

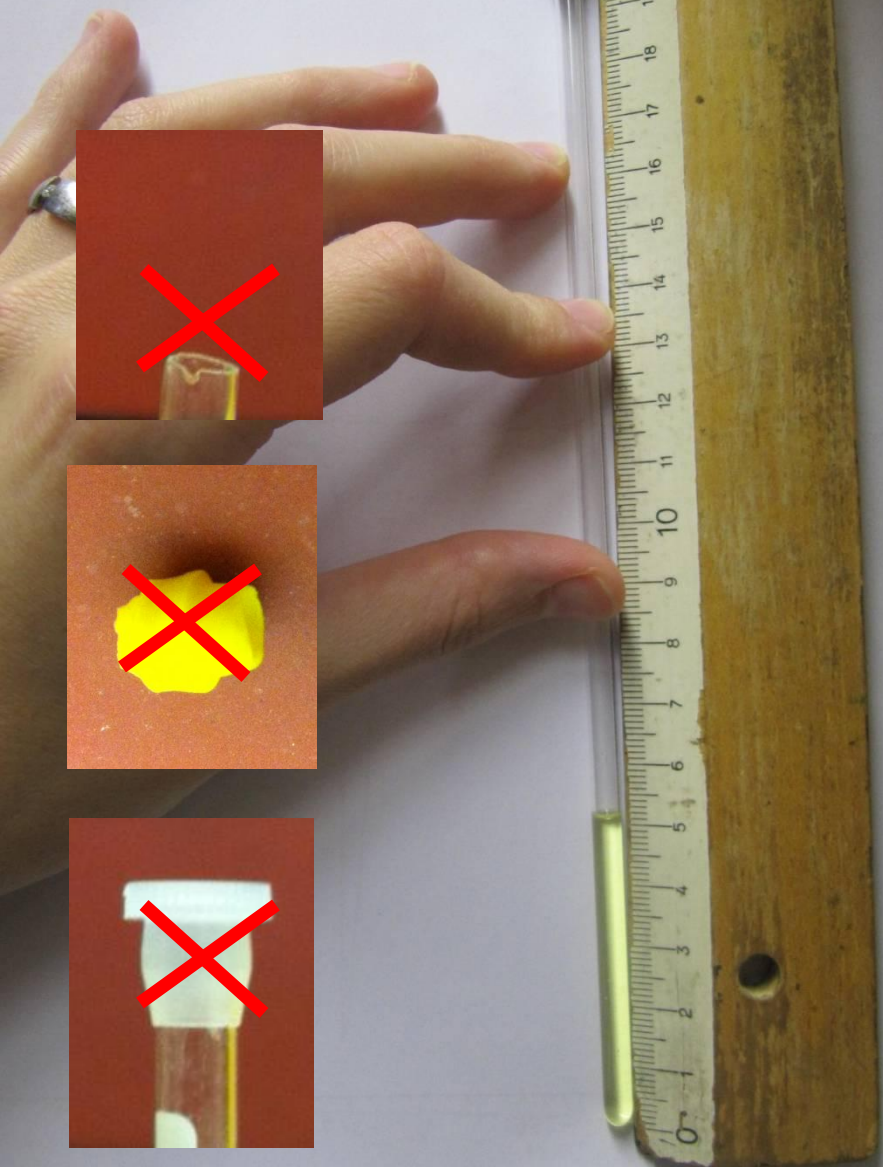


**The 300 MHz
Spectrometer
“NMR-Praktikum“**

M. John

February 2012

500
max
krocher



Sample

Standard NMR tubes only

Diameter: 5 mm

**Length: 18 - 24 cm
(7 - 9 inch)**

**No broken tubes and caps
Cap must sit firmly on tube**

**Minimum
Filling height: 4 cm**

Deuterated solvents only

**Homogenous solutions only
(no solids, no bubbles,
no two-phase mixtures)**

Label

Colour code

Name

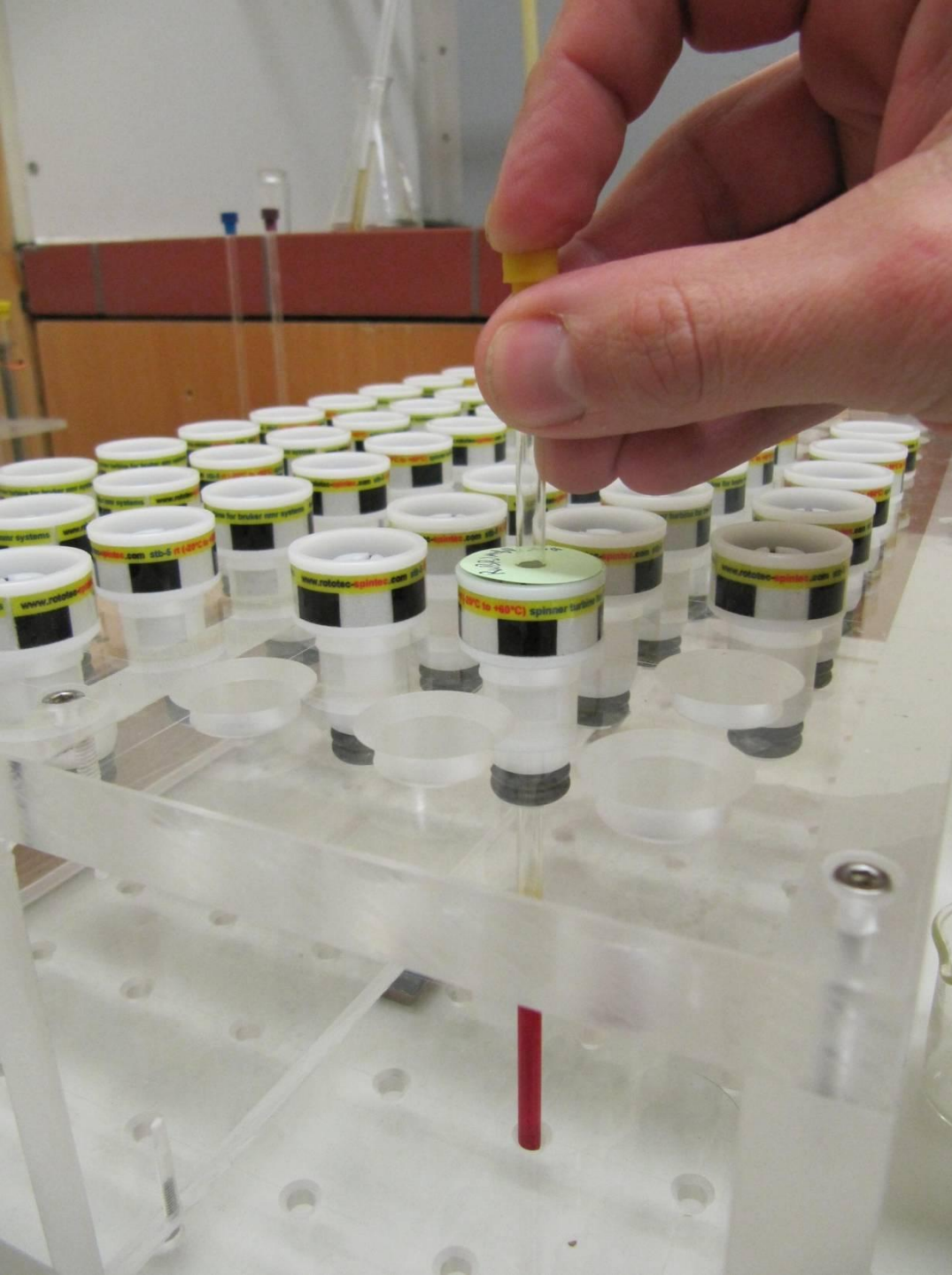
Personal sample number

Date

Use paper label only once

Any unlabelled, damaged or wrong tubes will be removed and discharged without further notice!





Spinner

Clean sample

**Push sample all the way
down in the spinner rack**

**Label must lie flat and firmly
on the spinner**

Avoid touching the spinner



Autosampler

Clean tube again!

**Use next free holder
(usually on the left side close
to magnet)**

**Inner circle: holder 1 - 60
Outer circle: holder 61- 120**



Hol...	Type	Status	Name	No.	Solvent	Experiment	Par	Title / Orig	Pri	Time	User
1		Available									
2		Available									
3		Automation Locked									
4											
5											
6											
7											
8											
9											
10											
11		Available									
12		Available									
13		Available									
14		Available									
15		Running	s_meyer300c	3	DMSO	13C					
16		Available									
17		Available									
18		Available									

Automation Locked

To unlock the automation window click on the change user button at the right hand side of the Automation Setup window.

