Test of Probe PH MASDVT1000S6 BL1.3 N/C/H NO_I/E , H170062/0001

1 Introduction

The probe was tested for achievable pulse width/RF field strength, resolution/line shape, signal-to-noise, stability during decoupling and CP, and double CP efficiency and stability. The tests were performed on 18th of August, 2020. This report was finished on the 13th of October, 2020.

2 Test Summary

2.1 Probe Performance

RF field strength and power requirements			
Irradiation type	pulse width/µs	RF field strength/kHz	Power (TopSpin) / W
^{1}H decoupling	1.8	140	45
^{13}C CP	3.0	83	40
^{15}N CP	3.5	70	82
Resolution			
nucleus	line width	Sample/parameters	comment
^{13}C	< 2.0 Hz	Adamantane, ns=16, d1=10s	40 kHz spinning, 20 kHz SPINAL64 decoupling
Signal to noise			
nucleus	S/N	Sample/parameters	comment
^{13}C	94	Glycine (^{13}C and ^{15}N in natural abundance), 10 kHz spinning, ns=64, aq=14 ms, 140 kHz decoupling	
^{15}N	15	Glycine (^{13}C and ^{15}N in natural abundance), 10 kHz spinning, ns=64, aq=29ms, 140 kHz decoupling	
CP stability			
nucleus	result	Sample/parameters	comment
^{15}N	ok	Glycine (^{13}C and ^{15}N in natural abundance), ns=4, aq=29 ms, 140 kHz decoupling, d1=5s 256 spectra with 4scans each at 5s recycle delay and 5.04 s repetition time	test duration: 1h 25 min

Double CP efficiency			
sample type	efficiency	Sample/ parameters	comment
Full rotor	52%	Glycine-1,2-$^{13}C_2$-^{15}N, full spinner, 10 kHz spinning, ^{15}N-^{13}C contact: 16 ms, 90%t to 100% ramped ^{13}C pulse centered at 25 kHz, square ^{15}N pulse at 35 kHz, ^{1}H-^{15}N contact: 3 ms 50% to 100% ramped ^{1}H pulse centered at 80 kHz, square ^{15}N pulse at 70 kHz, 140 kHz decoupling	^{13}C at -1 sideband condition in ^{15}N-^{13}C, contact parameters optimized on sample
	44%	Glycine-1,2-$^{13}C_2$-^{15}N, full spinner, 10 kHz spinning, ^{15}N-^{13}C contact: 20 ms, 90%t to 100% ramped ^{13}C pulse centered at 45 kHz, square ^{15}N pulse at 35 kHz, ^{1}H-^{15}N contact: 3 ms 50% to 100% ramped ^{1}H pulse centered at 80 kHz, square ^{15}N pulse at 70 kHz, 140 kHz decoupling	^{13}C at +1 sideband condition in ^{15}N-^{13}C, contact parameters optimized on sample
Double CP stability			
nucleus	result	sample/parameters	comment
^{1}H,^{15}N,^{13}C	ok	Glycine-1,2-$^{13}C_2$-^{15}N, full spinner, 10 kHz spinning, ^{15}N-^{13}C contact: 20 ms, 90% to 100% ramped ^{13}C pulse centered at 55 kHz, square ^{15}N pulse at 32 kHz, ^{1}H-^{15}N contact: 3 ms, 50% to 100% ramped ^{1}H pulse centered at 80 kHz, square ^{15}N pulse at 70 kHz, 140 kHz decoupling 512 spectra with 8 scans each at 5 s recycle delay, total number of scans: 4096	^{13}C at +2 sideband condition total experiment time: 5 h 40 min

Notes:

1) The ^{15}N signal to noise test was run at 10 kHz spinning as defined for a 1 GHz system. As the ^{15}N figure is rather low compared to the results obtained for comparable 800 MHz probes the test were run at 8 kHz spinning. An S/N of 17 was found (using optimized 50% to 100% or 70% to 100% ramped ^{1}H contact pulses, respectively). This figure is not significantly different from the figure of 15 determined at 10 kHz spinning.
2) All double CP efficiencies are determined by comparing the intensity of the C_α signal in the double cross polarization experiment ($^{1}H \rightarrow {}^{15}N \rightarrow {}^{13}C$) to the intensity in the single cross polarization experiment ($^{1}H \rightarrow {}^{13}C$) for the same sample. The single cross polarization experiment was optimized using the standard 50% to 100% ramp shaped ^{1}H contact.
3) The double CP stability test was run at the +2 sideband condition (which is not the most efficient one) for ^{13}C deliberately to stress the probe.

