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EFFECTS OF WALL COATINGS AND TEMPERATURE ON HYDROGEN ATOM SURFACE RECOMBINATION

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16. Abstract

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SUMMARY

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INTRODUCTION

The Lewis Research Center has recently begun a program to stabilize hydrogen atoms. One of the initial experiments was an attempt to confirm Hess' results (ref. 1). For this experiment hydrogen atoms were produced at a short distance from the region where these atoms were to be condensed at 1 K and stored in the presence of a strong magnetic field. Because it is important to maintain the hydrogen atoms at a high concentration during their travel to the stabilization region, information on the effect of cold wall surfaces on hydrogen atom surface recombination would be desirable. In a recent survey paper (ref. 2) some surface coatings and materials are described as being efficient inhibitors of hydrogen atom surface recombination at 300° K, but there is very little information of this nature at lower temperatures. The purpose of this investigation is to compare the efficiencies of these various surface coatings and materials (ref. 2) at lower temperatures. For this study a flow-discharge, mass-spectrometric technique is used. Relative hydrogen atom concentration is monitored directly using a quadrupole mass spectrometer. Comparison of the efficiencies of the various surface coatings and materials in reducing hydrogen atom surface recombination is reported for the temperature range from room temperature (298 K) to liquid nitrogen temperature (77 K). For one of the surface coatings the temperature was reduced further to liquid neon temperature (27 K).

EXPERIMENT AND PROCEDURES

Apparatus

The essential part of the experimental apparatus is shown in figures 1 and 2. Figure 1 shows the apparatus with a straight 1.27-centimeter (1/2-in.) outside diameter Pyrex tube for the preliminary experiment. In this study we compared the efficiency of an uncoated tube with one coated with ortho-phosphoric acid (H_3PO_4) at room temperature. The rotameter and the high vacuum needle valve are used to meter the appropriate flow into the Pyrex tube. Pressures inside the Pyrex tube can be measured by means of the tap leading to a calibrated Autovac vacuum gage. The microwave cavity (Broida type) is self-shielding and efficient in the production of hydrogen atoms. This cavity can be moved axially almost the full length of the Pyrex tube so that we could study the hydrogen decay as a function of distance from the discharge. A 125-watt diathermy microwave generator was used to energize the cavity. The mass spectrometer inlet adapter is made of Pyrex and is used to couple the Pyrex tube with the quadrupole mass spectrometer. One end of the adapter is fitted with an outer ground glass joint (19/38 standard taper) in order to accept the inner ground joint on one end of the Pyrex tube. The opposite end of the adapter is drawn down to a 0.127-millimeter (0.005-in.) diameter leak hole for mass-spectrometer sampling. This leak hole is located about 6.4 millimeters (1/4 in.) from the ionizing electron beam. The total length of the adapter from this leak hole to the rim of the ground joint is approximately 26.4 centimeters ($10\frac{3}{8}$ in.). Because the inlet adapter is attached more or less permanently to the mass spectrometer, it was coated with ortho-phosphoric acid to minimize atom surface recombination during all the runs. Thus, the various surface coatings were applied only to the portion of the apparatus that can be easily disconnected from the adapter, such as the flow tube shown in figure 1.

Figure 2 shows the 1.27-centimeter (1/2-in.) diameter U-tube that can be easily substituted for the straight tube (fig. 1). This U-tube was used in the second part of this work to determine the efficiencies of various surface coatings toward reducing hydrogen atom surface recombination as a function of temperature. The U-tube was designed so that it could be easily immersed in a standard Dewar flask.

Surface Coatings and Materials

The various surface coatings and materials used in this report are

(1) Concentrated and dilute (~ 20 percent) ortho-phosphoric acid (H_3PO_4; ref. 3)
(2) Teflon (polytetrafluoroethylene) suspension (duPont Teflon Clear Finish 852-201; ref. 4)
(3) Dri-film, SC 77 (General Electric Co.; a mixture of dimethyldichlorosilane and trichlorosilane; ref. 5)
(4) Metaphosphoric acid ($(HPO_3)_n$; ref. 6)
(5) Uncoated Pyrex tube
(6) 1.27-centimeter (1/2-in.) Teflon tube (straight tube)

Coating Procedures

All the Pyrex tubes were cleaned with hot chromic acid cleaning solution, followed by rinsing with distilled water before coating with the appropriate surface materials. For the concentrated and dilute ortho-phosphoric acid coatings, the wet tube was rinsed in the acid and air dried before being installed in the flow system. For the Teflon suspension coating, the dried tube was coated using the technique described in reference 4. The metaphosphoric acid coating, $(HPO_3)_n$, was fused to the Pyrex surface by heating. The Dri-film was heated, and its vapor permitted to condense inside the clean Pyrex surface.

