SN

中华人民共和国出入境检验检疫行业标准

SN/T 2918—2011

出口食品中亚硫酸盐的检测方法 离子色谱法

Determination of sulfite in food for export—Ion chromatography method

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前　言

本标准按照 GB/T 1.1—2009 给出的规则起草。

本标准由国家认证认可监督管理委员会提出并归口。

本标准起草单位：中华人民共和国海南出入境检验检疫局。

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出口食品中亚硫酸盐的检测方法 离子色谱法

1 范围

本标准规定了出口食品中亚硫酸盐残留量的离子色谱检测方法。

本标准适用于白砂糖、饼干、果脯、虾肉、柠檬茶饮料、啤酒、淀粉、葡萄、辣椒、白萝卜、魔芋精粉中亚硫酸盐残留量的检测。

2 规范性引用文件

下列文件对于本文件的应用是必不可少的。凡是注日期的引用文件，仅注日期的版本适用于本文件。凡是不注日期的引用文件，其最新版本(包括所有的修改单)适用于本文件。

GB/T 601 化学试剂 标准滴定溶液的制备

3 方法提要

3.1 辛辣类食品、高蛋白食品、高吸水膨胀性食品：在密闭容器中对样品进行酸化，在氮气流的保护下蒸馏，释放出其中的二氧化硫，释放物用甲醛溶液吸收，将吸收液用配有电导检测器的离子色谱仪测定，外标法定量。

3.2 除3.1以外的食品：用碱将食品中结合型的亚硫酸释放出来，与甲醛生成稳定的羟甲基磺酸，经ENVI-CARB活性碳小柱除去提取液中的色素，石油醚除去提取液中的油脂，用配有电导检测器的离子色谱仪测定，外标法定量。

4 试剂和材料

除特殊说明之外，所有试剂均为分析纯，所用水均为超纯水(电阻率为18.2 MΩ·cm)。

4.1 无水碳酸钠：基准试剂。

4.2 氢氧化钠：优级纯。

4.3 石油醚：色谱纯。

4.4 甲醛：37%。

4.5 磷酸：≥85%。

4.6 氢氧化钠溶液(1.0 mol/L)：称取4.0 g氢氧化钠，溶解，定容至100 mL。

4.7 磷酸溶液：1+1(体积比)。

4.8 Na_2Co_3-NaOH混合溶液：Na_2Co_3为8 mmol/L、NaOH为2.5 mmol/L。称取0.848 g无水碳酸钠溶解，并吸取2.5 mL 1.0 mol/L的氢氧化钠(4.6)于容量瓶中，定容至100 mL。

4.9 亚硫酸钠标准品(sodium sulfite，分子式：Na_2SO_3，CAS编号：7757-83-7)：含量需根据亚硫酸钠纯度校正，校正方法参见附录A。

4.10 二氧化硫标准储备溶液(1 000 μg/mL):准确称取 0.196 9 g 亚硫酸钠溶解,并吸取 2.0 mL 甲醛于容量瓶中,定容至 100 mL。

4.11 二氧化硫标准稀释液(100 μg/mL):吸取二氧化硫标准储备溶液(4.10)10.0 mL,加入 2.0 mL 甲醛,用水定容至 100 mL。

4.12 二氧化硫标准工作曲线溶液:分别取二氧化硫标准稀释液(4.11)0.2 mL、0.5 mL、1.0 mL、2.0 mL、3.0 mL、4.0 mL、6.0 mL 于 100 mL 的容量瓶中,加入 2.0 mL 甲醛,用水定容至刻度,该标准工作曲线浓度为 0.2 μg/mL、0.5 μg/mL、1.0 μg/mL、2.0 μg/mL、3.0 μg/mL、4.0 μg/mL、6.0 μg/mL。

4.13 ENVI-CARB 石墨化碳黑小柱:0.25 g,或相当者。

4.14 水相滤膜:0.2 μm。

5 仪器和设备

5.1 离子色谱仪:配电导检测器。

5.2 离心机:转速不低于 9 000 r/min。

5.3 组织捣碎机。

5.4 涡旋振荡器。

5.5 天平:感量分别为 0.01 g 和 0.000 1 g。

5.6 pH 计。

5.7 全玻璃蒸馏器。

5.8 超滤器:截留相对分子质量 10 000,使用容量为 4 mL 样品杯。

6 试样制备和保存

6.1 试样制备

6.1.1 水果蔬菜类

取葡萄、果脯、白萝卜、辣椒等水果及蔬菜样品至少 500 g,或去皮、去壳、去根、去冠、去茎(不可水洗),将可食部分切碎后,用组织捣碎机将样品加工成浆状,混匀后,均分为两份作为试样,分装入洁净盛样袋内,密闭并标识。

