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GUIDELINES FOR THE REPRESENTATION OF PULSE SEQUENCES FOR SOLUTION-STATE NUCLEAR MAGNETIC RESONANCE SPECTROMETRY

(IUPAC Recommendations 2001)

Prepared for publication by

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Guidelines for the representation of pulse sequences for solution-state nuclear magnetic resonance spectrometry

(IUPAC Recommendations 2001)

Abstract: In drawing up the specifications for a standard for multidimensional nuclear magnetic resonance spectroscopy (NMR) it became clear that the spectroscopic data content needed to be qualified by experimental condition information especially pertaining to the pulse sequences used to obtain the free induced decays or spectra. Failure to include this information not only severely inhibits the ability of subsequent data handling packages to work with the experimental data, but also makes interpretation of the final results virtually impossible.

This paper has been produced in collaboration with the NMR spectrometer manufacturers in an attempt to get agreement on a definitive list of the most frequently used pulse sequence programs. The list includes entries where common agreement has been reached as to the acronym to name the experiment and the key instrument independent parameters needed to report concisely.

It is not intended to restrict in any way the freedom of manufacturers or users to develop new and novel experimental pulse sequences, but should aid reporting of experimental data where the more common sequences are in use.

1. INTRODUCTION

The transfer of data between computers of different origin, running differing operating systems and application software has been greatly enhanced by the introduction of openly agreed standards. In the field of spectroscopy, the publication and successful implementation of the JCAMP-DX (Joint Committee on Atomic and Molecular Physical Data – Data Exchange) protocols has made it possible to transfer infrared and Raman spectroscopy [1], chemical structure [2], nuclear magnetic resonance spectroscopy [3], and mass spectrometry [4] data sets.

The JCAMP-DX protocols are based on delivering scientific data using ASCII encoding to ensure maximum compatibility and longevity of the encoded data. Depending on the encoding software, it is possible to produce data exchange files with absolutely no loss of information. In-built compression algorithms can often yield ASCII exchange files that are smaller than the original binary spectrometer files, again with no loss of data. More recent interest in the long-term, safe storage and archiving of original experimental data sets in electronic form has delivered a new dimension to the use of independent international standards.

Coordinated work on the first JCAMP-DX protocol for NMR began following the sixth INUM conference in Italy in 1990. During this meeting the major NMR manufacturers agreed to implement the protocol when finished. The protocol was published after several years work in 1993 [3] and is available online from <http://www.jcamp.org>

This first NMR protocol was specifically designed to handle conventional NMR spectra and free induction decays. Some additional NMR relevant labeled data records were added in JCAMP-DX 5.01 [5]. Recently, a task group has been working on a protocol to handle multidimensional NMR data sets [6]. This task group decided that it was important to define a recommended reporting format for the

most common NMR pulse sequences. IUPAC has also published in 1997 recommendations on the parameters and symbols for use in NMR, and where appropriate these have been incorporated here [7].

2. SCOPE

2.1 Copyright

This publication was drawn up by the International Union of Pure and Applied Chemistry (IUPAC) Standing Committee on Printed and Electronic Publications (CPEP) Working Party on Spectroscopic Data Standards (JCAMP-DX) following extensive consultation with users and manufacturers. The areas covered here involve extending the NMR standard. As with all such publications, they are meant for public use. The specifications are copyrighted by IUPAC. The right to copy these specifications for scientific use is hereby granted.

2.2 Use of name

The use of the name JCAMP-DX in the description of data files and software implies the capability of converting between internal data files and JCAMP-DX according to the various published protocols.

3. NMR PULSE SEQUENCES

JCAMP-DX version 5.01 [5] defined pulse sequence information as follows:

3.1.1 Pulse sequences

Unlike most other spectroscopic techniques, the signals observed in NMR are dependent upon the particular pulse sequence used. Therefore, an NMR spectrum, FID or relaxation signal has little or no meaning if the pulse sequence is not given. For this reason, a new labeled-data-record (LDR) has been created in the data type specific notes section to contain this information.

The new entry to the specification will be: ##.PULSE SEQUENCE= (TEXT)

This LDR will contain all pertinent information concerning pulse sequences, pulse delays, relaxation delays, pulse angles etc.. Whenever possible, this LDR should include the name of the pulse sequence. The text in this field should preferentially be based on the standard reporting format for NMR pulse sequences or may be written in the instrument's native macro language provided enough information is supplied to clearly enable post processing of the data by third parties. The LDR ##.SAMPLING PROCEDURE= will no longer be used to contain this information.

This document contains the recommended standard reporting formats referred to above.

To simplify matters, it was decided that a standard definition was desirable and could be achieved for the most common pulse sequences and their associated parameters. This list follows below and should be used for reporting pulse sequence information. Where a pulse sequence is used that does not belong to the list it should be reported in the instrument specific macro language with sufficient information to allow post-processing by a third party.

The nomenclature recommended has been chosen for clarity. In general, the term "p" followed by a pulse angle will denote a pulse of some nature and will have the units of microseconds (μs). The "d" terms are static delays in milliseconds (ms), with "rd" being the relaxation delay from the end of the acquisition to the start of the first pulse in the next pulse sequence and defined here as starting the pulse sequence in milliseconds (ms). For incremental delays, the "id" terms indicate the start delay values in milliseconds (ms). The various power attenuation levels are stored in "s" terms (dB), and the s1 and s2

power attenuation levels will always refer to the transmitter, and, conversely, the s3, s4, s5, and s6 power attenuation levels will always refer to the decoupler sequences.

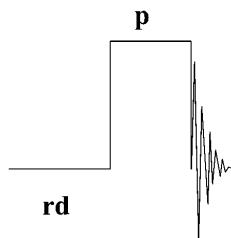
Each pulse sequence described below will contain the acronym to be reported as the title, a short definition of the pulse sequence followed by the usage, and the key parameters regarded as essential when reporting data measured in this way.

