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Analysis of Low Level Volatile Organic Compounds in Air

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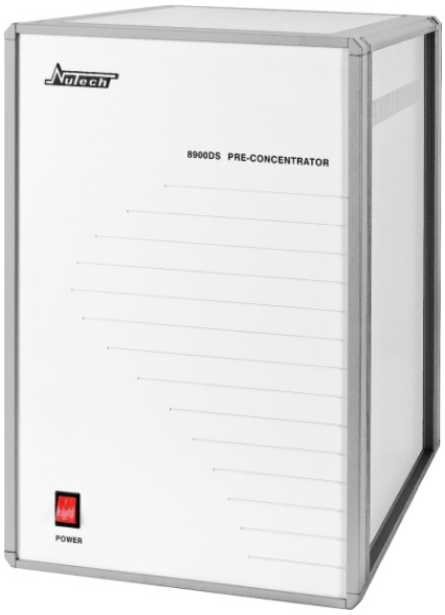
Introduction

The New Jersey Department of Environmental Protection (NJDEP) developed a new low level TO-15 method in order to address the needs of several programs. In comparing the NJDEP method and the United States Environmental Protection Agency (USEPA) TO-15 method it can be seen that the new method provides for additional quality control requirements and lower reporting limits. This study will explore low level TO-15 analysis and the requirements of the NJDEP method.

Discussion

Volatile organic compounds are often present in soils and ground water. These compounds have the potential to migrate from surface water and soil into the surrounding structures. This process is called vapor intrusion and refers to the indoor air quality of area buildings.



The NJDEP method for indoor air quality demands a 0.2ppbv detection limit for a majority of the compounds listed in the method. In order to detect the volatile organics down to such a low level, the pre-concentrator needs to be efficient at trapping the compounds. The 8900 pre-concentrator uses a three stage trapping system in order to trap the volatile compounds. The system also uses cryofocusing for volatile compound concentration prior to injection. Finally, the 8900 pre-concentrator has a unique feature that utilizes nitrogen gas to heat the cryofocuser before injection. This feature ensures rapid heating and cooling of the cryofocuser thus aiding moisture control, compound transfer to the GC and compound peak shape. This poster will demonstrate the effectiveness of the 8900 pre-concentrator for the NJDEP indoor air quality method.

Experimental

The sampling system used for this study was the 8900 pre-concentrator. The pre-concentrator was coupled to a Shimadzu QP 2010 SE GCMS analytical system. The transfer line of the pre-concentrator was configured directly to the Restek Rtx-5 60m x 0.32mm x 1.5um column in the GC. The pre-concentrator and GCMS parameters are listed in Tables 1 and 2 respectively.

8900 pre-concentrator Experimental Parameters	
Trap 1	
Cooling Temp.	-150°C
Preheat Temp.	10°C
Preheat Time	10 sec.
Desorb Temp.	20°C
Desorb Flow	10ml/min
Desorb Time	240 sec.
Bakeout Temp.	160°C
Flush Flow	100ml/min
Flush Time	10sec
Sweep Flow	100ml/min
Sweep Time	30sec
Timeout	15min.
Temp. Target Range	10°C
Stable Time	2 sec.
Enable Cooling w/He	no
Trap 2	
Cooling Temp.	-20°C
Desorb Temp.	180°C
Desorb Time	30 sec.
Bakeout Temp.	190°C
Bakeout Time	360 sec.
Timeout	15 min.
Temp. Target Range	10°C
Stable Time	2 sec.
Enable Cooling w/He	no
Focuser	
Cooling Temp.	-165°C
Inject Time	30 sec.
Timeout	15 min.
Temp. Target Range	10°C
Stable Time	30 sec.
Internal Standard	
Purge Flow	20ml/min
Purge Time	30 sec.
Volume	50ml
ISTD Flow	100ml/min
Sample	
Purge Flow	20ml/min
Purge Time	30 sec.
Sample Flow	100ml/min
GC	
GC Remote Start	yes
GC Run Time	28 min.
Flush Sample Line	no
GC Ready	yes
GC Ready Timeout	10 min.
Idle	
Cryotrap 1	100°C
Transfer Line	125°C
Valve Oven	125°C
Cryotrap 2	100°C
Sample Line	100°C
Sample Oven	100°C
Auxiliary	60°C

