SNRM
Sistema Nacional de Resonancia Magnética

Ministerio de Ciencia, Tecnología e Innovación Productiva
Secretaría de Articulación Científico Tecnológica

IFEG CONICET UNC

UNC FAMAF

Principios Básicos de RMN en sólidos destinado a usuarios

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SPECTRAL EDITING

Editing

- sideband suppression
- discrimination of $C_{(quarternary)}$, CH, CH2, CH3
  - Non Quarternary Suppression (NQS)
  - Polarization/Polarization Inversion (CPPI)

Relaxation (in CPMAS experiments)

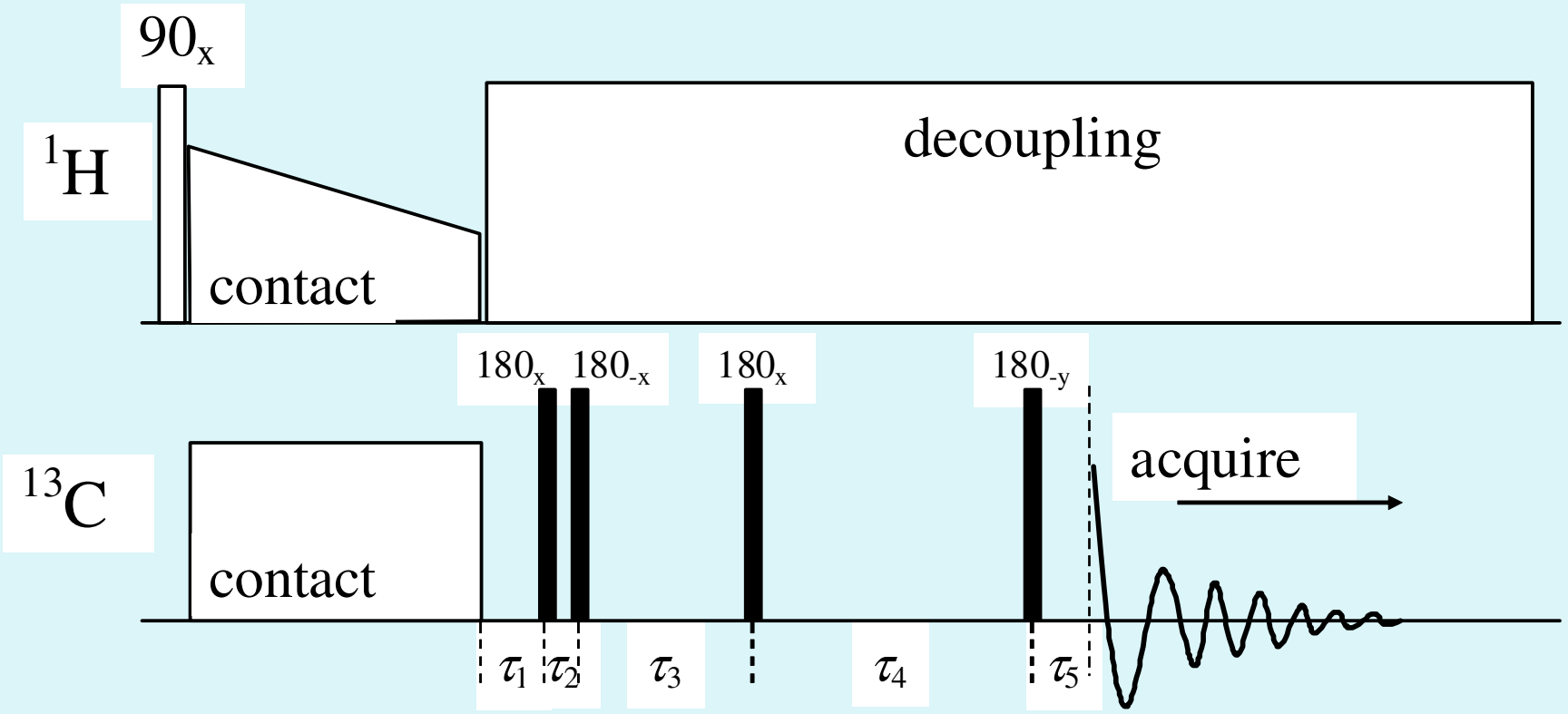
- T_1 and $T_{1\rho}$ measurement for X (e. g. 13C) using CP
- T_1 and $T_{1\rho}$ measurement for ^1H using CP

Spinning Sideband Suppression:

Spinning speed may be limited due to

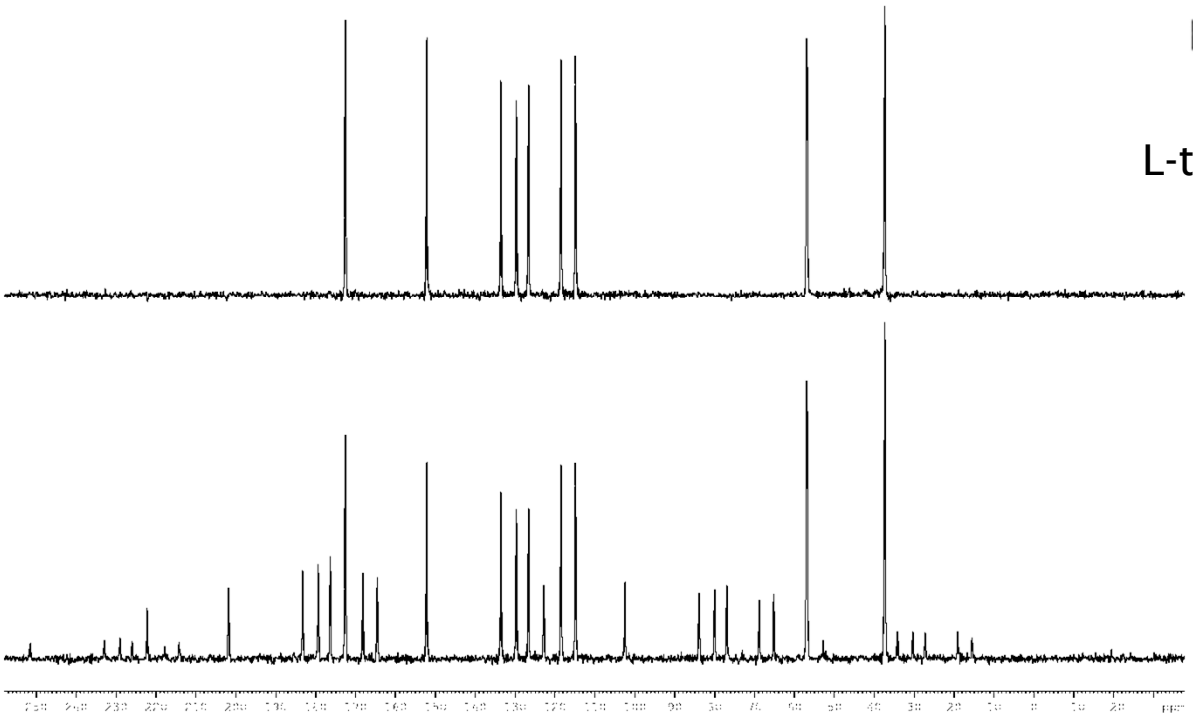
- hardware
- sample shape (e. g. spinning fibers or tablets)
- sample behaviour (e. g. phase changes under pressure)