6.1.2 饼干类

取饼干类样品至少 300 g,用粉碎机粉碎并通过 2.0 mm 圆孔筛,混匀,均分成两份作为试样,分装入洁净盛样袋内,密闭并标识。

6.1.3 肉及肉制品

取肉及肉制品至少 500 g,切碎后用组织捣碎机将样品加工成浆状,混匀,均分成两份作为试样,分装入洁净盛样袋内,密闭并标识。

6.1.4 淀粉、魔芋精粉、白砂糖类

6.1.4.1 取淀粉、魔芋精粉样品至少 500 g,充分混匀后,通过 2.0 mm 圆孔筛,均分成两份作为试样,

分装入洁净盛样袋内，密闭并标识。

6.1.4.2 取有代表性白砂糖样品至少 500 g，充分混匀后，均分成两份作为试样，分装入洁净盛样袋内，密闭并标识。

6.1.5 啤酒、柠檬茶饮料类

取啤酒、柠檬茶饮料类样品至少 500 g，混匀后，均分成两份作为试样，分装入洁净容器内，密闭并标识。

6.2 试样保存

6.2.1 饼干类、淀粉、魔芋精粉、白砂糖类、啤酒、柠檬茶饮料类、水果、蔬菜等试样在 4 ℃保存；肉和肉制品等试样在－18 ℃保存。

6.2.2 在制样过程中，应防止样品受到污染或发生残留物含量的变化

7 测定步骤

7.1 提取

7.1.1 啤酒、白砂糖、淀粉、果脯、饼干样品

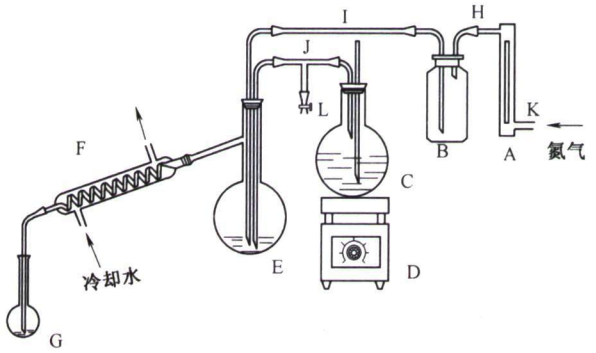
准确称取 2.5 g 均匀试样(精确至 0.001 g)置于 50 mL 的具塞刻度塑料离心管中，以少量水润湿，加入 1.0 mol/L 氢氧化钠溶液(4.6)1.0 mL，摇匀，加入 1.0 mL 甲醛，以水稀释至 25 mL。在涡旋振荡器上混匀 5 min，以 9 000 r/min 离心 30 min。上清液备用。

7.1.2 柠檬茶饮料、葡萄等酸性样品

准确称取 2.5 g 均匀试样(精确至 0.001 g)于 50 mL 具塞刻度塑料离心管中，加入 15 mL 水和 1.0 mol/L 氢氧化钠溶液 1.0 mL，摇匀，加入 1.0 mL 甲醛，摇匀，以 1.0 mol/L 氢氧化钠溶液(4.6)调节稀释液的 pH 大于 11，摇匀，以水稀释至 25 mL。在涡旋振荡器上混匀 5 min，以 9 000 r/min 离心 30 min。上清液备用。

7.1.3 辣椒、虾、白萝卜、魔芋精粉等辛辣类食品、高蛋白食品、高吸水膨胀性的样品

按图 1 所示，连接测定装置。K 管通入氮气，F 管通入冷却水，在 1 000 mL 的圆底烧瓶 C 中加入 500 mL 水，1 000 mL 的圆底蒸馏烧瓶 E 中加入 50 mL 水，在 200 mL 的茶色容量瓶 G 中加入的吸收液为 8 mL 甲醛和 7 mL 水，冷凝管下端应插入吸收液中。称取粉碎均匀的样品 10 g(精确至 0.001 g)置于圆底蒸馏烧瓶 E 中，快速加入 20 mL 磷酸溶液(4.7)，随即盖上瓶塞。接通 N_2 保护，控制其流量为 500 mL/min～2000 mL/min，经缓冲瓶 B、弯管 I 通入圆底蒸馏烧瓶 E；打开电炉加热瓶 C，使瓶内溶液保持沸腾，产生的水蒸气由 T 型管 J 通入圆底蒸馏烧瓶 E，T 型管下端连接一段带有螺旋夹 L 的乳胶管，打开螺旋夹 L，可以及时放掉蒸汽冷凝形成的水滴；待馏出液达到容量瓶 G 的刻度线时停止接收，混匀后供离子色谱仪测定。



A ——流量计；
B ——缓冲瓶；
C ——圆底烧瓶；
D ——电炉；
E ——圆底蒸馏烧瓶；
F ——冷凝管；
G ——容量瓶；
H、I——弯管；
J ——T 型管；
K ——直管；
L ——螺旋夹。

