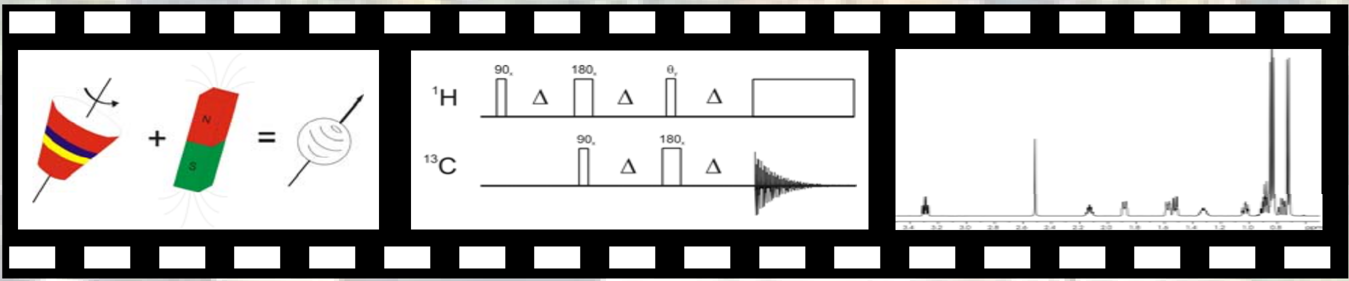
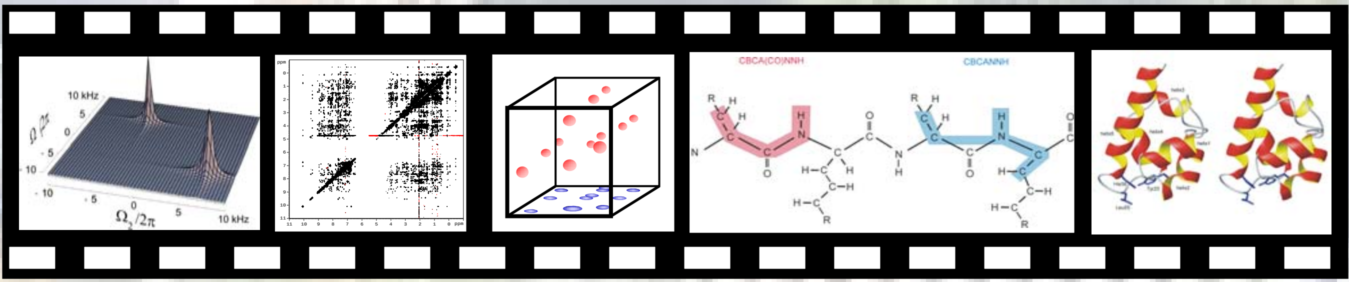


NUS:

Non-Uniform-Sampling in 3D-NMR

Gruppenseminar 18.08.2011



- some concepts
  - sampling vs. sensitivity limit
  - traditional sampling vs. full points
- the sampling schedule
- ct-HNCA @ 900 MHz
- processing

sampling limit

the sample provides sufficient S/N and the data have to be recorded far too long to achieve enough resolution

NUS can drastically shorten the experiment time

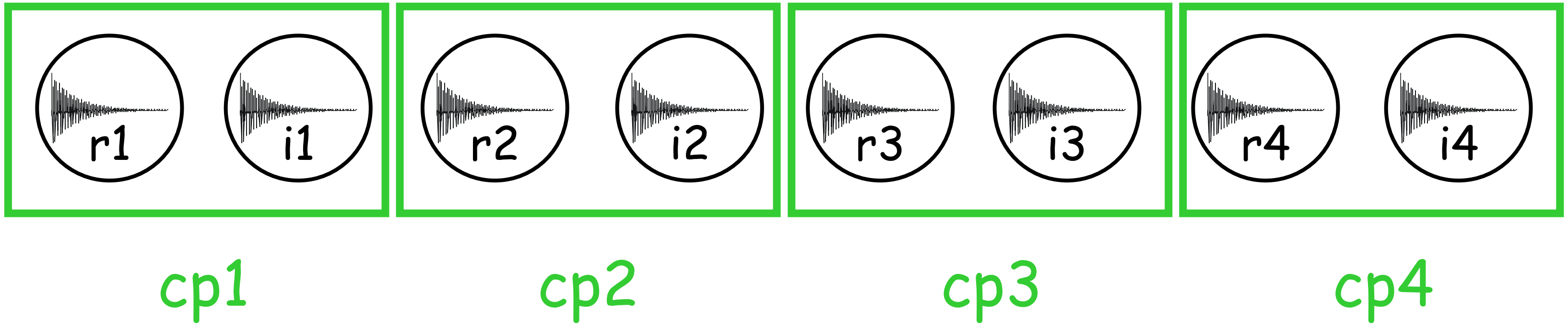
sensitivity limit

the sample requires an extended experiment time to achieve a sufficient S/N anyway and the required resolution can easily be obtained

NUS can provide more flexibility

"traditional sampling" in 2D experiments

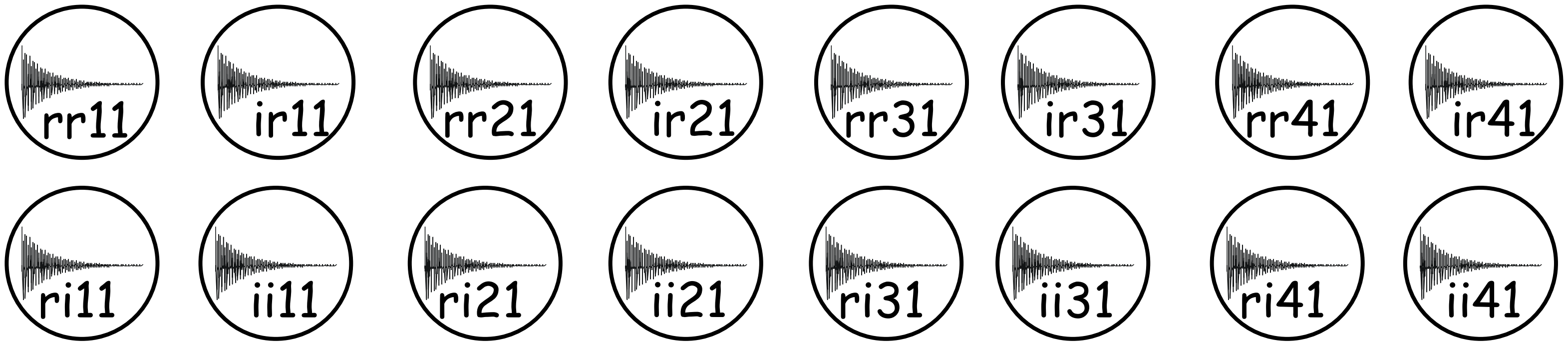
(there is no other way)



A real and an imaginary part is recorded in alternating manner

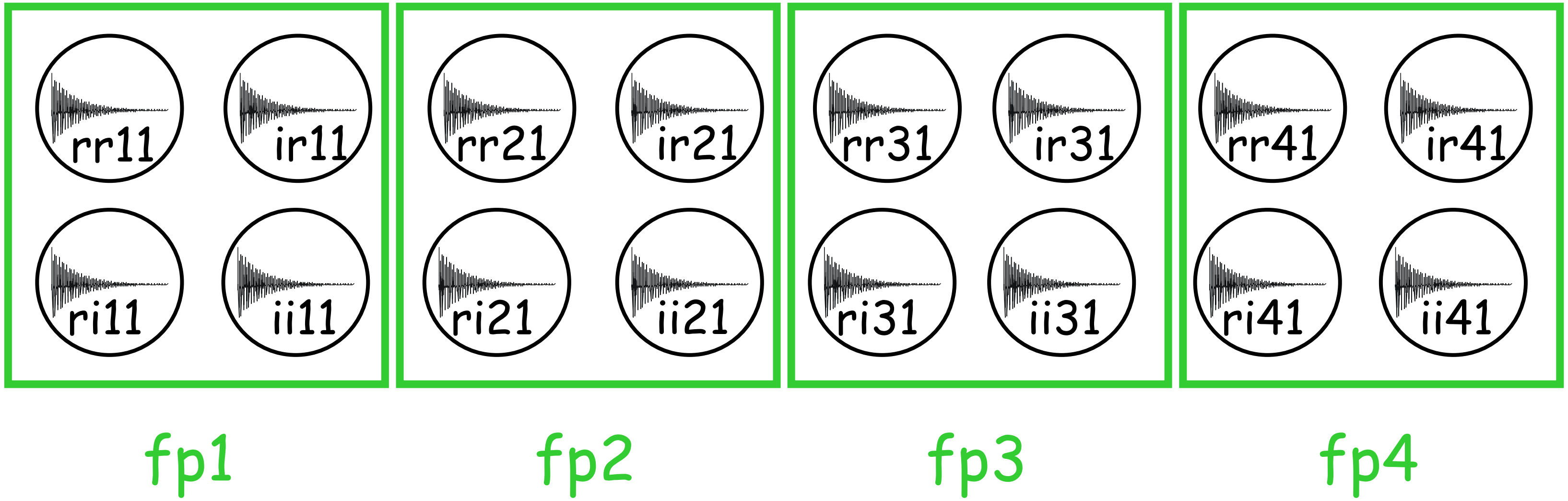
together they form a complex point

"traditional sampling" in 3D experiments



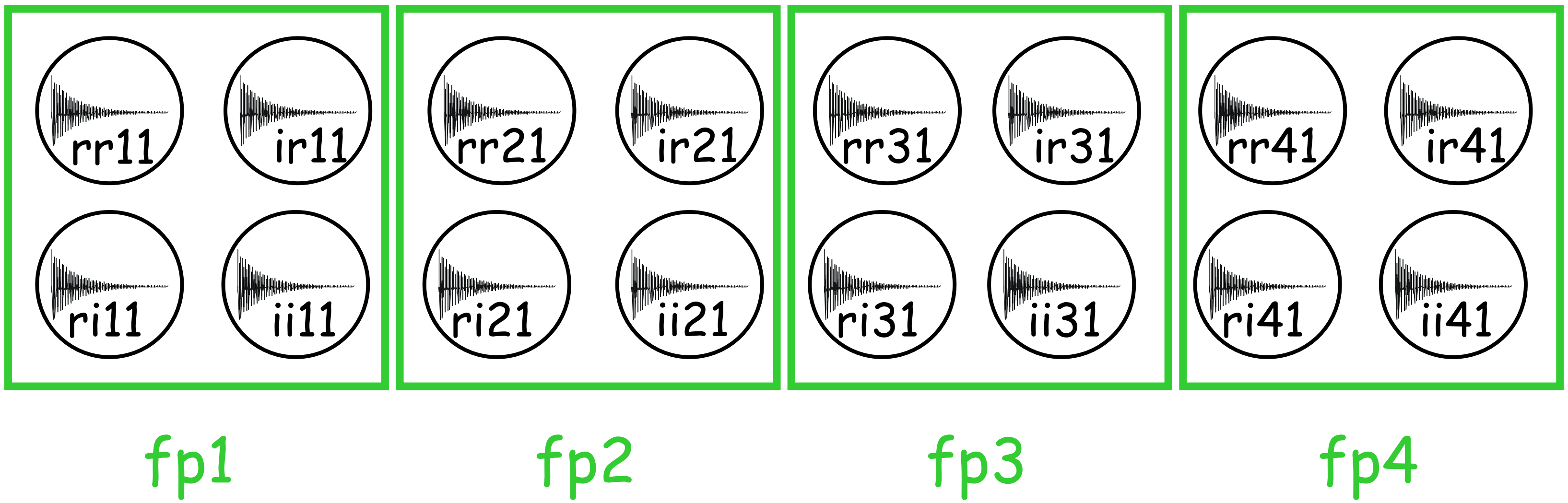
The logical extension of the traditional sampling is to record planes sequentially, with one fast changing dimension and one slow

