

Solution of multi-residue analysis in food safety

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Presentation Overview

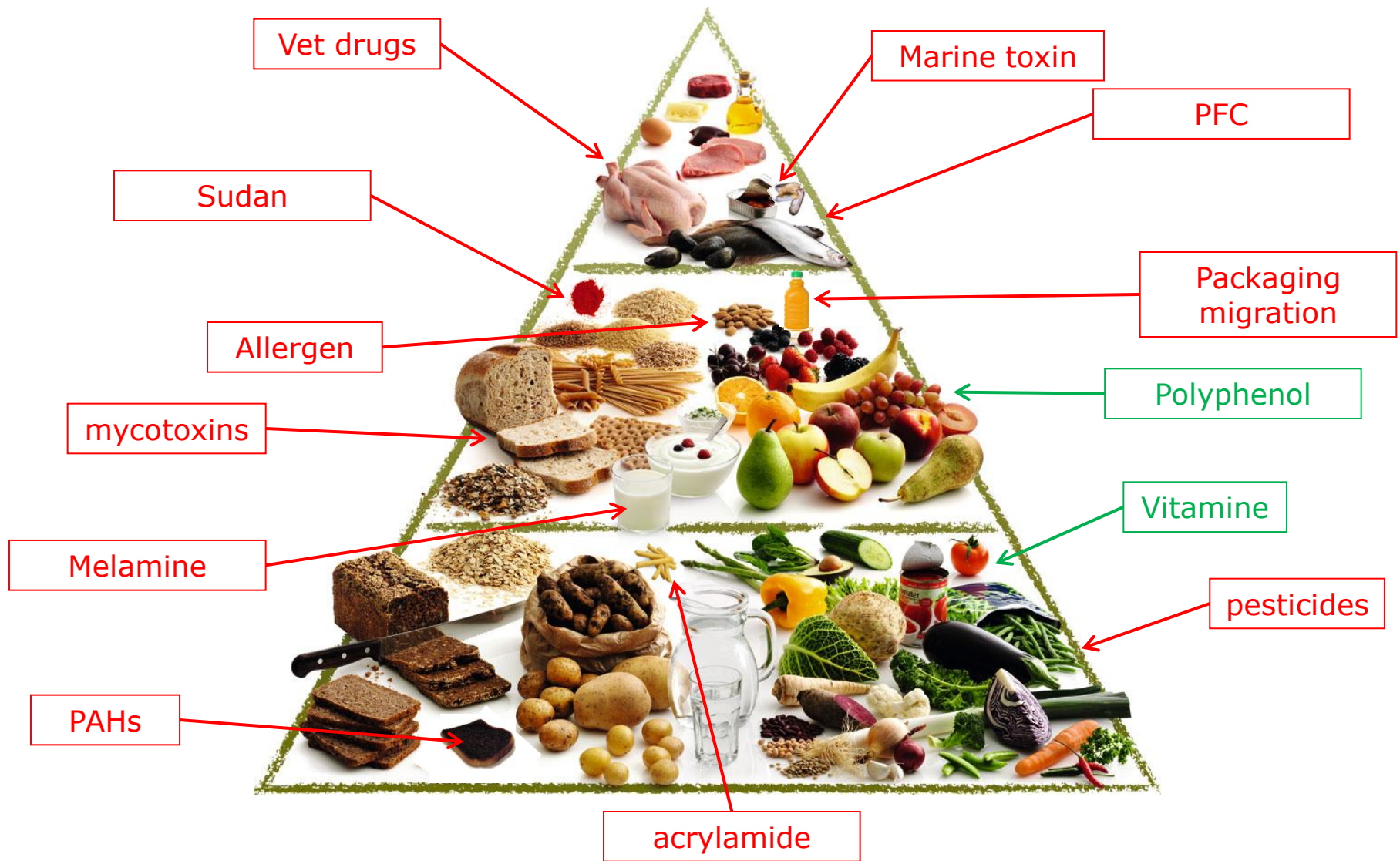


- Overview of Multi-residue analysis
 - Challenges in food safety analysis
 - customer's requirement
 - What should we focus on
- Experimental Information
 - Sample Preparation
 - Instruments conditions
- Results and Conclusions
 - Method performance
 - Advantage of the Multi-residue solutions



The view of food safety

—Multitude of Compounds & Variety of Food Types



What are the challenges?

Variety of methods

- mycotoxins
- Pesticides
- Veterinary drugs
- Food additives
- Contaminants
- • • • •

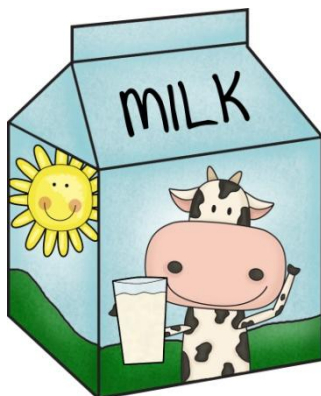
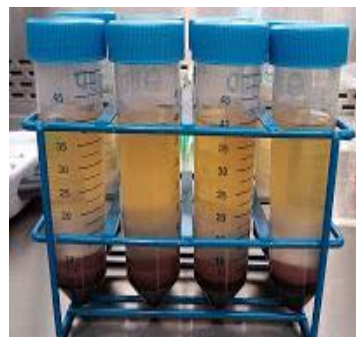
More than 6,000 compounds need to be detected

