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Application Note

Comparison of Helium and Nitrogen Purge Gases for the Analysis of Volatile Organic Compounds (VOCs) by Purge and Trap

Introduction

Although analysis of VOCs by purge and trap (P&T) is considered a mature technique, advances in methodology and instrumentation are still being developed. Most USEPA methods for this analysis call for extraction of the analytes by purging with helium for 11 minutes at 40 ml/minute making P&T one of the largest consumers of helium in the laboratory. Many labs are seeking a less expensive alternative to helium and have turned to nitrogen as a viable option. Nitrogen is abundantly available, inert, and currently a quarter of the price of helium, resulting in significant cost savings.

This study is based on one of the most widely used methods for volatiles analysis by P&T and GC/MS; USEPA Method 8260. Suggested operating conditions for the analysis and results comparing helium to nitrogen purge are presented.

Experimental

Instrumentation used for this study included an OI Analytical Eclipse 4760 Purge and Trap Sample Concentrator, an OI Analytical 4100 Water/Soil Sample Processor, and an Agilent 7890A/5975C GC/MS. Since the nitrogen molecule is larger than that of helium and has a higher heat capacity, it is able to draw more thermal energy from the purged solution. This makes it necessary to increase the thermal energy present in the purged volume. For that reason, several changes were made to the standard conditions including slowing the purge flow slightly, increasing the purge temperature, and increasing the desorb temperature.

A nine-point calibration from 1 ppb to 200 ppb was run using the conditions listed in **Table 1**. Acrolein and Acetonitrile were run at 2x and the ketones were run at 5x. Response factors were calculated using the internal standard approach. The same conditions were used for the helium and nitrogen purges. Initial Demonstrations of Proficiency (IDP) were run at the 50 ppb level.

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Table 1. Operating Conditions

Purge-and-Trap	Eclipse 4760 P&T Sample Concentrator
Trap	#10 trap; Tenax® / Silica gel / CMS
Purge Gas	Zero grade He/N_2 at 38 ml/min
Purge Time	11 min
Sparge Mount Temperature	50 °C
Sample Temperature	50 °C
Desorb Time	0.5 min
Bake Time	4 min
OI #10 Trap Temperature	Ambient during purge 180 °C during desorb pre-heat 200 °C during desorb 210 °C during bake
Water Management	120 °C during purge Ambient during desorb 240 °C during bake
Transfer Line Temperature	140 °C
Six-port Valve Temperature	140 °C

Autosampler	4100 Water/Soil Sample Processor
System Gas	Zero grade nitrogen
Purge Gas	Zero grade helium/nitrogen
LV20 Pressure	8.0 psi
Loop-based Time Settings	Default
Rinse Water	80 °C
Soil Sample Transfer	150 °C
Soil Oven	150 °C
Soil Lift Station	50 °C
Gas Chromatograph	**Agilent 7890A**
Column	Restek Rtx-VMS 30 meter, 0.25 mm ID, 1.4 µmdf
Carrier Gas	Zero grade helium
Inlet Temperature	250 °C
Inlet Liner	Agilent Ultra Inert, 1 mm straight taper
Column Flow Rate	0.8 mL/min
Split Ratio	100:1
Oven Program	Hold at 40 °C for 1.5 min 16 °C/minute to 180 °C 40 °C/minute to 220 °C Hold at 220 °C for 2 min Total GC Run is 13.25 min
Mass Spectrometer	**Agilent 5975C**
Mode	Scan 35 - 300 amu
Scans/Second	5.19
Solvent Delay	1.40 min
Transfer Line Temperature	250 °C
Source Temperature	300 °C
Quadrupole Temperature	200 °C
Draw Out Plate	6 mm

4100 Sample Processor Methods			
Sample Type	Waters Only	Soils Only	Blanks Only
Needle Rinses	1	1	0
SAM A (µL)	5	5	5
SAM B (µL)	0	0	0
SAM C (µL)	0	0	0
SAM D (µL)	0	0	0
Purge Time (min)	11.0	11.0	11.0
Desorb Time (min)	0.5	0.5	0.5
P&T Rinses	2	2	2
Rinse Water	Hot	Hot	Hot
Water Stir Time (min)	0.0		
Water Settle Time (sec)	0		
Soil Add Water to Vial (#loops)		*1 x 5 mL	
Soil Pre-Heat Stir		Yes	
Soil Pre-Heat/Purge Temp (˚C)		50 ˚C	
Soil Stir During Purge		Yes	

*Suggested initial volume in vial should be 5 mL and final volume 10 mL

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Results

Calibration results passed USEPA Method 8260D criteria. Please see **Table 2** and **Table 3**. IDP results were also well within method limits. Please see Table 4 and Table 5. When changing from He to N_2 purge, it has been observed that response declines 40-60%. Even with this reduction in response, good quality data was achieved.