3.1 Pulse acquisition

Definition: Simple pulse acquisition program

Usage: rd - p - acquisition

Key parameters:



p: transmitter high-power pulse in microseconds (μ s)

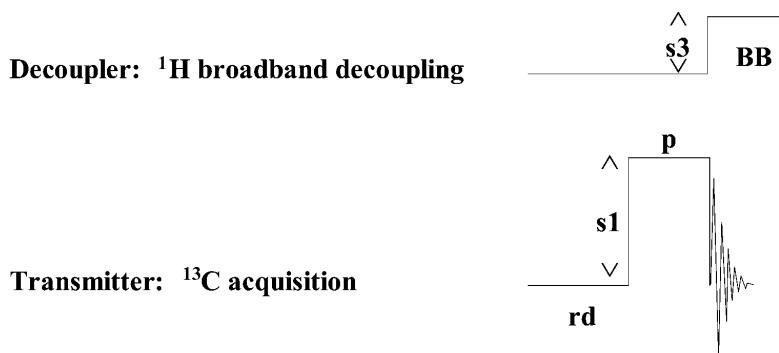
rd: relaxation delay in milliseconds (ms)

3.2 Pulse acquisition DEC

Definition: Simple pulse acquisition program with broadband decoupling

Usage: rd - s1 p - acquisition
.....s3 decoupling (broadband)

Key parameters:



s1: power attenuation level for radio frequency (RF) pulses (dB)

s3: decoupler power attenuation level (dB)

p: transmitter high-power pulse in microseconds (μ s)

rd: relaxation delay in milliseconds (ms)

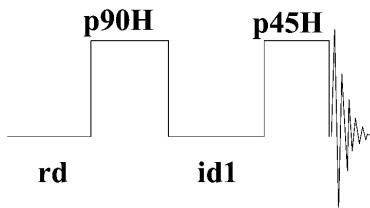
3.3 COSY

Definition: 2D homonuclear shift correlation

Reference: W. P. Aue, E. Bartholdi, R. R. Ernst. *J. Chem. Phys.* **64**, 2229 (1976)

Usage: rd - p90H - id1 - p45H - acquisition

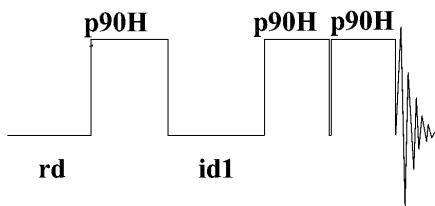
Key parameters:



- p90H: 90 degree transmitter high-power pulse in microseconds (μs)
 p45H: 45 degree transmitter high-power pulse in microseconds (μs)
 id1: incremented delay (2D) start value = 1/spectralwidth (1/sweepwidth) in milliseconds (ms)
 rd: relaxation delay in milliseconds (ms)

3.4 DQF-COSY

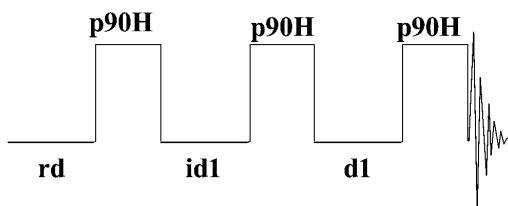
- Definition: 2D homonuclear shift correlation with double quantum filter
 References: A. Wokaun and R. R. Ernst. *Chem. Phys. Lett.* **52**, 407 (1977)
 A. J. Shaka and R. Freeman. *J. Magn. Reson.* **51**, 169 (1983)
 Usage: rd - p90H - id1 - p90H - p90H - acquisition
 Key parameters:



- p90H: 90 degree transmitter high-power pulse in microseconds (μs)
 id1: incremented delay (2D) start value = 1/spectralwidth in milliseconds (ms)
 rd: relaxation delay in milliseconds (ms)

3.5 NOESY

- Definition: 2D homonuclear correlation via dipolar coupling; dipolar coupling may be due to NOE or chemical exchange
 Reference: J. Jeener, B. H. Meier, P. Bachmann, R. R. Ernst. *J. Chem. Phys.* **71**, 4546 (1979)
 Usage: rd - p90H - id1 - p90H - d1 - p90H - acquisition
 Key parameters:

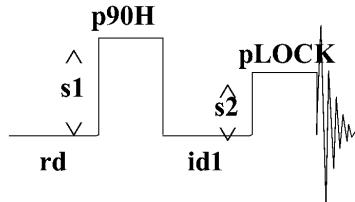


- p90H: 90 degree transmitter high-power pulse (μs)
 id1: incremented delay (2D) start value = 1/spectralwidth in milliseconds (ms)

- rd: relaxation delay in milliseconds (ms)
 d1: mixing time in milliseconds (ms)
 NS: number of transients (scans) (an integer multiple of 8)

3.6 ROESY

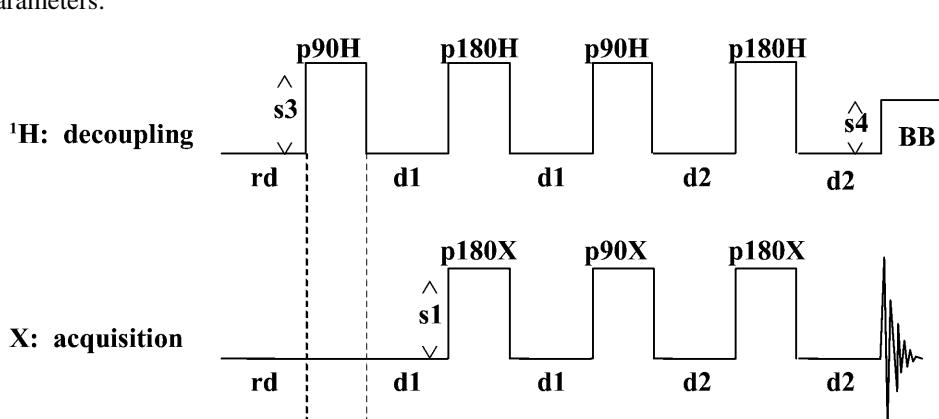
- Definition: 2D rotating frame NOE experiment with cw spinlock for mixing
 Reference: A. Bax and D. G. Davis. *J. Magn. Reson.* **63**, 207–213 (1985)
 Usage: rd - s1 p90H - id1 - s2 pLOCK - acquisition
 Key parameters:



