INSIDE THIS SECTION

SiliaQuick™
QuEChERS

SILICYCLE Inc.
UltraPure SILICA GELS

SiliaQuick™ QuEChERS

Food Environment

Using SiliaQuick QuEChERS ensures the following benefits:

- Clean extracts from pure products.
- High recovery and lot-to-lot reproducibility.
- Great variety of QuEChERS to cover full spectrum of food applications.
- Reduction of analysis cost.

SiliaQuick QuEChERS for Pesticide Residue Analysis

The QuEChERS technique was developed in 2003 by USDA scientists to simplify and accelerate the analysis of pesticides in various fruit and vegetable samples. The name QuEChERS is formed by an acronym of the properties that are observed with this technique: ***Qu***ick, ***E***asy, ***Ch***eap, ***E***ffective, ***R***ugged and ***S***afe.

The QuEChERS method has gained in popularity to become the most valuable alternative for determination of traces of analytes in a high throughput environment. Presently, scientists have expanded the use of this method to the analysis of a vast array of pesticides, herbicides, fungicides, antibiotics, drugs, and any other compounds present in all food, beverage, animal and human matrices.

The QuEChERS technique can be summarized as a three-step methodology, starting with a **Liquid Extraction**, followed by a **dispersive Solid-Phase Extraction** clean-up, and completed by a **LC or GC Analysis**:

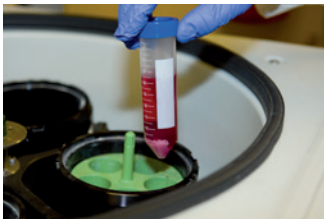
1) First step is to carry out the extraction of compounds of interest from your food or beverage matrix through a solvent (mainly *acetonitrile*).

2) The dispersive Solid-Phase Extraction clean-up is designed to remove specific undesired compounds such as sugars, lipids, organic acids, proteins, pigments and excess water from the solution.

3) Final analysis step consists in a simple injection into a LC or GC coupled with MS or MS/MS instrument to quantify the analyte concentration.

Step 1
Liquid Extraction

Extract pesticides or analytes of interest into an organic layer relying on the perfect combination of salts and acetonitrile.



Step 2
dispersive SPE Clean-Up

Subject organic layer from 1st step to further clean-up and selectively remove unwanted interferences such as lipids and pigments.

Step 3
LC or GC Analysis

Analyze your clean from last step and ready for GC or HPLC with MS/MS, or your selective detector.

SiliaQuick QuEChERS for Food Sample Treatments

SiliaQuick QuEChERS are designed to ensure ultimate performance in pesticide analysis.

***Quick*:** Pre-packed liquid extraction kits and dispersive solid-phase extraction clean-up kits contain the right amount of salts and/or sorbents to suit the specific food matrices, hence eliminating the sample preparation measurement step.

***Easy*:** Preweighed & ready-to-use tubes or packets for only 3 straightforward steps.

***Cheap*:** No specialized equipment or glassware is required to achieve the pesticide residue analysis.

***Effective*:** General procedure for all food and beverage matrices allowing a significant reduction of the analysis cost.

***Rugged*:** Useful for the treatment of complex food matrices such as fish, meat or nuts without the requirement of additional treatments.

***Safe*:** Limited time of contact with dangerous compounds and solvents.

Traditional Liquid & Solid Extractions for Sample Preparation involve a number of issues that QuEChERS cam sort out:

Liquid-Phase Extractions are:

- Labor intensive
- Necessitate large volumes of organic solvents, including halogenated solvents
- Difficult to automate
- Emulsion prone
- Not specific to a given analyte

Solid-Phase Extractions are:

- Complex and difficult to master
- Necessitate large volumes of organic solvents, including halogenated solvents
- Lengthy to develop: necessitate time & efforts in method development
- Offer too many possibilities to choose from
- Ineffective toward many analytes

Advantages of QuEChERS over Traditional Sample Preparation

Advantages of QuEChERS Over Traditional Sample Preparation			
	Traditional SPE	**QuEChERS**	**QuEChERS Benefits**
Estimated time to process 6 samples	120 minutes	20 minutes	About 6 times faster
Solvent used per sample	90 mL	10 - 15 mL	About 6 - 9 times less solvent needed
Chlorinated waste	30 mL	None	Safer, greener, less costly
Gassware and specialized equipment	Clean separatory funnels, waterbath, round bottom flasks, rotary evaporator…	Centrifuge	No additional supplies needed

