University of Kentucky
Regulatory Services
College of Agriculture, Food and Environment

HEMP
Proficiency Testing Program

Interpreting Hemp Proficiency Testing Reports

October, 2019

Statistical analysis of data in the Hemp Proficiency Testing Program follows guidelines in ISO 13528 (ISO, 2015). Laboratories are asked to provide the method performed and triplicate results for each sample. Laboratory results are evaluated for trueness and precision. This document presents information on interpreting each of the following reports.

a) Laboratory Trueness Report - Individualized lab report evaluating lab's trueness.
b) Laboratory Precision Report - Individualized lab report evaluating lab's precision.
c) Summary All Labs Trueness - Summary report evaluating trueness of all lab results.
e) Summary All Labs Precision - Summary report evaluating precision of all lab results.
e) Summary Statistics - Summary report comparing analytes and methods.
f) Summary Survey Results - Summary of laboratory responses to questions on methodology.
g) Certificate of Analysis - Analytical results and uncertainties of analytes in samples based on results submitted.

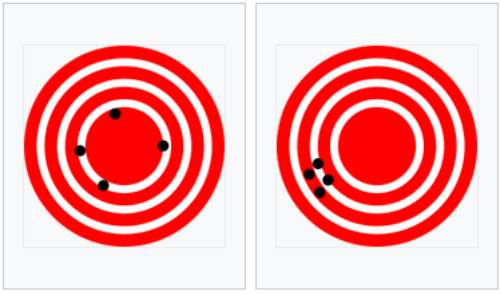
Method Codes, Analytes, and Method Groups

Laboratories report their results and the methods they used. The methods are defined with method codes as shown in Appendix A. Analytes in the program include Δ9-THC, Δ9-THCA, total Δ9-THC, CBD, CBDA, total CBD, and CBN. Laboratories report their results in units of % (w/w) on an as received or dry weight basis. Abbreviations of %AR and %DW are used in the reports to specify concentrations on as received or dry weight basis, respectively.

Laboratory Trueness and Precision Reports

Prior to 1994, accuracy referred to how close an average result was to the true value. This term was modified in ISO 5725 (ISO 1994) to include both the closeness of an average to the true value (trueness) and closeness of repeated results (precision). Trueness replaced accuracy as a term to describe the closeness of an average result to the true value. Both figures below display poor accuracy. The figure on the left has good trueness because the average location of the holes is close to the center target. However, there is poor accuracy because the holes have poor precision. The figure on the right has good precision because the holes are close

to one another. However, there is poor accuracy because the average location of the holes has poor trueness.



Low accuracy due to poor precision

Low accuracy due to poor trueness

Individualized lab reports are prepared that evaluates trueness and precision of lab results. Page 1 of a Laboratory Trueness Report is shown below. The laboratory number and sample identifications are identified in the banner. A table of data is presented for each sample. The three lab results and the average of the three results (Lab Value) are displayed for each method code which defines the analyte and method used to obtain the results

University of Kentucky
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Hemp Proficiency Testing

Laboratory Trueness Report

HEMP Proficiency Testing Program Participant

Lab #: 136 **Samples: HM19SEP** **Issue Date: 10/17/2019**

HM19SEP-1 (hemp)

Method Code	Analyte	Method	Result1	Result2	Result3	Lab Value	Robust Mean (#observations)		Z score	
							Analyte	Method	Analyte	Method
001.10	Δ9-THC (%AR)	LC-UV, other	0.3067	0.3133	0.2866	**0.302**	0.247 (41)	0.24 (18)	**1.79**	**1.62**
002.10	Δ9-THCA (%AR)	LC-UV, other	0.1233	0.1	0.11	**0.111**	0.0384 (35)	0.0419 (15)	**4.39**	**3.03**
003.10	CBD (%AR)	LC-UV, other	4.5766	4.3533	4.0366	**4.32**	5.21 (38)	5.18 (18)	**-2.06**	**-1.57**
004.10	CBDA (%AR)	LC-UV, other	3.1366	2.97	2.7833	**2.96**	3.59 (35)	3.56 (17)	**-1.37**	**-1.20**
005.10	CBN (%AR)	LC-UV, other	0.2866	0.2833	.2700	**0.28**	0.0365 (29)	0.038 (13)	**23.49**	**16.10**
006.10	Total Δ9-THC (%AR)	LC-UV, other	0.4148	0.4010	0.3830	**0.4**	0.286 (52)	0.271 (17)	**2.47**	**2.55**
007.10	Total CBD (%AR)	LC-UV, other	7.3273	6.9579	6.4775	**6.92**	8.42 (39)	8.31 (16)	**-1.85**	**-1.53**

A robust mean and number of observations is displayed for all Lab Values for an analyte regardless of method used (Analyte heading) and all Lab Values for an analyte in a Method (Method heading). Z scores are presented for each of these data sets. A Z score is a measurement of the agreement between the individual lab result and the robust mean considering data distribution of each set. An exact match between Lab Value and Robust Mean would result in a Z score of 0. Lab Values between -2 and +2 are within 2 standard deviations of the data distribution. Lab Values between -3 and +3 are within 3 standard deviations of the data distribution. Lab Values between -2 and +2 are green and considered acceptable. Lab values between -3 and -2 or +2 and +3 are colored orange and are a cautionary warning that the laboratory's procedure should be evaluated. Lab Values less than -3 or greater than +3 are

colored red and are considered unacceptable where action should be taken to correct the laboratory's procedure. A laboratory's proficiency in testing an analyte is evaluated with all values for an Analyte (Analyte Z score). Z scores for Method are for evaluating how a lab performed with other labs using the same method. Appendix B has information on robust statistics and Z score calculations that were used.

Flag indicators will appear in the far right hand column of the report for situations with limited data for statistical analysis. Robust means and Z scores are only calculated with 6 or more observations. Lab value is not used to determine robust means and Z scores if there are less than 2 numeric results for an analyte reported from the lab. A key to the flags is provided at the bottom of the reports when these situations arise. Rules used for reporting nonnumeric values are shown in Appendix C.

The Laboratory Precision Report, as shown below, is very similar to the Laboratory Accuracy Report. Instead of Lab Value, the Precision Report displays the lab's relative standard deviation for repeatability (Lab RSDr) from the three reported results for an analyte. All lab RSDr values are considered for calculating robust mean for all results for the analyte or method. The HorRat(r) value is a ratio of the Lab RSDr value to an expected Horowitz reproducibility value. Any value greater than 0 and less than or equal to 1.3 is in green and considered acceptable. Values that are greater than 1.3 and less than or equal to 4.9 are in orange and are a cautionary warning that the values are high. Values greater than 4.9 are in red and are a heightened warning that the variability of the three results is very high. Appendix B contains details on the calculation of HorRat(r) and an explanation of the warning levels used.

