IV. INEPT – Insensitive Nuclei Enhanced by Polarization Transfer

(22-Jun-98)

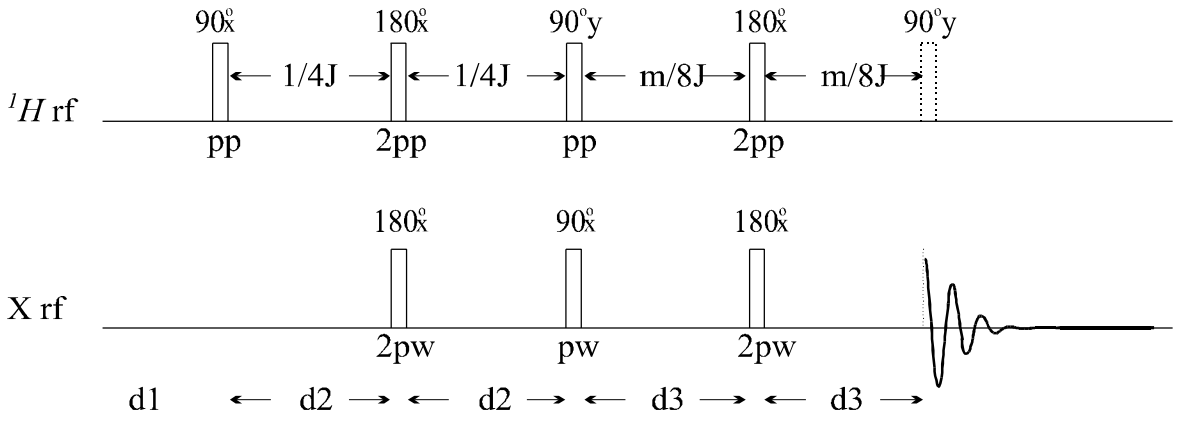
A. Discussion of INEPT

See the discussion section in the previous section on DEPT.

- INEPT should be used if one *J* value is involved and it's size is. Otherwise use of DEPT is recommended.
- The previous statement is not intended to dissuade students from experimenting with the different version of polarization transfer. (After all, the fearless facility manager has been known to give not-such-good advice on occasion. Please do let me know your observation/preferences if you try both INEPT and DEPT on same/similar compounds.)

Refocused Decoupled INEPT

(ineptrd.c [written by cgfry] from Bruker's INEPTRD.AU)



B. Critical Parameters

d1	–	relaxation delay; typically = 1-2s
j	=	140Hz; *change if you want to observe* X *with* J_{XH}*>180 Hz or <110 Hz*
	–	INEPT is sensitive to accurate *J* coupling being entered; use DEPT unless you are fairly certain you know *J* reasonably accurately
pw, tpwr	–	observe *X* 90° pulse width **pw** at power level **tpwr**
pp, pplvl	–	high power ^1H 90° pulse width **pp** at power level **pplvl**
mult	–	multipler for **d3** delay, where **mult= 1** all positive, **2** -CH2- nulled, **3** -CH2- inverted from -CH3 and -CH<

C. INEPT Acquisition

- the INEPTRD macro (run by the menu SETUP SEQUENCES INEPT) is ***not*** the standard Varian sequence, but in my opinion is superior; it incorporates all aspects of Bruker's INEPTRD and INEPTP sequences for decoupled and coupled acquisition, respectively
- for short runs, use facility calibrations for **pw, tpwr, pp, pplvl, dpwr (£46)** and **dmf**

- FILE SETUP SEQUENCES INEPT will setup Inept correctly, including for non-^{13}C acquisitions
  - **mult** is set by the number of coupled protons and **j** ; **d3 = mult/4j**
  - the **d2** interpulse delay is set according to **j**
- for overnights or longer runs, recalibrate (at least) observe and (best) decoupler pulse widths
- set **mult** as needed; see below

D. Calibration

- see ^{13}C section for nominal ^{13}C (X) and ^{1}H decoupler calibrations
- The delay **d2** and **mult*d3** are calculated by the DEPT macro as follows:

d2 = $\frac{1}{4J}$ echo period involving two **d2** delays creates ^{1}H antiphase state

d3 = $\frac{\text{mult}}{4J} = \frac{1}{2pJ}\left[\sin^{-1}\left(\frac{1}{\sqrt{n}}\right)(\text{rad})\right] = \frac{1}{360^{o}J}\left[\sin^{-1}\left(\frac{1}{\sqrt{n}}\right)(\text{deg})\right]$

where n = # I nuclei coupled with J_{XH}

example: suppose have trimethylsilyl, and want to detect ^{29}Si, and $J_{Si\text{-}H}$ = 2 Hz,

then **d2** = 1/(4×2 Hz) = 125 msec

and **d3** = $\frac{1}{360^{o}\times 2\text{Hz}}\sin^{-1}\left(\frac{1}{\sqrt{9}}\right) = \frac{19.47^{o}}{360^{o}\times 2\text{Hz}} = 27\text{msec}$

or **mult** = $\frac{2}{180^{o}}\sin^{-1}\left(\frac{1}{\sqrt{9}}\right) = \frac{2\times 19.47^{o}}{180^{o}} = 0.216$ (same as in DEPT setup)

These values are calculated internal in the /vnmr/psglib/ineptrd.c pulse sequence code.

E. Data Workup and Plotting

- workup is similar (identical) to ^{13}C 1d and DEPT