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EXPERIMENTAL STUDIES OF LOW TEMPERATURE SURFACE  
INTERACTIONS OF ATOMIC HY. (U) SMITHSONIAN  
ASTROPHYSICAL OBSERVATORY CAMBRIDGE MA  
R F VESSOT ET AL. JAN 85 N00014-77-C-0777

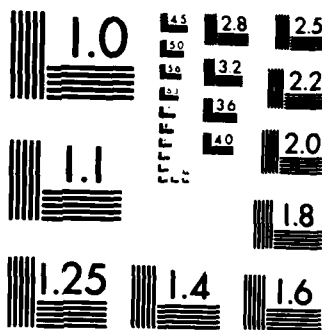
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EXPERIMENTAL STUDIES OF LOW TEMPERATURE SURFACE INTERACTIONS  
OF ATOMIC HYDROGEN USING MASER TECHNIQUE

ONR CONTRACT N00014-77-C-0777

FINAL REPORT

For the period 30 September 1977 through 30 September 1984

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Co-Investigator  
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JANUARY 1985

Prepared for  
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Astrophysical Observatory  
Cambridge, MA 02138

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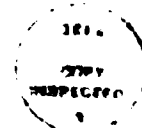
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EXPERIMENTAL STUDIES OF LOW TEMPERATURE SURFACE INTERACTIONS  
OF ATOMIC HYDROGEN USING MASER TECHNIQUE

FINAL REPORT

1. INTRODUCTION

The data and results presented in this report are the culmination of an ongoing study of the low temperature operation of the atomic Hydrogen masers performed by the Smithsonian Astrophysical Observatory on this contract over the past 7 years. The history of the contract is presented in Table 1. Interim progress reports (approximately every 1-1½ years) describing work performed were submitted in the proposal for continuing the work on cold maser operation and results have been published. [1-3] Funding from other agencies (Jet Propulsion Laboratory (JPL), National Aeronautics and Space Administration (NASA), Marshall Space Flight Center (MSFC), and Smithsonian Institution (SI) were integrated with the ONR support to build a test setup for performing this research. A number of modifications to the test setup were incorporated to overcome problems encountered during early observations. These include improvements developed with other support (i.e. in-vacuum cold RF dissociator developed on NASA-MSFC grant).

The principal objectives of the SAO cold maser research programs are:

1. To understand the behavior of the atomic hydrogen wall collision process and find better wall coating materials or processes.
2. To develop hydrogen masers of improved stability taking advantage of the following low temperature properties:
  - a) Lower thermal noise per unit bandwidth (kT). Both the signal-to-noise ratio of the output signal and the intrinsic stability limit vary as  $T^{3/2}$

- b) Smaller spin exchange cross section for atomic hydrogen. At 4<sup>deg</sup>K this cross section is about 200 times smaller than at room temperature. For a given rate of spin exchange quenching at room temperature we can obtain 200 times greater power output and thus reduce the signal-to-noise in the output signal.
- c) Slower speed of the atoms (proportional to  $T^{d/2}$ ), which reduces the wall collision rate.
- d) Better mechanical and thermal stability of materials at low temperatures. Superconducting magnetic shields can also be used at sufficiently low temperatures.

The work we report here is aimed principally at understanding more about the interaction of hydrogen atoms with wall coatings of fluorinated ethylene propylene (Dupont Teflon FEP-120 co polymer) and of carbon tetrafluoride (CF<sub>4</sub>). The principal measured quantity in these experiments is the "wall shift" of the maser's output frequency. To relate the present data to the study of cold Teflon surfaces made by Michel DeSaintfusien, [4] we calculated the wall shift per atomic collision from the measured wall frequency shift. As will be seen later, this assumes that the wall surface area is smooth on a molecular scale.

We calculate the average phase shift  $\Delta\Phi_g$  per geometrical collision determined from the dimensions of the storage bulb:

$$\Delta\Phi_g(T) = \frac{2\pi\Delta\nu_w(T)}{\nu_c(T)} \quad (1)$$

where  $\nu_c$  is the rate of atomic collisions with the storage bulb wall and  $\Delta\nu_w(T)$  is the frequency shift owing to wall collisions.  $\nu_w$  is obtained by measuring the maser output frequency  $\nu_{out}$  and correcting it for second-order Doppler shift, second order magnetic field dependence, and cavity resonator frequency offset pulling as follows:

$$\nu_{\text{out}} = \nu_0 + \frac{3kT\nu_0}{2mc^2} - 2752H^2 - (f_c - \nu_0) \frac{Q_c}{Q_l} + \Delta\nu_w(T) \quad (2)$$

Here  $\nu_0$  is the unperturbed hyperfine separation frequency of atomic hydrogen, [5]  $H$  is the static magnetic field strength in the storage volume,  $m$  is the hydrogen atom mass,  $f_c$  is the cavity resonance frequency,  $Q_c$  is the cavity  $Q$ , and  $Q_l$  is the atomic line  $Q$ .

