
**Animal and vegetable fats and oils —
Determination of Lovibond® colour —
Automatic method**

*Corps gras d'origines animale et végétale — Détermination de la
couleur Lovibond® — Méthode automatique*

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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 27608 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 11, *Animal and vegetable fats and oils*.

Introduction

This International Standard has been developed at the request of the industry for an automatic method to measure the colour of oils and fats. The traditional scale used for this parameter is the Lovibond® scale, as used in the manual method of colour measurement, ISO 15305^[5]. It has proved impossible to get an automated instrument which gives results that exactly match those of the manual method. However, the repeatability and reproducibility limits of the manual method are quite large, and thus the automated method specified in this International Standard has been developed to include instruments which produce results within these limits.

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Animal and vegetable fats and oils — Determination of Lovibond® colour — Automatic method

1 Scope

This International Standard specifies a method for the determination of Lovibond®¹⁾ colour of animal and vegetable fats and oils using automatic instrumentation.

NOTE 1 The interlaboratory trials have shown that these instruments are not suitable for use with oils and fats with a colour intensity above 4 Lovibond® red units, for which the manual reference method, ISO 15305^[5], is applicable.

NOTE 2 The trials also showed that these instruments are not suitable for samples with strong blue, green, and brown hues, for which the manual reference method, ISO 15305^[5], is applicable.

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*

3 Terms and definitions

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

3.1

Lovibond® colour value

value obtained by analysis of light transmitted through a known path length of liquid fat or oil and read from the display of a suitable instrument

NOTE Lovibond® colour value is displayed in terms of Lovibond® colour units.

4 Principle

The colour of the light transmitted through a known path length of liquid fat or oil is measured by the spectral response of the light arriving at a detector. The results of these calculations are expressed in terms of Lovibond® colour units.

1) Lovibond is the trade name of a colour scale developed and supplied by The Tintometer Ltd (<http://www.tintometer.com>). This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

5 Apparatus

5.1 Automatic colorimeter. An instrument capable of measuring transmitted colour over the range 400 nm to 700 nm, reporting the result in terms of Lovibond® colour units, being equivalent to those obtained using the manual equipment according to ISO 15305^[5]. It should also be capable of giving results equivalent to ISO 15305^[5] when optical glass cells ranging from 1,58 mm to 133 mm (1/16 inch to 5 1/4 inch) optical path length are used.

The following instruments have been found to be suitable:

- a) LICO 200, LICO 300, LICO 400, and LICO 500²⁾;
- b) PFX 880/L, PFX 950, and PFX 995³⁾.

5.2 Certified reference standards. Certified glass or liquid reference standards with defined or traceable Lovibond® colour values and optical path length details are suitable and can be obtained from the manufacturers of the instruments [see footnotes 2) and 3)]. It is recommended that reference standards be used for calibration and routine verification of automatic colorimeters, and these may assist laboratories to conform to their good laboratory practice requirements and ISO/IEC 17025^[6] and ISO 9000^[4] procedures.

5.3 Optical glass sample cells, made of good quality optical glass and with a good standard finish. The following optical path lengths may be used, or results expressed as equivalent to these lengths:

1,6 mm (1/16 inch);	3,2 mm (1/8 inch);	6,4 mm (1/4 inch);	12,7 mm (1/2 inch);
25,4 mm (1 inch);	76,2 mm (3 inch);	133,4 mm (5 1/4 inch).	

Borosilicate optical glass cells should be used if cells are subjected to thermal shock, in particular during the heating or measurement of hot samples.

6 Sampling

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 5555^[1].

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

7 Preparation of test sample

7.1 General

Prepare the sample in accordance with ISO 661. The fat or oil shall be completely liquid, clear, bright, and free from turbidity when the determination is performed.

2) LICO 200, LICO 300, LICO 400, and LICO 500 are trade names of products supplied by Hach Lange (<http://www.hach-lange.com>). This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

3) PFX 880/L, PFX 950, and PFX 995 are trade names of products supplied by The Tintometer Ltd (<http://www.tintometer.com>). This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

7.2 Samples requiring heating

Solid samples, such as fats and tallow, require some form of heating. If the prepared sample is not fully liquid at room temperature, heat it to a temperature approximately 10 °C above its melting point.

