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DYNAMICS OF LARGE MOLECULES AND MOLECULAR CLUSTERS(U)
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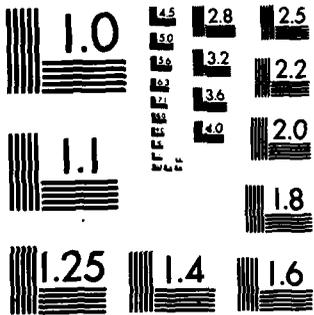
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DYNAMICS OF LARGE MOLECULES AND MOLECULAR CLUSTERS

Final Technical Report

by

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6. PROGRESS IN TECHNICAL APPROACH

New techniques for spectroscopy in supersonic expansions have been developed since the initiation of the research program. *include:*

6.A The Construction of a Pulsed, Planar, Supersonic, Slit Nozzle; was successfully accomplished. We developed a 12 Hz, 150 μ sec duration pulsed supersonic jet source based on expansion of a seeded gas through a 35 mm nozzle. The characteristics of this planar supersonic source are:

- (1) A long optical pathlength.
- (2) High densities of ultracold seeded molecules at the interrogation distance.

6.B Absorption Spectroscopy in Supersonic Expansions; The pulsed slit nozzle was utilized for absorption studies of ultracold large molecules.

6.C Determination of Fluorescence Quantum Yields in Jets; The simultaneous determination of absorption spectra and fluorescence excitation spectra enabled us to determine the fluorescence quantum yields from photoselected states of large molecules.

6.D "Laser-Free" Spectroscopy of Large Molecules in Planar, Supersonic Jets; Absorption spectra, fluorescence excitation spectra and fluorescence quantum yields from photoselected vibrational states of the S_1 manifold of large molecules cooled in planar, supersonic jets were determined using a pulsed xenon lamp and a monochromator. We have constructed a pulsed linear nozzle spectrometer for absorption and fluorescence excitation in jets. The use of a xenon lamp

6.D (cont'd)

as a light source made it possible to record the lamp-induced fluorescence spectrum simultaneously with the absorption spectrum. The spectroscopic resolution of this setup was $3-4 \text{ cm}^{-1}$, making it possible to interrogate individual vibronic features. The ratio between the lamp-induced fluorescence signal and the absorption signal of each spectral feature gives the relative quantum yields Y . These relative Y values were calibrated to give absolute quantum yields from photoselected vibrational states.

6.E Medium-Resolution Absorption and Quantum Yield Data in Jets; ^{and} The "laser-free" spectroscopic methods were improved by the use of a medium-resolution spectrograph. The spectral resolution accomplished for absorption and fluorescence excitation spectra in jets was 0.05 \AA . Rotational contours of large molecules and the dependence of quantum yields on rotational excitations can now be explored.

6.F Two-Photon Spectroscopy. The high local densities attained in the pulsed planar supersonic expansion are beneficial for two-photon spectroscopy. High-resolution vibrational two-photon spectra of ultracold large molecules, interrogated by monitoring two-photon fluorescence excitation spectra, were recorded.

6.G The Construction of a High-Temperature, Pulsed, Supersonic Nozzle. A pulsed supersonic nozzle, which can be operated routinely in the temperature range $20-520^\circ\text{C}$ was built. The gas pulse, which was interrogated by delayed laser-induced fluorescence from the seeded jet, was characterized by a width (fwhm) of 200 \mu sec . The nozzle diameter was 0.06 cm . This pulsed high-temperature nozzle enabled us to conduct studies of porphyrins in supersonic jets.

6.H psec Time-Resolved Spectroscopy in Jets. An experimental system was constructed consisting of a mode-locked dye laser in conjunction with a fast detection system. The pulse width of the laser was 7 psec in the range 5600-6400 Å and 5 psec in the range 2800-3200 Å. A time-resolved fluorescence was detected by a fast time analyzer, which consists of a photomultiplier (with a modified resistor chain), an amplifier and a pair of constant fraction discriminators. The response to the system was characterized by a pulse width of 100 psec (fwhm), allowing for the measurement of lifetimes in the range 10 nsec-100 psec.

