**Probes: 4mm HXY probe H13694; 4mm HX H13892**

Rotor: HR with zirconia cap

Samples: $Pb(NO_3)_2$, KBr and $Sm_2Sn_2O_7$

**Variable temperature calibration**

I started with $Pb(NO_3)_2$ on 4mm HXY probe H13694 and I measured the change of $^{207}Pb$ chemical shift with temperature. A very linear behaviour has been obtained however the slope of 1.05ppm/K is quite different from the slopes reported in the literature (between 0.7 and 0.775 ppm/K).

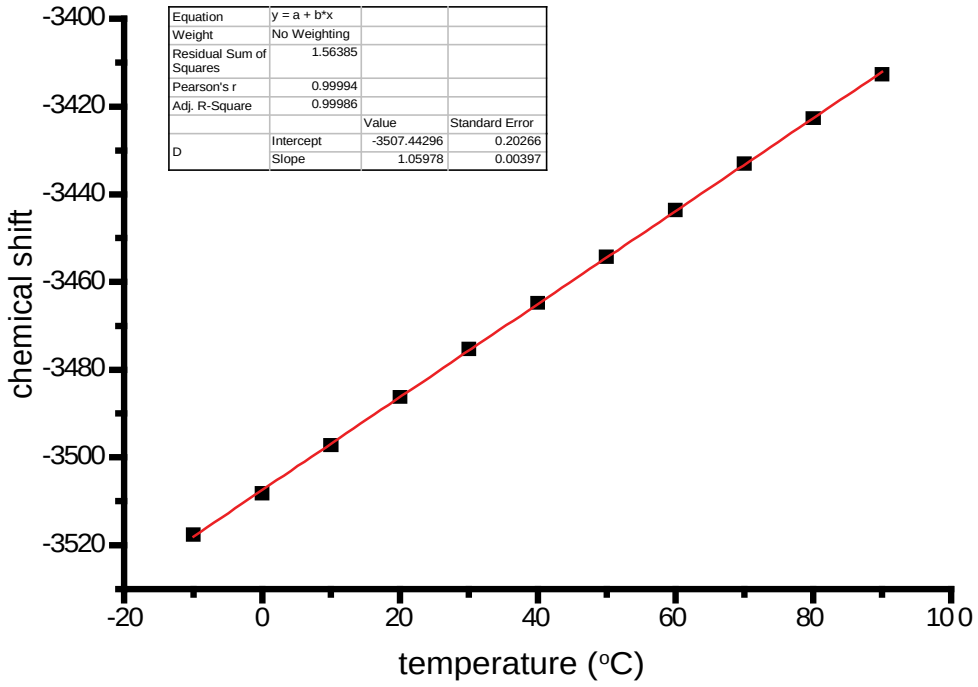



Figure 1 Chemical shift change of 207Pb $Pb(NO_3)_2$ with temperature

A calibration that brings the slope to a value between 0.7 and 0.775 means a temperature correction as large as 30°C (instead of 90 °C temperature indicated be Bruker the real temperature is 120°C). I was not ready to accept such a big difference between the shown temperature and the real temperature and for this reason on the 4mm HXY probe H13694 I measured the changes of chemical shift with temperature in KBr and $Sm_2Sn_2O_7$ and on the 4mm HX H13892 probe I measured $Pb(NO_3)_2$ and $Sm_2Sn_2O_7$. The data obtained are summarized in the figure 2 and 3 bellow. I also used a regular multimeter to check the temperature on the two 4mm probes and the results are shown in the table below.

| **4mm HXY probe H13694** | | **4mm HX H13892** | |
|---|---|---|---|
| Temperature indicated by Bruker | Temperature measured with a multimeter | Temperature indicated by Bruker | Temperature measured with a multimeter |
| 10.3 | 6.8 | 10.4 | 6 |
| 20.7 | 20.2 | 20.3 | 19.3 |
| 50 | 57 | 49.8 | 55.2 |
| 79.6 | 90 | 79.5 | 91 |