3 Experimental

3.1 Spectrometer setup

Instrument: Avance Neo 1000 SB

TopSpin: 4.0.9 (of 2020-05-25 14:09:09)

3.2 Standard Temperature settings

- T(set)= 20°C
- VT=500 l/h
- *p*(bearing) and *p*(drive) as set by the MAS III unit (rotation profile: Generic 0.7 mm)
- Regulation on TC2 (probe has a single thermocouple, only)
- Chiller=BCU II, power = strong

3.3 External Filters

Filters were used on all probe channels throughout:

^{15}N channel: ^{15}N LRP filter
^{13}C channel: ^{13}C-^{23}Na BP filter
^{1}H channel: ^{1}H BP filter

3.4 Double Cross Polarization Experiment

This experiment was optimized using a 90% to 100% ramped ^{13}C contact pulse for the $^{15}N \rightarrow ^{13}C$ contact, no other shapes (square, tangential) were tried.

The determined maximum efficiencies (-1, +1, and +2 sideband conditions) are good. Long contact pulses were possible indicating that the probe's tuning and matching are stable during the pulses.

4 Figures

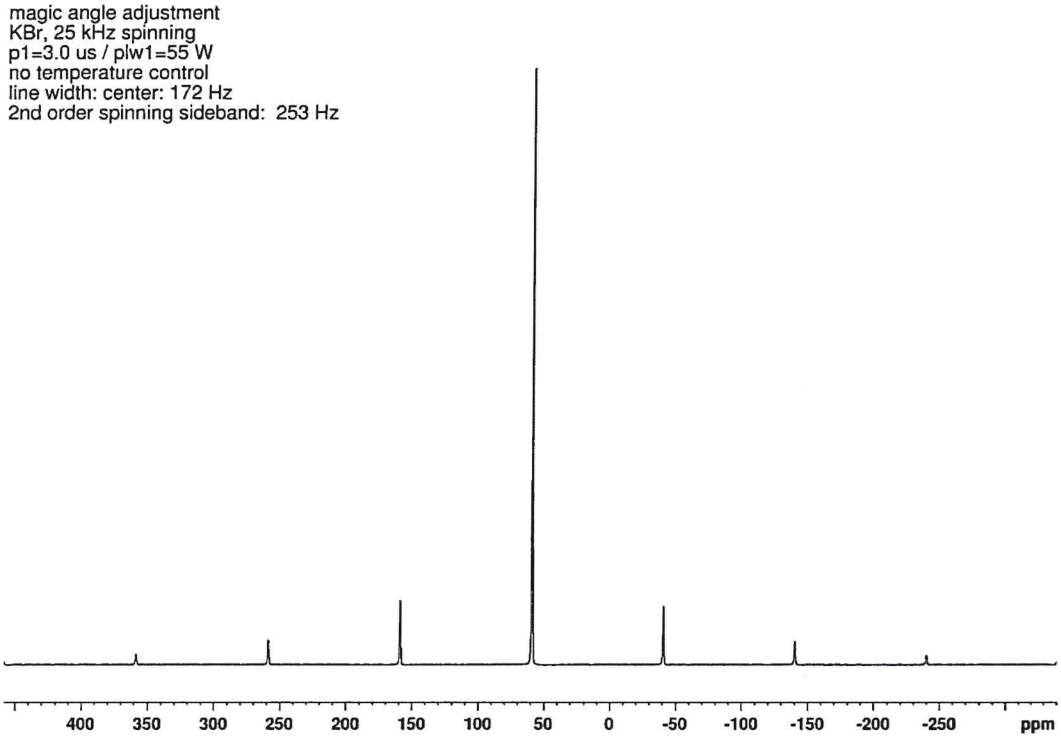

Figure 1: Magic angle adjustment: ^{79}Br spectrum of KBr at 25 kHz spinning.

