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Solid State Nuclear Magnetic Resonance 10 (1997) 45–51

SOLID STATE
Nuclear Magnetic
Resonance

Effects of the finite pulse widths on solid echo signals

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Received 5 March 1997; accepted 17 March 1997

Abstract

The quadrupolar and dipolar interactions of spins during the radio frequency (RF) pulses are considered. It is shown that due to these interactions the two-pulse echo signal is observed at the shifted time $t_e = \tau + t_1/2$ (t_1 = width of the first RF pulse, τ = time interval between the pulses). © 1997 Elsevier Science B.V.

Keywords: Solid echo signal; Finite widths RF pulses; Mori formalism

1. Introduction

At the present time, there have been published a great number of papers describing analysis and applications of two-pulses spin echoes in solids (solid echoes) [1–19]. Almost all of these papers consider the radio frequency (RF) pulses as delta-functions. [1] This approximation is not good enough for solids because the experimental RF pulses have values of their amplitudes comparable with NMR linewidths in solids. The effects of the finite RF pulse widths on the solid echoes were discussed first by Bloom et al. [20]. They had shown that for the nuclei with spin $I = 1$, their quadrupolar interaction during the RF pulses leads to the shift of the maximum echo signal from the time $t = \tau$. The obtained results are quite adequate for the case of the deuterium NMR, but at the present time, it is not known how the internal interactions of nuclei during the RF pulses distort the solid echo signals when nuclear spin, I, is greater than 1 and when the dipolar interactions taken into account.

In this paper, we consider the effects of quadrupolar and dipolar interactions of the spins during the RF pulses on solid echoes for general case, when nuclear spin $I > 1$ and when the interactions of multispin system are dipolar ones.

2. Spin echo after the delta-pulses

In this section, we consider formation of the two-pulse echo signals when the RF pulses are the delta-pulses. Our purpose is to briefly describe the formalism of solving the Liouville equation which was originally introduced by Mori [21,22] and later extensively developed by Dupuis, Lee, Grigolini, Sen and others ([23–30]). In the solid state NMR this formalism was also widely applied [31–41].

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[1] In the delta approximation of the RF pulse, it is assumed that $\omega_1 t_i$ = const at $t_i \to 0$ and $\omega_1 \to \infty$. ω_1 is the amplitude of the RF field and t_i is the width of pulse.

PII S0926-2040(97)00015-5

Consider an ensemble of nuclear spins in high static magnetic field B_0 ($B_0 \| OZ$). The reduced equilibrium density operator σ at $t_0 = 0$ can be written in high-temperature approximation as [42]:

$$\sigma(0) = I_Z \tag{1}$$

The first 90^0_Y pulse (Fig. 1a), RF field of which lies along the OY-axis in the rotating frame, transforms the density operator into:

$$\sigma(0^+) = I_X \tag{2}$$

After the RFD pulse end, the free evolution of the density operator is described by the Hamiltonian $\mathscr{H}_0$ and after the time τ (Fig. 1a), the density operator has the form:

$$\sigma(\tau) = \exp(-i\tau L_0)|I_X\rangle \equiv |I_X(\tau)\rangle \tag{3}$$

where:

$$L_0 = [\mathscr{H}_0, \dots] \tag{4}$$

is the Liouville superoperator [31].

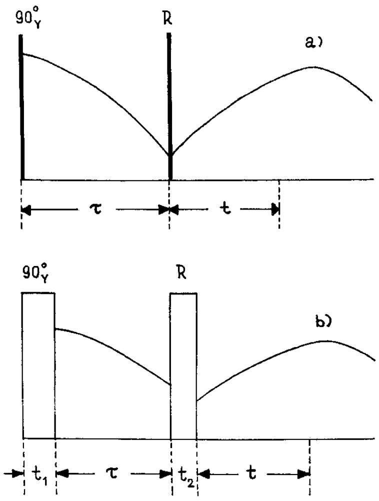

Fig. 1. Schematic representations of the two-pulse sequence: (a) for the delta-pulses, and (b) for the hard finite width pulses.

