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RJ LEE GROUP
DELIVERING SCIENTIFIC RESOLUTION

WEEK 3.2 REPORT – LCU AND PTR-MS VERIFICATION

February 22nd, 2017

Summary

The mobile lab was used at Columbia Basin Analytical Laboratories (CBAL) on February 22nd between 7:00 AM and 4:00 PM to conduct experiments with the FC Inlet flow rate on the IONICON Analytik Proton Transfer Reaction Time-of-Flight Mass Spectrometer 4000 (PTR-MS). The goal was to understand the effect of the FC Inlet flow on the signal of standard to verify the effectiveness of PTR-MS standard operating parameters.

During this time period the Ionicon Liquid Calibration Unit (LCU) was used to dilute the 2 parts per million by volume (ppmv) sensitivity check standard to 20 parts per billion by volume (ppbv) which was measured by the PTR-MS, as per normal operations. The concentration of benzene was then monitored at 0, 100, and 200 standard cubic centimeters per minute (sccm) FC Inlet flow rate. This experiment was repeated on February 27th due to analyst concern that the signal had not fully stabilized.

The 200 ppbv standard was also manually diluted to 20 ppbv and then directly measured by the PTR-MS, bypassing the LCU. The same experiment was performed with the PTR-MS inlet tubing at two different depths within the tubing leading from the 20 ppbv standard canister. It was found that tube depth did not affect signal stability or concentration. Lower FC Inlet flow rates, at a constant delivery flow from the air canister, resulted in higher concentration detected by the PTR-MS. This suggests that higher rates effectively dilute the standard by pulling in ambient air in addition to standard. This would only affect manual measurement of standard using an independent mass flow controller.

February 23rd, 2017

Summary

The mobile lab was used on at CBAL on February 23rd between 3:00 PM and 4:00 PM to conduct preliminary experiments for the verification of the LCU. These experiments were used to direct experiments on February 24th.

February 24th, 2017

Summary

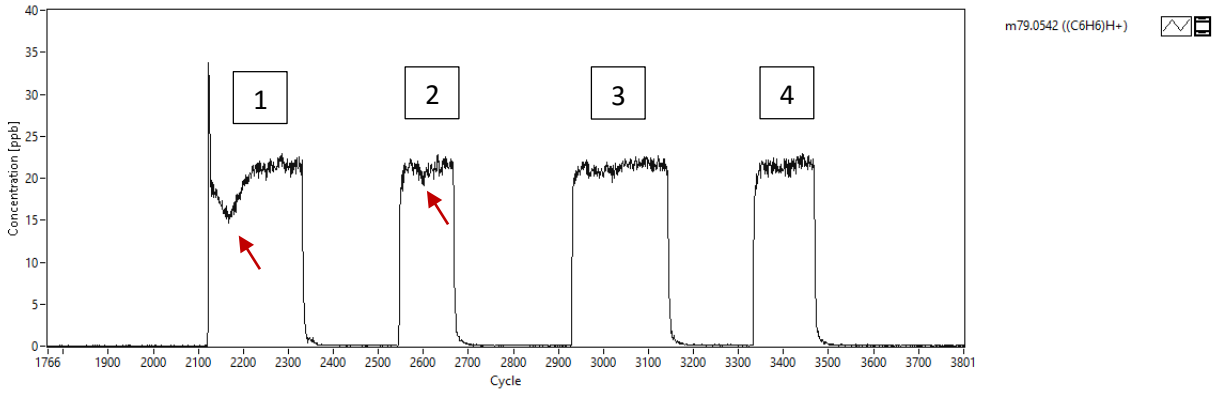
The mobile lab was used at CBAL on February 24th between 7:00 AM and 2:30 PM to perform experiments to verify LCU operation. In particular, an experiment was performed to test the effect of the evaporation chamber temperature concentration of standard produced by the LCU.

During this time period the LCU was used to dilute the 2 ppmv sensitivity check standard to 20 ppbv which was measured by the PTR-MS, as per normal operations. Benzene concentration was monitored when the evaporation chamber temperature was set to 30, 60, 80, and 100 °C. For at least 5 minutes between temperature changes zero air was drawn through the system to limit residual effects. Signal behavior was recorded and concentration was averaged. It was found that temperature did not alter the concentration of the standard, but it did alter the behavior of the signal. Visually, the signal is more erratic at lower temperatures and more stable at higher temperatures. The current standard operating condition of the evaporation chamber is (and has always been) 80 °C. Also, the LCU is only used during sensitivity checks, not during ambient air measurements. Thus, these findings do not affect data quality.

This behavior may indicate that at low temperatures condensation occurs, initially lowering the amount of standard in the gas phase.

Figure LCU 3.2.3a

Figure LCU 3.2.3a displays the PTR-MS signal for benzene in this experiment. PTR-MS Viewer 3 software was used to subtract the background and to produce this image. Signal 1 indicates evaporation chamber at 30 °C. Signal 2 indicates 60 °C, 3 indicates 80 °C, and 4 indicates 100 °C. Note the non-steady initial behavior of signal 1 compared to signals 3 and 4.



February 27th, 2017

Summary

The mobile lab was used at CBAL on February 27th from 8:00 AM to 3:00 PM to continue verification of the LCU and PTR-MS. The FC-Inlet experiment from February 22nd was repeated specifically for standard diluted by the LCU. This experiment was repeated because further examination of the data indicated that sufficient time for stabilization may not have occurred and results were the opposite of expectations.

