

Application Vapodest

C.11. TVB-N in Fish and Fish Products



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1 Principle

When determining the freshness of fish products, the main focus is on sensory evaluation criteria such as appearance, smell, taste or the texture of the fillets. In addition, also chemical parameters such as pH-value or the TVB-N content (Total Volatile Basic Nitrogen) as well as the bacterial count are determined.

In order to determine the TVB-N content, the volatile basic nitrogen compounds - after alkalization - are expelled from a perchloric acid extract of the sample using water steam and received in an acidic receiver. A titrimetric determination of the absorbed bases follows.

2 Methods

This application note is meant to be a guideline for the operation of your C. Gerhardt analysis system and has to be adapted to your sample matrix and the local circumstances in your laboratory.

This document is based on

- BVL-Methode L10.00-3 Bestimmung des Gehaltes von flüchtigen stickstoffhaltigen Basen (TVB-N) in Fischen und Fischerzeugnissen – Referenzverfahren
- Official Journal of the European Communities, No L 97/page 84 (procedure for TVB-N concentrations from 5 mg/100 g to at least 100 mg/100 g, Precision requirements: The applied method is correct if the difference of the duplicates is not higher than 2 mg/100 g.

3 Chemicals

Quality grade p. a.

- 3.1. Water: demineralized or distilled
- 3.2. Perchloric acid solution $c = 0,6 \text{ mol/l}$
- 3.3. Sodium hydroxide NaOH 20 %
- 3.4. Boric acid H_3BO_3 0,3 %
- 3.5. Tashiro mixed indicator
- 3.6. Standard solution: Hydrochloric acid $c(\text{HCl}) = 0.01 \text{ mol/l}$ or sulphuric acid $c(\text{H}_2\text{SO}_4) = 0.005 \text{ mol/l}$
- 3.7. Phenolphthalein solution
- 3.8. Silicone antifoam

4 Instruments

- Mechanical shredder, as e.g. Moulinette or a similar mechanical shredder, or dispenser as e.g. ULTRA TURRAX; IKA to prepare a homogeneous paste
- Analytical balance (reading accuracy 0.1 mg)
- VAPODEST Steam distillation system, models 200 to 450 without titrator, titration has to be performed by means of a manual burette (class A, according to ISO 385), 50 ml nominal volume, with volume scale in 0.05 ml steps or a titrator, or instead of indicator solution with a pH meter with a combination electrode.
- Recommendation distillation flask Kjeldahl-750 ml, with enlarged neck (12-0314) or Jumbo digestion tube 1200 ml (12-0243), for VAPODEST 500c Jumbo-digestion tube 400 ml (12-0310)

5 Procedure

5.1 Sample Preparation

A representative average sample is crushed e.g. with a Moulinette. Approx. 10 g (standard) of this sample are weighed into a vessel which is suitable for working with a dispensing device (such as Ultra-Turrax, IKA). Carefully add 90 ml of perchloric acid solution (3.2) to this fish mash. The mixture is then homogenized for 1 to 2 minutes with

the Ultra-Turrax. This solution is then filtered through a folded filter while the filter is discarded. 50 ml of the extract are filled into a Kjeldahl flask KTG 250 ml (12-0301). If the sample foams heavily, we recommend using a silicone based defoaming reagent. For later check of sufficient alkalinization, a few drops of phenolphthalein indicator solution (3.7) are added to the extract.

5.2 Distillation with VAPODEST

	Methode Fish TVB-N	VAP 200	VAP 300	VAP 400	VAP 450	VAP 500 / 500c
H ₂ O addition	0 ml	•	✓	✓	✓	✓
NaOH addition	10 ml	✓	✓	✓	✓	✓
Reaction time	0 s	✓	✓	✓	✓	✓
Distillation time	600 s	✓	✓	✓	✓	✓
Steam power	50 %	✓	✓	✓	✓	✓
Sample suction	0 s	-	✓	✓	✓	✓
H ₃ BO ₃ addition	80 ml	•	•	✓	✓	✓
Suction receiver solution	30 s	-	-	-	✓	✓
Titration	-	•	•	•	✓	✓
Calculation	-	•	•	•	•	✓
Reading pH value, fixed endpoint or automatic endpoint	-	-	-	-	✓	✓
Titration online	-	-	-	-	-	✓

✓ = automatic

• = manual

- = not applicable

Choose the method from the method library or program an older VAPODEST unit following the method „ Fish TVB-N“.