Gases

The hydrogen atom source was a premixed tanked gas mixture of 2-percent hydrogen in helium. This mixture was made up using commercially available hydrogen with a stated minimum purity of 99.95 percent and helium with a tested purity of 99.99 percent. A very dilute hydrogen gas mixture was used in order that the background hydrogen atom peak (contributed from molecular H_2) would be relatively small. For the present study the background hydrogen atom peak during the off discharge condition was only about 1 to 2 percent of the molecular hydrogen peak. A second reason is that the percentage of molecular hydrogen dissociated in a microwave discharge is much greater for a dilute mixture than for pure hydrogen. For our experimental conditions, a 2 percent hydrogen in helium mixture will have 30 to 35 percent of its hydrogen dissociated, whereas 100 percent hydrogen will have only 10 to 13 percent of its molecules dissociated.

Monitoring of Hydrogen Atom Concentration

As previously mentioned a quadrupole mass spectrometer was used to monitor the hydrogen (H) atom concentration in the flow apparatus shown in figures 1 and 2. For the present study it was sufficient to compare the effectiveness of the various surface coatings and materials on the basis of a relative H atom concentration measurement. This relative H atom measurement was expressed as the ratio of the peak heights for H atoms at mass 1 and molecular hydrogen (H_2) at mass 2, H_1/H_2. By measuring the ratio of the H_1 peak to the H_2 peak, any drift in the mass-spectrometer sensitivity during a run will be cancelled. From such data we could also compute the amount of H_2 dissociated when the discharge is turned on and off.

Figure 3 shows a partial mass spectrum for a 2 percent hydrogen in helium mixture during the on and off discharge conditions. The mass spectrometer was adjusted to display only masses 1 and 2 for atomic hydrogen and molecular hydrogen peaks, respectively. Each scan is separated by a dashed line. The first two scans for the on condition show the H atom peak (1) and the molecular hydrogen peak (2). The next two scans for the off conditions show that the H_1 peak has almost disappeared and the H_2 peak is now considerably larger. The last two scans for the on condition again show that the H_1 peak has reappeared and the H_2 peak is again reduced. For the preceding data

$$\frac{H_1}{H_2} = \frac{H_1(\text{on}) - H_1(\text{off})}{H_2(\text{on})} = \frac{1.4 - 0.1}{5.5} = 0.24 \tag{1}$$

In addition, the amount of H_2 dissociated by the discharge can be computed from figure 3 as follows:

$$H_2 \text{ dissociated} = \frac{8.9 - 5.5}{8.9} \times 100 = 38 \text{ percent} \tag{2}$$

where 8.9 units is the peak height of the H_2 peak during the off condition and 5.5 units is the peak height of the H_2 peak during the on condition. From the percent H_2 dissociated one can calculate an upper limit H_1/H_2 value, which, in this case, is equal to

$$\frac{2 \times 38}{100 - 38} = \frac{76}{62} = 1.2$$

It is now interesting to compare the measured H_1/H_2 peak ratio value of 0.24 with the calculated upper limit value of 1.2. This suggests that the quadrupole mass spectrometer used in the present study discriminates severely against H atoms.

RESULTS AND DISCUSSION

In the results and discussion that follow it should be emphasized that the H_1/H_2 ratios are computed from equation (1) using the actual peak heights observed. No attempt has been made to correct the peak heights for any differences in mass spectrometer sensitivies between atomic and molecular hydrogen. For the purposes of these studies, such relative values are entirely adequate.

