



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

GB 5009.190-2014

**National Food Safety Standard -- Determination of Indicator
Polychlorinated Biphenyls in Foods**

食品安全国家标准 食品中指示性多氯联苯含量的测定

(English Translation)

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1 Scope

Method I in this standard specifies the determination methods for polychlorinated biphenyls (PCBs), including indicator PCBs (PCB28, PCB52, PCB101, PCB118, PCB138, PCB153 and

PCB180) as well as PCB18, PCB33, PCB44, PCB70, PCB105, PCB128, PCB170, PCB187,

PCB194, PCB195, PCB199 and PCB206 in foods specified in the Global Environmental Monitoring System/Food Planning Part. Method II specifies the determination methods of PCB28, PCB52, PCB101, PCB118, PCB138, PCB153 and PCB180.

This standard is applicable to the determination of indicator PCBs in animal-based foods (like fish, shellfish, egg, meat, milk and their products) and oil and fat samples.

Method I Stable Isotope Diluted Gas
Chromatography-mass Spectrometry

2 Principle

Adopt stable isotope dilution technology; add $^{13}\text{C}_{12}$ labeled PCBs in the sample as the quantitative standard; carry out Soxhlet extraction; carry out chromatography purification, separation and concentration for the extracted sample solution with column chromatography, and then add it into the internal recovery standard; adopt gas chromatography - low-resolution gas chromatograph-mass spectrometer, analyze through quadrupole mass spectrum selected ion monitoring (SIM) or ion trap tandem mass spectrum multi-response monitoring (MRM), and then quantify through internal standard method.

3.1 Reagents

3.1.1

N-hexane (C_6H_{14})

Pesticide residue.

3.1.2

Dichloromethane (CH_2Cl_2)

Pesticide residue.

3.1.3

Acetone ($\text{C}_3\text{H}_6\text{O}$)

Pesticide residue.

3.1.4

Methanol (CH_3OH)

Pesticide residue.

3.1.5

Isooctane (C_8H_{18})

Pesticide residue.

3.1.6 Anhydrous sodium sulfate (Na_2SO_4): guaranteed reagent. Put the commercially

available anhydrous sodium sulfate into the chromatographic column, and successively elute it twice with n-hexane and dichloromethane; the volume of solvent used for each elution is about twice the volume of anhydrous sodium sulfate. After elution, transfer the anhydrous sodium sulfate into a flask; bake it to dry at 50°C and then bake it for 8~12h at

225°C; cool it and then store it in a dryer.

3.1.7 Sulfuric acid (H₂SO₄): with content of 95%~98%, guaranteed reagent.

3.1.8 Sodium hydroxide (NaOH): guaranteed reagent.

3.1.9 Silver nitrate (AgNO₃): guaranteed reagent.

3.1.10 Chromatographic silica gel (75~250μm). Put the commercially available silica gel

into the glass chromatographic column, and successively elute it twice with n-hexane and dichloromethane; the volume of solvent used for each elution is about twice the volume of silica gel. After elution, transfer the silica gel into flask, and cover the flask mouth with aluminum foil; put it into the oven, and bake at 50°C to dry; then heat up to 180°C and bake for 8~12h; cool it and then put it into a reagent bottle with ground stopper; store it in the dryer.

3.1.11 44% acid silica gel: weigh 100g of activated silica gel, dropwise add 78.6g of sulfuric acid and shake until it is free of cake; put in into the reagent bottle with ground stopper and store it in dryer.

3.1.12 33% alkaline silica gel: weigh 100g of activated silica gel, dropwise add 49.2g of 1mol/L sodium hydroxide solution and shake until it is free of cake; put in into the reagent bottle with ground stopper and store it in dryer.

3.1.13 10% silver nitrate silica gel: dissolve 5.6g of silver nitrate into 21.5mL of deionized water, dropwise add it into 50g of activated silica gel, and shake until it is free of cake; put in into the brown reagent bottle with ground stopper and store it in dryer.

3.1.14 Alkaline aluminum oxide: adopt alkaline aluminum oxide for chromatogram

chromatography, and bake it at 660°C for 6h; put it into the reagent bottle with ground stopper and store it in dryer.

3.2.1 Standard solution for the determination of the time window: composed of cognate

can lead to a good cleaning effect. Where brush is adopted for cleaning, special attention shall be paid to that the brush shall not damage the internal surface of glass ware.

5.1.1.1 Pack the site-collected sample with lucifugal material like aluminum foil and brown

bottle, put it in small refrigerator and then transport it to the laboratory; store it in a

cryogenic refrigerator below -10°C.

5.1.1.2 Solid sample like fish and meat may be dried by freezing or anhydrous sodium sulfate, and shall be mixed uniformly. Oil and fat may be directly dissolved in n-hexane for purification treatment.

5.1.2 Extraction

5.1.2.1

Before extraction, put an empty cellulose or glass fiber extraction sleeve into the Soxhlet extractor; use n-hexane + dichloromethane (50+50) as extraction solvent, pre-extract for 8h, and then take it out for air drying.

5.1.2.2 Put 5.0g~10.0g of pretreated sample into the extraction sleeve treated according to

Article 5.1.2.1, add $^{13}\text{C}_{12}$ labeled quantitative internal standard (Article 3.2.2); cover the sample with glass wool, balance for 30min and then put into Soxhlet extractor; use proper amount of n-hexane + dichloromethane (50+50) as extraction solvent and extract for 18~24h;

control the back flow speed within 3~4 times/h.

5.1.2.3 After extraction, transfer the extraction solution into a round-bottom flask, concentrate it with rotary evaporator to nearly dry. Where the analysis result is calculated according to fat, the fat content of sample shall be determined.

5.1.2.4 Fat determination: exactly weigh the mass of round-bottom flask before

concentration, and then exactly weigh the mass of the round-bottom flask again after the solvent is concentrated to dry; the difference of two weighed results is regarded as the fat content of the sample. After the fat content determination, add a small amount of n-hexane to dissolve the residue in flask.

5.1.3.1 Acidic silica gel column purification

Purification column filling: plug the bottom of glass column with glass wool, and then successively fill in 4g of activated silica gel, 10g of acid silica gel, 2g of activated silica gel and 4g of anhydrous sodium sulfate (see Figure D.1 in Appendix D) from bottom to top. Elute with 100mL of n-hexane in advance.

Purification: completely transfer the concentrated extraction solution to the column; wash the round-bottom flask for 3~4 times with 5mL of n-hexane, and transfer the washing liquid to the column. Where the liquid level falls to the anhydrous sodium sulfate layer, add 180mL of n-hexane to elute, and concentrate the eluent to about 1mL.