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Tema -5

Spectrometer calibrations

Spectrometer calibrations and specifications

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Spectrometer calibrations

- Observe Pulses
- Indirect Pulses
  - High abundance nuclides
  - Low abundance nuclides
- Decoupling pulses
  - homonuclear
  - heteronuclear
- Temperature calibration

Spectrometer Performance tests

- Relation signal to Noise (sensitivity)
- Line shape (homogeneity)
- Solvent presaturation

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Observe pulses calibration

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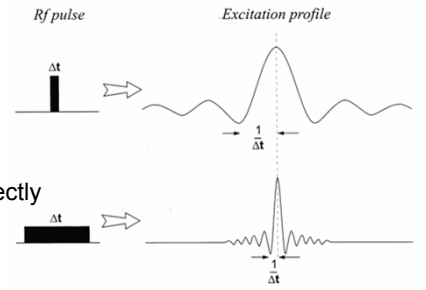
The RF pulse is defined by the lenght, power and shape

Varian : Coarse power 63 dB (high) to -10dB (low), Increment 1dB

Fine power

Hard pulse: calibrate directly

Soft or shape pulse : calibrate indirectly



For all frequencies can be excited in a homogeneous mode must be satisfied that $\gamma B_1 = 2\pi SW$, (SW equal to spectral window), $Pw_{90} << 1/4sw$

(1H at 500 MHz) SW=8000 ⟹ Pw90 << 31,24 us

Proton pulse calibration

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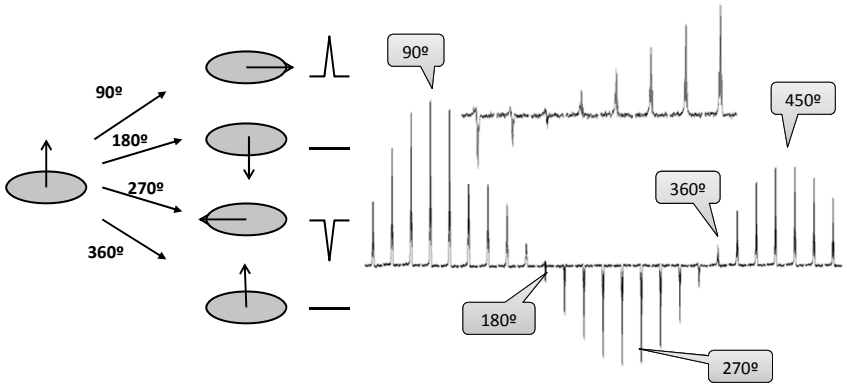
- Most hard (highest power) 90° pulses are typically from 5 us to 20 us.
- Direct observation for high power proton pulse calibration (or even for heteronuclei if sensitivity is sufficient)
  - 360° method (not quite sensitive to radiation damping or relaxation)
  - 180° method

First pulse with ≈2 us; 2 us increment

Refine with 1 us increment



Spectrometer Performance tests

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Standard sensitivity test samples of some common nucle

1H	0.1% Ethylbenzene in CDCl3
13C	40% dioxane in C6D6 (ASTM sample) No decoupling, C6D6 used for measurement
31P	0.0485 M triphenylphosphate in d6-acetone No decoupling
19F	0.05% trifluorotoluene in CDCl3 No decoupling
15N	90% formamide in d6-dmso. Use inverse gated dec. to suppress negative NOE
29	Si 85% hexamethyldisiloxane in d6-benzene No decoupling

Relation S/N (sensitivity)

