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Solution of multi-residue analysis in food safety

Dr Yu Yanling

Presentation Overview

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- 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

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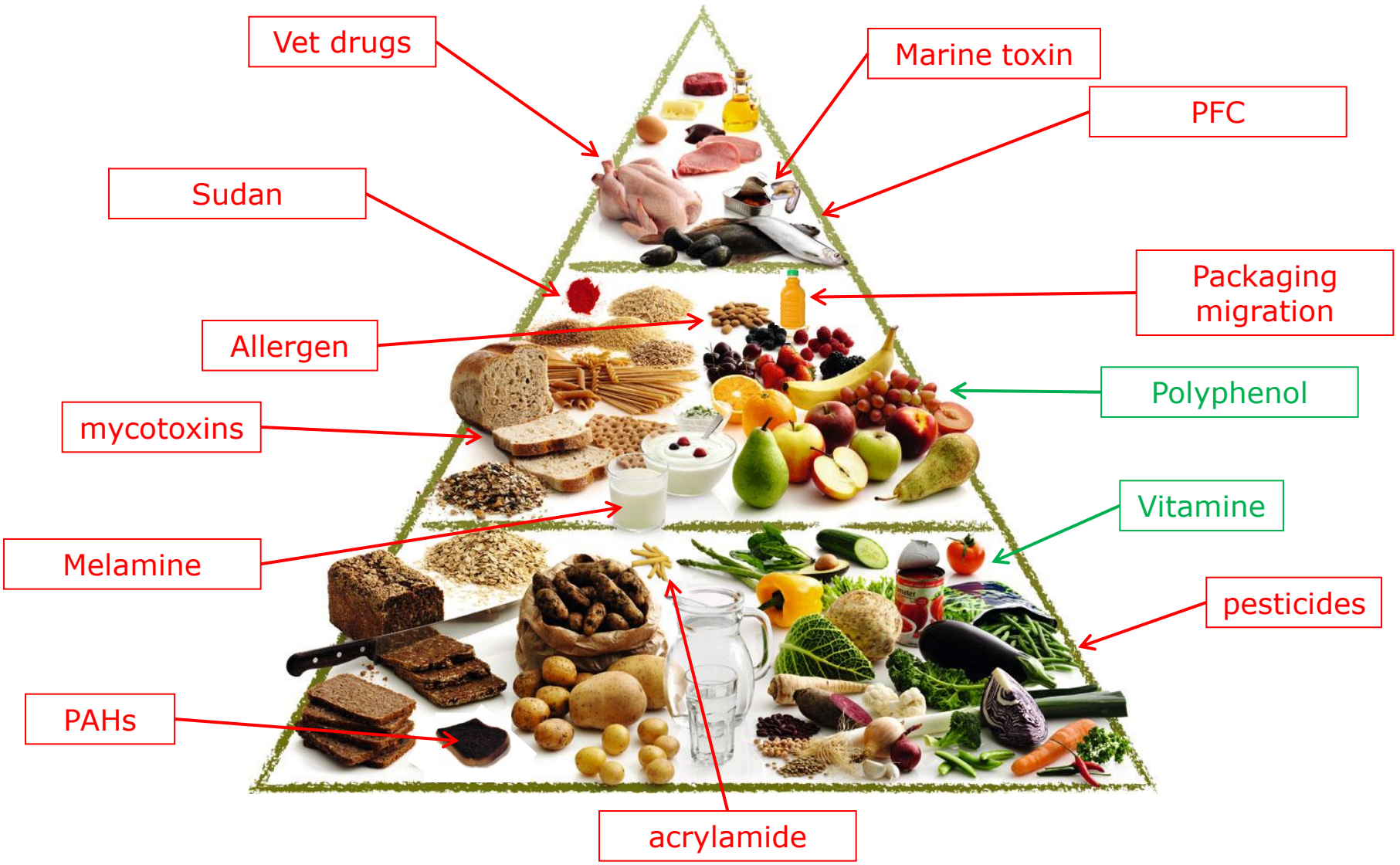

What are the challenges?

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Variety of methods

mycotoxins

Pesticides

Veterinary drugs

Food additives

Contaminants

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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?

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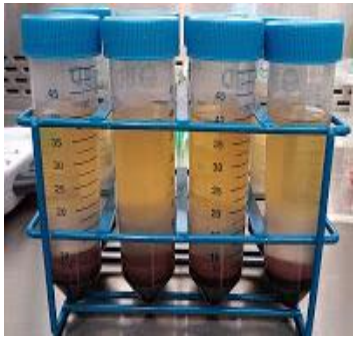
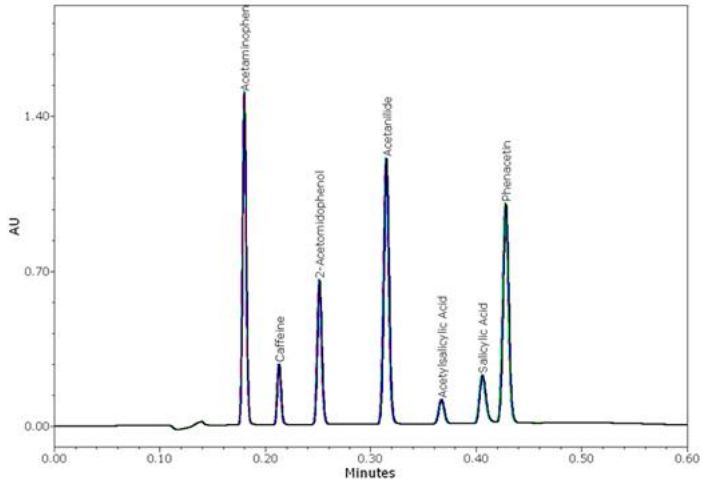
How can I turn this?



