5 kompletnih kopija

N.M.R. Spectrometer

Cat. No. NMR/11

CONTENTS

Circuit Diagram

TECHNICAL SPECIFICATIONS

Power Supply

Voltage	100...120V or 200...240V / 50/60Hz		
Power Consumption	max. 60vA		
Fuses	2 x 2A		
Dimensions overall	Height	7½"	(19cm)
	Width	12¼"	(31cm)
	Depth	10"	(25cm)
	Weight	7 kg	

Oscillator/Detector

Voltage	- 6V, + 100...150V
Operating Frequency	4...35MHz
Modulation Current	125...250mA

Circuit Operation

The circuit is basically that of a marginal oscillator and for maximum sensitivity, the oscillator feedback control must be adjusted to bring the unit to the verge of oscillation.

Dimensions overall	Height	22½"	(57cm)
	(sample probe including head only)	16"	(40cm)
	Width	3"	(8cm)
	Depth	5"	(13cm)
	Weight	1 kg	

Field Sweep Coils

Resistance	approx. 12 ohm per bobbin
Number of turns	400 per bobbin
d	3"
D	4½"

NMR-Magnet

Field strength	approx. 4300 gauss
Pole face diameter	3"
Pole gap	0.625"
Pole face finish	ground and optically finished
Yoke finish	grey-green enamel
Weight	approx. 40 lbs

METHOD OF OPERATION

Before using the spectrometer, a suitable plug must be attached to the power lead with the order connection as follows:

green - ground
red - live
black - neutral

A suitable coaxial plug and a lead for the oscilloscope must then be connected to the spectrometer's output coaxial plug. The next step is to plug the 8-pin connector of the oscillator section into the spectrometer power supply and the 4-pin plug into the oscillator section. The arrangement should be as shown in Fig. 1.

Obtaining an Absorption Signal

Plug the sample coil into the connector at the top of the oscillator probe. With the glycerol sample cell inserted into the probe socket the probe then should be positioned close to the center of the two pole faces. Both the power and modulation switches should be switched on and a few minutes allowed for the instrument to warm up. During this period the oscilloscope should be switched on, with the sensitivity set to read about 0.1V/cm and the time base set to lock-in at about one-fifth of mains supply frequency. Following this, the modulation potentiometer, H.T. potentiometer and the oscillator feedback control should be set to their maximum settings.

If the customer wishes to use his own magnet a field homogeneity of at least of the order of 1 part in 10^3 over the sample probe head volume is required.

From the NMR theory it can be shown that for a magnetic field strength of 4300 gauss the corresponding resonance for glycerol occurs at approx. 18MHz. To obtain this frequency the fine tune control should be set to 90 on the dial for the standard sample coil provided. The oscillator feedback control should be carefully reduced from maximum (fully clockwise) until the oscillation ceases. At this point, approx. 0.4V of noise should be visible on the oscilloscope. The fine tune control should be rotated slowly over the range from 85...100 whereupon the absorption signal should appear. The feedback control can now be adjusted to optimum.

The frequency of the oscillator can be measured with a wavemeter. Sufficient coupling for this measurement is provided by connecting a wire between the wavemeter and the oscillator instrument case.

N.M.R. THEORY

The atomic nucleus of an atom possesses a rotary spin which may be likened to the spin of a child's top. As a result of this spin the nucleus has angular momentum of value $I\ h/2\pi$, where I is the nuclear spin number which depending on the nucleus is equal to an integer or half integer, and h is Planck's constant. It is well known that an electric current is in fact a moving electric charge and that associated with a loop of current is a magnetic moment. So in the case of the nucleus, where the nuclear spin and electric charge constitute a circulating electric charge. This results in some nuclei having a magnetic moment whose value is approximately equal to the nuclear magneton (β_n). Where

$$\beta_n = \frac{eh}{4\pi m_n c} \qquad (1)$$

with e the electronic charge
m_n the mass of a proton
c the velocity of light

As in the case of the nucleus the electron also possesses angular momentum and electric charge which gives rise to a magnetic moment. The value of this is approximately equal to the Bohr magneton (β_e). where

$$\beta_e = \frac{eh}{4\pi m_e c} \qquad (2)$$

with m_e the mass of the electron,
equations 1 and 2 combine to give

$$\frac{\beta_e}{\beta_n} = \frac{m_n}{m_e} \qquad (3)$$

With the mass of the proton 1836 times greater than that of the electron it is seen that β_e is of the order of 10^3 greater than β_n.

