Application

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# ePrep | Application Note 2024

# Automated Sample Preparation for PFAS in Soils at 0.1-100ppb

with magnetic stirring

Pub No. 98-35040

## SUMMARY

This application note presents a fully automated workflow for PFAS extraction from soil matrices using the ePrep ONE workstation. The validated procedure covers a comprehensive range of PFAS compounds with minimal manual intervention, requiring only a single offline blow-down step. The method achieves impressive analytical sensitivity with LOQ of 0.02 µg/kg and LOR of 0.1 µg/kg, while delivering extract directly to autosampler vials for LC-MS/MS analysis.

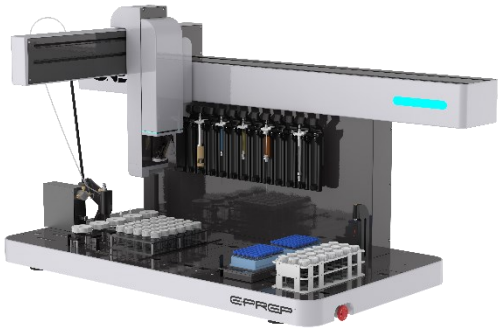


Figure 1 ePrep ONE Sample Preparation Workstation

Method validation demonstrates excellent performance with recoveries of 81-131% and RSDs below 5% for replicate extractions (n=10), confirming reliable quantification from complex soil matrices. The automated workflow accommodates laboratory-specific alterations while maintaining analytical rigor, making it suitable for high-throughput environmental analysis. The combination of automation, precision, and performance establishes a new benchmark for PFAS in soil analysis.

## INTRODUCTION

Perfluoroalkyl substances (PFAS) are synthetic chemicals known as "forever chemicals" due to their extreme persistence in the environment. These fluorine-based compounds are widely used in products like non-stick cookware, waterproof fabrics, and firefighting foams. Their ability to contaminate soil and water resources globally has raised significant environmental and health concerns, as PFAS can be found even in remote areas of the planet.

Traditional methods for analysing PFAS in contaminated soil samples often involve solid phase extraction (SPE) and filtration as part of the clean-up process. However, these techniques can be labour-intensive and time-consuming.

The ePrep ONE is a robotic sample preparation workstation with several unique advantages. One of its key features is the automated syringe exchange technology, which allows for the use of different volume syringes within the same workflow. This flexibility enhances the versatility of

the instrument. Additionally, the system uses Teflon-free syringes, which is crucial in preventing background contamination from Teflon, a potential source of PFAS.

The ePrep ONE also features multi-position magnetic stirring with polypropylene-coated beads to break down soil clumps, along with patented high-velocity mixing technology for improved extraction efficiency.

This application note demonstrates how ePrep ONE automated workstation extracts trace-level PFAS from soil samples, using integrated SPE to streamline sample preparation and reduce costs.

## INSTRUMENTATION

### *LCMS/MS*

| |
|---|
| Instrument: LCMS/MS |
| Column: C18 column, 2.1 mm × 50 mm, 2.7 µm particle size |
| Flowrate: 0.5mL/min |
| Injection vol: 10 µL |
| Column temp: 45°C |
| Mobile phase A: 5 mM ammonium acetate and 0.1% acetic acid in ultra-pure water |
| Mobile phase B: 0.1% acetic acid in 95% Acetonitrile and 5% A phase, Gradient |
| Gradient: 10-minute total runtime |

| Time (min) | A. Conc | B. Conc |
|---|---|---|
| 0 | 89 | 11 |
| 0.75 | 72 | 28 |
| 2 | 62 | 38 |
| 6 | 25 | 75 |
| 6.7 | 1 | 99 |
| 7.1 | 89 | 11 |