Into this?



Three major classes compounds in food safety

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Pesticides

Organophosphates
Chlorpyrifos, Diazinon
Carbamates
Aldicarb, Carbaryl
Organochlorine
DDT, Endosulfan
Pyrethroids(菊酯)
Cyfluthrin, Pyrethrin
Sufonylureas?
Rimsulfuron玉嘧磺隆,
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
Fumonisins
Fumonisin A
Trichothecenes
HT-2 Toxin,
Deoxynivalenol (DON)
Ochratoxins
Ochratoxin A
Estrogenic Metabolites
Zearalenone
Phenolic
Citrinin

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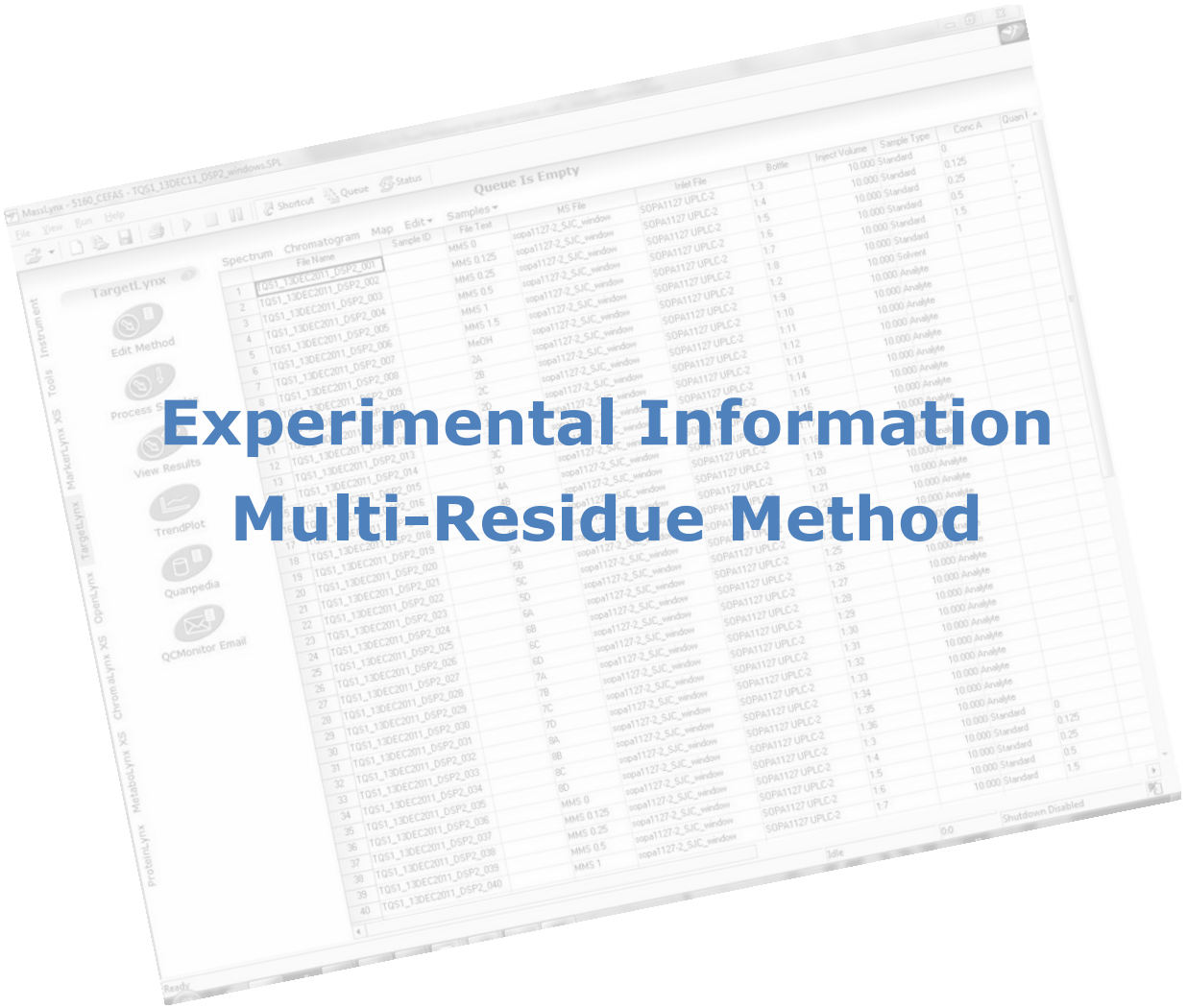

