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Application of Fructo-oligosaccharides Quality Control Chart in Milk Powder

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Abstract The quality control chart of fructo-oligosaccharides in milk powder was established to determine whether the detection process and results are in control state. The content of fructo-oligosaccharides in milk powder control samples was determined by ion chromatography, and the quality control chart of fructo-oligosaccharides was established to analyze the controlled state. The results indicate that the median of the quality control chart is 1 613.14 mg/100 g, and the standard deviation is 85.57 mg/100 g. The new quality control points were evaluated and analyzed, and the precision changed, but the mean value did not change. Further F test was conducted to determine that the precision did not change significantly, indicating that the test was in a statistical control state, and the detection process, method and results were controlled.

Key words Quality control chart, fructo-oligosaccharides, Milk powder

0 Introduction

Fructo-oligosaccharides is also known as fruit oligosaccharides, sugarcane fruit oligosaccharides, *etc.* Fructo-oligosaccharides is a mixture of trisugars, trasaccharides, pentasaccharides and hexasaccharides[1]. Fructo-oligosaccharides is a kind of prebiotics, not absorbed by the human small intestine into the human large intestine, selectively used by bifidobacterium, is a proliferation factor of bifidobacterium and fermented to produce short chain fatty acids to play a series of physiological functions[2]. It has been reported that fructo-oligosaccharides promote the proliferation of Bifidobacterium, reduce harmful bacteria[3], reduce glucose, total cholesterol, do not cause dental caries[4-5], improve intestinal flora, reduce blood triglyceride levels[6-7], *etc.* Therefore, fructo-oligosaccharides as a beneficial factor added to infant formula milk powder, is also one of the items that dairy enterprises must test.

The quality control chart is one of the main tools for internal quality control in dairy laboratories, which can reflect whether the daily testing process is in a stable and controlled state, continuously monitor the testing process and results for abnormalities, and promptly alert if there are abnormalities, so as to take corrective or preventive measures quickly[8-9].

In this study, the determination of fructo-oligosaccharides content in infant and toddler food by GB 5009.255-2016 *National Food Safety Standard—Determination of Fructo-oligosaccharides in Foods*[10] was taken as an example to make a quality control chart, monitor whether the daily test results are stable and reliable, and provide effective basis and reference for the quality control of dairy products laboratory.

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1 Materials and methods

1.1 Reagents and materials D-fructose standard substance, DR, Germany; the fructoglycan mix enzyme, Megazyme; sodium borohydride, glacial acetic acid, sodium acetate and maleic acid (AR); fructo-oligosaccharides control samples: infant formula milk powder (Inner Mongolia Yili Industrial Group Co., Ltd.); 1.0 c cRP purification column, Thermo Fisher.

1.2 Instruments and equipment Ion chromatograph (ampere detector), Thermo Fisher; analytical balance, METTLER-TOLEDO; ultrapure water machine, Thermo Fisher, USA; electric-heated thermostatic water bath, Beijing Xingwei Instrument Co., Ltd.; spinning evaporator IKA; vortex oscillator IKA; thermostatic water bath shake bed, Shanghai Boxun Medical Biological Instrument Co., Ltd.

1.3 Methods

1.3.1 Sample preparation. Weigh 1.0 - 2.0 g of sample in a 100 mL volumetric flask, add about 50 mL of hot water at 40 - 50 ℃, shake well, dissolve fully, cool to room temperature, then filter and centrifuge. Take 200 μL of the spare sample into 10 mL plug glass tube, add 400 μL of sucrose mixed enzyme solution and shake well. After 60 min of 150 r/min shaking, add 300 μL of 10 mg/mL sodium borohydride solution, place in 40 ℃ constant temperature water bath shaking bed, shaking at 150 r/min for 30 min, take out and cool to room temperature, add 750 mL of 200 mmol/L acetic acid solution, shake well, leave for 10 min, add 100 μL of 500 μL/mL Fructan mixed enzyme solution, shake well, put in 40 ℃ constant temperature water bath shaking bed, 150 r/min shake for 30 min, take out, and cool to room temperature, set volume to scale line. The sample solution was sequentially removed through a 0.45 μm aqueous phase filter membrane and an RP purification column (or a C_{18} column), and after discarding the initial 3 mL of eluent, other eluates were collected for testing.

1.3.2 Instrument operating conditionsChromatographic column:

CarboPacTM PA 20 (150 mm × 3 mm); Ampere detector: Au working electrode, Ag/AgCl reference electrode, and standard sugar four-potential waveform; Column temperature: 30 ℃ ; Detection pool temperature: 30 ℃. ; Injection volume: 10 μL; mobile phase: A: hyperpure water; B: 250 mmol/L NaOH; C: 1.0 mol/L NaOAc&100 mmol/L NaOH; Elution method: gradient elution.

