Tackling the Glyphosate Paradox with IC-MS/MS: Towards Enhanced Monitoring and Analysis of Polar Anionic Pesticides

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Polar anionic pesticides are widely used in agriculture. Residues can find their way into food commodities such as cereals, honey and processed foods. Despite controversy over these substances' potential toxicity and impact on human health, their chemical properties have traditionally made them particularly difficult and expensive to analyse in food products.

More sensitive, reliable, and cost-effective methods of analysis are needed to guarantee food safety and quality control, meet regulations, and protect the environment. This article highlights the potential of an integrated modular analytical workflow based on a modified version of the Quick Polar Pesticides Extraction (QuPPe) method for extraction, and determination using a compact ion chromatograph coupled with triple quadrupole (QQQ) mass spectrometer (IC-MS/MS). The workflow demonstrates the ability to directly and simultaneously analyse multi-residue polar anionic pesticides and their metabolites in the food supply.

Introduction

The polar anionic pesticide glyphosate is the most widely used herbicide in the world, but also one of the least determined - a dichotomy defined as the 'glyphosate paradox' [1]. Glyphosate is used in over 750 different products across agriculture, forestry, urban and home applications, and has been detected in the air (during spraying activity), water and food [2]. This is concerning given that the safety of glyphosate is unclear and hotly disputed. The International Agency for Research on Cancer (IARC) classified glyphosate as a probable carcinogen in March of 2015 [3]. However, the European Food Safety Authority (EFSA) came to a different conclusion, instead considering glyphosate unlikely to pose a carcinogenic hazard to humans [4].

Given this uncertainty, the EFSA and European Commission have called for more effective methods and increased monitoring of polar anionic pesticides. However, it has traditionally proved analytically challenging and costly to analyse polar pesticides, especially glyphosate. Polar pesticides have a range of properties that significantly increase the need for pretreatment and derivatisation in analytical techniques. They also lack chromophores or fluorophores, which are necessary for many methods of spectrophotometric detection, and are similar to many amino acids, natural plant components, and their own by-products, which can interfere with quantitative analysis [1].

Furthermore, due to their physicochemical properties, the complete analysis of these pesticides has historically required several single residue methods. Single residue analyses involve a great deal of labour and cost while only covering a single or limited number of pesticides, making them inefficient, resource-intensive methods. Such methods also do not always account for all metabolites listed in EU maximum residue level (MRL) definitions, or those with the potential to be included in future. Currently, MRLs are defined for glyphosate as a parent compound only and do not include any of the metabolites (although this definition may change in the future based on ongoing guidance from the EFSA). However, genetically modified (GM) crops have a deactivation pathway that converts glyphosate to the N-acetyl glyphosate and N-acetyl AMPA metabolites, and omitting these from risk assessments could, potentially, lead to an underestimate of the frequency and distribution of residues.

There is a pressing need for a sensitive, cost-effective and reliable way to characterise and quantify as many polar pesticides as possible, at low concentrations, in a diverse range of sample types - all in a single generic method. Currently, the Quick Polar Pesticides Extraction (QuPPe) method [5] of generic extraction with chromatographic separation based on either hydrophilic interaction liquid chromatography (HILIC) or ion chromatography (IC) is used, with detection, quantitation and identification performed by mass spectrometry (MS).

QuPPe is based on extraction with methanol/water without liquid/liquid partition or solid-phase extraction clean-up. As a result, extracts can contain high levels of co-extractives that contaminate chromatographic and detection systems, and suppress the MS response. In terms of chromatographic separation, reversed-phase liquid chromatography with tandem MS (LC-MS/MS) suffers from poor retention of polar anionic pesticides. Pre-or post-column derivatisation techniques can increase retention and selectivity, but also

Table 1: Summary of experimental conditions and settings.

