



WEEK 3.1 REPORT – LCU AND PTR-MS VERIFICATION AND ASSOCIATED GRAPHS

February 17TH, 2017

Summary

The mobile lab was used at Columbia Basin Analytical Laboratory (CBAL) on February 17th between 7:00 AM and 1:00 PM to conduct a series of preliminary experiments with the Ionicon Liquid Calibration Unit (LCU). The goal of these experiments were to create a streamlined series of experiments to be performed on February 20th to verify LCU operation and provide technical training for the analyst-in-training.

During this time period the LCU was used to dilute the 200 ppb sensitivity check standard to 20 parts per billion by volume (ppbv) which was measured by the Ionicon 4000 proton transfer reaction mass spectrometer (PTR-MS), as per normal operations. The 200 ppbv standard was also manually diluted to 20 ppb and then directly measured by the PTR-MS, bypassing the LCU.

February 20TH, 2017

Summary

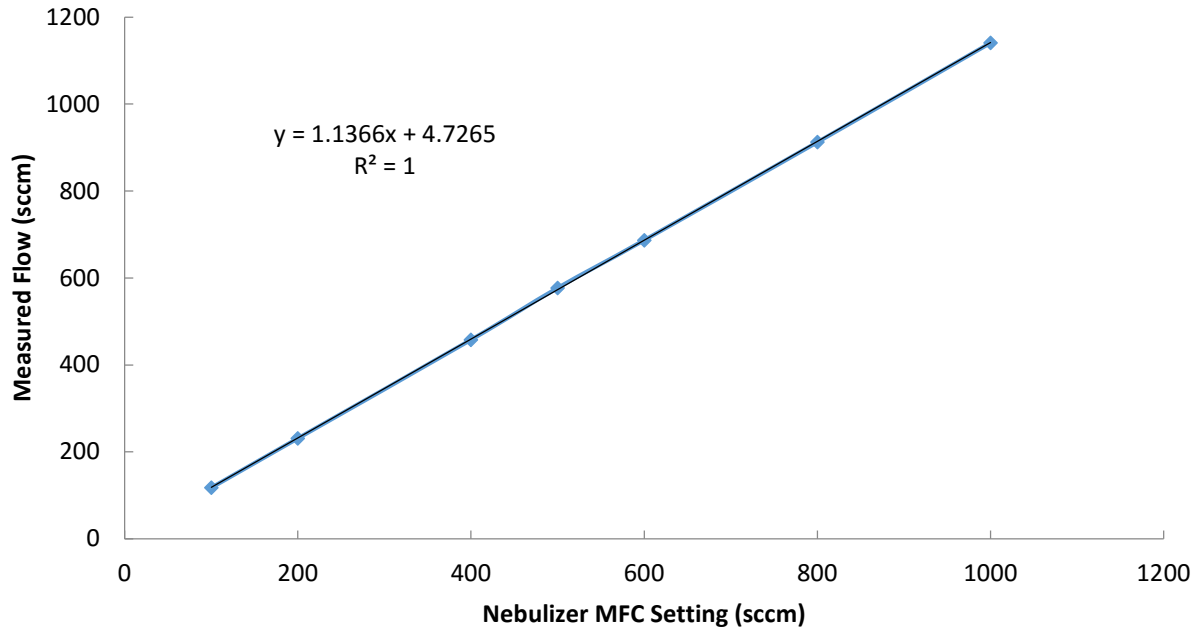
The mobile lab was used on at CBAL on February 20th between 7:00 AM and 4:30 PM to verify LCU operation and provide technical training for the analyst-in-training.

During this time period the flow of the standard and zero air flow control units were measured separately over expected operating ranges. Gas flow from the control units were measured using a Mesa Labs Defender 520 digital flow meter. Mixing ratios of both flow controllers were also tested at a total flow rate of 300, 500, and 1000 standard cubic centimeters per minute (sccm) to validate linearity over a range used for dilutions of the standard during a sensitivity check. The digital flow meter used in these experiments is due to be recalibrated in April. Once the flow meter is returned these experiments will be repeated to determine if the deviation from program settings (see LCU 3.1.1a and LCU 3.1.1b) is from the LCU or is a measurement error from drift on the meter.

