

## Polarization of Li Nuclei in Irradiated LiH\*

R. T. SCHUMACHER† AND J. L. HALL‡

*Department of Physics, Carnegie Institute of Technology, Pittsburgh, Pennsylvania*

(Received August 18, 1961)

The electron spin resonance in colloidal lithium metal produced in ultraviolet-irradiated LiH has been investigated at room temperature. The "Overhauser shift" of the electron resonance frequency due to nuclear polarization may be observed. By an electron-nuclear double resonance experiment it is shown that the polarization of the metallic lithium nuclei probably causes polarization of the lithium in the neighboring nonmetallic regions, i.e., the nonmetallic nuclei are in thermal contact with the nuclei in the metal, and tend to come to the same spin temperature.

### I. INTRODUCTION

SEVERAL years ago Doyle *et al.*<sup>1</sup> reported the observation of a narrow electron spin resonance line in lithium hydride after the crystal had been subjected to uv irradiation for several hours. By studying the temperature dependence of the intensity of the resonance, they concluded it was from the conduction electrons of lithium metal present in colloidal form within the LiH matrix. The resonance line width, although temperature independent, was narrow enough to permit Gueron and Ryter<sup>2</sup> to observe, at 4.2°K, the shift in the external field at which the resonant condition for the electrons was satisfied as the microwave power at the electron Larmor frequency was increased. This effect was predicted by Overhauser<sup>3</sup> in his paper on the "Overhauser effect." A consequence of saturating the electron spin resonance in a metal is to produce an appreciable polarization of the nuclei in the sample (the Overhauser effect). The conduction electrons move in an average internal field due to the nuclei which is considerably enhanced by this polarization. We propose in this paper to call the resultant shift in the apparent electron Larmor frequency the "Overhauser shift." The shift was measured quantitatively by Ryter<sup>4</sup> in a beautiful series of experiments on colloidal lithium in LiF at 4.2°K, and Overhauser's prediction was borne out in detail.

It occurred to several investigators, including the present authors, that the metallic nuclear polarization should be communicated to nonmetallic lithium nuclei in LiH by the process of spin diffusion.<sup>5</sup> The Knight shift of metallic lithium is small enough (0.025%) that at moderate laboratory fields the metallic and nonmetallic nuclei are on "speaking terms" even when the conduction electron polarization is only partly destroyed by saturation of the electron resonance.<sup>6</sup> The experi-

mental problem of demonstrating this "cooling" of the nonmetallic nuclear spins is the subject of this communication.

The most direct experimental attack on the problem would be to observe by nuclear magnetic resonance the enhanced lithium nuclear polarization. The distinction between the metallic and the nonmetallic nuclei would presumably be made on the basis of linewidth, position, and the sheer size of the signal from the more numerous nonmetallic nuclei. This direct experiment was performed, in fact, by Gueron and Ryter.<sup>2</sup> They observed the enhancement of the metallic lithium nuclear resonance in LiH at room temperature. Although there is no direct mention of the nonmetallic nuclei in their letter, their observations are clearly consistent with the conclusion that they failed to observe any enhancement of the nonmetallic lithium nuclear resonance.

Possible reasons for the failure of such a direct experiment are not hard to guess. If the colloidal particles are large enough, they can be separated by a considerable distance and still provide the sample with enough atoms in metallic form to produce a strong electron resonance. The diffusion constant associated with spin diffusion is extremely small, so it is conceivable that a large fraction of the nonmetallic nuclei are maintained at the lattice temperature by a spin-lattice relaxation mechanism much stronger than their contact with the cold metallic nuclear spins via spin diffusion. Such considerations produce a picture of polarized lithium nuclei in LiH near a metallic particle, with the degree of polarization decreasing as one proceeds farther away from the metal. We have performed an experiment which we believe shows that the latter picture is probably correct.

