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Experimental Verification of the Overhauser Nuclear Polarization Effect*

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The experimental verification of Overhauser's proposal for polarizing nuclear spins is described. The effect on the nuclear magnetic resonance of saturating the electron spin resonance in several appropriate systems was observed at low fields and room temperatures. The systems investigated were: metallic Li, metallic Na, and Na dissolved in anhydrous liquid ammonia. The nuclear resonances of Li^7, Na^{23}, and H^1 (in the ammonia) were observed at 50 kc/sec in fields of 30.3 gauss, 44.2 gauss, and 11.7 gauss, respectively, and the electron spin resonances were saturated with the corresponding applied frequencies of 84, 124, and 33 Mc/sec. The detailed predictions of Overhauser were confirmed as far as nuclear polarization is concerned, although other relaxation processes in Li reduce the polarization, and the difficulty in completely saturating the metallic Na electron resonance leads to a partial effect. Only in fairly concentrated solutions of Na in ammonia was a substantially complete effect observed in which the proton nuclear polarization increased by the ratio of the electron gyromagnetic ratio to the nuclear gyromagnetic ratio. The proton line widths in the Na-ammonia solutions further verify the theory of Kaplan and Kittel.

1. INTRODUCTION

OVERHAUSER has predicted[1] that the saturation of the conduction electron spin resonance (ESR) in a metal should produce an increase in the nuclear polarization by a factor of several thousand for metals in which the nuclei reach thermal equilibrium with the "lattice" by means of the magnetic hyperfine interaction with the conduction electrons. It was felt that such a surprising effect should be investigated in as direct and immediate a way as possible. Overhauser proposed a method which has been recently analyzed by Kaplan[2] to measure this effect. His scheme involved observing the shift of the ESR frequency brought about by the polarization of the nuclei (we have called this the Overhauser shift, and it has a nuclear resonance counterpart in the Knight shift[3]). Rather than using this method, we turned to the more direct procedure of observing the amplitude of the nuclear magnetic resonance (NMR) since it is directly proportional to the polarization. The latter method has the added advantage that it may be performed at room temperature.

There will be two sets of Zeeman levels established when the metal is placed in a magnetic field, and the transition frequency for the electrons in Li^7, for example, will be 1690 times as high as the NMR frequency. A choice of frequencies is necessary. One may either observe the NMR in the rf range, while saturating the ESR in the microwave band, or else saturate the electrons in the rf region which requires that the NMR be observed in the audio- or video-frequency range. Although the former is customary for nuclear and electron resonance work, and usually is better for good signal to noise, the choice of lower frequencies has some distinct advantages for our investigation. In particular, the conduction electron spin resonance had been saturated[4] in the rf range where more powerful oscillators may conveniently be built. It was desired to avoid line width effects resulting from the diffusion of the electrons in and out of the skin depth[5,6] particularly because the effects of saturation under these conditions are not known. The size of metal particles which were convenient to make (5 to 20 microns) was small compared to the skin depth at the lower frequencies. Finally, it was necessary to make the sample common to two resonant structures for the NMR and ESR, and from the standpoint of high sensitivity and ease of saturation, both structures should have as high a Q as possible. At the lower frequencies two coils can be used orthogonal to one another without interactions, and such things as filling factors and the magnitude of the ESR saturating field (H_1) actually produced in the sample can be measured or computed with more confidence. The frequencies finally chosen were based on observing the NMR of Li^7, Na^{23}, and H^1 at 50 kc/sec, and saturating the ESR at the frequencies corresponding to γ_e/γ_n.

2. EXPERIMENTAL TECHNIQUE

A general schematic diagram of the double resonance apparatus is shown in Fig. 1. A conventional type of absorption bridge nuclear resonance detection system

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[1] A. W. Overhauser, Phys. Rev. **91**, 476 (1953); **92**, 411 (1953). For further discussions see, for example: F. Bloch, Phys. Rev. **93**, 944(A) (1954); C. Kittel, Phys. Rev. **95**, 589 (1954); J. Korringa, Phys. Rev. **94**, 1388 (1954); A. Abragam, Phys. Rev. **98**, 1729 (1955).

[2] J. I. Kaplan, Phys. Rev. **99**, 1322 (1955).

