Preliminary experiments in FYS-KJM4740

Obective: Elucidation of some basic NMR concepts by practical NMR

The present NMR tutorial (room V170 in the Department of Chemistry) is an offer to students who want to learn some of the NMR concepts by a practical approach. The experimental results will be available on the home page of the course during January. All students must write up a short report based on the experimental findings.

Exercise 1

Experiments on water confined between glass beads:

Practical: Insert the sample in correct position
Acquisition mode → Sequence → Load → FID
Parameters: VT 30C, DW 0.5, RD 3s, NS 4, RG 10, SI 1K, P90 1.95. The t_d (rf pulse duration) is varied.
Start experiment: GO

a) Set t_p (0.5, 1.0, 1.5, 2.00, 2.50, 3.0, 3.50. 4.00, 4.50 and 5.00 μs) and determine the initial signal intensity I of the FID vs t_p. What is the strength B_1 (gauss) of the rf-field? Discuss your results.

b) Acquire an FID (real/imaginary) for different rf-frequency offsets (O1). Plot the FID for different O1 and discuss your results with respect to the rotating frame of reference.
Set: RD 3s, VT 30C, DW 0.5, SI 1K, P90 1.95, NS 4, RG 10). O1 is varied.

c) Fit a single exponential function and a Gaussian function to the on-resonance FID with t_p = 1.95μs. Discuss your results.

d) Acquire an FID for different repetition delays t_{RD} (= 3s, 2s, 1s and 0.5s). Plot the initial signal intensity I(0) of the FID as a function of t_{RD} and discuss your results.
Set: DW 2, SI 1K, VT 30C, P90 1.95, NS 4, RG 10, on resonance, vary t_{RD}.

Exercise 2

Inversion Recovery experiments on a series of CuSO4-solutions (Concentrations: 20mM, 10mM, 5mM, 2.5mM, 1.25mM and 0mM).

Practical: Insert the sample in correct position
Acquisition mode → Sequence → Load → INVREC
Set: **RD 1s**, **RG 1**, **VT 30, NS 4, DW 0.1s, SI 4K**
Write: .*T1* → *T1-4740* → open → OK→ write a file name.
A T1-plot will appear on the screen when the experiment is finished.
Plot T1 vs CuSO4-concentration and discuss your results.

Exercise 3

CPMG experiments on a series of CuSO4-solutions (Concentrations: 20mM, 10mM, 5mM, 2.5mM, 1.25mM and 0mM).
Practical**: Acquisition mode** → Sequence → Load → CPMGF
Set : NS 4, RD 1s, **RG 1.5**, **VT 30, P90 = 2.0** (μs), TAU **100** (μs), **NECH 4K.**
Start: GO.
When finished; Write T2.
A plot will appear on the screen. Make a "screen dump" (Press "Print Scrn" and paste the image into "Paint"). You will use the value of T_2 in the report.
Plot T2 vs CuSO4-concentration and discuss your results.

Write a report on your experimental work (include relevant Figures)!

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UiO, January 2015

Practical NMR

A Simplified User Manual for the Maran Ultra NMR Instrument (Non-expert User)

Initiate experiment

1. Insert a sample in correct position
2. **Acquisition mode**: sequence → load → FID.EXE
3. Set RD to ≈ 5·T1, i.e., **RD 1.0s**, **P90 = 2.15** (μs), **VT 30** (^{0}C) and **RG 1, DW 0.1, SI 4K.**

Optimizing relevant parameters

4. **Command** → ***Auto O1*** (wait)
5. **Command** → ***Auto RG*** (wait). Use this value of RG throughout if not otherwise stated in the text.
6. **Command** → ***Auto P90*** (wait)

T_1 measurement

7. Insert the sample in correct position
8. **Acquisition mode** → Sequence → Load → INVREC
9. Set: **RD 1s**, **RG 1**, **VT 30** and **NS 4, DW 0.1s, SI 4K**
10. **Write: *.T1* → *T1-4740*** → open → OK→ write a file name.
11. A T1-plot will appear on the screen when the experiment is finished. Make a "screen dump" (Press "Print Scrn" and paste the image into "Paint"). You will use the value of T_1 in the report.

T2-measurement

12. Insert the sample in correct position
13. **Acquisition mode** → Sequence → Load → CPMGF
14. Set: **RD 1s**, **RG 1**, **VT 30** and **P90 = 2.15** (μs), **tau 100** (μs), **NECH 4K**
15. **Write: GO**
16. **Store File:**
17. Write T2. A plot will appear on the screen. Make a "screen dump" (Press "Print Scrn" and paste the image into "Paint"). You will use the value of T_2 in the report.

(Miscellaneous)

Calculations using the NMR program WinFit

18. Open WinFit: C:\ProgramFiles\Resonance\WinFit\nmrfit (double click)
19. Retrieve a file: File → Open → Select file → data →
    (Select Fit Options) → number of exponents/DC-offset (if possible) →
    Auto Initialize → Fit → Data (Read out values) or print, i.e.; File → Print → ….