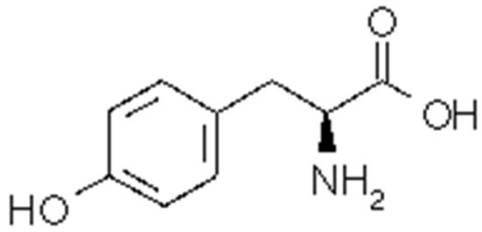
TOSS
Total Suppression of Spinning Sidebands



	τ_1/τ_{rot}	τ_2/τ_{rot}	τ_3/τ_{rot}	τ_4/τ_{rot}	τ_5/τ_{rot}	τ_{total}/τ_{rot}
TOSS A	**0.1885**	**0.0412**	**0.5818**	**0.9588**	**0.2297**	**2.0000**
TOSS B	**0.1225**	**0.0773**	**0.2236**	**1.0433**	**0.7744**	**2.2412**

TOSS
Total Suppression of Spinning Sidebands





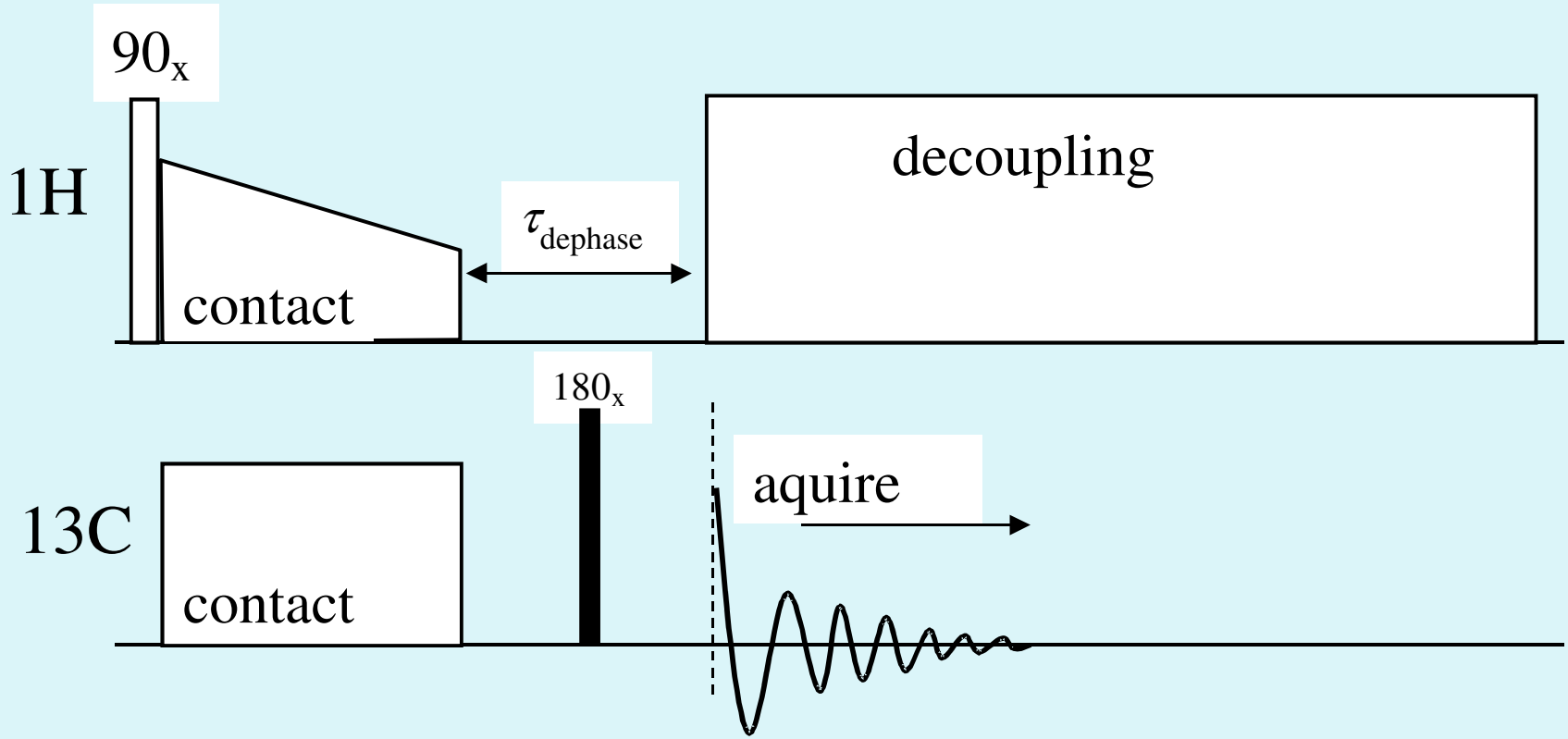
HCl

L-tyrosine hydrochloride

top:
TOSS A
4mm probe
5 kHz spinning
(t_{rot}= 200 μs)

