APPLICATION NOTE

Residual Pesticides in Grapes by GC-MS (SQ)

AN180v1; June 2025, SCION Instruments

Introduction

Pesticides are another term used for plant protection products which are used to control pests, prevent weeds and diseases. This is crucial for keeping crops healthy and improving crop yields. Pesticides play an important role in the food production process but have a toxicological effect. Death by self-poisoning due to excessive pesticide consumption is still prevalent in low and middle income countries.[1]

Testing for residual pesticides is important to maintain human health and food quality. In order to meet crop demand the use of pesticides is necessary but regulations have been put in place to minimize the effect on human health and the environment. The World Health Organization states it has two objectives in relation to pesticides. One is to eliminate the use of the most toxic pesticides to humans and ones which remain in the environment for the longest period of time. The other is to set limits for residual pesticides in food and water to protect human health.[1]

In this application note grape samples were analyzed for residual pesticides using a 8500 GC with 8700 MS single quad (SQ) and 8400 PRO autosampler.

Experimental

An internal standard (IS) was added to the samples to help improve the precision of results. The internal standard selected was triphenylmethane. A working standard was prepared with a concentration of 10 µg/ mL in toluene. For more information on the importance of using an internal standard, see our technical note.

A commercially available pesticide standard containing the compounds in Table 2 with a concentration of 10 µg/ mL were used to prepare linearity samples at 5 concentration levels: 50, 75, 100, 150, 200 and 250 ng/mL in toluene. Each linearity sample contained 100 ng/ mL of the internal standard.

To be able to analyze residual pesticides in grapes by GC-MS, sample preparation using the buffered AOAC method QuEChERS was conducted. 15 mL of 1% acetic acid in acetonitrile and 15 g homogenized grapes were added to a 50 mL QuEChERS extraction tube with AOAC buffered extraction salts in duplicate. To both extraction tubes 150 µL IS was added. To one extraction tube 150 µL pesticide standard (10 µg/ mL) was added. Samples were shaken for 1 min and then centrifuged >1500 rcf for 5 min. 1 mL acetonitrile extract from each sample was transferred to 2 mL PSA AOAC clean up tubes and shaken for 30 sec. 2 mL clean up tubes centrifuged >1500 rcf for 1 min. 0.5 mL extracted from clean up tubes and 0.25 mL toluene was added to each sample. The samples were blown down under nitrogen to <0.1 mL then brought up to 0.4 mL with toluene and transferred to vial with insert for analysis.

Instrument parameters can be found in Table 1. The instrument method shown in Table 1 gave excellent specificity with all compounds resolving from one another and exhibiting good peak shape.

In order to improve the sensitivity of the system, Selective Ion Monitoring (SIM) was used. For each compound a single qualifier ion was selected by choosing the most abundant ion for each pesticide. The 2nd and 3rd most abundant ions were used as qualifier ions which are important for analyte confirmation and preventing false positives. See Figure 1 for an example from MSWS of the quantifier and qualifier ions selected for terbuthylazine.

Table 1 Instrument parameters for GC-MS

Part	Settings
Injector	300 °C Pulsed splitless injection Splitless time 0.3 min Pulse 25 psi for 0.25 min
Injection Volume	1.0 µL
Column	SCION-5MS 30 m x 0.25 mm x 0.25 µm
Carrier Gas	Helium 1.5 mL/min
Oven Program	80°C (hold 2.0 min), 5°C /min to 200°C, 40 °C /min to 300°C (hold 1.5 min)
Run Time	30.0 min
Software	MSWS v8.2.1
Part	**Settings**
MS transfer line temp.	275 °C
Ion source temp.	300 °C
MS mode	Electron Ionization
Delay collection time	5.0 min
Scan mode	SIM mode

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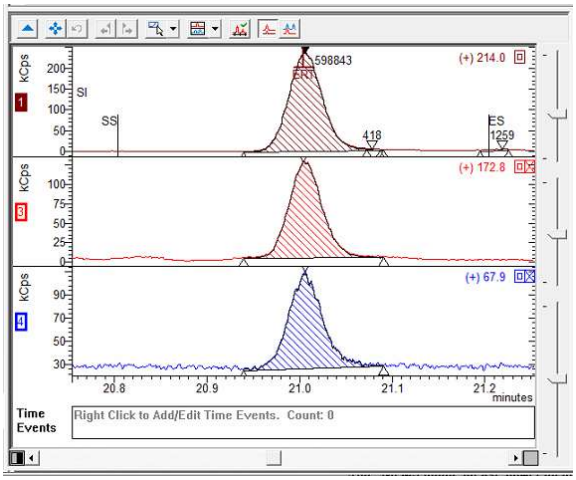

Figure 1 Example mass spectra of terbuthylazine from a linearity sample (L3) showing quantifier (top) and qualifier plots (middle and bottom)

Results

Table 2 shows the correlation coefficient (r^2) and the RSD for the system precision (n=6) conducted for the compounds present in the pesticide standard. This table also highlights the quantifier and qualifier ions selected for each compound.

