JOURNAL OF MAGNETIC RESONANCE **60,** 397–404 (1984)

A High-Resolution NMR Probe in Which the Coil and Preamplifier Are Cooled with Liquid Helium*

P. STYLES AND N. F. SOFFE

Department of Biochemistry, University of Oxford, South Parks Road,
Oxford OX1 3QU, United Kingdom

AND

C. A. SCOTT, D. A. CRAGG, F. ROW, D. J. WHITE, AND P. C. J. WHITE

Rutherford Appleton Laboratory, Chilton, Didcot, Oxfordshire OX11 0QX, United Kingdom

Received May 1, 1984

In a well designed NMR spectrometer, the noise originates predominantly from the resistance of the receiver coil. Significant improvements in sensitivity can be achieved by cooling the coil to cryogenic temperatures, provided that a preamplifier can be designed to match the coil's performance. A probe is described in which the coil and preamplifier are cooled with liquid helium, but the sample is maintained at room temperature. Carbon-13 spectra at 45 MHz demonstrate improved sensitivity over conventional probes at the same field. © 1984 Academic Press, Inc.

In a well designed NMR spectrometer the sensitivity is limited by the thermal noise, most of which comes from the resistance of the receiver coil. The analysis of Hoult and Richards (*1*) demonstrates that, for a nonconducting sample, significant improvements in sensitivity could be achieved by cooling the coil with liquid helium. An idea of the potential improvement can be obtained by considering the root mean square voltage (V) from a resistance (R). At temperature T the noise is given by

$$V = \sqrt{4kTRB} \qquad [1]$$

where k is Boltzmann's constant and B is the receiver bandwidth. Since the resistivity of normal coil materials falls at low temperatures the noise is reduced because of the fall in R as well as in T. For a reduction in coil temperature from 290 to 4.2 K the resistance at radiofrequencies of a typical coil in a magnetic field of a few tesla falls by a factor of about 7. The noise thus falls by a factor of about 22.

In practice this factor is reduced by the noise contribution from the preamplifier and by the loss of filling factor that will inevitably be suffered due to the necessity of placing the receiver coil in a vacuum vessel. With regard to the preamplifier

---

* This work was presented at the 24th ENC, Asilomar, California, April 1983.

0022-2364/84 $3.00

This article is a reprint of a previously published article.
For citation purposes, please use the original publication details;
Journal of Magnetic Resonance 60, 397-404 (1984)

DOI of original item: doi: 10.1016/0022-2364(84)90050-7

there are fortunately semiconductor devices which operate with acceptable noise performance at low temperatures (*2–5*). A theoretical assessment of these factors suggests that a cryogenically cooled receiver could achieve an improvement of some eight times over an equivalent room-temperature system.

In this report we describe an NMR probe in which both the receiver coil and preamplifier are enclosed in a cryostat and cooled with liquid helium. The sample, however, is at room temperature. This should be contrasted with other cooled NMR probes where the sample is also cooled and optimum receiver sensitivity is not of prime importance (*6–9*). The probe is designed for ^{13}C NMR at 45.9 MHz and fits in the 83 mm bore of a 4.3 T magnet. Facilities for sample spinning and proton decoupling are incorporated in the room-temperature part of the probe along with the transmitter coil. The probe is designed for 10 mm sample tubes. Larger tubes would have improved the filling factor, but considerations of sample availability and decoupling power reduce their usefulness.

CONSTRUCTION

The probe cryostat is designed to be completely independent of the magnet cryostat so that it can be easily interchanged with standard probes. The room-temperature assembly, incorporating the transmit and decoupling coil and the spinner, aligns with the cryostat and is inserted from the top of the magnet bore. The general arrangement is shown in Fig. 1 and details of the construction around the sample tube are shown in Fig. 2. The sample tube hangs inside a re-entrant glass cap which is part of the vacuum envelope of the cryostat. The saddle-shaped Helmholtz receiver coil is positioned in the annular vacuum space inside the cap, but makes no contact with the glass. A coaxial transmission line made from copper tubes supports the coil and connects it electrically to the preamplifier which is

