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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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National foreword

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TECHNICAL REPORT

RAPPORT TECHNIQUE

TECHNISCHER BERICHT

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English Version

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

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

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

This Technical Report was approved by CEN on 7 February 2012. It has been drawn up by the Technical Committee CEN/TC 338.

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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.

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

Modifications from CEN Correction Notice 18 September 2012

1) In Clause 2, in the paragraph relating to sample set B, second line, delete "resp.".

2) Regarding Figures 2, 5, 8, 11, 13, 16, 19, 22, 25, 28, 31, 34, 37 and 40, replace the explanation of Key X with "laboratory".

3) Replace all the occurrence of "[% W/W]" or "[W/W %]" with "[w/w %]" in the keys of the figures where this appears (i.e. Figure 2, 4, 5, 7, 8, 10, 11, 13, 15, 16, 18, 19, 21, 22, 24, 26, 27, 28, 30, 31, 33, 34, 36, 37, 39 and 40).

4) In the keys of Figures 3, 6, 9, 12, 14, 17, 20, 23, 26, 29, 32, 35, 38 and 41, write for the explanation of Y1 the Mandel's statistic k in italics and in lower case.

5) In the heading of Figure 13, replace "six test samples" with "four test samples".

6) In the heading of Table 22, correct "recision" with "Precision".

7) Replace Figure 42 with the new one.

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.

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. 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.

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[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.

[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.

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.

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"

Sample:	Single results of the dupliacte determination (results given in [w/w%])												Mean of duplicate determination (results given in [w/w%])					
Lab.No	A	A	B	B	C	C	D	D	E	E	F	F	A	B	C	D	E	F
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*
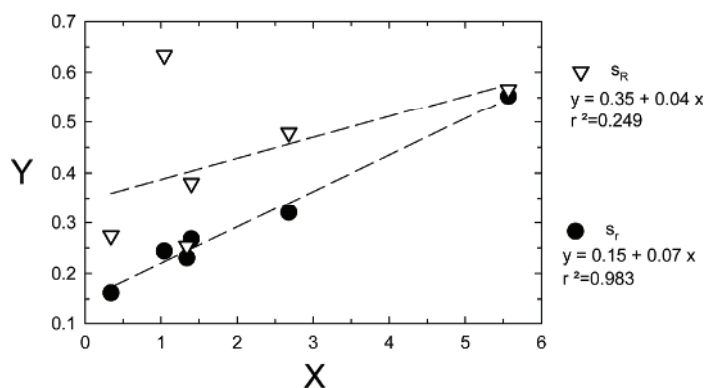
Key

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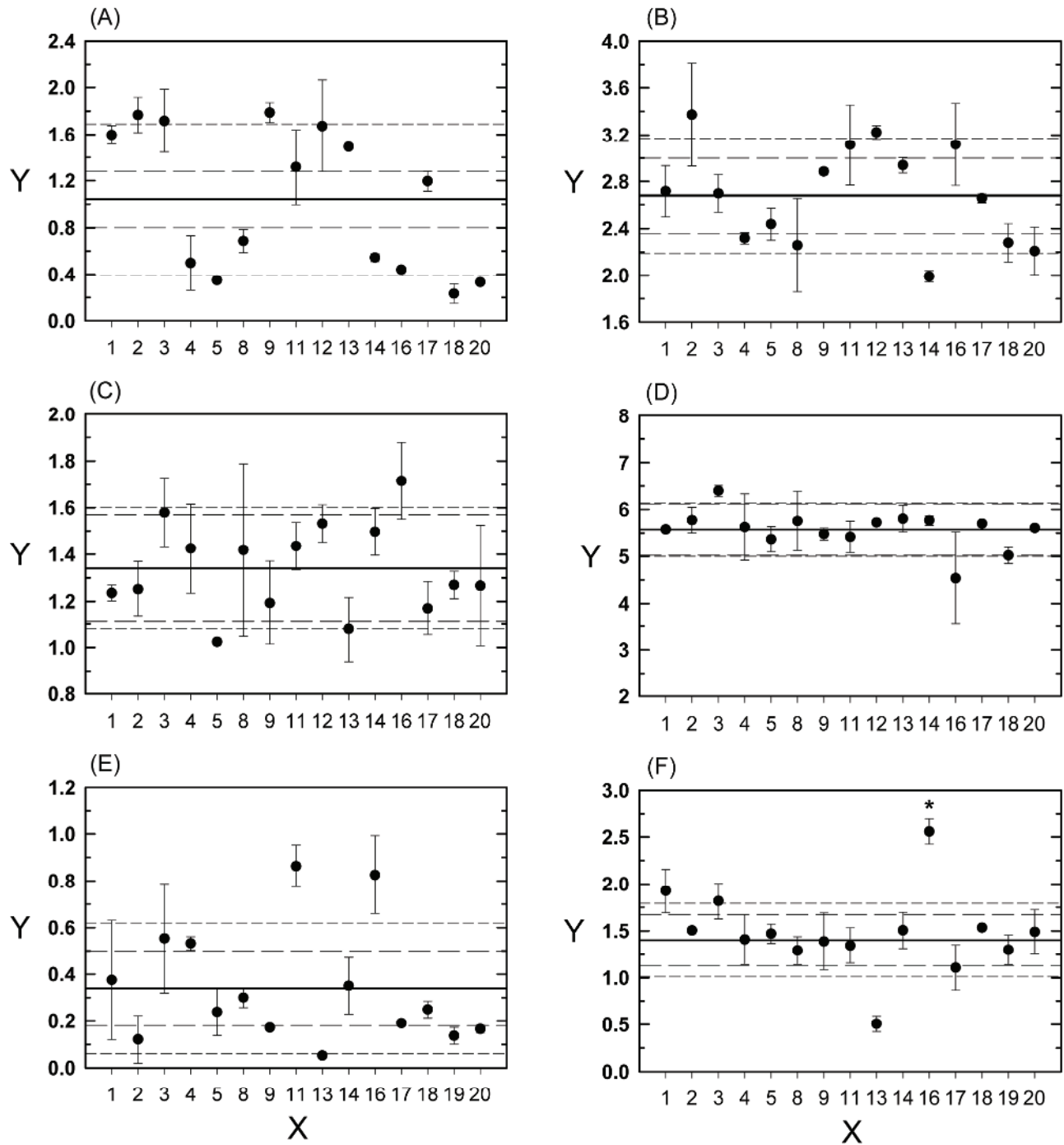
——— mean   — — s_r – – – s_R (values without eliminated outliers (*))

X   laboratory
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
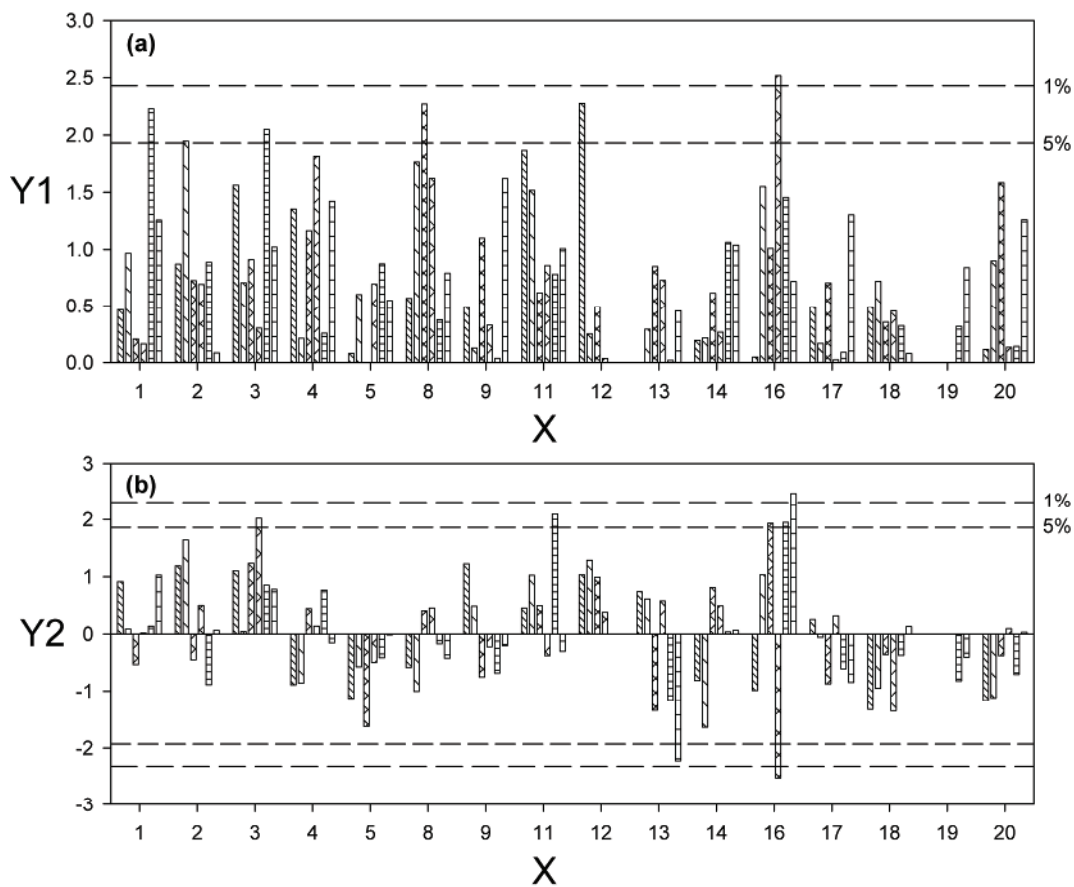
▨	A (wheat)
◨	B (wheat)
▨	C (rye)
▧	D (rye)
▤	E (durum)
▭	F (durum)

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
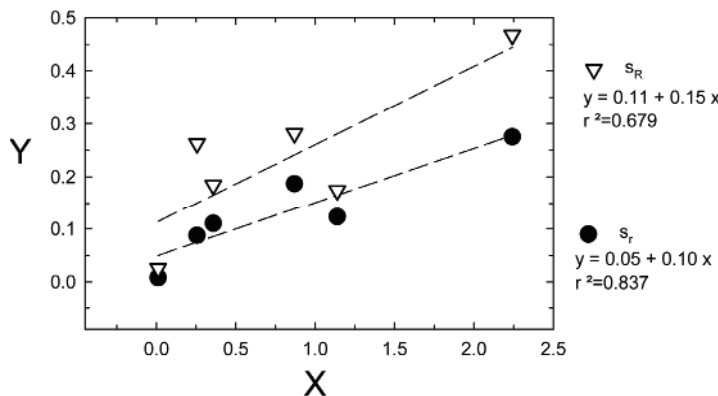
5 Results of the determination of "shrivelled grains"

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

Determination of "shrivelled grains"

Lab.No	A	A	B	B	C	C	D	D	E	E	F	F	A	B	C	D	E	F
	Single results of the dupliacte determination (results given in [w/w%])												Mean of duplicate determination (results given in [w/w%])					
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* G — *Grubbs outlier, 1%, data eliminated*

c — *Cochrans outlier, 5%, data eliminated* g — *Grubbs outlier, 5%, data eliminated*

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

s_R
y = 0.11 + 0.15 x
r^2=0.679

s_r
y = 0.05 + 0.10 x
r^2=0.837

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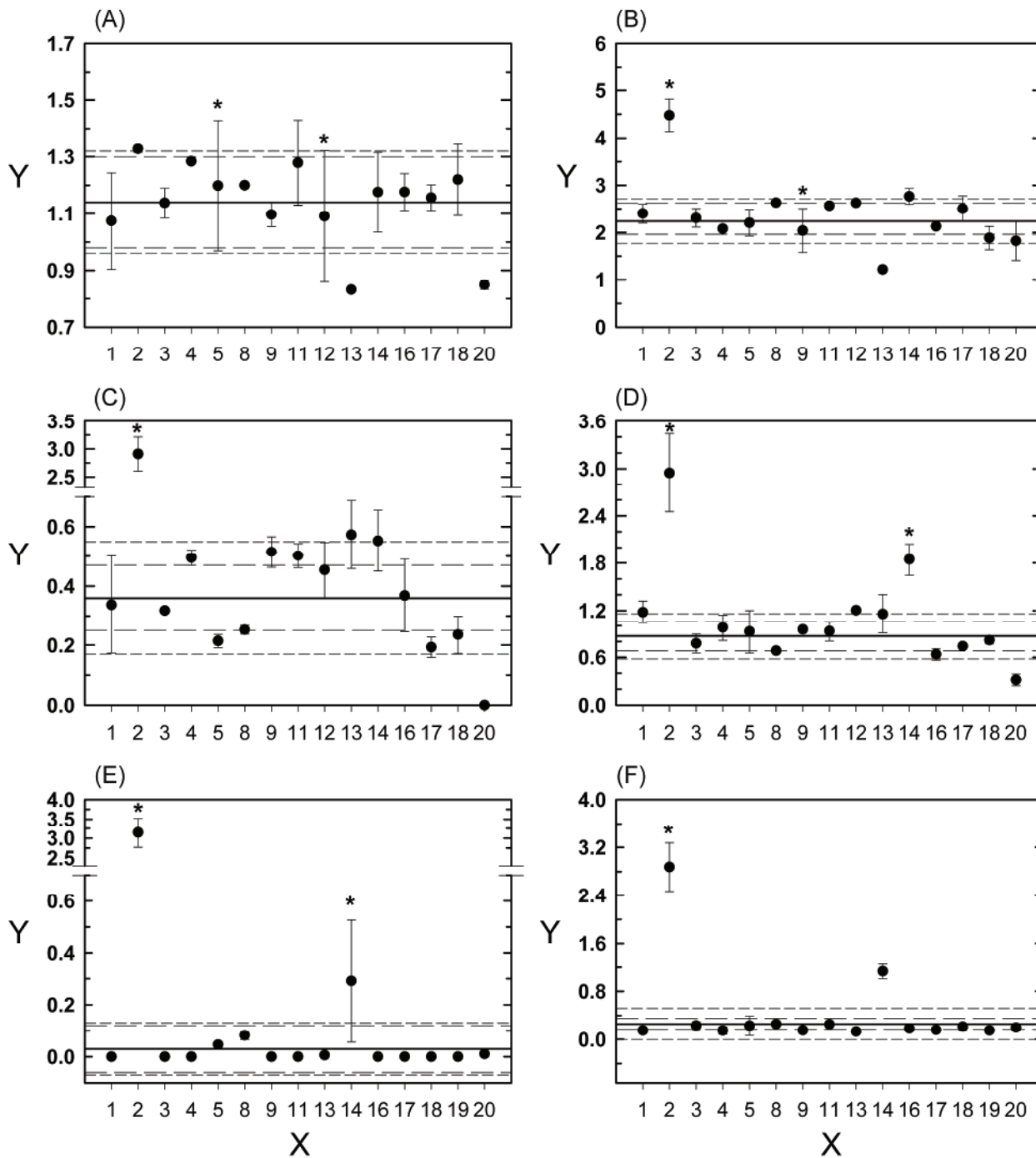
———— mean   — — s_r — — — — s_R (values without eliminated outliers (*))

X   laboratory
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
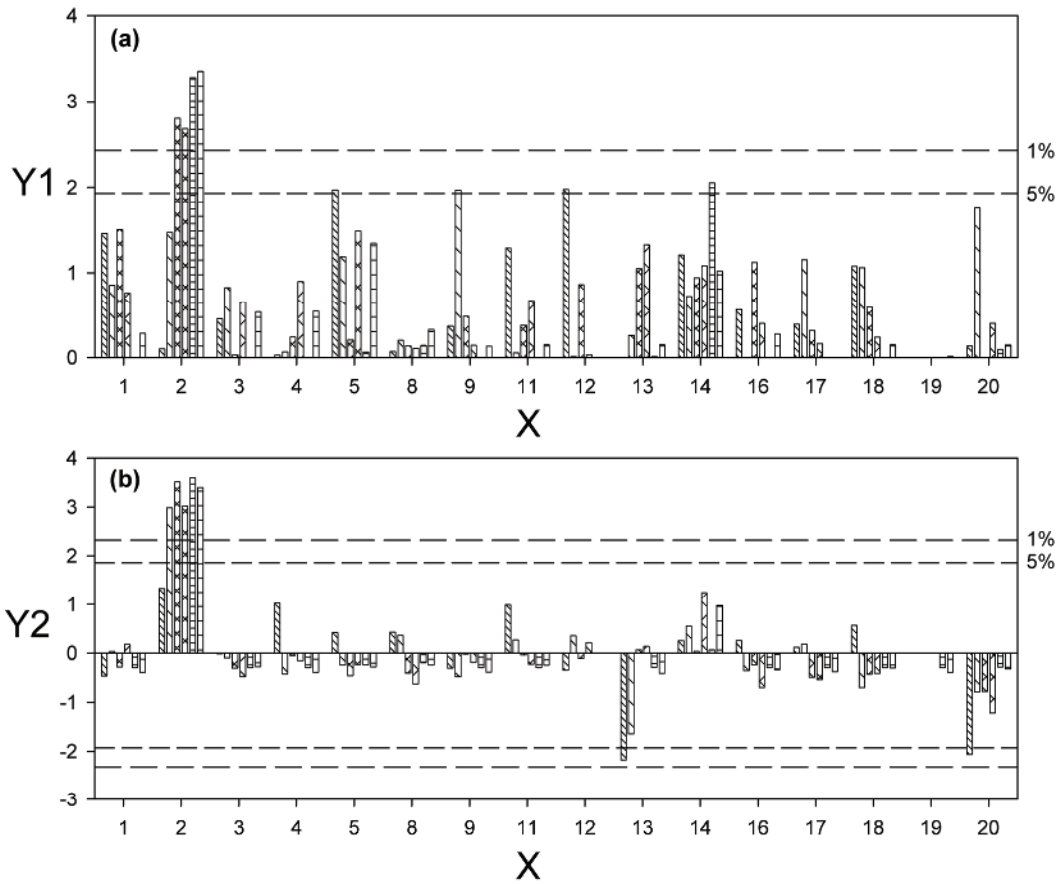
▨	A (wheat)
◪	B (wheat)
▩	C (rye)
▨	D (rye)
▤	E (durum)
▭	F (durum)

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

6 Results of the determination of "other cereals"

Table 3 — Collation of test results, outliers and precision data on the determination of "other cereals"

Determination of "other cereals"

	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	0.000	0.000	1.805	1.010	1.199	1.873	0.510	0.985	1.305	1.165	3.556	2.847	0.00	1.41	1.54	0.75	1.24	3.20
2	0.223	0.126	2.003	2.389	2.407	1.727	1.083	1.356	1.328	0.950	3.484	3.849	0.17	2.20	2.07	1.22	1.14	3.67
3	0.000	0.000	2.740	1.570	1.864	1.683	0.233	1.033	0.485	1.254	3.697	3.996	0.00	2.16	1.77	0.63	0.87	3.85
4	0.110	0.219	1.946	2.674	1.547	1.692	0.486	1.582	0.499	0.742	3.989	3.853	0.16	2.31	1.62	_1.03_ k	0.62	3.92
5	0.012	0.142	0.442	0.423	0.686	0.790	0.571	0.284	0.588	0.360	1.646	2.606	0.08	0.43	0.74	0.43	0.47	2.13
8	0.000	0.000	0.333	2.007	1.413	3.468	0.776	0.830	0.493	0.704	3.323	3.444	0.00	1.17	_2.44_ c	0.80	0.60	3.38
9	0.000	0.211	4.260	0.734	1.085	1.171	1.201	0.836	0.841	0.620	3.841	2.923	0.11	_2.50_ C	1.13	1.02	0.73	3.38
11	0.205	0.112	1.069	1.170	2.174	1.670	0.293	1.211	0.570	0.758	2.488	2.088	0.16	1.12	1.92	0.75	0.66	2.29
12	0.000	0.189	0.867	0.882	1.583	0.983	0.060	0.909	--	--	--	--	0.09	0.87	1.28	0.48	--	--
13	0.043	0.043	0.520	0.522	1.265	1.177	0.302	0.751	0.303	0.157	0.000	0.000	0.04	0.52	1.22	0.53	0.23	_0.00_ G
14	0.128	0.000	0.396	0.713	1.331	1.607	0.584	0.715	0.693	0.470	2.539	3.767	0.06	0.55	1.47	0.65	0.58	3.15
16	0.146	0.099	1.544	0.969	1.210	1.544	0.328	0.120	0.590	0.355	2.211	2.311	0.12	1.26	1.38	0.22	0.47	2.26
17	0.160	0.261	1.171	1.743	1.948	1.317	0.497	0.262	0.845	2.176	2.190	2.436	0.21	1.46	1.63	0.38	_1.51_ c	2.31
18	0.139	0.148	1.525	1.292	1.680	1.660	0.749	0.842	0.575	0.936	4.620	4.243	0.14	1.41	1.67	0.80	0.76	4.43
19	--	--	--	--	--	--	--	--	0.840	1.159	3.428	3.269	--	--	--	--	1.00	3.35
20	0.126	0.053	1.175	0.867	1.059	2.250	0.743	0.537	0.769	1.100	2.784	3.427	0.09	1.02	1.65	0.64	0.93	3.11

Sample					A	B	C	D	E	F
A	=	common wheat		**Mean of test results**	**0.10**	**1.28**	**1.51**	**0.66**	**0.74**	**3.17**
B	=	common wheat		Number of laboratories	15	14	14	14	14	14
C	=	rye		Repeatability standard deviation s_r	0.07	0.48	0.36	0.33	0.23	0.42
D	=	rye		Variation coefficient of repeatability	76 %	37 %	24 %	50 %	31 %	13 %
E	=	durum wheat		**Repeatability r**	**0.21**	**1.34**	**1.01**	**0.93**	**0.64**	**1.16**
F	=	durum wheat		Reproducibility standard deviation s_R	0.08	0.70	0.43	0.35	0.32	0.76
				Variation coefficient of reproducibility	88 %	55 %	28 %	53 %	44 %	24 %
				Reproducibility R	**0.24**	**1.95**	**1.19**	**0.98**	**0.90**	**2.13**
				Ratio (r/R)	0.87	0.69	0.84	0.95	0.71	0.55

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_
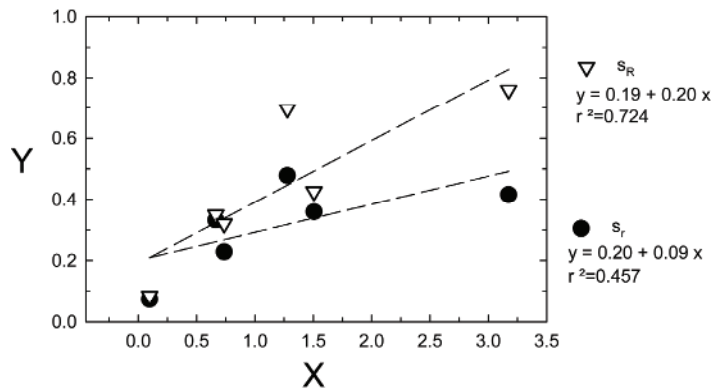
Key
X   mean value of "other cereals" [w/w %]
Y   standard deviation [w/w %]

