

DETERMINATION OF INDOLE AS A FRESHNESS ASSESSMENT OF SHRIMP

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ABSTRACT

Samples of raw and cooked shrimp (*Pandalus borealis*), caught in the Barents Sea, were stored at 0° and 5° for 20 days. The concentration of indole stayed at a low and constant level, increasing only as the shrimp became putrid. Total volatile bases increased evenly, while trimethylamine and trimethylamine oxide showed a lag period before increase, resp. decrease.

INTRODUCTION

Indole is found in shrimp as a result of bacterial decomposition of protein (PONDER, 1978). The concentration of indole increases as the spoilage progresses, and it was of interest to study whether the indole concentration could be used as an index for freshness of shrimp.

Official methods for analysis of indole in fish and marine products have been established (AOAC, 1980 a,b). In the present study, the gas-liquid chromatographic method (AOAC, 1980 b) was modified to permit the use of capillary columns.

In addition to indole, volatile nitrogen compounds were determined as a function of time and storage conditions, and sensory evaluations were performed.

EXPERIMENTAL

The shrimp (prawn, *Pandalus borealis*) used in this study were caught in the Barents Sea during the two last weeks of February 1981. Freshly caught shrimp were frozen immediately and were received frozen at the Central Laboratory. After rapid thawing the shrimp were divided into two portions, one of which was cooked. Samples of raw and cooked shrimp were stored for 20 days in a) ice (0°C) and b) in a refrigerator at 5°C. Samples were withdrawn for the determination of chemical and sensory parameters.

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Indole was determined by three methods:

- 1) Colorimetric method (AOAC, 1980 a)
- 2) Fluorometric method (Ponder, 1978)

These two methods were used without modification.

- 3) Gas liquid chromatographic (GLC) method (AOAC, 1980 b)

This method was modified to permit the use of capillary columns. A Perkin-Elmer F22 gas chromatograph was used, with Rb-silicate nitrogen detector and a Hewlett-Packard 25 m x 0.31 mm i.d. fused silica column coated with methyl silicone. Nitrogen was used as the carrier gas at a column head pressure of 0.6 bar. Temperatures were (°C): injector 220, oven 120, and detector 25. Samples (1 μ l) were injected onto column by the splitless technique.

GLC/mass spectrometry was done on a HP 5992 instrument equipped with a 50 m x 0.21 mm i.d. HP fused silica column coated with methyl silicone.

TVB (total volatile bases), TMA (trimethylamine), and TMAO (trimethylamine oxide) were determined by standard methods (Methods of Analysis, 1979).

Sensory evaluation of shrimp quality was performed by four panelists, who were asked to make a ranking of samples on the basis of appearance, odour, texture, and taste. In addition the panelists had to give a score for each of the samples on a scale where 9 indicated top quality whereas 5 indicated the limit of acceptability.

Table 1. Indole in Shrimp (μ g/100g).
Cooked and in parenthesis uncooked samples.

Days	Colorimetric	Fluorometric	GLC
Temperature 0°C			
0	0	7.0	< 3
3	0	6.8	< 3
6	4.8	7.6	
10	5.0	8.0	< 3
14	7.3	10.0	
17	8.9	8.5	
20	9.2	13.5	
Temperature 5°C			
0	0	7.0 (9.4)	< 3 (< 3)
3	0	7.2 (11.4)	< 3 (< 3)
5	4.0	7.5 (9.2)	< 3 (< 3)
7	4.8	8.1 (10.4)	< 3 (< 3)
10	5.6	9.5 (14.6)	< 3 (< 3)
12	5.3	10.6 (19.5)	< 3 (8)
14	7.5	11.1 (43.6)	< 3 (31)

RESULTS AND DISCUSSION

From table 2 is seen that the development of volatile nitrogen compounds followed the expected patterns. In all cases the concentrations were higher and the changes more pronounced in raw than in cooked samples. The concentration of TMAO decreased while the amounts of TMA and TVB increased with time. There was also a rise in pH with time. The concentrations of TMA and TMAO stayed relatively constant for two weeks at 0° and for 10 days at 5°. The concentration of TVB increased slowly for the first two weeks followed by a faster increase, and correlates well with the results of the sensory evaluation given in Table 3.

On the other hand the indole concentration stayed at a low and constant value beyond the limit of sensory acceptability (Table 1). The concentration rose steeply, however, as the shrimp became putrid. Cooked shrimp stored for 14 days at 5°, then for 2 days at room temperature held 9900 µg indole/100 g shrimp. The identity of indole was in this case established by GC/MS.

The concentration of indole in shrimp caught in the Barents Sea and stored at 0° and 5° is therefore not a suitable index for freshness. It may, however, be used to classify a shrimp sample as putrid.

Table 2. Volatile nitrogen compounds in shrimps (mg N/100g).
Cooked and in parenthesis uncooked samples.

Days	pH	TVB	TMA	TMAO
Temperature 0°C				
1	7.08 (7.00)	3.6 (11.3)	0 (0)	67.9 (188.2)
3	7.10 (7.80)	4.9 (23.5)	0 (0)	60.7 (144.3)
6	7.11 (7.98)	5.1 (31.2)	0 (0.9)	65.7 (145.4)
10	7.25 (8.06)	9.7 (42.5)	0 (0.7)	60.4 (139.2)
14	7.39 (8.04)	23.5 (89.6)	1.3 (4.7)	59.6 (123.8)
17	7.41 (8.18)	26.6 (183.5)	6.4 (19.7)	47.4 (24.4)
20	7.80 (8.14)	75.9 (195.2)	17.5 (64.9)	36.8 (56.5)
Temperature 5°C				
0	7.08 (7.00)	3.6 (11.3)	0 (0)	67.9 (188.2)
3	7.18 (7.89)	5.1 (33.0)	0 (0)	64.9 (154.9)
5	7.15 (8.10)	6.0 (39.2)	0 (0.5)	68.4 (151.6)
7	7.25 (7.90)	17.0 (61.1)	0 (1.8)	64.8 (129.0)
10	7.56 (8.30)	55.4 (199.5)	8.6 (20.1)	50.3 (59.5)
12	8.02 (8.40)	152.1 (245.1)	32.1 (55.8)	28.5 (48.4)
14	7.95 (8.29)	126.2 (255.4)	35.9 (50.7)	28.1 (44.3)

TVB: Total volatile bases

TMA: Trimethylamine

TMAO: Trimethylamine oxide

Table 3. Sensory evaluation of stored cooked shrimp.

Days	Appearance	Odour	Texture	Taste
Temperature 0°C				
0	9	9	9	9
3	9	8	8	8
6	8	7	6	7
10	7	5	5	6
14	7	4	—	—
17	6	4	—	—
20	6	4	—	—
Temperature 5°C				
0	9	9	9	9
3	8	8	7	8
5	8	7	6	7
7	8	5	5	5
10	5	4	—	—
12	5	3	—	—
14	4	3	—	—

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REFERENCES

- Methods of Analysis – Central Laboratory, Bergen. 1979. No. 7.
 Official Methods of Analysis, 13th Ed., 1980. AOAC, Washington DC, sec. 18.072.
 Official Methods of Analysis, 13th Ed., 1980. AOAC, Washington DC, sec. 18.075.
 PONDER, C., 1978. *J.A.O.A.C.* 61, 1089–1091.