



Breaking Bad: Change lab habits with more ions



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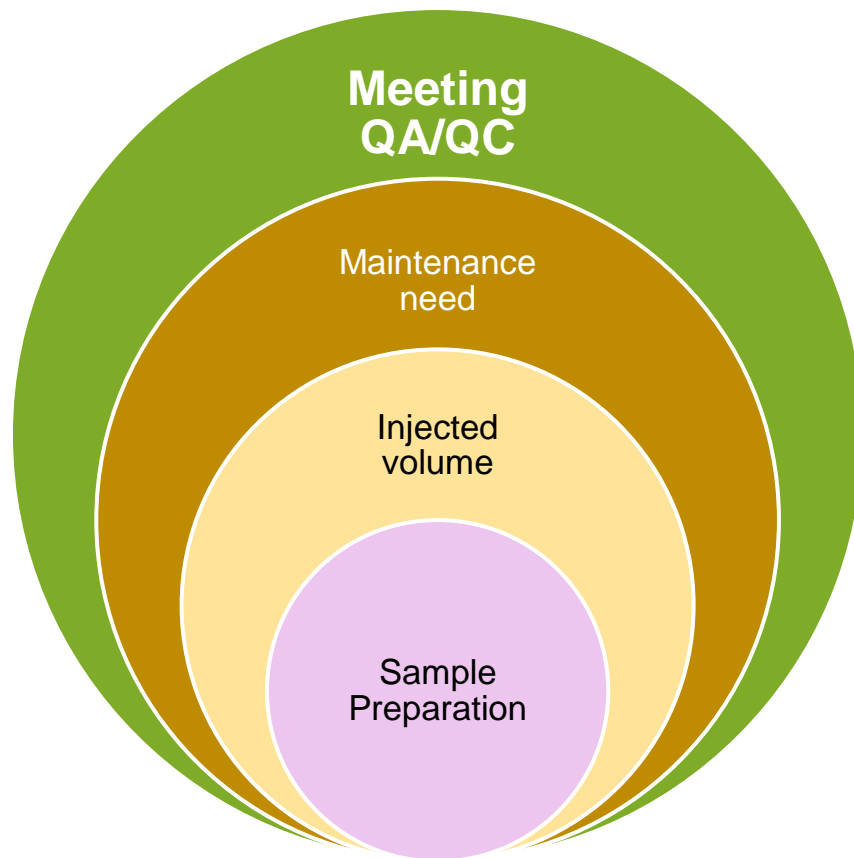


