Appendix 17

Standard Values for Testing

The standard

This publication describes the elements needed for best laboratory practice for grain testing. It gives straightforward guidance on the requirements for equipment, methods and operators. It is not designed to supersede any quality systems already in place and is subject to third party audit and accreditation. However, it provides a framework for those who do not operate to these standards (see page 2).

In addition to the framework, the publication describes targets for testing. These are measures of the proficiency of a laboratory and are given as the limits of variation that are acceptable for repeat testing by the laboratory. They are also given as limits of deviation from defined mean/median values or reference values when participating in proficiency test schemes and limits of variation for use with Internal Quality Control checks. Limits of variation for staff competency are also provided. These values have been derived from a very large pool of actual testing data. An explanation of terminology and derivation of standard value is given on page 3, followed by standard values in the table on page 4.

Who should adopt this standard?

All UK laboratories that test grain for trading purposes. Registers of participating laboratories (along with their testing protocols) can be found the relevant trade association websites. Click on the relevant link to view the register or scheme.

https://www.ukflourmillers.org/wheattestingatmillintake

AIC | Trade Assurance Schemes (agindustries.org.uk)

https://www.ukmalt.com/protocols-and-procedures

https://www.ukas.com/find-an-organisation/

Scope – tests to be included in the scheme

Ideally all participating laboratories should include the following tests within ring testing schemes. Those laboratories not undertaking the full range of tests will have a reduced scope.

Test	Wheat	Barley	OSR	Oats	Rye	Peas	Beans	Linseed	Maize
Moisture	✓	✓	✓	✓	✓	✓	✓	✓	✓
Specific weight	✓	✓		✓	✓				
Screenings	✓	✓		✓	✓				
Admixture	✓	✓	✓	✓	✓	✓	✓	✓	✓
Protein	✓			✓	✓		✓		
Nitrogen		✓							
Oil			✓					✓	
Falling number	✓				✓				
Gluten	✓								
Hardness	✓								
Germination		✓							
Mycotoxins	✓	✓		✓	✓				
Erucic Acid			✓						

Grain testing best practice

Equipment

1. All equipment used for testing must be fit for purpose, serviced and maintained in compliance with the laboratory's quality system and manufacturer's instructions. Requirements for equipment, eg balance accuracy, would be given in the test method
2. Equipment shall be calibrated according to routines within the method and/or against a defined schedule
3. Performance checks must be done before use. These checks should be performed daily for contractual testing or weekly, as a minimum, for non-contractual testing
4. When all the above (1–3) have been fulfilled the equipment is ready for use

Method

5. The method shall be documented – this can be an official method or an in-house procedure. If the latter is used, the relationship to the official method must be known. Procedures must be in place to ensure that only current methods are used
6. Is the method accepted by the grain industry? Ensure you only use methods that give results that others recognise, eg SKCS scale for wheat hardness

Operator

7. All staff must be competent in those tests that they are expected to undertake
8. All staff (full-time and temporary) shall be trained in the methods they are required to perform. A record of competence will be held by the laboratory manager and reviewed regularly. Preferably, staff will have undertaken the necessary laboratory training modules (available at https://ahdb.talentlms.com/)
9. All staff must ensure that any instrument of test performance checks are completed before routine testing can begin. These checks should be performed daily for contractual testing or weekly, as a minimum, for non-contractual testing

Sample, testing and reporting

10. Only when all the above elements have been fulfilled can the sample be taken and testing start. Ensure that the test sample is representative of the sample provided to the laboratory (either in bag or from the laboratory taken sample). Rules about sub-sampling, grinding and mixing must be followed
11. Undertake the test according to the documented method. Observe all the critical control points
12. Results shall be reported in a format agreed with the customer. All results shall be reported with units of measurement as recognised in the grain industry

