

**Loomasööt. Fluoriidi sisalduse määramine pärast soolhappega töötlemist ioontundliku elektrodmeetodiga (ISE)**

**Animal feeding stuffs - Determination of fluoride content after hydrochloric acid treatment by ion-sensitive electrode method (ISE)**

## EESTI STANDARDI EESSÕNA

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ICS 65.120

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English Version

## Animal feeding stuffs - Determination of fluoride content after hydrochloric acid treatment by ion-sensitive electrode method (ISE)

Aliments des animaux - Détermination de la teneur en fluorure, après traitement à l'acide chlorhydrique, selon la méthode utilisant une électrode sélective d'ions (ISE)

Futtermittel - Bestimmung des Fluoridgehaltes nach Salzsäure-Behandlung mit ionensensitiver Elektrode (ISE)

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## Foreword

This document (EN 16279:2012) has been prepared by Technical Committee CEN/TC 327 “Animal Feeding stuffs – Methods of sampling and analysis”, the secretariat of which is held by NEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by January 2013, and conflicting national standards shall be withdrawn at the latest by January 2013.

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## Introduction

Fluorine (F) is one of the most abundant elements in the environment. Animals are exposed to the ionic form of the element, fluoride (F<sup>-</sup>), which may be present in feeding stuffs. The toxicity of fluoride has already been established by EFSA [6]; and its availability greatly depends on the solubility of fluoride compounds.

In particular, fluoride compounds with low solubility are poorly absorbed while fluoride ions released from readily soluble compounds are almost completely absorbed from the gastrointestinal tract by passive diffusion in monogastric species. The extraction procedure of this method involves a mild acid treatment with hydrochloric acid solution of 1 mol/l which should reflect the gastric hydrochloric acid concentration of 0,1 mol/l– 0,3 mol/l.

## 1 Scope

This European Standard specifies an Ion-Selective Electrode method (ISE) after hydrochloric acid treatment for the determination of fluoride from animal feeding stuffs. The content of fluoride ( $F^-$ ) corresponds to that of fluorine (F) specified in Commission Regulation (EU) 574/2011[3].

This European Standard is strictly based on several conventions such as those contained in the following example:

**EXAMPLE** 0,5 g test portion for extraction of fluoride from animal feeds by means of an acid treatment with 20 ml of 1 mol/l hydrochloric acid solution at ambient temperature (20 °C to 25 °C) for 20 min. The pH is controlled and adjusted to 5,5 in the buffered test solution before determination of fluoride by ISE using standard addition technique.

The method was successfully tested in an interlaboratory study in concentrations between 100 mg/kg up to 500 mg/kg. If this method is followed strictly, then theoretically all concentrations from 40 mg/kg up to 4 000 mg/kg can be analysed within the linear calibration function.

Only for concentrations lower than 40 mg/kg is the use of an interpolation technique required instead of standard addition Annex C.

The quantification limit for fluoride using the conventions of the method including the standard addition technique is 40 mg/kg or lower than 2,5 mg/kg when using interpolation Annex C.

## 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*

EN ISO 6498, *Animal feeding stuffs — Guidelines for sample preparation*

## 3 Principle

For the determination of fluoride, a test portion of 0,5 g of the sample is treated with 20 ml of 1 mol/l hydrochloric acid solution for 20 min at ambient temperature (20 °C to 25 °C).

The amount of fluoride extracted from the sample is determined by means of a fluoride selective electrode.

**NOTE** This ISE method is based upon a potentiometric technique. This means that it is based upon the measurement of a concentration of an analyte present in solution, by means of an ion selective electrode. This electrode has a linear response within a working range of analyte concentrations, which is provided by the calibration curve. Thus, in general, the operator should take an adequate amount of sample to ensure that the final concentration of the analyte is within this working range given by the calibration step. To ensure satisfactory reproducibility of the method however, this extraction procedure as a convention method fixes the ratio of the amount of the test portion to the volume of the extraction solvent for all kind of feeds.

**WARNING — The use of this European Standard can involve hazardous materials, operations and equipment. This standard does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this European Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.**