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Aqueous Film Forming Foam (AFFF)
Reference Materials**

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Introduction

The measurement of per- and polyfluorinated substances (PFAS) in complex environmental matrices has been proven to have significant interlaboratory variability that can affect the confidence of quantitative measurements.¹⁻³ In addition to high variability in measurements, currently there are no EPA published methods for analysis of PFAS in media other than drinking water. Without published methods there is the potential for greater variability in analytical results from laboratory to laboratory. To this end, there have been multiple statements of needs from Department of Defense (DoD) agencies to focus on validation, accuracy, and reproducibility of PFAS data in a variety of matrices. The National Institute of Standards and Technology has provided measurements of PFAS on ten different reference materials, including human serum, human plasma, fish tissue, house dust, soil, and domestic sludge. These materials are useful for the development and validation of methods; however, the concentrations in these reference materials are significantly lower than the concentrations in source materials impacting contaminated DoD sites, such as aqueous film forming foam (AFFF). A SERDP (Strategic Environmental Research and Development Program) project was funded, starting in FY19, to develop an AFFF PFAS reference material (Project code ER18-1664). The goal is to provide a material to help evaluate the ability of laboratories to analyze high concentrations of PFAS in AFFF concentrates.

This summary report documents and discusses the results from the PFAS AFFF interlaboratory exercise. Twelve laboratories responded to the call for participants. Samples were shipped to participants in January 2020 and results were returned to NIST by December 31, 2020.

Materials and Data Treatment

Materials and Sample Preparation

Four different AFFF formulations were shipped to NIST Gaithersburg between March and April of 2019. Fisherbrand cryogenic storage vials (2 mL capacity), lot number 1244025, and were used to bottle the AFFFs. Fifty vials of each AFFF were created. Volumetrically, approximately 1 mL of each AFFF was added to the storage vials. The four materials were labeled as candidate Reference Materials (RMs) 8690 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation I, 8691 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation II, 8692 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation III, and 8693 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation IV. The homogeneity of these materials was assessed by examining PFAS in a stratified random sampling of the materials (n=5) before they were shipped out to participants. Samples were shipped to participants in January 2020 and results were returned to NIST by December 31, 2020.

Thirty-three PFAS were examined in the four candidate RMs. Participants were not required to measure every analyte specified in the study (Table 1) but were asked to provide data for the analytes they could measure. Participants were asked to use their in-house analytical methods to determine the mass fraction of the different PFAS in mg/kg in each of the materials. All values reported by the laboratories are totals, inclusive of the linear and branched isomers.

Table 1. Analytes (measurands) and their acronyms

Measurand ^a	Acronym
Perfluorobutanoic acid	PFBA
Perfluoropentanoic acid	PFPeA
Perfluorohexanoic acid	PFHxA
Perfluoroheptanoic acid	PFHpA
Perfluorooctanoic acid	PFOA
Perfluorononanoic acid	PFNA
Perfluorodecanoic acid	PFDA
Perfluoroundecanoic acid	PFUnA
Perfluorododecanoic acid	PFDoA
Perfluorotridecanoic acid	PFTrA
Perfluorotetradecanoic acid	PFTA
Perfluorobutanesulfonic acid	PFBS
Perfluoropentanesulfonic acid	PFPeS
Perfluorohexanesulfonic acid	PFHxS
Perfluoroheptanesulfonic acid	PFHpS
Perfluorooctanesulfonic acid	PFOS
Perfluorononanesulfonic acid	PFNS
Perfluorodecanesulfonic acid	PFDS
Perfluorododecanesulfonic acid	PFDoS
Perfluorooctanesulfonamide	FOSA
N-Methyl perfluorooctanesulfonamide	NMeFOSA
N-Ethyl perfluorooctanesulfonamide	NEtFOSA
N-Methyl perfluorooctanesulfonamidoacetic acid	NMeFOSAA
N-Ethyl perfluorooctanesulfonamidoacetic acid	NEtFOSAA
N-Methyl perfluorooctanesulfonamidoethanol	NMeFOSE
N-Ethyl perfluorooctanesulfonamidoethanol	NEtFOSE
1H,1H, 2H, 2H-Perfluorohexane sulfonic acid	4:2 FTS
1H,1H, 2H, 2H-Perfluorooctane sulfonic acid	6:2 FTS
1H,1H, 2H, 2H-Perfluorodecane sulfonic acid	8:2 FTS
4,8-dioxa-3H-perfluorononanoic acid	ADONA
Hexafluoropropylene oxide dimer acid	HFPO-DA
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	9Cl-PF3ONS
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUnDS

^a Measurands are totals, inclusive of linear and branched isomers

Data Treatment

Community tables and figures are provided using randomized laboratory codes, with identities only known to NIST and the individual laboratories. The statistical approaches are outlined below for each type of data representation.

Statistics

Data tables and graphs throughout this report contain information about the performance of each laboratory relative to that of the other participants in this study. All calculations were performed in PROLab Plus (QuoData GmbH, Dresden, Germany). The consensus means and standard deviation are calculated according to the robust Q/Hampel method outlined in ISO 13528:2015(E), Annex C.⁴ In the Q/Hampel method, the standard deviation is estimated by means of the Q method, based on the consideration of pairwise absolute difference. The standard deviation is then used for the Hampel estimation of the mean, which is based on the principle of limiting extreme values in the data.

Summary Data Tables

These data tables include a summary of all reported data for a particular analyte (measurand) in a particular AFFF candidate RM. Participants can compare the data for their laboratory to data reported by the other participating laboratories or to the consensus data. Consensus means and standard deviations are calculated using the laboratory means.¹

Graphs

Individual laboratory data (diamonds) are plotted with the individual laboratory standard deviation (rectangles). Laboratories reporting values as “below LOQ” can still be successful in the study if the target value is also below the laboratory LOQ. The black solid line represents the consensus mean, and the green shaded area represents the 95 % confidence interval for the consensus mean, based on the standard error of the consensus mean. The uncertainty in the consensus mean is calculated using the equation below, based on the repeatability standard deviation (s_r), the reproducibility standard deviation (s_R), the number of participants reporting data, and the average number of replicates reported by each participant. The uncertainty about the consensus mean is independent of the range of tolerance.

$$u_{mean} = \sqrt{\frac{s_R^2 - s_r^2}{n_{participants}} + \frac{s_R^2}{n_{participants} \times n_{Average\ Number\ of\ Replicates\ per\ Participant}}}$$

The solid red lines represent the range of tolerance (values that result in an acceptable Z' score, $|Z'| \leq 2$). If the lower limit is below zero, the lower limit has been set to zero. In this view, the relative locations of individual laboratory data and consensus zones with respect to the target zone can be compared easily.

Candidate RM 8690 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation I

For candidate RM 8690 a summary of the data report by the laboratories and the community results is provided in Table 2. The individual analytes are discussed in subsequent sections along with figures.

Table 2. Reported mass fraction of PFAS (mean \pm standard deviation in mg/kg as received) in candidate RM 8690 from participating laboratories

	Individual Results						Community Results	
	LC0003 (n=3)	LC0004 (n=3)	LC0006 (n=3)	LC0007 (n=3)	LC0011 (n=3)	LC0013 (n=3)	Consensus Mean	Consensus Standard Deviation
PFBA	0.707 \pm 0.145	1.25 \pm 0.04	1.33 \pm 0.07	1.37 \pm 0.50	1.11 \pm 0.01	1.20 \pm 0.00	1.18	0.22
PFPeA	1.20 \pm 0.33	1.37 \pm 0.03	1.84 \pm 0.06	1.40 \pm 0.05	1.15 \pm 0.03	1.33 \pm 0.01	1.33	0.16
PFHxA	2.44 \pm 0.57	3.11 \pm 0.16	3.81 \pm 0.28	3.06 \pm 0.46	2.96 \pm 0.08	2.93 \pm 0.06	3.02	0.38
PFHpA	0.487 \pm 0.146	0.830 \pm 0.053	1.52 \pm 0.07	0.457 \pm 0.010	0.928 \pm 0.017	0.859 \pm 0.017	0.808	0.318
PFOA	2.06 \pm 0.48	2.90 \pm 0.04	4.30 \pm 0.44	3.74 \pm 0.62	3.01 \pm 0.12	2.51 \pm 0.19	3.09	0.90
PFNA	NR	< 0.470	NR	0.02 \pm 0.00	0.01 \pm 0.00	NR	0.014	0.003
PFDA	0.190 \pm 0.000	< 0.470	NR	< 0.013	0.01 \pm 0.00	NR	0.098	0.306
PFUnA	0.130 \pm 0.000	< 0.470	NR	< 0.013	NR	NR		
PFDoA	NR	< 0.470	NR	< 0.050	NR	NR		
PFTra	NR	< 0.470	NR	< 0.050	NR	NR		
PFTA	NR	< 0.470	NR	< 0.250	NR	NR		
PFBS	4.18 \pm 0.92	5.25 \pm 0.23	5.58 \pm 0.52	5.42 \pm 0.05	4.81 \pm 0.22	5.14 \pm 0.16	5.06	0.59
PFPeS	6.26 \pm 1.37	3.60 \pm 0.08	7.34 \pm 0.36	4.01 \pm 0.08	4.22 \pm 0.13	3.83 \pm 0.18	4.45	0.84
PFHxS	21.8 \pm 5.0	22.1 \pm 0.6	35.1 \pm 2.9	31.7 \pm 1.4	27.2 \pm 0.9	23.7 \pm 0.6	26.9	7.0
PFHpS	0.657 \pm 0.045	2.73 \pm 0.07	8.54 \pm 1.02	4.22 \pm 0.13	3.28 \pm 0.16	2.51 \pm 0.07	3.38	2.35
PFOS	147 \pm 27	138 \pm 5	157 \pm 10	200 \pm 2	165 \pm 2	185 \pm 4	165	39
PFNS	NR	< 0.470	NR	0.075 \pm 0.004	0.163 \pm 0.012	NR	0.119	0.178
PFDS	0.070 \pm 0.026	< 0.470	NR	0.171 \pm 0.006	0.195 \pm 0.003	NR	0.145	0.064
PFDoS	NR	< 0.470	NR	NR	0.025 \pm 0.004	NR		
FOSA	NR	< 0.470	NR	< 0.013	0.010 \pm 0.001	NR	0.013	0.009
NMeFOSA	NR	NR	NR	< 0.050	0.001 \pm 0.000	NR		
NEtFOSA	NR	NR	NR	< 0.050	NR	NR		
NMeFOSAA	NR	< 0.470	NR	0.113 \pm 0.016	NR	NR		
NEtFOSAA	NR	< 0.470	NR	< 0.025	NR	NR		
NMeFOSE	NR	NR	NR	4.12 \pm 0.58	NR	NR		
NEtFOSE	NR	NR	NR	< 0.250	NR	NR		
4:2 FTS	NR	< 0.470	NR	< 0.012	0.011 \pm 0.001	NR		
6:2 FTS	0.430 \pm 0.030	1.92 \pm 0.19	2.31 \pm 0.36	1.16 \pm 0.04	1.69 \pm 0.06	3.42 \pm 1.56	1.82	1.18
8:2 FTS	4.02 \pm 1.19	< 0.470	NR	0.222 \pm 0.005	0.313 \pm 0.004	NR	0.268	0.214
ADONA	NR	< 0.470	NR	0.151 \pm 0.031	NR	NR		
HFPO-DA	NR	< 0.470	NR	3.82 \pm 0.20	NR	NR		
9Cl-PF3ONS	NR	< 0.470	NR	NR	NR	NR		
11Cl-PF3OU _n DS	NR	< 0.470	NR	NR	NR	NR		

Values shown as “<” a specified number indicate the actual reporting limit provided by the laboratory

NR: Not Reported

Candidate RM 8691 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation II

For candidate RM 8691 a summary of the data report by the laboratories and the community results is provided in Table 3. The individual analytes are discussed in subsequent sections along with figures.

Table 3. Reported mass fraction of PFAS (mean \pm standard deviation in mg/kg as received) in candidate RM 8691 from participating laboratories

	Individual Results						Community Results	
	LC0003 (n=3)	LC0004 (n=3)	LC0006 (n=3)	LC0007 (n=3)	LC0011 (n=3)	LC0013 (n=3)	Consensus Mean	Consensus Standard Deviation
PFBA	0.067 \pm 0.006	< 0.470	NR	< 1.00	0.071 \pm 0.002	0.066 \pm 0.001	0.068	0.006
PFPeA	0.353 \pm 0.012	< 0.470	0.247 \pm 0.020	0.094 \pm 0.006	0.065 \pm 0.001	0.062 \pm 0.003	0.146	0.078
PFHxA	0.137 \pm 0.015	< 0.470	0.223 \pm 0.004	0.195 \pm 0.014	0.157 \pm 0.002	0.166 \pm 0.006	0.175	0.042
PFHpA	0.030 \pm 0.000	< 0.470	NR	0.021 \pm 0.001	0.009 \pm 0.000	0.055 \pm 0.008	0.029	0.024
PFOA	0.027 \pm 0.006	< 0.470	NR	< 0.096	0.037 \pm 0.002	0.045 \pm 0.002	0.036	0.015
PFNA	NR	< 0.470	NR	< 0.013	0.003 \pm 0.000	0.017 \pm 0.002	0.014	0.004
PFDA	0.197 \pm 0.006	< 0.470	NR	0.024 \pm 0.002	0.021 \pm 0.000	0.020 \pm 0.002	0.022	0.007
PFUnA	0.130 \pm 0.000	< 0.470	NR	0.014 \pm 0.001	0.001 \pm 0.000	NR	0.011	0.028
PFDoA	NR	< 0.470	NR	< 0.050	0.009 \pm 0.001	NR		
PFTTrA	NR	< 0.470	NR	< 0.050	0.001 \pm 0.000	NR		
PFTA	NR	< 0.470	0.029 \pm 0.009	< 0.050	0.005 \pm 0.001	NR	0.017	0.04
PFBS	NR	< 0.470	0.347 \pm 0.026	0.761 \pm 0.665	NR	NR	0.554	0.686
PFPeS	NR	< 0.470	NR	0.029 \pm 0.004	NR	NR		
PFHxS	NR	< 0.470	NR	0.054 \pm 0.014	NR	NR		
PFHpS	NR	< 0.470	0.391 \pm 0.105	< 0.012	NR	NR		
PFOS	NR	< 0.470	1.43 \pm 1.06	< 0.023	NR	NR		
PFNS	NR	< 0.470	NR	< 0.024	NR	NR		
PFDS	NR	< 0.470	NR	< 0.012	NR	NR		
PFDoS	NR	< 0.470	NR	NR	NR	NR		
FOSA	NR	< 0.470	NR	< 0.013	NR	NR		
NMeFOSA	NR	NR	NR	< 0.050	NR	NR		
NEtFOSA	NR	NR	NR	< 0.050	NR	NR		
NMeFOSAA	NR	< 0.470	NR	< 0.100	NR	NR		
NEtFOSAA	NR	< 0.470	NR	< 0.025	NR	NR		
NMeFOSE	NR	NR	NR	< 0.050	NR	NR		
NEtFOSE	NR	NR	NR	< 0.050	NR	NR		
4:2 FTS	NR	< 0.470	NR	0.017 \pm 0.003	0.001 \pm 0.000	NR	0.009	0.031
6:2 FTS	0.630 \pm 0.061	0.563 \pm 0.059	NR	0.629 \pm 0.073	0.697 \pm 0.019	2.02 \pm 0.26	0.630	0.154
8:2 FTS	NR	< 0.470	NR	0.037 \pm 0.006	0.056 \pm 0.006	0.472 \pm 0.129	0.047	0.053
ADONA	NR	< 0.470	NR	< 0.047	NR	NR		
HFPO-DA	NR	< 0.470	NR	< 0.997	NR	NR		
9Cl-PF3ONS	NR	< 0.470	NR	NR	NR	NR		
11Cl-PF3OU _n DS	NR	< 0.470	NR	NR	NR	NR		

Values shown as “<” a specified number indicate the actual reporting limit provided by the laboratory
NR: Not Reported

Candidate RM 8692 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation III

For candidate RM 8692 a summary of the data report by the laboratories and the community results is provided in Table 4. The individual analytes are discussed in subsequent sections along with figures.

