Storage Technique for Atomic Hydrogen

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lattice or exchange specific heat.\textsuperscript{3} Substances with very short spin-lattice relaxation times such as the free radicals $\alpha,\alpha$-diphenyl $\beta$-picryl hydrazyl (DPPH) and $\alpha,\gamma$-bisdiphenylene $\beta$-phenyl allyl (BDPA)\textsuperscript{4} require large amounts of microwave power for saturation. Under these conditions the usual CW saturation experiment may be measuring the lattice-bath bottleneck and not the true spin-lattice relaxation time. In the case of DPPH and BDPA it was comparatively simple to recognize that, in attempting to saturate the resonance line, we were heating the lattice. The linewidths of DPPH and BDPA have an inverse temperature dependence in the liquid helium temperature range.\textsuperscript{3} In Fig. 1 we see what happens to the linewidth of DPPH saturated at 77°C where the lattice specific heat is large. The broadening of the resonance line as the incident power is increased is that predicted by the Bloch equations. At 4.2°C, however, we see that the linewidth narrows as the microwave power is increased. This narrower line is the unsaturated line corresponding to a higher lattice temperature.

We have developed a modulated CW method in which the microwave power remains on for a time long compared to that necessary for the electrons to reach equilibrium with the microwaves. This characteristic time, obtained by solving the rate equations for a spin $\frac{1}{2}$ system is given by $T_1/(1+\gamma^2H^2T_1T_2)$. However, the duty cycle is such that the average power is not sufficient to appreciably heat the lattice. One can make $T_1$ measurements at several values of the duty cycle to see if heating effects have been eliminated.

An X-band bridge spectrometer is used, as shown in Fig. 2. One sets the magnetic field on resonance and then moves the klystron by means of the reflector voltage off magnetic and klystron resonance. The pulse height is then turned up to bring the system back on magnetic resonance. Since the AFC can not be used during this operation an extremely stable klystron is required. We use magnetic field modulation with phase-sensitive detection. The use of magnetic field modulation requires that the pulse rate be greater than the modulation frequency. Instead of field modulation, the phase-sensitive detector could operate at the frequency of the pulse-repetition rate. Saturation measurements are made in the usual CW manner. As the power incident on the cavity is increased by decreasing the attenuation in attenuator 1, the setting of 2 is increased so that the power falling on the crystal is constant (see Fig. 2).

We have applied this technique to measure the relaxation time of polycrystalline DPPH and BDPA. At 300 and 77°C both the usual CW and our modulated CW method give the same results, $T_1\sim5\times10^{-4}$ sec for DPPH and $\sim1\times10^{-7}$ sec for BDPA. Previous microwave measurements, both CW and high power pulse,\textsuperscript{4} on DPPH and BDPA at 1.5°C had given values of $T_1$ around $10^{-6}$ sec, while our modulated CW method gives $T_1\sim10^{-8}$ sec.

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\textsuperscript{*} Supported by the U. S. Air Force Office of Scientific Research.
\textsuperscript{\dagger} Presently employed with General Telephone and Electronics Laboratories, Inc., Palo Alto, California.
\textsuperscript{1} M. W. P. Strandberg, Phys. Rev. 110, 65 (1958).
\textsuperscript{3} J. P. Goldsborough, thesis, Stanford University.
\textsuperscript{5} James W. Meyer, thesis, Massachusetts Institute of Technology.

\textbf{Storage Technique for Atomic Hydrogen*}

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\textbf{WE} have found it possible to inhibit adsorption and surface recombination of atomic hydrogen by covering the container with a thin film of Teflon. The storage time of hydrogen has been measured with the atomic hydrogen maser,\textsuperscript{1} and times of up to 2.5 sec have so far been obtained, corresponding to $7.5\times10^{14}$ wall collisions. This is appreciably longer than possible with a saturated hydrocarbon coating, such as paraffin, or with a surface treated with Dri-Film\textsuperscript{2} (dimethyl dichlorosilane). The pressure of atomic hydrogen in the maser is of the order of $10^{-7}$ mm Hg.
although much higher pressures could be used before three-
body collisions become important. On the basis of hyperfine
frequency measurements we conclude that wall collisions
not leading to chemical reaction are essentially elastic with
an interaction time of the order of $10^{-18}$ sec.

