NMR Spectroscopy of Organic Compounds

Lesson 1:
1D NMR pulse sequences

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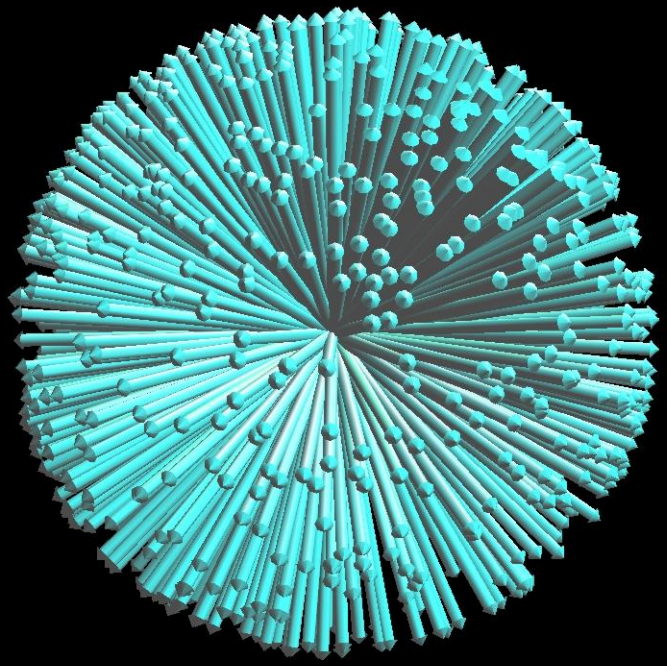
Overview

