

CHEMISTRY

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ABSTRACTS

RCHDCHDX Compounds as Stereochemical Probes: Avoiding Deuterium Decoupling. TED T. ASHBURN, SIMONE R. HEAD, and PAUL L. BOCK, Department of Chemistry, Ball State University, Muncie, IN 47306.—The use of RCHDCHDX compounds to examine the stereochemistry of reactions involving organic and organometallic compounds has been of somewhat restricted utility because deuterium decoupling is required in order to observe the proton-proton coupling constants which provide the stereochemical information. The MATHCAD simulations described here strongly suggest that the use of resolution enhanced FT-NMR spectra will allow the determination of the appropriate vicinal coupling constants for these compounds without requiring the use of deuterium decoupling.

Characterization of a Direct Injection Nebulizer. THOMAS W. AVERY, JOSEPH J. THOMPSON, and ESTHER R. SURFACE, Department of Chemistry, Ball State University, Muncie, IN 47306—A direct injection nebulizer (DIN) was constructed and evaluated as an interface for high performance liquid chromatography with inductively coupled plasma-atomic emission spectrometric detection. Detection limits, injection precision and other parameters were nearly identical to literature data for a commercial device, despite the fact that critical steps in the fabrication of the DIN were done in-house. In addition, the nebulizer demonstrated equivalent response for several phosphorous-containing compounds of varying volatility. This uniform response was not observed using a conventional concentric nebulizer with a Scott-type spray chamber.

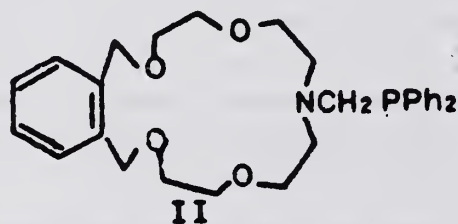
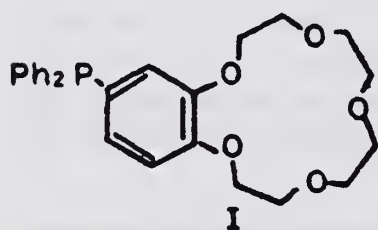
Technique for Improving Quantification of PPB Level Chloride Determinations in Multicomponent Anion Analysis Using Ion Chromatography with a Pre-concentration Column. DANIEL L. BURDEN and STANLEY L. BURDEN, Chemistry Department, Taylor University, Upland, IN 46989 and AuSable Institute of Environmental Studies, Mancelona, MI 49659.—Chloride ion is particularly troublesome to quantify accurately by ion chromatography when using a pre-concentration column. The limited capacity of the pre-concentration column and competition for sites on this column by more strongly retained ions, such as sulfate, cause severe non-linearity in the calibration curve for chloride. The current procedure for handling this problem is to use three or more standards and fit the PPB vs. area points with a second order least squares polynomial equation. This paper will show that significantly improved accuracy is realized by taking the logarithm of PPB before fitting the calibration points. Results from using this

technique on data from simultaneous analysis for PPB level mixtures of chloride, nitrate, phosphate, and sulfate will be presented and discussed.

Glucose Test Strips as Peroxide Indicators. R. DALE DELK, CAROL L. WALTERS, CHRISTOPHER YAMAMOTO, and BRUCE N. STORHOFF, Ball State University, Muncie, IN 47306.—Three types of commercially available test strips of the types used to monitor glucose levels in blood or urine have been evaluated as indicators for peroxides in aqueous and nonaqueous solutions. For aqueous solutions, the minimum detectable quantities of hydrogen peroxide for Diastix^R, Clinistix^R, and Chemstrip^R reagent strips were found to be 1.5 mg/dl, 0.2 mg/dl, and 0.8 mg/dl, respectively. These strips were also found to change color in response to the presence of benzoyl peroxide in ether. The Diastix^R, for example, responded to levels of this peroxide which were greater than 0.6 mg/dl.

