Procedure for Xe purity measurement with flow controller.

All values not automatically recorded in data files and every special occurrence need to be saved in an excel workbook (the measuana.xlsx contains template spreadsheets, and can be used as model). The RGA data must be saved in ASCII format, and snapshots of the RGA plots should be pasted in the excel spreadsheet of the measurements.

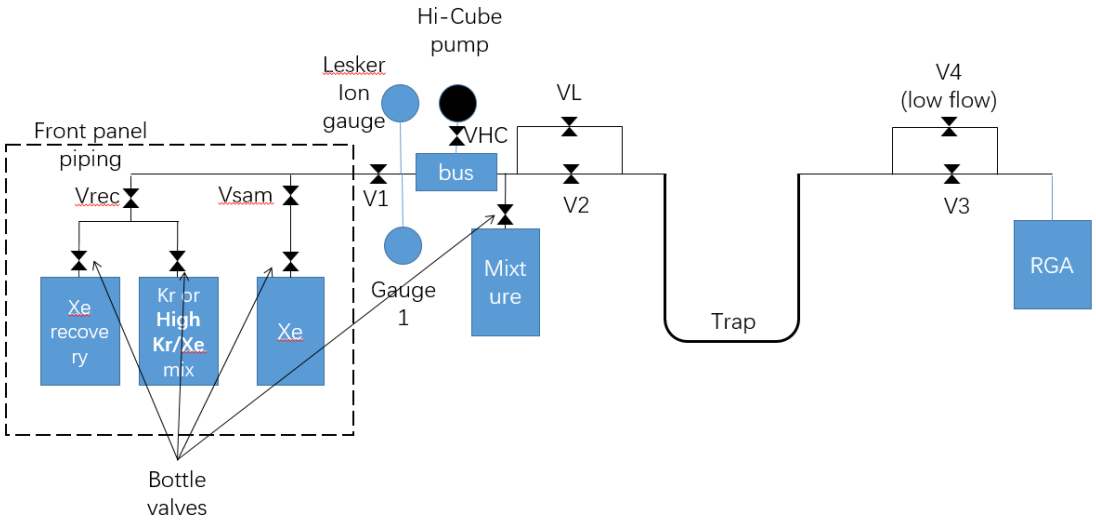

Fig. 1 Schema of the Xe purity system. V1 closes the connection between the front piping and the bus. Vsam is the sampling valve, ***not to be confused*** with the *sample bottle valve*., while Vrec is the valve shutting the path from either the recovery or the calibration bottle to the bus.

The valve nomenclature used below is displayed in Fig. 1. For the physical locations, you can refer to Fig.s 3-5. V1 is usually left open, unless it is necessary to completely isolate the main system from the front piping. If V1 is closed, you won't be able to inject gas into the system. Vsam is the sampling valve, ***not to be confused*** with the *sample bottle valve*. Fig.s 1 and 3 should make this clear. Valves on the bottles do not have specific names.

1. Initially the system should be under high vacuum, so check the various gauge pressures. The front panel has several displays, shown in the left Fig. 2. The normal pressures displayed in the vacuum state are clearly visible. If any of these pressures is significantly higher, find out why. If a filled bottle has its valve not fully closed, you probably lost a large fraction of its content. Other 2 pressures to check are behind the front panel, on the Lesker ion gauge (see Fig. 2, right side), and on the red HiCube pumping station. If the station's display shows something else (e.g., the rotation speed), browse through the parameters until you find the pressure. Note that the unit on the pump display is hPa, not Pa. The Lesker gauge and the pump should read similar pressures (within a factor of 2, after conversion to the same unit). ***Most important pressures***: ion gauge of RGA side $\leq$ 1e-5 Pa, pump (bus side) and Lesker ion gauge $\leq$ 2e-4 Pa.
2. Check the configuration: the valve of the HiCube pump (VHC in the schema of Fig. 1 and in Fig. 4) and valves Vsam, Vrec, V1, V3 and V4 should be open, while V2 and VL (leak vale)

should be closed, and the RGA off. The valves of any bottle that shouldn't be empty should be closed. If not, you have likely lost the bottle's content. *Note*: if Vsam was not open, you likely have air between the sample bottle valve and Vsam (in the elbow at the right of Vsam, see Fig. 3). In this case:

a) Turn off the lesker, the RGA ion gauge and the HiCube's turbo component (wait that the turbo's rotation speed is zero)
b) Slowly open Vsam
c) Turn the HiCube's turbo on and wait that the Pirani, Gauge 1, Gauge 2 and the HiCube return to the normal readings. Then turn the Lesker and the RGA ion gauge on and check that everything is back to normal.