Experimental Information
Multi-Residue Method

Multi-residue Veterinary drugs Sample preparation approach

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MILK

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

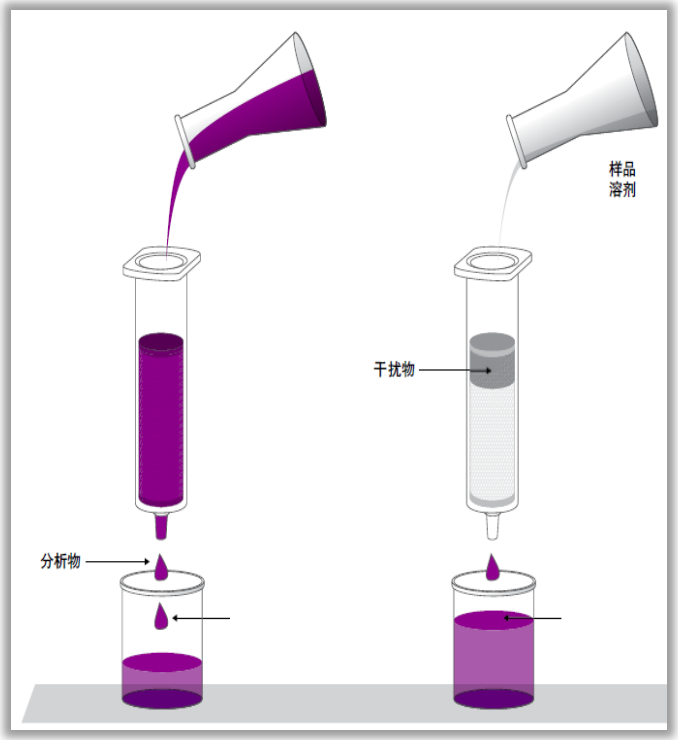
TISSUE

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 hrough



Multi-residue pesticides
Sample preparation approach

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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

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grain

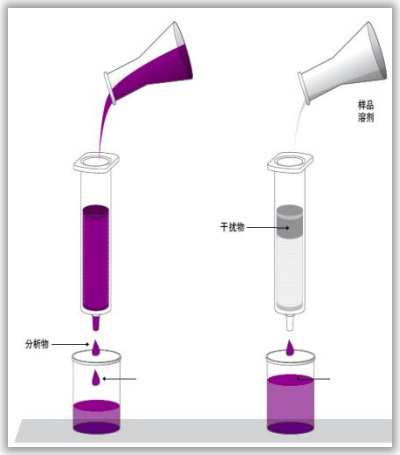
Extraction

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



Instrument Conditions

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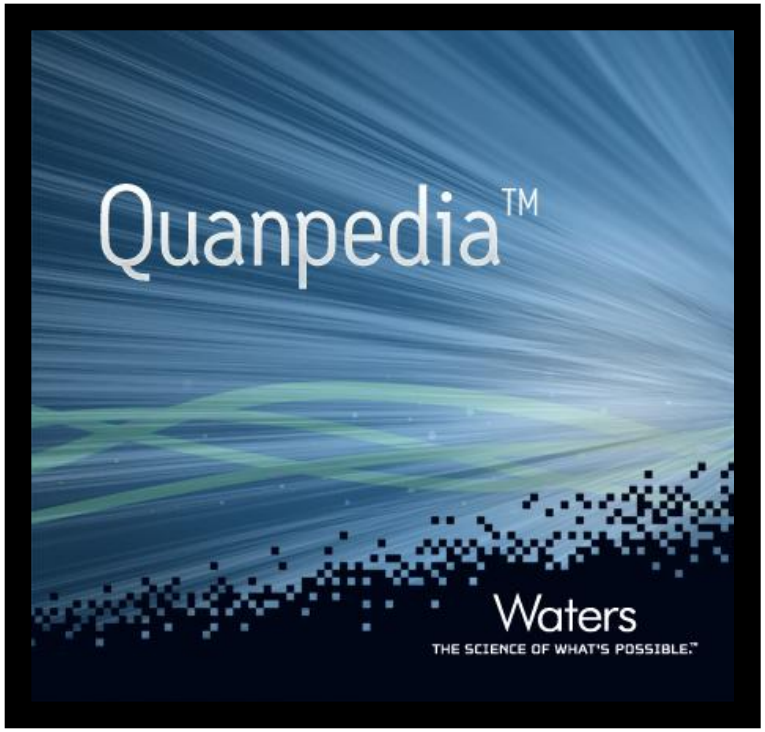
- A central data base for quantitative LC/MS methods
- Based on compound name or predefined analysis
- A tool to aid MS method creation
  - Automatically creates data **acquisition methods**
  - Automatically creates data **processing methods**



