Drying Solvent Extracts using DryDisk® Membrane

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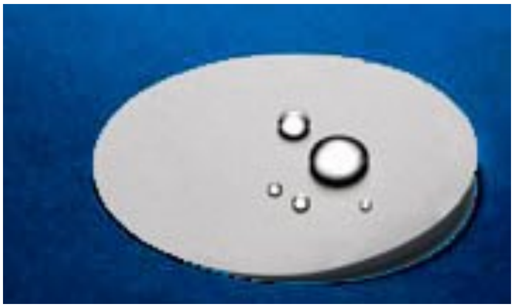


Key Words

Drying, Solvent Extracts, solid phase extraction, SPE, Water Extraction, Membrane, DryDisk, DryDisk-R

Introduction

When analytes are extracted from aqueous samples with solvent either in a liquid-liquid (LLE) format or using solid phase extraction (SPE) it is likely that a small amount of water will be carried into the extract. This water should be removed before the analytical step to ensure that there is no back extraction of analytes into the water and the water is not available to cause damage to the chromatography system.

Removing water with a membrane rather than the older technique of passing the solvent through a column of sodium sulfate (Na_2SO_4) brings several advantages. The most important analytically is that the membrane will not adsorb analytes or contaminate the extract with matrix or other potential interferences.

It is important that the membrane act effectively to keep the water on one side of the membrane and pass the solvent through.

Several experiments were performed to ensure proper operation of the DryDisk and DryDisk-R membranes. The first experiment was designed to evaluate the capacity of the membrane to separate water as a larger percentage of intermediate polar solvent, one that is both soluble in the non-polar extraction solvent and the water, was added to the solvent mixture. Water breakthrough was evaluated by looking for droplets of colored water pulled through the membrane (Figure 1).

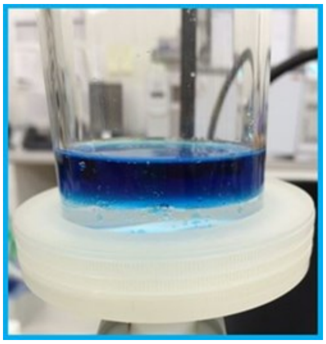


Figure 1: Water (with Blue Color) and Solvent, for Separation

Conditions: 30 mL of a Dichloromethane (DCM):acetone solvent mix, with 5 mL of

Table 1: Water Breakthrough with Varying Water-Soluble Solvent Ratios

DCM (%)	Acetone (%)	Water (mL)	Breakthrough
100	0	5	No
90	10	5	No
80	20	5	No
70	30	5	No
60	40	5	No
50	50	5	No
45	55	5	Yes

deionized water is pulled through the hydrophobic membrane. Table 1 shows the results.
This is an example of a range of solvent mixtures that may be encountered in developing an extraction process and the DryDisk-R performed well. These values will vary depending upon the exact conditions, so we recommend using less than the maximum acetone to avoid breakthrough.

