

SN

中华人民共和国出入境检验检疫行业标准

SN/T 4783—2017

出口食品中低聚三果糖、低聚四果糖、 低聚五果糖的测定 高效液相色谱法

Determination of kestose, nistose and fructofuranosylnystose in export food—
High performance liquid 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：中华人民共和国广东出入境检验检疫局、国际检验检疫标准与技术法规研究中心、中华人民共和国天津出入境检验检疫局、华南农业大学。

本标准主要起草人：韦晓群、郑璇、宋志刚、娄婷婷、杜斯航、吴志航、张玉文、黄维龙、陈文锐、刘青、易蓉、王志元、梁瑞婷、卢珍珠。

出口食品中低聚三果糖、低聚四果糖、 低聚五果糖的测定 高效液相色谱法

1 范围

本标准规定了婴幼儿配方米粉、米乳粉以及奶粉中低聚蔗糖三糖、四糖、五糖等3种低聚果糖的测定方法。

本标准适用于婴幼儿配方米粉、米乳粉以及奶粉中低聚蔗糖三糖、四糖、五糖的测定。

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 6682 分析实验室用水规格和试验方法

3 方法提要

应用既定 pH 的乙酸水和甲醇溶液沉淀去除蛋白和淀粉后,样品中的低聚果糖再用 β -半乳糖酶处理去除半乳糖和乳糖,过膜上机测试,外标法定量。

4 试剂和材料

除另有说明外,所用试剂均为分析纯,水采用 GB/T 6682 规定的二级水。

4.1 甲醇(CH_4O),分析纯。

4.2 乙酸($\text{C}_2\text{H}_4\text{O}_2$),分析纯。

4.3 乙腈($\text{C}_2\text{H}_3\text{N}$),色谱纯。

4.4 磷酸氢二钠(Na_2HPO_4),分析纯。

4.5 柠檬酸($\text{C}_6\text{H}_8\text{O}_7$),分析纯。

4.6 低聚果糖标准品:蔗糖三糖($\text{C}_{18}\text{H}_{32}\text{O}_{16}$, CAS No.: 470-69-9),蔗糖四糖($\text{C}_{24}\text{H}_{42}\text{O}_{21}$, CAS No.: 13133-07-8),蔗糖五糖($\text{C}_{30}\text{H}_{52}\text{O}_{26}$, CAS No.: 59432-60-9)3种标准品,纯度 $\geq 99\%$ 。

4.7 蔗糖三糖、蔗糖四糖、蔗糖五糖混合标准储备液溶液:分别称取蔗糖三糖、蔗糖四糖、蔗糖五糖的标准品 2.5 g,一起用水溶解并定容于 50 mL 容量瓶,配成浓度分别为 50 000 mg/mL 的蔗糖三糖、蔗糖四糖、蔗糖五糖混合标准溶液,2℃~4℃保存,有效期为 3 个月。

4.8 蔗糖三糖、蔗糖四糖、蔗糖五糖混合标准中间溶液:分别准确量取 50 000 mg/mL 的单标溶液(4.7) 5.0 mL,用水定容到 25 mL,配成浓度分别为 10 000 mg/mL 的蔗糖三糖、蔗糖四糖、蔗糖五糖混合标准溶液。

4.9 蔗糖三糖、蔗糖四糖、蔗糖五糖标准混合工作溶液:分别移取 10 000 mg/mL 蔗糖三糖、四糖、五糖标准中间溶液(4.8) 5 mL、2.5 mL、1 mL、0.5 mL、0.1 mL,用纯水定容至 10 mL 配成 5 000 mg/mL、2 500 mg/mL、1 000 mg/mL、500 mg/mL、100 mg/mL 的标准工作溶液,4℃保存,可使用 3 个月。

4.10 pH 为 5.0 的 1 mol/L 磷酸氢二钠缓冲液:先称取 1.92 g 柠檬酸溶解于 10 mL 纯水中,制得

1 mol/L的柠檬酸溶液 10 mL;称取 0.71 g 磷酸氢二钠溶解与 48 mL 纯水中,然后应用 1 mol/L 的柠檬酸溶液调节磷酸氢二钠溶液 pH 至 5.0,最后定容至 50 mL。

4.11 20 U/mL β -半乳糖苷酶溶液:称取 103 U/mg 的 β -半乳糖苷酶固体 2 mg,溶解在 10.3 mL 的缓冲溶液(4.10)中,2 °C~4 °C 保存,可使用 1 个月。