Each measurement required a determination of the output frequency at several known cavity frequency settings. The output frequency data were fit to a straight line as a function of cavity frequency  $f_c$ , and the frequency  $\Delta\nu_w$  corresponding to  $f_c = \nu_0$  was calculated in order to eliminate the effects of resonator pulling. From the average velocity of the atoms,  $\bar{v} = (8\frac{kT}{\pi m})^{1/2}$ , and the geometrical surface-to-volume ratio of the storage volume  $A_g/V_g$ , we determine the average collision rate,  $\bar{\nu}_c$ .

$$\bar{\nu}_c = \frac{\text{average velocity}}{\text{mean free geometrical distance}} = \frac{\bar{v}}{\frac{4V_g d}{A_g d}} = \frac{\bar{v}}{4} \frac{A_g}{V_g} \quad (3)$$

Following Hardy and Morrow [6] we assume a model for the hydrogen wall surface interaction where some fraction of the wall collisions result in momentary binding of the atom in a potential well characterized by energy  $E_b$ . While in the well the atoms have the properties of a two-dimensional gas. The time spent in this state is very much smaller than the mean time between collisions,  $\bar{\tau}_c$ . The ratio of  $\bar{\tau}_s$ , the time spent bound to the wall, to  $\bar{\tau}_b$ , the time between these events, is given by [6]

$$r = \frac{\bar{\tau}_s}{\bar{\tau}_b} = \Lambda \frac{A}{V} e^{-1E_b/kT-1} \quad (4)$$

where  $\Lambda = \frac{h}{(2\pi mkT)^{1/2}}$  is the thermal de Broglie wavelength.

Since an atom does not bind to the wall on each impact, the time  $\bar{\tau}_b$  may involve many collisions. The probability of binding per collision is

represented by the fraction  $\alpha = \frac{\bar{r}_c}{\bar{r}_b}$ . During a binding collision the phase of the hyperfine interaction is retarded an amount

$$\overline{\Delta\phi} = 2\pi\bar{r}_s\Delta_s$$

where  $\Delta_s$  is the frequency change of the atom while in the bound state. The wall collision frequency shift is

$$\Delta\nu_w = \frac{\nu_c\alpha}{2\pi} \overline{\Delta\phi} = \frac{h}{(2\pi m)^{1/2}} \frac{A}{V} e^{-1E_b/kT} 1\Delta_s \quad (5)$$

and the average phase shift per collision is

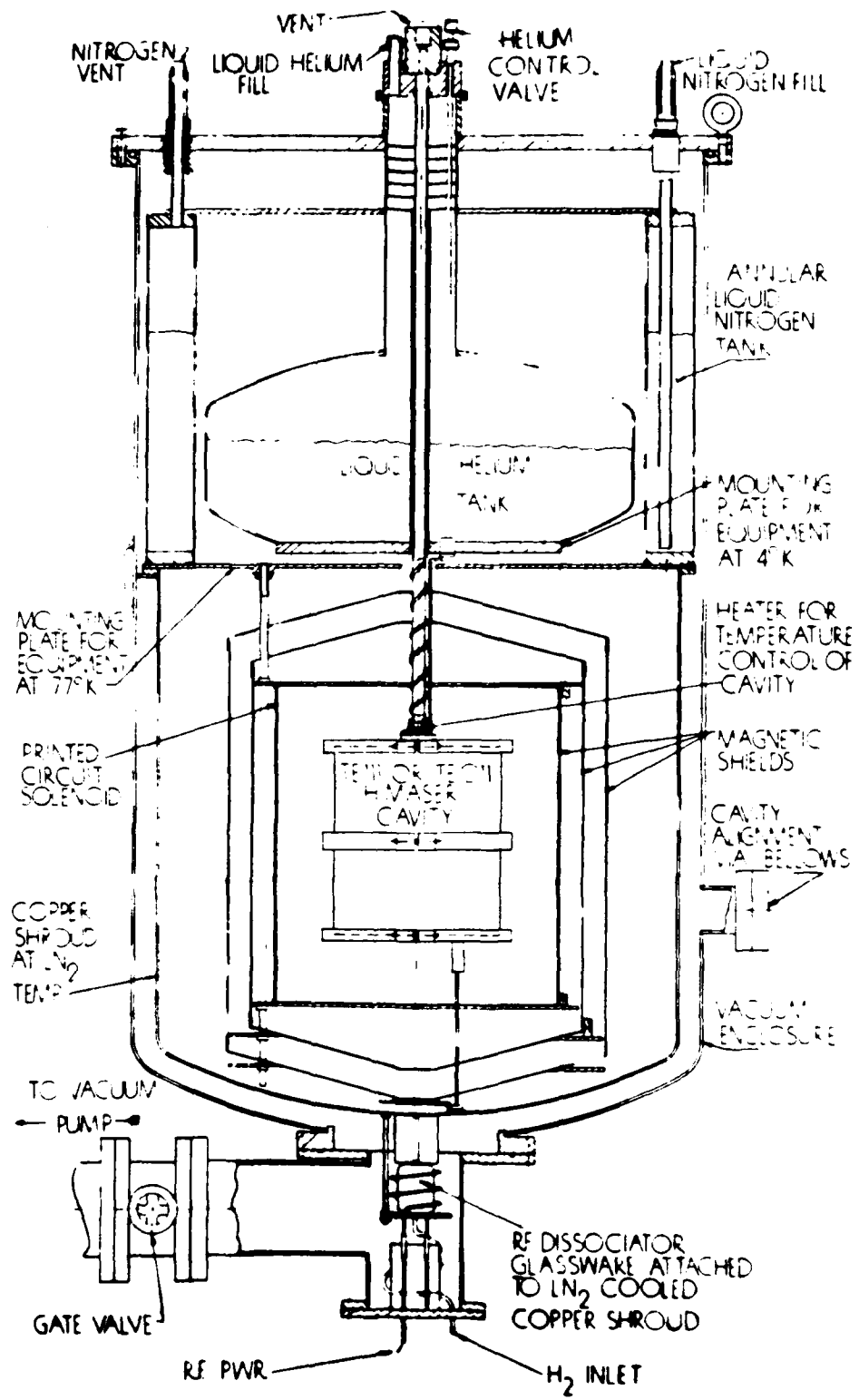
$$\Delta\phi_g(T) = \frac{V_g}{A_g} \frac{A}{V} \frac{2\pi h}{kT} e^{-1E_b/kT} 1\Delta_s \quad (6)$$

