ni(EMG)_2$ as described earlier.

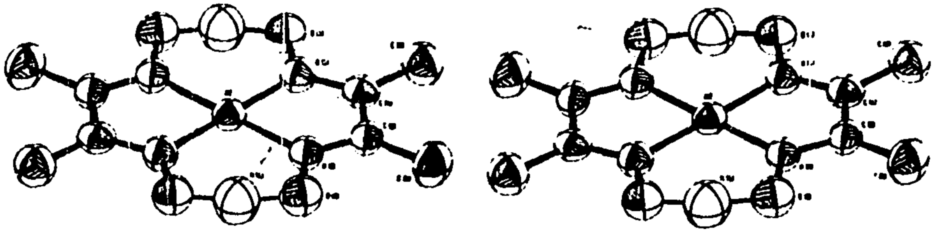


Figure 14. Stereo illustration of the thermal motion of $Ni(DMG)_2$ with ellipsoids scaled to enclose 50% probability

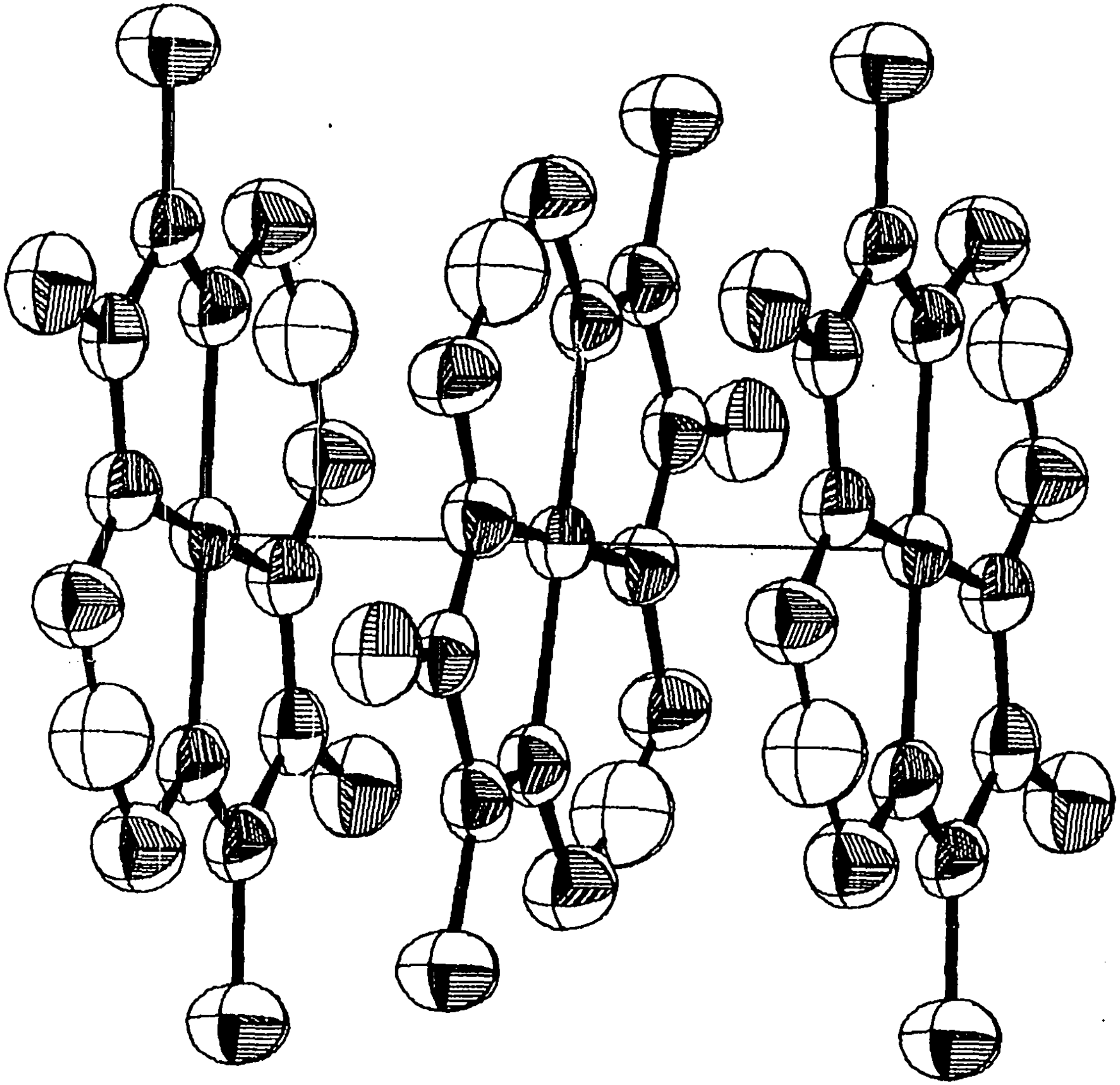


Figure 15. Illustration of the stacking of successive layers in Ni(DMG)$_2$ with ellipsoids scaled to enclose 50% probability

Figure 16. Stereo illustration of the crystal packing of $Ni(DMG)_2$

Delocalization of the C=N bonds over the chelate ring is apparently a feature common to all metal(II)-vic-dioximes with the possible exception of Cu(DMG)$_2$. This matter is discussed further below.

A careful examination of the thermal parameters reveals nothing unusual with the possible exception of the motion of atom C(1). This atom has an unusually large component of thermal motion along the C(1)-N(1) bond which cannot readily be accounted for and must therefore be ascribed to merely fitting some systematic error in the data. For all of the atoms at least one principal axis lies within 13° of a bond. The magnitudes of the root-mean-square components of vibration are reasonable and are larger for atoms O(1), O(2), C(3) and C(4) than for atoms in the ring. The motion of these latter four atoms is also more anisotropic, their motion being more restricted along the N-O and C-C bonds. The atoms in the chelate ring, with the exception of C(1), exhibit nearly isotropic motion with the smallest amplitude of motion normal to the ring.

Calculation of interatomic distances corrected for thermal motion assuming a riding model does not significantly change distances in the chelate ring, but results in increases of about 0.02 Å in the N-O bonds and the C-C bonds to the methyl carbon atoms.

E. Results and Discussion

For comparison purposes and easy reference, a summary of the most recently available structural information on metal(II)-vic-dioximes, in the form of interatomic distances and including the results of this study, is given in Table 16. In the case of $Cu(DMG)_2$ the results of another recent study (81) are also included. Estimated standard deviations as reported have also been included in order to permit statistical comparisons.

It seems apparent that the hydrogen bonding in the nickel(II)-vic-dioximes is symmetrical. If it is not, then the asymmetry must be very slight. It seems equally apparent that the hydrogen bonding in the palladium(II)- and platinum(II)-vic-dioximes is asymmetrical. For $Cu(DMG)_2$ the situation is complicated by dimer formation. In the solid complex the hydrogen bonds are apparently both asymmetrical.

The metal(II)-vic-dioximes are perhaps unique in permitting the direct observation of increasing oxygen-oxygen repulsions as the O---O distance decreases. Considering only the Ni, Pd, and Pt complexes for which the structures can be considered well determined [$Ni(G)_2$, $Ni(DMG)_2$, $Ni(EMG)_2$, $Pd(G)_2$ and $Pt(G)_2$], an examination of the C-N-O angles reveals an interesting trend. For the Ni, Pd, and Pt complexes these angles average 120.4 ± 0.2°, 122.0 ± 0.4° and 123.1 ± 1.06, respectively. The differences in the C-N-O angles for the pairs Ni-Pd, Ni-Pt, and Pd-Pt must be considered highly significant, significant, and not significant, respectively. The most logical explanation for this behavior is that oxygen-oxygen repulsions counteract, to a certain extent, the effect of decreasing size of the metal ion and result in a decrease in the C-N-O angle.

Another feature of the metal(II)-vic-dioximes is a delocalization of the C-N double bonds over the chelate rings. This effect has been

Table 16. Interatomic distances (Å) in some metal(II)-vic-dioximes

Compound	M-M[a]	M-N(1)[b]	M-N(2)	N(1)-O(1)
$Ni(G)_2$		1.868 (4)	1.880 (4)	1.343 (5)
$Ni(DMG)_2$	3.238 (1)	1.855 (6)	1.865 (7)	1.328 (9)
$Ni(EMG)_2$		1.861 (4)	1.862 (4)	1.350 (5)
$Pd(G)_2$		1.957 (7) 1.991 (8)	1.953 (9) 1.958 (7)	1.337 (12) 1.277 (13)
$Pd(DMG)_2$	3.26 (1)	1.99 (2)	1.93 (2)	1.33 (3)
$Pt(G)_2$		2.013 (14)	1.968 (14)	1.356 (21)
$Pt(DMG)_2$	3.23 (1)	1.95 (4)	1.93 (4)	1.24 (6)
$Cu(DMG)_2$	2.294 (3)	1.949 (3) 1.968 (3)	1.947 (3) 1.943 (3)	1.367 (4) 1.389 (4)
	2.301 (3)	1.946 (4) 1.968 (4)	1.950 (4) 1.946 (4)	1.373 (5) 1.385 (5)
$Cu(DMG)_2$-d_2	2.304 (7)	1.972 (9) 1.976 (9)	1.920 (11) 1.926 (11)	1.382 (10) 1.375 (11)

[a] For $Cu(DMG)_2$ and $Cu(DMG)_2$-d_2 the Cu-O(2') distance is given; all values in the table have units of Å.

[b] The lack of consistency in the literature with regard to labelling of atoms in metal(II)-vic-dioximes necessitated adoption of the scheme used in this work for $Ni(EMG)_2$.

Table 16. (Continued)

N(2)-O(2)	N(1)-C(1)	N(2)-C(2)	C(1)-C(2)	C(1)-C(3)
1.350 (5)	1.303 (6)	1.277 (7)	1.435 (7)	
1.354 (9)	1.302 (11)	1.287 (11)	1.443 (12)	1.507 (14)
1.344 (5)	1.295 (6)	1.301 (5)	1.462 (7)	1.499 (7)
1.384 (12) 1.372 (12)	1.321 (12) 1.319 (16)	1.273 (13) 1.271 (15)	1.471 (14) 1.492 (16)	
1.37 (3)	1.31 (4)	1.31 (4)	1.47 (4)	1.54 (5)
1.332 (22)	1.286 (28)	1.326 (30)	1.453 (31)	
1.31 (6)	1.38 (9)	1.27 (9)	1.57 (10)	1.47 (10)
1.353 (4) 1.333 (4)	1.289 (5) 1.283 (5)	1.294 (5) 1.299 (5)	1.481 (5) 1.486 (5)	1.483 (6) 1.494 (6)
1.345 (5) 1.331 (5)	1.284 (6) 1.278 (6)	1.294 (7) 1.304 (6)	1.495 (7) 1.487 (7)	1.488 (8) 1.491 (8)
1.378 (10) 1.326 (10)	1.265 (12) 1.296 (12)	1.314 (12) 1.305 (12)	1.508 (15) 1.445 (14)	1.493 (15) 1.513 (14)

Table 16. (Continued)

C(2)-C(4)	C(4)-C(5)	O(1)-O(2)	O(3)-O(4)	R(%)
		2.453 (6)		3.9
1.471 (13)		2.471 (9)		5.2
1.506 (7)	1.512 (8)	2.454 (5)		4.8
		2.599 (12) 2.659 (12)		4.6
1.55 (4)		2.59 (3)		7.
		2.655 (21)		4.3
1.46 (10)		3.03 (6)		7.
1.488 (6) 1.494 (5)		2.547 (4)	2.699 (4)	3.8
1.481 (7) 1.487 (7)		2.526 (5)	2.694 (5)	6.3
1.459 (13) 1.536 (15)		2.569 (11)	2.716 (11)	8.4

Table 16. (Continued)

Reference	Comments
18	
This work	
This work	
19	Molecule at 0, 0, 0 Molecule at $^1/_2$, $^1/_2$, $^1/_2$
90	
31	
34	
This work	Plane I[c] Plane II
81	Plane I Plane II
This work	Plane I Plane II

[c] Plane I contains the oxygen atom bound to the copper atom in the other half of the dimer.

observed for complexes of Ni(II), Pd(II), and Pt(II). In the case of Cu(DMG)$_2$ however, the amount of this delocalization is not as large as in other complexes for which the structures are well determined. This is apparent from an examination of the C-N and especially the C-C bond distances in the chelate rings of these complexes. A recent molecular orbital calculation (66) for Cu(DMG)$_2$, assuming a planar molecule, reveals a strong metal-ligand π bond and π delocalization over the entire ring. It seems reasonable, therefore, to conclude that, in solid Cu(DMG)$_2$, non-planarity resulting from distortions on dimerization may lead to a significant decrease in the π delocalization. The infrared spectra of Caton (20) reveal a shift in the C-C stretch in Cu(DMG)$_2$ on going from the solid to solution. The C-C stretching frequency shifts from 1544 cm^{-1} to 1555 cm^{-1}, suggesting that this bond is stronger in solution. Whether the increase in energy with greater delocalization is large is not certain, but some energy gain is certainly to be expected and would help to provide the energy necessary (69) for Cu(DMG)$_2$ to be as soluble as Ni(DMG)$_2$ in inert solvents.

VI. CHEMICAL REACTIVITY AND INFRARED SPECTRAL STUDIES OF SOME PALLADIUM(II)-*VIC*-DIOXIMES

Caton and Banks (21) interpreted the lack of reactivity of the hydroxyl hydrogen atoms of $Cu(DMG)_2$ in solution to mean that both of the hydrogen bonds in $Cu(DMG)_2$ rearrange on dissolution to become similar to those in $Ni(DMG)_2$ and $Ni(EMG)_2$. The implied, but unstated, assumption was that the hydroxyl hydrogen in the shorter hydrogen bond in solid $Cu(DMG)_2$ is reactive. The infrared spectra of $Cu(DMG)_2$ in the solid and in solution indicate that only the longer bond rearranges (21) so that both hydrogen bonds in solution are similar to the shorter bond in the solid. If this is indeed the case, then the hydroxyl hydrogen atom in the shorter hydrogen bond in solid $Cu(DMG)_2$ must necessarily be unreactive. Because the hydrogen bonds in $Pd(G)_2$ (19) and $Pd(DMG)_2$ (56, 90) were known to be only slightly longer than the shorter hydrogen bond in solid $Cu(DMG)_2$, the reactivities of the hydroxyl hydrogen atoms in several Pd(II)-*vic*-dioximes were studied in an attempt to show that only the longer bond in solid $Cu(DMG)_2$ is reactive. In order to confirm the similarity of the hydrogen bonds in the complexes studied, infrared spectra were obtained in the region from 4000 to 600 cm^{-1} and assignments were made for the OH stretching modes. The details and results of this study follow.

A. Apparatus and Materials

1. Instrumentation and apparatus

Infrared spectra in the region 4000-600 cm^{-1} were obtained on a Beckman IR-7 spectrophotometer.

2. Chemicals

Glyoxime was obtained from J. T. Baker as a 'Baker Grade' reagent. The commercial product was recrystallized from ethanol prior to use.

$Pd(DMG)_2$, $Pd(Niox)_2$, $Pd(Heptox)_2$, $Pd(\alpha\text{-Benzil})_2$, and $Pd(\alpha\text{-Furil})_2$ were prepared by R. P. Vander Wal (83) of this Laboratory according to the following procedures. $Pd(DMG)_2$ was prepared using the standard analytical procedure (28). $Pd(Niox)_2$ was prepared using the procedure of Voter, _et al_. (85). $Pd(Heptox)_2$ was prepared using the procedure of Voter and Banks (84). $Pd(\alpha\text{-Benzil})_2$ was prepared using the procedure of Banks, _et al_. (4). $Pd(\alpha\text{-Furil})_2$ was prepared using the procedure of Reed and Banks (64). All of the existing Pd(II) complexes had been recrystallized from 1,2-dichlorobenzene and did not require further treatment or purification.

B. Experimental Procedures

$Pd(G)_2$ was not available and therefore had to be synthesized prior to the reactivity and spectral studies. The following procedure was found to give good results. $PdCl_2$ was dissolved in the minimum necessary amount of 1:1 $HCl-H_2O$. To this solution, a stoichiometric amount of recrystallized glyoxime in ethanol solution was added. $Pd(G)_2$ was then precipitated by adjusting the acidity to a pH of 6 with NaOH. The finely divided amorphous precipitate was filtered and washed with 1:1 ethanol-water and then with ethanol and dried. The resulting dark brown solid was recrystallized from dilute acetic acid to give needle-shaped yellow crystals of very small cross section. The $Pd(G)_2$ thus prepared was used without further treatment in the spectral and reactivity studies.

1. Reactivity study

The reactivities of the six Pd(II) complexes were tested by placing approximately 20 mg. of the complexes in small test tubes and adding 20 drops of phenyl isocyanate or acetic anhydride. Any immediate reaction was noted and the test tubes were stoppered and allowed to stand for three days, at which time any subsequent reactions were noted.

2. Spectral studies

Infrared spectra in the region 4000-600 cm^{-1} were obtained by grinding small amounts of the complexes with KBr to a very fine particle size and pressing the mixture into a transparent disk. The resulting spectra contained a band near 1625 cm^{-1} due to water in the disk and spectra in the region 2000-1300 cm^{-1} were consequently obtained by mulling the complexes with halocarbon oil.

Due to an electronic imbalance and a contaminated atmosphere within the spectrophotometer, a CO_2 absorption band near 2370 cm^{-1} frequently appeared in the spectra as both positive and negative "peaks". Whenever possible, spectra were obtained which exhibited both additive and subtractive effects in order to aid in the location of absorption maxima. In such instances, the subtractive effect is indicated as a dotted line in the spectra.

C. Results

1. Reactivity study

The results of this study are summarized in Table 17. None of the six Pd(II) complexes studied was found to react with either acetic anhydrice or phenyl isocyanate. These reagents did dissolve significant amounts of the complexes but no decomposition of any of the complexes was noted, even on long standing.

Table 17. Results of reactivity studies of some Pd(II)-*vic*-dioximes

	Reactions[a]			
Complex	Acetic anhydride		Phenyl isocyanate	
	Immediately	On standing[b]	Immediately	On standing
$Pd(G)_2$	sol.[c]	sol.	sol.	sol.
$Pd(DMG)_2$	sl. sol.	v. sol.	sl. sol.	v. sol.
$Pd(Niox)_2$	sol.	v. sol.	sol.	v. sol.
$Pd(Heptox)_2$	sol.	v. sol.	sol.	v. sol.
$Pd(\alpha\text{-Benzil})_2$	sol.	sol.	sl. sol.	sol.
$Pd(\alpha\text{-Furil})_2$	sl. sol.	sl. sol.	sol.	sol.

[a] No decomposition of any of the complexes was observed, but both reagents dissolved significant amounts of nearly all of the complexes.

[b] Any reaction observed after three days standing.

[c] Sol. - soluble; sl. sol. - slightly soluble; v. sol. - very soluble.

2. Infrared spectral study

Infrared spectra of the six complexes studied are shown in Figures 17 through 22 (Appendix C). Frequency assignments were made for the principal absorptions in the spectra. The assignments were based on comparisons with the spectra reported in the literature (7, 8, 21) for several metal(II)-vic-dioximes and are given in Tables 18 through 23. The infrared spectra contain bands in the region 2300-2700 cm^{-1} which have been assigned to the OH stretching mode. These bands are very broad in all of the spectra. The frequencies reported are for absorption maxima.

For $Pd(G)_2$ two OH stretching frequencies were observed, one at 2510 cm^{-1} and the other at 2680 cm^{-1}. $Pd(G)_2$ contains two different hydrogen bonds, 2.599 Å and 2.659 Å in length, respectively. The observation of two OH stretching frequencies is consistent with the known structure. In addition, the frequencies observed for $Pd(G)_2$ agree well with those which are to be expected for O---O distances of 2.599 and 2.659 Å. The frequencies reported for the 2.547 and 2.699 Å hydrogen bonds in solid $Cu(DMG)_2$ were 2382 and 2650 cm^{-1}, respectively (21). For the remaining complexes studied the OH stretching frequencies occur in the region 2360-2410 cm^{-1}. Because of the interference of an atmospheric CO_2 absorption band which could not be balanced out of the spectra, the reported frequencies are not as precisely determined as those of the other assigned bands. The estimated error in the assignments of the OH stretching frequencies is still only on the order of 20 cm^{-1}, which is not enough to affect the conclusion that the hydrogen bonding in all of these complexes is quite similar, and that it is nearly as strong as in the shorter bond in solid $Cu(DMG)_2$.

Table 18. Infrared frequency assignments for $Pd(G)_2$

Frequency, cm^{-1}	Assignment
2680	OH stretch
2510	OH stretch
1571	CN stretch
1524	CC stretch
1348	CN stretch
1248	NO stretch
1110	NO stretch

Table 19. Infrared frequency assignments for $Pd(DMG)_2$

Frequency, cm^{-1}	Assignment
2390	OH stretch
1549	CN stretch
1507	CC stretch
1330	CN stretch
1254	NO stretch
1089	NO stretch

Table 20. Infrared frequency assignments for $Pd(Niox)_2$

Frequency, cm^{-1}	Assignment
2390	OH stretch
1547	CN stretch
1505	CC stretch
1357	CN stretch
1245	NO stretch
1086	NO stretch

Table 21. Infrared frequency assignments for $Pd(Heptox)_2$

Frequency, cm^{-1}	Assignment
2360	OH stretch
1547	CN stretch
1499	CC stretch
1340	CN stretch
1250	NO stretch
1076	NO stretch

Table 22. Infrared frequency assignments for $Pd(\alpha\text{-Benzil})_2$

Frequency, cm^{-1}	Assignment
2410	OH stretch
1579	CN stretch
1522	CC stretch
1328	CN stretch
1279	NO stretch
1076	NO stretch

Table 23. Infrared frequency assignments for $Pd(\alpha\text{-Furil})_2$

Frequency, cm^{-1}	Assignment
2370	OH stretch
1579	CN stretch
1513	CC stretch
1301	CN stretch
1227	NO stretch
1083	NO stretch

D. Discussion and Conclusions

The lack of reactivity of the hydroxyl hydrogen atoms in the Pd(II)-vic-dioximes coupled with the infrared spectral information confirms the presence of strong hydrogen bonds in these complexes. In addition, the data indicate that only the hydrogen atom in the longer hydrogen bond in solid $Cu(DMG)_2$ is reactive. This fact supports the conclusion, based on infrared spectral information, that only the longer hydrogen bond in solid $Cu(DMG)_2$ rearranges on dissolution.

VII. SUMMARY

The hydrogen bonding in the Ni(II)-, Pd(II)-, and, presumably, Pt(II)-vic-dioxime complexes in the solid state and in solution is apparently quite strong. The hydroxyl hydrogen atoms in the solid Ni(II)- and Pd(II)-vic-dioxime complexes are remarkably unreactive. The reactivity of these hydrogen atoms in Pt(II)-vic-dioximes has not been investigated, but structural studies, which reveal short hydrogen bonds in all of the complexes, show that the hydrogen bonds in the $Pt(G)_2$ complex are similar to those in the Pd(II)-vic-dioximes.

The hydrogen bonds in $Cu(DMG)_2$ in solution are also apparently quite strong, but not as strong as those in Ni(II)-vic-dioxime complexes. The infrared spectra of $Cu(DMG)_2$ in the solid and in solution suggest that only the longer hydrogen bond in the solid rearranges on dissolution. The lack of reactivity of the hydroxyl hydrogen atoms in the Pd(II)-vic-dioximes confirms the fact that this is possible, and also shows that the lack of reactivity of the hydroxyl hydrogen atoms of $Cu(DMG)_2$ in solution does not prove a similarity of the hydrogen bonds in the copper and nickel complexes in solution.

