Analytical Challenges for Food Safety in the Global Competitive Market



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About OMIC

>Overseas Merchandise Inspection Company

A Japanese Company

Established in 1954

Core Businesses:

- Inspection, testing, and appraisal of the quality, quantity, and weight of cargo
- Food Safety Inspection
- Price evaluation for imported and exported products
- Certification of Organic Products





Laboratory Network

supports Overseas Merchandise Inspection Company



Testing the limits

OMIC USA laboratory



Located in Portland, Oregon ca. 30 employees ca. 10,500 sample per year





OMIC USA 's purpose is...

Exporting

- Food and Feed Grains, Beans and Pellets
- Fruits and Vegetable
- •Tea and Coffee
- Food supplements

Importing

FDA – DWPE Program



Analytical Services

- FDA Nutrition Label
- Pesticides Residue
- Pathogen Testing
- Mycotoxins
- Food Additives
- ・GMO Testing

Soil Pesticides Testing

- rotating crops
- Drift problem

GLP Projects

FSMA (future work/accreditation)





Export Testing Service

- Through increase interaction with government offices in foreign countries (MHLW Japan and KFDA in Korea) we are able to assist clients to meet import requirements
- Our chemists received necessary analytical training in foreign government laboratory for their approved methods



Political Map of the World



R02373 (P00350: 4.95





Food Safety Testing

- Pesticides
- Natural Toxicants mycotoxins
- Additives colors
- Packing migration
- Environmental contaminants PCB's, PAH's, metals
- Authenticity and traceability
- Deliberate adulteration



Food Safety Analytical Challenges Methodology

Complexities

- ✓ multi- residue / multi elements
- ✓ matrix complexity (dry, high fat, spices)
- ✓ Iower Maximum Residue Limits (MRL)
- \checkmark not uniform MRL => more method validation

Challenges

 ✓ development of analytical method for analysis of multi residues pesticides in complex matrix
✓ need expensive equipment, MSMS, HRMS, TOF





Food Safety Analytical Challenges Method Validation

Complexities

- \checkmark suitable for given range
- \checkmark sensitivity: LOD = 3 x S/N, LOQ = 3 x LOD
- ✓ specificity/selectivity
- ✓ repeatability/precision
- ✓ recovery (usually 70 120 %)
- ✓ matrix standard vs solvent made standard

Challenges

✓ validated method for various parameters





Food Safety Analytical Challenges Sampling

Complexities

- ✓ homogeneous sample
- sample taking for testing must represent the entire lot
- ✓ multi portions sampling for different tests
 - GMO, mycotoxins, micro testing, pesticides
- \checkmark statistical sampling determination

Challenges

✓ Representative sample for the complete lot



Food Safety Analytical Challenges Sample extraction

Complexities

- \checkmark chemical nature of analytes
- \checkmark chemical type of matrix
- ✓ co-extractants problem
- \checkmark adsorption of analyte on the matrix

Challenges

✓ selection of the right solvent✓ optimize the extraction condition





Food Safety Analytical Challenges Clean up

Complexities

- ✓ number of compounds
- ✓ matrix interference
- \checkmark adsorption of the analytes onto the SPE powder

Challenges

✓ selection of the appropriate technique
✓ adsorbant powder selection
✓ solvent elution



Food Safety Analytical Challenges Sample Concentration

Complexities

- ✓ type of analyte
- \checkmark selection of technique (roto-evaporator, N₂-evaporator)

Challenges ✓ LOD /LOQ meet the MRL value ✓acceptable % recovery





Food Safety Analytical Challenges Analytical Instrumentation

Complexities

- ✓ instrument noise level (LOD)
- ✓ interference
- ✓ false negative / false positive
- \checkmark instrument cost / operation cost
- ✓ expiration date for reference standards

Challenges

✓ confirmation method (MSMS, TOF, HRMS)
✓ availability of reference standard (second source)
✓ data result interpretation





Food Safety Analytical Challenges Analyst

Complexities

- \checkmark training and experience
- \checkmark skill, attitude, problem solving worker
- \checkmark judgment, making the right decision

Challenges

✓ long-term employment employee





Testing the limits

We did all the testing



Cartoon reference: http://www.hospitalityguild.com/cartoon2.htm







Ref: David Ropeik, How Risky Is It Really? Why Our Fears Don't Always Match the Facts, McGraw Hill, 2010





Building consumer trust



From: Fear



To: Confidence

Cartoon www.panicbuster.com/grfx/phobias/jpg Photo http://www.thedailygreen.com/environmental-news/latest/organic-food-tips-47-040801





Challenges – Screening for 750 Pesticides

Complexities

- \checkmark increase number
- ✓ difficult matrices
- ✓ multi residue method or single analysis
- ✓ lower sensitivity (as low as 0.3 ppb)
- ✓ diversity of pesticide structure (organoclorine, organophosphates, carbamates, pyrethrum, etc.
- \checkmark no history of pesticide usage in foreign country







Challenges – Screening for 750 Pesticides



Challenges

- ✓ increased monitoring program (more samples)
- ✓ client demands low cost
- ✓ faster turnaorund time (7 w. days or less)
- \checkmark quick extraction





Testing the limits

OMIC USA trends in Multi pesticide screen method





Multi Residue Method Strategies





MASE – Microwave–Assisted Solvent Extraction

A process of heating solid sample and solvent in a sealed (closed) vessel with microwave energy and temperature controlled conditions.

PFL – Pressurized Fluid Extraction

A process similar to Soxhlet extraction except that the solvents are used near their supercritical region where they have high extraction properties.





SBSE – Stir Bar Sorptive Extraction

Extraction is performed using a special glass coated magnetic stir bar which is coated with polydimethylsiloxane (PDMS).