"traditional sampling" in 3D experiments



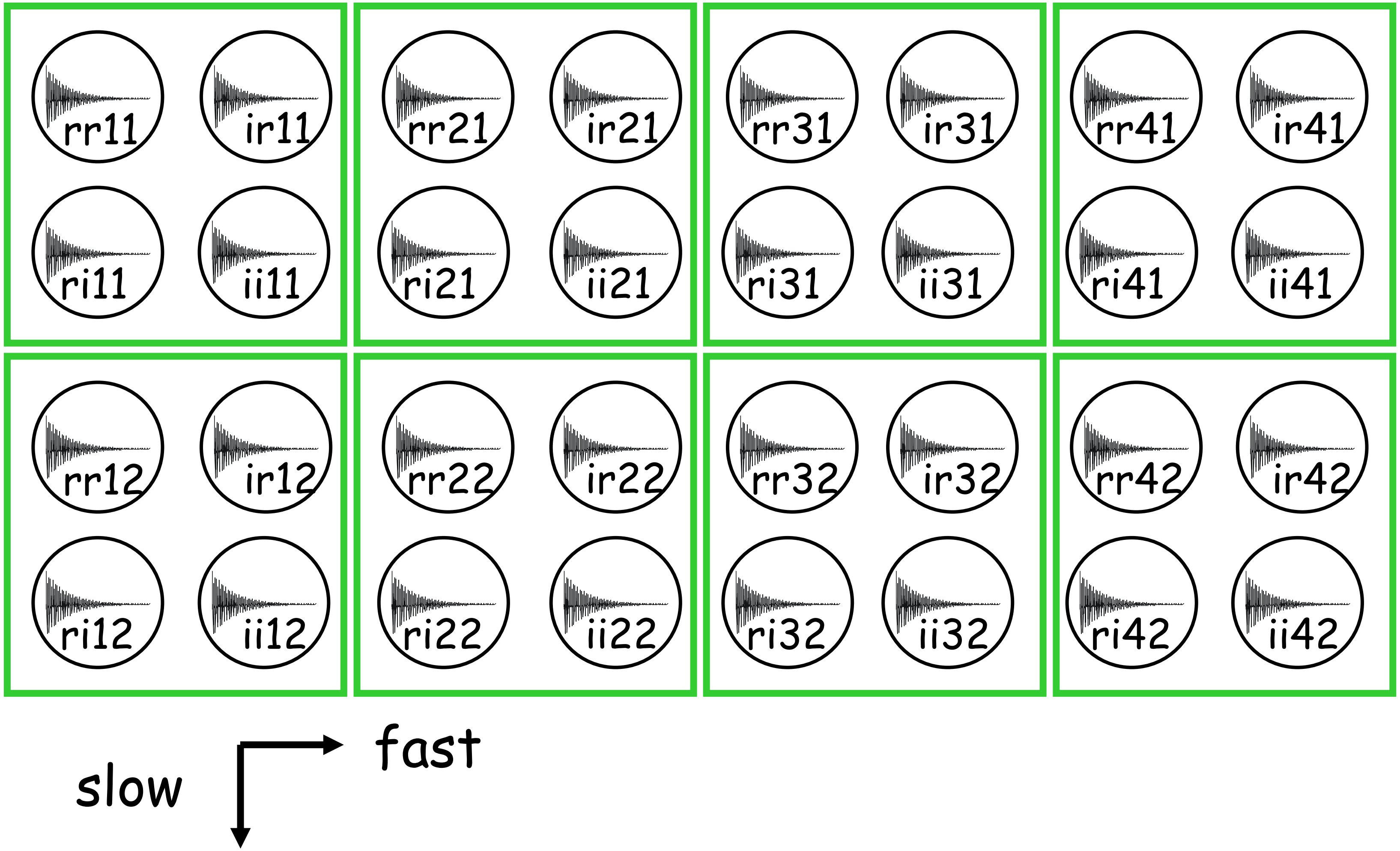
But 4 FIDs can also be grouped together to a "full point" since they belong to the same pair of evolution times

Sampling of full points in 3D experiments



It is then also possible to sample "full points" together, i.e. the 4 FIDs that belong to the same pair of evolution times

A 3D experiment is now a area of full points



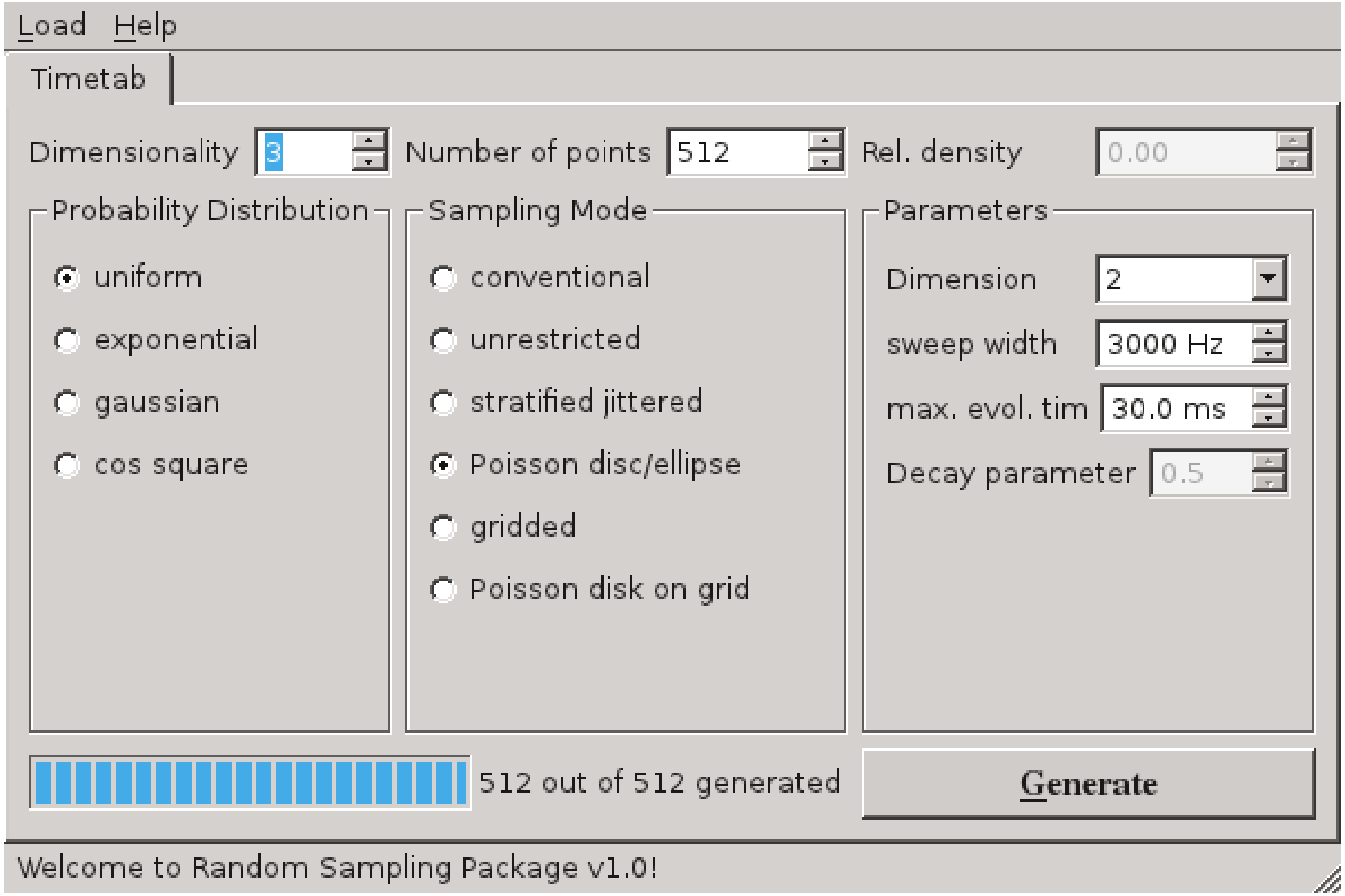
Sampling of full points in 3D experiments

- For conventional sampling the difference is the order of the FIDs in the ser-file (not really important) and the time between the recording of real and imaginary part of one time point in the formerly “slow” dimension
- Recording of “old-fashioned” planes is then not possible any more, two planes will always be recorded together

The sampling schedule

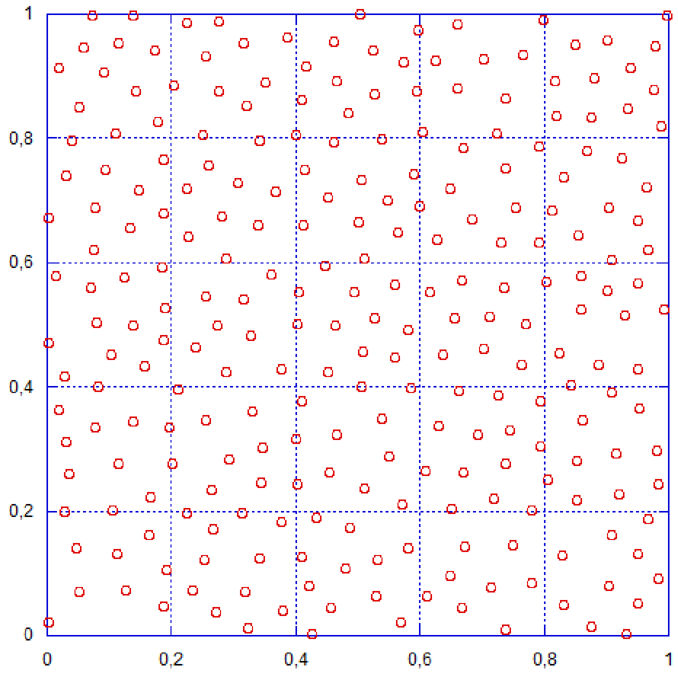
- The two indirect dimensions of a 3D are not viewed independently any more but as a two-dimensional area of “full points”
- The sampling schedule picks a certain distribution of “full points” from this area (e.g. 25% of the points)
- The least artifacts are obtained for a sampling schedule distributing the datapoints randomly

