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A nitrogen factor for Alaska pollack ingredient in fish products

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Analytical Methods Committee†

The Analytical Methods Committee has received and approved the following report from the Nitrogen Factors Sub-Committee.

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Summary

Alaska pollack (*Theragra chalcogramma* (*Pallas*)) has one of the largest catches on a global level (2.83 million tonnes in 2010), and is being increasingly used by European manufacturers as the raw material for fish products, replacing scarcer and more expensive white fish species. It is caught and prepared by Pacific Rim countries, especially Russia and the USA. Mince blocks are prepared from recovered flesh from the frames after filleting, and are used principally to prepare value-added coated fish products. Soluble nitrogen is lost during the preparation of these mince blocks, and their nitrogen content is lower than that of fillet blocks. Hence a nitrogen factor is very important in determining if good manufacturing practice has been used, and that the declaration of fish content in fish products is correct.

The experimental plan involved the preparation of 75 samples each of whole Alaska pollack, Alaska pollack fillet blocks, and Alaska pollack mince blocks achieved in two sessions, one in March and the other in September 2012. Whole Alaska pollack were obtained from the main fishing grounds in the Russian and US sectors of the Bering Sea, and filleted without using any water. Frozen Alaska pollack fillet and mince blocks were obtained from fish caught in both sectors of the Bering Sea during the two main seasons (March (A) and September (B)), and processed in factories or ships known to use good manufacturing practices.

All the samples were analysed for nitrogen content, moisture, ash and fat according to established British Standards Institute methods (or their equivalents) and the results were analysed statistically. All three laboratories participating in the study used the rapid Dumas combustion method for the determination of nitrogen content. This method gives slightly higher results (a factor of 1.014) than the Kjeldahl digestion method. Because of problems in sourcing material, it was not possible to obtain an equal distribution of samples of Alaska

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pollack that covered all the variables of season and origin. However, sufficient samples were obtained to look at trends and show that where significant differences were found, these were not of practical significance in the overall mean and uncertainty.

The recommended nitrogen factor for Alaska pollack fillet block is 2.7 (Kjeldahl 2.65), and Alaska pollack mince block 2.5 (Kjeldahl 2.45). The difference between the average nitrogen content of whole Alaska pollack and the commercial fillet or fillet block was only 5%, indicating that good manufacturing practice had been used in their preparation, and water uptake had been kept to a minimum. For Alaska pollack, the difference between whole fish and the mince block was 11%, which fits with the known loss of soluble protein during mince recovery.

Report

The membership of the Nitrogen Factors Sub-Committee responsible for the preparation of this report was: Dr Mark Woolfe, (Chairman), Dr Roger Wood (Secretary), Mr Andrew Caines, Ms Bhavna Parmar, Mrs Theresa Ekong (until 11 Oct 2013), Mr Charles Boardman, Mr David Keeble, Mr Duncan Campbell, Mr Jeremy Hall, Prof. Michael Thompson, Mr Robbie Beattie, Mrs Selvarani Elahi, Mr Steve Lamming, Mr Steve Moore, Ms Henrietta Tambo, Mr Andrew Furmage, and Ms Liz Moran. Participants in the study were: Dr Mark Woolfe, Mr Steve Lamming, Mr Robert Olivant, Mr Keith Flint, Mr Charles Boardman, Mr Chris Hunt, Dr Joanna Topping, and Prof. Michael Thompson, who was responsible for the statistical evaluation.

Introduction

Labelling rules¹ require that for meat products, the species of meat used should be declared. For fish products there is still the option to declare the species of fish or use the generic description "fish". Where ingredients are highlighted in the name of the food, the amount of that ingredient must be declared as a percentage of the final product

(QUID - quantitative ingredient declaration). Although the amount of an ingredient is calculated on a recipe basis, enforcement authorities usually check the declaration by analysis of the finished product. The analysis determines the nitrogen content (mainly on a fat free basis) of a meat or fish ingredient, and converts this to a meat or fish content using a previously determined nitrogen factor. Added water of a fish ingredient can be calculated by difference. Manufacturers of fish products usually check the specification of their raw materials, and nitrogen factors are used to determine if the fish raw material contains excess added water. The use of nitrogen factors to calculate the fish content of coated fish products has also been accepted at an international level, as detailed in Codex Alimentarius.2 A considerable amount of research has already been undertaken to determine robust nitrogen factors for common meat and poultry species, and some fish species. In this project the nitrogen factor for a new important species of fish for the UK fish product market was determined.