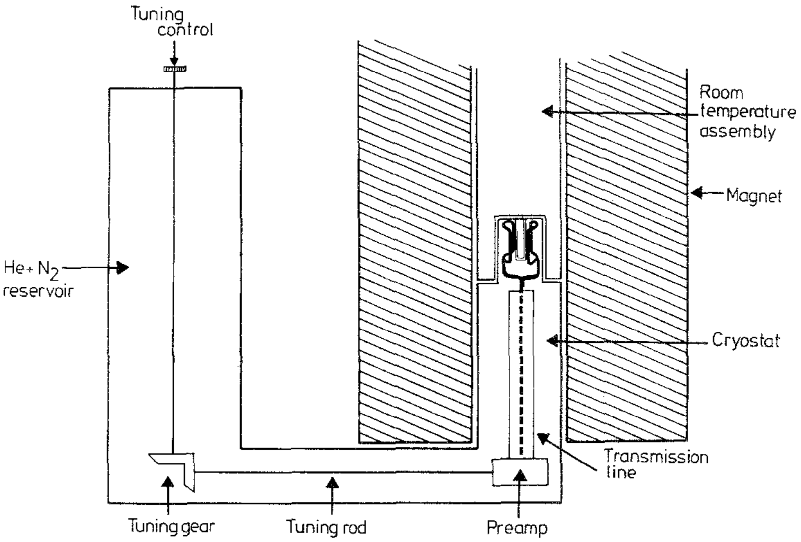

FIG. 1. Schematic representation of the arrangement of cryostat, magnet, and room-temperature assembly.

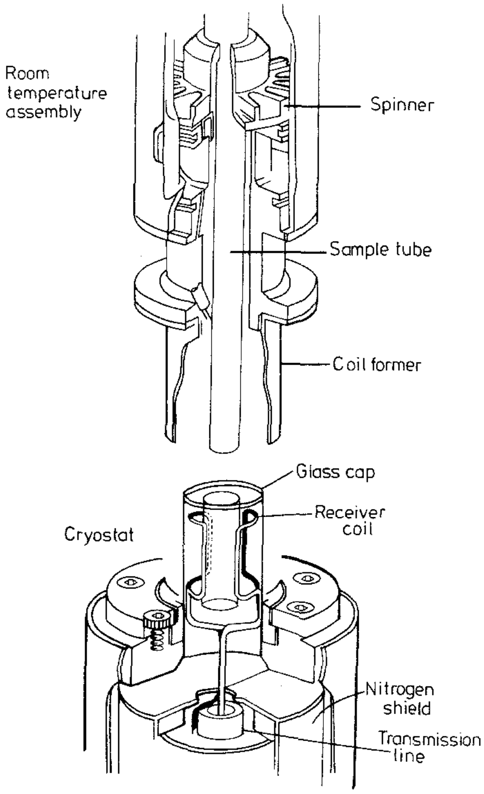

FIG. 2. Detail of the cryostat and room-temperature assembly in the vicinity of the coil.

immersed in helium. The coil is wound from 1 mm o.d. silver tube and the surface is polished to reduce the radiation heat load. Cooling of the receiver coil is effected by pumping liquid helium through the hollow centers of the inner transmission line conductor and the coil. A reservoir of liquid helium is held in the main part of the cryostat. The helium reservoir and the transmission line are surrounded by a nitrogen cooled radiation shield in keeping with normal cryogenic practice.

THE PREAMPLIFIER

Figure 3 shows the circuit of the preamplifier. The active device is a gallium arsenide field effect transistor (GAT1/010 from Plessey Optoelectronics and Microwave Limited, Wood Burcote Way, Towcester, Northamptonshire, UK). The coil is tuned with a combination of fixed capacitors at each end of the transmission line and a variable capacitor close to the FET. The variable capacitor can be adjusted from outside the cryostat, by means of rods and bevel gears, and tunes the probe over a range of about 4 MHz. At room temperature the Q of the coil and input circuit is about 150, but this rises to about 1000 in operation at cryogenic temperatures.