7. ACCOMPLISHMENTS OF OBJECTIVES

Some
 Some accomplishments since the initiation of the research project are listed below. *such as the records, etc.*

7.A Absorption Spectroscopy of Large Molecules. *S* Absorption spectra of ultracold aniline, anthracene, perdeuterated anthracene, *and other* 9-bromo-anthracene, 9,10 dichloro-anthracene, 9,10 dibromo-anthracene, fluorene, pyrene, perylene and tetracene in the vicinity of their $S_0 \rightarrow S_1$ vibrational excitations were recorded. Apart from demonstrating the potential of this new method, these spectroscopic data provide novel information regarding (i) relative quantum yields from different groups of rotational states, and (ii) quantum yields from photoselected vibrational states in the S_1 manifold.

7.B Rotational Profiles of Large Molecules. These involved medium-sized molecules (indole) and large molecules (anthracene and tetracene). The rotational contours of the $S_0 \rightarrow S_1$ transition of these molecules were interrogated using laser-induced fluorescence (resolution 0.3 cm^{-1}), lamp-induced fluorescence (resolution $0.3\text{-}0.5 \text{ cm}^{-1}$), and absorption (resolution $0.3\text{-}0.5 \text{ cm}^{-1}$) spectra. The rotational envelopes were fitted by computer-generated contours. These data pertain to rotational excitations of the largest molecules obtained up to date, providing spectroscopic information on excited-state molecular structure and directions of transition dipole moments in isolated, bare molecules.

7.C Absorption Line Profiles of Large Molecules. We have applied the techniques of absorption spectroscopy of large molecules in planar, supersonic jets to record the spectra involving excitation to "dark" states which are characterized by fluorescence quantum yields of $\sim 10^{-5} - 10^{-6}$. We have recorded the $S_0 \rightarrow S_1$ transition of the azulene molecule, which is characterized by a very low fluorescence quantum yield ($\sim 5 \times 10^{-6}$) and a low oscillator strength (9×10^{-3}). The absorption signals recorded were $(1-25) \times 10^{-4}$, being lower by 1-2 orders of magnitude than those recorded for the origin of other large molecules in jets. The absorption line profile of the origin of the $S_0 \rightarrow S_1$ transition of azulene is a Lorentzian originating from relaxation broadening. This Lorentzian lineshape qualitatively differs from the rotational contours observed for large molecules, which are characterized by long (nsec) decay lifetimes.

7.D Fluorescence Quantum Yields. Our new "laser-free" technique for fluorescence quantum yields from photoselected vibrational states of ultracold molecules provides a general method for the interrogation of excited-state relaxation in large molecules.

In comparison with the laser-induced fluorescence technique, the following advantages of the present combination of lamp-induced fluorescence and absorption spectroscopy should be emphasized: (a) Direct and accurate information is obtained concerning the ratio τ_{nr}/τ_r of the nonradiative (τ_{nr}) and pure radiative (τ_r) decay lifetimes. (b) Nonradiative lifetimes in the $\tau_{nr} = 10$ psec can be interrogated. (c) Easy tunability over a broad wavelength region, currently spanning the visible and UV regions. With small modifications the apparatus can span the VUV range. Three disadvantages of our approach should be noted: (1) The lamp-induced fluorescence signal is considerably weaker than a characteristic laser-induced fluorescence. (2) The overall sensitivity of the absorption signal and of γ was improved, being determined by the signal to noise ratio of $S/N = 5000$. (3) The material consumption rate is much higher (being up to 100 mg/hour) than in conventional jet spectroscopy.

- 7.E Absolute Fluorescence Quantum Yields. We have developed a method for the determination of absolute quantum yields from the electronic origin, $S_1(0)$, of the first electronically excited singlet state of isolated large molecules cooled in pulsed, planar, supersonic expansions. We have measured the absorption spectra, the fluorescence excitation spectra and the fluorescence quantum yields from the photoselected $S_1(0)$ states for pairs of large molecules seeded in planar supersonic expansions using a pulsed xenon lamp and a monochromator. The relative quantum yields thus obtained were calibrated using a reference molecule to give absolute quantum yields, Y , from the vibrationless S_1 states. Absolute Y values from the $S_1(0)$ state of thirteen aromatic molecules and their derivatives were determined. Information is inferred on the mechanism of intersystem crossing mediated by a nearby triplet state and on solvent effects on this process.
- 7.F Nonreactive Electronic Relaxation in the S_1 State of Large Molecules. Information on intramolecular dynamics in the S_1 manifold of a variety of large molecules was inferred from Y values. Our studies fall into two categories: (1) Manifestation of nonreactive intramolecular relaxation involving interstate electronic relaxation and intrastate vibrational energy redistribution (in the S_1 state of perylene, pyrene, tetracene, anthracene, perdeutero-anthracene, 9,10 dichloro-anthracene, 9 bromo-anthracene, fluorene, naphthalene and aniline). (2) Unimolecular rearrangement and isomerization in isolated molecules (S_1 state of trans-stilbene).