IMPORTANT — The sample shall not be heated to the extent that it becomes scorched or discoloured.

Stirring the sample in a heated cell prior to measurement can help to disperse thermal currents or bubbles and establish a uniform temperature throughout the sample. The temperature at which the colour measurement is carried out should be recorded in the test report for all samples.

7.3 Filtration

If turbidity is present after heating, the sample shall be filtered to an appropriate level. It is, however, advised to filter all samples prior to testing as slight turbidities are difficult to see with the naked eye. The filtration process shall not affect the colour of the sample.

NOTE Suspended material and bubbles, even if of colloidal size, cause light scattering and can result in inaccurate measurement results.

8 Procedure

8.1 Position the automatic colorimeter on a stable surface and ensure the following:

- a) the unit is away from bright, direct sunlight;
- b) the ambient temperature is between 0 °C and 40 °C;
- c) a regulated power supply is available.

8.2 Turn on the colorimeter and allow for initialization to be completed (refer to manufacturer's instruction manual).

8.3 Select the Lovibond® colour scale from the menu options.

8.4 Select or input the required optical path length.

8.5 Perform zero (baseline) calibration according to manufacturer's instructions. This calibration should be performed at regular intervals depending on instrument usage.

8.6 Measure at least one certified glass or liquid reference standard (5.2) to verify the performance of the colorimeter prior to sample measurement. It is recommended that any glass reference filters be re-certified regularly and that the shelf lives of liquid reference standards be observed.

8.7 If an integrated heating system is available on the instrument, set the heater controller to the required temperature. This is generally 10 °C above the melting point of the fat. This allows a constant sample temperature for the duration of the measurement (refer to manufacturer's instruction manual).

8.8 The optical glass cell (5.3) shall be thoroughly clean and dry and, if necessary, pre-warmed to the same temperature as the sample so that no solid matter separates from the sample during the colour determination. If heat loss occurs, re-heat the sample and cell simultaneously.

8.9 Pour the liquid sample into the cell. It is recommended the cell be filled except for the top 3 mm. The optical path length of the cell used should be related to the colour saturation of the sample. Reduce the path length if required. It is advisable to restrict the colour intensity of the sample to a maximum of 4 Lovibond® red units for optimum colorimeter performance. Using a shorter path length cell, or equivalent, reduces colour saturation and increases measurement precision. Trade contracts, however, often prescribe fixed cell lengths.

8.10 Place the cell containing the sample in the sample chamber, position correctly, and close the sample chamber lid (refer to manufacturer's instruction manual).

8.11 Press the read button and obtain the Lovibond® colour values from the display (refer to manufacturer's instruction manual).

9 Expression of results

Express the results in terms of the following:

- a) the Lovibond® colour values displayed by the instrument;
- b) the optical path length of the cell to which the result refers.

Only standard cell sizes shall be referred to in the results (see ISO 15305^[5]).

10 Precision

10.1 Interlaboratory test

Details of an interlaboratory test on the precision of the method are given in Annex A. The values derived from this interlaboratory test may not be applicable to concentration ranges and matrices other than those given.

10.2 Repeatability

The absolute difference between two independent single test results, obtained with 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 Annex A.

10.3 Reproducibility

The absolute difference between two single test results, obtained with 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 reproducibility limit, R , given in Annex A.

11 Test report

The test report shall contain at least the following information:

- a) all information necessary for the complete identification of the sample;
- b) apparatus (brand, type, etc., and a statement that the model used is automatic);
- c) sample preparation details (heating or filtration, and any comments on sample condition before filtration);
- d) sample temperature during the measurement;
- e) the test method used, with reference to this International Standard (ISO 27608:2010);
- f) the optical path length of the cell, or its equivalent, used for measurement;
- g) the results obtained, in Lovibond® units.

