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Thermodynamic theory of narrowing of NMR spectral lines in solids

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A new solution is proposed for the problem of narrowing of the many-pulse NMR spectra in solids. The dynamics of the spin system is investigated when the sample is acted upon by the pulse sequence $90°_y - \tau - (\varphi_x - 2\tau -)^N$ when the field goes off resonance by an amount Δ (φ_x denotes a pulse that rotates the spin through an angle φ around the x axis; 2τ is the distance between pulses). It is shown that when the system is acted upon by pulses and by detuning in a time $t >> \tau$, the spins precess around an effective field ω_{eff} whose magnitude and direction are determined by the parameters φ and $\Delta\tau$. In addition, the spins absorb the quanta of the dipole–dipole interaction modulated by the RF field and by the detuning; the magnitudes of these quanta depend only on the pulse repetition frequency. Within short times $\sim T_2$ ($T_2 \sim \|\mathcal{H}_d\|^{-1} \sim \omega_{\text{loc}}$) the system reaches a quasiequilibrium state [M. Goldman, Spin Temperature and Nuclear Magnetic Resonance in Solids, Oxford U. Press, 1970, Chap. 6] corresponding to thermal mixing of the Zeeman and dipole–dipole interaction reservoirs. The type of the quasiequilibrium depends on the ratio of ω_{eff} to the local field ω_{loc}. The quantum absorption process takes place in times $t >> T_2$ and at $\omega_{\text{eff}} >> \omega_{\text{loc}}$ it is connected with transfer of part of the energy into the dipole–dipole reservoir. This energy transfer does not take place under resonance conditions, i.e., when $n\omega_{\text{eff}} = m\pi/\tau$ (n and m are integers). The resonance conditions correspond to the experimentally observed [L. N. Erofeev *et al.*, Sov. Phys. JETP **48**, 925, (1978)] minima in the magnetization-damping times when φ and $\Delta\tau$ are varied. The variations of the magnetization damping times are calculated for different types of resonances (for different n and m). The kinetics of the damping of the magnetization near the resonances is investigated. The results differ substantially from those obtained by the average-Hamiltonian method (U. Haeberlen and J. S. Waugh, Phys. Rev. **175**, 453, 1968), and explain a number of experiments [W.-K. Rhim *et al.*, Phys. Rev. Lett. **37**, 1764 (1976)]; L. N. Erofeev and B. A. Shumm, JETP Lett. **27**, 149 (1978); L. N. Erofeev *et al.*, Sov. Phys. JETP **48**, 925 (1978)] that contradict this theory.

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1. INTRODUCTION

A number of experimental methods have been recently developed by which to improve considerably the resolution of the lines in nulcear magnetic resonance (NMR) spectra of solids.[1–7] Methods most widely used are many-pulse methods[4–7] of line narrowing, which make it possible in practice to narrow down the NMR spectral lines of solids from several kilohertz to several dozen hertz.[8] In view of the great increase of the resolution of the many-pulse method, it becomes important to develop a theory of line narrowing. The hitherto known theory of many-pulse experiments starts from the premise that the pulse sequence causes the dipole–dipole interaction or part of it to become dependent on the time and average out over the cycle time τ_c. In addition to the time τ_c, which characterizes the motion of the nuclear spins under the influence of the pulses, the many-pulse problem involves one other time T_2, which characterizes the motion of the spins in the local field. If the dipole–dipole interaction is averaged over the time of the cycle, the influence of the local fields on the motion of the spins is neglected. This, of course, is fully justified in the case of one or several cycles at $\tau_c \ll T_2$. However, the damping of the magnetization in many-pulse experiments takes place over time $t \gg T_2$, i.e., over hundreds or thousands of pulse cycles. Therefore the influence of the local fields on the spin dynamics becomes substantial and the abbreviated description of the system is permissible only in the case of averaging over a time interval $T_2 \gg \tau_c$.

It must also be noted that in the average-Hamiltonian theory one does not follow the evolution of the density matrix in time. To the contrary, it is customary to make with respect to the density matrix an additional assumption[9] which in many cases is not justified.[10–12]

On the other hand, there is deep analogy between the behavior of a spin system in fields produced by pulse sequences, on the one hand, and in continuous external RF fields, on the other. This can be particularly clearly traced using as an example the pulse sequence $90°_y - \tau - (\varphi_x - 2\tau -)^N$ as $\varphi \to 0$ and $\tau \to 0$.[10] This circumstance makes it possible to construct for many-pulse line narrowing a theory that is similar to a considerable degree to the theory of continuous "locking" of the spin

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magnetization (spin locking).[13] In a continuous field, over times $\sim T_2$, a quasiequilibrium state is established in the system and corresponds to thermal mixing of the Zeeman and nonsecular parts of the dipole–dipole reservior of the interactions[14]; at $t \gg T_2$ the system reaches a state with one temperature.[14] The experimentally observed[10,11] decrease of the observed magnetization in many-pulse experiments offers evidence that in the case of a pulse field, over times $\sim T_2$, a quasiequilibrium is likewise established in the system and is determined by the redistribution of the energy between the Zeeman and dipole–dipole reservoirs of the interactions. The character of the quasiequilibrium in many-pulse experiments differs at the same time substantially from the case of a continuous field in the presence of dipole echo,[10,11] this being a reflection of the external pulse actions.

Over times $t \gg T_2$, the behavior of the spin system in many-pulse experiments is determined both by the quasiequilibrium that is formed over times $\sim T_2$, and by the absorption of quanta of external fields. The nature of the physical processes that lead to damping of the magnetization cannot be understood without a correct description of the quasiequilibrium in the system. This description is closely connected with the choice of the averaging-time scale. The arguments presented above show that this time scale in the many-pulse problem is T_2.

The problem of line narrowing in the case of exact equality of the frequency of the RF field to the Larmor frequency of the spins was recently considered, and a new approach to the theory of narrowing of many-pulse NMR spectra was proposed,[12] based on the description of the quasiequilibrium in the system and of the multispin processes of absorption of quanta by the nuclear spins. The present paper is devoted to an exposition of a new theory of many-pulse narrowing of NMR spectra in solids, using as an example the pulse sequence $90°_y - \tau - (\varphi_x - 2\tau -)^N$ (Ref. 10) with the field off-resonance by an amount Δ (in frequency units). The described approach can be used also for other pulse sequences used to narrow down NMR lines in solids.

