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| 005 | Weekly Report 3.3 | | | | | | | 00 |
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RJ LEE GROUP
DELIVERING SCIENTIFIC RESOLUTION

# WEEK 3.3 REPORT – GC-PTR-MS EXPERIMENTS

## March 1st, 2017

### Summary

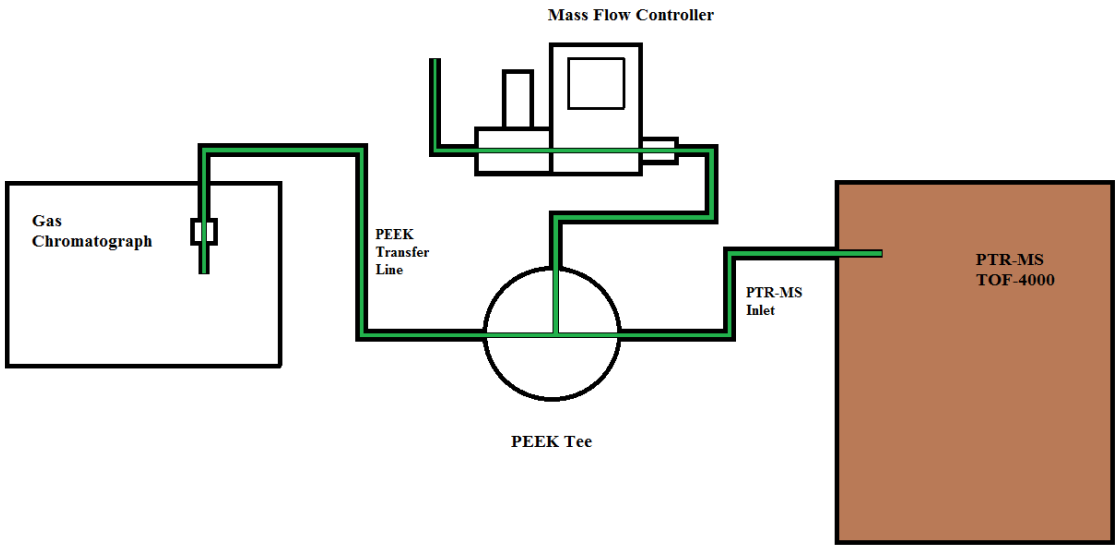
The mobile lab was used at CBAL on March 1st between 7:00 AM and 4:00 PM to conduct preliminary Gas Chromatography Proton Transfer Reaction Mass Spectrometer (GC-PTR-MS) experiments.

The GC column was connected to the PTR-MS inlet as shown in Figure GC-PTR-MS 3.3.1a. Samples in desorption tubes were prepared on the sample preparation interface. These samples varied in concentration and content. The Desorption Unit was then used to transfer samples to the GC column and from there to the PTR-MS to be monitored. Figure GC-PTR-MS 3.3.1b is an example of the PTR-MS signal. These were preliminary experiments and will inform future PTR-MS and GC interface experiments.

## March 1st Figures

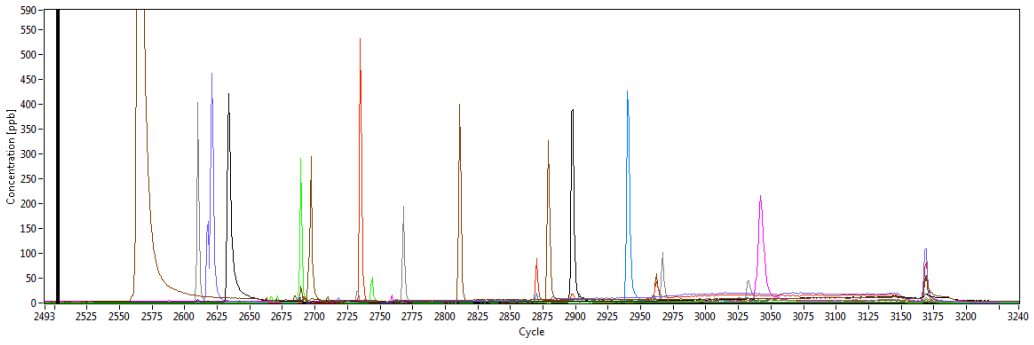
### Figure GC-PTR-MS 3.3.1a

This diagram displays the connection between the GC column and the PTR-MS inlet. The mass flow controller (MFC) was also connected to provide supplemental zero air to maintain proper drift tube pressure on the PTR-MS. The direction of the flow was as follows: the volatile organic compounds in the preconcentrated air sample flowed from the Desorption Unit to the GC and finally to the PTR-MS where signal was monitored.




