



A New Technology, Energized Dispersive Extraction, for One Step  
Extraction and Cleanup of Complex Matrices

# Food: Complex and Wide Variety of Applications



- **Pesticides**
- Fat
- Contaminants
- Nutrients
- Packaging





# Extraction of Pesticides from Food

## QuEChERS

**Quick Easy Cheap Effective Rugged and Safe**

Multi-matrix Multi-residue Method

- Salt partitioning extraction
- dSPE Cleanup

## EDGE

- Multi-matrix Multi-residue Method
- Pressurized Fluid Extraction with In-cell Cleanup

# Extraction of Pesticides from Food Matrices

- **QuEChERS** is an accepted extraction and matrix clean up procedure for multi-residue analytes in a variety of different matrices
  - Manual
  - Slow
  - Limited
  - Cost per test \$3-\$5
- **EDGE** is a rapid, efficient, and simple alternative method to QuEChERS
  - Automated
  - 5 min run time
  - Versatile
  - Low cost



# Extraction of Pesticides: QuEChERS Method

## AOAC 2007.01 Method Procedure

### Sample extraction

1. Transfer 15 g homogenized sample to 50 mL centrifuge tube
2. Add 15 mL 1% acetic acid in acetonitrile + 1.5 g NaAc + 6 g MgSO<sub>4</sub>
3. Shake vigorously 1 min
4. Centrifuge > 1500 U/min for 1 min

### Sample Cleanup

1. Transfer 1-8 mL of acetonitrile layer to tube with 150 mg MgSO<sub>4</sub> + 50 mg PSA per mL extract
2. Shake vigorously 30 sec
3. Centrifuge > 1500 U/min for 1 min
4. Transfer supernatant to a vial for concurrent analysis

**\*Entire process takes around 20 min  
of constant manual work**

# QuEChERS Kits and Sorbents Available

- Sigma Aldrich
- Thermo Fischer Scientific
- Agilent
- Waters
- Restek
- Phenomenex
- Silicycle
- United Chemical
- Thomas Scientific
- GL Sciences
- Environmental Express
- Etc.



**DisQUE™**  
Dispersive Sample Preparation



# QuEChERS Salts and Sorbents

- Sodium Chloride: Reduces polar interferences
- Sodium Acetate: Protects base sensitive analytes
- Magnesium Sulfate: Removes water
- Primary Secondary Amine (PSA): Removes sugars and fatty acid
- C<sub>18</sub> : Removes lipids
- Carbon Black: Removes pigments

Any combination compatible  
with the EDGE!



# Extraction of Pesticides: EDGE Method

## Sample extraction and sample clean up together

1. Add dSPE sorbent and transfer homogenized food sample to Q-Cup
2. Place Q-Cup in the EDGE
3. Run the 5 min EDGE method
4. Transfer extract to a vial for concurrent analysis

Entire process takes only 5 min per sample and is automated

# Pesticide Method

## Wet Samples

**Edit Method - pesticide**

Settings	Solvent	Acetonitrile >
Cycles	Top Volume (mL)	<input type="text" value="20.0"/>
Parameters	Bottom Volume (mL)	<input type="text" value="10.0"/>
Wash	Rinse Solvent	Acetonitrile >
	Volume (mL)	<input type="text" value="0.0"/>
	Temperature (°C)	<input type="text" value="100"/>
	Hold Time	2:00 >

2:48 PM

**Edit Method - pesticide**

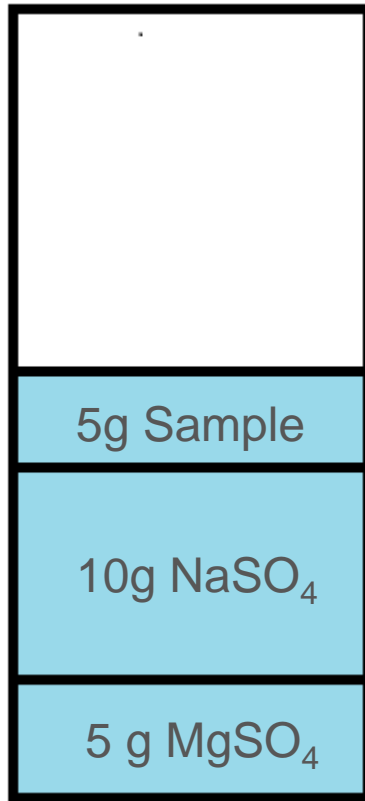
Settings	Wash	Solvent	Volume (mL)
Cycles	1	Water >	30.0
Parameters	2	Acetonitrile >	10.0
Wash			

