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ORGANISATION EUROPÉENNE POUR LA RECHERCHE NUCLÉAIRE CERN EUROPEAN ORGANIZATION FOR NUCLEAR RESEARCH

THE NUCLEAR MAGNETIC RESONANCE MAGNETOMETER TYPE 9298

K. Borer and G. Frémont

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K. Borer*) and G. Frémont

GENEVA

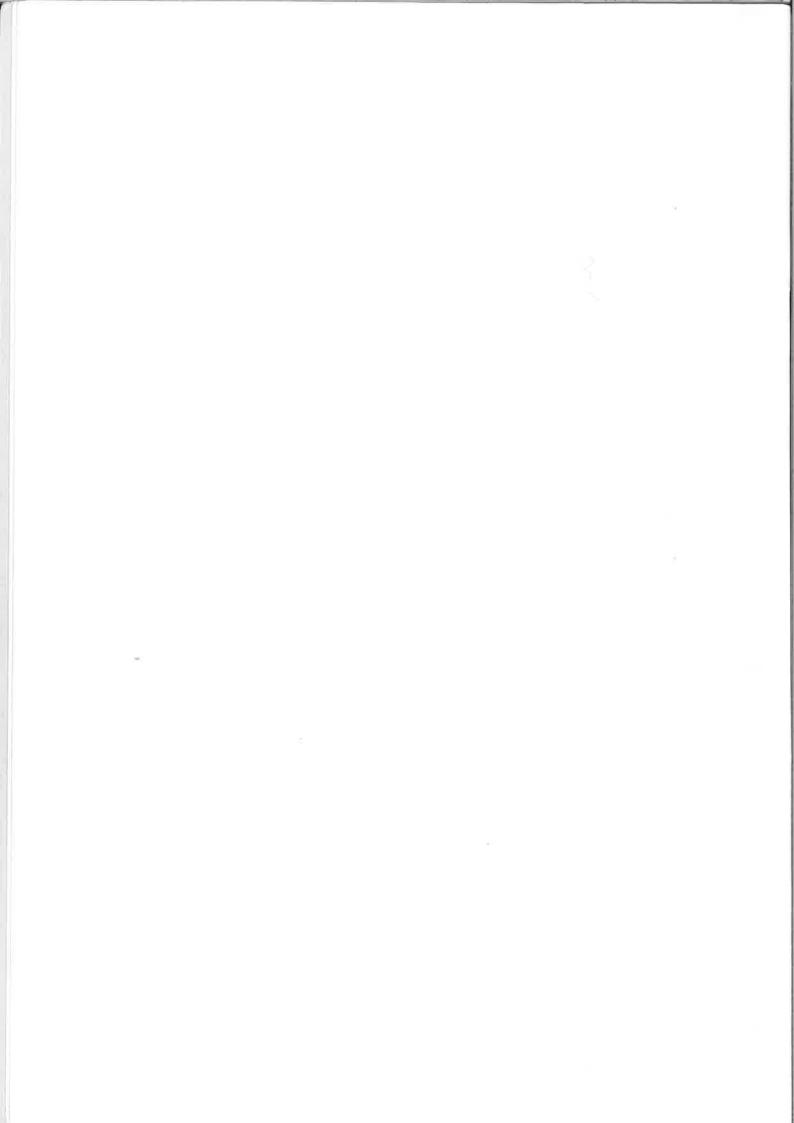
1977

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ABSTRACT

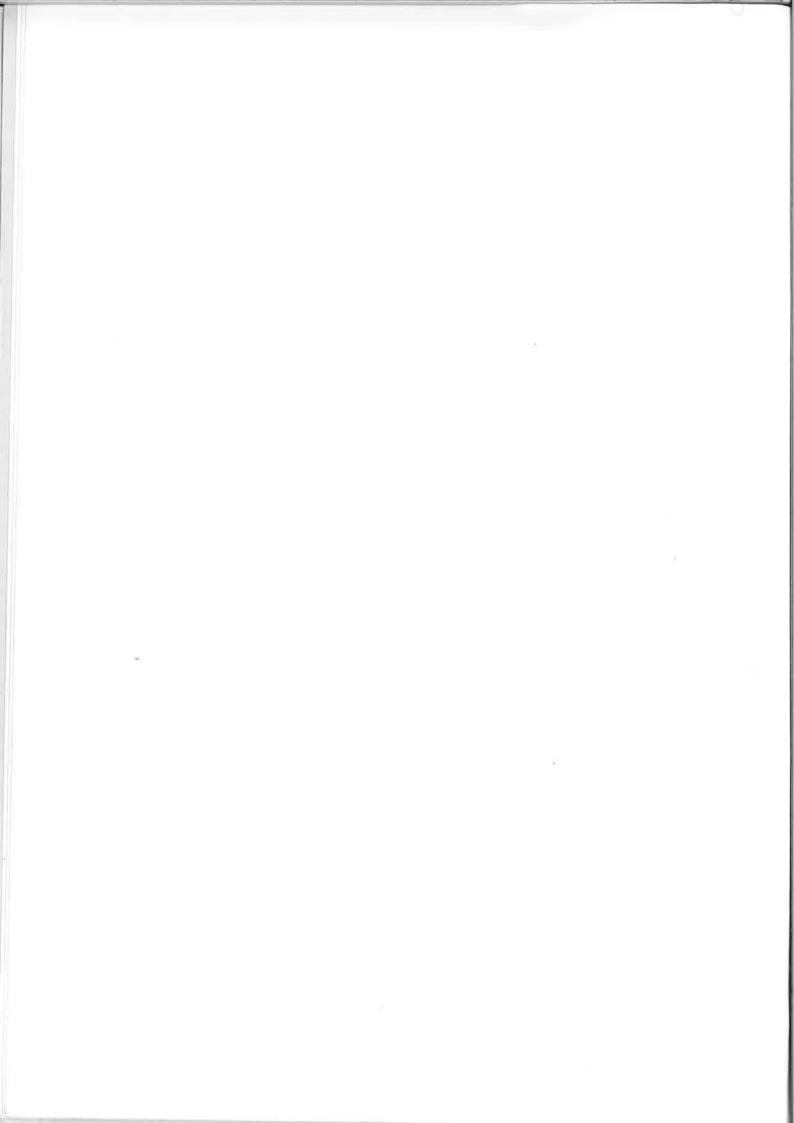
The nuclear magnetic resonance (NMR) magnetometer described here measures homogeneous magnetic fields at a ppm level precision. It offers automatic frequency tracking and digital read-out of the measured magnetic field strength, and is easier to use and much cheaper than existing commercial instruments of similar versatility. It consists of a dual-width NIM plug-in, a probe amplifier box, and various probes.

Fields between 1 kG and 21 kG can be measured with four proton probes, and two deuteron probes are used from 20 kG to 68 kG. The corresponding six field ranges overlap comfortably. The search and frequency tracking range is a few percent of full scale; the coarse adjustment of the frequency has to be done manually.



