Pulse NMR for the determination of the nuclear polarisation

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- Comparison between CW and pulse NMR
- Setup
- Linearity measurement
- Signals
- Conclusion / outlook

Comparison between CW and pulsed NMR

Because of the dynamic range between the TE signal and highly polarised signals the linearity of the detection system is important.

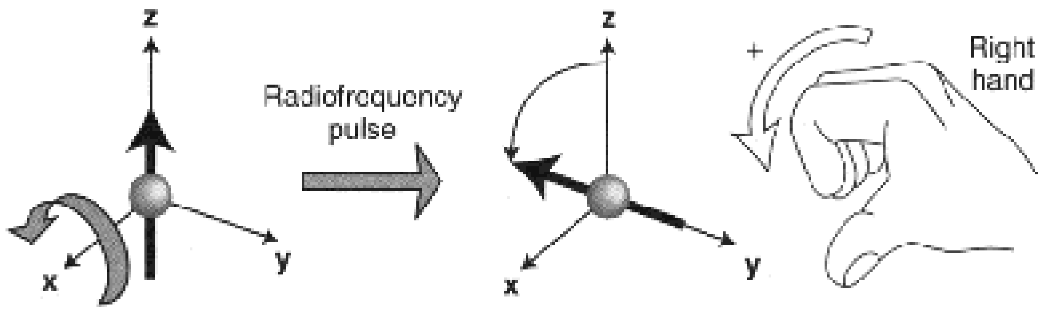
e.g. B= 2.5T and T = 1K $$\frac{P^{dyn}}{P^{TE}} = \frac{\geqslant 80\,\%}{0.052\,\%} \geqslant 1600$$

	CW-NMR (Liverpool-Box)	Pulsed NMR (low power)
saturation	negligible	depends on pulse length and rf power
sweep time	>50ms	1ms
Pulse length		µsec
ΔP/P proton	≈ΔT/T (TE)	?
ΔP/P deuteron	≈5%	?
price	≈15k€	≈30k€ (self made)

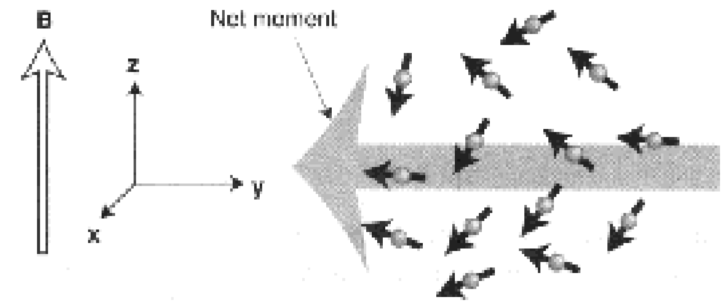
To increase S/N in CW-NMR the accumulation method is used
→
same in pulsed nm, but much faster (50 times)

$$\frac{S}{N} \propto \sqrt{n} \quad \textbf{and} \quad \frac{S}{N} \propto t_P \times n$$

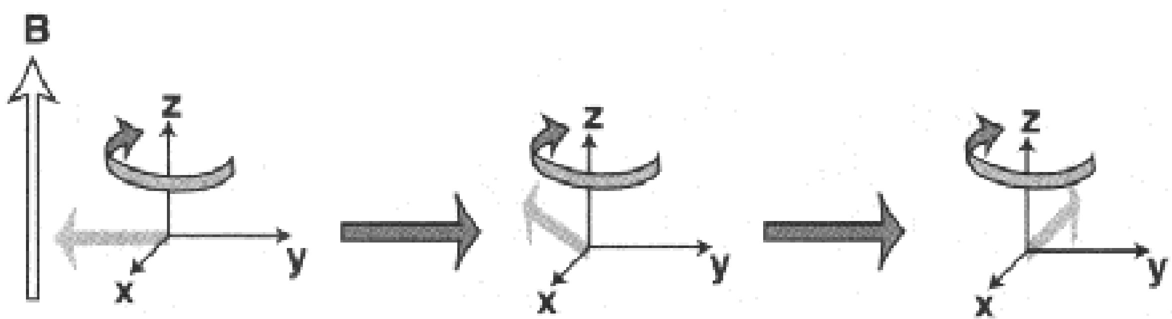
Principle of pulsed NMR



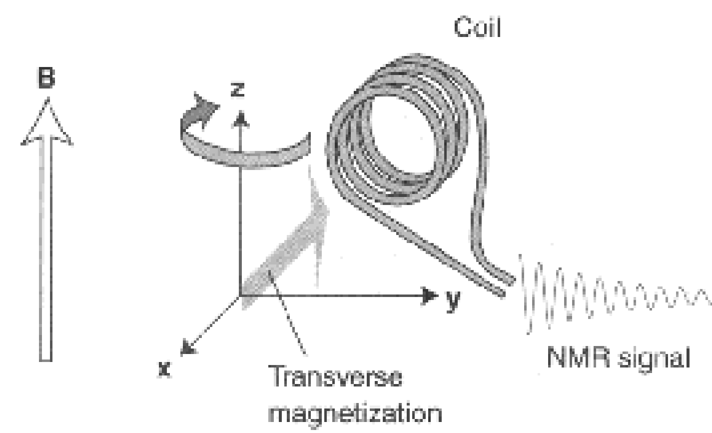
By applying a rf pulse every signal spin is rotated by $\pi/2$ about the *x*-axis.
The result is a spin polarization along the –*y*-axis



Since the pulse rotates the polarization of every spin the net polarization is therefore transformed along the –*y*-axis; thus a *transverse magnetization* arises.