The dependence of the fluorescence quantum yields on the excess vibrational energy, E_v , in the S_1 manifold of isolated large molecules reveals the following novel characteristics of nonreactive dynamics: (a) The Y versus E_v dependence in several molecules reveals a decrease at about $E_v = 1000-1500 \text{ cm}^{-1}$ followed by saturation of Y . A notable exception is fluorene, where Y is constant over the range $E_v = 0-5000 \text{ cm}^{-1}$. (b) The Y versus E_v dependence in the S_1 state of 9,10 dichloro-anthracene exhibits a "step-function-type" behavior, which is attributed to intersystem crossing mediated by the higher T_2 triplet state. (c) The comparison between Y data and direct decay lifetime data for the S_1 manifold of tetracene reveals that the pure radiative lifetime is independent of E_v in the range $0-1000 \text{ cm}^{-1}$, while in the range $E_v = 1000-1500 \text{ cm}^{-1}$ τ_r is higher by a numerical factor of 1.5. This

7.F (cont'd)

observation originates from a "dilution effect" resulting from interstate coupling. (d) A surprising large inverse isotope effect on Y from the $S_1(0)$ state of anthracene and perdeutero-anthracene was observed. The ratio of the quantum yields for the electronic origin is $Y_H/Y_D = 5$. (e) The $S_1(0)$ state of 9 bromo-anthracene is characterized by $Y = 2.5 \times 10^{-4}$ and $\tau_{nr} = 75$ psec, providing an example for the interrogation of a "dark" excited state. (f) The constant value of Y at high E_y values, $E_y \geq 1500$ cm^{-1} , is a universal feature reflecting the manifestation of intrastate vibrational relaxation. (g) The step-function behavior of Y of 9,10 dichloro-anthracene was utilized to probe the flow of vibrational energy in 9,10 dichloro-anthracene- Ar_n ($n = 1-4$) van der Waals complexes.

7.G Photochemistry in Large Isolated Molecules. The techniques of spectroscopy and quantum yield determinations in supersonic jets were applied for the exploration of photochemical rearrangements in isolated molecules. The first application involved trans-cis isomerization of trans-stilbene. The absorption spectra, the fluorescence excitation spectra and the relative fluorescence quantum yields of trans-stilbene in planar supersonic jets were determined for excess vibrational energies, $E_y = 0-5000$ cm^{-1} above the origin of S_1 . The nonradiative decay rates reveal an onset at $E_y = 900$ cm^{-1} , exhibit the erosion of vibrational photoselectivity for isomerization at $E_y = 900-500$ cm^{-1} and show a linear dependence on E_y for $E_y = 1500-5000$ cm^{-1} . A heuristic description of the photoisomerization dynamics is provided in the terms of the classical RRK formula. However, the dynamics of the trans-cis isomerization presumably involves coupling to a second excited singlet state.

7.H Subnsec Dynamics in jets. The fast time-resolved system enabled the interrogation of real-time intramolecular photochemistry in isolated large molecules. We measured the time-resolved fluorescence decay lifetimes from photoselected vibrational states in the S_1 manifold of trans-stilbene, 4-Cl trans-stilbene and 4-methyl trans-stilbene cooled in supersonic jets and excited by a mode-locked dye laser. Decay lifetimes as short as 100 ± 30 psec were

7.H. (cont'd)

recorded, using a fast single photon counting system. The energy dependence of the nonradiative decay rates, k_{nr} , of trans-stilbene is invariant with respect to the para-substitution of Cl, whereupon the intersystem crossing to the triplet in 4-Cl trans-stilbene does not provide the main route for photo-isomerization and constitutes a distinct, energy dependent, decay channel. The invariance of k_{nr} with respect to para-methyl substitution indicates that intrastate vibrational energy redistribution (IVR) does not involve all accessible molecular modes. The experimental data are consistent with a nonadiabatic trans-cis isomerization in the isolated molecule, which involves S_1 - S_2 internal conversion in conjunction with IVR.

