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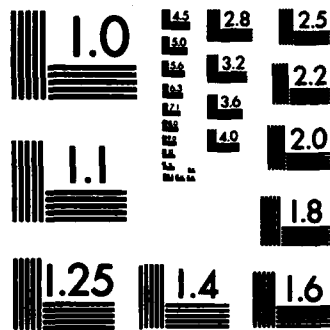
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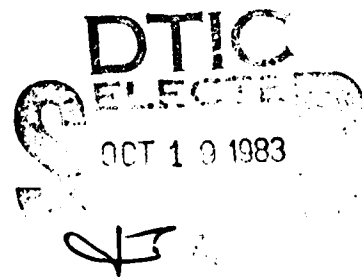
Collisional Energy Exchange in Polyatomic Molecules

by

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21 August 1983



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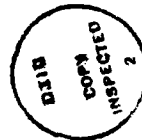
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<p>Fourier Transform Infrared Spectrometry (FTIS) has been used to study several kinds of gas-gas and gas-surface collision processes brought about with and in supersonic free jets in vacuo. Following are some key results:</p> <p>(1) Terminal distributions of rotational energy in free jets of CO and CO<sub>2</sub> show a non-Boltzmann distribution that can be characterized by a two-temperature model and explained in terms of competition between rotation-rotation and rotation-translation transfers. (2) Spectra of CO<sub>2</sub> molecules excited by collisions with N<sub>2</sub> molecules indicated similar two-temperature distributions, possibly due</p>		

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to secondary collisions between excited  $\text{CO}_2^*$  and  $\text{N}_2^*$ . (3) The accommodation of vibrational and rotational energy during collisions between  $\text{CO}$ ,  $\text{CO}_2^*$  and  $\text{NO}$  molecules and hot platinum surfaces has been determined over a range of surface temperatures. (4) Excess internal energies have been determined in nascent  $\text{CO}$  and  $\text{CO}_2^*$  molecules formed respectively by catalytic oxidation of  $\text{C}$  and  $\text{CO}$  on a platinum surface. An energy balance indicates that about  $1/3$  of the available reaction energy is absorbed by the surface, the remainder going mostly into vibrational modes of product molecules.



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## I. Introduction

For the past three years under this grant we have been pursuing a program of research relating to the details of energy exchange processes during collisions between molecules in the gas phase and between molecules and surfaces. This research has been characterized by a combination of methods based on supersonic free jet expansions in vacuum molecular beam methods with spectroscopy. In particular, we use supersonic free jets expanding in vacuo to bring about the collisions and Fourier Transform Infrared Spectrometry to determine the states of one or both collision partners before and/or after the encounter. In what follows we will set forth briefly what we have been able to accomplish in each of the several topics that we have investigated.

## II. Achievements and Results

1. TV Excitation Cross Sections. The ability to obtain high translational energies by aerodynamic acceleration provided the basis for some of the research that we have been carrying out under this program for the past several years. Our original objective was to determine the cross sections for TV excitation of molecules at collision velocities in the range from 2 to 8 kilometers per second. In particular, we wanted to measure cross sections for the excitation of CO<sub>2</sub> molecules during collisions with N<sub>2</sub>, O<sub>2</sub>, and Ar. In this velocity range, the center-of-mass collision energies for these species range from 0.1 to 5 or so eV. The reason for interest in these cross sections stems from the belief that TV excitation collisions between combustion product molecules and atmospheric species may account for some features of the infrared radiation from the exhaust plumes of rockets flying at high altitudes. If the excitation cross sections and their energy dependence were known, it would be possible to compute the amount and distribution of radiation to be expected.