Table 1: 8900 pre-concentrator Experimental Parameters

GC/MS	Shimadzu QP2010 SE
Injection Mode	Split
Injection Temp.	100ºC
Flow Control mode	Linear Velocity
Pressure	41.8 kPa
Total Flow	25.3ml/min
Column Flow	1.20ml/min
Linear Velocity	27.9 cm/sec
Split Ratio	20:1
Purge Flow	0.0 ml/min
Column	Rtxi-5 60m x 0.32mm I.D. 1.5µm film thickness
Oven Temp. Program	35ºC hold for 1 min, ramp 10ºC/min to 220ºC, hold for 4.5 min
Ion Source Temp.	185ºC
Interface Temp.	180ºC
Solvent Cut Time	1.5 min
Event Time	0.20 sec
ACQ Mode	Scan
Scan Speed	1250
Start m/z	35
End m/z	265

Table 2: GC/MS Experimental Parameters

Standards and certified summa cans were acquired from Restek. The summa cans were cleaned using the 2100B Canister Cleaning System. The IS was diluted to 20ppb and a 50ml aliquot of this standard was used in order to hold the IS and surrogate levels to a constant 5ppb. The TO-15 standard was diluted to 20ppb and to 1ppb respectively. Next, a series of standard volumes of these dilutions were used for the calibration curve. Refer to Table 3.

Calibration Curve		
Standard	Volume	Calibration Level
1ppb	40ml	0.2ppb
1ppb	100ml	0.5ppb
1ppb	200ml	1ppb
1ppb	400ml	2ppb
20ppb	20ml	2.5ppb
20ppb	50ml	5ppb
20ppb	100ml	10ppb
20ppb	200ml	20ppb
20ppb	400ml	40ppb

Table 3: Calibration Curve

After the calibration curve was established, a series of seven replicate 0.2ppb standards were run in order to establish the minimum detection limit (MDL) of the system. In order to determine the precision and accuracy of the system, a series of four replicate 10ppb standards were run. The curve, MDL and precision and accuracy results are listed in Table 4, while both the 0.2ppb and 10ppb chromatograms of the TO-15 standard are displayed in Figures 1 and 2.

