

MAFF VALIDATED METHODS FOR THE ANALYSIS OF FOODSTUFFS

No. V12

ICE-GLAZE ON QUICK FROZEN FISH FILLETS

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1. Scope and Field of Application

The method is designed to determine the net contents of quick frozen raw fish fillets covered by ice-glaze.

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 applying a gentle spray of cold tap-water and then drained, dried and weighed. The weight loss is assumed to be loss of ice-glaze.

4. Reagents

None

5. Apparatus

5.1 Analytical balance

5.2 Spray: a gentle spray of cold tap-water.

6. Procedure

6.1 Place the sample in a freezer of temperature $-18^{\circ}\text{C} \pm 2^{\circ}\text{C}$ and allow to equilibrate. For analysis, remove the sample from low temperature storage, open immediately, accurately weigh in g to one decimal place (m_0) and put it under a gentle spray of cold tap-water.

6.2 Agitate carefully so that the product is not broken. Spray until all the ice-glaze that can be seen or felt is removed.

6.3 Allow the sample to drain; remove adhering water by the use of a paper towel and weigh the deglazed product. 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:

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

where:

m_0 is the initial frozen weight taken (6.1);

m_1 is the observed deglazed weight (6.3).

9. References

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 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 Table 1. For both species, r may be taken to be 2.4% glaze at 4% levels, and 4% glaze at 8% levels. This precision corresponds to a relative standard deviation of repeatability (coefficient of variance of repeatability), RSD_r , of about 20%.

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 Table 1. For both species, R may be taken as 6% glaze at 8% levels; this corresponds to a relative standard deviation of reproducibility (coefficient of variance of reproducibility), RSD_R , of over 30%.

A3 Trueness (Bias)

The observed accuracy of the method may be assessed by comparing the overall mean of the results with the expected values given in Table 1. The cod fillets gave a recovery of 108% glaze, while the plaice fillets

(which had a larger surface area to weight ratio) gave 142%. The overestimates must be due to loss of physiological water, and are unsatisfactory for plaice fillets.

A4 Limit of Detection

This limit has not been established, but the poor accuracy demonstrated by the collaborative trial suggests that levels of ice glaze lower than 2.5% cannot be detected with confidence, and that even higher observed levels may not indicate glaze in samples with inappropriate physical properties.

A5 Statistical Data Derived from the Results of Interlaboratory Tests

Participants in the collaborative trial each analysed four samples of quick frozen whole uncooked fish fillets once. These comprised two species, each analysed in blind duplicate: "large" plaice fillets (2/11) and "small" cod fillets (9/15). The selected cod fillets were in fact thicker than the plaice fillets.

Table 1 summarises the statistical data; no outlying results were reported, though one sample was not analysed and its duplicate not accepted. The ice-glaze levels were expressed as a percentage by mass of the sample.

TABLE 1
Statistical Analysis of the % Ice-glaze in Quick-frozen Fish Samples

Sample	2/11	9/15
Number of laboratories	12	12
Number of results accepted	24	22
LEVEL OF ANALYTE		
Mean observed value	7.4	4.0
Actual (target) value	5.2	3.7
REPEATABILITY		
Standard Deviation S_r	1.32	0.86
Relative Standard Deviation RSD_r (%)	18	22
Repeatability r [$2.8 \times S_r$]	3.7	2.4
REPRODUCIBILITY		
Standard Deviation S_R	2.18	1.39
Relative Standard Deviation RSD_R (%)	28	35
Reproducibility r [$2.8 \times S_R$]	6.1	3.9

A6 Interpretation of observed levels

The subjective nature of the method is reflected in the poor levels of accuracy and precision established by the results of the collaborative trial; there is a distinct tendency towards overestimation, and the observed values of repeatability and reproducibility (Table 1) are larger than would

be considered acceptable in a conventional chemical method. Nevertheless the method is recommended for the analysis of quick frozen fish fillets until a more precise method is established.

The fish 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 fish content, since this is the parameter of interest to the consumer.

A7 Key to Table 1

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