Old Habits

New Approach

Maintained

Maintained



2 μ l

0.5 μ l



15 g

2 g

Optimizing your experience: Pesticide Residues in Foods

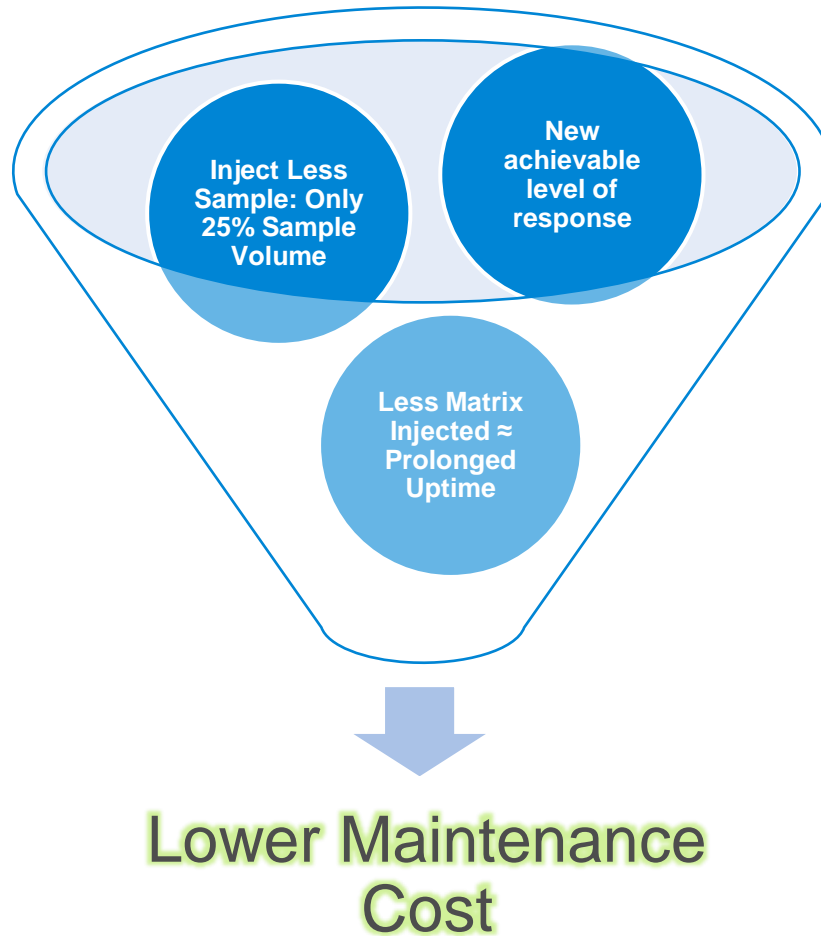
Highlights

- Advantages of the High Efficiency Source (HES) of the 7010
- GC/MS/MS QuEChERS sample: **15** g versus **2** g
- Injection volume: **2.0** μL versus **0.5** μL
- Maintain and reach lower LOQs



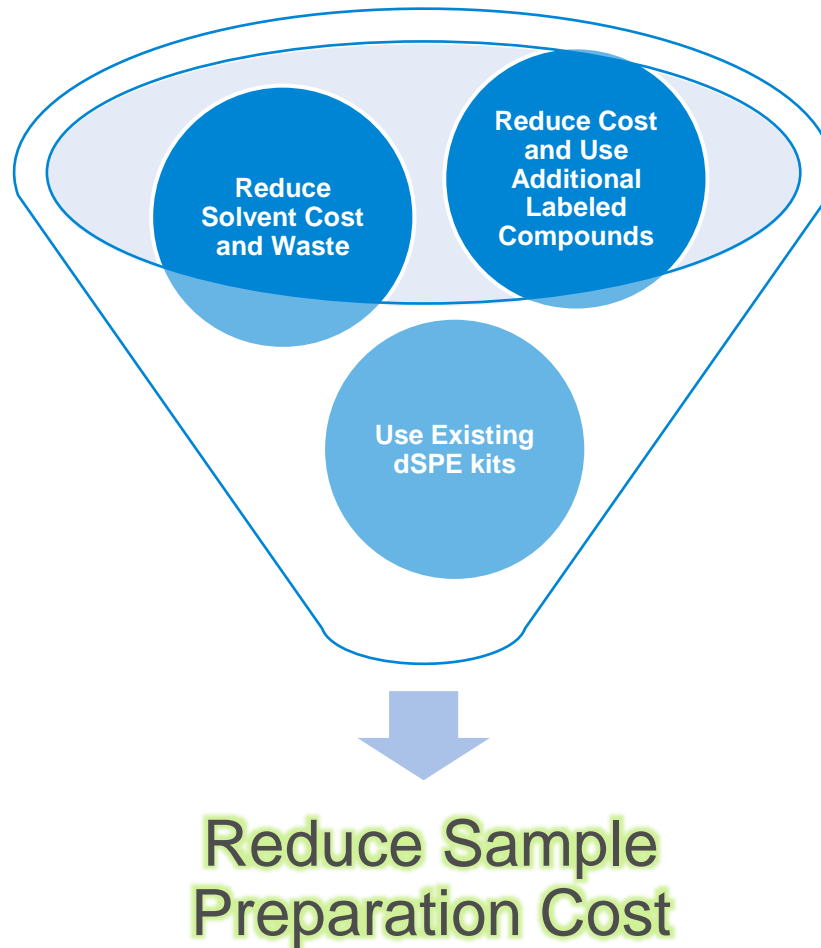
What if?

GCMS 7010 High Efficiency Source (HES)



What if?

