

Evaluation of Evaporative Sample Preparation Techniques

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Introduction

When introducing any new piece of instrumentation or equipment into a highly controlled environment such as an environmental analysis laboratory, the new unit should be validated to ensure that it has equivalent or better performance to the existing equipment. Any impact on the testing methods can then be fully understood and the testing methods re-validated if necessary. With regard to sample concentration and evaporation technology the most important issue is sample recovery, especially for very volatile analytes. As part of their evaluation of new equipment Laboratoire départemental d'analyses de la Drôme (LDA26) evaluated the new Genevac EZ-2 and compared it to two existing methodologies. A summary of the data is presented in this report.

Performance Benchmarking of Genevac EZ-2

Samples of pesticides were spiked (20mg/litre) into 50ml of a 50:50 volume:volume mixture of dichloromethane (DCM) and acetone. Each sample also had a drop of pentanol added as a solvent keep. Samples were evaporated using a Genevac EZ-2 (Figure 1) at 35°C to leave the sample concentrated in the drop of pentanol. The procedure follows a special environmental testing evaporation methodology developed by the Environmental Protection Agency of Tuscany¹ which preferentially evaporates the volatile solvents to leave just the less volatile solvent. Following evaporation the samples were made up to 1ml with ethyl acetate and injected into a Gas Chromatograph using an Electron Capture Detector (GC-ECD) for analysis. The recoveries are reported in Figure 2.

Figure 1 – Genevac EZ-2



Comparison of Genevac EZ-2 to existing equipment

Following on from this initial evaluation the EZ-2 was benchmarked against two existing evaporation methods traditionally employed at LDA26, the Zymark TurboVap and simple air evaporation in the fume hood.

Samples of the volatile environmental analytes were spiked (50mg/litre) into 50ml of a 50:50 volume:volume mixture of DCM and acetone. Each sample also had a drop of pentanol added as a solvent keep. Samples were evaporated using the Genevac EZ-2 as before, or the Zymark TurboVap at 35°C to leave just the drop of pentanol. Following evaporation the samples were made up to 1ml with ethyl acetate and dried. Those that had been initially dried in the Genevac EZ-2 were dried in the EZ-2 again.

Figure 2 – Percentage recovery of various analytes prepared in Genevac EZ-2
 % = Percentage Recovery SD = Standard Deviation

Analyte	%	SD	Analyte	%	SD	Analyte	%	SD
123TCB	83	9	HCB	101	11	pyrimiphos ethyl	110	17
124TCB	131	16	indeno 123cd pyrene	104	5	pyrimiphos methyl	115	2
135 TCB	105	28	malathion	100	8	quinalphos	106	6
2 methyl fluoranthene	102	4	metazachlore	110	15	quintozene	100	10
2 methyl naphtalene	94	15	methydathion	109	7	tetrachlorobenzene	110	12
acenaphtene	101	4	naphtalene	85	13	triadimefon	120	9
anthracene	92	5	op'DDD	96	8	triazophos	146	5
benzo a anthracene	90	4	op'DDE	95	8	vinchlozoline	100	9
benzo a pyrene	107	6	oxychlorane	105	1	diazinon	104	2
benzo b anthracene	102	4	penchlorobenzene	95	11	chlorpyriphos methyl	102	2
benzo ghi perylene	109	6	phenanthrene	101	28	parathion methyl	103	7
benzo k anthracene	116	5	phorate	102	5	fenitrothion	105	10
chlorothalonil	71	0	phorate oxon	94	13	chlorpyriphos ethyl	102	8
chrysene	101	5	phorate sulfone	112	7	parathion ethyl	89	10
dibenzo ah anthracene	108	5	phosalone	114	6	bromophos methyl	93	8
dimethoate	116	7	phosmet	106	12	chlorfenvinphos	118	21
disulfoton	105	8	pp'DDD	92	8	bromophos ethyl	83	14
disulfoton sulfone	113	7	pp'DDE	93	8	carbophenothion	95	1
fenthion	110	8	pp'DDT	93	8	azinphos methyl	132	20
fluoranthene	94	11	propetamphos	109	7	azinphos ethyl	107	9
fluorene	93	14	pyrene	117	6	mevinphos	103	4

at 35°C and those that had been initially dried in the TurboVap were air dried in the fume hood. Samples were then made up with water and acetonitile adjusted to pH 2 and injected into a gradient HPLC system using a multi wavelength fluorescence detector (HPLC-Fluor) for analysis. The recoveries are reported in figure 3.

Figure 3 – Percentage recovery of volatile analytes prepared by two stage evaporation using different methods

Analyte	Genevac & Genevac		TurboVap & Fume Hood	
	Test 1	Test2	Test 3	Test 4
naphtalene	75	79	28	21
2 methyl naphtalene	86	87	40	35
acenaphtene	101	101	50	47
fluorene	100	100	51	50
phenanthrene	119	120	69	61
anthracene	93	94	56	51
fluoranthene	103	104	74	64
pyrene	120	121	86	76
2 methyl fluoranthene	103	105	83	73
benzo a anthracene	92	93	80	73
chrysene	102	102	88	81
benzo b anthracene	105	105	96	87
benzo k anthracene	121	120	110	100
benzo a pyrene	105	105	93	82
dibenzo ah anthracene	122	120	112	100
benzo ghi perylene	107	105	96	86
indeno 123cd pyrene	110	110	102	91

An additional set of tests were performed using 1ml samples of ethyl acetate which had been spiked (50mg/litre) with a range of different volatile analytes. Each sample had a drop of pentanol added as a solvent keep. These samples were evaporated using the Genevac EZ-2 at 35°C or left to air dry in the fume hood. Following evaporation the samples were made up to 1ml with water and acetonitrile, adjusted to pH 2, and injected into the HPLC-Fluor system for analysis. The results are shown in Figure 4.

Figure 4 – Percentage recovery of volatile analytes evaporated by different methods

Analyte	Genevac		Fume Hood	
	Test 1	Test 2	Test 3	Test 4
naphtalene	93	92	66	61
2 methyl naphtalene	91	92	79	78
acenaphtene	103	106	95	97
fluorene	103	107	96	99
phenanthrene	120	124	112	116
anthracene	96	99	89	92
fluoranthene	101	104	95	98
pyrene	122	126	113	116
2 methyl fluoranthene	101	106	95	98
benzo a anthracene	91	94	85	86
chrysene	99	104	95	96
benzo b anthracene	101	105	96	97
benzo k anthracene	115	120	110	111
benzo a pyrene	106	110	100	100
dibenzo ah anthracene	109	113	103	105
benzo ghi perylene	110	115	104	104
indeno 123cd pyrene	104	109	100	101

Conclusions

The new method of evaporation using the Genevac EZ-2 delivers excellent sample recoveries and in the case of the most volatile environmental analytes provides enhanced recovery over the pre-existing methods of evaporation tested. In the EZ-2 vacuum evaporation causes the solvents to boil at a low temperature keeping the samples at the boiling point of the solvents. By comparison when using the other methods under test the samples are kept at the elevated temperature of 35°C for the duration of the evaporation process. This increased temperature of operation will increase the evaporation rate of both the solvents and the most volatile components in the sample. By careful control of evaporation conditions in the EZ-2, the solvents can be preferentially removed leaving the analytes and pentanol behind.

References

1. Marsico, Anna Maria, 2006. Improving Analysis of Pesticides – a new method development protocol to increase recovery of volatile compounds. First published in Lab Asia, August 2006 & available via <http://www.genevac.org/en/ArticleDetail.asp?S=6&V=1&ProductDownload=81>

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