Table 2. Liquid Calibration Data

Analyte	Compound	Liquid RF Helium	% RSD Helium	Liquid RF Nitrogen	% RSD Nitrogen
1	pentafluorobenzene (IS)	N/A	N/A	N/A	N/A
2	dichlorodifluoromethane	0.569	5.15	0.717	10.44
3	chloromethane	1.086	6.22	1.533	8.40
4	vinyl chloride	0.675	5.57	0.924	8.33
5	bromomethane	0.4	7.79	0.485	9.33
6	chloroethane	0.327	4.57	0.493	10.49
7	trichlorofluoromethane	0.448	6.70	0.577	9.96
8	ethyl ether	0.264	9.34	0.363	11.79
9	1,1-dichloroethene	0.438	8.60	0.617	10.16
10	carbon disulfide	1.271	5.47	1.747	9.93
11	1,1,2-trichloro-1,2,2-trifluoroethane	0.449	7.47	0.577	9.96
12	methyl iodide	0.866	5.60	1.244	9.18
13	acrolein(2x)	0.042	7.98	0.063	9.51
14	allyl chloride	0.25	3.57	0.369	11.80
15	methylene chloride	0.481	5.16	0.715	9.80
16	acetone(5x)	0.069	14.33	0.118	7.23
17	trans-1,2-dichloroethene	0.496	4.08	0.741	12.13
18	methyl tert-butyl ether	1.035	2.74	1.414	6.28
19	acetonitrile(2x)	0.108	18.36	0.216	13.15
20	chloroprene	1.112	5.50	1.375	6.37
21	1,1-dichloroethane	1.179	3.84	1.586	6.75
22	acrylonitrile	1.112	5.50	1.375	6.37
23	cis-1,2-dichloroethene	0.542	3.74	0.718	5.56
24	2,2-dichloropropane	0.484	10.34	0.737	10.39
25	bromochloromethane	0.31	4.84	0.436	8.28
26	chloroform	0.995	3.82	1.298	6.05
27	methyl acrylate	0.996	7.15	0.896	5.63
28	carbon tetrachloride	0.831	3.85	1.073	9.13
29	tetrahydrofuran	0.441	12.62	0.462	8.58
30	dibromofluoromethane (SS)	0.568	3.83	0.722	4.95
31	1,1,1-trichloroethane	0.825	4.16	1.075	6.03
32	2-butanone(5x)	0.086	12.78	0.08	15.87
33	1,1-dichloropropene	0.681	4.49	0.766	3.73
34	1,4-difluorobenzene (IS)	N/A	N/A	N/A	N/A
35	benzene	1.274	2.68	1.455	5.80
36	methacrylonitrile	0.506	8.04	0.530	5.20
37	1,2-dichloroethane-d4 (SS)	0.054	4.62	0.064	7.51
38	1,2-dichloroethane	0.59	10.84	0.702	12.46

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Table 2. **Liquid Calibration Data (continued)**

Analyte	Compound	Liquid RF Helium	% RSD Helium	Liquid RF Nitrogen	% RSD Nitrogen
39	trichloroethene	0.393	4.34	0.432	4.49
40	dibromomethane	0.261	4.49	0.294	7.84
41	1,2-dichloropropane	0.438	3.34	0.455	5.35
42	bromodichloromethane	0.509	4.73	0.556	6.04
43	methyl methacrylate	0.296	10.09	0.212	9.26
44	2-chloroethyl-vinyl-ether	0.288	8.68	0.186	15.08
45	cis-1,3-dichloropropene	0.516	4.16	0.453	7.42
46	chlorobenzene-d5 (IS)	N/A	N/A	N/A	N/A
47	toluene-d8(ss)	1.263	0.86	1.400	2.89
48	toluene	0.879	2.57	0.956	4.66
49	2-nitropropane	0.244	8.36	0.250	9.63
50	4-methyl-2-pentanone(5x)	0.050	7.39	0.058	9.18
51	tetrachloroethene	0.330	1.65	0.367	3.92
52	trans-1,3-dichloropropene	0.568	3.84	0.617	5.92
53	ethyl methacrylate	0.413	8.44	0.363	7.59
54	1,1,2-trichloroethane	0.320	4.19	0.327	8.22
55	chlorodibromomethane	0.501	3.85	0.555	7.26
56	1,3-dichloropropane	0.514	3.56	0.482	3.79
57	1,2-dibromoethane	0.410	3.76	0.382	5.82
58	2-hexanone(5x)	0.819	8.96	0.583	5.97
59	chlorobenzene	1.078	2.56	1.086	4.56
60	ethylbenzene	1.717	3.79	1.740	5.57
61	1,1,1,2-tetrachloroethane	0.406	4.00	0.579	12.22
62	m,p-xylenes(2x)	1.301	6.57	0.655	8.93
63	o-xylene	0.590	9.32	0.690	7.36
64	styrene	0.975	9.28	0.878	10.52
65	bromoform	0.367	4.57	0.347	7.51
66	isopropylbenzene	1.570	8.73	1.879	11.70
67	cis-1,4-dichloro-2-butene	0.168	12.81	0.103	15.64
68	1,4-dichlorobenzene-d4 (IS)	N/A	N/A	N/A	N/A
69	4-bromofluorobenzene (SS)	0.892	1.76	0.857	4.87
70	n-propylbenzene	3.875	5.05	4.490	9.07
71	bromobemzene	0.921	3.96	0.948	6.56
72	1,1,2,2-tetrachloroethane	1.115	7.19	1.323	9.47
73	2-chlorotoluene	2.432	3.79	3.053	6.72
74	1,3,5-trimethylbenzene	2.575	10.54	3.313	15.43
75	1,2,3-trichloropropane	0.282	9.37	0.305	6.50
76	trans-1,4-dichloro-2-butene	0.481	6.74	0.340	10.88
77	4-chlorotoluene	2.276	5.33	2.215	13.20
78	tert-butylbenzene	2.139	8.33	2.674	22.60 r=1.00
79	1,2,4-trimethylbenzene	2.575	10.54	3.313	15.43
80	sec-butylbenzene	3.268	8.06	4.388	16.58

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Table 2. Liquid Calibration Data (continued)

Analyte	Compound	Liquid RF Helium	% RSD Helium	Liquid RF Nitrogen	% RSD Nitrogen
81	p-isopropytoluene	2.629	11.87	3.423	18.93
82	1,3-dichlorobenzene	1.658	3.66	1.734	4.87
83	1,4-dichlorobenzene	1.697	2.55	1.695	3.83
84	n-butylbenzene	2.629	7.81	3.211	10.8
85	1,2-dichlorobenzene	1.563	2.42	1.947	6.69
86	1,2-dibromo-3-chloropropane	0.255	6.20	0.377	15.1
87	hexachlorobutadiene	0.459	7.92	0.613	5.63
88	1,2,4-trichlorobenzene	0.867	9.23	1.422	8.48
89	naphthalene	2.771	14.3	4.679	7.30
90	1,2,3-trichlorobenzene	0.826	9.16	1.531	5.79

