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Tehnično poročilo o medlaboratorijski študiji za ugotavljanje primesi v pšenici, rži in pšenici durum

Technical report of the interlaboratory study for the determination of Besatz in common wheat, rye and durum wheat

Technischer Bericht über Ringversuchsergebnisse zur Bestimmung von Besatz in Weizen, Roggen und Hartweizen

Rapport technique relatif à l'essai interlaboratoire portant sur la détermination du pourcentage d'impuretés dans le blé tendre, le seigle et le blé dur

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Technical report of the interlaboratory study for the determination of Besatz in common wheat, rye and durum wheat

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Technischer Bericht des Ringversuchs zur Bestimmung von Besatz in Weizen, Roggen und Hartweizen

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Foreword

This document (CEN/TR 16324:2012) has been prepared by Technical Committee CEN/TC 338 “Cereal and cereal products”, the secretariat of which is held by AFNOR.

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Introduction

The term “Besatz” applies to all components of a grain sample that differ from the normal basic cereal. The principle of the determination of Besatz is to separate all the groups of Besatz from the normal basic cereal grains of unimpaired quality by sieving and manual selection out of a subsample and to quantify them. The amount of Besatz and its constituent groups is important for health, cleaning, milling and further processing aspects. For these reasons Besatz is a part of contracts in grain trade and also of the grain intervention system of the EU. At present a European Standard for the determination of Besatz (EN 15587) has been developed.

An international interlaboratory test for the determination of Besatz, according to EN 15587 and involving 15 laboratories in 11 countries, was carried out with two common wheat, two rye and two durum wheat samples. Statistical analysis was possible for all Besatz fractions with the exception of grains overheated during drying, bunted grains, and impurities of animal origin.

Very high interlaboratory variation was found for the fractions mottled grains (including grains in which the germ is discoloured), unsound grains, and grains damaged by pests.

The lowest interlaboratory variation was found for the fractions extraneous matter, extraneous seeds, and ergot.

The coefficient of variability in most cases depended clearly on the amount of the respective Besatz group, whereby the amount of the respective Besatz group was in a normal range. As a result the coefficient of intralaboratory variability for the total Besatz was not higher than 10 %. The coefficient of interlaboratory variability for the total Besatz was not higher than 20 %, provided the amount of mottled grains or unsound grains or insect-damaged grain is not higher than 1 % (w/w).

1 Scope

The term "Besatz" applies to all components of a grain sample that differ from the normal basic cereal. It includes the following groups: broken grains, shrivelled grains, other cereals, grains damaged by pests, grains with discoloured germ, grains overheated during drying, sprouted grains, extraneous seeds, unsound grains, ergot, bunted grains, extraneous matter, husks and impurities of animal origin. The amount of Besatz and its constituent groups is important for health, cleaning, milling and further processing aspects. For these reasons Besatz is a component of contracts in grain trade and also of the grain intervention system of EU. The principle of the determination of Besatz is to separate all the groups of Besatz from the normal basic cereal grains of unimpaired quality by sieving and manual selection out of a subsample and to quantify them. There are various problems in the determination of Besatz: Firstly, the identification of the different groups of Besatz depends strongly on the experience and the knowledge of the investigator. Also experienced investigators can differ in their characterization of grains. Finally, one is faced with the fact that grain, even after mixing, is rarely homogenous. In other words, if a sample was divided by a sample divider into a number of portions, the amount of a specific group of Besatz in each portion could be different, even if absolutely no human or machine error occurred in each determination. These problems will result in variation of the results of the determination.

An international interlaboratory trial for the determination of Besatz in common wheat, durum wheat and rye was accomplished with 15 laboratories in order to get information on the intra- and interlaboratory variability of the determination of Besatz.

The Technical Report here describes the preparation and evaluation of the results of this interlaboratory test.

2 Design of the study

Each laboratory received two kinds of sample sets.

The **sample set A** consisted of six samples which had a mass of not less than 50g. The participants received two samples of common wheat, rye and durum wheat. The sample set A was prepared from basic cereal of unimpaired quality. Certain amounts of the 14 different Besatz groups [1)] were added to each sample of the set A, in the way that the two samples of the same cereal had the same composition. The composition of the spiked samples was chosen to ensure that i) laboratories obtaining outlying results would be identified and ii) the influence of sample division on the precision of the method would be recognizable.

