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ASSESSING PFAS GAS SAMPLING STANDARDS IN OPERATION

Avfall Sverige

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Avfall Sverige

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Förord

PFAS, per- och polyfluorerade alkylsubstanser har tidigare påträffats i flygaska, bottenaska och kondensvatten från svenska förbränningsanläggningar. Idag saknas tillförlitliga mätmetoder för att mäta PFAS-emissioner i rökgaser vilket gör det svårt att ta reda på vilka utsläpp som finns. Tidigare utförda undersökningar inom området ger motstridiga uppfattningar om hur nedbrytningen av PFAS sker under förbränningsprocessen och bättre mätmetoder är en viktig del för att kunna undersöka detta närmare.

Syftet med projektet har varit att utvärdera tillförlitligheten hos tre provtagningsmetoder (OTM-45, modifierad EN 1948:1 och OTM-50) för att karakterisera PFAS-emissioner i rökgaser från svenska avfallsförbrännings-anläggningar under verkliga driftförhållanden.

Projektet har genomförts av Johan Strandberg, Annika Potter, Omar Abdalal, Ioannis Liagkouridis, Thomas Ledbetter, Raed Awad, Jana Moldanova, Sara Jutterström, Emelie Ström och John Andersson från IVL, Stiftelsen för Vatten- och luftvårdsforskning.

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Summary

Per- and polyfluoroalkyl substances (PFAS) are thermally persistent and can form a range of degradation products during waste incineration. Despite this, standardised methods for measuring PFAS in flue gases have only recently been developed. In this study, three PFAS air sampling techniques – OTM-45, a modified EN 1948-1 method, and OTM-50 – were evaluated at four Swedish waste-to-energy plants, including a hazardous waste kiln where aqueous film-forming foam (AFFF) was co-incinerated. The overall aim was to assess how reliably these methods can characterise PFAS emissions under real operating conditions, rather than to establish definitive emission factors for individual facilities.

At all investigated plants, OTM-50 measurements showed that emissions consisted mainly of volatile halogenated compounds, with total concentrations ranging from 10^2 to 10^3 µg/Nm^3. In contrast, concentrations of semi-volatile polar PFAS measured with OTM-45 and EN 1948-1 were generally below 1 ng/Nm^3. The analysed target lists therefore indicate that incineration plants primarily emit volatile fluorinated compounds such as chlorodifluoromethane (HCFC-22) and trichlorofluoromethane (CFC-11), rather than perfluoroalkyl acids. The recurring presence of HCFC-22 and CFC-11 in both flue gas samples and blanks suggests contributions from incomplete destruction of CFC-containing materials, possible in-stack formation via radical reactions, and background concentrations. However, the available data do not allow a detailed quantification of the relative importance of each process.

For polar PFAS, the dataset was dominated by two clearly elevated samples: an OTM-45 sample at Site B with high concentrations of 6:2 fluorotelomer sulfonate (6:2 FTS), and an EN 1948-1 sample at Site C with elevated levels of short-chain perfluoroalkyl carboxylic acids. Both events are likely to represent genuine episodic emissions in a heterogeneous flue gas, although they are partly difficult to interpret due to missing fractions. When these outliers are excluded, total polar PFAS concentrations at all plants converge towards levels below 1 ng/Nm^3, with broadly similar chain-length profiles. This suggests that OTM-45 and EN 1948-1 yield broadly comparable results at these levels, but that the methods respond differently to local emission events and capture partly different PFAS groups.

The method comparison highlights several important limitations. First, ratios between OTM-45 and EN 1948-1 varied by up to two orders of magnitude between plants, and in opposite directions. Combined with the strong influence of individual outliers, this shows that single 2–4 hour grab samples have limited representativeness for PFAS emissions that may vary both in space and time. Second, internal mass balances show that both OTM-45 and EN 1948-1 in practice function primarily as impinger- and surface-based methods: the vast majority of PFAS mass was recovered in NaOH and in the solvent used for equipment rinsing, whereas XAD-2 sorbents contributed very little under the conditions studied.

From a practical perspective, OTM-50 proved relatively straightforward logistically but requires specialised canisters and GC–MS infrastructure. OTM-45 and EN 1948-1 are more complex, requiring extensive handling of glassware and solvents, as well as multi-step laboratory extractions. All three methods are labour-intensive and strongly dependent on careful quality control. Loss of a single canister or a missing fraction can substantially influence data interpretation at low concentration levels.

Taken together, the study shows that PFAS measurement methods can be applied at full-scale waste-to-energy plants and that OTM-45 and EN 1948-1 provide broadly comparable results at sub-ng/Nm^3 levels. At the same time, the data demonstrate that (i) volatile non-polar fluorinated compounds dominate among the compound classes measured here, (ii) all three methods are sensitive to episodic outliers and spatial variability, and (iii) single grab samples are likely to have limited representativeness, for example, in regulatory contexts. Future sampling strategies should therefore combine multi-point or long-duration sampling with target lists that also include volatile polar PFAS classes.

Sammanfattning

Per- och polyfluoralkylämnen (PFAS) är termiskt persistenta och kan bilda en rad nedbrytningsprodukter vid avfallsförbränning. Trots detta har standardiserade metoder för PFAS-mätningar i rökgaser först nyligen utvecklats. I denna studie utvärderas tre PFAS-provtagningstekniker i luft – OTM-45, en modifierad EN 1948:1-metod samt OTM-50 – vid fyra svenska avfallsförbränningsanläggningar, inklusive en farligt avfall-ugn där släckskum (AFFF) samförbrändes. Det övergripande syftet var att bedöma hur tillförlitligt dessa metoder kan karakterisera PFAS-emissioner under verkliga driftförhållanden, snarare än att fastställa definitiva emissionsfaktorer för enskilda anläggningar.

Vid samtliga undersökta anläggningar påvisades att utsläppen enligt OTM-50-metoden huvudsakligen utgörs av flyktiga halogenerade föreningar, med totala koncentrationer inom intervallet 10^2–10^3 µg/Nm^3. Koncentrationerna av semivolatila polära PFAS, uppmätta med OTM-45 och EN 1948:1, var däremot generellt lägre än 1 ng/Nm^3. De analyserade ämneslistorna indikerar således att förbränningsanläggningar primärt emitterar flyktiga fluorerade ämnen såsom klordifluormetan (HCFC-22) och triklorfluormetan (CFC-11), snarare än perfluoralkylsyror. Den återkommande förekomsten av HCFC-22 och CFC-11 i både rökgasprover och blankprover tyder på en kombination av bidrag från otillräcklig destruktion av CFC-innehållande material, möjlig nybildning via radikalreaktioner i skorstenen samt bakgrundskoncentrationer. Befintliga data tillåter dock inte någon detaljerad kvantifiering mellan dessa processer.

För polära PFAS dominerades analysdata av två tydligt förhöjda prover: ett OTM-45-prov vid anläggning B med höga halter av 6:2 fluorotelomersulfonat (6:2 FTS), samt ett EN 1948:1-prov vid anläggning C med förhöjda halter kortkedjiga perfluoralkylkarboxylsyror. Båda händelserna kan sannolikt vara sanna episodiska emissioner i en heterogen rökgas, även om båda är delvis svårtolkade på grund av saknade fraktioner. När dessa outliers exkluderas konvergerar de totala halterna av polära PFAS vid samtliga anläggningar mot nivåer under 1 ng/Nm^3, med grovt sett likartade kedjelängdsprofiler. Detta tyder på att OTM-45 och EN 1948:1 på dessa nivåer i stort sett ger jämförbara resultat, men att metoderna reagerar olika på lokala emissionshändelser och fångar delvis olika PFAS-grupper.

Metodjämförelsen belyser flera viktiga begränsningar. För det första varierade kvoterna mellan OTM-45 och EN 1948:1 med upp till två tiopotenser mellan anläggningarna, och dessutom i motsatta riktningar. I kombination med den starka påverkan från enskilda outliers visar detta att enstaka 2–4 timmars stickprov har begränsad representativitet för PFAS-emissioner som kan vara både rumsligt och tidsmässigt variabla. För det andra visar interna massbalanser att både OTM-45 och EN 1948:1 i praktiken fungerar främst som impinger- och ytbundna metoder: den helt dominerande delen av PFAS-massan återfanns i NaOH- och lösningsmedlet som användes för sköljning av utrustning, medan XAD-2-sorbenter bidrog mycket lite under de studerade förhållandena.

Ur ett praktiskt perspektiv visade sig OTM-50 vara logistiskt relativt enkel men kräver specialkärl och GC–MS-infrastruktur. OTM-45 och EN 1948:1 är mer komplexa, med omfattande hantering av glasvaror, lösningsmedel och flerstegsextraktioner i labbet. Samtliga tre metoder är arbetsintensiva och starkt beroende av noggrann kvalitetskontroll. Bortfall av en enskild kanister eller en förlorad fraktion kan kraftigt påverka tolkningen vid låga koncentrationsnivåer.

Sammantaget visar studien att mätmetoderna för PFAS kan tillämpas vid fullskaliga avfallsförbränningsanläggningar och att OTM-45 och EN 1948:1, ger i huvudsak jämförbara resultat på sub-ng/Nm^3-nivåer. Samtidigt visar data att (i) flyktiga opolära fluorerade föreningar dominerar de av de ämnesgrupper som mättes, (ii) alla tre metoderna är känsliga för episodiska outliers och rumslig variabilitet, och (iii) enstaka stickprov bör ha begränsad representativitet, exempelvis i regulatoriska sammanhang. Framtida provtagningsstrategier bör därför kombinera flerpunkts- eller långtidsprovtagningar med substanslistor som dessutom innehåller flyktiga polära PFAS-klasser.

Innehållsförteckning

1 Introduction

1.1 BACKGROUND

Per- and polyfluoroalkyl substances (PFAS) have been previously detected in fly ash, bottom ash, and condensate water from Swedish incineration plants (Awad et al., 2021). The findings revealed low concentrations in most plants, with a few pronounced outliers. Five plants had no detectable PFAS above the analytical limit of detection. The chemical distribution varied by matrix: bottom ash was dominated by PFCA precursors, fly ash primarily contained PFSA compounds, and condensate water was almost exclusively composed of PFCA species. The data suggested that neither high incineration temperatures nor a high proportion of a particular waste type consistently led to lower PFAS concentrations. These findings indicated a complex relationship between combustion conditions and PFAS fate. However, quantification of PFAS emissions from incineration plants was limited due to the absence of standardised sampling techniques for PFAS in flue gases.

The United States Environmental Protection Agency (USEPA) has developed several methodologies to assess PFAS air emissions, addressing this gap. The Other Test Method 45 (USEPA, 2021, p. 45), introduced in 2021, was designed to measure polar semi-volatile and nonvolatile PFAS compounds in emissions. In contrast, OTM-50 (USEPA, 2023) was developed for non-polar volatile PFAS using whole air canisters. In parallel, researchers at Umeå University adapted the EN 1948:1 dioxin sampling method to include PFAS, demonstrating its feasibility in a Swedish waste-to-energy plant (Björklund, Weidemann and Jansson, 2023). This modified method enabled the first detection of PFAS in flue gas from a full-scale incineration facility, with concentrations of 4.0–5.6 ng/Nm^3, and short-chain perfluorocarboxylic acids (PFCAs) as the dominant species. These advancements significantly shift the ability to monitor PFAS emissions from incineration. However, their practical application in full-scale incineration plants remains limited, as full-scale combustion trials have been limited both in the US and internationally.

