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Nuclear magnetism of solid hydrogen at reduced ortho concentration*

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The NMR of 200-ml STP of pure H_2, frozen in a cavity resonating at 310 MHz, was studied as the sample aged for 400 h while cooled by a dilution refrigerator. The frequency separation between points of maximum and minimum slope of the NMR line decreased linearly with time for $24 < t < 240$ h at the rate of 180 Hz/h. At the highest ortho concentrations and the lowest temperatures, the line shape corresponded closely to the doublet expected for interacting randomly oriented dipole pairs broadened by intermolecular dipolar interactions. However, as the ortho concentration decreased below 0.5 the line became more square than this model would predict. At several times during the run, the temperature was raised and again lowered. At the lowest temperature, the line changed with time through shapes very similar to those found with increasing temperature at an earlier time. Evidence from the integrated intensity of the line suggests that the ortho-para conversion during the run increased relative to the rate expected for a random lattice with interacting pairs. Studies of recovery from selective saturation showed that cross relaxation within the NMR line creates a spin temperature in about 5s.

INTRODUCTION

Studies of solid hydrogen at low temperatures have shown that a phase transition to an orientationally ordered structure occurs at a temperature T_c that depends on the fraction of molecules in the $J=1$ rotational state (ortho H_2).[1-5] In the ordered state, a sample containing randomly oriented microcrystals has a structured NMR absorption line with two main cusps resulting from the intramolecular dipole-dipole interactions. The frequency separation ν_{dd} between points of maximum and minimum slope of the doublet has been taken to be a measure of the orientational order of the molecules.

Ortho-para conversion is induced by the intermolecular dipolar interaction between pairs of ortho-molecules and the accepted rate is

$$\dot{X} = -kX^2, \tag{1}$$

where X is the fractional ortho-concentration and $k=0.019$ per hour.[6] Thus, by allowing the sample to age at low temperatures, one can examine the dependence of line shape and transition temperature on ortho concentration. Recent studies of solid H_2 at very low temperatures by means of NMR in high magnetic fields[7-9] have shown that the NMR line continues to be broad at fractional ortho concentrations X well below 0.56. Earlier studies had suggested[5] that the ordering would vanish at that "critical" concentration. As X decreased toward 0.56, ν_{dd} decreased rapidly followed by a slower rate down to $X=0.25$ (145 h age).[8]

The present paper describes similar measurements made as the sample aged at very low temperatures for 400 h, and X decreased to about 0.07. The magnet is now highly persistent and the line shapes are significantly more reproducible than previously. The present data show clearly that after 24 h, ν_{dd} decreased linearly with time at least to 240 h, and the NMR line continued to be broader than at 4 °K throughout the 400 h. However, the shape of the absorption line and the dependence of ν_{dd} on temperature at fixed ortho concentration suggest that the model of cooperative ordering of the rotational angular momenta of the ortho molecules may be invalid at the lower concentrations. We also present evidence that the clustering of the ortho molecules causes the ortho-para conversion to speed up relative to Eq. (1) when the sample is aged at $T < 0.1$ °K.

EXPERIMENTAL PROCEDURE

The H_2 was research-grade purity supplied in a glass bulb by Union Carbide, Linde Specialty Gas Division. Mass spectrographic batch analysis performed by Linde indicated 0.6-ppm O_2 contaminant. A 200-mL STP sample was condensed directly from the gas into the 310-MHz resonant cavity at 8 °K. As described previously,[10] the cavity was integral with the mixing chamber of a He^3-He^4 dilution refrigerator and had 15 cm^2 available surface area. The dilution refrigerator had a flow rate of 4×10^{-5} moles/s supplied by an Edwards 600 mechanical pump. Immediately following the condensation of the sample, the superconducting magnet was charged to 72 kG and left persistent throughout the run. The magnet had a homogeneity of about 2 parts in 10^6 over the dimensions of the sample. The observed drift of center frequency over the run corresponds to a mean decay rate in the persistent mode of 8×10^{-8}/h.

After 5 h at 4 °K, the refrigerator was started and the sample was allowed to age for most of the next 395 h at the lowest temperatures the refrigerator could maintain. The sample temperature early in the run was evaluated from three different indicators: (i) a 220-Ω Speer resistor mounted exterior to the sample chamber, (ii) the integrated intensity of the cw NMR lines, (iii) the relative heights of the cusps in the line shape predicted for randomly oriented dipole pairs. The three measurements agreed to better than ±5 mK. As shown in Fig. 1, the temperature achieved by the refrigerator dropped during the run from 110 to 58 mK as the heat from ortho-para conversion decreased. The solid line in Fig. 1 is a fit to the mixing chamber temperature assuming the ortho-para conversion obeys $\dot{X} = -kX^2$, and that the dilution refrigerator is ideal but has incoming heat that limits its coldest temperature to about 55 mK, as observed when empty. The step to lower temperature for $t < 310$ h results from a faster flow rate obtained by modifying the flow path.