Table 2 Correlation coefficient and system precision results for all pesticides

Pesticide	Correlation coefficient (r^2)	RSD (%)	SIM Ions (Quantifier and qualifiers)
Atrazine	0.9976	1.14	200, 58, 68
Cyanazine	0.9958	1.40	212, 68, 96
Desethylatrazine	0.9915	0.47	172, 68, 69
Hexazinone	0.9975	0.56	171, 128, 252
Isoproturon	0.9976	0.91	72, 146, 206
Linuron	0.9920	1.47	61, 124, 187
Metazachlor	0.9977	1.46	81, 133, 132
Methabenzthiazuron	0.9908	1.02	136, 164, 135
Metobromuron	0.9918	1.19	61, 90, 63
Metolachlor	0.9923	1.40	162, 238, 146
Monolinuron	0.9954	1.37	61, 126, 153
Sebuthylazine	0.9950	1.02	200, 202, 68
Simazine	0.9944	0.87	201, 68, 186
Terbuthylazine	0.9948	1.11	214, 173, 68

The r^2 values are all >0.99 which demonstrates excellent linearity. The system precision for all compounds is <2%, including many compounds ≤1%. The system is shown to have excellent repeatability for the results. An example plot for the linearity of terbuthylazine is shown in Figure 2.

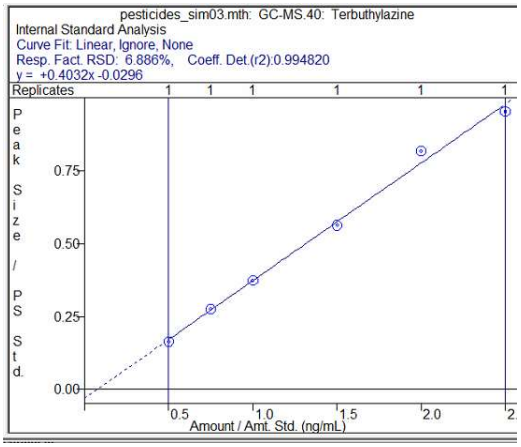

Figure 2 Example linearity (L3) for terbuthylazine, showing correlation coefficient (r^2) from data review in MSWS

Table 3 shows the recovery and precision results for the QC spiked samples (n=6). Recovery was 101.26–139.95 % for the compounds present in the pesticide standard with precision <4%. As stated in SANTE/11312/2021 (V2) a practical default range of 60-140 % may be used for individual recoveries in routine analysis and RSDs outside of ±20% should be investigated.[2] Therefore, the SCION GC-MS recovery % results are within the practical range with RSDs well below the set threshold.

Figures 3 and 4 show an example of the data review in MSWS for linuron in a QC spiked sample. Figure 3 displays the plot for the quantifier ion selected for linuron and Figure 4 demonstrates the average QC recovery for a spiked sample as 134.81%.

The QC blank was shown to have no residual pesticides present. An example of this can be seen in Figure 5 which shows the spectra of the QC blank (green) and QC spiked (red) overlaid for linuron. See our tech note on Recovery Spiked Sample. Spiking the sample allows the performance of the analytical method to be evaluated to ensure that the method produces accurate and valid results. By spiking your sample you increase the concentration of the target analytes by a known amount and therefore will be able to determine if the added analytes are recovered. It is key to spike your sample at a concentration within your linearity range and sample volume is not increased. This allows calculations to be consistent and avoids introducing unknown effects.

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Table 3 Recovery and precision results from spiked sample

Pesticide	Recovery (%)	RSD (%)
Atrazine	127.29	0.74
Cyanazine	136.33	0.96
Desethylatrazine	139.35	1.15
Hexazinone	138.35	1.24
Isoproturon	101.26	3.61
Linuron	133.26	3.22
Metazachlor	138.22	2.76
Methabenzthiazuron	139.67	0.31
Metobromuron	139.61	1.14
Metolachlor	139.95	0.32
Monolinuron	136.82	0.62
Sebuthylazine	133.48	0.68
Simazine	125.61	0.80
Terbuthylazine	128.27	0.21

Conclusion

A method was developed and validated for the analysis of residual pesticides in grapes by GC-MS (SQ). Samples were prepared using the buffered AOAC method for QuEChERS. For further information on using QuEChERS, see our technical note in the SCION Instruments' knowledge centre.

To validate the results, spiked samples were also analyzed. The spiked samples proved that sample preparation techniques were not interfering with the target analytes. The linearity showed that the instrument and method were in good working order and system precision results indicate excellent repeatability of the system. Recovery results found fell within the guidance outlined in SANTE/11312/2021 (V2).

The grape samples showed no residual pesticides were present which was expected. It is recommended to use quality control samples throughout your analytical run to monitor that your system remains consistent over many runs.

