TECHNICAL SPECIFICATION

ISO/TS 23471

First edition
2022-11

Experimental designs for evaluation of uncertainty — Use of factorial designs for determining uncertainty functions

Plans d'expériences pour l'évaluation de l'incertitude — Utilisation de plans factoriels pour la détermination des fonctions d'incertitude

Reference number
ISO/TS 23471:2022(E)

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Published in Switzerland

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Foreword

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This document was prepared by Technical Committee ISO/TC 69, *Applications of statistical methods*, Subcommittee SC 6, *Measurement methods and results*.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

This document has been elaborated in response to the need for standardized single laboratory designs to determine measurement uncertainty (JCGM 100[1]) by means of experiments. It applies in situations where the standard deviation of the observations is not constant but depends on the measurand and where the measurement uncertainty is derived by a top-down approach. This need has been expressed in such areas as consumer protection, food safety, environmental analytics and medical diagnostics.

Uncertainty evaluation usually requires the quantification and subsequent combination of uncertainties arising from random variation and uncertainties associated with corrections. Random variation may arise within a particular experiment under the same conditions, or across a range of conditions. The former kind of variation occurs under repeatability conditions, hence usually being characterised as repeatability standard deviation or repeatability coefficient of variation; precision across a range of conditions is generally termed intermediate precision or reproducibility (ISO 5725 (all parts)[3]).

The most common experimental design for estimating the laboratory variance and the repeatability variance is the ANOVA design described in ISO 5725-2. In this design, an equal number of observations are collected under repeatability conditions for each participating laboratory. Alternative designs for interlaboratory studies, in which other factors are varied in addition to the laboratory factor, are described in ISO 5725-3. Evaluation of uncertainties based on such a study design is discussed in ISO 21748[6]. Similarly, where the observations are not grouped in different laboratories but in groups of different measurement conditions (e.g. different weeks or technicians) within the same laboratory, the between-group variance component can be considered to represent the uncertainty contribution arising from random variation in the measurement condition which the grouping factor represents. For example, if test results are obtained under repeatability conditions once a week, analysis of variance can provide an estimate of the effect of variation between weeks.

While nested designs are among the most common designs for estimating random variation, they are not the only useful class of design. Consider, for example, an experiment conducted by using three instruments, three batches of reagents and three batches of a solid phase extraction (SPE) cartridges, where every possible combination is included in the design for a total 3 × 3 × 3 = 27 runs. As every possible combination has the same number of observations, this design is called balanced, and as factors are not nested within each other, the factors instrument, reagent and SPE cartridge are said to be ‘crossed’. This type of experiment is considered in ISO/TS 17503[5] for the uncertainty evaluation of the mean in two-factor crossed designs. Just as in the case of the nested design, the aim is to extract the variance components corresponding to the three factors. Suitable models are available and are referred to in the statistical literature as random-effects or (if one factor is a fixed effect) mixed-effects models. This approach can be extended to take more than three factors into account. However, if all factor level combinations are included in the design, the corresponding number of measurements can become very high. For example, for five factors, each with three levels, there are already $3^5 = 243$ factor level combinations. If it is necessary to include five or more factors in the experiment, the number of levels should be as low as possible (two levels), and it is recommended to implement an orthogonal design, whereby only a selection of factor level combinations is included.

It is assumed in this document that the measured values are non-negative numbers and that all variance components consist of two parts: one part which is proportional to the level of the measurand and another which is constant across levels. Estimation of variance components can be achieved by several methods. For balanced designs, computing expected mean squares from classical analysis of variance is straightforward. Restricted (sometimes also called residual) maximum likelihood estimation (REML) is widely recommended for estimation of variance components and is applicable to both balanced and unbalanced designs.

Experimental designs for evaluation of uncertainty — Use of factorial designs for determining uncertainty functions

1 Scope

This document specifies experimental procedures and statistical analysis for the determination of measurement uncertainty in situations where the following conditions are fulfilled:

Condition 1: The level of the measurand is non-negative, e.g. concentration level of a contaminant in a sample.

Condition 2: Measurement error consists of two independent components: for one of these components the relative standard deviation is constant (that is, the absolute deviation is proportional to the level of the measurand), whereas for the other component the absolute standard deviation is constant (that is, independent of the level of the measurand).

Condition 3: Samples for different levels of the measurand can be made available; if the level of the measurand is the concentration of a chemical substance, samples could be obtained e.g. by fortifying (spiking) blank samples.

Conditions 1 and 2 are met for most applications of instrumental chemical analyses. Condition 3 can be met for chemical analyses if blank samples are available.

This document can also be used to determine precision data for a particular laboratory for different technicians, different environmental conditions, the same or similar test items, with the same level of the measurand, over a certain period of time.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. 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 3534-2, *Statistics — Vocabulary and symbols — Part 2: Applied statistics*

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

ISO 3534-4, *Statistics — Vocabulary and symbols — Part 4: Survey sampling*

ISO/IEC Guide 98-3, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 3534-1, ISO 3534-2, ISO 3534-3, ISO 3534-4, ISO/IEC Guide 98-3 and the following apply. ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/