2:48 PM

## Dry Samples

Solvent: ACN, Temp: 100 °C, Hold: 2:00 min, Top: 20 mL, **Bottom: 5 mL, Rinse: 5 mL**  
Wash 1: 10mL ACN, Wash 2: 20mL ACN

# Wet Sample Tips



When using salts for drying a 30 mL water wash is mandatory!

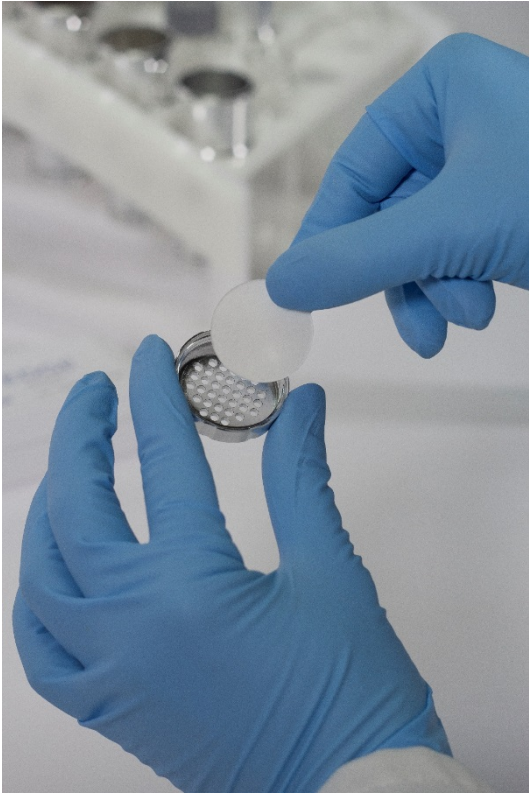
Wash 1: 30mL H<sub>2</sub>O

Wash 2: 15mL Extract Solvent

↑  
Sample Size

5g Sample = 10g NaSO<sub>4</sub>  
10g Sample = 15g NaSO<sub>4</sub>  
15g Sample = 20g NaSO<sub>4</sub>

# Assemble Q-Cup





# Add Sorbents and Sample





# Prepared Q-Cup



No Packing or Mixing!

# Prepared Q-Cup Tips



↑  
Water content  
In sample

↑  
Amount of  
salt layer

Compatible with  
QuEChERS Kits

Clean layers

Protect the Q-Disc with  
enough salt and sorbent

## Q-Disc Configuration

- M2 textured side up (do not heat above 100 °C)
- C9 Support

# Q-Disc Tips

## Porosity



LC Analysis



Membrane

- M2

GC Analysis



Cellulose

- C1
- C3
- C4
- C7
- C9

H<sub>2</sub>O present



# Q-Disc Tips

## Sample Matrix

Dry Foods

All C  
M2

Fine Powders

$\geq C4$   
M2

Wet Foods

M2

1) Porosity

LC or GC analysis

2) Sample Matrix

Dry or Wet, Fine Powder

# UPLC MS/MS Method

- Waters Acquity H Class, Xevo TQD
- Waters Acquity UPLC BEH C18 1.7  $\mu\text{m}$  2.1 x 50 mm column
- 10  $\mu\text{l}$  injection
- A: Water with 10 mM Ammonium Acetate, B: Methanol with 10 mM Ammonium Acetate
- Gradient