ICON-NMR: Identify User

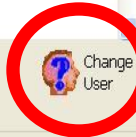
Help on logging in

User ID	User's Full Name
acf	ACF-Praktikum
meyer	Ak Meyer
nmrservice	AC NMRservice
nmrsu	NMR SuperUser
oca	OC-Analysenkurs
ocf	OCF-Praktikum
roesky	Ak Roesky
stalke	Ak Stalke
wenger	Ak Wenger

User ID:

OK Lock Icon-NMR

**click
"change user"**



**log in
with your
user ID**

Preceding Experiments

#	Date	Holder	Name	No.	Experiment	Lo
1	2009-12-03 14:56:04	15	s_meyer300c	3	13C	✓

Proc	User	Disk	Title / Orig	Remarks
	nmrservice	C:\Bruker\	TOPSPIN	

File Run Holder View Find Parameters Options Tools Help Processing started

Holder	Type	Status	Name	No.	Solvent	Experiment	Par	Title / Orig	Pri	Time	User
5		Available									
6		Available									
7		Available									
8		Available									
9		Available									
10		Available									
11		Available									
12		Available									
13		Available									
14		Available									
15		5Queued									
		Finished	s_meyer300c	3	DMSO	13C				00:32:53	nmrservice
		Queued	s_meyer300hsqc	5	DMSO	HSQC				00:14:37	nmrservice
		Queued	s_meyer300p	5	DMSO	19F				00:00:40	nmrservice
		Queued	s_meyer300f	5	DMSO	31P				00:00:48	nmrservice
		Queued	s_meyer300h	5	DMSO	1H				00:01:54	nmrservice
16		Available									
		Available	mueller300h	257	C6D6	b					nmrservice
17		Available									
18		Available									
19		Available									

select appropriate holder

here you can enter a title

N 1H	standard 1H nmr
n 13C	standard 13C 1H decoupling
N 19F	standard 19F 1H decoupling
N 31P	standard 31P 1H decoupling
n COSY	standard 1H-COSY
n HSQC	standard 13C-HSQC
n HMBC	standard 13C-HMBC

enter data file, personal sample no., solvent and experiment

username300experiment (e.g. mueller300h, mueller300hsqc)

#	Date	Holder	Name	No.	Experiment	Load	ATM	Rotation	Lock	Shim	Acq	Proc	User	Disk	Title / Orig	Remarks
1	2009-12-03 14:56:04	15	s_meyer300c	3	13C	✓	✓	✓	✓	✓	✓	✓	nmrservice	C:\Bruker\TOPSPIN		sref: no peak found default calibration done

Search Preceding include previous runs

Busy until: Thu 15:40 Day Experiments: 00:03 Night Experiments: 00:15 User: nmrservice



Holder	Type	Status	Name	No.	Solvent	Experiment	Par	Title / Orig	Pri	Time	User
12		Available									
13		Available									
14		Available									
15		5Queued									
		Finished	s_meyer300c	3	DMSO	13C				00:32:53	nmrservice
		Queued	s_meyer300hsqc	5	DMSO	HSQC				00:14:37	nmrservice
		Queued	s_meyer300p	5	DMSO	19F				00:00:40	nmrservice
		Queued	s_meyer300f	5	DMSO	31P				00:00:48	nmrservice
		Queued	s_meyer300h	5	DMSO	1H				00:01:54	nmrservice
16		1Available									
		Available	mueller300h	257	C6D6	b	N 1H			00:01:54	nmrservice
17		Available									
18		Available									
19		Available									
20		Available									
21		Available									
22		Available									
23		Available									
24		Available									
25		Available									
26		Available									

