

# Confirmation Study: No Background PFAS Contamination with EDGE PFAS

## Abstract

Per- and polyfluoroalkyl substances (PFAS) are a group of compounds that are used in many different industries due to their favorable properties, such as heat and grease resistance. Their resistance and prevalence in manufacturing has presented hurdles for their detection, as they are used in the manufacture of materials typically found in Liquid Chromatography–Mass Spectrometry (LC-MS) systems. This has caused LC-MS companies to suggest the substitution of these materials. Similarly, the EDGE®, an automated extraction system, was modified to prevent the contamination of the resultant extracts by PFAS. The EDGE PFAS™ system was designed specifically for PFAS sample prep and, when used with Q-Disc® PFAS, eCleanUP™, and a Q-Cup®, has been shown to be PFAS-free. A series of washes, soaking, and exposure to solvents typically used in PFAS extraction were performed and the resulting extract analyzed. It was found that EDGE PFAS, as well as the Q-Disc PFAS, eCleanUP, and Q-Cups were free of PFAS to the limit of detection. Thus, the EDGE PFAS is an excellent choice for laboratories that would like to automate their PFAS extractions without subjecting their samples to unwanted contamination.

## Introduction

Per- and polyfluoroalkyl substances (PFAS) are a class of man-made chemicals that are used in a wide variety of industries. They are resistant to high temperatures, stains, and grease, allowing for the performance of products such as nonstick cookware, firefighting foam, and stain-resistant fabrics and furnishings. PFAS molecules are made of linked carbon atoms with fluorine atoms branching off of the main chain. The strong bond of carbon and fluorine makes these compounds extremely stable. Their stability and widespread use presents difficulties for their analytical measurement, as they often leach out of the common materials used in analysis systems. Because of this, LC-MS companies recommend substituting these components with other materials, such as replacing Teflon®-coated solvent lines with polypropylene tubing, to remove the background contamination.

Built on the EDGE platform, the EDGE PFAS is an automated extraction system that has been shown to rapidly and efficiently extract PFAS molecules from samples. To ensure that the EDGE PFAS was free of any PFAS contamination, several substitutions were made to a traditional EDGE

system: any potentially contaminated materials (tubing) were exchanged, extra precautions were taken to prevent contamination (side enclosure), and consumables and Q-Cups were assessed for any of PFAS content.

The system was found to be free of any PFAS contamination, and the consumables and Q-Cups were also found to have no contamination. Thus, the modifications done to the EDGE PFAS ensure a clean background for the extraction of PFAS from a variety of matrices.

## Materials and Methods

### EDGE Modifications

Typically, the EDGE has solvent tubing lines, a dispense line, and other lines along its fluidic pathway made of polytetrafluoroethylene (PTFE). However, PFAS compounds, such as perfluorooctanoic acid, are involved in the manufacture of PTFE. This warrants the replacement of these lines. PEEK tubing was substituted for the PTFE solvent tubing, as the PEEK tubing is flexible enough to be used for this purpose. The PTFE tubing used in the dispense line and other lines along the pathway were substituted with polypropylene tubing because these are moving components that require more flexibility than PEEK tubing allows. Solvent caps containing PTFE seals were also substituted with polypropylene caps.

As a further safeguard against contamination, extra care was taken in a variety of ways. A side enclosure was utilized on the EDGE PFAS to prevent air contamination. Also, extracts were collected into polypropylene conical tubes shown to be PFAS-free (below level of quantification LOQ) previously.

### Extractions and Soak Tests

To confirm that the EDGE PFAS was PFAS-free, a series of 12 sequential extractions were done to clean the system. In these extractions, no Q-Cups were used and the EDGE was run according to the EDGE PFAS Method for System Analysis below. The 12th and final extract was used for analysis of the background contamination of PFAS on the EDGE by Pace Analytical.

## EDGE PFAS Method for System Analysis

Q-Disc: None\*

### Cycle 1

Extraction Solvent: Methanol  
Top Add: 20 mL  
Rinse: 0 mL  
Temperature: 65 °C  
Hold Time: 02:00 (mm:ss)

### Wash

Wash Solvent: Methanol  
Wash Volume: 10 mL  
Temperature: 65 °C  
Hold: 00:15 (mm:ss)

Individual samples of PEEK tubing, polypropylene tubing, Q-Disc PFAS, eCleanUP, and Q-Cups were screened for potential contamination via soak tests. For five days, 0.5 g each of PEEK and polypropylene tubing, and six Q-Disc PFAS were soaked in HPLC-grade methanol that was previously shown to be PFAS-free. A 5 g sample of eCleanUP was soaked similarly. The soaking methanol for each sample type was subjected to analysis by Pace Analytical.

The Q-Cups were extracted in the EDGE PFAS without a Q-Disc, using a two cycle method with two washes. The EDGE PFAS Method for Q-Cup Analysis parameters are shown below. The extract was subjected to analysis for its PFAS content by Pace Analytical.

