# Automatically Tuned Probe Head System for Pulsed NQR Spectroscopy in Extreme Thermodynamic Conditions *

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A complete NQR probe-head system operating in the frequency ranges 0.5–150 and 150–300 MHz is described. The head is particularly suited for NQR experiments carried out at a remote location, for example in a low-temperature cryostat or high-pressure chamber. Moreover, the head system includes a microprocessor-controller for automated tuning of the probe to the operating frequency of the associated NQR spectrometer. The controller can be easily interfaced to a PC via standard serial port.

*Key words:* Nuclear quadrupole resonance, Pulsed spectrometers, Instrumentation

## Introduction

A pulsed NQR experiment often involves searching for weak NQR lines of unknown frequencies. In particular, at low NQR frequencies (for example in the case of $^{14}$N NQR) the observation window on the frequency scale is determined by the bandwidth of the resonant circuit in the probe rather than by the effective bandwidth of the r.f. excitation pulse applied to the sample. In addition, for optimal detection of weak NQR signals the receiver bandwidth is usually decreased to match the NQR linewidth. In such cases one has to re-tune manually the probe approximately as many times, as many spectrometer frequency bands can be contained within a frequency range to be scanned. Therefore, automation of the tedious probe tuning procedure would facilitate automated search for unknown NQR lines in a computer-controlled NQR spectrometer.

On the other hand, NQR experiments may have to be carried out in extreme thermodynamic conditions: low temperature or high pressure. This may imply placing the sample coil and the probe head circuit 50–80 cm apart. In a simple and often used scheme the sample coil is connected to the end of the coaxial cable which forms part of the resonant circuit. The disadvantage of this solution is, however, worsening of the sensitivity inherent in the circuit [1]. The probe head system described in the present paper was designed to meet the above mentioned requirements.

## Circuit Description

The NQR probe uses a series-tuned resonant circuit with piston-type 5-100 pF variable capacitor (Figure 1). Instead of a conventional matching capacitor, a broadband transmission-line type r.f. transformer is used for matching the low impedance of the resonant circuit to a standard 50 Ω impedance of the r.f. pulse transmitter. The voltage transformation ratio of the transformer is 3:1, the corresponding impedance-transformation is 50/5.6. The NQR sample coil in series resonant circuit is connected to the low impedance (5.6 Ω) port of the transformer by means of a transmission line formed with nine coaxial 50 Ω -cables connected in parallel. The net characteristic impedance of this line is therefore 5.6 Ω. The line is

