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Wheat flour and durum wheat semolina — Determination of colour by diffuse reflectance colorimetry

Farine de blé tendre et semoule de blé dur — Détermination de la couleur par colorimétrie de réflectance diffuse

Reference number
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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

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Foreword

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This document was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 4, *Cereals and pulses*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 338, *Cereal and cereal products*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Wheat flour and durum wheat semolina — Determination of colour by diffuse reflectance colorimetry

1 Scope

This document specifies a method for the determination of colour in durum wheat semolina and wheat flour by diffuse reflectance colorimetry.

It is applicable to industrial semolina and flour.

The method can be applicable to flour obtained from experimental mill.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
colour metric space
expression of the colour of an object or of a light source by some parameters expressed by figures

3.2
illuminant
light source characterized by a spectral curve, where the energy relative distribution is defined in the field of wavelengths that are able to influence the object colour vision

4 Principle

The principle is based on the measurement of colour directly on semolina and flour by a reflectance colorimeter.

The colour of wheat milling product (semolina and flour) is due to the pigments naturally present in wheat grains. These pigments (xanthophyll's and carotenoids) are responsible for the colour visually perceived in milling products.

5 Apparatus

5.1 Reflectance colorimeter[1] with head of measurement suitable for carrying out measurements of absolute chromaticity. Consists of a setting system and a cell samples-driver.

The colorimeter shall be characterized by the following technical characteristics:

— system of measurement with pulsed xenon lamp diffused to the light and receipt of the radiation reflected to 0° (geometry d/0°);

— circular surface of measurement;

— measurements of chromaticity expressed as L^*, a^*, b^* (see CIE 1976) with the use of the illuminant CIE D_{65} (illuminant D_{65}: representing one of the relative spectral distribution of the day-light energy that corresponds to a proximal colour temperature similar to 6 504 K);

— time of measurement 1 *s*;

— possibility of calibration with reference plate;

— repeatability within a DE * 0,6 (30 measurements effected to an interval of 10 s on the reference plate).

5.2 Accessory samples-driver and cell for the measurement of granular materials.

The dimensions of the cell that defines the quantity of sample submitted to the test are: external diameter = 60 mm, diameter inside hole = 22 mm, thickness = 9 mm.

5.3 Reference plate in porcelain for the initial setting of the colorimeter.

6 Sample preparation

Before the analysis, the samples shall be carefully homogenized.

Sampling is not part of the method specified in this document. A recommended sampling method is given in ISO 24333.

7 Procedure

7.1 General

Before each series of measurements, the apparatus shall be calibrated.

The colorimeter calibration shall be made through opaque stable materials (as ceramics, glaze etc.) samples supplied by manufacturers. When the colorimeter (5.1) is used, a further calibration for better measurement accuracy can be performed using a reference material similar to the colour of the samples to be measured.

Before the calibration, verify the integrity of the reference plate (5.3) used as a reference (e.g. for the absence of linings or colour not homogeneous). Also, for the setting, verify that the coordinates are those reported on the reference standard.

1) Laboratories involved in the ring test nearly all used a colorimeter CR 400 or CR 410 Minolta. These are fit apparatus responding to the required technical characteristics. Minolta is a trade name and is an example of a suitable apparatus 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.

7.2 Setting of the colorimeter

To set the colorimeter (5.1), put the head of measurement on the centre of the reference plate (5.3) and select the colour metric space y, x, z with illuminant D_{65} in the system CIE Lab.

At the end, return in the colour metric space *L**, *a**, *b**, and therefore prepare the colorimeter to the reading of the samples.

The reference plate shall carefully be cleaned after every setting, being careful to avoid any rubbing or lining. After the setting, the plate shall be stored in the dark.

7.3 Colorimetric determination

From a homogeneous sample of at least 100 g, withdraw a share, fill with care the cell for granular samples and measure. Every determination shall be performed twice.

The cell has to be emptied completely and cleared between two determinations. The sample shall be mixed each time.

8 Expression of the results

The colour metric space CIELAB (1976) is the most used for measuring the colour of objects.

*L** indicates the lightness and extends from 0 (black) to 100 (white); *a** and *b** are chromaticity indexes, respectively; *a**: red/green (a > 0 red; a < 0 green); b*: yellow/blue (a > 0 yellow; a < 0 blue).

The results are expressed as yellow index (*b**). It is possible to have a datum of dark coloration through the measure "100-L*" and of red coloration through the measure "*a**."

