Less Solvent Emission using the Biotage® Solvent Recovery SVOC

(Previously Known as SolventTrapSVOC)

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Key Words

Method 8270, Solvent Emission, SolventTrap$_{SVOC}$, DryVap, Method 525.2

Introduction

In the laboratory many chemicals are used in small and larger volumes. Table 1 shows several of the typical kinds of analyses done for environmental compliance. Semivolatile organic compound extraction and evaporation can be a large source of solvent emission to the atmosphere if the solvent is allowed to go up the hood and is not recaptured.

Table 1: Typical Environmental Analyses

Class of Compounds	Materials Used	Emissions
Metals	Acids, water	Some acid vapors
Oil & Grease	Hexane, methanol, evaporation	Hexane evaporation
Volatile Organic Compounds	Purge and Trap Headspace	Samples eluted directly into GC, very little emission
Semivolatile Organic Compounds	Solvent extraction with dichloromethane, hexane, ethyl acetate, others	Solvent evaporation to increase concentration for better sensitivity, generating solvent vapors

Through Title V Federal US regulations and State Implementation Plans up to 10 tons of chlorinated solvent and up to 25 tons total of solvent may be emitted into the air each year with a valid permit. For extremely small emitters, no permit is required if they are not at risk of exceeding the limit. In 2014, four labs in Massachusetts were fined for exceeding this limit without a permit, raising awareness that environmental labs have grown and using a heavy solvent, such as dichloromethane (DCM) places them at risk of a violation.

The SolventTrap$_{SVOC}$ works with the DryVap® so that solvent evaporated through the DryVap is recaptured with the SolventTrap$_{SVOC}$. Figure 1 shows the system with a general description of how the solvent evaporates and is transferred for recapture in the condenser.

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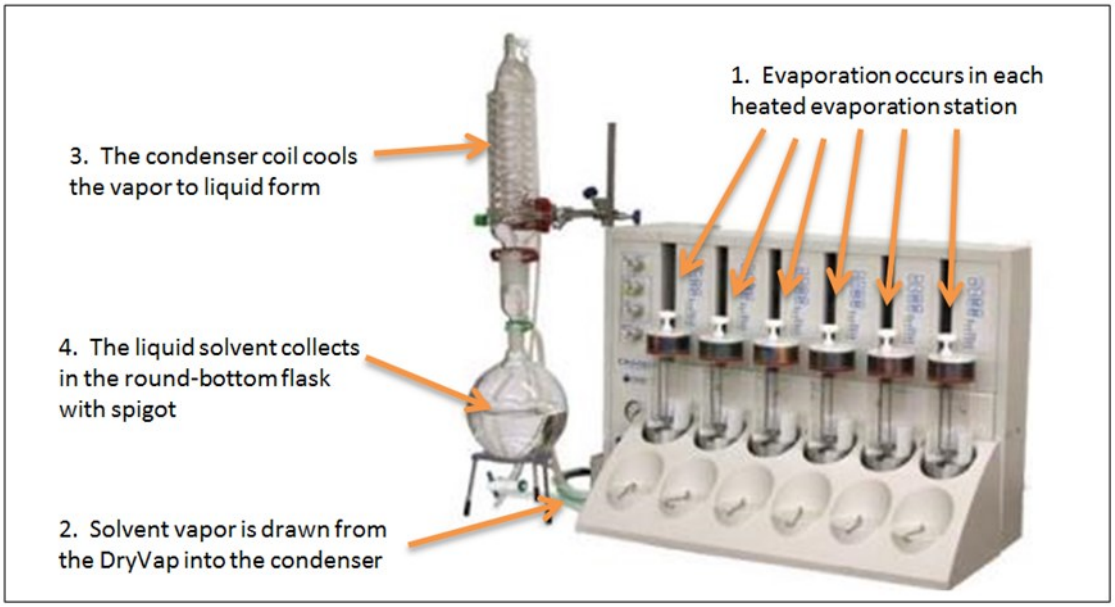

Figure 1. SolventTrap $_{SVOC}$ with the DryVap In-line Drying and Evaporation System.

