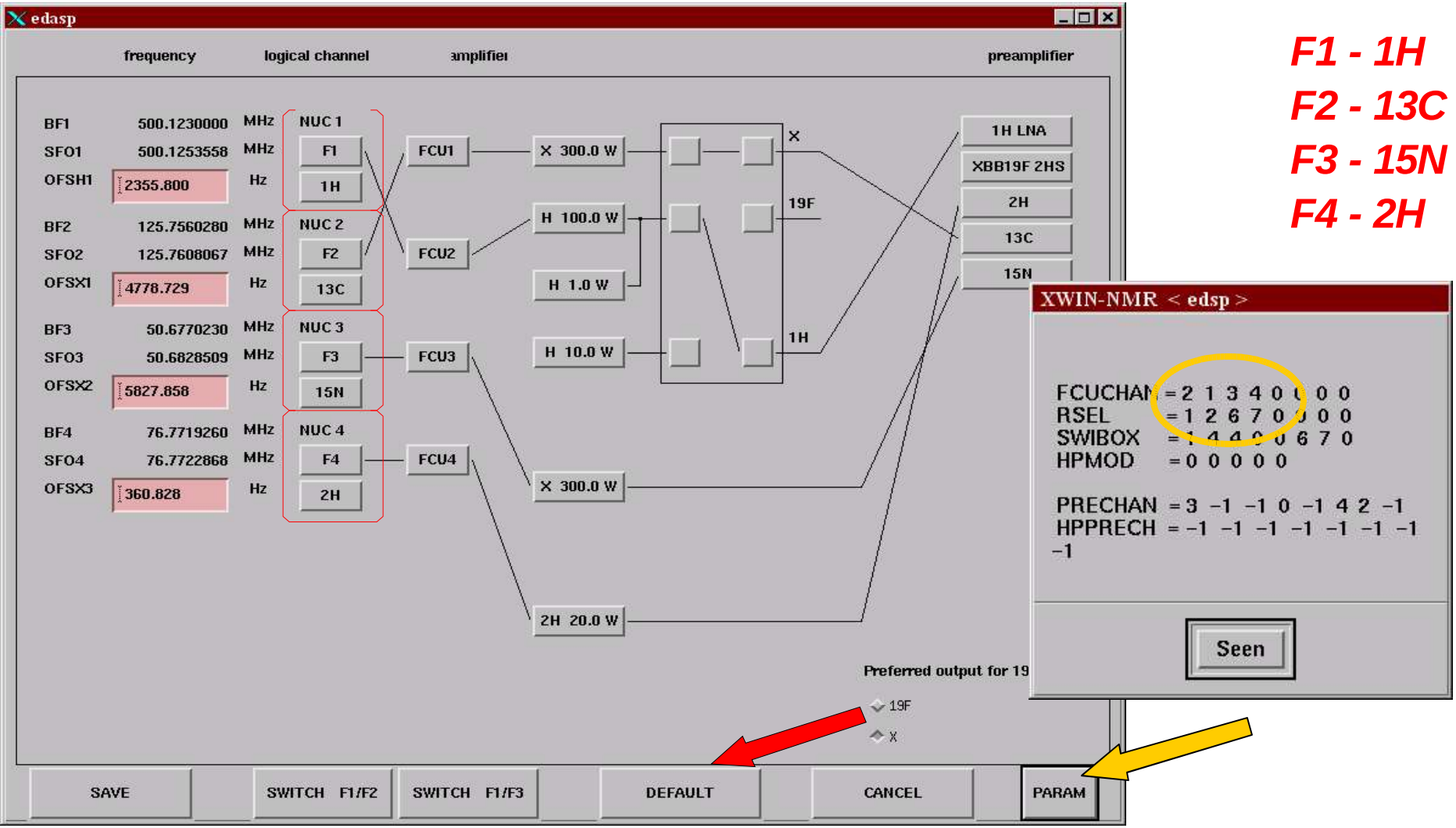
Pulse calibration

RF-routing for triple resonance, 2H-dec.



F1 - 1H
F2 - 13C
F3 - 15N
F4 - 2H

New: change HPPR routing with edasp setpreamp, cf

Pulse calibration: Check List

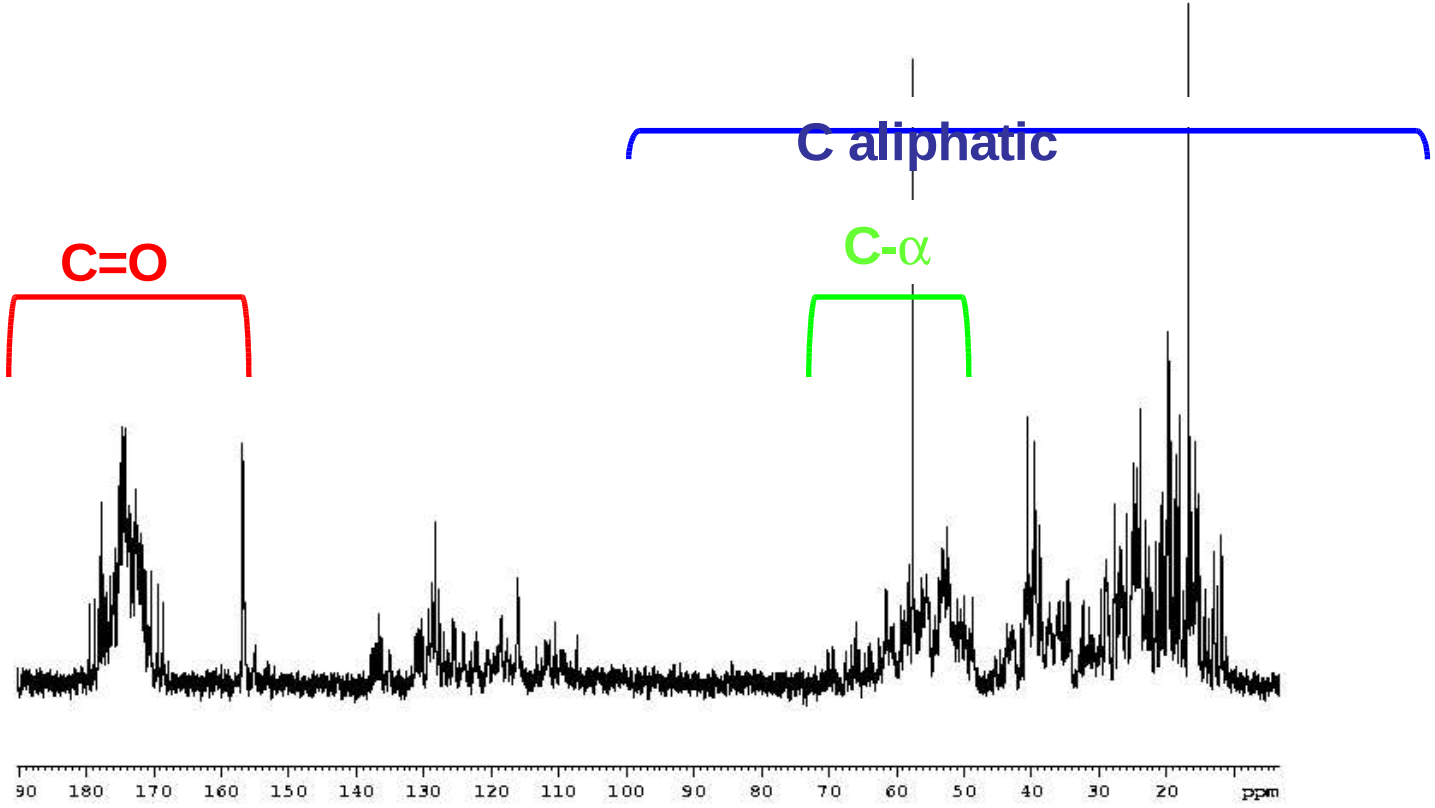
^{1}H	90° hard pulse	xx µsec	
	flipback pulse	Sinc or Square, 2 ms	
	TOCSY	25 - 35 µsec	
	ROESY	80-100 µsec	
	WALTZ decoupling pulse	80-100 µsec	
	DIPSI decoupling pulse	40 µsec	
^{15}N	90° hard pulse	xx µsec	decp90f3
	decoupling pulse	250 µsec	decp90f3
^{2}H	90° hard pulse	xx µsec	decp902hf4
	decoupling pulse	350-400 µsec	decp902hf4

Pulse calibration: Check List

^{13}C -selective pulses are required to excite / invert / refocus Cα, Cali and CO regions

-identical parameters are used for the CO/Cali selective pulses

-pulses with higher selectivity are used for Cα

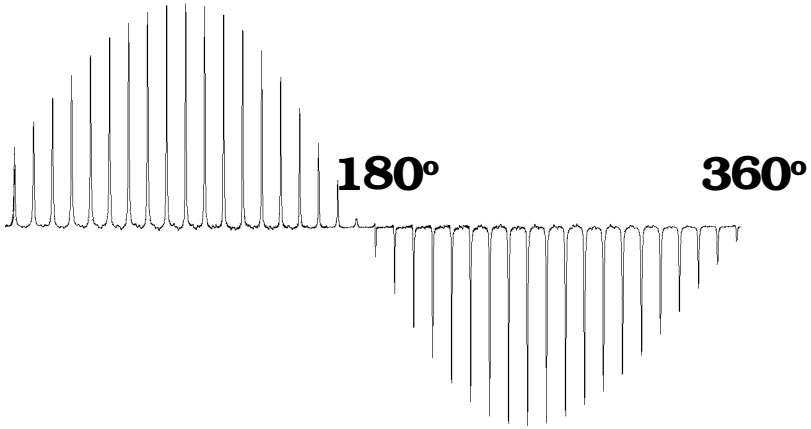


Pulse calibration: Check List

^{13}C	90° hard pulse	xx µsec	decp90
	CC-TOCSY pulse	22-25 µsec	decp90
	GARP decoupling pulse	55-65 µsec	decp90
	90° shape (Cali, CO)	Q5, 320 µsec /G4 308 µsec	decp90sp
	90° shape (Cα)	Q5, 410 µsec	decp90sp
	180° shape (Cali, CO)	Q3, 256/210 µsec	decp180sp
	180° shape (Cα)	Q3, 360 µsec	decp180sp
	180° shape (CO decoupling)	Gauss, truncation level 5%, 100 µsec	decp180sp
	adiabatic inversion	Crp60,0.5,20.1 or Crp80,0.5,20.1, 500 µsec	calculate
	adiabatic refocussing	Crp60comp.4 or Crp80comp.4, 2 msec	calculate
	adiabatic decoupling	CHIRP, 18-40kHz sweep 1.5 ms	calculate