1) The added material of the various Besatz groups comes from numerous Besatz-investigations of our own laboratory. The material was reviewed independently by three persons with regard to the unique assignment to a Besatz group.

The **sample set B** consisted of six samples which had a mass of not less than 1kg. The participants received two samples of common wheat, rye and durum wheat, resp.. The sample set B was prepared from clean basic cereal, which contained small amounts of Besatz [2)]. One bulk sample of slightly more than 40kg of each cereal (wheat, rye, and durum) was used. After careful mixing to make the bulk sample as homogeneous as possible it was divided using a sample divider to obtain 40 samples. Twenty of them were directly sealed in moisture proof plastic bags. The other 20 samples were spiked with certain Besatz groups, individually mixed and the sealed in moisture proof plastic bags. In this way we achieved different concentration levels of the various Besatz groups. The concentration levels were selected to represent the range of Besatz commonly found in grain samples. The results obtained for the sample set B were used for the calculation of the precision data given in Annex D of EN 15587:2008.

The participants were instructed to analyse the samples of set A only once and the samples of set B in duplicate. They had to divide the samples of set B to obtain the required size, before testing.

We sent the sample to 20 laboratories and after six weeks we received results of 15 laboratories for each sample.

The laboratories were instructed to report the weights of the analysed samples as well as of the separated Besatz groups. In addition, participants should calculate the results in terms of percentage weight. The obtained results of sample set B showed that the method of calculation was not described clearly enough in the draft standard. Many laboratories have not taken into account that extraneous matter was removed (by sieving) before testing the two sub-samples. Only five laboratories performed the calculations in the correct manner. The results of the other laboratories were recalculated before beginning the statistical evaluation.

3 Statistical evaluation

The statistical evaluation was performed according to ISO 5725-2. This requires the examination of data with the Cochran and Mandel's k test for non homogeneity of variances (5 % and 1 % level) and the examination of the data with the Grubbs and Mandel's h test for deviations of laboratory mean values (5 % and 1 % level). So, the analysis of the results for consistency and outliers was done by:

— numerical outlier tests (Cochran's test, Grubbs' test); and

— graphical consistency technique (Mandel's statistics, *k* and *h*).

The results obtained for the sample set B are presented in Clause 4 to 17. These results were used for the calculation of the precision data given in EN 15587. The single results of the duplicate determination and the calculated precision data (after outlier elimination) are always presented in a table and a figure. The results of the Mandel's statistic are presented in a separate figure. Abnormal values can be identified by the comparison with the critical values which are drawn as lines in the figure.

A value which was straggling (abnormal) according to one of these four statistical tests was removed only if the following two conditions were met:

a) the precision data without this abnormal values differed significantly from the precision data including it, so that its elimination gave significantly better values for precision;

b) the elimination of this abnormal value does not have the consequence that the ratio r / R (repeatability/Reproducibility) reaches the value 1.

2) The use of basic cereal of unimpaired quality was impossible, because the cleaning of 120 kg of grain material by manual selection was not realisable.

Only if both conditions were met, an abnormal value was eliminated. Therefore, there may be cases in which an abnormal value was not removed, because the precision data were not significantly improved or the ratio r/R reached the value 1.

The table which contains the collation of the test results and the calculated precision data shows also the eliminated outliers, which are underlined.

The results obtained for the sample set A were statistically evaluated in the same manner as described above, but the collation of test results and precision data are not presented as detailed as above. The precision data of the sample set A are presented in Clause 18 and shown together with those of the sample set B in Clause 19.

No statistical evaluation could be done for three fractions: grains overheated during drying, smutty grains and impurities of animal origin. In the first two cases we did not have enough material to spike the grain samples. In the case of impurities of animal origin there was a problem with the insufficient description in EN 15587. It was not clear how to handle the insects which fall through the 1 mm sieve. Some laboratories had counted these insects, others had not.

