

A More Comprehensive And Sensitive Method For CO₂ Quality Assurance

Application Note:

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Authors

Chris Casteel Dan Cardin Entech Instruments, Inc Simi Valley, CA 93065

Abstract

A technique for the comprehensive analysis of contaminants in Carbon Dioxide has been developed which quantifies a wide range of compounds that previously required multiple methods. Samples are collected in ultra inert fused silica lined stainless steel canisters for subsequent GC/MS analysis of most compound classes including aldehydes, ethers, ketones, alcohols, mercaptans, sulfides, hydrogen sulfide, and various classes of hydrocarbons and halocarbons. Detection levels are 10-100 times lower than current methods, allowing off-flavor and potentially toxic compounds to be monitored at levels which might affect product quality. Through sample preconcentration, LOD's in the sub-ppb range are easily achieved. Fused silica coating of the sampling container, sampling pathway, and sample inlet help to minimize contact with reactive surfaces, thus insuring sample integrity. The inertness of these surfaces acts to further reduce detection limits while allowing investigation of other compound classes for which no previous methodology has been developed. Finally, this



method is readily adaptable to other areas of quality control including testing of product reagents, packaging, and the final product. Multi-sample automation is available to reduce the cost associated with on-going Quality Assurance Programs.

Introduction

As a multi billion-dollar/year industry, beverage manufacturers' success depends on product development and quality assurance. Contamination events of the past have raised serious questions with both regulatory agencies and consumers. The Food and Drug Administration (FDA) requires all manufacturers to regularly test their products for potentially harmful contents. The FDA monitors compliance by performing site audits and random testing of their own. These measures help to minimize potential dangers to consumers. However, discovering a problem at the final product stage results in reduced productivity and the loss of The entire product, including materials and ingredients that did not originally contain the offending odors or contaminants. A more prudent approach involves testing of the starting materials before they are actually combined with and contaminate other ingredients. As an important ingredient in a wide variety of beverages, CO2 testing is a good place to begin.

Carbon Dioxide is used to provide effervescence and mild acidity that in-turn provides some protection against microbial growth.¹ Since the sources for CO2 can vary greatly, the potential for undesirable or harmful contamination is much greater than with most other ingredients. Contamination can occur from the initial CO₂ source as well as purification, delivery, and storage processes. Due to the number of different contaminants. several methods have been developed to monitor purity. Each method is specific to a certain number or class of contaminants. These methods are used to monitor voluntary guidelines for CO2 quality. The criteria for determining these guidelines are based on observed sensory effects, known process contamination, or regulatory enforcement. Unfortunately, these methods are often not sensitive enough to detect levels that could be harmful or which may affect product quality. In addition, performing several tests to cover the range of suspected contaminants is costly and time consuming.

In general, non-mass spectrometer based detectors produce small voltage variations as compounds pass through them. Different detectors vary in response, based on the type of compound or compound class. The more specific/sensitive to a certain class of compounds the detector is, the fewer classes of compounds to which it will respond. Conversely, the more classes of compounds the detector responds to, the more prone it is to coelutions and other chemical noise. Mass spectrometers, however, can detect a wide range of compounds with high specificity and relatively good sensitivity. They are limited by their reduced dynamic range and possible matrix interference from CO2 and water. By employing a sample inlet system that

reproducibly and quantitatively introduces small to large sample volumes, the method dynamic range can be significantly increased. Similarly reducing or eliminating CO2 and water interference allows the mass spectrometer to become more effective for high CO2/high humidity samples. With the right sample introduction technique, the GC/MS becomes an extremely powerful tool for quantitative analysis and qualitative identification of a diverse range of contaminants.

Sample integrity is best assured by introducing the sample directly from the source to the analytical system. Because analytical systems are rarely this portable, other means of bringing the sample to the equipment is necessary. Concentration onto sorbent media is made possible due to sample interactions with the media. Subsequent extraction from the media often introduces artifacts from the media or the extraction process itself. Artifacts interfere with the analysis and can raise detection limits. The reactivity of some of the potential contaminants is what prompted the ISBT to require all sample pathways to be deactivated. It only follows that the sampling container should likewise be deactivated and non-adsorptive.

The use of Silonite coated mini-cans offers the best solution to these requirements. The Silonite process deposits a uniform layer of fused silica onto stainless steel surfaces. The small size of the mini-can facilitates easy set-up and economical transport. The 400 ml capacity is more than enough to meet the detection limits necessary. Should lower detection limits be required, 1.0 Liter, 2.7 Liter, 3.2 Liter, and 6.0 Liter canisters are available with this coating. This coating is likewise deposited on all surfaces of the sample pathway of the 7100A Preconcentrator.

Experimental Section

EPA Method TO-15 standards containing 65 different aromatic, halogenated, and oxygenated compounds were prepared in 0.4 Liter Entech Minicans (29-MC400L). The standards were prepared at 6 different concentrations using an Entech 4600 Dynamic Diluter, 1.0 ppm Spectra Gas stock cylinders, a 5 ppm Sulfur standard from HP Gas Products, a 1-5 ppm carbonyl standard from Apel-Riemer Environmental, and CO2 from Airgas.

Sampling equipment for C02





Pressure Reducing Sampler For Nupro Valve Cans

Pressure Reducing Sampler For QC Valve Cans

MC400L MiniCan



With Nupro Valve

621 6 Liter Canister

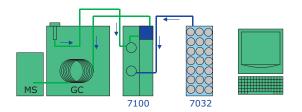
Analytical System

GCMS Inlet System: 7100 Preconcentrator and 7032L 21 Position Autosampler (Entech Instruments, Inc.)