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INTRODUCTION

The magnetic field in the muon storage ring of the latest (g-2) experiment at CERN 1) was stabilized and measured at a precision of ≤ 1 ppm with a large nuclear magnetic resonance (NMR) system 2) specially designed for this experiment. The 10 NMR magnetometers used during four years were shown to work very reliably and to be easier to use than similar commercial instruments. Moreover, the cost of the components and for wiring these dual-width NIM plug-ins is only a small fraction of the price of a typical commercial NMR magnetometer.

There are various applications for low-cost and easy-to-use NMR magnetometers; for example, calibration of Hall plates, monitoring or stabilization of the field in the spectrometer and bending magnets, etc. Unfortunately, because of the experiment for which they were designed, the (g-2) magnetometers have a too narrow field range (14.6 to 15.0 kG). Therefore a modified version for more general use, measuring fields between 1 kG and 68 kG, has been developed. For this, the frequency range of the NMR probes and the oscillator has been increased. Moreover, the electronics have been upgraded by making use of more modern integrated circuits, but the well-proved principle of operation of the (g-2) magnetometers has been fully preserved.

The new NMR magnetometer (Fig. 1) consists of a dual-width NIM plug-in, which is the main unit, four H_2O probes (for B=1-21~kG) and two D_2O probes (for B=20-68~kG), and one

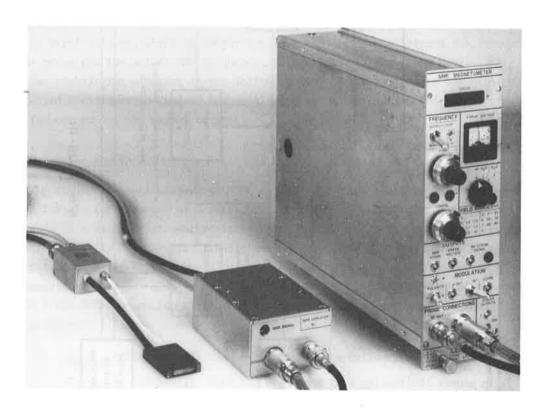


Fig. 1 NMR magnetometer with probe amplifier box and NMR probe

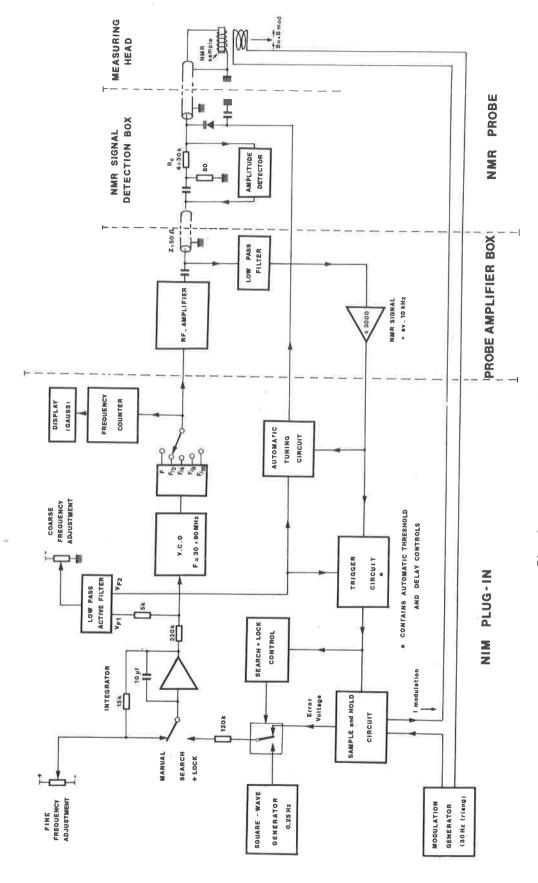


Fig. 2 Block diagram of the NMR magnetometer

probe amplifier box (the same for all probes). Each probe consists of a measuring head and a small box which contains the signal detection circuit. The maximum length of the two cables between the detection circuit and the measuring head is about 17 cm, whereas the cables between the probe and the probe amplifier box may be a few metres long. From there to the main unit any cable length is allowed, provided the RF signal is not damped more than 6 dB. A block diagram of the magnetometer, including probe and probe amplifier box is given in Fig. 2. The RF oscillator in the main unit has a frequency range of 30 to 90 MHz, which corresponds to the highest proton resonance field range of 7 to 21 kG. The other field ranges are obtained by dividing the frequency by 2, 4, or 8; f:4 and f:2 being used for the two D_2O probes. This results in a very comfortable overlap of the six field ranges (except below 20 kG, where the signal of the D_2O probe becomes too weak). An internal frequency counter measures the frequency which is sent to the probe, the result being displayed in gauss with a resolution of 0.01 G.

Coarse adjustment of the frequency is done manually with a 10-turn potentiometer. A second 10-turn potentiometer allows fine adjustment over 1 to 10% of full scale, depending on the coarse frequency setting and the type of probe $(H_2O \text{ or } D_2O)$ used. In the "search and lock" mode, the unit sweeps the frequency up and down through the fine adjustment range until a NMR signal appears. Then it "locks" automatically to this signal, i.e. feedback control adjusts the frequency such that it equals the NMR frequency of the connected probe. The resulting frequency tracking with any changes of the magnetic field at the probe is restricted to the fine frequency adjustment range.

Various other automatic controls simplify the use of the magnetometer; these are automatic probe tuning and automatic trigger threshold and timing of the NMR signal processing circuits.

An error voltage, which is proportional to the difference between the frequency applied to the probe and the actual NMR frequency, is available at the front panel. This can be used for stabilizing a magnet in the following way: the NMR probe is connected to a crystal-controlled oscillator instead of the oscillator in the main unit, and the error voltage is used for correcting the magnet current.

2. REMARKS ON THE NMR TECHNIQUE USED

The theory and applications of NMR are widely covered in the literature 3). Let us just recall that in the presence of a static magnetic field B_0 , a nucleus with magnetic moment μ can take (2T + 1) distinct energy states, I being the spin quantum number. The separation of these states is $\Delta E = \mu B_0/I$. Transitions between levels can be induced by applying an alternating magnetic field perpendicular to the static field if its frequency equals the resonance frequency $f = \Delta E/h = \Gamma B_0$, with $\Gamma = \mu/hI$. For magnetic fields of the order of 10,000 G the NMR frequencies lie in the RF region. For protons and deuterons Γ is known very precisely 4):

 $\Gamma_{\rm p,H_2O}$ = 4257.608(12) Hz/G for protons in a cylindrical sample of H₂O; $\Gamma_{\rm d,D_2O}$ = 653.569(2) Hz/G for deuterons in a cylindrical sample of D₂O .

For detecting the proton magnetic resonance *), a small water-filled coil is placed in the static field B_0 , with its axis perpendicular to B_0 . The magnetic moments of the protons in the water molecules point preferentially in the direction of B_0 , i.e. the lower energy magnetic states are more populated than the higher one. Therefore, if transitions are induced with an alternating field, those from lower to higher energy states are more frequent than the contrary. The protons absorb more energy from the alternating field than they supply to it, and the difference between the populations of the two energy states is reduced.

