Magnets and Magnetic Field Measurements

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The discovery of nuclear magnetic resonance by Bloch and Hansen (*1*) and by Purcell, Torrey, and Pound (*2*) in 1945 ushered in a new era in the development of laboratory magnets and magnetic measurements. Not only did it become possible to measure magnetic fields with ease to a much greater degree of precision, but the magnetic resonance experiments themselves demanded magnets with much more exacting requirements of field homogeneity and stability. This article is concerned with some of the new requirements that are placed on magnetic fields by nuclear magnetic resonance and the techniques for measuring these fields. The magnets under consideration are laboratory-sized magnets used for general experimental work wherever strong fields are needed in a relatively small volume. We do not discuss magnets of highly specialized design such as large cyclotron magnets.

Precision Measurements

The measurement of magnetic fields to high precision has not always been an easy task. Until recently, the only convenient methods of measuring magnetic fields have been classical ones such as the flip coil with ballistic galvanometer and the rotating coil. These methods are still useful, but even with recent improvements in technique they are capable of an accuracy of only about 0.1 percent, assuming proper calibration. More modern instruments that are occasionally used in strong fields include Hall-effect detectors for which an accuracy of about 5 percent is claimed (*3*) and detectors that employ changes in the ohmic resistance of bismuth as a function of the magnetic field. The latter detectors, when they are calibrated and operated under constant-temperature conditions, give an accuracy of 1 part in 5000 (*4*). In weak fields, such as the earth's magnetic field, the flux-gate magnetometer (*5*), a null-measuring instrument, is capable of high relative accuracy. This type of instrument often has a noise level of less than 10^{-5} gauss, but the actual accuracy is only as good as the known value of the bias field that is used to produce the null condition.

Nuclear magnetic resonance (NMR) makes available to the experimenter an instrument that converts magnetic field measurement into frequency measurement, one of the most easily measurable quantities. If the magnetic field is H, then the resonant angular frequency ω is given by

$$\omega = \gamma H,$$

where γ is the gyromagnetic ratio of the nucleus, which is given by the expression

$$\gamma = 2 \pi \mu / I h,$$

where μ is the magnetic moment of the nucleus, h is Planck's constant, and I is the spin of the nucleus expressed as a multiple of a half-integer. Because the frequency is related to the field only through atomic constants, which are fixed for the nuclei of any given isotope, a field-measuring instrument using nuclear magnetic resonance requires no calibration in the sense of initial comparison with a known magnetic field. Frequency standards, in the form of quartz crystal oscillators, on the other hand, are available in almost every laboratory and can easily be compared with primary frequencies broadcast by radio station WWV.

The standard techniques of inducing resonance by continuous radio-frequency excitation (*6, 7*) work best in fields of from approximately 200 to 20,000 gauss. At the low end, the limitation is the sample volume required to give a usable signal (for fixed sample volume, the voltage induced by the nuclei in the coil is proportional to the square of the field). At the high end one finds that the fields produced by existing magnets are usually not sufficiently homogeneous to justify an attempt at a precise measurement. Recently an instrument has been built that uses proton magnetic resonance to measure weak fields such as the earth's magnetic field (*8*). Here no exciting radio-frequency field is used. Instead, the nuclei are polarized perpendicular to the earth's field by a stronger field and are then allowed to precess freely in the earth's field; it is this precession frequency that is measured directly.

