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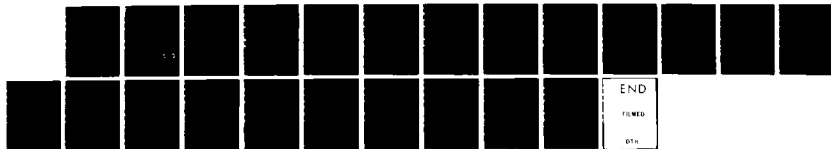
STUDY OF VIBRATIONAL ENERGY TRANSFER AT A LIQUID TIN
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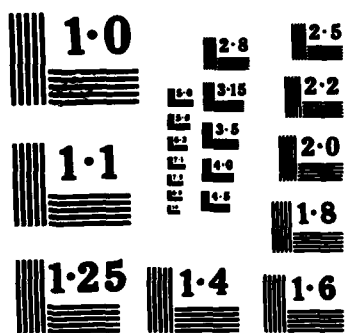
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Study of Vibrational Energy Transfer at a Liquid Tin Surface

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Study of Vibrational Energy Transfer at a Liquid Tin Surface*

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Abstract

Single collision vibrational energy transfer for a canonical ensemble ($T = 300$ K) of cyclobutene molecules at a liquid tin surface was studied at temperatures from 500 K to 775 K. Transport above the reaction threshold for isomerization to butadiene ($E_0 = 32.4 \text{ kcal mole}^{-1}$) was used as the criterion for efficiency of vibrational energy accommodation. The surface was found to function as a strong collider below 550 K. Experiments both above and below the freezing point revealed no discontinuity in the vibrational accommodation efficiency.



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Introduction

Thermal energy accommodation of molecular gases with solid surfaces has been an area of both experimental and theoretical interest for some time. Much attention has been focused on translational, and to a lesser extent, rotational energy accommodation at well characterized surfaces and, sometimes, in specific initial states¹⁻⁴. More recently, however, there has been an effort toward the measurement of internal energy accommodation by ensembles of gas molecules at a particular temperature that interact with less well-defined solid surfaces (polycrystalline materials in the low vacuum region). Rosenblatt and coworkers^{5,6} used a vibrating surface method to obtain translational and internal energy accommodation coefficients for a series of paraffins measured at relatively high pressures on a number of polycrystalline metal substrates. The lower limits of the room temperature vibrational accommodation coefficients (α_v) on Fe for CH_4 , $n\text{-C}_4\text{H}_{10}$, and $n\text{-C}_8\text{H}_{18}$ were estimated to be 0.36, 0.72, and 0.90, respectively. Foner and Hudson⁷, using a molecular beam sampling method, estimated a much lower value, $\alpha_v = 0.05$, for $n\text{-C}_4\text{H}_{10}$ on a Pt filament at 1273 K. A recent report that utilized a technique similar to that applied in the present work, gave $\alpha_v = 0.66$ for n -octane at 350 K on silica surfaces⁸.

Experiments in this laboratory using the single collision version of the variable encounter method (VEM)⁹ reveal a decrease of α_v with rise in temperature. The probability of excitation of gaseous molecules to energy levels above their reaction threshold by collision with a surface was measured. Some of these studies^{10,11} dealt with energy relaxation by cyclobutene at pyrex and quartz surfaces seasoned by reactant gas under low vacuum conditions, i.e. surfaces covered by a graphitic polymeric layer that gives

rise to reproducible behavior. Strong collider behavior was reported at temperatures below ~ 450 K. Polycrystalline gold surfaces appear to be even more efficient colliders; Yuan and Rabinovitch¹² reported very recently that strong collider behavior on gas-seasoned plane gold and wire surfaces was observed for cyclobutene up to 550-600 K. This led to speculation that metal surfaces may function as stronger colliders than pyrex and silica surfaces due to phonon and other relaxation mechanisms unique to metals. In any case, seasoning of the surface by the gaseous reactant evidently does not suffice to obscure all effects contributed by the underlying solid substrate.