Conditions for ion chromatography	
IC System:	Dionex Integrion HPIC system
Conductivity monitor	Conductivity detector
Columns:	Thermo Scientific™ Dionex™ IonPac™AG19-4µm Guard, 2 × 50 mm (P/N 083225) Thermo Scientific™ Dionex™IonPac™AS19-4µm Analytical, 2 × 250 mm (P/N 083223)
Eluent Source:	Dionex EGC 500 KOH Eluent Generator Cartridge with Dionex CR-ATC 600
KOH Gradient:	20–30 mM (0–2 min), 30 mM (2–8 min), 45-55 mM (8–12 min), 80 mM (12–14 min), 85 mM (14–19 min), 20 mM (19-21 min)
Flow Rate:	0.35 mL/min
Injection Volume:	25 µL
Temperature:	40 ºC (column oven), 20 ºC (compartment temperature) 35 ºC (conductivity detector cell)
System Backpressure:	~3900 psi (100 psi = 0.6894 MPa)
Suppressor:	Suppressed Conductivity, Dionex ADRS 600 Suppressor (2 mm) operated in constant current mode, AutoSuppression, 74 mA, external water mode via Dionex AXP Pump, external water flow rate (0.40 mL/min)
Background Conductance:	~0.3 µS/cm
Run Time:	21 min
IC-MS Interface:	Tee union (PEEK, P/N 00101-18204) to combine the analyte from conductivity detector via Thermo Scientific Viper fitting tubing
Post Suppressor Makeup Solution:	Acetonitrile at 0.2 mL/min via Dionex AXP-MS pump

Conditions or mass spectrometric detection	
Ion source settings	
Ion Source Type:	HESI
Spray Voltage:	Static
Negative Ion:	3250 V
Positive Ion:	3500 V
Sheath Gas (N_2):	60 Arbitrary units (Arb)
Aux Gas (N_2):	13 Arb
Sweep Gas (N_2):	1 Arb
Ion Transfer Tube Temp:	350°C
Vaporizer Temp:	250 °C
MS global settings	
Start Time:	0 min
End Time:	21 min
Master scan	
Scan Mode:	SRM
Use Cycle Time:	True
Cycle Time:	1.0 s
Q1 Resolution (FWHM):	0.7
Q3 Resolution (FWHM):	1.2
CID Gas:	2.0 mTorr
Source Fragmentation:	0 V
Chromatographic peak width:	6 s
Transition conditions:	Optimized for each compound using TSQ Altis mass spectrometer

limit the analyte scope of the method (number of compounds determined), increase the labour involved, and result in high methodological variability if the derivatisation in not precisely controlled. For example, glyphosate and the metabolite AMPA are often derivatised before quantitation with reversed-phase chromatography-MS, but the method is specific to glyphosate only, and therefore relatively costly on a per-analyte basis.

HILIC [6] and non-suppressed IC [7,8] have been used to analyse polar pesticides, but both options provide lower column capacity compared to IC with electrolytic suppression. Using the HILIC option, Herrera López et al. reported the analysis of 14 anionic pesticides in a number of different matrices [6]. The researchers evaluated several clean-up options, but with limited success, so opted to dilute the QuPPe extracts before LC-MS/MS only. Although dilution reduces the concentration of matrix co-extractives it also reduces the concentration of analytes, so the 0.01 mg/kg target reporting level could not be reached for all the analytes, especially in the more difficult matrices: cereal and soybean. With non-suppressed IC using weak bicarbonate eluent the gradient elution options are restricted, which limits the number of analytes that can be included in a single analysis.

Alternatively, ion chromatography with either triple quadrupole mass spectrometry (IC-MS/MS) [9] or high resolution accurate mass (HRAM) mass spectrometry [10] has emerged as a powerful and useful tool with which to analyse and monitor polar anionic pesticides. This approach aggregates several compound-specific methods into a single technique, while producing results compliant with residue definitions and regulations worldwide. It can simultaneously analyse several analytes at low concentrations (including glyphosate and glufosinate, and their metabolites, ethephon, fosetyl, chlorate, perchlorate and others). The use of IC, especially with post-column electrolytic ion suppression and tandem triple quadrupole (QQQ) MS, combines the benefits of both techniques to overcome common difficulties: the high sample capacity of IC columns brings excellent chromatographic retention and resolution in a wide range of matrices, while tandem MS enables high selectivity and sensitivity and, therefore, low µg/kg detection limits.