5 仪器和设备

- 5.1 高效液相色谱仪,配蒸发光散射检测器和柱恒温系统。
- 5.2 流动相真空抽滤脱气装置及 0.2 μ m 或 0.45 μ m 微孔膜。
- 5.3 色谱柱:改性酰胺柱或者氨基柱,5 μ m,250 mm \times 4.6 mm(内径)或性能类似的分析柱。
- 5.4 分析天平:感量为 0.01 mg 和 0.001 g 各一台。
- 5.5 离心机:转速 $>$ 10 000 r/min。
- 5.6 涡旋混合器。
- 5.7 旋转蒸发器。
- 5.8 恒温振荡水槽。

6 测定步骤

6.1 提取

6.1.1 纯奶粉样品

称取 0.25 g 样品于 50 mL 离心管中,加入 4.73 mL 水,加入 0.07 mL 醋酸溶液调节 pH 至 4~5 之间,溶解振荡 5 min,10 000 r/min 离心 5 min,用定量滤纸过滤,加入 0.2 mL 半乳糖苷酶,于 45 °C 下(是在恒温振荡水槽中)酶解 3 h,酶解后取 1 mL 过 0.22 μ m 滤膜于进样瓶中,供上机分析。

6.1.2 纯淀粉食品

称取 0.25 g 样品于 50 mL 离心管中,加入 4.73 mL 水,加入 0.07 mL 醋酸溶液调节 pH 至 4~5 之间,然后加入 20 mL 甲醇,振荡 5 min 静置 5 min,用定量滤纸过滤,旋干滤液后,用水定容至 4.8 mL,加入 0.2 mL 半乳糖苷酶,于 45 °C 下(是在恒温振荡水槽中)酶解 3 h,酶解后取 1 mL 过 0.22 μ m 滤膜于进样瓶中,供上机分析。

6.1.3 含奶粉和淀粉食品

称取 0.25 g 样品于 50 mL 离心管中,加入 4.93 mL 水,加入 0.07 mL 醋酸溶液调节 pH 至 4~5 之间,然后加入 20 mL 甲醇,振荡 5 min 后 10 000 r/min,用定量滤纸过滤,滤液旋蒸至于,用水定容至 4.8 mL,加入 0.2 mL 半乳糖苷酶,于 45 °C 下(是在恒温振荡水槽中)酶解 3 h,酶解后取 1 mL 过 0.22 μ m 滤膜于进样瓶中,供上机分析。

6.2 测定

6.2.1 液相色谱参考条件

由于测试结果取决于所用仪器,因此不可能给出色谱分析的普遍参数。采用下列参数已被证明对测试是合适的:

- a) 色谱柱:酰胺柱或者氨基柱,5 μ m,250 mm \times 4.6 mm(内径)或性能类似的分析柱。
- b) 柱温:30 °C。

c) 流动相梯度见表 1。

表 1 流动相梯度

时间/min	流动相 A:水/%	流动相 B:乙腈/%
0	20	80
18	41	59
18.1	80	20
23	80	20
23.1	20	80
30	20	80

d) 流速:1.0 mL/min。

e) 进样量:10 μ L。

6.2.2 检测仪器

蒸发光检测器。

6.2.3 液相色谱测定

在仪器最佳工作条件下,以蔗果三糖、蔗果四糖、蔗果五糖混合标准工作溶液(4.9)浓度为横坐标,以峰面积为纵坐标,绘制标准曲线。用标准工作曲线对样品进行定量。使样品中 3 种低聚果糖的响应值在仪器测定的线性范围内。在上述色谱条件下,蔗果三糖的参考保留时间为 11.7 min,蔗果四糖参考保留时间为 13.6 min、蔗果五糖的参考保留时间为 15.1 min,3 种低聚果糖标准溶液色谱图参见附录 A 中图 A.1。

6.2.4 标准曲线的绘制

按照 6.2.1 和 6.2.2 所列的测定条件,对提取液(6.1)依次进样测定。分别以标准工作溶液中每种低聚果糖浓度为横坐标,单位以“mg/kg”表示,以对应的峰面积为纵坐标,分别绘制标准工作曲线,得到线性方程。标准溶液色谱图参见附录 A。