Resonance

The magnetic moment μ of the nucleus is along the axis of angular momentum, that is, along the axis about which the nucleus spins. When this nucleus is placed in a magnetic field H, magnetic energy is given to the system of value

$$E \text{ (energy)} = -\mu H \cos \theta \tag{4}$$

where θ is the angle between the direction of H and μ. The gyromagnetic ratio γ is defined as the ratio of the nuclear magnetic moment μ to the nuclear angular momentum P. That is

$$\gamma = \frac{\mu}{P} \tag{5}$$

substituting this into equation 4 gives

$$E = -\gamma H P \cos \theta \tag{6}$$

where $P \cos \theta$ is the component of angular momentum along the direction of H, which can be written as $M_I \frac{h}{2\pi}$.

This then gives

$$E = -\gamma H M_I \frac{h}{2\pi} \tag{7}$$

where M_I is called the nuclear magnetic quantum number. Differentiating both sides of equation 7 leads to

$$d E = -d \left(\gamma H M_I \frac{h}{2\pi}\right) = -\gamma H \frac{h}{2\pi} dM_I \tag{8}$$

This equation expresses the change in energy associated with a change in orientation of the nuclear magnetic moment μ in the field H.

For the proton $dM_I = \pm 1$, or expressed classically, the proton's magnetic moment can have just one of two directions. This is either parallel or antiparallel to the magnetic field H. The parallel arrangement being the lower energy state, with the energy difference between the two states given by equation 8 as

$$d E = \gamma \ H \frac{h}{2\pi} \tag{9}$$

To bring about this energy transition it is necessary to feed to the system energy of value dE for every transition. Quantum mechanics gives the relation between this energy and the frequency of a radio wave needed to bring about the transition. This states that

$$d E = h\nu \tag{10}$$

where ν is the frequency of the radio wave. Equations 9 and 10 combine to give

$$\nu = \frac{\gamma \ H}{2 \ \pi} \tag{11}$$

In the NMR apparatus the probe head contains the resonant material which for glycerol is protons. Thus when the probe head containing glycerol is inserted into a magnetic field H, the protons line themselves along the direction of H. Also housed in the probe head is the oscillator coil which subjects the protons to a radio wave at the oscillator frequency. When the radio wave frequency is such that equation 11 is satisfied, movement of the protons from the lower to higher energy state occurs. This results in energy being absorbed from the oscillator circuit which is observed as a change in the operating condition of the oscillator. To obtain an alternating signal from the apparatus it is necessary for the resonance condition $\nu = \gamma \frac{H}{2\pi}$ to repeatedly occur. This is achieved by superimposing on H a small alternating magnetic field $H_0 \sin \omega t$, whose value should be of the order of a few gauss.

Determination of gyromagnetic constants

The proton's gyromagnetic ratio is given as 2.675×10^{4} radian $\sec^{-1}$ gauss^{-1}, using this value and the frequency of the oscillator, equation 11 gives the magnetic field as

$$H = 2.3487.10^{-4}\ \nu \quad \text{gauss} \tag{12}$$

where ν is in c/s. Alternatively the magnetic field H can be measured with a flux meter and the proton's gyromagnetic ratio obtained from equation 11. That is