Standard values for testing

1. **Repeatability** – permitted difference between two results on same sample done by the same person, using same instrument within a short timeframe
2. **Reproducibility** – permitted difference between two results on the same sample from different testing facilities
3. **Standard Deviation (SD) for Proficiency Testing Schemes (PT)** – maximum permitted value to be used to calculate z-scores (see section 5 below for explanation of z-scores). Providers are encouraged to use lower values for SD if participants are regularly achieving better performance than the standard set by these values
4. **Standard Deviation (SD) for Internal Quality Control (IQC)** – maximum suggested value to be used to calculate monitoring (±1SD), warning (±2SD) and action limits (±3SD) for IQC sample
5. **Staff competency** – Dependent on methodology, a combination of repeatability (column 1) and reproducibility (column 2) values for the test are used. Reproducibility can be checked by labs being members of a Proficiency "ring" check scheme. There are two main steps for checking staff competency:
    a. Repeatability – The first step is by repeat testing one sample a minimum of five times, ensuring all results fall within the repeatability value (column 1)
    b. Competency – The second step is by testing a minimum of five different samples, representing a range of values, and comparing the results obtained with those of a trained operator/established value for the sample. Difference between results should not exceed the figure given in column 5

Please refer to the Grain Analyst Training modules available at https://ahdb.talentlms.com/

Interpretation of z-score

$$\text{z-score} = \frac{\text{(lab value – mean value)}}{\text{standard deviation}}$$

Regardless of the sign (+/-):

- **|Z| ≤ 2:** performance is satisfactory
- **2 < |Z| ≤ 3:** is a warning (occurs 1 in 20) and suggest attention to equipment or procedures may be required
- **|Z| > 3**: requires immediate action and requires attention to equipment, procedure or operator

The figures presented in the following tables are based on proficiency testing schemes data provided by end-users where significant amounts of data, often a year's worth, was available. Reference has also been made to standard deviation data published in IOB, EBC, CCAT, BSI and ISO standards.

For a number of tests compromises have had to be made and these are based on the knowledge and experience of members of the Grain Sampling and Analysis Laboratory Working Party. This reflects the fact that the data available from different sources covered different ranges of test results.

Wheat

Commodity / Test	1 Repeatability (r)	2 Reproducibility (R)	3 SD for PT	4 SD for IQC	5 Competency
Moisture					
• Oven	0.20	0.36	0.20	N/A	0.36
• NIR	0.2	0.4	0.20	0.1	0.2
• Meter	0.3	0.5	0.30	0.15	0.3
Specific weight					
• Kern	0.40	0.90	0.40	0.20	0.70
• Other	0.5	1.5	0.70	0.25	0.5
Screenings	See Table 1	See Table 1	See Table 1	N/A	See Table 1
Falling number	See Table 4	See Table 4	See Table 4	See Table 4	See Table 4
Gluten	1.1	2.8	1.0	0.5	2.0
Hardness					
• SKCS	3.1	6.7	2.4	1.6	3.1
Protein					
• Dumas	0.25	0.40	0.14	0.125	0.35
• NIR (Transmission)	0.25	0.48	0.20	0.125	0.25
• NIR (Reflectance)	0.30	0.60	0.20	0.15	0.50
Admixture	See Table 1	See Table 1	See Table 1	N/A	See Table 1
Mycotoxins	No current data available				

Barley

Commodity / Test	1 Repeatability (r)	2 Reproducibility (R)	3 SD for PT	4 SD for IQC	5 Competency
Moisture					
• Oven	0.20	0.45	0.20	N/A	0.45
• NIR	0.2	0.4	0.20	0.1	0.2
• Meter	0.3	0.5	0.30	0.15	0.3
Specific weight					
• Kern	0.40	0.90	0.40	0.20	0.70
• Other	0.6	1.5	0.70	0.3	0.6
Screenings	See Table 1	See Table 1	See Table 1	N/A	See Table 1
Nitrogen					
• Dumas	0.05	0.09	0.03	0.025	0.07
• NIR (Transmission)	0.05	0.09	0.05	0.025	0.05
• NIR (Reflectance)	0.05	0.11	0.05	0.025	0.09
Germination capacity					
• Rapid	See Table 5	See Table 5	See Table 5	N/A	See Table 5
• Peroxide	See Table 5	See Table 5	See Table 5	N/A	See Table 5
• Energy (BRF)	See Table 6	See Table 6	See Table 6	N/A	See Table 6
Admixture	See Table 1	See Table 1	See Table 1	N/A	See Table 1
Mycotoxins	No current data available				

