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Submittal Number	Submittal Description	Specification Number	Specification Paragraph Number	Copies E / H	TOC Document Number	Rev	Supplier Document Number	Supplier Rev
005	Weekly Report 4.3							00

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WEEK 4.3 REPORT – Li-Cor Calibration

March 29th, 2017

Summary

On March 29th, the Mobile Lab was used at Columbia Basin Analytical Laboratories (CBAL) from 7:45 AM to 3:30 PM. Some troubleshooting work was performed on the sample preparation interface. Also, preliminary experiments for the binary comparison of compounds were performed.

During this time the Li-Cor CO_2 Analyzer underwent two calibration verifications (CVs) at a concentration of 1008 ppm. Both were within the 15 percent range indicated by the 2017 Test Plan until enough data points have been collected to establish a statistical limit based on control charting. This check was conducted with and without a digital flow meter. The data from these checks indicate that more accurate and precise data is collected without the flow meter. With the meter in line with the analyzer the CV resulted in a value of 1010.6 ppm with a standard deviation of 4.0 ppm. With the meter removed from the line the result of the CV was 1008.2 ppm with a standard deviation of 0.7 ppm. The purpose of the flow meter in this CV system is only to ensure optimum flow rate to the Li-Cor CO_2 Analyzer. It can be removed from the CV system after the flow rate has been established without impacting the analysis. No zero air checks were performed on this day.

Also, some compounds were analyzed via Gas Chromatography-Mass Spectrometry (GC-MS) as preliminary activities for building a relative retention time library started on March 31st.

WEEK 4.3 REPORT – Preliminary Picarro Calibration Check

March 30th, 2017

Summary

On March 30th, the Mobile Lab was used at CBAL from 8:00 AM to 4:30 PM. During this time the Li-Cor CO_2 Analyzer underwent a 1008 ppm CV. The result was 1007.9 ppm which was within the administrative accuracy limit of +/- 15 percent. No zero air check was performed on this day.

Also, some preliminary calibration activities were performed on the Picarro NH_3 Analyzer. A CV conducted at 9.887 ppm fell outside of the temporary 15 percent range indicated in the 2017 Test Plan, which indicated that a calibration needed to be performed. Work with the Picarro indicated response time of several seconds to changes in concentration, although stabilization of each signal could take 3 to 4 minutes. Zero air checks that took over 30 minutes indicated that the instrument requires much time to recover from high ammonia concentrations.

WEEK 4.3 REPORT – GC-MS Preparation

March 31st, 2017

Summary

On March 31st, the Mobile Lab was used at CBAL from 8:00 AM to 2:30 PM. During this time a preliminary calibration was run on the Picarro NH_3 Analyzer from 0 to 29.7 ppm. This calibration was later found to be unacceptable because a subsequent CV did not pass

Additionally, the GC-MS was used to identify retention times relative to benzene and 4-bromofluorobenzene for many compounds. These included toluene, methylvinylketone, 2,3-dihydrofuran, methyl acrylate, furan, methyl acetate, butyl acetate, ethyl acetate, cyclopentane, isoprene, 2,5-dihydrofuran, cyclohexane, 2-n-heptylfuran, heptanenitrile, valeronitrile, 2-methylfuran, 2 hexanone, tert-nitrobutane, 2-ethyl-2-hexenal, butyronitrile, 2-pentylfuran, ethanol, m-xylene, 1,2,3-trimethylbenzene, styrene, acetonitrile, ethyl acrylate, tetrachloroethylene, pyridine, 3-hexanone, 2-octanone, p-xylene, 2,2-dimethoxypropanone, 2-pentanone, and 2,4-lutidine. These activities will continue as scheduling allows. A table displaying the results of the data collected is included in Appendix A at the end of this report.

WEEK 4.3 REPORT – Picarro Calibration

April 3rd, 2017

Summary

On April 3rd, the Mobile Lab was used at CBAL from 8:00 AM to 3:00 PM. During this time a calibration was performed on the Picarro NH_3 Analyzer. The Picarro was recalibrated because a midrange CV did not pass. The calibration was performed from 0 to 29.7 ppm and 6 CVs were performed at various concentrations. The midrange CV of 4.94 ppm did not pass indicating that another calibration would need to be performed to improve the slope of the calibration curve. Furthermore, it was conducted with the Liquid Calibration Unit which was tagged out of service for calibration. Subsequent CVs could not be performed using the same system.