A robust understanding of PFAS emissions from incineration is essential due to concerns over incomplete degradation and potential atmospheric transformation products. The current lack of data regarding emission variability across different incineration technologies and operational conditions presents a critical knowledge gap. Additionally, there is limited information on the transformation pathways of PFAS during combustion and the potential formation of secondary fluorinated pollutants. Validating and refining sampling methodologies enhances the quantification of PFAS emissions and supports the development of effective mitigation strategies for PFAS in waste-to-energy facilities. This study addresses these gaps by systematically comparing three PFAS sampling methods across multiple full-scale incineration facilities in Sweden.

1.2 PFAS

Per- and polyfluoroalkyl substances (PFAS) represent a broad class of synthetic chemicals (>4500 known compounds) characterised by a carbon chain where hydrogen atoms are fully or partially replaced by fluorine. According to the OECD definition, a PFAS compound must contain at least one CF_3 group (fully fluorinated methyl group) or one CF_2 group (fully fluorinated methylene group) in its structure (Wang et al., 2021). The strength of the carbon-fluorine bond provides PFAS with exceptional chemical and thermal stability, making them desirable for industrial applications but also highly persistent in the environment. Due to their widespread use and resistance to degradation, PFAS are ubiquitous in environmental matrices, and many compounds bioaccumulate in wildlife and humans.

Several national, regional, and international regulatory measures have been implemented to address PFAS contamination. The Stockholm Convention on Persistent Organic Pollutants (POPs) listed perfluorooctanesulfonic acid (PFOS) in Annexe B in 2009, leading to a near-global phase-out of production and use. Similarly, perfluorooctanoic acid (PFOA) has been classified as a hazardous substance under the EU REACH regulation. In Sweden, significant attention has been devoted to PFAS contamination in drinking water, resulting in the establishment of guideline values and action limits.

Although there are no direct emissions of PFOS and PFOA, for example, from production in Sweden, substantial quantities of PFAS are still released from consumer products, industrial applications, and waste management processes. Among these sources, landfills and incineration plants are recognised as significant contributors to PFAS emissions (Masoner et al., 2020). Thermal treatment of PFAS-containing waste remains uncertain, particularly regarding the extent of decomposition, the potential release of secondary transformation products, and the adequacy of current sampling methods to capture these emissions.

Recent work by Ghasemi et al. (2025) shows that persistent organic pollutants (POPs), such as short-chain chlorinated paraffins, brominated flame retardants, PFOA and PFHxS, are still widespread in Swedish waste streams, particularly in construction and demolition waste, electronic waste, textile and furniture waste, and end-of-life vehicles. Their study highlights that the long service lives of building materials, combined with historical use of regulated substances, imply that legacy chemicals will continue to appear in demolition waste for decades, and that current sorting practices do not always ensure that POP-containing waste is routed to appropriate high-temperature treatment. Although CFCs and HCFCs were not analysed in that work, the same structural challenges in identifying and segregating legacy materials are likely relevant for products containing ozone-depleting substances and fluorinated blowing agents.

1.3 COMBUSTION OF PFAS

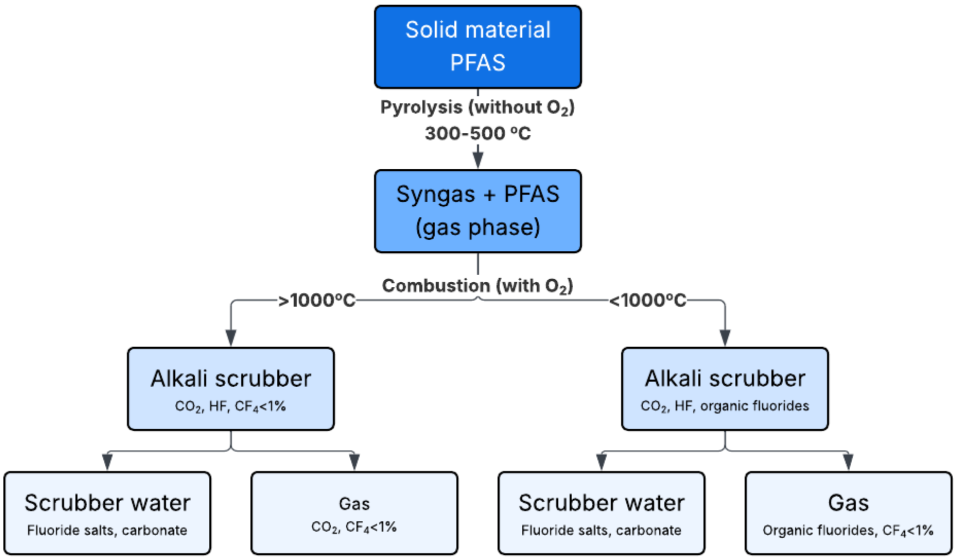
What makes the breakdown of PFAS difficult is the presence of numerous carbon-fluorine (C-F) bonds in the molecule, which require a very high energy to break. Particularly resistant to thermal treatment are fully fluorinated compounds, such as PFCA and PFSA. These lack hydrogen atoms, which further complicates their degradation. In terms of chemical stability, the compounds can be ranked in the following order, with the most stable first: PFSA > PFCA > perfluoroether carboxylic acids. An example of the latter is GenX. The stability of PFCA or PFSA also depends on the length of the carbon chain, with shorter chains such as CF_4 and C_2F_6 being more stable (Weitz et al., 2024). PFAS that contain functional groups which are not fully fluorinated are generally easier to break down, since these groups facilitate the initial degradation. Examples here include fluorotelomers (FTOH), such as 6:2 FTS.

Figure 1 presents a schematic overview of PFAS breakdown through pyrolysis and combustion in solid materials. A key conclusion is that temperature plays a critical role in the effectiveness of PFAS destruction. Decomposition of PFOA and PFOS begins at approximately 350–450 °C, but incomplete combustion can result in the formation of degradation products. Achieving complete mineralisation to carbon dioxide, water, and hydrogen fluoride requires temperatures of at least 1000 °C, at which point no organic fluorine residues can be detected (Longendyke, Katel and Wang, 2022; Zhang et al., 2023). Pilot-scale furnace experiments conducted by Shields et al. (2023) further confirmed that the total quantity of measurable PFAS following AFFF combustion varies as a function of combustion temperature.

Recent full-scale measurements from a Swedish waste-to-energy plant confirmed the presence of PFAS across multiple emission pathways, including flue gas, bottom ash, air pollution control residues, treated process water, and gypsum (Björklund, Weidemann and Jansson, 2023). Short-chain PFCAs (C4–C7) were found to be most abundant across all residual streams, with total annual PFAS releases estimated at 13–47 g depending on waste composition. These findings demonstrate that some PFAS are not fully degraded even at typical incineration temperatures (850–1100 °C), underscoring the importance of validated sampling methods in characterising emissions.

Oxygen-deficient conditions, such as those present during pyrolysis, can limit PFAS degradation and lead to the formation of partially decomposed products, including volatile organic fluorinated compounds (VOFs). However, controlled oxygen supply at high temperatures promotes efficient combustion and reduces the risk of PFAS compounds being converted into other fluorinated substances. Therefore, ensuring sufficient oxygen and maintaining high combustion chamber temperatures are crucial for effective PFAS mineralisation (Longendyke, Katel and Wang, 2022; Zhang et al., 2023).

Figure 1. Schematic overview of PFAS breakdown pathways during pyrolysis and combustion in solid materials, showing temperature zones, dominant reactions and formation or destruction of transformation products. *Adapted from Wang et al. (2024).*



1.4 RESEARCH QUESTIONS

Based on the identified knowledge gaps regarding PFAS combustion behaviour and measurement methodology limitations, this study addresses the following research questions:

1. How reliable and practical are OTM-45, OTM-50, and the modified EN 1948:1 methods for accurately measuring PFAS emissions in flue gases at various incineration plants, considering the influence of sampling surfaces, necessary cleaning steps, and real-world operational challenges?
2. How does the introduction of known PFAS concentrations into the combustion process affect the final concentration, composition, and transformation products of PFAS in emissions?
3. Based on project findings, what recommendations can improve PFAS monitoring and mitigation strategies at both national and international levels?

1.5 OBJECTIVES AND SCOPE

This study was performed with the following objectives:

1. Assessment of the reliability and limitations of OTM-45, OTM-50, and the modified EN 1948:1 methods for characterising PFAS emissions.
2. Conduct PFAS measurements across four furnaces, including the logistics and practical arrangements needed to evaluate usability.
3. Introduce known quantities of PFAS into the combustion process to evaluate method sensitivity and validate the results.

This study primarily evaluates methodological aspects rather than conducting a comprehensive investigation of PFAS fate and transport in incineration systems. The limited number of sampling campaigns and the inherent complexity of full-scale waste incineration systems—including variability in waste composition, combustion conditions, and plant-specific configurations—preclude definitive conclusions regarding the relationships between operational parameters (e.g., combustion temperature, residence time, waste type) and PFAS emission characteristics.

While metadata such as temperature, flow rates, and waste composition were recorded during sampling, the sample size and number of process variables are insufficient to establish statistically robust correlations or mechanistic insights into PFAS transformation pathways. The primary focus remains on evaluating the practical applicability, reliability, and comparative performance of the three sampling methods under real-world conditions at Swedish incineration facilities.

2 Experimental design and sampling locations

2.1 OVERVIEW OF THE TEST CAMPAIGN

This study was conducted as a systematic field evaluation of three PFAS sampling methods across multiple Swedish waste-to-energy facilities from September to December 2024. The test campaign was designed to assess the practical applicability, reliability, and comparative performance of OTM-45, OTM-50, and the modified EN 1948:1 methods under real-world operational conditions.

Full-scale, on-site testing at operational incineration plants is critical for several reasons. First, laboratory-scale experiments cannot replicate the complexity of actual combustion environments, including variable waste composition, dynamic temperature profiles, and the presence of possibly interfering matrices such as high humidity and particulate matter. Second, the logistical challenges of deploying sampling equipment, managing extended sampling durations, and coordinating with plant operations can only be properly evaluated in field settings. Third, understanding method performance under realistic emission concentrations—which are substantially lower than those typically used in laboratory validation studies—is essential for establishing robust detection limits and quantification capabilities.

The sampling campaigns were conducted during normal plant operations to ensure representative conditions were maintained. Each facility was operating under steady-state conditions with typical waste throughput and combustion parameters during the sampling periods.

2.2 SAMPLING LOCATIONS

Flue gas samples were collected at four incineration facilities in Sweden, representing different technologies and operational characteristics. The facilities are designated as Plants A, B, C, and D in this report. Table 1 summarises key operational parameters for each facility.

Table 1. Operational characteristics of the sampled incineration plants, including combustion technology, waste type and key process parameters.

