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THE MEASUREMENT OF LOW MAGNETIC FIELDS USING PARAMAGNETIC RESONANCE

Report Prepared for
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April 6, 1951

Berkeley, California

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THE MEASUREMENT OF LOW MAGNETIC FIELDS USING PARAMAGNETIC RESONANCE

E. C. Levinthal and E. H. Rogers of Varian Associates

April 6, 1951

Purpose of Report

This report covers the experimental and theoretical results obtained during a six week investigation of the feasibility of measuring magnetic fields from 5 to 50 gauss to ± 0.02 gauss with a sample no larger than 1 cc in volume in the presence of field inhomogeneities ranging between 1 gauss/in and 3 gauss/in. The conclusion reached in this report is that the field measuring requirements outlined above can be met in several ways using paramagnetic media presently available.

Introduction

In the years since the end of World War II a considerable advance has taken place in the field of nuclear magnetic resonance. The theory has been extensively developed and refined, the experimental apparatus has been improved enormously since the early devices, and a large and ever-growing tabulation of nuclear magnetic properties has been and is being compiled. This large body of knowledge has led to the rapid development of magnetic field measuring equipment utilizing the principles of nuclear magnetic resonance. Many such instruments are currently in use, principally in the United States, and certainly at the present time they provide the highest precision possible for determination of magnetic fields in the region for which they are useful.

A number of important factors, such as sample size, radio-frequency circuitry and techniques, availability of isotopes, and range of nuclear spin and

magnetic moment values have led to a practical limitation of conventional nuclear induction measuring equipment to magnetic fields above approximately 200 gauss. (The frequency range 1 mc. to 100 mc. corresponds to resonant fields of 235 gauss to 23,500 gauss for protons and 1530 gauss to 153,000 gauss for deuterons). It has been recognized that a device capable of measuring much lower fields would have value, especially if the techniques and relative accuracies of nuclear induction methods could be utilized. Since, with the exception of the triton, the proton has the largest measured nuclear gyromagnetic ratio, it seemed obvious that the solution of the low-field problem lay in the direction of paramagnetic resonance-----i.e., utilization of the large gyromagnetic ratio of the spinning electron.

To understand more fully the implications of the use of paramagnetic resonance, the following brief review of facts may be helpful. It is known that the nuclear magnetic moments can be most conveniently described in terms of a basic quantity

(1) $\mu_o = e\hbar/2Mc = 5.049 \times 10^{-24}$ erg/gauss

referred to as the nuclear magneton, in which e and M are the charge and mass of the proton, $\hbar$ is Planck's constant divided by 2π, and c is the velocity of light. If the magnetic moment of the proton were simply that of a spinning spherical shell of mass M and charge e, its value would be given exactly by the expression (1). Actually, careful measurements have determined that for the proton

(2) $\mu_p = 2.7935 \ \mu_o$

It has also been ascertained that for other nuclei which can be used in magnetic field measuring equipment the multiplying factor differs from, but is of the same order of magnitude as, the proton multiplying factor.

The theory of nuclear magnetic resonance[1] predicts that in a magnetic field H_o the nuclear induction signal will appear at a frequency ν given by

(3) $$2\pi\nu = \mu H_o / I\hbar$$

in which I is the spin of the nucleus and μ is the nuclear magneton multiplied by the factor described in the previous paragraph.

For the proton with spin 1/2, the resonance condition relationship is

(4) $$H_o = (2\pi I\hbar/\mu)\,\nu_p = 2.349 \times 10^{-4} \times \nu_p \text{ gauss}$$

Following this same line of reasoning, the spinning electron can be described in terms of a quantity

(5) $$\mu_b = e\hbar/2mc$$

known as the Bohr magneton, in which m is the mass of the electron. Comparison of (5) and (1) yields the information that the ratio of the Bohr magneton to the nuclear magneton is simply the ratio of the proton mass to the electron mass. Therefore, assuming that the magnetic moment of the electron is one Bohr magneton (which has been experimentally confirmed to be true to one part in 1000), and using the fact that the proton and electron spins are equal, we have for a given H_o field

(6) $$\frac{\nu \text{ proton}}{\nu \text{ electron}} = 2.79\,(m/M) \cong 1/660$$

Thus, for a field of 10,000 gauss, the proton nuclear induction signal will appear at 42.6 mc., whereas the paramagnetic resonance will occur at the microwave frequency of 28,100 mc. Conversely, at a given <u>frequency</u>, the electron signal will appear in a field 660 times <u>lower</u> than that required for proton resonance. As a typical example, for a frequency of 42.6 mc., the proton nuclear induction signal will appear at 10,000 gauss, whereas the electron resonance will appear at 15.2 gauss. Therefore, at least on the basis of the resonant frequency condition, paramagnetic resonance would seem to be ideally suited for measurement of low magnetic fields.

Unfortunately, "ideally suited" implies a great deal more than the mere existence of resonance at convenient radio-frequencies for low magnetic fields. There are, for example, such crucial matters as signal amplitude, signal width and sample size which affect the precision of field measurements. We would like to point out briefly some general considerations of these parameters insofar as they are connected with the precision.

Referring to the papers on Nuclear Induction[1,2], it is seen that the signal-to-noise ratio is proportional to the 5/6 power of the volume. If we assume, for the moment, that a signal-to-noise ratio of N:1 implies a possible precision for reading within the line width of 1/N, then, for a perfectly homogeneous field the precision, P, is proportional to $V^{5/6}$, where V = volume of spherical sample. If now we assume that the field is inhomogeneous for the simplicity is inhomogeneous in such a manner that the gradient across the sample is proportional to the radius of the sample, then, $P \sim \frac{V^{5/6}}{V^{1/3}} = V^{1/2}$ provided $T_1 = T_2$. T_1 equals the "thermal" or "longitudinal" relaxation time and T_2 equals the "transversal" relaxation time following the definition of Bloch[1]. If the field becomes sufficiently inhomogeneous or the sample large enough so that it is no longer possible to find samples with large natural line widths and with $T_1 = T_2$ (and thus the line width is being determined by the field inhomogeneities) then

$$P \sim \frac{V^{1/2}}{V^{1/6}} = V^{1/3}$$

What we have said above is that even for inhomogeneous fields and with samples which are being broadened by the field the signal gets larger in amplitude faster than it gets broader in line width so that the precision for reading within the line will improve with increasing volume. This statement requires however many qualifications. It implies that one's knowledge of the line shape is sufficient so that detailed analysis of the line is meaningful,

and more important for relative measurements, that the line shape is reproducible so that the predicted precision can really be achieved. For these reasons it is necessary to examine several of the factors which can affect the line shape. The resonance theory as shown by Bloch in his paper, and which applies both to nuclear resonance and paramagnetic resonance, demonstrated that the signal has two components. One is a symmetrical component called the V -mode and the other is an antisymmetrical component called the U -mode. With a bridge type or cross-coil resonance system either mode can be observed. The one which is observed is determined by the phase of the "leakage" signal which mixes in the detector with the r-f signal from the sample. For the V-mode, or symmetrical signal, the center of symmetry is resonance; and for the U-mode, or antisymmetrical signal, the center of asymmetry is resonance. Any mixture of modes will cause an uncertainty in determining which part of the signal indicates resonance. There are however several methods of observing only one mode at a time. In the first place, the phase of the leakage can be adjusted to be in quadrature with the phase of the undesired mode. These adjustments are usually frequency sensitive and would possibly have to be reset for each value of the field to be measured. There is no doubt, however, that the adjustments could be made to the required precision. One would reduce the leakage or homodyning signal until the detected signal was equal or less than the noise and then introduce leakage of the correct phase to observe the desired signal. With a crossed coil system such as described by Bloch, Hansen and Packard[3] the symmetric signal would be observed when the leakage was in phase with the r-f transmitter voltage. A reasonably frequency insensitive arrangement could be made to keep the amplitude of the U mode to between 1 percent and 10 percent of the V mode depending on the r-f level used. To do better would require critical adjustments. A regenerative system of detection such as used by Pound[4] will

detect only one mode; which of these modes is observed depends on whether or not the receiver is an a-m or f-m receiver. Such a system however has the limitation that it only has a good noise figure when used at low r-f levels. Thus such a system will work only for narrow lines, in which case it is not required to read within the line. Another possibility is to use the fact that the amplitude of the V -mode goes to zero for large r-f powers and the U -mode amplitude remains constant. It is possible to use large enough r-f powers so that only one mode would exist. However, this will present other problems that will be pointed out subsequently.