### Figure 7 — Relationship between the standard deviation (s_r, s_R) and the mean value of "other cereals"

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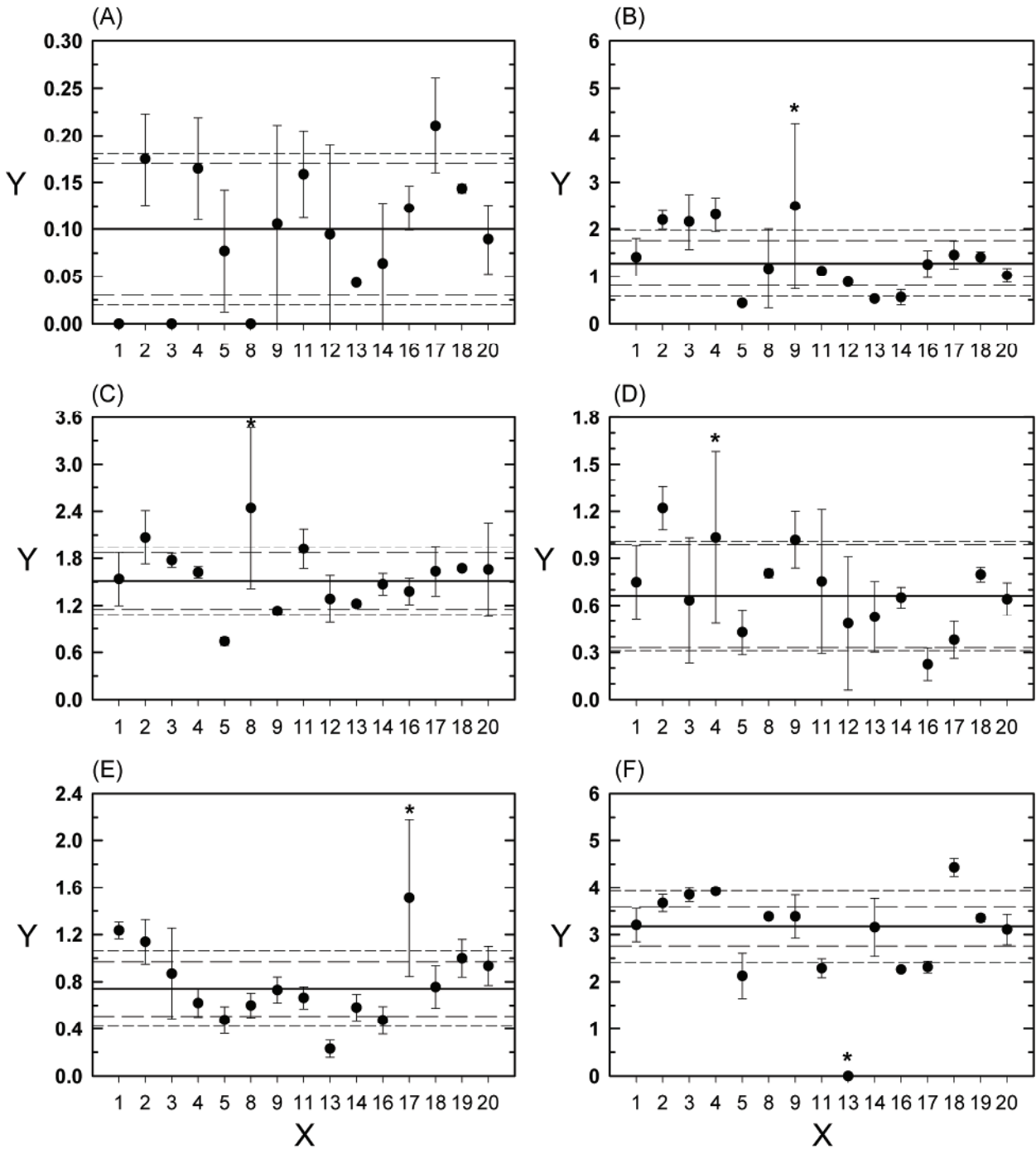
———— mean   — — s_r – – – – s_R (values without eliminated outliers (*))

X   laboratory
Y   other cereals [w/w %]

Figure 8 — Single results of the duplicate determination of "other cereals" including the overall mean, standard deviation of repeatability (s_r) and reproducibility (s_R) for the six test samples A to F

Key
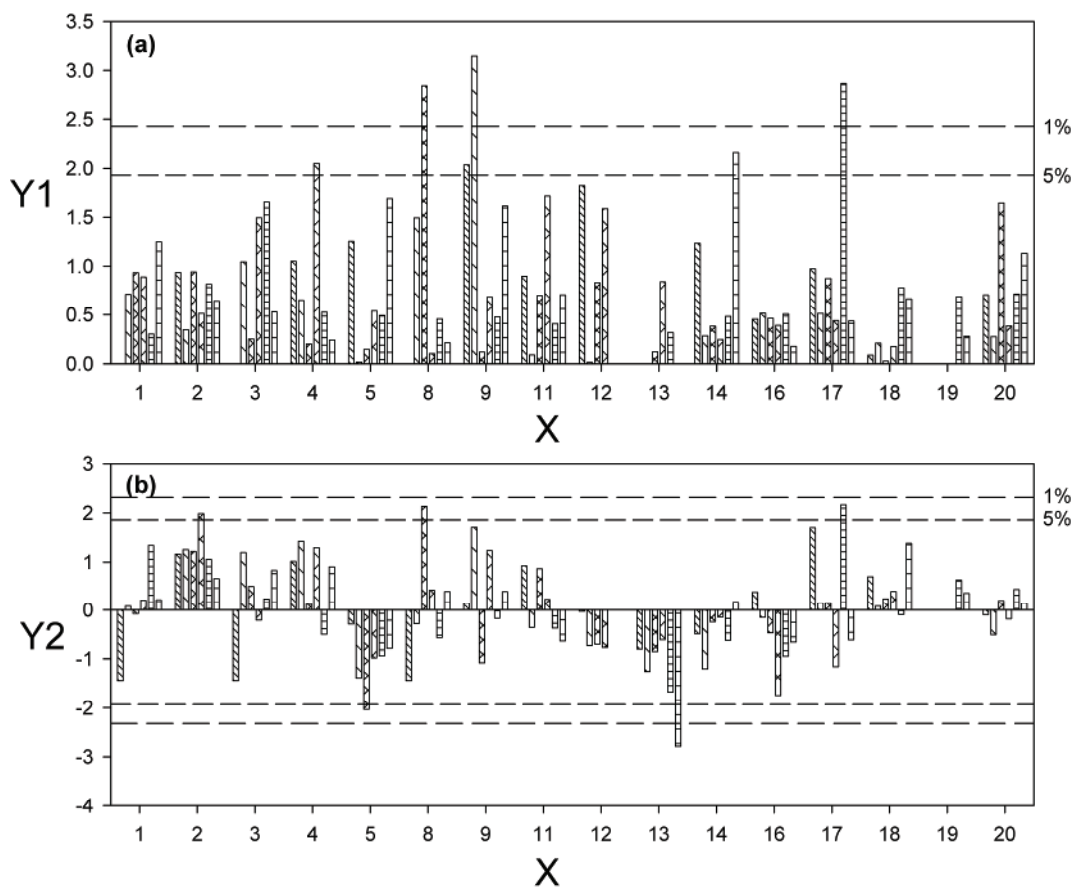
▨	A (wheat)
◹	B (wheat)
▧	C (rye)
◈	D (rye)
▤	E (durum)
▭	F (durum)

X   laboratory

Y1   Mandel's statistic, k

Y2   Mandel's statistic, h

Figure 9 — Determination of "other cereals": (a) Mandel's within-laboratory statistic, k, and (b) Mandel's between laboratory consistency statistic, h, each grouped by laboratories

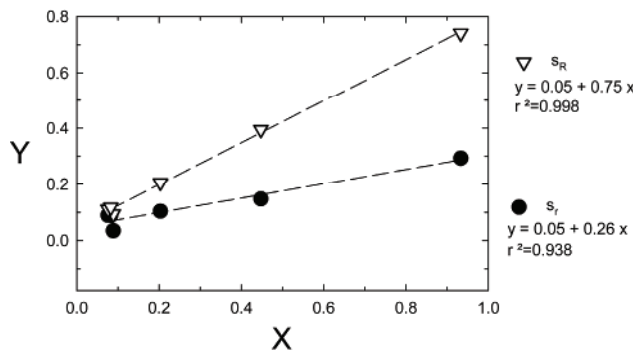# 7 Results of the determination of "grains damaged by pests"

Table 4 — Collation of test results, outliers and precision data on the determination of "grains damaged by pests"

Determination of "grains damaged by pests"

	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	0.487	0.272	0.404	0.276	0.043	0.000	0.000	0.072	0.051	0.086	0.000	0.000	0.38	0.34	0.02	0.04	0.07	0.00
2	0.342	0.431	0.241	0.090	0.101	0.089	0.000	0.053	0.074	0.137	0.000	0.095	0.39	0.17	0.10	0.03	0.11	0.05
3	0.253	0.401	0.147	0.440	0.069	0.000	0.086	0.150	0.063	0.046	0.046	0.063	0.33	0.29	0.03	0.12	0.05	0.05
4	2.019	1.296	1.056	0.889	0.624	0.164	0.346	0.548	0.000	0.000	0.000	0.091	1.66	0.97	*0.39* C	0.45	0.00	0.05
5	0.427	0.655	0.232	0.129	0.000	0.000	0.288	0.200	0.000	0.000	0.000	0.000	0.54	0.18	0.00	0.24	0.00	0.00
8	1.529	1.474	1.625	0.619	0.186	0.232	1.100	0.499	0.412	0.154	0.078	0.080	1.50	*1.12* C	0.21	*0.80* c	0.28	0.08
9	0.347	0.456	0.084	0.121	0.064	0.070	0.059	0.196	0.000	0.146	0.000	0.289	0.40	0.10	0.07	0.13	0.07	0.14
11	0.604	0.400	0.222	0.041	0.039	0.015	0.042	0.223	0.000	0.000	0.000	0.000	0.50	0.13	0.03	0.13	0.00	0.00
12	0.569	0.666	0.113	0.056	0.000	0.000	0.145	0.109	--	--	--	--	0.62	0.08	0.00	0.13	--	--
13	0.245	0.245	0.204	0.204	0.310	0.270	0.129	0.434	0.000	0.000	0.000	0.000	0.25	0.20	0.29	0.28	0.00	0.00
14	1.170	1.618	0.815	0.873	0.195	0.187	0.440	0.159	0.000	0.077	0.058	0.162	1.39	0.84	0.19	0.30	0.04	0.11
16	1.882	2.515	0.810	0.714	0.000	0.094	0.097	0.191	0.064	0.337	0.089	0.328	2.20	0.76	0.05	0.14	0.20	0.21
17	0.256	0.163	0.154	0.158	0.081	0.000	0.032	0.062	0.069	0.000	0.000	0.000	0.21	0.16	0.04	0.05	0.03	0.00
18	1.255	1.798	0.570	1.197	0.097	0.164	0.059	0.110	0.000	0.000	0.154	0.086	1.53	0.88	0.13	0.08	0.00	0.12
19	--	--	--	--	--	--	--	--	0.310	0.098	0.456	0.235	--	--	--	--	0.20	0.35
20	1.621	2.600	1.121	1.170	1.037	0.862	0.771	0.696	1.228	0.832	3.767	2.402	2.11	1.15	*0.95* G	0.73	*1.03* G	*3.08* C

Sample				Sample	A	B	C	D	E	F
A	=	common wheat		**Mean of test results**	**0.93**	**0.45**	**0.09**	**0.20**	**0.08**	**0.08**
B	=	common wheat		Number of laboratories	15	14	13	14	14	14
C	=	rye		Repeatability standard deviation s_r	0.29	0.15	0.03	0.10	0.09	0.09
D	=	rye		Variation coefficient of repeatability	31 %	33 %	39 %	51 %	118 %	108 %
E	=	durum wheat		**Repeatability r**	**0.82**	**0.41**	**0.10**	**0.29**	**0.25**	**0.25**
F	=	durum wheat		Reproducibility standard deviation s_R	0.74	0.40	0.09	0.21	0.11	0.12
				Variation coefficient of reproducibility	79 %	88 %	106 %	101 %	146 %	143 %
				Reproducibility R	**2.08**	**1.11**	**0.26**	**0.58**	**0.31**	**0.33**
				Ratio (r/R)	0.40	0.37	0.37	0.50	0.81	0.76

Abbreviations: C   *Cochtran's outlier, 1%, data eliminated*   G   *Grubbs outlier, 1%, data eliminated*

c   *Cochrans outlier, 5%, data eliminated*

Key

X   mean value of "grains damaged by pests" [w/w %]
Y   standard deviation [w/w %]

Figure 10 — Relationship between the standard deviation (s_r, s_R) and the mean value of "grains damaged by pests"
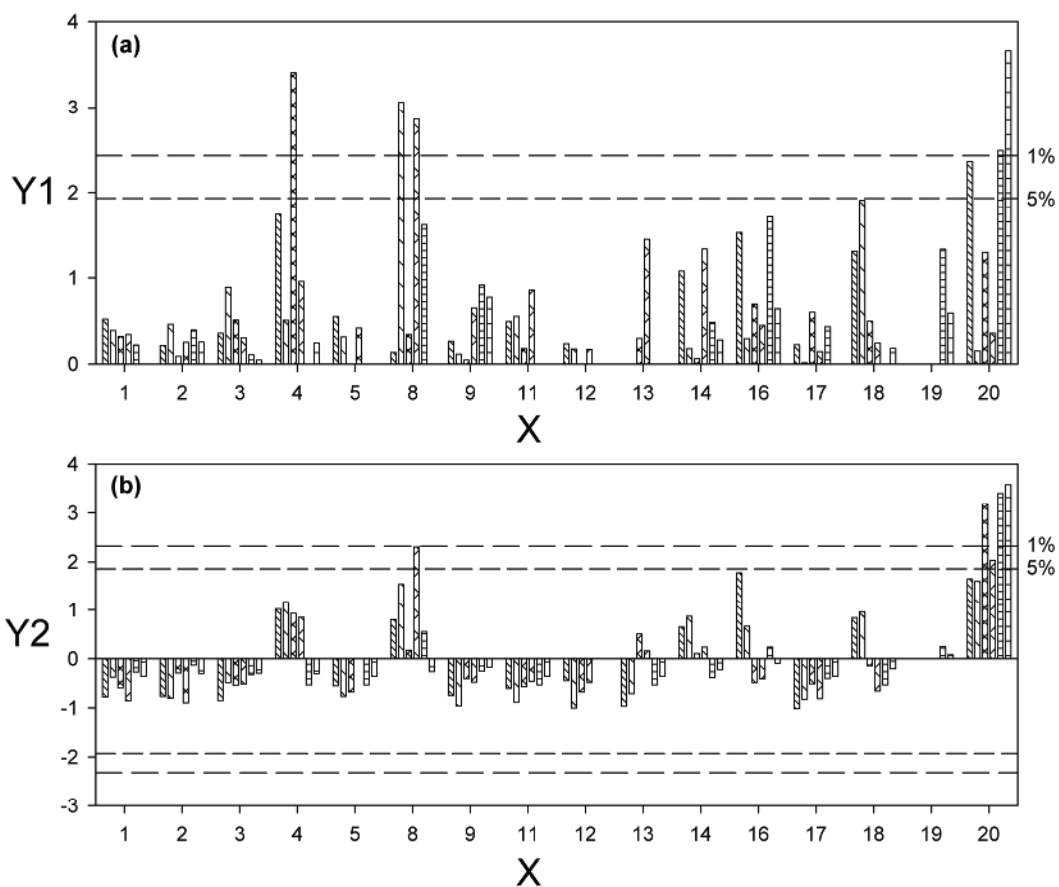
Key
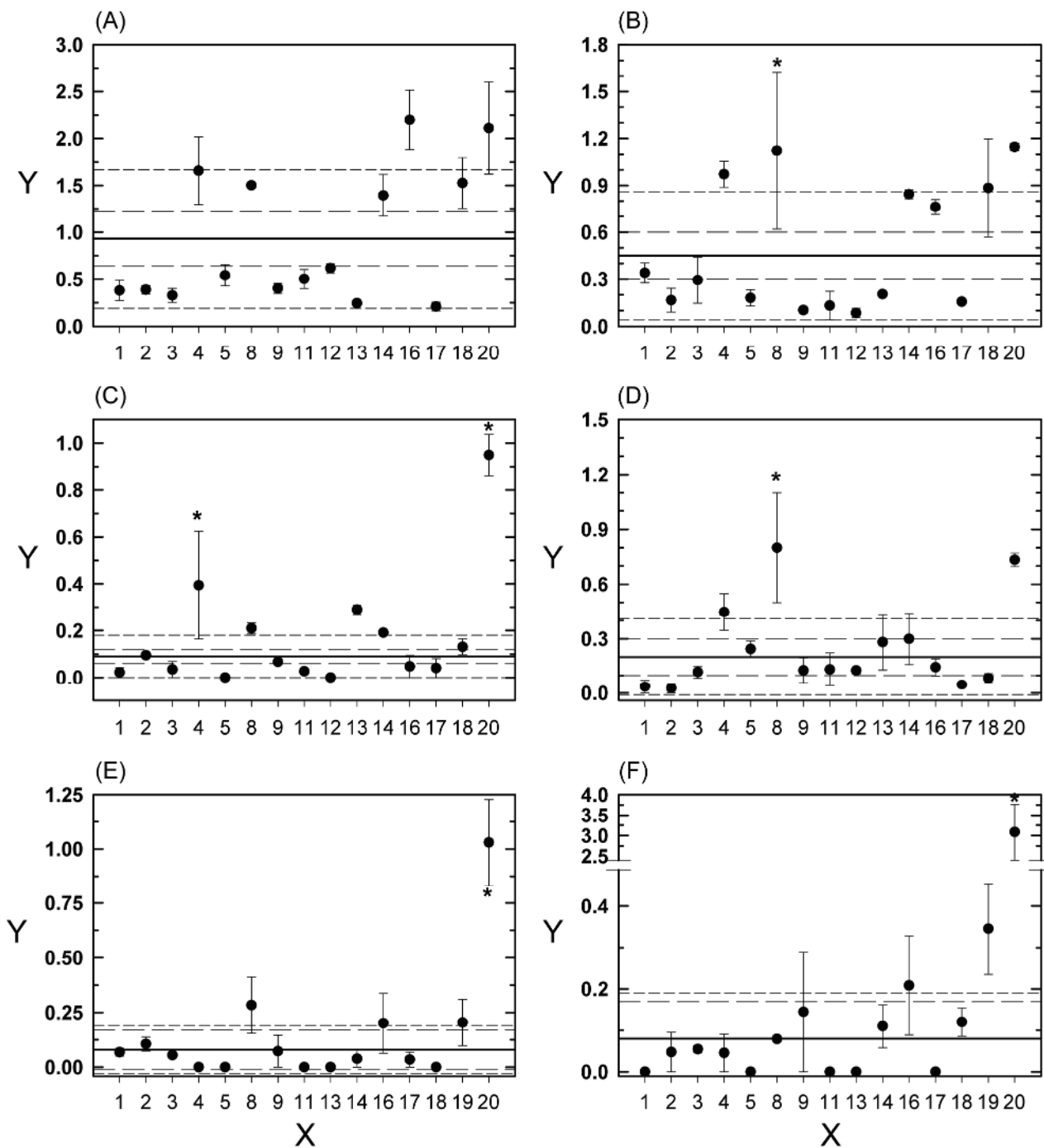
─────── mean   ─ ─ ─ s_r ─ ─ ─ ─ s_R (values without eliminated outliers (*))

X   laboratory
Y   grains damaged by pests [w/w %]

Figure 11 — Single results of the duplicate determination of "grains damaged by pests" 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 12 — Determination of "grains damaged by pests": (a) Mandel's within-laboratory statistic, *k*, and (b) Mandel's between laboratory consistency statistic, *h*, each grouped by laboratories

8 Results of the determination of "grains in which the germ is discoloured, mottled grains"

Table 5 — Collation of test results, outliers and precision data on the determination of "mottled grains"

Determination of "mottled grains, grains in which the germ is discoloured"

	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	E	E	F	F		A	B	E	F
Lab.No													
1	0.000	0.000	0.490	0.379	1.474	1.422	3.505	3.440		0.00	0.43	1.45	3.47
2	0.000	0.072	0.951	1.302	6.649	4.721	5.797	5.341		0.04	1.13	5.69	5.57
3	0.175	0.000	0.704	0.635	20.056	19.632	21.462	21.633		0.09	0.67	19.84	21.55 g
4	0.008	0.000	0.564	0.246	12.208	11.570	10.446	8.318		0.00	0.41	11.89	9.38
5	0.000	0.000	0.000	0.000	1.708	2.864	3.591	3.880		0.00	0.00	2.29	3.74
8	0.000	0.000	0.530	0.390	2.781	2.895	2.512	2.239		0.00	0.46	2.84	2.38
9	0.000	0.000	0.613	0.377	2.073	2.150	2.811	2.631		0.00	0.50	2.11	2.72
11	0.164	0.249	0.530	0.412	16.240	16.112	14.033	16.053		0.21	0.47	16.18	15.04
12	0.470	0.365	0.418	0.464	--	--	--	--		0.42	0.44	--	--
13	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000		0.00	0.00	0.00	0.00
14	0.212	0.000	0.301	0.698	3.264	3.104	5.209	3.775		0.11	0.50	3.18	4.49
16	0.000	0.000	0.199	0.486	5.582	6.731	4.110	4.360		0.00	0.34	6.16	4.24
17	0.000	0.065	0.616	0.238	4.684	4.083	2.459	3.159		0.03	0.43	4.38	2.81
18	0.180	0.000	0.436	0.270	0.000	0.000	0.842	1.800		0.09	0.35	0.00	1.32
19	--	--	--	--	15.504	15.055	14.120	14.351		--	--	15.28	14.24
20	0.000	0.518	0.317	0.206	5.578	6.489	8.691	7.338		0.26 C	0.26	6.03	8.01

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

Sample	A	B	E	F
Mean of test results	**0.07**	**0.43**	**6.49**	**5.53**
Number of laboratories	14	15	15	14
Repeatability standard deviation s_r	0.07	0.16	0.53	0.72
Variation coefficient of repeatability	99 %	37 %	8 %	13 %
Repeatability r	**0.19**	**0.44**	**1.48**	**2.01**
Reproducibility standard deviation s_R	0.13	0.29	6.31	4.59
Variation coefficient of reproducibility	181 %	67 %	97 %	83 %
Reproducibility R	**0.36**	**0.80**	**17.66**	**12.86**
Ratio (r/R)	0.55	0.56	0.08	0.16

Abbreviations: C   *Cochtran's outlier, 1%, data eliminated*   g   *Grubbs outlier, 5%, data eliminated*

Key

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

X   laboratory
Y   grains in which the germ is discoloured, mottled grains [w/w %]
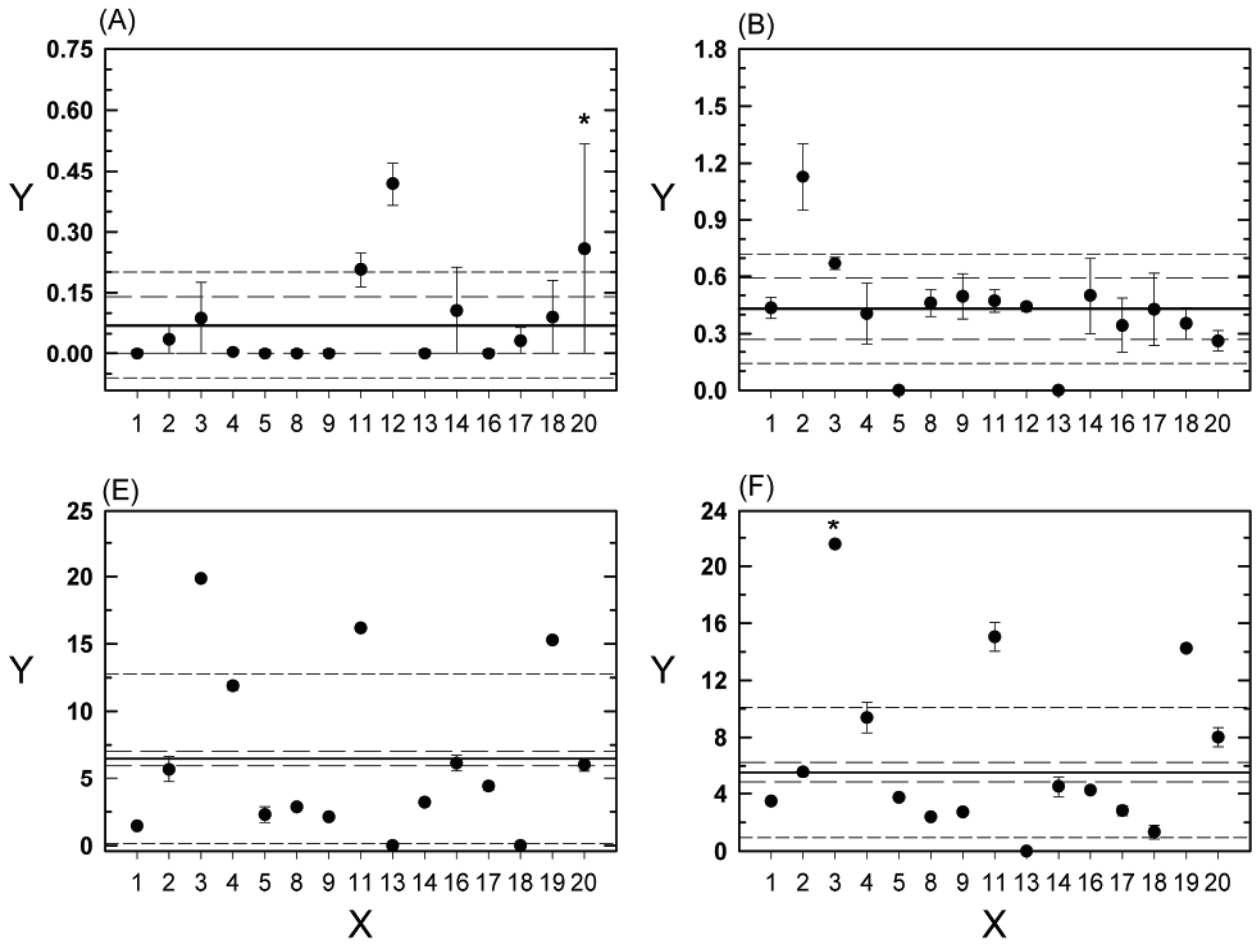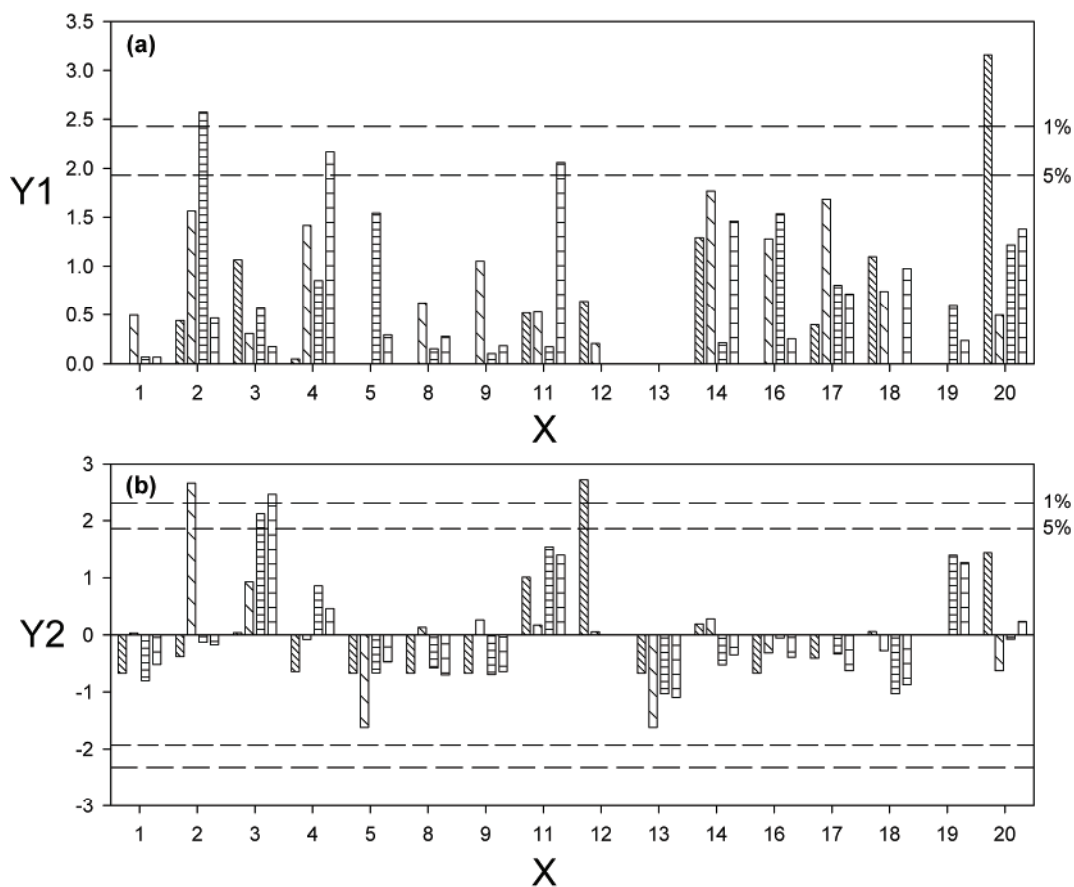
Figure 13 — Single results of the duplicate determination of "grains in which the germ is discoloured, mottled grains" including the overall mean, standard deviation of repeatability (s_r) and reproducibility (s_R) for the four test samples A, B, E and F