The results are expressed with two decimal places.

9 Precision

9.1 General

The values derived for the interlaboratory tests, given in Annex A, may not be applicable to concentration ranges and matrices other than those given, i.e. for *L** between 83,37 and 92,47; *a**: −2,17 and −0,18; *b**: 8,56 and 27,54.

9.2 Repeatability limit, *r*

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 be greater than the repeatability limit *r* given in Table 1.

Table 1 — Repeatability limit

Parameter	*L**	*a**	*b**
Repeatability standard deviation, s_r	−0,007 9*L** + 0,768 8	0,03	0,010 0*b** − 0,018 6
Repeatability limit, *r*	− 0,0219 *L** + 2,129 6	2,77 × 0,03 = 0,08	0,027 7*b** − 0,051 5

9.3 Reproducibility limit, *R*

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 be greater than the reproducibility limit *R* given in Table 2.

Table 2 — Reproducibility limit

Parameter	L^*	a^*	b^*
Reproducibility standard deviation, s_R	$-0{,}033\,6L^* + 4{,}680\,8$	0,63	$0{,}038\,2b^* + 0{,}057\,1$
Reproducibility limit, R	$-0{,}093\,1L^* + 12{,}965\,8$	2,77 × 0,63 = 1,75	$0{,}105\,8b^* + 0{,}158\,2$

9.4 Critical difference, d_C

9.4.1 General

Critical difference is the difference between two averaged values obtained from two test results under repeatability conditions.

9.4.2 Comparison of two groups of measurements in one laboratory

The critical difference ($d_{C,r}$) between two averaged values obtained in one laboratory from two test results under repeatability conditions is equal to Formula (1):

$$d_{C,r} = 2{,}77 s_r \sqrt{\frac{1}{2n_1} + \frac{1}{2n_2}} = 2{,}77 s_r \sqrt{\frac{1}{2}} = 1{,}98 s_r \quad (1)$$

where

s_r is the standard deviation of repeatability;

n_1 and n_2 are the number of test results corresponding to each of the averaged values ($n_1 = n_2 = 2$).

9.4.3 Comparison of two groups of measurements in two laboratories

The critical difference ($d_{C,R}$) between two averaged values obtained in two different laboratories from two test results under repeatability conditions is equal to Formula (2):

$$d_{C,R} = 2{,}77 \sqrt{s_R^2 - s_r^2 \left(1 - \frac{1}{2n_1} - \frac{1}{2n_2}\right)} = 2{,}77 \sqrt{s_R^2 - 0{,}5 s_r^2} \quad (2)$$

where

s_r is the standard deviation of repeatability;

s_R is the standard deviation of reproducibility;

n_1 and n_2 are the number of test results corresponding to each of the averaged values ($n_1 = n_2 = 2$).

9.5 Uncertainty, U

The measurement uncertainty, U, is a parameter representing the distribution of the values that may reasonably be attributed to the result.

It is possible to evaluate measurement uncertainties using data obtained from studies carried out in accordance with ISO 5725-2. The reproducibility standard deviation obtained during an interlaboratory test is a valid basis to evaluate measurement uncertainty because, by definition, uncertainty characterizes the dispersion of values that can be reasonably attributed to the parameter. The calculated expanded standard uncertainty shall be plus or minus twice the standard deviations of reproducibility given in this document.

10 Test report

The test report shall contain at least the following information:

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

b) the method used, with reference to this document, i.e. ISO 16624;

c) the final result obtained;

d) all operational details not specified in this document, or considered optional, as well as any incident that could have influenced the test(s) result(s);

e) the data of the test.

Annex A
(informative)

Results of the interlaboratory test

The values for repeatability limit and reproducibility limit have been derived from the results of interlaboratory test carried out in accordance with ISO 5725-1, ISO 5725-2 and ISO 5725-6.

An international interlaboratory test was organized by the CRA-Cereal Quality Research Unit (CRA-QCE), Italy.

This international interlaboratory test was carried out on four industrial samples of durum wheat semolina (FD) and four industrial samples of common wheat flour (FT).

Twenty-seven laboratories took part in this test and all of them sent their results. Specifically, the test was attended by 15 Italian laboratories, 5 French laboratories, 5 Spanish laboratories, 2 Portugal laboratories and 1 Philippine laboratory.