Miniaturize Sample Preparation



The High Efficiency Source (HES) of the Agilent 7010 maximizes ion formation



Revolutionary, true increase in sensitivity – more ions created

Achievable limits that exceed today's regulatory requirements

Increased sensitivity gives you options - use of the HES allows us to modify our sample prep and sample introduction

Analytical Approach: Miniaturization of QuEChERS

Sample Preparation:

Extraction/Partitioning (AOAC)

2 g of sample, 2 ceramic homogenizers

2 mL ACN (1% HAc), vortex

1 g of Agilent Bond Elut AOAC salts

shake, centrifuge

Dispersive SPE

Transfer 1 mL of extract to 2 mL Agilent Bond Elut dSPE
(G/FV or P/FV)

vortex, centrifuge

Transfer 250 μ L for analysis

Maintain Sample/Solvent/Salt Ratio



LOQs (ng/g) using 0.5 μ L injection: 10-fold lower than default MRL

	Carrot	Tomato	Celery
Bifenthrin	1	1	1
Bupirimate	1	1	1
Captan	5	5	1
Chlorothalonil	1	1	1
Chlorpropham	1	1	1
Clomazone	1	1	1
Cypermethrin	1	1	1
Cyprodinil	1	5	1
DDE-p,p'	1	1	1
Diazinon Results	1	1	1
Dicloran	1	1	1
Dieldrin	1	1	1
Difenoconazole I	1	1	1
Diphenylamine	1	1	1
Endosulfan I	5	1	1
Endosulfan II	1	1	1
Endosulfan Sulfate	1	1	1
Endrin	1	1	1
Etridiazole	1	1	5
Fenpropathrin	1	1	1
Fenvalerate	1	1	1
Fludioxonil	1	1	1
Folpet	1	1	1
Fuberidazole	5	5	1
Iprodione	1	1	1

LOQ is 1 ng/g for
86 - 90% of 126 pesticides

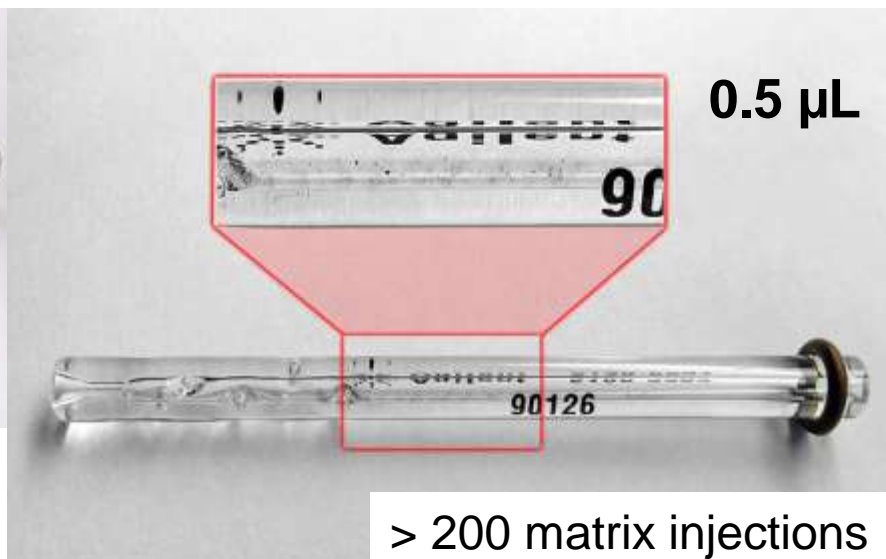
Recovery-based LOQs
(over 35 injections)



(2g sample, spiked blank matrix)

Analytical Approach: 7010 Triple Quadrupole GC/MS

- Columns: Agilent J&W Ultra Inert HP-5ms columns: 5 m x 0.25 mm and 15 m x 0.25 mm x 0.25 μm
- Agilent Ultra Inert dimpled liners, UltiMetal plus Flexible Metal ferrules at the PUU, **column backflushing**
- **0.5 μL** versus **2 μL** injection





Concerns About Miniaturizing QuEChERS



- Representative Sampling
 - Collection from different sources
 - Isolated positives
- Missing hits
- Sample amount: 10-15 g required
- Ease of use, handling sample size
- Available equipment