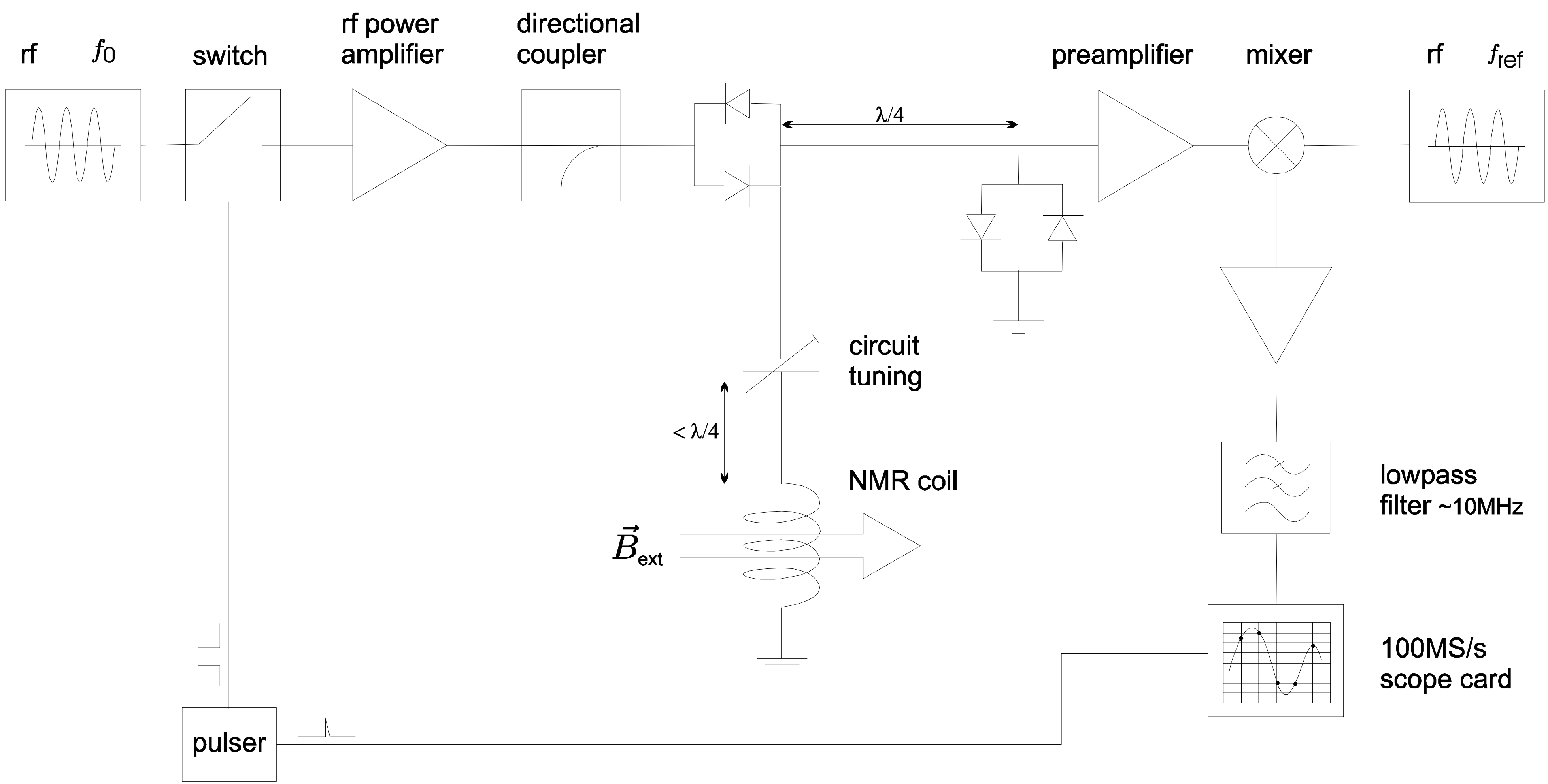


Because every signal spin starts its precession motion, the transverse magnetization also precesses in the *xy*-plane, perpendicular to the magnetic field.