### Figure GC-PTR-MS 3.3.1b

Figure GC-PTR-MS 3.3.1b is an example of the PTR-MS signal from sample gas separated on a GC column.



# WEEK 3.3 REPORT – GC-PTR-MS EXPERIMENTS

## March 2nd, 2017

### Summary

The mobile lab was used at CBAL on March 2nd between 7:00 AM and 1:15 PM to conduct more preliminary Gas Chromatography Proton Transfer Reaction Mass Spectrometer (GC-PTR-MS) experiments. The connection between the GC and PTR-MS was the same as that previously described in Figure GC-PTR-MS 3.3.1a with one major difference. The GC and PTR-MS were connected with a capillary tube up to the first Tee in the PTR-MS. The connection between the polyether ether ketone (PEEK) tee and the PTR-MS was made with a section of PEEK tubing containing the capillary tube.

Several samples of differing concentration and composition were analyzed via the system in an atempt to optimize conditions. The drift tube pressure of the PTR-MS and the GC flow rate were also varied to determine if they had an effect on peak width. At a later date these experiments will be expanded and the parameters evaluated.

Preliminary O2+ reagent ion experiments were also conducted.

# WEEK 3.3 REPORT – GC-PTR-MS AND PICARRO EXPERIMENTS

## March 3rd, 2017

### Summary

The mobile lab was used at CBAL on March 3rd between 6:30 AM and 2:00 PM to conduct Gas Chromatography Proton Transfer Reaction Mass Spectrometer (GC-PTR-MS) experiments.

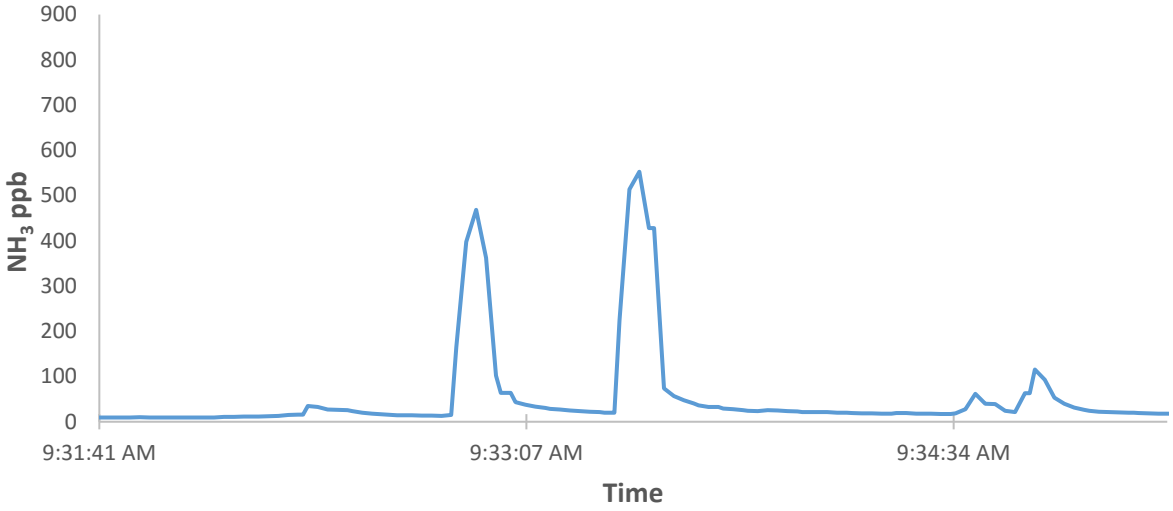
The GC-PTR-MS experimental setup was the same as that used on March 2nd. The focus during the experiments conducted on March 3rd was to collect and compare data in several different reagent ion modes. These reagent ions were H3O+, O2+, and NH4+. Two samples containing either the TO-15 standard or a nitrosamine mix were analyzed for each reagent ion. Different reagent ions are expected to have different advantages. For example, NH4+ should only protonate compounds with a greater proton affinity than NH3. This should eliminate signal from hydrocarbons and some chlorinated compounds while leaving all amines and some oxygenated species. This could potentially be used to eliminate the methyl acetate interference assocaited with m/z=75. While the background was much cleaner than that of H3O+, much noise was generated. The source of the noise is as yet unknown but was significant enough to hamper the ability to see a clear spectrum of the data. Due to the high noise level, it was difficult to derive any useful information from the data. Spectra from several of the O2+ and H3O+ experiments are included in Figures GC-PTR-MS 3.3.3a through GC-PTR-MS 3.3.3f.