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 14 — Determination of "grains in which the germ is discoloured, mottled grains": (a) Mandel's within-laboratory statistic, k, and (b) Mandel's between laboratory consistency statistic, h, each grouped by laboratories

9 Results of the determination of "sprouted grains"

Table 6 — Collation of test results, outliers and precision data on the determination of "sprouted grains"

Determination of "sprouted grains"

Sample:	A	A	B	B	C	C	D	D	E	E	F	F	A	B	C	D	E	F
	Single results of the dupliacte determination (results given in [w/w%])												Mean of duplicate determination (results given in [w/w%])					
Lab.No																		
1	0.581	0.868	0.192	0.391	0.000	0.064	2.257	1.834	0.162	0.214	0.902	1.194	0.72	0.29	0.03	2.05	0.19	1.05
2	0.941	1.066	0.524	0.322	0.158	0.200	1.580	1.238	0.080	0.111	1.464	1.324	1.00	0.42	0.18	1.41	0.10	1.39
3	1.146	0.750	0.415	0.329	0.205	0.135	2.492	2.392	0.779	0.487	1.832	1.514	0.95	0.37	0.17	2.44	0.63	1.67
4	0.761	0.910	0.378	0.353	0.370	0.189	2.013	2.823	0.485	0.673	1.454	2.023	0.84	0.37	0.28	2.42	0.58	1.74
5	1.389	0.902	0.115	0.248	0.000	0.000	1.938	1.849	0.000	0.527	0.764	0.597	1.15	0.18	0.00	1.89	0.26	0.68
8	0.751	1.135	0.689	0.446	0.000	0.000	2.371	1.916	0.000	0.000	0.511	0.781	0.94	0.57	0.00	2.14	0.00	0.65
9	0.986	0.775	0.195	0.549	0.192	0.108	1.960	1.828	0.151	0.303	0.637	0.827	0.88	0.37	0.15	1.89	0.23	0.73
11	0.762	1.078	0.338	0.401	0.200	0.523	2.342	1.591	0.987	1.093	0.834	0.766	0.92	0.37	0.36	1.97	1.04	0.80
12	0.598	0.424	0.328	0.396	0.052	0.049	1.832	1.931	--	--	--	--	0.51	0.36	0.05	1.88	--	--
13	0.669	0.669	0.523	0.491	0.121	0.426	2.082	1.515	0.000	0.249	0.240	0.339	0.67	0.51	0.27	1.80	0.12	0.29
14	1.126	1.646	0.673	0.316	0.076	0.104	1.720	1.923	0.000	0.000	0.854	1.154	1.39	0.49	0.09	1.82	0.00	1.00
16	1.748	0.770	0.551	0.855	0.138	0.296	2.827	2.095	0.839	0.457	1.100	0.826	*1.26* K	0.70	0.22	2.46	0.65	0.96
17	0.801	0.898	0.231	0.238	0.000	0.032	1.698	1.774	0.230	0.603	0.063	0.141	0.85	0.23	0.02	1.74	0.42	0.10
18	1.325	1.061	0.442	0.430	0.050	0.000	1.973	2.361	0.550	0.283	0.371	0.740	1.19	0.44	0.03	2.17	0.42	0.56
19	--	--	--	--	--	--	--	--	0.307	0.280	0.078	0.579	--	--	--	--	0.29	0.33
20	0.883	0.939	0.593	0.647	0.123	0.300	2.151	1.706	0.000	0.178	0.610	0.423	0.91	0.62	0.21	1.93	0.09	0.52

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	**0.92**	**0.42**	**0.14**	**2.00**	**0.33**	**0.83**
Number of laboratories	14	15	15	15	15	15
Repeatability standard deviation s_r	0.21	0.13	0.10	0.32	0.17	0.21
Variation coefficient of repeatability	22 %	31 %	74 %	16 %	51 %	25 %
Repeatability r	**0.58**	**0.37**	**0.28**	**0.89**	**0.48**	**0.58**
Reproducibility standard deviation s_R	0.26	0.17	0.14	0.37	0.32	0.50
Variation coefficient of reproducibility	29 %	40 %	100 %	18 %	94 %	61 %
Reproducibility R	**0.74**	**0.47**	**0.38**	**1.02**	**0.88**	**1.41**
Ratio (r/R)	0.78	0.78	0.74	0.87	0.55	0.41

Abbreviations:

Abbreviations: K   *Mandel's statistic,k, outlier, 1%, data eliminated*

s_R
$y = 0.21 + 0.10 x$
$r^2 = 0.259$

s_r
$y = 0.11 + 0.11 x$
$r^2 = 0.937$

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

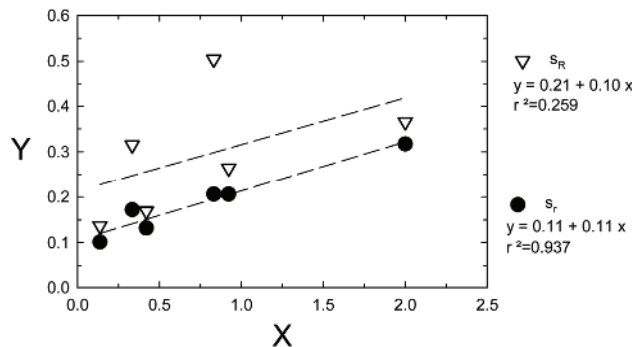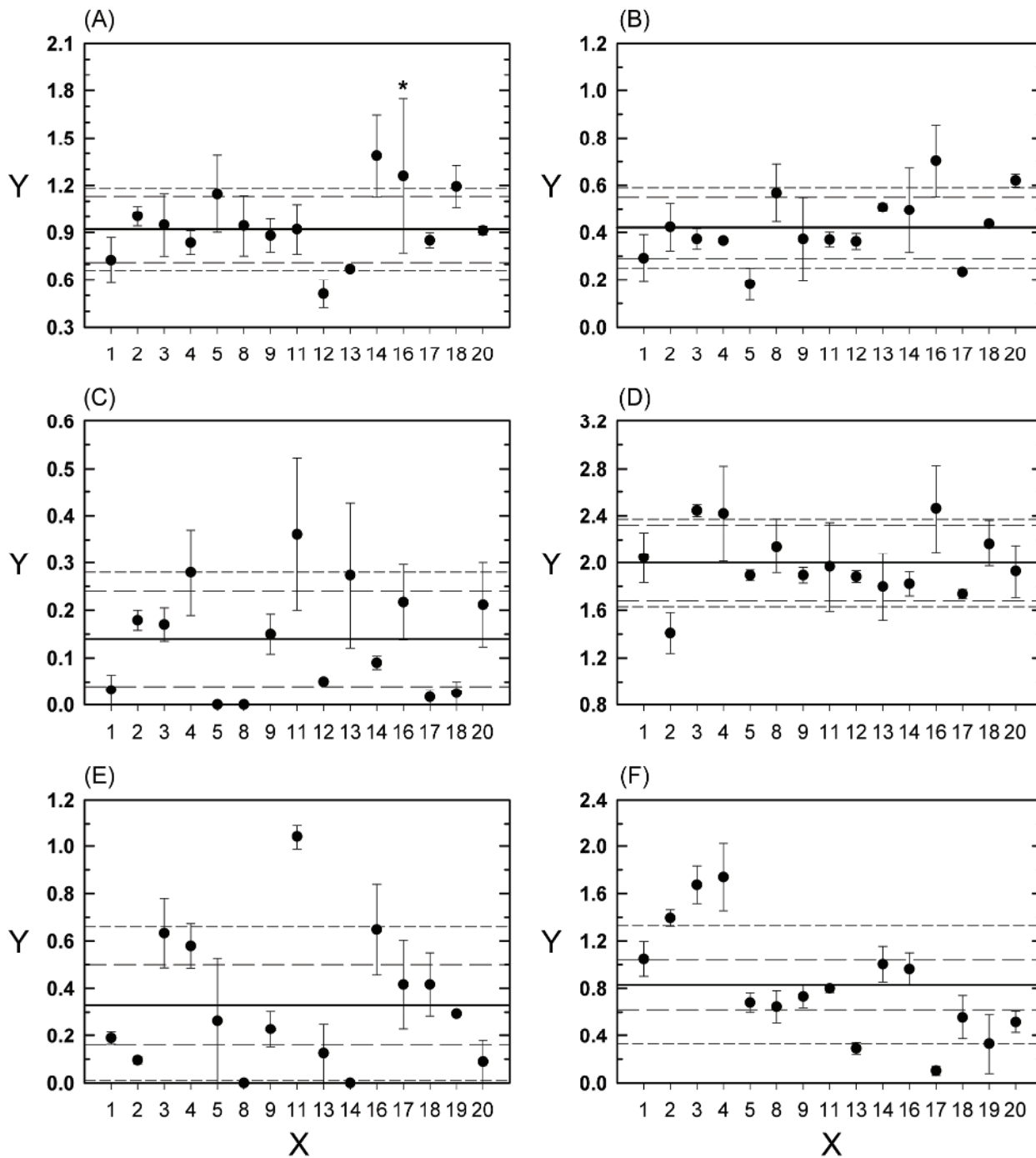**Figure 15 — Relationship between the standard deviation (s_r, s_R) and the mean value of "sprouted grains"**

Key

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

X   laboratory
Y   sprouted grains [w/w %]

Figure 16 — Single results of the duplicate determination of "sprouted grains" including the overall mean, standard deviation of repeatability (s_r) and reproducibility (s_R) for the six test samples A to F

Key
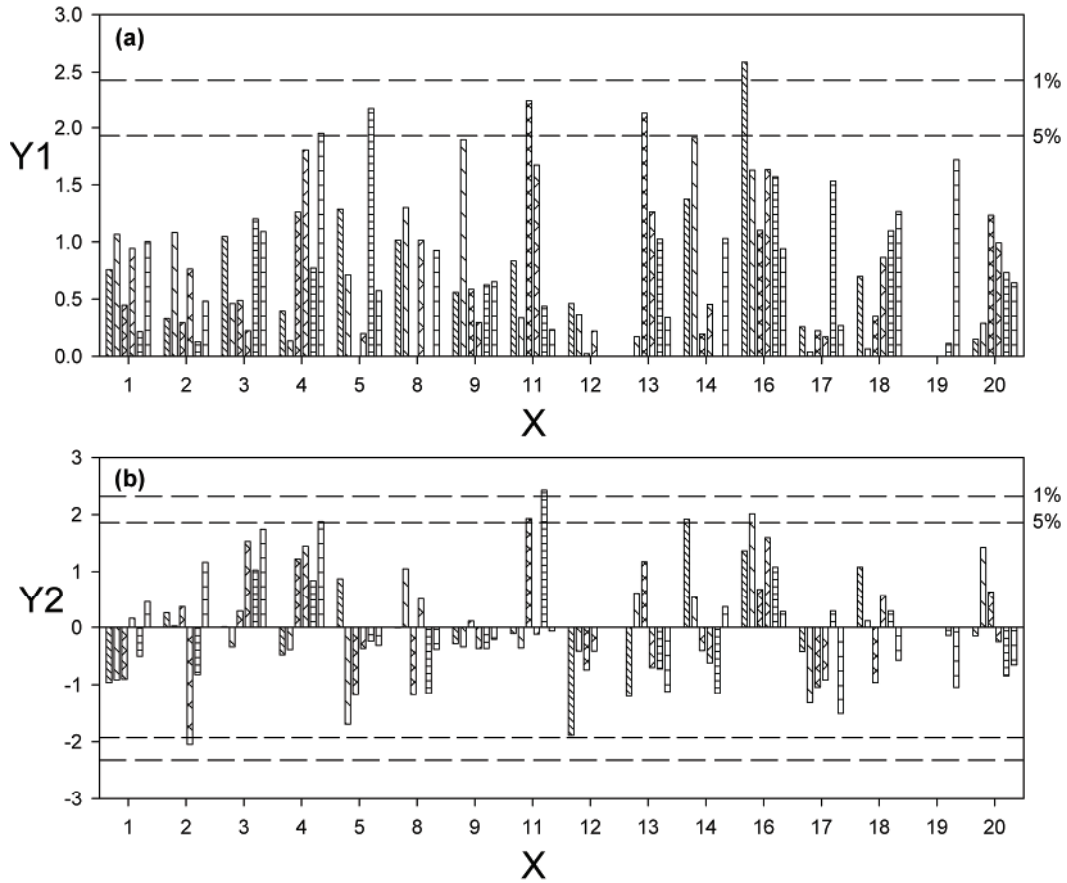
	A (wheat)
	B (wheat)
	C (rye)
	D (rye)
	E (durum)
	F (durum)

X   laboratory

Y1   Mandel's statistic, *k*

Y2   Mandel's statistic, *h*

Figure 17 — Determination of "sprouted grains": (a) Mandel's within-laboratory statistic, *k*, and (b) Mandel's between laboratory consistency statistic, *h*, each grouped by laboratories

10 Results of the determination of "extraneous seeds"

Table 7 — Collation of test results, outliers and precision data on the determination of "extraneous seeds"

Determination of "extraneous seeds"

Sample:	A	A	B	B	C	C	D	D	E	E	F	F	A	B	C	D	E	F
Lab.No																		
1	0.630	0.884	0.048	0.074	1.490	2.232	0.522	0.575	1.284	1.438	0.085	0.177	0.76	0.06	*1.86* K	0.55	1.36	0.13
2	0.758	0.735	0.085	0.038	1.487	1.424	0.403	0.324	1.301	0.887	0.118	0.093	0.75	0.06	1.46	0.36	1.09	0.11
3	0.929	0.951	0.047	0.090	1.666	1.820	0.627	0.551	1.388	1.314	0.196	0.177	0.94	0.07	1.74	0.59	1.35	0.19
4	0.969	0.611	0.088	0.085	1.868	1.914	0.651	0.504	1.446	1.488	0.164	0.133	0.79	0.09	1.89	0.58	1.47	0.15
5	0.773	0.908	0.140	0.103	1.512	1.503	0.541	0.647	1.404	1.572	0.201	0.271	0.84	0.12	1.51	0.59	1.49	0.24
8	0.877	1.068	0.147	0.068	1.552	1.789	0.360	0.514	1.557	1.344	0.139	0.179	0.97	*0.11* k	1.67	0.44	1.45	0.16
9	0.793	0.859	0.050	0.074	1.547	1.547	0.403	0.472	1.187	1.277	0.220	0.209	0.83	0.06	1.74	0.44	1.23	0.21
11	0.956	0.874	0.090	0.093	1.823	1.890	0.522	0.450	1.437	1.181	0.264	0.164	0.92	0.09	1.86	0.49	1.31	0.21
12	0.822	0.869	0.127	0.085	1.876	2.076	0.414	0.707	--	--	--	--	0.85	0.11	1.98	0.56	--	--
13	0.799	0.799	0.112	0.112	1.869	1.602	0.361	0.236	0.307	0.713	0.328	0.329	0.80	0.11	1.74	0.30	0.51	0.33
14	0.358	0.327	0.010	0.010	1.333	1.000	0.179	0.426	0.587	0.922	0.021	0.094	0.34	0.01	1.17	0.30	0.75	0.06
16	0.704	0.804	0.074	0.125	1.630	1.074	0.226	0.449	0.495	0.801	0.248	0.191	0.75	0.10	1.35	0.34	0.65	0.22
17	0.657	0.735	0.077	0.048	1.412	1.587	0.513	0.231	0.845	0.826	0.079	0.079	0.70	0.06	1.50	0.37	0.84	0.08
18	0.894	0.815	0.083	0.086	1.468	1.418	0.327	0.355	1.318	1.323	0.129	0.199	0.85	0.08	1.44	0.34	1.32	0.16
19	--	--	--	--	--	--	--	--	1.007	1.416	0.141	0.125	--	--	--	--	1.21	0.13
20	0.546	0.763	0.052	0.086	1.287	1.276	0.369	0.460	1.581	1.469	0.235	0.204	0.65	0.07	1.28	0.41	1.53	0.22

(Single results of the dupliacte determination (results given in [w/w%]) — columns A through F; Mean of duplicate determination (results given in [w/w%]) — columns A through F)

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	**0.78**	**0.08**	**1.59**	**0.44**	**1.17**	**0.17**
Number of laboratories	15	14	14	15	15	15
Repeatability standard deviation s_r	0.11	0.02	0.17	0.11	0.17	0.04
Variation coefficient of repeatability	13 %	28 %	11 %	25 %	15 %	22 %
Repeatability r	**0.29**	**0.06**	**0.47**	**0.32**	**0.49**	**0.10**
Reproducibility standard deviation s_R	0.17	0.03	0.27	0.13	0.35	0.07
Variation coefficient of reproducibility	21 %	41 %	17 %	30 %	30 %	43 %
Reproducibility R	**0.47**	**0.09**	**0.76**	**0.38**	**0.98**	**0.21**
Ratio (r/R)	0.63	0.67	0.62	0.84	0.49	0.50

Abbreviations: K   *Mandel's statistic,k, outlier, 1%, data eliminated*

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

Key
X   mean value of "extraneous seeds" [w/w %]
Y   standard deviation [w/w %]

Figure 18 — Relationship between the standard deviation (s_r, s_R) and the mean value of "extraneous seeds"

Key

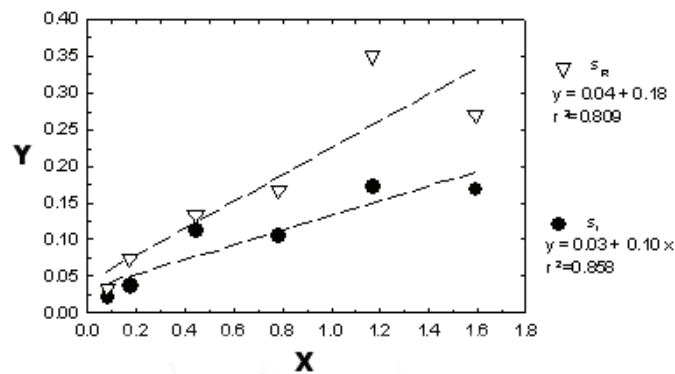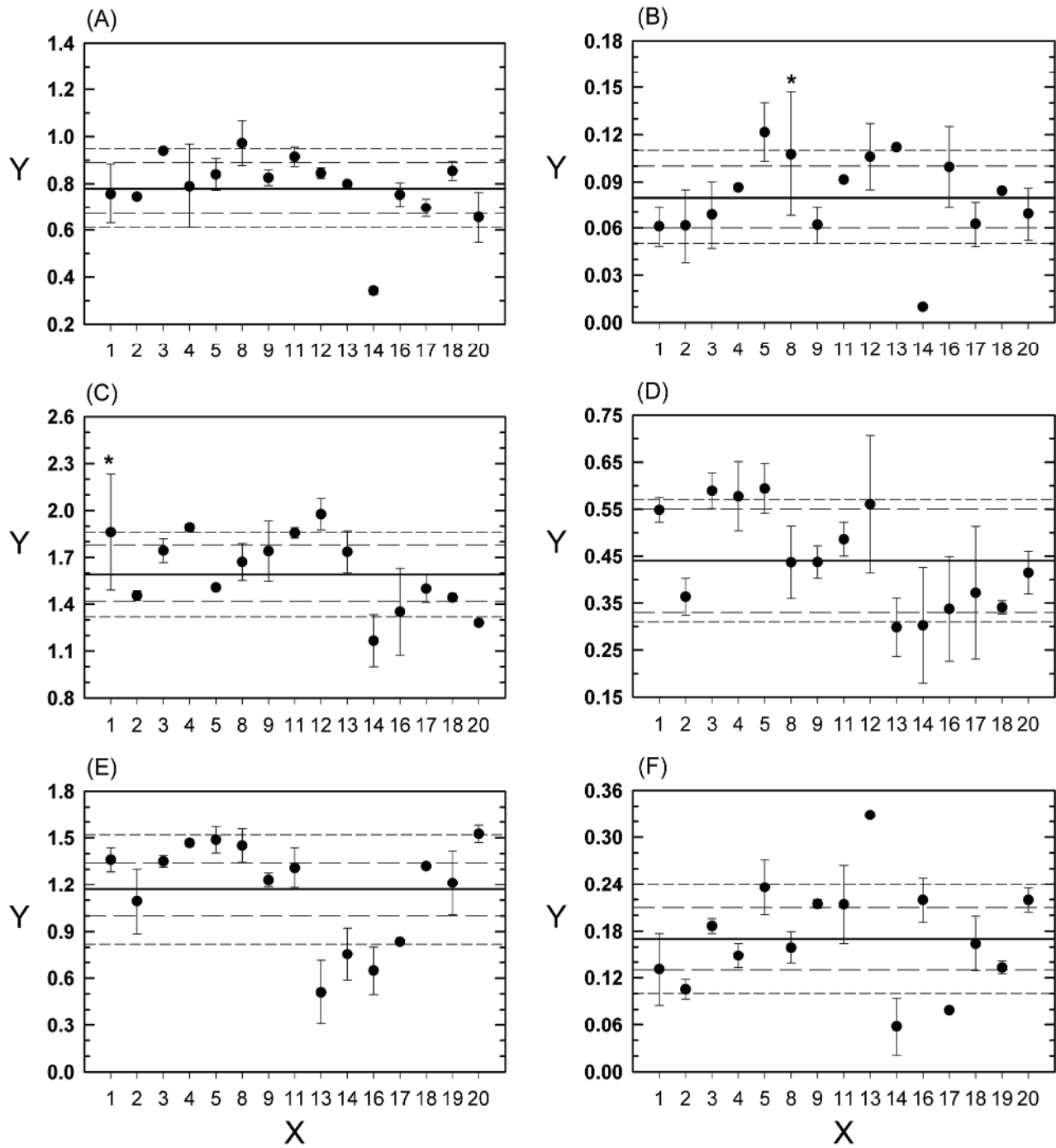―――― mean   ― ― s_r ‒ ‒ ‒ ‒ s_R (values without eliminated outliers (*))

X   laboratory
Y   extraneous seeds [w/w %]

Figure 19 — Single results of the duplicate determination of "extraneous seeds" including the overall mean, standard deviation of repeatability (s_r) and reproducibility (s_R) for the six test samples A to F

