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# MAGNETIC FIELD FLUCTUATIONS AND RESONANCE CONDITION STABILIZATION IN NMDR SPECTROMETERS

Proton high-resolution nuclear magnetic double resonance (NMDR) spectrometers require the highest possible degree of stabilization of the ratio $\omega/H_0$. During the period of scanning of the whole spectrum the permissible long-term instability is only a small fraction of the line width. Moreover, even magnetic field fluctuations of a quite short duration must be avoided. In case of monoresonance only fluctuations within the bandwidth of the recorder are of significance. In case of double resonance the perturbing *rf* field $H_2$ of much greater amplitude also affects the spectral line to be observed. This is due to the noise sideband produced by field fluctuations near the frequency difference between the two *rf* fields. The direct influence of strong *rf* field $H_2$ on the line to be observed via noise sideband can give considerable output within the bandwidth of the recorder and seriously disturb the investigation of a given double resonance effect.

In this paper we present some results of research on the frequency response and noise characteristics of the magnet, flux stabilizer and spin stabilizers within the frequency range of 0.01 . . . 1000 Hz.

## 1. Magnetic field characteristics

Two main sources perturbing the magnetic field in the air gap of NMR magnets are external magnetic fields and exciting current noise (in case of electromagnets). We investigated their influence on the magnetic field by exposing the magnet to artificial perturbations of those kinds. The parameters to be measured were total field perturbation in the central region of the air gap and also perturbation difference between two points in the same central region, the latter being of main interest in case of external (i. e. separate probe) NMR field-frequency stabilization.

1.1. E x p e r i m e n t a l   a r r a n g e m e n t. The 40-MHz NMDR spectrometer [1] formed the basic structure of the instrumentation (some blocks of it have been described in [2]). The electromagnet was the JEOL JM-300 fed from the JEOL JVR-3 regulated power supply. For total field perturbation measurement a free-running 5-kHz sideband spin generator was used and sideband frequency excursions were measured with the *lf* deviation meter [3]. The measurement of differential field perturbation was accomplished by the use of two sideband spin generators operating from separate NMR probes. These probes were fed from two frequency synthesizers with a $\sim$ 3 kHz frequency difference, the sideband frequencies being 5 and 8 kHz. The difference of these sideband frequencies was formed and its excursions were measured by the same meter [3]. In order to minimize frequency pulling, the *lf* circuits must not introduce any significant phase shift, i. e. they must not contain any frequency-dependent elements, the modulation coils being located inside the probes and the modulation amplifiers having degenerative modulation current feedback. A further improvement was obtained by using phase synchronization between one of the sideband frequencies and an independent *lf* generator. The residual frequency pulling was negligible as compared to the effect to be measured — the differential perturbation.

The external field perturbations were obtained by the use of a *vlf* generator, a transistorized 50-watt d. c. amplifier and an air coil of 50 cm diameter and of 1250 amper-turns exciting capability. The coil was placed at a distance of 1.1 m from the air gap on the axis of the magnet. The perturbations due to exciting current excursions were obtained simply by injecting the output of a *vlf* generator via a 20-Mohm resistor to input of the current regulator.

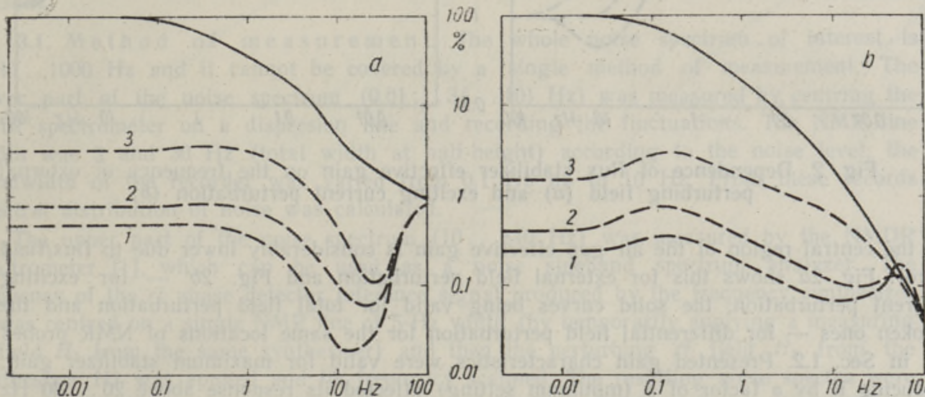


Fig. 1. Dependence of field perturbation on the frequency of external perturbing field (*a*) and exciting current perturbation (*b*).