In addition, the Picarro tubing experiments from February 28th were repeated and expanded. Over the course of this experiment a sampling tube was imursed in a nitrogen source. The amount of time the tube was within the source was kept constant, althought the ammonia concentration of the source was not necessarily constant. Both PEEK and perfluoroalkoxy alkane (PFA) tubing of differing diameter were used to conduct air to the analyzer. The signal was recorded and is displayed qualitatively in graphs Picarro 3.3.3a through Picarro 3.3.3f. Note that the same scale for both the x and y axes are equivalent in each graph and thus the signal quality can be directly compared. Ultimatley the data from this experiment indicate that long length PEEK tubing could be used as the sampling line. It also indicates that at least short lengths of 1mm inner diameter (ID) PFA tubing could be used. A future experiment will need to be conducted to determine if the high flow rate of the sampling manifold will provide adequate ammonia to the Picarro to produce a useable signal.

## March 3rd Figures

### Figure Picarro 3.3.3a

Figure 3.3.3a shows the signal produced with 2.5 ft of 1 mm ID PEEK tubing. This signal is sharp.




### Figure Picarro 3.3.3b

Figure 3.3.3b shows the signal produced with 2.5 ft 2 mm ID 1/8 in outer diameter (OD) PFA tubing. This signal is sharp.

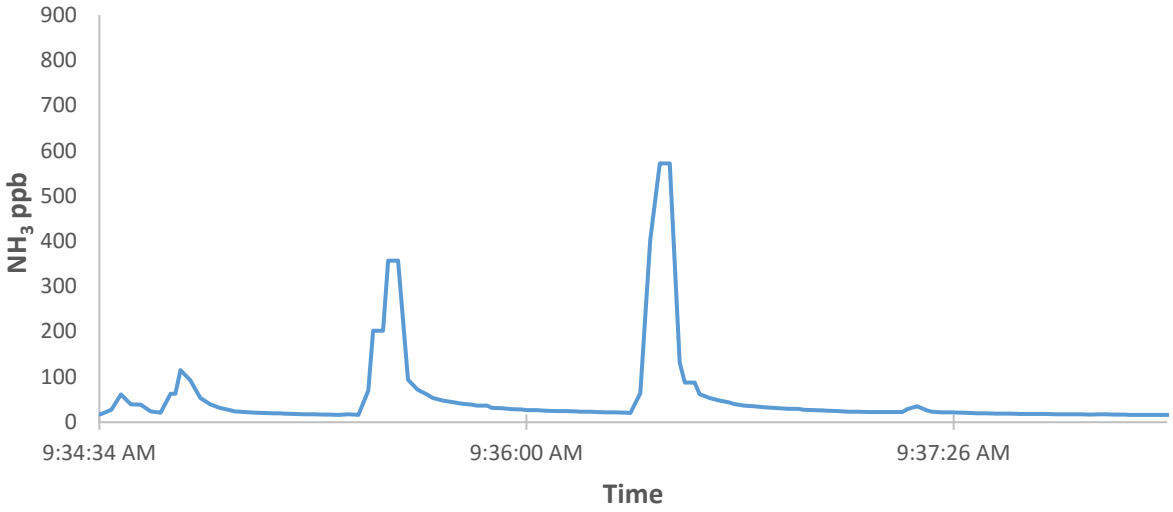


**Figure Picarro 3.3.3c**

Figure 3.3.3c shows the signal produced with 2.5 ft 2 mm ID 1/4 in OD PFA tubing. This signal is poor.

