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# Development of the Integrated Solution of Data Processing and Combined Reporting of Multi-Residue Pesticides Analysis using both GC/MS and LC/MS

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## Introduction

In order to cover the various properties (highly polar to non-polar) of multi-residue pesticides, their analysis requires the use of both GC/MS and LC/MS at the same time, which means operators will face the challenge of expending time and energy on results merging and its reporting. The development of a further streamlined and automated solution on data analysis and reporting becomes crucial for productivity improvement. A new automated and combined solution based on the MassHunter quantitation workflow and customized report template is developed to speed up the data processing and reporting for multi-residue pesticides analysis from GC/MS and LC/MS. This integrated solution greatly reduces labor-intensive manual steps and increases productivity.

## Experimental

### 1290 UHPLC System Conditions

Column: Agilent InfinityLab Poroshell 120 EC-18, 2.1x100mm, 2.7 $\mu$ m

Mobile phase A: (0.1% HCOOH+5mM NH<sub>4</sub>CO<sub>2</sub>H) in water

Mobile phase B: acetonitrile : mobile phase A= 95:5

Column Oven: 40 °C

Gradient program: (Flow rate: 0.3 mL min<sup>-1</sup>)

Time (min)	0	1	12	14
A (%)	70	70	0	0
B (%)	30	30	100	100

Post Time: 3min

### 6470 LC/TQ System Conditions

Ion source: AJS ESI, Positive Mode

Nebulizer gas: 35psi

Dry gas: 7 L min<sup>-1</sup>

Dry gas Temperature: 250°C

Sheath gas: 11 L min<sup>-1</sup>

Sheath gas Temperature: 325°C

Nozzle voltage: 0V

Capillary voltage: 3500V

Acquisition mode: Dynamic MRM

## Experimental

### 8890 GC System Conditions

Column: Agilent VF-17 MS, 30m x 0.25mm, 0.25 $\mu$ m

Injection volume: 1  $\mu$ L

Injection mode: splitless

Inlet temperature: 280°C

Carrier gas: He, constant pressure 146kPa

MS transfer line temperature: 310°C

Oven program:

60°C for 1 minutes;

then 30°C/min to 120°C;

then 10°C/min to 160°C;

then 2°C/min to 230°C;

then 15°C/min to 300°C(6 minutes hold).

### 7000D GC/TQ System Conditions

MS source: EI, 70 eV

Source temperature: 250°C

Quadrupole temperature: 150°C

Solvent delay: 3min

Acquisition mode: MRM



Figure 1. Agilent 1290/6470 Triple Quadrupole LC/MS



Figure 2. Agilent 8890/7000 Triple Quadrupole GC/MS

### Integrated data processing and reporting

According to the general principles 0212 of the Chinese Pharmacopoeia 2020(ChP 2020), 55 pesticides from 33 groups in Traditional Chinese Medicines (TCM) matrices need to be detected. Among them, 30 pesticides are analyzed by LC/MS, and 35 pesticides are analyzed by GC/MS. There are 10 compounds overlapping the techniques. Additionally, 10 groups should be summed first, then compared with the maximum residue limit (MRL). Compared to a general workflow, the combined report workflow provides a faster and automated solution using existing software functionality and a customized template.