We note in this expression that  $A/V$  is the actual area to volume ratio and  $A_g/V_g$  is the geometrically determined area to volume ratio.  $A$  is the effective surface area of the storage region coating, which may be microscopically rough, while  $A_g$  is the area of the storage region assuming perfectly smooth walls. Because the surface roughness has negligible effect on the storage volume, for practical purposes  $V = V_g$ .

## 2. EXPERIMENTAL PROCEDURE

The cryostat shown in Fig. 1 was equipped with a  $TE_{111}$ -mode cavity<sup>[7]</sup> whose interior surface was coated with FEP-120 Teflon and that had a Teflon FEP septum 0.25 mm thick. The atomic hydrogen source is cooled by contact with the copper shroud, and operates at about 80°K. Liquid helium admitted via a control valve into the sample holder cools the cavity.

The maser was operated in the range of 78°K to 50°K with the bare Teflon surface. After completing the data runs for bare Teflon, the system was stabilized at about 60°K and gaseous  $CF_4$  was beamed toward the cavity entrance



S.A.C. COLD MASER  
 FIGURE 1

aperture by a nozzle located in the shadow of one of the pole tips of the hexapole magnet. The system pressure rose to about  $10^{-3}$  torr and was kept there for about 4 minutes by the flow of  $CF_4$ .

The wallshift was measured and the coating process was repeated a second and third time. No further shift in frequency was observed. We conclude that we had completely and thoroughly coated the surface using this procedure.

### 3. EXPERIMENTAL RESULTS

Figure 2 shows the average phase shift per collision based on the geometrical surface to volume ratio of the storage volume. For Teflon we include the entire data set of 1983 and 1984 and the data point at 77.4K made by DeSaintfusien in 1976. [4] The carbon tetrafluoride data were obtained within 30 minutes of the time of injection of  $CF_4$ . The system was then cooled at a rate of  $5^\circ K$  per hour to  $52^\circ K$ , was allowed to equilibrate at  $52^\circ K$  for about 40 minutes, and was then again cooled at  $5^\circ K$  per hour until the cavity reached  $45^\circ K$ . We paused for equilibration for 20 minutes before cooling steadily to  $33.6^\circ K$  at a rate of about  $4^\circ K$  per hour. Frequency measurements were made with the continuously oscillating maser. Measurements at each temperature were made for three or more cavity frequency settings. Each data set was made within a two-minute time interval to minimize errors owing to the constant slow cooling of the cavity.

We found that the temperature gradient between the sample holder and the cavity caused magnetic quenching, probably due to thermoelectric currents. To keep the maser oscillating the field coil current was raised to produce a magnetic field as high as  $27 \times 10^{-3}$  gauss. The usual field setting was  $12.8 \times 10^{-3}$  gauss; when gradients were small, data were successfully taken at  $2 \times 10^{-3}$  gauss.

