

Call for AOAC INTERNATIONAL Collaborator

-- The collaborative study protocol of "High Throughput GC-MS(GC-MS/MS) and LC-MS-MS Method for Multi-classes and Multi-kinds of Residual Pesticides in Tea"

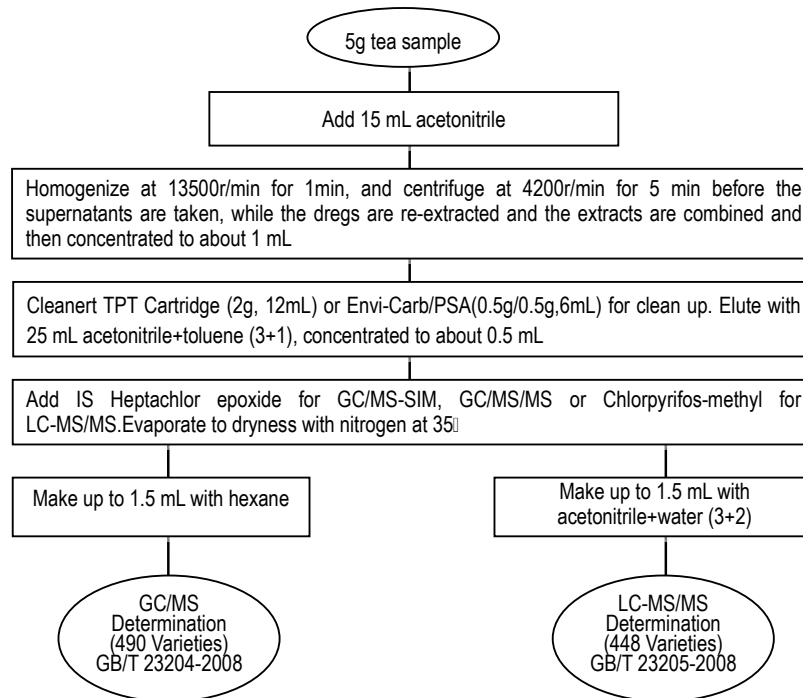
1. Principle of the method

Test samples are extracted with acetonitrile using homogenizer, the extracts are cleaned up with Cleanert TPT cartridge or Envi-Carb/PSA cartridges. The pesticides are eluted with acetonitrile-toluene (3:1,V/V), after concentration, dryness and dissolution, the samples are analyzed by GC-MS, GC-MS/MS or LC-MS-MS, quantified with matrix-matched internal standard calibration curve.

2. Application of the method

The method is applicable to determination of multi-classes and multi-Kinds of 668 residue pesticides and chemical pollutants in green tea, black tea, puer tea and wulong tea by GC-MS, GC-MS/MS & LC-MS/MS. The limits of detection (LOD) for 490 pesticides determined by GC-MS were between 1.0-500 µg/kg, and for 448 pesticides determined by LC-MS/MS were between 0.03-4820 µg/kg. There are 482 pesticides with LOD ≤ 100 µg/kg for GC-MS method, accounting for 98% of the pesticides tested and there are 417 pesticides for LC-MS/MS, accounting for 93% of the pesticides analyzed; there are 264 pesticides with LOD ≤ 10 µg/kg for GC-MS method, accounting for 54% of the pesticides tested, 325 pesticides for LC-MS/MS method, making up 73% of the pesticides analyzed.

3. GC-MS (GC-MS/MS) & LC-MS/MS analysis program



GC-MS (GC-MS/MS) & LC-MS/MS Analysis program

4. Equipment and materials

- 4.1 GC/MS system.—Agilent 7890A/5975C or equivalent.
- 4.2 GC-MS/MS system.—Agilent 7890A/7000B or equivalent.
- 4.3 LC-MS/MS system.—Agilent 1200/6430 or equivalent.
- 4.4 Capillary column.— DB-1701, 30 m×0.25 mm×0.25 µm (Agilent J&W Scientific) or equivalent.

- 4.5 Column.—Zorbax SB-C18, 2.1×100 mm, 3.5 μm (Agilent Technologies) or equivalent.
- 4.6 Homogenizer.—Rotational speed higher than 13500 r/min, T-25B or equivalent.
- 4.7 Rotary evaporator.—Buchi EL131 or equivalent.
- 4.8 Centrifuge. —Centrifugal speed higher than 4200 r/min, Z320 or equivalent.
- 4.9 Nitrogen evaporator.—EVAP 112 or equivalent.
- 4.10 Visiprep 5-port flask vacuum manifold.—RS-SUPELCO 57101-U or equivalent.
- 4.11 Balance: Capable of accurately measuring weights from 0.05 to 100g within ±0.01 g.
- 4.12 SPE cartridges. —Cleanert–TPT(2000mg, 12mL, Agela, China) or Envi-Carb/PSA (500mg/500mg, 6mL, Supelco,USA)or equivalent.

