
GUIDELINE FOR INTERLABORATORY TESTS

Introduction

An essential characteristic of a test method is the precision that may be achieved using the method under certain conditions (refer to the terminology booklet for precision, testing conditions).

Carrying out tests : by a representative number of participating laboratories repeatedly within each participating laboratory on identical samples under agreed conditions

is a suitable procedure to measure the precision of a test method.

This procedure is called an interlaboratory test.

The details of the interlaboratory test (number of participating laboratories, testing

conditions, number of repetitions,...) are expressed as the design of the interlaboratory test.

This document shall serve as a practical guideline in order to :

design,

prepare,

carry out

an interlaboratory test.

For definitions refer to :

- BISFA Terminology booklet

- Corresponding BISFA tests method booklet

- ISO 5725 part 1 – part 3

This guide shall be restricted to test methods, preferably standardised by BISFA (an

example is given in the appendix of the terminology booklet).

1. Initiation and Design of Interlaboratory trial

An Interlaboratory trial may be initiated by any institute, company or normative body

that regularly uses BISFA test methods. Recruitment of participating laboratories may

be done by seeking their participation.

Prior to the invitation a comprehensive layout of the scope has to be established.

This layout will be part of the invitation.

The basic structure of the Interlaboratory trial, as proposed by BISFA, is the n-Factor

fully nested experiment. ISO 5725 refers to factors that contribute to the variation of

test results. The graphic representation of the factors of interest within an

Interlaboratory trial is a straight forward way to lay down and explain the successive steps of operation of the Interlaboratory trial.

In the context of the following three examples please refer to the paragraphs

- Aspects of design of the test
- Aspects of evaluation

on page 5 of this guide.

Example 1 : Determine components of variation of test results caused by participating laboratories, sample, time and estimation of single operator component.

FACTOR

Laboratory i

0 (laboratory) (i = 1 to p)

1 (sample) sample 1 sample 2 sample 3

2 (day) day 1 day 2 day 3 day 4 day 5 day 6

3 (set/repetition) 1st 2nd 1st 2nd 1st 2nd 1st 2nd 1st 2nd 1st 2nd
test result

4 (specimen) (n) (n) (n) (n) (n) (n) (n) (n) (n) (n) (n) (n)

Example 2 : Determine components of variation of test results caused by participating laboratory, sample and time (strictly obeying the rules of reporting data as outlined in the BISFA booklets – ie only test results, which may be the average of several individual observations, are reported).

Example 3 : Determine components of variation of test results caused by participating laboratory, sample, operator and estimation of single operator component.

FACTOR

Laboratory i

FACTOR

Laboratory i

0 (laboratory) (i = 1 to p)

1 (sample) sample 1 sample 2 sample 3

2 (day) day 1 day 2 day 3 day 4 day 5 day 6

test result

3 (set/repetition) 1st 2nd 1st 2nd 1st 2nd 1st 2nd 1st 2nd 1st 2nd

FACTOR

Laboratory i

0 (laboratory) (i = 1 to p)

1 (sample) sample 1 sample 2 sample 3

2 (operator) operator 1 operator 2 operator 1 operator 2 operator 1 operator 2

test result

3 (set/repetition) 1st 2nd 1st 2nd 1st 2nd 1st 2nd 1st 2nd 1st 2nd

test result

4 (specimen) (n) (n) (n) (n) (n) (n) (n) (n) (n) (n) (n) (n)

An essential part of the design of an interlaboratory trial is checking all relevant standards in order to ensure that:

sampling,

sample preparation,

test,
calculation and interpretation of results
will be carried out under standardised procedures.
The initiator of the interlab trial shall send out an invitation (including the
graphical
layout of the design) to possible participants in the laboratory trial.
After studying this invitation, laboratories wishing to participate in the trial shall
communicate their interest to the initiator.

2. Preparation of Interlaboratory trial

Each participating laboratory shall nominate only one contact person who will
be
responsible for communicating to other participating laboratories.
The contact person is required to coordinate the interlaboratory trial within
their own
business organisation.

This shall be achieved through :

- Clear communication of requirements
- Ensuring correct test methods are used
- Providing statistical expertise

The initiator of the laboratory trial or one of the other contact persons, if
agreed, shall
report the terms of reference of the trial to the other contact persons for cross
checking.

This should include :

- a detailed instruction sheet referring to the test method to be used
- any other specific instructions for carrying out the trial (for example : test two
samples on the same day with the same operator...)
- a standard form(at) for the reporting of results

and ensures a consistent approach with sampling, testing and the
interpretation of
results. This is particularly important where a participating laboratory is used
to
dealing with a wide range of test procedures and sampling techniques eg
BISFA,
ISO, ASTM.

One of the contact persons shall be agreed to provide and distribute the
samples for
the trial

One of the contact persons shall be agreed to carry out the evaluation of the
results

The Contact persons from each participating laboratory shall agree :

Operational aspects :

- a realistic timescale for completion of Interlaboratory trial
- data exchange by means of hard copies instead of computer discs
- material to be tested and which participating laboratory will prepare and
distribute
samples
- what to do in case of receiving samples with damaged wrapping

Aspects of design of the test :

- what are the precision conditions to be tested
- what type of experiment (three factor fully nested experiment, two factor fully nested, etc.)
- how many individual tests per observation
- inherent inhomogeneities of presumed identical subsamples with respect to the property of interest especially in the case of destructive testing the design of an interlab trial has to be carried out with respect to inhomogeneous individual samples caused by inhomogeneities within the grand sample, sample handling and sample preparation.
- use the terminology “uncertainty of results” in preference to “uncertainty of measurement” to point out that a number of factors originating from test equipment, environment and also sample contribute to the precision of a test procedure.

Aspects of evaluation :

- to use similar format for all data reporting by all participating laboratories.

Only

the original data have to be reported.

- If an observation is the mean of a number n of individual tests, only the mean is reported.
- the procedure to handle outliers and what other testing techniques shall be used.

In the case of an outlier need to decide whether to cancel data or retest. (If a cell

contains an outlier and no retests are carried out, it is recommended to exclude

all data of the corresponding participating laboratory and level to simplify the evaluations).

- which participating laboratory will do the data evaluations and make the final report.

3. Carrying out the Interlaboratory trial Sampling

The amount of total sample taken, and sampling technique, depends on the property

to be tested and the number of samples required. Generally some spare sample

shall be taken, in the unfortunate event of unintended destruction or loss of a set of subsamples.

In the case of commercial mass it is recommended to sample several (3-4) subsamples at random from the “ready to distribute sample” and test under repeatability conditions to check equilibrium. The results should be within the repeatability limit as determined later with the Interlaboratory trial.

After the Contact persons have agreed on all details and the contact person in

charge of providing samples has checked all participating labs have sufficient correct samples (without damage etc), he gives the signal to start. No tests shall be done prior to that.

Performing the test

According to the agreed documents of relevance.

4. Completing the Interlaboratory trial

Data reporting and verification

Original data are put forward to the Contact person in charge of the evaluation.

Treatment of outliers as agreed. In case of suspect data the other Contact persons

should be contacted.

The Contact persons agree on the data to be further evaluated (Analysis of variation).

Data evaluation

The components of variation shall be determined using a commercial statistical

software capable of doing a multifactor analysis of variance. The Contact person in

charge of the evaluation is responsible for the correct application of the software to

the data (according to the design of the Interlaboratory trial).

Before the final report may be issued, the Contact persons shall agree on the original

data, including the statistical evaluation.

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