The thermal equilibrium populations are re-established, due to spin-lattice interactions, at a rate described by the so-called spin-lattice relaxation time T_1 . This is the reason why the protons continuously absorb energy from the alternating field if the coil is driven at the proton resonance frequency, thereby reducing slightly the quality factor Q of the coil.

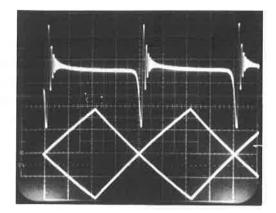
A practical way of detecting this effect is to tune a parallel LC resonant circuit to the proton resonance frequency, using the water-filled coil as the inductor, and to apply to this tank circuit a stable sine wave of that frequency via a resistor. The resistor value chosen should be high compared with the resonance impedance of the tank circuit in order to avoid damping. If the proton resonance frequency is now modulated by superimposing a modulating magnetic field parallel to the static field B_0 , the reduction of the Q factor due to the proton resonance can be detected as a small amplitude variation of the RF voltage across the tank circuit.

This signal can be enhanced by adding a paramagnetic salt to the water. This reduces the relaxation time T_1 and therefore increases the steady-state energy absorption of the protons at resonance. Our NMR probes contain 0.1 molar NiSO $_4$ instead of pure water.

The modulating field B_{mod} is produced by a small, flat coil in the NMR probes. Its frequency is 30 Hz and its amplitude 100 to 1000 ppm of B_0 . The magnetometer electronics detects and amplifies the nuclear resonance signals of the LC circuit and measures the current in the modulating coil at the instant when the resonance occurs. A voltage-controlled oscillator produces the RF voltage. Its frequency is controlled by a high-gain feedback loop, such that the resonance occurs at the instant when B_{mod} crosses zero. Therefore this frequency equals the proton resonance frequency of B_0 and automatically follows any changes in B_0 . The LC circuit is automatically tuned to the applied frequency by means of a varicap diode.

In concluding these general remarks, it should be mentioned that the field modulation in the NMR probes sweeps far too quickly through the resonance to obtain adiabatic conditions. Therefore, the observed signals have neither the form nor the width of the real proton or deuteron resonance curve (i.e. the form found for very slow crossing of the resonance). The width is several times the natural line width, and transient effects, like for example "wiggles", appear (Fig. 3). However, this fast modulation is convenient for practical reasons, and an accuracy better than 1 ppm is nevertheless achievable, using a symmetry criterion. This subject is discussed in more detail in Section 3.

^{*)} The following explanations are also true for deuterons, except that D_2O doped with $GdCl_3$ is used instead of H_2O + $NiSO_4$.

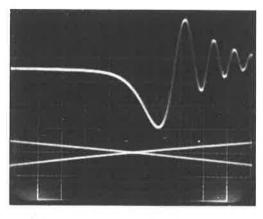


1 V/div.

Time scale: ∿ 4 msec/div.

2 G/div.

"alignment" of the NMR-pulses in the frequency tracking mode,

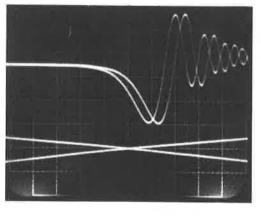


1 V/div.

Time scale: ○ 0.4 msec/div.

2 G/div.

magnified display of the central part,



1 V/div.

Time scale: ∿ 0.4 msec/div.

2 G/div.

"misalignment" resulting from a 1 ppm frequency error.

Fig. 3 NMR-signal and modulation field chopped y(t) display with \vec{B} = 15 kG. (The scope is triggered with the NMR-pulses, alternatively at upward and downward going modulation field.)

3. PRINCIPLE OF OPERATION OF THE NMR MAGNETOMETER

An over-all view is given in this section; some circuit details will be discussed later in Sections 4 and 5. It is convenient to explain the block diagram (Fig. 2) starting with the probe. The measuring head contains a flat coil for modulating the field in a small glass tube, which contains either H_2O or D_2O and around which an RF coil is wound. The applied field modulation is a symmetric 30 Hz triangular wave form with an amplitude of a few hundred ppm.

The RF coil, a tuning diode, and the coaxial cable from the NMR signal detection box to the measuring head form a parallel LC resonant circuit. This resonant circuit is weakly coupled, by means of a resistor, to the output of an RF amplifier with stabilized output amplitude, and is automatically tuned to the applied frequency. If this frequency is chosen close enough to the nuclear resonance frequency corresponding to the main field B_0 , an absorption signal (i.e. amplitude variation) appears in the LC resonant circuit every time the resonance is crossed due to the field modulation. This signal is amplified in the amplifier box and transmitted to the main unit.

A sample-and-hold circuit produces an "error voltage" which is proportional to the modulating field at the instant when the nuclear resonance occurs. With this error voltage, the frequency of the RF oscillator in the main unit is regulated in such a way that the nuclear resonance occurs exactly at the zero crossing of the modulation. This frequency is therefore equal to the nuclear resonance frequency of the field B_0 seen by the protons or deuterons in the sample without modulation. It follows automatically all changes of B_0 , within the range covered by the fine frequency adjustment.

The kind of field modulation used, which as already mentioned influences the signal shape and increases its line width, has been chosen for the following reasons: i) the modulation amplitude of a few hundred ppm makes locking of the RF to the field easy; ii) the modulation frequency of 30 Hz was found to be a reasonable compromise between RF tracking speed and signal line width; iii) a triangular wave crosses through zero more slowly than a sine wave of the same frequency and amplitude, which reduces the spreading of the line width. Moreover, it can be easily generated very symmetrically, thus improving the accuracy of the magnetometer.

The resulting line width is 10 to 100 ppm, depending on the measured field and the modulation amplitude setting. An accuracy better than 1 ppm can still be reached, provided that the LC resonance circuit in the probe is well tuned to the applied frequency and that the field modulation is symmetric in respect to zero, i.e. $B_{mod}(t+T) = -B_{mod}(t)$, 2T being the period of the modulation. With both conditions fulfilled, the NMR signals become identical in form and size and equally spaced in time if the resonance occurs at the zero crossing of the modulation (Fig. 3). If the LC resonance circuit in the probe is slightly mistuned a dispersion signal is mixed with the absorption signal, and the NMR signals at upward zero crossing of the modulation look different from those at downward zero crossing. This effect is eliminated by the automatic tuning of the probe.

It is therefore not necessary to know *a priori* at which point of the 10 to 100 ppm wide signal the applied frequency is equal to the proton resonance frequency. The criterion is simply that the time difference between any point of the NMR signal and the close-by zero



crossing point of the modulation is equal for the upward-going as well as for the downward-going modulating field. Then the applied frequency is equal to the proton resonance frequency of the field B_0 with B_{mod} = 0 and this is the criterion upon which the frequency control loop works.

