CHEMISTRY

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ABSTRACTS

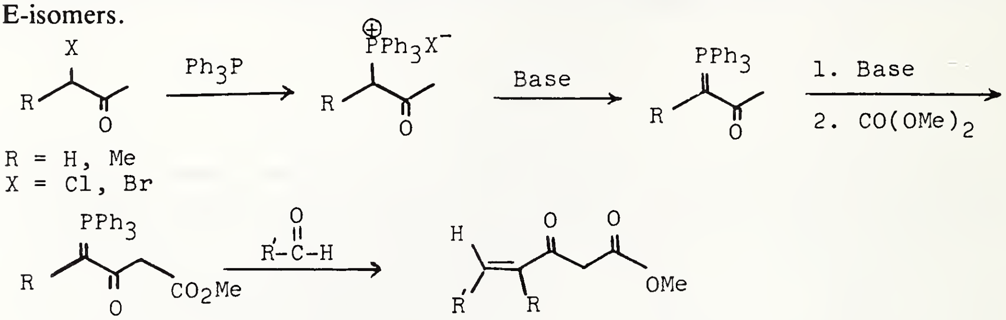
Ambidentate Phosphine Ligands: Phosphine-amine and Phosphine-imidate Complexes of Tungsten. Sepehra Akhavan, Kristen Faust and Bruce Storhoff, Department of Chemistry, Ball State University, Muncie, Indiana 47306.——The reaction of $Ph_2PCH_2CH(R)CN$ (R = CH_3,H) (L) with W(CO)6 with an excess of $NaBH_4$ in dry ethanol provides excellent yields of *cis*-coordinated $(CO)_4W[Ph_2PCH_2CH(R)CH_2NH_2]$ and $(CO)_4W[Ph_2PCH_2CH(R)C(OC_2H_5)NH$. The latter are converted to the corresponding phosphine-amine complexes upon reaction with additional $NaBH_4$. These results are rationalized in terms of a reaction scheme involving a side-on coordinated nitrile group which is susceptible to nucleophilic attack by ethanol. The coordinated amine and imidate groups are replaced by PMe_2Ph providing mixtures of *cis* and *trans* complexes.

The Synthesis of a Crown Ether that May Exhibit Metal Cation Enhanced Fluorescence. Stasia A. Barnell, Beth E. Beeson and Lynn R. Sousa, Department of Chemistry, Ball State University, Muncie, Indiana 47306.——A crown ether molecule that contains both a fluorescent chromophore and a potential quencher of that chromophore's fluorescence is being synthesized. The synthesis is convergent and involves several steps.

Based on literature data concerning the steric requirements for the quenching of fluorescent singlet states and our understanding of metal cation complexation by crown ethers, it is probable that some metal cations (Na^+, K^+, Ca^{+2}, etc.) will enhance the fluorescence of our crown ether compound. Such a "fluorogenic" crown ether could prove useful for the quantitative analysis of selected metal cations.