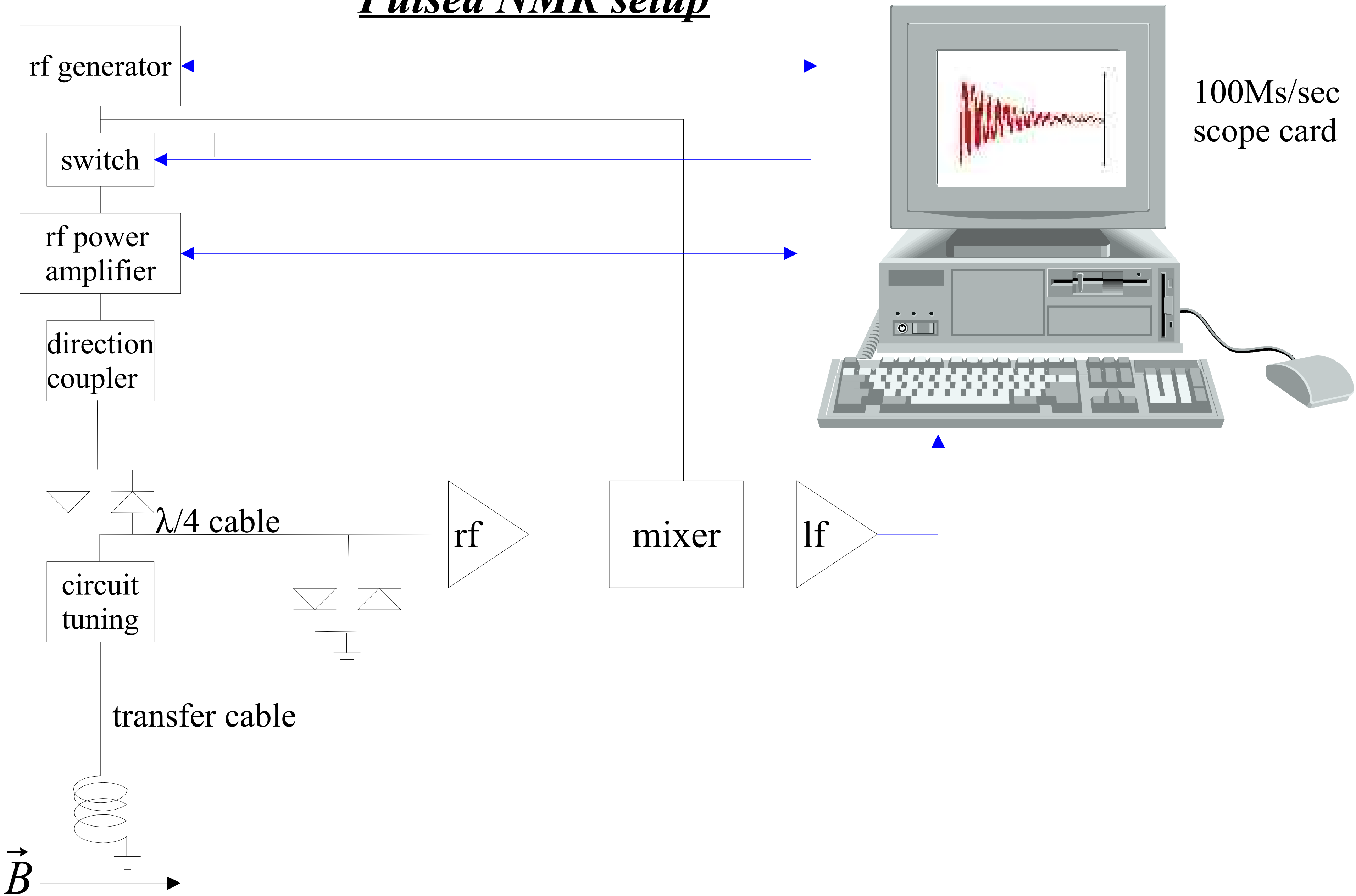


The rotating net magnetic moment induces an oscillating signal in the receiver coil
→ **free-inductance decay (FID)**