In addition to contributing to the line width, inhomogeneities of the magnetic field can also affect the symmetry of the signal, even though one is observing only the V -mode. If one plots a distribution function of the number of particles (nuclei or free electrons) in the sample in a particular increment of field versus the field, the observed signal will only be symmetrical if the distribution function is symmetrical. The magnitude of the effect will depend on the ratio of the change in field across the sample to the total field and the value of the odd moments of the above distribution curve. For the problem at hand where the gradients are as large as 3 gauss/in it is probable that the above effect would seriously limit the precision with which one could determine resonance within a line regardless of the signal amplitude. The effect is, however, subject to calculation and could be predicted provided the field shape was approximately known.

Wide signals require the use of large a-c sweep or modulation fields in order to obtain the maximum sensitivity. If the sweep is of the same order as the d-c field to be measured this will also introduce an asymmetry to the signal shape because of the field dependence of the signal amplitude.

Another difficulty in using wide signals can be found in the following:

It has been shown by Bloch and Siegert[4,5] that for particles with spin 1/2 and magnetic moment μ placed in a precission field H_o and an oscillating field of amplitude H_1, the condition of resonance is not given by the idealized Equation (3), but rather by

$$\omega = 2\pi\nu = \frac{(\mu)}{\frac{\hbar}{2}} \frac{H_o}{\left(1 - \frac{H_1^2}{16 H_o^2}\right)} \tag{7}$$

Since it was proposed to use conventional nuclear induction apparatus for paramagnetic resonance work, and since the required rotating field would then be derived from one component of an oscillating radio-frequency field (H_1), it appeared possible that, in view of the low H_o fields to be encountered, an annoying correction factor might be required which would involve some fairly accurate knowledge of the magnitude of the oscillating field. Equation (7) shows that the correction factor is completely negilgible for such usual nuclear induction situations as H_1 = 1 gauss and H_o = 1000 guass; but it also shows that for such possible low-field situations as H_1 = 1 gauss and H_o = 2 gauss, the correction factor can be of the order of 1.5 percent. Now it is generally true that for optimum signal amplitude with a particular sample it is desirable to excite the particles with an H_1 field whose amplitude is of the same order of magnitude as the natural line width of the signal. Further increase of the H_1 field amplitude beyond this optimum value will lead to undesirable signal broadening and amplitude saturation effects. Thus for wide signals where large r-f powers are required there will be a shift in the resonance value which will depend on the r-f level used. This can be corrected for or avoided by using a rotating instead of an oscillating r-f field. A direct method of avoiding the difficulty is to use samples producing narrow lines, and then to take advantage of the small r-f fields required for optimum amplitude. For example, an accuracy of at least one part in 1000 can be ob-

tained in an H_o field of 2 gauss if $H_1 \leq 0.25$ guass. Or, putting it more generally, an accuracy of at least one part in 1000 is assured if the ratio H_1/H_o is subject to the following limitations:

(8) $$H_1/H_o \leq 0.125$$

In view of the above difficulties encountered in determining resonance within a line width it is obvious that one should be cautions about going too far in this direction. It is also obvious that it is operationally simpler to deal with a signal whose width is equal to the required precision in gauss for the field measurement. The signal then merely has to be detectable, and simply finding the signal gives the desired accuracy. There are two difficulties that prevent the application of such a straight-forward procedure. Firstly, the inhomogeneity of the field broadens the line and hence imposes a limitation on the size of the sample which can be used for a particular line width. This could prevent one from obtaining a detectable signal. For example nuclear resonance with portons as a sample offers a large range of line widths from a fraction of a milligauss to several gauss depending on the concentration of paramagnetic salt used. However, at a field of 12 gauss a one liter sample would have to be used to get 15:1 signal-to-noise ratio. Besides being objectionable because of space requirements and the fact that the field measured is the average over a large volume, such a large sample in an inhomogeneous field would give a wide signal in spite of the small natural line widths possible.

The compromise between reading within a line and using narrow lines and small samples will be largely dictated by the paramagnetic media that are available. As will be pointed out in the discussion of the results, just a few useful possibilities exist. Unfortunately, with those paramagnetic materials that have been found so far, for optimum adjustment of the r-f field and for

homogeneous d-c fields, the media which give narrower lines also give proportionately small amplitudes. This is a different situation than one finds in the case of nuclear resonance where one changes the line width via the concentration of a paramagnetic salt.

Apparatus and Methods

In the search for an investigation of paramagnetic resonance effects, one requirement was a suitable magnet for producing the low fields in which the experiments were to be performed. Since the experiments were to be carried out in fields of 20 gauss or less, the solution was to design and construct an air-core magnet consisting of two coils separated by a distance determined from the Helmholtz condition. Stability was assured by the use of storage batteries as a current source. After weighting the various factors involved, a magnet with the following specifications was constructed:

Two coils, each consisting of 675 turns of Number 18 Formvar covered copper wire (total resistance: 20.8 ohms), and each of mean diameter 9 inches, were placed coaxially with a separation between their centers of 4.5 inches (helmholtz condition). The wooden spools upon which the coils had been wound were of such thickness as to leave a gap between the pole faces of 2.75 inches. A current of approximately 375 milliamperes was required to produce a field of 20 gauss at the magnet center. The inhomogeneity of the magnet was such that for deviations of $\pm$ 1 cm. from the magnet center, the field did not deviate from its central value by more than $\pm$ .006 percent. In a field of 20 gauss, this corresponded to an inhomogeniety of 1.2 milligauss over the region given. Since precession fields in excess of 20 gauss were not expected to be used, since the sample volume would not exceed $\pm$ 1 cm. in any dimension, and since natural line widths were expected to be somewhat greater than 1.2 milligauss,

the magnet was considered to be completely satisfactory for the present series of experiments. The magnet coils were connected in series and tuned by means of parallel condensers to the sweep frequency of 500 cps. A 500 cps audio oscillator was used to drive a power amplifier, the output of which was connected by means of d.c. blocking condensers to the tuned coils. In this way, the magnet coils themselves were modulated to provide continuously adjustable sweep amplitudes from zero to a maximum of 22 peak-to-peak gauss.

The precession field and sweep field circuits, meters and controls were mounted on a single chassis along with a bucking circuit which permitted observation of field changes (via current changes) of .01 gauss. The magnet was oriented in such a way as to minimize the superimposed effect of the earth's field, and great care was taken during the course of the measurements to make certain that the magnet's orientation with respect to that field remained fixed. (The horizontal component of the earth's field in the San Francisco Bay region is approximately 0.26 gauss. Therefore, an inadvertent reorientation of the magnet between readings could seriously affect accuracy measurements). The random presence of ferro-magnetic-objects, such as tools, iron stock, etc., was also carefully checked during the course of the precision measurements.

A highly stable Colpitts oscillator and power amplifier was constructed to operate at a frequency of 49.0 mc., which, through the relation

(9) $$H_o = 0.357 \times 10^{-6} \nu \text{ gauss}$$

for electrons (derived from Equations (4) and (6)) corresponded to a resonant field of 17.5 gauss. The output of the power amplifier was fed through a matched line to a pair of transmitter field coils located in a conventional nuclear induction type probe. The transmitter field coils were also tuned to 49 mc., the entire arrangement being capable of producing an oscillating r-f field of 0.91 rms gauss in the sample region, at full power output of 5 watts.

Fields of any lower value could be produced by the insertion of dropping resistors into the B-plus lead to the transmitter. A hole in the probe permitted insertion of samples into the field, the samples generally being contained in standard test tubes.

The receiver coil in the probe was also tuned to 49 mc. and the output fed into a two-stage tuned low noise receiver. The detected signal was then amplified by a conventional 400-7000 cps wide audio amplifier, the latter circuit also having an optional narrow-band filter for oscilloscope observation of the 500 cps component of the signal and a discriminator for obtaining a d.c. signal from the slow passage through resonance. In most of the measurements signals were observed on a cathode-ray tube at the output of the wide-band audio amplifier.

The tuned r-f stages were broad-banded enough so that no appreciable loss of gain occurred for small deviations of the oscillator frequency from 49.0 mc. Therefore, by making slight manual adjustments of the oscillator frequency, and by using the fact that for electrons a one milligauss field shift corresponded to a 2.8 kc. frequency shift, determinations of the precision within which an observer could reset the resonant condition were made.

A view of the laboratory arrangement for low-field studies is given in Figure 1. The precession field magnet with the probe placed in the field and a sample placed in the probe is shown in Figure 2.

Signal amplitudes were obtained in a relative manner by simple measurement of oscilloscope trace heights. Absolute signal widths were obtained in two ways, the first and most obvious method being straightforward measurement with a ruler of the full-width of the observed oscilloscope signal at the half-maximum amplitude point on a trace whose peak-to-peak sweep in gauss was known from previous search-coil calibration. The second method had a con-

siderable value in that it provided a check on both the previously described width and amplitude measurements, and in addition permitted observation of widths in conditions of small signal-to-noise ratio were conventional measurements would be out of the question. Since the reader may be interested in making such determinations himself, the second method is described in some detail in the following four paragraphs.

