NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB 5009.297-2023

National food safety standard - Determination of molybdenum in foods

食品安全国家标准 食品中钼的测定

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Standardization Administration of the PRC

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Foreword

This document was issued on 6 September 2023 by the State Administration for Market Regulation; Standardization Administration of the PRC and takes effect on 6 March 2024.

It is a GB standard without the /T suffix: compliance is mandatory in China.

1 Scope

GB 5009.297-2023 covers the determination of molybdenum in food by graphite furnace atomic absorption spectrometry and by inductively coupled plasma mass spectrometry. In the furnace route the digested sample is atomised and the absorbance read at 313.3 nanometres, the molybdenum content being proportional to that absorbance over a working range; the plasma route stands as the second method. The standard sets out the principle, the reagents and materials - guaranteed reagents and grade two water to GB/T 6682 - the instruments and equipment, the analysis steps, the presentation of results, the precision and further method information, with instrument reference conditions in Appendix A. Molybdenum in food is a trace element measured at trace levels, and at those levels the glassware carries more risk than the instrument does: the standard has all glassware and the polytetrafluoroethylene digestion vessels soaked overnight in nitric acid solution, rinsed repeatedly with tap water and finally with water, because whatever a previous sample left behind will be reported as molybdenum in this one. A mandatory national food safety standard, used by inspection laboratories and by makers of formula and fortified foods who have to substantiate a declared molybdenum figure.

This standard specifies the methods for the determination of molybdenum in foods by

using graphite furnace atomic absorption spectrometry and inductively coupled plasma mass spectrometry.

This standard applies to the determination of molybdenum in food.

Method I - Graphite furnace atomic absorption spectrometry

2 Principle

After digestion treatment, the sample is atomized in a graphite furnace, and the absorbance value is measured at 313.3 nm. Within a certain concentration range, the absorbance value of molybdenum is proportional to the molybdenum content, and the external standard method is used for quantitative analysis.

3 Reagents and materials

Unless otherwise stated, the reagents used in this method are guaranteed reagents and the water is secondary water specified in GB/T 6682.

3.1 Reagents

3.1.1 Nitric acid (HNO_3).

3.1.2 Perchloric acid ($HClO_4$).

3.1.3 Calcium chloride ($CaCl_2$).

3.2 Reagent preparation

3.2.1 Nitric acid solution (5:95, volume fraction): Measure 50 mL of nitric acid, slowly add it to 950 mL of water, and mix well.

3.2.2 Calcium chloride solution (1.00 g/L): Weigh 0.100 g of calcium chloride, dissolve it in a small amount of water, then make the volume up to 100 mL with the nitric acid solution (5:95, volume fraction), and mix well.

3.2.3 Nitric acid solution (1:5, volume fraction): Measure 100 mL of nitric acid, slowly add it to 500 mL of water, and mix well.

3.3 Standard product

Ammonium molybdate ($H_8MoN_2O_4$, CAS number: 13106-76-8): purity of >=99.98%, or a standard solution with national authentication and a Reference Material Certificate.

3.4 Preparation of standard solutions

3.4.1 Molybdenum standard stock solution (1000 mg/L): Accurately weigh 2.0430 g (accurate to 0.0001 g) of ammonium molybdate, dissolve it in 50 mL of water, transfer it to a 1000 mL volumetric flask, add the nitric acid solution (5:95, volume fraction) to make the volume up to the mark, and mix well. This solution can be stored refrigerated for 12 months.

3.4.2 Molybdenum standard intermediate solution (10.0 mg/L): Accurately draw 1.00 mL of molybdenum standard stock solution (1000 mg/L) into a 100 mL volumetric flask, add the nitric acid solution (5:95, volume fraction) to the mark, and mix well. This solution can be stored and refrigerated for 1 month.

3.4.3 Molybdenum standard solution (1.00 mg/L): Accurately pipette 10.0 mL of molybdenum standard intermediate solution (10.0 mg/L) into a 100 mL volumetric flask, add the nitric acid solution (5:95, volume fraction) to the mark, and mix well. Prepare the solution fresh just before use.

3.4.4 Molybdenum standard series working solution: Take 0 mL, 0.10 mL, 0.25 mL, 0.50 mL, 1.00 mL, and 2.50 mL of molybdenum standard solution (1.00 mg/L) into 100 mL volumetric flasks, add nitric acid solution (5:95, volume fraction) to the mark, and mix well. The mass concentrations of this molybdenum standard series solution are 0 ug/L, 1.00 ug/L, 2.50 ug/L, 5.00 ug/L, 10.0 ug/L and 25.0 ug/L. Prepare the solution fresh just before use.

NOTE: The mass concentration of molybdenum in the standard series working solution can be

determined based on the sensitivity of the instrument and the actual content of molybdenum in the sample.

4 Instruments and equipment

All glassware and polytetrafluoroethylene digestion inner tanks need to be soaked in nitric acid solution (1:5, volume fraction) overnight, rinsed repeatedly with tap water, and finally rinsed with water.

4.1 Atomic absorption spectrometer: equipped with a graphite furnace atomizer and a

Weigh 0.3 g~0.8 g of the sample (accurate to 0.001 g) or accurately pipette 0.500

mL~3.00 mL of the liquid sample into the microwave digestion tank, add 5 mL~10 mL of nitric acid, and digest the sample according to the operating steps of the microwave digestion system. Please refer to Table A.1 in Appendix A for reference conditions of the instrument. After cooling, take it out and reduce the acid to about 1 mL on the electric hot plate at 150 degrees C~170 degrees C. Remove the digestion tank, cool it, and transfer the

digestion liquid to a 10 mL or 25 mL volumetric flask. Wash the digestion tank 2 to 3 times with a small amount of water. Combine the washing liquid in the volumetric flask and make the volume up to the mark with water, mix well for later use. Do a blank test at the same time.

5.2.2 Wet digestion method

Weigh 0.5 g~3.0 g of the sample (accurate to 0.001 g) or accurately pipette 0.500 mL~5.00 mL of the liquid sample into the digestion tube, add 10 mL of nitric acid and 0.5 mL of perchloric acid, and digest on the adjustable electrothermal furnace (Reference conditions: rise the temperature to 120 °C and hold for 0.5 h~1 h, rise the temperature to 180 °C and hold for 2 h~4 h, and rise the temperature to 200 °C~220 °C until digestion is complete). If the digestive fluid is brown, add a small amount of nitric acid, and digest until white smoke is emitted; if the digestive fluid is still brown, repeatedly add a small amount of nitric acid to make the digestive fluid colorless and transparent or slightly yellow, and reduce the acid to about 1 mL, then take the digestive tube out. After cooling, transfer the digestive fluid to a 10 mL or 25 mL volumetric flask, wash the digestive tube 2 to 3 times with a small amount of water, combine the washing fluids in the volumetric flask, make to volume up to the mark with water, mix well for later use. Do a blank test at the same time. An Erlenmeyer flask can be used also to perform wet digestion on an adjustable electric hot plate according to the above operation method.

5.2.3 Pressure tank digestion method

Weigh 0.2 g~1.0 g of the sample (accurate to 0.001 g) or accurately pipette 0.500 mL~5.00 mL of the liquid sample into the digestion tank, add 5 mL~10 mL of nitric