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Establishment and Evaluation of Quality Control Chart of DHA in Milk Powder

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Abstract [**Objectives**] To establish a quality control chart of DHA in milk powder and determine whether the detection process and results are in control state. [**Methods**] The content of DHA in milk powder control samples was determined by gas chromatography, and the quality control chart of DHA was established to analyze the controlled state. [**Results**] the median of the quality control chart was 23.85 mg/100 g, and the standard deviation was 1.00 mg/100 g. In the evaluation and analysis of the new quality control points, it can be seen that the point beyond the warning limit was 1, and the outlier value was not beyond the action line, and the mean value did not change, indicating that the detection is in the statistical control state, and the detection process, method and results are controlled. [**Conclusions**] The establishment and review of the control chart in this study are expected to provide a reference for other subsequent quality controls.

Key words Quality control chart, DHA, Milk powder

1 Introduction

Docosahexaenoic acid (DHA) is an essential fatty acid for the human body, belonging to w-3 polyunsaturated fatty acid[1] commonly known as brain gold. It plays an important role in maintaining human health and is a major structural fatty acid in the cerebral cortex and retina. It is particularly important for brain development, vision improvement, promoting growth and development, and enhancing human immunity[2]. Due to the inability of the human body to synthesize DHA on its own, it needs to be absorbed through food intake. With the enhancement of people's awareness of health care, the application of DHA in food is becoming more and more widespread. Many countries around the world (including China) have approved DHA as a nutritional food additive and formulated corresponding additive standards. DHA is a natural component in breast milk and is crucial for the intellectual development and visual function of infants[3]. However, it is not found in milk, which has attracted attention from expert groups around the world. In 1994, the Food and Agriculture Organization of the United Nations and the World Health Organization (FAO/WHO) officially recommended the addition of DHA to infant formula. After artificial feeding formula milk powder is fortified with DHA, the growth and development of infants can achieve similar effects as breastfeeding[4]. Therefore, DHA is added to infant formula milk powder as a nutritional indicator, which is also one of the indicators 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[5-6].

In this study, the determination of DHA content in infant and toddler food by *National Food Safety Standard - Determination of Fatty Acids in Foods* (GB 5009.168-2016)[7] 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.

2 Materials and methods

2.1 Reagents and materials Thirty seven fatty acids, NU-CHEK, USA; hydrochloric acid (GR), sodium hydroxide, petroleum ether, ether, ammonia, 95% ethanol and absolute ethanol (AR), Methanol, n-hexane and n-heptane (chromatographic pure), DHA control samples: Infant formula milk powder (Inner Mongolia Yili Industrial Group Co., Ltd.).

2.2 Instruments and equipment Gas chromatograph (hydrogen flame ionization detector), Shimzu (China) Co., Ltd.; analytical balance METTLER-TOLEDO; Low-temperature, high-speed centrifuge, Sigma Company, German; ultrapure water machine Thermo Fisher, USA; electric-heated thermostatic water bath, Beijing Xingwei Instrument Co., Ltd.; Spinning evaporator IKA; vortex oscillator IKA.

2.3 Methods

2.3.1 Sample preparation. The samples were prepared with reference to the hydrolysis extraction method in the standard *Determination of Fatty Acids in Foods* (GB 5009.168-2016) with slight modifications. We weighed 10 g of milk samples, added 2 mL of ammonia, and perform hydrolysis in a (65 ± 1) ℃ water bath for 15 min to 20 min. The hydrolyzed samples were added 10 mL of

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ethanol and mixed. Then added 25 mL of diethyl ether and shaken for 1 min. 25 mL of petroleum ether was added, blocked and shaken for 1 min, centrifuged for 5 min at 600 rpm, and the organic layer was transferred into a rotary evaporation bottle. In the second extraction, 5 mL of ethanol, 15 mL of ether were added and shaken for 1 min. Added 15 mL of petroleum ether, plugged and shaken for 1 min, centrifuged for 5 min at 600 rpm, and transferred into the same rotary evaporation bottle. Concentrated to dry using a rotary evaporator. Applied 10 mL to n-hexane, absorbed 2 mL of sample into 10 mL centrifuge tube, added 200 μL potassium hydroxide methanol solution, covered violently for 30 sec and stood to clear, added about 1 g sodium hydrogen sulfate, shaken violently to neutralize potassium hydroxide. After salt precipitation, removed the upper solution to the upper bottle for testing.

2.3.2 Instrument operating conditions. Chromatographic column: CP-SiL 88 (100 m × 0.25 mm × 0.250 μm); temperature programming: 100 ℃ (3 min) − 3 ℃/min − 130 ℃ (1 min) −4 ℃/min − 230 ℃ (27 min); intake mouth temperature: 260 ℃; sampling method: split sampling; split ratio: 1 ∶ 100; Injection volume: 1 μL; detector temperature: 280 ℃.

2.4 Plotting of the quality control chart Control chart was plotted and analyzed using the Excel software.

3 Results and analysis

3.1 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[8]. 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[9-10]. 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.

3.2 Establishment of quality control chart In this study, the test results of 25 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 standard (*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 the detection process is stable, can be used as a control diagram for daily quality control.

