Table 11-6. PFAS Analytical Data Usability Table

This table belongs with the ITRC PFAS Tech Reg Document. The ITRC intends to update this table periodically as new information is gathered. This table includes a summary of key points from data validation guidance documents, USEPA (2018, 2019, 2020a, b) and USDOD (2021), focused on the analytical data results. It is not intended to address all data usability requirements across site investigation, risk assessment, or remediation projects. Information in the table that is not PFAS-specific is included for context when reviewing PFAS analytical results.

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Users who identify updates to the material in this table are encouraged to send that information to itrc@itrcweb.org

QC Check	QC Check Description	Data Quality Indicator	Potential Impact of Data Usability
Preparation of Aqueous Samples	Entire sample volume extracted including bottle rinsate (if method requires)	Analytical Accuracy	• If the bottle not rinsed with methanol, results may be biased low for ≥ C10 PFAS • If bottle is subsampled for extraction, results may be biased low
Preparation of Aqueous Samples with Solids	Aqueous or Aqueous + solids analyzed	Analytical Accuracy/Representativeness	• Project objectives will dictate whether only the aqueous or the aqueous + solids should be extracted and analyzed • If lab doesn't follow project requirements, data may be uncertain or objectives may not have been achieved
Preparation of Solid Samples	Sample thoroughly homogenized prior to sub-sampling for extraction	Analytical Accuracy/Representativeness	• If not homogenized, data may not be representative and may be uncertain
Preparation of Biota Samples	Sample prepared as defined by the project prior to sub-sampling for extraction	Analytical Accuracy	• Project objectives will dictate how samples should be prepared (e.g., filleted fish or whole fish) • If lab doesn't follow project requirements, data may be uncertain or objectives may not have been achieved

QC Check	QC Check Description	Data Quality Indicator	Potential Impact of Data Usability
Sample Preservation (*)	Chemical preservative (e.g., Trizma or ammonium acetate) added to drinking water samples during collection. Aqueous & solid samples transmitted to the lab cooled to method-required temperature (e.g., <10°C).	Field Collection Accuracy	• If drinking water samples are not chemically preserved, data may be biased (precursors may be biased low while other PFAAs may be biased high due to transformation from precursors) • If the cooler temperature exceeds method or project criteria, data may be biased low or high, depending on the analyte
Field Reagent Blank (FRB) (*)	To check for ambient levels of PFAS; PFAS results < LOQ	Analytical and Field Collection Accuracy	• If certain PFAS compounds are detected in FRB, those PFAS in samples associated with FRB may be biased high or may be false positives
Equipment Rinse Blank (ERB) (*)	To check equipment for potential PFAS contamination and effectiveness of decontamination procedure; PFAS results < LOQ	Analytical and Field Collection Accuracy	• If certain PFAS compounds are detected in ERB, those PFAS in samples associated with ERB may be biased high or may be false positives
Field Duplicate (FD) (*)	Sample/FD RPD defined by regulatory program and/or project requirements	Sampling and Analytical Precision/Representativeness	• If RPD exceeds criteria, potential uncertainty in PFAS result for site location
Holding Time (HT)	Time from collection to extraction and/or analysis	Analytical Accuracy	• If HT exceeded, data may be biased (precursors may be biased low while other PFAAs may be biased high due to transformation from precursors)
Method Blank (MB)	To check for potential PFAS contamination during preparation and analysis; MB acceptance criteria defined by method, regulatory program and/or project requirements	Analytical Accuracy	MB results affect all samples in the QC batch. • If certain PFAS compounds are detected in MB, those PFAS in all samples in the affected batch may be biased high or may be false positives