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Hemp Proficiency Testing

Laboratory Precision Report

Lab #: 136 **Samples:** HM19SEP **Issue Date:** 10/17/2019

HM19SEP-1 (hemp)

Method Code	Analyte	Method	Result1	Result2	Result3	Lab RSDr	HorRat(r)	Robust Mean (#observations) Analyte	Robust Mean (#observations) Method
001.10	Δ9-THC (%AR)	LC-UV, other	0.3067	0.3133	0.2866	**4.6**	**0.96**	4.24 (39)	3.29 (17)
002.10	Δ9-THCA (%AR)	LC-UV, other	0.1233	0.1	0.11	**10.5**	**1.9**	10.3 (34)	11.2 (15)
003.10	CBD (%AR)	LC-UV, other	4.5766	4.3533	4.0366	**6.28**	**1.96**	3.38 (36)	3.06 (17)
004.10	CBDA (%AR)	LC-UV, other	3.1366	2.97	2.7833	**5.96**	**1.76**	3.48 (34)	3.1 (16)
005.10	CBN (%AR)	LC-UV, other	0.2866	0.2833	.2700	**3.14**	**0.65**	5.81 (28)	5.43 (13)
006.10	Total Δ9-THC (%AR)	LC-UV, other	0.4148	0.4010	0.3830	**3.99**	**0.87**	3.87 (49)	3.33 (17)
007.10	Total CBD (%AR)	LC-UV, other	7.3273	6.9579	6.4775	**6.16**	**2.06**	3.54 (37)	2.94 (16)

As with the trueness report, flag indicators will appear in the far right hand column of the report for situations with limited data for statistical analysis. Robust Means are only determined with 6 or more observations. A value for RSDr and HorRat(r) is only determined when 3 nonzero numeric results are reported. A key to the flags is provided at the bottom of the reports when these situations arise. Rules used for reporting nonnumeric values are shown in Appendix C.

Summary All Labs Trueness Report

The Summary All Labs Trueness report is a multipage report displaying all lab results grouped by Analyte and Sample Number. Page 1 and 2 of the report is shown on page 5. For each set of Analyte and Sample Number, data is sorted by Lab Value. Z scores are also shown in green, orange, and red colors as described for Laboratory Trueness reports. Flag values other than 0 note Lab Values were not used to calculate robust mean or Z scores due to limited numeric results. This report is useful to determine where an individual Lab Value fell within the range of all Lab Values for an analyte. The report also provides useful information on lower and upper limits used by various labs where results are reported with "<" or ">".

Summary All Labs Precision Report

The Summary All Labs Precision report is a multipage report displaying all lab RSDr values grouped by Analyte and Sample Number. Page 1 and 2 of the report is shown on page 6. HorRat(r) values are shown in green, orange, and red colors as described for Laboratory Precision reports. For each set of Analyte and Sample Number, data is sorted by the HorRat(r) values. Flag values other than 0 note RSDr and HorRat(r) values were not calculated due to limited numeric results. This report is useful to determine where individual Lab RSDr and HorRat(r) values fell within the range of all Lab RSDr and HorRat(r) values for an analyte.

Summary Statistics Report

The Summary Statistics report presents robust means, number of observations (n), and robust standard deviation for Lab Values for trueness and robust means, minimum, and maximum RSDr Values precision. Page 1 of the report is shown on page 7. Robust means, n, and robust standard deviation of Lab Values are presented for an analyte tested by all methods (Analyte), analyte tested by method group such as LC or GC (Method Group), and analyte tested by a specific method (Method). The robust means and standard deviation for trueness in this report are used for determining lab Z scores in the other reports.

This report also shows % relative standard deviation (%RSD) and Horwitz %RSD for trueness. The %RSD is the trueness robust standard deviation divided by the trueness robust mean times 100. Horwitz was a scientist who studied results from several collaborative studies and found %RSD for reproducibility from those data followed the formula shown as

$$\text{Horwitz \%RSD} = 2 \times C^{-0.15}$$

where C is the concentration expressed as a dimensionless mass fraction (eg., C = 0.03 for 3%). The Horwitz %RSD is a benchmark value that the trueness %RSD values can be compared against. A reasonable goal would be to have trueness %RSD values for hemp analysis be approximately equal to or less than the Horwitz %RSD.

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Hemp Proficiency Testing

Summary All Labs Trueness

Samples: HM19SEP | **All Labs Trueness** | **Issue Date: 10/17/2019**

Δ9-THC (%AR)

HM19SEP-1

Sam Num	Analyte	Code	Method	Lab Num	Result1	Result2	Result3	Lab Value	Z score	Population of Lab Values: Rob Mean	n	Rob StDev	Flag
1	Δ9-THC (%AR)	001.01	AOAC 2018.10	107									1
1	Δ9-THC (%AR)	001.99	Other	148	0	0	0	0					2
1	Δ9-THC (%AR)	001.10	LC-UV, other	150	.1317	.1550	.1625	0.15	-3.43	0.248	41	0.0286	0
1	Δ9-THC (%AR)	001.02	AOAC 2018.11,	103	0.1627	0.1663	0.167	0.165	-2.89	0.248	41	0.0286	0
1	Δ9-THC (%AR)	001.10	LC-UV, other	163	0.2733	0.1261		0.2	-1.69	0.248	41	0.0286	0
1	Δ9-THC (%AR)	001.10	LC-UV, other	127	0.2075	0.2186	0.2110	0.212	-1.25	0.248	41	0.0286	0
1	Δ9-THC (%AR)	001.02	AOAC 2018.11,	155	0.218	0.239	0.195	0.217	-1.07	0.248	41	0.0286	0
1	Δ9-THC (%AR)	001.01	AOAC 2018.10	168	0.2180	0.2239	0.2262	0.223	-0.88	0.248	41	0.0286	0
1	Δ9-THC (%AR)	001.10	LC-UV, other	141	0.2462	0.2187	0.2092	0.225	-0.81	0.248	41	0.0286	0
1	Δ9-THC (%AR)	001.10	LC-UV, other	161	0.2260	0.2259	0.2286	0.227	-0.74	0.248	41	0.0286	0
1	Δ9-THC (%AR)	001.30	LC-MS, other	164	0.2408	0.2159	0.2268	0.228	-0.71	0.248	41	0.0286	0
1	Δ9-THC (%AR)	001.30	LC-MS, other	122	0.2117	0.237	0.2442	0.231	-0.6	0.248	41	0.0286	0
1	Δ9-THC (%AR)	001.10	LC-UV, other	125	0.2321	0.2278	0.2348	0.232	-0.57	0.248	41	0.0286	0