Prepare Less Sample in Conjunction with Injecting Less

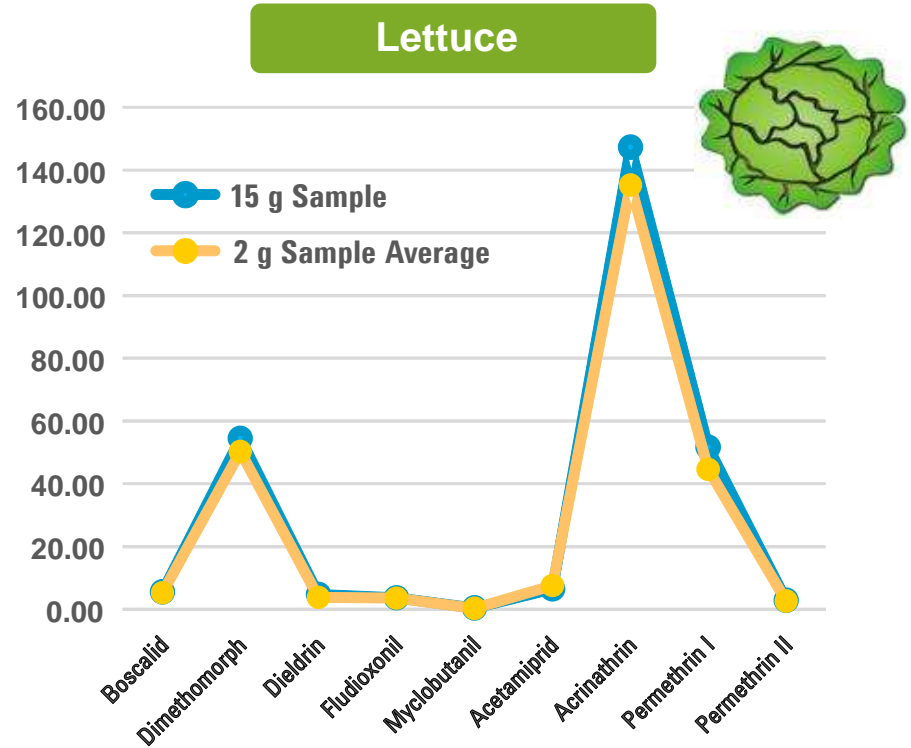
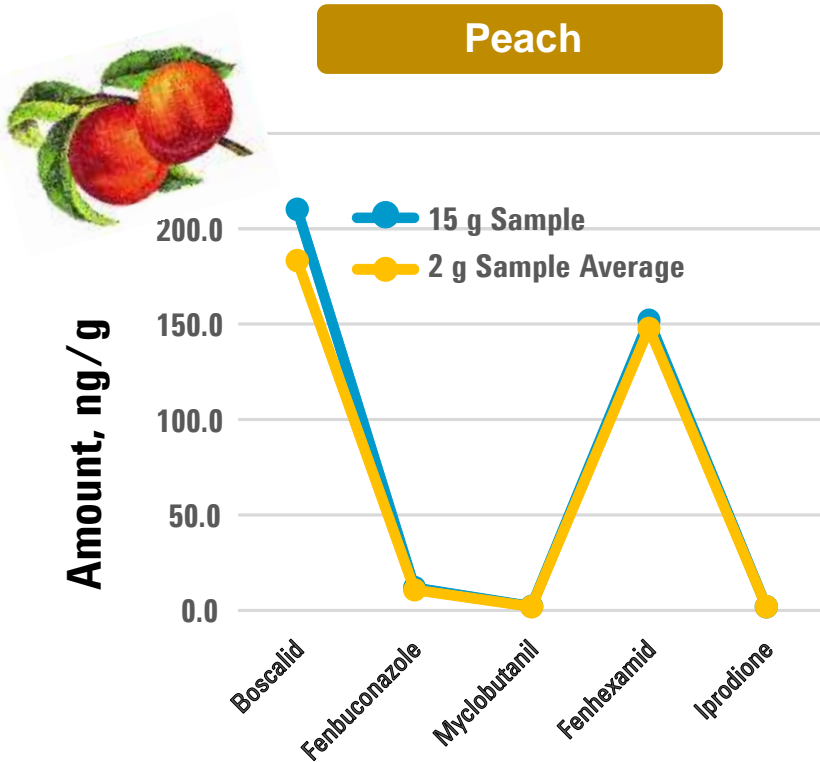


