Gasoline Range Organic Detection and Screening Using Static Headspace

EST analytical

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INTRODUCTION

In general, Gasoline Range Organic (GRO) compounds correspond to a range of hydrocarbons from C6 to C10. These hydrocarbons have boiling points below 170°C and are often found in ground water and soils. The cause of this water or soil contamination can vary from the simple leaking underground storage tank to catastrophic such as the Gulf Oil spill. When GRO compounds are found, the site of the pollution needs to be tested in order to determine the level of the contamination. One method to determine the amount of contamination is to perform static headspace sampling in conjunction with Gas Chromatography/ Mass Spectrometry analysis. This poster will explore static headspace sampling of GRO water samples using the EST Analytical LGX50.

DISCUSSION

Analysis of field samples can sometimes be complicated by how contaminated or "hot" the sample is. Many times the compounds contributing to the contamination are GRO compounds. In order to test these samples it can be helpful to know what kind of dilution needs to be done on the sample. The LGX50 can perform headspace screening on the sample in order to determine the dilution factor needed for purge and trap sampling so as to preserve the concentrator from high levels of GRO compounds.

The LGX50 is also an ideal instrument for high level GRO compound sampling. The system can perform static headspace sampling of a GRO sample by simply placing a portion of the water and/or soil in a 40mL vial and setting the vial on the sample tray. The LGX50 does the rest of the work. This paper will discuss static headspace sampling of GRO compounds in a water matrix using both a commercially purchased standard and a sample of raw gasoline.

EXPERIMENTAL

The LGX50 sampling system was configured with a 1mL sample loop and connected to a GC/MS for analysis. The GC column employed for this study was a Restek Rtx-624 20m x .180mmID x 1.0µm. The LGX50 was run in headspace screen mode. For this analysis, a sample was placed in a 40ml vial with a stir bar and positioned in the LGX50 sample tray. The LGX50 moved the vial from the sample tray to the sampling station where the sample was heated and stirred and sampled into a 1ml loop where the analytes were transferred to the GC/MS for calibration and analysis. See **Figure 1** for the LGX50 sampling graphic and **Tables 1 and 2** for LGX50 and GC/MS parameters respectively.

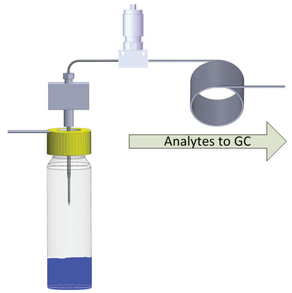

Figure 1: LGX50 Sampling

The Gasoline Range Organic standard was obtained from Restek. The standard was Wisc PVOC/GRO and contained ten GRO analytes at a concentration of 100ppm each for a total concentration of 1000ppm. A five-point curve was prepared with a concentration range of 100ppb to 2ppm for the individual analytes (200ppb to 4ppm for p&m-Xylene) and 1ppm to 20ppm for the C6-C10 gasoline range. Finally, seven consecutive low point calibration standards were run in order to establish MDLs and seven consecutive mid-point standards were run in order to verify the precision and accuracy of the analysis. The curve, MDL and precision and accuracy results are all listed in **Table 3** while a chromatogram is displayed in **Figure 2**.

Table 1. LGX50 Sampling Parameters

LGX50 Autosampler Parameter	Setting
Sample Type	Screen
Sample Fill Mode	Loop
Sample Volume	0ml
Syringe Prime	3 sec.
Syringe Needle Rinse	20ml
Rinse Cycles	Off
Sample Temperature	85°C
Stirrer	On/Medium
Sample Equilibration Time	15 min.
Vial Pressurization Time	3 sec.
Loop Fill Time	3 sec.
Loop Equilibration Time	3 sec.
Valve Temperature	115°C
GC Line Temperature	150°C
GC Cycle Time	18 min.
Rinse Water Temperature	65°C
IS	0µl