Plant	Technology	Share of household waste	Fuel throughput (tonnes/h)	Operating temperature (°C)
A	Grate furnace	50%	20	1 125
B	Grate furnace	10%	24	1 050
C	Grate furnace	10%	14	850
D	Rotary kiln	0%	7	1 100

Plants A, B, and C represent typical Swedish waste-to-energy operations processing mixed municipal solid waste in grate furnaces at different scales (15–27 tonnes per hour) and temperatures (850–1125 °C). Plant D, a specialised rotary kiln for hazardous waste with no household waste fraction, enabled controlled spiking experiments with AFFF firefighting foam.

Furnaces A and B were sampled at stack locations after the flue gas treatment systems, measuring emissions released into the atmosphere, while C and D were sampled before the scrubber. Sampling points were accessed via permanent stack testing ports in accordance with isokinetic sampling requirements for representative characterisation of the flue gas.

2.3 SAMPLING STRATEGY

At sites A, B, and C, OTM-45 and modified EN 1948:1 sampling trains were deployed to enable direct comparison under identical combustion conditions. OTM-50 canister sampling was conducted during the same operational period. At site D (the hazardous waste line), only OTM-45 and OTM-50 were deployed due to spatial constraints. The modified EN 1948:1 method was not used at site D. Table 2 summarises the samples collected per method and site.

Table 2. Overview of flue gas samples collected per site and method, indicating the number of OTM-45, EN 1948:1 and OTM-50 trains at each facility.

Site	OTM-50	OTM-45	EN 1948:1	Notes
A	2	1	1	OTM-50: A-1 excluded due to pressure gauge failure
B	2	1	1	Complete method comparison
C	2	1	1	Complete method comparison
D	1	1		Hazardous waste line with AFFF spiking
Total valid samples	**7**	**4**	**3**	
Ambient air samples	3	1		Site A, B and C (OTM 50) Site A (OTM 45)
Sampling train field blanks (STFB)(1)		3		Sites B, C and D
Laboratory blanks	2			Quality control

(1) Sampling train field blanks (STFB) consist of samples from the complete sampling train at the site, to assess potential background contamination and equipment artefacts. These do not represent stack emissions.

The sampling durations ranged from approximately 2 to 4 hours per sample. Key sampling parameters for OTM-45 included:

- Plant A: Sampling time 12:10–16:12 (4.03 hours), Stack temperature 80 °C
- Plant B: Sampling time 12:45–15:45 (3.00 hours), Stack temperature 60 °C
- Plant C: Sampling time 09:29–11:35 (2.10 hours), Stack temperature ~160 °C
- Plant D: Sampling time 16:32–20:39 (4.12 hours), Stack temperature ~143 °C

For OTM-45, sampling was conducted using extractive sampling with isokinetic flow control to match stack gas velocity and ensure representative sampling. Flow rates were adjusted at each site in accordance with the modified EN 1948:1 to match OTM-45. OTM-50 canister sampling employed critical-orifice flow control at approximately 83 mL/min, as specified in the method protocol, with sampling timing coordinated with the sorbent-based methods.

One OTM-50 canister sample from Plant A (designated A-1) was excluded from quantitative analysis due to a pressure gauge malfunction. Post-sampling pressure measurements did not show the expected increase from initial vacuum to near-atmospheric pressure, indicating either a leak or a flow restriction that compromised sample integrity. The remaining duplicate samples from Plant A (A-2, A-3) provided sufficient data for method evaluation at this site.

Field blanks and sampling train field blanks (STFB) were collected during sampling campaigns to assess potential contamination from sampling equipment, reagents, ambient air, and handling procedures. Procedural blanks, consisting of pre-cleaned glassware and sorbent media, were taken through the whole preparation and recovery protocol and analysed alongside field samples to evaluate background contamination levels.

To ensure that PFAS was actually included in the fuel at incineration plants, aqueous film-forming foam (AFFF) containing PFAS was introduced into the combustion process at Site D (the hazardous waste rotary kiln). One cubic meter of AFFF foam was continuously injected into the waste feed stream over 24 hours. The injection rate was limited to avoid increasing HF levels in the flue gases, as higher HF concentrations, formed when PFAS combusts, cause excessive wear on plant equipment and lead to increased maintenance. This spiking approach also enabled the identification of potential transformation products formed during combustion.

At the time of this study, the project absorbed a substantial fraction of Sweden's available capacity for specialised passivated canisters suitable for PFAS analysis, as these canisters cannot be readily substituted with standard air sampling canisters due to the requirement for silicon-ceramic passivation to prevent PFAS adsorption.

2.4 ANALYTES

The three sampling methods target different classes of PFAS compounds based on their volatility and polarity, providing complementary coverage of the PFAS spectrum that may be present in combustion emissions.

2.4.1 Volatile non-polar substances

OTM-50 was explicitly designed for volatile, fluorinated, non-polar compounds with high vapour pressures that are not effectively captured by traditional sorbent-based methods. The OTM-50 target list includes a range of volatile fluorinated compounds, such as CFCs, HFCs, PFCs, and fluoroethers. Many, but not all, of these meet recent OECD definitions of PFAS. The method utilises passivated stainless-steel canisters to collect whole air samples, which are then analysed by gas chromatography-mass spectrometry (GC-MS).

A total of 30 volatile non-polar PFAS compounds and other fluorinated substances were targeted according to the OTM-50 method protocol, including:

- Perfluorocarbons (PFCs): tetrafluoromethane (CF_4), hexafluoroethane, perfluoropropane, perfluorobutane, perfluoropentane, perfluorohexane, perfluoroheptane, perfluorooctane
- Hydrofluorocarbons (HFCs): fluoromethane (HFC-41), difluoromethane (HFC-32), fluoroform (HFC-23), pentafluoroethane (HFC-125), 1,1,1,2-tetrafluoroethane (HFC-134a), 1,1,1-trifluoroethane (HFC-143a), and various partially fluorinated hydrocarbons (1H-heptafluoropropane, 1H-nonafluorobutane, 1H-perfluoropentane through 1H-perfluorooctane)
- Chlorofluorocarbons (CFCs): trichlorofluoromethane (CFC-11), chlorotrifluoromethane (CFC-13), chlorodifluoromethane (HCFC-22)
- Fluorinated olefins and cyclic compounds: hexafluoropropylene, tetrafluoroethylene, octafluorocyclobutane (PFC-318), octafluorocyclopentene
- Fluoroethers: hexafluoropropene oxide (HFPO)

The complete target list is provided in Table OTM-50-1 of the OTM-50 method document (USEPA, 2023). These compounds represent potential products of incomplete PFAS combustion as well as industrial refrigerants and process chemicals that may be present in waste streams. Canister samples were analysed using GC-MS with cryogenic preconcentration at IVL. Detailed analytical procedures are described in Section 3.3.2.

2.4.2 Semi-volatile polar substances

Both the OTM-45 and the modified EN 1948:1 method target semi-volatile and polar PFAS, employing sorbent-liquid-based sampling trains to capture compounds across multiple phases (vapour, particle-bound, and water-soluble). A total of 54 polar PFAS compounds were included in the analytical panel, representing:

- Perfluoroalkyl carboxylic acids (PFCAs): C4-C14 chain lengths (PFBA, PFPeA, PFHxA, PFHpA, PFOA, PFNA, PFDA, PFUnDA, PFDoDA, PFTrDA, PFTeDA)
- Perfluoroalkyl sulfonic acids (PFSAs): C4-C12 chain lengths (PFBS, PFPeS, PFHxS, PFHpS, PFOS, PFNS, PFDS, PFDoDS)
- Fluorotelomer sulfonates (FTS): 4:2, 6:2, 8:2, 10:2 FTS
- Perfluorinated sulfonamides (FOSAs): FOSA, MeFOSA, EtFOSA, and related compounds
- Perfluorinated sulfonamide ethanols (FOSEs): MeFOSE, EtFOSE
- Perfluorinated sulfonamidoacetic acids (FOSAAs): MeFOSAA, EtFOSAA
- Fluorotelomer unsaturated acids (FTUAs)
- Ether-based PFAS: including GenX (HFPO-DA) and other replacement chemicals
- Chlorinated ether sulfonic acids
- Fluorinated replacement chemicals

The complete list of target analytes, along with their CAS numbers and isotope-labelled internal standards, is provided in the supplementary materials. Detailed sample preparation and analytical procedures are described in Sections 3.1.2 and 3.2.2

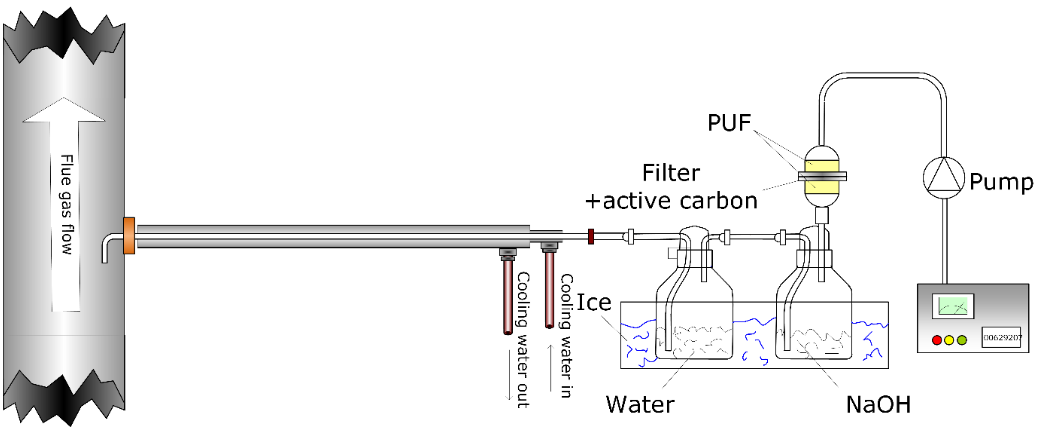
3 Methods

All methods were deployed at operational waste-to-energy facilities under normal operating conditions.

3.1 MODIFIED EN 1948:1

The sampling train was constructed around a cooled probe with a glass liner that fed gas samples through a cascade of glass impingers and a PUF holder with an integrated aerosol filter, as shown in Figure 2. Scott bottles containing absorption solutions (0.1 M NaOH and MilliQ water) were employed to capture soluble PFAS. The design is aimed at minimising thermal degradation and ensuring that particulate and gaseous PFAS fractions are effectively collected.

Figure 2. Schematic layout of the modified EN 1948:1 sampling train used for semi-volatile polar PFAS in flue gas.



3.1.1 Sampling

The pump flow was calibrated and adjusted to match the flue gas velocity of the OTM-45 sampler (typically 15–20 L/min). The cooled probe, featuring a glass liner, maintained low temperatures during sample collection to prevent the loss or degradation of thermally labile analytes. Two glass impingers were arranged in series downstream of the probe to trap PFAS in the condensating gas. In addition, a PUF holder equipped with an integrated aerosol filter was used to capture particulate-bound and less hydrophilic compounds.

Prior to each sampling campaign, an internal standard spike (3 ng M8PFOS in approximately 5 mL HPLC-grade methanol) was added to the water impinger to assess analyte recovery throughout the sampling and analytical process. The sampling system was mounted on a dedicated stand, which contributed to the mechanical stability of the train and reduced the risk of leaks or disturbances during field deployment.