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The idea of the experiment was to bring about the high energy collisions by opposing ~~two uncollimated molecular beams (free jets in the free molecular flow regime)~~. Each jet was obtained by expanding a mixture of one of the collision partners in hydrogen or helium. The intersection region of the two jets, where the high energy collisions occur, occupied the field of view of a sensitive infrared detector. Collision energy was varied by changing the concentration of the seed species in the carrier gas. To a more circumscribed extent, the source temperature could also be varied. There are two prerequisites to obtaining observable signals: molecules must become excited and they must radiate before they leave the field of view of the detector. This latter requirement compounds the problem of an already harassed experimenter because the radiative lifetimes of the expected states are at least several milliseconds. The residence time of an excited molecule in the field of view of the detector is of the order of a few times  $10^{-5}$  seconds. The net result is that one can only hope to see a small fraction of the excited molecules, the impatient ones that radiate early. By painstaking attention to many small details such as alignment, optical focussing, careful baffling and improved calibration procedures, and after many frustrations and disappointments, we finally succeeded in increasing signal and reducing noise to the point where we could measure cross sections as small as a few times  $10^{-20} \text{ cm}^2$ . We determined for the first time the TV cross section for the asymmetric stretch mode of  $\text{CO}_2$  by collisions with  $\text{N}_2$  and found that it rises steeply from a value of about  $3 \times 10^{-20} \text{ cm}^2$  at collision energies just above the threshold value of 0.288 eV to about  $5 \times 10^{-17} \text{ cm}^2$  at a collision energy of 2 eV. Most gratifying was the fact that measurements made when the jets intersect at 90 degrees were entirely consonant with those made when the intersection angle was 180 degrees. We also obtained cross sections for excitation of the same mode with argon, oxygen, and hydrogen as the collision partners. (2) We are now engaged in trying to carry out similar measurements with  $\text{H}_2\text{O}$  as the species to be excited.

These IV excitation cross sections were "total" in the sense that they related to all the radiation in the wavelength range of the InSb detector, 2 to 5 microns. It happens that for CO<sub>2</sub> the asymmetric stretch mode is the only one that can radiate in that interval. We then became interested in knowing the distribution of rotational energy in the collisionally excited molecules and were fortunate enough to obtain a Fourier Transform Infrared Spectrometer (FTIS) to see if we could spectrally resolve the radiation. This new task has proved to be inordinately more difficult than the measurement of total excitation cross sections. In that case the detector was in the vacuum system only a few centimeters from the collision region. In the case of the FTIS experiment we had to "pipe" the radiation out of the vacuum system and through a window before it could enter the spectrometer. In addition, only a small fraction of the total radiation fell within the wavelength range for each of twenty or more rotational lines. After nearly three years of trying, we finally obtained a useful spectrum for an optimum combination of collision velocity and flux density with CO<sub>2</sub> and N<sub>2</sub>.<sup>(3)</sup> We will have to achieve further improvements in signal-to-noise before we can make measurements at other velocities.

This first elusive spectrum reveals a distribution that can best be described in terms of two sub-populations, one at a rotational temperature of 64 K, the other at a temperature of 340 K. Our tentative explanation is that the molecules in the low temperature group represent those which have had a single exciting collision with an N<sub>2</sub> molecule. We would expect low rotational energies for these molecules because only collisions with small impact parameter would put enough energy in the center of mass for vibrational excitation. The high temperature population comprises those molecules which have had a second collision after being vibrationally excited. Such a second collision would have a large probability of exciting higher J levels. We emphasize the tentative nature of this explanation of what we have observed. Further work will be required before we can claim any confidence in such a preliminary result.



2. Terminal States in Free Jet Expansions. During our long struggle to obtain a TV excitation spectrum, we made some other kinds of FTIS studies that had their roots in what we learned during the original measurements of total TV excitation cross sections. One of the major problems in these measurements was the calibration of the detection system. Originally we had tried, without conspicuous success, to use a black body source at a known temperature. We then hit upon a scheme which had its origins in some of our earlier studies of internal energy relaxation during free jet expansion. Those studies were based on TOF measurements in beams extracted from free jet sources. Such measurements gave directly the amount of translational energy in the beam molecules. Because the free jet expansion is adiabatic, an energy balance allowed us to infer the amount of residual internal energy in the beam molecules. It is possible to determine the relaxation rate for that mode in which the residual energy is contained. Indeed, during the work reported here, we made a substantial advance in calculational methods for relating the residual energy to the relaxation rate. (1) We had found in our earlier studies that in the case of  $\text{CO}_2$  and  $\text{CO}$ , essentially none of their original vibrational energy was dissipated in the expansion. Thus, the terminal vibrational temperature of these species in the free jet was the same as the source temperature. Because we know with some precision the density distribution along the jet axis and thus in the field of view of the detector, we could calibrate the detector in situ simply by heating the source gas to an appropriate temperature and measuring the radiation from the free jet molecules.

