Designing a multi-component calibration experiment

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The necessity of multivariate design of experiment (DoE) for the efficiency of analysis and subsequent data modeling is commonly recognized [1]. However, in spite of its well-established theory and practice [2], the modern DoE is mainly focused at the optimization problem, i.e. finding a set of experimental parameters producing maximal or sufficiently high value of a chosen merit. The problem of designing an optimal calibration experiment stays almost beyond consideration by the theory of DoE.

One of the practically most important needs of quantitative mixture analysis is simultaneous calibration and prediction of several constituents from the same multivariate measurement, e.g. spectrum, using possibly few designed samples. Examples of this kind are numerous and include: two active ingredients in a tablet, fat and protein in milk, ethanol and glucose in the fermentation medium, to name a few. For robust regression modeling, the analyte concentrations (i.e. factors) in calibration samples should be spread on many levels. This distinction makes the classical experimental designs, typically operating in two to five levels, disadvantageous. Attempts to construct an economical DoE effective in the case of a few factors at many levels are rare [3, 4].

The present lecture reviews existing approaches to the calibration DoE and presents a new one. Suggested diagonal designs for a multi-component calibration experiment provide uncorrelated factor variations in as many levels as the number of samples. An independent validation set is provided by the very design scheme. Practicability of the diagonal designs is illustrated by selected applications.

References

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