Publications of the National Bureau of Standards*

Analytical Chemistry

A novel method for analyzing silver sediment with high precision, R. S. Davis and V. E. Bower, *J. Res. Nat. Bur. Stand. (U.S.),* **84,** No. 2, 157-160 (Mar.-Apr. 1979).

Key words: controlled potential; coulometry; electrochemical equivalent of silver; electrochemistry; Faraday; potentiostat; silver; silver analysis.

A technique has been devised which is sufficiently accurate to aid in an electrochemical determination of the Faraday constant using the silver coulometer. The technique is used to recover the silver residue which falls from the anode during operation of the silver coulometer. In contrast to previous efforts at recovery, which involved weighing of the silver residue, the method here described is to convert the silver atoms to ions and then to plate the silver onto a cathode held at constant potential with respect to a reference electrode. The current involved in the electrolysis is integrated electronically. An overall standard deviation of 5 μg is achieved for samples ranging in size from 400 μg to 1.8 mg.

18362. Durst, R. A., **Potential pitfalls in the use of ion-selective electrode-reference electrode pairs,** Paper in *Theory, Design, and Biomedical Applications of Solid State Chemical Sensors,* pp. 155-163 (CRC Press, Inc., West Palm Beach, FL, 1978).

Key words: electrodes, errors; electrodes, ion-selective; electrodes, reference; ion-selective electrodes; reference electrodes; sensors, chemical.

Ion-selective electrodes have demonstrated unique features for the measurement of a variety of ionic and gaseous species. While the advantages of these electrodes are widely known and appreciated, it is usually not until one is actually using these sensors that the problems and pitfalls become apparent and may result in serious errors and considerable aggravation. The causes of these problems are almost as varied as the sensors and the samples, but several common sources can be identified: interferences and/or fouling of the electrode, sample matrix effects, reference electrode instability, and improper calibration of the measurement system. These pitfalls are discussed in some detail with suggestions for overcoming or minimizing the problems and their associated errors.

These sensors can be a useful and convenient means for the determination and monitoring of selected species, but only if the potential pitfalls are recognized and eliminated. More than any other modern analytical technique, ion-selective electrodes require a sound knowledge of solution chemistry in order to achieve the maximum effectiveness of these sensors.

18375. Breiter, D. N., Roush, M. L., **Trace element characterization of thin polymer films by proton-induced x-ray emission spectrometry,** *Macromolecules* **10,** No. 4, 868-870 (Aug. 1977).

Key words: copper; iron; plastic; polymer; proton-induced x-ray fluorescence; thin films; trace element analysis; trace element concentrations; zinc.

Proton-induced x-ray fluorescence is a particularly well-suited technique for the simultaneous quantitative analysis of trace elements in thin polymer films. Since the sample is thin and of low atomic number, matrix effects due to proton energy loss and x-ray self absorption are small. Sensitivities are sufficient to allow for the 5-10 minute analysis of films of 1 mg/cm² areal density for iron, copper and zinc at concentration of several ng/cm².

18379. Durst R. A., Cali, J. P., **Recommended reference materials for realization of physicochemical properties,** *Pure Appl. Chem.* **50,** E. F. G. Herington, Ed., 1485-1517 (1978).

Key words: activity coefficient; hydrogen ion activity; ionic activity; ionic activity scales; pD; pD scale; pH; pH scales; potentiometric ion activity.

This report is a survey and compilation of pH and ionic activity reference materials in use throughout the world. An introductory section discusses the differences in the national pH scales and provides a brief review of the equations which define these operational scales. The major part of this report tabulates the reference materials (composition and values as a function of temperature) used to realize the pH scales presently being employed in Germany, Hungary, Japan, Poland, Rumania, USSR, UK, and the USA. Data sheets are also included for the pD and ionic activity reference materials used in the United States. This report was prepared under the auspices of the IUPAC Commission I.4 Subcommittee on Calibration and Test Materials.

Atomic and Molecular Studies

18359. Callcott, T. A., Arakawa, E. T., Ederer, D. L., **Emission and absorption x-ray edges of Li,** *Phys. Rev. B* **16,** No. 12, 5185-5192 (Dec. 15, 1977).