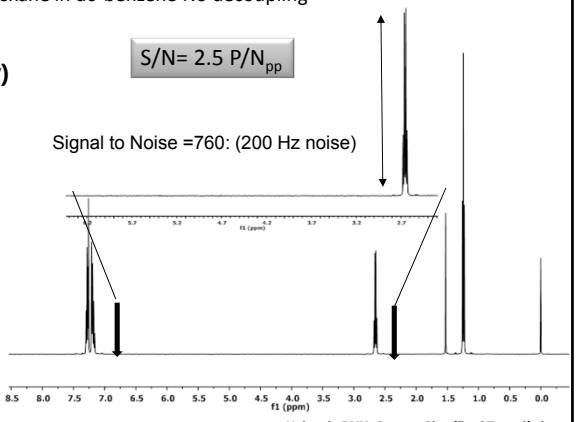
$S/N = 2.5\ P/N_{pp}$

Proton sensitivity test

0.1 % ETB sample in cdcl3

VNMRS500 One probe

Pw90 and d1=120s nt=1
lb=1.01 Hz



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Line Shape and Resolution

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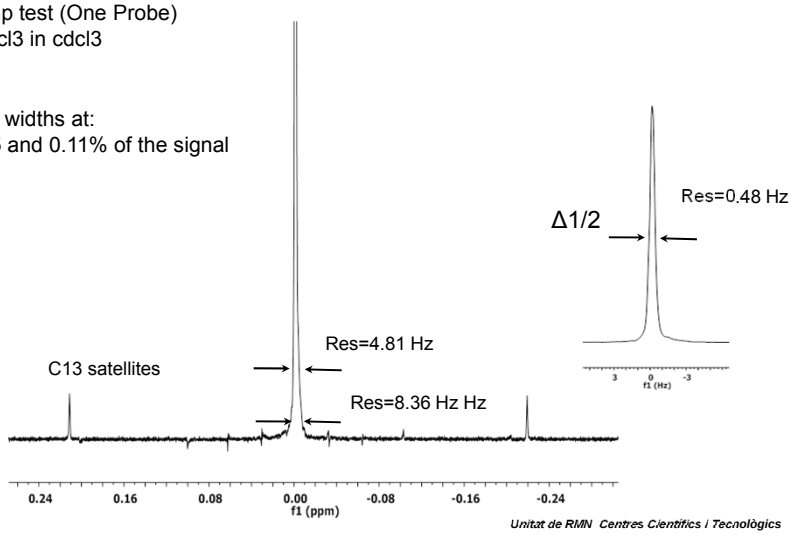
Resolution = the linewidth at Δ1/2 of the signal

VNMRS500
lshp test (One Probe)
chcl3 in cdcl3

lshp widths at:
0.55 and 0.11% of the signal



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Sensitivity test in 13C

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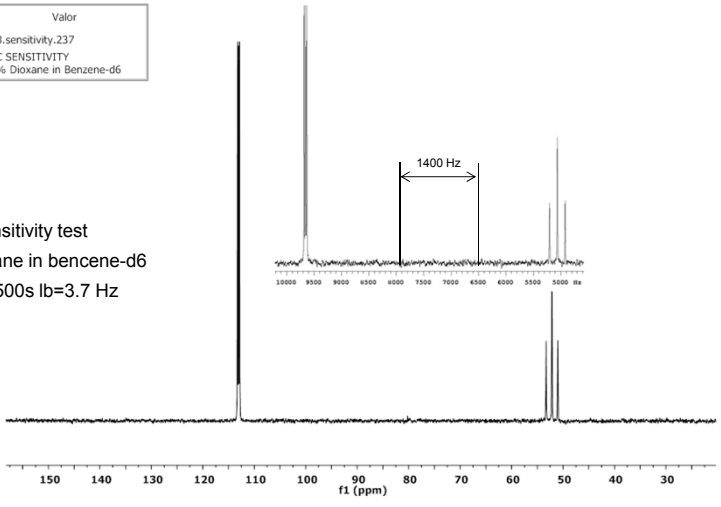
Parámetro	Valor
1 Title	C13.sensitivity.237
2 Comment	13C SENSITIVITY 40% Dioxane in Benzene-d6



Carbon sensitivity test
40% dioxane in bencene-d6
Pw90, d1=500s lb=3.7 Hz

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Solvent presaturation

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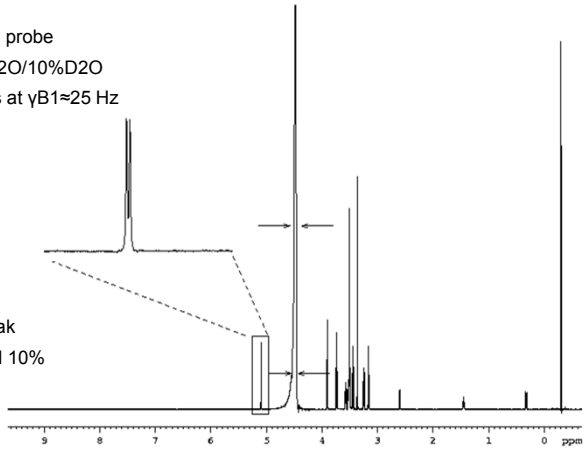
A good performance of the removal requires a high quality of the static field Bo and rf (B1) homogeneity

Presaturation test on B600 TXI probe
2mM sucrose and DSS in 90 H2O/10%D2O
presaturation water peak at 2 s at γB1≈25 Hz



Check the SN in anomeric peak
and the line width at 50% and 10% peak residual water

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