



**NBS TECHNICAL NOTE**

**986**

**U.S. DEPARTMENT OF COMMERCE / National Bureau of Standards**

# **NBS Special Foreign Currency Program in Yugoslavia 1973-1978**

53  
986  
8

## NATIONAL BUREAU OF STANDARDS

The National Bureau of Standards<sup>1</sup> was established by an act of Congress March 3, 1901. The Bureau's overall goal is to strengthen and advance the Nation's science and technology and facilitate their effective application for public benefit. To this end, the Bureau conducts research and provides: (1) a basis for the Nation's physical measurement system, (2) scientific and technological services for industry and government, (3) a technical basis for equity in trade, and (4) technical services to promote public safety. The Bureau's technical work is performed by the National Measurement Laboratory, the National Engineering Laboratory, and the Institute for Computer Sciences and Technology.

**THE NATIONAL MEASUREMENT LABORATORY** provides the national system of physical and chemical and materials measurement; coordinates the system with measurement systems of other nations and furnishes essential services leading to accurate and uniform physical and chemical measurement throughout the Nation's scientific community, industry, and commerce; conducts materials research leading to improved methods of measurement, standards, and data on the properties of materials needed by industry, commerce, educational institutions, and Government; provides advisory and research services to other Government Agencies; develops, produces, and distributes Standard Reference Materials; and provides calibration services. The Laboratory consists of the following centers:

Absolute Physical Quantities<sup>2</sup> — Radiation Research — Thermodynamics and Molecular Science — Analytical Chemistry — Materials Science.

**THE NATIONAL ENGINEERING LABORATORY** provides technology and technical services to users in the public and private sectors to address national needs and to solve national problems in the public interest; conducts research in engineering and applied science in support of objectives in these efforts; builds and maintains competence in the necessary disciplines required to carry out this research and technical service; develops engineering data and measurement capabilities; provides engineering measurement traceability services; develops test methods and proposes engineering standards and code changes; develops and proposes new engineering practices; and develops and improves mechanisms to transfer results of its research to the ultimate user. The Laboratory consists of the following centers:

Applied Mathematics — Electronics and Electrical Engineering<sup>2</sup> — Mechanical Engineering and Process Technology<sup>2</sup> — Building Technology — Fire Research — Consumer Product Technology — Field Methods.

**THE INSTITUTE FOR COMPUTER SCIENCES AND TECHNOLOGY** conducts research and provides scientific and technical services to aid Federal Agencies in the selection, acquisition, application, and use of computer technology to improve effectiveness and economy in Government operations in accordance with Public Law 89-306 (40 U.S.C. 759), relevant Executive Orders, and other directives; carries out this mission by managing the Federal Information Processing Standards Program, developing Federal ADP standards guidelines, and managing Federal participation in ADP voluntary standardization activities; provides scientific and technological advisory services and assistance to Federal Agencies; and provides the technical foundation for computer-related policies of the Federal Government. The Institute consists of the following divisions:

Systems and Software — Computer Systems Engineering — Information Technology.

<sup>1</sup>Headquarters and Laboratories at Gaithersburg, Maryland, unless otherwise noted; mailing address Washington, D.C. 20234.

<sup>2</sup>Some divisions within the center are located at Boulder, Colorado, 80303.

SEP 20 1978

# NBS Special Foreign Currency Program in Yugoslavia 1973-1978

*Technical note, 10, 986*

Doris M. Bluebond,  
Robert S. Marvin,  
and H. Steffen Peiser

Office of the Director  
National Bureau of Standards  
Washington, D.C. 20234



U.S. DEPARTMENT OF COMMERCE, Juanita M. Kreps, Secretary

Dr. Sidney Harman, Under Secretary

Jordan J. Baruch, Assistant Secretary for Science and Technology

NATIONAL BUREAU OF STANDARDS, Ernest Ambler, Director

Issued September 1978

Library of Congress Catalog Card Number 78-600093

**National Bureau of Standards Technical Note 986**

Nat. Bur. Stand. (U.S.), Tech. Note 986, 75 pages (Sept. 1978)

CODEN: NBTNAE

U.S. GOVERNMENT PRINTING OFFICE  
WASHINGTON: 1978

---

For sale by the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402  
Stock No. 003-003-01977-1 Price \$2.50  
(Add 25 percent additional for other than U.S. mailing).

## FOREWORD

This Publication reviews the National Bureau of Standards Special Foreign Currency Program in Yugoslavia since January 1973. The project descriptions presented here will help scientists and scientific administrators form opinions on the value of such programs, and help the more general reader examine an example of international cooperation and its success in contributing to the general betterment of the two countries involved and in improving international scientific relationships.

Grants from a Joint Fund, to which the U.S. and Yugoslavia contribute equally, are made for research by Yugoslav scientists working in Yugoslav institutions. Projects are approved by the Yugoslav Federal Administration for International Scientific, Educational, Cultural and Technical Cooperation and certain U.S. Scientific and Technological Agencies. Although the NBS allocations to the Joint Fund have now been fully committed and expended, an agreement between Presidents Carter and Tito to supplement the funding brings expectation of program continuation.



All the projects funded under this Program are summarized in this report. The summaries list the project goals, some major results, the publications which resulted from the specific support under this Program and the names of the NBS monitor and the principal investigators. From the NBS viewpoint the Program has been scientifically productive and has contributed to NBS mission and programmatic objectives. We understand that Yugoslav officials have concluded, in part from the previous report (Technical Note 753), that our collaborative program has contributed to their own goals as well.

Further, we consider that opportunities abound for a long continuing program of mutual interest. NBS intends to continue to explore these opportunities and we trust that the good technical cooperation that has developed under this program will continue and be strengthened in the future.

A handwritten signature in dark ink, appearing to read "E. Ambler.", with a stylized, flowing script.

Ernest Ambler  
Director

# TABLE OF CONTENTS

Page(s)

INTRODUCTION .....	1
STANDARD REFERENCE MATERIALS	
L. Kosta/P. D. LaFleur "Determination of Impurity Levels in Analytical Grade and Ultrapure Chemicals by Activation Analysis Using Triga Reactor" .....	7
M. Branica/J. K. Taylor "Electroanalytical Determination of Trace Elements" .....	11
MATERIALS MEASUREMENT METHODS AND STANDARDS	
R. Blinc/D. H. Reneker "Development and Application of Ultrasensitive Magnetic Resonance Methods" .....	15
I. Piljac/J. K. Taylor "Electrochemical Study of Metal Ion-ligand Interaction in Nonaqueous Solutions by Investigation of Electro-chemical Property of Ligand" .....	21
K. F. Zmbov/J. W. Hastie "Mass Spectrometric Studies of High Temperature Equilibria" .....	25
PROPERTIES OF MATTER	
A. Bonefacic/R. P. Reed "X-ray Diffraction and Electromicroscopic Studies on Aluminum and on the Metastable Phases of Aluminum Base Alloys, Obtained by Rapid Quenching from the Melt" .....	31
N. Konjevic/W. L. Wiese "Stark Broadening of Spectral Lines in Plasmas" .....	37
V. Vujnovic/W. L. Wiese "Measurement of f-values in Ultraviolet and Visible Region and Study of Continuous Emission of Hydrogen near the Balmer Limit" .....	43
V. V. Urosevic/G. Dunn "Electron-atom Excitation Cross Section Measurements" .....	49
EXPLORATORY RESEARCH	
V. Pravdic/W. Haller "Bulk Composition and Surface Properties of Glass" .....	51
M. Sunjic/J.W. Gadzuk "Theoretical Investigations on Physical Surfaces" .....	57

	Page(s)
J. Slivnik/T. D. Coyle "Syntheses and Properties of Fluoride Complexes" .....	59
 COMPUTER SCIENCE AND TECHNOLOGY	
Z. Damjanovic/T. N. Pyke, Jr. "A Modular Computer System for Real-Time Data Reduction and Control in Biomedical Experiments" .....	63
 NULCEAR AND RADIATION TECHNOLOGY	
I. Draganic/W. L. McLaughlin "Radiation Dosimetry and Radiation Chemistry" .....	67
APPENDIX I - SFCP (PL-480) BACKGROUND HISTORY .....	71
APPENDIX II - INDEX OF INSTITUTES .....	73
APPENDIX III - ALPHABETIC LIST OF PRINCIPAL INVESTIGATORS .....	74
APPENDIX IV - ALPHABETIC LIST OF NBS MONITORS .....	75



## ABSTRACT

An overview is given of grants awarded by the National Bureau of Standards under the Special Foreign Currency Program (SFCP) or by the U.S.-Yugoslav Joint Board. Each grant is identified by title, principal investigator, institution in Yugoslavia, NBS monitor, and the monitor's organizational unit within NBS. The work is then described briefly under the three headings "Summary Description of Project Goals," "Results and Implications to Date," and "List of Publications that Resulted from the Project." To demonstrate the relevance of such grants to the programs of NBS, the grant descriptions are ordered by NBS institutes and divisions. The significance and purpose of the NBS/SFCP grant programs are discussed in the Foreword and the Introduction. The NBS monitors and the program manager judge this grant program to have had a high benefit to cost ratio from the viewpoint of NBS.

Key words: Binational research cooperation; international scientific cooperation; physical science research administration; research planning; scientific research abstracts; Special Foreign Currency Program; Yugoslavia science and technology.



## NBS SPECIAL FOREIGN CURRENCY PROGRAM IN YUGOSLAVIA ]973/77

### 1. INTRODUCTION

This Technical Note is intended to provide a review of the scientific and technological grants program of the National Bureau of Standards (NBS) conducted in Yugoslavia by use of Special Foreign Currency Program (SFCP) funds. Such information is needed for any value judgment, be it from the viewpoint of the disciplines, the institutions or the technological plans of countries. Each grant funded by the NBS/SFCP since the last similar review (NBS Technical Note 753) is described briefly in terms of its initial project goals, its significant accomplishments, and its implications for science or technology at the cooperating institutions of both countries.

The NBS Special Foreign Currency Program discussed in this report can be traced to visits to Yugoslavia of two small NBS scientific teams in September 1970 and October 1972. These teams accumulated firsthand information about science and technology in Yugoslavia based on discussions with scientists, engineers, educators, R&D administrators, and governmental officials. Visits to more than two dozen selected laboratories and institutes in eight cities including Belgrade, Zagreb, Ljubljana, Sarajevo, and Zenica were completed in two two-week periods. In parallel with proposals by Yugoslav scientists through their institutions and committees in the Republics, recommendations were made to the Director of the National Bureau of Standards for funding projects in Yugoslavia under the Special Foreign

Currency Program based on the teams' evaluation of the projects in accordance with established criteria, and the capabilities of those submitting the proposals to undertake and successfully complete the work.

The Program seen from the point of view of NBS can be said to be of direct benefit to at least five major elements of the NBS mission. Several of the projects are involved with measurement techniques not yet exploited at NBS. Input for the National Standard Reference Data System is provided in areas not yet being investigated at NBS. Many NBS projects are currently involved with computer control of experiments. Closely related is the project in Yugoslavia on interactive control between experiments and computer. Finally, some of the Yugoslav projects are concerned with the development of standards and standard reference materials, supplementing NBS work and providing both new and better characterized standards.

The Yugoslav oriented reader should ask whether new and increased capabilities have been brought to Yugoslavia by these projects and whether the collaboration has been useful to institutional and national goals. Questions should be asked: Have new competences been built up and existing ones strengthened? Have scientific and technological talents been diverted from the national goals of Yugoslavia? Have neighboring fields been enriched in Yugoslavia by cross-fertilization? Have applications in industry been sparked and has the Program helped to develop a broadly based infrastructure of science and technology in Yugoslavia?



Readers who are experts in the fields of investigation may ask such questions as:

1. Were the projects in the fields familiar to you well executed?
2. Did the senior investigators demonstrate exceptional competence which could be of special service in the future?
3. Did the projects contribute to the solution of problems of known importance to science, technology, and society?
4. Is there evidence that contact between US and Yugoslav scientists was intellectually stimulating and helpful in related work?
5. Does the program appear to have contributed to a stimulating climate for scientific progress and technological innovation?
6. Did the program help to widen the horizons of scientists towards application of their expertise to urgent national needs?
7. Does this report suggest that bi-national programs of this type should be developed for mutual benefit?

This list of questions is certainly not exhaustive nor are their answers by any means obvious.

This report is addressed most particularly to scientifically trained science administrators. He should remember, however, that the financial support given to the NBS/SFCP has amounted to less than \$300,000 spread over all projects in the almost ten years. The extreme modesty of these funds has been the principal constraint on the program, but it must be acknowledged that institutions have in most cases added from their own resources so that the true cost of the research has been somewhat higher.



Several management details may be responsible for much of the success of this program. First of all, the NBS monitor has been strongly encouraged to communicate directly with the principal investigator. Secondly, proper scheduling of visits by the NBS monitors to Yugoslavia has broadened the basis of interaction. Thus, the input to the NBS/SFCP from the NBS monitor can be based upon his knowledge of the general Yugoslav scientific context of the particular project which he is monitoring. To promote widespread knowledge at NBS of Yugoslavian scientists and institutions, whenever it becomes necessary, for scientific reasons, for an NBS monitor to visit his project in Yugoslavia, he is urged to visit as many additional scientific institutions and laboratories as possible. Such visits are described in reports, on his return, which include his appraisal of the scientific competences and the particular strengths of individuals, laboratories, and institutions that he observed. In addition to serving to broaden the outlook of the particular NBS monitor, it increases the extent of the contacts between Yugoslav scientists and the NBS staff, thereby enabling the Yugoslav scientist to see NBS from the various points of view brought by the NBS visitors. This increases the likelihood that Yugoslav scientists involved in the program will, to a certain extent, insert an NBS point of view in addition to their own, into goals set for their projects. By careful planning of such NBS staff visits their cost has been kept to a fraction of the project funds available. The trip reports by the two NBS teams that visited Yugoslavia are recommended reading for NBS staff visiting Yugoslavia in the future.

The files of the program contain a growing number of examples of collaborative work which has been of direct utility to either or both sides. Such collaborative efforts are extremely important in today's world. The unit for scientific research is seldom an individual scientist, but rather a team. The complex and interdisciplinary nature of modern scientific work makes use of self-contained, local expertise of utility only as a component in collaborative efforts which cross lines normally separating institutions, countries and areas of research.

Some of the work reported here was completed as much or more than two years ago. Nevertheless, it is believed that these reports should be issued as a summary of the activities supported and encouraged by this program. The editors would greatly appreciate receiving any comments that the readers would care to make regarding any of the individual projects or the overall program.

Identification of commercial products does not imply recommendation or endorsement by the National Bureau of Standards nor that the product is necessarily the best available for the purpose.