- Basic 1D pulse sequences
- Relaxation
- Spin echo

Magnetization



$\vec{M} = 0$

Random orientation of magnetic moments

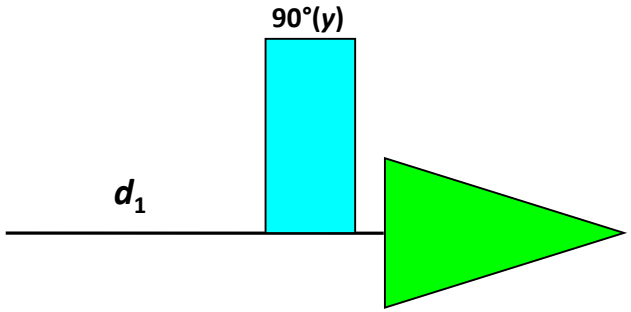
$\vec{M} = (0, 0, M_0)$

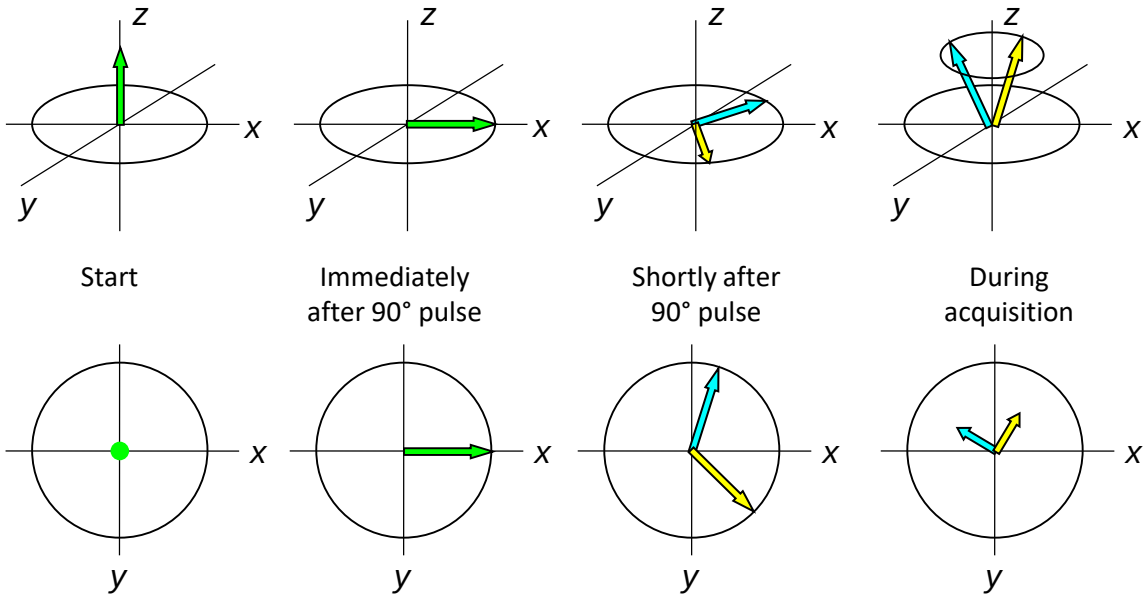
Larmor precession, magnetization

$\nu = \gamma \boldsymbol{B}_0 / 2\pi$

Basic 1D pulse sequences

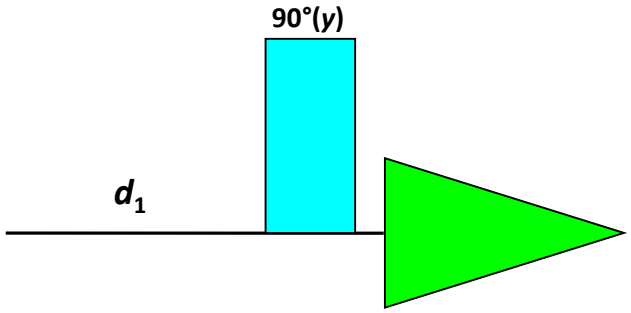
Measurement of ^{1}H, ^{19}F, …



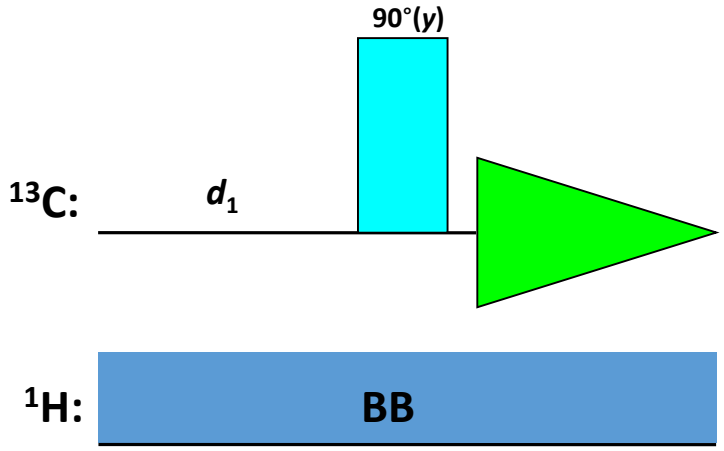


Basic 1D pulse sequences

Measurement of ^{1}H, ^{19}F, …