The tuning scheme that we have described differs from the conventional practice of treating probe and preamplifier as separate "black boxes" with each tuned to allow interconnection with 50 Ω cable. Instead, the tuning components including

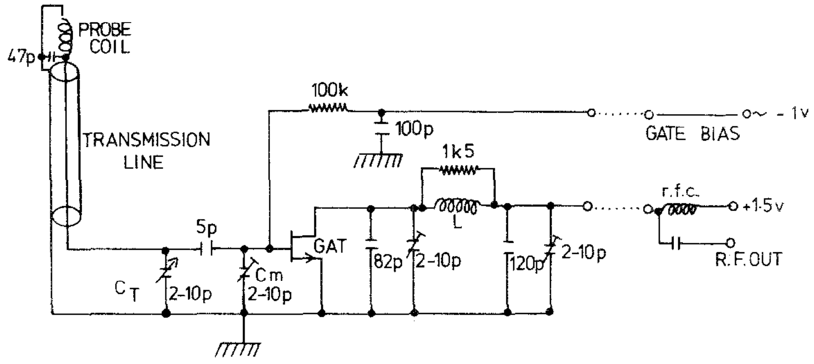

FIG. 3. Circuit diagram of the helium-cooled receiver coil and preamplifier. The inductance L consisted of 9 turns of 28 swg wire, close wound with a diameter of 6 mm.

the transmission line effect the impedance transformation required to present the FET with an impedance consistent with good noise performance. The change of approach is required because of the difficulty in providing rotating mechanical linkages to the coil end of the cryostat. It will be noted that no provision is made to adjust the impedance match after the preamplifier has been installed. This is because we have measured the noise performance of the FET to be almost constant in the range of 1 to 10 kΩ at helium temperature. Although the impedance transformation of the input circuit at resonance is dependent on the circuit Q, which will vary with coil temperature and sample, by choosing C_m to match the coil to 5 kΩ with a Q of 1000, variations of operating Q will affect the gain but not the noise performance of the circuit.

The most useful measure of the noise performance of an amplifier used with a variable temperature source impedance is its effective input noise temperature, T_a. A perfect amplifier with a source at temperature T_s gives a noise voltage at its output proportional to $\sqrt{T_s}$. T_a is defined by regarding the extra noise observed at the output of a real amplifier as being due to an apparent temperature increase at the source so that the total output noise is proportional to $\sqrt{(T_s + T_a)}$. In terms of the more usual noise figure F (dB) (measured with the source at 290 K), T_a is given by

$$T_a = 290(10^{F/10} - 1). \qquad [2]$$

The noise temperature of the amplifier shown in Fig. 3 has been measured as 7 K at 45.9 MHz. The measurement was made with the amplifier and all its input circuity immersed in liquid helium.

THE ROOM-TEMPERATURE ASSEMBLY

This part of the apparatus, which is inserted from the top of the magnet bore, incorporates the transmitter coil, tuning capacitors, spinner, and air jets for temperature control of the sample. The transmitter coil is a single-turn saddle-shaped coil of the type suggested by Dadok (*10*). It is double tuned to 45.9 MHz for ^{13}C and 182.4 MHz for proton decoupling. The assembly can be rotated with respect to the cryostat to make the transmitter and receiver coils orthogonal. The isolation of about 35 dB achieved by this crossed-coil arrangement is the only protection for the preamplifier from the transmitter pulse.

PERFORMANCE

The improvement in sensitivity that has been obtained by cryogenic cooling is demonstrated in Fig. 4. The sample is the ASTM ^{13}C test sample which contains 60% C_6D_6 and the two spectra were recorded using geometrically similar coils. The

FIG. 4. Single-scan ^{13}C spectra of the C_6D_6 triplet in the ASTM standard sample. The upper spectrum was recorded with the cryogenically cooled probe, and the lower spectrum with a room-temperature coil of similar geometry. The signal-to-noise ratios, as determined by the Nicolet software routine, were 250:1 and 30:1, respectively.