If the harmonic content of the pure symmetric signal is analyzed by a Fourier expansion, it will be found that at exact resonance this mode consists solely of the even harmonics of the sweep frequency. Therefore, for a 500 cps sweep field, the fundamental of the symmetric signal is the 1000 cps component. The amplitude of the second-harmonic component is given by

$$(10) \qquad a_2/A_o = (4/\pi) \int_o^{\frac{\pi}{2}} \frac{\cos 2x \, dx}{1 + (p \sin x/h_o)^2}$$

in which

a_2 = peak amplitude of second harmonic signal

A_o = peak amplitude of total signal

h_o = half-width in gauss of the total signal at half-maximum amplitude

p = peak sweep field in gauss

$x = \omega_s t$

ω_s = angular sweep frequency

t = time

The integral (10) cannot be evaluated directly, but a numerical integration gives the result plotted graphically in Figure 3. It is seen there that the second harmonic signal amplitude continues to increase with increasing sweep field, until at a peak sweep field of 2.2 times the half-width of the true

signal, a maximum occurs. At this point, the second harmonic amplitude is 34 percent of the total signal amplitude. The second harmonic amplitude then drops off rather slowly with further increase of the sweep field.

This dependence of the second harmonic signal on the sweep field points to a simple way for determination of signal widths. The sample producing the signal of unknown line width is placed in the field, and, by means of leakage adjustment, a pure symmetric mode is obtained and set to exact resonance. The audio output which normally goes to the oscilloscope is then switched over to the input terminals of a narrow-band amplifier (in the present case a Hewlett-Packard Harmonic Wave Analyzer) which is tuned to 1000 cps. The sweep field amplitude is then adjusted until the output meter of the narrow-band amplifier reads a maximum. From the preceding analysis, it follows that for this optimum condition the full-width of the total signal is 1/2.2 times the peak-to-peak sweep field. A measurement of the sweep field for maximum harmonic and the application of a simple multiplying factor therefore provides a knowledge of the signal line width. Relative signal amplitudes may also be checked from the fact that for the optimum sweep condition, the second harmonic amplitude is 0.34 times the total signal amplitude. Therefore, the narrow-band meter reading at the maximized sweep condition is a relative measure of total signal amplitude for any signal regardless of width.

In situations where the signal may be only one or two times the noise, the method described in the preceding paragraphs may be the only possible one. The use of the narrow-band amplifier can greatly increase the signal-to-noise ratio and thereby enable the observer to make width measurements where a simple laying off of distances on an oscilloscope would be impossible. On the other hand, some difficulties may arise because the method depends on the ability to adjust the leakage to get a symmetric signal.

There are several methods of making certain that the frequency is set to the resonant value of the field being measured. The methods differ in the precision with which they enable one to adjust to resonance. All the methods rely on the fact that the center of symmetry of a symmetric signal due only to the "V mode" determines the resonance value. Thus all the methods depend on being able to adjust the leakage so that only the V mode is observed. It also was pointed out that even for a pure V mode the inhomogeneities of the field being measured can introduce asymmetries.

A simple method of setting to resonance is by visual observation of the signal on the oscilloscope. One adjusts the frequency until the signal appears symmetrical. The audio bandwidth can be reduced so that only the first harmonic is observed on the oscilloscope and thus the signal-to-noise somewhat improved. This does not increase the precision very much since even with the larger audio bandwidth the eye serves as an integrating device and hence reduces the bandwidth also.

A better method of reducing the bandwidth than narrowing the audio bandwidth is to use a phase detector or "lock-in amplifier" the output of which has a time constant long enough so that a very narrow band about the first harmonic frequency of 500 cps is examined. The signal-to-noise will be improved compared to wide band oscilloscope observation by the square root of the ratio of the bandwidths.

If the harmonic content of the symmetric mode signal is analyzed, a simple application of the Fourier Theorem shows that the first harmonic (frequency equal to the sweep frequency) has an amplitude relative to the total signal amplitude given by

$$(11) \qquad a_1/A_o = 2/\pi \int_{-\frac{\pi}{2}}^{\frac{\pi}{2}} \frac{\sin x \, dx}{1 + (r/h_o + p \sin x/h_o)^2}$$

In this expression the symbols have the following meaning:

a_1 = peak amplitude of first harmonic signal

A_o = peak amplitude of total signal

h_o = half-width in gauss of total signal at half-maximum amplitude

p = peak sweep field in gauss

r = deviation from resonance in gauss

$x = \omega_s t$

ω_s = angular sweep frequency

t = time

The evaluation of the integral must take the form of an approximation in the region of interest. Figure 4 is a plot of the shapes of first harmonic signals for various sweeps between 0.1 times the half-width of the total signal and 0.8 times the half-width of the total signal. The ordinate is the ratio of first harmonic amplitude to total signal amplitude, and the abscissa is the ratio of deviation from resonance to half-width of total signal. All the curves represent odd functions -- i.e., for negative deviations from resonance the amplitudes become negative (phase shift of π).

The principal feature to observe is that, for sweeps less than the half-width of the total signal, the amplitude of the first harmonic signal increases markedly with sweep amplitude at very little sacrifice in first harmonic signal "width". (Here "width" means the distance in gauss between the resonant condition and deviation from resonance for maximum first harmonic amplitude). Subsequent analysis indicates that the optimum first harmonic signal occurs for sweep amplitude equal to half-width of total signal, and that for sweeps larger than this the amplitude does not decrease too rapidly, but the "width" of the first harmonic signal broadens considerably. In other words, is one is interested in looking at first harmonic signals for precision measurement of

fields, one should use a peak-to-peak sweep equal to the full-width of the original signal before extraction of first harmonic.

This first harmonic signal can be observed with the Standard Fluxmeter using the meter marked Discriminator Output. Additional gain and thus an improvement in sensitivity can be had by using auxiliary equipment.

This first harmonic method was used to re-set a magnetic field to one part in 20,000 as described in a subsequent section on metal-ammonia solutions. An oscilloscope presentation of a truly symmetric signal was first obtained, the sweep called for in the previous paragraph was applied, and then a Hewlett-Packard Harmonic Wave Analyzer was used to reject all but the first harmonic of the signal. This narrowing of bandwidth increased the signal-to-noise ratio considerably. Then, in accordance with the curves of Figure 4, resonance was set by slightly varying the precession field until the output meter of the Harmonic Wave Analyzer read the zero between two peaks (the Harmonic Wave Analyzer read only absolute values, and could not take into account sign changes; therefore, a first harmonic signal was characterized by a dip to zero between two adjacent maxima). Of course, one might also have considered using the maximumat resonance exhibited by the second harmonic of the symmetric signal, but it turnes out that the second harmonic signal is somewhat wider than the true signal when a sweep is applied which produces maximum second harmonic amplitude.

In a manner similar to proton field control devices the first harmonic signal offers excellent possibilities for control and regulation of low magnetic fields.

A word of caution concerning an interesting effect should be interjected here. When one is hunting for resonances in low magnetic fields, extreme care may be necessary to avoid confusion with signals observed due to resonances in

the sweep field. If, for example, the probe containing the paramagnetic sample is removed from all d.c. fields and a peak-to-peak sweep field of 20 gauss is applies, two signals symmetrically located with respect to the center of the oscilloscope trace will appear for all transmitter frequencies below 28 mc. Obviously, the reason for this is found in the fact that the sweep field, in passing from zero to + 10 gauss, back to zero, and then to -10 gauss, provides just the field required for resonance with the existing radio-frequency (10 gauss corresponds to a resonant frequency of 28 mc. for electrons). This effect is immediately recognizable, and a simple way to eliminate confusion is to use peak-to-peak sweep fields which are less than twice the precession field required for resonance with the existing radio-frequency.

Measurements and Results

In the summary which follows, the reader must bear in mind that some of the results are necessarily of a qualitative nature, and that the emphasis has been placed on the solution of the particular field measuring problem which forms the purpose of this report. In particular, no attempt was made to measure electron g-values at low fields, principally because of the difficulty of establishing magnetic field magnitudes to a high degree of accuracy. Such measurements are much more readily carried out at fields of the order of 3000 gauss, in which region the magnitude of the field may be conveniently determined from a simple proton nuclear induction experiment. The principal purpose of the present series of experiments was to find substances which would produce paramagnetic resonance signals, and, once found, to investigate line widths, signal amplitudes, chemical stability of samples and usefulness of signal for field measuring purposes.