Part of the Vet Drugs Methods

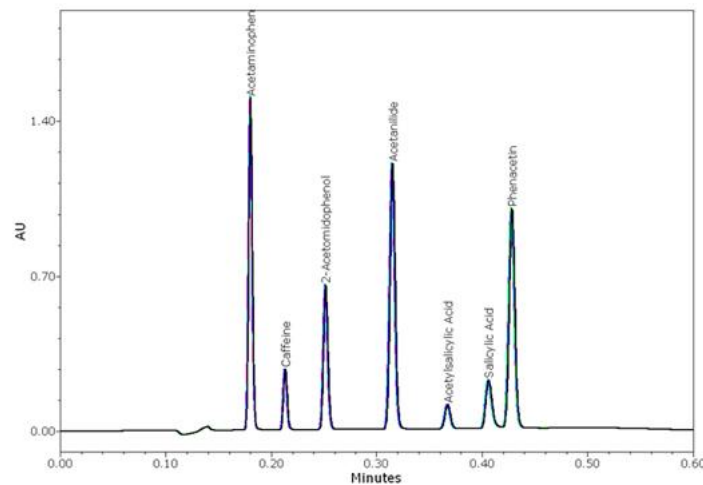
Method	Analytes	Sample Matrix
GB/T22286	β-Adrenergic agonists	Muscle
GB/T21315	β-Lactam	Kidney and muscle
GB/T22338	Chloramphenicol	Muscle
GB/T20762	Macrolide	Liver, kidney, and muscle
GB/T21316	Sulfonamides	Liver and muscle
GB/T20366	Fluoroquinolone	Liver and muscle

What 's the customer focus on?

How can I turn this?



Into this?



Three major classes compounds in food safety

Pesticides

Organophosphates

Chlorpyrifos, Diazinon

Carbamates

Aldicarb, Carbaryl

Organochlorine

DDT, Endosulfan

Pyrethroids (菊酯)

Cyfluthrin, Pyrethrin

Sfonylureas ?

Rimsulfuron 玉密磺隆,

Phenoxyacid Herbicides

2,4-D

Triazines

Atrazine

Ureas

Diuron

Acetanilides

Metolachlor, alachlor

Neonicotinoids

Imidacloprid

Imidazolinones

Imazapyr

Veterinary Drugs

Tetracyclines

Oxytetracycline

Fluoroquinolones

Enrofloxacin

Sulfonamides

Sulfamerazine

Macrolides

Erythromycin

Beta-Lactams

Amoxicillin

Amphenicols

Chloramphenicol

Steroids

Dexamethasone

Beta-Adrenergics

Albuterol

Aminoglycosides

Streptomycin

Mycotoxins

Aflatoxins

Aflatoxin B

Fumonisin

Fumonisin A

Trichothecenes

HT-2 Toxin,
Deoxynivalenol (DON)

Ochratoxins

Ochratoxin A

Estrogenic Metabolites

Zearalenone

Phenolic

Citrinin

Experimental Information Multi-Residue Method

Sample ID	File Name	MS File	Inlet File	Bottle	Inlet Volume	Sample Type	Conc. A	Quan
1	T051_13DEC2011_DSP2_001	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.3	10.000	Standard	0	
2	T051_13DEC2011_DSP2_002	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.4	10.000	Standard	0.125	
3	T051_13DEC2011_DSP2_003	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.5	10.000	Standard	0.25	
4	T051_13DEC2011_DSP2_004	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.6	10.000	Standard	0.5	
5	T051_13DEC2011_DSP2_005	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.7	10.000	Standard	1	
6	T051_13DEC2011_DSP2_006	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.8	10.000	Standard	1	
7	T051_13DEC2011_DSP2_007	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.9	10.000	Standard	1	
8	T051_13DEC2011_DSP2_008	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.10	10.000	Standard	1	
9	T051_13DEC2011_DSP2_009	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.11	10.000	Standard	1	
10	T051_13DEC2011_DSP2_010	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.12	10.000	Standard	1	
11	T051_13DEC2011_DSP2_011	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.13	10.000	Standard	1	
12	T051_13DEC2011_DSP2_012	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.14	10.000	Standard	1	
13	T051_13DEC2011_DSP2_013	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.15	10.000	Standard	1	
14	T051_13DEC2011_DSP2_014	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.16	10.000	Standard	1	
15	T051_13DEC2011_DSP2_015	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.17	10.000	Standard	1	
16	T051_13DEC2011_DSP2_016	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.18	10.000	Standard	1	
17	T051_13DEC2011_DSP2_017	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.19	10.000	Standard	1	
18	T051_13DEC2011_DSP2_018	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.20	10.000	Standard	1	
19	T051_13DEC2011_DSP2_019	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.21	10.000	Standard	1	
20	T051_13DEC2011_DSP2_020	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.22	10.000	Standard	1	
21	T051_13DEC2011_DSP2_021	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.23	10.000	Standard	1	
22	T051_13DEC2011_DSP2_022	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.24	10.000	Standard	1	
23	T051_13DEC2011_DSP2_023	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.25	10.000	Standard	1	
24	T051_13DEC2011_DSP2_024	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.26	10.000	Standard	1	
25	T051_13DEC2011_DSP2_025	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.27	10.000	Standard	1	
26	T051_13DEC2011_DSP2_026	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.28	10.000	Standard	1	
27	T051_13DEC2011_DSP2_027	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.29	10.000	Standard	1	
28	T051_13DEC2011_DSP2_028	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.30	10.000	Standard	1	
29	T051_13DEC2011_DSP2_029	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.31	10.000	Standard	1	
30	T051_13DEC2011_DSP2_030	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.32	10.000	Standard	1	
31	T051_13DEC2011_DSP2_031	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.33	10.000	Standard	1	
32	T051_13DEC2011_DSP2_032	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.34	10.000	Standard	1	
33	T051_13DEC2011_DSP2_033	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.35	10.000	Standard	1	
34	T051_13DEC2011_DSP2_034	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.36	10.000	Standard	1	
35	T051_13DEC2011_DSP2_035	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.37	10.000	Standard	1	
36	T051_13DEC2011_DSP2_036	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.38	10.000	Standard	1	
37	T051_13DEC2011_DSP2_037	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.39	10.000	Standard	1	
38	T051_13DEC2011_DSP2_038	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.40	10.000	Standard	1	
39	T051_13DEC2011_DSP2_039	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.41	10.000	Standard	1	
40	T051_13DEC2011_DSP2_040	mspa1127-2_SJC_window	SOPA1127 UPLC-2	1.42	10.000	Standard	1	