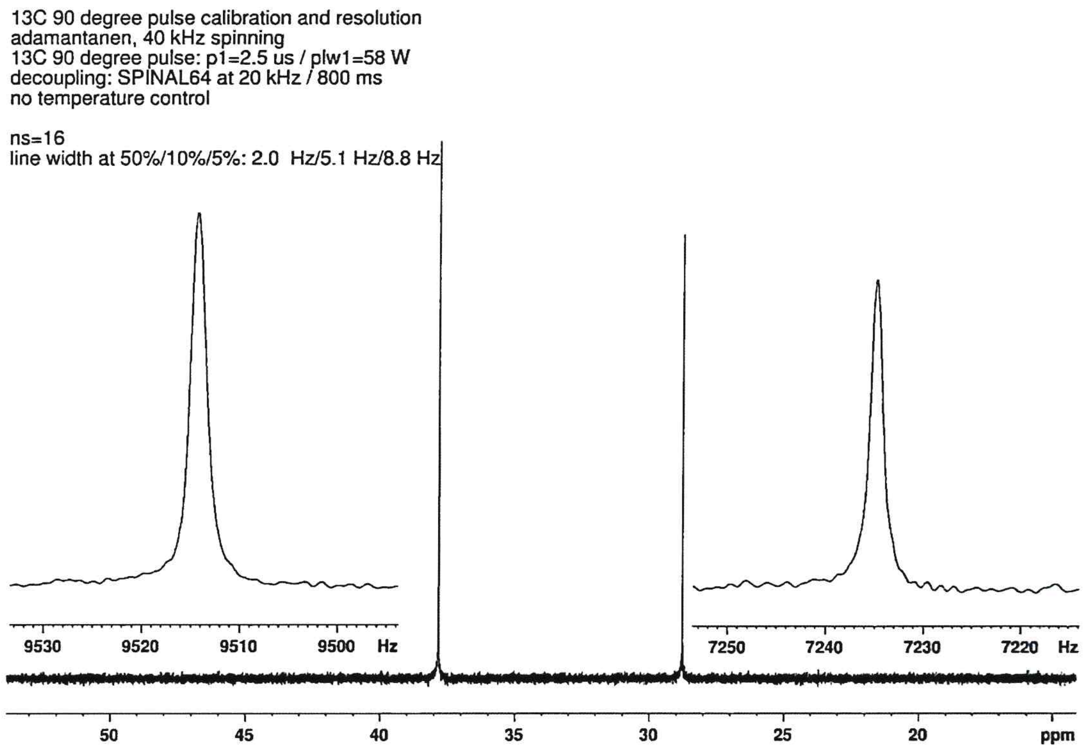

Figure 2: ^{13}C resolution test: ^{13}C spectrum (direct excitation) of adamantane at 40 kHz spinning.

