EVAPORATION

APPLICATION NOTE

Using Labconco's RapidVap Vertex Dry Evaporator per US EPA Method 8270

Protocol: *US EPA Method 8270 requires an evaporation or concentration step when extracting various matrices for semi-volatile compounds. For this application note, Chemir Analytical Services, Maryland Heights, Missouri, used Labconco's RapidVap Vertex Dry Evaporator for the evaporation step of this method. The data presented are compounds from the matrix spike list including surrogates.*

Provisions

Rapid Vap Vertex Evaporator (Cat# 7320020 / SN-111150275 A) with 40 mL block.

40 mL I-Chem vials with Teflon lined septa.

Chromatographic grade Methylene Chloride. Prepurified or ultra-high purity compressed nitrogen.

Agilent 6890 GC with 5973 MSD.

DB-5 GC column.

Analytical Standards purchased from reputable vendor.

Procedure

40 mL vials were filled with 10.0 mL of methylene chloride spiked with 5ug/mL standards and concentrated down between 0.5 mL - <1.0mL, approximately 0.6 mL. The block preheat and temperature was 35°C with 10 p.s.i. of nitrogen which took about ten minutes to complete. The 0.6 mL was brought to 1.0 mL with vial rinsing and spiked with 40 ug/mL of Internal Standard. Three preparations were concentrated, injected in duplicate, and compared to 50 ug/mL standard for percent recovery determinations. Method 8270C was used as a reference.

The following table represents the recoveries of the matrix spike compounds and surrogates using the Ave response factor (R.F.) from three injections of 50 ug/mL standard.

Refer to Table I on reverse.

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APPLICATION NOTE

Table I: Average Recoveries

Compound	1	2	3	4	5	6	6	Ave.	%RSD
2-Flourophenol	71.78	74.98	66.26	65.4	65.64	61.76	61.76	68	1.7
Phenol	71.24	73.66	68.58	67.2	65.76	65.2	65.2	69	4.4
Phenol-d6	74.40	75.58	70.8	68.32	66.84	64.56	64.56	70	3.0
2-Chlorophenol	70.06	70.26	65	65.38	62.88	62	62	66	3.9
1,4-Dichlorobenzene	69.8	70.2	61.96	61.54	64.06	63.72	63.72	65	3.2
N-nitrosodi-n-propylamine	81.42	79.68	75.12	73.86	71.42	71.72	71.72	76	3.5
Nitrobenzene-d5	73.3	74.8	66.7	67.78	68.16	67.1	67.1	70	3.8
1,2,4-Trichlorobenzene	71.88	74.46	70	65.46	67.18	67	67	69	3.2
4-Chloro-3-methylphenol	85.4	83.74	85.7	85	81.4	82.52	82.52	84	3.1
2-Fluorobiphenyl	82.1	83.58	79.3	76.82	75.86	75.24	75.24	79	1.6
Acenaphthene	86.66	87.42	84.94	83.74	81.56	80.9	80.9	84	3.1
4-Nitrophenol	88.9	97.72	102.52	93.02	98.82	93.12	93.12	96	2.4
2,4-Dinitrotoluene	94.34	96.46	93.5	92.3	90.94	89.3	89.3	93	4.5
2,4,6-Tribromophenol	92.52	93.8	98.12	95.24	91.58	87.36	87.36	93	2.3
Pentachlorophenol	96.1	93.4	101.12	96.36	98.22	97.92	97.92	97	3.3
Pyrene	96.22	95.52	92.58	96.56	91.76	93.92	93.92	94	2.4
Terphenyl-d14	100.1	100.1	96.76	99.48	97.42	97.7	97.7	99	1.8

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EVAPORATION

APPLICATION NOTE

Using RapidVap Vertex Dry Evaporator for Evaporation per US EPA Method 550.1

Protocol: *US EPA Method 550.1 requires an evaporation or concentration step when extracting polyaromatic hydrocarbons from liquid. For this application note, Chemir Analytical Services used Labconco's RapidVap Vertex Dry Evaporator for the evaporation step of this method. The data presented are compounds from the matrix spike.*

Materials:

RapidVap Vertex Evaporator (Cat#7320020/ SN-111150275 A) with 40 mL block.

40 mL I-Chem vials with Teflon lined septa.

Chromatographic grade Methylene Chloride and Acetonitrile.

Agilent 1100 HPLC with Ultraviolet and Fluorescence Detection

Polyaromatic Hydrocarbon standards purchase from Supelco Analytical

Procedure:

40mL vials were filled with 10.0mL of methylene chloride spiked with 5ng/mL – 100ng/mL depending on the concentration of the analytical standard and concentrated down to 1mL. The block preheat and temperature was 40°C with 10 p.s.i. of nitrogen. The 0.5mL was brought to 3mL with acetonitrile and then concentrated to 0.5mL. Three preparations were injected in duplicate and compared to a 1ug/mL standard of polyaromatic hydrocarbons for percent recovery determinations. Table 1 shows the recoveries of the matrix spike. Method 550.1 was used as a reference.