Table 3. Solid Calibration Data

Analyte	Compound	Solid RF Helium	% RSD Helium	Solid RF Nitrogen	% RSD Nitrogen
1	pentafluorobenzene (IS)	N/A	N/A	N/A	N/A
2	dichlorodifluoromethane	0.635	9.53	0.785	6.94
3	chloromethane	1.117	9.70	1.529	9.55
4	vinyl chloride	0.703	14.46	0.906	8.23
5	bromomethane	0.412	10.68	0.505	7.34
6	chloroethane	0.329	10.61	0.488	9.73
7	trichlorofluoromethane	0.481	13.67	0.621	7.78
8	ethyl ether	0.260	4.79	0.300	4.44
9	1,1-dichloroethene	0.447	11.25	0.621	8.05
10	carbon disulfide	1.389	10.50	1.837	7.18
11	1,1,2-trichloro-1,2,2-trifluoroethane	0.482	13.94	0.621	7.78
12	methyl iodide	0.926	9.90	1.266	4.70
13	acrolein(2x)	0.044	10.92	0.052	10.52
14	allyl chloride	0.260	12.50	0.346	6.72
15	methylene chloride	0.533	6.41	0.722	4.79
16	acetone(5x)	0.061	6.94	0.074	12.94
17	trans-1,2-dichloroethene	0.516	6.45	0.692	9.04
18	methyl tert-butyl ether	1.068	8.52	1.332	5.78
19	acetonitrile(2x)	0.103	11.50	0.141	9.21
20	chloroprene	1.109	10.13	1.281	7.16
21	1,1-dichloroethane	1.228	6.65	1.502	4.61
22	acrylonitrile	1.109	10.13	1.281	7.16
23	cis-1,2-dichloroethene	0.579	8.54	0.698	4.96
24	2,2-dichloropropane	0.567	16.13	0.823	6.55
25	bromochloromethane	0.352	10.01	0.395	4.82
26	chloroform	1.050	6.60	1.266	5.95
27	methyl acrylate	0.707	12.00	0.575	14.79

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Table 3. Solid Calibration Data (continued)

Analyte	Compound	Solid RF Helium	% RSD Helium	Solid RF Nitrogen	% RSD Nitrogen
28	carbon tetrachloride	0.878	7.48	1.058	5.90
29	tetrahydrofuran	0.287	9.06	0.284	15.46
30	dibromofluoromethane (SS)	0.657	2.17	0.743	4.28
31	1,1,1-trichloroethane	0.860	7.20	1.057	5.55
32	2-butanone(5x)	0.055	9.00	0.052	10.06
33	1,1-dichloropropene	0.698	8.79	0.732	7.14
34	1,4-difluorobenzene (IS)	N/A	N/A	N/A	N/A
35	benzene	1.221	6.33	1.371	4.83
36	methacrylonitrile	0.343	6.96	0.327	8.52
37	1,2-dichloroethane-d4 (SS)	0.053	3.93	0.056	6.98
38	1,2-dichloroethane	0.563	8.27	0.596	6.47
39	trichloroethene	0.370	5.48	0.392	5.14
40	dibromomethane	0.247	5.64	0.251	4.52
41	1,2-dichloropropane	0.442	6.35	0.434	3.81
42	bromodichloromethane	0.504	6.91	0.513	2.93
43	methyl methacrylate	0.203	13.07	0.155	10.28
44	2-chloroethyl-vinyl-ether	0.207	14.52	0.132	20.71 r=0.997
45	cis-1,3-dichloropropene	0.511	5.91	0.427	9.25
46	chlorobenzene-d5 (IS)	N/A	N/A	N/A	N/A
47	toluene-d8(ss)	1.244	1.47	1.405	3.04
48	toluene	0.823	6.57	0.900	4.97
49	2-nitropropane	0.153	13.03	0.156	13.56
50	4-methyl-2-pentanone(5x)	0.029	11.84	0.033	8.48
51	tetrachloroethene	0.314	8.74	0.348	6.23
52	trans-1,3-dichloropropene	0.548	5.82	0.566	5.77
53	ethyl methacrylate	0.324	10.71	0.263	18.86
54	1,1,2-trichloroethane	0.282	5.19	0.282	6.46
55	chlorodibromomethane	0.467	7.33	0.484	3.82
56	1,3-dichloropropane	0.447	5.36	0.416	5.69
57	1,2-dibromoethane	0.331	7.69	0.316	6.11
58	2-hexanone(5x)	0.411	11.98	0.323	10.45
59	chlorobenzene	1.032	7.56	1.005	5.27
60	ethylbenzene	1.577	8.30	1.572	8.00
61	1,1,1,2-tetrachloroethane	0.409	6.33	0.540	8.45
62	m,p-xylenes(2x)	0.600	9.36	0.587	11.22
63	o-xylene	0.559	9.87	0.641	9.15
64	styrene	0.910	11.37	0.811	13.58
65	bromoform	0.293	6.75	0.285	9.03
66	isopropylbenzene	1.451	11.05	1.703	11.29
67	cis-1,4-dichloro-2-butene	0.115	13.47	0.083	14.93
68	1,4-dichlorobenzene-d4 (IS)	N/A	N/A	N/A	N/A
69	4-bromofluorobenzene (SS)	0.883	1.92	0.886	3.95

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Table 3. Solid Calibration Data (continued)