2. QUASIEQUILIBRIUM OF SPIN SYSTEMS. THE EFFECTIVE FIELD

We consider a spin system acted upon by a pulse sequence $90°_y - \tau - (\varphi_x - 2\tau -)^N$, where φ_x denotes the pulse that rotates the spins through an angle φ about the x axis; 2τ is the distance between the pulses (Fig. 1). It is assumed that the constant field directed in the laboratory frame along the z axis is detuned from the resonance value ω_0/γ (ω_0 is the Larmor-precession frequency of the nuclear spins and γ is the gyromagnetic ratio) by an amount Δ (in frequency units). In a coordinate system that rotates with Larmor frequency around the z axis, the equation for the spin-system density matrix $\rho(t)$ takes the form ($\hbar = 1$)

$$i\frac{d\rho}{dt} = [-f(t)\hat{S}_x + \Delta\hat{S}_z + \hat{\mathcal{H}}_d^z, \rho(t)], \tag{1}$$

where $f(t)$ is a pulse function defined by the formula

$$f(t) = \varphi \sum_{k=0}^{\infty} \delta(\tau + 2k\tau - t), \tag{2}$$

and $\hat{\mathcal{H}}_d^z$ is the secular (with respect to the z axis) part of the dipole–dipole interaction. We proceed to the interaction representation in the pulses and in the detuning, i.e., we make the substitution

$$\rho(t) = \hat{L}(t)\bar{\rho}(t)\hat{L}^{-1}(t), \tag{3}$$

where

$$\hat{L}(t) = T\exp\left\{-i\int_0^t [-f(t')\hat{S}_x + \Delta\hat{S}_z]dt'\right\}, \tag{4}$$

and T stands for a product ordered in time. We introduce now the effective pulse

$$\exp(-2i\tau\omega_{eff}\hat{S}) = \exp(-i\Delta\tau\hat{S}_z)P_{-\varphi}\exp(i\Delta\tau\hat{S}_z), \tag{5}$$

which characterizes the external action on the system over the interval 2τ. It follows from (5) that ω_{eff} is defined by

$$\cos(2\omega_{eff}\tau) = \cos\varphi\cos^2(\Delta\tau) - \sin^2(\Delta\tau), \tag{6}$$

and its direction is obtained from the formulas

$$n_x = \frac{\sin\varphi\cos(\Delta\tau)}{\sin(2\omega_{eff}\tau)}, \quad n_y = 0, \quad n_z = \frac{\sin(2\Delta\tau)\cos^2(\varphi/2)}{\sin(2\omega_{eff}\tau)}. \tag{7}$$

(It turned out in a preliminary discussion that an analogous value of ω_{eff} was obtained independently[15] on the basis of an analysis of the experimental data.) We introduce a pulse function $g(t)$ analogous to (2)

$$g(t) = \theta\sum_{k=0}^{\infty}\delta(t - 2k\tau - \tau), \tag{8}$$

so that

$$\exp\left[i\int_0^t g(t')dt'\,\hat{S}_n\right] = \exp(i\theta\hat{S}_n)\exp(i\theta\hat{S}_n)\ldots\exp(i\theta\hat{S}_n), \tag{9}$$

where $\theta = 2\omega_{\text{eff}}\tau$ is the angle of rotation of the effective pulse about the axes $\mathbf{n}(n_x, n_y, n_z)$, and the number of factors in the right-hand side of (9) is determined by the instant of time t. We can then rewrite (1) in the form

$$i\frac{d\bar{\rho}}{dt} = \left[\exp\left\{-i\int_0^t g(t')dt'\,\hat{S}_n\right\}\hat{\mathcal{H}}_d^z\exp\left\{i\int_0^t g(t')dt'\,\hat{S}_n\right\}, \bar{\rho}(t)\right]. \tag{10}$$

It is convenient to expand the operator $\hat{\mathcal{H}}_d^z$ as follows

$$\hat{\mathcal{H}}_d^z = A_0\hat{\mathcal{H}}_d^0 + A_1\hat{\mathcal{H}}_d^1 + A_{-1}\hat{\mathcal{H}}_d^{-1} + A_2\hat{\mathcal{H}}_d^2 + A_{-2}\hat{\mathcal{H}}_d^{-2}, \tag{11}$$

where $\hat{\mathcal{H}}_d^0$ is the secular part and $\hat{\mathcal{H}}_d^{\pm1}$ and $\hat{\mathcal{H}}_d^{\pm2}$ the nonsecular parts of the dipole–dipole interaction relative to the n axis and are defined by

$$\hat{\mathcal{H}}_d^0 = \sum_{i>j} b_{ij}\left\{2\hat{S}_{ni}\hat{S}_{nj} - \frac{1}{2}(\hat{S}_{ni}^+\hat{S}_{nj}^- + \hat{S}_{ni}^-\hat{S}_{nj}^+)\right\},$$

$$\hat{\mathcal{H}}_d^1 = -\frac{3}{2}\sum_{i>j} b_{ij}(\hat{S}_{ni}\hat{S}_{nj}^+ + \hat{S}_{ni}^+\hat{S}_{nj}), \hat{\mathcal{H}}_d^{-1} = -\frac{3}{2}\sum_{i>j} b_{ij}(\hat{S}_{ni}\hat{S}_{nj}^- + \hat{S}_{ni}^-\hat{S}_{nj}), \tag{12}$$

$$\hat{\mathcal{H}}_d^2 = -\frac{3}{4}\sum_{i>j} b_{ij}\hat{S}_{ni}^+\hat{S}_{nj}^+, \hat{\mathcal{H}}_d^{-2} = -\frac{3}{4}\sum_{i>j} b_{ij}\hat{S}_{ni}^-\hat{S}_{nj}^-; \quad \hat{S}_n^{\pm} = \hat{S}_{ny} \pm i\hat{S}_{nz}.$$

The coefficients A_0, $A_{\pm1}$, and $A_{\pm2}$ in (11) can be easily determined by changing over to a coordinate system in which the x axis coincides with the n direction, with

$$A_0 = (3n_z^2 - 1)/2, \quad A_1 = n_z(1-n_z^2)^{1/2}e^{-i\psi}, \quad A_{-1} = A_1^* = n_z(1-n_z^2)^{1/2}e^{i\psi},$$
$$A_2 = (1-n_z^2)e^{-2i\psi}, \quad A_{-2} = A_2^* = (1-n_z^2)e^{2i\psi}, \tag{13}$$
$$\cos\psi = n_z n_y/\{(1-n_z^2)^{1/2}(1+n_z)\}.$$

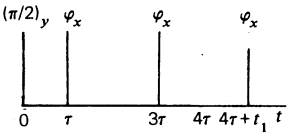

FIG. 1.