To ensure that the recovery of solvent necessary to help laboratories stay in compliance, several tests at customer sites using real samples were performed. The goal is to get the best recapture of solvent possible without sacrificing either the analyte recoveries or the speed of evaporation.

Conditions:

Chiller	0° Celsius
Vacuum	-5 in Hg
DryVap Settings	
Dry Volume	OFF
Heat Power	5
Heat Timer	OFF
Auto-Rinse Mode	OFF
DryVap N2 Regulator	Set to 0 psig during the heat state
DryVap N2 Regulator	Set to 20 psig in all 6 stations after heaters turned off

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Lab personnel measured and filled six Evaporation Tubes (PN 03-1588-04) with 1 mL tips and 0.9-mL optical endpoints with 200 mL DCM each. They used a 1-liter graduate cylinder to measure the starting volume, totaling 1200 mL. Each tube was spiked with 50 µg/L of typical semivolatile mix used in US EPA method 8270.1

The total evaporation time to reduce the 200-mL sample to approx. 0.9 mL was about 47 minutes, which is approximately the same as the time without solvent recovery.

The analyte recoveries and RSD for six replicates are shown in Table 2 and a graph of the data is shown in Figure 2.

Table 2. Average Recoveries and %RSD of Six Replicate Semivolatile Spikes

	Target Compounds	Avg n=6	%RSD
1	NDMA	96.5	6.5
2	Pyridine	90.8	4.9
3	2-Picoline	97.8	6.4
4	N-Nitrosomethyl ethylamine	95.8	6.7
5	Methyl Methane Sulfonate	97.8	8.6
6	2-Fluorophenol	88.4	6.1
7	N-Nitroso-diethylamine	96.3	6.5
8	Ethylmethane Sulfonate	96.2	6.0
9	Phenol-d5	93.8	6.5
10	Phenol	93.3	6.3
11	Aniline	100.9	6.7
12	Bis (2-chloroethyl) ether	95.5	5.5
13	Pentachloroethane	90.9	6.3
14	2-Chlorophenol	93.1	6.0
15	1,3-Dichlorobenzene	93.9	6.2
16	1,4-Dichlorobenzene	93.7	6.7
17	1,2-Dichlorobenzene	93.4	6.4
18	Benzyl alcohol	97.5	6.5
19	2-Methyl phenol	95.7	6.1
20	Bis(2chloroisopropyl) ether	92.9	6.8
21	N-Nitrosopyrrolidine	101.9	6.6
22	N-Nitroso-morpholine	95.8	6.3
23	Acetophenone	96.1	6.5

	Target Compounds	Avg n=6	%RSD
24	3+4 Methyl phenol	96.8	6.8
25	N-nitroso-di-n-propylamine	95.1	6.1
26	Hexachloroethane	92.7	6.5
27	O-toluidine	102.0	6.5
28	Nitrobenzene-d5	96.0	6.9
29	Nitrobenzene	95.6	7.1
30	N-Nitroso-piperidine	100.6	7.2
31	Isophorone	98.2	6.4
32	2-Nitrophenol	99.6	7.0
33	2,4-Dimethylphenol	96.4	6.3
34	Bis(2-chlorethoxy)methane	95.3	6.5
35	Benzoic acid	111.8	6.3
36	2,4-Dichlorophenol ether	96.5	6.6
37	1,2,4-Trichlorobenzene	93.6	7.0
38	Naphthalene	94.8	6.7
39	2,6-Dichlorophenol	95.5	6.6
40	4-Chloroaniline	100.1	6.3
41	Hexachloropropene	94.1	6.3
42	Hexachlorobutadiene	92.8	6.6
43	N-nitroso-di-n-butylamine	100.8	5.0
44	4-Chloro-3-methylphenol	102.4	5.9
45	Cis-Isosafrole	95.9	6.2
46	2-Methylnaphthalene	96.2	6.6