February 20th Graphs

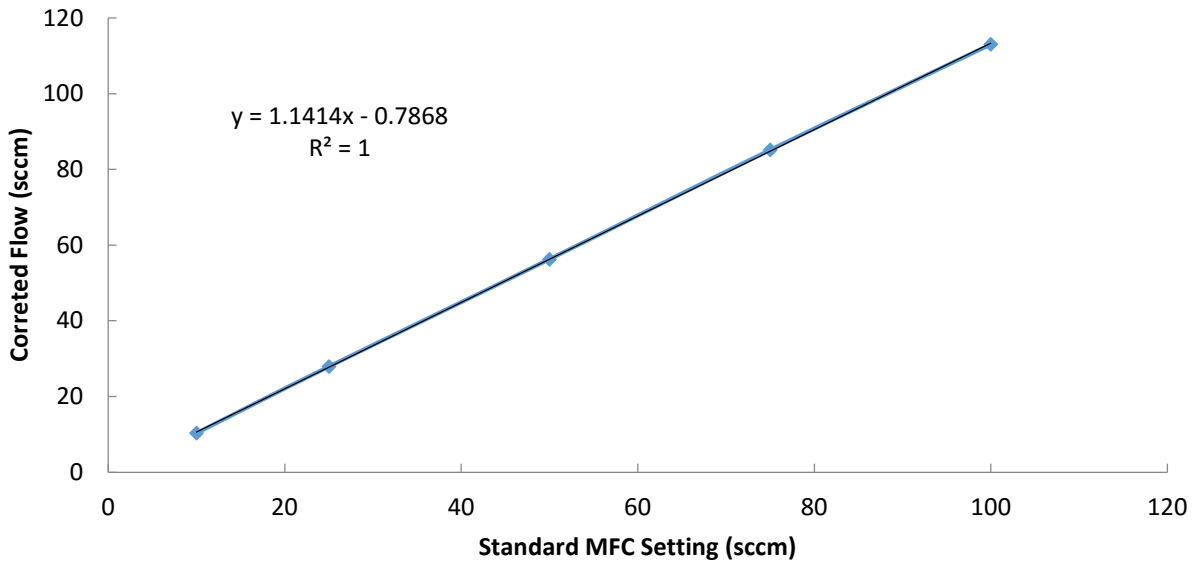
LCU 3.1.1a

Graph LCU 3.1.1a displays the measured flow of the zero air flow controller vs. the theoretical air flow. The behavior of the flow controller is linear as indicated by the R^2 value. The slope indicates percent deviation from the program settings, which is 14% higher than the theoretical value. Subsequent experiments indicate that this does not affect data quality, see LCU 3.1.1b. During sensitivity checks the zero air flow setting is 975 sccm which is within the range of this graph.



