

POLICY BRIEF

Development of methods for PFAS analyses in articles and chemical products

is needed

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Per- and polyfluoroalkyl substances (PFAS) are a group of more than 10 000 substances that have been used in articles and chemical products for decades. Due to concerns for the environment and human health, PFAS have become subject to regulation in the EU and globally. The newly published report "Analysis of needs for enforcement of PFAS in articles and chemical products" shows a need for development of standardized and validated analytical methods for enforcement and compliance with current and coming PFAS regulations in the EU.

RECOMMENDATIONS

FOR THE EUROPEAN COMMISSION

The European commission should initiate further development of validated and standardised protocols for analysis of PFAS in articles and chemical products to improve enforcement of and compliance with regulations in the EU.

2. FOR THE SCIENTIFIC COMMUNITY AND COMMERCIAL LABORATORIES

The scientific community, including the Partnership for the Assessment of Risks from Chemicals (PARC) and commercial laboratories should work to fill the gaps in analytical methods and matrices mapped in the report and strive towards standardisation and validation of developed methods.







RECOMMENDATION 1

The European commission should initiate further development of validated and standardised protocols for analysis of PFAS in articles and chemical products to improve enforcement of and compliance with regulations in the EU.

Why?

National competent authorities for REACH enforcement face difficulties with enforcement of PFAS restrictions. These include lack of standardised analytical methods for many matrices, making it difficult to compare results from different laboratories. Sufficiently low limits of quantification (LOQs) in relation to set limit values in different matrices was pointed out as another major hurdle. Similarly, companies that want to comply with EU regulations need reliable methods for testing of PFAS in their products. The European Commission (EU COM) has the mandate and possibility to initiate processes to develop and standardise methods for PFAS analysis in relation to current and upcoming PFAS-restrictions.

Background

Effective enforcement of PFAS restrictions relies on accurate and reproducible analytical methods for PFAS. In analytical chemistry, samples are often complex mixtures containing various components, and the matrix is the combination of all these components. The matrix can affect the performance of analytical instruments and methods in several ways, leading to challenges in obtaining reliable and accurate results. When testing PFAS, analytical methods are lacking for many types of articles and chemical products. Experiences from enforcement of current PFAS restrictions were provided by the Nordic Competent Authorities for REACH and important difficulties brought up included:

- 1. Lack of standardised methods to analyse PFAS in articles and chemicals products,
- 2. Different analytical methods used by different analytical laboratories, making it difficult to compare results,
- 3. Lack of methods with sufficiently low limits of quantification (LOQ) to meet regulatory limit values.

Hence, there is a need to further develop analytical methods for PFAS in articles and chemical products to reliably enforce the PFAS restrictions.

VISIONING A WAY FORWARD

EU COM has successfully demonstrated the development of new solutions in the recast of EU Drinking Water Directive (DWD)¹ where PFAS was included as a parameter for surveillance. To enable implementation of the DWD, the DG Environment of the EU COM assigned the IWW (Rheinisch-Westfälisches Institut für Wasser) to develop a new standardized method² for targeted analysis of 20 PFAS in drinking water. In addition, a working group was established under the DWD (article 13(7)) to further develop technical guidelines regarding methods for analysis of 'PFAS Total' and 'Sum of PFAS', in drinking water. These are important parameters for enforcement of PFAS not only in drinking water but in other matrices as well. REACH restrictions on PFAS require similar development of standardized methods and technical guidelines. Therefore, a similar approach should be adopted to ensure efficient enforcement of the current and upcoming regulations.

- Directive (EU) 2020/2184 of the European Parliament and of the Council of 16 December 2020 on the quality of water intended for human consumption (recast)
- 2. DIN CEN/TC 230, entry nr. 14

RECOMMENDATION 2

The scientific community, including PARC, and commercial laboratories should work to fill the gaps in analytical methods and matrices that are currently lacking and strive towards standardisation and validation of developed methods.

Why?

