

ICS 67.040
CCS C 53



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

GB 14883.4-2016

Replacing GB 14883.4-1994

**Examination of radioactive materials for foods -- Determination
of promethium-147**

食品安全国家标准 食品中放射性物质钷-147的测定
(English Translation)

Issued on: August 31, 2016

Implemented on: March 1, 2017

Issued by: National Health and Family Planning Commission

Table of Contents

Foreword
1 Scope
3 Reagents and materials
3.1 Reagents
3.2 Reagent preparation
3.3 Standard
3.4 Standard Solution
4 Instruments and equipment
5 Step, Analysis
5.2 Determination of counting efficiency
5.3 Sampling and pretreatment
5.4 Preparation of sample and assay
5.5 Determination of the chemical recovery
6 Expression analysis
7 Other

Foreword

This standard replaces GB 14883.4-1994 "test radioactive materials for foods - Determination of promethium -147." this standard compared with GB 14883.4-1994, the main changes are as follows.

--- Standard name was changed to "national food safety standard radioactive materials for foods - Determination of promethium -147";

--- National food safety standards in accordance with the format of the text has been adjusted;

--- Delete Appendix A, Appendix A of the original content is placed in the body of the respective terms.

National Food Safety Standard Determination of radioactive substances promethium -147

1 Scope

This standard applies to promethium -147 (147Pm) Determination of various foods.

Principle 2

Neodymium and samarium as ^{147}Pm carrier, food ash leaching with nitric acid and hydrogen peroxide, promethium and other heavy rare earth elements in the form of oxalate Lake, then adsorbed coated with titanium - (2-ethylhexyl) phosphoric acid polychlorotrifluoroethylene (referred HDEHP-Kel-F) column. Color paper layer method the ^{147}Pm separated from other rare earth elements. With low background beta beta measuring instrument measuring the radioactivity ^{147}Pm .

3 Reagents and materials

Unless otherwise indicated, the methods used were of analytical grade reagents and water as a water GB/T 6682 regulations.

3.1 Reagents

3.1.1

two - (2-ethylhexyl) phosphoric acid ($\text{C}_{16}\text{H}_{35}\text{O}_4\text{P}$). also known as double iso-octyl phosphate, chemically pure.

3.1.2

n-heptane (C_7H_{16}).

3.1.3

hydroxylamine hydrochloride ($\text{NH}_2\text{OH} \cdot \text{HCl}$).

3.1.4

anhydrous sodium acetate ($\text{C}_2\text{H}_3\text{NaO}_2$).

3.1.5

Uranium reagent III ($\text{C}_{22}\text{H}_{18}\text{As}_2\text{N}_4\text{O}_{14}\text{S}_2$).

3.1.6

monochloroacetic acid ($\text{C}_2\text{H}_3\text{ClO}_2$).

3.1.7

Uranium reagent I ($\text{C}_{16}\text{H}_{11}\text{AsN}_2\text{Na}_2\text{O}_{11}\text{S}_2$).

3.1.8

Ammonium thiocyanate (NH_4SCN).

3.1.9

butanone ($\text{C}_4\text{H}_8\text{O}$).

3.1.10

absolute ethanol ($\text{C}_2\text{H}_6\text{O}$).

3.1.11

hexamethylenetetramine ($\text{C}_6\text{H}_{12}\text{N}_4$).

3.1.12

nitric acid (HNO_3).

3.1.13

sulfosalicylic acid ($\text{C}_7\text{H}_6\text{O}_6\text{S} \cdot 2\text{H}_2\text{O}$).

3.1.14

ascorbic acid ($\text{C}_6\text{H}_8\text{O}_6$).

3.1.15 2,4-dinitrophenol ($\text{C}_6\text{H}_4\text{N}_2\text{O}_5$).

3.2 Reagent preparation

3.2.1

hydroxylamine hydrochloride - sodium acetate buffer. 1L of water was added to hydroxylamine hydrochloride and 9g 10g of anhydrous sodium acetate, pH adjusted with nitric acid solution to 1.5.

3.2.2

a buffer acid - a mixture of uranium reagent III. the 1.00g dissolved uranium reagent III 120mL 1mol/L sodium hydroxide solution.

In a 500mL water dissolve 100g acid, mixing the two, diluted with water to 1L.

3.2.3

eluent. Add 2.5g 300mL ammonium thiocyanate in methyl ethyl ketone was added 4mL of water and dissolved, stirring constantly added 3mL of concentrated nitric acid. Use the supernatant.

3.2.4

uranium reagent layer was I agent. Weigh 0.10g uranium reagent I, dissolved in 35mL saturated aqueous solution of hexamine, diluted with anhydrous ethanol to 100mL, mix, clarified and filtered. When using a volume of about 150mL prosper in the nebulizer.

3.3 Standard

^{147}Pm standard solution. radioactive decay intensity of about $1 \times 10^3/(\text{min} \cdot \text{mL})$.

3.4 Standard Solution

Neodymium, samarium standard solution. Weigh accurately spectroscopically pure neodymium oxide (Nd_2O_3) and samarium oxide (Sm_2O_3) each 1.0000g, was dissolved in a

small amount of concentrated nitric Acid, with 0.5mol/L nitrate formulated into 1L, this solution per ml of stock standard solution containing 1.0mg of each Nd₂O₃ and Sm₂O₃. With shift Pipette accurately Pipette 10.0mL standard stock solution was diluted to 100mL with 0.5mol/L nitrate. This solution per milliliter and Nd₂O₃ Sm₂O₃ 100μg of each standard solution.

4 Instruments and equipment

4.1

chromatographic column. Weigh 3g particle diameter of 180μm ~ 250μm of polychlorotrifluoroethylene powder, dried into a small beaker. Join 6mL 25% of the two - (2-ethylhexyl) phosphoric acid - heptane solution, mix well placed 24h, placed in 80 °C ~ 90 °C oven drying. use 0.1mol/L nitrate load lower packed with glass wool 25mL acid Buret bed height 13cm ~ 14cm, on top of the bed with a small glass Glass wool. Before use of hydroxylamine hydrochloride 20mLpH1.5 - sodium acetate buffer solution with 1mL/min flow rate through the column.

4.2

paper chromatography tube. diameter 20cm, 100cm tall glass cylinder with a glass cylinder tripod supporting a large glass dish Sheng Put eluent. Put a dish on the glass rod made of a triangular frame for support with a colored layer of paper. Catchy separator tube with perforated vacuum dried Cover seal, cover the hole with a separating funnel with a rubber stopper plugged separating funnel for accession eluent used. For the whole barrel is poured Lotion "steam" saturated, to be hung in the barrel a few soaked with eluent color layer paper.

4.3

colored layer of paper. coated paper with a medium-speed color and cut into 60cm, width of 7.5cm long. After immersion in 15% ammonium nitrate, immediately remove the dry preparation use. Folded from paper at the end of 6cm so anchored in the upper triangular frame. Away from the paper side 7.5cm, 1.5cm from the edge of the pencil lightly Crossed, as a coating liquid analysis reticle. 1cm from the end of the paper with two holes for hanging glass wear aggravated by hook (see Figure 1).

Figure 1 colored layer paper

4.4

Spectrophotometer.

4.5

low background beta ray measuring instrument. The gas-flow proportional counter (background count rate of not more than 3 counts/min) or other suitable low 147Pm Energy beta-ray measuring instruments (such as low background beta liquid scintillation counter).