

# NEW METHODS IN NUCLEAR QUADRUPOLE RESONANCE MEASUREMENT

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New methods in nuclear quadrupole resonance measurement on the basis of digital procedure were developed. It was proved that the system was stable and the repeated accumulation of signal led to an improvement in sensitivity. These methods have an advantage of application for rapid scanning.

**INTRODUCTION** In the conventional nuclear quadrupole resonance (NQR) spectrometer using a superregenerative oscillator, without distinction of either self-quench or external-quench, the radio frequency is always swept with a low frequency modulation. 1~3) After the suppression of the quench frequency components by low pass filter, only the modulated low frequency component is amplified and recorded. The recovery of interested signal out of background noises is effectively performed with the help of a Lock-in amplifier in spite of prolonged recording time due to unavoidable large time constant. These methods are consisted of essentially analog procedures. On the other hand, we thought why not the digital procedure could be applied as another way of noise rejection, ect.. Then, we have attempted to develop new methods on the basis of the digital procedure by time-averaging, different from those conventional Lock-in recording system and proved that these methods are applicable in practical use.

**RESULTS and DISCUSSION** The super-regenerative oscillator is obtained from periodic interruption of continuous oscillation, that is to say, from quenching. In spite of its unstable characters, it is commonly used in NQR measurement because of its high sensitivity. There are two kinds of superregenerative oscillator, namely, external-quenching and internal- or self-quenching. For the self-quenching, a change of quench frequency occurs with signal arrival, on the other hand a change of quench amplitude in external-quenching. 4)

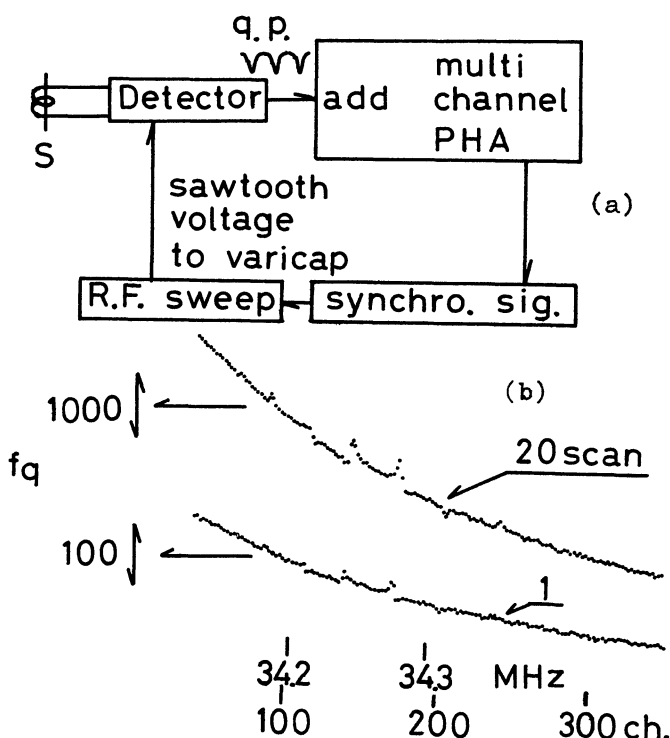


Fig.1 Simple Repetition Method

The primary step of our developments is shown in Fig.1(a). Detector is the self-quenching superregenerative oscillator, which is used throughout this study. As was mentioned, the quench frequency in self-quenching mode markedly changes with NQR signal. It is directly counted by and is stored in multi-channel analyzer(PHA) operated in multi-scaler mode. The radio frequency is swept linearly against the time by application of sawtooth voltage to the varicap placed in the tank circuit of detector. The radio frequency is synchronized to the channel address in PHA by the control circuit represented as synchro. sig.. The repeated sweeps permit an accumulation of signal in PHA. Fig.1(b) is the spectra of  $^{35}\text{Cl}$  in p-dichlorobenzene obtained in this way from 1 scan and 20 scans, respectively. The peak at  $34.27 \sim 34.28$  MHz is the main signal we are interested in and others are trivial sidebands due to the characteristics of detection.  $f_q$  means the quench frequency and it is noticed that the ordinate scale of 20 scans is ten times as large as the other. As is evident from Fig.1(b), the quench frequency slowly changes with the radio frequency sweep due to the circuit characteristics. Therefore, the simple repetition emphasizes an inclination of spectrum background and makes it difficult to discern a resonant peak unless the peak has high intensity. In order to overcome this difficulty, we have developed the following two methods. First one which we term the difference method is given in Fig.2(a). In this method, we make use of the fact that the resonant peaks apparently vanish and leave only the inclined background when a low frequency of an optional amplitude is superposed on the ordinal radio frequency sweep. The control circuit alternatively commands an additive and subtractive action of PHA and simultaneously turns the low frequency oscillator on and off. The oscillator is on during sub-period, but is off during add-period. Of course, the radio frequency is swept in the same way during both periods. By the repetition of the add and sub action, we obtain the resonant peaks on the roughly flat background and observe an explicit interaction of NQR signal with the superregenerative oscillator. That is, it is noticed that the levels