Table 4. Reported mass fraction of PFAS (mean \pm standard deviation in mg/kg as received) in candidate RM 8692 from participating laboratories

	Individual Results						Community Results	
	LC0003 (n=3)	LC0004 (n=3)	LC0006 (n=3)	LC0007 (n=3)	LC0011 (n=3)	LC0013 (n=3)	Consensus Mean	Consensus Standard Deviation
PFBA	0.127 \pm 0.023	< 0.470	NR	< 1.000	0.156 \pm 0.001	0.146 \pm 0.003	0.143	0.017
PFPeA	NR	< 0.470	0.193 \pm 0.011	0.034 \pm 0.004	0.011 \pm 0.000	NR	0.066	0.058
PFHxA	0.073 \pm 0.012	< 0.470	NR	0.103 \pm 0.007	0.091 \pm 0.003	0.101 \pm 0.006	0.092	0.019
PFHpA	NR	< 0.470	2.32 \pm 0.09	< 0.050	0.002 \pm 0.000	0.038 \pm 0.004	0.020	0.085
PFOA	NR	< 0.470	NR	< 0.096	NR	NR		
PFNA	NR	< 0.470	NR	< 0.013	NR	NR		
PFDA	NR	< 0.470	NR	0.037 \pm 0.009	NR	NR		
PFUnA	0.130 \pm 0.000	< 0.470	NR	< 0.013	NR	NR		
PFDoA	NR	< 0.470	NR	< 0.025	NR	NR		
PFTra	NR	< 0.470	NR	< 0.050	NR	NR		
PFTA	NR	< 0.470	NR	< 0.050	NR	NR		
PFBS	NR	< 0.470	0.404 \pm 0.037	0.049 \pm 0.008	NR	NR	0.227	0.718
PFPeS	NR	< 0.470	NR	0.023 \pm 0.007	NR	NR		
PFHxS	NR	< 0.470	NR	0.021 \pm 0.005	NR	NR		
PFHpS	NR	< 0.470	NR	< 0.012	NR	NR		
PFOS	NR	< 0.470	4.14 \pm 0.19	0.020 \pm 0.002	NR	NR	2.08	8.75
PFNS	NR	< 0.470	0.144 \pm 0.030	< 0.024	NR	NR		
PFDS	NR	< 0.470	NR	< 0.012	NR	NR		
PFDoS	NR	< 0.470	NR	NR	NR	NR		
FOSA	NR	< 0.470	NR	< 0.013	NR	NR		
NMeFOSA	NR	NR	NR	< 0.050	NR	NR		
NEtFOSA	NR	NR	NR	< 0.050	NR	NR		
NMeFOSAA	NR	< 0.470	NR	< 0.100	NR	NR		
NEtFOSAA	NR	< 0.470	NR	< 0.025	NR	NR		
NMeFOSE	NR	NR	NR	< 0.050	NR	NR		
NEtFOSE	NR	NR	NR	< 0.050	NR	NR		
4:2 FTS	NR	< 0.470	NR	0.019 \pm 0.003	NR	NR		
6:2 FTS	0.357 \pm 0.067	0.573 \pm 0.025	NR	0.292 \pm 0.011	0.503 \pm 0.006	1.28 \pm 0.09	0.527	0.256
8:2 FTS	NR	< 0.470	NR	< 0.012	NR	0.228 \pm 0.114		
ADONA	NR	< 0.470	NR	< 0.047	NR	NR		
HFPO-DA	NR	< 0.470	NR	< 0.997	NR	NR		
9Cl-PF3ONS	NR	< 0.470	NR	NR	NR	NR		
11Cl-PF3OU _n DS	NR	< 0.470	NR	NR	NR	NR		

Values shown as “<” a specified number indicate the actual reporting limit provided by the laboratory

NR: Not Reported

Candidate RM 8693 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation IV

For candidate RM 8693 a summary of the data report by the laboratories and the community results is provided in Table 5. The individual analytes are discussed in subsequent sections along with figures.

Table 5. Reported mass fraction of PFAS (mean \pm standard deviation in mg/kg as received) in candidate RM 8693 from participating laboratories

	Individual Results						Community Results	
	LC0003 (n=3)	LC0004 (n=3)	LC0006 (n=3)	LC0007 (n=3)	LC0011 (n=3)	LC0013 (n=3)	Consensus Mean	Consensus Standard Deviation
PFBA	0.347 \pm 0.296	0.547 \pm 0.015	0.990 \pm 0.018	< 1.000	0.462 \pm 0.012	0.497 \pm 0.045	0.528	0.174
PFPeA	0.443 \pm 0.038	< 0.470	0.586 \pm 0.033	0.183 \pm 0.016	0.142 \pm 0.003	0.161 \pm 0.017	0.260	0.104
PFHxA	1.46 \pm 0.30	1.94 \pm 0.04	3.40 \pm 0.14	1.70 \pm 0.12	1.60 \pm 0.04	1.65 \pm 0.19	1.72	0.58
PFHpA	0.037 \pm 0.006	< 0.470	4.12 \pm 0.24	< 0.050	0.021 \pm 0.001	0.057 \pm 0.010	0.041	0.041
PFOA	NR	< 0.470	NR	< 0.096	NR	NR		
PFNA	NR	< 0.470	NR	< 0.013	NR	NR		
PFDA	NR	< 0.470	NR	< 0.013	NR	NR		
PFUnA	NR	< 0.470	NR	< 0.013	NR	NR		
PFDoA	NR	< 0.470	NR	< 0.050	NR	NR		
PFTTrA	NR	< 0.470	NR	< 0.050	NR	NR		
PFTA	NR	< 0.470	0.030 \pm 0.001	< 0.050	NR	NR		
PFBS	NR	< 0.470	0.547 \pm 0.015	< 0.013	NR	NR	0.282	1.155
PFPeS	NR	< 0.470	NR	< 0.012	NR	NR		
PFHxS	NR	< 0.470	NR	< 0.013	NR	NR		
PFHpS	NR	< 0.470	NR	0.049 \pm 0.002	NR	NR		
PFOS	NR	< 0.470	4.33 \pm 0.25	< 0.012	NR	NR		
PFNS	NR	< 0.470	NR	< 0.012	NR	NR		
PFDS	NR	< 0.470	NR	< 0.012	NR	NR		
PFDoS	NR	< 0.470	NR	NR	NR	NR		
FOSA	NR	< 0.470	NR	< 0.013	NR	NR		
NMeFOSA	NR	NR	NR	< 0.050	NR	NR		
NEtFOSA	NR	NR	NR	< 0.050	NR	NR		
NMeFOSAA	NR	< 0.470	NR	< 0.100	NR	NR		
NEtFOSAA	NR	< 0.470	NR	< 0.025	NR	NR		
NMeFOSE	NR	NR	NR	0.139 \pm 0.008	NR	NR		
NEtFOSE	NR	NR	NR	< 0.050	NR	NR		
4:2 FTS	0.300 \pm 0.066	< 0.470	0.733 \pm 0.030	0.324 \pm 0.008	0.323 \pm 0.026	0.372 \pm 0.052	0.33	0.08
6:2 FTS	37.5 \pm 4.0	128 \pm 2	168 \pm 7	130 \pm 2	152 \pm 0.371	137 \pm 10	133	34
8:2 FTS	NR	NR	250 \pm 9	0.013 \pm 0.001	0.030 \pm 0.001	0.293 \pm 0.017	0.112	0.579
ADONA	NR	< 0.470	NR	< 0.047	NR	NR		
HFPO-DA	NR	< 0.470	NR	< 0.997	NR	NR		
9Cl-PF3ONS	NR	< 0.470	NR	NR	NR	NR		
11Cl-PF3OU _n DS	NR	< 0.470	NR	NR	NR	NR		

Values shown as “<” a specified number indicate the actual reporting limit provided by the laboratory

NR: Not Reported

Individual Analytes

Perfluorobutanoic acid (PFBA)

- Six laboratories reported results for PFBA in candidate RMs 8690 and 8693, while five laboratories reported results for PFBA in candidate RMs 8691 and 8692
- The between-laboratory variability was good for candidate RMs 8690, 8691, and 8692 (19 %, 9 %, and 12 % relative standard deviation (RSD), respectively)
- The between-laboratory variability was poor for candidate RM 8693 (33 % RSD)



Figure 1-1. PFBA in candidate RM 8690 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation I. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

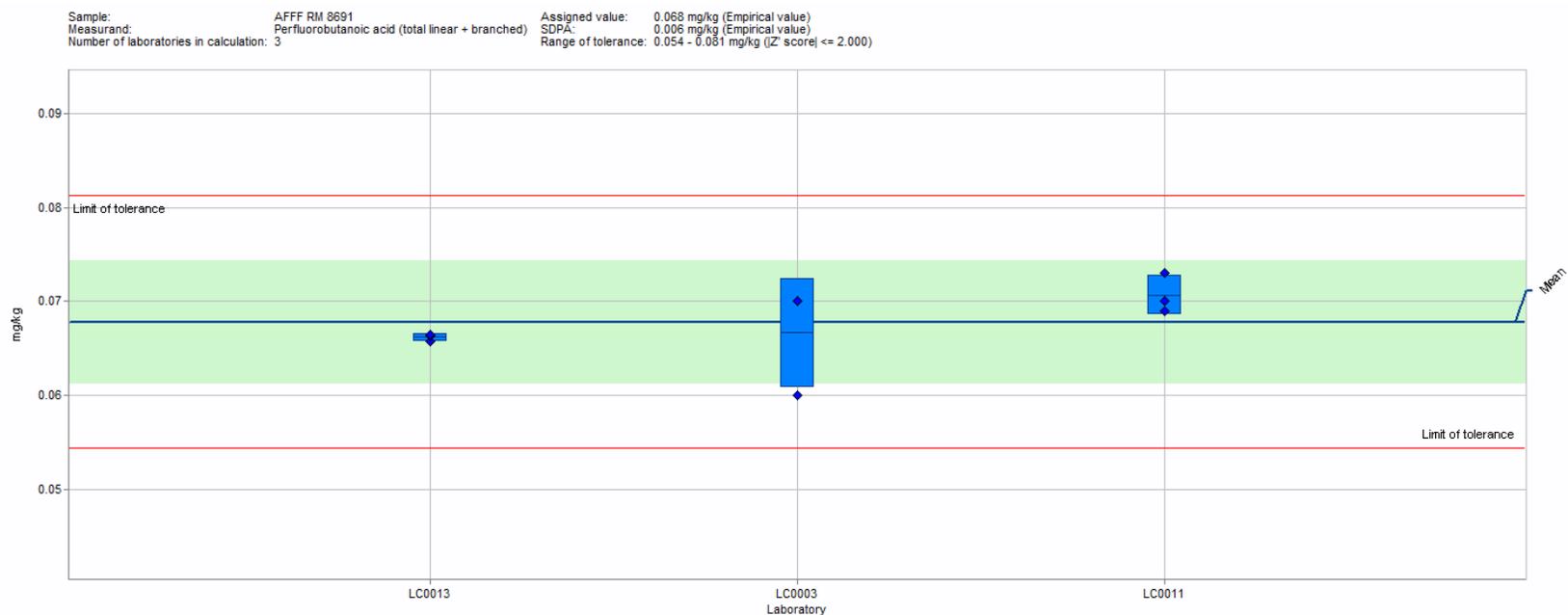


Figure 1-2. PFBA in candidate RM 8691 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation II. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

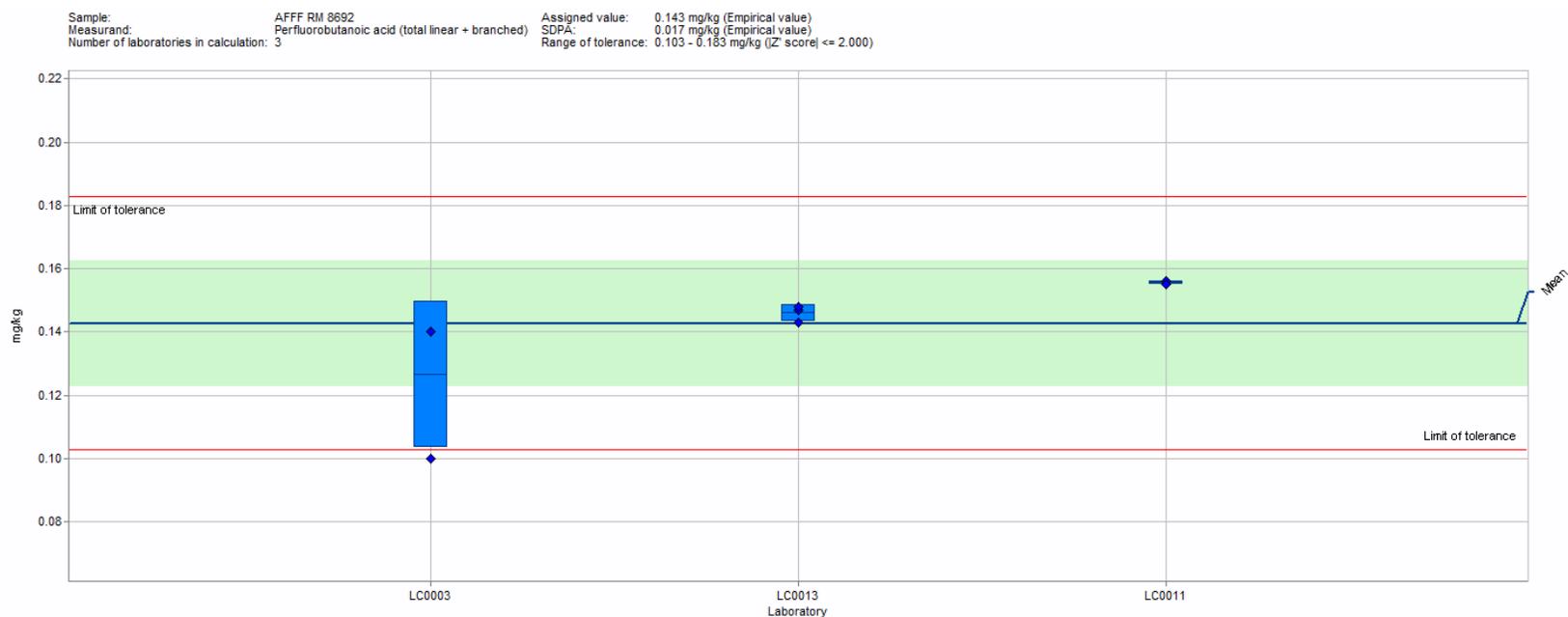


Figure 1-3. PFBA in candidate RM 8692 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation III. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Sample: AFFF RM 8693 Assigned value: 0.528 mg/kg (Empirical value)
 Measurand: Perfluorobutanoic acid (total linear + branched) SDPA: 0.174 mg/kg (Empirical value)
 Number of laboratories in calculation: 5 Range of tolerance: 0.148 - 0.908 mg/kg ($|Z'_{\text{comm}}| \leq 2.000$)

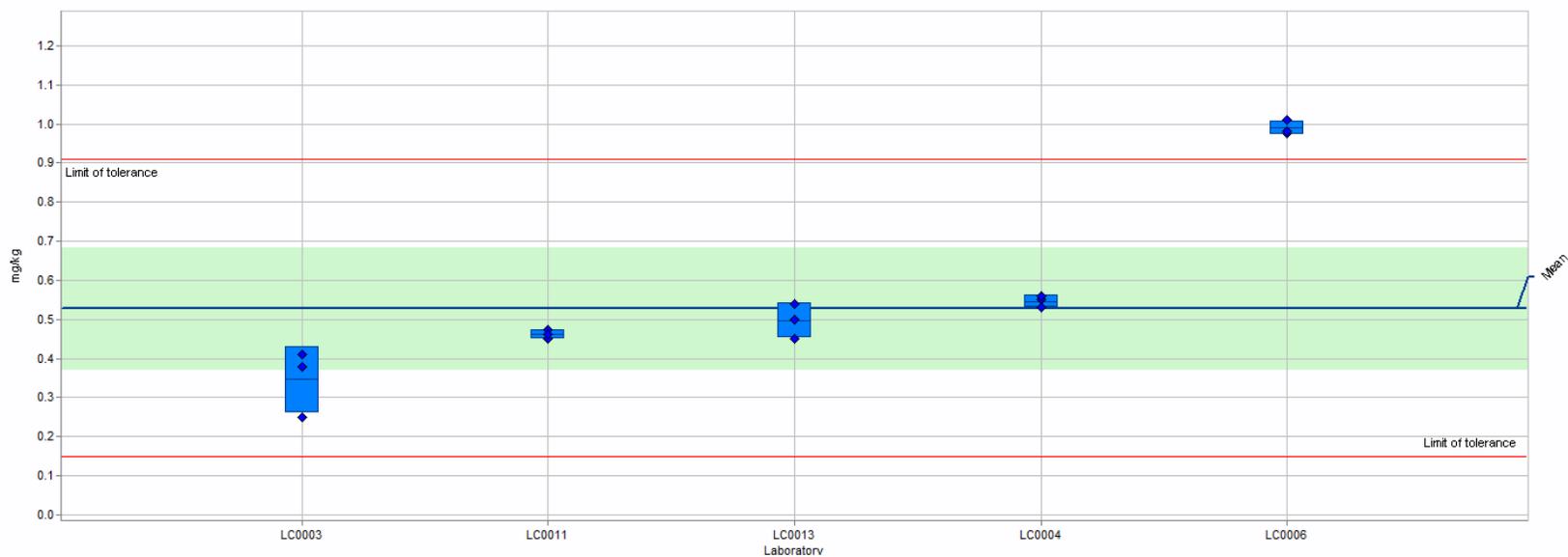


Figure 1-4. PFBA in candidate RM 8693 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation IV. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Perfluoropentanoic acid (PFPeA)

- Six laboratories reported results for PFPeA in candidate RMs 8690, 8691, and 8693. Four laboratories reported results for PFPeA in candidate RM 8692.
- The between-laboratory variability was good for candidate RMs 8690 (12 % RSD)
- The between-laboratory variability for candidate RMs 8691, 8692, and 8693 was poor (53 %, 88 %, and 40 % RSD, respectively).