This section of the report has been divided into several sub-sections,

each of which is devoted to a particular material investigated for paramagnetic resonance. The results include data received as the result of consultation with other workers in the field of paramagnetic resonance.

Organic Free Radicals

The first paramagnetic resonance was observed using a sample consisting of the organic free radical diphenyl-trinitro phenyl hydrazyl $(C_6H_5)_2N\text{-}NC_6H_2(NO_2)_3$. This substance, of molecular weight 394, has been found to be stable. The signal was observed in a precession field of 17.5 gauss at a resonant frequency of 49.0 mc. The best width measurements gave a value for the full-width of the signal at half-maximum amplitude of 2.2 gauss. Using the tuned probe described in a previous section and a 0.3 cubic centimeter sample an estimated signal-to-noise of 800 was obtained for the largest available r-f power (which was estimated to be close to the maximum required).

The 0.3 cc. sample was sealed and used in the experiments as a standard of comparison. For simplicity, in subsequent paragraphs and sections this standard sample will be referred to as the "hydrazyl sample".

An untuned probe was constructed which could be used with the standard r-f units of a Varian Associates Model F-6 Nuclear Fluxmeter. With this probe the 2.25 - 4.5, 4.5 - 9, 9 - 18 and 18 - 23 kilogauss r-f units were changed to cover ranges of approximately 3.4 - 6.8, 6.8 - 13.6, 13.6 - 27.2 and 27.2 - 35 gauss respectively. The signals were excellent down to the lowest fields measured, namely 3 gauss. Since line width was large compared to the precision required and the leakage balance adjustments were not adequate to insure a pure V mode some difficulty was encountered in using this probe.

Several laboratories were visited which were working with free radicals. The hydrazyl free radical used was discussed in an article by Holden, Kittel, Merritt and Yager[6].

The unusual narrowness of the line is ascribed to the Gorter-Van Vleck process of exchange narrowing. The character of this process seems to be such that the narrower lines are associated with the more stable organic free radicals. The exchange narrowing is evidenced by the widening of the line width with dilution. During a visit to Prof. Clyde A. Hutchinson, Jr., of Chicago he pointed out that a 0.0002 moles/liter hydrazyl solution gave a line width of 20 gauss. The advantage of such compounds are that while they give relatively narrow lines they have a high concentration of free electrons and hence give large signals. Up until recently not many of these compounds have been examined for the property of line width. It was the opinion of Dr. Kittel that it was not impossible or improbable that if the absorption widths of enough compounds were observed one might be found with even narrower line widths then the hydrazyl. As a result of consultations with Dr. Kittel, Prof. Dicke of Princeton, Prof. Hutchinson of Chicago, and Dr. Garstens of the Navel Research Laboratories, who kindly supplied the hydrazyl samples used, we concluded that the hydrazyl compound gave the narrowest line width of the organic free radicals studied at this time.

A communication which arrived during the writing of this report indicated the discovery of a free radical with a line width of 0.35 gauss. The information from Mr. N. O. Schuster of the laboratories of Prof. Geroge Pake at Washington University, Saint Louis, was that an 80 milligram sample of tri (para nitro phenyl) methyl gave large signal-to-noise ratio at a precession field of 2 gauss and a line width of 0.35 gauss.

Solutions of Alkali Metals in Liquid Ammonia

The first paramagnetic resonance signals from these solutions were observed in open test tubes at a frequency of 49.0 mc. in a field of 17.5 gauss.

Since the boiling point of ammonia is -34 deg. C., and since therefore the rapid boiling away of ammonia caused indeterminate concentration changes during the course of the observations, the first experiments were necessarily limited to mere observance of the existence of signals with both sodium and lithium as metallic solutes. On the other hand, it was immediately evident that extremely narrow signal widths (of the order of 0.1 gauss) could be obtained from metal-ammonia solutions, and therefore immediate proparation of a sealed Faraday tube was begun under the direction of Professor R. A. Ogg, Jr. of Stanford University.

A note in handling these solutions may be interjected at this point. In working with sealed tubes containing metal-ammonia solutions, considerable precaution is recommended in that the pressure in a closed vessel at room-temperature rises to approximately 10 atmospheres. It is therefore advisable to use extreme care in handling the sealed glass samples, and the wearing of a face mask is strongly recommended. In certain types of "low Q" untuned probes, some heating may occur due to the large amount of power required to set up the radio-frequency field. In this case the probe should be designed so that the pressurized sample experiences a miminum terperature rise, as a 10 deg. C. rise in the region of room temperature can result in a nearly two-fold pressure rise.

The life of these samples can be greatly extended by storing them when not in use in a dry-ice acetone bath (-78 deg. C.). Such storage not only prolongs sample life, but also reduces the vapor pressure to a negligible value and thereby lowers the probability of glass breakage. Under the best care, the life of these samples at room temperature is not expected to exceed two or three months due to an irreducible velocity of decomposition. This lifetime figure is not very well known. At the time of writing of this report, two samples prepared six weeks prior to this date have not shown measurable deterioration.

The Faraday tube was a sealed v-shaped glass tube which contained a fixed amount of liquid ammonia and a fixed quantity of the metal. In this particular case the metal was sodium. The concentration of the solution could be varied at will by simple distillation techniques, and temperature effects could be observed by placing the tube in several calibrated baths, one of which was that somewhat variable temperature bath defined as "room temperature". Again, it should be emphasized here that the results were of a qualitative nature----- for example, the concentrations of sodium-liquid ammonia solutions in the Faraday tube were judged to order of magnitude only by coloration estimates. The nature of the program made it inadvisable to prepare numerous separate sealed samples each of known measured concentration. A quantitative investigation would serve a valuable purpose in helping to explain the origin and behavior of paramagnetic resonance in metal-ammonia solutions.

Repeated measurements with the Faraday tube indicated that the amplitude and width of the electron signal from sodium-liquid ammonia were strongly dependent upon concentration and temperature. In considering the amplitude effects first, it was observed that when the tube was placed in the probe directly from a dry ice-acetone bath the signal was at first barely visible, but that as the solution warmed the signal grew, continuing to do so up to room temperature. In other words, metal-ammonia solutions have susceptibilities exhibiting a temperature behavior opposite to that prescribed by Curie's Law. It was also observed that the ratio of signal amplitude at room temperature to that at dry ice-acetone temperature was markedly dependent upon concentration, being much greater for high concentrations than for low.

The signal amplitude was strongly dependent upon concentration, being barely visible at a concentration of the order of 10^{-4} M, growing until a maximum amplitude was reached at approximately 0.1 M, and decreasing in amplitude

with further increase in concentration. It is entirely possible that this optimum concentration effect may not be due to any decrease for high concentrations in the number of "free" electrons available for "flipping", but that it is due to the rather marked increase in electrical conductivity of the solutions at high concentration. Such high conductivities (of the order of magnitude of that of mercury) would affect the penetration of the sample by the radio-frequency field, the Q of the resonant circuit, and thus the sensitivity of the system. That this may indeed be true was evidenced by the change in both the phase and amplitude of the r-f "leakage" from the transmitter to receiver coil.

At room temperature and concentrations below the maximum amplitude concentration described above, the natural full-width of the sodium-ammonia line was found to be 0.08 gauss. By continued increase in concentration beyond the maximum signal point, a considerable broadening was observed, attaining values as high as 0.7 gauss-----or nearly 9 times that of the narrowest line. In making these broad lines, some anomalous behavior was noted in the so-called "two-phase" region of the phase diagram of sodium and liquid ammonia. Below certain temperatures in this region, the solution physically separates into two liquid phases, the less concentrated heavier solution sinking to the bottom of the sample container. Since the design of the probe was such that the receiver and transmitter coils only "saw" that portion of the sample lying in the bottom of the tube, the signal appeared to be the very narrow one (0.8 gauss) from the dilute phase. However, the broad signal was immediately obtained when the Faraday tube was removed from the field, warmed, shaken thoroughly to mix the two phases, and then quickly replaced in the field.

At concentrations below the maximum amplitude concentration, the width of the signal was observed to decrease by an approximate factor of 2 in warming from dry ice-acetone temperature (-78 deg. C.) to room temperature.

From what has been described qualitatively above, it must be obvious to the reader that the behavior of metal-ammonia solutions with respect to temperature is an extremely favorable one from the point of view of field-measuring convenience. In using the maximum amplitude concentration, the signal both narrows and grows as the temperature approaches that of the room from that of the dry ice-acetone bath. The one drawback, of course, is the rise in pressure as the temperature reaches and passes the liquid ammonia boiling point of -34 deg. C. Nevertheless, it was felt that it would be a relatively simple matter to form and seal off a glass capsule capable of withstanding 10 atmospheres. Therefore a sample, which will be referred to hereafter in this report as the "sodium-ammonia sample", was sealed off, the volume being approximately 0.3 cc and the concentration approximately 0.1 M. (If or when the electron signal from this sample disappears, its concentration will be carefully measured by quantitative methods).

