SDPD Forensic Chemistry Section
Forensic Alcohol Analysis – Reestablishment of the Uncertainty of Measurement of Ethanol in Blood By Headspace Gas Chromatography using GC3

Introduction

The previous calculation of the Uncertainty of Measurement (UM) for GC3 was done in 2017. The current evaluation was performed using control chart data collected from November 2019 through March 2021. This incorporates data from 161 runs covering two diluters, four analysts, over 100 different days, 12 internal standard lots, multiple reference material lots, and various room temperatures and times of day. This data also includes both successful and unsuccessful runs (ex: a control was outside of acceptable limits).

Procedure

Each run consists of certified reference material (CRM) calibrators of 0.100g%, 0.200g%, and 0.300g%. A series of CRM controls are run after the line and include at least 0.050g%, 0.080g%, and 0.400g%. In October 2020, a CRM control of 0.150g% was added to the initial set of controls. Additional 0.080g% and 0.150g% CRM controls are run depending on the number of subject samples included.

Defining Factors of Uncertainty of Measurement

In estimating measurement uncertainty, the following factors that affect the measurement were assessed:

- **Reported Accuracy of Certified Reference Material Ethanol Standards:**
  The standards are CRMs with values traceable to NIST Standardized Reference Material. Since various lots were utilized, the following are the highest manufacturer established values (± margin of error is for a 95% confidence interval) for each level.

  1. 0.050g% Standard (± 0.00019)
  2. 0.080g% Standard (± 0.00031)
  3. 0.100g% Standard (± 0.00040)
  4. 0.150g% Standard (± 0.00050)
  5. 0.200g% Standard (± 0.00080)
  6. 0.300g% Standard (± 0.00120)
  7. 0.400g% Standard (± 0.00160)

  The uncertainty of the standards is accounted for in the results collected in the control chart data and is reflected in the measurements of accuracy and precision. These values were not separately included into the overall expanded uncertainty calculations.

- **Accuracy and Linearity:**
  The instrument's ability to measure ethanol levels accurately and throughout a range of values represents the accuracy and linearity. The average difference of each of the four CRM control levels was calculated to determine accuracy and linearity.

- **Precision (repeatability)**
  The instruments ability to consistently deliver the same reading of a known amount represents precision. The standard deviation of each of the four CRM control levels was calculated to determine precision.

- **The sample diluter and the Headspace GC/FID instrument:**
  The uncertainty associated with these aspects of sample preparation and quantitation is accounted for in the results collected in the control chart data and is reflected in the measurements of accuracy and precision. These were not separately included into the overall expanded uncertainty calculations

Calculating the Uncertainty of Measurement

The control chart data was analyzed to calculate the standard deviation and average difference for each control level. A normal distribution was assumed and standard uncertainties were calculated and combined. The combined uncertainty for each level was calculated at a coverage factor of k = 2. Table 1 summarizes these calculations. See Appendix A for calculation sheets.

Table 1

Control level	Run Count	Standard Deviation	Average Difference	Combined Uncertainty	k = 2	k = 2 Percentage
0.050	161	0.000775	-0.001014	0.00128	0.0026	5.1%
0.080	303	0.001281	-0.001676	0.00211	0.0042	5.3%
0.150	186	0.002669	-0.002049	0.00336	0.0067	4.5%
0.400	161	0.005943	0.000128	0.00594	0.0119	3.0%

Reporting the Uncertainty of Measurement

The highest combined uncertainty under a 0.100g% was determined to be +/-0.004g% at the 0.080g% level. The highest combined uncertainty at or above a 0.100g% as determined to be +/-4.5% at the 0.150g% level.

The uncertainty will be expressed as +/-0.004g% for values under 0.100g% and +/-4.5% for values greater than or equal to 0.100g%, at a coverage level of k = 2 and a confidence level of approximately 95%. Both of these values are within the acceptable limits set by Title 17.

