MASTER

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Vacuum Ultraviolet Electronic Properties of Liquids

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Electronic Properties of Liquids

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I. Introduction

The principal aim of this program for the contract period November 1, 1968 to October 31, 1980 has been to study the electronic structure of liquids over a broad energy range, 2 to 25 eV. These studies basically consist of measuring the reflectance, trasmittance, photoionization and photoemission of liquids in the vacuum ultraviolet spectral region as a function of angle of incidence and photon energy. Such measurements are analyzed to yield the optical and dielectric functions of the liquid as functions of photon energy.

While considerable data has been accumulated on the vacuum UV electronic properties of solids, comparable data on liquids has been scarce. This group has been responsible for pioneering efforts in the field of UV liquid studies and for the development of many techniques associated with experimental research in this area. This apparently is one of the few groups with instrumentation to study optical and dielectric properties of liquids beyond 10 eV. These functions describe electron motion in liquids as stimulated by nuclear radiation, visible and ultraviolet light, and charged particles. A knowledge of the interactions of radiation with molecular liquids has direct biological application in terms of understanding radiation damage to living material, to therapy, to radiation dosimetry, to problems in cell biology, and to calculation of the actual degree of adhesive interactions between biological cells and between cells and artifical material. Applications are made to radiation chemistry, upper atmospheric physics, and aerosol formation.

The UV and VUV spectra of organic molecules is valuable in the characterization of atmospheric emissions. These spectra complement analysis by other techniques, including spectrometry, fluorescence, column chromatography and IR-VIS spectroscopy. The UV-VUV is the region of maximum absorption for organic molecules. The absorption spectra of different compounds yield different energies and magnitude of absorption. As such, these spectra are more sensitive for compound identification than visible or IR.

Several methods have been developed for studying liquid samples. In the transmission[1] method a cell is formed of thick planar slabs separated by as little as 500 Å of liquid. The method cannot be used beyond ≈ 10 eV photon energy, the limit of slab transparency. A second closed cell technique is the semicylinder[2] method. The liquid sample is placed in contact with the flat side of the semicylinder. Light is incident normally on the curved surface, is reflected specularly at the cylinder-liquid interface, and exits normally through the semicylindrical surface again. This method, using semicylinders of magnesium fluoride, calcium fluoride, quartz and sapphire, features the possibility of employing reflection at angles around the critical angle, and of avoiding optical absorption in the vapor. As with the transmission method it is limited to the region below ≈ 10 eV. In the open-dish[3] method, light reflected from the surface of a liquid is measured while the liquid is in equilibrium with its vapor. Absolute reflectance values of the liquid are determined from normalization to a reflecting surface of known optical properties. Although the

the partial absorption of the light by the vapor is a problem, the open-dish method is the only one capable of being used at energies beyond 10 eV. A more recently developed open-dish method is the ionization[4] method. The ionization in the vapor adjacent to the liquid is used to monitor the photon beam strength before and after reflection. The ratio of ion currents is related to the reflectance.

The transmission method yields the optical absorption directly. Examination of the absorption spectrum of a liquid over an extended energy range yields the energies of single-electron excitations. The optical constants at each incident photon energy may be obtained from measurements of reflectance as a function of photon angle of incidence by a least-squares fit to Fresnel's equations. From n and k, the real and imaginary parts, respectively, of the complex index of refraction, $\tilde{n}$, one can calculate the real and imaginary parts ε_1 and ε_2, respectively, of the complex dielectric function ε, where $\varepsilon = \tilde{n}^2$, and the energy loss function, $-\mathrm{Im}\ 1/\varepsilon$. This calculated energy loss function can be compared with the characteristic electron-energy-loss spectrum for the liquid. Thus, optical measurements in the vacuum UV give information similar to that obtained by measuring the distribution of energy losses experienced by a fast electron in going through a thin layer of matter.

Supplementing techniques for studying liquids through measurement of reflectance and transmission is the photoemission method. Electrons ejected from the liquid by UV photons are measured. In photoelectron studies it is possible to explore the energy region containing plasma excitations to investigate the dependency of electron emission on this important process. From photoyield data it is also possible to calculate the photon and photoelectron mean free paths.