At the time of sampling, ambient conditions (barometric pressure and temperature) and stack parameters (O_2, CO_2, and H_2O content, as well as temperature) were recorded through the facility's continuous emission monitoring system. Sample volumes were normalised to standard conditions (0°C, 1 atm, dry gas) using the measured parameters.

3.1.2 Analysis

Sample fractions collected via the modified EN 1948:1 method were processed separately according to their matrix type.

The water and NaOH impinger fractions were filtered through glass fibre filters (GF/A, Whatman, 1.6 μm) and then through nylon filters (0.45 μm) to remove particulate matter. Filters were retained for separate extraction if warranted. Following filtration, pH was adjusted to 3 using acetic acid (MilliQ fraction) or hydrochloric acid (NaOH fraction). Samples were subjected to solid-phase extraction (SPE) using Oasis WAX cartridges (6 cc, 150 mg, 30 μm). SPE cartridges were conditioned with 4 mL of 0.1% ammonium hydroxide in methanol, 4 mL of methanol, and 4 mL of MilliQ water. Samples were loaded at a flow rate of no more than 1 drop per second, followed by washing with 4 mL MilliQ water and 4 mL 25 mM ammonium acetate (pH 4). After drying under vacuum for 30 minutes, the analytes were eluted into two fractions: neutral PFAS (4 mL of methanol) and anionic PFAS (4 mL of 0.1% ammonium hydroxide in methanol).

The PUF adsorbents and activated carbon filter were extracted together by Soxhlet extraction using HPLC-grade methanol. Internal standards were added to the extraction solvent prior to extraction. Samples were extracted for a total of 16 hours (8 hours, followed by fresh solvent, and an additional 8 hours) to ensure complete recovery. Following extraction, samples were concentrated by rotary evaporation to approximately 5 mL, diluted to a maximum of 10% methanol with ultrapure water, and adjusted to a pH of 3–4 with acetic acid. The diluted extracts underwent SPE cleanup using the same Oasis WAX protocol as the aqueous fractions, with separate elution of neutral and anionic PFAS fractions.

All extracts were concentrated under nitrogen gas to 150–200 μL, transferred to LC vials with methanol to a final volume of 500 μL, and prepared for analysis. For the neutral PFAS fraction, 160 μL of extract was combined with 40 μL of 2 mM ammonium acetate. For the anionic PFAS fraction, 80 μL of extract was combined with 120 μL of 2 mM ammonium acetate. Samples were analysed by liquid chromatography-tandem mass spectrometry (LC-MS/MS, Shimadzu LCMS-8060NX) operating in multiple reaction monitoring (MRM) mode with electrospray ionisation in negative mode. Isotope dilution calibration was employed for quantification, with isotopically labelled internal standards added to each sample fraction prior to extraction to monitor recovery and correct for matrix effects.

Quality assurance protocols included analysis of field blanks, procedural blanks, and duplicate extractions to assess method performance, quantify background contamination levels, and evaluate analytical precision.

3.2 OTM-45

The OTM-45 sampling train featured a heated quartz-glass probe to withstand elevated temperatures. The system integrated a filter assembly (utilising glass fibre filters) to capture particulate matter, a primary XAD-2 adsorbent module for gaseous-phase PFAS, a series of impingers to enhance capture efficiency, and a secondary XAD-2 module intended to detect breakthrough of analytes, as shown in Figure 3.

Figure 3. Schematic layout of the OTM-45 sampling train, showing the heated probe, filter, impingers and primary/backup XAD-2 sorbent modules from USEPA (2021).

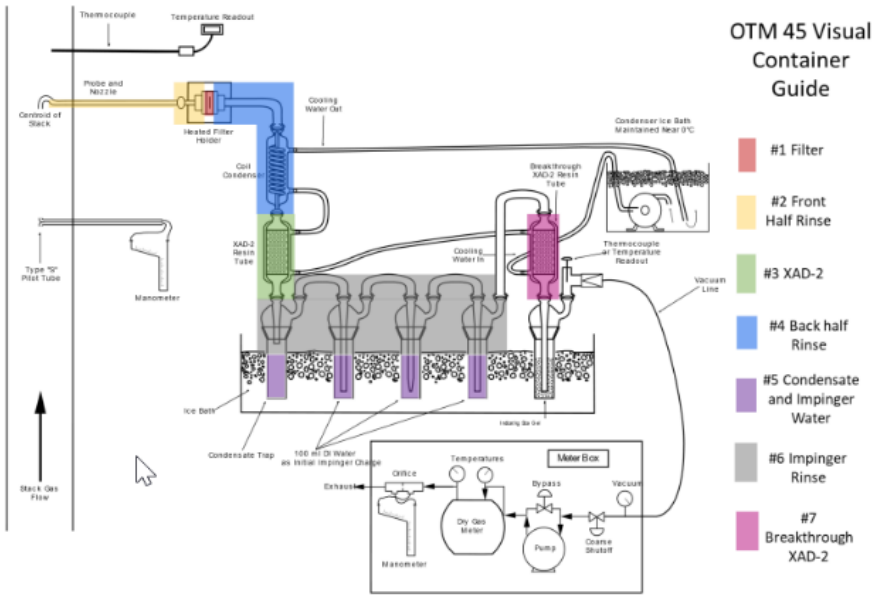


Figure OTM-45-1. Sampling Train

The Belgian reference method LUC/VI/003 (Vito, 2023) builds on EPA OTM-45 but introduces additional sampling train variants to address moisture-related issues. In the original OTM-45 configuration, the condensate formed in the train passes through the primary XAD-2 cartridge, which can cause the adsorbent to become heavily wetted in stacks with high moisture content and may affect both capture efficiency and the required underpressure of the sampling train. To mitigate this, LUC/VI/003 defines two alternative sampling trains in which the condensate is collected in a separate flask before the sampled gas is directed through the XAD-2 cartridge (an "OTM-45 variant" and a "cooled probe" configuration). The equivalence of these variants to the original OTM-45 train has been demonstrated for selected C4–C8 PFAS in simultaneous stack measurements and further evaluated as part of the Belgian reference method development and interlaboratory comparison exercises (Vito, 2023; Hofman, Baeyens, et al., 2025; Hofman, Jacobs, et al., 2025).

3.2.1 Sampling

Sampling was performed under nominally isokinetic conditions with flow rates adjusted to match stack gas velocity and ensure representative sampling. Protocols, including leak checks after assembly and validation of isokinetic conditions during the run, were followed throughout the sampling procedure. Field blanks were also collected to assess background contamination and to validate the data.

3.2.2 Analysis

Samples collected using OTM-45 were processed using tailored extraction procedures, following the general guidelines outlined in OTM-45 Revision 0 (January 2021). Before extraction, samples were spiked with mass-labelled internal standards (IS) for selected PFAS compounds to enable isotope-dilution quantification and to assess recovery throughout the analytical process.

Filter and XAD-2 adsorbent samples were extracted separately. Glass fibre filters were extracted overnight (minimum 18 hours) with methanolic ammonium hydroxide (5% v/v) according to the OTM-45 protocol. XAD-2 adsorbent samples were extracted overnight with methanolic ammonium hydroxide (5% v/v). For the XAD-2 samples, the extraction procedure was repeated with fresh solvent to ensure complete recovery of the target analytes.

Impinger water samples were subjected to solid-phase extraction (SPE) using weak anion-exchange (WAX) sorbent cartridges. Following the OTM-45 protocol, extracts were cleaned, concentrated, and prepared for instrumental analysis. The use of dichloromethane (DCM) for cleaning XAD-2 sorbents, as specified in the original OTM-45 method, was omitted due to health and environmental concerns. Laboratory validation studies at IVL demonstrated that thorough rinsing procedures provided equivalent performance without the use of this hazardous solvent.

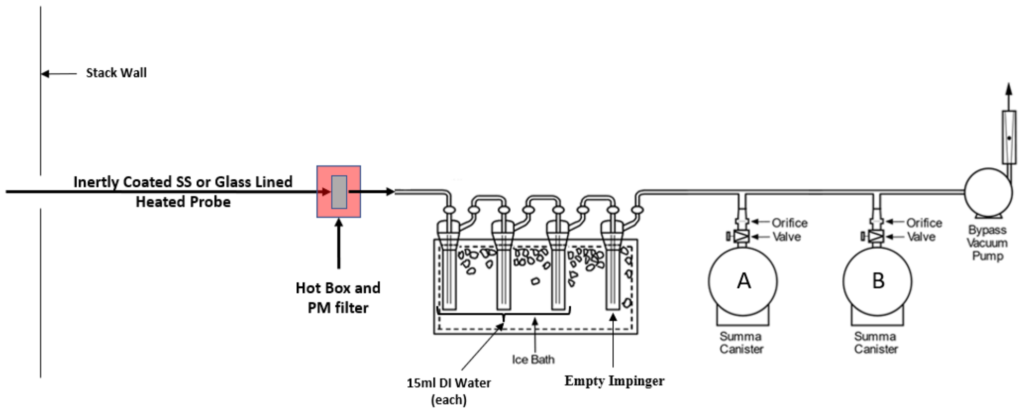
The cleaned extracts were subsequently concentrated and analysed for 54 target PFAS using ultra-high-performance liquid chromatography-tandem mass spectrometry (UHPLC–MS/MS, Shimadzu LCMS-8060NX) operating in multiple reaction monitoring (MRM) mode. Isotope dilution techniques were employed to enhance quantification accuracy and correct for matrix effects.

Rigorous quality assurance protocols were implemented, including the use of laboratory-fortified media blanks (LFMB) and the routine analysis of field and laboratory blanks and duplicate samples. Periodic use of continuing calibration check (CCC) standards provided ongoing verification of instrument performance and analytical consistency throughout the sample analysis sequence. Typical method quantification limits (MQLs) for individual polar PFAS ranged from 0.015 to 0.1 ng per sample, corresponding to approximately 0.01–0.05 ng/Nm^3 for the sampling volumes used in this study.

3.3 OTM-50

The OTM-50 sampling train was explicitly designed to measure volatile, non-polar fluorinated compounds. It consisted of a heated sample probe, stainless-steel tubing leading to a critical orifice, and 3-L SiloniteTM-coated passivated stainless-steel canisters, as shown in Figure 4. Integrated water and acid gas management components, including heated filters and conditioning systems, were incorporated to protect volatile analytes from moisture and corrosive interference during sampling.

Figure 4. Design of the OTM-50 sampling train, including impingers for humid gases and multiple evacuated canisters for non-polar volatile PFAS collection.



3.3.1 Sampling

Sampling for OTM-50 was conducted at stack locations using passivated canisters to collect whole-air samples. A stainless-steel tube, mounted parallel to the OTM-45 sampling probe, directed humid gases through impingers for drying, then through a critical orifice to control the flow rate, ensuring a consistent gas volume entered the silicon-ceramic-lined, passivated stainless steel canisters. The critical orifice precisely regulated the gas flow (approximately 38 mL/min), thereby maintaining sample integrity and minimising the risk of analyte degradation.

Detailed pre-sampling calibrations and system leak tests were performed to safeguard sample quality. Post-sampling quality checks included verification of final canister pressure to confirm adequate sample volume collection and identify potential leaks or flow restrictions. These field-level quality control measures proved essential, as demonstrated by the successful identification of one compromised sample (Site A, sample A-1) through pressure verification before laboratory analysis.