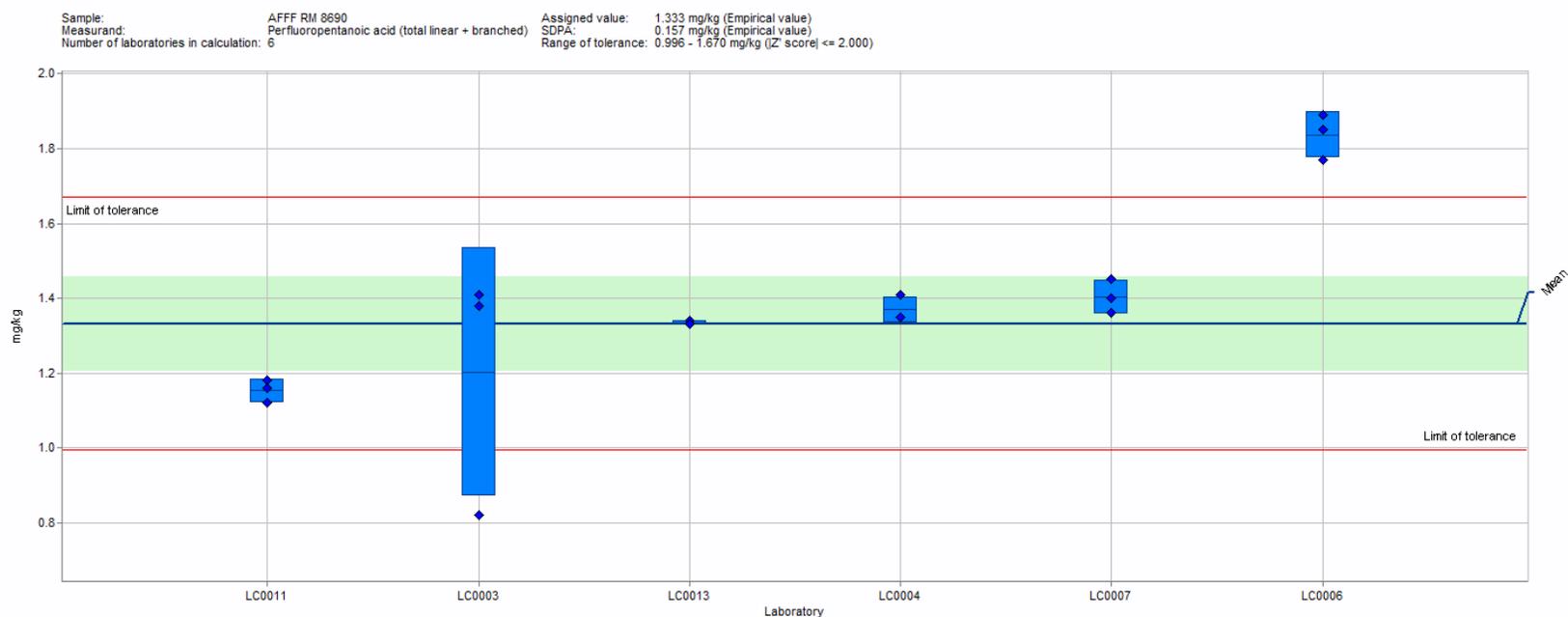


Figure 2-1. PFPeA in candidate RM 8690 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation I. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

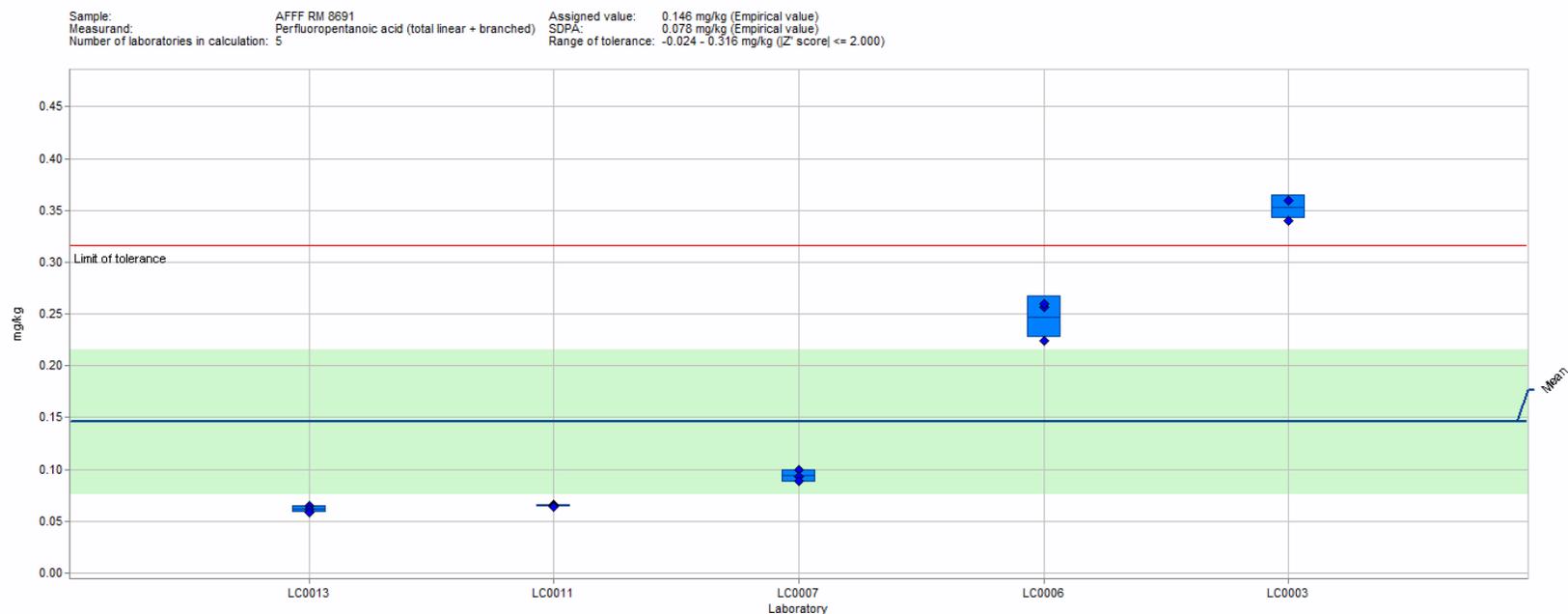


Figure 2-2. PFPeA in candidate RM 8691 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation II. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Sample: AFFF RM 8692 Assigned value: 0.086 mg/kg (Empirical value)
 Measurand: Perfluoropentanoic acid (total linear + branched) SDPA: 0.058 mg/kg (Empirical value)
 Number of laboratories in calculation: 3 Range of tolerance: -0.067 - 0.199 mg/kg (Z' score ≤ 2.000)



Figure 2-3. PFPeA in candidate RM 8692 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation III. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

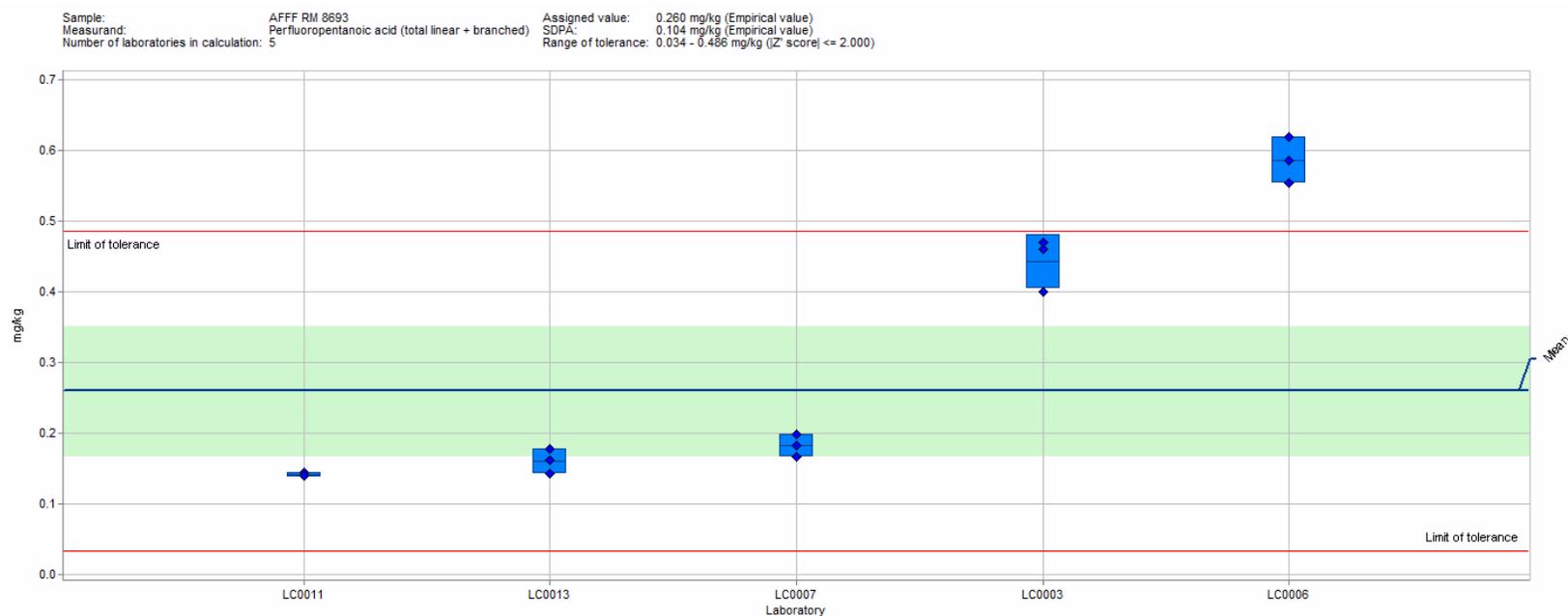


Figure 2-4. PFPeA in candidate RM 8693 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation IV. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Perfluorohexanoic acid (PFHxA)

- Six laboratories reported results for PFHxA in candidate RMs 8690, 8691, and 8693. Five laboratories reported results for PFHxA in candidate RM 8692.
- The between-laboratory variability was good for candidate RM 8690 (13 % RSD).
- The between-laboratory variability was poor for candidate RMs 8691, 8692, and 8693 (24 %, 21 %, and 33 % RSD, respectively).



Figure 3-1. PFHxA in candidate RM 8690 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation I. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

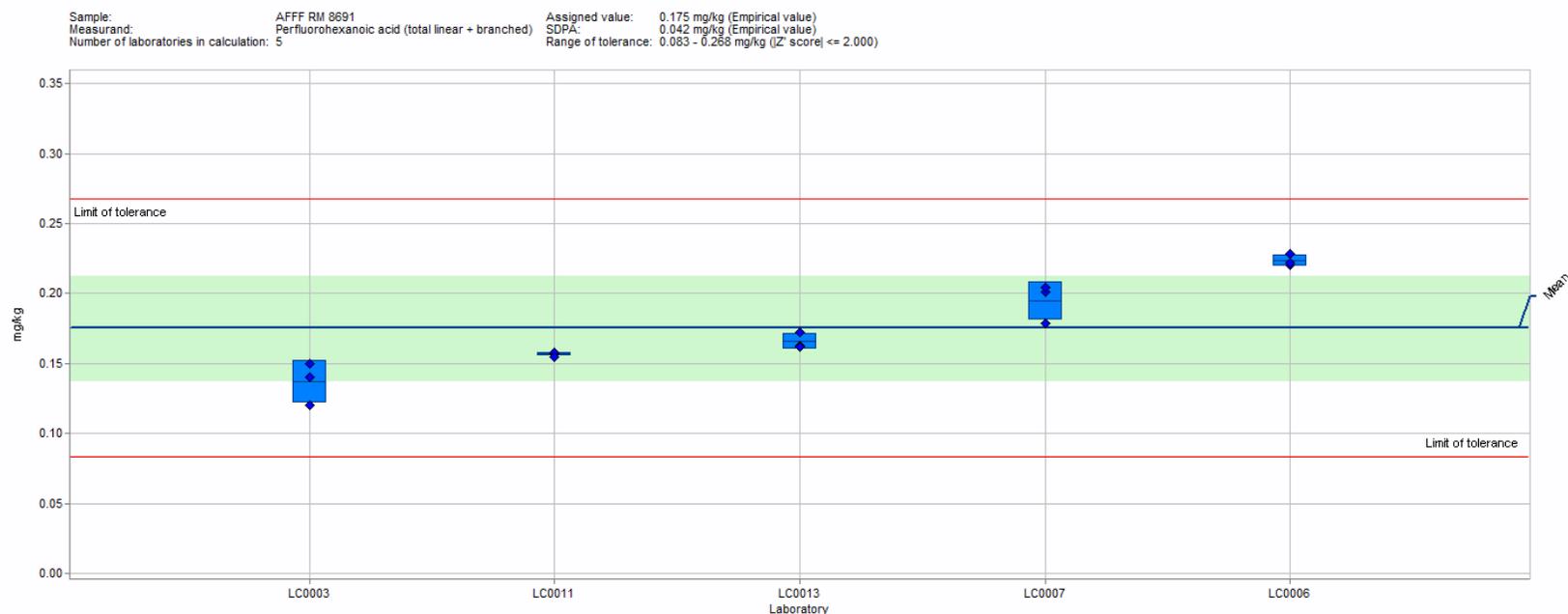


Figure 3-2. PFHxA in candidate RM 8691 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation II. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.



Figure 3-3. PFHxA in candidate RM 8692 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation III. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

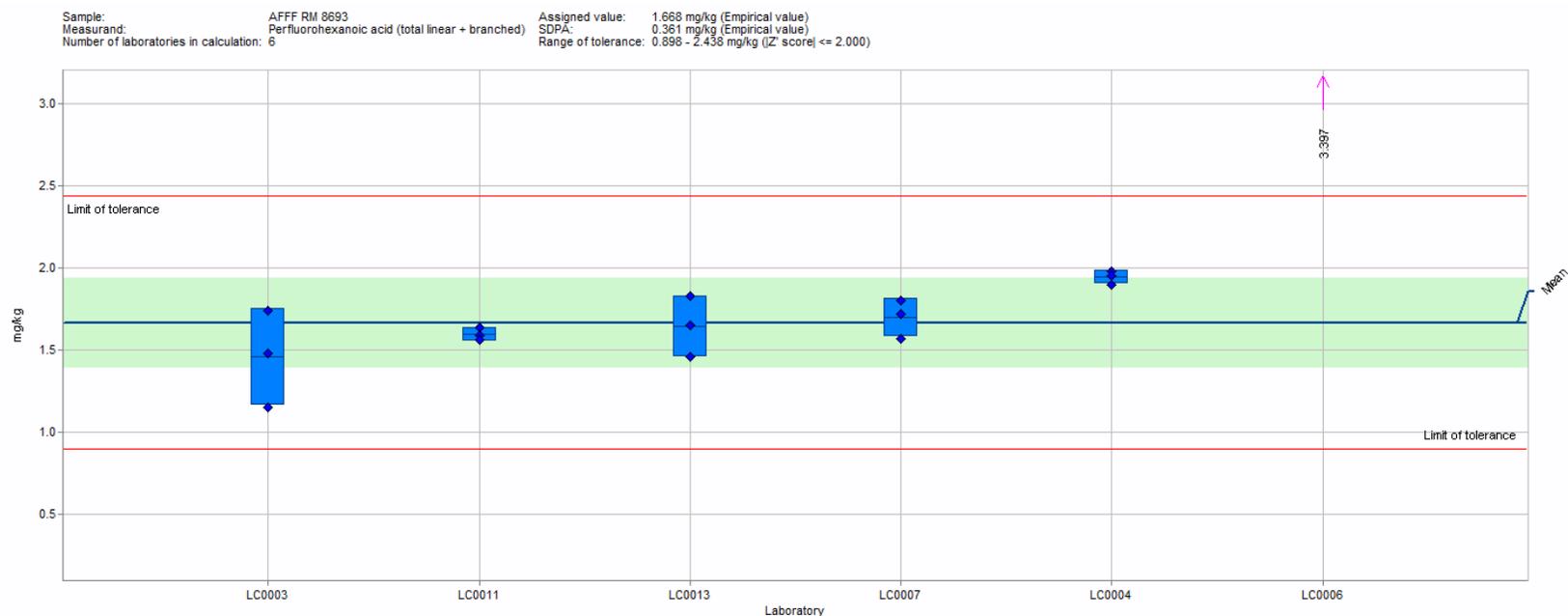


Figure 3-4. PFHxA in candidate RM 8693 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation IV. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Perfluoroheptanoic acid (PFHpA)

- Six laboratories reported results for PFHpA in candidate RMs 8690 and 8693. Five laboratories reported results for PFHpA in candidate RMs 8691 and 8692.
- The between-laboratory variability was poor for candidate RMs 8690, 8691, 8692, and 8693 (39 %, 83 %, 425 %, and 100 % RSD, respectively)



Figure 4-1. PFHpA in candidate RM 8690 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation I. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Sample: AFFF RM 8691
 Measurand: Perfluoroheptanoic acid (total linear + branched)
 Number of laboratories in calculation: 4
 Assigned value: 0.029 mg/kg (Empirical value)
 SDPA: 0.024 mg/kg (Empirical value)
 Range of tolerance: -0.025 - 0.082 mg/kg ($|Z'_{\text{comm}}| \leq 2.000$)