It is seen from (5) and (9) that the spins rotate in the average field ω_{eff}. To take this rotation into account, it is convenient to change to a coordinate system that rotates at a frequency ω_{eff} :

$$\rho^*(t)=\exp(i\omega_{eff}t\hat{S}_n)\tilde{\rho}(t)\exp(-i\omega_{eff}t\hat{S}_n). \tag{14}$$

The density matrix satisfies then the equation

$$i\frac{d\rho^*}{dt}=[-\omega_{eff}\hat{S}+A_0\hat{\mathcal{H}}_d^0+A_1\Phi(t)\hat{\mathcal{H}}_d^1+A_{-1}\Phi^*(t)\hat{\mathcal{H}}_d^{-1}$$
$$+A_2\chi(t)\hat{\mathcal{H}}_d^2+A_{-2}\chi^*(t)\hat{\mathcal{H}}_d^{-2},\rho^*], \tag{15}$$

where

$$\Phi(t)=\exp\left\{-i\left[\int_0^t g(t')dt'-\omega_{eff}t\right]\right\},\ \chi(t)=\exp\left\{-2i\left[\int_0^t g(t')dt'-\omega_{eff}t\right]\right\} \tag{16}$$

are periodic functions with a pulse repetition period 2τ. It is seen from (15) that the fields acting on the spins can be divided into constant fields ω_{eff} and $A_0\hat{\mathcal{H}}_d^0$ and the fields $\sim\Phi(t)$ and $\chi(t)$ that which fluctuate rapidly in time. The greatest influence on the motion of the nuclear spins is exerted by the field ω_{eff} and by the constant local field, while the contribution of the rapidly oscillating fields can be accounted for by perturbation theory. To take into account the fluctuating fields, we expand $\Phi(t)$ and $\chi(t)$ in Fourier series:

$$\Phi(t)=\sum_{n=-\infty}^{\infty}c_ne^{-in\pi t/\tau},\quad \chi(t)=\sum_{n=-\infty}^{\infty}b_ne^{-in\pi t/\tau},$$
$$c_n=\frac{(-1)^n\sin\omega_{eff}\tau}{n\pi+\omega_{eff}\tau},\quad b_n=\frac{(-1)^n\sin 2\omega_{eff}\tau}{n\pi+2\omega_{eff}\tau}, \tag{17}$$

and express (15) in the form

$$i\frac{d\rho^*}{dt}=\Big[\hat{\mathcal{H}}_0+A_1\sum_{n=-\infty}^{\infty}{}'c_ne^{-in\pi t/\tau}\hat{\mathcal{H}}_d^1+A_{-1}\sum_{n=-\infty}^{\infty}{}'c_n^*e^{in\pi t/\tau}\hat{\mathcal{H}}_d^{-1}$$
$$+A_2\sum_{n=-\infty}^{\infty}{}'b_ne^{-in\pi t/\tau}\hat{\mathcal{H}}_d^2+A_{-2}\sum_{n=-\infty}^{\infty}{}'b_n^*e^{in\pi t/\tau}\hat{\mathcal{H}}_d^{-2},\rho^*\Big], \tag{18}$$

where

$$\hat{\mathcal{H}}_0=-\omega_{eff}\hat{S}_n+A_0\hat{\mathcal{H}}_d^0+\frac{\sin\omega_{eff}\tau}{\omega_{eff}\tau}(A_1\hat{\mathcal{H}}_d^1+A_{-1}\hat{\mathcal{H}}_d^{-1})$$
$$+\frac{\sin 2\omega_{eff}\tau}{2\omega_{eff}\tau}(A_2\hat{\mathcal{H}}_d^2+A_{-2}\hat{\mathcal{H}}_d^{-2}) \tag{19}$$

and the prime on the summation sign will denote hereafter that the zeroth harmonic is left out of the summation.

It is known[14] that certain terms of the Hamiltonian can be accounted for by perturbation theory only if they are much smaller than each of the operators contained in the principal Hamiltonian $\hat{\mathcal{H}}_0$. During the same time, the time-dependent part of the Hamiltonian in (18) is comparable in order of magnitude with the dipole–dipole terms contained in $\hat{\mathcal{H}}_0$. We therefore carry out first a number of canonical transformations aimed at lowering the order of the time-dependent terms of the Hamiltonian in (18).[12]

The remainder of the analysis is substantially different in the cases when $\omega_{loc}=[\mathrm{Sp}(\hat{\mathcal{H}}_d^z)^2/\mathrm{Sp}(\hat{S}_z)^2]^{1/2}\sim\omega_{eff}$ and when $\omega_{loc}\ll\omega_{eff}$. Let first $\omega_{eff}\sim\omega_{loc}$. For each nonzero harmonic of $\Phi(t)$ we carry out the following canonical transformations of Eq. (18):

$$\tilde{\rho}(t)=\exp\left(-i\frac{m\pi t}{\tau}\hat{S}_n\right)\exp(i\hat{R}_m^1)\exp\left(i\frac{m\pi t}{\tau}\hat{S}_n\right)$$
$$\times\rho^*(t)\exp\left(-i\frac{m\pi t}{\tau}\hat{S}_n\right)\exp(-i\hat{R}_m^1)\exp\left(i\frac{m\pi t}{\tau}\hat{S}_n\right), \tag{20}$$

where

$$\hat{R}_m^1=\frac{i\tau}{m\pi+\omega_{eff}\tau}(A_1c_m\hat{\mathcal{H}}_d^1-A_{-1}c_m^*\hat{\mathcal{H}}_d^{-1}). \tag{21}$$

It is easy to show that

$$i[\hat{R}_m^1,\ (\omega_{eff}+m\pi/\tau)\hat{S}_n]=A_1c_m\hat{\mathcal{H}}_d^1+A_{-1}c_m^*\hat{\mathcal{H}}_d^{-1}. \tag{22}$$

