食品中殘留農藥檢驗方法－多重殘留分析方法(五)
衛生福利部食品藥物管理署
第 1031900615 號公告修正.(2014_0703)
Method of Test for Pesticide Residues in Foods-Multiresidue Analysis (5)
Taiwan Food and Drug Administration
Correction Announcement No. 1031900615.

1. 適用範圍：本檢驗方法適用於蔬果類、穀類、乾豆類、茶類、香辛植物及其他草本植物等食品中 310 項農藥多重殘留分析。

2. 檢驗方法：檢體採用 QuEChERS 方法(Quick, Easy, Cheap, Effective, Rugged, Safe) 前處理後， 以液相層析串聯質譜儀(Liquid Chromatograph/tandem Mass Spectrometer, LC/MS/MS)及氣相層析串聯質譜儀(gas chromatograph/tandem mass spectrometer, GC/MS/MS)分析之方法.

1. Scope: The test method is suitable for 310 pesticide Multi-residues analysis in foof from fruits and vegetables, cereals, dried beans, tea, spice plants and other herbs etc.

2. Test methods: the sample is pre-treated using QuEChERS method(Quick-Easy-Cheap-Effective-Rugged-Safe) and then for the method of analysis with Liquid Chromatography Tandem Mass Spectrometry LC/MS/MS) and Gas Chromatograph/Tandem mass spectrometer, GC/MS/MS).

2.1. 裝置：
2.1.1. 液相層析串聯質譜儀：
2.1.1.1. 離子源：電灑離子化(Electrospray Ionization, ESI)。
2.1.1.2. 層析管：Acquity UPLCR HSS T3，1.8um，內徑 2.1 mm × 10 cm，或同級品。
2.1.2. 氣相層析串聯質譜儀：
2.1.2.1. 離子源：電子撞擊游離(electron impact ionization, EI)。
2.1.2.2. 層析管：DB-5MS UI 毛細管，內膜厚度 0.25um，內徑 0.25mm × 30m，或同級品。
2.1.3. 攪拌均質器(Blender)。
2.1.4. 粉碎機(Grinder)。
2.1.5. 高速組織研磨振盪均質機(SPEX SamplePrep 2010 GenoGrinderR)：1000 rpm 以上，或同級品。
2.1.6. 離心機(Centrifuge)：可達 3000 × g 以上，控制溫度可達 15℃以下者。
2.1.7. 氮氣濃縮裝置(Nitrogen evaporator)。

2.1 Device:
2.1.1 Liquid Chromatograph/Tandem Mass Spectrometer:

2.1.1.1 Ion Source: Electrospray Ionization, ESI).
2.1.1.2 Chromatography Column: Acquity UPLCR HSS T3,1.8um, an inner diameter of 2.1 mm × 10 cm, or products with same quality.
2.1.2 Gas Chromatography Tandem Mass Spectrometry:
2.1.2.1 Ion Source: Electron Impact Ionization, EI.
2.1.2.2 Chromatography Column: DB-5MS UI capillary, inner film layer thickness 0.25um, 0.25 mm ID × 30 m, or products with same quality..
2.1.3. Stirring homogenizer (Blender).
2.1.4 Grinder.
2.1.5 High-speed oscillation grinding tissue homogenizer (SPEX SamplePrep 2010 GenoGrinderR): 1000 rpm or more, or products with same quality.
2.1.6 Centrifuger: Up to 3000 × g or more, control the temperature up to those below 15℃.
2.1.7. Nitrogen evaporator for concentration.

2.2. 試藥：
冰醋酸、甲酸及醋酸銨均採試藥特級；正己烷及丙酮均採用殘留量級；
乙腈及甲醇均採液相層析級。無水醋酸鈉、無水硫酸鎂、primary secondary amine (PSA) 、octadecysilane, end-capped (C18 EC) 及 graphitized carbon black (GCB)均採用分析級；去離子水(比電阻於 25℃可達 18M ohm・cm 以上)；
農藥對照用標準品 3-酮加保扶(3-keto carbofuran)等 310 項(品項見表一、表二及表三)；
磷酸三苯酯(triphenylphosphate,TPP)內部標準品。