Refer to Table I on the back.

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APPLICATION NOTE

Table I: Average Recoveries

Compound/Recovery	1	2	3	4	5	6	Average Recovery	%RSD
Acenapthene	76%	76%	77%	78%	81%	80%	78%	2.8
Acenapthylene	75%	76%	77%	77%	80%	81%	78%	2.9
Anthracene	72%	75%	74%	81%	80%	79%	77%	4.8
Benzo(a)anthracene,Chyrsene[1]	90%	90%	97%	97%	109%	109%	99%	8.5
Benzo(a)pyrene	89%	90%	90%	89%	87%	87%	89%	1.5
Benzo(b)fluoranthene	111%	111%	102%	102%	90%	90%	101%	9.4
Benzo(g,h,i)perylene	92%	94%	93%	91%	91%	89%	92%	1.7
Benzo(k)fluoranthene	90%	90%	90%	90%	88%	89%	90%	1.0
Dibenz(a,h)anthracene	91%	91%	92%	91%	89%	89%	91%	1.2
Fluoranthene	80%	81%	94%	92%	92%	85%	87%	7.3
Fluorene	78%	80%	82%	73%	76%	84%	79%	5.3
Indeno(1,2,3-cd)pyrene[2]	0%	0%	0%	0%	0%	0%	0%	0.0
Napthalene	74%	75%	77%	78%	79%	79%	77%	2.8
Phenanthrene	79%	80%	80%	81%	83%	82%	81%	1.8
Pyrene	86%	87%	87%	86%	86%	85%	86%	0.8

[1]Compounds elute together

[2]Indeno(1,2,3-cd)pyrene was not observed in the standard as well as the matrix spiked samples.

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APPLICATION NOTE

Using RapidVap Vertex Dry Evaporator for Concentrator per US FDA LIB No.# 4438

Protocol: *US FDA analytical method LIB NO# 4438 requires a concentration step when extracting antibiotics from Distillers Grains (DDG). For this application note, Chemir Analytical Services, Maryland Heights, Missouri, used Labconco's RapidVap Vertex Dry Evaporator for the concentration step of this method. The data presented are compounds from the matrix spike.*

Materials:

RapidVap Vertex Evaporator (Cat#7320020/ SN-111150275 A) with 40 mL block.
40 mL I-Chem vials with Teflon line septa.
Chromatographic grade acetonitrile and water.
Formic Acid
Thermo Finnigan Surveyor HPLC with Thermo Finnigan LCQ Deca Mass Spectrometer

Procedure:

40 mL vials were filled with 2.5 mL of a 87/13% water/acetonitrile mixture spiked with approximately 50 µg/mL and evaporated to residue. The block preheat temperature was 50°C with 12 psi of nitrogen which took approximately 70 minutes to complete. The residue was then reconstituted with 5g of a 87/13% water/ acetonitrile mixture. Three preparations were concentrated and compared to a 25 µg/mL standard for percent recovery determinations. Method LIB NO 4438 was used as a reference. The following table represents the recoveries of the matrix spike compounds compared to a single injection of a 25 µg/mL standard.

Refer to Table I on the back.

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APPLICATION NOTE

Table I: Recoveries using MS detection

Analyte/Recovery	Ion	1	2	3	Average	% RSD
Chloramphenicol	-321	83	77	93	84	9.7
Oxyetracycline	+461.1	76	72	71	73	3.5
Monensin	+693.4	76	75	88	80	9.3
Clarithromycin	+748.2	74	78	73	75	3.6
Virginiamycin	526.1	74	65	62	67	9.4
Amplicillian	+350	80	80	78	79	1.5
Bacitracin	++712.1	89	96	88	91	4.8
Streptomycin	+582.2	74	80	81	79	4.8
Erythromycin	+734.2	91	69	69	77	16.8

Table 2: Recoveries using MS-MRM Detection

Analyte/Recovery	Ion	1	2	3	Average	% RSD
Chloramphenicol	-321	95	99	107	101	6.1
Oxyetracycline	+461.1	93	102	99	98	4.4
Monensin	+445.0	95	101	96	98	3.2
Clarithromycin	+693.4	105	115	86	102	14.3
Virginiamycin	+748.2	93	86	86	88	4.3
Amplicillian	526.1	96	101	97	98	2.3
Bacitracin	+350	82	86	98	89	9.3
Streptomycin	++712.1	93	97	77	89	11.7
Erythromycin	+582.2	76	91	90	85	9.9

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