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Palm oil — Determination of the deterioration of bleachability index (DOBI) and carotene content

Huile de palme — Détermination de la détérioration de l'indice de blanchiment (DOBI) et de la teneur en carotène

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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 2.

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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 17932 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 11, *Animal and vegetable fats and oils*.

This second edition cancels and replaces the first edition (ISO 17932:2005), which has been technically revised.

Palm oil — Determination of the deterioration of bleachability index (DOBI) and carotene content

1 Scope

This International Standard specifies a method for the determination of the deterioration of bleachability index (DOBI) of crude palm oil and the carotene content of crude or bleached palm oil and their fractions by spectrophotometric examination in the ultraviolet and visible range of the spectrum.

2 Normative references

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

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

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

ISO Guide 34, *General requirements for the competence of reference material producers*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
deterioration of bleachability index
DOBI
I_{DoB}
ratio of the absorbance of the test portion at 446 nm to that at 269 nm, as determined spectrophotometrically in a 10 mm (1 cm) pathlength cell

NOTE DOBI is expressed to one decimal place without a dimension.

3.2
carotene content of vegetable oil
w_C
mass fraction of β-carotene in oil

NOTE The carotene content of vegetable oil is expressed in milligrams per kilogram.

4 Principle

A homogenized sample is dissolved in isooctane and the absorbance is measured spectrophotometrically at 446 nm and 269 nm. The DOBI value is the ratio of the absorbance at 446 nm to that at 269 nm. The test is a measure of the ease of refining crude palm oil. A low DOBI value can indicate difficulty in refining the oil to a low Lovibond colour. The absorbance at 446 nm is used for calculation of the carotene content.

5 Reagents

WARNING — Attention is drawn to the regulations which specify the handling of dangerous substances. Technical, organizational and personal safety measures shall be followed.

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and distilled or demineralized water or water of equivalent purity.

5.1 Solvent: isooctane (2,2,4-trimethylpentane), having an absorbance less than 0,12 at 230 nm and less than 0,05 at 250 nm against water, measured in a cell with a pathlength of 10 mm (1 cm).

6 Apparatus

The glassware used for the determination shall be thoroughly cleaned and rinsed with the solvent (5.1) before use, so that it is free from impurities having an absorbance within the wavelength range of 220 nm to 500 nm.

Usual laboratory apparatus and, in particular, the following.

6.1 Spectrometer, capable of operating in the ultraviolet and visible range and using 10 mm (1 cm) pathlength quartz cells and preferably having a recording instrument.

Before use, it is recommended that the wavelength and absorbance scales of the spectrometer be checked as follows.

a) **Wavelength scale**: This may be checked using a reference material consisting of an optical glass filter containing holmium oxide which has distinct absorption bands. The reference material is designed for the verification and calibration of the wavelength scales of visible and ultraviolet spectrophotometers having nominal spectral bandwidths of 5 nm or less. The holmium glass filter is measured in the absorbance mode against an air blank over the wavelength range of 640 nm to 240 nm. For each spectral bandwidth (0,10 – 0,25 – 0,50 – 1,00 – 1,50 – 2,00 and 3,00), a baseline correction is performed with an empty cell holder. The wavelengths of the spectral bandwidth are listed in the certificate of the reference material[1)]. All procedures shall be implemented in accordance with ISO/IEC 17025 and ISO Guide 34.

b) **Absorbance scale**: Secondary calibration standards can be used to check the ordinate accuracy. The set of standards consisting of gray glass filters[2)] provide nominal absorbance, A, values of 0,3A, 0,5A, and 1,0A respectively. The ordinate readings of the filters are measured at each of the selected wavelengths and the readings obtained compared with those listed in the certificate of the secondary calibration standards.

Alternatively, prepare a 200 mg/l solution of potassium chromate[3)] in 0,05 mol/l potassium hydroxide solution. Transfer 25 ml of this solution to a volumetric flask, capacity 500 ml, ISO 1042[1], class A, and make up to the mark with additional 0,05 mol/l potassium hydroxide solution. The absorbance of this solution, measured in a cell 10 mm thick at 275 nm against the 0,05 mol/l potassium hydroxide solution, should be 0,200 ± 0,005.