Multi-residue Veterinary drugs

Sample preparation approach

MILK

Protein Precipitation

Add 4 mL of 0.2 % formic acid ACN to 1 mL of sample

TISSUE

Extraction/Precipitation

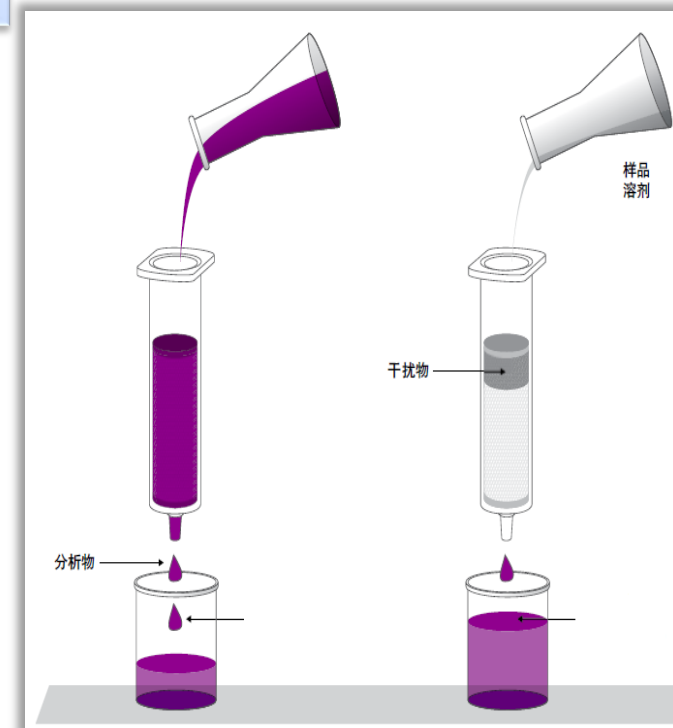
Add 10 mL of 0.2 % formic acid in 80:20 ACN/water to 2.5 g of sample

Centrifuge

Take aliquot supernatant

Pass-Through
PRiME HLB SPE
Cleanup

Pass through



Multi-residue pesticides

Sample preparation approach

samples

Extraction

Measure out 10 g sample +10 mL water+ 15mL of 1% acetic acid in Acetonitrile into DisQuE tube 1



Centrifuge

Centrifuge and transfer 1 mL of the extraction into DisQuE tube 2



Centrifuge

Dilute 10 times with water (~ 15x dilution)

Dispersion SPE



Mycotoxins

Sample preparation approach

grain

Extraction

Measure out 10 g of sample+10 mL water+ 10mL of 10% acetic acid in Acetonitrile into DisQuE tube 1