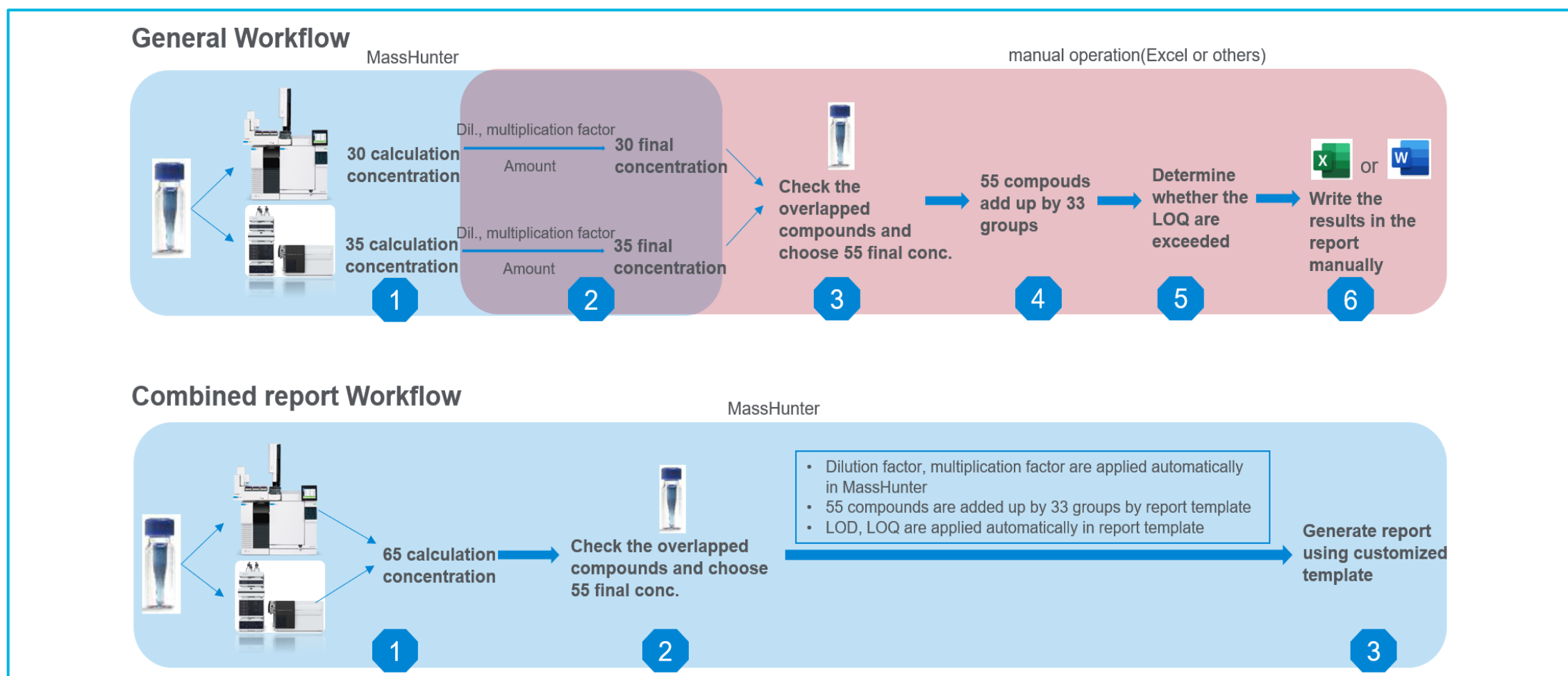


Figure 3. The comparison between general workflow and combined report workflow

The Quant-my-way feature of MassHunter software makes interface customization easy to achieve. In this solution, several parameters were designed for calculation and sample group; all of them were language localized. The final concentration(mg/Kg) is equal to Calculation Concentration x Dilution Factor x Multiplication Factor / Amount.

Batch Table

Sample: JL-1-2

Parameters designed for Chinese users: Amount, Dilution factor, Multiplication factor, GCMS or LCMS, Report No., Sample Group

ISTD: [ ]

