EAST AFRICAN COMMUNITY

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EAS 318 (2002) (English): Animal and vegetable fats and oils — Determination of soap content

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EAS 318:2002
ICS 67.200.10

# EAST AFRICAN STANDARD

## Animal and vegetable fats and oils — Determination of soap content

## EAST AFRICAN COMMUNITY


First Edition 2002

## Foreword

Development of the East African Standards has been necessitated by the need for harmonizing requirements governing quality of products and services in East Africa. It is envisaged that through harmonized standardization, trade barriers which are encountered when goods and services are exchanged within the Community will be removed.

In order to achieve this objective, the Partner States in the Community through their National Bureaux of Standards, have established an East African Standards Committee.

The Committee is composed of representatives of the National Standards Bodies in Partner States, together with the representatives from the private sectors and consumer organizations. Draft East African Standards are circulated to stakeholders through the National Standards Bodies in the Partner States. The comments received are discussed and incorporated before finalization of standards, in accordance with the procedures of the Community.

East African Standards are subject to review, to keep pace with technological advances. Users of the East African Standards are therefore expected to ensure that they always have the latest versions of the standards they are implementing.



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INTERNATIONAL STANDARD

**ISO 10539**

First edition
2002-03-15

# Animal and vegetable fats and oils — Determination of alkalinity

*Corps gras d'origines animale et végétale — Détermination de l'alcalinité*

Reference number
ISO 10539:2002(E)

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# Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 10539 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 11, *Animal and vegetable fats and oils*.

Annex A of this International Standard is for information only.

# Animal and vegetable fats and oils — Determination of alkalinity

## 1 Scope

This International Standard specifies a method for the determination of the alkalinity of animal and vegetable fats and oils without distinguishing between the various constituents. The method is not applicable to dry melted animal fats, nor to oils and fats with an acidity greater than 60 % (mass fraction) as determined in accordance with ISO 660.

NOTE Fats and oils can contain alkaline constituents either naturally (e.g. calcium soaps from bones) or accidentally (e.g. sodium soaps in imperfectly refined fats and oils).

## 2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 660:1996, *Animal and vegetable fats and oils — Determination of acid value and acidity*

ISO 661:1989, *Animal and vegetable fats and oils — Preparation of test sample*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

## 3 Term and definition

For the purposes of this International Standard, the following term and definition apply.

**3.1**
**alkalinity**
proportion of alkali in a fat or oil determined by titration with hydrochloric acid, in accordance with the method specified in this International Standard

## 4 Principle

A test portion is dissolved in warm aqueous acetone and titrated with hydrochloric acid.

## 5 Reagents

Use only reagents of recognized analytical grade, unless otherwise stated.

**5.1 Water**, conforming to grade 3 of ISO 3696.

### 5.2 Aqueous acetone

Mix 970 ml of acetone, 20 ml of water and 10 ml of bromophenol blue solution (2 g/l in acetone). Add sodium hydroxide solution [$c$(NaOH) = 0,1 mol/l] to give a blue colour, then add hydrochloric acid [$c$(HCl) = 0,1 mol/l] until a yellowish green colour appears.

**5.3 Hydrochloric acid standard volumetric solution**, $c$(HCl) = 0,01 mol/l, standardized not more than 7 days before use.

## 6 Apparatus

Usual laboratory apparatus and, in particular, the following.

**6.1 Borosilicate glass apparatus**, for aqueous acetone (5.2).

**6.2 Conical flasks**, with wide necks, and of capacities 400 ml to 500 ml.

## 7 Sampling

It is important that the laboratory receive a sample which is truly representative and has not been damaged or changed during transport or storage.

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 5555.

## 8 Preparation of test sample

Prepare the test sample in accordance with ISO 661.

## 9 Procedure

### 9.1 Preparation of the apparatus

Rinse each conical flask (6.2) and stirrer to be used with successive 20 ml amounts of aqueous acetone (5.2) until the aqueous acetone does not change colour. Allow the flasks and stirrer to dry.

### 9.2 Test portion

From the test sample (clause 8) weigh, to the nearest 0,1 g, a test portion of up to 40 g (see Table 1) according to the expected result and colour of the sample. Melt a solid test portion at below the boiling point of acetone. Add 100 ml of the acetone (5.2) at 40 °C and stir. Allow to settle until the solution splits in two layers; in the presence of soap, the upper layer is blue-coloured.