Considerable data concerning the sodium-ammonia sample were taken. Using the probe described in Section II, a signal-to-noise ratio at room-temperature of 25 was observed with an oscillating H_1 field of approximately .03 rms gauss. The full-width was measured to be .08 gauss. A series of measurements were taken at room temperature to investigate the effect of H_1 field magnitude on broadening and amplitude. The results are tabulated below.

Table I

H_1	Full-width	Signal Amplitude
.01 rms gauss	.08 gauss	0.22 arbitrary units
.03	.08	0.64
.05	.09	0.88
.08	.11	1.00
.15	.23	0.82
.48	.33	0.27

It is apparent from the table that maximum amplitude without regard to broadening occurs at an r-f field of approximately .08 rms gauss, whereas maximum amplitude in conjunction with no boradening occurs at an r-f field of approximately .03 rms gauss.

Another measurement with the sodium-ammonia sample yielded the following data concerning the effect of temperature on amplitude and width:

Table II

Temperature	Full-width	Signal Amplitude
-78 deg. C.	0.13 gauss	.08 arbitrary units
0 deg. C.	0.08	.79
room (approx. 21 deg. C.)	0.08	1.00

The table clearly demonstrates the width and amplitude temperature effect for the particular concentration of the sodium-ammonia sample.

A measurement was made to determine the accuracy within which one could re-set a particular homogeneous precession field using the sodium-ammonia sample in the previously described tuned probe. The experiment was carried out in a precession field of 17.5 gauss at a resonant frequency of 49.0 mc., with a peak-to-peak sweep of 0.3 gauss and an oscillating r-f field of .03 rms gauss. Employing the straightforward visual method of setting the signal to a given position on the oscilloscope, a precision of one part in 5000 was easily attainable. By using the method which was described in Section II of this report and which depended upon the observation of the harmonic of the signal, an ability to re-set a field to one part in 20,000 seemed possible.

To investigate the possibility of using metal ammonia solutions for general low-field exploration and measurement, a special untuned probe was designed and constructed for the sodium-ammonia sample. A photograph of this probe appears in Figure 5. The low-Q feature of the probe permitted its use with the conventional r-f units of a Varian Associates Model F-6 Nuclear Fluxmeter. The device was used extensively in a low-field exploration, but unfortunately this particular application involved measurement of fields which were everywhere considerably more inhomogeneous over the sample than the natural line width of the signal. The consequence was that the narrow-line advantage of metal ammonia was somewhat lost, and precision to .04 gauss in the presence of field inhomogeneities of 1 gauss/in. was all that was attained. (Fields of the order of 20 gauss were being measured). Signal-to-noise ratios in the neighborhood of 4 to 1 were observed.

Metal-ammonia solutions offer considerable promise as samples in untuned probes in low-field measurement applications were the inhomogeneities over the sample do not appreciably exceed 0.1 gauss. For such applications, full advantage may be taken of the narrow-line characteristic of a properly prepared metal-ammonia solution. Much greater signal-to-noise ratios can be obtained with a tuned probe, but the clumsiness of its use and possible physical limitations of size in the field to be measured may rule out a probe of this sort.

In describing the characteristics of metal-ammonia solutions, some comparison between the paramagnetic resonance signals produced by the hydrazyl

sample and the sodium-ammonia sample may be valuable. The two samples had equal volumes of 0.3 cc. The comparison was made at a frequency of 49.0 mc. in a precession field of 17.5 gauss, using the tuned probe described in Section II. The sweep frequency was 500 cps, and in each case a peak-to-peak sweep amplitude was employed which was 2.2 times the full-width of the signal under investigation. The result is tabulated below:

Table III

H_1	Signal Amplitude from Sodium-Ammonia		Signal Amplitude from Hydrazyl
.03 rms gauss	1.00	All Amplitudes measured in the same arbitrary units	0.95
.05	1.40		1.90

It will be noted that when only small r-f fields are available and narrow lines are of advantage, the experimenter should by all means use a metal-ammonia sample if precision is the prime requirement. However, an increase in the r-f field beyond .05 gauss will cause an increase in the hydrazyl signal, whereas the saturation effect will set in for the metal-ammonia resonance. For optimum adjustment of the r-f field and the same size sample we have the following observed ratio:

$$\frac{\text{Signal amplitude from hydrazyl}}{\text{Signal amplitude from sodium-ammonia}} = \frac{800}{25} = 32$$

There are several references on the properties, preparation, and handling of liquid ammonia solutions. There is a series of articles on this subject by W. Johnson and W. C. Fernelius which has appeared in the Journal of Chemical Education[7]. Recent work on paramagnetic resonance in metal ammonia solutions has been reported by Hutchison[8] and Garstens[9]. These experiemnts confirm the results reported here. Conversations with Prof. Ogg of Stanford and Prof. Hutchison of Chicago indicate that if prepared with great care the metal ammonia solutions will be stable enough to last one to three months. While no very great effort had been made in this direction, it seems that no one has made solutions which have remained stable for longer periods.

Solutions of Lithium in Methylamine

As in the case of the metal-ammonia solutions, the first observation of a signal from lithium in methylamine (CH_3NH_2) was made using an open test tube. Such experiments are extremely difficult to perform because of the pervasive foul odor (similar to that of decaying fish) which emanates from methylamine. However, the boiling point of this organic liquid (-6.5 deg.C.) is such that the pressure in a closed container is fairly low at room temperature (approximately 2 or 3 atmospheres). A Faraday tube was constructed, and the investigations indicated that the dependence of width and amplitude on temperature and concentration was generally similar to that of the metal-ammonia solutions. An increase of width and amplitude with concentration was observed, the amplitude reaching an optimum value in the neighborhood of 1 M. Lithium-methylamine showed a less pronounced temperature effect on amplitude than was shown by metal-ammonia solutions, a typical ratio of amplitude at room temperature to amplitude at dry ice-acetone temperature being 1.3. Full-width measurements ranged between 0.4 gauss and 0.7 gauss, the former value occurring for a dilute

solution. An estimated signal-to-noise ratio using the tuned probe was 45. An anomalous behavior, similar to that displayed by metal-ammonia, was observed in the "two-phase region."

A small, 0.3 cc., 1.2 M, sample of lithium-methylamine was sealed off for investigation as to use with the untuned probe of a Model F-6 Nuclear Fluxmeter. Using comparable sweep amplitudes and equal r-f fields (.03 rms gauss) the signal from the lithium-methylamine sample was found to be approximately twice as large as that from the sodium-ammonia sample. Its somewhat greater width made it better suited for moderately inhomogeneous fields than the sodium-ammonia sample. The lithium-methylamine sample was actually used for several hours with considerable success in an exploration of an inhomogeneous low field. It was estimated that precisions were achieved of $\pm$ 0.02 gauss for 20 gauss fields in the presence of field inhomogeneities of 3 gauss/in. Unfortunately after this time the signal suddenly disappeared due to chemical deterioration. This event occurred 24 hours after the sealing off of the small sample, and only after the sample had necessarily been exposed to room temperature for several hours. A Faraday tube containing lithium-methylamine was kept for at least four days, with good signals being seen at the end of that period. The principal difference here, though, was that the Faraday tube was exposed to room temperature only for very brief periods during signal observation, and was at all other times carefully kept at -78 deg. C.

Since the measurement and exploration of magnetic fields obviously requires considerable periods of operation at room temperature, it would seem evident that, unless methods of chemical preparation can be greatly improved lithium-methylamine solutions do not offer considerable promise in low-field measurement work.

In the case of the solutions of alkali metals in liquid-ammonia, the method

of preparation was to make several vacuum distillations of the metal as well as the liquid-ammonia. Since the melting point of lithium is considerably higher than sodium, much more elaborate techniques would have to be used to prepare the lithium-methylamine solution to the same purity as the sodium-ammonia solutions. Inasmuch as such improved techniques would, if the samples were sufficiently stable, give the desired precision, an investigation of the techniques of preparation seems worthwhile. In any case, further investigation of the characteristics of lithium-methylamine solutions would shed light on the mechanism of paramagnetic resonance.