3.3.2 Analysis

All OTM-50 analytical work was performed at IVL. Following quality verification, sample aliquots were extracted from canisters, dried and pre-concentrated using a cryogenic focusing trap to enable detection at sub-ppbv concentrations. This preconcentration step is critical for achieving the method detection limits required for ambient and low-concentration industrial source measurements. The CO_2 interference was handled on the focusing trap by purging the sample at an elevated temperature.

Analysis was performed using gas chromatography-mass spectrometry (GC-MS) in both full-scan and selected-ion monitoring (SIM) modes to facilitate the identification and quantification of volatile fluorinated compounds.

During analysis of the last two sites (C and D), the GC-MS system was calibrated with a traceable, certified calibration gas, ensuring accurate and reproducible quantification across the concentration ranges encountered in this study. Since the certified calibration gas could not be retrieved until after the analysis of the first two sites (A and B), a surrogate calibration standard containing volatile organic compounds as ozone precursors was used. A number of surrogate components were calibrated at each analysis occasion, and the responses of these surrogate components, along with the internal standard injections, were later used to calculate concentrations of PFAS at sites A and B after retrieving the PFAS calibration gas.

Quality assurance protocols included the analysis of field blanks, sampling train field blanks (STFB), laboratory blanks, and duplicate samples, as well as duplicate analysis of at least one sample in each round. Regular verification of instrument performance was conducted using continuing calibration verification (CCV) standards, which were analysed at specified intervals throughout the analytical sequence.

3.4 PRINCIPAL DIFFERENCES BETWEEN OTM-45 AND EN1948:1

The EN 1948:1 and OTM-45 methods were both developed to capture equivalent proportions of PFAS. However, several key differences were noted between them, which are shown in Table 3.

Table 3. Key similarities and differences between the modified EN 1948:1 and OTM-45 methods for polar PFAS in flue gas.

Parameter	Modified EN 1948:1	OTM-45
Sampling Probe	Cooled sampling probe with a glass liner; condensation starts already in the probe	Heated sampling probe with a quartz liner to maintain temperature and prevent condensation
Collection Media	Two 2-L glass impingers with aqueous solutions (0.1 M NaOH and MilliQ water); PUF holder with integrated aerosol filter and activated carbon	Multi-stage system: glass fibre filter to capture particulates; primary XAD-2 adsorbent module for gaseous-phase PFAS; series of impingers; secondary XAD-2 module to detect breakthrough
Rinsing		
Extraction Procedure	Aqueous fractions: filtration, pH adjustment, SPE using Oasis WAX cartridges; Solid sorbents: Soxhlet extraction (16 h total) with methanol, concentration, pH adjustment, followed by SPE cleanup	Filter and XAD-2: overnight extraction with methanolic ammonium hydroxide (5% v/v), repeated for XAD-2; Impingers: SPE using WAX sorbent; All fractions concentrated and prepared for LC-MS/MS

4 Results

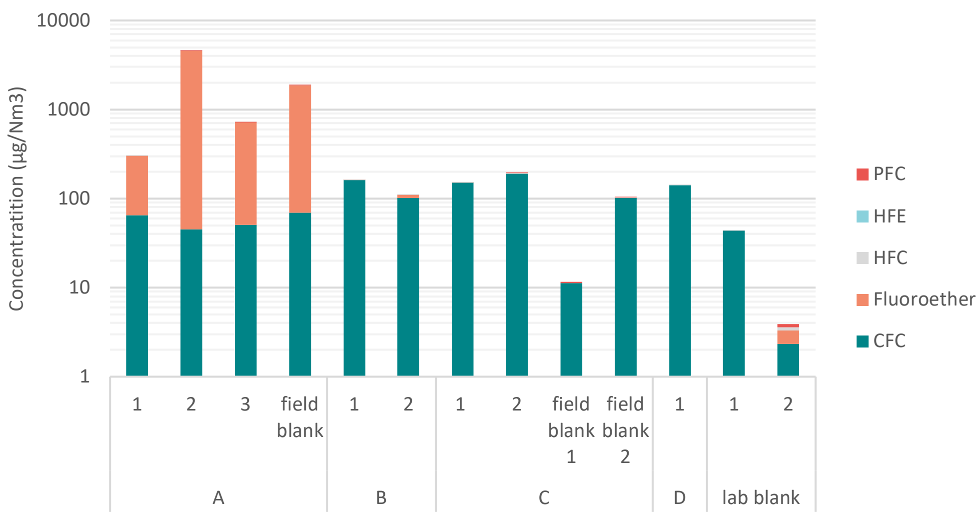
4.1 VOLATILE FLUORINATED COMPOUNDS: OTM-50

4.1.1 Concentration ranges and detection patterns

OTM-50 analysis targeted 29 volatile fluorinated compounds, including chlorofluorocarbons (CFCs), hydrofluorocarbons (HFCs), perfluorocarbons (PFCs), and fluoroethers. Total volatile PFAS concentrations at background facilities (Sites B, C, D) ranged from 110 to 196 µg/Nm^3, indicating relatively consistent emissions from municipal solid waste and hazardous waste processing, as shown in Figure 5. These concentrations exceed polar PFAS emissions (typically <1 ng/Nm^3) by factors of 100,000–1,000,000, indicating that, within the set of compounds targeted in this study, emissions are dominated by volatile fluorinated compounds (including CFCs and HFCs) rather than the polar PFAS that are the focus of most regulatory attention.

Site A exhibited substantially higher concentrations in one of two duplicate samples (Sample A-2: 4,605 µg/Nm^3) due to hexafluoropropene (HFP) variability. When HFP is excluded, site A concentrations align with other facilities (79 µg/Nm^3 average).

Figure 5. Total concentrations of non-polar volatile PFAS measured by OTM-50 at all sites, highlighting the HFPO-dominated event at Site A. *Canister A-2 was excluded from the analysis due to a malfunctioning valve.*



Hexafluoropropene (HFP) exhibited highly episodic behaviour. At Site A, one canister (A-2) contained HFP at 4,500 µg/Nm^3, compared with 240 and 660 µg/Nm^3 in the other two canisters and 1,800 µg/Nm^3 in the site A field blank. At the remaining sites and blanks, HFP concentrations were generally below 10 µg/Nm^3. This pattern suggests a short-lived, site-specific emission event rather than a universal feature of waste combustion. In contrast, hexafluoropropylene oxide (HFPO) was typically close to method quantification limits, with only modest elevations at Site A and in its field blank.

Hydrofluorocarbons (HFCs) and perfluorocarbons (PFCs) contributed minor fractions (<5% each) across all sites. Detected HFCs included pentafluoroethane and 1H-nonafluorobutane. Hexafluoropropylene was the primary PFC detected, though predominantly at Site A.

4.1.2 Duplicate precision and HFP variability

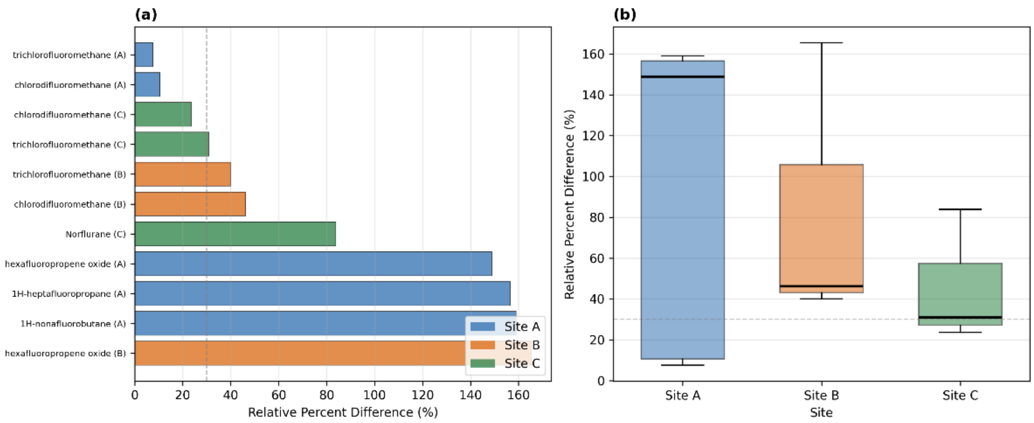
Duplicate OTM-50 canisters were deployed simultaneously at Sites A–C. At Sites B and C, total non-polar volatile PFAS showed good duplicate agreement, with relative per cent differences (RPDs) typically within 10–30%. At Site A, however, the duplicate pair A-1 and A-2 exhibited poor overall agreement (RPD 146%), as shown in Table 4, driven almost entirely by hexafluoropropene variability. When HFP was excluded from the total, the RPD for Site A dropped to levels comparable to those of the other facilities.

Table 4. Relative per cent differences (RPDs) in total non-polar volatile PFAS concentrations between duplicate OTM-50 canisters at each site.

Site	Total PFAS RPD	Median compound RPD	n compounds	Assessment
A	146%	153%	4	Unacceptable (HFP-driven)
B	38%	46%	3	Acceptable
C	25%	31%	3	Good

Compound-specific RPD analysis confirmed that the poor overall precision at Site A was almost entirely attributable to HFP. Most other target compounds showed acceptable duplicate agreement, with a median RPD of 10.5% when hexafluoropropene was excluded from the calculation (Figure 6). This indicates that the OTM-50 method itself performs well for most analytes, and that the apparent precision problem at Site A reflects a single, highly variable compound rather than a systematic issue.

Figure 6. OTM-50 duplicate precision for non-polar volatile PFAS. *Panel A shows compound-specific RPDs for key analytes at each site, and Panel B shows the distribution of RPD values, with most compounds exhibiting acceptable precision.*



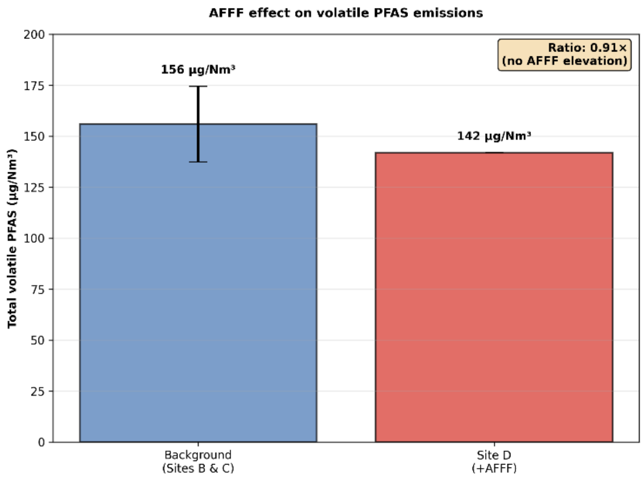
The extreme variability observed for hexafluoropropene suggests episodic emissions occurring on time scales shorter than the sampling period, likely linked to specific waste batches or operating conditions. This illustrates how short-lived events can strongly influence both total concentrations and duplicate precision for individual canisters, and highlights the importance of considering temporal variability when interpreting OTM-50 data (see also Section 6).