按式(1)计算回归参数:

$$y = a \times x + b \quad \dots\dots\dots(1)$$

式中:

y ——标准工作溶液中每种低聚果糖的峰面积;

a ——回归曲线的斜率;

x ——标准工作溶液中每种低聚果糖的浓度,单位为毫克每千克(mg/kg);

b ——回归曲线的截距。

7 结果计算和表述

试样中低聚果糖(包括低聚三果糖、低聚四果糖、低聚五果糖等)含量由色谱数据处理软件或按式(2)计算获得,计算结果需扣除空白值,并保留 3 位有效数字:

按式(2)计算试样中待测物残留量(mg/kg):

$$X = \frac{c \times V \times 100}{M \times 1\,000} \dots\dots\dots(2)$$

式中:

X —— 试样中待测物的含量,单位为毫克每千克(mg/kg);

c —— 从标准工作曲线得到的被测组分溶液的浓度,单位为毫克每升(mg/L);

V —— 样液溶液定容体积,单位为毫升(mL);

M —— 最终定容体积试样溶液所代表试样的质量,单位为克(g)。

FOS 含量 Z(mg/100 g)按式(3)计算:

$$Z = X_{\text{蔗果三糖}} + X_{\text{蔗果四糖}} + X_{\text{蔗果五糖}} \dots\dots\dots(3)$$

8 方法的检测范围和精密度

8.1 检测范围

采用本方法对奶粉中 3 种低聚果糖含量进行测定,每种低聚果糖的检测范围在 1 g/kg~70 g/kg。

8.2 精密度

采用本方法对样品进行添加回收实验,添加水平为每种低聚果糖分别添加 1 g/kg、35 g/kg、70 g/kg的回收率及精密度数据参见附录 B。

附录 A

(资料性附录)