The newly published report highlights that standardised methods are not available for all types of matrices relevant for market surveillance and compliance (TABLE 1). This issue is also raised by the Nordic Competent Authorities for REACH enforcement. Effective enforcement of PFAS restrictions is dependent on accurate and reproducible analytical methods. Thus, the authors state that a commitment to continued research and improvement in the field of PFAS analysis is needed, where collaboration between regulators, laboratories and research institutions is essential to achieve these goals. Academic research institutions and commercial laboratories would make valuable contributions by expanding the types of matrices/samples that could be analysed for PFAS.

Background

Standardised methods are not available for all types of matrices that may be relevant for market surveillance. For many matrices there is an identified lack of commercially available methods. Thus, testing of PFAS for enforcement and market compliance cannot reliably be performed for many types of articles and chemical products with the methods available today.

Such activities could be undertaken e.g. within PARC (Partnership for the Assessment of Risks from Chemicals), which is an EU-wide research and innovation partnership programme with more than 200 partners mostly from academia, research institutions and health authorities. PARC promotes dialogue between scientific and regulatory bodies and one of the main objectives is to provide new data, methods and innovative tools to those responsible for assessing and managing the risks of chemical exposure.

Effective enforcement of PFAS restrictions is dependent on accurate and reproducible analytical methods.

REPORT PROVIDES A TOOLBOX FOR COMPANIES AND AUTHORITIES

The report "Analysis of needs for enforcement of PFAS in articles and chemical products" provides a good overview of the analytical methods available for analyses of PFAS in various types of matrices (TABLE 1). The output of the project, presented in this report, consists of summaries of the various methods available for PFAS analysis, including their applications, commercial availability as well as advantages/disadvantages and limitations. This compilation can be used for identifying available methods for different matrices and assessing the advantages and disadvantages of given methods.

The report describes a variety of analytical methods:

Total fluorine content

Analytical methods that provide information on total fluorine content without the need to identify and quantify each individual PFAS compound separately.

Non-target and suspect screening analysis with HRMS

High-resolution mass spectrometry-based methods that provide high accuracy mass and structural data on both known and unknown PFAS compounds. Information obtained from non-target and suspect screening are very rich in information and allow identification of new PFAS compounds in complex matrices.

Targeted analysis

Targeted methods are very specific approaches that aim to identify and quantify PFAS compounds at the individual level with a high degree of precision and sensitivity.

Structural analysis

Structural methods can be run in parallel to HRMS analysis to confirm the identity of PFAS compounds based on complementary structural tools such as nuclear magnetic resonance spectroscopy and Fourier transform infrared spectroscopy.

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of	avai	ilab	le m	eth	ods

Yes for targeted and total PFAS
= Only for targeted PFAS
× = No

* It is assumed that methods used for other matrices (e.g. consumer products) can also be applied to this matrix.