The limit of the *absolute* accuracy to which a magnetic field can be measured by nuclear magnetic resonance is the accuracy to which the gyromagnetic ratio γ is known. By an absolute measurement, we mean one in which the measuring instrument is not initially calibrated in a known "standard" field. The most accurate measurement of the proton gyromagnetic ratio, by Thomas, Driscoll, and Hipple (*9*), is listed with an uncertainty of 1 part in 40,000; this is the best that can be expected of an absolute measurement by nuclear magnetic resonance at the present time (*10*). One must remember that nuclear magnetic resonance is basically a form of spectroscopy, and, as in other forms of spectroscopy, one is always uncertain about the exact location of the center of the line (or resonance) by approximately the natural width of the line. However, the natural line width in nuclear magnetic resonance spectroscopy can be so narrow that this uncertainty is much less than the uncertainty in the absolute value of γ. The natural line width is the inverse of the transverse relaxation time T_2 in a homogeneous field. One of the longest transverse relaxation times known at present is for protons in pure benzene (*11*) and is of the order of 15 to 20 seconds, equivalent to a width in magnetic field units of 2×10^{-6} gauss.

In *relative* or comparison field measurements, the situation regarding accuracy is quite different. If line width considerations are neglected, the accuracy with which one can compare two magnetic fields is limited only by the accuracy with which one can compare frequencies. Even if the lines are relatively broad, one can make assumptions regarding their structure that will remain constant from one measurement to another and so allow a reduction of the uncertainty to a small fraction of a line width. For example, in many experiments, the method of presentation introduces transient oscillations of the nuclear polarization that complicate the line shape considerably. Jacobsohn and Wangsness (*12*) have shown how simple symmetry considerations allow an exact determination of the center of the resonance even in the presence of a large number of such oscillations. Another example occurs in the case of free precession in the earth's field. Here one expects, from both experimental and theoretical considerations, that in a moderately homogeneous field the signal output will take the form of an exponentially damped sine wave. Use is made of this fact to allow measure-

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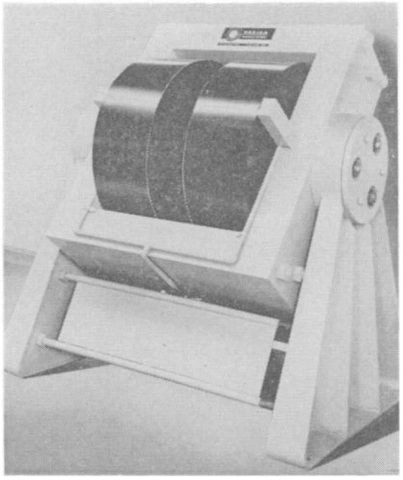

Fig. 1. Varian 12-inch electromagnet, an example of double-yoke construction.

ment of changes in the earth's field to 1 part in 250,000, limited only by the signal-to-noise ratio, and there is no reason why this precision cannot be improved in the near future.

Magnet Design

During the last few years the need for large research magnets of various geometries and precisions has led to the development and manufacture of several different types of magnets. The properties of a magnet that are of interest to the experimenter are generally the volume and homogeneity of the field, the intensity of the field, and the stability, both short- and long-time. The size of a magnet is usually described most simply in terms of the diameter of the magnet pole piece. Research-type magnets are available in sizes that range from a 4-inch diameter to a 12-inch diameter pole piece. These can be procured in permanent magnets as well as in electromagnet types.

Modern magnets usually have the general geometry shown in Fig. 1, which shows a Varian 12-inch electromagnet. The magnet is constructed with a double yoke, not so much to improve the magnetic circuit but rather to improve the mechanical stability. The magnetic forces tending to pull the pole pieces together are tremendous, amounting to about 7 tons for a 12-inch magnet at a field of 14,000 gauss. The effect of this force on a single-yoke magnet is to destroy the parallelism of the pole pieces, and thereby the homogeneity, as the field is changed. In the double-yoke magnet, only the spacing between pole tips is altered; this does not affect the homogeneity of the field.

The efficiency of an electromagnet at low or moderate fields is not particularly important. However, at higher fields it is important to attain as high a field as possible for a given power input, since cooling rapidly becomes a problem at higher currents. The general geometry of the magnet shown in Fig. 1 was chosen to produce a magnet of high efficiency compatible with good accessibility and ease of manufacture. For the production of very high fields, the fully enclosed yoke of the A. D. Little magnet shown in Fig. 2 is advantageous. Marked deviation from the geometry shown in Fig. 1 will generally result in a reduced efficiency; for this reason, most new designs of different sized magnets are uniformly scaled copies of this geometry. Scaling is the most satisfactory way to design new magnets because of the difficulty of calculating leakage flux.