5.3 Titration

Add 3-4 drops of Tashiro indicator (3.5) to the receiver solution (3.4) and titrate with standard solution (3.6) until the colour changes from green to grey. If you determine the endpoint with a pH meter or a titrator, you do not have to add the Tashiro indicator.

5.4 Determination of Blank Value

For blank value determination perform the analysis just with the indicated chemicals for distillation and titration. The received consumption of the titration solutions has to be taken into account for the calculation of the result.

6 Calculation

6.1 TVB-N calculation in mg/100g

The percentage mass fraction of TVB-N nitrogen in the sample is calculated with the following equation:

$$TVB - N \left(\frac{mg}{100g} \right) = \frac{(V_1 - V_0) \times c_{eq,soll} \times t \times 100 \times 2 \times 14}{m_{Sample}}$$

V_1 = Volume standard solution which has been used for amount of sample [ml]

V_0 = Volume standard solution, which has been used for blank test [ml]

$c_{eq,soll}$ = equivalent concentration standard solution

t = Titer standard solution

14 = Factor for recalculating N content

2 = Aliquot

100 = Factor for the Unit

m = Sample Weight [g]

6.2 Accuracy

6.2.1 Repeatability

The maximum deviation of duplicate determinations respectively repeat determinations must not exceed the following values:

$r=3$ mg/100 g

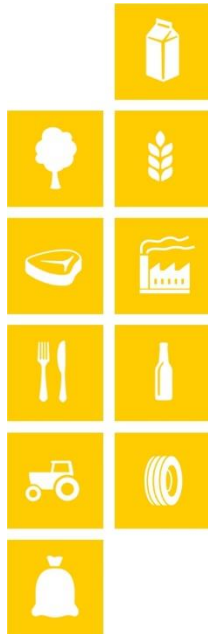
$S_{(r)}=\pm 1$ mg/100 g

6.2.2 Reproducibility

Reproducibility is defined as the absolute difference between two single test results, performed by different operators in different laboratories, following the same method with identical samples.

$R=8$ mg/100 g

$S_{(R)}=\pm 3$ mg/100 g



UMFANGREICHE APPLIKATIONS DATENBANK

C. Gerhardt bietet umfangreiche Applikationen zu den unterschiedlichsten Methoden und Verfahren. Bitte fragen Sie nach weiteren Applikationen, z.B:

- Stickstoffbestimmung in Lebens- und Futtermitteln nach Kjeldahl und Dumas
- Rohfaser, ADF und NDF in Futtermitteln
- Fettbestimmung in Lebens- und Futtermitteln
- Alkoholbestimmung
- Gesamtcyanidbestimmung in Wasser
- Schwermetall in Böden und Schlämmen
- CSB-Wert von Wasser
- Gesamtstickstoff in Wasser, Böden und Pflanzen
- uvm.

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HYDROLYSE VOLLAUTOMATISCH

HYDROTHERM – automatisches Säure-Hydrolysesystem für die Fettbestimmung nach Weibull-Stoldt. Zusammen mit SOXTHERM ist HYDROTHERM eine ideale Systemlösung zur Gesamtfettbestimmung.

FETTEXTRAKTION VOLLAUTOMATISCH

SOXTHERM – automatisches Schnell-Extraktionssystem zur Fettbestimmung.

WASSERDAMPF-DESTILLATION VOLLAUTOMATISCH

VAPODEST – Schnell-Destilliersystem zur Stickstoff- und Proteinbestimmung nach Kjeldahl und Wasserdampf-destillation als Probenvorbereitung für weitere Analysen.

STICKSTOFFANALYSE VOLLAUTOMATISCH

DUMATHERM – Stickstoff-/Proteinbestimmung von festen und flüssigen Proben nach der Verbrennungsmethode von Dumas. Für fast alle Probenmatrices eine schnelle und komfortable Alternative zur klassischen Kjeldahl-Methode.

ROHFASEREXTRAKTION VOLLAUTOMATISCH

FIBRE THERM – vollautomatische Abarbeitung der Koch-, Wasch- und Filtrationsvorgänge bei der Rohfaser-, ADF- und NDF-Bestimmung.

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