Key words: core hole lifetime; core hole relaxation; *K* x-ray absorption spectrum; *K* x-ray emission spectrum; many body effects; rhonon interactions.

The *K* x-ray emission edge of Li is measured between 85 and 490 K and analyzed to obtain the temperature dependences of the edge position and of a Gaussian edge-broadening function. Full width at half-maximum edge widths range from 0.22 ± 0.05 eV at 85 K to 0.38 ± 0.05 eV at 440 K. Edge positions shift from 54.81 ± 0.02 eV at 85 K to 54.86 ± 0.02 eV at 440 K and to 54.91 ± 0.02 eV after melting. Emission and absorption spectra measured with the same spectrometer establish a 0.10 ± 0.02 eV separation between the emission and absorption edges. These results are compared with recent theories describing incomplete lattice relaxation about the core hole. We conclude that for a core-hole lifetime of 0.016 to 0.030 eV the theories give a satisfactory explanation of the premature peak in the emission spectra. We suggest also that phonon-core-hole interactions provide the dominant edge-broadening mechanism, and that many-body effects are small.

18366. Younger, S. M., Wiese, W. L., **Systematic trends for the oscillator strengths of resonance transitions in the Cu and Zn isoelectronic sequences,** *Phys. Rev. A* **18,** No. 5, 2366-2368 (Nov. 1978).

Key words: Cu sequence; oscillator stength; Zn sequence.

**This listing includes recent NBS papers in the various NBS publications—defined at end of the listing—as well as NBS-authored papers in non-NBS media—identified by 5-digit numbers. Publications with prices and SD stock numbers may be purchased directly from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402 (foreign: one-fourth additional). Microfiche copies of these GPO-published documents, as well as paper copy and microfiche versions of NBS Interagency Reports (NBSIR), may be purchased from the National Technical Information Service (NTIS), Springfield, VA 22161. Reprints of papers in the NBS Journal of Research and non-NBS media, may often be obtained directly from the authors.*

New systematic trend curves for the oscillator stengths of the 4s-4p resonance transitions of Cu- and Zn-like ions have been derived from a critical analysis. This analysis takes into account recent theoretical results as well as a simulation study of cascade effects in beam-foil experiments which showed that the currently available experimental data for high ions may be too low by as much as 40-70%. The new analysis yields reliable interpolated data for many ions within the Cu and Zn isoelectronic sequences.

18369. Kaufman, V., Sugar, J., **Analysis of the spectrum of four-times-ionized lutetium (Lu v)**, *J. Opt. Soc. Am.*, **68**, No. 11, 1529-1541 (Nov. 1978).

Key words: energy levels; lutetium; spectrum; wavelengths.

Spectra of Lu obtained with a sliding spark discharge at peak currents of 50-500 A were recorded with a 10.7 m normal incidence spectrograph in the range of 500-2100 Å. Intercomparison of spectra revealed a distinct separation of Lu III, IV, and V, the first two of which have already been analyzed. The present work contains an interpretation of Lu V in which 419 lines are classified as transitions among 136 energy levels of the $4f^{13}$, $4f^{12}5d$, $4f^{12}6s$, and $4f^{12}6p$ configurations. Calculated energy levels and eigenvectors, obtained with fitted values for the radial integrals, are given.

18388. Horwitz, A. B., Leone, S. R., **Laser-excited resonant isotopic $V \rightarrow V$ energy transfer: $H^{35}Cl$-$H^{37}Cl$, $H^{79}Br$-$H^{81}Br$, $D^{35}Cl$-$D^{37}Cl$, and $D^{79}Br$-$D^{81}Br$**, *J. Chem. Phys.* **69**, No. 12, 5319-5328 (Dec. 15, 1978).

Key words: hydrogen halides; isotopes; laser; vibrational energy transfer.