bottom:
standard CP

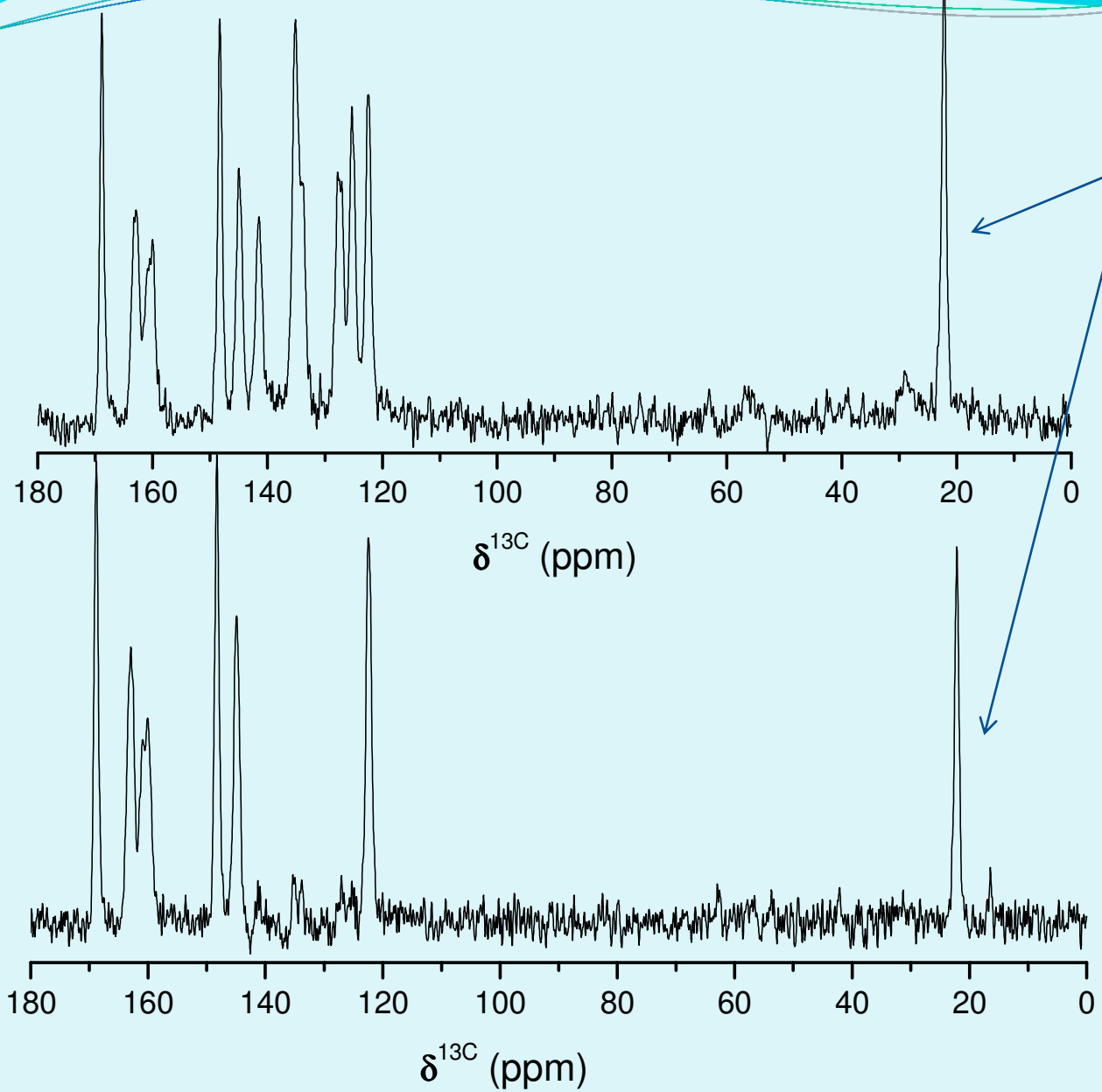
NQS: Non Quarternary Suppression



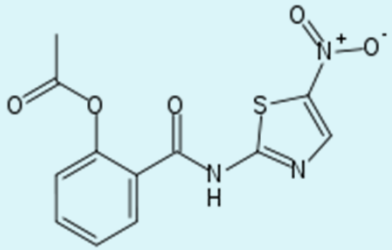
refocussed (Hahn echo) version to avoid phase distortion