Interactions of Iron and Copper with Some Sulfur Oxyanions. KRIS DHAWALE, I.U. East, 2325 Chester Blvd., Richmond, IN 47374-9979.—Aqueous corrosion of iron, copper and their alloys in the presence of sulfur oxyanions is under investigation in our laboratory. This paper deals with the use of Ferrozine and Bathocuproine reagents for the determination of Fe^{2+} and Cu^{1+} ions during corrosion of those metals, and some copper base alloys were also tested in the presence of sulfite, hydrosulfite, tetrathionate, thiosulfate and metabisulfite. The metals are exposed to aqueous solutions of sodium salts of sulfur oxyanions. Then filtered solutions are assayed for Fe^{2+} and Cu^{1+} using the Spectronic 20 for quantitative determination. This work was undertaken to see if these assays can be used to determine the rate of dissolution of the metals. In the absence of any other precise analytical techniques, such assays can give a good estimate of the rate of dissolution.

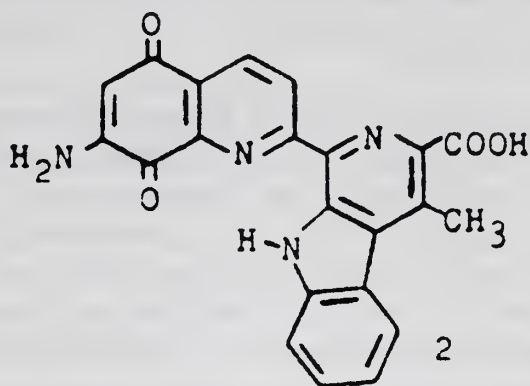
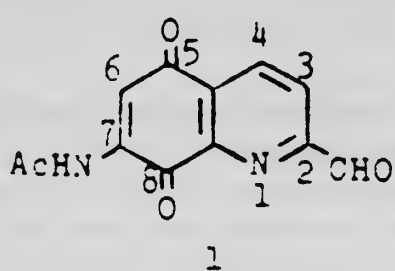
Phosphine-Functionalized Crown Ethers. JAMES E. FRITZ, MARY KAY NEEB, and BRUCE N. STORHOFF, Ball State University, Muncie IN 47306.—Both of the phosphine-functionalized crown ethers shown below have been obtained in modest yields from readily available starting materials. The precursor of II, the corre-



sponding amine, was isolated in modest yield from $\text{HN}[(\text{CH}_2)_2\text{O}(\text{CH}_2)_2\text{OH}]_2$, α , α' -dibromo-o-xylene, and potassium-t-butoxide in t-butanol. Reaction of the amine with diphenylphosphine and aqueous formaldehyde provided II. Compound I was obtained from benzo-15-crown-5 *via* a reaction sequence which involved bromination, Li-Br exchange and attack on ClPPh_2 . The spectroscopic and P-binding properties of these ligands will be discussed.

Synthesis of 7-Acetamido-2-formylquinoline-5,8-dione. ZHENGXIANG GU, MACKLIN B. ARNOLD, ARON C. SOUSA, and MOHAMMAD BEHFOROZ, Department of Chemistry, Ball State University, Muncie, IN 47306.—The title aldehyde (1) an intermediate in the total synthesis of antibiotic antitumor agent lavendamycin (2) was prepared through the Diels-Alder reaction of a 1,3 azadiene with a brom-

quinone followed by oxidation. Compound 1 was also prepared by nitration of 8-hydroxy-2-methylquinoline followed by reduction, acetylation and oxidation.



The Nickel (II)-Dimethylglyoxime Complex: A New Use for an Old Compound. A.J.C.L. HOGARTH, Ross Laboratories, Department 417, 625 Cleveland Avenue, Columbus, Ohio 43216.—Typically, in undergraduate laboratories, the gravimetric determination of nickel is used to demonstrate precipitation from homogeneous solution. The strawberry-red nickel (II)-dimethylglyoxime complex produced is easily dried and weighed, it is heat stable to at least 121 C, it resists atmospheric oxidation and it is analytically pure. The last property has to be true otherwise its use as a quantitative measure of nickel would be of little value. With all these desirable properties, those of a chemical standard material, the complex is normally thrown away after many undergraduates have labored to produce it, and little thought is given to using it. This paper explores a possible use, that of a standard material against which to standardize solutions of EDTA.