4.1.3 AFFF effect assessment

Site D processed hazardous waste spiked with aqueous film-forming foam (AFFF) during the sampling campaign, allowing for the assessment of AFFF's impact on volatile PFAS emissions. Site D showed no elevation in total volatile PFAS (142 µg/Nm^3) compared to other facilities processing municipal solid waste (Sites B and C averaged 156 µg/Nm^3, ratio 0.91×), shown in Figure 7.

At the facility's combustion temperature (1100°C), thermal degradation of AFFF constituents to volatile fluorinated fragments was anticipated but not observed within the OTM-50 target analyte list and detection limits, suggesting either (i) complete mineralisation to HF and CO_2, (ii) insufficient AFFF loading to produce detectable degradation products above background, (iii) effective capture of degradation products by downstream equipment, or (iv) transformation to species outside the current OTM-50 target panel. The effect of AFFF on polar PFAS emissions is discussed in Section 4.3.5.

Figure 7. Total non-polar volatile PFAS concentrations at the AFFF incineration site (Site D, red bar) compared with municipal solid waste sites B and C (blue bars), illustrating that AFFF co-incineration did not increase total volatile PFAS emissions. *Error bars show standard deviations of duplicate canisters.*

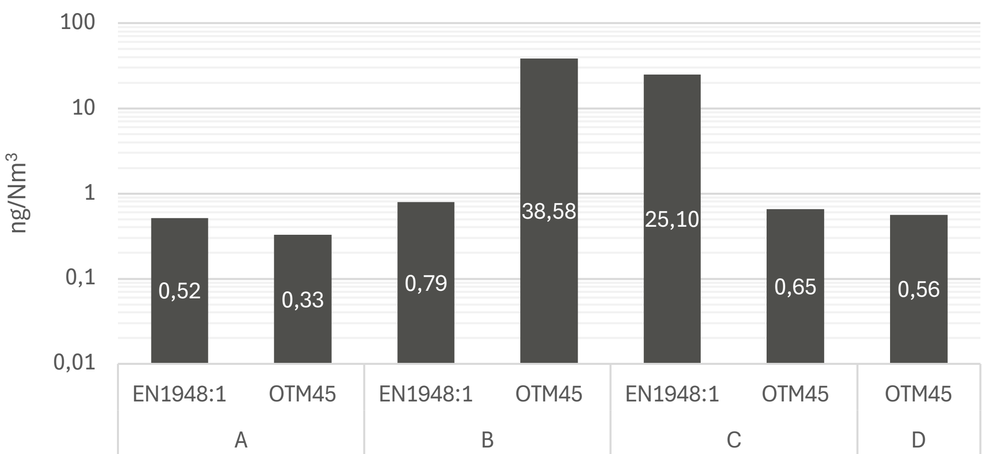


4.2 SEMI-VOLATILE POLAR PFAS: OTM-45 AND MODIFIED EN 1948:1

4.2.1 Total concentrations by facility and method

Total polar PFAS concentrations ranged from 0.33 to 36 ng/Nm^3, with substantial variation both between sites and between methods deployed at the same site (Figure 8). Site A measured 0.33 ng/Nm^3 with OTM-45 and 0.52 ng/Nm^3 with EN 1948:1. Site B measured 36 ng/Nm^3 with OTM-45 and 0.79 ng/Nm^3 with EN 1948:1. Site C measured 0.65 ng/Nm^3 with OTM-45 and 25 ng/Nm^3 with EN 1948:1. Site D measured 0.56 ng/Nm^3 with OTM-45.

Figure 8. Total concentrations of semi-volatile polar PFAS in flue gas by site and method, comparing results from OTM-45 and the modified EN 1948:1 method.



Two elevated samples dominated the concentration distributions. At Site B, OTM-45 measured 38.58 ng/Nm^3 in a single sampling event, with 6:2 fluorotelomer sulfonate (6:2 FTS) contributing 28.74 ng/Nm^3 (75%). This compound was concentrated in equipment rinses (MeOH+NH_4OH fractions), with two rinse bottles contributing 24.19 and 14.24 ng/Nm^3, respectively. At Site C, EN 1948:1 measured 24.99 ng/Nm^3 in one sample, dominated by pentafluoropropanoic acid (PFPeA, 12.95 ng/Nm^3, 52%), perfluorobutanoic acid (PFBA, 4.25 ng/Nm^3, 17%), and perfluoroheptanoic acid (PFHpA, 3.89 ng/Nm^3, 16%). This elevation appeared in the NaOH impinger fraction. All other sample matrices showed concentrations below 1 ng/Nm^3.

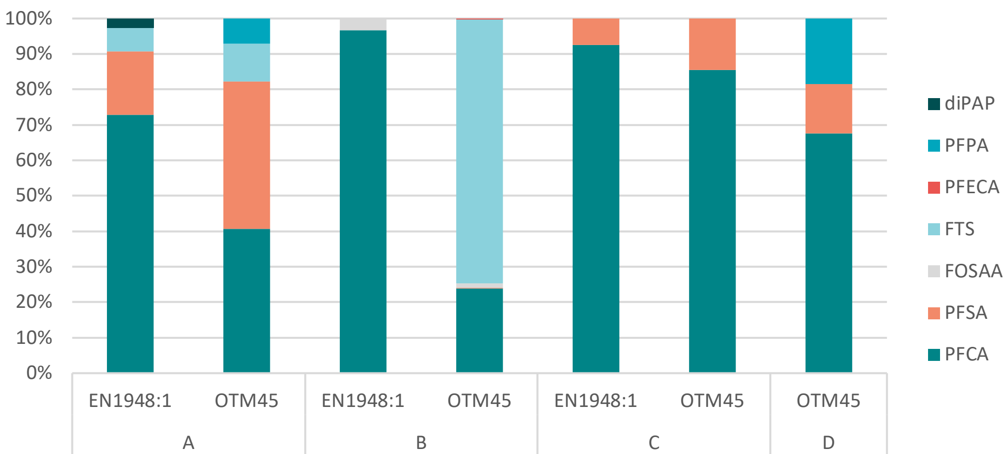
4.2.2 Compositional patterns and method selectivity

Substance group distributions

Perfluoroalkyl carboxylic acids (PFCAs) and perfluoroalkyl sulfonic acids (PFSAs) dominated emissions at Sites A, C, and D, together accounting for 50-90% of total PFAS depending on site and method. Fluorotelomer sulfonates (FTSs) and fluorotelomer acids (FTAs) contributed minor fractions (<10%) at these sites. The two elevated samples showed distinct compositional signatures: Site B OTM-45 contained 75% FTSs (exclusively 6:2 FTS). In contrast, Site C EN 1948:1 contained 84% PFCAs with short-chain compounds (C3-C5), accounting for 81% of the PFCA total, as shown in Figure 9.

EN 1948:1 samples consistently showed higher PFCA fractions than OTM-45 samples from the same sites. At Site A, EN 1948:1 measured 47% PFCAs versus 41% in OTM-45. At Site B, the background EN 1948:1 sample measured 66% PFCAs, while the simultaneously deployed OTM-45 (excluding the elevated sample) showed different distributions. This pattern was observed across all sites where both methods were deployed, suggesting systematic differences in compound-class capture efficiency or selectivity between the methods.

Figure 9. Composition of semi-volatile polar PFAS by substance group (e.g. *PFCAs, PFSAs, FTS, PAPs) for each site and method.*

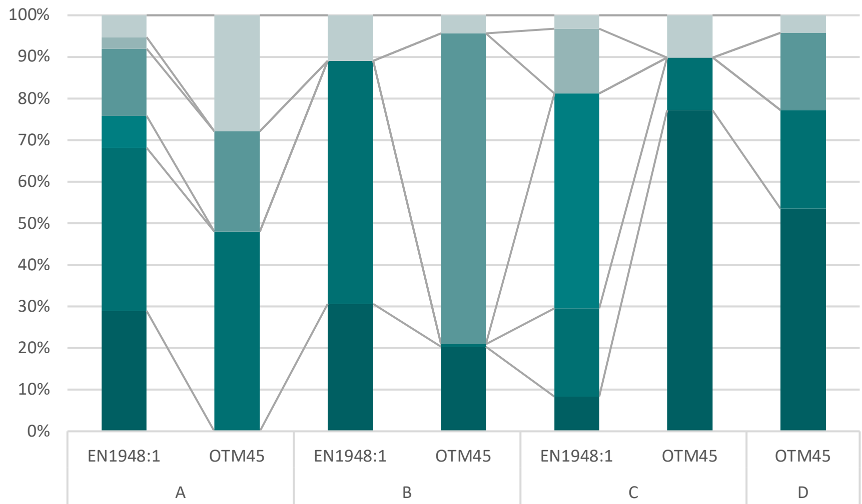


Chain length distributions

Short-chain PFAS (C3-C5) dominated background samples (excluding the two outliers), accounting for 70-90% of compounds with identifiable chain lengths, as shown in Figure 10. C4 compounds (primarily PFBA and PFBS) represented the most abundant single chain length at Sites A and B background samples (40-60% of total). C3 compounds (PFPrA) contributed 30-80% at Sites C and D. Longer-chain compounds (C6-C10) appeared at low concentrations (<10% of total) in most samples.

The elevated samples showed distinct patterns in chain length. Site B OTM-45 contained 76% C6 compounds (reflecting the 6:2 FTS dominance), a pattern not observed in any other sample. Site C EN 1948:1 showed 52% C5 compounds (driven by PFPeA), significantly higher than any background sample. C8 compounds (PFOA, PFOS) contributed <10% across all samples and sites.

Figure 10. Chain-length distribution of semi-volatile polar PFAS by site and method, showing the relative contribution of short- and long-chain homologues.



Influence of elevated samples

The compositional differences between OTM-45 and EN 1948:1 reflect both episodic emission events captured by individual samples and systematic method selectivity visible across all samples. At Site B, the OTM-45 outlier (38.58 ng/Nm^3, 75% FTS) captured an emission event with a compound signature completely absent from the simultaneously deployed EN 1948:1 sample (0.79 ng/Nm^3, 66% PFCAs, 0% FTS). This indicates spatial or temporal variability in emissions during the sampling period.

At Site C, the EN 1948:1 outlier (24.99 ng/Nm^3, 84% PFCAs) showed enrichment in short-chain PFCAs compared to background EN 1948:1 samples at the same site (0.10 ng/Nm^3, 32% PFCAs). The background sample showed 68% unclassified compounds (“Others”), suggesting the elevated sample represents a distinct emission event rather than a simple concentration increase of the same mixture.

