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CONCEPTS IN NUCLEAR SPIN DYNAMICS OF LIQUIDS. 5

(Presented by E. Lippmaa)

Establishment of the master equation is «a necessary but not sufficient condition» to understand phenomena. The understanding holds only to the extent one knows the solutions of this equation, its dependence upon external magnetic field and upon characteristics of spin systems. It is the aim of this and of the following papers to consider outlines of these relationships. In the first place, we shall concern ourselves with the problem of how the motions and the observed signal patterns of a typical d-level spin system depend upon the character of time-dependence of the external magnetic field.

In this paper we consider experiments carried out in a magnetic field that retains its direction. In case the strength of this field is also time-independent we have the relaxation process (Secs 3.7; 3.8) or the free induction decay (FID) — the basis of the FID FT NMR Spectroscopy (Sec. 5.1). Time-dependence of the strenght of the external magnetic field induces nonresonant phenomena discussed in Secs 5.2 and 5.3 of this paper. For preceding papers of this series see [1] and references therein.

5. Nonresonant Phenomena

5.1. Free Induction Decay FT NMR Spectra. One starts at $t=0$ from the thermal equilibrium state P_0, and applies a resonant alternating magnetic field during a short preparation period $0 \leqslant t \leqslant t_1$ in order to get a nonequilibrium initial state $P(t_1)$ for the following free induction decay (FID) period $t \geqslant t_1$. The Fourier transform (FT) of NMR signals accumulated during this latter period gives the FID FT NMR spectrum of the substance under study. In this paper we consider $P(t_1)$ as given and discuss the FID period only. A treatment of the preparation period will be possible after a detailed study of several kinds of resonant excitation processes.

During the FID period the spin system is affected by the strong static magnetic field $\vec{b}_0 = b_0 \vec{e}_z$ only. Therefore, the Hamiltonian H_0 of a typical molecular nuclear spin system of an isotropic liquid is time-independent and symmetric relative to the z-axis —

$$[H_0, I_z] = 0. \tag{5.1}$$

For the thermal equilibrium state of a homonuclear d-level system we get from Eqs (4.46), (5.1)

$$P_0 = I_0 - (\hbar \omega_L / dkT) I_z, \tag{5.2}$$

where $\omega_L = \gamma b_0$.

According to Eq. (4.47) the Superhamiltonian $\mathcal{H}_0$ and the superoperator of relaxation $\mathcal{R}_0$ are, now, also time-independent and symmetric relative to the z-axis

$$[\mathcal{H}_0, \mathcal{I}_z] = [\mathcal{R}_0, \mathcal{I}_z] = 0. \tag{5.3}$$

Moreover, accepting Eq. (4.16), we have

$$[\mathcal{H}_0, \mathcal{R}_0] = 0. \tag{5.4}$$

As already explained in Sec. 3.8, Eq. (5.4) allows us to represent the induced propagator of the FID period in exponential form

$$\mathcal{L}^0(t, t_1) = \exp(t_2(\mathcal{H}_0^0 + \mathcal{R}_0^0)), \tag{5.5}$$

where $t_2 = t - t_1$.

In terms of the time-independent R—A basis

$$(\mathcal{H}_0^0 + \mathcal{R}_0^0) R_j = -(1/\tau_j) R_j, \tag{5.6}$$

$$(\mathcal{H}_0^0 + \mathcal{R}_0^0) A_{mn} = -(1/\tau_{mn} + i\omega_{mn}^0) A_{mn}; \tag{5.7}$$

the supermatrix Eq. (5.5) is diagonal

$$\mathcal{L}^0(t, t_1) R_j = \exp(-t_2/\tau_j) R_j, \tag{5.8}$$

$$\mathcal{L}^0(t, t_1) A_{mn} = \exp(-(1/\tau_{mn} + i\omega_{mn}^0) t_2) A_{mn}. \tag{5.9}$$