7100 Mode of Operation: Microscale Purge and Trap

GCMS: Agilent 6890/5973N (Palo Alto, CA) Column: DB-5MS, 0.32mm ID, 60 m, 1um Temperature Program: 35C (5 min), 6C/min to 140C, 15C/min to 220C. Hold @ 220C for 3

Diagram of Analytical Equipment



Preconcentration and GC/MS Analysis

In order to reach low ppb or ppt detection limits, an aliquot of the CO2 sample must be concentrated before injection into the GCMS. The concentration procedure eliminates most of the water and CO2 as these compounds can interfere with GCMS analyses. To minimize interference, a 3 stage-trapping procedure called "Microscale Purge and Trap" was utilized. The first stage (M1) uses a glass bead/Tenax filled trap cooled down to -80°C. As the sample is passed through this trap, the majority of Carbon Dioxide passes through the trap while water, some Carbon Dioxide, and all the Volatile Organic Compounds (VOCs) are condensed onto the glass beads and Tenax. The heavier compounds are trapped onto the glass bead portion while the lighter end including H2S and some CO2 are trapped onto the Tenax. This trap is then warmed to 10C. For dry CO2 samples, the M1 trap can be desorbed at higher temperatures. A small volume (40 cc) of helium is then back-flushed through M1 to transfer the carbon dioxide and compounds of interest to a second Tenax trap (M2) which has been cooled to -60°C. Most of the water remains condensed on the glass beads in module 1 and is baked off later. Most of the remaining CO2 passes through the tenax in M2 at -60°C and is pumped away. At -60°C the break through volume for H2S is greater than 40cc so it is retained in M2. After transfer to the tenax in M2, an open tubular

third trap (M3) is cooled to -170° C. M2 is then heated to $+180^{\circ}$ C and back-flushed with Helium to transfer the sample from M2 to the pre-column focusing trap (M3). At -170° C the VOCs and any remaining water and CO₂ are condensed onto a $1/32^{\circ}$ OD Silonite coated stainless steel transfer line to refocus the sample before introduction to the GC. M3 is then rapidly heated to above 50° C to inject the VOCs onto the GC column. The first two traps (M1 and M2) are then baked out to prepare them for the next sample.

As required by the ISBT all sample pathway lines are deactivated. The Silonite gas phase deposition process developed at Entech achieves this deactivation. This inert surface allows reactive compounds such as H2S and Formaldehyde to be quantitatively transmitted to the analytical system.





Results and Discussion

A chromatogram of a 40 ppb standard is shown in Figure 1. Observable at the beginning of the run, the CO2 has been reduced to a level that it is not a factor in the quantitative analysis of the 68 component polar/non-polar VOC standard. Had H2S not been a compound of interest, trapping conditions could have been changed to eliminate even more of the CO2. Conditions for these tests were set to quantitatively transfer H2S while eliminating as much CO2 as possible. The quantitative nature of this method was tested by running a 5-point calibration curve.

The results of a 5-point calibration (Table 1) shows the effectiveness in quantifying a wide range of compounds. With a percent relative standard deviation (%RSD) of 8.9%, H2S is shown to be quantitatively transferred and recovered. A wide range of volatile organics with higher boiling points and varying polarities are similarly transferred and recovered. Although not quite as impressive, the %RSD for Formaldehyde is well within EPA and other method guidelines for limits of precision. Considering that no other method has demonstrated formaldehyde at these levels using underivitized GC injection, this %RSD is quite impressive. Generally another form of trapping called cold trap dehydration (CTD) is recommended for formaldehyde due to ts solubility in liquid water. CTD works well with high CO2 matrices, but not as well with some of the other compounds. Using CTD, %RSDs below 10% for formaldehyde, and most of the other VOCs listed are readily achieved. The purpose of this test was to show that a wide range of compounds, which otherwise would have taken several analyses, can be tested for in a single analysis.

Table 2 contains the results of a detection limit study performed in a 99+% CO2 matrix. Standard criteria for instrument detection limits were applied. This procedure calls for seven replicates of a standard at or near the expected detection limit. The instrument detection limit (IDL) is defined as 3 times the standard deviation of the results for each compound. For this determination, 100 ml of a 68 compound 2.0 ppb standard in CO2 was analyzed seven times. The results in Table 2 show that all IDLs are well below the detection limits currently listed in the ISBT methods. Concentrating a larger volume of sample can lower these detection limits even further. Using this instrumentation, sample volumes up to 1.0 Liter can be concentrated, thereby reducing these detection limits by another order of magnitude, if needed.

The real power in using full scan GC/MS is the ability to reduce or eliminate false positives and to identify unknown peaks. With other techniques, a detector response at a certain time is considered a positive identification of a contaminant. However, this assumes that nothing else in the sample could create a response at that time. Since this isn't always the case, confirmatory analyses using a different column and/or set of conditions would be necessary. GC/MS yields not only time/response data, but also mass spectral data that can be compared against the 100,000+ compound National Institute of Science Technology (NIST) Library to determine the possible identity of the unknown compound. Semi-quantitative results for these non-calibrated responses can also be determined since the Total Ion Current (TIC) produced by most compounds in a mass spectrometer are within a factor of 2-3 of each other.

Conclusion

Current methodologies only allow Levels of Detection (LOD's) down to 20 ppb⁻¹. However, compounds such as hydrogen sulfide, mercaptans, and thiophenes have olfactory LOD's down to sub-ppb levels and may alter product quality if not monitored at these levels. Other compounds such as vinyl chloride and 1,3butadiene may create health hazards unless limited to low or sub-ppb levels. Reaching these LOD's requires the use of more inert sampling media, more inert sample pathways to the analytical instrumentation, and a means of introducing larger volumes of sample without compromising the sample integrity. The Entech 7100A Preconcentor meets these requirements offering improved sensitivity and extended

By preconcentrating a larger volume of sample before injection into a GC or GC/MS, the 7100A reduces detection limits regardless of what type of detector is used. However, the almost complete elimination of water and C02 in the sample allows GC/MS to be used for sensitive, confirmatory detection of target compounds, while offering tentative identification and semiquantitative analysis of contaminants not included in the calibration mixture. Available automation allows increased sample throughput to make use of analytical instrumentation outside of normal work hours.