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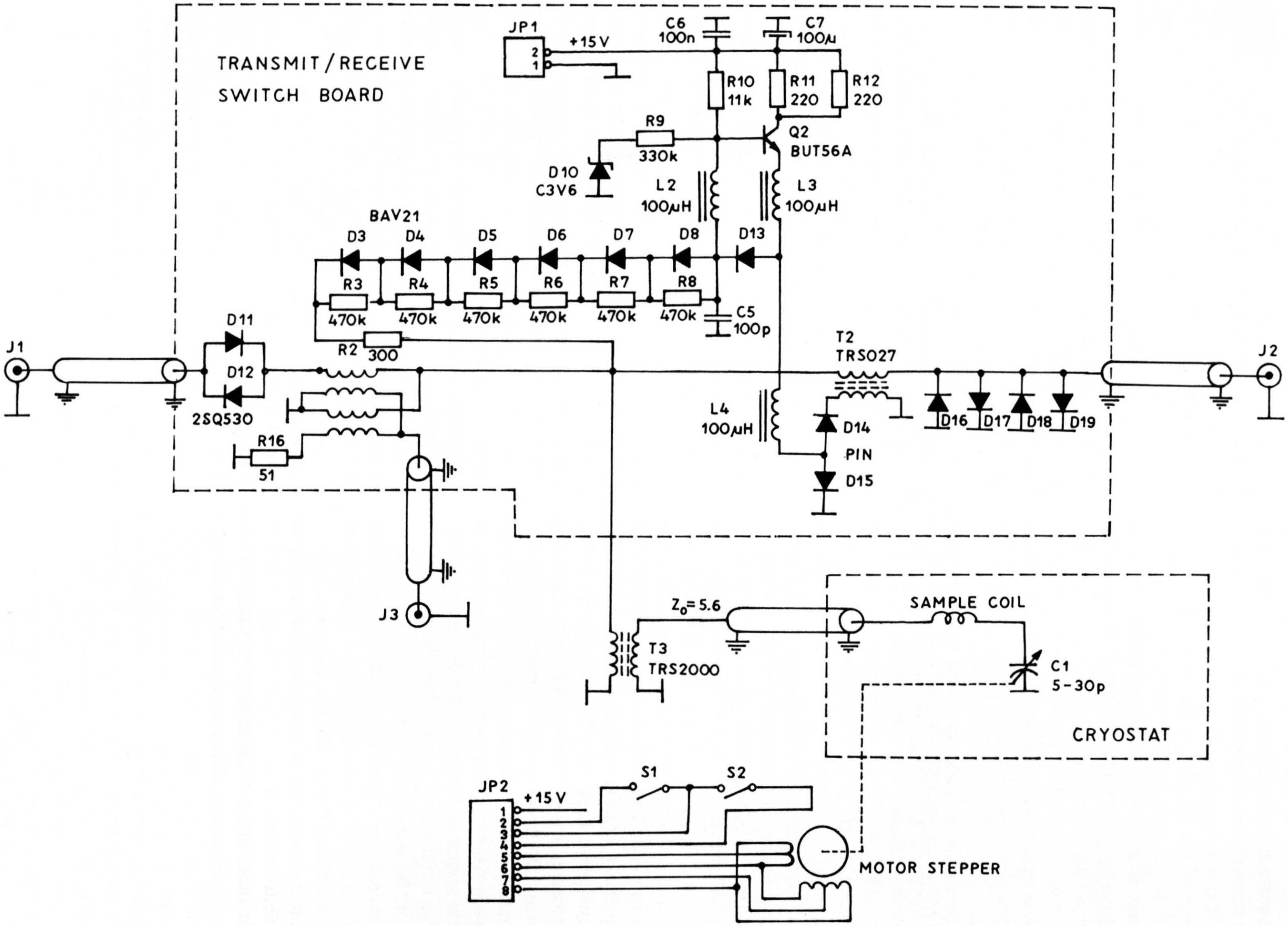


Fig. 1. Circuit diagram of the probe head. J1 – r.f. input (from transmiter); J2 – r.f. output (to receiver); J3 – r.f. tuning voltage output (from directional coupler to stepper motor controller).

matched both to the transformer and the resonant circuit, therefore its length (approx. 80 cm) does not affect the inductance of the sample coil. Also, because this line is not resonant, it does not deteriorate the noise figure of the system. In fact, perfect matching assumes that the characteristic impedance of the resonant circuit is also 5.6 Ω over the whole operating frequency range of the probe. Although this condition cannot be fulfilled accurately, because the circuit L/C ratio varies when tuning the probe; the resulting probe mismatch is negligible as indicated by the reasonable value of the SWR (smaller than 1.5) measured at the probe input. A broadband transmit/receive circuit, developed by Engle [2] for applications in NMR spectrometer for liquids, is used for protecting the receiver in the transmit mode. The original circuit has been slightly modified to withstand the increased power of the r.f. transmitter ($P \geq 2$ kW) in the present case. For the duration of the transmitter r.f. pulse the diodes D3–D8 in series and the 100 pF capacitor C5 make up a peak detector. The negative voltage on the peak detector turns the Q2 transistor off and the D13 diode conducts, allowing the peak detector's negative voltage to turn each of the PIN diodes (D14 and D15) hard off, so that they disconnect the primary winding of the transmission line transformer T2 from ground. With its primary winding open the secondary winding of the transformer acts like a high-impedance choke, and therefore as an open circuit. Thus the receiver is protected and the high power transmitter pulse passes almost unattenuated to the probe. The crossed diodes D16–D19 serve to ground any residual transmitter pulse at that point and protect the receiver input transistor. The receive time occurs after about 2 μs after the end of the transmitter pulse when the peak detected voltage at the 100 pF capacitor disappears. The transistor Q2 is then turned on by a bias current through the R10 resistor into the base. The current out of the emitter is set by the 110 Ω resistor to be about 100 mA. Each of the PIN diodes gets one half of this forward current causing it to be equivalent to a very low resistance. The transmission line transformer thus has its primary winding effectively at ground during receive time. The transformer's secondary winding now presents a low impedance and the NQR signal from the probe passes practically undiminished to the receiver. Because this signal is much weaker then the transmitter residual voltage, both pairs of crossed diodes (D16–D19) are nonconducting and effectively out of the circuit.