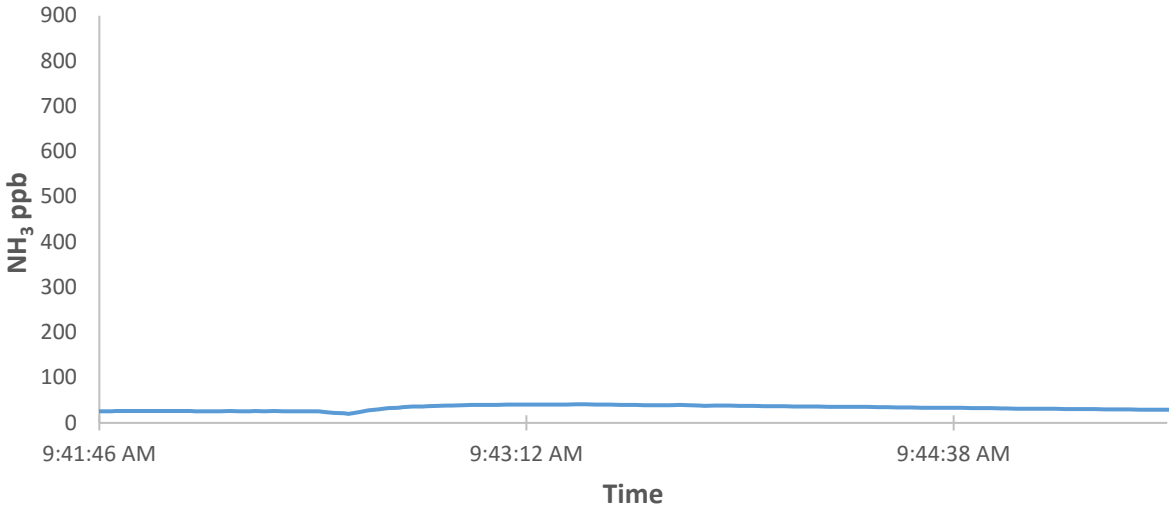



**Figure Picarro 3.3.3d**

Figure 3.3.3d shows the signal produced with 8.5 ft 4 mm ID PFA tubing. This signal is poor.

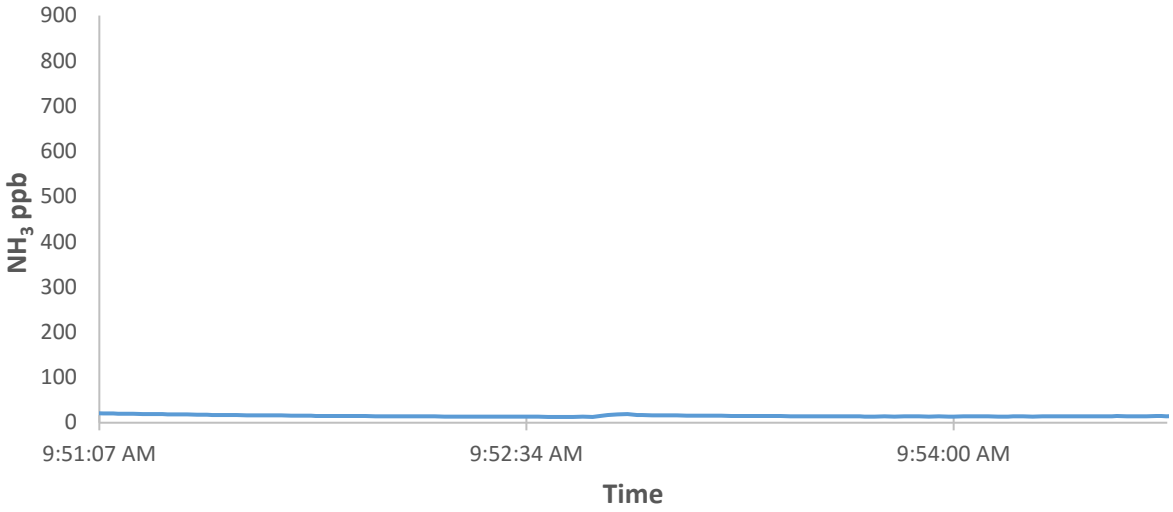


**Figure Picarro 3.3.3e**

Figure 3.3.3e shows the signal produced with 1.75 ft 1 mm ID PFA tubing. This signal is sharp.