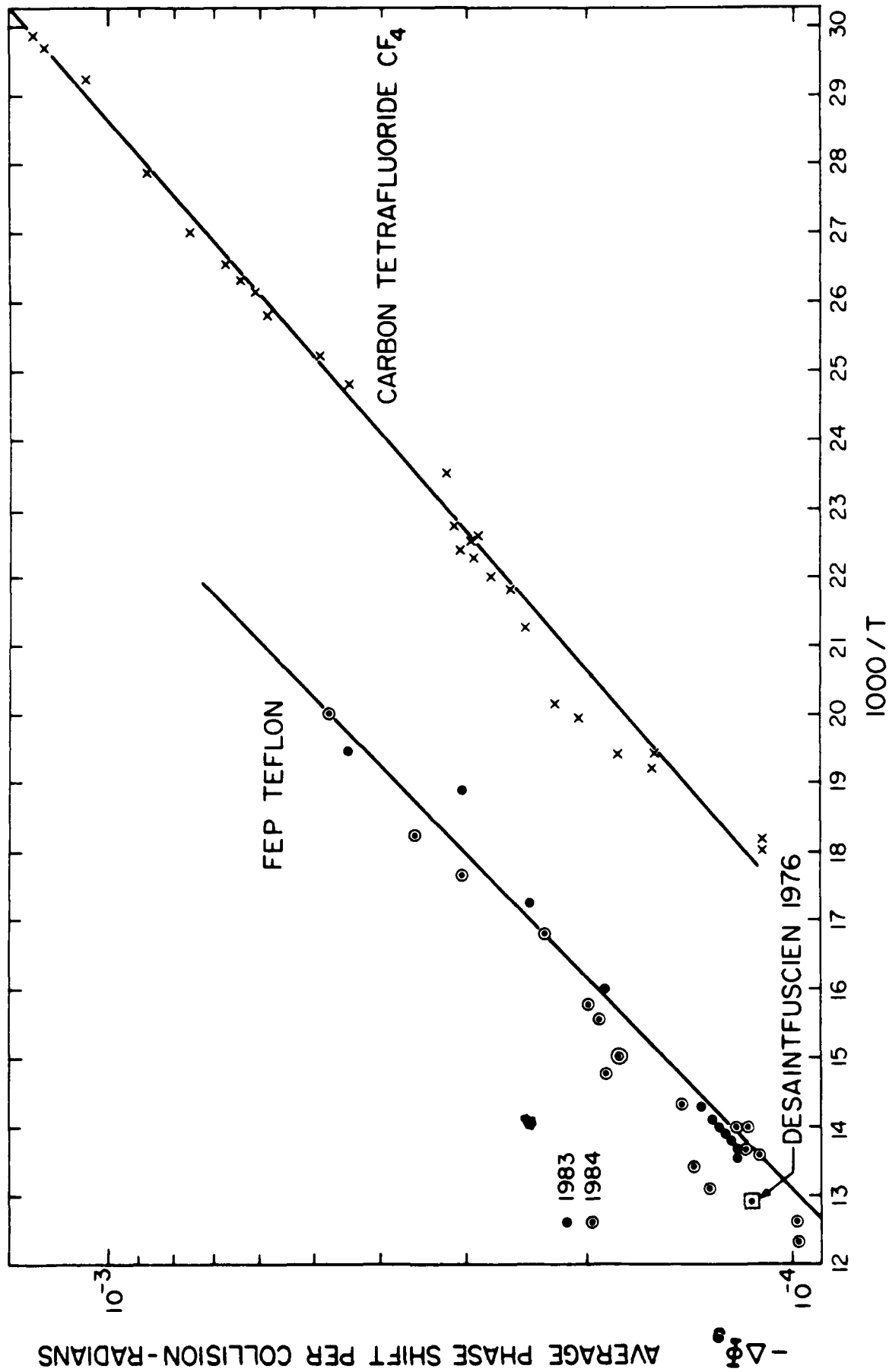


FIGURE 2

The plot of  $-T\Delta\Phi_g(T)$  vs  $1000/T$  shown in Fig. 3 was calculated from measurements taken between noon and 7 P.M. on October 15, 1984. The straight line is a least squares fit to  $-T\Delta\Phi_g(T) = 0.42 \times 10^{-3} e^{154.6/T}$ . Measurements made at 43°K, 44°K and 46°K on October 16 fell into the spread of the October 16 data set. Measurements made on October 17 at 53°K and 55°K and on October 18 at 60°K lie well below expected values. We can perhaps conclude that at the higher temperatures the  $CF_4$  surface sublimates away. The jagged edges and prominences of this surface would disappear first, leaving a progressively smoother surface until all of the  $CF_4$  had been pumped away. This behavior upon warming the  $CF_4$  surfaces has been observed on at least three previous occasions. To date, we have not been able to find reliable values of the  $CF_4$  vapor pressure for temperatures below its melting point of 89°K. From the pressure behavior observed when we warm up the system, we believe that the vapor pressure is significant, perhaps on the order of  $10^{-5}$  torr, at temperatures around 60°K and that at higher temperatures the surface is sufficiently volatile to change its structure significantly in an interval of several days.