On several occasions, in the course of aging, the cavity temperature was raised briefly to $T \sim 0.7$ °K to examine the line shape as a function of temperature. Each thermal cycle was performed slowly (~3 h) to insure thermal equilibrium. Between 340 and 360 h the temperature was raised and held at 1 °K, and between 364 and 388 h, the temperature was 4 °K. After each excursion to high temperatures, the wide line shape was recovered on lowering the temperatures again.

A Q meter (or Rollin circuit) was used for observing the NMR line shapes, while, for the spin-lattice relaxation time measurements, a uhf bridge was used to obtain rf levels large enough to cause saturation. The derivative of the NMR signal was observed using modulation of the magnetic field and coherent phase detection. The field modulation was kept less than 1 G peak-to-peak to avoid distortion of the line shape. A uhf generator (Marconi TF 1066 $B/6$) frequency modulated at 60 Hz was used without field modulation for oscilloscope display of the NMR absorption signal.

ORTHO-CONCENTRATION DEPENDENCE OF ν_{dd}

The separation in frequency of the points of maximum and minimum slope ν_{dd} are plotted in Fig. 2 against the sample age. The accuracy is $\sim\pm 1.0$ kHz for $t < 200$, but for $t > 300$ h the accuracy is only $\sim\pm 3.0$ kHz due to the widening and flattening of the derivative peaks. As in previous work, the data show that after the initial rapid decrease in linewidth at $t \sim 20-24$ h ($X \sim 0.56$), the linewidth continues to decrease but at a slower rate. The new data show more clearly that the continued slow decrease with time is linear, with a slope $d\nu_{dd}/dt = 18$ kHz/100 h. The data on linewidth vs. temperature of Fig. 7 show that for $t < 240$ h the curve of ν_{dd} vs temperature appears to approach its temperature independent limit at our lowest temperatures. Thus, the decrease in splitting with time cannot be due just to thermal excitations of the higher states of molecular orientation. As in our previous work, the primary dependence of the splitting on X is thought to arise from the reduction in the order parameter at $T = 0$ caused by an increase, as the ortho concentration decreases, of the mixing of states with $J_z \neq 0$ into the ground-state molecular wave function.

The change in slope after about 240 h seems to occur because the refrigerator is no longer cold enough to attain the low temperature limit for the order parameter. The inability to reach tempera-

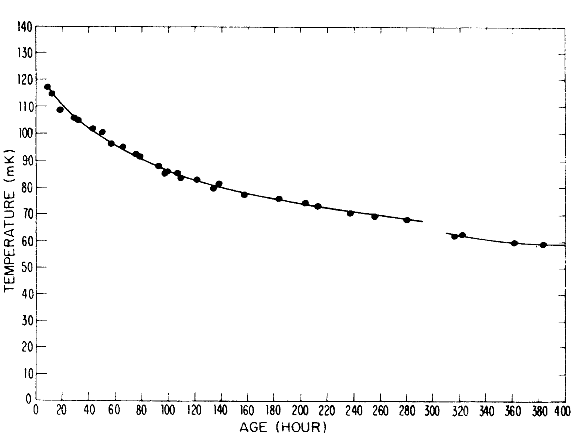

FIG. 1. Temperature achieved by dilution refrigerator as the sample aged.

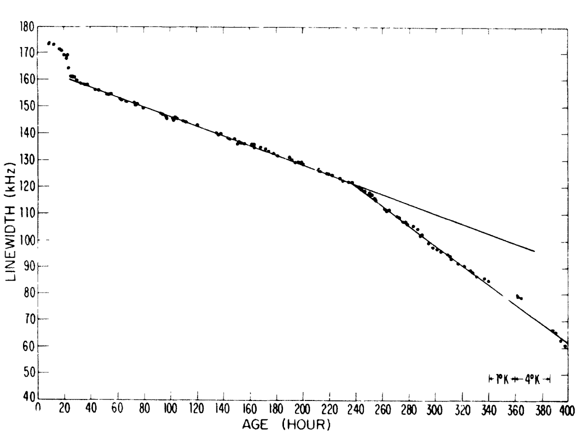

FIG. 2. Separation in frequency of maximum and minimum of the derivative of the absorption line as the sample aged.

tures sufficiently low to observe the zero temperature limit results in an additional concentration dependence in the data for $t \gtrsim 240$ h.

ORTHO-CONCENTRATION DEPENDENCE OF LINE SHAPE

At the highest concentration and the lowest temperature, the line shape corresponded quite closely to the expected dipolar doublet broadened by random intermolecular dipolar interactions. However, as the concentration fell below 0.5, the line departed more noticeably from such a shape. Figure 3 reproduces oscilloscope pictures taken at eight different times during 400 h all at the lowest temperatures the refrigerator could attain. All the photographs except the last were taken before 240 h, i.e., in the region where the linewidth retained little temperature dependence at the lowest temperature achieved. As the sample aged, the deep valley between the cusps, characteristic of the shape at high concentration, filled in rapidly. In about 120 h, the absorption spectra were essentially flat topped. The outermost steps of the predicted line shape became broadened and within the first 100 h they were no longer distinguishable from the sides of the central part of the line. Note the appearance of a weak component of only about 8 kHz width in the center of the resonance after about 50 h. After 150 h, the absorption spectra had rounded tops and became bell shaped. At the longest times, the sides of the absorption curve were remarkably straight.

