Automated Sample Cleanup system – for Pesticides and Organic Metabolites in Environmental and Food Samples

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For the past 14 years the PowerPrep™ system has built a reputation as a reliable and efficient analytical instrument for providing analytical laboratories automation solutions for the extraction and cleanup phase of analysis.

The most widely used version of the Power-Prep is the Power-Prep/Dioxin™, used in the analysis of Dioxins/PCB's.

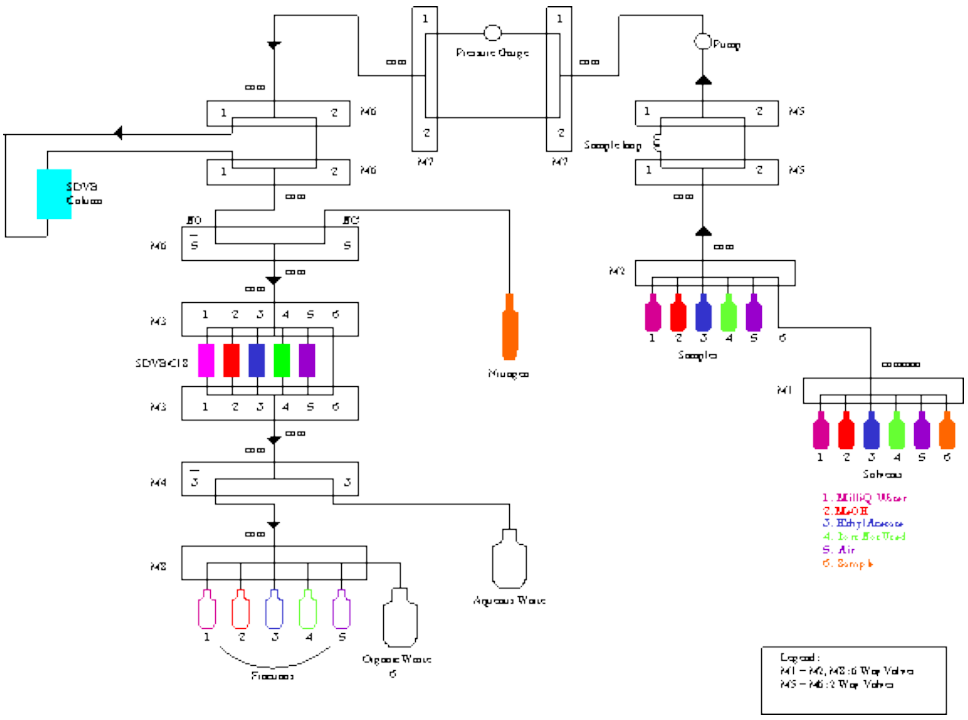
Because of the growing interest in food safety and soil quality for food product and animal feed, the analytical market has a need to test for contaminants other than PCBs and Dioxins, including pesticides, phthalates, poly-aromatic hydrocarbons etc.

It is known that PCBs and Dioxins can be found in fats, lipids and oil materials due to their hydrophobic properties, but different types of pesticides (hydrophilic and hydrophobic) can be found either in lipids or aqueous type materials. The assessment of the potential hazards of pesticides is closely associated with the identification and determination of the present concentrations. Therefore, developing an analytical method that is capable of detecting the traces of pesticides is needed to meet the objectives for the surveillance and monitoring of the present concentrations. [2]

The number of pesticides (chlorinated and non-chlorinated) cannot be separated from the fatty background with acidic media, which is typical for PCB and/or Dioxins. Many of these pesticides are unstable and would be degraded by acid.

An alternative approach is to do the separation using the Power-Prep/GPC-SPE system. This system is a combination of the Gel Permeation Chromatograph and Solid Phase Extraction and it is designed and modeled based on traditional Power-Prep/Dioxin configuration.

Fig 1. PowerPrep™ Automated GPC-SPE system .



The system can perform both GPC (US EPA 3640B) and SPE (methods based on the US EPA Method 3535B and its modification, depending on application requirements)

GPC-SPE

GPC cleanup is performed by introducing 9 mls of the standard mixture of pesticides (tetra-chloromethaxylene, a,b,g-BHC, Heptachlor, Heptachlorepoxide, Endrin, EndrinAldehyde, EndrinKetone, p,p,'-DDD, p,p'-DDE, p,p'-DDT, Endosulfan, EndosulfanKeton, Methoxichglor, decachlorobyphenyl)which has been spiked in 25% solution of corn oil in dichloromethane.

After a short operation of rinsing the sample loop and tubes, and removing excessive bubbles of air, an exact amount of 5 ml of the spiked solution is introduced into the sample loop.

The auto sampler is not a necessary part because the extract is introduced to the loop by suction.

After concentrating the target fraction and solvent exchange the sample was tested both on GC/ECD and GC/MS.