Key
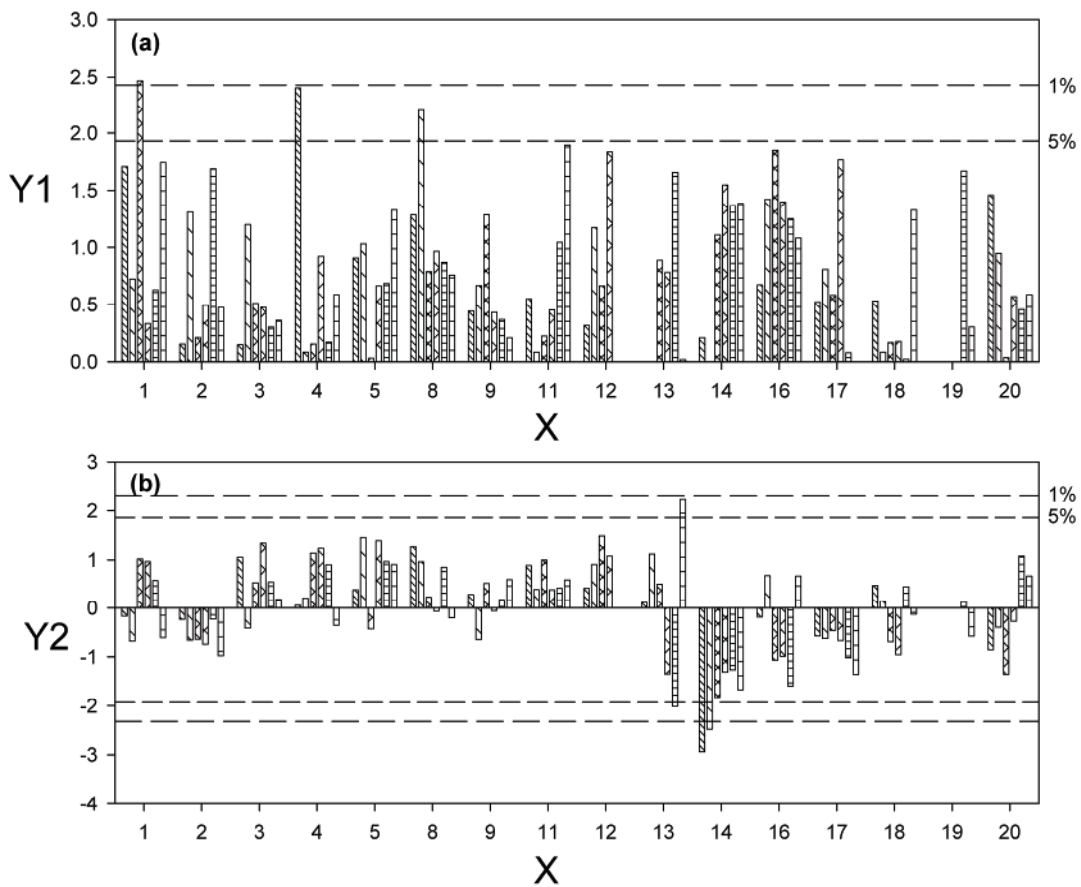
▨	A (wheat)
▨	B (wheat)
▨	C (rye)
▨	D (rye)
▤	E (durum)
▤	F (durum)

X   laboratory

Y1   Mandel's statistic, *k*

Y2   Mandel's statistic, *h*

Figure 20 — Determination of "extraneous seeds": (a) Mandel's within-laboratory statistic, *k*, and (b) Mandel's between laboratory consistency statistic, *h*, each grouped by laboratories

11 Results of the determination of "unsound grains"

Table 8 — Collation of test results, outliers and precision data on the determination of "unsound grains"

Determination of "unsound grains"

Sample:	\multicolumn{12}{c}{Single results of the dupliacte determination (results given in [w/w%])}	\multicolumn{6}{c}{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	0.207	0.191	0.980	0.571	0.661	0.485	0.957	1.193	0.318	0.174	1.381	0.999	0.20	0.78	0.57	1.08	0.25	1.19
2	0.246	0.071	0.012	0.020	0.026	0.027	0.066	0.131	0.237	0.262	0.237	0.164	0.16	0.02	0.03	0.10	0.25	0.20
3	0.206	0.131	0.720	0.897	0.546	0.448	0.771	1.372	0.927	1.253	1.943	1.826	0.17	0.81	0.50	1.07	1.09	1.88
4	0.071	0.016	1.290	1.538	0.515	0.240	0.897	0.851	0.838	0.600	0.908	2.160	0.04	1.41	0.38	0.87	0.72	1.53
5	0.000	0.000	0.542	0.612	0.000	0.000	0.000	0.095	0.000	0.000	0.000	0.154	0.00	0.58	0.00	0.05	0.00	0.08
8	0.073	0.076	0.193	0.294	0.271	0.117	0.641	0.383	0.222	0.299	0.292	0.623	0.07	0.24	0.19	0.51	0.26	0.46
9	0.000	0.000	0.365	0.462	0.101	0.145	0.000	0.000	0.369	0.364	0.141	0.116	0.00	0.41	0.12	0.00	0.37	0.13
11	0.548	0.412	2.033	1.561	1.141	2.074	4.423	1.972	3.990	6.098	4.767	5.868	*0.48* g	1.80	*1.61* c	*3.20* C	*5.04* C	*5.32* G
12	0.108	0.051	0.806	0.594	0.110	0.195	0.271	0.174	--	--	--	--	0.08	0.70	0.15	0.22	--	--
13	0.049	0.049	0.702	0.679	0.000	0.000	0.094	0.207	0.000	0.000	0.000	0.000	0.05	0.69	0.00	0.15	0.00	0.00
14	0.000	0.000	0.107	1.423	0.451	0.408	1.853	1.214	1.308	0.954	0.991	1.041	0.00	*0.77* c	0.43	1.53	1.13	1.02
16	0.173	0.248	1.270	0.946	0.194	0.631	0.555	0.252	0.190	0.000	0.621	0.471	0.21	1.11	0.41	0.40	0.10	0.55
17	0.000	0.098	0.847	0.951	0.179	0.159	0.401	0.231	0.138	0.600	0.476	0.550	0.05	0.90	0.17	0.32	0.37	0.51
18	0.000	0.000	1.169	1.929	0.000	0.152	0.513	0.394	0.403	0.728	1.586	1.864	0.00	1.55	0.08	0.45	0.57	1.73
19	--	--	--	--	--	--	--	--	0.400	0.258	0.398	0.512	--	--	--	--	0.33	0.46
20	0.326	0.261	0.919	1.432	0.718	1.267	2.290	1.500	0.489	0.790	2.040	1.502	0.29	1.18	*0.99* g	1.90	0.64	1.77

Sample				Sample	A	B	C	D	E	F
A	=	common wheat		**Mean of test results**	**0.09**	**0.87**	**0.23**	**0.62**	**0.43**	**0.82**
B	=	common wheat		Number of laboratories	14	14	13	14	14	14
C	=	rye		Repeatability standard deviation s r	0.05	0.23	0.12	0.25	0.17	0.29
D	=	rye		Variation coefficient of repeatability	50 %	27 %	51 %	40 %	39 %	35 %
E	=	durum wheat		**Repeatability r**	**0.13**	**0.65**	**0.33**	**0.69**	**0.47**	**0.80**
F	=	durum wheat		Reproducibility standard deviation s R	0.10	0.53	0.22	0.61	0.38	0.71
				Variation coefficient of reproducibility	106 %	61 %	93 %	99 %	87 %	87 %
				Reproducibility R	**0.28**	**1.47**	**0.61**	**1.71**	**1.06**	**1.99**
				Ratio (r/R)	0.47	0.44	0.55	0.40	0.44	0.40

Abbreviations:
- C *Cochtran's outlier, 1%, data eliminated*
- c *Cochrans outlier, 5%, data eliminated*
- G *Grubbs outlier, 1%, data eliminated*
- g *Grubbs outlier, 5%, data eliminated*

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

Figure 21 — Relationship between the standard deviation (s_r, s_R) and the mean value of "unsound grains"
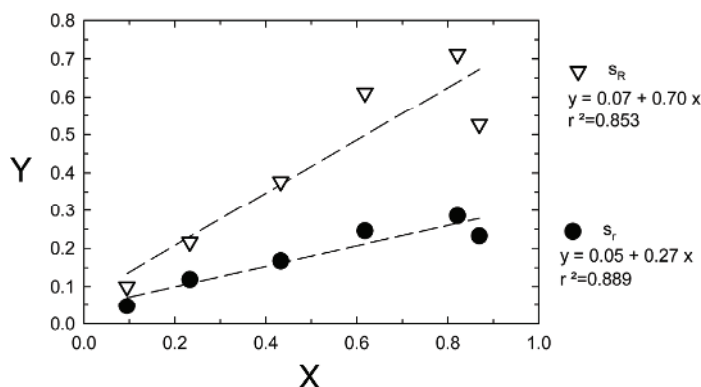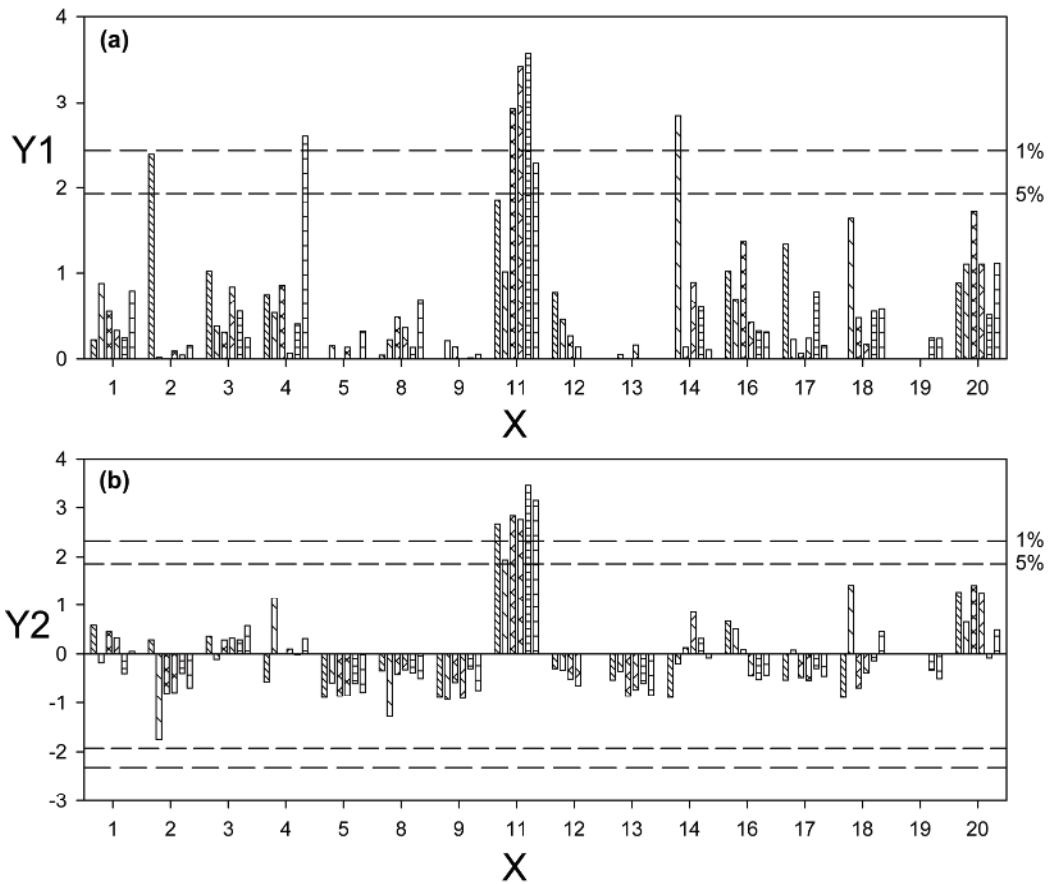
Key
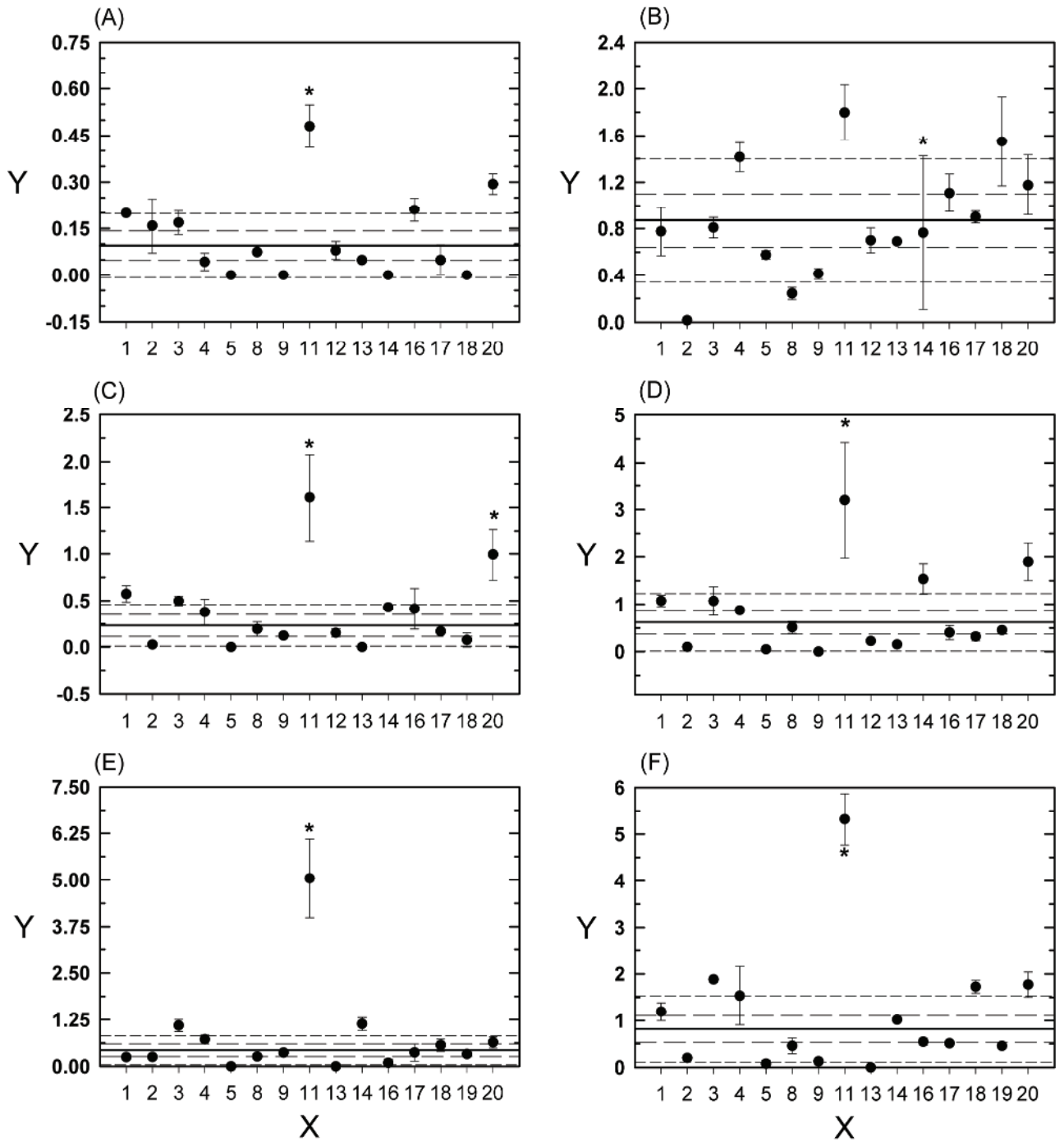
—— mean   — — s_r — — — — s_R (values without eliminated outliers (*))

X   laboratory
Y   unsound grains [w/w %]

Figure 22 — Single results of the duplicate determination of "unsound 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 23 — Determination of "unsound grains": (a) Mandel's within-laboratory statistic, *k*, and (b) Mandel's between laboratory consistency statistic, *h*, each grouped by laboratories

12 Results of the determination of "extraneous matter"

Table 9 — Collation of test results, outliers and precision data on the determination of "extraneous matter"

Determination of "extraneous matter"

Lab.No	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
1	0.557	0.581	1.287	1.309	1.412	1.359	4.473	4.166	1.532	1.475	0.253	0.281	0.57	1.30	1.39	4.32	1.50	0.27
2	0.499	0.478	1.103	1.092	1.573	1.670	3.863	3.803	1.562	1.591	0.260	0.225	0.49	1.10	1.62	3.83	1.58	0.24
3	0.592	0.598	0.823	0.829	2.025	2.071	4.475	4.446	1.598	1.647	0.178	0.162	0.60	0.83	2.05	4.46	1.62	0.17
4	0.426	0.455	1.054	1.080	2.091	2.178	3.747	3.483	1.711	1.570	0.211	0.273	0.44	1.07	2.13	3.62	1.64	0.24
5	0.627	0.627	1.107	1.106	1.735	1.735	4.279	4.279	1.900	1.900	0.310	0.310	0.63	1.11	1.73	4.28	1.90	0.31
8	0.521	0.511	0.950	0.930	1.970	1.912	3.689	3.551	2.115	1.989	0.225	0.232	0.52	0.94	1.94	3.62	2.05	0.23
9	0.459	0.492	2.072	1.994	2.118	2.048	4.015	3.970	1.780	1.759	0.176	0.170	0.48	_2.03_ G	2.08	3.99	1.77	0.17
11	1.049	1.054	1.393	1.376	3.017	2.831	4.883	4.892	2.547	2.063	0.367	0.341	_1.05_ g	1.38	2.92	4.89	_2.30_ C	0.35
12	0.683	0.614	0.993	1.008	1.977	1.946	3.312	3.355	--	--	--	--	0.65	1.00	1.96	3.33	--	--
13	0.545	0.589	0.857	0.781	2.750	3.130	1.689	2.284	0.648	1.445	1.775	1.387	0.57	0.82	_2.94_ K	_1.99_ c	_1.05_ C	_1.58_ g
14	0.249	0.268	0.509	1.088	2.424	2.129	3.032	2.965	0.876	0.652	0.124	0.097	0.26	_0.80_ C	2.28	3.00	0.76	0.11
16	0.460	0.242	1.038	2.329	2.496	1.737	1.848	0.736	0.947	1.021	0.547		_0.35_ C	1.20	2.41	_1.79_ g	0.84	0.78
17	0.700	0.717	0.940	0.918	2.564	2.514	4.491	4.477	1.965	1.959	0.456	0.751	0.71	0.93	2.54	4.48	1.96	0.60
18	0.532	0.535	1.375	1.399	2.313	2.273	3.518	3.761	1.954	1.976	0.223	0.219	0.53	1.39	2.29	3.64	1.97	0.22
19	--	--	--	--	--	--	--	--	1.809	1.786	1.157	1.345	--	--	--	--	1.80	_1.25_ G
20	0.734	0.699	1.005	0.984	2.363	2.383	4.141	4.224	2.085	2.070	0.244	0.222	0.72	0.99	2.37	4.18	2.08	0.23

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	**0.55**	**1.08**	**2.12**	**3.97**	**1.65**	**0.30**
Number of laboratories	13	13	14	13	13	13
Repeatability standard deviation s_r	0.02	0.07	0.08	0.10	0.07	0.11
Variation coefficient of repeatability	4 %	6 %	4 %	3 %	4 %	37 %
Repeatability r	**0.06**	**0.19**	**0.23**	**0.28**	**0.20**	**0.31**
Reproducibility standard deviation s_R	0.12	0.20	0.40	0.53	0.42	0.20
Variation coefficient of reproducibility	22 %	18 %	19 %	13 %	26 %	67 %
Reproducibility R	**0.34**	**0.55**	**1.12**	**1.49**	**1.18**	**0.57**
Ratio (r/R)	0.17	0.34	0.20	0.19	0.17	0.55

Abbreviations:
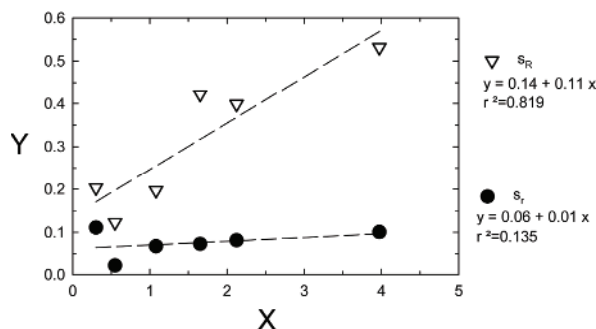
C	_Cochtran's outlier, 1%, data eliminated_	G _Grubbs outlier, 1%, data eliminated_
c	_Cochrans outlier, 5%, data eliminated_	g _Grubbs outlier, 5%, data eliminated_
K	_Mandel's statistic,k, outlier, 1%, data eliminated_	

▽ s_R
y = 0.14 + 0.11 x
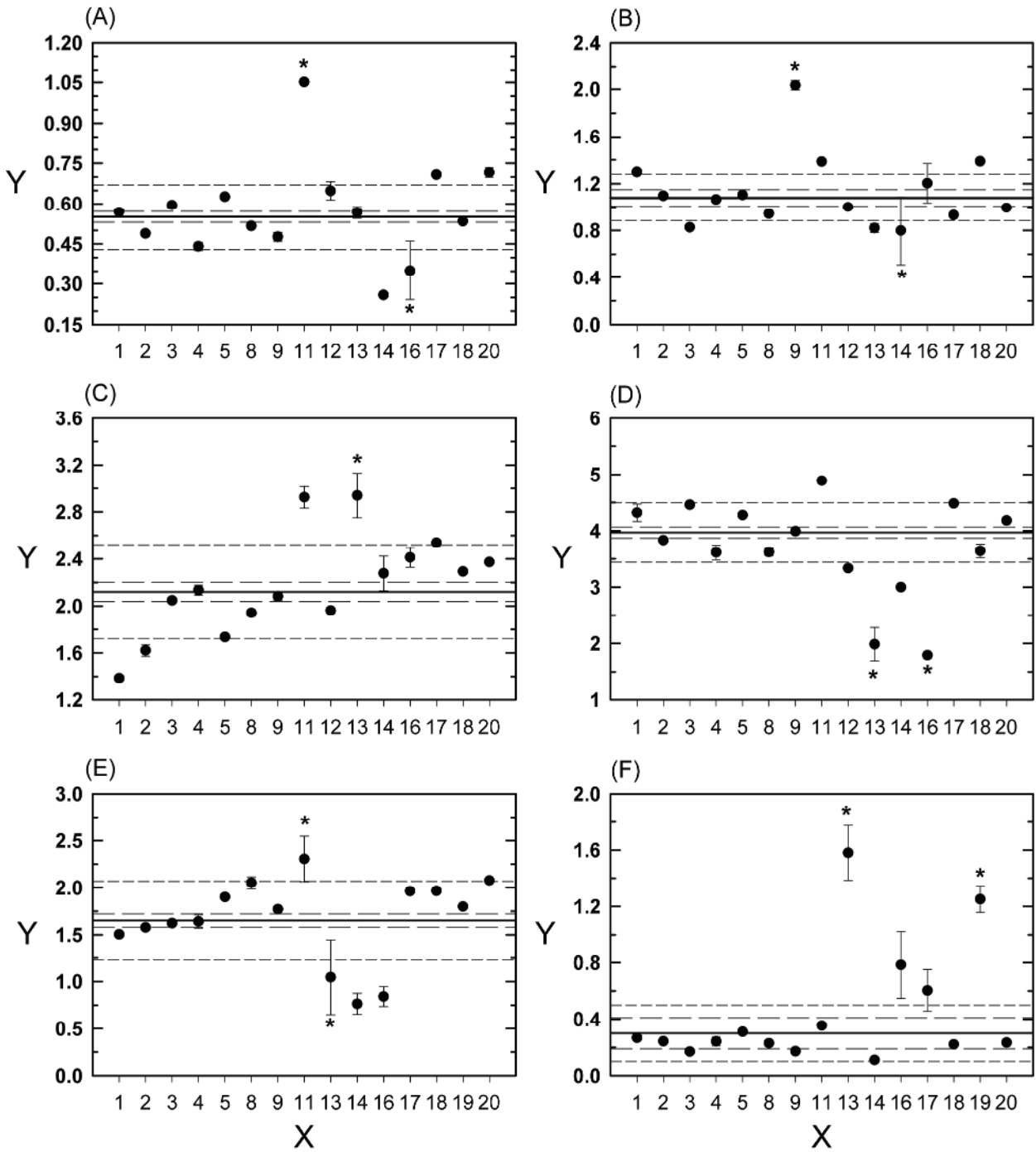r^2=0.819

● s_r
y = 0.06 + 0.01 x
r^2=0.135

Key
X   mean value of "extraneous matter" [w/w %]
Y   standard deviation [w/w %]