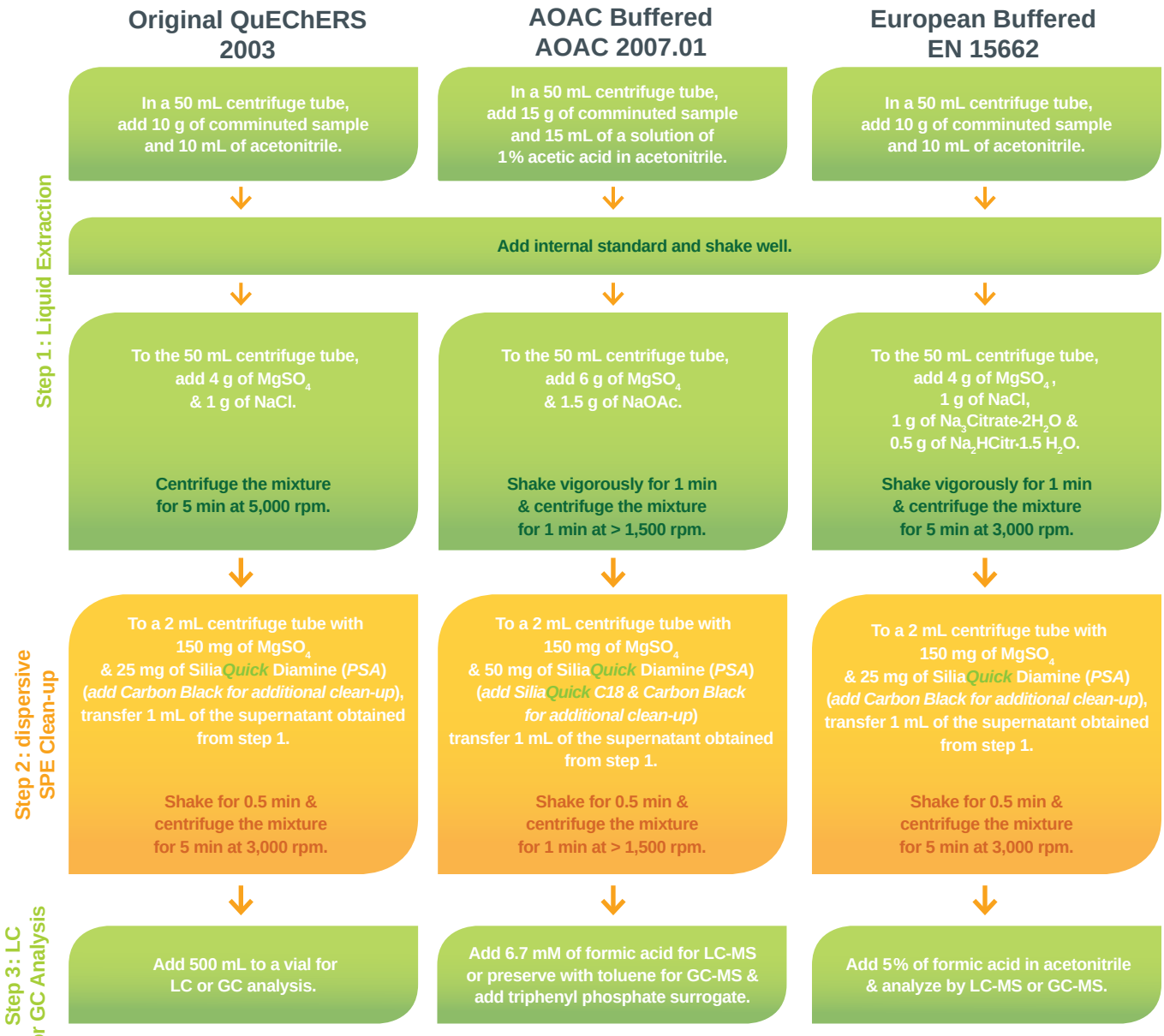
Remember, QuEChERS is the only sample preparation technique in line with green chemistry principles and it is highly effective.

Extraction and Dispersive Reagents

The following table presents each extraction and dispersive reagent and their specific functions in the QuEChERS technique.

