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FINAL TECHNICAL REPORT ON

"A PHOTOIONIZATION STUDY OF CLUSTER SPECIES IN A SUPERSONIC MOLECULAR BEAM"

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August 26, 1982

Final Report for Period May 1, 1975 to August 26, 1982

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) This report summarizes the research designed to explore the heats of formation of van der Waals and hydrogen bonded clusters which are present at low pressures in the nozzle chamber of a supersonic molecular beam. Data are reported on equilibria, ionization, cross sections, and fragmentation for monomers and clusters of CO ₂ , alcohols, ammonia, acetic acid, nitriles and water.		

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A. A SUMMARY OF THE FINAL REPORT ENTITLED "A PHOTOIONIZATION STUDY OF CLUSTER SPECIES IN A SUPERSONIC MOLECULAR BEAM"

The work summarized here is an effort which began with an attempt at characterizing those cluster species which were already present in the nozzle chamber of a supersonic molecular beam as contrasted with those species which formed as a result of condensation in the beam process. Primarily the approach has been to employ mass spectrometry in conjunction with photoionization produced by dispersed synchrotron radiation or electron impact to monitor the changes in intensities produced by a variation in the equilibrium vapor in the nozzle chamber. These variations can be produced by precise nozzle temperature changes, and the heats of formation of the cluster species can be derived by application of the van't Hoff relationship. The measured heats of formation are given in the listed published papers for carbon dioxide, methanol, ethanol, trifluoroethanol, acetic acid, and ammonia. Recent work in the just-completed PhD dissertation of Susan Johanningsmeier Olesik describes the efforts with water, acetonitrile, propionitrile, and compares the effects of fragmentation of the gas phase cluster species with the fragmentation from solid surfaces. (Completion of this PhD dissertation is the reason for the delay in this final report. The abstract lists data which are not yet published.)

Work is also described in the use of a supersonic molecular beam for analytical sampling of gases by mass spectrometry. In conjunction with this work was a large effort directed toward measurement of absolute ionization cross sections with laboratory line sources which could be used to calibrate a mass spectrometer.

The effort initiated by the Office of Naval Research is continuing under sponsorship of the National Science Foundation.

B. PUBLICATIONS FROM THIS RESEARCH

1. Taylor, J. W. and Jones, G. G. "Photoionization Mass Spectrometry of Methanol Clusters Using Supersonic Molecular Beam Sampling and Synchrotron Radiation", in Proc. 5th Intl. Symp. on Molecular Beams, (Ed.) F. M. Devienne, Laboratoire de Physique Moléculaire des Hautes Energies, Peymeinade, France, E₂ (1975).
2. Taylor, J. W. and Jones, G. G. "Photoionization Mass Spectrometry of Alcohols, Alcohol Clusters, and Carbon Dioxide Dimers", J. Photochem. 5, 195 (1976).
3. National Academy of Sciences Report, "An Assessment of the National Need for Facilities Dedicated to the Production of Synchrotron Radiation", Solid State Sciences Committee, National Research Council, Washington, DC, 1976 (JWT was a member of the panel which prepared the report).
4. Jones, G. G. and Taylor, J. W. "A Photoionization Study of Carbon Dioxide Dimers in a Supersonic Molecular Beam", J. Chem. Phys. 68, 1768 (1978).
5. Wood, K. V., Grange, A. H., Taylor, J. W. "Mass Discrimination Effects in a Quadrupole Mass Spectrometer", Anal. Chem. 50, 1652 (1978).

6. Cook, K. D. and Taylor, J. W. "Note Concerning the Dissociation Energy of the Hydrogen-Oxygen Bond in Acetic Acid", *Inter. J. Mass Spectrom. Ion Phys.* 30, 93-95 (1979).
7. Cook, K. D. and Taylor, J. W. "A Supersonic Molecular Beam Mass Spectrometric Study of Hydrogen Bonding in Ammonia", *Inter. J. Mass Spectrom. Ion Phys.* 30, 345-357 (1979).
8. Wood, Karl V. and Taylor, J. W. "A Photoionization Mass Spectrometric Study of Autoionization in Ethylene and *trans*-2-Butene", *Inter. J. Mass Spectrom. Ion Phys.* 30, 307-318 (1979).
9. Cook, K. D. and Taylor, J. W. "A Mass Spectrometric Study of the Effect of Supersonic Molecular Beam Sampling on the Clustering of Acetic Acid Vapor", *Inter. J. Mass Spectrom. Ion Phys.* 35, 259-271 (1980).
10. Cook, Kelsey D., Jones, Gilbert G. and Taylor, James W. "A Photoionization Study of Hydrogen-Bound Clusters in a Supersonic Molecular Beam", *Inter. J. Mass Spectrom. Ion Phys.* 35, 273-292 (1980).
11. Taylor, J. W., Cook, K. D. and Johanningsmeier, S. V. "Photoionization Mass Spectrometry of Hydrogen Bound Clusters", VI International Conference on Vacuum Ultraviolet Radiation Physics, Charlottesville, VA, June 2-6, 1980, Paper II-91.

C. COMPLETED PAPERS UNDER CONSIDERATION BY JOURNALS

1. A. H. Grange and J. W. Taylor, "Consideration of Design of Photoionization Chambers for Cross Section Measurements". The abstract follows:

"Design parameters of photoionization chambers which influence the accuracy of cross section measurements are considered. Among these parameters are the interelectrode spacing, the nature and construction of the end plates, pressure and temperature effects, and the effective guard plate lengths. A three-cathode plate system was developed to test for deviation from assumed Beer's law behavior and to test designs which could provide cross section accuracies in the range $\pm 2\%$ for spectral regions without sharp spectral structures."