## EDGE PFAS Method for Q-Cup Analysis

Q-Disc: None\*

### Cycle 1

Extraction Solvent: Methanol/Water (80:20) with 0.3% ammonium hydroxide  
Top Add: 10 mL  
Rinse: 0 mL  
Temperature: 65 °C  
Hold Time: 03:00 (mm:ss)

### Cycle 2

Extraction Solvent: Methanol/Water (80:20) with 0.3% ammonium hydroxide  
Top Add: 10 mL  
Rinse: 0 mL  
Temperature: 65 °C  
Hold Time: 04:00 (mm:ss)

*\*We chose not to run a Q-Disc for this study however, in the software, it is required that you choose a Q-Disc. CEM recommends the use of Q-Disc PFAS for PFAS extractions.*

### Wash 1

Wash Solvent: Methanol  
Wash Volume: 10 mL  
Temperature: 50 °C  
Hold: 00:03 (mm:ss)

### Wash 2

Wash Solvent: Methanol/Water (80:20) with 0.3% ammonium hydroxide  
Wash Volume: 10 mL  
Temperature: - - -  
Hold: - :- -

## Results and Discussion

The 12th and final extract obtained from the EDGE PFAS system was analyzed. The list of compounds assessed, their detection limits, and the PFAS content from the extraction of the system and individual soak tests are in **Table 1** (page 3). The data showed that the EDGE PFAS system was free of PFAS molecules to the limit of detection. Furthermore, soak tests of the samples of PEEK and polypropylene tubing, the Q-Disc PFAS, their packaging separators, and eCleanUP indicated that these consumables did not have any background PFAS contamination. The Q-Cups were also shown to have no PFAS contamination. Thus, the EDGE PFAS free from PFAS contamination.

## Conclusion

PFAS testing is extremely important because of the widespread nature of the compounds and their stability. Their use in the manufacture of materials that are commonly used in sample preparation and LC-MS analysis has presented a hurdle for their low level detection. Thus, instrumentation used in the extraction and analysis of PFAS molecules must be free of any prior PFAS contamination. The modifications made to the EDGE were effectively able to eliminate PFAS contamination. The consumables and Q-Cups used by the EDGE PFAS were also found to be PFAS free. The EDGE PFAS is an ideal choice for any laboratory seeking to automate their PFAS extractions without risking PFAS contamination.

**Table 1.** PFAS Compounds Assessed, Detection Limit, and Soak Test Results

Compound	Limit of Detection (ng/L)	EDGE PFAS System	PEEK Tubing	Polypropylene Tubing	Q-Disc PFAS	eCleanUP	Q-Cup
9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF30NS)	25	ND	ND	ND	ND	ND	ND
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF30UdS)	25	ND	ND	ND	ND	ND	ND
1H, 1H, 2H, 2H-perfluorodecane sulfonic acid (8:2 FTS)	50	ND	ND	ND	ND	ND	ND
1H, 1H, 2H, 2H-perfluorooctane sulfonic acid (6:2 FTS)	50	ND	ND	ND	ND	ND	ND
1H,1H,2H,2H-perfluorododecane sulfonic acid (10:2 FTS)	50	ND	ND	ND	ND	ND	ND
1H,1H,2H,2H-perfluorohexane sulfonic acid (4:2 FTS)	50	ND	ND	ND	ND	ND	ND
Hexafluoropropylene oxide dimer acid (GenX)	50	ND	ND	ND	ND	ND	ND
4,8-dioxa-3H-perfluorononanoic acid (ADONA)	50	ND	ND	ND	ND	ND	ND
N-ethylperfluoro-1-octanesulfonamide (EtFOSA)	50	ND	ND	ND	ND	ND	ND
N-ethylperfluoro-1-octanesulfonamidoacetic acid (EtFOSAA)	50	ND	ND	ND	ND	ND	ND
2-N-ethylperfluoro-1-octanesulfonamido-ethanol (EtFOSE)	50	ND	ND	ND	ND	ND	ND
N-methylperfluoro-1-octanesulfonamide (MeFOSA)	100	ND	ND	ND	ND	ND	ND
N-methylperfluoro-1-octanesulfonamidoacetic acid (MeFOSAA)	50	ND	ND	ND	ND	ND	ND
2-N-methylperfluoro-1-octanesulfonamido-ethanol (MeFOSE)	50	ND	ND	ND	ND	ND	ND
Perfluoro-1-butanefulfonic acid (PFBS)	25	ND	ND	ND	ND	ND	ND
Perfluoro-1-decanesulfonic acid (PFDS)	25	ND	ND	ND	ND	ND	ND
Perfluoro-1-heptanesulfonic acid (PFHpS)	25	ND	ND	ND	ND	ND	ND
Perfluoro-1-nonanesulfonic acid (PFNS)	53	ND	ND	ND	ND	ND	ND
Perfluoro-1-octanesulfonamide (PFOSA)	50	ND	ND	ND	ND	ND	ND
Perfluoro-1-pentanesulfonic acid (PFPeS)	25	ND	ND	ND	ND	ND	ND
Perfluorohexanesulfonic acid (PFHxS)	25	ND	ND	ND	ND	ND	ND
Perfluoro-n-butanoic acid (PFBA)	25	ND	ND	ND	ND	ND	ND
Perfluoro-n-decanoic acid (PFDA)	25	ND	ND	ND	ND	ND	ND
Perfluoro-n-dodecanoic acid (PFDoA)	25	ND	ND	ND	ND	ND	ND
Perfluoro-n-heptanoic acid (PFHpA)	25	ND	ND	ND	ND	ND	ND
Perfluoro-n-hexanoic acid (PFHxA)	25	ND	ND	ND	ND	ND	ND
Perfluoro-n-nonanoic acid (PFNA)	25	ND	ND	ND	ND	ND	ND
Perfluoro-n-octanoic acid (PFOA)	25	ND	ND	ND	ND	ND	ND
Perfluoro-n-pentanoic acid (PFPeA)	25	ND	ND	ND	ND	ND	ND
Perfluoro-n-tetradecanoic acid (PFTeDA)	50	ND	ND	ND	ND	ND	ND
Perfluoro-n-tridecanoic acid (PFTrDA)	25	ND	ND	ND	ND	ND	ND
Perfluoro-n-undecanoic acid (PFUdA)	25	ND	ND	ND	ND	ND	ND
Perfluorooctanesulfonic acid (PFOS)	38	ND	ND	ND	ND	ND	ND

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