Pulsed NMR setup

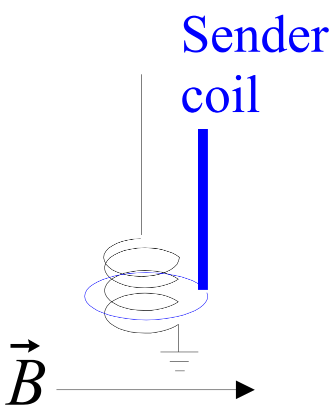


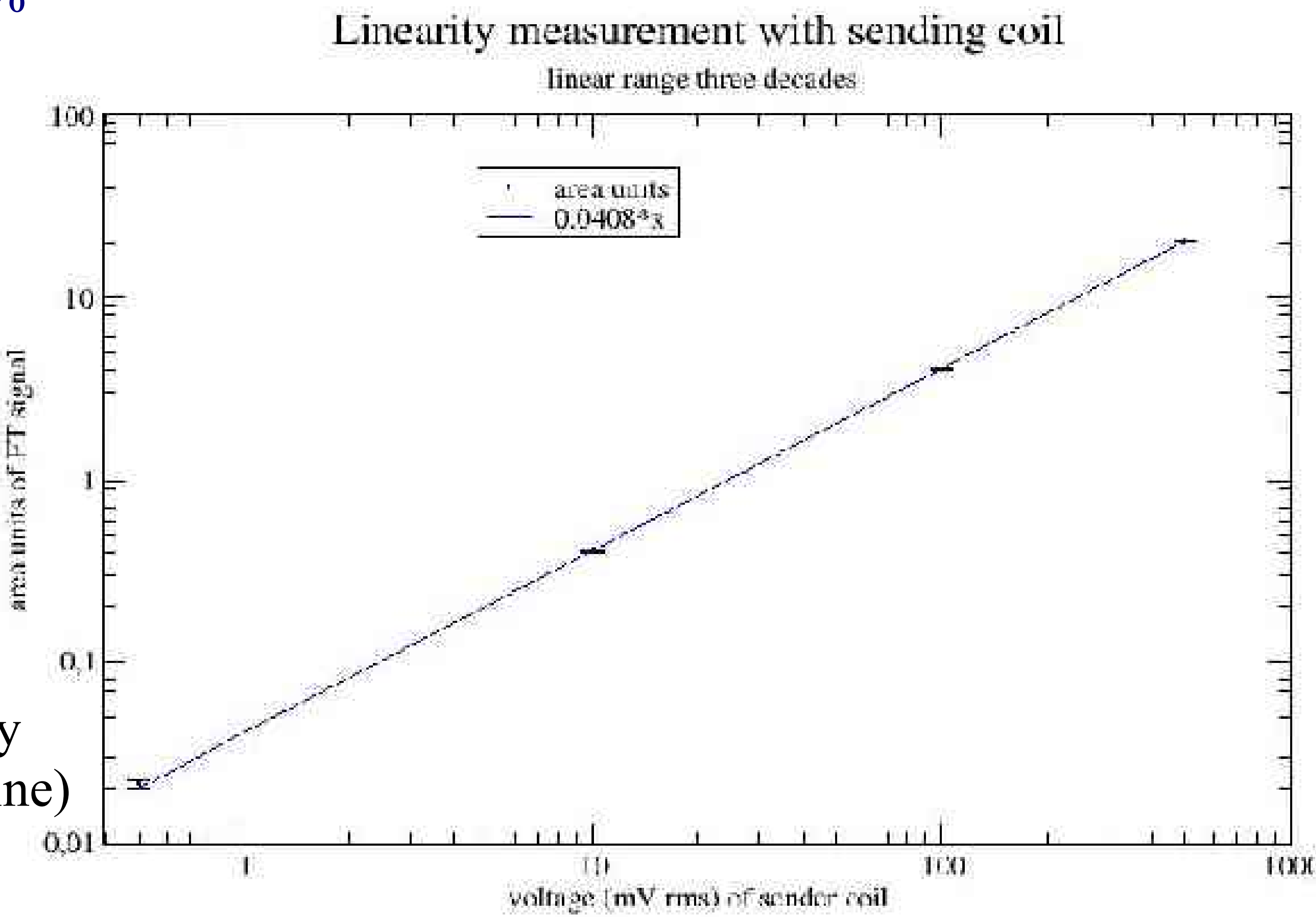
Pulsed NMR setup



Linearity measurement with sending coil

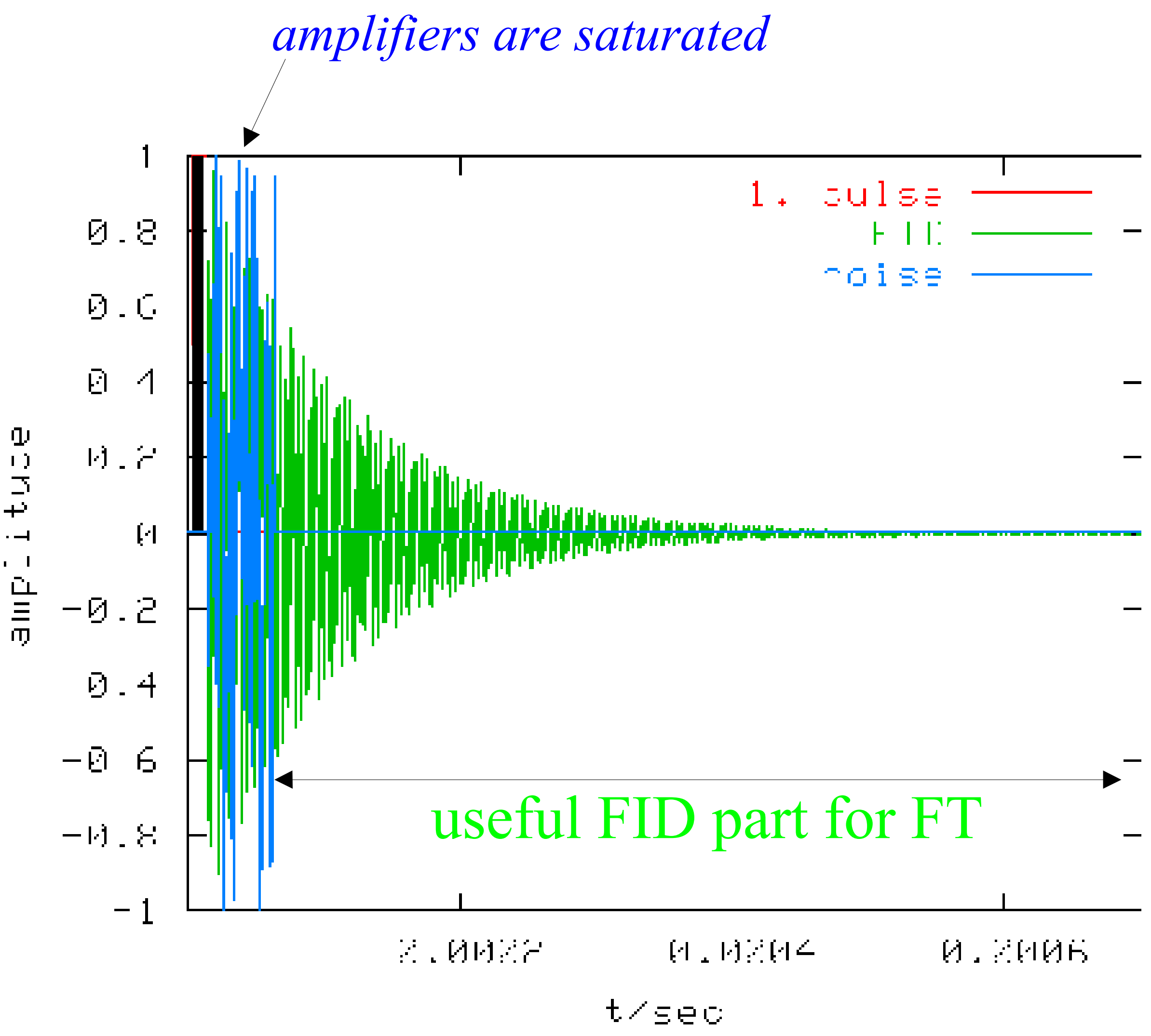
$$\frac{P_{dyn}}{P_{TE}} = \frac{\geq 80\,\%}{0.052\,\%} \geq 1600$$