References

1. <u>Carbon Dioxide</u>, International Society of Beverage Technologists (ISBT) March 2001

Table 1 - T015 Initial Calibration Report

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isopropi Akchol3.2233.2233.0383.0963.1683.3813.1873.471.1-Dichloroethene3.7413.3403.6463.5963.5783.3145.1214.47Dirnethyl Suffide3.4032.8422.8212.8062.4881.9572.7201.7.52Trichhorotifhorethane1.1861.0331.0231.0481.060.9631.0537.01MethyleeCholred2.5852.2072.0832.1012.0771.8782.15510.95Carbon Disulfide8.8457.5206.1256.4696.0445.3376.72218.77Trans-1,2-Dichorethene3.4713.1263.0813.1843.882.8913.1575.97Methyltert-Butyl Ether7.9066.8846.4847.0417.0336.4197.0226.96Vinyl Acetate4.5503.9704.0044.1174.1253.7454.0776.112-Butanone1.2241.0201.6121.2141.1201.1451.1392.661-Dichloroethane3.9293.6903.0673.8623.6883.6183.7643.352Chloroform5.7614.9174.9545.1445.1464.745.1165.7441,1-1*richloroethane3.5283.1023.1663.2753.2823.083.2325.37Berzone8.697.5967.7357.9815.7425.1165.745.3125	Trichlorofluoromethane	6.742	5.961	6.033	6.177	6.327	5.664	6.151	5.94		
1,1-Dichloroethene 3,741 3,380 3,460 3,596 3,578 3,314 3,512 4,47 Dinnchyl Sulfide 3,481 2,842 2,812 2,848 4,367 4,780 6,322 Trichborotfilloorethane 5,272 4,646 4,679 4,887 4,88 4,367 4,780 6,323 Methylene Choride 2,885 2,207 2,083 2,101 2,071 1,878 2,155 10,95 Carbon Disulfide 8,487 7,520 6,125 6,469 6,034 5,373 6,722 8,97 Methyl tert-lavil ther 7,906 6,884 6,448 7,041 7,033 6,419 7,022 6,90 Vinyl Acetate 4,855 4,433 4,397 4,888 4,584 4,241 4,561 5,551 1,-Dichloroethane 1,224 1,202 1,162 1,114 1,110 1,145 1,173 2,666 Hexane 3,767 3,463 3,460 3,457 3,232 3,015 5,454 Chiroothane 5,764 5,432 3,566	Acetone	3.706	3.602	2.809	2.791	2.860	2.687	3.076	14.71		
Dimethyl sulfide3.4032.8422.8212.8062.8881.9272.72017.52Allyl Choride1.1861.0331.0231.0481.0660.9631.0537.01Methylenc Choride1.882.0311.0231.0481.0660.9631.0537.01Methylenc Choride8.8457.5206.1256.4696.0345.3376.72218.77Trans-1.2b/lehrorethene3.7473.1263.0813.1843.1882.3813.1575.571Methylenc Furyl tert-Buryl Ether7.9066.8846.8487.0417.0336.4197.0226.961.p.Dichlorroethane4.5003.9704.0044.1174.1211.1451.1451.1452.Burane1.3763.3823.3763.4773.2532.9663.3727.86Hexne3.2922.892.9503.0693.0572.8253.0155.45Ethyl Acetate3.9293.6663.8223.8683.6183.7643.52Chloroform5.7614.9174.9545.1445.1465.1472.48I.p.1-Tichlorothane5.3335.275.2085.3094.325.311.p.2-Ichloroethane3.5283.1023.1663.2753.2823.0383.2325.371.p.2-Ichloroethane3.5283.1023.1663.2753.2825.0880.5877.861.p.2-Ichloroethane0.6350.577 <td>Isopropyl Alcohol</td> <td>3.223</td> <td></td> <td>3.038</td> <td>3.096</td> <td>3.150</td> <td>3.351</td> <td>3.187</td> <td>3.47</td>	Isopropyl Alcohol	3.223		3.038	3.096	3.150	3.351	3.187	3.47		
Trichlworethane 5.272 4.646 4.679 4.897 4.818 4.367 4.780 6.63 Methylene Choride 2.885 2.097 2.083 2.101 2.077 1.878 2.155 10.95 Carbon Disulfide 3.471 3.126 3.081 3.184 3.184 2.881 2.897 1.877 Methyler-Ruyl Eher 7.906 6.84 6.448 7.041 7.033 6.619 7.022 6.96 Vinyl Acetat 4.855 4.433 4.397 4.888 4.844 4.241 4.561 5.55 1,-Dichloroethane 4.224 1.202 1.162 1.144 1.145 1.145 1.193 2.66 Hexane 3.761 4.332 3.76 3.361 3.618 3.764 3.35 Chioroform 5.761 4.917 4.954 5.144 5.146 5.144 5.141 6.142 1,1-Dichloroethane 5.934 5.227 5.285 5.649 4.760 1,2-	1,1-Dichloroethene	3.741	3.380	3.460	3.596	3.578	3.314	3.512	4.47		