A CV was also performed on the Li-Cor CO_2 analyzer at 1008 ppm. The Li-Cor passed this CV with 1014 which is within administrative accuracy limit of +/- 15 percent . No zero air check was performed. Work was done to prepare the mobile lab to return to the field including securing items and adjusting insulation.

WEEK 4.3 REPORT – Picarro Calibration

April 4th, 2017

Summary

On April 4th, the Mobile Lab was used at CBAL from 6:45 AM to 6:00 PM. During this time tubing for the CO_2 and NH_3 calibration verification gases were plumbed to the instrument to increase efficiency and reduce tripping hazards. Then a system of mass flow controllers was used to create a calibration and calibration verification system to be used until such a time as the Liquid Calibration Unit is returned to service. Information on the identification of these two flow controllers is included in Table 4.3.5a.

The Picarro NH_3 Analyzer was then recalibrated using this new configuration and a calibration verification was passed. The calibration was conducted per the instructions in the Picarro G2103 Analyzer for NH_3/H_2O User's Guide (WI-019.00). Concentrations used in this calibration were 0, 4.9, 14.8, and 29.7 ppm NH_3. This calibration is recorded in CBAL-413. The CV was performed at 9.9 ppm and the result was 9.0 ppm. This falls within the administrative accuracy limit of +/- 15 percent.

The Li-Cor CO_2 Analyzer passed a calibration verification of 1008 ppm with a concentration of 1013.7 which is within the administrative accuracy limit of +/- 15 percent. Furthermore, the lab was prepared to return to the field.

April 4th Figures

Table 4.3.5a

Table 4.3.5a displays identification information for the Mass Flow Controllers used to performs calibrations and calibration verifications in the Mobile Lab while the LCU is out of service. This information is also available in WI-024.00.

M&TE	Model #	Serial #	Unique ID #	Location
Mass Flow Controller	FMA-2605A-TOT	22790	CLS-22790	Calibration and CV System
Mass Flow Controller	FMA-2605A-TOT	29005	CLS-29005	Calibration and CV System

WEEK 4.3 REPORT – Appendix A

March 31st, 2017

Table 4.3.Aa

Table 4.3.Aa lists compounds analyzed via GC-MS. Also displayed are the relative retention times of each compound to both benzene and 4-bromofluorobenzene. This information will be used to more accurately identify compounds measured via the GC-PTR-MS system.

Compounds	RT Benzene	RT 4-Bromofluorobenzene
Benzene	1.0000	0.5741
4-Bromofluorobenzene	1.7419	1.0000
Toluene	1.2995	0.7460
Methylvinylketone	0.8385	0.4814
2,3-Dihydrofuran	0.7571	0.4347
Methyl Acrylate	0.8755	0.5026
Furan	0.5687	0.3265
Methyl Acetate	0.6523	0.3751
Butyl Acetate	1.4110	0.8113
Ethyl Acetate	0.8656	0.4977
Cyclopentane	0.6914	0.3975
Isoprene	0.5704	0.3279
2,5-Dihydrofuran	0.8628	0.4961
2,3 Dihydrofuran	0.7535	0.4312
2-Methylfuran	0.8148	0.4663
2-n, Heptylfuran	2.2862	1.3081
Cyclohexane	0.9597	0.5491
2 Hexanone	1.3998	0.8009
Tert-Nitrobutane	1.3176	0.7531
2-Ethyl-2-Hexenal	1.9405	1.1091
Butyronitrile	1.1365	0.6496
2-Pentylfuran	1.8470	1.0557
Ethanol	0.5289	0.3026
M-Xylene	1.5782	0.9030
1,2,3-Trimethyl Benzene	1.9730	1.1289
Styrene	1.6517	0.9451
Acetonitrile	0.6407	0.3666
Ethyl Acrylate	1.1018	0.6304
Tetrachloroethylene	1.3892	0.7949

Pyridine	1.2792	0.7321
3-Hexanone	1.3790	0.7892
2-Octanone	1.9011	1.0880
P-Xylene	1.5806	0.9046
2,2-Dimethoxypropanone	0.9436	0.5401
2-pentanone	1.1158	0.6386
2,4-Lutidine	1.7594	1.0069