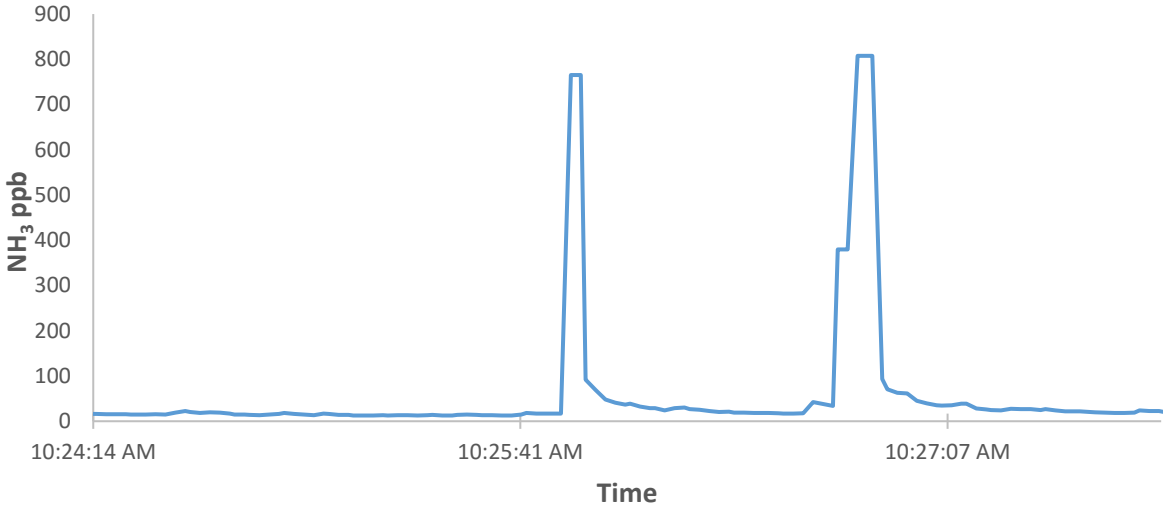



**Figure Picarro 3.3.3f**

Figure 3.3.3f shows the signal produced with 20ft 1 mm ID PEEK tubing. This signal is sharp.

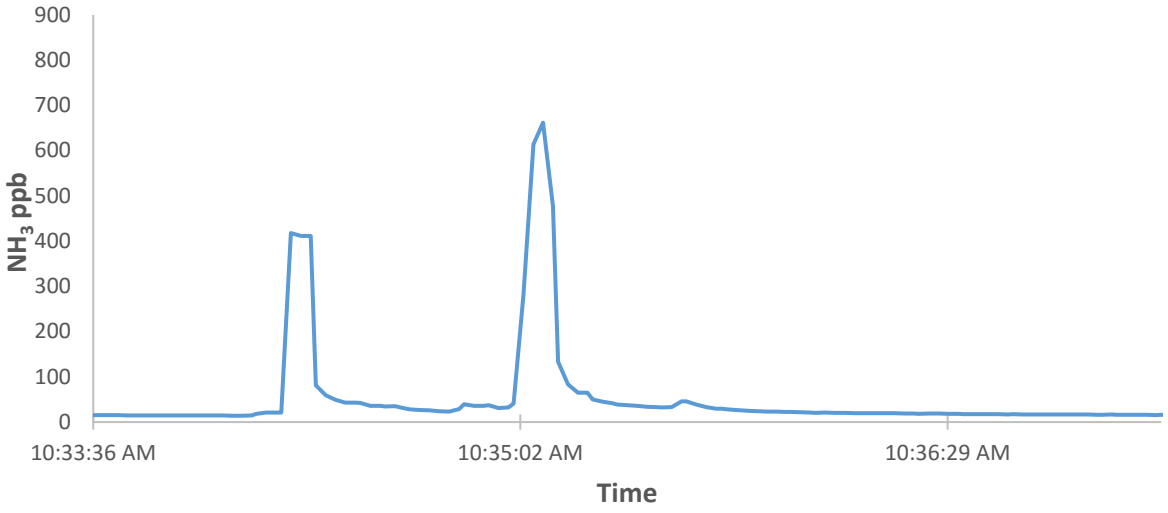


**Figure GC-PTR-MS 3.3.3a**

Figure GC-PTR-MS 3.3.3a displays an O2+ mode GC-PTR-MS analysis of a TO-15 standard containing 64 components. Signals displayed in red indicate aromatic compounds. Those in green are halogenated. The black signals are miscellaneous hydrocarbons. Each component has an approximate concentration of 250 pptv and the GC-PTR-MS run time was 26 minutes.