The hydrogen bonds in solid Ni(II)-vic-dioximes are apparently symmetrical. The symmetry of the nickel complexes supports this conclusion. The hydrogen bonds in solid $Cu(DMG)_2$ are apparently both asymmetrical. Differences in the N-O distances in the complex, as well as the refined positions of the hydroxyl hydrogen atoms support this conclusion. Expansion of the hydrogen bonds in Cu(DMG)$_2$ on deuteration is apparently slight and only marginally significant.

All of the metal(II)-vic-dioximes show signs of significant π delocalization in the chelate rings, a fact which helps to stabilize the complexes. This is indicated by C-N bond lengths which are longer than the normal double-bond length, and C-C bond lengths in the chelate rings which are shorter than the normal single-bond length.

The unusual specificity of the vic-dioximes for such metals as nickel and palladium is apparently the result of a combination of factors, the most important being a tendency toward square-planar coordination in the divalent state, and an ability to participate in metal-ligand π bonding. These factors combine to provide extremely stable square planar complexes with the vic-dioximes, complexes which are stabilized by significant metal-ligand π bonding, a delocalized π system over the chelate ring, and strong intramolecular hydrogen bonds.

VIII. SUGGESTIONS FOR FUTURE WORK

1) The vic-dioximes are able to form particularly strong complexes with transition metals in those instances when the vic-dioximes can assume a co-planar configuration. It seems reasonable that one might extend the utility of the vic-dioximes by developing extraction and/or spectrophotometric procedures for divalent metal ions which show tendencies toward coordination numbers higher than four. The use of an organic solution of a suitable vic-dioxime and some organic base, which would function as a neutral donor, would permit the formation and extraction of six-coordinate complexes with a square-planar metal(II)-vic-dioxime skeleton plus two axially coordinated neutral donor molecules. Appropriate selection of the vic-dioxime and neutral donor might also permit reasonably efficient separations to be performed rapidly and with ease.

2) The vic-dioxime complexes of the divalent metals provide a unique series of compounds in which it is possible to study relatively strong hydrogen bonds covering a reasonably wide range of O---O distances (2.45-2.70 Å). In particular, neutron diffraction studies of a few selected complexes could provide valuable information on short hydrogen bonds, including the nature of the potential well for the hydrogen atom. A careful study of normal and deuterated copper dimethylglyoxime could provide valuable information on the source of the deuterium isotope effect found in short hydrogen bonds, an effect which is not well understood.

3) Far infrared spectrophotometers are now available which permit the study of this region of the electromagnetic spectrum with a great deal of resolution and sensitivity. A careful and systematic study of the far infrared spectra of the metal(II)-vic-dioximes might provide considerable information on the hydrogen bonding in these complexes. Frequency assignments for the stretching and bending modes associated with the H•••O or hydrogen bond would be particularly valuable. By comparing the spectra of the complexes with different metals it should be possible to overcome the problem usually encountered in making such assignments, namely, the unusually small magnitude of deuteration shifts in these frequencies. In addition to information on hydrogen bonding, a careful study of this spectral region could also provide much information on the nature and strengths of the metal-nitrogen bonds in these complexes, as the metal-nitrogen stretching frequencies lie in the far infrared.

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X. ACKNOWLEDGMENTS

I am particularly indebted to the late Professor Charles V. Banks who, throughout this research, provided the suggestions and encouragement so necessary for the successful completion of this work. His untimely death saddened all of us who knew and worked with him and leaves a void which is only partially filled by the memory of many pleasant hours of conversation and discovery.

I also wish to express my thanks to the members of Analytical Chemistry Group I and X-Ray Chemistry Group I for their helpful discussions and assistance. I am particularly indebted to Dr. John E. Caton, Jr., and Dr. Marvin L. Hackert who provided much valuable assistance and advice, and to Dr. Robert F. Sieck, Dr. Charles J. Hensler, Dr. James W. Mitchell, James N. Story, and Charles A. Burgett who provided encouragement and advice and with whom I spent many pleasant hours.

A particular debt of gratitude is owed to Dr. Robert A. Jacobson for advice during the course of this research, and to he and Dr. Dennis C. Johnson for considerable advice and assistance following the death of Dr. Banks.

I would especially like to thank my wife, Judy, for encouragement and understanding, without which this work would not have been possible.

XI. APPENDIX A

Table 24. Final refined positional and thermal parameters for $Cu(DMG)_2$-d_2 with the significant figures of the standard deviations

Table 25. Final refined positional and thermal parameters for $Cu(DMG)_2$ with the significant figures of the standard deviations

Table 26. Final refined positional and thermal parameters for $Ni(EMG)_2$ with the significant figures of the standard deviations

Table 27. Final refined positional and thermal parameters for $Ni(DMG)_2$ with the significant figures of the standard deviations

Table 24. Final refined positional and thermal parameters for $Cu(DMG)_2$-d_2 with (below) the significant figures of the standard deviations

	X/A	Y/B	Z/C	B11	B22	B33	B12	B13	B23
CU	0.0268	0.0935	0.3650	0.0080	0.0030	0.0225	-0.0006	0.0065	-0.0003
	1	1	2	2	1	5	1	2	2
C(1)	0.2269	0.0135	0.2272	0.0190	0.0040	0.0254	0.0028	0.0057	0.0010
	16	10	18	26	8	36	12	23	15
C(2)	0.3639	-0.0074	0.1886	0.0171	0.0061	0.0475	0.0010	0.0187	-0.0032
	13	8	17	21	8	41	10	25	14
C(3)	0.1139	-0.0457	0.2297	0.0204	0.0029	0.0196	-0.0013	0.0103	-0.0008
	15	8	15	26	7	32	11	23	12
C(4)	0.1167	-0.1252	0.1585	0.0165	0.0038	0.0244	0.0022	0.0049	-0.0031
	12	8	15	20	6	31	9	20	13
N(1)	0.2035	0.0795	0.2906	0.0127	0.0045	0.0281	-0.0029	0.0096	0.0014
	10	8	13	16	6	28	9	17	11
N(2)	0.0140	-0.0149	0.2952	0.0133	0.0066	0.0244	-0.0000	0.0091	0.0008
	11	7	12	18	8	29	9	19	11
O(1)	0.2974	0.1407	0.3035	0.0154	0.0062	0.0509	-0.0019	0.0177	-0.0039
	9	6	12	14	5	29	7	17	10
O(2)	-0.0938	-0.0643	0.3071	0.0124	0.0056	0.0317	-0.0010	0.0076	-0.0003
	8	5	10	12	5	23	6	14	9
C(5)	-0.2126	0.1828	0.3438	0.0135	0.0048	0.0207	-0.0004	0.0020	-0.0002
	14	10	16	23	8	33	12	21	14
C(6)	-0.3583	0.2089	0.3505	0.0135	0.0058	0.0422	0.0016	0.0080	-0.0051
	13	7	16	21	7	41	10	23	15
C(7)	-0.0976	0.2369	0.3562	0.0161	0.0041	0.0167	-0.0011	-0.0039	0.0016
	17	8	14	26	8	30	12	23	13
C(8)	-0.1180	0.3257	0.3396	0.0262	0.0059	0.0391	0.0003	0.0196	0.0026
	14	9	19	29	8	40	12	26	16
N(3)	-0.1780	0.1105	0.3306	0.0115	0.0053	0.0229	0.0002	0.0048	0.0011
	10	9	12	17	6	27	9	16	12
N(4)	0.0259	0.2058	0.3683	0.0116	0.0063	0.0293	-0.0008	0.0051	0.0017
	14	7	13	17	7	29	9	18	11
O(3)	0.1379	0.2495	0.3706	0.0155	0.0049	0.0441	-0.0022	0.0134	-0.0035
	9	5	11	13	4	27	7	15	9
O(4)	-0.2758	0.0527	0.3258	0.0088	0.0069	0.0407	-0.0026	0.0067	0.0009
	8	5	10	12	5	26	6	14	9

Table 25. Final refined positional and thermal parameters for Cu(DMG)$_2$ with (below) the significant figures of the standard deviations

	X	Y	Z	B11	B22	B33	B12	B13	B23
CU	0.02769	0.09384	0.36725	0.00681	0.00204	0.02216	-0.00044	0.00578	-0.00022
	4	2	7	5	2	13	2	6	3
C(1)	0.22893	0.01497	0.23573	0.00865	0.00308	0.01819	0.00018	0.00596	0.00070
	38	23	57	41	13	87	19	50	28
C(2)	0.36153	-0.00505	0.18597	0.01164	0.00454	0.03747	0.00026	0.01287	-0.00078
	50	29	86	56	20	155	26	79	44
C(3)	0.11433	-0.04249	0.22738	0.00868	0.00253	0.01541	0.00017	0.00460	0.00053
	37	20	53	38	12	81	17	46	25
C(4)	0.11806	-0.12394	0.15646	0.01468	0.00270	0.02411	0.00073	0.00659	-0.00083
	49	24	69	58	13	110	23	66	31
N(1)	0.20297	0.08337	0.29268	0.00830	0.00284	0.02294	-0.00064	0.00655	0.00006
	32	18	50	33	11	80	15	43	24
N(2)	0.01280	-0.01583	0.29163	0.00761	0.00225	0.02095	-0.00027	0.00459	0.00030
	30	17	48	32	10	74	14	41	23
O(1)	0.29925	0.14251	0.30670	0.01077	0.00349	0.03530	-0.00201	0.01085	-0.00028
	31	17	51	34	11	93	16	47	26
O(2)	-0.09550	-0.06203	0.30670	0.00834	0.00271	0.02137	-0.00147	0.00452	-0.00051
	26	15	39	27	9	63	13	34	20
C(5)	-0.21280	0.18391	0.34700	0.00797	0.00298	0.01646	0.00070	0.00281	0.00051
	37	22	57	38	13	82	18	46	27
C(6)	-0.35621	0.20999	0.35610	0.00921	0.00416	0.02836	0.00168	0.00538	0.00081
	43	27	74	45	17	122	23	61	37
C(7)	-0.09554	0.24024	0.35700	0.01004	0.00271	0.01949	0.00034	0.00419	0.00058
	41	22	60	46	13	92	19	53	28
C(8)	-0.11867	0.32657	0.34294	0.01618	0.00235	0.03324	0.00124	0.00650	0.00061
	53	24	82	65	13	140	23	79	35
N(3)	-0.17577	0.11249	0.33719	0.00633	0.00288	0.02267	-0.00020	0.00414	0.00011
	30	18	50	29	11	81	14	40	24
N(4)	0.02666	0.20733	0.37188	0.00954	0.00234	0.02158	-0.00052	0.00505	0.00018
	32	17	49	34	10	78	16	43	23
O(3)	0.14090	0.25023	0.37508	0.01126	0.00253	0.03616	-0.00131	0.00898	0.00019
	31	15	53	35	9	93	14	47	23
O(4)	-0.27668	0.05433	0.32649	0.00735	0.00350	0.02777	-0.00090	0.00410	0.00079
	27	17	46	28	11	77	14	38	24

Table 26. Final refined positional and thermal parameters for Ni(EMG)$_2$ with (below) the significant figures of the standard deviations

	X	Y	Z	B11	B22	B33	B12	B13	B23
NI	0.00000	0.00000	0.00000	0.04101	0.00741	0.00532	-0.00205	0.00296	-0.00034
	0	0	0	48	8	7	17	12	6
N(1)	-0.02701	-0.15244	-0.04141	0.03311	0.00794	0.00672	-0.00127	0.00374	0.00040
	76	35	32	200	36	29	69	65	26
N(2)	0.26856	0.00248	-0.11016	0.05166	0.00790	0.00602	-0.00187	0.00296	0.00012
	89	36	30	228	35	28	89	66	30
O(1)	-0.20713	-0.22715	0.00387	0.06962	0.00723	0.00999	-0.00524	0.00760	-0.00032
	85	28	29	246	29	32	71	74	25
O(2)	0.41503	0.09580	-0.13782	0.06345	0.00947	0.00800	-0.00513	0.00817	-0.00032
	81	30	29	227	34	29	77	67	24
C(1)	0.13442	-0.18584	-0.12036	0.05236	0.00724	0.00664	0.00138	0.00017	-0.00013
	113	42	39	289	43	38	97	89	33
C(2)	0.31007	-0.09348	-0.16140	0.04260	0.00790	0.00579	0.00249	-0.00039	-0.00026
	107	42	38	259	45	36	91	79	33
C(3)	0.14082	-0.30575	-0.16311	0.07652	0.00762	0.01079	0.00182	0.00557	-0.00121
	133	45	47	394	47	52	117	120	40
C(4)	0.50873	-0.10602	-0.25632	0.05147	0.01039	0.00753	0.00221	0.00490	-0.00044
	122	44	43	287	52	42	106	89	37
C(5)	0.35938	-0.08927	-0.36816	0.09196	0.01585	0.00735	0.00561	0.00166	-0.00055
	154	57	48	465	77	47	158	122	46

Table 27. Final refined positional and thermal parameters for $Ni(DMG)_2$ with (below) the significant figures of the standard deviations

	X	Y	B11	B22	B33	B12
NI	0.00000	0.00000	0.00326	0.00726	0.01563	-0.00010
	0	0	7	18	40	13
N(1)	0.11175	-0.00746	0.00369	0.00834	0.01845	0.00021
	40	94	31	83	173	63
N(2)	0.01047	-0.17787	0.00358	0.00836	0.01701	0.00055
	53	68	36	81	169	53
O(1)	0.15989	0.09416	0.00384	0.00886	0.02964	-0.00110
	43	68	30	79	227	45
O(2)	-0.05313	-0.25920	0.00458	0.00970	0.03068	-0.00106
	41	72	31	83	224	49
C(1)	0.14206	-0.12243	0.00361	0.00980	0.01457	-0.00014
	61	99	46	134	254	63
C(2)	0.08262	-0.22335	0.00375	0.00831	0.01661	0.00106
	59	94	41	112	249	63
C(3)	0.23194	-0.14364	0.00431	0.01335	0.02840	0.00023
	62	102	50	145	320	70
C(4)	0.10118	-0.36115	0.00566	0.00829	0.03627	-0.00004
	67	97	56	113	379	67

XII. APPENDIX B

Table 28. Final observed and calculated structure amplitudes in electrons for $Cu(DMG)_2$-d_2

Table 29. Final observed and calculated structure amplitudes in electrons for $Cu(DMG)_2$

Table 30. Final observed and calculated structure amplitudes in electrons for $Ni(EMG)_2$

Table 31. Final observed and calculated structure amplitudes in electrons for $Ni(DMG)_2$

Table 28. Final observed and calculated structure amplitudes in electrons for Cu(DMG)$_2$-d$_2$

H	K	L	FO	FC
2	0	0	34	29
4	0	0	31	31
6	0	0	29	32
8	0	0	20	17
1	1	0	150	147
2	1	0	6	6
3	1	0	71	70
4	1	0	38	38
5	1	0	39	37
6	1	0	19	19
7	1	0	15	13
8	1	0	30	29
0	2	0	64	60
1	2	0	5	2
2	2	0	13	11
3	2	0	38	36
4	2	0	52	52
5	2	0	57	56
6	2	0	6	2
7	2	0	37	38
8	2	0	7	3
1	3	0	2	2
2	3	0	4	1
3	3	0	12	12
4	3	0	52	53
5	3	0	15	11
6	3	0	57	57
8	3	0	9	8
-1	4	0	10	10
0	4	0	21	17
1	4	0	10	10
2	4	0	24	22
3	4	0	33	32
4	4	0	44	42
5	4	0	29	27
6	4	0	21	22
7	4	0	20	18
8	4	0	15	12
-2	5	0	79	81
-1	5	0	91	92
1	5	0	95	92
2	5	0	78	81
3	5	0	10	9
4	5	0	33	30
5	5	0	10	10
6	5	0	21	20
7	5	0	17	21
-2	6	0	64	61
-1	6	0	24	25
0	6	0	74	75
1	6	0	20	25
2	6	0	57	61
3	6	0	6	7
4	6	0	15	17
5	6	0	14	12
6	6	0	17	17
7	6	0	9	8
-2	7	0	20	20
-1	7	0	64	62
1	7	0	58	62
2	7	0	21	20
3	7	0	22	21
4	7	0	27	28
5	7	0	10	13
6	7	0	23	22
8	7	0	23	24
-3	8	0	33	30
-1	8	0	3	3
0	8	0	8	9
3	8	0	30	30
4	8	0	13	13
5	8	0	46	42
6	8	0	17	16
7	8	0	30	30
-3	9	0	28	27
1	9	0	44	42
2	9	0	34	35
3	9	0	25	27
4	9	0	30	31
5	9	0	36	34
6	9	0	29	32
7	9	0	6	5
-4	10	0	42	42
-2	10	0	51	50
0	10	0	39	38
1	10	0	23	22
2	10	0	47	50
3	10	0	35	37
4	10	0	45	42
5	10	0	5	4
6	10	0	29	29
1	11	0	37	38
2	11	0	14	10
3	11	0	31	31
4	11	0	10	8
5	11	0	48	47
6	11	0	8	3
-4	12	0	19	16
-3	12	0	9	14
-2	12	0	23	22
0	12	0	33	32
2	12	0	24	22
3	12	0	11	14
4	12	0	13	16
5	12	0	15	16
1	13	0	4	5
2	13	0	5	12
3	13	0	11	10
4	13	0	10	12
-2	14	0	11	9
0	14	0	11	11
1	14	0	19	17
2	14	0	9	9
3	14	0	7	5
4	14	0	7	1
-3	15	0	14	13
1	15	0	22	20
2	15	0	15	15
3	15	0	12	13
0	16	0	15	12
1	16	0	15	13
-9	0	1	30	31
-7	0	1	25	26
-5	0	1	7	7
-3	0	1	20	20
-1	0	1	88	88
1	0	1	137	141
3	0	1	66	66
5	0	1	56	53
7	0	1	47	45
-9	1	1	13	13
-8	1	1	19	19
-7	1	1	33	32
-6	1	1	27	26
-5	1	1	50	46

Table 28. (Continued)

H	K	L	FO	FC
-4	1	1	29	27
-3	1	1	56	51
-2	1	1	14	12
-1	1	1	67	60
0	1	1	87	86
1	1	1	67	65
2	1	1	53	52
3	1	1	8	5
4	1	1	67	64
5	1	1	5	7
6	1	1	48	47
7	1	1	6	7
8	1	1	15	15
-9	2	1	12	13
-8	2	1	15	13
-7	2	1	4	4
-6	2	1	62	57
-5	2	1	3	3
-4	2	1	139	136
-3	2	1	8	5
-2	2	1	29	26
-1	2	1	35	39
0	2	1	115	108
1	2	1	51	50
2	2	1	47	48
3	2	1	40	40
4	2	1	35	35
5	2	1	13	13
6	2	1	12	10
7	2	1	8	13
8	2	1	19	20
-8	3	1	18	18
-7	3	1	34	35
-6	3	1	40	41
-5	3	1	84	83
-4	3	1	31	30
-2	3	1	28	31
-1	3	1	24	22
0	3	1	33	32
1	3	1	4	9
2	3	1	19	19
3	3	1	9	11
4	3	1	44	39
5	3	1	22	20
6	3	1	11	10
-8	4	1	7	3
-7	4	1	15	14
-6	4	1	44	42
-5	4	1	3	3
-3	4	1	30	30
-2	4	1	11	7
-1	4	1	12	11
0	4	1	17	12
1	4	1	10	7
2	4	1	9	12
3	4	1	39	39
4	4	1	29	29
5	4	1	44	45
6	4	1	11	10
7	4	1	12	12
-8	5	1	14	16
-7	5	1	9	10
-6	5	1	12	10
-5	5	1	8	9
-4	5	1	7	4
-3	5	1	18	16
-2	5	1	26	26
-1	5	1	8	8
0	5	1	43	40
1	5	1	4	4
2	5	1	36	35
3	5	1	16	15
4	5	1	39	42
5	5	1	10	10
6	5	1	35	32
7	5	1	4	3
-8	6	1	6	1
-7	6	1	20	18
-6	6	1	15	19
-4	6	1	11	11
-3	6	1	11	11
-2	6	1	24	23
-1	6	1	59	58
0	6	1	21	17
1	6	1	51	48
2	6	1	34	35
3	6	1	42	38
4	6	1	17	16
5	6	1	24	25
6	6	1	5	11
-8	7	1	11	11
-7	7	1	23	25
-6	7	1	25	23
-5	7	1	42	38
-4	7	1	29	27
-3	7	1	36	37
-2	7	1	6	6
-1	7	1	90	89
0	7	1	65	63
1	7	1	39	39
2	7	1	38	41
3	7	1	12	10
4	7	1	3	3
5	7	1	7	2
6	7	1	23	26
7	7	1	4	2
-8	8	1	25	26
-7	8	1	17	17
-6	8	1	60	55
-4	8	1	38	31
-3	8	1	12	11
-2	8	1	87	79
-1	8	1	47	39
0	8	1	86	83
1	8	1	38	37
2	8	1	46	45
3	8	1	6	4
5	8	1	4	3
6	8	1	4	2
-3	9	1	49	44
0	9	1	26	25
1	9	1	50	48
2	9	1	25	25
3	9	1	11	13
4	9	1	21	27
5	9	1	9	7
6	9	1	23	23
0	10	1	7	11
1	10	1	28	32
2	10	1	5	3
3	10	1	39	41
4	10	1	6	6
5	10	1	46	49
0	11	1	24	26
1	11	1	5	8

Table 28. (Continued)

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
2	11	1	35	36	2	2	2	3	4	3	5	2	23	23
3	11	1	4	4	3	2	2	47	47	4	5	2	9	7
4	11	1	34	33	4	2	2	7	2	5	5	2	29	29
-4	12	1	22	24	5	2	2	41	41	6	5	2	10	9
0	12	1	21	21	6	2	2	20	20	7	5	2	16	14
1	12	1	37	37	7	2	2	16	14	-8	6	2	21	19
2	12	1	15	14	-8	3	2	10	4	-7	6	2	9	8
5	12	1	12	13	-7	3	2	8	8	-6	6	2	36	42
1	13	1	33	36	-6	3	2	16	15	-4	6	2	6	5
0	14	1	26	26	-5	3	2	8	7	-2	6	2	35	35
0	15	1	16	13	-4	3	2	75	75	-1	6	2	22	22
-8	0	2	64	61	-2	3	2	25	25	1	6	2	44	39
-6	0	2	40	49	-1	3	2	6	5	2	6	2	21	17
-4	0	2	47	47	0	3	2	58	58	4	6	2	16	13
-2	0	2	20	22	1	3	2	28	29	5	6	2	8	6
0	0	2	174	180	2	3	2	32	33	6	6	2	32	29
2	0	2	27	26	4	3	2	59	58	-8	7	2	4	1
4	0	2	26	26	5	3	2	12	14	-7	7	2	16	19
6	0	2	35	34	6	3	2	25	30	-6	7	2	28	28
-9	1	2	28	30	-8	4	2	4	3	-5	7	2	20	18
-8	1	2	7	8	-7	4	2	19	19	-4	7	2	7	8
-7	1	2	53	51	-6	4	2	40	42	-3	7	2	8	3
-6	1	2	30	28	-5	4	2	10	9	-2	7	2	45	49
-5	1	2	61	55	-3	4	2	19	20	-1	7	2	7	7
-4	1	2	4	4	-2	4	2	53	52	1	7	2	12	14
-3	1	2	33	32	-1	4	2	38	39	2	7	2	48	49
-2	1	2	36	36	0	4	2	9	8	3	7	2	20	19
-1	1	2	12	11	1	4	2	39	40	4	7	2	17	16
0	1	2	180	187	2	4	2	16	16	5	7	2	18	17
1	1	2	75	76	3	4	2	26	26	6	7	2	18	18
2	1	2	12	14	4	4	2	38	36	-7	8	2	9	7
3	1	2	17	17	5	4	2	18	16	-5	8	2	42	38
4	1	2	15	18	6	4	2	16	15	-4	8	2	12	11
5	1	2	26	27	7	4	2	14	14	-3	8	2	39	34
6	1	2	18	18	-8	5	2	16	18	-2	8	2	6	1
7	1	2	25	28	-7	5	2	34	35	-1	8	2	83	75
-8	2	2	11	8	-6	5	2	8	8	1	8	2	65	67
-7	2	2	14	14	-5	5	2	22	26	2	8	2	6	5
-6	2	2	32	30	-4	5	2	28	27	3	8	2	41	43
-5	2	2	38	37	-3	5	2	4	4	4	8	2	5	1
-4	2	2	22	22	-2	5	2	15	22	5	8	2	32	32
-3	2	2	67	68	-1	5	2	31	36	6	8	2	13	12
-1	2	2	70	72	0	5	2	15	15	-6	9	2	14	11
0	2	2	65	67	1	5	2	8	7	-3	9	2	7	10
1	2	2	81	84	2	5	2	25	24	2	9	2	51	48