For comparison, shapes of the absorption lines and of the first derivatives for randomly oriented dipole pairs, convoluted with various broadening functions, were evaluated numerically on a Data General Corp. Nova II computer. Care was taken to study the influence of the size of the steps in

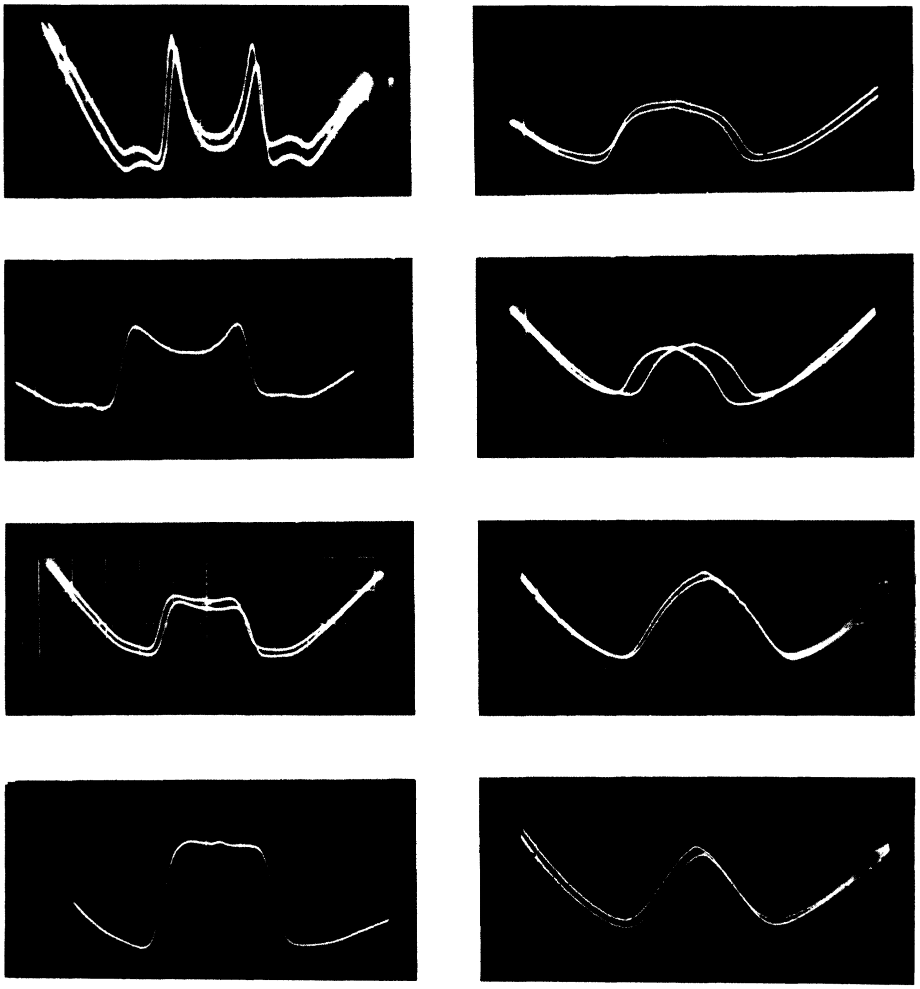

FIG. 3. NMR absorption lines at several ages. All lines are superposed on the downward resonance of the cavity. In some displays, a small phase shift of the sinusoidal oscilloscope horizontal sweep and a distortion separates the signals for up-going and down-going frequency sweeps. The ages and temperatures are from top to bottom on the left: 19 h, 110 mK; 45 h, 102 mK; 98 h, 86 °K; 119 h, 84 mK. On the right: 158 h, 77 mK; 183 h, 76 mK; 234 h, 73 mK; 334 h, 62 mK. The overall gains and the sweeps vary from picture to picture.

the numerical integration procedure, especially at the cusps. For Gaussian broadening functions, as an approximation for the effect of intermolecular dipole-dipole interations,[11]

$$g(\nu)=[1/\sigma(2\pi)^{1/2}]\,e^{-\nu^2/2\sigma^2}, \qquad (2)$$

the correction to the distance between derivative extrema caused by the intermolecular broadening is found numerically to be