[3] W. D. Knight Phys. Rev., **76**, 1259 (1949).

[4] T. R. Carver, thesis, University of Illinois, 1954 (unpublished); Carver, Holcomb, and Slichter (to be published).

[5] G. Feher and A. F. Kip, Phys. Rev. **98**, 337 (1955).

[6] F. J. Dyson, Phys. Rev. **98**, 349 (1955).

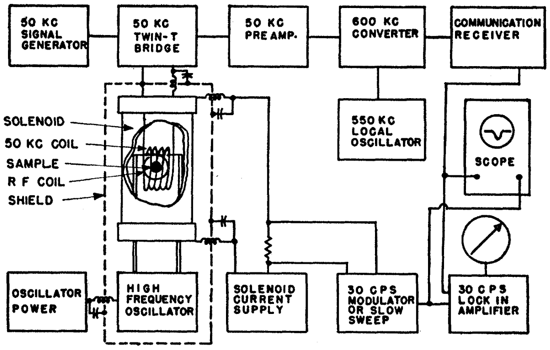

FIG. 1. Block diagram of the nuclear and electron double resonance apparatus.

designed for 50 kc/sec was used. The saturation of the electrons was accomplished by a push-pull oscillator capable of supplying 59 watts of rf power. The metal sample is contained in the tank coil. Although an alternating magnetic field, H_1, of only a few gauss is required to saturate the electron resonance, and the spin system actually absorbs only fractions of a watt, there is a considerable power loss to heating effects in the coil and sample. Because of the broad ESR line width in metals no special frequency stability is required of the saturation oscillator, and it could be conveniently tuned from 84 to 124 Mc/sec for the Li and Na resonances. When more easily saturated Na-ammonia solutions were studied, an ordinary signal generator at 33 Mc/sec was used to replace the oscillator. The sensitive nuclear resonance detection system was easily jammed by the high rf power levels used to saturate the electron spins, so that for shielding purposes the entire oscillator, sample, nuclear resonance coil, and low-field solenoid were placed in a copper box, and all leads including that to the nuclear resonance bridge were heavily filtered.

2.1 Low-Frequency Nuclear Resonance Detection

The essential components of the 50-kc/sec nuclear resonance apparatus are shown in Fig. 2. A twin T bridge employing a completely resistive null T is practical at low frequencies using a standard Helipot for adjustment, and was used instead of a Wheatstone bridge so that a common ground could be established. A simple tuned preamplifier was made to improve the noise figure at 50 kc/sec and the signal was then converted to 600 kc/sec where a standard communications receiver could be used. The signal was observed on the oscilloscope or recorded with a 30-cps lock-in amplifier.

Such a low-frequency bridge with large lumped circuit elements is unusually free from mechanical drift and microphonics, and can be easily balanced to 1 part in 10^6. However, the bridge was frequency-sensitive, and an ordinary audio-frequency signal generator proved to have not only too much noise amplitude modulation but frequency modulation and drift as well. To eliminate this source of noise, a 100-kc/sec crystal-controlled oscillator was constructed and the 50 kc/sec was obtained from it by means of a conventional frequency division circuit.[7] The use of batteries on the signal generator helped to eliminate all traces of frequency modulation. With this apparatus it proved possible to observe the proton resonance in a 30-cm^3 sample of glycerine with a signal-to-noise ratio of about 30 when using the lock-in amplifier with a 10-second time constant.

2.2 Magnetic Field and Modulation

The magnetic fields of 10 to 50 gauss were produced by a solenoid $6\frac{1}{2}$ in. long with additional end windings to improve the homogeneity.[8] Batteries were adequate and most convenient for the current supply. Observations of the proton resonance in distilled water using this solenoid indicated that the homogeneity was about 2 milligauss in 10 gauss over a 30-cm^3 sample.

Either slow sweep or 30-cps modulation could be applied in series with the solenoid current supply. In actual observations of the Overhauser effect in the metal samples, heating occurred during the saturation of the electron spin resonance and the balance of the bridge changed. Consequently, observations using the slow sweep were more convenient and reliable than longer observations necessary with the lock-in. The slow sweep was taken directly from a Dumont 304H oscilloscope and applied to the solenoid through the cathode resistor of a cathode follower which also carried the solenoid current. When the slow sweep was used, RC filtering of the signal from the receiver was the only method used to reduce the band width after detection. In general the sweep rates were approximately 20 milligauss per second.