Oilseed rape

Commodity / Test	1 Repeatability (r)	2 Reproducibility (R)	3 SD for PT	4 SD for IQC	5 Competency
Moisture					
• Oven	0.20	0.40	0.20	N/A	0.40
• NIR	0.3	0.5	0.30	0.15	0.3
• Meter	0.3	0.5	0.30	0.15	0.3
Admixture					
• Reference method	See Table 2	See Table 2	See Table 2	N/A	See Table 2
• Mechanical method	See Table 2	See Table 2	See Table 2	N/A	See Table 2
• Manual method	See Table 2	See Table 2	See Table 2	N/A	See Table 2
Oil content					
• Extraction	0.27	1.54	0.54	N/A	1.54
• NMR	0.6	1.5	0.53	0.3	0.6
• NIR	0.6	1.2	0.50	0.3	0.6
Erucic acid (NIR)	See Table 8	See Table 8	See Table 8	See Table 8	See Table 8

Oats

Commodity / Test	1 Repeatability (r)	2 Reproducibility (R)	3 SD for PT	4 SD for IQC	5 Competency
Moisture					
• Oven	0.32	0.54	0.20	N/A	0.54
• NIR	0.2	0.4	0.20	0.15	0.2
• Meter	0.3	0.5	0.30	0.15	0.3
Bulk Density					
• Kern	0.50	1.80	1.0	0.25	1.10
• Other	0.9	1.9	1.0	0.45	0.9
Screenings	See Table 1	See Table 1	See Table 1	N/A	See Table 1
Admixture	See Table 1	See Table 1	See Table 1	N/A	See Table 1
Protein					
• Dumas	0.35	0.83	0.30	0.15	0.54
• NIR (Transmission)	0.51	0.57	0.25	0.25	0.51

Rye

Commodity / Test	1 Repeatability (r)	2 Reproducibility (R)	3 SD for PT	4 SD for IQC	5 Competency
Moisture					
• Oven	0.20	0.36	0.20	N/A	0.36
• NIR	0.2	0.4	0.20	0.1	0.2
• Meter	0.3	0.5	0.30	0.15	0.3
Bulk Density					
• Kern	0.40	0.90	0.40	0.20	0.70
• Other	0.5	1.5	0.70	0.25	0.5
Falling number	See Table 4	See Table 4	See Table 4	See Table 4	See Table 4
Protein					
• Dumas	0.25	0.40	0.14	0.125	0.35
• NIR (Transmission)	0.25	0.48	0.20	0.125	0.25
• NIR (Reflectance)	0.30	0.60	0.20	0.15	0.50
Screenings	See Table 1	See Table 1	See Table 1	N/A	See Table 1
Admixture	See Table 1	See Table 1	See Table 1	N/A	See Table 1

Peas

Commodity / Test	1 Repeatability (r)	2 Reproducibility (R)	3 SD for PT	4 SD for IQC	5 Competency
Moisture					
• Oven	0.30	0.50	0.20	N/A	0.50
• Rapid	0.3	0.8	0.30	0.15	0.3
Waste (and Admixture)	See Table 3	See Table 3	See Table 3	N/A	See Table 3
Cracked seed coats	See Table 3	See Table 3	See Table 3	N/A	See Table 3
Soak test	See Table 3	See Table 3	See Table 3	N/A	See Table 3

Beans

Commodity / Test	1 Repeatability (r)	2 Reproducibility (R)	3 SD for PT	4 SD for IQC	5 Competency
Moisture					
• Oven	0.30	0.50	0.20	N/A	0.50
• Rapid	0.3	0.8	0.30	0.15	0.3
Admixture	See Table 3	See Table 3	See Table 3	N/A	See Table 3
Protein					
• Dumas	0.6	0.6	0.5	0.3	0.6
• Rapid	0.6	0.6	0.5	0.3	0.6
Staining and Colour	See Table 5	See Table 5	See Table 5	See Table 5	See Table 5
Insect Test	See Table 7	See Table 7	See Table 7	See Table 7	See Table 7

Linseed

Commodity / Test	1 Repeatability (r)	2 Reproducibility (R)	3 SD for PT	4 SD for IQC	5 Competency
Moisture					
• Oven	0.20	0.40	0.20	N/A	0.40
• Rapid	0.3	0.5	0.30	0.15	0.3
Admixture	See Table 2	See Table 2	See Table 2	N/A	See Table 2
Oil content					
• Extraction	0.27	1.54	0.54	N/A	1.54
• Other	0.6	1.5	0.54	0.3	0.6