Samples: HM19SEP | **All Labs Trueness** | **Issue Date: 10/17/2019**

Sam Num	Analyte	Code	Method	Lab Num	Result1	Result2	Result3	Lab Value	Z score	Population of Lab Values: Rob Mean	n	Rob StDev	Flag
1	Δ9-THC (%AR)	001.10	LC-UV, other	124	0.2367	0.2254	0.2342	0.232	-0.56	0.248	41	0.0286	0
1	Δ9-THC (%AR)	001.10	LC-UV, other	129	0.2297	0.2430	0.2250	0.233	-0.54	0.248	41	0.0286	0
1	Δ9-THC (%AR)	001.30	LC-MS, other	149	0.2241	0.2354	0.2475	0.236	-0.43	0.248	41	0.0286	0
1	Δ9-THC (%AR)	001.30	LC-MS, other	102	0.2459	0.2283		0.237	-0.38	0.248	41	0.0286	0
1	Δ9-THC (%AR)	001.02	AOAC 2018.11,	145	0.2426	0.2330	0.2392	0.238	-0.34	0.248	41	0.0286	0
1	Δ9-THC (%AR)	001.02	AOAC 2018.11,	137	0.2450	0.2360	0.2398	0.24	-0.27	0.248	41	0.0286	0
1	Δ9-THC (%AR)	001.02	AOAC 2018.11,	146	0.2454	0.2405	0.2367	0.241	-0.25	0.248	41	0.0286	0
1	Δ9-THC (%AR)	001.02	AOAC 2018.11,	118	0.231	0.231	0.262	0.241	-0.23	0.248	41	0.0286	0
1	Δ9-THC (%AR)	001.10	LC-UV, other	149	0.2425	0.2422	0.2433	0.243	-0.19	0.248	41	0.0286	0
1	Δ9-THC (%AR)	001.10	LC-UV, other	154	0.258	0.238	0.236	0.244	-0.14	0.248	41	0.0286	0
1	Δ9-THC (%AR)	001.30	LC-MS, other	159	0.238	0.2458	0.2586	0.247	-0.02	0.248	41	0.0286	0
1	Δ9-THC (%AR)	001.10	LC-UV, other	142	0.2453	0.2486	0.2511	0.248	0.01	0.248	41	0.0286	0
1	Δ9-THC (%AR)	001.10	LC-UV, other	101	0.2385	0.2523	0.2548	0.249	0.02	0.248	41	0.0286	0
1	Δ9-THC (%AR)	001.10	LC-UV, other	152	0.2497	0.2492	0.2479	0.249	0.03	0.248	41	0.0286	0
1	Δ9-THC (%AR)	001.02	AOAC 2018.11,	113	0.2486	0.2495	0.2512	0.25	0.06	0.248	41	0.0286	0
1	Δ9-THC (%AR)	001.01	AOAC 2018.10	115	0.2489	0.2528	0.2573	0.253	0.17	0.248	41	0.0286	0
1	Δ9-THC (%AR)	001.30	LC-MS, other	139	0.256	0.2493	0.257	0.254	0.21	0.248	41	0.0286	0
1	Δ9-THC (%AR)	001.10	LC-UV, other	135	0.2521	0.2626	0.2507	0.255	0.25	0.248	41	0.0286	0
1	Δ9-THC (%AR)	001.01	AOAC 2018.10	162	0.2606	0.2540	0.2607	0.258	0.36	0.248	41	0.0286	0
1	Δ9-THC (%AR)	001.30	LC-MS, other	119	0.2646	0.2611	0.2867	0.271	0.79	0.248	41	0.0286	0
1	Δ9-THC (%AR)	001.01	AOAC 2018.10	160	0.26	0.29	0.28	0.277	1	0.248	41	0.0286	0
1	Δ9-THC (%AR)	001.99	Other	173	0.3107	0.2588	0.2622	0.277	1.02	0.248	41	0.0286	0