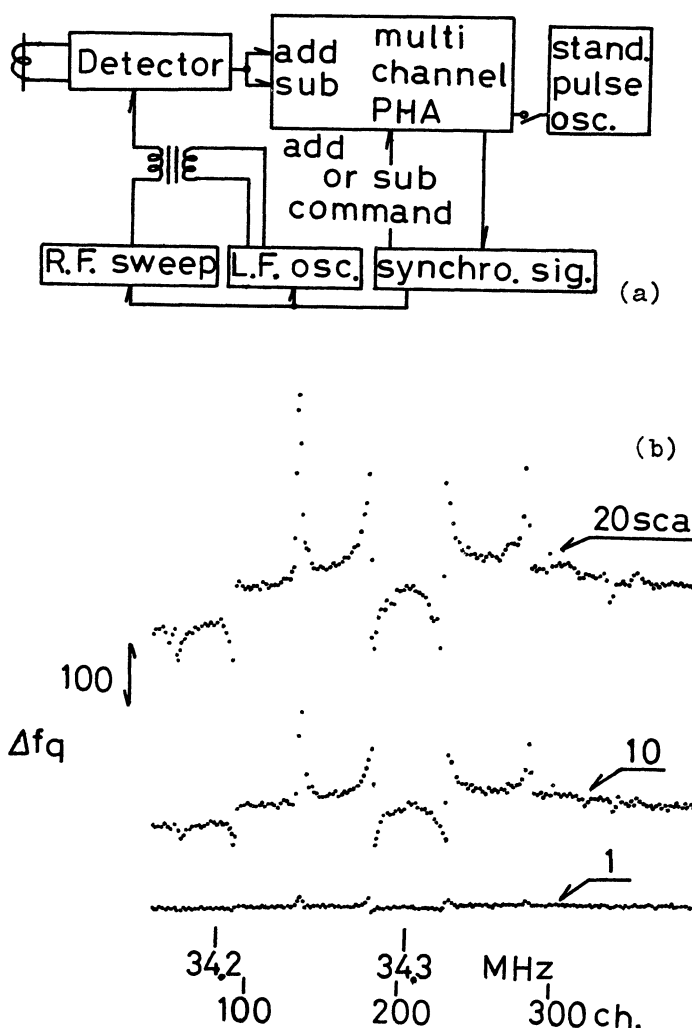


Fig.2 The Difference Method

on both sides of a peak shift to each other. By the way, in order to give PHA sufficient room for subtraction, an equal amount of input is previously stored into each channel from standard pulse oscillator. The spectra obtained in this way are shown in Fig.2(b).  $\Delta f_q$  means a variation of quench frequency. The ordinate scales are common to three cases, but the levels are shifted to each other. If a weak external magnetic field is on during sub-period instead of superposition of low frequency, it may be expected that the flatness of background is more complete. In this method we waste the half of total measuring time. In order to improve this point, we developed following modification shown in Fig.3(a). In this method, the radio frequency is swept in above-mentioned way for addition period, but for subtraction period the radio frequency sweep is performed with the slightly constant-difference ( $\Delta\omega$ ) from that of the add action period. As is evident from Fig.3(b) obtained in this way, it

is reasonable that we term this the derivative method.  $\Delta f_q$  corresponds to the derivative of quench frequency variation. The ordinate scales are common but the levels are shifted like in Fig.2(b). It is noticed that the outermost sidebands which are uncertain from 1 scan become evident in measurements of 10 and 20 scans. The flatness of background is better than the difference method. Table 1 gives comparison of the difference method with the derivative method and effects of repetition.  $\sigma$  is the standard deviation calculated from