Each laboratory analysed each sample five times in duplicate. The processing of the data showed that the accuracy of the results is the same when comparing the average of the ten or the two first measurements. The statistic results presented were therefore determined from the analysis of each sample in duplicate.

The results are given in Tables A.1, A.2 and A.3 and Figures A.1, A.2 and A.3.

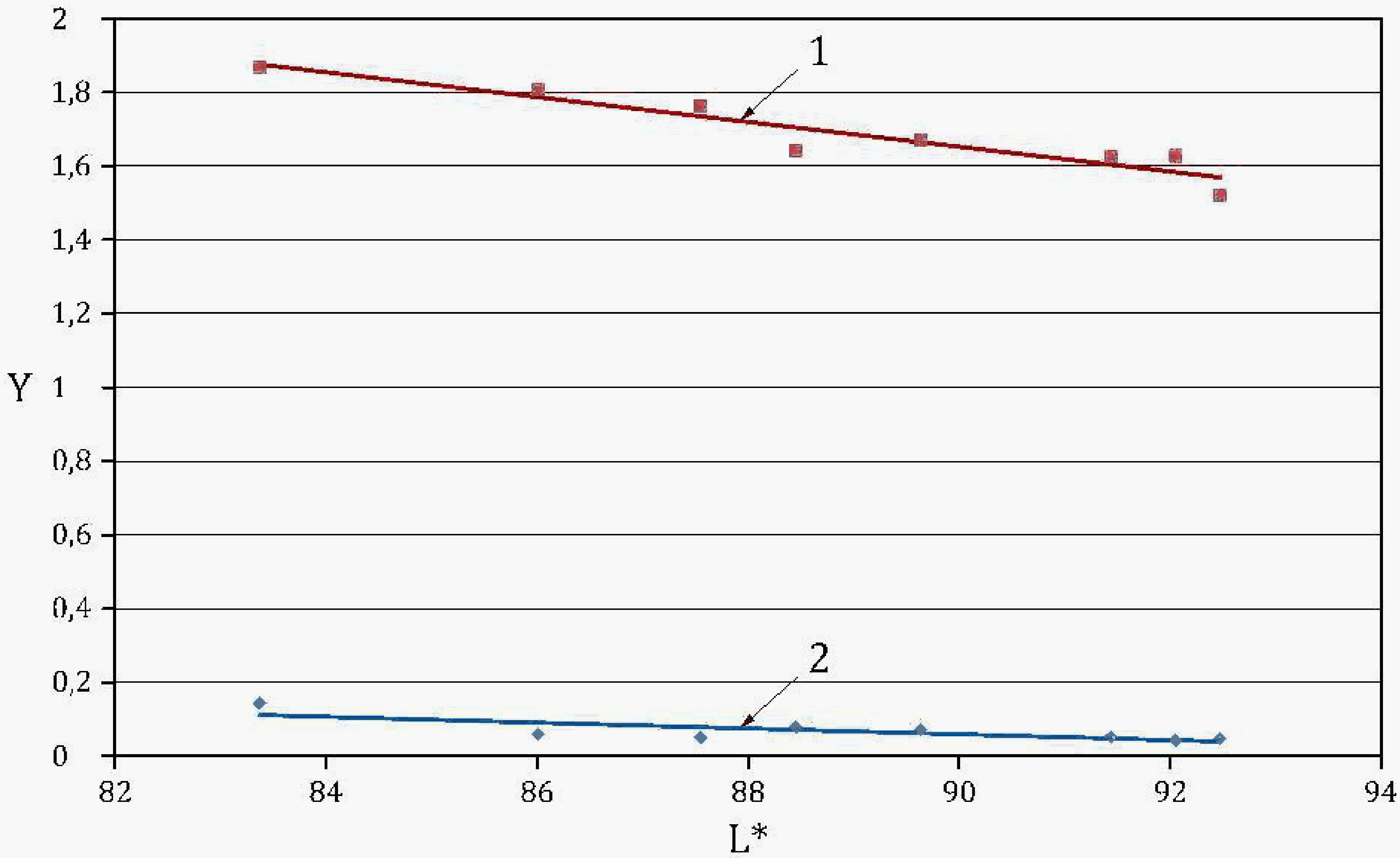
Table A.1 — Statistical results of the interlaboratory test for L^*

