FIELD APPLICATION REPORT

Gas Chromatography

Analysis of Organophosphorus and Organonitrogen Herbicides in Water using GC/MS with Selected Ion Recording

Introduction

Laboratories monitoring trace level pesticides and herbicides by gas chromatography/mass spectrometry (GC/MS) frequently utilize Selected Ion Recording (SIR) techniques to maximize analytical sensitivity for quantification. With analytical standards, precision is limited to a large extent only by the autosampler and other instrumental effects. However, real-world samples pose a greater challenge. In this latter case, matrix effects have a detrimental effect on both sensitivity and precision.

Often, such analyses are carried out using on-column injection, as classical (hot) splitless injection can result in the loss of thermally labile species. The practical disadvantages of on-column injection usually manifest themselves in the form of degraded separation and peak tailing as the retention gap rapidly becomes contaminated with matrix components. Chromatographic performance may be restored by cleaning or replacement of the retention gap, but this is time consuming and requires a significant level of operator skill.

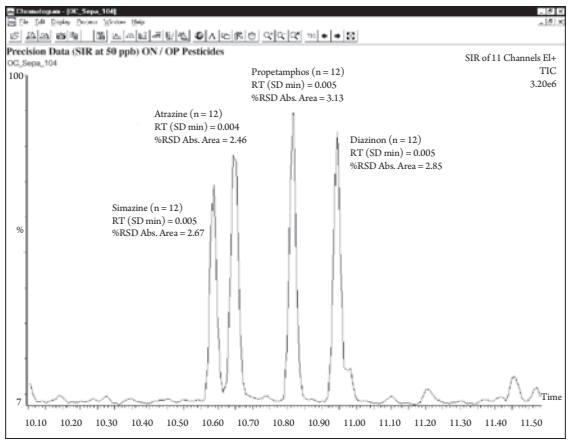

Figure 1. SIR chromatogram of herbicides in water.

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In programmed splitless injection the sample is introduced into a cool (quartz) injector liner packed with quartz or silanized glass wool. The packed liner has a much greater sample capacity than the retention gap used in on-column injection methods. After injection, the temperature of the injector is rapidly heated to transfer the analytes to the head of the chromatographic column. This approach exposes the sample to substantially milder conditions than the flash vaporization used in classical splitless injection. It eliminates possible decomposition caused by contact of the analytes with a heated metal syringe. Another advantage of this approach is that the same experimental setup may also be used for large-volume injection with solvent purging.

Results

In the following example, a spiked (50 ppb) river water extract has been analyzed 12 times using programmable splitless injection. The chromatographic conditions are listed in Table 1, and a typical SIR chromatogram is shown in Figure 1. These 12 replicate injections were made after some 40 to 50 previous injections into the same liner. Some of these earlier injections involved large-volume injection techniques. Despite this, the precision data in Table 2 shows no trends, and the quality of the chromatographic separation is unchanged throughout the test.

Table 1. **Instrument Conditions:** The following conditions can be used with any recent model PerkinElmer® GC/MS system (Autosystem XL/TurboMass Gold or Clarus® 500 GC/MS).

Gas Chromatograph	
Column	Elite-5ms; 30 m x 0.25 mm ID x 0.25 µm film thickness (5%-Diphenyl)
Carrier	Helium, constant flow at 1.0 mL/min
Oven temperature	60 °C for 3 min, 25 °C/min to 200 °C, then 4 °C/min to 260 °C, hold for 6.4 min
PSS (Programmable Split/Splitless)	60 °C initial, 100 °C/min to 260 °C injector temperature
Split flow	0 mL/min at -0.5 minutes, 50 mL/min after 3 minutes
Mass Spectrometer	
Acquisition mode	Selected Ion Recording
Masses monitored	137, 138, 173, 179, 186, 194, 200, 201, 215, 236, 304
Dwell time	0.04 sec
Solvent delay	5 minutes
Transfer line temperature	225 °C
Ion source temperature	150 °C

Table 2. **Herbicide Compound Summary Report:** Replicate injections of river water spiked with 50 ppb of herbicides.

	Simazine (m/z 201)		Atrazine (m/z 200)		Propetamphos (m/z 194)		Diazinon (m/z 179)	
	Ret. Time	**Abs. Area**	**Ret. Time**	**Abs. Area**	**Ret. Time**	**Abs. Area**	**Ret. Time**	**Abs. Area**
	10.587	15491	10.651	22801	10.817	14653	10.945	22237
	10.596	15040	10.661	23339	10.826	15385	10.954	20246
	10.587	15167	10.652	23914	10.817	14746	10.945	20937
	10.587	15505	10.652	23264	10.826	15055	10.955	21302
	10.587	14726	10.651	23572	10.817	15206	10.954	21991
	10.587	15281	10.651	23449	10.826	15805	10.945	21662
	10.587	15093	10.652	24337	10.817	15143	10.955	22422
	10.597	15784	10.652	23709	10.826	15611	10.955	22216
	10.587	15439	10.651	23970	10.817	16119	10.945	21941
	10.587	15537	10.651	23623	10.826	14768	10.954	22040
	10.587	16306	10.652	24893	10.817	15715	10.945	21877
	10.578	15743	10.643	24522	10.817	15790	10.945	21775
Mean	**10.588**	**15426**	**10.652**	**23783.24**	**10.821**	**15333**	**10.950**	**21721.04**
SD	**0.005**	**411**	**0.004**	**584.17**	**0.005**	**479**	**0.005**	**619.48**
RSD %	**0.05**	**2.67**	**0.04**	**2.46**	**0.04**	**3.13**	**0.05**	**2.85**

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