2.3 Samples and Resonance Coils

The metallic Li and Na samples were in the form of particle dispersions in mineral oil. It was found that

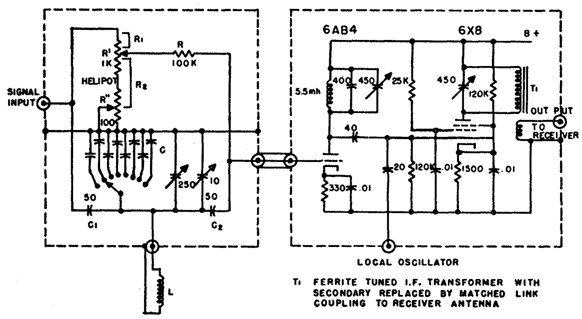

FIG. 2. Details of 50-kc/sec nuclear magnetic resonance twin-T bridge, preamplifier, and converter.

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[7] Chance, Hughes, MacNichol, *et al.*, *Waveforms* (McGraw-Hill Book Company, Inc., New York, 1948), Vol. 19, p. 578.

[8] W. Garrett, J. Appl. Phys. **22**, 1091 (1951).

particles of 10-micron diameter could be produced by stirring the metal when melted in hot mineral oil by a small sharp-bladed stainless steel stirrer spinning at about 20 000 rpm. This technique produces a range of particle sizes, so that a small fraction of 1-micron particles may be obtained by separation. With lithium samples in particular it was necessary to stir and handle the metal in an inert atmosphere (helium). After the excess oil was removed, the samples were sealed in small (about 10 cm^3) glass ampules. The electron line width in lithium is partially dependent on the amount of reaction with nitrogen, and since narrower and more easily saturated lines were desired, such precautions were necessary.

Sodium-ammonia solutions were prepared in the manner described by Hutchison and Pastor.[9] Samples of three different concentrations were used in the investigations and were sealed in 3 to 5 cm^3 Pyrex ampules. They were kept at room temperatures only during the time the resonances were observed. Concentrations of the solutions were measured after they were studied by opening the ampules and titrating the NaOH produced in exposure to the atmosphere.

The ampules containing the lithium dispersion were clipped directly in a $1\frac{1}{2}$-turn coil of the saturating oscillator. The sodium samples were similarly placed in a $\frac{1}{2}$-turn coil to provide the higher frequency necessary. Sodium ammonia solutions were placed directly in a five-turn coil driven directly by a signal generator capable of supplying about a volt to a matched load.

The low-frequency nuclear resonance coil was wound on a 4-cm diameter form into which both the sample and high-frequency coil could fit. This is a comparatively small size for a 50-kc coil, and it was necessary to wind it with some attention to eliminating distributed capacity in order to obtain a high Q. The coil used consisted of two 250-turn pi or universal wound sections connected in parallel, and had a Q of 80 and an inductance of 4 millihenries.

The magnitude of the rf magnetic field in the electron saturating coils was measured by a set of small probe coils of known area less than 1 cm^2 connected by twisted leads to a vacuum tube voltmeter. Induced voltages were in fact proportional to area, so that the reactance of the probe itself could be neglected in the frequency range used.

3. EXPERIMENTAL RESULTS AND INTERPRETATION

The general method of observation was to compare the NMR absorption signal amplitude when the conduction electron population was in thermal equilibrium and when it was saturated. The nuclear resonance absorption amplitude is directly proportional to χ'' which in turn depends on the polarization or effective susceptibility of the nuclear paramagnetism. Since the line widths were not expected to increase when the electrons were saturated and did not in fact do so, the enhancement of the nuclear resonance signal was a direct measure of the nuclear polarization produced. This was a practical method in studying metallic Na or Na-ammonia solutions where the nuclear resonance apparatus was sufficiently sensitive to observe directly the Na^{23} or H^1 resonance. However, in lithium the line width is so much broader[10] than in sodium that it proved impossible to compare the lithium resonance directly, since it could not be observed. The enhanced lithium resonance was therefore compared with a known amount of glycerine in which the proton resonance was observed. If two substances are observed at the same resonant frequency and H_1, in a coil whose Q does not change, the nuclear resonance signals differ only in proportion to T_2, the inverse at the half-maximum half-width in angular frequency, and χ_0, the nuclear susceptibility. χ_0 is given by the expression

$$\chi_0 = (N/3kT)\gamma_n^2\hbar^2 I(I+1).$$

Here N is the concentration of nuclei, I is the nuclear spin and γ_n the nuclear gyromagnetic ratio. The signal in unsaturated lithium was obtained in this manner from known[10] and measured values of T_2. In general, however, the lithium samples when observed with the electrons saturated were at a temperature of about 70°C.

