June 2004

Grain testing: Standards for testing

HGCA is the cereals and oilseeds division of the Agriculture and Horticulture Development Board.

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Introduction

One of the objectives of the HGCA Grain Sampling Analysis Project is to improve the agreement of test results across the UK Cereals Industry. Part of the approach to this was to establish standards for all grain testing laboratories. A working party with representatives from all sectors of the industry has developed these standards.

The standard

This document describes the elements that are needed for best laboratory practice. It gives straightforward guidance on the requirements for equipment, methods and people. It is not designed to supersede any quality systems that are already in place and are subject to third party audit and accreditation. However, it will provide a framework for those who do not operate to these standards (see pages 3 and 4).

In addition to the framework it includes targets for testing. They 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 and also limits of deviation from mean/median values or reference values when participating in proficiency test schemes. 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 in page 5 followed by standard values in the table on page 6. The standard for running proficiency testing scheme is given by VAM (Valid Analytical Measurement) and can be found on http://www.vam.org.uk

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.
http://www.nabim.org.uk/millintake.asp
http://www.agindustries.org.uk/document.aspx?fn=load&media_id=2426&publicationId=424
http://www.ukmalt.com/LABS/Labs1.html
http://www.lgc.co.uk/pts_schemes_children.asp?id=53_2_3_4

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.

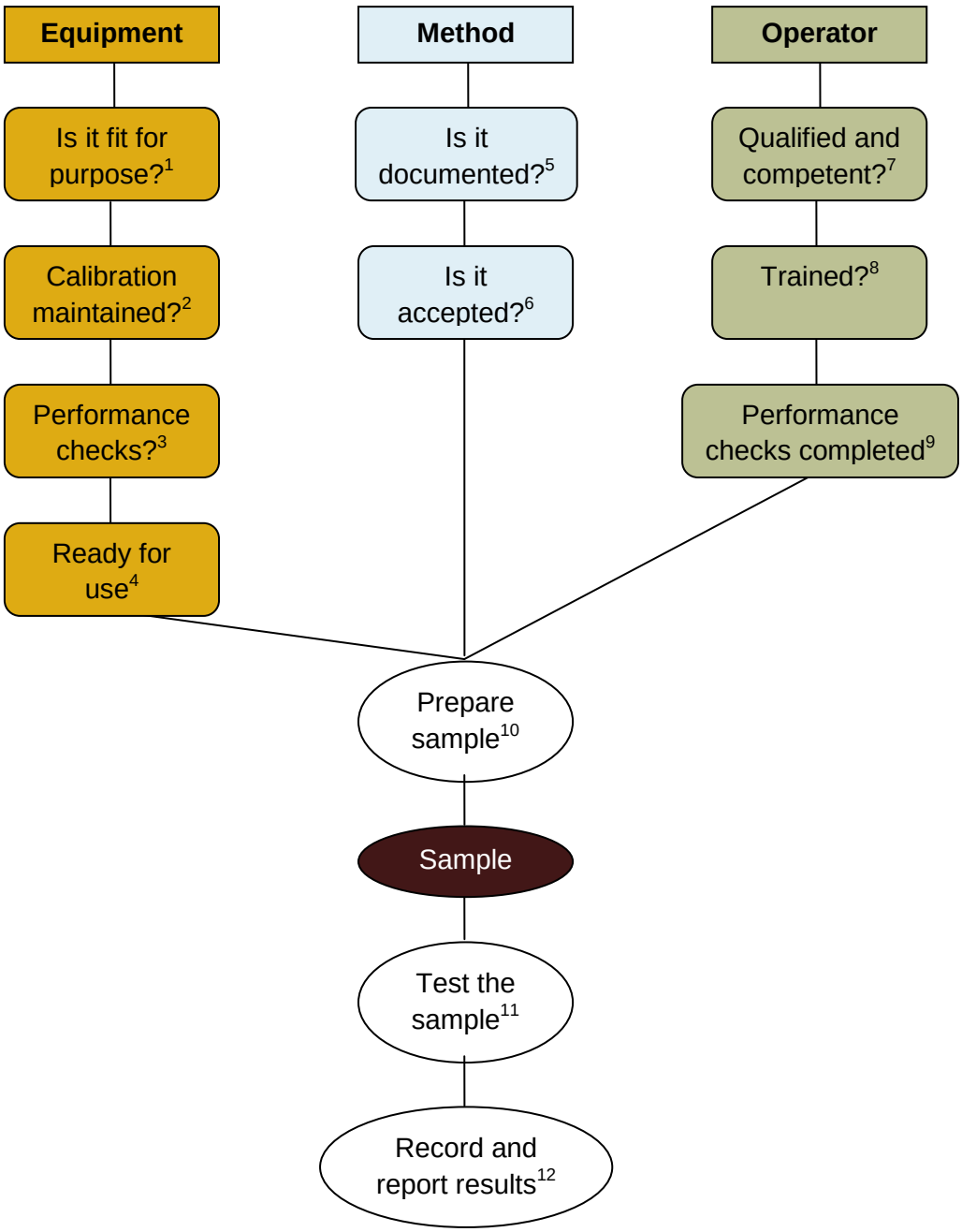
Test	Barley	Wheat
Nitrogen, %	✓	
Moisture content, %	✓	✓
Specific weight, kg/hl	✓	✓
Germinative capacity	✓	
Sieving test >2.5mm; <2.25mm; <2.20mm	✓	
Protein, %		✓
Falling Number, Sec		✓
Hardness, SKCS		✓