Submit all selected experiments for run

Submit Cancel Edit Delete Add Copy 1

Change User

Submit all selected experiments for run

Preceding Experiments

#	Date	Holder	Name	No.	Experiment	Load	ATM	Rotation	Lock	Shim	Acq	Proc	User	Disk	Title / Orig	Remarks
1	2009-12-03 14:56:04	15	s_meyer300c	3	13C	✓	✓	✓	✓	✓	✓	✓	nmrservice	C:\Bruker\TOPSPIN		sref: no peak found default calibration done

submit

**if needed
add further
experiment for
same sample**

log off

Holder	Type	Status	Name	No.	Solvent	Experiment	Par	Title / Orig	Pri	Time	User
12	Available	Available									
13	Available	Available									
14	Available	Available									
15	5Queued	Finished	s_meyer300c	3	DMSO	13C				00:00:37	nmrsvic
		Queued	s_meyer300hsqc	5	DMSO	HSQC				00:00:40	nmrsvic
		Queued	s_meyer300p	5	DMSO	19F				00:00:48	nmrsvic
		Queued	s_meyer300f	5	DMSO	31P				00:01:54	nmrsvic
		Queued	s_meyer300h	5	DMSO	1H					
16	2Queued	Queued	mueller300h	257	C6D6	1H				00:01:54	nmrsvic
	Available	Available	mueller300c	257	C6D6						nmrsvic



experiments > 5 min (¹³C, ²⁹Si, DEPT, 2D) will be run overnight

some accounts have priority option

- N 1H standard 1H nmr
- n 13C standard 13C 1H decoupling
- N 19F standard 19F 1H decoupling
- N 31P standard 31P 1H decoupling
- n COSY standard 1H-COSY
- n HSQC standard 13C-HSQC
- n HMBC standard 13C-HMBC

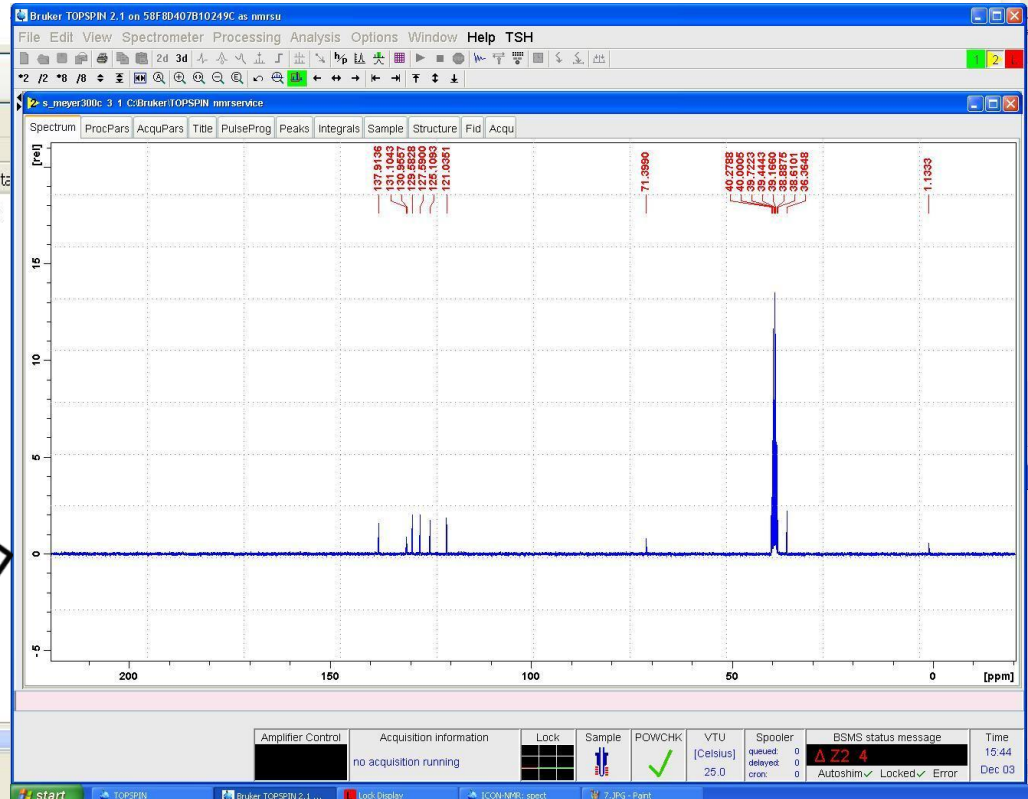
Submit Cancel Edit Delete Add 1

Preceding Experiments

#	Date	Holder	Name	No.	Experiment	Load	ATM	Rot
1	2009-12-03 14:56:04	15	s_meyer300c	3	13C	✓	✓	✓

↑ finished experiments appear here

double click to display spectrum



ICON-NMR on 58F8D407B10249C: spect - Windows Internet Explorer

http://10.76.64.151:8015/.../template-automation.htm

Automation - Running - Busy until : Wed 12:30 - Day Experiments : 00:02 - Night Experiments : 06:28