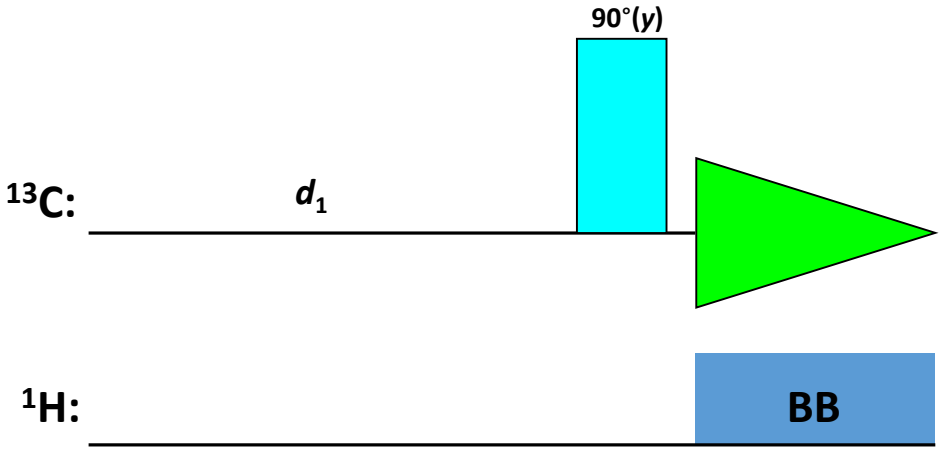


Decoupling – measurement of ^{13}C, ^{31}P, …

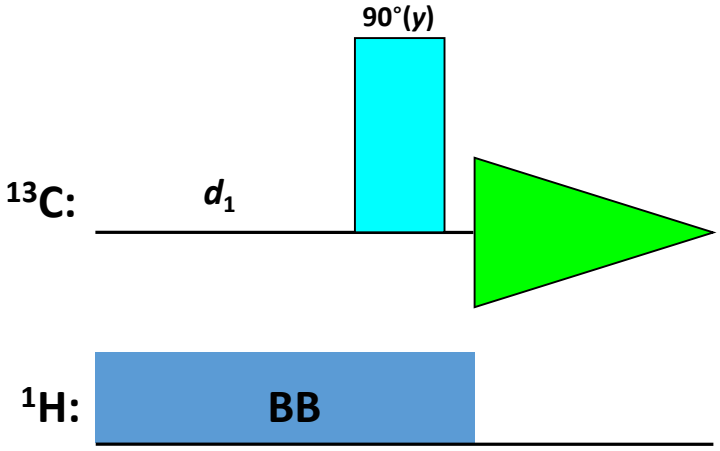


Basic 1D pulse sequences

Quantitative ^{13}C

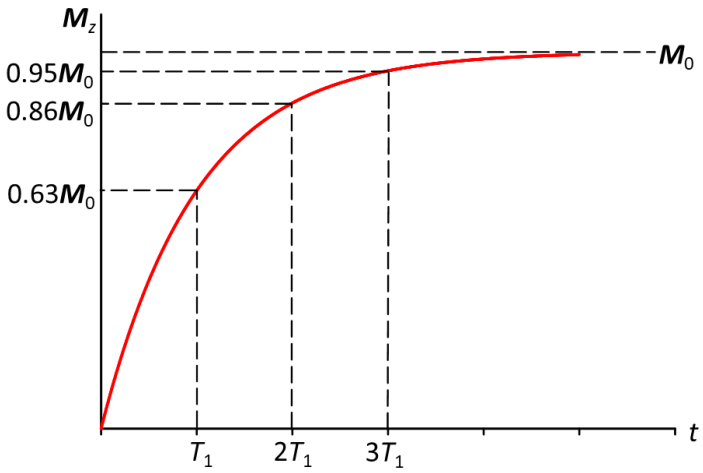


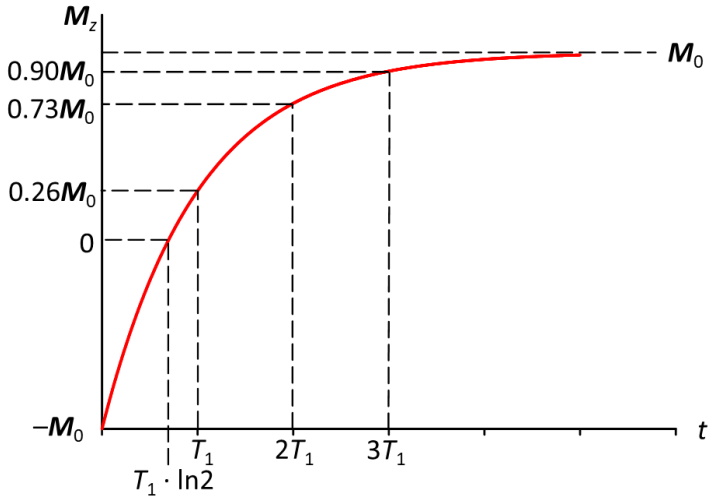
Coupled ^{13}C



Relaxation

– Longitudal (T_1) – building of magnetization along *z* axis



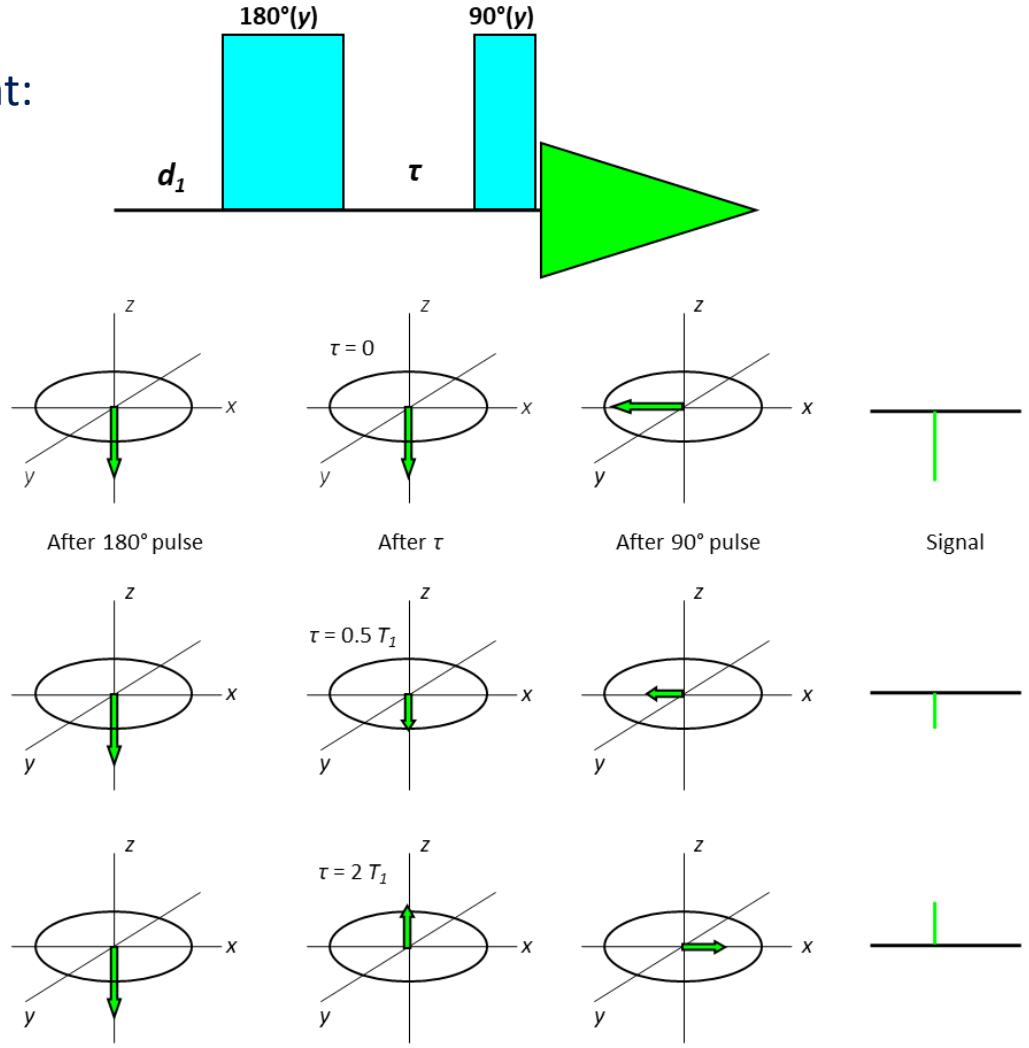


$$M_z = M_0 \left(1 - e^{-t / T1}\right)$$

Relaxation

– Longitudal (T_1) measurement:

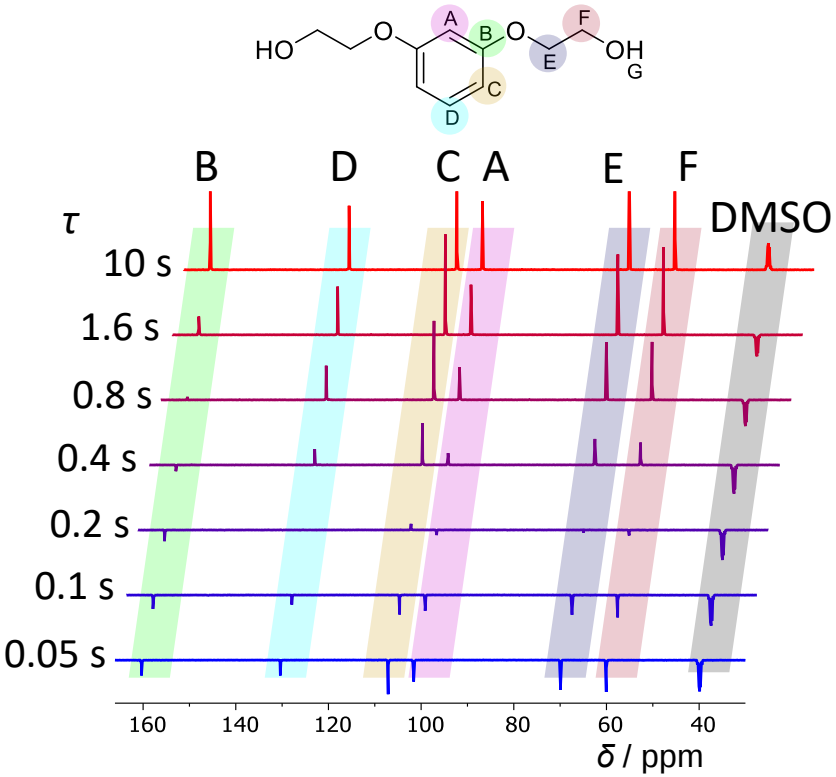
inversion recovery

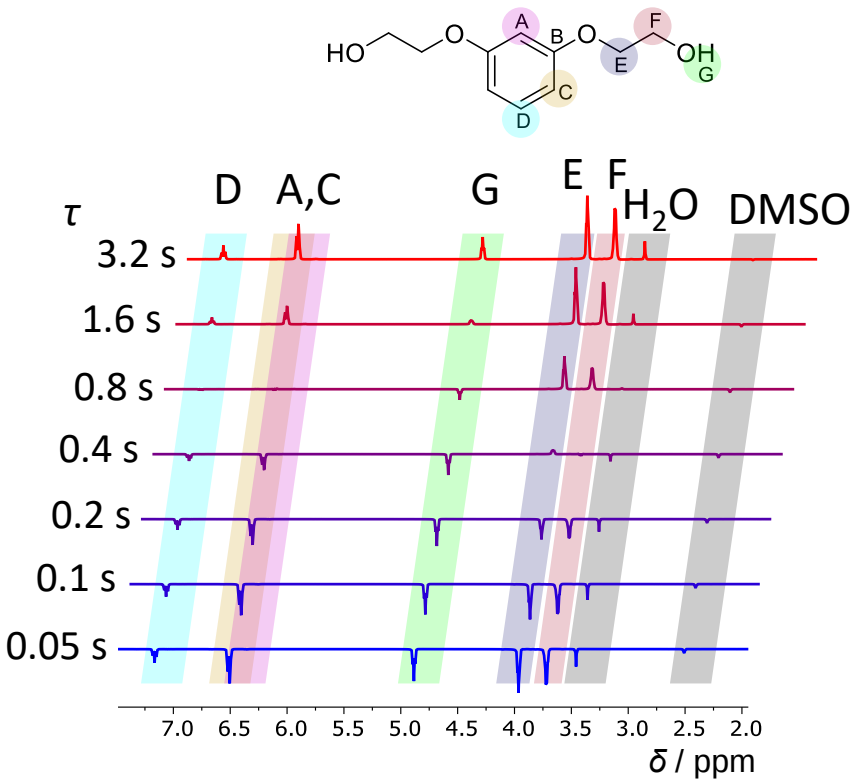


Relaxation

– Longitudal (T_1) measurement: inversion recovery

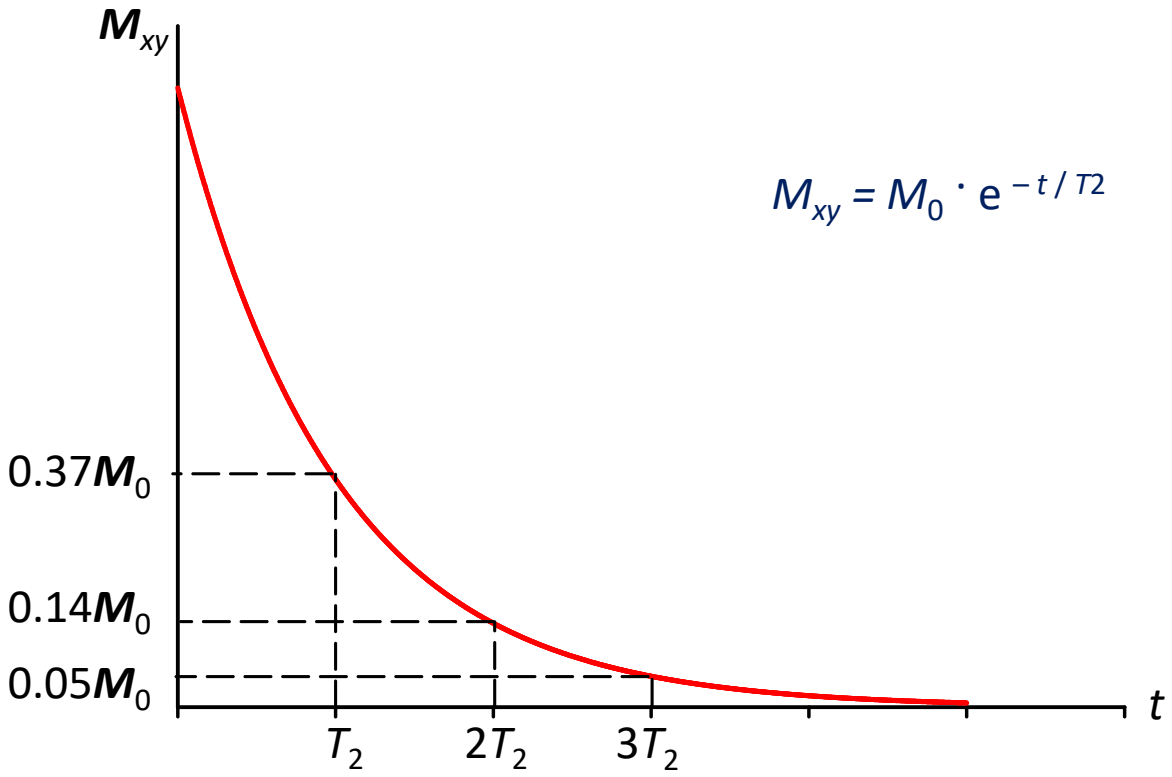
Ernst angle: $\cos \alpha = \exp(-t_r/T_1)$