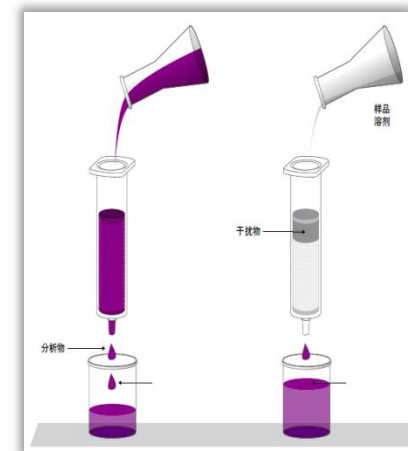
Centrifuge

Centrifuge and transfer 1 mL of the extraction **into PRiME HLB**



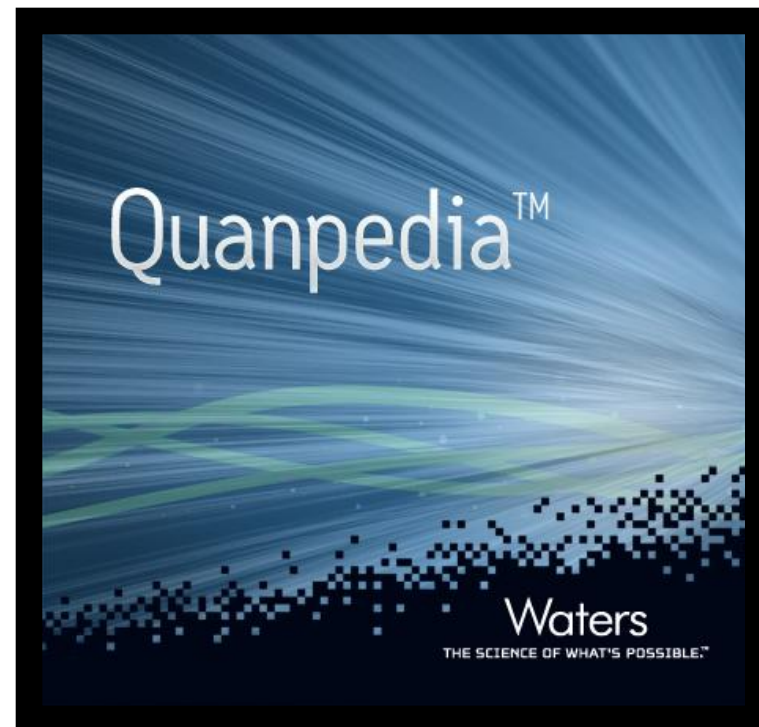
Pass-Through SPE Cleanup

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Instrument Conditions

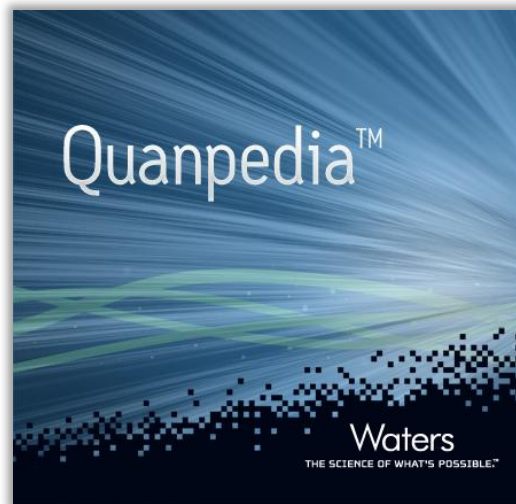
- A central data base for quantitative LC/MS methods
- Based on compound name or predefined analysis
- A tool to aid MS method creation
 - Automatically creates data **acquisition methods**
 - Automatically creates data **processing methods**



Quanpedia database

—set up & update for China customer needs

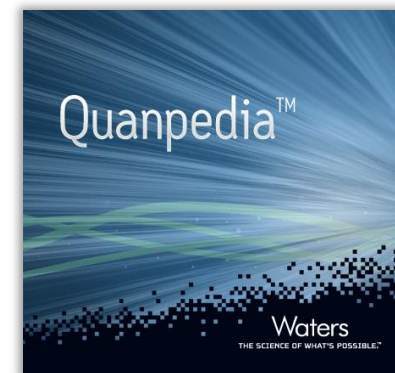
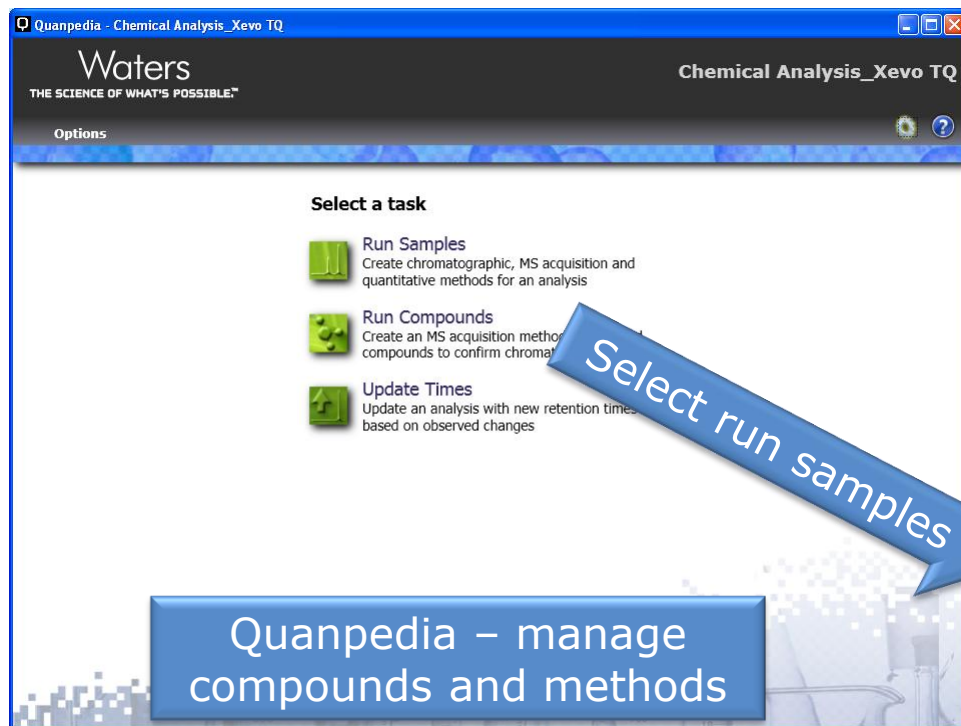
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- mycotoxins.qdb
- 327 Pesticides Screen TQ-S.qdb
- 110 vet drug Screen TQ-S.qdb

create multi-residue methods from Quanpedia database directly

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Configure Analysis

Method files and a sample batch list are required to run this analysis.

Enter the name to use for all the files created, the project in which they will be created and specify which files to create.

Name:

Project:

Files to create:

- ☒ MS method
- ☒ Chromatography method
- ☒ TargetLynx method
- ☒ Sample list

IntelliStart



UPLC Conditions

- Fixed retention time based on given UPLC condition
- Should check R-T before injection sample ($\pm 0.2 \sim 0.5$ min)

MassLynx - quanpedia - 327 pesticides_test.spl

File View Run Help

Queue Is Empty

Spectrum Chromatogram Map Edit Samples

	File Name	Sample ID	File Text	MS File	Inlet File	Bottle
1				327 pesticides_test	327 pesticides...	