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Hemp Proficiency Testing

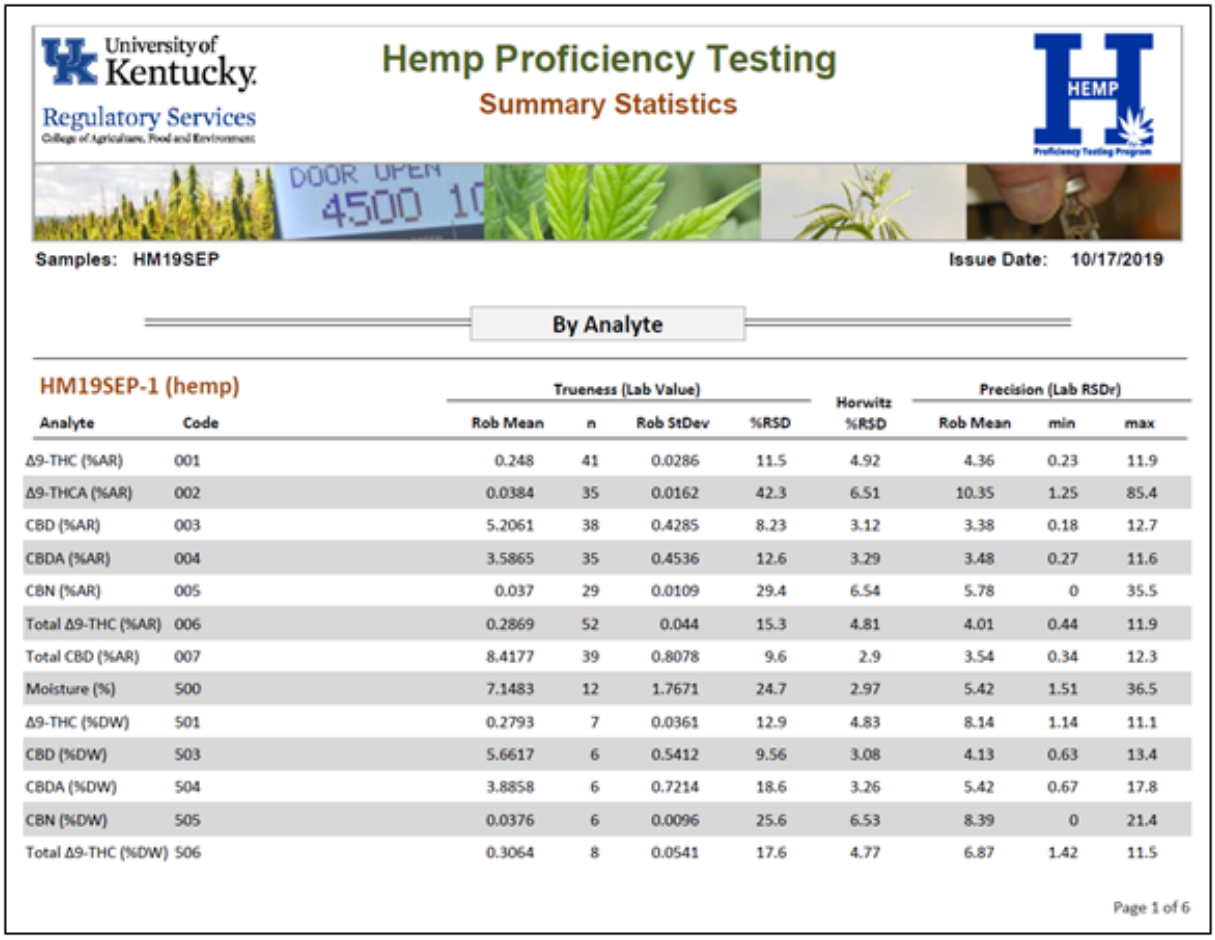
Summary All Labs Precision

Samples: HM19SEP | All Labs Precision | Issue Date: 10/17/2019

Sam Num	Analyte	Code	Method	Lab Num	Result1	Result2	Result3	Lab RSDr	HorRat(r)	Population of Lab RSDr			Flag
										Rob Mean	min	max	
					Δ9-THC (%AR)								
HM19SEP-1													
1	Δ9-THC (%AR)	001.10	LC-UV, other	163	0.2733	0.1261							1
1	Δ9-THC (%AR)	001.99	Other	148	0	0	0						1
1	Δ9-THC (%AR)	001.01	AOAC 2018.10	107									1
1	Δ9-THC (%AR)	001.30	LC-MS, other	102	0.2459	0.2283							1
1	Δ9-THC (%AR)	001.10	LC-UV, other	149	0.2425	0.2422	0.2433	0.23	0.05	4.36	0.23	11.9	0
1	Δ9-THC (%AR)	001.10	LC-UV, other	152	0.2497	0.2492	0.2479	0.37	0.08	4.36	0.23	11.9	0
1	Δ9-THC (%AR)	001.02	AOAC 2018.11,	113	0.2486	0.2495	0.2512	0.53	0.11	4.36	0.23	11.9	0
1	Δ9-THC (%AR)	001.10	LC-UV, other	161	0.2260	0.2259	0.2286	0.68	0.14	4.36	0.23	11.9	0
1	Δ9-THC (%AR)	001.10	LC-UV, other	142	0.2453	0.2486	0.2511	1.17	0.24	4.36	0.23	11.9	0
1	Δ9-THC (%AR)	001.02	AOAC 2018.11,	123	0.2810	0.2840	0.2880	1.24	0.26	4.36	0.23	11.9	0
1	Δ9-THC (%AR)	001.02	AOAC 2018.11,	103	0.1627	0.1663	0.167	1.4	0.27	4.36	0.23	11.9	0
1	Δ9-THC (%AR)	001.01	AOAC 2018.10	162	0.2606	0.2540	0.2607	1.49	0.3	4.36	0.23	11.9	0
1	Δ9-THC (%AR)	001.10	LC-UV, other	125	0.2321	0.2278	0.2348	1.52	0.31	4.36	0.23	11.9	0
1	Δ9-THC (%AR)	001.30	LC-MS, other	139	0.256	0.2493	0.257	1.65	0.34	4.36	0.23	11.9	0

Samples: HM19SEP | All Labs Precision | Issue Date: 10/17/2019

Sam Num	Analyte	Code	Method	Lab Num	Result1	Result2	Result3	Lab RSDr	HorRat(r)	Population of Lab RSDr			Flag
										Rob Mean	min	max	
1	Δ9-THC (%AR)	001.01	AOAC 2018.10	115	0.2489	0.2528	0.2573	1.66	0.34	4.36	0.23	11.9	0
1	Δ9-THC (%AR)	001.02	AOAC 2018.11,	146	0.2454	0.2405	0.2367	1.81	0.37	4.36	0.23	11.9	0
1	Δ9-THC (%AR)	001.01	AOAC 2018.10	168	0.2180	0.2239	0.2262	1.9	0.38	4.36	0.23	11.9	0
1	Δ9-THC (%AR)	001.02	AOAC 2018.11,	137	0.2450	0.2360	0.2398	1.88	0.38	4.36	0.23	11.9	0
1	Δ9-THC (%AR)	001.02	AOAC 2018.11,	145	0.2426	0.2330	0.2392	2.04	0.41	4.36	0.23	11.9	0
1	Δ9-THC (%AR)	001.10	LC-UV, other	124	0.2367	0.2254	0.2342	2.56	0.51	4.36	0.23	11.9	0
1	Δ9-THC (%AR)	001.10	LC-UV, other	135	0.2521	0.2626	0.2507	2.55	0.52	4.36	0.23	11.9	0
1	Δ9-THC (%AR)	001.10	LC-UV, other	127	0.2075	0.2186	0.2110	2.67	0.53	4.36	0.23	11.9	0
1	Δ9-THC (%AR)	001.10	LC-UV, other	101	0.2385	0.2523	0.2548	3.53	0.72	4.36	0.23	11.9	0
1	Δ9-THC (%AR)	001.10	LC-UV, other	116	0.2974	0.3107	0.2896	3.57	0.75	4.36	0.23	11.9	0
1	Δ9-THC (%AR)	001.10	LC-UV, other	129	0.2297	0.2430	0.2250	4.01	0.81	4.36	0.23	11.9	0
1	Δ9-THC (%AR)	001.30	LC-MS, other	159	0.238	0.2458	0.2586	4.2	0.85	4.36	0.23	11.9	0
1	Δ9-THC (%AR)	001.10	LC-UV, other	136	0.3067	0.3133	0.2866	4.6	0.96	4.36	0.23	11.9	0
1	Δ9-THC (%AR)	001.30	LC-MS, other	149	0.2241	0.2354	0.2475	4.97	1	4.36	0.23	11.9	0
1	Δ9-THC (%AR)	001.10	LC-UV, other	154	0.258	0.238	0.236	4.99	1.01	4.36	0.23	11.9	0
1	Δ9-THC (%AR)	001.30	LC-MS, other	119	0.2646	0.2611	0.2867	5.13	1.06	4.36	0.23	11.9	0
1	Δ9-THC (%AR)	001.10	LC-UV, other	106	0.2635	0.2751	0.2917	5.12	1.06	4.36	0.23	11.9	0
1	Δ9-THC (%AR)	001.30	LC-MS, other	164	0.2408	0.2159	0.2268	5.48	1.1	4.36	0.23	11.9	0
1	Δ9-THC (%AR)	001.01	AOAC 2018.10	160	0.26	0.29	0.28	5.52	1.14	4.36	0.23	11.9	0
1	Δ9-THC (%AR)	001.30	LC-MS, other	122	0.2117	0.237	0.2442	7.39	1.49	4.36	0.23	11.9	0
1	Δ9-THC (%AR)	001.02	AOAC 2018.11,	118	0.231	0.231	0.262	7.42	1.5	4.36	0.23	11.9	0
1	Δ9-THC (%AR)	001.10	LC-UV, other	141	0.2462	0.2187	0.2092	8.55	1.71	4.36	0.23	11.9	0