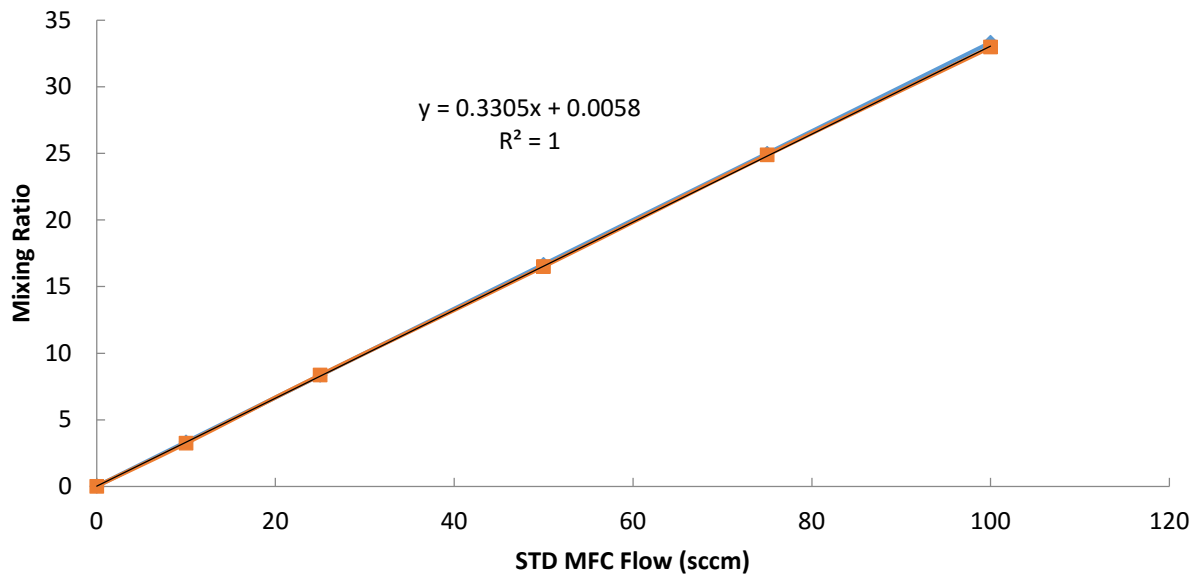
LCU 3.1.1b

Graph LCU 3.1.1b displays the measured flow of the standard flow controller vs. the theoretical air flow. The behavior of the flow controller is linear as indicated by the R^2 value. The slope indicates percent deviation from the program settings, which is 14% higher than the theoretical value. During sensitivity checks the zero air flow setting is 25 sccm which is within the range of this graph. The deviation in the zero air and standard flow controllers is the same (14%), indicating that dilutions made with the two flow controllers will be linear and that the LCU will deliver the expected standard concentration to the PTR-MS. This is verified with the following experiments, see LCU 3.1.1c, LCU 3.1.1d, and LCU 3.1.2e.



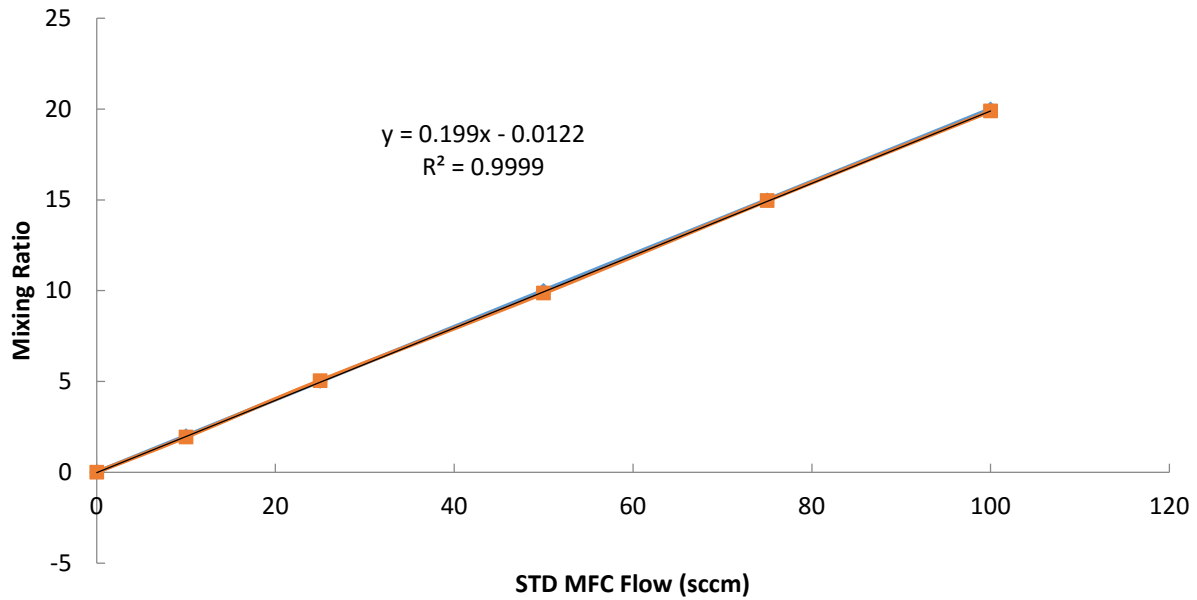
LCU 3.1.1c

Graph LCU 3.1.1c displays the theoretical and measured mixing ratios of zero air and standard at a total flow rate of 300 sccm. The linear regression within the graph is indicative of the measured values. Theoretical values are plotted in blue and measured values in orange. The behavior of the flow controllers is linear as indicated by the R^2 value. This data indicates that at 300 sccm total flow rate the flow controllers from the LCU are mixing the zero air and standard gas at the appropriate ratio for a dilution.