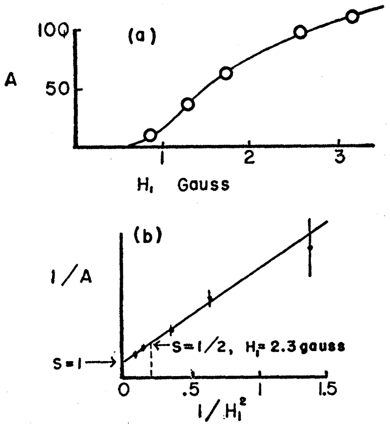

FIG. 3. Nuclear resonance absorption signal in metallic lithium as a function of electron saturating field, H_1. The unit of the ordinate, A, is the enhancement of the signal relative to the unsaturated, thermal equilibrium resonance.

3.1 Lithium

In Fig. 3(a) is shown the increase of the NMR signal as the electron saturating radio-frequency field is increased. The sample is one of the lithium samples showing the narrowest electron line width and was most easily saturated. The detailed prediction of Overhauser is that the nuclear polarization should increase as though the nuclei had an effective gyromagnetic ratio

$$\gamma_{\text{eff}} = \gamma_n + s|\gamma_e| T_n/T_n',$$

where γ_n is the normal nuclear gyromagnetic ratio, s

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[9] C. A. Hutchison, Jr., and R. C. Pastor, Revs. Modern Phys. **25**, 285 (1953).

[10] D. F. Holcomb and R. E. Norberg, Phys. Rev. **98**, 1074 (1955).

is the saturation factor, and T_n/T_n' is the ratio of the nuclear relaxation time arising from all nuclear relaxation processes with the "lattice," to the nuclear relaxation time arising only from the conduction electron nuclear hyperfine interaction. (It should not be thought, of course, that the effective gyromagnetic ratio implies a changed nuclear resonance frequency, but only that the population difference given by the nuclear Boltzmann distribution is increased.) The saturation factor s is $1-n/n_0$, where n is the population difference in the electron spin populations, and n_0 is the thermal equilibrium value of the population difference. The saturation factor may also be written in the form

$$s=\gamma_e^2H_1^2T_1T_2/(1+\gamma_e^2H_1^2T_1T_2).$$

Thus a plot of the reciprocal of the NMR signal *versus* the reciprocal of the square of H_1, the ESR rf magnetic field strength, should be a straight line, and is shown in Fig. 3(b). From this plot the saturation factor corresponding to the maximum H_1 available from the saturation oscillator may be calculated, and also an estimate of the H_1 required for half-saturation may be made. This latter value is 2.3 gauss. Since the half-maximum half-width of the ESR for this particular sample was measured directly to be about 2.5 gauss, this saturation value for H_1 is good experimental evidence that $T_1=T_2$ for the conduction electrons in metals, as generally expected. Since the maximum H_1 available was 3.3 guass, the maximum saturation factor is thus given by

$$s=(3.3/2.3)^2/[1+(3.3/2.3)^2]\approx 0.7.$$

Under these conditions, the nuclear resonance signal in lithium would be expected to increase by 0.7 γ_e/γ_n or about 10^3 if no nuclear relaxation processes were present other than the hyperfine interaction between nuclei and electrons.