BRUKER icon web

Automation - Running - Busy until : Wed 12:30 - Day Experiments : 00:02 - Night Experiments : 06:28

Help
Logoff

Logged in as acf

Instrument Name
58F8D407B10249C: spect

check the status of your measurements online (within gönet)

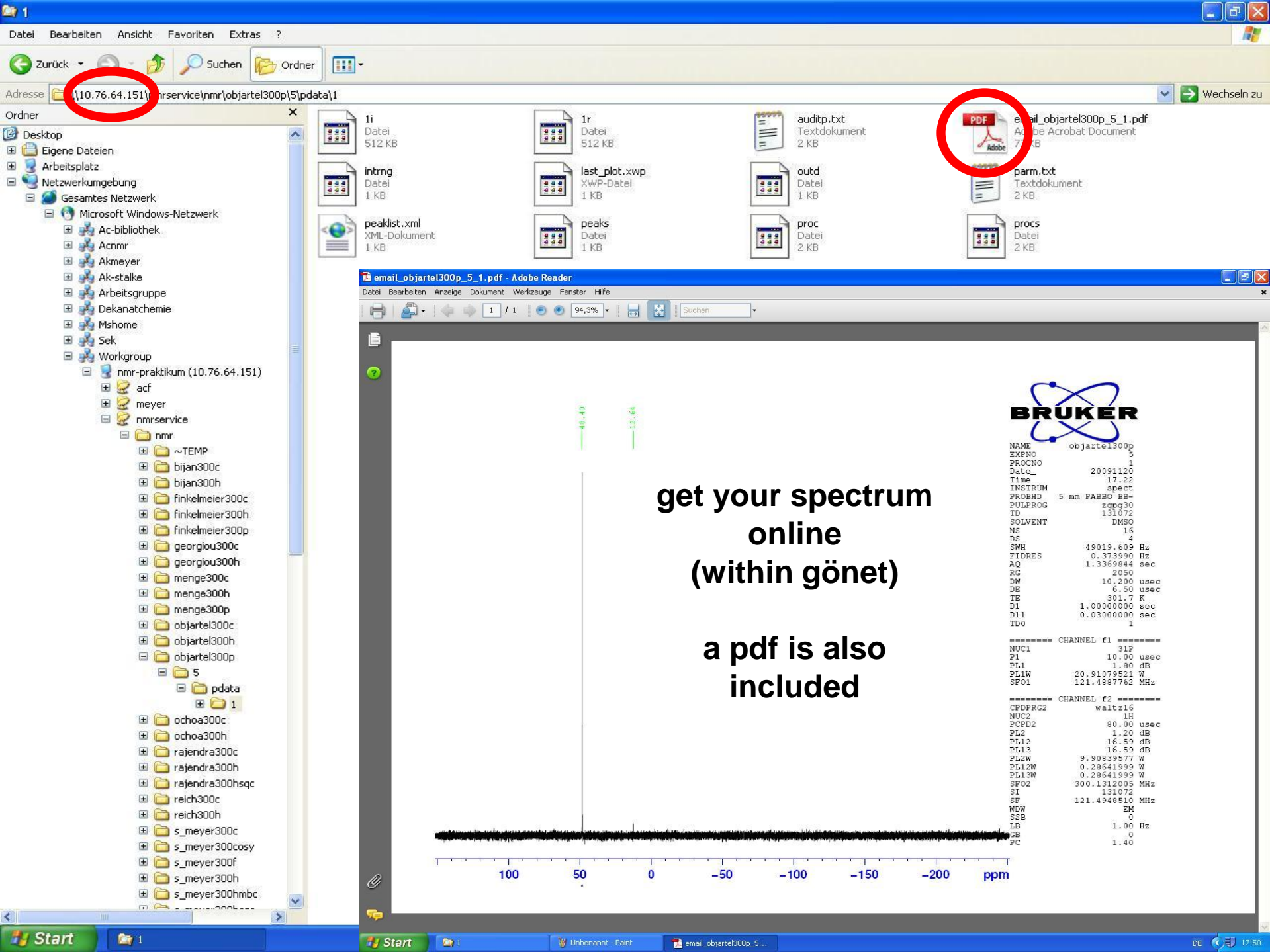
ATM Running	Lock Field	Shim	Receiver Gain	Acquire Data	Process Data						
<input type="checkbox"/> 8		Completed	ab300h	81	CDCI3	1H		AB20-1		00:01:54	wenger
<input type="checkbox"/> 9		Completed	sfp300h	51	CD3CN_SPE	1H		SFP 21A Zugabe 160uL BF4		00:01:54	clever
<input type="checkbox"/> 10		Completed	sfp300h	52	CD3CN	1H		SFP 23 Pt-CD3CN		00:01:54	clever
<input type="checkbox"/> 11		Completed	sfp300h	53	CD3CN	1H		SFP 05 Pt-CD3CN		00:01:54	clever
<input type="checkbox"/> 12		Completed	sfp300h	54	DMSO	1H		SFP 27 BF4-DMSO		00:01:54	clever
<input type="checkbox"/> 13		Completed	sfp300h	55	DMSO	1H		SFP 28 NO3-DMSO		00:01:54	clever
<input type="checkbox"/> 14		Completed	hschmidt	6	CDCI3	1H		Stufe3		00:01:54	ocf
<input type="checkbox"/> 15		Queued	walli300h	104	D2O	1H		HN01-a		00:01:54	meyer
<input type="checkbox"/> 15		Queued	walli300c	104	D2O	13C{1H}		HN01-a		00:14:08	meyer
<input type="checkbox"/> 16		Completed	walli300h	105	D2O	1H		HN01-b		00:01:54	meyer