$$\nu_{dd}=3d+1.1\sigma, \qquad (3)$$

in agreement with Ref. 12. The differentiated absorption line recorded at 0.12 °K, 9.5 h after freezing the sample, is compared in Fig. 4 with a computed derivative adjusted to match the amplitude and splitting of the experimental curve at the extrema. A Gaussian broadening of second moment $\sigma=7$ kHz, corresponding to the value derived from the Van Vleck formula at about 67% concentration was folded with the shape function corresponding to randomly oriented proton pairs. In the immediate region of the extrema, the fit is good but the broadening of the outermost steps is greater than in the calculated function. The bump displayed at the step should be an almost direct representation of the broadening function operating at that part of the line. The experimental width is about twice the width of a Gaussian function of the calculated second moment. The derivative curve calculated with $\sigma/d=0.25$, makes a better fit to the position and width of those parts of the observed line, but is much broader around the main cusps. At 4.2 °K and 4 h, the line was also broader than expected with width between extrema of the derivative of 26 kHz rather than 16 kHz for a Gaussian of the calculated second moment. Various factors complicate the prediction of the second moment in the ordered phase because the width prevents spin-spin exchanges from taking place among all interacting pairs. Such factors, however, would decrease the second moment by allowing some terms to be dropped. Observation of the narrow line (~8 kHz) in the dilute orthohydrogen at the end of the run absolves the magnet inhomogeneity of responsibility for the excess broadening.

As the sample aged, the observed line shape at the lowest temperatures departed increasingly from a dipolar doublet with Gaussian broadening. For the center of the doublet to be completely filled in, as it was observed to be after 120 h, the computed shapes represented by surface of the solid of Fig. 5 show that σ/d must have increased to at least 0.85. The line observed at 120 h, however, was still so wide that an unreasonably large value $\sigma\approx 31$ kHz would be required to fit it. This is more than twice even the broadening seen in the wings at 95 h, whereas the decrease of the ortho concentration should result in a corresponding decrease in the second moment. We conclude that the model of a dipolar doublet with Gaussian broadening is inadequate for $X<0.50$. Alternative broadening functions, for instance, a Lorentzian, also failed to fit the observed shapes in a similar manner.

The small 8-kHz wide line superimposed on the spectra for $t>50$ h is presumed to result from or-

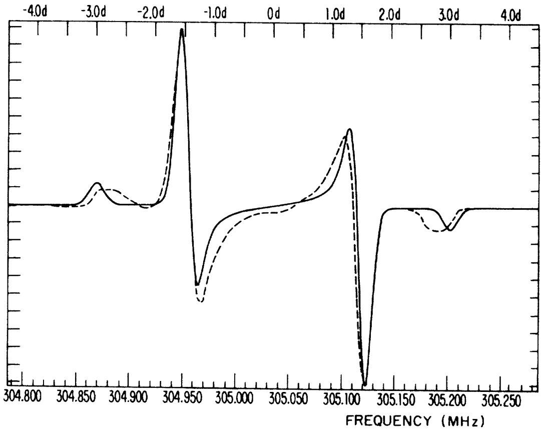

FIG. 4. Comparison of the experimental differential absorption line at 0.12 °K and 9.5 h (dashed line) with the derivative curve computed using Gaussian broadening with $\sigma=0.125d$, or about 7 kHz, corresponding to the calculated second moment at $X\approx 0.67$.

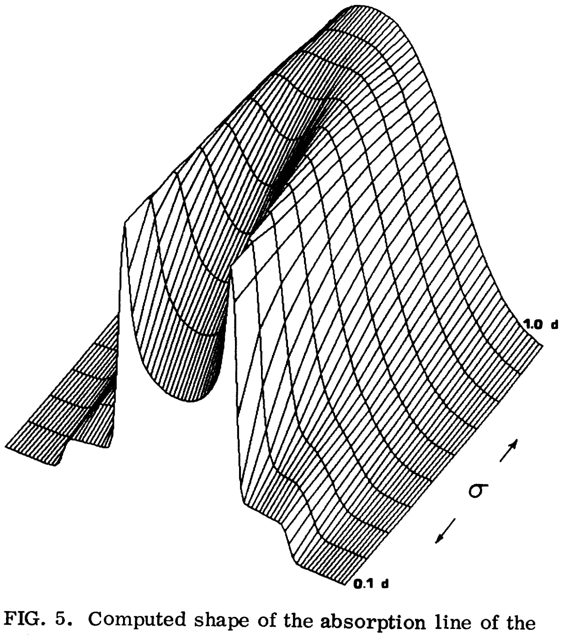

FIG. 5. Computed shape of the absorption line of the dipolar pair convoluted with a Gaussian-broadening function displayed as a figure of three dimensions. The axis normal to the front face corresponds to the range $0.1<\sigma/d<1.0$.

tho molecules without ortho neighbors in nearest-neighbor sites. Its relative intensity at $T < 0.1\,^\circ$K grows slowly as the sample ages and is about 2% at 120 h age. Examination of the derivative curves at the lowest concentration reveals structure corresponding not only to that central weak line but evidence of components about 40 kHz width, and other less well resolved and broader components. These may correspond to isolated ortho pairs and larger clusters, respectively.

TEMPERATURE DEPENDENCE OF THE LINE SHAPE

At fixed ortho concentration, the line shape showed a surprisingly different temperature dependence for $X < 0.56$ than for $X > 0.56$. For $X > 0.56$, as the sample was cooled to the transition temperature, well-resolved cusps grew on either side of the central line. On further cooling, the central line became small and disappeared entirely. The transition was reversible, and on warming toward the transition the cusps moved inward only by about 20% as the central line regrew.