Table 28. (Continued)

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
3	9	2	4	3	-2	1	3	37	37	-1	4	3	13	13
4	9	2	31	38	-1	1	3	47	47	0	4	3	21	22
5	9	2	11	9	0	1	3	93	94	1	4	3	7	5
6	9	2	19	18	1	1	3	35	35	2	4	3	47	48
-6	10	2	39	35	2	1	3	18	17	3	4	3	7	8
-5	10	2	26	26	3	1	3	29	28	4	4	3	12	11
-4	10	2	38	44	4	1	3	22	19	6	4	3	19	20
-2	10	2	24	26	5	1	3	21	18	-8	5	3	11	11
0	10	2	6	1	-9	2	3	8	5	-7	5	3	12	7
1	10	2	9	14	-8	2	3	14	11	-6	5	3	42	41
2	10	2	11	11	-7	2	3	18	19	-5	5	3	7	8
3	10	2	23	25	-6	2	3	7	6	-4	5	3	44	44
4	10	2	21	20	-5	2	3	18	21	-3	5	3	19	18
5	10	2	7	14	-4	2	3	39	42	-2	5	3	32	34
1	11	2	10	7	-3	2	3	30	30	-1	5	3	22	25
3	11	2	19	24	-2	2	3	18	18	0	5	3	47	49
4	11	2	4	3	-1	2	3	3	3	1	5	3	25	25
-6	12	2	17	16	0	2	3	34	33	2	5	3	12	11
-4	12	2	18	17	1	2	3	28	28	3	5	3	22	16
-3	12	2	18	13	2	2	3	68	69	4	5	3	5	3
-2	12	2	15	16	3	2	3	13	12	5	5	3	7	8
0	12	2	4	4	4	2	3	43	42	-7	6	3	17	18
1	12	2	22	23	5	2	3	10	2	-5	6	3	39	36
2	12	2	12	12	6	2	3	17	17	-4	6	3	4	4
3	12	2	9	13	-9	3	3	17	13	-3	6	3	41	43
4	12	2	4	3	-8	3	3	6	4	-2	6	3	12	12
0	13	2	20	31	-7	3	3	9	15	-1	6	3	64	68
-2	14	2	4	3	-6	3	3	32	32	0	6	3	14	15
0	14	2	6	2	-5	3	3	19	20	1	6	3	32	27
0	15	2	4	4	-4	3	3	8	8	2	6	3	12	9
-9	0	3	11	21	-3	3	3	11	9	3	6	3	4	6
-7	0	3	50	51	-1	3	3	14	14	4	6	3	24	24
-5	0	3	46	50	0	3	3	28	28	5	6	3	9	4
-3	0	3	83	83	1	3	3	42	44	-8	7	3	21	22
-1	0	3	84	85	2	3	3	19	20	-7	7	3	7	6
1	0	3	113	113	3	3	3	60	60	-6	7	3	17	11
3	0	3	8	11	4	3	3	5	6	-5	7	3	14	13
5	0	3	6	7	5	3	3	21	25	-4	7	3	30	26
-9	1	3	12	12	-8	4	3	10	8	-3	7	3	10	8
-8	1	3	18	15	-7	4	3	12	9	-2	7	3	20	23
-7	1	3	16	15	-6	4	3	33	34	-1	7	3	4	4
-6	1	3	34	34	-5	4	3	55	51	0	7	3	58	56
-5	1	3	14	12	-4	4	3	8	7	1	7	3	32	35
-4	1	3	98	96	-3	4	3	47	47	2	7	3	11	13
-3	1	3	6	6	-2	4	3	15	14	3	7	3	14	17

Table 28. (Continued)

H	K	L	FO	FC
4	7	3	8	6
5	7	3	19	20
-7	8	3	12	11
-6	8	3	29	26
-5	8	3	9	8
-4	8	3	7	11
-3	8	3	6	2
-2	8	3	9	9
-1	8	3	5	1
0	8	3	26	27
2	8	3	25	27
4	8	3	29	28
5	8	3	10	6
-6	9	3	28	27
-3	9	3	11	15
0	9	3	10	19
2	9	3	19	19
3	9	3	34	31
4	9	3	7	4
5	9	3	25	25
-5	10	3	61	51
0	10	3	4	5
1	10	3	19	28
2	10	3	8	15
3	10	3	6	9
4	10	3	11	9
0	11	3	23	24
2	11	3	17	17
3	11	3	14	13
-3	12	3	8	13
0	12	3	7	10
1	12	3	20	27
2	12	3	12	11
3	12	3	7	6
-8	0	4	8	7
-6	0	4	11	14
-4	0	4	16	17
-2	0	4	72	76
0	0	4	73	76
2	0	4	28	27
4	0	4	20	18
-8	1	4	24	28
-7	1	4	8	9
-6	1	4	25	26
-5	1	4	13	13
-4	1	4	23	21
-3	1	4	49	48
-1	1	4	57	56
0	1	4	40	40
1	1	4	13	13
2	1	4	18	12
3	1	4	26	25
4	1	4	12	9
5	1	4	17	17
-8	2	4	9	9
-7	2	4	37	39
-6	2	4	6	8
-5	2	4	47	47
-4	2	4	28	30
-3	2	4	42	41
-2	2	4	11	10
0	2	4	26	26
1	2	4	16	17
2	2	4	12	13
3	2	4	22	19
4	2	4	11	10
-8	3	4	16	18
-6	3	4	49	49
-5	3	4	16	19
-4	3	4	59	59
-3	3	4	10	10
-2	3	4	7	8
-1	3	4	12	13
0	3	4	20	19
1	3	4	29	29
2	3	4	14	15
3	3	4	4	3
4	3	4	13	11
5	3	4	11	11
-7	4	4	26	27
-6	4	4	11	7
-5	4	4	25	23
-4	4	4	13	19
-3	4	4	23	19
-2	4	4	44	45
-1	4	4	8	10
0	4	4	29	31
1	4	4	3	3
2	4	4	13	13
3	4	4	14	13
4	4	4	26	25
5	4	4	4	5
-8	5	4	16	13
-7	5	4	13	13
-6	5	4	7	2
-5	5	4	14	15
-4	5	4	8	11
-3	5	4	38	39
-2	5	4	14	12
-1	5	4	35	35
0	5	4	5	7
1	5	4	45	46
2	5	4	13	13
3	5	4	16	17
5	5	4	16	15
-8	6	4	17	11
-7	6	4	10	14
-6	6	4	4	1
-5	6	4	12	12
-4	6	4	38	39
-3	6	4	6	7
-2	6	4	10	37
-1	6	4	16	17
0	6	4	41	46
1	6	4	10	10
2	6	4	15	29
3	6	4	5	1
4	6	4	6	9
-7	7	4	10	3
-6	7	4	35	34
-5	7	4	19	17
-4	7	4	17	20
-3	7	4	33	35
-2	7	4	16	17
-1	7	4	28	29
0	7	4	15	14
1	7	4	17	19
2	7	4	5	7
3	7	4	17	18
4	7	4	6	7
-7	8	4	29	27
-6	8	4	9	14
-5	8	4	59	54
-4	8	4	34	31
-3	8	4	12	12

Table 28. (Continued)

H	K	L	FO	FC
-2	8	4	7	1
-1	8	4	20	16
0	8	4	10	5
1	8	4	10	11
2	8	4	8	1
-6	9	4	28	29
-3	9	4	12	12
1	9	4	21	21
3	9	4	9	9
-6	10	4	15	14
-5	10	4	23	23
-4	10	4	6	12
-2	10	4	12	19
0	10	4	30	29
1	10	4	8	4
2	10	4	33	28
0	11	4	4	4
1	11	4	15	25
2	11	4	6	3
-4	12	4	6	5
-3	12	4	13	8
-2	12	4	6	11
0	12	4	15	16
1	12	4	6	5
-7	0	5	33	31
-5	0	5	11	11
-3	0	5	15	11
-1	0	5	32	28
1	0	5	8	5
3	0	5	24	22
-8	1	5	11	12
-7	1	5	16	18
-6	1	5	13	16
-5	1	5	19	19
-4	1	5	28	27
-3	1	5	23	20
-2	1	5	13	13
-1	1	5	31	32
1	1	5	27	26
2	1	5	15	13
3	1	5	10	7
4	1	5	13	14
-8	2	5	21	20
-7	2	5	13	15
-6	2	5	39	40

H	K	L	FO	FC
-5	2	5	4	4
-4	2	5	40	37
-3	2	5	10	7
-2	2	5	25	27
-1	2	5	5	6
0	2	5	40	38
1	2	5	6	7
2	2	5	16	16
3	2	5	7	9
4	2	5	14	13
-7	3	5	27	30
-6	3	5	6	7
-5	3	5	39	39
-4	3	5	4	4
-3	3	5	31	32
-1	3	5	16	17
1	3	5	23	22
2	3	5	16	17
3	3	5	17	18
-6	4	5	14	11
-5	4	5	15	12
-4	4	5	20	21
-3	4	5	7	5
-2	4	5	14	14
-1	4	5	10	6
0	4	5	18	17
1	4	5	17	15
2	4	5	20	20
3	4	5	20	22
-7	5	5	18	16
-6	5	5	18	15
-2	5	5	13	16
-1	5	5	15	15
0	5	5	8	1
1	5	5	10	10
2	5	5	22	23
3	5	5	8	7
-7	6	5	7	1
-6	6	5	6	14
-5	6	5	14	14
-4	6	5	9	11
-3	6	5	9	8
-2	6	5	18	15
-1	6	5	8	9
0	6	5	7	3

H	K	L	FO	FC
1	6	5	21	19
-6	7	5	8	6
-5	7	5	14	17
-4	7	5	9	7
-3	7	5	30	31
-2	7	5	12	13
-1	7	5	20	23
0	7	5	4	4
1	7	5	17	16
2	7	5	15	12
-6	8	5	21	19
-4	8	5	32	33
-3	8	5	8	11
-2	8	5	32	30
-1	8	5	4	8
0	8	5	29	28
1	8	5	8	8
2	8	5	8	14
-3	9	5	28	29
0	9	5	14	9
1	9	5	14	14
0	10	5	6	5
-6	0	6	21	25
-4	0	6	22	22
-2	0	6	18	18
0	0	6	31	28
2	0	6	12	3
-5	1	6	21	21
-3	1	6	29	27
-2	1	6	26	25
-1	1	6	15	15
0	1	6	22	22
-6	2	6	11	8
-5	2	6	9	9
-4	2	6	17	17
-3	2	6	17	14
-2	2	6	9	8
-1	2	6	33	31
1	2	6	17	15
-6	3	6	7	1
-5	3	6	11	12
-4	3	6	12	10
-2	3	6	25	24
-1	3	6	4	2
0	3	6	13	12

Table 28. (Continued)

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
1	3	6	6	11	-4	5	6	9	5	-2	6	6	14	14
-6	4	6	16	21	-3	5	6	17	15	-1	6	6	7	3
-4	4	6	13	18	-1	5	6	15	12	0	6	6	8	8
-3	4	6	11	9	0	5	6	8	8	-4	7	6	4	1
-2	4	6	8	5	1	5	6	10	9	-3	7	6	18	16
-1	4	6	13	12	-5	6	6	5	4	-2	7	6	20	19
0	4	6	8	11	-4	6	6	27	26	-1	7	6	4	4
-5	5	6	18	18	-3	6	6	11	9	-3	8	6	21	22

Table 29. Final observed and calculated structure amplitudes in electrons for Cu(DMG)$_2$

H	K	L	FO	FC
-12	0	0	13	14
-10	0	0	9	8
-8	0	0	21	21
-6	0	0	37	37
-4	0	0	34	36
-2	0	0	36	35
2	0	0	36	35
4	0	0	33	36
6	0	0	36	37
8	0	0	21	21
10	0	0	8	8
12	0	0	13	14
-12	1	0	0	4
-11	1	0	0	2
-10	1	0	20	20
-9	1	0	9	8
-8	1	0	38	38
-7	1	0	14	14
-6	1	0	25	24
-5	1	0	43	41
-4	1	0	43	40
-3	1	0	83	78
-2	1	0	8	7
-1	1	0	152	154
0	1	0	0	0
1	1	0	147	154
2	1	0	8	7
3	1	0	79	78
4	1	0	42	40
5	1	0	42	41
6	1	0	24	24
7	1	0	14	14
8	1	0	37	38
9	1	0	9	8
10	1	0	19	20
11	1	0	4	2
12	1	0	5	4
-12	2	0	0	2
-11	2	0	23	22
-10	2	0	5	4
-9	2	0	25	25
-8	2	0	8	7
-7	2	0	47	47
-6	2	0	0	2
-5	2	0	69	66
-4	2	0	60	55
-3	2	0	46	44
-2	2	0	18	16
-1	2	0	6	6
0	2	0	64	62
1	2	0	6	6
2	2	0	18	16
3	2	0	44	44
4	2	0	56	55
5	2	0	65	66
6	2	0	0	2
7	2	0	46	47
8	2	0	7	7
9	2	0	24	25
10	2	0	6	4
11	2	0	21	22
12	2	0	3	2
-12	3	0	14	13
-11	3	0	0	3
-10	3	0	26	26
-9	3	0	6	7
-8	3	0	10	9
-7	3	0	4	2
-6	3	0	70	70
-5	3	0	16	15
-4	3	0	64	61
-3	3	0	15	14
-2	3	0	3	3
-1	3	0	2	2
0	3	0	2	0
1	3	0	2	2
2	3	0	3	3
3	3	0	14	14
4	3	0	60	61
5	3	0	15	15
6	3	0	68	70
7	3	0	1	2
8	3	0	10	9
9	3	0	7	7
10	3	0	24	25
12	3	0	11	13
-12	4	0	5	6
-11	4	0	12	12
-10	4	0	5	4
-9	4	0	13	13
-8	4	0	18	18
-7	4	0	27	26
-6	4	0	24	24
-5	4	0	32	32
-4	4	0	51	48
-3	4	0	36	36
-2	4	0	28	27
-1	4	0	10	10
0	4	0	25	23
1	4	0	11	10
2	4	0	27	27
3	4	0	36	36
4	4	0	48	48
5	4	0	30	32
6	4	0	22	24
7	4	0	24	26
8	4	0	18	18
9	4	0	12	13
10	4	0	4	4
11	4	0	13	12
12	4	0	5	6
-12	5	0	0	2
-11	5	0	6	5
-10	5	0	11	10
-9	5	0	8	9
-8	5	0	5	3
-7	5	0	24	25
-6	5	0	25	25
-5	5	0	14	14
-4	5	0	40	35
-3	5	0	9	11
-2	5	0	96	88
-1	5	0	106	103
0	5	0	0	0
1	5	0	106	103
2	5	0	95	88
3	5	0	8	11
4	5	0	39	35
5	5	0	12	14
6	5	0	24	25
7	5	0	24	25
8	5	0	3	3
9	5	0	7	9
10	5	0	9	10
11	5	0	6	5

Table 29. (Continued)

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
12	5	0	2	2	10	7	0	24	25	9	9	0	5	4
-11	6	0	12	11	11	7	0	5	1	10	9	0	17	17
-10	6	0	0	3	-11	8	0	21	22	11	9	0	0	2
-9	6	0	11	12	-10	8	0	4	3	-11	10	0	5	7
-8	6	0	4	3	-9	8	0	31	31	-10	10	0	7	7
-7	6	0	12	12	-8	8	0	0	3	-9	10	0	11	0
-6	6	0	20	20	-7	8	0	43	42	-8	10	0	2	3
-5	6	0	16	17	-6	8	0	24	23	-7	10	0	8	7
-4	6	0	19	21	-5	8	0	57	57	-6	10	0	40	39
-3	6	0	11	10	-4	8	0	14	13	-5	10	0	0	2
-2	6	0	73	72	-3	8	0	37	36	-4	10	0	58	58
-1	6	0	29	27	-2	8	0	5	3	-3	10	0	45	45
0	6	0	85	84	-1	8	0	0	3	-2	10	0	66	66
1	6	0	30	27	0	8	0	13	13	-1	10	0	27	25
2	6	0	73	72	1	8	0	3	3	0	10	0	47	49
3	6	0	11	10	2	8	0	3	3	1	10	0	26	25
4	6	0	18	21	3	8	0	36	36	2	10	0	65	66
5	6	0	15	17	4	8	0	13	13	3	10	0	44	45
6	6	0	19	20	5	8	0	55	57	4	10	0	56	58
7	6	0	11	12	6	8	0	23	23	5	10	0	0	2
8	6	0	4	3	7	8	0	40	42	6	10	0	39	39
9	6	0	12	12	8	8	0	5	3	7	10	0	8	7
10	6	0	1	3	9	8	0	29	31	8	10	0	2	3
11	6	0	11	11	10	8	0	0	3	9	10	0	10	9
-11	7	0	0	1	11	8	0	20	22	10	10	0	10	7
-10	7	0	25	25	-11	9	0	5	2	11	10	0	8	7
-9	7	0	0	1	-10	9	0	18	17	-10	11	0	8	7
-8	7	0	32	33	-9	9	0	6	4	-9	11	0	3	1
-7	7	0	4	3	-8	9	0	23	23	-8	11	0	6	6
-6	7	0	32	30	-7	9	0	4	5	-7	11	0	5	5
-5	7	0	14	14	-6	9	0	44	43	-6	11	0	9	8
-4	7	0	33	32	-5	9	0	47	46	-5	11	0	64	64
-3	7	0	25	25	-4	9	0	41	40	-4	11	0	15	14
-2	7	0	27	25	-3	9	0	35	34	-3	11	0	41	41
-1	7	0	76	71	-2	9	0	46	45	-2	11	0	15	14
0	7	0	7	0	-1	9	0	59	56	-1	11	0	48	49
1	7	0	75	71	0	9	0	9	0	0	11	0	0	0
2	7	0	27	25	1	9	0	58	56	1	11	0	48	49
3	7	0	25	25	2	9	0	45	45	2	11	0	14	14
4	7	0	32	32	3	9	0	34	34	3	11	0	41	41
5	7	0	15	14	4	9	0	39	40	4	11	0	14	14
6	7	0	28	30	5	9	0	45	46	5	11	0	62	64
7	7	0	3	3	6	9	0	42	43	6	11	0	9	8
8	7	0	32	33	7	9	0	4	5	7	11	0	2	5
9	7	0	3	1	8	9	0	21	23	8	11	0	5	6

Table 29. (Continued)

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
9	11	0	4	1	-8	14	0	5	6	0	16	0	20	18
10	11	0	9	7	-7	14	0	19	19	1	16	0	17	18
-10	12	0	3	2	-6	14	0	7	7	2	16	0	35	37
-9	12	0	20	19	-5	14	0	23	23	3	16	0	7	7
-8	12	0	0	1	-4	14	0	2	2	4	16	0	34	33
-7	12	0	12	12	-3	14	0	4	3	5	16	0	8	8
-6	12	0	9	9	-2	14	0	13	12	6	16	0	15	16
-5	12	0	26	25	-1	14	0	26	26	7	16	0	2	2
-4	12	0	21	21	0	14	0	18	18	8	16	0	0	1
-3	12	0	13	12	1	14	0	26	26	-8	17	0	10	10
-2	12	0	29	29	2	14	0	13	12	-7	17	0	7	6
-1	12	0	4	3	3	14	0	5	3	-6	17	0	14	15
0	12	0	43	42	4	14	0	4	2	-5	17	0	17	17
1	12	0	0	3	5	14	0	22	23	-4	17	0	10	10
2	12	0	30	29	6	14	0	8	7	-3	17	0	27	28
3	12	0	12	12	7	14	0	19	19	-2	17	0	4	1
4	12	0	20	21	8	14	0	6	6	-1	17	0	24	25
5	12	0	24	25	9	14	0	14	15	0	17	0	1	0
6	12	0	9	9	-9	15	0	0	1	1	17	0	24	25
7	12	0	11	12	-8	15	0	9	9	2	17	0	0	1
8	12	0	6	1	-7	15	0	10	9	3	17	0	27	28
9	12	0	20	19	-6	15	0	14	14	4	17	0	11	10
10	12	0	2	2	-5	15	0	21	22	5	17	0	18	17
-10	13	0	10	13	-4	15	0	9	9	6	17	0	15	15
-9	13	0	0	1	-3	15	0	16	17	7	17	0	7	6
-8	13	0	25	24	-2	15	0	18	18	8	17	0	10	10
-7	13	0	6	5	-1	15	0	29	29	-7	18	0	19	19
-6	13	0	23	24	0	15	0	0	0	-6	18	0	6	4
-5	13	0	3	2	1	15	0	30	29	-5	18	0	19	18
-4	13	0	13	12	2	15	0	17	18	-4	18	0	10	9
-3	13	0	18	18	3	15	0	16	17	-3	18	0	12	12
-2	13	0	10	10	4	15	0	7	9	-2	18	0	9	8
-1	13	0	0	3	5	15	0	21	22	-1	18	0	4	4
0	13	0	5	0	6	15	0	15	14	0	18	0	11	11
1	13	0	4	3	7	15	0	8	9	1	18	0	5	4
2	13	0	9	10	8	15	0	10	9	2	18	0	8	8
3	13	0	18	18	9	15	0	0	1	3	18	0	13	12
4	13	0	12	12	-8	16	0	0	1	4	18	0	9	9
5	13	0	3	2	-7	16	0	4	2	5	18	0	19	18
6	13	0	23	24	-6	16	0	16	16	6	18	0	5	4
7	13	0	4	5	-5	16	0	9	8	7	18	0	18	19
8	13	0	22	24	-4	16	0	33	33	-6	19	0	13	13
9	13	0	4	1	-3	16	0	6	7	-5	19	0	3	2
10	13	0	12	13	-2	16	0	38	37	-4	19	0	12	12
-9	14	0	15	15	-1	16	0	17	18	-3	19	0	9	9

Table 29. (Continued)