Sample				Monocrotophos_L...		Monocrotophos_LCMS Results											
Name	Data File	Type	Level	样品重量	稀释比	样品体积	GCMS 或 LCMS	报告编号	样品组	残留物	Exp. Conc.	RT	Resp.	MI	Calc. Conc.	最终结果 (mg/kg)	Accuracy
YL1-1	YL1-1.d	Blank		1000	1.0	0.1	LCMS	20210713		久效磷		0.953	35.6393		0.1635		0.0000
YL1-2	YL1-2.d	Blank		1000	1.0	0.1	LCMS	20210713		久效磷		0.923	14.9742		0.1625		0.0000
BL-1-1	BL-1-1.d	Cal	5	1000	1.0	0.1	LCMS	20210713		久效磷	100.0000	0.933	2089550.40...		99.8693	0.0100	99.9
BL-1-2	BL-1-2.d	Cal	4	1000	1.0	0.1	LCMS	20210713		久效磷	50.0000	0.933	1051997.84...		50.3602	0.0050	100.7
BL-1-3	BL-1-3.d	Cal	3	1000	1.0	0.1	LCMS	20210713		久效磷	25.0000	0.933	518133.4200		24.8857	0.0025	99.5
BL-1-4	BL-1-4.d	Cal	2	1000	1.0	0.1	LCMS	20210713		久效磷	12.5000	0.933	254005.7915		12.2822	0.0012	98.3
BL-1-5	BL-1-5.d	Cal	1	1000	1.0	0.1	LCMS	20210713		久效磷	6.2500	0.923	129739.0057		6.3526	0.0006	101.6
JL-1-1	JL-1-1.d	Sample		1000	1.0	0.1	LCMS	20210713	1	久效磷		0.938	119682.9790		5.8727	0.0006	
JL-1-2	JL-1-2.d	Sample		1000	1.0	0.1	LCMS	20210713	2	久效磷		0.938	118688.5904		5.8253	0.0006	
1-YG-1	1-YG-1.D	Blank		1000	1.0	0.1	GCMS	20210713		久效磷							
1-YG-2	1-YG-2.D	Blank		1000	1.0	0.1	GCMS	20210713		久效磷							
1-BG-1	1-BG-1.D	Cal	L5	1000	1.0	0.1	GCMS	20210713		久效磷							
1-BG-2	1-BG-2.D	Cal	L4	1000	1.0	0.1	GCMS	20210713		久效磷							
1-BG-3	1-BG-3.D	Cal	L3	1000	1.0	0.1	GCMS	20210713		久效磷							
1-BG-4	1-BG-4.D	Cal	L2	1000	1.0	0.1	GCMS	20210713		久效磷							
1-BG-5	1-BG-5.D	Cal	L1	1000	1.0	0.1	GCMS	20210713		久效磷							
1-JG-1	1-JG-1.D	Sample		1000	1.0	0.1	GCMS	20210713	1	久效磷							
1-JG-2	1-JG-2.D	Sample		1000	1.0	0.1	GCMS	20210713	2	久效磷							

Customer can switch to different compounds in this view. And this compound(Monocrotophos) was analyzed by LCMS, so there is no result for GCMS samples.

Figure 4. Customized interface for combined report workflow

## Results and Discussion

The same set of data from GC/MS and LC/MS are imported into the same batch, then the combined quantitative method is applied simultaneously. In the method, each compound is designated a number. Compounds with the same number – those detected by both GC/MS and LC/MS – will have the best results reported. In order to avoid false positives, the report will automatically choose the higher concentration for the overlapped compounds to report. Additionally, operators can also choose them manually if needed. The Chinese name, Group name, LOD and maximum residue limit(MRL) are also set in the method.

