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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

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Determination of HCH and DDT in feeds

饲料中六六六、滴滴涕的测定

(English Translation)

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Table of Contents

Foreword...	3
1 Scope...	5
2 Normative References...	5
3 Terms, Definitions and Symbols...	5
4 Principle...	6
5 Reagents or Materials...	6
6 Instruments and Equipment...	8
7 Samples...	8
8 Test Procedures...	8
9 Test Data Processing...	11
10 Precision...	12
Appendix A (normative) Detection Limit and Limit of Quantitation...	13
Appendix B (informative) Gas Chromatogram of Standard Solutions...	14

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 alpha-HCH, beta-HCH, gamma-HCH, delta-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)

3.1 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. alpha-, beta-, gamma-, and delta- 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]

alpha-HCH. alpha-HCH (Chemical name. alpha-1,2,3,4,5,6-hexachlorocyclohexane, CAS No.. 319-

84-6)

beta-HCH. beta-HCH (Chemical name. beta-1,2,3,4,5,6-hexachlorocyclohexane, CAS No.. 319-85-

7)

gamma-HCH. gamma-HCH (chemical name. gamma-1,2,3,4,5,6-hexachlorocyclohexane, CAS No.. 58-89-

9)

delta-HCH. delta-HCH (chemical name. delta-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 $\mu\text{g/mL}$). accurately weigh-take appropriate amounts of alpha-

HCH, beta-HCH, gamma-HCH, delta-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 alpha-HCH, gamma-HCH, delta-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 beta-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.