Our qualitative experimental demonstration that at least the lithium nuclei in LiH near the metallic particles are polarized rests on the following observations. At room temperature the nuclear resonance in metallic lithium is motionally narrowed<sup>7</sup>; the linewidth of the nuclear resonance in the metal is ten to one hundred times narrower than in the LiH solid. This characteristic narrow line was observed by Gueron and Ryter<sup>2</sup> in their

simple spin flips between two spins, one in the metal and one in the insulator, are not possible unless the relative shift of the resonance frequencies is destroyed by saturating the electron resonance.

<sup>7</sup> D. F. Holcomb and R. E. Norberg, Phys. Rev. **98**, 1074 (1955).

\* Work supported in part by a grant from the National Science Foundation.

† Alfred P. Sloan Foundation Fellow.

‡ National Carbon Predoctoral Fellow.

<sup>1</sup> W. T. Doyle, D. J. E. Ingram, and M. J. A. Smith, Phys. Rev. Letters **2**, 497 (1959).

<sup>2</sup> M. Gueron and Ch. Ryter, Phys. Rev. Letters **3**, 338 (1959).

<sup>3</sup> A. W. Overhauser, Phys. Rev. **92**, 411 (1953).

<sup>4</sup> Ch. Ryter, Phys. Rev. Letters **5**, 10 (1960).

<sup>5</sup> N. Bloembergen, Physica **15**, 386 (1949).

<sup>6</sup> It was pointed out to us by A. Abragam that in metals for which the Knight shift is much greater than rigid-lattice linewidth,

LiH sample. It is possible to make the Overhauser shift disappear by destroying the nuclear polarization with rf at the nuclear resonance frequency. If the metallic particles were isolated from their surroundings (i.e., the metallic and nonmetallic spins "insulated" from each other), the destruction of the Overhauser shift would occur over a narrow range of frequencies of the saturating nuclear frequency rf determined by the narrow metallic nuclear resonance line width. If, on the other hand, the metallic nuclei are in good thermal contact with the lithium nuclei in the LiH surrounding the metal particles, destruction of the nonmetallic nuclear polarization will result in depolarization of the metallic nuclei, and the consequent destruction of the Overhauser shift. The point is that the depolarization of the lithium nuclei in the rigid LiH lattice may be achieved with rf applied at five to ten metallic resonance linewidths away from the lithium nuclear resonance frequency, where the direct transition probability for metallic spins in the rf field is very small.

In Sec. II the essential experimental evidence is described in detail, and in Sec. III these results are discussed and our semi-quantitative conclusions are presented.

## II. EXPERIMENTAL DETAILS

Colloidal metallic lithium was produced in single crystals of lithium hydride by irradiation for 24 to 48 hr with light from a GE H100-A4 mercury lamp. The crystal of LiH was obtained from a one pound can of LiH purchased some years ago from the Metal Hydrides Corporation. The unirradiated crystals are opaque and appear light gray in reflected light. After irradiation they appear quite black.

The electron spin resonances were observed with a more or less conventional X-band spectrometer using superheterodyne detection. The resonances may be observed with good signal-to-noise on the oscilloscope. Data were taken on two samples. In sample one, the line shape was a function of microwave power. At very low powers, 30 db down from 30 mw incident on the cavity, the line has broad wings with a sharp center. The peak-to-peak derivative linewidth was about 0.15 gauss. At above 300  $\mu$ w the sharp center saturates, and the remaining line is about 0.5 gauss broad at the absorption half-maxima. It seems clear this sample contains particles of widely varying linewidths, possibly reflecting a wide range of particle sizes. This fact has important consequences for the measurement of the Overhauser shift, particularly under the circumstances that the maximum expected shift is much less than the linewidth, such as at room temperature and also at 77°K. The resonance line shape in sample two was more nearly Lorentzian, and the low-power half-maximum full width was 0.15 gauss. The resonance was easier to saturate completely than in sample one, but the signal-to-noise of the residual signal at high power was too poor to be useful.