**Table 1 — Mass of test portion**

| Colour of sample | Expected result<br>mg sodium oleate/kg of sample | Mass<br>g |
|---|---|---|
| Light | up to 500 | 40 |
| Light | over 500 to 1 000 | 10 |
| Light | over 1 000 | 4 |
| Dark | not relevant | 2 |
| Very dark | not relevant | 1 |

For test portions of less than 10 g, take replicate portions totalling at least 5 g for titration and sum the masses and titres to give the result for one determination.

### 9.3 Determination

Titrate the warm (30 °C to 40 °C) solution with the hydrochloric acid (5.3) while stirring until the colour of the indicator changes from blue (or blue green) to the yellowish green of the acetone (5.2) added. Allow the mixture to settle in order to observe the colour clearly.

NOTE An explosion-resistant hotplate with magnetic stirrer and burette support is particularly useful.

### 9.4 Number of determinations

Carry out two determinations on the same test sample, treating the replications for small test portions as one determination (see Table 1).

## 10 Expression of results

Alkalinity may be expressed as a mass, in milligrams, of sodium oleate per kilogram of fat or oil, or as a percentage (mass fraction) of sodium hydroxide:

a) the alkalinity in terms of sodium hydroxide is given by the expression

$$A = \frac{4Vc}{m}$$

b) the alkalinity in terms of sodium oleate is given by the expression

$$S = 304\ 000\ \frac{Vc}{m}$$

where

$A$ is the alkalinity expressed as a percentage (mass fraction) of sodium hydroxide;

$S$ is the alkalinity expressed as milligrams of sodium oleate per kilogram of sample;

$m$ is the mass of the test portion, in grams;

$c$ is the exact concentration of the standard volumetric hydrochloric acid solution used, in moles per litre;

$V$ is the volume of hydrochloric acid standard volumetric solution used to titrate the test portion, in millilitres.

Take as the result the mean of two determinations, provided that the requirement for repeatability (see 11.2) is met.

## 11 Precision

### 11.1 Interlaboratory test

Details of an interlaboratory test are summarized in annex A. The values derived from this interlaboratory test may not be applicable to concentration ranges other than those given.

### 11.2 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will in not more than 5 % of cases exceed the repeatability limit $r$ given in Table A.1 for the relevant level of the mean value determined.

### 11.3 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories by different operators using different equipment, will in not more than 5 % of cases exceed the reproducibilty limit $R$ given in Table A.1 for the relevant level of the mean value determined.

## 12 Test report

The test report shall specify:

— all information necessary for the complete identification of the sample;

— the sampling method used, if known;

— the test method used, with reference to this International Standard;

— all operating conditions not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the test results;

— the test results obtained or, if the repeatability has been checked, the final result obtained.

# Annex A
(informative)

# Results of an interlaboratory trial

An international collaborative test has been carried out on the method given in this International Standard. The test, involving fifteen laboratories in six countries, was organized by AFNOR and the results obtained were subjected to statistical analysis in accordance with ISO 5725-2 to give the precision data reported in Table A.1.

**Table A.1 — Precision data**

| | Sample D | Sample E | Sample H |
|---|---|---|---|
| Number of laboratories | 15 | 15 | 15 |
| Number of laboratories retained after eliminating outliers | 14 | 11 | 12 |
| Mean alkalinity (mg/kg) | 8,3 | 216,3 | 12,5 |
| Repeatability standard deviation, $s_r$ (mg/kg) | 0,7 | 0,6 | 0,5 |
| Repeatability coefficient of variation (%) | 8,1 | 0,3 | 3,6 |
| Repeatability limit, $r$ (mg/kg) | 1,9 | 1,8 | 1,3 |
| Reproducibility standard deviation, $s_R$ (mg/kg) | 2,3 | 8,4 | 9,5 |
| Reproducibility coefficient of variation (%) | 27,1 | 3,9 | 76,1 |
| Reproducibility limit, $R$ (mg/kg) | 6,4 | 23,7 | 27,0 |

# Bibliography

[1] ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

[2] ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

[3] ISO 5555, *Animal and vegetable fats and oils — Sampling*

[4] ISO/TC 34/SC 11 N 683, *Animal and vegetable fats and oils — Determination of alkalinity — Results of an international collaborative test*

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