Date	Time	Holder Name	No.	Experiment Load	ATM	Rotation	Lock	Shim	Acq	Proc	User	Title
2011-11-23	12:24:28	42 MaassJW	15	1H							stalke	Carben_neu
2011-11-23	12:18:21	27 sietzen300h	168	1H	✓	✓	✓	✓	✓	✓	siewert	MS90_2
2011-11-23	12:12:54	25 Steinert300h	252	1H	✓	✓	✓	✓	✓	✓	meyer	MS147
2011-11-23	12:07:37	23 kuegler300h	180	1H	✓	✓	X	✓	✓	✓	siewert	MK 117-1
2011-11-23	12:01:06	21 klawitter300h	260	1H	✓	✓	✓	✓	✓	✓	meyer	IK197-1
2011-11-23	11:55:22	20 klawitter300h	259	1H	✓	✓	✓	✓	✓	✓	meyer	IK170
2011-11-23	11:49:30	28 JW2	9	1H	✓	✓	✓	✓	✓	✓	ocf	
2011-11-23	11:44:07	19 schneider300h	195	1H	✓	✓	✓	✓	✓	✓	meyer	BS447-2 H2L7
2011-11-23	11:38:00	18 Steinert300h	251	1H	✓	✓	✓	✓	✓	✓	meyer	MS152

Version: 4.2.5
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Start TOPSPIN Bruker TOPSPIN 2.1 ... ICON-NMR on 58F8D... DE 12:28

File Explorer window showing a network path `\\10.76.64.151\nmr\objartel300p\5\data\1`. A file named `email_objartel300p_5_1.pdf` is circled in red. The file is opened in Adobe Reader.