1.4 Drawing of the quality control chart Control chart was drawn and analyzed using the Excel software.

2 Results and analysis

2.1 The selection of quality control samples The matrix of the quality control samples should be as consistent as the sample to be tested, and should be stable, uniform and sufficient[11]. However, it is difficult to find the control samples that meet the above requirements for each test item, so the certified standard substance, standard solution, blank sample and sample to be tested can also be selected as the quality control sample[12-14]. The milk powder itself is stable, uniform and easy to preserve, so the infant milk powder was selected as the control sample in this study.

2.2 Establishment of quality control chart In this study, the test results of 20 control samples were collected to determine the control limit (CL), upper warning limit (UWL), upper action limit (UAL), lower warning limit (LWL), and the control limit to identify the subsequent test results. The experimental results are shown in Table 1. Quality control chart was performed using Excel software, as shown in Fig. 1. According to 8 criteria in GB / T 4091-2001[15] (*i. e.* 1 point falls outside the control line; 9 consecutive points fall on the same side of the center line; 6 consecutive points are monotonically increasing or decreasing; adjacent points in 14 consecutive points; 2 of 3 consecutive points fall outside the warning line on the same side of the center line; 4 consecutive points fall outside the auxiliary limit on the same side of the center line; 15 consecutive points fall within the upper and lower auxiliary limit; 8 consecutive points fall on both sides of the center line and none within the auxiliary limit), all points are randomly arranged, no abnormal points, and deviation phenomenon, can determine whether the detection process is stable and can be used as a control diagram for daily quality control.

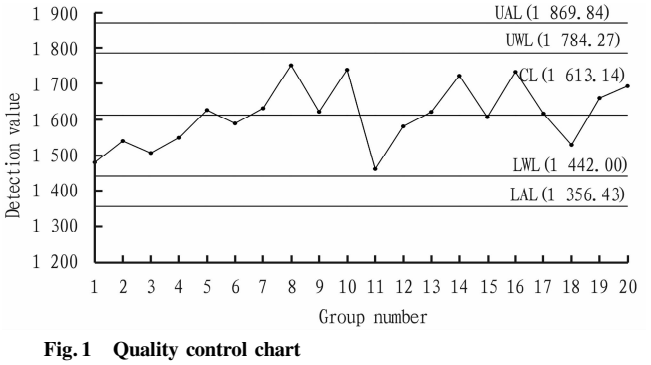

Fig. 1 Quality control chart

Table 1 The test results of 20 control samples

No.	Detection value	CL	UWL	LWL	UAL	LAL
1	1 480.2	1 613.14	1 784.27	1 442.00	1 869.84	1 356.43
2	1 540.6	1 613.14	1 784.27	1 442.00	1 869.84	1 356.43
3	1 506.8	1 613.14	1 784.27	1 442.00	1 869.84	1 356.43
4	1 549.8	1 613.14	1 784.27	1 442.00	1 869.84	1 356.43
5	1 627.0	1 613.14	1 784.27	1 442.00	1 869.84	1 356.43
6	1 589.9	1 613.14	1 784.27	1 442.00	1 869.84	1 356.43
7	1 631.8	1 613.14	1 784.27	1 442.00	1 869.84	1 356.43
8	1 750.3	1 613.14	1 784.27	1 442.00	1 869.84	1 356.43
9	1 622.4	1 613.14	1 784.27	1 442.00	1 869.84	1 356.43
10	1 738.0	1 613.14	1 784.27	1 442.00	1 869.84	1 356.43
11	1 461.8	1 613.14	1 784.27	1 442.00	1 869.84	1 356.43
12	1 581.7	1 613.14	1 784.27	1 442.00	1 869.84	1 356.43
13	1 621.9	1 613.14	1 784.27	1 442.00	1 869.84	1 356.43
14	1 721.2	1 613.14	1 784.27	1 442.00	1 869.84	1 356.43
15	1 607.7	1 613.14	1 784.27	1 442.00	1 869.84	1 356.43
16	1 732.2	1 613.14	1 784.27	1 442.00	1 869.84	1 356.43
17	1 617.3	1 613.14	1 784.27	1 442.00	1 869.84	1 356.43
18	1 529.8	1 613.14	1 784.27	1 442.00	1 869.84	1 356.43
19	1 659.7	1 613.14	1 784.27	1 442.00	1 869.84	1 356.43
20	1 692.6	1 613.14	1 784.27	1 442.00	1 869.84	1 356.43

2.3 Assessment of quality control chart After the control data is run for a period of time, the data during this period is evaluated. The purpose of the review is to continuously monitor the measurement process and the measurement results to determine whether the results are sufficiently reliable. Maintaining the stability of the control limit and the median line over a long time period is very important for the successful use of the quality control chart. The median line and control limits should not change frequently, otherwise it will be difficult to monitor the gradient of analysis quality, and are generally recommended to evaluate after each year or at least 20 new control values. This study was evaluated after adding 20 new data, as shown in Table 2, and the control chart after running 20 new data of the control data, as shown in Fig. 2.