The "warsaw" software

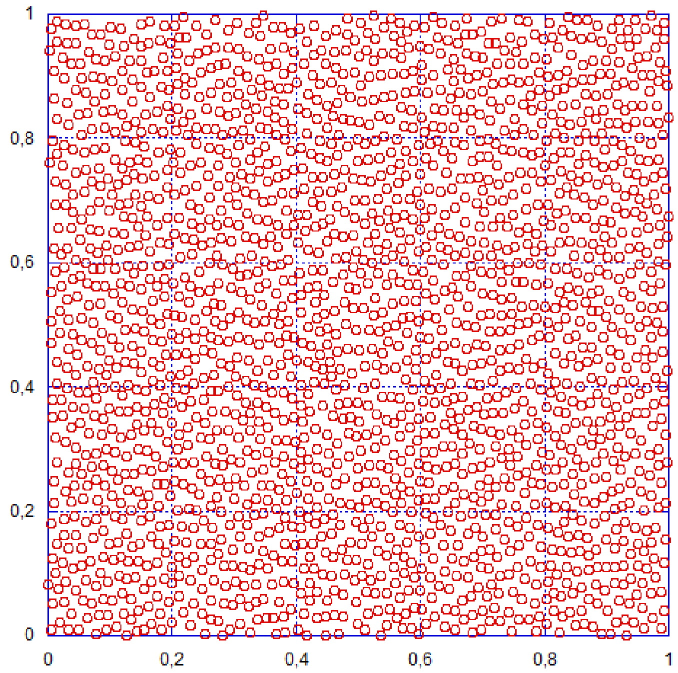


(runs under linux)

