Das dreimalige Schütteln im Trockenschrank nach A reicht dagegen wohl nicht aus, um das Fett vollständig herauszulösen. Wird dagegen das Gemisch während des Aufschlusses im Rundkolben auf einem heizbaren Magnetrührer kräftig gerührt, werden praktisch die gleichen Gehalte wie bei B gefunden.
Die erhaltenen Werte lassen sich natürlich nur begrenzt mit denen vergleichen, die mit Methoden auf anderer Grundlage gefunden wurden.

Dank
Frau *H. Bendig* danke ich für die zuverlässige und engagierte Mitarbeit.

Literatur

1) *Weibull, M.*: Über Fettbestimmungen im Brot. Z. Angew. Chem. **1892**, 450–451.
2) *Weibull, M.*: Weitere Versuche über Fettbestimmungen im Brot. Z. Angew. Chem. **1894**, 199–202.
3) *Großfeld, J.*: Die Bestimmung des Fettgehaltes in Nahrungsmitteln und Seife. Z. Unters. Nahr. Genussm. **44**, 193–203 (1922).
4) *Großfeld, J.*: Erfahrungen bei der Untersuchung von fetthaltigen Backwaren. Z. Unters. Lebensm. **74**, 284–291 (1937).
5) *A. Beythien* und *W. Diemair*: Laboratoriumsbuch für den Lebensmittelchemiker. **8.** Aufl., Verlag Steinkopff, Dresden u.a. 1963, S. 38, 202, 749.
6) *Stoldt, W.*: Fettbestimmung in Lebensmitteln. Dtsch. Lebensm. Rdsch. **45**, 41–46 (1949).
7) *Stoldt, W.*: Fettbestimmung bei Lebensmitteln. Dtsch. Lebensm. Rdsch. **48**, 39–40 (1952).
8) Amtliche Sammlung von Untersuchungsverfahren nach § 35 LMBG, Herausg.: Bundesinstitut für gesundheitlichen Verbraucherschutz und Veterinärmedizin (BgVV). Methode L 01.00-20 (5/1988): Bestimmung des Fettgehaltes von Milch und Milchprodukten – Verfahren nach Weibull (= DIN 10342). Beuth Verlag, Berlin, Wien, Zürich.
9) ibid. Methode L 06.00-6 (9/1980): Bestimmung des Gesamtfettgehaltes in Fleisch und Fleischerzeugnissen.
10) ibid. Methode L 17.00-4 (5/1982): Bestimmung des Gesamtfettgehaltes in Brot einschließlich Kleingebäck aus Brotteigen.
11) ibid. Methode L 20.01/02-5 (5/1980): Bestimmung des Gesamtfettgehaltes in Mayonnaise und emulgierten Soßen.
12) ibid. Methode L 44.00-4 (12/1985): Bestimmung des Gesamtfettgehaltes in Schokolade.
13) ibid. Methode L 05.00-14 (6/1991): Bestimmung des Gesamtlipidgehaltes in Eiern und Eiprodukten.
14) Schweizerisches Lebensmittelbuch, Band 2, Kapitel 11-5.7 (7/1995): Fleisch und Fleischwaren – Bestimmung des Gesamtfettes – Säureaufschlussmethode. Eidg. Drucksachen- und Materialzentrale, Bern.
15) ibid. Kapitel 36A-6 (5/1992): Kakao, Kakaomasse, Kakaopulver und Schokoladepulver – Bestimmung des Gesamtfettes – Säureaufschlussmethode.
16) Deutsche Forschungsgemeinschaft: MAK- und BAT-Werte-Liste 2000. Wiley-VCH, Weinheim 2000.
17) Verordnung über gefährliche Stoffe (Gefahrstoffverordnung – GefStoffV) vom 26. 8. 1986, BGBl. I S. 1470 sowie Anhang VI, S. 234.
18) Anhang I zur EG-Richtline 67/548/EWG des Rates vom 27. 06. 1967 zur Angleichung der Rechts- und Verwaltungsvorschriften für die Einstufung, Verpackung und Kennzeichnung gefährlicher Stoffe. ABl. No. 196 vom 16. 08. 1967, S. 1.

Determination, Spatial Variation and Distribution of Iodine in Fish

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Summary
Iodine is a very important essential trace element and marine fish is one of the rare natural food containing relatively large amounts of iodine.
In this study a fast and reliable determination method of iodine in marine species has been developed and validated. The method based on a procedure of *Gu* et al.[18] published in 1997 and avoids ashing of the sample which often leads to iodine losses.
Furthermore, various biological factors, effecting the iodine content in fish have been studied and the distribution in fish muscle was analysed. The iodine content of various flat, lean and fatty fish species from different fishing grounds has been compared, yielding no clear indication of a relation of iodine concentration to the fishing ground. But large variations were found between cod samples in fishes of a single catch.
Distribution within fish muscle showed an iodine gradient, decreasing from skin to the inner part of the fillet and iodine levels in the skin of marine fishes can be up to twentifold of the content of the muscle tissue, depending on the species.
No differences were observed between left and right fillets, ventral and dorsal parts and head and tail parts of cod fillets, respectively.