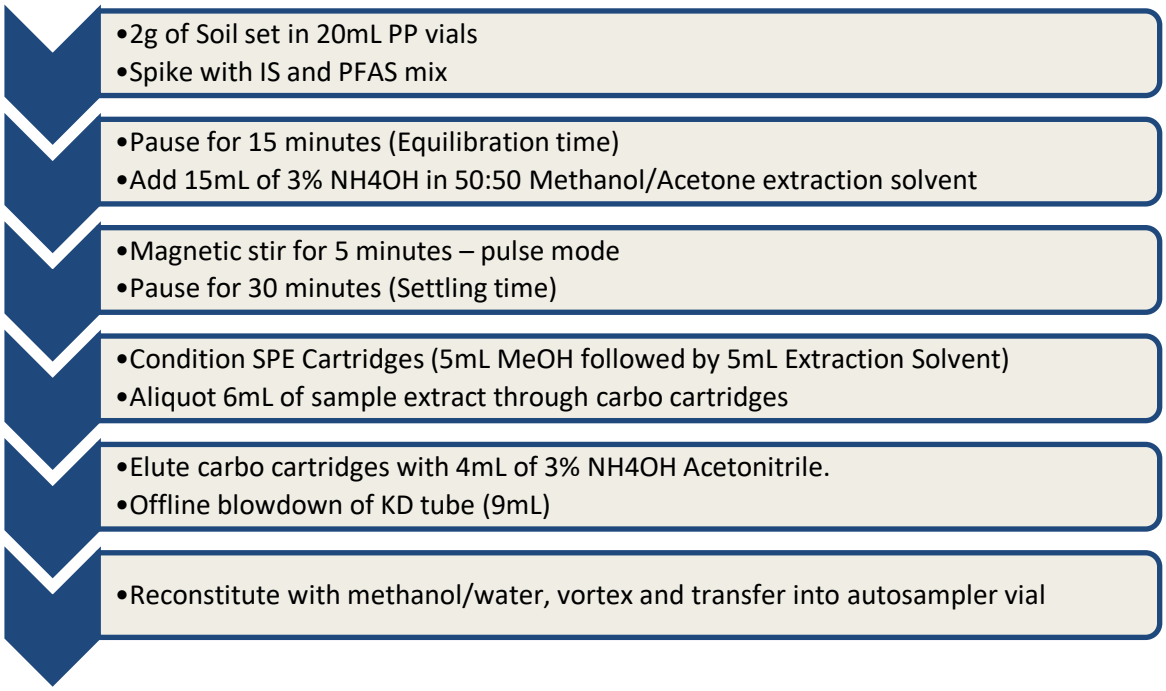
### *MS/MS*

| |
|---|
| Model: triple quadrupole mass spectrometer |
| Polarity: Negative |
| Curtain Gas: 30psi |
| Collision Gas: 9L/min |
| Ion Spray Voltage: -4500V |
| Source Temp: 450°C |
| Ion Source Gas 1: 60psi |
| Ion Source Gas 2: 50psi |

## WORKFLOW

*Automated ePrep Workflow of Sample Preparation*

The following describes the workflow for the preparation and subsequent extraction of Laboratory Control Samples (LCS) using soil. It offers unattended processing other than for the preparation of soil sample 2g into the 20 mL PP (polypropylene) extraction vial and offline blowdown.




Sample spiking process for both the PFAS spike mix and internal standard solution (IS) is optimized for precision. By setting aspiration rates at 30 µL/sec with a 1-second pause, the system minimizes solvent cavitation in the syringe, ensuring quantitative transfer. This level of control is crucial for maintaining the accuracy of spike and standard additions.

A key advantage of the ePrep system is its capacity for 12 syringes or tools, providing significant flexibility in syringe use. The system's ability to dedicate specific syringes to tasks, identified by unique serial numbers and RFID tags, is a notable feature. This dedication of syringes to specific solutions (e.g., internal standard mixture) greatly enhances the integrity of the analytical method by preventing cross-contamination between the PFAS spike solution and the internal standard.

The extraction process is optimized for speed and efficiency. The rapid addition of 15mL of extraction solvent using a probe dispenser, rather than a slower syringe method, saves time. The magnetic stirring approach is particularly efficient, typically requiring only 5 minutes compared to traditional tumbling processes that can take up to an hour.

The incorporation of solid phase extraction (SPE) into the automated workflow is a significant feature. The system transfers 6mL of the sample extract through an SPE cartridge into a 10mL KD tube. The on-deck conditioning of the SPE cartridge with methanol and extraction solvent (3% NH4OH in 50:50 Methanol/Acetone) ensures optimal performance of the extraction process.

The solvent manifold with multiple inlets for different solvents and a waste line facilitates efficient solvent delivery and waste management, particularly useful for conditioning and washing the SPE cartridges. The thorough washing of the syringe with a 50:50 Isopropyl alcohol or methanol: water mixture after each SPE process is a critical step in minimizing background contamination.

The software's validation feature is a valuable tool for ensuring the sufficiency of reagents throughout the workflow. By calculating the required volumes of solutions and comparing them to the set total volume, it prevents potential workflow interruptions due to insufficient reagents.