An isotopically selective transverse discharge chemical laser coupled with infrared fluorescence techniques is used to measure vibration-to-vibration energy transfer rates between $v = 1$ levels of the isotopic species $H^{35}Cl \rightarrow H^{37}Cl$, $H^{79}Br \rightarrow H^{81}Br$, $D^{35}Cl \rightarrow D^{37}Cl$, and $D^{79}Br \rightarrow D^{81}Br$. The measured rate constants are $k_{HCl} = (1.91 \pm 0.04) \times 10^{-11}$, $k_{HBr} = (1.50 \pm 0.06) \times 10^{-11}$, $k_{DCl} = (1.18 \pm 0.08) \times 10^{-11}$, and $k_{DBr} = (8.34 \pm 0.17) \times 10^{-12}$ cm^3 molecule^{-1} sec^{-1}. All of the processes are nearly resonant and the rates for the hydrogenated species are on the order of one-tenth gas-kinetic. The rates for DCl and DBr are found to be 1.6 and 1.8 times slower than the corresponding hydrogen halides. Direct comparison of the results with theoretical predictions for such resonant energy transfer processes shows disagreement, indicating that further development of theoretical methods for these systems is desirable.

18389. McCann, K. J., Flannery, M. R., **Elastic scattering and rotational excitation in ion-molecule collisions. II. Li^+-H_2 and H^+-H_2 collisions**, *J. Chem. Phys.* **69**, No. 12, 5275-5287 (Dec. 15, 1978).

Key words: ion-molecule collisions; rotational excitation; semiclassical treatment.

A general semiclassical treatment of elastic scattering and of rotational excitation in ion-molecule collisions is presented. When the orbits associated with the different channels corresponding to the internal modes do not differ significantly, simplification occurs and the internal degrees of freedom can then be coupled to the relative motion via the introduction of an optical potential (which in turn depends on the transition amplitudes). Total energy is consequently conserved. An expression is derived for the inelastic scattering amplitude which acknowledges various interference effects and possible rainbow scattering. With all phase-information supressed, the procedure, when compared with the full quantum-mechanical results, reproduces the background elastic and inelastic scattering in Li^+-H_2 and in H^+-H_2 collisions. Restoration of the phases, particularly of the eikonal or action phases associated with the different classical paths that contribute to a specified scattering angle, produces the interference oscillations present in the differential cross section for scattering angles less than the rainbow angle. The method, when compared with the full quantal procedure, is remarkably efficient and accurate.

18392. Celotta, R. J., Swanson, N., Kurepa, M., **Electron scattering from ozone**, *Proc. Int. Conf. on the Physics of Electronic and Atomic Collisions, Paris, France, July 21-27, 1977*, pp. 656-657 (Commissariat a L'Energie Atomique, Paris, France, 1978).

Key words: electron scattering; energy loss; excited states; negative ions; ozone; resonances.

We present recent electron scattering measurements in ozone which (1) resolve vibrational structure in the energy loss range of 1.3-2.4 eV and (2) exhibit resonances in the incident energy range of 0.9-1.9 eV.

Building Technology

TN979. **Lead paint abatement costs: Some technical and theoretical considerations**, R. E. Chapman and J. G. Kowalski, *Nat. Bur. Stand. (U.S.), Tech. Note 979*, 153 pages (Feb. 1979) SN003-003-02022-2, $3.25.

Key words: abatement; building economics; building materials; cost estimation; econometric models; economic analysis; housing; lead-based paint; lead poisoning.

Public and private concern about the potential for lead poisoning in children due to the ingestion of lead-based paint chips has resulted in a Federally sponsored program to develop technologies by which the lead-based paint hazard may be eliminated from the nation's housing. The nature and design of the Experimental Hazard Elimination Program (EHEP) is thought to be unique in that it permitted the costs of the alternative lead paint abatement techniques to be rigorously analyzed.

The focus of this report is on the design, implementation and analysis of EHEP and the cost information it produced. Statistical analyses which permitted the development of econometric models capable of estimating abatement technique costs and expected contractor markup are described. Structural equations relating changes in the values of certain key factors to variations in direct cost and contractor markup are also presented. Guidelines, including a national deleading cost estimate, are given so that these econometric models can be used by municipal officials and building owners to estimate deleading costs as well as provide input to policy evaluation and formulation.

Computer Science and Technology

SP500-39. **Computer science & technology: Computer model documentation: A review and an approach**, S. I. Gass, *Nat. Bur. Stand. (U.S.), Spec. Publ. 500-39*, 89 pages (Feb. 1979) SN003-003-02020-6, $2.75.

Key words: computer; computer model; documentation; documentation procedures; model; model documentation.