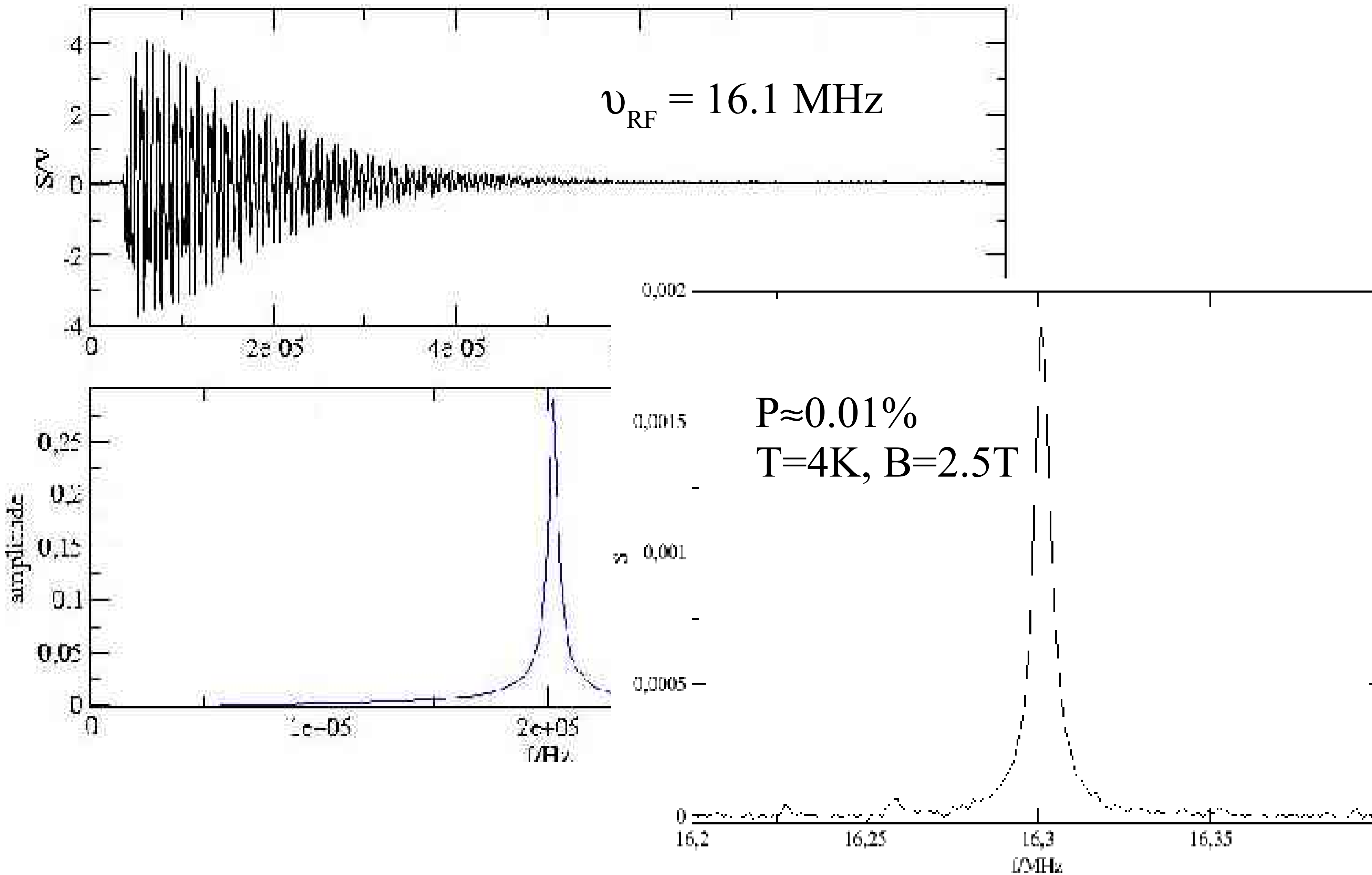


Linearity range can be extend by reducing t_P (online)

