Application Note No. 013

TRIAZINES WITH ON-LINE COUPLING SPE-GC

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Purpose:

Solid -phase extraction is a powerful technique for the clean-up and trace enrichment of samples before GC or GC-MS. Unfortunately, up until recently, its use with GC was strictly limited to off-line mode, since no standard instrumentation for hyphenated SPE-GC was commercially available.

This note presents a fully automated SPE-GC system. The PROSPEKT for automated SPE is interfaced to the GC using the OPTIC2 injector for large volume injection.

Equipment:

The system (see fig. 1) used is a HP6890 equipped with a FID detector. The SPE system used is a PROSPEKT (Spark Holland) and a solvent delivery system with a syringe pump.
For the large volume injection a OPTIC2 injector is used as interface between GC and SPE.

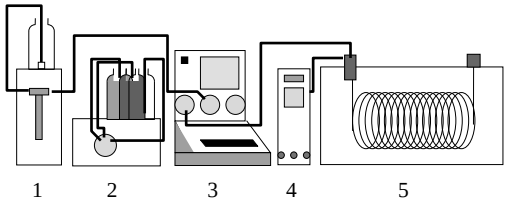

Fig. 1: System used in our experiments. 1:syringe pump, 2:solvent delivery system, 3:PROSPEKT, 4:OPTIC2, 5:HP6890 GC

The column in the GC oven is a 15m DB-1 x 0.32 mm i.d. and a film thickness of 0.25µm.

Method development:

First:
The triazines were analysed on the GC to examine the right GC conditions for this application.

Second:
Optimize the parameters for the large volume injection with the OPTIC.
- Establish the maximum volume to be injected.
- The solvent venting time.
- Temperature programming.

Third:
Optimize the method on the PROSPEKT SPE.
- Optimize the drying time of the cartridges.
- Optimize the washing steps of the cartridges.

Fourth:
Combine SPE with GC. Important steps are:
- The internal volume of the line between SPE-GC.
- The rate of the eluent to the cartridge and into the GC.
- Internal volume of tubing at the valves.

Results and discussion:

The valve connections on the PROSPEKT to do on-line coupling SPE-GC is shown in figure 2.

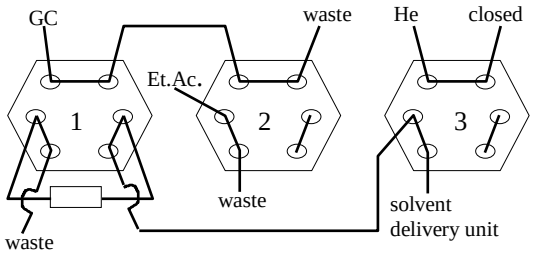

Fig. 2: PROSPEKT valve configuration for injection into a GC with OPTIC2 injector.

Method SPE:

- The cartridge is solvated with Acetonitril
- The cartridge is equilibrated with water
- The sample is loaded on the cartridge for 5 minutes with 3 ml/min.
- The cartridge is dried for 15 min with Helium
- The sample is eluted with 200µl Ethylacetate at a rate of 3 ml/min.

The concentration of the sample is 8 ppb.

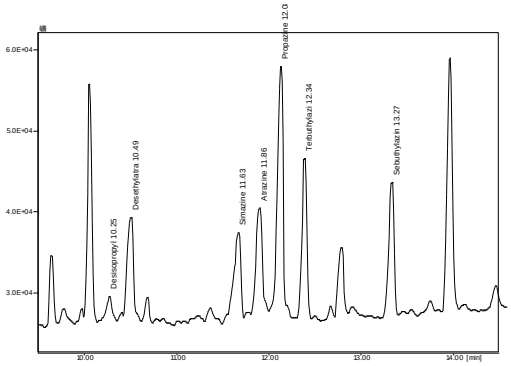

Figure 3: On-line SPE-GC chromatogram of triazines (8 ppb); Desisopropyl 10.23, Desethylatrazine 10.48, Simazine 11.63, Atrazine 11.86, Propazine 12.08, Terbuthylazine 12.35, Sebuthylazine 13.27

This eluate is immediately transferred to the GC via a capillary column. In the OPTIC2 injector it is held on a Supelcoport packed liner on 45°C. The solvent is evaporated and vent away by the special vent exit.
By temperature programming of the liner, the analytes are transferred to the GC column.

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The resulting chromatograms are given in figure 3 and 4.

Component	Recovery (%)
Desisopropyl atrazine	-[(a)]
Desethyl atrazine	100
Simazine	85
Atrazine	88
Propazine	100
Terbuthyl azine	91
Sebuthyl azine	94

Table 1: Recovery of triazines.
[(a)] Data not available due to co-elution
With impurity in ethyl acetate

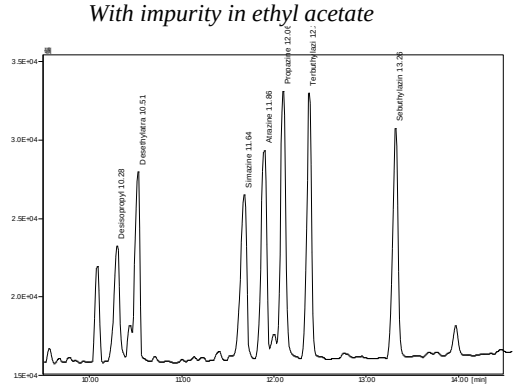

Figure 4: 50 µl injection of triazines on the GC.
Desisopropyl 10.28, Desethylatrazine 10.51, Simazine 11.64, Atrazine 11.86, Propazine 12.06, Terbuthylazine 12.33, Sebuthylazine 13.26

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