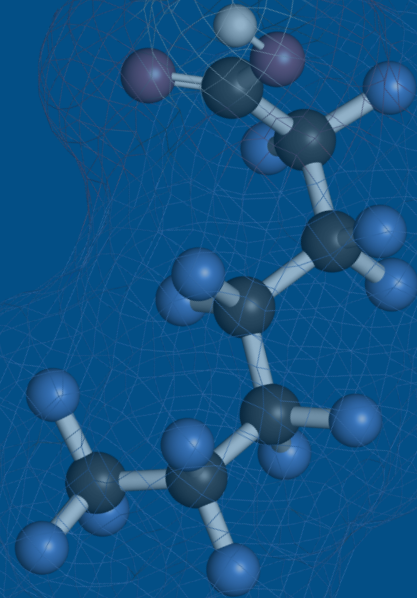


Assessing PFAS Data Quality for Compliance to USEPA Method 1633A



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Ensuring the lab's quality control data provided with the sample results meet certain standards is necessary when assessing or remediating a site. Environmental practitioners use quality procedures such as field blanks, trip blanks, or field duplicates and the laboratories provide quality data such as control standards, laboratory duplicates and sample spikes amongst others. These requirements ensure the data can be used and ensure the data are fit for intended purpose. This becomes even more important if data are to be used in a legal setting where defensible data are of critical importance.



PFAS analytical methodology uses Extracted Internal Standards (EIS) to assess how the method is performing against the sample. For this reason, it is important that EIS recovery data is reviewed for every sample.

What is an EIS? USEPA Method 1633 describes it as the following: Extracted Internal Standard (EIS) – An isotopically-labeled analog of a target analyte that is structurally identical to a native (unlabeled) analyte. The EIS compounds are added to the sample at the beginning of the sample preparation process and are used to quantify the native target analytes.

The EIS recoveries ideally would all be near 100% and determine that the PFAS methodology was fit for purpose for the samples analysed. In reality, there are matrix affects, pH changes, differing moisture content – all which can affect these recoveries and why USEPA Method 1633A has recovery limits typically in the range of 40-130%. However, some are as low as 5%, and some as high as 365%, depending on matrix. Tables 6 and 8 of the [USEPA 1633A method](#) detail the acceptance criteria.

The EIS recoveries indicate how the submitted samples are performing against the method. The laboratory reported results for target analytes are recovery corrected using this EIS method of quantification. As an example, to determine an EIS for PFOS, a synthetic EIS PFOS compound is added prior to the sample undergoing the analytical process. If the EIS recovery at the end of the analytical process is 50% and the PFOS compound is 1 ug/kg, then the laboratory would adjust the result to 2 ug/kg to account for the 50% loss in the EIS.

Painstakingly reviewing EIS recoveries and ensuring there are no issues, while important, can take significant time if done manually. The EQulS® Data Qualification Module (DQM) can make this task much easier. EIS recovery data can be assessed to a method such as USEPA Method 1633A as sample results are received.

Assessing EIS Data: An Example

The table to the right is a small example dataset of PFAS results. We quickly see there are some EIS recovery issues flagged with 18% for PFOA and 30% for PFOS. This could be difficult to see in a dataset of hundreds or thousands of samples.

EQulS DQM can quickly highlight the data issues. The DQM image below shows this when looking at one sample, MW-2-05-2024.

Sample Date	14 May 2024	14 May 2024	14 May 2024
Location	MW1	MW2	MW3
Sample Name	MW1-05-2024	MW2-05-2024	MW3-05-2024
Analyte			
Perfluorobutanesulfonic Acid (PFBS)	0.12 ug/L	1.34 ug/L	0.88 ug/L
Perfluorohexanoic acid (PFHxA)	0.22 ug/L	0.55 ug/L	1.29 ug/L
Perfluorooctanesulfonic Acid (PFOS)	0.08 ug/L	0.98 ug/L	0.09 ug/L
Perfluorooctanoic acid (PFOA)	0.47 ug/L	2.56 ug/L	4.21 ug/L
13C3-PFBS (surr.)	145 %	123 %	130 %
13C5-PFHxA (surr.)	187 %	115 %	56 %
13C8-PFOS (surr.)	106 %	30 %	192 %
13C8-PFOA (surr.)	18 %	110 %	22 %

Sample	Analyte	Result	Apply Remark	Apply
MW2-05-2024	Perfluorooctanoic acid (PFOA)	2.56		
MW2-05-2024	Perfluorooctanesulfonic Acid (PFOS)	0.98	Associated Result from: Associated Detected Result of a Surrogate Recovery less than the Lower Surrogate Cutoff.	J-
MW2-05-2024	Perfluorohexanoic acid (PFHxA)	0.55		
MW2-05-2024	Perfluorobutanesulfonic Acid (PFBS)	1.34		
MW2-05-2024	13C8-PFOS (surr.)	30	The Surrogate Recovery is less than the Lower Surrogate Cutoff.	J-
MW2-05-2024	13C8-PFOA (surr.)	110		
MW2-05-2024	13C5-PFHxA (surr.)	115		
MW2-05-2024	13C3-PFBS (surr.)	123		

We have a result of 0.98 for PFOS and a remark that the result is affected by a low surrogate cutoff; in other words, the recovery for the surrogate was below our set limit. This aligns with the flagged recovery of the PFOS EIS at 30%. We set DQM to insert a qualifier code of "J-" next to any such result.

