

SLOVENSKI STANDARD
oSIST prEN ISO 19343:2016
01-junij-2016

Mikrobiologija v prehranski verigi - Odkrivanje prisotnosti in kvantifikacija histamina v ribah in ribjih proizvodih - Metoda HPLC (ISO/DIS 19343:2016)

Microbiology of the food chain - Detection and quantification of histamine in fish and fishery products - HPLC method (ISO/DIS 19343:2016)

Mikrobiologie der Nahrungskette - Nachweis und Bestimmung von Histamin in Fisch und Fischereierzeugnissen - HPLC-Methode (ISO/DIS 19343:2016)

Microbiologie de la chaîne alimentaire - Détection et quantification de l'histamine par méthode HPLC (ISO/DIS 19343:2016)

Ta slovenski standard je istoveten z: prEN ISO 19343

ICS:

07.100.30	Mikrobiologija živil	Food microbiology
67.120.30	Ribe in ribji proizvodi	Fish and fishery products

oSIST prEN ISO 19343:2016

en

DRAFT INTERNATIONAL STANDARD

ISO/DIS 19343

ISO/TC 34/SC 9

Secretariat: **AFNOR**Voting begins on:
2016-03-31Voting terminates on:
2016-06-29

Microbiology of the food chain — Detection and quantification of histamine in fish and fishery products — HPLC method

Microbiologie de la chaîne alimentaire — Détection et quantification de l'histamine par méthode HPLC

ICS: 07.100.30

iTeh STANDARD PREVIEW
(standards.iteh.ai)

SIST EN ISO 19343:2017

<https://standards.iteh.ai/catalog/standards/sist/a26f396e-09fb-4210-a271-692294fbb88c/sist-en-iso-19343-2017>

THIS DOCUMENT IS A DRAFT CIRCULATED FOR COMMENT AND APPROVAL. IT IS THEREFORE SUBJECT TO CHANGE AND MAY NOT BE REFERRED TO AS AN INTERNATIONAL STANDARD UNTIL PUBLISHED AS SUCH.

IN ADDITION TO THEIR EVALUATION AS BEING ACCEPTABLE FOR INDUSTRIAL, TECHNOLOGICAL, COMMERCIAL AND USER PURPOSES, DRAFT INTERNATIONAL STANDARDS MAY ON OCCASION HAVE TO BE CONSIDERED IN THE LIGHT OF THEIR POTENTIAL TO BECOME STANDARDS TO WHICH REFERENCE MAY BE MADE IN NATIONAL REGULATIONS.

RECIPIENTS OF THIS DRAFT ARE INVITED TO SUBMIT, WITH THEIR COMMENTS, NOTIFICATION OF ANY RELEVANT PATENT RIGHTS OF WHICH THEY ARE AWARE AND TO PROVIDE SUPPORTING DOCUMENTATION.

ISO/CEN PARALLEL PROCESSING

This draft has been developed within the European Committee for Standardization (CEN), and processed under the **CEN lead** mode of collaboration as defined in the Vienna Agreement.

This draft is hereby submitted to the ISO member bodies and to the CEN member bodies for a parallel three month enquiry.

To expedite distribution, this document is circulated as received from the committee secretariat. ISO Central Secretariat work of editing and text composition will be undertaken at publication stage.



Reference number
ISO/DIS 19343:2016(E)

© ISO 2016

iTeh STANDARD PREVIEW (standards.iteh.ai)

SIST EN ISO 19343:2017

<https://standards.iteh.ai/catalog/standards/sist/a26f396e-09fb-4210-a271-692294fbb88c/sist-en-iso-19343-2017>



COPYRIGHT PROTECTED DOCUMENT

© ISO 2016, Published in Switzerland

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

Contents

Foreword.....	4
Introduction	5
1 Scope.....	1
2 Principle	1
3 Reagents and products	1
4 Apparatus	2
5 Procedure	3
5.1 Sample preparation	3
5.2 Extraction	3
5.3 Derivatization	3
5.4 Purification	3
5.5 LC conditions.....	4
5.6 Standard samples.....	4
6 Calculation.....	4
6.1 Calibration line (curve)	4
6.2 Histamine quantification	4
Annex A (informative) Recommendations for HPLC separation	6
Annex B (informative) Precision and trueness	8
Bibliography	11

SIST EN ISO 19343:2017

<https://standards.iteh.ai/catalog/standards/sist/a26f396e-09fb-4210-a271-692294fbb88c/sist-en-iso-19343-2017>

ISO/DIS 19343:2016(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: [Foreword - Supplementary information](#).

ISO 19343 was prepared the European Committee for Standardization (CEN) in collaboration with Technical Committee TC 34, *Food products*, Subcommittee SC 9, *Microbiology*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

Introduction

Histamine is a causative agent of scombroid poisoning or histamine fish poisoning. Histamine can be present mainly in Scombridae (Tuna, Mackerel) and Clupeidae (Herring, Sardine), in species which contain a high level of free histidine. Histamine is formed through the decarboxylation of histidine by microbiological histidine decarboxylase.