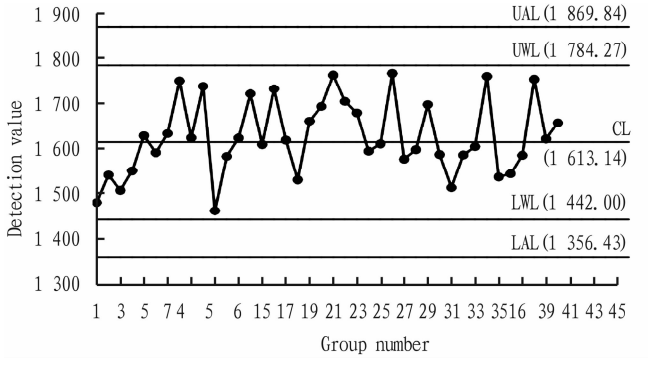

Fig. 2 Quality control chart after running 20 new data

2.3.1 Assessment of the quality of the current analysis. To calculate the number of points falling outside the warning limit, if the number of points falling outside the warning limit is greater than 6 or less than 1, it indicates that the precision of the analysis changes. As can be seen from Fig. 2, no points fall outside the warning

limit, indicating a change in precision for the 40 data points; the mean of the 40 results was calculated, compared with the mean of the initial 25 data points (median line), if the difference between the two is greater than 0.35 S, indicating a change in the mean: $0.35 \times 85.57 = 29.95$ ($S = 85.57$), $\bar{x}_{40} - \bar{x}_{20} = 1\,624.64 - 1\,613.14 = 11.5 < 29.95$, the mean did not change.

Table 2 The test results of 45 control samples

No.	Detection value	No.	Detection value
1	1 480.2	21	1 763.0
2	1 540.6	22	1 704.0
3	1 506.8	23	1 678.1
4	1 549.8	24	1 593.1
5	1 627.0	25	1 609.2
6	1 589.9	26	1 766.9
7	1 631.8	27	1 575.5
8	1 750.3	28	1 596.5
9	1 622.4	29	1 696.9
10	1 738.0	30	1 585.7
11	1 461.8	31	1 512.8
12	1 581.7	32	1 584.6
13	1 621.9	33	1 603.2
14	1 721.2	34	1 760.2
15	1 607.7	35	1 536.2
16	1 732.2	36	1 543.4
17	1 617.3	37	1 583.9
18	1 529.8	38	1 753.8
19	1 659.7	39	1 620.0
20	1 692.6	40	1 656.0

2.3.2 Assessment of control limit. Due to changes in precision, further F-tests are required for precision (F-tests are performed bilaterally, and a 95% confidence level is conventionally taken) to see if the changes are significant. First, the outliers were evaluated: from Fig.2, it can be seen that there are no outliers in 40 data; secondly, the standard deviation review (F-test) was conducted: Based on the 40 data in Table 2, the new mean and standard deviation were recalculated, $\bar{x}_{new} = 1\,624.64$ mg/kg, $S_{new} = 82.93$ mg/kg. Compare the initial standard deviation with the new standard deviation using the F-test: $F = \frac{s^2_{origina}}{s^2_{new}} = \frac{85.57^2}{82.93^2} = 1.06$. The degrees of freedom of $s^2_{origina}$ and s^2_{new} are 20 and 40, respectively, and the critical value of F is 2.07 when the critical value of F test with a confidence level of 95% (two-sided test) and a degree of freedom of 4 – 120 is found. The calculated F-number (1.06) was smaller than the look-up value (2.07), so there was no significant change in precision.

According to the above review, there is no significant change in precision or mean value, according to the control limit change as required by CNAS-GL39 9.3: the control limit can only be considered for a significant change in precision or bias, so fructo-oligosaccharides does not need to change the control limit, indicating that the control chart is running well.

3 Conclusion

The quality control chart for the detection of fructo-oligosaccharides in milk powder was established using 20 measurement data, and the quality control chart was reviewed for the newly generated 20 new data points. The results showed that the median line value of the established control chart was 1 613.14 mg/100 g, and the standard deviation was 85.57 mg/100 g. After evaluating the new quality control point, the control limit is not changed, the control chart is running well, and the detection process, method and results are controlled. The establishment and review of the control chart in this study will provide a reference for other subsequent quality control.

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