Alaska pollack (Theragra chalcogramma (Pallas)) production

Alaska pollack is sold in the UK under the commercial names Pacific pollack, Pacific pollock, and Atlantic pollock.3 On a global level, Alaska pollack is the second highest caught species after anchoveta. Russia, USA, Korea and Japan are the major fishing nations catching and preparing this (headed and gutted) fish. Russia and the US also have factory vessels producing single frozen fillet and mince blocks. Frozen de-headed and gutted fish are sent to China for filleting as well as double frozen block production. Given the decline and cost of gadoid species (cod, haddock, whiting, and hake) production, Alaska pollack serves as a major raw material for coated fish products. It is imported mainly in the form of frozen blocks of both filleted fish and mince. Table 1 shows that the global catch of Alaska pollack in 2010 was three times that of Atlantic cod. In 2007, the catch of Alaska pollack reached a peak of nearly 3 million tonnes, but has failed to exceed that since, probably due to quota management. Alaska pollack has increasingly become the raw material of choice for white fish products because of its lower price.

Alaska pollack spawn from mid March to June in the Bering Sea, and commercial fisheries try to avoid catching fish immediately post-spawning because of their low quality. There are two main catching seasons - January to the beginning of April (A season), and June to October (B season). The fish are caught both in the Alaska (US) sector of the Bering Sea (FAO67) and the Russian sector (FAO61). Some fish are filleted and frozen into fillet blocks on factory/fishing vessels, whilst other fish are processed in land factories in Alaska or Russia. Nearly 60% of the imported frozen fillets (usually in the form of 10 kg fish blocks) are prepared in China where it is more difficult to inspect and control manufacturing practices. Mince blocks are prepared from recovered flesh from the frames after filleting, and are used principally to prepare value-added coated fish products.

Table 1 Global fisheries catch of cod and Alaska pollack in 2006–2010, in millions of tonnes^a

Fish	2006	2007	2008	2009	2010
Alaska pollack	2.86	2.91	2.65	2.50	2.83
Atlantic cod	0.83	0.78	0.77	0.86	0.95

 $[^]a$ FAO yearbook fisheries and a quaculture statistics 2010 – FAO Rome 2012.

Experimental

Alaska pollack samples

1. Whole Alaska Pollack. Seventy five samples of whole Alaska pollack, sourced from both the Russian sector (FAO61) and the US sector (FAO67) of the Bering Sea, were prepared in September 2012. The frozen fish were carefully thawed, and whilst still frozen, filleted and the fillets trimmed without using any water. The two fillets from one fish were homogenised to give a 250 g sample.

2. Alaska pollack fillet and mince blocks. Seventy five samples each of the fillet and mince blocks were prepared in March and September 2012. The samples were prepared from separate cartons containing a 7.5 kg block of Alaska pollack fillets and a 7.5 kg block of mince. These were obtained from both US and Russian processor ships and a factory in the A and B seasons. The factory had been approved and inspected for good manufacturing practices. On factory ships, approval is by selfassessment, but checks are made on all consignments to ensure they are within specification. It was not possible to obtain equal numbers of A and B season fish, and those from the A season predominated. Only single frozen fillet blocks were obtained and no double frozen blocks were used in this study. A section of frozen block was cut by a band saw to give a 240 g piece, which then was thawed, homogenised, and put into sample pots. All samples were randomised and sent to three labs for analysis.

Methods of analysis

Samples of whole Alaska pollack, fillet and mince blocks were analysed in duplicate using the following chemical analysis methods:

British Standards Institution, analytical methods for meat and meat products.

Part 1 1970 (1993) determination of ash.4

Part 2 1980 (1993) determination of nitrogen or equivalent method.⁵ All labs used an equivalent method of determining nitrogen by Dumas combustion using a LECO 2000 CNS.

Table 2 Summary of chemical analyses of whole Alaska pollack

Variable	No.	Mean	SD	SEM
Nitrogen %	75	2.81	0.20	0.023
Fat %	75	0.96	0.32	0.0363
Moisture %	75	81.6	0.90	0.10
Ash %	75	1.22	0.05	0.0062

Table 3 Two way ANOVA analysis of results from 3 labs and season of whole Alaska pollack

	N %	Fat %	Moisture %	Ash %
Season				*
Α	2.82	0.94	81.5	1.20
В	2.83	0.87	81.2	1.26
Lab	*	*	*	*
Α	3.00	0.74	80.6	1.21
В	2.73	0.70	81.5	1.23
C	2.75	1.26	82.0	1.26

^{* =} Differences significant at 95% confidence.