不同样品中低聚果糖(蔗果三糖、蔗果四糖、蔗果五糖)的色谱图

不同样品中低聚果糖(蔗果三糖、蔗果四糖、蔗果五糖)的色谱图见图 A.1~图 A.3。

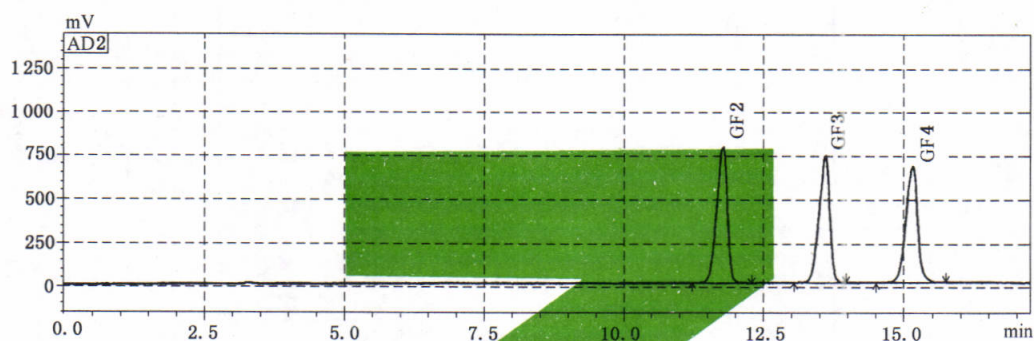


图 A.1 低聚果糖(蔗果三糖、蔗果四糖、蔗果五糖)的标准品色谱图

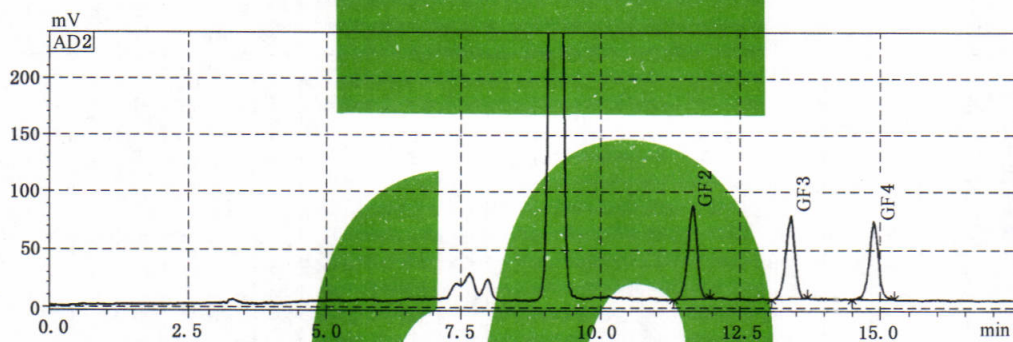


图 A.2 米粉中低聚果糖(蔗果三糖、蔗果四糖、蔗果五糖)的色谱图

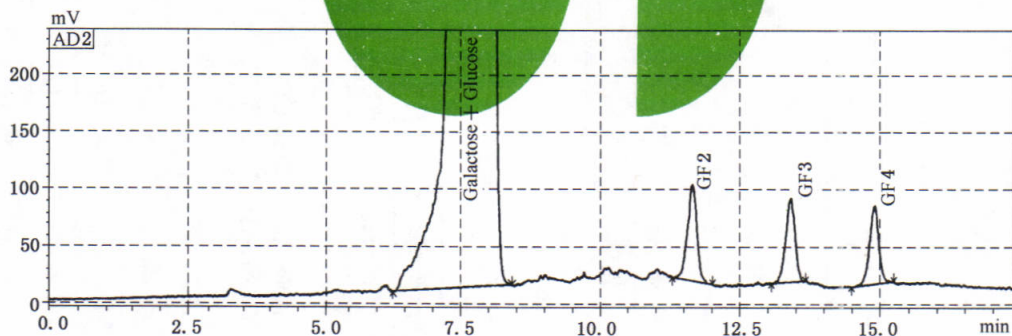


图 A.3 奶粉中低聚果糖(蔗果三糖、蔗果四糖、蔗果五糖)的色谱图

附录 B
(资料性附录)

不同基质中低聚果糖不同添加水平回收率数据

食品基质	添加水平 mg/kg	低聚果糖种类	测得值						测得平均值	回收率范围 %	RSD
			1	2	3	4	5	6			
米粉	1 000	蔗果三糖	1 010.1	1 030.1	1 005.9	1 023.8	998.1	978.3	1 007.7	98~100	1.8
		蔗果四糖	977.9	997.7	972.2	992.8	968.0	979.3	981.3	96~100	1.2
		蔗果五糖	1 157.3	1 178.9	1 160.6	1 166.7	1 136.7	1 141.3	1 156.9	100~118	1.4
		蔗果三糖	34 531.7	32 747.4	34 841.8	32 886.7	33 890.5	33 250.7	33 691.7	93~99	2.6
		蔗果四糖	31 971.1	30 366.7	32 255.3	30 556.4	31 458.0	30 997.4	31 267.6	86~92	2.4
米粉	35 000	蔗果五糖	33 120.5	31 435.6	33 416.6	31 602.2	32 550.0	32 008.9	32 355.4	88~95	2.5
		蔗果三糖	66 405.5	66 124.1	64 382.5	64 787.8	67 767.0	63 789.6	65 542.4	91~95	2.3
		蔗果四糖	63 532.7	62 964.3	62 055.7	63 351.4	64 227.8	62 367.9	63 083.3	88~91	1.3
		蔗果五糖	65 581.6	652 181.6	63 715.4	64 376.2	66 752.0	63 382.2	64 837.5	91~95	1.9
		蔗果三糖	964.5	92 527.5	919.5	926.6	939.6	962.1	939.6	91~96	2.1
奶粉	1 000	蔗果四糖	931.5	893.9	881.6	897.6	909.1	930.4	907.4	88~93	2.2
		蔗果五糖	963.5	924.3	918.4	925.8	938.6	961.2	938.6	91~96	2.1
		蔗果三糖	31 941.7	32 960.2	32 088.0	32 117.4	31 168.9	33 342.4	3 227.0	88~94	2.4
		蔗果四糖	29 841.7	30 839.2	30 137.1	30 185.4	29 061.9	31 293.5	30 226.0	84~88	2.6
		蔗果五糖	34 828.5	35 875.7	34 770.4	34 772.5	34 066.2	36 159.2	35 079.1	96~102	2.2
奶粉	70 000	蔗果三糖	63 220.5	62 855.8	67 420.5	64 718.5	6 454.7	64 814.4	64 596.1	89~96	2.5
		蔗果四糖	59 949.4	59 486.7	63 869.4	61 286.4	60 702.6	61 371.1	61 110.7	84~91	2.5
		蔗果五糖	64 850.8	64 535.1	69 190.1	66 428.6	66 462.2	66 530.1	66 332.7	92~98	2.5

Foreword

This section is written according to GB/T 1.1—2009.

This part is put forward and under centralized by the state certification and accreditation supervision and management committee.