In the present paper we report the results of a study of vibrational relaxation of cyclobutene at a liquid tin surface. Thermal accommodation studies of gases at liquid metal surfaces, besides being of intrinsic interest, have an interesting feature. In principle (but not achieved here), by rapid stirring of the metal at not-too-high pressures of gas, a "clean" surface may be continually generated and, ideally, one may obtain information concerning energy transfer on a clean liquid metal surface. In addition, it is possible, in principle, to determine the dependence of vibrational energy transfer efficiency upon phase. This can be investigated by measurements just above and just below the metal melting point.

Very little work appears to have been done on even conventional gas accommodation coefficients at liquid surfaces. The earliest work we have found concerned accommodation coefficients for several covalent liquids and their own vapors, as well as mercury and its vapor; only for water vapor was reflection, as opposed to penetration and absorption into the liquid, proposed^{13,14}. In all cases the values were unity. Translational accommodation coefficients for He on a tungsten filament coated with potassium metal just above and below its melting point (335 K) have been described by

Thomas and Petersen¹⁵. This study reportedly gave $\alpha_t(338 \text{ K}) = 0.095$ and $\alpha_t(328 \text{ K}) = 0.089$. These workers observed no discontinuity at the melting point.

For studies at higher temperatures where reaction takes place, reactive metals must be avoided. Tin was chosen as the trial liquid metal in the present work because of its convenient melting point (505 K) and its low vapor pressure, reported to be less than 10^{-6} mm Hg up to 1000 K¹⁶. Cyclobutene is convenient as the collider molecule because of its low critical threshold ($E_0 \sim 32 \text{ kcal mole}^{-1}$ ¹⁷) for isomerization to 1,3-butadiene, a reaction which is close to the low-pressure fall-off region at the reactant pressures in this study. An added advantage of the use of cyclobutene is that previous studies that utilized pyrex, silica, and crystalline gold surfaces revealed no catalytic behavior for cyclobutene isomerization. We emphasize that the present technique is a more stringent test of vibrational transfer at high energy levels (above E_0) than are conventional bulk energy accommodation studies.

Experimental

Cyclobutene was synthesized according to the procedure of Fadel, et al.¹⁸ Gas-liquid chromatographic and GCMS analysis showed that it contained less than 0.05% methylcyclobutene and butadiene as impurities. The sample was used without further purification. Tin metal (>99.8%) was obtained from Aldrich.

Two arrangements were used. In the studies above 520 K (Sets 1 and 2), the liquid tin sample was contained in a series 304 stainless steel well, 4 cm i.d. and 1.5 cm in depth. At the top of the well, which corresponded to the liquid surface, the stainless steel was flared for a 5 cm length and sealed

into the bottom of a spherical 5-l pyrex bulb via a stainless steel-pyrex seal. In order to maintain the stainless steel flare beyond the liquid surface at, or near, room temperature, the perimeter of the well just above the liquid surface was internally water cooled. Even at the highest surface temperature in this work, (775 K), the exposed surface temperature of the stainless steel flare never exceeded 320 K.

The liquid tin was stirred magnetically at a rate of ~ 120 rpm in most cases. The steel stirrer was constructed such that an 0.5 mm wide strip 2.2 cm in length, which was welded to the stirrer base, just broke the liquid surface. The base was designed such that, on rotation, liquid tin near the bottom of the well was driven towards the surface while surface tin was driven downward. This facilitated both temperature uniformity and also constant regeneration of a fresh metal surface.

The tin was heated by four cartridge heaters encased in the heavy bottom wall of the well. Temperature measurement was made by means of a chromel-alumel thermocouple in a thin-wall quartz well tube sealed into the top of the pyrex bulb that extended downward into the liquid metal; unfortunately the thermocouple well tube was positioned very close to the wall, which led to a correction described below.

For experiments below 520 K (Set 3), the vessel consisted of a 5-l quartz bulb, the lower part of which was sealed to a 4 cm i.d. flat bottom quartz well containing the solid sample. In these experiments, the volume of tin was only that necessary to cover the bottom of the well. Therefore, it was not stirred. The wall directly above the tin surface was wrapped with plastic tubing and water cooled. Temperature measurement was made with a 40 gauge

chromel-alumel thermocouple sealed into the vessel and immersed directly into the metal.