The tuning capacitor is driven by a stepper motor via the reduction gear. The position of the capacitor piston is controlled by an appropriate current pulse sequence produced by a microprocessor-controller and applied to stepper motor coils. This controller also realizes the algorithm of automatic tuning of the probe to the frequency of r.f. pulses applied to the sample coil. The tuning signal is derived from a directional coupler, shown in Fig. 1, or from a small pick-up loop located near the coil. The advantage of using the coupler is that no additional seal leads from inside the cryostat or pressure chamber are required. On the other hand, the coincidence of the maximum of the r.f. signal induced in the pick-up loop with optimal tuning of the resonant circuit is not affected by impedance matching conditions in the probe. For a directional coupler this is not the case because the shape of the tuning signal is frequency dependent.

Figure 2 shows the block diagram of the stepper motor controller with automatic tuning control circuit. Because this circuit uses a pulsed tuning voltage, the sample-and-hold (S & H) circuit of the ADC must be switched synchronously with the output of the pulse programmer of the associated NQR spectrometer. The S & H sampling pulse is normalized to optimal duration by the monostable multivibrator shown in Figure 2. The r.f. output of the transmitter is automatically attenuated by 10 dB by the spectrometer control software until the probe tuning process is completed. Otherwise the excessive r.f. power reflected from the probe input could cause electric damage to the probe or the transmitter. After broadband r.f. amplification and diode detection the pulse tuning voltage is converted into DC voltage measured by the 8-bit AD converter (ADC). A peak-picking algorithm programmed in the microprocessor ROM is used to find the maximum (or minimum when using a directional coupler) of the ADC code, which corresponds to probe tuning condition. This algorithm also accounts for the total mechanical clearance in the gear when the direction of the stepper motor revolution is reversed. According to Faraday's law, the tuning voltage induced in the pick-up loop varies significantly with frequency, and the ADC output is also used to control the gain of the broadband r.f. amplifier, so that the optimal tuning conditions are independent of the spectrometer operating frequency. For this purpose the digital automatic gain control (AGC) loop is formed by an electronic r.f. attenuator, r.f. amplifier, diode detector, ADC, microprocessor, and