327 pesticides_test.vvhp, 327 pesticides_test.ft, Inlet Method

File View Tools LC Acq

Status

Ind

Det

Se

For Help, press F1

Ready

No Instrument

0:0

Shutdown Disabled

Modify ACQUITY Binary Solvent Manager Instrument Method

Run Time: 17.00 min

General Data Analog Out Events

Solvents

A2 water with 10mM NH4

B2 MeOH with 10mM NH4

Pressure Limits

Low: 0 psi

High: 0 psi

Seal Wash: 5.0 min

Gradient:

	Time (min)	Flow (mL/min)	%A	%B	Curve
1	Initial	0.450	98.0	2.0	Initial
2	0.25	0.450	98.0	2.0	6
3	12.25	0.450	1.0	99.0	6
4	13.00	0.450	1.0	99.0	6
5	13.01	0.450	98.0	2.0	6
6	17.00	0.450	98.0	2.0	6

Comment:

OK Cancel

TargetLynx Edit Method

Process Samples

View Results

TrendPlot

Quanpedia

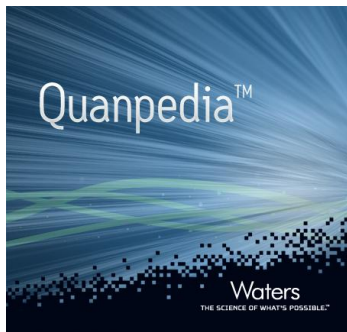
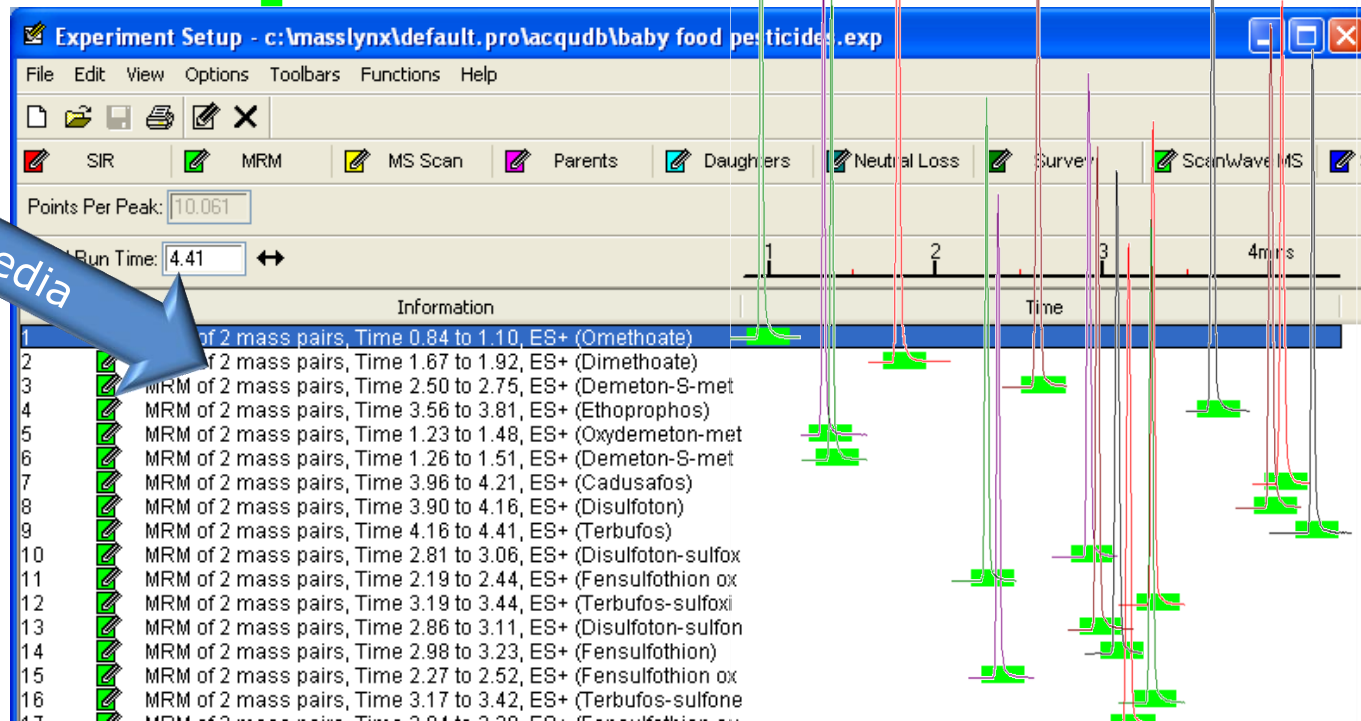
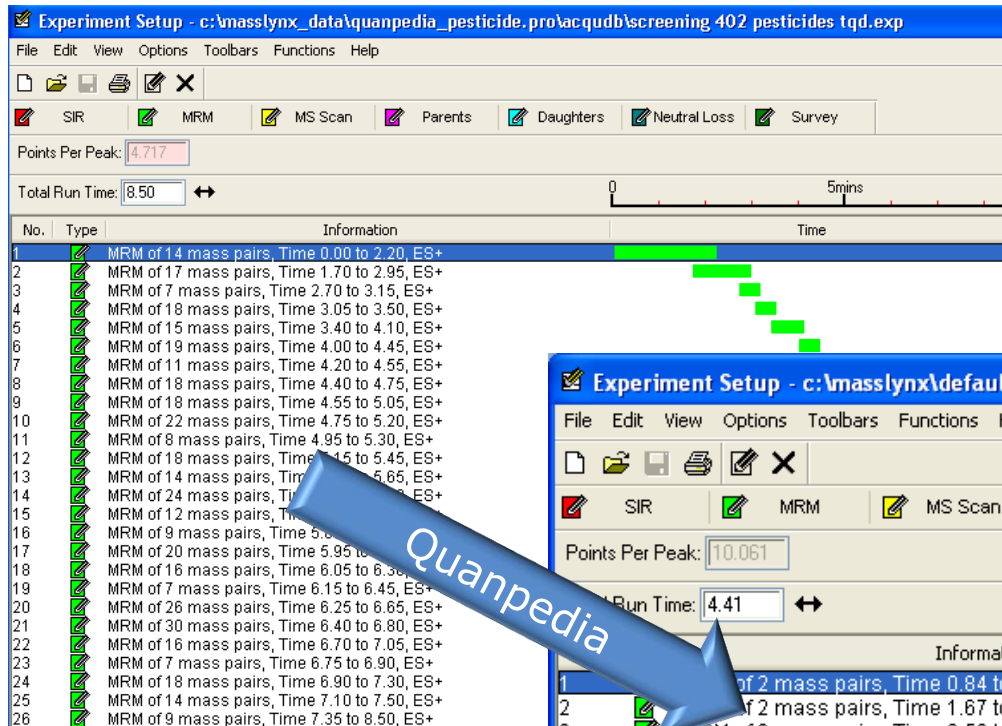
System Status