For $X < 0.56$, as the sample was warmed, the center of the doublet filled in and the line gradually narrowed with increasing temperature until $\nu_{dd} = 40$ kHz at $T > 0.7\,^\circ$K. Figure 6 reproduces a set of photographs taken at various temperatures at a sample age of 57 h. There is a striking similarity between these and lines seen in Fig. 3. The temperature dependence of ν_{dd} for several different concentrations $X < 0.56$ is shown in Fig. 7. It is tempting to assume that if the refrigerator could have reached lower temperatures, $\nu_{dd}(T=0)$ for $t > 240$ h would be given by the continuation of the straight line in Fig. 2 drawn through the data points for $t < 240$ h. The values of $\nu_{dd}(T=0)$ so obtained are indicated by arrows in Fig. 7. The solid curves have been fitted to the data assuming a splitting of an anisotropic crystal field[13]

$$V_i = \tfrac{1}{3}\Delta_c(3J_{iz}^2 - 2) \tag{4}$$

which allows an increasing thermal population of the $J_z = \pm 1$ states as the temperature is raised. The function describing these curves is

$$\nu_{dd} = A[1 - 3e^{-\Delta_c/kT}/(1 + 2e^{-\Delta_c/kT})] \tag{5}$$

obtained from $1 - \frac{3}{2}\langle J_z^2\rangle_T$ where A was adjusted to fit $\nu_{dd}(T=0)$. The fit is good for $X < 0.25$, but becomes poorer for the curves at higher ortho concentration, which have a steeper temperature dependence. This may be because the transition has a more cooperative character at larger ortho concentrations. At low ortho concentrations a field of lower symmetry, fully raising the rotational degeneracy and resulting in two different energy intervals would be more realistic and makes possible a large posible range of dependences of ν_{dd} on temperature.

In a recent publication, Sullivan describes results from a similar experiment performed with a

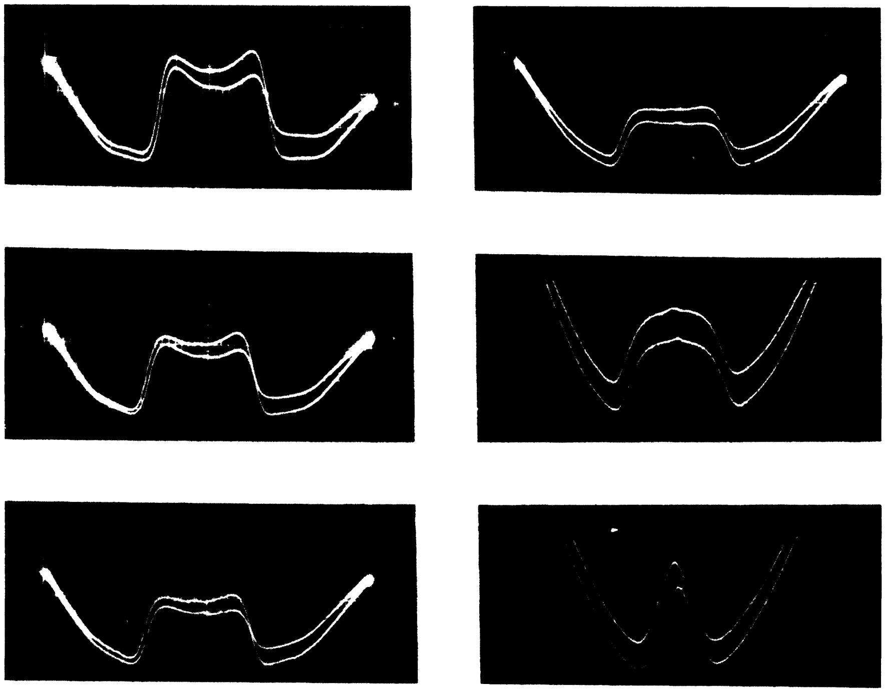

FIG. 6. Sequence of absorption lines at 57 h as the temperature is increased. The sweep amplitude is 45 kHz per division. The temperatures are 90, 115, 130 mK from top to bottom on the left and 160, 270, and 570 mK on the right. The vertical gain is doubled in the last photographs. Evidence of the sample polarization is seen in the higher right-hand side in the first two pictures.

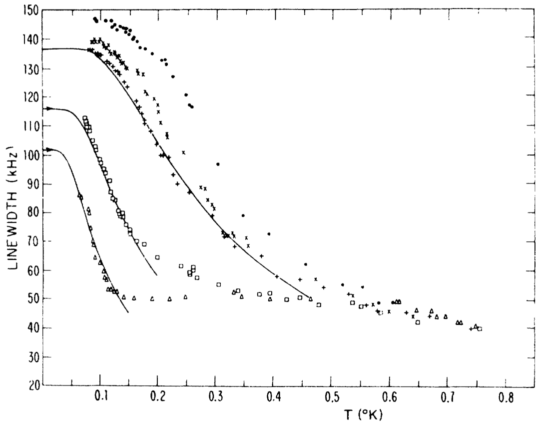

FIG. 7. Distance between extrema of the derivative curves vs temperature at several sample ages. The dots correspond to the sample of age in the range 92–95 h; the points plotted with × were found at an age 137–141 h; the crosses 162–166 h; the open squares 265–269 h and the triangles at 340–346 h. Solid lines calculated from Eq. (5) approximate some of the crosses for $\Delta_c = 0.475$ °K, the open squares for $\Delta_c = 0.275$ and the triangles for $\Delta_c = 0.18$ °K.