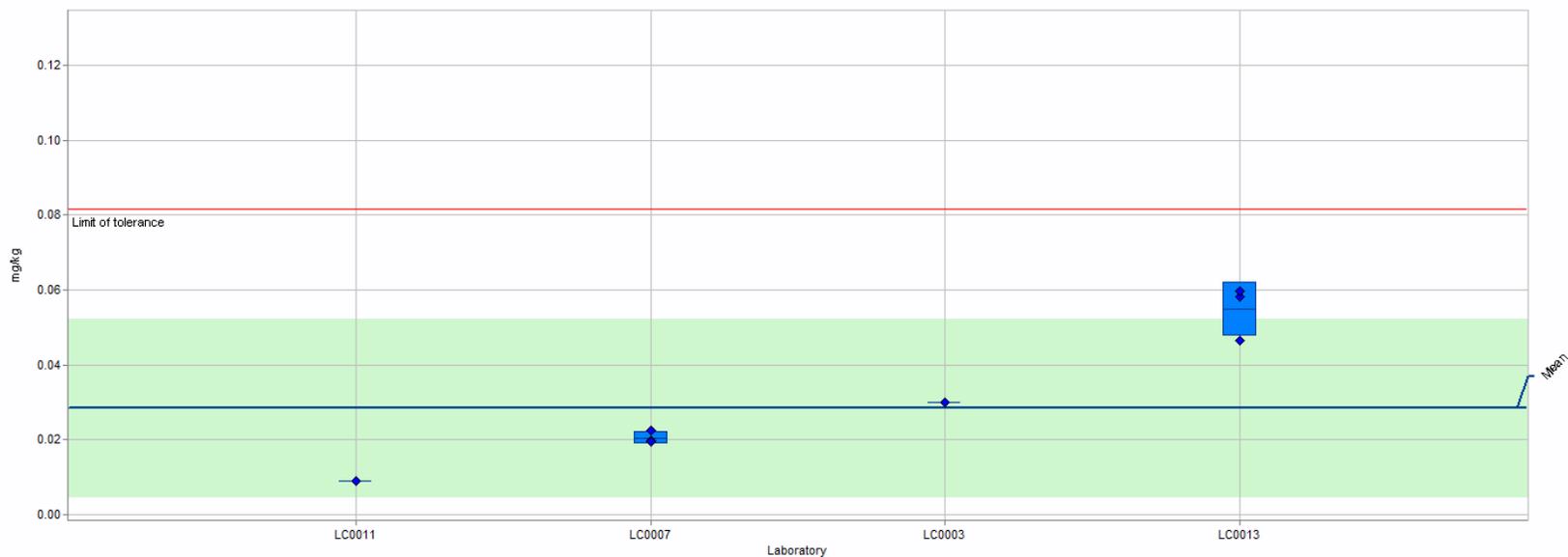


Figure 4-2. PFHpA in candidate RM 8691 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation II. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Sample: AFFF RM 8692
 Measurand: Perfluoroheptanoic acid (total linear + branched)
 Number of laboratories in calculation: 3
 Assigned value: 0.020 mg/kg (Empirical value)
 SDPA: 0.085 mg/kg (Empirical value)
 Range of tolerance: -0.175 - 0.215 mg/kg (Z' score ≤ 2.000)



Figure 4-3. PFHpA in candidate RM 8692 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation III. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

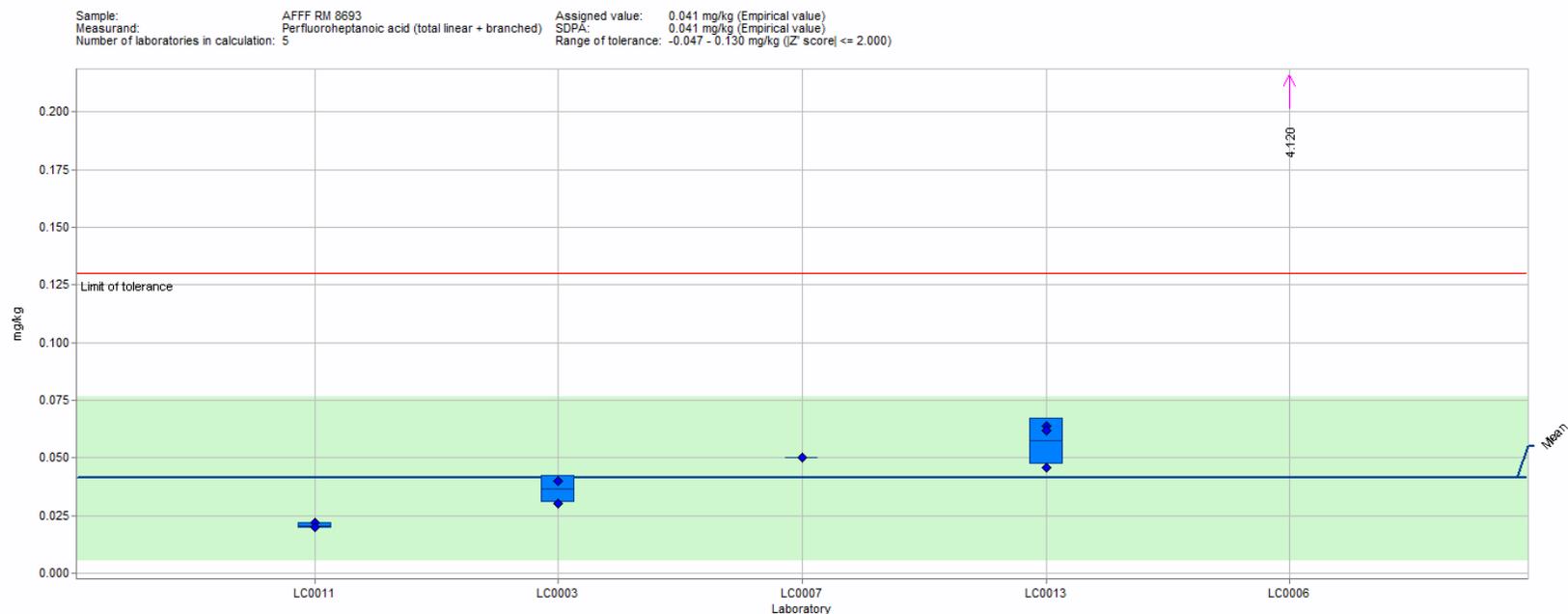


Figure 4-4. PFHpA in candidate RM 8693 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation IV. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Perfluorooctanoic acid (PFOA)

- Six laboratories reported results for PFOA in candidate RM 8690. Five laboratories reported results for PFOA in candidate RM 8691
- The between-laboratory variability was poor for candidate RMs 8690 and 8691 (29 % and 42 % RSD, respectively)

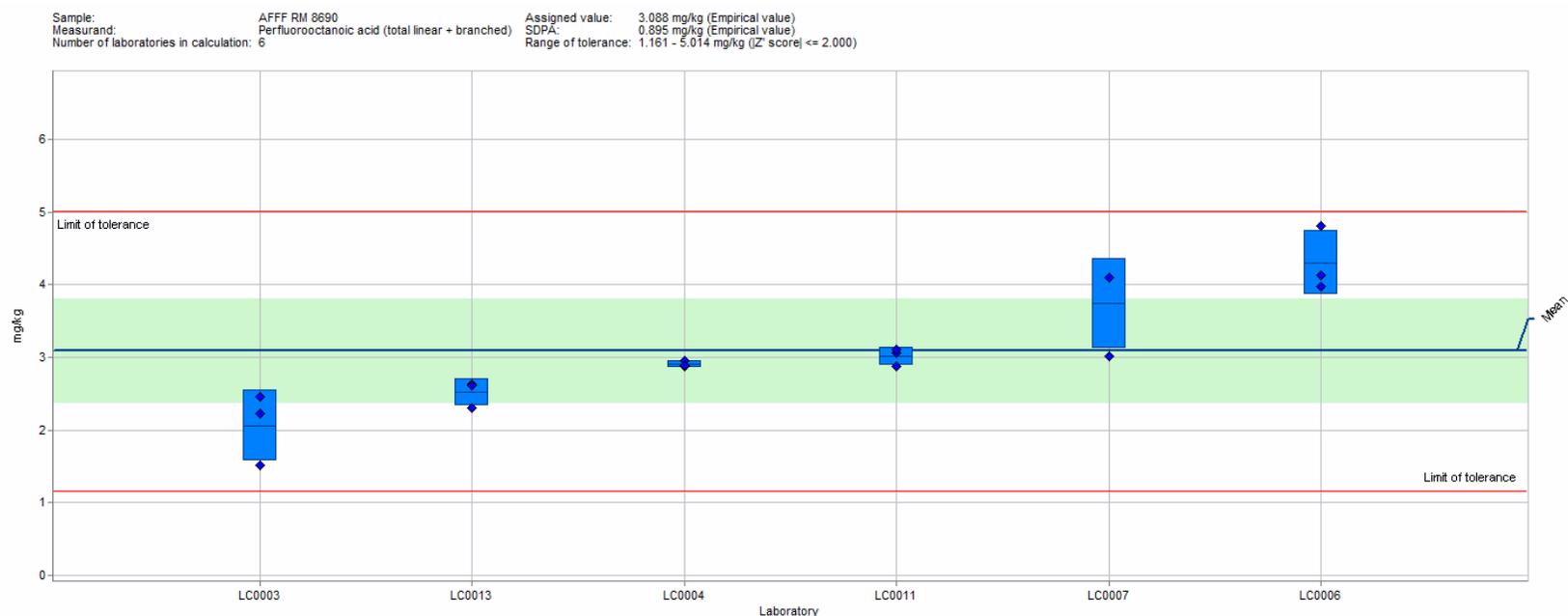


Figure 5-1. PFOA in candidate RM 8690 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation I. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

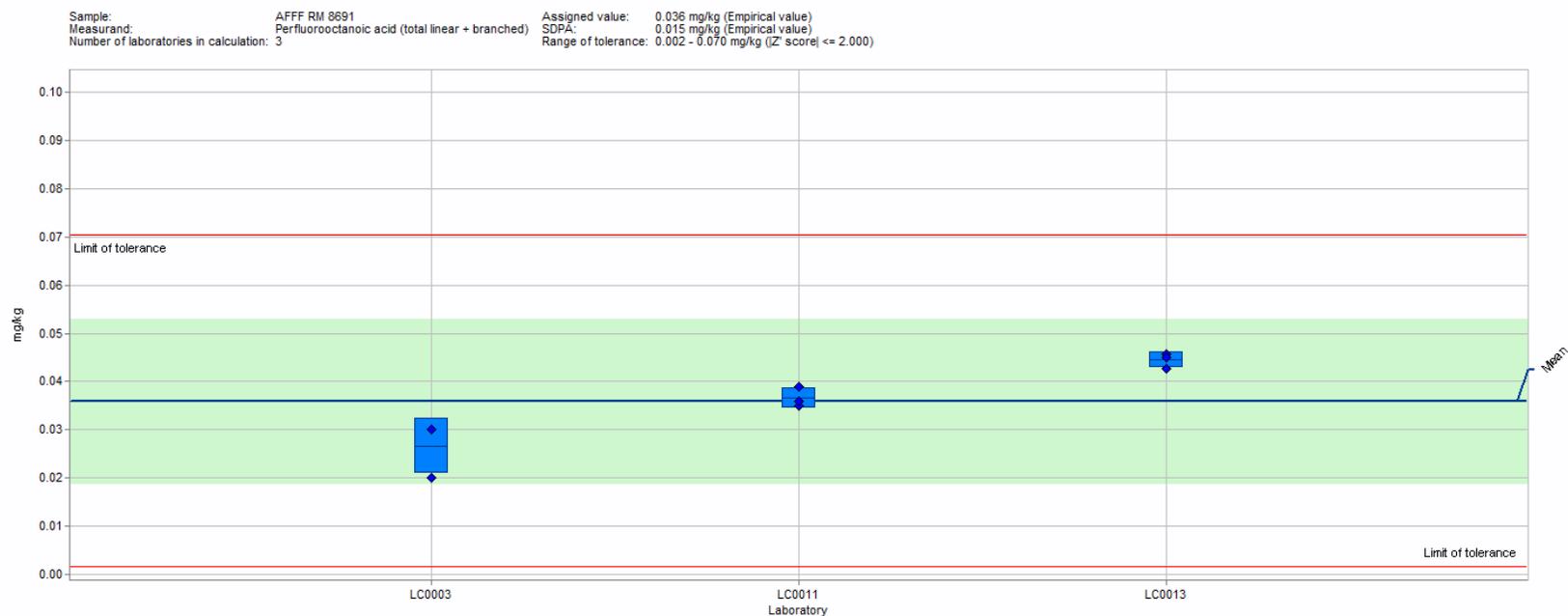


Figure 5-2. PFOA in candidate RM 8691 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation II. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Perfluorononanoic acid (PFNA)

- Four laboratories reported results for PFNA in candidate RM 8691. Three laboratories reported results for PFNA in candidate RM 8690
- Although laboratories reported results for PFNA, only two laboratories reported results for PFNA above their detection limits

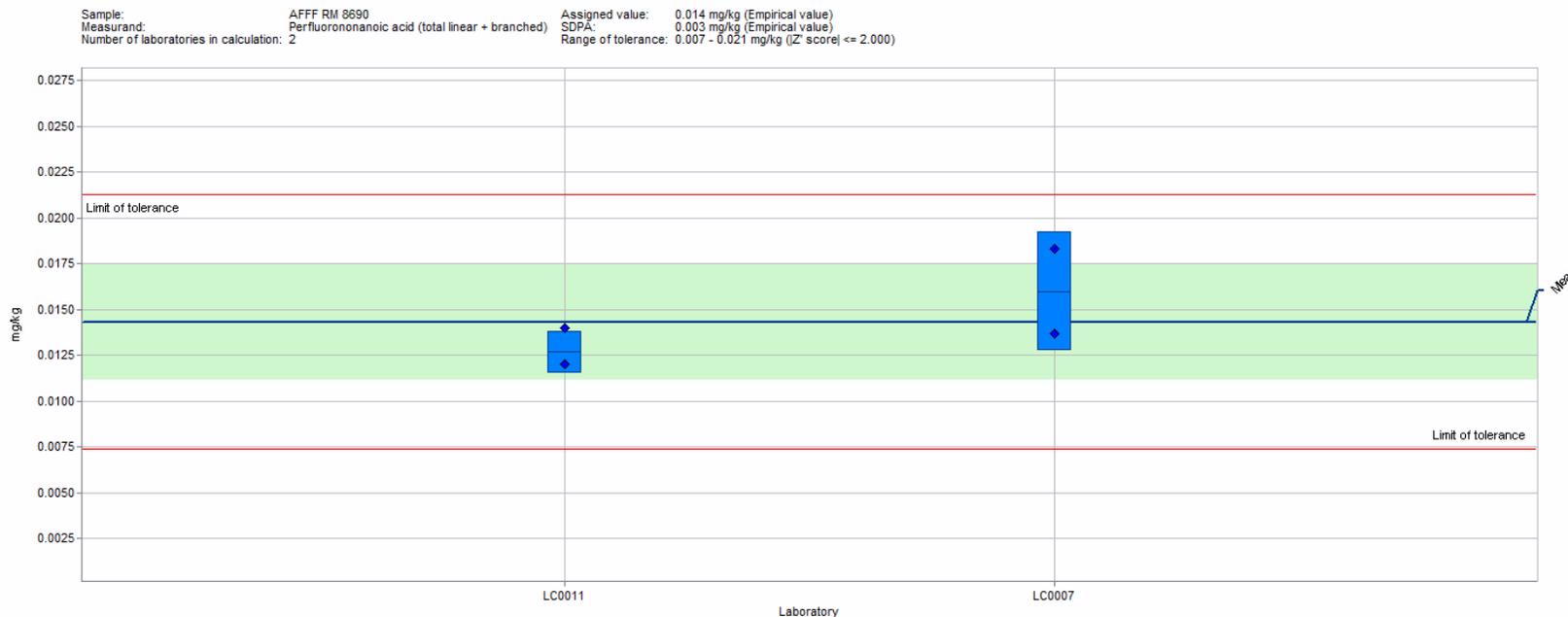


Figure 6-1. PFNA in candidate RM 8690 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation I. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Sample: AFFF RM 8691
 Measurand: Perfluorononanoic acid (total linear + branched)
 Number of laboratories in calculation: 3
 Assigned value: 0.014 mg/kg (Empirical value)
 SDPA: 0.004 mg/kg (Empirical value)
 Range of tolerance: 0.004 - 0.023 mg/kg ($|Z'_{\text{score}}| \leq 2.000$)