**1.2.** Results. The results of the performed measurements are shown in Fig. 1. Fig. 1*a* represents frequency response of magnetic field perturbation for constant amplitude external field perturbation and Fig. 1*b* represents the same for exciting current perturbation caused through constant amplitude input to the current regulator. The solid curves represent total field perturbation in the central region of the air gap (the d. c. response taken for 100 per cent) and the broken curves — differential perturbation for various locations of the NMR probes. For Curve *1*, both probes were side by side along the direction of the magnetic field at a distance of 20 mm; for Curves *2* and *3*, one of the probes was in the centre of the air gap and the other moved from the centre to distances of 18 and 54 mm, respectively. The distances were measured from sample centre to sample centre. Sufficiently slow (below 0.1 Hz) external field perturbations had 3.5 times greater effect caused by the iron yoke of the magnet as compared to the hypothetical case of an ironless magnet.

## 2. Flux stabilizer

In principle the main limitation of a flux stabilizer is its sensitivity to total magnetic flux perturbations through its pick-up coils only and not to field perturbations at the gap centre. Such "false action" we shall call flux/field error and it constitutes the subject of investigation of the following section. The flux stabilizer used was the JEOL JNS-H (with a galvanometer and photovoltaic amplifier). Experimental arrangement was the same as described in Sec. 1.1.

**2.1.** Characteristics. The gain of the flux stabilizer for flux perturbation through the "back out" coils is shown in Fig. 2 by the dotted curve. It was measured by injecting a perturbing current from a *vlf* generator via a 50-kohm resistor into the "back out" coils and monitoring the current in the same coils. But for a field perturbation

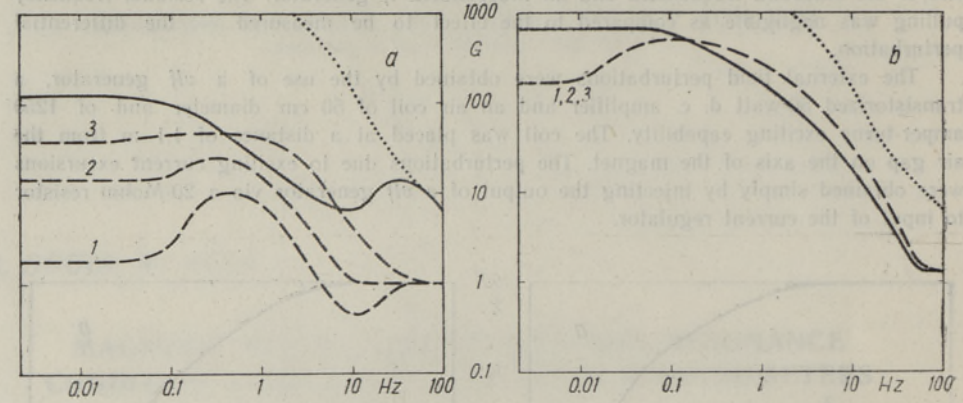


Fig. 2. Dependence of flux stabilizer effective gain on the frequency of external perturbing field (*a*) and exciting current perturbation (*b*).

in the central region of the air gap effective gain is considerably lower due to flux/field error. Fig. 2*a* shows this for external field perturbation and Fig. 2*b* — for exciting current perturbation, the solid curves being valid for total field perturbation and the broken ones — for differential field perturbation for the same locations of NMR probes as in Sec. 1.2. Presented gain characteristics were valid for maximum stabilizer gain; reducing it by a factor of 5 (minimum setting) affected its response above 20...30 Hz only, slightly reducing effective gain. $G<1$ means that the stabilizer increased perturbation (see Fig. 2*a*, Curve *1*).