RED: values for 800 MHz

Pulse calibration - observe channel



Pulse sequences: ***zg*** ***[hard pulse]***

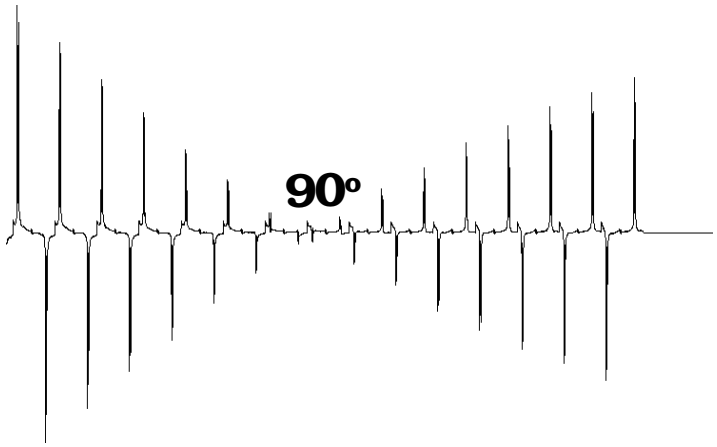
selzg ***[for selective pulses]***

Note: ***- on- resonance***

- 6dB attenuation doubles the pulse length

- excitation bandwidth $\gamma B_1/2\pi = 1/(4*\tau_p^{90})$

Pulse calibration - indirect mode, excitation

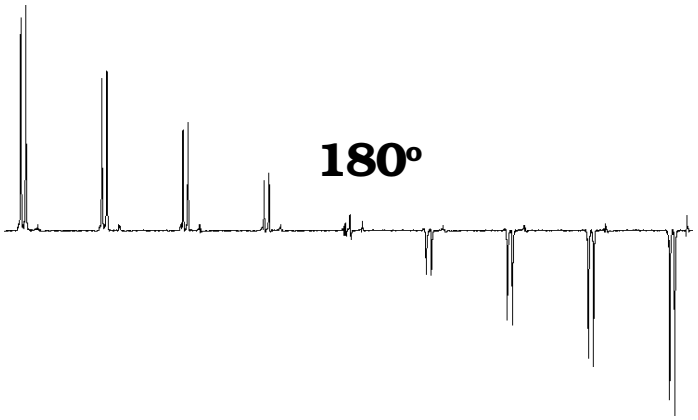


Pulse sequences:	***decp90***	***2nd channel***	***^{13}C***
	decp90f3	***3rd channel***	***^{15}N***
	decp902hf4	***4th channel***	***^{2}H***
	decp90sp	***2nd/selective***	***^{13}C***

Note: ***- on- resonance on the other channel***

- anti-phase signal

Pulse calibration - indirect mode, inversion

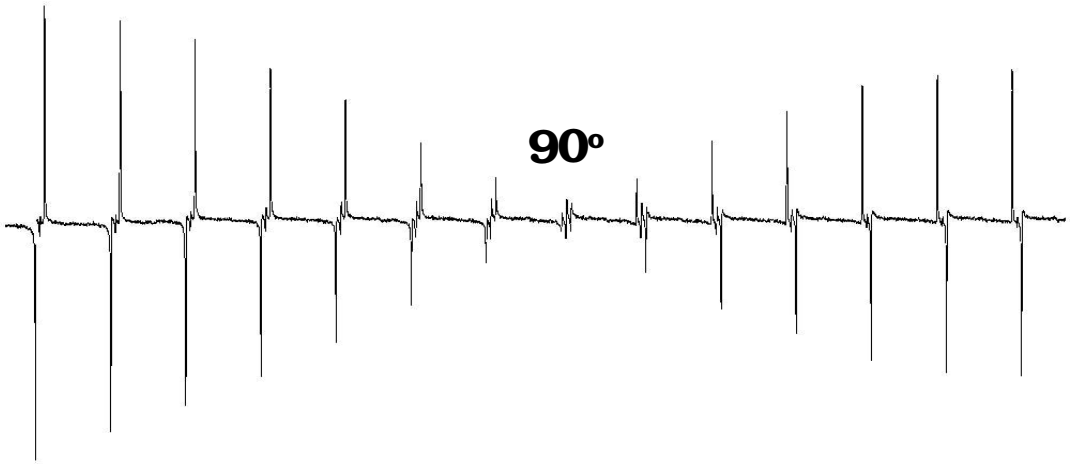


Pulse sequence: ***decp180sp 2nd channel selective ^{13}C***

Note: ***- on-resonance on the 2nd channel (o2p)***

- in-phase signal

Pulse calibration - indirect mode, 2H-dec.