SECTORS	METHOD FOR COMMERCIAL USE AVAILABLE	SUITABLE METHODS ACCORDING TO LITERATURE
Consumer products	 CEN/TS 15968 (LC-MS/MS)(A), CEN/EN 17681-1 (LC-MS/MS), CEN/EN 17681-2 (GC-MS), ISO 23702-1 (LC-MS/MS), CEN/EN 14582 (CIC) 	 LC-MS/MS, LC-HRMS (NTS), GC-MS, APPI-HRMS, non-target workflow, TOPA, TF, TOF, EOF, AOF, XRF, XPS, TSF
Textiles, leather, carpets	 CEN/TS 15968 (LC-MS/MS) (A), CEN/EN 17681-1 (LC-MS/MS), CEN/EN 17681-2 (GC-MS), ISO 23702-1 (LC-MS/MS), CEN/EN 14582 (CIC) 	 LC-MS/MS, TOPA, THP, TOF, GC-MS, pyr-GC-MS, GC-PARCI-MS, PIGE, XPS, CIC
Food contact material	CEN/TS 15968 (LC-MS/MS)(A), CEN/EN 14582 (CIC)	LC-MS/MS, LC-HRMS, TF, TOF (F-ISE), EOF, GC-MS, PIGE, CIC, XPS, INAA
Cosmetics	×	LC-MS/MS, GC-MS, TF, EOF, TOF, PIGE, CICGC-MS
Ski wax	×	LC-MS/MS, GC-MS, EOF, TOF, WD-XRF
Metal plating	×	● LC-MS/MS, GC-MS
Medical devices & medicinal products	×	● LC-MS/MS, GC-MS
Electric & electronic components	×	LC-MS/MS, LC-HRMS (NTS), GC-MS, TF
Chemical products	×	LC-MS/MS, NMR
Energy sector	×	X *
Transport	×	× *
Firefighting foams	 CEN/TS 15968 (LC-MS/MS)(A), DoD AFFF01 (LC-MS/MS) 	 LC-MS/MS, LC-HRMS, LCxLC-HRMS, LC-IMS-MS, Nano-ESI-HRMS, TD-pyr-DART-MS, EOF, TOPA, photo-TOPA, CIC, SERS
Flame retardants & resins	×	X *
F-gases	×	● GC-MS, Sensors, UV/Vis, IR
Construction products	• CEN/TS 15968 (LC-MS/MS)(A)	LC-MS/MS, GC-MS, DESI-MSI, TOF, TF, IF, TOPA
Lubricants	×	LC-MS/MS, TOF-SIMSv, NMR, GPC
Petroleum & mining	×	LC-HRMS, GC-MS, NTS, TF (HR-CS-MS), CIC
Waste	 ASTM D7979-20 (LC-MS/MS), DIN 38407-42 (LC-MS/MS), DIN 38414-14 (LC-MS/MS), USEPA SW-846 Method 3512 and 8327 (LC-MS/MS) 	LC-MS/MS, LC-HRMS, GC-MS, TOPA, EOF, CIC

TABLE 2: Needs and proposal for an efficient PFAS enforcement regarding analytical methods

ASPECT	NEED	PROPOSAL
Standardized Analytical Methods	Develop and establish standardized analytical methods for PFAs analysis to ensure consistency and comparability of results across laboratories and regulatory agencies.	Collaborate with international standards organizations to create and update standardized methods for PFAs analysis, such as ASTM International and ISO. These methods should cover a wide range of PFAS compounds and matrices.
Method Validation and Certification	Rigorously validate analytical methods to demonstrate their accuracy, precision, sensitivity, and selectivity for various PFAS compounds and matrices.	Regulatory agencies and accredited laboratories should conduct method validation studies, and certified reference materials (CRMs) for PFAS should be developed and made available to laboratories for calibration and quality control.
Accredited Laboratories	Ensure that laboratories conducting PFAS analysis are accredited and follow strict quality assurance/quality control (QA/QC) procedures.	Establish accreditation programs specific to PFAS analysis and regularly assess laboratory performance through proficiency testing programs. Encourage laboratories to participate in interlaboratory studies for method validation and improvement.
Non-Targeted Screening Methods	Develop and refine non-targeted screening methods to identify known and emerging PFAS compounds in complex matrices.	Collaborate with researchers and analytical experts to advance non-targeted screening techniques, such as high-resolution mass spectrometry, and establish data libraries for PFAS compounds.
Method Harmonization	Harmonize analytical methods and reporting criteria among regulatory agencies and regions to facilitate data sharing and comparison.	Collaborate with international organizations and adopt standardized reporting formats and units of measurement for PFAS data. Develop mechanisms for sharing (FAIR – findability, accessibility, interoperability and reusability) data among regulatory agencies and laboratories.
Method Detection and Reporting Limits	Establish method detection limits (MDLs) and reporting limits (RLs) that are appropriate for PFAs analysis in different matrices.	Regulatory agencies should define MDLs and RLs based on method performance data and the specific requirements of PFAS regulations.
Data Quality Assurance	Implement robust data quality assurance practices to ensure the accuracy and reliability of PFAs data.	Develop and enforce QA/QC protocols, including the use of CRMs, blank samples, and internal standards, to monitor and verify data quality throughout the analytical process.
Method Updates and Research	Stay updated on advancements in PFAS analysis and continuously improve analytical methods to address emerging PFAS compounds.	Establish research programs and collaborations to explore new analytical techniques and improve existing methods. Encourage the publication of method updates and improvements.