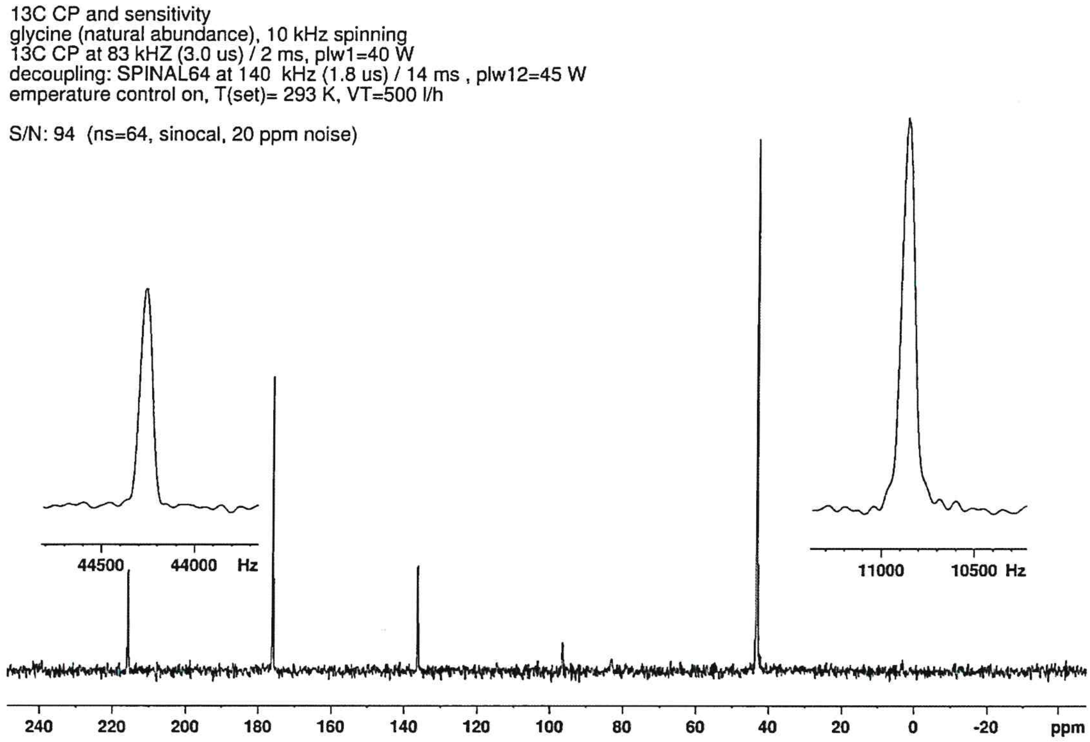

Figure 3: ^{13}C sensitivity (S/N) test: ^{13}C spectrum of glycine (natural abundance) at 10 kHz spinning.

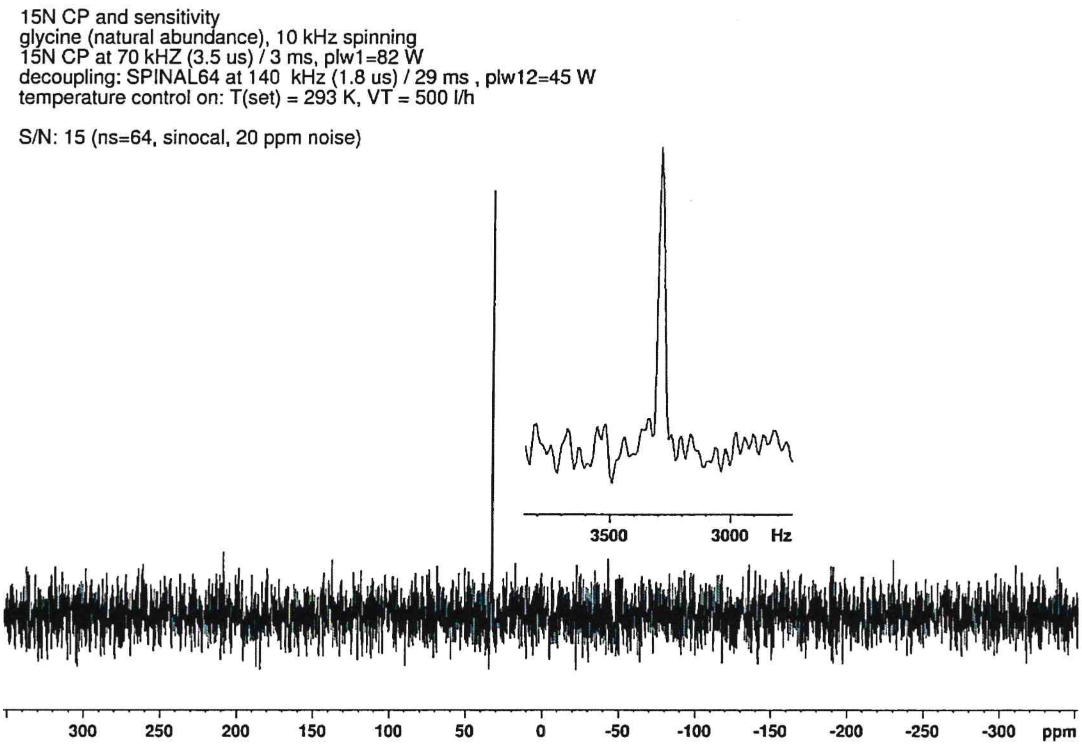

Figure 4: ^{15}N sensitivity (S/N) test: ^{15}N spectrum of glycine (natural abundance) at 10 kHz spinning.