Therefore, if one expands the deviation operator in terms of the R—A basis

$$\Delta(t_2) = P(t_2) - P_0 = \sum_j \delta_j(t_2) R_j + \sum_{m \neq n} \sum \pi_{mn}(t_2) A_{mn} \tag{5.10}$$

and calculates the transformation Eq. (3.37) one gets, for the components of the deviation operator, an exponential decay law —

$$\delta_j(t_2) = \delta_j(t_1) \exp(-t_2/\tau_j), \tag{5.11}$$

$$\pi_{mn}(t_2) = \pi_{mn}(t_1) \exp(-(1/\tau_{mn} + i\omega_{mn}^0) t_2). \tag{5.12}$$

Of course, the decay of nonequilibrium populations $\pi_{mm}(t_2)$ of energy levels ω_m^0 is described by a superposition of exponential decays Eq. (5.11)

$$\pi_{mm}(t_2) - \pi_{mm}^0 = \sum_j (R_j, A_{mm}) \delta_j(t_2). \tag{5.13}$$

Thus, Eqs (5.11) and (5.13) represent the solutions of the Pauli equation (3.70).

Due to the axial symmetry Eq. (5.1) of the static Hamiltonian H_0, the operator of longitudinal magnetization $M_z = \gamma \hbar I_z$ is spanned by the basis operators R_j, $(j = 1, 2, \ldots, d-1)$ only, whereas the operator of transversal magnetization $M_x = \gamma \hbar I_x$ is spanned by such A_{mn} which satisfy the selection rules

$$\mu_{mn} = \pm 1. \tag{5.14}$$

Now, the longitudinal relaxation is given by

$$\langle M_z(t_2) \rangle - \langle M_z^0 \rangle = \sum_j (R_j, M_z) \delta_j(t_1) \exp(-t_2/\tau_j), \tag{5.15}$$

where

$$\langle M_z^0 \rangle = (P_0, M_z) = -(\hbar\omega_L/dkT)(M_z, I_z) \tag{5.16}$$

denotes the equilibrium nuclear magnetization.

During the FID period the transversal magnetization decays as follows —

$$\langle M_x(t_2)\rangle = \sum_m \sum_n (A_{mn}, M_x)\pi_{mn}(t_1) \exp\left(-(1/\tau_{mn}+i\omega^0_{mn})t_2\right). \quad (5.17)$$

In order to calculate the Fourier transform of this decay function let us, first, represent Eq. (5.17) in terms of real numbers

$$\langle M_x(t_2)\rangle = \sum_{m>n}\sum \exp(-t_2/\tau_{mn})\,(a_{mn}(t_1)\cos\omega^0_{mn}t_2 +$$
$$+b_{mn}(t_1)\sin\omega^0_{mn}t_2). \quad (5.18)$$

In Eq. (5.18) the real numbers $a_{mn}(t_1)$ and $b_{mn}(t_1)$ are defined by

$$2(A_{mn}, M_x)\pi_{mn}(t_1) = a_{mn}(t_1)+ib_{mn}(t_1). \quad (5.19)$$

The numeration of energy levels has been chosen such that for $\omega^0_{mn}>0$ we have $m>n$ and the allowed transitions are given by $\mu_{mn}=-1$.

Now, the Fourier transform

$$M_x(\omega) = (2/\pi)\int_0^\infty \langle M_x(t_2)\rangle \cos' \omega t_2\, dt_2 \quad (5.20)$$

of the signal Eq. (5.18) is given by

$$M_x(\omega) = \sum_{m>n}\sum (\tau_{mn}/\pi)\,\frac{a_{mn}(t_1)+b_{mn}(t_1)\Delta\omega_{mn}\tau_{mn}}{1+(\Delta\omega_{mn}\tau_{mn})^2}, \quad (5.21)$$

where

$$\Delta\omega_{mn}=\omega^0_{mn}-\omega, \quad (5.22)$$

provided

$$1/\tau_{mn}\ll\omega^0_{mn}. \quad (5.23)$$

If the initial state $P(t_1)$ is generated by an «ideal» pulse, such that

$$P(t_1)=I_0+(\hbar\omega_L/dkT)I_x, \quad (5.24)$$

we call the spectrum an ideal FID FT NMR spectrum.