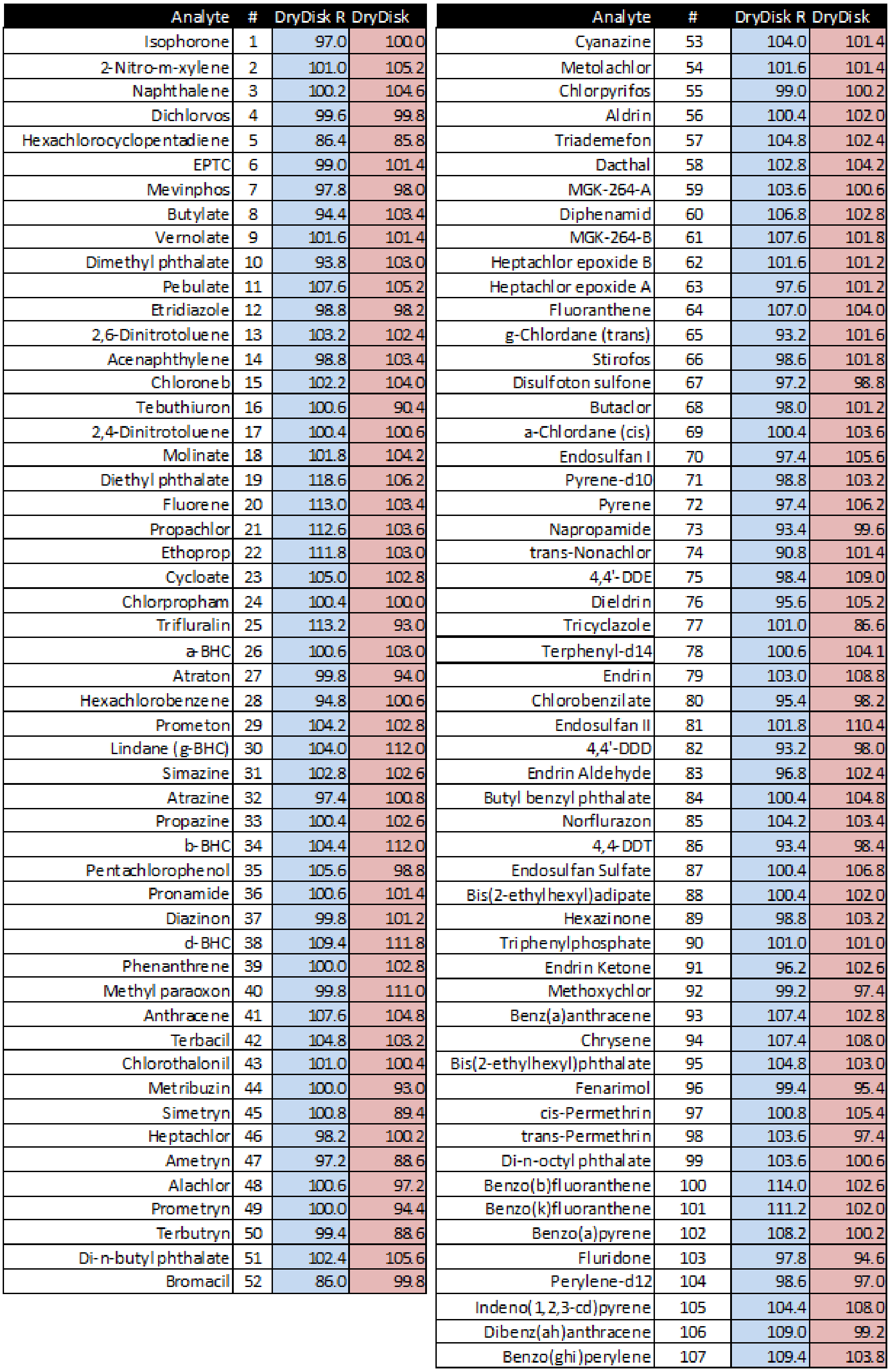
A second experiment was performed to see if both types of the DryDisk gave satisfactory recoveries of a full list of analytes that might be extracted into a solvent such as DCM. The conditions of the experiment are as follows:

Experimental Conditions

Solvent:	25% ethylacetate: 75% DCM. Total volume 30 mL
DryVap Setting	Vacuum –10 in Hg, Nitrogen Pressure set at 27 psi. , Heat power 5, 5 µg spike of 525.2 analytes

The recoveries , shown in Table 2, are excellent and don’t significantly differ from one type of DryDisk to the other.