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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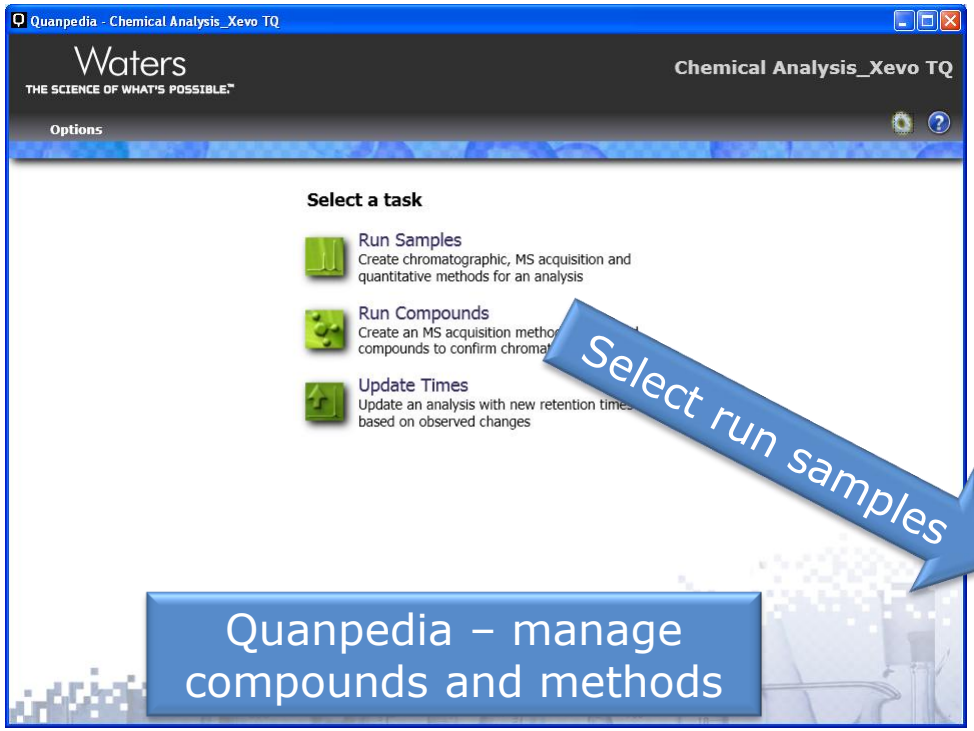
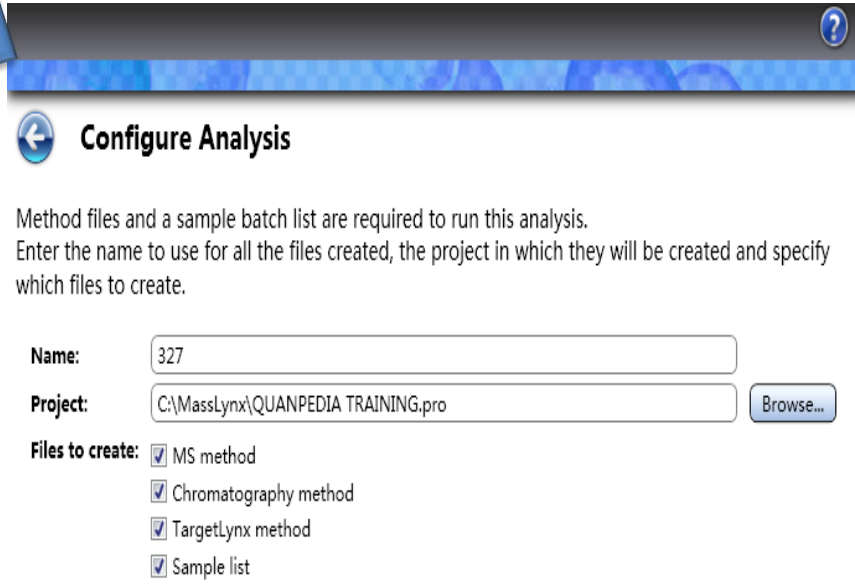