- s1: high-power attenuation level (dB)
 s2: low-power attenuation level for ROESY spinlock (dB)
 p90H: 90 degree transmitter high-power pulse in microseconds (μ s)
 pLOCK: low-power continuous wave (cw) pulse for ROESY spinlock in microseconds (μ s)
 id1: incremented delay (2D) start value = 1/spectralwidth in milliseconds (ms)
 rd: relaxation delay in milliseconds (ms)
 NS: number of transients (an integer multiple of 8)

3.7 INEPT-DEC

- Definition: INEPT for nonselective polarization transfer with decoupling during acquisition
 Reference: G. A. Morris and R. Freeman. *J. Am. Chem. Soc.* **101**, 760 (1979)
 Usage: rd-s3 p90H-d1 - p180H-d1-p90H-d2-p180H-d2-s4 decoupling
 rd.....d1-s1 p180X-d1-p90X-d2-p180X-d2 - acquisition
 Key parameters:

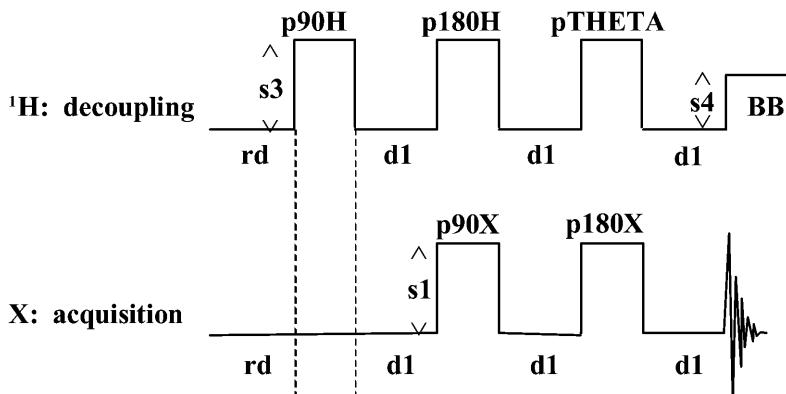


- s1: high-power attenuation level for RF pulses X (dB)
 s3: high-power attenuation level for RF pulses ^1H (dB)
 s4: low-power attenuation level for broadband decoupling (dB)

- p90X: 90 degree transmitter high-power pulse in microseconds (μ s)
 p180X: 180 degree transmitter high-power pulse in microseconds (μ s)
 p90H: 90 degree decoupler high-power pulse in microseconds (μ s)
 p180H: 180 degree decoupler high-power pulse in microseconds (μ s)
 rd: relaxation delay in milliseconds (ms)
 d1: delay normally derived from the relation $1/(4J(XH))$ in milliseconds (ms)
 d2: delay normally derived from the relations:
 $1/(6J(XH))$ XH, XH₂, XH₃ positive
 $1/(4J(XH))$ XH only
 $1/(3J(XH))$ XH, XH₃ positive, XH₂ negative
 in milliseconds (ms)
 NS: number of transients (an integer multiple of 4)

3.8 DEPT-DEC

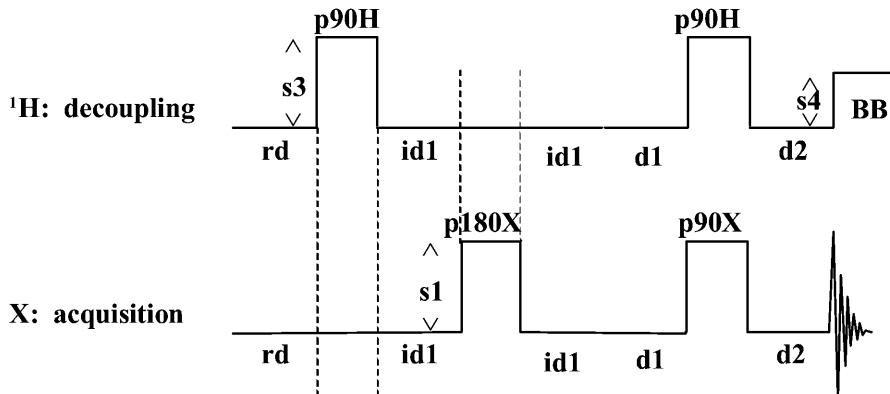
- Definition: DEPT polarization transfer with decoupling during acquisition
 Reference: M. R. Bendall, D. M. Doddrell, D. T. Pegg. *J. Am. Chem. Soc.* **103**, 4603 (1981)
 Usage: rd - s3 p90H - d1 - p180H - d1 - pTHETA - d1 - s4 decoupling
 rd.....d1-s1 p90X - d1 - p180X - d1 - acquisition
 Key parameters:



- s1: high-power attenuation level for RF pulses X (dB)
 s3: high-power attenuation level for RF pulses ¹H (dB)
 s4: low-power attenuation level for broadband decoupling (dB)
 p90X: 90 degree transmitter high-power pulse in microseconds (μ s)
 p180X: 180 degree transmitter high-power pulse in microseconds (μ s)
 p90H: 90 degree decoupler high-power pulse in microseconds (μ s)
 p180H: 180 degree decoupler high-power pulse in microseconds (μ s)
 pTHETA: 45, 90 or 135 degree decoupler high-power pulse in microseconds (μ s)
 45 degree - all positive
 90 degree - XH only
 135 degree - XH, XH₃ positive, XH₂ negative
 rd: relaxation delay in milliseconds (ms)
 d1: delay normally derived from the relation $1/(2J(XH))$ in milliseconds (ms)
 NS: number of transients (an integer multiple of 4)