Not Ready

MRM Conditions

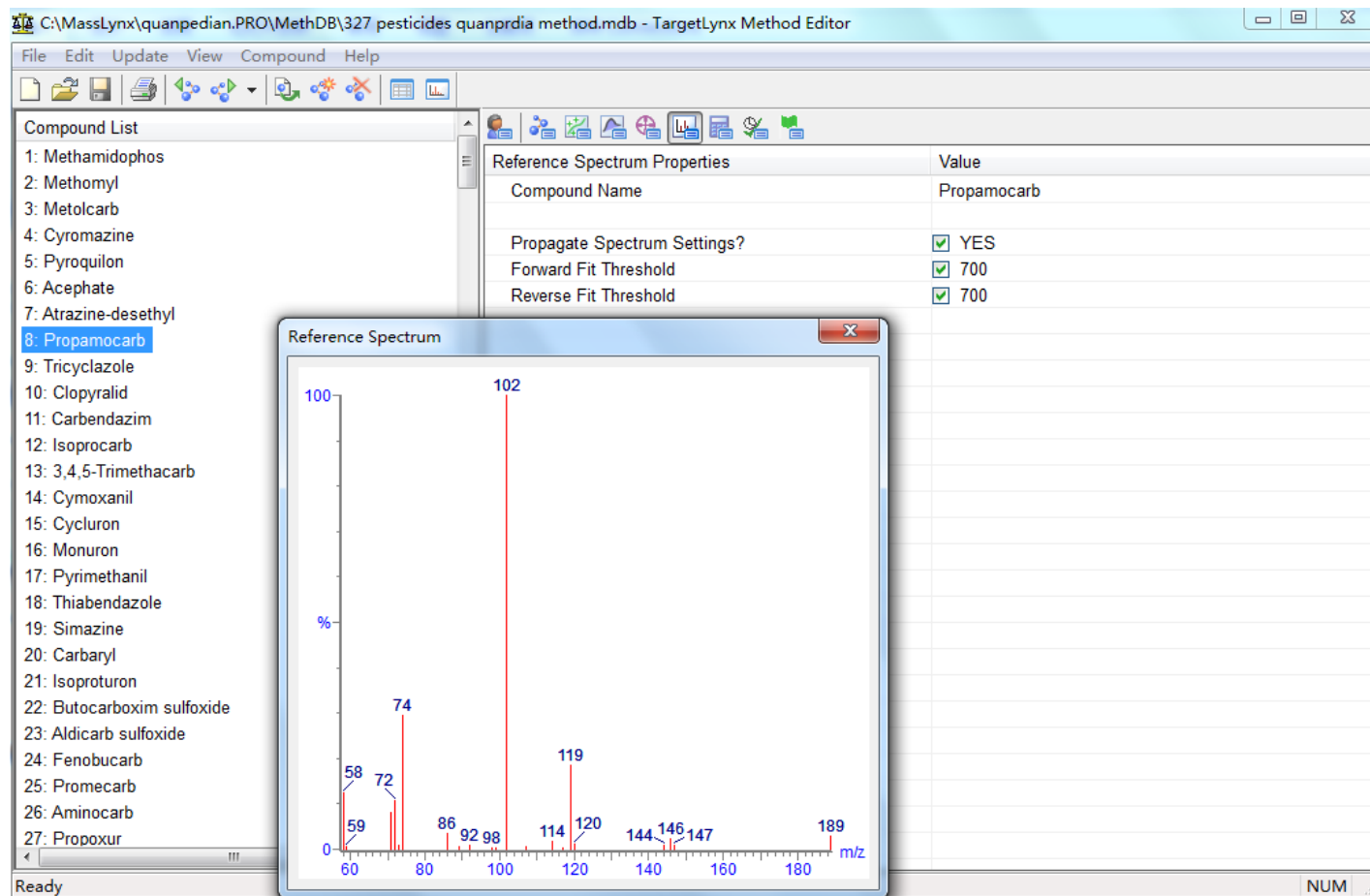
—completely pre-defined analysis

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Data processing methods include PICs reference spectrum

- Acquire all compounds' PICs reference spectrum and predefined in Quanpedia database





Results and Conclusions

[APPLICATION NOTE]

Rapid Detection of Pesticide Residues in Fruit Juices Without Sample Extraction Using UPLC-MS/MS

Dimple Shah, Jinchuan Yang, Gordon Fujimoro, Lauren Mullin, and Jenn Waters Corporation, Milford, MA, USA

APPLICATION BENEFITS

Pesticides can be detected below legislative limits in fruit juices using a "dilute and shoot" approach with the ACQUITY UPLC® I-Class System coupled to the Xevo® TQ-S Mass Spectrometer.

- Ultra-sensitive Xevo TQ-S facilitates trace level detection of pesticides.
- Dilute and shoot approach reduces sample preparation time and improves laboratory efficiency.
- Dilute and shoot approach provides excellent repeatability.
- Simple QuEChERS extraction can be employed prior to dilution for complex matrices.