Fig. 2: Left: main displays of the front panel, with their normal reading when the system is under a good vacuum. Gauge 1 is the high P (range up to 10 bar) gauge of the bus, Gauge 2 is the front piping gauge with same range. Both displays are in bar. The controller in the top displays on the left the pressure (in Pa) of the Pirani gauge of the bus (range 0.1-1e5 Pa), which in these conditions is normally below range, and on the right the pressure (in Pa) of the ion gauge on the RGA side (range to 10 Pa. ***Never*** turn this gauge on if you think the pressure could be above 5-10 Pa). Right: Lesker ion gauge, placed in the back of the front panel. It measures the bus pressure (in Pa) when it is under high vacuum. Its range is up to 6.7 Pa. ***Never*** turn this gauge on if you think the pressure could be above 3-6.5 Pa (yes, the Lesker gauge takes less pressure than the RGA side ion gauge).

3. Close or make sure that the valves Vsam, Vrec, V2, *V3*, and VL are closed. Also make sure that the recovery bottle valve (not shown) is closed. V4 should remain fully open. Turn the ***Lesker*** gauge ***off*** and close its valve.
4. Check the vacuum with the RGA. When the RGA filament is on, the ion gauge must be off. Make 2 measurements, one spectral (for the residual gas composition) and one P vs time of the masses of interest (for stability in time. After a night in this configuration, the state should be steady). Save each vacuum (blank) measurement, ***turn the RGA off*** and its ion

gauge **on**.

5. Cool down the trap by submersion in LN. This means:
   a) Place the lifting pad below the trap pipe
   b) Place the green dewar on the pad, tilting it so that the bottom of the trap gets into it
   c) Fill the green dewar with LN to a reasonable level
   d) Use the lifting pad to lift the dewar until the trap is well submerged in LN
   e) If the LN level is too low, add some to the dewar
6. ***Close VHC***. If you don't, your gas sample will be *pumped out* by the HiCube pump.
7. *Configuration with flow controller (MFC) between sample bottle and Vsam.* ***Do not use sample bottles with pressure above 9.5 bar***. Make sure that the MFC valve is ***CLOSED***, then open (not fully) the sample bottle valve with Vsam ***CLOSED***, check that the pressures of gauge 1 and RGA ion gauge do not change, and the reading of the MFC is 0. If not, ***promptly*** close Vsam, then recheck all valves (most likely V2 or VL is not well closed *and* one of the front bottles/valves is not properly closed, but better make sure every valve is as should. The worst case scenario is if the MFC valve is not fully closed. In this case, make sure the MFC is not broken or defective, and learn to operate it more properly). And, of course, restore the initial vacuum conditions, then restart from step 1. Advice: to avoid having to restart from scratch, feel free to check the valves and the MFC before doing this step. Same advice for the next step.
8. Make sure that the MFC can, when closed, stop the flow completely. This means with the MFC closed, you can gradually open Vsam until fully open ***without an increase*** of the RGA's ion gauge reading. Keeping the MFC closed, ***and with the trap already cold***, gradually open Vsam and the leak valve. Ideally, you should be able to fully open Vsam and VL without problems, but if the RGA's ion gauge reading increases, *stop immediately, promptly closing Vsam*. Then retry, until you find the configuration with Vsam and VL open as much as possible without an increase of the RGA's ion gauge reading.
9. Gradually and *slowly* open the MFC valve. When the RGA ion gauge reads 3 to 5 E -5, make a 1 minute pause to let some Xe ice to form, then resume opening the MFC, still *gradually* and slowly, until you reach the desired flow (typically between 0.5 and 1 slpm), ***watching the RGA pressure with the ion gauge***, and keeping an eye also on the bus pressure. If the RGA pressure ***exceeds 1 E -4 Pa you can't use the RGA in the electron multiplier mode***, if it exceeds 1e-2 Pa ***you can't use the RGA at all***[1] of (it is prudent to not use the RGA above 5 mPa anyway). The bus pressure is less critical, but it shouldn't grow above a few bar.
10. Once you achieved a stable inflow to the trap and a stable pressure at the RGA, record in the excel spreadsheet the RGA ion gauge pressure, and *turn it off.* Turn the RGA on, and start a PvsT measurement to check the gas composition stability (PvsT template). Save the RGA data, then open the massSpecTemplate.rga file and connect the RGA. The filament should have remained on.
11. Start a 3 pass mass scan, then save the mass spectrum.
12. Change back to PvsT (close the mass spectrum file and open the PvsT template), and measure 1-2 more minutes before recording G1 and closing V4, VL, and the MFC. Keep

[1] The MKS RGA datasheet states a max operating P of 1e-4 Torr, the same of the SRS for Faraday cup mode, without specifying anything for the EM mode. So, conservatively assume that the max operating P for EM mode is also 1e-6 Torr, as for the SRS.

measuring, to record the drop to normal vacuum.