3.9 HETCOR

Definition: 2D heteronuclear shift correlation
 Reference: A. Bax and G. A. Morris. *J. Magn. Reson.* **42**, 501 (1981)
 Usage: rd-s3 p90H - id1 - d1 - p90H - d2 - s4 decoupling
 rd.....id1 - s1 p180X - id1 - d1 - p90X - d2 - acquisition
 Key parameters:



- s1: high-power attenuation level for RF pulses X (dB)
 s3: high-power attenuation level for RF pulses ^1H (dB)
 s4: low-power attenuation level for broadband decoupling (dB)
 p90X: 90 degree transmitter high-power pulse in microseconds (μs)
 p180X: 180 degree transmitter high-power pulse in microseconds (μs)
 p90H: 90 degree decoupler high-power pulse in microseconds (μs)
 id1: incremented delay (2D) start value = $1/(2 \times \text{spectralwidth})$ in milliseconds (ms)
 rd: relaxation delay in milliseconds (ms)
 d1: delay normally derived from the relation $1/(2J(XH))$ in milliseconds (ms)
 d2: delay normally derived from the relation $1/(3J(XH))$ in milliseconds (ms) for all multiplicities
 NS: number of transients (an integer multiple of 4)

3.10 HMQC

Definition: 2D inverse detected heteronuclear direct bond shift correlation
 Reference: L. Müller. *J. Am. Chem. Soc.* **101**, 4481 (1979)
 Usage: rd-s1 p90H - d1.....id1 - p180H - id1.....d1 - acquisition
 rd.....d1 - s3 p90X - id1.....id1 - p90X - d1 - s4 decoupling (GARP)

GARP

(Globally optimized Alternating-phase Rectangular Pulses) is a cyclic windowless sequence for broadband decoupling giving minimal residual splittings over a wide range of decoupler resonance offsets. The sequence is constructed from building blocks P and Q of the type

$$P = 27.1 \text{ } \mathbf{57.6} \text{ } 122.0$$

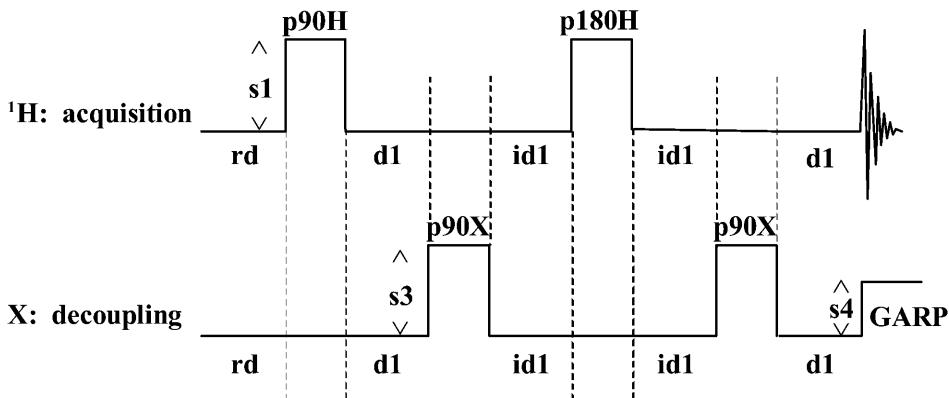
$$Q = 130.8 \text{ } \mathbf{262.8} \text{ } 65.9 \text{ } \mathbf{64.6} \text{ } 87.0 \text{ } \mathbf{98.0} \text{ } 137.2 \text{ } \mathbf{256.5} \text{ } 71.6 \text{ } \mathbf{51.1}$$

Here, the numbers denote the pulse angles; bold numbers denote phase inversion. P and Q are then incorporated into a decoupling cycle of the type

$$R \text{ } R \text{ } \mathbf{R} \text{ } R, \text{ where } R = P \text{ } Q \text{ } \mathbf{P} \text{ } Q$$

Reference: A. J. Shaka, J. Keeler, R. Freeman. *J. Magn. Reson.* **64**, 547 (1985)

Key parameters:

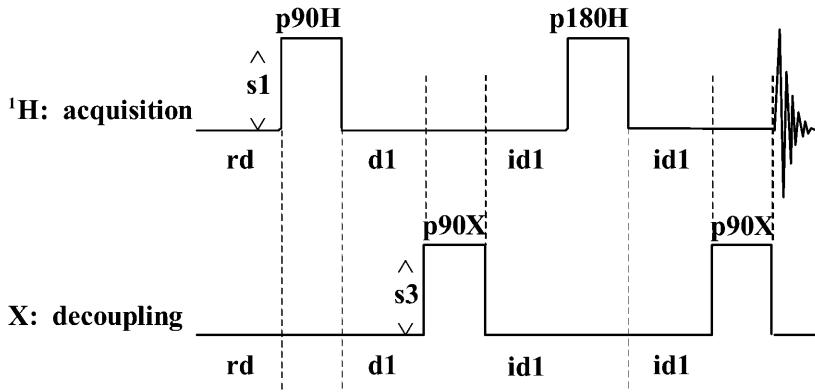


- s1: high-power attenuation level for RF pulses ^1H (dB)
- s3: high-power attenuation level for RF pulses X (dB)
- s4: low-power attenuation level for GARP decoupling (dB)
- p90H: 90 degree transmitter high-power pulse in microseconds (μs)
- p180H: 180 degree transmitter high-power pulse in microseconds (μs)
- p90X: 90 degree decoupler high-power pulse in microseconds (μs)
- p90GARP: 90 degree low-power decoupler pulse for calculating the GARP pulse angles (μs)
- id1: incremented delay (2D) start value = $1/(2 \times \text{spectralwidth})$ in milliseconds (ms)
- rd: relaxation delay in milliseconds (ms)
- d1: delay normally derived from the relation $1/(2J(\text{XH, one bond}))$ in milliseconds (ms)
- NS: number of transients (an integer multiple of 8)