schedules are "general", they range from 0 to 1



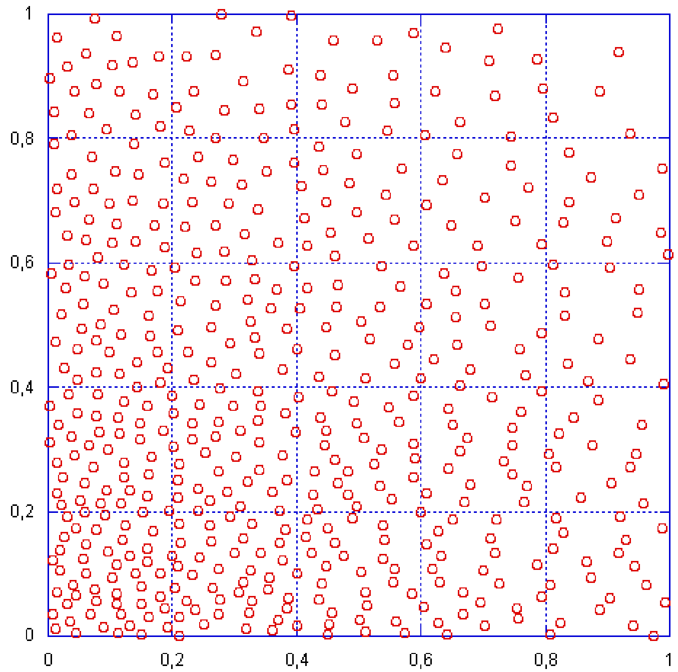
pois_uni_256:
sparse sampling



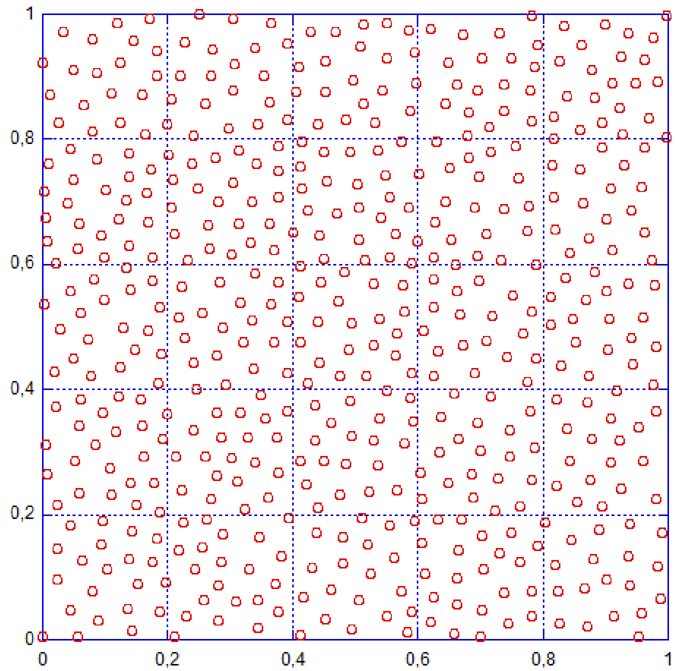
pois_uni_2048:
dense sampling

now only „full points" (o) are considered

schedules are "general", they range from 0 to 1

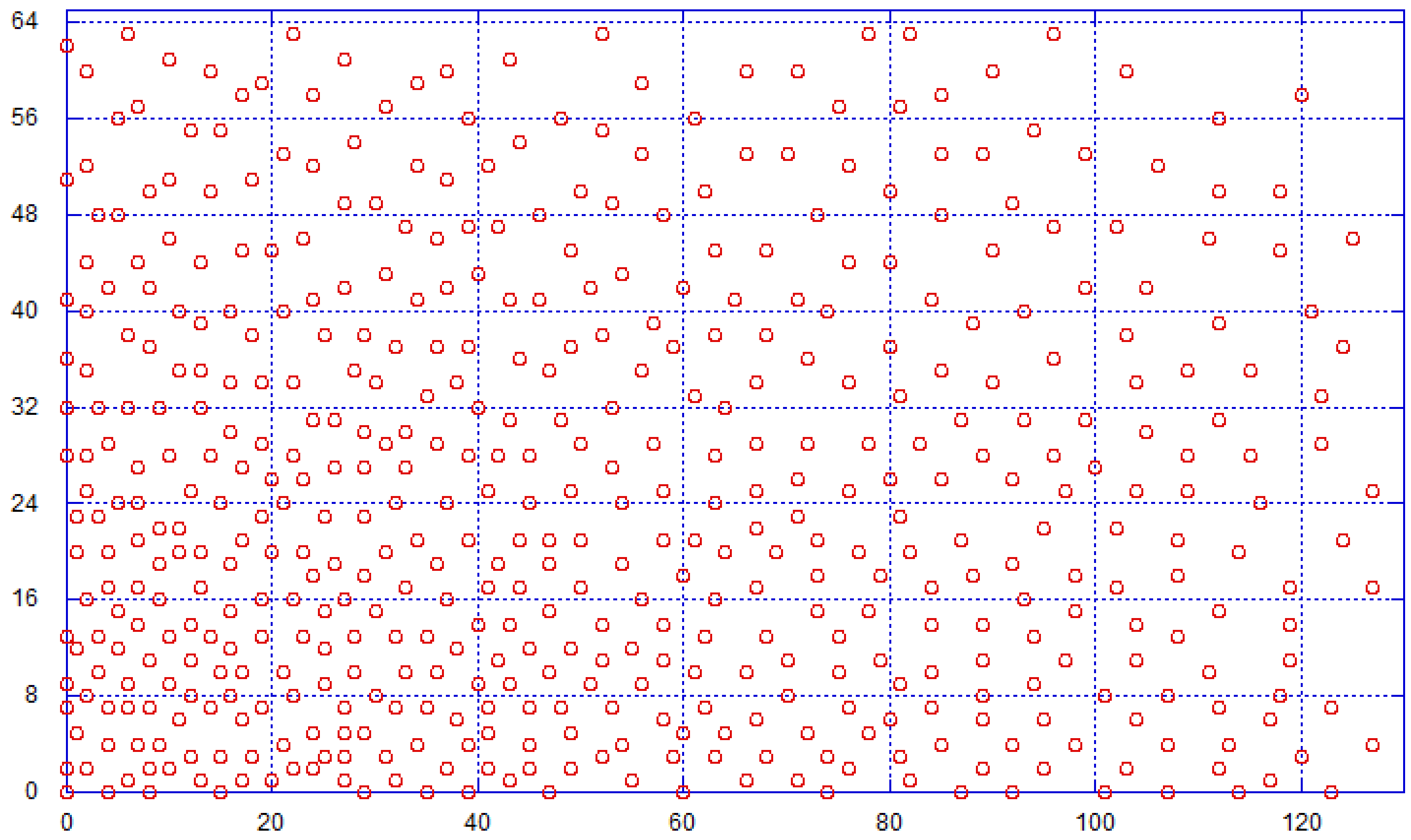


pois_exp_08_512:
exponential weight



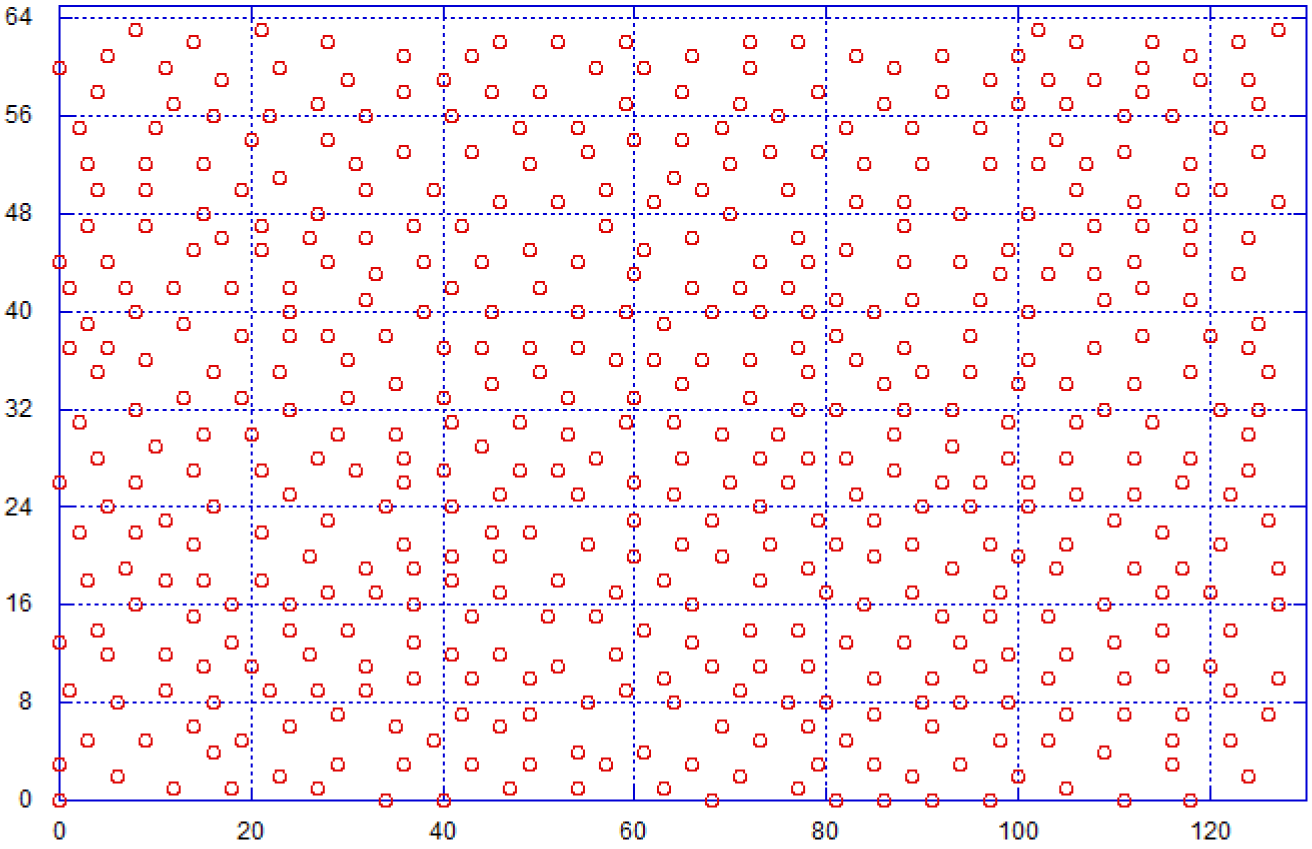
pois_uni_512:
uniform weight

The general schedule is converted to a specific one



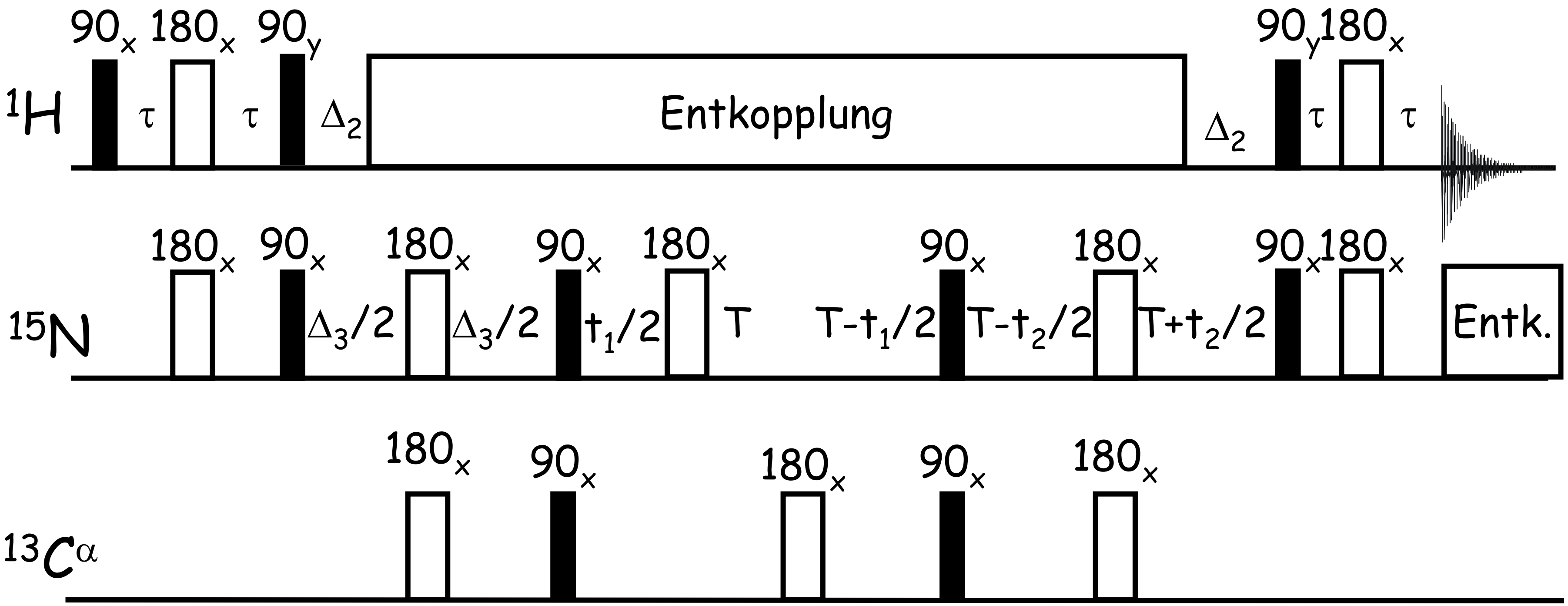
pois_exp_08_512 with 64 x 128 complex points

The general schedule is converted to a specific one



pois_uni_512 with 64 x 128 complex points

Das ct-HNCA mit zwei indirekten ct-Dimensionen ^{13}C und ^{15}N



ct-HNCA of SH3 @900 MHz

We are clearly in the sampling limit !

^{13}C: the ct-delay is 27 msec, the increment is 132 μsec

we can collect 200 complex points (400 FIDs)

^{15}N: the ct-delay is 22 msec, the increment is 220 μsec

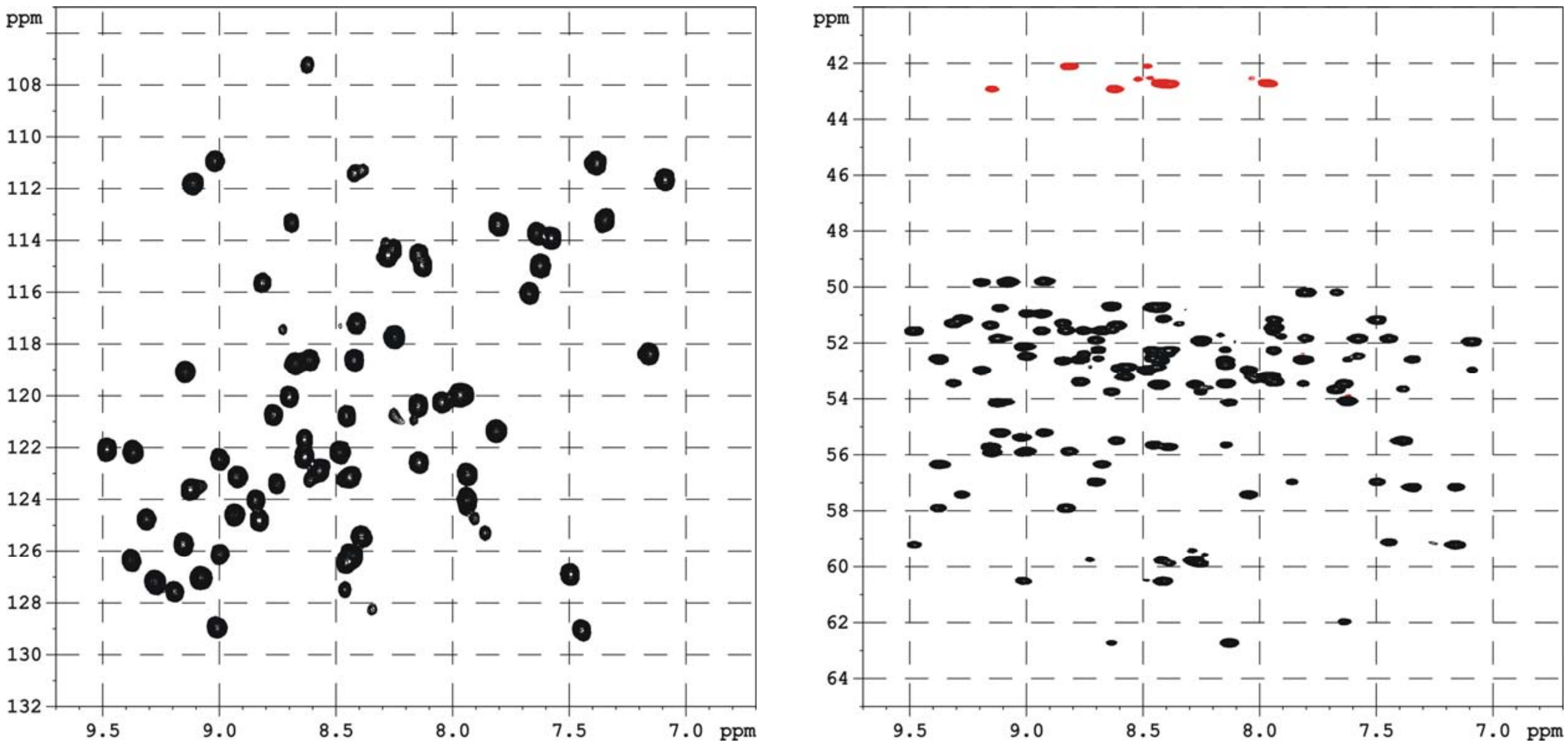
we can collect 100 complex points (200 FIDs)

The full 3D would therefore consist of 80 000 FIDs or 20 000 „full points"

to speed things up

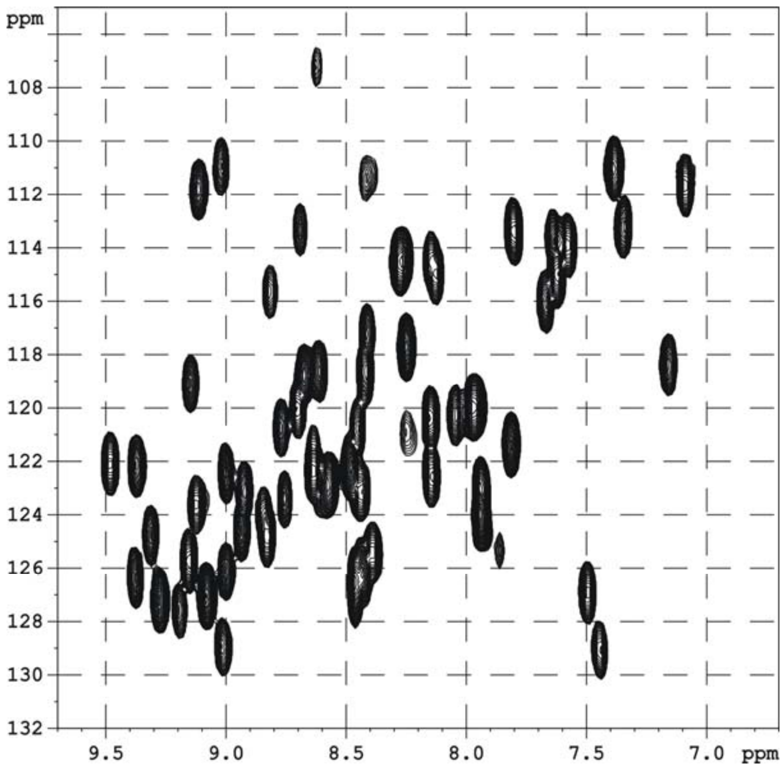
Assuming 4 scans and 0.8 sec relaxation delay this amounts to 80 hours, while 1 hour is enough for S/N !