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	Target Compounds	Avg n=6	%RSD
47	Hexachlorocyclopentadiene	92.5	7.7
48	1,2,4,5 Tetrachlorobenzene	96.5	7.2
49	Trans-Isosafrole	102.4	6.3
50	2,4,6-Trichlorophenol	102.2	6.1
51	2,4,5-Trichlorophenol	103.8	5.8
52	2-Fluorobiphenyl	98.1	6.2
53	Safrole	100.8	5.8
54	2-Chloronaphthalene	98.5	6.1
55	2-Nitroaniline	108.7	5.8
56	1,4-Naphthaquinone	107.4	5.7
57	Dimethyl phthalate	105.5	5.8
58	1,3-Dinitrobenzene	110.0	5.9
59	2,6-Dinitrotoluene	106.7	6.5
60	Acenaphthylene	102.5	6.0
61	3-Nitroaniline	109.4	6.0
62	Acenaphthene	102.1	5.6
63	2,4-Dinitrophenol	114.5	6.4
64	Pentachlorobenzene	102.9	6.6
65	4-Nitrophenol	104.9	6.9
66	Dibenzofuran	102.8	5.2
67	2,4-Dinitrotoluene	110.2	5.8
68	2,3,4,6-Tetrachlorophenol	107.1	6.1
69	1-Naphthylamine	103.1	5.8
70	2-Naphthylamine	108.5	5.5
71	Diethylphthalate	105.4	5.9
72	Fluorene	103.7	5.9
73	4-Chlorophenyl phenyl ether	102.4	5.4
74	4-Nitroaniline	110.6	6.9
75	5-nitro-o-toluidine	112.4	6.2
76	4,6-Dinitro-2-methylphenol	111.8	5.3
77	Diphenylamine	105.5	5.6
78	Azobenzene	104.6	5.9
79	2,4,6-Tribromophenol	106.8	5.8
80	1,3,5-Trinitrobenzene	115.6	7.2
81	Phenacetin	111.6	6.8

	Target Compounds	Avg n=6	%RSD
82	4-Bromophenyl phenyl ether	104.8	5.8
83	Hexachlorobenzene	105.3	6.9
84	Pentachlorophenol	105.9	6.2
85	Pentachloronitrobenzene	105.4	6.1
86	4 Aminobiphenyl	112.1	5.8
87	Dinoseb	111.3	6.7
88	Phenanthrene	106.2	6.8
89	Anthracene	105.0	6.0
90	Carbazole	109.9	6.8
91	Di-n-butyl phthalate	109.7	7.0
92	4-Nitroquinoline-1-oxide	122.4	7.3
93	Methapyrilene	109.9	7.1
94	Fluoranthene	108.2	6.1
95	Benzidine	98.3	8.1
96	Pyrene	107.7	6.4
97	P-Terphenyl-d14	108.2	6.6
98	Dimethylaminoazobenzene	111.4	6.7
99	3,3'-Dimethylbenzidine	104.7	6.4
100	Butyl benzyl phthalate	108.3	6.4
101	Acetylaminofluorene	120.1	6.7
102	3,3'-Dichlorobenzidine	112.5	7.1
103	Benz(a)anthracene	107.7	6.8
104	Chrysene	107.2	6.6
105	Bis(2-ethylhexyl)phthalate	109.5	6.8
106	Di-n-octyl phthalate	110.7	6.7
107	7,12-Dimethylbenz(a)anthracene	106.1	6.6
108	Benzo(b)fluoranthene	109.4	6.3
109	Benzo(k)fluoranthene	110.0	6.8
110	Benzo(a)pyrene	107.5	6.8
111	3-Methylcholanthrene	108.6	7.2
112	Indeno(1,2,3-cd)pyrene	106.0	7.4
113	Dibenz(ah)anthracene	105.4	7.2
114	Benzo(ghi)perylene	106.3	7.4