Analyte	Compound	Solid RF Helium	% RSD Helium	Solid RF Nitrogen	% RSD Nitrogen
70	n-propylbenzene	3.670	7.61	4.090	11.50
71	bromobenzene	0.909	6.87	0.944	7.57
72	1,1,2,2-tetrachloroethane	0.818	10.01	1.022	8.01
73	2-chlorotoluene	2.346	7.26	2.867	8.87
74	1,3,5-trimethylbenzene	2.497	12.06	3.161	14.13
75	1,2,3-trichloropropane	0.202	8.53	0.212	9.16
76	trans-1,4-dichloro-2-butene	0.328	6.71	0.258	10.52
77	4-chlorotoluene	2.175	7.31	2.169	11.41
78	tert-butylbenzene	2.084	10.21	2.641	19.66
79	1,2,4-trimethylbenzene	2.497	12.06	3.161	14.13
80	sec-butylbenzene	3.249	7.84	4.363	12.98
81	p-isopropytoluene	2.621	9.90	3.368	16.29
82	1,3-dichlorobenzene	1.631	5.04	1.673	4.21
83	1,4-dichlorobenzene	1.659	8.17	1.607	5.65
84	n-butylbenzene	2.630	7.86	3.154	6.12
85	1,2-dichlorobenzene	1.549	5.23	1.790	5.09
86	1,2-Dibromo-3-chloropropane	0.150	10.30	0.213	15.64
87	hexachlorobutadiene	0.537	14.23	0.800	13.49
88	1,2,4-trichlorobenzene	0.930	7.36	1.434	9.45
89	naphthalene	2.127	15.81	3.593	5.91
90	1,2,3-trichlorobenzene	0.897	9.11	1.583	9.90

Table 4. Liquid IDPs

Analyte	Compound	Liquid % Recovery Helium	% RSD Helium	Liquid % Recovery Nitrogen	% RSD Nitrogen
2	dichlorodifluoromethane	91.69	5.01	86.44	3.99
3	chloromethane	96.67	3.56	96.12	1.50
4	vinyl chloride	98.00	3.05	98.01	1.77
5	bromomethane	96.88	2.02	96.63	0.48
6	chloroethane	100.76	3.64	94.52	2.32
7	trichlorofluoromethane	95.54	4.99	91.28	4.46
8	ethyl ether	96.47	2.85	95.95	1.64
9	1,1-dichloroethene	98.61	3.31	95.60	4.14
10	carbon disulfide	98.62	3.33	95.84	3.05
11	1,1,2-trichloro-1,2,2-trifluoroethane	95.21	5.00	91.28	4.46
12	methyl iodide	97.65	3.43	99.18	2.16
13	acrolein	93.47	8.00	100.49	4.14
14	allyl chloride	100.29	2.07	94.40	2.20
15	methylene chloride	100.29	4.30	97.73	2.75

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Table 4. **Liquid IDPs (continued)**

Analyte	Compound	Liquid % Recovery Helium	% RSD Helium	Liquid % Recovery Nitrogen	% RSD Nitrogen
16	acetone	98.93	8.39	93.89	2.12
17	trans-1,2-dichloroethene	99.91	2.41	94.12	2.58
18	methyl tert-butyl ether	100.04	2.41	101.88	1.25
19	acetonitrile	77.01	5.87	100.65	5.46
20	chloroprene	103.16	3.01	103.87	2.30
21	1,1-dichloroethane	98.97	2.59	99.43	1.55
22	acrylonitrile	103.66	2.97	103.87	2.30
23	cis-1,2-dichloroethene	99.12	2.80	101.81	1.57
24	2,2-dichloropropane	92.85	6.09	91.41	3.12
25	bromochloromethane	97.30	3.99	97.21	1.57
26	chloroform	97.92	3.23	99.45	1.71
27	methyl acrylate	99.52	1.64	101.56	1.67
28	carbon tetrachloride	98.15	3.71	96.08	3.47
29	tetrahydrofuran	87.39	9.07	100.00	8.61
30	dibromofluoromethane (SS)	96.47	2.90	96.24	1.79
31	1,1,1-trichloroethane	99.56	3.25	98.98	2.18
32	2-butanone	93.34	4.44	99.71	2.43
33	1,1-dichloropropene	102.85	2.80	102.09	2.66
35	benzene	101.02	2.33	101.01	1.07
36	methacrylonitrile	97.60	1.15	101.74	1.24
37	1,2-dichloroethane-d4 (SS)	98.88	4.50	98.46	1.87
38	1,2-dichloroethane	94.24	1.91	95.31	0.81
39	trichloroethene	100.69	2.15	98.53	2.35
40	dibromomethane	98.55	1.42	100.12	1.66
41	bromodichloromethane	101.33	2.05	101.13	0.89
42	1,2-dichloropropane	98.88	1.48	98.98	0.97
43	methyl methacrylate	104.39	2.43	104.13	0.72
44	2-chloroethyl-vinyl-ether	105.62	3.18	99.71	2.85
45	cis-1,3-dichloropropene	101.43	2.10	101.17	1.77
47	toluene-d8(ss)	100.83	1.17	100.40	0.56
48	toluene	100.99	2.06	103.38	1.78
49	2-nitropropane	96.14	2.56	98.87	1.51
50	4-methyl-2-pentanone	103.50	2.02	109.23	2.46
51	tetrachloroethene	101.18	2.43	99.25	2.90
52	trans-1,3-dichloropropene	102.25	2.42	101.07	2.04
53	ethyl methacrylate	107.33	2.53	107.87	1.53
54	1,1,2-trichloroethane	101.15	1.87	102.57	0.44
55	chlorodibromomethane	101.63	1.32	100.37	0.74

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Table 4. **Liquid IDPs (continued)**