Figure 24 — Relationship between the standard deviation (s_r, s_R) and the mean value of "extraneous matter"
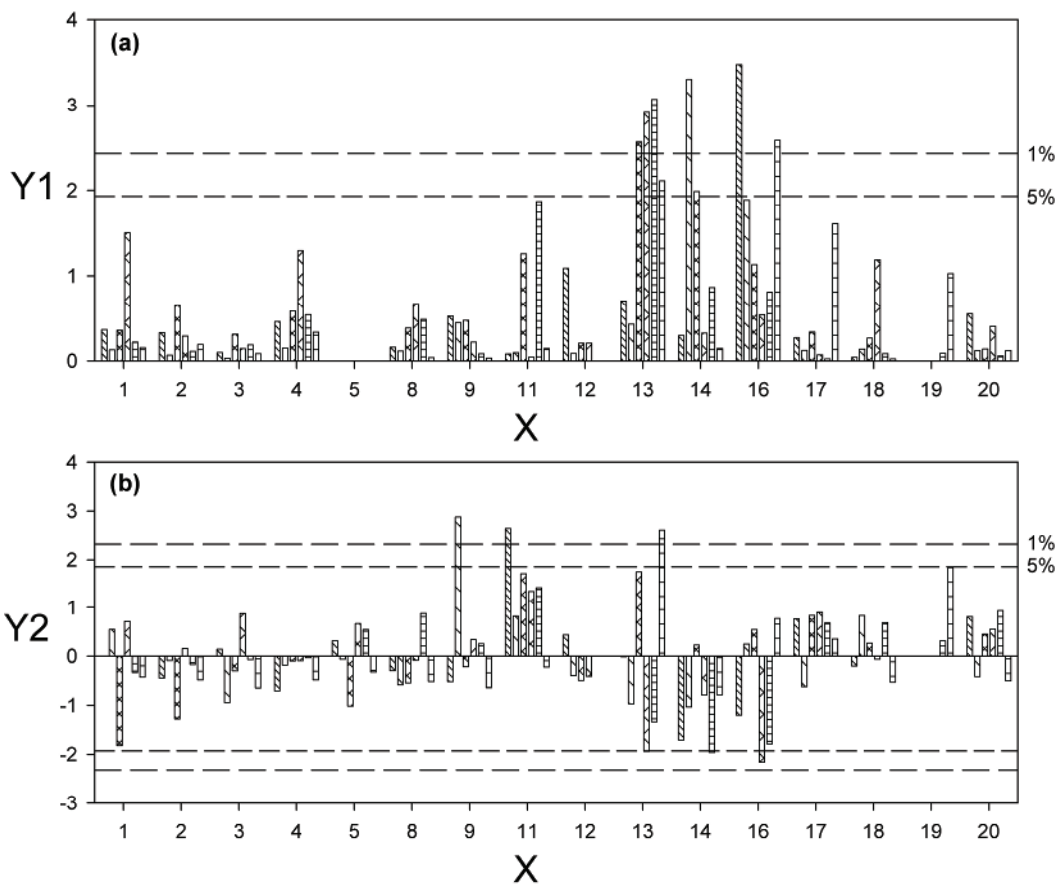
Key

———— mean   — — sr   ———— sR (values without eliminated outliers (*))

X   laboratory
Y   extraneous matter [w/w %]

Figure 25 — Single results of the duplicate determination of "extraneous matter" including the overall mean, standard deviation of repeatability (sr) and reproducibility (sR) 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 26 — Determination of "extraneous matter": (a) Mandel's within-laboratory statistic, *k*, and (b) Mandel's between laboratory consistency statistic, *h*, each grouped by laboratories

13 Results of the determination of "husks"

Table 10 — Collation of test results, outliers and precision data on the determination of "husks"

Determination of "husks"

Sample:	A	A	B	B	C	C	D	D	E	E	F	F	A	B	C	D	E	F
Lab.No					Single results of the dupliacte determination (results given in [w/w%])								Mean of duplicate determination (results given in [w/w%])					
1	0.162	0.177	0.174	0.222	0.195	0.136	0.080	0.080	0.151	0.122	0.231	0.476	0.17	0.20	0.17	0.08	0.14	0.35
2	0.221	0.064	0.269	0.226	0.143	0.172	0.174	0.041	0.240	0.092	0.407	0.406	_0.14_ C	0.25	0.16	0.11	0.17	0.41
3	0.076	0.084	0.280	0.164	0.247	0.140	0.091	0.128	0.061	0.108	0.164	0.210	0.08	0.22	0.19	0.11	0.08	0.19
4	0.182	0.154	0.184	0.216	0.072	0.138	0.178	0.303	0.171	0.110	0.548	0.449	0.17	0.20	0.11	_0.24_ g	0.14	0.50
5	0.088	0.102	0.171	0.077	0.378	0.384	0.127	0.163	0.150	0.089	0.230	0.739	0.10	0.12	0.38	0.15	0.12	_0.48_ C
8	0.123	0.173	0.160	0.128	0.196	0.184	0.112	0.111	0.134	0.174	0.442	0.320	0.15	0.14	0.19	0.11	0.15	0.38
9	0.068	0.097	0.070	0.127	0.253	0.135	0.111	0.051	0.114	0.094	0.383	0.403	0.08	0.10	0.19	0.08	0.10	0.39
11	0.060	0.097	0.148	0.162	0.209	0.348	0.195	0.174	0.149	0.144	0.543	0.455	0.08	0.16	0.28	0.18	0.15	0.50
12	0.152	0.126	0.116	0.117	0.275	0.330	0.141	0.119	--	--	--	--	0.14	0.12	0.30	0.13	--	--
13	0.050	0.050	0.109	0.062	0.227	0.153	0.054	0.052	0.006	0.005	0.034	0.021	0.05	0.09	0.19	0.05	0.01	0.03
14	0.181	0.212	0.165	0.387	0.160	0.222	0.101	0.091	0.163	0.077	0.578	0.547	0.20	_0.28_ c	0.19	0.11	0.12	0.56
16	0.009	0.014	0.081	0.010	0.019	0.051	0.058	0.028	0.026	0.001	0.062	0.057	0.01	0.05	0.04	0.04	0.01	0.06
17	0.056	0.082	0.077	0.095	0.081	0.143	0.080	0.062	0.031	0.030	0.238	0.283	0.07	0.09	0.11	0.07	0.03	0.26
18	0.143	0.136	0.110	1.462	0.204	0.073	0.068	0.105	0.014	0.071	0.275	0.370	0.14	_0.79_ C	0.14	0.09	0.04	0.32
19	--	--	--	--	--	--	--	--	0.084	0.051	0.384	0.255	--	--	--	--	0.07	0.32
20	0.075	0.099	0.111	0.174	0.150	0.220	0.087	0.165	1.502	0.057	0.337	0.282	0.09	0.14	0.19	0.13	_0.78_ C	0.31

| Sample					A	B	C	D	E	F
A	=	common wheat		**Mean of test results**	**0.11**	**0.14**	**0.19**	**0.10**	**0.10**	**0.33**
B	=	common wheat		Number of laboratories	14	13	15	14	14	14
C	=	rye		Repeatability standard deviation s $_r$	0.02	0.04	0.06	0.03	0.04	0.07
D	=	rye		Variation coefficient of repeatability	17 %	29 %	30 %	34 %	43 %	21 %
E	=	durum wheat		**Repeatability r**	**0.05**	**0.11**	**0.16**	**0.10**	**0.11**	**0.19**
F	=	durum wheat		Reproducibility standard deviation s $_R$	0.05	0.07	0.09	0.04	0.06	0.16
				Variation coefficient of reproducibility	50 %	46 %	49 %	44 %	65 %	50 %
				Reproducibility R	**0.15**	**0.19**	**0.26**	**0.13**	**0.17**	**0.46**
				Ratio (r/R)	0.33	0.62	0.60	0.78	0.66	0.41

Abbreviations: C _Cochtran's outlier, 1%, data eliminated_   g _Grubbs outlier, 5%, data eliminated_

c _Cochrans outlier, 5%, data eliminated_
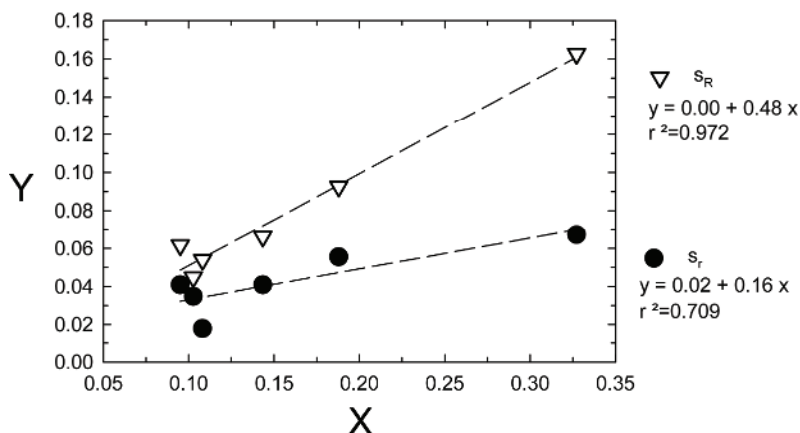
Key
X   mean value of "husks" [w/w %]
Y   standard deviation [w/w %]

Figure 27 — Relationship between the standard deviation (s_r, s_R) and the mean value of "husks"
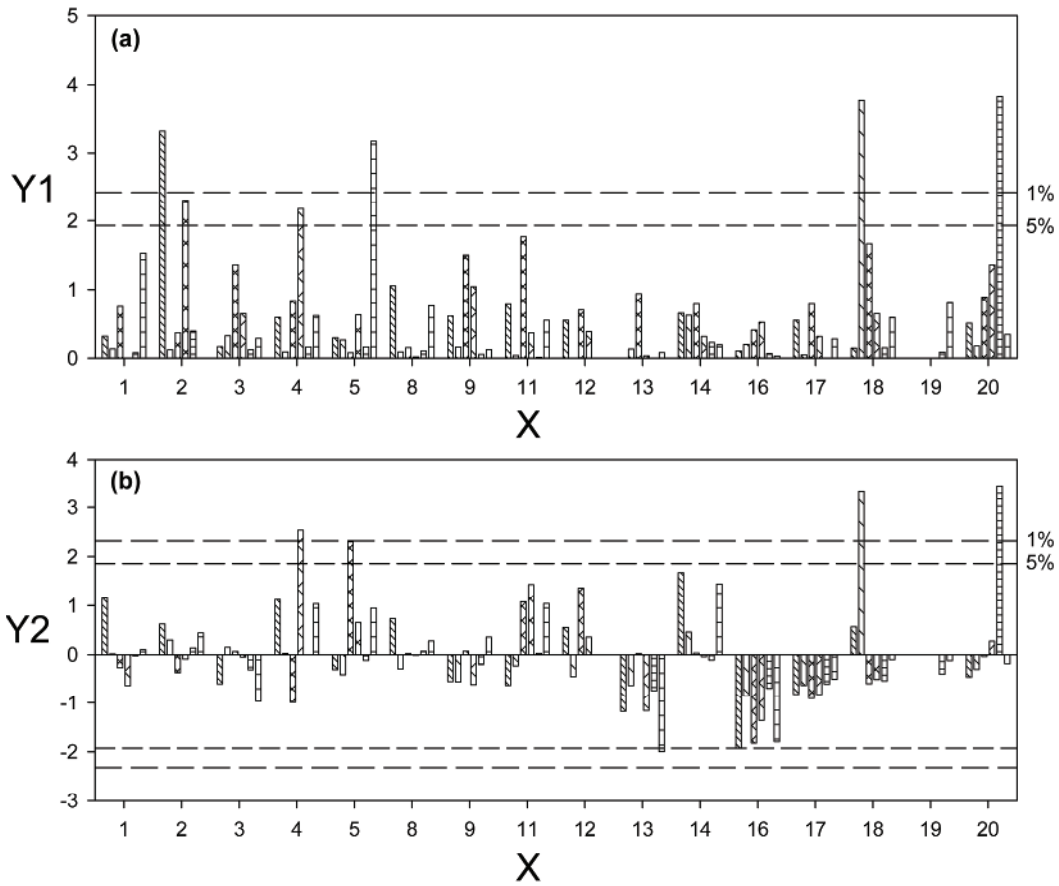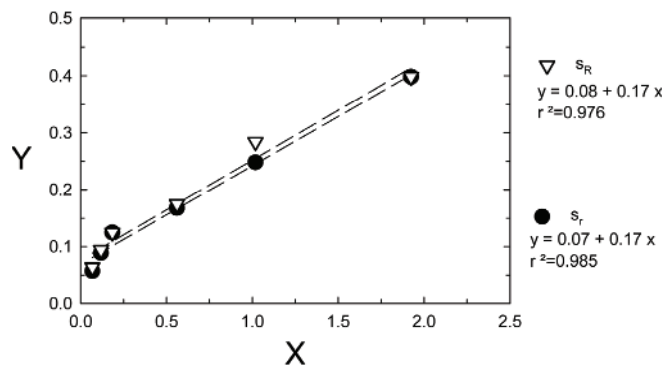
Key
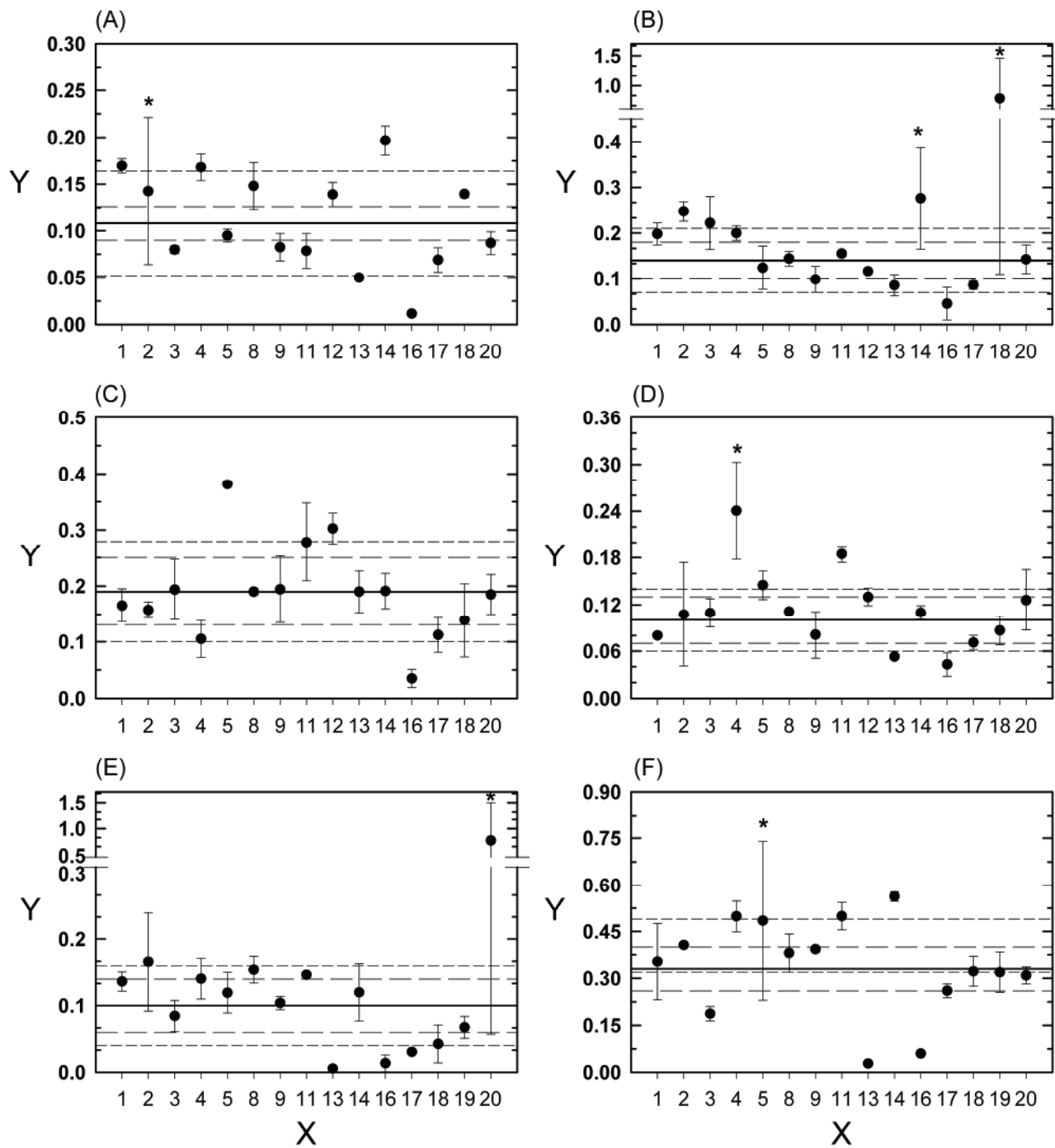
——— mean　　— — s_r　　— — — — s_R (values without eliminated outliers (*))

X　laboratory
Y　husks [w/w %]

Figure 28 — Single results of the duplicate determination of "husks" 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 29 — Determination of "husks": (a) Mandel's within-laboratory statistic, *k*, and (b) Mandel's between laboratory consistency statistic, *h*, each grouped by laboratories

14 Results of the determination of "ergot"

Table 11 — Collation of test results, outliers and precision data on the determination of "ergot"

Determination of "ergot"

Sample:	A	A	B	B	C	C	D	D	E	E	F	F	A	B	C	D	E	F
	over Single results of the dupliacte determination (results given in [w/w%])												Mean of duplicate determination (results given in [w/w%])					
Lab.No																		
1	0.628	0.314	0.025	0.000	0.328	0.047	1.398	1.869	0.152	0.055	0.947	1.038	0.47	0.01	0.19	1.63	0.10	0.99
2	0.608	0.452	0.040	0.058	0.346	0.257	1.537	1.676	0.190	0.018	1.123	0.562	0.53	0.05	0.30	1.61	0.10	0.84
3	0.624	0.625	0.041	0.000	0.080	0.328	2.056	2.140	0.167	0.177	1.025	1.107	0.62	0.02	0.20	2.10	0.17	1.07
4	0.536	0.492	0.222	0.101	0.360	0.241	1.967	1.793	0.190	0.022	0.558	1.725	0.51	0.16	0.30	1.88	0.11	*1.14* K
5	0.439	0.795	0.075	0.104	0.090	0.206	2.024	1.952	0.045	0.069	1.274	2.028	0.62	0.09	0.15	1.99	0.06	1.65
8	0.422	0.527	0.127	0.000	0.062	0.191	2.391	1.492	0.117	0.000	0.974	0.896	0.47	0.06	0.13	1.94	0.06	0.94
9	0.612	0.351	0.086	0.127	0.182	0.300	2.223	1.766	0.175	0.050	1.116	1.049	0.48	0.11	0.24	1.99	0.11	1.08
11	0.177	0.492	0.000	0.060	0.163	0.111	1.914	2.365	0.154	0.103	1.139	1.431	0.33	0.03	0.14	2.14	0.13	1.29
12	0.601	0.927	0.000	0.056	0.413	0.081	1.965	2.277	--	--	--	--	0.76	0.03	0.25	2.12	--	--
13	0.621	0.621	0.217	0.081	0.202	0.067	1.575	2.061	0.301	0.301	0.846	0.861	0.62	0.15	0.13	1.82	0.30	0.85
14	0.760	0.684	0.048	0.008	0.212	0.064	2.572	1.615	0.000	0.279	1.449	0.779	0.72	0.03	0.14	2.09	0.14	1.11
16	0.412	0.373	0.037	0.164	0.089	0.351	2.207	0.946	0.073	0.233	0.979	0.824	0.39	0.10	0.22	1.58	0.15	0.90
17	0.785	0.751	0.077	0.048	0.049	0.143	2.083	1.836	0.061	0.150	0.714	1.179	0.77	0.06	0.10	1.96	0.11	0.95
18	0.294	0.719	0.000	0.022	0.076	0.105	2.040	2.194	0.260	0.105	0.878	0.804	0.51	0.01	0.09	2.12	0.18	0.84
19	--	--	--	--	--	--	--	--	0.000	0.000	0.926	0.786	--	--	--	--	0.00	0.86
20	0.759	0.420	0.181	0.048	0.115	0.281	2.176	1.603	0.036	0.069	0.948	0.815	0.59	0.11	0.20	1.89	0.05	0.88

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	**0.56**	**0.07**	**0.18**	**1.92**	**0.12**	**1.02**
Number of laboratories	15	15	15	15	15	14
Repeatability standard deviation s_r	0.17	0.06	0.12	0.40	0.09	0.25
Variation coefficient of repeatability	30 %	83 %	67 %	21 %	75 %	24 %
Repeatability r	**0.47**	**0.16**	**0.35**	**1.11**	**0.25**	**0.70**
Reproducibility standard deviation s_R	0.17	0.06	0.12	0.40	0.09	0.28
Variation coefficient of reproducibility	31 %	93 %	67 %	21 %	79 %	28 %
Reproducibility R	**0.49**	**0.18**	**0.35**	**1.11**	**0.26**	**0.80**
Ratio (r/R)	0.96	0.90	1.00	1.00	0.94	0.87

Abbreviations: K   *Mandel's statistic,k, outlier, 1%, data eliminated*

Key

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

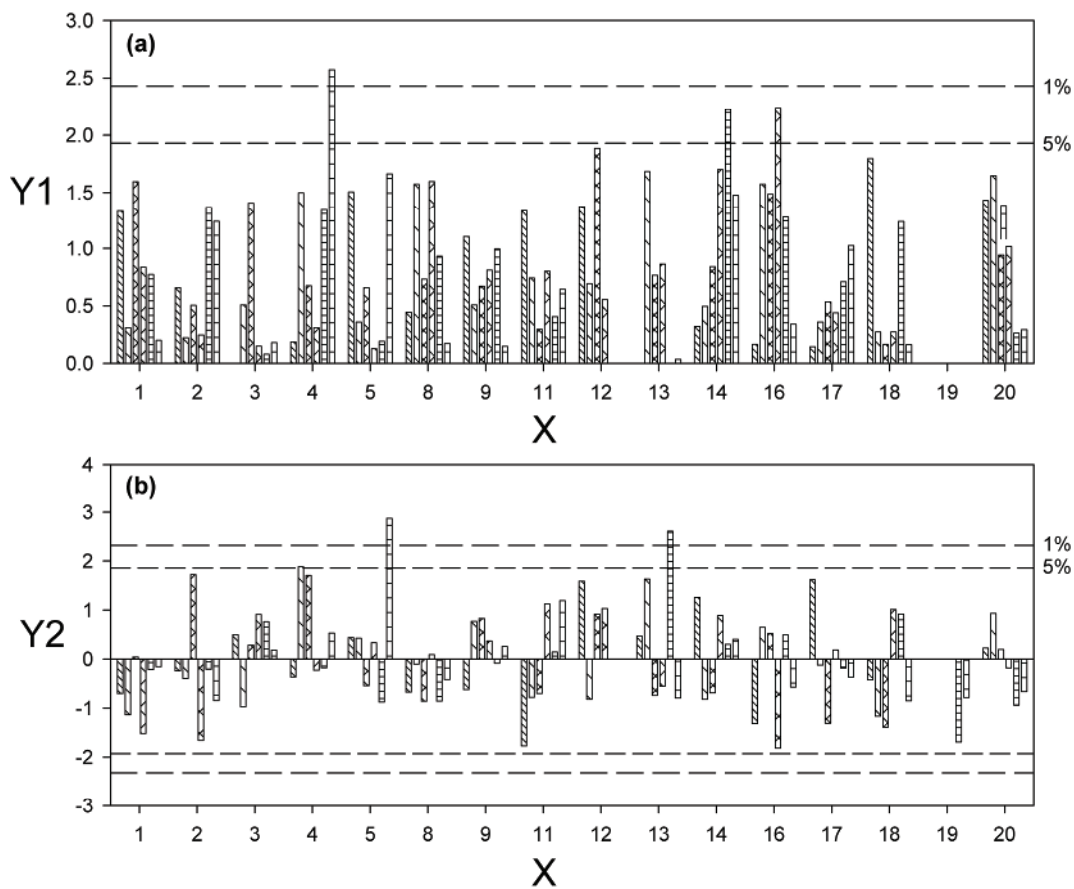**Figure 30 — Relationship between the standard deviation (s_r, s_R) and the mean value of "ergot"**