Maize

Commodity / Test	1 Repeatability (r)	2 Reproducibility (R)	3 SD for PT	4 SD for IQC	5 Competency
Moisture					
• Oven	0.30	0.60	0.20	N/A	0.60
• Rapid	0.3	0.8	0.3	0.15	0.3
Admixture	See Table 3	See Table 3	See Table 3	N/A	See Table 3

Balance checks

(Balances are checked with known value weights in the critical range, upper and lower action lines are set using the figures in the table below)

Number of decimal places of balance (dp)	Maximum tolerance for the check weight
1 dp (0.1g)	+/- 0.3g
2 dp (0.01g)	+/- 0.03g
3 dp (0.001g)	+/- 0.003g
4 dp (0.0001g)	+/- 0.0003g

Dispenser checks

(Dispensers are checked by weighing the volume of dispensed water)

(based on 1ml = 1g)

Volume of dispenser	Below weights are based on a tolerance of 5%
100 µl	0.1g +/- 0.005g
200 µl	0.2g +/-0.01g
300 µl	0.3g +/- 0.015g
400 µl	0.4g +/- 0.02g
500 µl	0.5g +/-0.025g
600 µl	0.6g +/-0.03g
700 µl	0.7g +/- 0.035g
1 ml	1g +/-0.05g
For dispensers used with 70% Methanol	
10 ml (70% Methanol)	8.72g +/-0.44g
For dispensers used with Falling Number apparatus	
25 ml	25g +/- 0.20g

Table 1. Wheat, barley, oats, rye screenings and admixture

(Including skinned barley and broken grains)

Result range		1 Repeatability (r)	2 Reproducibility (R)	3 SD for PT	4 SD for IQC	5 Competency
0.0 to 1.0	99.0 to 100	0.4	0.7	0.25	N/A	0.7
1.1 to 2.0	98.0 to 98.9	0.7	1.4	0.50	N/A	1.4
2.1 to 5.0	95.0 to 97.9	1.1	2.1	0.75	N/A	2.1
5.1 to 10.0	90.0 to 94.9	1.4	2.8	1.0	N/A	2.8
10.1 to 20.0	80.0 to 89.9	2.1	4.2	1.5	N/A	4.2
20.1 to 30.0	70.0 to 79.9	3.5	7.1	2.5	N/A	7.1
30.1 to 50.0	50.0 to 69.9	7.1	14.2	5.0	N/A	14.2

Table 2. Oilseeds admixture

Result range	1 Repeatability (r)	2 Reproducibility (R)	3 SD for PT	4 SD for IQC	5 Competency
0.0 to 0.5	0.2	0.4	0.14	N/A	0.4
0.6 to 1.0	0.4	0.8	0.28	N/A	0.8
1.1 to 2.0	0.6	1.8	0.64	N/A	1.8
2.1 to 3.0	0.8	2.4	0.85	N/A	2.4
3.1 to 4.0	1.0	3.0	1.06	N/A	3.0
4.1 to 5.0	1.2	3.6	1.27	N/A	3.6
5.1 to 6.0	1.4	4.2	1.48	N/A	4.2
Over 6.0	1.6	4.8	1.70	N/A	4.8

Table 3. Pea, bean and maize admixture (Including pea insect damage and beans waste (admixture and screenings)).

Result range		1 Repeatability (r)	2 Reproducibility (R)	3 SD for PT	4 SD for IQC	5 Competency
0.0 to 1.0	99.0 to 100	0.4	1.4	0.50	N/A	1.4
1.1 to 2.0	98.0 to 98.9	0.7	2.1	0.75	N/A	2.1
2.1 to 3.5	96.5 to 97.9	1.1	3.5	1.25	N/A	3.5
3.6 to 5.0	95.0 to 96.4	1.4	4.2	1.50	N/A	4.2
5.1 to 10.0	90.0 to 94.9	2.1	5.0	1.75	N/A	5.0
10.1 to 20.0	80.0 to 89.9	3.5	7.1	2.50	N/A	7.1
20.1 to 30.0	70.0 to 79.9	5.3	10.6	3.75	N/A	10.6
30.1 to 50.0	50.0 to 69.9	7.1	14.2	5.00	N/A	14.2