II. Progress

At the time of initiation of this contract, techniques had been developed for measuring the UV electronic structure of condensed materials including metals, semiconductors and insulators. The possibility of utilizing such techniques in the study of the electronic properties of liquids was first examined by this group during the period 1966-1968 wherein we were able to determine the dielectric response of pure water up to 10 eV.[5]

Two interpretations of the data were presented, one in which the structure in the optical constants was compared with the known absorption characteristics of water vapor, i.e. water is considered to be a condensed gas with the condensation changing only slightly the electronic structure of the molecules. In the other interpretation the liquid water is likened to an electronic insulator such as aluminum oxide. Initial studies of the dielectric properties of liquid water were limited to wavelengths greater than 1050 Å due to the onset of ionization in the vapor at this wavelength — with a consequent increase in the attenuation of light in the vicinity of the liquid sample.

One of the purposes of the present contract was to extend the observations of liquid water to higher energy (shorter wavelength) to see if there were collective effects present. Such an effect was considered to represent an absorption of energy by many electrons operating collectively in the condensed state (the "plasmon" response). To extend the measurements on liquid water to higher energy require the design, construction and assembly of new reflectance equipment consisting of a movable monochromator, sample chamber, and associated equipment. The new cell and Seya-monochromator embodies several improvements over the apparatus used in the initial studies. It has a shorter path in the vapor thus reducing absorption in the vapor. Rather than being limited to three values, as with the initial apparatus, the angle of incidence may be varied continuously between 15 and 70 degrees thus enabling more precise values of absorption and index of refraction to be obtained. Using the new reflectometer it was possible to extend the studies on water to 14.6 eV. However, no pronounced structure was found in the optical constants near 12.5 eV as had been speculated based on studies of ionization in water vapor near the ionization threshold. Considerable effort was expended in carefully aligning the reflectometer so that the optical properties of the liquid were accurately represented by the measured reflectance. The system had to be stabilized to minimize vibrations in the liquid and an auxiliary cooling system incorporated to control the temperature and thereby maintain the system in thermodynamic equilibrium. A description of the construction and alignment of the reflectometer appeared in Review of Scientific Instruments.[6]

Refinements were made in the open-dish reflectance technique which permitted more accurate measurements than those obtained in the earlier years of this contract. A refrigerator circulator was added for more adequate control of the temperature within the chamber. Modifications were made in the light pipe collecting radiation reflected from the liquid or gold reference surface so as to minimize detection of non-specularly reflected light. A gold replica grating was employed in preference to the previously used platinized grating so as to increase the ratio of the s-polarized to p-polarized light and to reduce grating changes due to oxidation by vapors pumped into the monochromator from the sample chamber. A modification was made to the McPherson-Hinteregger-type capillary discharge lamp which converted it from a cold cathode discharge to a hot filament discharge. With the approximately three-fold intensity increase and improved stability it was possible to extend reflectance measurements on water to 25.6 eV.

Optical and dielectric data were obtained from these measurements. A prominent peak in the energy loss function at about 21.2 eV was interpreted as a collective electronic oscillation. This corresponds closely to the value of 21 eV calculated by Platzman[7] for the plasma frequency in water on the assumption that the electrons are not bound in atoms. This loss was acknowledged to represent an extremely important energy absorption mechanism in water. The results on water, including identification of the first reported collective effect in a liquid were reported by Heller, et al.[8]

A second reflectance chamber was constructed for mounting to the exit arm of the Seya monochromator. Though initially designed to measure the optical properties of the gold films used as a reference in the open dish liquid studies the chamber was also used to study the optical properties of solid films and of liquids in the region below 10 eV using closed cell techniques. The paper, "Automatic Concentric Shaft Reflectometer for Use in the Vacuum UV"[9] concerns the automation of processes on the data acquisition from the angular reflectometer.

For one closed cell technique, the semicylinder method, a cell was constructed which contains the liquid sample in contact with the flat side of a quartz, calcium fluoride, lithium fluoride or sapphire semicylinder. A cell was also constructed to hold thick planar slabs for transmission measurements. The spacing between plates which determines liquid sample thickness can be as narrow as 500 Å. The thickness of cell spacing is determined by interference measurements. A triple reflection polarizer was constructed for use in measuring the grating polarization. A vacuum evaporator was purchased for preparing gold reference films and thin films of organic materials. It was intended that films of materials to be used as solutes would be studied so that properties of solute and solvent would be available for interpretation of solution data.