Relaxation

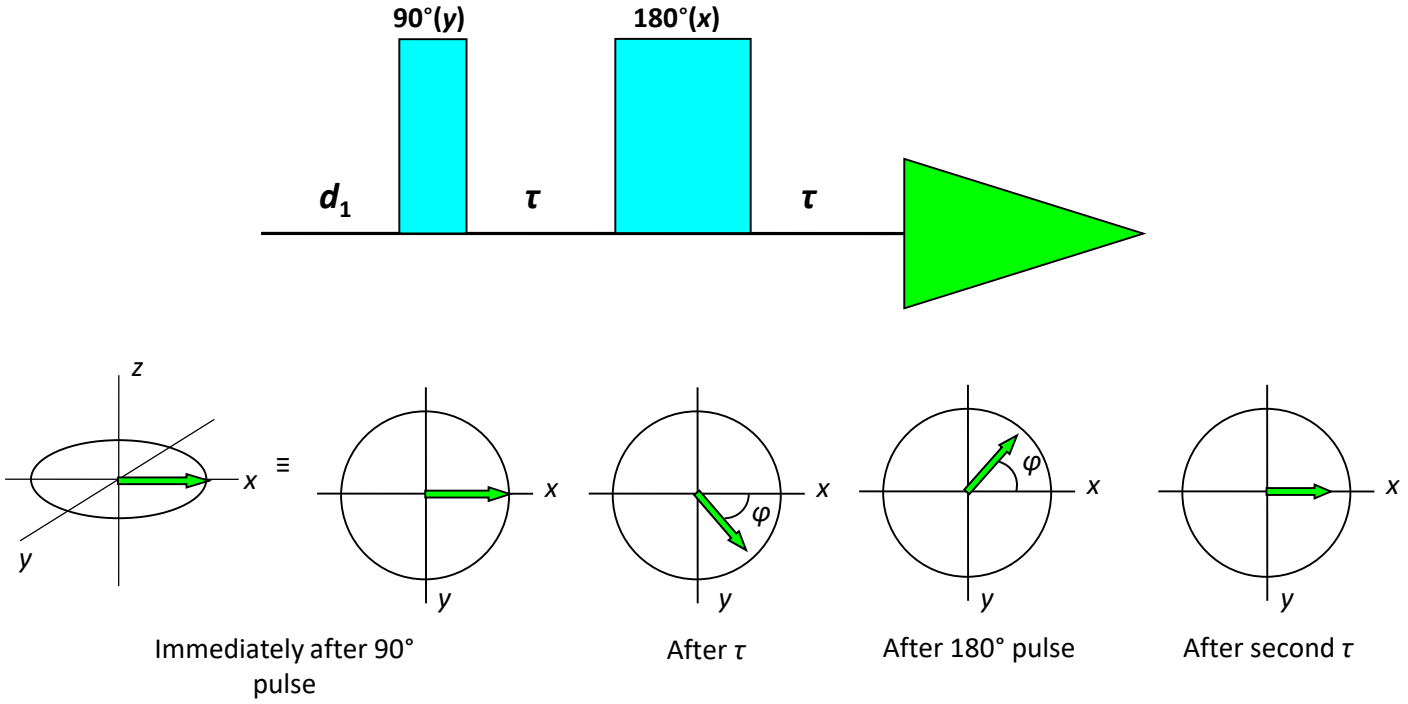
– Transversal (T_2) – loss of magnetization in *xy* plane