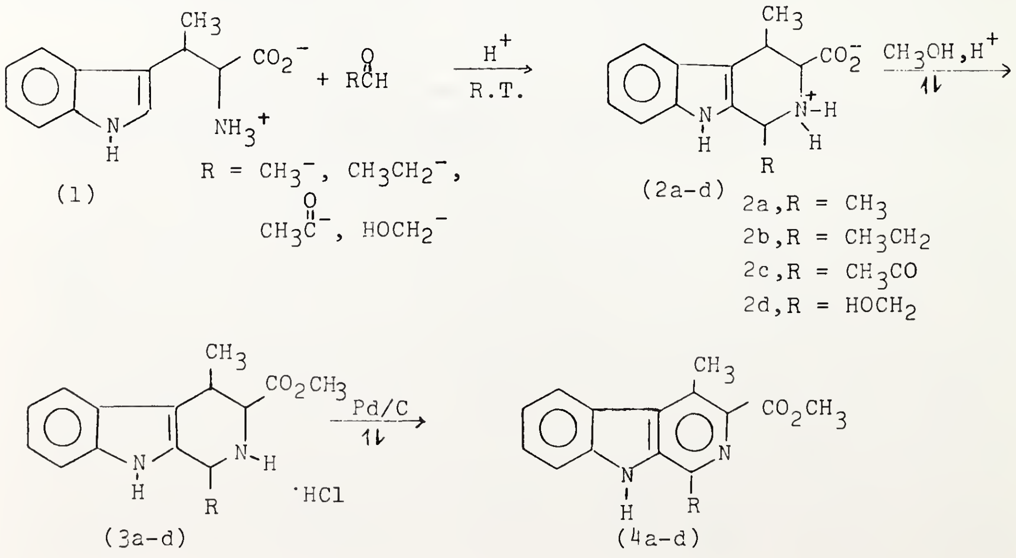
2,4-Dinitrophenylhydrazones: A Modified Method for the Preparation of these Derivatives and an Explanation of Previous Conflicting Results. Mohammad Behforouz, Joseph L. Bolan and Michael S. Flynt, Department of Chemistry, Ball State University, Muncie, Indiana 47306.——We have found that the conventional methods for forming 2,4-dinitrophenylhydrazones (2,4-DNPs) usually leave traces of acids complexed with the derivatives and that this has been the major cause for the melting point discrepancies and controversy throughout the 50 year history of their application. A simple modification of the original method, a bicarbonate wash of the 2,4-DNP crystals, removes the acid and reproducibly gives derivatives with previously reported or higher melting ranges. A series of aldehydes and ketones was selected and the 2,4-DNPs were prepared by both the conventional and the modified methods. In nearly all cases the modified method gave products with higher melting ranges. 2,4-DNPs of several hydroxy ketones previously unattainable by the standard method were also prepared. Careful studies of the 2,4-DNPs of acetaldehyde and 3-hydroxy-3-methyl-2-butanone by NMR spectroscopy and differential scanning calorimetric analysis showed that traces of acids incorporated in the crystals catalyze the interconversion of the syn and anti forms of the 2,4-DNPs and promote the dehydration of the hydroxycarbonyl derivatives thus lowering or changing the melting behaviors of the products.

Wittig Reaction: Stable Ylides in the Preparation of γ,δ-unsaturated-β-Ketoesters. MOHAMMAD BEHFOROUZ AND K.E. MENNEN, Department of Chemistry, Ball State University, Muncie, Indiana 47306.——Stable ylides of β-ketoesters are prepared and their condensations with aromatic and aliphatic aldehydes to give γ,δ-unsaturated-β-ketoesters are discussed. These Wittig reactions are sterospecific and give mainly the E-isomers.



Synthesis of β-Carbolines Derived from 2-Amino-3-(3-indolyl)-butyric Acid (β-Methyltryptophan). MOHAMMAD BEHFOROUZ AND M.E. OGLE, Department of Chemistry, Ball State University, Muncie, Indiana 47306.——Although β-carbolines are common structural units and their chemistry has been well documented, their preparations from β-methyltryptophan (1) have never been reported. Four aldehydes were reacted with β-methyltryptophan via the Pictet-Spengler reaction to form the corresponding salts of tetrahydro-β-carbolines (2a-d). The formation and subsequent esterification and dehydrogenation of these compounds were studied.

An example of such a synthetic route involves the reaction of acetaldehyde with β-methyltryptophan in aqueous sulfuric acid at 25° to yield 2a-d. The resulting salt (2a) was filtered, dried, and dissolved in saturated methanolic HCl and refluxed under nitrogen to yield the salt 3a-CHl. The salt was converted to the free base on treatment with 14% NH_4OH to give 3a. The resulting free base was refluxed over 10% Pd/C in dioxane to yield 4a. Reactions involving the other aldehydes were conducted in an identical fashion.