Project Title: DETERMINATION OF IMPURITY LEVELS IN ANALYTICAL  
GRADE AND ULTRAPURE CHEMICALS BY ACTIVATION  
ANALYSIS USING TRIGA REACTOR

Principal Investigator: Professor Dr. Lado Kosta

Institution: Jozef Stefan Institute, Ljubljana

NBS Monitor: Dr. P. D. LaFleur

NBS Institute or Center and Division: Institute for Materials Research  
Analytical Chemistry Division

#### Summary Description of Project Goals

The goals of this project are the development of methods of trace element analysis and certification of the content of important trace elements in certain Standard Reference Materials. The analyses are based on radioactive tracer and activation analysis (both slow and fast neutrons) techniques, which permit the coverage of a wide spectrum of materials and a large number of elements critical in many applications--quality control of agricultural products and in the food industry, the suitability of raw materials for particular purposes, aspects of process control, and in the evaluation of import and export products.

The SRM's include some from the NBS series, particularly biological materials and glasses, plus a new series which are being prepared by NBS, IAEA, and SEV, which are likely to be specifically recognized by organizations such as IUPAC. This new series includes glasses, calcite, zinc, kaolin, magnesite, quartz, river sediments, homogenized and freeze-dried tissues, and some basic foods such as powdered human milk, and vegetables (i.e. spinach). The methods



developed should also be important in implementing the new Yugoslav Federal Law on Standardization and Quality Norms, which requires certification of a wide range of materials used in agricultural, food, chemical, metals, paper, and electronic industries.

#### Results and Implications to Date

The various methods of analysis and separation developed during this project are shown by the titles of the publications listed in the next section. Most of these methods have been applied by the authors to assist in the characterization of various NBS SRM's, and all have important potential for future characterization work.

There is an increasing emphasis in setting tolerance limits for trace elements in a wide range of materials of commerce. This requires reliable measurements, measurements which can successfully be carried out in many laboratories if they have reliable SRM's for the calibration of their analytical processes and devices. But very few laboratories are capable of carrying out the absolute measurements required to characterize these SRM's. NBS is one, JSI another. The collaboration of these two laboratories has yielded significant advantages to both, and to the larger analytical community which they serve, by speeding the development of adequate absolute techniques which have been successful both in extending the range to lower concentrations and in overcoming difficult separation problems.

#### List of Publications that Resulted from the Project

1. V. Ravnik, M. Dermelj and L. Kosta, "A Highly Selective Diethyldithiocarbamate Extraction System in Activation Analysis of Copper, Indium, Manganese and Zinc," J. Radioanalytical Chem., 20 pp 443-453 (1974).



2. A. R. Byrne and L. Kosta, "Simultaneous Neutron-Activation Determination of Selenium and Mercury in Biological Samples by Volatilization, "Talanta, Vol. 21, pp 1083-1090 Pergamon Press (1974).
3. A. R. Byrne, "Neutron Activation Analysis of Tin in Biological Materials and Their Ash Using  $^{123}\text{Sn}$  and  $^{125}\text{Sn}$ ," J. Radioanalytical Chemistry, Vol. 20 627 (1974).
4. A. R. Byrne and V. Ravnik, "Analysis of Arsenic in Glass Standards by Neutron Activation Analysis," Radiochem. Radioanal. Letters 20/2 pp 117-136 (1974).
5. A. R. Byrne and A. Vakselj, "Rapid Neutron Activation Analysis of Arsenic in A Wide Range of Samples by Solvent Extraction of the Iodide," Croatica Chemica Acta CCACAA 46 (3) pp 225-235 (1974).
6. V. Ravnik, M. Dermelj and L. Kosta, "Determination of Some Trace Elements (Fe, Co, Cr, Zn, Cu, Mn, and In) in Different Series of Standard Reference Samples by Neutron-Activation Analysis," Mikrochimica Acta (Wien) pp 153-164 (1976).
7. M. Dermelj, V. Ravnik and L. Kosta, "A Fast Isolation and Determination of Cd in Some Fungi, Other Biological Materials, Soil and Zn Metal by NAA," Radiochem. Radioanal. Letters 24(2) pp 91-102 (1976).



Project Title: ELECTROANALYTICAL DETERMINATION OF TRACE ELEMENTS

Principal Investigator: Dr. Marko Branica

Institution: Rudjer Boskovic Institute, Zagreb

NBS Monitor: Dr. John K. Taylor

NBS Institute or Center and Division: Institute for Materials Research  
Analytical Chemistry Division

### Summary Description of Project Goals

The aim of this work is to investigate and establish the applicability, advantages, and limitations of electroanalytical procedures in comparison with other well known methods for the trace analysis of natural waters.

Electroanalytical methods have often been used successfully in the detection and determination of traces of heavy metals in aqueous solutions. These rather simple and sensitive methods are specially convenient for direct determination of trace metals in natural waters (rivers, lakes, sea) and waste industrial waters. The present research is directed to a better understanding of the methodology.

To achieve reliable and successful electroanalytical determination of trace metals in natural waters, it is necessary to investigate simultaneously:

- (a) the development of very sensitive analytical techniques and procedures, which enable direct determination at a concentration level of  $10^{-8}$  M or less,
- (b) the characterization of the species actually present in the sample, and
- (c) the study of chelation, hydrolization and other interactions between metallic ions and inorganic and organic ligands in natural aquatic systems.

In addition it is desirable to take into consideration the possible changes in sample composition due to adsorption and interaction with container walls.

#### Results and Implications to Date

The results obtained were reviewed at the 7th IMR Symposium, NBS/Gaithersburg (1). Although anodic stripping methods have been widely applied during the last ten years for determination of some heavy metals in natural water systems, relatively little has been done on this use in physico-chemical characterization and on determinations of special importance for elucidation of respective biochemical cycles and the fate of radioactive or industrial wastes. The experience in application of electroanalytical techniques in the laboratory were presented and discussed with regard to: determination of concentrations of some metal ions; characterizations of the species actually present; and study of chelation, hydrolyzation and other interactions between metallic ions and organic ligands in natural aquatic systems.

Anodic stripping voltammetry (ASV) with the rotating glassy carbon electrode has been investigated and used for determination of cadmium and lead, and the slowly dropping mercury electrode was used for determination of zinc in the sea water samples from the North Adriatic. The reproducibility of measurements, behaviour of electrodes, the role of cell and storage bottle materials, and sampling procedures were discussed.

The principle and the applicability in trace metals analysis of differential stripping voltammetry (DSV) with two symmetrical rotating glassy-carbon electrodes covered by a thin mercury film were studied as a new development of electroanalytical methods (2).

A new method for characterization of the ionic state of a metal at very low concentrations, based on voltammetric measurements has been investigated. The formation of complexes of cadmium at a concentration of  $5 \times 10^{-9}$  M with chloride in the solution of constant ionic strength,  $\mu = 0.7$  ( $\text{NaCl} + \text{NaClO}$ ), and with thiocyanate,  $\mu = 2.0$  ( $\text{KNO}_3 + \text{KSCN}$ ), as well as complexes of lead with chloride were studied to illustrate the possible application of the method (5).

In many natural waters and especially in polluted waters, the presence of dissolved organic matter, which could act as a complexing or chelating agent for metallic ions, can be expected. Surface active substances (organic matter, detergents, oils), dissolved or colloidally dispersed in natural water also can be adsorbed on the electrode surface. These adsorption processes change the capacity current and suppress (or even eliminate) the faradaic current, which means that they strongly influence the electroanalytical results, changing the sensitivity, potential and velocity of electrode processes.

It is very probable that synthetic detergents represent the most significant surface active compounds among various organic pollutants entering natural waters. The polarographic method of discontinuously changing potential, known as the Kalousek commutator technique, was



applied to the estimation of surface active substances in samples of polluted seawater on the basis of the adsorption effects at the mercury electrode.

Future research will study the stability of sea water samples when stored under various conditions, with the objective of evaluating the feasibility of using this material as an SRM for trace metals. Electroanalytical determinations of trace elements in natural waters are influenced by many different factors, as are results obtained by any other well known methods. Therefore, the proposed research involves many complex and mutually interacting investigations, and merits a larger allocation of staff and funds.

#### List of Publications which Resulted from the Project

1. M. Branica, L. Sipos, S. Bubic and S. Kozar, "Electroanalytical Determination and Characterization of Some Heavy Metals in Seawater," 7th Materials Research Symposium, National Bureau of Standards, Gaithersburg, Maryland, USA, October 7-11, 1974. NBS Special Publication 422, 1977 "Accuracy in Trace Analysis: Sampling, Sample Handling, Analysis," Vol. II, pp 917-928.
2. L. Sipos, I. Kontusic and M. Branica, "Differential Stripping Voltammetry with Two Rotating Glassy-Carbon Electrodes," Meeting of Chemists of Croatia 1975, Zagreb, February 12-14, 1975.
3. S. Kozar, L. Sipos and M. Branica, "Determination of Traces of Heavy Metals by Differential Stripping Voltammetry," Meeting of Chemists of Croatia 1975, Zagreb, February 12-14, 1975.
4. Z. Kozarac, B. Cosovic and M. Branica, "Estimation of Surfactant Activity of Polluted Seawater by Kalousek Commutator Technique," J. Electroanal. Chem. 64, pp 75-83 (1976).
5. M. Branica, D. M. Novak and S. Bubic, "Application of Anodic Stripping Voltammetry to Determination of the State of Complexation of Traces of Metal Ions at Low Concentrations Levels," submitted for publication in Croatia Chem. Acta.

Project Title:: DEVELOPMENT AND APPLICATION OF ULTRASENSITIVE MAGNETIC RESONANCE METHODS

Principal Investigator: Professor R. Blinc

Institution: Jozef Stefan Institute, University of Ljubljana,  
Ljubljana

NBS Monitor: Dr. D. H. Reneker

NBS Institute or Center and Division: Institute for Materials Research  
Polymers Division

### Summary Description of Project Goals

The purpose of this project is the development of new nuclear-nuclear double resonance methods which allow a simple measurement of the NMR and NQR spectra of nuclei which are too weak to be detected by conventional magnetic resonance techniques as well as the development of new multipulse NMR spin-echo methods for the measurements of the translational self diffusion in systems where normal NMR spin-echo methods cannot be applied because spin-spin relaxation times are too short. The multipulse NMR spin echo methods are being used to measure the segmental diffusion of polyethylene molecules in the melt, and to characterize the diffusion of small molecules, particularly butane in solid polyethylene. The advantages of the NMR methods for characterizing diffusion processes promise to be of great value in relation to NBS programs concerned with the prediction of migration of additives from polymers, particularly those that create health hazards by migrating from packaging materials into foodstuffs.

## Results and Implications to Date

The following results have been obtained so far:

1. A new nuclear-nuclear double resonance technique (1) has been developed which allows the measurement of the pure NQR spectra of integer spin nuclei (like  $^{14}\text{N}$  and  $^2\text{H}$ ) by double resonance with half integer spin nuclei like protons in cases where spin quenching prevented the use of classical nuclear-nuclear double resonance techniques. The new technique is based on the solid effect, i.e., on strong r.f. magnetic field induced coupling between spin systems. It has been applied in several investigations, including the first measurement of the nuclear quadrupole resonance spectra of  $^{14}\text{N}$  in polypeptides (3).

2. A new multipulse NMR spin-echo technique (4) has been developed which allows for self-diffusion measurements in systems where this was not possible previously because the spin-spin relaxation times are too short. The new technique extends the range of the NMR determination of selfdiffusion coefficients from liquids and gases to adsorbed molecules, biological polymers, liquid crystals, and in favorable cases even crystalline solids. The pulse sequence used consists of: (i) A Waugh-type multiple  $90^\circ$  rf pulse sequence removing dipolar interactions:  $-P_Y - (t-P_X - 2t-P_X - t-P_Y)$ ; (ii) a pulsed, linear, magnetic-field-gradient sequence placed between the rf pulses at such intervals that its effect is not averaged out by (i); (iii) a slow, refocusing, Carr-Purcell train of  $180^\circ$  rf pulses. Steps (ii) and (iii) are similar to the technique used by Tanner, whereas step

(i) is new and essential for the observation of spin echoes which make the measurement of the self-diffusion coefficients feasible. This technique was used to measure the diffusion coefficient of butane in polyethylene as a function of pressure. The results show that the method provides new insight into the diffusion process since by measuring diffusion over small distances it avoids problems of interpretation which arise because spherulite boundaries are difficult to characterize. The NMR methods also measure the diffusion coefficient at a particular concentration of butane instead of averaging over a wide concentration range as is necessarily the case with alternative methods. Other advantages of the NMR method are that it provides relaxation time information and it may be used in a straight forward way to measure anisotropy in the diffusion coefficient of certain kinds of oriented polymer samples. It appears probable that these measurements will have direct beneficial impact on NBS programs concerned with the prediction of durability and safety of plastics where migration of small molecule additives are important, as is frequently the case.

The new NMR methods were also shown to be useful for measuring the self diffusion of molten polymers. This information, not previously obtainable by straightforward experiments, bears on polymer mixing and flow problems frequently encountered in the processing of polymers.



Still another application was the study of the self-diffusion of adsorbed water in biological polymers like oriented Na-DNA where a significant anisotropy of the self-diffusion coefficient was detected as well as the presence of restricted diffusion due to the fibrous structure of the sample. The typical fibre diameter necessary to explain the restriction of the diffusion was found to be about one micrometer. It should be mentioned that this research is part of a long range research program of the Jozef Stefan Institute. About half of its effort has been funded by Institute sources and the Slovenian Research Community.

#### List of Publications Resulting from Project

1. J. Seliger, R. Blinc, M. Mali, R. Osredkar and A. Prelesnik, "Nuclear Magnetic Double Resonance Based on Strong rf Magnetic-Field-Induced Coupling Between Spin Systems," Phys. Rev. B 11, 27 (1975).
2. R. Blinc, M. Mali, R. Osredkar and J. Seliger, "Nitrogen and Chlorine NQR in Paraelectric and Ferroelectric Tris-Sarcosine Calcium Chloride," Journal of Chem. Phys. 63, 35 (1975).
3. R. Blinc, M. Mali, R. Osredkar, J. Seliger and L. Ehrenberg, " $N^{14}$  Quadrupole Resonance in Polyglycine," Chem. Phys. Letters 28, 158 (1974).
4. R. Blinc, M. Burgar, M. Luzar, J. Pirs, I. Zupancic and S. Zumer, "Anisotropy of Self-Diffusion in the Smectic A and Smectic C Phases," Phys. Rev. Letters 33, 1192 (1974).
5. R. Blinc, J. Seliger, R. Osredkar and M. Mali, " $O^{17}$  Quadruple Resonance Study of the Ferroelectric Phase Transitions in  $KH_2PO_4$ ," Phys. Letters 47A, 131 (1974).
6. J. Seliger, R. Osredkar, M. Mali and R. Blinc, " $N^{14}$  Quadrupole Resonance of Some Liquid Crystalline Compounds in the Solid," Journal of Chem. Phys. 65, 2887 (1976).



7. R. Blinc, Proceedings of the Enrico Fermi International Summer School on Local Properties at Phase Transitions, Varenna, Italy, 1973, 165 (1975).
8. I. Zupancic, G. Lahajnar, V. Rutar, Lj. Miljkovic and A. Rupprecht, paper presented at the XIXth Congress Ampere, Heidelberg 1976, to be published in the proceedings of the Congress.
9. I. Zupancic, G. Lahajnar, R. Blinc, D.H. Reneker and A. Peterlin, "NMR Study of Diffusion of Butane in Linear Polyethylene," to be published, J. Polymer Sci. (1978).
10. I. Zupancic, G. Lahajnar, R. Blinc, D. H. Reneker and A. Peterlin, "NMR Line Shape, Relaxation, and Self Diffusion of Polyethylene in the Melt," in draft.



