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Statistical methods of uncertainty evaluation — Guidance on evaluation of uncertainty using two-factor crossed designs

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National foreword

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 69, *Applications of statistical methods*, Subcommittee SC 6, *Measurement methods and results*.

Introduction

Uncertainty estimation usually requires the estimation and subsequent combination of uncertainties arising from random variation. Such random variation may arise within a particular experiment under repeatability conditions, or over a wider range of conditions. Variation under repeatability conditions is usually characterized as repeatability standard deviation or coefficient of variation; precision under wider changes in conditions is generally termed intermediate precision or reproducibility.

The most common experimental design for estimating the long- and short-term components of variance is the classical balanced nested design of the kind used by ISO 5725-2. In this design, a (constant) number of observations are collected under repeatability conditions for each level of some other factor. Where this additional factor is 'Laboratory', the experiment is a balanced inter-laboratory study, and can be analysed to yield estimates of within-laboratory variance, σ_{r}^2, the between-laboratory component of variance, σ_{L}^2, and hence the reproducibility variance, $\sigma_{\mathrm{R}}^2 = \sigma_{\mathrm{L}}^2 + \sigma_{\mathrm{r}}^2$. Estimation of uncertainties based on such a study is considered by ISO 21748. Where the additional grouping factor is another condition of measurement, however, the between-group term can usefully be taken as the uncertainty contribution arising from random variation in that factor. For example, if several different extracts are prepared from a homogeneous material and each is measured several times, analysis of variance can provide an estimate of the effect of variations in the extraction process. Further elaboration is also possible by adding successive levels of grouping. For example, in an inter-laboratory study the repeatability variance, between-day variance and between-laboratory variance can be estimated in a single experiment by requiring each laboratory to undertake an equal number of replicated measurements on each of two days.

While nested designs are among the most common designs for estimation of random variation, they are not the only useful class of design. Consider, for example, an experiment intended to characterize a reference material, conducted by measuring three separate units of the material in three separate instrument runs, with (say) two observations per unit per run. In this experiment, unit and run are said to be 'crossed'; all units are measured in all runs. This design is often used to investigate variation in 'fixed' effects, by testing for changes which are larger than expected from the within-group or 'residual' term. This particular experiment, for example, could easily test whether there is evidence of significant differences between units or between runs. However, the units are likely to have been selected randomly from a much larger (if ostensibly homogeneous) batch, and the run effects are also most appropriately treated as random. If the mean of all the observations is taken as the estimate of the reference material value, it becomes necessary to consider the uncertainties arising from both run-to-run and unit-to-unit variation. This can be done in much the same way as for the nested designs described previously, by extracting the variances of interest using two-way analysis of variance. In the statistical literature, this is generally described as the use of a random-effects or (if one factor is a fixed effect) mixed-effects model.

Variance component extraction can be achieved by several methods. For balanced designs, equating expected mean squares from classical analysis of variance is straightforward. Restricted (sometimes also called residual) maximum likelihood estimation (REML) is also widely recommended for estimation of variance components, and is applicable to both balanced and unbalanced designs. This Technical Specification describes the classical ANOVA calculations in detail and permits the use of REML.

Note that random effects rarely include all of the uncertainties affecting a particular measurement result. If using the mean from a crossed design as a measurement result, it is generally necessary to consider uncertainties arising from possible systematic effects, including between-laboratory effects, as well as the random variation visible within the experiment, and these other effects can be considerably larger than the variation visible within a single experiment.

This present Technical Specification describes the estimation and use of uncertainty contributions using factorial designs.

Statistical methods of uncertainty evaluation — Guidance on evaluation of uncertainty using two-factor crossed designs

1 Scope

This Technical Specification describes the estimation of uncertainties on the mean value in experiments conducted as crossed designs, and the use of variances extracted from such experiments and applied to the results of other measurements (for example, single observations).

This Technical Specification covers balanced two-factor designs with any number of levels. The basic designs covered include the two-way design without replication and the two-way design with replication, with one or both factors considered as random. Calculations of variance components from ANOVA tables and their use in uncertainty estimation are given. In addition, brief guidance is given on the use of restricted maximum likelihood estimates from software, and on the treatment of experiments with small numbers of missing data points.

Methods for review of the data for outliers and approximate normality are provided.

The use of data obtained from the treatment of relative observations (for example, apparent recovery in analytical chemistry) is included.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3534-1, *Statistics — Vocabulary and symbols — Part 1: General statistical terms and terms used in probability*

ISO 3534-3, *Statistics — Vocabulary and symbols — Part 3: Design of experiments*

3 Terms and definitions

For the purposes of this document, the terms and definitions in ISO 3534-1, ISO 3534-3 and the following apply.

3.1
factor
predictor variable that is varied with the intent of assessing its effect on the response variable

Note 1 to entry: A factor may provide an assignable cause for the outcome of an experiment.

Note 2 to entry: The use of factor here is more specific than its generic use as a synonym for predictor variable.

Note 3 to entry: A factor may be associated with the creation of blocks.

[SOURCE: ISO 3534-3:2013, 3.1.5, modified — cross-references within ISO 3534-3 omitted from Notes to entry]

3.2
level
potential setting, value or assignment of a factor

Note 1 to entry: A synonym is the value of a predictor variable.