Such is not the case, however, and we must consider the other processes. Fortunately, the detailed measurements of Holcomb and Norberg[10] on relaxation times in the alkali metals can be used to make a reasonable estimate of T_n/T_n'. Their data indicates that the electron nuclear relaxation time in Li^7 at 25°C and in a magnetic field of 5400 gauss is about 130 milliseconds. This time is in good agreement with the theoretical calculations of Korringa[11] and Overhauser,[12] and is inversely proportional to temperature, but should in theory be independent of static magnetic field. While the former feature has been verified experimentally, there appears to be a magnetic field dependence which is noticeable for low values of H_0 in both sodium and lithium. Since the deviations are the same in lithium and sodium, and since line width measurements made in this research at 50 kc/sec on sodium indicate that the relaxation time is half as long as reported at 9 Mc/sec by Holcomb and Norberg, a reasonable estimate for T_n' in lithium at room temperature and at 50 kc/sec is 65 milliseconds. At a temperature of 70°C, where observations on the saturated lithium were actually made because of rf heating, the T_n' is about 55 msec.

The actual line width of the NMR in lithium is caused by nuclear dipolar interactions between diffusing nuclei in the lattice.[10] At room temperature the value of T_2, the inverse line width, is 0.5 msec.[10] However, at 70°C, the lithium is above the T_1 minimum and T_1 is equal to T_2. Thus the total effective nuclear relaxation time T_n may be measured directly from the line width. It was found to be 6 msec. T_n/T_n' is about 0.12 and the value of the enhancement of the nuclear resonance to be expected under the conditions described is given by

$$A=1+s\frac{T_n}{T_n'}\frac{|\gamma_e|}{\gamma_n}\approx 0.7\times 0.12\times 1690$$

and is about 140.

The actual enhancement observed may be obtained from the comparison in Fig. 4 between the lithium signal with electrons saturated and the glycerine signal. Taking into account the sample volumes, line widths, nuclear susceptibility, and temperature in the two cases, the lithium signal was found to be increased by a factor of about 110, a value which is perhaps fortuitous within a factor of 2 considering the uncertainty in the assumed value of T_n', but which is nevertheless in general agreement with the features of the Overhauser theory. One can see from this figure, too, that under the conditions of identical bridge balance for both

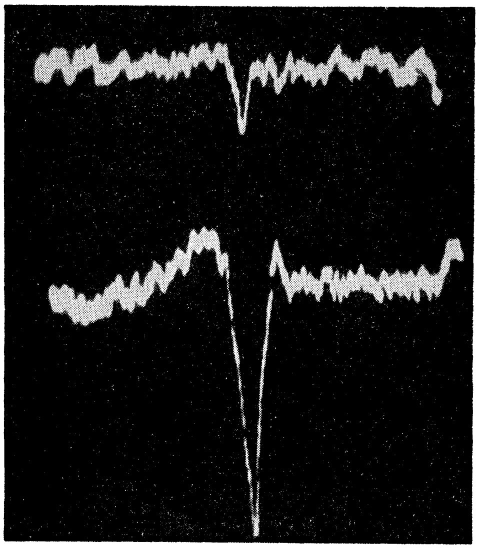

FIG. 4. *Top*.—Calibration proton resonance in 28 cm^3 of glycerine. Static magnetic field of 11.7 gauss with 0.4-gauss sweep. 25°C. *Bottom*.—Li^7 nuclear resonance with electron saturation factor of 0.7. Static magnetic field of 30.3 gauss with 0.6-gauss sweep. About 70°C. 3 cm^3 sample volume.

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[11] J. Korringa, Physica **16**, 601 (1950).

[12] A. W. Overhauser, Phys. Rev. **89**, 689 (1953).

the lithium and the glycerine, the signal obtained from the enhanced lithium resonance is an *absorption* signal as is expected in metals where the nuclear magnetic moment is positive. The Overhauser effect carried out in metals where the nuclear magnetic moment is negative should result in a stimulated emission signal from the nuclei. It should be noted from the short values of T_2 quoted from Norberg and Holcomb for lithium at room temperature that the dipolar relaxation would almost completely bypass the Overhauser effect at room temperature. This was observed and the NMR enhancement did in fact increase from about 10 to 130 as the lithium was heated by the rf currents in the sample. This behavior with heating, which is in agreement with what is to be expected from the temperature dependence of nuclear dipole relaxation effects, is good confirmation that it is this mechanism which is by-passing the nuclear electron relaxation essential to the Overhauser effect in metals. Other lithium samples of lesser purity showed the same behavior for the nuclear relaxation times, but the electrons were not so easily saturated and the samples showed correspondingly less enhancement.