upper spectrum was obtained with the cryogenic probe that we have described, while the lower one was obtained with the coil at room temperature, matched to 50 Ω in the conventional manner and connected to a preamplifier with a noise temperature of 180 K (2.1 dB noise figure). The expected improvement in sensitivity can be obtained from the analysis of Hoult and Richards (*1*) which shows that the ratio of signal to noise is given by

$$S/N \propto \frac{1}{a}\left(\frac{p}{(T_a + T_c)l\zeta}\right)^{1/2} \frac{\omega_0^{7/4}}{\rho(T_c)^{1/4}} \qquad [3]$$

for our coil geometry and a standard sample of fixed volume. In this expression a is the radius of the coil; p and l are the perimeter and length of the conductor from which the coil is wound; ζ is the proximity factor for the coil; T_a is the effective noise temperature of the preamplifier; T_c is the temperature of the coil; $\rho(T_c)$ is the resistivity of the coil material and ω_0 is the operating frequency of the spectrometer. Since the geometry of the room temperature and cold coils is similar, only the terms $(T_a + T_c)$ and $\rho(T_c)$ are important in comparing the spectra in Fig. 4. For a temperature change from 290 to 4.2 K the resistivity changes by a factor of 50 in a field of 4.3 T. Thus we expect S/N to improve by a factor of 17 on cooling. This is about double the value that we have obtained so far.

Additionally, this result makes no allowance for filling factor, or the relative efficiency of our saddle-shaped coil compared to a modern commercial probe. Direct comparisons with the latter are further complicated because different manufacturers use different lengths of sample within the coil. Nevertheless, comparisons cannot be avoided, and current literature from Bruker and Varian quote sensitivities of 90 and 150, respectively, at 4.8 T. These figures extrapolate to 75 and 120 at 4.3 T using the $\omega^{7/4}$ frequency dependence given in Eq. [3]. The effect of using larger coils with a fixed sample volume can also be obtained from Eq. [3]. As well as the obvious inverse dependence of S/N on coil radius, a, there is a further decrease because the length of conductor, l, in the coil increases with radius. The total effect is that S/N is proportional to $a^{-3/2}$. Our coil has a mean radius of 9.5 mm compared with about 5.5 mm in a commercial coil. Hence we expect to lose a factor of about 2.3 because of our necessarily oversize coil. The implications of these results and predictions are discussed below.

The C_6D_6 spectrum is an artificial case in ^{13}C spectroscopy in that no proton decoupling is involved. Since it is common to use decoupling powers of several watts it might be expected that this would degrade the performance of the cooled receiver. The dioxan spectra of Fig. 5 demonstrates that this is not the case. There is no extra noise in the decoupled spectrum.

DISCUSSION

If a cryogenically cooled probe is to give a significant improvement in sensitivity over conventional technology, there are two requirements to be met. First, the theoretical low-temperature electrical performance must be realizable in the environment of an NMR experiment. Second, the receiver coil design must deviate as