4 Results of the determination of "broken grains"

Table 1 — Collation of test results, outliers and precision data on the determination of "broken grains"

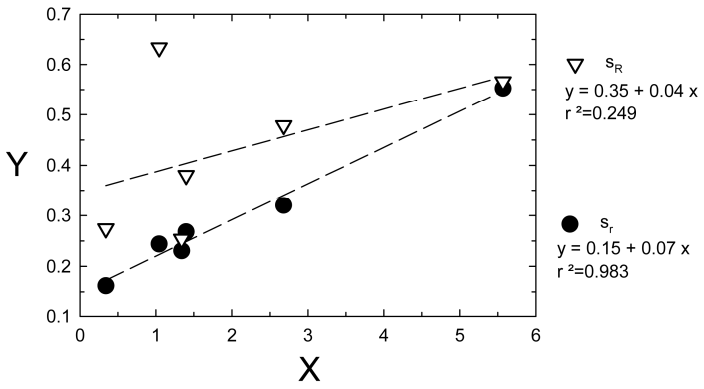
Determination of "broken grains"

	Single results of the dupliacte determination (results given in [w/w%])												Mean of duplicate determination (results given in [w/w%])					
Sample:	A	A	B	B	C	C	D	D	E	E	F	F	A	B	C	D	E	F
Lab.No																		
1	1.676	1.517	2.935	2.498	1.202	1.269	5.512	5.639	0.120	0.630	2.158	1.690	1.60	2.72	1.24	5.58	0.38	1.92
2	1.917	1.616	3.813	2.932	1.133	1.370	5.500	6.042	0.222	0.019	1.519	1.488	1.77	3.37	1.25	5.77	0.12	1.50
3	1.985	1.446	2.858	2.538	1.430	1.727	6.278	6.512	0.319	0.788	1.626	2.006	1.72	2.70	1.58	6.40	0.55	1.82
4	0.265	0.732	2.270	2.365	1.235	1.614	6.332	4.915	0.561	0.503	1.673	1.144	0.50	2.32	1.42	5.62	0.53	1.41
5	0.363	0.336	2.574	2.301	1.025	1.023	5.636	5.093	0.338	0.138	1.572	1.367	0.35	2.44	1.02	5.36	0.24	1.47
8	0.588	0.785	2.656	1.858	1.788	1.048	5.121	6.387	0.342	0.257	1.439	1.144	0.69	2.26	1.42	5.75	0.30	1.29
9	1.872	1.702	2.913	2.856	1.014	1.372	5.605	5.350	0.175	0.167	1.688	1.085	1.79	2.88	1.19	5.48	0.17	1.39
11	0.996	1.639	3.456	2.770	1.334	1.536	5.748	5.076	0.774	0.953	1.156	1.531	1.32	3.11	1.44	5.41	0.86	1.34
12	2.064	1.279	3.280	3.167	1.612	1.451	5.735	5.709	--	--	--	--	1.67	3.22	1.53	5.72	--	--
13	1.491	1.491	3.004	2.873	0.940	1.217	5.517	6.087	0.050	0.054	0.422	0.590	1.49	2.94	1.08	5.80	0.05	0.51
14	0.578	0.512	2.039	1.942	1.597	1.396	5.665	5.871	0.472	0.229	1.311	1.698	0.55	1.99	1.50	5.77	0.35	1.50
16	0.432	0.448	2.766	3.467	1.550	1.879	3.551	5.519	0.660	0.993	2.692	2.424	0.44	3.12	1.71	4.54	0.83	*2.56* g
17	1.281	1.111	2.619	2.694	1.055	1.285	5.688	5.707	0.200	0.180	1.349	0.864	1.20	2.66	1.17	5.70	0.19	1.11
18	0.318	0.149	2.116	2.442	1.327	1.212	5.191	4.840	0.285	0.212	1.518	1.547	0.23	2.28	1.27	5.02	0.25	1.53
19	--	--	--	--	--	--	--	--	0.172	0.100	1.454	1.141	--	--	--	--	0.14	1.30
20	0.351	0.312	2.005	2.412	1.008	1.524	5.555	5.658	0.149	0.181	1.723	1.254	0.33	2.21	1.27	5.61	0.17	1.49

Sample		
A	=	common wheat
B	=	common wheat
C	=	rye
D	=	rye
E	=	durum wheat
F	=	durum wheat

Sample	A	B	C	D	E	F
Mean of test results	**1.04**	**2.68**	**1.34**	**5.57**	**0.34**	**1.40**
Number of laboratories	15	15	15	15	15	14
Repeatability standard deviation s_r	0.24	0.32	0.23	0.55	0.16	0.27
Variation coefficient of repeatability	23 %	12 %	17 %	10 %	47 %	19 %
Repeatability r	**0.68**	**0.90**	**0.65**	**1.55**	**0.45**	**0.75**
Reproducibility standard deviation s_R	0.63	0.48	0.25	0.57	0.27	0.38
Variation coefficient of reproducibility	61 %	18 %	19 %	10 %	80 %	27 %
Reproducibility R	**1.77**	**1.34**	**0.71**	**1.58**	**0.77**	**1.06**
Ratio (r/R)	0.38	0.67	0.91	0.98	0.59	0.71