Zusammenfassung
Iod gehört zu den essenziellen Elementen, die mit der Nahrung aufgenommen werden. Zu den wenigen Lebensmitteln, die relativ hohe Iodgehalte aufweisen, zählen Meeresfische und andere Nahrungsmittel aus dem Meer. Es wurde eine schnelle und zuverlässige Methode zur Bestimmung von Iod in Meeresorganismen entwickelt und validiert. Die Methode basiert auf einem von *Gu* et al.[18] 1997 publizierten gaschromatografischen Nachweisverfahren.
Mit der Methode wurden die Abhängigkeit des Iodgehaltes von verschiedenen biologischen Faktoren, die den Iodgehalt von Fischen beeinflussen können, und die Iodverteilung im Fischmuskel untersucht.
Ein Zusammenhang zwischen dem Iodgehalt und dem Fangplatz konnte nicht festgestellt werden. Bereits Fische aus einem Fang zeigten starke Schwankungen in den Gehalten.

Die Haut von Seefischen kann bis zu 20-fach höhere Iodkonzentrationen als der Fischmuskel aufweisen. Die Iodverteilung im Filet nimmt von der Hautseite zum inneren Filet deutlich ab, während keine Unterschiede zwischen linken und rechten Filets, dem Vorder- und Schwanzteil sowie dem dorsalen und ventralen Anteil festgestellt wurden.

Introduction

Iodine is a very important essential trace element. The recommended dietary allowance for iodine has been set at 150–200 µg daily for adults[1]. Iodine is an integral part of the thyroid hormones that play an important role in controlling the rate of basic metabolism and reproduction[2,3]. Chronically low intake leads to iodine deficiency disorders, which remains a major problem in many regions of the world. According to WHO[4] more than 1500 Mio humans suffer from iodine deficiency disorders. Fish and other marine products are one of the rare natural food items containing relatively large amount of iodine[5,6]. However, comparing different publications, large variations in iodine content have been reported for the same fish species[6-14]. Data on iodine in cod muscle e. g. vary between low 21 µg iodine/100 g wet weight (w. w.) and high 652 µg iodine/100 g w. w.[5]. Meanwhile these variations could be confirmed by own investigations.

The composition of fish and thus the iodine content may be related to different factors like feeding habits, fishing area, maturation state and age[15]. Additionally distribution within the fish can vary considerably. Recently *Eckhoff* and *Maage*[16] reported large differences in iodine content of skin and muscle of fishes from the African lake Victoria.

Also analytical uncertainties have to be taken into account when discussing possible reasons for variation. Most of results which have been reported are based on a complete thermal decomposition of the matrix before actual iodine determination. It is well known that mineralisation is the most critical operation in iodine determination of biological materials[17] and can lead to iodine losses. Thus any analytical problems have to be excluded before studying the different biological effects.

Recently *Gu* et al.[18] published a method for gaschromatographic determination of iodine in milk and oyster tissue based on decomposition of the organic matrix by peroxydisulfate oxidation in alkaline solution followed by derivatisation of iodine to 2-iodo-pentane-3-one, according to a method of *Mitsuhashi* and *Kaneda*[19]. The derivative can be analysed using gas chromatography and ECD detection. *Gu* et al.[18] reported reproducible results for non fat milk powder and lean oyster tissue, but no performance data are available for fat containing matrices which one often encounters when analysing fish and fishery products.

In this study a modified procedure of *Gu* et al.[18] is introduced which has been adopted to fatty fish matrix and optimised for the determination of iodine in fish and fishery products.

The method was applied to study some biological parameters which may effect the iodine content of fish and to determine the distribution of iodine within the fish.

Method and Material

Evaluation of a modified GC MS/ECD method for determination of iodine in sea food

Tests with the original method of *Gu* et al.[18] on fish samples spiked with iodine showed, that the procedure failed to analyse fatty fish species. Thus some modifications were necessary to overcome this problem. The introduction of following modifications allows a reliable analysis of iodine in all kinds of fish species:

- Increase of the amount of peroxydisulfate and potassium hydroxide to yield a complete oxidation of fatty fishes and
- Introduction of a surrogate standard

Chemicals

Potassium hydroxide p. a.
Potassium peroxydisulfate p. a. (*Fluka*)
Sodium sulfite p. a.
Sulfuric acid 5 M
Hydrogen peroxide solution, 30 %
Pentan-3-one
Pentachlorbenzene
Sodium sulfate p. a.
Sodium iodide p. a.
Iso-octane pesticide residue analysis grade

Solution:
Pentan-3-one (4 %, v/v):
4 ml pentane-3-one were diluted in 96 ml H_2O (desalted)

Stock solutions were diluted to following concentrations:
Pentachlorobenzene solution: 100 mg pentachlorobenzene/100 ml iso-octane were diluted to 0.032 µg pentachlorobenzene/ml iso-octane
Iodine standard solution A: 118.117 mg NaI/100 ml H_2O = 1 mg I^-/ml were diluted to 0.1 µg I^-/ml H_2O
Iodine standard solution B: 118.117 mg NaI/100 ml H_2O = 1 mg I^-/ml were diluted to 10 µg I^-/ml H_2O

Devices

Normal laboratory equipment including brown glassware, magnetic stirrer plate, automatic shaker for separatory funnels, a capillary gas chromatograph *Carlo Erba* HRGC 5160 Mega equipped with a FS-SE-54 capillary column (50 m, 0.2 mm ID, 0.25 µm film thickness), ECD-detector, auto sampler (AS 800, *Fisons Instruments*), and a computer based data acquisition. For peak confirmation a *Finnegan* Mat IST 40 ion trap mass spectrometer equipped with a DB 5 capillary column (60 m x 0.25 mm ID, 0.25 µm film thickness) was used in the electron ionisation mode (EI/MS).

