План статьи

Введение

1. Какую информацию об образце мы можем получить, используя метод ЯМР в низких полях?
2. О молекулярных движениях, как источнике информации, с точки зрения ЯМР-релаксометрии. О видах движения (работы Кая).
3. О влиянии времени корреляции на T1, T2 твердых и жидких фаз образцов и форму спектральной линии.
4. Корреляция структуры и свойств веществ с амплитудными и временными характеристиками спадов и параметров спектра.
5. Микрообзор того, что кто смотрит по фиду.
6. ГЭП – разброд и шатание в подходах к обработке фидов.
7. Цель работы.

Результаты и Дискуссия

1. Твердые тела. Подбор ЯМР-экспериментов с кратким описанием их особенностей.
2. Обработка данных во временной области.
3. Модели Абрагам, Гаусс, Вейбулл, Эксп. в зависимости от молекулярной динамики. Комбинации моделей в гетерофазных образцах. Модели не всегда идеально подходят – наши экспериментальные данные по разным видам твердых образцов.
4. Обработка данных в частотной области.
5. Формы спектральных линий. Второй момент. Взаимосвязь второго момента с временем поперечной релаксации.
6. Новый метод: Second moment approximation.
7. Идея.
8. Примеры применения к спадам с осцилляцией и без.
9. Сопоставление с результатами подходов Интегрирования и Усреднения.

10. Возможные будущие задачи. Может, как-то корреляцию с DQ поизучать.

11. Медленные движения молекул твердых тел эффективнее исследовать по спаду свободной индукции.

12. Есть модификации как RK, Solid Echo (самые сильные дипольные взаимодействия), MSE (вроде, слабые).

13. На каждом из упомянутых экспериментов по-разному отражается константа остаточных дипольных взаимодействий.

Analysis of FID shapes of Solids in glassy condition

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Abstract

Why is it important
What has been done
What was found
What is the general result
How it can be used in the future

1 Introduction

2 Experiment details

3 Results and discussion

3.1 FID processing in Time-Domain NMR

The recorded FID signal in NMR experiments typically consists of time, and real and imaginary components. Before analyzing the shape of FID, it undergoes preprocessing steps, including baseline subtraction, phase and frequency adjustments in the time domain. The NMR signal for baseline is recorded in the empty ampule at exactly the same conditions as the sample. Phase adjustment addresses phase errors in the time domain data. These errors can arise from imperfections in the experimental setup or sample characteristics. The process involves finding the optimal phase angle that minimizes the difference between the real component of the FID signal and the overall amplitude of the signal within the initial part (typically before 30 microseconds). By adjusting the phase angle, we aim to align the real component of the signal with its amplitude, ensuring accurate representation of the signal's characteristics.

Frequency adjustment ensures that the frequency spectrum of the signal is correctly positioned and scaled. This adjustment is crucial due to potential imperfections in the equipment or sample, which can cause the signal to deviate from its ideal state. The Fast Fourier Transform (FFT) procedure transforms the normalized time-domain signal into a frequency-domain in the form of a spectrum. The frequency axis is determined based on the time axis, following the Nyquist theorem. The spectrum is then shifted so that the maximum aligns with zero frequency, ensuring an even representation of positive and negative frequencies, i.e. symmetric spectrum. Finally, an inverse FFT (iFFT) is performed to obtain the FID signal with the corrected frequency.