Pulse sequence: ***decp902hf4***

Note: ***ASTM-sample: 60% D_6C_6***

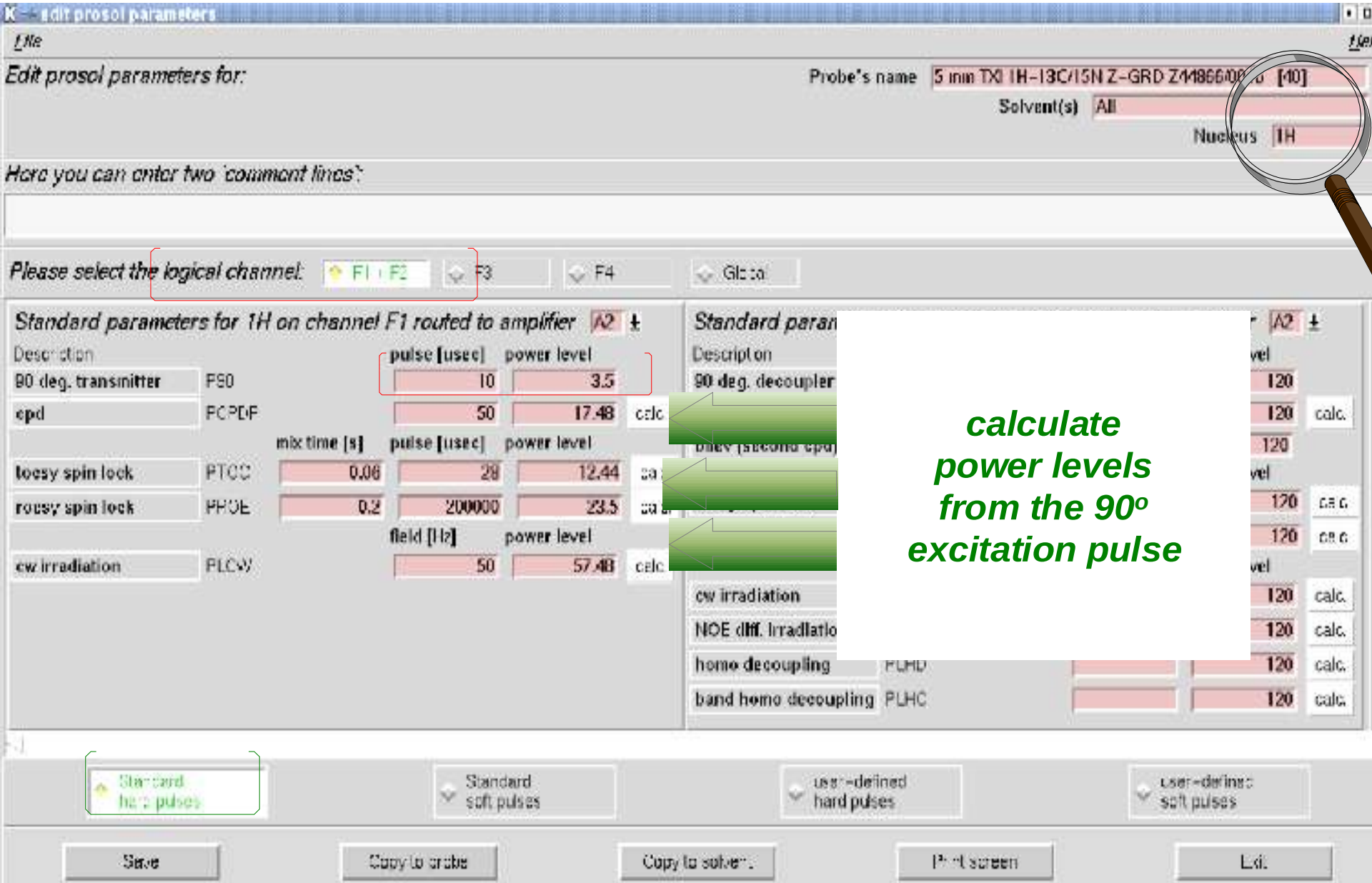
- ^{13}C-observe

- ^{2}H on-resonance

- anti-phase triplet

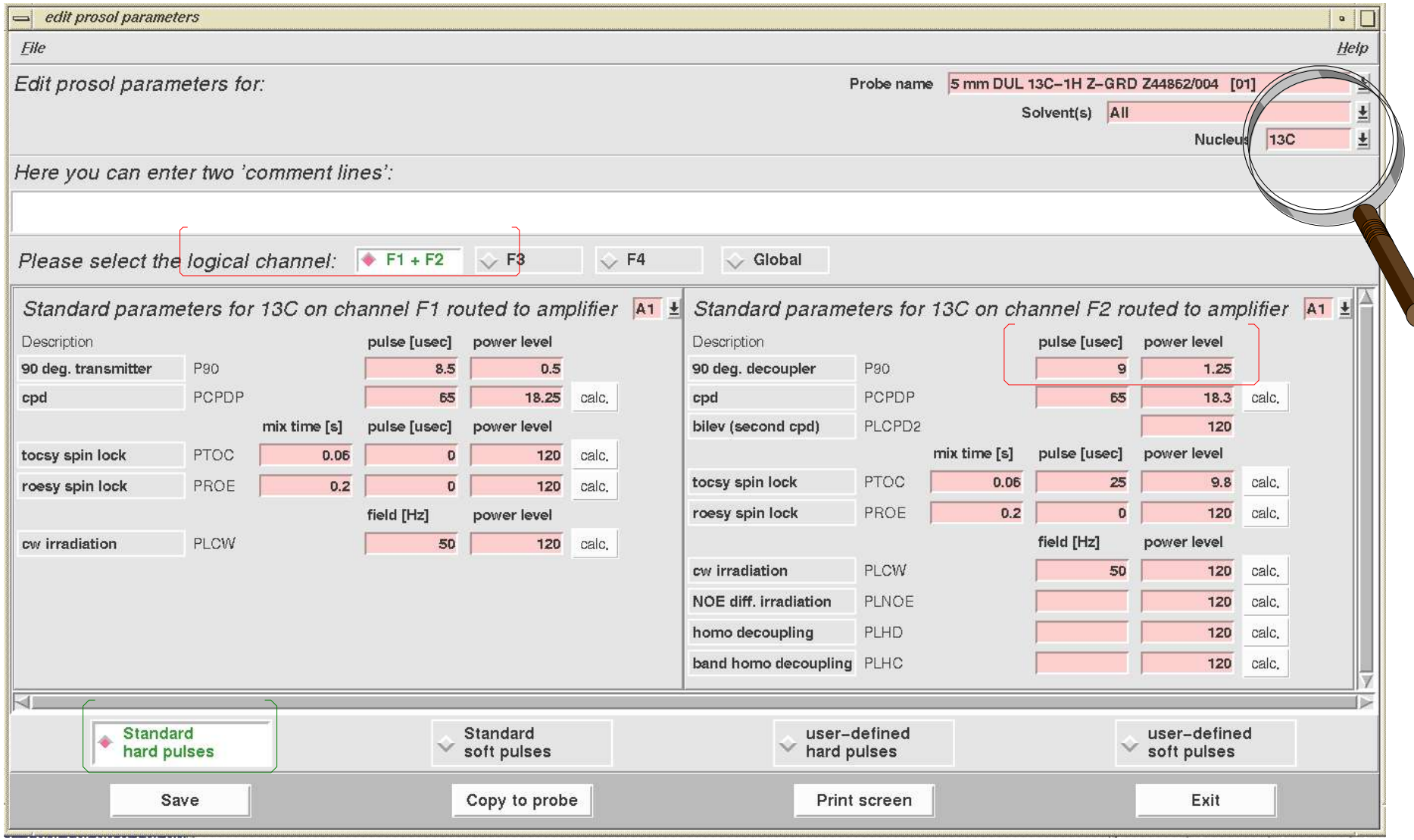
Parameter	Value	Comments
PULPROG	decp902hf4	pulse program
NUC1	13C	nucleus on f1 channel
O1P	128 ppm	^{13}C offset
NUC4	2H	nucleus on f4 channel
O4P	7.28 ppm	^{2}H offset
NS	1	
DS	0	
CNST5	24	$^{1}J_{CD}$