This summary has importantly not flagged any other results as the EIS recoveries are within acceptance criteria. This is expected as we only want to flag the results affected by the EIS failures and the PFOS EIS failure only affects the PFOS result. While this is fairly simple for one sample, see below how the three samples in our original dataset appear:

Sample	Analyte	Result	Apply Remark	Apply
MW1-05-2024	13C3-PFBS (surr.)	145	The Surrogate Recovery exceeds the Upper Cutoff.	R
MW1-05-2024	13C5-PFHxA (surr.)	187	The Surrogate Recovery exceeds the Upper Cutoff.	R
MW1-05-2024	13C8-PFOA (surr.)	18	The Surrogate Recovery is less than the Lower Surrogate Cutoff.	J-
MW1-05-2024	13C8-PFOS (surr.)	106		
MW1-05-2024	Perfluorooctanesulfonic Acid (PFOS)	0.08		
MW1-05-2024	Perfluorohexanoic acid (PFHxA)	0.22	Associated Result from: Associated Detected Result of a Surrogate Recovery exceeding the Upper Cutoff.	R
MW1-05-2024	Perfluorooctanoic acid (PFOA)	0.47	Associated Result from: Associated Detected Result of a Surrogate Recovery less than the Lower Surrogate Cutoff.	J-
MW1-05-2024	Perfluorobutanesulfonic Acid (PFBS)	0.12	Associated Result from: Associated Detected Result of a Surrogate Recovery exceeding the Upper Cutoff.	R
MW2-05-2024	13C3-PFBS (surr.)	123		
MW2-05-2024	13C5-PFHxA (surr.)	115		
MW2-05-2024	13C8-PFOA (surr.)	110		
MW2-05-2024	13C8-PFOS (surr.)	30	The Surrogate Recovery is less than the Lower Surrogate Cutoff.	J-
MW2-05-2024	Perfluorooctanesulfonic Acid (PFOS)	0.98	Associated Result from: Associated Detected Result of a Surrogate Recovery less than the Lower Surrogate Cutoff.	J-
MW2-05-2024	Perfluorohexanoic acid (PFHxA)	0.55		
MW2-05-2024	Perfluorooctanoic acid (PFOA)	2.56		
MW2-05-2024	Perfluorobutanesulfonic Acid (PFBS)	1.34		
MW3-05-2024	13C3-PFBS (surr.)	130		
MW3-05-2024	13C5-PFHxA (surr.)	56		
MW3-05-2024	13C8-PFOA (surr.)	22	The Surrogate Recovery is less than the Lower Surrogate Cutoff.	J-
MW3-05-2024	13C8-PFOS (surr.)	192	The Surrogate Recovery exceeds the Upper Cutoff.	R
MW3-05-2024	Perfluorooctanesulfonic Acid (PFOS)	0.09	Associated Result from: Associated Detected Result of a Surrogate Recovery exceeding the Upper Cutoff.	R
MW3-05-2024	Perfluorohexanoic acid (PFHxA)	1.29		
MW3-05-2024	Perfluorooctanoic acid (PFOA)	4.21	Associated Result from: Associated Detected Result of a Surrogate Recovery less than the Lower Surrogate Cutoff.	J-
MW3-05-2024	Perfluorobutanesulfonic Acid (PFBS)	0.88		

The picture becomes more complex with multiple recovery issues. This is only assessing EIS recoveries. There are many other quality checks in DQM, including field duplicates and laboratory control standards.

Table summaries can quickly outline the data quality issues and communicate clearly where the issues are and for what reason.

Sample Code	MW1-05-2024	MW2-05-2024	MW3-05-2024
Analyte			
Perfluorobutanesulfonic Acid (PFBS)	0.12 ug/L R This result is associated with the failing Surrogate.	1.34 ug/L	0.88 ug/L
Perfluorohexanoic acid (PFHxA)	0.22 ug/L R This result is associated with the failing Surrogate.	0.55 ug/L	1.29 ug/L
Perfluorooctanesulfonic Acid (PFOS)	0.08 ug/L	0.98 ug/L J- This result is associated with the failing Surrogate.	0.09 ug/L R This result is associated with the failing Surrogate.
Perfluorooctanoic acid (PFOA)	0.47 ug/L J- This result is associated with the failing Surrogate.	2.56 ug/L	4.21 ug/L J- This result is associated with the failing Surrogate.
13C3-PFBS (surr.)	145 % R Surrogate recovery of 145% > Upper Cutoff of 135%.	123 %	130 %
13C5-PFHxA (surr.)	187 % R Surrogate recovery of 187% > Upper Cutoff of 130%.	115 %	56 %
13C8-PFOS (surr.)	106 %	30 % J- Surrogate recovery of 30% < Lower Cutoff of 40%.	192 % R Surrogate recovery of 192% > Upper Cutoff of 130%.
13C8-PFOA (surr.)	18 % J- Surrogate recovery of 18% < Lower Cutoff of 40%.	110 %	22 % J- Surrogate recovery of 22% < Lower Cutoff of 40%.

Conclusion

A systematic approach to data quality assessment is needed that compares your data to agreed quality guidelines. This short example of DQM with PFAS data demonstrates the overwhelming task without a standard approach for large amounts of field and lab data.

About the Author

Mr. Tomlins has more than 15 years of senior management experience and has worked with EQulS for more than a decade. He has been a voice in the environmental industry, presenting at technical conferences and on social media on a variety of topics including PFAS analytical methodology and quality assessments.