13. Turn ***off*** the RGA's filament, save the data and turn the RGA ***off***.
14. Recover the Xe from the cold trap, as follows
    a) At room temperature and with Vsam ***and Vrec*** closed ***but MFC open***, open the recovery bottle's valve so gauge 2 can measure its pressure. If MFC is closed the pressure difference between the sample and the recovery bottle may damage the MFC. If the recovery bottle pressure is high, you need to empty it to a "Xe dump" bottle before proceeding.
    b) With Vrec still closed, cool the recovery bottle to 77 K.
    c) When the recovery bottle pressure (gauge 2) falls below the bus pressure (gauge 1) open Vrec.
    d) When the bus pressure becomes low, lower the trap dewar, so that the trap starts warming up, and open V2, so that the bus pressure (gauge 1) equals the trap pressure.
    e) Warm up the trap with a heat gun. This will keep the trap pressure above the recovery pressure until there's significant Xe in the trap.
    f) When the recovery is complete, close the recovery bottle valve and V2 (make sure VL is also closed). Restore the valve configuration of step 1 (Vrec, Vsam, V3 and V4 are open, while V2 and VL are closed) and ***open VHC***, so the HiCube can pump down the bus and front piping. Make sure every bottle you don't want empty has its valve closed. When the pressure read by the HiCube pump is well below 1 Pa, open the Lesker gauge valve.
    g) Plug in the heating tapes, except that of the RGAs (these should be baked only seldom)