Therefore, as a result of the performance of the canonical transformations (20), the terms $A_1c_me^{-im\pi t/\tau}\hat{\mathcal{H}}_d^1$ and $A_{-1}c_m^*e^{im\pi t/\tau}\hat{\mathcal{H}}_d^{-1}$ will drop out of (18). Next, for each nonzero harmonic of $\chi(t)$ we carry out similarly the transformations

$$\rho'(t)=\exp\left(-i\frac{m\pi t}{2\tau}\hat{S}_n\right)\exp(i\hat{R}_m^2)\exp\left(i\frac{m\pi t}{2\tau}\hat{S}_n\right)$$
$$\times\tilde{\rho}(t)\exp\left(-i\frac{m\pi t}{2\tau}\hat{S}_n\right)\exp(-i\hat{R}_m^2)\exp\left(i\frac{m\pi t}{2\tau}\hat{S}_n\right), \tag{23}$$

where

$$\hat{R}_m^2=\frac{i\tau}{m\pi+\theta}(A_2b_m\hat{\mathcal{H}}_d^2-A_{-2}b_m^*\hat{\mathcal{H}}_d^{-2}). \tag{24}$$

Following the canonical transformations (20) and (23), we can rewrite (18) in the form

$$i\frac{d\rho'}{dt}=[\hat{\mathcal{H}}_0+\hat{V}(t),\rho']. \tag{25}$$

Here

$$\hat{V}(t)\sim\frac{\sin\omega_{eff}\tau}{\pi}\tau\omega_{loc}^2\sim\frac{\tau^2\omega_{loc}^3}{\pi},$$

since $\omega_{eff}\sim\omega_{loc}$ and $\omega_{loc}\tau<1$. Thus, the canonical transformations (20) and (23) decrease the time dependent part of the Hamiltonian by a factor $\varepsilon=(\tau\omega_{loc})^2/\pi$, and this part can now be treated by perturbation theory.

Since the order of magnitude of the perturbation $\hat{V}(t)$ is now much smaller than the order of magnitude of the interactions that enter in $\hat{\mathcal{H}}_0$, we can assume[14] that at $\omega_{eff}T_2\sim1$ the density matrix of the system assumes after a time $\sim T_2$, accurate to small off-diagonal terms, the form

$$\rho_{st}'=1-\alpha_{st}\hat{\mathcal{H}}_0,\quad \mathrm{Sp}\,\rho_{st}'=1. \tag{26}$$

Over times $\sim T_2$ we can also neglect the energy absorbed by the system from the external RF fields, and use the energy conservation law (which is accurate here up to terms $\sim\tau^4\omega_{loc}^5$):

$$\mathrm{Sp}\,\rho'(0)\hat{\mathcal{H}}_0=\mathrm{Sp}\,\rho_{st}'\hat{\mathcal{H}}_0, \tag{27}$$

where $\rho'(0)$ is the density matrix $\rho'(t)$ at the instant $t=0$. In a coordinate system that rotates with Larmor frequency around the z axis, the density matrix $\rho(t)$ at the instant of time $t=0$ is given by

$$\rho(0)=1-\alpha_0\omega_0\hat{S}_x,\quad \mathrm{Sp}\,\rho=1 \tag{28}$$

(ρ_0 is the initial temperature of the Zeeman reservoir). Taking (3) and (14) into account, we find that

$$\rho^*(0)=\rho(0)=1-\alpha_0\omega_0(n_x\hat{S}_n+\gamma_1\hat{S}_n^++\gamma_2\hat{S}_n^-), \tag{29}$$

where the coefficients γ_1 and γ_2 can be easily determined

by resolving the operator $\hat{S}_x$ along the axes of a coordinate system in which the z axis is directed along the effective field; their values, however, are not used in the subsequent calculations. We now obtain for the density matrix $\rho'(0)$

$$\rho'(0)=\prod_{l=-\infty}^{\infty}{}'\prod_{m=-\infty}^{\infty}{}'\exp(i\hat{R}_l^2)\exp(i\hat{R}_m^1)\rho^*(0)\exp(-i\hat{R}_m^1)\exp(-i\hat{R}_l^2). \quad (30)$$

We see therefore that the density matrix $\rho'(0)$ differs from $\rho^*(0)$ only by the terms $\sim\omega_{loc}^3\tau^2$ that are responsible for the dipole echo. Accurate to these terms we obtain, using (19) and (29),

$$\mathrm{Sp}\{\rho'(0)\hat{\mathcal{H}}_0\}=\alpha_0\omega_0\omega_{eff}n_z\,\mathrm{Sp}\{\hat{S}_n\}^2. \quad (31)$$

In the derivation of (31) we took into account the following easily verified relations:

$$\mathrm{Sp}\{\hat{S}_n\hat{\mathcal{H}}_d^0\}=\mathrm{Sp}\{\hat{S}_n+\hat{\mathcal{H}}_d^{-1}\}=\mathrm{Sp}\{\hat{S}_n-\hat{\mathcal{H}}_d^1\}=0. \quad (32)$$

In Appendix A it is shown[formulas (A.2) and (A.4)]that

$$\mathrm{Sp}\{\hat{\mathcal{H}}_d^1\hat{\mathcal{H}}_d^{-1}\}/\mathrm{Sp}\{\hat{S}_n\}^2=3\omega_{loc}^2, \quad (33)$$

$$\mathrm{Sp}\{\hat{\mathcal{H}}_d^2\hat{\mathcal{H}}_d^{-2}\}/\mathrm{Sp}\{\hat{S}_n\}^2=^3/_8\omega_{loc}^2, \quad (34)$$

where

$$\omega_{loc}^2=\mathrm{Sp}\{\hat{\mathcal{H}}_d^0\}^2/\mathrm{Sp}\{\hat{S}_n\}^2.$$

From (27), taking relations (29)–(34) into account, we obtain

$$\alpha_{st}/\alpha_0=\omega_0\omega_{eff}n_z$$
$$\times\left\{\omega_{eff}^2+\left[A_0^2+6A_1A_{-1}\frac{\sin^2(\theta/2)}{(\theta/2)^2}+\frac{3}{4}A_2A_{-2}\frac{\sin^2\theta}{\theta^2}\right]\omega_{loc}^2\right\}^{-1}. \quad (35)$$