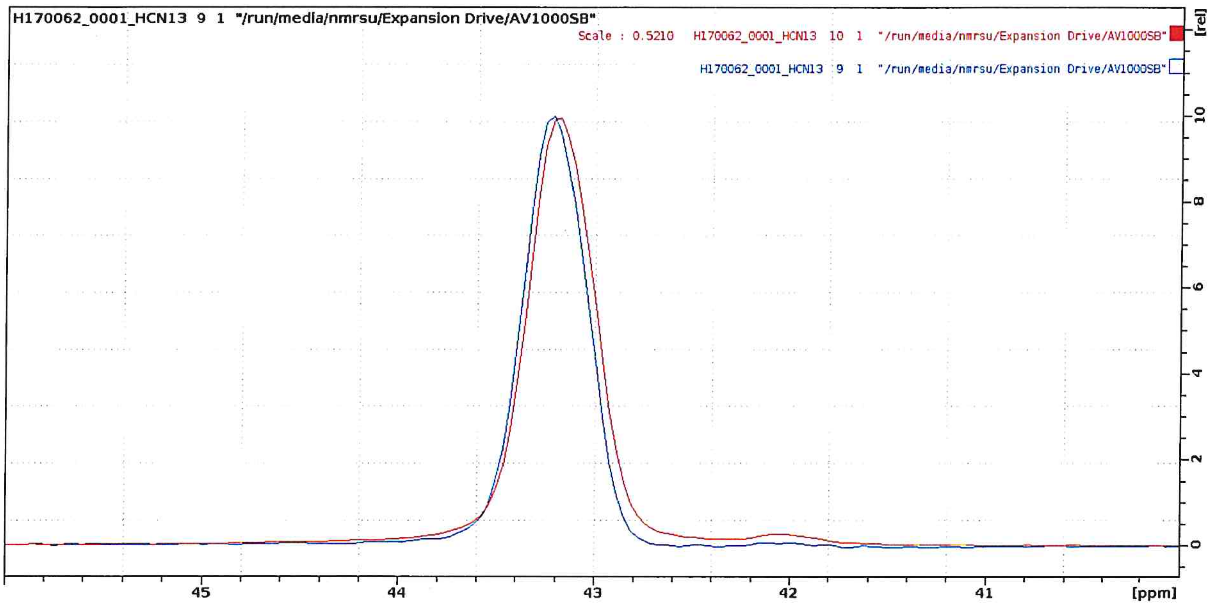

Figure 5: ^{1}H-^{15}N-^{13}C double CP efficiency test: Glycine-1,2-$^{13}C_2$-^{15}N at 10 kHz spinning. Blue line: double CP experiment at the -1 side band CP condition for ^{13}C, red line: standard CP experiment scaled by down by a factor of 0.52