Concern of PFAS

Per- and polyfluoroalkyl substances (PFAS) are a large group of man-made substances that have been widely used for decades, primarily due to their surface-active properties and resistance to degradation. PFAS are persistent and exposure to certain PFAS has been linked to a number of toxicological effects (e.g., immunotoxicity, reproductive toxicity and carcinogenicity). Hence, PFAS pose a concern for human health and the environment. This has resulted in regulatory actions towards this group of substances. In the EU and globally, groups of PFAS have been restricted and additional restriction proposals are underway.

The current state assessment of PFAS analysis methods

The newly published report describes the regulatory prerequisites for ensuring effective enforcement and compliance verification of restricted PFAS in chemical products and articles. The current state of PFAS analysis methods was assessed, identifying the need for further method development, standardization, and validation of analyses for individual PFAS, precursor substances, and total organic fluorine/total fluorine (including screening methods). This was done by extensive literature reviews and interviews with experts in the field of PFAS analyses as well as with stakeholders from regulatory agencies.

Identified challenges and needs for enforcement and compliance

Challenges to testing of PFAS in articles and chemical products for enforcement and market compliance serve as an obstacle to effective implementation of the restrictions on PFAS in the EU. The following overarching needs to overcome these challenges are proposed:

- Development towards commercially available and standardized analyses that can identify and quantify all regulated PFAS and their derivatives (including polymeric PFAS).
- Further development towards accredited analytical methods for all relevant matrices.
- Achievable LOQs that are lower than the enforcement limit.
- An approach for analysing PFAS at a reasonable cost level, including a potentially easy, low cost and fast screening method (preferable to be performed in the field, e.g. XRF), to be used as indicator for further testing.

To meet these needs, initiatives need to be taken by different actors on EU authority, academic as well as on a commercial level to improve analytical methods and testing of PFAS in articles and chemical products in the EU. This report and its conclusions, highlighting identified needs, could serve as a basis for such activities.

Effective enforcement is key to reduce intentional use of restricted PFAS in articles and chemical products

To analyse PFAS accurately and reliably in different matrices, by regulatory agencies as well as by companies for compliance with legislations, there is a need for reliable analytical methods. With existing and upcoming PFASrestrictions, not the least restriction proposals containing limit values for the total sum of PFAS and total fluorine, there is a need for development and improvement of analytical methods for PFAS analysis.

INFO ABOUT THE NORDIC PROJECT Analysis of needs for enforcement of PFAS in articles and chemical products

The report, initiated by the Nordic Working Group for Chemicals, Environment, and Health (NKE), evaluates the current situation related to PFAS-analyses and enforcement and proposes measures/strategies to enable and/or improve enforcement and compliance with current and future PFAS restrictions. The work in the project and preparation of this report were carried out jointly by Ramboll Deutschland GmbH and the Flemish Institute for technological research (VITO), in contact with a project group consisting of NKE members from Sweden, Norway, Denmark and Finland. The project was funded by Nordic Council of Ministers for Environment and Climate. Analytic methods for PFAS have previously been studied in e.g. the Nordic Council of Ministers reports: "Analytical methods for PFAS in products and the environment" and "Nordic Enforcement project on PFOS and PFOA in chemical products and articles"

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REPORT



Analysis of needs for enforcement of PFAS in articles and chemical products (norden.org)

OTHER LINKS



Analytical Methods for PFAS in Products and the Environment (norden.org)



Nordic enforcement project on PFOS and PFOA in chemical products and articles (norden.org)







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