For frequencies below 10 Hz the main feedback path of the flux stabilizer is closed through the current regulator to the exciting coils. When this circuit was broken, stabilizer gain for flux perturbation still exceeded 300 (at frequencies below 0.1 Hz), but for total field perturbation it was reduced from 125 (Fig. 2*a*) or from 640 (Fig. 2*b*) to 10...12. So we can conclude that this was due to increased flux/field error when the feedback path was closed only through the "back out" coils of the flux stabilizer. These coils create a magnetic field of increased inhomogeneity, yielding a flux/field error of $\sim$10% for both kinds of perturbing actions. In normal operation for external field perturbation a flux/field error of 0.8% is chiefly due to the exciting coils, whereas for exciting current perturbation its value is 0.16% and it is inherently due to the "back out" coils of the flux stabilizer.

2.2. N o i s e. The magnetic field noise spectra with and without flux stabilizer were measured as described later (Sec. 3.1.). In order to solve the alternative whether the residual field noise is due to noise of the flux stabilizer itself or it is of a different origin, the input signal from pick-up coils was divided by a factor of 5 (or 25) and the noise spectra were measured. From these measurements we concluded that the flux stabilizer has a margin of order of magnitude over its internal noise for frequencies above 0.03 Hz, while the long-term drift ($<$ 0.003 Hz) is due to the flux stabilizer itself. The galvanometer (with the stabilizer in operation) has a mechanical resonance at 10 Hz and a noise peak at this frequency occurs (see Fig. 3*a*) when external mechanical vibration is present.

From Fig. 2 (the solid curves) and Fig. 3*a* (the dotted curve) it should be clear that at frequencies above 1 Hz residual field noise is caused by external field fluctuations and/or by exciting current noise due to an insufficiently high effective gain of the flux stabilizer. A gradual decrease of the ratio of noise levels without/with the flux stabilizer for frequencies from 1 Hz to 0.01 Hz is not yet caused by the rise of flux stabilizer noise but is due to the magnet itself. The magnet acts as a source of noise which can be characterized by the fact that field noise at the air gap centre is to some extent (or even entirely) independent of flux noise, i. e. this noise is due to the redistribution of the field distribution diagram.

## 3. Spin stabilizers

A spin stabilizer has two main characteristics: noise performance and frequency response of stabilization factor. In case of external (separate probe) stabilizer, noise arises from thermal (Johnson s) noise of the input circuit and from magnetic field differential noise as well. It is true for fast-acting stabilizers; if the stabilizer is not fast enough or even with insufficient d. c. gain, some fraction of total field noise is also present.

**3.1.** Method of measurement. The whole noise spectrum of interest is 0.01 ... 1000 Hz and it cannot be covered by a single method of measurement. The lower part of the noise spectrum (0,01 ... 3(... 10) Hz) was measured by centring the NMR spectrometer on a dispersion line and recording the fluctuations. The NMR line width was 3 and 30 Hz (total width at half-height) according to the noise level; the bandwidth of the recorder was varied from 0.1 Hz up to 10 Hz. From these records spectral distribution of noise was calculated.

The upper part of the noise spectrum (10 ... 840 Hz) was measured by the NMDR spectrometer [1], which can be used as a noise sideband spectrum analyzer. The frequency of the *rf* phase detector reference signal produced by the frequency synthesizer II was centred on a single NMR line of 5-Hz width (by temporarily applying a measuring *rf* field $H_1$ from the same synthesizer) and then the perturbing *rf* field $H_2$ from the synthesizer III was applied and its frequency scanned. The resulting noise spectra were recorded at various scan ranges and strengths of $H_2$. Best discrimination against spurious base-line shifts and zero-beats was obtained by using centreband modulation technique at 3.5 kHz with the well-balanced *lf* phase detector [2].