We have recalculated  $-T\Delta\Phi_g(T)$  vs  $1000/T$  using DeSaintfuscién's data, our 1983 and 1984 Teflon data, and the October 15 data of Fig. 3. The results are plotted in Fig. 4.

From this plot we conclude that the interaction energy of atomic hydrogen on carbon tetrafluoride is 154.6°K, and that of FEP-120 Teflon is 143.4°K. These differ by only about 8 percent, and lead us to the conclusion that the surface interaction of hydrogen colliding with a fluorine atom bound to carbon does not significantly depend on the internal carbon structure. However, we observe a significant difference between the magnitudes of the phase shift per collision for Teflon and  $CF_4$ . There is about three times less phase shift per collision for frozen-in-place  $CF_4$  than for Teflon.

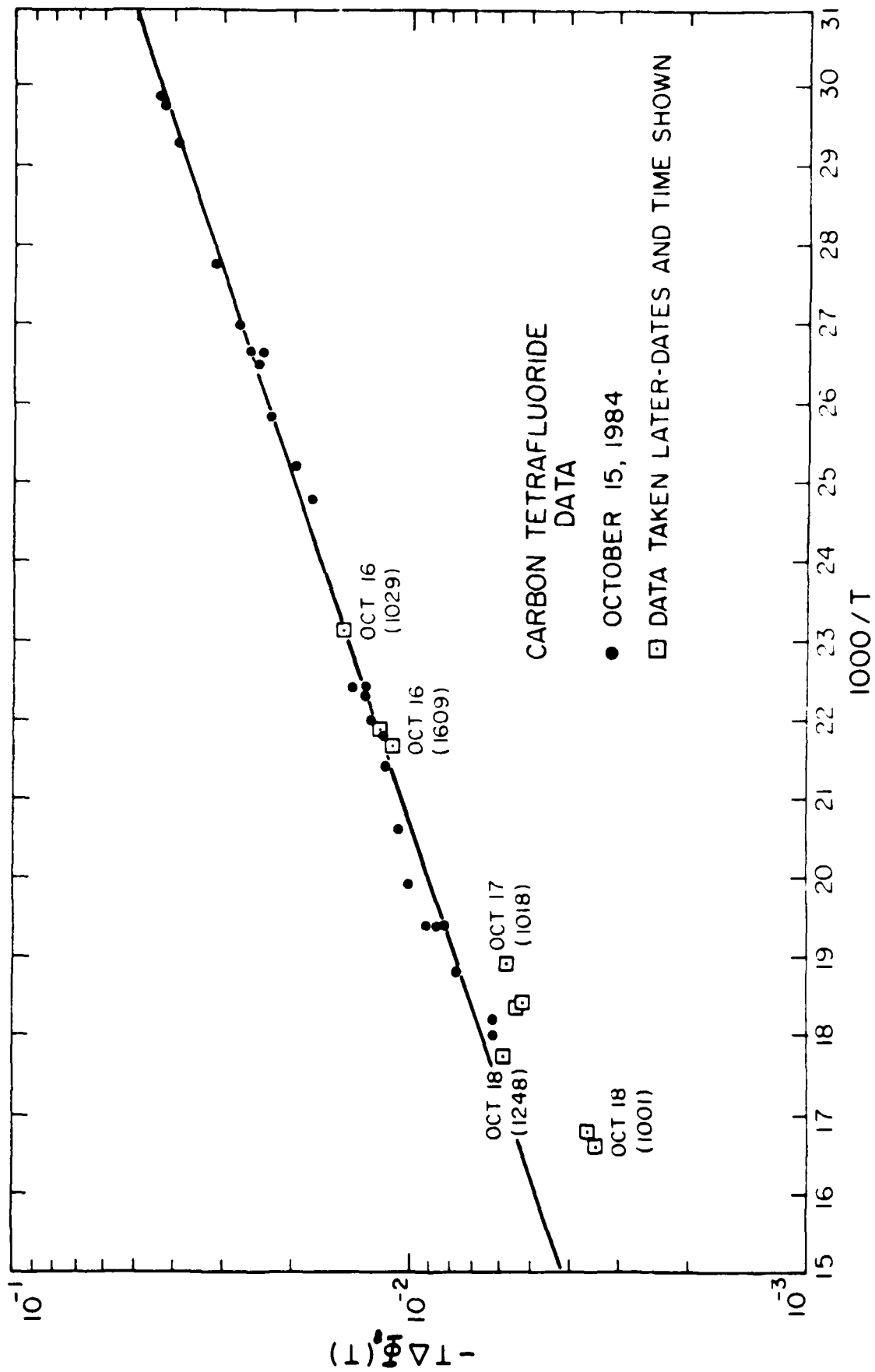


FIGURE 3

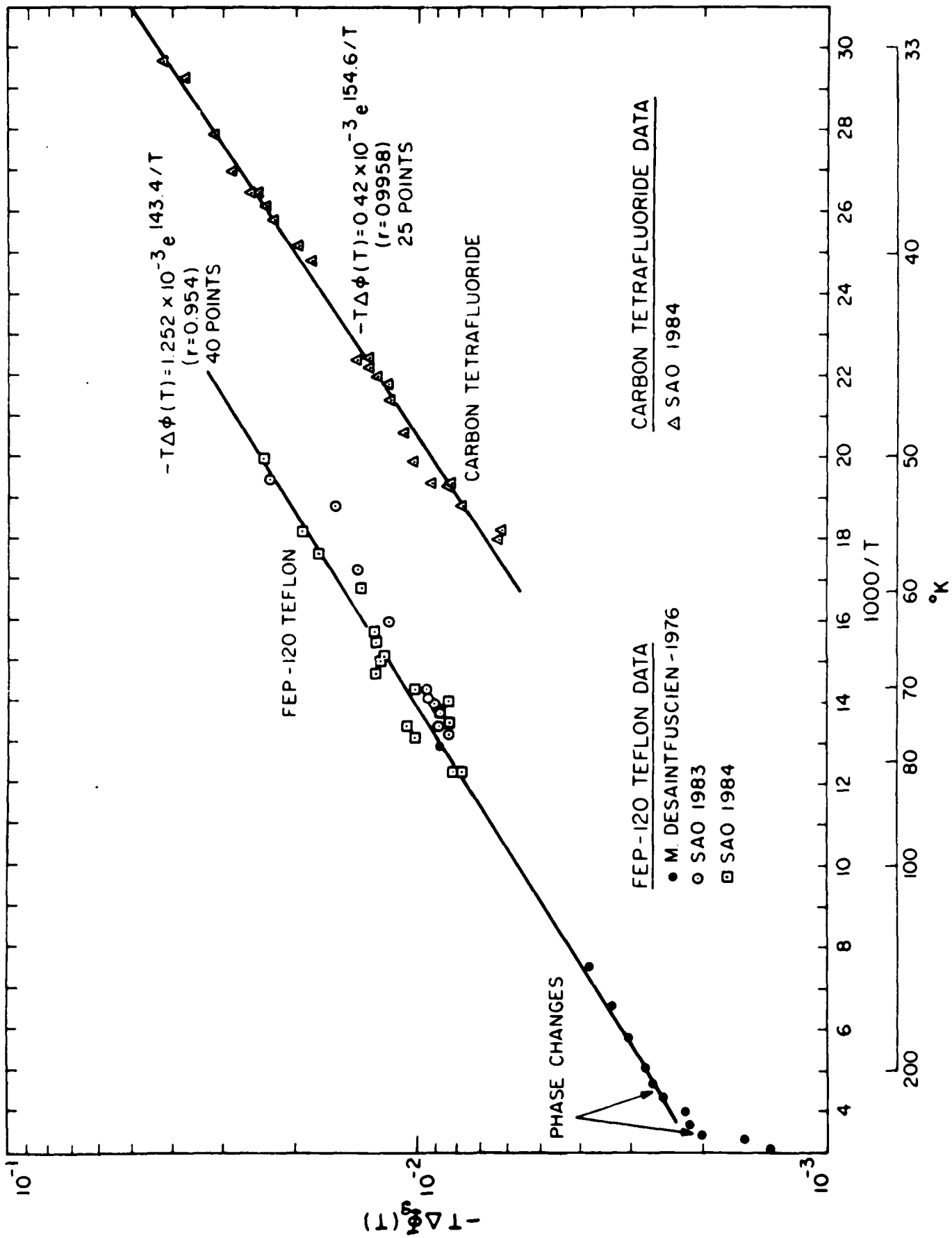


FIGURE 4

From Equation 6 we see that the terms that govern this shift are the actual surface area and the hyperfine frequency shift of the hydrogen atom while in the bound state,  $\Delta_S$ . Calculations of the hyperfine shift<sup>[8]</sup> have been made with reasonable agreement with experiments using the assumption that the inter-atomic hyperfine shift is proportional to the interaction potential. When the hyperfine shift is calculated for collisions with an aggregate of atoms, such as a Teflon molecule, the actual structure of the surface must be modeled in the same way. This micro model of the collision process where hydrogen is considered to interact with, say, 10 fluorine atoms bound to a carbon core must be contrasted with a macro model where the surface is irregular on a much greater scale involving, say, tens of millions of atoms. The situation involving the latter scale can be represented in terms of surface area-to-volume and related to free molecular flow collision processes. In either case it is a question of the actual physical configuration of the fluorine atoms encountered by the infringing hydrogen atom.