L^*	FD4	FD3	FD2	FT1	FD1	FT3	FT4	FT2
Number of laboratories after outlier elimination	20	25	24	26	26	25	22	23
Global mean	83,37	86,01	87,55	88,45	89,63	91,44	92,05	92,47
Repeatability standard deviation, s_r	0,14	0,06	0,05	0,08	0,07	0,05	0,04	0,05
Repeatability coefficient of variation, $C_{V,r}$, in %	0,17	0,07	0,06	0,09	0,08	0,06	0,05	0,05
Limit of repeatability, $r = (2,77 \times s_r)$	0,39	0,16	0,14	0,21	0,19	0,15	0,12	0,13
Reproducibility standard deviation, s_R	1,87	1,81	1,76	1,64	1,67	1,62	1,63	1,52
Reproducibility coefficient of variation, $C_{V,R}$, in %	2,24	2,10	2,02	1,86	1,86	1,78	1,77	1,65
Limit of reproducibility, $R = (2,77 \times s_R)$	5,18	5,01	4,89	4,55	4,63	4,50	4,51	4,22

Key

FD: industrial samples of durum wheat semolina

FT: industrial samples of common wheat flour



Key

1 reproducibility standard deviation, s_R = −0,033 6x + 4,680 8R^2 = 0,8904

2 repeatability standard deviation, s_r = −0,007 9x + 0,768 8 R^2 = 0,5972

Figure A.1 — Relationship between precision standard deviations and *L**

Figure A.1 shows that repeatability and reproducibility standard deviations are dependent of the average value *L**.

Reproducibility limit, $R = 2{,}77 \times (-0{,}033\,6L^* + 4{,}680\,8) = -0{,}093\,1L^* + 12{,}965\,8$

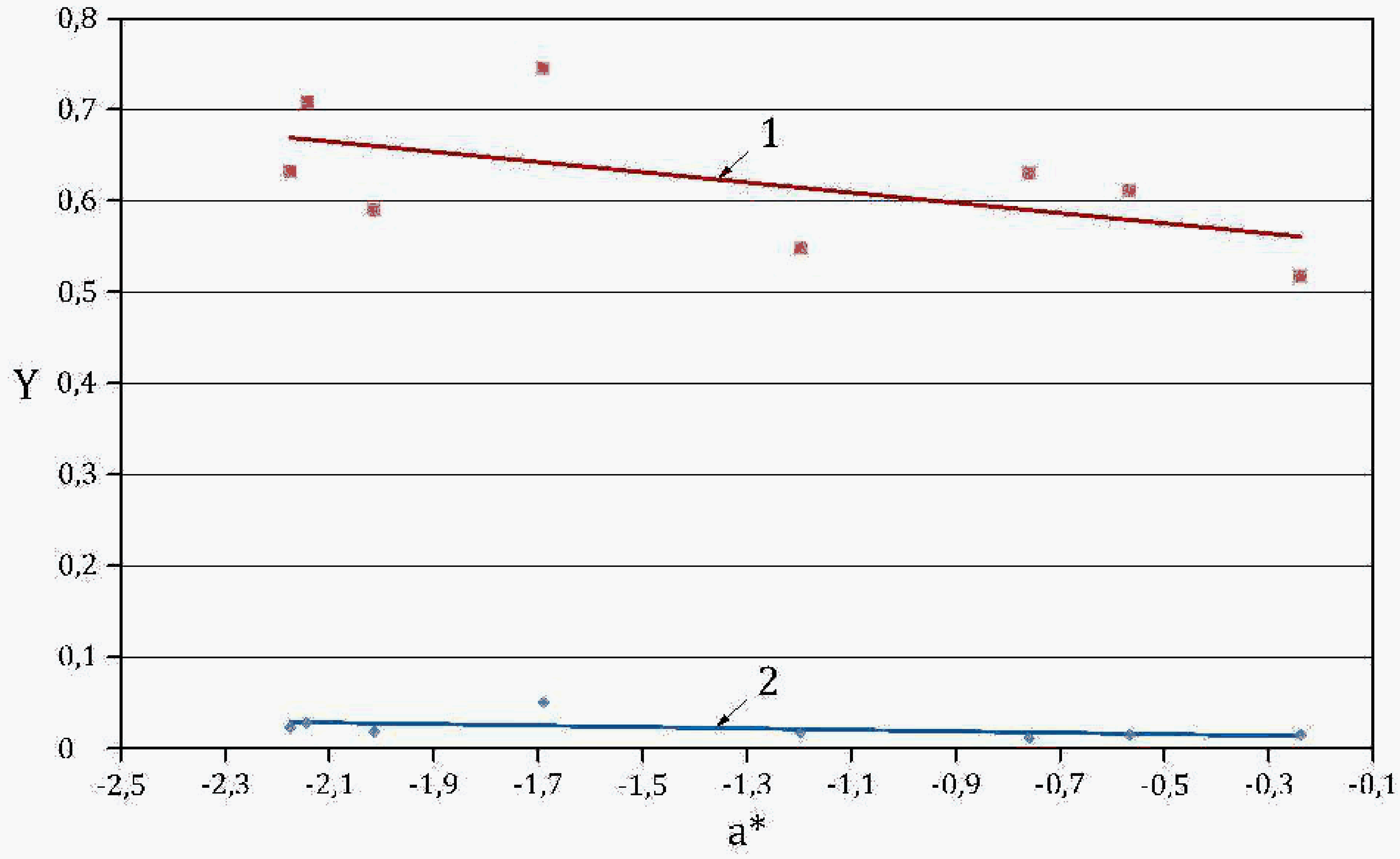
Repeatability limit, $r = 2{,}77 \times (-0{,}007\,9L^* + 0{,}768\,8) = -0{,}021\,9L^* + 2{,}129\,6$