Histamine is defined as a biologically active low molecular weight basic nitrogenous molecule. The consumption of food containing significant concentration of histamine can cause symptoms similar to those associated to seafood allergies.

This standard was developed in response to the need of standardizing a method for histamine detection and quantification in fish and fishery products, in particular for the European regulation 2073/2005 on microbiological criteria for foodstuffs.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

SIST EN ISO 19343:2017

<https://standards.iteh.ai/catalog/standards/sist/a26f396e-09fb-4210-a271-692294fbb88c/sist-en-iso-19343-2017>

Microbiology of the food chain — Detection and quantification of histamine in fish and fishery products — HPLC method

1 Scope

This International Standard specifies a High Performance Liquid Chromatography (HPLC) method to titrate histamine in fish and fishery products intended for human consumption.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN ISO 3696, *Water for analytical laboratory use — Specification and test methods (ISO 3696)*

3 Principle

This method enables to separate histamine among biogenic amines of fish and fishery products. The sample is extracted by mixing with perchloric acid. Precolumn derivatization is performed using dansyl chloride. The biogenic amines and the components in the solution are separated by HPLC with appropriate column, using UV detection. After derivatization, histamine mass concentration is calculated from the peak area ratio of histamine and internal standard, with a calibration curve.

4 Reagents and materials

Use only reagents of recognized analytical grade and water complying with grade 1 of EN ISO 3696, unless otherwise specified. Solvents shall be of quality for HPLC analysis, unless otherwise specified.

4.1 Acetone, CH_3COCH_3 .

4.2 Acetonitrile, HPLC quality, CH_3CN .

4.3 Toluene, C_7H_8 .

4.4 Water HPLC quality.

4.5 Bidistilled water.

4.6 Nitrogen.

4.7 Perchloric acid, substance concentration $c(\text{HClO}_4) = 0,2 \text{ mol/l}$ (recommended).

Add 19,5 ml of HClO_4 65 % or 17,2 ml of HClO_4 70 % to 1 000 ml of water (3.4). The solution is stable for six months if stored at room temperature (15°C to 25°C).

ISO/DIS 19343:2016(E)

4.8 Sodium carbonate, (Na₂CO₃) solution.

Dissolve 110 g of sodium carbonate until complete saturation in about 150 ml of water (3.4). The solution is stable for three months if stored at 5 °C.

4.9 Dansyl chloride solution, mass concentration $\rho = 7,5$ mg/ml.

Dissolve 0,375 g of dansyl chloride in 50 ml of acetone (3.1). The solution is stable for three weeks if stored in the dark at a temperature lower than $-18\text{ °C} \pm 2\text{ °C}$.

4.10 L-proline solution, $\rho = 100$ mg/ml.

Dissolve 1 g of L-proline in 10 ml of water (3.4). The solution is stable for three weeks if stored at $5\text{ °C} \pm 2\text{ °C}$.

4.11 Histamine stock solution, $\rho = 500$ mg/kg.

Dissolve 1,034 g of histamine dihydrochloride in 50 ml of water (3.4). The solution is stable for one year if stored at $-18\text{ °C} \pm 2\text{ °C}$.

4.12 Internal Standard (IS) 1,7-diaminoheptane stock solution, $\rho(\text{C}_7\text{H}_{18}\text{N}_2) = 6,4$ mg/ml. (recommended)

Dissolve 0,320 g of 1,7-diaminoheptane in 50 ml of water (3.5). The solution is stable for three weeks if stored at $5\text{ °C} \pm 2\text{ °C}$.

5 Apparatus**5.1 Grinder, e.g. mixer, blender.** [SIST EN ISO 19343:2017](https://standards.iteh.ai/catalog/standards/sist/a26f396e-09fb-4210-a271-19343-2017)**5.2 Balances, (precisions 0,1 g and 0,001 g).** <https://standards.iteh.ai/catalog/standards/sist/a26f396e-09fb-4210-a271-19343-2017>**5.3 Crusher, (ultra-turrax) with metallic rods.****5.4 Centrifuge, refrigerated capable of a centrifugal force of $8\,000 \times g$ for 100 ml tubes.****5.5 Centrifuge tubes, (plastic) with closing caps, 100 ml.****5.6 Pipettes, ranges 10 μl , 20 μl to 200 μl and 100 μl to 1000 μl .****5.7 Tubes, (temperature resistant glass) with closing caps, 10 ml.****5.8 Vortex.****5.9 Water bath, $60\text{ °C} (\pm 1\text{ °C})$ with dark cover or equivalent.****5.10 Refrigerator, capable of temperatures between 0 °C and 10 °C** **5.11 Freezer, capable of temperatures between -20 °C and -30 °C** **5.12 Gas flow evaporator.**