National Institute of Standards of the People's Republic of China

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Determination of iron，magnesium and manganese in foods

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Preface

This standard institute GB/T 12396-1990 " Determination mehhod of iron，magnesium and manganese in foods ".
Combine with GB/T 12396-1990, this standard's main changes as follows:
---Change the name to "Determination of iron，magnesium and manganese in foods ".
---according to GB/T 20001. 4-2001 "Rules for drafting standards, Part 4: Methods for chemical analysis". Change the structure of the original standard.
This standard is proposed and managed by the Ministry of Health of the People's Republic of China.
This standard is drafted by the Chinese Academy of Preventive Medicine Nutrition and Food Hygiene Institute.
This standard's main writer: ZhouXingHan, door JHF, wang guangya.

The original standard issued in 1990 for the first time, this is the first fix statins.

GB/T 5009.90-2003

Determination of iron，magnesium and manganese in foods

1. Scope

This standard stimulates the spectrophotometry by atomic absorption to determine the iron ,magnesium and manganese in food.

This standard applies to iron, magnesium and the determination of manganese in the food .

This method detection: iron: 0.2 p g/ml magnesium: 0.05 p g/mL manganese: 0.1 j .g/mL.

2. Principle

The wet sample digestion, guide people atomic absorption spectrophotometer, is initiated by flame atomic, iron, magnesium, manganese respectively absorbs 248. 3 nm,

285. 2 nm, 279.5 nm resonance line, the uptake and their content is proportional to the standard series more quantitative.

3. Reagent

3.1 Nitric acid

3.2 hydrochloric acid

3.3 high chlorine acid

3.4 Mixed acid digestion fluid: Nitric acid+high chlorine acid=4+1

3.5 0.5 mol/L Nitric acid solution :get 32 ML hydrochloric acid,plus Deionized water and dilute by 1000ML.

3.6 Standard solution

iron ,magnesium and manganese's standard solution:take Iron, magnesium metal, manganese metal（Purity: ≥99.99%）is about 1.0000g,or oxide which is including 10000g purity metal.Seperately add nitric acid to solute and move into three 1000ml volumetric flask,add 0.5 mol/L nitric acid solution to dilution to scale.Stored in polyethylene bottle,4 C to save. In the three solutions,each is equal to 1mg Iron,Mg,Mn.

3.7 The standard application solution

Iron,Mg,Mn standard application solution,see table 1.

Tabel 1 Preparation for standard application solution

Element	Density of standard solution/ (ug/ml)	The quantity of drain standard solution/ml	Dilution Volume (volumetric flask) / mL	Density of standard application solution/ (ug/mL)	Dilute solution
Iron	1000	10.0	100	100	0.5 mol/L nitric acid solution
Mg	1000	5.0	100	50	
Mn	1000	10.0	100	100	

Iron,Mg,Mn starndard application solution configure,stored in polyethylene bottle,4 C saved.

4.Instrument

All the glass instrument has been soaked many hours by vitriol-potassium dichromate solutions,and then wash fully by washing powder,and repeat wash by water,finally,use the deionized water to wash or dry,then can to be used.

4.1 Commonly used lab equipment.

4.2 Atomic absorption spectrophotometer

5.Analysis Steps

5.1 Sample processing

5.1.1 Specimen preparation:It should prevent all kinds of pollution in the sample preparation process for the analysis of trace elements.The equipment,like electric grinder,meat grinder,homogenizer and beating crusher etc.,must be stainless steel.The instrument must is glass and polyethylene products.

Fresh wet sample (like vegetable,fruit,fresh fish,fresh meat etc.) is washed by water,wash fully by deionized water. Dry powder sample (flour,milk powder),get the sample and then put into the container to seal and save,prevent the pollution of ash and water in the air.

5.1.2 Sample Digestion: Weight the uniform sample exactly 0.5g – 1.5g,wet sample 2.0g-4.0g,drinking ect. Liquid sample 5.0g-10.0g to 250ml beaker in tall form,add the mixed acid digestion fluid 20ml – 30ml,put the watch-glass.Put on the electric hot plate or electric sand bath to heat.If it do not digest well and the acid liquor is little,and then add some ml mixed acid digestion fluid,go on to heat to digest,until to be colorless transparent.Then add some mL water to heat to remove the left nitric acid.When the liquid in the beaker is near 2mL to 3mL,take down and cooling.Use the deionized water to wash and move to the 10 mL graduated test tube,add the water to the graduatation.

Take the mixed acid digestion fluid which is the same quantity with digested sample,and test as above operation.

5.2 Testing.

Equipped with different concentration series standard diluents by Iron,Mg,Mn standard application liquid,the test way pls see the table 2,Operation parameters determination see table 3.

Tabel 2 The way of different concentration series standard diluents

Element	**Density of standard solution/(ug/ml)**	**The quantity of drain standard solution/ml**	**Dilution Volume (volumetric flask) / mL**	**Dilute solution**
Iron	100	0.5	100	0.5 mol/L nitric acid solution
		1		
		2		
		3		
		4		
Mg	50	0.5	500	
		1		
		2		
		3		
		4		
Mn	100	0.5	200	
		1		
		2		
		3		
		4		

Table 3 Operation parameters determination

Element	Wavelength/ nm	Light source	Flame	Standard concentration range	Dilute solution
Iron	248.3	ultraviolet	Air-Acetylene	0.5-4.0	0.5 mol/L nitric acid solution
Mg	285.2	ultraviolet		0.05-1.0	
Mn	279.5	ultraviolet		0.25-2.0	

Other experiment condition:Instrument slit,the flow of air and acetylene,lamp height,element lamp current etc. moves to the best state as the explanation instruments.

5.3 The result calculation.

Drawing the standard curve as the each concentration series standard solution and the corresponding spectrophotometry.

According to the standard curve,test the sample liquid and reagent blank liquid,and check the concentration value(C & C_0),and calculate as the fomula (1).

$$X = \frac{(c - c_0) \times V \times f \times 100}{m \times 1\,000} \quad \cdots\cdots(1)$$

In the folula:

X--- The purity of element in the sample,the unit is mg/100g;

c--- The purity of element in the sample liquid (Checked by the standard curve),the unit is ug/ml;

c_0--- The purity of the element in the blank sample liquid (Checked by the standard curve),the unit is ug/ml;

V--- The capacity sample volume,the unit is mL;

f--- Dilution fator;

m--- The quantity of sample,the unit is g.

The calculate result is to be the decimal places.

6.Degree of precision

Under the repeat conditions,geting the absolute differences of two independetnt determination result,should not be more than 10% of arithmetic mean value.