A Computer-controlled, Fully Automatic NMR/NQR Double Resonance Spectrometer *

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A completely automatic computer-controlled NMR/NQR double resonance spectrometer is described. It features automatic tuning of the low, variable frequency power amplifier, thus permitting untended use over long periods, with high sensitivity and signal reproducibility. The sample is transferred between the low-frequency, zero-field region and the high-field region using compressed air and the possibility of switching on a field of several tens of gauss during the transfer of the sample is also included.

Introduction

One of us (F.Z.) has previously described [1] a relatively-easily constructed NMR/NQR double resonance spectrometer. In common with similar devices, in order to apply the low-frequency R.F. (NQR) field of sufficient amplitude a tuned power-amplifier is used. Such amplifiers must therefore be continually adjusted manually and the spectrometer cannot be left untended. In order to overcome this disability we have constructed a power amplifier whose tuning can be adjusted by a microcomputer according to a previously-determined calibration curve. At the same time we took the opportunity of controlling all the functions of the spectrometer, including data-acquisition, by means of the micro-computer. The sample-transfer system was also changed so as to use compressed air as the driving force.

Experimental

A block-diagram of the spectrometer is shown in Figure 1. Since many of the details are similar to those in the previously-described spectrometer, we limit ourselves here to commenting on those points which are specific to the present system.

The first version of this equipment was constructed around a Hewlett-Packard HP 86 micro-computer which was programmed using the built-in HP Basic interpreter. In order to achieve a more universal equipment, this version was later adapted to an IBM-compatible (Olivetti M24) personal computer; it is this version we report here. In both cases the computer was connected to the various components of the spectrometer through the industry-standard HP-IB IEEE 488 and Centronics parallel interfaces. The computer generates all the pulses necessary for measurement of the proton free-induction decay (FID), for actuating the valves of the compressed-air sample transfer device, for stepping the frequency and adjusting the pulse-width of the NQR signal generator while at the same time maintaining the tuning of the corresponding power amplifier. Should a transfer-field be required a Hewlett-Packard HP 6632A computer-controllable (HP-IB) power-supply which supplies current to a solenoid wrapped around the transfer tube is switched on just before the sample enters the transfer tube and off as soon as the sample is in place in the high-field or the zero-field regions.

The magnet was a 1.41 Tesla permanent magnet which originally formed part of a Perkin-Elmer R12

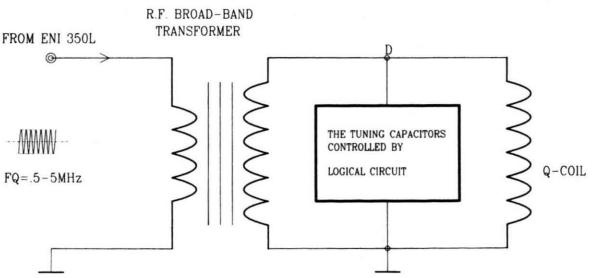

Fig. 2. LC matching network for coupling to Q-coil.