Compound	Compound RF	Curve %RSD	MDL (0.2ppb)	Precision (10ppb)	Accuracy (10ppb)	Compound	Compound RF	Curve %RSD	MDL (0.2ppb)	Precision (10ppb)	Accuracy (10ppb)
propene	0.558	16.32	0.03	1.41	100.91	Trichloroethene	0.296	7.08	0.03	1.03	104.70
Dichlorodifluoromethane	1.458	15.33	0.02	0.79	107.37	1,2-Dichloropropane	0.286	9.18	0.01	2.02	112.00
Chloromethane	0.749	16.02	0.03	0.58	94.69	methyl methacrylate	0.238	10.66	0.03	2.25	116.46
Vinyl Chloride	0.855	14.47	0.03	0.84	97.38	1,4-Dioxane	0.038	15.80	0.13	1.41	129.77
1,3-Butadiene	0.627	13.62	0.02	0.64	109.06	Bromodichloromethane	0.503	8.45	0.02	1.80	124.31
Bromomethane	0.642	15.03	0.02	0.51	97.66	cis-1,3-Dichloropropene	0.406	10.50	0.02	2.20	119.54
Chloroethane	0.468	13.19	0.03	0.48	100.86	4-methyl-2-pentanone	0.308	25.12	0.05	0.40	127.63
Ethanol	0.108	18.83	0.04	0.65	99.51	Toluene	0.909	9.91	0.02	2.36	113.59
Trichlorofluoromethane	1.516	15.07	0.03	0.50	107.28	trans-1,3-Dichloropropene	0.405	9.70	0.03	2.04	123.47
1,2-Dichlorotetrafluoroethane	0.490	15.27	0.02	0.43	107.37	1,1,2-Trichloroethane	0.294	9.53	0.03	1.58	108.82
1,1,2-trichlorofluoroethane	1.218	15.35	0.03	0.46	103.03	Tetrachloroethene	0.257	8.74	0.03	1.46	105.36
Acrolein	0.202	19.46	0.07	1.58	102.68	Dibromochloromethane	0.375	7.72	0.02	1.33	107.07
1,1-Dichloroethene	0.701	13.96	0.04	0.88	101.66	2-Hexanone	0.219	28.09	0.05	1.18	144.79
Acetone	0.805	20.70	0.02	1.73	126.48	1,2-Dibromoethane	0.409	9.18	0.03	1.80	111.33
2-propanol	0.783	19.59	0.03	0.54	111.55	Chlorobenzene	0.825	8.54	0.02	2.28	112.61
Carbon Disulfide	2.083	16.34	0.03	0.76	105.25	Ethylbenzene	1.372	9.08	0.02	2.63	114.95
Methylene Chloride	0.667	19.83	0.02	1.10	96.71	Xylene (m+p)	1.108	12.70	0.03	2.49	117.99
MTBE	1.698	20.89	0.03	1.76	120.82	Styrene	0.880	8.97	0.02	2.71	112.04
cis-1,2-Dichloroethene	1.100	17.52	0.02	0.82	105.08	Xylene (o)	1.255	7.98	0.02	2.37	115.53
Vinyl acetate	1.521	18.77	0.03	1.54	116.23	Bromoform	0.341	7.20	0.02	1.84	112.10
1,1-Dichloroethane	1.248	17.61	0.03	1.45	112.01	1,1,2,2-Tetrachloroethane	0.688	6.68	0.02	2.17	116.90
trans-1,2-Dichloroethene	1.172	18.65	0.04	0.93	108.10	1,3,5-Trimethylbenzene	1.057	7.34	0.03	2.13	119.27
ethyl acetate	0.157	21.24	0.05	1.36	112.46	1,2,4-Trimethylbenzene	1.221	7.62	0.03	2.46	118.08
2-Butanone	1.521	18.78	0.03	1.54	116.25	4-Ethyl Benzene	1.002	9.19	0.03	2.13	111.02
THF	0.550	18.10	0.04	1.83	118.89	Benzyl Chloride	0.838	11.52	0.02	2.65	128.15
Chloroform	1.213	16.52	0.03	1.65	119.03	1,3-Dichlorobenzene	0.627	6.52	0.02	1.73	113.08
1,1,1-Trichloroethane	1.125	19.42	0.03	1.72	117.58	1,4-Dichlorobenzene	0.622	5.96	0.02	2.09	113.96
Carbon Tetrachloride	0.897	19.88	0.03	1.50	116.00	1,2,-Dichlorobenzene	0.567	6.52	0.02	2.38	115.56
Cyclohexane	1.319	20.52	0.03	1.80	97.81	1,2,4-Trichlorobenzene	0.311	26.56	0.01	0.96	124.45
Benzene	2.075	17.65	0.04	2.07	117.22	Naphthalene	0.795	29.04	0.02	0.66	125.38
1,2-Dichloroethane	0.925	17.69	0.04	1.20	116.44	Hexachlorobutadiene	0.246	17.88	0.03	1.21	124.00
Heptane	1.156	17.74	0.04	1.95	115.86	Average	0.798	14.58	0.03	1.50	113.07