Table 2. GC/MS Experimental Parameters

GC/MS	Agilent 7890A/5975C inert XL
Inlet	Split/Splitless
Inlet Temp.	200°C
Inlet Head Pressure	9.64 psi
Mode	Split
Split Ratio	30:1
Column	Rtx-624 20m x 0.18mm I.D. x 1.0µm film thickness
Oven Temp. Program	45°C hold for 1 min., ramp 20°C/min to 220°C, hold for 1.25 min.
Column Flow Rate	1.0mL/min
Gas	Helium
Total Flow	34.0mL/min
Source Temp.	230°C
Quad Temp.	150°C
MS Transfer Line Temp.	180°C
Scan Range	m/z 35-300
Scans	2.73 scans/sec
Solvent Delay	1.0 min

Table 3: Experimental Results Summary

Compound	Curve %RSD	Curve Linear Regression	Average Response Factor	MDL Spike Level	MDL	Precision and Accuracy Spike Level	Precision (%RSD)	Accuracy (%Recovery)
C6-C10 (GRO Standard)	3.52	1.000	3.971	1000.00	95.68	10000.00	3.21	94.90
MTBE	3.62	1.000	1.169	100.00	10.31	1000.00	2.78	95.17
Benzene	4.57	1.000	1.716	100.00	9.01	1000.00	3.17	94.07
Toluene	2.53	1.000	1.039	100.00	14.48	1000.00	2.99	94.34
Ethylbenzene	2.41	1.000	1.877	100.00	12.95	1000.00	3.14	98.65
p&m-Xylene	4.63	1.000	1.451	200.00	28.01	2000.00	3.17	98.76
o-Xylene	2.18	1.000	1.496	100.00	7.28	1000.00	3.02	97.50
1,3,5-Trimethylbenzene	3.39	1.000	1.478	100.00	14.61	1000.00	3.23	100.79
1,2,4-Trimethylbenzene	2.90	1.000	1.473	100.00	11.46	1000.00	3.03	100.43
Naphthalene	5.07	1.000	1.239	100.00	17.46	1000.00	3.05	102.95
C6-C10 (Gasoline)	4.67	0.999	2.209	1000.00	87.52	10000.00	3.93	94.57

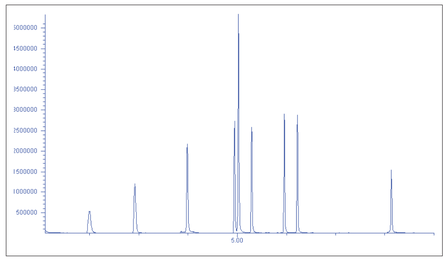

Figure 2. Chromatogram of GRO Standard

After LGX50 screen parameters were identified for the GRO standard, a raw gasoline sample was obtained. The raw gasoline was diluted to 5000ppm and used to make a series of standards from 1ppm to 20ppm in order to establish a five point C6-C10 gasoline curve. Furthermore, a series of seven low, 1ppm gasoline standards were run in order to establish MDLs, while seven 10ppm standards were run for the verification of precision and accuracy. The results of this study are also listed in **Table 3**. The 10ppm raw gasoline chromatogram is shown in **Figure 3**.

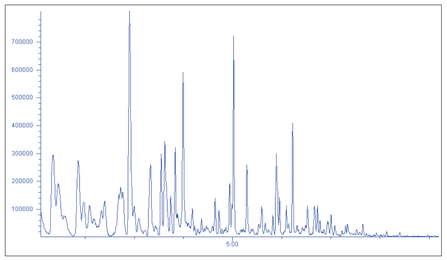

Figure 3. Chromatogram of Raw Gasoline

CONCLUSION

The LGX50 proved to be an excellent system for GRO headspace sampling. The system provided linear curves with the %RSD at less than 5% for all of the compounds and the linear regression at 0.999 or better. The precision and accuracy results were also outstanding with precision at better than 5% and percent recovery between 94 and 105% for all of the compounds. The LGX50 is also versatile in the fact that it can be used for both GRO sampling and headspace screening of "hot" samples thus making it an excellent addition to any environmental lab.

REFERENCE

Volatile Organic Compounds in Soils and Other Solid Matrices using Equilibrium Headspace Analysis, Method 5021, Revision 0, December, 1996.