WATERS SOLUTIONS

ACQUITY UPLC I-Class System

Xevo TQ-S Mass Spectrometer

ACQUITY UPLC BEH Column

Masslynx™ Software

Quanpedia™ Database

DtsQuE™ Sample Preparation Kit

KEY WORDS

Pesticides, fruit juice, MS, Quanpedia, QuEChERS, food safety, carbendazim, rotenone

[APPLICATION NOTE]

A Simple Cleanup Protocol Analysis of Multi-Residue

DeFeng Huang, Kim Van Tran, and Michael Waters Technologies, Ltd., Shanghai, China

APPLICATION BENEFITS

- Enable simultaneous determination of multi-class of veterinary drugs using an innovative solid phase extraction device
- Simple, fast, pass-through SPE cleanup prior to UPLC-MS/MS analysis
- The matrix interference from fatty/non-polar materials and phospholipids are removed together in one straightforward SPE cleanup for longer column life and less maintenance of the mass spectrometer

INTRODUCTION

Pesticide residues especially taking in report concerning widespread fruits in the United (FDA) began testing

Many published methods for regulatory purposes in order to technologies, name screening methods multi-residue analysis



Figure 1. Partial list of

WATERS SOLUTIONS

ACQUITY UPLC® I-Class System

Xevo® TQ-S Mass Spectrometer

ACQUITY UPLC BEH C₁₈ Column

Oasis® PRIME HLB 3 cc 60 mg cartridges

TriView™ LC/MS Certified Vials

Masslynx® v4.1 data system with

Quanpedia™ database

KEY WORDS

Oasis PRIME HLB, multi-residue, veterinary drug, SPE, milk, UPLC-MS/MS

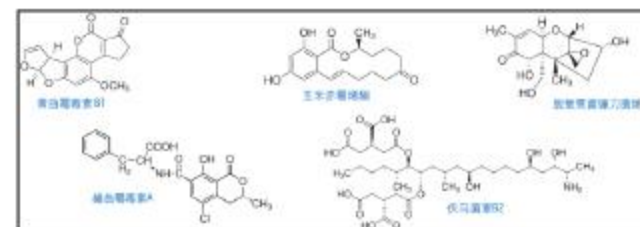


1 简介

1.1 应用背景

霉菌毒素是由霉菌或真菌产生的存在于动物食品或其他人类消费的食品上的有毒化合物，摄入的食物中即使仅含十亿分之一浓度的霉菌毒素也有可能引发严重疾病。因此，我们需要灵敏可靠的分析方法来确定食品和饲料中的霉菌毒素。下文简要介绍了一些重要的霉菌毒素种类，图 1 展示了一些重要霉菌毒素的结构。

图 1. 一些重要的真菌毒素



黄曲霉毒素：被世界卫生组织（WHO）的癌症研究机构划定为 1 类致癌物，是毒性最强、危害最大的一类霉菌毒素，接触后可能引发肝癌、生殖问题、贫血症、免疫系统抑制和黄疸等疾病。GB 2761-2011 规定黄曲霉毒素 B1 在谷物类食品中的限值为 5.0 µg/kg。

伏马菌素：伏马菌素 B1 最为常见，玉米是最易受其感染的作物，接触后可能导致摄食量和体重减少、肝损伤以及肺水肿。伏马菌素还是潜在致癌物。FDA 指导原则规定人类摄入的食品中伏马菌素总量的限值为 2 µg/kg。中国尚未针对食品中的 OTA 水平建立相关规定。

China GB Methods for multi-residue Veterinary Drug Analysis for example

Method	Analytes	Sample Matrix
GB/T22286	β-Adrenergic agonists	Muscle respectively
GB/T21315	β-Lactam	Kidney and muscle
GB/T22338	Chloramphenicol	Muscle
GB/T20762	Macrolide	Liver, kidney, and muscle
GB/T21316	Sulfonamides	Liver and muscle
GB/T20366	Fluoroquinolone	Liver and muscle

