Pesticide Quantitation with LC/MS/MS and GC/MS for 419 Compounds

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1 Introduction

Detecting pesticides in the world around us and in the products we consume is becoming more and more important in today's world. It is very important that a simple and robust analytical solution exists. Pesticide use is so prevalent that the environmental and drinking water samples have to regularly collected and analyzed to ensure they are not contaminated with pesticides. PerkinElmer developed a robust analytical method with simple sample prep that enables a greater number of pesticides to be analyzed faster than ever before that utilizes LC/MS/MS and GC/MS.

Key Features

- Robust analytical methods and instrumentation.
- Ability to create large libraries of compounds of interest such as pesticides.
- Simple sample prep that saves sample prep time and can adapted to fit any number matrices.

PerkinElmer QSight 420
Triple Quadrupole
Mass Spectrometer

PerkinElmer Clarus 680
Gas Chromatograph and
SQ8 Mass Spectrometer

2 Experimental Conditions

GC Clarus 680 Conditions	
Injector Type:	PSSI
Carrier Gas:	Helium, 1 mL/min
Injector Temperature:	225°C, ramp to 250° during run.
Injection Volume:	1 µL
Injection Mode:	Splitless with Pressure Pulse
Glass Liner:	2mm Focus with Wool (N9306232)
Analytical Column:	PerkinElmer – Elite™-5ms 30 m x 0.25 mm x 0.25 µm
Oven Program:	Initial 100°C hold for 2 minutes, ramp to 300° C at 5°C/min and hold for 8 minutes, 50 minutes total.
MS SQ8 Conditions	
Transfer Line Temperature:	200°C
Source Temperature:	180°C
Multiplier:	1500 V
Solvent Delay:	5.75 min
Acquisition Mode:	SIR, 32 groups

Table 1.GC Instrument Parameters

LC Conditions	
Mobile Phase A ESI/APCI	LC/MS grade Water + 0.1 % formic acid + 2 mM ammonium formate/LC/MS grade Water
Mobile Phase B ESI/APCI	LC/MS grade Methanol + 0.1 % formic acid + 2 mM ammonium formate/LC/MS grade Methanol
Gradients used	For the ESI method, the 19 minute run had initial conditions of 5 % B at 0.8 mL/min for a 0.5 minute hold, with a ramp to 50 % B by 4 minutes, followed by a ramp to 100 % B by 17.5 minutes, with a 1.5 minute re-equilibration period at initial conditions. For the APCI method, the 12 minute run had initial conditions of 30 % B at 0.8 mL/min for a 0.5 minute hold, followed by a ramp to 95 % B by 8 minutes, with a hold for 2 minutes before a 2 minute re-equilibration at initial conditions.
Column Oven Temperature	40 °C
Sample Tray Temperature	5 °C
Injection Volume ESI/APCI	3 µL/10 µL
MS Conditions	
Positive ESI Voltage	+ 5100 V
Negative ESI Voltage	-4500 V
Negative APCI Current	-3 µA
Drying Gas	150 arbitrary units
Nebulizer Gas	350 arbitrary units
Source Temperature ESI/APCI	315 °C/250 °C
HSID Temperature ESI/APCI	200 °C/180 °C
Detection Mode	Time-managed MRM

Table 2. LC Instrument Parameters

3 Sample Preparation

1. Measure 1 g of sample.
2. Spike with internal standards and addition of 5 mL of acetonitrile and 0.1 % formic acid.
3. Agitate to aid extraction at 1500 rpm for 30 minutes.
4. Centrifuge and filter.
5. The extract is now ready for LC/MS/MS analysis via ESI and APCI methods + GC/MS analysis.