Some relative information about the noise spectrum between 3 and 10 Hz was gained by the third method — by slow scanning through an absorption line of narrow natural width ($<$ 0.1 Hz) with a strong (3 Hz) *rf* field.

The "spectrum" probe was located in the air gap centre, the stabilizer probe being at a distance of 22 mm. Field inhomogeneity at the stabilizer probe location was reduced by a factor of 4 using an additional single "X" current shim. The sample for the stabilizer had a natural line width of 10 Hz, broadened by residual field inhomogeneity to 25 Hz.

**3.2.** Characteristics. Two kinds of spin stabilizers were used: the phase-synchronized sideband spin generator and the spin stabilizer with a "passive" NMR dispersion line and sideband modulation, both using only frequency regulation of the synthesizer I [1]. The first can be converted into the second by switching field modulation from the NMR sideband signal to the *lf* generator (so far used only for phase synchronization) and by introducing a 90° phase shift into the *rf* reference signal.

In the upper region their frequency response characteristics were found to be identical, but a difference occurs due to the integrating action of the phase-synchronizing phase detector for the lower region. The frequency deviation of the spin generator is integrated, yielding infinite d. c. gain of the spin generator quite irrespectively of the actual d. c. gain of the regulating circuit. For the "passive" stabilizer sufficient d. c. gain must be guaranteed; its response can be made deliberately slow by using a proper integrating circuit without affecting the regulation range. In case of the spin generator, its response can be made slow only by decreasing the regulation range; when simply integrating filters with decreased upper-frequency response are used, peaking of the response curve near the filter cutoff frequency results, and a further decrease leads to self-excitation.

The upper limit of frequency response (the same for both kinds of stabilizers) is restricted only by the necessity of a good filtering of the 5-kHz modulation frequency after phase detection and it can be expanded up to 500 Hz, if necessary. The upper-frequency gain must be held in proper limits, otherwise similar peaking and self-excitation result, but now on the frequency (~450 Hz), which is determined by the phase detector and frequency synthesizer time constants.

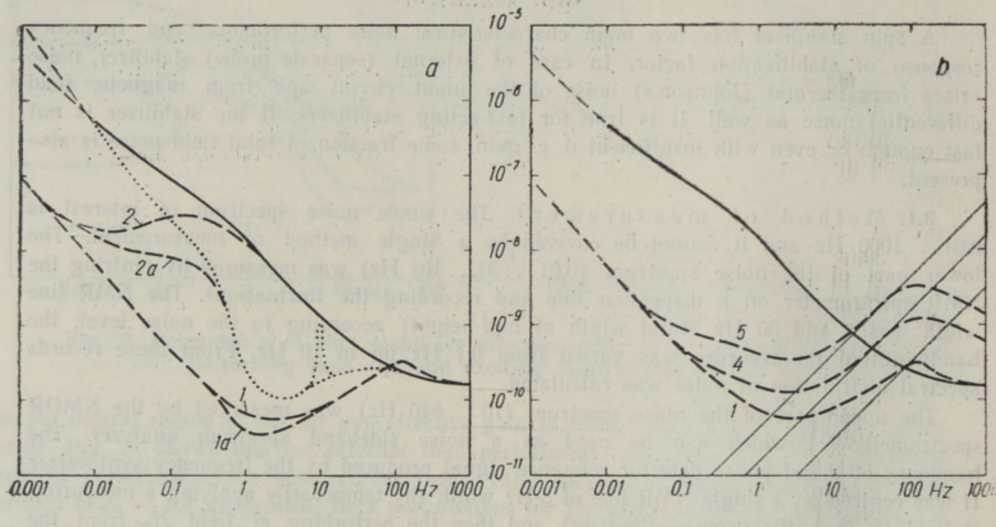


Fig. 3. Spectral density of noise (relative units per $\sqrt{\text{Hz}}$).