Solutions of Lithium in Ethylenediamine

Paramagnetic resonance was observed in an open test tube solution of lithium in ethylenediamine ($NH_2CH_2CH_2NH_2$). Signal widths of the order of 0.6 gauss (full-width) were seen. The extreme difficulty of preparing stable solutions led to the abandonment of further research with this substance on the present contract. The literature on the preparation of such solutions seemed meagre, and therefore considerable time, effort and experimentation would have been required for preparation of a sealed sample.

Materials with Negative Result

In addition to the substances described above which produced electron signals, a number of other materials were investigated with negative result. In many cases the failure to observe signals may have been due to the fact that the investigation was performed at low fields. An enormous gain in sensitivity can be obtained by working at microwave frequencies and high fields. In addition, extremely broad lines can be resolved much more readily at high fields.

In any case, the following materials appear to be useless for low-field work:

Silicon

Germanium

Iron Sulfide

Copper Sulfide

Lead Sulfide

Titanium Hydride

Zirconium Hydride

Potassium Super-Oxide

Oxygen (gaseous)

F-center Crystals[10], prepared both by x-radiation and by chemical means.

Conclusions

The apparatus actually tested in the fields to be measured almost meets the requirements for precision as outlines in the beginning of this report. The difficulties that remain are mainly due to the field inhomogeneities. With the sample sizes used it has been observed in the best part of the field that, with the metal-ammonia solution as a paramagnetic media, the line width was 0.7 gauss. This width, being almost a factor of ten greater than the width observed in a homogeneous field, must be essentially a measure of the field gradient across the sample. There are several possibilities which exist for improving the precision in the most inhomogeneous of the fields to be measured. They are listed below in the order of merit.

(1) If it is established that the organic free radical tri (para nitro phenyl) methyl has a full width at half amplitude of 0.35 gauss, and has an

amplitude comparable with the hydrazyl signal, use of this as an paramagnetic media will certainly give the required precision.

If one wanted to decrease the precision with which one had to read within the line width to get the required accuracy, one would be able to use a sample of this organic free radical approximately 1/100 the volume of that previously used. This would give a line width one-half of that presently observed even in the fields with inhomogeneities of 3 gauss/in. The signal-to-noise under these conditions would remain approximately the same as with the metal-ammonia sample in the best field as observed with the low Q probe. The precision could be increased still further by using a tuned, or high Q, probe with the disadvantages that more adjustments would be required and the leakage balance would be more critical. If one did not change the sample size, one could use the same probe as used for the metal-ammonia sample. The line width would remain the same as presently observed (0.7 gauss in the best field and correspondingly greater in the more inhomogeneous fields). The signal-to-noise ratio would be approximately one hundred times improved and by reading within the line width the required precision should be achieved provided the necessary care was taken with balancing, and the shape of the field did not introduce too large an asymmetry.

(2) The metal-ammonia sample could be used in a high-Q probe. It would be reasonable to expect Q's of the order of fifty. If the noise observed was all Johnson noise this would give an improvement in signal-to-noise of approximately seven. If the noise was largely hum or spurious 500 cps signals the improvement observed would be greater. This, as any tuned probe, would have the disadvantages pointed out previously. The signal-to-noise ratio in any case should be improved sufficiently to get at least the same precision in the 3 gauss/in fields as now observed in the best fields. One could reduce the

sample size by a factor of Q and get a line width approximately one-half that observed with the metal-ammonia sample and at the same time get an increase of about a factor of two in the signal amplitude. This would be expected to just barely give the required precision in the worst field but would have the advantage of less critical balancing adjustments. It would have the disadvantage of requiring a new probe. It is possible also that the use of high-Q probes would require modification of the existing r-f units used.

(3) A hydrazyl sample could be used which was made sufficiently large so that the field broadening in the best field was of the same order as the natural width of 2.2 gauss. This would give an improvement in signal-to-noise ratio over the metal ammonia sample of at least 300: 1. If enough care were taken in balancing this should give the required accuracy. It would have the advantages of using low-Q probes and the operational disadvantages of reading well within a line width. There is also the possible disadvantage that at the lowest fields to be measured a correction factor would have to be used to take account of the perturbation due to using an oscillating field instead of a rotating field.

(4) It is possible that further work on the preparation of the lithium solution in methylamine would result in a sample of sufficient stability to be useful. This would result in at least a four to one improvement in signal-to-noise compared to the metal-ammonia sample as measured in the 1 gauss/in. field. This larger natural width would mean there would be less of a reduction in signal-to-noise in going to the 3 gauss/in. fields than observed for the metal-ammonia sample. This would probably give the required precision.

There are many investigators performing experiments with paramagnetic resonance effects. More understanding of line widths should result from these studies as well as the discovery of new media with narrow lines.

The most useful discoveries from the point of view of field measurements

will be the finding of new organic free radicals such as the hydrazyl compound or possibly the tri (para nitro phenyl) methyl which have a high density of free electrons but with even more exchange narrowing. Such discoveries will immediately lead to more precision in field measurements. In very inhomogeneous fields there is an optimum line width determined by the field gradient across the smallest size sample which will give a detectable signal.

In addition to the continued investigation of new paramagnetic media, a program for future work should include measurements of g-factors so that absolute field measurements can be made. At high fields, the g-factor for free electrons in diphenyl-trinitro phenyl hydrazyl[6] and potassium in liquid ammonia[8] has been measured.* These measurements should be made at low fields so that no question of field dependence arises. Proton resonances have been observed in fields as low as 10 gauss, so that it is entirely possible to measure the electron g-factors in terms of the proton moment at low fields. Thus, absolute measurements of fields can be made to the precision with which the g-value of the proton is known.

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*Using the latest value for the gyromagnetic ratio of the proton and the results of references 6 and 8, one obtains the following relations:

1. Diphenyl-trinitro phenyl hydrazyl

   $H_o = (3.5654 \pm .0008) \times 10^{-7} \times \nu$ gauss

   (Average of two measurements made at 24,000 mc. in a field of approximately 8560 gauss)

2. Potassium in Ammonia

   $H_o = (3.5701 \pm .0006) \times 10^{-7} \times \nu$ gauss

   (Measurement made at 23,700 mc. in a field of approximately 8460 gauss)

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Figure Captions

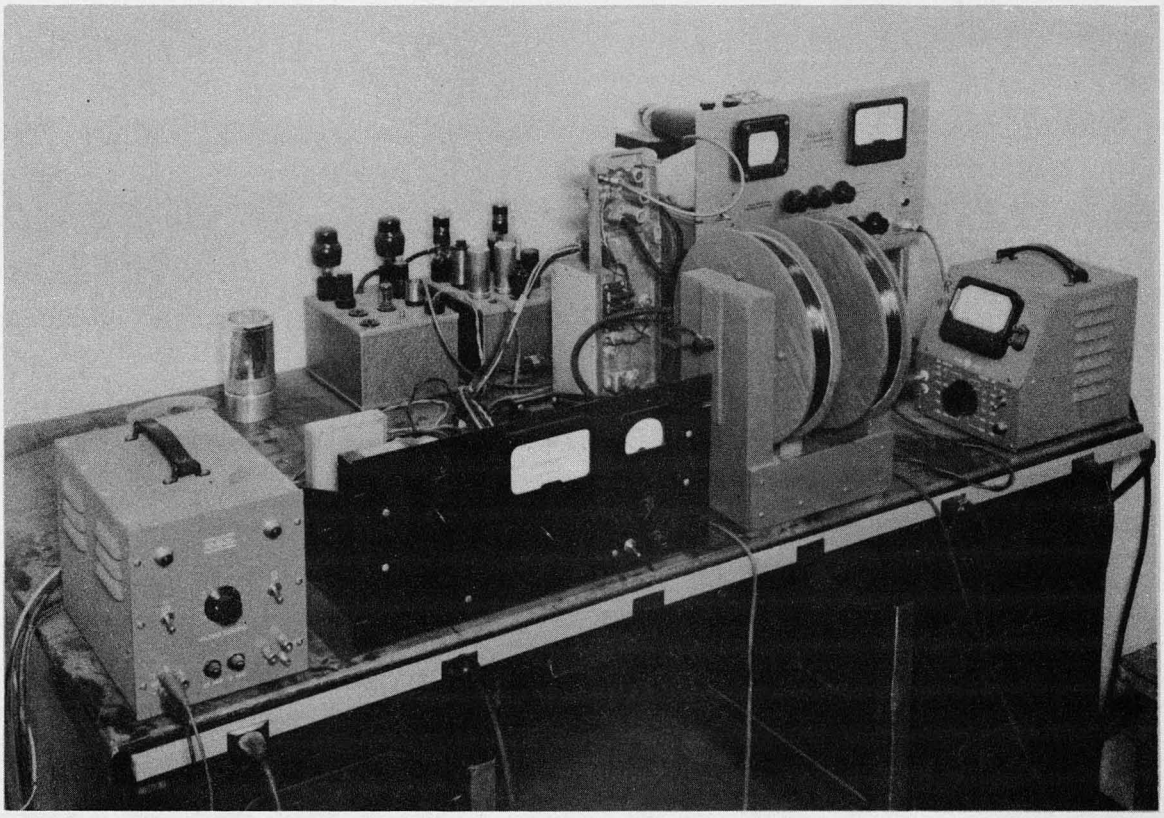
Figure 1 Overall view of laboratory arrangement for low field measurements

Figure 2 View of precession field magnet, with probe and sample inserted in the field.