SPE – Solid Phase Extraction

Well established procedure used for isolating and concentrating analytes at low detection levels because it eliminates the interferences that contribute to signal suppression.





LLE – Liquid–Liquid Extraction

A mass transfer operation in which a liquid solution (the feed) is contacted with an immiscible or nearly immiscible liquid (solvent) that exhibits selectivity toward one or more of the components in the feed.

SPME – Solid Phase Micro Extraction

A solvent-less extraction procedure that involves exposure of a probe (coated fused silica fiber) to a gaseous or liquid sample or the headspace above a liquid or solid sample





SFE – Supercritical Fluid Extraction

A supercritical carbon dioxide is used as solvent. This solvent has penetration and transport properties similar to a gas but acts as a liquid when dissolving analytes from matrix.

QuEChERS-Quick Easy Cheap Effective Rugged Safe

An extraction method using dispersive SPE clean -up.

M. Anastassiades, S.J. Lehotay, D. Stajnbaher and F.J. Schenck, J AOAC Int 86 (2003) 412.







SBSE – Stir Bar Sorptive Extraction

- A PolyDimethylSiloxane (PDMS) coated stir bar is placed in a liquid (water sample or sample extract) and sir for several minutes.
- The analytes of interest are extracted from matrix into the PDMS phase
- The analytes are thermally desorbed from the stir bar in a GC Thermo Desorption Unit (TDU) made by GERSTEL and transferred to a GC capillary column.
- Gerstel's Twister SBSE is an effective extraction and rapid extraction technique.





SBSE – Stir Bar Sorptive Extraction



SBSE – Stir Bar Sorptive Instrument Injection

GERSTEL Thermal Desorption Unit (TDU) with MPS-2 robot



Auto sampler rack for MPS-2-TDU

98 positions

Twisters are put in a clean empty glass liner and capped with special tube head



MPS-2-TDU on top of GC





QuEChERS

Anastassiades, S.J. Lehotay, D. Stajnbaher and F.J. Schenck, J AOAC Int. 86 (2003) 412.

Extraction/Partition

- Sample + ACN:H₂O
- Citrate (pH=6.4), or Acetate (pH=4.8)
- Dispersive clean-up of ACN extract PSA/C₁₈ or GCB
- Concentration/ solvent exchange (optional)









Pesticide residue analysis: ~740 compounds QuEChERS ~520

QuEChERS

(70 % of all compounds)





GC – Detection Challenges

- > GC work common problem
 - Peak tailing or analyte lost due to undesired interaction with active sites in the inlet column.
 - Higher Detection limit for these compounds and difficult to identify and calculate.
- > Analyte Protectants provide an effective solution to the problem.
 - They are added to extracts and matrix free standards to enhance the chromatography effect for analytes in a very dirty GC system.







Slide adapted from Steven Lehotay, USDA-ARS





LC-MSMS Detection Challanges

Ion suppression on LC/MS/MS



- Commodity
- Impacts accuracy:
 - S/N and LOQ for screening
 - Quantitation for positive samples



Strawberry Profile LC-MSMS Chromatogram







Cyprodinyl Chromatogram



Insecticide Trade name: Vangard WP



Matrix std (berry juice) 0.02 ppm

Matrix blank (berry juice)

Sample, 10 x dil. (strawberry)





ANALYTICAL LABORATORY

Fenhexamid Chromatogram

ECIALTY CROPS COUNCI



Fungicide Trade name: Elevate 50 WDG



Fludioxonil Chromatogram



Fungicide Trade name: Maxim, Switch







Reporting Pesticides

Analyte	Result	LOQ	Unit	EPA CFR #	Tolerance USA	Tolerance Canada	Tolerance Japan
Abamectin	0.01	0.01	ppm	180.476	0.02	0.02	0.02
Bifenazate	0.68	0.05	ppm	180.572	1.5	1.5	5
Captan**	10.2	0.01	ppm	180.103	20	5	20
Cyprodinyl	0.23	0.01	ppm	180.532	5	3.5	1
Fenhexamid	0.16	0.01	ppm	180.553	3	3	10
Fludioxonil	0.22	0.01	ppm	180.516	2	2	5
Myclobutanil	0.07	0.01	ppm	180.443	0.5	0.5	1





Strawberry Detected Pesticides (ppm)



Strawberry Detected Heavy Metals (ppb)







177

7 w.days

23

Export Wheat Profile

- Total Number Compounds
- Number of Methods per sample
- TurnAround Time (TAT)
- Equipment use:
 - Gas Chromatograph MS and MSMS detectors (5)
 - Ultra Precision Liquid Chromatograph MSMS (5)



Wheat Profile



- Multi Pesticide Screen GC / LC (122)
- Phenoxy Herbicides (21)
- Individual Test (13)
- Multi Pesticide Screen SU (8)
- Premier LC MSMS (3)
- Glyphosate / Glufosinate (2)
- Mycotoxins (2)
- OrganoTin (2)
 - Quaternary Ammine (2)
- Volatiles (2)





Export Testing Service Wheat Profile - Quality Assurance

- Internal Quality Data (minimum 20 spike recoveries) submitted to MAFF on annually basis
- Annually Internal and External audit
- Re-validation method data
 - Ten replicates at MRL level and LOQ level



Wheat Detected Pesticides (ppm)







Food Safety Requires . . .

THE POWER OF COLLABORATION...







Food Safety Testing Requires . . .

One accreditation standard

- ✓ ISO 17025
- ✓ KFDA
- ✓ NELAC / ORELAP
- ✓ MHLW
- ✓ FSMA

Uniform MRLUniform analytical methods





Thank you for your attention