QC Check	QC Check Description	Data Quality Indicator	Potential Impact of Data Usability
Instrument Blank (IB)	To check instrument for potential PFAS contamination; IB acceptance criteria defined by method, regulatory program and/or project requirements	Analytical Accuracy	• If certain PFAS compounds are detected in IB, those PFAS in all samples in the affected batch may be biased high or may be false positives
Surrogate (non-Isotope Dilution or non-ID)	Surrogate recoveries within method, project, or regulatory requirements	Analytical Accuracy	Professional judgment should be used in judging impact of a non-isotope dilution surrogate on PFAS. • If recovery high, no effect on non-detects but PFAS detects may be biased high in affected sample • If recovery low but > 10%, PFAS may be biased low in affected sample • If recovery < 10%, non-detects may not be usable (false negatives) and detects may be biased low in affected sample
Extraction Internal Standards (EIS) (Isotope Dilution or ID)	Isotope recoveries within method, project, or regulatory requirements	Analytical Accuracy	Isotope dilution recovery outside criteria only affects those specific PFAS quantitated using that isotope. Professional judgment should be used in evaluating impact of isotope dilution recovery on PFAS. • If recovery high, no effect on non-detects but detects for the specific PFAS may have indeterminate bias in affected sample • If recovery low but > 10%, nondetects and detects for specific PFAS may have indeterminate bias in affected sample • If recovery < 10%, non-detects for specific PFAS may not be usable (false negatives) and detects for specific PFAS may have indeterminate bias in affected sample
Laboratory Control Sample (LCS) or Ongoing Precision and Recovery (OPR) Sample, Low-Level LCS (LLLCS) and Low-Level OPR (LLOPR)	All target PFAS spiked and recoveries within method, project, or regulatory requirements	Analytical Accuracy	OPR/LCS results affect all samples in the QC batch. • If a PFAS OPR/LCS recovery high, no effect on non-detects but detects for that PFAS may be biased high • If a PFAS OPR/ LCS recovery low but > 10%, all data for that PFAS may be biased low • If a PFAS OPR/LCS recovery < 10%, non-detects for that PFAS may not be usable (false negatives) and detects may be biased low Same possible effects apply to LLOPR or LLLCS.

QC Check	QC Check Description	Data Quality Indicator	Potential Impact of Data Usability
Matrix Spike and Matrix Spike Duplicate (MS/MSD)	All target PFAS spiked; recoveries and MS/MSD precision within method, project, or regulatory requirements	Analytical Accuracy and Precision	MS/MSD results affecting parent sample results may be applied to other samples if a physical interference can be observed in sample or chromatography. • If a PFAS MS recovery high, no effect on non-detects but detect for that PFAS may be biased high • If a PFAS MS recovery low but > 10%, data for that PFAS may be biased low • If a PFAS MS recovery < 10%, non-detect for that PFAS may not be usable (false negatives) and a detect may be biased low • If MS/MSD RPD > criteria, data for that PFAS is imprecise and uncertain
Injection Internal Standard (IIS) or Non-extracted Internal Standard (NIS)	Added to every extract prior to LC/MS-MS analysis. ***Non-ID methods***: used for quantitation of analytes ***ID methods***: used for quantitation of isotopes only Abundance within method or project requirements	Analytical Accuracy	• If IS outside criteria in non-ID methods, affected analytes may be biased • If IS outside criteria for ID methods, no direct effect on sample data if isotope recoveries acceptable
Mass Calibration	Mass calibration and mass calibration verification must meet the method or project requirements	Analytical Accuracy	• If not calibrated and verified, data may be unusable
Ion Transitions (Precursor → Product)	Quantitation Ion (QI) transitions must be per method or project requirements Confirmation Ion (CI) transitions should be monitored for all compounds, if applicable (**)	Analytical Accuracy	• Data may be biased if different ion transitions used