The sample cavity was designed to allow application of considerable low-frequency rf power for double resonance experiments. The low-frequency current flows in a No. 16 copper wire that runs the length of the rectangular half-wave resonant cavity about 2 mm from a narrow face and midway between the broad faces of the cavity. The wire is grounded inside the cavity near the iris coupling the cavity to the waveguide, and is brought into the cavity at the bottom through a microwave choke. The low-frequency rf power is brought down the outside of the waveguide to the wire by a low-characteristic impedance ( $\sim 0.5$  ohm) transmission line. For the experiments reported here the rf is furnished by a Hewlett-Packard 606A signal generator which couples to the transmission line via a 17:1 step-down transformer. This arrangement allows about 50 milligauss (rotating) to be produced at the sample. If a power amplifier is inserted between the 606A and the transmission line, several gauss may be produced at the sample.

The Overhauser shifts were measured in the double resonance cavity described above by the following technique. The magnetic field was locked to the electron spin resonance by modulating the field a small fraction of the line width at 346 cps and applying the dc signal obtained from lock-in detection of the resonance to the "NMR" input of the Varian magnet supply. The saturating rf field was applied at the lithium metal nuclear resonance frequency (about 5.5 Mc/sec). This field destroyed the nuclear polarization and hence the Overhauser shift, as described in the introduction. The external magnetic field was forced by the circuitry to maintain itself in the center of the electron resonance. The change in field upon saturation of the lithium nuclear resonance was measured by measuring the change in frequency of a proton nuclear resonance system which was adjusted to keep the proton resonance pattern constant on the oscilloscope. All frequency

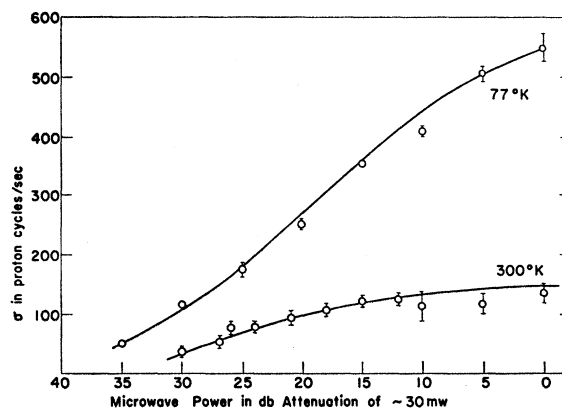


FIG. 1. Overhauser shift vs microwave power at 77°K and 300°K. The shifts were measured by the method described in the text. The smooth curves are drawn arbitrarily. The precision of the measurements at microwave power levels greater than 10 db is poor because of noise problems at high powers.

measurements were made with a Hewlett-Packard 524C electronic counter.

The results of these measurements on sample one at room temperature and 77°K are displayed in Fig. 1. The maximum shifts of 130 "proton cps" at room temperature, and 550 cps at 77°K, are smaller by a factor of 3 to 4 than the shifts of 476 cps and 1850 cps at the two temperatures as calculated from the results of Ryter<sup>4</sup> at 4.2°K. Our measurements imply an enhancement of the nuclear polarization by a factor of about 300. This is to be compared with the enhancement of 150 seen directly at room temperature by Gueron and Ryter.<sup>2</sup> Caution should be exercised, however, in interpreting the measurement reported here too quantitatively. We would like to emphasize again that the particular change of line shape of the electron resonance we observe upon increasing microwave power implies a wide distribution of saturation parameters over the colloidal particles. A little thought convinces one that under these circumstances, bearing in mind that the maximum possible Overhauser shift is much less than a linewidth, a rather complicated average of the shifts produced in the various particles is measured. Aside from the demonstration that the shift at room temperature is observable in a semi-quantitative way, we make no use of the quantitative results displayed in Fig. 1. The corresponding measurements were not made on sample two since the residual signal from the nearly completely saturated resonance was too small to lock the field. Qualitative observations indicated that the theoretical maximum shift was more nearly attained with sample two than in sample one. The matter was not pursued further since the measurements of Ryter<sup>4</sup> on the Overhauser shift are much more satisfactory than any results we could expect to get by our method.