Effect of Distance from Discharge for an Uncoated Pyrex Tube and H_3PO_4 Coated Tube at Room Temperature

The first experiment was conducted using the apparatus shown in figure 1. The efficiency of a coated tube (concentrated H_3PO_4) toward inhibiting H atom surface recombination as a function of distance was compared with an uncoated Pyrex tube. The distance D (see fig. 1) is measured from the downstream end of the Broida cavity to the leak hole of the adapter. For the coated surface, the Pyrex tube was rinsed with a concentrated ortho-phosphoric acid solution and air dried. These results are shown in figure 4, plotted as measured mass-spectrometer peak ratio H_1/H_2 against distance D. For this test the 2 percent hydrogen in helium flow was 0.8 cubic centimeter per second (at standard temperature and pressure (STP)), and pressure was 1.2 torr. The effectiveness of the ortho-phosphoric acid coating is such that even at 100 centimeters the H_1/H_2 value remains unchanged; for the uncoated Pyrex surface at 100 centimeters the H_1/H_2 value has been reduced by about 50 percent. During the course of these tests we observed that for the uncoated Pyrex tube a conditioning process occurred. When the discharge is first turned on, the measured H_1/H_2 value is low. After an interval of about 10 to 15 minutes with the discharge on, this measured H_1/H_2 value can increase as much as 10 times the initial H_1/H_2 value. No conditioning was required for the ortho-phosphoric acid coated tube.

Effect of Temperature on Efficiencies of Various Surface Coatings and Materials

The next series of experiments were conducted to compare temperature effects on the efficiencies of various surface coatings and materials toward inhibiting H atom surface recombination. For these tests the U-tube shown in figure 2 was used. The approximate linear distance from the downstream end of the cavity to the leak is 80 centimeters ($31\frac{1}{2}$ in.). Gas flow of the hydrogen-helium mixture was maintained at 0.5 cubic centimeter per second (STP) and the pressure at 0.7 torr. All the results are listed in table I. At room temperature (298 K) the concentrated phosphoric acid and the Teflon suspension were the most efficient. Of the two, the Teflon suspension is preferred because it is a relatively clean, stable, low-vapor-pressure coating. The results at 298 K can be compared with the work of reference 7 in which the coatings were ranked according to their efficiencies as follows: Teflon suspension > phosphoric acid > quartz rinsed with HNO_3 ≈ Pyrex rinsed with HNO_3 ≈ Pyrex rinsed with HF >> Dri-film, where > means "better than" and ≈ means "approximately equivalent to."

Our results are in general agreement with these findings. Two of the coatings, the Teflon suspension and Dri-film, required a conditioning period before the maximum H_1/H_2 ratio was obtained. This was also true for the uncoated Pyrex tube as noted previously. During the conditioning the surface is exposed to a flow of hydrogen atoms. During the first few minutes of exposure, most of the H atoms are "lost" on the surface (the H_1/H_2 ratio is only 0.02 to 0.03). The H_1/H_2 ratio then increases gradually until an equilibrium value is reached, usually after about 20 minutes exposure. Presumably, this occurs when a uniform, inert surface is produced by the action of hydrogen atoms on the coatings. The need for a conditioning period could explain the unfavorable results for Dri-film reported in reference 9. Because the test surface was not exposed to a direct flow of hydrogen atoms, it seems reasonable that the surface may never have been completely conditioned. This is consistent with the fact that investigators studying hydrogen masers (ref. 5) have reported Dri-film to be superior to most other coatings in its ability to inhibit H atom recombination.

As the temperature is reduced to liquid-nitrogen temperature (77 K), none of the coatings or materials were very efficient at preventing recombination. In the case of the ortho-phosphoric acid coating the temperature was further reduced to liquid neon temperature (27 K), which resulted in a further decrease in H_1/H_2 value.

Effects of Temperature on Efficiency of Uncoated Pyrex Tube Over Extended Temperature Range

In an attempt to compare the present data for an uncoated Pyrex tube with both theoretical and some experimental results described in the literature (refs. 8 and 9), the experiments dealing with the uncoated Pyrex tube were repeated including additional data points at temperatures of 111, 133, and 475 K. These data are shown in figure 5 plotted as measured H_1/H_2 values against temperature. For this plot a maximum or transition temperature of approximately 240 K was obtained. This temperature can be compared with a transition temperature of approximately 111 K predicted for such a surface (refs. 8 and 9). This transition temperature marks the point at which the mechanism for H atom surface recombination changes from one that is independent of atom concentration (first order kinetics) to one that is directly dependent on atom concentration (second order kinetics). Part of the difference between the observed and predicted temperatures may have been our use of a flowing system to obtain the data. Thus, there could have been additional factors, such as residence time, affecting the measured H_1/H_2 values.