Project Title: ELECTROCHEMICAL STUDY OF METAL ION-LIGAND INTERACTION  
IN NONAQUEOUS SOLUTIONS BY INVESTIGATION OF ELECTRO-  
CHEMICAL PROPERTY OF LIGAND

Principal Investigator: Professor Ivan Piljac

Institution: Institute of Inorganic Chemistry, Faculty of Technology,  
University of Zagreb

NBS Monitor: Dr. John K. Taylor

NBS Institute or Center and Division: Institute for Materials Research  
Analytical Chemistry Division

#### Summary Description of Project Goals

This research was undertaken for the purpose of finding a correlation between the strength of metal-ion-ligand interactions and the changes in electrochemical properties of ligands.

Investigations of hydroxy-quinone and of their conjugate base in non-aqueous media was performed by polarography and other electrochemical methods. Electrochemical measurements were combined with spectroscopic ones using the thin-layer, optically transparent electrochemical cell.

This is a new approach to the problem of studying metal ion and organic ligand interactions with the effect of coordination reactions of the molecular orbitals in the ligand which can be related to the electrochemical properties (e.g. half-wave potential). In addition, important information can be obtained concerning the ability of the ligand to donate electrons to metal ions in the process of forming chemical bonds, and further, to identify the type of molecular species resulting from such reactions. The kinds of information and chemical data which can be expected to be generated in this project should be of fundamental interest in several areas of electrochemical analysis

and of importance to scientists working in coordination chemistry, especially those engaged in chemical problems in such fields as coordination polymers and biomedical research involving metal ion-ligand interactions.

### Results and Implications to Date

The work completed to date represents the first phase of the electrochemical and spectroelectrochemical studies of metal ion - ligand interactions based on an investigation of the electrochemical property of ligand.

1. By application of DC polarography and cyclic voltametry on the 1-hydroxy-9,10-anthraquinone in DMF solutions in the presence of  $\text{Li}^+$  ion, the complexity of the electrode reduction of HOAQ to radical ion was demonstrated. The study established that the following factors must be considered in establishing exact quantitative relationships between the electrochemical magnitudes and the strength of metal ion - ligand interactions:
  - (a) The association of the metal ion with the electrode reaction product. This factor shifts the reduction potential in the positive direction. It is important to analyze the kinetic aspect of this interaction, especially with respect to the speed of reaching equilibrium.
  - (b) The association of the metal ion with the unreacted organic molecules by forming a covalent bond. This factor is especially important with aromatics carrying electron-donor groups or atoms and causes a shift of the reduction potential in the negative direction.
  - (c) The direct participation in electrochemical reaction of the complex species formed by association of organic molecules with metal ion. This process may be of considerable importance, together with the free electroactive species reduction, especially when more than one electroactive group resides on the organic molecule. The complex species will be reduced at a more positive potential than that of the free depolarizer. This factor is likely to be manifested as a change in DC polarographic reversibility.



Surely, all these factors cannot be adequately studied by using only electrochemical techniques. The necessity of taking recourse to other physiochemical methods, particularly to methods based on spectrophotometric and especially simultaneous spectroelectrochemical techniques, is quite obvious. In our own work , the optically transparent thin-layer cell proved advantageous in providing a satisfactory explanation of results observed, but not explained, by other techniques.

2. Mathematical models were constructed by deriving a theoretical expression from the Nernst equation for different types of metal ion - ligand interactions, and on these models a computer simulation of polarographic waves, as well as calculation of "theoretical" half-wave shifts was made.
3. Analog circuitry for electrochemical measurements based on operational amplifiers was manufactured and its DC polarographic part was connected on-line to a digital computer. Appropriate programs for data collection and analysis were worked out.
4. The use of optically-transparent thin-layer (OTTL) cells for a study of metal ion - ligand interaction with electrode reaction products was demonstrated and an appropriate OTTL cell was constructed.

#### List of Publications that Resulted from the Project

1. I. Piljac, M. Tkalcec and B. Grabaric, "Use of Optically Transparent Thin-Layer Cells for Determination of Composition and Stability Constants of Metal Ion Complexes with Electrode Reaction Products," Anal. Chem. 47, 1369 (1975).





Project Title: MASS SPECTROMETRIC STUDIES OF HIGH TEMPERATURE  
EQUILIBRIA

Principal Investigator: Dr. Kiro F. Zmbov

Institution: Boris Kidric Institute of Nuclear Sciences, Vinca

NBS Monitor: Dr. J. W. Hastie

NBS Institute or Center and Division: Institute for Materials Research  
Inorganic Materials Division

### Summary Description of Project Goals

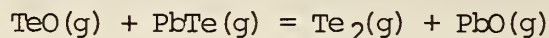
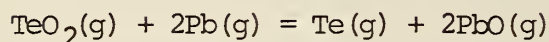
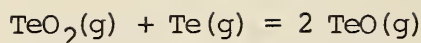
A Knudsen-effusion-mass spectrometric technique will be used to monitor chemical equilibria at high temperatures and low pressures. The bond dissociation energies of participating molecules will be determined.

An electron monochromator will be developed and utilized as an extension of existing characterization techniques for high temperature vapors. This will allow more accurate determination of molecular ionization and dissociation energies than existing techniques which utilize non-monochromatic low-resolution electron sources. Such a device should prove useful in the mass spectrometric detection of radical species, e.g., in flame studies.

### Results and Implications to Date

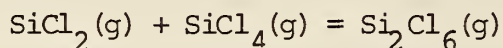
1. A Knudsen effusion cell for the study of high temperature equilibria has been constructed and attached to a mass spectrometer which has been designed previously in this laboratory.

2. The mass spectrometer-Knudsen effusion apparatus has been employed to study the following high temperature equilibria:



From the measured enthalpies and known heats of formation of PbO and PbTe molecules, the heats of atomization of TeO(g) and TeO<sub>2</sub> (g) molecules have been determined by isomolecular exchange reactions

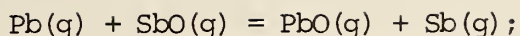
3. The composition of the gas phase above the reaction mixture CuCl + Si in a copper Knudsen cell has been investigated. From the mass spectra and appearance potentials of the ions the following parent molecules have been identified at temperatures near 200°C: SiCl<sub>2</sub>, SiCl<sub>4</sub>, Si<sub>2</sub>Cl<sub>6</sub>, Si<sub>3</sub>Cl<sub>8</sub> and Si<sub>2</sub>OCl<sub>6</sub>. The ionization potentials of these molecules have been measured and the reaction



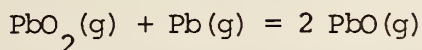
has been studied over a range of temperatures (160°- 200°C). Equilibrium constants for this reaction have been measured, and the reaction enthalpy determined by the second-law method. The data were used to obtain the Si-Si bond dissociation energy in the silicon hexachloride molecule.

4. Isomolecular exchange reactions involving gaseous atoms and oxides of lead and antimony at high temperatures have been studied.

Preliminary results have been obtained on the equilibrium constants and enthalpy change for the reaction -



Gaseous PbO has been detected for the first time at temperatures of  $800^{\circ}\text{--}950^{\circ}\text{K}$  and its ionization potential measured by semilog and extrapolated voltage difference methods. The isomolecular exchange reaction -



was studied and the dissociation energy of the  $\text{PbO}_2$  molecule was determined by the third law method.

These thermochemical studies are of direct interest to NBS in relation to its efforts to model the flame inhibiting effects of antimony.

5. A trochoidal electron monochromator (TEM) has been designed and attached to the ionization chamber of a Nier-type ion source of the mass spectrometer. The initial design has been modified in order to shield the electron beam from stray magnetic fields. The electron stopping curves have been measured by the retarding potential method, and the energy spread of the electron beam has been measured at both the entrance and exit of the monochromator. Beams with an energy spread of 40 mV were obtained, but their intensity was only of the order of  $10^{-8}$  amps.

The trochoidal electron monochromator has been used to study formation of negative ions by electron impact of  $\text{SF}_6$  and  $\text{SO}_2$ . The studies of  $\text{SO}_2$  gave interesting results. Besides the expected stable negative ions, ions were also detected which are analogous to

metastable ions in the positive ion spectra. The origin of these ions is still uncertain, and it is assumed that they are formed by decomposition of some primary ions of masses higher than the mass of the parent molecular ion. This indicates the existence of heavy ions formed either by ion-molecular reactions or by electron attachment to the dimer  $(\text{SO}_2)_2$  molecule. Further work is under way to elucidate the origin of these "metastable" ions.

This monochromator is of potential interest to NBS (and others) for molecular studies in high temperature and combustion chemistry. Further work is needed to demonstrate its utility in molecular beam systems.

6. A large number of stable inorganic molecules have been detected in high temperature vapors, but precise measurements of their ionization potentials are still lacking. The surface ionization method offers a possibility for measuring the ionization potentials and it has been applied in many cases, but only for atoms. It was therefore of interest to investigate the possibility of applying the method to determine the ionization potentials of gaseous inorganic molecules.

$\text{LaS}$  and  $\text{ThO}$  molecules were chosen for initial study. A surface ionization ion source was built in which the vapor of the sample was produced by heating in a tantalum oven and a heated metal ribbon served as the ionizing surface. The ions formed on the surface were analyzed by a conventional magnetic mass spectrometer equipped with an electron multiplier for ion detection.



Ions  $\text{La}^+$ ,  $\text{LaS}^+$  and  $\text{LaO}^+$  were detected when an LaS sample was heated in the Ta-oven and the vapors ionized on Ta and Re surfaces at temperatures between 1800 and 2500 K. Linear plots of  $\ln I(\text{LaS})^+$  vs  $1/T$  were obtained in the temperature range 2200 to 2500 K. From the slope of this plot, the ionization potential of the LaS molecule was calculated to be  $6.5 \pm 0.2$  eV.

When ThO vapors interact with a heated Re surface, ions Th and ThO are produced. The plot  $\ln I(\text{Th}^+)$  vs  $1/T$  is linear in the temperature range 2200 - 2600 K with an activation energy of  $9.8 \pm 0.2$  eV. This value can be explained as a sum of the dissociation energy of ThO and ionization potential of Th reduced by the work function of Re:

$$-E_a = W_{\text{Re}} - [ \text{I.P.}(\text{Th}) + D(\text{ThO}) ] .$$

Thus, the unusually high activation energy for the production of Th ions by surface ionization of ThO molecules offers a possibility of determining either the ionization potential of Th or  $D(\text{Th-O})$ .

Both ionization potentials and bond dissociation energies are needed in the interpretation of mass spectra and chemical processes for the molecular beam sampling of combustion systems.

#### List of Publications that Resulted from the Project

1. K.F. Zmbov, M. Miletic and M. Hofman, "Thermochemistry of  $\text{SiCl}_2$  and  $\text{Si}_2\text{Cl}_6$  Molecules and Bond Dissociation Energy of Si-Si in  $\text{Si}_2\text{Cl}_6$ ," Third Yugoslav Congress of Pure and Applied Chemistry, Ljubljana, June 1972.

2. R. Maksic, M. Miletic, A. Stamatovic and K.F. Zmbov, "Metastable Transitions in Negative Ion Spectra of  $\text{SO}_2$ ," 8th International Conf. Electron Atom Collisions, Belgrade, p. 463 (1973).
3. R. Maksic, M. Miletic, A. Stamatovic and K. F. Zmbov, "Negative Ions in  $\text{SO}_2$ ," 18th Annual Meeting of Serbian Chemists, Novi Sad (1974).
4. M. Miletic, A. Stamatovic, R. Maksic and K.F. Zmbov, "Modification of an Ion Source for Improvement of the Electron Energy Resolution," Adv. Mass Spectrometry 6, 523 (1974).
5. N. Djuric-Pregar, A. Stamatovic, M. Miletic and R. Maksic, "Mass Spectrometric Study of Negative Ion Formation in  $\text{SO}_2$ ," Proc. 7th Symp. Phys. Ionized Gases 7, 55 (1974).
6. A. Neubert and K. F. Zmbov, "Mass Spectrometric Determination of the Dissociation of  $\text{ThO}$  Molecule," High Temp. Sci., 6, 303 (1974).
7. A. Stamatovic and M. Miletic, "Metastable Ions from  $\text{SiCl}_4$ ," Proc. 8th Symp. Phys. Ionized Gases 8, 105 (1976).
8. K. F. Zmbov and M. Miletic, "Mass Spectrometric Determination of the Dissociation Energy of  $\text{PbO}_2$  and Ionization Potentials of  $\text{PbO}$  and  $\text{PbO}_2$  Molecules," Adv. Mass Spectrom. 7 (1976).
9. H.R. Ihle, C.H. Wu, M. Miletic and K. F. Zmbov, "Mass Spectrometric Study of Vapor Species in Si-Li and Si-Cl System," Adv. Mass Spectrometry 7 (1976).

Project Title: X-RAY DIFFRACTION AND ELECTROMICROSCOPIC STUDIES ON ALUMINUM AND ON THE METASTABLE PHASES OF ALUMINUM BASE ALLOYS, OBTAINED BY RAPID QUENCHING FROM THE MELT

Principal Investigator: Professor Dr. A. Bonefacic

Institution: Institute of Physics of the University of Zagreb, Zagreb

NBS Monitor: Dr. Richard P. Reed

NBS Institute or Center and Division: Institute for Basic Standards  
Cryogenic Division

### Summary Description of Project Goals

By rapid quenching from the melt, supersaturated solid solutions, new metastable phases and amorphous phases unobtainable by classical quenching procedures may be obtained.\* Quenching from the liquid state results in a high concentration of defects and small grain size formation, so that pure materials splatcooled from the melt differ in certain properties from the recrystallized samples.

The principal purposes of the project are:

1. Examination of the structural properties of aluminum-based supersaturated solid solutions directly after quenching and of the diffraction, electron-microscopy and hardness measurements.
2. Comparison of the structural and mechanical properties of alloys quenched from the liquid state with those of alloys quenched from the solid.
3. Investigation of pure aluminum quenched from the liquid.
4. Study of the applicability of X-ray cameras for quantitative scattering measurements, with respect to the error introduced by the distortion of the true scattering curves. Errors due to different forms and sizes of aperture and the collimation error due to the width of the incident beam and its divergence are to be considered.

---

\* Pol Duwez, R. H. Willens and W. Klement Jr., J. Appl. Physics 31, 1136 (1960).

Pol Duwez, R. H. Willens and W. Klement, Jr., Ibid, p. 1137.

Pol Duwez and R. H. Willens, Trans. Metallurgical Soc. of AIME, 227, 362-365 (April 1963).