Table 4 Summary of chemical analyses of Alaska pollack fillet block

Variable	No.	Mean	SD	SEM
Nitrogen %	75	2.68	0.181	0.0209
Fat %	75	0.71	0.314	0.0362
Moisture %	75	82.1	0.682	0.079
Ash %	75	1.31	0.086	0.0099

Table 5 Three way ANOVA analysis of results from 3 labs, origin and season

	N %	Fat %	Moisture %	Ash %
Season		*		*
A	2.69	0.76	82.1	1.30
В	2.65	0.62	82.1	1.34
Lab		*		*
A	2.71	0.46	81.9	1.27
В	2.62	0.58	82.1	1.34
C	2.67	1.03	82.3	1.35
Catch area		*		*
FAO61	2.64	0.75	82.2	1.30
FAO67	2.70	0.63	82.0	1.34
* = Difference	(s) significar	nt at 95% con	fidence.	

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Table 6 Summary of chemical analyses for Alaska pollack mince block

Variable	No.	Mean	SD	SEM
Nitrogen %	75	2.51	0.179	0.021
Fat %	75	0.70	0.224	0.026
Moisture %	75	83.1	0.748	0.086
Ash %	75	1.47	0.100	0.012

Part 3 1970 (1997) determination of moisture.6

Part 4 1970 (1993) determination of total fat.⁷

The detailed requirements for analysis included:

Two replicates of the four analyses are required per sample, which should add up to 100% \pm 2%. The duplicates should be

Table 7 Two way ANOVA of means of catch area FAO67 mince block with labs and season

	N %	Fat %	Moisture %	Ash %
Season	*			
A	2.48	0.68	83.2	1.49
В	2.39	0.60	83.5	1.53
Lab		*		*
A	2.44	0.50	83.1	1.46
В	2.42	0.56	83.3	1.50
\mathbf{C}	2.46	0.85	83.6	1.57

^{* =} Difference(s) significant at 95% confidence.

Table 8 Two way ANOVA of means of season A with catch area and laboratory

	N %	Fat %	Moisture %	Ash %
Catch area	*			
FAO61	2.61	0.79	82.6	1.39
FAO67	2.49	0.68	83.2	1.49
Lab		*		*
A	2.61	0.63	82.63	1.41
В	2.50	0.62	82.82	1.42
\mathbf{C}	2.54	0.97	83.25	1.49

^{* =} Difference(s) significant at 95% confidence.

randomised within each batch. Analytical agreement is required between the duplicates – the repeatability limits are 0.5 g/100 g (moisture and fat) and 0.1 g/100 g (nitrogen and ash).

The laboratories analysed a standard reference material in duplicate with each batch of fish samples. Laboratories assessed their performance against the above limits, and decided whether repeats were necessary. All three laboratories used the rapid Dumas method for nitrogen determination. This measures the non-protein nitrogen as well as the protein nitrogen, and hence gives higher results than the Kjeldahl method.⁸ The difference is small, and Dumas is higher by a factor of 1.014, which is more important for fish than for meat. Therefore it is usual to quote the nitrogen result for both methods of analysis as Kjeldahl is still widely used in laboratories worldwide.

Results

(a) Whole Alaska pollack

The chemical analyses of the 75 whole Alaska pollack samples gave an average nitrogen content of 2.81 g/100 g, and a fat content of less than 1% (Table 2). Comparison of the results from the 3 labs showed a significant difference in all four chemical analyses, but no difference between seasons except for ash (Table 3).

Table 9 Means of chemical analyses and standard error for Alaska pollack products

	N %		Fat %		Moisture	%	Ash %	
Product	Mean	SEM	Mean	SEM	Mean	SEM	Mean	SEM
Whole Alaska pollack	2.81	0.023	0.96	0.04	81.6	0.10	1.22	0.006
Alaska pollack fillet block Alaska pollack mince block	2.68 2.51	0.021 0.021	0.71 0.70	0.04 0.03	82.1 83.1	0.08 0.09	1.31 1.47	0.010 0.012

Table 10 Recommended nitrogen factors for Pangasius and Alaska pollack

Product	Nitrogen factor (Kjeldahl)
Whole Alaska pollack	$2.80~(2.75)\pm0.05$
Alaska pollack fillet block	$2.70~(2.65)\pm0.04$
Alaska pollack mince block	$2.50~(2.45)\pm0.04$

(b) Alaska pollack fillet block

The chemical analyses of the 75 samples of commercial fillet blocks gave an average nitrogen content of 2.68 g/100 g with a fat content of 0.7 g/100 g (Table 4). There were no significant differences in nitrogen content with season or catch area/ process plant (Table 5).