图 1 水蒸气蒸馏法测定二氧化硫的装置

7.2 净化

7.2.1 饼干等含油脂较多的样品

取出上清液(7.1.1)10 mL 于另一 50 mL 具塞塑料离心管中，加入 10 mL 石油醚，在涡旋振荡器上混匀 1 min，以 9 000 r/min 离心 10 min。弃去上层有机相，再加入 10 mL 的石油醚，重复提取一次。弃去上层有机相，收集下清液。

7.2.2 果脯、葡萄、柠檬茶饮料等含色素较多的样品

取上清液(7.1.1、7.1.2) 5 mL 过 ENVI-CARB 石墨化碳黑小柱(以 5 mL 水预淋洗)，调整流速在 1.5 mL/min 左右，弃去前 3 mL 样品流出液，收集后 2 mL 样品溶液于具塞玻璃管中备用。

7.2.3 超滤法去除样品提取液中的水溶性大分子

将 7.1.1、7.2.1、7.2.2 中收集液经 0.2 μm 的水相滤膜过滤后，注入 5.8 超滤器样品杯中，于 9 000 r/min 下离心 30 min 进行超滤，超滤液供离子色谱仪测定。

注 1：如样品中的二氧化硫含量高，超出标准工作曲线的浓度范围，可减少样品的取样量或增加其稀释倍数。

注 2：由于亚硫酸盐容易氧化成硫酸盐，因此样品和标准溶液都应是新鲜配制的，并减少暴露在空气中的时间。样品和标准溶液在甲醛稳定液中的稳定时间是 24 h。样品开封后应尽快分析。

7.3 离子色谱测定

7.3.1 色谱条件

7.3.1.1 色谱柱：AS 9-HC 高容量阴离子分离柱，4 mm×250 mm(带 AG9-HC，4 mm×50 mm 保护柱)，或性能相当的离子色谱柱。

7.3.1.2 流动相：8 mmol/L Na_2CO_3－2.5 mmol/L NaOH。

7.3.1.3 流速：1.0 mL/min。

7.3.1.4 抑制器：4 mm 阴离子抑制器；外加水抑制模式，抑制电流 50 mA。

7.3.1.5 检测器：电导检测器，检测池温度为 30 ℃。

7.3.1.6 进样量：进样 25 μL(可根据样液中二氧化硫含量进行调整)。

7.3.2 测定

根据样液中二氧化硫含量情况，选定浓度相近的标准工作溶液，标准工作溶液和待测样液中二氧化硫的响应值均应在仪器检测的线性范围内。标准工作溶液和样液等体积穿插进样测定。在上述色谱条件下二氧化硫的保留时间约为 16.6 min，标准品的色谱图参见图 B.1。

8 结果计算和表述

按式(1)计算样品中二氧化硫的残留含量：

$$X=\frac{A\times c\times V}{A_s\times m} \qquad \cdots\cdots(1)$$

式中：

X ——试样中二氧化硫的残留含量，单位为毫克每千克(mg/kg)；

A ——样液中二氧化硫的峰面积；

c ——标准工作液中二氧化硫的浓度，单位为微克每毫升(μg/mL)；

V ——样液最终定容体积，单位为毫升(mL)；

A_s——标准工作液中二氧化硫的峰面积；

m ——最终样液所代表的试样质量，单位为克(g)。

若结果以亚硫酸钠计时，除以系数 0.508。

9 测定低限、回收率

9.1 测定低限

本方法的测定低限为 4.0 mg/kg。

9.2 回收率

二氧化硫添加浓度及回收率的实验数据见表 1。

表 1 二氧化硫添加浓度及回收率的实验数据

样品名称	添加水平 mg/kg	回收率范围 %	样品名称	添加水平 mg/kg	回收率范围 %
淀粉	4.0	81.0～94.5	果脯	4.0	86.2～101.5
	10.0	85.6～96.6		10.0	82.1～97.3
	30.0	90.7～94.0		350.0	96.5～100.1
啤酒	4.0	87.0～97.8	葡萄	4.0	81.5～96.5
	10.0	81.3～94.0		10.0	85.7～95.9
	30.0	89.9～94.3		50.0	84.6～94.0

表 1（续）

样品名称	添加水平 mg/kg	回收率范围 %	样品名称	添加水平 mg/kg	回收率范围 %
白萝卜	4.0	89.0～105.8	饼干	4.0	87.8～101.2
	10.0	89.0～105.2		10.0	84.4～96.3
	500.0	85.7～89.1		100.0	92.3～96.2
摩芋精粉	4.0	87.0～107.5	辣椒	4.0	90.2～107.8
	10.0	86.7～102.0		10.0	88.2～102.1
	500.0	93.5～97.3		500.0	90.0～93.5
柠檬茶饮料	4.0	80.5～99.5	虾	4.0	84.0～99.5
	10.0	86.1～101.8		10.0	81.3～96.5
	50.0	91.0～94.4		100.0	91.1～95.1
白砂糖	4.0	82.5～101.8			
	10.0	87.6～98.7			
	100.0	90.8～94.6			