A Quest for Flashy Crowns: Progress on a "Clip-On" Approach. SUZY POPE, M. CATHERINE COSBY, AND LYNN R. SOUSA, Department of Chemistry, Ball State University, Muncie, IN 47306.—A long-time goal of our research program is the invention of compounds that will signal the amount of potassium ion present in solution by emitting fluorescence. One scheme we are investigating uses a crown ether compound that has a fluorescent chromophore on one edge of the crown ring and a quencher of fluorescence on the other edge. In the absence of K^+ , the crown ring is flexible, and the quencher can effectively stop the fluorescence of the chromophore. However, when K^+ is present, the crown ether complexes the ion and the crown ring is stiffened as a result. This stiffening separates the chromophore from the quencher and leads to an increase in fluorescence. To speed the research process, we are synthesizing a crown ether key intermediate with two different reactive sites. A number of different fluorescent chromophore-quencher pairs could be readily prepared from this "clip-on" crown ether. Progress on the synthesis will be described.

Molar Refractions of Metal Ions in Complexes. EUGENE P. SCHWARTZ, Department of Chemistry, DePauw University, Greencastle, IN 46135.—Molar refractions (electronic polarizations) of a number of neutral complexes in benzene were measured with a differential refractometer and calculated by the method of Halverstadt and Kumler. Ring refractions were obtained from the beryllium complexes. If the central metal has none or has ten d electrons (Al^{+3} , Sc^{+3} , Ga^{+3}) the apparent molar refraction of the central metal is smaller in the dipivaloyl-methane (2,2,6,6-tetramethyl-3,5-heptanedione) complex than in the acetylacetone (2,4-pen-

tanedione) complex, whereas these ligands show little effect for the other transition elements studied (Cr^{+3} , Mn^{+3} , Fe^{+3} , Co^{+3}). The apparent molar refraction of the metal when complexed with dibenzoylmethane (1,3-diphenyl-1,3-propanedione) is much smaller than when complexed with acetylacetone for all of the metals studied.

Determination of Molar Absorptivities and Formation Constants for Complexes of Copper (II) and Cobalt (II) with 3-Aminopyridine. JOSEPH R. SIEFKER AND RIE-WHUN GIHL, Department of Chemistry, Indiana State University, Terre Haute, IN 47809—The coordination of some transition metal ions with 3-aminopyridine was studied. Spectrophotometric data were obtained with aqueous solutions. The molar absorptivities and formation constants were calculated with a computer program using a CDC Cyber 171 computer.

Speciation of Cisplatin and Related Compounds by High Performance Liquid Chromatography With ICP-AES Detection. JEFFREY J. STADICK, JOSEPH J. THOMPSON, and BRUCE N. STORHOFF, Department of Chemistry, Ball State University, Muncie, IN 47306—Cisplatin (a drug used in cancer therapy), transplatin and their hydrolysis products were separated on a carbohydrate column and detected at ppb levels by an inductively coupled plasma-atomic emission spectrometer. A direct injection nebulizer comprised the interface between column and spectrometer. The method is evaluated as a means of monitoring levels of this drug and its degradation products in body fluids.

The Identification of Superoxide Dismutase in Human Serum Using Polyacrylamide Gel Electrophoresis. EUGENE S. WAGNER, Center for Medical Education, Ball State University, Muncie, IN 47306.—During the past 30 years biochemists have isolated types of molecules known as free radicals which purloin electrons from other molecules and disrupt healthy cell function. Restoration of normal cell function can be accomplished by the interaction of body-produced enzymes called antioxidants which neutralize these free radical toxins. If the body does not produce enough of these antioxidant enzymes naturally because of diet, disease, trauma, chemicals, environment, or age, this cellular underproduction must be provided by other sources. One such source is wheat sprout which is rich in Superoxide Dismutase, SOD, a potent antioxidant enzyme. The claims of many in the health food industry that dietary supplementation of SOD increases erythrocyte levels of SOD prompted this study which outlines a procedure, employing polyacrylamide gel electrophoresis, to measure the presence of SOD in human serum.