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Automatic Liner Exchange (ALEX) to Deal with Dirty Matrices Using the GC/Q-TOF

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Introduction

The GC-MS analysis of pesticides in dirty matrices is challenging because the non-volatile components collect in the inlet liner causing gradual deterioration of chromatographic performance such as tailing peak shapes and irreproducible response. This can be due to active sites forming on the liner due to the build-up of the non-volatile components. A set of coriander QuEChERS extracts with no clean up were supplied from FERA Science Ltd. A set of pesticides were spiked at various concentrations from 10 ng/l to 200 ng/l into the extracts.

The Cool Inlet System (CIS) is a Programmable Temperature Vaporiser which allows samples to be injected cold. The CIS can then be heated rapidly with a controlled temperature ramp to vent the solvent and then volatilize the pesticides onto the GC column.

The GERSTEL Automated Liner EXchange (ALEX) technology automates the task of replacing liners in the CIS. A whole series of liners can be changed within a given sequence of analysis.

The Agilent 7200 is a GC accurate mass QuadrupoleTime of Flight Mass Spectrometer (GC/Q-TOF) which is shown below in Figure 1. Mass accuracy is typically 5ppm. By extracting a known accurate mass with a narrow mass window e.g. 20ppm from the total ion current (TIC) improved signal:noise and improved selectivity is observed when compared to a single quadrupole instrument.

Figure 1 shows the set up for ALEX in our laboratory.

Figure 1 – ALEX set up on the GC/Q-TOF

Figure 2 shows a close up of how ALEX works. The liner with a transport adaptor is moved into the CIS using the GERSTEL Gripper. The liner is then locked in position. Within the transport adaptor, there is a septa and QuEChERS extracts are injected directly cold and then rapidly heated using the CIS inlet.

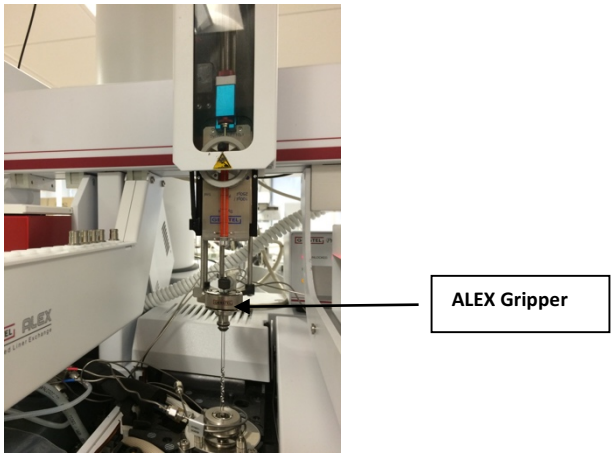


Figure 2 – MultiFlex GC/Q-TOF with DHS and Static Headspace at Anatune

During the sequence, the liner can be changed without any human intervention. Figure 3 shows a photo of a liner after only a few injections. This highlights the need to change the liner during the sequence.

Figure 3 – Dirty liner after a few injections of the dirty QuEChERS extracts

Instrumentation

Dual Head GERSTEL MPS 2
GERSTEL Automatic Liner Exchange (ALEX)
Agilent 7890 GC with a 7200 GC/Q-TOF
Agilent Mass Profiler Professional

Method

Linearity and precision experiments were performed on dirty QuEChERS extracts. The liner was changed automatically using ALEX after every 5 injections. Figure 4 shows a photo of the dirty QuEChERS extract.

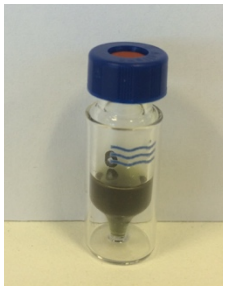


Figure 4 – Dirty QuEChERS extract

CIS conditions

Program guide:	10 °C ramped to 240 °C
Liner exchange:	ALEX (used after every 5 injections)
Liner:	Baffled liner
Inlet mode:	Splitless

Results

No deterioration in chromatography was observed due to frequent changing of the liners. Linearity was performed on some of the pesticides that had been spiked. Figure 5 shows linearity observed for Clomazone.