附 录 A
（资料性附录）
亚硫酸钠纯度的测定

A.1 方法提要

以间接碘量法测定亚硫酸钠的含量。碘与亚硫酸钠发生氧化还原反应，以淀粉作指示剂，再以硫代硫酸钠滴定过剩的碘，测定亚硫酸钠的含量。

A.2 试剂和材料

A.2.1 碘。

A.2.2 碘化钾。

A.2.3 硫代硫酸钠。

A.2.4 浓盐酸：36.5%。

A.2.5 碘溶液(0.1 mol/L)：称取 13.5 g 碘，加入 36 g 碘化钾和 50 mL 水，溶解后加入 3 滴盐酸及适量水稀释至 1 000 mL，置于阴凉处，密闭，避光保存。

A.2.6 硫代硫酸钠标准溶液(0.1 mol/L)：按 GB/T 601 制备并标定。

A.2.7 淀粉指示剂(1%)：称取 1 g 的可溶性淀粉，用少许水调成糊状，缓缓倾入 100 mL 沸水中。随加随搅拌，煮沸，放冷备用，此溶液临用时现配。

A.3 滴定

称取约 0.25 g(精确至 0.001 g)亚硫酸钠于盛有 0.1 mol/L 碘溶液(A.2.5)50 mL 的碘量瓶中，在室温下放置 5 min，加入 1 mL 浓盐酸，摇匀，立即用 0.1 mol/L 的硫代硫酸钠标准溶液(A.2.6)滴定过剩的碘至淡黄色，加入 0.5 mL 淀粉指示剂(A.2.7)，继续滴定至无色。同时做试剂空白试验。

A.4 计算

按照式(A.1)计算亚硫酸钠的含量：

$$X=\frac{c\times(V_1-V_2)\times 63.02}{m}\times 100 \quad \cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots\cdots(A.1)$$

式中：

X ——亚硫酸钠的含量，%；

c ——硫代硫酸钠标准溶液浓度，单位为摩尔每升(mol/L)；

V_1 ——试剂空白消耗硫代硫酸钠标准溶液的体积，单位为毫升(mL)；

V_2 ——加入亚硫酸钠消耗硫代硫酸钠标准溶液的体积，单位为毫升(mL)；

63.02 ——每毫升 1 mol/L 硫代硫酸钠溶液相当的亚硫酸钠的毫克数；

m ——亚硫酸钠的质量，单位为毫克(mg)。

附 录 B
（资料性附录）
亚硫酸盐标准品的离子色谱图

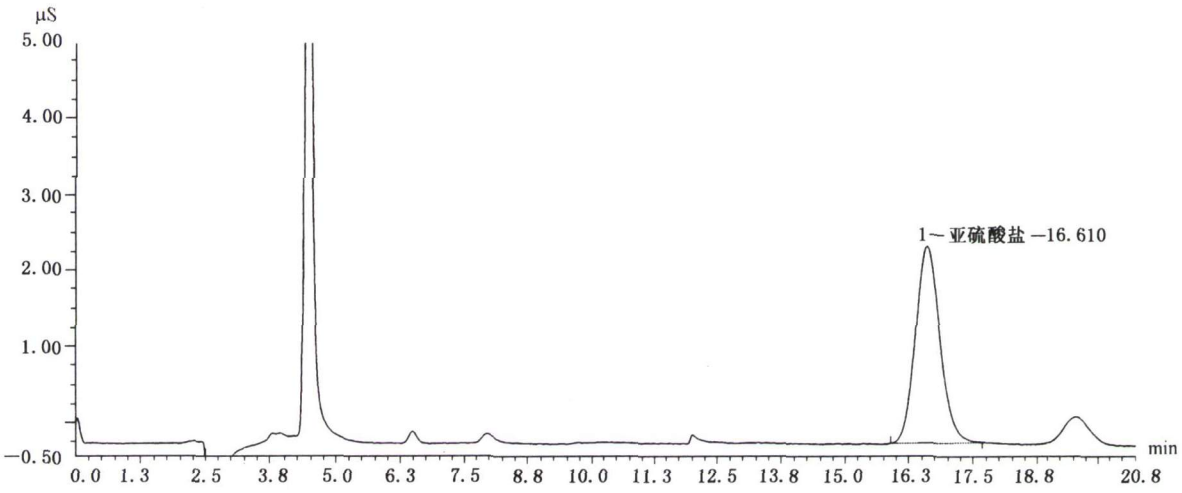


图 B.1 亚硫酸盐标准品的离子色谱图

Foreword

This standard was drafted in accordance with the GB/T 1.1—2009.

The standard was proposed by and is under the charge of the Certification and Accreditation Administration of the People's Republic of China.