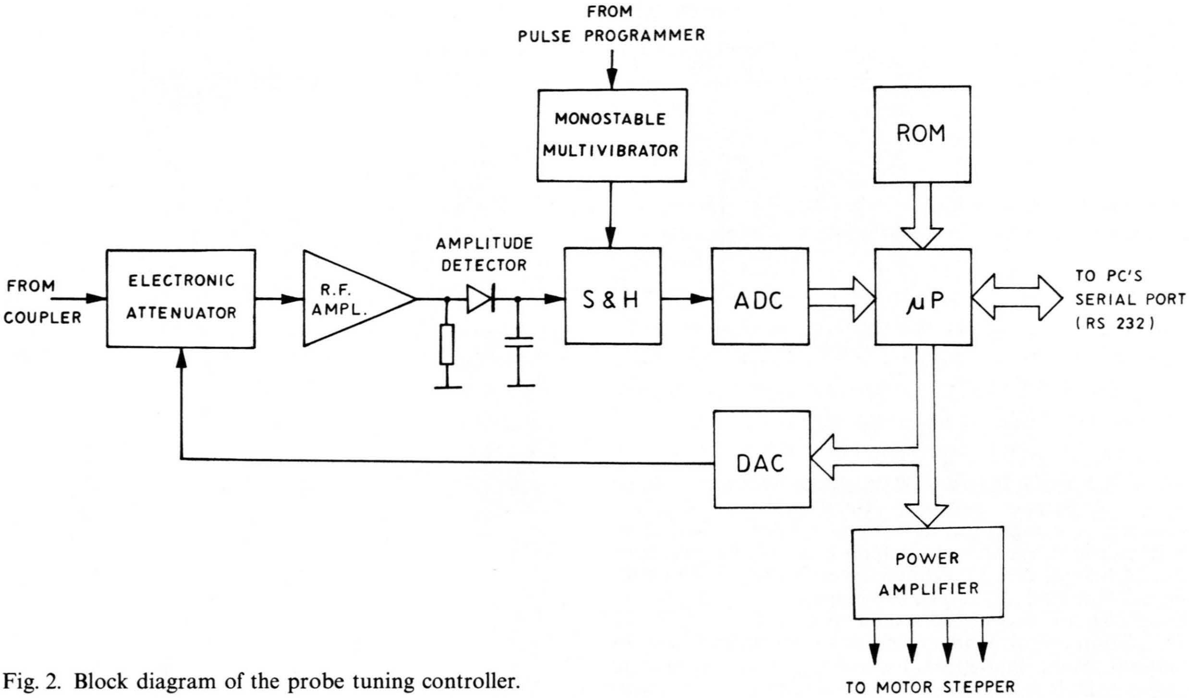


Fig. 2. Block diagram of the probe tuning controller.

DAC which produces DC control voltage for the r.f. attenuator. The electronic attenuator (using PIN diode network), broadband r.f. amplifier, and matching transformer are packaged components manufactured by MBC Electronics (Warsaw, Poland). The controller receives one-character ASCII commands from a PC's serial port and performs the required tuning operations. A command for reading the tuning voltage code is also provided. This can be used for plotting the probe frequency response on a computer's screen when the operating frequency is swept by spectrometer control software.

We have also constructed a simplified version of the probe head, using a commercial vacuum variable capacitor (Jennings) and a simple heat-leak type cryostat operating down to 77 K. Both versions operate in the frequency range 0.5 to 150 MHz. For the frequency range of 150–300 MHz a third probe head version has been constructed, in which the lumped parameters resonant circuit is replaced by a conventional piston-tuned cavity resonator. This version is equipped with an above mentioned heat-leak type cryostat.

The mechanical construction of the probe is shown in Figure 3. The electronic parts and the stepper motor with a gear assembly are encapsulated into a $(6.5 \times 12.5 \times 26.5)\ cm^3$ brass casing. A combined toothed-worm-and-nut steering gear (3) is used to transform the rotational motion of the stepper motor (2) to a linear motion of the brass rod (8) connected to the piston capacitor (10). When the capacitor piston reaches its upper or lower end positions, one of the corresponding limit switches (4) will be activated to stop the stepper motor unconditionally in order to prevent mechanical damage of the probe. The cold part of the probe has the form of a 60 cm long tail (9) which can be inserted into a commercial cryostat. The cryostat is fastened to the probe by the flanged nut (5) with "O-ring" gasket (6).

## Application and Performance

The described NQR probe head system has been used in cooperation with the MBC's NQS 1/150 and NQS 1/300 FT NQR spectrometers. The probe-head has been inserted into a commercial NMR helium-flow cryostat CF 1200 of internal diameter 6 cm and length of 88 cm produced by Oxford Instruments. The good performance of the system was indicated by reliable and reproducible results of tuning the probe and