One way to estimate this temperature effect on the present data for a flowing system is to consider that this effect is the combined result of the frequency of atomic collisions with the wall surface ω and of residence time τ. Since $\omega \propto T^{1/2}$ and $\tau \propto (1/T)$, measured H_1/H_2 values should be $\propto T^{1/2}$. Results of such an analysis are shown in figure 6. The solid curve is the experimental data shown in the previous figure. The dot-dashed curve shows H_1/H_2 values obtained by normalizing the room temperature H_1/H_2 value according to the equation

$$\left(\frac{H_1}{H_2}\right)_T = \left(\frac{H_1}{H_2}\right)_{300\text{ K}} \left(\frac{T}{300}\right)^{1/2} \tag{3}$$

where T is the absolute surface temperature. This equation gives the change in H_1/H_2 that would be expected if residence time and collision frequency were the only factors affecting recombination. The dashed curve is the experimental data from which this temperature effect has been removed using the following equation:

$$\left(\frac{H_1}{H_2}\right)_{T,\text{corr}} = \left(\frac{H_1}{H_2}\right)_{T,\text{exp}} + \left[\left(\frac{H_1}{H_2}\right)_{300\text{ K}} - \left(\frac{H_1}{H_2}\right)_{T,\text{eq. (3)}}\right] \tag{4}$$

It is apparent that this correction has shifted the transition temperature about 50 K closer to the theoretically predicted transition temperature of 111 K. It should be mentioned, however, that in experiments such as those to stabilize atomic hydrogen, a flowing gas is involved. Thus, the pertinent information for such experiments are the flow results.

Additional Efficiency Data for Teflon and Concentrated Ortho-Phosphoric Acid Coatings

For the two most efficient surface coatings, the Teflon suspension and the concentrated ortho-phosphoric acid, the measurements were repeated to include an additional data point at temperature of 133 K. These results are presented in figure 7. Also included are the data for the uncoated Pyrex tube shown as a dashed line. Apparently, as temperature is lowered coated surfaces are changed either chemically or physically, or both, in such a manner that they can no longer inhibit H atom surface recombination.

Calibration of Mass Spectrometer Hydrogen Atom Concentration Measurement

Previously, we found that the measured H_1/H_2 values differed considerably from the calculated upper limit H_1/H_2 values based on hydrogen dissociation. This suggested that the quadrupole mass spectrometer must discriminate severely against H atoms. To check this we calibrated the mass spectrometer for H atom concentration measurement at room temperature using the gas titration reaction of H atoms with nitrosyl chloride (NOCl) described in reference 10. For this calibration the inlet adapter (figs. 1 and 2) was modified as shown in figure 8. The main gas flow (2 percent H_2 - He) enters at point A, and the microwave discharge is at point B. The titrating gas NOCl enters the inlet adapter by the Pyrex feed tube. One end is joined to an appropriate pressure-drop type of metering system. The other part of the feed tube, which extends the entire length of the adapter, is a 3.18-millimeter (1/8-in.)diameter Pyrex tube. Nitrosyl chloride (further purified by fractional distillation from its stated purity of 97 percent) is fed into the reactor by means of three small holes placed on the sides of the small tube near its end. The end of this tube is about 1.9 centimeters (3/4-in.) from the leak hole into the mass spectrometer.

In this calibration technique the H atoms are titrated on a one-for-one basis with NOCl via the following reaction:

$$H + NOCl \rightarrow NO + HCl$$

By measuring the pressure drop in a calibrated volume containing an NOCl - argon mixture, the flow of NOCl required to produce an observed drop in the H_1 peak can be computed. Combining this with the measured H_2 flow yields the actual H_1/H_2 ratio. In general, for these experimental conditions, the measured H_1/H_2 ratio values were about 0.20, and the H_1 sensitivity to H_2 sensitivity ratio was about 6. (Sensitivity here is defined as being the amount of H_1 or H_2 in cm^3/sec, STP, necessary to produce one unit of peak height.) Thus, the actual H_1/H_2 ratio would be $6 \times 0.20 = 1.2$. This value of 1.2 can be compared with an average calculated upper limit value of 1.0, which was obtained on the basis of the percent of H_2 dissociated as measured mass spectrometrically during the calibration. This agreement in the two numbers is reasonably good if one considers the difficulties in making direct measurements of H_1, the percent of H_2 dissociated, and the relatively large errors involved in calculating this upper limit H_1/H_2 value.