## Results and Implications to Date

The project was signed by the U.S.-Yugoslav Joint Board on Scientific and Technological Cooperation on 13 May 1974, but work related to this investigation began earlier and funds from other sources were intermixed with those from this project. The present project is part of a long-term research program on metastable metallic alloys of the Institute of Physics of Zagreb University. The results obtained during the recent period are described in the same order as listed in the summary description of the project goals.

1. Aluminium-based supersaturated solid solutions Al-Ni, Al-Sn and Al-Pt were studied.

The solid solubility of Pt in Al was extended from 0 to 2 atomic percent by the "two-piston" quenching technique. The stability of the Al-Pt solid solutions during annealing was examined. During the decomposition an intermediate metastable phase was detected, and its unit cell constants were determined (2).

The intensity of scattering of monochromatic X-rays at small angles from Al-3.6 at.% Ni and Al-0.26 at.% Sn solid solutions was studied in order to determine if the solutes are dispersed. Electron-microscopic examinations were also carried out. Both alloys were obtained by rapid quenching from the melt to obtain the optimum dissolution. No perfectly homogeneous solid solutions were obtained, but agglomerations of solute atoms were detected. The evolution of clusters during annealing was examined and their influence on microhardness and microstrain properties evaluated (7).

Fourier analysis of the shape of diffraction peaks showed that in splat-quenched Al-Sn and Al-Ni solid solutions the major sources of peak broadening is microstrains which increase with increasing concentration of solute atoms (3).

2. The age hardening of aluminium-4.5 at.% silver alloys quenched from the liquid state (LQ) indicates that the decomposition processes in rapidly cooled specimens differ from those in conventionally quenched ones. Guinier-Preston (G.P.) zones were present in the as-quenched state and were about half the size of those in the specimens quenched from the equilibrium solid solution state (SQ). The precipitates nucleated, grew, and disappeared more readily in the liquid-quenched than in the solid-

state-quenched specimens, causing an overlap of cold and warm hardening stages and an earlier attainment of maximum hardness with aging (1).

G.P. zones which form in certain solid solutions may be represented by several models of particles of non-uniform electron density. Small-angle X-ray scattering curves for various models of spherically symmetric particles have been calculated. The scattering functions for particles having a Gaussian and a cosine distribution of electron density, and for dilute systems of such particles of various degree of polydispersity were calculated and compared with the scattering of spheres of uniform electron density, considering the statistics both of mass and of the number of particles (4).

Other models being studied are:

- (a) the gradual transition of electron density is restricted to the outer shell of the particle;
- (b) the electron-density transition is partly gradual and partly sharp; and
- (c) the particle consists of two parts of uniform electron density which may be of opposite sign.

The latter model includes the Guinier-Walker model which has been proposed to represent the G.P. zones in aluminum-silver alloys. In the large-angle region all scattering curves oscillate around a curve which runs proportionally to  $(2\theta)^{-n}$ , where  $n$  is an even number. Whenever the transition of electron density is gradual, the exponent  $n$  exceeds 4. However, even if a small abrupt drop of electron density is present, the value of  $n$  becomes 4 (Porod's Law). In the small-angle region the scattering curves are approximately of the Gaussian form, except for some models of particles which have an excess of electrons in the inner part and a deficit in the outer part (and vice versa) with respect to the surrounding medium. For these particles, some parameters characterizing the scattering curves were calculated to facilitate the evaluation of the experimental scattering curves.

An attempt was made to show whether the early stage of decomposition of the quenched Al-Ag alloy could be explained by the linear spinodal (LS) theory. Only the initial time dependence of small-angle-scattering spectra recorded in the lower-angular range explored was in rather good agreement with the LS theory. The interdiffusion coefficients were determined from these data. The value for the SQ specimen was one order of magnitude greater than the corresponding value for the IQ specimen, both annealed at 140°C. For lower temperatures, this difference



became smaller.

3. Measurements of the broadening of X-ray diffraction peaks were made for splat-cooled pure aluminum samples (3). Using the Warren-Averbach profile analysis method, it was found that the major source of broadening is strain. The residual strains calculated from the dislocation density and measured with an electron microscope were of the same order of magnitude as that obtained by the line-broadening measurements ( $10^{-4}$ ). Microstrains may appear as a consequence of a large temperature gradient during quenching. No preferred orientations of crystallites were observed in the quenched samples. The deformation fault probability determined by the peak-shift method was very small (0.004). No faulting in splat-cooled aluminum samples could be detected by an electron microscopic examination.

4. The error introduced by distortion of the true X-ray scattering curves as measured with the Levelut-Guinier camera was considered. The distortion of different types of curves, representing different scattering systems, was found by calculation, and the character and the amount of distortion evaluated (5).

The study of the distortions of scattering curves which emerged when applying convergent and divergent incident beams showed that the distortion of the Gaussian curves is greater in the case of the convergent beam. The presence of  $s^{-4}$  dependence in the tail of the scattering curve (Porod's Law) was more clearly revealed in the case of the convergent beam (6).

To summarize, the principal results of the project have been:

A variety of super-saturated solid solutions and new metastable phases, unobtainable by classical procedures, were obtained. Comparative studies of their mechanical and structural properties have been made.

Special efforts were made to correlate the microhardness, X-ray small-angle scattering intensity, and X-ray peak-broadening microstrain measurements, to indicate solute-atom clustering in the series of new solid solutions.

Theoretical and experimental substantiation was made of the small-angle scattering X-ray technique to identify solute atom

clustering.

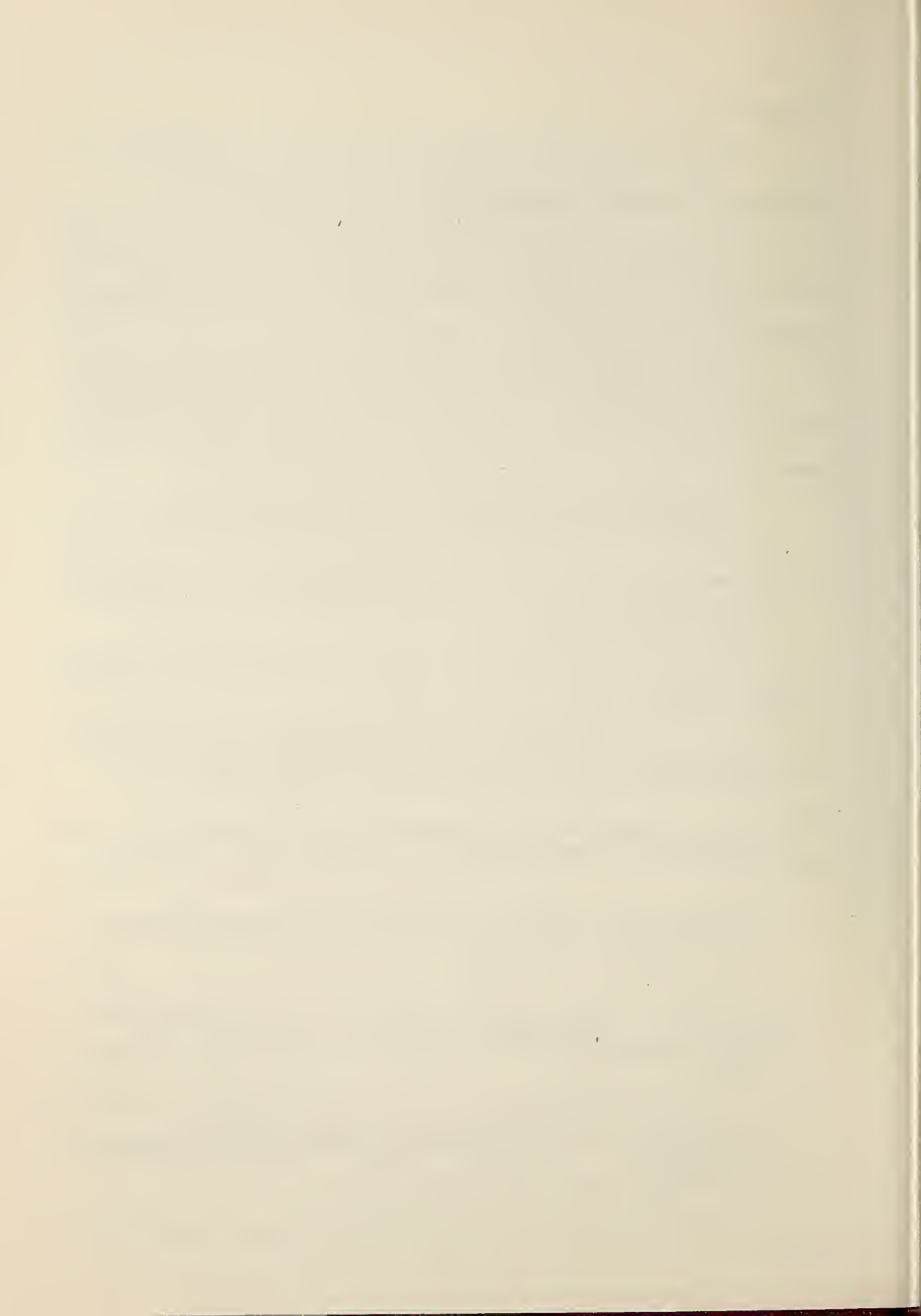
Electron-microscopic examinations were used to study the evolution of clusters during ageing.

The techniques of alloy characterization used in this project may prove of great utility in further studies of stress-corrosion, metallic glasses, and in alloy development.

By systematic and careful measurements on judiciously chosen series of new splat-cooled alloys, important regularities and laws governing the general behavior of alloys may be clarified.

#### List of Publications that Resulted from the Project

1. K. Kranjc and M. Stubicar, "X-ray Diffraction Study of Decomposition in Aluminium-Silver Alloys Quenched from the Melt," Met. Trans. 4, 2631 (1973).
2. A. M. Tonejc and A. Bonefacic, "Nonequilibrium Phases in Al-rich Al-Pt Alloys, Mat. Sci. 9, 523 (1974).
3. A. Kirin and A. Bonefacic, "X-ray Line Broadening Study of Splat Cooled Aluminium and Aluminium-tin and Aluminium Nickel Solid Solutions," J. Phys. F: Metal Phys. 4, 1608 (1974).
4. K. Kranjc, "Small-Angle X-ray Scattering from Spherical Particles of Non-uniform Electron Density," J. Appl. Cryst. 7, 211-218 (1974).
5. K. Kranjc and A. Bonefacic, "Distortion of X-ray Small Angle Scattering Curves Measured by a Levelut-Guinier Camera," J. Appl. Cryst. 7, 259 (1974).
6. K. Kranjc, "Distortion of X-ray Small-Angle Scattering Curves Measured by a Levelut-Guinier Camera when the Primary Beam Converges at a Point on the Counter," J. Appl. Cryst. 7, 498 (1974).
7. A. Bonefacic, M. Kerenovic, A. Kirin and D. Kunstelj, "Segregation of Solutes in Al-Ni and Al-Sn Alloys," J. Mat. Sci. 10, 243 (1975).



Project Title: STARK BROADENING OF SPECTRAL LINES IN PLASMAS

Principal Investigator: Dr. N. Konjevic

Institution: Institute of Physics, Belgrade

NBS Monitor: Dr. W. L. Wiese

NBS Institute or Center and Division: Institute for Basic Standards  
Optical Physics Division

### Summary Description of Project Goals

This project covers a study of the Stark broadening and shift of prominent lines of some singly, doubly and triply ionized atoms in plasmas. The experimental results are compared with semiclassical calculations based on various theoretical approximations.

The principal goal of this work is to supply new experimental Stark broadening data for spectral lines of ionized atoms and to test the various theoretical approaches for line width calculations. A part of the work is devoted to the search for the systematic trends of the Stark line widths within an element and within a group of elements. Critical evaluations of existing experimental Stark broadening data are also carried out.

### Results and Implications to Date

Work in the last three years was concentrated on the following tasks:

1. obtaining new Stark broadening data for lines of elements for which measurements did not exist and comparing them with existing theories
2. finding possible regularities of Stark broadened line widths within an element and within a group of elements, and



3. critical evaluation of existing Stark broadening data

The last two parts of the Project were carried out in collaboration with scientists from the National Bureau of Standards.

1. During the course of this work a number of singly, doubly and triply ionized atoms were investigated as given in Table 1.

Table 1

Element	Ionization State	Number of Lines Investigated
Beryllium	Be II	2
Nitrogen	N II	9
	N III	4
Oxygen	O II	21
	O III	6
Fluorine	F II	5
Magnesium	MgII	2
Chlorine	Cl III	15
Argon	Ar III	5
	Ar IV	2
Calcium	Ca II	4
Strontium	Sr II	2
Barium	Ba II	5

A low pressure pulsed arc was used as a light source in these experiments. The electron density was measured by laser interferometry while electron temperatures were determined from the Boltzmann plots of relative intensities of spectral lines. Electron densities were typically around  $5 \times 10^{17} \text{ cm}^{-3}$  and temperatures in the range 22000-26000 K.

Results for singly ionized lines of Be II, Ca II and FII agree within the limits of experimental error with semiclassical calculations by Griem.<sup>1</sup> In the case of F II, in addition, calculations of the line widths have been performed according to the theory of Baranger<sup>2</sup> where hyperbolic perturber-path trajectories were used. Theoretical results were also obtained combining Baranger's approach with GBKO straight perturber-path approximations for higher perturber velocities.<sup>3,4</sup>

The agreement between the F II experiment and our semiclassical calculations for singly ionized fluorine lines is good while the results based on Baranger's theoretical approach are systematically higher. It follows from this comparison that Baranger's perturber-path approximation (hyperbolic trajectories) is not adequate for singly ionized atoms.

However, for the doubly and triply ionized atoms Ar III, Ar IV and Cl III the experiments agree best with theoretical results based only on hyperbolic perturber-path trajectories while the results of other theoretical approximations are systematically lower. This suggests that the approach of Baranger is much better for higher ionization stages than one would expect on the basis of comparison

with results for singly ionized atoms. This conclusion may be of some practical importance since Baranger's approach offers by far the simplest method to evaluate line widths.

2. Stark broadening theory predicts regularities and systematic trends for the Stark widths of isolated neutral atom and ion lines on the basis of aspects of plasma as well as atomic physics. The systematic behaviour of Stark widths with regard to plasma parameters has been proven in a number of experiments. However it is still a largely unsettled question what regularities should be exhibited in the width data as a consequence of atomic physics factors contained in the line width expression.

On the basis of the present Stark broadening theory one would expect:

I. Some similarities within a given spectrum:

- a. The line widths of all lines in a multiplet should be practically identical.
- b. The width of all lines in a supermultiplet should be approximately equal, and
- c. Groups of supermultiplets having the same parent term in a transition array should possess Stark widths of about the same size.

II. Regularities within homologous atoms, and

III. Regularities within isoelectronic sequences.

Unfortunately a fairly large number of reliable experimental data are needed in order to check theoretical predictions of the systematic trends. The whole work in this field is in an early stage but the first results are very encouraging. As an example, Table 2 gives our results for the line widths of resonance lines of alkaline earth

atoms (homologous atoms). From the table one can readily notice a systematic increase of the line width with increase in atomic number (and with decrease of ionization potential).