Select run samples

Quanpedia – manage compounds and methods

IntelliStart



UPLC Conditions

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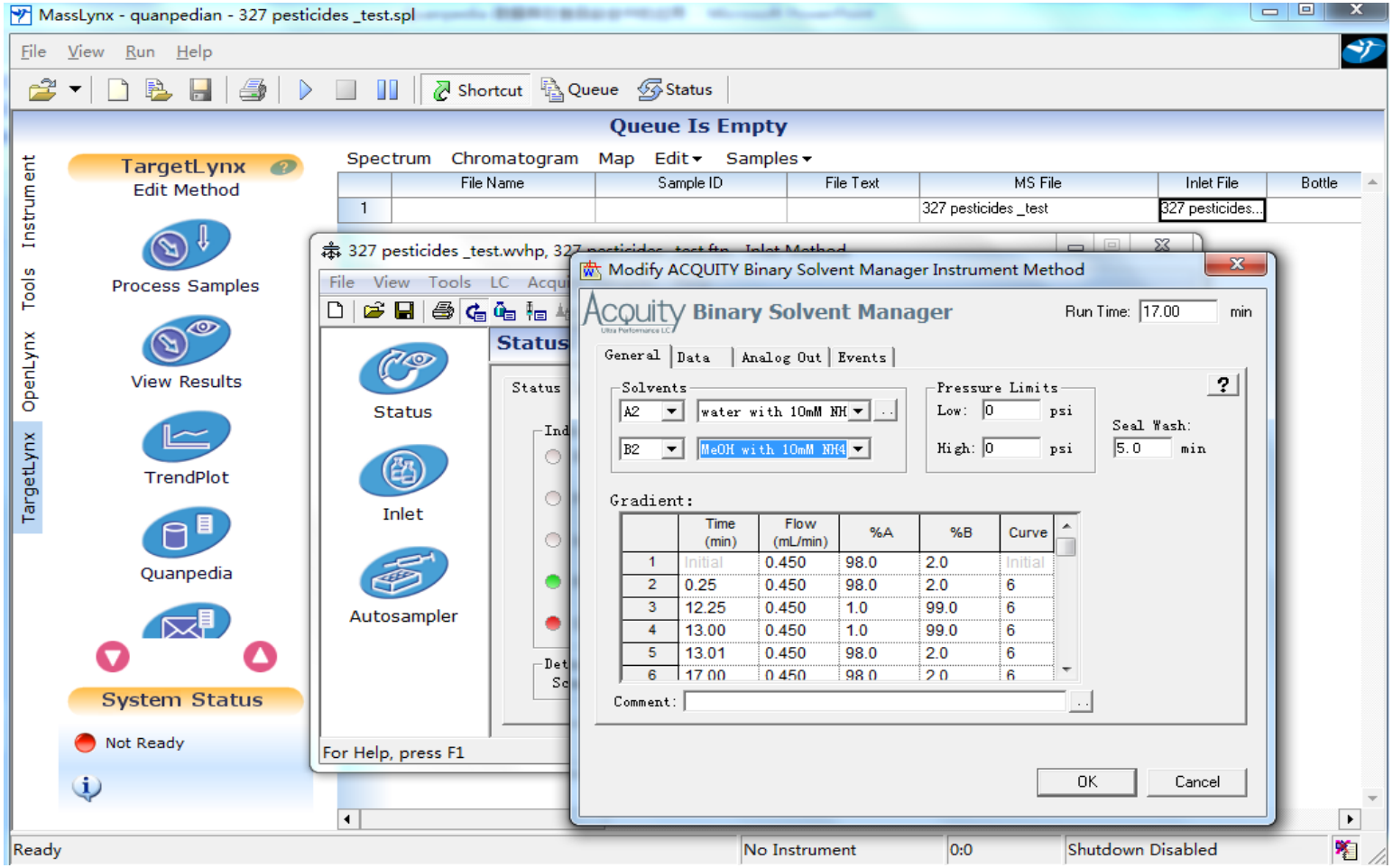
- Fixed retention time based on given UPLC condition
- Should check R-T before injection sample (±0.2~0.5min)



