A118 Estimating the Uncertainty of Purity Measurements in the Forensic Chemistry Laboratory: Application of the Horwitz Model

Sandra E. Rodriguez-Cruz, PhD, Drug Enforcement Administration, Southwest Laboratory, 2815 Scott Street, Vista, CA 92081*

The goal of this presentation is to illustrate the application of the Horwitz model for the estimation of the uncertainty associated with purity measurements.

This presentation will impact the forensic science community by creating the interest to other forensic chemistry analysts and laboratory personnel involved in the evaluation of measurement uncertainty, as required for accreditation under the ISO 17025:2005 standard.

Accreditation under the ISO/IEC 17025:2005 standard requires testing and calibration laboratories to have established procedures for estimating the uncertainty of their measurements. In order to meet this requirement and those of the American Society of Crime Laboratory Directors/Laboratory Accreditation Board (ASCLD/LAB)-*International* Program, the Drug Enforcement Administration (DEA) Office of Forensic Sciences has established procedures for reporting uncertainty of measurement estimates associated with both weight and purity determinations. This presentation will describe the top-down approach used for the estimation of the uncertainty of measurement associated with the quantitation of controlled substances.

Evaluation of six years (2003-2008) of proficiency testing program (PTP) samples and laboratory data from collaborative studies indicates that the relative standard deviation (RSD) of quantitative measurement among DEA laboratories is a function of analyte concentration, with higher RSD values observed as the concentration of the analyte decreases. This behavior has been previously characterized and documented by Horwitz and collaborators.[1 2] During the evaluation of numerous inter- laboratory studies in the early 1980's, Horwitz observed that an approximately 2-fold increase in RSD occurred for each 100-fold decrease in analyte concentration. Notably, Horwitz's studies also demonstrated that the RSD associated with purity determinations is independent of analyte, matrix, or analytical technique used. Horwitz's empirically-derived equation (equation 1) summarizes the experimental findings and defines the mathematical relationship between RSD and purity, where *C* is the analyte concentration expressed as a decimal fraction, i.e., 100 % = 1.00.

$$\% RSD_H = 2^{1-0.5\log_{10} C}$$

Equation 1 (Horwitz curve) RSD values obtained from DEA PTP samples and collaborative studies performed during 2003-2008 are found to be consistent with the curve defined by the Horwitz model. Furthermore, analysis of the data also indicates that an upper limit for RSD values can be established, regardless of controlled substance, matrix, or analytical technique used, as predicted by Horwitz. This upper limit is defined by equation 2.

$$\% RSD_{2H} = 2^{2-0.5\log_{10} C}$$

Equation 2 (2x the Horwitz curve) Therefore, the Horwitz model provides a reasonable basis for the estimation of the uncertainty of measurement associated with purity analyses performed by DEA laboratories. Uncertainty values reported are concentration dependent, and are calculated using equation 3.

$$U = x \cdot RSD_{2H} \cdot k_{95\%}$$

Equation 3

Where, x is the purity of the analyte, RSD_{2H} is the relative standard deviation (or coefficient of variation) defined by 2 times the Horwitz curve, and k is the coverage factor for a 95% confidence level ($k = 2$).

Results from the historical data evaluated will be presented and the validity of using the Horwitz model as the basis for the uncertainty of purity determinations will be demonstrated. This presentation is expected to be of interest to other forensic chemistry analysts and laboratory personnel involved in the evaluation of measurement uncertainty, as required for accreditation under the ISO 17025:2005 standard.

References:

1 Horwitz W. Evaluation of Methods Used for Regulation of Foods and Drugs. Anal Chem 1982;54(1):67A-76A.

2 Boyer KW, Horwitz W, Albert R. Interlaboratory Variability in Trace Element Analysis. Anal Chem 1985;57:454-459.

Uncertainty, Controlled Substances, Purity