Extraction and Dispersive Reagents	
Extraction Reagents	**Specific Function**
Anhydrous Magnesium Sulfate ($MgSO_4$)	Facilitates solvent partitioning.
Acetic Acid	Used for pH adjustment.
Acetonitrile	Solvent providing the best characteristics for extracting a wide variety of pesticides. Amenable for both LC and GC analysis.
Buffers	Maintain optimal pH and prevent pH degradation of sensitive analytes.
Sodium Chloride (*NaCl*)	Limits the amount of polar interferences.
Dispersive Reagents	**Specific Function**
Silia*Quick* Diamine (Primary Secondary Amine)	Removes sugars, fatty acids, organic acids, lipids, and some pigments. Sterols and additional lipids can also be removed in combination with Silia*Quick* C18.
Silia*Quick* Amine	Removes sugars and fatty acids as well as the Silia*Quick* Diamine but is less likely to catalyze degradation of base sensitive analytes.
Silia*Quick* C18	Removes long chain, non-polar compounds, and sterols.
Graphitized Carbon Black	Removes pigments, polyphenols, and other polar compounds.
Anhydrous Magnesium Sulfate ($MgSO_4$)	Removes residual water from the organic phase.

Schematic Flow Chart of the Most Used QuEChERS Technique



Note : Please note that these procedures are a convenient starting point for method development but are only meant to orient. Further optimization depending on the analyte of interest may be required to tailor the method to your application needs and maximize your LC-MS or GC-MS analysis.

$MgSO_4$: Anhydrous Magnesium Sulfate
NaCl : Sodium Chloride
$Na_3Citrate•2H_2O$: Trisodium Citrate Dihydrate
$Na_2HCitr•1.5H_2O$: Disodium Hydrogencitrate Sesquihydrate

PSA : Primary Secondary Amine - Silia*Quick* Diamine
GCB : Graphitized Carbon Black
C18 : C18 Fonctionnalized Silica - Silia*Quick* C18

How to Choose the Proper Silia*Quick* QuEChERS Kit

Step 1: For Liquid Extraction

Extract the pesticides or analytes of interest into an organic layer by relying on the perfect combination of salts and organic solvents (*usually acetonitrile*).

The table below presents the Silia*Quick* QuEChERS Liquid Extraction kits specially pre-packed with anhydrous salts and/or sorbents to suit the QuEChERS technique of your choice.

Selection Criteria:

- For base or heat-sensitive analytes, use buffered method.
- With doubt, use buffered method.

BLEND AND WEIGH

Original Method	Buffered Methods AOAC 2007.01 Method	 EN 15662 Method
10 g Sample	15 g Sample	10 g Sample
4 g $MgSO_4$; 1.5 g NaCl	6 g $MgSO_4$; 1.5 g NaOAc	4 g $MgSO_4$; 1 g NaCl; 1 g SCTD; 0.5 g SCDS
PN: QE-0001-100P (*packets only*) PN: QE-0001-100K (*packets & tubes*)	PN: QE-0002-100P (*packets only*) PN: QE-0002-100K (*packets & tubes*)	PN: QE-0003-100P (*packets only*) PN: QE-0003-100K (*packets & tubes*)

$MgSO_4$ = Anhydrous Magnesium Sulfate , **NaCl** = Sodium Chloride, **NaOAc** = Sodium Acetate, **GCB** =Graphitized Carbon Black, **SCTD**=Sodium Citrate Dibasic Sesquihydrate, **SCDS**=Sodium Citrate Tribasic Dihydrate

Step 2: For dispersive Solid-Phase Extraction Clean-Up

An aliquot of the organic layer from the 1st step is subjected to further clean-up. This step helps selectively remove unwanted interferences such as lipids and pigments but NOT your analytes of interest!

The following table presents the 4 types of SiliaQuick QuEChERS dispersive Solid-Phase Extraction clean-up kits to match your food matrices.

dSPE kits contain pre-weighed sorbents/salts inside 2 mL or 15 mL centrifuge tubes. We recommend using 2 mL dispersive tubes for an extract volume of 1 mL and 15 mL dispersive tubes for extract volumes higher than 3 mL.

Selection Criteria:

- Aliquot size is specified by the method.
- Kits are created for these specific amounts.
- 4 easy-to-choose typed of food matrices.
- Colour-coded caps that match matrix category for reduced error.
- Two methods depending on molecules to be removed.