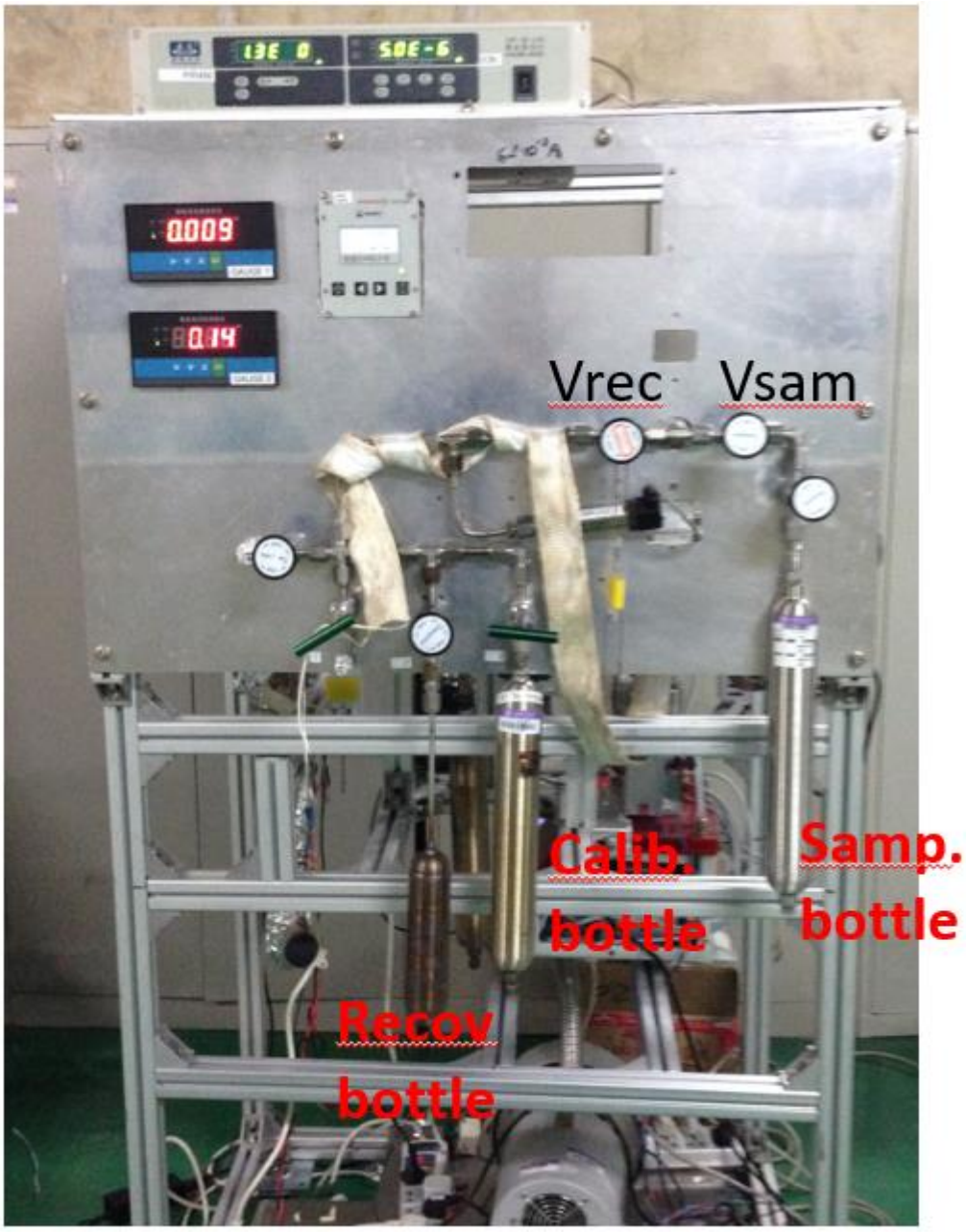

Fig. 3: Vrec and Vsam on the front panel

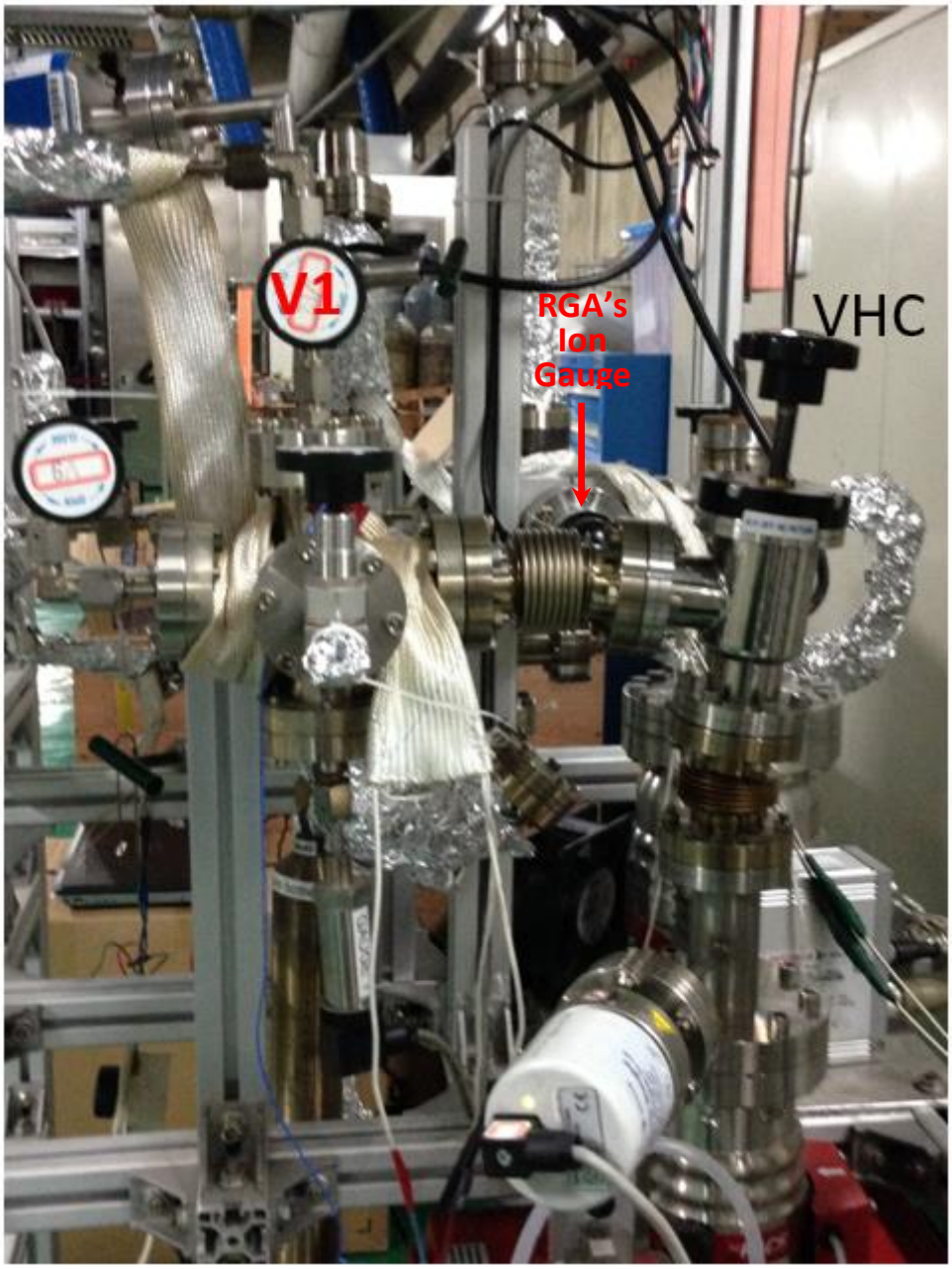

Fig. 4: V1 and VHC on the left side

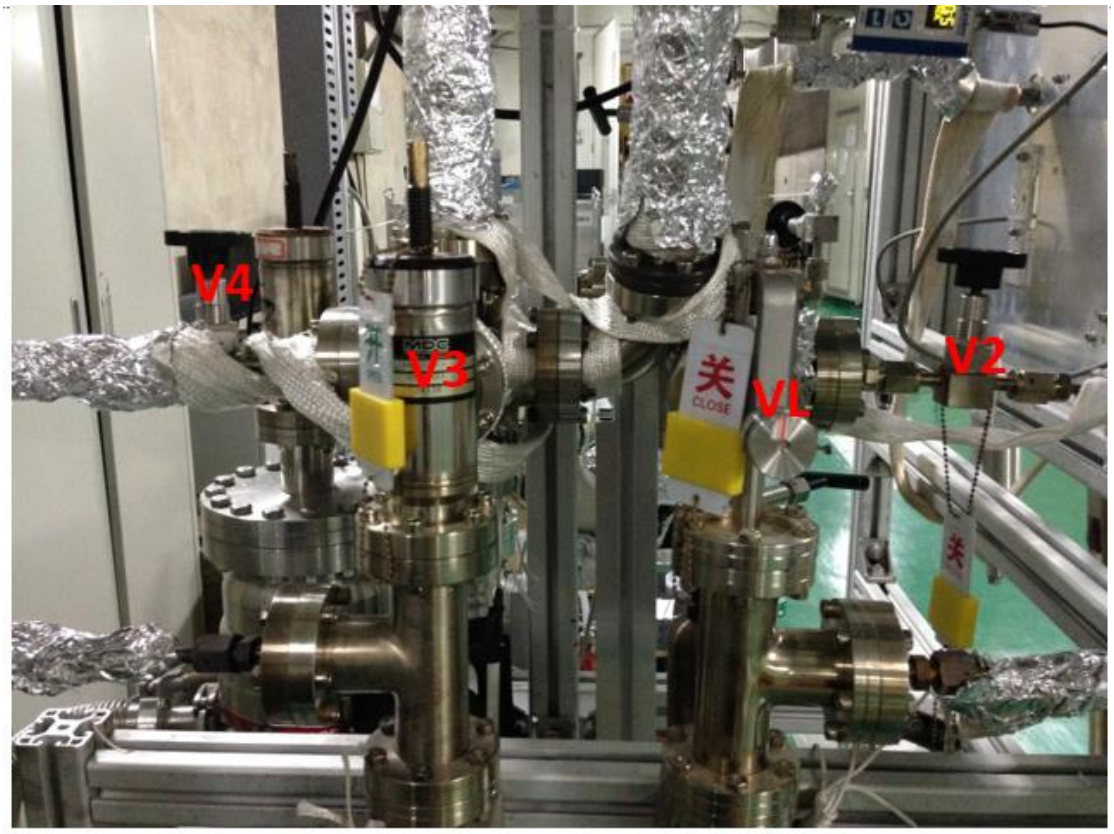