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-2	19	0	11	11	11	0	1	17	18	8	2	1	29	30
-1	19	0	6	4	-12	1	1	13	13	9	2	1	7	7
0	19	0	5	0	-11	1	1	8	8	10	2	1	18	19
1	19	0	4	4	-10	1	1	33	34	11	2	1	8	8
2	19	0	9	11	-9	1	1	17	18	-12	3	1	5	4
3	19	0	10	9	-8	1	1	22	23	-11	3	1	10	11
4	19	0	13	12	-7	1	1	36	37	-10	3	1	4	2
5	19	0	1	2	-6	1	1	32	30	-9	3	1	3	3
6	19	0	13	13	-5	1	1	55	53	-8	3	1	24	24
-5	20	0	7	5	-4	1	1	31	28	-7	3	1	39	39
-4	20	0	11	10	-3	1	1	59	55	-6	3	1	46	45
-3	20	0	2	2	-2	1	1	16	14	-5	3	1	100	98
-2	20	0	13	14	-1	1	1	66	64	-4	3	1	38	33
-1	20	0	6	3	0	1	1	89	92	-3	3	1	110	105
0	20	0	19	19	1	1	1	68	68	-2	3	1	32	28
1	20	0	3	3	2	1	1	59	58	-1	3	1	28	28
2	20	0	13	14	3	1	1	7	6	0	3	1	37	34
3	20	0	0	2	4	1	1	77	77	1	3	1	7	5
4	20	0	9	10	5	1	1	6	4	2	3	1	20	19
5	20	0	4	5	6	1	1	57	57	3	3	1	10	10
-4	21	0	0	2	7	1	1	12	11	4	3	1	48	47
-3	21	0	12	14	8	1	1	15	16	5	3	1	22	20
-2	21	0	5	7	9	1	1	15	16	6	3	1	11	12
-1	21	0	15	17	10	1	1	27	28	7	3	1	6	6
0	21	0	1	0	11	1	1	11	9	8	3	1	13	13
1	21	0	18	17	-12	2	1	0	0	9	3	1	24	26
2	21	0	7	7	-11	2	1	10	10	10	3	1	0	1
3	21	0	12	14	-10	2	1	24	23	11	3	1	14	16
4	21	0	2	2	-9	2	1	18	19	-12	4	1	0	1
-2	22	0	7	10	-8	2	1	13	14	-11	4	1	18	16
-1	22	0	0	0	-7	2	1	4	2	-10	4	1	8	7
0	22	0	11	12	-6	2	1	70	68	-9	4	1	8	8
1	22	0	4	0	-5	2	1	4	3	-8	4	1	3	3
2	22	0	9	10	-4	2	1	157	150	-7	4	1	18	19
-11	0	1	27	28	-3	2	1	10	7	-6	4	1	50	50
-9	0	1	42	42	-2	2	1	33	33	-5	4	1	5	5
-7	0	1	33	33	-1	2	1	38	36	-4	4	1	72	68
-5	0	1	9	9	0	2	1	116	117	-3	4	1	33	31
-3	0	1	26	23	1	2	1	52	51	-2	4	1	13	14
-1	0	1	94	93	2	2	1	53	50	-1	4	1	14	14
1	0	1	150	152	3	2	1	50	43	0	4	1	18	18
3	0	1	74	72	4	2	1	36	35	1	4	1	13	14
5	0	1	63	63	5	2	1	18	17	2	4	1	14	13
7	0	1	55	56	6	2	1	12	12	3	4	1	44	45
9	0	1	31	32	7	2	1	14	15	4	4	1	34	33

Table 29. (Continued)

H	K	L	FO	FC
5	4	1	52	54
6	4	1	14	15
7	4	1	18	18
8	4	1	5	6
9	4	1	6	2
10	4	1	17	16
11	4	1	8	8
-12	5	1	13	13
-11	5	1	3	1
-10	5	1	23	23
-9	5	1	0	0
-8	5	1	20	19
-7	5	1	9	10
-6	5	1	16	15
-5	5	1	13	13
-4	5	1	4	4
-3	5	1	19	19
-2	5	1	31	29
-1	5	1	6	6
0	5	1	50	49
1	5	1	5	5
2	5	1	42	43
3	5	1	18	18
4	5	1	45	47
5	5	1	15	16
6	5	1	42	44
7	5	1	4	4
8	5	1	14	14
9	5	1	11	11
10	5	1	13	14
11	5	1	3	4
-12	6	1	4	5
-11	6	1	17	18
-10	6	1	15	14
-9	6	1	26	26
-8	6	1	4	4
-7	6	1	25	25
-6	6	1	24	24
-5	6	1	0	1
-4	6	1	11	13
-3	6	1	12	11
-2	6	1	28	27
-1	6	1	72	67
0	6	1	24	23
1	6	1	57	57
2	6	1	43	41
3	6	1	48	48
4	6	1	23	21
5	6	1	32	34
6	6	1	10	10
7	6	1	34	35
8	6	1	7	6
9	6	1	15	16
10	6	1	0	1
11	6	1	9	10
-12	7	1	8	8
-11	7	1	14	14
-10	7	1	17	17
-9	7	1	26	26
-8	7	1	16	16
-7	7	1	34	35
-6	7	1	29	30
-5	7	1	48	47
-4	7	1	35	32
-3	7	1	44	44
-2	7	1	9	8
-1	7	1	112	107
0	7	1	75	70
1	7	1	53	51
2	7	1	50	50
3	7	1	10	10
4	7	1	5	3
5	7	1	4	2
6	7	1	33	34
7	7	1	5	4
8	7	1	20	21
9	7	1	13	15
10	7	1	9	8
11	7	1	10	9
-11	8	1	4	4
-10	8	1	18	19
-9	8	1	8	6
-8	8	1	38	38
-7	8	1	21	21
-6	8	1	73	71
-5	8	1	3	4
-4	8	1	43	45
-3	8	1	11	12
-2	8	1	98	96
-1	8	1	50	47
0	8	1	101	97
1	8	1	50	48
2	8	1	56	55
3	8	1	7	7
4	8	1	4	4
5	8	1	0	1
6	8	1	4	2
7	8	1	4	3
8	8	1	15	15
9	8	1	1	3
10	8	1	13	14
11	8	1	0	3
-11	9	1	9	8
-10	9	1	15	14
-9	9	1	19	20
-8	9	1	12	12
-7	9	1	26	26
-6	9	1	2	1
-5	9	1	87	86
-4	9	1	13	13
-3	9	1	57	57
-2	9	1	36	34
-1	9	1	51	50
0	9	1	32	31
1	9	1	63	61
2	9	1	34	35
3	9	1	14	14
4	9	1	37	38
5	9	1	8	7
6	9	1	35	35
7	9	1	15	17
8	9	1	13	13
9	9	1	7	6
10	9	1	9	8
-11	10	1	17	17
-10	10	1	4	4
-9	10	1	18	18
-8	10	1	9	10
-7	10	1	16	16
-6	10	1	20	20
-5	10	1	7	7
-4	10	1	32	31
-3	10	1	15	15
-2	10	1	31	31
-1	10	1	33	32

Table 29. (Continued)

H	K	L	FO	FC
0	10	1	13	13
1	10	1	47	47
2	10	1	4	4
3	10	1	54	55
4	10	1	12	12
5	10	1	70	72
6	10	1	3	2
7	10	1	16	17
8	10	1	3	1
9	10	1	15	16
10	10	1	5	2
-11	11	1	4	6
-10	11	1	13	14
-9	11	1	4	4
-8	11	1	17	17
-7	11	1	9	10
-6	11	1	19	18
-5	11	1	30	30
-4	11	1	5	6
-3	11	1	12	12
-2	11	1	21	22
-1	11	1	3	3
0	11	1	32	33
1	11	1	10	10
2	11	1	49	50
3	11	1	2	1
4	11	1	46	46
5	11	1	4	2
6	11	1	35	37
7	11	1	11	10
8	11	1	16	17
9	11	1	2	3
10	11	1	8	8
-10	12	1	7	8
-9	12	1	14	12
-8	12	1	16	14
-7	12	1	17	15
-6	12	1	13	14
-5	12	1	7	8
-4	12	1	31	30
-3	12	1	8	8
-2	12	1	28	29
-1	12	1	20	19
0	12	1	27	26
1	12	1	50	50
2	12	1	17	17
3	12	1	2	3
4	12	1	4	5
5	12	1	14	15
6	12	1	5	5
7	12	1	23	23
8	12	1	10	11
9	12	1	13	13
-10	13	1	0	2
-9	13	1	25	24
-8	13	1	6	7
-7	13	1	18	18
-6	13	1	18	19
-5	13	1	25	25
-4	13	1	8	9
-3	13	1	6	4
-2	13	1	15	15
-1	13	1	55	55
0	13	1	7	6
1	13	1	44	45
2	13	1	3	1
3	13	1	15	15
4	13	1	7	7
5	13	1	3	1
6	13	1	12	12
7	13	1	16	17
8	13	1	6	5
9	13	1	8	7
-10	14	1	7	6
-9	14	1	8	8
-8	14	1	18	18
-7	14	1	4	4
-6	14	1	19	19
-5	14	1	4	6
-4	14	1	20	19
-3	14	1	21	20
-2	14	1	26	25
-1	14	1	23	23
0	14	1	31	31
1	14	1	5	6
2	14	1	18	18
3	14	1	10	11
4	14	1	3	2
5	14	1	9	8
6	14	1	16	17
7	14	1	3	4
8	14	1	10	11
9	14	1	7	7
-9	15	1	7	7
-8	15	1	10	8
-7	15	1	9	8
-6	15	1	1	2
-5	15	1	18	19
-4	15	1	3	1
-3	15	1	15	14
-2	15	1	6	6
-1	15	1	20	19
0	15	1	18	17
1	15	1	7	5
2	15	1	30	30
3	15	1	9	10
4	15	1	23	24
5	15	1	12	11
6	15	1	16	16
7	15	1	13	13
8	15	1	9	10
-9	16	1	8	6
-8	16	1	0	1
-7	16	1	10	11
-6	16	1	4	2
-5	16	1	10	10
-4	16	1	0	3
-3	16	1	15	16
-2	16	1	4	4
-1	16	1	10	9
0	16	1	7	7
1	16	1	32	30
2	16	1	8	8
3	16	1	32	32
4	16	1	8	7
5	16	1	33	33
6	16	1	5	6
7	16	1	16	15
8	16	1	2	1
-8	17	1	11	9
-7	17	1	14	13
-6	17	1	2	4
-5	17	1	20	20
-4	17	1	2	3
-3	17	1	23	24

Table 29. (Continued)

H	K	L	FO	FC
-2	17	1	6	5
-1	17	1	10	11
0	17	1	19	20
1	17	1	17	17
2	17	1	33	32
3	17	1	16	16
4	17	1	22	22
5	17	1	0	1
6	17	1	18	18
7	17	1	1	1
-7	18	1	6	7
-6	18	1	25	25
-5	18	1	5	0
-4	18	1	22	24
-3	18	1	3	2
-2	18	1	32	33
-1	18	1	7	7
0	18	1	25	25
1	18	1	22	22
2	18	1	19	18
3	18	1	5	7
4	18	1	0	3
5	18	1	8	7
6	18	1	0	1
-6	19	1	2	0
-5	19	1	16	18
-4	19	1	0	0
-3	19	1	26	27
-2	19	1	4	4
-1	19	1	29	29
0	19	1	4	5
1	19	1	14	15
2	19	1	5	5
3	19	1	12	12
4	19	1	6	5
5	19	1	4	0
6	19	1	7	6
-5	20	1	1	0
-4	20	1	12	12
-3	20	1	7	1
-2	20	1	12	13
-1	20	1	7	9
0	20	1	15	15
1	20	1	15	15
2	20	1	10	11
3	20	1	15	15
4	20	1	4	2
5	20	1	9	8
-3	21	1	3	3
-2	21	1	9	9
-1	21	1	0	3
0	21	1	14	14
1	21	1	2	3
2	21	1	11	10
3	21	1	4	3
0	22	1	11	7
1	22	1	9	10
2	22	1	0	3
-10	0	2	58	59
-8	0	2	81	82
-6	0	2	56	58
-4	0	2	57	57
-2	0	2	25	22
0	0	2	178	178
2	0	2	34	33
4	0	2	31	30
6	0	2	45	44
8	0	2	38	38
10	0	2	17	17
-11	1	2	27	28
-10	1	2	2	3
-9	1	2	42	43
-8	1	2	7	7
-7	1	2	61	60
-6	1	2	33	31
-5	1	2	67	63
-4	1	2	0	2
-3	1	2	36	35
-2	1	2	38	38
-1	1	2	12	12
0	1	2	178	199
1	1	2	77	75
2	1	2	12	13
3	1	2	20	20
4	1	2	20	20
5	1	2	35	36
6	1	2	23	23
7	1	2	37	36
8	1	2	7	7
9	1	2	14	14
11	1	2	9	9
-11	2	2	11	11
-10	2	2	18	18
-9	2	2	0	1
-8	2	2	10	11
-7	2	2	16	15
-6	2	2	36	35
-5	2	2	46	45
-4	2	2	23	22
-3	2	2	79	75
-2	2	2	3	1
-1	2	2	72	78
0	2	2	73	73
1	2	2	95	96
2	2	2	5	4
3	2	2	55	55
4	2	2	4	1
5	2	2	47	47
6	2	2	28	28
7	2	2	15	16
8	2	2	9	8
9	2	2	3	5
10	2	2	5	5
11	2	2	0	1
-11	3	2	7	5
-10	3	2	5	7
-9	3	2	0	1
-8	3	2	5	5
-7	3	2	10	9
-6	3	2	18	18
-5	3	2	10	11
-4	3	2	89	85
-3	3	2	7	7
-2	3	2	27	28
-1	3	2	7	5
0	3	2	64	66
1	3	2	37	36
2	3	2	42	43
3	3	2	11	11
4	3	2	65	65
5	3	2	16	16
6	3	2	33	34
7	3	2	4	3
8	3	2	7	8
9	3	2	11	12

Table 29. (Continued)

H	K	L	FO	FC
10	3	2	2	1
11	3	2	7	4
-11	4	2	4	6
-10	4	2	19	20
-9	4	2	1	4
-8	4	2	5	4
-7	4	2	21	22
-6	4	2	51	50
-5	4	2	12	12
-4	4	2	63	59
-3	4	2	22	22
-2	4	2	60	55
-1	4	2	46	46
0	4	2	9	8
1	4	2	45	45
2	4	2	15	15
3	4	2	28	28
4	4	2	47	47
5	4	2	19	18
6	4	2	21	22
7	4	2	19	19
8	4	2	19	19
9	4	2	9	9
10	4	2	8	9
11	4	2	0	3
-11	5	2	21	22
-10	5	2	12	11
-9	5	2	27	27
-8	5	2	23	21
-7	5	2	44	43
-6	5	2	12	12
-5	5	2	32	33
-4	5	2	31	30
-3	5	2	4	3
-2	5	2	21	19
-1	5	2	34	33
0	5	2	18	19
1	5	2	11	11
2	5	2	28	28
3	5	2	28	28
4	5	2	10	9
5	5	2	35	36
6	5	2	13	14
7	5	2	23	23
8	5	2	5	4
9	5	2	23	24
10	5	2	4	4
-11	6	2	9	8
-10	6	2	36	37
-9	6	2	4	0
-8	6	2	25	25
-7	6	2	11	10
-6	6	2	53	52
-5	6	2	4	3
-4	6	2	4	5
-3	6	2	0	3
-2	6	2	41	42
-1	6	2	29	29
0	6	2	7	6
1	6	2	49	47
2	6	2	26	26
3	6	2	7	5
4	6	2	16	17
5	6	2	9	8
6	6	2	40	40
7	6	2	12	11
8	6	2	25	26
9	6	2	4	4
10	6	2	18	18
-10	7	2	8	8
-9	7	2	24	25
-8	7	2	0	1
-7	7	2	26	27
-6	7	2	32	32
-5	7	2	20	20
-4	7	2	8	10
-3	7	2	4	4
-2	7	2	65	64
-1	7	2	4	5
0	7	2	81	79
1	7	2	19	18
2	7	2	61	60
3	7	2	29	29
4	7	2	21	21
5	7	2	24	25
6	7	2	25	25
7	7	2	25	26
8	7	2	5	6
9	7	2	14	15
10	7	2	0	2
-10	8	2	0	0
-9	8	2	4	0
-8	8	2	2	4
-7	8	2	8	7
-6	8	2	3	4
-5	8	2	49	48
-4	8	2	15	14
-3	8	2	44	45
-2	8	2	4	4
-1	8	2	92	91
0	8	2	1	1
1	8	2	87	86
2	8	2	1	2
3	8	2	53	53
4	8	2	5	3
5	8	2	41	41
6	8	2	19	20
7	8	2	10	10
8	8	2	4	0
9	8	2	6	5
10	8	2	4	3
-10	9	2	7	6
-9	9	2	18	18
-8	9	2	3	3
-7	9	2	20	19
-6	9	2	16	16
-5	9	2	68	68
-4	9	2	62	62
-3	9	2	11	12
-2	9	2	34	34
-1	9	2	0	1
0	9	2	45	45
1	9	2	5	4
2	9	2	63	64
3	9	2	4	1
4	9	2	46	46
5	9	2	15	16
6	9	2	21	22
7	9	2	14	14
8	9	2	2	3
9	9	2	9	9
10	9	2	6	1
-10	10	2	19	19
-9	10	2	0	3
-8	10	2	30	30

Table 29. (Continued)

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-7	10	2	4	1	0	12	2	5	5	-6	15	2	8	7
-6	10	2	50	48	1	12	2	32	32	-5	15	2	19	20
-5	10	2	32	32	2	12	2	16	15	-4	15	2	22	23
-4	10	2	61	60	3	12	2	7	8	-3	15	2	15	14
-3	10	2	4	3	4	12	2	0	1	-2	15	2	17	16
-2	10	2	33	33	5	12	2	24	25	-1	15	2	12	12
-1	10	2	6	2	6	12	2	11	10	0	15	2	7	6
0	10	2	3	0	7	12	2	0	2	1	15	2	5	5
1	10	2	15	15	8	12	2	16	16	2	15	2	27	27
2	10	2	17	17	9	12	2	4	4	3	15	2	16	15
3	10	2	32	32	-8	13	2	5	3	4	15	2	10	10
4	10	2	29	29	-7	13	2	5	5	5	15	2	9	8
5	10	2	16	17	-6	13	2	5	6	6	15	2	5	5
6	10	2	12	11	-5	13	2	7	8	7	15	2	7	7
7	10	2	3	1	-4	13	2	19	18	8	15	2	6	2
8	10	2	20	19	-3	13	2	0	2	-7	16	2	0	2
9	10	2	0	1	-2	13	2	39	39	-6	16	2	27	27
-9	11	2	28	28	-1	13	2	11	12	-5	16	2	3	5
-8	11	2	0	1	0	13	2	45	44	-4	16	2	28	27
-7	11	2	13	13	1	13	2	15	14	-3	16	2	13	13
-6	11	2	11	11	2	13	2	33	32	-2	16	2	18	19
-5	11	2	62	62	3	13	2	9	9	-1	16	2	14	14
-4	11	2	7	8	4	13	2	7	7	0	16	2	2	2
-3	11	2	40	41	5	13	2	5	0	1	16	2	9	8
-2	11	2	26	26	6	13	2	10	11	2	16	2	13	13
-1	11	2	17	18	7	13	2	8	9	3	16	2	3	2
0	11	2	12	12	8	13	2	6	4	4	16	2	13	13
1	11	2	13	14	-8	14	2	10	10	5	16	2	0	0
2	11	2	8	9	-7	14	2	2	3	6	16	2	13	14
3	11	2	28	29	-6	14	2	8	8	7	16	2	5	3
4	11	2	2	3	-5	14	2	14	13	-6	17	2	6	5
5	11	2	4	4	-4	14	2	6	7	-5	17	2	24	24
6	11	2	8	8	-3	14	2	35	35	-4	17	2	16	16
7	11	2	20	21	-2	14	2	0	2	-3	17	2	15	15
8	11	2	5	0	-1	14	2	21	20	-2	17	2	22	22
9	11	2	17	16	0	14	2	2	3	-1	17	2	18	17
-9	12	2	2	2	1	14	2	41	40	0	17	2	14	14
-8	12	2	21	21	2	14	2	8	7	1	17	2	15	15
-7	12	2	4	2	3	14	2	14	15	2	17	2	15	14
-6	12	2	18	18	4	14	2	10	10	3	17	2	9	8
-5	12	2	26	25	5	14	2	15	15	4	17	2	15	15
-4	12	2	20	19	6	14	2	0	4	5	17	2	13	13
-3	12	2	21	21	7	14	2	1	1	6	17	2	8	8
-2	12	2	22	21	8	14	2	5	3	-5	18	2	13	13
-1	12	2	41	42	-7	15	2	16	16	-4	18	2	13	12

Table 29. (Continued)

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-3	18	2	27	27	-5	1	3	14	13	-4	3	3	11	11
-2	18	2	9	9	-4	1	3	110	107	-3	3	3	12	12
-1	18	2	29	29	-3	1	3	5	4	-2	3	3	4	3
0	18	2	1	0	-2	1	3	38	39	-1	3	3	16	19
1	18	2	22	23	-1	1	3	50	52	0	3	3	30	29
2	18	2	10	10	0	1	3	98	103	1	3	3	54	54
3	18	2	24	23	1	1	3	45	44	2	3	3	22	21
4	18	2	0	2	2	1	3	17	17	3	3	3	71	71
5	18	2	13	13	3	1	3	32	32	4	3	3	5	5
6	18	2	0	1	4	1	3	22	21	5	3	3	30	30
-3	19	2	7	6	5	1	3	24	24	6	3	3	0	2
-2	19	2	24	25	6	1	3	4	4	7	3	3	16	17
-1	19	2	1	3	7	1	3	17	17	8	3	3	7	7
0	19	2	27	27	8	1	3	15	16	9	3	3	8	9
1	19	2	3	2	9	1	3	11	11	10	3	3	6	6
2	19	2	19	19	10	1	3	0	1	-11	4	3	8	7
3	19	2	1	3	-11	2	3	4	4	-10	4	3	20	20
4	19	2	15	14	-10	2	3	38	38	-9	4	3	12	12
5	19	2	8	7	-9	2	3	8	10	-8	4	3	12	12
0	20	2	5	1	-8	2	3	15	15	-7	4	3	11	10
1	20	2	16	16	-7	2	3	24	24	-6	4	3	39	39
2	20	2	10	9	-6	2	3	8	7	-5	4	3	63	61
3	20	2	6	5	-5	2	3	24	23	-4	4	3	8	8
4	20	2	7	6	-4	2	3	43	39	-3	4	3	54	55
0	21	2	4	3	-3	2	3	35	33	-2	4	3	15	15
1	21	2	0	1	-2	2	3	22	24	-1	4	3	15	17
2	21	2	0	1	-1	2	3	4	5	0	4	3	25	27
0	22	2	3	2	0	2	3	41	43	1	4	3	8	9
-11	0	3	11	11	1	2	3	33	31	2	4	3	57	57
-9	0	3	28	27	2	2	3	79	79	3	4	3	8	8
-7	0	3	61	59	3	2	3	14	12	4	4	3	11	11
-5	0	3	59	59	4	2	3	52	52	5	4	3	7	4
-3	0	3	96	95	5	2	3	2	2	6	4	3	28	28
-1	0	3	90	92	6	2	3	21	21	7	4	3	0	4
1	0	3	123	120	7	2	3	4	5	8	4	3	4	1
3	0	3	10	9	8	2	3	17	17	9	4	3	14	12
5	0	3	5	4	9	2	3	6	3	10	4	3	9	9
7	0	3	8	7	10	2	3	13	13	-11	5	3	5	2
9	0	3	11	11	-11	3	3	27	27	-10	5	3	16	16
-11	1	3	18	19	-10	3	3	9	10	-9	5	3	3	5
-10	1	3	18	17	-9	3	3	18	17	-8	5	3	14	13
-9	1	3	17	17	-8	3	3	5	4	-7	5	3	8	8
-8	1	3	20	20	-7	3	3	13	13	-6	5	3	49	50
-7	1	3	20	19	-6	3	3	36	35	-5	5	3	8	9
-6	1	3	40	40	-5	3	3	21	20	-4	5	3	52	51

Table 29. (Continued)