In addition to water, a number of liquids have been studied using these techniques. Reflectance measurements were made on Dow Corning 704 and 705[10] silicone pump oils, tetramethyletetraphenyltrisiloxane and trimethylpentaphenyltrisiloxane, respectively. The optical properties of vacuum pump oils are of particular interest since they are always present to a certain extent as contaminants in chambers evacuated by oil diffusion pumps. Pumping fluids may condense in vacuum monochromators, in space craft simulation chambers, on spacecraft instrumentation, in evacuated cathode ray tubes, or atomic particle accelerators. They may be present as contaminants on films prepared by deposition for optical devices or electronic circuits. The polarization of gratings used in vacuum monochromators is dependent on surface contamination as is the change in reflectance of optical films. The optical and dielectric properties of silicone oils were studied not only to determine the effects of these surface contaminants but most important to see if the optical properties could be understood in terms of the response of the individual molecular groups, i.e. methyl, phenyl, and the trisiloxane backbone.

Studies were made from 2.6 to 22.4 eV on liquid glycerol, $C_3H_4(OH)_3$.[11] The study of glycerol was recommended by Dr. D. Dupourque of The University of Tennessee Medical Hospital in view of its important role as an essential ingredient of animal and vegetable oils. It has been shown that the presence of glycerol in a number of biological systems gives considerable protection against the effects of ionizing radiation. A knowledge of the electronic properties of glycerol may be important in understanding the mechanisms responsible for the radioprotective capacity of glycerol.

Measurements were made on two hydrocarbons, normal tetradecane, $C_{14}H_{30}$, and normal heptadecane, $C_{17}H_{21}$. Structure in these σ electron systems was compared to lower order alkanes from methane to octadecane for systematic shift and intensity changes in absorption peaks. The data was reported in "Collective Oscillation in Liquid Normal Tetradecane and Normal Heptadecane."[12]

Absorption measurements were made on aqueous solutions of DNA in the region from 2000 to 1220 Å. The absorption spectrum was basically the same as that observed for water, indicating an inability to separate the DNA contribution from that of the strongly absorbing solvent.

Attention was shifted to transmission measurements on dry films of DNA on CaF_2 substrate. Results appeared in "Optical and Dielectric Properties of DNA in the Extreme Ultraviolet."[13]

New techniques were explored in order that measurements could be made on solutions. Studies of solutions are tremendously important to any theoretical treatment of the attractive forces between cells in biological systems since substances added to water (or any organic solvent) change the index of refraction of the solvent and consequently the dispersion forces. Studies were made on bovine serum albumin in .9% NaCl irrigating solution on 2-chloroethanol in collaboration with Dr. S. Nir, Roswell Park Institute of Theoretical Biology, Buffalo, N.Y. The dielectric properties of albumin are of value to Dr. Nir in the calculation of dispersion forces between cells in vivo. The completed work appeared in print: "Dispersion Equation and Polarizability of Bovine Serum Albumin from Measurements of Refractive Indices."[14] Similar studies on solutions of organic material have been reported in "Optical Constants and Dispersion Equations of Lecithin, Cholesterol, Fucose and Chloroform; Measurements in Vacuum-Ultraviolet to Visible Wavelength Regions."[15]

Optical and dielectric functions of liquid H_2O and D_2O were obtained in the 2-9.3 eV spectral region. The principal difference observed was the contraction of the D_2O spectra due to the mass effect of the heavier isotope. This work appeared in publication: "Isotopic Effects on the Electronic Properties of H_2O and D_2O in the Vacuum UV."[16]

During the last decade members of this group have measured the optical and dielectric properties of over a dozen liquids; all have shown a strong energy absorption at ∿ 21 eV, attributed to excitation of a collective electronic excitation. The experimental spectra for several of these molecular liquids in the energy region from 0 to 26 eV are analyzed for the degree of collective behavior exhibited in a Robert Platzman memorial paper.[17]

A major thrust of the last several years has been the modification of the existing open-dish reflectance chamber. Beyond 10 eV, the cutoff for windows in the vacuum ultraviolet, reflectance measurements on liquids must be made at the free surface of the liquid in equilibrium with its vapor. The difficulties of making reflectance measurements to high accuracy (less than 1% uncertainty) are exacerbated for liquid surfaces because of the presence of the vapor in the optical path. In an effort to improve the accuracy of the reflectance measurements and hence the derived dielectric functions, a technique was devised whereby the ionization of the vapor along the optical path both before and after reflection at the liquid is measured with a double ion chamber. The instantaneous ratio of these ion currents is the required reflection. The ion chamber reflectometer is described in the article "Measurement of Reflectance by Gas Ionization."[18]

A new instrument was developed to measure the ratio of currents in the 10^{-13} ampere range or larger. The electrometer-ratiometer, used to simultaneously measure the currents in the incident and reflected ion chamber is described in "A Simple Electrometer-Ratiometer of High

Sensitivity and Precision."[19] Studies using this technique have been completed on glycerol[20], formamide[21] and hexamethylphosphoric triamide[22] with an order-of-magnitude improvement in accuracy over data taken previously.