edprosol - set hard pulses for ^{1}H

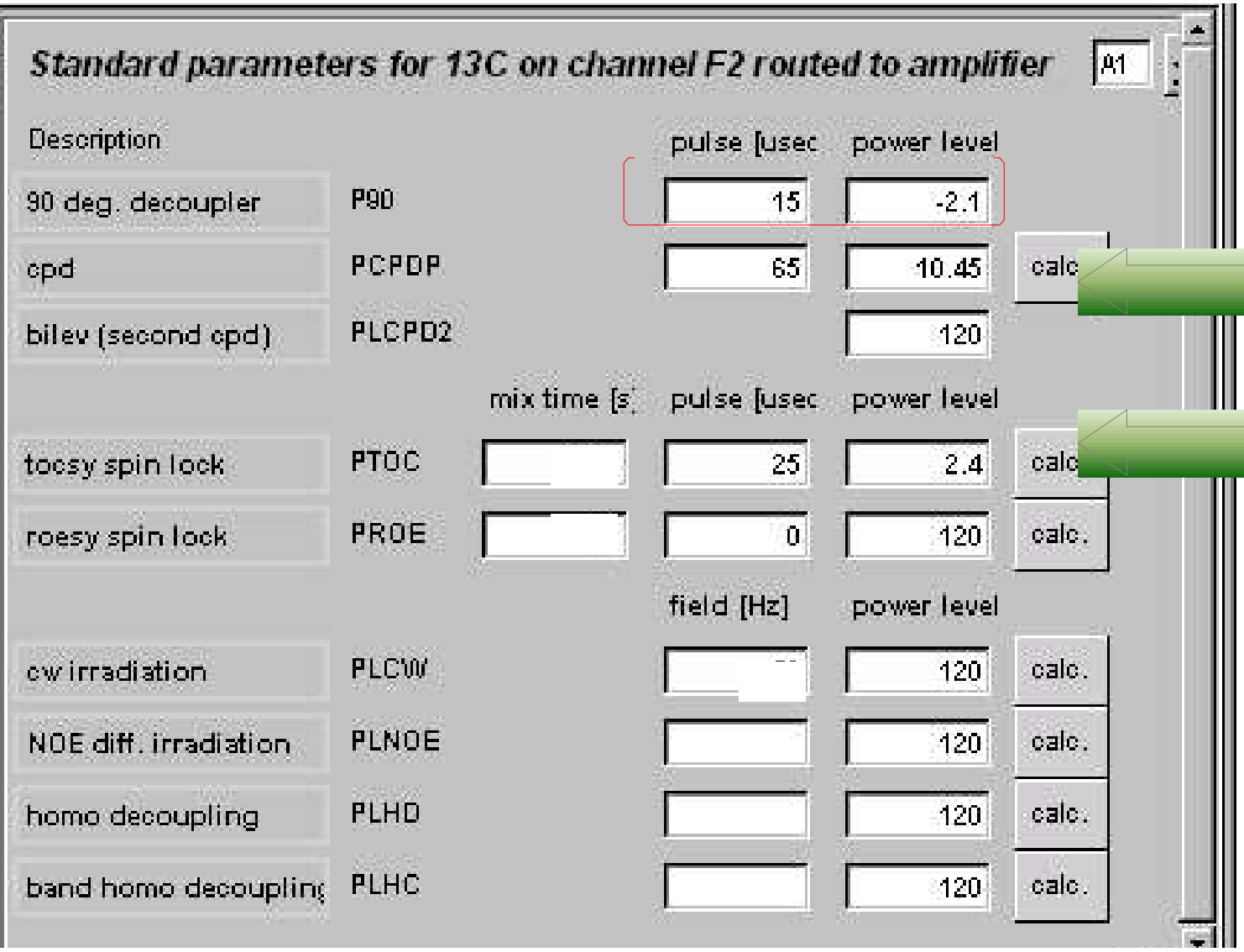


calculate power levels from the 90° excitation pulse

edprosol - set hard pulses for ^{13}C

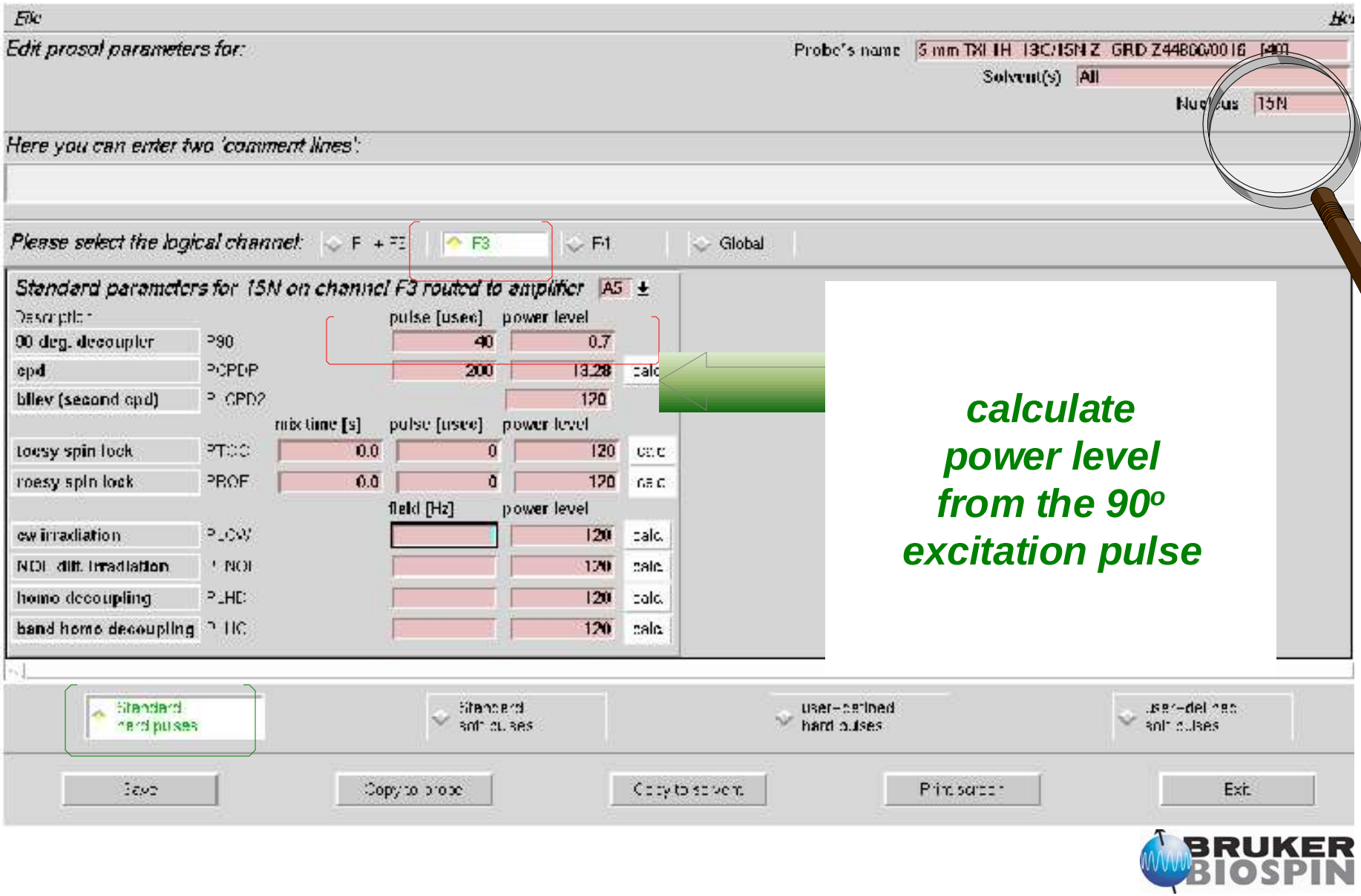


edprosol - set hard pulses for ^{13}C, cont.