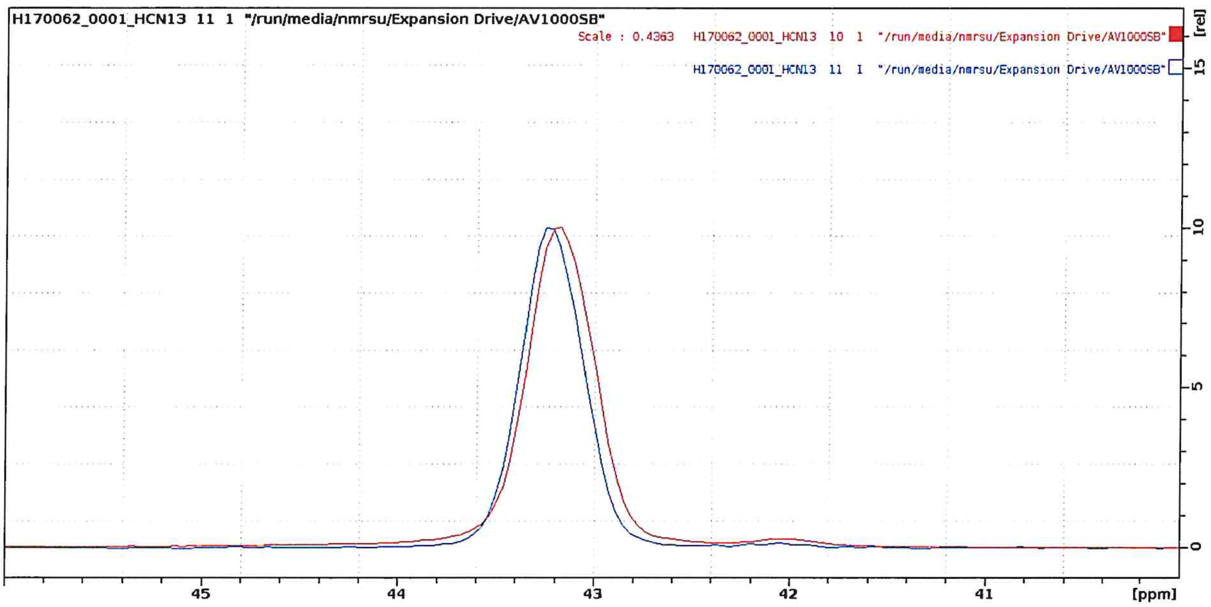

Figure 6: ^{1}H-^{15}N-^{13}C double CP efficiency test: Glycine-1,2-$^{13}C_2$-^{15}N at 10 kHz spinning. Blue line: double CP experiment at the +1 side band CP condition for ^{13}C, red line: standard CP experiment scaled by down by a factor of 0.44.