Ally Chloride 1.86 1.033 1.023 1.048 1.066 0.963 1.033 7.01 Methylenc Chloride 8.845 7.520 6.125 6.469 6.034 5.337 6.722 18.77 Trans1.2-Dichlorochtene 3.845 6.323 6.484 3.188 3.188 2.891 3.157 5.977 Methylenc Chloride 4.856 6.434 6.438 7.041 7.033 6.419 7.022 6.96 1/Dichlorocthane 4.850 3.970 4.808 4.584 4.241 4.561 5.55 1/Dichlorocthane 4.850 3.970 4.004 4.117 4.125 3.745 4.077 6.16 Peanone 3.276 3.323 3.376 3.477 3.323 2.966 3.372 7.88 Eisl.2-Dichlorocthane 3.292 3.69 3.663 3.822 3.868 3.618 3.764 3.375 1/L-Tricthorothane 5.345 5.14 5.14 5.14 5.14 5.14 5.14 5.14 5.14 5.14 5.14 5.14 5.14	Dimethyl Sulfide	3.403	2.842	2.821	2.806		1.957		17.52		
Methylene Chloride 2.885 2.207 2.083 2.101 2.077 1.878 2.155 10.95 Carbon Disulfide 3.471 3.126 3.081 3.184 3.184 2.891 3.157 5.77 Wethyl terk tryl Ether 7.06 6.84 6.448 7.041 7.033 6.419 7.022 6.96 Vinyl Acetate 4.855 4.433 4.397 4.888 4.844 4.241 4.561 5.55 JDichloroethane 1.224 1.202 1.162 1.145 1.193 2.66 Lexane 3.76 3.382 3.76 3.471 2.813 3.764 3.352 Chiorofram 5.761 4.917 4.954 5.144 5.144 4.742 5.111 6.917 L-2-Dichloroethane 5.934 5.227 5.326 5.514 5.518 5.074 5.432 5.369 4.762 L-2-Dichloroethane 5.232 7.391 7.922 7.391 7.922 7.391 7.924	Trichlorotrifluoroethane	5.272	4.646	4.679	4.897	4.818	4.367	4.780	6.32		
Carbon Disulfide 8.845 7.520 6.125 6.469 6.034 5.377 6.722 18.77 Methyl tert-Dutyl Etchar 7.906 6.884 6.848 7.041 7.033 6.419 7.022 6.96 Vinyl Accitate 4.855 4.433 4.974 4.117 4.125 3.745 4.077 6.11 Jehntone 1.244 1.202 1.162 1.214 1.120 1.145 1.193 2.66 Hexane 3.767 3.382 3.376 3.477 3.283 3.018 3.74 3.35 Chibrotorthane 3.029 3.679 3.666 3.822 3.088 3.018 3.74 3.35 Chibrotortane 5.934 5.217 5.326 5.514 5.148 5.414 5.418 5.414 5.418 5.438 5.432 5.338 3.232 5.37 1.2-Dichibrorthane 3.528 3.102 3.166 3.275 3.282 3.038 3.322 5.37 12-Jothibrore											
trans.12-bichborochane3.4713.1263.0813.1842.8812.8175.97Wethy Iter-Buryl Ether4.8554.4334.3974.8854.8444.2414.5616.565Vinyl Acetare4.8503.9704.0044.1174.1253.7454.0776.112-Burnone1.2241.2021.1621.2141.2101.1451.1932.661-Dickhorochtane3.2922.8992.9503.0693.0672.8253.0155.45Ethyl Acetare3.2923.6993.6663.8223.8683.6183.7643.35Charoform5.7614.9174.9545.1445.1464.7425.1116.91Terrahydrofaran1.1211.1141.1541.1461.1471.0601.1272.481,1-Trichtorochtane3.5283.1023.1663.2753.2823.0383.3225.511,2-Dichtorochtane3.5283.1023.1663.2753.2823.0383.3225.57Carbon Tetrachoride5.7445.1155.2795.4685.5225.0894.766Carbon Tetrachoride0.6730.5660.5770.5600.5900.5350.5877.88Trichtorochane0.6250.5330.5420.5670.5190.5576.70L2-Dichtoropropane0.6250.5310.5420.6570.5190.5576.70L3-Dichtoropropane0.7840.785		2.585	2.207	2.083	2.101		1.878	2.155	10.95		
Methyl tert-Buryl Ethor 7.906 6.884 6.488 7.041 7.033 6.419 7.022 6.96 Vinyl Accitate 4.500 3.970 4.004 4.117 4.125 3.745 4.077 6.11 2-Butanone 1.224 1.202 1.162 1.214 1.212 3.745 3.072 7.88 cis.1_2-Dichloroethane 3.202 2.899 2.956 3.063 3.087 2.825 3.015 5.45 Ethyl Accitate 3.292 2.899 3.066 3.822 3.868 3.618 3.774 3.253 Chioroform 5.761 4.917 4.954 5.144 5.146 4.742 5.111 6.91 1,1-Trichloroethane 5.528 3.023 3.033 3.232 5.30 4.76 1,2-Dichloroethane 5.538 7.596 7.735 7.981 7.922 7.299 7.904 6.85 2,4-Trinethylpentane 0.633 0.737 0.570 0.580 0.597 0.587 6.7	Carbon Disulfide	8.845	7.520		6.469	6.034		6.722			
Vinyi Acetate 4.855 4.433 4.397 4.858 4.584 4.241 4.561 5.55 1.1-Dichloroethane 1.224 1.202 1.162 1.214 1.103 2.66 Hxane 3.276 3.382 3.376 3.477 3.235 2.966 3.372 7.88 cis-12-Dichloroethene 3.292 2.899 2.950 3.067 2.825 3.015 5.45 Ethyl Acetate 3.292 2.899 2.950 3.066 3.822 3.868 3.618 3.764 3.35 Chloroform 5.761 4.917 4.954 5.144 5.146 4.742 5.111 6.91 1,1-1 Trichloroethane 3.528 3.102 3.166 3.275 3.282 3.038 3.232 5.57 Benzene 8.899 7.536 7.7981 7.922 7.299 7.046 6.85 Carbon Tetrachloride 5.734 5.145 5.177 5.66 5.572 5.885 5.694 4.762											