The automatic tuning of the probe and the good symmetry of the field modulation are the basis for the high accuracy of the magnetometer. To achieve a short response time of the frequency control loop, the sample-and-hold circuit mentioned above is used to produce an 'error voltage', which indicates after each NMR pulse how far away the resonance was from zero modulation, the sensitivity being 8 V/G for the lowest and 0.3 V/G for the highest field range. This error voltage is integrated and then sent to the frequency control input of the RF oscillator. By choosing the gain and the integration time constant appropriately, the error can be corrected entirely within the time between two consecutive NMR signals. Therefore, for this optimum loop gain setting, the time lag of the frequency tracking is equal to the spacing of the NMR signals, which is roughly 17 msec. The loop gain at d.c. is of the order of 10⁶.

The size and width of the NMR signals depend strongly on the field strength and homogeneity. During field mapping, for example, the amplitude may vary by a factor of ten and the width by a factor of four. Therefore the trigger level and the timing of the sample-and-hold circuit are adjusted automatically in the main unit in order to maximize the range of operation of the magnetometer. The trigger level is set automatically to about half the signal amplitude, the latter being measured with a special peak detector circuit, which is insensitive to possible occasional large, single, parasitic signals.

The trigger point may be early or late with respect to the proper proton resonance. In order to correct this, both the strobe pulse as well as the triangular wave voltage, proportional to the modulating field, are delayed appropriately before being fed to the sample-and-hold circuit which produces the error voltage. Wrong timing does not change the mean value of the error voltage, but produces a 30 Hz rectangular signal superimposed on it and synchronous to the modulating field (see Fig. 4). It is, therefore, the speed rather than the accuracy of the field measurement which would deteriorate, because a larger integration time constant would be needed. However, the delay of the strobe pulse is adjusted automatically by the magnetometer such that the above-mentioned 30 Hz component of the error voltage disappears.

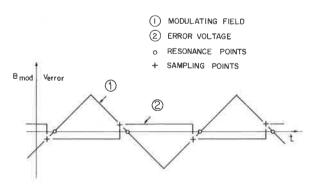


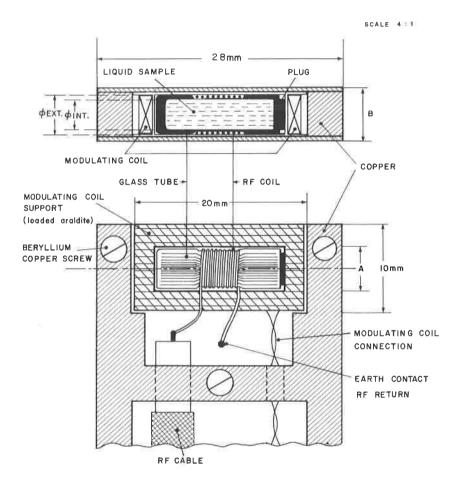
Fig. 4 A 30 Hz square-wave component in the error voltage indicates wrong timing of the sample-and-hold circuit. In the example shown, the sampling pulses are assumed to be early in respect to the nuclear resonance.

4. THE PROBES AND THE NMR AMPLIFIER BOX

4.1 General remarks on the probes

Six probes are necessary for a field range of 1 to 68 kG. Each probe consists of a measuring head and a NMR detection box, which are interconnected by a short 50 Ω coaxial cable, which is part of the LC resonant circuit, and a screened cable with two wires for the modulation. The probe and the NMR amplifier box are interconnected with a four-wire cable (for the modulation, a negative supply, and the tuning diode bias voltage) and a 50 Ω double-screened coaxial cable which transmits both the RF and the NMR signals (+ any detected 10 kHz signal which is used for the automatic tuning).

Figure 5 shows the mechanical drawings of the measuring head. It contains a small glass tube filled with the liquid NMR sample, H_2O + 0.1 m NiSO₄ for fields between 1 kG and



DD005 No	GLASS TUBE				COIL TURN NUMBER		
PROBE No.	φ EXT.	φ INT.	mm	mm	RF	MODULAT.	NMR SAMPLE
2	6	5	6,2	7	44	8	H_O+NISO4,0.1 m
3	5	3.2	5.1	6	32	19	т.
4	- 11	11	11		15	35	TE .
5	В	6 .	44.		5	92	ii.
6	11	10			32	35	D ₂ O + Gd Cl ₃ . Sat
7	п	ŭ.	n	ш	12	92	и

Fig. 5 Measuring head

21 kG, or D_2O saturated with gadolinium chloride for fields between 20 kG and 68 kG. One end of the glass tube is sealed by fusion of the glass and the other with a rubber plug. The RF coil is placed midway along the tube, wound in a 5.5 mm long and 0.2 mm deep groove which improves the "filling factor".

The number of turns of the RF coil is defined for each probe by its highest operation frequency and the lowest attainable value of the capacitance of the LC resonance circuit. This capacitance is essentially the sum of the capacitances of the coaxial cable and of the tuning diode. With the type of tuning diode used, a frequency range of a factor of three can be covered with a maximum cable capacitance of 17 pF, i.e. a maximum length of 17 cm. With this cable length, the probe which works at the highest frequency range needs an RF coil with five turns. For the lowest frequency range, 44 turns are needed.

The flat modulating coil is wound on a support made of loaded araldite with a rectangular hole for the sample tube. The number of turns of the modulating coil depends on the field range of the probe, and is chosen such that a field modulation of 100 ppm is produced by a current of a few tens of mA. This number of turns is also important for the loop gain of the frequency control loop (see Section 5.2). The modulating field in the sample is not homogeneous. This does not harm the accuracy, as the resonance occurs when the modulating field goes through zero, but it has the welcome effect of damping the "wiggles".

4.2 The automatic probe tuning

Although for space reasons the circuits for generating the varicap voltage are located in the main unit, their operation is discussed in this section, as the automatic tuning is a very essential feature of the probes. The simplified circuit diagram is given in Fig. 6.

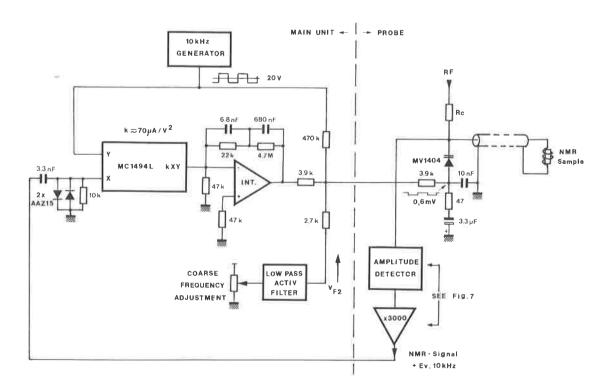


Fig. 6 Simplified circuit diagram of automatic tuning circuit

The bias voltage of the tuning diode in the probe is composed of the voltage V_{F2} given by the coarse frequency adjustment and the output of an integrator (INT in Fig. 6). A square wave signal of 0.6 mV amplitude is superimposed on it, and modulates very weakly the capacitance of the tuning diode ($\Delta C/C \sim 10^{-4}$). This results in a 10 kHz amplitude modulation if the resonant circuit is slightly mistuned, being in phase or 180° out of phase with respect to the injected square wave, depending on whether the capacitance is too small or too large.