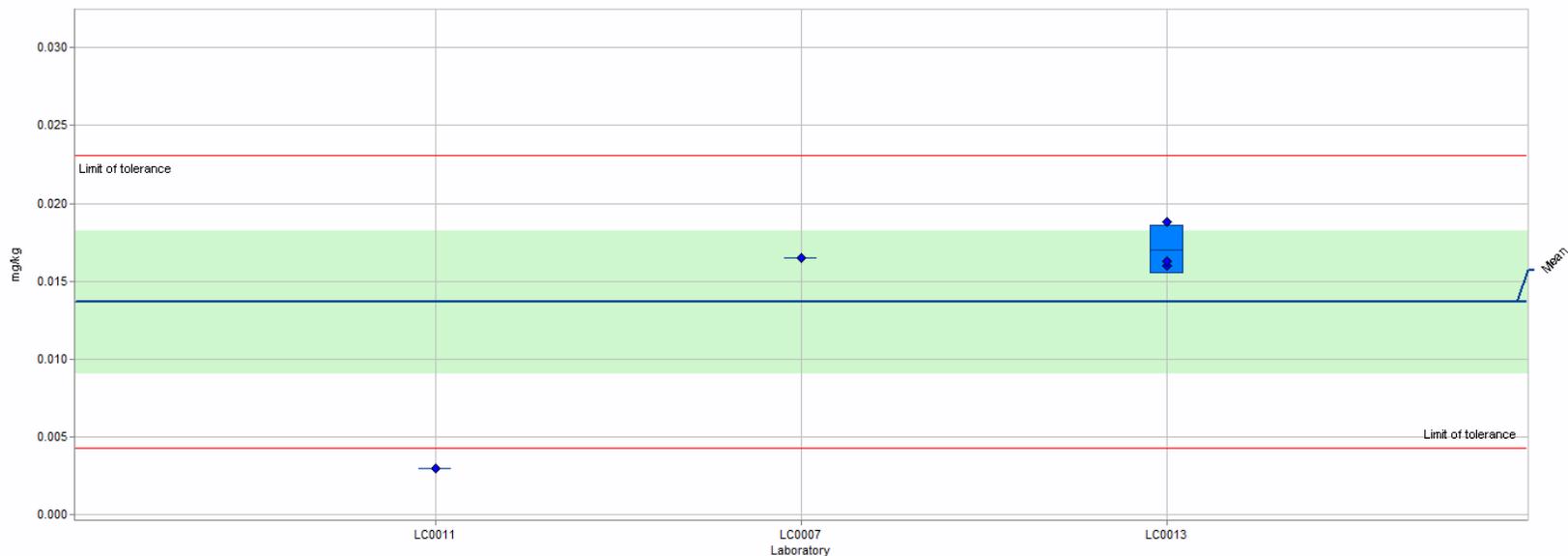


Figure 6-2. PFNA in candidate RM 8691 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation II. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Perfluorodecanoic acid (PFDA)

- Five laboratories reported results for PFDA in candidate RM 8691. Four laboratories reported results for PFDA in candidate RM 8690
- The between-laboratory variability was poor for candidate RMs 8690 and 8691 (312% and 32 % RSD, respectively)

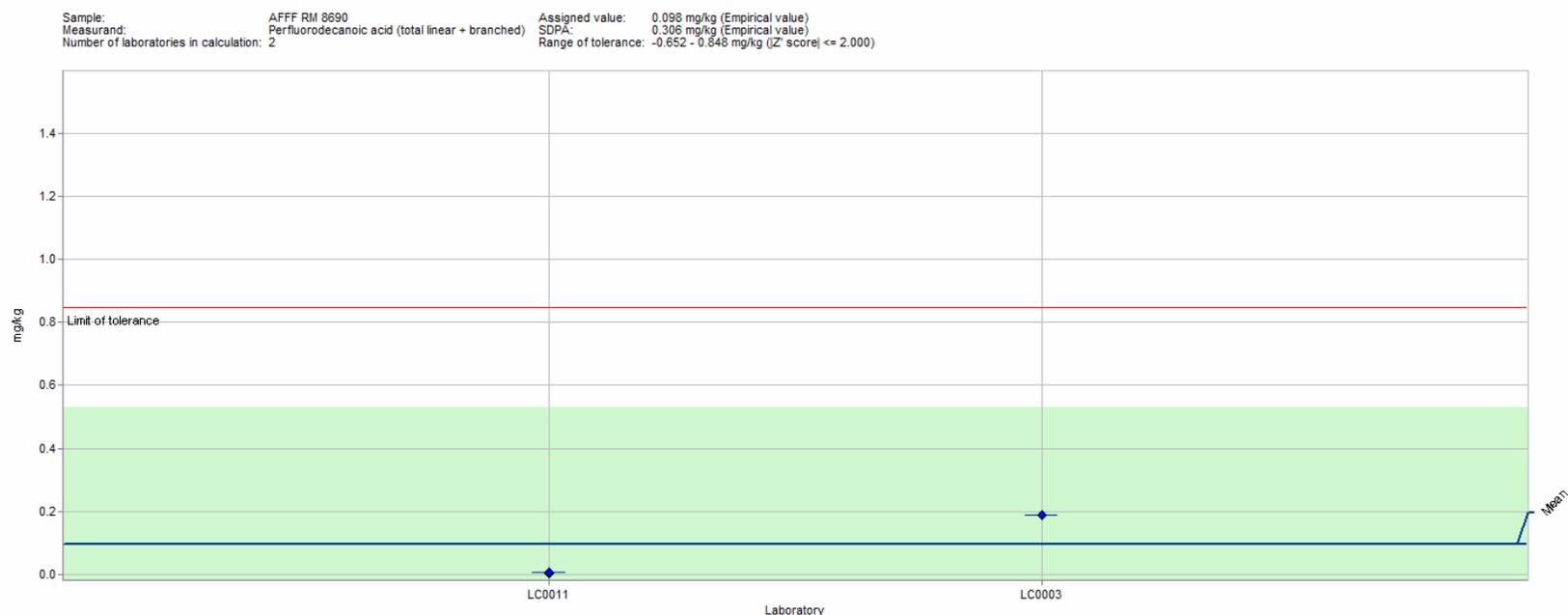


Figure 7-1. PFDA in candidate RM 8690 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation I. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

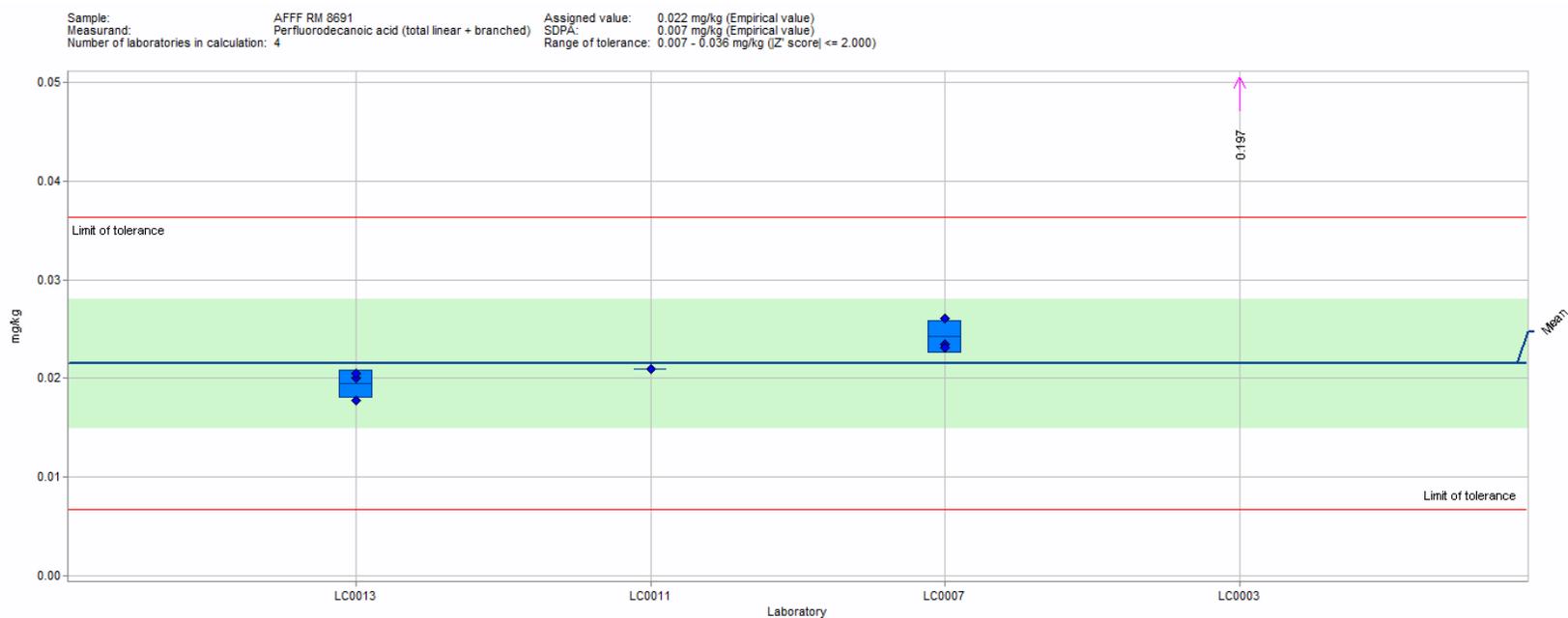


Figure 7-2. PFDA in candidate RM 8691 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation II. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Perfluoroundecanoic acid (PFUnA)

- Three laboratories reported results for PFUnA in candidate RM 8691.
- The between-laboratory variability was poor for candidate RM 8691 (255 % RSD)

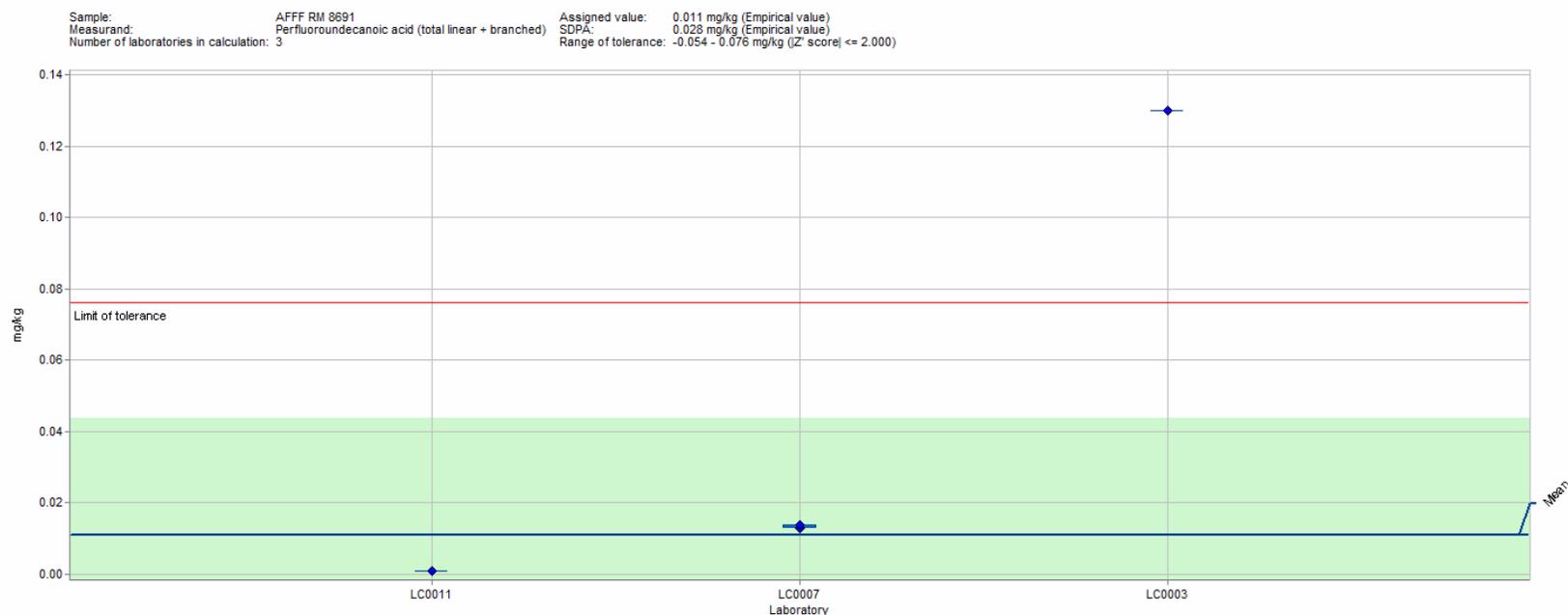


Figure 8. PFUnA in candidate RM 8691 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation II. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Perfluorotetradecanoic acid (PFTA)

- Four laboratories reported results for PFTA in candidate RM 8691.
- The between-laboratory variability was poor for candidate RM 8691 (235 % RSD)

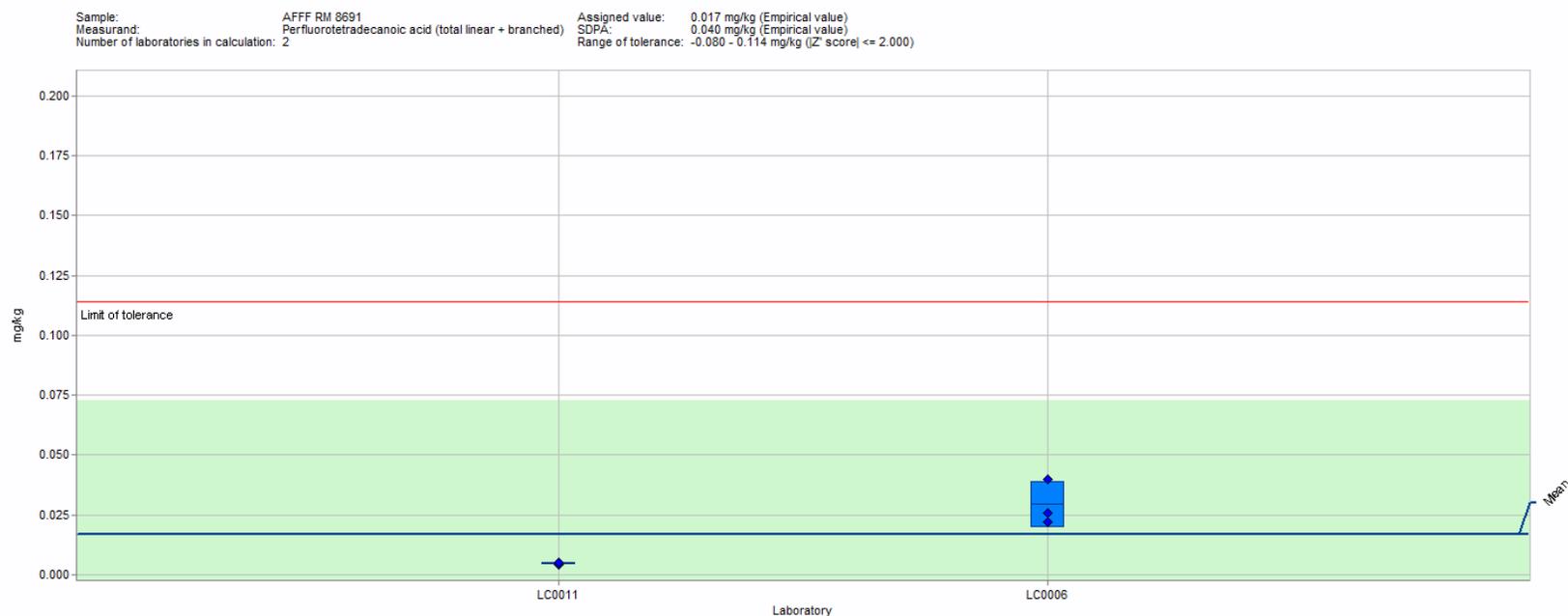


Figure 9. PFTA in candidate RM 8691 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation II. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Perfluorobutanesulfonic acid (PFBS)

- Six laboratories reported results for PFBS in candidate RM 8690. Three laboratories reported results for PFBS in candidate RMs 8691, 8692, and 8693.
- The between-laboratory variability was good for candidate RM 8690 (12 % RSD)
- The between-laboratory variability was poor for candidate RMs 8691, 8692, and 8693 (124 %, 316 %, and 410 % RSD, respectively)

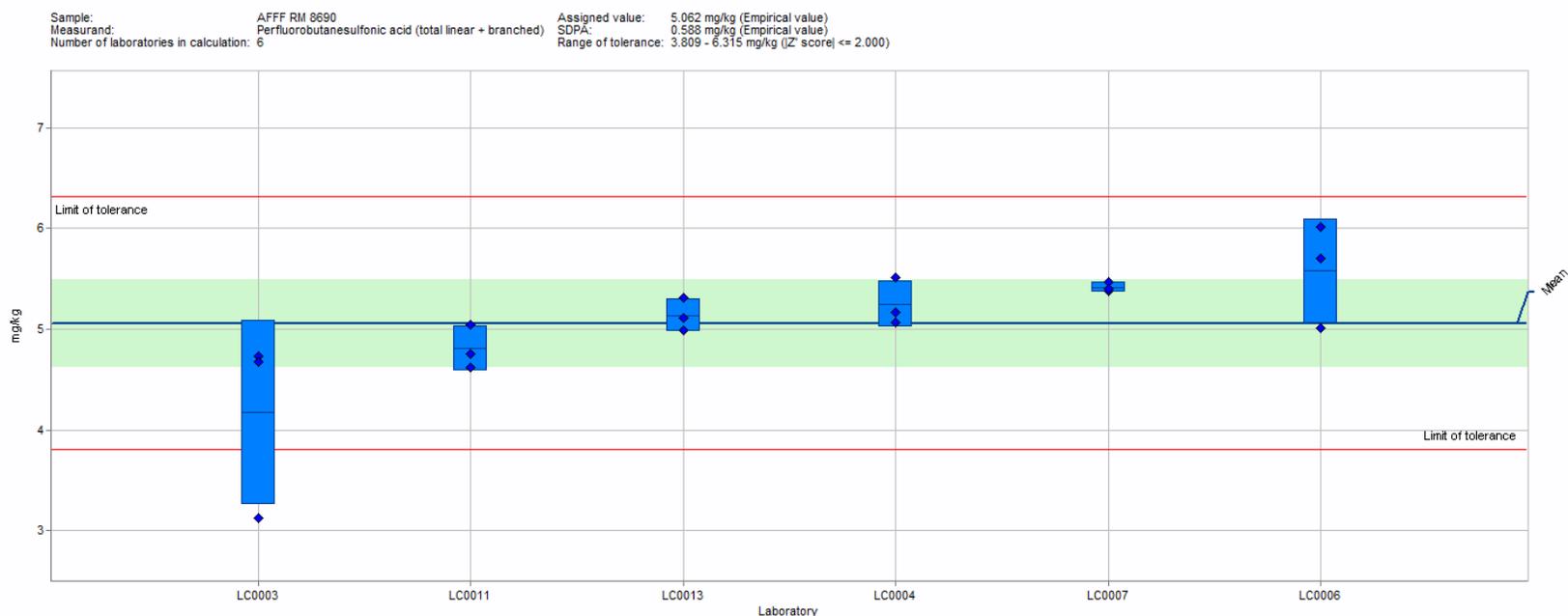


Figure 10-1. PFBS in candidate RM 8690 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation I. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

