

Application News

No. LCMS-083

Liquid Chromatography Mass Spectrometry

Determination of Selected ASTM Perfluorinated Compounds (PFCs) Using the Shimadzu LCMS-8060



■ Summary

ASTM methods D7968-14 and D7979-15 are used to determine the same list of PFCs but in different matrices. ASTM method D7968-14 lists soil as a matrix while ASTM method 7979-15 lists water, sludge, influent, effluent and waste water as matrix options. These methods use different extraction techniques but both evaluate the samples by liquid chromatography/tandem mass spectrometry (LC/MS/MS). We evaluated the instrumental portion of the methods using the Shimadzu LCMS-8060 triple quadrupole mass spectrometer. Calibration ranges and estimated detection limits were well within method specified values.

■ Background

There has been an increasing awareness of PFCs in soil, sludge and ground water. The need for a quick, easy and robust method to determine these compounds has become apparent.

The LCMS-8060 is used to identify analytes based on their mass, fragmentation, and retention time. Surrogates are present to monitor performance and recovery. The ASTM methods include both perfluorinated and fluorotelomer acids but this study tested the perfluorinated alkyl acids only.

■ Method

In this study, the performance of Shimadzu's most sensitive LC-MS/MS, the LCMS-8060, was evaluated. Since extraction efficiency varies by laboratory, and by analyst, only the instrumental portion of the method was evaluated. In this study, direct injection of fortified reagent water samples diluted with methanol was performed.

Using heated ESI in negative mode, MRM transitions were adjusted using Flow Injection Analysis (FIA) to optimize sensitivity for the PFCs and the surrogate analytes that are listed in Table 1.

Following MRM optimization, chromatography was adjusted for resolution and peak shape by optimizing instrumentation parameters. Near-baseline resolution was accomplished in a 20-minute LC method by using a Phenomenex Kinetex Phenyl-Hexyl 2.6 μ m analytical column coupled with a Shim-pack GIST C18 3 μ m isolator column, as seen in the chromatogram in Figure 1.

Samples were prepared by performing a 70:30 dilution of the water sample using MeOH. The calibration standards were injected at a volume of 30 μ L.

A Shimadzu Nexera UHPLC system was used; operating pressures were about 7000 psi.

■ **Results and Discussion:**

A series of four initial calibration standards across the range of 0.5 to 500 ng/L (parts per trillion, ppt) were prepared. The surrogates were spiked at the same level throughout the curve.

Calibration curves were evaluated using linear regression. Figure 2 shows the calibration curves for the analytes evaluated in this study; corresponding r^2 values are listed.