3.2 Sodium

In sodium the electron nuclear hyperfine relaxation is the dominant one.[10] The inverse line width, T_2, has been measured to be 10 msec and T_1 is 15 msec in Na^{23} at room temperature and 8000 gauss. The value of T_2 observed in this research at 50 kc/sec was half as much, but the line width was sufficiently narrow to permit a direct comparison between the sodium NMR with and without the ERS saturated. The ESR was saturated at 124 Mc/sec in a field of 44.2 gauss. At this frequency the saturation oscillator could produce an H_1 of only 1 to 2 gauss over the sample volume. The electron line width is about 8 gauss at room temperature. However, the sample was strongly heated by the rf and since the electron line width is proportional to temperature,[4,5] it was nearly 12 gauss under conditions of observation. Therefore a saturation factor of 0.01 was all that could be obtained. An increase in the NMR signal of about 20 is all that could be expected with this saturation factor. The actual increase produced was about 10.

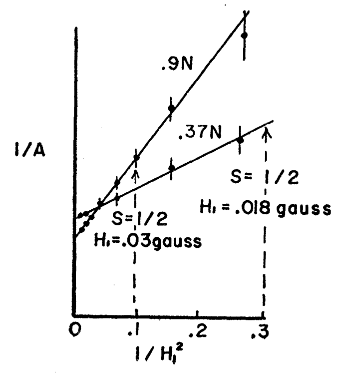

FIG. 5. Relative proton resonance absorption, A, as a function of electron saturating field, H_1, in solutions of sodium in ammonia. Units of the abscissa are 10^{-4} gauss^{-2}.

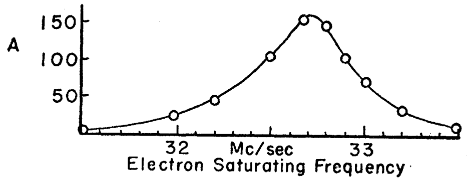

FIG. 6. Relative proton resonance absorption, A, as a function of electron saturating frequency, in the 0.37N solution of sodium in ammonia. Static magnetic field of 11.7 gauss. H_1 is 0.09 gauss.

3.3 Na-Ammonia Solutions

An analysis of the physical basis of Overhauser's polarization method led us to the conclusion that the effect could be found in any system in which the nuclear relaxation was dominated by a coupling to electrons in which the nuclear spin flip requires a simultaneous electron flip. Thus, for example, the ordinary magnetic dipolar coupling should make possible an Overhauser effect.[13,14] Other investigators have independently come to the same conclusion.[1]

To verify these conclusions, we performed experiments on systems of Na dissolved in anhydrous ammonia. This system has the added interest that the nucleus to be polarized is the proton. Beljers, Van der Kint, and Van Wieringen have independently studied proton polarization in a free radical.[15] We carried out investigations on three samples of concentrations approximately 0.01N, 0.4N and 0.9N. The solutions exhibit paramagnetic line widths as small as 20 milligauss[9] and can be easily saturated. Comparison of the enhanced resonance with normal resonance was made either directly using the lock-in amplifier or indirectly by comparison with a larger glycerine sample using the slow sweep. Both methods were in substantial agreement. It was found that the proton resonance enhancement was proportional to the saturation factor s in the same manner as illustrated for lithium in Figs. 3(a) and 3(b). Figure 5 shows the results for the two most concentrated solutions. The saturation was almost complete for the 0.01N sample and the maximum saturation values obtained for the 0.4N and 0.9N solutions were 0.96 and 0.88.

Figure 6 shows the NMR enhancement as a function of electron saturating frequency. The particular sample shown is the 0.4N sample and the maximum enhancement at the center is about 150. This curve actually represents an indirect observation of the electron resonance when greatly saturated. Consequently, the half-maximum half-width is equal to the H_1 used to saturate the electron resonance.

The data observed with the sodium-ammonia solutions are summarized in Fig. 7. Although the line widths observed at this low frequency are not too precise

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[13] I. Solomon, Phys. Rev. **99**, 559 (1955).

[14] D. F. Holcomb, thesis, University of Illinois, 1954 (unpublished).

[15] Beljers, Van der Kint, and Van Wieringen, Phys. Rev. **95**, 1683 (1954).