In a typical run, the reactor was pumped to 2×10^{-6} torr prior to introduction of the substrate. The typical substrate pressure was 3×10^{-4} torr although sample pressures in the range $(0.8-5) \times 10^{-4}$ torr were employed. The reaction gas was transferred for analysis after a predetermined run time. Product analysis was performed using gas liquid chromatography on a squalane SCOT column at 0 C. The detector was FID.

Results and Discussion

The reaction probability per collision, P_c , (i.e. the probability of transport of bulk molecules above E_0 in one collision with the surface) was obtained from the observed first order rate constant k , with use of the relation $P_c = 4kV/S\bar{c}$; where V is the reaction vessel volume, S is the surface area of the liquid tin, and $\bar{c}$ is the average translational velocity of the reactant molecules which have equilibrated with the cold wall prior to collision with the hot surface.

Initial data (Set 1) obtained between 525 K and 600 K gave values of P_c greater than those predicted by the theoretical strong collider curve for $E_0 = 32.4$ kcal, although lying parallel to that curve. This observation, coupled with good reproducibility of the data at these temperatures, suggested that the origin of this deviation from strong collider values was not surface catalysis. The extent of deviation of the low temperature experimental P_c values from the theoretical curve indicated that the measured temperature was in error by ~ 20 K. To leave sufficient room for the stirrer, it had been chosen to locate the thermocouple well next to the wall, the top of which was

internally water cooled. Therefore, the occurrence of a systematic error of this kind was not unreasonable. Moreover, in a later series of runs (Set 2) in the same apparatus between 600 K and 700 K, faster stirring and lower cooling water flow rate were used which resulted in more accurate temperature measurements. The apparent temperature discrepancy was then essentially eliminated. Substantial evidence that the correction as made was justified also was obtained in the final set of experiments (Set 3) at 505 K, using the quartz well reactor. In this case the thermocouple was in direct contact with the liquid, eliminating the temperature measurement error of the aforementioned type and P_c values in agreement with the theoretical strong collider curve were obtained. On the basis of this evidence, a correction was added to all observed temperatures for the first set of runs between 525 K and 775 K. In fact, if ascribed to an error in E_0 for the strong collider curve, this discrepancy would have simply led to the value 31 kcal mole⁻¹ rather than the literature value of 32 kcal mole⁻¹ itself a not-extreme possible error; neither possibility has any further substantive effect on the significance of the data.

The condition of the liquid metal surface proved to be a rather vexatious problem. When the solid was melted, the surface had a light coating of dross which could be effectively removed by skimming to give a clean, mirror-like surface. However, the surface was observed to develop a hazy, gray appearance with time. This was most likely due to the formation of tin (II) oxide¹⁹. Contamination of the surface by this material, which was accelerated at higher temperatures, necessitated frequent replacement of the metal sample. Somewhat surprisingly, however, no dependence of P_c upon surface quality was ever observed. This shows that despite the presence of particulate matter on the surface, which introduces roughness, the colliding molecules effectively still suffer only a single collision in an encounter with the hot surface.

Experiments on surface cleaning by doubling of the stirring rate were made. This had no observed effect on surface clarity. In addition, P_c values demonstrated no dependence on this experimental variable, nor did initial seasoning of the unstirred liquid have any apparent effect. Neither result is particularly surprising. Simple calculations of the kinetic theory collision rate, assuming a sticking coefficient of unity and a monolayer coverage of 10^{15} molecules cm^{-2} , indicates the formation of a monolayer in about 0.03 s.