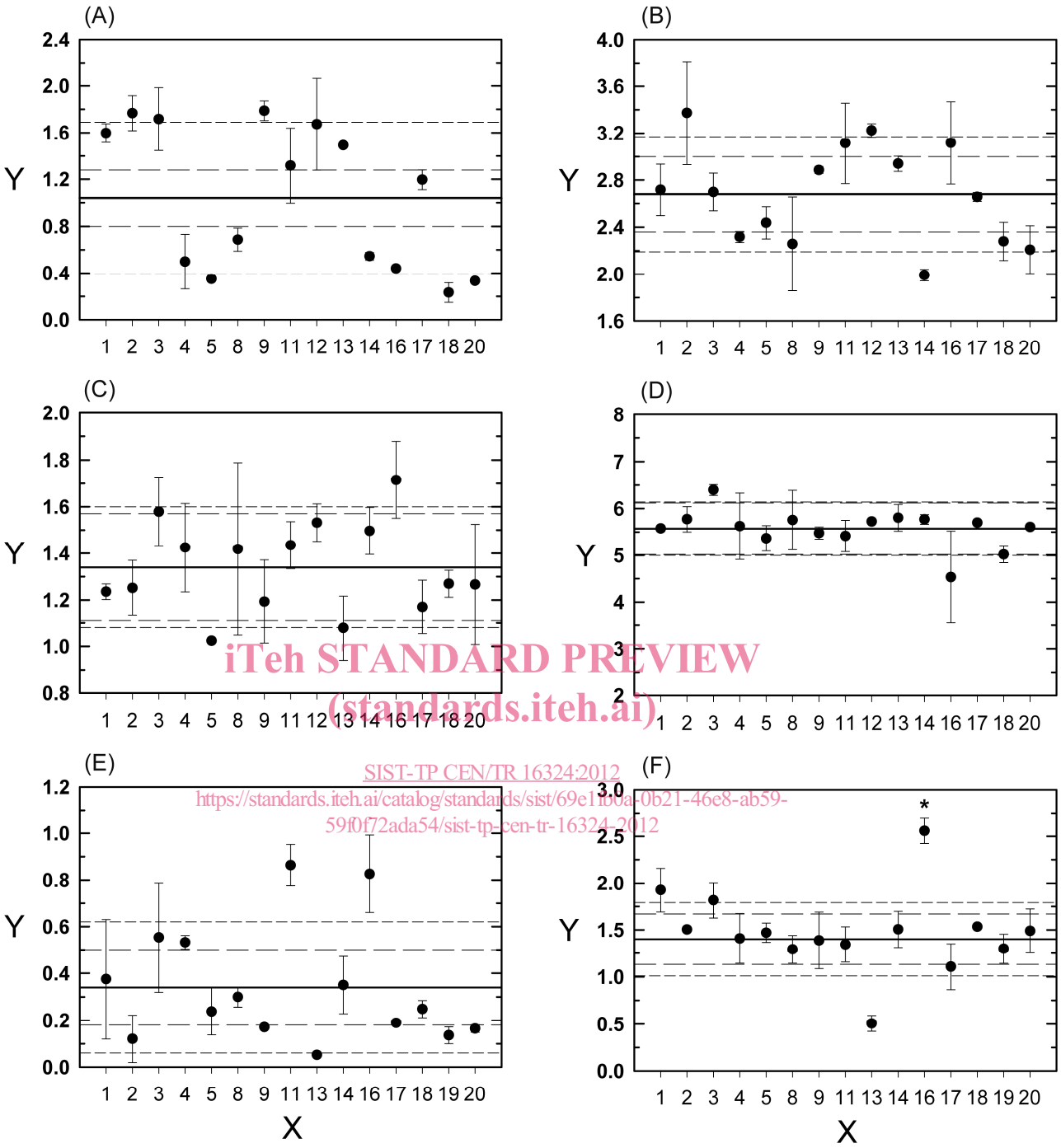
Abbreviations: g *Grubbs outlier, 5%, data eliminated*



Key

X mean value of "broken grains" [w/w%]
Y standard deviation [w/w%]