In case of an ideal spectrum

$$\langle M_x(t_2)\rangle = -\langle M^0_z\rangle/(I_z, I_z)\sum_m |(I_x; A_{mn})|^2 \exp(-t_2/\tau_{mn})\cos\omega^0_{mn}t_2, \quad (5.25)$$

and so the FT of this signal is given by

$$M_x(\omega) = -\langle M^0_z\rangle/(I_z, I_z)\sum_{m>n}\sum |(I_x, A_{mn})|^2\frac{(\tau_{mn}/\pi)}{1+(\Delta\omega_{mn}\tau_{mn})^2}. \quad (5.26)$$

Notice that in case of an ideal FID FT NMR spectrum Eq. (5.26) the line positions ω^0_{mn}, the relative line intensities $|(I_x, A_{mn})|^2$ and the «absorption like» Lorentzian line shapes coincide with those of a slow sweep absorption mode single resonance NMR spectrum calculated in the linear response approximation [2, 3].

Although the idea of FT NMR Spectroscopy has been proposed very early [4], it becomes a major tool of NMR in the seventies. The main argument for its use is in the increased sensitivity of this method [5]. Taking this into account, we consider the ideal FID FT NMR spectrum as basic for the substance under study. We shall discuss the problem of how this spectrum can be transformed by changing the conditions of the experiment.

Comparison of Eqs (5.21). (5.26) shows that the transformation of an ideal spectrum caused by modifications of the preparation period of a FID experiment are restricted to changes of line intensities and line shapes only. In order to change the number and positions of spectral lines, an additional time-dependent magnetic field must be applied during the decay period. The frequency-dependence of the effect of this field can show resonant as well as nonresonant character.

5.2. Zeeman modulation. Let us now modify the conditions of the FID experiment so that the decay period occurs in the presence of a time-dependent magnetic field which, yet, retains their direction

$$\vec{b}(t_2) = b(t_2)\vec{e_z}, \tag{5.27}$$

$$b(t_2) = b_0 + b_M(t_2). \tag{5.28}$$

Due to this specific feature of the alternating magnetic field the Hamiltonian of the decay period

$$H(t_2) = H_0 + H_M(t_2). \tag{5.29}$$

$$H_M(t_2) = -\omega_M(t_2)I_z; \tag{5.30}$$

$$\omega_M(t_2) = \gamma b_M(t_2), \tag{5.31}$$

does not change its unitary orientation. Thus

$$[H_0, H_M(t_2)] = 0. \tag{5.32}$$

We call an excitation $H_M(t_2)$ which possess the property Eq. (5.32) a modulation. Especially, the excitation Eq. (5.30) is a Zeeman modulation.

In case of a modulation decay experiment, the induced superoperator of relaxation $\mathcal{R}^0$ is time-independent and coincides with the superoperator $\mathcal{R}^0_0$ introduced in Sec. 5.1. According to Eq. (4.16) the Superhamiltonian $\mathcal{K}(t_2)$ commutes with $\mathcal{R}^0_0$. As a consequence, we get an exponential induced propagator

$$\mathcal{L}^0(t, t_1) = \exp \int_0^{t_2} (\mathcal{K}(t'_2) + \mathcal{R}^0_0) \, dt'_2. \tag{5.33}$$

Due to Eq. (5.32) the R—A basis defined in Sec. 5.1 is at the same time the R—A basis of the modulation decay experiment. The time-dependence of the Superhamiltonian $\mathcal{K}(t_2)$ originates from time-dependent eigenvalues $\omega_m(t_2)$ of $H(t_2)$ only. We have

$$\omega_m(t_2) = \omega_m^0 - \mu_m\omega_M(t_2), \tag{5.34}$$

$$\omega_{mn}(t_2) = \omega_{mn}^0 - \mu_{mn}\omega_M(t_2). \tag{5.35}$$

Knowledge of the induced propagator Eq. (5.33) enables one to predict the time-dependence of the deviation operator

$$\Delta(t_2) = P(t_2) - P_s(t_2), \tag{5.36}$$

provided the latter is expanded in terms of the R—A basis as shown by Eq. (5.10). In doing so, we obtain for the component $\delta_j(t_2)$ again Eq. (5.11), but now

$$\pi_{mn}(t_2) = \pi_{mn}(t_1)\exp(-t_2/\tau_{mn})\exp\left(-i\int_0^{t_2} \omega_{mn}(t'_2)\,dt'_2\right), \tag{5.37}$$

where the time-dependent transition frequencies are given by Eq. (5.35). Notice that in Eq. (5.37)

$$\int_0^{t_2} \omega_{mn}(t'_2)\,dt'_2 = \omega^0_{mn}\,t_2 - \mu_{mn}\int_0^{t_2}\omega_M(t'_2)\,dt'_2. \tag{5.38}$$

Therefore, the Zeeman modulation causes a similar modulation of all such ω^0_{mn} which refer to the same value of μ_{mn}.