$t_{dephase}$ for ^{13}C: typically 20 - 100 µs

NQS: Non Quarternary Suppression



CH3 not supressed

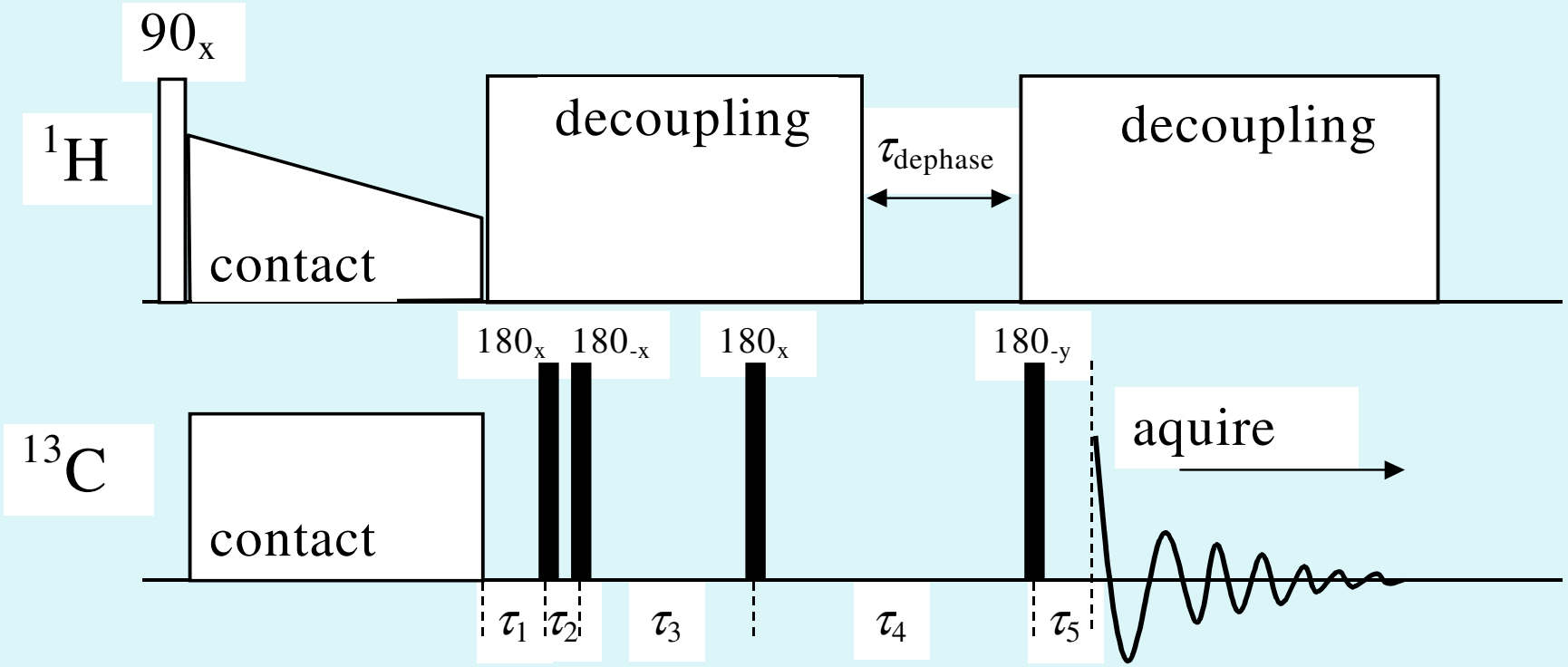


Nitazoxanide

Top: Standard CP
Bottom: NQS

40us delay

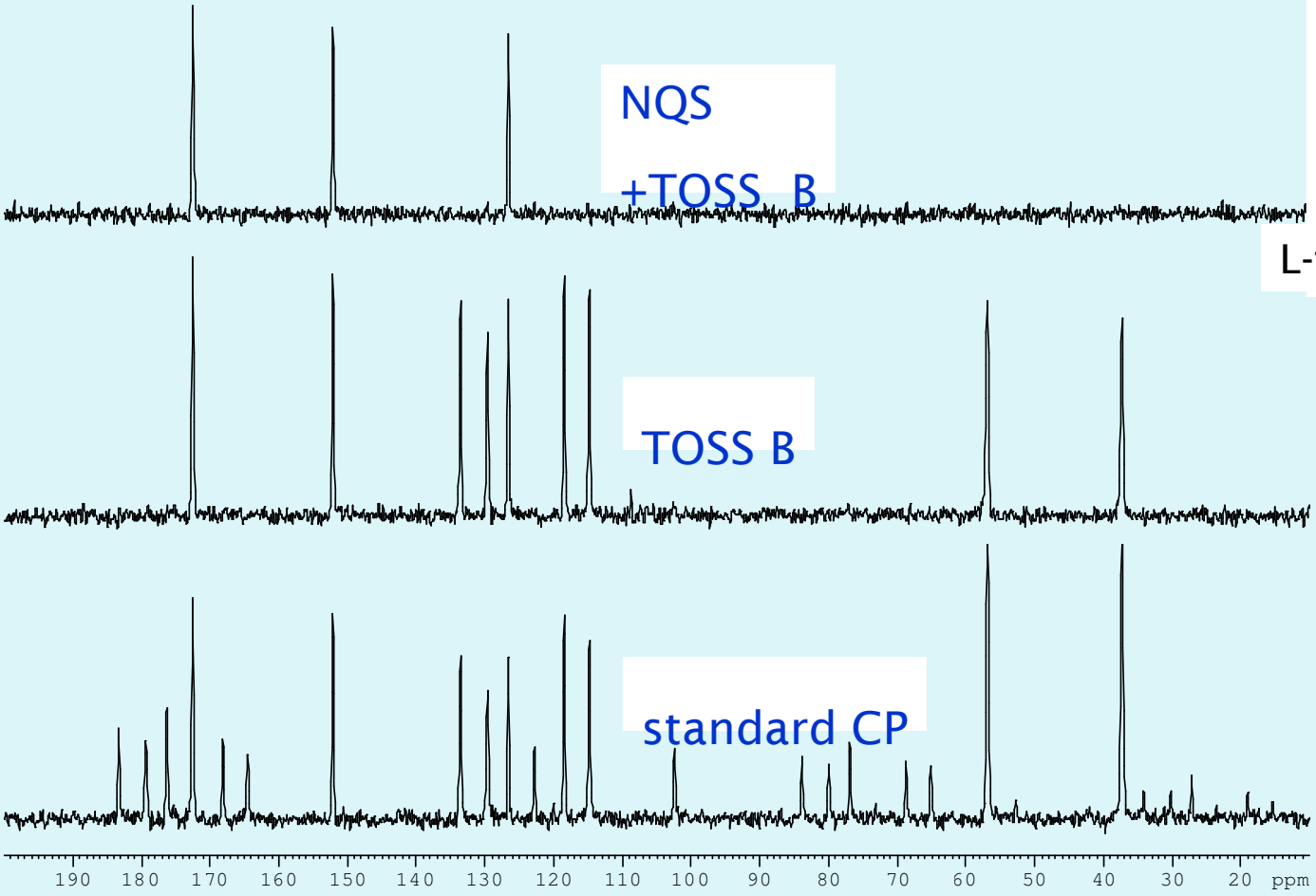
TOSS: Combined with NQS

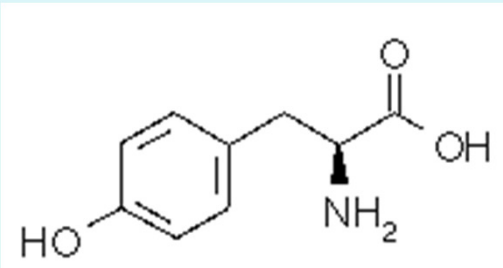


dephasing delay *within* TOSS timing

	τ_1/τ_{rot}	τ_2/τ_{rot}	τ_3/τ_{rot}	τ_4/τ_{rot}	τ_5/τ_{rot}	τ_{total}/τ_{rot}
TOSS A	**0.1885**	**0.0412**	**0.5818**	**0.9588**	**0.2297**	**2.0000**
TOSS B	**0.1225**	**0.0773**	**0.2236**	**1.0433**	**0.7744**	**2.2412**