Overall, this automated system represents a significant advancement in PFAS analysis from soil samples, offering improved precision, reduced contamination risks, and enhanced efficiency compared to traditional manual methods.

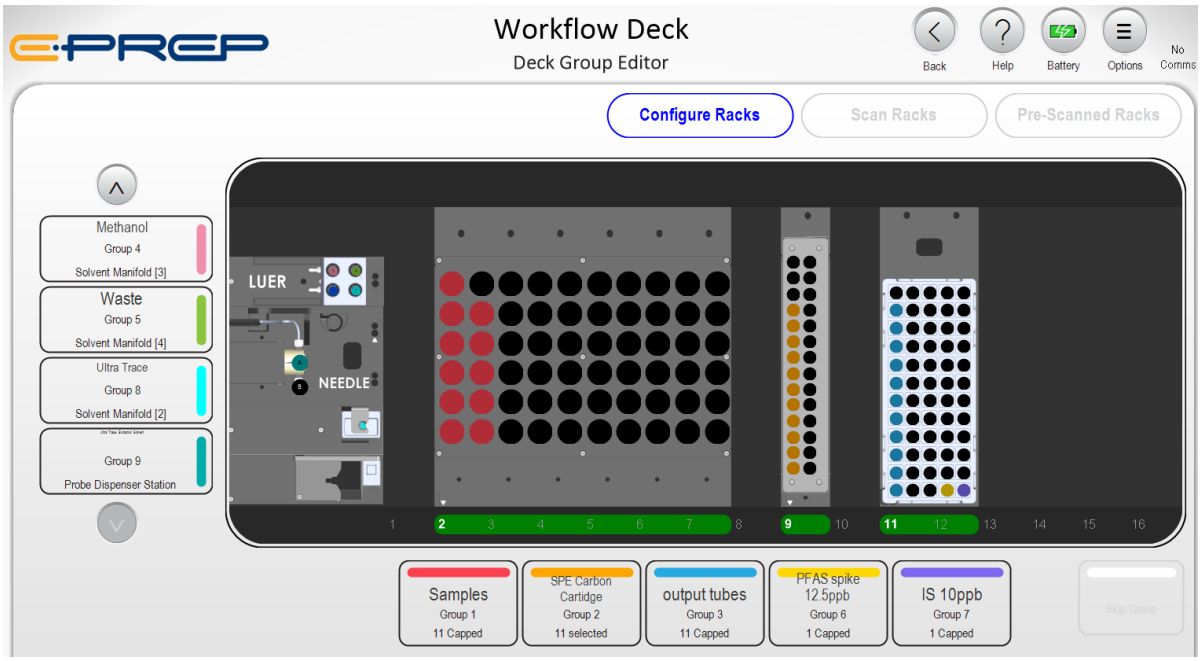



Figure 2 *Image of the ePrep deck (software screen capture).*

## EXPERIMENTAL

Ten Laboratory Control Samples were prepared. 2g of Soil Sample was spiked with mass labelled surrogates at 10 µg/L conc. and PFAS analytes at 12.5 µg/L conc. The percent recovery and RSD for each compound over the 10 samples was then determined and shown in Table 1. A total of 30 PFAS compounds were analysed.

Samples were prepared including method blanks, duplicates, matrix spikes and duplicate matrix spikes. Measurement of uncertainty were evaluated using 7 replicates of clean matrix spiked at low, middle and high range of PFAS at 12.5, 250 and 1000 µg/L. Ruggedness was evaluated by analysing four different types of soils (sandy, clay, loamy and dark with organic matter).

## RESULTS AND DISCUSSION

Table 1 . *Data showing the percentage recovery and percentage RSD of 30 PFAS compounds in Laboratory Control spikes (LCS) at 12.5 µg/L concentration n=10*