Check pH and adjust to pH 5 – 5.5
Add internal standards aliquot
SHAKE AND CENTRIFUGE

Cap Color for 2 mL tubes	Matrix	2 mL tubes for small extract volumes		15 mL tubes for large extract volumes	
		AOAC 2007.01	EN 15662	AOAC 2007.01	EN 15662
Clear	General matrices •Apples •Bananas •Broccoli •...	150 mg $MgSO_4$ 50 mg PSA PN: QD-1000-2T	150 mg $MgSO_4$ 25 mg PSA PN: QD-1001-2T	1200 mg $MgSO_4$ 400 mg PSA PN: QD-2000-15T	900 mg $MgSO_4$ 150 mg PSA PN: QD-2001-15T
Pink	Pigmented matrices •Lettuces •Peppers •Strawberries •...	150 mg $MgSO_4$ 50 mg PSA 50 mg GCB PN: QD-1002-2T	150 mg $MgSO_4$ 25 mg PSA 2.5 mg GCB PN: QD-1003-2T	1200 mg $MgSO_4$ 400 mg PSA 400 mg GCB PN: QD-2002-15T	900 mg $MgSO_4$ 150 mg PSA 15 mg GCB PN: QD-2003-15T
Green	Highly pigmented matrices •Urine •Avocados •Coffee •...	150 mg $MgSO_4$ 50 mg PSA 50 mg GCB 50 C18 PN: QD-1004-2T	150 mg $MgSO_4$ 25 mg PSA 7.5 mg GCB PN: QD-1005-2T	1200 mg $MgSO_4$ 400 mg PSA 400 mg GCB 400 mg C18 PN: QD-2004-15T	900 mg $MgSO_4$ 150 mg PSA 45 mg GCB PN: QD-2005-15T
Blue	Fatty and waxed matrices •Milk •Shrimps •Blood •Liver •...	150 mg $MgSO_4$ 50 mg PSA 50 mg C18 PN: QD-1006-2T	150 mg $MgSO_4$ 25 mg PSA 25 mg C18 PN: QD-1007-2T	1200 mg $MgSO_4$ 400 mg PSA 400 mg C18 PN: QD-2006-15T	900 mg $MgSO_4$ 150 mg PSA 150 mg C18 PN: QD-2007-15T

$MgSO_4$ = anhydrous Magnesium Sulfate, **PSA** = SiliaQuick Primary Secondary Diamine, **GCB** = Graphitized Carbon Black, **C18** = SiliaQuick C18

Step 3: For dispersive Solid-Phase Extraction Clean-Up

The extract, containing the analyte(s) is ready to be analysed by GC or HPLC with MS, MS/MS or your selective detector.

Selection Criteria:

- Consider your application (*analyte MW, solubility*).
- Consider the stationary phase (*column chemistry, separation mode, particle size, retention capacity*).
- Column dimensions (*internal diameter, length*).

SHAKE AND CENTRIFUGE

SiliaChrom dt C18	SiliaChrom XDB C18	SiliaChrom SCX
Universal C18 for most popular applications (*highest purity silica gel*)	Ideal for barbiturates, fat-soluble vitamins, fatty acids, steroids	Ideal for charged analytes

Check pages 112 - 165 of our Analytical Catalog, visit our website or contact us for more information

Bulk Sorbents Available for Your Own Recipe Creation

SiliCycle Bulk Sorbents for QuEChERS		
Description	**Available Quantities**	**Product Number**
Silia*Quick*™ C18	1 g 5 g 10 g 25 g 50 g 100 g 250 g 500 g 1 kg 5 kg 10 kg 25 kg … up to multi-ton Contact us for details	AUT-1313
Silia*Quick*™ Anhydrous Magnesium Sulfate ($MgSO_4$)		AUT-0310
Silia*Quick*™ Primary Secondary Amine (*PSA*)		AUT-0312
Silia*Quick*™ Amine		AUT-0412
Silia*Quick*™ Graphitized Carbon Black (*GCB*)		AUT-0311

New to QuEChERS Technology? We Have Starter Kits to Introduce This New Technique:

Sili*Quick* QuEChERS Starter Kits			
PN	**Product Name**	**Tube Volume (*mL*)**	**Qty/box**
QD-2001-15K	Silia*Quick*™ Extraction + dSPE Clean-up Kit for EN 15662 (*QE-0003-100K* + *QD-2001-15T*)	50 + 15	50 + 50
QD-1004-2K	Silia*Quick*™ Extraction + dSPE Clean-up Kit for AOAC 2007.01 (*QE-0002-100K* + *QD-1004-2T*)	50 + 2	100 + 100
QD-2004-15K	Silia*Quick*™ Extraction + dSPE Clean-up Kit for AOAC 2007.01 (*QE-0002-100K* + *QD-2004-15T*)	50 + 15	50 + 50
QD-1007-2K	Silia*Quick*™ Extraction + dSPE Clean-up Kit for EN 15662 (*QE-0003-100K* + *QD-1007-2T*)	50 + 2	100 + 100
QD-2007-15K	Silia*Quick*™ Extraction + dSPE Clean-up Kit for EN 15662 (*QE-0003-100K* + *QD-2007-15T*)	50 + 15	50 + 50

SiliaQuick QuEChERS Tips & Troubleshooting

When facing poor recovery of pesticide compounds:

Step 1:

- Each sample has to be at the minimum 80% hydrated to perform optimal liquid extraction.
- Homogenization is a key step and can significantly impact your results.
- Freezing sample at -20 °C can significantly improve the breaking behavior of the sample in order to obtain a higher fineness and homogeneity.
- Great care must be taken in keeping the sample cool, as many pesticides are volatile and some analytes are heat-sensitive. Some mills will continuously cool the sample with liquid nitrogen while grinding.
- Homogenize with dry ice until a fine powder is obtained.
- For base sensitive compounds use buffered method. If you don't know, use buffered method.
- If analyte is sensible, always mix the sample with the solvent first to reduce the exothermic reaction produced by the addition of Magnesium Sulfate (*hence, choose packets instead of pre-filled tubes*).

Step 2:

- Choose minimal possible tube size. Aliquot size is specified by the method, and kits are created for these specific amounts.
- You can build your own recipe in function of your unwanted interferences using bulk sorbents.

Step 3:

- Add an analyte protector like toluene or sorbitol to prevent loss of thermally unstable pesticides in the GC inlet.
- Add formic acid after the dispersive SPE clean-up step to limit the degradation of base sensitive compounds prior the LC analysis.

Select the Right Silia*Quick* QuEChERS dSPE Clean-Up Kit According to your Matrix Type

The Silia*Quick* QuEChERS dispersive Solid-Phase Extraction clean-up kits are assembled to match different types of matrices. Here is below a table with multiple examples of typical matrices, to help you choose the right kit and combination of sorbents.

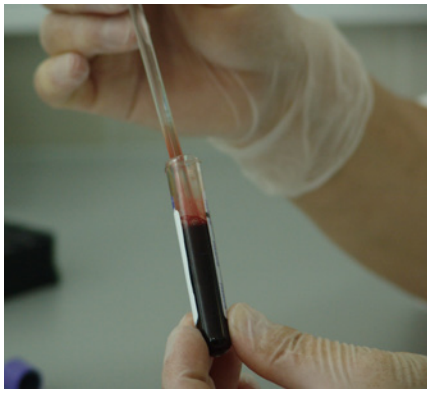
Silia*Quick* QuEChERS Dispersive Solid-Phase Extraction Kits				
Food Matrices	**General Fruits & Vegetables**	**Pigmented Fruits & Vegetables**	**Highly Pigmented and Fatty Fruits & Vegetables**	**Fatty and Waxed Fruits & Vegetables**
Root and Tuber Vegetables				
Beets				
Carrot				
Radish				
Potato				
Fruiting Vegetables				
Eggplant				
Cucumber				
Pepper *(green or red)*				
Pumpkin				
Tomato				
Cabbage				
Broccoli				
Brussels sprouts				
Cauliflower				
Stem Vegetables				
Aparagus				
Celery				
Leek				
Rhubarb				
Leafy Vegetables				
Lettuce				
Basil				
Parsley				
Spinach				
Leek Plants				
Garlic				
Onion				
Shallot				

Choose your Silia*Quick* QuEChERS Dispersive SPE Clean-Up Kits by Food Types *(con't)*

