

A GENERAL STRATEGY FOR OPTIMIZING SUPERCRITICAL FLUID EXTRACTION OF ORGANOCHLORINE COMPOUNDS

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The optimization of pressure and temperature conditions for supercritical fluid extraction (SFE) is an area of considerable interest to chemists performing environmental analyses. A number of anecdotal "rules" have been developed over the years (e.g., higher density is better, etc.), however, these general approaches often fail for particular matrix/analyte combinations. When confronted with a difficult extraction problem, most chemists will try a trial-and-error approach. Clearly, there is a need for a more systematic approach to the problem. Statistical experimental design strategies represent a well-established formal procedure for understanding multivariate systems. After a relatively few number of experiments (usually < 15), it is possible to perform a mathematical regression on the data to determine optimum conditions. However, most chemists do not have ready access to such software (and have an inherent distrust of anything which involves too much use of "statistics").

Fortunately, there are simple experimental design strategies which do not require special software. The results can be readily calculated and understood. The approach used here employed a two-level, two-factor (2^2) or two-level, three-factor (2^3) factorial design. The accompanying figure shows the eight conditions used for the latter approach. The eight points formed a "cube", representing a simultaneous variation in all variables. The results could be used to calculate the "main effect" of each variable, which could then be used to estimate the optimum conditions.

This presentation will summarize our efforts to use this approach for a variety of applications of environmental interest. For example, we have used this approach to optimize extraction of EPA Method 608 pesticides from Empore™ extraction disks, following extraction of wastewater samples. Polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) have been extracted using the same procedure. The approach has also been successful for extraction of a primary amine hydrochloride pesticide from feed. The general approach represents a quick method for optimizing extraction conditions using a minimum number of experiments.

A Two-Level Three-Factor (2^3) Factorial Design