Coulometric Titrations: Low Cost Alternatives for Computer Controlled Titrations. STANLEY L. BURDEN AND PHILLIP W. SCHULTZ, Department of Chemistry, Taylor University, Upland, Indiana 46989.——Software and hardware for several different system configurations to carry out coulometric titrations under control of an Apple II or IIe computer will be presented. One of the main advantages of the coulometric approach is the elimination of costly titrant delivery systems. The systems described will accommodate a variety of budget and equipment limitations. The simplest systems use, in addition to the Apple and pH meter with BCD readout, only a battery, electrodes and simple interfacing costing less than $50 to construct. Such a system is useful for classroom demonstrations or experiments in which shapes of titration curves are the primary data of interest as opposed to highly accurate end point determinations. The software presented will plot the data collected in real time and will compute and display first and second deratives as well as Gran plots in different colors using the high resolu-resolution graphics. A somewhat more expensive system uses operational amplifiers for the constant current source and a relay and timer, controlled by the computer, to stop the titration at a user selected endpoint potential or pH. The software for this system also permits specifying a pH or potential at which current will begin to be delivered in short pulses, with user selected intervals between pulses, to permit the endpoint to be approached slowly. Applications of the use of these systems and typical data will be presented.

Temperature Dependent Infrared Studies of the Hydrogen Bonding in Aliphatic Alcohols. MARK CISNEROS AND JOE KIRSCH, Department of Chemistry, Butler University, Indianapolis, Indiana 46208.——The extent of the hydrogen bonding of aliphatic alcohols in dilute solutions has been studied through the use of temperature dependent infrared spectroscopy. The equilibrium constants for the hydrogen bonding process have been determined as a function of temperature. The enthalpy and entropy for the hydrogen bonding process has been calculated from the temperature dependent equilibrium constants. The equilibrium constants, enthalpies, and entropies for the hydrogen bonding process are related to the molecular structure of the alcohols and the steric hindrance at the hydrogen bonding site.

Spectra and Equilibria of the Thiocyanato Complexes of Copper (I) in Aqueous Solution. SALLY K. DOTTERER AND KENNETH L. STEVENSON, Department of Chemistry, Indiana University-Purdue University at Fort Wayne, Fort Wayne, Indiana 46805.——The objective of this research was to determine the spectra of the copper (I)-thiocyanato complexes and recheck the published value of the equilibrium constant for the reaction:

$$Cu(SCN)_3^{2-} + SCN^- = Cu(SCN)_4^{3-}$$

According to Ahrland and Tageson, there are normally three complexes present in aqueous solutions of CuSCN and SCN^-: $Cu_2(SCN)_6^{4-}$, $Cu(SCN)_3^{2-}$, and $Cu(SCN)_4^{3-}$. Of these, based on the reported values for equilibrium constants, only $Cu(SCN)_3^{2-}$ and $Cu(SCN)_4^{3-}$ are present in significant concentrations. To determine the spectrum of each complex in the equilibrated, anaerobic solution, spectra were run on solutions of copper (I) thiocyanate, at a constant ionic strength of 5.0M ($NaClO_4$ medium) while varying the thiocyanate concentration between 0.2M and 5.0M. The most significant data appeared at thiocyanate concentrations lower than 1.0M, but the very low solubility of CuSCN at these thiocyanate concentrations caused difficulty in obtaining these data. This was done by filtering saturated CuSCN solutions, taking the spectra, and determining the copper concentration with atomic absorption spectrophotometry. At thiocyanate concentrations above 2.0M, the copper concentration was maintained at 0.005M.

From the spectra, the molar extinction coefficient was calculated at a specific wavelength for each solution, and, using a computer technique, an attempt was made at recalculating the value for the equilibrium constant. From this value, the spectra of each of the two complexes can be determined.

Steric and Electronic Effects upon *cis:trans* Distributions in $W(CO)_4(L)(L')$ Complexes when L and L′ are Phosphorus Ligands. JENNIFER L. DYKE AND JOHN A. MOSBO, Department of Chemistry, Ball State University, Muncie, Indiana 47306.———The tungsten complexes $W(CO)_4(L)(Py)$ (Py = pyridine and L = PPh_2Et or $PPhMe_2$) have been reacted with two series of electronically and sterically divergent ligands, $PPh_x(OMe)_{3-x}$ and $PPH_x(NMe_2)_{3-x}$ (where x = 0, 1 or 2). *Cis:trans* ratios of the $W(CO)_4(L)(L')$ products decrease in the order $x = 0 > 1 > 2$ for the OMe-containing series, but increase with the number of phenyl groups for the MNe_2-containing ligands. These results are consistent with increasing *trans* preference as the size of L′ increases, but are also consistent with increasing *trans* preference as the Tolman electronic parameter (ν) decreases.