We now turn to the experiment of primary interest. The object was to investigate the range of rf powers and frequencies near the lithium Larmor frequency with

which one could destroy the Overhauser shift by depolarizing the metallic nuclei. For this experiment the Varian magnet was stabilized by a proton resonance obtained from a Pound-Knight spectrometer locked to a Hewlett-Packard 608D signal generator. Frequency drifts of the 608D were much smaller than those in the microwave system. The rf power near the lithium Larmor frequency was again provided by the Hewlett-Packard 606A signal generator. The frequency of this generator was swept mechanically through the region of the lithium nuclear resonance. The shifts were recorded as a function of the 606A frequency by observing the derivative of the electron resonance on a recorder. For this purpose, and also to provide a dc feedback signal from the proton system, the field was modulated at 346 cps. The field was set so the electron resonance was exactly in the center; the absorption derivative was zero. As the 606A oscillator swept slowly through the region of the lithium nuclear resonance, the Overhauser shift was progressively destroyed, causing the electron resonance to shift away from dead center. The resultant signal on the recorder may be seen to be approximately proportional to the Overhauser shift  $\sigma$  (or to  $(\sigma_{\max} - \sigma)$ ) by noting that for a Lorentz absorption line the first derivative is nearly linearly proportional to frequency between the peaks.

The resultant curves are plotted in Fig. 2. These curves are, in fact, composite curves taken on sample two. Small frequency drifts in the microwave system, perhaps caused by temperature variations of the sample cavity to which the klystron was stabilized, made determination of the baseline to which we refer the curves of Fig. 2 somewhat difficult for a single run. The microwave power was adjusted so that the shift  $\sigma$  was much less than the peak-to-peak derivative width of about 500-proton cps ( $\sim 0.1$  gauss). The ordinate,  $(\sigma_{\max} - \sigma)$ , is not intended to be quantitatively accurate.

We have searched for a similar effect on the Overhauser shift from possible polarization of the proton system in LiH. The rf power at the proton Larmor frequency was applied while we observed the electron spin resonance as before. No effect at all was observed. One can conclude from this that the proton system polarization, if any, is independent of the nuclear polarization of the metallic lithium.

It will turn out to be necessary, even for qualitative analysis of the data, to know the magnitude of the rotating component of the low-frequency field. The configuration of the low-frequency field at the sample is such that by rotating the magnet 90° fields originally perpendicular to  $H_0$  become parallel to  $H_0$ . The effect of these 5.5-Mc/sec fields parallel to  $H_0$  may be described in terms of frequency modulation of the electron Larmor frequency. If the low-frequency power is increased until the modulation index is greater than  $\frac{1}{10}$ , electron spin resonances appear at fields displaced from the main resonant field by the equivalent in field of 5.5 Mc/sec. The intensities of the sideband resonances as a function

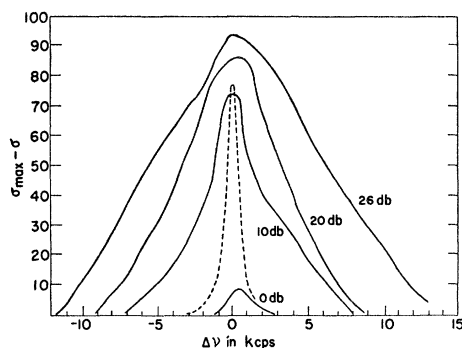


FIG. 2. The effect of low-frequency rf on the position of the electron resonance.  $\Delta\nu=0$  corresponds to the lithium Larmor frequency of  $\sim 5.5$  Mc/sec. Microwave power down 15 db from 30 mw, room temperature, gives  $\sigma_{\max} \sim 100$  proton cps. The ordinate is only approximately calibrated in proton cycles. (4.26 proton kc/sec = 1 gauss). The curves labeled 0 db, 10 db, 20 db, and 26 db correspond to an  $H_1$  at 5.5 Mc/sec of 5.7, 20, 58, and 115 milligauss, respectively. The dashed curve is explained in the caption to Fig. 3.

of low-frequency power relative to the intensity of the center line allows one to deduce the modulation index and hence the 5.5-Mc/sec rf field amplitude. It was possible to infer from these measurements that this field varied by a factor of 1.5 over the sample, a result consistent with the design of the double resonance cavity. The variation of a factor of 1.5 in the amplitude of the low-frequency rf unfortunately results in a variation of a factor of  $\sim 2$  in the saturation parameter for the nuclear resonance transitions. The amplitudes of the rotating components listed in the figure caption for the various curves in Fig. 2 represent an arbitrary mean of the possible values for each power level of the 606A oscillator.

