

EFFECTIVE DATE	N P Analytical Laboratories	METHOD CODE
REVISED: 11/19/25	LABORATORY TEST METHOD SUMMARY	PEST
REPLACES: 04/01/24	Multiresidue pesticide analysis by GC/MS	PAGE 1 OF 2
KEY WORDS: QuEChERS, GC-MS/MS, PCB's, pesticides, organochlorine, organophosphorus		

1. SCOPE AND PURPOSE:

This is a multiresidue method for the low level determination of selected organochlorine, organophosphorus, and polychlorinated biphenyl (PCB) pesticides in feed, food ingredients, grain, plant, and paper-based bedding materials. There is no assurance that matrices other than those listed can be assayed using this method.

2. PRINCIPLE:

- 2.1. The sample is hydrated with water and extracted with acetonitrile. After the initial extraction, magnesium sulfate, sodium chloride, sodium citrate, and disodium citrate sesquihydrate are added. The mixture is shaken vigorously and centrifuged for phase separation. An aliquot of the upper organic layer is then cleaned up by dispersive solid phase extraction containing sorbents (e.g. primary secondary amine sorbent (PSA), graphitized carbon black (GCB) and magnesium sulfate). The extracts are acidified by the addition of formic acid to improve the stability of base sensitive pesticides.
- 2.2. Using a 2 g sample, the lowest confidence levels of this method are:

Matrix	LCL	% recovery range
Non-Alfalfa	10ppb-if not stated below 2,2',4,6-Tetrachlorobiphenyl (#50)-20ppb 2,2',3,4,5'-Pentachlorobiphenyl (#87)-20ppb 2,2',4,4',5,6'-Hexachlorobiphenyl (#154)-20ppb 2,2',3,4,5,6'-Heptachlorobiphenyl (#186)-30ppb 2,2',3,3',4,5',6,6'-Octachlorobiphenyl (#201)-30ppb Decachlorobiphenyl (#209)-50ppb	60-140%
Paper	10ppb-if not stated below 2,2',4,6-Tetrachlorobiphenyl (#50)-20ppb 2,2',3,4,5'-Pentachlorobiphenyl (#87)-20ppb 2,2',4,4',5,6'-Hexachlorobiphenyl (#154)-20ppb 2,2',3,4,5,6'-Heptachlorobiphenyl (#186)-30ppb 2,2',3,3',4,5',6,6'-Octachlorobiphenyl (#201)-30ppb Decachlorobiphenyl (#209)-50ppb	60-140%
Alfalfa	10ppb-if not stated below 2,2',4,6-Tetrachlorobiphenyl (#50)-20ppb 2,2',3,4,5'-Pentachlorobiphenyl (#87)-20ppb 2,2',4,4',5,6'-Hexachlorobiphenyl (#154)-20ppb	60-140%
Alfalfa	2,2',3,4,5,6'-Heptachlorobiphenyl (#186)-30ppb 2,2',3,3',4,5',6,6'-Octachlorobiphenyl (#201)-30ppb Decachlorobiphenyl (#209)-50ppb Methoxychlor-10ppb Mirex-10ppb o,p'-DDT-10ppb p,p'-DDT-10ppb	40-120%

- 2.3. These lowest confidence levels may vary with sample matrix. Lower detection levels may be achieved for selected compounds and matrices upon request.
- 2.4. Known Interferences: Certain types of plastics, rubber, soaps, detergents, and impurities in solvents can cause interferences that may mask or coelute with some pesticide peaks. Avoid allowing samples, solvents or glassware to come into direct contact with rubber, plastics other than those indicated or soaps and detergents.

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3. **PRECISION:**

Records of method precision based on Method Validation and/or known control summaries are located in the QA Master file for this test method. Assay precision may vary with test matrix and analyte level. Terms used to describe method precision are defined in NPSOP3000, *Validation of Quantitative Chemical Tests*.

4. **REFERENCES:**

- 4.1. LI-00.140-01 "Multiresidue Method for Pesticides by GC", Nov 2017.
- 4.2. EN 15662 "Foods of Plant Origin-Determination of pesticide residues using GC-MS and/or LC-MS/MS following acetonitrile extraction/partitioning and clean up by dispersive SPE-QuEChERS-method." Oct 2018.
- 4.3. NQA-54.0005-3 "Pesticide Residues by GC-MS/MS."
- 4.4. NQA-54.0002-6 "Multiresidue Extraction method for Pesticides-QuEChERS Method."
- 4.5. SANCO/12571/2013 "Guidance document on analytical quality control and validation procedures for pesticide residue analysis in food and feed."