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Hemp Proficiency Testing

Summary Statistics

HEMP
Proficiency Testing Program

Samples: HM19SEP

Issue Date: 10/17/2019

By Analyte

HM19SEP-1 (hemp)

Analyte	Code	Trueness (Lab Value) Rob Mean	n	Rob StDev	%RSD	Horwitz %RSD	Precision (Lab RSDr) Rob Mean	min	max
Δ9-THC (%AR)	001	0.248	41	0.0286	11.5	4.92	4.36	0.23	11.9
Δ9-THCA (%AR)	002	0.0384	35	0.0162	42.3	6.51	10.35	1.25	85.4
CBD (%AR)	003	5.2061	38	0.4285	8.23	3.12	3.38	0.18	12.7
CBDA (%AR)	004	3.5865	35	0.4536	12.6	3.29	3.48	0.27	11.6
CBN (%AR)	005	0.037	29	0.0109	29.4	6.54	5.78	0	35.5
Total Δ9-THC (%AR)	006	0.2869	52	0.044	15.3	4.81	4.01	0.44	11.9
Total CBD (%AR)	007	8.4177	39	0.8078	9.6	2.9	3.54	0.34	12.3
Moisture (%)	500	7.1483	12	1.7671	24.7	2.97	5.42	1.51	36.5
Δ9-THC (%DW)	501	0.2793	7	0.0361	12.9	4.83	8.14	1.14	11.1
CBD (%DW)	503	5.6617	6	0.5412	9.56	3.08	4.13	0.63	13.4
CBDA (%DW)	504	3.8858	6	0.7214	18.6	3.26	5.42	0.67	17.8
CBN (%DW)	505	0.0376	6	0.0096	25.6	6.53	8.39	0	21.4
Total Δ9-THC (%DW)	506	0.3064	8	0.0541	17.6	4.77	6.87	1.42	11.5

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Summary Survey Results

Survey questions were asked of the laboratories regarding details on the methods they used such as extraction solvent, mass of sample, and volume of extractant. This report summarizes the responses from each of the laboratories with each response identified by laboratory number and method the laboratory used for THC analysis.

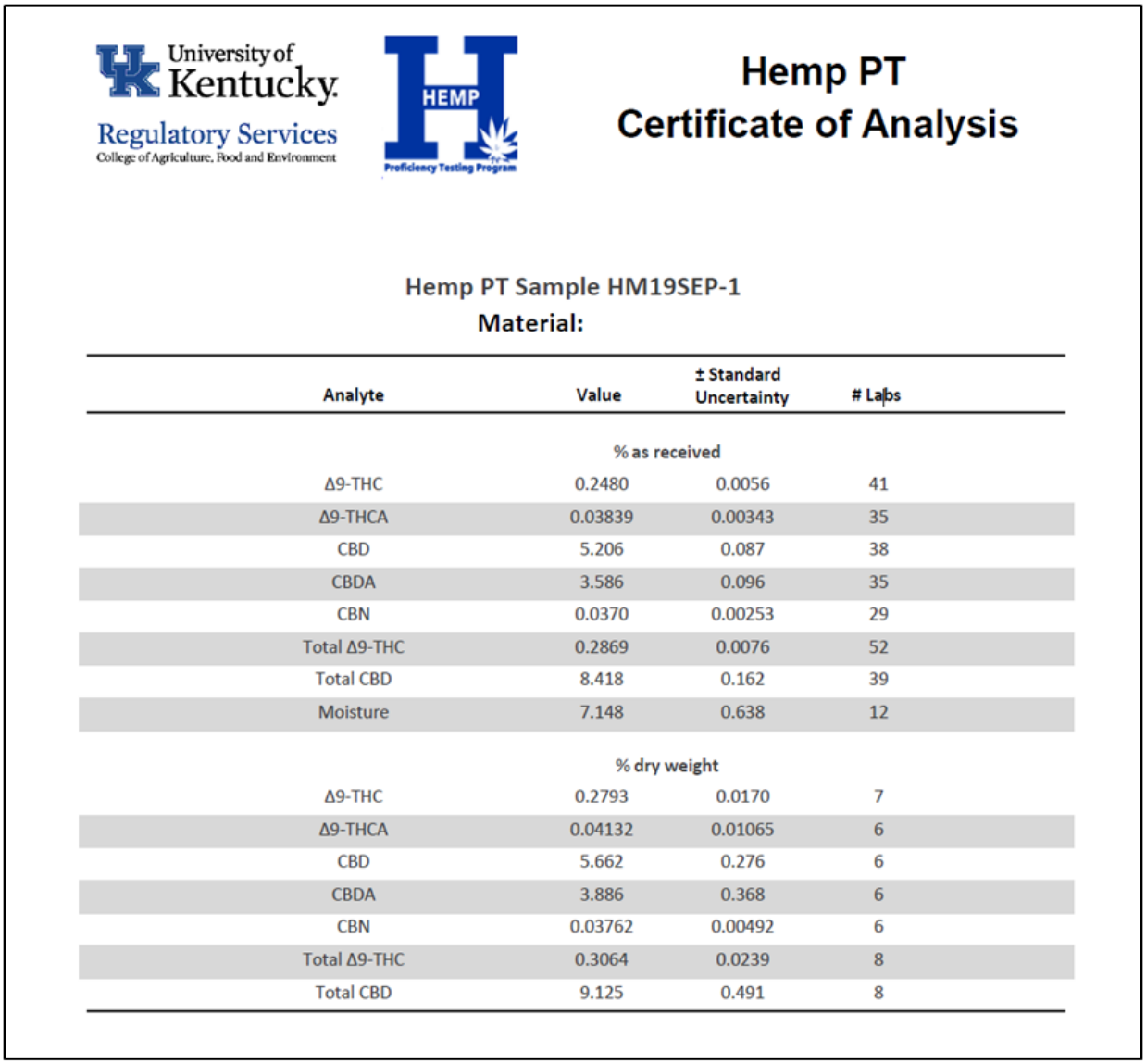
Certificate of Analysis

Samples in the program have a certificate of analysis with concentrations based on results submitted by the laboratories. An example of a certificate of analysis is shown below. The COA presents standard uncertainties for concentration of analytes in the sample.