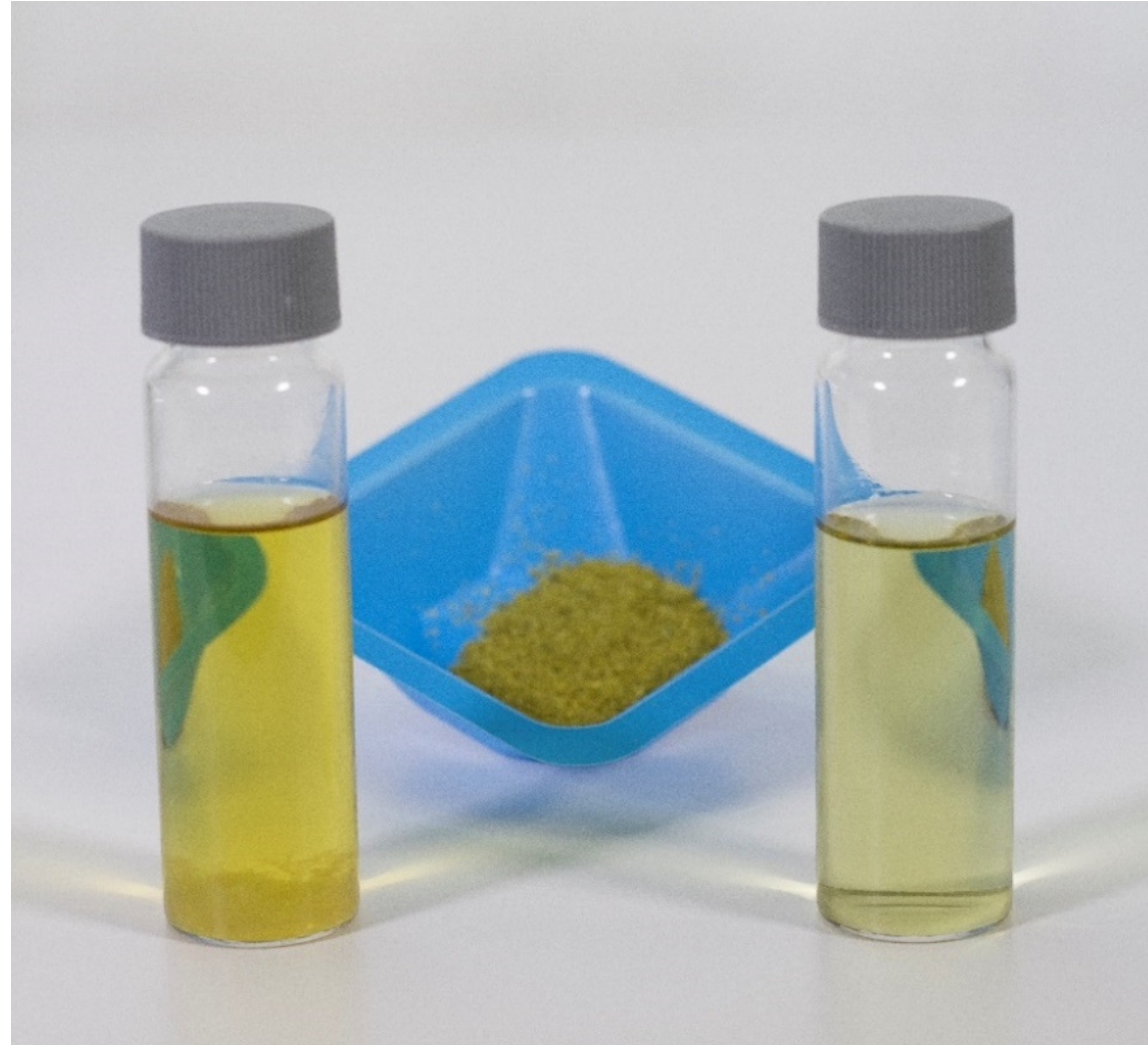
Time (min)	Flow (mL/min)	%A	%B
Initial	0.45	95	5
4	0.45	5	95
5.5	0.45	5	95
5.75	0.45	95	5
6	0.45	95	5

- Quantitation was based on a 6 point multi-level calibration curve using Multiple Reactions Monitoring