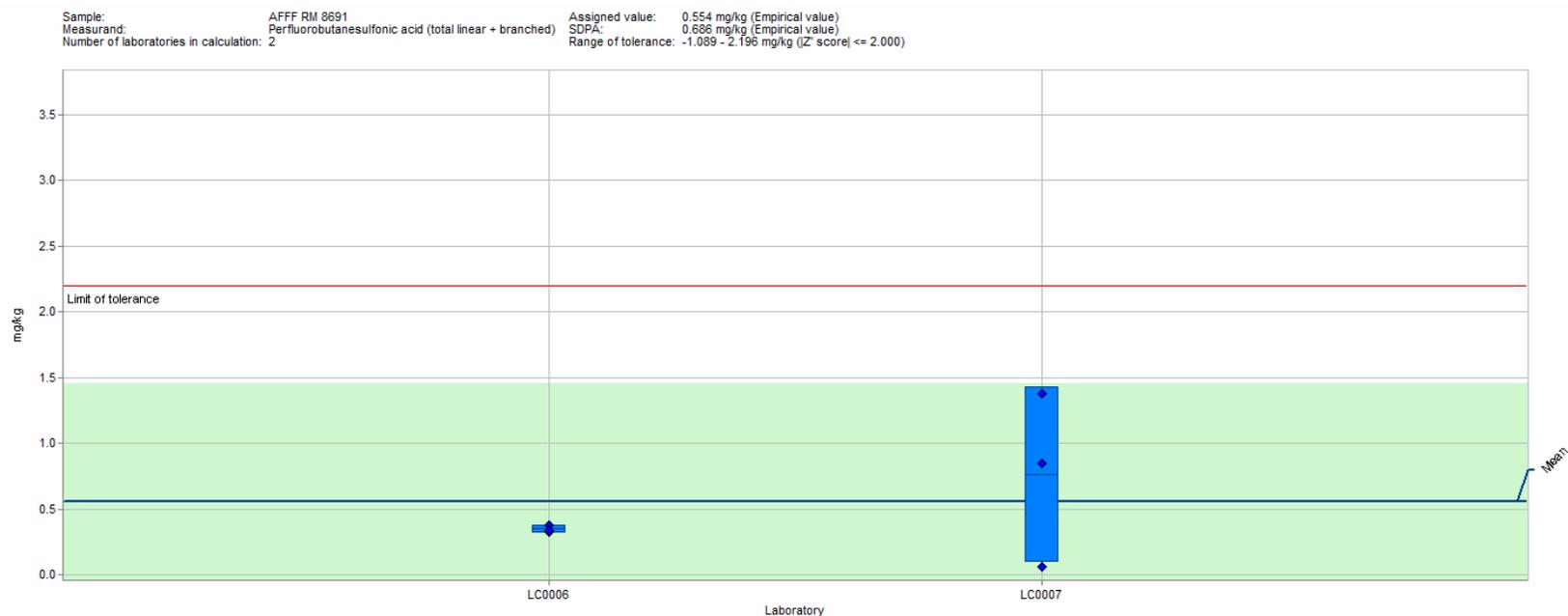


Figure 10-2. PFBS in candidate RM 8691 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation II. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

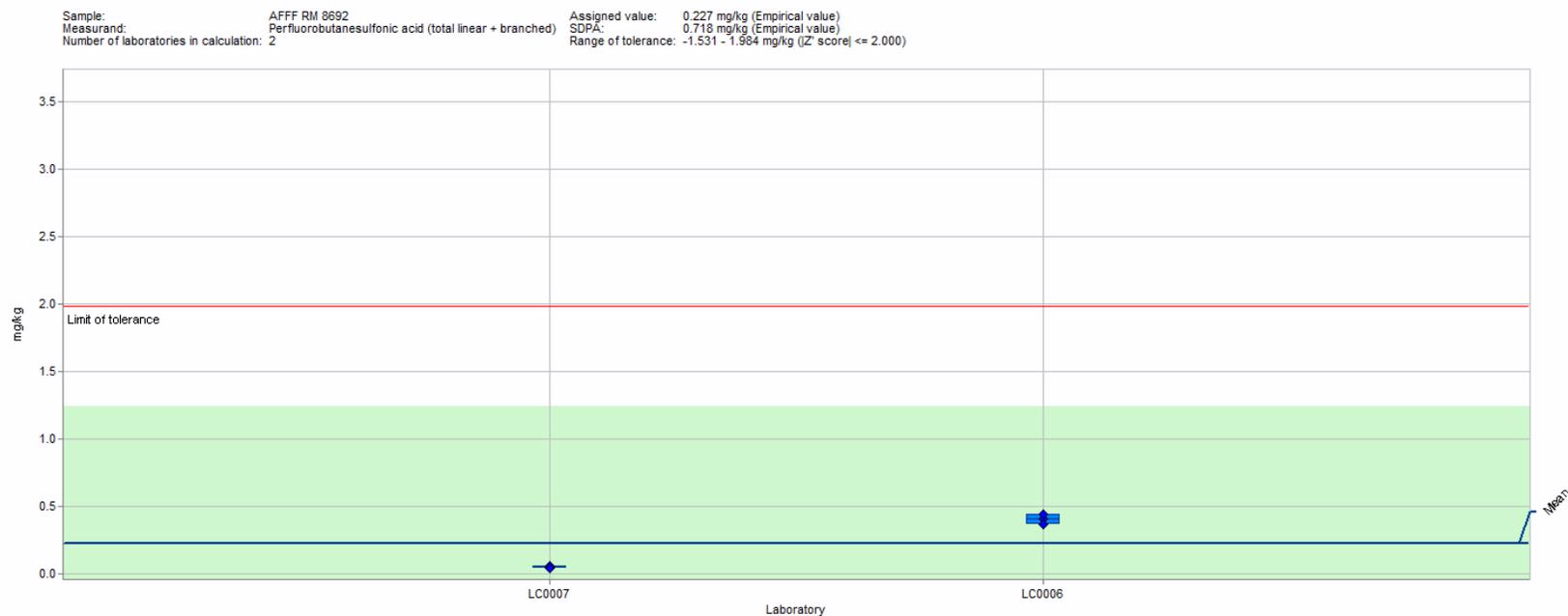


Figure 10-3. PFBS in candidate RM 8692 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation III. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Sample: AFFF RM 8693 Assigned value: 0.282 mg/kg (Empirical value)
 Measurand: Perfluorobutanesulfonic acid (total linear + branched) SDPA: 1.155 mg/kg (Empirical value)
 Number of laboratories in calculation: 2 Range of tolerance: -2.547 - 3.112 mg/kg ($|Z'_{\text{comm}}| \leq 2.000$)

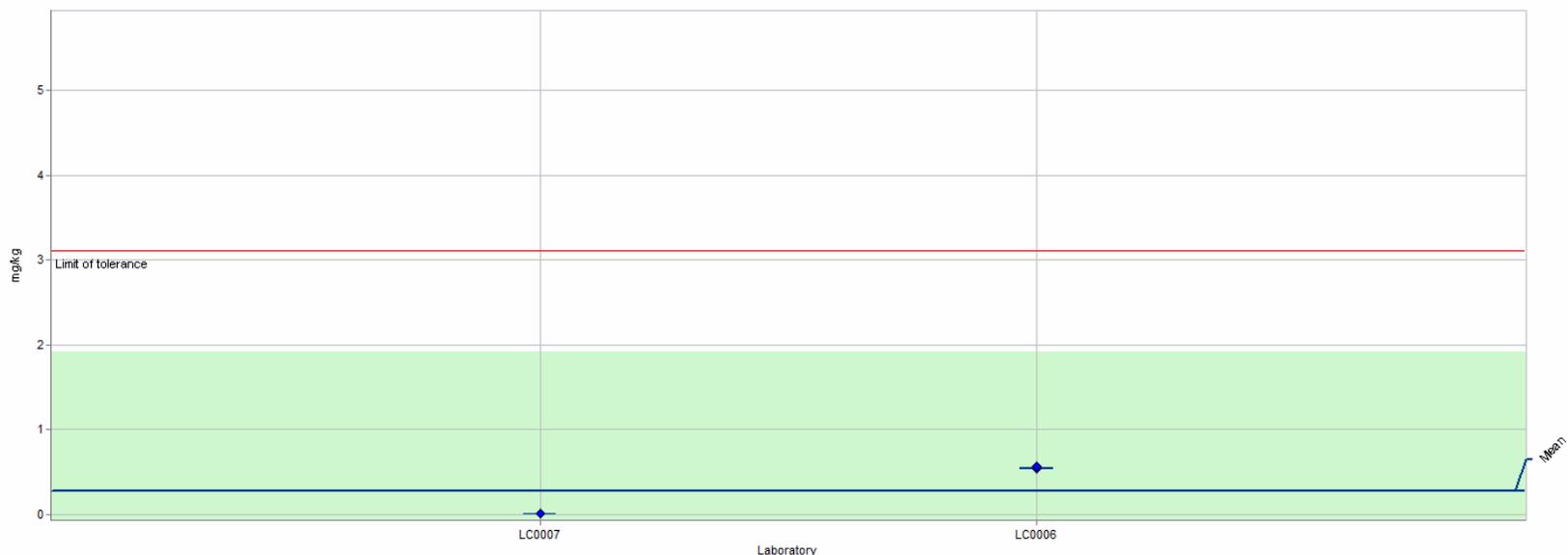


Figure 10-4. PFBS in candidate RM 8693 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation IV. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Perfluoropentanesulfonic acid (PFPeS)

- Six laboratories reported results for PFPeS in candidate RM 8690. Two laboratories reported results for PFPeS in candidate RMs 8691, 8692, and 8693.
- The between-laboratory variability was good for candidate RM 8690 (19 % RSD)



Figure 11. PFPeS in candidate RM 8690 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation I. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Perfluorohexanesulfonic acid (PFHxS)

- Six laboratories reported results for PFHxS in candidate RM 8690. Two laboratories reported results for PFHxS in candidate RMs 8691, 8692, and 8693.
- The between-laboratory variability was poor for candidate RM 8690 (26 % RSD)

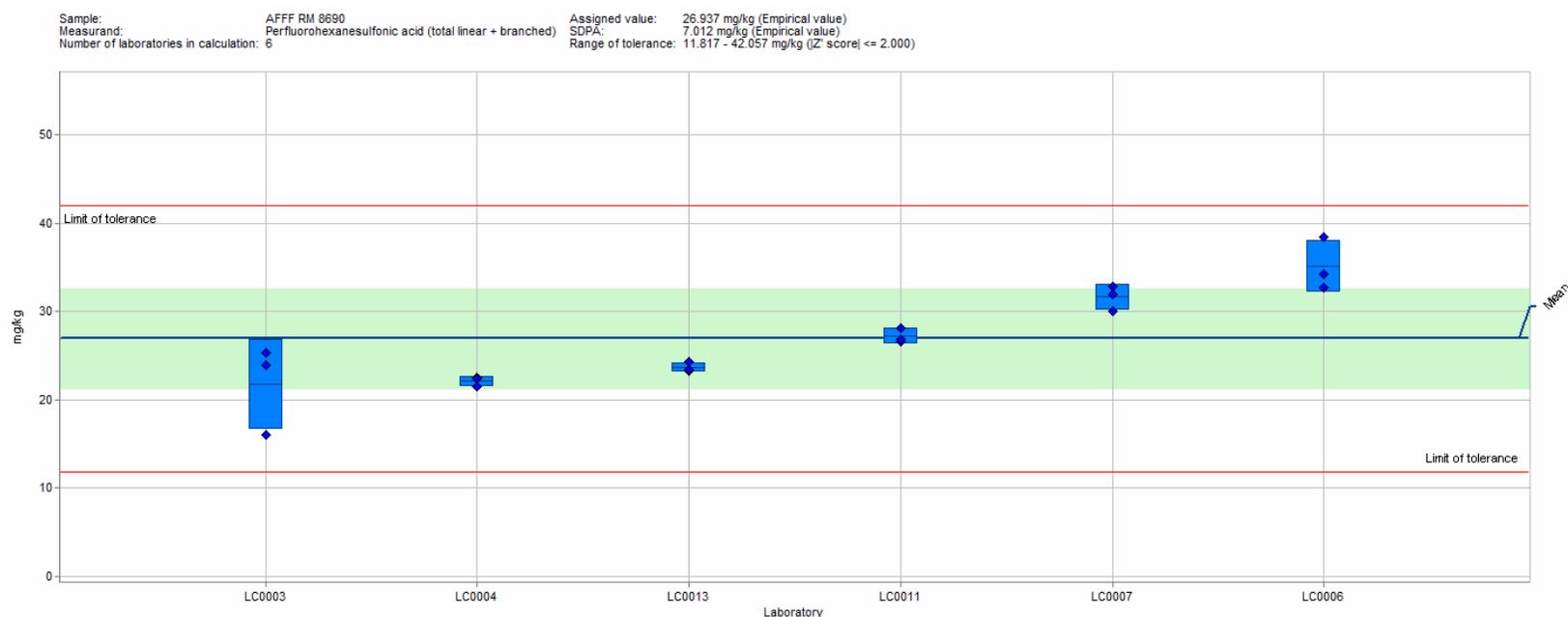


Figure 12. PFHxS in candidate RM 8690 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation I. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Perfluoroheptanesulfonic acid (PFHpS)

- Six laboratories reported results for PFHpS in candidate RM 8690. Three laboratories reported results for PFHpS in candidate RM 8691. Two laboratories reported results for PFHpS in candidate RMs 8692 and 8693.
- The between-laboratory variability was poor for candidate RM 8690 (69 % RSD)

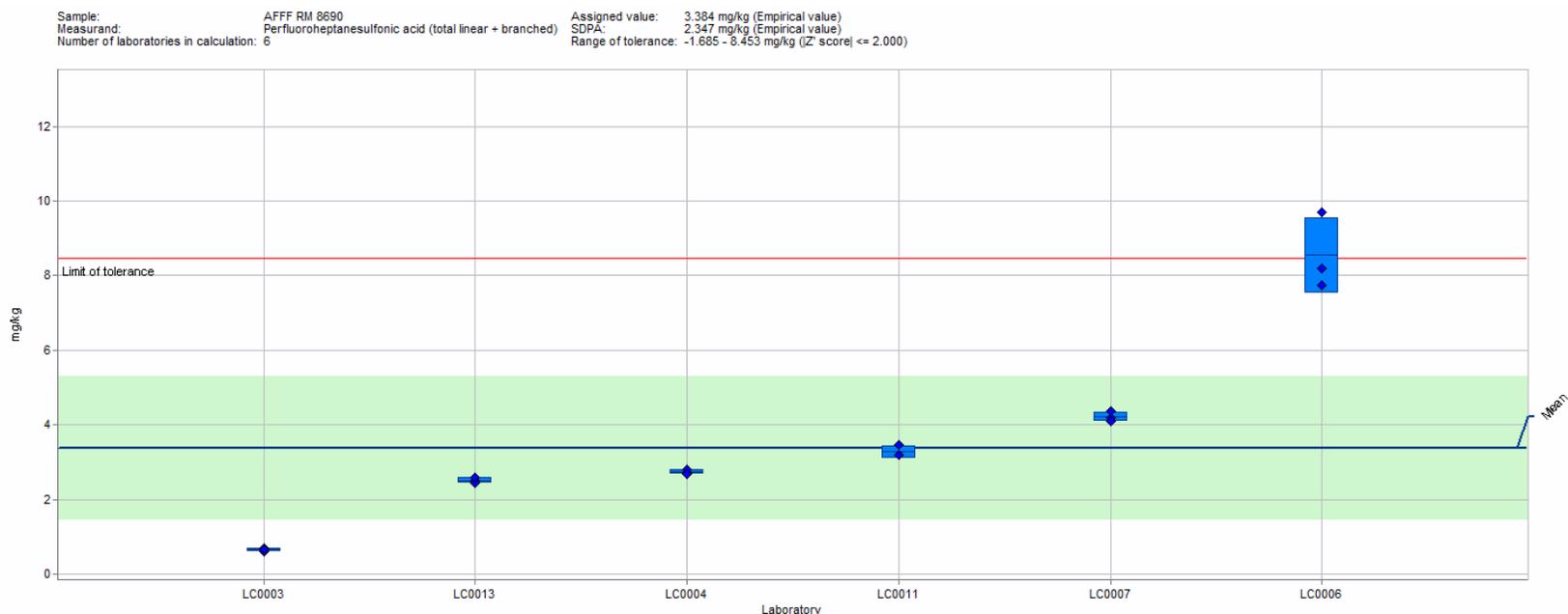


Figure 13. PFHpS in candidate RM 8690 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation I. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Perfluorooctanesulfonic acid (PFOS)

