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MAFF VALIDATED METHODS FOR THE ANALYSIS OF FOODSTUFFS

No. V 14

ICE-GLAZE ON QUICK FROZEN PRAWNS

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1. Scope and Field of Application

The method is designed to determine the ice-glaze content of quick frozen cooked and peeled prawns.

2. Definition

Content of ice-glaze: the percentage weight of ice-glaze as determined by the method specified.

3. Principle

The sample is thawed by immersion in tap-water at 27°C for a set time, during which it is gently agitated. It is then drained in a sieve and weighed. The weight loss is assumed to be loss of ice-glaze.

4. Reagents

None

5. Apparatus

5.1 Analytical balance

5.2 Sieve: Clean and dry, of diameter 200 mm and nominal aperture size 2.8 mm (conforming to the requirements of ISO R565), or of aperture size 2.38 mm (conforming to the requirements of US No 8 Standard Screen).

5.3 Water bath: a vessel containing tap-water at 27 ± 1°C equal in weight to 8 times the weight of sample taken (**6.1**).

6. Procedure

6.1 Place the sample in a freezer of temperature -18°C ± 2°C and allow to equilibrate. For analysis, remove the sample from low temperature storage, open immediately and accurately weigh in g to one decimal place (m_0).

6.2 Transfer the weighed portion to the water bath (**5.3**) and leave it in the water for two min. with occasional gentle stirring.

6.3 Empty the contents of the water bath into a sieve (**5.2**); incline the sieve at an angle of about 20° and allow to drain for two minutes.

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6.4 Remove the sample from the sieve and reweigh. Let the final weight in g, to one decimal place, be m_1.

7. COSHH

Analysts are reminded that appropriate hazard and risk assessments required by the Control of Substances Hazardous to Health Regulations, 1988 (See "Control of Substances Hazardous to Health - Approved Code of Practice, Control of Substances Hazardous to Health Regulations, 1988") must be made before using this method.

8. Expression of Results

The ice-glaze content of the original sample, expressed as a percentage by weight, is given by:

$$\% \text{ ice-glazed content} = 100 \times (m_0 - m_1) / m_0$$

where:

m_0 is the initial frozen weight taken (**6.1**);

m_1 is the observed deglazed weight (**6.4**).

9. Reference

9.1 GC Hodson, MJ Scotter and R Wood, J. Assoc. Publ. Analysts, 1989,**27**, 85-108.

9.2 Ministry of Agriculture, Fisheries and Food, Food Safety Directorate, MAFF Validated Methods for the Analysis of Food, Introduction, General Considerations and Analytical Quality Control, J. Assoc. Publ. Analysts, 1992, **28**, 11-16

APPENDIX 1

Analytical Quality Control

General principles of analytical quality control are outlined in protocol V.0 of the series[2].

A1 Repeatability

The absolute difference between two test results obtained under repeatability conditions should not be greater than the repeatability, r, deduced from the collaborative trial data summarised in Tables 1 and 2. Overall, r may be taken to be 3.8% glaze at levels between 15% and 30% glaze. This corresponds to a relative standard deviation of repeatability (coefficient of variance of repeatability), RSD_r, of 3 - 6%. The analysis of large prawns may be expected to be more precise, with a lower target for r (1.6% glaze).

A2 Reproducibility

The absolute difference between two test results carried out under reproducibility conditions should not be greater than the reproducibility, R deduced from the collaborative trial data summarised in Tables 1 and 2. Overall, R may be taken as about 4% glaze at the appropriate levels; this corresponds to a relative standard deviation of reproducibility (coefficient of variance of reproducibility), RSD_R, of 4 - 6%.

A3 Trueness (Bias)

The observed accuracy of the method may be assessed by comparing the overall mean of the somewhat imprecise results with the expected values given in Tables 1 and 2. The cold-water prawns gave a recovery of 150% glaze or more, while the larger warm-water prawns gave more satisfactory results (110%).

A4 Limit of Detection

This limit has not been established, but the collaborative trial data suggest that levels of ice glaze lower than 4% cannot be detected with confidence. The limit of detection in large prawns may be lower (2% glaze).

A5 Statistical Data Derived from the Results of Interlaboratory Tests

Participants in the collaborative trial each analysed four samples of quick frozen cooked cold-water prawns once, and four samples of quick frozen cooked warm-water prawns once. These comprised small whole cold-water prawns analysed in blind duplicate (1/5; 7/18) and whole warm-water prawns analysed similarly (small, 12/16; large, 10/13).

Tables 1 and 2 summarise the statistical data; four outlying results were reported. The ice-glaze levels were expressed as a percentage by mass of the sample.

A6 Interpretation of Observed Levels

The subjective nature of the method is reflected in the poor overall levels of accuracy and precision established by the results of the collaborative trial; there is a distinct tendency towards massive overestimation, and the observed values of repeatability and reproducibility (Tables 1 and 2) are larger than would be considered acceptable in a conventional chemical method. Nevertheless the method is recommended for the analysis of quick frozen shrimps and prawns until a more precise method is established.

The shellfish content of the original sample, expressed as a percentage by weight, is given by subtracting the glaze content from one hundred. It is recommended that the results should normally be interpreted in terms of shellfish content, since this is the parameter of interest to the consumer.

TABLE 1
Statistical Analysis of the % Ice-glaze in Quick-frozen Cooked Cold-water Prawn Samples

Sample	1/5 Small	7/18 Small
Number of Laboratories	12	12
Number of results accepted	24	24
LEVEL OF ANALYTE		
Mean observed value $\bar{x}$	26.9	22.8
Actual (target) value	20.7	13.3
REPEATABILITY		
Standard Deviation S_r	1.2	0.79
Relative Standard Deviation RSD_r (%)	4.5	3.4
Repeatability r [2.8 x S_r]	3.4	2.2
REPRODUCIBILITY		
Standard Deviation S_R	1.2	1.0
Relative Standard Deviation RSD_R(%)	4.4	4.5
Reproducibility R [2.8 x S_R]	3.3	2.9

TABLE 2
Statistical Analysis of the % Ice-glaze in Quick-frozen Cooked Warm-water Prawn Samples

Sample	12/16 Small	10/13 Large
Number of Laboratories	12	12
Number of results accepted	22	22
LEVEL OF ANALYTE		
Mean observed value $\bar{x}$	25.2	18.7
Actual (target) value	22.9	16.9
REPEATABILITY		
Standard Deviation S_r	1.4	0.57
Relative Standard Deviation RSD_r(%)	5.4	3.1
Repeatability r [2.8 x S_r]	3.8	1.6
REPRODUCIBILITY		
Standard Deviation S_R	1.5	0.89
Relative Standard Deviation RSD_R(%)	6.0	4.8
Reproducibility R [2.8 x S_R]	4.2	2.5

A7 Key to Tables 1 and 2

Symbol	Definition
$\bar{x}$	Overall mean value
S_r	The standard deviation of repeatability
RSD_r	The relative standard deviation of repeatability, expressed as a percentage of the mean (coefficient of variance of repeatability CV_r)
r	Repeatability
S_R	The standard deviation of reproducibility
RSD_R	The relative standard deviation of reproducibility, expressed as a percentage of the mean (coefficient of variance of reproducibility CV_R)
R	Reproducibility