Knowledge of the deviation operator enables one to calculate the time-development of the transversal nuclear magnetization

$$\langle M_x(t_2)\rangle = \sum_m \sum_n (A_{mn}, M_x)\,\pi_{mn}(t_1) \times$$

$$\times \exp\left(-(1/\tau_{mn} + i\omega^0_{mn})t_2\right)\exp\left(i\mu_{mn}\int_0^{t_2}\omega_M(t'_2)\,dt'_2\right). \tag{5.39}$$

Of course, only terms which satisfy the selection rules Eq. (5.14) appear in Eq. (5.39). Consequently, all lines of the FID spectrum will be similarly affected by the frequency modulation Eq. (5.38).

Knowledge of the deviation operator Eq. (5.36) enables also to calculate the time-development of $\langle M_z(t_2)\rangle$ toward the value $\langle M_z^s(t_2)\rangle$ it takes in case of the reference trajectory $P_s(t_2)$ —

$$\langle M_z(t_2)\rangle - \langle M_z^s(t_2)\rangle = \sum_j (R_j, M_z)\,\delta_j(t_1)\exp(-t_2/\tau_j). \tag{5.40}$$

In order to found a suitable reference trajectory, let us first establish the quasi-static process $P_0(t_2)$ in case of Zeeman modulation. Replacing Eqs (5.29), (5.30) into Eqs (4.4), (4.5), we have in a good approximation

$$P_0(t_2) = P_0 + \bar{M}_0(t_2) = P_0 + (\hbar\omega_M(t_2)/dkT)\,I_z = P_0 + \sum_j \tilde{\pi}^0_j(t_2)\,R_j, \tag{5.41}$$

where

$$\tilde{\pi}^0_j(t_2) = (I_z; R_j)\,(\hbar\omega_M(t_2)/dkT). \tag{5.42}$$

Note that due to Eq. (4.13)

$$\mathcal{R}(t_2)P_0 = -\mathcal{R}^0_0\,\bar{M}_0(t_2). \tag{5.43}$$

In principle, any solution of the master equation (4.1) could serve for reference. However, we regard a solution $P_s(t_2)$ of Eq. (4.1) as suitable for reference in modulation decay experiment if it satisfies the condition

$$[H(t_2), P_s(t_2)] = 0. \tag{5.44}$$

Due to Eq. (5.32) such trajectories exist always. In case Eq. (4.1) possesses a steady state solution, we choose this for reference.

Representing, now, the reference trajectory as follows —

$$P_s(t_2) = P_0 + \bar{M}_s(t_2) = P_0 + \sum_j \tilde{\pi}^s_j(t_2)\,R_j, \tag{5.45}$$

and taking into account Eq. (5.43), we obtain from Eq. (4.1) the following inhomogeneous master equation for $\bar{M}_s(t_2)$ —

$$\frac{d\bar{M}_s}{dt_2} = \mathcal{R}^0_0\,(\bar{M}_s(t_2) - \bar{M}_0(t_2)). \tag{5.46}$$

If both $\tilde{M}_0(t_2)$ and $\tilde{M}_s(t_2)$ are decomposed, as shown by Eq. (5.41), (5.45) the master equation (5.46) splits into a system of $d-1$ independent equations

$$\frac{d\tilde{\pi}_j^s}{dt_2} = -(1/\tau_j)\,(\tilde{\pi}_j^s\,(t_2) - \tilde{\pi}_j^0\,(t_2)). \tag{5.47}$$

The general solution of Eq. (5.47) is given by

$$\tilde{\pi}_j^s\,(t_2) = \exp\,(-t_2/\tau_j)\left\{\tilde{\pi}_j^s\,(t_1) + (1/\tau_j)\int_0^{t_2}\tilde{\pi}_j^0\,(t'_2)\,e^{t'_2/\tau_j}\,dt'_2\right\}, \tag{5.48}$$

where $\tilde{\pi}_j^0\,(t_2)$ is given by Eq. (5.42).