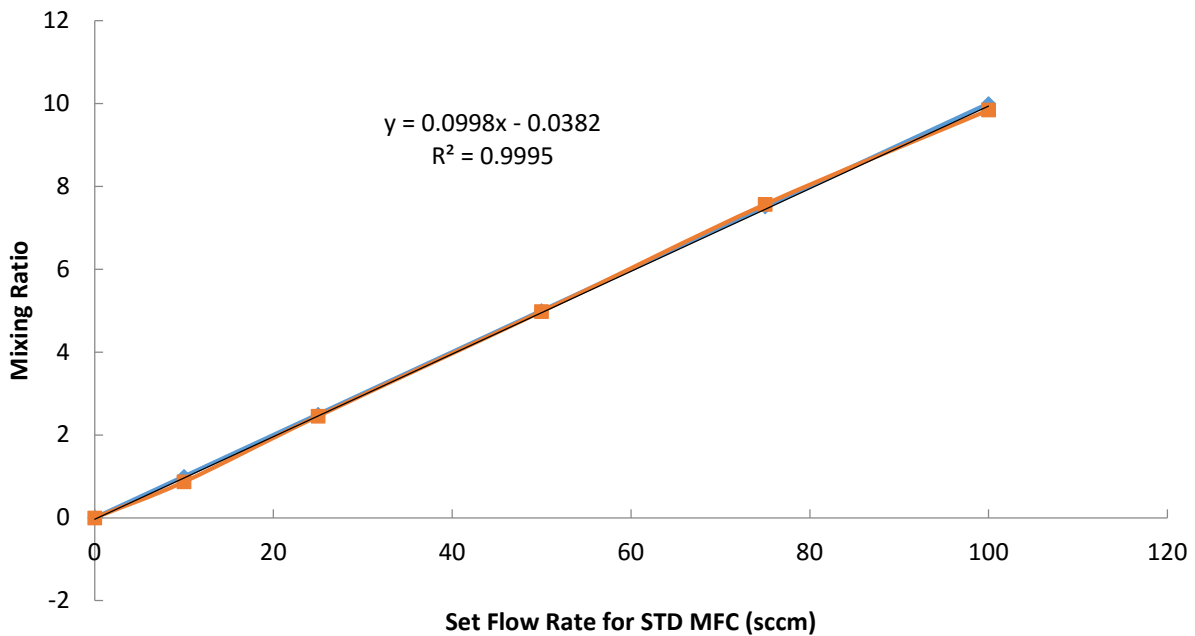
LCU 3.1.1d

Graph LCU 3.1.1d displays the theoretical and measured mixing ratios of zero air and standard at a total flow rate of 500 sccm. The linear regression within the graph is indicative of the measured values. Theoretical values are plotted in blue and measured values in orange. The behavior of the flow controllers is linear as indicated by the R^2 value. This data indicates that at 500 sccm total flow rate the flow controllers from the LCU are mixing the zero air and standard gas at the appropriate ratio for a dilution.



LCU 3.1.1e

Graph LCU 3.1.1e displays the theoretical and measured mixing ratios of zero air and standard at a total flow rate of 1000 sccm. The linear regression within the graph is indicative of the measured values. Theoretical values are plotted in blue and measured values in orange. The behavior of the flow controllers is linear as indicated by the R^2 value. This data indicates that at 1000 sccm total flow rate the flow controllers from the LCU are mixing the zero air and standard gas at the appropriate ratio for a dilution. Sensitivity checks are performed with a ratio of 975 sccm zero air to 25 sccm standard gas using the LCU, which falls within the range of this graph.