2.2 Reagent:
Acetic acid,
Formic acid and ammonium acetate are of GR/Guaranteed Reagent Grade;
Hexane and Acetone are of residual grade;
Acetonitrile and methanol are of Liquid Chromatography Grade.
Anhydrous sodium acetate, Anhydrous magnesium sμLfate, Primary secondary amine(PSA), Octadecysilane, end-capped(C18 EC) and Graphitized Carbon Black(GCB) are of analytical grade;
Deionized water (resistivity at 25℃ Up to 18 M ohm・cm or above);
310 pesticide Reference Standards as 3-keto carbofuran (items shown in Table I. Table 2 and Table 3);
Triphenyl phosphate (triphenylphosphate, TPP) internal standard.

2.3. 器具及材料：
2.3.1. 離心管：15 mL 及 50 mL，PP 材質。
2.3.2. 濾膜：孔徑 0.22um，PVDF 材質。
2.3.3. 容量瓶：25 mL 及 50 mL，褐色。
2.3.4. 陶瓷均質石(Ceramic homogenizer)(註 1)：採用 Bond Elut QuEChERS

P/N 5982-9313，或同級品。
2.3.5. 萃取用粉劑(註 2)：含無水硫酸鎂 4 g 及無水醋酸鈉 1 g。
2.3.6. 淨化用離心管 I (註 2)：含 PSA 300 mg 及無水硫酸鎂 900 mg，檢液負荷量 6 mL，適用於水分含量高之蔬果類檢體。

2.3.7. 淨化用離心管 II (註 2)：含 PSA 300 mg、C18EC 300 mg 及無水硫酸鎂 900 mg，檢液負荷量 6 mL，適用於蠟、油脂及醣類含量高之穀類檢體。

2.3.8. 淨化用離心管 III (註 2)：含 PSA 450 mg、無水硫酸鎂 900 mg、C18 EC 300 mg 及 GCB 50 mg，檢液負荷量 6 mL，適用於高色素含量及茶葉類檢體。
註 1：陶瓷均質石可視檢體黏稠度自行評估使用。
註 2：可依需求自行評估使用市售各種萃取及淨化用組合套組。

2.3 equipment and materials:
2.3.1 Centrifuge tubes: 15 mL and 50 mL, PP material.
2.3.2 Membrane: Pore size 0.22um, PVDF material.
2.3.3 Volumetric flasks: 25 mL and 50 mL, amber.
2.3.4 Homogeneous ceramic stone (Ceramic homogenizer) (Note 1): Using Bond Elut QuEChERS P/N 5982-9313, or products with same quality.
2.3.5 Extracting powder (Note 2): with Anhydrous magnesium sµLfate (MgSO4) 4 g, and Anhydrous sodium acetate (NaOAc) 1 g.
2.3.6 Purification centrifuge tube TypeI (Note 2): with PSA 300 mg and Anhydrous magnesium sµLfate (MgSO4) 900 mg,
extracting solution loading capacity 6 mL, suitable for fruits and vegetables samples with high water content.

2.3.7 Purification centrifuge tube TypeII (Note 2): with PSA 300 mg, C18EC 300 mg and Anhydrous magnesium sµLfate (MgSO4) 900 mg,
extracting solution loading capacity 6 mL, suitable for cereals samples with high wax, fat and sugar content.

2.3.8 Purifying centrifuge tube TypeIII (Note 2): with PSA 450 mg, Anhydrous magnesium sµLfate (MgSO4)900 mg, C18 EC
300 mg and GCB 50 mg, extracting solution loading capacity 6 mL, suitable for samples with high pigment content and tea analogues.

Note 1: The Ceramic homogenizer coµLd be arbitrarily used according to the user's assessment of the viscosity of sample.
Note 2: According to their demands, the users coµLd make their assessment for appling the various extraction and purification kits commercially available.