Table 2: Recoveries using DryDisk Membranes for Drying



Analyte	#	DryDisk R	DryDisk
Isophorone	1	97.0	100.0
2-Nitro-m-xylene	2	101.0	105.2
Naphthalene	3	100.2	104.6
Dichlorvos	4	99.6	99.8
Hexachlorocyclopentadiene	5	86.4	85.8
EPTC	6	99.0	101.4
Mevinphos	7	97.8	98.0
Butylate	8	94.4	103.4
Vernolate	9	101.6	101.4
Dimethyl phthalate	10	93.8	103.0
Pebulate	11	107.6	105.2
Etridiazole	12	98.8	98.2
2,6-Dinitrotoluene	13	103.2	102.4
Acenaphthylene	14	98.8	103.4
Chloroneb	15	102.2	104.0
Tebuthiuron	16	100.6	90.4
2,4-Dinitrotoluene	17	100.4	100.6
Molinate	18	101.8	104.2
Diethyl phthalate	19	118.6	106.2
Fluorene	20	113.0	103.4
Propachlor	21	112.6	103.6
Ethoprop	22	111.8	103.0
Cycloate	23	105.0	102.8
Chlorpropham	24	100.4	100.0
Trifluralin	25	113.2	93.0
a-BHC	26	100.6	103.0
Atraton	27	99.8	94.0
Hexachlorobenzene	28	94.8	100.6
Prometon	29	104.2	102.8
Lindane (g-BHC)	30	104.0	112.0
Simazine	31	102.8	102.6
Atrazine	32	97.4	100.8
Propazine	33	100.4	102.6
b-BHC	34	104.4	112.0
Pentachlorophenol	35	105.6	98.8
Pronamide	36	100.6	101.4
Diazinon	37	99.8	101.2
d-BHC	38	109.4	111.8
Phenanthrene	39	100.0	102.8
Methyl paraoxon	40	99.8	111.0
Anthracene	41	107.6	104.8
Terbacil	42	104.8	103.2
Chlorothalonil	43	101.0	100.4
Metribuzin	44	100.0	93.0
Simetryn	45	100.8	89.4
Heptachlor	46	98.2	100.2
Ametryn	47	97.2	88.6
Alachlor	48	100.6	97.2
Prometryn	49	100.0	94.4
Terbutryn	50	99.4	88.6
Di-n-butyl phthalate	51	102.4	105.6
Bromacil	52	86.0	99.8
Cyanazine	53	104.0	101.4
Metolachlor	54	101.6	101.4
Chlorpyrifos	55	99.0	100.2
Aldrin	56	100.4	102.0
Triademefon	57	104.8	102.4
Dacthal	58	102.8	104.2
MGK-264-A	59	103.6	100.6
Diphenamid	60	106.8	102.8
MGK-264-B	61	107.6	101.8
Heptachlor epoxide B	62	101.6	101.2
Heptachlor epoxide A	63	97.6	101.2
Fluoranthene	64	107.0	104.0
g-Chlordane (trans)	65	93.2	101.6
Stirofos	66	98.6	101.8
Disulfoton sulfone	67	97.2	98.8
Butaclor	68	98.0	101.2
a-Chlordane (cis)	69	100.4	103.6
Endosulfan I	70	97.4	105.6
Pyrene-d10	71	98.8	103.2
Pyrene	72	97.4	106.2
Napropamide	73	93.4	99.6
trans-Nonachlor	74	90.8	101.4
4,4'-DDE	75	98.4	109.0
Dieldrin	76	95.6	105.2
Tricyclazole	77	101.0	86.6
Terphenyl-d14	78	100.6	104.1
Endrin	79	103.0	108.8
Chlorobenzilate	80	95.4	98.2
Endosulfan II	81	101.8	110.4
4,4'-DDD	82	93.2	98.0
Endrin Aldehyde	83	96.8	102.4
Butyl benzyl phthalate	84	100.4	104.8
Norflurazon	85	104.2	103.4
4,4-DDT	86	93.4	98.4
Endosulfan Sulfate	87	100.4	106.8
Bis(2-ethylhexyl)adipate	88	100.4	102.0
Hexazinone	89	98.8	103.2
Triphenylphosphate	90	101.0	101.0
Endrin Ketone	91	96.2	102.6
Methoxychlor	92	99.2	97.4
Benz(a)anthracene	93	107.4	102.8
Chrysene	94	107.4	108.0
Bis(2-ethylhexyl)phthalate	95	104.8	103.0
Fenarimol	96	99.4	95.4
cis-Permethrin	97	100.8	105.4
trans-Permethrin	98	103.6	97.4
Di-n-octyl phthalate	99	103.6	100.6
Benzo(b)fluoranthene	100	114.0	102.6
Benzo(k)fluoranthene	101	111.2	102.0
Benzo(a)pyrene	102	108.2	100.2
Fluridone	103	97.8	94.6
Perylene-d12	104	98.6	97.0
Indeno(1,2,3-cd)pyrene	105	104.4	108.0
Dibenz(ah)anthracene	106	109.0	99.2
Benzo(ghi)perylene	107	109.4	103.8

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An easier way to visualize this data to see the graphic representation of the compounds listed in Table 2. The graph below shows the recoveries in Figure 2.