Analyte	Compound	Liquid % Recovery Helium	% RSD Helium	Liquid % Recovery Nitrogen	% RSD Nitrogen
56	1,3-dichloropropane	101.64	2.20	101.74	0.89
57	1,2-dibromoethane	101.04	0.59	100.35	1.11
58	2-hexanone	102.97	3.92	106.74	1.22
59	chlorobenzene	100.52	1.68	99.97	1.28
60	ethylbenzene	102.87	1.25	105.17	0.64
61	1,1,1,2-tetrachloroethane	97.65	1.74	98.62	2.31
62	m,p-xylenes	104.88	2.05	107.67	1.25
63	o-xylene	105.16	1.34	108.74	1.33
64	styrene	106.88	1.61	109.95	0.65
65	bromoform	101.22	2.44	100.94	1.24
66	isopropylbenzene	105.69	2.45	108.05	8.89
67	cis-1,4-dichloro-2-butene	93.48	3.72	97.55	6.55
69	4-bromofluorobenzene (SS)	102.52	1.30	102.64	2.14
70	bromobenzene	103.98	1.01	106.44	1.57
71	n-propylbenzene	100.83	1.89	101.96	1.84
72	1,1,2,2-tetrachloroethane	97.38	0.57	100.52	0.67
73	2-chlorotoluene	103.81	1.35	104.61	1.06
74	1,3,5-trimethylbenzene	108.49	1.59	113.92	1.59
75	1,2,3-trichloropropane	98.40	2.78	101.88	1.64
76	trans-1,4-dichloro-2-butene	103.01	3.69	97.49	3.20
77	4-chlorotoluene	105.54	1.15	108.24	0.70
78	tert-butylbenzene	107.72	2.17	94.53	1.99
79	1,2,4-trimethylbenzene	108.49	1.59	113.92	1.59
80	sec-butylbenzene	106.97	1.82	111.26	1.82
81	p-isopropytoluene	108.94	2.01	113.98	1.63
82	1,3-dichlorobenzene	101.79	1.08	100.81	2.20
83	1,4-dichlorobenzene	100.42	1.06	99.67	0.49
84	n-butylbenzene	104.10	2.24	107.89	0.49
85	1,2-dichlorobenzene	100.10	1.45	100.23	1.06
86	1,2-Dibromo-3-chloropropane	98.06	2.77	95.85	4.44
87	hexachlorobutadiene	96.86	2.61	98.40	0.98
88	1,2,4-trichlorobenzene	100.08	2.26	98.22	2.05
89	naphthalene	103.18	2.43	106.62	2.47
90	1,2,3-trichlorobenzene	100.53	3.66	101.50	2.69

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Table 5. Solid IDPs

Analyte	Compound	Solid % Recovery Helium	% RSD Helium	Solid % Recovery Nitrogen	% RSD Nitrogen
2	dichlorodifluoromethane	92.46	5.28	87.83	4.20
3	chloromethane	91.54	2.85	87.08	2.67
4	vinyl chloride	91.81	3.45	88.90	1.68
5	bromomethane	90.71	2.89	91.10	3.79
6	chloroethane	92.44	2.90	87.43	3.71
7	trichlorofluoromethane	93.09	4.34	88.00	2.89
8	ethyl ether	93.78	2.82	92.60	2.87
9	1,1-dichloroethene	94.27	2.51	88.75	3.68
10	carbon disulfide	92.60	3.22	90.42	4.19
11	1,1,2-trichloro-1,2,2-trifluoroethane	92.95	4.35	88.00	2.89
12	methyl iodide	92.32	2.15	93.66	3.82
13	acrolein	89.52	6.96	92.44	8.40
14	allyl chloride	96.05	2.94	88.54	2.18
15	methylene chloride	91.53	3.34	93.85	3.51
16	acetone	92.39	2.34	87.89	6.63
17	trans-1,2-dichloroethene	93.94	2.53	87.17	3.45
18	methyl tert-butyl ether	94.54	3.74	94.64	3.12
19	acetonitrile	93.69	4.92	97.77	7.51
20	chloroprene	99.18	2.26	91.94	1.46
21	1,1-dichloroethane	93.95	1.87	90.54	2.52
22	acrylonitrile	99.18	2.26	91.94	1.46
23	cis-1,2-dichloroethene	94.05	2.55	91.80	2.90
24	2,2-dichloropropane	95.51	5.46	89.99	3.12
25	bromochloromethane	88.46	2.65	94.03	1.82
26	chloroform	94.49	2.02	90.99	1.92
27	methyl acrylate	97.93	3.35	98.42	2.53
28	carbon tetrachloride	93.61	3.56	89.21	2.54
29	tetrahydrofuran	92.12	5.04	91.64	2.80
30	dibromofluoromethane (SS)	96.65	0.25	98.01	2.78
31	1,1,1-trichloroethane	95.00	2.82	91.38	3.45
32	2-butanone	98.15	2.28	90.69	2.33
33	1,1-dichloropropene	99.06	2.62	92.60	2.18
35	benzene	96.02	2.72	90.22	1.40
36	methacrylonitrile	94.82	3.38	98.34	0.59
37	1,2-dichloroethane-d4 (SS)	97.18	1.53	97.58	3.45
38	1,2-dichloroethane	89.76	2.75	90.91	1.12
39	trichloroethene	95.57	0.79	91.37	2.26
40	dibromomethane	91.70	5.28	92.46	2.16
41	bromodichloromethane	94.11	2.41	92.78	1.40
42	1,2-dichloropropane	95.01	6.38	93.24	0.79
43	methyl methacrylate	100.96	3.74	93.48	2.24
44	2-chloroethyl-vinyl-ether	99.73	2.86	77.47	2.40
45	cis-1,3-dichloropropene	96.33	2.40	93.89	1.09

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Table 5. Solid IDPs (continued)