$$M_{xy} = M_0 \cdot e^{-t/T2}$$

Spin echo

– Measurements of T_2 relaxation times



Immediately after 90° pulse

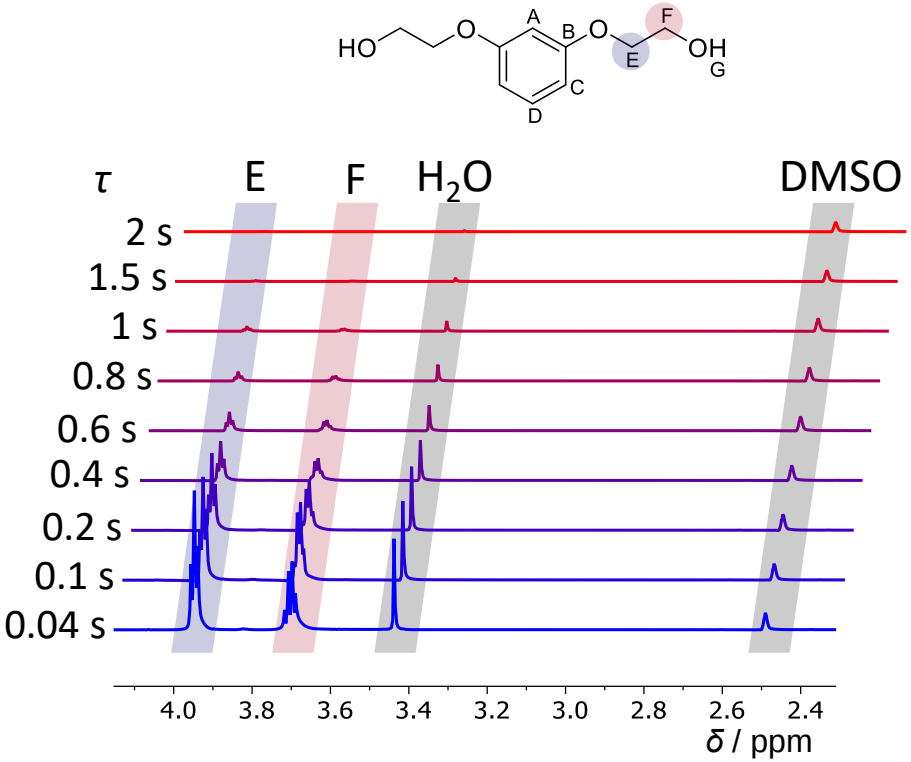
After τ

After 180° pulse

After second τ

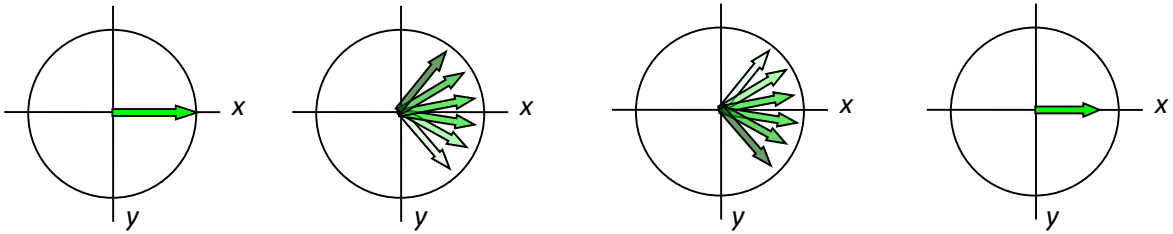
Spin echo

– Measurements of T_2 relaxation times



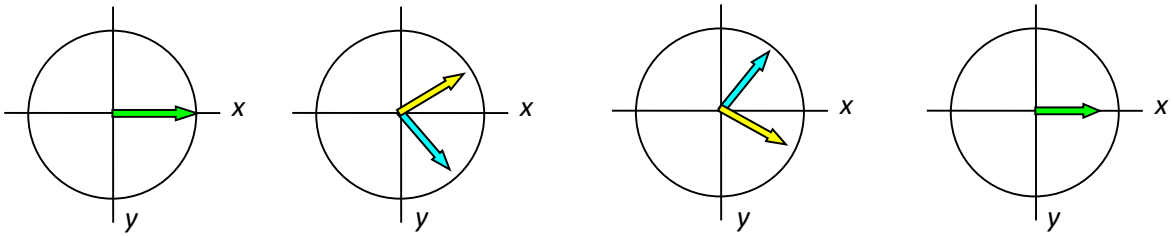
Spin echo

– Refocusing of magnetic-field inhomogeneities and of chemical shifts



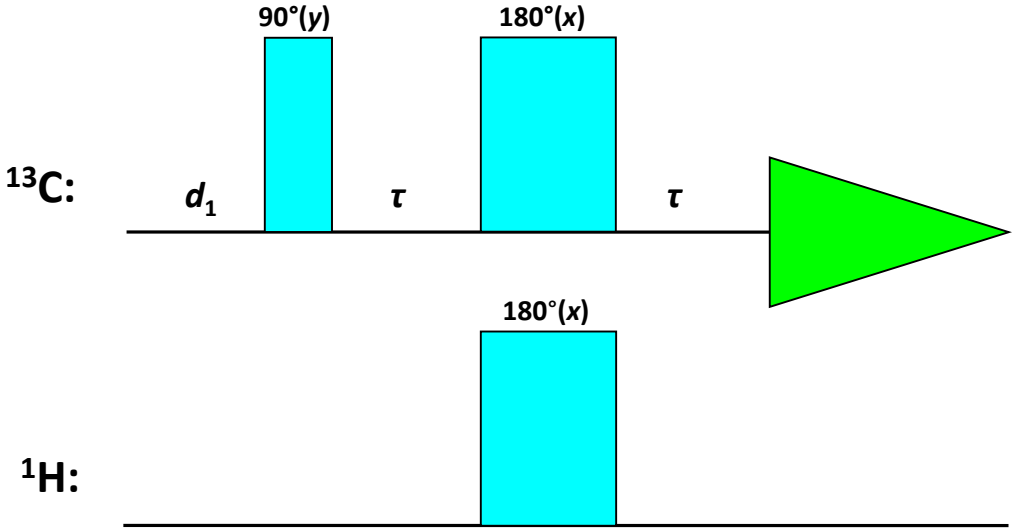
After 90° pulse | After τ | After 180° pulse | After second τ