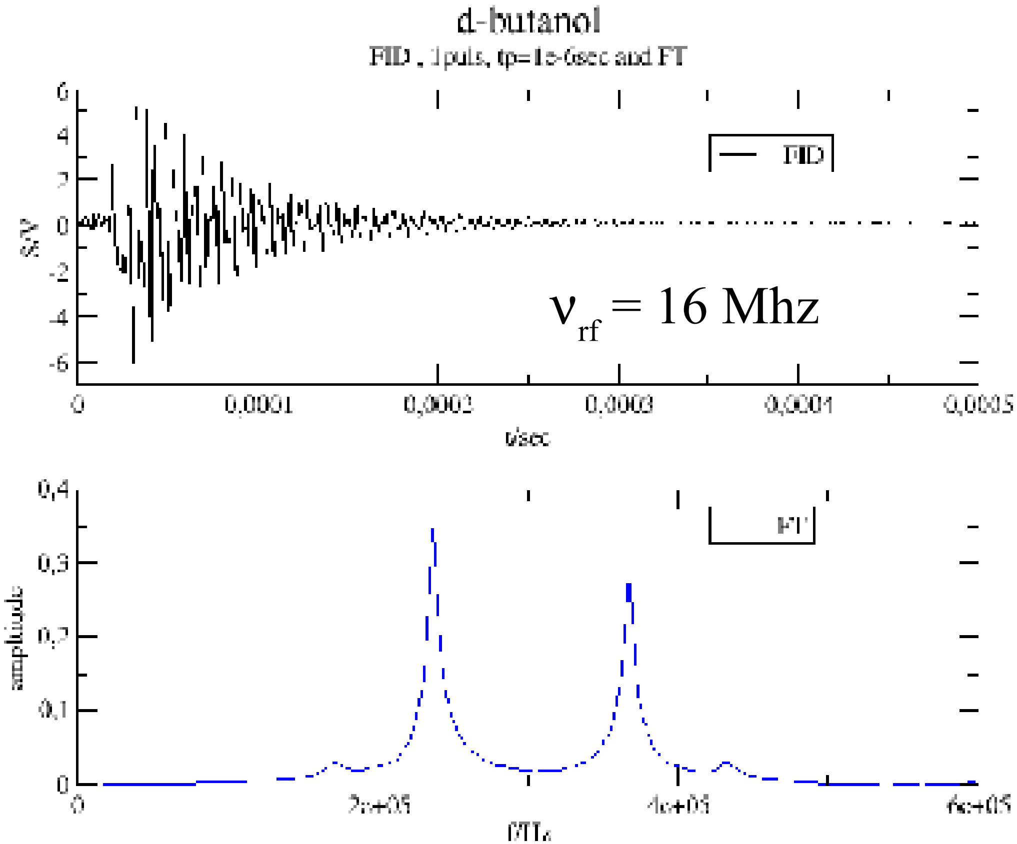
Signal detection



^{6}LiD, t_P = 200nsec, FID and FT, P ≈ 0.5%



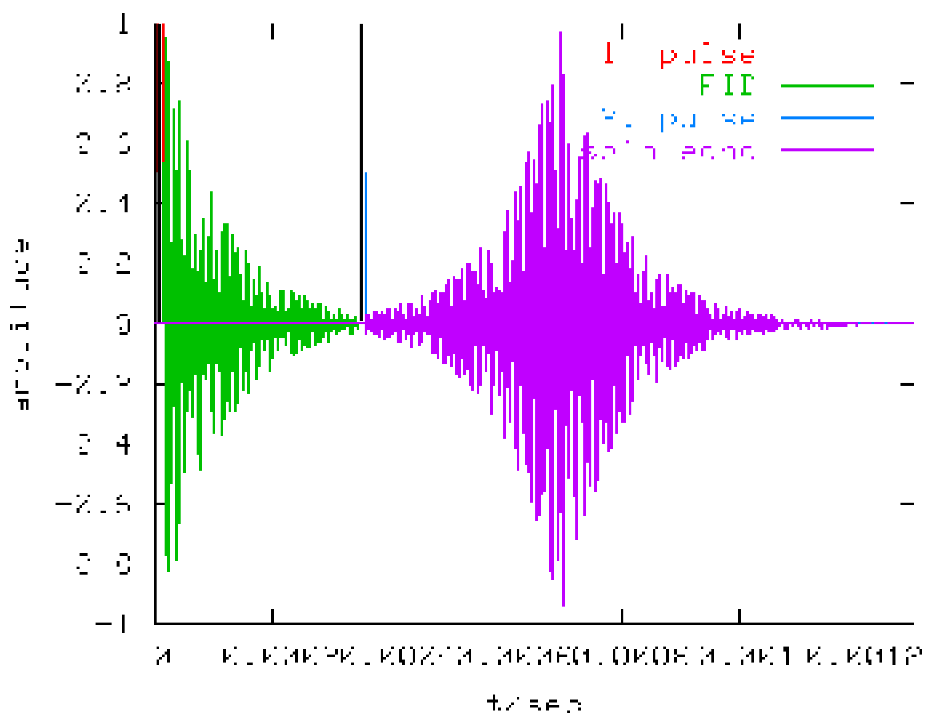
Quadrupol effects



To optain the lineshape of the quadrupol splitting in the correct way, one has to detect the