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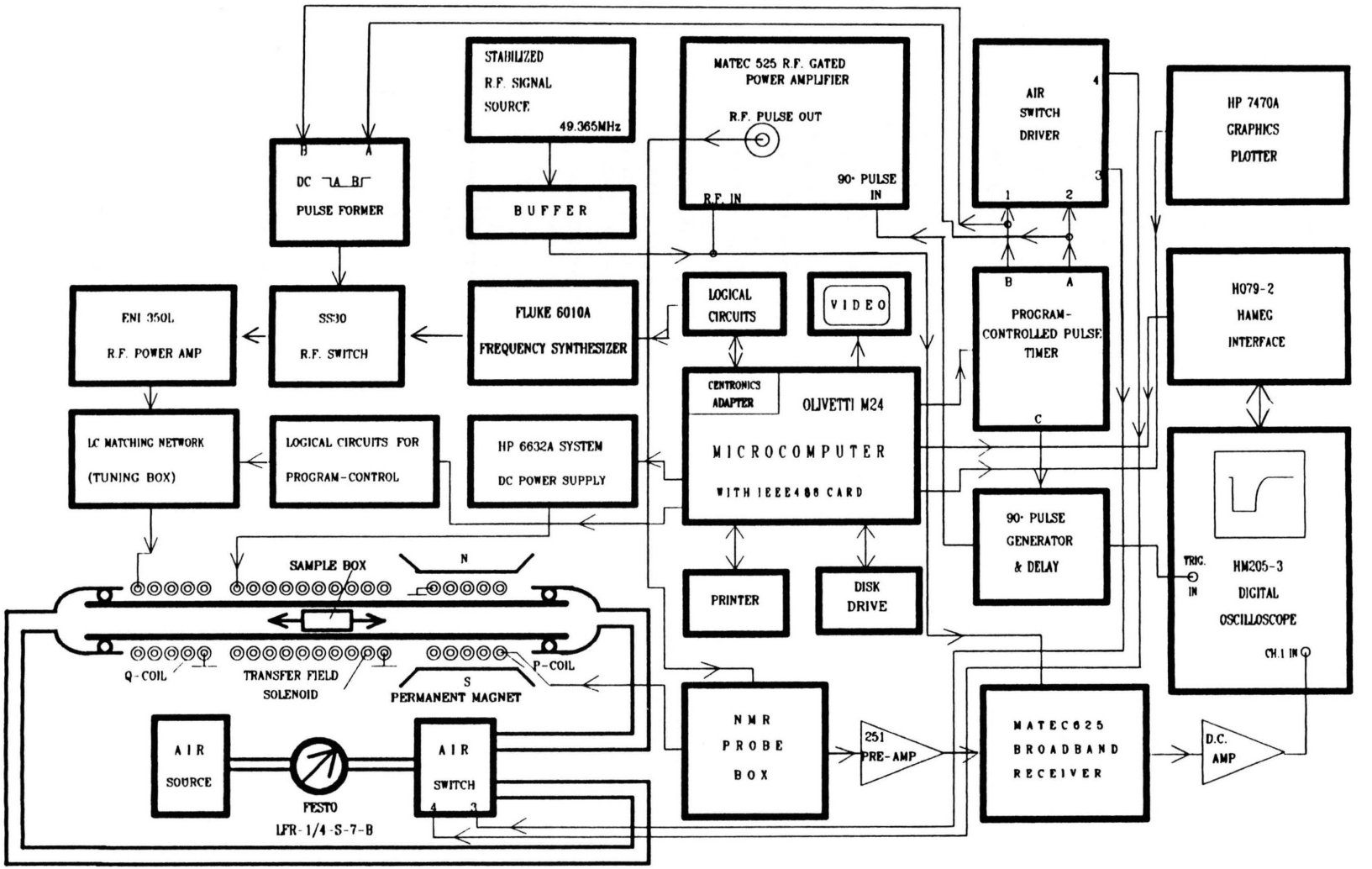

Fig. 1. Block diagram of the NMR/NQR double resonance spectrometer.

