

LABORATORY MANAGEMENT SYSTEM
OF THE DEPARTMENT OF ENVIRONMENT PROTECTION
MARITIME INSTITUTE, GDYNIA MARITIME UNIVERSITY



Edition No. 3
Date 05.10.2019
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RESEARCH PROCEDURE PB-45

DETERMINATION OF ORGANOCHLORINE PESTICIDES IN SOIL SAMPLES

Supervised document

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1. PURPOSE

The purpose of the procedure is to determine the method for analysis the content of organochlorine pesticides in agricultural soil samples by means of gas chromatography with mass spectrometry (GC-MS).

2. SUBJECT - SCOPE

The subject of the study are soils.

Lower limit of quantification: 1,0 mg·kg⁻¹.

3. PERSONNEL QUALIFICATIONS

The employee conducting the study should have: secondary chemical education and analytical experience.

4. DESIGNATION

Organochlorine pesticides: a-HCH, b-HCH, g-HCH, d-HCH, Aldrin, Izodrin, Dieldrin, Endrin, DDE, DDD, DDT

5. REAGENTS AND SUPPORT MATERIALS

➤ Reagents and standards:

- Hexane for gas chromatography
- Acetone 99.5%
- Sulfuric acid (VI) min. 95%
- Silica gel columns
- Mixtures of pesticide standards

➤ Support materials:

- 22 cm³ stainless extraction cells
- Cellulose filters
- 60 cm³ vials with screw cap and septum
- Thermo Scientific Dionex ASE Prep DE (diatomaceous earth)
- 2 cm³ vial with cap, septum and inner cartridge
- Chromatographic syringe with capacity 10 mm³.

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6. EQUIPMENT

- Thermo Scientific Dionex ASE 350 Accelerated Solvent Extractor
- Gas chromatograph with mass spectrometer (GC-MS)
- Analytical balance
- System for evaporating in an inert gas stream (nitrogen)
- Rotary evaporator

7. TEST CONDITIONS

The tests are carried out at ambient temperature.

8. DESCRIPTION OF THE PROCEDURE

8.1 Soil sampling

- take a general sample from a 0-30 cm layer of soil
- to take a general sample, up to 20 incremental (single) samples should be taken evenly from the field surface
- the general (average) sample should represent the area of agricultural use with similar natural conditions (i.e. soil type, terrain type) and agrotechnical conditions (fore crop, cultivation, fertilization)
- the usable area per general sample, with an aligned soil surface and similar terrain, may not exceed 4 ha.
- the general sample should be prepared separately for each crop.
- general samples should be marked on a precisely made terrain sketch and numbered respectively to the field surface they represent. samples taken from grassland must be marked with X in addition to the number.

8.2 Preparation of the test sample

- the sample should be stored at 2–5°C until analysis
- the soil sample after quartering should be dried (air drying according to PPN-ISO 11464:1999).

8.3 Stages of proceedings

Extraction

- Weigh 10 g of dry and ground sample (after humidity determination) into a 22 cm³ extraction cell
- Extract the sample in an extractor (ASE - Accelerated Solvent Extraction)

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- ASE extraction conditions:
 - solvent - hexane/acetone (1:1 v/v)
 - temperature - 100°C
 - pressure - 1500 psi
 - static time - 5 min
 - static cycles - 1-2
 - flush - 60%
- purge - 60-120 s
- clean the obtained extract with sulfuric acid and silica gel columns
- concentrate the extract to 1 cm³ (in a rotary evaporator and under a stream of nitrogen).

Analysis

- Determine pesticides in the obtained extract by gas chromatography with mass spectrometry.

8.4 Expression of results

Pesticide concentration is given in mg·kg⁻¹ dry weight of sample.

8.5 Quality assurance

A standard solution Pesticide Mix 323 is used to ensure the quality and check the measuring system (reagents, measuring device, sample preparation) and sample handling mode.

Parallel samples should also be analysed (every 10 samples per measurement series).

8.6 Control and finding of irregularities in tests

Test control or verification must be carried out in accordance with the program for monitoring the validity of measurements results in accordance with Management System Book. Irregularities in tests found by the Staff or the Head of the Laboratory cause the repeated analysis by an authorized person.

9. LIST OF REFERENCED DOCUMENTS

9.1 Normative documents

- PN-ISO 11464:1999 - Polish version. Soil quality - Initial sample preparation for physico-chemical tests
- PN-R-04031:1997 – Polish version. Chemical and agricultural analysis of soil. Sampling.

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9.2 Laboratory Management System documents

Management System Book.

10. ADDITIONAL INFORMATION

Not included.

Verification:

K. Galer-Tatarowicz, G. Pazikowska-Sapota, G. Dembska, L. Dzierzbicka-Głowacka. Impact of Puck commune farms on the quality of soils, inland and marine waters - pesticides, XII Conference 'CHEMISTRY, GEOCHEMY AND PROTECTION OF THE MARINE ENVIRONMENT' SOPOT, Institute of Oceanology PAS 19.10.2018 - presentation

G. Pazikowska-Sapota, K. Galer-Tatarowicz, G. Dembska, M. Wojtkiewicz, E. Duljas, S. Pietrzak, L. Dzierzbicka-Głowacka. 2020. The impact of pesticides used at the agricultural land of the Puck commune on the environment of the Puck Bay. PeerJ 8:e8789 <http://doi.org/10.7717/peerj.8789>