The ket-vector $|I_X(t)\rangle$ in the Liouville space may be expressed as superposition [31]:

$$|I_X(\tau)\rangle = \sum_{(n)} G_n(\tau)|n\rangle \tag{5}$$

of the ket-vectors $|n\rangle$ which form an orthogonal set:

$$\langle n|m\rangle / \langle n|n\rangle = \delta_{nm} \tag{6}$$

with inner product defined as:

$$\langle n|m\rangle = Tr(n^+ m)$$

These vectors satisfy the recurrence relation:

$$|n\rangle = L_0|n-1\rangle - \nu^2_{n-2}|n-2\rangle \tag{7}$$

where:

$$\nu^2_n = \langle n+1|n+1\rangle / \langle n|n\rangle \tag{8}$$

and:

$$\nu^2_{-1} = \nu^2_{-2} = 0$$

The functions $G_n(t)$ in Eq. (5) satisfy the system of equations [31]

$$\begin{aligned} &idG_0(t)/\mathrm{d}t = \nu^2_0\, G_1(t) \\ &\dots \\ &idG_n(t)/dt = G_{n-1}(t) + \nu^2_n\, G_{n+1}(t) \end{aligned} \tag{9}$$

From Eq. (5), we see that shape of the free induction decay (FID) after hard delta-pulse is described by the function $G_0(t)$.

If at the time τ (Fig. 1a) after the first pulse, the second delta-pulse is applied, [2] the density operator becomes:

$$\sigma(\tau^+) = \sum_{(n)} G_n(\tau)|RnR^{-1}\rangle \tag{10}$$

where unitary operator R describes the action of the second pulse [42].

After the second RF pulse, the free evolution of the density operator is again described by the Hamiltonian $\mathscr{H}_0$ and at the time t (Fig. 1a), the density operator has the form:

$$\sigma(t) = \sum_{(n)} G_n(\tau)\exp(-itL_0)|RnR^{-1}\rangle \tag{11}$$

[2] At the time τ, the cluster of closely spaced pulses-composite pulse [43,44] may be applied to the ensemble of spins. However, the combination of two or more pulses may be replaced by the one pulse [36], so we write in Eq. (10) one pulse operator R and so for the composite pulse.

The observed transient response of the ensemble of spins, two-pulse signal, is given by [42]:

$$V(t) = \langle \sigma(t)|0\rangle / \langle 0|0\rangle \tag{12}$$

Using Eqs. (5) and (11), we obtain from Eq. (12) the following general expression for the two-pulse signal:

$$V(t) = \sum_{(n,m)} G_m(t) G_n(\tau) \langle m|RnR^{-1}\rangle / \langle 0|0\rangle \tag{13}$$

If the two-pulse signal $V(t)$ contains the function $G_0(t-\tau)$, we would say that the two-pulse sequence leads to the formation of the echo signal.

From Eq. (13), it is easily to obtain the conditions at which the echo signal may be formed. Indeed, as was shown in Ref. [39], for the FID signal $G_0(t+\tau)$ we may write

$$G_0(t+\tau) = G_0(t)G_0(\tau) + \nu_0^2\, G_1(t)G_1(\tau) + \ldots + \nu_0^2\, \nu_1^2 \ldots \nu_{n-1}^2\, G_n(t)G_n(\tau) + \ldots. \tag{14}$$

Replacing $\tau \to -\tau$ and using parity properties of the functions $G_n(t)$:

$$\begin{aligned} G_{2k}(t) &= G_{2k}(-t) \\ G_{2k+1}(t) &= -G_{2k+1}(-t) \end{aligned} \tag{15}$$

for each $k = 0, 1, 2, \ldots$, we obtain:

$$G_0(t-\tau) = G_0(t)G_0(\tau) - \nu_0^2 G_1(t)G_1(\tau) + \nu_0^2\nu_1^2 G_2(t)G_2(\tau) - \ldots. \tag{16}$$

The function $G_0(t-\tau)$ has a maximum at $t=\tau$, $G_0(0)=1$.