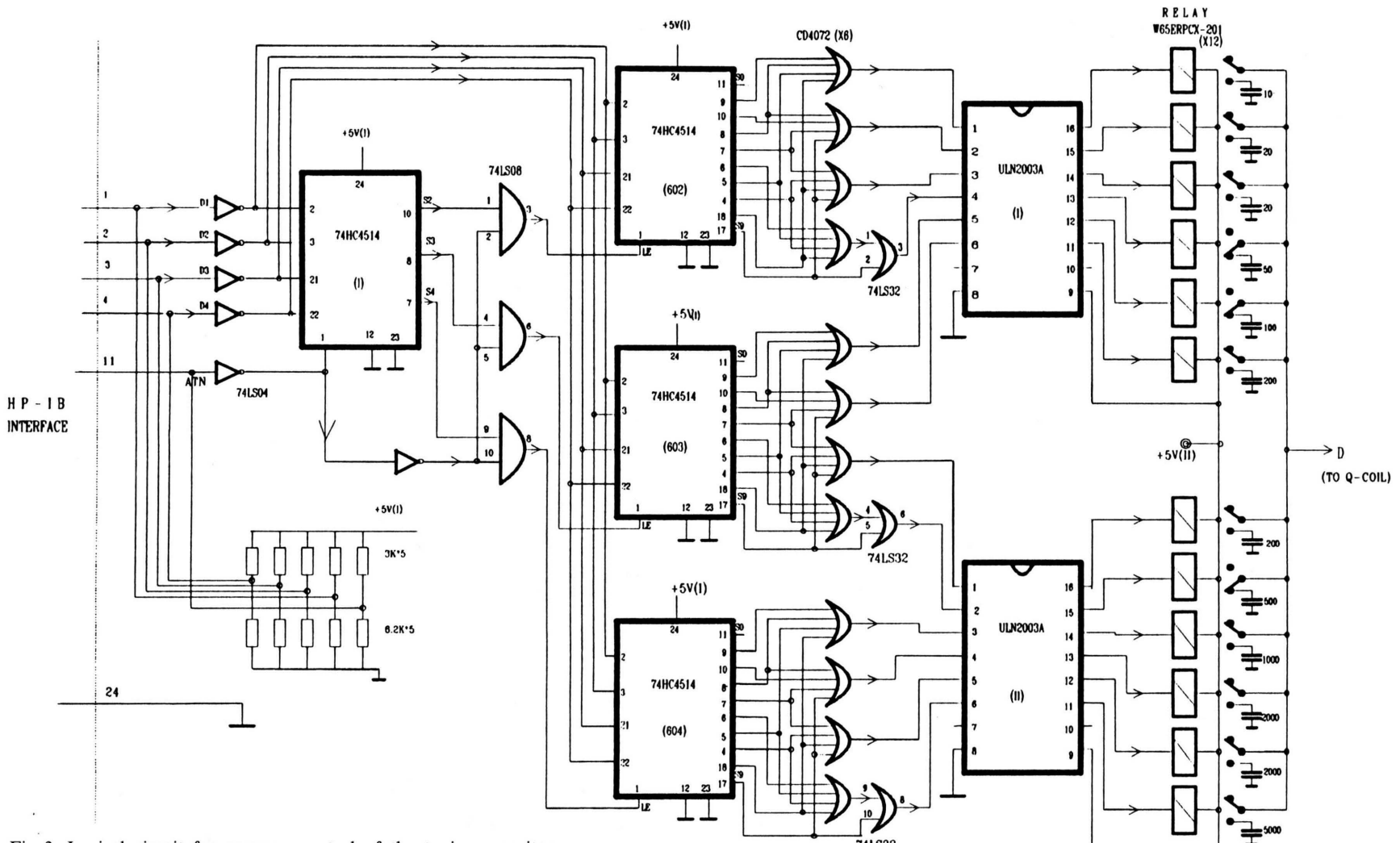

Fig. 3. Logical circuit for program-control of the tuning capacitor.