Silia*Quick* QuEChERS Solid-Phase Extraction Kits				
Food Matrices	**General Fruits & Vegetables**	**Pigmented Fruits & Vegetables**	**Highly Pigmented and Fatty Fruits & Vegetables**	**Fatty and Waxed Fruits & Vegetables**
Small Fruits				
Blackberry				
Blueberry				
Grapes *(red)*				
Cranberry				
Strawberry				
Pome Fruits				
Apple				
Pear				
Quince				
Citrus Fruits				
Grapefruit				
Lemon & Lime				
Orange				
Tangerine				
Stone Fruits				
Apricot				
Cherry				
Peach				
Plum				
Other Fruits				
Avocado				
Banana				
Mango				
Pineapple				
Other				
Cereals *(wheat, corn, rice)*				
Coffee beans				
Tea Leaves				

Choose your Silia*Quick* QuEChERS Dispersive SPE Clean-Up Kits by Food Types *(con't)*

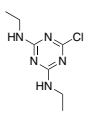
SiliaQuick QuEChERS Solid-Phase Extraction Kits				
Food Matrices	**General Fruits & Vegetables**	**Pigmented Fruits & Vegetables**	**Highly Pigmented and Fatty Fruits & Vegetables**	**Fatty and Waxed Fruits & Vegetables**
Animal Proteins				
Organs (*liver, intestine...*)				
Milk				
Shrimps & seafood				
Biological Fluids				
Blood				
Urine				



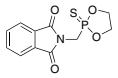
Case study: Detection of Multiple Pesticides Residues in Canadian Apples

The QuEChERS approach was used by SiliCycle scientists for the extraction and clean-up of nine of the most used pesticides for apple-growing sector in Canada. The application outlines the AOAC 2007.01 methodology, and exacts from this procedure were then diverted to HPLC/MS/MS for analysis.

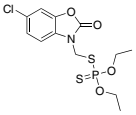
Pesticides Analyzed



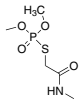
Simazine



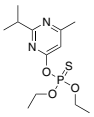
Phosmet



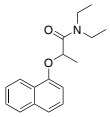
Phosalone



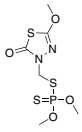
Dimethoate



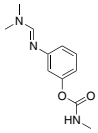
Diazinon



Napropamide



Methidation



Formetanate

Carbaryl

Silia*Quick* Kits Used

- **STEP 1: PN: QE-0002-100P (*6 g $MgSO_4$ & 1.5 g NaOAc*)**
    - 10 g of apple matrix was weighed in a 50 mL centrifuge tube.
    - 10 mL of H_2O was added.
    - Mixture was homogenized.
    - Salt packet was added.
    - Tube was vortexed for 30 sec and then centrifuged at 3,000 rpm for 5 min.

- **STEP 2**: **PN: QD-2000-15T (*«AOAC method for General Fruits & Veggies» : 1,200 mg $MgSO_4$, 400 mg PSA*)**
    - Supernatant was transfered into a 15 mL dSPE.
    - Tube was vortexed for 30 sec and then centrifuged at 3,000 RPM for 5 min.

- **STEP 3**: **Silia*Chrom* dt C18 PN: H141802E-G050 *(3.0 x 50 mm, 2.5 µm, 100 Å)***
    - Extract, containing the pesticides, was transferred to a 2 mL, 9 mm wide opening vial PN: **2SW-C9-C** and then injected for subsequent analysis.

Instruments Conditions

HPLC Conditions

MOBILE PHASE	MPA: 1 mM Ammonium Formate in 95/5 H_2O/MeCN, 0.1 % Formic Acid (v/v) MPB: 1 mM Ammonium Formate in 5/95 H_2O/MeCN, 0.1 % Formic Acid (v/v)
COLUMN	Silia*Chrom*® dt C18, 2.5 µm
COLUMN SIZE	3.0 x 50 mm
SILICYCLE PN	H141802E-G050
FLOW RATE	0.600 mL/min
TEMPERATURE	23°C
INJECTION VOL	2 µL

MS/MS Detection

DETECTOR	Sciex API 3000
IONISATION MODE	ESI+
GAS FLOW	8,000 cc/min
TEMPERATURE	375°C

Experimental Results

Quality Control					
Pesticide	***LLQC**	***QC1** (*3 X LLOQ*)	***QC2** (*30% ULQC*)	***QC3** (*70% ULQC*)	***ULQC**
Carbaryl	92 ± 3	103 ± 9	96 ± 5	95 ± 3	95 ± 2
Diazinon	102 ± 7	104 ± 2	96 ± 1	97 ± 2	98 ± 2
Dimethoate	106 ± 3	105 ± 5	98 ± 3	93 ± 1	95 ± 1
Formetanate	101 ± 6	99 ± 3	93 ± 2	97 ± 2	97 ± 2
Methidation	95 ± 7	103 ± 4	101 ± 2	96 ± 4	96 ± 3
Napropamid	92 ± 10	102 ± 6	95 ± 3	95 ± 2	95 ± 1
Phosalone	83 ± 4	103 ± 6	94 ± 5	98 ± 1	94 ± 5
Phosmet	85 ± 5	96 ± 6	102 ± 5	100 ± 6	98 ± 6
Simazine	106 ± 8	103 ± 4	97 ± 1	94 ± 2	95 ± 2