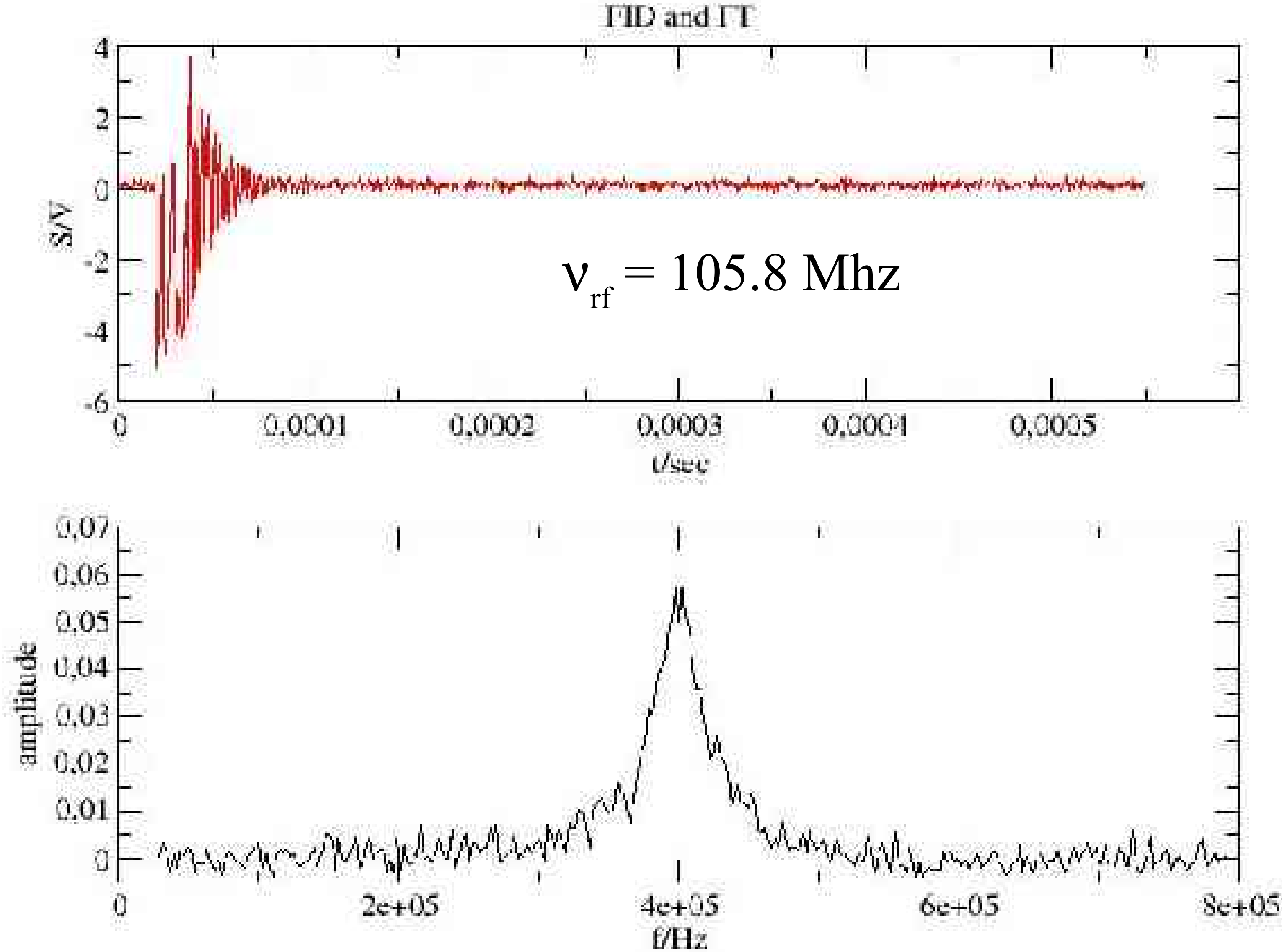
'spin echo'

after a second pulse.