The drafting units of this part is Inspection and quarantine technology center of Guangdong entry-exit inspection and quarantine bureau of the People's Republic of China, The center of the international inspection and quarantine standards and technical regulations of China, Inspection and quarantine technology center of Tianjin entry-exit inspection and quarantine bureau of the People's Republic of China, South China Agricultural University.

The main draftsmen of this part are: Xiaoqun Wei, Xuan Zheng, Zhigang Song, Tingting Lou, Sihang Du, Zhihang Wu, Yuwen Zhang, Weilong Hang, Wenrui Chen, Qing Liu, Rong Yi, Ruiting Liang, Zhenzhu Lu.

Note: this English version, a translation from the Chinese text, is solely for guidance.

Determination of kestose, nistose and fructofuranosyl nistose in export food— High performance liquid chromatography method

1 Scope

This standard specifies the determination of kestose, nistose and fructofuranosyl nistose of syrup and formula rice flour for infants.

This standard applies to the determination of kestose, cane fruit tetrasaccharide, cane fruit pentasaccharide of syrup and formula rice flour for infants.

2 Normative reference

The following documents for the application of this document is essential. For cited documents with dates, only the dated edition applies to this document. For undated references, the latest edition (including any amendments) applies to this document.

GB/T 6682 Analysis laboratory water specifications and test method

3 Abstract of the method

After removing protein and starch by acidified water oscillation and methanol, fructooligosaccharide in the sample was adding a given volume of beta galactosidase enzyme to remove lactose and galactose, and then followed by high performance liquid chromatography determination, quantified by external standard method.

4 Reagents and materials

Unless otherwise specified, the reagents used for the analysis are analytical, water used for the analysis was water level 2 set by GB/T 6682.

4.1 methanol (CH_4O), analytic grade.

4.2 Acetic Acid ($\text{C}_2\text{H}_4\text{O}_2$), analytic grade.

4.3 Ammonium acetate ($\text{CH}_3\text{COONH}_4$), HPLC grade.

- 4.4 Dibasic Sodium Phosphate(Na_2HPO_4), analytic grade.
- 4.5 1,2,3-Tricarboxy-2-hydroxypropane($\text{C}_6\text{H}_8\text{O}_7$), analytic grade.
- 4.6 Kestose($\text{C}_{18}\text{H}_{32}\text{O}_{16}$, CAS No.: 470-69-9), cane fruit tetrasaccharide($\text{C}_{24}\text{H}_{42}\text{O}_{21}$, CAS No.: 13133-07-8), cane fruit pentasaccharide($\text{C}_{30}\text{H}_{52}\text{O}_{26}$, CAS No.: 59432-60-9), standard, a purity of $\geq 99\%$.
- 4.7 500 00 mg/kg FOS standard stock solution; was prepared by dissolving 2.5 mg (accurate to 0.001 g). Standard substance (4.6) in water and diluting it with water to 50 mL.
- 4.8 10 000 mg/kg FOS standard intermediate solution; 2 mL 50 000 mg/kg FOS standard stock solution(4.7) diluting to 10 mL.
- 4.9 FOS Standard working solution; 5 mL, 2.5 mL, 1 mL, 0.5 mL, 0.1 mL of standard solution(4.8) were diluting it with water to 10 mL separately, to achieved 5 000 mg/kg, 2 500 mg/kg, 1 000 mg/kg, 500 mg/kg, 100 mg/kg Standard working solution, $2\text{ }^\circ\text{C} \sim 4\text{ }^\circ\text{C}$ preservation, valid for three months.
- 4.10 pH 5.0 mol/L disodium hydrogen phosphate buffer solution; first Weighing 1.92 g 1,2,3-Tricarboxy-2-hydroxypropane and dissolving in 10 mL pure water, to achieve 10 mL 1 mol/L 1,2,3-Tricarboxy-2-hydroxypropane; then taking 0.71 g disodium hydrogen phosphate and dissolving at 48 mL pure water, and then applying 1 mol/L 1,2,3-Tricarboxy-2-hydroxypropane to adjust the solution's pH to 5.0, final volume up to 50 mL.
- 4.11 20 U/mL β -galactosidase buffer solution; weighing 103 U/mg β -galactosidase 2 mg, and then dissolving in 10.3 mL buffer solution (4.10), $2\text{ }^\circ\text{C} \sim 4\text{ }^\circ\text{C}$ preservation, valid for one months.