Solid curves, total field noise; dotted curves, total field noise with flux stabilizer; Curves *1*, residual noise with fast-acting spin stabilizer; *1a* — flux stabilizer added; *2* — slow-acting spin stabilizer; *2a* — flux stabilizer added; *4* and *5* — fast-acting spin stabilizer with gradually reduced *S/N*.

**3.3.** N o i s e. The noise spectra were measured for various frequency response characteristics of both kinds of the spin stabilizers with and without the flux stabilizer. The typical cases are presented in Fig. 3 and Fig. 4. The solid curves represent total field noise and Curves *1* — residual noise with the spin stabilizer, having optimum response and maximum signal-to-noise ratio *S/N* (~100 in 30 kHz noise bandwidth).

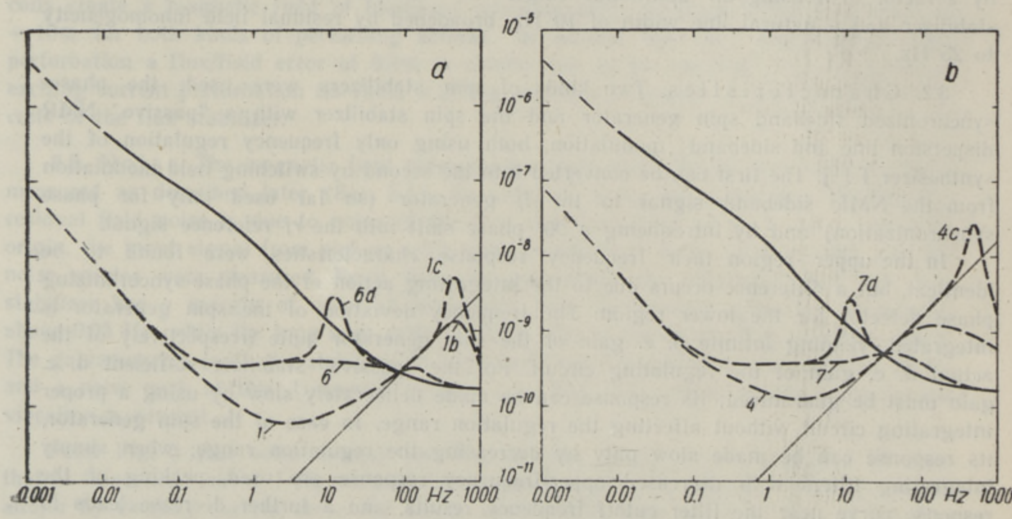


Fig. 4. Spectral density of noise (relative units per $\sqrt{\text{Hz}}$).

Solid curves, total field noise; Curve *1*, residual noise with spin stabilizer having optimum response; *1b* — faster response, non-peaking; *1c* — peaking at 450 Hz; *6* — slower response, non-peaking; *6d* — spin generator, peaking at 12 Hz; *4* — reduced *S/N*, fast but flat response; *4c* — peaking at 450 Hz; *7* — slower, non-peaking; *7d* — spin generator, peaking at 12 Hz.

Above 10 Hz the resulting noise spectrum is determined by the thermal noise of the stabilizer itself. For lower frequencies, differential field noise gives an increasing contribution. The influence of the flux stabilizer on the noise spectrum is represented by Curve *1a*; resulting decrease is a contribution from external field fluctuations and/or exciting current noise; for lower frequencies (below 0.1 Hz) we have concluded that main contribution is due to field redistribution noise, because the flux stabilizer is ineffective for such kind of noise.

The influence of the *S/N* of the stabilizer is illustrated in Fig. 3*b*, Curves *4* and *5* representing the case of decreased *S/N* of $\sim 25$ and $\sim 7$, respectively. For these cases, frequency response was the same as for Curve *1* and can equally be realized for both kinds of the stabilizers.