2.4. 移動相溶液之調製：

2.4.1. 移動相溶液 A：

取甲醇 50 mL 與去離子水 450 mL 混合後，加入醋酸銨 0.19 g，溶解並混合均勻，以濾膜過濾，取濾液供作移動相溶液 A。

2.4.2. 移動相溶液 B：

取甲醇 450 mL 與去離子水 50 mL 混合後，加入醋酸銨 0.19 g，溶解並混合均勻，以濾膜過濾，取濾液供作移動相溶液 B。

2.4 Mobile Phase Solution Preparations:

2.4.1 Mobile Phase Solution A:

Take 50 mL of Methanol and mix with 450 mL of deionized water, add and to dissolve Ammonium Acetate 0.19g
and mixed completely, filter with membrane. The filtrate is for mobile phase A.

2.4.2 Mobile Phase Solution B:

Take 450 mL of Methanol and mix with 50 mL of deionized water, add and to dissolve Ammonium Acetate 0.19g
and mixed completely, filter with membrane. The filtrate is for mobile phase B.

2.5. 內部標準溶液之配製：

取磷酸三苯酯內部標準品約 40 mg，精確稱定，以甲醇溶解並定容至
50 mL，作為內部標準原液，於-18 度 C 避光貯存備用。

2.5.1. 取適量內部標準原液以甲醇稀釋至 75 μg/mL，供作 2.8.節檢液調製使用之內部標準溶液。

2.5.2. 取適量內部標準原液以甲醇稀釋至 7.5 μg/mL，供作 2.9.1.節 LC/MS/MS 分析用內部標準溶液。

2.5.3. 取適量內部標準原液以丙酮稀釋至 7.5 μg/mL，供作 2.9.2.節 GC/MS/MS 分析用內部標準溶液。

2.5 Internal Standard solution preparation:

Take Triphenylphosphate(TPP) internal standard about 40 mg, accurately weighed,
dissolved in methanol and dilute to 50 mL, as an Internal Standard Stock Solution, stored at -18℃ in dark for use.

2.5.1 Take appropriate amount of Internal Standard Stock Solution and then dilute with Methanol to 75μg/mL, that will be the Internal Standard Solution used for the Test Solution Preparation in section 2.8.

2.5.2 Take appropriate amount of Internal Standard Stock Solution and then dilute with Methanol to 7.5μg/mL,
that will be the Internal Standard Solution used for LC/MS/MS analysis in section 2.9.1

2.5.3 Take appropriate amount of Internal Standard Stock Solution and then dilute with Acetone to 7.5μg/mL, that will be the Internal Standard Solution used for GC/MS/MS analysis in section 2.9.2

2.6. 試劑之調製：
2.6.1. 含 1%醋酸之乙腈溶液：
取冰醋酸 10 mL 與乙腈 990 mL 混合均勻。
2.6.2. 含 5%甲酸之乙腈溶液：
取甲酸 5 mL 與乙腈 95 mL 混合均勻。
2.6.3. 丙酮：正己烷(1:1, v/v)溶液：
取丙酮與正己烷以 1：1 (v/v)比例混勻。

2.6 Reagents Preparation:
2.6.1 1% Acetic Acid(HOAc) in Acetonitrile(MeCN) solution:
Take 10 mL of Glacial Acetic Acid(HOAc) and mixed with 990 mL of Acetonitrile(MeCN).
2.6.2 5% Formic Acid in Acetonitrile(MeCN) solution:
Take 5 mL Formic acid and mixed with 95 mL of Acetonitrile(MeCN).
2.6.3 Acetone: Hexane(1:1,v/v)solution:
Take Acetone and n-Hexane mixed to 1:1 (v/v)ratio.

2.7. 標準溶液之配製：
2.7.1 取農藥對照用標準品各約 25 mg，精確稱定，以乙腈溶解並定容至 25 mL，作為標準原液，於-18℃ 避光貯存備用。取適量標準原液以甲醇稀釋至 1 μg/mL，供作 2.9.1.節 LC/MS/MS 分析用標準溶液。
2.7.2 取農藥對照用標準品各約 25 mg，精確稱定，以丙酮或正己烷溶解並定容至 25 mL，作為標準原液，於-18℃ 避光貯存備用。取適量標準原液以丙酮：正己烷(1:1, v/v)溶液稀釋至 1 μg/mL，供作 2.9.2.節 GC/MS/MS 分析用標準溶液。