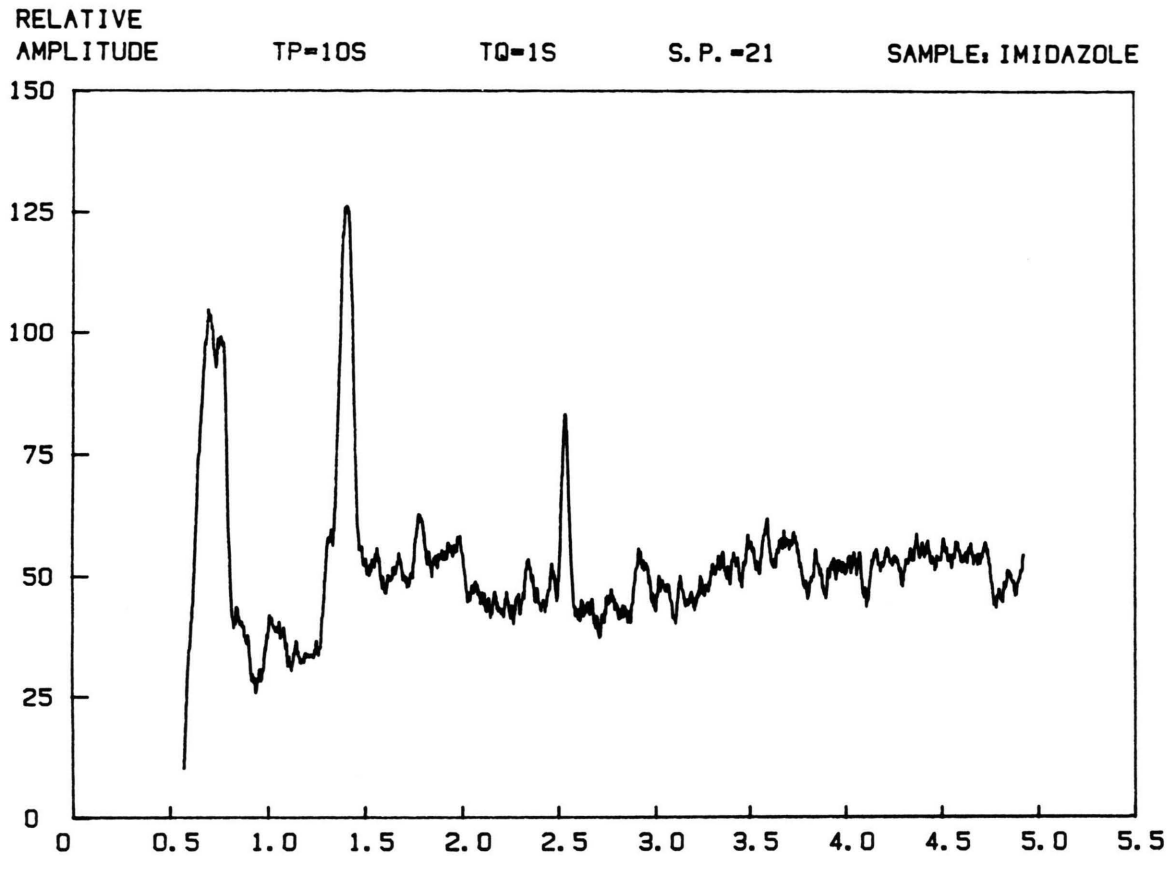

Fig. 4.

high-resolution 60 MHz NMR spectrometer. The original pole-gap of 15 mm was increased to 20 mm by inserting an annular soft-iron spacer between the two halves of the barrel magnet; the field then dropped to 1.17 Tesla corresponding to a proton NMR frequency of 49.9 MHz. The compact design of this magnet already provides an appreciable thermal stability with only a very small stray field. The thermal stability was further improved by wrapping the magnet with glass wool and enclosing the whole unit in a box made of sheets of expanded polystyrene of 5 cm thickness. No further stabilisation of the temperature of the magnet or of the magnetic field itself proved to be necessary.

The proton free-induction decay (FID) was observed using a MATEC 5100 pulse modulator and a MATEC 625 receiver, preceded by a MATEC 251 preamplifier, and finally displayed on a HAMEG HM205-3 digital oscilloscope. An appropriate portion of the FID was then transferred to the microcomputer via the HP-IB interface.

The most important modification of the previous spectrometer concerns the tuning of the power amplifier. The frequency source for this amplifier is a Fluke Model 6010A frequency-synthesizer, a model dating from 1972, whose functions can be controlled by a non-standard parallel interface for which we have adapted the parallel Centronics printer interface of the computer. The only reason for using this equipment was that it was to hand; had we been starting from scratch one of the frequency-synthesizers fitted with an HP-IB interface which are currently available would have been used instead. The output from the signal generator is fed to an R.F. switch which is opened and closed by pulses of appropriate width generated by the microcomputer and transferred by the HP-IB interface. The resulting R.F. pulses are fed to a wide-band amplifier (ENI Model 350L) to bring