The case of a combination of a slow spin stabilizer with a flux stabilizer is of certain interest. It is illustrated by Curve *2* — only slow spin stabilizer operating and by Curve *2a* — the same plus flux stabilizer. As can be seen, this combination is unconditionally inferior in performance to the fast spin stabilizer of the same *S/N*. Curve *2a* is valid for frequency regulation; if the spin stabilizer is acting through the flux stabilizer, as is done in the majority of NMR spectrometers, the performance is even worse due to peaking at spin stabilizer cutoff frequency, unless this cutoff is very low ($< 0.01$ Hz).

For maximum *S/N* (Curve *1*) optimum response was obtained by attenuating upper frequencies to some extent. Noise spectrum for maximally fast response without peaking is presented by Curve *1b* (Fig. 4*a*) and it is worse at the given level of total field noise; but it can be better than in the conditions of Curve *1* for increased upper-frequency total field noise. Curve *1c* represents fast response with peaking and it is inferior in any case, because of amplification of thermal noise. Using medium-speed response (Curve *6*) gives rise to uncancelled medium-frequency total field noise. The noise spectrum for lower-peaking response (Curve *6d*) is typical for the slower-acting spin generator but amplification of total field noise must be considered.

For reduced *S/N* (of $\sim 25$, Curve *4*) some compromise can be obtained between noise performance at higher and medium frequencies depending on the conditions. Such a spectrum is represented by Curve *7*, valid for medium-speed response. Curves *4c* and *7d* show the influence of peaking as described before (Curves *1c* and *6d*).

An experiment was made to establish an eventual improvement at the lowest-frequency end of the residual noise spectrum in case of combined regulation. This regulation includes exciting current regulation for lower frequencies while higher frequencies are channelled through the frequency regulation circuit. No improvement was noticed, in accordance with the values of the ratio of total and differential perturbation ($> 100$, Fig. 1) and of the ratio of total and residual noise ($< 100$, Fig. 3).

## 4. Conclusions

1. Total magnetic field perturbation at frequencies above 50 Hz has quite different phases at the locations of either probes, thus differential perturbation is comparable with total one.

2. Effective gain of the flux stabilizer for differential field perturbation in case of external perturbing field is rather low and it shows strong dependence on the locations of the probes.

3. In case of exciting current perturbation this gain is rather high and almost the same as for total field perturbation; it does not depend on the locations of the probes to any great extent.

4. The noise of the flux stabilizer does not contribute to total field noise at frequencies above 0.03 Hz.

5. The fast-acting spin stabilizer gives the lowest residual noise spectrum; the phase-synchronized spin generator and the "passive" spin stabilizer give equal noise

performance. The combination of the slow-acting "passive" spin stabilizer with the flux stabilizer is remarkably worse.

6. The signal-to-noise ratio and the shape of frequency response are important parameters of noise spectra. Any peaking of frequency response is harmful to noise performance.

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### MAGNETVÄLJA FLUKTUATSIOONID JA RESONANTSTINGIMUSE STABILISEERIMINE TMTR-SPEKTROMEETRITES

Esitatakse uurimistulemusi TMR-magneti, superstabilisaatori ja spinstabilisaatorite alalt.

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### ФЛУКТУАЦИИ МАГНИТНОГО ПОЛЯ И СТАБИЛИЗАЦИЯ РЕЗОНАНСНОГО УСЛОВИЯ В СПЕКТРОМЕТРАХ ЯМДР

Обоснованы требования к стабилизации магнитного поля в спектрометрах ядерного магнитного двойного резонанса. Исследованы амплитудно-частотные зависимости магнитных помех в зазоре магнита от внешних магнитных помех и от помех в токе возбуждения (рис. 1), а также влияние суперстабилизатора на эти помехи (рис. 2). Установлено, что собственный шум суперстабилизатора не имеет практического значения из-за значительной ошибки, обусловленной различным поведением полного потока и напряженности поля в центре магнита. Измерены спектры шума магнитного поля без стабилизаторов, с суперстабилизатором и со спиновыми стабилизаторами (рис. 3 и 4). Обсуждается связь между амплитудно-частотными характеристиками спиновых стабилизаторов и спектрами остаточного шума.