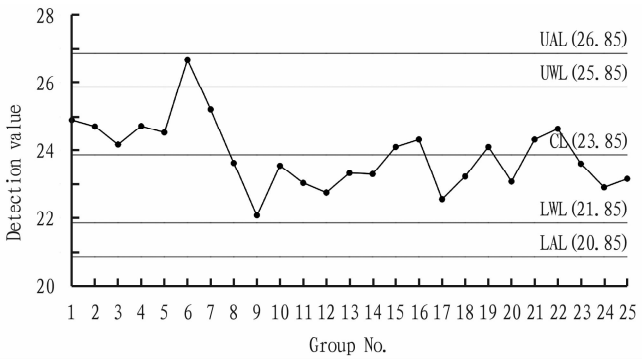

Fig. 1 Quality control chart of 25 control samples

Table 1 Test results of 25 control samples

No.	Detection value	CL	UWL	LWL	UAL	LAL
1	24.89	23.85	25.85	21.85	26.85	20.85
2	24.70	23.85	25.85	21.85	26.85	20.85
3	24.16	23.85	25.85	21.85	26.85	20.85
4	24.71	23.85	25.85	21.85	26.85	20.85
5	24.52	23.85	25.85	21.85	26.85	20.85
6	26.66	23.85	25.85	21.85	26.85	20.85
7	25.20	23.85	25.85	21.85	26.85	20.85
8	23.61	23.85	25.85	21.85	26.85	20.85
9	22.07	23.85	25.85	21.85	26.85	20.85
10	23.53	23.85	25.85	21.85	26.85	20.85
11	23.02	23.85	25.85	21.85	26.85	20.85
12	22.74	23.85	25.85	21.85	26.85	20.85
13	23.31	23.85	25.85	21.85	26.85	20.85
14	23.28	23.85	25.85	21.85	26.85	20.85
15	24.09	23.85	25.85	21.85	26.85	20.85
16	24.31	23.85	25.85	21.85	26.85	20.85
17	22.55	23.85	25.85	21.85	26.85	20.85
18	23.21	23.85	25.85	21.85	26.85	20.85
19	24.09	23.85	25.85	21.85	26.85	20.85
20	23.06	23.85	25.85	21.85	26.85	20.85
21	24.31	23.85	25.85	21.85	26.85	20.85
22	24.63	23.85	25.85	21.85	26.85	20.85
23	23.59	23.85	25.85	21.85	26.85	20.85
24	22.89	23.85	25.85	21.85	26.85	20.85
25	23.14	23.85	25.85	21.85	26.85	20.85

3.3 Assessment of quality control chart After the control data were 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.

Table 2 Test results of 45 control samples

No.	Detection value	No.	Detection value	No.	Detection value
1	24.89	16	24.31	31	22.70
2	24.70	17	22.55	32	23.80
3	24.16	18	23.21	33	23.00
4	24.71	19	24.09	34	22.50
5	24.52	20	23.06	35	23.90
6	26.66	21	24.31	36	24.40
7	25.20	22	24.63	37	22.40
8	23.61	23	23.59	38	22.90
9	22.07	24	22.89	39	23.90
10	23.53	25	23.14	40	23.80
11	23.02	26	23.49	41	22.50
12	22.74	27	24.50	42	23.00
13	23.31	28	23.86	43	24.30
14	23.28	29	23.95	44	23.10
15	24.09	30	22.59	45	24.80

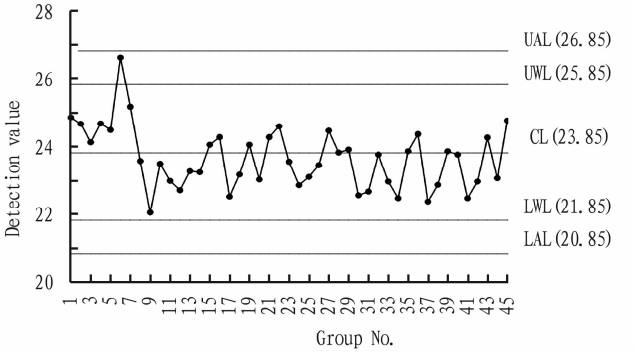

Fig. 2 Quality control chart after running 20 new data

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 the Fig. 2, 1 point falls outside the warning limit and did not exceed the action line outlier, indicating no change in precision. The mean of the 45 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: $0.35 \times 1.00 = 0.35$ ($S = 1.00$), $\bar{x}_{25} - \bar{x}_{45} = 23.85 - 23.68 = 0.17 < 0.37$, the mean did not change. According to the above review, there is no significant change in precision or mean value, according to the control limit change as required by 9.3 in CNAS-GL39: the control limit can only be considered for a significant change in precision or bias, so DHA does not need to change the control limit, indicating that the control map is running well.

4 Conclusions

Twenty five determination data were used to establish the quality control chart of DHA in milk powder, and the quality control chart was reviewed for 20 new data points. The results show that the median line value of the established control chart was 23.85 mg/100 g, and the standard deviation was 1.00 mg/100 g. After evaluating the new quality control point, the control limit was not change, 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 are expected to provide a reference for other subsequent quality controls.

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