CONCLUDING REMARKS

The results of the calibration described in the preceding section indicated that, at room temperature, the actual H_1/H_2 ratio is very close to the upper limit H_1/H_2 value calculated from the percent molecular hydrogen dissociated. On this basis, we conclude that there is only a small percentage of hydrogen atoms lost in traveling from the discharge region, through the inlet system, and into the ion source of the mass spectrometer. Furthermore, even when the U-tube (fig. 2) was cooled to liquid-nitrogen temperature, the H_1/H_2 ratio was actually about six times larger than the measured value, that is, approximately 0.18 instead of approximately 0.03. Thus, with regard to the hydrogen atom stabilization experiment, the feasibility of producing the hydrogen atoms some distance from the low-temperature stabilization region appears more promising than the earlier results indicated.

Finally, on the basis of this work we recommend the Teflon coating for inhibiting surface recombination of hydrogen atoms for most applications. This coating is easy to apply, very inert, and has a low vapor pressure. Moreover, it inhibits recombination as well as any of the coatings studied.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, November 29, 1972,
501-24.

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TABLE I. - EFFECT OF TEMPERATURE ON H ATOM SURFACE RECOMBINATION FOR VARIOUS SURFACE COATINGS

Surface coatings on 1.27 cm (1/2 in.) Pyrex U-tube	Measured mass spectrometer peak ratio,[a] H_1/H_2			
	Room temperature (298 K)	Dry ice temperature (195 K)	Liquid-nitrogen temperature (77 K)	Liquid neon temperature (27 K)
Concentrated ortho-phosphoric acid	0.20	0.10	0.03	0.01
Teflon suspension	0.02 to 0.20	.16	.01	----
20 Percent ortho-phosphoric acid	.15	.15	.03	----
Dri-film	0.03 to 0.09	----	.03	----
No coating (Pyrex tube)	0.01 to 0.09	.06	$<.01$	----
Meta-phosphoric acid, $(HPO_3)_n$	.02	----	-----	----
Teflon (1.27-cm (1/2-in.) Teflon tube)	.02	----	.004	----

[a] The H_1/H_2 values shown were computed from eq. (1) using the observed peak heights for H_1 and H_2. Absolute H_1/H_2 values require a knowledge of the sensitivity of the mass spectrometer toward H_1 and H_2.