Table 2

Ion	Transition	Stark halfwidths ( $\text{\AA}$ )
Be II	$2s^2S - 2p^2P^\circ$	0.05
Mg II	$3s^2S - 3p^2P^\circ$	0.08
Ca II	$4s^2S - 4p^2P^\circ$	0.20
Sr II	$5s^2S - 5p^2P^\circ$	0.23
Ba II	$6s^2S - 6p^2P^\circ$	0.25

3. Within the scope of our work on the Stark broadening of atomic lines in plasmas an attempt was made to evaluate critically all existing experimental data on the Stark broadening of nonhydrogenic spectral lines of neutral and ionized atomic species. The relevant literature compiled by the NBS Data Center on Atomic Line Shapes and Shifts was therefore critically evaluated and 68 papers dealing with neutral and 54 with ionized atoms were selected. The experimental data were then arranged according to the spectra and elements presented in alphabetical order. The accuracy of the experimental data was estimated on the basis of guidelines developed in the reviews, and comparisons with theoretical results were made whenever possible (6,7).



As a result of this work two review papers are now in print. To our knowledge these two reviews represent the first attempt to assemble and evaluate critically the experimental material on the Stark broadening parameters of isolated lines.

#### References:

1. H.R. Griem, Spectral Line Broadening by Plasmas Academic Press, New York (1974).
2. M. Baranger, Atomic and Molecular Processes edited by D.R. Bates, Academic Press Inc., New York (1962).
3. J. Cooper and G.K. Oertel, Phys. Rev. Let. 18, 985 (1967).
4. J. Cooper and G.K. Oertel, Phys. Rev. 180, 286 (1969).

#### List of Publications that Resulted from the Project

1. D. Hadziomerspahic, M. Platisa, N. Konjevic and M. Popovic, "Stark Broadening and Shift of Some Isolated Spectral Lines of Singly Ionized Alkaline Earth Metals," Z. Phys. 262, 169 (1973).
2. M. Platisa, M. Popovic, M. Dimitrijevic and N. Konjevic, "Stark Broadening of AIII and AIV Lines," z. Naturforsch. 30a, 212 (1975).
3. M. V. Popovic, M. Platisa and N. Konjevic, "Stark Broadening of NII and NIII Lines" Astron. Astrophys. 41, 463 (1975).
4. M. Platisa, M. V. Popovic and N. Konjevic, "Stark Broadening of OII and OIII Lines," Astron. Astrophys. 45, 325 (1975).
5. M. Platisa, M. Dimitrijevic, M. V. Popovic and N. Konjevic, "Stark Broadening of FII and CIII Lines," in press. (To be published in Astron. Astrophys.)
6. N. Konjevic and J. R. Roberts, "A Critical Review of the Stark Widths and Shifts of Neutral Non-Hydrogenic Spectral Lines," J. Chem. Phys. Ref. Data, 5, 209 (1976).
7. N. Konjevic and W. L. Wiese, "Experimental Stark Widths and Shifts for Isolated Ionic Lines," J. Chem. Phys. Ref. Data, 5, 259 (1976).

Project Title: MEASUREMENT OF F-VALUES IN THE ULTRAVIOLET AND VISIBLE REGION AND STUDY OF CONTINUOUS EMISSION OF HYDROGEN NEAR THE BALMER LIMIT

Principal Investigator: Dr. Vladis Vujnovic

Institution: Institute of Physics, University of Zagreb

NBS Monitor: Dr. Wolfgang L. Wiese

NBS Institute or Center and Division: Institute for Basic Standards  
Optical Physics Division

### Summary Description of Project Goals

The primary aim of the experimental investigation is to provide data on atomic transition probabilities. As an element of special interest, phosphorus is introduced into a wall stabilized arc chamber and the oscillator strengths are determined on a relative scale in the visible region. The oscillator strengths will be placed on an absolute scale by using an available absolute value of one of the lines, which has been determined by another technique (e.g. a life-time measurement).

The theoretical part of the project is undertaken to shed more light on the processes responsible for the hydrogen spectrum formation near the Balmer limit. The experimentally acquired data from the NBS Plasma Spectroscopy Section are fitted on the basis of a theoretical analysis.

### Results and Implications to Date

Measurements were made in a wall stabilized arc. The phosphorus pentafluoride was introduced into the central portion of the discharge column, where it mixed with argon introduced through the electrode ends. For the measurements, four well isolated spectral lines of P I and four of P II were chosen in the range from 400 to 650 nm.

Optical diagnostics and measurements were performed at the axis of the arc whose diameter was 3 mm. Test measurements showed a linear increase of the phosphorus spectral line intensities with the length of the phosphorus-gas layer, and no more than a 5% variation of intensity during the experimental runs. This observation assures good mixing of the phosphorus vapour. The optical depth of the spectral lines was checked. The relative spectral response of the monochromator was determined by using a tungsten strip lamp and a carbon arc. The relative spectral response of the monochromator was determined by using a tungsten strip lamp and a carbon arc. The oscillator strengths were normalized to the absolutely known oscillator strength of the spectral line P I 547.8nm ( $f=0.038$ ). The relative values of the other lines had an error corresponding mainly to the error of temperature measurements ( $11500 \text{ K} \pm 800 \text{ K}$ );

	LINE	f	
PI	507.9 nm	$0.0067 \pm 0.0020$	
PI	609.7	0.025	0.008
PI	619.9	0.022	0.007
PII	515.2	0.016	0.008
PII	529.6	0.030	0.015
PII	531.6	0.054	0.027
PII	541.0	0.073	0.037

During the measurements, corrosion of the gas-filling system was observed and a decision was made to rebuild the experimental system in order to prevent any of the corrosive influence and eventual poisonous effects of the gas used. For this purpose noncorrosive reduction valves were built in, as well as noncorrosive flow meters and a filter for HF, which constitutes the greatest impurity in the PF gas. In the vacuum system, electromagnetic valves were included which render faulty operation of pumps almost impossible (this applies equally to the mechanical and the diffusion oil pumps). The overall system is remotely controlled from the main control board. In the spring of 1976, the arc instrumentation was refined by a novel high-voltage ignition of the arc, a remotely controlled high current rectifier, a 0.001 ohm standard resistor, and a digital voltmeter for current reading to 0.01 A, and a smoothing filter which diminishes the voltage ripple to less than 1% at 500 V. By operating all components from a main control panel, and using foolproof sub-systems, a high degree of reproducibility was obtained and at the same time current adjustments were facilitated.

Further measurements will be done with this improved version of the experimental assembly, aimed at the determination of more oscillator strengths with higher precision and under conditions of higher safety for the operators. During the construction of the assembly, a 1-m visible monochromator made by McPherson was obtained and tested, together with several smaller items, including a deuterium lamp calibrated at the NBS.



Calculation of the Balmer recombination spectrum in the statistical interpretation was extended to four experimental situations (ion concentrations of 1.8, 2.9, 5.7 and  $9.3 \times 10^{16} \text{ cm}^{-3}$  with the corresponding temperatures of 9900, 10600, 12000 and 13000 K). By variations of semi-empirical parameters describing the ionization energy decrease and the quasi-static influence of electrons, the statistically formed recombination of ionization energies helped to evaluate the Balmer recombination spectrum which does not show a sharp limit. In order to match the experimental situations to the calculated recombination continuum, four other contributions were added: the  $\text{H}^+$  continuum,  $\text{H}^-$  continuum,  $\text{H}$  continuum and the line sum (all these contributions according to evaluations of Roberts and Voigt from NBS\*). However, the essential step was in further applications of the dissolution to the line sum. With no dissolution, any choice of ionization energy decrease produced a nonobservable increase of the spectrum intensity on the long wavelength side of the Balmer limit. After comparison of the summed calculated spectra with the spectra observed at NBS\*\*, agreement was found for all four experimental situations, using constant semi-empirical parameters, with 5-10% differences in various spectral regions.

The project in Zagreb materially assisted the training of five physics students and the results are incorporated in one diploma (ref. 1) and one master's thesis (ref. 2). The determination of Atomic Transitions Probabilities for phosphorus is important to NBS in

continuing work on the critical compilation of atomic reference data for the NSRDS system. A first critical compilation on phosphorus several years ago showed large gaps in the data, probably because this element is chemically rather aggressive and thus difficult to work with. The resourceful work of the Yugoslavian group will soon provide accurate data for the neutral phosphorus spectrum, and thus constitutes a valuable service for atomic reference data.

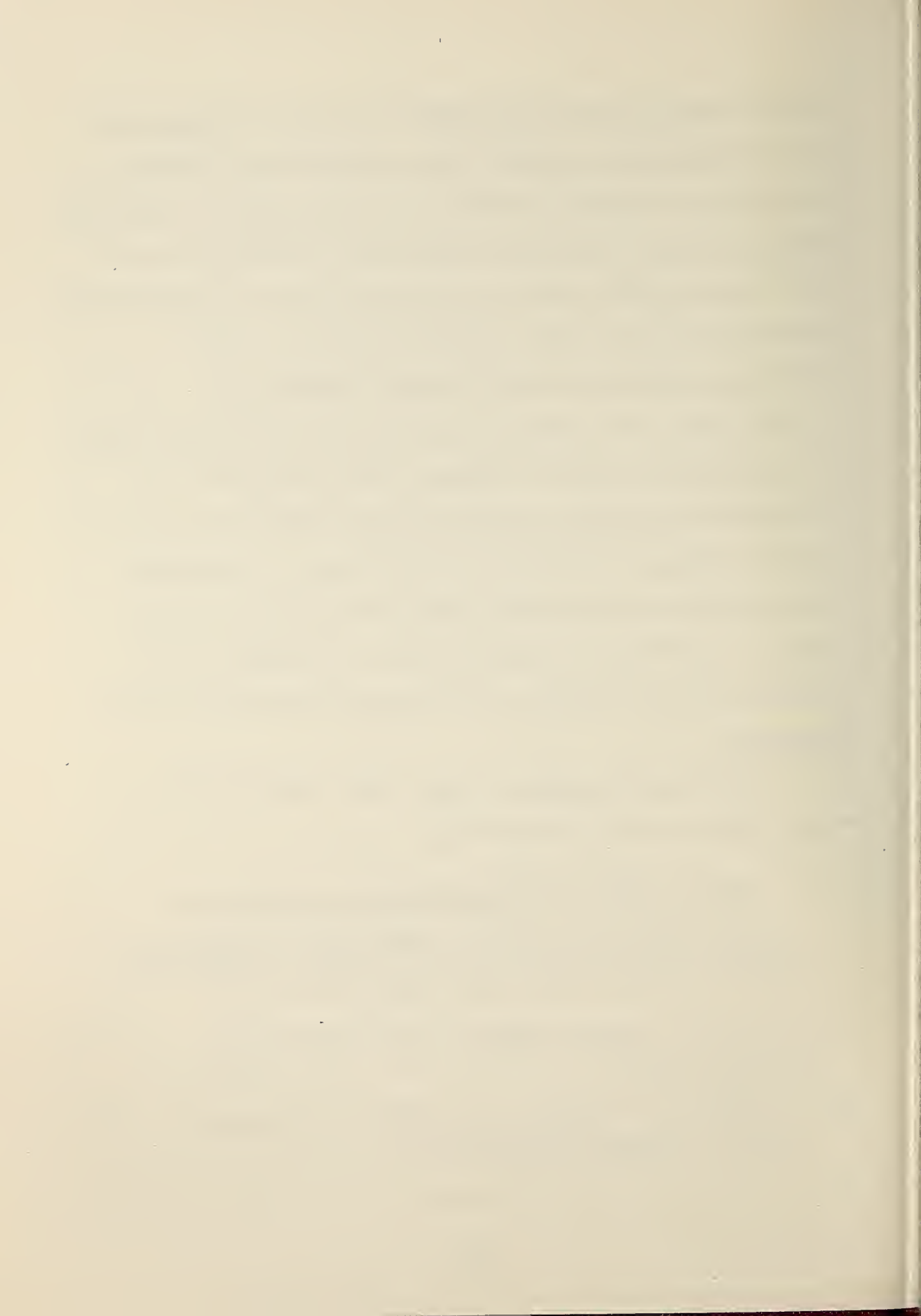
The high-current hydrogen-arc was developed at NBS several years ago as a standard of spectral radiance in the ultraviolet. The emitted continuum radiance must be thoroughly understood in all wavelength ranges for quantitative applications. Dr. Vujnovic's theoretical work on this subject appears to be the most promising solution to provide a reliable quantitative understanding of the continuous hydrogen spectrum in the principal recombination regions.

#### References:

- \* J.R. Roberts and P.A. Voigt, J. Res., NBS, 75A,291 (1971).
- \*\* W. L. Wiese, private communication.

#### List of Publications that Resulted from the Project

1. V. Lokner, "Relative Transition Probabilities and Oscillator Strengths of PI and PII in the Visible Region," Diploma Thesis (in Croatian), the University of Zagreb (January 1975).
2. V. Ruzdjak, "Hydrogen Spectrum at the Ionization Limit," (in Croatian), Master's Science Thesis, the University of Zagreb (1975).
3. V. Ruzdjak and V. Vujnovic, "Astronomy and Astrophysics," (in press, accepted for publication).



Project Title: ELECTRON-ATOM EXCITATION CROSS SECTION MEASUREMENTS

Principal Investigator: Dr. V. V. Urosevic

Institution: Institute of Physics, Belgrade University

NBS Monitor: Dr. G. Dunn

NBS Institute or Center and Division: Joint Institute for Laboratory  
Astrophysics

### Summary Description of Project Goals

The project is concerned with the absolute cross section excitation measurement of some hydrocarbons using He excitation cross section of  $4^1D - 2^1P$  and  $4^1S - 2^1P$  transition as the "excitation standard."

### Results and Implications to Date

During 1975 the experimental apparatus for cross section measurements of excitation of atoms and molecules was transformed into apparatus for automatic accumulation of experimental data. The automation of the apparatus has been performed in the first phase as an experiment for automatic measurements of optical spectra obtained from the excitation chamber of the apparatus at a defined electron energy. By changing in succession the electron energy in this apparatus in the interval 50-500 eV it is possible to measure simultaneously several excitation cross sections, i.e. for each spectral line detected, a measured optical spectrum.

The second phase of automization of this apparatus is also being planned. The purpose of this phase is to measure automatically the excitation functions of one spectral line for the energy interval of electrons from 1 - 1000 eV.



List of Publications that Resulted from the Project

1. B. Levi, J. Kurepa and B. Panic, "Automatic Measurements of Optical Spectra of very Small Intensities," XIX Yugoslav Conference on ETAN, Ohrid, pp 2-5 (June 1975).
2. J. M. Kurepa, S. S. Manola and V.V. Urosevic, "The Excitation Processes of Ammonia Molecules in the Interaction with Electrons." To be reported on VIth Congress of Mat. Phys. Astr. of Yugoslavia, Novi Sad (September 1975).

Project Title: BULK COMPOSITION AND SURFACE PROPERTIES OF GLASS

Principal Investigator: Dr. Velimir Pravdic

Institution: Rudjer Boskovic Institute, Zagreb

NBS Monitor: Dr. Wolfgang Haller

NBS Institute or Center and Division: Institute for Materials Research  
Inorganic Materials Division

### Summary Description of Project Goals

The objectives of this project have been to study the surfaces of glasses of well defined bulk composition and to find correlations between selected surface parameters and the bulk chemical composition.