A plot of P_c vs. temperature is given in Figure 1 along with the theoretical strong collider curve for a reaction threshold of 32.4 kcal mole⁻¹ (solid curve). The dashed curve represents a summary of all of the experimental data. The experimental scatter is somewhat disappointing and most likely due to residual temperature errors of the kind discussed above. However, this does not detract from the significant result that the surface behaves as a strong collider up to about 550 K. This is in agreement with the findings of Yuan and Rabinovitch for cyclobutene vibrational accommodation on seasoned gold wire and on plane gold surfaces¹². In fact, the behavior of the liquid metal surface, which is likely that of tin (II) oxide floating on liquid tin¹⁹, is most reminiscent of the oxygen-processed gold surfaces investigated in their work. Analogous to some published results of Somorjai and coworkers^{20,21}, Yuan and Rabinovitch found that preadsorbed oxygen enhances the rate of energy transfer. An equally important result of this study is that points just above and below the freezing point of tin show that there is no discontinuity in the vibrational accommodation coefficient. This finding conforms with a related observation by Thomas and Petersen for translational accommodation of He on liquid and solid potassium surfaces¹⁵.

It is useful to note that measurement of the extent of thermal accommodation by determination of transport to energy levels above the critical threshold, E_0 , is a more severe test than measurement of the average energy of the final distribution as a whole as in conventional accommodation coefficient measurements. In the latter case, relatively large deviations from the strong collider behavior for transitions at higher energy may not greatly alter the average energy of the distribution²². However, the same behavior can significantly alter the population above E_0 and thus lead to more sensitive determinations of the collisional efficiency¹⁰. After a single collision the new population vector N^1 , is given by $N^1 = PN^0$, where N^0 is the Boltzman population vector corresponding to the original temperature and P is the transition probability matrix. Strong collider behavior, observed in this work below ~ 550 K, corresponds to complete vibrational accommodation. N^1 is therefore simply a Boltzman distribution characteristic of the surface temperature. The value of α_{vib} deduced above ~ 550 K depends on the form of P , which is presently uncertain. Several studies in this laboratory have sought the correct analytical form of P using both the $m=1$ and $m>1$ variants of the VEM method^{22,23}. If P is partitioned at E_0 , one can describe four submatrices.

$$P = \begin{pmatrix} P_I & P_{II} \\ P_{III} & P_{IV} \end{pmatrix}$$

P_I contains the elements for transition probabilities between levels below E_0 . Such transitions are the important contributors to N^1 , and thus to the average energy change ΔE_{av} . Knowledge of P_I is most helpful in deducing values of α_{vib} . The $m > 1$ variant of the VEM method yields this information, but not directly. The form of P_{II} , which describes transition probabilities from levels below E_0 to levels above E_0 , is deduced from $m = 1$ studies. Once P_{II} is characterized, P_{III} is given by detailed balance. The two variants of the VEM method are complementary in determining the correct analytical form of P ^{22,23}.

Our results indicate that the liquid surface behaves much like the solid metal surface with respect to energy transfer by polyatomic gas molecules. The structure of liquid tin has been studied by x-ray diffraction by Furukawa, et al.¹⁹. The liquid tin was reduced in the presence of hydrogen at 775 K to provide an oxide-free surface. Intensity patterns were found to exhibit more structure than is found for "simple" liquids such as potassium and sodium, where atomic packing is observed. Their analysis of the liquid at 232 C indicated that there are at least two distinct internuclear separations present, in a ratio of about 19:1. The predominant of these, at 3.27 Å, does not greatly differ from those found in β -tin. This view is supported by NMR²⁴ and optical reflectivity²⁵ studies which indicate that the electronic states of the liquid do not greatly differ from those of β -tin.

It is of interest to extend the present work to include other appropriate liquid metals. These include Ga (m.p. = 29.8 C), currently under investigation, and In (m.p. = 157 C), both of which have conveniently low vapor pressures up to at least 900 K¹⁶. The technique used in this work could be useful in future studies of this type with some modifications. The stirring rate used here was not rapid enough to clean the surface. However, in principle, sufficiently clean surfaces might be attainable by rapid stirring at relatively high pressures ($\sim 10^{-6}$ torr), thus obviating the need for ultra-high vacuum techniques.