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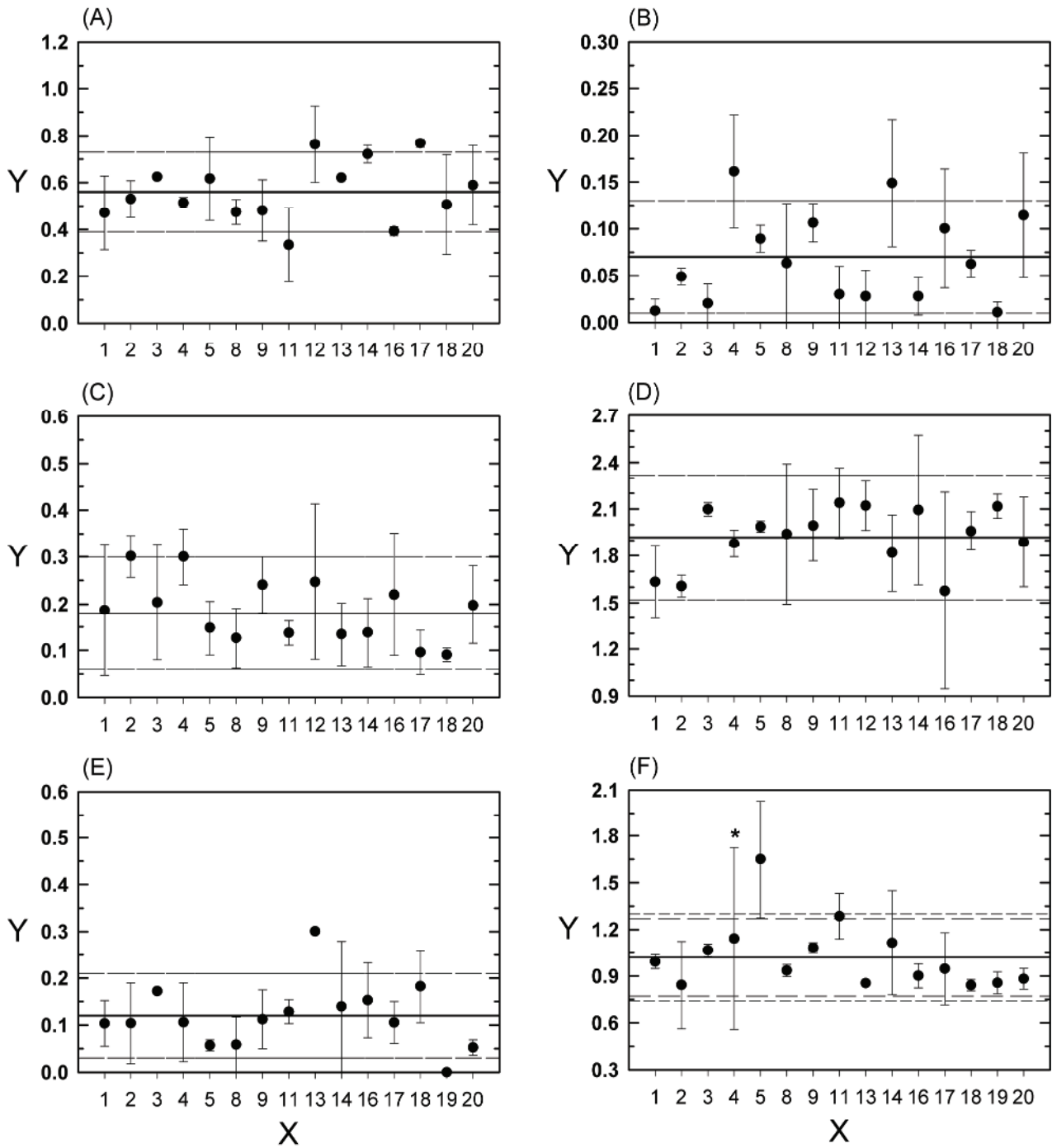
———— mean   — — s_r – – – – s_R (values without eliminated outliers (*))

X   laboratory
Y   ergot [w/w %]

Figure 31 — Single results of the duplicate determination of "ergot" 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 32 — Determination of "ergot": (a) Mandel's within-laboratory statistic, *k*, and (b) Mandel's between laboratory consistency statistic, *h*, each grouped by laboratories
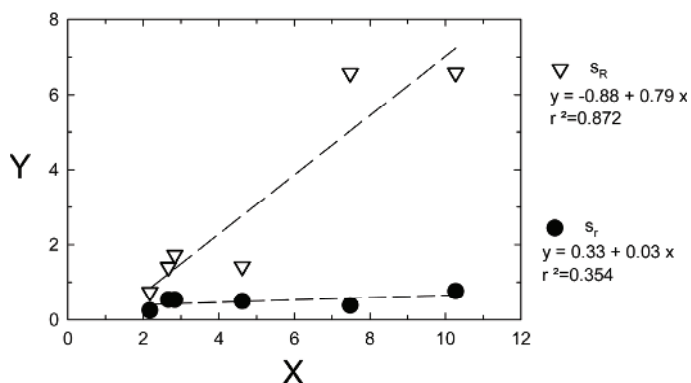
15 Results of the determination of "grain impurities"

Table 12 — Collation of test results, outliers and precision data on the determination of "grain impurities"

Determination of "grain impurities"

Sample:	A	A	B	B	C	C	D	D	E	E	F	F	A	B	C	D	E	F
Lab.No	Single results of the dupliacte determination (results given in [w/w%])												Mean of duplicate determination (results given in [w/w%])					
1	1.391	1.516	5.300	3.866	1.743	2.047	1.832	2.097	2.830	2.673	7.244	6.403	1.45	4.58	1.90	1.96	2.75	6.82
2	1.881	1.968	8.016	7.915	7.217	7.436	5.092	6.455	11.572	8.578	12.556	11.761	1.92	7.97	7.33	5.77	_10.08_ C	12.16
3	1.513	1.591	6.093	4.759	2.255	1.998	0.975	2.079	20.604	20.932	25.498	25.859	1.55	5.43	2.13	1.53	20.77	25.68
4	3.424	2.796	5.659	5.872	2.789	2.512	3.320	3.936	12.707	12.312	14.520	12.475	3.11	5.77	2.65	3.63	12.51	13.50
5	1.867	1.766	3.159	2.481	1.137	1.379	2.212	1.728	2.349	3.263	5.305	6.874	1.82	2.82	1.26	1.97	2.81	6.09
8	2.721	2.682	5.164	5.599	1.836	3.966	2.540	2.032	3.784	3.819	6.207	5.980	2.70	5.38 C	2.90	2.29	3.80	6.09
9	1.401	1.806	7.457	2.811	1.986	2.333	3.209	3.121	2.914	2.916	6.819	5.979	1.60	_5.13_ C	2.16	3.17	2.92	6.40
11	2.102	2.190	4.401	4.178	9.016	10.567	6.794	10.254	16.810	16.870	16.755	18.410	2.15	4.29	_9.79_ g	_8.52_ C	16.84	17.58
12	1.899	2.541	4.022	4.030	1.943	1.531	1.416	2.218	--	--	--	--	2.22	4.03	1.74	1.82	--	--
13	1.120	1.120	1.885	2.006	2.996	2.324	2.329	3.950	0.308	0.164	0.114	0.149	1.12	1.95	2.66	3.14	0.24	0.13
14	2.545	2.934	4.108	5.220	2.658	3.317	3.427	4.203	4.013	4.177	8.815	8.956	2.74	4.66	2.99	3.82	4.10	8.89
16	3.139	3.855	4.683	4.296	1.456	2.128	0.989	1.022	6.236	7.423	6.627	7.151	3.50	4.49	1.79	1.01	6.83	6.89
17	1.617	1.600	4.175	4.912	2.256	1.476	1.298	1.034	5.598	6.259	4.808	5.752	1.61	4.54	1.87	1.17	5.93	5.28
18	2.919	3.040	4.664	4.396	2.606	2.503	1.912	1.724	0.575	0.936	5.811	6.359	2.98	4.53	2.55	1.82	0.76	6.09
19	--	--	--	--	--	--	--	--	16.654	16.312	18.152	18.006	--	--	--	--	16.48	18.08
20	2.611	4.004	4.025	4.476	5.390	6.251	4.193	4.303	7.575	8.442	15.457	13.348	_3.31_ C	4.25	5.82	4.25	8.01	14.40

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	**2.18**	**4.62**	**2.84**	**2.67**	**7.48**	**10.27**
Number of laboratories	14	14	14	14	14	15
Repeatability standard deviation s_r	0.25	0.49	0.53	0.54	0.38	0.77
Variation coefficient of repeatability	11 %	11 %	19 %	20 %	5 %	7 %
Repeatability r	**0.69**	**1.38**	**1.48**	**1.50**	**1.06**	**2.14**
Reproducibility standard deviation s_R	0.74	1.42	1.72	1.40	6.58	6.59
Variation coefficient of reproducibility	34 %	31 %	61 %	53 %	88 %	64 %
Reproducibility R	**2.07**	**3.99**	**4.83**	**3.93**	**18.41**	**18.44**
Ratio (r/R)	0.33	0.35	0.31	0.38	0.06	0.12

Abbreviations: C _Cochtran's outlier, 1%, data eliminated_   g _Grubbs outlier, 5%, data eliminated_

Key
X   mean value of "grain impurities" [w/w %]
Y   standard deviation [w/w %]

**Figure 33 — Relationship between the standard deviation (s_r, s_R) and the mean value of "grain impurities"**

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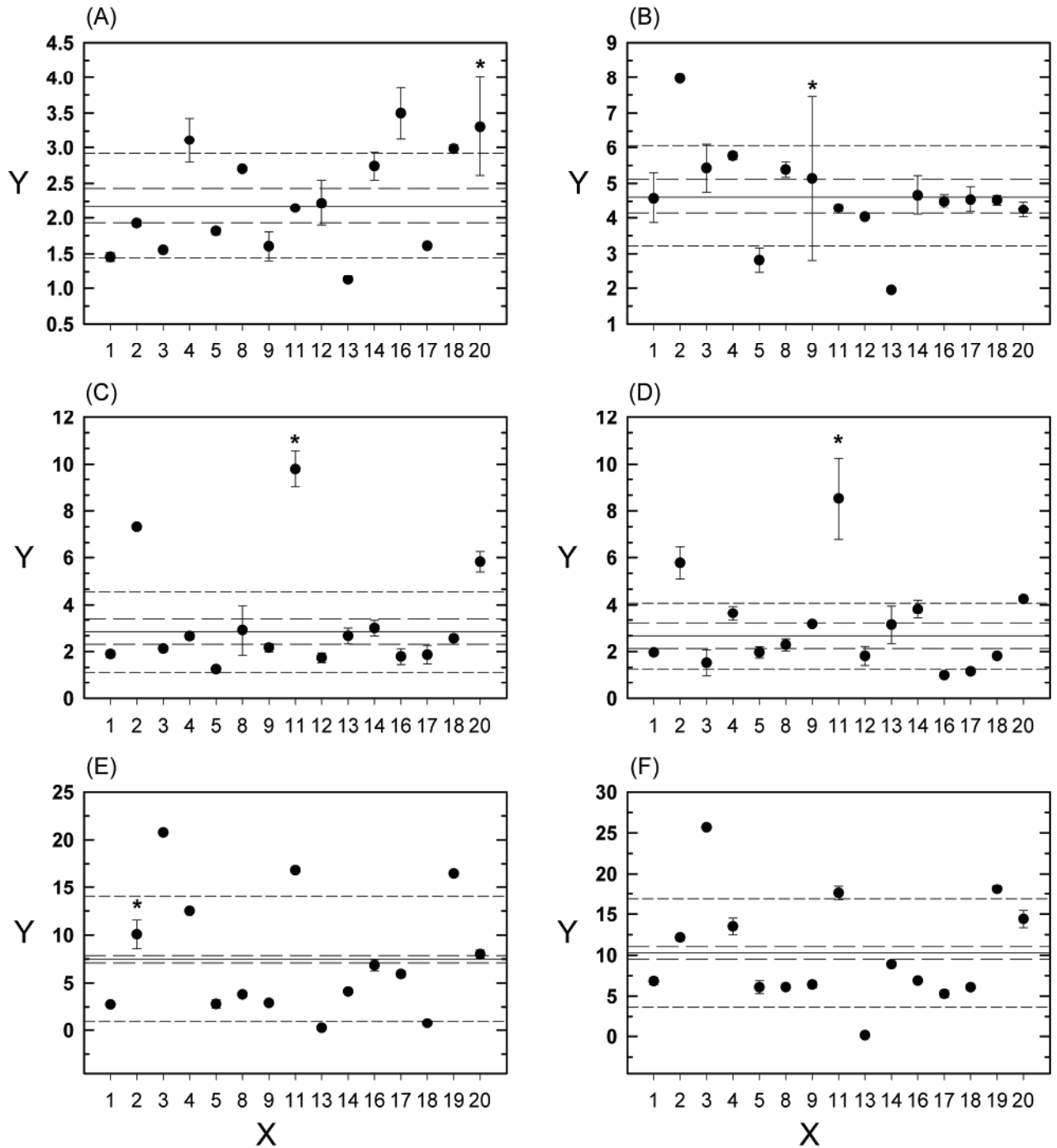
——— mean   — — s_r — — — — s_R (values without eliminated outliers (*))

X   laboratory
Y   grain impurities [w/w %]

Figure 34 — Single results of the duplicate determination of "grain impurities" including the overall mean, standard deviation of repeatability (s_r) and reproducibility (s_R) for the six test samples A to F

Key
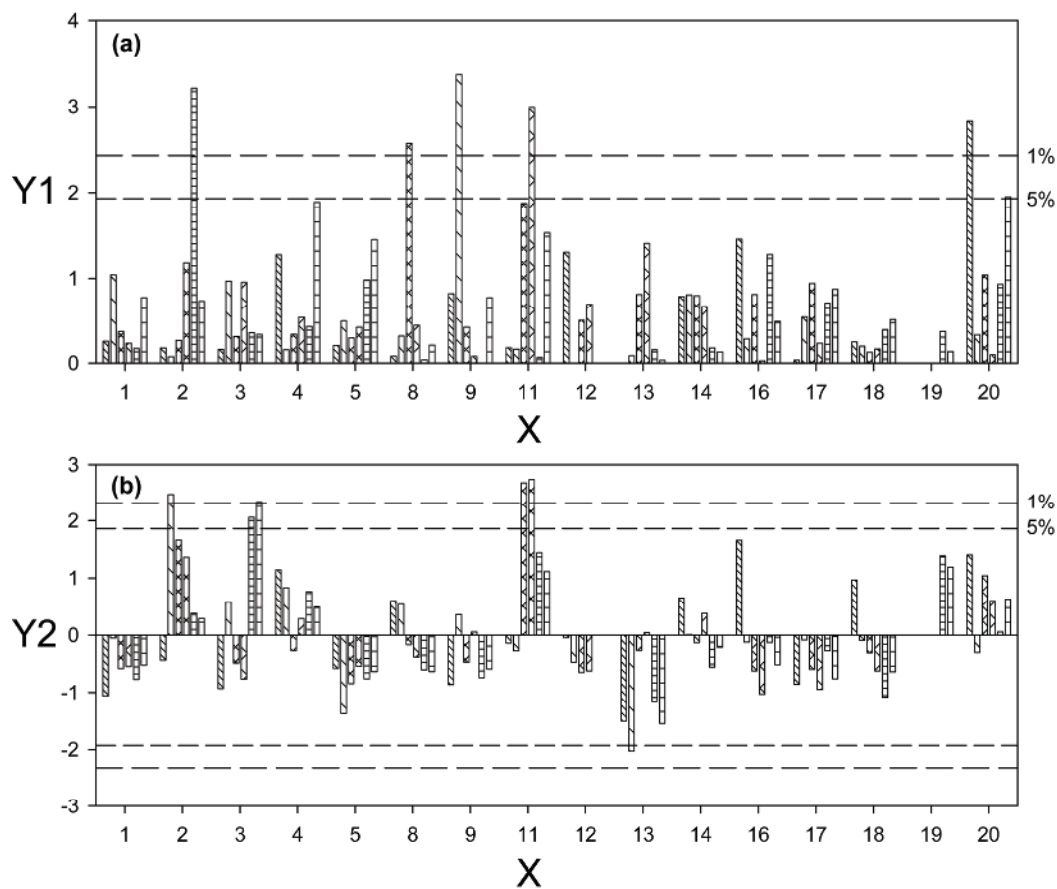
▨	A (wheat)
◹	B (wheat)
▩	C (rye)
◈	D (rye)
▤	E (durum)
▭	F (durum)

X laboratory

Y1 Mandel's statistic, *k*

Y2 Mandel's statistic, *h*

Figure 35 — Determination of "grain impurities": (a) Mandel's within-laboratory statistic, *k*, and (b) Mandel's between laboratory consistency statistic, *h*, each grouped by laboratories

16 Results of the determination of "miscellaneous impurities"

Table 13 — Collation of test results, outliers and precision data on the determination of "miscellaneous impurities"
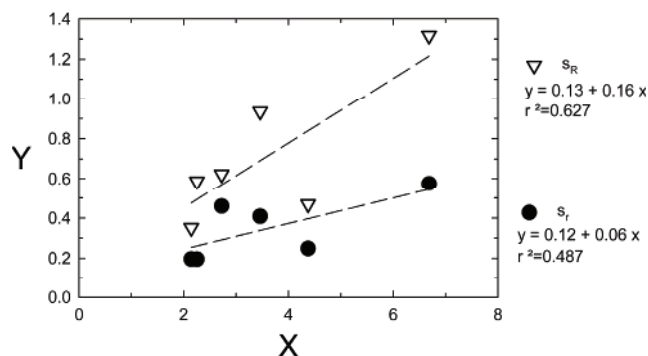
Determination of "miscellaneous impurities"

Sample:	A	A	B	B	C	C	D	D	E	E	F	F	A	B	C	D	E	F
Lab.No																		
1	2.184	2.147	2.514	2.176	4.086	4.259	7.430	7.883	3.437	3.264	2.897	2.971	2.17	2.34	4.17	7.66	3.35	2.93
2	2.332	1.800	1.509	1.434	3.575	3.550	6.043	5.975	3.530	2.850	2.145	1.450	2.07	1.47	3.56	6.01	3.19	1.80
3	2.427	2.389	1.911	1.980	4.564	4.807	8.020	8.637	4.141	4.499	3.506	3.482	2.41	1.95	4.69	8.33	4.32	3.49
4	2.184	1.728	2.838	3.020	4.906	4.711	7.440	6.934	4.356	3.790	2.389	4.740	1.96	2.93	4.81	7.19	4.07	*3.56* K
5	1.927	2.432	2.035	2.002	3.715	3.828	6.971	7.136	3.499	3.630	2.015	3.502	2.18	2.02	3.77	7.05	3.56	2.76
8	2.016	2.355	1.577	1.420	4.051	4.193	7.193	6.051	4.145	3.806	2.072	2.250	2.19	1.50	4.12	6.62	3.98	2.16
9	1.932	1.799	2.643	2.784	4.587	4.175	6.752	6.259	3.625	3.544	2.036	1.947	1.87	2.71	4.38	6.51	3.58	1.99
11	2.790	2.929	3.664	3.252	6.353	7.254	11.937	9.853	8.277	9.589	7.080	8.259	2.86	3.46	*6.80* G	*10.90* g	*8.93* G	*7.67* G
12	2.366	2.587	2.042	1.860	4.651	4.628	6.103	6.632	--	--	--	--	2.48	1.95	4.64	6.37	--	--
13	2.064	2.108	1.997	1.715	5.048	4.952	3.773	4.840	1.262	2.464	2.983	2.598	2.09	1.86	5.00	4.31	1.86	2.79
14	1.548	1.491	0.839	2.916	4.580	3.823	7.737	6.339	2.934	2.884	3.163	2.558	1.52	*1.88* K	4.20	7.04	2.91	2.86
16	1.758	1.681	2.830	2.283	4.261	4.603	4.783	3.523	1.520	1.982	2.931	2.090	1.72	2.56	4.43	4.15	1.75	2.51
17	2.198	2.383	2.018	2.060	4.285	4.546	7.568	6.837	3.040	3.565	1.963	2.842	2.29	2.04	4.42	7.20	3.30	2.40
18	1.863	2.205	2.737	4.898	4.061	4.021	6.466	6.809	3.949	4.203	3.091	3.456	2.03	*3.82* K	4.04	6.64	4.08	3.27
19	--	--	--	--	--	--	--	--	3.300	3.511	3.006	3.023	--	--	--	--	3.41	3.01
20	2.440	2.242	2.268	2.724	4.633	5.427	9.063	7.952	5.693	4.455	3.804	3.025	2.34	2.50	5.03	8.51	5.07	3.41

Column groups: Single results of the dupliacte determination (results given in [w/w%]) | Mean of duplicate determination (results given in [w/w%])

Sample				A	B	C	D	E	F
A	=	common wheat	**Mean of test results**	**2.14**	**2.25**	**4.38**	**6.68**	**3.46**	**2.72**
B	=	common wheat	Number of laboratories	15	13	14	14	14	13
C	=	rye	Repeatability standard deviation s r	0.20	0.20	0.25	0.58	0.41	0.46
D	=	rye	Variation coefficient of repeatability	9 %	9 %	6 %	9 %	12 %	17 %
E	=	durum wheat	**Repeatability r**	**0.55**	**0.55**	**0.70**	**1.61**	**1.14**	**1.28**
F	=	durum wheat	Reproducibility standard deviation s R	0.35	0.59	0.47	1.32	0.93	0.62
			Variation coefficient of reproducibility	16 %	26 %	11%	20 %	27 %	23 %
			Reproducibility R	**0.98**	**1.64**	**1.31**	**3.69**	**2.62**	**1.74**
			Ratio (r/R)	0.56	0.33	0.53	0.44	0.44	0.74

Abbreviations: K *Mandel's statistic,k, outlier, 1%, data eliminated*
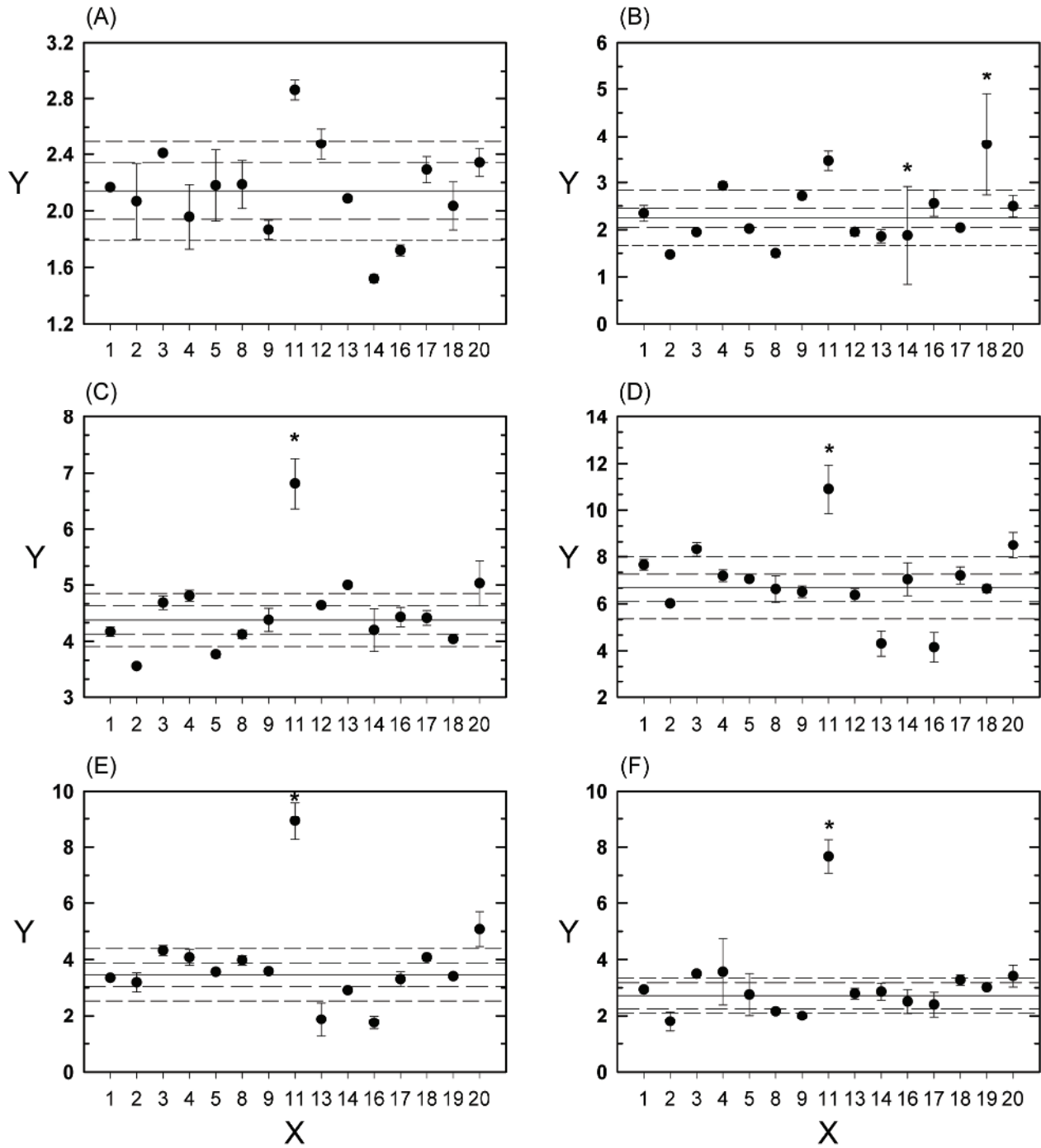
G *Grubbs outlier, 1%, data eliminated*

g *Grubbs outlier, 5%, data eliminated*

$$s_R \quad y = 0.13 + 0.16 \, x \quad r^2 = 0.627$$

$$s_r \quad y = 0.12 + 0.06 \, x \quad r^2 = 0.487$$

Key
X   mean value of "miscellaneous impurities" [w/w %]
Y   standard deviation [w/w %]

