DOCUMENT RESUME

ED 024 580 24 SE 005 063

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An Advanced Chemistry Laboratory Program.

Washington and Lee Univ., Lexington, Va.
Spons Agency- Office of Education (DHEW), Washington, D.C. Bureau of Research.
Bureau No- BR-5-8262
Pub Date 68
Note- 10p.
EDRS Price MF-$0.25 HC-$0.60
Descriptors- *Chemistry, *College Science, Curriculum Development, Instruction, *Laboratory Experiments, Undergraduate Study

The Advanced Chemistry Laboratory Program is a project designed to devise experiments to coordinate the use of instruments in the laboratory programs of physical chemistry, instrumental analysis, and inorganic chemistry at the advanced undergraduate level. It is intended that such experiments would incorporate an introduction to the instrument employing a descriptive as well as a theoretical point of view and use of the instrument as a physical tool as well as an analytical device. Also of interest in this project was the possibility of including the preparation of inorganic or organo-metallic compounds as the working substances for the instrumental experiments. The results of the laboratory program revealed that (1) an inorganic preparation followed by instrumental experiments to confirm structure or to provide analytical information can be used as the basis of an integrated experiment for advanced chemistry students, and (2) most of the more rapid preparations require very little variety in the laboratory techniques of preparative chamistry. (RP)

BR-5-8262
PA-24

# AN ADVANCED CHEMISTRY LABORATORY PROGRAM

Cooperative Research Project Number S-421

(Bureau Number: 5-8262)
PA-24

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1968

The research reported herein was supported by the Cooperative Research Program of the Office of Education, U. S. Department of Health, Education, and Welfare.

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## Introduction

The laboratory program in the training of chemists is continuously being evaluated by chemical educators. Questions of how much laboratory, what topics to include, what order of presentation, etc. are frequently raised, and a variety of approaches to answering these questions is found. One particular problem in the laboratory program at present is associated with the use of instruments in the laboratory courses of the advanced undergraduate years.

Within the past 25 years there has been a marked change in the field of chemistry in regard to the use of instruments. Many of these instruments have become commercially available during this time, and applications and techniques are continuously being developed. The separate category of instrumental analysis has become recognized as a specific requirement in the training of a modern chemist. Many colleges and most universities have acquired a variety of instruments, most frequently with support from the National Science Foundation.

The instruments used in the laboratory are frequently involved in several different courses, and often there is considerable overlap in the presentation. For example, an infrared spectrometer might be described and used in the organic laboratory to identify functional groups in an organic preparation, described and used in the physical chemistry laboratory to analyze the structure of a particular molecule, described and used in the instrumental analysis laboratory to determine the composition of a sample, and possibly described and used in an inorganic laboratory to confirm the structure of a preparation.

The particular problem to which this project was directed is that of the overlap in the use of the individual instruments in the various courses. Often a single instrument is described and used in several courses with only a small difference in the point of view. With some coordination, it should be possible to minimize the repetition of description and techniques which can exist with multiple use of the instrument.

## Objectives

The laboratory program in advanced undergraduate chemistry is conceived, from the aspect of the use of instruments, as involving the physical chemistry, the instrumental analysis, and the inorganic courses. At the present time, this institution does not offer laboratory training in the advanced inorganic chemistry course; however, some such training should be included in the program. The main objective was to devise experiments to coordinate the use of instruments in the advanced undergraduate laboratories.

Experiments to be used would be designed to introduce an instrument in both a descriptive and a theoretical manner. The instrument would then be used in both a physical chemical type procedure and as an analytical device. In some cases, these aspects of the useage could be combined (e.g., a kinetics experiment involves both analytical and physical ideas).

Another objective was to examine the possibility of including inorganic or organo-metallic preparations in the experiment. Substances prepared would then be the working materials for the instrumental experiments.

## Procedure

Literature sources were examined for possible experiments to be incorporated into the laboratory program. There are a number of existing textbooks and laboratory manuals in the fields of inorganic, physical, and instrumental analysis. The "Journal of Chemical Education" contains numerous articles and discussions pertaining both to possible experiments and to the problems being examined in this project. It should also be mentioned that the Advisory Council on College Chemistry is engaged in a project similar to the proposals given here.