(c) Alaska pollack mince block

The overall average nitrogen content of mince blocks was 2.51 g/100 g with a fat content of 0.7 g/100 g (Table 6). As all the mince blocks from the Russian sector (FAO61) were A season, statistical analyses were carried out on two subsets of the total data, one using the means of analyses for all samples from the catch area FAO67 (Table 7), and the means of all season A samples (Table 8). For mince blocks caught and produced in the US sector of the Bering Sea, there was a significant difference in nitrogen content between seasons but not between laboratories. Examining mince blocks from fish caught and processed in the A season, there was a significant difference between catch areas/processors in the US and Russian sector.

Discussion and conclusions

It was not possible to obtain an equal distribution of samples covering all the variables of season and origin for Alaska pollack. However, sufficient samples were obtained to look at trends in the overall mean and uncertainty and show that where significant differences were found, these were not of

practical significance. A summary of the means of the nitrogen, moisture, ash and fat contents is shown in Table 9. The recommended nitrogen factors for whole Alaska pollack, Alaska pollack fillet block and mince block for both the Dumas and Kjeldahl methods are given in Table 10. These values have been rounded up or down to the nearest 0.05. The values take into account the low fat contents (less than 1%) of the fish, with the standard uncertainty (2 \times SEM) for means of duplicate analyses. Whole Alaska pollack is not normally sold on the UK market, but the nitrogen factor for whole fish can be used as a comparison with the processed products fillet and mince blocks. The nitrogen content of the fish ingredient produced by good manufacturing practice for Alaska pollack fillet block is 5% less than for the whole fish. This figure compares favourably with the interim factor for Alaska pollack (2.59) in the UK Code of Practice,9 which indicates a 6% difference in nitrogen factor between whole fish and commercial fillets assuming a Kjeldahl analysis. For mince block fish ingredient, the recommended factor is 11% less than for the whole fish, which is acceptable given the loss of nitrogen during preparation.

The nitrogen factor values obtained in this study also compare favourably with historic data provided by Young's Seafood (Table 11).10 Double frozen Alaska pollack fillet blocks were not analysed in this study, but it can be seen from historic data that the nitrogen factor value of 2.70 for the Alaska pollack fillet block is applicable to both single and double frozen fillet blocks.

Acknowledgements

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Table 11 Average nitrogen values of analytical data provided by Young's Seafood

Product	Sample period	No. of samples	Mean N %	SD
Alaska pollack fillet blocks – single frozen	1997–2013	1006	2.69	0.167
Alaska pollack fillet blocks – double frozen	2003-2013	138	2.71	0.15
Alaska pollack mince blocks - single frozen	1997-2013	748	2.54	0.176

References

- 1 The Food Labelling Regulations 1996 (SI 1996 No. 1499), as amended, ISBN: 0-11-035941-0.
- 2 Codex Standard for Quick Frozen Fish Sticks (Fish Fingers), Fish Portions and Fish Fillets – Breaded or in Batter, CODEX STAN 166 – 1989, Rev 2011.
- 3 The Fish Labelling (England) Regulations 2010. SI 210 No. 420, HMSO.
- 4 BS4401-1: 1998, ISO 936:1998, Methods of Test for Meat and Meat Products, Part 1: Determination of Total Ash.
- 5 British Standard, BS 4401:1980 ISO 937:1978, "Methods of Test for Meat and meat products Part 2: Determination of nitrogen content (reference method)".
- 6 BS4401-3: 1997, ISO 1442:1997, Methods of Test for Meat and Meat Products, Part 3: Determination of Moisture Content (Reference Method).

- 7 BS 4401: Part 4: Method A 1970 (Weibull Stoldt, acid hydrolysis) UKAS accredited method based on BS4401-4: 1970, Methods of Test for Meat and Meat Products, Part 4: Determination of Total Fat Content: Method A (Weibull Stoldt).
- 8 M. Thompson, L. Owen, K. Wilkinson, R. Wood and A. Damant, A Comparison of the Kjeldahl and Dumas Methods for the Determination of Protein in Foods, using Data from a Proficiency Testing Scheme, *Analyst*, 2002, **127**, 1666–1668.
- 9 Code of Practice on the Declaration of Fish Content of Fish Products, UKAFFP, BFFF, BHA, BRC, Sea Fish Industry Authority, LACOTS, Association of Public Analysts, http://www.seafish.org/media/Publications/Fish_Content_CoP.pdf.
- 10 Personal communication with Young's Seafood, May 2013.