

Concentration of Samples Complying to US EPA 8082 Parallel evaporation technology for high solvent and analyte recoveries

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Introduction

US EPA method 8082 is used to determine the concentration of polychlorinated biphenyls (PCBs) in extracts prepared from many types of solid, tissue, and aqueous matrices. A solvent exchange from dichloromethane (DCM) to *n*-hexane is required so the sample can be efficiently analyzed through a gas chromatography / electron capture detector (GC / ECD).

A reproducible concentration of surrogate compounds 2,4,5,6-tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB), is necessary to obtain accurate data of the testing compounds, Aroclor 1016 and Aroclor 1260. When performing concentrations in compliance with US EPA 8082 it is important to comply with the EPA's "Emissions Reduction Program."

A collaboration with BUCHI Corporation and Accutest[®] Laboratories was developed to optimize analyte and solvent recoveries for US EPA 8082. Accutest[®] Laboratories is one of the nation's largest environmental testing laboratories, combining advanced technology and experienced personnel to deliver "Total Performance" You Can Count On." The BUCHI Syncore[®] Analyst, a parallel vortex evaporator equipped with a Solvent Vapor Recovery system with integrated control (SVR-N), was used to evaporate up to 12 samples simultaneously. A programmed pressure and time profile optimized for a DCM and *n*-hexane mixture eliminates the otherwise required solvent exchange, which drastically saves time and yields high analyte recoveries.

Experimental set-up





Recirculating chiller

Syncore[®] parallel vortex evaporator

Method 3500C summary of methods; section 2.2

"The resultant extract is dried and concentrated in a Kuderna-Danish (K-D) apparatus. Other concentration devices or techniques may be used in place of the Kuderna-Danish concentrator if they give acceptable results for the intended application."

"NOTE: Solvent recovery apparatus is recommended for use in methods that use Kuderna-Danish or other evaporative concentrators. EPA recommends the incorporation of this type of reclamation system as a method to implement an emissions reduction program."

Source: http://www.epa.gov/osw/hazard/testmethods/sw846/pdfs/3500c.pdf



Due to the cooled appendix technology the sample is automatically concentrated to the predefined volume of either 0.3, 1 or 3 mL ($A \rightarrow B$). The appendix is actively cooled by the BUCHI Recirculating Chiller. After concentration the sample is transferred to the sample vial ($C \rightarrow D$).



Concentration

Flushback effect

The Flushback module partially condenses the solvent vapor at the top of the sample vessel generating a continuous rinsing along the glass wall. With this, adsorption of analytes at the glass wall is avoided and high analyte recovery rates are obtained.





Application specific on-site support is offered to effectively prepare your environmental sample.

An environmental benign sample concentration process is performed by applying the follwing parameters and a programmed vacuum gradient.

| Solvent | DCM |
|------------------|--------|
| Starting volume | 110 ml |
| Final volume | 10 mL |
| Heating plate | 60 °C |
| Cover temp. | 60 °C |
| Cooling temp. | 10 °C |
| Orbital movement | 250 rp |
| | |

Twelve samples are concentrated in parallel using the Syncore[®] Analyst R-12 in only 28 minutes, *i.e.*, 2:20 minutes per sample. The solvents, dichloromethane and *n*-hexanes, are recovered at 95% efficiency without the use of nitrogen.

Vacuum source





Analytical results

Spiked (0.5/0.05 ng) and concentrated laboratory control samples were analyzed by GC/MS for several volatile surrogate compounds and target analytes found in US EPA 8082.

| | Analytical Method | CAS Number Equivalent | Parameter Name | Spike Level (ng) | Perc Recc LCS | | | |
|---------------------------------------------------|----------------------|--------------------------|---------------------------------|------------------------|---------------------|--|--|--|
| | | | Surrogate Compounds | | | | | |
| | SW8082 | 877-09-8 | % Tetrachloro- <i>m</i> -xylene | 0.05 | 86 | | | |
| | SW8082 | 2051-24-3 | % Decachlorobiphenyl | 0.05 | 134 | | | |
| | | | | | | | | |
| | | | Target Analytes | | | | | |
| | SW8082 | 12674-11-2 | Aroclor 1016 | 0.5 | 94 | | | |
| | SW8082 | 11096-82-5 | Aroclor 1260 | 0.5 | 124 | | | |
| * Elevated % recovery is due analysis uncertainty | | | | | | | | |
| | | | | | | | | |

Conclusion

- High solvent recovery > 95%
- High analyte recovery for PCBs
- Excellent reproducibility
- Compliance with waste minimization and pollution prevention
- biphenyls, and 8015 nonhalogenated organics



Percent

2:1

Automation and intensification —> cost reduction Also applicable for US EPA 8270/625 SVOC, 8081A, B/608 organochlorine pesticides, 8082/608 polychlorinated

| tes | | | | | | | | |
|--------------|-----|-----|-----|-----|-----|--|--|--|
| | 0.5 | 94 | 96 | 86 | 86 | | | |
| | 0.5 | 124 | 113 | 107 | 109 | | | |
| suncertainty | | | | | | | | |

Spike Percent Percent Recovery Recovery

2:1

114

Level Recovery Recovery LCS #3/ LCS #4/

LCS #2

120

Time / min

LCS #1

Vacuum gradient