1,1-Dickhoroethane 4.500 3.970 4.004 4.117 4.125 3.745 4.077 6.11 2-Burtanone 3.776 3.382 3.376 3.471 1.214 1.210 1.145 1.132 Cis-1.2-Dichloroethene 3.292 2.899 2.950 3.069 3.075 2.825 3.015 5.45 Chiroroform 5.761 4.917 4.954 5.144 1.141 1.160 1.127 2.48 1,1-17richloroethane 5.934 5.227 5.326 5.514 5.141 5.843 2.531 1,2-Dichloroethane 5.934 5.227 5.326 5.514 5.143 5.432 5.518 1,2-Dichloroethane 3.528 3.102 3.166 5.275 3.282 3.038 3.323 5.37 Benzene 8.889 7.596 7.735 7.981 5.926 5.668 5.522 5.085 7.88 1,2-Dichloropropane 0.653 0.577 0.580 0.690 0.555 0.567 7.86 1,2-Dichloropropane 0.653 0.737 0.758											
2-Buranne 1.24 1.202 1.162 1.214 1.210 1.145 1.193 2.66 Hexane 3.376 3.382 3.376 3.477 3.235 2.805 3.067 2.825 3.015 5.45 Ethyl Acetate 3.929 3.679 3.666 3.822 3.868 3.618 3.764 3.35 Chloroform 5.761 4.917 4.954 5.144 5.144 1.146 1.147 1.080 1.127 2.48 I.1.J. Trichhorethane 5.934 5.227 5.326 5.514 5.518 5.074 5.432 5.51 I.2.Otichloroethane 3.528 3.102 3.166 3.275 3.282 3.038 3.232 5.57 I.2.Otichloroethane 5.534 5.17 5.86 5.56 5.66 5.77 7.88 7.88 Carbon Tetrachloride 5.734 5.435 5.67 0.519 0.557 6.70 L2.4-Trimethylpentane 0.625 0.533 0.542 0.556 0.567 0.519 0.557 6.70 J.2-Dichloropropane											
Hexne3.7763.3823.3763.4773.2532.9663.3727.88cis-1,2-Dichloroethene3.2922.8992.9503.0673.0673.2823.8683.6183.7643.355Chloroform5.7614.9174.9545.1445.1464.7425.1116.91Tetrahydrofuran1.1211.1141.1541.1461.1471.0801.1272.4881,1-Drichloroethane5.9345.2275.3265.5145.5185.0745.4325.517Benzene8.8897.5967.7357.9817.9227.2997.9046.85Carbon Tetrachloride5.7445.1795.4685.5225.0855.3694.7662,2,4-Trimethylpentane2.6330.7350.7370.5800.5900.5350.5877.88Heptane0.6730.7350.7670.6930.7577.0661.777.0561,2-Dichloropropane0.6250.5330.5420.5660.5670.5190.5576.701,3-Dichloropropene1.1710.9941.1611.0421.0650.9791.0456.641,2-Dichloropropene1.1710.9941.0161.0421.0650.9791.0456.641,2-Dichloropropene1.1710.9941.0161.0421.0650.9791.0456.641,2-Dichloropropene1.1710.9941.0161.0421.0650.9791.0456.64 </td <td>·</td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>	·										
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Ethyl Acetate3.9293.6793.6663.8223.8683.6183.7643.35Chloroform5.7614.9174.9545.1445.1464.7425.1116.91Tetrahydrofuran1.1211.1411.1541.1461.1471.0801.1272.481,1.1-Trichloroethane5.9345.2275.3265.5145.5185.0745.4325.517Benzene8.8897.5967.7357.9817.9227.2997.9046.85Carbon Tetrachloride5.7345.1155.2795.4685.5225.0855.3694.762,2,4-Trimethylpentane2.5382.1902.1742.2142.2682.0422.2387.38Trichloroethene0.6530.7350.7370.7590.7670.6930.7577.0661,2-Dichloropropane0.6620.5330.5420.5660.5670.5190.5576.701,3-Dichloropropane0.6730.7880.8800.8990.9230.9577.061,2-Dichloropropane0.7940.8510.8800.8990.9330.9448.38trans-1,3-Dichloropropene0.7940.8510.8830.6990.9330.9448.38trans-1,3-Dichloropropene0.7850.6830.6930.6460.5117.79Dibromochhane1.3661.1371.1501.1651.2281.1188.341,2-Trichloroethane0.3340.4660.583 </td <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>											
Chiroform 5.761 4.917 4.954 5.144 5.146 4.742 5.111 6.91 Tetrahydrofuran 1.121 1.114 1.154 1.146 1.147 1.080 1.127 2.48 1,1.Trichloroethane 3.528 3.102 3.166 3.275 3.282 3.038 3.232 5.37 Benzene 8.889 7.596 7.735 7.921 7.299 7.904 6.85 Carbon Tetrachloride 5.744 5.115 5.279 5.468 5.522 5.085 5.369 4.76 2,2,4-Trimethylpentane 0.673 0.566 0.577 0.800 0.550 0.587 7.88 Trichloroethane 0.625 0.533 0.737 0.769 0.650 0.519 0.587 6.70 Bromodichloromorpane 0.425 0.533 0.542 0.566 0.577 0.680 0.979 0.948 0.642 Cis-1.3-Dichloropropene 0.797 0.995 0.989 0.923 0.854 0.	-										