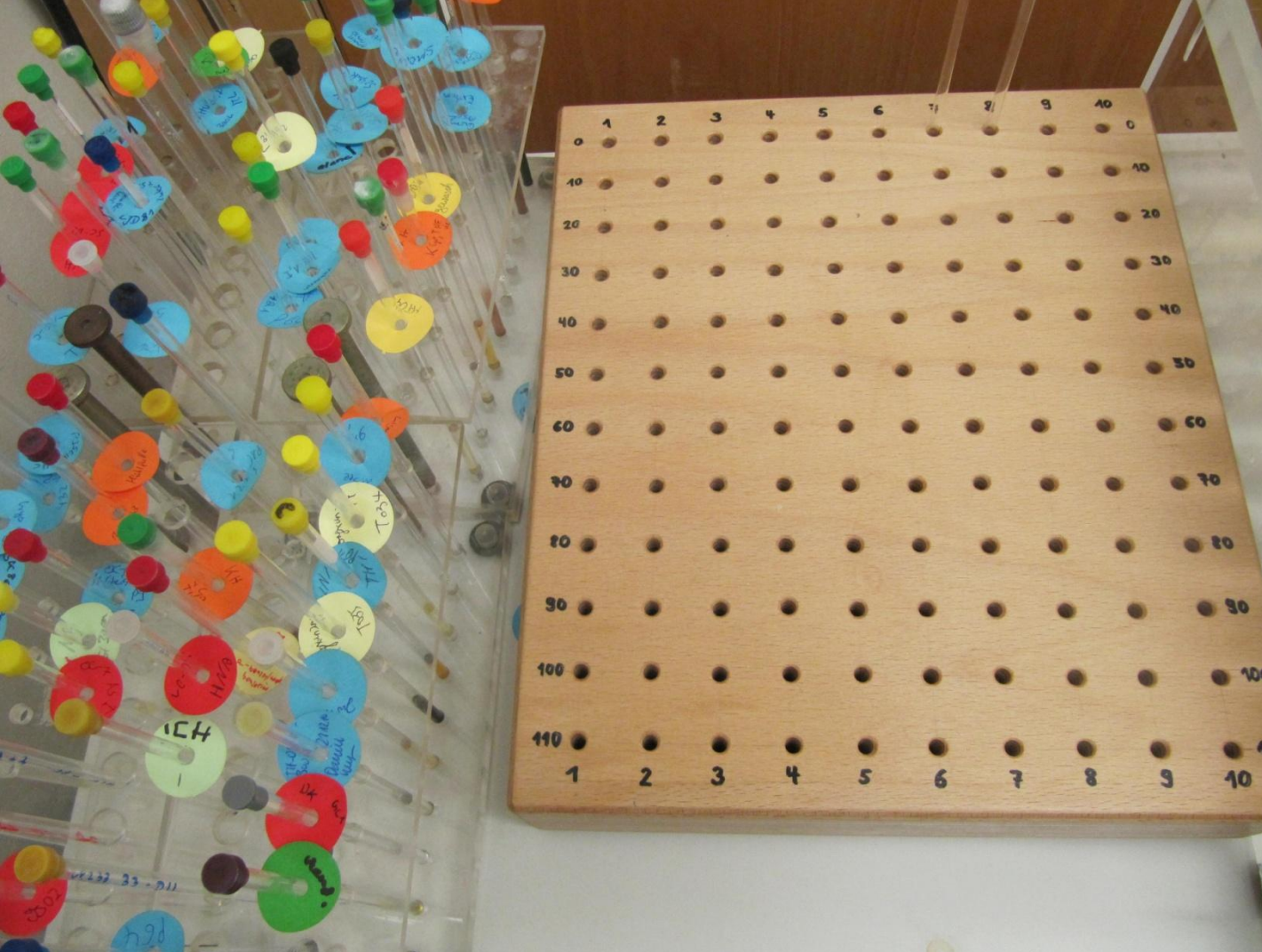
**get your spectrum
online
(within gönet)**

**a pdf is also
included**

NAME	objartel300p
EXPNO	5
PROCNO	1
Date_	20091120
Time	17.22
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	131072
SOLVENT	DMSO
NS	16
DS	4
SWH	49019.609 Hz
FIDRES	0.373990 Hz
AQ	1.3369844 sec
RG	2050
DW	10.200 usec
DE	6.50 usec
TE	301.7 K
D1	1.00000000 sec
D11	0.03000000 sec
TD0	1

===== CHANNEL f1 =====	
NUC1	31P
PL	10.00 usec
PL1	1.80 dB
PL1W	20.91079521 W
SFO1	121.4887762 MHz

===== CHANNEL f2 =====	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	1.20 dB
PL12	16.59 dB
PL13	16.59 dB
PL2W	9.90839577 W
PL12W	0.28641999 W
PL13W	0.28641999 W
SFO2	300.1312095 MHz
SI	131072
SF	121.4948510 MHz
WDW	EM
SFB	0
IS	3.00 Hz
CB	0
PC	1.40



Pick up

finished samples will be placed in timber pick up rack under holder no. (early morning)

Samples not picked up will end up in the storage rack on the left

Have fun!