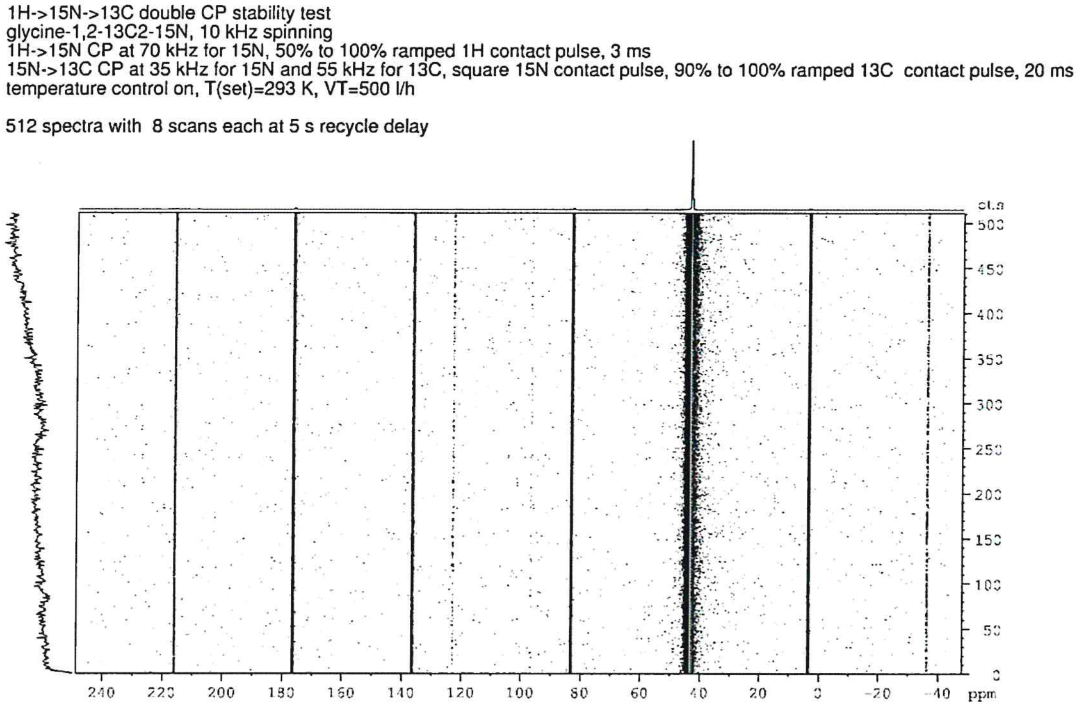

Figure 7: ^{1}H-^{15}N-^{13}C double CP stability test for glycine-1,2-$^{13}C_2$-^{15}N spinning at 10 kHz: 512 spectra with 8 scans each at 5 s recycle delay. Column projection (left) shows amplitude stability of CP signal, row projection (top) shows maximum noise compared to maximum signal.

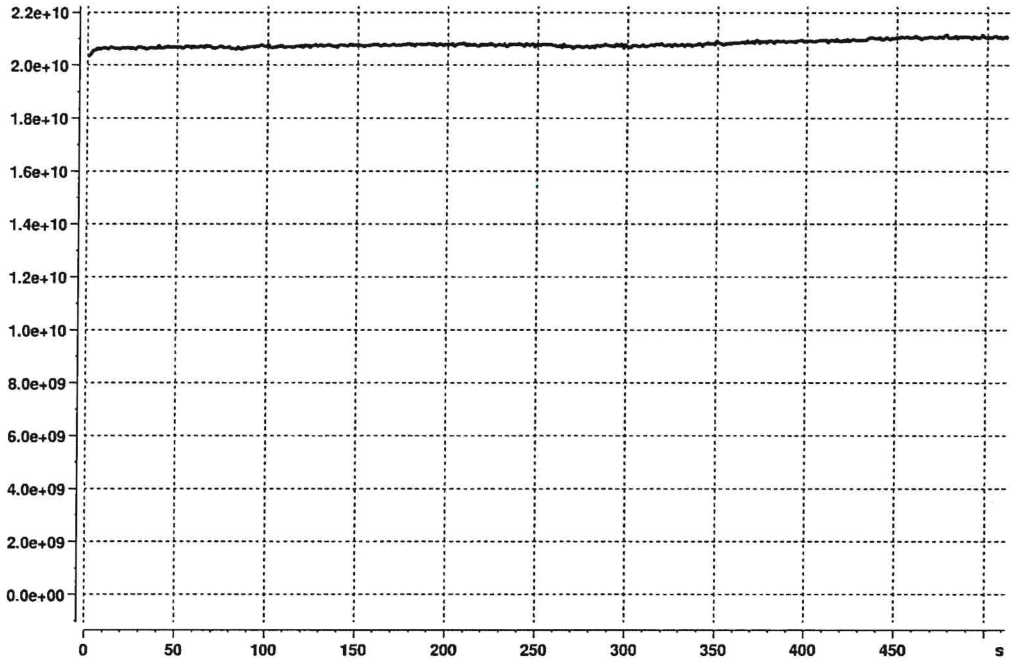

Figure 8: Expanded display of the column projection in Figure 7: signal amplitude is stable over time and the noise stays constant.

