SIKKER JOBBANALYSE

Navn på enhet: **Fysikalsk kjemi – reaktivitet og dynamikk**
Navn på deltakerne:
Navn på arbeidsoppgaven som sikker jobbanalysen gjelder: **Time-Resolved NMR (Maran Ultra)**
Dato: Oktober 2013

Deloppgaver	**Hva som kan føre til en uønsket hendelse**	**Mulige forebyggende tiltak**
Metallgjenstander	Metaller/magnetiske materialer påvirker magnetfeltet. I verste fall kan de ødelegge magneten.	• Legg fra deg metallgjenstander utenfor NMR laben.
Undersøkelser av ukjente prøver	Prøver kan potensielt være toksiske/helseskadelige	• Alle prøver skal beskrives i forhold til helserisiko eller annen fare i eget skjema, hvis relevant. • Advarsel i lab-journal • Farlige prøver skal ikke ligge framme på labben annet enn når målinger på prøvene utføres. • Prøvene skal oppbevares på sikker måte.
Oppvarming/avkjøling	Korrodere prøveholder/spole	• Benytt nitrogengass når t > 100C • Benytt flytende nitrogen/nitrogen-gass når t < 0C. • Benytt «batch»-programmer når du utfører høytemperaturmålinger over natta (husk å avslutte batch-programmet med å redusere temperaturen til romtemperatur). • Vær alltid til stede når du utfører lavtemperaturforsøk (t < 0C)
Bruk av flytende N2	Frostskader ved søl	• Alle setter seg inn i felles KI dokument (SJA/SOP) for håndtering av flytende nitrogen.

SOP for the Maran Ultra Time-Resolved NMR instrument

• **Training required:** No one is allowed to use the instruments before having received appropriate training. The lowest operating level requires that one of the staff (Eddy W. Hansen or any other certified person (Master/PhD) in the group is present during start-up of the instrument to confirm that everything is according to good laboratory practice and safety requirements before start-up.

User Guide – Maran Ultra. The Instrument User Guide (attachment) is part of the User Training. All users should have their own copy. The User guide is not meant to be exhaustive neither for SOP nor as a technical guide, but serves as an "up-and-go" document. A more detailed and complete description of the experimental procedures can be found in the manual "Maran Ultra" (in the lab).

1. Operating the Maran Ultra instrument

For every application a Safe Job Analysis (Sikker Jobb Analyse) is required. This means that every sample brought into the lab should be checked for health hazards, and all experimental set-ups needs to be evaluated for potential risks.
Check Log Book before starting an experiment.
When in doubt always call one of the responsible staff (Eddy W. Hansen） before starting the experiments.

2. Instrument Log Book

Note that every operation in the NMR laboratory must be documented in the Instrument Log Book. A minimum requirement is that the following is specified in the Log Book:
1. Date and time, and name of operator

2. Type of sample / sample ID, and specification of health and or hazard risks involved in handling the samples

3. Optical set-up of the FTIR

4. A summary of the state of the system as observed on start-up and by the end of the session.

Attachment

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

M: \KJM-MEF4010\2013\Maran User manual – simplified alternative - 2013.doc

Before initiating new experiments do the following

1. Insert sample (ca. 10 mm height) in correct position
2. Choose "acquisition mode"
3. Sequence → load → FID.EXE → open
4. Set RD to approximately 5*T1 (7s)
5. Set P90 (2.15 (μs)) and RG (1)

Optimize Receiver Gain (RG), Offset (O1) and Pulse length (P90)

1. Commands → Auto O1 (wait)
2. Commands → Auto RG (wait)
3. Commands Auto P90 (wait)

Acquiring an FID

1. Sequence → load → FID.EXE → Open
2. GO

Store the FID

1. File → Save As → (file name)

T_1 Measurement

1. Acquisition → Sequence → Load → INVREC.EXE → Open
2. .T1 → τ-list (file name) → Open → OK→ File name (for storing T1 data) → save
3. A plot will appear on the display at the end of the experiment", press; OK.

Calculating T_1

1. Process → T1 → Enter file name (T1 data) → Yes → T1 is calculated and presented on the display.
2. Alternatively; WinFit → File → Open → Select file → data → Select Fit Option → Auto Initialize → Fit → Data (Read out values), or print, i.e.; File → Print
3. You may also read the T1 data within an excel spread sheet; Retrieve excel → File → Open → File name (*.INT)

T_2 measurement

1. Acquisition → Sequence → Load → CPMG.EXE → Open
2. Check that SI = 1
3. TAU (Set a τ-value in microseconds)
4. NECH (number of echoes), which must be chosen according to the expected T2 and the TAU (NECH*TAU*2 ≈ 5*T2)
5. GO
6. File → Save As → (file name…..)
7. Store as *.xls file (File → Export → file name)

Calculating T_2

1. After acquisition, type; T2 (T2 based on a single exponential fit).
2. File → Save As → M:\..... (file name)
3. Alternative; WinFit → File → Open → Select file → data → Select Fit Option → Auto Initialize → Fit → Data (Read out values), or print, i.e.; File → Print

PFG NMR - Initial

1. Acquisition → Sequence → Load → CPMGB13.EXE → Open
2. Set: SI 512, DW 0.1, NS 8, RD 7000000, DEAD1 3, DEAD2 5, D1 200, D2 600, D3 300, G3 25, D7 100, G1 100, G2 -100, RG 5, C1 4, C2 2, FW 1E6
3. Commands → Auto RG
4. Commands → Auto O1 (wait)
5. Commands Auto P90 (wait)

Measurement

1. PFG_DIFF_AT_EWH
2. You are asked for a list of gradients; write:
3. f7_list → Open → OK
4. g7_list → Open → OK
   Experiment starts)[1)]
5. File → Save As → File name.
6. Change D7 and repeat the measurement by starting from point 1 above.