Due to Eq. (5.44) the reference trajectory possesses only the longitudinal magnetization.

$$\langle M_z^s\,(t_2)\rangle = \langle M_z^0\rangle + \sum_j\,(R_j, M_z)\tilde{\pi}_j^s\,(t_2). \tag{5.49}$$

In case the modulation $\omega_M(t_2)$ is periodic, it is possible to choose such $P_s(t_2)$ that its components Eq. (5.48) are also periodic functions with the same value of the period as $\omega_M(t_2)$. In this case $P_s(t_2)$ describes the steady state to which all trajectories tend as time proceeds. In order to give a simple illuminating example of periodic Zeeman modulation, the case of sinusoidal (harmonic) modulation will be discussed in the next section.

5.3. Harmonic Zeeman modulation. So, let the time-dependent part of the magnetic field Eqs (5.27), (5.28) be such that in Eq. (5.31) we have

$$\omega_M\,(t_2) = \omega_M^0\,\cos\,(\nu_M t_2). \tag{5.50}$$

In this case Eq. (5.38) leads to

$$\int_0^{t_2}\omega_{mn}\,(t'_2)\,dt'_2 = \omega_{mn}^0\,t_2 - (\mu_{mn}\omega_M^0/\nu_M)\,\sin\,\nu_M t_2 \tag{5.51}$$

and so Eq. (5.37) can be replaced by

$$\pi_{mn}\,(t_2) = \pi_{mn}\,(t_1)\,\sum_{k=-\infty}^{+\infty}\mathfrak{J}_k\,(x_{mn})\,\exp\,(-(1/\tau_{mn} + i\omega_{mn}^{(k)})\,t_2), \tag{5.52}$$

where

$$x_{mn} = |\mu_{mn}|\,(\omega_M^0/\nu_M), \tag{5.53}$$

$$\omega_{mn}^{(k)} = \omega_{mn}^0 \mp k\nu_M. \tag{5.54}$$

In Eq. (5.54) the upper sign is valid if $\mu_{mn} > 0$, otherwise the lower sign holds. In deriving Eq. (5.52) we used the Jacoby expansion [6]

$$\exp\,(\pm ix_{mn}\,\sin\,\nu_M t_2) = \sum_{k=-\infty}^{+\infty}\mathfrak{J}_k\,(x_{mn})\,\exp\,(\pm ik\nu_M t_2), \tag{5.55}$$

where $\mathfrak{J}_k\,(x_{mn})$ denotes the Bessel function of the k-th order and that of the first kind [6].

The time-development of the transversal nuclear magnetization Eq. (5.39) calculated on the basis of Eq. (5.52) for the ideal initial state Eq. (5.24) is given by

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$$\langle M_x(t_2)\rangle = -\langle M_z^0\rangle/(I_z, I_z) \sum_{m>n}\sum |(I_x, A_{mn})|^2 \exp(-t_2/\tau_{mn}) \times$$

$$\times \sum_k \mathfrak{I}_k(\omega_M^0/v_M)\cos(\omega_{mn}^{(k)} t_2). \tag{5.56}$$

Hence, for the Fourier transform Eq. (5.20) of this decay function we obtain

$$M_x(\omega) = -\langle M_z^0\rangle/(I_z, I_z) \sum_{m>n}\sum |(I_x, A_{mn})|^2 (\tau_{mn}/\pi) \times$$

$$\times \sum_k \frac{\mathfrak{I}_k(\overset{0}{\omega}_M/v_M)}{1+(\Delta\omega_{mn}^{(k)} \tau_{mn})^2}, \tag{5.57}$$

where

$$\Delta\omega_{mn}^{(k)} = \omega_{mn}^0 + k v_M - \omega, \tag{5.58}$$

$$k = 0, \pm 1, \pm 2, \dots . \tag{5.59}$$

In drawing comparisons between Eq. (5.26) and Eq. (5.57), we are forced to conclude: the harmonic Zeeman modulation Eq. (5.50) splits each «resonance frequency» ω_{mn}^0 into an infinite set of equidistant sideband frequencies Eq. (5.54). In such an infinite multiplet the relative line intensities are given by Bessel functions. This splitting of resonance frequencies does not depend upon ω_{mn}^0. In case the modulation frequency v_M exceeds the width of the whole FID FT spectrum, one speaks about side band spectra of different order caused by the harmonic modulation. In such a side band spectrum of a given order the dependence of relative line intensities upon initial state $P(t_1)$ is similar to that in the FID spectrum.