2.7 Standard Solution Preparation:
2.7.1 Take about 25 mg pesticide reference standards, accurately weighed and dissolved in Acetonitrile(MeCN) to 25 mL
as the Standard Stock solution, store at -18℃ in dark for use.
Take appropriate amount of the Standard Stock solution and diluted with Methanol to 1 μg/mL,
as the Standard solution used for LC/MS/MS analysis in section 2.9.1.
2.7.2 Take about 25 mg pesticide reference standards, accurately weighed and dissolved in Acetone or Hexane to 25 mL
as the Standard Stock solution, store at -18℃ in dark for use.
Take appropriate amount of the Standard Stock solution and diluted with Acetone: Hexane (1:1, v/v) solution

to 1 μg/mL,
as the Standard solution used for GC/MS/MS analysis in section 2.9.2.

2.8. 檢液之調製：
2.8. Test Solution Preparation:
2.8.1. 蔬果類、香辛植物及其他草本植物(鮮食)：
取均質之檢體約 10 g，精確稱定，置於離心管中，冷凍後加入含 1%醋酸之乙腈溶液 10 mL 及 75μg/mL 內部標準溶液 10 μL，
再依序加入陶瓷均質石 1 顆及萃取用粉劑，蓋上離心管蓋，隨即激烈振盪數次，防止鹽類結塊，再以高速組織研磨振盪均質機於 1000 rpm 振盪或以手激烈振盪 1 分鐘後，於 15℃，3000 × g 離心 1 分鐘。

取上清液 6 mL，置於淨化用離心管 I，以高速組織研磨振盪均質機以 1000 rpm 振盪或以手激烈振盪 1 分鐘後，於 15℃，3000 × g 離心 2 分鐘。

取上清液 1 mL，以氮氣吹至剛乾，殘留物以適量甲醇溶解，加入含 5%甲酸之乙腈溶液 10 μL，使體積為 1 mL，混合均勻，以濾膜過濾，供作檢液 I，以 LC/MS/MS 分析。

另取上清液 1 mL，以氮氣吹至剛乾，殘留物以適量丙酮：正己烷(1:1, v/v)溶液溶解，加入含 5%甲酸之乙腈溶液 10 μL，使體積為 1 mL，混合均勻，以濾膜過濾後，供作檢液 II，以 GC/MS/MS 分析。

2.8.1 Fruits and vegetables, spice plants and other herbs(fresh meal):
Accurately weigh about 10g homogeneous sample into a centrifuge tube,
frozen and then add 1% Acetic Acid(HOAc) in Acetonitrile(MeCN) solution 10 mL,
and 75μg/mL Internal Standard Solution 10μL, and then sequentially add one Ceramic homogenizer and extracting powder.
Close the cap and immediately followed with intense oscillations for times to prevent from caking of the salt, and then vigorously shaken by hand or by high speed tissue grinding and oscillating homogenizer in 1000rpm for 1 minute,
and centrifuged at 3000 × g for 1 min at 15℃.

Transfer the supernatant 6 mL to a Purification centrifuge tube(TypeI)
and then vigorously shaken by hand or by high speed tissue grinding and oscillating homogenizer in 1000rpm for 1 minute,
and centrifuged at 3000 × g for 2 min at 15℃.

Transfer the supernatant 1 mL to a scaled vials and evaporated with nitrogen to just dried,

and the residue was dissolved in an appropriate amount of Methanol, with 5% Formic Acid in Acetonitrile(MeCN) solution 10 µL added,
to get 1mL volume. Mix homogenously and filter with membrane to get the Test Solution I, for the LC/MS/MS analysis.

Transfer another supernatant 1 mL to a scaled vials and evaporated with nitrogen to just dried,
and the residue was dissolved in an appropriate amount of Acetone: Hexane (1: 1, v/v) solution, with 5% Formic Acid in Acetonitrile(MeCN) solution 10 µL added,
to get 1mL volume. Mix homogenously and filter with membrane to get the Test Solution II, for the GC/MS/MS analysis.

