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.

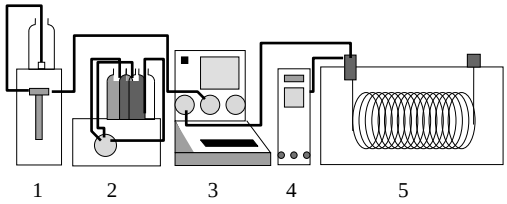

Fig. 1: System used in our experiments. 1:syringe pump, 2:solvent delivery system, 3:PROSPEKT, 4:OPTIC2, 5:HP6890 GC

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.

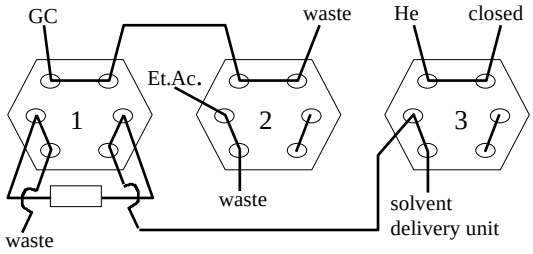

Fig. 2: PROSPEKT valve configuration for injection into a GC with OPTIC2 injector.

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.

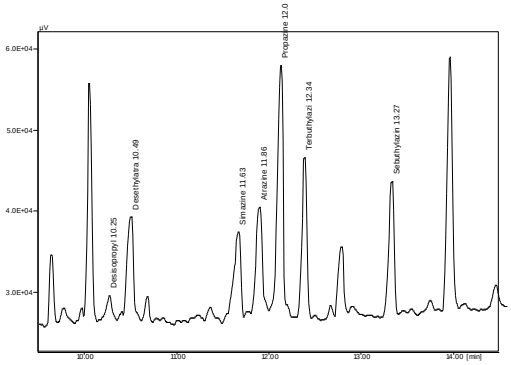

Figure 3: On-line SPE-GC chromatogram of triazines (8 ppb); Desisopropyl 10.23, Desethylatrazine 10.48, Simazine 11.63, Atrazine 11.86, Propazine 12.08, Terbuthylazine 12.35, Sebuthylazine 13.27

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.

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

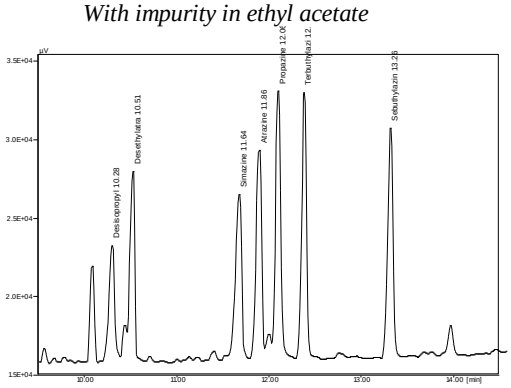

Figure 4: 50 µl injection of triazines on the GC.
Desisopropyl 10.28, Desethylatrazine 10.51, Simazine 11.64, Atrazine 11.86, Propazine 12.06, Terbuthylazine 12.33, Sebuthylazine 13.26