5. Testing materials for collaborator shipped by Study Director

- 5.1 Three standard stock solutions: pesticide mixed standard stock solutions, internal standard stock solutions and quality control standard stock solutions, used for quantification of collaborator samples, collaborators practice on their own and checking the instrumental sensitivity and stability.
- 5.2 Standard fortified solutions: used for preparation of fortified samples.
- 5.3 Blank samples of green tea and wulong tea: used respectively for establishing 5 point matrix-matched internal standard calibration curve, and the fortified test for collaborator.
- 5.4 Pesticide aged samples: used for practice examination for pre-collaborative study and used for the official collaborative study.

6. An outline of the collaborative study protocol

6.1 Analytical matrixes: green tea and wulong tea.

6.2 Analytical techniques: ①GC-MS or GC-MS/MS 和②LC-MS/MS (alternative or both).

6.2 Target pesticides: 20 representative pesticides were selected from the 490 pesticides determined by GC-MS (GC-MS/MS) and 448 pesticides determined by LC-MS/MS. See the table as follow:

GC-MS (GC-MS/MS)		LC-MS/MS	
1	2,4'-DDE	11	pirimiphos-methyl
2	benalaxyl	12	bifenthrin
3	bromophos-ethyl	13	propyzamide
4	bromopropylate	14	pyrimethanil
5	chlorfenapyr	15	quinoxifen
6	diflufenican	16	tebufenpyrad
7	dimethenamid	17	tefluthrin
8	fenchlorphos	18	tolclofos-methyl
9	picoxystrobin	19	trifluralin
10	pirimicarb	20	vinclozolin
1	acetochlor	11	imidacloprid
2	benalaxyl	12	indoxacarb
3	bensulide	13	kresoxim-methyl
4	butralin	14	phenothrin
5	chlorpyrifos	15	picoxystrobin
6	clomazone	16	pirimiphos methyl
7	diazinon	17	propoxur
8	ethoprophos	18	tebufenpyrad
9	fenazaquin	19	triadimefon
10	flutolanil	20	trifloxystrobin

6.3 Examination samples for pre-collaborative study: 6.

6.4 The official collaborative study samples: 16.

7. Content of the pre-collaborative study

- 7.1 Preparing the three kinds of standard working solutions (pesticides mixed standard working solutions, internal standard working solutions and quality control standard working solutions).
- 7.2 Establishing GC-MS (GC-MS/MS) and LC-MS/MS analytical procedures.
- 7.3 Checking instrumental sensitivity and stability using quality control standard working solutions.
- 7.4 Establishing 5 point matrix-matched internal standard calibration curve.
- 7.5 Collaborators practice on their own: the criteria of practice test: $R^2 > 0.995$, RSD:70-120%, RSD < 15% (n=5).
- 7.6 Examination for pre-collaborative study: 3 fortified samples, 3 aged samples.

8. Content of the official collaborative study

- 8.1 Checking instrumental sensitivity and stability using quality control standard working solutions.
- 8.2 Establishing 5 point matrix-matched internal standard calibration curve.
- 8.3 Determine fortified sample: 10 (5 each for green tea and wolong tea).
- 8.4 Determine aged sample: 6 (3 each for green tea and wolong tea).

9. Time frame for completion of the collaborative study

This study was expected to begin on Mar, 2011. According to the stability of the sample and the standard solution, the collaborative study should be finished in 3 months after the collaborative samples and materials were sent. If you are interested in this collaborative study and want to participate in it, please fill the application form (See attachment) and submit to us as soon as possible. If you need any additional details of this study, please contact the AOAC Study Director Dr. Guo-Fang Pang at any time.

10. Contact information

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Registration Form on AOAC INTERNATIONAL Collaborative Study

Title of method (copy attached): High-Throughput Analytical Method for Determination of multi-Classes and multi-Kinds of 668 Residue Pesticides and Chemical Pollutants in Tea by GC-MS, GC-MS/MS and LC-MS/MS

1. Our laboratory is willing to participate in the international collaborative study on AOAC Official method.

- | | | | |
|--------------------------|----------|------------------------------|--|
| <input type="checkbox"/> | GC-MS | YES <input type="checkbox"/> | NO <input type="checkbox"/> (tick the appropriate box) |
| <input type="checkbox"/> | GC-MS/MS | YES <input type="checkbox"/> | NO <input type="checkbox"/> (tick the appropriate box) |
| <input type="checkbox"/> | LC-MS/MS | YES <input type="checkbox"/> | NO <input type="checkbox"/> (tick the appropriate box) |

2. As a participant, we understand that

- a) All essential apparatus, chemicals and other requirements specified in the method shall be available in our laboratory when the programme begins;
- b) Specified "timing" requirements, such as starting date and finishing date of the programme, shall be rigidly met;
- c) The method shall be strictly adhered to;
- d) Samples shall be handled in accordance with instructions;
- e) A qualified operator shall perform the test.

Having studied the method and having made a fair appraisal of our capabilities and facilities, we feel that we will be adequately prepared for cooperative testing of this method.

3. Comments:

Signature:	_____	Date:	_____
Tel:	_____	Fax:	_____
Organization:	_____	Email:	_____

4. Communicational contact:

Dr. Guo-Fang Pang
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