[DISQUE DISPERSIVE SAMPLE PREPARATION EXTRACTION PRODUCTS]

QuEChERS Simplifed

DisQuE™
Dispersive Sample Preparation

Waters
THE SCIENCE OF WHAT'S POSSIBLE.®

DisQuE™
Dispersive Sample Preparation

The QuEChERS method (an acronym for **Q**uick, **E**asy, **C**heap, **E**ffective, **R**ugged, and **S**afe) followed by dispersive solid-phase extraction (d-SPE), is a simple and straightforward sample preparation technique ideal for multi-residue analysis for pesticides, veterinary drugs, and mycotoxins in a wide variety of food and agricultural products. DisQuE Dispersive Sample Preparation Extraction Products are conveniently packaged with pre-weighed sorbents and buffers in pouches and tubes as described in regulatory methods and protocols.

The Science Behind DisQuE

To overcome the deficiencies of traditional methods, an improved extraction procedure was developed (Anastassiades et al.)* to extract pesticides from fruits and vegetables. The procedure is based on an initial single-phase extraction using 10 or 15 g sample with acetonitrile at 1 mL acetonitrile per 1 g of sample. A liquid-liquid partition is created by adding excess salts and buffers to the extract. After centrifugation, the acetonitrile layer, containing the pesticide, is collected. The matrix can be further cleaned and the excess water removed with a single d-SPE step by mixing acetonitrile extract with anhydrous $MgSO_4$ and primary secondary amine (PSA) sorbents. The biggest advantage, besides time and effort, is that the final extract can be analyzed directly by either GC-MS or by LC-MS/MS technology with a simple dilution.

Some QuEChERS methods add buffers during the extraction step. Today, the two most common methods are the European Committee for Standardization (CEN) Method 15662 which uses citrate buffer for extraction and the Association of Analytical Communities (AOAC) Official Method 2007.01 using acetic acid buffer.

*Anastassiades, M., Lehotay, S.J., Tajnbaher, D., & Schenck, F.J. (2003) *J. AOAC. Int.* **86**, 412-431

Method	Sample Size	Solvent	Tube Content
CEN Method 15662 Citrate Buffer	10 g	10 mL acetonitrile	4 g $MgSO_4$, 1 g NaCl 1.5 g sodium citrate
AOAC Method 2007.01 Acetate Buffer	15 g	15 mL 1% acetic acid in acetonitrile	6 g $MgSO_4$ 1.5 g sodium acetate

Kit choice based on preferred method

Low Water Content Commodity Extraction

QuEChERS extraction is designed for multi-residue pesticide analysis of fruits and vegetables with high water content. For QuEChERS analysis of commodities with low-water content, additional water is added to optimize the extraction. CEN method guidelines for adding water are shown below.

Sample Type	Sample Weight	Water Added	Note
Fruits and vegetables > 80% water content	10 g	–	
Fruits and vegetables 25-80% water content	10 g	X g	X = 10 g - water amount in 10 g sample
Cereals	5 g	10 g	
Dried fruits	5 g	7.5 g	Water can be added during comminution step
Honey	5 g	10 g	
Spices	2 g	10 g	

CEN Method Guidelines for Adding Water into Commodities with Low Water Content

DisQuE Workflow

Simplified QuEChERS Method Sample Preparation Protocol

402 Pesticide Residues at 10 ppb ng/g In One 10 Minute Run

UPLC® Conditions

LC system:	Waters ACQUITY UPLC® System
Column:	ACQUITY UPLC BEH C_{18} 1.7 µm, 2.1 x 100 mm
Part number:	186002352
Column temp.:	40 °C
Sample temp.:	4 °C
Flow rate:	0.450 mL/min
Mobile phase A:	98:2 water: methanol + 0.1% formic acid
Mobile phase B:	Methanol + 0.1% formic acid

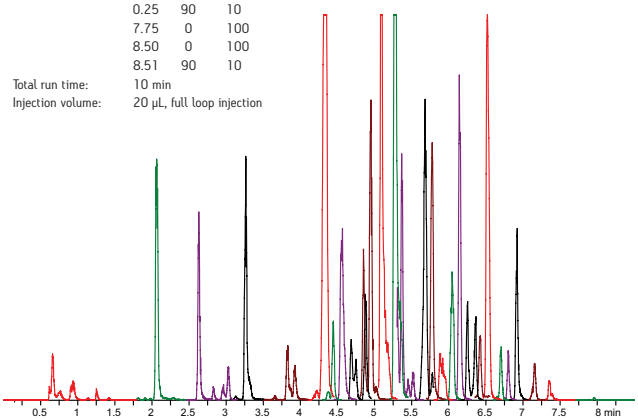
Gradient:

Time	A%	B%
0.00	90	10
0.25	90	10
7.75	0	100
8.50	0	100
8.51	90	10

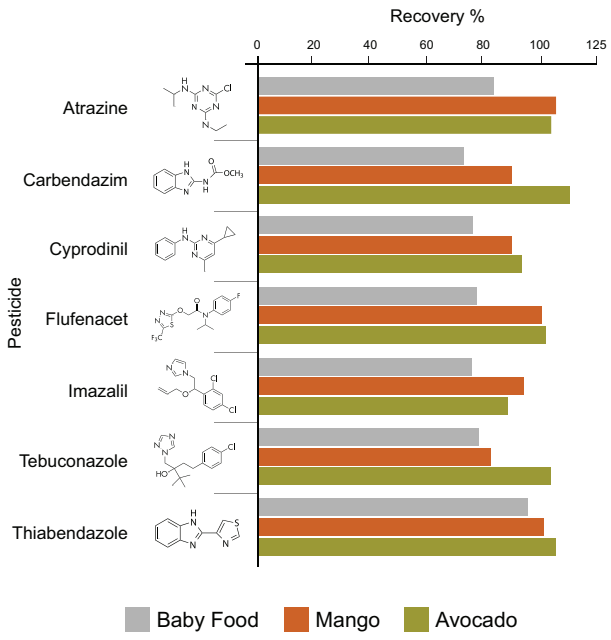
Total run time:	10 min
Injection volume:	20 µL, full loop injection

MS Conditions

MS system:	Waters ACQUITY® TQ Detector
Ionization mode:	ESI positive polarity
Capillary voltage:	1 kV
Desolvation gas:	Nitrogen, 800 L/hr, 400 °C
Cone gas:	Nitrogen, 5 L/hr
Source temp.:	120 °C
Acquisition:	Multiple Reaction Monitoring (MRM)
Collision gas:	Argon at 3.5 mBar



Recovery Data for Three Types of Sample Matrices Fortified at 10 ng/g



Selection of d-SPE Product for Sample Cleanup

Commodity Type	Cleanup Provided	Size	AOAC Method 2007.01	CEN Method 15662
General Fruits and Vegetables (Celery, Head Lettuce, Melon)	Removes polar organic acids, some sugars and lipids	2 mL Tubes	50 mg PSA, 150 mg $MgSO_4$ Part #186004572	25 mg PSA, 150 mg $MgSO_4$ Part #186004831
		15 mL Tubes	400 mg PSA, 1200 mg $MgSO_4$ Part #186008072	150 mg PSA, 900 mg $MgSO_4$ Part #186004833
Fruits and Vegetables with Fats and Waxes (Cereals, Nuts, Dairy, Avocado)	Removes polar organic acids, some sugars, more lipids and sterols	2 mL Tubes	50 mg PSA, 50mg C_{18}, 150 mg $MgSO_4$ Part #186004830	25 mg PSA, 25 mg C_{18}, 150 mg $MgSO_4$ Part #186004832
		15 mL Tubes	400 mg PSA, 400 mg C_{18}, 1200 mg $MgSO_4$ Part # 186008073	150 mg PSA, 150 mg C_{18}, 900mg $MgSO_4$ Part #186004834

Cleaning Up Pigmented Fruits and Vegetables

Some fruits and vegetables, such as spinach, red sweet pepper, and carrots, have high content of non-polar pigments, such as carotenoids or chlorophyll. QuEChERS effectively removes some common matrix constituents, such as fatty acids and sugars; however, samples that contain high levels of pigment require additional treatment. Graphitized carbon black (GCB) effectively removes these pigments, and can be added to the clean-up tube with PSA and $MgSO_4$ during the d-SPE cleanup step. The clean-up of pigments is especially critical for gas chromatographs because pigments often accumulate in the injection port, quickly fouling the injection liner.

The effect of GCB amounts on the de-coloring of spinach extracts.

No Cleanup | PSA Only | PSA + 2.5 mg GCB | PSA + 7.5 mg GCB | PSA + 12.5 mg GCB | PSA + 25 mg GCB | PSA + 50 mg GCB

Successful laboratories demand timely, high quality results. Waters scientists carefully evaluate product performance and can recommend methods for sample preparation, analytical separation, and instrument optimization for a wide array of food adulterants. This expert advice is readily available in our Food Testing Application Notebook.