Analytical procedure

All fish samples were homogenised before analysis. 0.5 g fat fish sample or 1.4 g lean fish sample (< 1 % fat content) and 140 ml desalted water were transferred into a 300 ml Erlenmeyer flask. Alternatively corresponding amounts of freeze dried fish samples can be weighed into the flask. 17 g potassium hydroxide is added under continuous stirring followed by 20 g of potassium peroxydisulfate. The mixture is heated to gentle boil under reflux for about 60 min. The solution must become completely clear, otherwise the charge must be discarded and the sample weight has to be reduced.

The clear hot solution is transferred into a 250 ml calibrated flask, containing 5 g sodium sulfite. The flask is filled with water up to mark.

25 ml aliquot of sample solution is transferred into a 100 ml brown glass separatory funnel, 3 ml of 5 M sulfuric acid, 3 ml 30 % H_2O_2 and 1 ml pentan-3-one solution is added and the mixture is shaken for 5 min. 10 ml iso-octane containing pentachlorobenzene (surrogate standard) (0.032 µg/ml) is added and the 2-iodo-pentane-3-one is extracted shaking for another minute.

The organic layer is washed 4 times with 10 ml desalted water, respectively, transferred into a 50 ml flask and dried over sodium sulfate. 2 µl aliquot is injected into the gas chromatograph.

Calibration

For calibration aliquots of the stock solution are diluted to obtain iodine concentrations between 0.006–0.06 µg I/ml. 25 ml of each dilution is processed as indicated for the sample aliquot.

For daily routine quantification a 1 point calibration standard is used, consisting of 7.5 ml iodine standard solution A, 0.25 g sodium sulfite and 17.5 ml desalted water, being worked up as a sample aliquot. The routine calibration standard equals an iodine concentration of 0.03 µg I/ml.

Gas chromatographic conditions

- Injector: 2 µl split less, 200°C
- Detector: 63 Ni-ECD, 300°C, make up gas: nitrogen
- Oven temperature profile: 50°C (3 min) → 20°C/min to 140°C → 10 °C/min to 220°C → 30°C/min to 280°C (8 min)
- Carrier gas: nitrogen (3 ml/min)

Quantification and analytical quality control

For routine analysis a 1 point calibration was used. The standard was measured at least once a day and samples were quantified by comparison of peak heights taking the surrogate standard into account. A calculation based on the daily standard was found to be advantageous, as variations which can occur in the daily performance of the ECD response can be corrected immediately.

The accuracy of the daily iodine standard is checked by a quality chart, basing on the relation of peak heights of 2-iodo-pentane-3-one and pentachlorobenzene.

Samples

Fish samples were collected during several cruises of the research vessel „Walther Herwig III". Fishes were characterised, hand filleted and skinned. All samples were deep frozen and stored at – 26°C until analysis. Skins were freeze dried and powdered before analysis.

Results and Discussion

Validation

Linearity

In the range of 0.006–0.03 µg I/ml the linearity of calibration curve was good with R^2 (coefficient of determination) of 0.998. Higher concentrations should be diluted before measurement.

Recovery

For determination of recovery rates a cod reference material CRM 422[20] with a certified iodine content of 4.95 µg I/g dry weight and different standard solutions, corresponding to iodine concentrations of 375 and 3750 µg I/100 g fish muscle, respectively, were analysed repeatedly (Table 1). The recovery rates varied between 93 and 109 %.

Tab. 1 Recovery rates [%] of various samples

Sample	Replicates	Recovery [%]	RSD [1] [%]
CRM 422	4	109.4	3.1
Standard solutions:			
3750 µg I/100 g	3	96.6	7.1
375 µg I/100 g	4	93	5

[1] RSD: relative standard deviation

The recovery rate of the reference material was within the given uncertainty of 10 %[20].

Routine application of the method included a frequent determination of the reference material CRM 422. Depending on the batch of chemicals used in the decomposition step of the organic matrix, occasionally an increase of the recovery rate up to 130 % and more was observed. A careful check revealed that the derivatisation of the liberated iodine was obviously influenced by unknown matrix compounds, resulting in larger peaks. Further investigations could exclude the fish matrix as possible source, the matrix effect tends to be created by trace impurities of the chemicals applied.

The problem was solved by introducing a matrix matched calibration standard instead of the common external standard. The matrix matched standard consists of 0.75 ml of iodine standard solution B, which is worked up like a sample. Due to similar conditions of standard and sample the matrix effect was neutralised and recovery rates of 100 % were achieved.

As long as the recovery rates of the reference material keep within the acceptable uncertainty range of 10 %, quantification based on the common external calibration standard can be used for routine analysis, as it is easier to handle.