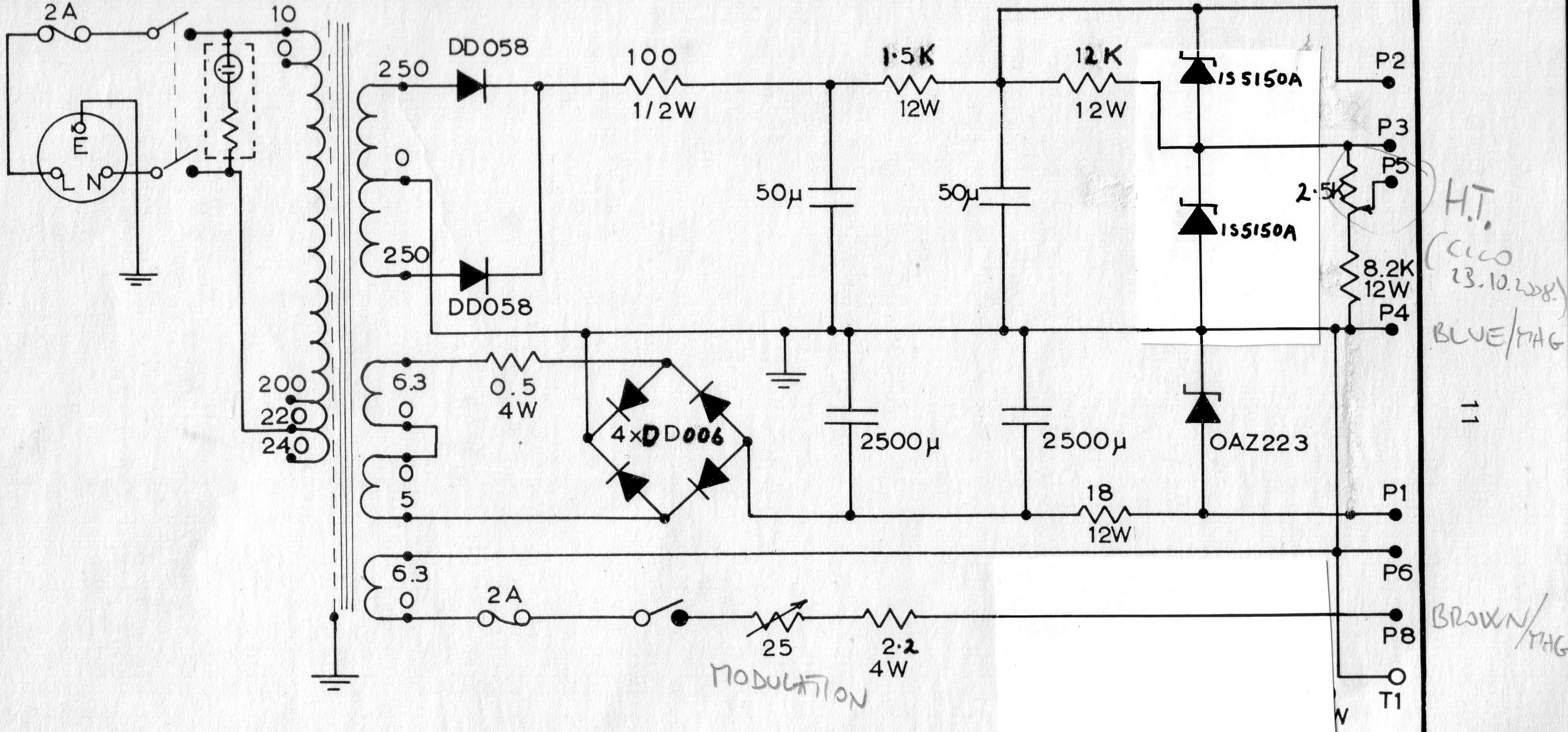
$$\gamma = \frac{2\pi\nu}{H} \tag{13}$$

Having obtained the gyromagnetic ratio γ of the proton the magnetic moment can be obtained. Equation 5 gives

$$\mu = \gamma\ p = \gamma\ \frac{I\ h}{2\pi} \tag{14}$$

Use as a proton magnetometer

As there is a linear relationship between the magnetic field strength and the absorption frequency it is possible to use the NMR as a magnetometer. The frequency range corresponding to a magnetic field strength of 1 to 10 K gauss is 4...40MHz. This would necessitate the use of several different probe heads. Using glycerol as the sample the conversion factor is $4.25\frac{\text{MHz}}{\text{KGauss}}$ e.g. 18 MHz corresponds to a field strength of 4300 gauss. Hence by measuring the absorption frequency it is possible to calibrate the fine tune control in gauss.



NMR POWER SUPPLY SCHEMATIC

200/250 Volt model shown; 100/130 Volt model transformer primary: 0, 5, 105, 115, 125 Volts

23-24.10.2008. (M.FURIĆ, M.BASLETIĆ, R.VARGA)
+ OSTALI

* NIJE RADILA VJEŽBA

* RAZLOG: 1K POT JE BIO PREMOŠTEN S (VELIKIM) OTPORNIKOM OD 100 Ω (VIDI SHEMU ZA OSC)

* NAPRAVILI: · BACILI 100Ω OTPORNIK VAN
· POŠPRICALI POSTOJEĆI 1K POT

* NAKON TOGA STVAR RADI:

FINE TUNE ≃ 24
OSC FEEDBACK = FULL CLOCKWISE
} OSCILLATOR

H.T. = 10
MODULATION = 10
(WHATEVER)
} NMR POWER SUPPLY

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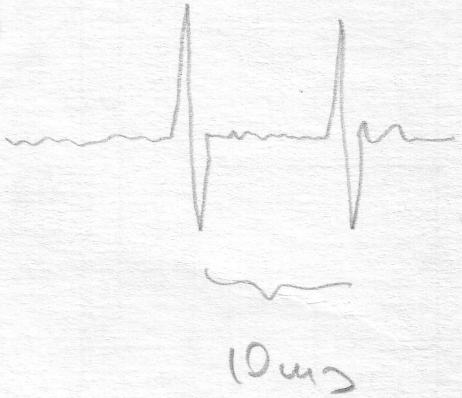
1 V/DIV
2 ms/DIV
} OSC



NMR-OSCILLATOR SCHEMATIC