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FC	FC
-3	5	3	21	21	-1	7	3	3	3	4	9	3	4	3
-2	5	3	40	42	0	7	3	62	62	5	9	3	35	35
-1	5	3	30	31	1	7	3	41	41	6	9	3	12	12
0	5	3	54	55	2	7	3	12	13	7	9	3	16	15
1	5	3	28	28	3	7	3	23	21	8	9	3	7	7
2	5	3	15	14	4	7	3	11	11	9	9	3	6	6
3	5	3	24	26	5	7	3	30	29	-10	10	3	4	7
4	5	3	2	1	6	7	3	11	10	-9	10	3	17	16
5	5	3	14	13	7	7	3	17	17	-8	10	3	7	7
6	5	3	4	4	8	7	3	5	6	-7	10	3	23	23
7	5	3	9	9	9	7	3	11	11	-6	10	3	20	20
8	5	3	10	10	-10	8	3	33	32	-5	10	3	75	75
9	5	3	8	4	-9	8	3	1	1	-4	10	3	0	1
10	5	3	10	10	-8	8	3	31	31	-3	10	3	47	48
-11	6	3	12	12	-7	8	3	13	13	-2	10	3	17	17
-10	6	3	14	15	-6	8	3	34	35	-1	10	3	39	39
-9	6	3	23	23	-5	8	3	11	9	0	10	3	7	6
-8	6	3	14	14	-4	8	3	12	12	1	10	3	41	40
-7	6	3	23	22	-3	8	3	3	3	2	10	3	18	18
-6	6	3	5	2	-2	8	3	12	11	3	10	3	8	8
-5	6	3	45	45	-1	8	3	3	1	4	10	3	12	11
-4	6	3	5	6	0	8	3	35	35	5	10	3	18	18
-3	6	3	48	49	1	8	3	0	1	6	10	3	5	6
-2	6	3	17	17	2	8	3	37	37	7	10	3	9	8
-1	6	3	77	78	3	8	3	4	1	8	10	3	4	2
0	6	3	16	15	4	8	3	37	37	9	10	3	6	5
1	6	3	31	31	5	8	3	13	12	-9	11	3	8	7
2	6	3	15	14	6	8	3	32	32	-8	11	3	21	21
3	6	3	7	6	7	8	3	7	8	-7	11	3	5	5
4	6	3	28	29	8	8	3	18	18	-6	11	3	24	25
5	6	3	3	5	9	8	3	1	2	-5	11	3	3	2
6	6	3	6	6	-10	9	3	6	7	-4	11	3	56	57
7	6	3	8	8	-9	9	3	20	20	-3	11	3	15	15
8	6	3	6	4	-8	9	3	18	17	-2	11	3	47	48
9	6	3	16	14	-7	9	3	25	25	-1	11	3	15	14
10	6	3	5	2	-6	9	3	38	38	0	11	3	27	27
-10	7	3	6	7	-5	9	3	30	30	1	11	3	5	4
-9	7	3	31	33	-4	9	3	24	25	2	11	3	22	23
-8	7	3	28	28	-3	9	3	14	15	3	11	3	18	18
-7	7	3	14	14	-2	9	3	47	47	4	11	3	5	5
-6	7	3	15	15	-1	9	3	22	22	5	11	3	17	17
-5	7	3	15	14	0	9	3	22	23	6	11	3	6	5
-4	7	3	32	33	1	9	3	11	11	7	11	3	0	3
-3	7	3	13	13	2	9	3	25	26	8	11	3	9	8
-2	7	3	29	20	3	9	3	42	41	-9	12	3	9	17

Table 29. (Continued)

H	K	L	FO	FC
-8	12	3	15	15
-7	12	3	18	17
-6	12	3	6	4
-5	12	3	11	8
-4	12	3	0	1
-3	12	3	15	15
-2	12	3	19	19
-1	12	3	38	40
0	12	3	17	17
1	12	3	32	33
2	12	3	17	17
3	12	3	9	10
4	12	3	27	27
5	12	3	3	1
6	12	3	14	14
7	12	3	3	2
8	12	3	11	11
-8	13	3	2	2
-7	13	3	4	9
-6	13	3	4	4
-5	13	3	3	5
-4	13	3	5	4
-3	13	3	18	18
-2	13	3	15	14
-1	13	3	22	23
0	13	3	8	8
1	13	3	22	21
2	13	3	5	4
3	13	3	18	18
4	13	3	4	4
5	13	3	17	17
6	13	3	5	3
7	13	3	21	21
8	13	3	0	1
-8	14	3	18	19
-7	14	3	12	11
-6	14	3	10	10
-5	14	3	12	13
-4	14	3	8	7
-3	14	3	7	8
-2	14	3	25	25
-1	14	3	18	18
0	14	3	12	12
1	14	3	8	8
2	14	3	21	20
3	14	3	1	1
4	14	3	16	16
5	14	3	7	7
6	14	3	22	21
7	14	3	3	2
-7	15	3	10	11
-6	15	3	23	24
-5	15	3	7	7
-4	15	3	20	21
-3	15	3	25	25
-2	15	3	16	17
-1	15	3	2	3
0	15	3	15	14
1	15	3	19	18
2	15	3	17	17
3	15	3	15	14
4	15	3	8	6
5	15	3	17	16
6	15	3	4	1
7	15	3	12	11
-6	16	3	3	1
-5	16	3	33	32
-4	16	3	3	0
-3	16	3	32	31
-2	16	3	7	9
-1	16	3	16	16
0	16	3	2	3
1	16	3	18	17
2	16	3	9	9
3	16	3	16	16
4	16	3	5	2
5	16	3	0	1
6	16	3	5	4
-5	17	3	8	8
-4	17	3	16	20
-3	17	3	3	1
-2	17	3	18	22
-1	17	3	10	10
0	17	3	17	18
1	17	3	6	3
2	17	3	13	13
3	17	3	9	7
4	17	3	1	1
5	17	3	10	10
-4	18	3	3	2
-3	18	3	12	12
-2	18	3	0	3
-1	18	3	6	5
0	18	3	14	14
1	18	3	7	6
2	18	3	16	15
3	18	3	3	1
4	18	3	14	12
5	18	3	4	3
-1	19	3	5	5
0	19	3	5	5
1	19	3	20	19
2	19	3	2	1
3	19	3	9	8
4	19	3	1	1
0	20	3	7	6
1	20	3	9	8
2	20	3	4	4
0	21	3	14	13
1	21	3	0	0
-10	0	4	24	25
-8	0	4	12	12
-6	0	4	20	20
-4	0	4	23	23
-2	0	4	80	84
0	0	4	88	87
2	0	4	28	28
4	0	4	20	19
6	0	4	25	26
8	0	4	11	10
-11	1	4	11	12
-10	1	4	28	28
-9	1	4	14	13
-8	1	4	37	37
-7	1	4	10	10
-6	1	4	30	29
-5	1	4	16	16
-4	1	4	27	27
-3	1	4	54	56
-2	1	4	5	4
-1	1	4	51	63
0	1	4	35	41
1	1	4	17	17
2	1	4	20	20
3	1	4	29	28

Table 29. (Continued)

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
4	1	4	12	12	7	3	4	7	6	-10	6	4	17	17
5	1	4	22	21	8	3	4	12	11	-9	6	4	12	12
6	1	4	8	7	9	3	4	7	5	-8	6	4	15	16
7	1	4	16	16	-11	4	4	15	15	-7	6	4	14	13
8	1	4	16	16	-10	4	4	2	3	-6	6	4	3	2
9	1	4	0	2	-9	4	4	17	17	-5	6	4	16	15
-11	2	4	22	22	-8	4	4	9	9	-4	6	4	46	47
-10	2	4	13	13	-7	4	4	33	32	-3	6	4	5	6
-9	2	4	33	33	-6	4	4	11	10	-2	6	4	43	47
-8	2	4	14	13	-5	4	4	27	27	-1	6	4	19	20
-7	2	4	47	47	-4	4	4	23	24	0	6	4	56	59
-6	2	4	10	9	-3	4	4	19	22	1	6	4	10	11
-5	2	4	54	54	-2	4	4	40	49	2	6	4	36	36
-4	2	4	35	35	-1	4	4	5	6	3	6	4	0	2
-3	2	4	43	46	0	4	4	33	37	4	6	4	14	12
-2	2	4	11	12	1	4	4	5	3	5	6	4	7	7
-1	2	4	3	3	2	4	4	17	17	6	6	4	20	20
0	2	4	27	31	3	4	4	17	17	7	6	4	3	1
1	2	4	14	15	4	4	4	33	32	8	6	4	0	3
2	2	4	16	16	5	4	4	8	8	9	6	4	6	2
3	2	4	25	24	6	4	4	10	9	-10	7	4	27	27
4	2	4	10	9	7	4	4	5	4	-9	7	4	10	9
5	2	4	16	15	8	4	4	2	2	-8	7	4	25	25
6	2	4	12	13	9	4	4	12	11	-7	7	4	1	1
7	2	4	14	14	-11	5	4	15	14	-6	7	4	42	42
8	2	4	5	4	-10	5	4	7	5	-5	7	4	25	26
9	2	4	17	15	-9	5	4	5	5	-4	7	4	21	22
-11	3	4	6	5	-8	5	4	19	18	-3	7	4	39	42
-10	3	4	27	28	-7	5	4	16	17	-2	7	4	21	25
-9	3	4	9	10	-6	5	4	4	3	-1	7	4	35	39
-8	3	4	21	20	-5	5	4	20	20	0	7	4	14	13
-7	3	4	6	5	-4	5	4	9	8	1	7	4	23	22
-6	3	4	58	58	-3	5	4	41	43	2	7	4	6	6
-5	3	4	23	22	-2	5	4	15	16	3	7	4	26	24
-4	3	4	65	67	-1	5	4	40	45	4	7	4	8	9
-3	3	4	12	12	0	5	4	9	11	5	7	4	11	11
-2	3	4	9	12	1	5	4	55	55	6	7	4	10	10
-1	3	4	10	13	2	5	4	18	19	7	7	4	6	6
0	3	4	20	22	3	5	4	19	19	8	7	4	13	12
1	3	4	38	38	4	5	4	3	3	-10	8	4	2	0
2	3	4	19	18	5	5	4	19	17	-9	8	4	37	36
3	3	4	6	7	6	5	4	2	2	-8	8	4	0	1
4	3	4	17	17	7	5	4	14	13	-7	8	4	36	35
5	3	4	13	13	8	5	4	4	5	-6	8	4	15	15
6	3	4	13	12	9	5	4	5	2	-5	8	4	68	68

Table 29. (Continued)

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
-4	8	4	41	41	4	10	4	24	24	-1	13	4	3	4
-3	8	4	14	14	5	10	4	7	8	0	13	4	3	4
-2	8	4	0	2	6	10	4	9	9	1	13	4	11	12
-1	8	4	19	20	7	10	4	1	3	2	13	4	4	2
0	8	4	5	2	8	10	4	2	4	3	13	4	0	1
1	8	4	15	15	-9	11	4	5	5	4	13	4	17	16
2	8	4	4	3	-8	11	4	6	6	5	13	4	7	5
3	8	4	8	8	-7	11	4	12	11	6	13	4	11	10
4	8	4	6	4	-6	11	4	10	8	7	13	4	0	3
5	8	4	14	14	-5	11	4	14	14	-7	14	4	16	16
6	8	4	5	4	-4	11	4	3	3	-6	14	4	0	1
7	8	4	17	17	-3	11	4	19	19	-5	14	4	17	17
8	8	4	0	0	-2	11	4	5	5	-4	14	4	2	1
-10	9	4	18	17	-1	11	4	27	27	-3	14	4	14	14
-9	9	4	6	2	0	11	4	0	3	-2	14	4	9	9
-8	9	4	27	27	1	11	4	35	34	-1	14	4	5	6
-7	9	4	11	10	2	11	4	4	2	0	14	4	15	16
-6	9	4	39	39	3	11	4	28	28	1	14	4	5	2
-5	9	4	8	7	4	11	4	18	18	2	14	4	0	1
-4	9	4	49	49	5	11	4	15	15	3	14	4	10	10
-3	9	4	15	16	6	11	4	0	1	4	14	4	8	8
-2	9	4	15	15	7	11	4	1	3	-6	15	4	15	14
-1	9	4	32	34	-8	12	4	4	1	-5	15	4	13	12
0	9	4	7	7	-7	12	4	9	8	-4	15	4	12	12
1	9	4	31	31	-6	12	4	1	3	-3	15	4	9	10
2	9	4	5	3	-5	12	4	24	25	-2	15	4	9	8
3	9	4	15	14	-4	12	4	6	7	-1	15	4	15	14
4	9	4	10	9	-3	12	4	12	13	0	15	4	6	5
5	9	4	13	12	-2	12	4	16	15	1	15	4	20	19
6	9	4	8	8	-1	12	4	8	8	2	15	4	6	5
7	9	4	4	3	0	12	4	20	21	3	15	4	17	16
8	9	4	10	10	1	12	4	5	5	-6	16	4	8	8
-9	10	4	11	11	2	12	4	24	24	-5	16	4	0	0
-8	10	4	0	0	3	12	4	20	19	-4	16	4	19	18
-7	10	4	5	6	4	12	4	1	3	-3	16	4	4	4
-6	10	4	21	22	5	12	4	7	5	-2	16	4	19	19
-5	10	4	26	26	6	12	4	3	2	-1	16	4	7	8
-4	10	4	18	19	7	12	4	11	11	0	16	4	14	14
-3	10	4	18	18	-8	13	4	26	26	1	16	4	2	3
-2	10	4	25	26	-7	13	4	5	1	2	16	4	28	27
-1	10	4	4	5	-6	13	4	14	15	-4	17	4	12	11
0	10	4	36	36	-5	13	4	5	6	-3	17	4	20	20
1	10	4	8	8	-4	13	4	16	16	-2	17	4	12	11
2	10	4	42	42	-3	13	4	12	13	-1	17	4	8	6
3	10	4	6	4	-2	13	4	13	13	0	17	4	6	6

Table 29. (Continued)

H	K	L	FO	FC
1	17	4	24	23
-11	0	5	27	28
-9	0	5	34	33
-7	0	5	38	38
-5	0	5	8	8
-3	0	5	14	13
-1	0	5	38	38
1	0	5	4	4
3	0	5	30	28
5	0	5	21	19
-10	1	5	33	33
-9	1	5	22	21
-8	1	5	17	17
-7	1	5	21	21
-6	1	5	15	15
-5	1	5	24	25
-4	1	5	28	27
-3	1	5	26	27
-2	1	5	17	19
-1	1	5	28	32
0	1	5	0	1
1	1	5	29	29
2	1	5	18	18
3	1	5	6	7
4	1	5	18	17
5	1	5	5	3
6	1	5	12	12
-10	2	5	7	8
-9	2	5	9	6
-8	2	5	25	25
-7	2	5	18	17
-6	2	5	48	48
-5	2	5	0	0
-4	2	5	46	46
-3	2	5	9	8
-2	2	5	24	28
-1	2	5	4	5
0	2	5	41	44
1	2	5	11	10
2	2	5	18	18
3	2	5	11	11
4	2	5	15	14
5	2	5	3	3
6	2	5	5	1
-10	3	5	10	12
-9	3	5	9	10
-8	3	5	4	4
-7	3	5	38	38
-6	3	5	9	8
-5	3	5	47	47
-4	3	5	3	3
-3	3	5	33	36
-2	3	5	7	8
-1	3	5	15	19
0	3	5	5	6
1	3	5	29	28
2	3	5	22	22
3	3	5	20	20
4	3	5	15	14
5	3	5	4	2
6	3	5	6	1
-10	4	5	3	3
-9	4	5	16	15
-8	4	5	30	29
-7	4	5	0	1
-6	4	5	16	16
-5	4	5	13	13
-4	4	5	26	27
-3	4	5	4	5
-2	4	5	14	15
-1	4	5	7	7
0	4	5	18	19
1	4	5	22	21
2	4	5	26	25
3	4	5	25	24
4	4	5	2	1
5	4	5	18	18
6	4	5	0	1
-10	5	5	31	29
-9	5	5	9	7
-8	5	5	8	8
-7	5	5	21	22
-6	5	5	19	19
-5	5	5	3	3
-4	5	5	3	1
-3	5	5	4	3
-2	5	5	18	19
-1	5	5	16	17
0	5	5	4	5
1	5	5	10	9
2	5	5	29	28
3	5	5	13	12
4	5	5	25	23
5	5	5	9	9
6	5	5	18	17
-10	6	5	0	1
-9	6	5	28	29
-8	6	5	12	11
-7	6	5	2	2
-6	6	5	14	14
-5	6	5	16	16
-4	6	5	11	12
-3	6	5	11	11
-2	6	5	19	21
-1	6	5	12	13
0	6	5	3	3
1	6	5	26	25
2	6	5	1	3
3	6	5	23	21
4	6	5	6	7
5	6	5	19	17
6	6	5	5	4
-10	7	5	17	16
-9	7	5	9	9
-8	7	5	20	20
-7	7	5	17	16
-6	7	5	8	8
-5	7	5	22	22
-4	7	5	12	12
-3	7	5	34	37
-2	7	5	11	12
-1	7	5	26	27
0	7	5	8	8
1	7	5	20	19
2	7	5	20	19
3	7	5	5	3
4	7	5	13	12
5	7	5	8	8
6	7	5	18	16
-9	8	5	3	1
-8	8	5	24	23
-7	8	5	0	0
-6	8	5	26	24
-5	8	5	0	2
-4	8	5	41	43

Table 29. (Continued)

H	K	L	FO	FC
-3	8	5	11	11
-2	8	5	34	37
-1	8	5	9	9
0	8	5	33	34
1	8	5	10	9
2	8	5	17	17
3	8	5	5	5
4	8	5	2	3
5	8	5	7	7
-9	9	5	12	10
-8	9	5	11	12
-7	9	5	13	13
-6	9	5	11	11
-5	9	5	33	34
-4	9	5	9	8
-3	9	5	34	34
-2	9	5	0	0
-1	9	5	19	20
0	9	5	15	16
1	9	5	18	17
2	9	5	7	7
3	9	5	9	8
4	9	5	12	13
5	9	5	8	8
-9	10	5	22	21
-8	10	5	8	8
-7	10	5	12	12
-6	10	5	5	4
-5	10	5	22	22
-4	10	5	22	22
-3	10	5	5	5
-2	10	5	5	3
-1	10	5	8	8
0	10	5	4	5
1	10	5	10	10
2	10	5	5	5
3	10	5	29	27
4	10	5	4	4
5	10	5	15	13
-8	11	5	18	18
-7	11	5	6	5
-6	11	5	9	8
-5	11	5	26	24
-4	11	5	13	13
-3	11	5	14	14
-2	11	5	0	1
-1	11	5	9	9
0	11	5	13	13
1	11	5	0	1
2	11	5	15	14
3	11	5	6	4
4	11	5	20	20
-8	12	5	7	7
-7	12	5	11	10
-6	12	5	19	20
-5	12	5	4	4
-4	12	5	24	24
-3	12	5	1	2
-2	12	5	26	26
-1	12	5	3	1
0	12	5	14	14
1	12	5	7	6
2	12	5	7	6
3	12	5	9	10
4	12	5	3	3
-7	13	5	17	16
-6	13	5	1	3
-5	13	5	16	16
-4	13	5	3	2
-3	13	5	20	21
-2	13	5	3	1
-1	13	5	32	32
0	13	5	0	2
1	13	5	18	17
2	13	5	0	2
3	13	5	0	2
-6	14	5	16	15
-5	14	5	9	8
-4	14	5	19	19
-3	14	5	1	0
-2	14	5	30	30
-1	14	5	11	10
0	14	5	11	11
1	14	5	4	3
2	14	5	12	12
-5	15	5	13	13
-4	15	5	2	3
-3	15	5	26	26
-2	15	5	8	7
-1	15	5	4	3
0	15	5	11	10
1	15	5	6	8
-4	16	5	0	1
-3	16	5	8	6
-2	16	5	9	9
-1	16	5	9	8
0	16	5	6	4
-10	0	6	26	26
-8	0	6	30	29
-6	0	6	30	30
-4	0	6	26	26
-2	0	6	19	19
0	0	6	32	30
2	0	6	13	10
4	0	6	13	12
-10	1	6	9	10
-9	1	6	18	18
-8	1	6	0	1
-7	1	6	17	16
-6	1	6	9	8
-5	1	6	23	22
-4	1	6	0	1
-3	1	6	32	32
-2	1	6	29	30
-1	1	6	13	13
0	1	6	27	28
1	1	6	1	1
2	1	6	13	12
3	1	6	9	7
4	1	6	9	8
5	1	6	8	7
-10	2	6	2	1
-9	2	6	5	4
-8	2	6	7	6
-7	2	6	0	2
-6	2	6	10	10
-5	2	6	14	14
-4	2	6	18	17
-3	2	6	17	17
-2	2	6	8	8
-1	2	6	33	38
0	2	6	5	4
1	2	6	22	21
2	2	6	0	0
3	2	6	25	23

Table 29. (Continued)

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
4	2	6	2	4	3	5	6	11	10	-6	9	6	0	2
5	2	6	12	12	4	5	6	3	1	-5	9	6	16	17
-10	3	6	16	14	-9	6	6	5	4	-4	9	6	11	11
-9	3	6	14	14	-8	6	6	31	30	-3	9	6	3	2
-8	3	6	6	5	-7	6	6	2	1	-2	9	6	13	14
-7	3	6	12	11	-6	6	6	13	13	-1	9	6	4	6
-6	3	6	3	3	-5	6	6	6	7	0	9	6	16	16
-5	3	6	16	16	-4	6	6	28	30	1	9	6	8	8
-4	3	6	14	14	-3	6	6	9	10	2	9	6	17	16
-3	3	6	2	2	-2	6	6	15	18	3	9	6	1	2
-2	3	6	27	29	-1	6	6	5	3	-8	10	6	23	22
-1	3	6	3	5	0	6	6	9	9	-7	10	6	0	2
0	3	6	15	14	1	6	6	6	6	-6	10	6	32	31
1	3	6	14	13	2	6	6	2	3	-5	10	6	5	2
2	3	6	27	27	3	6	6	4	6	-4	10	6	19	19
3	3	6	7	6	4	6	6	6	8	-3	10	6	0	1
4	3	6	19	18	-9	7	6	12	13	-2	10	6	6	8
5	3	6	0	0	-8	7	6	3	3	-1	10	6	0	1
-9	4	6	6	5	-7	7	6	15	14	0	10	6	5	6
-8	4	6	15	15	-6	7	6	4	2	1	10	6	7	7
-7	4	6	8	8	-5	7	6	20	20	2	10	6	4	4
-6	4	6	26	25	-4	7	6	1	3	3	10	6	7	7
-5	4	6	5	3	-3	7	6	17	18	-7	11	6	17	16
-4	4	6	24	24	-2	7	6	23	25	-6	11	6	11	11
-3	4	6	9	9	-1	7	6	6	7	-5	11	6	24	25
-2	4	6	3	3	0	7	6	18	19	-4	11	6	11	10
-1	4	6	19	19	1	7	6	4	3	-3	11	6	13	15
0	4	6	13	14	2	7	6	11	10	-2	11	6	7	6
1	4	6	17	17	3	7	6	5	6	-1	11	6	6	6
2	4	6	3	2	4	7	6	6	5	0	11	6	8	7
3	4	6	20	18	-8	8	6	5	3	1	11	6	2	3
4	4	6	7	6	-7	8	6	6	5	2	11	6	3	2
5	4	6	0	3	-6	8	6	13	13	-6	12	6	10	10
-9	5	6	22	21	-5	8	6	5	5	-5	12	6	12	11
-8	5	6	3	4	-4	8	6	21	21	-4	12	6	8	8
-7	5	6	32	30	-3	8	6	27	30	-3	12	6	12	10
-6	5	6	5	2	-2	8	6	2	0	-2	12	6	3	6
-5	5	6	23	23	-1	8	6	21	25	-1	12	6	17	17
-4	5	6	6	6	0	8	6	0	2	0	12	6	3	2
-3	5	6	17	17	1	8	6	20	19	1	12	6	8	8
-2	5	6	3	2	2	8	6	8	8	-5	13	6	0	3
-1	5	6	12	14	3	8	6	13	13	-4	13	6	9	10
0	5	6	12	12	4	8	6	0	2	-3	13	6	6	5
1	5	6	11	11	-8	9	6	0	2	-2	13	6	17	17
2	5	6	15	15	-7	9	6	18	18	-1	13	6	0	1

Table 29. (Continued)

