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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB 5009.27-2016

National Food Safety Standard – Determination of Benzo(a)Pyrene in Foods

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China Food and Drug Administration.

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National Food Safety Standard – Determination of Benzo(a)Pyrene in Foods

1 Scope

This Standard specifies the determination method for the benzo(a)pyrene in foods.

This Standard is applicable to determination of the benzo(a)pyrene of the grains and their products (rice, brown rice, rice, wheat, wheat flour, corn, cornmeal, corn residues AND corn flakes), meat and meat products (smoked, roasted and barbecued), aquatic animals and their products (smoked and boiled aquatic products) AND grease & their products.

2 Principle

Samples shall be extracted by organic solvents, purified by neutral alumina or molecularly imprinted cartridges AND concentrated to dryness; use acetonitrile to dissolve it; it shall be separated by reversed-phase liquid chromatography AND detected by fluorescence detector. Use the retention time of chromatographic peaks to determine the quality; use the external standard method to determine the quantity.

3 Reagents and Materials

Unless otherwise stated, the used reagents in this method shall all be analytical purity; the water shall be the Grade-1 water which is specified in GB/T 6682.

3.1 Reagents

- **3.1.1** Toluene (C₇H₈): Chromatographically pure.
- **3.1.2** Acetonitrile (CH₃CN): Chromatographically pure.
- **3.1.3** n-hexane (C_6H_{14}) : chromatography.
- **3.1.4** Dichloromethane (CH₂Cl₂): Chromatographically pure.

3.2 Standards

Benzo(a)pyrene standard ($C_{20}H_{12}$, CAS No.: 50-32-8): the purity shall be $\geq 99.0\%$; or

solution into the chromatograph's sample vial again; concentrate it until it is near dry. Accurately pipette 1mL of acetonitrile into the chromatograph's sample vial, vortex redissolve it for 0.5min; after passing the microporous membrane, it shall be used for liquid chromatography.

Purification method 2: adopt the benzo(a)pyrene molecular imprinting column; orderly use 5mL of dichloromethane and 5mL of n-hexane to activate the column. Transfer the liquid to be purified into the column; when the liquid-level falls to the column bed, use 6mL of n-hexane to rinse the column; discard the effluent. Use 6mL of dichloromethane to elute and collect the purified solution into the test tube. Under 40°C, use nitrogen to dry the purification solution; accurately pipette 1mL of acetonitrile; vortex re-dissolve it for 0.5min; after passing the microporous membrane, it shall be used for liquid chromatography.

5.1.2 Smoked, roasted & barbecued AND smoked & grilled aquatic products

Pretreatment: remove the bones of the meat; remove the thorns of fish; remove the shell of shellfish; grind the edible portion evenly; store it in clean sample bottles; mark it; at -16° C $\sim -18^{\circ}$ C, stored it in the refrigerator.

Extraction: same as the extraction in 5.1.1.

Purification method 1: Except that the volume of n-hexane's eluent is 70 mL, the rest of the operation shall be the same as the purification method 1 in 5.1.1.

Purification method 2: the operation shall be the same as the purification method 2 in 5.1.1.

5.1.3 Grease and its products

Extraction: weigh and take 0.4g (accurate to 0.001g) of sample; add 5mL of n-hexane; vortex-mix it for 0.5min. To be purified.

Note: If the samples are the hydrous oil products, such as margarine, etc., emulsification will occur. It shall perform 4000r/min centrifugalization for 5min; transfer out the n-hexane layer. To be purified.

Purification method 1: Except for using 0.4mL of acetonitrile to vertically re-dissolve the sample in the last, other operations shall be the same as the purification method 1 in 5.1.1.

Purification method 2: Except for using 0.4mL of acetonitrile to vertically re-dissolve the sample in the last, other operations shall be the same as the purification method 2 in 5.1.1. When the sample is prepared, pretreatments of different samples need to have sample's blank test at the same time.

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