Table 4: Experimental Results

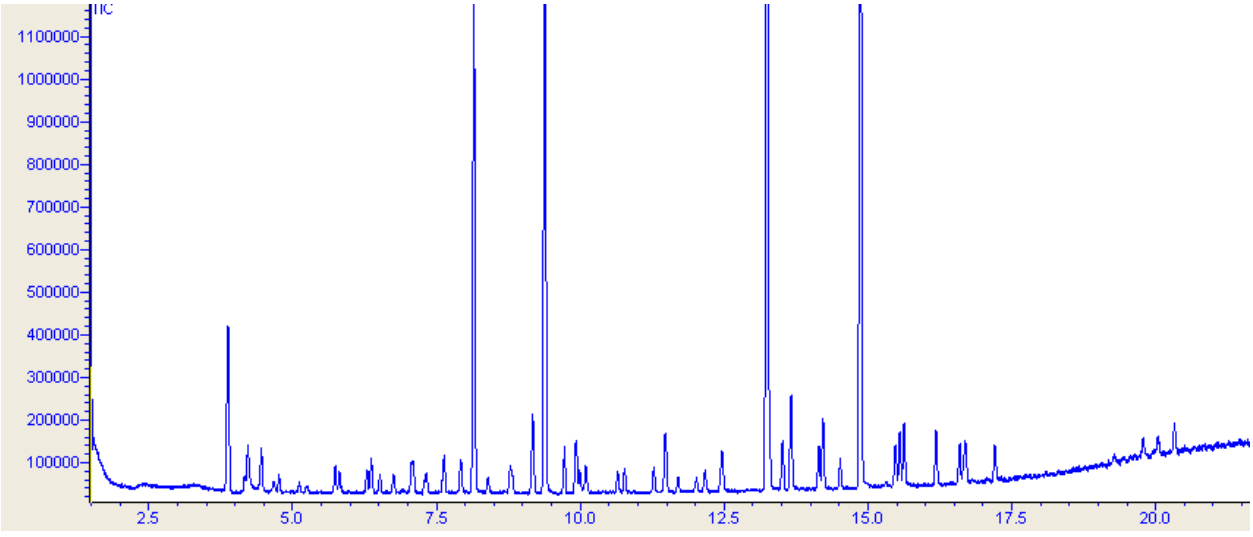


Figure 1: 0.2ppb Standard Chromatogram

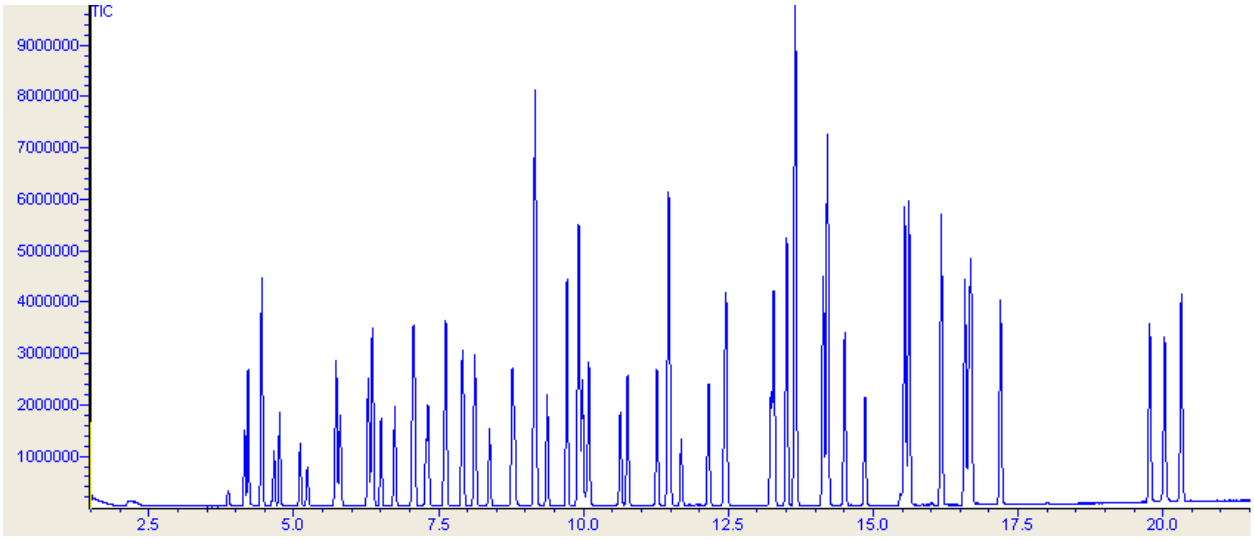


Figure 2: 10ppb Standard Chromatogram

Conclusions

The 8900 pre-concentrator met all of the requirements set by the NJDEP for low level USEPA TO-15. The results displayed excellent precision at less than 2% and percent recovery averaging at about 113%. The average linearity over the 0.2ppbv to 40ppbv range was less than 15%; these results far exceeded the less than 30% requirements. The 8900 pre-concentrator proved to be an excellent system for this analysis.

References

Determination of Volatile Organic Compounds (VOCs) in Air Collected In Specially-Prepared Canisters and Analyzed By Gas Chromatography/Mass Spectrometry (GC/MS), Compendium Method TO-15, United States Environmental Protection Agency, January, 1999.

New Jersey Department of Environmental Protection Modified Low Level TO-15 Method, Office of Data Quality Division of Remediation Management and Response, Trenton, New Jersey, March, 2007.

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