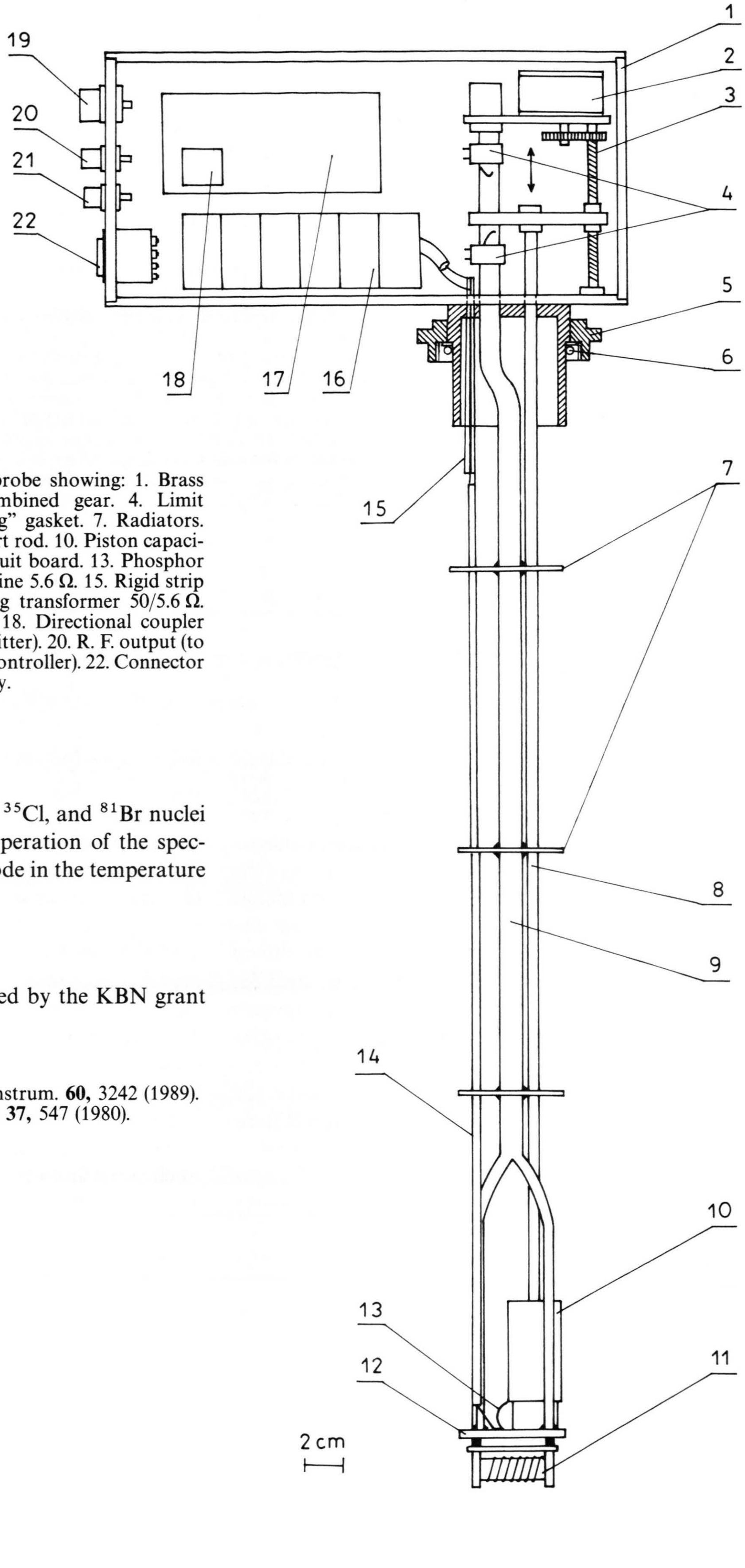


Fig. 3. Schematic diagram of the probe showing: 1. Brass casing. 2. Stepper motor. 3. Combined gear. 4. Limit switches. 5. Flanged nut. 6. "O-ring" gasket. 7. Radiators. 8. Brass rod. 9. Stainless steel support rod. 10. Piston capacitor. 11. Sample coil. 12. Printed circuit board. 13. Phosphor bronze tape lead. 14. Transmission line 5.6 Ω. 15. Rigid strip line 5.6 Ω. 16. Broadband matching transformer 50/5.6 Ω. 17. Transmit/receive circuit board. 18. Directional coupler (option). 19. R.F. input (from transmitter). 20. R. F. output (to receiver). 21. R.F. tuning output (to controller). 22. Connector for stepper motor and power supply.

recording NQR spectra of $^{14}N$, $^{35}Cl$, and $^{81}Br$ nuclei during long-term unattended operation of the spectrometers in fully automated mode in the temperature range 4.2–400 K.

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