After we obtained the FTIS we examined in some detail the spectral character of the radiation from  $\text{CO}_2$  and  $\text{CO}$  molecules after free jet expansion from a heated nozzle. The results were very interesting. We found that the distributions were markedly non-Boltzmann and therefore could not be characterized by a single temperature. Other investigators have found similar departures from a Boltzmann distribution. The advantage of FTIS is that it provides a much wider range of J values than other methods can conveniently embrace. It turned out that over a wide range of source conditions, giving

rise to a wide range of departures from "Boltzmannality," we could always achieve an excellent description of the observed rotational distributions in terms of a linear combination of two Boltzmann distributions, a concept already mentioned in the above discussion of the TV excitation spectrum. This two-temperature model may simply be regarded as an empirically useful device but it led us to the possibility of characterizing the departure from a Boltzmann distribution in terms of a single parameter, the molar rotational entropy deficit in terminal jet molecules, a sort of integrated surprisal. An investigation of the dependence of this entropy deficit upon source conditions seems to be explainable in terms of a competition between RT and RR transfers in rotational relaxation during free jet expansion. Some of these results have already been published. (4) A paper on subsequent work is in preparation. We believe that further studies of this kind may allow us to evaluate the magnitude of these RR transfer cross sections. We know of only one other experiment that provides information on such exchanges, a recent elegant measurement by Roger Miller. Using the laser-bolometer technique, he determined the change of rotational state in a beam of HF molecules scattered through very small angles by a crossing beam of HF molecules.

3. FTIS Applied to Gas-Surface Interactions. In order to cover as wide a range of collision energies as possible in our original TV excitation studies, we sometimes resorted to raising the temperature of the source from which came the collision partners for the vibrationally cold CO<sub>2</sub> molecules, i.e., the N<sub>2</sub>, Ar, O<sub>2</sub> or H<sub>2</sub> free jet that opposed the CO<sub>2</sub> free jet. Consequently, part of the calibration procedure consisted in determining the contribution of radiation from the hot nozzle to the detector signal. During one of these calibration procedures, we turned off the gas flow in the heated nozzle and let the cold chopped CO<sub>2</sub> jet to continue to run. The result was a fairly strong detector signal that we traced to CO<sub>2</sub> molecules rebounding from the heated end of the opposing nozzle. In short, a substantial fraction of the CO<sub>2</sub> molecules were being

vibrationally excited by a single collision with a hot surface. This observation led to the series of experiments on gas-surface interactions. In one, we studied the vibrational and rotational accommodation of molecules incident on heated surfaces. In the other, we examined the internal energy states of nascent molecules formed by catalytic reaction on a platinum surface.

A. Accommodation Studies. In these experiments, we intercepted the free jet with an electrically heated surface far enough from the nozzle exit so that there were essentially no encounters between incident and rebounding molecules. The FTIS was focussed on the region immediately above the surface. We will summarize briefly some of the highlights of results which have been obtained at surface temperatures ranging from 700 K to 1450 K.

(a) CO<sub>2</sub> on Platinum. The asymmetric stretch mode became excited in about 20 per cent of the incident molecules. The absence of hot bands indicated that bending and symmetric stretch modes did not become excited in these molecules that were asymmetrically excited which are the only ones we see. The rotational energies of these excited molecules had a Boltzmann distribution corresponding to a temperature about half the surface temperature.

(b) CO on Platinum. About 70 per cent of the incident molecules became vibrationally excited. The relative populations of the first and second levels revealed in the spectrum indicated a vibrational temperature identical with the surface temperature. The distribution of rotational energy was non-Boltzmann and corresponded to roughly 60 per cent accommodation.

(c) NO on Platinum. The vibrational temperature of the excited molecules was about 90 per cent of the surface temperature, which was 1430 K. The distribution of rotational energy was non-Boltzmann and corresponded to an energy accommodation of about 60 per cent. Spin orbit temperatures were determined from the relative

populations in the  $2s\ 1/2$  and  $2p\ 3/2$  states. They varied with J level, decreasing from about 1100 K at the highest J's to about 230 K at the lowest. It is interesting that  $T_{2s}$  for the low J levels is well below both  $T_{\text{surface}}$  and " $T_{\text{rotation}}$ ".

These accommodation studies have been more completely described in two papers already published. (5,6) Our results differ from and are supplementary to those obtained by various investigators who use LIF probing of the departing molecules. Because of greater sensitivity they can obtain angular distribution information and can work with clean surfaces at room temperature under ultra-high vacuum conditions. We must work with high surface temperatures, both to avoid contamination and to excite the molecules to radiating states. Fortunately, we can study the behavior of some species that do not lend themselves conveniently to LIF methods with lasers now available.