Table A.2 — Statistical results of the interlaboratory test for *a**

*a**	FD2	FD3	FD1	FD4	FT2	FT4	FT3	FT1
Number of laboratories after outlier elimination	24	25	22	23	25	25	26	25
Global mean	−2,17	−2,13	−2,00	−1,67	−1,16	−0,71	−0,52	−0,18
Repeatability standard deviation, s_r	0,02	0,03	0,02	0,05	0,02	0,01	0,02	0,02
Repeatability coefficient of variation, $C_{V,r}$, in %	−1,05	−1,30	−0,93	−3,02	−1,47	−1,60	−2,93	−8,56
Limit of repeatability, $r = (2{,}77 \times s_r)$	0,06	0,08	0,05	0,14	0,05	0,03	0,04	0,04
Reproducibility standard deviation, s_R	0,63	0,71	0,59	0,75	0,55	0,63	0,61	0,52
Key FD: industrial samples of durum wheat semolina FT: industrial samples of common wheat flour								

Table A.2 *(continued)*

a^*	FD2	FD3	FD1	FD4	FT2	FT4	FT3	FT1
Reproducibility coefficient of variation, $C_{V,R}$, in %	−29,24	−33,22	−29,54	−44,75	−47,17	−88,76	−118,70	−290,44
Limit of reproducibility, $R = (2{,}77 \times s_R)$	1,76	1,96	1,64	2,07	1,52	1,75	1,69	1,44
Key FD: industrial samples of durum wheat semolina FT: industrial samples of common wheat flour								



Key

1 reproducibility standard deviation, $s_R = -0{,}054\,6x + 0{,}551\,7$ $R^2 = 0{,}318\,3$

2 repeatability standard deviation, $s_r = -0{,}007\,7x + 0{,}012\,1$ $R^2 = 0{,}238\,5$

Figure A.2 — Relationship between precision standard deviations and mean values and a^*

Figure A.2 shows that repeatability and reproducibility standard deviations are independent of the average value a^*.