Repeatability

Repeatabilities of the procedure were determined from various lean and fat fish species as well as in cod reference material spiked with vegetable oil. Results are compiled in Table 2 and showed variations (RSD = relative standard deviation) between 5.6 % and 12.6 %.

Tab. 2 Repeatability of the iodine determination in various fish species

Sample	Replicates	Average iodine content [µg I/ 100 g w. w.][1)]	SD [µg I/ 100 g w. w.]	RSD [%]
Cod	5	122	7.04	5.8
Greenland halibut	3	109	13.8	12.6
Mackerel	3	73	8.9	12.2
CRM 422 + vegetable oil (0.2 g + 0.1 g)	4	115	7.6	6.6

[1)] w.w.: wet weight

Limit of detection

A small signal was observed in the blanks at the same retention time of the iodine peak. This was already found by *Gu* et al.[18]. A careful check of each chemical used in the procedure showed that the peak resulted from a minor contamination of the potassium peroxydisulfate, which could not be avoided by using potassium peroxydisulfate from different suppliers. The peak height of the blank corresponds to an average iodine amount of 0.000215 µg I/ml digestion solution. The peak height can slightly vary within different batches of peroxydisulfate from one supplier, thus the blank should be determined with every new batch.

The limit of detection was determined by the height of the blank + 3 times of the standard deviation of the blank.

Based on an average iodine amount of 0.000215 µg I/ml in the blank, the limit of detection was calculated to 14.35 µg I/100 g fat fish sample (sample weight: 0.5 g) and 5.1 µg I/100 g lean fish (sample weight: 1.4 g)

Precision of the method

The precision of the method was tested by comparative analysis of various samples applying neutron activation analysis (NAA) as non destructive independent analytical method. The neutron activation analysis was performed at the Institut für Radiochemie der Technischen Universität München.

Freeze dried samples of whiting, herring and mackerel muscle meat, as well as the cod reference material were analysed with both methods.

Results are summerized in Table 3. Good agreement was found for all samples.

All results of the validation show that the modified peroxydisulfate oxidation method is suitable to analyse the iodine content in fish and yields reproducible and quantitative results.

The described analytical procedure was applied to study the effect of various factors which can influence the iodine content in fish.

Tab. 3 Comparative analysis of iodine in freeze dried fish samples applying peroxydisulfate method and neutron activation analysis (NAA)

Sample	Fat content [%]	Peroxide method [µg I/100 g w. w.]	NAA [µg I/100 g w. w.]
Whiting	0.2	589	631
Herring	8.7	37	41
Mackerel	15.0	44	49
CRM 422	0.3	115	95

Variation of iodine in fish species

To attempt a systemic appraisal of all biological effects which can influence the chemical constituents of fish is very difficult, since the effects embrace not only all of the environmental influences but also seasonal variations due to maturity, spawning, feeding and age accumulation. We focused on few selected parameters, knowing that others have to be realised in future to receive a better understanding of the mechanism of variation of iodine in fish.

Iodine content in fish in relation to the fishing ground

The fishing ground can influence the composition of fishes, as the diet may change according to the locality. Also salinity, oxygen concentration, temperature and mineral composition of seawater can vary between different areas and may play a part in iodine uptake.

During several cruises of our research ship „Walther Herwig III" fish samples were taken from different fishing grounds and the edible part (regularily the skinless fillets) were analysed for iodine content. To reduce variation between single fishes, each sample consisted of at least three individuals. The results are compiled in Table 4.

Most data were collected from cod and large variations in iodine content were observed between samples from different fishing grounds. The iodine content of cod fillets of two closely related fishing grounds of the Norwegian coast of the North Sea, Viking Bank and Eigersund Bank, varied between 29 µg I/100 g w. w. and 482 µg I/100 g w. w., respectively. On the other side no differences were observed between three other areas of the North Sea and between samples from the German Bight and the Baltic Sea. All cod samples from Buchan Deep, Utsira Ground and Wie Bankie contained between 179 and 231 µg I/100 g w. w., whereas samples from the German Bight and the Kieler Bucht contained high iodine levels of around 820 µg I/100 g w. w..

Tab. 4 Average iodine content in fish muscle from various fishing grounds (n = 3–5 fishes/ pooled sample)

Fish species	Fishing ground	Date of catch	Water content [%]	Iodine content [µg I/100 g w. w.]
Cod *(Gadus morhua)*	North Sea (German Bight)	Jan. 1998	79.7	831
	North Sea (Viking Bank)	Nov. 1998	79.8	29
	North Sea (Eigersund Bank)	Feb. 1997	80.6	482
	North Sea (Buchan Deep)	April 2000		179
	North Sea (Utsira Ground)	April 2000		184
	North Sea (Wie Bankie)	April 2000		231
	Baltic Sea Kieler Bucht	Sept. 1999		813
	Baltic Sea (Adler Grund)	Sept. 1999		1063
Haddock *(Melanogrammus aeglefinus)*	North Sea (Turbot Bank)	Jan. 1995		24
	North Sea (Copinsay)	May 1997		104
	Faroe Islands	April 1999	82.3	267
	Faroe Islands	April 1999	80.5	136
Plaice *(Pleuronectes platessa)*	North Sea (German Bight)	Feb. 1998	80.8	19
	Faroe Islands	April 1999	82	32
Dab *(Limanda limanda)*	North Sea (German Bight)	Jan. 1998	79	45
	North Sea (Clay Deeps)	Feb. 1997	78.7	31
	Faroe Islands	April 1999	83.7	18
Lemon sole *(Microstomus kitt)*	North Sea	Feb. 1995		64
	Faroe Islands	April 1999	79.7	94
Herring *(Clupea harengus)*	North Sea	Jan. 1995		25
	North Sea	May 1998		55
	Baltic Sea	April 1999	65	34
Mackerel *(Scomber scombrus)*	Bay of Biscay	March 1998	65.2	44
	Faroe Islands	April 1999	68.9	68

