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Determination of HCH and DDT in Feeds

饲料中六六六、滴滴涕的测定

(ISO 14181:2000, Animal feeding stuffs - Determination of residues of organochlorine pesticides - Gas chromatographic method, MOD)

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Determination of HCH and DDT in Feeds

1 Scope

This document describes the gas chromatography method for the determination of HCH and DDT in feeds.

This document is applicable to the determination of α-HCH, β-HCH, γ-HCH, δ-HCH, *p*,*p*′-DDE, *o*,*p*′-DDT, *p*,*p*′-DDD and *p*,*p*′-DDT in compound feeds, feed concentrates, concentrate supplements, premixes containing additives and raw materials of feeds.

The detection limit and limit of quantitation in this document are in accordance with Appendix A.

2 Normative References

The contents of the following documents constitute indispensable clauses of this document through the normative references in the text. In terms of references with a specified date, only versions with a specified date are applicable to this document. In terms of references without a specified date, the latest version (including all the modifications) is applicable to this document.

GB/T 6682 Water for Analytical Laboratory Use - Specification and Test Methods (GB/T 6682-2008, ISO 3696:1987, MOD)

GB/T 20195 Animal Feed - Preparation of Test Samples (GB/T 20195-2024, ISO 6498:2012, MOD)

3 Terms, Definitions and Symbols

3.1 Terms and Definitions

This document does not have terms or definitions that need to be defined.

3.2 Symbols

The following symbols apply to this document.

DDT: dichlorodiphenyltrichloroethane (a general term for four chemical compounds: o, p′-DDT, p, p-DDE, p, p′-DDD, and p, p′-DDT)

HCH: benzene hexachloride (a general term for four chemical compounds: α-, β-, γ-, and δ-benzene hexachloride)

o, *p*'-DDT: *o*-, *p*'-DDT [chemical name: 1,1,1-trichloro-2-(2-chlorophenyl)-2-(4-chlorophenyl)

ethane, CAS No.: 789-02-6]

p, *p*'-DDE: *p*-, *p*'-DDE [chemical name: 1,1-dichloro-2,2-bis(4-chlorophenyl) ethylene, CAS No.: 72-55-9]

p, *p*'-DDD: *p*-, *p*'-DDD [chemical name: 1,1-dichloro-2,2-bis(4-chlorophenyl) ethane, CAS No.: 72-54-8]

p, *p*'-DDT: *p*-, *p*'-DDT [chemical name: 1,1,1-trichloro-2,2-bis(4-chlorophenyl) ethane, CAS No.: 50-29-3]

α-HCH: alpha-HCH (Chemical name: *α*-1,2,3,4,5,6-hexachlorocyclohexane, CAS No.: 319-84-6)

β-HCH: beta-HCH (Chemical name: *β*-1,2,3,4,5,6-hexachlorocyclohexane, CAS No.: 319-85-7)

γ-HCH: gamma-HCH (chemical name: *γ*-1,2,3,4,5,6-hexachlorocyclohexane, CAS No.: 58-89-9)

δ-HCH: delta-HCH (chemical name: *δ*-1,2,3,4,5,6-hexachlorocyclohexane, CAS No.: 319-86-8)

4 Principle

In accordance with the principle of like dissolves like, the analyte in the specimen is extracted with a mixture of acetone and n-hexane. The extracting solution is purified by chromatographic column solid-liquid separation (or use concentrated sulfuric acid to remove interfering substances by dehydration and sulfonation). The purification solution is separated by capillary gas chromatography and detected by electron capture detector. Conduct qualitative determination based on the retention time of the chromatographic peaks, and quantitative determination based on the external standard method.

5 Reagents or Materials

Unless otherwise specified, only analytically pure reagents are used.

5.1 Water: GB/T 6682, Grade 1.

5.2 Isooctane: chromatographically pure.

5.3 n-Hexane: chromatographically pure.

5.4 Acetone: chromatographically pure.

5.5 Dichloromethane: chromatographically pure.

5.6 Concentrated sulfuric acid: guaranteed reagent.

5.7 Phosphoric acid.

5.8 Anhydrous sodium sulfate: dry at 500 °C for 4 hours, remove, place in a desiccator, cool, and seal for later use.

5.9 Sodium sulfate solution (20 g/L): weigh-take 20 g of anhydrous sodium sulfate, use water to dissolve it and dilute to 1,000 mL, and thoroughly mix it.