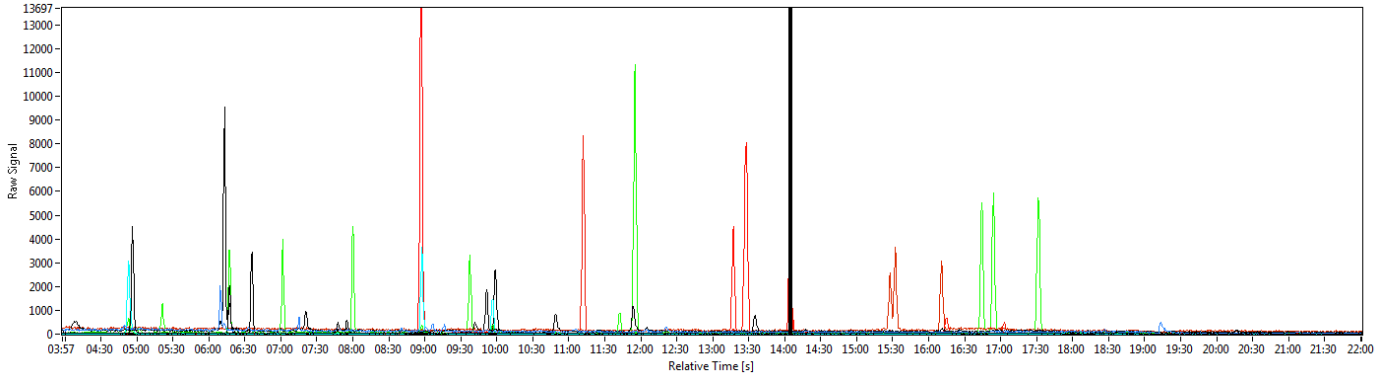



**Figure GC-PTR-MS 3.3.3b**

Figure GC-PTR-MS 3.3.3b displays an H3O+ mode GC-PTR-MS analysis of a TO-15 standard containing 64 components. Each component has an approximate concentration of 250 pptv and the GC-PTR-MS run time was 26 minutes.

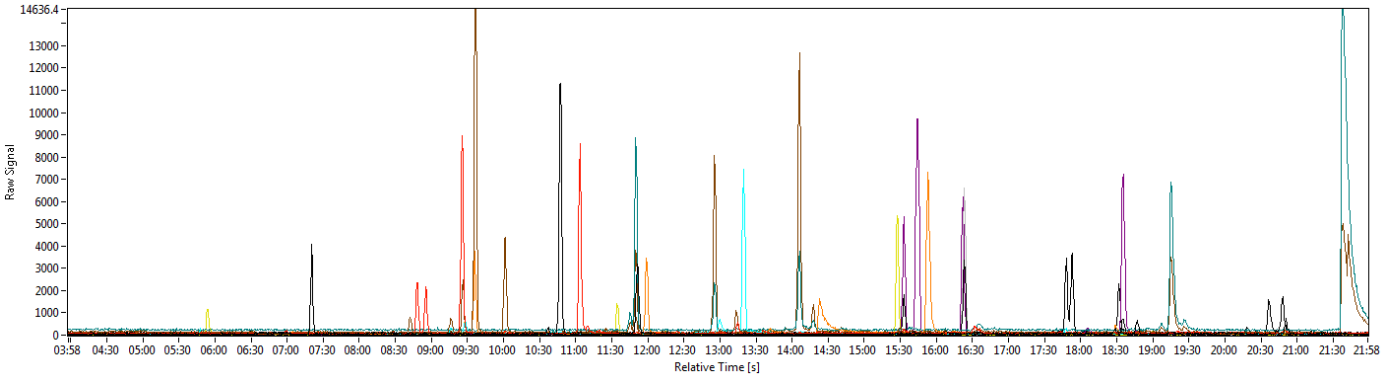


**Figure GC-PTR-MS 3.3.3c**

Figure GC-PTR-MS 3.3.3c displays an O2+ mode GC-PTR-MS analysis of a VOC mixture.