Analyte	Compound	Solid % Recovery Helium	% RSD Helium	Solid % Recovery Nitrogen	% RSD Nitrogen
47	toluene-d8(ss)	100.71	0.97	98.29	5.47
48	toluene	95.73	2.25	91.06	1.66
49	2-nitropropane	87.67	3.06	90.71	2.05
50	4-methyl-2-pentanone	99.83	2.83	100.89	1.69
51	tetrachloroethene	95.86	2.14	87.59	1.41
52	trans-1,3-dichloropropene	97.52	2.52	92.90	1.20
53	ethyl methacrylate	101.27	1.95	102.12	0.98
54	1,1,2-trichloroethane	95.13	3.31	93.40	1.89
55	chlorodibromomethane	94.24	2.45	93.31	0.25
56	1,3-dichloropropane	97.04	2.84	93.60	1.71
57	1,2-dibromoethane	97.80	2.72	92.39	1.79
58	2-hexanone	102.44	2.13	97.63	1.31
59	chlorobenzene	94.60	2.34	91.21	0.88
60	ethylbenzene	99.31	1.94	93.59	0.84
61	1,1,1,2-tetrachloroethane	90.81	1.79	92.75	2.07
62	m,p-xylenes	102.74	2.38	97.66	1.21
63	o-xylene	101.46	1.75	97.31	0.91
64	styrene	103.02	1.69	97.30	1.41
65	bromoform	95.63	1.72	90.74	1.38
66	isopropylbenzene	104.88	2.59	100.34	1.79
67	cis-1,4-dichloro-2-butene	94.51	5.61	82.66	7.98
69	4-bromofluorobenzene (SS)	102.01	1.96	103.93	1.73
70	bromobenzene	101.46	2.25	97.64	2.07
71	n-propylbenzene	93.71	2.29	91.50	2.46
72	1,1,2,2-tetrachloroethane	94.62	3.06	92.83	3.76
73	2-chlorotoluene	99.39	2.01	97.50	2.82
74	1,3,5-trimethylbenzene	105.09	2.71	102.81	2.41
75	1,2,3-trichloropropane	89.77	2.11	97.99	3.93
76	trans-1,4-dichloro-2-butene	97.45	2.61	88.71	1.27
77	4-chlorotoluene	100.66	1.65	94.92	1.91
78	tert-butylbenzene	101.89	2.35	102.07	3.14
79	1,2,4-trimethylbenzene	105.09	2.71	102.81	2.41
80	sec-butylbenzene	102.73	3.27	100.85	1.99
81	p-isopropytoluene	104.49	2.86	100.69	2.13
82	1,3-dichlorobenzene	96.84	2.08	91.13	1.66
83	1,4-dichlorobenzene	95.00	1.21	89.73	0.70
84	n-butylbenzene	102.18	3.16	93.28	1.30
85	1,2-dichlorobenzene	95.33	2.07	94.19	1.62
86	1,2-Dibromo-3-chloropropane	92.88	4.24	92.67	4.36
87	hexachlorobutadiene	93.46	2.03	86.02	2.22
88	1,2,4-trichlorobenzene	97.45	2.29	88.35	1.89
89	naphthalene	99.28	4.33	96.29	2.76
90	1,2,3-trichlorobenzene	95.10	3.46	92.04	2.23

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Figure 1. Liquid with He Purge

