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Foreword

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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 $3 \times 3 \times 3 = 27$ 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.