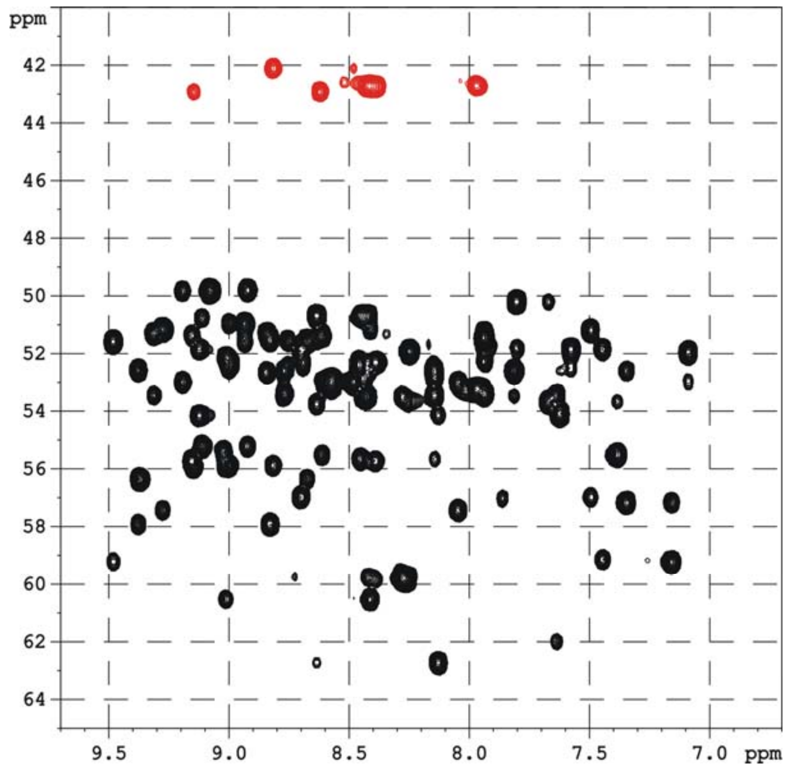
ct-HNCA of SH3 @900 MHz



Projections of a dataset with 100* x 200* = 20 000 full points, i.e. 80 hours spectrometer time

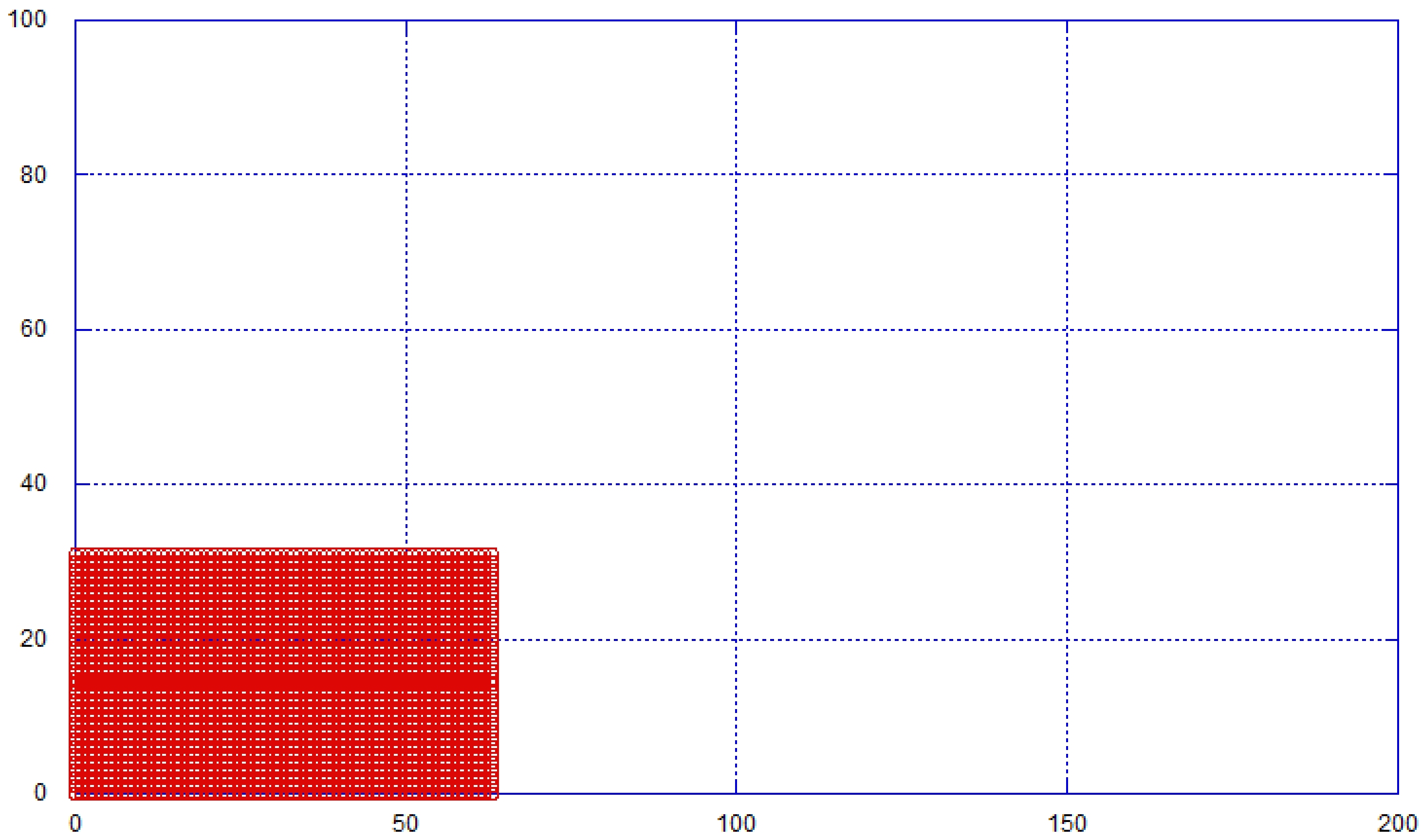
ct-HNCA of SH3 @900 MHz





if we cut this to 8 hours "classically" (32* x 64* = 2048 full points) we have less resolution