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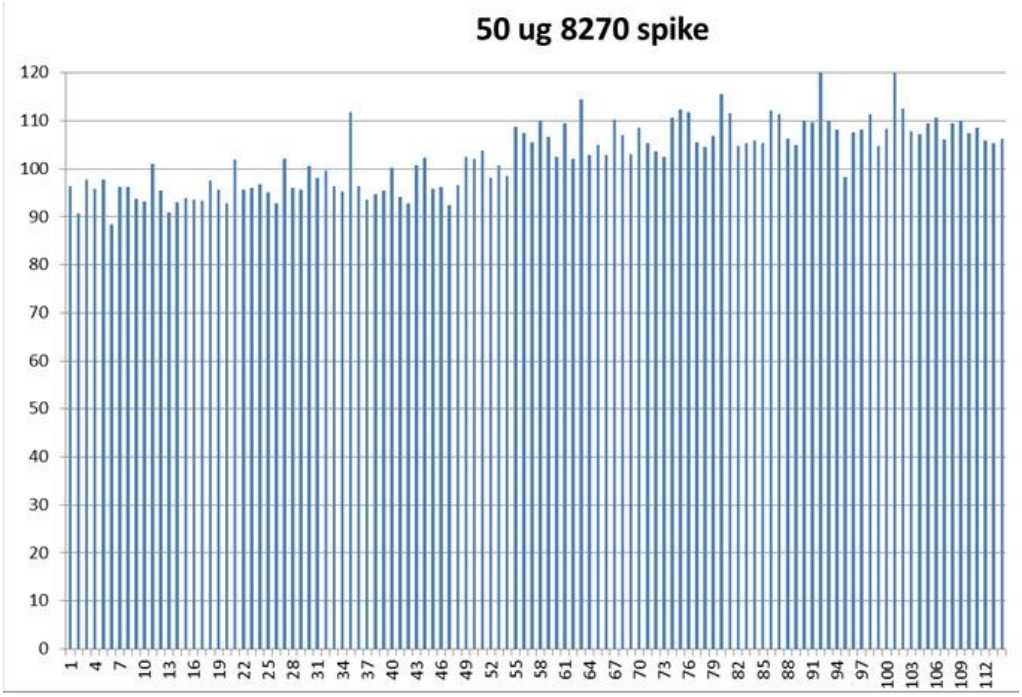

Figure 2. Average percent recovery of six spike replicates of a suite of semivolatile compounds.

A similar experiment was performed to test the performance of the system under conditions of low concentration spikes, such that might be seen if a drinking water sample was extracted. The solvent for evaporation consisted of 25 mL DCM with 5 mL of ethyl acetate and a spike of 5µg/mL. The analyte labels and chart are shown in Figure 3.