Combined

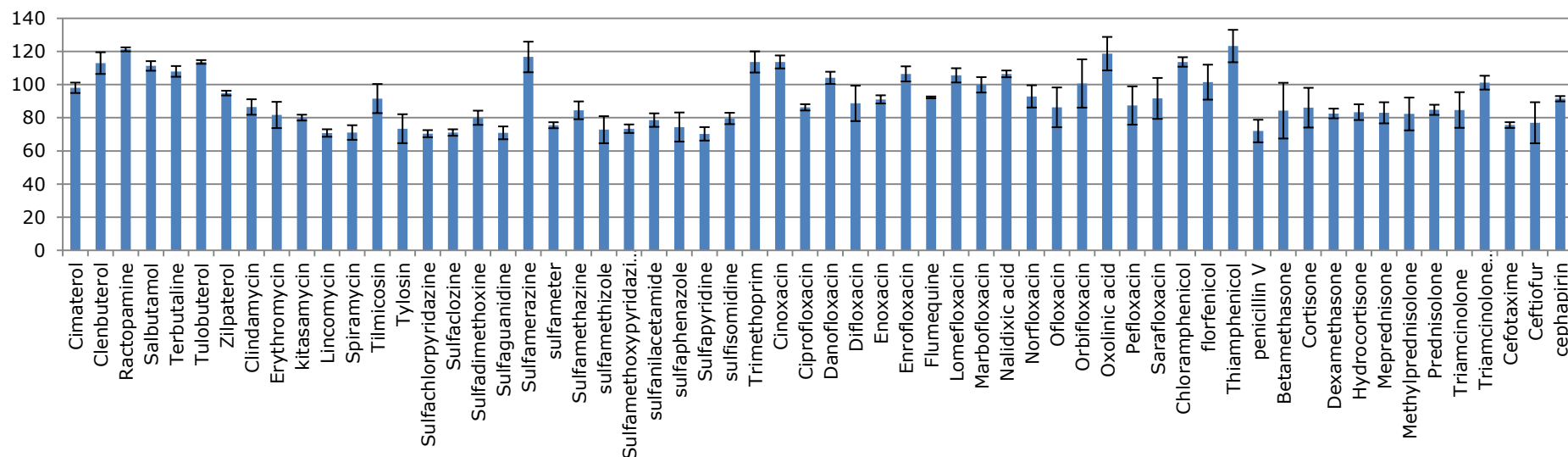
Using Oasis PRiME HLB

-Enable multi-class compounds to be analyzed using one sample prep method.

Using Oasis Xevo TQ-S with Quanpedia database

-Enable multi-class compounds to be analyzed by one injection.

Recovery of Multi-residue Veterinary from Milk (80 compounds in 9 drug classes)



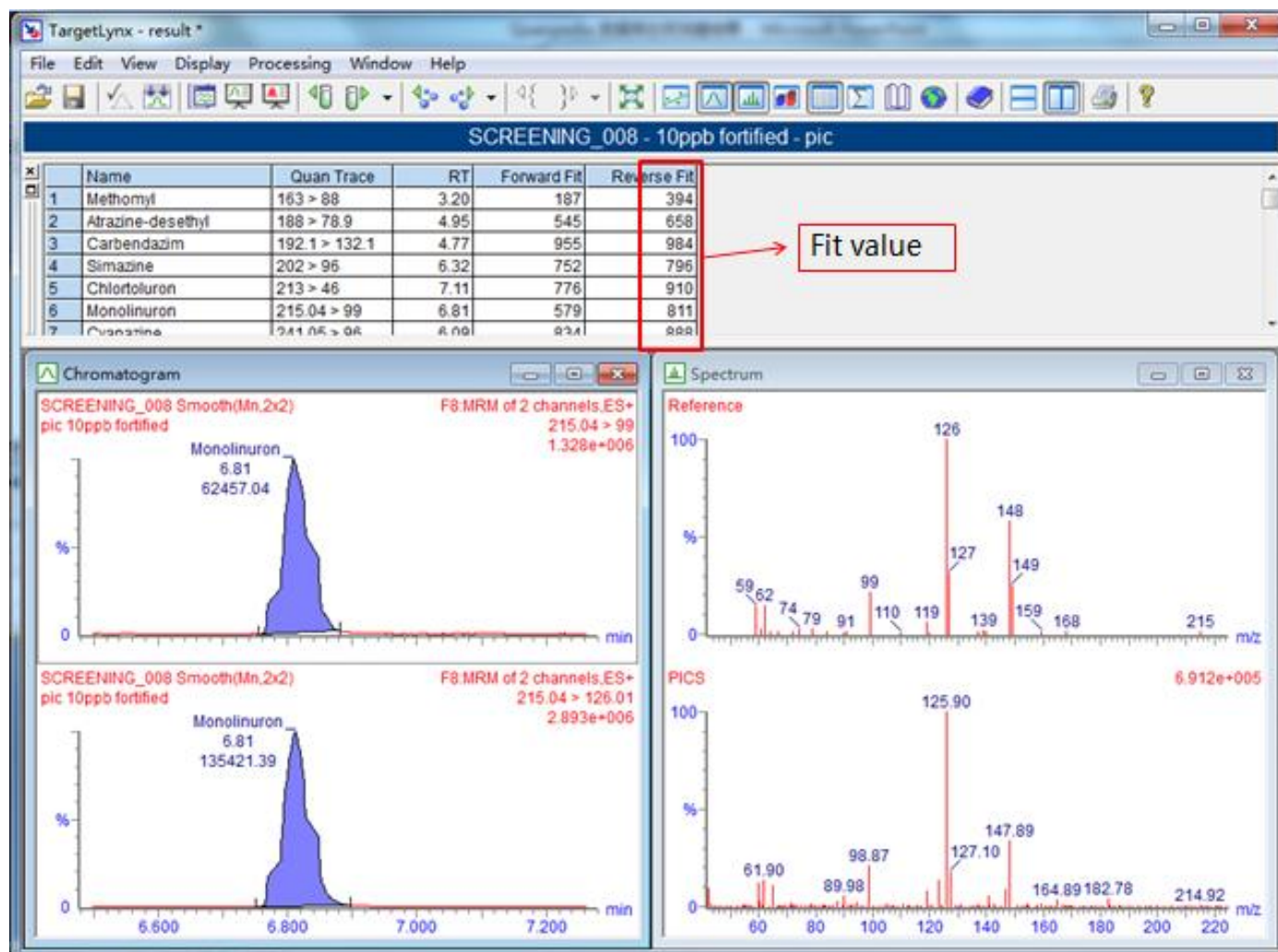
One single method replaces 9 separate methods!!!

Excellent recoveries ranging from 50% to 130% with precision (RSD) < 20% (n=5) for all compounds (Average recovery 91%, %RSD @ 6 (n=5))

Recovery values are a subject to the initial milk extraction efficiency

TargetLynx Reporting

- Predefined PICs reference spectrum make less false positive results
- Extended application of target screening



Conclusions

- Variety of different multi-residue solution include pesticides, Veterinary drugs and mycotoxins have been created.
- Simple sample preparation approach and Quanpedia database make customer easier to use the solution.
- Predefined PICs reference spectrum makes less false positive results.



谢谢！