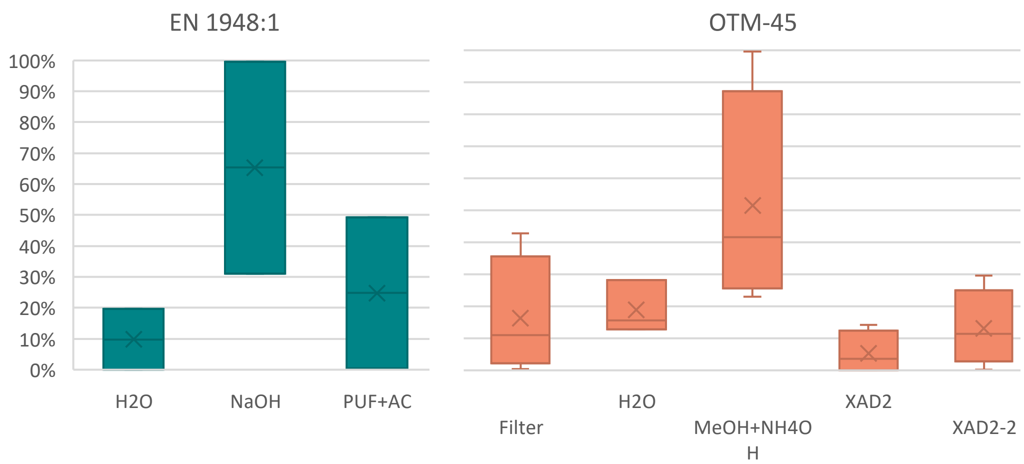
However, method-specific compositional differences persist even after excluding elevated samples. At Site A, where all samples showed background concentrations (<0.3 ng/Nm^3), EN 1948:1 measured 47% PFCAs, while OTM-45 measured 41% PFCAs and 41% PFSAs. This indicates systematic differences in method selectivity independent of concentration level or emission variability.

4.2.3 Internal distribution in OTM-45 sampling train

When all four OTM-45 stack samples are pooled, ≈97 % of the total polar PFAS mass appears in the MeOH+NH_4OH rinses. A single elevated event strongly drives this pattern at Site B, where 6:2 FTS was almost exclusively recovered in the rinse fractions. If the Site B outlier is excluded, the internal distribution across OTM-45 train components becomes much more balanced (MeOH+NH_4OH ≈40 %, H_2O and XAD-2 backup ≈20 % each, and filters ≈20 %), still supporting a dominant role of surface adhesion and rinse recovery but without the extreme skew.

Our internal mass balances show that the vast majority of polar PFAS mass in OTM-45 and EN 1948-1 samples resides in the alkaline aqueous and methanolic rinses (NaOH and MeOH+NH_4OH). In contrast, the primary and breakthrough XAD-2 cartridges typically contribute only a minor fraction of the total. This indicates that, under the studied conditions, the methods operate primarily as impinger- and surface-based collection systems, with XAD-2 functioning mainly as a backup medium rather than the dominant sink for PFAS. The pattern is consistent with observations from the Belgian LUC/VI/003 development work, where condensate passing through the primary XAD-2 cartridge was shown to make the adsorbent substantially wet in moist stacks, prompting the introduction of sampling train variants in which condensate is collected before XAD-2 (Vito, 2023; Hofman, Jacobs, et al., 2025). The subsequent interlaboratory comparison further demonstrated that the original OTM-45, the OTM-45 variant and the cooled probe configuration are analytically equivalent for C4–C18 PFAS, while providing additional insight into the retention behaviour of filters, XAD-2 and impinger water (Hofman, Baeyens, et al., 2025). Our findings therefore provide independent field-based support for the conclusion that XAD-2 performance is sensitive to moisture management in the sampling train, and that design choices that minimise adsorbent wetting are likely to be beneficial without compromising comparability.

Figure 11. Distribution of total polar PFAS mass across EN 1948:1 and OTM-45 sampling train components. *The MeOH used for rinsing in the EN 1948:1 method is included in the PUF+AC fraction; therefore, it is not possible to determine surface interferences for this method.*



4.2.4 AFFF incineration comparison

Site D processed hazardous waste spiked with aqueous film-forming foam (AFFF) during the sampling campaign. The total polar PFAS concentration measured by OTM-45 (summed across all sampling train fractions) was 0.56 ng/Nm^3. For municipal solid waste grate plants without AFFF, total OTM-45 concentrations were 0.33 ng/Nm^3 at site A and 0.65 ng/Nm^3 at site C, i.e. site D falls within the same range as these background sites. Site B showed a much higher total concentration (38.6 ng/Nm^3), dominated by 6:2 FTS, in the MeOH+NH_4OH rinse fractions and is treated as an episodic outlier rather than a typical background condition.

Using Sites A and C as a simple background reference, the mean total polar PFAS concentration is 0.49 ng/Nm^3, and the Site D value corresponds to a ratio of 1.14× relative to this mean and 0.86× relative to Site C. Given that only one OTM-45 train was collected per site, these differences are well within the limited inter-site variability of the dataset and do not provide evidence that AFFF incineration at Site D increased total polar PFAS emissions above typical background levels for municipal waste combustion.

4.3 QUALITY CONTROL AND METHOD PERFORMANCE

4.3.1 Sampling train field blanks and contamination assessment

Sampling train field blanks were collected at Sites A, B, and C to assess background equipment contamination during sampling operations. At Site B, the field blank showed 6:2 fluorotelomer sulfonate (6:2 FTS) contamination in the aqueous impinger fraction (H_2O). In contrast, the elevated sample from the same site showed 6:2 FTS predominantly in equipment rinses (MeOH+NH_4OH fractions). This different distribution pattern between blank and sample suggests distinct contamination sources rather than simple carryover. However, interpretation is complicated by the loss of the impinger water fraction from the elevated sample during transport.

Other field blanks showed low concentrations (<0.3 ng total across all fractions) with detection primarily in equipment rinses, consistent with trace contamination from solvents, glassware, or handling procedures. The sporadic appearance of elevated samples (2 of approximately 20 samples collected) with different compound profiles (6:2 FTS at Site B versus short-chain PFCAs at Site C) and different sampling methods (OTM-45 versus EN 1948:1) suggests genuine emission variability rather than systematic contamination artefacts. However, the limited number of field blanks (one per site) prevents definitive conclusions about contamination versus emission signals at concentration levels below 5 ng/Nm^3.

4.3.2 Internal distribution and recovery

Analysis of PFAS distribution across sampling train components provides insight into collection mechanisms and potential losses. In OTM-45 samples in which multiple fractions contained quantifiable concentrations, equipment rinsing recovered approximately 60% of the total PFAS, while the primary XAD-2 sorbent contributed less than 1%. The particulate filter and aqueous impinger (H_2O) together accounted for only a few per cent of the recovered PFAS, both secondary to surface adhesion and to the impinger/rinse fractions.

This distribution indicates that surface adhesion is the dominant collection mechanism, with chemical recovery via basic solvent rinses essential for quantitative collection. The minor role of gas-phase sorbent capture suggests potential for breakthrough losses during extended sampling periods, though breakthrough assessment was not included in this study design. The effectiveness of methanolic ammonium hydroxide for surface recovery indicates that basic conditions are necessary for deprotonating acidic perfluoroalkyl compounds and mobilising them from glass and metal surfaces.

For EN 1948:1, the elevated sample at Site C appeared predominantly in the NaOH impinger (24.99 ng/Nm^3 total concentration), with minimal contribution from other fractions. This differs from the OTM-45 distribution pattern, suggesting that the collection mechanism varies by method design or that different compound classes partition differently across sampling train components.

4.3.3 Method comparison and concentration variability

Comparison between OTM-45 and EN 1948:1 revealed substantial concentration differences that likely reflect both spatial/temporal emission variability and systematic method selectivity for different compound classes. At Site B, OTM-45 measured 38.58 ng/Nm^3 while EN 1948:1 measured 0.79 ng/Nm^3—a 49-fold difference. At Site C, EN 1948:1 measured 8.37 ± 11.76 ng/Nm^3 (range 0-24.99 ng/Nm^3) while OTM-45 measured 0.11 ± 0.10 ng/Nm^3—a 76-fold difference in mean concentrations.

These differences correlate with distinct compositional patterns. Site B OTM-45 contained 75% fluorotelomer sulfonates while Site B EN 1948:1 contained 66% perfluoroalkyl carboxylic acids. Site C EN 1948:1 contained 84% perfluoroalkyl carboxylic acids, while Site C OTM-45 contained 77% unclassified compounds. Method-specific compositional differences persist even at Site A, where both methods measured background concentrations, with EN 1948:1 showing 47% perfluoroalkyl carboxylic acids versus 41% in OTM-45.

The 100-fold variation in concentration ratios across sites (49× at Site B, 76× at Site C, and 2× at Site A) underscores the fundamental challenges of method comparison when sampling trace contaminants from complex emission sources. Contributing factors include spatial concentration gradients within stack cross-sections, temporal emission variability during 2-4 hour sampling periods, and method-specific selectivity for different compound classes or physical forms. Stack probe positioning was not systematically documented during this study, preventing evaluation of spatial gradient effects. The different compound profiles observed support episodic emissions of specific materials combined with systematic method selectivity rather than simple measurement error.

At background concentration levels (<1 ng/Nm^3), both methods show comparable detection capabilities but different compositional signatures. The practical implication is that single-point grab sampling may inadequately characterise trace PFAS emissions when spatial or temporal variability is present, and that method selection influences which compound classes are preferentially detected.

5 Observations and experiences from field implementation

The field deployment of the three PFAS sampling methods—OTM-45, modified EN 1948:1, and OTM-50—across four Swedish waste-to-energy facilities provided practical insight into their operational performance. Observations reflect the actual implementation rather than theoretical expectations, addressing time requirements, logistics, safety, costs, complexity, and reliability. Detailed descriptions of resources, costs and time requirements can be found in the appendix.

5.1 TIME REQUIREMENTS AND LOGISTICS

Field operations differed markedly in duration and complexity, directly affecting planning, costs, and the feasibility of routine monitoring. OTM-45 required the longest setup and recovery times owing to its multi-component assembly and chemical rinsing, typically occupying a full working day. Modified EN 1948:1 involved moderately complex procedures with fewer parts and robust components, whereas OTM-50 proved to be the simplest, requiring only canister connection and minimal recovery.

In the laboratory, both OTM-45 and EN 1948:1 demanded extensive extraction and cleanup. The latter was most time-consuming due to its prolonged Soxhlet extraction, while OTM-50 required no extraction or cleanup—only passive equilibration before automated GC-MS analysis. The overall elapsed time per sample ranged from about 1 day for OTM-50 to 3 or 4 days for the extraction-based methods.

Logistical demands mirrored methodological complexity. OTM-45 necessitated significant transport of glassware, XAD-2 modules, heating and cooling systems, and chemical reagents. EN 1948:1 required moderate transport and access to cooling water, whereas OTM-50 was compact, limited mainly to canisters and basic leak-check equipment. Personnel needs followed the same hierarchy: two operators for OTM-45, typically two for EN 1948:1 when handling caustic solutions, and one (optionally two) for OTM-50. Sampling throughput reflected this gradient, with OTM-50 achieving up to three samples per day, EN 1948:1 achieving one to two, and OTM-45 generally one sample. Regardless of the method, complete site campaigns typically spanned 2–3 days, encompassing setup, sampling, and demobilisation.

5.2 CHEMICAL SAFETY AND ENVIRONMENTAL ASPECTS

The three methods differed substantially in chemical use and related hazards. OTM-45 originally included dichloromethane (DCM) for resin cleaning, but IVL substituted methanol to mitigate toxic and regulatory risks without compromising analytical quality. Methanol use across all methods required strict flammability controls and adequate ventilation, particularly during the extended Soxhlet extraction process outlined in EN 1948:1. The use of sodium hydroxide in EN 1948:1 introduced corrosivity hazards, demanding personal protection and effective spill management. In contrast, ammonium hydroxide vapours in both extraction-based methods required respiratory precautions. OTM-50 used minimal chemicals, primarily small quantities of methanol and water, confining risks mainly to compressed-gas handling.