TOSS and NQS

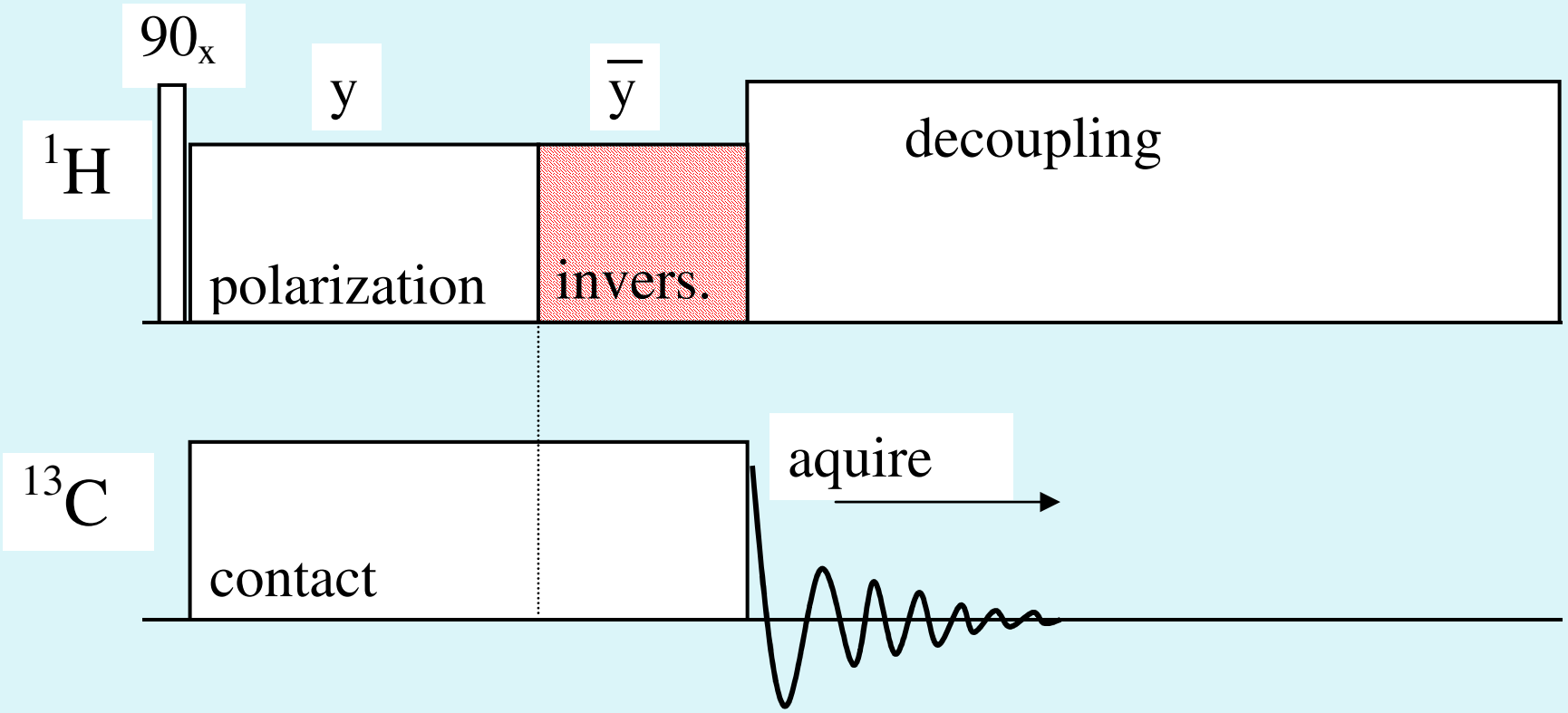




HCl

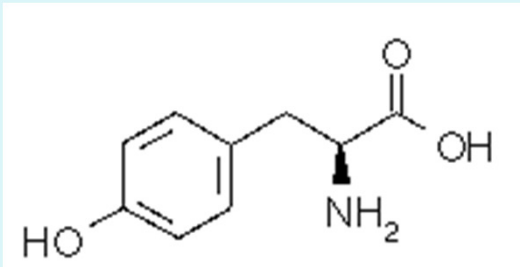
L-tyrosine hydrochloride

Polarisation/Polarisation Inversion Pulse Sequence



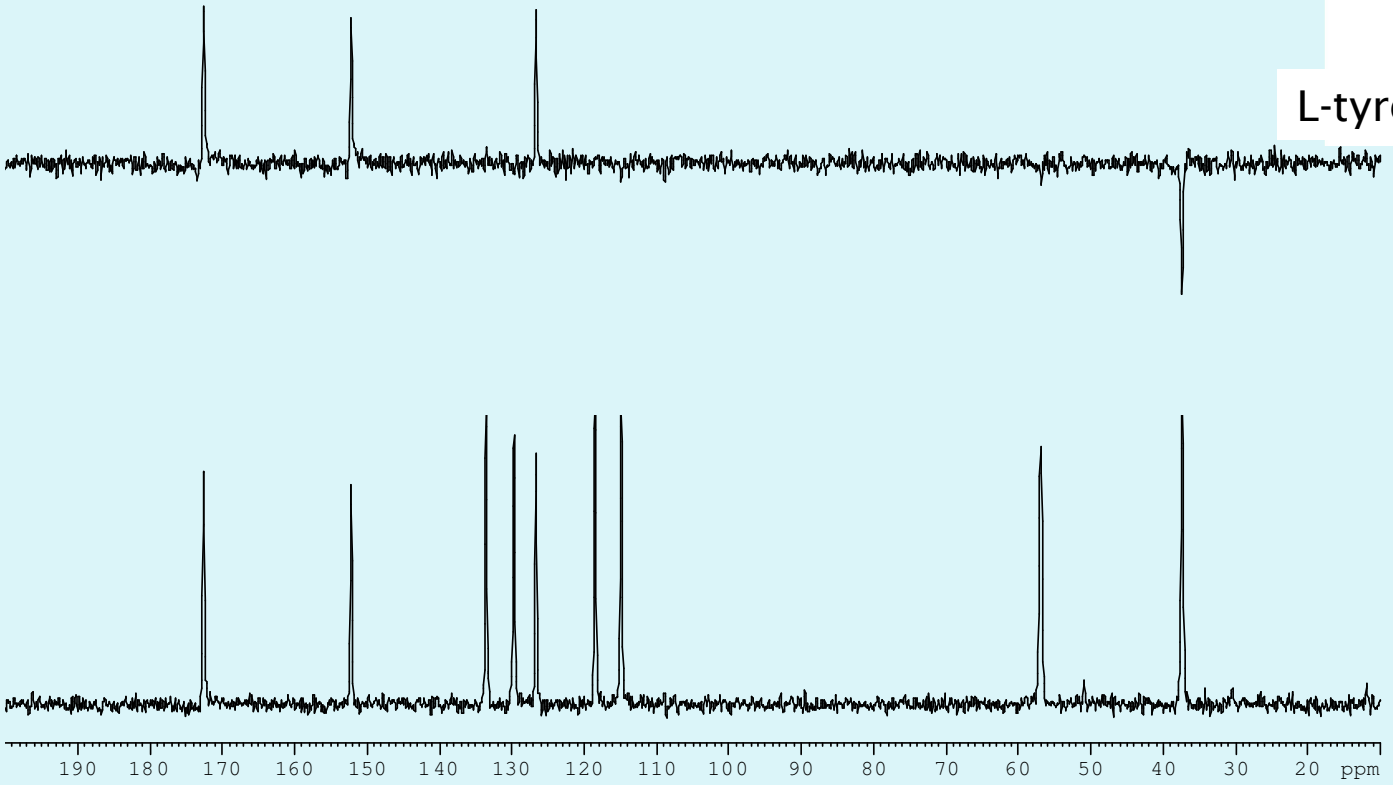
inversion time for ^{13}C: typically 20 - 400 µs