MRM Conditions ——completely pre-defined analysis

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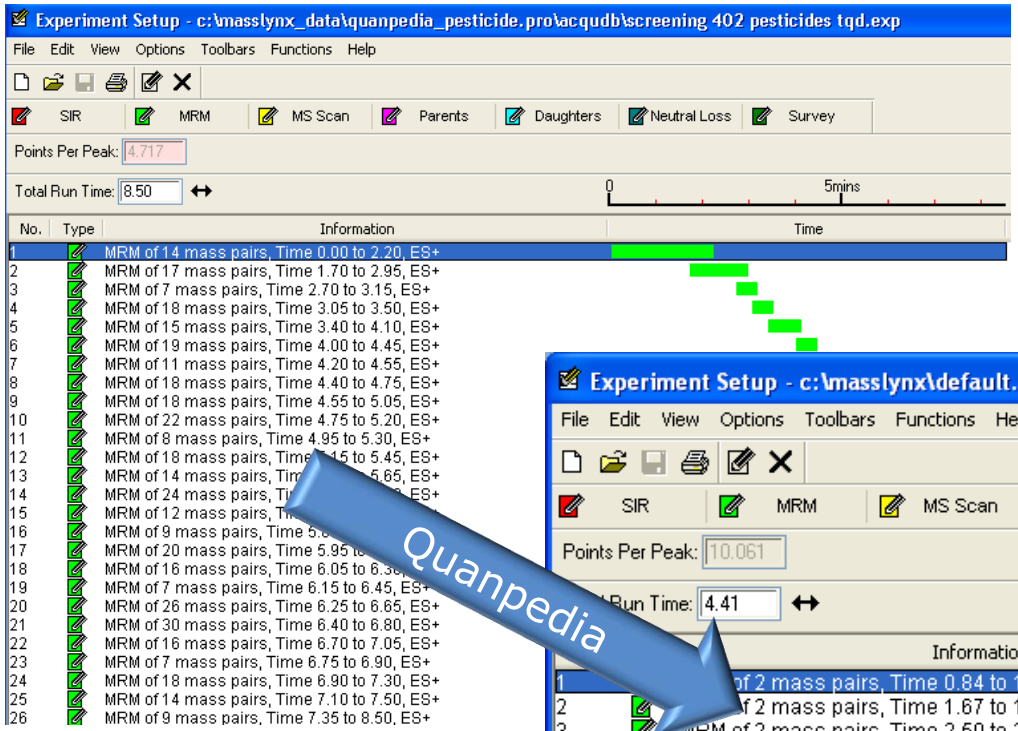
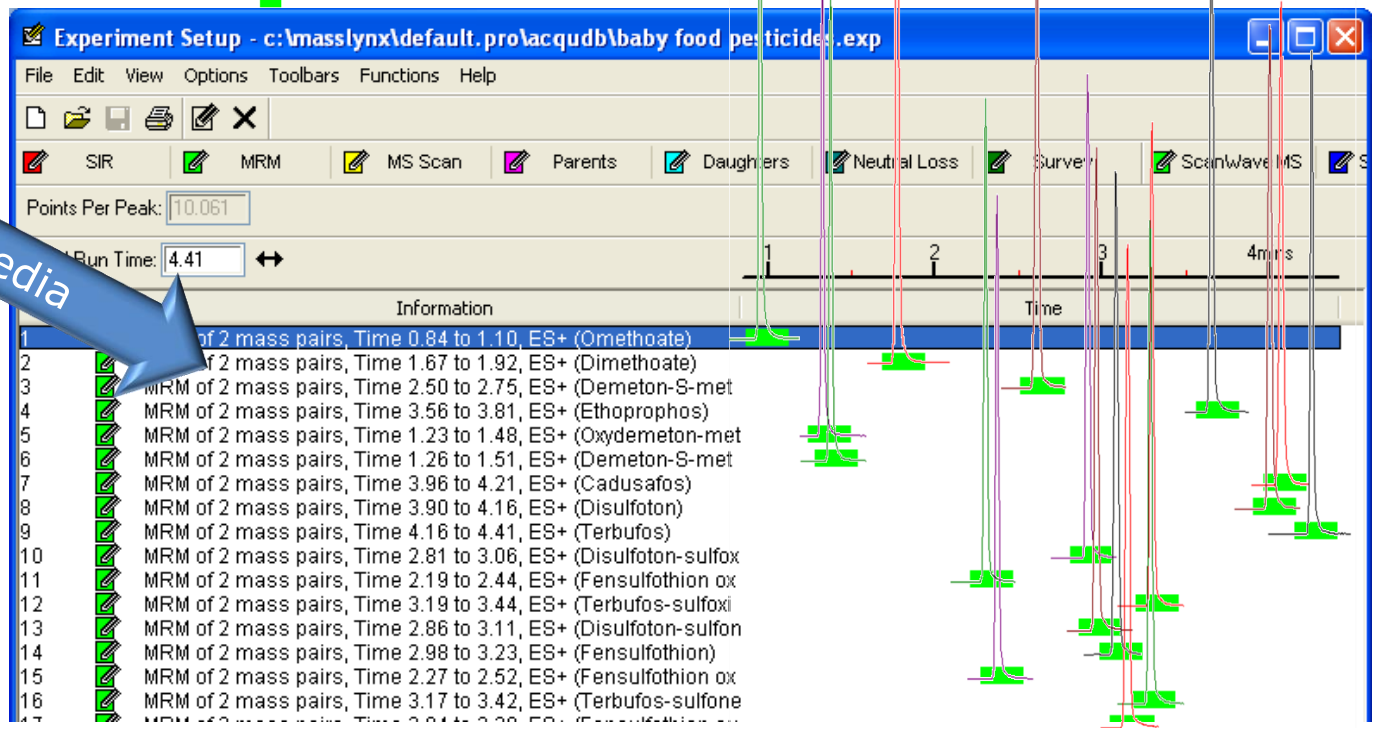
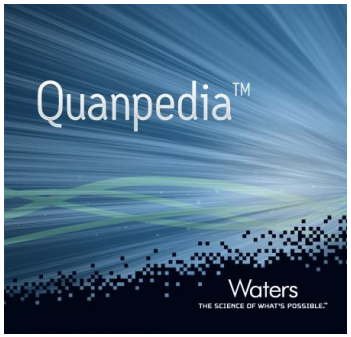


