

# Analysis of VOC by P&T and GC/MS using nitrogen as a purge gas

Using Nitrogen as a purge gas



## Introduction

Helium has been successfully used as a purge gas for volatile organic analysis for many years because it is inert and works extremely well for P&T concentration. Traditionally, VOCARB traps or mixed bed traps containing Tenax, silica gel, and carbon molecular sieve (CMS) have been used. Despite the success of this pairing, Helium (He) is a non-renewable resource and supplies will eventually run out. Decreasing availability has caused significant price increases and long lead times for He cylinders, prompting many labs to look for an alternative source.

Nitrogen (N<sub>2</sub>) is inert, plentiful, and relatively inexpensive making it a reasonable alternative purge gas. However, we have found that the VOCARB and mixed bed traps do not work as well when using N<sub>2</sub> instead of He as a purge gas. Increased tailing has been observed as well as more compounds having high Relative Standard Deviation (RSDs) in the calibrations using these traps. It is best to avoid manual integrations because of tailing as well as having the need for linear regression when calibration RSDs are too high.

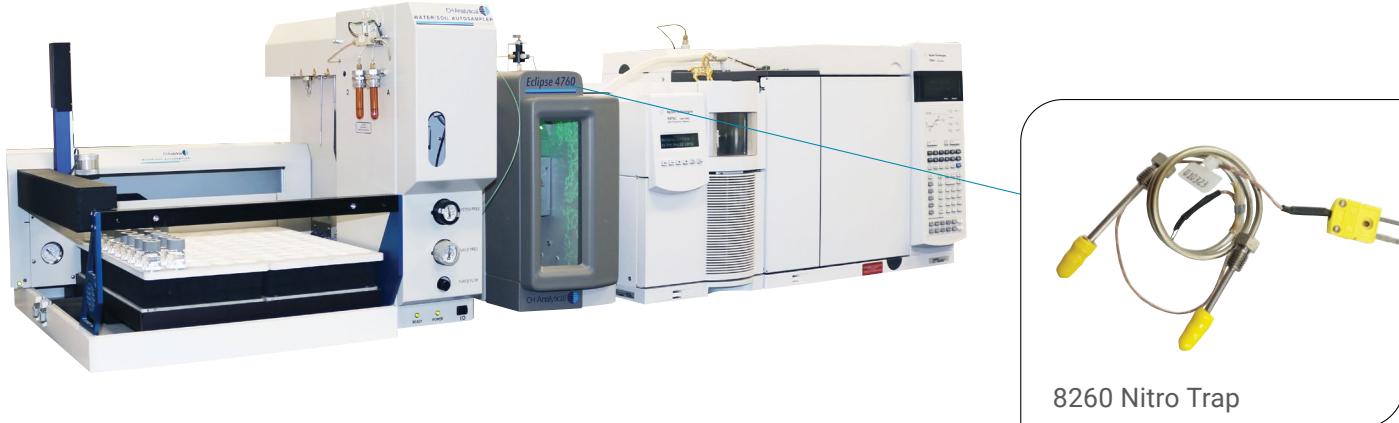
After exhaustive testing it was found that the silica gel in the #10 trap absorbs N<sub>2</sub> during purge and during desorb this causes erratic behavior within the GC inlet. This results in a 30-40% drop in response when switching to N<sub>2</sub> as a purge gas so that the lower concentrations in the calibration are less responsive and some compounds have a higher %RSD. Countless sorbents and configurations were tested and the effort was worthwhile. The 8260 Nitro Trap was specifically designed for N<sub>2</sub> purge which gives similar excellent results as the #10 with He.

This application notes presents data using EPA Method 8260D. Calibrations and QC using the #10 trap with He purge and 8260 Nitro Trap with N<sub>2</sub> purge are presented.

## Methodology

EPA Method 8260D was used as guidance for testing. The only changes labs will need to make to their current method are in the P&T temperature parameters. GC/MS parameters can remain the same. As with the #10 trap, the 8260 Nitro Trap does not require a dry purge for moisture removal which keeps the cycle time fast. There is no need for the lab to change current GC/MS parameters or do method development. An OI Analytical 4760 P&T was used for sample concentration and a 7890A/5975C GC/MS was used for chromatographic separation and detection.

A 4100 Sample Processor was used to automate the analysis. Please see Table 1 for instrument parameters. A multipoint calibration from 1 ppb to 200 ppb was analyzed for most compounds. Method Detection Limit (MDL) studies and Initial Demonstrations of Proficiency (IDC) were analyzed. Calibrations and QA/QC samples were analyzed in both water and soil mode.