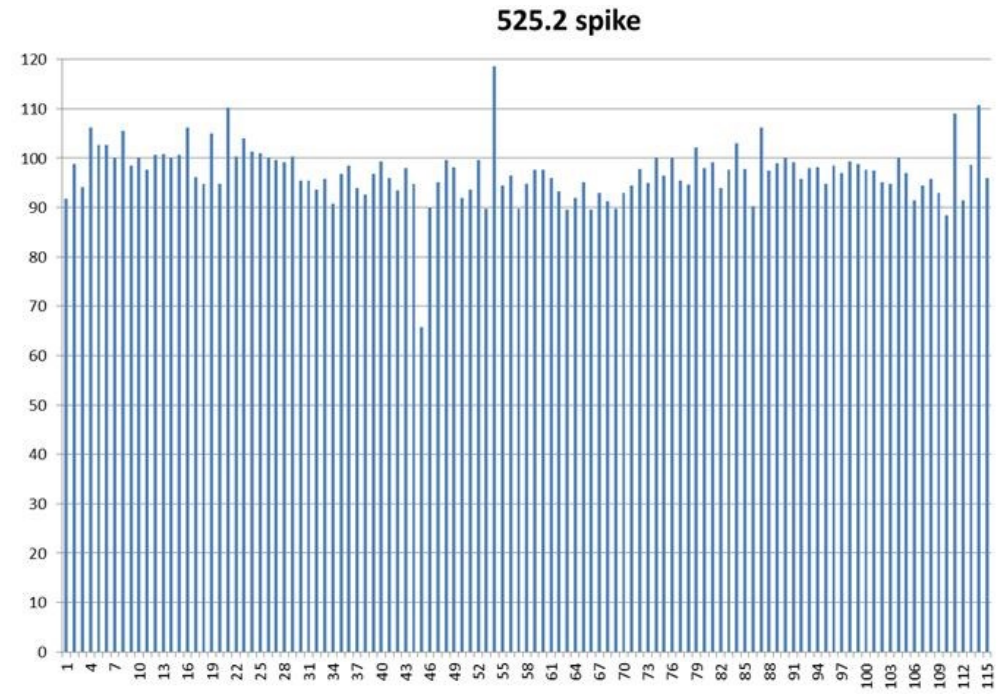

Figure 3. Recoveries of low-concentration analytes with DryVap System evaporation and SolventTrap $_{SVOC}$ solvent recovery.

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Analyte Labels for Figure 3

Acenaphthene d10	1	Ethoprop	26	Metribuzin	51	Disulfoton sulfone	76	Methoxychlor	101
Phenanthrene d10	2	Cycloate	27	Simetryn	52	Butaclor	77	Benz(a)anthracene	102
Chrysene d12	3	Chlorpropham	28	Heptachlor	53	Cis(1)-Chlordane	78	Chrysene	103
Isophorone	4	Trifluralin	29	Ametryn	54	Endosulfan I	79	Fenarimol	104
2-Nitro-m-xylene	5	Dimethoate	30	Alachlor	55	Pyrene-d10	80	Cis-Permethrin	105
Naphthalene	6	a-BHC	31	Prometryn	56	Pyrene	81	Trans-Permethrin	106
Dichlorvos	7	Atraton	32	Terbutryn	57	Napropamide	82	Di-n-octyl phthalate	107
Hexachlorocyclopentadiene	8	Hexachlorobenzene	33	Di-n-butyl phthalate	58	Trans-Nonachlor	83	Benzo(b)fluoranthene	108
EPTC	9	Prometon	34	Bromacil	59	4,4'-DDE	84	Benzo(k)fluoranthene	109
Mevinphos	10	Lindane (g-BHC)	35	Cyanazine	60	Dieldrin	85	Benzo(a)pyrene	110
Butylate	11	Simazine	36	Malathion	61	Tricydazole	86	Fluridone	111
Vernolate	12	Atrazine	37	Metolachlor	62	Terphenyl-d14	87	Perylene-d12	112
Dimethylphthalate	13	Propazine	38	Thiobencarb	63	Endrin	88	Indeno(1,2,3-cd)pyrene	113
Pebulate	14	B-BHC	39	Chlorpyrifos	64	Chlorobenzilate	89	Dibenz(ahanthracene	114
Etridiazole	15	Pentachlorophenol	40	Aldrin	65	Endosulfan II	90	Benzo(ghi)perylene	115
2,6-Dinitrotoluene	16	Pronamide	41	Triadimefon	66	4,4'-DDD	91		
Acenaphthene	17	Diazinon	42	Dacthal	67	Endrin Aldehyde	92		
Acenaphthylene	18	D-BHC	43	MGK-264-A	68	Butyl benzyl phthalate	93		
Chlorone b	19	Phenanthrene	44	Diphenamid	69	Norflurazon	94		
Tebuthiuron	20	Methyl paraoxon	45	MGK-264-B	70	4,4-DDT	95		
2,4-Dinitrotoluene	21	Anthracene	46	Heptachlor epoxide B	71	Endosulfan Sulfate	96		
Molinate	22	Terbacil	47	Heptachlor epoxide A	72	Bis(2-ethylhexyl)adipate	97		
Diethylphthalate	23	Chlorothalonil	48	Fluoranthene	73	Hexazinone	98		
Fluorene	24	Caffeine	49	Trans(g)-Chlordane	74	Triphenylphosphate	99		
Propachlor	25	Acetoclor	50	Stirofos	75	Endrin Ketone	100		

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Recent work, done over the course of two days with two operators shows the reproducibility that can be expected from the system. Each run consists of 6 stations filled with approximately 200 mL of DCM evaporated with the conditions shown in Table 3.