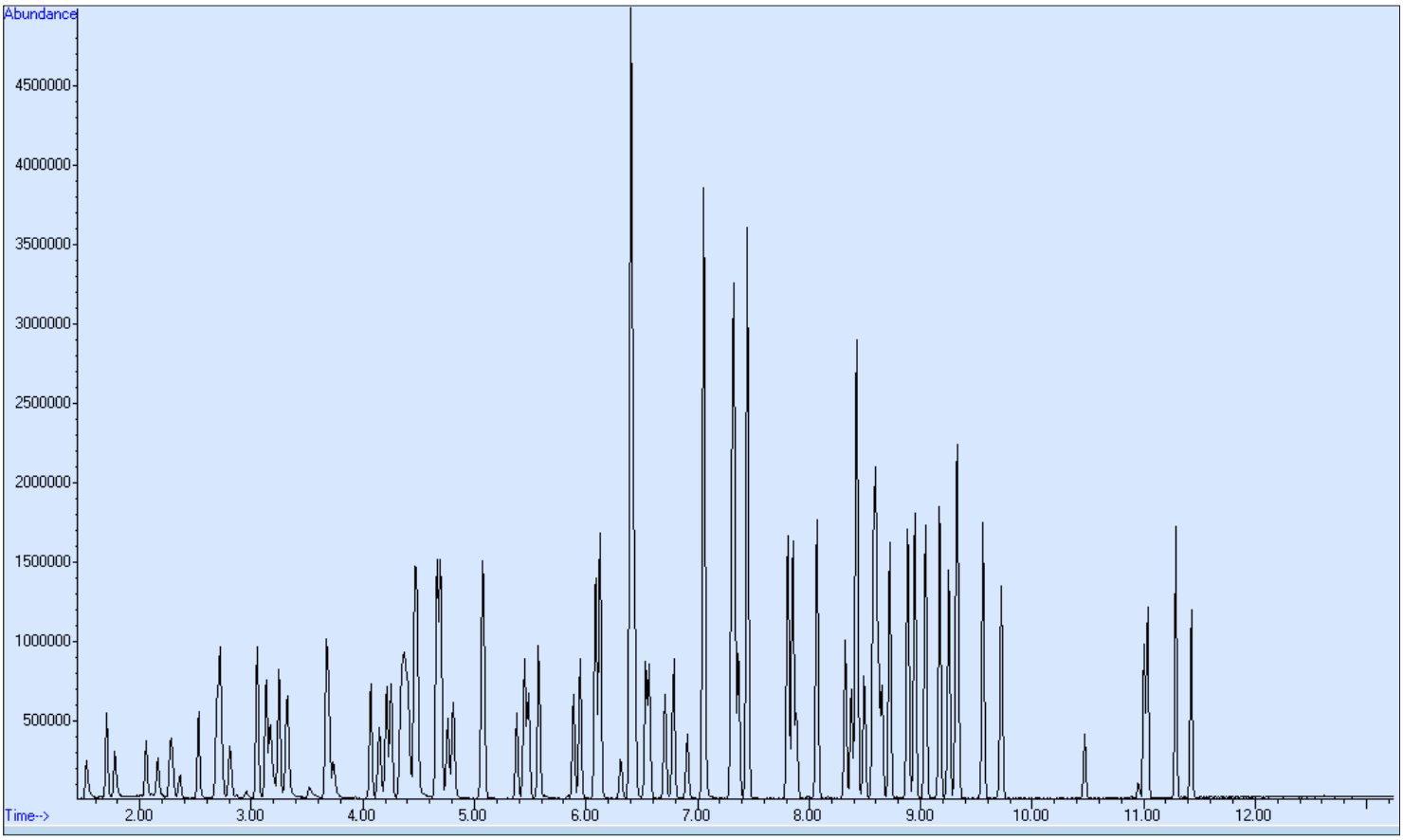

Figure 2. Solid with He Purge

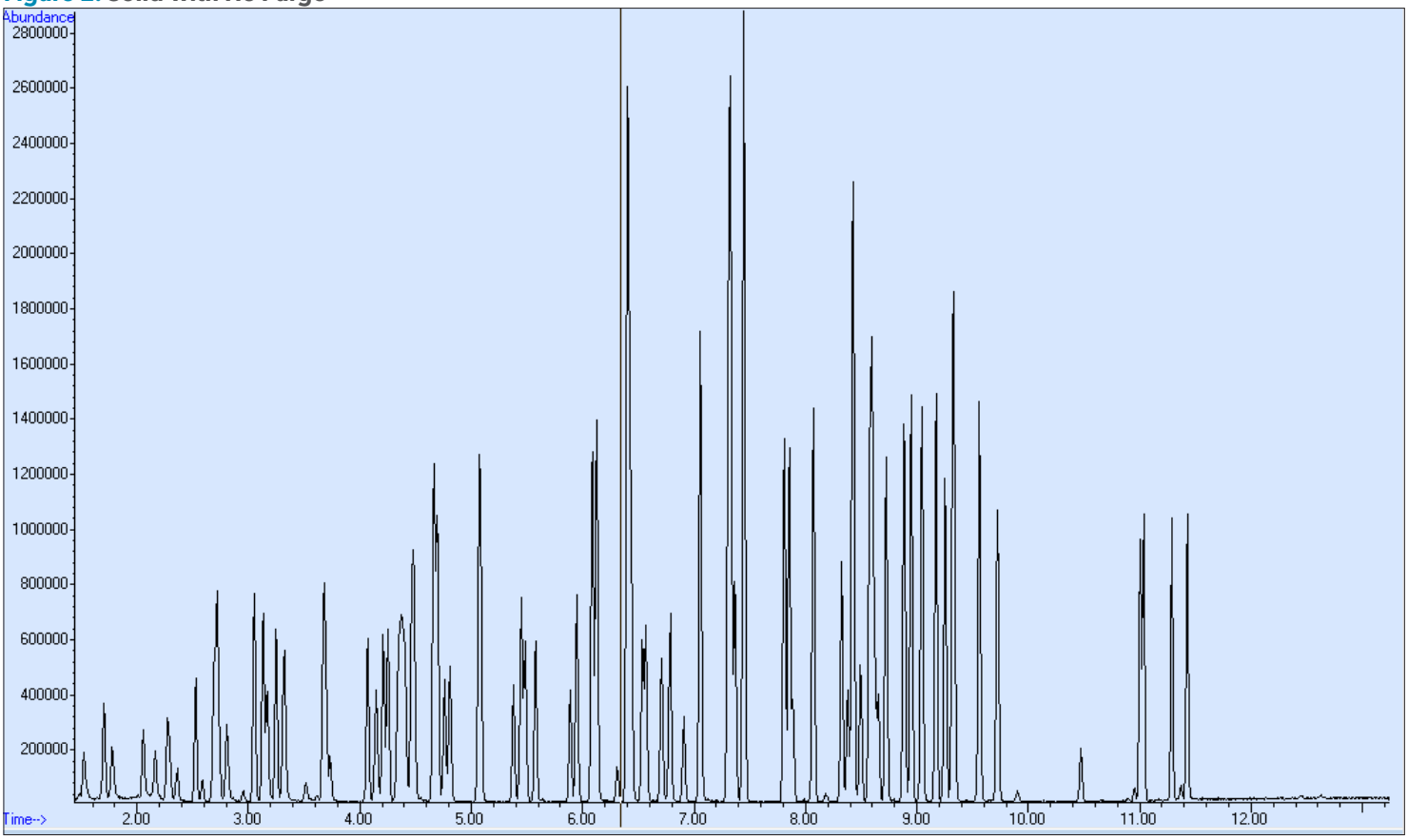

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Figure 3. Liquid with N_2 Purge