Recent studies and surveys have concluded that, in general, the documents produced to support the understanding and use of computer models are inadequate. This paper describes the issues and concerns of computer model documentation and proposes an approach for the development of adequate documentation. First, a number of documentation studies and reports are reviewed, including software documentation guidelines and model documentation procedures. Then, based

of tests—are given here. Criteria for assessing tests are presented.

18391. Sachse, W., Golan, S., **The scattering of elastic pulses and the non-destructive evaluation of materials,** *Proc. American Society of Mechanical Engineers Winter Annual Meeting, San Francisco, CA, Dec. 10-15, 1978*, pp. 11-31 (American Society of Mechanical Engineers, New York, NY, 1979).

Key words: acoustic; crack; defects; elastic solid; pulses; scattering; ultrasonic.

This paper is a review of the results of experiments and their interpretation of the scattering of bulk ultrasonic pulses from various smooth and crack-like obstacles imbedded in an elastic solid. The scattering by circular and elliptical cylinders and multiple inclusions possessing a wide range of acoustic properties and crack-like defects of various sizes and orientations is described. For the smooth obstacles, it is shown that both arrival-time and spectral analysis of the scattered signals contain information regarding the characteristics of the scatterer and either can be used to characterize such obstacles. For the crack-like scatterers, the time-delays of various signals scattered by the crack are measured relative to reference signals from the specimen surfaces from which the crack size can be ascertained.

Fluids: Liquids, Gases and Plasmas

18367. Chang, S. J., Zapas, L. J., **Several relations for comparison of a general rate fluid and the BKZ fluid,** *Proc. VIIth Int. Congress of Rheology, Gothenburg, Sweden, Aug. 23-27, 1976*, C. Klason and J. Kubat, Eds., pp. 566-567 (Tages-Anzeiger/Regina-Druck, Zurich, Switzerland, 1976).

Key words: BKZ fluid; constitutive equation; non-linear flow histories; rate fluid; simple shear.

In the framework of a general rate fluid and a BKZ fluid the stress components for a shearing flow history can be obtained from a knowledge of the behavior in other flow histories. In some cases the two fluids are indistinguishable. To avoid specialized forms of either fluid we obtained a set of rheological relations (relations which are independent of the material properties) for two flow histories, stress relaxation after shear and suddenly applied steady shear. It is found that the relations are identical for the two fluids only in shear rates in the range of a second order fluid.

Health and Safety

TN971. **Guidelines for cost-effective lead paint abatement,** R. E. Chapman and J. G. Kowalski, *Nat. Bur. Stand. (U.S.), Tech. Note 971*, 88 pages (Jan. 1979) SN003-003-02014-1, $2.75.

Key words: abatement; building economics; building materials; economic analysis; housing; lead-based paint; lead poisoning.

Public and private concern about the potential for lead poisoning in children due to the ingestion of lead-based paint chips has resulted in a Federally sponsored program to develop technologies by which the lead-based paint hazard may be eliminated from the nation's housing. Through this program lead-based paint abatement techniques were tested in field deleading operations conducted in Boston, Massachusetts. The major focus of the program was on the collection of data on the direct costs of labor, materials and special equipment associated with these abatement techniques. Data were also collected on contractors' bids so that markup ratios could be calculated.

This report provides an overview of the statistical analysis of these direct cost data by abatement technique and building component (i.e, walls, doors and frames, windows and frames, and miscellaneous trim). An overview of the statistical analysis of the markup ratio is also included. Cost models are developed for each abatement technique which identify the key factors which affect direct cost and markup. Guidelines are given so that these models can be used by municipal officials and building owners to estimate deleading costs as well as provide input to policy evaluation and formulation.

NBSIR 78-1573. **Properties and interactions of oral structures and restorative materials,** J. M. Cassel, 94 pages (Dec. 1978). Order from NTIS as PB289913, $6.00.

Key words: accelerator; alloy; base metal; casting; composite; cyanoacrylate; dental alloy; initiator; resin; wear.