Table 3. DryVap Operating Conditions

DryVap Parameters	Setting
~200 mL DCM in each EV Tube	
Chiller	0°C
Heat Power	5
Vacuum	-5 inches
Sparge Heat	Enabled
Nitrogen	0 psig heat state
Nitrogen	20 psig sparge state

Table 4. Statistics on solvent recovery over several days

Run	% Recovery of DCM
1	97.5
2	97.0
3	97.7
4	97.6
5	97.4
Average	97.4 SD 0.27

The results obtained are shown in Table 4 and show both excellent recovery and precision for recovery of the solvent among the runs.

This data shown for medium and low-level concentrations demonstrates that two specified goals can be achieved at the same time. The statistics show the system operates reproducibly over the course of several days. However, these are clean samples and the question remains how much solvent can be recovered with more complex and “dirty” extracts.

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Soil extracts from continuous liquid-liquid extraction were evaporated using the DryVap and the solvent recovery measured. The types of extracts are shown in Figure 4. Even with the complexity of the extracts the solvent recovery was excellent for DCM.

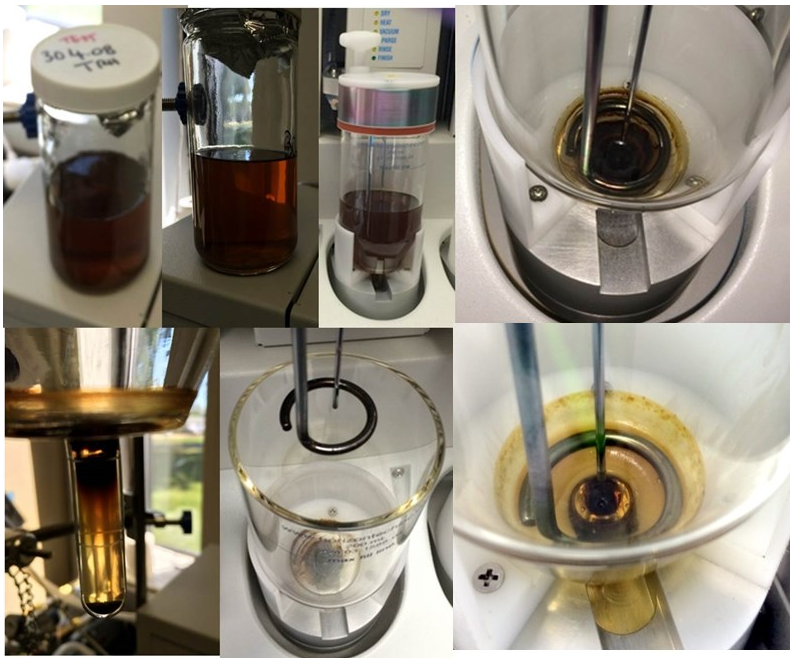

Figure 4: Examples of complex soil extracts evaporated with the DryVap system.

Several other solvents were evaluated in the customer's lab with difficult samples and recovery results for hexane, methanol, and acetone ranged from 93-95%.

The addition of solvent recovery has been shown not to compromise either time for evaporation or recovery of analytes. In addition it provides a way to operate as a "greener" lab and reduce risk of solvent emission regulatory violations.

References

USEPA Method 8270, Methods for Waste Analysis (SW-846) available from the US EPA at http://www.epa.gov/epawaste/hazard/testmethods/sw846/pdfs/8270d.pdf .

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