Quanpedia





Data processing methods include PICs reference spectrum

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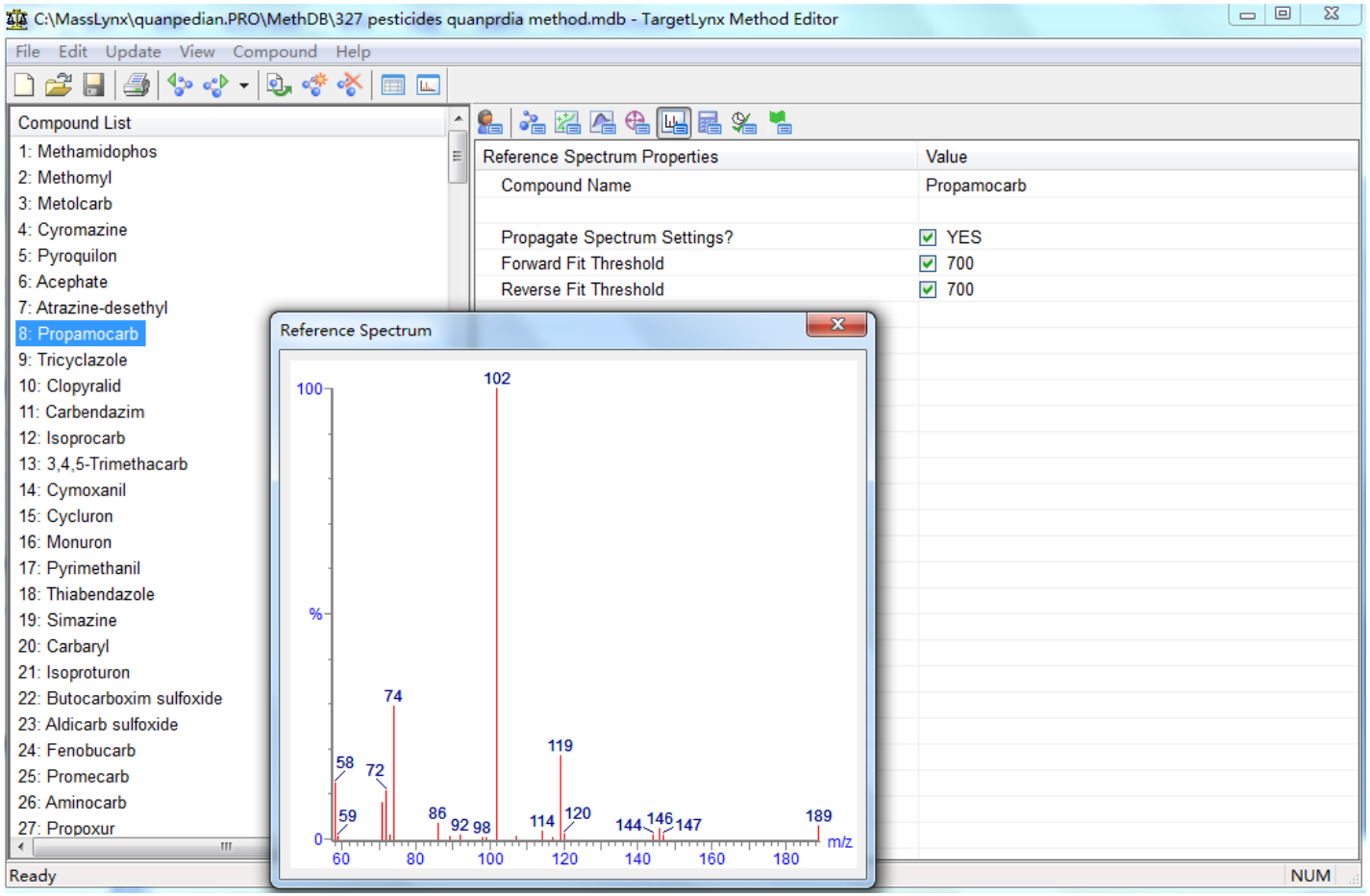
- Acquire all compounds' PICs reference spectrum and predefined in Quanpedia database



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Results and Conclusions

Application Notes

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[APPLICATION NOTE]

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

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

APPLICATION BENEFITS

Pesticides can be detected below legislative limits in fruit juice 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

DisQuE™ Sample Preparation Kit

KEY WORDS

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

INTRODUCTION

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

Many published me
for regulatory purp
methods in order t
technologies, name
screening method
multi-residue anal

Figure 1. Partial list of

[APPLICATION NOTE]

A Simple Cleanup Protoco
Analysis of Multi-Residue

DeFeng Huang,[1] Kim Van Tran,[2] and Michael
[1]Waters Technologies, Ltd., Shanghai, Chin

APPLICATION BENEFITS

- Enable simultaneously 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-pol materials and phospholipids are removed together in one straightforward SPE cleanu for longer column life and less maintenanc of the mass spectrometer

WATERS SOLUTIONS

ACQUITY UPLC® I-Class System

Xevo® TQ-S Mass Spectrometer

ACQUITY UPLC BEH C_{18} Column

Oasis® PRiME HLB 3 cc 60 mg cartridges

TruView™ LCMS Certified Vials

MassLynx® v4.1 data system with Quanpedia™ database

KEY WORDS

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

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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，摄取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测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 5.0μg/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 2μg/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

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Method	Analytes	Sample Matrix
GB/T22286	β-Adrenergic agonists	Muscle respectively
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GB/T22338	Chloramphenicol	Muscle
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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)

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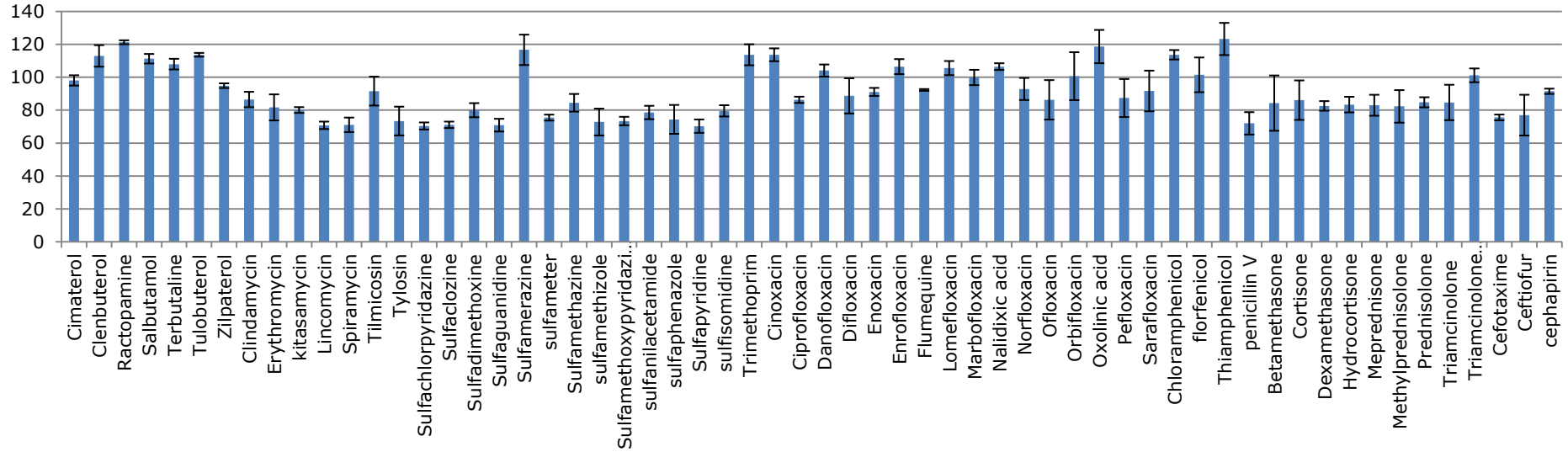