Figure 3 Second harmonic signal amplitude at resonance vs. sweep field amplitude.

Figure 4 First harmonic signal amplitude vs. deviation from resonance for several different sweep field amplitudes.

Figure 5 Three views of the special low-Q probe designed for field measurement with the sodium-ammonia sample. The upper photograph shows the sweep coils, paddle adjustment and hole for insertion of sample. The bottom photographs show the probe with lids removed, revealing the lucite block containing the transmitter and receiver coils, and the various resistors and capacitors associated with the low-Q circuit.



ZN 32

Figure 1

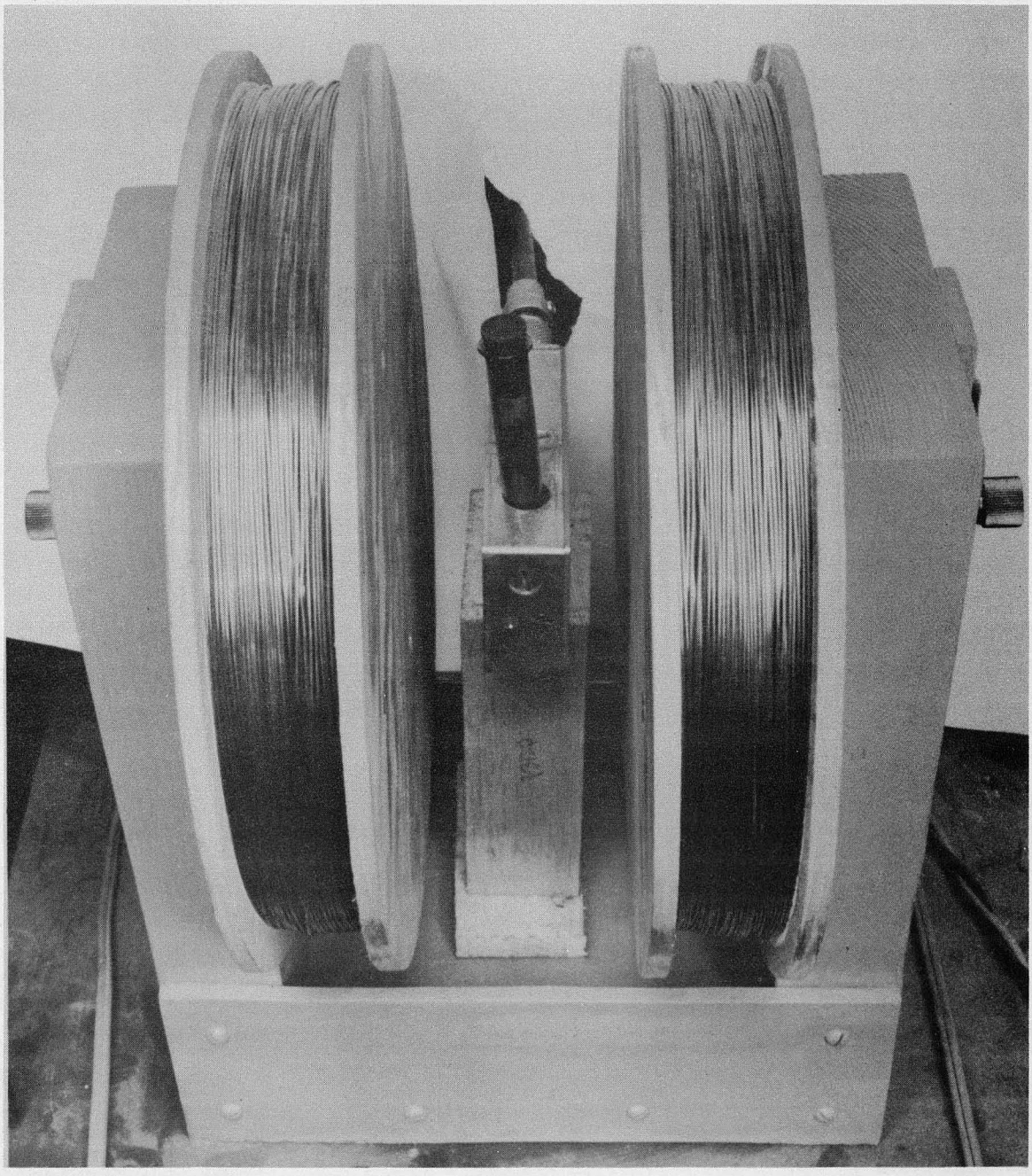
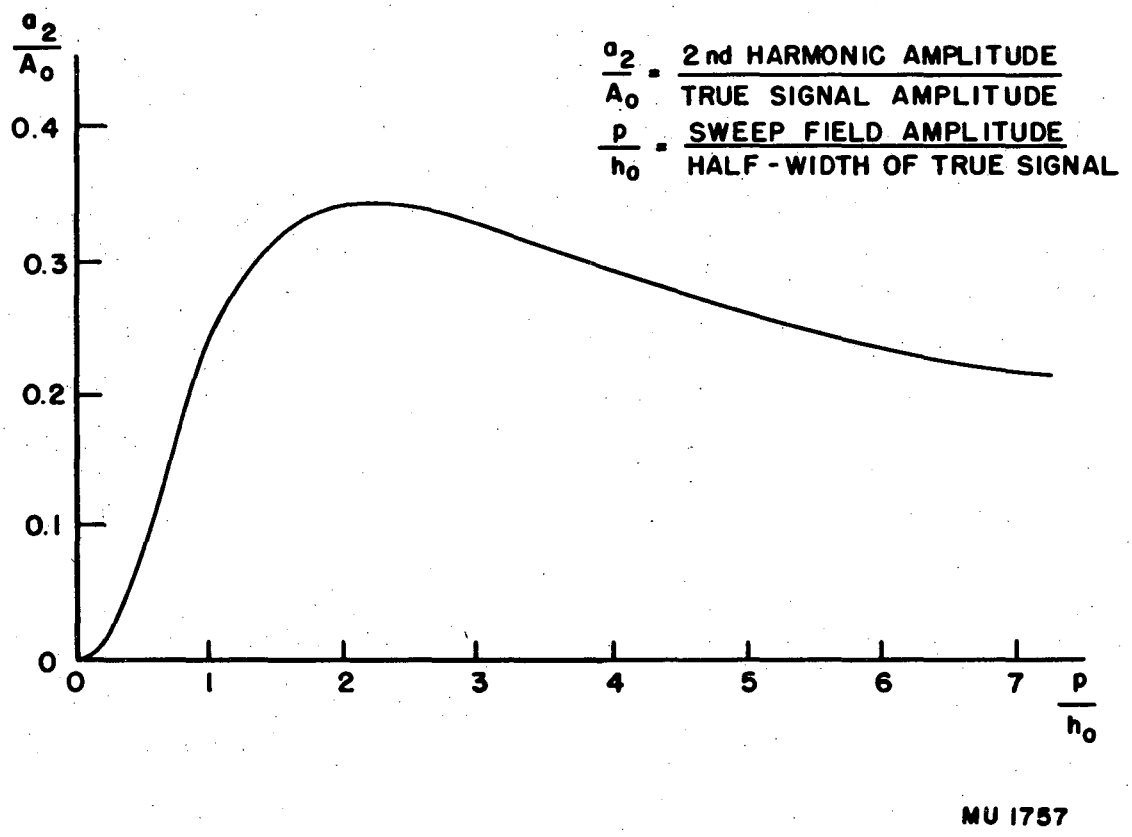