3.11 HMBC

- Definition: 2D inverse detected heteronuclear long-range shift correlation
- Reference: A. Bax and M. F. Summers. *J. Am. Chem. Soc.* **108**, 2093 (1986)
- Usage: rd - s1 p90H - d1.....id1 - p180H - id1.....acquisition
rd.....d1 - s3 p90X - id1.....id1 - p90X

Key parameters:

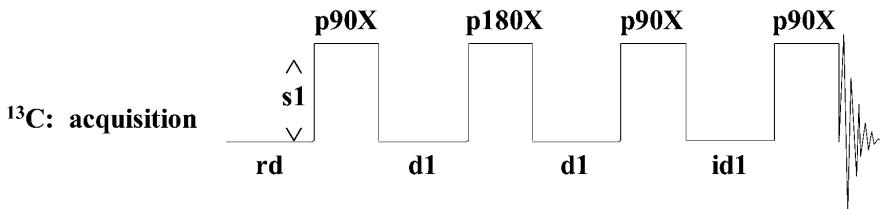


- s1: high-power attenuation level for RF pulses ^1H (dB)
- s3: high-power attenuation level for RF pulses X (dB)

p90H:	90 degree transmitter high-power pulse in microseconds (μ s)
p180H:	180 degree transmitter high-power pulse in microseconds (μ s)
p90X:	90 degree decoupler high-power pulse in microseconds (μ s)
id1:	incremented delay (2D) start value = $1/(2 \times \text{spectralwidth})$ in milliseconds (ms)
rd:	relaxation delay in milliseconds (ms)
d1:	delay normally derived from the relation $1/(2J(\text{XH, long range}))$ in milliseconds (ms)
NS:	number of transients (an integer multiple of 8)

3.12 INADEQUATE

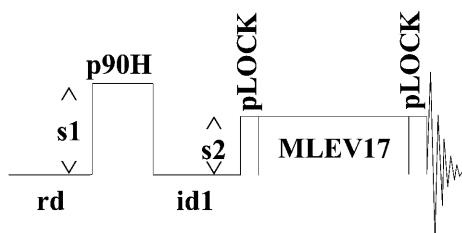
Definition:	2D carbon–carbon connectivity mapping
Reference:	A. Bax, R. Freeman, S. Kempsell. <i>J. Am. Chem. Soc.</i> 102 , 4849 (1980)
Usage:	rd - s1 p90X - d1 - p180X - d1 - p90X - id1 - p90X - acquisitions3 decoupling (broadband)
Key parameters:	



s1:	high-power attenuation level for RF pulses (dB)
s3:	low-power attenuation level for broadband decoupling (dB)
p90X:	90 degree transmitter high-power pulse in microseconds (μ s)
p180X:	180 degree transmitter high-power pulse in microseconds (μ s)
id1:	incremented delay (2D) start value = $1/(2 \times \text{spectralwidth})$ in milliseconds (ms)
rd:	relaxation delay in milliseconds (ms)
d1:	delay normally derived from the relation $1/(4J(\text{CC}))$ in milliseconds (ms)
NS:	number of transients (an integer multiple of 128)

3.13 TOCSY

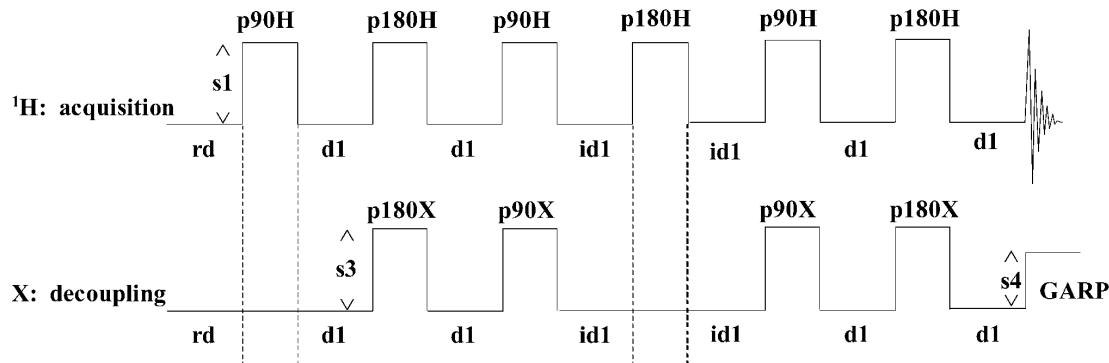
Definition:	Homonuclear Hartman–Hahn transfer using MLEV17 sequence for mixing
Reference:	A. Bax and D.G. Davis. <i>J. Magn. Reson.</i> 65 , 355–360 (1985)
Usage:	rd - s1 p90H - id1 - s2 pLOCK - (MLEV17) - pLOCK - acquisition
Key parameters:	



s1:	high-power attenuation level (dB)
s2:	low-power attenuation level for MLEV spinlock (dB)
p90H:	90 degree transmitter high-power pulse in microseconds (μ s)
pLOCK:	transmitter spin lock low-power pulse in microseconds (μ s)
id1:	incremented delay (2D) start value = $1/(2 \times \text{spectralwidth})$ in milliseconds (ms)
rd:	relaxation delay in milliseconds (ms)
MLEV17:	number of MLEV17 loops (integer)
NS:	number of transients (an integer multiple of 8)