Our apparatus for measuring reflectance of liquids in the vacuum UV was modified to permit absolute photoemission measurements. Considerable time has been devoted to overcoming formidable problems associated with the photoemission experiment. A new sample cup was required to support a larger volume of liquid above the cup edge, and to allow the ground support to be hidden from exposure to spilled liquid so no short can occur. Problems associated with electrical and vacuum leakage of BNC feedthroughs and with sweating of feedthroughs resulting in shorting of the electrometers had to be solved. Questions concerning appropriate sample cup potential had to be resolved. The theory of operation of the double ionization chamber when used for photoemission measurements has been incorporated in a paper submitted to The Physical Review on photoemission from liquid formamide.[23] Photon fluxes are determined by filling the dual ion chambers with a filling gas such as argon or benzene and connecting an electrometer to the properly biased sample cup to read electron currents leaving the cup. After ion drift into the cup of the filling gas is taken into account, the number of photo-electrons per incident photon and per absorbed photon are obtained. Photoemission measurements have been made from liquid formamide[23] and hexamethylphosphoric triamide.[24] A model for photoemission has been proposed whereby the photon and photoelectron mean free paths in liquids can be determined from the yield data.

As a common effort to work on liquid insulators studies have been made on solid insulators. Techniques were developed for the preparation of dry films of organic material including DNA,[13] chlorophyll and lecithin. The absorption spectrum of solutions of these materials were in many cases not appreciably different from that observed for the solvent, indicating an inability to separate the solute contribution from that of the strongly absorbing solvent. For this reason, attention was shifted to transmission measurements on dry films of the organic solute on an appropriate substrate to supplement solution data.

These techniques were applied to the study of polycrystalline anthracene in the 3.2-9.3 eV spectral region.[25] This information is relevant, for example, to studies on particulate matter containing anthracene from by-products of coal-combustion or conversion. Also, when used as phosphors, films of anthracene prepared by vacuum deposition are polycrystalline in structure.

Studies were completed on two polymers, polyethylene[26] and Kapton Type H polyimide film (manufactured by E.I. duPont de Nemours and Co.).[27] Results on the optical properties of polyethylene films, monomeric unit $(CH_2 \cdot CH_2)$, were determined by transmission and reflectance measurements between 0.7 and 82 eV. The results for the extinction coefficient were subsequently combined with calculated values in the soft and hard x-ray regions and both internal consistency and their consistency with data obtained previously for polystyrene, verified through sum rule calculation. Based on these optical data, calculations were made of the mean excitation energy for polyethylene, and of the inverse mean free paths and energy losses per unit path length for low-energy electrons.

Kapton films, with monomeric unit $-(C_{22}H_{10}N_2O_5)-$ are used extensively in situations demanding radiation resistance. The optical parameters of these polyimide films were determined from a Kramers-Kronig analysis of measured reflectance values from 25,000 Å to 163 Å. These data were used to calculate the dielectric functions, mean excitation energy of Kapton, electron mean free paths and stopping power of Kapton for electrons. A description of the Kapton data is appended in the extended abstract from the Basic Optical Properties of Materials Conference.[27]

Appended to this report is a listing of publications and papers of work performed during the contract period Nov. 1, 1968 to Oct. 31, 1980 with the relevant reprints and preprints.

III. Projected Studies

During the next three years additional work is proposed for initiation or completion.

(1) Transmission Grating Soft X-ray Monochromator: The major thrust of the next few years will be to design, construct and make operational a transmission grating soft x-ray monochromator and an associated exposure chamber. At present, our experiments are conducted using a reflection grating in a monochromator of the

Seya-Namioka geometry. Beyond 25 eV the grating efficiency is too low for measurements. This is a consequence of the low reflectance of solids above 25 eV. Our photoemission data on formamide and hexamethylphosphoric triamide indicate strong plasmon effects below 25 eV. The aluminum data of Sampson and Cairni[28] indicate multiple plasmon effects above 25 eV. Presumably, multiple plasmon effects are present in the liquid insulators. The transmission grating soft x-ray monochromator system will be used to extend photoemission measurements beyond 25 eV to investigate collective effects.

