NQR Spectrometer with a Two Integrated Circuits Radio Frequency Head*

Yushi Zikumaru

Faculty of Education, Oita University, 700 Dannoharu Oita-shi, 870-11, Japan

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An NQR spectrometer has been constructed using two linear integrated circuits in its oscillator-detector. This is very simple and compact and works in range 3-65 MHz. The radio frequency voltage can be varied from 10 mV_{p-p} to 15 V_{p-p} by changing the supply-voltage of an integrated circuit μ A 733. The utility of the spectrometer is demonstrated by recording ³⁵Cl NQR spectra in p-C₆H₄Cl₂, NaClO₃, and KClO₃.

1. Introduction

Marginal oscillator-detector type NQR spectrometers with field effect transistors (FET) [1–8] have particular advantages. However, they also have certain disadvantages: the frequency range is narrow, the oscillation voltage varies with temperature, and the circuit is not only complicated but inconvenient in the adjustment and control of the oscillation-voltage. To remove these defects, a spectrometer was constructed using linear integrated circuits. Although the circuit is simple, the signal-to-noise ratio (S/N) of this spectrometer was found to be one hundred or more in a wide frequency range.

2. The Radio Frequency Circuit

The oscillation energy of the marginal oscillator, where an amplifier LH 0063 (from National Semiconductor) was used as buffer, must be decreased sufficiently to prevent saturation due to a slow relaxation of the samples. To get enough S/N, however, the oscillation energy has to be increased. Namely, oscillation-voltage, which is adjusted by changing the supply voltage of the limiter, must be set at optimum for the type of sample.

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Reprint requests to Prof. Y. Zikumaru, Faculty of Education, Oita University, 700 Dannoharu Oita-shi 870-11, Japan.

used. The buffer amplifier has a high input impedance $(10^{10} \, \Omega)$, high speed, FET input and voltage follower, and can be used from DC to 200 MHz. This buffer amplifier was connected to both a detector circuit for the NQR signal and a second differential amplifier μA 733 (fairchild) used as a voltage-limiter amplifier. When the LC tank circuit is connected directly to the input terminal of the LH 0063, its circuit oscillates because of the positive feedback. Therefore an infinitesimal condenser must be inserted between the tank circuit and the LH 0063.

The original function of the μA 733 was differential video amplification. In this spectrometer, however, it was used as a voltage-limiter amplifier. One output terminal was used for feedback and the other was connected to a frequency counter. As the oscillation voltage can be controlled externally with the selection of three possible input-gain terminals, the μA 733 is quite suitable for the NQR spectrometer.

In our spectrometer, the differential video amplifier is used as a limiter of the oscillation voltage. The output voltage dependence on input voltage is measured at several supply-voltages of the μA 733, as shown in Figure 1. The measurement is made with the amplifier μA 733 in position 3 at 35 MHz. Judging from the results the output voltage of the μA 733 reaches saturation. Therefore the oscillation voltage can be controlled by adjusting the supply voltage (+1 to +12 V).

3. The Spectrometer

Figure 2 shows the circuit diagram of the spectrometer using an LH 0063 and a μA 733. The sample coil

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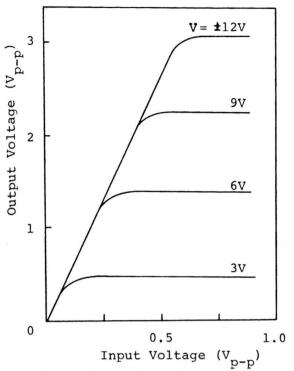


Fig. 1. Output voltage vs. input voltage for various supply-voltages of a differential video amplifier μA 733 at 35 MHz.

is connected to a variable condenser of 200 pF yielding an oscillation frequency in the range 15–45 MHz. By using coils of different numbers of turns, the spectrometer can be used in the range 3–65 MHz. The sensitivity of the spectrometer is tested by detecting ³⁵Cl NQR signals at 54, 30, and 8 MHz in (CH₂CO)₂NCl, p-C₆H₄Cl₂ and CsPbCl₃, respectively. The S/N ratios were one hundred or more at all frequencies. For the ³⁵Cl NQR measurements a modulation voltage of 0.1 V_{p-p} is applied to the diode 1S 1765 (NEC Co.).

The detection is made with a 1N60H (NEC) diode fed from the output of the LH 0063 through a 100 pF capacitor. According to the kind of diodes, the intensity of the output signal changes. Therefore a highest quality diode must be chosen. The above-mentioned circuits of the NQR spectrometer are shielded by copper plates surrounding each block, as shown in Fig. 2 with broken lines.

The detected output NQR signal is amplified and subjected to phase sensitive detection in the lock-in amplifier. The signal from the lock-in amplifier is introduced into the microcomputer through a GP-IB interface. The signal is calculated by the method explained in [9]. This method is used in order to increase S/N and the speed of measurement.