B. Reactive Scattering Studies. Having been able to obtain useful signals from molecules excited by single collisions with other surfaces, we were encouraged to attempt a study of the internal states of molecules formed by reaction on a catalytic surface. Becker et al. at Chicago had found by TOF measurements that  $\text{CO}_2$  formed by catalytic oxidation of CO on a platinum surface came off with about 4 kcal/mol more translational energy than thermal equilibrium with the surface would account for. Accordingly, we expanded a mixture of CO and  $\text{O}_2$  in a free jet incident on a polycrystalline platinum surface at 700 K. A typical spectrum of emission from the nascent product molecules is very "busy" with lots of hot bands indicating vigorous excitation of all vibrational modes. Analysis of this spectrum leads to reasonably reliable values of "temperature" for the symmetric stretch, bending and rotational modes. With these temperatures, we can compute a spectrum using the asymmetric stretch temperature as a fitting parameter spectrum. Superposition of the simulated and experimental spectra shows a remarkably good match. From this fitting, we are able to obtain with some confidence the amount of energy in each mode. It emerges that relative to thermal equilibrium at the surface temperature

there are excess energies of about 3.8 kcal/mol in bending, 3.4 kcal/mol in asymmetric stretching, 0.8 kcal/mol in symmetric stretching and 0.3 kcal/mol in rotation. The energy of the transition complex at the top of the activation barrier is known to be 29.2 kcal/mol. The Chicago group's TOF results, as already noted, show 4.0 kcal/mol in translation. An energy balance lets us conclude that about 10.3 kcal/mol is absorbed by the surface. A preliminary report of these findings is already in print. (7) A more complete exposition has been submitted for publication. (8) Although these results, of which we are very proud, provide the most comprehensive accounting of energy distribution yet achieved for a surface catalyzed reaction, that accounting cannot yet be considered complete. There remains the possibility that some of the available reaction energy could have been stored in symmetric stretch, bending and rotational modes of molecules that were in the ground state with respect to asymmetric stretch. One of the things we hope to do in future research is to develop methods of using absorption spectrometry to analyze the energy distribution of molecules that are not excited in the asymmetric stretch mode.

Kori and Halpern in our laboratory have carried out an analogous study of CO oxidation on a platinum surface in which O atoms from a microwave discharge instead of O<sub>2</sub> molecules were the oxidizing agent. The results were similar, except that reaction occurred even when the surface was at room temperature. An interesting observation was that if the platinum surface was completely covered with oxygen, no CO<sub>2</sub> was formed, but the rebounding molecules of CO were highly excited both vibrationally and rotationally. Indeed, the spectrum was essentially a continuum. We are a bit mystified by this result and speculate that by some unknown mechanism the recombination energy of the O atoms ended up in rotational and vibrational modes of the CO. A more interesting result was obtained when the platinum surface contained carbon atoms at concentrations substantially below a monolayer. Under these conditions an incident flux of O or O<sub>2</sub> resulted in the formation of CO molecules excited up to the seventh vibrational level. A suprisal analysis of these

observations led to the conclusion that the transition complex probably comprised a carbon atom bound to three surface Pt atoms and one O atom. A paper on this work has been submitted for publication. (10)

A very recent and exciting extension of this work has recently been carried out in our laboratory. A pulsed nozzle in conjunction with a soft ware FTIS program for time-resolution of spectral components has revealed the dependence on surface coverage of the internal state of product molecules. It emerges that there is less excess energy at low coverages than at high coverages. A paper has been submitted to Surface Science. (9)

Another result of interest in the surface reaction arena made possible by the FTIS was the finding that a flux of N atoms and O<sub>2</sub> molecules on a platinum surface resulted in the formation of copious quantities of N<sub>2</sub>O. (11) The conditions of this early experiment were such that any radiation due to excitation in the nascent N<sub>2</sub>O was overwhelmed by radiation from product molecules that were excited by collisions with vibrationally excited N<sub>2</sub> from the microwave atom source. In the future we hope to re-examine this reaction using the "cleaner" techniques we have developed in the CO oxidation studies.

It seems clear that the combination of FTIS with free jet molecular beams comprises a powerful method of probing the details of at least some important surface reactions. We hope to extend our studies in this exciting arena.