For your copy, visit **www.waters.com/FT**

Ordering Information

Kits

Name	Description	Qty	Part No.	
DisQuE Dispersive SPE Kit–Pouch Format (AOAC Method 2007.01)	Pouch: 1.5 g sodium acetate and 6 g $MgSO_4$ 50 mL Tube: Empty 2 mL Tube: 150 mg $MgSO_4$ and 50 mg PSA	100	176002922	
DisQuE Dispersive SPE Kit–Pouch Format (CEN Method 15662)	Pouch: 1 g trisodium citrate dihydrate, 0.5 g disodium hydrogencitrate sesquihydrate, 1 NaCl and 4 g $MgSO_4$ 50 mL Tube: Empty 2 mL Tube: 150 mg $MgSO_4$, 25 mg PSA, and 25 mg C_{18}	100	176002923	
DisQuE Dispersive SPE Kit (AOAC Method 2007.01)	50 mL Tube: 1.5 g sodium acetate, 6 g $MgSO_4$ 2 mL Tube: 150 mg $MgSO_4$, 50 mg PSA	100	176001676	
DisQuE Dispersive SPE Kit (CEN Method 15662)	50 mL Tube: 4 g $MgSO_4$, 1 g NaCl, 1 g trisodium citrate dehydrate, 0.5 g disodium hydrogencitrate sesquihydrate 2 mL Tube: 150 mg $MgSO_4$, 25 mg PSA, 25 mg C_{18}	100	176001903	

Extraction Tubes

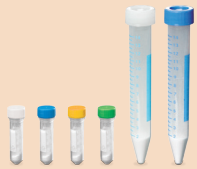
Name	Description	Qty	Part No.	
50 mL Empty Tube for QuEChERS Extraction	—	50	186006814	
50 mL Tube–Blue Cap (AOAC Method)	1.5 g sodium acetate, 6 g $MgSO_4$	100	186004571	
50 mL Tube–White Cap (CEN Method)	4 g $MgSO_4$, 1 g NaCl, 1 g trisodium citrate dehydrate, 0.5 g disodium hydrogencitrate sesquihydrate	100	186004837	

Sorbent Pouches

Name	Contents	Qty	Part No.	
DisQuE Pouch (AOAC Method)	1.5 g sodium acetate, 6 g $MgSO_4$	50	186006812	
DisQuE Pouch (CEN Method)	4 g $MgSO_4$, 1 g NaCl, 1 g trisodium citrate dehydrate, 0.5 g disodium hydrogencitrate sesquihydrate	50	186006813	

Clean-Up Tubes

Name	Contents	Qty	Part No.
2 mL Tube–White Cap	150 mg $MgSO_4$, 50 mg PSA	100	186004572
2 mL Tube–Blue Cap	150 mg $MgSO_4$, 50 mg PSA, 25 mg C_{18}	100	186004830
2 mL Tube–Yellow Cap	150 mg $MgSO_4$, 25 mg PSA	100	186004831
2 mL Tube–Green Cap	150 mg $MgSO_4$, 25 mg PSA, 25 mg C_{18}	100	186004832
2 mL Tube–Blue Cap	150 mg $MgSO_4$, 25 mg PSA, 25 mg C_{18}, 7 mg GCB	100	186008071
2 mL Tube–Yellow Cap	150 mg $MgSO_4$, 50 mg C_{18}	100	186008075
2 mL Tube–Blue Cap	150 mg $MgSO_4$, 25 mg PSA, 2.5 mg GCB	100	186008076
2 mL Tube–Blue Cap	150 mg $MgSO_4$, 50 mg PSA, 30 mg C_{18}, 30 mg Al-N	100	186008081
15 mL Tube–White Cap	900 mg $MgSO_4$, 150 mg PSA	50	186004833
15 mL Tube–Blue Cap	900 mg $MgSO_4$, 150 mg PSA, 150 mg C_{18}	50	186004834
15 mL Tube–Clear Cap	1200 mg $MgSO_4$, 400 mg PSA	50	186008072
15 mL Tube–Blue Cap	1200 mg $MgSO_4$, 400 mg PSA, 400 mg C_{18}	50	186008073
15 mL Tube–Blue Cap	1200 mg $MgSO_4$, 400 mg PSA, 400 mg C_{18}, 400 mg GCB	50	186008074
15 mL Tube–Clear Cap	900 mg $MgSO_4$, 300 mg PSA	50	186008077
15 mL Tube–Blue Cap	900 mg $MgSO_4$, 300 mg PSA, 300 mg C_{18}	50	186008078
15 mL Tube–Blue Cap	900 mg $MgSO_4$, 450 mg PSA, 300 mg C_{18}, 50 mg GCB	50	186008079
15 mL Tube–Blue Cap	750 mg $MgSO_4$, 250 mg PSA, 150 mg C_{18}, 150 mg Al-N	50	186008080



Al-N = Alumina N

Bulk Sorbent

Name	Qty	Part No.
Graphitized Carbon Black	25 g	186004835
Trifunctionally-bonded C_{18} Silica	100 g	WAT035672

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July 2016 720003048EN AW-SIG