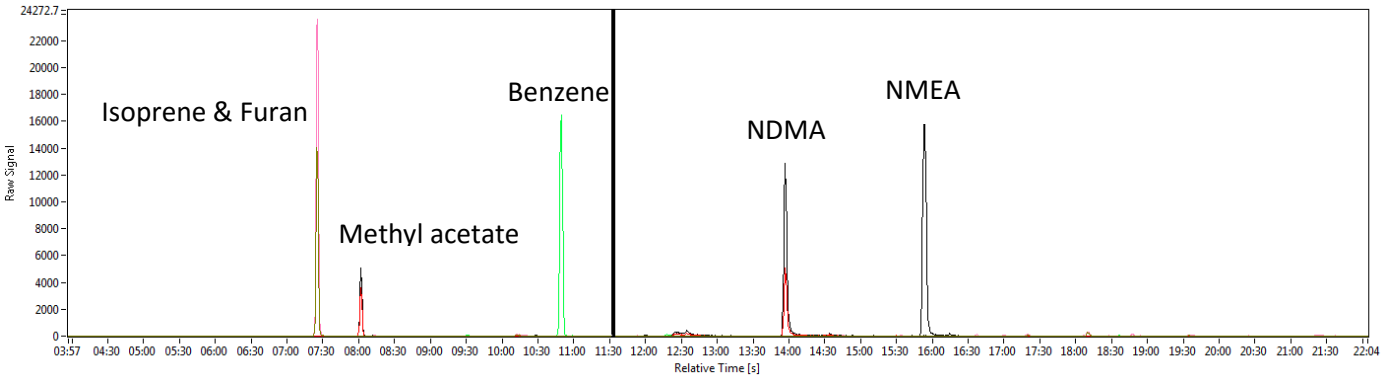



**Figure GC-PTR-MS 3.3.3d**

Figure GC-PTR-MS 3.3.3d displays a magnification of the furan and isoprene signals for an O2+ mode GC-PTR-MS analysis of a VOC mixture. This shows their mass separation in this mode of operation.

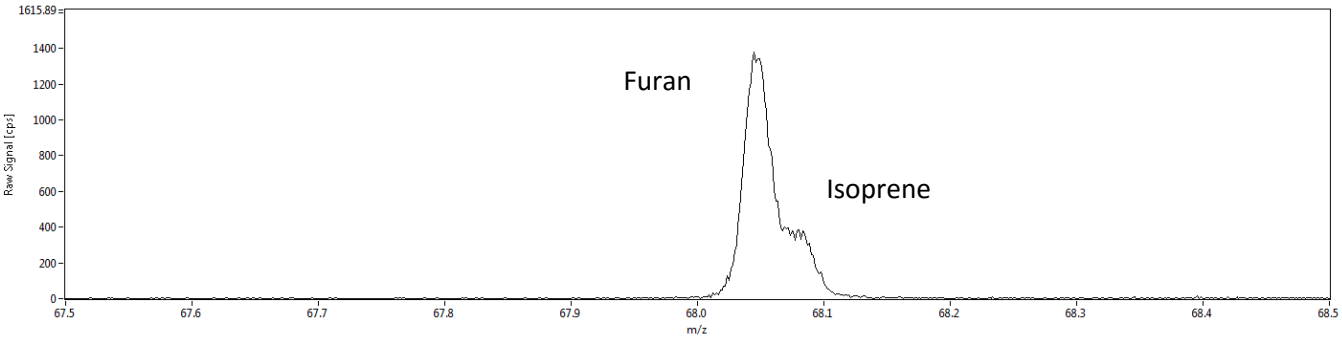


**Figure GC-PTR-MS 3.3.3e**

Figure GC-PTR-MS 3.3.3e displays H3O+ mode GC-PTR-MS analysis of a VOC mixture.