Polarisation/Polarisation Inversion



HCl

L-tyrosine hydrochloride



+: C, CH_3

0: CH

-: CH_2

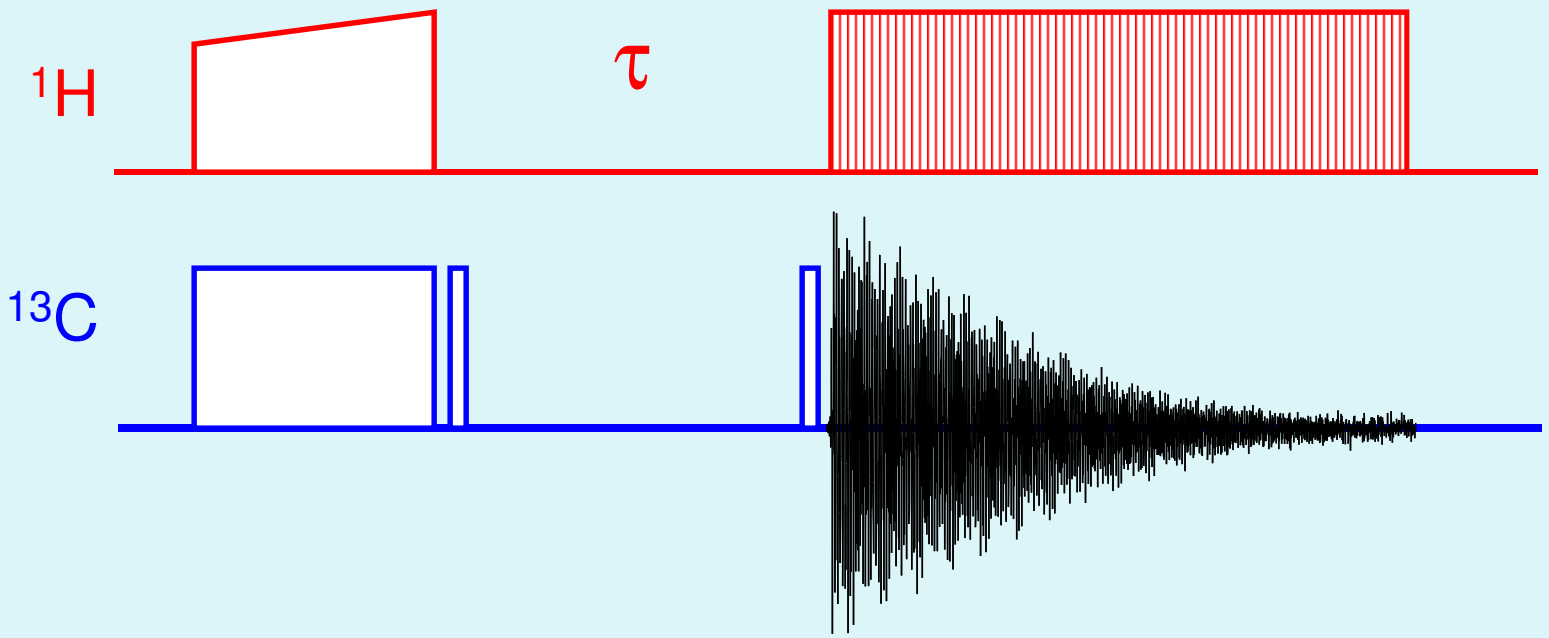
CP-Enhanced Relaxation Time Measurement

Enhancement

= sensitivity (and faster recycling) for X

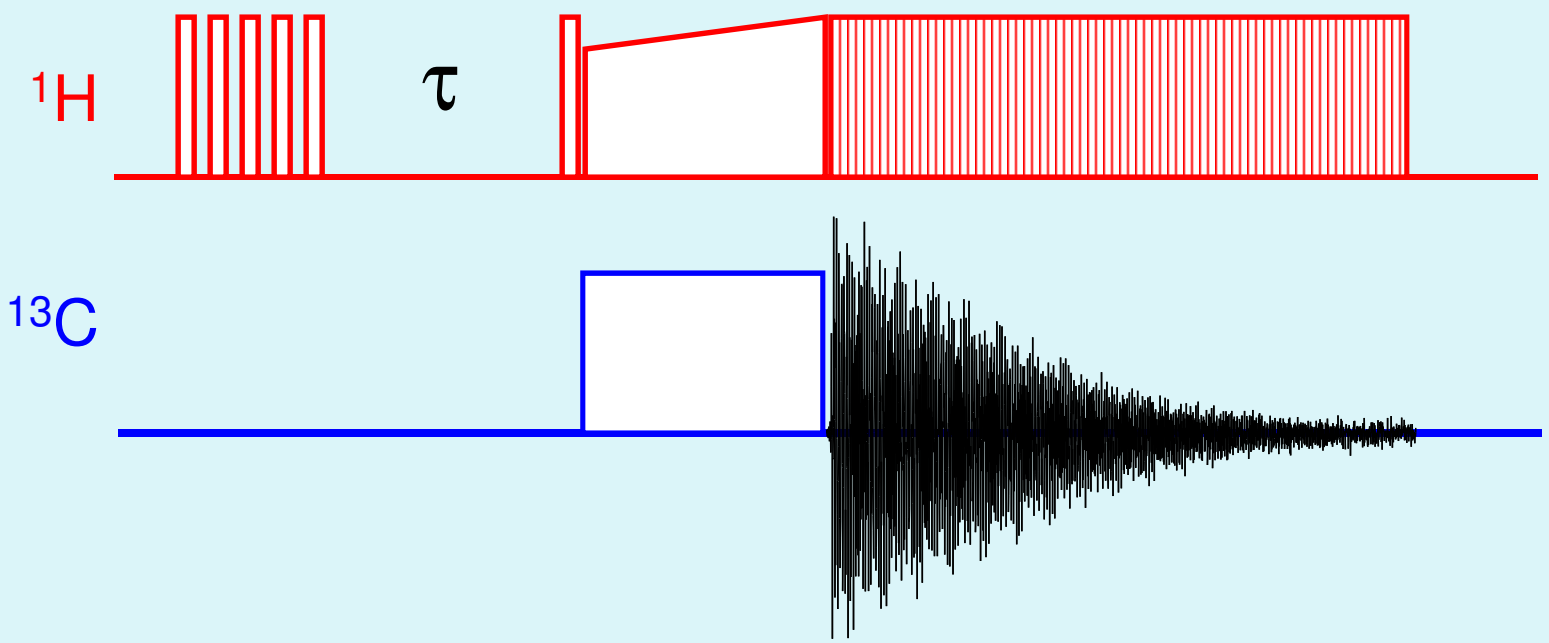
= resolution (for ^{1}H)