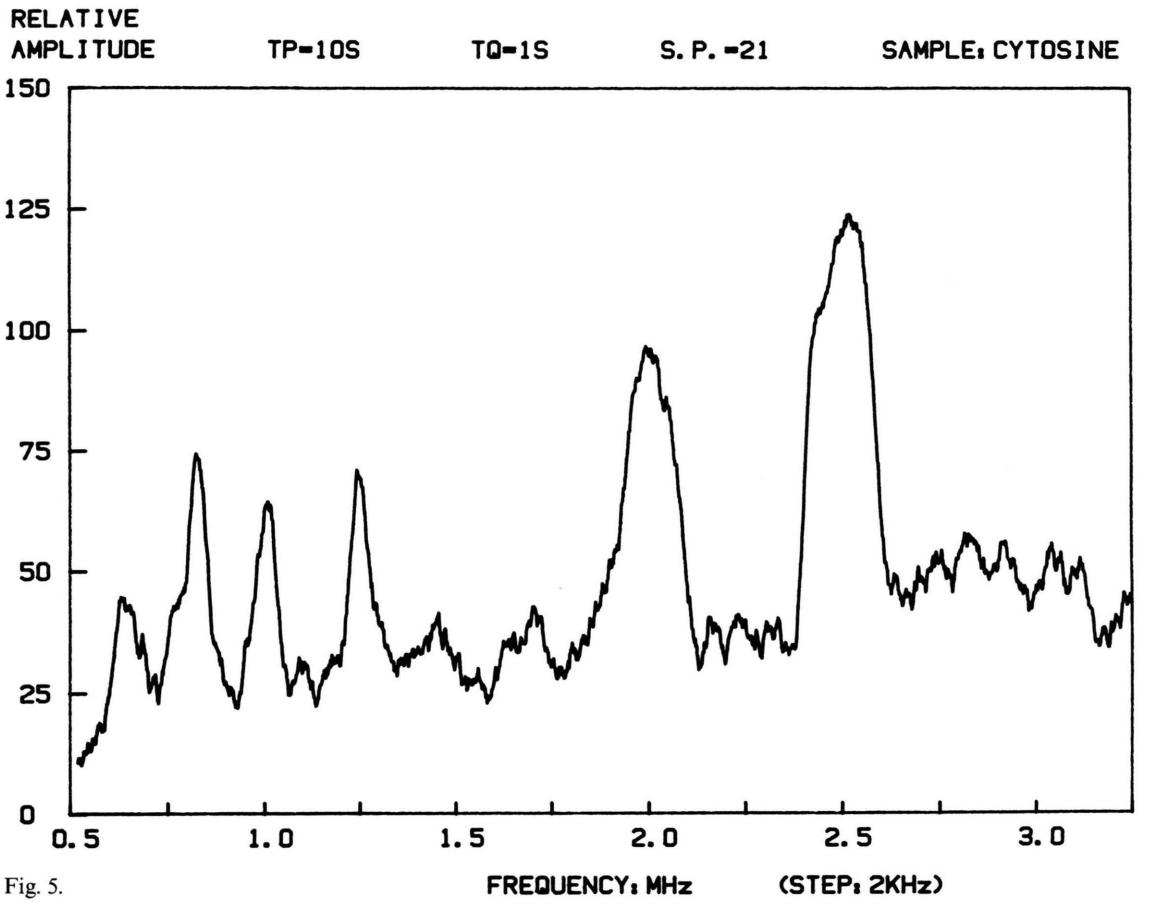

Fig. 5.

them to a suitable level to drive the final tuned circuit to which it is coupled by a broad-band matching transformer (Figure 2). In the previous version of this spectrometer tuning was achieved by switching in capacitors manually. This switching is now achieved when an appropriate signal is sent from the microcomputer causing one of the relays which now form the switch bank to open or close, thus adding or removing one of the high-voltage capacitors which are placed in parallel across the sample coil (Figure 3). The points at which switching of the capacitors occurs are determined by running a calibration curve and storing the appropriate frequency/capacity data in the microcomputer. Absolutely constant output is, of course, impossible to achieve since the total tuned circuit capacity is varied in a step-wise manner, but, as long as the R.F. field is sufficiently intense to thoroughly mix the populations of the various nuclear spin level, this will not affect the amplitude of the double-resonance signal.