The chief mechanism by which hydrogen is lost on colli-
sion with a saturated hydrocarbon surface is thought to be
chemical reaction with the surface. A typical reaction
would be of the form $\text{H} + \text{CH}_4 \rightarrow \text{H}_2 + \text{CH}_3$. The activation
energy for such a reaction is consistent with the relaxation
rate measured with the maser. The activation energy for
the corresponding reaction with a saturated fluorocarbon
is substantially higher and vastly longer lifetimes are there-
fore to be expected. The measured lifetime of $7.5 \times 10^4$
collisions is a lower limit, since the geometry of the bulb and
several other factors serve to limit the hyperfine radiation
lifetime of the atoms, which is the quantity measured.

The Teflon we have used is an aqueous suspension made
by duPont (Teflon Clear Finish 852-201). The container
to be coated, which in our case is a quartz bulb, is cleaned
with hot glass cleaning solution (sulfuric acid-dichromate),
rinsed several times, and drained. The same treatment is
satisfactory with a Pyrex vessel. The Teflon suspension is
strained and diluted with distilled water to about the con-
istency of skim milk. A thin coating is applied by swishing
the solution in the bulb and then draining it out. The coat-
ing is quickly dried by flowing dry $N_2$ through the bottle
and heating the outside with a hot air gun. The coating
must be fused, and this is accomplished by heating the bulb
locally with an acetylene torch. Teflon fuses to a clear,

nearly colorless film at about 400°C, and decomposes rapidly
only well above this temperature. The finished film is
cleaned gently with hot cleaning solution and rinsed.

If the coating is too thick it tends to turn brown and
crack; if it is too thin it is very fragile. The consistency of
the Teflon suspension is therefore critical.

The authors wish to thank G. B. Kistiakowsky for a very
helpful discussion on surface reactions.

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Fast-Response Temperature-Control Unit
for a Spectroscopic Sample Chamber

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Temperature-dependent changes in certain
protein solutions may be studied by spectroscopic
analysis. The temperature of 1.5 cc of liquid in a standard
optical cuvette may be controlled to within 1°C at any
setting above the ambient temperature, between 20 and
100°C, with the instrument described in this note. The

![Fig. 1. Heating coil in a 1-cm³ cuvette.](image)

The temperature increases at a 5°C/sec rate toward the de-
sired preset temperature value. The unit includes four
unorthodox features, as follows:

(1) The heating coil is directly immersed in the liquid.
As shown in Fig. 1, the container is a 1-cm³ cuvette. The
oval opening in the front view allows unobstructed passage
of an infrared beam for spectroscopic analysis of the liquid.
The heating coil is wound on a Kel-F form that is hollowed
out so that only a negligible portion of the coil is lost for
heating purposes. The wire criss-crosses along a path that
runs parallel to the oval opening. A rigid extension of the
Kel-F frame that is used to support lead wires as well as
various solution tubes has been omitted from the drawing.

To prevent chemical interactions with the liquid, a rela-
tively inert platinum wire (No. 40 bare, 0.003 in. diam.)
is used. The coil resistance is about 7.2 ohms. The small
stirrer at the bottom of the cuvette is magnetically rotated.

(2) The heating coil also acts as an arm of the temper-
ature-control Wheatstone bridge. The circuit is illustrated
in Fig. 2. Sixty-cycle heater power is supplied to $R_1$ through
$R_H$ and $C_R$ at the same time that a 2-v dc bridge supply
is applied through $L_F$. The latter blocks ac while $C_R$ blocks
dc.

(3) The reactive elements $L_R$ and $C_R$ form a partially
series-resonant circuit at 60 cps. This allows 30 w of heater
power to reach $R_1$ with a relatively small value for $C_R$.

(4) The 30 w of power are applied only to $R_2$, in contrast
with a balanced arrangement in which an additional 30 w
would be dissipated in $R_2$. A balanced circuit would also
require an isolation transformer because neither side of the
heater supply could be at ground. The relatively large ac
output of the circuit shown is removed by a 2-sec $R_FC_F$
time constant. The use of only 200 ohms for $R_F$ yields a
low source resistance for the amplifier. $C_F$ consists of two
low-voltage electrolytics in series, with reversed polarities,
to handle either dc polarity.

Before a temperature run, the experimenter places the
unit in a “stand-by” position, thereby returning the Variac