Comparison of Eqs. (16) and (13) yields the following conditions at which the echo signal may be obtained:

$$\langle m|RnR^{-1}\rangle = 0,\ if\, m \neq n \tag{17}$$

$$\langle m|RmR^{-1}\rangle / \langle m|m\rangle = 1,\ if\, m = 2k \tag{18a}$$

$$\langle m|RmR^{-1}\rangle / \langle m|m\rangle = -1,\ if\, m = 2k+1 \tag{18b}$$

2.1. Echo signals for quadrupolar nuclei with I = 1

We apply now the obtained above results to the quadrupolar nucleus with spin $I = 1$, whose Hamiltonian has the form [42]:

$$\mathscr{H}_0 = (\omega_q/3)\left[3I_Z^2 - I(I+1)\right] \tag{19}$$

The dipolar interaction Hamiltonian of two spin system with spins $I_1 = I_2 = 1/2$ may also be written in the form Eq. (19), as was shown first by Metzger and Gaines [45].

The Liouville space of nucleus with $I = 1$ has the dimension $(2I+1)^2 = 9$ [46]. However, as shown in Appendix A, to describe the free evolution of I_X operator in the Liouville space only two vectors $|0\rangle$ and $|1\rangle$ are needed. Using Hamiltonian Eq. (19) in Eq. (9) gives:

$$G_0(\tau) = \cos(\omega_q \tau) \tag{20}$$

Then Eq. (5) reduces to:

$$|I_X(\tau)\rangle = \cos(\omega_q\tau)|0\rangle + \sin(\omega_q\tau)|\tilde{1}'\rangle \tag{21}$$

where $|\tilde{1}'\rangle = -i|1\rangle/\omega_q(\langle\tilde{1}'|\tilde{1}'\rangle/\langle 0|0\rangle = 1)$

We see that after the first 90^0_Y pulse, the ket-vector $|I_X(\tau)\rangle$ rotates with the angular velocity ω_q in the plane spanned by two vectors $|0\rangle$ and $|\tilde{1}'\rangle$ (Fig. 2a).

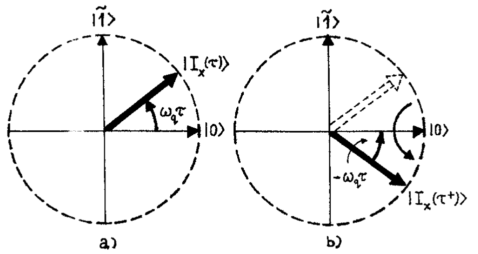

Fig. 2. Trajectories of the vector $|I_X(\tau)\rangle$ for the quadrupolar nucleus with spin $I = 1$ after the first 90^0_Y pulse (a) and after the second 90^0_X pulse (b).

According to Eqs. (17), (18a) and (18b), the two-pulse signal contains the echo signal, when:

$$\langle 0|R1R^{-1}\rangle = \langle R^{-1}0R|1\rangle = 0$$
$$\langle 0|R0R^{-1}\rangle/\langle 0|0\rangle = 1 \qquad (22)$$
$$\langle 1|R1R^{-1}\rangle/\langle 1|1\rangle = -1$$

Using the properties of the rotating operator R [36], it may be shown that only the operator:

$$R_X = \exp(\pm i\pi I_X/2) \qquad (23)$$

satisfies conditions Eq. (22). Then from Eq. (21), it follows:

$$|R_X I_X(\tau) R_X^{-1}\rangle = \cos(\omega_q\tau)|0\rangle - \sin(\omega_q\tau)|\tilde{1}\rangle \qquad (24)$$

Thus the second 90^0_X pulse rotates the ket-vector $|I_X(\tau)\rangle$ around the axis $|0\rangle$ at 180^0 (Fig. 2b). We see that 90^0_X RF pulse acts in the Liouville space as $180^0_{|0\rangle}$ pulse so that we may identify the solid echo signal with Hahn's echo signal $(90^0 - \tau - 180^0 - t)$ in the Liouville space.