Standard uncertainty is different from robust standard deviation shown on the Summary Statistics report. The robust standard deviation is a measure of the variability of all results submitted by laboratories. Approximately 67% of the results are within the robust mean ± robust standard deviation. Approximately 95% of the results are within the robust mean ± 2 × robust standard deviation. The standard uncertainty on COA reports is a measure of where the true analyte concentration is expected to be and is calculated using the robust standard deviation (robust stdev) and number of laboratory results (n) as shown below (ISO 13528:2015).

$$\text{Standard Uncertainty} = 1.25 \times \text{robust stdev} / \sqrt{n}$$

Since the number of laboratory results is in the denominator, there is greater certainty on the location of the true analyte concentration with an increased number of laboratory results.



University of Kentucky
Regulatory Services
College of Agriculture, Food and Environment

HEMP
Proficiency Testing Program

Hemp PT
Certificate of Analysis

Hemp PT Sample HM19SEP-1
Material:

Analyte	Value	± Standard Uncertainty	# Labs
	% as received		
Δ9-THC	0.2480	0.0056	41
Δ9-THCA	0.03839	0.00343	35
CBD	5.206	0.087	38
CBDA	3.586	0.096	35
CBN	0.0370	0.00253	29
Total Δ9-THC	0.2869	0.0076	52
Total CBD	8.418	0.162	39
Moisture	7.148	0.638	12
	% dry weight		
Δ9-THC	0.2793	0.0170	7
Δ9-THCA	0.04132	0.01065	6
CBD	5.662	0.276	6
CBDA	3.886	0.368	6
CBN	0.03762	0.00492	6
Total Δ9-THC	0.3064	0.0239	8
Total CBD	9.125	0.491	8

The standard uncertainty can be used to predict where the true concentration lies at different confidence intervals. A 67% confidence interval ranges from the robust mean – standard uncertainty and robust mean + standard uncertainty. An approximate 95% confidence interval ranges from the robust mean – (2 × standard uncertainty) and robust mean + (2 × standard uncertainty).

A laboratory can evaluate their laboratory bias using uncertainty in the Certificate of Analysis. Laboratory bias is one component of measurement uncertainty for an analytical method. Other components of measurement uncertainty include variability in preparing an analytical sample from the laboratory sample and reproducibility of results from the analytical sample. ISO 11352 and NORDTEST (2017) provide detailed information on how to use uncertainties from a proficiency test program to determine bias.

References

AOAC. 2016. Official Methods of Analysis of AOAC INTERNATIONAL. Appendix F: Guidelines for Standard Method Performance Requirements. AOAC INTERNATIONAL, Gaithersburg, MD, USA.

ISO. 1994. Accuracy (trueness and precision) of measurement methods and results – Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method. ISO 5725-2:1994(E). Geneva, Switzerland.

ISO. 2012. Water quality – Estimation of measurement uncertainty based on validation and quality control data. ISO 11352:2012(E).

ISO. 2015. Statistical methods for use in proficiency testing by interlaboratory comparison. ISO 13528:2015(E). Geneva, Switzerland.

Horwitz, W. and R. Albert. 2006. The Horwitz ratio (HorRat): A useful index of method performance with respect to precision. J AOAC Int.: 1095-1099.

NORDTEST. 2017. NORDTEST: Handbook for Calculation of Measurement Uncertainty in Environmental Laboratories, Edition 4 (2017). Available at www.nordtest.info

Rivera, C. and R. Rodriguez. Horwitz Equation as Quality Benchmark in ISO/IEC 17025 Testing Laboratory. http://bii.mx/documentos/horwitzCf11.pdf (verified Dec. 2018).

Thompson, M. 2004. The amazing Horwitz Function. AMC Technical Brief, Royal Society of Chemistry. http://www.rsc.org/images/horwitz-function-technical-brief-17_tcm18-214859.pdf (verified Dec. 2018).

APPENDIX A
List of Method Codes, Analytes, Method Groups and Methods in the Program

The first three numbers in the Method Code identifies the analyte and concentration basis. Values up to 500 are as received % w/w. Values greater than 500 are dry weight % w/w. The last two numbers identifies the method. The methods are grouped according to whether it is liquid chromatography (LC) or gas chromatography (GC).

Method Code	Analyte	Method Group	Conc. Basis (% w/w)	Method; Description
001.01	Δ9-THC	LC	as received	AOAC 2018.10; LC, UV detection
001.02	Δ9-THC	LC	as received	AOAC 2018.11, diode array; LC, UV diode array detection
001.03	Δ9-THC	LC	as received	AOAC 2018.11, mass spec; LC, mass spec detection
001.10	Δ9-THC	LC	as received	LC-UV, other; other LC, UV detection
001.30	Δ9-THC	LC	as received	LC-MS, other; other LC, mass spec detection
001.99	Δ9-THC	none	as received	Other;
002.01	Δ9-THCA	LC	as received	AOAC 2018.10; LC, UV detection
002.02	Δ9-THCA	LC	as received	AOAC 2018.11, diode array; LC, UV diode array detection
002.03	Δ9-THCA	LC	as received	AOAC 2018.11, mass spec; LC, mass spec detection
002.10	Δ9-THCA	LC	as received	LC-UV, other; other LC, UV detection
002.30	Δ9-THCA	LC	as received	LC-MS, other; other LC, mass spec detection
002.99	Δ9-THCA	none	as received	Other;
003.01	CBD	LC	as received	AOAC 2018.10; LC, UV detection
003.02	CBD	LC	as received	AOAC 2018.11, diode array; LC, UV diode array detection
003.03	CBD	LC	as received	AOAC 2018.11, mass spec; LC, mass spec detection
003.10	CBD	LC	as received	LC-UV, other; other LC, UV detection
003.30	CBD	LC	as received	LC-MS, other; other LC, mass spec detection
003.99	CBD	none	as received	Other;
004.01	CBDA	LC	as received	AOAC 2018.10; LC, UV detection
004.02	CBDA	LC	as received	AOAC 2018.11, diode array; LC, UV diode array detection
004.03	CBDA	LC	as received	AOAC 2018.11, mass spec; LC, mass spec detection
004.10	CBDA	LC	as received	LC-UV, other; other LC, UV detection
004.30	CBDA	LC	as received	LC-MS, other; other LC, mass spec detection
004.99	CBDA	none	as received	Other;
005.01	CBN	LC	as received	AOAC 2018.10; LC, UV detection
005.02	CBN	LC	as received	AOAC 2018.11, diode array; LC, UV diode array detection
005.03	CBN	LC	as received	AOAC 2018.11, mass spec; LC, mass spec detection
005.10	CBN	LC	as received	LC-UV, other; other LC, UV detection
005.20	CBN	LC	as received	LC-MS, other; other LC, mass spec detection
005.30	CBN	GC	as received	GC-FID; GC, flame ionization detection
005.40	CBN	GC	as received	GC-MS; GC, mass spec detection