One single method replaces 9 separate methods!!!

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

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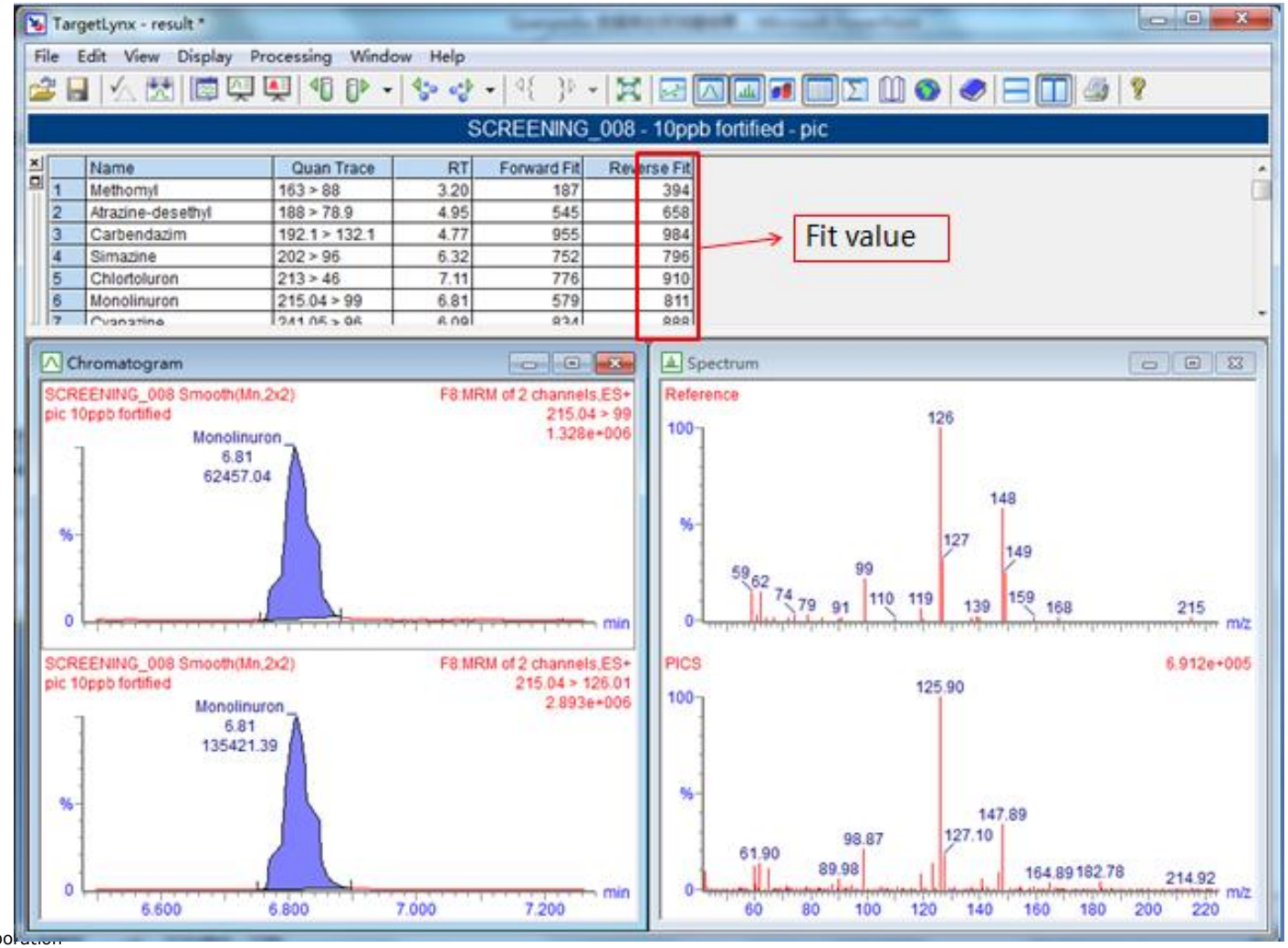
- Predefined PICs reference spectrum make less false positive results
- Extended application of target screening



Conclusions

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- 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.

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谢 谢！