We can now easily find that

$$M_{st}^n/M_0=\alpha_{st}\omega_{eff}/\alpha_0\omega_0. \quad (36)$$

Here M_{st} is the stationary value of the magnetization along the direction of the effective field, and M_0 is the magnetization at $t=0$. In experiment, however, one measures the ratio M_{st}^x/M_0,[11] where M_{st}^x is the projection of M_{st}^n on the x axis of the coordinate system that rotates with Larmor frequency ω_0 around the z axis. The canonical transformations (3) and (14) show that at the instants of time $t_N=2N\tau$ we have $\rho^*(t_N)=\rho(t_N)$. Therefore, accurate to small corrections connected with the transformations (20) and (23), the following relation holds true at these instants of time: $M_{st}^x=M_{st}^n n_x$. At an arbitrary instant of time $t=2N\tau+t_1(-\tau\leqslant t_1\leqslant\tau)$ the canonical transformations (3) and (14) lead to an additional rotation of the magnetization through an angle Δt_1 about the z axis of the coordinate frame that rotates with Larmor frequency. The final expression for the ratio M_{st}^x/M_0 is therefore

$$M_{st}^x/M_0=\omega_{eff}^2n_z^2\cos\Delta t_1$$
$$\times\left\{\omega_{eff}^2+\left[A_0^2+6A_1A_{-1}\frac{\sin^2(\theta/2)}{(\theta/2)^2}+\frac{3}{4}A_2A_{-2}\frac{\sin^2\theta}{\theta^2}\right]\omega_{loc}^2\right\}^{-1}. \quad (37)$$

Formula (37) shows that when a quasiequilibrium state is established in the system the stationary magnetization decreases for two reasons. First, within a time $\sim T_2$ the magnetization initially parallel to the observation axis becomes parallel to the direction of the effective field; the magnetization component perpendicular to the effective field vanishes during that time.[11] For this reason, the observed magnetization decreases after a time $\sim T_2$ by a factor n_x^2. Second, over times $\sim T_2$ energy exchange takes place between the Zeeman and the dipole–dipole interaction reservoirs.[14] This exchange is not connected with the change of the orientation of the magnetization and leads likewise to a decrease of the observed signal. Figure 2 shows plots of M_{st}^x/M_0 against the parameter $\Delta\tau$ for different angles φ, as calculated from formula (37) with $t_1=0$. A comparison of (37) with the experimental data of Fig. 2 shows, in the main, agreement between theory and experiment.[15] The deviation of the experimental data from the theoretical curves are due to errors in the orientation of the crystal.[15]

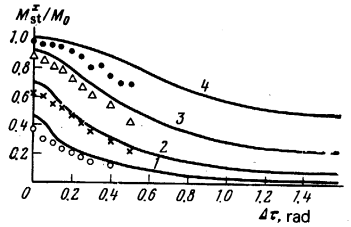

FIG. 2. Plot of M_{st}^x/M_0 against the parameter Δt for a CaF_2 crystal whose [111] axis is oriented along H_0 (H_{loc}= 0.86 Oe): curve 1—for $\varphi=22.5°$, 2—for $\varphi=36°$, 3—for $\varphi=60°$, 4—for $\varphi=90°$. Experimental points: ○—$\varphi=22.5°$; ×—$\varphi=36°$; Δ—$\varphi=60°$, ●—$\varphi=90°$.

It was shown previously[12] for the on-resonance problem that the magnetization M_x is a periodic function with a pulse repetition period 2τ, and the waveform of the signal between the pulses was calculated. In our case the magnetization is also quasiperiodic with period 2τ, but its variation in the intervals between the pulses is determined not only by the dipole–dipole interactions, but above all by the action of the detuning ($M_x\sim\cos\Delta(t-2\tau)$, $\tau\leqslant t\leqslant3\tau$).

In the case $\omega_{loc}\sim\omega_{eff}$ considered, the absorption of quanta from the external fields $\pi/\tau\gg\omega_{eff}$ and $\pi/\tau\gg\omega_{loc}$ is accompanied by a change in the orientation of a large number of spins, and is primarily connected with a change of the energy of the dipole–dipole interactions. At $\omega_{eff}\gg\omega_{loc}$ the absorption of the quanta is determined by transitions of the system to different energy levels of the Zeeman reservoir; only a small fraction of the absorbed energy enters the dipole–dipole reservoir. The canonical transformations (20) and (23) of Eq. (18) should be supplemented[12] at $\omega_{eff}\gg\omega_{loc}$ by canonical transformations for the zeroth harmonics of $\Phi(t)$ and $\chi(t)$, whose amplitudes are $\pi/\omega_{loc}\tau$ times larger than of the remaining harmonics, and which were previously taken into account accurately. Accordingly, we obtain in place of the density matrix $\rho'(t)$ (25)

$$t\frac{d\rho'}{dt}=[-\omega_{eff}\hat{S}_n+A_0\hat{\mathcal{H}}_d^0+\hat{V}_1(t),\rho'], \quad (38)$$

where $\hat{V}_1(t)\sim\tau\omega_{loc}^2$ and can also be regarded as a small perturbation. The spin system is now characterized by two integrals of motion, and in times $\sim T_2$ its density matrix takes the form

$$\rho_{st}=1+\alpha_{st}\omega_{eff}\hat{S}_n-^1/_2\beta_{st}A_0\hat{\mathcal{H}}_d^0,\quad \mathrm{Sp}\,\rho_{st}=1. \quad (39)$$

The condition for the conservation of $\hat{\mathcal{H}}_d^0$ over times $\sim T_2$ leads to $\beta_{st}=0$. The decrease of the stationary magnetization from its initial value M_0 is determined almost completely by the fact that the magnetization assumes in the time $\sim T_2$ the direction of the effective field:

$$M_{st}^z/M_0=n_z^2\cos\Delta t_1. \tag{40}$$

3. RESONANCE EFFECTS

We consider now the behavior of the spin at times $t\gg T_2$. In addition to the effective field, the time-dependent terms of the perturbation $\hat{V}_1(t)$ in (38) begin to influence strongly the dynamics of the system. Each of the terms contained in $\hat{V}_1(t)$ is a certain part of the dipole–dipole interaction modulated by the pulses and by the detuning. The nonsecular terms of the perturbation $\hat{V}_1(t)$ are sources of quanta absorbed by the nuclear spins that interact via $\hat{\mathcal{H}}_d^0$. As already noted, at $\omega_{eff}\sim\omega_{loc}$ the bulk of the absorbed energy goes to the Zeeman reservoir, and only a small fraction of the energy is absorbed by the dipole–dipole reservoir. It is therefore clear that the most effective action on the system should be exerted by terms $\hat{V}_1(t)$ that ensure absorption of the energy exclusively by the Zeeman reservoir and exclude fully any transfer of energy to the dipole–dipole reservoir. Such an absorption process is possible at