Training for analysts

To support the standardisation of training in the UK cereals industry, two training CD ROMs have been produced. When requesting a CD, please quote wheat or barley. These can be obtained from HGCA free of charge:
E-mail publications@hgca.ahdb.org.uk or call 024 7647 8757.

Grain testing best practice

These elements are essential for the reliable testing of grain. See following page for explanation of each element.



The standards to be applied to grain tests are given on page 6.

Notes to accompany each element of grain testing

Equipment

1. All equipment used for testing must be fit for purpose, serviced and maintained in compliance with the laboratory's quality system. Requirements for equipment e.g. balance accuracy, should 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.
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, e.g. 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. Preferably staff will have undertaken the necessary HGCA laboratory training modules.
9. All staff must ensure that any instrument of test performance checks are completed before routine testing can begin.

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.

Standards to be achieved when testing grain

The performance of a laboratory and its staff can be measured in a number of ways.

There are two measure of proficiency:

1. The first is **repeatabilit**y, a measure of the competence of a single analyst in one laboratory. The repeatability r_{95} value (given in the table on page 6) represents the difference between two independent single test results using identical material. *One in 20 results can exceed these values.*
2. The second compares a laboratory with other laboratories testing the same samples. This is usually carried out by using a ring test and operated by proficiency schemes. Laboratories can be judged in two different ways.
    a. Where participants are judged against the other participants in the proficiency scheme. In this case either a mean or median value is used to calculate z-scores. The results are relative to the group taking part in the scheme and are a measure of **reproducibility.** This approach only gives a measure of agreement with others. In these cases the standard deviation of test results is applicable for establishing a value for z and the guideline maximum values are given in the following table column labelled v (versus) mean. Generally the exact method used is not stipulated and hence can be either a reference or secondary method.
    b. Where participants using secondary methods are assessed against a reference test value. In this case, as participants are judged against the agreed reference results for each test there is an element of accuracy included in the scheme. In these cases the **Root Mean Squared Deviation** (RMSD) is applicable and these values are given in the column labelled v reference.

Most proficiency schemes use z-scores to assess proficiency. Values are given in the table to be used for z-score calculation of ring test results. These values are for z=1. For an explanation of z-scores see page 7.