In spite of extensive research into the structure, chemical composition, and properties of glasses, a clear understanding on how the bulk composition and the chemical and physical treatments influence surface properties is still largely missing. A fuller understanding of the correlations between bulk and surface properties could be of considerable technological importance wherever glass is being used.

The properties of glass surfaces studied include adsorption phenomena and charge density and distribution, as measured by electrokinetic techniques of streaming potential and surface conductivity, by ESR hyperfine splitting, and by heats of immersion and continuous flow microcalorimetry.

### Results and Implications to Date

The samples of glass, in powder or capillary forms, were supplied by the NBS Inorganic Glass Section. They were well characterized with respect to their chemical composition, concentration of minor

constituents (impurities), and structural elements (pore size and volume, domain development). The surface parameters were determined in the Laboratory of Electrochemistry and Surface Phenomena, of the Rudjer Boskovic Institute, using the techniques and the expertise developed in this group.

Glasses so far studied have been, partly, commercially available samples such as crystalline quartz, fused silica, doped silica, Vycor, Pyrex (7740), as well as a selection of controlled pore glasses produced in the NBS Laboratories.

It has been shown that electrokinetic measurements do not reveal large differences between pure and doped silica and quartz. Alumina impurities tend to decrease the negative potentials in quartz, but do not influence the properties of fused silica. Application of surface charge formation criteria based on the model of the diffuse double layer indicate that the mechanism of pH dependent adsorption of potential determining ions is dominant for quartz and silica. The incidence of surface boronol groups in glasses with an appreciable amount of  $B_2O_3$  (as in Vycor and Pyrex) results in a behavior characteristic for a constant surface charge model. Effective charge densities, based on observations on both these models, should not exceed  $10^{13}$  unit charges per  $cm^2$ . Indeed, these observations were borne out by experiments involving both measurements of electrokinetic streaming current and of ESR hyperfine splitting for  $^{107}Ag$  adsorbed from aqueous solutions. Characteristic values found were  $3 \times 10^{12}$  and  $7 \times 10^{12}$  unit charges per  $cm^2$ , respectively.

Heats-of-immersion microcalometry with a series of dipolar liquids using controlled pore glasses revealed that the contribution of polar forces to total interfacial energy was increasing from 35% for a mean pore size of 225 nm, to 38% for 55 nm, and to 45% for 15 nm, the smallest pore size material studied. The integral heats of immersion into water were measured and found in the range of  $0.30 \pm 0.03 \text{ J/m}^2$  for all the 4 types of controlled pore glasses studied, identifying these as exhibiting moderately high energy surfaces.

Flow microcalorimetric studies were used to investigate the energies of adsorption of a homologous series of lower normal aliphatic alcohols from hexane solutions. It was found that the integral molar heats of adsorption are almost constant in the series from methanol to n-pentanol, and amounts to  $15.4 \pm 0.5 \text{ kJ/mol}$ . It was also found that the surface area occupied per alcohol molecule is  $0.17 \pm 0.01 \text{ nm}^2$ .

Surface conductivity studies for fused silica and pyrex surfaces in methanol and acetonitrile indicated a twofold mechanism: one operative at lower temperatures (below  $10^\circ\text{C}$ ) characterized with low energies of activation, and another at higher temperatures with high energies of activation. Surface conductivities at silica surfaces were one order of magnitude lower than those observed at pyrex surfaces.

Continuation of the present research work is directed primarily to further investigation of adsorption properties at the glass/ solution interface. Modified surfaces with inorganic and organic ligands will



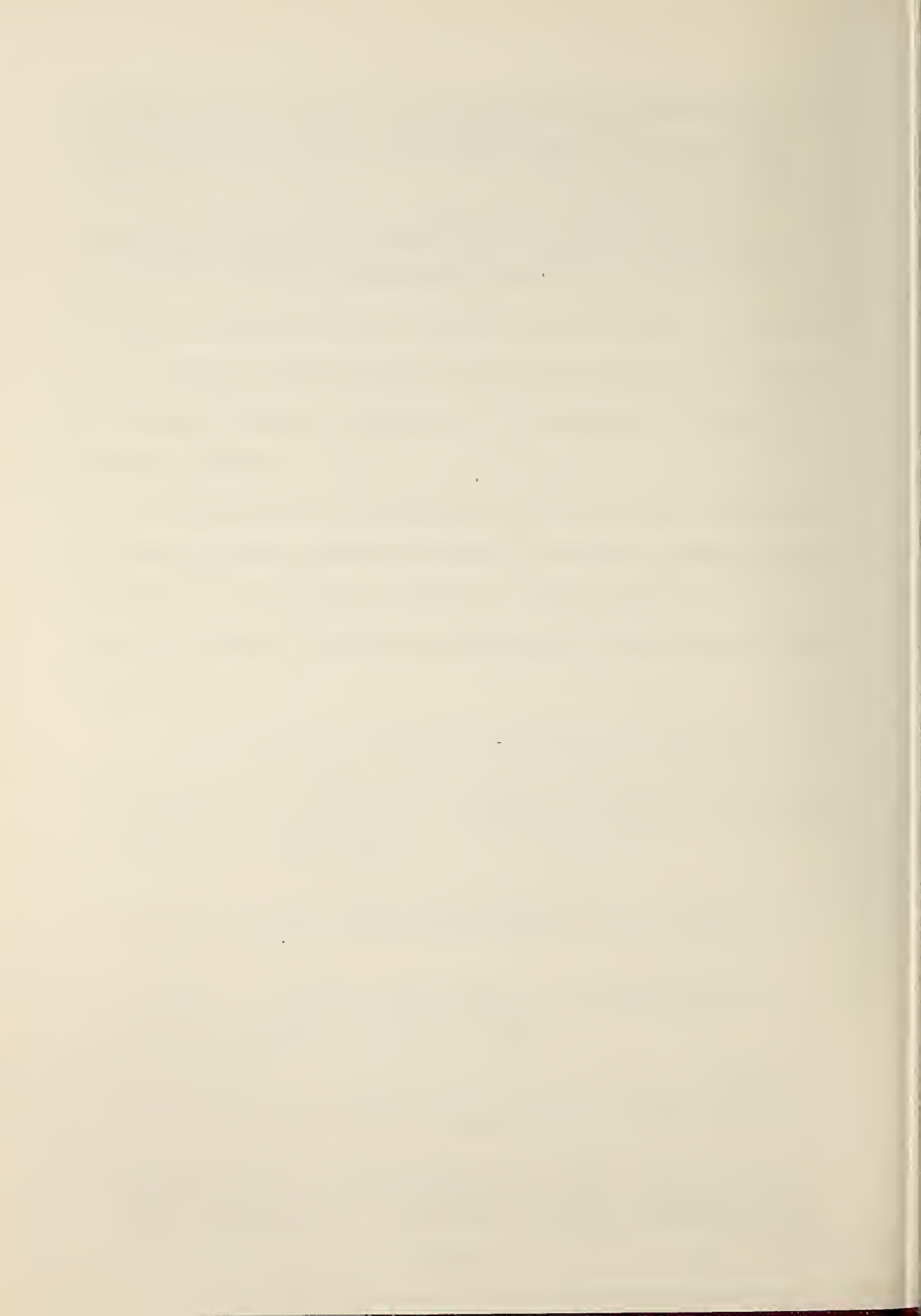
be studied, using as adsorbates polyelectrolytes, organic dyes, polypeptides, and, possibly, some well characterized biological substances. This research is aimed at understanding the behavior of glass in the presence of biologicals, in terms of adsorption energetics and interfacial electrification, and to see to what degree the surface ligands screen the influence exerted by bulk properties of glass. Results of this research could provide useful data for the application of glass as containers in the biological and chemical container industry.

A further area of application is the use of quartz, silica and some aluminosilicate glasses as models in studies of adsorption and immobilization of organic substances in marine sediments, a contribution towards problems of environmental effects and pollution control.

#### List of Publications Which Resulted from the Project

1. J. Jednacak, M. Hudomalj, V. Pravdic and W. Haller, Electrokinetics of Glass Surfaces. I. Charge Densities at the Solid/Gas and the Solid/Liquid Interfaces of Porous Glasses," Croat. Chem. Acta 44, 149 (1972).
2. J. Jednacak and V. Pravdic, "Electrokinetics of Glass Surfaces," Proc. 3rd. Yugoslav Congress for Pure and Applied Chemistry, Ljubljana, June 1972.
3. J. Jednacak, V. Pravdic and W. Haller, "The Electrokinetic Potential of Glasses in Aqueous Electrolyte Solutions," J. Colloid Interface Sc. 49, 16 (1974).
4. V. Mikac-Dadic, J. Jednacak-Biscan and V. Pravdic, "Surface Conductivity at the Glass/Liquid Interface," Meeting of the Chemical Society of Croatia, Zagreb, February 1975.
5. V. Mikac-Dadic, J. Jednacak-Biscan, V. Pravdic and W. Haller, "Mechanism of Electric Conductance of a Glass Surface in Contact with a Liquid Phase," 4th Yugoslav Symposium on Electrochemistry, Dubrovnik, June 1975.

6. J. Jednacak-Biscan, V. Mikac-Dadic, V. Pravdic and W. Haller,  
"Surface Conductivity of Glass at the Solid/Liquid Interface"  
52nd Symposium on Colloid and Surface Science, Knoxville,  
Tenn., June 1978.
7. J. Jednacak-Biscan, V. Mikac-Dadic, V. Pravdic and W. Haller,  
"Surface Conductivity of Glass at the Solid/Liquid Interface,"  
(submitted to J. of Colloid and Interface Science).



Project Title: THEORETICAL INVESTIGATIONS ON PHYSICAL SURFACES

Principal Investigator: Prof. M. Sunjic

Institution: Rudjer Boskovic Institute, Zagreb

NBS Monitor: Dr. J. W. Gadzuk

NBS Institute or Center and Division: Institute for Basic Standards  
Optical Physics Division

### Summary Description of Project Goals

A theoretical investigation of crystal surfaces by means of spectroscopic methods will be carried out. Applying the many-body theory, the x-ray photoemission and photoabsorption phenomena will be analyzed. Special attention will be paid to the determination of electron attenuation lengths in x-ray absorption and Auger processes, the excitation of both volume and surface plasmons in x-ray photoemission, and inelastic processes in field-electron and -ion emission.

This work represents a continuation of a scientific collaboration between Dr. J. W. Gadzuk (NBS) and Dr. M. Sunjic which started two years ago.

### Results and Implications to Date

The major problem we have been considering over the past year pertains to the many-body modifications to the photoelectron lineshape of core state electrons of atoms in solids and on surfaces. Amongst the important processes, we include electron-hole pair shakeup, plasmon satellites, phonon broadening, and Auger decay broadening. We have chosen to formulate our problems in a space-time (rather than momentum energy) picture. The added clarity (at the



expense of some mathematical rigor) has made our work considerably more accessible and thus useful to non-many-body theorists.

Specifically, we have continued our studies into time dependent relaxation effects which were first published in our Physical Review paper.\* As a result, we have been invited to give back to back lectures at such meetings as the "International Photoemission Sympsiu" which was held in Noordwijk, Netherlands in September 1976. We held special tutorial sessions on many-body effects in surface spectroscopies at a July 1976 Gordon Conference on Electron Spectroscopy. We have broken new ground in the area of coupled phonon excitation hole decay processes in x-ray photoemissions\*\* and will continue work in this area until a satisfactory resolution of some apparent paradoxes in existing theories is obtained. Our plans are to spend time together in September 1977 for our, at least yearly, coordination and writing.

#### List of Publications and Lectures that Resulted from the Project

1. J.W. Gadzuk, "Time Scales and Plasmon Satelllites in XPS" and M. Sunjic, "Scattering Effects in Photoemission from Localized Levals in Solids" in "Proc. of an International Symposium on Photoemission," European Space Agency Spec. Pub. 118 (Dec. 1976).
2. J. W. Gadzuk and M. Sunjic, Special tutorial lectures at a July 1976 Gordon Conference on Electron Spectroscopy (1976).

---

\* J. W. Gadzuk and M. Sunjic, Phys. Rev. B12, 524 (1975).

\*\* M. Sunjic and A. Lucas, Chem. Phys. Letters 42, 462 (1976).

Project Title: SYNTHESSES AND PROPERTIES OF FLUORIDE COMPLEXES

Principal Investigator: Professor Dr. Joze Slivnik

Institution: Jozef Stefan Institute, Ljubljana

NBS Monitor: Dr. T. D. Coyle

NBS Institute or Center and Division: Institute for Materials Research  
Inorganic Materials Division

#### Summary Description of Project Goals

This work deals with investigations of reactions of transition element fluorides with anhydrous hydrazine, the synthesis and isolation of hydrazinium fluorometalates containing first row transition elements, the preparation and characterization of xenon (II) and xenon(VI) fluorometalates of the first row transition series for the following purposes:

1. to develop new or improved methods of chemical synthesis for inorganic fluorine containing compounds
2. to advance the techniques of sample purification
3. to determine accurately the chemical and physical properties of isolated compounds where data will contribute to the understanding of such compounds

#### Results and Implications to Date

This is a third year report (the final report) and contains new results on the following topics:

1. Investigations of reactions of transition element fluorides with anhydrous hydrazine.
2. Syntheses of hydrazinium fluorometalates of the first transition series.
3. Synthesis and property studies of compounds formed between xenon difluoride and fluorides of elements of the first transition series.

In the course of the present work four MF-NH systems have been investigated, with M being nickel, chromium or iron. The following metal fluoride-hydrazine addition compounds have been isolated, described and characterized:  $\text{Ni}(\text{N}_2\text{H}_4)_2 \cdot 6\text{F}_2$ ,  $\text{CrF}_3 \cdot 3\text{N}_2\text{H}_4$ ,  $2\text{CrF}_2 \cdot 3\text{N}_2\text{H}_4$ , and  $\text{FeF}_2 \cdot 2\text{N}_2\text{H}_4$ . In addition some other compounds (e.g.  $\text{Ni}(\text{N}_2\text{H}_4)_3(\text{H}_2\text{O})_2\text{F}_2$ ,  $(\text{N}_2\text{H}_5)\text{Ni}(\text{N}_2\text{H}_4)_2(\text{OH})_5$ ,  $(\text{N}_2\text{H}_5)_3\text{Cr}_2\text{F}_4(\text{OH})_5 \cdot n\text{H}_2\text{O}$ ) were found as products of partial hydrolysis in these systems. Preliminary experiments were carried out with iron (III) fluoride and hydrazine, and it was found that a reduction to iron (II) took place.

The details of the preparative procedures are given in the publications listed, as are the chemical analyses of the isolated compounds. Some compounds were characterized also by thermal analysis, i.r. spectroscopy and magnetic measurements.