To improve the storage stability of dental composite resin systems, alternative catalyst systems to the commonly used benxoyl peroxide-tertiary amine are being developed. Increased storage stability is attainable with organic peresters or hydroperoxides but requires more efficient accelerators than the presently used amines. Ascorbic acid or ascorbyl palmitate offer promise as useful accelerators which give good biocompatibility. To develop more reactive amine accelerators with perhaps less toxicity than those currently in use, p-dimethylamino-phenylacetic acid (or ester derivatives) and dimethylaminoglutethimide were synthesized and shown to yield composite restorative specimens of good mechanical strength and color stability. A wide range of cyanoacrylates from the simplest, 2-ethyl cyanoacrylate, to the more complex type such as viscous isoamyl product have been examined for their capacity to achieve and retain bonding between acrylic resin and dentin. Very high initial bond strengths were achieved with isobutyl-2-cyanoacrylate on dentin pretreated with dilute acid but a one-month thermal cycling test in water indicated the bonding was not sufficiently stable. Initial pin on disc wear measurements with a synthetic hydroxyapatite pin have shown promise.

A pattern modification to reduce variability in the alloy castability test has been developed. Variations in the number of firing cycles and amount of condensation of green porcelain have been shown to cause significant differences in dimensional characteristics on cooling of fused dental porcelain.

18381. Treado, M. J., Diamond, J. J., Taggart, H. E., Jones, R. N., Saulsbury, L. F., Workman, J. L., **NILECJ standard for body-worn FM transmitters,** *NILECJ-STD-0214.00*, 20 pages (U.S. Department of Justice, Law Enforcement Assistance Administration, National Institute of Law Enforcement and Criminal Justice, Washington, DC, Dec. 1978).

Key words: body-worn transmitters; communications equipment; FM transmitters; law enforcement equipment; standards; surveillance; undercover equipment.

This is a voluntary national standard that establishes minimum performance requirements and methods of test for body-worn FM transmitters used by law enforcement personnel in undercover operations. The standard specifies the test equipment needed, the test methods, and the minimum performance requirements necessary for satisfactory performance.

18397. Reite, M., Edrich, J., Zimmerman, J. T., Zimmerman, J. E., **Human magnetic auditory evoked fields,** *Electroencephalogr. Clin. Neurophysiol.* 45, 114-117 (1978).

Key words: auditory evoked response; magnetoencephalography; SQUID magnetometer.

Several laboratories have demonstrated that both visual and somatosensory magnetoencephalographic (MEG) averaged

evoked fields (AEF) are more localized in their scalp distribution than are their more conventional EEG evoked potential counterparts (Brenner et al. 1975; Teyler et al. 1975; Brenner et al. 1978; Reite and Zimmerman 1978; Zimmerman). In this report, we present the first evidence of MEG auditory evoked fields. Our data indicate that they, too, are highly localized in their scalp distribution.

Instrumentation and Experimental Methods

18376. Chang, S. S., **Automated adiabatic calorimetric system for heat capacity measurement,** *Proc. 7th Symp. on Thermophysical Properties, Gaithersburg, MD, May 10-12, 1977,* pp. 75-82 (The American Society of Mechanical Engineers, New York, NY, 1977).

Key words: adiabatic calorimetry; automated calorimetry; calorimetry; heat capacity; measurement automation; p-Terphenyl; thermodynamic properties; λ-type transition.

A fully automated adiabatic calorimetric system for heat capacity measurements from 2 to 380 K is described. The system includes both automatic analog adiabatic shield control and automatic digital data acquisition and experimental control. The system incorporates a self-balancing, high resolution potentiometric system as the main instrument. The automated system is capable of making continuous, unattended calorimetric measurements over long periods of time. The precision of the heat capacity measurements is about 0.02% when the temperature increment of each measurement is greater than 1 K. For higher temperature resolutions the precision of the present system is apparently limited by an uncertainty of 0.2 mK in the estimation of the temperature increment.

18399. Boettinger, W. J., Burdette, H. E., Kuriyama, M., **X-ray magnifier,** *Rev. Sci. Instrum.* **50,** No. 1, 26-30 (Jan. 1979).

Key words: asymmetric diffraction; dynamical diffraction; microradiography; radiography; radiology; real-time radiography; resolution; x-ray image magnifier.