Only 2 g of sample, with cryomilling, was used for miniaturized QuEChERS extraction, while still delivering reliable sampling

- an 87% weight reduction compared to the AOAC



Similar Results with 15 g Homogenized and 2 g Cryomilled Matrix Samples



Incurred samples were pre-tested by a monitoring agency

Same pesticide residues as determined by the independent laboratory

Comparison of Pesticide Residue Analysis from 15 g and 2 g Homogenized and 2 g Cryomilled

Incurred Residues in Tomato, ng/g						
	2g Cryo	% RSD (n=6)	2g homogenized	% RSD (n=3)	15g homogenized	% RSD (n=2)
Chlorothalonil	7.32	20.3	8.49	7.0	6.56	2.2
Chlorpyrifos	0.32	17.9	0.39	13.1	0.35	16.6
Myclobutanil	0.13	11.6	0.15	7.3	0.10	12.4
Buprofezin (Z-isomer)	45.22	3.6	54.82	10.6	41.51	1.2
Tebuconazole	0.36	12.9	0.32	7.2	0.25	0.1
Pyriproxyfen	12.63	6.1	14.05	2.7	10.50	2.5

Incurred Residues in cucumber, ng/g						
	2g Cryo	% RSD (n=6)	2g homogenized	% RSD (n=3)	15g homogenized	% RSD (n=2)
Chlorpropham	0.544	12	0.528	2.6	0.674	3.8
Chlorothalonil	44.4	7.6	51.4	7.5	51.5	7.7
Metalaxyl	35.0	7.7	37.6	9.5	37.0	5.3
Dieldrin	1.86	7.4	1.92	12	1.83	0.4
Boscalid	14.4	6.1	15.3	7.7	15.1	7.6
Pyraclostrobin	1.73	7.6	1.89	15	1.86	0.3
Dimethomorph	7.65	7.5	7.42	3.1	7.18	10.2

Incurred Residues in Strawberry, ng/g						
	2g Cryo	% RSD (n=6)	2g homogenized	% RSD (n=3)	15g homogenized	% RSD (n=2)
Chlorpropham	0.28	12.1	0.14	22.5	0.18	0.6
Cyprodinil	1.15	5.1	1.16	3.0	1.10	3.2
Fludioxonil	1.90	9.9	2.13	14.8	1.79	4.2
Myclobutanil	3.80	6.6	3.52	5.4	3.42	0.5
Bifenthrin	3.87	6.3	3.67	4.4	3.54	1.5
Boscalid Results	2.28	4.6	2.38	7.53	2.26	2.0
Pyraclostrobin	1.04	11.8	0.92	11.7	0.97	5.1

Reduce Sample Prep Costs by Over 40%

Cost Breakdown and Cost Savings for Sample Preparation with QuEChERS and Mini-QuEChERS Technique

Sample Preparation Cost/Sample	Centrifuge Tube	ACN	Salts	Internal Std: Captan-d6, Folpet-d4	dSPE Gen F&V or Universal	Total Cost/Sample	Cost Savings
QuEChERS	\$0.43	\$1.50	\$2.96	\$0.30	\$1.32/\$1.96	\$6.51/\$7.15	-
Mini-QuEChERS	\$0.42	\$0.20	\$0.80	\$0.04	\$1.32/\$1.96	\$2.78/\$3.42	43%/48%



What have We Learned

- Miniaturized QuEChERS **delivers: easier sample handling, reduced cost** of labeled compounds, salts, solvent, waste, and used existing dSPE
- 7010 Triple Quadrupole GC/MS High Efficiency Source has several advantages: new achievable level of response, **inject less sample**, only 25% of the **extract** volume, less matrix injected offers prolonged uptime, increased performance
- Initial data presented suggest that a 2 gram sample with mini-QuEChERS was capable of producing similar results as found with a 15 gram sample amount used in AOAC QuEChERS. **No sampling error** was observed due to smaller sample size.
- Initial comparison of 2 gram sample homogenized versus homogenized and cryomilled produced similar results suggesting that **cryomilling is not required for a 2 gram sample** size employed in mini-QuEChERS, which offers laboratories an alternative approach to multi-residue analysis

Thank you!