2.8.2. 穀類及乾豆類：
取磨粉後之檢體約 5 g，精確稱定，置於離心管中，加入冷藏預冷之去離子水 10 mL，
靜置 20 分鐘，加入含 1%醋酸之乙腈溶液 10 mL 及 75µg/mL 內部標準溶液 10 µL，
再依序加入陶瓷均質石 1 顆及萃取用粉劑，蓋上離心管蓋，隨即激烈振盪數次，防止鹽類結塊，
再以高速組織研磨振盪均質機於 1000 rpm 振盪或以手激烈振盪 1 分鐘後，
於 15℃，3000 × g 離心 1 分鐘。取上清液 6 mL，置於淨化用離心管 II，
以高速組織研磨振盪均質機以 1000 rpm 振盪或以手激烈振盪 1 分鐘後，於 15℃，3000 × g 離心 2 分鐘。

取上清液 1 mL，以氮氣吹至剛乾，殘留物以適量甲醇溶解，加入含 5%甲酸之乙腈溶液 10 µL，
使體積為 1 mL，混合均勻，以濾膜過濾後，供作檢液 I，以 LC/MS/MS 分析。

另取上清液 1 mL，以氮氣吹至剛乾，殘留物以適量丙酮：正己烷(1:1, v/v)溶液溶解，加入含 5%甲酸之乙腈溶液 10µL，
使體積為 1 mL，混合均勻，以濾膜過濾後，供作檢液 II，以 GC/MS/MS 分析。

2.8.2 Cereals and dried beans:
Accurately weigh about 5g homogeneous grinded sample into a centrifuge tube,
add pre-cooling deionized water 10 mL and stand for 20 minutes,
and then add 1% Acetic Acid(HOAc) in Acetonitrile(MeCN) solution 10 mL.
The following procedures are the sample as that in the section 2.8.1,
except that the Purification centrifuge tube(TypeII) is used instead.

2.8.3. 茶類、香辛植物及其他草本植物(乾燥)：
取磨粉後之檢體約 2 g，精確稱定，置於離心管中，加入冷藏預冷之去離子水 10 mL，靜置 20 分鐘，
加入含 1%醋酸之乙腈溶液 10 mL 及 75µg/mL 內部標準溶液 10 µL，再依序加入陶瓷均質石 1 顆及萃取用粉劑，

蓋上離心管蓋，隨即激烈振盪數次，防止鹽類結塊，再以高速組織研磨振盪均質機於 1000 rpm 振盪或以手激烈振盪 1 分鐘後，
於 15℃，3000 × g 離心 1 分鐘。取上清液 6 mL，置於淨化用離心管 III，
以高速組織研磨振盪均質機以 1000 rpm 振盪或以手激烈振盪 1 分鐘後，於 15℃，3000 × g 離心 2 分鐘。

取上清液 1 mL，以氮氣吹至剛乾，殘留物以適量甲醇溶解，加入含 5%甲酸之乙腈溶液 10 µL，使體積為 1 mL，
混合均勻，以濾膜過濾後，供作檢液 I，以 LC/MS/MS 分析。

另取上清液 1 mL，以氮氣吹至剛乾，殘留物以適量丙酮：正己烷(1:1, v/v)溶液溶解，加入含 5%甲酸之乙腈溶液 10µL，
使體積為 1 mL，混合均勻，以濾膜過濾後，供作檢液 II，以 GC/MS/MS 分析。

2.8.3 Tea, spice plants and other herbs (dried):
Accurately weigh about 2g homogeneous grinded sample into a centrifuge tube,
add pre-cooling deionized water 10 mL and stand for 20 minutes,
and then add 1% Acetic Acid(HOAc) in Acetonitrile(MeCN) solution 10 mL.
The following procedures are the sample as that in the section 2.8.1,
except that the Purification centrifuge tube(TypeIII) is used instead.