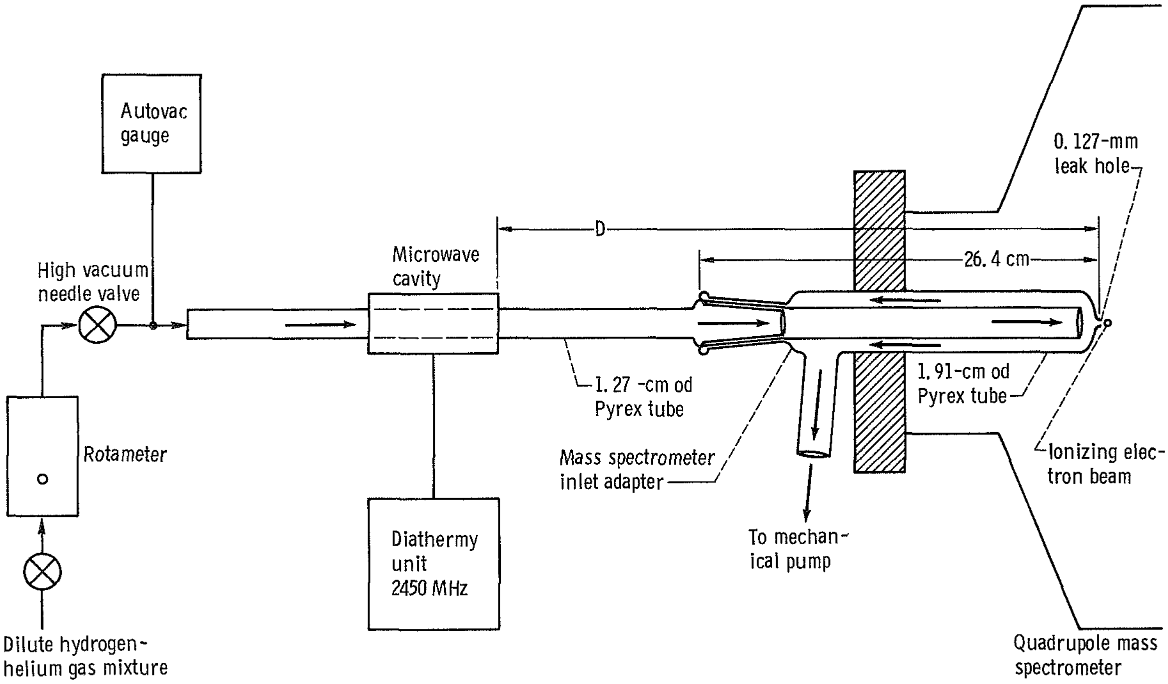

Figure 1. - Flow discharge-mass spectrometer apparatus using a straight Pyrex tube.

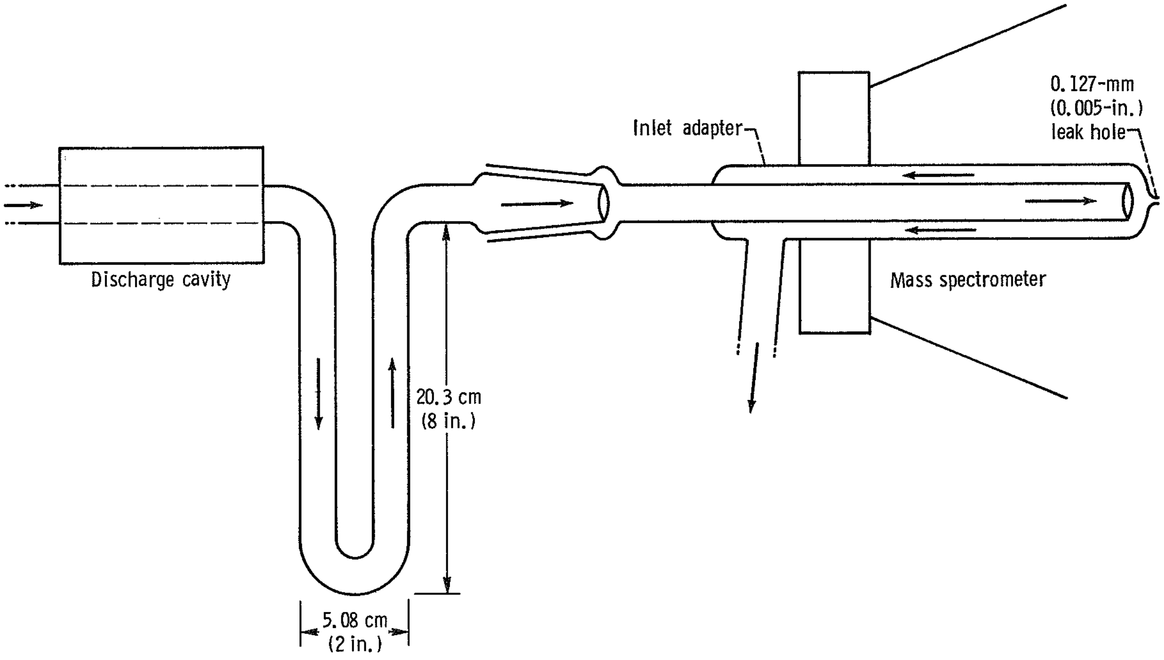

Figure 2. - Pyrex U-tube.