3.14 HSQC

Definition:	2D inverse detected heteronuclear direct bond shift correlation; with INEPT/reverse INEPT sequence
Reference:	G. Bodenhausen and D. J. Ruben. <i>Chem. Phys. Lett.</i> 69 , 185 (1980)
Usage:	rd-s1 p90H-d1 - p180H - d1-p90H - id1- p180H-id1 - p90H - d1- p180H- d1 - acquisition rd.....d1- s3 p180X - d1- p90X - id1.....id1 - p90X - d1- p180X -d1-s4 decoupling (GARP)
Key parameters:	

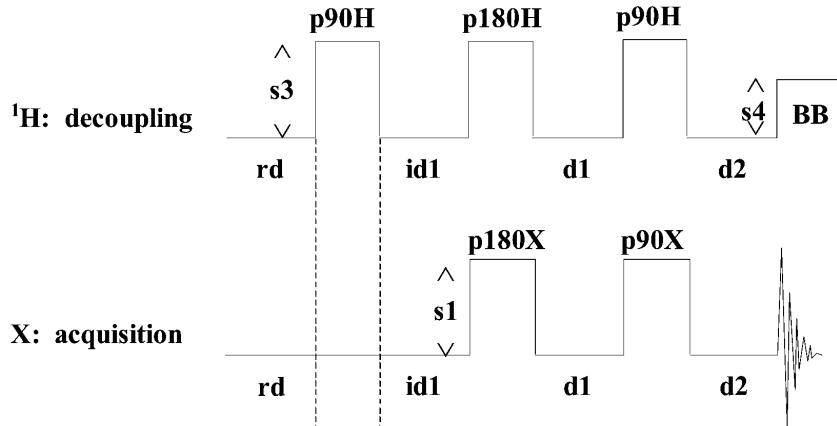


s1:	high-power attenuation level for RF pulses ^1H (dB)
s3:	high-power attenuation level for RF pulses X (dB)
s4:	low-power attenuation level for GARP decoupling (dB)
p90H:	90 degree transmitter high-power pulse in microseconds (μ s)
p180H:	180 degree transmitter high-power pulse in microseconds (μ s)
p90X:	90 degree decoupler high-power pulse in microseconds (μ s)
p180X:	180 degree decoupler high-power pulse in microseconds (μ s)
p90GARP:	90 degree low-power decoupler pulse for calculating the GARP pulse angles (μ s)
id1:	incremented delay (2D) start value = $1/(2 \times \text{spectralwidth})$ in milliseconds (ms)
rd:	relaxation delay in milliseconds (ms)
d1:	delay normally derived from the relation $1/(4J(\text{XH, one bond}))$ in milliseconds (ms)
NS:	number of transients (an integer multiple of 8)

3.15 COLOC

Definition:	2D HX correlation via long-range couplings with H–H decoupling in F1
Reference:	H. Kessler, C. Griesinger, J. Zarbock, H. Loosli. <i>J. Magn. Reson.</i> 57 , 331 (1984)
Usage:	rd - s3 p90H - id1 - p180H - d1 - p90H - d2 - s4 broadband decoupling rd.....id1 - s1 p180X - d1 - p90X - d2 - acquisition

Key parameters:

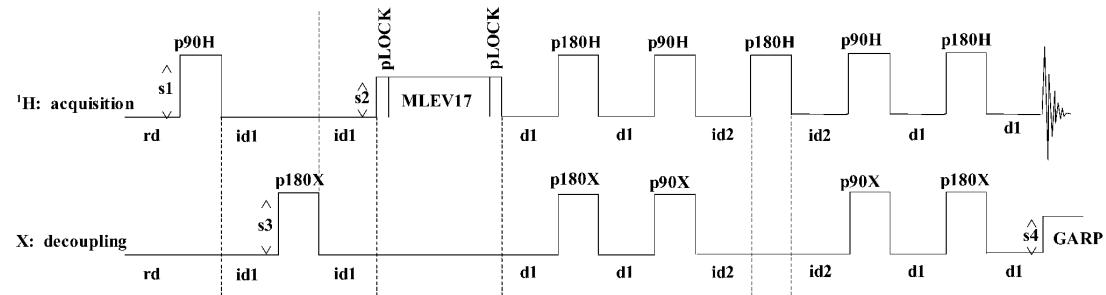


- s1: high-power attenuation level for RF pulses X (dB)
- s3: high-power attenuation level for RF pulses ^1H (dB)
- s4: low-power attenuation level for broadband decoupling (dB)
- p90X: 90 degree transmitter high-power pulse in microseconds (μs)
- p180X: 180 degree transmitter high-power pulse in microseconds (μs)
- p90H: 90 degree decoupler high-power pulse in microseconds (μs)
- p180H: 180 degree decoupler high-power pulse in microseconds (μs)
- id1: incremented delay (2D) start value = $1/(2 \times \text{spectralwidth})$ in milliseconds (ms)
- rd: relaxation delay in milliseconds (ms)
- d1: delay for evolution of long-range couplings in milliseconds (ms)
- d2: delay for evolution of long-range couplings in milliseconds (ms)
- NS: number of transients (an integer multiple of 16)

3.16 3D HSQC-TOCSY

- Definition: 3D homonuclear correlation combined with inverse detected heteronuclear direct bond shift correlation.
- Reference: D. Marion, P. C. Driscoll, L. E. Kay, P. T. Wingfield, A. Bax, A. M. Gronenborn, G. M. Clore. *Biochemistry* **28**, 6150 (1989)
- Usage:
- rd - s1 p90H - id1.....id1 - s2 pLOCK-MLEV17- pLOCK - d1 - p180H - d1 - p90H - id2 - p180H - id2 - p90H - d1 - p180H - d1 - acquisition
 - rd.....id1 - s3 p180X - id1.....d1 - p180X - d1 - p90X - id2.....id2 - p90X - d1 - p180X - d1 - s4 decoupling (GARP)

Key parameters:

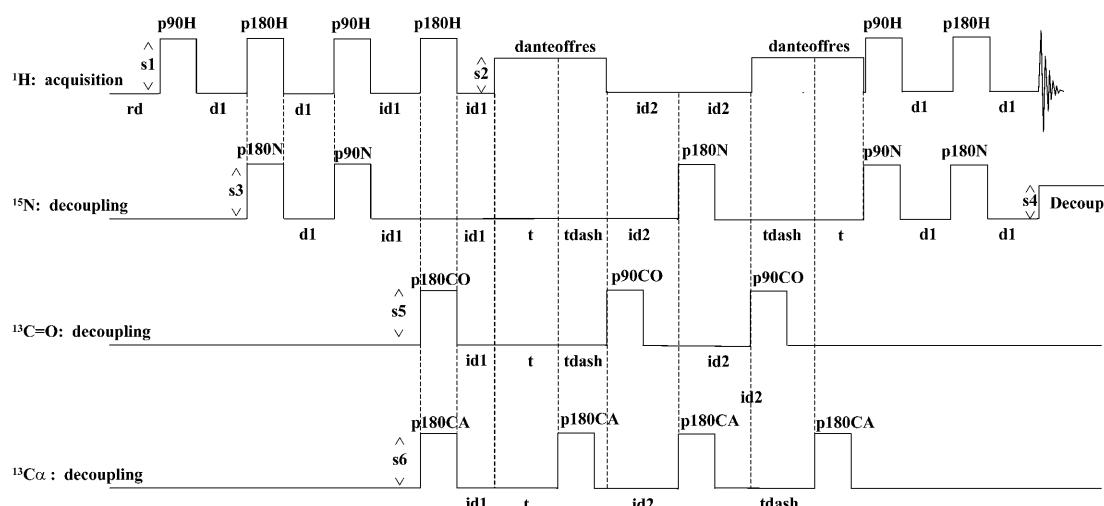


s1:	high-power attenuation level for RF pulses ^1H (dB)
s2:	low-power attenuation level for MLEV spinlock (dB)
s3:	high-power attenuation level for RF pulses X (dB)
s4:	low-power attenuation level for GARP decoupling (dB)
p90H:	90 degree transmitter high-power pulse in microseconds (μs)
p180H:	180 degree transmitter high-power pulse in microseconds (μs)
p90X:	90 degree decoupler high-power pulse in microseconds (μs)
p180X:	180 degree decoupler high-power pulse in microseconds (μs)
p90GARP:	90 degree low-power decoupler pulse for calculating the GARP pulse angles (μs)
pLOCK:	transmitter spinlock low-power pulse in microseconds (μs)
id1:	incremented delay start value = $1/(2\text{SW}1)$ in milliseconds (ms)
id2:	incremented delay start value = $1/(2\text{SW}3)$ in milliseconds (ms)
rd:	relaxation delay in milliseconds (ms)
d1:	constant delay derived from the relation $1/(4J(\text{XH, one bond}))$ in milliseconds (ms)
NS:	number of transients
MLEV17:	number of MLEV17 loops (integer)

3.17 3D-HNCO

Definition:	3D experiment for the correlation of the amide proton shift and the nitrogen-15 shift of one amino acid residue with the amide carbon shift of the preceding amino acid residue.
Reference:	L. E. Kay, M. Ikura, R. Tschudin, A. Bax. <i>J. Magn. Res.</i> 89 , 496–514 (1990)
Usage:	
1H:	rd - s1 p90H - d1 - p180H - d1 - p90H - id1 - p180H - id1 - s2 danteoffres - id2 - id2 - danteoffres - p90H - d1 - p180H - d1 - acquisition
15N:s3 p180N - d1 - p90N - id1.....id1 - t - tdash - id2 - p180N - tdash - t - p90N - d1 - p180N - d1 - s4 decoupling
13CO:s5 p180CO - id1 - t - tdash - p90CO - id2 - p90CO.....
13CA:s6 p180CA - id - t - p180CA - p180CA - tdash - p180CA.....

Key parameters:



s1:	high-power attenuation level for RF pulses ^1H (dB)
s2:	low-power attenuation level for ^1H pulses of the DANTE off-resonance sequence (dB)
s3:	high-power attenuation level for RF pulses nitrogen-15 (dB)
s4:	high-power attenuation level for nitrogen-15 broadband decoupling (dB)
s5:	low-power attenuation level for carbon-13 pulses selective for amide carbonyl of amino acids (dB)
s6:	low-power attenuation level for carbon-13 pulses selective for alpha carbons of amino acids (dB)
p90H:	90 degree high-power proton pulse in microseconds (μs)
p180H:	180 degree high-power proton pulse in microseconds (μs)
p90N:	90 degree high-power nitrogen-15 pulse in microseconds (μs)
p180N:	180 degree high-power nitrogen-15 pulse in microseconds (μs)
p90CO:	90 degree carbon-13 pulse in microseconds (μs) selective for amide carbonyl of amino acids
p180CO:	180 degree carbon-13 pulse in microseconds (μs) selective for amide carbonyl of amino acids
p180CA:	180 degree carbon-13 pulse in microseconds (μs) selective for alpha carbons of amino acids
t, tdash:	delay times for DANTE off-resonance water saturation sequence in milliseconds (ms)
d1:	delay time for coherence transfer from proton to nitrogen in milliseconds (ms)
id1:	incrementable delay start value for nitrogen-15 chemical shift evolution in milliseconds (ms)
id2:	incrementable delay start value for amide carbonyl chemical shift evolution in milliseconds (ms)

Description of the off-resonance DANTE sequence:

The sequence consists of four pulses of length P90H with phases incremented in steps of 90 degrees, starting at 0 degrees. The four-pulse sequence is repeated n times and is given by:

$$[(\text{p90H})_0 (\text{p90H})_{90} (\text{p90H})_{180} (\text{p90H})_{270}]_n$$

The length of the pulses P90H is given by:

$$\text{p90H} = 1 / (4 * \delta(\text{H}_2\text{O}))$$

where $\delta(\text{H}_2\text{O})$ denotes the proton chemical shift of the water signal with respect to the carrier.