Figure 36 — Relationship between the standard deviation (s_r, s_R) and the mean value of "miscellaneous impurities"

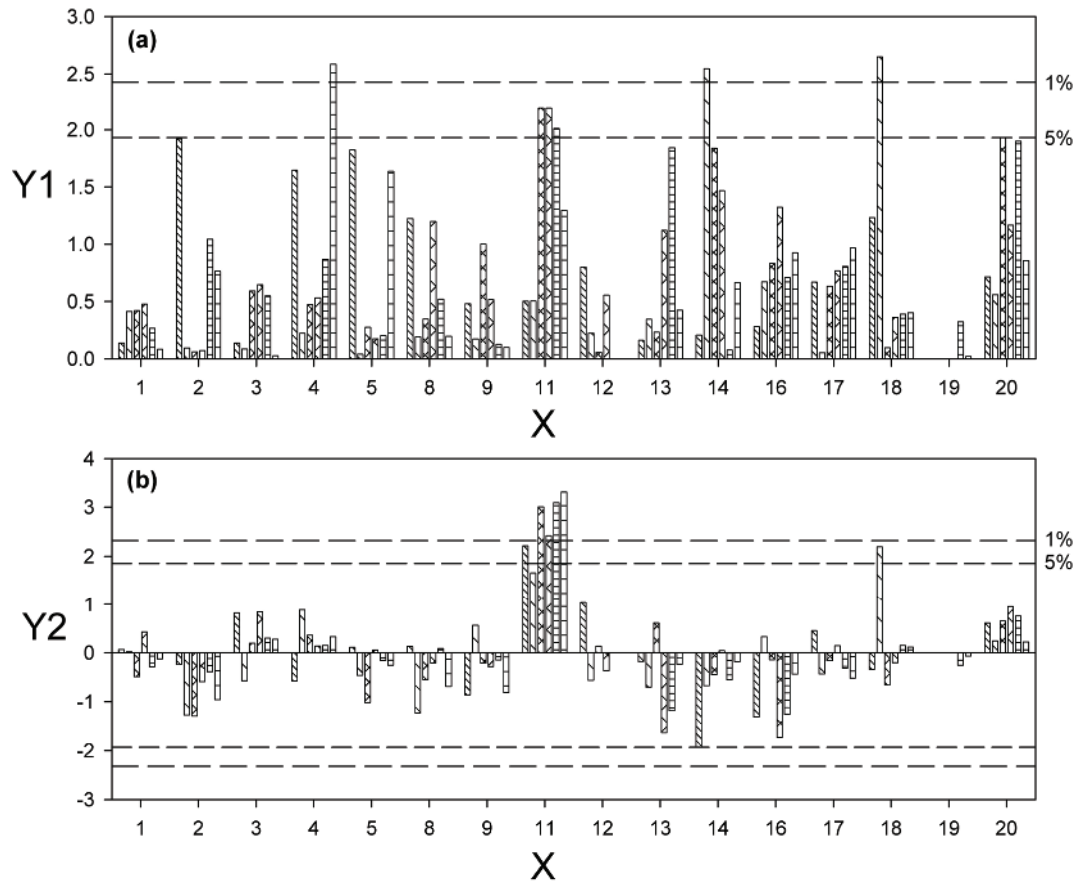**Key**

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

X   laboratory

Y   miscellaneous impurities [w/w %]

Figure 37 — Single results of the duplicate determination of "miscellaneous impurities" 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 38 — Determination of "émiscellaneous impurities": (a) Mandel's within-laboratory statistic, *k*, and (b) Mandel's between laboratory consistency statistic, *h*, each grouped by laboratories

17 Results of the determination of "total Besatz"

Table 14 — Collation of test results, outliers and precision data on the determination of "total Besatz"

Determination of "total Besatz"

	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	5.832	6.048	10.941	8.931	7.031	7.639	17.031	17.453	6.549	6.781	13.201	12.258	5.94	9.94	7.34	17.24	6.67	12.73
2	7.071	6.450	13.862	12.603	12.083	12.556	18.215	19.710	15.404	11.558	17.684	16.023	6.76	13.23	12.32	18.96	*13.48* C	16.85
3	7.071	6.176	11.277	9.606	8.454	8.667	17.765	19.620	25.843	26.706	32.462	32.861	6.62	10.44	8.56	18.69	26.27	32.66
4	6.634	6.166	11.145	11.610	9.300	9.026	19.105	18.608	18.109	17.278	20.036	20.382	6.40	11.38	9.16	18.86	17.69	20.21
5	5.546	5.436	7.883	7.032	5.877	6.230	16.757	15.806	6.186	7.558	9.656	12.340	5.49	7.46	6.05	16.28	6.87	11.00
8	6.076	6.957	10.086	9.323	7.675	9.207	17.225	16.386	8.271	7.882	10.229	10.155	6.52	9.70	8.44	16.81	8.08	10.19
9	6.191	6.082	13.208	9.000	7.779	7.988	17.526	16.558	6.865	6.930	11.180	9.838	6.14	*11.10* K	7.88	17.04	6.90	10.51
11	6.650	7.836	11.859	10.601	16.903	19.880	26.821	26.774	26.848	28.505	25.825	28.966	7.24	11.23	*18.39* G	*26.80* G	*27.68* h	27.40
12	6.927	6.831	9.672	9.453	8.258	7.659	15.086	16.490	--	--	--	--	6.88	9.56	7.96	15.79	--	--
13	5.344	5.388	7.409	7.085	9.105	8.919	13.701	16.392	1.620	2.931	3.759	3.676	5.37	7.25	9.01	15.05	2.28	3.72
14	5.797	6.583	7.659	10.394	8.911	8.640	18.549	18.336	7.419	7.290	14.143	14.366	6.19	9.03	8.78	18.44	7.35	14.25
16	7.077	6.754	10.830	10.901	7.405	8.906	12.150	12.159	9.255	10.855	13.350	12.491	6.92	10.87	8.16	12.15	10.05	12.92
17	5.897	5.992	9.043	9.904	7.596	7.339	16.252	15.352	9.068	10.607	8.183	9.599	5.94	9.47	7.47	15.80	9.84	8.89
18	6.425	6.455	9.959	12.166	8.044	7.736	15.542	15.734	5.359	5.634	10.791	12.102	6.44	11.06	7.89	15.64	5.50	11.45
19	--	--	--	–	--	--	--	--	20.433	20.203	22.690	22.749	--	--	--	--	20.32	22.72
20	6.285	7.497	8.891	10.259	11.154	13.502	20.962	19.619	13.417	13.256	21.594	18.050	6.89	9.57	*12.33* k	20.29	13.34	19.82

| Sample					Sample	A	B	C	D	E	F
A	=	common wheat			**Mean of test results**	**6.38**	**10.01**	**8.39**	**16.93**	**10.86**	**15.69**
B	=	common wheat			Number of laboratories	15	14	13	14	13	15
C	=	rye			Repeatability standard deviation s_r	0.44	0.98	0.48	0.86	0.63	1.15
D	=	rye			Variation coefficient of repeatability	7 %	10 %	6 %	5 %	6 %	7 %
E	=	durum wheat			**Repeatability r**	**1.24**	**2.73**	**1.36**	**2.40**	**1.77**	**3.23**
F	=	durum wheat			Reproducibility standard deviation s_R	0.62	1.71	1.48	2.16	6.80	7.69
					Variation coefficient of reproducibility	10 %	17 %	18 %	13 %	63 %	49 %
					Reproducibility R	**1.74**	**4.78**	**4.14**	**6.04**	**19.05**	**21.53**
					Ratio (r/R)	0.71	0.57	0.33	0.40	0.09	0.15

Abbreviations:

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

K *Mandel's statistic,k, outlier, 1%, data eliminated*   h *Mandel's statistic,h, outlier, 5%, data_eliminated*

k *Mandel's statistic,k, outlier, 5%, data_eliminated*
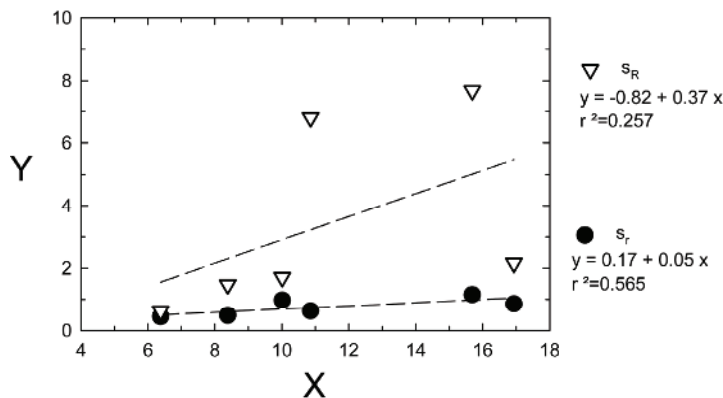
Key
X   mean value of "total Besatz" [w/w %]
Y   standard deviation [w/w %]

Figure 39 — Relationship between the standard deviation (s_r, s_R) and the mean value of "total Besatz"
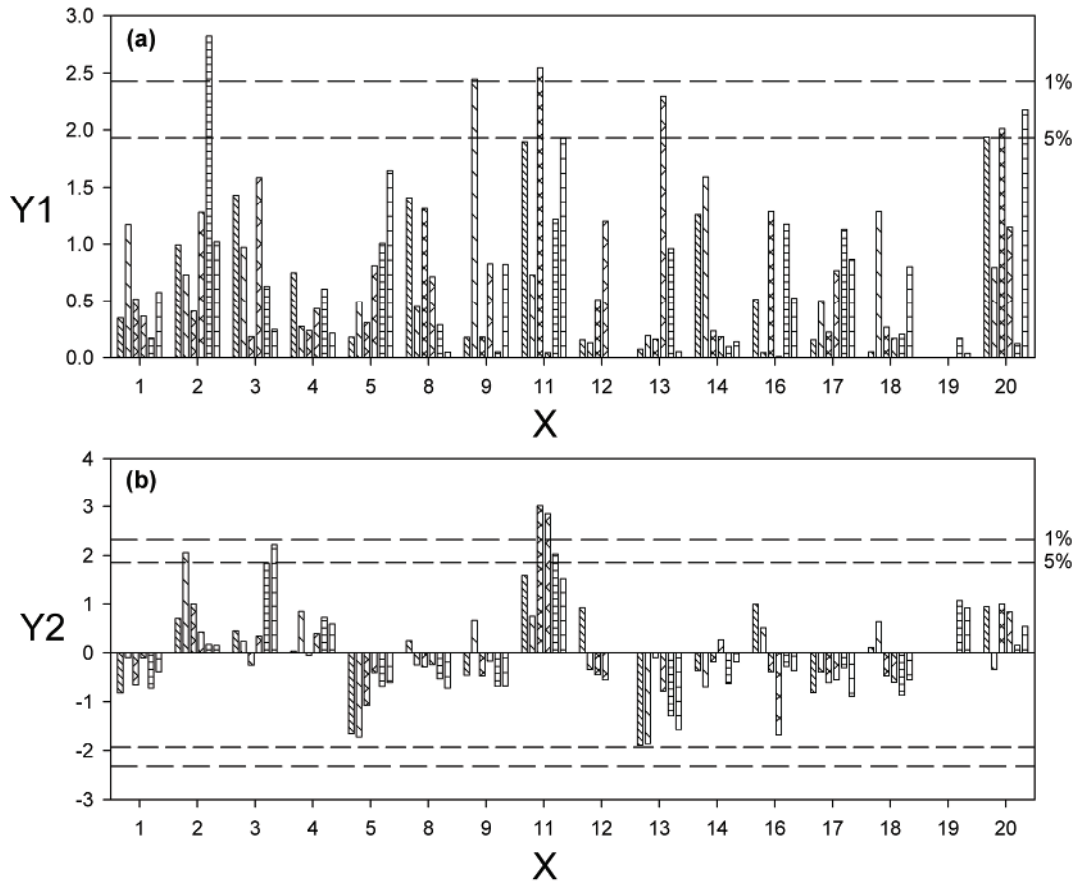
Key
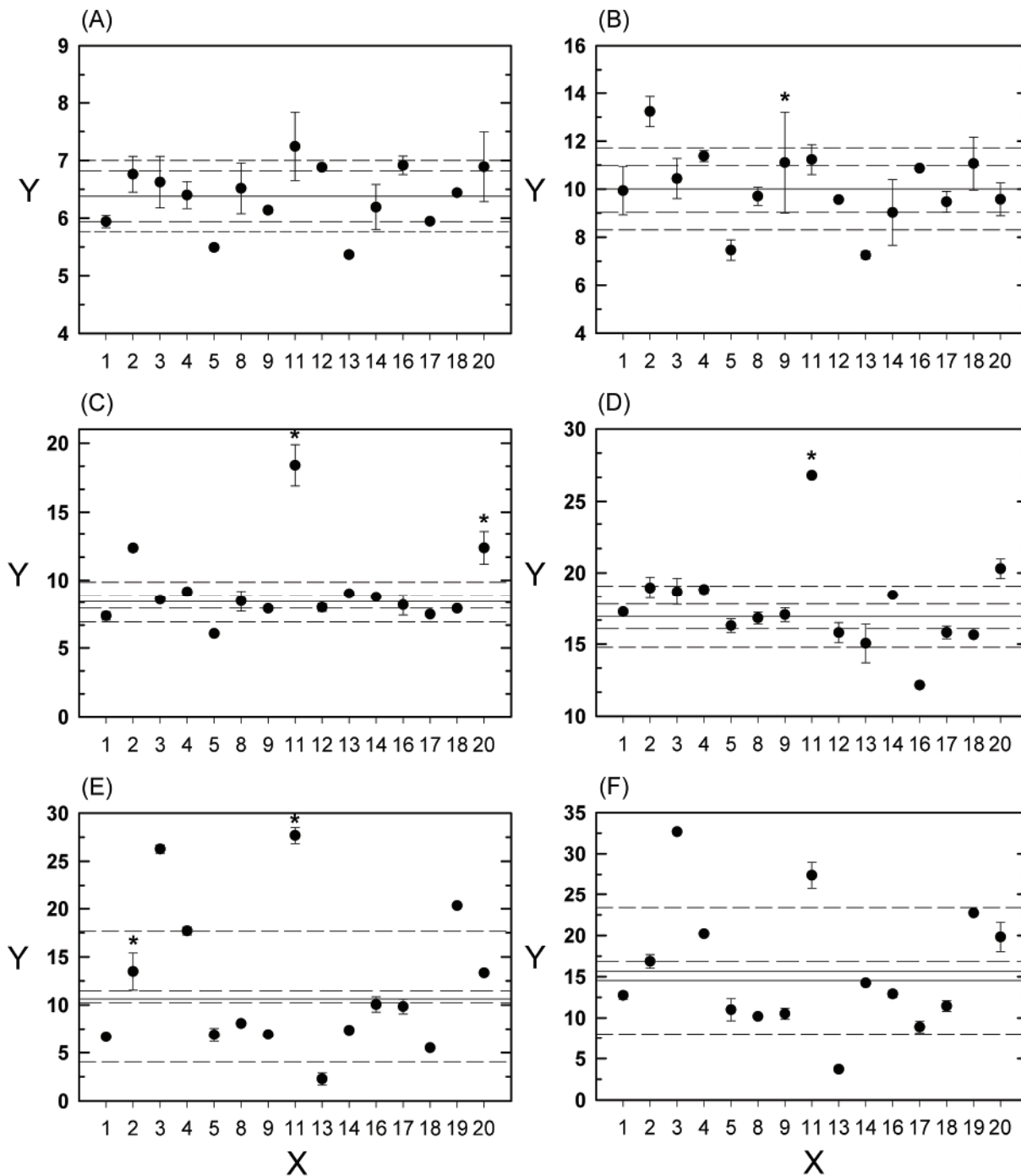
───── mean   ─ ─ sᵣ   ─ ─ ─ ─ s_R (values without eliminated outliers (*))

X   laboratory
Y   total Besatz [w/w %]

Figure 40 — Single results of the duplicate determination of "total Besatz" including the overall mean, standard deviation of repeatability (sᵣ) 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 41 — Determination of "total Besatz": (a) Mandel's within-laboratory statistic, k, and (b) Mandel's between laboratory consistency statistic, h, each grouped by laboratories

18 Precision data of sample set A

Table 15 — Precision data of the sample set A for "broken grains"

Cereal	Wheat	Rye	Durum
Outliers	-	-	-
Mean value, % w/w	1,55	3,14	1,98
Number of laboratories, p	15	15	15
Repeatability standard deviation s_r	0,17	0,15	0,13
Coefficient of variation of repeatability	10,9 %	4,9 %	6,6 %
Repeatability r	0,48	0,43	0,37
Reproducibility standard deviation s_R	0,41	0,15	0,15
Coefficient of variation of reproducibility	26,6 %	4,9 %	7,8 %
Reproducibility R	1,16	0,43	0,43
Ratio (r/R)	0,41	1,00	0,85
Added amount, % w/w	1,50	3,00	1,99
"Recovery", %	103	105	99

Table 16 — Precision data of the sample set A for "shrivelled grains"

Cereal	Wheat	Rye	Durum
Outliers	Lab.No. 20	Lab.No. 2,5,20	Lab.No. 2
Mean value, % w/w	1,09	1,10	0,98
Number of laboratories, p	14	12	14
Repeatability standard deviation s_r	0,18	0,13	0,32
Coefficient of variation of repeatability	16,1 %	11,6 %	33,1 %
Repeatability r	0,49	0,35	0,90
Reproducibility standard deviation s_R	0,19	0,14	0,41
Coefficient of variation of reproducibility	17,4 %	13,2 %	42,5 %
Reproducibility R	0,53	0,40	1,16
Ratio (r/R)	0,93	0,88	0,78
Added amount, % w/w	1,00	1,00	1,00
"Recovery", %	109	110	98

Table 17 — Precision data of the sample set A for "other cereals"

Cereal	Wheat	Rye	Durum
Outliers	-	-	Lab.No. 14
Mean value, % w/w	0,60	2,20	2,72
Number of laboratories, p	15	15	14
Repeatability standard deviation s_r	0,12	0,17	0,40
Coefficient of variation of repeatability	19,5 %	7,8 %	14,9 %
Repeatability r	0,33	0,48	1,13
Reproducibility standard deviation s_R	0,26	0,45	0,98
Coefficient of variation of reproducibility	43,8 %	20,7 %	36,1 %
Reproducibility R	0,73	1,27	2,75
Ratio (r/R)	0,45	0,38	0,41
Added amount, % w/w	0,65	2,06	2,55
"Recovery", %	92	107	109

Table 18 — Precision data of the sample set A for "grains damaged by pests"

Cereal	Wheat	Rye	Durum
Outliers	Lab.No. 20	-	Lab.No. 19,20
Mean value, % w/w	0,44	0,70	0,07
Number of laboratories, p	14,00	15,00	13,00
Repeatability standard deviation s_r	0,17	0,23	0,09
Coefficient of variation of repeatability	37,6 %	32,9 %	130 %
Repeatability r	0,46	0,64	0,26
Reproducibility standard deviation s_R	0,31	0,23	0,10
Coefficient of variation of reproducibility	70,9 %	32,9 %	145 %
Reproducibility R	0,88	0,64	0,29
Ratio (r/R)	0,53	1,00	0,90
Added amount, % w/w	0,75	1,00	0,40
"Recovery", %	59	70	18

Table 19 — Precision data of the sample set A for "grains in which the germ is discoloured, mottled grains"

Cereal	Wheat	Durum
Outliers	Lab.No. 12	Lab.No. 19
Mean value, % w/w	0,68	0,82
Number of laboratories, p	14	14
Repeatability standard deviation s_r	0,14	0,39
Coefficient of variation of repeatability	20,2 %	47,5 %
Repeatability r	0,38	1,09
Reproducibility standard deviation s_R	0,44	0,62
Coefficient of variation of reproducibility	65,7 %	75,6 %
Reproducibility R	1,24	1,74
Ratio (r/R)	0,31	0,63
Added amount, % w/w	0,50	0,50
"Recovery", %	136	164

Table 20 — Precision data of the sample set A for "sprouted grains"

Cereal	Wheat	Rye	Durum
Outliers	-	Lab.No. 9	Lab.No. 18,9,14
Mean value, % w/w	0,86	0,99	0,79
Number of laboratories, p	15	14	12
Repeatability standard deviation s_r	0,17	0,18	0,18
Coefficient of variation of repeatability	19,7 %	18,3 %	22,4 %
Repeatability r	0,47	0,51	0,49
Reproducibility standard deviation s_R	0,23	0,25	0,21
Coefficient of variation of reproducibility	27,4 %	24,7 %	26,8 %
Reproducibility R	0,66	0,69	0,59
Ratio (r/R)	0,72	0,74	0,84
Added amount, % w/w	1,00	1,00	1,00
"Recovery", %	86	99	79

Table 21 — Precision data of the sample set A for "extraneous seeds"

Cereal	Wheat	Rye	Durum
Outliers	-	Lab.No. 17,14	Lab.No. 14,17
Mean value, % w/w	0,90	1,83	1,91
Number of laboratories, p	15	13	13
Repeatability standard deviation s $_r$	0,06	0,07	0,07
Coefficient of variation of repeatability	7 %	4 %	3,9 %
Repeatability r	0,18	0,20	0,21
Reproducibility standard deviation s $_R$	0,17	0,14	0,16
Coefficient of variation of reproducibility	18,7 %	7,8 %	8,3 %
Reproducibility R	0,47	0,40	0,45
Ratio (r/R)	0,37	0,51	0,47
Added amount, % w/w	1,00	2,00	2,00
"Recovery", %	90	91	95

Table 22 — Precision data of the sample set A for "unsound grains"

Cereal	Wheat	Rye	Durum
Outliers	-	Lab.No. 5,11	Lab.No. 2
Mean value, % w/w	0,74	1,17	0,34
Number of laboratories, p	15	13	14
Repeatability standard deviation s $_r$	0,48	0,24	0,22
Coefficient of variation of repeatability	64,5 %	19,2 %	64,4 %
Repeatability r	1,34	0,63	0,62
Reproducibility standard deviation s $_R$	0,68	0,78	0,28
Coefficient of variation of reproducibility	91,8 %	67,1 %	82,2 %
Reproducibility R	1,90	2,20	0,79
Ratio (r/R)	0,70	0,29	0,78
Added amount, % w/w	1,00	1,50	0,40
"Recovery", %	74	79	86

Table 23 — Precision data of the sample set A for "extraneous matter"

Cereal	Wheat	Rye	Durum
Outliers	-	Lab.No. 2,14	Lab.No. 9,14
Mean value, % w/w	0,97	1,56	1,51
Number of laboratories, p	15	13	13
Repeatability standard deviation s $_r$	0,10	0,09	0,10
Variation coefficient of repeatability	10,2 %	5,5 %	6,6 %
Repeatability r	0,28	0,24	0,28
Reproducibility standard deviation s $_R$	0,32	0,19	0,28
Variation coefficient of reproducibility	32,7 %	12,1 %	18,6 %
Reproducibility R	0,89	0,53	0,79
Ratio (r/R)	0,31	0,45	0,35
Added amount, % w/w	1,00	1,50	1,50
"Recovery", %	97	104	101

Table 24 — Precision data of the sample set A for "husks"

Cereal	Wheat	Rye	Durum
Outliers	Lab.No. 4,5	Lab.No. 14	-
Mean value, % w/w	1,00	0,22	0,54
Number of laboratories, p	13	14	15
Repeatability standard deviation s_r	0,07	0,05	0,06
Coefficient of variation of repeatability	7,1 %	23 %	10,4 %
Repeatability r	0,20	0,14	0,16
Reproducibility standard deviation s_R	0,08	0,06	0,10
Coefficient of variation of reproducibility	8 %	25,9 %	19 %
Reproducibility R	0,22	0,16	0,29
Ratio (r/R)	0,88	0,89	0,55
Added amount, % w/w	1,00	0,20	0,50
"Recovery", %	100	109	108

Table 25 — Precision data of the sample set A for "ergot"

Cereal	Wheat	Rye	Durum
Outliers	-	-	Lab.No. 9,5
Mean value, % w/w	0,99	1,47	2,00
Number of laboratories, p	15	15	13
Repeatability standard deviation s_r	0,02	0,05	0,02
Coefficient of variation of repeatability	2,1 %	3,6 %	0,9 %
Repeatability r	0,06	0,15	0,05
Reproducibility standard deviation s_R	0,03	0,05	0,02
Coefficient of variation of reproducibility	3 %	3,6 %	1,2 %
Reproducibility R	0,08	0,15	0,07
Ratio (r/R)	0,70	1,00	0,74
Added amount, % w/w	1,00	1,50	2,00
"Recovery", %	99	98	100

Table 26 — Precision data of the sample set A for "grain impurities"

Cereal	Wheat	Rye	Durum
Outliers	-	-	-
Mean value, % w/w	2,90	4,51	4,97
Number of laboratories, p	15	15	15
Repeatability standard deviation s_r	0,50	0,56	1,07
Variation coefficient of repeatability	17,3 %	12,4 %	21,5 %
Repeatability r	1,40	1,57	2,99
Reproducibility standard deviation s_R	0,72	1,99	1,63
Variation coefficient of reproducibility	25 %	44 %	32,7 %
Reproducibility R	2,03	5,56	4,55
Ratio (r/R)	0,69	0,28	0,66
Added amount, % w/w	2,9	4,06	4,45
"Recovery", %	100	111	112

Table 27 — Precision data of the sample set A for "miscellaneous impurities"

Cereal	Wheat	Rye	Durum
Outliers	-	Lab.No. 11	Lab.No. 9
Mean value, % w/w	4,66	6,23	6,38
Number of laboratories, p	15	14	14
Repeatability standard deviation s r	0,50	0,56	0,29
Variation coefficient of repeatability	10,8 %	9 %	4,6 %
Repeatability r	1,41	1,57	0,81
Reproducibility standard deviation s R	0,68	0,88	0,41
Variation coefficient of reproducibility	14,6 %	14,1 %	6,5 %
Reproducibility R	1,90	2,45	1,15
Ratio (r/R)	0,74	0,64	0,70
Added amount, % w/w	5,0	6,7	6,4
"Recovery", %	93	93	100

Table 28 — Precision data of the sample set A for "total Besatz"

Cereal	Wheat	Rye	Durum
Outliers	-	Lab.No. 20	-
Mean value, % w/w	9,97	14,93	14,04
Number of laboratories, p	15	14	15
Repeatability standard deviation s $_r$	0,66	0,96	1,17
Variation coefficient of repeatability	6,6 %	6,4 %	8,3 %
Repeatability r	1,84	2,69	3,27
Reproducibility standard deviation s $_R$	1,03	2,20	1,80
Variation coefficient of reproducibility	10,3 %	14,7 %	12,8 %
Reproducibility R	2,88	6,16	5,04
Ratio (r/R)	0,64	0,44	0,65
Added amount, % w/w	10,4	14,76	13,84
"Recovery", %	96	101	101

The results of this preliminary test showed that no laboratory reported such striking results that it should be excluded from the ring trial evaluation.