The experiment was repeated at 8:30 AM and at 9:30 AM. At 8:30 AM data from the experiment indicated that the PTR-MS and LCU had not sufficiently warmed-up and stabilized. At 9:30 AM data from the experiment indicated that varying FC Inlet flow rates from 200 sccm to 100 sccm and 0 sccm had no effect on benzene concentration, provided that the PTR-MS inlet line placement in the mass flow controller exit line is deep enough to prevent back diffusion of ambient air. Additionally, the PTR-MS signal appeared to stabilize. Note that field measurements are conducted well-after the warm-up period of this experiment.

An additional experiment was conducted to test the separation of the peaks of several compounds of interest. A standard (CBAL-413-74B) was produced that contained methanol, furan, benzene, isoprene, methyl acetate, N-nitrosodi-N-propylamine, N-nitrosomethylethylamine, and N-nitrosodimethylamine (NDMA). The LCU was used to dilute this standard to an appropriate concentration and the signal was monitored on the PTR-MS. Isoprene and furan were sufficiently resolved at m/z=69 which provides high confidence in their identification in the data sets when present individually or as a mixture. This is augmented with the high stability of the mass scale from the PTR-TOF-4000 instrument. The other peaks of interest at m/z=75 for protonated methyl acetate and NDMA were not resolved. This experiment will further inform future experiments.

February 27th, Figures

Figure PTR-MS 3.2.4a

Figure PTR-MS 3.2.4a shows the peak separation achieved at m/z=69 by the IONICON Analytik PTR-TOF 4000. PTR-MS Viewer 3 software was used to subtract the background and to produce this image.

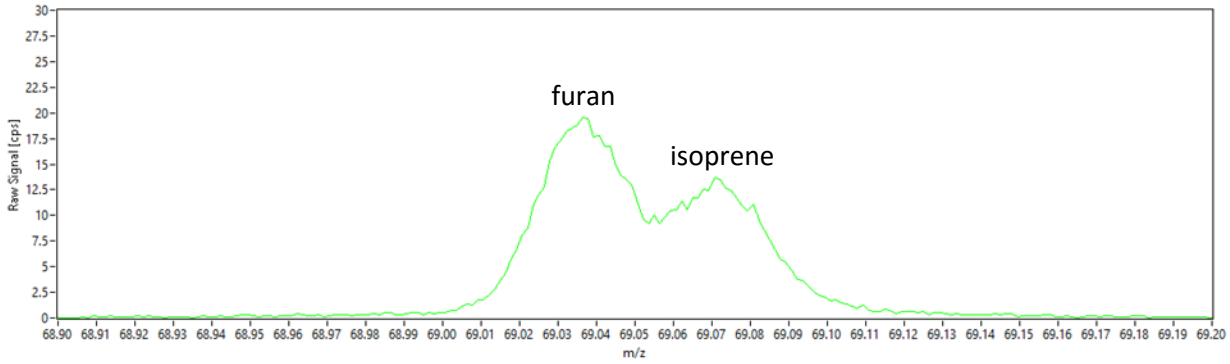
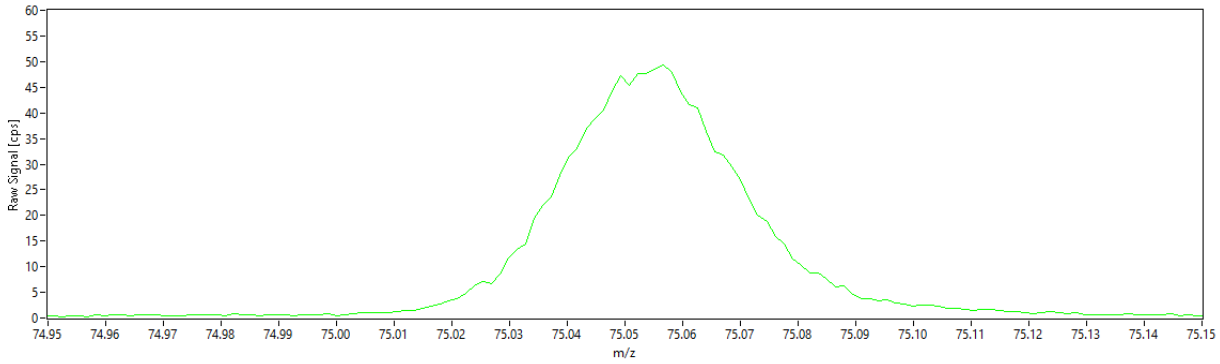

furan
isoprene
Raw Signal [cps]
m/z

Figure PTR-MS 3.2.4b

Figure PTR-MS 3.2.4b shows the lack of separation between peaks at m/z=75, NDMA and methyl acetate. PTR-MS Viewer 3 software was used to subtract the background and to produce this image.



February 28th, 2017

Summary

The mobile lab was operated on February 28th from 8:00 AM to 3:00 PM to verify the operation of the GC-MS system. Part of this process was to perform preliminary experiments to provide data for experiments conducted on March 1st. Approximately 100 mL of a 200 ppbv gas standard (CBAL-413-71B) was collected on 2 desorption tubes and then desorbed. The resulting 20 ppbv standard was then monitored via the Agilent 6890 GC and 5973 MS GC-MS.

Also, on February 28th the Picarro Cavity Ring-Down spectrometer was unpacked and set up to perform preliminary bench scale experiments. Some preliminary experiments with tubing were performed. These experiments will be repeated and reported in the Week 3.3 Report for March 3rd. Note that this is not the installation date of the Picarro in the mobile lab.