- Six laboratories reported results for PFOS in candidate RM 8690. Three laboratories reported results for PFOS in candidate RMs 8691, 8692, and 8693.
- The between-laboratory variability was poor for candidate RMs 8690 and 8692 (24 % and 421 % RSD, respectively)



Figure 14-1. PFOS in candidate RM 8690 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation I. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Sample: AFFF RM 8692 Assigned value: 2.080 mg/kg (Empirical value)
 Measurand: Perfluorooctanesulfonic acid (total linear + branched) SDPA: 8.749 mg/kg (Empirical value)
 Number of laboratories in calculation: 2 Range of tolerance: -19.351 - 23.510 mg/kg (Z' score ≤ 2.000)

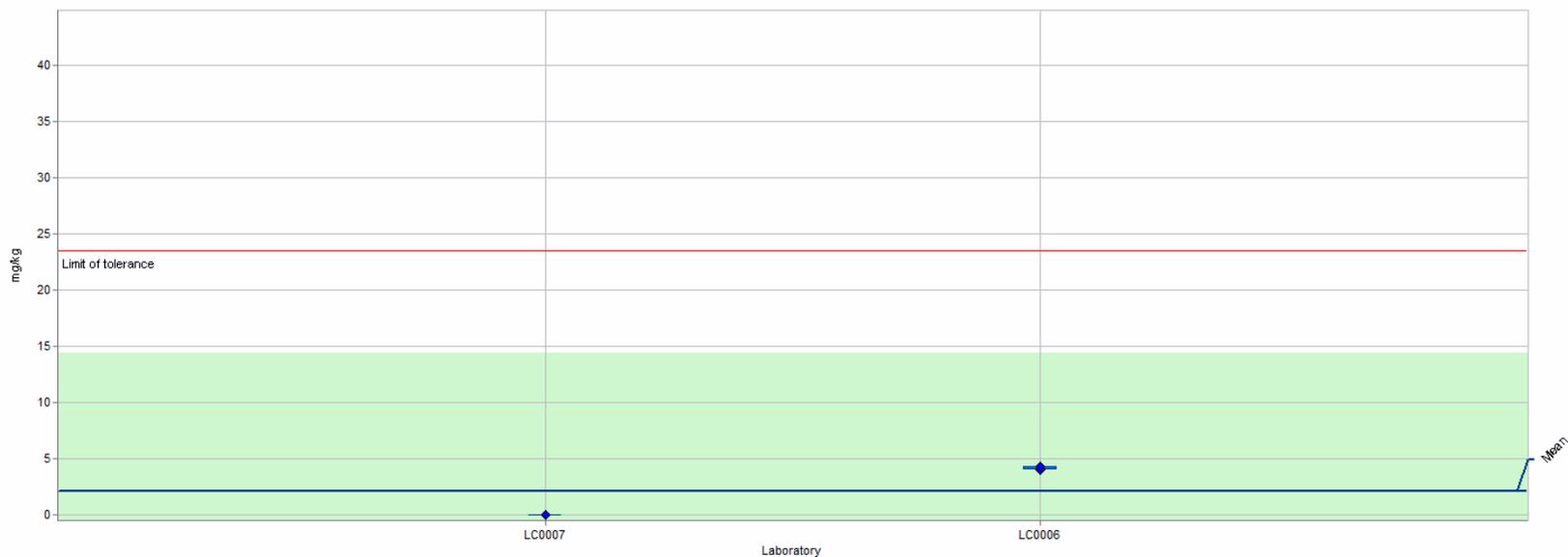


Figure 14-2. PFOS in candidate RM 8692 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation III. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Perfluorononanesulfonic acid (PFNS)

- Three laboratories reported results for PFNS in candidate RMs 8690 and 8692. Two laboratories reported results for PFNS in candidate RMs 8691 and 8693.
- The between-laboratory variability was poor for candidate RM 8690 (150 % RSD)

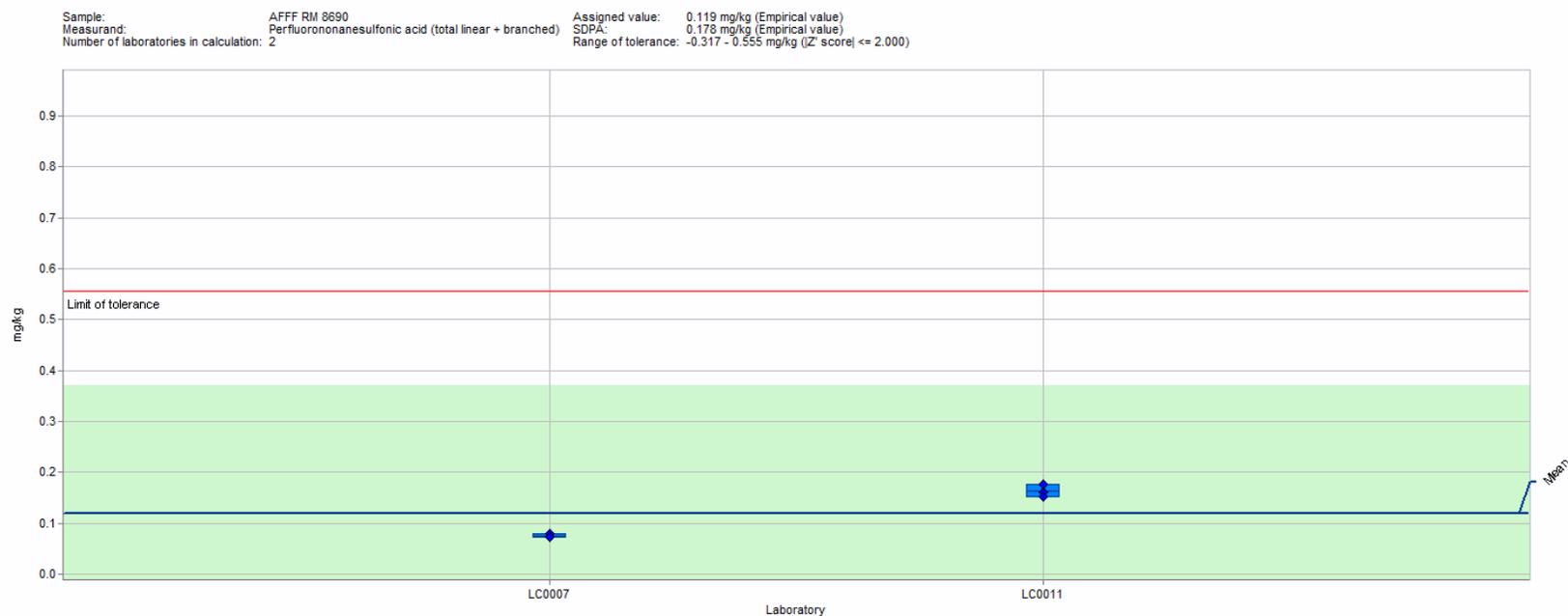


Figure 15. PFNS in candidate RM 8690 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation I. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Perfluorodecanesulfonic acid (PFDS)

- Four laboratories reported results for PFDS in candidate RM 8690. Two laboratories reported results for PFDS in candidate RMs 8691, 8692, and 8693.
- The between-laboratory variability was poor for candidate RM 8690 (44 % RSD)

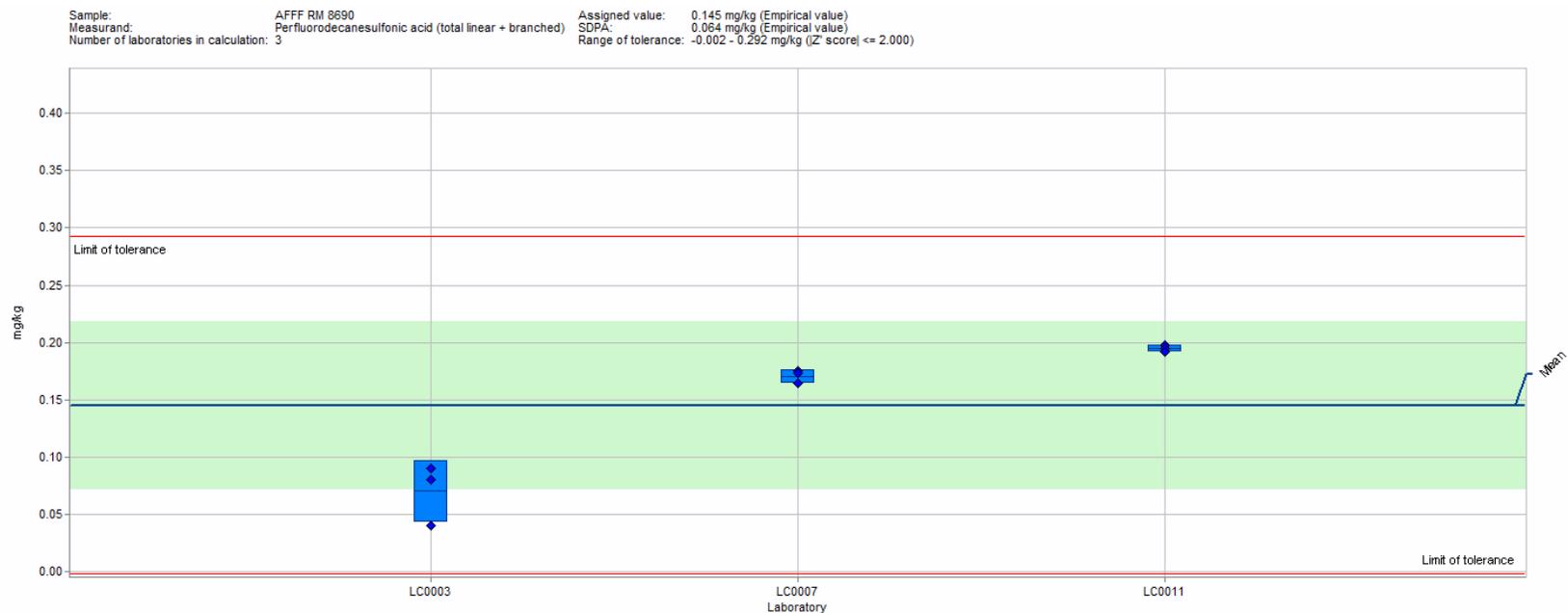


Figure 16. PFDS in candidate RM 8690 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation I. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

1H,1H, 2H, 2H-Perfluorohexane sulfonic acid (4:2 FTS)

- Six laboratories reported results for 4:2 FTS in candidate RM 8693. Four laboratories reported results for 4:2 FTS in candidate RM 8690. Three laboratories reported results for 4:2 FTS in candidate RM 8691. Two laboratories reported results for 4:2 FTS in candidate RM 8692.
- The between-laboratory variability was poor for candidate RM 8690, 8691, and 8693 (77 %, 344 %, and 44 % RSD, respectively).

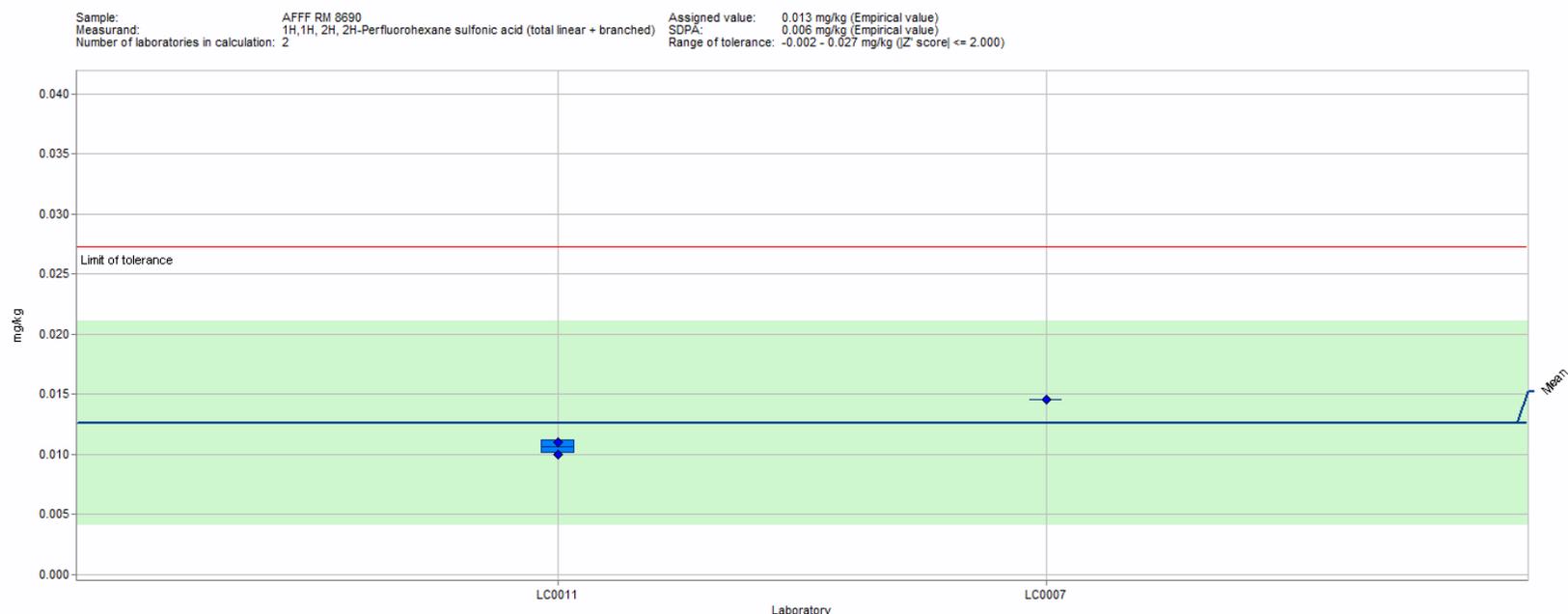


Figure 17-1. 4:2 FTS in candidate RM 8690 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation I. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Sample: AFFF RM 8691
 Measurand: 1H,1H,2H,2H-Perfluorohexane sulfonic acid (total linear + branched)
 Number of laboratories in calculation: 2

Assigned value: 0.009 mg/kg (Empirical value)
 SDPA: 0.031 mg/kg (Empirical value)
 Range of tolerance: -0.067 - 0.085 mg/kg ($|Z'_{\text{score}}| \leq 2.000$)



Figure 17-2. 4:2 FTS in candidate RM 8691 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation II. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

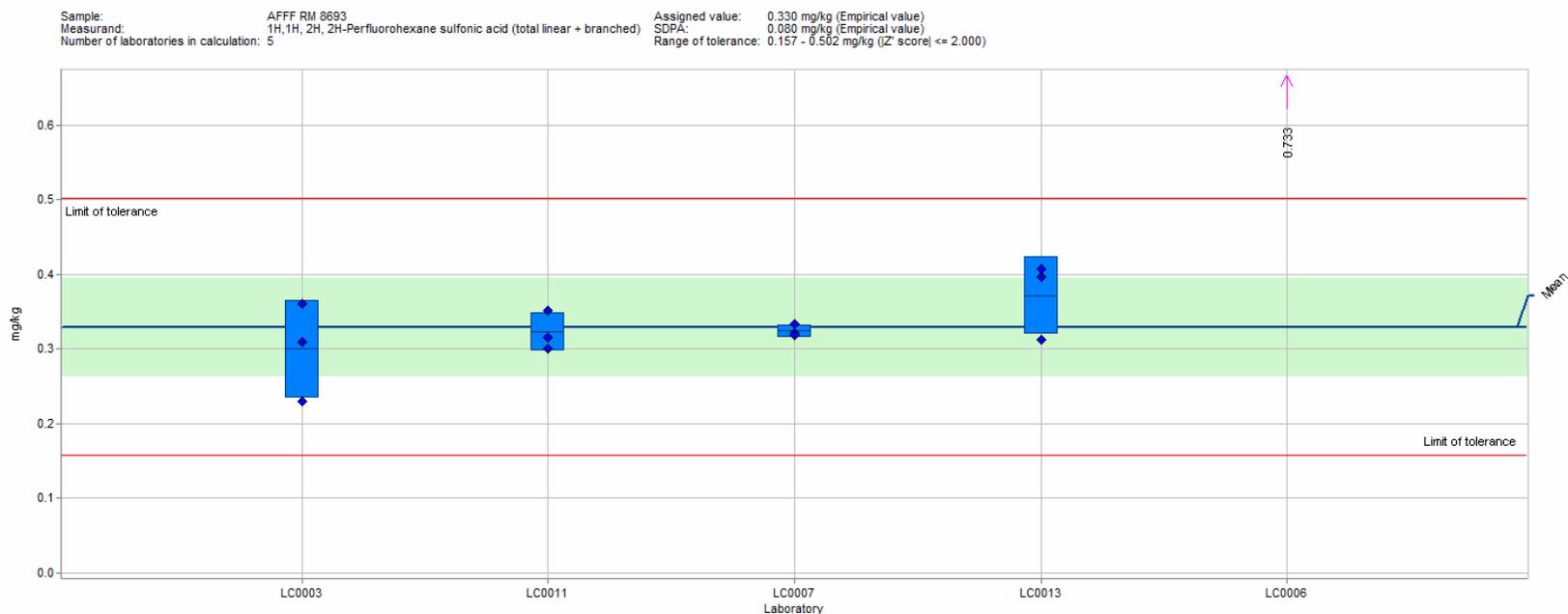


Figure 17-3. 4:2 FTS in candidate RM 8693 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation IV. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

1H,1H, 2H, 2H-Perfluorooctane sulfonic acid (6:2 FTS)

- Six laboratories reported results for 6:2 FTS in candidate RMs 8690 and 8693. Five laboratories reported results for 6:2 FTS in candidate RMs 8691 and 8692.
- The between-laboratory variability was poor for candidate RM 8690, 8691, 8692 and 8693 (65 %, 24 %, 49 %, and 26 % RSD, respectively).