### III. DISCUSSION OF THE RESULTS

We will show first that the curves of Fig. 2 are qualitatively inconsistent with an assumption of no interaction between the metallic lithium and the LiH matrix. If we assume the metallic lithium to be completely insulated, in the sense that our experiments detect interactions with surroundings, then the Overhauser shift as a function of low-frequency rf power is easy to deduce. We need only extend the rate equations of Overhauser, Eqs. (9) and (11) of his paper,<sup>3</sup> to include the effect of the rf transition probability  $W = \gamma^2 H_1^2 (\pi/2) f(\Delta\omega)$ .  $f(\Delta\omega)$  is the line shape of the metallic lithium nuclear resonance. The result for the Overhauser shift  $\sigma$  is

$$\sigma \propto \frac{S_e N H_0 \hbar \gamma_e}{(2I+1)k\theta} \left[ \frac{1}{1+2WT_n} \right] - \frac{N \hbar H_0 \gamma_n}{(2I+1)k\theta} \left[ \frac{2WT_n}{1+2WT_n} \right]. \quad (1)$$

$S_e$  is the electron resonance saturation parameter as defined by Overhauser,  $0 \leq S_e \leq 1$ ,  $N$  the density of spins,  $H_0$  the external field,  $\gamma_e$  the electron gyromagnetic ratio,  $\gamma_n$  the nuclear gyromagnetic ratio,  $I$  the nuclear spin, and  $\theta$  the absolute temperature.  $T_n$  is the nuclear relaxation time, which we will assume to be caused entirely by the conduction electrons, an assumption which is not quite correct at room temperature.<sup>7</sup> The second term is much smaller than the first except when  $2WT_n = \gamma_e/\gamma_n$ . We will neglect it in the discussion. The first term is essentially the Overhauser nuclear polarization multiplied by a saturation term which expresses the effect on the nuclear polarization of the rf field. From the work of Holcomb and Norberg,<sup>7</sup> we know that  $f(\Delta\omega) = (T_2/\pi)(1+T_2^2\Delta\omega^2)^{-1}$ , where  $\Delta\omega$  is the deviation from the angular Larmor frequency and  $T_2 = 10^{-3}$  sec. Thus we have

$$\frac{1}{1+2WT_n} = \frac{1+4\pi^2 T_2^2 \Delta\nu^2}{1+4\pi^2 T_2^2 \Delta\nu^2 + \gamma_n^2 H_1^2 T_n T_2}. \quad (2)$$

We use  $T_n = 0.140$  sec,<sup>7</sup> and plot  $(\sigma_{\max} - \sigma)$  vs  $\Delta\nu$  for several values of  $\gamma_n^2 H_1^2 T_n T_2$  in Fig. 3. The values of  $\gamma_n^2 H_1^2 T_n T_2$  of 0.46, 5.35, 47.6, and 185 were chosen to correspond to the values of  $H_1$  for the various curves in

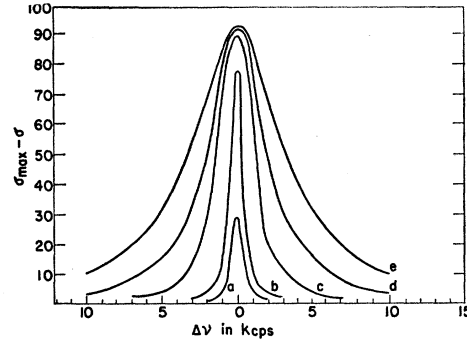


Fig. 3. Curves derived from Eq. (1) of the text. Under the assumption that  $\sigma_{\max} = 95$  proton cps, an ordinate scale designed to match that of Fig. 2 was used. Curves (a) through (e), respectively, correspond to values of  $2W = \gamma_n^2 H_1^2 T_n \pi f(0) = 0.46, 5.35, 47.6, 185$ , and  $500$ . Values of  $2W$  for curves (a) through (d) were chosen to match the rf powers of the 0 db, 10 db, 20 db, and 26 db curves of Fig. 2. Curve (b) is drawn dashed in Fig. 2, where it is to be compared to the 10 db experimental curve.