H	K	L	FO	FC
0	13	6	18	17
-4	14	6	6	6
-3	14	6	14	15
-2	14	6	2	0
-1	14	6	11	12
-9	0	7	11	11
-7	0	7	10	9
-5	0	7	28	26
-3	0	7	21	20
-1	0	7	33	31
1	0	7	10	8
3	0	7	6	5
-9	1	7	15	15
-8	1	7	10	11
-7	1	7	11	11
-6	1	7	14	13
-5	1	7	9	7
-4	1	7	18	17
-3	1	7	3	1
-2	1	7	21	20
-1	1	7	14	14
0	1	7	14	14
1	1	7	3	1
2	1	7	2	1
3	1	7	7	7
-9	2	7	7	5
-8	2	7	17	16
-7	2	7	4	1
-6	2	7	13	13
-5	2	7	12	9
-4	2	7	13	12
-3	2	7	4	3
-2	2	7	11	11
-1	2	7	1	3
0	2	7	3	5
1	2	7	6	4
2	2	7	14	13
3	2	7	5	2
-8	3	7	0	3
-7	3	7	18	18
-6	3	7	19	18
-5	3	7	12	12
-4	3	7	8	8
-3	3	7	3	3
-2	3	7	7	6
-1	3	7	8	9
0	3	7	7	6
1	3	7	8	7
2	3	7	7	6
3	3	7	11	11
-8	4	7	13	12
-7	4	7	19	18
-6	4	7	15	15
-5	4	7	20	19
-4	4	7	3	4
-3	4	7	19	20
-2	4	7	8	9
-1	4	7	8	8
0	4	7	5	4
1	4	7	12	11
2	4	7	3	3
3	4	7	8	6
-8	5	7	15	13
-7	5	7	13	12
-6	5	7	21	19
-5	5	7	4	2
-4	5	7	23	23
-3	5	7	5	6
-2	5	7	22	24
-1	5	7	4	6
0	5	7	8	8
1	5	7	5	2
2	5	7	14	13
-8	6	7	7	6
-7	6	7	26	23
-6	6	7	0	3
-5	6	7	10	10
-4	6	7	6	5
-3	6	7	26	29
-2	6	7	1	4
-1	6	7	17	19
0	6	7	0	1
1	6	7	12	12
2	6	7	5	5
-7	7	7	0	2
-6	7	7	11	9
-5	7	7	9	8
-4	7	7	18	19
-3	7	7	3	5
-2	7	7	14	16
-1	7	7	0	2
0	7	7	12	13
1	7	7	10	9
2	7	7	7	7
-7	8	7	1	1
-6	8	7	7	7
-5	8	7	4	2
-4	8	7	8	8
-3	8	7	9	8
-2	8	7	0	4
-1	8	7	5	4
0	8	7	6	7
1	8	7	5	2
-6	9	7	12	12
-5	9	7	10	11
-4	9	7	4	4
-3	9	7	4	1
-2	9	7	8	9
-1	9	7	7	8
0	9	7	10	10
1	9	7	2	3
-5	10	7	19	21
-4	10	7	9	9
-3	10	7	15	16
-2	10	7	6	5
-1	10	7	9	10
0	10	7	4	2
-4	11	7	16	16
-3	11	7	3	1
-2	11	7	11	11
-1	11	7	3	3
-6	0	8	5	5
-4	0	8	6	5
-2	0	8	16	15
0	0	8	13	11
-6	1	8	11	10
-5	1	8	8	6
-4	1	8	6	4
-3	1	8	3	3
-2	1	8	8	5
-1	1	8	12	11
0	1	8	8	9
-6	2	8	0	0
-5	2	8	19	17
-4	2	8	6	4

Table 29. (Continued)

H	K	L	FO	FC
-3	2	8	9	9
-2	2	8	0	3
-1	2	8	4	6
0	2	8	3	2
-6	3	8	20	18
-5	3	8	4	2
-4	3	8	15	15
-3	3	8	3	5
-2	3	8	8	6
-1	3	8	8	9
0	3	8	3	3
-6	4	8	2	2
-5	4	8	9	8
-4	4	8	0	2
-3	4	8	6	6
-2	4	8	14	14
-1	4	8	2	5
-5	5	8	0	2
-4	5	8	8	6
-3	5	8	9	9
-2	5	8	3	1
-1	5	8	10	10
-4	6	8	5	2
-3	6	8	5	3
-2	6	8	9	12

Table 30. Final observed and calculated structure amplitudes in electrons for $Ni(EMG)_2$

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
0	0	-14	4	6	1	7	-12	1	1	2	2	-10	18	17
1	0	-14	4	4	0	0	-11	0	0	3	2	-10	16	16
0	1	-14	3	0	1	0	-11	2	0	4	2	-10	10	10
1	1	-14	0	4	2	0	-11	0	0	0	3	-10	8	9
0	2	-14	7	7	3	0	-11	0	0	1	3	-10	9	10
0	0	-13	0	0	0	1	-11	14	15	2	3	-10	0	1
1	0	-13	0	0	1	1	-11	10	10	3	3	-10	4	1
2	0	-13	3	0	2	1	-11	9	8	0	4	-10	30	32
0	1	-13	7	7	3	1	-11	13	12	1	4	-10	8	8
1	1	-13	10	9	0	2	-11	0	1	2	4	-10	13	13
2	1	-13	8	7	1	2	-11	9	9	3	4	-10	15	15
0	2	-13	0	1	2	2	-11	5	5	0	5	-10	6	6
1	2	-13	4	4	3	2	-11	3	4	1	5	-10	5	5
2	2	-13	0	0	0	3	-11	18	19	2	5	-10	1	3
0	3	-13	11	11	1	3	-11	17	17	3	5	-10	0	0
1	3	-13	11	11	2	3	-11	20	20	0	6	-10	16	17
2	3	-13	14	14	3	3	-11	16	16	1	6	-10	0	1
0	4	-13	0	3	0	4	-11	7	7	2	6	-10	10	10
1	4	-13	5	3	1	4	-11	2	3	3	6	-10	12	12
0	5	-13	5	3	2	4	-11	3	2	0	7	-10	5	6
1	5	-13	6	7	3	4	-11	3	3	1	7	-10	0	0
0	0	-12	10	11	0	5	-11	12	12	2	7	-10	2	2
1	0	-12	8	7	1	5	-11	2	2	0	8	-10	11	11
2	0	-12	6	6	2	5	-11	9	8	1	8	-10	8	9
3	0	-12	9	9	3	5	-11	11	10	2	8	-10	7	7
0	1	-12	3	2	0	6	-11	3	3	0	9	-10	2	2
1	1	-12	4	4	1	6	-11	3	4	1	9	-10	0	1
2	1	-12	0	1	2	6	-11	0	0	0	0	-9	1	0
3	1	-12	0	2	0	7	-11	5	7	1	0	-9	0	0
0	2	-12	9	10	1	7	-11	5	5	2	0	-9	0	0
1	2	-12	12	12	2	7	-11	10	11	3	0	-9	3	0
2	2	-12	15	15	0	8	-11	0	2	4	0	-9	0	0
3	2	-12	13	13	1	8	-11	0	1	0	1	-9	21	23
0	3	-12	5	3	0	0	-10	8	9	1	1	-9	17	17
1	3	-12	1	0	1	0	-10	22	22	2	1	-9	20	20
2	3	-12	0	2	2	0	-10	3	3	3	1	-9	8	8
0	4	-12	12	12	3	0	-10	9	9	4	1	-9	12	12
1	4	-12	10	10	4	0	-10	14	13	0	2	-9	7	7
2	4	-12	12	13	0	1	-10	3	4	1	2	-9	0	1
0	5	-12	3	1	1	1	-10	4	2	2	2	-9	11	10
1	5	-12	3	2	2	1	-10	1	0	3	2	-9	6	6
2	5	-12	0	0	3	1	-10	5	6	4	2	-9	4	4
0	6	-12	4	4	4	1	-10	3	2	0	3	-9	22	23
1	6	-12	3	3	0	2	-10	15	16	1	3	-9	20	20
0	7	-12	5	2	1	2	-10	20	21	2	3	-9	17	17

Table 30. (Continued)

H	K	L	FO	FC
3	3	-9	21	20
4	3	-9	14	13
0	4	-9	3	2
1	4	-9	9	9
2	4	-9	4	5
3	4	-9	4	4
4	4	-9	0	1
0	5	-9	32	35
1	5	-9	21	21
2	5	-9	0	2
3	5	-9	12	13
0	6	-9	1	3
1	6	-9	0	0
2	6	-9	1	1
3	6	-9	5	3
0	7	-9	11	11
1	7	-9	9	10
2	7	-9	7	7
3	7	-9	14	14
0	8	-9	1	1
1	8	-9	6	6
2	8	-9	0	1
3	8	-9	0	1
0	9	-9	13	12
1	9	-9	8	9
2	9	-9	7	7
0	10	-9	1	1
1	10	-9	3	4
0	0	-8	54	59
1	0	-8	7	8
2	0	-8	19	20
3	0	-8	8	7
4	0	-8	14	15
0	1	-8	7	8
1	1	-8	1	2
2	1	-8	6	5
3	1	-8	4	6
4	1	-8	7	7
0	2	-8	25	27
1	2	-8	22	22
2	2	-8	28	28
3	2	-8	19	18
4	2	-8	12	12
0	3	-8	0	2
1	3	-8	6	7
2	3	-8	3	3
3	3	-8	3	3
4	3	-8	4	4
0	4	-8	22	24
1	4	-8	37	38
2	4	-8	2	2
3	4	-8	13	13
4	4	-8	14	13
0	5	-8	6	6
1	5	-8	10	11
2	5	-8	8	9
3	5	-8	1	1
4	5	-8	0	0
0	6	-8	11	12
1	6	-8	28	29
2	6	-8	6	5
3	6	-8	12	12
4	6	-8	11	10
0	7	-8	1	2
1	7	-8	6	7
2	7	-8	0	4
3	7	-8	3	3
0	8	-8	15	15
1	8	-8	12	13
2	8	-8	9	10
3	8	-8	9	8
0	9	-8	0	1
1	9	-8	0	1
2	9	-8	2	1
3	9	-8	2	2
0	10	-8	14	15
1	10	-8	6	5
2	10	-8	9	9
0	11	-8	3	1
1	11	-8	0	2
0	0	-7	8	0
1	0	-7	2	0
2	0	-7	2	0
3	0	-7	0	0
4	0	-7	4	0
0	1	-7	38	41
1	1	-7	31	31
2	1	-7	29	28
3	1	-7	17	17
4	1	-7	14	14
0	2	-7	0	2
1	2	-7	0	2
2	2	-7	9	10
3	2	-7	7	7
4	2	-7	6	8
0	3	-7	22	24
1	3	-7	32	31
2	3	-7	16	16
3	3	-7	11	11
4	3	-7	14	12
0	4	-7	4	5
1	4	-7	3	3
2	4	-7	2	3
3	4	-7	11	11
4	4	-7	0	3
0	5	-7	17	18
1	5	-7	40	42
2	5	-7	16	16
3	5	-7	10	10
4	5	-7	13	13
0	6	-7	8	8
1	6	-7	11	12
2	6	-7	3	3
3	6	-7	3	3
4	6	-7	4	3
0	7	-7	9	10
1	7	-7	20	22
2	7	-7	12	14
3	7	-7	8	9
4	7	-7	8	9
0	8	-7	5	4
1	8	-7	8	9
2	8	-7	1	4
3	8	-7	0	2
0	9	-7	18	19
1	9	-7	12	12
2	9	-7	5	7
3	9	-7	7	6
0	10	-7	0	0
1	10	-7	2	1
2	10	-7	4	2
0	11	-7	9	9
1	11	-7	9	8
2	11	-7	10	10
0	12	-7	5	3

Table 30. (Continued)

H	K	L	FO	FC
0	0	-6	49	54
1	0	-6	60	58
2	0	-6	17	17
3	0	-6	29	29
4	0	-6	10	10
5	0	-6	16	15
0	1	-6	8	7
1	1	-6	7	7
2	1	-6	7	7
3	1	-6	14	13
4	1	-6	7	7
5	1	-6	3	2
0	2	-6	14	15
1	2	-6	31	31
2	2	-6	30	30
3	2	-6	22	21
4	2	-6	10	10
5	2	-6	10	9
0	3	-6	21	22
1	3	-6	9	8
2	3	-6	2	4
3	3	-6	3	5
4	3	-6	0	1
5	3	-6	2	3
0	4	-6	28	29
1	4	-6	32	33
2	4	-6	26	26
3	4	-6	2	2
4	4	-6	16	15
0	5	-6	3	3
1	5	-6	12	13
2	5	-6	7	8
3	5	-6	1	3
4	5	-6	0	0
0	6	-6	8	9
1	6	-6	30	32
2	6	-6	32	34
3	6	-6	7	8
4	6	-6	13	13
0	7	-6	6	7
1	7	-6	6	7
2	7	-6	5	5
3	7	-6	0	4
4	7	-6	2	2
0	8	-6	12	13
1	8	-6	18	18
2	8	-6	8	8
3	8	-6	8	9
4	8	-6	8	8
0	9	-6	2	2
1	9	-6	3	3
2	9	-6	2	1
3	9	-6	4	1
0	10	-6	12	13
1	10	-6	15	15
2	10	-6	7	6
3	10	-6	9	9
0	11	-6	1	2
1	11	-6	2	1
2	11	-6	0	0
0	12	-6	7	7
1	12	-6	9	8
0	0	-5	0	0
1	0	-5	2	0
2	0	-5	0	0
3	0	-5	2	0
4	0	-5	2	0
5	0	-5	0	0
0	1	-5	27	28
1	1	-5	70	67
2	1	-5	18	18
3	1	-5	33	32
4	1	-5	16	15
5	1	-5	13	12
0	2	-5	21	22
1	2	-5	3	3
2	2	-5	13	12
3	2	-5	4	3
4	2	-5	6	6
5	2	-5	4	4
0	3	-5	24	24
1	3	-5	34	35
2	3	-5	38	38
3	3	-5	9	8
4	3	-5	4	4
5	3	-5	7	6
0	4	-5	6	6
1	4	-5	7	7
2	4	-5	9	9
3	4	-5	7	6
4	4	-5	2	2
5	4	-5	0	0
0	5	-5	17	18
1	5	-5	9	10
2	5	-5	38	40
3	5	-5	8	8
4	5	-5	14	14
0	6	-5	8	8
1	6	-5	9	9
2	6	-5	11	12
3	6	-5	3	2
4	6	-5	0	1
0	7	-5	12	13
1	7	-5	22	23
2	7	-5	35	36
3	7	-5	10	11
4	7	-5	7	8
0	8	-5	0	2
1	8	-5	11	12
2	8	-5	0	3
3	8	-5	3	5
4	8	-5	0	1
0	9	-5	16	15
1	9	-5	19	20
2	9	-5	5	5
3	9	-5	7	6
0	10	-5	2	1
1	10	-5	5	4
2	10	-5	0	0
3	10	-5	0	2
0	11	-5	4	2
1	11	-5	12	12
2	11	-5	7	7
0	12	-5	3	0
1	12	-5	0	1
2	12	-5	0	0
0	13	-5	10	9
0	0	-4	3	3
1	0	-4	70	69
2	0	-4	55	51
3	0	-4	25	23
4	0	-4	35	30
5	0	-4	13	12
0	1	-4	1	2
1	1	-4	37	34

Table 30. (Continued)

H	K	L	FO	FC	H	K	L	FC	FC	H	K	L	FO	FC
2	1	-4	5	3	2	9	-4	5	3	5	4	-3	3	3
3	1	-4	10	10	3	9	-4	0	2	0	5	-3	57	58
4	1	-4	4	4	4	9	-4	0	2	1	5	-3	17	19
5	1	-4	6	4	0	10	-4	0	1	2	5	-3	12	12
0	2	-4	4	5	1	10	-4	12	13	3	5	-3	24	25
1	2	-4	45	43	2	10	-4	12	13	4	5	-3	8	9
2	2	-4	46	43	3	10	-4	7	6	5	5	-3	9	9
3	2	-4	19	18	0	11	-4	5	4	0	6	-3	0	5
4	2	-4	8	8	1	11	-4	2	4	1	6	-3	11	12
5	2	-4	6	6	2	11	-4	0	1	2	6	-3	6	7
0	3	-4	7	7	3	11	-4	0	1	3	6	-3	4	3
1	3	-4	16	15	0	12	-4	2	3	4	6	-3	2	3
2	3	-4	17	17	1	12	-4	10	10	0	7	-3	21	21
3	3	-4	3	1	2	12	-4	8	8	1	7	-3	21	21
4	3	-4	9	9	0	13	-4	2	1	2	7	-3	31	33
5	3	-4	0	3	1	13	-4	0	0	3	7	-3	26	27
0	4	-4	42	45	0	0	-3	0	0	4	7	-3	9	9
1	4	-4	16	17	1	0	-3	1	0	0	8	-3	9	8
2	4	-4	22	23	2	0	-3	2	0	1	8	-3	5	5
3	4	-4	20	20	3	0	-3	0	0	2	8	-3	11	11
4	4	-4	3	3	4	0	-3	0	0	3	8	-3	8	9
5	4	-4	10	9	5	0	-3	0	0	4	8	-3	4	4
0	5	-4	11	11	0	1	-3	35	37	0	9	-3	15	15
1	5	-4	14	14	1	1	-3	33	31	1	9	-3	13	14
2	5	-4	0	3	2	1	-3	66	58	2	9	-3	12	13
3	5	-4	2	1	3	1	-3	28	25	3	9	-3	6	5
4	5	-4	0	0	4	1	-3	21	19	4	9	-3	5	6
5	5	-4	3	0	5	1	-3	11	11	0	10	-3	4	4
0	6	-4	21	21	0	2	-3	1	2	1	10	-3	5	4
1	6	-4	9	10	1	2	-3	36	34	2	10	-3	4	1
2	6	-4	39	41	2	2	-3	19	18	3	10	-3	3	3
3	6	-4	30	30	3	2	-3	19	19	0	11	-3	0	1
4	6	-4	7	8	4	2	-3	8	8	1	11	-3	7	6
0	7	-4	3	0	5	2	-3	0	1	2	11	-3	13	13
1	7	-4	2	2	0	3	-3	11	12	3	11	-3	7	6
2	7	-4	4	2	1	3	-3	17	19	0	12	-3	5	2
3	7	-4	0	3	2	3	-3	38	37	1	12	-3	7	5
4	7	-4	1	4	3	3	-3	26	26	2	12	-3	0	1
0	8	-4	25	26	4	3	-3	4	5	0	13	-3	10	8
1	8	-4	18	19	5	3	-3	4	3	1	13	-3	8	8
2	8	-4	18	19	0	4	-3	20	20	0	0	-2	56	56
3	8	-4	5	5	1	4	-3	2	2	1	0	-2	38	35
4	8	-4	6	6	2	4	-3	11	11	2	0	-2	30	26
0	9	-4	5	5	3	4	-3	9	8	3	0	-2	35	30
1	9	-4	8	9	4	4	-3	4	4	4	0	-2	24	24

Table 30. (Continued)

H	K	L	FO	FC	H	K	L	FC	FC	H	K	L	FO	FC
5	0	-2	16	17	3	8	-2	17	18	1	4	-1	5	3
0	1	-2	27	25	4	8	-2	5	6	2	4	-1	2	3
1	1	-2	11	10	0	9	-2	3	3	3	4	-1	3	4
2	1	-2	24	19	1	9	-2	5	7	4	4	-1	6	5
3	1	-2	0	2	2	9	-2	6	6	5	4	-1	6	0
4	1	-2	0	1	3	9	-2	2	2	0	5	-1	53	52
5	1	-2	4	3	4	9	-2	4	3	1	5	-1	53	55
0	2	-2	18	20	0	10	-2	5	5	2	5	-1	2	4
1	2	-2	39	38	1	10	-2	3	1	3	5	-1	23	24
2	2	-2	62	59	2	10	-2	7	7	4	5	-1	15	16
3	2	-2	36	35	3	10	-2	9	8	5	5	-1	6	6
4	2	-2	8	9	0	11	-2	2	0	0	6	-1	10	9
5	2	-2	0	3	1	11	-2	0	0	1	6	-1	7	7
0	3	-2	34	36	2	11	-2	0	0	2	6	-1	3	3
1	3	-2	35	36	3	11	-2	0	0	3	6	-1	11	11
2	3	-2	2	1	0	12	-2	10	10	4	6	-1	0	0
3	3	-2	5	5	1	12	-2	9	7	5	6	-1	0	3
4	3	-2	0	1	2	12	-2	12	11	0	7	-1	29	29
5	3	-2	3	2	0	13	-2	0	1	1	7	-1	29	30
0	4	-2	64	64	1	13	-2	3	2	2	7	-1	21	22
1	4	-2	12	12	0	0	-1	0	0	3	7	-1	25	26
2	4	-2	9	10	1	0	-1	2	0	4	7	-1	12	13
3	4	-2	18	18	2	0	-1	1	0	0	8	-1	4	1
4	4	-2	12	13	3	0	-1	0	0	1	8	-1	12	12
5	4	-2	4	5	4	0	-1	0	0	2	8	-1	2	3
0	5	-2	20	19	5	0	-1	0	0	3	8	-1	2	1
1	5	-2	16	15	0	1	-1	97	101	4	8	-1	4	6
2	5	-2	7	7	1	1	-1	14	14	0	9	-1	27	27
3	5	-2	7	7	2	1	-1	59	54	1	9	-1	6	6
4	5	-2	4	5	3	1	-1	34	34	2	9	-1	10	9
5	5	-2	0	2	4	1	-1	18	19	3	9	-1	10	10
0	6	-2	41	40	5	1	-1	9	10	4	9	-1	7	6
1	6	-2	16	17	0	2	-1	30	32	0	10	-1	1	2
2	6	-2	23	25	1	2	-1	34	32	1	10	-1	7	7
3	6	-2	28	29	2	2	-1	7	8	2	10	-1	2	0
4	6	-2	13	15	3	2	-1	5	6	3	10	-1	5	2
5	6	-2	6	7	4	2	-1	5	6	0	11	-1	14	12
0	7	-2	20	19	5	2	-1	6	7	1	11	-1	3	2
1	7	-2	6	6	0	3	-1	28	26	2	11	-1	12	12
2	7	-2	8	8	1	3	-1	15	15	3	11	-1	9	8
3	7	-2	6	6	2	3	-1	17	17	0	12	-1	3	1
4	7	-2	6	4	3	3	-1	24	24	1	12	-1	4	2
0	8	-2	28	28	4	3	-1	12	13	2	12	-1	5	3
1	8	-2	25	26	5	3	-1	2	4	0	13	-1	14	13
2	8	-2	22	23	0	4	-1	15	14	1	13	-1	8	7

Table 30. (Continued)