The maximum field intensity attainable by a particular magnet is determined by the volume of the field and the maximum amount of heat that can be removed from the coils by the cooling system. The curves of Fig. 3, which are typical for a 6-inch magnet, show the great decrease in maximum achievable field as the gap is increased. At a ¼-inch gap, the maximum field is 18,000 gauss, while for a 6-inch gap the field is only 2000 gauss. The production of very high fields—above 15 kilogauss, where saturation of the iron becomes important—can be achieved only by the use of tapered pole pieces and narrow gaps or by the use of large input powers. For example, a 12-inch magnet that is dissipating 4.0 kilowatts will produce a field of about 13,500 gauss for a 1¾-inch gap, while tapering the pole pieces to a 1-inch diameter and a ¼-inch gap will increase the field to 38,000 gauss.

The A. D. Little magnet shown in Fig. 2 can be operated at input powers of 100 kilowatts and will produce a field of 22 kilogauss in an 11-inch diameter by 1¾-inch gap. The fact that the field is proportional to the square root of input power is a great limitation in the design and use of large electromagnets.

The coils of electromagnets can usually

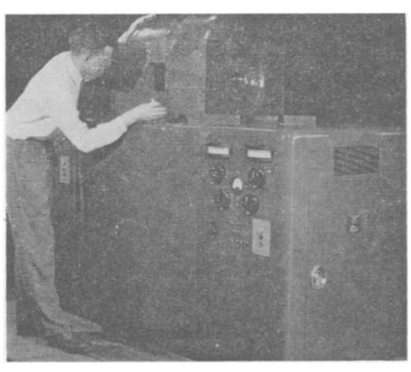

Fig. 2. A. D. Little electromagnet, rotating, adjustable-height model, a type especially designed to produce very high fields. [Courtesy Arthur D. Little, Inc.]

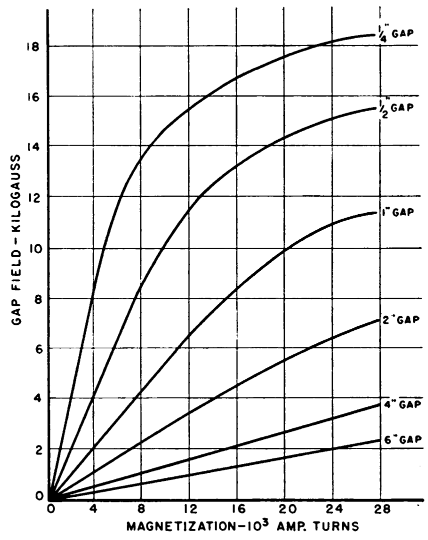

Fig. 3. Magnetic field as a function of magnetization current and gap width for the Varian 6-inch electromagnet. These curves can be used in general for any size electromagnet of the same design provided that the gap width is scaled accordingly.

be classified as having either high- or low-current windings, depending on whether the magnet current is more or less than about 2 amperes. For extremely high input powers, it is advantageous to use a high-current winding in order to achieve good heat transfer by assuring intimate copper-to-coolant contact. On the other hand, in magnet systems that require high stability, it is desirable to use a low-current winding in order to facilitate magnetic field regulation by passing the total magnet current through controlled vacuum tubes.

Permanent magnets are ideal for many applications because of their inherent stability and freedom from power supplies. Their usefulness is generally limited to producing moderate fields because the length of the Alnico-V pole pieces becomes inconveniently long at high levels. Figure 4 shows an experimental permanent magnet that has Alnico-V pole pieces each 19 inches long. This magnet produces a field of about 8000 gauss in a 1-inch by 10-inch diameter gap. This field can be increased to 14,000 gauss by tapering the pole pieces to 5-inch diameter and narrowing the gap to ¼ inch. Permanent magnets have been built that have square pole pieces measuring 12 by 12 inches; these produce a field of 7000 gauss in a 1½-inch gap.