Annex A (informative)

Results of an interlaboratory trial

An international collaborative trial involving 34 laboratories from nine countries analysing six samples of oils and fats was organized by DIN/DGF. The samples were analysed by three groups of laboratories:

- Group A using automatic instruments PFX 880/L, PFX 950 or PFX 995³⁾;
- Group B using automatic instruments LICO 200, LICO 300, LICO 400 or LICO 500²⁾;
- Group C using the manual instruments Model F or Model E⁴⁾ according to ISO 15305:1998⁵⁾.

The samples analysed were:

- Sample A distilled lauric fatty acid
- Sample B crude pressed rapeseed oil
- Sample C refined palm oil
- Sample D hardened palm oil and hardened rapeseed oil
- Sample E palm oil, rapeseed oil, and hardened rapeseed oil
- Sample F rapeseed oil and palm oil

The results obtained were subjected to statistical analysis in accordance with ISO 5725-1^[2] and ISO 5725-2^[3] to give the precision data reported in Tables A.1 and A.2.

NOTE Sample B was a crude, dark and rather cloudy sample, and various path lengths could have been used. This was probably the cause of the poor reproducibility results, but emphasizes the intensity limit of 4 Lovibond[®] red units included in this International Standard.

4) Tintometer Model F and Tintometer Model E are trade names of products supplied by The Tintometer Ltd (<http://www.tintometer.com>). This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

Table A.1 — Results of a collaborative trial

Group A — Instruments PFX 880/L, PFX 950 or PFX 995						
Sample	A		B		C	
Colour	Red	Yellow	Red	Yellow	Red	Yellow
Number of participating laboratories, N	9	9	5	5	10	10
Number of laboratories retained after eliminating outliers, n	7	8	5	5	10	9
Number of individual test results in all labs on each sample	14	16	10	10	20	18
Mean, μ	0,07	0,20	6,21	70,1	3,46	42,8
Repeatability standard deviation, s_r	0,00	0,09	0,51	0,0	0,07	0,0
Repeatability coefficient of variation, $C_{V,r}$, %	0,0	46,8	8,2	0,0	2,2	0,0
Repeatability limit, r ($s_r \times 2,8$)	0,00	0,26	1,42	0,0	0,21	0,0
Reproducibility standard deviation, s_R	0,05	0,18	1,81	0,1	0,89	10,8
Reproducibility coefficient of variation, $C_{V,R}$, %	68,3	89,8	29,1	0,2	25,8	25,3
Reproducibility limit, R ($s_R \times 2,8$)	0,14	0,50	5,06	0,4	2,49	30,3
Group B — Instruments LICO 200, LICO 300, LICO 400 or LICO 500						
Sample	A		B		C	
Colour	Red	Yellow	Red	Yellow	Red	Yellow
Number of participating laboratories, N	23	23	21	20	24	24
Number of laboratories retained after eliminating outliers, n	23	23	21	20	19	20
Number of individual test results in all labs on each sample	46	46	42	40	38	40
Mean, μ	0,11	0,22	12,7	89,4	2,75	36,6
Repeatability standard deviation, s_r	0,02	0,06	0,1	0,0	0,13	2,3
Repeatability coefficient of variation, $C_{V,r}$, %	18,5	26,0	1,0	0,0	4,6	6,2
Repeatability limit, r ($s_r \times 2,8$)	0,06	0,16	0,4	0,0	0,35	6,4
Reproducibility standard deviation, s_R	0,08	0,16	6,6	33,0	0,20	5,5
Reproducibility coefficient of variation, $C_{V,R}$, %	72,0	73,6	51,8	37,0	7,4	15,1
Reproducibility limit, R ($s_R \times 2,8$)	0,23	0,45	18,4	92,5	0,57	15,5
Group C — Manual instruments Model F (BS684) or Model E according to ISO 15305:1998 ^[5]						
Sample	A		B		C	
Colour	Red	Yellow	Red	Yellow	Red	Yellow
Number of participating laboratories, N	11	12	9	9	13	13
Number of laboratories retained after eliminating outliers, n	11	12	8	9	13	13
Number of individual test results in all labs on each sample	22	24	16	18	26	26
Mean, μ	0,13	0,32	8,46	59,5	2,90	29,9
Repeatability standard deviation, s_r	0,02	0,06	0,16	6,4	0,17	2,1
Repeatability coefficient of variation, $C_{V,r}$, %	16,2	18,2	1,9	10,7	5,9	6,9
Repeatability limit, r ($s_r \times 2,8$)	0,06	0,16	0,44	17,9	0,48	5,8
Reproducibility standard deviation, s_R	0,14	0,27	4,24	19,3	0,36	7,8
Reproducibility coefficient of variation, $C_{V,R}$, %	102,8	86,5	50,1	32,4	12,5	26,1
Reproducibility limit, R ($s_R \times 2,8$)	0,38	0,77	11,88	54,0	1,02	21,6