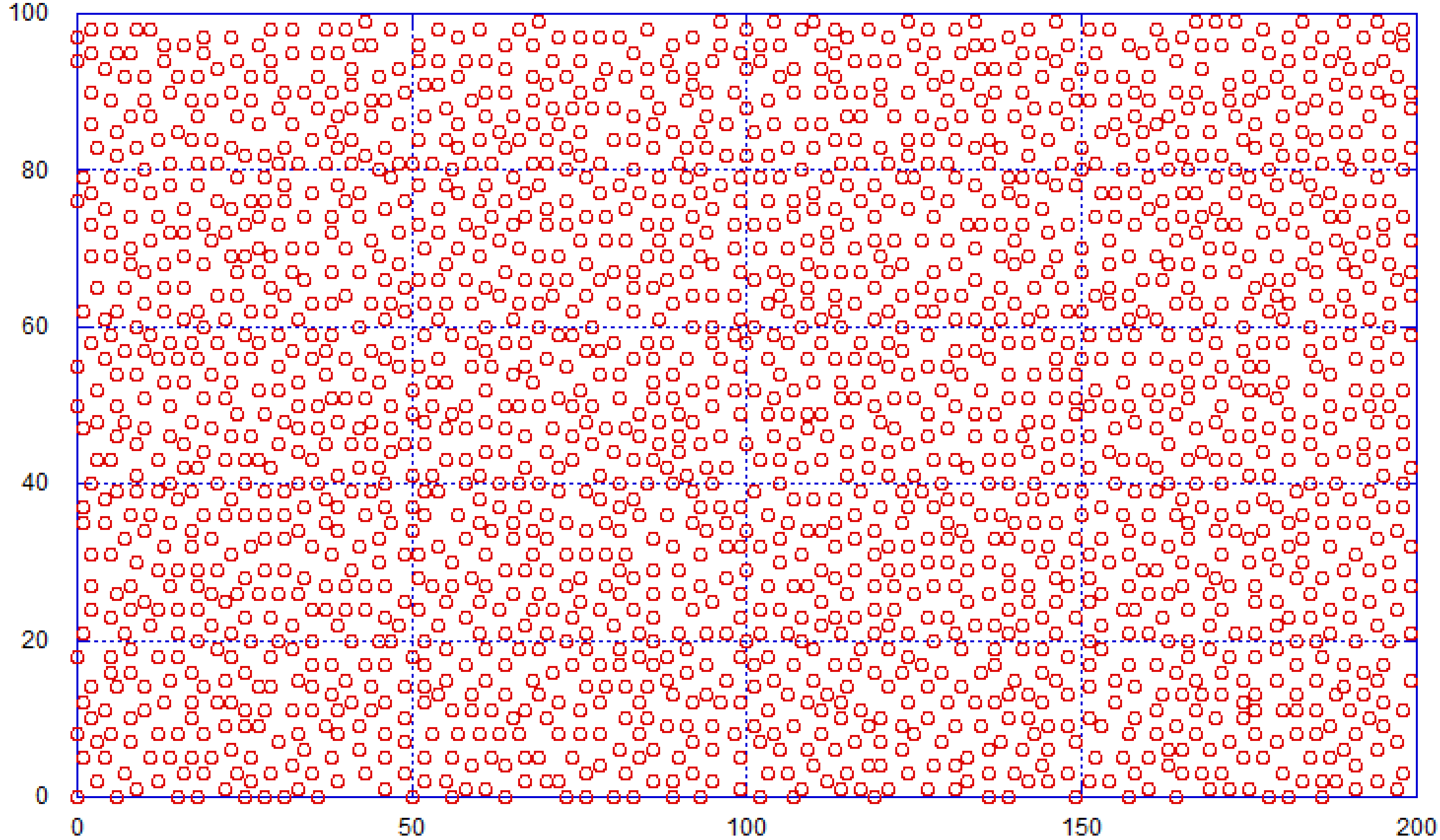
ct-HNCA of SH3 @900 MHz



by cutting to 8 hours the sampling is drastically reduced

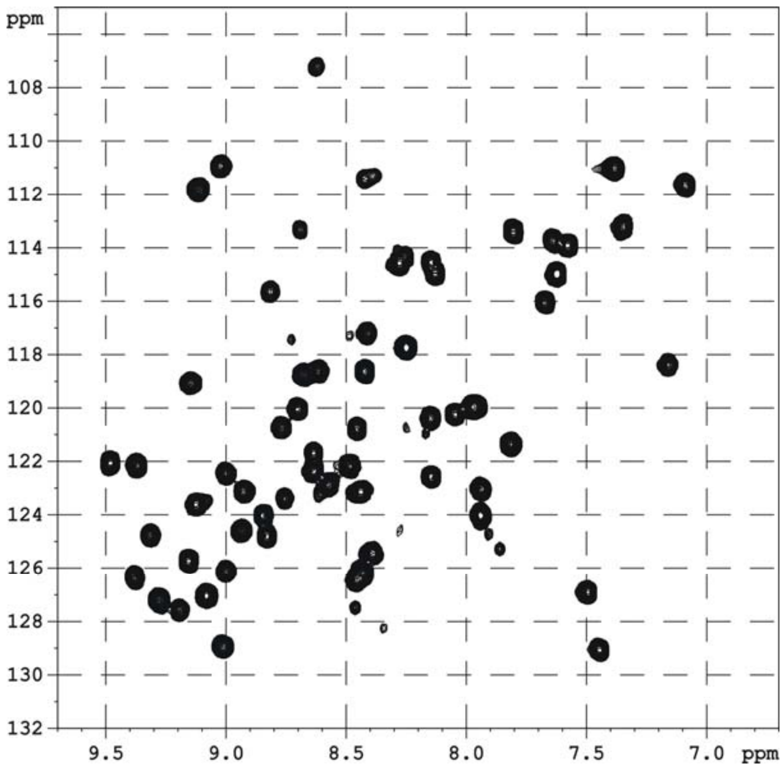
32* x 64* = 2048 full points

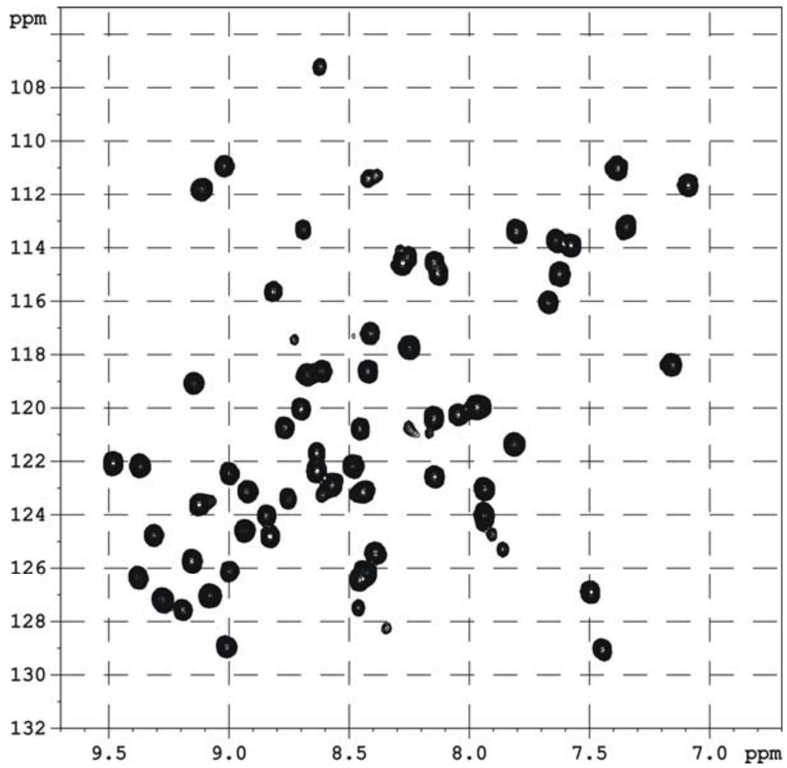
ct-HNCA of SH3 @900 MHz



pois_uni_2048 ⟶ pois_uni_2048_200_100

ct-HNCA of SH3 @900 MHz



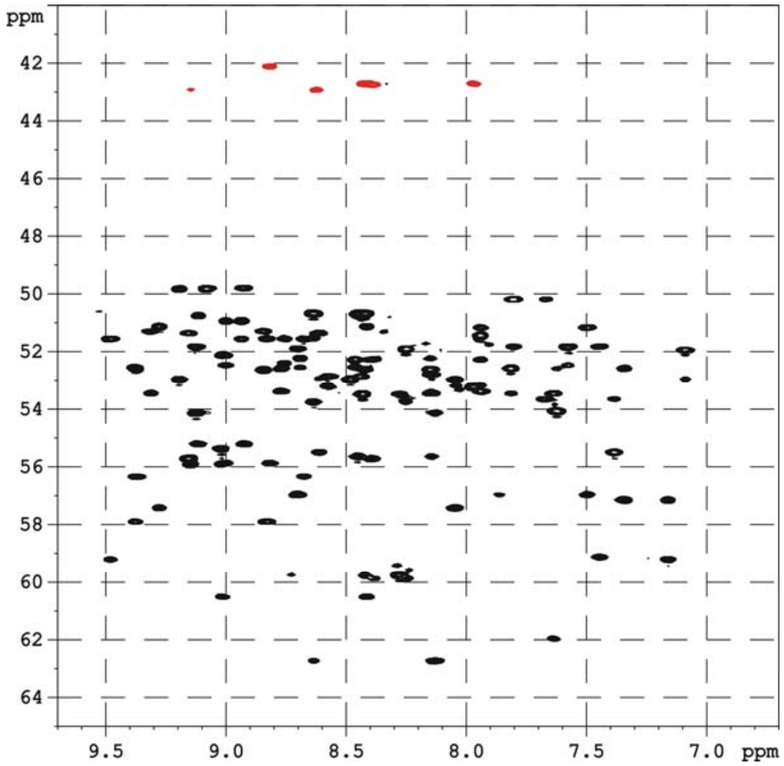


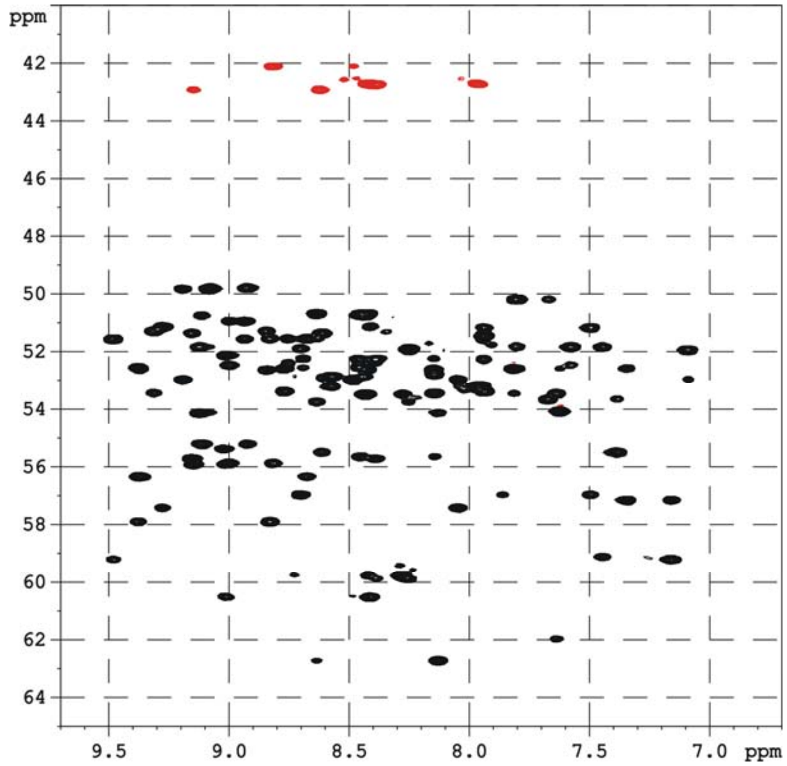
2048 instead of 20 000:

8 hours instead of 80

traditional

ct-HNCA of SH3 @900 MHz



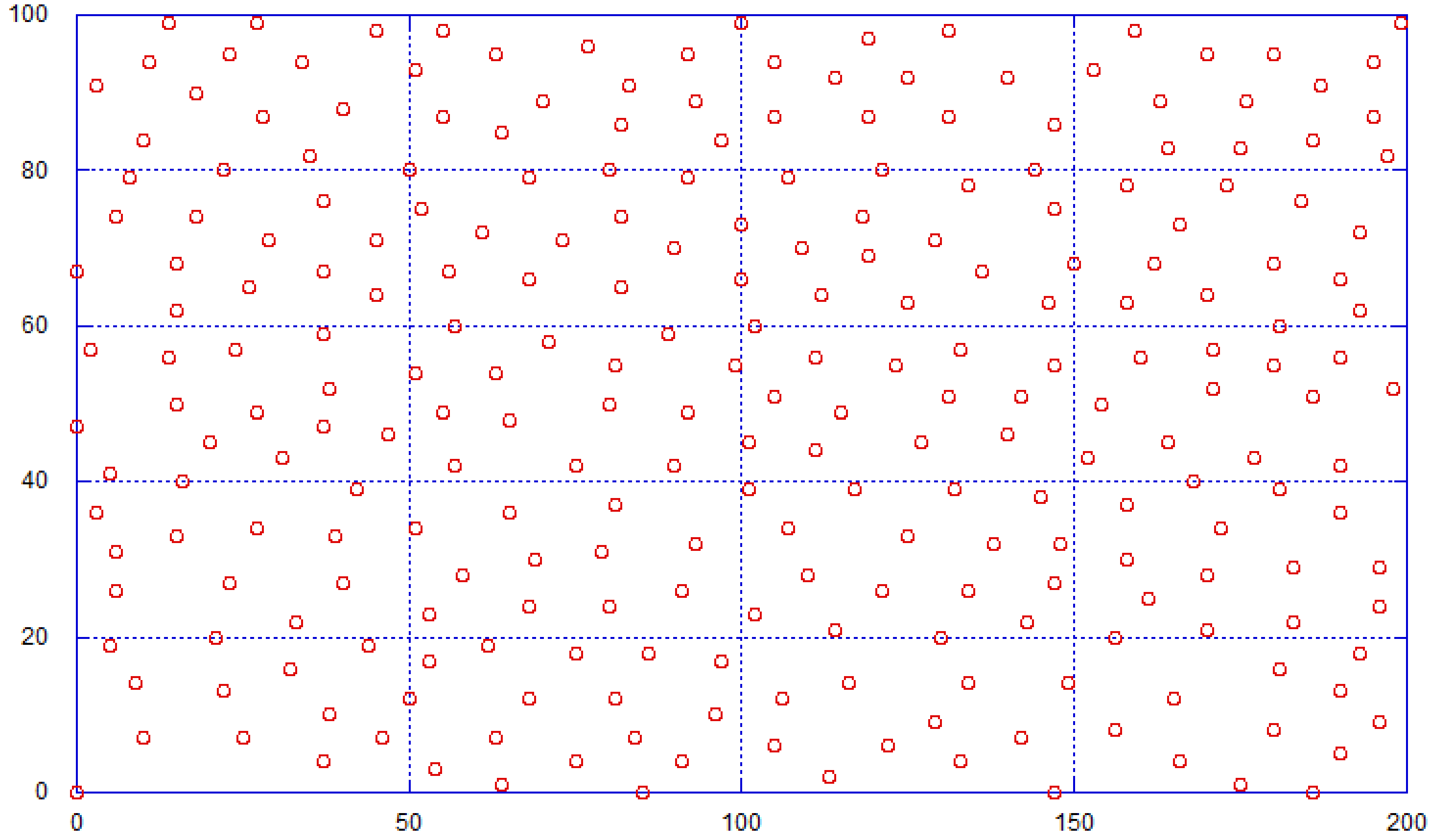


2048 instead of 20 000:

8 hours instead of 80

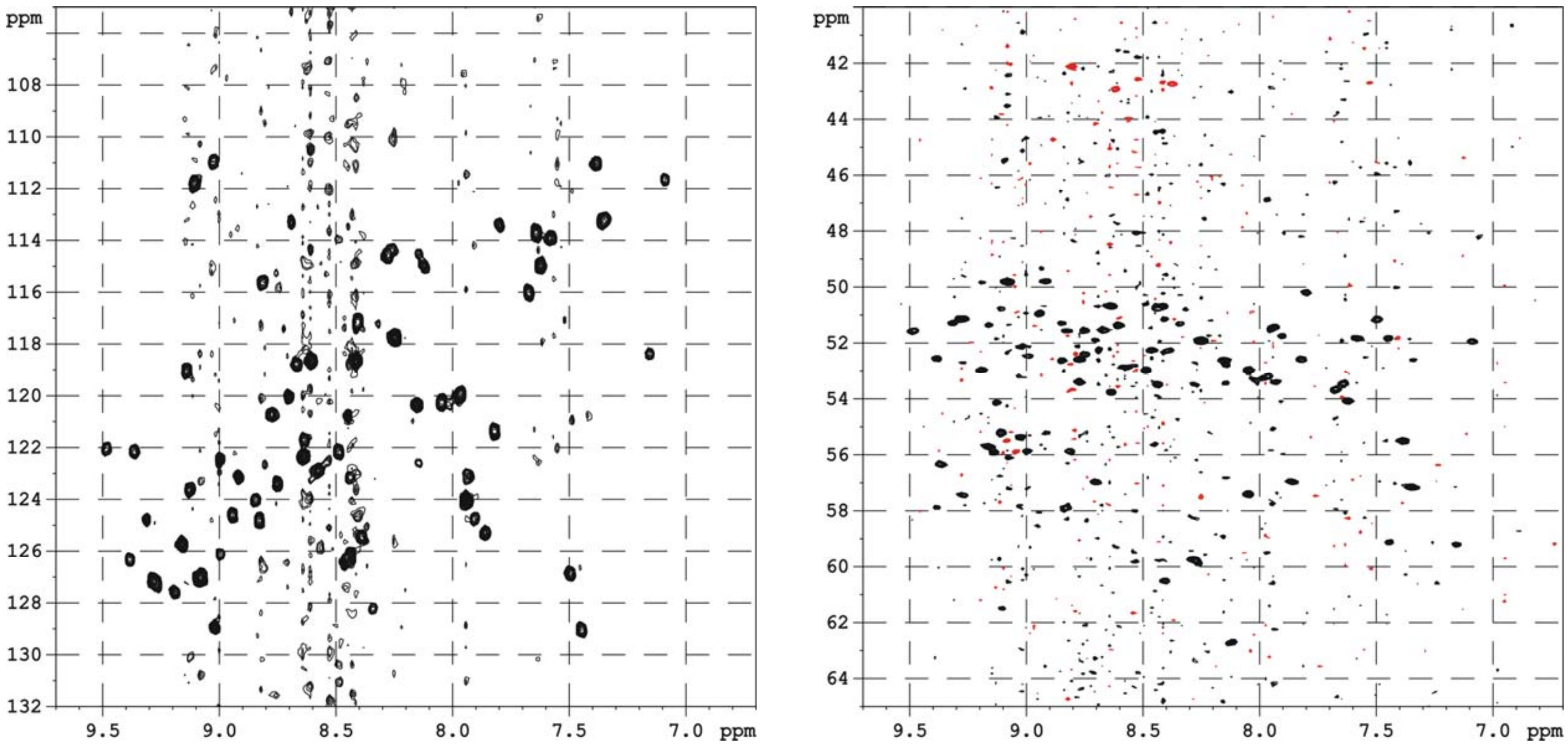
traditional

ct-HNCA of SH3 @900 MHz



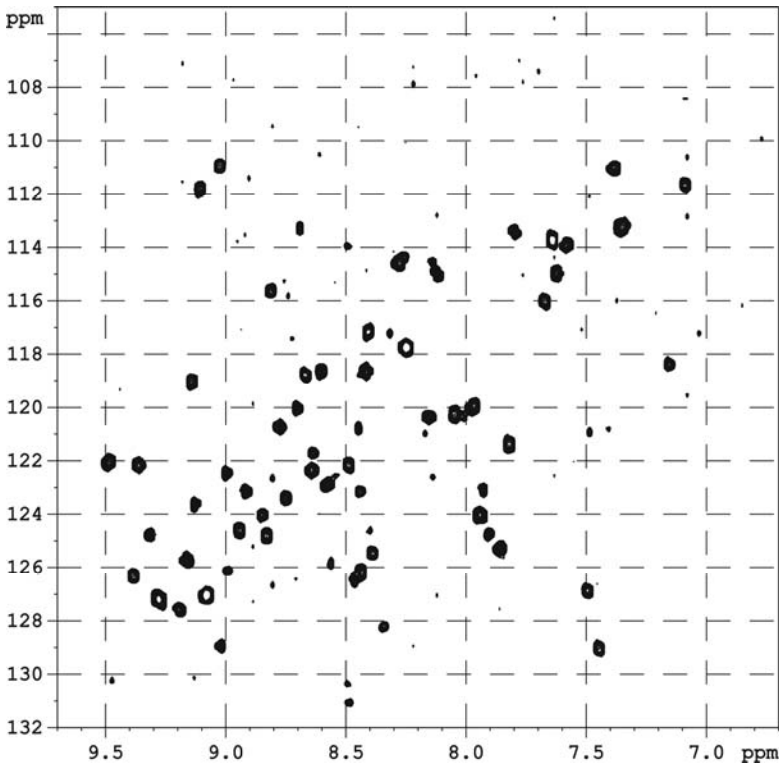
pois_uni_256 → pois_uni_256_200_100

ct-HNCA of SH3 @900 MHz



256 instead of 20 000: 1 hour instead of 80

ct-HNCA of SH3 @900 MHz



The quality can be improved by noise-subtraction

Increasing the number of scans (4 to 32) does not help

256 instead of 20 000: 1 hour instead of 80

Acquisition of NUS data

Bruker has introduced the "Orekhov-Software" (MDD) in topspin 3.1 together with acquisition tools

Lists

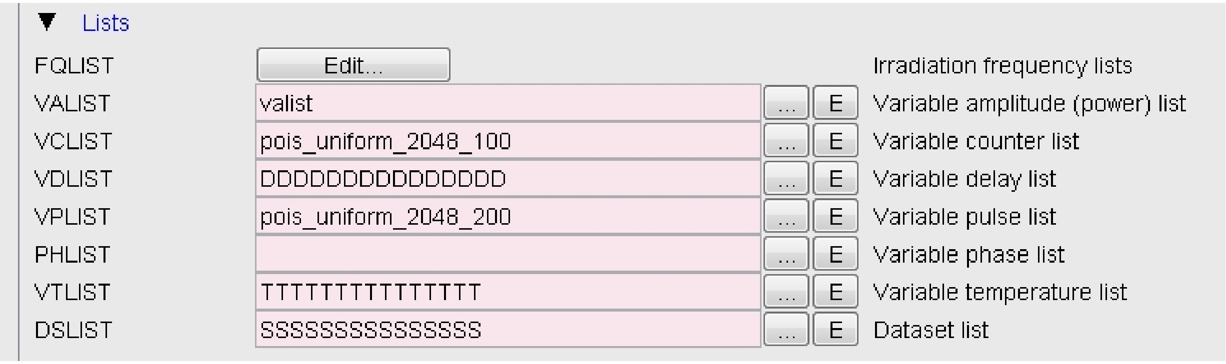
FQLIST	Edit...				Irradiation frequency lists
VALIST	valist			... E	Variable amplitude (power) list
VCLIST	lista			... E	Variable counter list
VDLIST	DDDDDDDDDDDDDDD			... E	Variable delay list
VPLIST	listb			... E	Variable pulse list
PHLIST				... E	Variable phase list
VTLIST	TTTTTTTTTTTTTTTT			... E	Variable temperature list
NUSLIST	automatic				Name of loopcounter list for NUS (Non Uniform Sampling)

NUS (Non Uniform Sampling) parameters

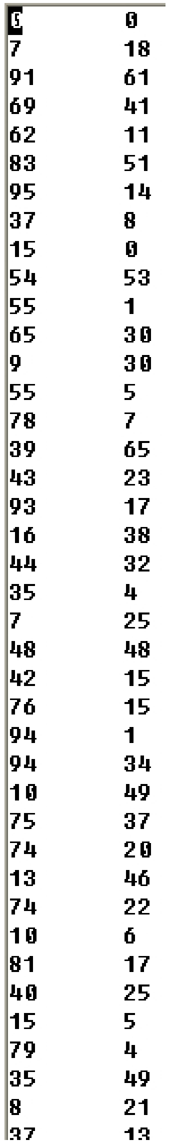
NusAMOUNT [%]	25			Amount of sparse sampling
NusPOINTS	64			Number of hypercomplex points in indirect dimension
NusJSP [Hz]	0	0	0	J-coupling
NusT2 [sec]	1	1	1	T2 relaxation
NusSEED	54321			Random generator seed
	Calculate			Calculate point spread function

Acquisition of NUS data

Using topspin 2.1 requires some tricks but works



Lists are created from the sampling schedule and the individual points are recorded



0	0
7	18
91	61
69	41
62	11
83	51
95	14
37	8
15	0
54	53
55	1
65	30
9	30
55	5
78	7
39	65
43	23
93	17
16	38
44	32
35	4
7	25
48	48
42	15
76	15
94	1
94	34
10	49
75	37
74	20
13	46
74	22
10	6
81	17
40	25
15	5
79	4
35	49
8	21
37	13