2. K. V. Wood and J. W. Taylor, "Skimmer Induced Effects on Analytical Sampling by a Supersonic Molecular Beam". The abstract follows:

"High pressure (20-100 torr) gas sampling with a supersonic molecular beam is studied for binary mixtures by mass spectrometry as a function of the nozzle-skimmer distance. It was found that at the minimum in the plot of intensity *versus* nozzle-skimmer separation the heavier species is enriched relative to the light species by the square root of the molecular weight ratio. Mass separation is attributed to the interaction of the molecules in the expanding jet with molecules reflected off the skimmer and is analyzed in terms of a diffusion-like separation process."

3. T. Ibuki and J. W. Taylor, "Photoabsorption Cross Sections of CO_2 , O_2 , C_2H_4 , C_3H_6 and $i\text{-C}_4\text{H}_8$ Between 175 and 780 Å Using a Triple Cathode Plate Ionization Chamber". The abstract follows:

"The total photoabsorption cross sections have been measured for CO_2 , O_2 , C_2H_4 , C_3H_6 , and $i\text{-C}_4\text{H}_8$ over the wavelength range of 175-780 Å using a three cathode plate and dispersed synchrotron radiation. The accuracies of the cross sections obtained are in the range $\pm 2\%$ where there is no sharp absorption and where the photon source is monochromatic. The discrete structure observed for CO_2 and O_2 are consistent with the Rydberg series reported previously. C_2H_4 , C_3H_6 and $i\text{-C}_4\text{H}_8$ show monotonical increasing cross sections with increasing wavelengths from 175 to 700 Å."

D. COMPLETED PhD DISSERTATIONS UNDER SPONSORSHIP OF THIS CONTRACT

1. September 1975; Gilbert G. Jones, NSF Fellow, PhD, University of Wisconsin. "A Photoionization Mass Spectrometric Study of Cluster Species in a Supersonic Molecular Beam: Carbon Dioxide and Methanol". (Presently Research Scientist, American Oil Company, Naperville, IL.)
2. July 1978; Kelsey D. Cook, WARF Fellow, PhD, University of Wisconsin. "A Photoionization Study of Hydrogen Bound Clusters in a Supersonic Molecular Beam". (Presently Assistant Professor, University of Illinois, Urbana, IL.)
3. July 1978; Karl V. Wood, PhD, University of Wisconsin. "Part I: Mass Discrimination in a Mass Spectrometer System and a Supersonic Molecular Beam Sampling System. Part II: Excited States in the Ionization Process of Alkyl Olefins and 2-Pentanone". (Presently Group Leader, Fuels Analysis Laboratory, Purdue University, West Lafayette, IN.)
4. August 1982; Susan V. (Johanningsmeier) Olesik, PhD, University of Wisconsin. "A Study of Intermolecular Bonding Using Supersonic Molecular Beam Mass Spectrometry". The abstract of the dissertation follows because the oral exam was held August 26, 1982:

"A supersonic molecular beam mass spectrometer was used to study hydrogen and dipole-dipole bonding. The ionizing radiation used was either wavelength dispersed vacuum ultraviolet radiation produced by an electron storage ring or electrons produced by a heated rhenium filament. The focusing of the mass spectrometer ion optics was optimized using a two-level factorial design.

"The autoassociation of acetonitrile and propionitrile was studied. Ions corresponding to the series $(\text{RCN})_n\text{H}^+$ were attributed to fragmentation of the unique neutral clusters present in the molecular beam. The enthalpies of cluster formation were determined by applying the van't Hoff relation to the temperature dependence of the ion intensities. Estimated dimerization energies for acetonitrile and propionitrile were -8.9 and -9.5 kcal/mole, respectively. A lower limit of -9.0 kcal/mole was determined for the enthalpy of formation of acetonitrile trimer.

The energetics of water cluster formation were also studied. Ions corresponding to the series $(\text{H}_2\text{O})_n\text{H}^+$ (with $n=1$ to 5) were observed. The photoionization efficiency curves were measured for each of these ions and the ionization and appearance potentials were 12.6 eV, 11.7 eV, and 10.4 eV for H_2O^+ , $(\text{H}_2\text{O})\text{H}^+$, and $(\text{H}_2\text{O})_2\text{H}^+$, respectively. Step structure was observed in the photoionization efficiency curves of the large oligomer ions (trimer, tetramer, pentamer) at energies corresponding to the small oligomer ions (monomer, dimer, trimer). Fragmentation of the larger clusters was observed at energies lower than the ionization potential of the monomer. The enthalpies of water cluster formation were determined at low source pressure to minimize the contribution of large cluster fragmentation. The measured enthalpies of formation were -3.18, -3.12, -3.06, -2.68 kcal/mole for dimer, trimer, tetramer, and pentamer, respectively.

The fragmentation pathways of hydrogen bonded clusters of methanol, ammonia, and water were also studied. The relative intensities of the monomer fragment ions were monitored as a function of pressure. Increases in the relative ion intensities of the monomer fragment ions with pressure were attributed to fragmentation of neutral clusters. The observed fragmentation processes indicated that the cluster fragment ions possess considerable internal energy. The degree of cluster fragmentation was found to decrease in the order of methanol > ammonia > water.

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