The amplitude modulation is detected and amplified by a factor of 3000 together with the NMR signal. The superposition of both is fed to the x input of an analogue multiplier MC1494L, where the NMR part is reduced by RC differentiation and diode clipping, the latter being necessary in order to avoid spurious signals when there are large and slowly decaying wiggles. The y input of the multiplier is connected to the 10 kHz square wave generator, which produces a bi-polar output signal of ± 10 V. The same signal is used with ~ 90 dB attenuation for modulating the varicap. This attenuation is split into two steps: ~ 50 dB in the main unit and ~ 40 dB in the probe, where a low-pass filter is placed for reducing the noise pick-up in the long interconnecting cables.

A positive or negative current is produced at the multiplier output whenever the resonance circuit is mistuned. This current is fed to the integrator INT, which changes the tuning diode bias voltage until the multiplier output current falls to zero, i.e. the 10 kHz signal at the x input disappears. The time constant has been chosen such that the automatic tuning easily follows the fastest frequency variations in the search mode. The effect of the low-pass filter in the probe on the frequency characteristic of the loop gain is compensated by an appropriate feedback network of the integrator.

4.3 The NMR signal and RF amplifiers

A simplified circuit diagram of the NMR probe and NMR amplifier box is shown in Fig. 7. The complete diagrams of these circuits are given in Figs. 8 and 9, respectively. The NMR

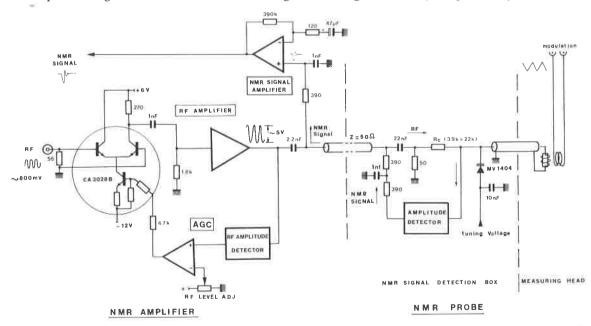


Fig. 7 Simplified circuit diagram of NMR amplifier and NMR probe





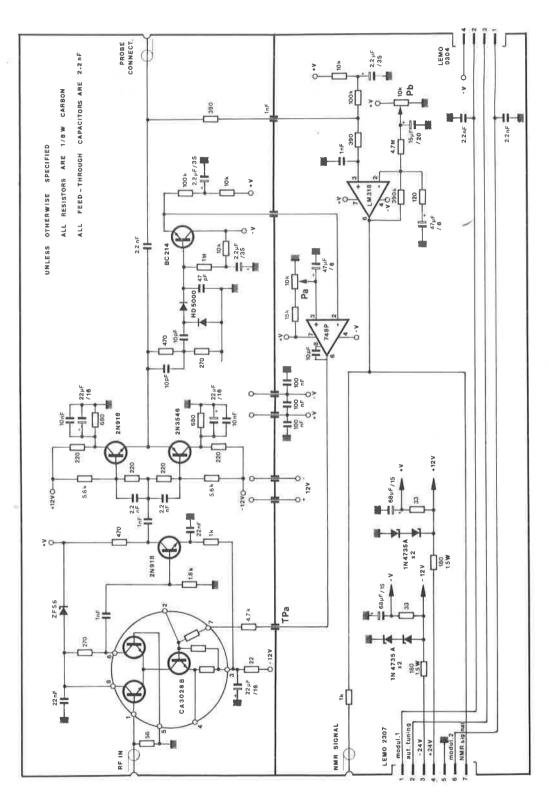


Fig. 8 Circuit diagram of probe amplifier box

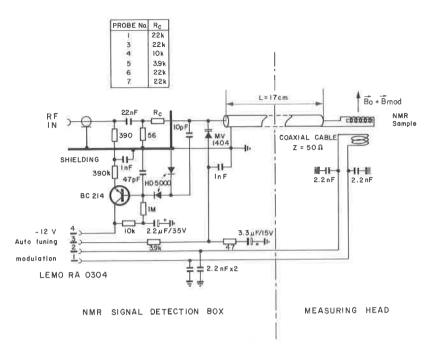


Fig. 9 Circuit diagram of NMR probe

absorption signal is an amplitude variation of the RF voltage of the LC circuit and is very small, typically of the order of 0.1%. It is detected in a conventional way with two Schottky diodes and transmitted by means of an emitter follower through the coaxial cable and a low-pass filter to the NMR amplifier box. The a.c. part of the detected voltage is amplified by a factor of 3000, whilst unity gain is provided for the d.c. component. The upper frequency limit is ~ 20 kHz, i.e. higher than the 10 kHz frequency used for the automatic tuning.

After this amplifier the amplitude of the NMR signal may vary from about 100 mV, which is near the lower limit for locking the RF to the field, to several volts. A slightly smoothed output (RC integration with 10 k Ω and 10 nF) is available at the front panel of the main unit for scope inspection of the NMR signal. Its d.c. component indicates the RF voltage amplitude at the LC resonance circuit in the probe, which should be about 0.1 to 1 V depending on the frequency. For example, this checks very quickly whether the connected probe corresponds to the selected frequency range and whether the automatic tuning works properly.

Because of the weakness of the NMR signal the RF voltage must be extremely clean with respect to any spurious amplitude or frequency modulation and noise, otherwise the signal-to-noise ratio becomes bad. The wave form of the RF signal, however, is not important since the LC resonant circuit and the NMR sample in the probe are insensitive to any harmonics. The precautions taken in the oscillator design will be discussed in Section 5.2. In the NMR amplifier box the RF signal is amplified to the level needed for the probe, which is about 5 V peak-to-peak. The RF amplifier consists of a fast differential amplifier (CA3028A) with voltage-controlled gain, a common emitter stage, and a push-pull output stage which is able to drive a 50 Ω load at the required level.

With a typical input signal of 0.5 V peak-to-peak amplitude, the differential amplifier works in a switching mode rather than linearly. Its sensitivity to amplitude variations of the input signal is therefore reduced. Moreover, the signal output at the RF amplifier is measured with a diode detector circuit and compared with a clean, adjustable reference voltage. Any difference is amplified and fed back to the gain control. This feedback control of the amplitude in addition to the switching of the input transistors, smooths any amplitude modulation of the input signal by a factor of 50 to 100. This helps, in particular, to reduce the very disturbing interference effects ("beating") when more than one probe operating at slightly different frequencies is used.



The output signal of the RF amplifier looks more like a badly shaped square wave than like a sine wave. As already mentioned, this is of no disadvantage, since the probe is hardly sensitive to the wave form. The output level is roughly the same for all frequencies. For obtaining the best signal-to-noise ratio, however, the optimum RF voltage of the LC resonance circuit in the different probes is set by an appropriate choice of the coupling resistor R_{C} to the RF input.

5. CIRCUIT DETAILS

5.1 Automatic trigger threshold and delay circuits

The reason for automatic controls of the trigger threshold and the timing of the sample-and-hold circuit has been explained in Section 3. Figure 10 shows the diagram of the corresponding circuits.