We must remember that our plots of  $\log [T\Delta\Phi_g(T)]$  vs  $T^{-1}$  are normalized to a collision rate estimated from geometrical surface and volume. We have used equation 6 to show the connection between DeSaintfusciens's data and ours; we see from this equation that, in principle,  $\Delta\Phi_g(t)$  is independent of  $A/V$ .

A more realistic way to plot the data is to use equation 5, plotting  $\log[T^{3/2}\Delta\nu_w]$  vs  $T^{-1}$ . This has the same slope as the  $T\Delta\Phi_g(T)$  data, and the prefactor contains  $A/V$  and  $\Delta_S$ .

We conclude from the close agreement between DeSaintfusciens's data and ours that the FEP-120 Teflon dispersion has produced a fairly reproducible surface in these two applications.

From the similarity in the experimental terms shown in the FEP-120 and  $CF_4$  data plotted in Figure 4, we see that the energy of interaction is very similar for these two surfaces. The question of the three-fold to four-fold disparity in the wall shift remains.

Fluorine, the most electronegative of all elements, forms extremely stable carbon compounds (fluorocarbons) whose molecular attraction (as well as repulsion) are entirely governed by fluorine. The long chains of the  $CF_2$  (tetrafluor-ethylene) homopolymer (TFE Teflon) or the shorter branched chains of the FEP co-polymer (fluorinated ethylene-propylene) have substantially similar structure in that they can be either crystalline or largely amorphous, depending on their condition after melting, i.e. whether slowly annealed or quenched. There are substantial differences in wallshift between these two physical states<sup>[9,8,10]</sup>, and these differences have been associated with the surface structure. The crystalline surface has the larger wallshift owing to the growth of platelets that produce a rougher surface on a macroscopic scale and to microscopic textural effects of the platelets when pictured as a stack of cordwood where the Teflon chains (logs) of various lengths protrude from the sides of the generally well organized stack.

The homopolymer is obtained in the form of a water dispersion of particles about 0.22 micro meters average diameter stabilized with a soap-like substance to prevent coagulation. The co-polymer has 0.1 micro meter sized particles and is also available in a water dispersion. These dispersions are cast on the surface to be coated, the water evaporated, and the particles sintered or melted together to form a film. The homopolymer has a high viscosity of about  $10^{11}$  poises in the melted state. Its consistency is "more like a frame than a liquid".<sup>[8]</sup> The co-polymer has melt viscosity of about  $10^6$  poises and flows more easily. In 1978, the surface of thin films, say 0.002 inches in thickness, were

found to be porous from tests made at the U.S. Naval Research Laboratory using electron scattering and low energy electron diffraction techniques.<sup>[11]</sup> This has led us to increase the coating thickness of our films from about 1 milligram per  $\text{cm}^3$  to well over 3 milligrams per  $\text{cm}^3$  by applying successive coatings of the Dupont FEP-120 co-polymer. Whether or not the density of the films has been improved is open to question. What has probably happened under these multiply coated conditions is that we have piled up more and more platelets but still have a porous and, to the hydrogen atom, macroscopically rough surface.

The threefold decrease in apparent surface area by freezing  $\text{CF}_4$  probably results from the smoothing of the Teflon surface by a build-up of  $\text{CF}_4$  "frost" on the interior of the storage chamber. That this "frosted"  $\text{CF}_4$  surface itself is not as smooth as it could be is strongly evidenced by the further reduction in wallshift when the surface is warmed to  $60^\circ\text{K}$ . Here we envision a further smoothing of the surface either by melting, or more probably, by sublimation where the pointed peaks are first to go, leaving a smoother surface that lasts as long as there is  $\text{CF}_4$  available.

The fact that the Teflon surface is porous and that the collision rate apparently can be reduced by a factor of as much as four times leads us to reconsider the present status of wall coatings created by fusing granules of Teflon applied from a water dispersion. This is a difficult process to control and has a great deal of variability.

The importance of wall coatings is crucial to the hydrogen maser storage technique, where the linewidth of the oscillator is limited by the wall relaxation process. Remarkably little has been done to improve wall coatings since the invention of the maser by Kleppner, Goldenberg and Ramsey in 1969. With support from the Office of Naval Research included as a small part of a

contract with the U.S. Naval Research Laboratory (N00014-71-A-0110-0003) an attempt was made in 1975-1976 by our group at SAO to obtain bulbs coated with Teflon polymerized in place using  $C_2F_4$  monomer gas. The technique was developed by the Laboratoire Suisse de Recherches Hologeres (L.S.R.H.), Neuchatel, Switzerland in 1975 and patented in the U.S.A., France, Japan and Germany. SAO engaged the L.S.R.H. to coat two quartz bulbs with a film of 1000Å to 3000Å thickness made from polymerized  $C_2F_4$ , using ultraviolet surface photo polymerization. This was to be done along with samples of glass and  $KB_r$  for analyses by pyrolysis to determine molecular weight, and other measurements to determine molecular structure and film thickness.