Fig. 2. The curve for  $\gamma_n^2 H_1^2 T_n T_2 = 500$  is included for purposes of later discussion. The values of  $(\sigma_{\max} - \sigma)$  at  $\Delta\nu = 0$  do not agree very well with the experimental observations, although the value corresponding to the 10-db curve in Fig. 2 is nearly quantitatively correct.

We contend that the data of Fig. 2 are in qualitative disagreement with Fig. 3. Particularly striking is the +10-db curve, which should be compared to  $\gamma_n^2 H_1^2 T_n T_2 = 5.35$  shown dashed in Fig. 2. The value of  $(\sigma_{\max} - \sigma)$  at  $\Delta\nu = 0$  agrees with Fig. 3, and even some suggestion of the sharpness of Fig. 3 is preserved for  $\Delta\nu \lesssim \pm 1$  kc/sec. Particularly for  $\Delta\nu$  greater than  $\pm 3$  kc/sec the two curves show completely different behavior. The higher power curves of Fig. 2 show this disagreement with their corresponding numbers of Fig. 3 to a similar, but less striking degree.

We shall next formulate in more concrete terms our expectations of the effect of saturating rf on  $\sigma$  in the presence of spin-spin interactions between metallic and nonmetallic lithium nuclei. We shall write rate equations for a highly idealized model: two homogeneous spin systems with an interaction between them which tends to keep them at the same spin temperature. With respect to the metal system the assumption of homogeneity is probably not bad. Even at room temperature the diffusion constant  $D$  for atomic (not spin) diffusion is so large that  $(2DT_2)^{1/2} \cong 3$  microns, which is several particle diameters. With respect to the nonmetallic nuclei, we are more vague. At a minimum, we refer to the lithium nuclei in the hydride which are nearest to the metallic particle. Let  $\delta$  be the population difference between adjacent spin levels in the hydride, let  $\Delta$  and  $D$  refer to the corresponding quantities in the nuclear spin system of the metal, and the conduction electron system of the metal, respectively. Our rate equations are:

$$\frac{d\Delta}{dt} = \frac{(\Delta_0 - \Delta)}{T_n} + \frac{(D_0 - D)}{\frac{2}{3}I(I+1)(2I+1)T_1'} - 2W\Delta - \frac{(\Delta - \delta)}{T_2^*}, \quad (3)$$

$$\frac{d\delta}{dt} = \frac{(\delta_0 - \delta)}{T_\delta} - 2W'\delta - \frac{(\delta - \Delta)}{T_2^*}. \quad (4)$$

$\Delta_0$ ,  $\delta_0$ , and  $D_0$  are thermal equilibrium values.  $T_n$  is the metal nuclear spin-lattice relaxation time, assumed as before to be entirely due to conduction electrons.  $T_1'$  is the conduction electron relaxation time due to the same mechanism.  $T_\delta$  is the spin-lattice relaxation time of the relevant hydride lithium nuclei.  $W$  and  $W'$  are the direct rf transition probabilities for the metal and hydride lithium nuclei respectively.  $T_2^*$  is a time characterizing the interaction between the metallic and nonmetallic systems. It is determined by the nuclear dipole-dipole interaction and the motional narrowing effects of atomic diffusion. The correlation time relevant to the latter is about twice the nuclear spin-spin correlation time in the metal since the nuclei in the hydride are, we assume, static. A reasonable estimate of  $T_2^*$  might be several times the metal nuclear  $T_2$ . Except for the last two terms, Eq. (3) is Eq. (11) of Overhauser.<sup>3</sup>