Table A.2 — Results of a collaborative trial

Group A — Instruments PFX 880/L, PFX 950 or PFX 995						
Sample	D		E		F	
Colour	Red	Yellow	Red	Yellow	Red	Yellow
Number of participating laboratories, N	10	10	10	10	10	10
Number of laboratories retained after eliminating outliers, n	10	10	10	10	10	9
Number of individual test results in all labs on each sample	20	20	20	20	20	18
Mean, μ	1,81	16,0	2,78	30,5	1,37	12,7
Repeatability standard deviation, s_r	0,00	0,2	0,08	2,1	0,06	0,3
Repeatability coefficient of variation, $C_{V,r}$, %	0,0	1,4	3,0	6,8	4,6	2,6
Repeatability limit, r ($s_r \times 2,8$)	0,00	0,6	0,23	5,8	0,18	0,9
Reproducibility standard deviation, s_R	0,37	2,9	0,53	6,7	0,36	2,0
Reproducibility coefficient of variation, $C_{V,R}$, %	20,6	17,9	18,9	21,9	26,2	15,5
Reproducibility limit, R ($s_R \times 2,8$)	1,04	8,0	1,47	18,7	1,00	5,5
Group B — Instruments LICO 200, LICO 300, LICO 400 or LICO 500						
Sample	D		E		F	
Colour	Red	Yellow	Red	Yellow	Red	Yellow
Number of participating laboratories, N	24	24	24	24	24	24
Number of laboratories retained after eliminating outliers, n	19	19	19	20	19	19
Number of individual test results in all labs on each sample	38	38	38	40	38	38
Mean, μ	1,49	14,8	2,39	28,2	1,18	12,7
Repeatability standard deviation, s_r	0,13	0,7	0,08	1,5	0,07	1,0
Repeatability coefficient of variation, $C_{V,r}$, %	8,7	4,5	3,3	5,5	6,1	7,7
Repeatability limit, r ($s_r \times 2,8$)	0,36	1,9	0,22	4,3	0,20	2,8
Reproducibility standard deviation, s_R	0,18	1,9	0,20	4,1	0,19	1,5
Reproducibility coefficient of variation, $C_{V,R}$, %	12,3	12,9	8,5	14,6	15,8	12,1
Reproducibility limit, R ($s_R \times 2,8$)	0,51	5,4	0,57	11,5	0,52	4,3
Group C — Manual instruments Model F or Model E according to ISO 15305:1998 ^[5]						
Sample	D		E		F	
Colour	Red	Yellow	Red	Yellow	Red	Yellow
Number of participating laboratories, N	13	13	13	13	13	13
Number of laboratories retained after eliminating outliers, n	12	10	12	12	13	11
Number of individual test results in all labs on each sample	24	20	24	24	26	22
Mean, μ	1,63	14,9	2,45	25,6	1,17	11,9
Repeatability standard deviation, s_r	0,04	0,4	0,12	0,8	0,08	0,8
Repeatability coefficient of variation, $C_{V,r}$, %	2,5	2,6	4,7	3,0	6,5	6,4
Repeatability limit, r ($s_r \times 2,8$)	0,11	1,1	0,32	2,2	0,21	2,1
Reproducibility standard deviation, s_R	0,22	1,0	0,30	5,6	0,34	1,3
Reproducibility coefficient of variation, $C_{V,R}$, %	13,8	6,8	12,2	22,0	28,7	11,2
Reproducibility limit, R ($s_R \times 2,8$)	0,63	2,8	0,84	15,7	0,94	3,7