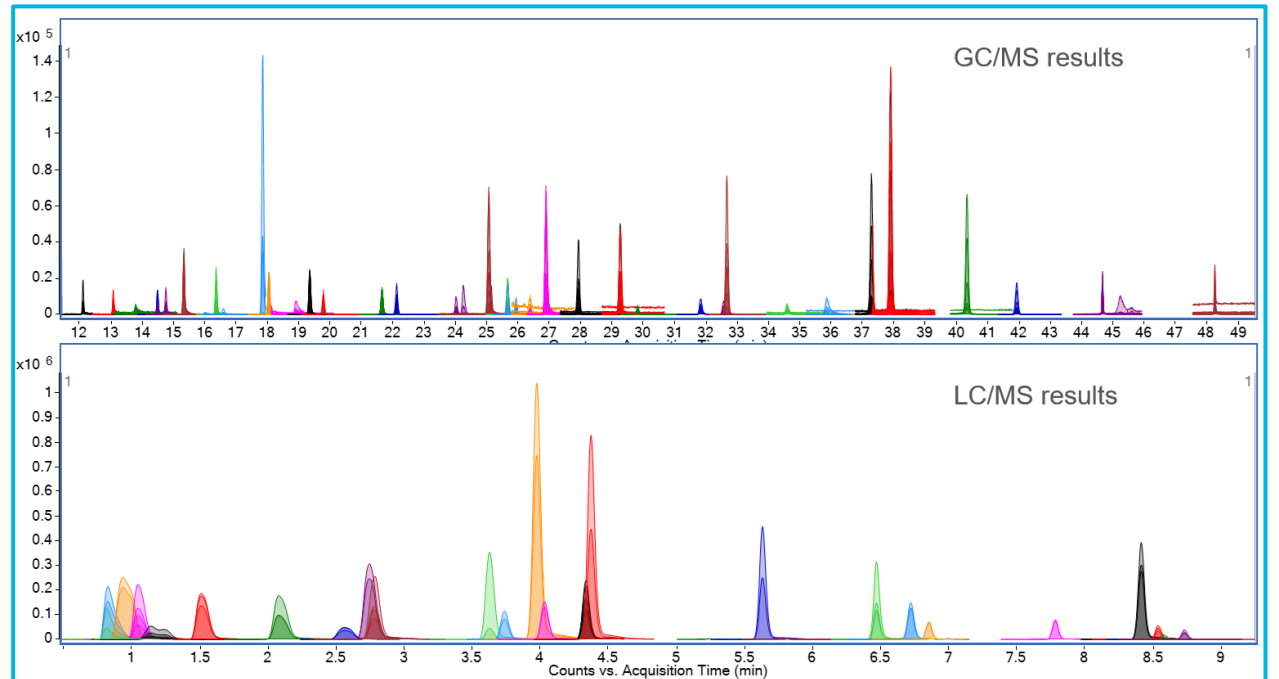


Figure 5. The MRM chromatography of 55 compounds in GC/MS and LC/MS

## Results and Discussion

In addition to the customized interface and the combined quantitative method, the customized report template is the third important part in this solution. Pesticides are separated to 33 groups according to ChP 2020, and some of them are summed by template automatically. For example, the result of BHC consists of  $\alpha$ -BHC,  $\beta$ -BHC,  $\gamma$ -BHC, and  $\delta$ -BHC. Then the result will compare with MRL recorded in the quantitative method. A "Pass" or "Fail" will be reported in the last column.

## Conclusions

An integrated solution has been successfully applied for multi-pesticides analysis on TCMs to follow Chinese Pharmacopoeia 2020 (ChP 2020). The general principles 0212 of ChP 2020 describes a procedure to monitor fifty-five pesticides of thirty-three groups in TCM matrices. Ten groups should be summed first, then compared with the MRL. The newly developed workflow of data processing offered the combined quantitative analysis in TCM extracts and use the report template to integrate the results of duplicated samples and the summation of grouped compounds, as needed. This rapid and automation solution improves the efficiency of multi-residue pesticides analysis, data consolidation and reporting.