A Simple, Reproducible High Performance Liquid Chromatography Separation of Amino Acids with Picomole Sensitivity. BERNICE ELLIS, KEVIN COOKSY, JAMES M. ANDERSON, AND HARRY W. JARRETT, Department of Biology, Indiana University-Purdue University at Indianapolis, Indianapolis, Indiana 46223 and Alltech Associates/Applied Science Labs, Deerfield, Illinois 60015.———A method for separating and quantitating the amino acids commonly found in protein acid hydrolysates has been developed. The amino acids are derivatized using o-phthaldehyde reagent and separated using reverse phase by a method similar to published reports. Our method differs from these in that it uses a C8 reverse phase column, 0.1% triethylamine in the buffers to improve the peak shape of HIS, and achieves baseline separation of all amino acids in 16 min total analysis time. The column (4 x 15 cm) used is inexpensive and various lots of the column have been shown to behave identically. The limits of detection are in the 10^{-12} to 10^{-14} mole range. The same column has been used for over 100 analyses without a guard column or any special precautions with no degradation of performance noted. The detailed method and factors which influence resolution will be discussed.

An Electron Spin Resonance Method for the Measurement of Liposomal Leakage. MAUREEN L. HILL, PATRICK GALLAGHER AND JEFF MACRI, Department of Medical Research, Methodist Hospital of Indiana, Inc., 46202, F.W. KLEINHANS, Department of Medical Research, Methodist Hospital of Indiana Inc., and Department of Physics, Indiana University-Purdue University at Indianapolis, Indianapolis, Indiana 46223.———The effectiveness of different compositions of liposomes as drug delivery systems is dependent on their leakage properties. Diffusion into multilamellar liposomes was examined by recording the increase in ESR signal amplitude as the spin label, 3-carboxy-Proxyl, diffused into the lipid vesicle. The external spin label signal was quenched with chromium oxalate. Values for leakage into vesicles obtained via ESR were compared with studies of leakage out of vesicles by the conventional radioactive tracer method using $^{99m}TcO_4$. Both radioactive tracer and spin label have a negative one charge and comparable molecular weights of 163 and 169, respectively. They yielded qualitatively similar results; however, the ESR data exhibited significantly less scatter of ± 2% vs. ± 5% with the radioactive tracer method. The data exhibit a fast initial rate of leakage followed by a slow long-term component. This does not fit a simple Fick's Law process. It suggests a distribution of liposomal diffusion rates or sizes, thus a nonhomogeneous system. Membrane compositions considered included distearoyl, dipalmitoyl, and dimystearoyl phosphatidylcholines with varying ratios of cholesterol. ESR is superior to the radioactive method as it decreases scatter, eliminates the hazards of working with radioactive materials, and also eliminates the difficulties associated with disposal of the radioactive wastes.

Hindered Ligand Systems: Structure of the *cis,cis*-1,3,5-Tris(pyridine-2-carboxaldimine) cyclohexane Complexes of Fe(II) and Ni(II) Ions. J.C. HUFFMAN, R.A.D. WENTWORTH, W.E. STREIB AND C.J. HUFFMAN, Molecular Structure Center, Department of Chemistry, Indiana University, Bloomington, Indiana 47405.——Structures of the perchlorate salts of the title compounds have been determined by single crystal X-ray crystallography. The Fe(II) complex is monoclinic, space group Cc with a = 17.230(10), b = 9.729(4), c = 16.061(7)A, beta – 104.79(2)° (at –130 C.); D_{calc} = 1.662 gm/cm^3 for Z = 4. The Ni(II) complex is cubic, space group $P2_13$ with a = 14.337(6)A (at 20 C.) and D_{calc} = 1.474 gm/cm^3 for Z = 4. The Fe(II) complex has approximate octahedral coordination while the Ni(II) complex lies intermediate between the trigonal prismatic coordination previously found for the Zn(II) and Co(II) complexes and that of the Fe(II) ion. A detailed comparison is made of the inter- and intramolecular distortions present in this unusual system.