**Figure 1.** 4100, 4760, 7890A/5975C

**Table 1.** Instrument Parameters

Purge-and-Trap	Eclipse 4760 P&T Sample Concentrator		Autosampler	4100 Water/Soil Sample Processor
Trap	#10 Trap or 8260 Nitro Trap		System Gas	Zero grade Nitrogen
Purge gas	Zero grade Helium or Nitrogen at 40 mL/min		Purge Gas	Zero grade Helium or Nitrogen
Purge time	11 min		LV20 pressure	8.0 psi
Sparge mount temperature	45 °C		Loop-based time settings	Default
Sample temperature (purge)	45 °C		Rinse water	80 °C
Sample temperature (bake)	45 °C		Soil sample transfer	150 °C
Desorb time	0.5 min		Soil oven	150 °C
Bake time	6 min		Soil lift station	45 °C
Trap temperature	Ambient during purge 180 °C (#10) or 210 °C (8260 Nitro) during desorb pre-heat  190 °C (#10) or 220 °C (8260 Nitro) during desorb  210 °C (#10) or 230 °C (8260 Nitro) during bake		Gas Chromatograph	Agilent 7890A
Water management	120 °C during purge Ambient during desorb 240 °C during bake		Column	Restek Rtx-VMS 30 meter, 0.25 mm ID, 1.4 µm df
Transfer line temperature	140 °C		Carrier gas	Zero grade Helium
Six-port valve temperature	140 °C		Inlet temperature	240 °C
Autosampler	4100 Sample Processor Methods		Inlet liner	Agilent Ultra Inert, 1 mm straight taper
Sample type	Waters only	Soils only	Column flow rate	0.8 mL/min
Needle rinses	1	0	Split ratio	125:1
SAM A (µL)	5	5	Oven program	Hold at 40 °C for 1.5 min 18 °C/minute to 180 °C 40 °C/minute to 220 °C Hold at 220 °C for 4 min Total GC run is 15.75 min
SAM B (µL)	0	0		
SAM C (µL)	0	0		
SAM D (µL)	0	0		
Purge time (min)	11.0	11.0		
Desorb time (min)	0.5	0.5		
P&T rinses	3	2		
Rinse water	Hot	Hot		
Water stir rime (min)	0.0	N/A		
Water settle time (sec)	5	N/A		
Soil add water to vial (#loops)	N/A	1		
Soil pre-heat stir (min)	N/A	0.5		
Soil pre-heat/purge temp °C	N/A	45		
Soil stir during purge	N/A	Yes		