In the steady state, one may solve for  $\Delta$ . We obtain

$$\Delta = \frac{[\Delta_0 + 2S_e N \beta_e H_0 / (2I + 1) k \theta]}{1 + 2WT_n + T_n g(W', T_2^*)}, \quad (5)$$

where

$$g(W', T_2^*) = \frac{1 + 2W'T_\delta}{T_\delta + T_2^*(1 + 2W'T_\delta)}.$$

We have neglected additive terms on the order of  $\delta_0 T_n / T_\delta$  in the numerator. If  $W = (T_2^*)^{-1} = W' = 0$ , (5) reduces to the Overhauser polarization. If only  $(T_2^*)^{-1} = 0$ , (5) reduces to (1). One sees that for situations such as the 10 db curve beyond  $\Delta\nu = \pm 2$  kc/sec,  $T_n g(W', T_2^*)$  can dominate the denominator, since because of the difference between metal and nonmetal linewidths one may have  $2W'T_\delta \gg 1$  and  $2WT_n \ll 1$  satisfied simultaneously. However, one may destroy completely the Overhauser shift via saturation of the nonmetallic nuclei only if  $T_2^* = 0$ . In view of the unknown quantities  $W'$ ,  $T_\delta$ , and  $T_2^*$ , quantitative speculation is useless. We believe, nevertheless, that the experimental results disagree with the curves of Fig. 3 [ $T_2^* \rightarrow \infty$  in (5)] and are susceptible to interpretation in terms of the model presented here.

One further warning against taking (5) quantitatively too seriously should be made. We can expect (4) to be valid only if  $2W'T \lesssim 1$ . Redfield<sup>8</sup> showed conclusively that this approach is invalid for  $2W'T \gg 1$ .<sup>9</sup>

<sup>8</sup> A. G. Redfield, Phys. Rev. **98**, 1787 (1955).

<sup>9</sup> But Eq. (3) is valid, at least on this score, since the metal system at room temperature is a "viscous liquid" in the sense

Finally, it should be pointed out that for sufficiently large nuclear frequency fields the curve for  $\gamma_n^2 H_1^2 T_2 T_2 = 500$  of Fig. 3 is applicable. The saturation factor is so large that appreciable depolarization may result directly from the term  $2WT_n$  in (5), and this effect may be larger than the indirect depolarization via the hydride spins because of the  $T_2^*$  bottleneck. Thus it is conceivable the experimental curve labeled +26 db in Fig. 2 may be approaching this limit.

#### IV. CONCLUSION

We have presented the idea that Overhauser polarization of the colloidal metal nuclei may result in polarization of lithium nuclei in the nonmetallic part of the sample. The fraction of the nonmetallic nuclei to be polarized depends sensitively on the number density of the metallic particles and on strength of the relaxation mechanisms of the nonmetallic nuclei. It seems clear that the dipole-dipole interaction between the lithiums in the metal and in the hydride must cause at least local polarization of lithium in the hydride. A simple model of the metal particle and its immediate surroundings was presented, which because of the motionally narrowed resonance nuclear resonance line width in the metal, suggested that selective depolarization of the hydride lithiums by an rf field could be achieved. The subsequent depolarization of the metal lithium nuclei due to the dipolar interaction was detected by monitoring the conduction electron spin resonance. Since the simplest mutual spin-flip interaction between protons and lithium nuclei does not conserve energy, no enhancement of the normal proton polarization is expected. In agreement with the model, no effect on the Overhauser shift was observed upon application of rf at the proton resonance frequency.

The preparation of samples of this type is, at least in this laboratory, still a random art. It is obvious that if samples of LiH could be prepared consistently with a sufficient number density of colloidal particles, polarization of all the lithium nuclei in the sample could be achieved. Polarization of the protons could then be achieved by the Abragam and Proctor<sup>10</sup> technique of reducing the applied fields to zero periodically.

#### ACKNOWLEDGMENTS

We wish to acknowledge useful conversations on this and related experiments between one of us (R.T.S.) and Professor A. Abragam and Dr. J. Winter.

discussed by A. Abragam, *Nuclear Magnetism* (Clarendon Press, Oxford, 1961), Chap. XII.

<sup>10</sup> A. Abragam and W. G. Proctor, Phys. Rev. **109**, 1441 (1958).