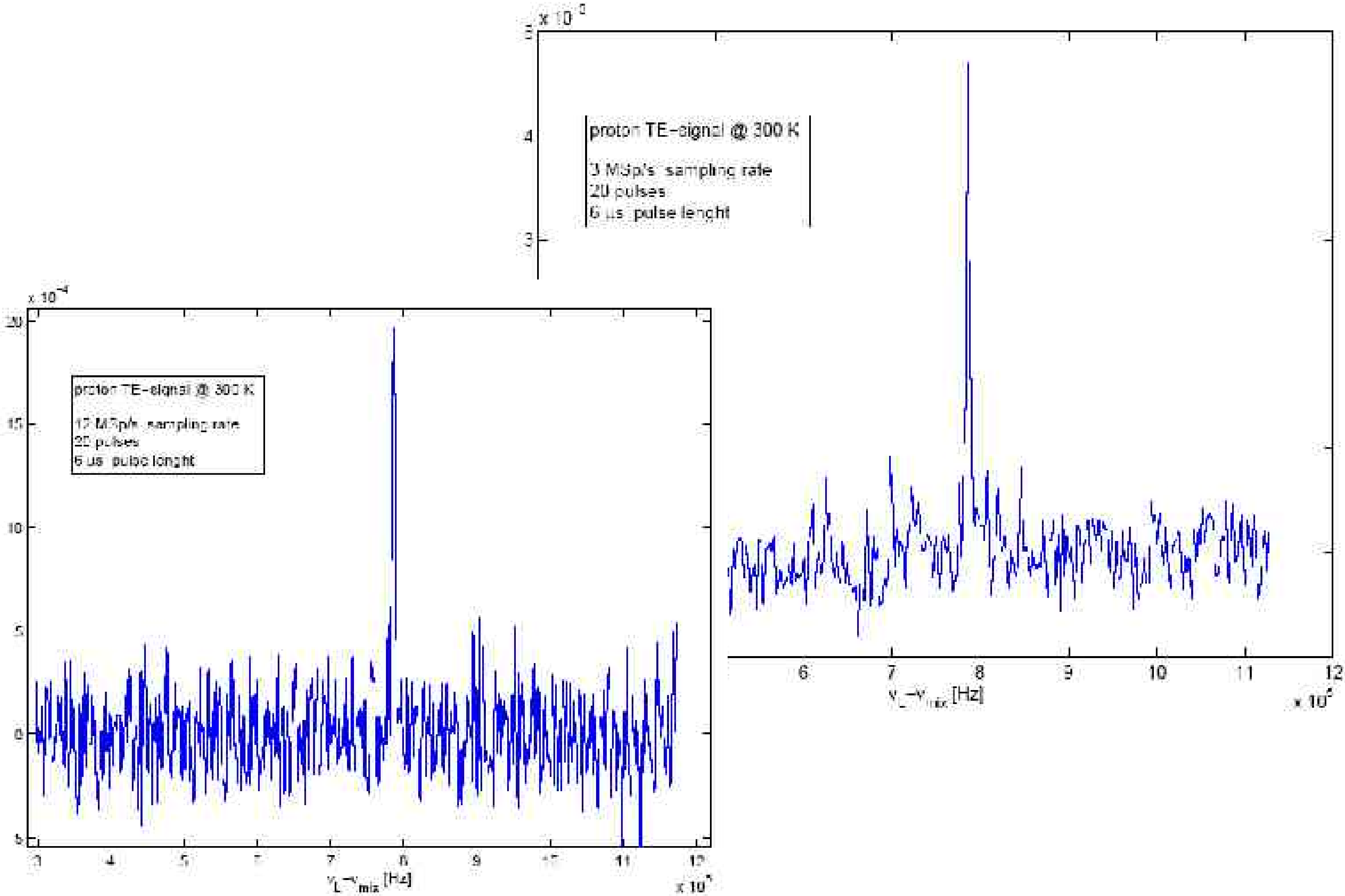


A finite time after the pulse the preamplifier is saturated and the detected FID is unusable, but the whole FID must be Fourier trans-formated to optaine the complete line shape.

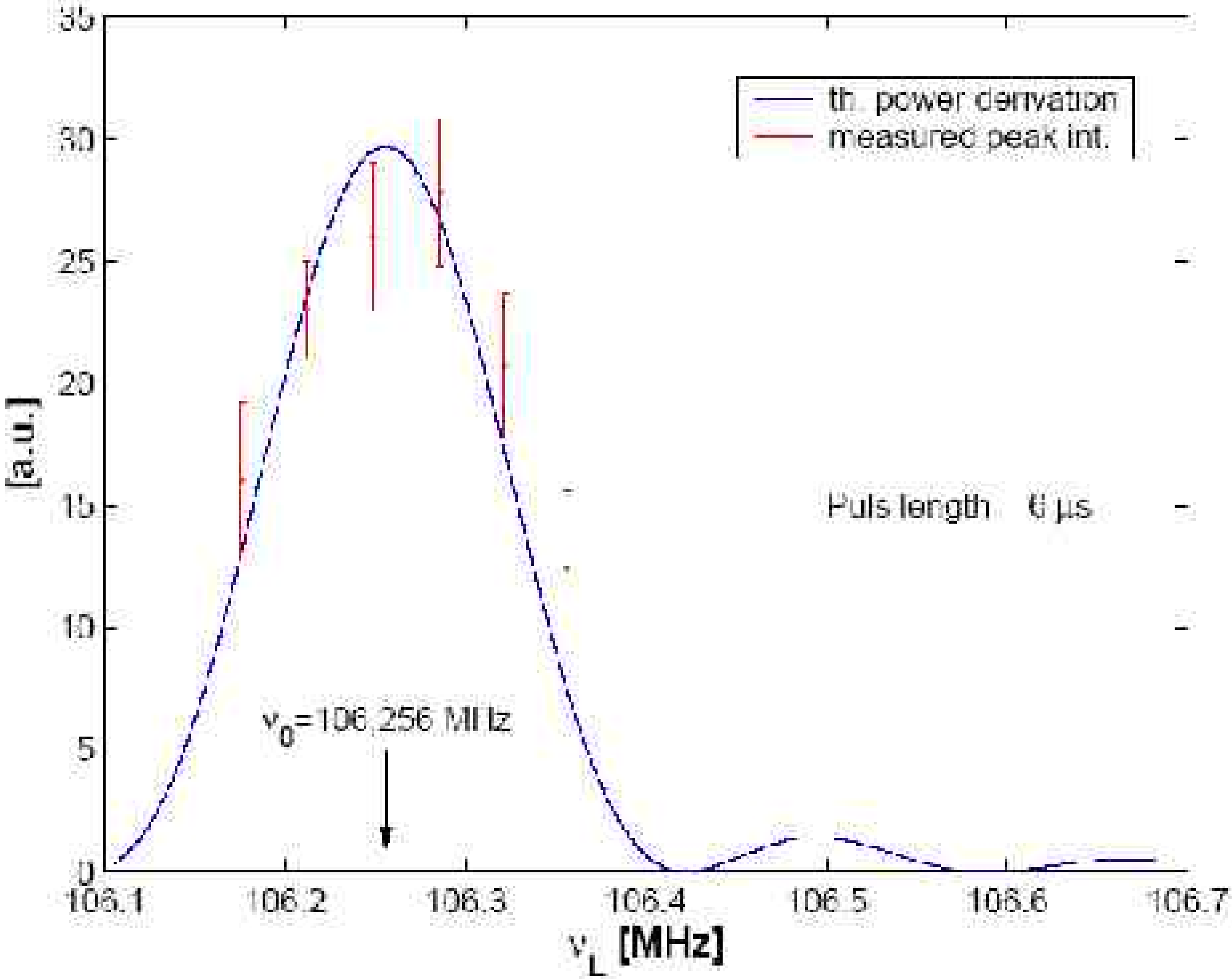
Residue protons in d-butanol **(0.5%)**



Proton signals of Butanol at room temperature!



Power derivation versus frequency



Summery

- Pulse NMR was build to test their application in polarisation measurement

- Linearity measurements with sending coil are made and a linearity over 3 decades was obtained

Conclusion

- When obtaining the FID the one pulse NMR is only useful for narrow signals (^{6}LiD)

- In flexibility and speed the pulse NMR is the better choice

- With the same depolarising amplitude the sensitivity is approximately the same for both methods ?!

Outlook

- In the near future <u>multi pulse</u> option will be implemented to acquire signals with wide line shape