The same confusing situation applies to other fish species like haddock, plaice or herring. As mentioned above the fishing ground is only one of various parameters, which can influence the iodine content of fish and other biological effects may interfere. Thus the results can only give a first indication that there tends to be no relation between iodine concentrations in fish muscle and fishing ground.

Variation within a single fishing ground

The large variation of iodine found in cod from closely related fishing grounds indicated a possible inhomogenity within a fish population of a single fishing ground. Therefore, five pooled cod samples of comparable length (35 – 44 cm) were taken from two different batches of fishes, respectively, caught by bottom trawl during the 210. cruise of the research ship „Walther Herwig III". The sampling was repeated at two well separated areas of the Baltic Sea. The first fishing ground „Kieler Bucht" was close to the city of Kiel in the western part of the Baltic Sea, whereas the second ground „Adler Grund" was chosen south-west of Bornholm. Results are compiled in Figure 1 and 2.

The iodine content in five randomly chosen pooled cod samples from the Kieler Bucht ranged only between 941 and 1124 µg I/100 g w. w., presenting a relative homogeneous distribution of iodine in this batch of fishes. Samples from the Adler Grund gave a different picture. The iodine

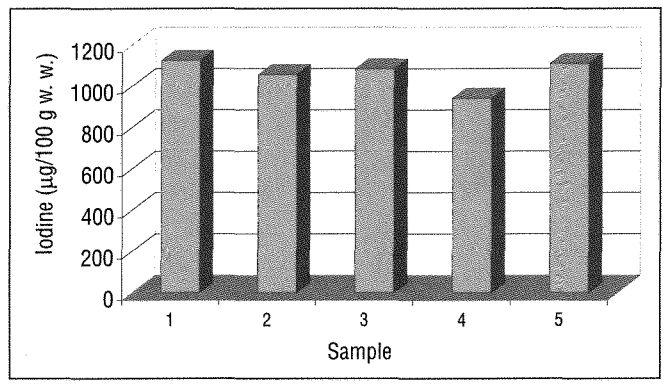

Fig. 1 Variation of iodine in 5 pooled cod samples from one catch (Kieler Bucht, n = 3 fishes/pooled sample)

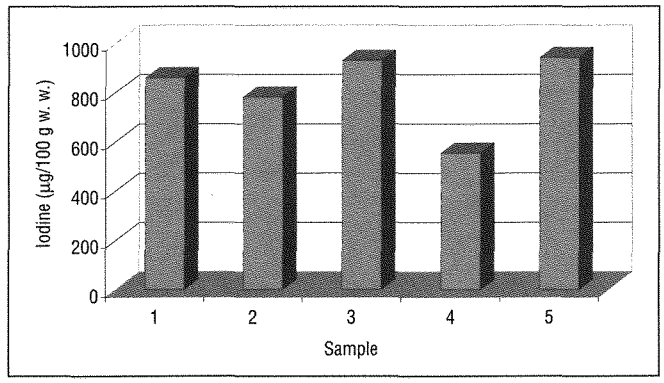

Fig. 2 Variation of iodine in pooled samples from one catch (Adler Grund, n = 3 fishes/pooled sample)

Tab. 5 Iodine in muscle and skin of marine fishes (pooled samples of 3–10 fishes)

Species	No. of fishes	Muscle tissue			Skin	
		Water [%]	Iodine [µg I/100 g w. w.]	% Skin[a)]	Water [%]	Iodine [µg I/100 g w. w.]
Plaice	3	82.0	32	24	79.0	58
Dab	5	84.0	18	22.5	76.0	32
Lemon sole	5	79.7	94	11.5	76.5	70
Long rough dab[b)]	5	81.1	28	19.2	73.2	78
Megrim[c)]	4	77.5	17	16.8	72.9	26
Herring	5	65.0	34	9	64.1	100
Mackerel	4	68.9	68	8.2	54.6	308
Haddock	5	82.3	266	10.9	77.2	713
Haddock	10	80.5	136	9.8	76.9	274
Saithe	5	n.d.[d)]	68	10.8	73.5	1326
Cod	5	79.8	29	12.5	70.7	595
Cod	3	n.d.	889	8.9	n.d.	2510
Cod	3	n.d.	827	8.6	n.d.	1813
Cod	3	n.d.	950	9.1	n.d.	1480

[a)] in relation to skin-on fillets, [b)] *Hippoglossoides platessoides*, [c)] *Lepidorhombus whiffiagonis*, [d)] not determined

content varied from 554 to 941 µg I/100 g w. w., which implies a large difference in iodine levels between individuals. Taking into account that cod from both fishing grounds presumely belong to the same Baltic cod stock[21)] the differences are even more pronounced. The results demonstrate that the iodine content can vary considerably within one fish species, even within one batch.