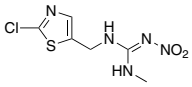
Concentration Levels						
Pesticide	**LLOQ** (*%*)	**3 X LLOQ**	**30% ULOQ**	**70% ULOQ**	**ULOQ**	**LOQ** (*ppt*)
Carbaryl	100 ± 3	113 ± 2	104 ± 3	103 ± 3	103 ± 3	0.05
Diazinon	94 ± 4	101 ± 2	99 ± 2	102 ± 1	103 ±1	0.03
Dimethoate	109 ± 8	96 ± 7	110 ± 3	104 ± 2	105 ± 2	0.10
Formetanate	88 ± 6	87 ± 1	81 ± 1	86 ± 1	89 ± 1	0.50
Methidation	109 ± 2	110 ± 3	109 ± 2	106 ± 1	108 ± 2	0.06
Napropamid	91 ± 9	103 ± 5	107 ± 2	106 ± 1	107 ± 1	0.05
Phosalone	97 ± 8	106 ± 4	110 ± 2	115 ± 3	111 ± 4	0.20
Phosmet	112 ± 2	94 ± 7	100 ± 1	101 ± 1	106 ± 4	0.25
Simazine	107 ± 10	107 ± 7	106 ± 3	106 ± 2	106 ± 2	0.25

- The **accuracy** of the method, expressed as **recovery**, was between 81 and 113 %
- The **precision**, expressed as RSD, was between 0.3 and 11.6 %
- The established limit of quantification (*LOQ*) was 25 ng/g, which is significantly lower than the respective Maximum Residue Limit for such pesticides in food by the FDA and Santé Canada

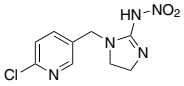
Case Study: Detection of Common Neonicotinoids Residues in Honey

Clothianidin, Imidacloprid and Thiamethoxan are three of the most common neonicotinoids, which is a type of neuroactive insecticide. In January 2013, the European Food Safety Authority declared that these three neonicotinoids pose a high risk for bees and set up maximum residue limits in honey. The QuEChERS approach was used by SiliCycle scientists for the extraction and clean-up of nine of the most used pesticides in apple-growing sector in Canada. The QuEChERS AOAC technique coupled with LDTDMS/ MS was used to obtain a clean extract of honey and a high extraction recovery for these insecticides.

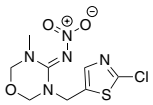
Pesticides Analyzed



Clothianidin



Imidacloprid



Thiamethoxam

Silia*Quick* Kits Used

- **STEP 1**: **PN: QE-0002-100K *(tube with 6 g $MgSO_4$ & 1.5 g NaOAc)***
    - 1 g of honey was weighed in a 50 mL centrifuge tube containing the salts
    - Sample was diluted and homogenized in 10 mL of saturated H_2O with NaCl and spiked with the desired concentration
    - 3 mL of ACN was added and tube was vortex

- **STEP 2**: **PN: QD-1006-2T (*«AOAC method for Fatty and Waxed Fruits & Veggies» : 150 mg $MgSO_4$, 50 mg PSA, 50 mg 18*)**
    - Supernatant was transfered into a 2 mL dSPE tube
    - Tube was vortexed for 30 sec and then centrifuged at 14,000 RPM for 2 min

- **STEP 3**: **Silia*Chrom* dt C18 PN: H141802E-G050 (*3.0 x 50 mm, 2.5 µm, 100 Å*)**
    - A 4 µL extract, containing the pesticides, was transferred to a 2 mL, 9 mm wide opening vial PN: **2SW-C9-C** and then injected for subsequent analysis