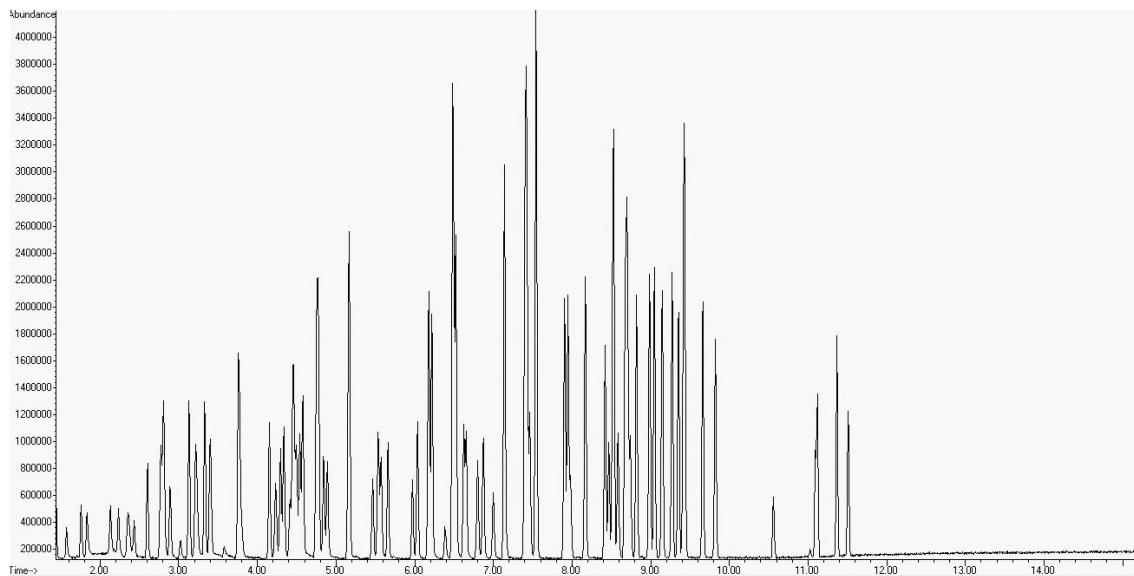




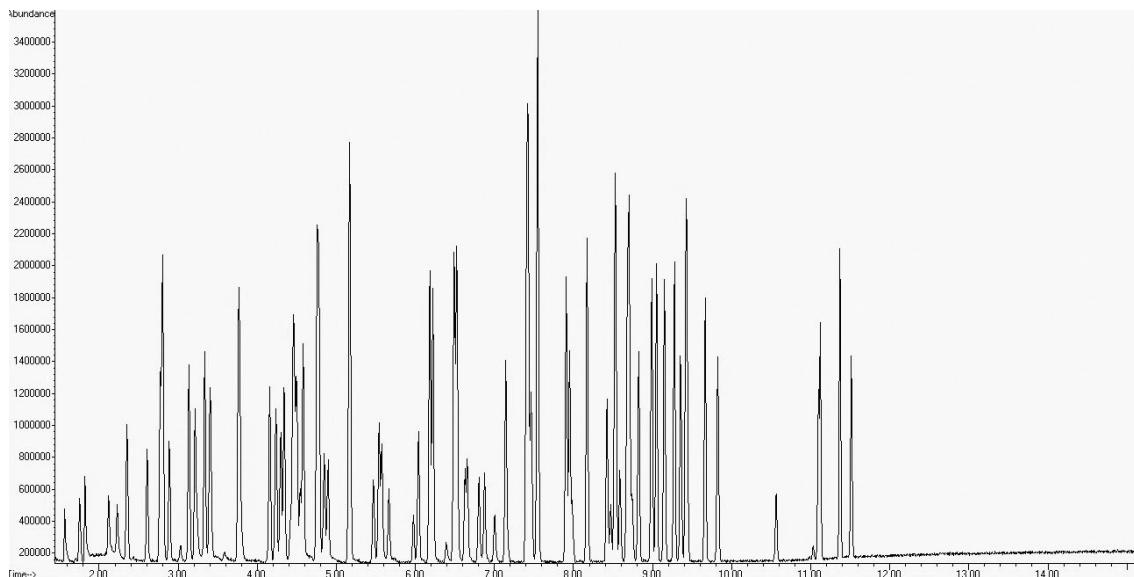


Analyte	Compound	#10 MDL % Recovery	#10 MDL (ppb)	#10 IDC % Recovery	#10 IDC % RSD	8260 Nitro MDL % Recovery	8260 Nitro MDL (ppb)	8260 Nitro IDC % Recovery	8260 Nitro IDC % RSD
79	1,2,4-Trimethylbenzene	91.0	0.09	99.9	1.25	92.3	0.13	101	2.06
80	sec-Butylbenzene	89.3	0.11	97.1	1.74	85.5	0.10	106	2.16
81	p-Isopropyltoluene	89.8	0.08	98.3	0.77	86.3	0.07	104	2.09
82	1,3-Dichlorobenzene	100	0.11	101	1.31	96.0	0.18	98.1	0.78
83	1,4-Dichlorobenzene	109	0.15	100	1.01	110	0.16	99.6	0.82
84	n-Butylbenzene	93.5	0.06	97.5	2.15	97.0	0.14	100	1.67
85	1,2-Dichlorobenzene	102	0.08	99.8	1.59	101	0.06	100	1.35
86	1,2-Dibromo-3-chloropropane	88.0	0.22	98.1	2.28	88.3	0.19	102	3.09
87	Hexachlorobutadiene	99.8	0.27	91.1	3.80	96.3	0.27	98.6	1.95
88	1,2,4-Trichlorobenzene	92.8	0.12	91.0	3.33	110	0.16	98.8	2.35
89	Naphthalene	94.5	0.08	93.7	2.11	101	0.09	102	1.93
90	1,2,3-Trichlorobenzene	98.0	0.13	90.1	1.76	107	0.17	99.8	2.64