$$n\omega_{eff}=m\pi/\tau, \tag{41}$$

where n is the number of absorbing spins and m is the number of quanta absorbed by them. Relation (41) indicates the conditions that must be satisfied by the perturbation terms $\hat{V}_1(t)$ that lead to absorption of energy from the external fields only by the Zeeman part of the interaction reservoir. First, these terms must serve as a source of m quanta; second, they must change the spin projection on the ω_{eff} axis by n units; in the case of $S=\frac{1}{2}$ this is equivalent to spin operators that change the orientation of n spins (m and n are integers). The corresponding term of the perturbation $\hat{V}_1(t)$ will be called the n-spin resonance term that causes absorption of m quanta. Formula (41) shows also that the resonant term defined in this manner leads to an effective resonant absorption only at certain values of the pulse rotation angles φ and of the parameter $\Delta\tau$, namely, when ω_{eff} satisfies (41) for the given n and m. The corresponding value of the effective field will be called resonant. The values of effective fields for certain resonant processes are listed in the table. When ω_{eff} deviates from the resonant values, a fraction of the energy of the quanta of the external fields should be transferred to the dipole–dipole reservoir and this leads (since $\omega_{loc}\ll\omega_{eff}$) to an increase of the damping time of the magnetization.[12] Thus, if the parameters $\Delta\tau$ and φ have values such that $\omega_{eff}=m\pi/n\tau$, i.e., as follows from formula (6), if

$$\cos\varphi\cos^2\Delta\tau-\sin^2\Delta\tau=\cos(2\pi m/n) \tag{42}$$

minima (relative to φ and $\Delta\tau$) should be observed in the magnetization damping times.

We now determine the order of magnitude of certain resonant terms. The term $\sim\tau\omega_{loc}^2$ can be only a three-spin resonant term. We confine ourselves hereafter to the resonant terms responsible for absorption of not more than two quanta. Processes with absorption of many quanta have a negligibly small probability and are not observed in experiment.[15] The three-spin resonant term that causes absorption of one quantum takes the following form:

$$\hat{R}_3(t)=\tau K_3 e^{i\pi t/\tau}[\hat{\mathcal{H}}_d^1,\hat{\mathcal{H}}_d^2],\quad K_3=-A_1A_2\sum_{m=-\infty}^{\infty}\frac{c_m b_{-m-1}}{\omega_{eff}\tau+m\pi}. \tag{43}$$

The only resonant term $\sim\tau\omega_{loc}^3$ is the three-spin resonant term

$$\hat{R}_4(t)=\tau^2K_4e^{i\pi t/\tau}[\hat{\mathcal{H}}_d^2,[\hat{\mathcal{H}}_d^2,\hat{\mathcal{H}}_d^0]],\quad K_4=\text{const}. \tag{44}$$

It is interesting to note that formula (42) means (at $n=4$ and $m=1$) that in the considered cycle without detuning, in the case of 90° pulses, the main contribution to the damping is made by the four-spin resonant process. This is in full agreement with the conservation of the dipole energy in the investigated cycle without detuning for 90° pulses. It can be shown directly from (1) that at $\varphi=\pi/2$ and at $\Delta=0$ the energy of the dipole reservoir is in fact conserved (this circumstance was pointed out to the authors by V. E. Zobov). Finally, we indicate that the resonant term $\sim\tau^3\omega_{loc}^4$ is a five-spin resonant term. The resonant terms of higher order of smallness lead to processes that have not been observed in experiment.

If we confine ourselves to not more than two-quantum resonant processes produced by resonant terms with order of magnitude not lower than $\tau^3\omega_{loc}^4$, and if we also recognize that the four-spin resonant process with absorption of two quanta is realized only at $\Delta\tau=\pi/2$, then we can see from the table that there should be four resonance curves in the $(\varphi,\Delta\tau)$ plane. Figure 3 shows

TABLE I.

Number of absorbing spins	Number of absorbed quanta	ω_{eff}	$\cos(2\omega_{eff}\tau)$	Number of absorbing spins	Number of absorbed quanta	ω_{eff}	$\cos(2\omega_{eff}\tau)$
3	1	$\pi/3\tau$	−0.5	4	2	$\pi/2\tau$	−1.0
3	2	$2\pi/3\tau$	−0.5	5	1	$\pi/5\tau$	0.309
4	1	$\pi/4\tau$	0.0	5	2	$2\pi/5\tau$	−0.809

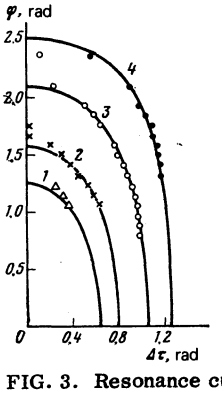

FIG. 3. Resonance curves for single-quantum five-, four-, and three-spin resonances (curves 1—3, respectively) and two-quantum five-spin resonance (curve 4). The experimental points correspond to: Δ—single-quantum five-spin resonances, ×—single-quantum four-spin resonance, ○—single-quantum three-spin resonance, ●—two-quantum five-spin resonance.

these curves, as well as the experimental points[15] at which minima were actually observed in the damping times of the magnetization. The agreement with the experimental data[15] is good.