WARNING — Potassium chromate is very toxic, dangerous to the environment and a carcinogen by inhalation. Attention is drawn to regulations which specify handling and disposal procedures for toxic and dangerous substances. Users should be aware of and comply with technical, organizational, and personal safety measures.

6.2 Matched quartz cells, of pathlength 10 mm (1 cm), suitable for measurements at ultraviolet and visible wavelengths.

6.3 Volumetric flask, capacity 25 ml, ISO 1042[1], class A.

6.4 Analytical balance, capable of being read to the nearest 0,001 g.

1) Starna Scientific Ltd (www.starna.com) is an example of a supplier of suitable holmium filters and potassium dichromate sealed cell available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.

2) A suitable supplier is PerkinElmer Ltd. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the supplier named.

3) NIST 935a is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named.

7 Sampling

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 5555[2].

A representative sample should have been sent to the laboratory. It should not have been damaged or changed during transport or storage.

Ensure that the sample is taken and stored away from strong light, kept cold and contained in completely filled glass containers, hermetically sealed with ground glass or waxed cork stoppers.

8 Preparation of test sample

Prepare the test sample in accordance with ISO 661.

9 Procedure

9.1 General

Melt the sample at 60 °C to 70 °C and homogenize thoroughly before taking a test portion. Filter through a fast filter paper [Whatman No. 1[4)]] if the sample contains impurities and is not clear.

9.2 Test portion and preparation of the test solution

Weigh, to the nearest 0,1 mg, about 0,1 g to 0,5 g of the test sample (Clause 8), sufficient to obtain absorbance values between 0,2 and 0,8, into a 25 ml volumetric flask (6.3).

Dissolve the test portion in a few millilitres of isooctane (5.1) at ambient temperature and then make up to the mark with the same solvent. Mix thoroughly.

9.3 Determination

Rinse a quartz cell (6.2) three times with the test solution (9.2). Fill the cell with the test solution and fill a second matched cell with isooctane (5.1). Measure the absorbance of the test solution against the solvent by means of the spectrometer (6.1) at wavelengths 446 nm and 269 nm. If necessary, dilute the original test solution (9.2) to a measured volume and take further readings so that the observed absorptions are between (0,2 and 0,8) absorbance values.

10 Calculation

10.1 The deterioration of bleachability index (DOBI), I_{DoB}, is calculated as follows:

4) Example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product. Equivalent products may be used if they can be shown to lead to the same results.

$$I_{DoB} = \frac{A_{446}}{A_{269}}$$

where

A_{446} is the absorbance at 446 nm;

A_{269} is the absorbance at 269 nm.

Express the results to one decimal place.

10.2 Calculate the total carotene content, w_c, of oil as β-carotene, in milligrams per kilogram, using the following formula:

$$w_c = \frac{383\ \Delta A}{l\ \rho}$$

where

383 $= 10^6/2\ 610$, in which 2 610 is the percentage solution extinction coefficient of β-carotene in isooctane at 446 nm;

ΔA is the observed difference in absorption, A, between the sample solution and the solvent (5.1);

l is the pathlength, in centimetres, of the cell;

ρ is the concentration, in grams per 100 ml, used for absorption measurement.

Express the results to the nearest whole number.

11 Precision

11.1 Interlaboratory test

Details of an interlaboratory test on the precision of the method for the DOBI and carotene content are summarized in Annex A and Annex B respectively. It is possible that the values derived from these interlaboratory tests are not applicable to concentration ranges and matrices 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 values of r given in Table A.1 and Table B.1.

11.3 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment, will in not more than 5 % of cases exceed the values of R given in Table A.1 and Table B.1.

12 Test report

The test report shall contain at least the following information:

a) all information necessary for the complete identification of the sample;

b) the sampling method used, if known;

c) the test method used, with reference to this International Standard (ISO 17932:2011);

d) all operating details not specified in this International Standard, or regarded as optional, together with any details of any incident which may have influenced the test result;

e) the test result obtained;

f) if the repeatability has been checked, the final quoted result obtained.