Tetrahydrofuran 1.121 1.114 1.154 1.146 1.147 1.080 1.127 2.48 1,1,1-Trichloroethane 5.934 5.227 5.326 5.514 5.518 5.074 5.432 5.511 1,2-Dichloroethane 3.528 3.102 3.166 3.275 3.282 3.038 3.232 5.37 Benzene 8.889 7.596 7.735 7.981 7.922 7.299 7.904 6.85 2,4-Frimethylpentane 2.538 2.190 2.174 2.214 2.268 2.042 2.238 7.38 Trichloroethene 0.663 0.737 0.759 0.767 0.693 0.757 7.86 1,2-Dichloropropane 0.625 0.533 0.542 0.566 0.567 0.519 0.557 6.70 Bromodichloromethane 1.326 1.135 1.142 1.168 1.207 1.045 6.64 4-Methyl-2-pentanone 0.799 0.995 0.980 0.903 0.944 8.38 trans-1,3-Dichloropropene 1.71 0.881 0.880 0.899 0											
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Benzene8.8897.5967.7357.9817.9227.2997.9046.85Carbon Tetrachloride5.7445.1155.2795.4685.5225.0855.3694.76Q.2,4-Trimethylpentane0.6730.5660.5770.5800.5900.5350.5877.88Heptane0.66730.5660.5770.7590.7670.6030.7577.061,2-Dichloropropane0.6250.5330.5420.5560.5670.5190.5576.70Bromodichloromethane1.3261.1351.1421.1681.2071.0861.1777.055cis-1,3-Dichloropropene0.7790.9950.9890.9930.9800.9030.9448.38trans-1,3-Dichloropropene0.9740.8510.6830.6990.7050.6490.7016.521,12-Trichloroethane0.7850.6830.6830.6990.7050.6490.7016.522-Hexanone0.3340.4660.5510.5680.5410.5740.5011.779Dibromochloromethane1.3061.1371.1501.1651.2281.1181.1845.96Tetrachloroethene1.2961.0811.0831.1021.1060.9961.1118.931,2-Dirbromochane2.6672.3932.4102.5122.3072.1653.168.25m-Xylene2.9082.5232.5042.5422.4672.2192.5268.75											
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p-Xylene2.6672.3932.4102.5122.3072.1652.4097.13Styrene2.2101.9361.9401.9881.9651.7611.9677.32o-Xylene2.9992.5442.5532.6062.5602.2742.5899.00Bromoform1.4141.2501.2841.2931.3971.2381.3135.711,1,2,2-Tetrachloroethane1.9201.6711.6481.6751.6641.4631.6748.694-Ethyltoluene3.8403.3633.3363.3243.3302.9733.3618.231,3,5-Trimethylbenzene3.5203.0023.0313.0823.0072.6603.0509.021,2,4-Trimethylbenzene3.4933.0393.0473.0893.0272.6883.0648.361,3-Dichlorobenzene2.6922.4742.5352.5222.6042.4662.5493.371,4-Dichlorobenzene2.3682.0182.0162.0231.9951.7582.0309.601,2-Dichlorobenzene2.2041.8771.8691.8811.8631.6341.8889.641,2,4-Trichlorobenzene2.0091.7781.7161.7431.5611.3881.69912.35	m-Xylene	2.908							8.75		
o-Xylene2.9992.5442.5532.6062.5602.2742.5899.00Bromoform1.4141.2501.2841.2931.3971.2381.3135.711,1,2,2-Tetrachloroethane1.9201.6711.6481.6751.6641.4631.6748.694-Ethyltoluene3.8403.3633.3363.3243.3302.9733.3618.231,3,5-Trimethylbenzene3.5203.0023.0313.0823.0072.6603.0509.021,2,4-Trimethylbenzene3.4933.0393.0473.0893.0272.6883.0648.361,3-Dichlorobenzene2.3592.0262.0052.0191.9991.7562.0279.49Benzyl Chloride2.6922.4742.5352.5222.6042.4662.5493.371,4-Dichlorobenzene2.3682.0182.0162.0231.9951.7582.0309.601,2-Dichlorobenzene2.2041.8771.8691.8811.8631.6341.8889.641,2,4-Trichlorobenzene2.0091.7781.7161.7431.5611.3881.69912.35		2.667	2.393	2.410	2.512	2.307	2.165	2.409	7.13		
Bromoform1.4141.2501.2841.2931.3971.2381.3135.711,1,2,2-Tetrachloroethane1.9201.6711.6481.6751.6641.4631.6748.694-Ethyltoluene3.8403.3633.3363.3243.3302.9733.3618.231,3,5-Trimethylbenzene3.5203.0023.0313.0823.0072.6603.0509.021,2,4-Trimethylbenzene3.4933.0393.0473.0893.0272.6883.0648.361,3-Dichlorobenzene2.3592.0262.0052.0191.9991.7562.0279.49Benzyl Chloride2.6922.4742.5352.5222.6042.4662.5493.371,4-Dichlorobenzene2.3682.0162.0231.9951.7582.0309.601,2-Dichlorobenzene2.2041.8771.8691.8811.8631.6341.8889.641,2,4-Trichlorobenzene2.0091.7781.7161.7431.5611.3881.69912.35	Styrene	2.210	1.936	1.940	1.988	1.965	1.761	1.967	7.32		
