

OTM-45 Frequently Asked Questions (FAQs)

- 1. If the intent of the proof blank is to check the laboratory glassware cleaning procedures, including the sampling media makes it impossible to determine a specific source of contamination if contamination is identified. Can you please comment on the intent of the proof blank?**

The Sampling Train Proof Blank (STPB) is one in a series of quality control samples (blanks) collected to evaluate the presence of and potentially isolate the source of sample collection related contamination. The purpose of the STPB combines the glassware that is cleaned before sampling, with all of other sample train assembly, preparation and recovery procedures. The STPB samples, in conjunction with the Laboratory Sample Media Blanks (LSMB) and Field Sampling Media Blanks (FSMB) serve to evaluate the presence of and potentially isolate the source of sample collection related contamination.

- 2. When is the field blank required/recommended?**

The Sampling Train Field Blank (STFB) is intended for when sampling train glassware that has been initially cleaned is re-used after sample collection. The primary tenet is that sufficient, cleaned glassware will be taken to the field such that sampling glassware will not need to be re-used. However, it is recognized that this cannot always be the case. While not required, the collection of a field blank is highly recommended such that the data quality of emission samples collected using re-used glassware can be evaluated.

- 3. Suggest calling impinger contents "DI water" and rinsing solvents "methanol/5% NH₄OH" to distinguish from impinger contents, which would be interpreted as contents after sampling.**

OTM-45 has been revised to better distinguish between the DI water and collected condensate and the methanol/5% NH₄OH

- 4. Why is a titanium nozzle specified in OTM-45?**

A sampling option is needed for potential high temperature sampling applications. Borosilicate glass or quartz (nozzle combined with liner) are alternatives limited only by their temperature suitability. Titanium is an option for where temperatures exceed where borosilicate glass or quartz can be used. Stainless-steel (SS) is not appropriate for measurement of organic compounds at high temperatures.

- 5. Are PTFE filter supports and other PTFE materials allowed?**

Yes, when necessary. However, they must be evaluated as sources of contamination. PTFE materials (filter supports, ferrules, fittings, transfer lines, Teflon tape, etc.) have been shown or are suspected to be sources of PFAS contamination. Moreover, contamination may be evident on the surface as well as upon heating. In many instances these materials can be conditioned so as to eliminate or minimize contamination. In order

for these materials to be used, they should be cleaned or conditioned and then checked for contamination. In addition, if these materials are used, they must be included in the Sampling Train Proof Blank (STPB), which requires heating of all train components for a time duration consistent with that of an actual run. The STPB must meet the requirements of OTM-45 Section 9.2.2.1.

6. How was the 30 day holding time established?

In the absence of empirical data supporting OTM-45 sample holding times, a 30 day hold time has been established until such time as empirical data are available. The 30 day hold time is consistent with other organic sampling method sample hold times.

7. Please describe how OTM-45 has been made compatible with Office of Water Method 1633.

As now stated in revised OTM-45, the OTM-45 target analytes and respective pre-extraction isotopic labeled compounds used have been revised to be inclusive and consistent with EPA Office of Water (OW) Method 1633. As a result, some commercially available standard mixes used for 1633 analyses may also be suitable for performing OTM-45 sample analyses. However, it should be noted that in order to expand the presampling standard compounds now required, the specific OTM-45 pre-analysis standards and Method 1633 non-extracted internal standards (NIS) are not consistent and separate mixes will be required.

8. Does the availability of PFAS standards limit what PFAS compounds can be targeted for analysis?

Yes. This includes both natives and labeled isotopologues. As new compounds of interest are identified, they will need to be synthesized and made available. There is currently a need for additional labeled isotopologues (e.g., PFOS and HFPO-DA) to support assessment of overall measurement data quality. With the incorporation of presampling standards, 3 different labeled isotopologue standards are needed. Their availability is limited. As a result, the number of presampling standards that can be incorporated into the method is limited. In addition, not all current native target analytes have labeled isotopologues for quantitative purposes. Additional labeled isotopologues are needed for true isotope dilution quantitation.

9. Have the method's methanolic solutions changed?

No. The OTM-45 extraction and cleanup reagents have been renamed "methanolic ammonium hydroxide" with the actual concentration of ammonium hydroxide in solution as opposed to the percentage of concentrated ammonium hydroxide in solution. This revision is also consistent with the nomenclature used in the EPA OW Method 1633.

10. Can additional analytes be added to the OTM-45 target list?

Yes. The target list currently presented in OTM-45 represents the minimum compounds to be targeted for analysis. Additional target analytes of interest may be included. Ideally, any target PFAS compounds added will be quantified by isotope dilution, meaning that it has a labeled isotopologue for appropriate quantitation. However, that may not always be possible. In those instances where an isotopologue is not available, the isotopically labeled PFAS compound used for quantitation should be similar in chemical composition and properties and its recovery performance assessed and deemed appropriate until such time as an isotopologue has been synthesized and made commercially available. Any analytes added to the OTM-45 target list should be identified and the approach used for the quantitation described.

11. Describe how OTM-45 is a performance-based method?

EPA's performance-based methods are intended to establish Measurement Quality Objectives (MQOs) that, when achieved, result in the collection of measurements data of known and acceptable quality. These methods are intended to offer flexibility and encourage innovation, where appropriate, with respect to the procedures and equipment necessary to achieve these MQOs. With respect to OTM-45, specific MQOs have been established for assessing individual as well as overall measurement performance. These are presented in Section 9 as well as Table 45.5. While some MQOs do have specific approaches associated with them presented (e.g., blanks, MDLs, etc), others such as the sample extraction, sample concentration, etc equipment and approaches, are amenable to alternative equipment and procedures as long as the critical MQOs for pre-extraction and pre-sampling standard recoveries can be achieved. In these instances, the procedures presented, though somewhat prescriptive, are provided so that procedures that have data supporting their successful application can be provided at this time.

12. For HFPO-DA (Gen-X) to be a viable target analyte, the probe and filter box (and heated sample transfer line if used) must remain several degrees below 90°C (194°F) to avoid thermal decomposition (decarboxylation).

OTM-45 has been modified to limit the probe, filter box and heated sample transfer line (if used) heated temperature to 85°C for those emissions sampling environments conducive to HFPO-DA being potentially present. That is, non-combustion, (e.g., industrial, chemical manufacturing) emission sources. The intent is for the OTM-45 sampling train to not contribute to thermal decomposition, though HFPO-DA thermal decomposition may indeed be occurring prior to the sampling location. As a result, OTM-45 has been revised to include probe and filter operating temperatures for both combustion and non-combustion sampling environments.

13. The calculation of breakthrough (BT) can result in a “false positive” at the low concentration limits identified in 9.1.6.

EPA acknowledges that BT can indeed be artificially biased at low fraction 1-3 mass totals, especially at similar, fraction 4 mass levels, possibly as a result of contamination. OTM-45 has been revised to better minimize that scenario.

14. Section 9.5 of the method presents the requirements for flagging compiled data. Are these flags expected to be appended to data that already carry "J", "E", and "B" flags? Are these flags expected to be added to the stack sampling reports?

Yes, when fractions are summed and reported, the expectation is that the stack sampling reports include data flags associated with both laboratory reporting and fraction summing reporting.