Visits were made to nine representative colleges and universities to explore current laboratory practices and contemplated curricular revisions. Informal discussion and correspondence with other faculty members also contributed to the project. A number of institutions are actively engaged in attempts to revise laboratory programs; and at least two which were contacted, Lehigh University and Northwestern University, are well along in new programs of the type proposed.

Experimental work on this project was performed by undergraduate students working under the auspices of the Robert E. Lee Research program at Washington and Lee University and of the National Science Foundation summer undergraduate research project at W. and L. In a few cases, experiments in the regular instrumental analysis course were performed with materials prepared by students for this project.

Of major emphasis in the experimental phase of this work was the preparation of compounds. An attempt was made to find preparations which would be relatively rapid (within a single, standard laboratory period) and which would illustrate a variety of preparative techniques. Materials so prepared would then be the working substances for typical experiments in physical chemistry and/or in instrumental analysis.

## Results

A number of inorganic preparations were tested under student laboratory conditions. Several of these compounds were then used as working substances for instrumental experiments either by a student or by students in the regular courses. The preparations represented a variety of techniques and of ease of completion. Each of the preparations completed is described briefly below.

$CoHg(SCN)_4$ was found to be rather easy to prepare. A sample of this material was used in an X-ray diffraction experiment. A preparation of this material using radioactive cobalt was used in a pulse height analysis experiment with a scintillation well.

$Cu_2HgI_4$ was found to be rather easy to prepare. A sample of this material was used for a differential thermal analysis experiment.

$NH_2SO_3H$ requires somewhat more than a single period for preparation, but illustrates use of a gas as a preparative technique. This material was also used in an X-ray diffraction experiment.

Co and Cr ammine complexes of various formulas were prepared. Several of these complexes can be prepared easily while others require longer periods of time. These complexes represent good materials for spectroscopic experiments since some are rather stable in solutions. Samples of these complexes were used in X-ray diffraction experiments, but not all are well characterized in the literature.

$Cr(OAc)_2$ can be prepared in a single period, and the preparation illustrates several techniques (Jones reductor and dry bag). The material is air sensitive; thus making handling more difficult. The preparation requires some practice to be performed readily, but the product has interesting possibilities for further experimentation.

$MoOCl_3 \cdot 2DMSO$ requires several days for preparation and involves fumes which may be hazardous. The compound has some interesting possibilities as a sample for spectroscopic experiments, but it seems too involved and possibly too dangerous for further work.

The use of the materials mentioned on previous pages in instrumental experiments has not been attempted in a complete manner; however, a number of these are known to be useful for such extended experimental projects. Further work with the materials prepared is contemplated. An annotated bibliography contains further observations about preparations and possible useage.

## Conclusions and Implications

It was found that a useful number of experiments can be devised which integrate laboratory work in inorganic, physical, and instrumental analysis. Preparations requiring approximately one laboratory period followed by use of the compound in a physical and/or analytical experiment represent a useful way to focus attention on a particular instrument. On the other hand, many of the preparations which are completed rapidly offer a limited variety of preparative techniques for illustration of inorganic laboratory procedures. Because of this latter difficulty, it was found that experiments of the type visualized would be difficult to incorporate into an existing laboratory program of separate courses with separate laboratory periods. The most convenient arrangement would be a single integrated advanced laboratory program of lectures and laboratory work incorporating elements of inorganic, physical, and instrumental analysis.

This project was undertaken with some view towards establishing a listing of the techniques and instruments which should be included in the advanced laboratory program. Although not all of these were investigated directly, nor are the facilities available at all institutions, the following partial list contains the items deemed important enough for intensive consideration.

a) High temperature techniques

b) Low temperature techniques

c) Vacuum line manipulations

d) Anhydrous and/or inert atmosphere conditions

e) Non-aqueous procedures and solvent extraction

f) Electrolytic preparations

g) NMR experiments

h) Mass spectrometry

i) X-ray diffraction

j) Radioactivity procedures

k) Calorimetry

l) Spectrometry (infrared, visible ultraviolet) including absorption and emission

m) Gas chromatography

n) Gouy balance

o) Ion exchange procedures

p) pH techniques

q) Polarography and electrochemistry

r) Coulometry

s) Electronics

In all cases, considerable attention should be placed on the analysis of errors and the limitations of the instruments employed.