Iodine in muscle and skin of marine fishes

Eckhoff et al.[16)] reported higher iodine content in skin of some African fresh water fishes, which raised the question of the distribution of iodine in skin and muscle tissue of marine fish species. Thus, the distribution was analysed in pooled samples of 5–10 fishes of various species. Sampling included 5 flat fish species, 3 lean and 2 fat fish species.

The results obtained (Table 5) revealed that iodine can be highly concentrated in the skin of marine fishes. Depending on the species, the skin can obtain up to twentifold of the muscle tissue. These findings may explain some of the contrary results reported in the literature. The skin can obviously contribute considerably to the total iodine content, but often it is not stated if a sample was analysed skin on or off.

Furthermore, the findings could have a beneficial effect for the consumer. Consuming skin-on fillets instead of skinless fillets will improve the daily iodine intake and may lead to a better exploitation of fish resources.

Distribution of iodine in fish muscle

The high iodine concentrations in skin of marine fishes raised the question of the distribution of iodine in the remaining muscle tissue.

Thus single cod fillets from Baltic cod were sliced into three horizontal layers, consisting of the skin, a layer sided towards the skin (skin layer) and a layer sided towards the bone (bone layer) and repeatedly analysed. Figure 3 gives an outline of the selected layers. Data of each layer are compiled in Table 6.

The iodine concentrations found in the different layers are presented in Figure 4, demonstrating an iodine gradient within the muscle tissue.

Calculation the relative amount of iodine in each layer, the skin contains 15–20% of the total iodine, the upper layer approximately 50–60% and the layer sided towards the bones around 30%.

To get a more profound insight on the distribution of iodine in fish muscle, also the variation of iodine content between

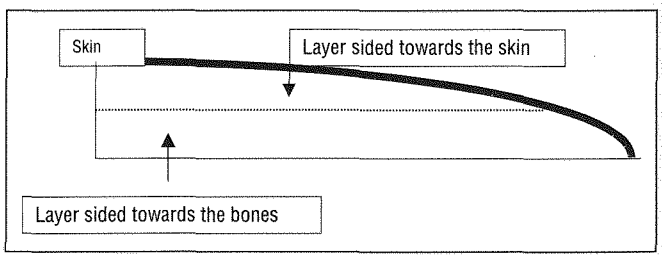

Fig. 3 Cross-section of cod fillets

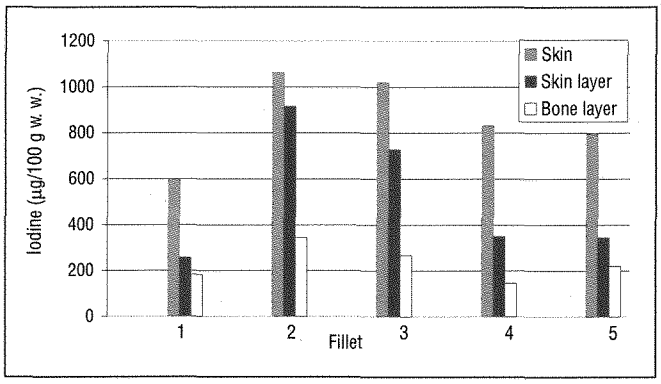

Fig. 4 Iodine content in different layers of cod fillets

Tab. 6 Data on cod fillets sliced into horizontal layers

Fillet	1		2		3		4		5	
	g	% [a)]	g	%	g	%	g	%	g	%
Skin	10.20	8.40	8.30	8.90	9.30	9.50	10.05	8.80	8.32	7.30
Skin Layer	49.10	40.30	39.50	42.10	44.30	45.20	48.53	42.30	52.32	45.70
Bone Layer	62.50	51.30	46.00	49.00	44.60	45.50	56.06	48.90	53.84	47.00
Total Fillet	121.80	100.00	93.80	100.00	98.20	100.00	114.64	100.00	114.48	100.00

[a)] relative amount

left and right skinless fillets, the dorsal and ventral part and between head and tail part of the muscle was investigated.
Taking the revealed iodine gradient within a cod muscle into account, the influence of filleting and deskinning had to be considered. It was tried to minimize variations due to handling by pooling each sample consisting of three left and three right fillets, three dorsal and three ventral parts or three head and tail parts of the fillets, respectively.
All samples were hand filleted on bord of our research ship.
The results are compiled in Figures 5, 6 and 7.
Although some samples showed different iodine levels of corresponding pairs, there is no uniform tendency between left and right fillets, between dorsal and ventral parts and head and tail parts of fillets, respectively.
Notable differences were probably caused by sample preparation, although care was taken. Under board conditions it is difficult to standardize hand filleting, resulting sometimes in different filleting yields.
Generally the iodine distribution in cod muscle tends to be uniform except for the revealed vertical gradient.