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
2	13	-1	7	5	3	7	0	8	8	1	3	1	33	33
1	0	0	52	38	4	7	0	5	6	2	3	1	22	22
2	0	0	35	29	0	8	0	25	25	3	3	1	16	17
3	0	0	32	27	1	8	0	15	15	4	3	1	15	15
4	0	0	22	21	2	8	0	13	14	5	3	1	7	8
5	0	0	8	9	3	8	0	15	16	0	4	1	15	14
0	1	0	2	0	4	8	0	14	14	1	4	1	12	12
1	1	0	8	8	0	9	0	4	0	2	4	1	13	14
2	1	0	9	10	1	9	0	10	11	3	4	1	8	7
3	1	0	1	1	2	9	0	6	7	4	4	1	3	3
4	1	0	4	4	3	9	0	1	1	5	4	1	3	4
5	1	0	4	6	4	9	0	0	2	0	5	1	51	52
0	2	0	38	40	0	10	0	30	31	1	5	1	32	32
1	2	0	7	6	1	10	0	5	5	2	5	1	26	26
2	2	0	33	35	2	10	0	6	6	3	5	1	10	10
3	2	0	35	39	3	10	0	6	7	4	5	1	14	15
4	2	0	16	19	0	11	0	1	0	5	5	1	7	8
5	2	0	4	6	1	11	0	1	2	0	6	1	10	9
0	3	0	0	0	2	11	0	5	5	1	6	1	6	6
1	3	0	21	20	3	11	0	1	2	2	6	1	3	1
2	3	0	2	2	0	12	0	17	15	3	6	1	6	6
3	3	0	14	16	1	12	0	9	7	4	6	1	7	6
4	3	0	4	5	2	12	0	8	7	5	6	1	0	1
5	3	0	1	2	0	13	0	1	0	0	7	1	28	29
0	4	0	16	17	1	13	0	1	2	1	7	1	32	32
1	4	0	56	60	2	13	0	3	1	2	7	1	29	30
2	4	0	3	3	0	0	1	0	0	3	7	1	16	17
3	4	0	12	12	1	0	1	1	0	4	7	1	11	11
4	4	0	14	14	2	0	1	0	0	0	8	1	2	1
5	4	0	7	8	3	0	1	3	0	1	8	1	4	4
0	5	0	0	0	4	0	1	3	0	2	8	1	0	3
1	5	0	13	12	5	0	1	3	0	3	8	1	0	2
2	5	0	7	7	0	1	1	97	101	4	8	1	3	2
3	5	0	6	6	1	1	1	97	93	0	9	1	27	27
4	5	0	7	7	2	1	1	12	13	1	9	1	18	18
5	5	0	0	2	3	1	1	28	29	2	9	1	1	1
0	6	0	28	27	4	1	1	20	22	3	9	1	8	8
1	6	0	61	63	5	1	1	6	9	4	9	1	9	9
2	6	0	22	23	0	2	1	30	32	0	10	1	3	2
3	6	0	20	20	1	2	1	8	7	1	10	1	5	4
4	6	0	13	14	2	2	1	6	7	2	10	1	4	5
5	6	0	4	7	3	2	1	9	9	3	10	1	6	5
0	7	0	3	0	4	2	1	2	3	0	11	1	13	12
1	7	0	11	11	5	2	1	0	1	1	11	1	15	13
2	7	0	10	11	0	3	1	27	26	2	11	1	7	6

Table 30. (Continued)

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
3	11	1	8	8	3	6	2	18	18	2	2	3	6	6
0	12	1	0	1	4	6	2	12	13	3	2	3	2	0
1	12	1	5	5	5	6	2	4	4	4	2	3	3	4
2	12	1	4	4	0	7	2	20	19	5	2	3	0	2
0	13	1	14	13	1	7	2	5	4	0	3	3	11	12
1	13	1	11	10	2	7	2	4	4	1	3	3	27	28
0	0	2	58	56	3	7	2	0	2	2	3	3	29	28
1	0	2	156	146	4	7	2	3	5	3	3	3	13	12
2	0	2	29	26	0	8	2	28	28	4	3	3	11	11
3	0	2	31	29	1	8	2	26	26	5	3	3	9	9
4	0	2	19	18	2	8	2	4	4	0	4	3	21	20
5	0	2	13	12	3	8	2	6	7	1	4	3	15	15
0	1	2	27	25	4	8	2	8	8	2	4	3	8	8
1	1	2	79	75	0	9	2	2	3	3	4	3	0	2
2	1	2	11	12	1	9	2	7	8	4	4	3	0	2
3	1	2	9	6	2	9	2	6	7	5	4	3	0	2
4	1	2	0	0	3	9	2	2	3	0	5	3	60	58
5	1	2	2	1	4	9	2	4	0	1	5	3	34	34
0	2	2	19	20	0	10	2	6	5	2	5	3	29	30
1	2	2	46	45	1	10	2	23	22	3	5	3	11	12
2	2	2	23	22	2	10	2	8	7	4	5	3	11	12
3	2	2	17	17	3	10	2	9	9	5	5	3	7	7
4	2	2	15	14	0	11	2	2	0	0	6	3	5	5
5	2	2	9	10	1	11	2	0	1	1	6	3	1	1
0	3	2	35	36	2	11	2	0	2	2	6	3	0	0
1	3	2	0	2	3	11	2	3	4	3	6	3	7	7
2	3	2	17	17	0	12	2	11	10	4	6	3	5	5
3	3	2	7	7	1	12	2	14	13	0	7	3	21	21
4	3	2	9	10	2	12	2	8	6	1	7	3	23	24
5	3	2	2	3	0	13	2	0	1	2	7	3	26	28
0	4	2	64	64	1	13	2	3	1	3	7	3	11	12
1	4	2	24	24	0	0	3	0	0	4	7	3	8	9
2	4	2	27	26	1	0	3	3	0	0	8	3	9	8
3	4	2	5	5	2	0	3	2	0	1	8	3	6	6
4	4	2	16	16	3	0	3	0	0	2	8	3	4	4
5	4	2	7	7	4	0	3	0	0	3	8	3	5	5
0	5	2	20	19	5	0	3	3	0	4	8	3	0	0
1	5	2	0	0	0	1	3	36	37	0	9	3	15	15
2	5	2	4	5	1	1	3	99	96	1	9	3	23	23
3	5	2	5	6	2	1	3	29	29	2	9	3	7	7
4	5	2	6	5	3	1	3	19	19	3	9	3	3	3
5	5	2	0	3	4	1	3	15	14	4	9	3	6	6
0	6	2	40	40	5	1	3	11	10	0	10	3	5	4
1	6	2	33	33	0	2	3	3	2	1	10	3	4	1
2	6	2	39	41	1	2	3	34	31	2	10	3	1	0

Table 30. (Continued)

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
3	10	3	3	1	0	6	4	22	21	1	2	5	5	3
0	11	3	1	1	1	6	4	20	20	2	2	5	35	35
1	11	3	16	15	2	6	4	22	22	3	2	5	4	4
2	11	3	12	11	3	6	4	19	20	4	2	5	2	3
3	11	3	7	6	4	6	4	8	8	5	2	5	0	2
0	12	3	2	2	0	7	4	1	0	0	3	5	25	24
1	12	3	4	2	1	7	4	1	2	1	3	5	34	32
2	12	3	4	4	2	7	4	0	0	2	3	5	33	33
0	13	3	10	8	3	7	4	7	8	3	3	5	16	17
1	13	3	9	9	4	7	4	2	1	4	3	5	6	7
0	0	4	4	3	0	8	4	26	26	5	3	5	6	7
1	0	4	43	41	1	8	4	25	25	0	4	5	6	6
2	0	4	47	45	2	8	4	19	20	1	4	5	28	29
3	0	4	8	7	3	8	4	5	4	2	4	5	3	1
4	0	4	20	18	4	8	4	5	4	3	4	5	10	11
5	0	4	9	9	0	9	4	6	5	4	4	5	5	1
0	1	4	2	2	1	9	4	0	1	0	5	5	16	18
1	1	4	43	41	2	9	4	1	3	1	5	5	32	34
2	1	4	30	28	3	9	4	0	1	2	5	5	16	16
3	1	4	0	2	0	10	4	1	1	3	5	5	16	15
4	1	4	2	3	1	10	4	9	8	4	5	5	10	10
5	1	4	3	1	2	10	4	15	14	0	6	5	8	8
0	2	4	3	5	3	10	4	6	5	1	6	5	11	11
1	2	4	52	51	0	11	4	4	4	2	6	5	5	5
2	2	4	48	49	1	11	4	3	4	3	6	5	0	1
3	2	4	14	14	2	11	4	0	1	4	6	5	4	3
4	2	4	7	8	3	11	4	2	2	0	7	5	14	13
5	2	4	8	7	0	12	4	5	3	1	7	5	22	23
0	3	4	8	7	1	12	4	12	10	2	7	5	17	18
1	3	4	7	7	2	12	4	10	9	3	7	5	10	11
2	3	4	5	3	0	13	4	0	1	4	7	5	5	4
3	3	4	6	6	1	13	4	2	0	0	8	5	3	2
4	3	4	5	5	0	0	5	0	0	1	8	5	5	4
5	3	4	2	1	1	0	5	2	0	2	8	5	8	8
0	4	4	44	45	2	0	5	2	0	3	8	5	8	8
1	4	4	38	39	3	0	5	0	0	4	8	5	0	2
2	4	4	15	15	4	0	5	0	0	0	9	5	16	15
3	4	4	18	17	5	0	5	0	0	1	9	5	13	12
4	4	4	7	8	0	1	5	29	28	2	9	5	14	14
5	4	4	9	10	1	1	5	12	12	3	9	5	6	7
0	5	4	11	11	2	1	5	45	45	0	10	5	0	1
1	5	4	4	4	3	1	5	17	18	1	10	5	0	0
2	5	4	8	9	4	1	5	8	8	2	10	5	3	3
3	5	4	7	9	5	1	5	5	6	3	10	5	0	2
4	5	4	5	4	0	2	5	22	22	0	11	5	0	2

Table 30. (Continued)

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
1	11	5	3	3	2	7	6	4	4	0	5	7	18	18
2	11	5	14	12	3	7	6	2	1	1	5	7	14	15
0	12	5	0	0	4	7	6	4	5	2	5	7	9	9
1	12	5	4	5	0	8	6	14	13	3	5	7	12	12
2	12	5	0	1	1	8	6	15	15	4	5	7	4	5
0	13	5	11	9	2	8	6	15	16	0	6	7	8	8
0	0	6	55	54	3	8	6	11	11	1	6	7	5	5
1	0	6	22	21	0	9	6	0	2	2	6	7	4	2
2	0	6	7	8	1	9	6	10	9	3	6	7	1	5
3	0	6	16	16	2	9	6	0	0	4	6	7	3	1
4	0	6	4	6	3	9	6	3	2	0	7	7	11	10
5	0	6	6	5	0	10	6	14	13	1	7	7	11	11
0	1	6	8	7	1	10	6	7	6	2	7	7	15	14
1	1	6	2	2	2	10	6	7	7	3	7	7	11	11
2	1	6	11	11	3	10	6	9	8	0	8	7	5	4
3	1	6	6	6	0	11	6	4	2	1	8	7	3	2
4	1	6	0	0	1	11	6	5	4	2	8	7	3	3
5	1	6	3	3	2	11	6	3	4	3	8	7	3	3
0	2	6	15	15	0	12	6	8	7	0	9	7	20	19
1	2	6	20	20	1	12	6	0	3	1	9	7	8	9
2	2	6	37	36	0	0	7	7	0	2	9	7	10	9
3	2	6	26	26	1	0	7	0	0	3	9	7	9	8
4	2	6	8	8	2	0	7	2	0	0	10	7	2	0
0	3	6	22	22	3	0	7	2	0	1	10	7	0	3
1	3	6	18	18	4	0	7	1	0	2	10	7	3	0
2	3	6	2	3	0	1	7	42	41	0	11	7	10	9
3	3	6	4	5	1	1	7	10	9	1	11	7	4	3
4	3	6	4	5	2	1	7	17	18	0	12	7	3	3
0	4	6	29	29	3	1	7	14	13	0	0	8	61	59
1	4	6	25	25	4	1	7	9	9	1	0	8	15	16
2	4	6	25	26	0	2	7	2	2	2	0	8	10	10
3	4	6	11	12	1	2	7	12	13	3	0	8	8	7
4	4	6	9	9	2	2	7	4	5	4	0	8	6	7
0	5	6	2	3	3	2	7	11	12	0	1	8	8	8
1	5	6	4	4	4	2	7	2	1	1	1	8	8	9
2	5	6	2	2	0	3	7	23	24	2	1	8	1	1
3	5	6	6	6	1	3	7	25	25	3	1	8	0	0
4	5	6	6	5	2	3	7	29	29	4	1	8	1	0
0	6	6	10	9	3	3	7	22	22	0	2	8	27	27
1	6	6	13	13	4	3	7	9	9	1	2	8	11	11
2	6	6	19	19	0	4	7	5	5	2	2	8	17	17
3	6	6	8	8	1	4	7	0	0	3	2	8	18	16
4	6	6	5	5	2	4	7	11	11	4	2	8	10	10
0	7	6	7	7	3	4	7	4	1	0	3	8	2	2
1	7	6	1	0	4	4	7	4	5	1	3	8	0	2

Table 30. (Continued)

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
2	3	8	5	5	2	2	9	5	5	1	3	10	5	5
3	3	8	4	4	3	2	9	4	3	2	3	10	1	2
4	3	8	0	1	4	2	9	3	2	3	3	10	4	3
0	4	8	24	24	0	3	9	23	23	0	4	10	33	32
1	4	8	22	22	1	3	9	14	14	1	4	10	16	16
2	4	8	14	13	2	3	9	15	14	2	4	10	10	9
3	4	8	15	15	3	3	9	15	15	3	4	10	8	7
4	4	8	8	8	4	3	9	10	11	0	5	10	7	6
0	5	8	5	6	0	4	9	3	2	1	5	10	0	1
1	5	8	2	0	1	4	9	4	3	2	5	10	0	0
2	5	8	2	1	2	4	9	0	2	3	5	10	2	1
3	5	8	0	2	3	4	9	2	2	0	6	10	18	17
4	5	8	2	2	0	5	9	34	35	1	6	10	10	9
0	6	8	12	12	1	5	9	12	12	2	6	10	5	6
1	6	8	12	12	2	5	9	10	10	3	6	10	5	5
2	6	8	9	9	3	5	9	8	7	0	7	10	5	6
3	6	8	9	9	0	6	9	4	3	1	7	10	3	4
0	7	8	2	2	1	6	9	0	2	2	7	10	3	1
1	7	8	3	4	2	6	9	3	3	0	8	10	12	11
2	7	8	2	1	3	6	9	0	2	1	8	10	11	10
3	7	8	3	3	0	7	9	11	11	2	8	10	3	3
0	8	8	15	15	1	7	9	3	4	0	9	10	2	2
1	8	8	7	7	2	7	9	6	7	1	9	10	3	3
2	8	8	7	7	3	7	9	6	7	0	0	11	0	0
3	8	8	8	8	0	8	9	2	1	1	0	11	2	0
0	9	8	3	1	1	8	9	0	3	2	0	11	0	0
1	9	8	1	1	2	8	9	0	1	3	0	11	0	0
2	9	8	2	4	0	9	9	14	12	0	1	11	16	15
0	10	8	16	15	1	9	9	12	11	1	1	11	15	14
1	10	8	7	8	2	9	9	3	4	2	1	11	9	9
2	10	8	8	7	0	10	9	0	1	3	1	11	7	7
0	11	8	0	1	1	10	9	3	0	0	2	11	1	1
1	11	8	0	1	0	0	10	10	9	1	2	11	1	3
0	0	9	1	0	1	0	10	22	22	2	2	11	4	4
1	0	9	0	0	2	0	10	7	8	3	2	11	3	3
2	0	9	1	0	3	0	10	7	6	0	3	11	20	19
3	0	9	0	0	0	1	10	4	4	1	3	11	17	17
4	0	9	3	0	1	1	10	11	10	2	3	11	8	8
0	1	9	24	23	2	1	10	3	2	3	3	11	5	5
1	1	9	26	26	3	1	10	5	4	0	4	11	8	7
2	1	9	5	6	0	2	10	17	16	1	4	11	4	6
3	1	9	9	8	1	2	10	19	18	2	4	11	1	1
4	1	9	8	6	2	2	10	12	11	3	4	11	4	0
0	2	9	7	7	3	2	10	8	9	0	5	11	13	12
1	2	9	4	4	0	3	10	9	9	1	5	11	18	17

Table 30. (Continued)

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
2	5	11	8	8	1	2	12	14	13	2	0	13	0	0
0	6	11	3	3	2	2	12	11	10	0	1	13	9	7
1	6	11	2	2	0	3	12	0	3	1	1	13	6	7
2	6	11	0	0	1	3	12	6	6	2	1	13	8	7
0	7	11	7	7	2	3	12	3	1	0	2	13	0	1
1	7	11	9	8	0	4	12	14	12	1	2	13	4	4
2	7	11	2	4	1	4	12	18	16	2	2	13	0	2
0	8	11	5	2	2	4	12	9	8	0	3	13	12	11
1	8	11	3	1	0	5	12	2	1	1	3	13	14	13
0	0	12	11	11	1	5	12	2	0	0	4	13	4	3
1	0	12	9	10	2	5	12	0	0	1	4	13	4	4
2	0	12	8	8	0	6	12	6	4	0	5	13	4	3
0	1	12	0	2	1	6	12	8	8	0	0	14	7	6
1	1	12	0	0	0	7	12	0	2	0	1	14	4	0
2	1	12	4	3	0	0	13	0	0	0	2	14	8	7
0	2	12	11	10	1	0	13	0	0					

Table 31. Final observed and calculated structure amplitudes in electrons for Ni(DMG)$_2$

H	K	L	FO	FC
4	0	0	34	33
6	0	0	56	58
8	0	0	108	107
10	0	0	38	40
12	0	0	64	63
14	0	0	36	37
16	0	0	23	20
18	0	0	25	26
1	1	0	187	192
3	1	0	28	28
5	1	0	100	101
7	1	0	124	124
9	1	0	69	71
11	1	0	38	42
13	1	0	46	51
15	1	0	23	23
17	1	0	25	24
19	1	0	16	18
0	2	0	31	30
2	2	0	15	22
4	2	0	19	20
6	2	0	107	107
8	2	0	94	96
10	2	0	63	63
12	2	0	34	33
14	2	0	21	18
16	2	0	22	20
18	2	0	23	21
1	3	0	80	79
3	3	0	65	64
5	3	0	3	6
7	3	0	44	46
9	3	0	54	57
11	3	0	48	49
13	3	0	0	3
15	3	0	20	19
17	3	0	26	23
19	3	0	23	19
0	4	0	56	57
2	4	0	150	147
4	4	0	80	80
6	4	0	17	18
8	4	0	21	23
10	4	0	58	57
12	4	0	22	21
14	4	0	18	19
16	4	0	32	31
18	4	0	21	19
1	5	0	103	100
3	5	0	108	106
5	5	0	34	33
7	5	0	36	38
9	5	0	87	89
11	5	0	57	60
13	5	0	16	19
15	5	0	23	24
17	5	0	15	21
0	6	0	55	57
2	6	0	66	63
4	6	0	66	68
6	6	0	46	44
8	6	0	84	85
10	6	0	66	69
12	6	0	23	24
14	6	0	18	16
16	6	0	19	20
1	7	0	54	50
3	7	0	53	52
5	7	0	52	51
7	7	0	42	43
9	7	0	43	42
11	7	0	6	7
13	7	0	17	15
15	7	0	13	18
0	8	0	82	79
2	8	0	41	40
4	8	0	11	11
6	8	0	14	14
8	8	0	26	30
10	8	0	16	16
12	8	0	8	12
14	8	0	15	15
1	9	0	40	41
3	9	0	10	9
5	9	0	10	11
7	9	0	29	29
9	9	0	27	23
11	9	0	18	20
13	9	0	20	18
0	10	0	34	31
2	10	0	28	23
4	10	0	15	14
6	10	0	38	35
8	10	0	28	27
10	10	0	20	19
1	11	0	44	35
3	11	0	23	21
5	11	0	27	21
7	11	0	32	26
9	11	0	17	17
0	12	0	19	23
2	12	0	17	21
4	12	0	25	19
1	0	1	3	0
3	0	1	0	0
5	0	1	5	0
7	0	1	7	0
9	0	1	3	0
11	0	1	6	0
13	0	1	0	0
15	0	1	0	0
17	0	1	0	0
19	0	1	6	0
0	1	1	1	0
2	1	1	42	42
4	1	1	3	6
6	1	1	31	28
8	1	1	47	45
10	1	1	23	22
12	1	1	14	14
14	1	1	20	22
16	1	1	16	14
18	1	1	10	7
1	2	1	12	12
3	2	1	43	40
5	2	1	57	54
7	2	1	14	12
9	2	1	28	26
11	2	1	9	10
13	2	1	5	8
15	2	1	0	6
17	2	1	14	12
19	2	1	6	0
0	3	1	0	0
2	3	1	17	14

Table 31. (Continued)

H	K	L	FO	FC
4	3	1	46	44
6	3	1	14	15
8	3	1	0	6
10	3	1	23	24
12	3	1	6	8
14	3	1	21	20
16	3	1	10	6
18	3	1	8	0
1	4	1	34	34
3	4	1	10	7
5	4	1	13	11
7	4	1	26	25
9	4	1	12	9
11	4	1	13	11
13	4	1	0	9
15	4	1	0	10
17	4	1	0	1
0	5	1	3	0
2	5	1	39	40
4	5	1	6	8
6	5	1	19	19
8	5	1	13	13
10	5	1	12	13
12	5	1	0	9
14	5	1	9	2
16	5	1	10	5
1	6	1	11	9
3	6	1	37	39
5	6	1	12	9
7	6	1	10	1
9	6	1	15	14
11	6	1	4	4
13	6	1	11	11
15	6	1	13	1
17	6	1	12	3
0	7	1	7	0
2	7	1	16	15
4	7	1	10	7
6	7	1	13	15
8	7	1	14	16
10	7	1	6	1
12	7	1	15	2
14	7	1	12	3
16	7	1	8	4
1	8	1	0	10
3	8	1	15	16
5	8	1	7	15
7	8	1	13	8
9	8	1	11	13
11	8	1	0	1
13	8	1	0	4
0	9	1	0	0
2	9	1	17	18
4	9	1	14	13
6	9	1	14	14
8	9	1	8	4
10	9	1	13	1
12	9	1	0	0
1	10	1	10	11
3	10	1	5	8
5	10	1	7	2
7	10	1	0	0
9	10	1	13	1
11	10	1	3	7
0	11	1	0	0
2	11	1	0	7
4	11	1	0	5
6	11	1	0	2
8	11	1	0	0
1	12	1	0	3
3	12	1	9	1
0	0	2	410	402
2	0	2	140	135
4	0	2	31	33
6	0	2	65	62
8	0	2	87	87
10	0	2	40	40
12	0	2	57	54
14	0	2	38	34
16	0	2	19	18
18	0	2	28	23
1	1	2	164	157
3	1	2	29	29
5	1	2	83	81
7	1	2	106	103
9	1	2	63	63
11	1	2	38	37
13	1	2	44	45
15	1	2	24	21
17	1	2	29	21
0	2	2	34	35
2	2	2	21	23
4	2	2	24	25
6	2	2	91	89
8	2	2	84	83
10	2	2	55	54
12	2	2	29	29
14	2	2	12	16
16	2	2	17	18
18	2	2	21	19
1	3	2	65	65
3	3	2	58	58
5	3	2	6	13
7	3	2	41	41
9	3	2	52	50
11	3	2	43	44
13	3	2	0	3
15	3	2	15	17
17	3	2	23	21
0	4	2	57	60
2	4	2	118	119
4	4	2	66	67
6	4	2	5	17
8	4	2	22	23
10	4	2	49	51
12	4	2	23	20
14	4	2	5	17
16	4	2	28	27
1	5	2	83	82
3	5	2	87	92
5	5	2	29	33
7	5	2	33	35
9	5	2	76	76
11	5	2	49	53
13	5	2	21	17
15	5	2	14	21
17	5	2	19	19
0	6	2	51	53
2	6	2	56	56
4	6	2	59	59
6	6	2	36	39
8	6	2	72	74
10	6	2	61	60
12	6	2	20	22
14	6	2	10	15

Table 31. (Continued)

H	K	L	FO	FC
16	6	2	18	18
1	7	2	45	45
3	7	2	43	44
5	7	2	47	45
7	7	2	38	39
9	7	2	37	38
11	7	2	0	7
13	7	2	11	13
15	7	2	17	16
0	8	2	67	68
2	8	2	34	35
4	8	2	9	11
6	8	2	17	13
8	8	2	25	27
10	8	2	12	14
12	8	2	3	11
14	8	2	0	13
1	9	2	37	37
3	9	2	12	8
5	9	2	7	9
7	9	2	26	26
9	9	2	19	20
11	9	2	22	18
0	10	2	27	27
2	10	2	22	21
4	10	2	18	13
6	10	2	31	31
8	10	2	24	24
10	10	2	13	18
1	11	2	29	31
3	11	2	25	19
5	11	2	15	19
7	11	2	22	24
1	0	3	0	0
3	0	3	0	0
5	0	3	0	0
7	0	3	4	0
9	0	3	0	0
11	0	3	9	0
13	0	3	0	0
15	0	3	0	0
17	0	3	12	0
0	1	3	0	0
2	1	3	16	17
4	1	3	6	2
6	1	3	12	16
8	1	3	25	27
10	1	3	17	15
12	1	3	0	9
14	1	3	19	17
16	1	3	3	11
18	1	3	7	5
1	2	3	8	6
3	2	3	5	8
5	2	3	27	26
7	2	3	0	6
9	2	3	14	17
11	2	3	8	7
13	2	3	8	6
15	2	3	12	6
17	2	3	0	9
0	3	3	7	0
2	3	3	3	3
4	3	3	19	23
6	3	3	7	10
8	3	3	5	3
10	3	3	16	18
12	3	3	6	6
14	3	3	17	14
16	3	3	7	5
1	4	3	18	19
3	4	3	5	5
5	4	3	8	6
7	4	3	15	17
9	4	3	9	6
11	4	3	6	7
13	4	3	0	7
15	4	3	0	8
17	4	3	0	1
0	5	3	0	0
2	5	3	26	23
4	5	3	0	4
6	5	3	11	12
8	5	3	10	10
10	5	3	9	10
12	5	3	13	6
14	5	3	11	2
16	5	3	3	4
1	6	3	7	4
3	6	3	22	25
5	6	3	8	8
7	6	3	0	1
9	6	3	11	11
11	6	3	0	3
13	6	3	4	9
15	6	3	12	1
0	7	3	0	0
2	7	3	11	10
4	7	3	7	5
6	7	3	9	11
8	7	3	15	11
10	7	3	3	1
12	7	3	0	3
14	7	3	0	3
1	8	3	10	7
3	8	3	15	13
5	8	3	6	10
7	8	3	0	6
9	8	3	11	10
11	8	3	7	0
0	9	3	0	0
2	9	3	14	13
4	9	3	5	9
6	9	3	11	10
8	9	3	0	3
10	9	3	0	1
1	10	3	13	9
3	10	3	0	7
5	10	3	9	2
7	10	3	11	1
0	11	3	5	0
2	11	3	8	6
4	11	3	9	3
0	0	4	203	194
2	0	4	86	84
4	0	4	28	30
6	0	4	48	47
8	0	4	56	57
10	0	4	33	32
12	0	4	40	37
14	0	4	24	26
16	0	4	18	14
1	1	4	96	94
3	1	4	28	28
5	1	4	51	51

Table 31. (Continued).