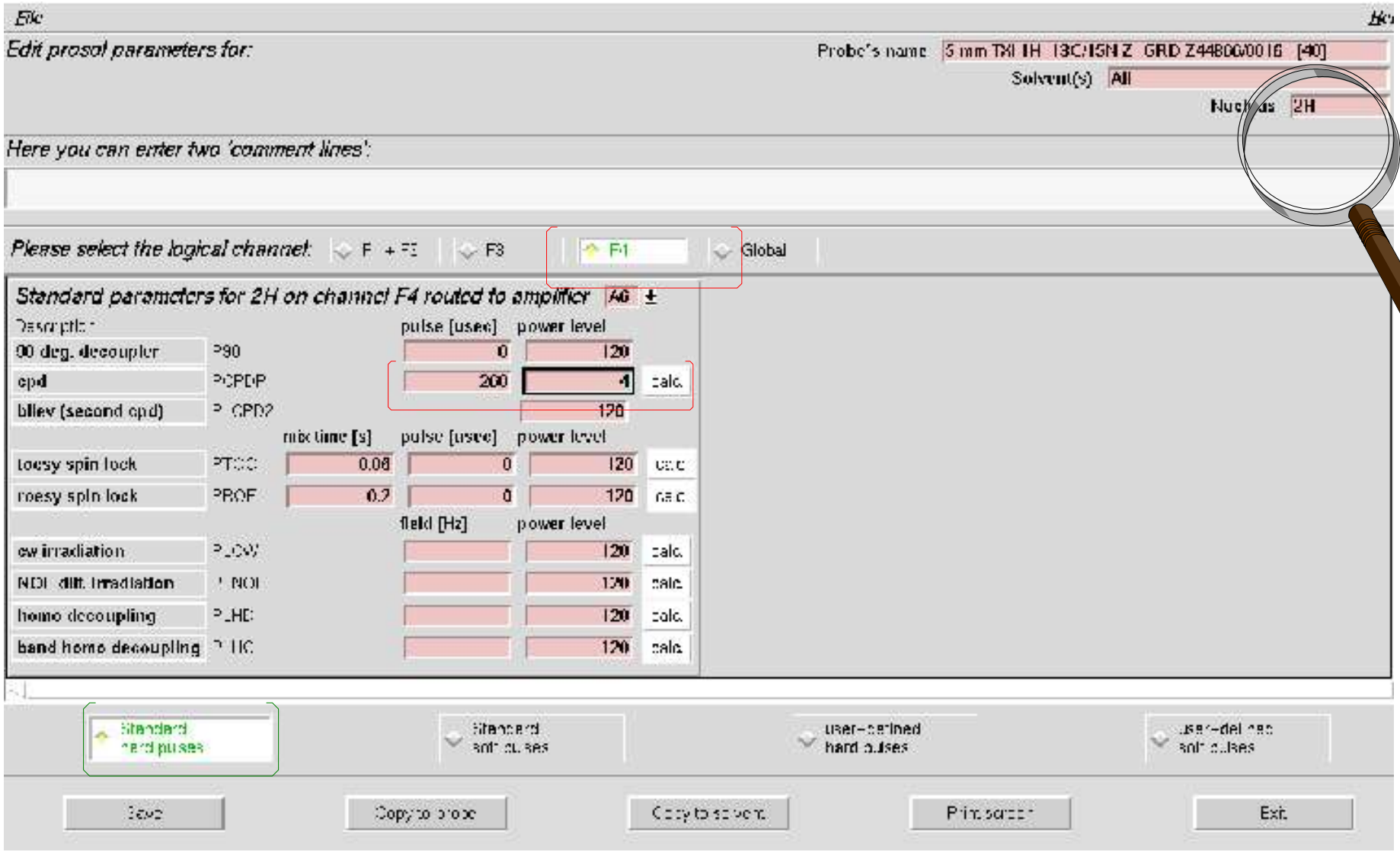


calculate power levels from the 90° excitation pulse

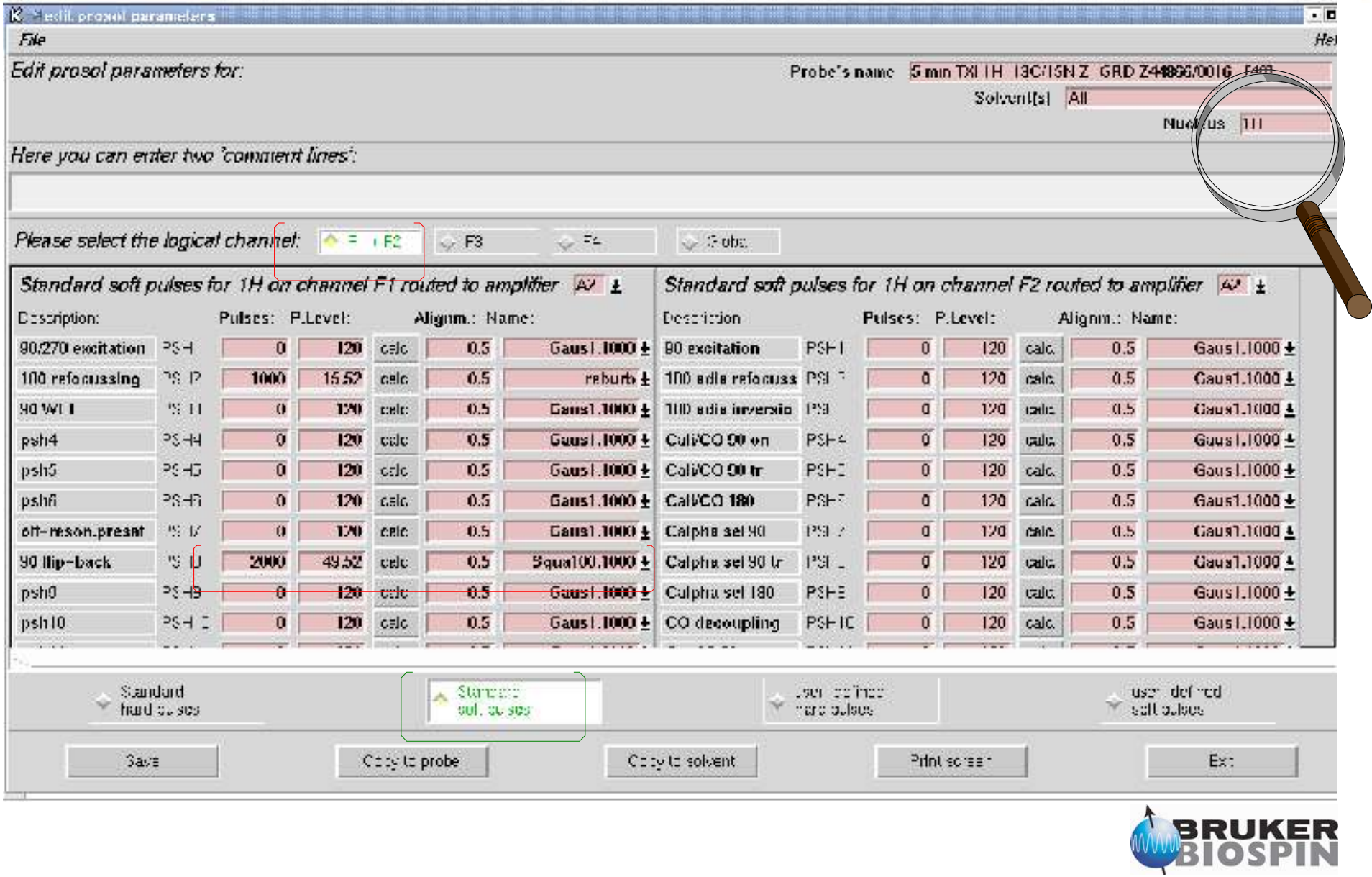
edprosol - set hard pulses for ^{15}N



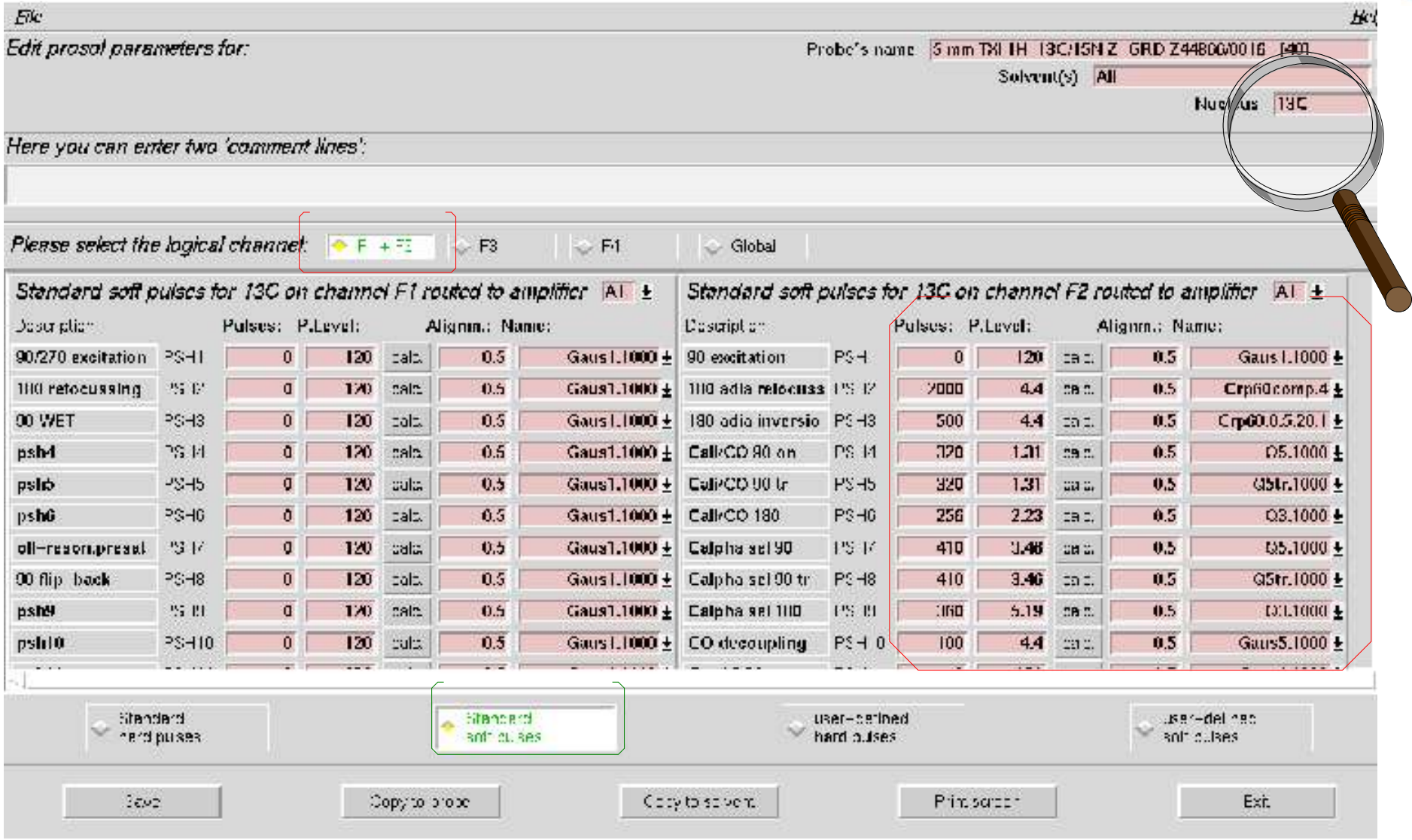
edprosol - set decoupling pulse for ^{2}H



edprosol - set water flip-back pulse for ^{1}H



edprosol - set soft pulses for ^{13}C



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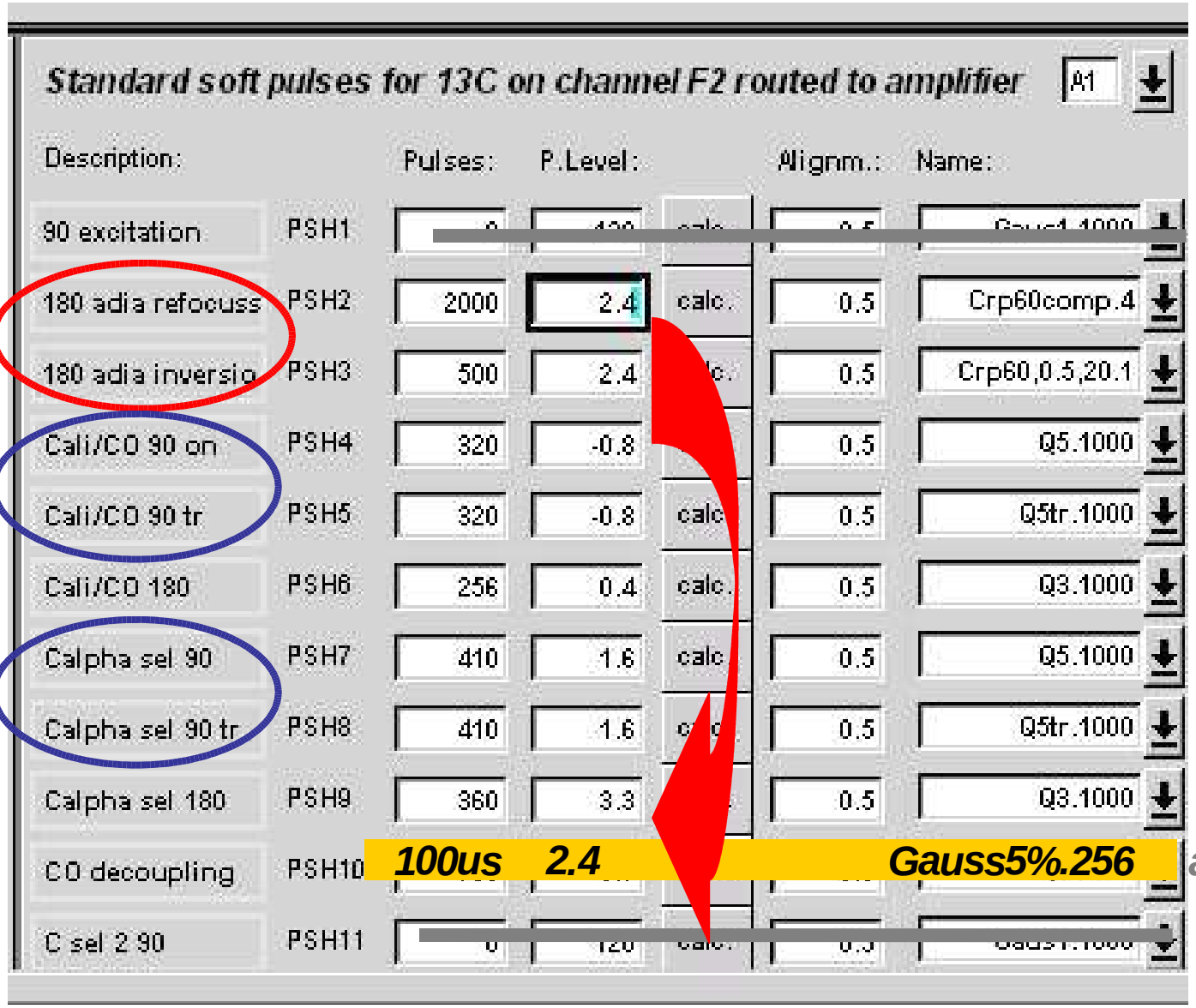
edprosol - set soft pulses for ^{13}C, cont.