Presented are the results of an integrated sample-to-result analytical workflow based on IC coupled with QQQ MS. Crucially, this workflow also includes a cartridge solid phase extraction step, which reduces the amount of matrix co-extractives and hence contamination of the analytical system. The workflow was developed and validated for multi-residue analysis of polar anionic pesticides in representative food matrices [11]. It demonstrated the potential of the technique for robust, efficient, compliant, accurate and precise quantitation.

Experimental

Materials

Samples were chosen to represent two groups in the EU SANTE guidelines [12]: wheat flour (representative of dry commodities, group 5), and leek (representative of green vegetables, group 1). Samples were purchased from retail outlets in Beijing, China.

The chemicals and consumables used included deionised (DI) water, methanol and acetonitrile, and Thermo Scientific Dionex OnGuard II RP cartridges. Isotopically labelled standards were obtained from various sources: glyphosate-$^{13}C_2$,15N, 3-methylphosphinicopropionic acid-d_3 sodium salt and glufosinate-d_3 hydrochloride

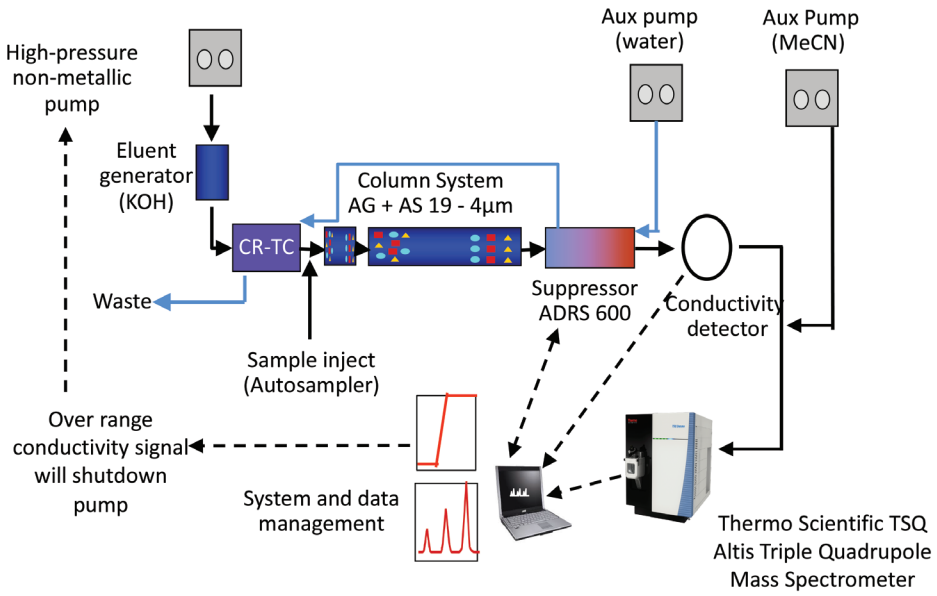

Figure 1: Configuration of the fully integrated IC-MS/MS system.

from Trc-Canada; aminomethylphosphonic acid-^{13}C, potassium chlorate $^{18}O_3$, ^{15}N, ^{2}D and perchloric acid sodium salt ($^{18}O_4$) from Cambridge Isotope Laboratories Inc.; and ethephon-d_4 from A ChemTek, Inc. The performance of the instrument modules was tested using the QPP-Lab Standard Kit QuPPe EURL v.10-1.3 Method Compliance stock solution, obtained from Lab Instruments, Italy.

Conditions, calibration and data

Analysis followed the Thermo Scientific Anionic Pesticides Explorer workflow, comprising a Thermo Scientific Dionex Integrion High-Pressure Ion Chromatography (HPIC) system fitted with a Thermo Scientific Dionex electrolytic eluent generator cartridge (EGC) and conductivity cell, coupled to a Thermo Scientific Dionex AS-AP Autosampler and Thermo Scientific TSQ Altis Triple Quadrupole Mass Spectrometer (table 1). The separation method used a Thermo Scientific Dionex IonPac AG19-4 µm Guard column (2×50 mm) coupled to a Thermo Scientific Dionex IonPac AS19-4 µm Analytical column (2×250 mm) held at 40°C. Elution of polar anionic analytes used a potassium hydroxide gradient at a flow rate of 0.35 mL/min. A 2 mm Thermo Scientific Dionex ADRS 600 Anion Dynamically Regenerated Suppressor, fed with DI water at 0.7 mL/min by an auxiliary Thermo Scientific Dionex AXP pump and connected to the outlet of the column, converted KOH eluent to water before it flowed through the conductivity detector and mass spectrometer (connected in series). Acetonitrile was delivered at a make-up flow rate of 0.2 mL/min by an auxiliary Dionex AXP-MS pump.