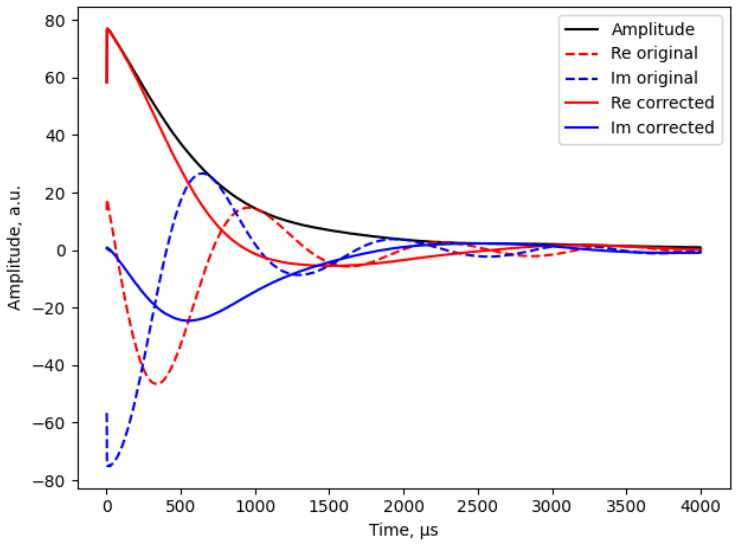

Рис. 3.1: The comparison between original (solid lines) and corrected (dashed lines) signals

To mitigate the effects of magnet inhomogeneity at short time scales (before $30\mu s$), where

the fast relaxation occurs, it is recommended to subtract the long component. Glycerol, known for its long relaxation times, serves as a suitable reference. Alternatively, any substance with a transverse relaxation time exceeding 2 milliseconds can be used. The Glycerol's reference FID is normalized, adjusted for frequency and phase in the same manner as it was done with Sample. Subsequently, the real component of the Sample is divided by the amplitude of the Glycerol FID.

To eliminate the long component, an exponential fitting of the real part of the FID corresponding to the slow decay within the time range before $200\mu s$ should be conducted and the fitted curve should be subtracted from the original data.

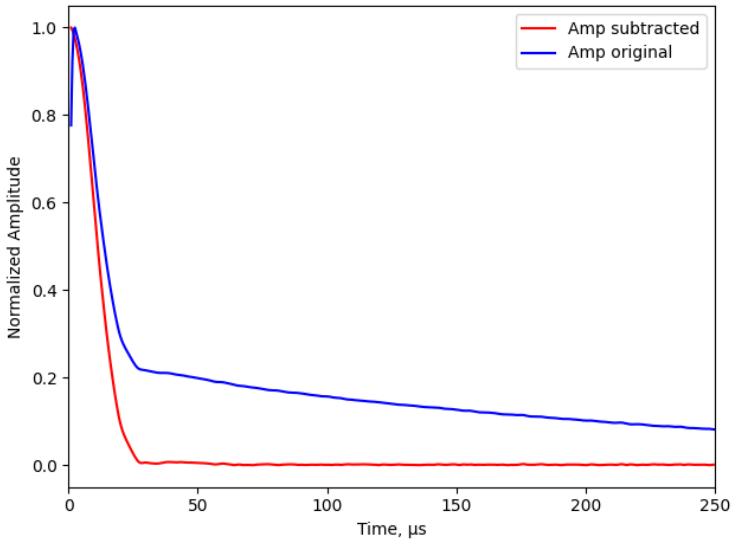

Рис. 3.2: The comparison of the original FID (red) and subtracted FID (blue)

The concluding steps in the time-domain involve apodizing the real and imaginary components of the FID, followed by zero-filling to ensure that the final FID comprises a power of 2 number of data points (2^n). This step is crucial as FFT computation is more efficient with data containing this specific number of points. Subsequently, the FFT procedure is applied, yielding the resulting spectrum in the frequency-domain.

$$f(apodization) = \exp\left(-\left[\frac{Time}{\sigma}\right]^4\right) \tag{1}$$

3.2 FID processing in Frequency-Domain NMR

The spectra derived from the FFT procedure can be manually phased using linear or polynomial functions. Subsequently, the resulting real and imaginary components of the spectra can be recalculated accordingly.

$$
\begin{aligned}
Re &= Re \cdot \cos(\phi) - Im \cdot \sin(\phi) \\
Im &= Re \cdot \sin(\phi) + Im \cdot \cos(\phi) \\
Amplitude &= \sqrt{Re^2 + Im^2}
\end{aligned}
\tag{2}
$$

Additionally, apodization of the spectrum, akin to the time-domain signal, should be performed to ensure zero amplitude at high frequencies, utilizing similar equations as in Eq. 1. Once apodized, the acquired spectrum is prepared for further analysis, such as the calculation of the second moment.

4 Conclusions

5 References