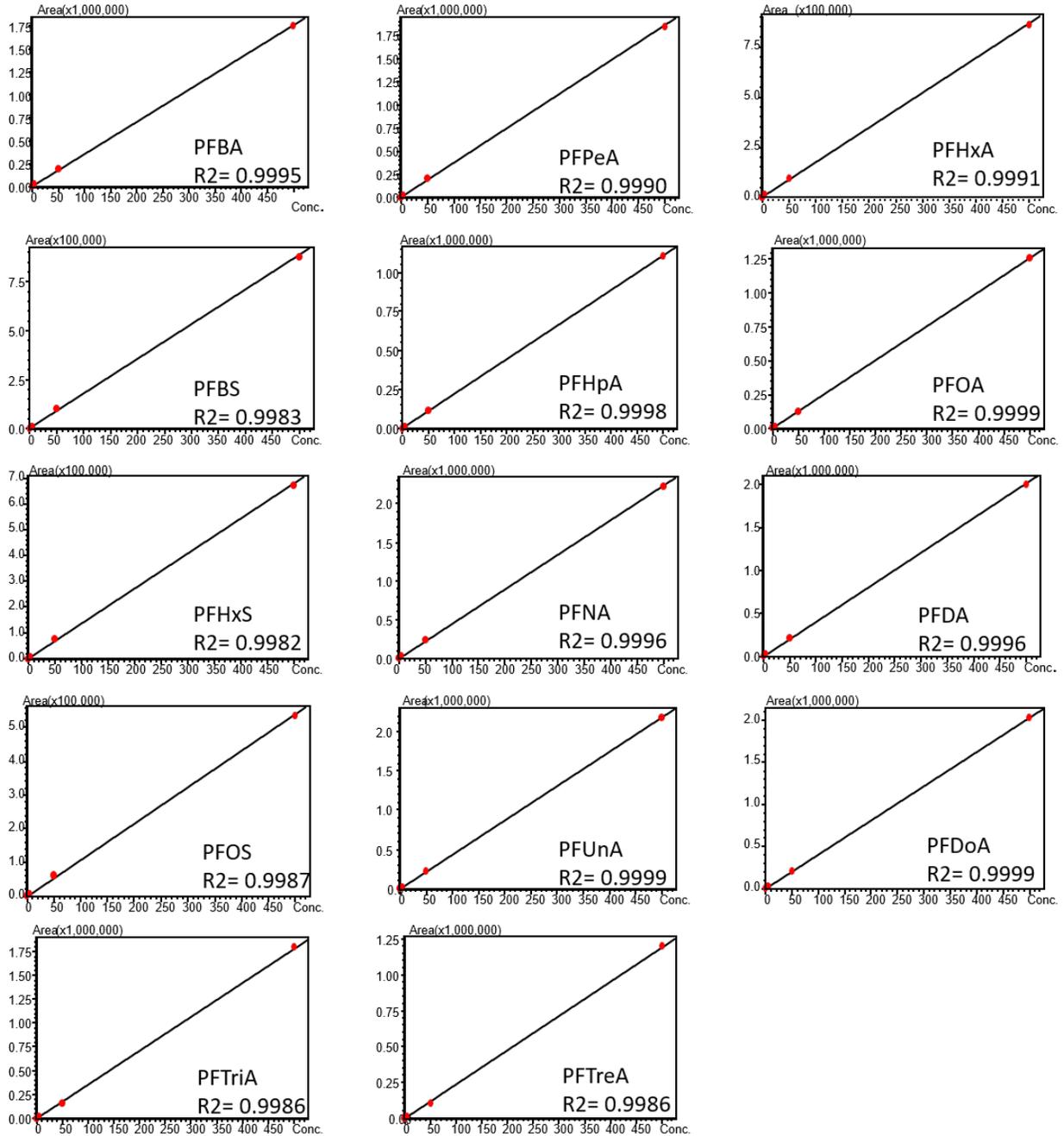


Figure 2: Calibration curves for analytes in the LCMS-8060 method.

Figure 3 shows the chromatogram of a blank water sample evaluated for PFOA and a blank water sample that was spiked with 20 ppt PFOA. The blank sample shows no reportable contamination from the system or method.

The use of an isolator column is important in this method due to potential contamination from ubiquitous plasticizers. The isolator column retards the retention time of PFOA in the mobile phase, and thus eliminates the interference.

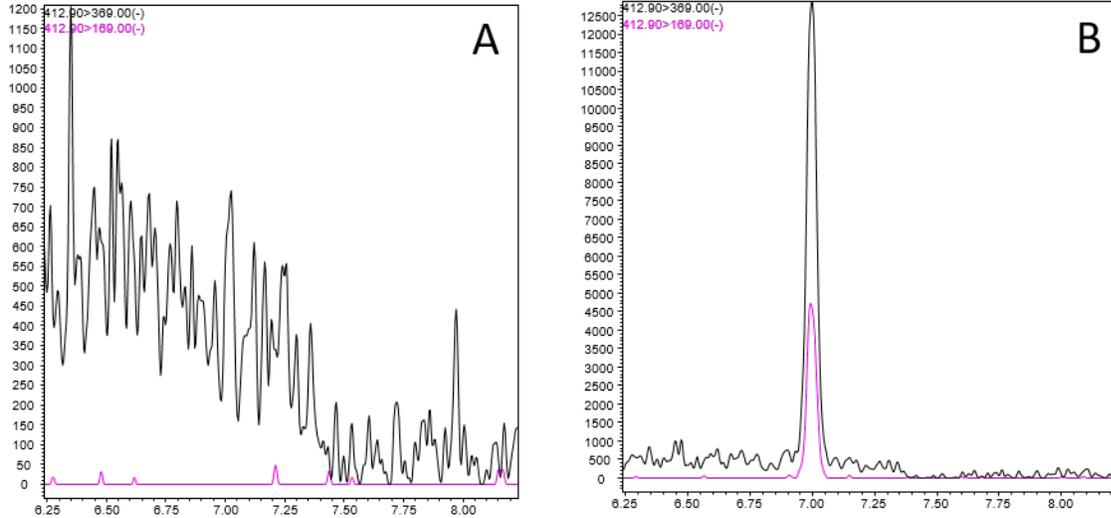


Figure 3: (A) Representative blank chromatogram for PFOA (B) Representative chromatogram of PFOA at 20 ppt.

■ Summary and Conclusions

The Shimadzu LCMS-8060 demonstrates excellent sensitivity for compounds listed in ASTM methods 7968 and 7979. Because of the high sensitivity performance of the LCMS-8060, it is suitable for the analysis of trace levels of PFCs.

UAFMS

ULTRA FAST MASS SPECTROMETRY



LCMS-8040



LCMS-8050



LCMS-8060



LCMS-2020



LCMS-IT-TOF

Founded in 1875, Shimadzu Corporation, a leader in the development of advanced technologies, has a distinguished history of innovation built on the foundation of contributing to society through science and technology. Established in 1975, Shimadzu Scientific Instruments (SSI), the American subsidiary of Shimadzu Corporation, provides a comprehensive range of analytical solutions to laboratories throughout North, Central, and parts of South America. SSI maintains a network of nine regional offices strategically located across the United States, with experienced technical specialists, service and sales engineers situated throughout the country, as well as applications laboratories on both coasts.

For information about Shimadzu Scientific Instruments and to contact your local office, please visit our Web site at www.ssi.shimadzu.com



Shimadzu Corporation
www.shimadzu.com/an/

SHIMADZU SCIENTIFIC INSTRUMENTS, INC.
Applications Laboratory
7102 Riverwood Drive, Columbia, MD 21045
Phone: 800-477-1227 Fax: 410-381-1222
URL <http://www.ssi.shimadzu.com>

For Research Use Only. Not for use in diagnostic procedures. The content of this publication shall not be reproduced, altered or sold for any commercial purpose without the written approval of Shimadzu. The information contained herein is provided to you "as is" without warranty of any kind including without limitation warranties as to its accuracy or completeness. Shimadzu does not assume any responsibility or liability for any damage, whether direct or indirect, relating to the use of this publication. This publication is based upon the information available to Shimadzu on or before the date of publication, and subject to change without notice.

© Shimadzu Scientific Instruments, 2016
First Edition: December, 2016