One bulb was received and tested in December 1975. Samples tested at L.S.R.H. by infrared spectroscopy showed proper Teflon polymer lines. The high temperature pyrolysis test showed no deterioration over  $3\frac{1}{2}$  hours at  $220^\circ C$ , indicating satisfactory molecular weight. As part of the contract, a complete report of the coating process from L.S.R.H. was received. Tests run at SAO on the bulbs initially showed excellent line Q results, but in less than a week the coating had failed.

While the maser was oscillating we measured the wallshift and found it to be in the range of normal values for P.T.F.E. Teflon. However, in a few days the line Q diminished steadily from a relatively high value of  $1.5 \times 10^9$  to well below  $1 \times 10^9$ . We took the maser apart to examine the bulb and found that its surface had deteriorated and was very easily wetted, and that a loose powder appeared on the surface of the water drop we had introduced. We concluded that the film on the bulbs was not the same as on the test samples. L.S.R.H. agreed to recoat the bulbs after testing their system with other bulbs. This work was not done and we were forced to conclude our contract with L.S.R.H. in August of 1977.

To the best of our knowledge, no further work on Teflon-like coatings for hydrogen masers has been done since this date. The present results with frozen  $\text{CF}_4$  suggest that another attempt at polymerizing  $\text{C}_2\text{F}_4$  in place would be worthwhile. The prospect of a possible fourfold improvement in storage time and developing a controllable means of coating hydrogen maser bulbs is a strong motivation for this renewed effort.

#### 4. A LOOK AT THE FUTURE

With the completion of this work on carbon tetrafluoride coatings, the Cold Maser Program at SAO has taken a new direction. We have joined forces with Professor Isaac F. Silvera's low temperature physics group at Harvard University's, Lyman Physics Laboratory, to work on an ultra-cold Hydrogen maser using wall coatings of superfluid helium 4 at  $0.4^\circ\text{K}$ . The dielectrically-loaded cavity resonator, made of sapphire, and the required magnetic field coils and low temperature shields are now at hand. This system will be tested at  $50^\circ\text{K}$  in the SAO cryostat using  $\text{CF}_4$  coatings to calibrate the fields and tune the cavity before being installed in the Lyman Laboratory dilution refrigerator for operation at  $0.4^\circ\text{K}$ . If time permits we plan to try operation at about  $9^\circ\text{K}$  with coatings of frozen neon. [12]

#### 5. ACKNOWLEDGEMENTS

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TABLE 1  
HISTORY  
ONR CONTRACT NO0014-77-C-0777  
STUDIES OF LOW TEMPERATURE OPERATION OF THE ATOMIC HYDROGEN MASER FREQUENCY STANDARD

DATE -----	ORIGINAL CONTRACT -----	FUNDING -----	CONTRACT VALUE -----
9/30/77	9/30/77 through 9/30/78 (Per SAO Proposal P731-8-77)	\$100,000	\$100,000
10/1/78	Mod 1 - No-cost Extension to 12/31/78	--	--
1/1/79	Mod 2 - No-cost Extension to 6/30/79	--	--
4/79	SAO Proposal P875-4-79: (a) Interim Progress Report (9/30/77 through 3/79) (b) Continuation of work 7/1/79 through 9/30/80	--   +92,166	
7/1/79	Mod 3 - Funds work through 6/30/81	+41,184	192,166
9/1/79	Mod 4 - Funding for low noise preamplifier	+10,930	203,096
6/1/80	Mod 5 - Funds balance of work to 6/30/81	+50,982	203,096
5/81	SAO Proposal P1088-5-81: (a) Interim Progress Report through 4/30/81 (b) Continuation of work through 6/30/82 (\$55,000)	--  --	--  --
7/1/81	Mod 6 - funds work through 6/30/82	+55,000	258,096
4/82	SAO Proposal P1179-4-82: (a) Interim Progress Report through 5/31/82 (b) Continuation of work through 6/30/83 (\$55,000)	--  --	--  --
7/1/82	Mod 7 - Funds work through 6/30/83 (Includes funds for summer high school student)	+57,421	315,517
4/83	SAO Proposal P1291-4-83: (a) Interim Progress Report through 5/31/83 (b) Continuation of work through 6/30/84 (\$64,984)	--  --	
7/1/83	Mod 8 - Funds work through 6/30/84 (Includes funds for summer high school student)	+67,586	383,103
7/1/84	Mod A01 - No-Cost Extension to 9/30/84	--	--

**END**

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