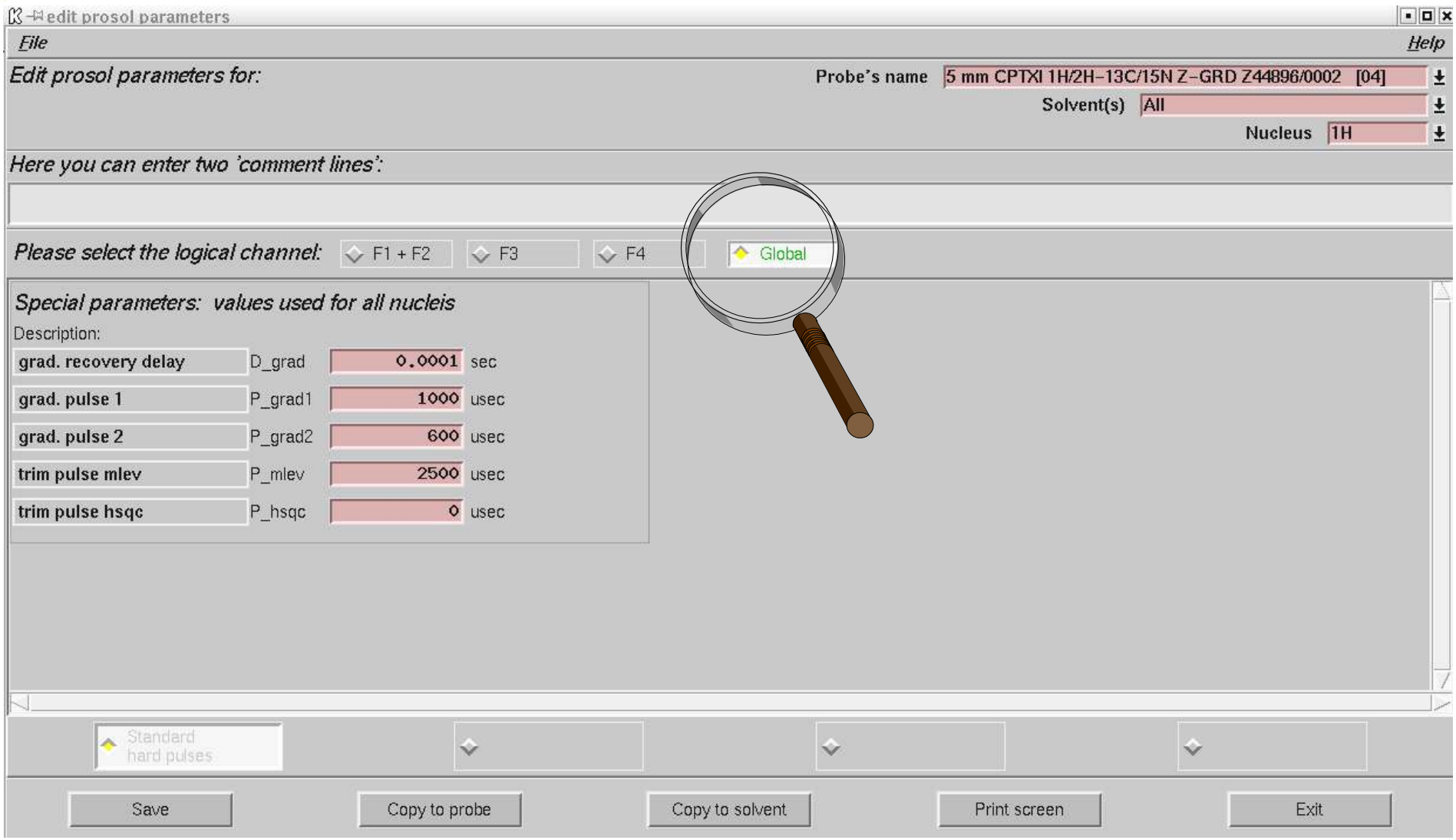
not used

note: same as the power of 25us 90°-pulse

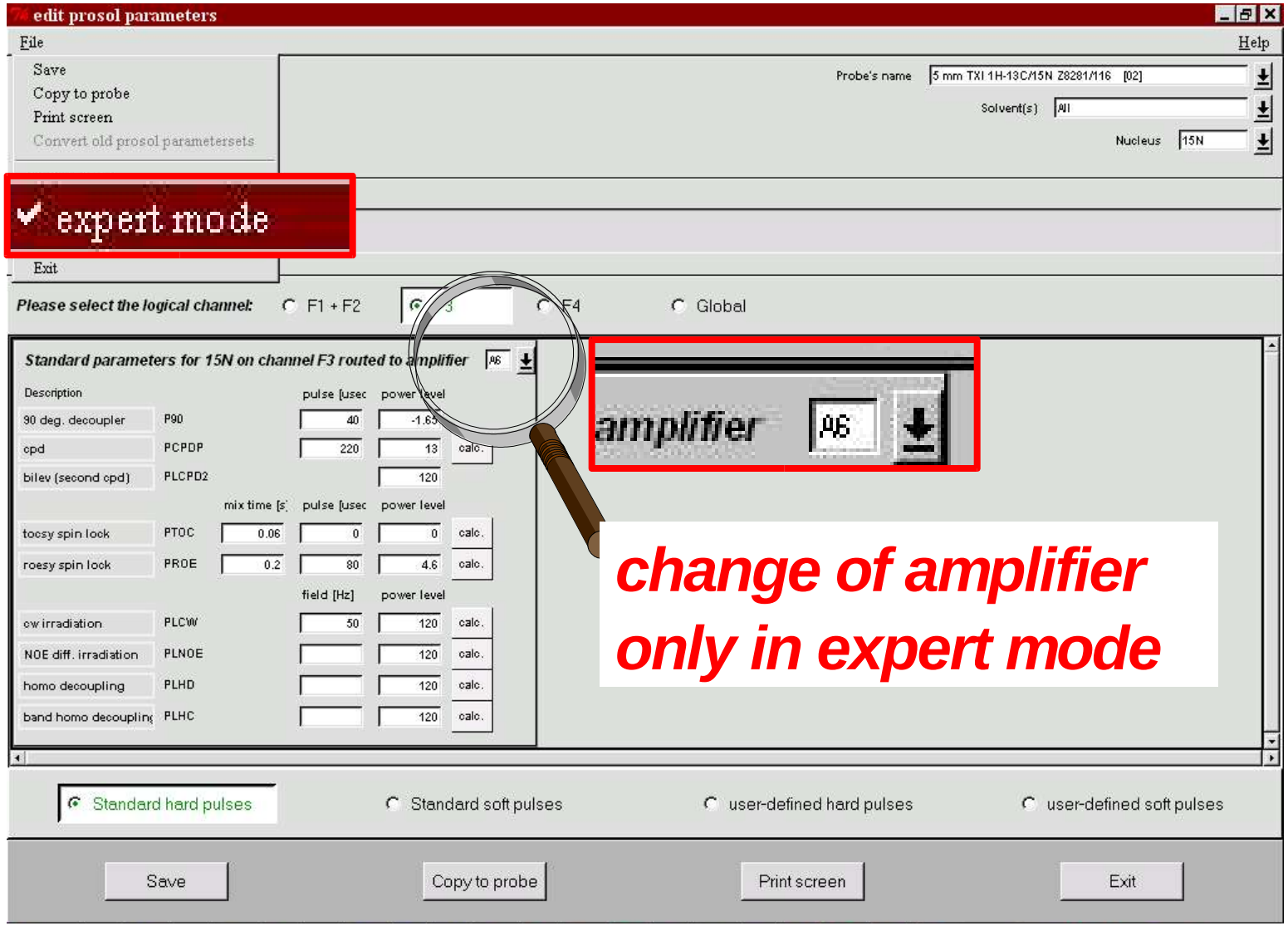
advantage: shorter

not used

edprosol - global settings



edprosol - expert mode



GETPROSOL

getprosol without options:

- loads parameters for pulses according to prosol-tables created with ‘edprosol’
- prosol-tables are global, any changes will apply to all users and data set

getprosol <nuc> <P90> <PL90>

- recalculate power levels of the prosol-table for all pulses of selected nucleus
- standard prosol-tables are NOT modified
- changes act on current data set only

GETPROSOL

Example:

- Hard ^{1}H-pulse had been re-calibrated:
  - 90^{o} = 9.7µs @ pl1 = 2.3dB
- ^{1}H-pulse differs from value stored in prosol-table, which is:
  - 90^{o} = 7.2µs @ pl1 = 1.8dB
- Recalculate all power levels for all pulses stored in the prosol-table of ^{1}H:

  enter: getprosol 1H 9.7us 2.3dB
- Now all ^{1}H pulses have been recalculated and set in the current dataset

AU programs for calculations on pulses

pulse

- calculate power level, pulse duration or excitation bandwidth
- calculations are based on transmitter pulse P1 and PL1
- examples:
  - pulse 25us : power level and bandwidth for 25us pulse
  - pulse 12.3khz : power level and duration for a pulse with a bandwidth of γB_1 = 12.3kHz
  - pulse 5.3db : pulse duration and bandwidth for a pulse applied with 5.3 dB RF power

calcpowlev

- calculate power for a new pulse, related to reference pulse

Before you start….