Fig. 5: VL, V2, V3, V4

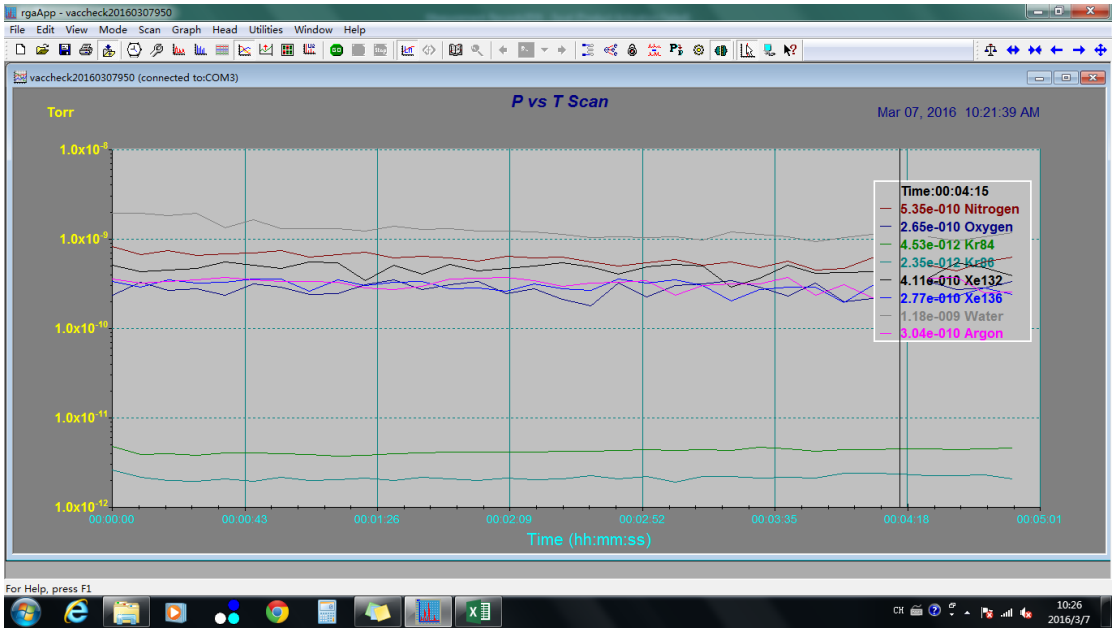

Fig 6. Example of vacuum & RGA stability check

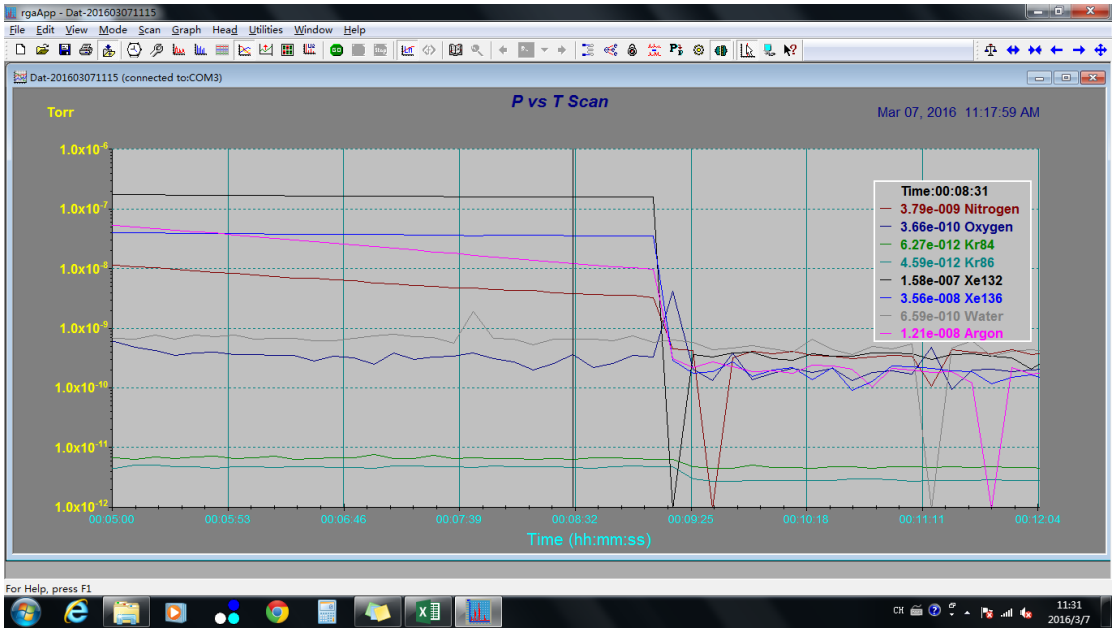

Fig. 7: Data recorded during a measurement. The acquisition continues after closing V4 (no more flow to the RGA), to check that the vacuum is restored, so presumably the background remained the same during the measurement.