The standard was drafted by Hainan Entry-Exit Inspection and Quarantine Bureau of the People's Republic of China.

The standard was mainly drafted by Huang Huiling, Wang Yujian, Zhuo Haihua, Liu Hongzhuan, Ji Shaofan, Xu Li, Zeng Min, Lin Zhengfeng.

Determination of sulfite in food for export—Ion chromatography method

1 Scope

This standard specifies the method for the determination and comfirmation of sulfite in foods for export by ion chromatography method.

This standard is applicable to the determination and confirmation of sulfite in granulated sugar, biscuit, preserved fruit, shrimps, lemonade, beer, starch, grape, chilli, radish, Konjak refined powder.

2 Quoted normative documents

The following documents are necessary for this standard. For dated references, only dated editions shall apply to this standard. For undated references, the latest edition of the normative document (including subsequent amendments) referred to applies.

GB/T 601 Chemical reagent—Preparatoins of standard volumetric solutions

3 Principle

3.1 Piquant, high-protein, super absorbent and expanding food: The sample was acidified in airproof container and distilled under the protection of nitrogen flow to release sulfur dioxide. Then sulfur dioxide reacted with formaldehyde. The derivation was determined by ion chromatography equipped with conductance detector, quantified by external standard method.

3.2 Food not included in 3.1: The combined sulfurous acid was released with alkali from the sample and reacted with formaldehyde to form hydroxymethyl sutfonic acid. The pigment and fat were removed with ENVI-CARB cartridge and petroleum ether respectively. Then the derivation was determined by ion chromatography equipped with conductance detector, quantified by external standard method.

4 Reagents and materials

Unless otherwise specified, the reagents used should be analytical pure. And the water should be ultra-pure grade water with 18.2 MΩ · cm resistivity.

4.1 Anhydrous sodium carbonate: Reference reagent.

4.2 Sodium hydroxide: Guaranteed reagent.

4.3 Petroleum ether: HPLC grade.

4.4 Formaldehyde: 37%.

4.5 Phosphoric acid: ≥85%.

4.6 Sodium hydroxide solution (1.0 mol/L): Weigh 4.0 g sodium hydroxide and dissolve it. Transfer it into 100 mL volumetric flask and dillnte to volume.

4.7 Phosphoric acid solution: 1+1 (V/V).

4.8 Na_2CO_3-NaOH mixed solution(8 mmol/L Na_2CO_3, 2.5 mmol/L NaOH): Weigh 0.848 g anhydrous sodium carbonate and dissolve it. Then mix it with 2.5 mL sodium hydroxide(4.6) and transfer them into 100 mL volumetric flask and dilute to volume.

4.9 Sodium sulfite standard (Na_2SO_3, CAS No.: 7757-83-7): The calibration of its purity should refer to annex A.

4.10 Sulfur dioxide storage solution (1 000 μg/mL): Weigh accurately 0.196 9 g sodium sulfite and dissolve. Then mix it with 2.0 mL formaldehyde and transfer them into 100 mL volumetric flask and dillute to volume.

4.11 Sulfur dioxide standard solution (100 μg/mL): Draw sulfur dioxide storage solution (4.10) 10.0 mL and 2.0 mL formaldehyde (4.4) into 100 mL volumetric flask, mix thoroughly and dillute to volume.

4.12 Sulfur dioxide standard working solution: Draw sulfur dioxide standard solution (4.11) 0.2 mL, 0.5 mL, 1.0 mL, 2.0 mL, 3.0 mL, 4.0 mL, 6.0 mL and 2.0 mL formaldehyde (4.4) into 100 mL volumetric flask, mix thoroughly and dillute to volume. The concentration of sulfur dioxide are 0.2 μg/mL, 0.5 μg/mL, 1.0 μg/mL, 2.0 μg/mL, 3.0 μg/mL, 4.0 μg/mL, 6.0 μg/mL respectively.

4.13 ENVI-CARB cartridge: 0.25 g or equivalent.

4.14 Inorganic filter membrane: 0.2 μm.

5 Apparatus and equipment

5.1 Ion chromatography, equipped with conductance detector.

5.2 Centrifuge: ≥9 000 r/min.

5.3 Tissue blender.

5.4 Vortex mixer.

5.5 Balance: With 0.01 g and 0.000 1 g sensivity.

5.6 pH meter.

5.7 Vitreous distiller.

5.8 Superfiltrater: To cutoff the molecular with mass above 10 000, the sample cup capacity should be 4 mL.

6 Sample preparation and storage

6.1 Sample preparation

6.1.1 Fruits and vegetable

For fruits and vegetable, such as grape, preserved fruit, radish, chilli, about 500 g representative sample should be taken from all samples. After remove the peel, shell, root, coronal, stem (wash forbidden!), the edible parts should be blended with a tissue blender and grinded into paste. Mix thoroughly, the paste should be divided into two equal portions. Each portion of the test sample is put into a clean container, then sealed and labeled.