CP Enhanced T_1 Relaxation Pulse Sequence



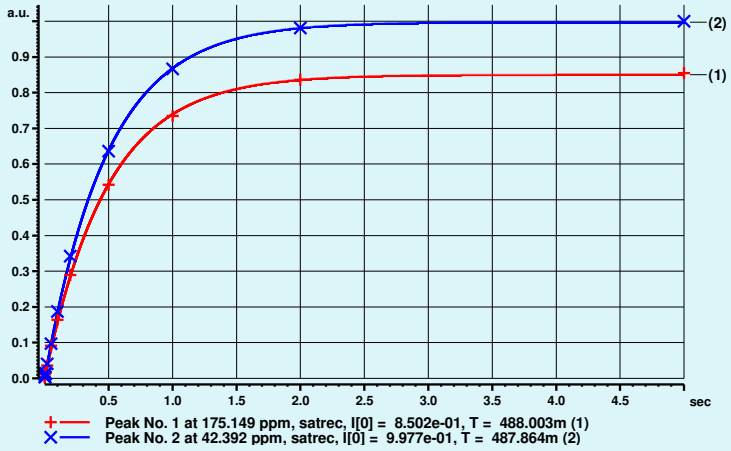
13C T1 measurement

Saturation Recovery Pulse Sequence

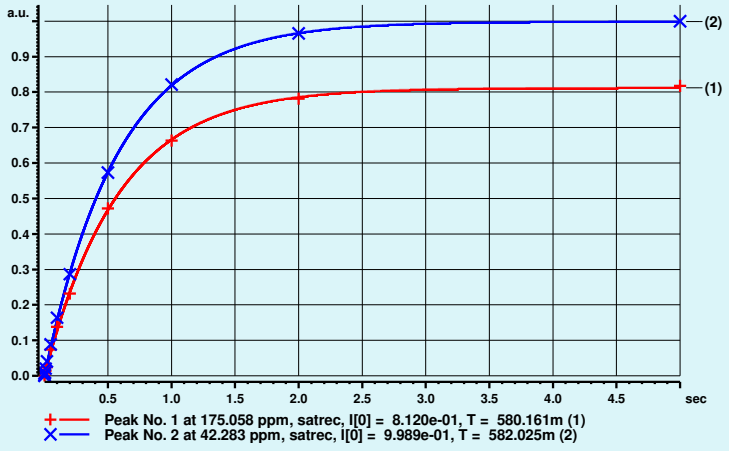


1H T1 measurement via 13C spectrum –resolution
Drawback: spin diffusion

Glycine ^{1}H T_1 Relaxation via CP to ^{13}C

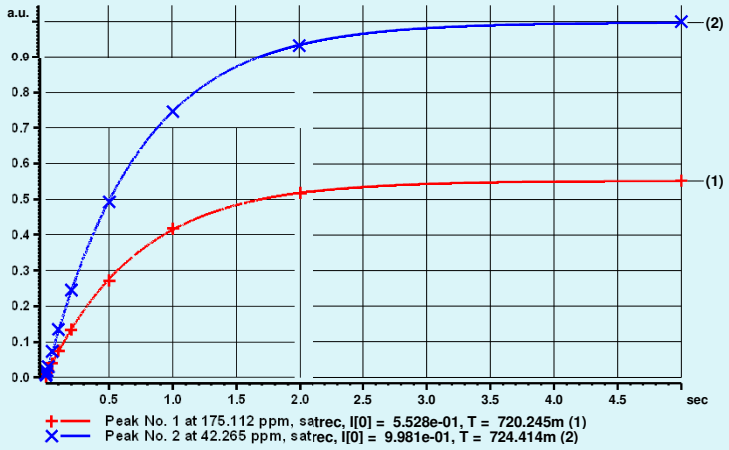


12.5 kHz



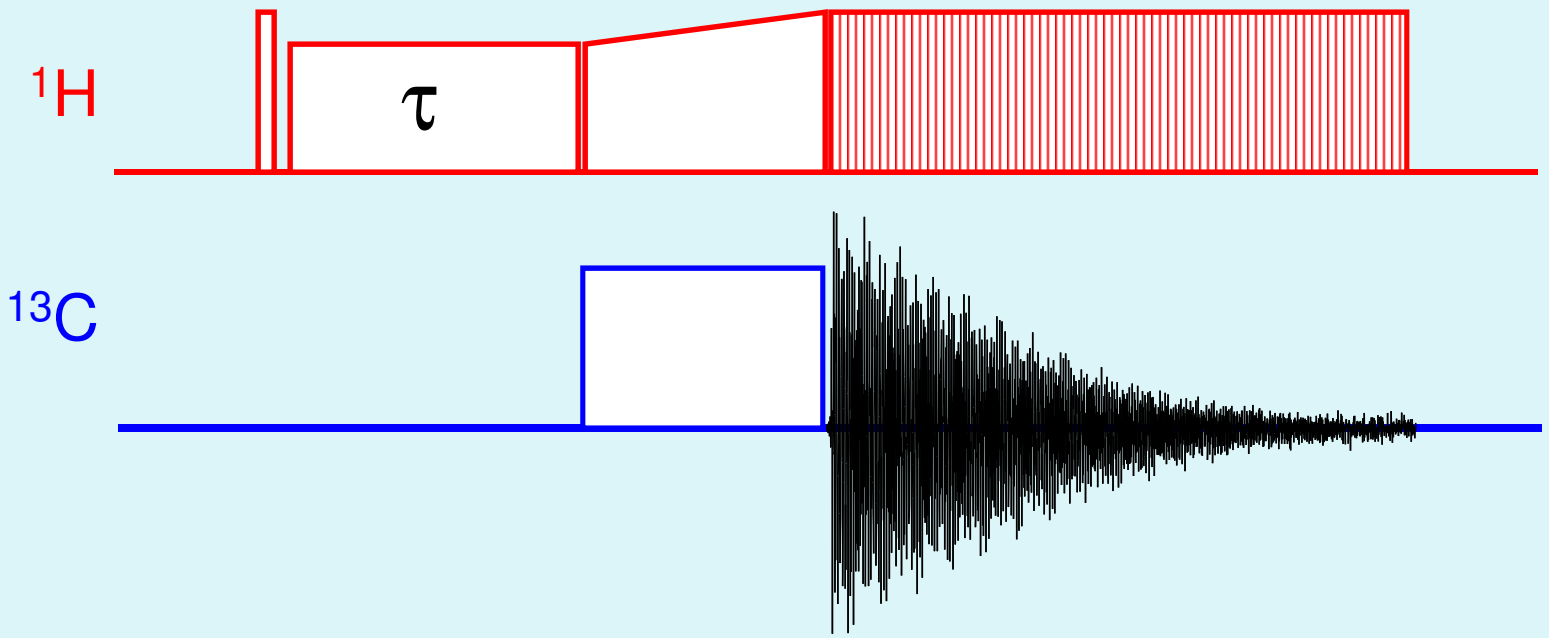
10.0 kHz

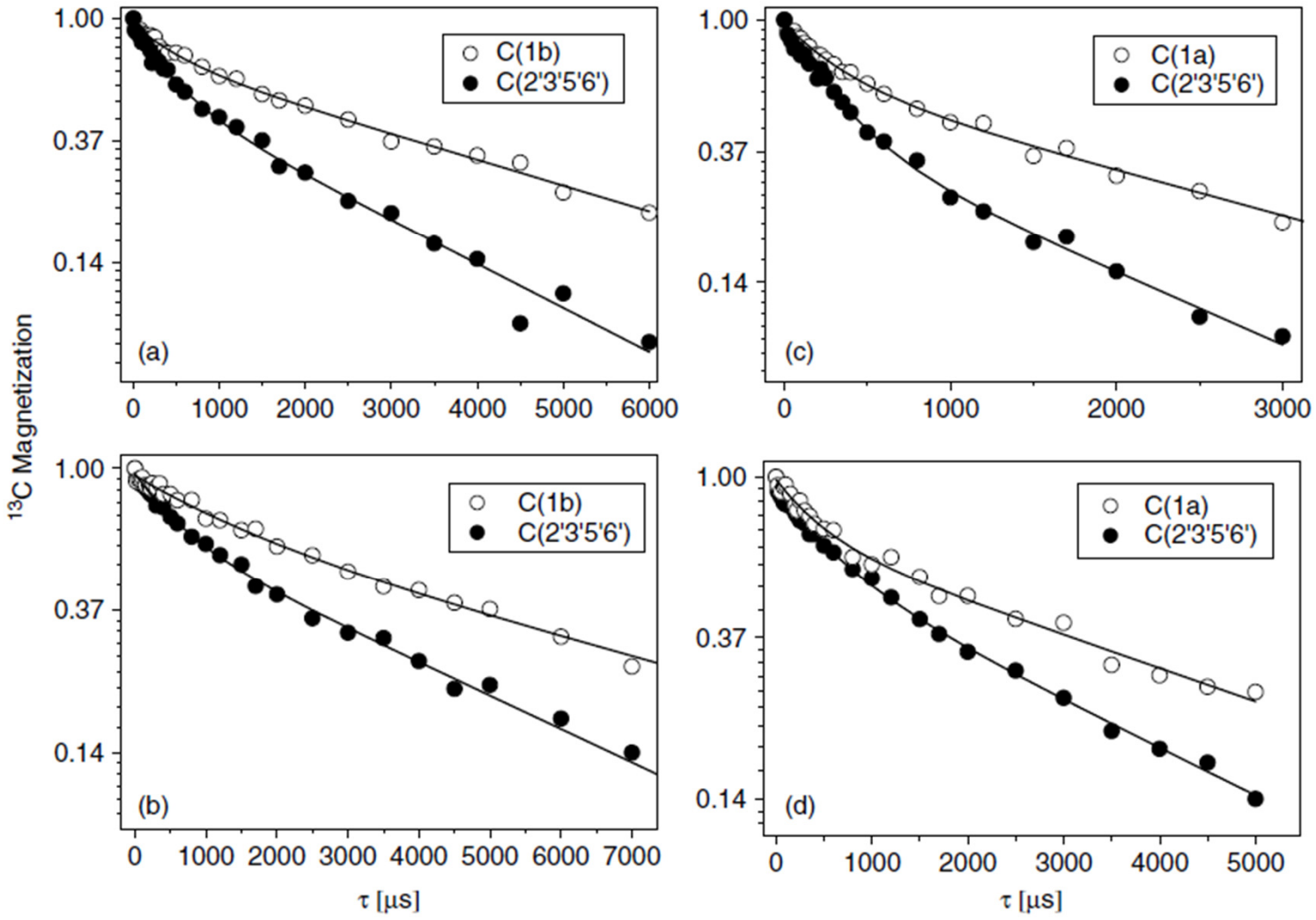
$\omega_{MAS}/2\pi$ [kHz]	T_1 [ms]
5	720
10	580
12.5	490