4. KINETICS OF RESONANT PROCESSES

The kinetics of the damping of magnetization at $t \gg T_2$ will be considered using as an example a three-spin resonant single-quantum process. The equation for the density matrix takes in this case the form

$$i\frac{d\rho'}{dt}=\Big[-\Big(\frac{\pi}{3\tau}-\Delta\omega_{eff}\Big)S_n+A_0\hat{\mathcal{H}}_d^0-\tau A_1A_2Q\Big(\frac{\pi}{3}\Big)e^{i\pi t/\tau}[\hat{\mathcal{H}}_d^1,\hat{\mathcal{H}}_d^2]$$

$$-\tau A_1A_2Q\Big(\frac{\pi}{3}\Big)e^{-i\pi t/\tau}[\hat{\mathcal{H}}_d^{-1},\hat{\mathcal{H}}_d^{-2}],\rho'\Big], \qquad (45)$$

where

$$Q\Big(\frac{\pi}{3}\Big)=\sum_{m=-\infty}^{\infty}\frac{c_mb_{-1-m}}{\pi/3+m\pi}. \qquad (46)$$

It is shown in Appendix B that $Q(\pi/3)=3^{-1/2}$. In (45), $\Delta\omega_{eff}$ is the deviation from the three-spin resonant field $\omega_{eff}=\pi/3\tau$. It is assumed that $\Delta\omega_{eff}$ is small enough to be able to neglect the influence of other resonant processes at momentum rotation angles φ and at detunings Δ connected by relations (42) (at $m=1$ and $n=3$). The problem under consideration is quite analogous to the problem solved in saturation theory.[16] The main results of this theory also remain valid. In particular, the spin system absorbs the quanta of the dipole–dipole interaction modulated by the pulses and by the detuning prior to equalization of the temperatures of the Zeeman and dipole–dipole reservoirs. The residual magnetization M_{res}^x is then connected with the quasistationary magnetization M_{st}^x by the formula

$$\frac{M_{res}^x}{M_{st}^x}=\frac{(\Delta\omega_{eff})^2}{(\Delta\omega_{eff})^2+A_0^2\omega_{loc}^2}. \qquad (47)$$

We see therefore that only at exact resonance ($\Delta\omega_{eff}=0$, $\omega_{eff}=\pi/3\tau$) does the magnetization attenuate to zero. This conclusion agrees well with the experimental data.[15] In practice a deviation from the resonant field value can be obtained either by changing the pulse rotation angle φ or by changing the parameter $\Delta\tau$. Figure 4a shows plots of M_{res}^x/M_{st}^x at $\varphi=72°$ against the parameter $\Delta\tau$, while Fig. 4b shows the analogous dependence of M_{res}^x/M_{st}^x on the angle φ at the value of $\Delta\tau$ corresponding to the investigated resonant process at $\varphi=72°$. The experimental data shown in Fig. 4 (Ref. 15) agrees satisfactorily with the theory. Some discrepancy between theory and experiment can apparently be attributed to the influence of other resonant processes, also to the fact that the pulses are not ideal.

It is easy to write out for our problem the saturation equations[16] and find that equalization of the temperatures of the Zeeman and dipole–dipole reservoirs proceeds exponentially, and the rate of this process is given by

$$\Big(\frac{1}{T_2}\Big)_{res}=6\pi\tau^2|A_1A_2|^2\frac{\mathrm{Sp}(\hat{G}^3_{-3\Delta\omega_{eff}}\hat{G}^{-3}_{3\Delta\omega_{eff}})}{\mathrm{Sp}(\hat{S}_n)^2}\Big[1+\frac{(\Delta\omega_{eff})^2}{A_0^2\omega_{loc}^2}\Big]. \qquad (48)$$

The operators $\hat{G}^{\pm3}_{\mp3\Delta\omega_{eff}}$ are defined here by the relation

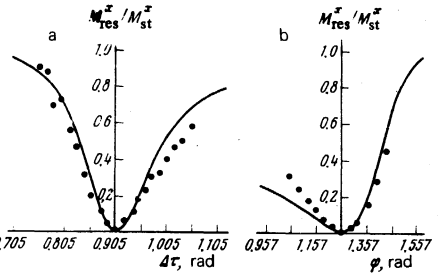

FIG. 4. Dependence of M_{res}^x/M_{st}^x in three-spin resonance for single-crystal CaF_2 ($H_{res}=0.86$ Oe): a) on Δt at $\varphi=72°$, b) on the angle φ at $\Delta t=0.905$; ●–experimental points.

$$G_\omega^{\pm3}=\int_{-\infty}^{\infty}e^{i\omega t}G^{\pm3}(t)\,dt, \qquad (49)$$

where

$$G^3(t)=[\exp(iA_0\hat{\mathcal{H}}_d^0t)\hat{\mathcal{H}}_d^1\exp(-iA_0\hat{\mathcal{H}}_d^0t),\ \exp(iA_0\hat{\mathcal{H}}_d^0t)\hat{\mathcal{H}}_d^2\exp(-iA_0\hat{\mathcal{H}}_d^0t)],$$
$$G^{-3}(t)=\{G^3(t)\}^+. \qquad (50)$$

The plus sign denotes a transition to the complex-conjugate operator. The main conclusion of (48) is that the magnetization damping time in three-spin resonance is $\sim\tau^{-2}$. In perfect analogy, we can find also:

a) that the magnetization damping time in four-spin resonance is $\sim\tau^{-4}$;

b) that the magnetization damping time in five-spin resonance is $\sim\tau^{-6}$.

The experimental data[15] obtained for cases a) and b) are close to the theoretical results. The magnetization damping time in three-spin resonance, according to the data of Ref. 15, is $\sim\tau^{-2.5}$. The difference between theory and experiment is due here apparently to the influence of other resonant processes and to inhomogeneity of the field H_1. From formula (48) we can also determine the dependence of the magnetization damping time in the three-spin resonant process on the pulse rotation angle φ:

$$T_{2\,res}\sim\sin^{-6}{}^1/_2\varphi(4\cos^2{}^1/_2\varphi-1)^{-1}. \qquad (51)$$

Formula (51) agrees with the experimental data.[15] A corresponding comparison of theory with experiment is shown in Fig. 5.

We note that at $\varphi=\pi/2$ and $\Delta\tau=\pi/4$ we have $A_0=0$ [see formula (7) and (13)]. Under these conditions the quasiequilibrium (39) can therefore not be established because there is no mixing interaction $\hat{\mathcal{H}}_d^0$, and the foregoing treatment of three-spin resonance at this point of the $(\varphi,\Delta t)$ plane is no longer correct.