Conclusions

According to literature the iodine content in marine fishes can vary considerably at high levels. To study this natural variation in detail a reliable gaschromatographic method was developed, basing on the determination procedure of *Gu* et al.[18].
The methods permits the determination of iodine in lean and fatty fishes with good accuracy (recovery 93–109%).
Results confirm a large variation of iodine content between fishes of the same species. In cod from different fishing areas concentrations between 29 and 1124 µg iodine/100 g w. w. were observed, but a relation to the fishing ground could not be derived from the data.
The amount of iodine varies already considerably within fishes from the same catch, e. g. ranging in Baltic cod muscle between 554 and 941 µg iodine/100 g w. w.. The iodine distribution within a fillet is inhomogeneous with respect of a vertical gradient. The skin contains considerable amounts of iodine, whereas the lowest concentrations are found towards the backbone sided inner part of the fillets. Consequently sample preparation can influence the iodine content to a large extent, which has to be considered when comparing iodine data.
The finding can also have an additional beneficial effect for the consumer. By consuming skin – on fillets, the intake of iodine from fish can be improved up to 20 %.
Although some progress was achieved to explain variations of iodine in marine fish, further investigations are needed to receive a complete understanding of the influence of all biological effects on iodine in fish.

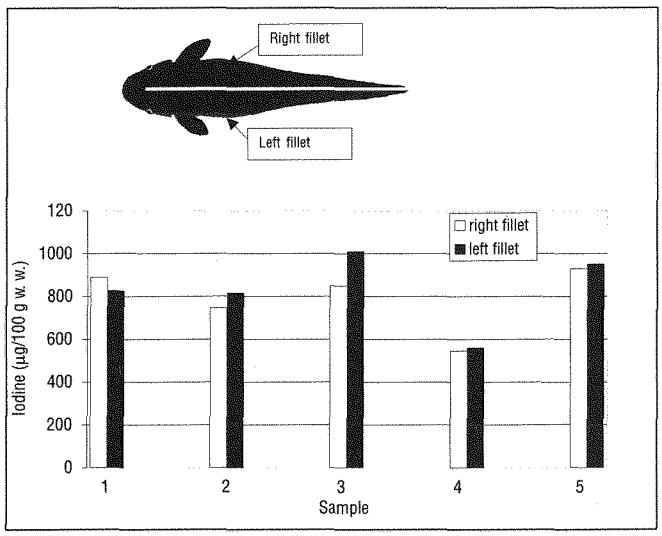

Fig. 5 Variation of iodine content in right and left cod fillets

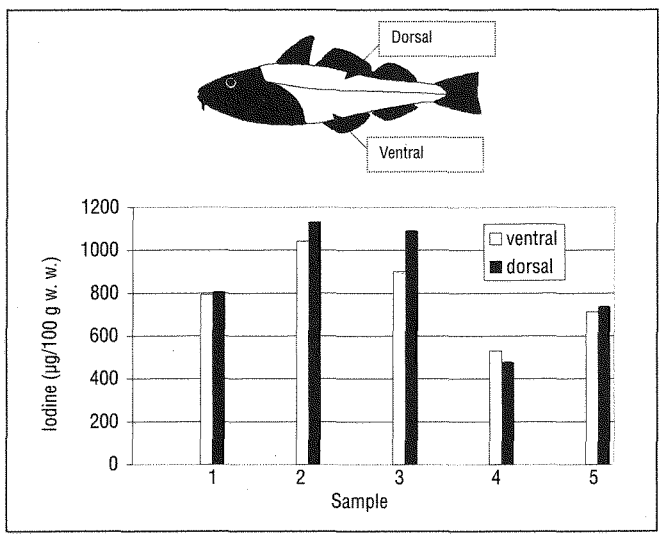

Fig. 6 Distribution of iodine between ventral and dorsal part of cod fillets

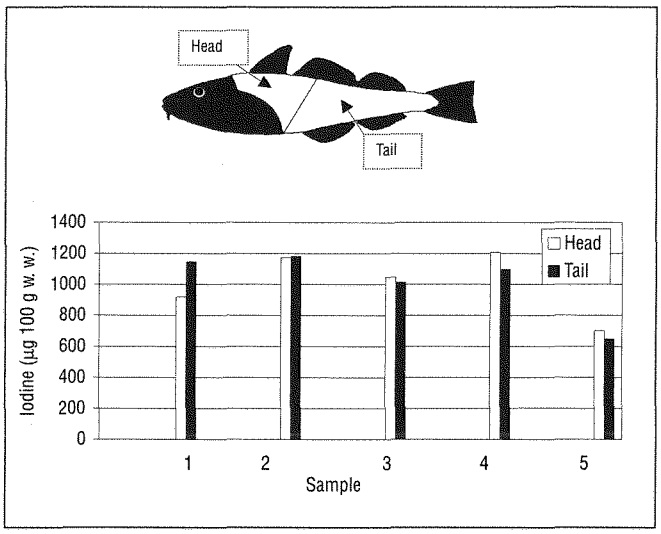

Fig. 7 Distribution of iodine between head and tail part of fillets

References

1) DGE (Deutsche Gesellschaft für Ernährung), Referenzwerte für die Nährstoffzufuhr. 1. Auflage, Umschau-Verlag, Frankfurt/Main (2000).
2) *Heseker, H.*: Iod – Funktion, Physiologie, Stoffwechsel, Empfehlungen und Versorgung in der Bundesrepublik Deutschland. Ernährungs-Umschau **46**, 55–59 (1999).
3) *Sumar, S.* and *H. Ismail*: Iodine in food and health. Nutrition and Food Science **5**, 175–183 (1997).
4) *Donahue, J., A. Robertson* and *D. B. de Benoist*: Comparative analysis of progress on elimination of iodine deficiency disorders (IDD). World Health Organization. Regional Office for Europe, Copenhagen and WHO headquarters, Geneva, 1–37 (1999).
5) *Karl, H.* und *W. Münkner*: Iod in marinen Lebensmitteln. Ernährungs-Umschau **46** (8), 288–291 (1999).
6) *Wenlock, R. W., D. H. Buss, R. E. Moxon* and *N. G. Bunton*: Trace nutrients. 4. Iodine in British food. British J. Nutr. **47**, 381–390 (1982).
7) DFU (Danish Institute of Fisheries Research) pers. communication, Lyngby (1995).
8) *Holland, B., J. Brown* and *D. H. Buss*: Fish and fish products. Third Supplement to the 5th edition of the composition of foods. Ed.: Royal Society of Chemistry, Land and Unwin Ltd., Bugbrooke (1993).