Results

Figures 4, 5 and 6 show double resonance spectra for the ^{14}N quadrupole transitions in imidazole, cytosine, and in ethylenethiurea. They were obtained over a period of 8 hours during which there were absolutely no adjustments made to the spectrometer. The reproducibility of the details of the spectrum is much higher than in the previous version but, above all, the spectrometer can now be run unattended and even overnight, thus allowing us to concentrate on preparing samples and analysing the results. In a word being spectroscopists rather than spectrometer-watchers.

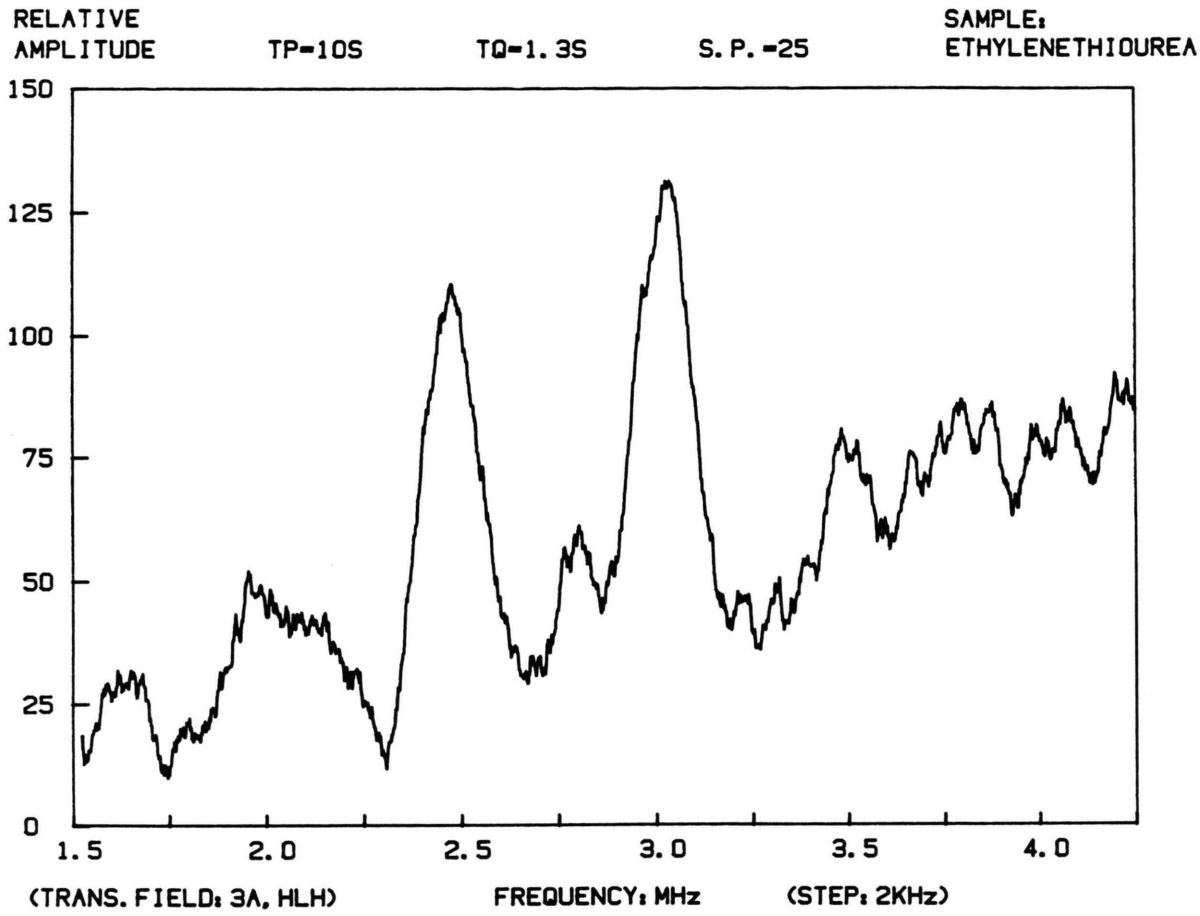

Fig. 6.

Acknowledgement

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[1] M. M. P. Khurshid, F. Zhenye, and J. A. S. Smith, Z. Naturforsch. **45a**, 595 (1990).