At arbitrary values of φ and $\Delta\tau$, the damping time of the observed signal is influenced simultaneously by several resonant processes. We account for their influence on the damping of the magnetization using as an example the joint action of three- and four-spin resonances. The equation for the density matrix in this case can be written in the form

$$i\frac{d\rho'}{dt}=[-\omega_{eff}S_n+A_0\hat{\mathcal{H}}_d^0+\hat{R}_3(t)+\hat{R}_{-3}(t)+\hat{R}_4(t)+\hat{R}_{-4}(t),\rho'], \qquad (52)$$

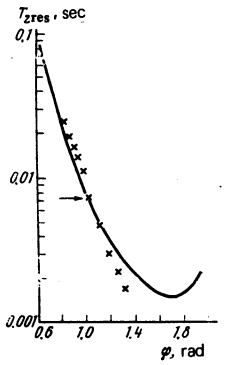

FIG. 5. Dependence of the magnetization damping time in three-spin resonance on the angle φ. The theoretical curve corresponds to expression (51) referred to the point marked by the arrow; ×—experimental points.

where $\hat{R}_3(t)$ and $\hat{R}_4(t)$ are defined by (43) and (44), while $\hat{R}_{-3}(t)$ and $\hat{R}_{-4}(t)$ are the corresponding complex-conjugate operators. The saturation equations[16] are obtained here in standard fashion[16] and take the form

$$\frac{d\alpha}{dt}=-2\pi\tau^2\Big[(9K_3^2F_3+16\tau^2K_4^2F_4)\alpha(t)+\Big(9K_3^2\frac{\pi/3\tau-\omega_{eff}}{\omega_{eff}}F_3+16\tau^2K_4^2\frac{\pi/4\tau-\omega_{eff}}{\omega_{eff}}F_4\Big)\beta(t)\Big], \tag{53}$$

$$\frac{d\beta}{dt}=-\frac{2\pi\tau^2}{A_0^2\omega_{loc}^2}\Big\{\Big[9K_3^2\omega_{eff}\Big(\frac{\pi}{3\tau}-\omega_{eff}\Big)F_3+16\tau^2K_4^2\omega_{eff}\Big(\frac{\pi}{4\tau}-\omega_{eff}\Big)F_4\Big]\times\alpha(t)+\Big[9K_3^2\Big(\frac{\pi}{3\tau}-\omega_{eff}\Big)^2F_3+16\tau^2K_4^2\Big(\frac{\pi}{4\tau}-\omega_{eff}\Big)^2F_4\Big]\beta(t)\Big\}.$$

In (53),

$$F_3=\frac{\mathrm{Sp}(\hat{G}^3_{-(\pi/\tau-3\omega_{eff})}\hat{G}^{-3}_{(\pi/\tau-3\omega_{eff})})}{\mathrm{Sp}(\hat{S}_n)^2}, \tag{54}$$

and the operators $\hat{G}^{\pm3}_{\mp(\pi/\tau-3\omega_{eff})}$ are specified by formulas (49) and (50). To obtain F_4 we must use the same formulas, with the index 3 replaced by the index 4 and with $\hat{G}^4(t)$ defined as

$$G^4(t)=[\hat{\mathcal{H}}_d^2(t),[\hat{\mathcal{H}}_d^2(t),\hat{\mathcal{H}}_d^0]],\qquad \hat{\mathcal{H}}_d^2(t)=\exp(iA_0\hat{\mathcal{H}}_d^0t)\hat{\mathcal{H}}_d^2\exp(-iA_0\hat{\mathcal{H}}_d^0t). \tag{55}$$

An investigation of Eqs. (53) shows that when two resonant processes are taken into account the magnetization attenuates in accordance with a double exponential law. The indicated resonance processes always lead to a complete vanishing of the magnetization. We can treat simultaneously the simultaneous action of any number of resonant processes.

In conclusion, the authors thank G. B. Manelis for constant attention to the work and L. N. Erofeev and B. A. Shumm for useful discussions.

APPENDIX A

To calculate expressions (33) and (34) we use formulas (12). Let $\omega_{loc}^2=\mathrm{Sp}(\hat{\mathcal{H}}_d^0)^2/\mathrm{Sp}(\hat{S}_n)^2$. Standard calculations[7] then lead to the formulas

$$\omega_{loc}^2=\frac{\mathrm{Sp}(\hat{\mathcal{H}}_d^2)^2}{\mathrm{Sp}(\hat{S}_z)^2}=\frac{3}{4}\sum_i b_{1i}^2 \tag{A.1}$$

and

$$\frac{\mathrm{Sp}(\hat{\mathcal{H}}_d^1\hat{\mathcal{H}}_d^{-1})}{\mathrm{Sp}(\hat{S}_z)^2}=\frac{9}{4}\sum_i b_{1i}^2=3\omega_{loc}^2. \tag{A.2}$$

We use next relations verified by direct calculation:

$$\hat{\mathcal{H}}_d^2=-\tfrac{1}{2}\hat{\mathcal{H}}_d^{-2}+\hat{\mathcal{H}}_d^1+\hat{\mathcal{H}}_d^{-3}. \tag{A.3}$$

Scoring (A.3) and recognizing that $\mathrm{Sp}(\hat{\mathcal{H}}_d^n)^2=\mathrm{Sp}(\hat{\mathcal{H}}_1^x)^2$, we get

$$\frac{\mathrm{Sp}(\hat{\mathcal{H}}_d^2\hat{\mathcal{H}}_d^{-2})}{\mathrm{Sp}(\hat{S}_z)^2}=\frac{3}{8}\frac{\mathrm{Sp}(\hat{\mathcal{H}}_d^2)^2}{\mathrm{Sp}(\hat{S}_z)^2}=\frac{3}{8}\omega_{loc}^2. \tag{A.4}$$

APPENDIX B

We calculate the sum $Q(\pi/3)$ [Eq. (46)]:

$$Q\Big(\frac{\pi}{3}\Big)=\sum_{m=-\infty}^{\infty}\frac{c_mb_{-1-m}}{\pi/3+m\pi}=\frac{81}{4\pi^2}\sum_{m=-\infty}^{\infty}\frac{1}{(3m+1)^2}. \tag{B.1}$$

We use next the known formula[18]

$$\frac{1}{\sin^2z}=\sum_{m=-\infty}^{\infty}\frac{1}{(z-m\pi)^2}. \tag{B.2}$$

Differentiating (B.2) with respect to z we get

$$\frac{\cos z}{\sin^2z}=-\frac{27}{\pi^3}\sum_{m=-\infty}^{\infty}\frac{1}{(3m-3z/\pi)^3}. \tag{B.3}$$

Substituting $z=-\pi/3$ in (B.3) we obtain

$$\sum_{m=-\infty}^{\infty}\frac{1}{(3m+1)^3}=\frac{4\pi^3}{81\cdot3^{1/2}}. \tag{B.4}$$

Using (B.1) and (B.4) we establish finally that

$$Q(\pi/3)=3^{-1/2}. \tag{B.5}$$

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