H_2 sample frozen on fine copper wires and aged for times up to 210 h.[14] On warming, he observed, even for $X < 0.5$, a rapid but smooth initial decrease of ν_{dd} by about 25%, followed by a much more rapid decrease in linewidth. The transition temperatures he measured from the discontinuities are shown in Fig. 8 as a function of ortho concentration, assuming the ortho-para conversion obeys Eq. (1). Although our data of $\nu_{dd}(T)$ do not show sudden discontinuities, we have also plotted in Fig. 7, the temperatures at which our $\nu_{dd}(T)$ have decreased by 25% from $\nu_{dd}(T=0)$, and find good agreement with Sullivan's transition temperatures. In calculating the ortho concentration of our sample, we have also used the equation $\dot{X} = -kX^2$, but as will be shown later, there is evidence that the conversion accelerates for $t > 200$ h relative to the quadratic rate when the sample is aged in the ordered phase. The ortho concentration of our lowest data point in Fig. 8 is thus $X = 0.07$ rather than $X = 0.12$. With this correction, $T_c(X)$ becomes approximately a straight line through the origin.

The differences between N. S. Sullivan's results and ours are not understood, although an important factor could be that his samples are frozen onto fine wires. The larger cooling surface and shorter path through hydrogen to that surface could result in more uniform temperatures in his samples than in ours, with thermal inhomogeneity precluding observation of an abrupt transition. We assumed the main thermal bottleneck to be between the hydrogen and the metal and not in the bulk, however. Sullivan's samples were aged at 4 °K instead of at $T < 0.1$ °K. Finally, there might be an effect dependent on field strength. He observed the H_2 NMR in a field only $\frac{1}{3}$ as large as ours.

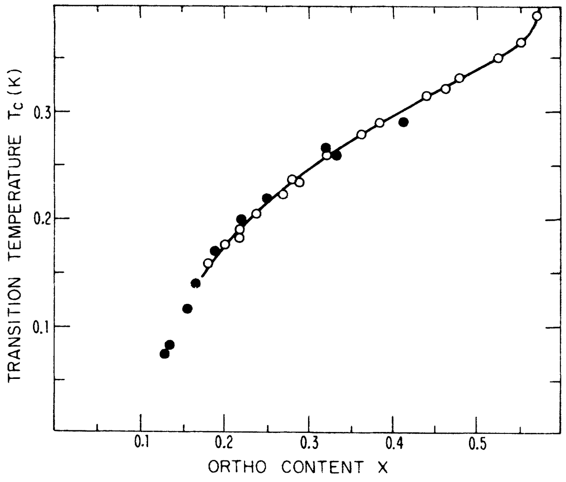

FIG. 8. Transition temperature vs ortho concentration. The open circles are from Sullivan, Ref. 14, and the solid circles are from this work, derived as described in the text. The ortho concentration used is derived from the age assuming $\dot{X} = kX^2$, $k = 0.019$ per hour.

ORTHO-PARA CONVERSION AND MOLECULAR MOTION

After aging for 340 h at the lowest temperatures, the sample was warmed to 1 °K, $\nu_{dd} \cong 40$ kHz, but during the next 16 h the 8-kHz central component, initially very small, grew at the expense of the 40-kHz component. The ratio of the areas under the 40-kHz curve compared to the 8-kHz curve as a function of time at 1 °K is shown in Fig. 9. When

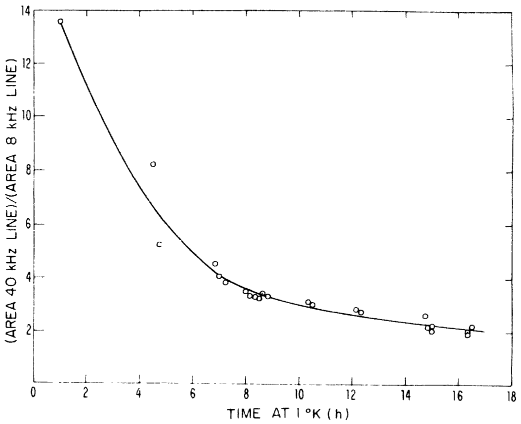

FIG. 9. Ratio of the areas of the broad (40 kHz) compared to that of the narrow (8 kHz) component vs time after being brought to 1 °K from the temperature of aging below 100 mK.

the sample was warmed to 4.2 °K after 360 h, the line was 8 kHz wide with little remnant of the 40-kHz curve.