5 Instruments and equipment

- 5.1 High performance liquid chromatography (equipped with evaporative light scattering detector and column thermostat system).
- 5.2 The mobile phase vacuum filtration degasser and 0.2 μm or 0.45 μm microporous membrane.
- 5.3 Chromatographic column; Modified Amide or amino column, 5 μm , 150 mm \times 4.6 mm or similar performance analysis.
- 5.4 Analytical balance; the amount of sense of 0.01 mg and 0.001 g each one.
- 5.5 Centrifuge.
- 5.6 Vortex mixer.
- 5.7 Rotary evaporator.
- 5.8 Termolstat water bath.

6 Measuring steps

6.1 extract

6.1.1 For pure milk

As a typical procedure, 0.25 g sample was put into a 50 mL centrifuge tube and mixed with 4.73 mL water and 0.07 mL acetic acid, vortexed for 5 minutes, following a 10-minute 10 000 r/min centrifugalization. After then, it was added 0.2 mL galactosidase, and enzymed at 45 °C for 3 h and 1 mL was filtered through a syringe filter for HPLC test.

6.1.2 For pure Starch food

For milk: As a typical procedure, 0.25 g sample was put into a 50 mL centrifuge tube and mixed with 5 mL water and 20 mL methanol, vortexed for another 5 minutes following a 10-minute 10 000 r/min centrifugalization. After the centrifugalization, filter and spin dry, dilute with water to 4.8 mL. After that, it was added 0.2 mL galactosidase, and enzymed at 45 °C for 3 h and 1 mL was transferred to an autosampler vial. After being shaken well and filtered through a syringe filter, the sample was transferred to an autosampler vial and was ready for HPLC test.

6.1.3 For milk and starch-based mix

For milk: As a typical procedure, 0.25 g sample was put into a 50 mL centrifuge tube and mixed with 4.93 mL water and 0.07 mL acetic acid, vortexed for 5 minutes. Then, 20 mL methanol was pipetted into the tube vortexed for another 5 minutes following a 10-minute 10 000 r/min centrifugalization. After the centrifugalization, filter and spin dry, dilute with water to 4.8 mL. After that, it was added 0.2 mL galactosidase, and enzymed at 45 °C for 3 h and 1 mL was transferred to an autosampler vial. After being shaken well and filtered through a syringe filter, the sample was transferred to an autosampler vial and was ready for HPLC test.

6.2 Measure

Due to the test results depends on the instrument, so it's impossible to give general parameters of chromatographic analysis. The parameters used following had been proved that is suitable for the test.

6.2.1 Liquid chromatography reference conditions

- a) Chromatographic column: Amide or amino column, 5 μm , 150 mm \times 4.6 mm or similar performance analysis.
- b) Column temperature: 30 °C.
- c) The gradient of flow phase (see Table 1).

Table 1 the gradient of flow phase

Time/min	A: water/%	B: acetonitrile/%
0	20	80
18	41	59
18.1	80	20
23	80	20
23.1	20	80
30	20	80

d) Flow rate: 1.0 mL/min.

e) Sample injection: 10 μ L.

6.2.2 Detector

Evaporative light detector, Detection limit of 100 mg/kg.

6.2.3 Liquid Chromatography

Under optimum working conditions, quantified the sample with standard curve. Concentration of Kestose, cane fruit tetrasaccharide, cane fruit pentasaccharide in standard working solution as the abscissa, unit expressed in mg/kg, with the corresponding peak area as the ordinate, standard working curve drawing respectively, linear equation is obtained. The response of kestose, cane fruit tetrasaccharide, cane fruit pentasaccharide should be within the linear measurement range of the instrument. Under the above chromatographic conditions, the reference retention time of kestose was 11.7 min, cane fruit tetrasaccharide was 13.6 min, cane fruit pentasaccharide was 15.1 min. Standard solution chromatogram of 3 kinds of fructooligosaccharide refer to appendix A.1.

6.2.4 Drawing of the standard curve

According to 6.2.1 and 6.2.2 determination conditions, measure the determination of fructooligosaccharide of the standard working solution in turn. Concentration of fructooligosaccharide in standard working solution as the abscissa, unit expressed in mg/kg, with the corresponding peak area as the ordinate, standard working curve drawing respectively, linear equation is obtained. Standard solution chromatogram refer to appendix A.