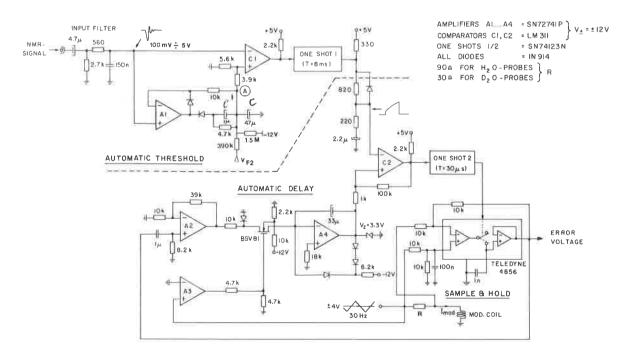


Fig. 10 Circuits for generating the error voltage, including the automatic threshold and timing controls

The NMR signals are fed to the input of the comparator Cl via a filter network which eliminates the d.c. component and reduces the noise. The trigger level is set automatically to about half the signal amplitude: the threshold of comparator Cl is 0.6 times the voltage at point A, which is produced by the circuit around Al in a charge pumping mode, and which is slightly less than the NMR signal amplitude. This kind of amplitude detection has the advantage of not being very sensitive to occasional large, single, parasitic signals, since the voltage at point A can change at most by 0.2 V per input pulse of any size.

The lowest trigger level, which is set with no NMR signal, is kept safely above the noise level. Since the noise increases with the RF, the minimum threshold is derived from the coarse frequency adjustment ($V_{\rm F2}$) and varies between 40 mV and 100 mV.

The modulating current I mod is sampled during the 30 µsec pulses produced by the one-shot 2 (shown in Fig. 10). The pulse width is not critical, but has to be long enough to allow the sample-and-hold amplifier to settle. The delay relative to the instant when the NMR signal crosses the threshold at C1 is determined by the voltage at the output of the integrator A4. This voltage is regulated so that no 30 Hz component appears at the error voltage output. If there is a signal in phase or 180° dephased relative to the modulation, it is amplified by A2 and integrated by A4 during only the positive half waves of the modulation, the MOS-FET switch being controlled by A3. This results in a decrease or increase, respectively, of the output voltage of A4 and therefore in a decrease or increase, respectively, of the delay, until the 30 Hz component disappears.

The three diodes and the Zener diode limit the output voltage of A4 to values safely above the base line and below the top of the pulse at the inverting input of C2. The range of the automatic delay is about 0 to 5 msec, which is, considering the fixed delay of 0.5 msec at the sample-and-hold input, equivalent to -0.5 msec to +4.5 msec. This is quite sufficient for all practical operating conditions of the magnetometer.

5.2 Frequency control and loop gain

As any frequency drifts of the voltage-controlled oscillator (VCO) are corrected by the frequency control loop, the problem of long-term stability of the VCO is not very critical. Any frequency modulation or noise above about 1 Hz is, however, very harmful; therefore, the following precautions are taken (see bottom of circuit diagram Fig. 14):

- very careful filtering of the varicap bias voltage and of the supply voltage of the oscillator;
- the oscillator is enclosed in a copper box for RF screening and for avoiding thermal convection effects.

The various frequency ranges are obtained by division in steps of two, using MECL 10,000 flip-flops. The selection of these ranges is done with MECL 10,000 gates. A long-tailed transistor pair produces two NIM outputs for external CAMAC or other counters, whereas an MECL 10,000 gate is used as output stage for the probe RF signal. Using a well-filtered supply voltage for this gate results in the necessary cleanness of the amplitude of the RF signal. Its square-wave-like shape does not cause any disturbance (see also Section 4.3).



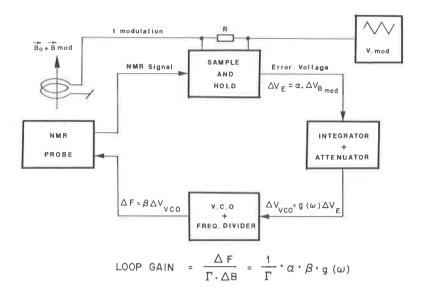


Fig. 11 Block diagram of frequency loop

The frequency control-loop diagram is given in Fig. 11. The sample-and-hold circuit produces an error voltage ΔV_E , proportional to the modulating field at the instant when the nuclear resonance occurs: $\Delta V_E = \alpha \ \Delta B_{mod}$. The frequency control voltage of the oscillator is derived from ΔV_E by integration and attenuation: $\Delta V_{CO} = g(\omega) \ \Delta V_E$, which results in a frequency change of $\Delta F = \alpha \beta g(\omega) \ \Delta B$. By choosing an appropriate integration time constant and attenuation, ΔF reaches $\Delta B_{mod}/\Gamma$ just when the next NMR signal appears, i.e. the frequency error which produced $\Delta V_E \neq 0$ is entirely corrected by this time. This is the optimum loop-gain setting for fast frequency tracking (see Section 3). Owing to the nonlinearity of the oscillator frequency control curve, the optimum loop-gain setting at a medium frequency is not valid for the full frequency range. The loop gain decreases in the worst case by a factor of five close to the upper and lower limits of the oscillator frequency range.

For all frequency ranges except one, the oscillator frequency is divided by a factor of $2^{\rm n}$, which results in a reduction of B by the same factor. This is compensated by the factor α , i.e. the product α times β is made constant for all field ranges between 1 kG and 21 kG (the D_2O probes need some extra explanation, see below) by using an appropriate number of turns of the modulating coil of the different probes. The number of turns is chosen such that at a given current in the modulating coil the ratio of B_{mod} to B_0 is the same for all H_2O probes (at the same VCO setting). Hence the number of turns decreases roughly linearly with the decreasing field range of the probe, whilst $\alpha = \Delta V_E/\Delta B_{mod}$ increases inversely, R being constant. Therefore, switching the frequency range and changing corresponding H_2O probes does not change the loop gain.

For the D_2O probes the resistor R for limiting and measuring the modulating current is switched to a three times lower value (30 Ω instead of 90 Ω), in order to keep the necessary number of turns of the modulating coil below impracticable limits. The ratio of B_{mod} to B_0 at a given voltage drop over the resistor R is the same for the D_2O probes as for the H_2O probes, and the resulting factor α compensates for the frequency division and the lower





gyromagnetic ratio of the deuterons. To understand this point, the following argument may be helpful: the sensitivity of the error voltage to a frequency error in relative terms (e.g. ppm) is the same for all H_2O probes and D_2O probes at a given VCO setting, and the relative change of the probe frequency $\Delta f/f$ produced by $\Delta V_{\rm VCO}$ does not depend on the frequency-dividing factor.

The signal-to-noise ratio is much smaller for the $\rm D_2O$ probes than for the $\rm H_2O$ probes. Therefore, for the $\rm D_2O$ probes an additional attenuation factor of three is switched in the frequency control loop, in order to facilitate locking of the magnetometer to the field. This reduces by the same factor of three the rate of the frequency variation in the search mode, the loop gain, and the frequency tracking range. The accuracy of the magnetometer is not influenced by the lower loop gain, which is still > 10^5 at d.c.