| Number | PFAS Compound | %Recovery | %RSD (n=10) |
|---|---|---|---|
| 1 | PFBA | 104.1 | 1.0 |
| 2 | PFPeA | 105.2 | 2.2 |
| 3 | PFHxA | 106.1 | 2.6 |
| 4 | PFHpA | 101.0 | 3.9 |
| 5 | PFOA | 117.2 | 3.8 |
| 6 | PFNA | 111.0 | 2.7 |
| 7 | PFDA | 101.0 | 2.4 |
| 8 | PFUdA | 119.3 | 1.9 |
| 9 | PFDoA | 108.9 | 1.3 |
| 10 | PFTrDA | 131.0 | 2.8 |
| 11 | PFTeDA | 100.2 | 1.7 |
| 12 | PFPrS | 80.6 | 1.6 |
| 13 | PFBS | 87.3 | 1.9 |
| 14 | PFPeS | 86.3 | 4.1 |
| 15 | PFHxS | 101.1 | 2.5 |
| 16 | PFHpS | 96.2 | 1.9 |
| 17 | PFOS | 103.1 | 1.8 |
| 18 | PFNS | 107.2 | 2.2 |
| 19 | PFDS | 105.8 | 2.6 |
| 20 | 4:2 FTS | 99.6 | 4.8 |
| 21 | 6:2 FTS | 100.0 | 1.4 |
| 22 | 8:2 FTS | 104.6 | 3.7 |
| 23 | 10:2 FTS | 102.2 | 2.5 |
| 24 | N-MeFOSAA | 104.4 | 2.6 |
| 25 | N-EtFOSAA | 99.6 | 2.6 |
| 26 | PFOSA | 108.7 | 2.7 |
| 27 | MeFOSA | 108.9 | 2.5 |
| 28 | EtFOSA | 106.8 | 2.8 |
| 29 | N-MeFOSE | 98.2 | 2.6 |
| 30 | N-EtFOSE | 94.3 | 2.0 |

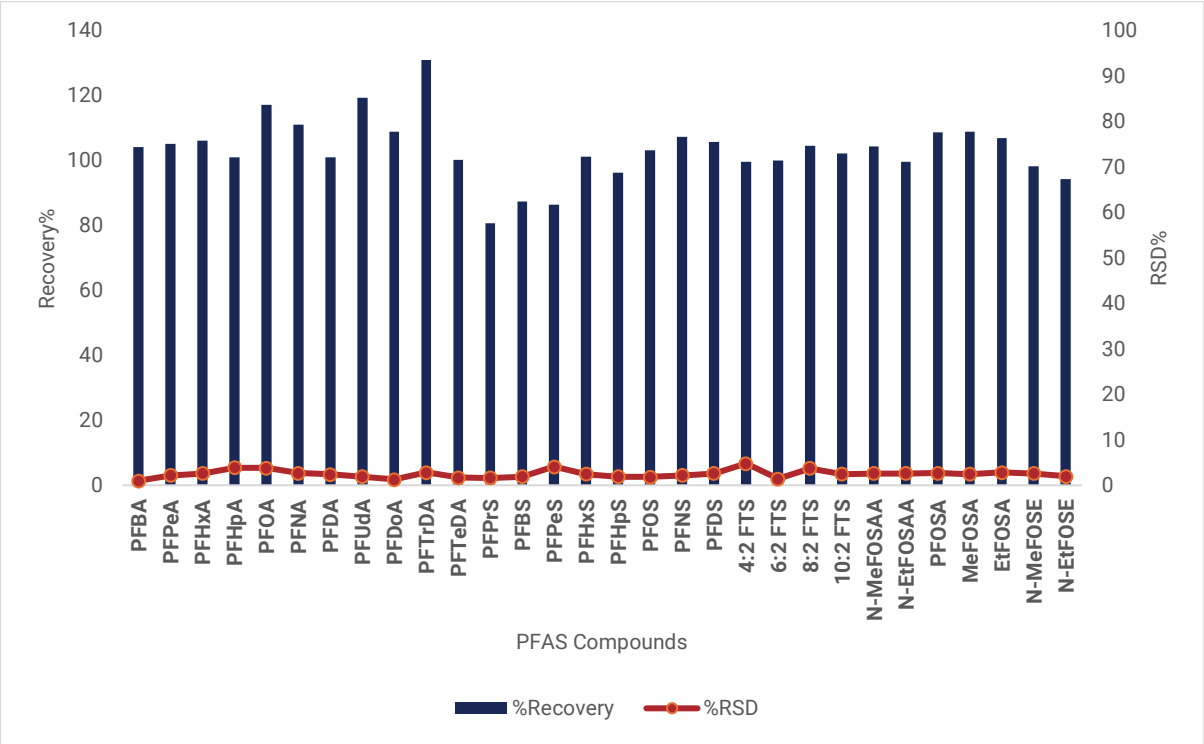



Figure 3 Recovery (%) and RSD (%) for 30 PFAS compounds in LCS spike at 12.5 µg/L n=10

Figure 3 demonstrates the performance of the ePrep ONE workstation for PFAS analysis through Laboratory Control Sample (LCS) studies conducted at 12.5 µg/L concentration (n=10). The data presents two key performance metrics: percent recovery (blue bars) and relative standard deviation (RSD%, orange line) across a comprehensive suite of PFAS compounds.