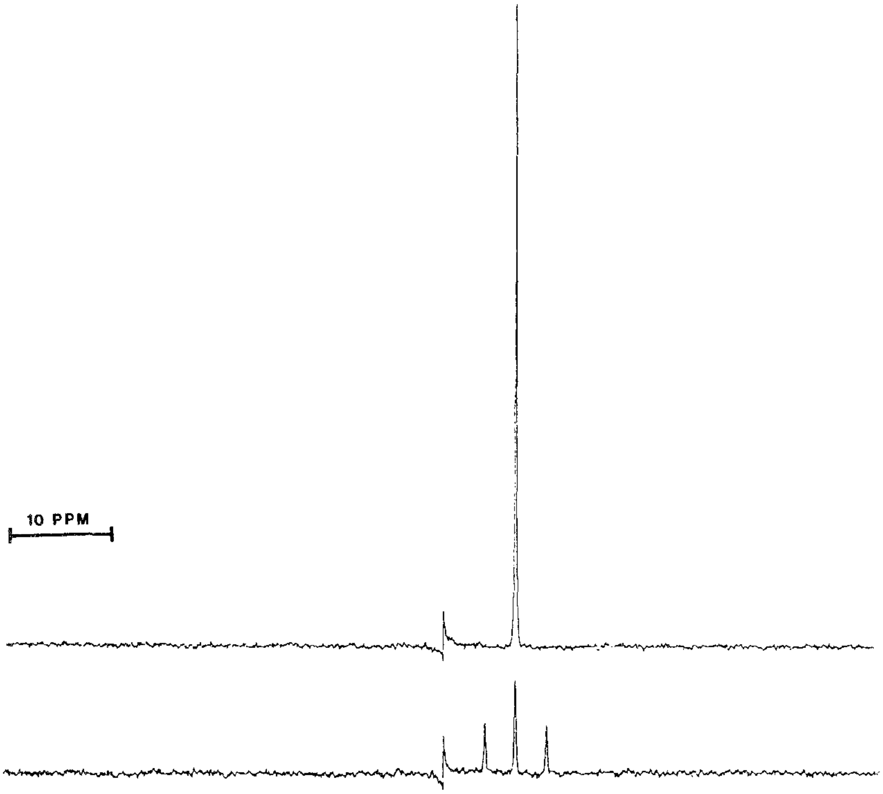

FIG. 5. Single-scan spectra of a dilute dioxan sample showing the results of phase-modulated broadband proton decoupling.

little as possible from a conventional coil in terms of filling factor and Q (measured at room temperature).

Low-temperature electrical performance. The results that we have so far achieved show a disappointing discrepancy between the predicted and realised improvement in sensitivity due to cooling. We appreciate that there are several mechanisms which could account for this, including inductive coupling between the coil and surrounding conducting materials (*1*), interaction between transmitter and receiver circuits and thermal gradients in our cryostat. We are currently modifying the cryostat to assess the relative contributions of such effects.

Receiver coil design. Using the known $a^{-3/2}$ dependence of sensitivity on coil size, the receiver coil performance at room temperature is in reasonable agreement with prediction based on the published Bruker sensitivity. However, it is unlikely that our coil represents the best design that can be achieved. In particular, we use relatively thin capillary tubing (selected because of availability, and to minimize the radiation heat load), and the coil down leads are rather long. A further problem is that differential thermal contractions in our cryostat make it difficult to position the coil accurately which limits the filling factor. All of these aspects are amenable to improvement, and it is hoped that further gains in sensitivity will be possible.

CONCLUSIONS

We have demonstrated that it is possible to construct a high-resolution NMR probe in which the sample is at room temperature, but the receiver coil is cooled with liquid helium. This arrangement has been shown to give a significant improvement in sensitivity over conventional probes operating at the same frequency (between 2.5 and 4 times better). We believe that these figures can be further improved, perhaps by a factor of 2, which would mean at least a 25-fold reduction in experimental time.

It must also be noted that these experiments were performed at a relatively modest field strength. Operating frequency was chosen partially on the basis of magnet availability, but mainly because we wished to evaluate the technique without the added difficulties of working at higher frequencies. Now that the feasibility of constructing a viable cryogenically cooled probe has been demonstrated, we hope to extend the technique to higher frequencies.

ACKNOWLEDGMENTS

This work was supported by the Science and Engineering Research Council, UK in the form of a collaborative grant to the Rutherford Appleton Laboratory and the University of Oxford. The authors thank Sir Rex Richards, Dr. George Radda, Dr. Iain Campbell, and Dr. Gordon Walker for their help and support.

REFERENCES

1. D. I. Hoult and R. E. Richards, *J. Magn. Reson.* **24,** 71 (1976).
2. B. Lengeler, *Cryogenics* **14,** 439 (1974).
3. D. I. Hoult and R. E. Richards, *Electron. Lett.* **11,** 596 (1975).
4. H. Ahola, G. J. Ehnholm, P. Östman, and B. Rantala, *J. Low Temp. Phys.* **35,** 313 (1979).
5. A. Long, T. D. Clark, R. J. Prance, and M. G. Richards, *Rev. Sci. Instrum.* **50,** 1376 (1979).
6. D. S. Miyoshi and R. M. Cotts, *Rev. Sci. Instrum.* **39,** 1881 (1968).
7. D. W. Alderman, *Rev. Sci. Instrum.* **41,** 192 (1970).
8. M. Linder, A. Höhener, and R. R. Ernst, *J. Magn. Reson.* **35,** 379 (1979).
9. B. M. Wood and R. F. Code, *Rev. Sci. Instrum.* **52,** 386 (1981).
10. J. Dadok in D. I. Hoult, *Prog. Nucl. Magn. Reson. Spectros.* **12,** 41 (1978).