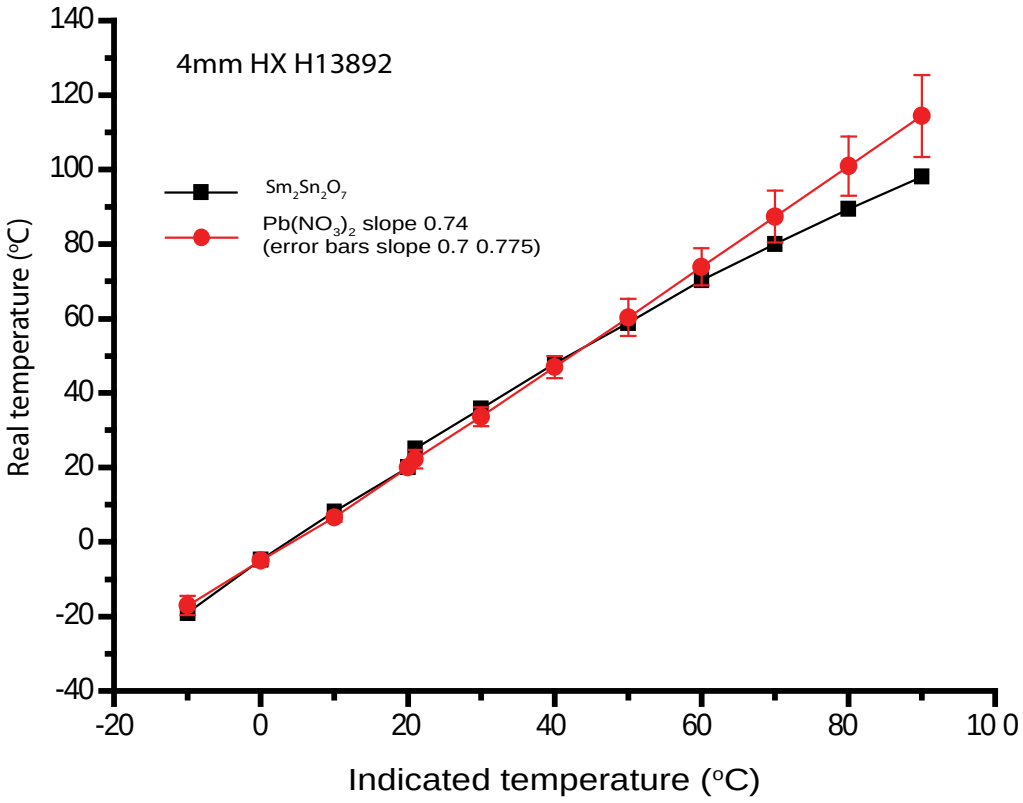



**Figure 2. Temperature calibration probe 4mm HX H13892**

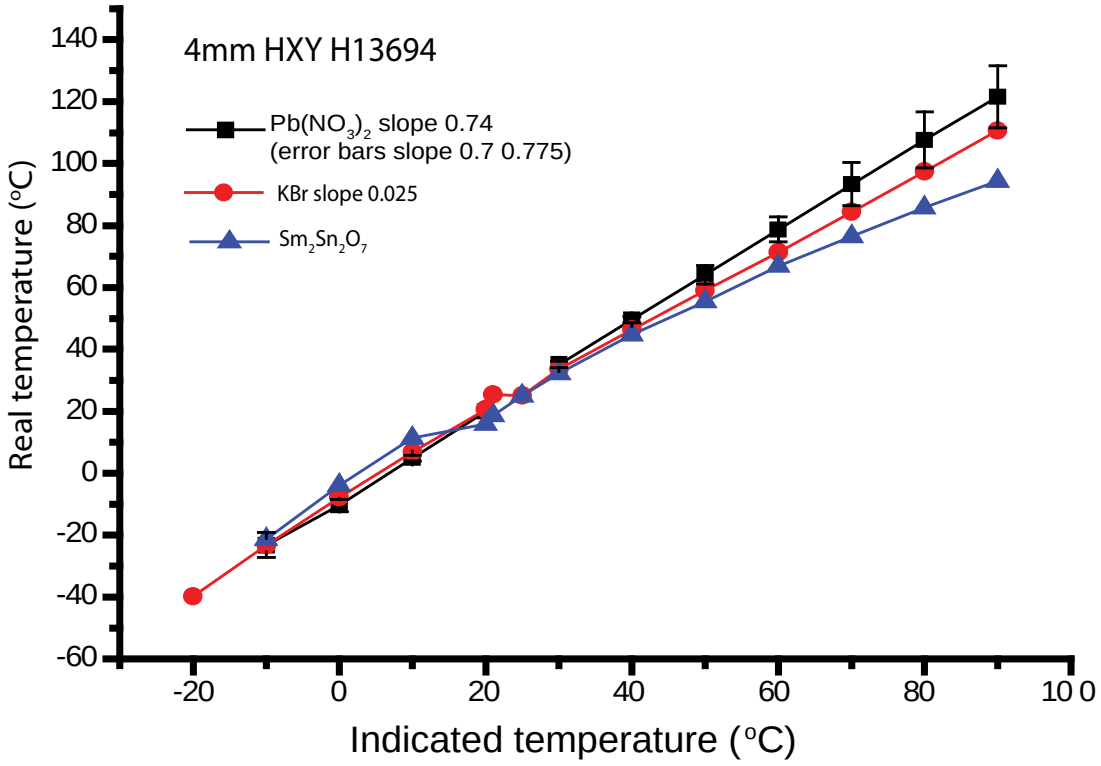


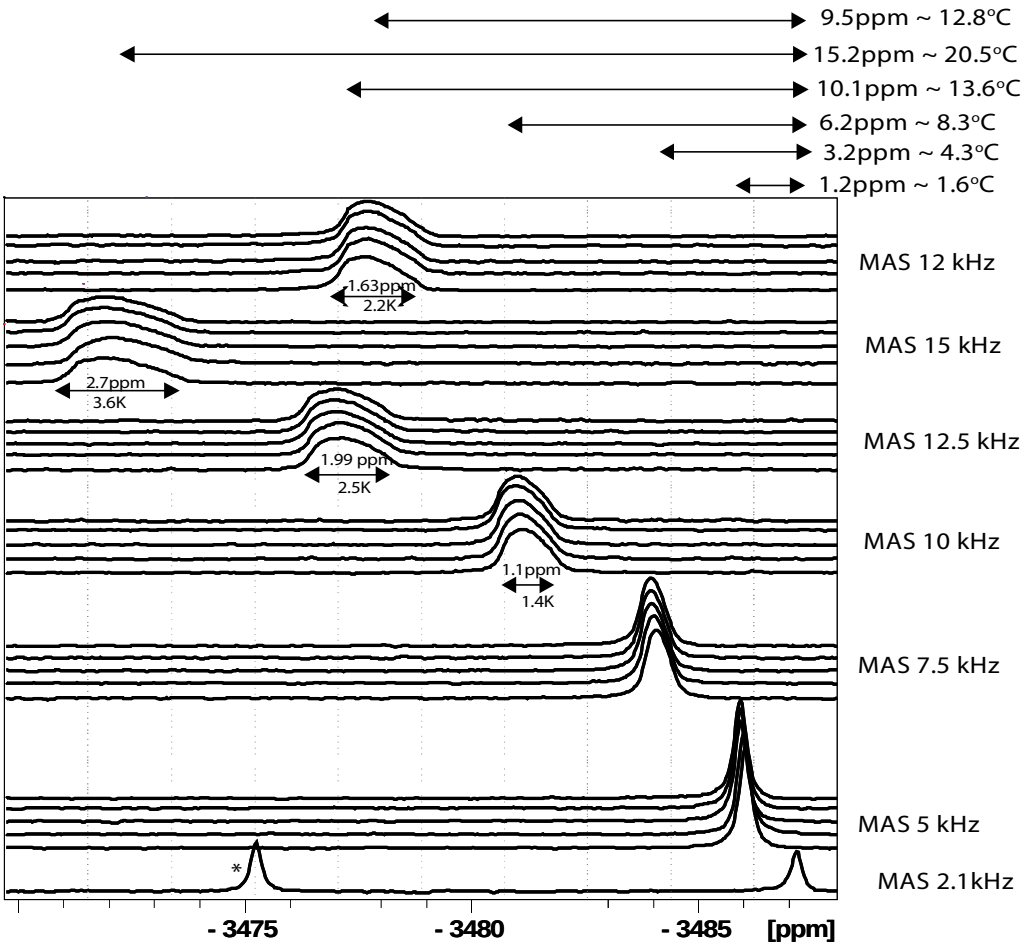

**Figure 3. Temperature calibration probe 4mm HXY H13694**

**Temperature change during spinning**

| MAS (kHz) | $^{207}Pb(NO_3)_2$ shift (ppm) | Change in temperature* ($^oC$) | Line width | Temperature gradient* ($^oC$) |
|---|---|---|---|---|
| 2.1 | | | Less than 0.7ppm | |
| 5 | 1.2 | 1.6 | Less than 0.7ppm | |
| 7.5 | 3.2 | 4.3 | Less than 0.7ppm | |
| 10 | 6.2 | 8.37 | 1.1 | 1.4 |
| 12.5 | 10.1 | 13.64 | 1.99 | 2.5 |
| 15 | 15.2 | 20.54 | 2.7 | 3.6 |
| 12 | 9.5 | 12.83 | 1.63 | 2.2 |

* Change in temperature is calculated as the change in chemical shift (ppm) compared with position at 2.1 kHz divided by 0.74

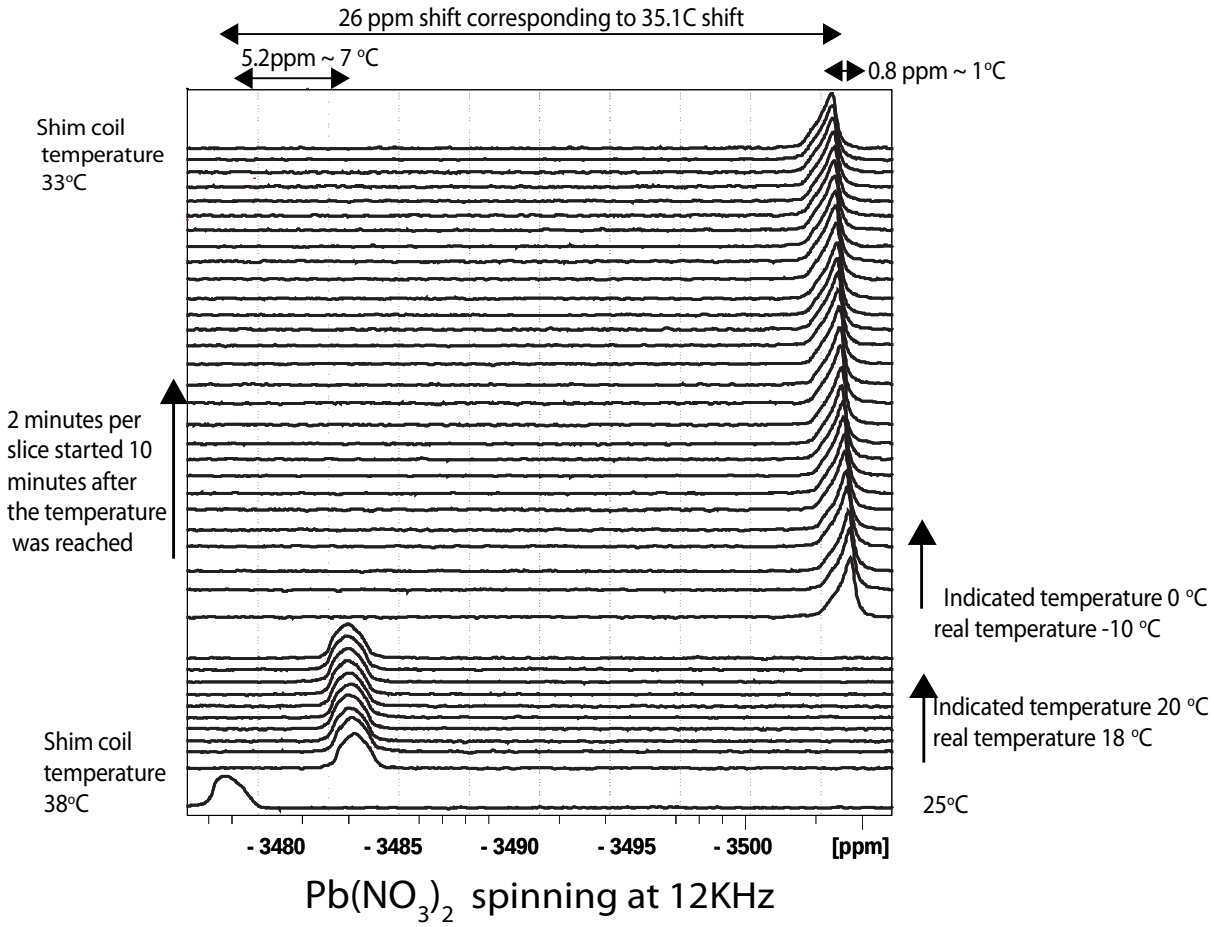
**Temperature gradient is calculated as linewidth(ppm)/0.74