2.2. Homonuclear spin system with dipolar interactions

The dipolar Hamiltonian of the homonuclear spin system has the form [42]:

$$\mathscr{H}_0 \equiv \mathscr{H}_Z = \sum_{(i,j)} b_{ij}\left[2I_Z^i I_Z^j - I_X^i I_X^j - I_Y^i I_Y^j\right] \qquad (25)$$

From Eq. (7), it follows that vector $|2k+1\rangle$ contains only 'odd' superoperators L_0^{2k+1}, L_0^{2k-1}, ... and the vector $|2k\rangle$ contains only 'even' ones L_0^{2k}, $L_0^{2(k-1)}$, ... Thus, the conditions Eqs. (17), (18a) and (18b) are fulfilled if:

$$RI_XR^{-1} = I_X \qquad (26a)$$

$$R\mathscr{H}_0R^{-1} = -\mathscr{H}_0 \qquad (26b)$$

The first condition Eq. (26a) may be satisfied only by the RF pulse of the type:

$$R_X = \exp(-i\beta I_X) \qquad (27)$$

The Hamiltonian Eq. (25) may be expressed through the components of irreducible tensor operator of the second rank T_{2m}^{ij} ($m = \pm 2, \pm 1, 0$) as [36]:

$$\mathscr{H}_0 = \sum_{(i,j)} A_{20}^{ij} T_{20}^{ij} \qquad (28)$$

This representation of $\mathscr{H}_0$ allows to analyse transformations of the $\mathscr{H}_0$ under acting of the RF pulses [36,46–48]. Using Wigner matrices [49] for description of the RF pulse effect, we conclude that the RF pulse Eq. (27) cannot fulfils the second condition Eq. (26b).

However, as we will show now, it is possible to satisfy both conditions Eqs. (17), (18a) and (18b) for some vectors $|n\rangle$ with small index n. Indeed, for the rotating operator Eq. (27) and $\beta = \pi/2$, we have:

$$R_X\mathscr{H}_0R_X^{-1} \equiv \mathscr{H}_Y = \sum_{(i,j)} b_{ij}\left[2I_Y^i I_Y^j - I_X^i I_X^j - I_Z^i I_Z^j\right] \qquad (29)$$

Using the property of dipolar Hamiltonian [37]:

$$\mathscr{H}_X + \mathscr{H}_Y + \mathscr{H}_Z = 0 \qquad (30)$$

where:

$$\mathscr{H}_X = \sum_{(i,j)} b_{ij}\left[2I_X^i I_X^j - I_Y^i I_Y^j - I_Z^i I_Z^j\right] \qquad (31)$$

and Hamiltonians $\mathscr{H}_Z$ and $\mathscr{H}_Y$ are defined by Eqs. (25) and (29), we obtain:

$$|R_X 0 R_X^{-1}\rangle = |0\rangle \qquad (32a)$$

$$|R_X 1 R_X^{-1}\rangle = -|1\rangle \qquad (32b)$$

Hence for the short enough times, τ, $t \ll \|\mathscr{H}_0\|^{-1}$ ($\|\mathscr{H}_0\| \equiv \langle \mathscr{H}_0|\mathscr{H}_0\rangle^{1/2}$), we may write:

$$V(t) = G_0(t)G_0(\tau) - \nu_0^2 G_1(t)G_1(\tau) + \ldots \approx G_0(t-\tau) \qquad (33)$$

This result is also well known [3–5,16,17,38]. However, written in the form Eq. (33), it will be important for our considerations below because in analysis of the solid echo signal we may now account in Eq. (13) only the functions $G_0(t)$ and $G_1(t)$.

3. Solid echo after the hard finite widths pulses

In this section, we consider the calculation of the solid echo $(90^0_Y - \tau - 90^0_X - t)$ signal for the case when RF pulses are not the delta-pulses. Using the Liouville superoperator formalism, we may write for the two pulses signal (Fig. 1b) the following expression:

$$V(t) = \langle I_X|\exp(-iL_0t)\exp(-iL_2t_2) \times\exp(-iL_0\tau)\exp(-iL_1t_1)|I_Z\rangle/\langle I_X|I_X\rangle \qquad (34)$$

where:

$$L_1 = [\mathscr{H}_0 - \omega_1 I_Y, \dots] \quad (35)$$

$$L_2 = [\mathscr{H}_0 - \omega_1 I_X, \dots] \quad (36)$$

and $\omega_1 = \gamma B_1$ is the amplitude of RF fields.