TCMX 90%

Lindane 93%

α-HBC 85%

β-HBC 87%

δ-HBC 89%

Dieldrin 94%

Endrin 94%

Endrin Aldehyde 87%

Endrin Ketone 90%

pp'- DDE 92%

pp'- DDD 89%

pp'- DDT 94%

Heptachlor 91%

Heptachlor Epoxide 86%

Mirax 87%

Methoxichlor 79%

DCBP 84%

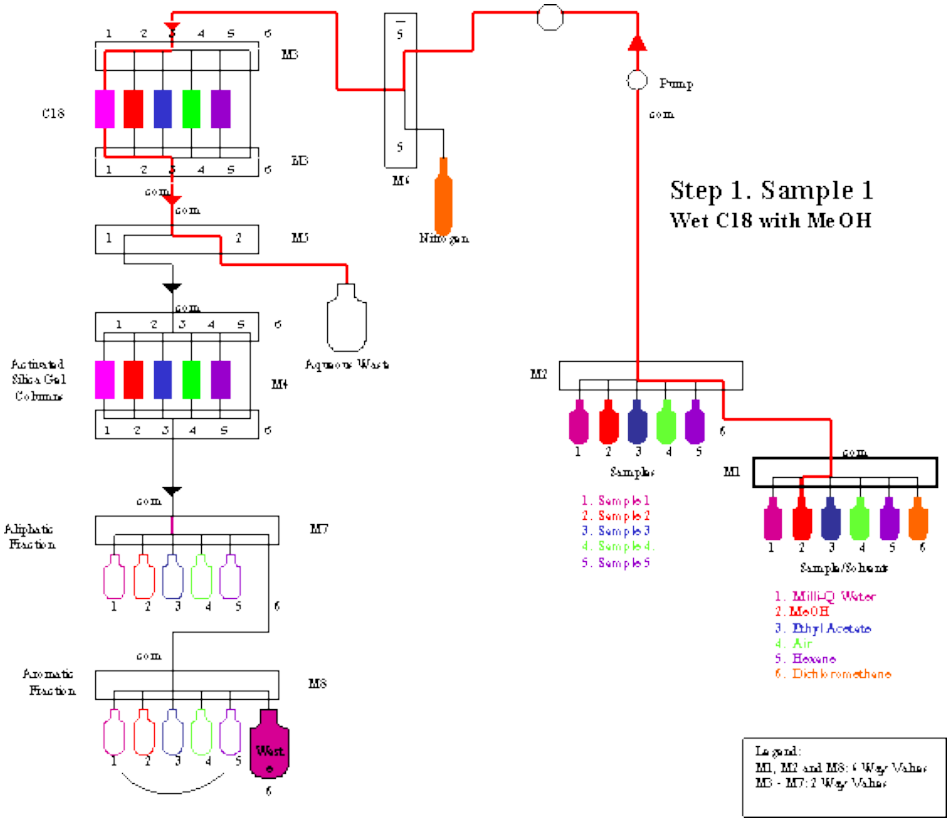
Power-Prep/SPE.

Power-Prep/SPE is designed to perform either extraction and/or fractionation of extracts.

The automated PowerPrep™ system has been evaluated for SPE extraction and clean-up of 32 pesticides and

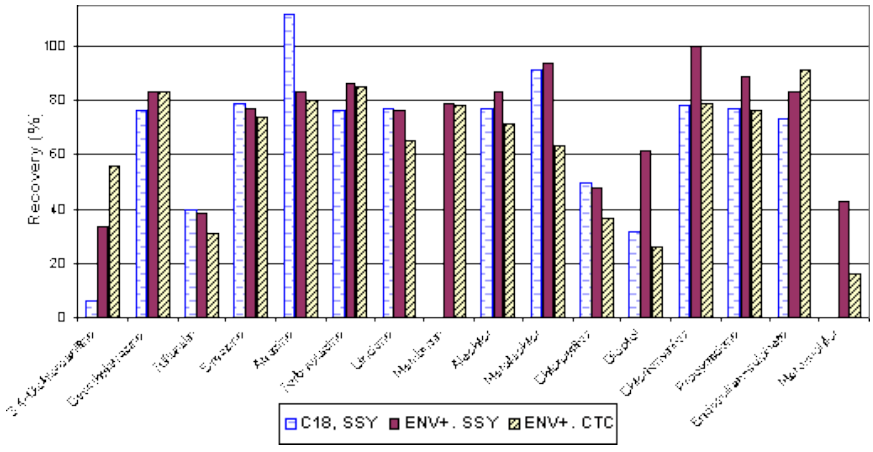
metabolites. The optimized method was applied to the analysis of surface water samples using d_5-atrazine and d_{10}-parathion as internal standards for isotope dilution-GC/MS analysis. [3]

Fig. 2.PowerPrep™ SPE-SPE extraction and Clean-up System



The highest recoveries were obtained for the styrene-divinylbenzene cartridge, with 1% of methanol at a flow rate of 5mL/min, and using a piston pump. The 12 samples of surface water were tested and The results related to 16 priority and suspected endocrine disrupting pesticides are summarized in figure 2. [2]

Figure 2. Automatic SPE extraction: Sorbent phase and Pump material.



The presented configuration is also applicable to the fractionation of PAH's (extractable petroleum hydrocarbons, Extractable Petroleum Hydrocarbons (EPH) method).

References:

[1] US EPA SW-846

[2] Analysis of some Chlorinated Pesticides in Jordanian Ground

and Surface Waters by SolidPhase Extraction and Mass Spectrometric Detection

A Method Development. Lara Shahin. *Department of Water and Environmental Studies*

Linköping University, Sweden. August 2004.

[3]Automated Solida-Phase Extractio of Priority and Suspected Endocrine Disrupting Pesticides And Metabolites. C.Planas, J.Sauló, J.Rivera, J.Caixach, ass Spectrometry Lab., Dept. of Ecotechnologies, IIQAB-CSIC