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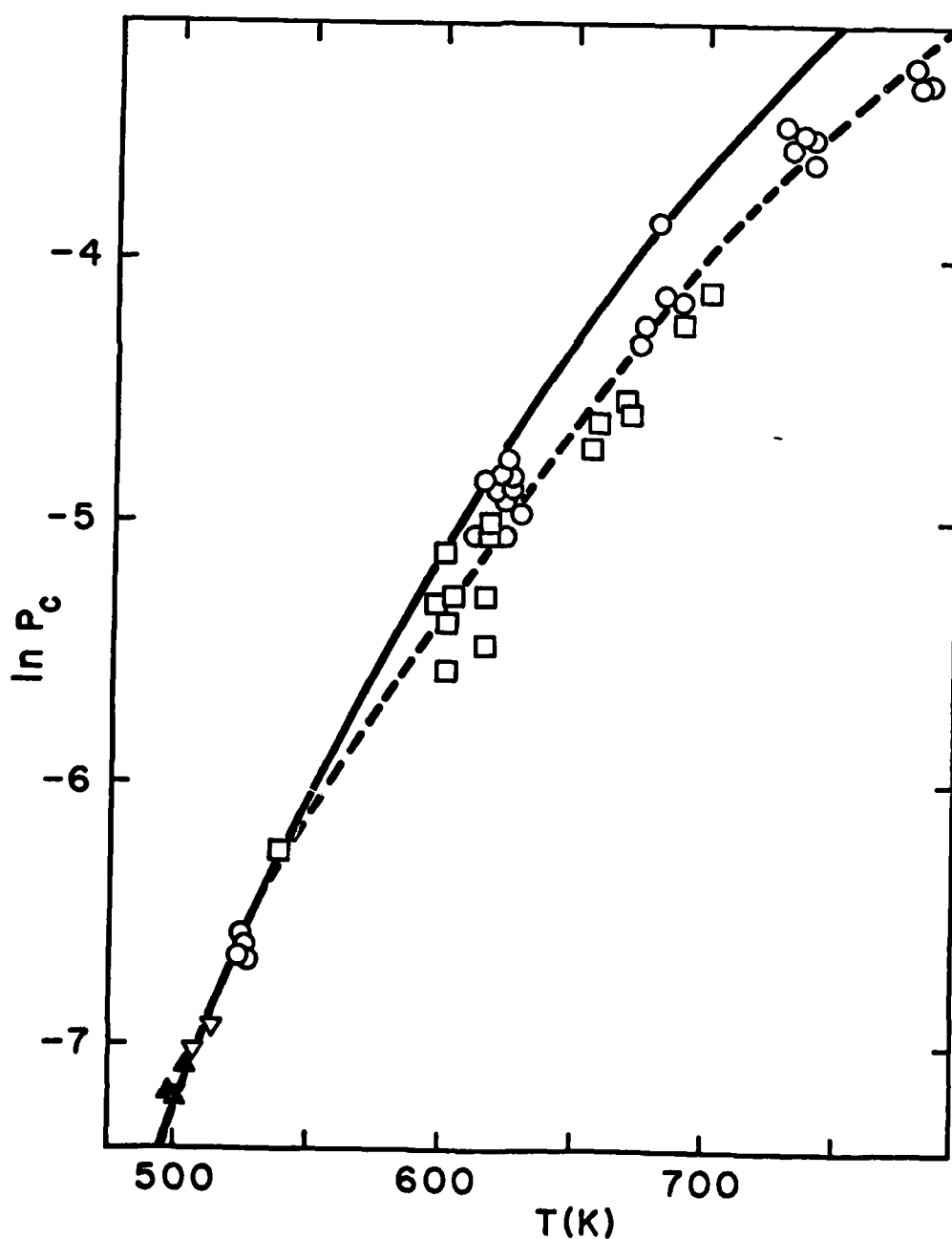
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Figure Captions

Figure 1. Plot of Experimental Values of P_c vs T ;
 0 and $\square$ represent values from data sets 1 and 2, respectively,
 see text). $\blacktriangle$ and ∇ are values from set 3, below and above the
 freezing point of tin (505 K), respectively; --- is a fit of the
 experimental data; — is the theoretical strong collider curve
 ($E_0 = 32.4 \text{ kcal mole}^{-1}$).



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