Reevaluating Uncertainty of Measurement

The uncertainty of measurement will be re-established whenever any part of the process is altered so that it will affect the measurement quantitation. This will include repair or upgrade of GC headspace instruments with new hardware or new software that would affect quantitation, and repairing or replacing the sample diluter. Additionally, prior to a new analyst being signed

off in blood alcohol analysis, they will be required to run the controls as listed, calculate the uncertainties, and ensure that they are within the established limits.

Analyst _____ Date 3/11/21

Technical Review _____ Date 03521

Administrative Review _____ Date 3/15/21

Quality Assurance Manager _____ Date 3/16/21

Appendix A

Headspace GC3
Under 0.1000%
Acceptable Range: +/- 0.005†

Control	Factor	Value (x)	Standard Uncertainty (u)	Distribution	Relative Contribution*
0.0500	Repeatability[1]	0.00078	0.00078	Normal	43%
	Accuracy and Linearity[2]	0.00101	0.00101	Normal	57%
0.0800	Repeatability[1]	0.00128	0.00128	Normal	43%
	Accuracy and Linearity[2]	0.00168	0.00168	Normal	57%

†As per Title 17
[1]Standard deviation
[2]Average difference
*Each standard uncertainty value divided by the total standard uncertainty multiplied by 100.

Calculation of Combined Standard Uncertainty:

$$U_c = \sqrt{u(repeatability)^2 + u(accuracy\ and\ linearity)^2}$$

Calculation of Expanded Uncertainty:

U = k × *Uc*
U is the expanded uncertainty
k is the coverage factor (approximately 95% when k = 2)

0.0500%

Calculation of Combined Standard Uncertainty:

$$Uc = \sqrt{u(0.00078)^2 + (0.00101)^2} = 0.00128$$

Calculation of Expanded Uncertainty:

$$U = 2 * 0.00128 = 0.0026$$

0.0800%

Calculation of Combined Standard Uncertainty:

$$Uc = \sqrt{u(0.00128)^2 + (0.00168)^2} = 0.00211$$

Calculation of Expanded Uncertainty:

$$U = 2 * 0.00211 = 0.0042$$

The highest calculated expanded uncertainty is within the acceptable range as defined by Title 17, +/- 0.005.

Analyst _____ Date 3/11/21

Technical Review _____ Date 031521

Administrative Review _____ Date 3/15/21

Headspace GC3
0.1000% and Over
Acceptable Range: +/- 5%†

Control	Factor	Value (x)	Standard Uncertainty (u)	Distribution	Relative Contribution*
0.1500	Repeatability[1]	0.002669	0.002669	Normal	57%
	Accuracy and Linearity[2]	0.002049	0.002049	Normal	43%
0.4000	Repeatability[1]	0.00594	0.00594	Normal	98%
	Accuracy and Linearity[2]	0.00013	0.00013	Normal	2%

†As per Title 17
[1]Standard deviation
[2]Average difference
*Each standard uncertainty value divided by the total standard uncertainty multiplied by 100.

Calculation of Combined Standard Uncertainty (0.200):

$$U_c = \sqrt{u(repeatability)^2 + u(accuracy\ and\ linearity)^2}$$

Calculation of Expanded Uncertainty:

U = k × *Uc*
U is the expanded uncertainty
k is the coverage factor (approximately 95% when k = 2)

0.1500%

Calculation of Combined Standard Uncertainty:

$$Uc = \sqrt{u(0.002669)^2 + (0.002049)^2} = 0.00336$$

Calculation of Expanded Uncertainty:

$$U = 2 * 0.00336 = 0.0067 = 4.5\%$$

0.4000%

Calculation of Combined Standard Uncertainty:

$$Uc = \sqrt{u(0.00594)^2 + (0.00013)^2} = 0.00594$$

Calculation of Expanded Uncertainty:

$$U = 2 * 0.00594 = 0.0119 = 3.0\%$$

The highest calculated expanded uncertainty is within the acceptable range as defined by Title 17, +/- 5%.

Analyst _____ Date 3/11/21

Technical Review _____ Date 031521

Administrative Review _____ Date 3/15/21