For $\omega_1 \gg \|\mathscr{H}_0\|$, the elements of the Hamiltonian $\mathscr{H}_0$ noncommuting with $I_{X,Y}$ may be ignored in Eqs. (35) and (36) and effective superoperators L_1 and L_2 may be written as [39]:

$$L_1 = -[\omega_1 I_Y + \mathscr{H}_Y/2, \dots] \quad (37a)$$

$$L_2 = -[\omega_1 I_X + \mathscr{H}_X/2, \dots] \quad (37b)$$

The dipolar Hamiltonians $\mathscr{H}_Y$ and $\mathscr{H}_X$ were defined by Eqs. (29) and (31). In the case of quadrupolar interactions, Hamiltonians $\mathscr{H}_X$ and $\mathscr{H}_Y$ have the form [39]:

$$H_{X,Y} = (1/3) \sum_{(i)} \omega_q^i \left[3\left(I_{X,Y}^i\right)^2 - I^i(I^i+1)\right] \quad (38)$$

Assuming $\omega_1 t_1 = \omega_1 t_2 = \pi/2$ and using Eq. (5), we obtain from Eq. (34):

$$\begin{aligned} V(t) &= G_0(t)G_0(t_1/2)G_0(\tau) \\ &\quad + \nu_0^2 G_0(t)G_1(t_1/2)G_1(\tau) \\ &\quad - G_1(t)G_0(t_1/2)G_1(\tau) \\ &\quad \times \langle 1|\exp(iL_X t_2/2)|1\rangle / \langle 0|0\rangle \\ &\quad - G_1(t)G_1(t_1/2)G_0(\tau) \\ &\quad \times \langle 1|\exp(iL_X t_2/2)|1\rangle / \langle 0|0\rangle + \dots \quad (39) \end{aligned}$$

where:

$$L_X = [\mathscr{H}_X, \dots] \quad (40)$$

In the approximation $\omega_1 \gg \|\mathscr{H}_0\|$ for which the superoperators Eqs. (37a) and (37b) were introduced, we have:

$$\|\mathscr{H}_X\| t_2 < 1$$

so to a good approximation, we may write:

$$\begin{aligned} &\langle 1|\exp(L_X t_2/2)|1\rangle / \langle 0|0\rangle \\ &\quad = [\langle 1|1\rangle + i(t_2/2)\langle 1|L_X|1\rangle \\ &\quad + \dots]/\langle 0|0\rangle \approx \langle 1|1\rangle / \langle 0|0\rangle = \nu_0^2 \quad (41) \end{aligned}$$

Inserting Eq. (41) in Eq. (39), using properties Eq. (15) of the functions $G_n(t)$ and Eq. (A2.1), obtained in Appendix B, we have:

$$\begin{aligned} V(t) &= G_0(t)G_0(-t_1/2)G_0(-\tau) \\ &\quad + \nu_0^2[G_0(t)G_1(-t_1/2)G_1(-\tau) \\ &\quad + G_1(t)G_0(-t_1/2)G_1(-\tau) \\ &\quad + G_1(t)G_1(-t_1/2)G_0(-\tau)] \\ &\quad + \dots \approx G_0(t - \tau - t_1/2) \quad (42) \end{aligned}$$

As was mentioned in Section 1, the same result was first obtained by Bloom et al. [20] for the quadrupolar nucleus with spin $I = 1$. [3] It is interesting to note that predicted time $t_e = \tau + t_1/2$ when the maximum of solid echo signal is observed does not depend on the width t_2 of the second RF pulse.

4. Conclusions

We have calculated the effects of the internal interactions of spins during the hard finite widths RF pulses on the solid echo signals. The most important result of our consideration is that for homonuclear dipolar system and for the quadrupolar nuclei with $I \geq 1$, the solid echo signal after the sequence $90_Y^0 - \tau - 90_X^0 - t$, has its maximum at $t_e = \tau + t_1/2$.