Figure 2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1
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3
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7
$\frac{p}{h_0}$
$\frac{a_2}{A_0}$ = 2nd HARMONIC AMPLITUDE / TRUE SIGNAL AMPLITUDE
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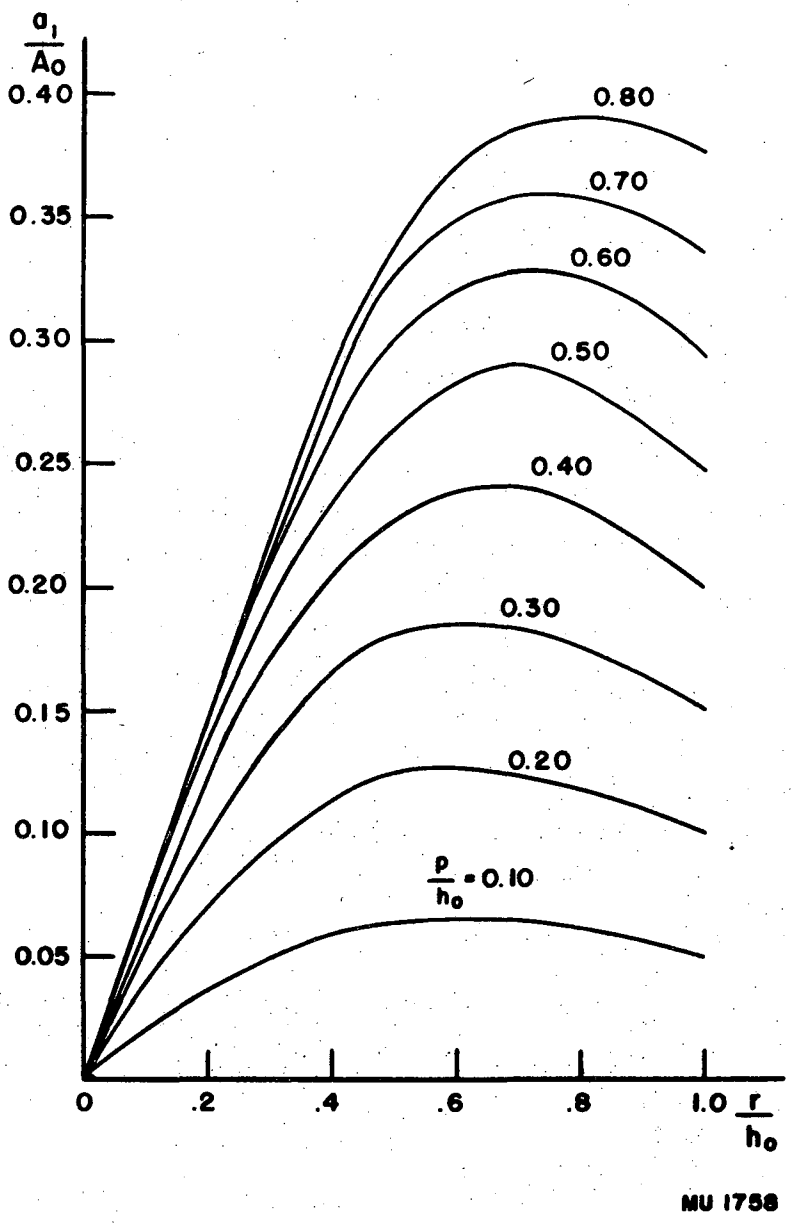
MU 1757

Figure 3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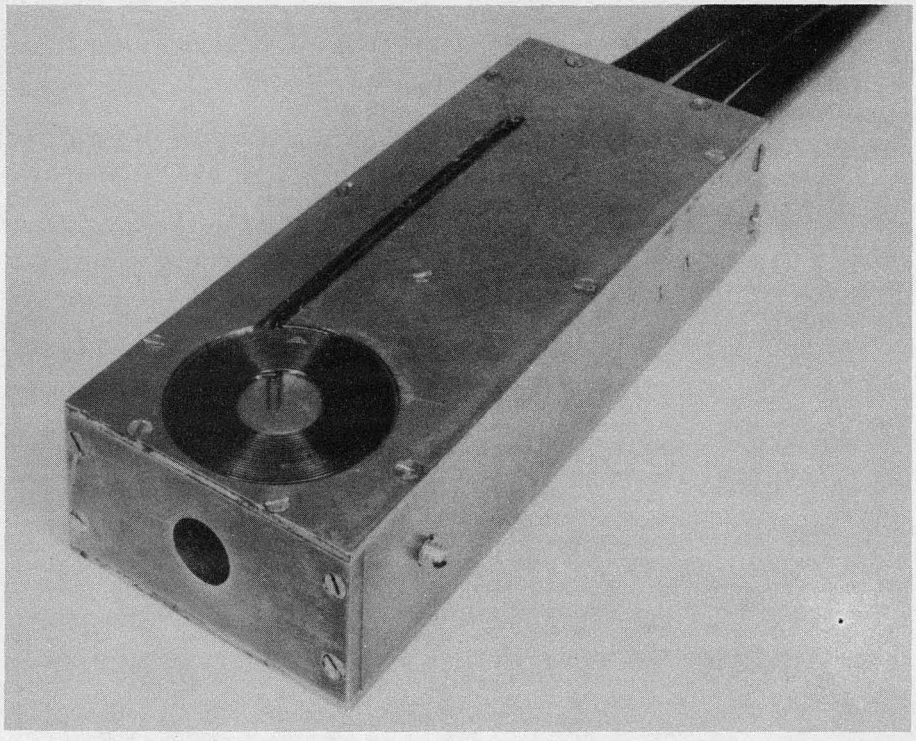
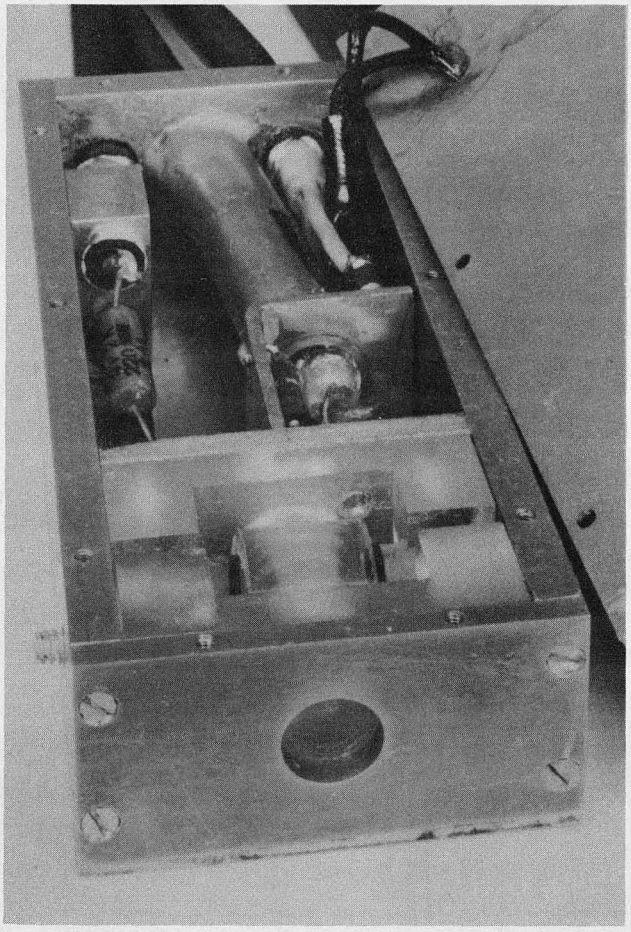
MU 1758

Figure 4





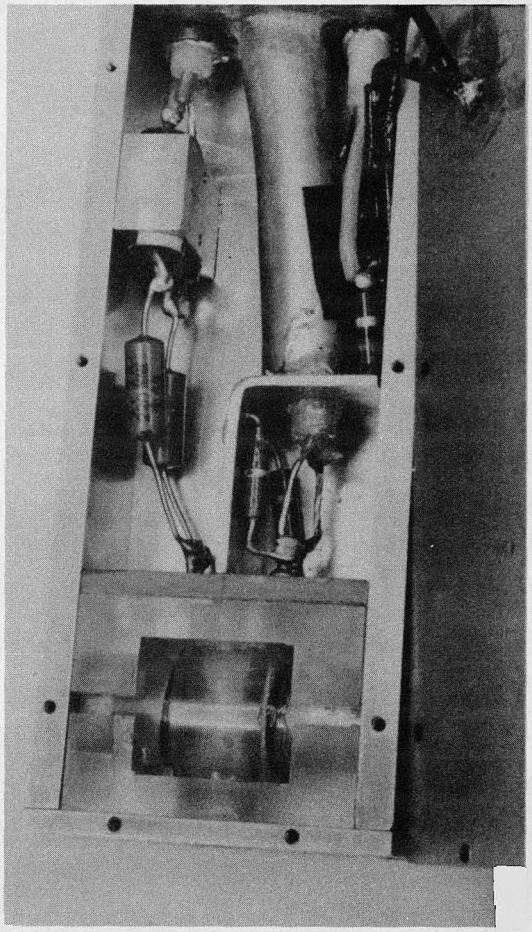


Figure 5