Figure 1 — Relationship between the standard deviation (s_r, s_R) and the mean value of "broken grains"

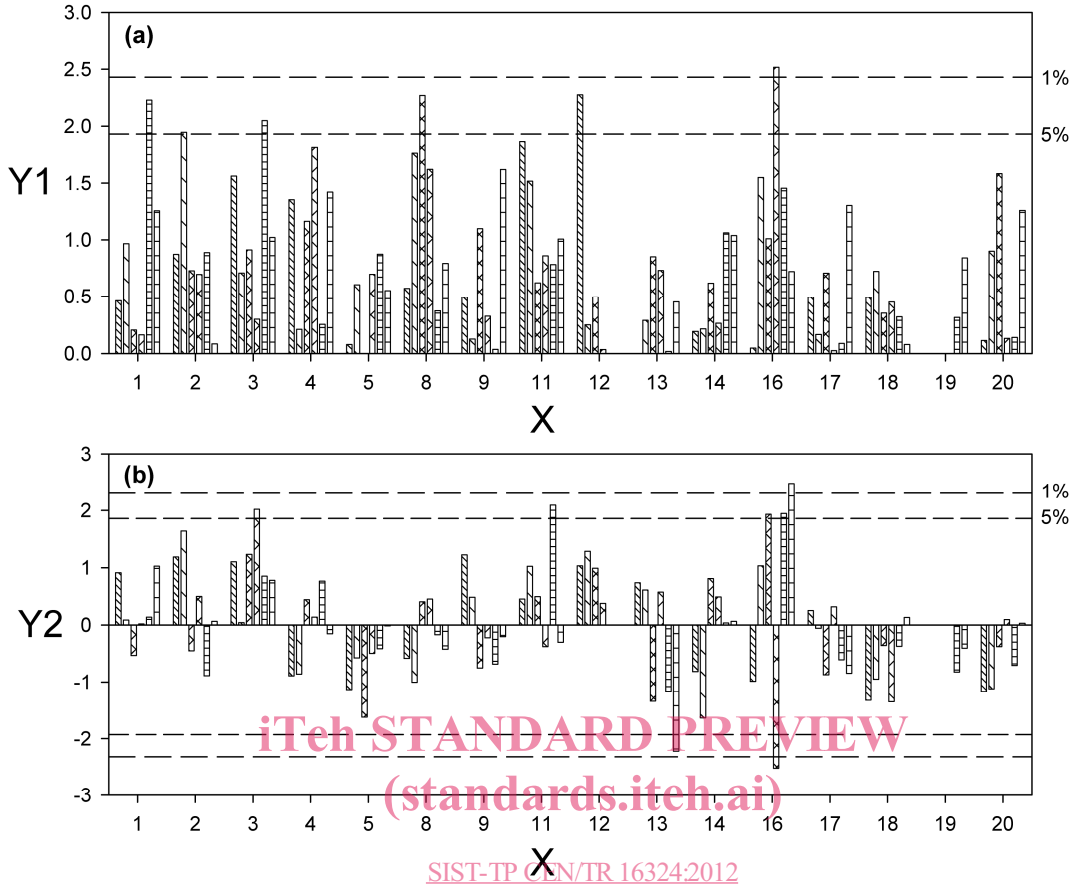


Key

mean s_r s_R (values without eliminated outliers (*))

X lab. No.
Y broken gains [% W/W]

Figure 2 — Single results of the duplicate determination of "broken grains" including the overall mean, standard deviation of repeatability (s_r) and reproducibility (s_R) for the six test samples A to F



Key

A (wheat)

B (wheat)

C (rye)

D (rye)

E (durum)

F (durum)

X laboratory

Y1 Mandel's statistic, *K*

Y2 Mandel's statistic, *h*

Figure 3 — Determination of "broken grains": (a) Mandel's within-laboratory statistic, k, and (b) Mandel's between laboratory consistency statistic, h, each grouped by laboratories

5 Results of the determination of "shrivelled grains"

Table 2 — Collation of test results, outliers and precision data on the determination of "shrivelled grains"

Determination of "shrivelled grains"