The recovery values consistently fall within method acceptance criteria (70-130%) for all compound classes, including PFCAs (PFBA through PFDoA), PFSAs (PFBS through PFDS), fluorotelomer sulfonates (4:2-10:2 FTS), and sulfonamides/sulfonamidoacetic acids. Recovery percentages range from approximately 80% to 120%, demonstrating excellent accuracy across the analyte suite.

Method precision, represented by RSD%, shows consistency with values typically below 4% for most compounds, highlighting the reproducibility of the automated sample preparation process. This combination of high recovery and low RSD values validates its capability to deliver reliable and consistent PFAS analysis across multiple compound classes.

Table 2 Data showing the percentage recovery and percentage RSD of 30 PFAS analytes in Laboratory Control samples (LCS) and Soil samples across the three levels (spiked PFAS) - Analysis by LC-MS/MS

| Number | PFAS Compound | %Recovery | %RSD ( n = 29) |
|---|---|---|---|
| 1 | PFBA | 101.3 | 23.1 |
| 2 | PFPeA | 103.9 | 21.6 |
| 3 | PFHxA | 96.7 | 14.6 |
| 4 | PFHpA | 89.1 | 10.8 |
| 5 | PFOA | 98.1 | 12.4 |
| 6 | PFNA | 94.9 | 10.7 |
| 7 | PFDA | 94.0 | 12.7 |
| 8 | PFUdA | 86.1 | 15.2 |
| 9 | PFDoA | 88.6 | 13.6 |
| 10 | PFTrDA | 97.6 | 15.5 |
| 11 | PFTeDA | 91.1 | 14.3 |
| 12 | PFPrS | 81.9 | 12.9 |
| 13 | PFBS | 81.9 | 20.1 |
| 14 | PFPeS | 79.3 | 18.0 |
| 15 | PFHxS | 96.6 | 24.6 |
| 16 | PFHpS | 86.1 | 12.8 |
| 17 | PFOS | 83.8 | 20.8 |
| 18 | PFNS | 83.3 | 29.5 |
| 19 | PFDS | 80.4 | 26.9 |
| 20 | 4:2 FTS | 85.5 | 12.8 |
| 21 | 6:2 FTS | 334.1 | 247.3 |
| 22 | 8:2 FTS | 106.2 | 60.5 |
| 23 | 10:2 FTS | 87.0 | 11.5 |
| 24 | N-MeFOSAA | 92.7 | 12.4 |
| 25 | N-EtFOSAA | 96.3 | 17.6 |
| 26 | PFOSA | 96.2 | 9.8 |
| 27 | MeFOSA | 96.2 | 10.5 |
| 28 | EtFOSA | 97.0 | 9.6 |
| 29 | N-MeFOSE | 96.7 | 12.2 |
| 30 | N-EtFOSE | 89.4 | 10.1 |

