

Analysis of Oxygenates in Environmental Water Samples utilizing the EST Encon Purge and Trap Concentrator

Jeff Sheriff – Application Specialist

Abstract

A Purge and Trap system comprised of an EST Encon Concentrator and a Centurion Autosampler was interfaced to a 6890/5793 GC/MS to analyze Oxygenates in water along with 92 other common Volatile Organic Compounds. Data showing the linearity, accuracy, and precision of the Oxygenates will be presented.



Keywords

Oxygenates, MTBE, EPA 8260B, EST Analytical, Encon, VOC, Environmental, Purge and Trap, Concentrator, Underground Storage Tank

Introduction

Hydrocarbon compounds containing 1 or more Oxygen atoms which are added to fuels to increase the Oxygen content are collectively referred to as Oxygenates. This increase in Oxygen content allow motor fuels to burn more cleanly thereby reducing pollution. Methyl Tertiary Butyl Ether (MTBE), a common Oxygenate has been blended into motor gasoline since 1979 to replace the use of lead. Unfortunately, as a result of MTBE and the other Oxygenates being very soluble in water coupled with the fact that they do not readily breakdown there are an increasing number of Oxygenate detections found in ground water and drinking water caused from leaking underground storage tanks or spills. As a result of this contamination more samples are analyzed for Oxygenate compounds than ever before.

Discussion

Since Oxygenates are extremely polar compounds they are considered to be “poor purging compounds”. Modifications to the typical Purge and Trap system parameters such as increased flow rate, increased purge temperature and increased sample size can be made to improve the response of these compounds. Another modification to improve analyte response is to add Sodium Chloride (NaCl) to the sample. However, to remain productive and competitive most Environmental Laboratories chose to analyze for these Oxygenate compounds with the same Purge and Trap system used for the analysis of the routine list of Target analytes found in US EPA Method 8260B. The purpose of this paper is to show the typical results expected for the Oxygenate compounds when using typical Purge and Trap parameters.

Experimental

The EST Centurion Autosampler was interfaced to the EST Encon Purge and Trap Concentrator. The Encon heated transfer line was connected to the GC injection port carrier line with a low dead volume connecting union. To reduce the injection port dead volume a 1mm I.D. liner was installed to improve the resolution of the early eluting gases. Five common Oxygenate compounds will be analyzed in accordance to US EPA 8260B/5030 to illustrate the system’s ability to analyze Oxygenate compounds at low level concentrations utilizing typical Purge and Trap conditions. Ethanol even though considered to be a compound inappropriate to be analyzed by 8260B/5030 will also be studied. Instrument conditions used for this work are listed in Tables 1 and 2.

Table 1. Purge and Trap Conditions

Purge & Trap Concentrator	EST Encon
Trap	Vocarb 3000 Type “K” Carbopack B Carboxen 1000 Carboxen 1001
Sample Size	5 ml
Purge Temp	Ambient
Purge Rate	40 ml/min 4 psi TBC
Purge Time	11 minutes
Dry Purge Time	2 minutes
Dry Purge Temp	Ambient
Desorb Preheat	260 degrees C
Desorb Temp	265 degrees C
Desorb Time	2 minutes
Moisture Reduction	Purge-40 degrees C
Trap Temp	Bake- 260 degrees C
Line Temp	130 degrees C
Valve Temp	130 degrees C
Bake Temp	270 degrees C
Bake Time	10 minutes
Vial Autosampler	EST Centurion
Internal Standard	Fluorobenzene 10 ug/L

Table 2. GC/MSD Conditions

GC	Agilent 6890
Inlet	EPC S/SS
Mode	Split
Inlet Temperature	180 degrees C
Split Ratio	50:1
Split Flow	30.0 ml/min
Oven Temp Program	
Initial Temperature	45 degrees C
Initial Time	1.00 minute
Ramp Rate	18.0 degrees C/minute
Final Temperature	220 degrees C
Column	DB-624 20 meters 0.18 mm I.D. 1.00 Film Thickness
Carrier Gas	Helium UHP
Column Flow	0.6 ml/min
Linear Velocity	31 cm/sec
Mode	Constant Flow
MSD	Agilent 5973
EM Voltage	BFB.U + 100 volts
Solvent Delay	0.7 minute
Mass Range	35-265 amu
Threshold	150
Sampling Rate	3
Quad Temperature	150 degrees C
Source Temperature	230 degrees C
Aux Temperature	200 degrees C

Table 3. QA/QC DATA

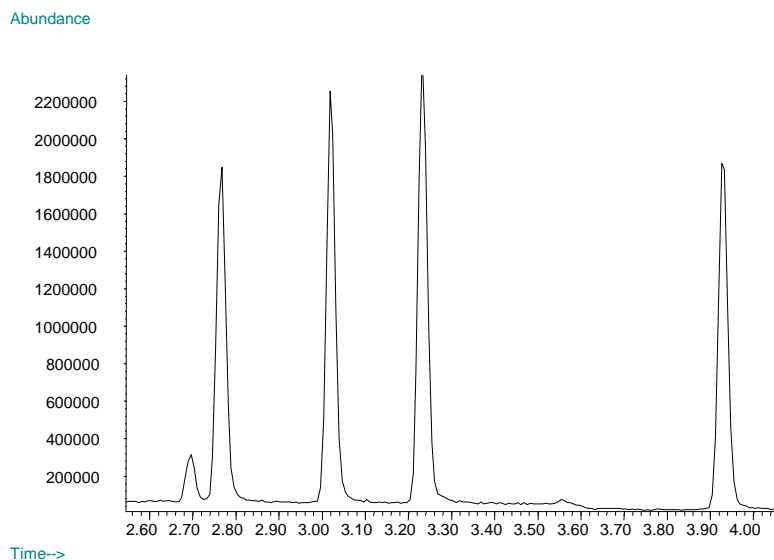
COMPOUND NAME	CALIBRATION RANGE (PPB)	AVG RF	% RSD	MDL
Methyl Tertiary Butyl Ether MTBE	1 – 100	1.647	4.35	0.149
Tertiary Amyl Methyl Ether TAME	1 – 100	1.475	5.12	0.330
Tertiary Butyl Ether TBA	5 – 500	0.070	13.2	1.041
Di-Isopropyl Ether DIPE	1 – 100	1.524	7.19	0.096
Ethyl Tertiary Butyl Ether ETBE	1 – 100	1.800	3.94	0.215
Ethyl Alcohol Ethanol	40 - 4000	0.089	11.1	1.33

Conclusions

The EST Encon, designed for the production Environmental Laboratory, is a reliable Purge & Trap Concentrator which can be used to successfully perform low level Oxygenate analysis without changing the typical Purge and Trap parameters used for routine 8260B work. All system performance and quality assurance criteria can be easily and routinely met. In addition to its superior analytical performance, the EST Encon also has a number of unique user features such as the GC and On-trap injection ports for easy trouble-shooting and the optional foam sensor.

Results

To demonstrate the system's linearity an Initial Calibration curve was constructed consisting of 7 levels. A Method Detection Limit study was then undertaken to determine the system's accuracy and precision by analyzing 7 replicates at a concentration of 1 ug/L. The Calibration Range, %RSD of Initial Calibration Curve, Average Response Factor, and calculated MDL for each compound are outlined in Table 3.

**Figure 1. Oxygenate TIC – 50 ppb**

503 Commercial Drive
Fairfield, Ohio 45014
Phone: 800-283-3510
Email: est@estanalytical.com
Web: www.estanalytical.com