Robots in the Chemistry Laboratory, Part I: A High Speed RS-232C Serial Communications Link for Controlling a HERO I Robot from an Apple II Plus Microcomputer. NATHAN E. KASTELEIN, PHILLIP E. KLUNZINGER, EDWARD J. CIESLA, CLAUDIA RISHAW, CYNTHIA L. ROTH AND STANLEY L. BURDEN. Digital Equipment Corporation, St. Louis, Missouri and the Department of Information Science, Physics and Chemistry, Taylor University, Upland, Indiana 46989.——Small table-top robots are beginning to be used in the analytical chemistry laboratory to minimize routine and mundane sample preparation tasks. Although its capabilities are not well-suited for many laboratory tasks, the low price and variety of sensors on the HERO I robot make it attractive for introducing laboratory robotics to undergraduate students. As the first phase of developing an instructional laboratory robotics system, a method of minimizing the constraints of limited memory and inconvenient programming associated with the HERO I was needed. To overcome these limitations, an RS-232C communications interface which runs at 9600 baud has been designed and installed in a HERO I robot. The interface allows the robot to be controlled via a single RS-232C cable from any computer with RS-232C output capability. The interface and associated cable can be constructed for approximately $60. In our application, an Apple II Plus microcomputer was used to write programs and then download them, one command at a time, to the robot over the serial communications line. This presentation will focus on the hardware of the communications link. The initial phase of a robotized solution preparation system was implemented as the first application of this system.

Robots in the Chemistry Laboratory, Part II: Software for Controlling a HERO I Robot from an Apple II Plus Microcomputer via a High Speed RS-232C Communications Link. NATHAN E. KASTELEIN, PHILLIP E. KLUNZINGER, EDWARD J. CIESLA, CYNTHIA L. ROTH, CLAUDIA RISHAW AND STANLEY L. BURDEN. Digital Equipment Corporation, St. Louis, Missouri, and the Department of Information Science, Physics and Chemistry, Taylor University, Upland, Indiana 46989.——Software has been written for both the HERO I robot and the Apple II Plus microcomputer which enables programs to be written on the Apple and downloaded to the robot over an RS-232C line operating at 9600 baud. A short assembly language routine which is loaded into the robot memory from a cassette tape enables the more lengthy command receiving and interpreting routines to be downloaded frcm the Apple. To activate the robot, commands are sent from the Apple to the robot as a sequence of escape characters which indicate the appropriate motor to activate as well as the direction, speed and extent of movement. Commands are sent and executed by the robot individually. Typical command formats and capabilities will be discussed. Software was also written for the HERO I robot which will accept these escape sequences, acknowledge to the Apple the reception of the command and activate the appropriate motor. Since robot movement is much slower than transmission time plus interpretation time, the next interpreted command is always available before the robot needs it and the robot motion occurs just as if all of the commands were residing in the on-board robot memory. This presentation will focus on the software involved in this system.

Reaction Sequence Alteration in the Acetoacetic Ester Synthesis of Ketones. RICHARD A. KJONAAS, Department of Chemistry, Indiana State University, Terre Haute, Indiana 47809.———The acetoacetic ester synthesis of ketones is a very important method not only of making ketones, but also of making new carbon-carbon bonds. This classical method and its well known modifications, such as the use of β-ketosulfoxides and β-ketosulfones, require hydrolysis, saponification, aluminum amalgam reduction, or other such treatment to remove the stabilizing group after alkylation has been achieved. Thus, these methods cannot be used with substrates that are sensitive to these post-alkylation treatments. We have found at ISU, however, that deprotonation of acetoacetic acid with two equivalents of base, followed by alkylation, gives an adduct which readily decarboxylates *in situ* to give good yields of methyl ketones. This method, which is essentially an alteration of the reaction sequence of the acetoacetic ester synthesis of ketones, provides a way of achieving the same goals as that synthesis but without carrying the organo halide moiety through harsh reaction conditions.