The IC injection volume was 25 µL; the compartment temperature was held at 20°C and conductivity detector cell at 35°C; the system back pressure was ~3900 psi; and the run time was 21 minutes.

The mass spectrometer was operated in selected reaction monitoring (SRM) mode to ensure high selectivity and low detection limits. Because the target analytes are small molecules with low mass-to-charge (m/z) product ions, the MS was calibrated using the Thermo Scientific Pierce Triple Quadrupole Calibration Solution, Extended Mass Range. This improved mass accuracy and transmission compared to conventional polytyrosine mass calibration solution, especially in the low m/z range.

The system control, data acquisition and data processing was done using Thermo Scientific Chromeleon Chromatography Data System software (version 7.2.9), Thermo Scientific Xcalibur software (version 4.1) and Thermo Scientific TraceFinder software (version 4.1).

Standards

Matrix-matched standards (MMS) were prepared by spiking the diluted, cleaned-up extract with native standards and isotope-labelled internal standards (ILIS) where available. Procedural standards (PS), offer an alternative method of calibration in certain situations where ILIS are either unavailable or unsuitable. Procedural standards are prepared by spiking blank sub-samples, before extraction, with native pesticide standards over the calibration range of interest. The procedural standards are then extracted alongside samples and the responses used to construct calibration curves for quantitation. Since these standards are subject to the exact same extraction conditions as samples, and hence any losses of analytes, the residue concentrations in the samples are effectively corrected for recovery improving accuracy of the results. However, analysts need to be aware that, in cases where there is significant variation between the matrix composition of different individual samples of the same commodity (e.g. wheat from different sources), the use of ILIS or the Standard addition approach will provide more accurate results than procedural standards.

Method

Within the fully integrated IC-MS/MS system (figure 1), deionised water was pumped into the EGC cartridge to facilitate the automatic generation of the eluent. By removing manual preparation protocols, this automated approach not only saved time, but also reduced the risk of human error, which in turn enabled high levels of reproducibility. The eluent then exited the cartridge, underwent purification, and was passed through EG degas tubing to remove any hydrogen gas produced during KOH generation.

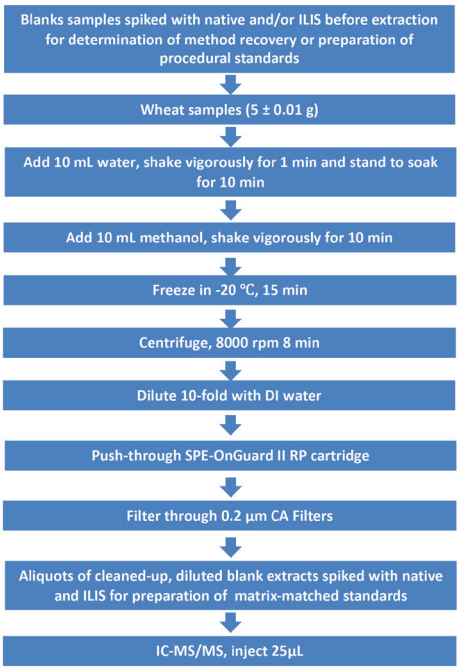

Figure 2: Flow diagram of the Modified QuPPe Extraction Method.