## Bibliography

Metal-ammine complexes
Preparation: "Inorganic Syntheses", McGraw-Hill, N.Y.
Experiments: Poteniometric: J. Chem. Ed. 39, 329 (1962)
J.A.C.S. 81, 529 (1959)
J. Chem. Ed. 40, 341 (1963)
J.A.C.S. 67, 1334 (1945)
Acta Chem. Scand. 2, 297 (1948)
(computerized calculations are possible)
Isopiestic: J. Chem. Ed. 38, 28 (1961)
(useful for activity coefficients)

Cr(III) complexes
Preparation and experiments: J. Chem. Ed. 44, 101 (1967)
(compounds possibly useful for additional experimental procedures, including kinetics)

Co(III) complexes
Preparation and experiments:
J.A. Bell, "Chemical Principles in Practice", Addison-Wesley
(again capable of modifications such as J. Chem. Ed. 43, 93 (1966))

$NH_2SO_3H$
Preparation: "Inorganic Syntheses", II, p. 176, McGraw-Hill, N.Y.
(the related compound, $NH_2CH_2SO_3H$, could also be prepared and contrasted with $NH_2SO_3H$. W.G. Palmer, "Experimental Inorganic Chemistry", p. 359, Cambridge)

$Cu_2HgI_4$
Preparation: G. Brauer, "Handbook of Preparative Inorganic Chemistry", 2nd edition, Vol. II, p. 1110, Academic Press, N.Y.

$Cr(OAc)_2$
Preparation: "Inorganic Syntheses, I, p. 122; III, p. 148; VI, p. 145; VIII, p. 125, McGraw-Hill, N.Y.
Experiments: Non-aqueous titration: J. Chem. Ed. 43, 215 (1966)
Reilly and Sawyer, "Experiments for Instrumental Methods", p. 26, McGraw-Hill, N.Y.

$MoOCl_3 \cdot 2DMSO$
Preparation: S.M. Horner, Ph.D. Dissertation, Univ. of North Carolina, 1962

$CuC_2O_4$
Preparation and analysis: Meites and Thomas, "Advanced Analytical Chemistry", McGraw-Hill, N.Y.
(also possible DTA experiment sample)

Kinetics experiments
J. Chem Ed. 40, 264 (1963) (could be modified for additional instrumental approaches)

Univ. of North Carolina procedure using gas chromatography in an esterification reaction

J. Chem. Ed. 33, 552 (1956) (variety of possible instrumental approaches)

J. Chem. Ed. 38, 571 (1961) (DTA techniques)

J. Chem. Ed. 40, 607 (1963) (instrumentation is not particularly involved)

Gas chromatography
(See kinetics experiment above)

J. Chem. Ed. 40, 541 (1963) (activity coefficients are determined)

J. Chem. Ed. 43, 321 (1966) (infrared and ultraviolet spectra could also be obtained)

Fused salts
J. Chem. Ed. 43, 363 (1966) (an EMF titration)

Infrared
J. Chem. Ed. 43, 476 (1966) (gaseous reaction products - although corrosive - and solid residues for further analysis)

Shoemaker and Garland, "Experiments in Physical Chemistry", 2nd edition, p. 329, McGraw-Hill, N.Y. (possibly as a dry-lab experiment)

Radiochemical
J. Chem. Ed. 41, 604 (1964) (used for the $CoHg(SCN)_4$ experiment)

J. Chem. Ed. 37, 391 (1960) (neutron activation)

X-ray diffraction
J. Chem. Ed. 42, 77 (1965) (offered as a dry-lab possibility)

NMR
J. Chem. Ed. 44, 200 (1967) (another dry-lab possibility)

Gouy balance
J. Chem. Ed. 39, 574 (1962)

J. Chem. Ed. 39, 577 (1962)

J. Chem. Ed. 44, 142 (1967) (includes a possible preparation and CTA sample)