	Single results of the dupliacte determination (results given in [w/w%])												Mean of duplicate determination (results given in [w/w%])					
Sample:	A	A	B	B	C	C	D	D	E	E	F	F	A	B	C	D	E	F
Lab.No																		
1	0.904	1.244	2.601	2.201	0.501	0.174	1.322	1.040	0.000	0.000	0.183	0.116	1.07	2.40	0.34	1.18	0.00	0.15
2	1.316	1.339	4.821	4.134	2.605	3.215	2.451	3.442	3.521	2.770	3.275	2.476	1.33	*4.48* G	*2.91* c	*2.95* c	*3.15* C	*2.88* C
3	1.085	1.190	2.502	2.114	0.322	0.315	0.656	0.896	0.000	0.000	0.293	0.167	1.14	2.31	0.32	0.78	0.00	0.23
4	1.287	1.281	2.093	2.063	0.469	0.521	1.143	0.811	0.000	0.000	0.085	0.213	1.28	2.08	0.50	0.98	0.00	0.15
5	1.428	0.969	2.485	1.929	0.236	0.192	1.202	0.653	0.053	0.039	0.068	0.388	*1.20* k	2.21	0.21	0.93	0.05	0.23
8	1.192	1.208	2.676	2.583	0.237	0.266	0.664	0.703	0.098	0.066	0.294	0.217	1.20	2.63	0.25	0.68	0.08	0.26
9	1.054	1.139	2.500	1.579	0.567	0.463	0.927	0.979	0.000	0.000	0.167	0.136	1.10	*2.04* k	0.52	0.95	0.00	0.15
11	1.129	1.429	2.580	2.555	0.543	0.462	0.807	1.056	0.000	0.000	0.234	0.269	1.28	2.57	0.50	0.93	0.00	0.25
12	0.860	1.321	2.624	2.628	0.360	0.548	1.211	1.200	--	--	--	--	*1.09* k	2.63	0.45	1.21	--	--
13	0.832	0.832	1.161	1.280	0.687	0.459	0.912	1.402	0.005	0.007	0.114	0.149	0.83	1.22	0.57	1.16	0.01	0.13
14	1.035	1.316	2.596	2.936	0.450	0.655	1.645	2.045	0.056	0.526	1.009	1.252	1.18	2.77	0.55	*1.85* g	*0.29* C	1.13
16	1.111	1.241	2.130	2.127	0.246	0.490	0.564	0.711	0.000	0.000	0.217	0.152	1.18	2.13	0.37	0.64	0.00	0.18
17	1.201	1.111	2.234	2.773	0.227	0.159	0.769	0.710	0.000	0.000	0.159	0.157	1.16	2.50	0.19	0.74	0.00	0.16
18	1.345	1.094	2.133	1.637	0.172	0.299	0.859	0.772	0.000	0.000	0.195	0.230	1.22	1.89	0.24	0.82	0.00	0.21
19	--	--	--	--	--	--	--	--	0.000	0.000	0.148	0.151	--	--	--	--	0.00	0.15
20	0.864	0.833	1.412	2.233	0.000	0.000	0.390	0.243	0.000	0.021	0.215	0.181	0.85	1.82	0.00	0.32	0.01	0.20

Sample		
A	=	common wheat
B	=	common wheat
C	=	rye
D	=	rye
E	=	durum wheat
F	=	durum wheat

Sample	A	B	C	D	E	F
Mean of test results	**1.14**	**2.24**	**0.36**	**0.87**	**0.01**	**0.26**
Number of laboratories	13	13	14	13	13	14
Repeatability standard deviation s_r	0.12	0.28	0.11	0.19	0.01	0.09
Variation coefficient of repeatability	11 %	12 %	31 %	22 %	72 %	34 %
Repeatability r	**0.34**	**0.77**	**0.31**	**0.52**	**0.02**	**0.24**
Reproducibility standard deviation s_R	0.17	0.47	0.18	0.28	0.03	0.26
Variation coefficient of reproducibility	15 %	21 %	51 %	32 %	229 %	103 %
Reproducibility R	**0.49**	**1.31**	**0.52**	**0.79**	**0.07**	**0.74**
Ratio (r/R)	0.70	0.59	0.60	0.66	0.31	0.33

Abbreviations:

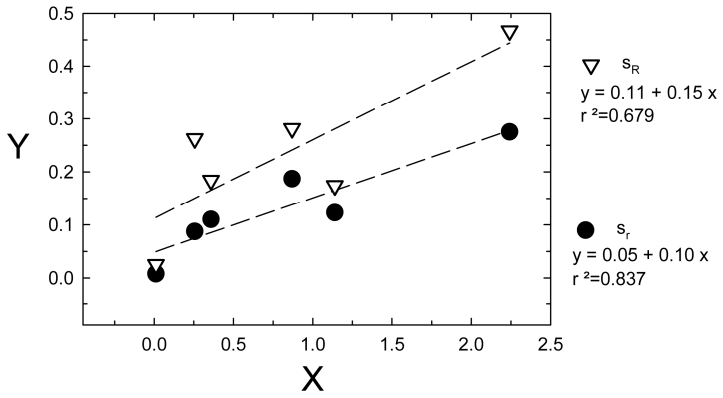
C *Cochtran's outlier, 1%, data eliminated*

c *Cochrans outlier, 5%, data eliminated*

k *Mandel's statistic,k, outlier, 5%, data eliminated*

G *Grubbs outlier, 1%, data eliminated*

g *Grubbs outlier, 5%, data eliminated*

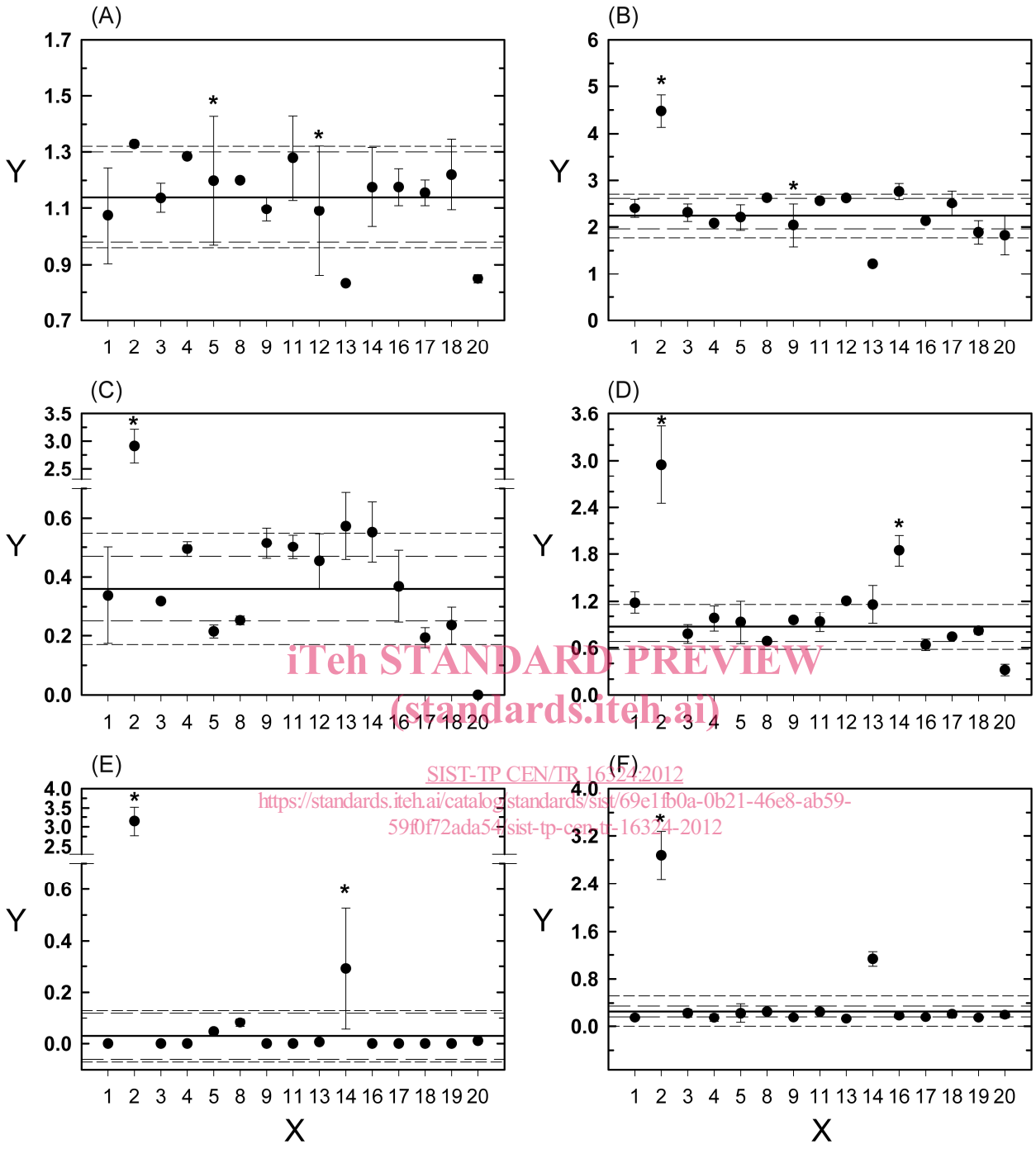


Key

X mean value of "shrivelled grains" [W/W%]