1,1,2,2-Tetrachloroethane1.9201.6711.6481.6751.6641.4631.6748.694-Ethyltoluene3.8403.3633.3363.3243.3302.9733.3618.231,3,5-Trimethylbenzene3.5203.0023.0313.0823.0072.6603.0509.021,2,4-Trimethylbenzene3.4933.0393.0473.0893.0272.6883.0648.361,3-Dichlorobenzene2.3592.0262.0052.0191.9991.7562.0279.49Benzyl Chloride2.6922.4742.5352.5222.6042.4662.5493.371,4-Dichlorobenzene2.3682.0182.0162.0231.9951.7582.0309.601,2-Dichlorobenzene2.2041.8771.8691.8811.8631.6341.8889.641,2,4-Trichlorobenzene2.0091.7781.7161.7431.5611.3881.69912.35	o-Xylene	2.999	2.544	2.553	2.606	2.560	2.274	2.589	9.00		
4-Ethyltoluene3.8403.3633.3363.3243.3302.9733.3618.231,3,5-Trimethylbenzene3.5203.0023.0313.0823.0072.6603.0509.021,2,4-Trimethylbenzene3.4933.0393.0473.0893.0272.6883.0648.361,3-Dichlorobenzene2.3592.0262.0052.0191.9991.7562.0279.49Benzyl Chloride2.6922.4742.5352.5222.6042.4662.5493.371,4-Dichlorobenzene2.3682.0182.0162.0231.9951.7582.0309.601,2-Dichlorobenzene2.2041.8771.8691.8811.8631.6341.8889.641,2,4-Trichlorobenzene2.0091.7781.7161.7431.5611.3881.69912.35	Bromoform	1.414	1.250	1.284	1.293	1.397	1.238	1.313	5.71		
1,3,5-Trimethylbenzene3.5203.0023.0313.0823.0072.6603.0509.021,2,4-Trimethylbenzene3.4933.0393.0473.0893.0272.6883.0648.361,3-Dichlorobenzene2.3592.0262.0052.0191.9991.7562.0279.49Benzyl Chloride2.6922.4742.5352.5222.6042.4662.5493.371,4-Dichlorobenzene2.3682.0182.0162.0231.9951.7582.0309.601,2-Dichlorobenzene2.2041.8771.8691.8811.8631.6341.8889.641,2,4-Trichlorobenzene2.0091.7781.7161.7431.5611.3881.69912.35	1,1,2,2-Tetrachloroethane	1.920	1.671	1.648	1.675	1.664	1.463	1.674	8.69		
1,2,4-Trimethylbenzene3.4933.0393.0473.0893.0272.6883.0648.361,3-Dichlorobenzene2.3592.0262.0052.0191.9991.7562.0279.49Benzyl Chloride2.6922.4742.5352.5222.6042.4662.5493.371,4-Dichlorobenzene2.3682.0182.0162.0231.9951.7582.0309.601,2-Dichlorobenzene2.2041.8771.8691.8811.8631.6341.8889.641,2,4-Trichlorobenzene2.0091.7781.7161.7431.5611.3881.69912.35		3.840	3.363		3.324		2.973		8.23		
1,2,4-Trimethylbenzene3.4933.0393.0473.0893.0272.6883.0648.361,3-Dichlorobenzene2.3592.0262.0052.0191.9991.7562.0279.49Benzyl Chloride2.6922.4742.5352.5222.6042.4662.5493.371,4-Dichlorobenzene2.3682.0182.0162.0231.9951.7582.0309.601,2-Dichlorobenzene2.2041.8771.8691.8811.8631.6341.8889.641,2,4-Trichlorobenzene2.0091.7781.7161.7431.5611.3881.69912.35	1,3,5-Trimethylbenzene	3.520	3.002	3.031	3.082	3.007	2.660	3.050	9.02		
Benzyl Chloride2.6922.4742.5352.5222.6042.4662.5493.371,4-Dichlorobenzene2.3682.0182.0162.0231.9951.7582.0309.601,2-Dichlorobenzene2.2041.8771.8691.8811.8631.6341.8889.641,2,4-Trichlorobenzene2.0091.7781.7161.7431.5611.3881.69912.35		3.493	3.039	3.047	3.089	3.027	2.688	3.064	8.36		
1,4-Dichlorobenzene2.3682.0182.0162.0231.9951.7582.0309.601,2-Dichlorobenzene2.2041.8771.8691.8811.8631.6341.8889.641,2,4-Trichlorobenzene2.0091.7781.7161.7431.5611.3881.69912.35	1,3-Dichlorobenzene	2.359	2.026	2.005	2.019	1.999	1.756	2.027	9.49		
1,2-Dichlorobenzene2.2041.8771.8691.8811.8631.6341.8889.641,2,4-Trichlorobenzene2.0091.7781.7161.7431.5611.3881.69912.35	Benzyl Chloride	2.692	2.474	2.535	2.522	2.604	2.466	2.549	3.37		
1,2,4-Trichlorobenzene 2.009 1.778 1.716 1.743 1.561 1.388 1.699 12.35	1,4-Dichlorobenzene		2.018		2.023	1.995	1.758		9.60		
	1,2-Dichlorobenzene		1.877		1.881						
Hexachlorobutadiene 1.809 1.580 1.562 1.476 1.233 0.992 1.442 19.99	1,2,4-Trichlorobenzene		1.778	1.716	1.743	1.561		1.699			
	Hexachlorobutadiene	1.809	1.580	1.562	1.476	1.233	0.992	1.442	19.99		