Repeatability standard deviation, $s_r = 0{,}03$

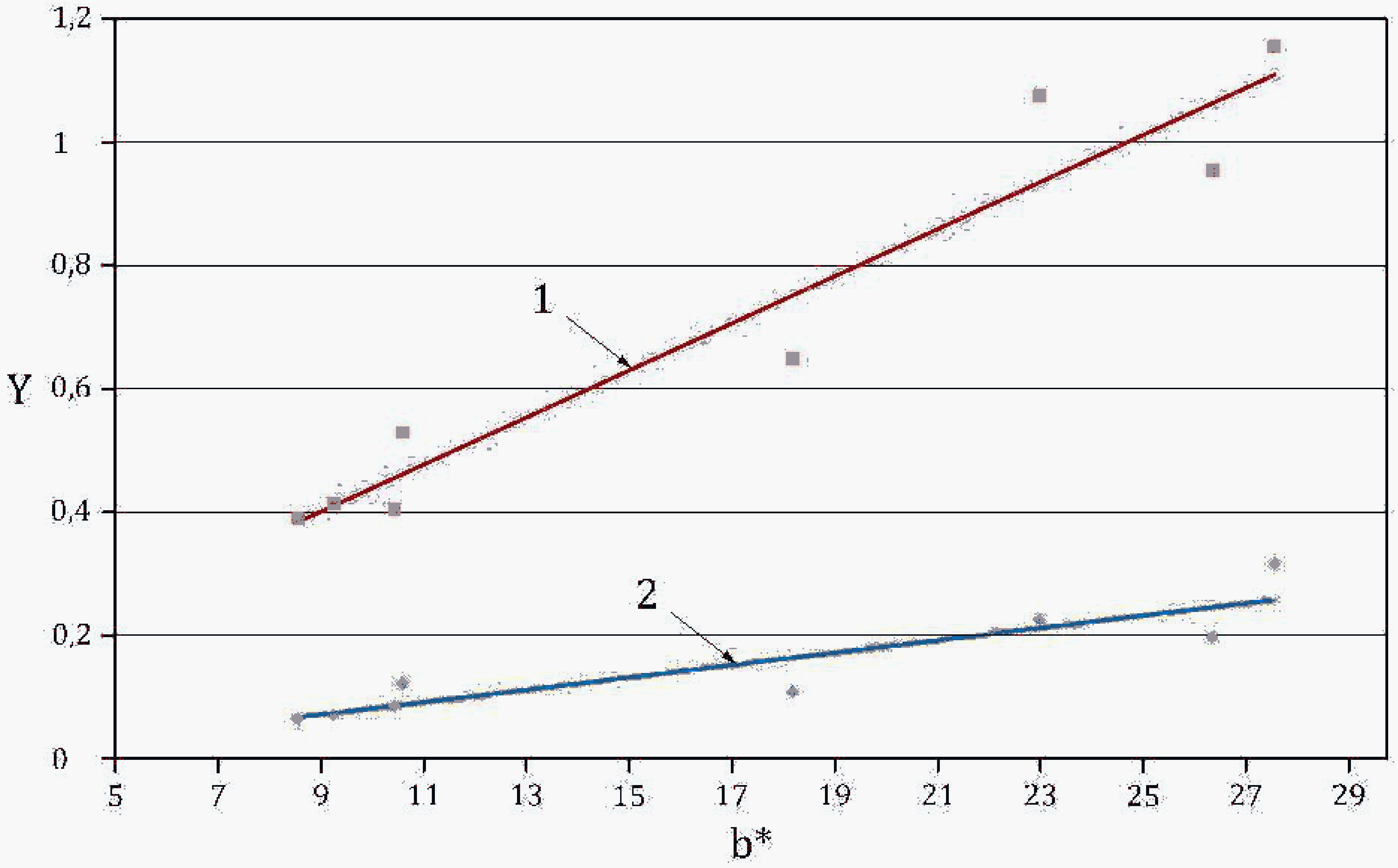
Reproducibility standard deviation, $S_R = 0{,}63$

Repeatability limit, $r = 2{,}77 \times s_r = 0{,}08$

Reproducibility limit, $R = 1{,}75$

Table A.3 — Statistical results of the interlaboratory for *b**

*b**	FT4	FT3	FT1	FT2	FD1	FD2	FD3	FD4
Number of laboratories after outlier elimination	25	25	25	26	26	26	23	20
Global mean	8,56	9,25	10,43	10,59	18,17	22,98	26,33	27,54
Repeatability standard deviation, s_r	0,07	0,07	0,09	0,12	0,11	0,23	0,20	0,32
Repeatability coefficient of variation, $C_{V,r}$, in %	0,76	0,77	0,82	1,17	0,60	0,98	0,75	1,15
Limit of repeatability, $r = (2{,}77 \times s_r)$	0,18	0,20	0,24	0,34	0,30	0,63	0,55	0,88
Reproducibility standard deviation, s_R	0,39	0,41	0,40	0,53	0,65	1,07	0,95	1,15
Reproducibility coefficient of variation, $C_{V,R}$, in %	4,57	4,48	3,87	5,00	3,57	4,68	3,63	4,19
Limit of reproducibility, $R = (2{,}77 \times s_R)$	1,08	1,15	1,12	1,47	1,80	2,98	2,65	3,20
Key FD: industrial samples of durum wheat semolina FT: industrial samples of common wheat flour								



Key

1 reproducibility standard deviation, $s_R = 0{,}038\,2x + 0{,}057\,1$ $R^2 = 0{,}927\,9$

2 repeatability standard deviation, $s_r = 0{,}010\,0x - 0{,}018\,6$ $R^2 = 0{,}815\,3$

Figure A.3 — Relationship between precision standard deviations and mean values and *b**

Figure A.3 shows that repeatability and reproducibility standard deviations are dependent of the average value *b**.