February 21ST, 2017

Summary

The mobile lab was used on at CBAL on February 21st between 7:00 AM and 4:30 PM to continue studying LCU operation and provide technical training for the analyst. The ion source was removed and replaced with a clean source. The Agilent 6890 gas chromatograph and Agilent 5973 mass spectrometer (GC-MS) was also turned on to begin analyst training on the instrument.

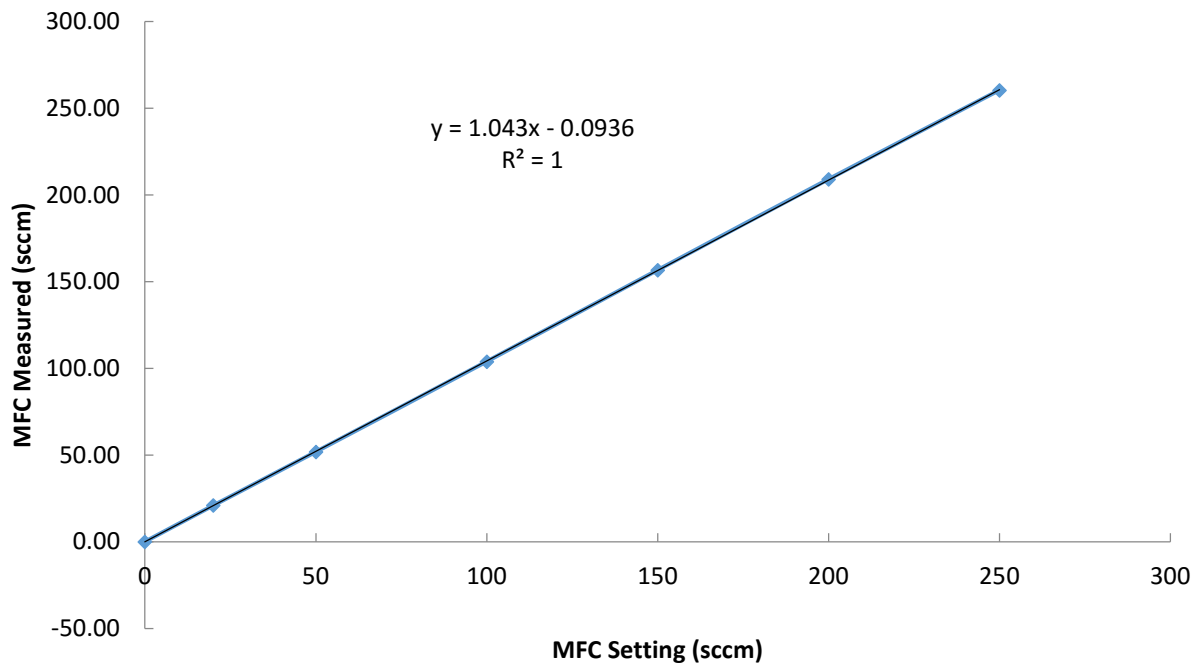
In addition, the PTR-MS inlet flow controller flow rate was manipulated in several experiments to verify PTR-MS operation. The experiments performed were at 2 different drift tube pressures from 0 to 250 sccm and at one drift tube pressure up to 500 sccm. Data from the experiments indicate that the flow is linear up to at least flow rates of 500 sccm, see LCU 3.1.2c. A flow of 200 sccm is the current operating condition and falls within this acceptable range.

Data from the experiment also indicated that the rate could be lowered to 100 sccm. This lower flow rate would reduce strain on the rough pump and also reduce gas use. However, higher flow rates decrease compound transit time in the tubing leading up to the PTR-MS which increases the ability to measure bolus events. Thus while conducting field monitoring 200 sccm will still be used.