Hydrazinium fluorometalates, with the metal being nickel, cobalt, or copper, were isolated and characterized. In order to synthesize hydrazinium fluorocobaltate and fluoronickelate first the reaction between a water solution of hydrazinium (1+) fluoride and cobalt (2+) hydroxide or nickel (2+) hydroxide or nickel (2+) hydroxide respectively was tried. In the case of cobalt,  $(\text{N}_2\text{H}_5)_2\text{CoF}_2 \cdot \text{H}_2\text{O}$  was obtained while in the case of nickel,  $\text{NiF}_2 \cdot 2\text{N}_2\text{H}_4 \cdot 2\text{H}_2\text{O}$  was formed. Anhydrous compounds were obtained by dehydration in vacuum.  $\text{N}_2\text{H}_5\text{CoF}_3$  was synthesized by dissolving cobalt (II) fluoride in the melt of hydrazinium (1+) fluoride. In an analogous way  $3\text{NiF}_2 \cdot 2\text{N}_2\text{H}_4$  was synthesized, while the analogous cobalt compound was obtained by thermal decomposition of hydrazinium (1+) trifluorocobaltate (II).



Hydrazinium (1+) fluorocuprate was isolated by the reaction between hydrofluoric acid and copper hydrazidocarbonate hydrate. All isolated compounds were characterized by chemical analysis, x-ray powder diffraction patterns, i.r. spectroscopy, magnetic measurements and thermal analyses.

It was found that hydrazinium fluorometalates are very interesting starting materials for the reactions with xenon difluoride, or xenon hexafluoride, because the corresponding fluorides are formed during this reaction. Fluorides thus obtained are very reactive and combine to form complex compounds. For example, in the case of the reaction between iron(III) fluoride and xenon hexafluoride the starting materials were recovered unchanged while in the case of the reaction between  $\text{N}_2\text{H}_5\text{FeF}_5$  and xenon hexafluoride the complex compound  $\text{XeF}_6 \cdot \text{FeF}_3$  was formed. It was also found that during the reactions between hydrazinium fluorometalates (manganese and chromium) and xenon difluoride, the corresponding ammonium fluorometalates are formed ( $\text{NH}_4\text{MnF}_5$  and  $\text{NH}_4\text{CrF}_4$ ) instead of xenon fluorometalates ( $\text{XeF}_2 \cdot \text{MnF}_4$  and  $\text{XeF}_2 \cdot 2\text{CrF}_4$ ) which are obtained during the reactions between corresponding fluorides and xenon difluoride.

In the case of the reactions between hydrazinium fluorometalates and xenon hexafluorides the same complex compounds are formed as with corresponding fluorides if the reaction takes place. The following xenon(VI) fluorometalates were isolated during this study:

$4\text{XeF}_6 \cdot \text{TiF}_4$ ,  $\text{XeF}_6 \cdot \text{TiF}_4$ ,  $\text{XeF}_6 \cdot 2\text{TiF}_4$ ,  $4\text{XeF}_6 \cdot \text{MnF}_4$ ,  $2\text{XeF}_6 \cdot \text{MnF}_4$ ,  $\text{XeF}_6 \cdot \text{MnF}_4$ ,  $\text{XeF}_6 \cdot 2\text{MnF}_4$ ,  $\text{XeF}_6 \cdot \text{CoF}_3$ , and  $\text{XeF}_6 \cdot \text{FeF}_3$ .



This project, in addition to generating data and information on the chemistry of fluorine compounds, has also resulted in the development of methods for preparation, purification and characterization of fluorine compounds.

List of Publications that Resulted from the Project

1. P. Glavic, A. Bole and J. Slivnik, "Manganese(II) Fluoride - Hydrazine Complexes," J. Inorg. Nucl. Chem., 35, 3979 (1973).
2. P. Glavic, A. Bole and J. Slivnik, "Reactions of Zirconium, Hafnium and Cerium Tetrafluoride with Hydrazine," J. Inorg. Nucl. Chem., 37, 1316 (1975).
3. P. Glavic, J. Slivnik and A. Bole, "Cadmium(II) Fluoride - Hydrazine Complexes," J. Inorg. Nucl. Chem., 37, 345 (1975).
4. P. Glavic, J. Slivnik and A. Bole, "Reaction of Nickel(II) Fluoride with Hydrazine," J. Fluorine Chm., (in press).
5. J. Slivnik and A. Rahten, "Die Synthese und einige Eigenschaften von Hydrazinium(1+) Tetrafluoromanganat(II) und Hydrazinium(2+) Pentafluoroferrat(III). Vestn. Slov. Kem. Drustva (Bull. Slov. Chem. Soc.), 22 36 (1975).
6. J. Slivnik and A. Rahten, "Die Verbindungen von Elementen der ersten Ubergangsreihe mit Hydrazin und Fluor; Verbindungen von Kobalt und Nickel," Vestn. Slov. Kem. Drustva (Bull. Slov. Chem. Soc.), (submitted for publication June 1976).
7. B. Zemva and J. Slivnik, "On the Xenon - Fluorine Reactions," J. Inorg. Nucl. Chem. Supplement 173 (1976).
8. B. Zemva, J. Slivnik and M. Bohinc, "On the Syntheses of Xenon Fluorotitanates(IV)," J. Inorg. Nucl. Chem., 38, 73 (1976).
9. M. Bohinc, J. Grannec, J. Slivnik and B. Zemva, "On the Syntheses of Xenon Fluoromanganates(IV)," J. Inorg. Nucl. Chem., 38, 75 (1976).
10. J. Slivnik, B. Zemva, M. Bohinc, D. Hanzel, J. Grannec, P. Hagenmuller, J. Inorg. Nucl. Chem., 38, 997 (1976); "On the Syntheses and Some Properties of Xenon(VI) Fluoroferrate(III) and Fluorocobaltate(III).

Project Title: A MODULAR COMPUTER SYSTEM FOR REAL-TIME DATA REDUCTION  
AND CONTROL IN BIOMEDICAL EXPERIMENTS

Principal Investigator: Professor Zvonimir Damjanovic

Institution: Multidisciplinary Centre of Belgrade University

NBS Monitor: Mr. T. N. Pyke, Jr.

NBS Institute or Center and Division: Institute for Computer Sciences  
and Technology, Computer Systems  
Engineering Division

### Summary Description of Project Goals

The main purpose of the project is to improve interaction between the experimenter and the computer in biomedical or other laboratory experiments. The basic assumption is that this goal can be achieved by designing a modular structure, either in the hardware, the software or combined hardware-software sense, that will give the experimenter flexible command over the experimental apparatus without the need for him to become familiar with the detailed operation of the supporting computer. The investigations were carried out in four basic directions:

- a. Theoretical research in the area of formal languages with the purpose of providing a sound critical basis for the evaluation of alternatives in eventual future development of suitable problem-oriented languages.
- b. Research in hardware implementation of high-level programming languages with the purpose of providing enough elements for eventual future synthesis of computing structures.
- c. Examination of representative biomedical processes and development of suitable mathematical models with the purpose of providing enough material for the development of some basic modular programs applicable to several biomedical experiments.

- d. Practical computer applications in processing signals from biomedical experiments with the basic purpose of studying input-output devices necessary for the realisation of mutual communication in the system composed of man, computer and the biological object.

### Results and Implications to Date

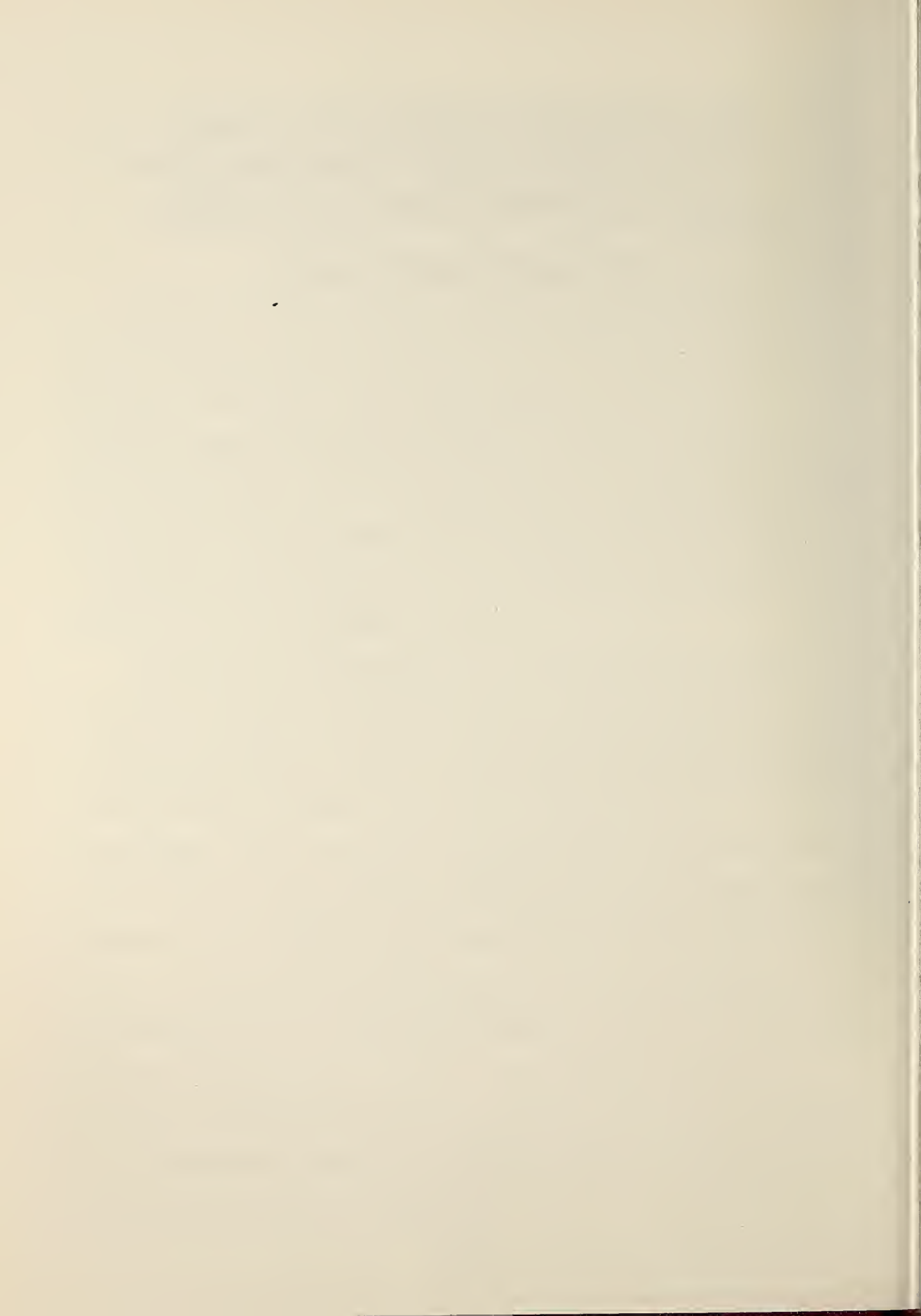
Several major steps have been completed in the development of a modular computer system for real time data reduction and control in biomedical experiments. This modular system requires that the interfaces between the human scientist or laboratory technician and the computer system be well tailored to human use and to each specific application. Special attention must be given in this man-computer relationship to the triad involving the human user, the control computer, and the experimental apparatus.

Three specific accomplishments have laid the basis for the flexible development of experiment control languages that are individually tailored to specific experimental applications. Specific results in formal language theory and in hardware/ software implementation schemes for high level programming languages have been combined with a unique approach to generating an application-specific compiler for the laboratory data acquisition and control environment. These first steps have been combined with the in-house testing of a minicomputer-based data acquisition and control system with which these techniques will be used to develop and demonstrate the project's ultimate goal--that of significantly improved control and flexibility in an experimenter's work with a laboratory control computer and with the associated experimental apparatus.

List of Publications that Resulted from the Project

1. J. Polajnar, M.A. Thesis on results in formal language theory.
2. B. Jankovic, M.A. Thesis on high level programming language structures.
3. D. Braticevic, M.A. Thesis on compiler generation.





Project Title: RADIATION DOSIMETRY AND RADIATION CHEMISTRY

Principal Investigator: Dr. I. Draganic

Institution: Boris Kidric Institute of Nuclear Sciences, Vinca

NBS Monitor: William L. McLaughlin

NBS Institute or Center and Division: Institute for Basic Standards  
Center for Radiation Research  
Applied Radiation Division

### Summary Description of Project Goals

The project concerns work on chemical dosimetry and radiation chemistry in high-intensity radiation fields. The studies are focused on the most promising measurement systems, whose practical ranges of reliable application are to be determined. This mainly comprises ranges of absorbed dose, dose rate, and also some practical conditions, e.g. in-situ and/or bulk measurements in industrial radiation processing. During the first year, suitable methods for absolute calibration were established and have subsequently been used for testing chosen chemical dosimetry systems. The later stages have been devoted to the radiation-chemical studies of processes and mechanisms of the overall "dosimetric" reaction, in order to achieve improved measurement capability and metrology standards for industrial applications.

### Results and Implications to Date

The results of this collaborative program have proven to be highly successful in attaining the initial goals of the project. In fact, the mutual benefit of achieving new, practical methods of accurate measurement for radiation processing industries goes far beyond the original scope of the program. Some of the methods

developed here, using solid and liquid forms of dye derivatives, will provide NBS with improved measurement capability for intense radiation fields. They will also be used routinely in a new Yugoslav program supported in part by the United Nations Development Program for improving industrial and medical materials by means of radiation treatment. Beyond that, the methods are expected to play an important role on an international scale and contribute to applied radiation metrology and quality control.

A breakthrough was achieved in the final stages of the work, in that the kinetics of radiolytic dye formation in leuco forms of triphenylmethane dye systems were explained for the first time, thus giving credence to the observation of the absence of rate dependence of the response of these systems in high flux density radiation fields.

The collaboration is expected to continue on a basis of mutual scientific interest.

#### List of Publications Which Resulted from the Project

1. B. Radak, P.E. Hjortenbergl and N.W. Holm, "A Calorimeter for Absolute Calibration of Thin Film Dosimeters in Electron Beams," Proceedings of the IAEA Symposium on Dosimetry in Agriculture, Industry, Biology and Medicine, pp. 311-318, IAEA, Vienna (1973).
2. I. Draganic and B.L. Gupta, "Some Current Tendencies in Chemical Dosimetry," Ibid., pp. 351-361.
3. W.L. McLaughlin, P.E. Hjortenbergl and B.B. Radak, "Problems in Absorbed Dose Measurements by Thin Films," Ibid., 557.
4. W.L. McLaughlin, M. Kosanic and I. Draganic, "Extending the Linear Gamma-Ray Response of Pararosaniline Cyanide Solutions from 0.1 to 1 Megarad," Ibid., pp. 362-366.