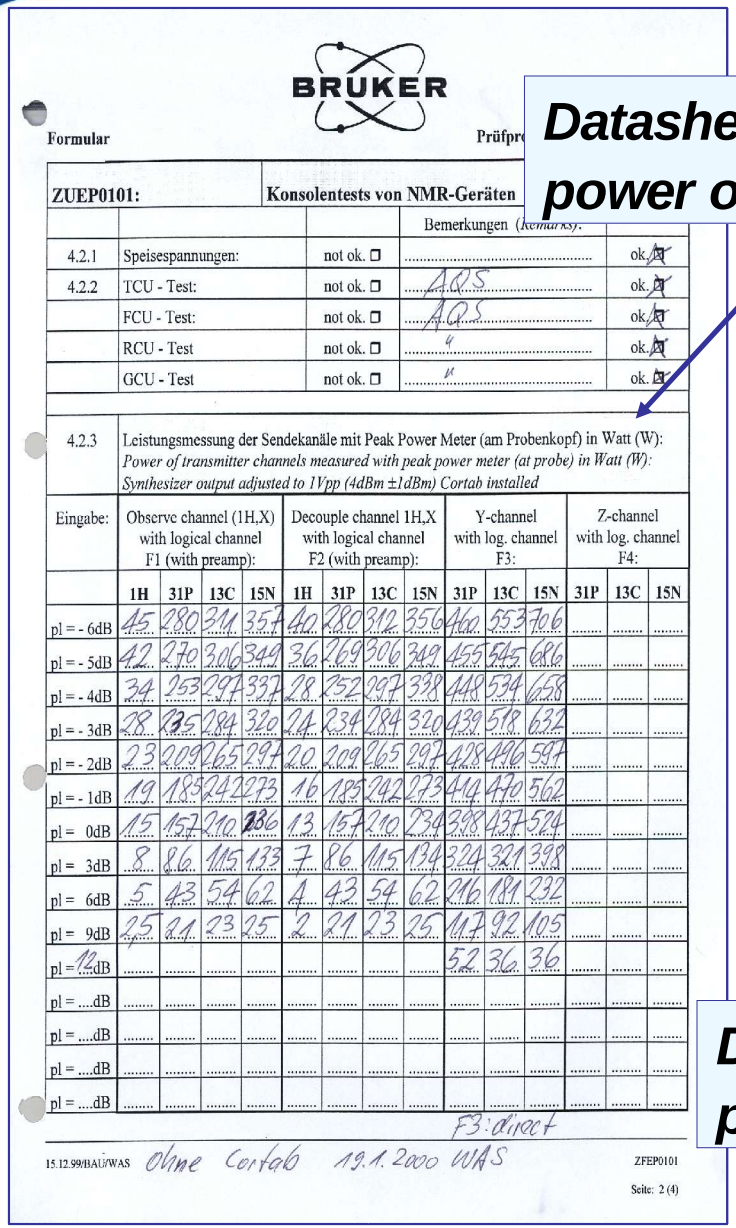
Undamaged coil in Probe

Heat damaged Coil

Before you start - maximum power

Datasheet output power of amplifiers

Datasheet allowed power for probe



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Formular Prüfpr

ZUEP0101: Konsolentests von NMR-Geräten

			Bemerkungen (remarks):	
4.2.1	Speisespannungen:	not ok. ☐	.....	ok. ☒
4.2.2	TCU - Test:	not ok. ☐	..... AQS	ok. ☒
	FCU - Test:	not ok. ☐	..... AQS	ok. ☒
	RCU - Test	not ok. ☐	 4	ok. ☒
	GCU - Test	not ok. ☐	..... "	ok. ☒

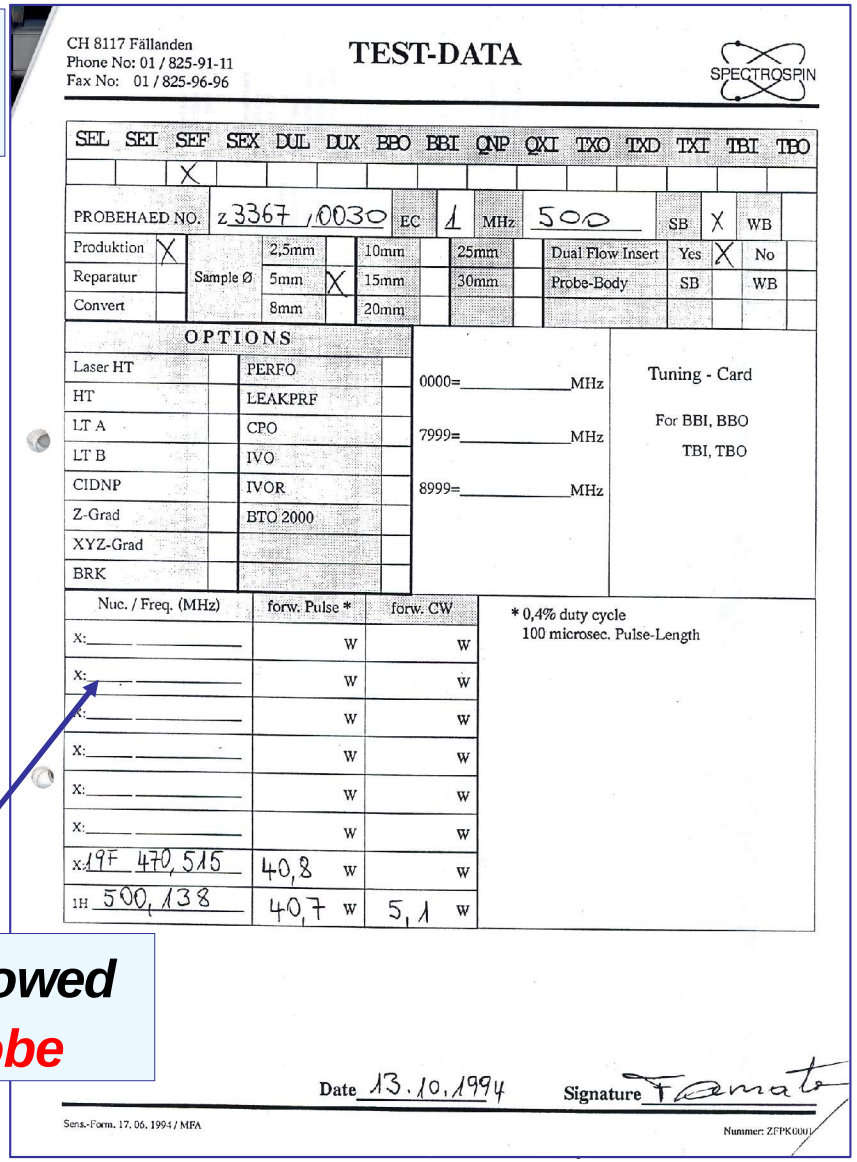
4.2.3 Leistungsmessung der Sendekanäle mit Peak Power Meter (am Probenkopf) in Watt (W):
Power of transmitter channels measured with peak power meter (at probe) in Watt (W):
Synthesizer output adjusted to 1Vpp (4dBm ±1dBm) Cortab installed

Eingabe:	Observe channel (1H,X) with logical channel F1 (with preamp):				Decouple channel 1H,X with logical channel F2 (with preamp):				Y-channel with log. channel F3:			Z-channel with log. channel F4:		
	1H	31P	13C	15N	1H	31P	13C	15N	31P	13C	15N	31P	13C	15N
pl = - 6dB	45	280	311	357	40	280	312	356	460	553	706	.....	.....	.....
pl = - 5dB	42	270	306	349	36	269	306	349	455	545	686	.....	.....	.....
pl = - 4dB	34	253	297	337	28	252	297	338	448	534	658	.....	.....	.....
pl = - 3dB	28	235	284	320	24	234	284	320	439	518	632	.....	.....	.....
pl = - 2dB	23	209	265	297	20	209	265	297	428	496	597	.....	.....	.....
pl = - 1dB	19	185	242	273	16	185	242	273	414	470	562	.....	.....	.....
pl = 0dB	15	157	210	236	13	157	210	234	398	437	524	.....	.....	.....
pl = 3dB	8	86	115	133	7	86	115	134	324	321	398	.....	.....	.....
pl = 6dB	5	43	54	62	4	43	54	62	216	181	232	.....	.....	.....
pl = 9dB	25	21	23	25	2	21	23	25	117	92	105	.....	.....	.....
pl = 12dB	.....	.....	.....	.....	.....	.....	.....	.....	52	36	36	.....	.....	.....
pl = ...dB	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
pl = ...dB	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
pl = ...dB	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
pl = ...dB	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....

F3: direct

15.12.99/BAU/WAS ohne Cortab 19.1.2000 WAS ZFEP0101

Seite: 2 (4)



CH 8117 Fällanden
Phone No: 01 / 825-91-11
Fax No: 01 / 825-96-96

TEST-DATA

SPECTROSPIN

SEL	SEI	SEF	SEX	DUL	DUX	BBO	BBI	QNP	QXI	TXO	TXD	TXI	TBI	TBO
		X												

PROBEHAED NO. z3367/0030 EC 1 MHz 500 SB X WB

Produktion	X	Sample Ø	2,5mm		10mm		25mm	Dual Flow Insert	Yes X No
Reparatur			5mm	X	15mm		30mm	Probe-Body	SB WB
Convert			8mm		20mm				

OPTIONS			
Laser HT		PERFO	
HT		LEAKPRF	
LT A		CPO	
LT B		IVO	
CIDNP		IVOR	
Z-Grad		BTO 2000	
XYZ-Grad			
BRK			

0000=_____MHz
7999=_____MHz
8999=_____MHz

Tuning - Card
For BBI, BBO
TBI, TBO

Nuc. / Freq. (MHz)	forw. Pulse *	forw. CW
X:_____	W	W
X:_____	W	W
X:_____	W	W
X:_____	W	W
X:_____	W	W
X:_____	W	W
X:19F 470,515	40,8 W	W
1H 500,138	40,7 W	5,1 W

* 0,4% duty cycle
100 microsec. Pulse-Length

Date 13.10.1994 Signature Tamato

Sens.-Form. 17.06.1994 / MFA Nummer: ZPPK0001

Before you start - maximum power

Datasheet output power of amplifiers

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Formular Prüfpr

ZUEP0101: Konsolentests von NMR-Geräten

			Bemerkungen	
4.2.1	Speisespannungen:	not ok. ❒	.....	ok. ☒
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	FCU - Test:	not ok. ❒	..... AQS	ok. ☒
	RCU - Test	not ok. ❒	..... "	ok. ☒
	GCU - Test	not ok. ❒	..... "	ok. ☒

4.2.3 Leistungsmessung der Sendekanäle mit Peak Power Meter (am Probenkopf) in Watt (W):
Power of transmitter channels measured with peak power meter (at probe) in Watt (W):
Synthesizer output adjusted to 1Vpp (4dBm ±1dBm) Cortab installed

Eingabe:	Observe channel (1H,X) with logical channel F1 (with preamp):				Decouple channel 1H,X with logical channel F2 (with preamp):				Y-channel with log. channel F3:			Z-channel with log. channel
	1H	31P	13C	15N	1H	31P	13C	15N	31P	13C	15N	31P
pl = - 6dB	45	280	311	357	40	280	312	356	460	553	706	.....
pl = - 5dB	42	270	306	349	36	269	306	349	455	545	686	.....
pl = - 4dB	34	253	297	337	28	252	297	338	448	534	658	.....
pl = - 3dB	28	235	284	320	24	234	284	320	439	518	632	.....
pl = - 2dB	23	209	265	297	20	209	265	297	428	496	597	.....
pl = - 1dB	19	185	242	273	16	185	242	273	414	470	562	.....
pl = 0dB	15	157	210	236	13	157	210	234	398	437	524	.....
pl = 3dB	8	86	115	133	7	86	115	134	324	321	398	.....
pl = 6dB	5	45	54	62	4	43	54	62	216	181	232	.....
pl = 9dB	25	21	23	25	2	21	23	25	117	92	105	.....
pl = 12dB	.....	.....	.....	.....	.....	.....	.....	.....	52	36	36	.....
pl = ...dB	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
pl = ...dB	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
pl = ...dB	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
pl = ...dB	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....