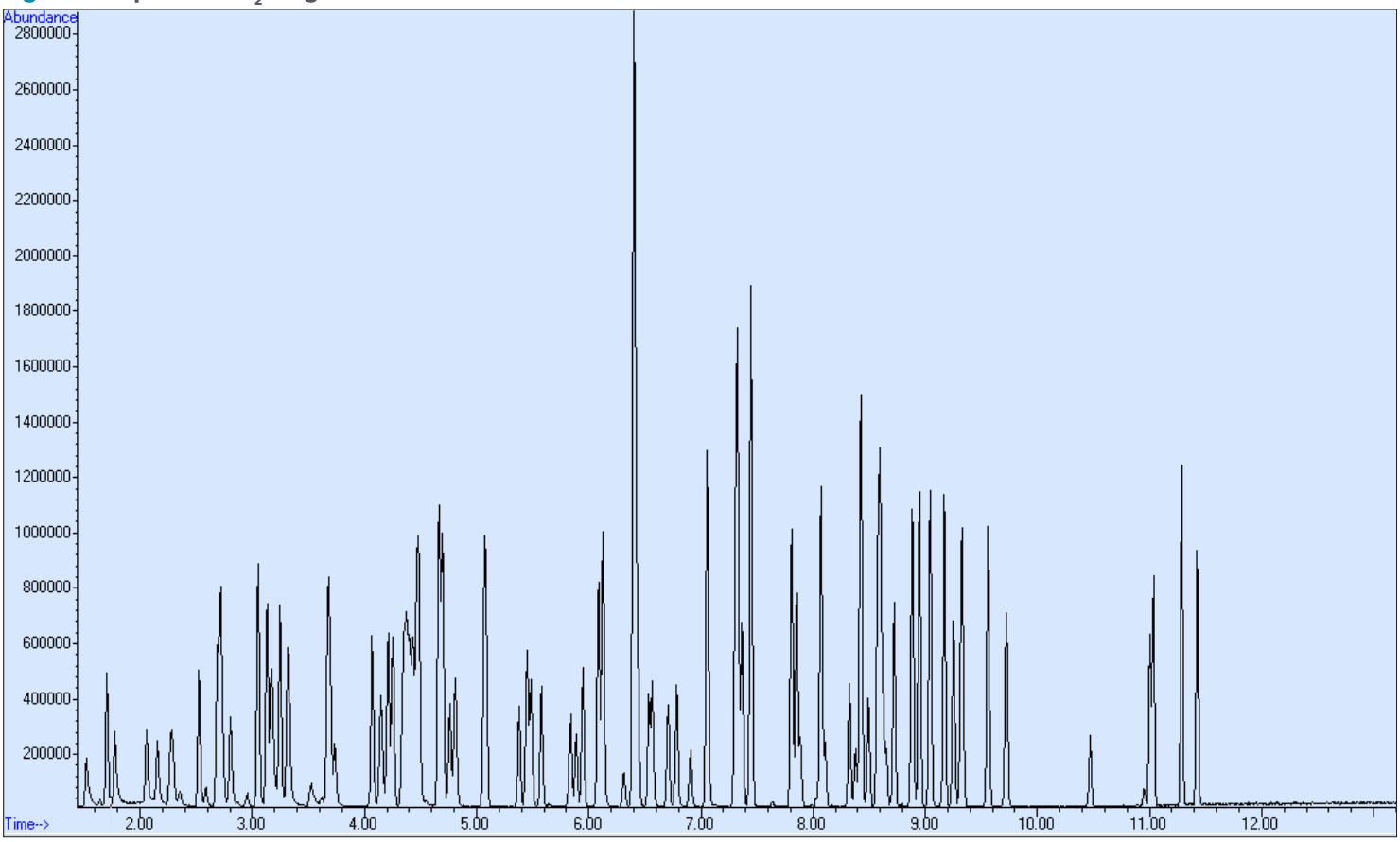
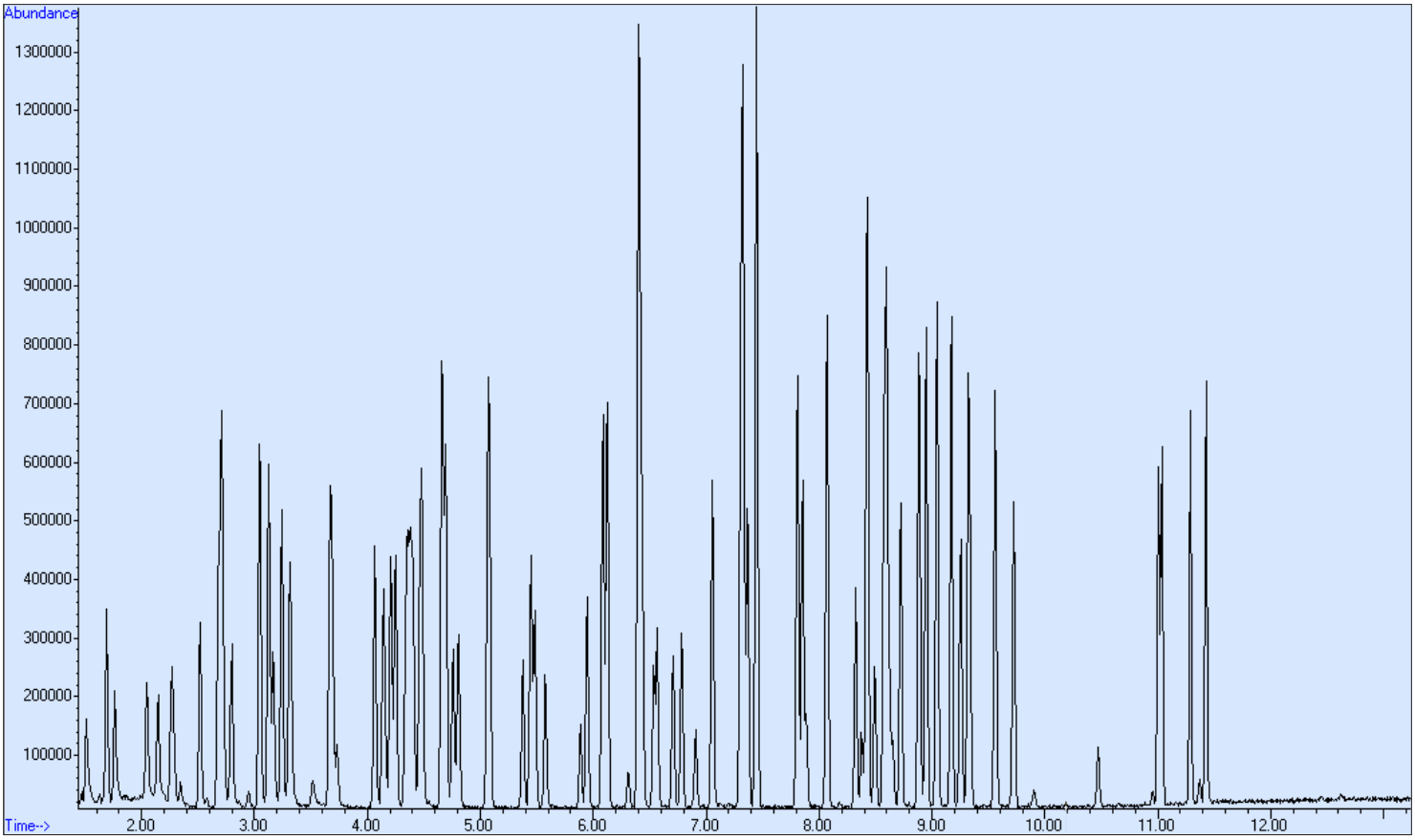


Figure 4. Solid with N_2 Purge



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Conclusions

Nitrogen is a viable alternative purge gas for the analysis of volatile compounds by USEPA Method 8260D which may result in cost savings for the laboratory. It has been observed, however, that using nitrogen as a purge gas may result in more frequent source cleaning and calibrations to be performed.

References

Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry. USEPA Office of Solid Waste, SW 846, Method 8260D, Rev 4, February 2017.

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