SCION instruments recommends checking with local regulatory authorities to ensure all testing and reporting requirements are met, or contact the SCION applications team for assistance.

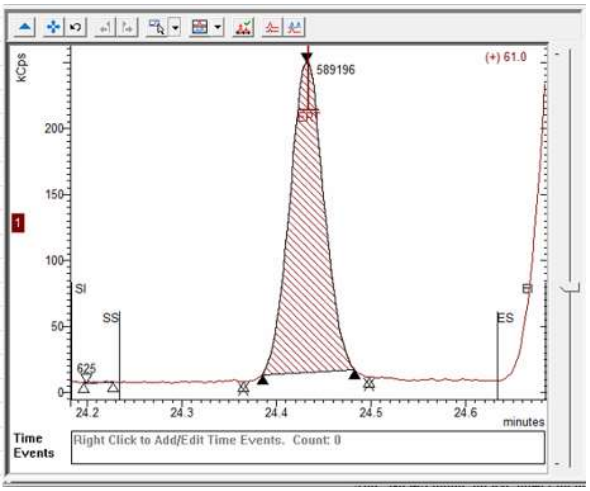

Figure 3 Quantifier ion plot for Linuron

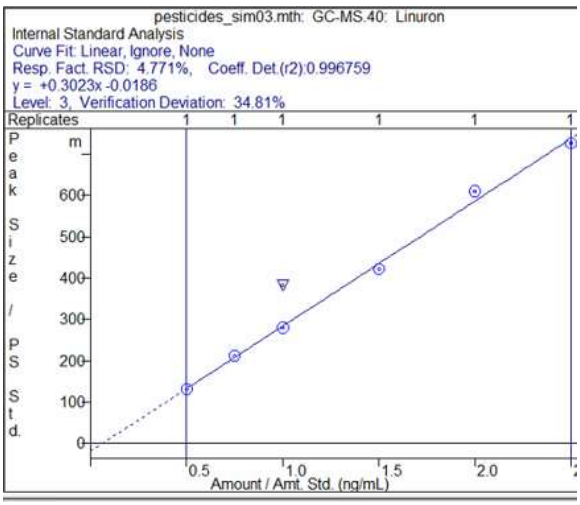

Figure 4 Example QC spiked sample showing recovery of 134.81%

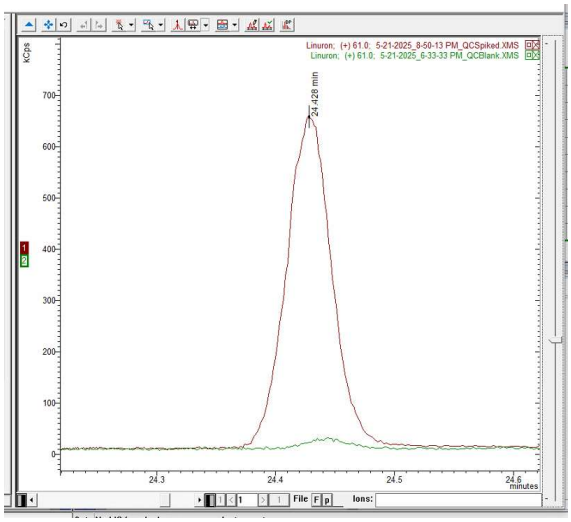

Figure 5 Overlaid spectra of QC spiked sample (red) and QC blank sample (green) for linuron

Ordering Information

Ordering Information for the 8300 GC	
Part	**Part Number**
SCION SQ SELECT, w/8300, SSL-T21; 120V	SCIONSQ83SEL311
SCION SQ SELECT, w/8300, SSL-T21; 230V	SCIONSQ83SEL312
8400 PRO Autosampler for 8300 and 8500 GC	840000001
Suggested Consumables	
Part	**Part Number**
SCION-5MS 30m x 0.25mm x 0.25µm	SC32223
Liner TAPER QW ULTRA PK/5	41312117
15% Graphite/85% Vespel Ferrule 1/16" with 0.4 mm hole pk/10	41312148
BTO Septa 9 mm, pk/50	CR298713
10 µL fixed needle syringe, 5 cm, 0.47 mm OD, 26 g conical needle	41312133
Vial, 9-425 Screw Thread, 2 mL Clear Glass 12 x 32 mm Flat Base with Label, pk/100	41311000
Cap, Screw, Blue 9-425 Open Top Ribbed with 9mm Red PTFE/Sil Septa 1mm Thick. 100pcs/pk	41311002

For ordering info on the SCION 8500 GC, which offers greater functionality with the option of up to 4 detectors (including MS), please contact your local SCION sales representative.

References

1. World Health Organization, https://www.who.int/news-room/fact-sheets/detail/pesticide-residues-in-food, (accessed 06 May 2025)

2. European Commission, https://eurl-pesticides.eu/docs/public/tmplt_article.asp?CntID=727, (accessed 02 Jul 25)

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