Functionalized Crown Ethers. LEROY KROLL AND BRUCE STORHOFF, Ball State University, Muncie, Indiana 47306.———The previously reported 15-crown-5 ethers functionalized with -CH_2OH or -CH_2O-$CH_2CH=CH_2$ have been studied as potential starting materials for phosphine- and phosphinite-crown ethers. In accord, the reaction of 15-crown-5-CH_2OPPh_2 which has been identified by spectroscopic measurements. In contrast, the corresponding phosphine derivative, 15-crown-5-CH_2PPh_2 has yet to be identified from the reaction between 15-crown-5-CH_2OTs and Ph_2P^-. The reaction of 15-crown-5-$CH_2OCH_2CH=CH_2$ with 9-BBN has also been investigated. This yields a surprisingly water soluble compound which has been tentatively identified as the corresponding alcohol, 15-crown-5-$CH_2OCH_2CH_2CH_2OH$.

A Trace Metal Analysis of Coal and Acid Rain. STEVE NEWNAM AND JAMES P. RYBARCZYK, Department of Chemistry, Ball State University, Muncie, Indiana 47306.———In an attempt to determine the origins of acid precipitation collected in Central Indiana, a thorough trace metal analysis was performed on the samples provided by volunteer members of the Central Indiana District 656 of the International Rotary Club. The 2000 samples from this study were analyzed for such metal ions as Ca, Mg, Na, K, Fe, V, Mn, etc., on a graphite furnace atomic absorption system and an inductively coupled plasma. The ratios between the amounts of these various trace metals found in the samples were found to be related to seasonal, geographical, and pH variations and thus provide an indirect means of "tracing" the source of the metals. With the volunteer assistance of numerous electric power companies, coal samples were obtained from various geographic Midwest locations. These coal samples were thoroughly digested in a modified procedure and then analyzed for their trace metal content. Utilizing the variable geographical and meteorological data for the acid rain samples, the metal ratios from the coal samples were compared to those measured from the acid rain.

Conclusion of Acid Rain Monitoring in Central Indiana. LAURA POKORNEY AND JAMES P. RYBARCZYK, Department of Chemistry, Ball State University, Muncie, Indiana 47306.——With the volunteer assistance of the Central Indiana District 656 of the International Rotary Club, thirty precipitation collection stations have been in operation for the past two years. This collection process has just been concluded with over 2000 samples being analyzed for pH, conductivity, depth, trace metal ion and anion concentrations. These data have been related to the various meteorological, geographical, and seasonal conditions at the time of collection. The resulting statistical data base has revealed definite trends in acid precipitation within Central Indiana, with an overall volume-weighted average pH of 3.9 for the two-year precipitation study. Several individual events have been recorded in the highly acidic pH = 2.8 to 2.0 region.

One of the most obvious effects of acid rain in Central Indiana is structural damage. In conjunction with the above monitoring study, carefully-controlled laboratory weathering studies of Indiana limestone have been conducted. Various types of Indiana limestone were subjected to pH = 4.0 and 3.0 simulated rain, and the results quantitatively monitored.

Atomic Polarizations of Transition Metal *tris*-3-Pentanedionates. EUGENE SCHWARTZ, Department of Chemistry, DePauw University, Greencastle, Indiana 46135.——Results are presented for the radiofrequency and electronic polarizations of the *tris*-3-pentanedionates of vanadium(III), manganese(III), and ruthenium(III) in benzene solution. The atomic polarizations (the difference between the radiofrequency and the visible frequency or electronic polarization) for these compounds are, for vanadium(III), manganese(III), and ruthenium(III), 39 cc, 81 cc, and 35 cc, respectively. The atomic polarizations for the *tris*-3-pentanedionates of the series vanadium(III) through cobalt(III) peak at manganese(III). The second transition series compound *tris*-3-pentanedionato-ruthenium(III) has an atomic polarization considerably smaller than its iron(III) analogue. These results are discussed in terms of contributions to the atomic polarization arising from absorptions in the high frequency microwave region and in the far-infrared spectral region.