2.9. 基質匹配檢量線製作
2.9 Matrix-matched calibration curve preparation

2.9.1. LC/MS/MS：
取空白檢體，依 2.8.節調製未添加內部標準品之淨化後上清液，分別量取 1 mL，以氮氣吹至剛乾，
分別加入適量甲醇、1 μg/mL(註 3)標準溶液 2～200 μL、7.5 μg/mL 內部標準溶液 10 μL
及含 5%甲酸之乙腈溶液 10µL，使體積為 1 mL，混合均勻。
依下列條件進行分析，就各農藥與內部標準品波峰面積比，與對應之各農藥濃度，製作 0.002～0.2 μg/mL (芬普尼為 0.0004～0.04 μg/mL)之基質匹配檢量線。

2.9.1 LC/MS/MS:
Take a blank sample and process according to the 2.8.Test Solution Preparation steps
without the addition of Internal Standard solution to produce the supernatant after SPE purification.
For each standard levels, transfer 1mL supernatant to scaled vial and evaporate with nitrogen to just dried,
and the residue was dissolved in an appropriate amount of Methanol, with 1µg/mL (Note 3) Standard Solution 2 ~ 200µL espectively,
7.5µg/mL of Internal Standard solution 10µL, and 5% Formic Acid in Acetonitrile(MeCN) solution 10µL added,

to get 1mL volume. mix homogenously.

The prepared Matrix-matched calibration standards are analyzed by the following conditions.
The Matrix-matched calibration curves are produced as a plot of (peak area/internal standard peak area)ratio of each pesticides
versus the corresponding concentration of each pesticides in the 0.002～0.2 μg/mL range (Fipronil is 0.0004 ~ 0.04 μg/mL).

液相層析串聯質譜分析測定條件(註 4)：
層析管：Acquity UPLCR HSS T3，1.8um，內徑 2.1 mm ×10 cm。
移動相溶液：A 液與 B 液以下列條件進行梯度分析
移動相流速：0.3 mL/min。
毛細管電壓(Capillary voltage)：
電灑離子化正離子(ESI＋)採用 3.5 kV，
電灑離子化負離子(ESI－)採用 1.6 kV。
離子源溫度(Ion source temperature)：150℃。
溶媒揮散溫度(Desolvation temperature)：450℃。
進樣錐氣體流速(Cone gas flow)：30 L/hr。
溶媒揮散流速(Desolvation flow)：900 L/hr。
偵測模式：多重反應偵測(Multiple reaction monitoring, MRM)。
偵測離子對、進樣錐電壓(cone voltage)與碰撞能量
(collision energy)如表一及表二。

Liquid Chromatography Tandem Mass Spectrometry measurement conditions (Note 4):
Chromatography Column: Acquity UPLCR HSS T3, 1.8um, 2.1mm(ID) × 10 cm.
Mobile phase solution: Analysis with the following Gradient conditions for A abd B solution
Mobile phase flow rate: 0.3 mL/min.
Capillary voltage :
Positive ion electrospray ionization (ESI +) using 3.5 kV,
Negative ion electrospray ionization (ESI-) using 1.6 kV.
Ion source temperature (Ion source temperature): 150℃.
Solvent volatilization temperature (Desolvation temperature): 450℃.
Injection cone gas flow rate (Cone gas flow): 30 L/hr.
Flow rate of vaporized solvent (Desolvation flow): 900 L/hr.
Detection mode: Multiple reaction monitoring(MRM).
Detective Measured ion pair, injection cone voltage (cone voltage) and collision energy
(collision energy) as shown in Table 1 and Table 2.

2.9.2. GC/MS/MS：
取空白檢體，依 2.8.節調製未添加內部標準品之 GC/MS/MS 檢液原液，分別量取 1 mL，
以氮氣吹至剛乾，分別加入適量丙酮：正己烷(1:1, v/v)溶液、1 μg/mL 標準溶液 4～500 μL、7.5 μg/mL 內部標準溶液 10 μL
及含 5%甲酸之乙腈溶液 10μL，使體積為 1 mL，混合均勻。
依下列條件進行分析，就各農藥與內部標準品波峰面積比，與對應之各農藥濃度，製作 0.004～0.5 μg/mL 之基質匹配檢量線。

2.9.2 GC/MS/MS:
Take a blank sample and process according to the 2.8.Test Solution Preparation steps
without the addition of Internal Standard solution to produce the supernatant after SPE purification.
For each standard levels, transfer 1mL supernatant to scaled vial and evaporate with nitrogen to just dried, and the residue was dissolved in an appropriate amount of Acetone: Hexane(1:1, v/v)solution, with 1μg/mL Standard Solution 4 ~ 500μL espectively,
7.5μg/mL of Internal Standard solution 10μL, and 5% Formic Acid in Acetonitrile(MeCN) solution 10μL added, to get 1mL volume. mix homogenously.