F3: direct

15.12.99/BAU/WAS ohne Cortab 19.1.2000 WAS

Eingabe:	Observe channel (1H,X) with logical channel F1 (with preamp):				Decouple channel 1H,X with logical channel F2 (with preamp):				Y-channel with log. channel F3:		
	1H	31P	13C	15N	1H	31P	13C	15N	31P	13C	15N
pl = - 6dB	45	280	311	357	40	280	312	356	460	553	706
pl = - 5dB	42	270	306	349	36	269	306	349	455	545	686
pl = - 4dB	34	253	297	337	28	252	297	338	448	534	658
pl = - 3dB	28	235	284	320	24	234	284	320	439	518	632
pl = - 2dB	23	209	265	297	20	209	265	297	428	496	597
pl = - 1dB	19	185	242	273	16	185	242	273	414	470	562

Typical Pulses for the 5mm CryoProbe™

CryoProbe RF power handing

Typical Pulses for the 5mm CryoProbe™ 500MHz and 600MHz

Instructions (NMR-Applications)

4.2 Pulse and power recommendations

Note: all values require a minimum repetition rate appr. *d1+aq* of 1sec [2]
Important: observe the separate notes on the following page!

	5mm 500MHz TXI CryoProbe™	5mm 600MHz TXI CryoProbe™
^{1}H		
hard pulse [3] (max. length 360°)	8.0 µsec	8.0 µsec
hard pulse for lossy samples	Power level corresponding to 8.0 µsec pulse for non-lossy sample	Power level corresponding to 8.0 µsec pulse for non-lossy sample
trim pulse p28 [4]	2 msec @ 10 µsec	2 msec @ 10 µsec
TOCSY spin lock	200 msec @ 25 µsec 400 msec @ 35 µsec	200 msec @ 25 µsec 400 msec @ 35 µsec
ROESY spin lock	Up to CW for power level corresponding to a 100 µsec pulse	Up to CW for power level corresponding to a 100 µsec pulse
WALTZ16 decoupling during 13C-detection	Up to CW for power level corresponding to a 100 µsec pulse	Up to CW for power level corresponding to a 100 µsec pulse
DIPSI2-decoupling in triple resonance	400 msec @ 50 µsec	400 msec @ 50 µsec
^{13}C		
hard pulse (max. length 360°)	15.0 µsec	15.0 µsec
trim pulse [5]	2msec @ 25 µsec	2msec @ 25 µsec
CC spin lock [5]	20msec @ 25 µsec	20msec @ 25 µsec
GARP decoupling [6]	140msec @ 65 µsec	140msec @ 55 µsec
selective pulses [7]	Q5: 320 µsec Q3: 256 µsec CHIRP: 2ms @ 25 µsec	Q5: 320 µsec Q3: 256 µsec CHIRP: 2ms @ 25 µsec
^{15}N		
hard pulse (max. length 360°)	40.0 µsec	40.0 µsec
WALTZ16 decoupling [6]	140 msec @ 200 µsec	140 msec @ 170 µsec
CPMG T2 [8]	250 msec @ 80 µsec	250 msec @ 80 µsec
^{2}H		
hard pulse (max. length 360°)	150usec	150usec
WALTZ16 decoupling	100 msec @ 250 µsec	100 msec @ 250 µsec
Z-Gradient		
Absolute max. current	10A	10A
Max.overall length [9]	10ms @ 10A	10ms @ 10A

	5mm 500MHz TXI CryoProbe™	5mm 600MHz TXI CryoProbe™
^{1}H		
hard pulse [3] (max. length 360°)	8.0 μsec	8.0 μsec
hard pulse for lossy samples	Power level corresponding to 8.0 μsec pulse for non-lossy sample	Power level corresponding to 8.0 μsec pulse for non-lossy sample
trim pulse p28 [4]	2 msec @ 10 μsec	2 msec @ 10 μsec
TOCSY spin lock	200 msec @ 25 μsec 400 msec @ 35 μsec	200 msec @ 25 μsec 400 msec @ 35 μsec
ROESY spin lock	Up to CW for power level corresponding to a 100μsec pulse	Up to CW for power level corresponding to a 100μsec pulse
WALTZ16 decoupling during 13C-detection	Up to CW for power level corresponding to a 100μsec pulse	Up to CW for power level corresponding to a 100μsec pulse
DIPSI2-decoupling in triple resonance	400 msec @ 50 μsec	400 msec @ 50 μsec
^{13}C		
hard pulse (max. length 360°)	15.0 μsec	15.0 μsec
trim pulse [5]	2msec @ 25 μsec	2msec @ 25 μsec
CC spin lock [5]	20msec @ 25 μsec	20msec @ 25 μsec
GARP decoupling [6]	140msec @ 65 μsec	140msec @ 55 μsec
selective pulses [7]	Q5: 320 μsec Q3: 256 μsec CHIRP: 2ms @ 25 μsec	Q5: 320 μsec Q3: 256 μsec CHIRP: 2ms @ 25 μsec
^{15}N		
hard pulse (max. length 360°)	40.0 μsec	40.0 μsec
WALTZ16 decoupling [6]	140 msec @ 200μsec	140 msec @ 170μsec
CPMG T2 [8]	250 msec @ 80 μsec	250 msec @ 80 μsec
^{2}H		
hard pulse (max. length 360°)	150usec	150usec
WALTZ16	100 msec @ 250 μsec	100 msec @ 250 μsec

CryoProbe RF power handling

2. Observe the notes!

1. Repetition time

Note: all values require a minimum repetition rate appr. *d1+aq* of 1sec [2)]
Important: observe the separate notes on the following page!

	5mm 500MHz TXI CryoProbe™	5mm 600MHz TXI CryoProbe™
^{1}H		
hard pulse [3)] (max. length 360°)	8.0 µsec	8.0 µsec
hard pulse for lossy samples	Power level corresponding to 8.0 µsec pulse for non-lossy sample	Power level corresponding to 8.0 µsec pulse for non-lossy sample
trim pulse p28 [4)]	2 msec @ 10 µsec	2 msec @ 10 µsec
TOCSY spin lock	200 msec @ 25 µsec	200 msec @ 25 µsec

CryoProbe RF power handling

3. Max. pulse length

Note: all values require a minimum repetition rate appr. *d1+aq* of 1sec [2)]
Important: observe the separate notes on the following page!

	5mm 500MHz TXI CryoProbe™	5mm 600MHz TXI CryoProbe™
^{1}H		
hard pulse [3)] (max. length 360°)	8.0 µsec	8.0 µsec
hard pulse for lossy samples	Power level corresponding to 8.0 µsec pulse for non-lossy sample	Power level corresponding to 8.0 µsec pulse for non-lossy sample
trim pulse p28 [4)]	2 msec @ 10 µsec	2 msec @ 10 µsec
TOCSY spin lock	200 msec @ 25 µsec	200 msec @ 25 µsec

CryoProbe RF power handling

Note: all values require a minimum repetition rate appr. *d1+aq* of 1sec [2)]
Important: observe the separate notes on the following page!

	5mm 500MHz TXI CryoProbe™	5mm 600MHz TXI CryoProbe™
^{1}H		
lse [3)] ngth 360°)	8.0 µsec	8.0 µsec
hard pulse for lossy samples	Power level corresponding to 8.0 µsec pulse for non-lossy sample	Power level corresponding to 8.0 µsec pulse for non-lossy sample
trim pulse p28 [4)]	2 msec @ 10 µsec	2 msec @ 10 µsec
TOCSY spin lock	200 msec @ 25 µsec	200 msec @ 25 µsec

4. ^{1}H trim pulses

CryoProbe RF power handling

^{1}H pulses:

- shortest pulse allowed for non-lossy samples:

8usec (max. 360^{o})

- hard pulse for lossy samples:

power level corresp. to 8usec for non-lossy sample:

- trim pulse 'P28':

2ms @ 10usec [1)]

- TOCSY spin lock:

200ms @ 25usec [1)], 400ms @ 35usec [1)]

- ROESY, Waltz16-decoupling:

up to CW @ 100usec [1)]

[1)] power and pulse of non-lossy sample

CryoProbe RF power handling

^{13}C pulses:

- shortest pulse allowed:

15usec (max. 360°)

- trim pulses:

2ms @ 25usec

- CC spin lock:

20ms @ 25usec

- GARP decoupling:

140ms @ 65usec (500MHz) / 55usec (600-800MHz)

- selective pulses:

500-600 MHz: Q5: 320usec Q3: 256usec

700-800 MHz: G4: 308usec Q3: 210usec

CryoProbe RF power handling

^{15}N pulses:

- shortest pulse allowed:

40usec (max. 360^{o})

- WALTZ16 decoupling:

140ms @ 200usec (500MHz) / 170usec (600-800MHz)

- simultaneous ^{13}C, ^{15}N pulses:

attenuate power by 3dB on BOTH, ^{13}C and ^{15}N channel

CryoProbe RF power handling

Z-gradient:

- absolute max. current:

10A

- max. overall length:

10ms @ 10A