The figures presented in the following table 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, 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.

In addition, for some secondary tests e.g. moisture, protein and nitrogen, it has been necessary to group together a range of equipment.

The figures given on the following table are for guidance and are the maximum values that should be achievable by individual analysts (r_{95}) or maximum values to use for z scores when assessing laboratories. When running proficiency schemes where lower values than those shown can be achieved, the scheme administrators are encouraged to implement these tighter criteria.

Standard values

The repeatability r_{95} values given below should be used to assess the competence of a trainee analyst. The values represent the difference that should not be exceeded for two independent single test results using identical material. Statistically one in 20 results can exceed these values.

When assessing a laboratory's proficiency the values to be used for z-score calculation of the individual laboratory's ring test results are given for the two differing approaches. When comparing with your peers (v mean) or against reference values (v reference). See page 7 for an explanation of z-score calculation. The values given in this table are for s. The values for s given in the table below are for guidance for organisations running proficiency schemes. All schemes should review scheme operations (ideally annually) and set s values that are appropriate. When participants in a scheme perform significantly better than the standard set then **s** values should be reduced accordingly.

Wheat tests	**Repeatability, r_{95}**	**Values of s for z-score calculation**		
		v mean[1]	**v reference[2]**	**dps[3]**
Protein (N*5.7), dm %, Dumas	0.23	0.14	–	1
Protein (N*5.7), dm %, NIR	–[4]	0.22	0.26	1
Moisture %, Oven	0.11	0.20	–	1
Moisture Others[5]	–	0.22	0.24	1
Falling Number, sec	24	26.5	25	0
Specific weight, kg/hl, Kern	0.37	0.30	–	1
Specific weight, kg/hl, Others	–	0.70	0.85	1
Hardness units, SKCS	3.1	2.4	–	1
Hardness SKCS units, NIR	–	–	–	–
Gluten %, wet weight	4.87	3.67	–	1
Barley tests				
Nitrogen dm %, Dumas	0.056	0.030	–	2
Nitrogen dm %, NIR	–	0.033	0.048	2
Moisture %, Oven	0.11	0.20	–	1
Moisture %, NIR	–	0.17	0.29	1
Moisture %, meter	–	0.21	0.32	1
Specific weight, kg/hl	–	0.70	–	1
Germination capacity %, Rapid[6]	5.7	2.4	–	0
Germination capacity %, Peroxide	–	2.2	–	0
Screenings >2.5mm %	–	–	1.00	1
Screenings <2.25mm %	–	–	0.35	1
Screenings <2.2mm %	–	–	0.30	1

[1] Based on R sd. Applicable to ring tests that assess results against either mean or median values.
[2] Based on RMSD. Applicable to ring tests that assess results against reference method values.
[3] Number of decimal places to be used for test results in proficiency testing and when calculating z scores. When reporting z-scores round to 1dp.
[4] No data available to the GSAP Laboratory Working Party. This applies to all boxes containing a dash (–).
[5] Covers a range of methods incl. NIR and meters.
[6] Applicable to barley with a germination capacity of >90%

Proficiency testing schemes

Using z-scores

Participants results are collated by the scheme administrator and a z-score calculated for each result, using the equation:

$$z = \frac{x_i - assigned\ value}{s}$$

Where x_i is the individual result. The assigned value will be either the median value for that analyte, or the 'established value' derived by reference testing, and s is a target value for the acceptable deviation from the assigned value. Values in the table on page 6 in the column headed v (versus) mean applies to schemes that operate using a mean value whereas those who compare individual test results against a reference (established) values should use values in the column headed v (versus) reference.

In the case of these value for each test has been derived from a very large body of proficiency scheme test data and should be achievable by laboratories.

Z-scores are normally interpreted as follows:
$|Z| \leq 2$ is satisfactory
$2 < |Z| < 3$ is questionable
$|Z| \geq 3$ is unsatisfactory