Permanent magnets need to be energized with about 6000 ampere turns per inch of Alnico-V. The peak power is high but the average power is low because magnetization is accomplished in less than ½ second. The field is usually reduced from the maximum value in order to stabilize the system against mechanical and thermal shocks.

Magnetic Field Homogeneity

Ever since the discovery of nuclear magnetic resonance, investigators have discovered new and ramifying phenomena with each significant improvement in field homogeneity and stability. With a definition of about 1 part in a million, Arnold, Dharmatti, and Packard (*13*) discovered fine structure in the proton resonance owing to differences in electronic magnetic shielding about otherwise identical nuclei. A further improvement by a factor of 10 revealed the existence of hyperfine structure owing to indirect interactions between nuclear spins in the same molecule (*14*). Some of the most precise work to date, by Anderson (*15*) and Arnold (*16*), has involved definition of about 1 part in 100 million and has revealed hyperfine structure details to second order, indicating not only very small energy differences owing to spin interactions, but also differences in the lifetimes of the individual energy states. This is evidently not the limit to which useful information may be gained by an increase in precision. The afore-mentioned experiments seem to indicate that there is much to be learned with a precision of 1 part in 10^9 or better.

The volume over which a field is homogeneous will depend on the ratio of the pole-piece diameter to the gap width. To obtain a first approximation, well-known mathematical methods can be used to calculate the region of homogeneity for flat pole faces or for ring shims that are used to increase the field at the edges of the gap (*17*). In order to achieve good homogeneity, great care must be taken to assure that the pole faces are flat and parallel and that they are made of metallurgically uniform material. Mechanical tolerances cannot be held closely enough to insure parallelism of the pole pieces, and therefore the pole pieces must be adjusted after manufacture. This adjustment is best done by tilting the pole pieces until a nuclear magnetic resonance plot of the field shows concentric circles of constant field centered about the geometric center of the magnet. Figure 5 shows a 12-inch magnet with field measuring probe inserted. The probe is 5/16 inches in diameter and contains a ⅛-inch diameter spherical water sample at the end. An example of the type of field plot made with this probe is shown in Fig. 6. This plot not only shows the concentric circles but also gives an idea of the homogeneity that can be achieved over a rather large volume.

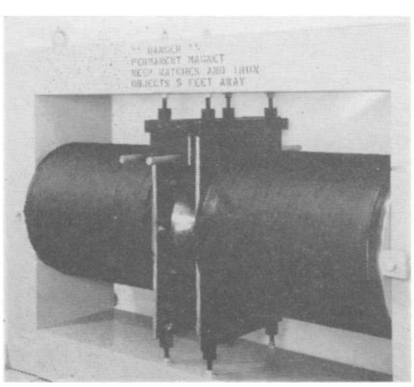

Fig. 4. Experimental permanent magnet intended to produce a 7000-gauss field in a gap 6 inches in diameter and 1.75 inches wide. The over-all dimensions of the magnet yoke are 39 by 56 by 13 inches.

For high homogeneity of 1 part in 10 million over a volume of 0.1 cubic centimeters, pole faces should be almost optically flat and free of machining marks. These conditions can be met only by lapping the pole faces and inspecting them optically for flatness. The field distribution will be dependent somewhat on the magnetic history of the magnet. For each magnet, one can develop a recipe for varying the magnetism in such a way that the end result is an optimum shaped field. The field pattern in the gap may show long-time drifts (of the order of hours) because of magnetic hysteresis of the iron. This effect will be most pronounced at the edges. Permanent magnets show much smaller regions of homogeneity than electromagnets for the same ratio of gap width to pole face diameter and must always be corrected by using ring shims.