Reproducibility limit, $R = 2{,}77 \times (0{,}038\,2b^* + 0{,}057\,1) = 0{,}105\,8b^* + 0{,}158\,2$

Repeatability limit, $r = 2{,}77 \times (0{,}010\,0b^* - 0{,}018\,6) = 0{,}027\,7b^* - 0{,}051\,5$

The conversion of precision data into formulae for the calculation of repeatability, reproducibility and critical difference are given in Tables A.4 and A.5.

Table A.4 — L^*

L^*	s_r	r	s_R	R	$d_{C,r}$	$d_{C,R}$
83,37	0,11	0,31	1,88	5,21	0,22	5,19
84,00	0,11	0,29	1,86	5,15	0,21	5,13
84,50	0,10	0,28	1,84	5,10	0,20	5,09
85,00	0,10	0,27	1,82	5,05	0,19	5,04
85,50	0,09	0,26	1,81	5,01	0,18	5,00
86,00	0,09	0,25	1,79	4,96	0,18	4,95
86,50	0,09	0,24	1,77	4,92	0,17	4,90
87,00	0,08	0,23	1,76	4,87	0,16	4,86
87,50	0,08	0,21	1,74	4,82	0,15	4,81
88,00	0,07	0,20	1,72	4,78	0,15	4,77
88,50	0,07	0,19	1,71	4,73	0,14	4,72
89,00	0,07	0,18	1,69	4,68	0,13	4,68
89,50	0,06	0,17	1,67	4,64	0,12	4,63
90,00	0,06	0,16	1,66	4,59	0,11	4,58
90,50	0,05	0,15	1,64	4,54	0,11	4,54
91,00	0,05	0,14	1,62	4,50	0,10	4,49
91,50	0,05	0,13	1,61	4,45	0,09	4,45
92,00	0,04	0,12	1,59	4,40	0,08	4,40
92,47	0,04	0,11	1,57	4,36	0,08	4,36

Table A.5 — b^*

b^*	s_r	r	s_R	R	$d_{C,r}$	$d_{C,R}$
8,56	0,07	0,19	0,38	1,06	0,13	1,06
9,00	0,07	0,20	0,40	1,11	0,14	1,10
10,00	0,08	0,23	0,44	1,22	0,16	1,21
11,00	0,09	0,25	0,48	1,32	0,18	1,31
12,00	0,10	0,28	0,52	1,43	0,20	1,41
13,00	0,11	0,31	0,55	1,53	0,22	1,52
14,00	0,12	0,34	0,59	1,64	0,24	1,62
15,00	0,13	0,36	0,63	1,75	0,26	1,72
16,00	0,14	0,39	0,67	1,85	0,28	1,82
17,00	0,15	0,42	0,71	1,96	0,30	1,93
18,00	0,16	0,45	0,74	2,06	0,32	2,03
19,00	0,17	0,47	0,78	2,17	0,34	2,13
20,00	0,18	0,50	0,82	2,27	0,36	2,23
21,00	0,19	0,53	0,86	2,38	0,38	2,33
22,00	0,20	0,56	0,90	2,49	0,40	2,43
23,00	0,21	0,59	0,94	2,59	0,42	2,53

Table A.5 *(continued)*

b^*	s_r	r	s_R	R	$d_{C,r}$	$d_{C,R}$
24,00	0,22	0,61	0,97	2,70	0,44	2,63
25,00	0,23	0,64	1,01	2,80	0,46	2,73
26,00	0,24	0,67	1,05	2,91	0,48	2,83
27,00	0,25	0,70	1,09	3,02	0,50	2,93
27,54	0,26	0,71	1,11	3,07	0,51	2,98

Bibliography

[1] ISO 5725-2, *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*

[2] ISO 5725-3, *Accuracy (trueness and precision) of measurement methods and results — Part 3: Intermediate measures of the precision of a standard measurement method*

[3] ISO 5725-6, *Accuracy (trueness and precision) of measurement methods and results — Part 6: Use in practice of accuracy values*

[4] ISO 24333, *Cereals and cereal products — Sampling*

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