Table 2 - T015 Detection Limit Study

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Compound	Level 1	Level 2	Level 3	Responses Level 4	(ppbv) Level 5	Level 6	Level 7	Ave Resp	SD	IDL
Compound	Level I	Leverz	Levers	Level 4	Levero	Levero	Lever	Avencesp	50	IDL
Propene	0.12	0.10	0.08	0.11	0.10	0.09	0.09	0.10	0.01	0.04
Dichlorodifluoroethane	0.11	0.09	0.08	0.10	0.09	0.08	0.07	0.09	0.01	0.04
Chloromethane	0.11	0.12	0.10	0.12	0.08	0.10	0.10	0.10	0.01	0.04
Dichlorotetrafluourethar	ne 0.10	0.09	0.08	0.11	0.10	0.08	0.08	0.09	0.01	0.04
Vinyl Chloride	0.11	0.09	0.08	0.11	0.08	0.08	0.08	0.09	0.01	0.04
1,3-Butadiene	0.10	0.09	0.04	0.10	0.08	0.08	0.08	0.08	0.02	0.06
Bromoethane	0.11	0.08	0.07	0.10	0.07	0.08	0.07	0.08	0.02	0.05
Chloroethane	0.10	0.09	0.07	0.10	0.07	0.08	0.08	0.08	0.01	0.04
Bromoethene	0.09	0.08	0.07	0.08	0.05	0.07	0.07	0.07	0.01	0.04
Trichlorofluoromethane		0.08	0.07	0.09	0.07	0.07	0.06	0.08	0.03	0.08
Acetone	0.10	0.12	0.10	0.13	0.07	0.10	0.10	0.10	0.02	0.06
1,1-Dichloroethene	0.11	0.09	0.08	0.11	0.10	0.08	0.07	0.09	0.02	0.05
Trichlorotrifluoroethane		0.09	0.08	0.11	0.08	0.09	0.08	0.09	0.02	0.05
Allyl Chloride	0.20 0.29	0.10	0.09	0.12	0.08	0.10	0.09	0.11	0.04	0.12
Methylene Chloride Carbon Disulfide	0.29	0.25 0.15	0.24 0.25	0.20 0.27	0.09 0.20	0.20 0.24	0.13 0.24	0.20 0.21	0.07 0.06	0.21 0.18
trans-1,2-Dichloroethen		0.15	0.25	0.27	0.20	0.24	0.24	0.21	0.06	0.18
Methyl tert-Butyl Ether	0.11	0.09	0.08	0.12	0.24	0.09	0.08	0.12	0.00	0.05
Vinyl Acetate	0.11	0.09	0.08	0.07	0.08	0.09	0.08	0.09	0.02	0.03
1,1-Dichloroethane	0.11	0.09	0.08	0.07	0.08	0.09	0.08	0.09	0.01	0.04
2-Butanone	0.15	0.00	0.09	0.11	0.08	0.00	0.08	0.10	0.02	0.07
Hexane	0.10	0.09	0.07	0.10	0.08	0.08	0.08	0.09	0.01	0.03
cis-1,2-Dichloroethene	0.10	0.09	0.08	0.11	0.08	0.08	0.08	0.09	0.01	0.04
Ethyl Acetate	0.11	0.10	0.08	0.10	0.08	0.08	0.08	0.09	0.01	0.04
Chloroform	0.10	0.09	0.08	0.11	0.08	0.08	0.07	0.09	0.01	0.04
Tetrahydrofuran	0.10	0.12	0.08	0.11	0.08	0.10	0.09	0.10	0.01	0.04
1,1,1-Trichloroethane	0.10	0.09	0.07	0.10	0.07	0.08	0.07	0.08	0.01	0.04
1,2-Dichloroethane	0.12	0.09	0.07	0.10	0.07	0.07	0.07	0.08	0.02	0.06
Benzene	0.11	0.10	0.08	0.12	0.09	0.09	0.09	0.10	0.01	0.04
Carbon Tetrachloride	0.11	0.09	0.08	0.10	0.07	0.08	0.07	0.09	0.02	0.05
Cyclohexane	0.11	0.10	0.09	0.12	0.09	0.09	0.09	0.10	0.01	0.04
2,2,4-Trimethylpentane	0.10	0.09	0.08	0.11	0.08	0.08	0.08	0.09	0.01	0.04
Heptane	0.11	0.09	0.08	0.11	0.07	0.08	0.07	0.09	0.02	0.05
Trichloroethene	0.11	0.09	0.08	0.11	0.08	0.08	0.08	0.09	0.01	0.04
1,2-Dichloropropane	0.10	0.09	0.08	0.11	0.08	0.08	0.08	0.09	0.01	0.04
Bromodichloromethane		0.08	0.07	0.10	0.07	0.07	0.07	0.08	0.01	0.04
cis-1,3-Dichloropropene		0.09	0.08	0.11	0.08	0.08	0.08	0.09	0.01	0.03
4-Methyl-2-pentanone	0.10	0.11	0.08	0.08	0.09	0.10	0.09	0.09	0.01	0.03
trans-1,3-Dichloroprope		0.09	0.08	0.11	0.07	0.08	0.07	0.09	0.02	0.05
Toluene 1,1,2-Trichloroethane	0.11 0.10	0.10 0.09	0.08 0.08	0.12 0.11	0.08 0.08	0.09 0.09	0.08 0.08	0.09 0.09	0.02 0.01	0.05 0.03
2-Hexanone	0.10	0.09	0.08	0.10	0.08	0.09	0.08	0.09	0.01	0.03
Dibromochloromethane		0.12	0.07	0.10	0.07	0.07	0.07	0.08	0.01	0.05
Tetrachloroethene	0.11	0.10	0.08	0.12	0.08	0.07	0.07	0.09	0.02	0.05
1,2-Dibromoethane	0.11	0.09	0.08	0.12	0.08	0.08	0.08	0.09	0.01	0.00
Chlorobenzene	0.11	0.10	0.08	0.12	0.08	0.09	0.08	0.09	0.02	0.05
Ethylbenzene	0.11	0.09	0.08	0.12	0.08	0.09	0.09	0.09	0.02	0.05
m-Xylene	0.10	0.09	0.08	0.10	0.09	0.08	0.07	0.09	0.01	0.03
p-Xylene	0.11	0.10	0.08	0.11	0.08	0.08	0.08	0.09	0.01	0.04
Styrene	0.11	0.10	0.08	0.11	0.08	0.08	0.08	0.09	0.01	0.04
o-Xylene	0.11	0.09	0.08	0.12	0.08	0.09	0.08	0.09	0.02	0.05
Bromoform	0.10	0.10	0.08	0.10	0.07	0.08	0.07	0.09	0.01	0.04
1,1,2,2-Tetrachloroetha	ne0.11	0.11	0.09	0.12	0.08	0.09	0.08	0.10	0.02	0.05
4-Ethyltoluene	0.12	0.10	0.08	0.12	0.09	0.09	0.08	0.10	0.02	0.05
1,3,5-Trimethylbenzene		0.11	0.09	0.12	0.08	0.09	0.08	0.10	0.02	0.05
1,2,4-Trimethylbenzene		0.11	0.09	0.12	0.09	0.09	0.09	0.10	0.01	0.04
1,3-Dichlorobenzene	0.13	0.10	0.09	0.12	0.09	0.09	0.09	0.10	0.02	0.05
Benzyl Chloride	0.11	0.11	0.09	0.11	0.08	0.08	0.07	0.09	0.02	0.05
1,4-Dichlorobenzene	0.12	0.11	0.08	0.12	0.09	0.09	0.09	0.10	0.02	0.05

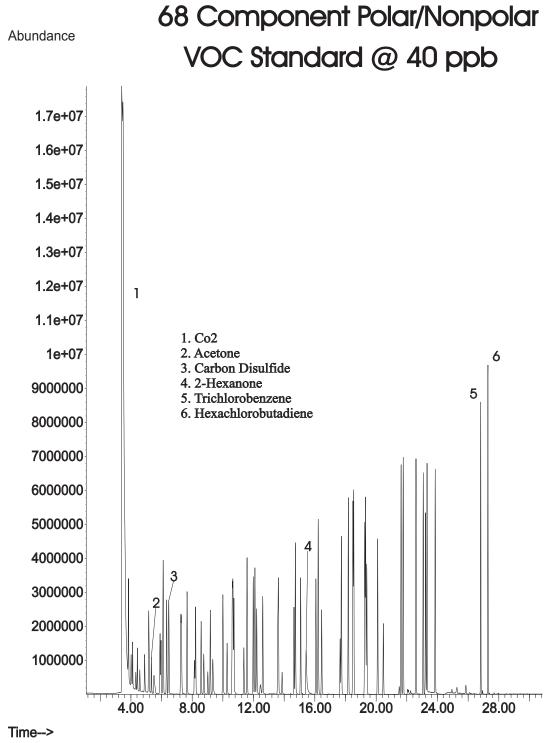


Figure 1

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