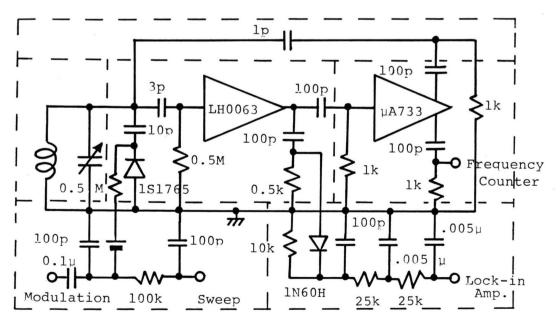


Fig. 2. Diagram of the oscillator made with two integrated circuits. Capacitance in F, resistances in Ω .

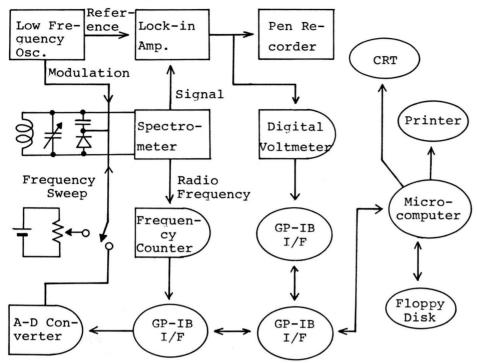


Fig. 3. Block diagram of the NQR measurement system. \square means analogue device, \square means digital device, \square means AD converter.

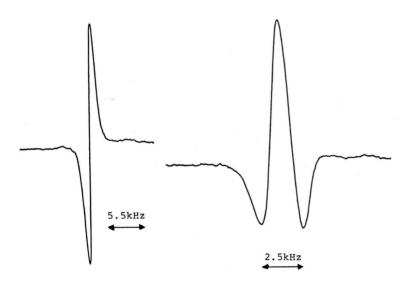


Fig. 4. Left: First derivative curve of the absorption line of ³⁵Cl in NaClO₃. Right: Second derivative curve of the absorption line of ³⁵Cl in p-C₆H₄Cl₂. Both curves are recorded on a pen recorder at room temperature. The time constant is 1 s.

Figure 3 shows a block diagram of the system for measuring NQR signals. The detected signal is converted digitally through an AD converter. When a microcomputer is not used, the frequency sweep can be caused by mechanical means, namely by variation of the DC voltage added to the diode 1S1765.

4. Operation and Performance

The first- and second-derivative absorption curves of ³⁵Cl NQR in p-C₆H₄Cl₂ and NaClO₃, recorded by the NQR spectrometer at room temperature, are shown in Figure 4. Judging from the measurements,

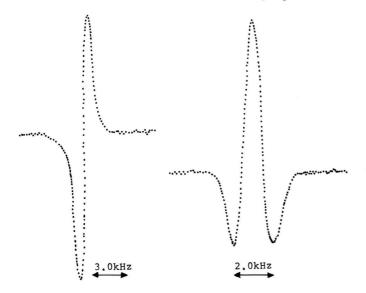


Fig. 5. Left: First derivative absorption line of ³⁵Cl in KClO₃. Right: Second derivative absorption line of ³⁵Cl in NaClO₃. Both curves are recorded on the online line-printer at room temperature. Time constant is 0.5 s.

the S/N ratios obtained for both cases are over one hundred. These are higher than those found by other authors (see [1-6]).

The first- and second-derivative absorption curves of ³⁵Cl NQR in NaClO₃ and KClO₃ at room temperature, measured by polynomial curve fitting [10] using an online microcomputer, are shown in Figure 5. The

S/N ratios are higher than those found by H. Niki et al. [8].

The temperature dependence of the 35 Cl NQR frequencies in p-C₆H₄Cl₂ at various temperatures from 77 to 326 K were measured for the α , β , and γ phases and the results agree completely with those reported by Moross and Story [11].

- T. L. Viswanathan, T. R. Viswanathan, and K. V. Sane, Rev. Sci. Instrum. 39, 472 (1968).
- [2] T. L. Viswanathan, T. R. Viswanathan, and K. V. Sane, Rev. Sci. Instrum. 41, 477 (1970).
- [3] N. Sullivan, Rev. Sci. Instrum. 42, 462 (1971).
- [4] R. A. Klein, Adv. Nucl. Quadrupole Reson. 7, 159 (1974).
- [5] J. Lee and S. H. Choh, Rev. Sci. Instrum. 53, 232 (1982).
- [6] F. N. H. Robinson, J. Phys. E, Sci. Instrum. 15, 814 (1982).
- [7] O. N. Bryukhanov and T. N. Rudakov, Instrum. & Exp. Tech. 25, 947 (1983).
- [8] H. Niki, M. Hashimoto, and K. Mano, J. Association of Personal Computer for Chemists 11, 3 (1989).
- [9] Y. Zikumaru and S. Yamashita, Research Bull. Fac. Educ. Oita Univ. 7, 7 (1985).
- [10] A. Savitzky and M. J. E. Golay, Anal. Chem. 36, 1627 (1964).
- [11] G. G. Moross and H. S. Story, J. Chem. Phys. 45, 3370 (1966).