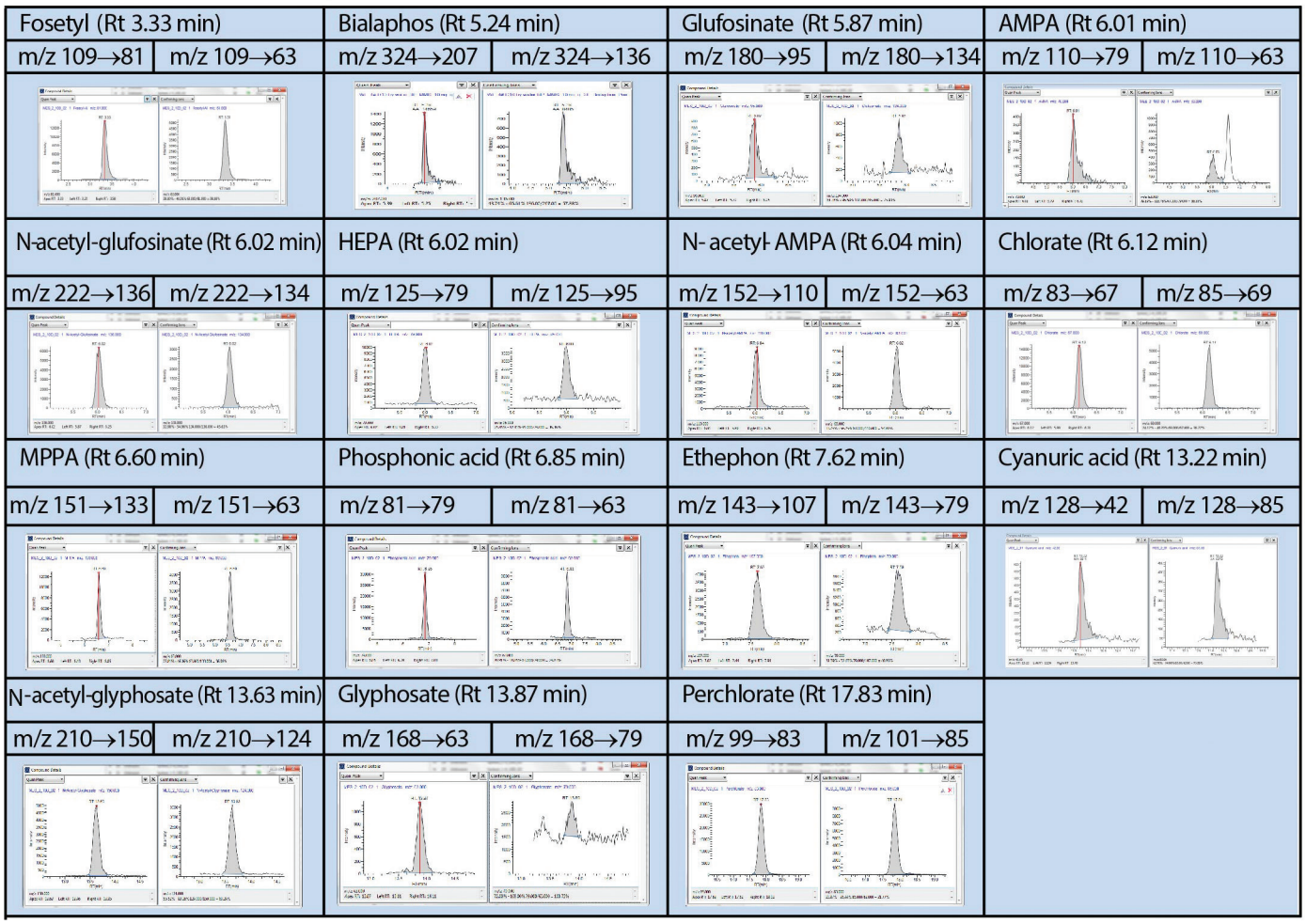

Figure 3. Response for quantification and qualifier product ions for individual anionic pesticides equivalent to 10 ng/g in wheat flour.

After then entering the injection valve, the sample was pumped, along with the eluent, through the Guard and Analytical columns and suppressor. In the suppressor, cations from both the eluent and sample were replaced with hydronium ions; this neutralised the high-pH eluent and optimised its compatibility with the mass spectrometer. The eluent entered the conductivity detector (to measure background conductivity levels of typically below 1.5 µS/cm pre-injection), before acetonitrile was added (at a rate of 0.2 mL/min) to increase the signal intensity of the analyte before it entered the electrospray interface. The addition of acetonitrile between the suppressor and mass spectrometer improved electrospray aerosol desolvation and increased the response of most analytes by three- to four-fold.

The extraction of the samples was based on a modified QuPPe method (Figure 2), to offer a straightforward, streamlined workflow that applies to a wide range of complex matrices.