6.1.2 Biscuit

About 300 g representative sample should be smashed and screened through a sieve with 2.0 mm aperture. Then the sample should be divided into two equal portions. Each portion of the test sample is put into a clean container, then sealed and labeled.

6.1.3 Meat and meat products

About 500 g representative sample should be blended with a tissue blender and grinded into paste.

Mix thoroughly, the paste should be divided into two equal portions. Each portion of the test sample is put into a clean container, then sealed and labeled.

6.1.4 Starch, Konjak refined powder and white granulated sugar

6.1.4.1 For starch, Konjak refined powder, about 500 g representative sample should be mixed thoroughly and screened through a sieve with 2.0 mm aperture. Then the sample should be divided into two equal portions. Each portion of the test sample is put into a clean container, then sealed and labeled.

6.1.4.2 For white granulated sugar, about 500 g representative sample should be mixed thoroughly and divided into two equal portions. Each portion of the test sample is put into a clean container, then sealed and labeled.

6.1.5 Beer and lemonade

About 500 g representative sample should be mixed thoroughly and divided into two equal portions. Each portion of the test sample is put into a clean container, then sealed and labeled.

6.2 Sample storage

6.2.1 Samples including biscuits, starch, Konjak refined powder, white granulated sugar, beer, lemonade, fruits and vegetable should be stored in 4 ℃ refrigerator. Meat and meat products should be stored in −18 ℃ refrigerator.

6.2.2 Sample contamination or changes in residue content shall be avoided during the process of sample preparation.

7 Analytical procedure

7.1 Extraction

7.1.1 Beer, white granulated sugar, starch, preserved fruit, biscuits

Accurately weigh 2.5 g test sample (accurate to 0.001 g) into a 50 mL polypropylene centrifuge scale tube with cap and add a little water to wet the sample. Add 1.0 mL sodium hydroxide solution(4.6) into the tube and mix. Then add 1.0 mL formaldehyde and dillute to 25 mL. Shake it on the vortex mixer for 5 min and centrifuge at 9 000 r/min for 30 min. The supernatant is collected for the next step.

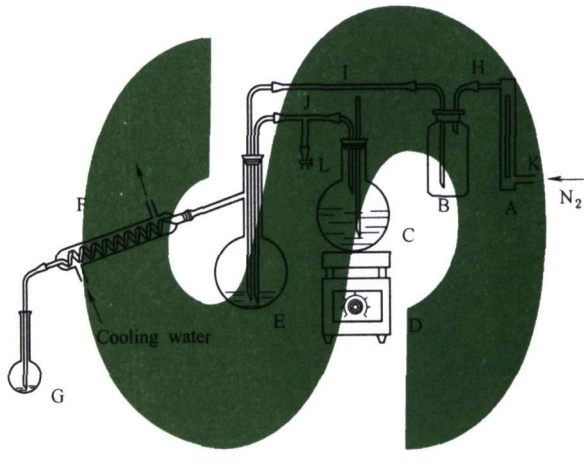
7.1.2 Acidic sample (including lemonade and grape)

Accurately weigh 2.5 g test sample (accurate to 0.001 g) into a 50 mL polypropylene centrifuge scale tube with cap, add 15 mL water and 1.0 mL sodium hydroxide solution(4.6). Then add 1.0 mL

formaldehyde and mix. Adjust the pH of the mixture above 11 with sodium hydroxide solution(4.6) and dillute to 25 mL. Shake it on the vortex mixer for 5 min and centrifuge at 9 000 r/min for 30 min. The supernatant is collected for the next step.

7.1.3 Piquant, high-protein, super absorbent and expanding food (including chilli, shrimps, radish, Konjak refined powder)

Connect the equipment according to figure 1. K pipeline is utilized to aerating nitrogen. F pipeline is for cooling water. Add 500 mL and 50 mL water into round bottom flask C and E respectively. Add 8 mL formaldehyde and 7 mL water in 200 mL brown glass volumetric flask G. The condensate pipe should be dipped under water. Pipe I and J should be dipped below the fluid. Accurately weigh 10.0 g test sample (accurate to 0.001 g) in round bottom flask E. Promptly add 20 mL phosphoric acid solution(4.7) and fasten the cap. Turn on the nitrogen flow and control its speed between 500 mL/min～2 000 mL/min. The nitrogen flow from buffer bottle B, bent pipeline I to round bottom flask E. Heat the round bottom flask C to keep the water boiling. Then the vapor formed was led to round-bottom distilling flask E from a T-shape tube J. The T-shape tube J was connected with a alatex tube equipped a screw clamp L. When the screw clamp L was opened, the waterdrop condensed can be exhausted in time. Stop receive the distillate when it reaches 200 mL volume. Mix the solution and determine with ion chromatography.