The prepared Matrix-matched calibration standards are analyzed by the following conditions.
The Matrix-matched calibration curves are produced as a plot of (peak area/internal standard peak area)ratio of each pesticides
versus the corresponding concentration of each pesticides in the 0.004～0.5 μg/mL range.

氣相層析串聯質譜分析測定條件(註 4)：
層析管：DB-5MS UI 毛細管，內膜厚度 0.25um，內徑 0.25 mm × 30 m。
層析管溫度：初溫：60℃，1 min；
升溫速率：40℃/min；
中溫：170℃；
升溫速率：10℃/min；
終溫：310℃，2.25 min。
移動相流速：氦氣，1 mL/min。
注入器溫度(Injector temperature)：280℃。
注入模式：不分流。
離子化模式：電子撞擊(electron impact)，70 eV。
離子源溫度：300℃。
偵測模式：多重反應偵測，偵測離子對及碰撞能量如表三。
註 3：芬普尼之基質匹配檢量線製作時，選擇適當之標準溶液添加。
註 4：上述測定條件分析不適時，可依所使用之儀器，設定適合之測定條件。

Gas Chromatography Tandem Mass Spectrometry measurement conditions (Note 4):
Chromatography Column: DB-5MS UI capillary, inner film layer thickness 0.25um, 0.25 mm ID × 30 m.
Chromatography Column temperature: initial temperature: 60℃, 1 min;
Heating rate: 40℃/min;
Temperature: 170℃;
Heating rate: 10℃/min;
Final temperature: 310℃, 2.25 min.
Mobile phase flow rate: Helium, 1 mL/min.
Injector temperature : 280℃.
Injection mode: splitless.
Ionization modes: electron impact, 70 eV.
Ion source temperature: 300℃.
Detection mode: Multiple reaction monitoring, MRM, as detection ion pairs and collision energy shown in Table III.
Note 3: Select appropriate standard solution spikings for producing the Matrix-matched calibration curve of Fipronil.
Note 4: Setting suitable measurement conditions according to the instrument used
if the measurement conditions listed above are not suitable.

2.10. 鑑別試驗及含量測定：
2.10 Identification Test and Quantitation:

2.10.1. LC/MS/MS：
精確量取檢液及標準溶液各 10 μL，分別注入液相層析串聯質譜儀中，
依 2.9.1.節條件進行分析，就檢液與標準溶液所得波峰之滯留時間及多重反應偵測相對離子強度(註 5)鑑別之，
並依下列計算式，求出檢體中各農藥之含量(ppm) :
檢體中各農藥之含量(ppm) = C x V / M

C：由各農藥之基質匹配檢量線求得檢液中各農藥之濃度(μg/mL)
V：萃取檢體之含 1%醋酸之乙腈溶液體積(10 mL)
M：取樣分析檢體之重量(g)

2.10.1 LC/MS/MS: ,
Exactly measure 10μL of Test solution and Standard solution and inject into the Liquid Chromatography Tandem Mass spectrometry respectively.
Perform analysis in accordance with the measurement conditions from section 2.9.1,
Identifications are based on the peak retention time and the Multiple reaction monitoring(MRM) relative ion

strength
of the Test solution and Standard solution(Note 5), and calculated in accordance with the following formula, to obtain the content of each pesticides(ppm) of samples :
The content of each pesticide in a sample (ppm) = C x V / M

C: Each pesticides concentrations(μg/mL) of Test solution obtained/indexed from corresponding Matrix-Matched calibration curve for each pesticides.
V: Volume of extracted sample(10 mL) with 1% Acetic Acid(HOAc) in Acetonitrile(MeCN) solution.
M: Sampled weight of the sample for analysis(g)

2.10.2. GC/MS/MS：
精確量取檢液及標準溶液各 1 μL，分別注入氣相層析串聯質譜儀中，
依 2.9.2.節條件進行分析，就檢液與標準溶液所得波峰之滯留時間及多重反應偵測相對離子強度(註 5)鑑別之，
並依下列計算式，求出檢體中各農藥之含量(ppm)：
檢體中各農藥之含量(ppm) = C x V / M