检测报告					
样品名称	1-YG-2 + YL1-2				
农药名称	残留物	检测方法	定量限	检测结果	结果判定
1. 呋喃	1. 呋喃	GCMS	0.05 mg/kg	未检出(检测限为 0.05 mg/kg)	符合
甲基对硫磷	甲基对硫磷	GCMS	0.02 mg/kg	未检出(检测限为 0.005 mg/kg)	符合
对硫磷	对硫磷	GCMS	0.02 mg/kg	未检出(检测限为 0.005 mg/kg)	符合
久效磷	久效磷	GCMS	0.02 mg/kg	未检出(检测限为 0.005 mg/kg)	符合
Compound Group, Name, Test Method, LOQ, Results, Pass or Fail					
六六六	$\beta$ -六六六	GCMS	0.1 mg/kg	未检出(检测限为 0.02 mg/kg)	符合
	$\gamma$ -六六六	GCMS		未检出(检测限为 0.02 mg/kg)	符合
	$\delta$ -六六六	GCMS		未检出(检测限为 0.02 mg/kg)	符合
滴滴涕	4,4'-滴滴涕	GCMS	0.1 mg/kg	未检出(检测限为 0.02 mg/kg)	符合
	2,4'-滴滴涕	GCMS		未检出(检测限为 0.02 mg/kg)	符合
	4,4'-滴滴伊	GCMS		未检出(检测限为 0.02 mg/kg)	符合
杀虫脒	杀虫脒	GCMS	0.02 mg/kg	未检出(检测限为 0.005 mg/kg)	符合
	除草脒	GCMS	0.05 mg/kg	未检出(检测限为 0.02 mg/kg)	符合
	艾氏剂	GCMS	0.05 mg/kg	未检出(检测限为 0.02 mg/kg)	符合
苯线磷	苯线磷	GCMS	0.02 mg/kg	未检出(检测限为 0.005 mg/kg)	符合
	苯线磷亚砷	GCMS		未检出(检测限为 0.005 mg/kg)	符合
	苯线磷砷	GCMS		未检出(检测限为 0.005 mg/kg)	符合
地虫硫磷	地虫硫磷	GCMS	0.02 mg/kg	未检出(检测限为 0.005 mg/kg)	符合
	硫线磷	GCMS	0.02 mg/kg	未检出(检测限为 0.005 mg/kg)	符合
	磷毒磷	GCMS	0.05 mg/kg	未检出(检测限为 0.02 mg/kg)	符合
治螟磷	治螟磷	GCMS	0.02 mg/kg	未检出(检测限为 0.005 mg/kg)	符合
	特丁硫磷	GCMS	0.02 mg/kg	未检出(检测限为 0.005 mg/kg)	符合
	特丁硫磷亚砷	GCMS		未检出(检测限为 0.005 mg/kg)	符合
氯磺隆	氯磺隆	GCMS	0.05 mg/kg	未检出(检测限为 0.02 mg/kg)	符合
	胺苯磺隆	GCMS	0.05 mg/kg	未检出(检测限为 0.02 mg/kg)	符合
	甲磺隆	GCMS	0.05 mg/kg	未检出(检测限为 0.02 mg/kg)	符合
甲拌磷	甲拌磷	GCMS	0.02 mg/kg	未检出(检测限为 0.005 mg/kg)	符合
	甲拌磷亚砷	GCMS		0.24 mg/kg	不符合
	甲拌磷砷	GCMS		未检出(检测限为 0.005 mg/kg)	符合
甲基异柳磷	甲基异柳磷	GCMS	0.02 mg/kg	未检出(检测限为 0.005 mg/kg)	符合
	内吸磷	GCMS	0.02 mg/kg	未检出(检测限为 0.005 mg/kg)	符合
	克百威	GCMS	0.02 mg/kg	未检出(检测限为 0.02 mg/kg)	符合
克百威	2-羟基克百威	GCMS	0.05 mg/kg	未检出(检测限为 0.02 mg/kg)	符合
	涕灭威	GCMS	0.1 mg/kg	未检出(检测限为 0.02 mg/kg)	符合
	涕灭威亚砷	GCMS		未检出(检测限为 0.02 mg/kg)	符合
灭线磷	灭线磷	GCMS	0.02 mg/kg	未检出(检测限为 0.005 mg/kg)	符合
	氟啶脲	GCMS	0.01 mg/kg	未检出(检测限为 0.003 mg/kg)	符合
	水胺硫磷	GCMS	0.05 mg/kg	未检出(检测限为 0.02 mg/kg)	符合
硫丹	$\alpha$ -硫丹	GCMS	0.05 mg/kg	未检出(检测限为 0.013 mg/kg)	符合
	$\beta$ -硫丹	GCMS		未检出(检测限为 0.013 mg/kg)	符合
	硫丹硫酸酯	GCMS		未检出(检测限为 0.013 mg/kg)	符合
氟虫腈	氟虫腈	GCMS	0.02 mg/kg	未检出(检测限为 0.005 mg/kg)	符合
	氟虫腈亚砷	GCMS		未检出(检测限为 0.005 mg/kg)	符合
	氟虫腈砷	GCMS		未检出(检测限为 0.005 mg/kg)	符合
三氯杀螨醇	$o,p'$ -三氯杀螨醇	GCMS	0.2 mg/kg	未检出(检测限为 0.02 mg/kg)	符合
	$p,p'$ -三氯杀螨醇	GCMS		未检出(检测限为 0.02 mg/kg)	符合
	硫环磷	GCMS	0.03 mg/kg	未检出(检测限为 0.005 mg/kg)	符合
甲氨基环磷	甲氨基环磷	GCMS	0.03 mg/kg	未检出(检测限为 0.005 mg/kg)	符合

备注: 方法依据2020版《中国药典》第四部通则0212药材和饮片检定通则及2341农药残留量测定法 第五法 1. 气相色谱-串联质谱法 2. 高效液相色谱-串联质谱法

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Figure 6. The combined report of GC/MS and LC/MS results designed for Chinese users. Also, it can be other languages.