The phenomenon of side band resonances has been first observed in slow sweep single resonance NMR spectra taken in the presence of harmonic Zeeman modulation. In case of single spin systems, the theory of this phenomenon has been improved by several authors and reviewed in [7, 8]. The case of multispin systems is descussed in [9, 10]. Comparison of the latter paper (in which relaxation is also taken into account) with Eq. (5.57) brings out the full similarity between slow sweep single resonance side-band spectrum and the corresponding Zeeman modulation decay spectrum Eq. (5.57).

In conclusion, let us turn to the steady state longitudinal nuclear magnetization.

In case of harmonic modulation Eq. (5.50) the quasi-static process Eq. (5.41) is given by

$$P_0(t_2) = P_0 + (\hbar\omega_M^0/dkT)\cos(v_M t_2)I_z. \tag{5.60}$$

Using this expression in calculating the steady state Eqs. (5.45), (5.48) we have

$$P_s(t_2) = P_0 + (\hbar\omega_M^0/dkT)\sum_j \frac{(I_z, R_j)}{\sqrt{1+(v_M\tau_j)^2}}\cos(v_M t_2 - \varphi_j)R_j, \tag{5.61}$$

where

$$\tan\varphi_j = v_M\tau_j. \tag{5.62}$$

Hence, the steady state longitudinal magnetization

$$\langle M_z^s(t_2)\rangle = \langle M_z^0\rangle + (\hbar\omega_M^0/dkT)\sum_j \frac{(R_j, M_z)(I_z, R_j)}{\sqrt{1+(v_M\tau_j)^2}}\cos(v_M t_2 - \varphi_j) \tag{5.63}$$

indeed oscillates with the same frequency ν_M as the modulation. Note the typical nonresonant character of the dependence of the amplitude of this oscillation upon ν_M.

In the extreme case of very low frequencies

$$\nu_M \ll 1/\tau_j \qquad (5.64)$$

we get from Eq. (5.63)

$$P_s(t_2) \approx P_0(t_2), \qquad (5.65)$$

whereas in the other extreme

$$\nu_M \gg 1/\tau_j \qquad (5.66)$$

we have

$$P_s(t_2) \approx P_0. \qquad (5.67)$$

Therefore, our first formulation of the dynamical axiom V given in Sec. 3.2 is justified, provided Eq. (5.66) is valid. Moreover, so far as

$$\omega_M^0 \ll \omega_L \qquad (5.68)$$

the amplitude of the steady state oscillations is negligible also in the case Eq. (5.64) holds.

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V. SINIVEE

VEDELIKE TUUMASPINNIDE DÜNAAMIKA PÕHIMÕISTED. 5

Vaba induktsiooni kustumise Fourier' teisenduse spektrite ergutuse meetodist sõltuvad spektraaljoonte intensiivsused ja kujud, kuid mitte nende asupaigad. Alalisele magnetväljale samasuunalise harmooniliselt moduleeriva välja lisamine kutsub esile külgriba spektrite tekkimise. Viimaste sõltuvusel modulatsiooni sagedusest ei ole resonantsiseloomu.

В. СИНИВЕЭ

ОСНОВНЫЕ ПОНЯТИЯ В ЯДЕРНОЙ СПИНОВОЙ ДИНАМИКЕ ЖИДКОСТЕЙ. 5

Способ возбуждения спектров преобразования Фурье сигнала затухания свободной индукции определяет интенсивность и форму спектральных линий, но не их положение. Дополнительное гармонически модулированное магнитное поле, имеющее такое же направление как и постоянное поле, обусловливает появление спектров боковой полосы. Зависимость последних от частоты модуляции имеет нерезонансный характер.