4 Results

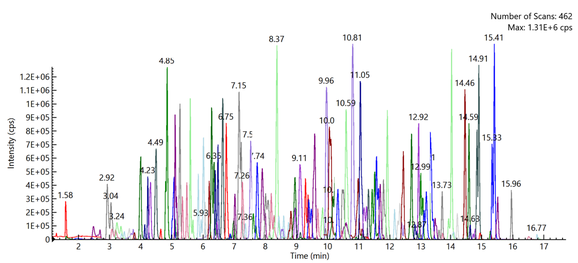

Figure 1. One ESI injection for 347 compounds on the Qsight 420

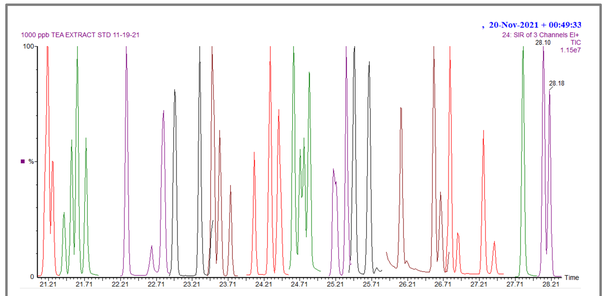

Figure 2. A sample GC chromatogram from the Clarus 680 SQ8

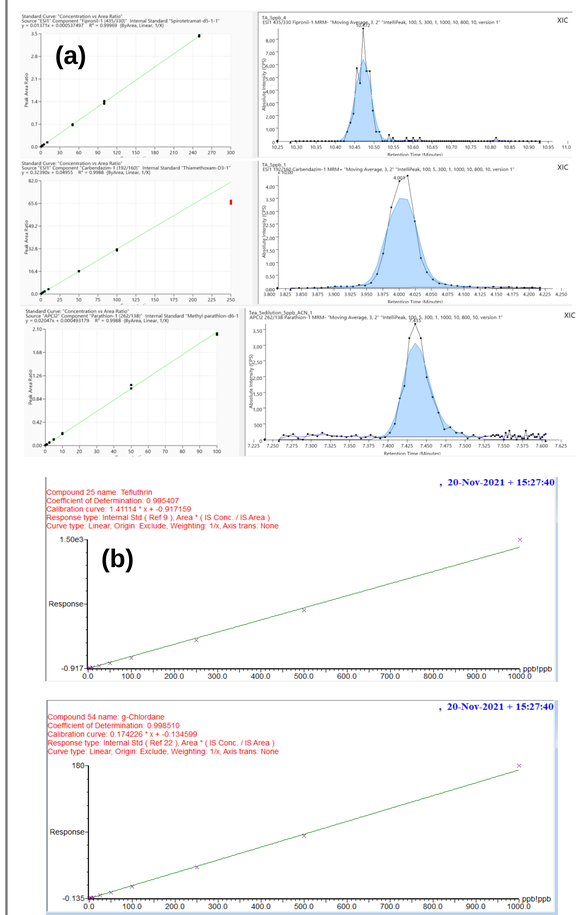

Figure 3. Example LC/MS/MS (a) and GC/MS (b) calibration curves showing excellent linearity

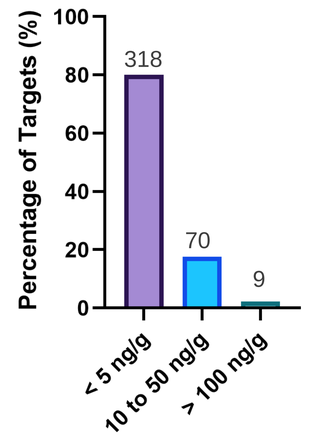

Figure 4. Limits of Quantification for pesticides analyzed by LC/MS/MS

5 Conclusions

The need to detect pesticides at lowest concentrations possible in several matrices is higher than ever before. PerkinElmer has created an analytical solution that allows for the 419 different pesticides to be analyzed simply and easily. Historically, most pesticides analyses were performed by Gas Chromatography but now we are seeing Liquid Chromatography being utilized more than ever before. Most of the pesticides shown here were analyzed with LC/MS/MS and compounds that showed poor performance on the LC/MS/MS showed strong performance when analyzed on the GC/MS.

The analytical solution presented here showcases how PerkinElmer's QSight 420 LC/MS/MS can be used with GC/MS to easily analyze more pesticides than ever before. The simple sample preparation and reliable instrumentation allows for a robust analytical solution with the potential to analyze hundreds of pesticides in many different matrices.