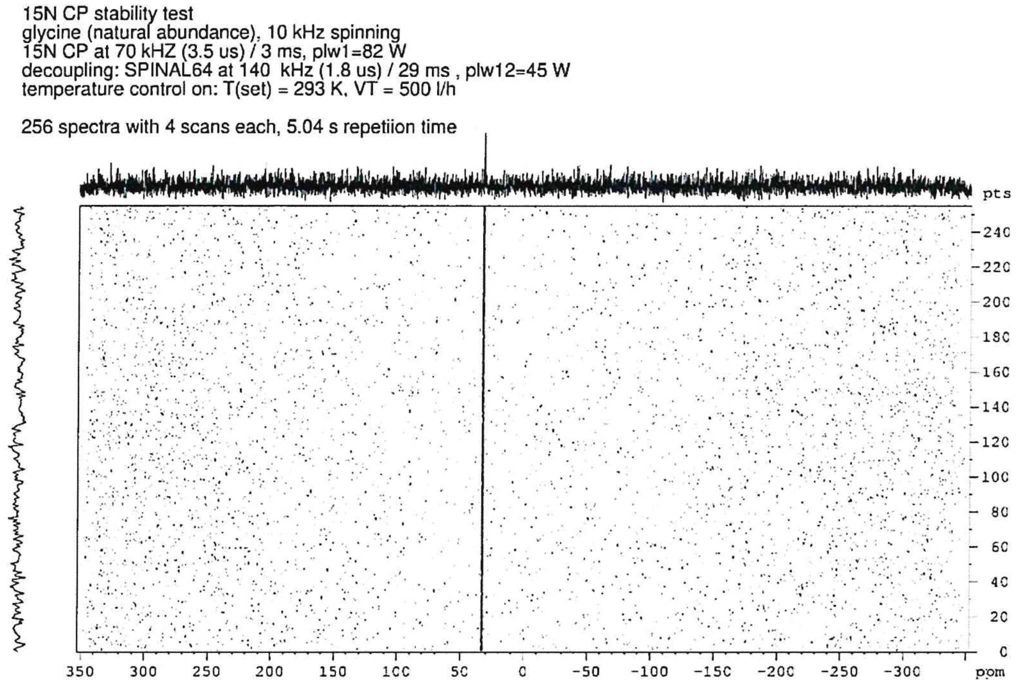

Figure 9: ^{15}N CP stability test run on glycine (natural abundance) at 10 kHz spinning: 256 spectra with 4 scans each at 5 s recycle delay and 5.04 s repetition time. Column projection (left) shows amplitude stability of CP signal, row projection (top) shows maximum noise compared to maximum signal.

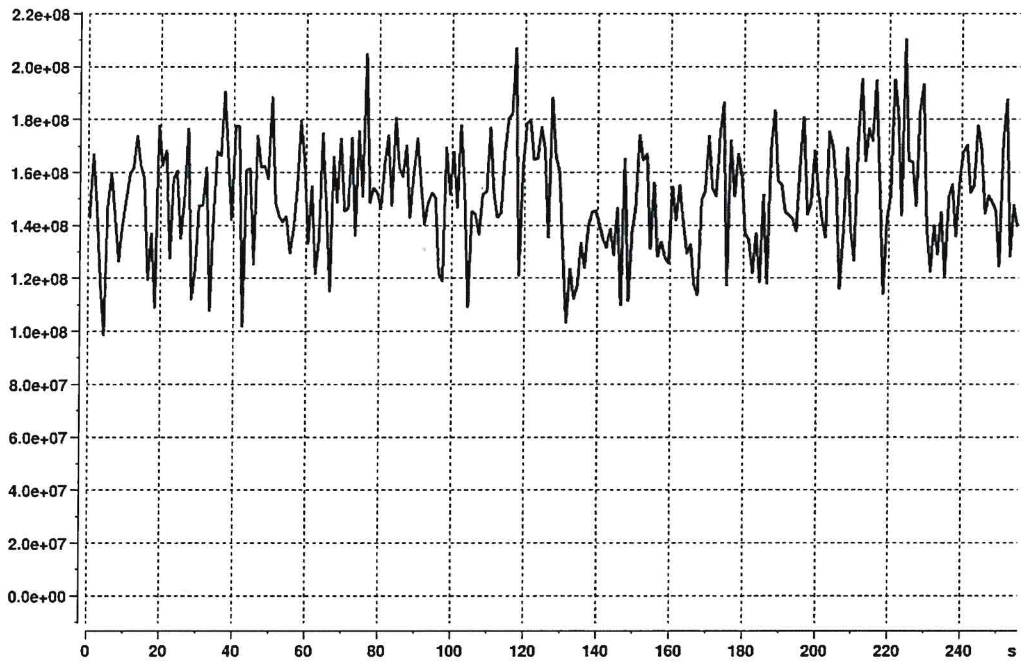

Figure 10: Expanded display of the column projection in Figure 9: signal amplitude is stable over time and the noise level stays constant.