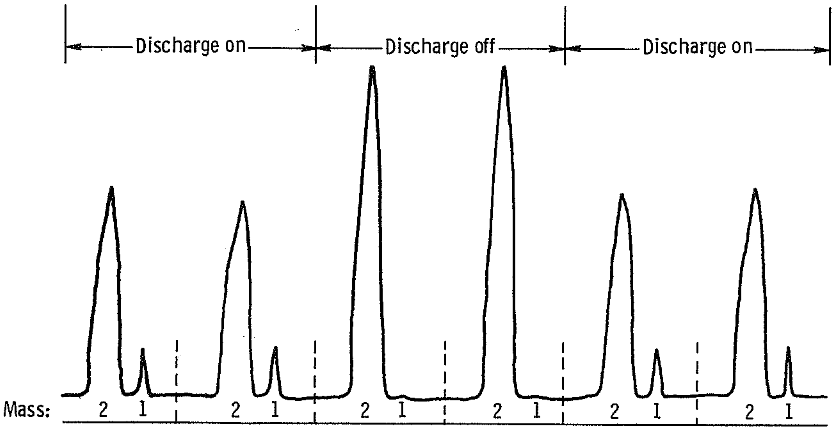

Figure 3. - Mass spectrum for atomic hydrogen (mass 1) and molecular hydrogen (mass 2) during on and off discharge conditions.

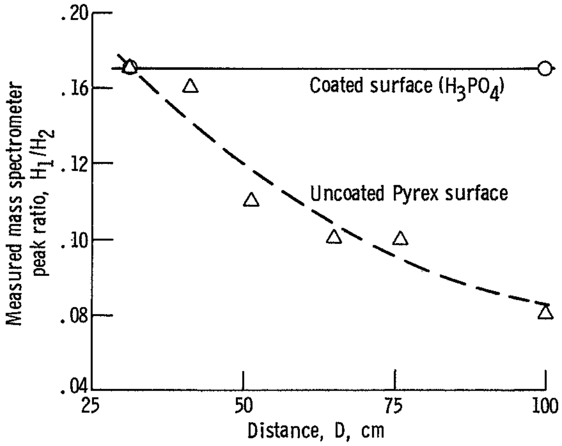

Figure 4. - Effect of distance between discharge and leak hole on mass spectrometer peak ratios for coated (concentrated H_3PO_4) and uncoated Pyrex surface. Temperature, 298 K.

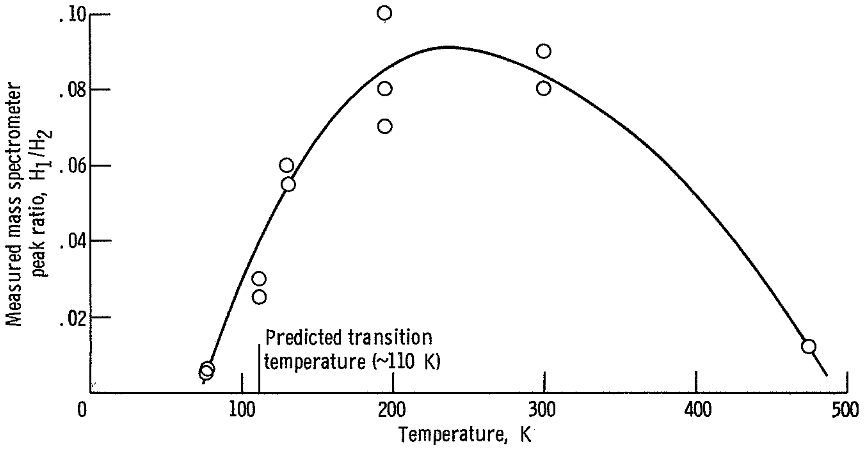

Figure 5. - Effect of temperature on measured mass spectrometer peak ratios for uncoated Pyrex tube.