Several authors have pointed out that the electric quadrupole-quadrupole interaction between ortho molecules can alter the random distribution of molecules and trap the ortho molecules in clusters as the temperature decreases.[6,15,16] The ortho molecules fall into lower energy states of pairs and triples, from which they have insufficient energy to escape at very low temperatures. We interpret the 40-kHz line observed on first warming to 1 °K as representing the pairs formed by clustering during the 340-h aging at $T < 0.1$ °K. At 1 °K, the molecules have sufficient energy to break the extra pairs in a quantum diffusion process which has a time constant of about 6 h. Quantum diffusion in H_2 has been observed previously by Amstutz *et al.* in the range $0.4 < T < 4.2$ °K for $X < 0.01$, but was observed by them not to occur at higher concentrations.[15]

Evidence from the integrated intensity of our NMR lines at 4.2 °K suggests that the conversion in our sample increased relative to the rate expected for a random lattice with interacting pairs. Thus, our ortho concentration at 378 h age was found to be 0.07 rather than 0.12 predicted from Eq. (1). Schmidt observed a similar acceleration of the conversion rate in a sample aged at 1.57 °K, which led to an ortho concentration of ~0.09 after 400 h.[6] Our line shapes at 1 °K observed at 356 h age are similar to those of Harris *et al.* for $0.06 < X < 0.09$ at $T = 1$ °K.[17] This is further evidence that the conversion rate has increased, because $\dot{X} = -kX^2$ would predict $X = 0.125$ at $t = 356$ h.

STUDIES OF SATURATION EFFECTS

The recovery of the NMR line after selective saturation was observed on an oscilloscope by sweeping through the spectrum with a frequency modulated uhf generator and saturating at a fixed frequency with another uhf generator. Homogeneous saturation over the whole spectrum was observed following the application of a fixed frequency near the center of the doublet. All points of the spectrum then recovered in the same time T_1 which was similar in magnitude to that seen by Sullivan and Pound,[7] and showed the same strong concentration and temperature dependence. At high ortho concentrations when T_1 was long, it was possible to saturate one cusp and simultaneously cause the enhancement of the other cusp. This is shown in Fig. 10. The molecules in the two cusps then recover to a common spin temperature in a short time T_x by adiabatic nuclear spin flip-flops between neighboring molecules induced by the $I_1^+ I_2^-$ terms of the dipolar coupling. The entire spectrum recovers to thermal equilibrium with the lattice in the longer time T_1. We find T_x is approximately 5 s, independent of ortho concentration and temperature over the observable range $0.70 < X < 0.50$. Saturation and recovery could not be carried out at lower concentrations without overloading the dilution refrigerator.

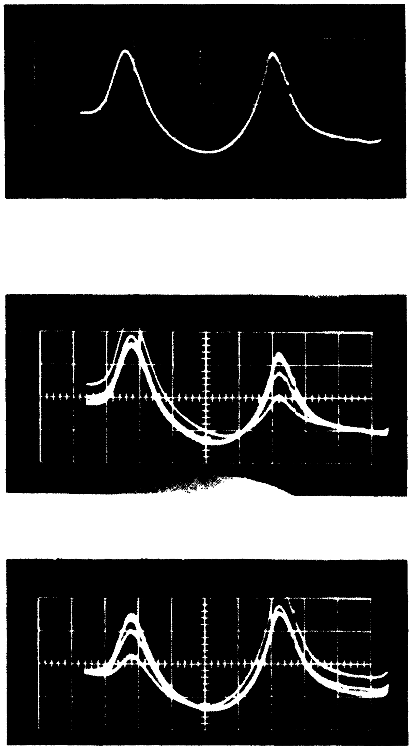

FIG. 10. Photographs showing recovery after saturation of one cusp. The upper trace is the equilibrium at $T = 0.42$ °K for the sample at 7 h age before saturation. In the center picture are four scans made at 5 S. intervals after saturating the right-hand cusp. The bottom exposure has the same procedure but follows saturation of the left-hand cusp.

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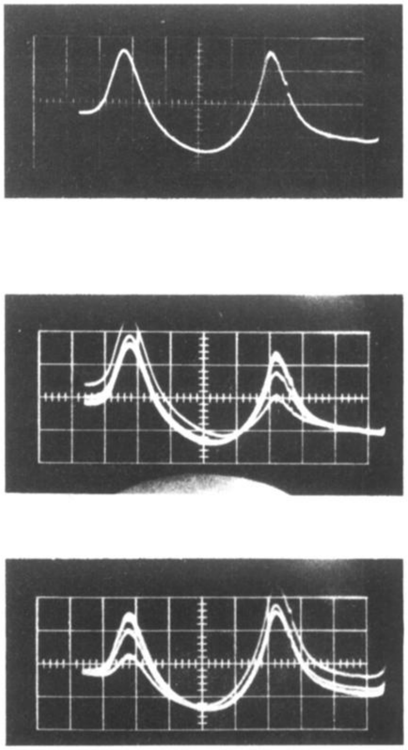