9) *Sidwell, V. D.*: Chemical and nutritional composition of fin fishes, whales, crustaceans, mollusks, and their products. NOAA Technical Memorandum NMFS, F/sec-11, Seattle (1981).
10) *Souci,S. W., W. Fachmann* und *H. Kraut*: Die Zusammensetzung der Lebensmittel-Nährwert-Tabellen. Medpharm GmbH, Scientific Publishers, Stuttgart (1994).
11) *Varo, P., E. Saari, A. Paaso* and *P. Koivistoinen*: Iodine in Finnish foods. Int. J. Vit. Nutr. Res. **52**, 80–89 (1982).
12) *Lee, S. M., J. Lewis* and *D. H. Buss*: Iodine in British foods and diets. British J. Nutr. **72**, 435–446 (1994).
13) *Manthey, M.*: Gehalte an Natrium, Kalium, Jod und Fluorid in Fischerzeugnissen. Deutsche Lebenmittel-Rundschau **85** (10), 318 –321 (1989).
14) *Montag, A.* und *B. Grote*: Untersuchungen zur Iod-Brom-Relation in Lebensmitteln. Z. Lebensm. Unters. Forsch. **172**, 123–128 (1981).
15) *Love, M.*: The chemical biology of fishes. Academic Press, London and New York (1974).
16) *Eckhoff, K. M.* and *A. Maage*: Iodine content in fish and other food products from East Africa analyzed by ICP-MS. J. Food Composition and Analysis **10**, 270–282 (1997).
17) *Fiedlevova, V.*: Spectrophotometric determination of iodine and its content and stability in selected food raw materials and products. Czech. J. Food. Sci. **16**, 163–167 (1998).
18) *Gu, F., A. A. Marchetti* and *T. Straume*: Determination of iodine in milk and oyster tissue samples using combustion and peroxydisulfate oxidation. Analyst **122**, 535–537 (1997).
19) *Mitsuhashi, T.* and *Y. Kaneda*: Gas chromatographic determination of total iodine in foods. J. Assoc. Off. Anal. Chem. **73**, 790–792 (1990).
20) BCR reference material CRM 422, European Commission, Institute for Reference Materials and Measurements (IRMM), Geel, Belgium.
21) Rechlin, O.: Fischbestände in der Ostsee, ihre Entwicklung in den Jahren seit 1970 und Schlußfolgerungen für ihre fischereiliche Nutzung – Teil 1: Dorsch. Inf. Fischwirtsch. Fischereiforsch. **46**, 10–15 (1999).

Europäische Beurteilungsmerkmale für Mayonnaise – Code of Practice

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Einleitung

Seit zwei Jahrhunderten ist Mayonnaise bekannt. Heute ist sie ein Lebensmittel des täglichen Bedarfs, wird industriell hergestellt und international gehandelt. Das Grundrezept ist aber bis heute das gleiche geblieben: Aus Eigelb, Öl, Essig, Salz und Gewürzen wird eine Emulsion hergestellt. Die Produktionsmenge in Deutschland beträgt ca. 28000 t/Jahr.

Seit dem 1. 1. 1993 gibt es den gemeinschaftlichen Binnenmarkt aller EG-Mitgliedstaaten. Jedes ordnungsgemäß nach den Vorschriften des Herstellerlandes hergestellte Erzeugnis kann ohne Behinderung in jedem anderen Mitgliedstaat vermarktet werden. Die EG-Kommission will durch Gemeinschaftsrecht nur noch solche Bereiche regeln, die für alle Lebensmittel in der Gemeinschaft einheitlich gelten sollen. Sofern Regelungen für einzelne Produkte für erforderlich gehalten werden, sollen diese von den beteiligten Wirtschaftsgruppen aufgestellt werden.

Unter dem Begriff „Mayonnaise" wurden in Europa emulgierte Soßen mit einem Fettgehalt zwischen 25% und 80% verstanden. Die deutschen Hersteller haben die Zusammensetzung von Mayonnaise bereits 1968 in den „Leitsätzen für Mayonnaise, Salatmayonnaise und Remou-