$\Delta f_q$  on the channels which are not affected by resonant peaks and P stands for the height of main peak. And then, it is considered that  $P/\sigma$  corresponds to signal to noise ratio. It shows how the sensitivity is improved by the repetition of scanning in either difference or derivative method, and it is evident that the derivative method is more sensitive than the difference method. The center frequency of detector is selected by adjusting another variable capacitor. The measurement of radio frequency is easily performed

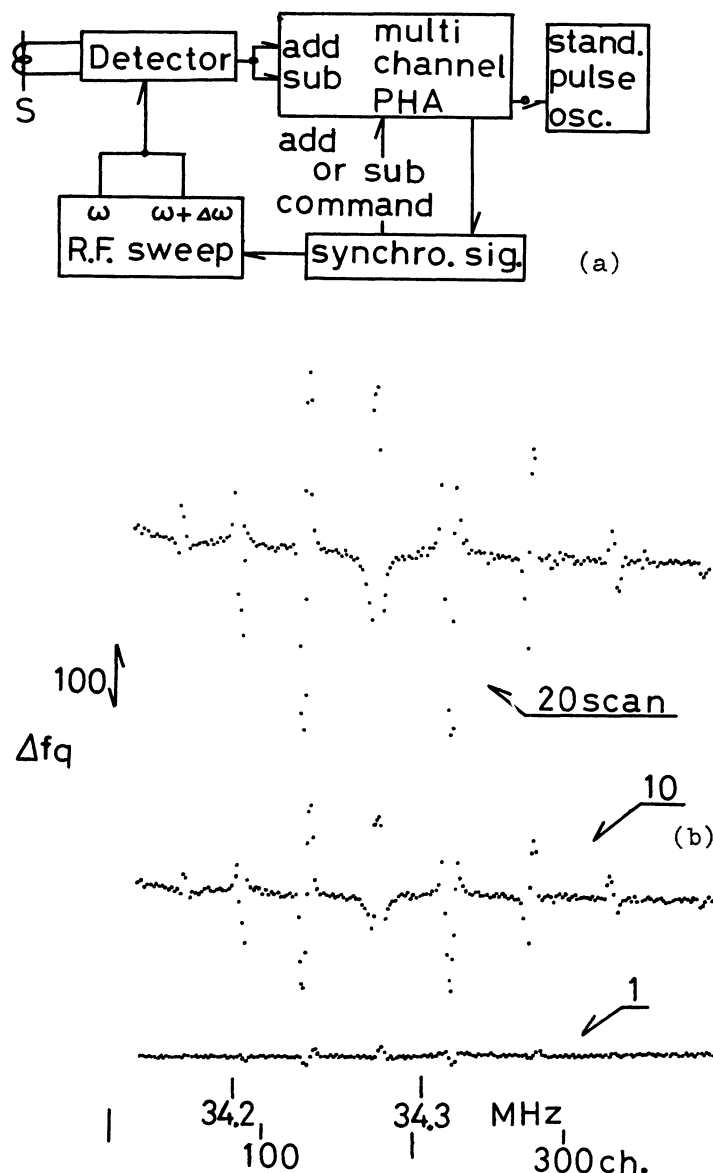


Fig.3 The Derivative Method

| Difference Method |          |             | Derivative Method |          |             |
|-------------------|----------|-------------|-------------------|----------|-------------|
| scan              | $\sigma$ | P/ $\sigma$ | scan              | $\sigma$ | P/ $\sigma$ |
| 1                 | 0.74     | 24          | 1                 | 0.91     | 23          |
| 10                | 3.07     | 52          | 10                | 2.65     | 70          |
| 20                | 3.95     | 73          | 20                | 2.96     | 130         |

Table1 Effects of Repetition for The Difference Method and The Derivative Method

$\sigma$  ; standard deviation on 360  $\sim$  389ch.

P ; height of main peak

with an interaction of the detector with the signal from standard high frequency generator for only one scanning. These results were obtained from the experiments which take 40 seconds per one scanning. The experiments of 4 seconds per one scan also give similar results. So it is worthwhile to suggest that these methods have an advantage of application for rapid scanning.

But at the present stage, an automatic gain control is not combined into this system, and it is difficult to spread the scanning range of radio frequency only by means of supplying a sweep voltage on varicap.

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