Table 4. Falling number

Result range	1 Repeatability (r)	2 Reproducibility (R)	3 SD for PT	4 SD for IQC	5 Competency
60 to 79	10	10	3.5	5	10
80 to 109	13	21	7.4	7	17
110 to 139	15	30	10.6	8	23
140 to 169	17	38	13.4	9	28
170 to 199	19	46	16.3	10	33
200 to 229	21	54	19.1	11	38
230 to 259	23	62	21.9	12	43
260 to 289	25	70	24.7	13	48
290 to 319	27	78	27.6	14	53
320 to 349	30	86	30.4	15	58
350 to 379	32	94	33.2	16	63
Over 379	40	100	35.3	20	70

Table 5. Barley germinative capacity – peroxide and rapid Beans – Staining and colour

Result range	1 Repeatability (r)	2 Reproducibility (R)	3 SD for PT	4 SD for IQC	5 Competency
100	1	2	0.6	N/A	2
99	1	2	0.8	N/A	2
98	2	3	1.0	N/A	3
97	2	3	1.1	N/A	3
96	3	4	1.3	N/A	4
95	3	4	1.5	N/A	4
94	4	5	1.7	N/A	5
93	5	5	1.9	N/A	5
92	5	6	2.0	N/A	6
91	6	6	2.2	N/A	6
90	7	7	2.4	N/A	7
89	7	7	2.6	N/A	7
88	8	8	2.7	N/A	8
87	9	9	2.9	N/A	9
86	9	9	3.1	N/A	9

Table 6. Barley germinative energy – BRF

Result range	1 Repeatability (r)	2 Reproducibility (R)	3 SD for PT	4 SD for IQC	5 Competency
100	3	3	1.1	N/A	3
99	3	3	1.1	N/A	3
98	4	4	1.4	N/A	4
97	4	5	1.8	N/A	5
96	5	6	2.1	N/A	6
95	5	7	2.5	N/A	7
94	6	7	2.5	N/A	7
93	6	8	2.8	N/A	8
92	6	8	2.8	N/A	8
91	7	9	3.2	N/A	9
90	7	9	3.2	N/A	9
89	7	10	3.5	N/A	10
88	8	10	3.5	N/A	10
87	8	11	3.9	N/A	11
86	8	11	3.9	N/A	11

Table 7. Beans – Insect test (Bruchid Beetle)

Result	1 Repeatability (r)	2 Reproducibility (R)	3 SD for PT	4 SD for IQC	5 Competency
0	5.0	8.6	3.1	N/A	8.6
1	5.7	9.7	3.5	N/A	9.7
2	6.3	10.8	3.9	N/A	10.8
3	7.0	11.9	4.3	N/A	11.9
4	7.5	12.8	4.6	N/A	12.8
5	8.2	13.9	5.0	N/A	13.9
6	8.8	15.0	5.4	N/A	15.0
7	9.3	15.8	5.7	N/A	15.8
8	9.9	16.9	6.1	N/A	16.9
9	10.6	18.0	6.5	N/A	18.0
10	11.2	19.1	6.9	N/A	19.1
11	11.7	20.0	7.2	N/A	20.0
12	12.4	21.1	7.6	N/A	21.1
13	13.0	22.2	8.0	N/A	22.2
14	13.5	23.0	8.3	N/A	23.0
15	14.1	24.1	8.7	N/A	24.1
16	14.8	25.2	9.1	N/A	25.2
17	15.4	26.3	9.5	N/A	26.3
18	16.0	27.2	9.8	N/A	27.2
19	16.6	28.3	10.2	N/A	28.3
20	17.2	29.4	10.6	N/A	29.4
21	17.7	30.2	10.9	N/A	30.2
22	18.4	31.3	11.3	N/A	31.3
23	19.0	32.4	11.7	N/A	32.4
24	19.5	33.3	12.0	N/A	33.3
≥25	20.2	34.4	12.4	N/A	34.4

Table 8. Oilseed Rape – Erucic acid (NIR)

Result range	1 Repeatability (r)	2 Reproducibility (R)	3 SD for PT	4 SD for IQC	5 Competency
0.00 - 1.00	1.0	2.0	0.7	0.4	1.0
1.01 - 3.00	1.5	2.5	0.9	0.5	1.5
3.01 - 6.00	2.0	3.0	1.2	0.7	2.0
>6.01	3.0	4.5	1.6	1.0	3.0