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
7	1	4	64	65	3	7	4	26	30	8	3	5	0	1
9	1	4	44	44	5	7	4	28	32	10	3	5	7	11
11	1	4	29	27	7	7	4	27	30	12	3	5	10	4
13	1	4	31	31	9	7	4	27	28	14	3	5	15	8
15	1	4	14	16	11	7	4	17	6	1	4	5	15	9
0	2	4	32	34	0	8	4	46	46	3	4	5	0	3
2	2	4	27	25	2	8	4	21	24	5	4	5	0	4
4	2	4	24	25	4	8	4	16	9	7	4	5	0	8
6	2	4	56	56	6	8	4	13	11	9	4	5	0	3
8	2	4	57	54	8	8	4	24	20	11	4	5	0	3
10	2	4	38	37	10	8	4	14	11	13	4	5	11	5
12	2	4	18	20	1	9	4	31	28	0	5	5	8	0
14	2	4	16	12	3	9	4	9	7	2	5	5	12	10
16	2	4	12	14	5	9	4	10	7	4	5	5	5	0
1	3	4	42	43	7	9	4	21	19	6	5	5	0	5
3	3	4	42	41	0	10	4	12	21	8	5	5	14	7
5	3	4	16	17	2	10	4	12	17	10	5	5	7	7
7	3	4	30	30	4	10	4	12	10	12	5	5	5	4
9	3	4	34	35	1	0	5	5	0	1	6	5	7	1
11	3	4	33	31	3	0	5	11	0	3	6	5	10	13
13	3	4	0	3	5	0	5	0	0	5	6	5	0	6
15	3	4	9	12	7	0	5	3	0	7	6	5	0	1
0	4	4	45	45	9	0	5	0	0	9	6	5	0	8
2	4	4	72	73	11	0	5	0	0	11	6	5	0	2
4	4	4	41	44	13	0	5	0	0	0	7	5	12	0
6	4	4	16	15	15	0	5	7	0	2	7	5	8	4
8	4	4	18	20	0	1	5	0	0	4	7	5	0	3
10	4	4	36	37	2	1	5	13	11	6	7	5	8	6
12	4	4	14	17	4	1	5	0	3	8	7	5	11	6
14	4	4	8	12	6	1	5	9	7	10	7	5	0	2
1	5	4	54	55	8	1	5	11	12	1	8	5	3	3
3	5	4	59	60	10	1	5	3	7	3	8	5	10	7
5	5	4	26	26	12	1	5	13	4	5	8	5	8	5
7	5	4	23	26	14	1	5	10	10	7	8	5	8	3
9	5	4	50	51	1	2	5	5	6	0	9	5	8	0
11	5	4	36	38	3	2	5	10	1	2	9	5	11	8
13	5	4	15	14	5	2	5	13	10	4	9	5	10	5
0	6	4	37	40	7	2	5	6	3	0	0	6	90	92
2	6	4	37	40	9	2	5	11	10	2	0	6	45	44
4	6	4	39	41	11	2	5	0	3	4	0	6	18	19
6	6	4	26	30	13	2	5	0	3	6	0	6	29	27
8	6	4	50	50	0	3	5	0	0	8	0	6	35	34
10	6	4	41	41	2	3	5	10	2	10	0	6	20	20
12	6	4	8	16	4	3	5	5	7	12	0	6	25	22
1	7	4	33	32	6	3	5	0	2	1	1	6	46	49

Table 31. (Continued)

H	K	L	FO	FC	H	K	L	FO	FC	H	K	L	FO	FC
3	1	6	23	20	4	4	6	19	25	7	0	7	0	0
5	1	6	27	28	6	4	6	14	10	0	1	7	11	0
7	1	6	36	36	8	4	6	19	13	2	1	7	0	8
9	1	6	26	27	10	4	6	18	24	4	1	7	0	2
11	1	6	21	16	1	5	6	34	33	6	1	7	3	3
0	2	6	21	22	3	5	6	33	34	8	1	7	6	4
2	2	6	21	18	5	5	6	21	17	1	2	7	11	5
4	2	6	20	17	7	5	6	17	16	3	2	7	12	1
6	2	6	31	31	9	5	6	29	29	5	2	7	0	3
8	2	6	31	30	0	6	6	26	25	7	2	7	12	1
10	2	6	22	21	2	6	6	26	24	0	3	7	11	0
1	3	6	23	24	4	6	6	23	24	2	3	7	0	1
3	3	6	26	24	6	6	6	9	19	4	3	7	5	0
5	3	6	11	13	1	7	6	15	20	6	3	7	3	1
7	3	6	23	18	3	7	6	14	17	1	4	7	7	4
9	3	6	26	21	5	7	6	15	19	3	4	7	10	2
11	3	6	24	18	1	0	7	5	0	5	4	7	13	3
0	4	6	23	26	3	0	7	0	0	0	5	7	13	0
2	4	6	38	39	5	0	7	0	0	2	5	7	0	4

XIII. APPENDIX C

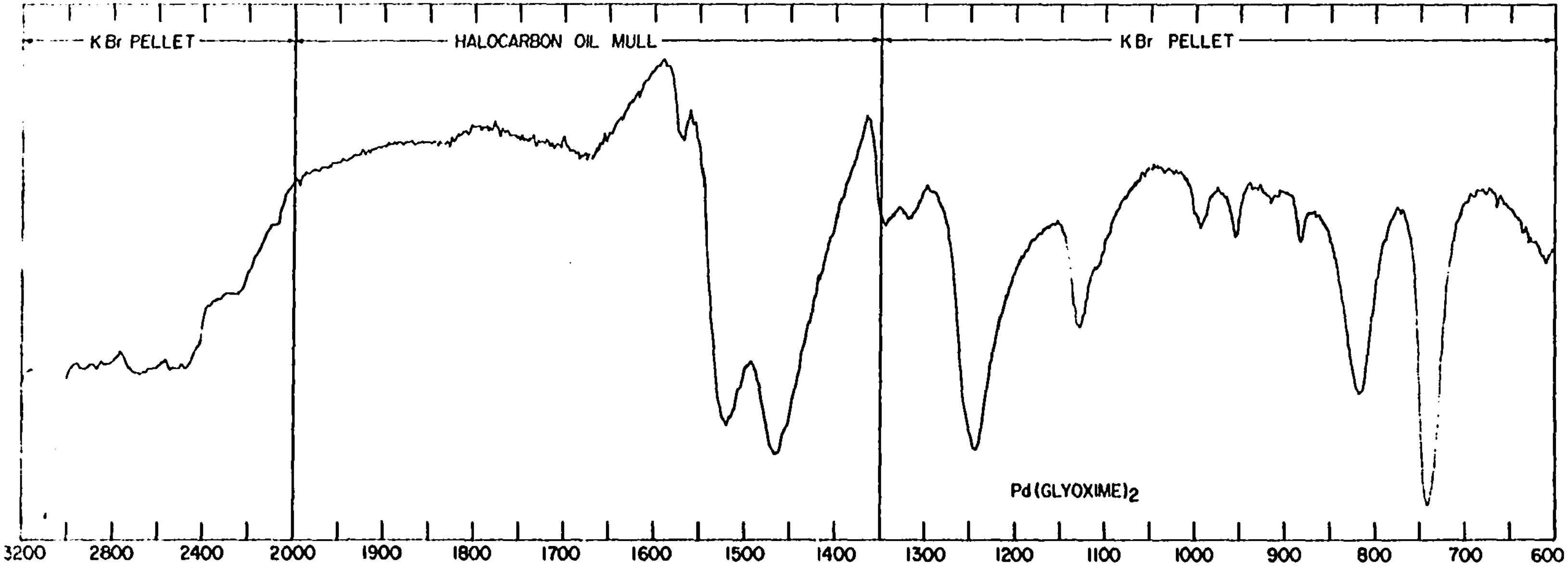

Figure 17. Infrared spectrum of Pd(G)$_2$

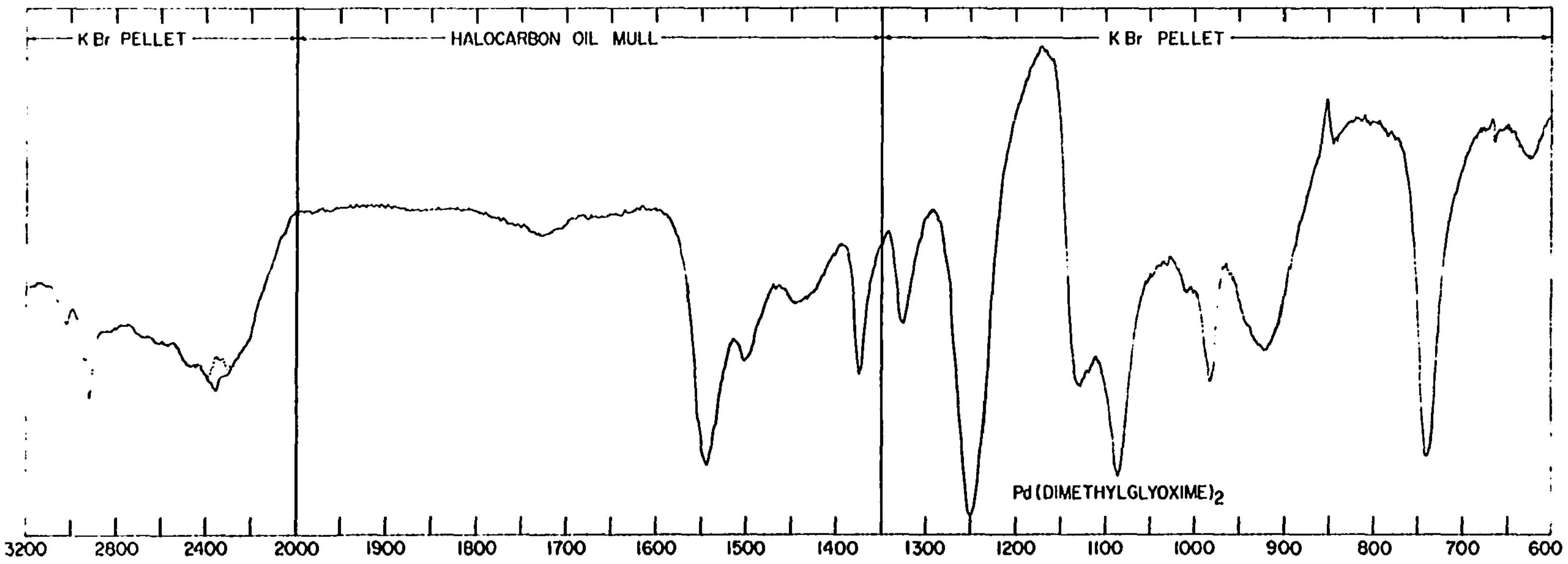

Figure 18. Infrared spectrum of $Pd(DMG)_2$

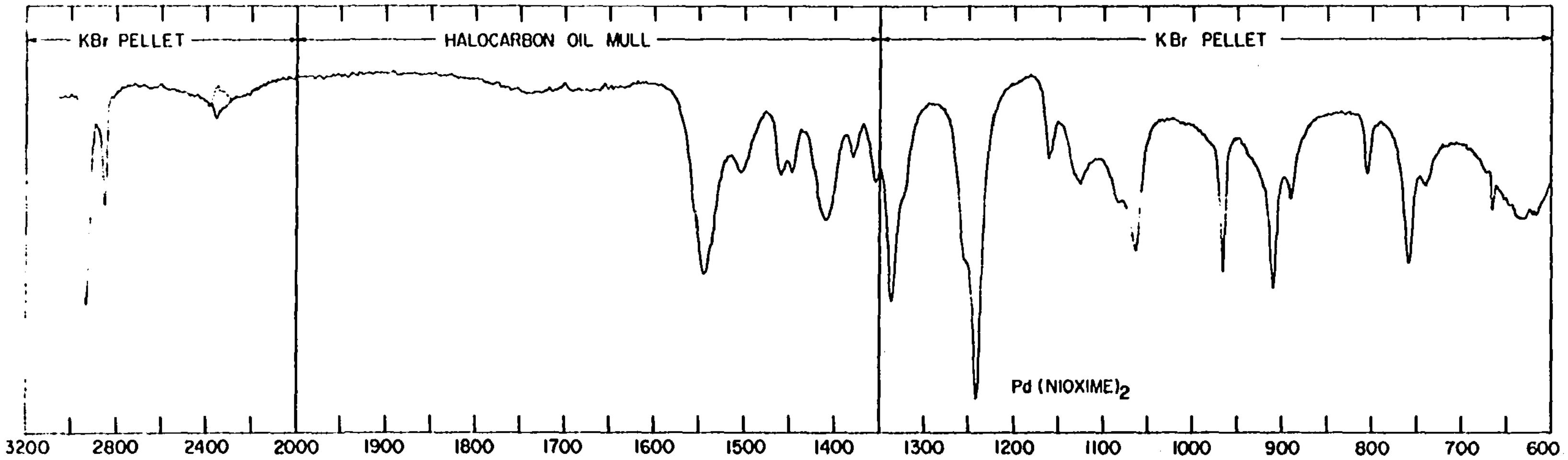

Figure 19. Infrared spectrum of Pd(Niox)$_2$

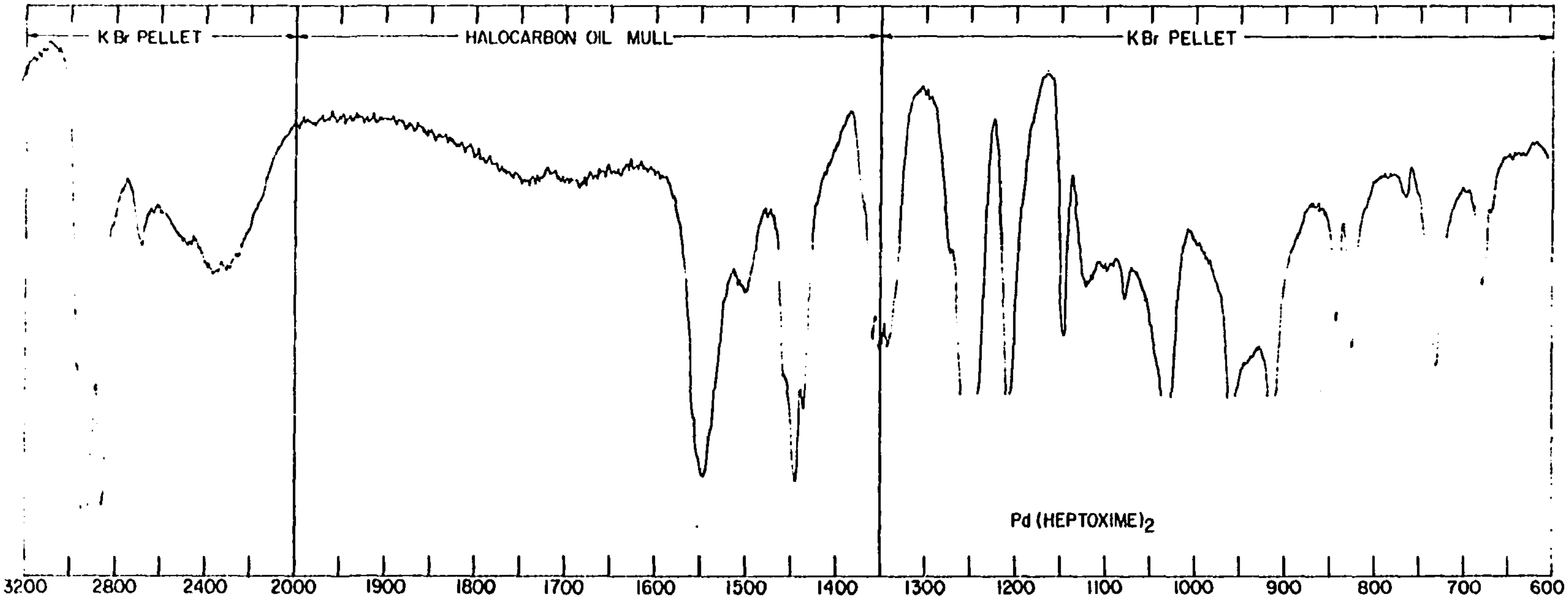

Figure 20. Infrared spectrum of Pd(Heptox)$_2$

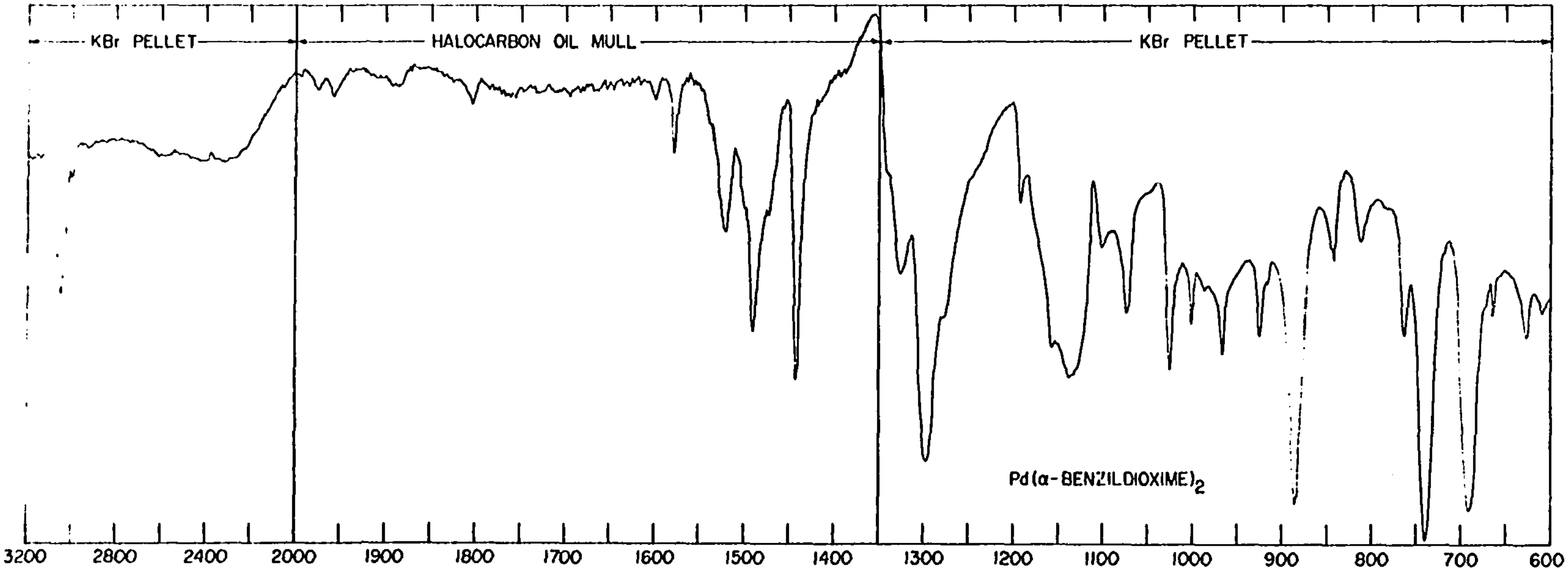

Figure 21. Infrared spectrum of Pd(α-Benzil)$_2$

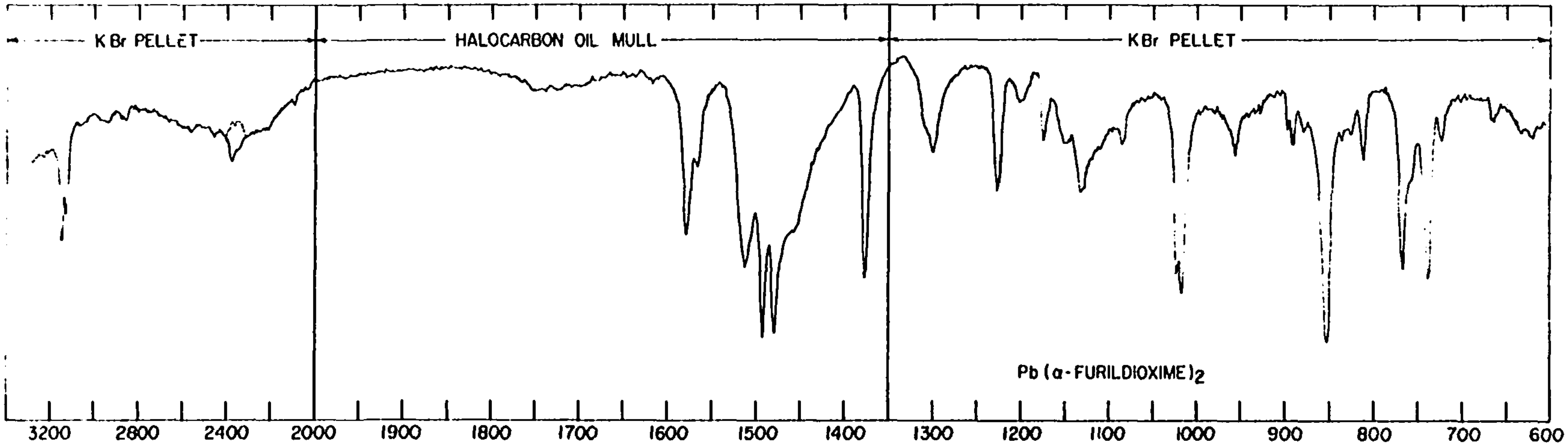

Figure 22. Infrared spectrum of Pd(α-Furil)$_2$