According to the type (1) the regression parameters:

$$y = a \times x + b \quad \dots\dots\dots (1)$$

Type:

y —standard working solution of each nucleotide in peak area;

a —regression curve slope;

x —standard working solution concentration of kestose, cane fruit tetrasaccharide or cane fruit or pentasaccharide, the unit is mg per liter (mg/kg);

b —the intercept of regression curve.

7 Results calculation and expression

Fructooligosaccharide content in sample by chromatography data processing software or by type (2) calculation, the calculation results to deduct blank value, and keep the three significant figures;

Calculated by (2) in the sample under test content residue (mg/kg):

$$X = \frac{c \times V \times 100}{M \times 1\,000} \dots\dots\dots (2)$$

Type:

X —the content of the object under test in the sample, the unit is mg per liter (mg/kg);

c —standard solution concentration of the object under test, the unit is mg per liter (mg/kg);

V —the capacity volume, the samples, the final units as liter (L);

M —the volume of the sample, the unit is liter (L).

The content of (3) kinds of FOS *Z* (mg/100 g):

$$Z = X_{GF2} + X_{GF3} + X_{GF4} \dots\dots\dots (3)$$

8 The method of quantitative restrictions and recovery

8.1 LOQ

Fructooligosaccharide content in milk powder determined of this method, its quantitative limit of each kind of fructooligosaccharide was 1 g/kg~70 g/kg.

8.2 Precision

Add the recycling experiment; added level of 1 g/kg, 35 g/kg, 70 g/kg using this method.

Appendix B was the data of the recovery rate of 3 kinds of fructooligosaccharide.

Appendix A

(Reference appendix)

3 kinds of fructooligosaccharide(kestose,cane fruit tetrasaccharide,cane fruit pentasaccharide)in different samples liquid chromatogram

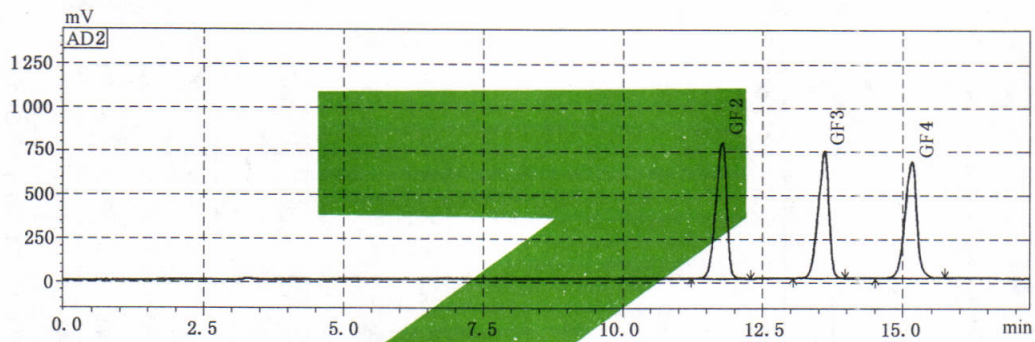


Figure A.1 Fructooligosaccharide(kestose,cane fruit tetrasaccharide,cane fruit pentasaccharide)standard substance liquid chromatogram

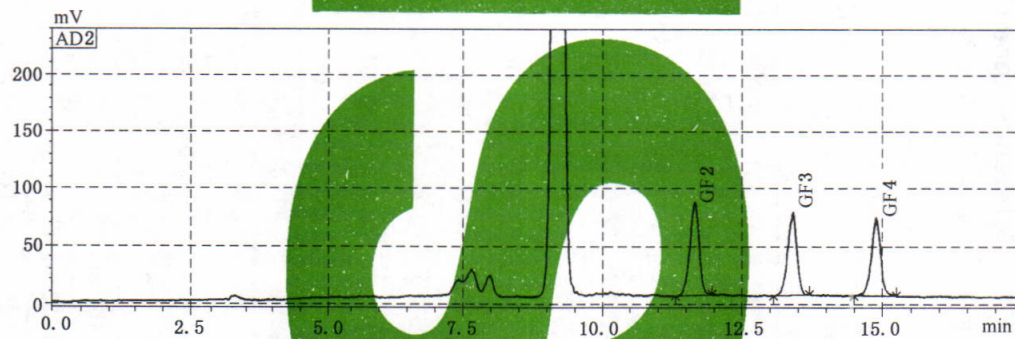


Figure A.2 starch-based samples measured 5 kinds of nucleotides liquid chromatogram