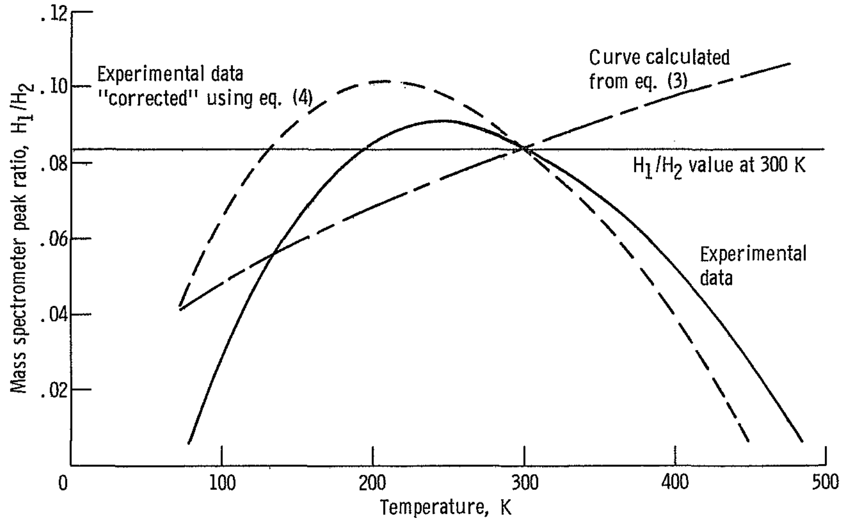

Figure 6. - Calculated effects of temperature on measured mass spectrometer peak ratios for uncoated Pyrex tube.

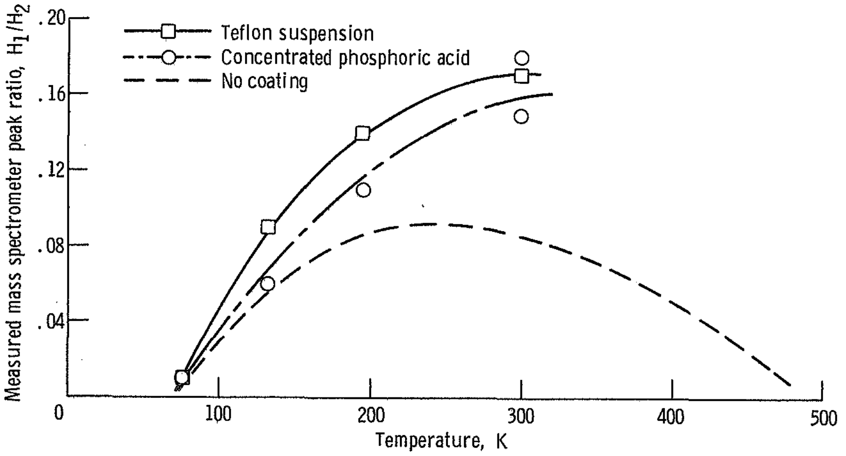

Figure 7. - Effect of temperature on measured mass spectrometer peak ratios for several surfaces.

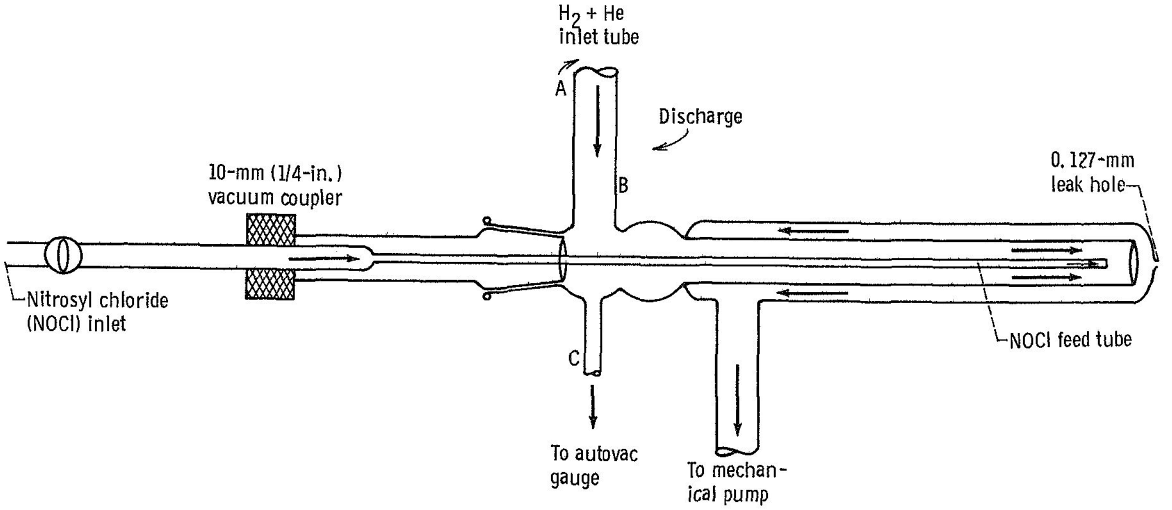

Figure 8. - Mass spectrometer hydrogen atom calibration apparatus.

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