A —flow meter;
B —buffer bottle;
C —round bottom flask;
D —electric oven;
E —round bottom flask;
F —condensate pipeline;
G —brown glass volumetric flask;
H、I —bent pipeline;
J —T-shape tube;
K —straight pipeline;
L —screw clamp.

Figure 1—The vapor-distillation equipment for sulfur dioxide determination

7.2 Cleanup

7.2.1 Sample with abundant fat including biscuit

Draw 10 mL supernatant (7.1.1) into another 50 mL polypropylene centrifuge scale tube with cap, add 10 mL petroleum ether and mix on the vortex mixer for 1 min. Centrifuge at 9 000 r/min for 10 min. Abandon the upper organic layer and extract with another 10 mL petroleum ether. Collect the underlayer for superfiltration.

7.2.2 Sample with abundant pigment including preserved fruit, grape, lemonade

ENVI-CARB cartridge is preconditioned with 5 mL water. Load 5 mL the extract solution (7.1.1、7.1.2) to the cartridge and adjust the flow rate to 1.5 mL/min. Abandon 3 mL the initial elution and collect 2 mL the subsequent elution to a glass tube with cap.

7.2.3 Superfiltration procedure for removing the water-soluble macromolecular

After filtrate the elution in 7.1.1、7.2.1、7.2.2 with 0.2 μm membrane filter, the solution should be injected into the sample cup of 5.8 and centrifugated at 9 000 r/min for 30 min. The superfiltrate is acquired for determination.

Note 1: If the sulfur dioxide exceeds the range of the standard curve, the sample amount should be reduced or the multiple should be increased.

Note 2: Because of the oxidation of sulfite to sulfate, the sample and the standard solution should be fresh. Furthermore, the exposed time of the solution in air should be shortened. The sample and the standard solution can be stored in formaldehyde for 24 h. The sample unsealed should be determined as soon as possible.

7.3 Determination by ion chromatography

7.3.1 Chromatography operating conditions

7.3.1.1 Chromatography column: AS 9-HC, 4 mm × 250 mm (with AG 9-HC, 4 mm × 50 mm guard column).

7.3.1.2 Mobile phase: 8 mmol/L Na_2CO_3 – 2.5 mmol/L NaOH.

7.3.1.3 Flow rate: 1.0 mL/min.

7.3.1.4 Suppressor: 4 mm anion suppressor; external water suppression mode, suppression current is 50 mA.

7.3.1.5 Detector: Conductance detector, the temperature of detector cell is 30 ℃.

7.3.1.6 Injection volume: Injection volume should be 25 μL(could adjust according to the concentration of sulfur dioxide).

7.3.2 Determination

Choose the standard solution close to the sample according to the concentration of sulfur dioxide. The responses of the standard solution and the sample should be in the linearity of the instrument. The standard solution and the sample are injected in turn with equal-volume. Under the above condition, the retention time of sulfur dioxide is about 16.6 min. The chromatogram is showed in annex B.

8 Calculation and expression of the result

The concentration of sulfur dioxide can be calculated according to formula (1):

$$X = \frac{A \times c \times V}{A_s \times m} \qquad \cdots\cdots(1)$$

Where

X —the concentration of sulfur dioxide in the sample solution, mg/kg;

A —the peak area of sulfur dioxide in the sample solution;

c —the concentration of sulfur dioxide in the standard solution, μg/mL;

V —the final volume of the sample solution, mL;

A_s—the peak area of sulfur dioxide in the standard solution;

m —the corresponding mass of test sample in the final sample solution, g.

The result should be divided by 0.508 when it is calculated as sodium sulfite.

9 Limit of quantification and recovery

9.1 Limit of quantification

The limit of determination of this method is 4.0 mg/kg.

9.2 Recovery

The fortifying concentrations of sulfur dioxide and its corresponding recovery data are showed as table 1.

Table 1—The results of the sulfur dioxide addition concentration and recovery data

Sample	Addition concentration mg/kg	Recovery data %	Sample	Addition concentration mg/kg	Recovery data %
Strach	4.0	81.0～94.5	Lemonade	4.0	80.5～99.5
	10.0	85.6～96.6		10.0	86.1～101.8
	30.0	90.7～94.0		50.0	91.0～94.4
Beer	4.0	87.0～97.8	White granule sugar	4.0	82.5～101.8
	10.0	81.3～94.0		10.0	87.6～98.7
	30.0	89.9～94.3		100.0	90.8～94.6
Preserved fruit	4.0	86.2～101.5	Biscuit	4.0	87.8～101.2
	10.0	82.1～97.3		10.0	84.4～96.3
	350.0	96.5～100.1		100.0	92.3～96.2
Grape	4.0	81.5～96.5	Chilli	4.0	90.2～107.8
	10.0	85.7～95.9		10.0	88.2～102.1
	50.0	84.6～94.0		500.0	90.0～93.5
Radish	4.0	89.0～105.8	Shrimp	4.0	84.0～99.5
	10.0	89.0～105.2		10.0	81.3～96.5
	500.0	85.7～89.1		100.0	91.1～95.1
Konjak refined powder	4.0	87.0～107.5			
	10.0	86.7～102.0			
	500.0	93.5～97.3			