Temperature Dependent Infrared Studies of the Hydrogen Bonding in Aliphatic Alcohols. JOHN SCIRCLE AND JOE KIRSCH, Department of Chemistry, Butler University, Indianapolis, Indiana 46208.——The extent of the hydrogen bonding of aliphatic alcohols in dilute solutions has been studied through the use of temperature dependent infrared spectroscopy. The equilibrium constants for the hydrogen bonding process have been determined as a function of temperature. The enthalpy and entropy for the hydrogen bonding process has been calculated from the temperature dependent equilibrium constants. The equilibrium constants, enthalpies, and entropies for the hydrogen bonding process are related to the molecular structure of the alcohols and the steric hindrance at the hydrogen bonding site.

A study of the Coordination Compounds of Some of the Transition Metals Using 2(2-Aminoethoxy)-Ethanol as a Ligand and 1-Methyl-2-Pyrrolidinone as a Solvent. JOSEPH R. SIEFKER AND KENNETH R. KIMMERLE, Department of Chemistry, Indiana State University, Terre Haute, Indiana 47809.——The purpose for this study was to investigate the coordination compounds of some of the transition metals using

2(2-aminoethoxy)-ethanol as a ligand and 1-methyl-2-pyrrolidinone as a solvent. The formation of a coordination compound in solution is an equilibrium process. For metal complexes the central elements of coordination, the transition metal ions, are surrounded by the coordinating groups or ligands, which at first are the solvent molecules but later are replaced by the ligand molecules. The formation of the complex may be represented by the equation:

$$M + nL \rightleftarrows ML_n$$

and the corresponding formation constant is:

$$K_f = \frac{[ML_n]}{[M]\,[L]^n}$$

Two independent spectrophotometric methods were used to measure the variation of the metal ion and metal complex concentrations during the formation of the complex. Job's method of continuous variations as modified by Vosburgh and Cooper was the major method used to determine the coordination formula and the formation constant for the complex. The second method, called the mole ratio or fixed metal method, was tested for its applicability to this case and was used to give a check for the results of Job's method. The spectra were recorded with a Cary Model 14 Spectrophotometer.

The coordination formulas and average formation constants from Job's method are presented below: Nickel (II) Perchlorate and 2(2-Aminoethoxy)-Ethanol Complex

Formula = $[Ni(2(2\text{-Aminoethoxy})\text{-Ethanol})_2](ClO_4)_2$

$K_f = 6.5\ 10^4$

Copper (II) Nitrate and 2(2-Aminoethoxy)-Ethanol Complex

Formula = $[Cu(2(\text{-Aminoethoxy})\text{-Ethanol})_2](NO_3)_2$

$K_f = 2.2 \times 10^5$

Cobalt (II) Perchlorate and 2(2-Aminoethoxy)-Ethanol Complex

Formula = $[Co(2(2\text{-Aminoethoxy})\text{-Ethanol})_3](ClO_4)_2$

$K_f = 1.9 \times 10^{10}$

Manganese (II) Perchlorate and 2(2-Aminoethoxy)-Ethanol Complex

Formula = $[Mn(2(2\text{-Aminoethoxy})\text{-Ethanol})_3](ClO_4)_2$

$K_f = 1.5 \times 10^9$

An Investigation of Aluminum Concentrations in Water. DANIEL K. WUNDERLICH, Department of Science, Terre Haute South High School, Terre Haute, Indiana 47802 and MYONG-KU AHN, Department of Chemistry, Indiana State University, Terre Haute, Indiana 47809.——In this project we investigated the amount of aluminum obtainable from water solutions under conditions similar to those used in cooking with aluminum utensils. The aqueous aluminum concentrations were examined as a function of exposed surface area and pH. The aluminum from sample runs was complexed with 8-hydroxyquinoline and concentrated by extracting with chloroform. The concentrations of aluminum were determined by the molecular fluorescence of the complex, tris(8-hydroxyquinolato)aluminum(III), at 509 nm. The total aluminum concentrations ranged from 0.38 to 1.9 ppm for the aluminum surface area between 125 cm^2 and 625 cm^2, respectively, in 200 ml of distilled water. Aluminum levels in the diet have recently been suspected of being connected with health hazards including senile dementia and osteomalacia.