Y standard deviation [W/W%]

Figure 4 — Relationship between the standard deviation (s_r, s_R) and the mean value of "shrivelled grains"

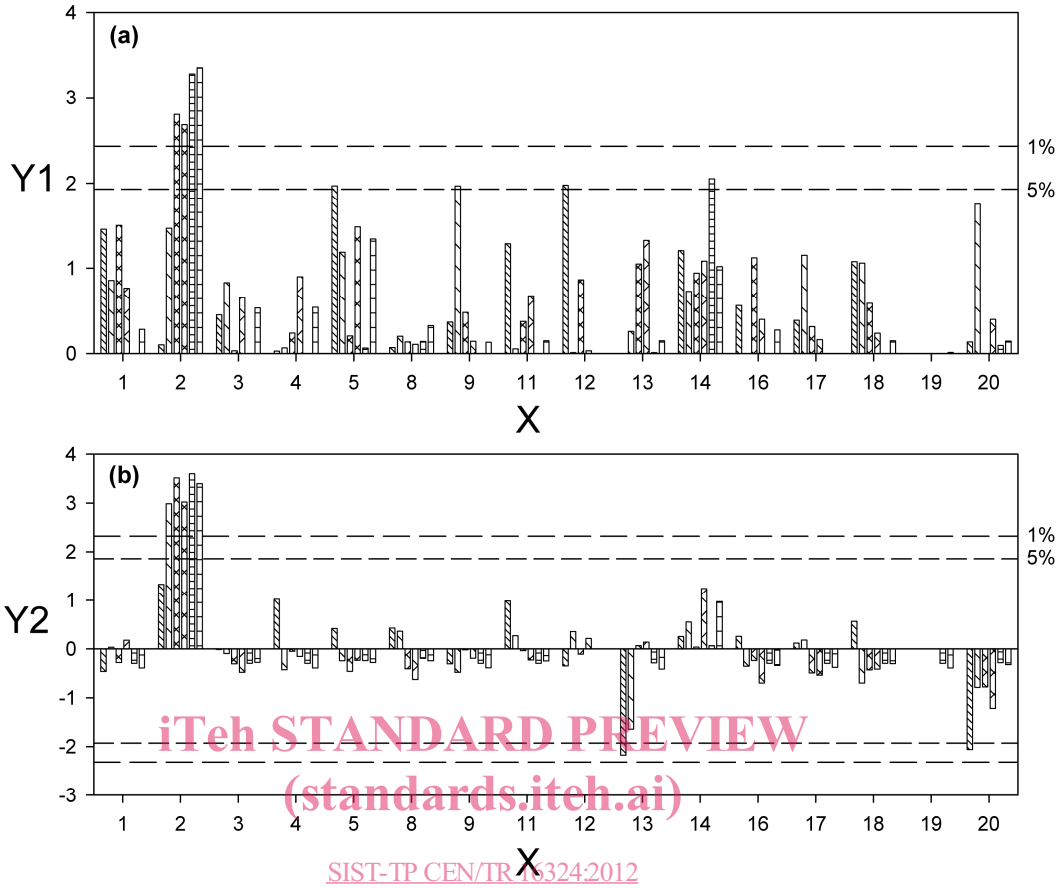


Key

——— mean — — s_r – – – – s_R (values without eliminated outliers (*))

X lab. No.
Y shrivelled grains [W/W%]

Figure 5 — Single results of the duplicate determination of "shrivelled grains" including the overall mean, standard deviation of repeatability (s_r) and reproducibility (s_R) for the six test samples A to F



Key

A (wheat)

B (wheat)

C (rye)

D (rye)

E (durum)

F (durum)

X laboratory

Y1 Mandel's statistic, *K*

Y2 Mandel's statistic, *h*

Figure 6 — Determination of "shrivelled grains": (a) Mandel's within-laboratory statistic, k, and (b) Mandel's between laboratory consistency statistic, h, each grouped by laboratories