A method for the magnification of x-ray radiographic images is described and demonstrated. This magnifier employs two successive asymmetric diffractions of an x-ray beam from highly perfect silicon crystals. The two diffractions magnify the beam in two perpendicular directions. A device with a magnification of 25× is demonstrated for Cu K_α radiation. This device preserves and sometimes improves the resolution inherent in the radiographic technique. The x-ray magnifier is particularly useful in circumventing the relatively poor spatial resolution of electro-optical imaging systems needed for real-time observations. Basic limits on magnification and resolution using this method are described.

Lasers and Their Applications

18364. Keller, R. A., Travis, J. C., **Recent advances in analytical laser spectroscopy,** Paper in *Analytical Laser Spectroscopy,* N. Omenetto, Ed., Chapter 8, 493-538 (John Wiley & Sons, Inc., New York, NY, 1979).

Key words: atomic flame fluorescence; intracavity absorption; isotope analysis; laser analysis; laser fluorescence; laser spectroscopy; microfluorescence; saturation spectroscopy; tunable laser.

A review of new, potentially analytical applications of lasers is presented. Topics include: description of laser properties, direct absorption measurement, frequency modulation, intracavity absorption, fluorescence measurements, saturation spectroscopy, isotope analysis, CARS, flame fluorescence, condensed phase fluorimetry, energy state population distribution, and microfluorescence.

Mathematical and Statistical Methods

Non-free groups generated by two parabolic matrices, R. J. Evans, *J. Res. Nat. Bur. Stand. (U.S.),* **84,** No. 2, 179-180 (Mar.-Apr. 1979).

Key words: free groups; matrix groups; roots of unity.

In 1974, M. Newman conjectured that for any root of unity ζ, the matrix group generated by $\begin{pmatrix}\zeta^1 & 1\\ 0 & \zeta^0\end{pmatrix}$ and $\begin{pmatrix}\zeta^1 & 0\\ 1 & \zeta^0\end{pmatrix}$ is non-free. This conjecture is proved here.

Metrology: Physical Measurements

Psychrometric wet elements as a basis for precise physico-chemical measurements, R. G. Wylie, *J. Res. Nat. Bur. Stand. (U.S.),* **84,** No. 2, 161-177 (Mar.-Apr. 1979).

Key words: convective heat transfer; convective mass transfer; emissivity of water; evaporation coefficient; humidity measurement; monomolecular film; psychrometer; psychrometry; radiative heat transfer; temperature depression; water vapor measurement; wet bulb; wet element.

Under appropriate conditions, psychrometric wet elements of simple design can be highly reproducible in behavior. A temperature depression of 10 K can be reproducible from element to element within 2 mK. The properties of a wet element can be determined very accurately by direct comparisons with other wet elements in a common airstream. Comparisons with one specially developed type show the effects of practical water-retaining coverings. Comparisons with another type, which simulates the fully calculable flat-plate system, then give the behavior in absolute terms.

A cotton-yarn covering increases the psychrometer coefficient A by only 0.2 percent. The departure of the flow around a cylinder from laminar boundary-layer flow increases it by 0.7 percent.

The background and theory are outlined. The detailed behavior of a cotton-yarn covered cylinders is deduced from element comparisons; and their absolute value of A obtained as a function of diameter, airspeed, and the temperature, pressure and water content of the airstream. The dependence of A on these parameters is essentially simple. The work leads to a large increase in the accuracy of water vapor measurements and to new methods of measuring some other physico-chemical quantities.

18393. Hanson, D. W., Davis, D. D., Cateora, J. V., **Microprocessor-based satellite-controlled clock,** *Proc. of the NBS-IEEE Microcomputer-Based Instrumentation Conf., Gaithersburg, MD, June 12-13, 1978,* pp. 83-86 (Institute of Electrical and Electronics Engineers, Silver Spring, MD, 1978).

Key words: clock; GOES; microprocessors; satellite; synchronization; time.

As a demonstration of the usefulness of the NBS-sponsored time signals from two U.S. weather satellites, NBS designed and built a "Satellite-Controlled Clock." The initial design used random logic requiring nearly 80 packages of TTL logic. With the advent of microprocessors, the clock was redesigned using a four bit microprocessor and a scientific calculator chip.

Today, commercial versions of the NBS-designed clock are available and are finding use in the electric power industry, radio and television broadcasting, defense, communications, and geophysical monitoring.