Calculation

There exists an Excel spreadsheet which you may use for this purpose

Alternative PFG NMR experiment

Initial

1. Acquisition → Sequence → Load → CPMGB13.EXE → Open
2. Set: SI 512, DW 0.1, NS 8, RD 7000000, DEAD1 3, DEAD2 5, D1 200, D2 600, D3 300, G3 25, D7 100, G1 100, G2 -100, RG 5, C1 4, C2 2, FW 1E6
3. Commands → Auto RG
4. Commands → Auto O1 (wait)
5. Commands Auto P90 (wait)

Measurement

1. Use a batch-file denoted: ***CPMGB13-variableG***
2. Set: SI 512, DW 0.1, NS 8, RD 7000000, DEAD1 3, DEAD2 5, D1 200, D2 600, D3 300, G3 25, D7 100, G1 100, G2 -100, RG 5, C1 4, C2 2, FW 1E6
3. As can be noticed from the batch file (see next page) the value of the two pulse gradient strengths G1 and G2 (G1 = - G2) are varied during the 15 experiments. In particular, the parameter denoted D7 is kept fixed (= 1000) but must be changed for each different batch-run in order to probe the diffusion time dependence of the diffusivity.
4. Start the experiment
5. Write: .CPMGB13-variableG
6. *Enter* (start experiment)
7. During the experiment, the intensity data are stored in a file denoted: C:\name1\name2\Name3-G

Calculation

After the experiment is performed you must load each set of data files (same D7) and analyze them.

Batch file: CPMGB13-variableG

```
~AMODE
LOAD CPMGb13
VT 35
C1 4
C2 2
d1 125
d2 600
d3 500
DW 0.1
SI 512
RD 7000000
D7 1000
G1  1000
G2 -1000
G3 25
ns 16
GO
wr C:\KJM-MENA4010\Miscellaneous\GK-24-1000
EX C:\KJM-MENA4010\Miscellaneous\GK-24-1000 E

~AMODE
G1 2000
G2 -2000
GO
wr C:\KJM-MENA4010\Miscellaneous\GK-24-2000
EX C:\KJM-MENA4010\Miscellaneous\GK-24-2000 E

~AMODE
G1 3000
G2 -3000
GO
wr C:\KJM-MENA4010\Miscellaneous\GK-24-3000
EX C:\KJM-MENA4010\Miscellaneous\GK-24-3000 E

~AMODE
G1 4000
G2 -4000
GO
wr C:\KJM-MENA4010\Miscellaneous\GK-24-4000
EX C:\KJM-MENA4010\Miscellaneous\GK-24-4000 E

~AMODE
G1 5000
G2 -5000
GO
wr C:\KJM-MENA4010\Miscellaneous\GK-24-5000
EX C:\KJM-MENA4010\Miscellaneous\GK-24-5000 E

~AMODE
G1 6000
G2 -6000
GO
wr C:\KJM-MENA4010\Miscellaneous\GK-24-6000
EX C:\KJM-MENA4010\Miscellaneous\GK-24-6000 E

~AMODE
G1 7000
G2 -7000
GO
wr C:\KJM-MENA4010\Miscellaneous\GK-24-7000
EX C:\KJM-MENA4010\Miscellaneous\GK-24-7000 E

~AMODE
G1 8000
G2 -8000
GO
wr C:\KJM-MENA4010\Miscellaneous\GK-24-8000
EX C:\KJM-MENA4010\Miscellaneous\GK-24-8000 E

~AMODE
G1 9000
G2 -9000
GO
wr C:\KJM-MENA4010\Miscellaneous\GK-24-9000
EX C:\KJM-MENA4010\Miscellaneous\GK-24-9000 E

~AMODE
G1 10000
G2 -10000
GO
wr C:\KJM-MENA4010\Miscellaneous\GK-24-10000
EX C:\KJM-MENA4010\Miscellaneous\GK-24-10000 E

~AMODE
G1 11000
G2 -11000
GO
wr C:\KJM-MENA4010\Miscellaneous\GK-24-11000
EX C:\KJM-MENA4010\Miscellaneous\GK-24-11000 E

~AMODE
G1 12000
G2 -12000
GO
wr C:\KJM-MENA4010\Miscellaneous\GK-24-12000
EX C:\KJM-MENA4010\Miscellaneous\GK-24-12000 E

~AMODE
G1 13000
G2 -13000
GO
wr C:\KJM-MENA4010\Miscellaneous\GK-24-13000
EX C:\KJM-MENA4010\Miscellaneous\GK-24-13000 E

~AMODE
G1 14000
G2 -14000
GO
wr C:\KJM-MENA4010\Miscellaneous\GK-24-14000
EX C:\KJM-MENA4010\Miscellaneous\GK-24-14000 E

~AMODE
G1 15000
G2 -15000
GO
wr C:\KJM-MENA4010\Miscellaneous\GK-24-15000
EX C:\KJM-MENA4010\Miscellaneous\GK-24-15000 E
```