7.I Spectroscopic Manifestation of Intramolecular Relaxation. We have observed line profiles in the absorption spectra for the $S_0 \rightarrow S_1$ electronic origin and for the 659 cm^{-1} and 1394 cm^{-1} vibrational excitations in the S_1 manifold of azulene in pulsed, planar, supersonic expansions. The S_1 electronic origin exhibits a Lorentzian lineshape, whose homogeneous linewidth results in the lifetime $\tau = 0.8 \pm 0.2$ psec for interstate electronic relaxation in the isolated molecule. An interesting effect of shortening the electronic relaxation decay lifetime in the isolated molecule (0.8 psec) relative to the medium-perturbed molecule in a low-temperature mixed crystal (3 psec) was established.

7.J Ultrafast Relaxation of the S_2 State of Aromatic Molecules. Absorption studies of the $S_0 \rightarrow S_2$ transition of several large molecules (fluorene, anthracene and tetracene) in jets revealed dramatic line broadening due to electronic relaxation. Of particular interest is the $S_0 \rightarrow S_2$ transition of a phenantrene. The line profile of the electronic origin of this $S_0 \rightarrow S_2$ transition is Lorentzian with a homogeneous width of $\Delta = 11 \text{ cm}^{-1}$ for electronic relaxation in the isolated molecule. The $S_0 \rightarrow S_2$ origin of isolated phenantrene,

7.J (cont'd)

which corresponds to the statistical limit ($\Delta = 11 \text{ cm}^{-1}$), drastically differs from the same transition of phenantrene in durene ($\Delta = 220 \text{ cm}^{-1}$), which reveals the features of the intermediate level structure. The dramatic medium-enhanced intramolecular S_2 - S_1 coupling of phenantrene is attributed to the modification of the electronic energy gap.

7.K Excited-State Energetics of Porphyrins. We have studied laser-induced fluorescence spectra of several porphyrins in supersonic jets. Detailed information on the electronic-vibrational level structure of free-base porphine, Zn-tetrabenzoporphyrin, free-base tetraphenylporphyrin, Mg-tetraphenylporphyrin and Zn-tetraphenylporphyrin was obtained. With regard to the electronic level structure, we have attained evidence for the electronic degeneracy of the S_1 and S_2 states in symmetrically substituted metal porphyrins and have explored symmetry breaking effects on the level structure. Concerning the vibrational level structure, detailed information was obtained on vibrational modes of isolated porphyrins and on novel aspects of low-energy motion.

7.L Ultrafast Electronic Relaxation from Higher Singlet States of Porphyrins. Quantitative information on psec and sub-psec lifetimes of the second excited singlet state of porphyrins was obtained for the Lorentzian optical lineshape data. The lifetime of the S_2 state of Zn-tetrabenzoporphyrin is 3.2 ± 0.2 psec, while the lifetime of the S_1 state of free-base porphine is 0.5 ± 0.1 psec.

7.M van der Waals Complexes of Large Molecules with Rare Gases. Energy-resolved and mass-resolved studies by two-photon ionization techniques were applied for the elucidation of the excited-state energetics of fluorene rare-gas complexes. Information was obtained on the low-energy nuclear excitations involving the motion of the rare-gas atoms relative to the large molecule.