Each wheat flour sample was mixed thoroughly before being portioned, and leek samples were thoroughly homogenised using a blender. Sub-samples of homogenised leek (10 ± 0.01 g) and wheat flour (5 ± 0.01 g) were weighed into 50 mL polypropylene centrifuge tubes. DI water (1.5 mL for leek, 10 mL for wheat flour) was added to adjust the water content of each tube to 10 mL, followed by the addition of methanol (10 mL). The hydrated samples were then mixed vigorously for 10 minutes using a vortex mixer. The extract was subsequently placed in a freezer for 15 minutes before being centrifuged at 8000 rpm for 8 minutes at 5°C.

The resulting supernatant was then diluted with DI water 10-fold and pushed through a preconditioned Thermo Scientific Dionex OnGuard II RP cartridge coupled to a 0.2 µm Thermo Scientific Titan3 CA Membrane Syringe Filter (connected in series). The first 3 mL of filtrate were discarded, and the subsequent 1.5 mL collected in plastic vials. Plastic was used throughout to minimise and avoid adsorption of analytes onto glass.

Simultaneous Analysis of 15 Analytes Without Derivatisation

The QQQ IC-MS/MS workflow offered direct analysis of 15 polar anionic pesticides simultaneously (Figure 3), and without the need for derivatisation: AMPA, chlorate, cyanuric acid, ethephon, glufosinate, glyphosate, MPPA, perchlorate, bialaphos, fosetyl-Al, HEPA, phosphoric acid, N-acetyl AMPA, N-acetyl glufosinate and N-acetyl glyphosate.

High selectivity and sensitivity were achieved, with the QQQ IC-MS/MS system returning satisfactory separation for all 15 pesticides, including metabolites of interest, in 18 minutes. These were identified based on the presence of transition ions (quantifier and qualifier) at retention times corresponding to those of each pesticide, and with ion ratios within 30% (relative) of average calibration standards in the same sequence. Peak shapes and sensitivities were satisfactory for most of the anionic pesticides present in the wheat extract at 0.25 ng/mL (equivalent to 10 ng/g in the sample), with the response for the quantifier

Table 2. Summary of results for recovery and precision using different calibration approaches for wheat flour.

	Spiked 10 ng/g								Spiked 50 ng/g							
	MMS no ILIS		MMS +ILIS		PS no ILIS		PS +ILIS		MMS no ILIS		MMS +ILIS		PS no ILIS		PS +ILIS	
	Rec. %	RSD %	Rec. %	RSD %	Rec. %	RSD %	Rec. %	RSD %	Rec. %	RSD %	Rec. %	RSD %	Rec. %	RSD %	Rec. %	RSD %
Fosetyl-Al	93	2.7	-	-	96	2.7	-	-	89	1.9	-	-	94.6	1.08	-	-
Bialphos	96	6.4	-	-	95	5.7	-	-	90	3.2	-	-	76	9.5	-	-
Glufosinate	85	12	92	8.6	87	12	92	8.6	76	4.5	94	3.0	95	6.0	111	4.8
AMPA	65	6.6	115	6.1	104	6.5	115	6.1	61	4.9	108	9.0	98	1.5	98	6.1
HEPA	86	2.4	-	-	96	2.6	-	-	80	0.7	-	-	79	2.5	-	-
N-acetyl AMPA	85	1.0	-	-	98	1.1	-	-	81	0.6	-	-	89	1.0	-	-
N-acetyl Glufosinate	79	2.4	-	-	87	2.8	-	-	72	2.9	-	-	83	2.4	-	-
Chlorate	77	2.2	96	1.7	100	2.3	96	1.7	73	2.0	92	0.8	89	2.3	96	1.5
MPPA	71	1.0	96	1.4	95	1.1	96	1.4	63	2.5	94	1.9	101	2.3	101	2.0
Phosphonic acid	36	25*	-	-	84	14	-	-	69	3.4	-	-	85	3.5	-	-
Ethephon	79	1.4	97	2.1	100	1.4	97	2.1	74	0.9	92	0.3	99	0.4	99	2.0
Cyanauric Acide	87	12	-	-	95	12	-	-	89	1.8	-	-	87	5.0	-	-
N-acetyl - glyphosate	60	2.9	-	-	100	3.0	-	-	53	1.7	-	-	98	1.2	-	-
Glyphosate	40	4.5	111	2.2	104	5.4	111	2.2	34	2.0	100	1.5	100	2.2	101	2.0
Perchlorate	66	4.2	100	0.9	90	5.2	100	0.9	63	3.0	96	0.7	101	0.6	100	0.3