5.0 kHz

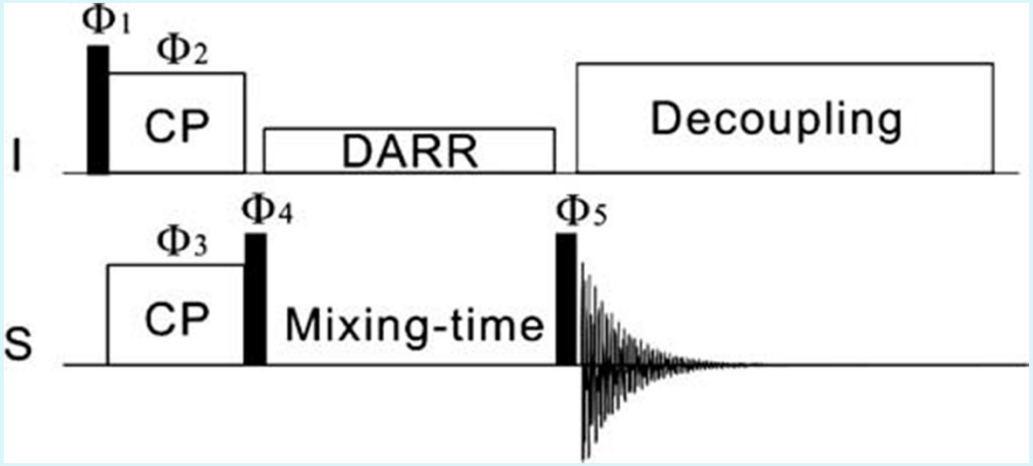
^{13}C detected ^{1}H $T_{1\rho}$ pulse sequence





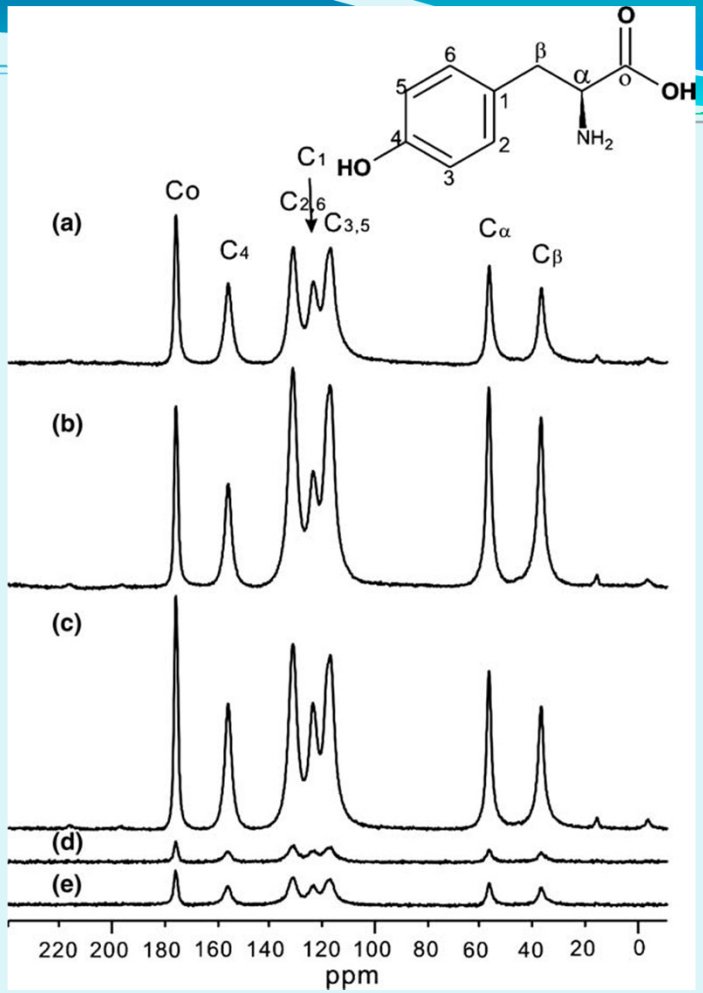
ure 6. ^{13}C magnetization as a function of the spin-lock time τ for selected carbons in the $T_{1\rho C}$ experiment. The fittings of the erimental data to the Eqn (1) are shown. (a) $(HCl.CIP)_3Al$ (I), (b) $(HCl.CIP)_3Al$ (II), (c) $(HCl.NOR)_3Al$ (I), (d) $(HCl.NOR)_3Al$ (II). Th ues obtained from the fittings are given in Table 4.

Quantitative CP: QUCP



The QUCP pulse sequence. The DARR irradiation with intensity of $\omega_{1H} = \nu_{MAS}$ is applied on I channel during the mixing time. Solid bars denote $\pi/2$ pulses. Phase cycles: $\Phi1$ = x,x; $\Phi2$ = y; $\Phi3$ = y,y,y,y,x,x,x,x; $\Phi4$ = x,x,x, x,y,y,y,y; $\Phi5$ = x,x,y,y,x,x,y,y; receiver = y,y,x,x,y,y,x,x.

G. Hou et al. / Chemical Physics Letters 421 (2006) 356–360



13C MAS spectra of uniformly 13C, 15N-labeled tyrosine acquired with a single 90 pulse with proton decoupling (a), CP (b), QUCP (c) and NOP (d, e). Eight FIDs were accumulated for each experiment, and the spectra were plotted on the same amplitude scale. The relaxation interval was 500 s for (a), 16 s for (b–e). The contact time was 0.5 ms for (b) and (c). For QUCP experiment, the DARR irradiation time was 1 s. The mixing times are 10 and 20 s for (d) and (e), respectively.