- 7.N Spectral Shifts of van der Waals Complexes. These spectral shifts provide information on microscopic solvation effects. In this context, the additivity of the spectral shifts bears on structural information, which can be inferred for such data. Accurate information on the magnitude of the microscopic spectral shifts of tetracene-Ar_n (n = 1-6) complexes emerges from fluorescence excitation spectra in pulsed planar jets, which were obtained using a pulsed xenon lamp and a monochromator. The nonadditivity of the spectral shifts per added Ar atom was established, providing structural information on these large complexes.
- 7.O Large Molecular Complexes. We have conducted studies of laser-induced fluorescence spectroscopy of M·X_n van der Waals complexes, consisting of medium-sized molecules (X = methane, carbon tetrachloride, ammonia, water, methanol, acetonitrile, benzene and toluene) bound to a large aromatic molecule (M = tetracene and fluorene). These large complexes were synthesized in pulsed supersonic jets of He seeded with M and X. The spectral features assigned to S₀ → S₁ electronic-vibrational excitation of these large M·X₁ complexes, which are characterized by narrow line widths (fwhm) of 2-4 cm⁻¹, were attributed to three distinct categories: (i) A vibrationless excitation of M·X₁, (ii) vibrational excitations of M in M·X₁, and (iii) excitations of the vibrations involving relative motion of M and X in the M·X₁ complex.
- 7.P Absorption Spectra of van der Waals Complexes. We have extended the arsenal of experimental techniques for the interrogation of excited states of large complexes to include absorption spectroscopy. This development enabled us to explore "dark" excited states. Furthermore, absorption spectroscopy in conjunction with fluorescence excitation spectroscopy of complexes provides quantum yield data, which are of considerable interest for the elucidation of reactive vibrational predissociation and nonreactive intramolecular vibrational energy redistribution in complexes. A detailed study of absorption spectra of the complexes of anthracene and its derivatives with Ar have been performed.

- 7.Q Excited-State Dynamics of Complexes of Large Molecules with Rare Gases. The absorption spectra and the fluorescence excitation spectra of the complexes of 9,10 dichloro-anthracene (DCA) with Ar atoms were studied for the $S_0 \rightarrow S_1(0)$ vibrationless transition of $DCA \cdot Ar_n$ ($n = 1-6$), and from the $S_0 \rightarrow S_1$ (1390 cm^{-1}) transition of $DCA \cdot Ar_n$ ($n = 1-4$). Information on the structure of these complexes was inferred from the additivity of the spectral shifts per added rare-gas atom (ASSRA) for $DCA \cdot Ar_n$ ($n = 1,2$) and from deviations from the ASSRA for $DCA \cdot Ar_n$ ($n = 3-6$). The vibrational predissociation (VP) dynamics of $DCA \cdot Ar_n$ ($n = 1-3$) complexes was interrogated by fluorescence quantum yield, Y , measurements. The value of the S_1 (1390 cm^{-1}) state of $DCA \cdot Ar_n$ ($n = 1-3$) exhibits a dramatic enhancement relative to that of DCA. Utilizing the dependence of Y on the excess vibrational energy of bare DCA, we were able to estimate the internal energy of the fragments resulting from VP of $DCA \cdot Ar_n$ ($n = 1-3$). An upper limit of ~ 100 psec was estimated for the VP (and/or vibrational energy redistribution) lifetime from the S_1 (1390 cm^{-1}) state of $DCA \cdot Ar_3$.
- 7.R Complexing of Porphyrins in Supersonic Jets. The complexing of porphyrins is a subject of considerable interest in the areas of physical chemistry and biophysical diagnosis. We have applied the techniques of laser spectroscopy in seeded supersonic expansions for the synthesis, identification and exploration of excited-state energetics of isolated ultracold complexes of porphyrins with medium-sized molecules ($L =$ water, methanol, acetonitrile, benzene and pyridine), providing a new approach for the elucidation of solvent perturbations on porphyrins, as explored from the microscopic point of view.
- 7.S Aromatic Molecules in Large Argon Clusters. We have studied the excited-state energetics and dynamics of several large molecules, i.e., anthracene, tetracene and pentacene, in large clusters of Ar, which were synthesized in high-flow supersonic jets (stagnation pressure $p = 3000-14000$ torr expanded through a $D = 150\mu$ nozzle).

- 7.T Level Structure and Dynamics of Clusters. Some features of the structure, the dynamics of nuclear motion, the nature of electronic states, excited-state energetics and relaxation phenomena in "isolated" nonmetallic clusters were explored from a theoretical point of view with an emphasis on the interrelationship between the characteristics of molecular and condensed phase systems.
- 7.U Analytical Applications. We have considered some applications of the techniques of laser spectroscopy in supersonic jets to analytical chemistry focusing attention on:
- (1) Identification of large molecules.
 - (2) Sensitive detection of minor traces of aromatic molecules.
 - (3) Isotopic analysis.

8. PUBLICATIONS

The following manuscripts supported by this research grant were prepared and submitted for publication.

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