Some magnets, although they appear perfectly symmetrical on both sides of the gap, actually show a relatively large field gradient tending to concentrate the field at one of the pole faces. The total difference in fields at the two pole faces may be as much as 0.5 gauss. This is presumably the result of differences in the permeability of the iron in the two pole pieces or of unequal windings in the two halves of the magnet.

Some experimenters have had success in the use of special shimming techniques to assist in obtaining homogeneity. Inhomogeneity can be reduced by the use of thin metallic shims placed either near the edge of the pole face as ring shims, or on the pole face in order to increase the field over small regions. The judicious use of emery paper will reduce the field over small regions and sometimes increase homogeneity. Flexibility in shimming can be achieved by the use of small coils pasted to the magnet. Different currents can be passed through the coils in order to produce quickly a number of shimming patterns. Needless to say, these techniques can be laborious and are best suited to trimming a good magnet.

In nuclear magnetic resonance experiments, the effect of a homogeneous field

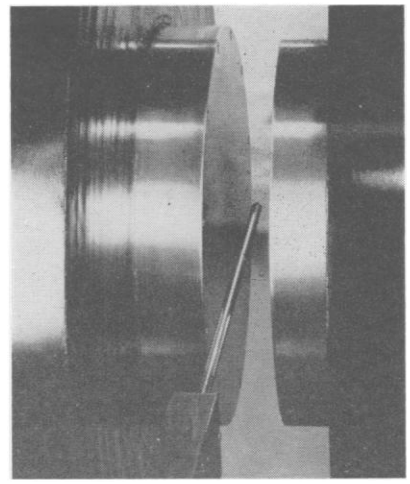

Fig. 5. Pole faces of Varian 12-inch electromagnet and nuclear magnetic resonance field-measuring probe. Note reflection of tip of the field-measuring probe in the left-hand pole face, an indication of the polish necessary for high homogeneity.

can be obtained by using a rotating sample holder (*18*). When the sample is rotated at a rate fast compared with the apparent T_2 in the inhomogeneous field, the nuclei experience all possible fields in the sample, but only the average, which is the same for all nuclei, is effective in the resonance.

Magnetic Field Stability

Whereas the problem of making the field homogeneous is mostly one concerning design of the magnet, the problem of making the field stable is primarily one of controlling external influences. For example, a permanent magnet has a negative temperature coefficient of 1 part in 5000 per degree Celsius, and an electromagnet is only as stable as the current that passes through its coils. Other influences such as external fields will affect both types of magnets.

The stability that is desired in the high-resolution nuclear magnetic resonance experiments of interest in physical chemistry is about 1 part in 10 million for a period of 30 seconds or more. At this stability, storage batteries by themselves are quite inconvenient, for the voltage "runs down" perceptibly and the resistance of the magnet increases. The most satisfactory answer seems to be a highly regulated constant-current power supply deriving its power from the alternating-current mains. Regulation is achieved by passing the magnet current through a small resistor; the resultant voltage across the resistor is compared with a reference voltage from a battery, and the difference is greatly amplified. The error voltage is then fed back by being impressed on the grids of vacuum tubes through which the magnet current is passed. The implementation of this deceptively simple feedback system is complicated by phase shifts that lead to oscillation and by vacuum tube and circuit noise and instabilities. The drift in ordinary direct-current amplifiers is avoided by the use of choppers, or synchronous converters. All voltages in the power supply must be highly regulated, as well as the heater currents in the vacuum tubes.

At the stability level of 1 part in 10 million, the effects of completely external influences become extremely critical. Alternating-current fields from nearby transformers, metal objects being moved in adjacent laboratories, and elevators in the building—all have been known to plague the researcher in one installation or another. Another form of external influence is distortion of the magnet as a result of thermal effects or external forces. At the level of 1 part in 10 million, the pressure of one finger on a magnet yoke 6 by 12 inches in cross section produces a relatively large change in field.