# Strawberry Extracts



# Hops Extracts



# %Recovery Data of Spiked Rice and Strawberries

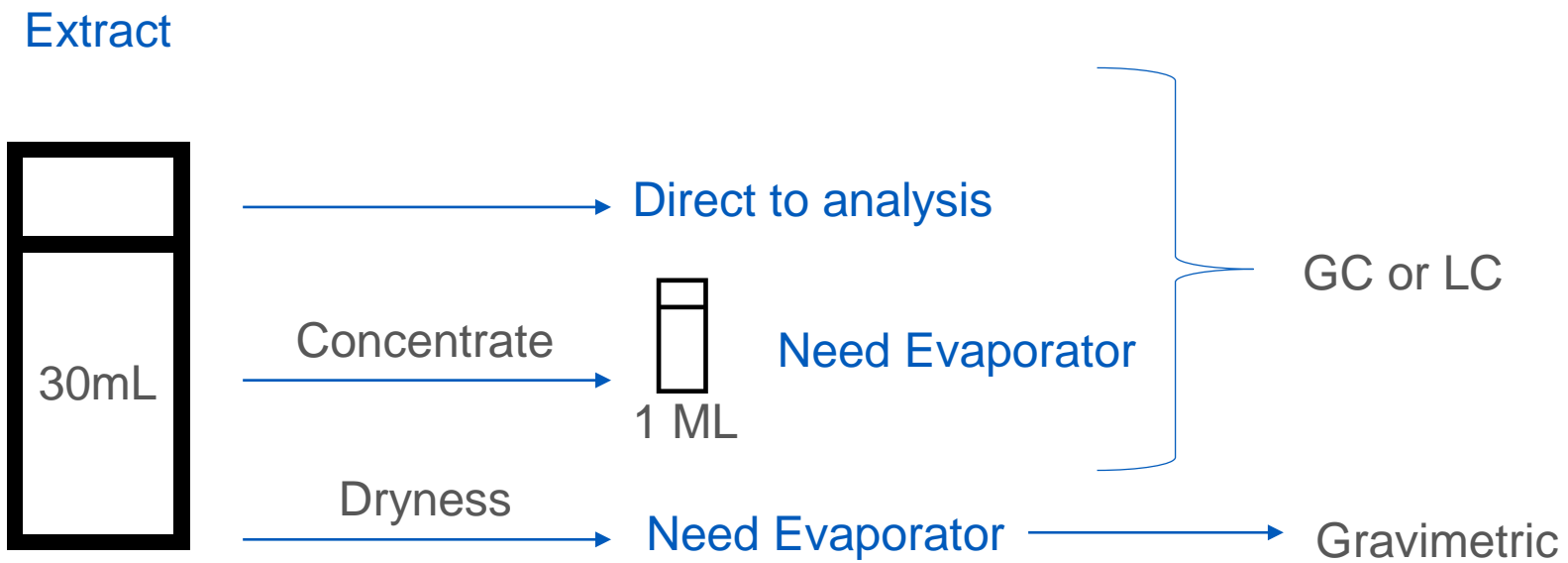
Pesticide	Rice	Strawberry
Tokuthion	87	93
Guthion	90	90
Dichlorvos	88	120
Methyl parathion	95	107
Dursban	89	100
Ronnel	90	102
Disulfoton	92	92
Mocap	94	109



# %Recovery Data of Spiked Avocado and Hops

Pesticide	Avocado	Hops
Tokuthion	86	102
Guthion	85	102
Dichlorvos	116	98
Methyl parathion	107	98
Dursban	93	107
Ronnel	97	105
Disulfoton	89	101
Mocap	93	102