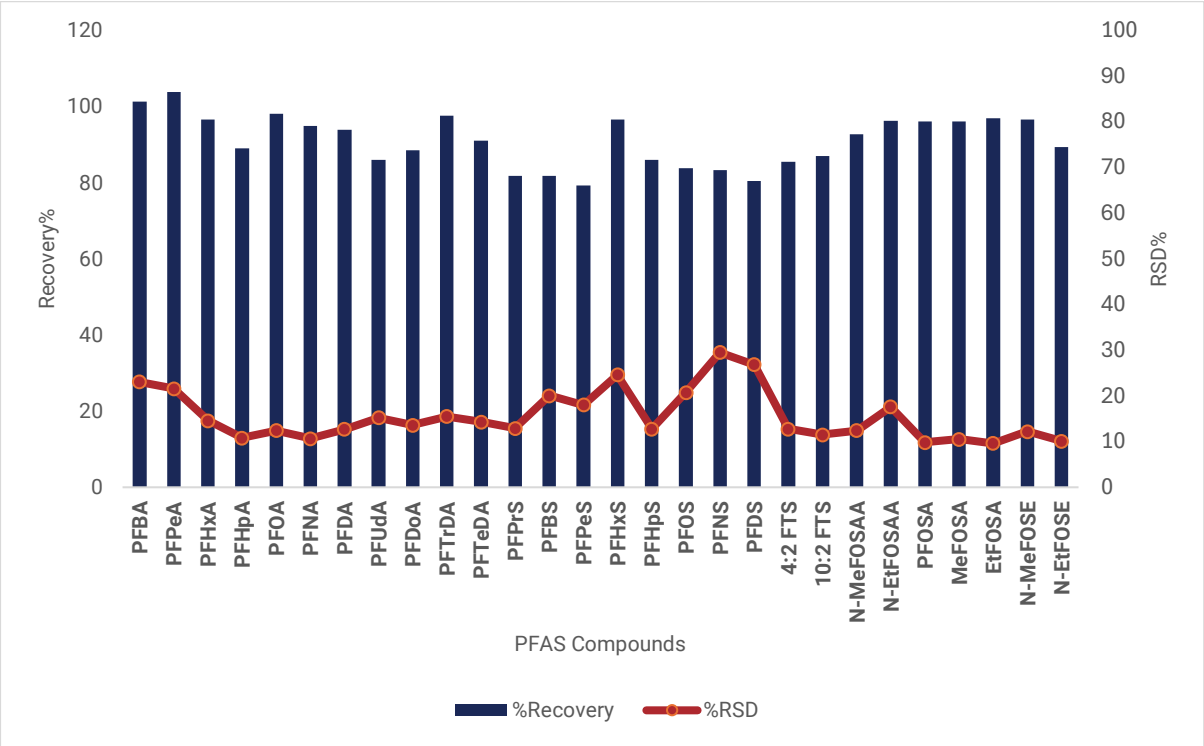



Figure 4 Recovery (%) and RSD (%) for 30 PFAS compounds across three conc. levels in LCS samples.

The figure demonstrates the robust analytical performance of the ePrep ONE workstation across multiple PFAS concentration levels (12.5, 250, and 1000 µg/L, n=10). Recovery values (blue bars) consistently fall within method acceptance criteria (70-130%) across all PFAS classes, from short-chain PFCAs to complex sulfonamides. Method precision, indicated by RSD% (orange line), shows excellent reproducibility with most compounds exhibiting RSDs below 15%.

## CONCLUSION

This application note demonstrates the performance of the ePrep ONE workstation for extraction of PFAS in soil. The described workflow requires minimal manual intervention, with automation handling all steps from extraction through to final transfer into autosampler vials. Method validation shows excellent performance with recoveries of 81-131% and RSD's of 1-4.8% across 10 replicate extractions, demonstrating superior precision despite complex soil matrices.

The method's scalability enables unattended preparation of up to 60 soil samples, significantly enhancing laboratory productivity. with average recoveries of 79.3-106.2% and RSD's below 15% for most compounds, the automated workflow meets rigorous validation criteria while delivering consistent analytical performance.

## APPENDIX 1

Products List for PFAS Soils application:

| Description | ePrep Part Number |
|---|---|
| ePrep One Sample Preparation Workstation | 01-01000 |
| 60 position magnetic stirrer kit | 01-04502 |
| | |
| Magnetic stirrer Adapter Plate for 20-40 mL Enviro Vials | 01-04516 |
| spSPE Rack | 01-04184 |
| Luer Enabling Kit | 01-04160 |
| Solvent manifold | 01-04205 |
| Adapter Plate for Bel-Art 16mm KD Tube | 01-03048 |
| RFID Tag for Bel-Art 16mm Rack | 01-03055 |
| | |
| **Teflon Free Syringes** | |
| 100 µL ePrep (PFAS Free) syringe, Cone 80 mm Removable needle | 01-09046 |
| 1 ml ePrep (PFAS Free) syringe, Cone 80 mm Removable needle | 01-09047 |
| 10 ml ePrep (PFAS Free) syringe, Cone 80 mm Removable needle | 01-09048 |
| | |
| **Consumables** | |
| Bel-Art Rack for 16mm KD Tube or 15mL Falcon Tube {BelArt P/N 18747001} | n/a |
| 20mL PP container (PFAS Free) | 01-03140 |
| Polyimide lined Caps for 20mL PP container (PFAS Free) | 01-18002 |
| Magnetic stirrer beads | 01-04521 |
| KD 10mL tubes, {Thermo P/N LBS1004 (Box 1000)} | n/a |
| Polyimide Lined Caps for KD tube {ALWSCI, PART No. C0003182 (Pkt 100)} | n/a |
| SPE cartridge S*Pure, 220mg carbo {S*Pure P/N 8186557 (100pkt)} | n/a |
| ePrep Needles for SPE cartridge | 01-10996 |
| ePrep Aspiration Needles (Spare) | 01-10995 |