Determination of Mercury in Milk Powder by HG-AFS

Mercury is measured in milk powder by means of H_2O_2-HNO_3 extraction followed by atomic fluorescence measurement.

1.Materials and methods of equipment and reagents

1.1 equipment and reagents
AI3300 Atomic fluorescence spectroscopy;
Water bath with heater;
Adjustable micro pipettes (1mL, 100ul, 50ul);
Volumetric flask (10mL);
Concentrated HNO_3;
30% H_2O_2;
Reserved mercury standards solution (1000ug/m_L = 1000ppm),
working mercury standards (0, 0.2, 0.5, 1.0, 2.0 ppb);
$SnCl_2$ solution:
Add 100gram $SnCl_2$ in 100 mL concentrated HCl, water bath to totally dissolved, add water to 1000 mL.
High pure argon (>99.99%)
High pure distilled water.

2 Method

Accurately weight 1.00g milk powder in a 50 ml beaker, add a small amount of distilled water to moisten, add 1m**L** H_2O_2 and wait 5 min, then add 5m_L HNO_3, mix well, wait 20 min until the reaction stops. Place the beaker in a water bath and heat for 1 hour. Move the flask away from the water bath and let it cool down. Transfer all contents to a 10mL volumetric flask; add distilled water to make the total volume 10mL. At the same time, prepare the blank solution in the same way.

3. Instrument parameters

Carrier gas	300mL/min
Shield gas	800mL/min
HCL current	30mA
PMT voltage	360V
Integration time	6 s
Pump speed	40 r/min
Reducing reagent solution	10% $SnCl_2$ in 10% HCl (V/V)

4. Results

This method gives:
Detection limit: 0.3ppb,
Recovery rate: 95~110%
Relative standard deviation: 2~5%