Acknowledgements

The author is indebted to Prof. Ya.I. Granovskii for the critical reading of the manuscript and helpful discussions. This research was supported by the University of Szczecin.

Appendix A

In this appendix, we show that for nucleus with spin $I = 1$ only two vectors $|0\rangle$ and $|1\rangle$ in Eq. (5) are not equal to zero.

[3] In [20], the times τ and t were measured from the beginning of the first pulse. We are measuring the time t from the end of the second pulse and τ from the end of the first pulse (Fig. 1b). Replacing in our expressions (t) with $(t - \tau - t_2)$ and (τ) with $(\tau - t_1)$, we obtain that maximum of solid echo signal has been observed at $t_e = 2\tau + t_2 - t_1/2$ [20].

For Hamiltonian Eq. (19), we have:

$$|0\rangle = I_X$$
$$|1\rangle = L_0|0\rangle = i\omega_q(I_Z I_Y + I_Y I_Z)$$
$$|2\rangle = L_0|1\rangle - \nu_0^2|0\rangle = \omega_q^2(I_Z^2 I_X + 2I_Z I_X I_Z + I_X I_Z^2 - I_X) \quad (A1.1)$$

In the case of spin $I = 1$, the dimension of the Hilbert space is $(2I+1) = 3$. Then for any linear spin operator A, we can write:

$$\begin{vmatrix} A_{11} - A & A_{10} & A_{1-1} \\ A_{01} & A_{00} - A & A_{0-1} \\ A_{-11} & A_{-10} & A_{-1-1} - A \end{vmatrix} = 0 \quad (A1.2)$$

where:

$$A_{mn} = \langle m|A|n\rangle$$

and m, $n = 1, 0{-}1$

From Eq. (A1.2) for $A = I_{X,Y,Z}$, we have:

$$I_X^3 = I_X, I_Y^3 = I_Y, I_Z^3 = I_Z \quad (A1.3)$$

Using Eq. (A1.3), it is easy to obtain:

$$(I_Z^2 I_X + 2I_Z I_X I_Z + I_X I_Z^2) = I_X \quad (A1.4)$$

Inserting Eq. (A1.4) into Eq. (A1.1), we see that state $|2\rangle$ is the zero operator:

$$|2\rangle = 0 \quad (A1.5)$$

Hence, from Eqs. (8) and (7), we obtain:

$$\nu_1^2 = \langle 2|2\rangle/\langle 1|1\rangle = 0$$
$$|3\rangle = L_0|2\rangle - \nu_1^2|1\rangle = 0$$
$$|4\rangle = L_0|3\rangle - \nu_2^2|2\rangle = 0$$
$$\ldots$$

Appendix B

In this appendix, we show that the function $G_0(t)$ may be written in the form:

$$G_0(t_1 + t_2 + t_3) = G_0(t_1)G_0(t_2)G_0(t_3) + \nu_0^2(G_0(t_1)G_1(t_2)G_1(t_3) + G_1(t_1)G_0(t_2)G_1(t_3) + G_1(t_1)G_1(t_2)G_0(t_3)) + \ldots \quad (A2.1)$$

From Eq. (5), it follows:

$$G_1(t_1 + t_2) = \langle 1|\exp(-iL_0 t_1) \times \exp(-iL_0 t_2)|0\rangle/\langle 1|1\rangle \quad (A2.2)$$

Using Eqs. (5) and (7), we have from Eq. (A2.2):

$$G_1(t_1 + t_2) = \sum_{(m)} \left[G_m(t_1)G_{m-1}(t_2) + \nu_m^2 G_m(t_1)G_{m+1}(t_2)\right] \times \langle m|m\rangle/\langle 1|1\rangle = G_0(t_1)G_1(t_2) + G_1(t_1)G_0(t_2) + \ldots \quad (A2.3)$$

Inserting Eq. (A2.3) into Eq. (14), we obtain Eq. (A2.1).

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