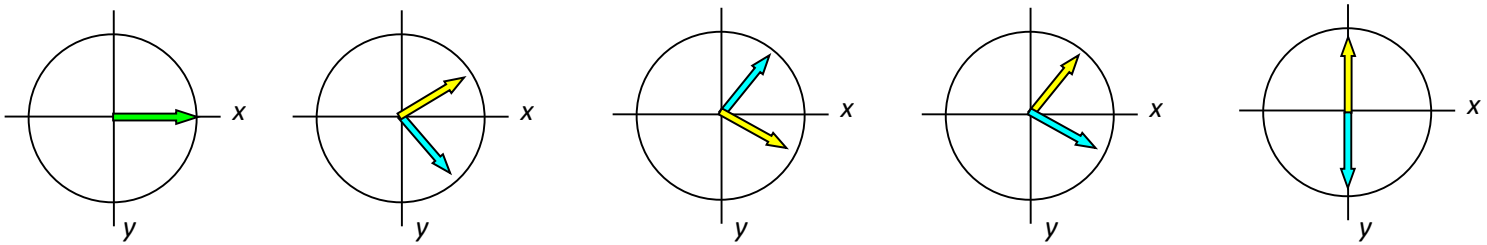
– Refocusing heteronuclear coupling



After 90° pulse | After τ | After 180° pulse | After second τ

Modified spin echo

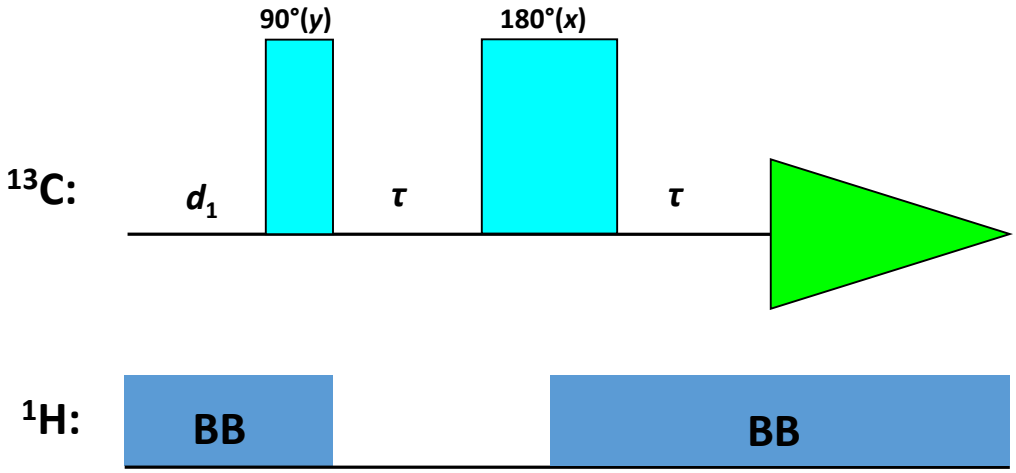




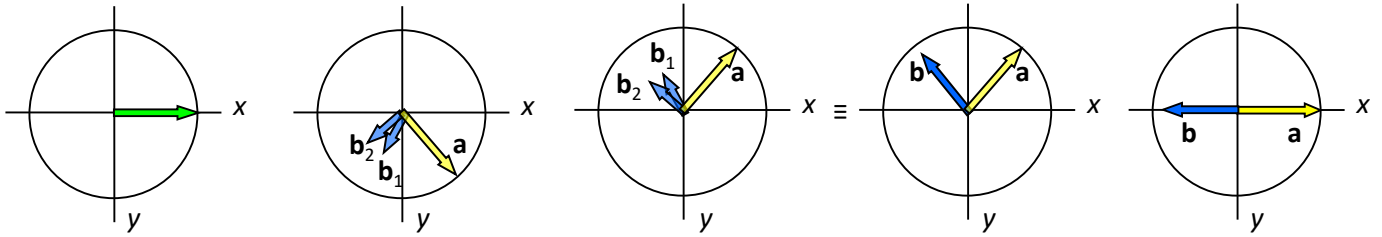
After 90° pulse | After first τ | After 180° pulse | "relabeling" of the vectors by the ^{1}H 180° pulse | After second τ

Heteronuclear couplings do not refocus

Attached proton test (APT)