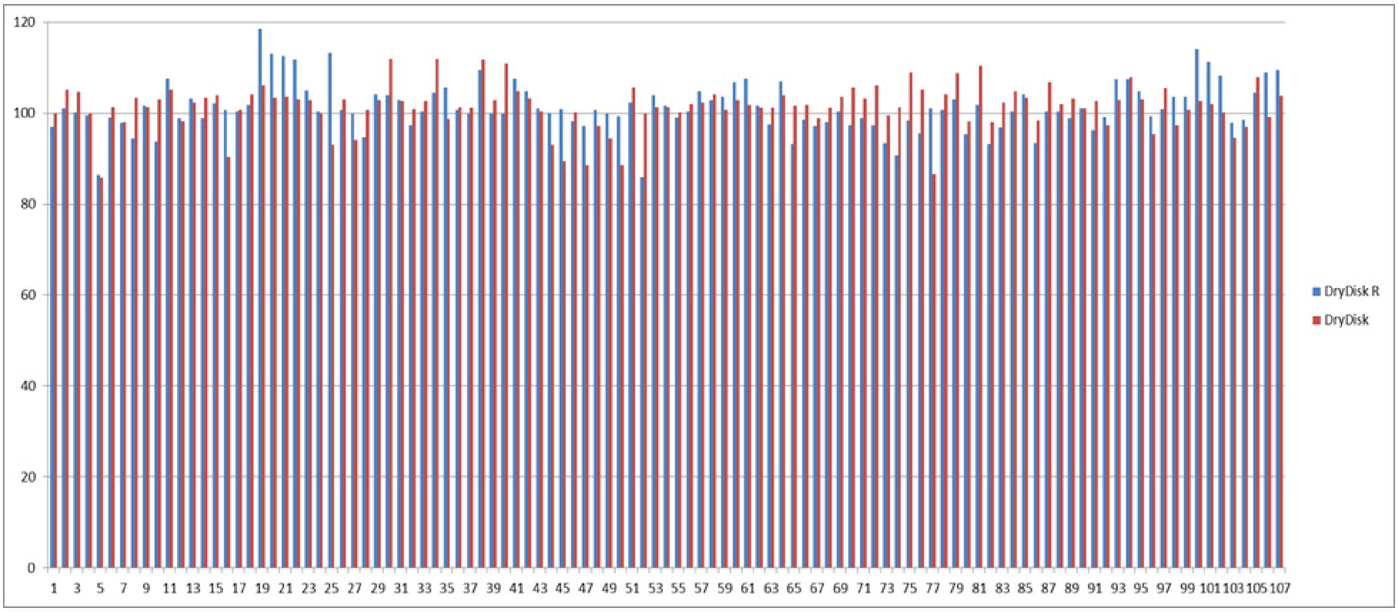

Figure 2. Recoveries of a large suite of compounds after drying with DryDisk

One concern when using a drying material is if the material will introduce contamination into the extract. A test was done to see if analytes of interest spiked into an extract at low concentrations, in fact yielded more than 100% recovery.[1]

Procedure

Complete Assembly Test

1. Prepare two 1000-mL reagent water aliquots
2. Adjust the samples' pH to be less than 2
3. Spike the complete analyte mix into the samples
4. Extract the samples with conventional LLE
5. Dry one sample with sodium sulfate
6. Dry the other sample with DryDisk
7. Analyze both samples with GC/MS and compare results

The samples were spiked with a full semivolatile (method 8270) mix but, as most contamination typically appears in the form of phthalates, the data in Table 4 was condensed to focus on these compounds. As seen from the data, the recoveries for the various phthalate compounds closely match those from the sodium sulfate run. This indicates that the DryDisk membrane and holder assembly introduced no additional phthalates.

Table 4: Contamination Evaluation, Analyte Percentage Recoveries

Analytes	DryDisk		Na_2SO_4	
	Conc (ugL)	% Recovery	Conc (ug/L)	% Recovery
Phenol	6.05	37.8	6.10	38.1
Napththalene	12.03	75.2	12.74	79.6
2,6-Dichlorophenol	12.23	76.4	12.97	81.1
Dimethylphthalate	12.80	80.0	17.20	107.5
2,4-Dinitrophenol	8.43	52.7	6.39	39.9
Pentachlorobenzene	12.64	79.0	13.22	82.6
4-Nitrophenol	14.31	89.4	13.92	87.0
2,3,4,6-Tetrachlorophenol	13.86	86.6	13.81	86.3
Diethylphthalate	13.50	84.4	13.31	78.6
Pentachlorophenol	13.80	86.3	12.57	78.6
Methylparathion	16.20	101.3	15.61	97.6
Heptachlor	13.92	87.0	14.39	89.9
Di-n-butylphthalate	16.26	95.4	16.35	102.2
Aldrin	14.30	89.4	14.65	91.6
Bis(2-ethylhexyl) adipate	14.93	93.3	15.66	97.9
Butyl benzyl phthalate	15.13	94.6	15.81	98.8
Chrysene	14.86	92.9	14.94	93.4
3, 3’-Dichlorobenzidine	13.70	85.6	13.84	86.5
Bis(2-ethylhexyl) phthalate	16.55	103.4	15.34	95.9
Di-n-octyl-phthalate (CCC)	14.87	92.9	15.06	94.1

Conclusions

The tests done here show the DryDisk membranes perform very well in all the aspects considered. They remove water, even when a significant percentage of a water soluble solvent is mixed with the nonsoluble solvent used for extraction. In addition, the analytes show excellent recovery with both the DryDisk and DryDisk-R. The DryDisk was further compared to sodium sulfate to see if recoveries could be distinguished and base compounds pass through the DryDisk, while they show significant loss on sodium sulfate.

DryDisk membranes are a useful way to quickly remove water from organic extracts and do not add contamination or retain analytes. In addition, because the separation is physical and not chemical the amount of water that can be removed is unlimited.

Reference

1. Susan Petitti, A Study of DryDisk Background Contamination, AN051-091214.

Literature Number: UI405