5.10 Acetone/n-hexane mixed solution: measure-take 30 mL of acetone (5.4) and 220 mL of n-hexane (5.3), thoroughly mix it.

5.11 Perchloric acid/glacial acetic acid mixed solution: measure-take 50 mL of perchloric acid and 50 mL of glacial acetic acid, thoroughly mix it.

5.12 Silica gel: 63 μm ~ 100 μm, dry overnight at 130 °C, remove, place in a desiccator, cool, and seal for later use.

5.13 10% hydrous silica gel: place 90 g of silica gel (5.12) in a stoppered round-bottom flask, slowly add 10 mL of water, shake while adding water (to prevent agglomeration), put on the stopper, and shake in an oscillator for 30 minutes. Prepare immediately before use.

5.14 Dichloromethane/n-hexane mixed solution: measure-take 20 mL of dichloromethane (5.5) and 80 mL of n-hexane (5.3), thoroughly mix it.

5.15 Acidified diatomite: weigh-take 1.5 g of diatomite with a median particle size of 7 μm, dried at 500 °C for 4 h, and then, cooled. Place in a 50 mL centrifuge tube and add 0.6 mL of concentrated sulfuric acid (5.6). Tighten the cap, and conduct vortex oscillation for 30 min. This is an individual portion and reserved for later use.

5.16 Standard stock solutions (100 μg/mL): accurately weigh-take appropriate amounts of *α*-HCH, *β*-HCH, *γ*-HCH, *δ*-HCH, *p*, *p*'-DDE, *o*, *p*'-DDT, *p*, *p*'-DDD and *p*, *p*'-DDT (purity not less than 99.0%) (accurate to 0.01 mg), and respectively place them in 100 mL brown volumetric flasks. Use isooctane (5.2) to dissolve *α*-HCH, *γ*-HCH, *δ*-HCH, *p*, *p*'-DDE, *o*, *p*'-DDT, *p*, *p*'-DDD and *p*, *p*'-DDT, reach a constant volume and thoroughly mix them; use acetone (5.4) to dissolve β-HCH, reach a constant volume and thoroughly mix it. Store below −18 °C in the dark, and they shall remain valid for 12 months. Alternatively, use standard substances.

5.17 Mixed standard intermediate solutions: respectively and accurately transfer-take appropriate amounts of the standard stock solutions (5.16) into 100 mL brown volumetric flasks, use isooctane (5.2) to dilute them, reach a constant volume and thoroughly mix them. The mass concentration of *β*-HCH, *p*, *p*'-DDD and *p*, *p*'-DDT is 2 μg/mL; the mass concentration of *α*-HCH, *γ*-HCH, *δ*-HCH, *p*, *p*'-DDE and *o*, *p*'-DDT is 1 μg/mL. Store below −18 °C in the dark, and they shall remain valid for 6 months. Alternatively, use standard substances.

5.18 Mixed standard series solutions: accurately transfer-take appropriate amount of the mixed

standard intermediate solutions (5.17) and use isooctane (5.2) to dilute them in series into mixed standard series solutions with mass concentrations of 2 ng/mL, 4 ng/mL, 10 ng/mL, 20 ng/mL, 50 ng/mL, 100 ng/mL and 200 ng/mL respectively for *β*-HCH, *p*, *p*'-DDD and *p*, *p*'-DDT; and 1 ng/mL, 2 ng/mL, 5 ng/mL, 10 ng/mL, 25 ng/mL, 50 ng/mL and 100 ng/mL respectively for *α*-HCH, *γ*-HCH, *δ*-HCH, *p*, *p*'-DDE and *o*, *p*'-DDT. Prepare immediately before use.

5.19 Absorbent cotton: soak in acetone (5.4) for 30 min, discard the acetone, and then, soak in n-hexane (5.3) for 30 min, discard the n-hexane, and air-dry for later use.