Method Code	Analyte	Method Group	Conc. Basis (% w/w)	Method; Description
005.99	CBN	none	as received	Other;
006.01	Total Δ9-THC	LC	as received	AOAC 2018.10; LC, UV detection, %Δ9-THC+(%Δ9-THCA x 0.877)
006.02	Total Δ9-THC	LC	as received	AOAC 2018.11, diode array; LC, UV diode array detection, %Δ9-THC+(%Δ9-THCA x 0.877)
006.03	Total Δ9-THC	LC	as received	AOAC 2018.11, mass spec; LC, mass spec detection, %Δ9-THC+(%Δ9-THCA x 0.877)
006.10	Total Δ9-THC	LC	as received	LC-UV, other; other LC, UV detection, %Δ9-THC+(%Δ9-THCA x 0.877)
006.30	Total Δ9-THC	LC	as received	LC-MS, other; other LC, mass spec detection, %Δ9-THC+(%Δ9-THCA x 0.877)
006.40	Total Δ9-THC	GC	as received	GC-FID; GC, flame ionization detection
006.50	Total Δ9-THC	GC	as received	GC-MS; GC, mass spec detection
006.99	Total Δ9-THC		as received	Other;
007.01	Total CBD	LC	as received	AOAC 2018.10; LC, UV detection, %CBD+(%CBDA x 0.877)
007.02	Total CBD	LC	as received	AOAC 2018.11, diode array; LC, UV diode array detection, %CBD+(%CBDA x 0.877)
007.03	Total CBD	LC	as received	AOAC 2018.11, mass spec; LC, mass spec detection, %CBD+(%CBDA x 0.877)
007.10	Total CBD	LC	as received	LC-UV, other; other LC, UV detection, %CBD+(%CBDA x 0.877)
007.30	Total CBD	LC	as received	LC-MS, other; other LC, mass spec detection, %CBD+(%CBDA x 0.877)
007.40	Total CBD	GC	as received	GC-FID; GC, flame ionization detection
007.50	Total CBD	GC	as received	GC-MS; GC, mass spec detection
007.99	Total CBD	none	as received	Other;
500.20	Moisture	none	dry weight	AOAC 2001.12, Method I; Karl-Fisher, Extraction into Methanol-Formamide
500.30	Moisture	none	dry weight	AOAC 2001.12, Method II; Karl-Fisher, Boiling Methanol Extraction
500.40	Moisture	none	dry weight	AOAC 934.01; Loss on Drying, 95 to 100 C under pressure
500.50	Moisture	none	dry weight	AOAC 930.15; Loss on Drying, 135 C for 2 hours
500.99	Moisture	none	dry weight	Other;
501.01	Δ9-THC	LC	dry weight	AOAC 2018.10; LC, UV detection
501.02	Δ9-THC	LC	dry weight	AOAC 2018.11, diode array; LC, UV diode array detection
501.03	Δ9-THC	LC	dry weight	AOAC 2018.11, mass spec; LC, mass spec detection
501.10	Δ9-THC	LC	dry weight	LC-UV, other; other LC, UV detection
501.30	Δ9-THC	LC	dry weight	LC-MS, other; other LC, mass spec detection
501.99	Δ9-THC	none	dry weight	Other;
502.01	Δ9-THCA	LC	dry weight	AOAC 2018.10; LC, UV detection
502.02	Δ9-THCA	LC	dry weight	AOAC 2018.11, diode array; LC, UV diode array detection

Method Code	Analyte	Method Group	Conc. Basis (% w/w)	Method; Description
502.03	Δ9-THCA	LC	dry weight	AOAC 2018.11, mass spec; LC, mass spec detection
502.10	Δ9-THCA	LC	dry weight	LC-UV, other; other LC, UV detection
502.30	Δ9-THCA	LC	dry weight	LC-MS, other; other LC, mass spec detection
502.99	Δ9-THCA		dry weight	Other;
503.01	CBD	LC	dry weight	AOAC 2018.10; LC, UV detection
503.02	CBD	LC	dry weight	AOAC 2018.11, diode array; LC, UV diode array detection
503.03	CBD	LC	dry weight	AOAC 2018.11, mass spec; LC, mass spec detection
503.10	CBD	LC	dry weight	LC-UV, other; other LC, UV detection
503.30	CBD	LC	dry weight	LC-MS, other; other LC, mass spec detection
503.99	CBD	none	dry weight	Other;
504.01	CBDA	LC	dry weight	AOAC 2018.10; LC, UV detection
504.02	CBDA	LC	dry weight	AOAC 2018.11, diode array; LC, UV diode array detection
504.03	CBDA	LC	dry weight	AOAC 2018.11, mass spec; LC, mass spec detection
504.10	CBDA	LC	dry weight	LC-UV, other; other LC, UV detection
504.30	CBDA	LC	dry weight	LC-MS, other; other LC, mass spec detection
504.99	CBDA	none	dry weight	Other;
505.01	CBN	LC	dry weight	AOAC 2018.10; LC, UV detection
505.02	CBN	LC	dry weight	AOAC 2018.11, diode array; LC, UV diode array detection
505.03	CBN	LC	dry weight	AOAC 2018.11, mass spec; LC, mass spec detection
505.10	CBN	LC	dry weight	LC-UV, other; other LC, UV detection
505.20	CBN	LC	dry weight	LC-MS, other; other LC, mass spec detection
505.30	CBN	GC	dry weight	GC-FID; GC, flame ionization detection
505.40	CBN	GC	dry weight	GC-MS; GC, mass spec detection
505.99	CBN	none	dry weight	Other;
506.01	Total Δ9-THC	LC	dry weight	AOAC 2018.10; LC, UV detection, %Δ9-THC+(%Δ9-THCA x 0.877)
506.02	Total Δ9-THC	LC	dry weight	AOAC 2018.11, diode array; LC, UV diode array detection, %Δ9-THC+(%Δ9-THCA x 0.877)
506.03	Total Δ9-THC	LC	dry weight	AOAC 2018.11, mass spec; LC, mass spec detection, %Δ9-THC+(%Δ9-THCA x 0.877)
506.10	Total Δ9-THC	LC	dry weight	LC-UV, other; other LC, UV detection, %Δ9-THC+(%Δ9-THCA x 0.877)
506.30	Total Δ9-THC	LC	dry weight	LC-MS, other; other LC, mass spec detection, %Δ9-THC+(%Δ9-THCA x 0.877)
506.40	Total Δ9-THC	GC	dry weight	GC-FID; GC, flame ionization detection
506.50	Total Δ9-THC	GC	dry weight	GC-MS; GC, mass spec detection
506.99	Total Δ9-THC	none	dry weight	Other;
507.01	Total CBD	LC	dry weight	AOAC 2018.10; LC, UV detection, %CBD+(%CBDA x 0.877)

