Evaluation of a Novel, Large Volume Evaporative Sample Preparation Technique

Mr Felix Massat, Benoît Planel, & Antoine Venezia
Laboratoire départemental d'analyses de la Drôme, Valence, France

Introduction

When introducing any new piece of instrumentation or equipment into a highly controlled environment such as an environmental analysis laboratory, the new unit must evaluated 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 Rocket evaporation system and its ability to prepare large volume samples containing volatile analytes. A summary of the data is presented in this report.

Rocket Evaporation System

The Rocket evaporation system is a new parallel evaporation system which evaporates up to six flasks each containing up to 400ml in parallel. The system uses vacuum to evaporate the samples, which spin in a centrifuge to ensure that there is no sample loss or cross contamination due to "bumping". The samples spin in a low pressure, low temperature steam bath, which provides precise, even and very high energy heating, making sample concentration very fast. [1] The Rocket evaporator has two styles of flask, a traditional bottle shaped design, and SampleGenie™ style flasks which act like a funnel and permit the large volume sample to be concentrated directly to a gas chromatograph (GC) autosampler vial. Speed an reduction of sample transfer steps are desirable in a sample preparation method to help streamline workflow in the laboratory.

Figure 1 – Genevac Rocket

Evaluation of Rocket and Comparison to existing equipment

Samples of the volatile environmental analytes were spiked (50mg/litre) into 100ml of a 50:50 volume:volume mixture of DCM and acetone. Each sample also had a 50µl drop of pentanol added as a solvent keep. Samples were evaporated in SampleGenie style flasks in the Genevac Rocket set at 35°C using the HPLC FRACTION method, to leave just the drop of pentanol. The Rocket is designed to stop the evaporation automatically at this point. Following evaporation the samples were made up to 1ml with a 50:50 volume:volume mixture of water and acetonitrile 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 2.

Figure 2 – Percentage recovery of volatile analytes prepared in Genevac Rocket
SD = Standard Deviation

	Assay 1	Assay 2	Assay 3	SD
Naphthalene	82	94	92	6.43
Methyl 2 naphthalene	92	92	96	2.31
Acetonaphthene	95	94	97	1.53
Fluorene	96	99	105	4.58
Phenanthrene	104	96	99	4.04
Anthracene	101	96	101	2.89
Fluoranthene	99	100	101	1.00
Pyrene	103	97	97	3.46
Methyl 2 fluoranthene	99	102	102	1.73
Benzo a Anthracene	101	98	100	1.53
Chrysene	100	101	101	0.58
Benzo b Fluoranthene	102	104	103	1.00
Benzo k Fluoranthene	98	104	103	3.21
Benzo a Pyrene	104	97	99	3.61
Dibenzo ah Anthracene	100	100	101	0.58
Benzo ghi Perylene	101	104	100	2.08
Indeno 123 cd Pyrene	101	104	103	1.53

By way of comparison these results were compared to earlier work done evaluating another vacuum evaporation system for multiple small volumes with an alternative method in use at LDA26, the TurboVap. The samples were of 50ml volume, and prepared and analysed similarly to those in the Rocket[2], results are shown in figure 3.

Figure 3 – Percentage recovery of volatile analytes prepared by two stage evaporation using different methods, Genevac EZ-2 and TurboVap

	Genevac EZ-2		**TurboVap**	
Analyte	**Test 1**	**Test2**	**Test 3**	**Test 4**
naphthalene	75	79	28	21
2 methyl naphthalene	86	87	40	35
acetonaphthene	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

Conclusions

The new method of evaporation using the Genevac Rocket 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 Rocket, 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 Rocket, the solvents can be preferentially removed leaving the analytes and solvent keep behind.

References

1. Lee-Smith, Roger, 2008. Development of a Novel Parallel Evaporator. First Published in SP2, Vol. 7, No. 39, November, pp 38-39, and also available via www.Genevac.com/rocketmovie
2. Massat, F, Planel, B & Venezia, A, 2007, Evaluation of Evaporative Sample Preparation Techniques. First published in International Environmental Technology, March/April 2008, pp 36, and also available via http://genevac.org/en/ArticleDetail.asp?S=6&V=1&ProductDownload=134

About the Authors

Mr Felix Massat is head of the Laboratory; Benoît Planel is a Chemist; and Antoine Venezia is head of Research and Development; all at: Laboratoire départemental d'analyses de la Drôme, Valence BP118, 26904F-91, France