Instruments Conditions

LTDT Conditions

FLOW RATE	3 mL/min
TEMPERATURE	22°C
INJECTION VOL	4 µL
LASER PATTERN	

Time (*s*)	Power (%)
0.0	0
2.0	0
5.0	65
6.0	65
6.1	0
8.0	0

MS/MS Detection

Insecticide	Transition	CE	DP
Clothianidin	250.1 -> 169.1	17	80
Imidacloprid	292.1 -> 211.1	17	80
Thiamethoxan	256.1 -> 209.1	20	80

- MODE : Positive

Experimental Results

Linearity Results

Excellent linearity (*r2 > 0.99*) with no sign of carryover effect was achieved within the quantification range (*10 to 500 ng/g of honey for Clothianidin and Thiamethoxan and 25 to 1,250 ng/g of honey for Imidacloprid*) without the use of an internal standard.

Clothianidin					
	LLOQ	**Low-QC**	**Mid-QC**	**High-QC**	**ULOQ**
Conc. (*ng/g honey*)	10	25	100	250	500
N	3	3	3	3	3
Mean (*ng/g honey*)	9.18	28.42	89.13	277.41	497.44
% RSD	7.3	4.5	11.1	3.4	10.1
% Nom	91.83	113.67	89.13	110.96	99.49

Thiamethoxan					
	LLOQ	**Low-QC**	**Mid-QC**	**High-QC**	**ULOQ**
Conc. (*ng/g honey*)	10	25	100	250	500
N	3	3	3	3	3
Mean (*ng/g honey*)	9.50	27.11	110.37	276.45	497.98
% RSD	14.3	9.9	4.5	2.7	10.7
% Nom	94.85	108.44	110.37	110.58	99.60

Imidacloprid					
	LLOQ	**Low-QC**	**Mid-QC**	**High-QC**	**ULOQ**
Conc. (*ng/g honey*)	25	63	250	625	1250
N	3	3	3	3	3
Mean (*ng/g honey*)	23.70	73.48	224.95	690.64	1213.12
% RSD	9.3	4.1	8.1	5.2	12.3
% Nom	94.81	117.56	89.98	110.50	97.05

- The accuracy of the method, expressed as recovery, was between 89 and 117 %.
- The precision, expressed as RSD, was between 2.7 and 14.3 %.
- The established limit of quantification (*LOQ*) for Clothianidin, imidacloprid and thiamethoxan respectively was found to be 9, 24 and 9 ng/g, hence falling within the respective maximum residue limit for such insecticide in honey set by the FDA, Santé Canada and the European Food Safety Authorities.

Matrix Effect

Matrix effect was also evaluated by adding a known concentration of neonicotinoids (*50 ng/g of honey for Clothianidin and Thiamethoxan, and 125 ng/g of honey for Imidacloprid*) in different honey brands. All non-spiked compounds were negative for neonicotinoids. As shown in tables below, no matrix effect was observed between four different honey brands.

Clothianidin				
	A	B	C	D
Conc. (*ng/g honey*)	50	50	50	50
N	3	3	3	3
Mean (*ng/g honey*)	45.44	57.74	48.28	57.28
% RSD	9.1	2.1	14.4	10.6
% Nom	90.88	115.47	96.57	114.57

Imidacloprid				
	A	B	C	D
Conc. (*ng/g honey*)	125	125	125	125
N	3	3	3	3
Mean (*ng/g honey*)	116.79	132.30	122.20	138.16
% RSD	12.7	14.5	14.1	3.4
% Nom	93.43	105.84	97.76	110.52

Thiamethoxan				
	A	B	C	D
Conc. (*ng/g honey*)	50	50	50	50
N	3	3	3	3
Mean (*ng/g honey*)	42.62	57.60	51.22	48.83
% RSD	13.4	10.7	13.2	9.2
% Nom	85.25	115.20	102.44	97.66

Hence, no matrix effect was observed. LDTD-MS/MS provides the high-throughput analysis of clothianidin, imidacloprid and thiamethoxan in honey in **9 seconds sample-to-sample** without carryover.

Please contact us for more applications and case studies, such as:

- Detection and analysis of Oxytetracyclines and Sulfadiazines in Shrimps.
- Detection and analysis of Plant Protection Products (*PPPs*) in different Tobacco Grades.
- Detection and analysis of Pesticides Residues in Green Tea.
- Detection and analysis of Nicotine and Nicotine Metabolites in Biological Fluids.
- Detection and analysis of Pesticides Residues in Rice.
- Detection and analysis of over 20 Veterinary Drugs in Animal Food.