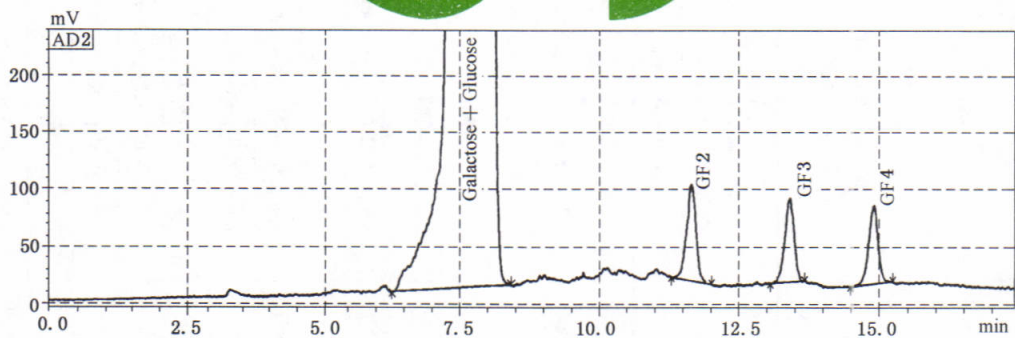


Figure A.3 Milk powder samples measured 5 kinds of nucleotides liquid chromatogram

Appendix B
(Reference appendix)

Recovery rate data of different matrix of fructooligosaccharide adding level

Typical matrix	adding level mg/kg	Fructooligosaccharide Species	The measured value mg/kg						Average mg/kg	Range recovery %	RSD
			1	2	3	4	5	6			
Starch-based infant food	1 000	kestose	1 010.1	1 030.1	1 005.9	1 023.8	998.1	978.3	1 007.7	98~100	1.8
		cane fruit tetrasaccharide	977.9	997.7	972.2	992.8	968.0	979.3	981.3	96~100	1.2
		cane fruit pentasaccharide	1 157.3	1 178.9	1 160.6	1 166.7	1 136.7	1 141.3	1 156.9	100~118	1.4
	35 000	kestose	34 531.7	32 747.4	34 841.8	32 886.7	33 890.5	33 250.7	33 691.7	93~99	2.6
		cane fruit tetrasaccharide	31 971.1	30 366.7	32 255.3	30 556.4	31 458.0	30 997.4	31 267.6	86~92	2.4
		cane fruit pentasaccharide	33 120.5	31 435.6	33 416.6	31 602.2	32 550.0	32 008.9	32 355.4	88~95	2.5
70 000	kestose	66 405.5	66 124.1	64 382.5	64 787.8	67 767.0	63 789.6	65 542.4	91~95	2.3	
	cane fruit tetrasaccharide	63 532.7	62 964.3	62 055.7	63 351.4	64 227.8	62 367.9	63 083.3	88~91	1.3	
	cane fruit pentasaccharide	65 581.6	652 181.6	63 715.4	64 376.2	66 752.0	63 382.2	64 837.5	91~95	1.9	
Milk-based infant food	1 000	kestose	964.5	92 527.5	919.5	926.6	939.6	962.1	939.6	91~96	2.1
		cane fruit tetrasaccharide	931.5	893.9	881.6	897.6	909.1	930.4	907.4	88~93	2.2
		cane fruit pentasaccharide	963.5	924.3	918.4	925.8	938.6	961.2	938.6	91~96	2.1
	35 000	kestose	31 941.7	32 960.2	32 088.0	32 117.4	31 168.9	33 342.4	3 227.0	88~94	2.4
		cane fruit tetrasaccharide	29 841.7	30 839.2	30 137.1	30 185.4	29 061.9	31 293.5	30 226.0	84~88	2.6
		cane fruit pentasaccharide	34 828.5	35 875.7	34 770.4	34 772.5	34 066.2	36 159.2	35 079.1	96~102	2.2
70 000	kestose	63 220.5	62 855.8	67 420.5	64 718.5	6 454.7	64 814.4	64 596.1	89~96	2.5	
	cane fruit tetrasaccharide	59 949.4	59 486.7	63 869.4	61 286.4	60 702.6	61 371.1	61 110.7	84~91	2.5	
	cane fruit pentasaccharide	64 850.8	64 535.1	69 190.1	66 428.6	66 462.2	66 530.1	66 332.7	92~98	2.5	

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