# Evaluation of Evaporative Sample Preparation Techniques

***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.

| 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 | 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 |

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

*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 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.

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.

| 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 |

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

| Analyte | Genevac | | Fume Hood | |
|---|---|---|---|---|
| | Test 1 | Test2 | 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 |

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

## 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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