Acquisition of NUS data

```
define list<loopcounter> t1list=<$VCLIST>
define list<loopcounter> t2list=<$VPLIST>
```

```
;-----------------------------------
  20u
  "cnst7 = (t1list%2)*180"
  20u
  "cnst17 = (t2list%2)*180"
  20u
  "cnst31 = cnst7 + cnst17"

  3m ip5+cnst7
  3m ip8+cnst17
  3m ip31+cnst31
;-----------------------------------
```

```
;----------------------------------------NUS-Cal
  20u
  "d28 = d0 + in0*t1list"
  20u
  "d29 = d0 + in0*t1list - d12 - p19 - TAU"
  20u
  "d30 = d0 - in0*t1list"
  20u
  "d18 = d10 + in10*t2list"
;----------------------------------------NUS-del
```

3m t1list.inc
3m t2list.inc

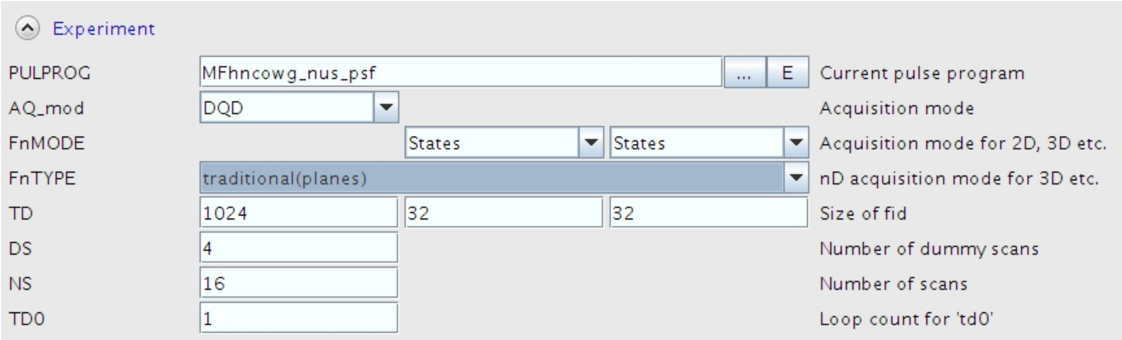
Acquisition of NUS data

Acquisition is now always done as full points

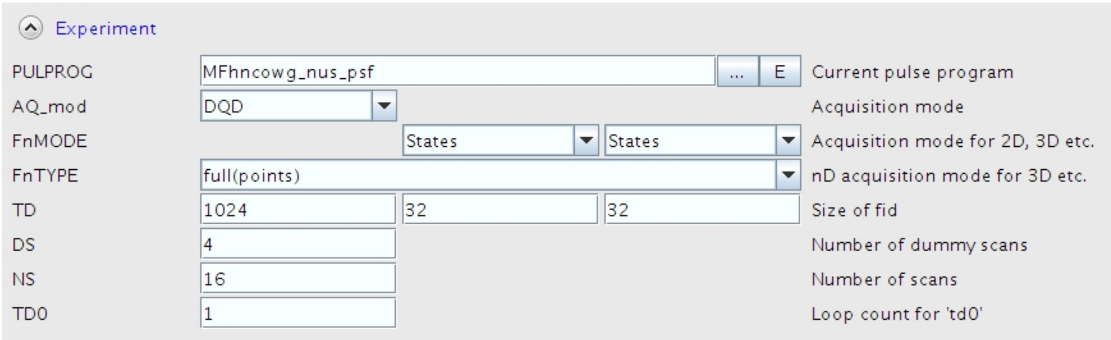
Recording "planes" gets more complicated since a full point plane consists of two traditional planes

Processing on the spectrometer is currently not trivial since 2.1 does not know what full points are

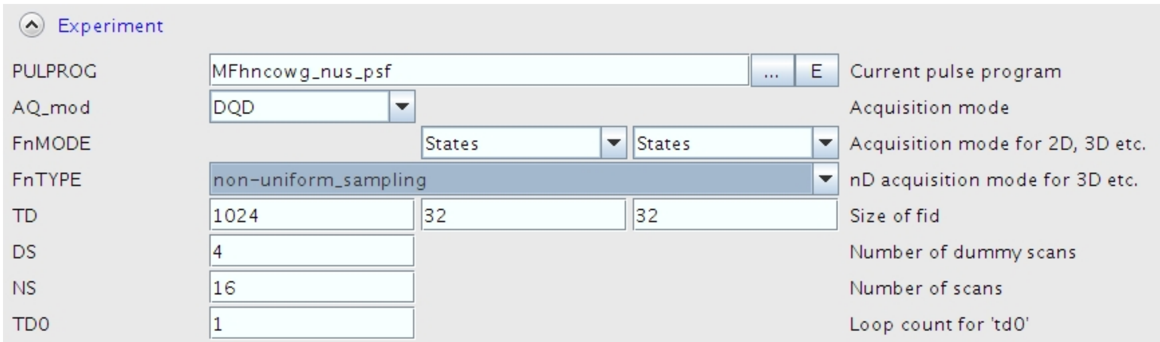
Processing of NUS data



The FnTYPE !



Monika has written a script to make the changes with „one click“

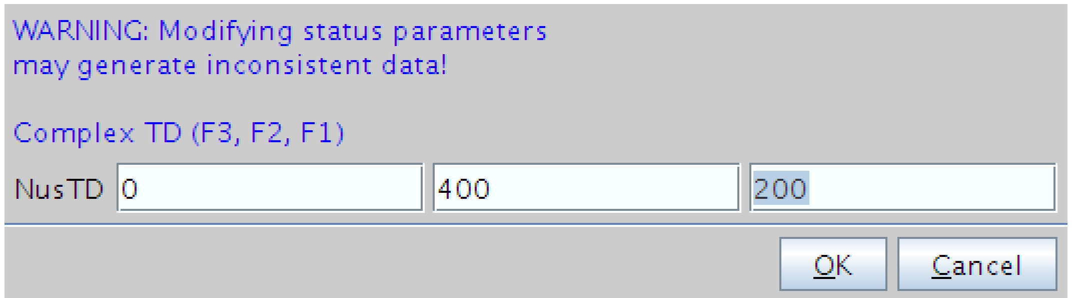


Processing of NUS data

The handling of TD is currently more complicated, for 2048 full points (with 4 FIDs each) the product of TD has to be 8192, e.g. 64 x 128, to be consistent with the size of the ser-file

But if NUS was done, the software needs to now the TD that was assumed during the selection of points

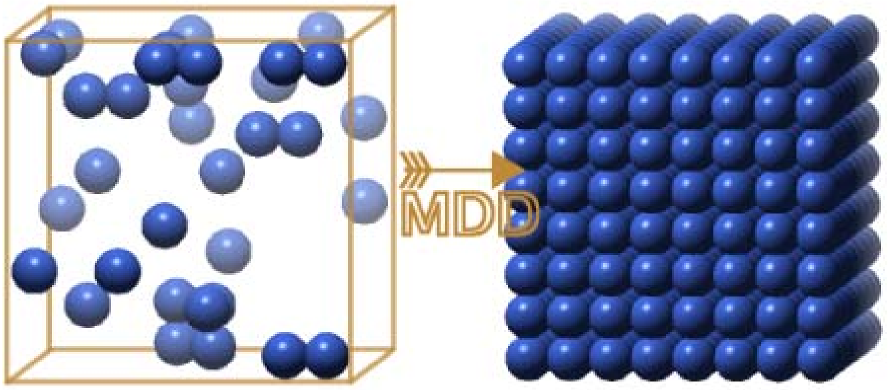
NUS-TD !!



Processing of NUS data

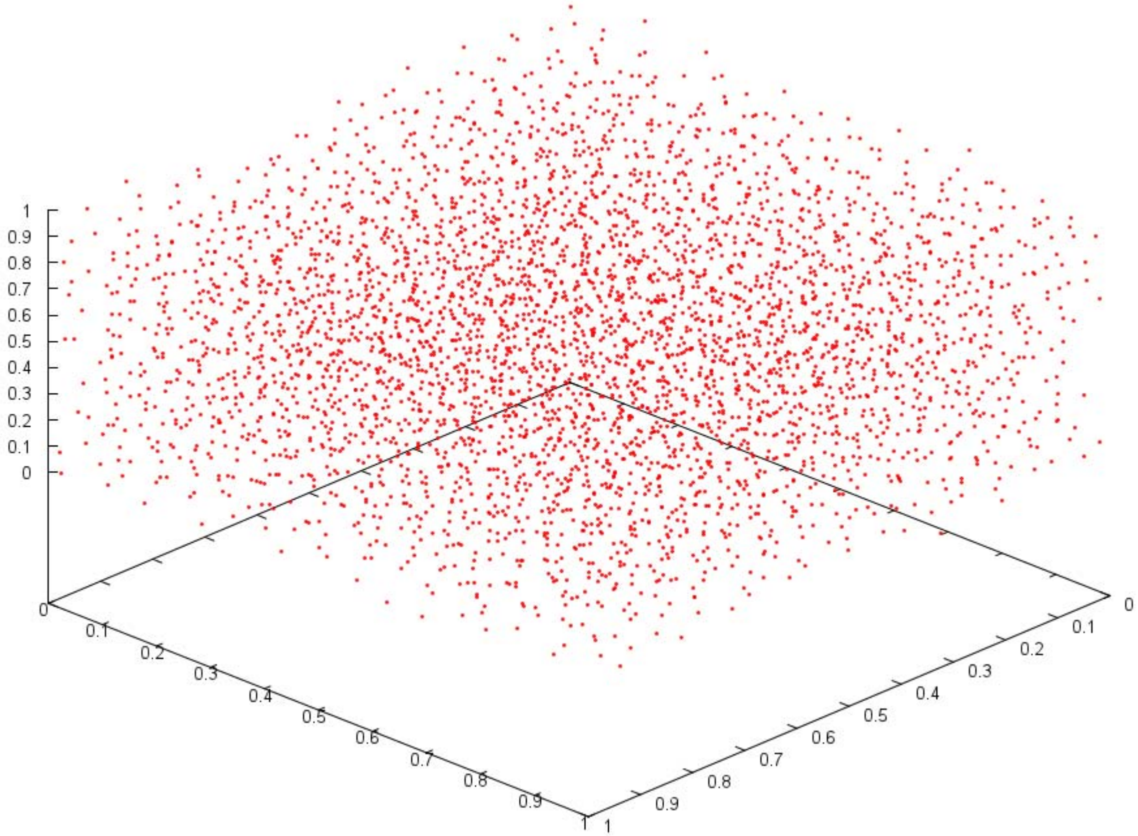
In the EXPNO directory there has to be a file "nuslist" (containing the sampling schedule) together with the acquisition parameter

With FnTYPE set to "non-unifom_sampling" the command "ftnd 0" starts the "MDD" software (Orekhov) and then a normal ft with windows functions and potential LP



Multi-Dimensional Decomposition

NUS is even more important in 4D spectra



pois_4D_uni_4096: a schedule for a 16* x 16* x 16* 4D

That's it