Annex A
(Informative annex)
Determination of the purity of sodium sulfite

A. 1 Principle

Determination of the purity of sodium sulfite is according to indirect iodometry. Iodine can oxidize sodium sulfite. And the reaction can be indicted by starch. The excessive iodine is titrated with sodium thiosulfate. Then the purity of sodium sutfite can be specified.

A. 2 Reagents and materials

A. 2. 1 Iodine.

A. 2. 2 Potassium iodine.

A. 2. 3 Sodium thiosulfate.

A. 2. 4 Concentrated hydrochloric acid: 36. 5%.

A. 2. 5 Iodine solution(0. 1 mol/L): Weigh 13. 5 g iodine, 36 g potassium iodide, dissolve with 50 mL water. Add 3 drops hydrochloric acid and transfer to 1 000 mL volumetric flask. Then dilute the solution to the volume and store the solution away from light.

A. 2. 6 Sodium thiosulfate(0. 1 mol/L): Prepare it according to GB/T 601.

A. 2. 7 Starch indicator(1%): Mix 1 g soluble starch with a little water to paste. Transfer it into 100 mL boiling water with stirring. Heat it to boil and cool it. The indicator should be prepared before use.

A. 3 Titration

Weigh accurately 0. 25 g(accurate to 0. 001 g)sodium sulfite into a iodineflask with 50 mL iodine solution (A. 2. 5). Leave it in room temperature for 5 min. Add 1 mL concentrated hydrochloric acid and shake. Then titrate the excessive iodine with sodium thiosulfate (A. 2. 6) to faint yellow. Add 0. 5 mL starch indicator (A. 2. 7) and titrate to colourless. The reagent blank sample should be carried out simultaneously.

A.4 Calculation

The purity of sodium sulfite can be calculated according to formula(A.1):

$$X = \frac{c \times (V_1 - V_2) \times 63.02}{m} \times 100 \quad \cdots\cdots (A.1)$$

Where

X —the purity of sodium sulfite, %;

c —the concentration of sodium thiosulfate standard solution, mol/L;

V_1 —the sodium thiosulfate volume consumed by the reagent blank sample, mL;

V_2 —the sodium thiosulfate volume consumed by the solution with sodium sulfite, mL;

63.02 —the corresponding sodium sulfite milligrame of 1 mL sodium thiosulfate solution;

m —the mass of sodium sulfite, mg.

Annex B
(Informative annex)
The ion chromatogram of sodium sulfite

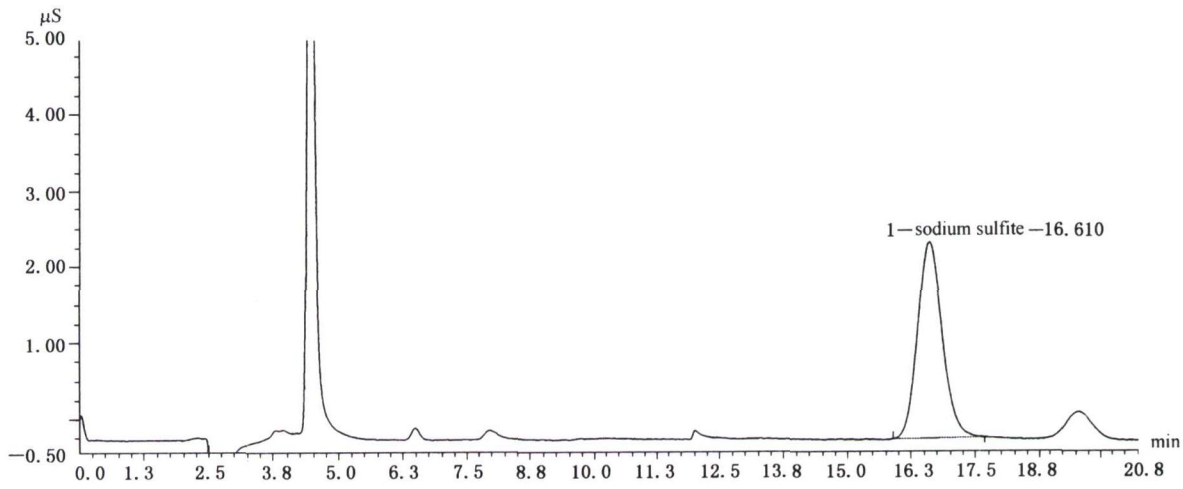


Figure B. 1—The ion chromatogram of sodium sulfite

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