* spinning side band

**Temperature stability**

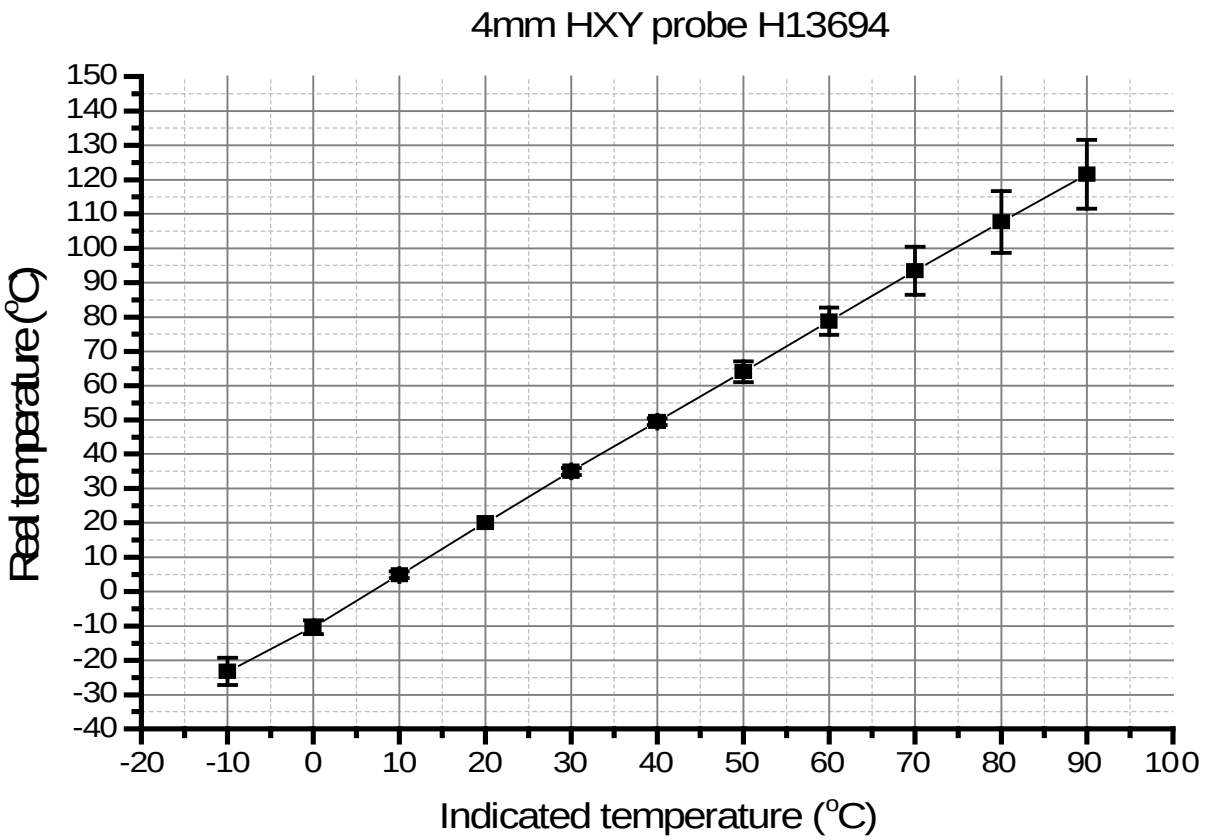
The metal holding the probe and the shim coils changes temperature gradually and therefore there will be a temperature drift that can last several hours. I started at 25°C with the shim temperature 38°C. I dropped the temperature to 0°C (indicated by Bruker) after 1 hour the shim coil temperature dropped to 33°C and the temperature of the sample changed by 1°C.