$\tau \sim 1/{}^{1}J_{C\text{-}H}$

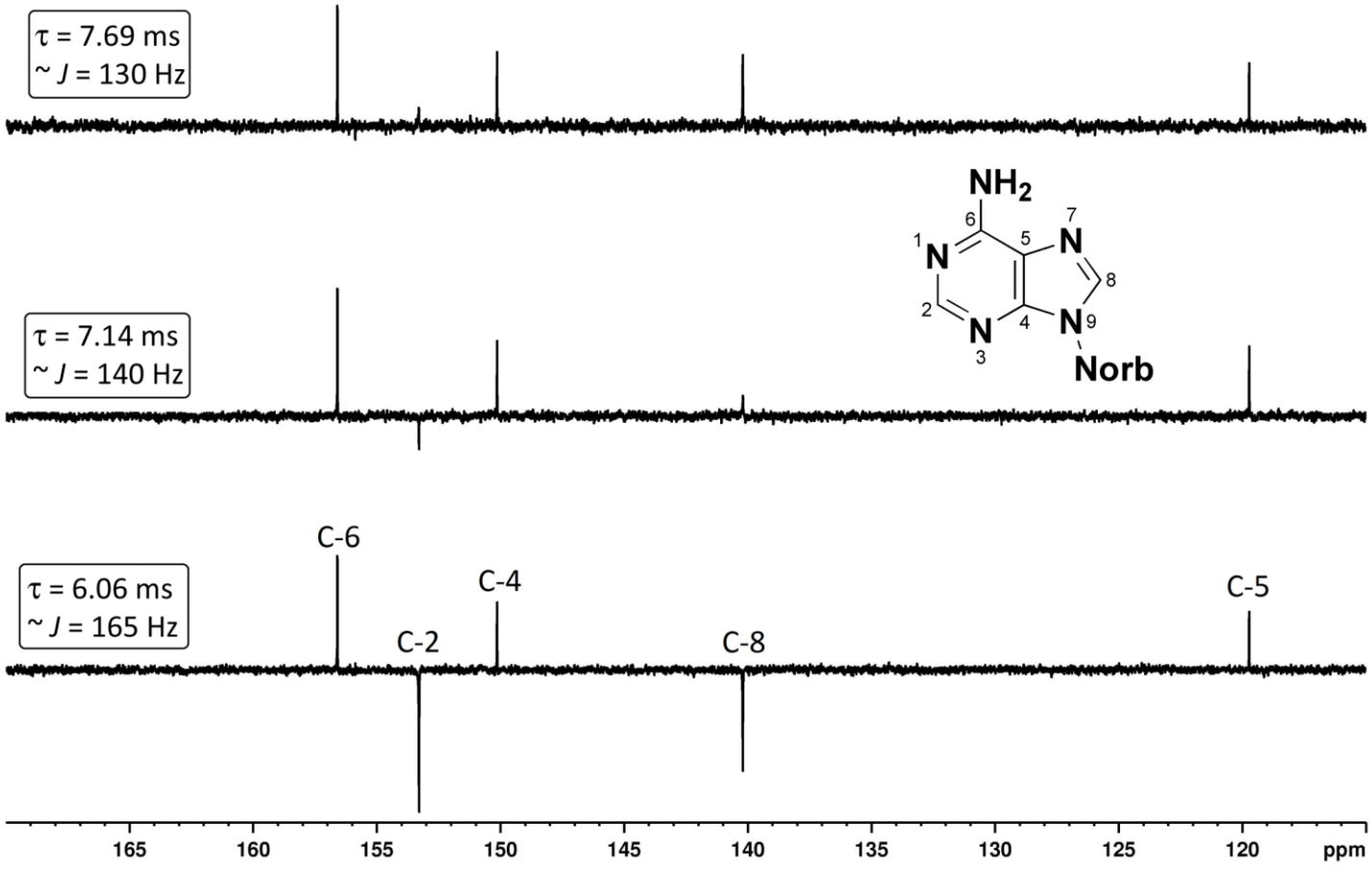


After 90° pulse | After first τ | After 180° pulse | After second τ

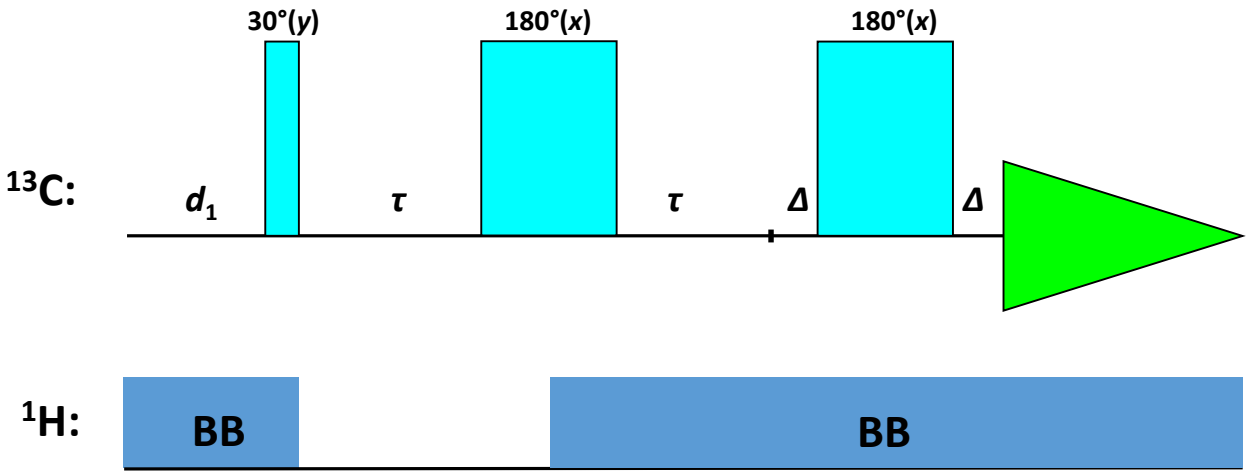
$${}^{1}J_{C-H} = 5 \cdot \%s$$

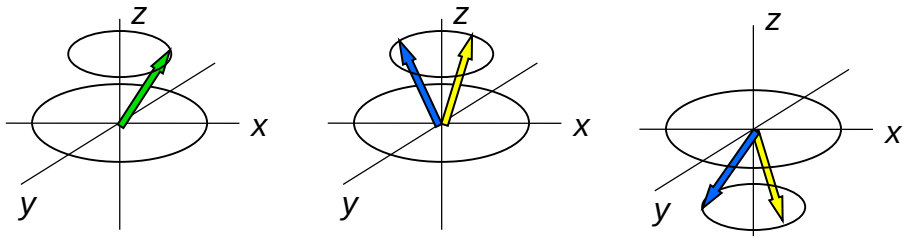
	$H_3C–CH_3$	$H_2C=CH_2$	C_6H_6	HC≡CH
Hybridization	sp^3	sp^2	sp^2	sp
%s	25	33,3	33,3	50
Calculated ${}^{1}J_{C,H}$	125	166,5	166,5	250
Experimental ${}^{1}J_{C,H}$	124,9	156,4	158,4	249,0

Attached proton test (APT)



Attached proton test (APT)





After 30° pulse

After first τ

After first 180° pulse