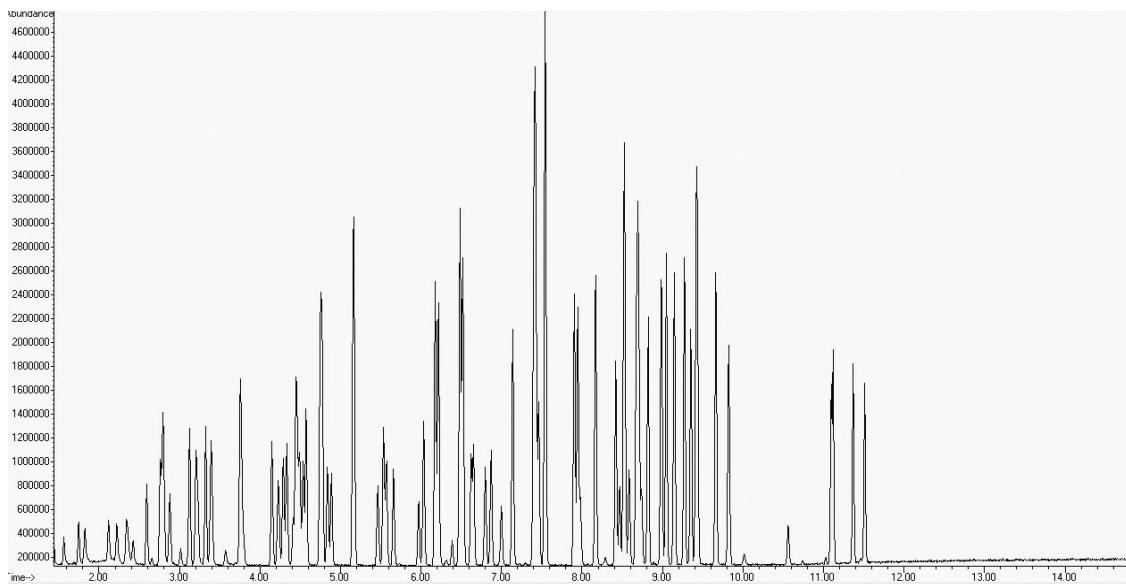
**Figure 1.** 50 ppb Water Standard Using #10 Trap and Helium



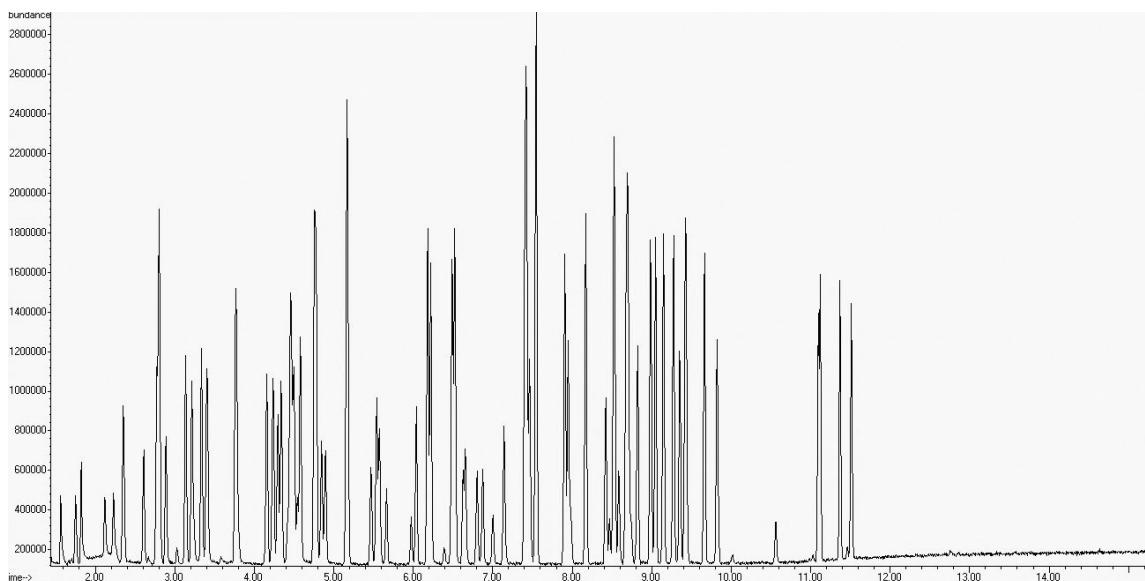
**Figure 2.** 50 ppb Soil Standard Using #10 Trap and Helium



**Figure 3.** 50 ppb Water Standard Using 8260 Nitro Trap and Nitrogen



**Figure 4.** 50 ppb Soil Standard Using 8260 Nitro Trap and Nitrogen



## Conclusion

The results demonstrate that the 8260 Nitro Trap delivers acceptable performance when using Nitrogen as a purge gas without compromising data quality.