5. P.E. Hjortenbergs and W.L. McLaughlin, "Use of Radiochromic Dye Systems for Dosimetry," Proceedings of Regional Conference of Radiation Protection, Jerusalem, Israel, March (1973).
6. W. L. McLaughlin and M.M. Kosanic, "The Gamma-Ray Response of Pararosaniline Cyanide Dosimeter Solutions," Int. J. Appl. Radiat. Isotopes, 25, pp 249-62 (1974).
7. B. Radak, M.M. Kosanic, M.B. Sestic and W.L. McLaughlin, "A Calorimetric Approach to the Calibration of Liquid Dosimeters in High-Intensity Electron Beams," Advances in Bio-Medical Dosimetry (IAEA Symposium), paper IAEA-SM-193/70, IAEA, Vienna, pp. 633-641 (1975).
8. M.M. Kosanic, M.T. Nenadovic, B.B. Radak, V.M. Markovic and W.L. McLaughlin, "Liquid Radiochromic Dye Dosimetry for Continuous and Pulsed Radiation Fields Over a Wide Range of Energy Flux Densities," Int. J. Appl. Radiat. Isotopes, 28, pp 313-21 (1977).
9. W.L. McLaughlin, K. Schested, V.M. Markovic, M.M. Kosanic and M.T. Nenadovic, "The Kinetics of Dye Formation by Pulse Radiolysis of Leuco Cyanides of Triarylmethane Dyes in Aqueous or Organic Solution" Rad. Phys. Chem., in press (1978).
10. W. L. McLaughlin, A. Miller, M.M. Kosanic, M.M. Markovic and B.B. Radak, "Kinetics of Dye Formation by Pulse Radiolysis of Leucocyanides of Triarylmethane Dyes in Polymeric Solution" (to be published in Rad. Phys. Chem., 1978).
11. W. L. McLaughlin, "Solid-Phase Chemical Dosimeters," Sterilization by Ionizing Radiation (eds. E.R.L. Faughnan and A.J. Goudie, Multiscience Publishers Ltd., Montreal, pp. 219-252 (1974).
12. I. Draganic, "Liquid Chemical Dosimeters," Ibid, pp. 253-269.
13. W. L. McLaughlin, "Radiation Measurements and Quality Control," Proceedings of International Meeting on Radiation Processing (eds. J. Silverman and A. van Dyken), Pergamon Press, Oxford (1976). (Rad. Phys. Chem. 9, pp 147-181, 1977).
14. J. C. Humphreys, W. L. McLaughlin, B. B. Radak, A. Miller and T. A. Olejnik, "The Response of Plastic Dosimeters to Gamma Rays and Electrons at High Absorbed Dose Rates," (to be published in Rad. Phys. Chem, 1979).



15. B. B. Radak, W. M. Markovic and W. L. McLaughlin, "Commissioning Dosimetry of a Versatile Irradiation Plant at the Boris Kidric Institute of Nuclear Sciences - Vinca, Yugoslavia" (to be published in Rad. Phys. Chem, 1979).
16. V. M. Markovic, R. Eymery and H. C. Yuan, "A New Approach to Co-60 Plant Design for Introduction of Radiation Sterilization in Developing Countries," Rad. Phys. Chem. 9, pp 625-631, 1977.

## APPENDIX I

### SFCP (PL-480) BACKGROUND HISTORY

Since Fiscal Year (FY) 1961 the National Bureau of Standards has had an active Special Foreign Currency Program (SFCP) in India, Israel, and Pakistan. During 1970, Yugoslavia was added to the list of countries. Projects in Poland and the Arab Republic of Egypt became active after 1972.

The relevant US laws, such as PL-480 of the 83rd Congress pertaining to the use of SFCP funds, challenge imaginative implementation at the same time as they offer the opportunity for international collaboration of universal benefit. In the mission area of NBS and in joint research with the available countries, successive directors of NBS have called for an effective program. To this end, Mr. H. Steffen Peiser, as designated program manager, under the supervision of Dr. E. L. Brady, Associate Director for International Affairs, has received wide-ranging support, which is hereby gratefully acknowledged.

#### Stated Policy for NBS/SFCP Grants

NBS/SFCP grants provide supplementary resources that provide an appropriate means for accomplishing NBS objectives, consistent with U.S. foreign policy objectives. NBS criteria for this purpose are:

- "1. All projects must be of scientific or technological excellence.
2. All projects must be within the mainstream of NBS interests or contribute directly to NBS effectiveness.

3. All projects must benefit both the U.S. and the participating countries:
  - (a) NBS/SFCP funds must not merely replace otherwise available country funds; and
  - (b) Individuals or institutions abroad must not become primarily dependent upon NBS for long term support.
4. All projects must be followed closely by NBS staff members working in related areas. Thus, one or more NBS staff members should maintain a continuing direct relationship with the foreign person or group. This relationship should be strengthened by visits in either or both countries.
5. NBS involvement with a technical institution of another nation should be consistent with U.S. foreign policy. For example, relationships with sister institutions, staff exchanges, and visits should be carried on in the context of overall U.S. objectives in its relationships with the other country.
6. NBS staff travel to participating foreign countries should be exploited, when practical, by suitable additional visits to identify additional targets for bilateral benefit or to provide additional strength to existing relationships. Such travel should be coordinated with the NBS/SFCP Manager."

APPENDIX II  
INDEX OF INSTITUTES

Institute	Page(s)
BELGRADE	
University of Belgrade	
Institute of Physics	37,49
Multidisciplinary Center	63
LJUBLJANA	
Jozef Stefan Institute	7,15,59
VINCA	
Boris Kidric Institute	25,67
ZAGREB	
Rudjer Boskovic Institute	11,51,57
University of Zagreb	21,31,43



## APPENDIX III

## ALPHABETIC LIST OF PRINCIPAL INVESTIGATORS

Principal Investigator	University, Institute or Laboratory	Page(s)
Blinč, R.	Jozef Stefan	15
Bonefacic, A.	University of Zagreb	31
Branica, M.	Rudjer Boskovic Institute	11
Damjanovic, Z.	University of Belgrade (Multidisciplinary Center)	63
Draganic, I.	Boris Kidric	67
Konjevic, N.	University of Belgrade (Institute of Physics)	37
Kosta, L.	Jozef Stefan	7
Piljac, I.	University of Zagreb	21
Pravdic, V.	Rudjer Boskovic Institute	51
Slivnik, J.	Jozef Stefan	59
Sunjic, M.	Rudjer Boskovic Institute	57
Urosevic, V.	University of Belgrade (Institute of Physics)	49
Vujnovic, V.	University of Zagreb (Institute of Physics)	43
Zmbov, K. F.	Boris Kidric	25

APPENDIX IV  
ALPHABETIC LIST OF NBS MONITORS

NBS Monitor	Division	Page(s)
Coyle, T. D.	313.01	15
Dunn, G. H.	274.00 (Boulder)	49
Gadzuk, J. W.	232.00	57
Haller, W.	313.02	51
Hastie, J. W.	313.01	25
LaFleur, P. D.	310.00	7
McLaughlin, W. M.	243.02	67
Pyke, T. N.	650.02	63
Reed, R. P.	275.03 (Boulder)	31
Reneker, D. H.	311.03	15
Taylor, J. K.	310.00	11, 21
Wiese, W. L.	232.07	37, 43

U.S. DEPT. OF COMM. BIBLIOGRAPHIC DATA SHEET		1. PUBLICATION OR REPORT NO. NBS TN 986	2. Gov't Accession No.	3. Recipient's Accession No.
4. TITLE AND SUBTITLE  NBS SPECIAL FOREIGN CURRENCY PROGRAM IN YUGOSLAVIA 1973-1978				5. Publication Date September 1978
				6. Performing Organization Code
7. AUTHOR(S) Doris M. Bluebond, Robert S. Marvin, and H. Steffen Peiser				8. Performing Organ. Report No.
9. PERFORMING ORGANIZATION NAME AND ADDRESS  NATIONAL BUREAU OF STANDARDS DEPARTMENT OF COMMERCE WASHINGTON, D.C. 20234				10. Project/Task/Work Unit No.
				11. Contract/Grant No.
12. Sponsoring Organization Name and Complete Address (Street, City, State, ZIP)  Same as No. 9				13. Type of Report & Period Covered
				14. Sponsoring Agency Code
15. SUPPLEMENTARY NOTES  Library of congress Catalog Card Number 78-600093				
16. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here.)  An overview is given of grants awarded by the National Bureau of Standards under the Special Foreign Currency Program (SFCP) or by the U.S.-Yugoslav Joint Board. Each grant is identified by title, principal investigator, institution in Yugoslavia, NBS monitor, and the Monitor's organizational unit within NBS. The work is then described briefly under the three headings "Summary Description of Project Goals," "Results and Implications to Date," and "List of Publications that Resulted from the Project." To demonstrate the relevance of such grants to the programs of NBS, the grant descriptions are ordered by NBS institutes and divisions. The significance and purpose of the NBS/SFCP grant programs are discussed in the Foreword and the Introduction. The NBS monitors and the program manager judge this grant program to have had a high benefit to cost ratio from the viewpoint of NBS.				
17. KEY WORDS (six to twelve entries; alphabetical order; capitalize only the first letter of the first key word unless a proper name; separated by semicolons) Binational research cooperation; international scientific cooperation; physical science research administration; research planning; scientific research abstracts; Special Foreign Currency Program; Yugoslavia science and technology.				
18. AVAILABILITY  <input checked="" type="checkbox"/> Unlimited  <input type="checkbox"/> For Official Distribution. Do Not Release to NTIS  <input checked="" type="checkbox"/> Order From Sup. of Doc., U.S. Government Printing Office Washington, D.C. 20402, SD Stock No. SN003-003  <input type="checkbox"/> Order From National Technical Information Service (NTIS) Springfield, Virginia 22151		19. SECURITY CLASS (THIS REPORT)  UNCLASSIFIED		21. NO. OF PAGES 75
		20. SECURITY CLASS (THIS PAGE)  UNCLASSIFIED		22. Price  \$2.50



## PERIODICALS

**JOURNAL OF RESEARCH**—The Journal of Research of the National Bureau of Standards reports NBS research and development in those disciplines of the physical and engineering sciences in which the Bureau is active. These include physics, chemistry, engineering, mathematics, and computer sciences. Papers cover a broad range of subjects, with major emphasis on measurement methodology, and the basic technology underlying standardization. Also included from time to time are survey articles on topics closely related to the Bureau's technical and scientific programs. As a special service to subscribers each issue contains complete citations to all recent NBS publications in NBS and non-NBS media. Issued six times a year. Annual subscription: domestic \$17.00; foreign \$21.25. Single copy, \$3.00 domestic; \$3.75 foreign.

Note: The Journal was formerly published in two sections: Section A "Physics and Chemistry" and Section B "Mathematical Sciences."

### DIMENSIONS/NBS

This monthly magazine is published to inform scientists, engineers, businessmen, industry, teachers, students, and consumers of the latest advances in science and technology, with primary emphasis on the work at NBS. The magazine highlights and reviews such issues as energy research, fire protection, building technology, metric conversion, pollution abatement, health and safety, and consumer product performance. In addition, it reports the results of Bureau programs in measurement standards and techniques, properties of matter and materials, engineering standards and services, instrumentation, and automatic data processing.

Annual subscription: Domestic, \$11.00; Foreign \$13.75

## NONPERIODICALS

**Monographs**—Major contributions to the technical literature on various subjects related to the Bureau's scientific and technical activities.

**Handbooks**—Recommended codes of engineering and industrial practice (including safety codes) developed in cooperation with interested industries, professional organizations, and regulatory bodies.

**Special Publications**—Include proceedings of conferences sponsored by NBS, NBS annual reports, and other special publications appropriate to this grouping such as wall charts, pocket cards, and bibliographies.

**Applied Mathematics Series**—Mathematical tables, manuals, and studies of special interest to physicists, engineers, chemists, biologists, mathematicians, computer programmers, and others engaged in scientific and technical work.

**National Standard Reference Data Series**—Provides quantitative data on the physical and chemical properties of materials, compiled from the world's literature and critically evaluated. Developed under a world-wide program coordinated by NBS. Program under authority of National Standard Data Act (Public Law 90-396).

NOTE: At present the principal publication outlet for these data is the Journal of Physical and Chemical Reference Data (JPCRD) published quarterly for NBS by the American Chemical Society (ACS) and the American Institute of Physics (AIP). Subscriptions, reprints, and supplements available from ACS, 1155 Sixteenth St. N.W., Wash., D.C. 20056.

**Building Science Series**—Disseminates technical information developed at the Bureau on building materials, components, systems, and whole structures. The series presents research results, test methods, and performance criteria related to the structural and environmental functions and the durability and safety characteristics of building elements and systems.

**Technical Notes**—Studies or reports which are complete in themselves but restrictive in their treatment of a subject. Analogous to monographs but not so comprehensive in scope or definitive in treatment of the subject area. Often serve as a vehicle for final reports of work performed at NBS under the sponsorship of other government agencies.

**Voluntary Product Standards**—Developed under procedures published by the Department of Commerce in Part 10, Title 15, of the Code of Federal Regulations. The purpose of the standards is to establish nationally recognized requirements for products, and to provide all concerned interests with a basis for common understanding of the characteristics of the products. NBS administers this program as a supplement to the activities of the private sector standardizing organizations.

**Consumer Information Series**—Practical information, based on NBS research and experience, covering areas of interest to the consumer. Easily understandable language and illustrations provide useful background knowledge for shopping in today's technological marketplace.

Order above NBS publications from: Superintendent of Documents, Government Printing Office, Washington, D.C. 20402.

Order following NBS publications—NBSIR's and FIPS from the National Technical Information Services, Springfield, Va. 22161.

**Federal Information Processing Standards Publications (FIPS PUB)**—Publications in this series collectively constitute the Federal Information Processing Standards Register. Register serves as the official source of information in the Federal Government regarding standards issued by NBS pursuant to the Federal Property and Administrative Services Act of 1949 as amended, Public Law 89-306 (79 Stat. 1127), and as implemented by Executive Order 11717 (38 FR 12315, dated May 11, 1973) and Part 6 of Title 15 CFR (Code of Federal Regulations).

**NBS Interagency Reports (NBSIR)**—A special series of interim or final reports on work performed by NBS for outside sponsors (both government and non-government). In general, initial distribution is handled by the sponsor; public distribution is by the National Technical Information Services (Springfield, Va. 22161) in paper copy or microfiche form.

## BIBLIOGRAPHIC SUBSCRIPTION SERVICES

The following current-awareness and literature-survey bibliographies are issued periodically by the Bureau:

**Cryogenic Data Center Current Awareness Service.** A literature survey issued biweekly. Annual subscription: Domestic, \$25.00; Foreign, \$30.00.

**Liquidified Natural Gas.** A literature survey issued quarterly. Annual subscription: \$20.00.

**Superconducting Devices and Materials.** A literature survey issued quarterly. Annual subscription: \$30.00. Send subscription orders and remittances for the preceding bibliographic services to National Bureau of Standards, Cryogenic Data Center (275.02) Boulder, Colorado 80302.



**U.S. DEPARTMENT OF COMMERCE**  
**National Bureau of Standards**  
Washington, D.C. 20234

OFFICIAL BUSINESS

Penalty for Private Use, \$300

POSTAGE AND FEES PAID  
U.S. DEPARTMENT OF COMMERCE  
COM-215



SPECIAL FOURTH-CLASS RATE  
BOOK