6. FREQUENCY COUNTER

A seven-digit frequency counter with a special time-base measures the NMR frequency in gauss with a resolution of 0.01 G (see Fig. 15). About 3 measurements per sec are displayed in the case of the $\rm H_2O$ probes, and ~ 1.6 per sec in the case of the $\rm D_2O$ probes, the gate length being defined by the gyromagnetic ratios and the chosen predividing factors of 16 for the $\rm H_2O$ probes and 4 for the $\rm D_2O$ probes.

The frequency counter is built up with TTL (Schottky Low-Power) circuits. The data transfer signal for its display register and the reset signal are generated by the time-base circuit, which is discussed in some detail below. A multiplexed miniature LED display is used, and a BCD output of the display register is available.

Figure 12 shows a block diagram of the frequency counter time-base. A 100 kHz crystal oscillator is used as the clock frequency for generating the required gate lengths. This

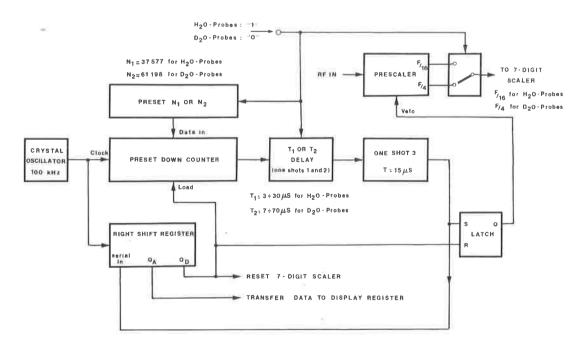


Fig. 12 Block diagram of the time base of the frequency counter

low clock frequency value has been chosen in order to avoid interference with the RF signal of the probe (risk of "beating"). The required gate lengths are 375798 µsec for the H_2O probes and 612024 µsec for the D_2O probes. The clock period of 10 µsec is too long for generating these times sufficiently accurately by simple countdown, therefore two one-shots for fine adjustment of the gate lengths (± 40 ppm) have been added. One-shot 1 (3 to 30 µsec) is used for calibrating the gauss reading of the H_2O probes, and one-shot 2 (7 to 70 µsec) for the D_2O probes. The stability of these one-shots (typically $\leq 1\%$) is not critical, since they add only a few tens of ppm to the total gate width. This interpolation technique has also the advantage that the frequency tolerance of the 100 kHz crystal is relaxed.

Figure 13 shows a pulse sequence diagram of some lines in the time-base of the frequency counter.

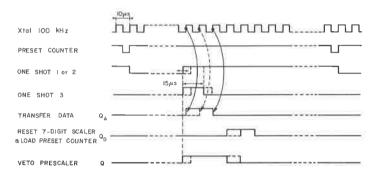


Fig. 13 Pulse sequence diagram of the time base of the frequency counter

7. SPECIFICATIONS

Probe No.	Field range (kG)	Probe type	Frequency range (MHz)
(1) a)	(0.45-1.3)	H ₂ O	(1.9-5.6)
2 b)	1.0-2.6	H ₂ O	(3.8)-11.3
3	1.7-5.2	H ₂ O	7.5-22.5
4	3.5-10.5	H ₂ O	15-45
5	7-21	H ₂ O	30-90
6 b)	20-34	D_2O	(7.5)-22.5
7 b)	30-68	D_2O	(15)-45
(8) c)	(46-138)	D_2O	(30-90)

- a) This probe is not yet constructed, and the signal-to-noise ratio at the lower end of the indicated field range might be too small for automatic frequency tracking.
- b) For these probes the signal-to-noise ratio is very small at the lower end of their frequency range, and automatic frequency tracking is only possible within the indicated field range.
- c) No adequate field was available for testing this probe, which is identical to probe No. 5 except that $\rm D_2O$ is used instead of $\rm H_2O$ and the number of turns of the modulating coil is doubled.

Absolute accuracy: better than $\pm 10^{-5}$; can be improved by absolute calibration of the probes.

Relative accuracy and stability: $\sim \pm 5 \times 10^{-7}$.

<u>Note:</u> The relative accuracy means the equality of the readings of different magnetometer units connected to the same probe in the same field. This accuracy and especially the stability depend on the signal-to-noise ratio and therefore on the field intensity.

The specified value holds for a signal-to-noise ratio safely above the limit for automatic frequency tracking.

Signal-to-noise ratio (in a highly homogeneous field)

 H_2O probes at min. of field range ~ 10 at max. of field range ~ 100 D_2O probes at min. of field range ~ 5 at max. of field range not measured.

Frequency tracking speed

f/f: up to 1%/sec.

Time lag: min. 17 msec.

Both depend on the loop gain, and the maximum tracking speed $(\dot{f}/f)_{max}$ also on the setting of the modulation amplitude. Therefore, the frequency tracking speed and the time lag may be an order of magnitude worse than the optimum values given above.

Loop gain at d.c.: $> 10^5$ (worst case for D_2O probes), typically $> 10^6$

Front panel potentiometer for max. 10 times attenuation of the loop gain.

Coarse-frequency adjustment: 10-turn precision potentiometer.

Fine-frequency adjustment: 10-turn precision potentiometer range: see "Field tracking range".

Error ∀oltage output:

Impedance = $1 k\Omega$

Sens	it	iν	ri	tv

Probe No.	Sensitivity (V/G)
2	8
3	4
4	2
5	1
6	0.65
7	0.32

Maximum output voltage equal to the amplitude of the modulation signal at the scope output, i.e. ± 8 V at maximum modulation setting.

Error voltage indicator

Full scale corresponds to ±10 ppm of the maximum field value of the range.

Required homogeneity of the field

The following table gives the maximum field gradients (in ppm/cm) for which a signal-to-noise ratio results, which just allows automatic frequency tracking.

Probe No.	Field range				
Probe No.	High	Midd1e	Low		
2	600	300	0		
3	1200	900	300		
4	1300	1300	700		
5	750	450	200		
6	250	a)	a)		
7	a)	a)	a)		

te: The field gradient effect on the NMR can be compensated with an appropriate coil, see Appendix.

a) Not measured.

Field tracking range (= search-mode range = fine frequency adjustment range)

 H_2O probes: from 20% to 80% of the frequency range: $\sim \pm 5\%$

: at the extremities of the frequency range: $\sim \pm 1\%$

 D_2O probes: from 20% to 80% of the frequency range: $\sim \pm 1.5\%$

: at the extremities of the frequency range: $\sim \pm 0.3\%$.

Two LEDs indicate the approach of the upper or lower limit, respectively, of the frequency tracking range.

NMR signal output

For scope inspection of the NMR signal.

Output resistance = 10 $k\Omega$ + 10 nF to ground for noise filtering.

NMR signal: negative pulses of 100 mV to 7 V (see Fig. 3).