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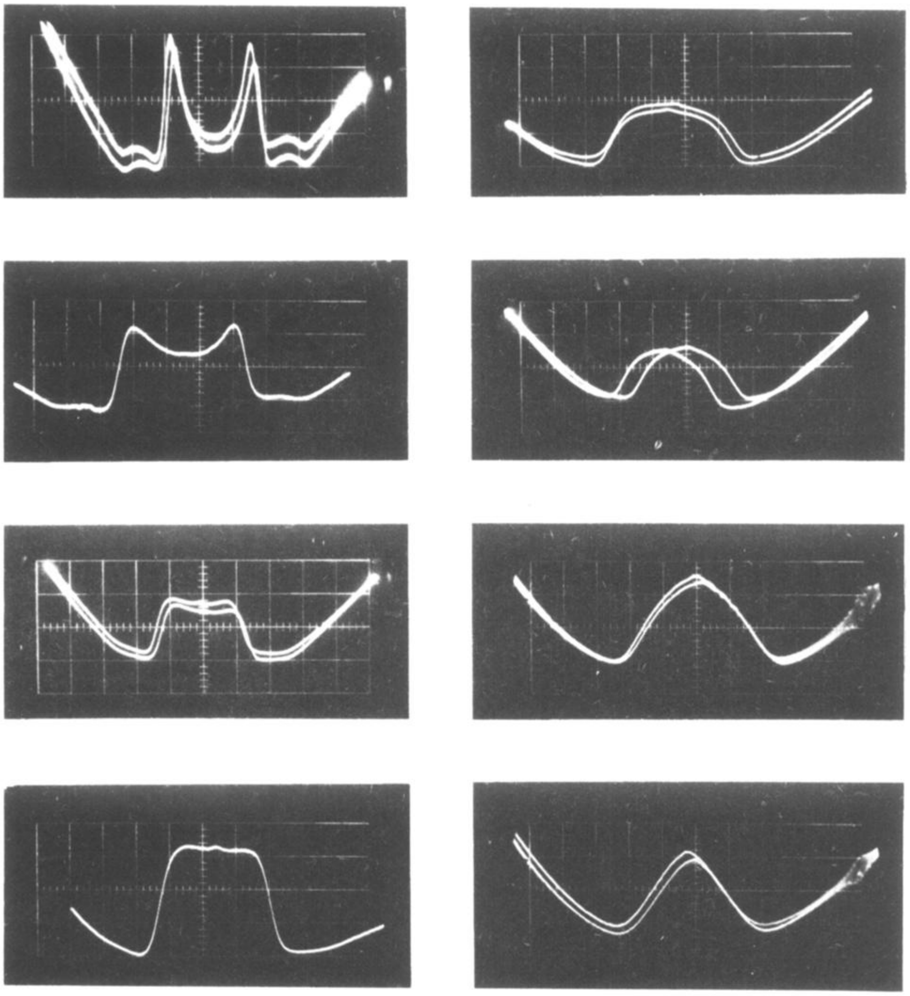

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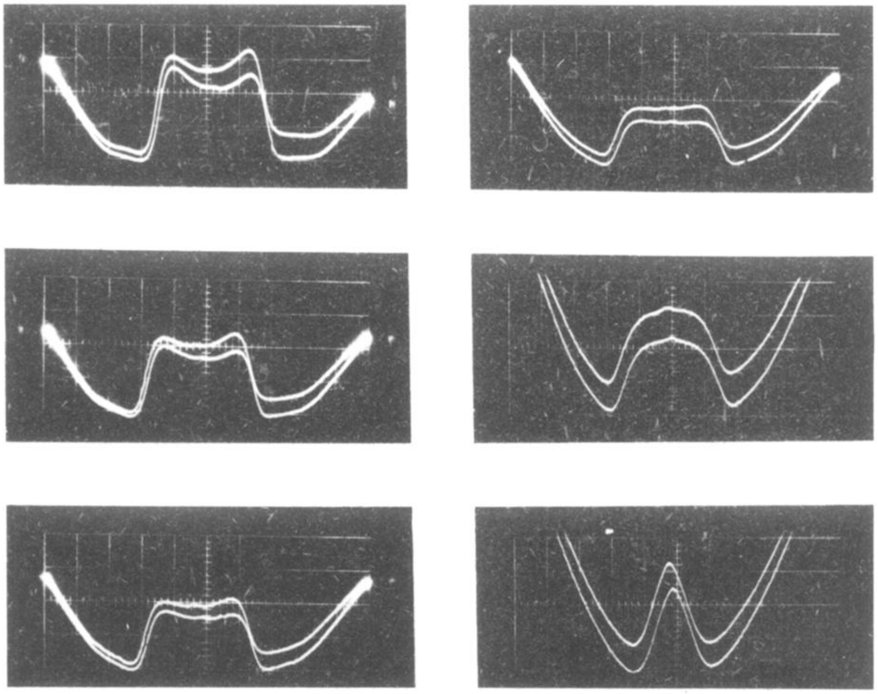

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