C：由各農藥之基質匹配檢量線求得檢液中各農藥之濃度(μg/mL)
V：萃取檢體之含 1%醋酸之乙腈溶液體積(10 mL)
M：取樣分析檢體之重量(g)

註 5：相對離子強度由定性離子對與定量離子對之波峰面積相除而得(<=100%)，容許範圍如下：
相對離子強度(%) 容許範圍(%)

2.10.2 GC/MS/MS:
Exactly measure 10μL of Test solution and Standard solution and inject into the Gas Chromatography Tandem Mass spectrometry respectively.
Perform analysis in accordance with the measurement conditions from section 2.9.2,
Identifications are based on the peak retention time and the Multiple reaction monitoring(MRM) relative ion strength
of the Test solution and Standard solution(Note 5), and calculated in accordance with the following formula, to obtain the content of each pesticides(ppm) of samples :
The content of each pesticide in a sample (ppm) = C x V / M

C: Each pesticides concentrations(μg/mL) of Test solution obtained/indexed from corresponding Matrix-Matched calibration curve for each pesticides.
V: Volume of extracted sample(10 mL) with 1% Acetic Acid(HOAc) in Acetonitrile(MeCN) solution.
M: Sampled weight of the sample for analysis(g)

Note 5: Relative Ionic Strength is obtained from the from the ratio of the peak areas from the qualitative and quantitative ion pair (<=100%),
acceptable range is as follows:
Relative Ionic strength(%) Acceptable range(%)

1. 本檢驗方法之定量極限如表一、表二及表三。
2. 本檢驗方法所列品項可依需求評估以 GC/MS/MS 或 LC/MS/MS 分析。
3. 本檢驗方法不適用於茶葉基質中免扶克及派滅淨之檢驗。
4. 食品中有影響檢驗結果之物質時，應自行探討。

1. The Quantitation Limits of this test method are shown in Table I, Table 2 and Table 3.
2. The items listed in this test method could be assessed for the demands to the analysis with GC/MS/MS or LC/MS/MS.
3. The method does not apply to analysis of Benfuracarb and Pymetrozine in Tea matrix.
4. If there are intefering substances in the food that affect the resdult of analysis,
   the practioner should check and study the cause and solution.

表一、3-酮加保扶等 144 項農藥及內部標準品之多重反應偵測模式參數及定量極限(LC/MS/MS 正離子模式)
TableI, The Multiple reaction monitoring(MRM) parameters and Quantitative detection limits(by LC/MS/MS in positive ion mode) for 144 pesticide items as 3-keto carbofuran and Internal Standard.

項次 Item
分析物 定量離子對 定性離子對 定量極限(ppm)
前驅離子(m/z)＞
產物離子(m/z)
進樣錐電壓(V)
Analyte, Quantitation ion pair, Qualitative ion pair, Quantitation limits(ppm)
English name Chinese name
Precursor ion (m/z)>
The Product ion (m/z)
Injection Cone voltage (V)
碰撞能量(eV)
Collision energy(eV)

蔬果類 a
穀類 b

茶類 c
Fruits and vegetables
Cereal
Tea

表二、本達隆等 6 項農藥之多重反應偵測模式參數及定量極限(LC/MS/MS 負離子模式)
TableII, The Multiple reaction monitoring(MRM) parameters and Quantitative detection limits(by LC/MS/MS in negative ion mode) for 6 pesticide items as Talon.

表三、Acetochlor 等 160 項農藥及內部標準品之多重反應偵測模式參數及定量極限(GC/MS/MS)
TableIII, The Multiple reaction monitoring(MRM) parameters and Quantitative detection limits(by LC/MS/MS in positive ion mode) for 160 pesticide items as Acetochlor and Internal Standard.

a 適用於蔬果類、香辛植物及其他草本植物(鮮食)。
b 適用於穀類及乾豆類。
c 適用於茶類、香辛植物及其他草本植物(乾燥)。

a Suitable for vegetables and fruit, spice plants and other herbs(fresh meal).
b Applies to cereals and dried beans.
c Suitable for tea, spice plants and other herbs(dried).