The recoveries were in the range 90-110 percent for most of the Besatz groups. Whereas for the Besatz groups "unsound grains", "grains damaged by pest" and "grains in which the germ is discoloured, mottled grains", the recoveries varied in the wide range of 18 to 164 percent.

19 Results of sample set A and B

In the following section, the variability of results is shown in relation to the amount of the various Besatz fractions. In the most cases an exponential relationship exists between the amount of a Besatz group and the relative standard deviation of the results.

The results from sample set A and B are presented together in one figure. In this way, it is clear that the sample division had a significant influence on the variability of the results in the groups of broken grains and ergot. The variability of the results for the other Besatz groups does not differ remarkably between the two sample sets.

Key

A intralaboratory
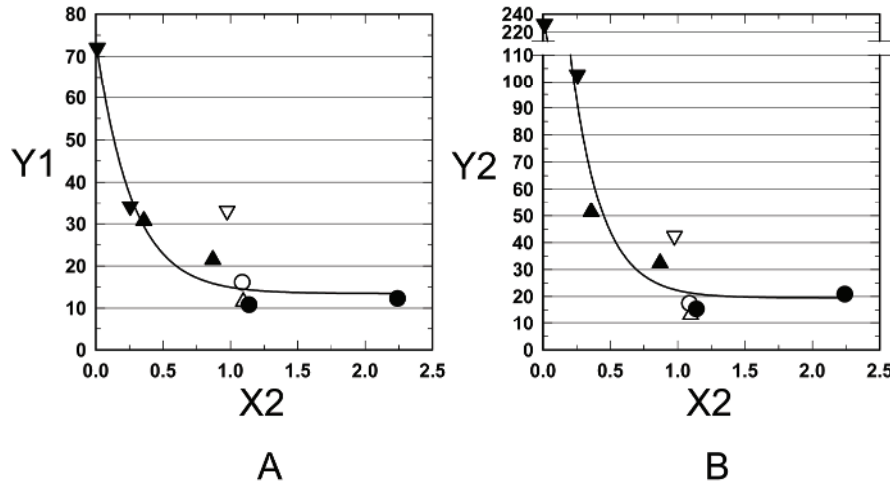B interlaboratories

a) broken grains

Key

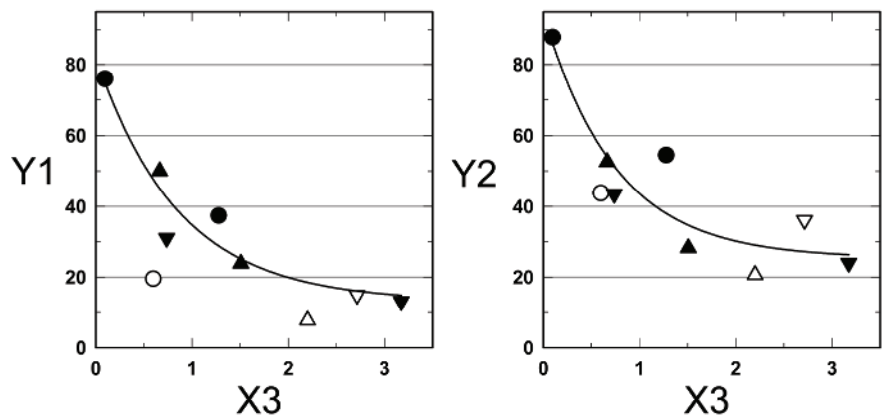X1 broken grains [% w/w]
Y1 RSD_r [%]
Y2 RSD_R [%]
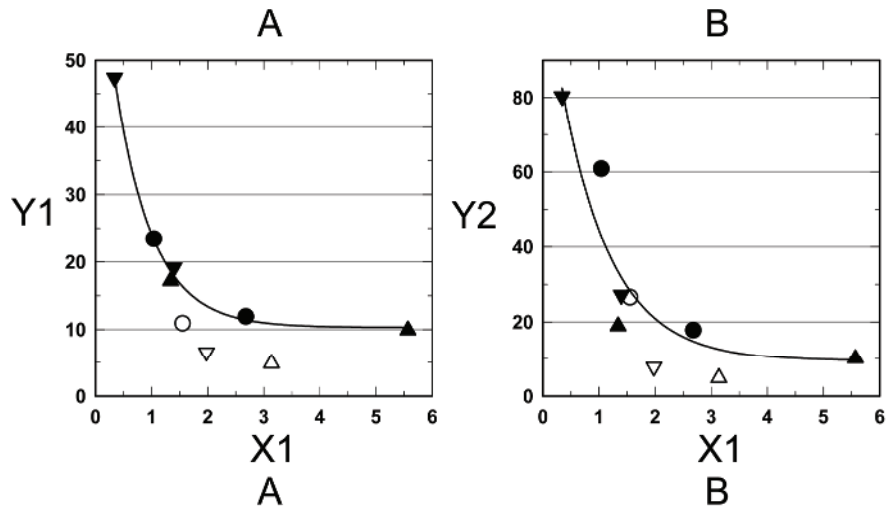
b) shrivelled grains

Key

X2 shrivelled grains [% w/w]

Y1 RSD_r [%]

Y2 RSD_R [%]

c) other cereals

Key

X3 other cereals [% w/w]

Y1 RSD_r [%]

Y2 RSD_R [%]

d) grains damaged by pests

Key

X1 grains damaged by pests [% w/w]
Y1 RSD$_r$ [%]
Y2 RSD$_R$ [%]
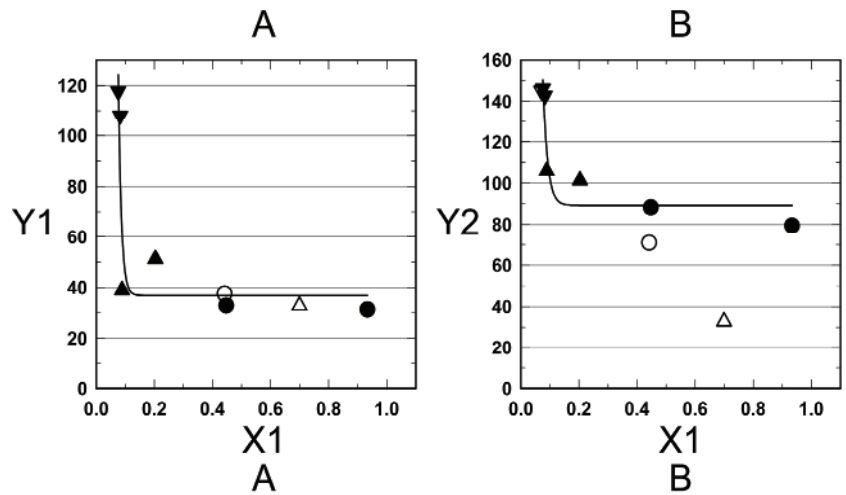
e) grains in which the germ is discoloured, mottled grains

Key

X2 grains in which the germ is discoloured, mottled grains [% w/w]
Y1 RSD$_r$ [%]
Y2 RSD$_R$ [%]
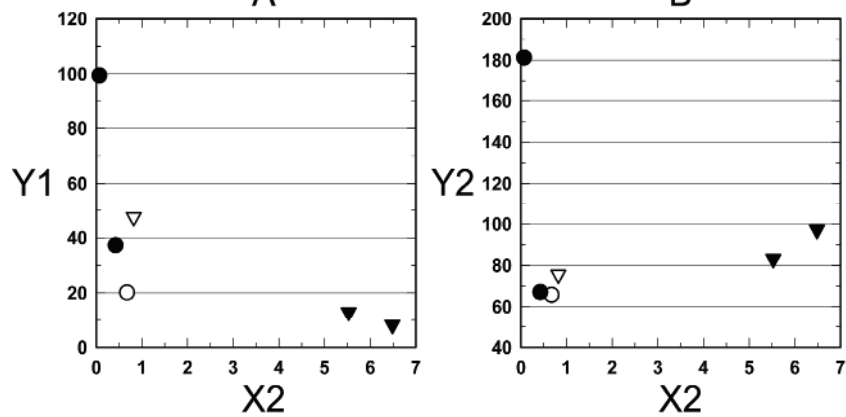
f) sprouted grains
Key

X3 sprouted grains [% w/w]
Y1 RSD$_r$ [%]
Y2 RSD$_R$ [%]
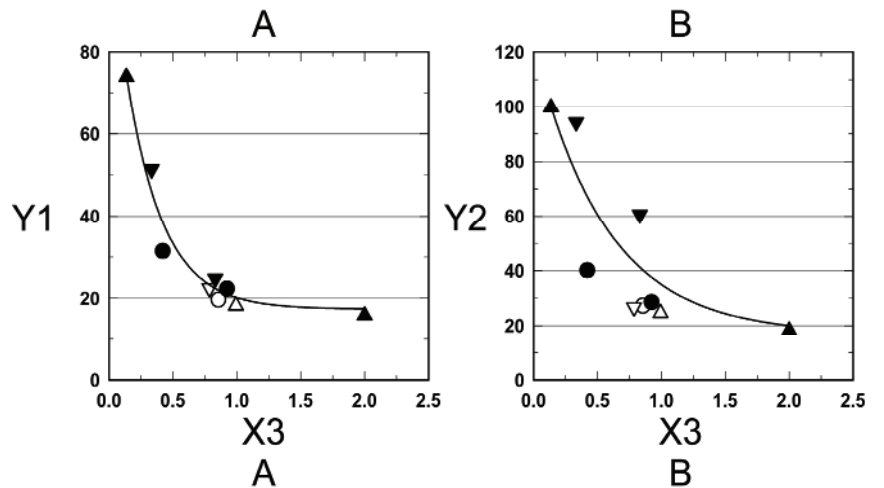
g) extraneous seeds
Key
X4 extraneous seeds [% w/w]
Y1 RSD$_r$ [%]
Y2 RSD$_R$ [%]
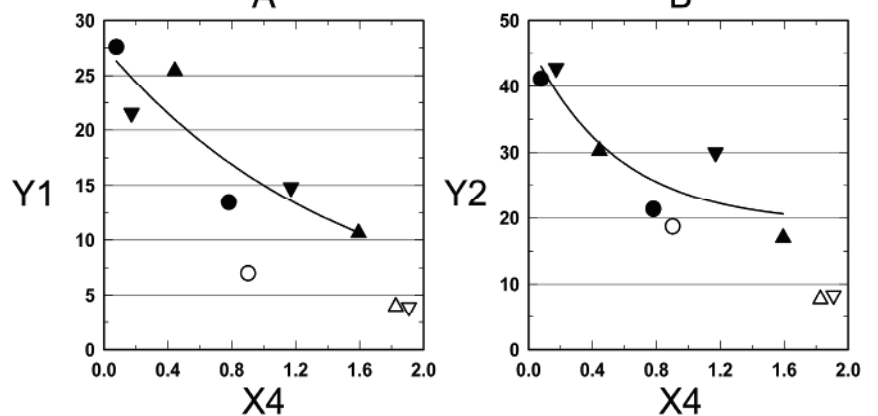
h) unsound grains

Key

X1 unsound grains [% w/w]
Y1 RSD_r [%]
Y2 RSD_R [%]
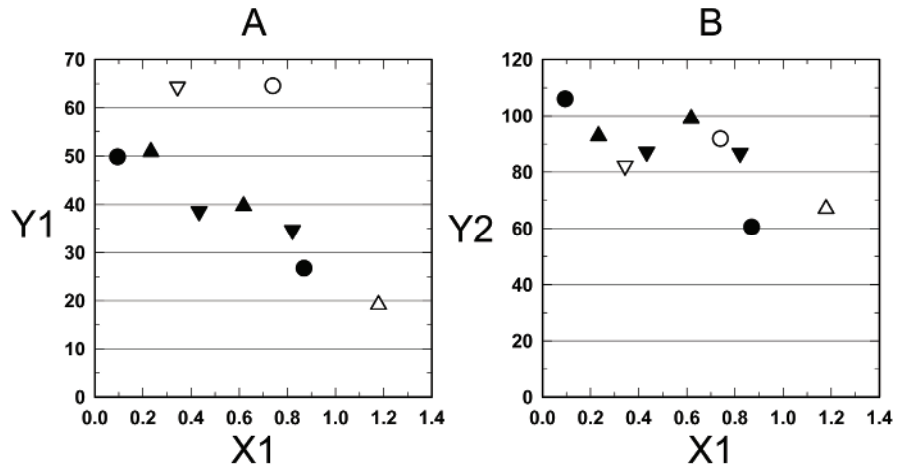
i) extraneous matter

Key

X2 extraneous matter [% w/w]
Y1 RSD_r [%]
Y2 RSD_R [%]
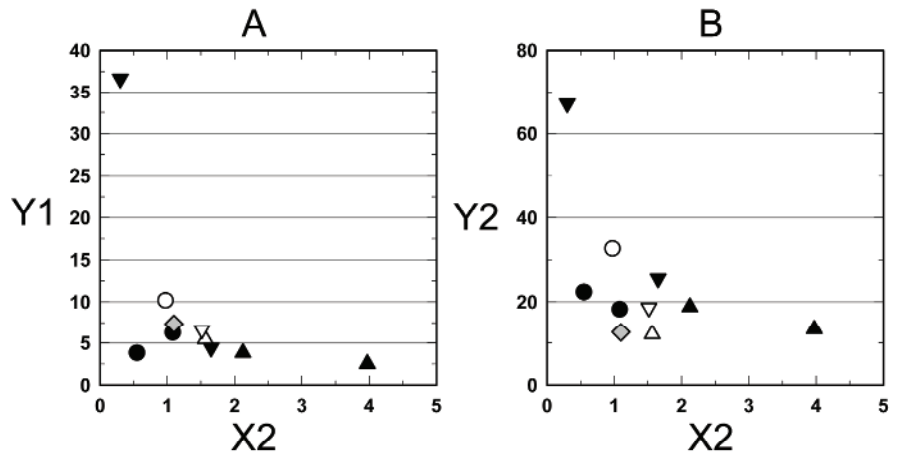
j) husks

Key

X3 husks [% w/w]
Y1 RSD_r [%]
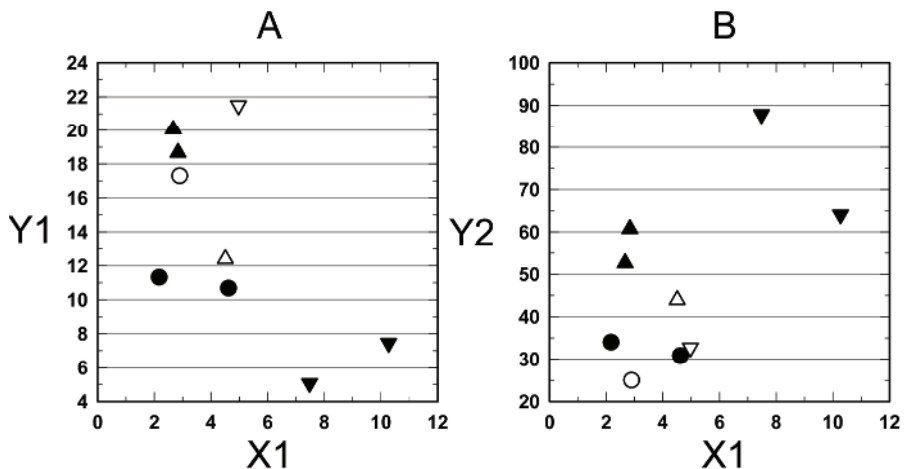Y2 RSD_R [%]
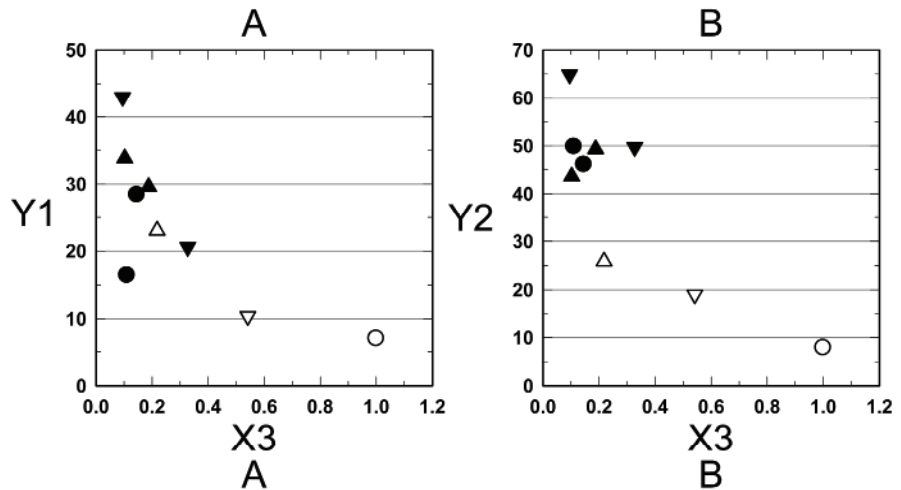
k) ergot

Key

X4 ergot [% w/w]
Y1 RSD_r [%]
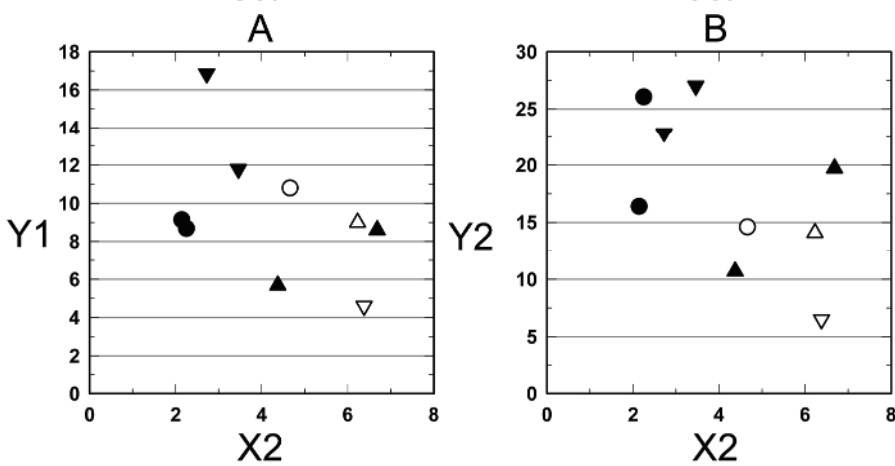Y2 RSD_R [%]
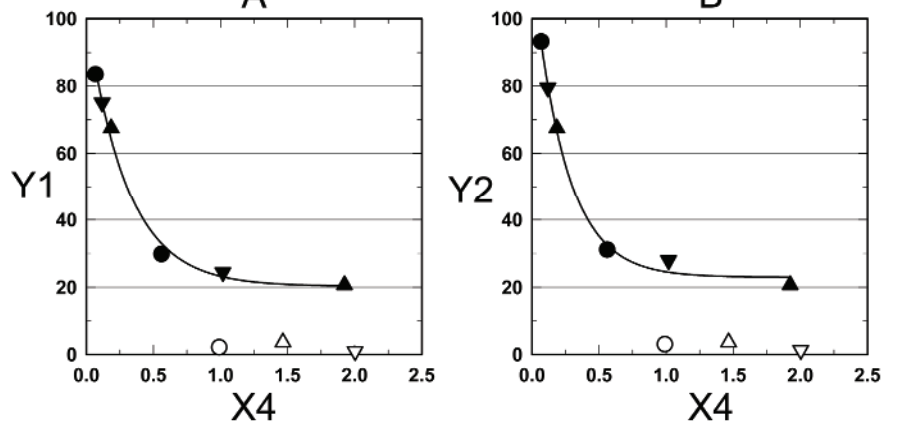
l) grain impurities

Key

X1 grain impurities [% w/w]
Y1 RSD$_r$ [%]
Y2 RSD$_R$ [%]

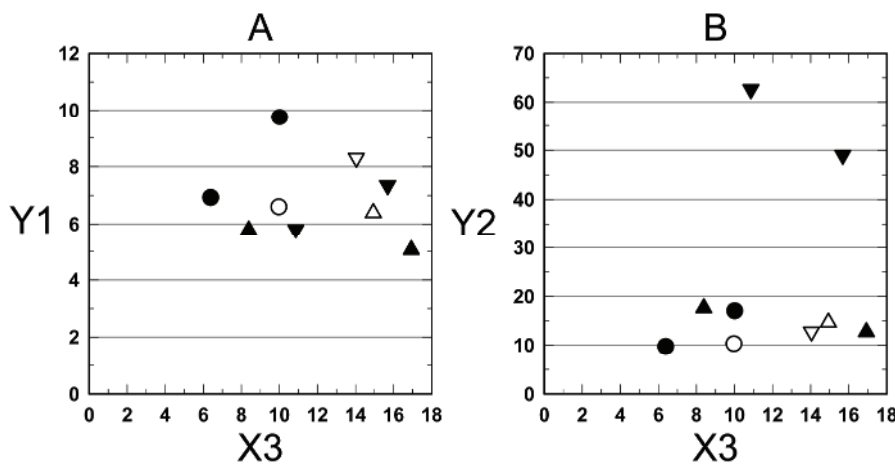m) miscellaneous impurities

Key

X2 miscellaneous impurities
[% w/w]
Y1 RSD$_r$ [%]
Y2 RSD$_R$ [%]

n) total Besatz

Key

X3 total Besatz [% w/w]
Y1 RSD$_r$ [%]
Y2 RSD$_R$ [%]

Key
Sample set B (with sample division)   ● wheat   ▲ rye   ▼ durum
Sample set A (without sample division)   ○ wheat   △ rye   ▽ durum

Figure 42 — Intralaboratory (RSD$_r$) and interlaboratory (RSD$_R$) relative standard deviation of sample set B (closed symbols) and sample set A (open symbols) in dependence of the amount of the Besatz group (the regressions curves include only results of sample set B)

Annex A
(informative)

Participants

Sixteen participants from eleven countries submitted their test results. One laboratory sent only results of the durum samples and another laboratory sent only results of the wheat and rye samples. In total we received results of 15 laboratories for each sample.

Special thanks must be given to the following participating laboratories:

— Raiffeisen Ware Austria (AT);

— National Grain Feed Service of Bulgaria (BG);

— Agrotest (CZ);

— Laboratory for Plant Production (EE);

— Centre Gallardonnais D'Analyses Cerealieres (FR);

— Crecerpal (FR);

— Laboragro – In Vivo (FR);

— SGS Agri Min (FR);

— Bundeslehranstalt Burg Warberg (GE);

— DiGeFa (GE); Oberfinanzdirektion Cottbus (GE);

— The State Seed and Grain Service (LT);

— Rikilt (NL); Stacja Doswiadczalna Oceny Odmian (PL);

— VNIIZ (RU); Campden BRI (UK).

Bibliography

[1]   EN 15587:2008, *Cereals and cereal products — Determination of Besatz in wheat (Triticum aestivum L.) durum wheat (Triticum durum Desf.), rye (Secale cereal L.) and feed barley (Hordeum vulgare L.)*

[2]   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*

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