No strobe signal output

CMOS switch to ground open in the absence of NMR signal:

- switch open: I = 5-500 nA at max. $\pm 20 \text{ V}$
- switch closed: 80 Ω to 100 Ω to ground at max. ± 20 mA.

No strobe signal indicator

LED is ON in absence of NMR signal.

Modulation output

Lemo connector for scope inspection.

Impedance = $1 k\Omega$.

Modulation signal is a 30 Hz triangular waveform.

Amplitude adjustable with external potentiometer: max. ± 8 V, normal setting $\sim \pm 4$ V. Calculation of $B_{\rm mod}$: see "Error voltage sensitivity".

Ext. modulation

INT./EXT. front panel switch.

EXT. input: Lemo connector, $Z_{in} \approx 25 \text{ k}\Omega$, voltage gain = 1, max. input voltage = $\pm 8 \text{ V}$.

RF output for NMR amplifier box

Square wave of 0.8 V peak-to-peak amplitude into 50 Ω .

RF scaler output

NIM standard signal.

Internal frequency counter

7-digit LED display indicating the field strength in gauss.

Resolution: 0.01 G.

Temperature stability: 3×10^{-6} from 0 to 50°C.

Time-base gate length:

- with H₂O probes ~ 0.4 sec
- with D₂O probes ~ 0.6 sec.

NMR amplifier box

RF input: 0.4 V peak-to-peak into 50 Ω (sine or square wave).

RF output: 5 V peak-to-peak square wave into 50 Ω .

NMR signal output: 1 $k\Omega$ output impedance, no noise filter.

Power consumption

+24 V 0.1 A -24 V 0.1 A +12 V 0.1 A -12 V 0.5 A +6 V 0.6 A -6 V 0.2 A.

Figure 14 shows the complete circuit diagram of the NMR electronics in the NIM plug-in and Fig. 15 the frequency counter with display in gauss.

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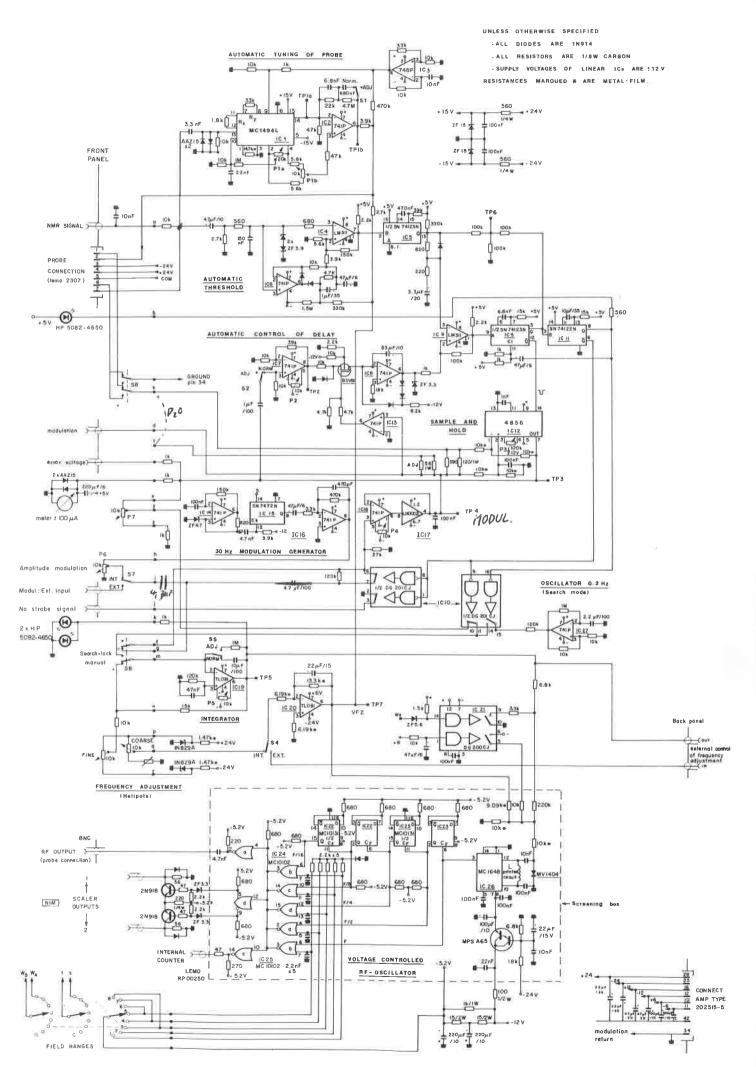


Fig. 14 Circuit diagram of the NMR electronics in the NIM plug-in

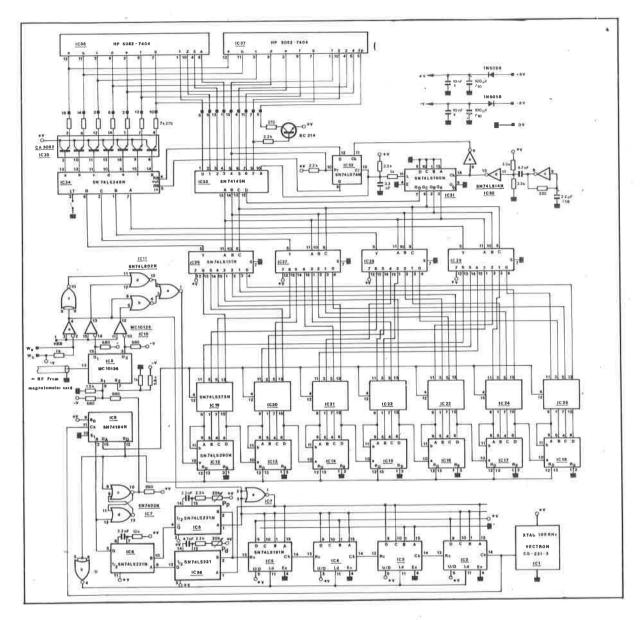


Fig. 15 NMR magnetometer - Frequency counter with display in gauss

COMPENSATION OF FIELD GRADIENTS

' Figure Al shows a possible arrangement for local compensation of field gradients. Two printed circuit coils are mounted, sandwich-fashion, close to the measuring head. The windings of these coils are arranged such that they produce fields parallel to B_0 but of opposite sign on opposite sides of the NMR sample, falling to zero at the centre of symmetry of the windings. A current of 100 mA produces a field gradient of about 3 G/cm and does not change the field reading if the NMR sample is situated in the centre of symmetry of the windings. In this way gradients of B_0 up to about 20 G/cm can be opposed and cancelled by choosing an appropriate current.

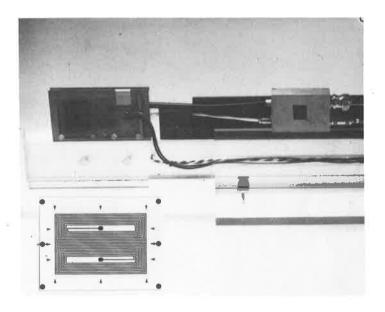


Fig. Al Printed coils for field gradient compensation