A description of a new far UV transmission grating monochromator has been reported by Flodstrom and Bachrach[29] and by Källne, et al.[30] Caldwell and Arakawa reported at the VI VUV Conference on a new method of making transmission gratings for the high energy region (to 50 eV). These are made in photoresist exposed in the beam of a scanning electron microscope. An electron microscope at ORNL, accessible to our group, is currently being fitted with a digital scan generator and PDP/1123 computer to custom manufacture any surface, hole(s), slit or grating to specifications. Transmission gratings for use in the proposed monochromator will be made using the facilities at ORNL. It is anticipated that photoemission measurements will be extended on those liquids for which data exists below 25 eV.

(2) Organic Solvents: Several low vapor pressure organic solvents are proposed for study including triethylphosphate N,N-dimethylacetamide, and N-methylformamide. The latter, N-methylformamide, $HOCNH(CH_3)$, is

a generically related liquid to the previously studied formamide. This liquid is distinguished by having an extremely high static dielectric constant, 182.4,surpassing even that of formamide, 110.0.

The low vapor pressure of these liquids makes them amenable to open dish techniques and attributed to their selection for study. Liquids having high vapor pressure are difficult to maintain under near-vacuum conditions. The reduction in intensity with increasing vapor pressure makes measurements prohibitive for high vapor pressure liquids. Other factors important in determining whether or not a liquid is accessible to open dish studies includes the electrical conductivity, surface tension, viscosity, toxicity, and corrosiveness of the liquid.

Closed cell and photoionization measurements will be made on these liquids in the energy region from 0 to 25 eV. From optical properties, the electron mean free path and stopping power will be calculated for electrons of energy 10^1 to 10^4 eV. It will be possible for some liquids to combine absorption spectrum determined from transmission and/or reflectance measurements with estimated values of k at shorter wavelengths to form a spectrum extending over the oscillator strenght of the valence electrons. As a consistency check on the data, one can perform a "k-sum rule" on these data to yield the total number of valence electrons per molecule.

It is anticipated that photoemission measurements will be made in the $\sim$ 16 to 50 eV region. Photon and photoelectron mean free paths in liquids will be determined from yield data. The yield curves will also be analyzed for multiple plasmon effects.

(3) Polycyclic Organic Molecules: Studies have been initiated on the polycyclic aromatic hydrocarbons benzene, toluene (methybenzene), xylene (diemthylbenzene) and trimethylbenzene. Concurrent with these liquid studies measurements will be initiated on the simple ring structures phenanthrene (an isomer of the previously studied anthracene molecule), pyrene (benzo [d,e,f] phenanthrene), picene and chrysene. These have been identified as important polycyclic aromatic hydrocarbons in coal conversion processes and as pollutants of industrial gasifiers. Studies on these liquids will be limited to closed cell techniques. The high vapor pressures of the liquids make them inaccessible to open dish studies.

(4) Liquid Semiconductors: As a common effort to the existing work on liquid insulators attention will be given to the study of liquid semiconductors and to the identification of these materials that would lend themselves to open dish measurements.

It is hoped that application of these basic physical techniques to the study of pure liquids and solutions will bring about a thorough and complete understanding of electronic energy levels in liquids which are excited by the passage of ionizing radiation.

IV. Personnel

During the contract period three students completed research for the Ph.D. dissertation. George D. Kerr and James M. Heller, Jr. were students of L.R. Painter. Martha Anderson was a student of Dr. S. Nir of the Roswell Park Institute of Theoretical Biology, Buffalo, N.Y. The experimental aspects of her thesis were done under L.R. Painter at The University of Tennessee. A fourth student, Jaspal Attrey is currently working on research for her Ph.D. dissertation. James M. Heller, Jr. completed a 2 year post-doctoral tenure upon completion of his Ph.D. degree. A number of other students have been involved with the program on a short-term basis, e.g. as summer participants.

This project, begun in 1968, was an outgrowth of optical studies carried out in the Biological and Radiation Physics Section in the Health and Safety Research Division at Oak Ridge National Laboratory. The program has continued to have access to the extensive ultraviolet facilities and to the five senior experimental physicists who have been working in the vacuum ultraviolet area for 15 or more years. The ORNL group is backed up by seven theorists of whom three are in molecular physics and four are concerned principally with solid state activities. The research proposed here falls naturally between the molecular and solid state fields and will look to research personnel at ORNL for both experimental and theoretical support.

The Physics Department is well equipped with regard to machine shops and electronic shops. Competent technical assistance, available to the project, is furnished by the Department in connection with these facilities.

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