February 21st Graphs

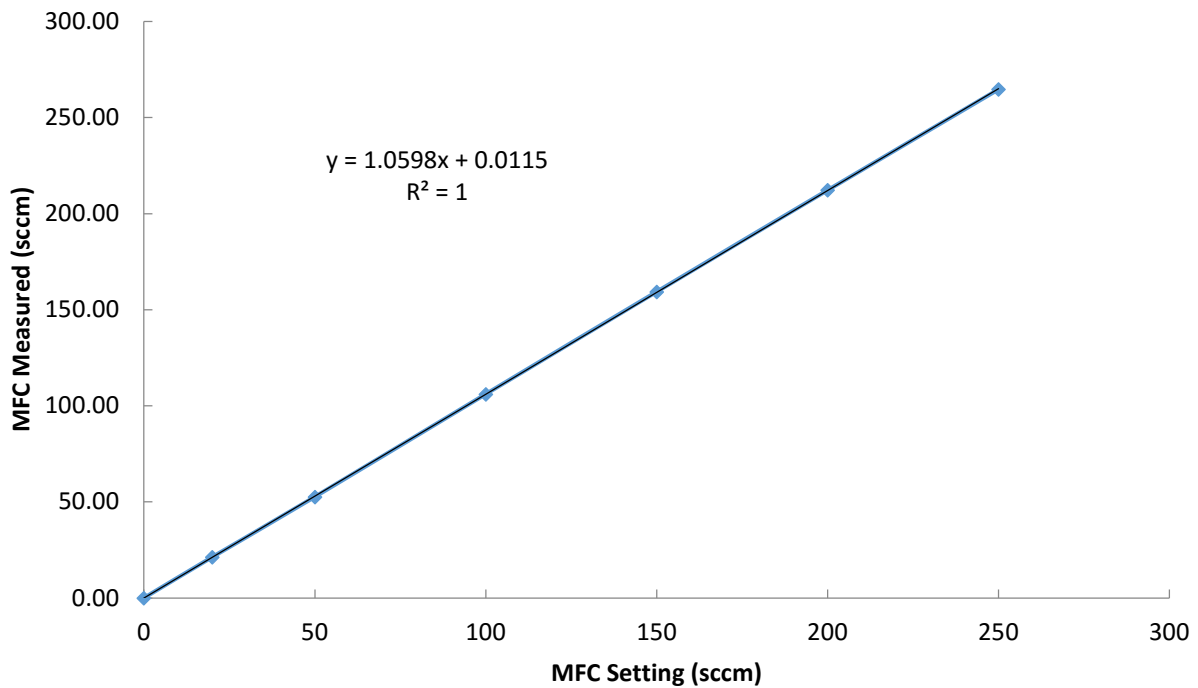
LCU 3.1.2a

Graph LCU 3.1.2a displays the measured flow rate of the PTR-MS inlet flow controller vs. theoretical values based on software settings for a drift tube pressure of 2.8 mbar. The behavior of the flow controller is linear as indicated by the R^2 value. The slope indicates percent deviation from the program settings. Note that deviation for this flow rate does not affect data quality.



LCU 3.1.2b

Graph LCU 3.1.2b displays the measured flow rate of the PTR-MS inlet flow controller vs. theoretical values based on software settings for a drift tube pressure of 2.2 mbar. The behavior of the flow controller is linear as indicated by the R^2 value. The slope indicates percent deviation from the program settings. Note that deviation for this flow rate does not affect data quality.



LCU 3.1.2c

Graph LCU 3.1.2c displays the measured flow rate of the PTR-MS inlet flow controller vs. theoretical values based on software settings for a drift tube pressure of 2.8 mbar. The behavior of the flow controller is linear as indicated by the R^2 value. The slope indicates percent deviation from the program settings. Note that deviation for this flow rate does not affect data quality. Any deviation from linearity at the high flow rates would suggest that the system rough pump had reached its pumping capacity. The data is linear which means that the PTR-MS system is capable of handling high inlet flow rates (up to 500 sccm) for rapid flushing of the sample lines with no degradation of overall instrument performance. The intersection of the Y-axis is the 'normal' sample flow to the PTR-MS through the 1.2 meter sampling line without added flow from the FC-inlet mass flow controller (MFC) (~74 sccm).