Figure 18-1. 6:2 FTS in candidate RM 8690 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation I. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

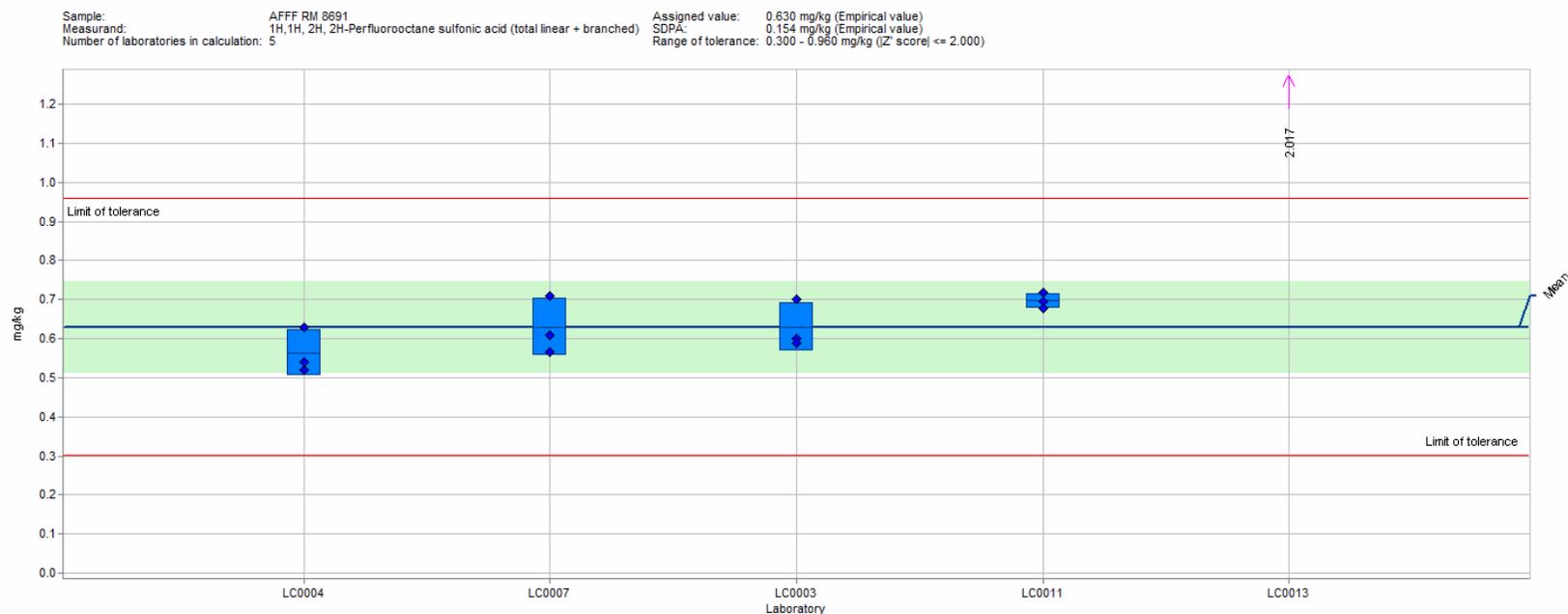


Figure 18-2. 6:2 FTS in candidate RM 8691 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation II. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

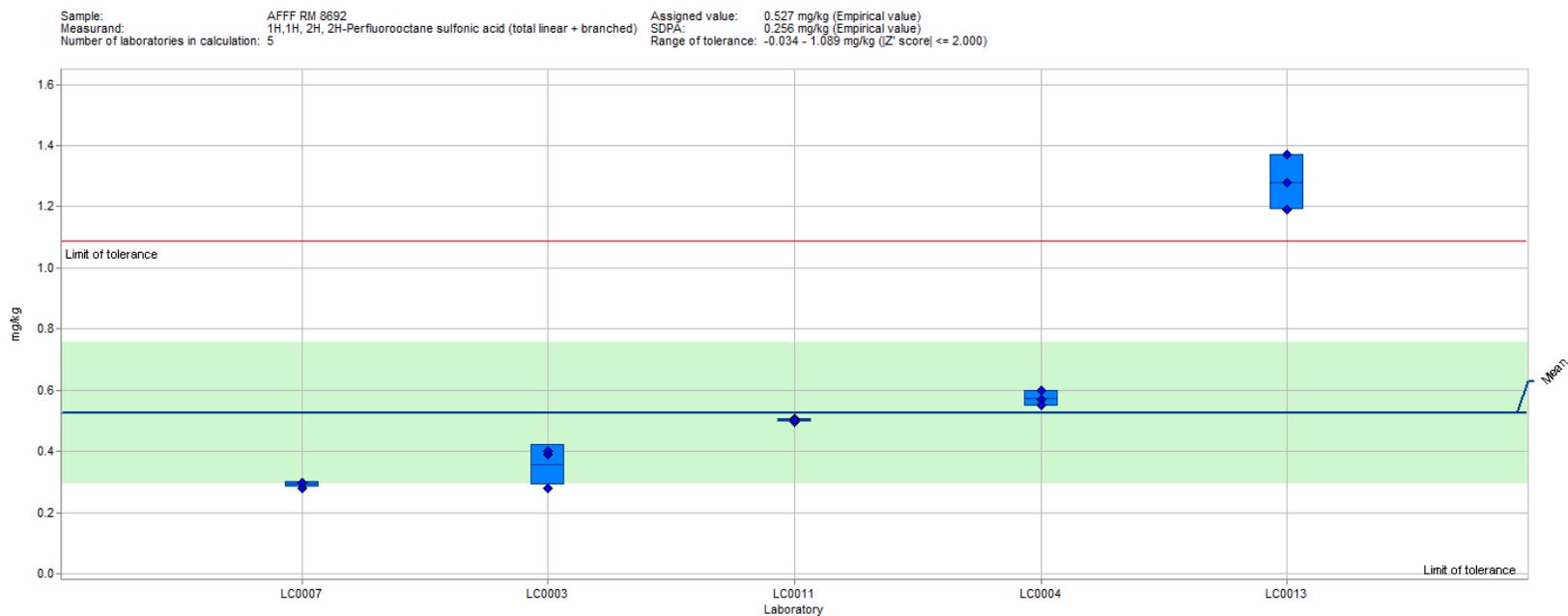


Figure 18-3. 6:2 FTS in candidate RM 8692 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation III. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Sample: AFFF RM 8693
 Measurand: 1H,1H, 2H, 2H-Perfluorooctane sulfonic acid (total linear + branched)
 Number of laboratories in calculation: 6
 Assigned value: 132.722 mg/kg (Empirical value)
 SDPA: 34.038 mg/kg (Empirical value)
 Range of tolerance: 59.253 - 206.191 mg/kg ($|Z'_{\text{score}}| \leq 2.000$)

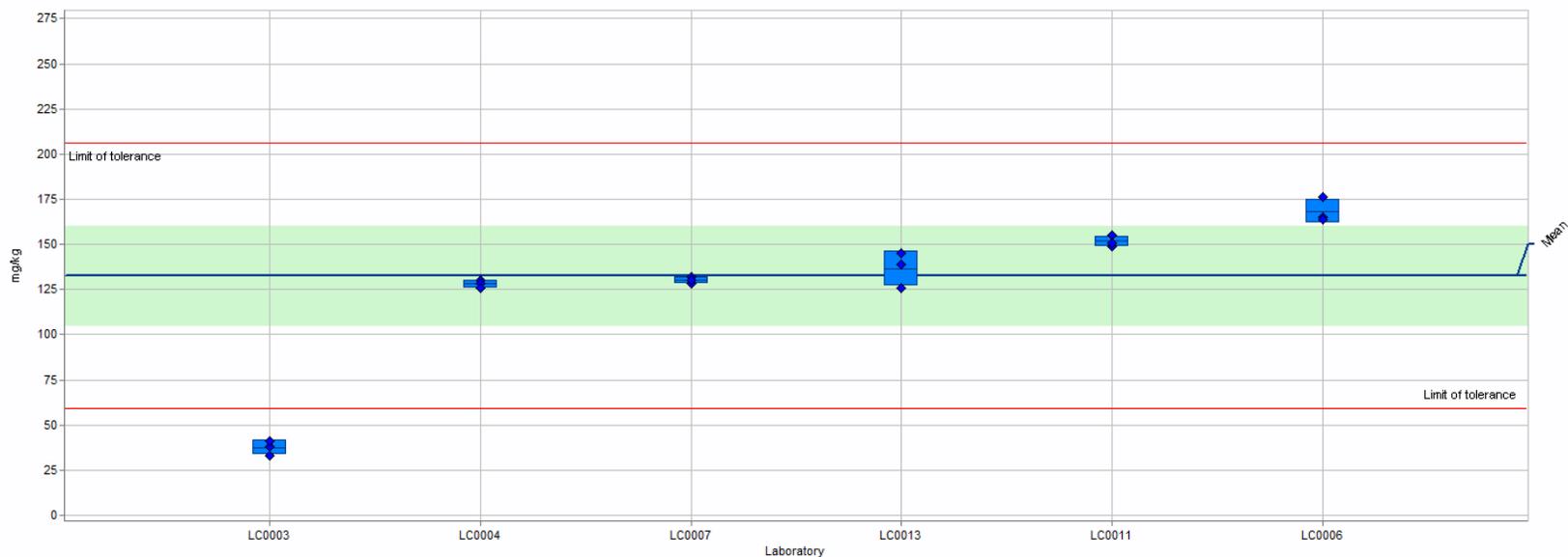


Figure 18-4. 6:2 FTS in candidate RM 8693 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation IV. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

1H,1H, 2H, 2H-Perfluorodecane sulfonic acid (8:2 FTS)

- Four laboratories reported results for 8:2 FTS in candidate RMs 8690, 8691, and 8693. Three laboratories reported results for 8:2 FTS in candidate RM 8692.
- The between-laboratory variability was poor for candidate RM 8690, 8691, and 8693 (80 %, 113 %, and 517 % RSD, respectively).
- One laboratory reported results of over 250 mg/kg in candidate RM 8693. Since this was substantially higher compared to the other reported values (around 0.112 mg/kg), we went back to the material and found a chemical interferant that when not fully separated from 8:2 FTS, gave a substantially higher reported value. This chemical interferant is not 8:2 FTS, but does have a similar retention time and primary MS/MS transition.

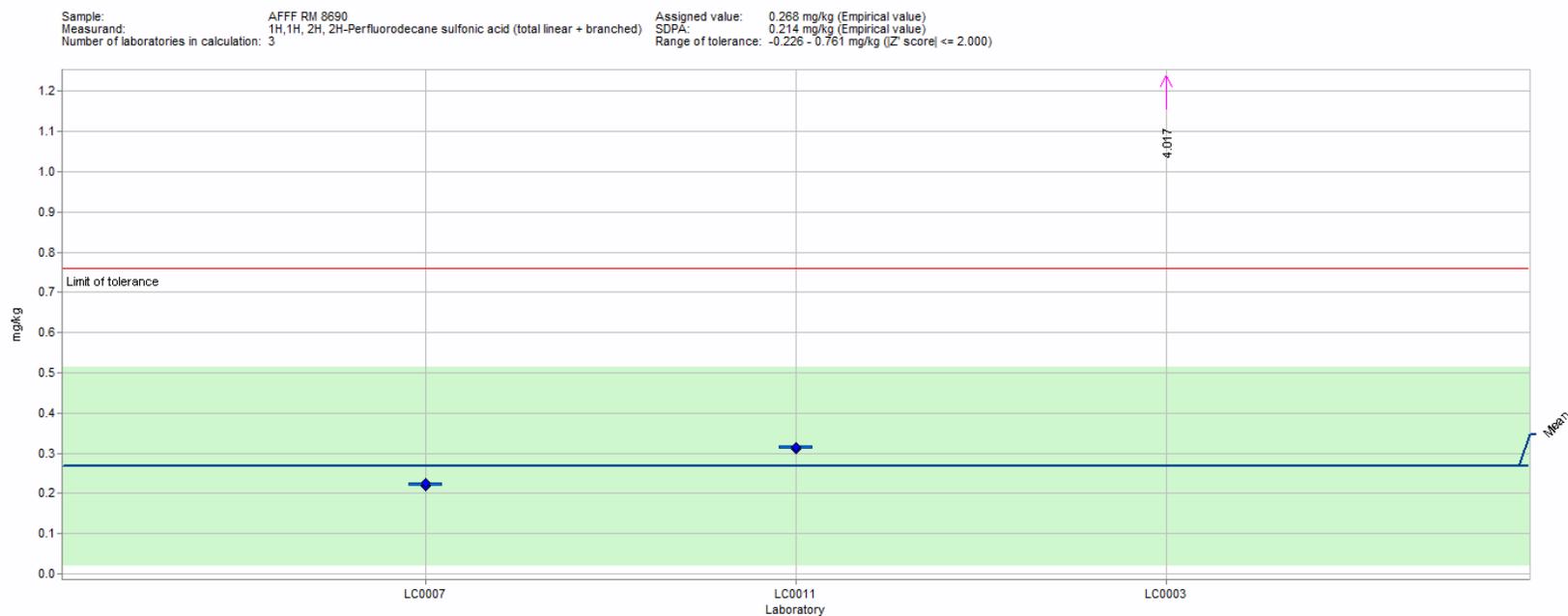


Figure 19-1. 8:2 FTS in candidate RM 8690 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation I. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.



Figure 19-2. 8:2 FTS in candidate RM 8691 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation II. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Sample: AFFF RM 8693
 Measurand: 1H,1H,2H,2H-Perfluorodecane sulfonic acid (total linear + branched)
 Number of laboratories in calculation: 4

Assigned value: 0.112 mg/kg (Empirical value)
 SDPA: 0.579 mg/kg (Empirical value)
 Range of tolerance: -1.183 - 1.407 mg/kg ($|Z'_{\text{score}}| \leq 2.000$)



Figure 19-3. 8:2 FTS in candidate RM 8693 Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Film-Forming Foams (AFFF) Formulation IV. In this view, individual laboratory data are plotted (diamonds) with the individual laboratory standard deviation (rectangle). The solid blue line represents the consensus mean, and the green shaded region represents the 95 % confidence interval for the consensus mean. The red solid lines represent the consensus range of tolerance, calculated as the values above and below the consensus mean that result in an acceptable Z'_{comm} score, $|Z'_{\text{comm}}| \leq 2$.

Conclusions

There are technical recommendations based on feedback from some of the participants in this study.

- 1) Laboratories reported foaming of the candidate materials which made it difficult to subsample from the vials. It is recommended that the candidate RMs be diluted (approximately 1:10 v:v) to make material handling easier and reduce the chance of foaming.
- 2) Given the large % RSD values for most analytes, measuring PFAS in AFFFs is clearly a challenging measurement. AFFF RMs will be useful for to validate future measurements of PFAS in AFFF or other commercial formulations.
- 3) The use of matrix RMs for method validation and quality assurance of the measurement process is recommended.

References

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²Reiner, J.L.; O'Connell, S.G.; Butt, C.; Mabury, S.; Small, J.; De Silva, A.O.; Muir, D.; Delinsky, A.; Strynar, M.J.; Lindstrom, A.B.; Reagen, W.; Malinsky, M.; Schäfer, S.; Kwadijk, C.; Schantz, M.M.; Keller, J.M. Determination of Perfluorinated Alkyl Acid Concentrations in Biological Standard Reference Materials. *Anal Bioanal Chem.* 2012. 9, 2683-2692.

³Reiner, J.L.; Blaine, A.C.; Higgins, C. P.; Huset, C.; Jenkins, T.M.; Kwadijk, C.; Lange, C.; Muir, D.C.G.; Reagen, W.; Rich, C.; Small, J.M.; Strynar, M.J.; Washington, J.W.; Yoo, H.; Keller, J.M. Polyfluorinated Substances in Abiotic Standard Reference Materials. *Anal Bioanal Chem.* 2015. 407, 2975-2983.

⁴ISO 13528:2015(E), *Statistical methods for use in proficiency testing by interlaboratory comparisons*, pp. 53-54.