Note: PS= procedural standards

* Poor precision to phosphonic acid contribution from blank

and qualifier ions and ion ratios meeting the requirements for compliance with substantive and default (set at 0.01 mg/kg) MRLs in wheat. This demonstrates the method's excellent sensitivity across a range of complex sample-pesticide combinations.

The use of matrix-matched standards in combination with ILIS produced excellent results in both matrices, and the use of ILIS substantially improved recovery when compared to matrix-matched calibration without ILIS (tables 2 and 3). Recovery improved from 40% to 111% for glyphosate, for instance, and 60% to 100% for perchlorate. As appropriate and affordable labelled standards are not consistently available in some parts of the world, the use of procedural standards was also evaluated (table 4). Excellent results were obtained for all analytes in wheat, with recoveries of 84% to 104% and associated RSDs of 0.9% to 7%, using the same sample matrix for calibration and spiking (table 2).

However, results were more variable between wheat samples from different sources. The use of procedural standards without ILIS may potentially be sufficient for screening wheat samples. Still, quantitation with ILIS or standard addition, as performed in this study, is necessary for improved accuracy.

By contrast, the results for leek were more consistent due to the lower matrix effects compared to wheat flour (table 3). Results

Table 3. Summary of results for recovery and precision using different calibration approaches for leek.

	Spiked 10 ng/g								Spiked 50 ng/g							
	MMS no ILIS		MMS +ILIS		PS no ILIS		PS +ILIS		MMS no ILIS		MMS +ILIS		PS no ILIS		PS +ILIS	
	Rec. %	RSD %	Rec. %	RSD %	Rec. %	RSD %	Rec. %	RSD %	Rec. %	RSD %	Rec. %	RSD %	Rec. %	RSD %	Rec. %	RSD %
Fosetyl-Al	97	0.8	-	-	93	0.8	-	-	96	1.1	-	-	95	1.1	-	-
Bialphos	122	8.3	-	-	82	9.0	-	-	98	8.2	-	-	76	9.5	-	-
Glufosinate	90	2.9	94	7.1	96	7.5	119	8.0	92	2.9	85	5.1	95	6.0	111	4.8
AMPA	100	7.6	111	8.4	96	8.4	94	9.6	95	2.5	104	6.1	98	1.5	98	6.1
HEPA	99	7.5	-	-	87	6.1	-	-	100	2.4	-	-	79	2.5	-	-
N-acetyl AMPA	91	1.0	-	-	100	0.7	-	-	101	1.0	-	-	89	1.0	-	-
N-acetyl Glufosinate	102	1.9	-	-	88	1.6	-	-	105	2.3	-	-	83	2.4	-	-
Chlorate	93	2.6	86	2.1	86	2.6	105	1.8	97	2.2	90	1.5	89	2.3	96	1.5
MPPA	94	1.9	89	1.7	96	1.9	92	1.8	100	2.3	95	2.0	101	2.3	101	2.0
Phosphonic acid*	64	12.3	-	-	85	8.1	-	-	87	3.6	-	-	85	3.5	-	-
Ethephon	97	2.5	104	2.7	97	2.7	102	2.8	96	0.4	98	1.9	99	0.4	99	2.0
Cyanauric Acide	118	4.6	-	-	104	4.4	-	-	100	4.9	-	-	87	5.0	-	-
N-acetyl - glyphosate	93	0.9	-	-	103	0.8	-	-	96	1.2	-	-	98	1.2	-	-
Glyphosate	91	1.6	90	1.5	95	1.6	94	1.6	95	2.2	94	2.0	100	2.2	101	2.0
Perchlorate	93	0.6	89	0.4	95	0.6	98	0.4	96	0.6	90	0.3	101	0.6	100	0.3

* Recovery and precision are less accurate for phosphonic acid because of an incurred residue in the blank

Table 4: Wheat flour data summary- procedural standards.