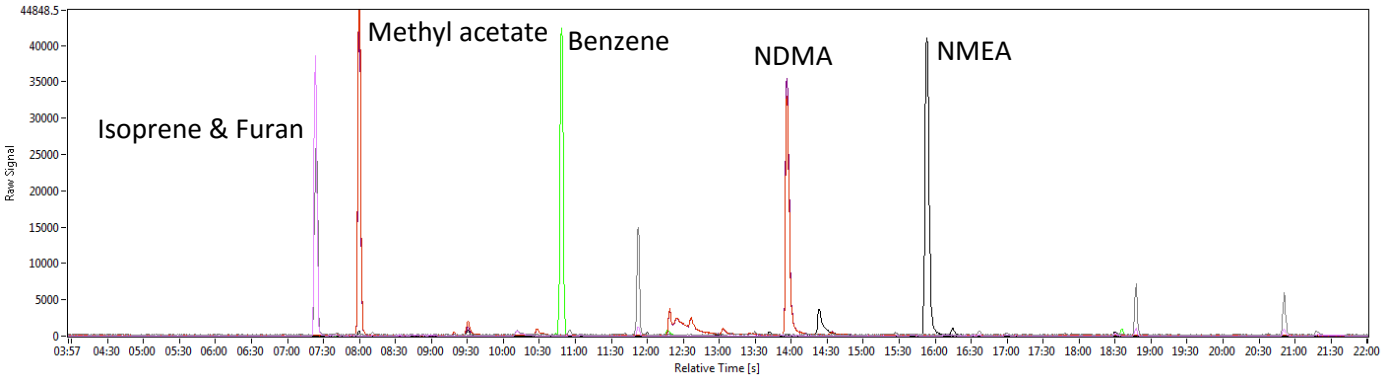
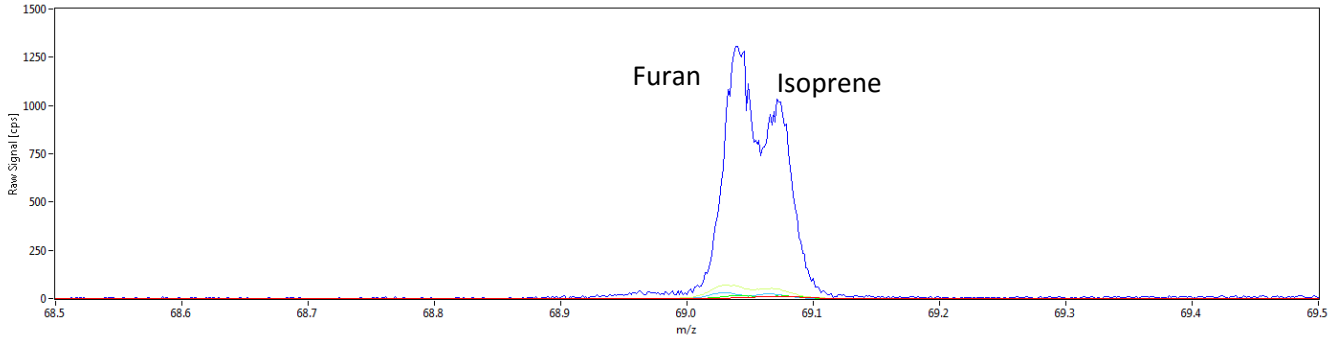



**Figure GC-PTR-MS 3.3.3f**

Figure GC-PTR-MS 3.3.3f displays a magnification of the furan and isoprene signals for an H3O+ mode GC-PTR-MS analysis of a VOC mixture. This shows their mass separation in this mode of operation.



# WEEK 3.3 REPORT – PICARRO INSTALLATION

## March 7th, 2017

### Summary

The mobile lab was used at CBAL on March 7th between 12:20 PM and 3:30 PM to install the Picarro G2103 Cavity Ring Down Spectrometer Ammonia Analyzer.

The Mobile Lab was prepared for the Picarro installation by moving the electronics cabinet forward, rearranging power cords, and adding a shelf for the Picarro. The cabinet was then moved back into place. Two representatives of WRPS QA were present to witness the installation. The Picarro was moved from the CBAL conference room and then transported to the Mobile Lab where it was installed. The instrument was powered up to display functionality and then powered down at the end of the day.