6 Instruments and Equipment

6.1 Gas chromatograph: equipped with electron capture detector (ECD).

6.2 Analytical balance: with an accuracy of 0.1 mg, 0.01 mg.

6.3 Oscillator: not less than 180 r/min.

6.4 Constant-temperature water bath: 80 °C ± 2 °C.

6.5 Vortex mixer.

6.6 Multi-tube vortex oscillator: with a speed not less than 2,500 r/min.

6.7 Centrifuge: with a speed not less than 8,000 r/min.

6.8 Nitrogen blowing instrument.

6.9 Rotary evaporator.

6.10 Box-type resistance furnace: 500 °C ± 25 °C.

6.11 Chromatographic column: with an internal diameter of 8 mm ~ 10 mm, a length of 15 cm ~ 20 cm. Top with a cylindrical funnel reservoir, with a capacity of approximately 30 mL.

7 Samples

In accordance with GB/T 20195, prepare samples of at least 200 g. Solid samples shall be pulverized, until they all pass through a test sieve with a 1 mm pore size, thoroughly mixed, and stored in a sealed container for future use. Liquid samples and pastes with uneven distribution shall be homogenized before testing. Gum samples need to be chopped into particles with a particle size less than 0.5 cm.

8 Test Procedures

8.1 Extraction

8.1.1 Grease samples

Perform two tests in parallel. Weigh-take 1 g of specimen (accurate to 0.1 mg) into a 25 mL (V_1) volumetric flask. Add the acetone/n-hexane mixed solution (5.10) and reach a constant volume, thoroughly mix it and reserve for purification.

8.1.2 Gum, liquid and paste samples (except grease)

Perform two tests in parallel. Weigh-take 5 g of specimen (accurate to 0.1 mg) into a 100 mL stoppered conical flask. Add 20 mL of the perchloric acid/glacial acetic acid mixed solution (5.11). Heat and digest in an 80 °C water bath for 30 min. Remove it, cool to room temperature, and transfer the digestion solution to a 50 mL centrifuge tube. Use 5 mL of water to rinse the conical flask and transfer it to the centrifuge tube. Respectively add 10 mL of the acetone/n-hexane mixed solution (5.10) and vortex to mix for 30 seconds. At 3,000 r/min, centrifuge for 2 minutes, and collect the upper layer solution. Successively use 8 mL and then 5 mL of the acetone/n-hexane mixed solution (5.10) to repeat the extraction twice; combine the extracts. Insert a small amount of absorbent cotton (5.19) and anhydrous sodium sulfate (1 cm thick) (5.8) into a cylindrical funnel. Filter the extracts into a 25 mL (V_1) volumetric flask. Use a small amount of the acetone/n-hexane mixed solution (5.10) to wash the anhydrous sodium sulfate layer, reach a constant volume, thoroughly mix it and reserve for purification.

8.1.3 Other samples

Perform two tests in parallel. Weigh-take 5 g of specimen (accurate to 0.1 mg) into a 100 mL stoppered conical flask. Accurately add 25 mL (V_1) of the acetone/n-hexane mixed solution (5.10) and 4 ~ 5 drops of phosphoric acid (5.7). At 180 r/min, oscillate and extract for 30 min. Remove and allow it to stand for 10 min. Transfer the upper layer extracting solution to a 50 mL centrifuge tube, and at 8,000 r/min, centrifuge for 5 min. Collect the supernatant for purification.

8.2 Purification

Use one of the following three methods for purification.

a) Chromatographic column purification method I (not suitable for grease samples): insert a small amount of absorbent cotton (5.19) into the chromatographic column (6.11), successively add 5 g of silica gel (5.13) and 5 g of anhydrous sodium sulfate (5.8), and use 20 mL of n-hexane (5.3) to rinse the chromatographic column. Accurately transfer-take 5 mL of the extracting solution (8.1), at below 40 °C, use nitrogen to blow it, until it is nearly dry. Use 5 mL (V_2) of n-hexane (5.3) to re-dissolve it and transfer the entire amount to the top of the chromatographic column. Use 50 mL of the dichloromethane/n-hexane mixed solution (5.14) to elute it and collect the eluate. At below 40 °C, perform rotary evaporation to approximately 2 mL, and use 5 mL of n-hexane (5.3) to transfer in several small portions to a 10 mL graduated tube. At below 40 °C, use nitrogen to blow it, until it is nearly dry. Accurately add 1 mL (V_3) of n-hexane (5.3) to re-dissolve it and perform vortex. Reserve it for later use.

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