QC Check	QC Check Description	Data Quality Indicator	Potential Impact of Data Usability
Initial Calibration (ICAL)	Minimum number of concentration levels for branched and linear isomers (if available) for each PFAS as required by the method or project requirements with lowest level at concentration ≤ LOQ Acceptance criteria for ICAL per method or project requirements Ion Ratio (e.g., QI/CI abundances) criteria per method or project requirements Signal to Noise Ratio (S/N) for QIs and CIs within method or project requirements (**)	Analytical Accuracy	• If ICAL doesn't meet criteria, data may be biased or may not be usable
Initial Calibration Verification (ICV)	Second source standard concentrations within method or project requirements	Analytical Accuracy	• If ICV doesn't meet criteria, data may be biased or if <10% recovery, may not be usable
Instrument Sensitivity Check (ICS)	Concentration at LOQ Recovery within method or project requirements	Analytical Accuracy	• If ICS doesn't meet criteria, data may be biased or if <10% recovery, may not be usable

QC Check	QC Check Description	Data Quality Indicator	Potential Impact of Data Usability
Retention Time (RT) Window	RT of PFAS set as RT in mid-point ICAL standard on days an ICAL is performed or RT of PFAS in initial CCV on analytical sequences to follow RT in sample should fall within method or project requirements RT of PFAS compared to associated EIS within method or project requirements	Analytical Accuracy	• If RT outside criteria, data may be biased or may be false positive
Continuing Calibration Verification (CCV) or Calibration Verification (CV)	Standard near mid-level concentration Recovery within method or project requirements	Analytical Accuracy	• If CCV doesn't meet criteria, data may be biased or may not be usable • If %D indicates enhanced sensitivity to detection of PFAS, no effect on non-detects but associated detected results may be biased high • If %D indicates loss in sensitivity, associated detects and non-detects may be biased low

QC Check	QC Check Description	Data Quality Indicator	Potential Impact of Data Usability
Quantitation and General Reporting Issues	Average RRF or calibration equation from ICAL used to quantitate results QI and CI should be present and RT must maximize within method or project requirements (**) Signal to Noise Ratio (S/N) for QI and CI within method or project requirements (**) QI/CI ratio should be within method or project requirements (**) Results reported < LOQ should be qualified "J" by the lab Samples with PFAS reported above the calibration range should be diluted to bring response within the calibration range	Analytical Accuracy	• If a lab result is qualified "E" or "J" by the lab indicating quantitation outside the calibration range, the result is uncertain with indeterminate bias • If S/N criteria not met, data may be biased • If QI/CI ion ratio in sample is outside ICAL established criteria, data may be biased (possible ion suppression or interferences) • If QI and CI RT are not within method or project requirements, data may be biased • If branched isomers not included in quantitation, data for the PFAS may be biased low

Notes:

* Review method, project, and regulatory program requirements

** Non-Isotope Dilution (non-ID) methods (e.g., EPA Method 537.1) do not require monitoring of Confirmation Ions (CI)

References:

USEPA. 2018. Data Review and Validation Guidelines for Perfluoroalkyl Substances (PFASs) Analyzed Using EPA Method 537. EPA 910-R-18-001.

USEPA. 2019. Technical Brief: Per- and Polyfluoroalkyl Substances (PFAS): Reviewing Analytical Methods Data for Environmental Samples. EPA 600-F-19-056.

USEPA. 2020a. National Functional Guidelines for Organic Superfund Methods Data Review. EPA 540-R-20-005. Washington, DC: U.S Environmental Protection Agency. https://www.epa.gov/sites/default/files/2021-03/documents/nfg_for_organic_superfund_methods_data

USEPA. 2020b. National Functional Guidelines for High Resolution Superfund Methods Data Review. EPA 542-R-20-007. Washington, DC: U.S. Environmental Protection Agency. https://www.epa.gov/sites/default/files/2021-03/documents/nfg_for_hrsm_superfund_methods_data_review_november_2020.pdf.
USDOD. 2021. Module 3: Data Validation Procedure for Per- and Polyfluoroalkyl Substances Analysis by Quality Systems Manual for Environmental Laboratories (QSM) Table B-15. Version 5.4: Department of Defense/Department of Energy.