4. CONCLUSIONS AND FUTURE WORK

The proposal presented here is limited to the most frequently used pulse sequences. A description based on acronyms of standard pulse sequences as suggested in this paper is suitable for the vast majority of spectra produced by the normal NMR user.

It is not difficult, however, to design new pulse techniques or at least minor modifications of standard techniques. Moreover, even pulse sequences having the same acronym may be implemented in a different way depending on the NMR instrument manufacturer. As there are numerous custom developed pulse sequences working with parameter sets different from the standard ones described here, a fully generic description of NMR pulse sequences should be attempted in future. The description should be short, transparent, and flexible enough to incorporate all types of pulses including shaped pulses, selective excitation, supercycles, and phase cycling procedures, as well as pulsed field gradients in a manufacturer-independent fashion.

This list should be used in the reporting of multiple pulse and multidimensional NMR experiments. As mentioned above, a task force including all of the major NMR manufacturers and independ-

ent software houses has been established. Their goal is to develop and validate a multidimensional NMR data standard. This publication should be a major step toward this goal.

Those wishing to help in this activity can register at <http://www.jcamp.org>.

In the immediate future, the relevant pulse sequences for solid-state NMR will be addressed. As these lists will require periodic maintenance, this will be undertaken by expert limited-term tasks groups reporting in the usual manner through the CPEP.

Finally, a warning: The attenuation levels reported in a JCAMP-DX NMR file should be used as a documentation of the experiment, which has been conducted. **ON NO ACCOUNT** should the values reported in the file be used to program a spectrometer. Always refer to the spectrometer handbook for the appropriate power attenuation levels appropriate for your spectrometer and coils!

5. ACKNOWLEDGMENTS

The authors wish to thank the many NMR specialists, academic, industrial and manufacturer, who have contributed to this sometimes very lively discussion on this document over recent years. Many will find their suggestions incorporated—some may be disappointed that their particular proposal has not been adopted. However, the need to produce a compromise acceptable to all NMR instrument manufacturers and third-party software vendors must take priority over academic perfection if the solution is to be implemented throughout the NMR industry to the benefit of all. Most of all, we must thank the manufacturers for encouraging and supporting the development of this standard, which goes to show that many can look beyond their balance sheets to the good of the NMR community as a whole. Thank you!

6. EXAMPLES

Some simple examples of the use of the labeled data record ##.PULSE SEQUENCE= are as follows:

6.2 Example 1

This example is taken from the JCAMP-DX v. 5.01 publication [5].

```
##.PULSE SEQUENCE= PULSE ACQUISITION DEC
s1=      0          $$ power attenuation level for radio frequency pulse (dB)
s3=      14         $$ decoupler power attenuation level (dB)
p=       5.0        $$ transmitter high-power pulse in microseconds
rd=     3000        $$ relaxation delay in milliseconds
```

6.2 Example 2

This example is taken from the first nD-NMR test files

```
##.PULSE SEQUENCE= HETCOR
s1=      0.0        $$ high-power attenuation level for RF pulses X (dB)
s3=      0.0        $$ high-power attenuation level for RF pulses 1H (dB)
s4=      14         $$ low-power attenuation level for broadband decoupling (dB)
p90X=   10.500     $$ 90 degree transmitter high-power pulse in microseconds
p180X=  21.000     $$ 180 degree transmitter high-power pulse in microseconds
p90H=   28.700     $$ 90 degree decoupler high-power pulse in microseconds
id1=    0.114       $$ incremented delay (2D) start value = 1/(2*spectralwidth) in milliseconds
rd=    1000.000    $$ relaxation delay in milliseconds
d1=    0.114       $$ delay normally derived from the relation 1/(2J(XH)) in milliseconds
d2=    10.000       $$ delay normally derived from the relation 1/(3J(XH)) in milliseconds for all
                     multiplicities
NS=    2720        $$ number of transients (an integer multiple of 4)
```

7. REFERENCES

1. R. S. McDonald and P. A. Wilks Jr. "JCAMP-DX: A standard form for exchange of infrared spectra in computer readable form", *Appl. Spectrosc.* **42**, 151–162 (1988) and "JCAMP-DX. A standard format for the exchange of infrared spectra in computer readable format (IUPAC Recommendations 1991)", *Pure Appl. Chem.* **63**, 1781–1792 (1991).
2. J. Gasteiger, B. M. P. Hendriks, P. Hoever, C. Jochum, H. Somberg. "JCAMP-CS: A standard exchange format for chemical structure information in computer-readable form", *Appl. Spectrosc.* **45**, 4–11 (1991).
3. A. N. Davies and P. Lampen. "JCAMP-DX for NMR", *Appl. Spectrosc.* **47**, 1093–1099 (1993).
4. P. Lampen, H. Hillig, A. N. Davies, M. Linscheid. "JCAMP-DX for Mass Spectrometry", *Appl. Spectrosc.* **48**, 1545–1552 (1994).
5. P. Lampen, J. Lambert, R. J. Lancashire, R. S. McDonald, P. S. McIntyre, D. N. Rutledge, T. Fröhlich, A. N. Davies. "An extension to the JCAMP-DX standard file format, JCAMP-DX V.5.01 (IUPAC Recommendations 1999)", *Pure Appl. Chem.* **71**, 1549–1556 (1999).
6. A. N. Davies. "A new NMR data standard for the exchange and archiving for multi-dimensional data sets", *Spectrosc. Eur.* **11**(1), 14–20 (1999). (See also http://www.spectroscopyeurope.com/td_col.html) and *Chem. Int.* **21**(2), 37–39 (1999).
7. R. K. Harris, J. Kowalewski, S. Cabral de Menezes. "Parameters and symbols for use in nuclear magnetic resonance", *Pure Appl. Chem.* **69**, 2489–2495 (1997).