# Post Extraction Tips





# Automated QuEChERS Systems



Gerstel MPS3



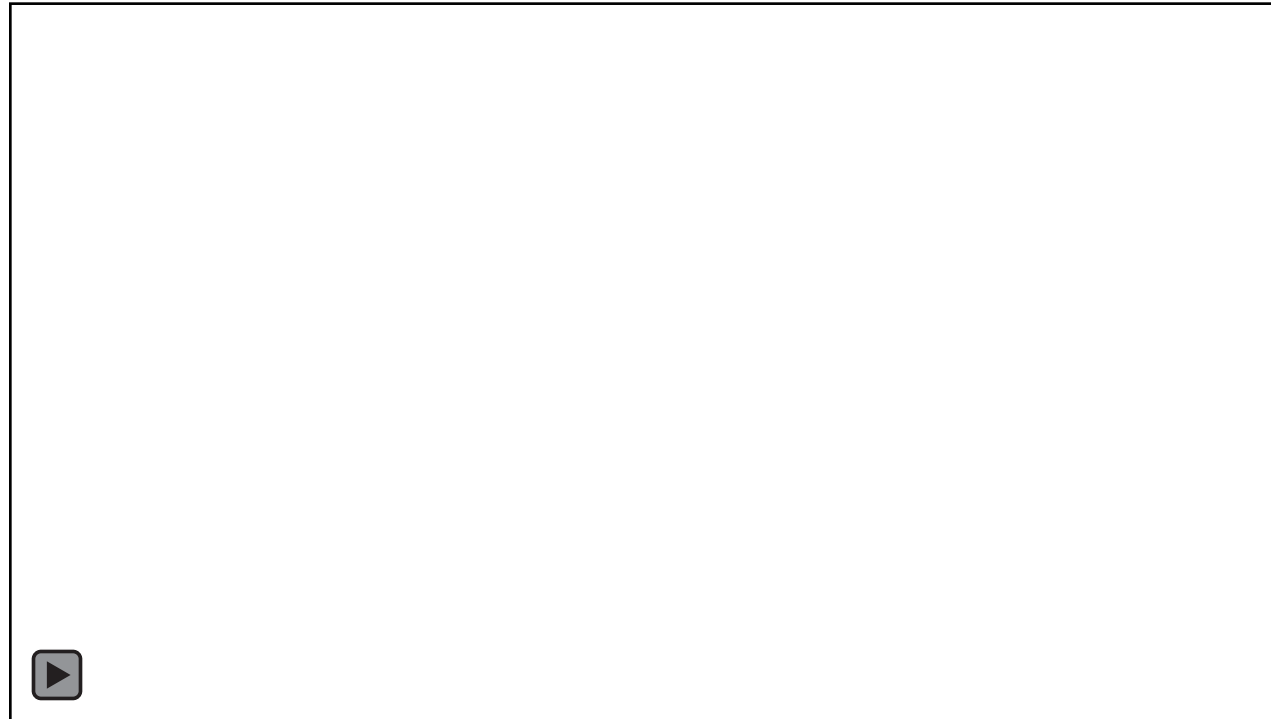
Teledyne Tekmar AutoMate-Q40

44”H x 64”W x 27”D

- J2 Scientific PrepLink
- Tecan Freedom EVO

# Automated QuEChERS Video

<https://www.youtube.com/watch?v=82OpFGSgBIQ>



# Automated QuEChERS Systems



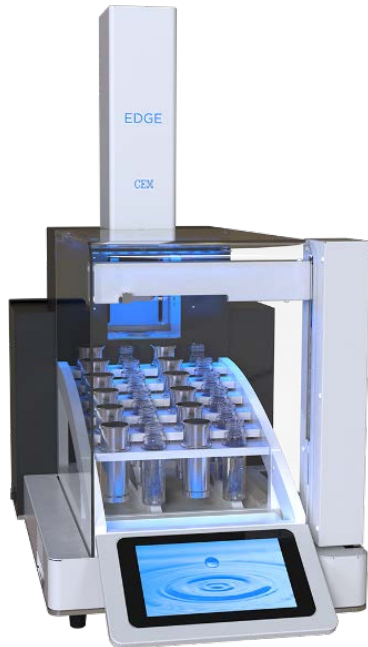
Teledyne Tekmar AutoMate-Q40

The AutoMate-Q40 system automates the following sample prep functions

- Liquid dispensing/pipetting
- Vortex mixing
- Vial shaking
- Opening/closing sample vials
- Addition of solid reagents (ex. salts, buffers)
- Identifying liquid levels
- Decanting
- Centrifugation
- Matrix spiking
- dSPE cleanup

# EDGE Versus Automated QuEChERS

- Automated systems robotically automate the QuEChERS process
- EDGE is a new technology and alternative to QuEChERS



Rapid, Simple, and Efficient!