Method Code	Analyte	Method Group	Conc. Basis (% w/w)	Method; Description
507.02	Total CBD	LC	dry weight	AOAC 2018.11, diode array; LC, UV diode array detection, %CBD+(%CBDA x 0.877)
507.03	Total CBD	LC	dry weight	AOAC 2018.11, mass spec; LC, mass spec detection, %CBD+(%CBDA x 0.877)
507.40	Total CBD	GC	dry weight	GC-FID; GC, flame ionization detection
507.50	Total CBD	GC	dry weight	GC-MS; GC, mass spec detection
507.99	Total CBD	none	dry weight	Other;

APPENDIX B
Statistics used to evaluate trueness and precision

Trueness of Lab Values, as the average of three lab results for each analyte, was evaluated with robust statistics and Z scores. Proficiency test data often include outliers which can cause a misleadingly large spread in a bell curve used to evaluate lab values. There are several methods outlined in ISO 17025 (ISO, 2015) to analyze data with outliers to avoid the large spread and to achieve a more reasonable bell curve to evaluate lab values. The approach used in this Proficiency Program is Algorithm A found on page 53 of ISO 13528 (ISO, 2015). The method is an iterative process where outliers are adjusted to values closer to the central value and new mean and standard deviations are calculated. The process continues until the differences between old and new mean and standard deviations are minimal. The mean and standard deviations from this procedure are given the adjective "robust" to differentiate them from commonly used calculations for mean and standard deviation. The average of three results (Lab Value) was considered in robust statistic calculations. Calculations were only performed if there were 6 or more observations.

Z score to evaluate trueness is determined using the robust mean and standard deviation as shown below.

Z score = (LabValue – robust mean) / standard deviation

A Z score of -1 or +1 means the difference between the Lab Value and robust mean is equal to 1 standard deviation. A Z score of -2 or +2 means the difference between the Lab Value and robust mean is equal to 2 standard deviations, and so forth. The greater the absolute magnitude of the Z score, the further away the Lab Value is from the robust mean and the center of the bell curve.

Z scores between -2 and +2 are colored green and considered acceptable. Lab values between -3 and -2 or +2 and +3 are colored orange and are a cautionary warning that the laboratory's procedure should be evaluated. Lab Values less than -3 or greater than +3 are colored red and are considered unacceptable where action should be taken to correct the laboratory's procedure.

Precision of the three results submitted from a laboratory was evaluated using Horwitz formulas (AOAC, 2016 and Horowitz and Albert, 2006). Relative standard deviation for repeatability (RSDr) was determined using standard deviation and average of the three results as shown below.

RSDr = (standard deviation) / average × 100

Horowitz found the following formula to describe reproducibility (R) among lab results in many interlaboratory studies.

Horwitz %RSD = $2 \times C^{-0.15}$

The symbol C is the concentration expressed as a dimensionless mass fraction (eg., C = 0.03 for 3%). The ratio of RSDr to Horowitz %RSD is the Horwitz ratio for repeatability (HorRat(r)).

HorRat(r) = RSDr / (Horwitz %RSD)

AOAC advises that this ratio should be between 0.3 and 1.3 (AOAC, 2016). For the Hemp PT program, the upper limit of 1.3 is used to warn users that there repeatability exceeds the guidance from AOAC. The lower limit of 0.3 is not used to avoid warning laboratories that there repeatability is too good. The program assumes that each lab result reported is a single analysis result and does not represent an average of several results which AOAC warns can lead to erroneously low HorRat(r) values. A cautionary limit is imposed for HorRat(r) values of 0. This is a result of exactly the same result obtained in triplicate. The probability of this occurring is highly unlikely.

Another upper limit is used for HorRat(r) to warn laboratories that their values are exceedingly high. From an analysis of HorRat(r) values for all analytes in the 2018 Hemp PT program, 95% of all the data had HorRat(r) value of 4.9 or less.

Any HorRat(r) value greater than 0 or less than or equal to 1.3 is colored green signifying an acceptable value. A HorRat(r) greater than 1.3 and less than or equal to 4.9 is colored orange signifying a warning that the value is above the guidance level from AOAC. A HorRat(r) above 4.9 is colored red signifying a warning that the value is very high compared to the population of all HorRat(r) values from 2018. HorRat(r) values of 0 are colored orange warning laboratories that this result is highly unlikely from three individual lab results.

APPENDIX C
Rules for Nonnumeric Lab Reported Values

Laboratories can report values less than detection or quantitation limit, 0, or nonnumeric entry such as “na”. Entries can also be left blank with laboratory reporting only one or two results rather than three. There can also be a combination of numeric and nonnumeric values for the three results. Only numeric entries greater than zero were considered in the statistical evaluation. A Lab Value was used in statistical analysis if there were two or more numeric results greater than zero. Relative standard deviation for repeatability (RSDr) was calculated and used in statistical analysis if there were three numeric results greater than zero. Flag indicators are present on the Laboratory Reports for instances were Lab Value was not used and RSDr was not calculated.