Sample No.7 used as calibration curve matrix	PS Curve	Spiked level (10 ng/g)							
		Sample No. 7 (n=5)		No. 4 (n=3)		No. 6 (n=3)		No. 9 (n=3)	
		Rec (%)	RSD (%)	Rec (%)	RSD (%)	Rec (%)	RSD (%)	Rec (%)	RSD (%)
AMPA	ISTD	108	6.5	114	7.6	86	6.5	111	4.1
Chlorate	ISTD	98	1.7	84	2.0	87	2.4	77	0.8
Ethephon	ISTD	97	2.2	103	3.7	100	7.6	103	1.5
Glufosinate	ISTD	98	8.7	88	8.7	95	5.5	100	7.3
Glyphosate	ISTD	101	2.4	90	4.1	93	9.9	99	7.3
MPPA	ISTD	96	1.4	102	3.1	116	2.2	97	1.7
Perchlorate	ISTD	86	1.0	95	1.1	88	4.0	77	2.0
Bialaphos	No ILIS	95	5.7	67	0.3	58	4.5	68	2.0
Fosetyl-Al	No ILIS	96	2.7	85	1.8	75	2.0	49	1.3
HEPA	No ILIS	95	2.6	85	2.6	80	6.5	87	4.6
N-acetyl AMPA	No ILIS	97	1.1	95	4.0	79	1.0	91	0.9
N-Acetyl-Glufosinate	No ILIS	87.	2.8	94	0.9	68	2.4	92	1.6
N-Acetyl-Glyphosate	No ILIS	100	3.0	68	3.7	59	2.7	87	2.3
Phosphonic acid	No ILIS	84	14	87	1.7	79	1.9	93	2.2

Over-spiking/or standard addition is the only option without availability of IUS

with MMS or PS, with or without ILIS, were all satisfactory. The high precision of the QQQ IC-MS/MS results is in part due to the inert peek flow path in the IC system, which negated column contamination and metal ion chelation (chelation is a common issue in some LC systems as metal ions leach from equipment, and can negatively impact results).

Crucially, the robustness of the workflow was significantly increased compared to other methods of analysis due to the inclusion of the OnGuard cartridge for sample clean-up. After 500 injections of matrix extracts, retention times and peak shapes remained stable for two test analytes (fosetyl-Al and perchlorate), demonstrating the reliable and robust nature of the QQQ IC-MS/MS approach. Additionally, the column and mass spectrometer source remained clean with no required maintenance, and the pressure in the suppressor was consistent. Given this increased efficiency and reduced labour, the method could offer routine laboratories highly sensitive ion analysis, accurate quantitation, and a robust workflow that is flexible and easy to implement.

Analysis of polar molecules, via methods such as LC–MS/MS, typically includes several different methods that employ various columns. By contrast, IC–MS/MS offers direct analysis of many polar pesticides simultaneously, bringing excellent chromatographic retention and resolution in a wide range of matrices with high selectivity and low detection limits.

Conclusion

Precise and simultaneous multi-residue analysis of 15 polar ionic pesticides in complex food samples was achieved using integrated QQQ IC-MS/MS. All chromatographic and mass spectrometer parameters were optimised so the workflow provided excellent sensitivity, recovery, selectivity and precision, met EU Maximum Residue Levels, and was compliant with EU SANTE guidelines. Extensive testing over several months and more than 1,500 sample injections proved the workflow to be reliable, reproducible, robust and suitable for routine analysis. Inclusion of the OnGuard cartridge for sample clean-up minimised levels of contaminating co-extractives, leading to a highly effective and time-saving approach to sample preparation and extraction. Workflows based on QQQ IC-MS/MS could, therefore, improve analysis that is key to guaranteeing food safety, meeting changing regulations, and ensuring quality control for food and pesticides: critical to safeguarding human and environmental health.

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This article is based on research conducted by Yingchen Li[1], Qilei Guo[1], Fausto Pigozzo2, Richard J. Fussell[3], and Beibei Huang[4].

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[3]Thermo Fisher Scientific, Hemel Hempstead, UK.

[4]Thermo Fisher Scientific, Sunnyvale, CA, USA.

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