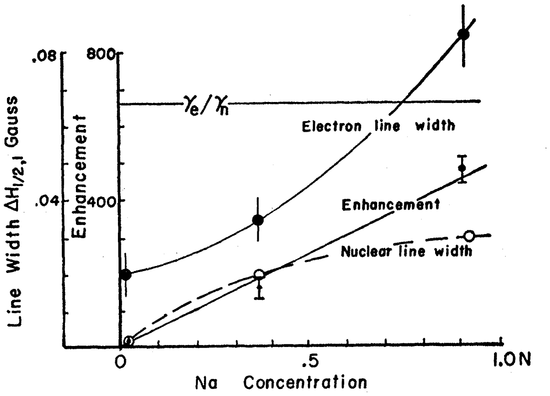

FIG. 7. Proton resonance enhancement, proton resonance line width, and electron spin responance line width shown for the three samples of sodium dissolved in ammonia.

because of the poor signal-to-noise ratio, the general behavior of the resonances and the concentration dependence of the Overhauser effect is indicated. At a concentration of about $1N$ or higher the solutions become brassy and exhibit the properties of a metal. Our observations indicate that as the concentration is increased through the transition range, the Overhauser polarization becomes complete.

It is suggested by Kaplan and Kittel[16] in a model for these solutions intended to explain, among other things, why the ESR line width is constant and independent of electron concentration at low concentrations, that the electrons trapped in large "cavities" in the solution interact by the s-state hyperfine interaction with the protons of the ammonia surrounding the cavity. If this is true, and the protons in turn are relaxed by the electrons, it is difficult to see why there is not a complete Overhauser effect even at lower concentrations, since the essential electron nuclear hyperfine interaction is present. Nuclear relaxation by dipolar coupling to other nuclei is not an effective short circuit of the nuclear-electron hyperfine interaction, so that effects such as we found in lithium should not be important. Moreover, the dipolar coupling to the electrons (which would polarize the nuclei in the *opposite* direction from the $\mathbf{I}\cdot\mathbf{S}$ hyperfine term) should be much less effective than the hyperfine coupling, and have a rather similar concentration dependence.

Our line-width data enables us to check the model of Kaplan and Kittel. That is, the nuclear line width ΔH_n due to coupling to the electrons via the hyperfine interaction is related to ΔH_e the electron line width contributed by the hyperfine coupling to the nuclei. The relation is

$$\Delta H_n = \Delta H_e \alpha \gamma_e / \gamma_n,$$

where α is the ratio of single electrons to protons. ΔH_e is independent of concentration and is presumably the observed electron line width at low concentrations. As Kaplan and Kittel show, α should vary as the square root of the sodium concentration. When one uses these facts, both the magnitude and concentration dependence of the nuclear line width is reasonable.

Kaplan and Kittel propose that the electron line broadens at high concentration owing to magnetic dipolar coupling among the electrons. This sort of process should likewise go as the square root of the sodium concentration. It appears that the electron line actually broadens much more rapidly with increasing concentration, suggesting a possible connection with the formation of a conduction band in which the electronic relaxation may be more effective, or possibly electron relaxation by spin-orbit coupling with sodium ions. The authors hope eventually to make a further study of these solutions under conditions where enhancements at low concentrations may be observed with better signal to noise.

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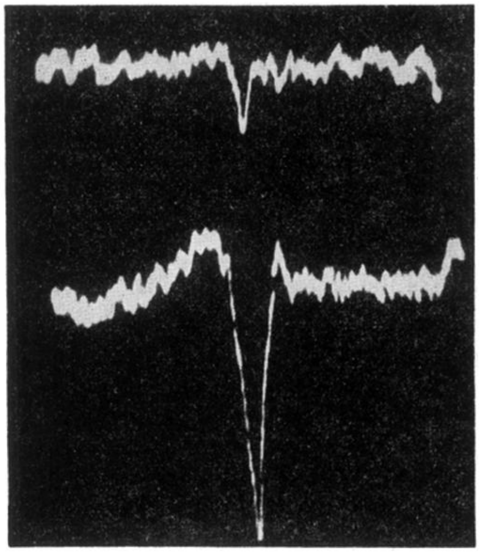
[16] J. Kaplan and C. Kittel, J. Chem. Phys. 21, 1429 (1953).



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