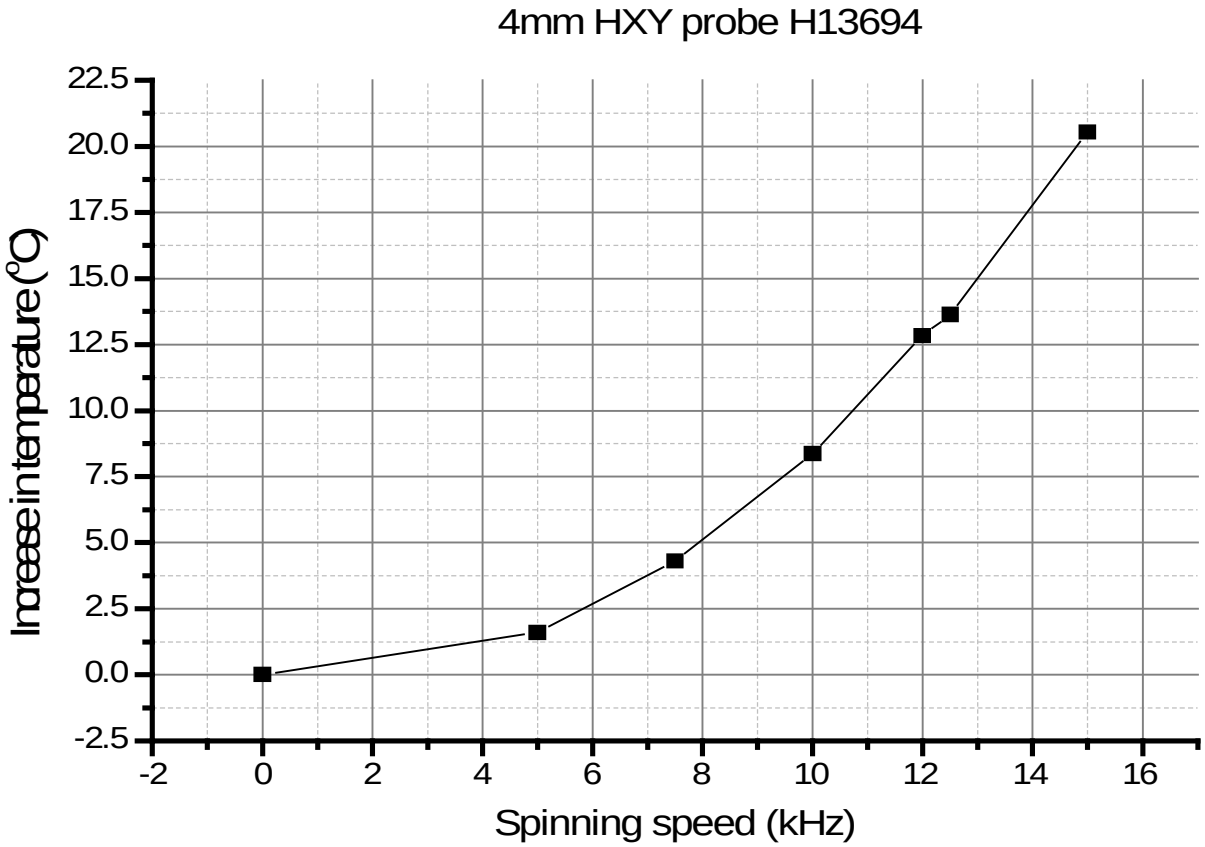



**Conclusions**

The users will receive two graphs shown below, and they will be informed about the long time required by the shim-stack temperature to reach thermal equilibrium with the VT gas flow. The users who perform VT measurements below -10°C will be asked to check the temperature of the shim-stack with 'coiltemp' command and stop their experiments if the shim coil temperature goes below -5°C.

Dinu Iuga
15/09/2011


4mm HXY probe H13694
Real temperature (°C)
Indicated temperature (°C)
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-20
-10
0
10
20
30
40
50
60
70
80
90
100


4mm HXY probe H13694
Increase in temperature (°C)
Spinning speed (kHz)
22.5
20.0
17.5
15.0
12.5
10.0
7.5
5.0
2.5
0.0
-2.5
-2
0
2
4
6
8
10
12
14
16