(a) Review Papers

- (i) A chapter entitled "Collision Kinetics in Gas Dynamics" was prepared for the five volume treatise on Applied Atomic Collision Physics edited by Massey, Bederson and McDaniel and published by Academic Press. It treated supersonic free jet expansions as an example of the interplay between the continuum and particulate

- (ii) A tutorial review article entitled "Order from Chaos with Molecular Beams" was prepared for the Proceedings of the Indian Academy of Science. It comprised an expanded version of an invited lecture delivered to the Academy. (13)
- (iii) A review paper entitled "Where Are we Going with Molecular Beams" was presented by invitation at the 13th International Symposium on Molecular Beams at Novosibirsk in July 1983. It will appear in the proceedings to be published by Plenum Press. (14)

4. Other Related Efforts. In addition to the specific research studies summarized in the previous paragraphs, we also carried out several general tasks made possible in part by the support of this grant. These include:

(b) Miscellaneous Articles

- (i) A note entitled "Clusters as a Possible Source of Error in Molecular Beam Scattering Experiments" was based on results obtained in part under AFOSR sponsorship. (15)
- (ii) A letter to the editor was published in Review of Scientific Instruments entitled "Further Reflections on the Role of a Mass Flux Gauge in the Calibration of Mass Spectrometric for Dimers." It was in response to a letter by W. R. Gentry on the problem of calibrating a mass spectrometer for dimer assay. (16)
- (iii) A note was submitted to the Journal of Chemical Physics entitled "On the Time Required to Reach Fully Developed Flow in Pulsed Supersonic Free Jets" with K. L. Saenger of MIT as co-author. (17)
- (iv) An article entitled "Research is Engineering" was published in Chemical Engineering Education. It argued that research in general and on Molecular Beams in particular was indeed important practical educational experience for engineers. (18)

### III. Professional Personnel

Except where otherwise noted, all of the following participants in this program are associated with the Department of Chemical Engineering at Yale:

Principal Investigators: S. B. Ryali and J. B. Fenn

Faculty Collaborators: G. L. Haller and B. L. Halpern, Dhan Sethi (Dept. Chemistry, University of Bridgeport)

Postdoctoral Associates: S. P. Venkateshan, M. L. Labowsky

Graduate Students: Yih Fen Maa, Moris Kori, D. A. Mantell (Physics; Ph.D. recipient, Spring 1983)

### IV. Interactions

This program had its origins in a joint venture with Aerodyne Research Laboratories of Bedford, Mass. We have maintained a working relation with that group by visits and telephone calls, in particular with Drs. C. E. Kolb and J. A. Silver. We have also maintained communications with Dr. A. Rahbee and his colleagues at the Air Force Geophysical Laboratories at Hanscomb Air Force Base.

Lectures on various aspects of this research have been given at the following places: FOM Laboratory of Atomic and Molecular Physics in Amsterdam, Max Planck Institute for Fluid Mechanics in Goettingen, University of Marburg (West Germany), University of Munich, University of Bern, University of Lausanne, University of Kaiserslautern, University of Karlsruhe, Munich Chemical Society, University of Sydney, Australian National University (Canberra), Australian Physical Society (Adelaide), University of Queensland (Brisbane), University of Canterbury (Christ Church, New Zealand), Indian Institute of Science (Bangalore), Baba Research Institute (Bombay), University of Patras (Greece), University of Waterloo (Ontario), and Bell Telephone Laboratories.



## V. Publications

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3. S. P. Venkateshan, S. B. Ryali and J. B. Fenn, "Rotational Energy Distribution in CO<sub>2</sub> Vibrationally Excited by Collision with N<sub>2</sub>," Chem. Phys. Lett. 92, 606 (1982).
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9. D. A. Mantell, S. B. Ryali and G. L. Haller, "Time Resolved Infrared Emission Studies of CO<sub>2</sub> Formed by CO Oxidation on Pt and Pd," submitted to Chem. Phys. Lett.
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16. J. B. Fenn and M. Lee, "Further Reflections on the Role of a Mass Flux Gauge in the Calibration of Mass Spectrometers for Dimers," Rev. Sci. Instr. 53, 1494 (1982).
17. K. L. Seeger and J. B. Fenn, "On the Time Required to Reach Fully Developed Flow," J. Chem. Phys. (submitted).
18. J. B. Fenn, "Research Is Engineering," Chem. Eng. Educ., Fall 1982, 190.

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