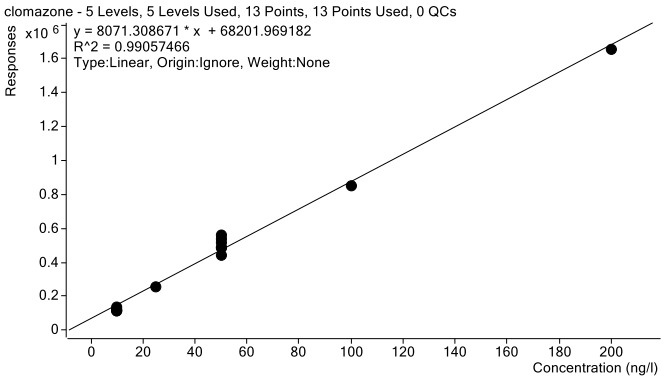


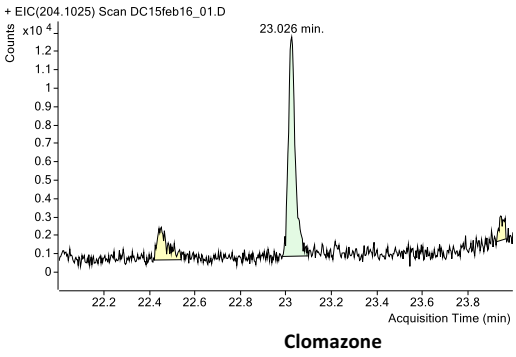
Figure 5 – Shows linearity observed for Clomazone

Table 1 shows precision for Clomazone without internal standard. ALEX makes no fundamental change to the inlet so you should expect to see no change in precision compared to not using the liner exchange.

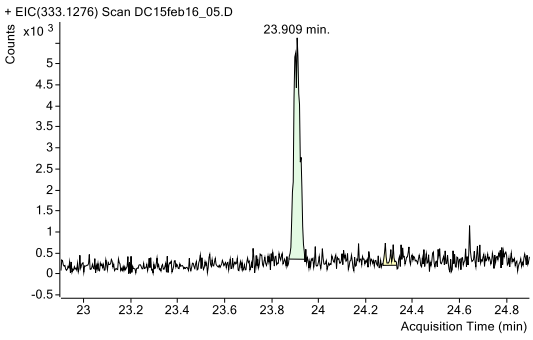
Description	Calc. Conc. ng/l	Accuracy %
50 ppb	57.9	116
50 ppb	60.6	121
50 ppb	55.2	111
50 ppb	51.3	103
50 ppb	51.7	103
50 ppb	45.9	92
Mean	53.8	
sd	5.25	
% RSD	9.8	

Table 1 – 50ng/l spike in QuEChERS extract results for Clomazone with no internal standard

Figure 6 shows extracted ion chromatograms for selected pesticides at10 ng/l with an extraction window of 20ppm.



Clomazone



Pirimiphos-ethyl

Figure 6 – Extracted ion chromatograms for selected pesticides at 10 ng/l

Figure 7 shows how the GC/Q-TOF can be used to give extra selectivity. Below shows the same chromatogram extracting with a unit mass window and also with a relative mass extraction window of 20ppm. As you can see when dealing with dirty samples, there is a high chance of observing analytes with the same unit mass. By extracting with a narrow extraction window, you can limit the amount of interfering ions observed for Dichlorvos.

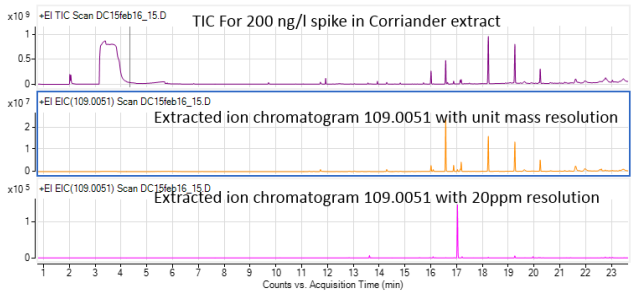


Figure 7 – 200 ng/l spike of pesticide test mixture in coriander extract

Discussion

This application note details how Automatic Liner Exchange (ALEX) can be used to deal with dirty samples by periodically changing the liner. No retention time drift was observed due to the liner change. The GC/Q-TOF gives extra selectivity over a GC Single Quadrupole instrument.

We would like to thanks FERA Science Ltd for supplying the spiked pesticide extracts for this work.