K3 Measurement Uncertainty of GC Method in Determining Ethanol Concentration of In- House Prepared Aqueous Standards for use with Evidentiary Breath Test Instruments

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After attending this presentation, attendees will better understand the principles of ISO "Guide to the Expression of Uncertainty in Measurement" as applied to a specific gas chromatography (GC) method for ethanol chemical measurement. This paper defines the measurement problem, describes the evaluation steps involved, shows the largest sources of uncertainty, and demonstrates how the formal process led to changes to the method that resulted in achieving a combined uncertainty goal.

This presentation will impact the forensic science community by giving an example of how to determine and express the confidence of results as a combined uncertainty based on traceability to a certified reference material (CRM).

The objective of this work was to establish the uncertainty of the concentration of ethanol in aqueous solution standards prepared by our laboratory. The principle goals of this study were: (1) to estimate the combined standard uncertainty of in-house standard solution using a GC method; and (2) to see if it was possible to develop a new GC method with tighter quality controls than available with the present method.

The measurement was the mass concentration of ethanol in water in which the concentration is a function of the uncertainty sources of the batch sampling, the GC method, which included the entire sequence of steps from sample auto-dilution through final GC analysis, the uncertainty of the certified reference material used in determining the linear calibration slope and the uncertainty of the calibration least squares fit process.

The measurement problem was a concentration of ethanol analyte in single sample matrix with a range of analyte concentrations. The GC measurement is calibrated against traceable CRMs. Because the method has been under statistical control, the precision information from previous runs includes the *combined* effect of nearly all of the potential sources of uncertainty. Precision estimates used were over an extended period of time, by different analysts using different equipment and the replicate analysis of several samples was the choice for precision data.

The precision of the measurement was found to vary both proportionally with analyte concentration level (level dependant

contributions of analyte) and a constant related to the calibration regression method that is independent of analyte concentration. The three largest sources of uncertainty were determined to be Calibration Slope (1.2%), GC Analytical (0.6%), and CRM (0.7%). The Combined Standard Uncertainty was calculated as U = 1.6% (95% C.L., K=2).

The results show that the proposed method is suitable to expect GC calibrators (ones above 0.098 g/100ml) to be within 2% (k=2) of their target value vs. the present method of 5%; and the sample solution measurement to be within 2% (k=2) of target vs. the present method of 10% of QCs. Below 0.098 g/100ml concentration, the acceptance criteria will need to be established that is not based on a percentage since the uncertainty is not linearly proportionally to concentration in the extremes.

Based on this study, routine 0.080 g/210L and 0.160 g/210L breath alcohol standard solutions are estimated to have a combined standard uncertainty of < 2 % at a 95% confidence limit.

Measurement Uncertainty, Standards, Gas Chromatography

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