A somewhat different approach to the stability problem is to devise means to control the magnetic field itself rather than just the current in the windings. This can be accomplished rather easily by using nuclear magnetic resonance to generate an error signal that is injected into the input of the power supply regulator amplifier (*19*). Many installations of this type have been made to control long-time magnet field drifts to 1 part in a million or less, and most have been highly successful. For the ultimate short-time stability, the idea loses its simplicity because the signal-to-noise ratio of the nuclear magnetic resonance control signal may be low enough to introduce noise and hunting that may be comparable to the original instabilities. Briefly, the steps needed to insure correct operation require that the control probe be in about as homogeneous a field as the research probe and that both probes be at the same field value. This puts severe homogeneity requirements on the magnet. The most stable system is one in which the greatest pains have been taken to make an intrinsically stable magnet system, with nuclear magnetic resonance feedback added as a final touch to take care of slow drifts.

An example of the type of magnet stability that one can achieve practically is shown by the following experiment. The signal of the narrow 1-milligauss proton resonance of acetone was used to monitor changes in a 7000-gauss field. With no nuclear magnetic resonance feedback, the root-mean-square magnetic field fluctuation was about 0.5×10^{-3} gauss, although occasional peaks, apparently caused by transients on the alternating-current mains, were as high as 2×10^{-3} gauss. A metal chair moved 10 feet away from the magnet, or a bench voltmeter with its small permanent magnet, 5 feet away, would each cause a deviation of about 10^{-2} gauss. In addition, there was a slow diurnal temperature effect about 10 times larger. When nuclear magnetic resonance feedback was added, the field was maintained within a range of 2×10^{-3} gauss continuously for several weeks.

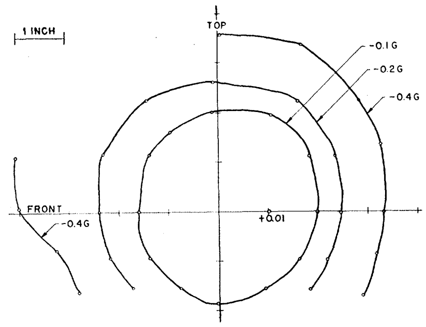

Fig. 6. Example of an experimental field plot of a Varian 12-inch electromagnet with a field of approximately 7000 gauss. The plot was taken halfway between the pole faces in a magnet that had ring shims to improve homogeneity. Contours are in fractions of a gauss with the center taken as the zero of reference. The isolated point labeled + 0.01 is the field maximum.

There is a still different approach to the stability problem that is now being tried out in several laboratories. It involves a feedback loop in which special windings are used as pickup coils to detect changes in total flux across the gap; the loop is closed with an integrator and amplifier that passes small currents through another winding to compensate for field changes. This method appears capable of suppressing field variations, both internal and external, by a factor of 10 to 100. It seems most useful for controlling rapid variations and transients, such as those induced by power line fluctuations, whereas nuclear magnetic resonance feedback is best suited for controlling long-time drifts.

The problems involved in stabilizing a permanent magnet are different in nature from those involved in stabilizing an electromagnet. The short-time stability is excellent if external field influences can be eliminated. The long-time drift may be troublesome because of the high temperature coefficient of 1 part in 5000 per degree Celsius. However, this may be minimized by temperature lagging or thermostating. Despite the relatively large thermal coefficient, thermostating is needed only to an accuracy of about 1°C, owing to the enormous heat capacity of the magnet. Occasionally, as Wertz points out (*20*), a nearby open window is used as a control, presumably with a nearby graduate student as thermostat. The drift and the external field interferences can be reduced either by nuclear magnetic resonance or by the use of a feedback loop consisting of pickup coil, integrator, and amplifier.

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Nourished by knowledge patiently won, bounded and conditioned by co-ordinate reason, the imagination becomes the prime mover of scientific discovery.—John Tyndall.