The elimination of DCM significantly improved occupational safety and reduced hazardous waste. Overall, OTM-50 presented the least demanding safety profile, whereas OTM-45 and EN 1948:1 remained dependent on solvent and reagent control, ventilation, and operator experience.

5.3 ECONOMIC AND INFRASTRUCTURAL CONSIDERATIONS

The cost analysis indicated that labour accounted for the majority of total expenditures, outweighing consumables. A comprehensive sampling campaign, including duplicate OTM-50 and one OTM-45 train, totalled approximately 300,000 SEK and covered personnel, consumables, analysis, and logistics. Because each method required 2–3 days of fieldwork per site, personnel time determined feasibility more than the costs of chemicals or equipment.

Capital investment differed sharply. OTM-50 required specialised passivated canisters and cleaning infrastructure—a substantial upfront cost but one offset by reusability. Conversely, facilities already equipped with LC-MS/MS instrumentation could adopt OTM-45 or EN 1948:1 without major investment, while those lacking GC-MS with pre-concentrator capability faced barriers to implementing OTM-50.

OTM-50 demonstrated the highest efficiency and the fastest turnaround time, potentially reducing overall monitoring costs for large programs. All three methods benefited from batch laboratory processing, though field labour requirements limited economies of scale. Hidden costs included personnel training, quality-control samples, waste disposal, and equipment maintenance, which varied most strongly with procedural complexity.

6 Discussion

6.1 WHAT THE DATA TELLS US

Volatile fluorinated compounds dominated emissions across all facilities, with concentrations ranging from 100 to 5,000 µg/Nm^3, while polar PFAS concentrations were typically below 1 ng/Nm^3—a difference of 100,000–5,000,000 fold. Within the limits of this dataset, this indicates that waste-to-energy facilities emit predominantly volatile halogenated compounds rather than the polar perfluoroalkyl acids that are the focus of most regulatory attention. Given the low method quantification limits (down to ≈0.01–0.05 ng/Nm^3 for most target compounds), the generally low polar PFAS concentrations observed here are unlikely to be an artefact of insufficient sensitivity.

Chlorodifluoromethane (HCFC-22) and trichlorofluoromethane (CFC-11) together accounted for approximately 95% of total volatile PFAS mass at background facilities, with HCFC-22 typically exceeding CFC-11 by about 100-fold. This stable dominance of HCFC-22 and CFC-11, combined with the non-detection of chlorotrifluoromethane (CFC-13) at our limit of quantification and generally low levels of other halomethanes and haloethanes, defines a distinct CFC/HCFC fingerprint. Several, not mutually exclusive, mechanisms could produce this pattern: (i) incomplete destruction or release of pre-existing HCFC-22- and CFC-11-containing materials in the waste stream, (ii) in-stack formation via halogen radical recombination, and (iii) regional and system background contributions, as suggested by measurable levels of HCFC-22 and CFC-11 in field and laboratory blanks. Because the current dataset does not allow these contributions to be separated unambiguously, we interpret the CFC/HCFC pattern in Section 6.2 in an exploratory rather than definitive way.

Hexafluoropropene (HFP) showed pronounced episodic emission behaviour in the OTM-50 data, with one Site A canister and the corresponding field blank containing much higher concentrations than other samples. This pattern, together with generally low HFP levels at the other sites, suggests that specific waste streams or operating conditions can generate transient peaks of short-chain fluorinated olefins. HFPO itself was detected only at low levels close to method quantification limits and did not exhibit the same pronounced episodic pattern.

Comparison between OTM-45 and modified EN 1948:1 revealed up to 100-fold variation in concentration ratios across sites, with no consistent bias toward either method. These differences likely reflect spatial concentration gradients, temporal variability in emissions, or method-specific selectivity. For a complete discussion of spatial variability and its implications for method comparison, see Section 6.4.

Internal distribution analysis of OTM-45 samples showed that equipment rinsing with methanolic ammonium hydroxide recovered about 60% of total PFAS from sampling train surfaces, while the primary XAD-2 sorbent contributed less than 1%.

This indicates that surface adhesion followed by chemical recovery is the dominant collection mechanism, not gas-phase sorbent capture. The particulate filter and aqueous impinger (H_2O) each contributed about 4–5%, but both are secondary to surface adhesion. These findings have practical implications for OTM-45 implementation, as discussed in Section 6.3.

OTM-50 generally demonstrated good precision for most volatile compounds, with relative per cent differences of 10–40% for trichlorofluoromethane and chlorodifluoromethane at Sites B and C. However, HFPO showed extreme variability, highlighting the limitations of grab sampling for compounds with high temporal emission variability. Continuous or composite sampling would better characterise such emissions. The observed 12.5% canister failure rate underscores the importance of field quality control and redundant sampling.

6.2 SOURCES OF CFCS

HCFC-22 and CFC-11 were detected in all OTM-50 stack samples, with HCFC-22 concentrations ranging from 11–190 µg/Nm^3 and CFC-11 from 0.55–2.2 µg/Nm^3. Both compounds were also present in field and laboratory blanks, generally within the same order of magnitude as stack concentrations. This indicates that the sampling system and/or ambient air carry a measurable background load of these species, while also pointing to net emissions above background at most sites.

Across sites, HCFC-22 exceeded CFC-11 by roughly two orders of magnitude, and CFC-13 remained below the limit of quantification in all samples. In addition, several "neighbouring" halomethanes and haloethanes (e.g., HFC-23, HFC-32, HFC-143a, and other target C1–C2 species) rarely exceeded the reporting limits and did not show the same consistent enhancement as HCFC-22 and CFC-11. The combination of (i) a stable HCFC-22:CFC-11 ratio, (ii) non-detection of CFC-13, and (iii) an absence of a broader homologous series of C1–C2 halocarbons suggests a relatively selective source. At the same time, the lack of a whole "ladder" of related compounds weakens a simple interpretation in terms of a fully developed radical pool generating a broad spectrum of halomethanes.

Taken together, the data support several plausible, non-exclusive scenarios:

1. **Incomplete destruction or release of legacy products.**
   HCFC-22 and CFC-11 have historically been used as refrigerants and blowing agents. Emissions could therefore reflect partial survival or release of these substances from waste containing old equipment, foams, or other legacy materials, with destruction efficiencies high but not complete. Similar concerns have been raised for POP-containing materials in construction and demolition waste. Ghasemi et al. (2025) documented that products and materials containing regulated POPs are not always correctly identified

or segregated, and can enter general waste streams rather than dedicated hazardous waste treatment. While their study focused on POPs rather than ozone-depleting substances, it illustrates how limitations in information on chemical content, guidance and sorting practices can allow legacy halogenated materials to reach waste-to-energy plants. In this context, incomplete destruction of CFC- and HCFC-containing building products in mixed waste streams appears as a plausible contributor to the HCFC-22 and CFC-11 patterns observed here, alongside in-stack formation and background.

2. **In-stack formation via radical chemistry.**
   Combustion of halogenated materials can generate Cl-, F-, and C-centred radicals. One conceivable pathway is that difluorocarbene-type species (CF_2) formed during fluoropolymer decomposition react with hydrogen chloride or other chlorine sources to yield HCFC-22, with subsequent chlorination producing CFC-11. Under this scenario, the observed ~100:1 HCFC-22:CFC-11 ratio and the absence of CFC-13 could be qualitatively consistent with kinetic and thermodynamic constraints that favour HCFC-22 and disfavour further chlorination. However, the limited occurrence of chemically related “neighbour” species in our dataset argues against this pathway being the only or dominant source.

3. **Background and system contamination.**
   The presence of HCFC-22 and CFC-11 in both field and laboratory blanks shows that background contamination cannot be neglected. This may reflect regional atmospheric levels, emissions from on-site infrastructure, or adsorption/desorption on sampling materials. For some samples, the difference between stack concentrations and blanks is modest, implying that net emissions and background contributions are difficult to separate with high confidence.

Because these processes cannot be disentangled with the available data, we interpret the observed CFC/HCFC pattern as resulting from a combination of legacy emissions, possible in-stack formation, and background loads, rather than as definitive evidence for any single mechanism. From an atmospheric perspective, even modest de novo formation of CFC-11 and HCFC-22 in waste-to-energy plants could be relevant if it proves widespread and persistent. At present, however, our dataset is too limited to distinguish quantitatively between formation and survival, or to extrapolate robustly to regional or national emission inventories.

Current emission inventories primarily attribute CFC and HCFC releases to leaks from equipment and foams. Our observations show that stack samples from modern combustion sources can contain measurable levels of HCFC-22 and CFC-11, at or above background levels. However, the relative contributions of incomplete destruction, in-stack formation, and background remain unresolved. Potential mitigation strategies—regardless of the dominant mechanism—include reducing chlorine and fluorine inputs (e.g., separating PVC- and fluoropolymer-rich fractions), optimising combustion conditions to enhance destruction efficiency, and considering post-combustion treatment for halomethanes.

The broader volatile fluorinated fingerprint provides additional context. We detect multiple perfluorocarbons and hydrofluorocarbons at low concentrations, often close to blank levels. These may arise from analogous formation pathways, from survival of fluorinated constituents in the waste stream, from background, or from a combination thereof. The generally low abundance and proximity to blanks make clear attribution difficult. The absence of highly chlorinated species in our target list may indicate efficient destruction of heavier chlorinated organics under the studied conditions, or may reflect limitations of the analytical panel and detection limits.

Overall, the data suggest that waste-to-energy facilities can act as sources of CFCs and HCFCs. However, the balance between de novo formation and incomplete destruction remains an open question. Resolving this will require targeted experiments with upstream/downstream sampling, better characterisation of background levels, and expanded speciation of co-emitted halogenated compounds.

6.3 METHOD PERFORMANCE AND LIMITATIONS

OTM-50 performs well for volatile compounds, with adequate concentrations and good precision. The 12.5% canister failure rate is manageable with field QC and redundant sampling. Temporal variability is high for some compounds, such as HFPO.

OTM-45 and EN 1948:1 are technically sound for polar compounds but operationally complex, involving multiple matrices in which surface adhesion appears critical. Within our OTM-45 dataset, impinger- and rinse-based fractions (NaOH, H_2O, and MeOH+NH_4OH) account for approximately 95–97% of the recovered PFAS mass, while XAD-2 sorbents contribute well below 1%. This indicates that, under the conditions studied, impinger-based collection and surface recovery dominate over gas-phase sorbent capture. For short-chain PFAS (C3–C4), most of the mass was observed in downstream liquid and rinse fractions rather than in upstream filters or sorbents, consistent with substantial breakthrough under extended sampling. PM extraction appears to show a positive bias and would benefit from further optimisation and, where possible, correction. Spatial sampling variability and episodic emission events likely dominate much of the apparent method disagreement between simultaneous samples.

6.4 SPATIAL VARIABILITY AND SINGLE-POINT SAMPLING LIMITATIONS

Spatial concentration variability is significant. Polar PFAS varied 30-100 fold between simultaneously collected samples at different probe locations, demonstrating that flue gas is not perfectly mixed. Even in turbulent duct flow, recent combustion events and temperature gradients create concentration “pockets” that may be captured or missed depending on probe position. This variability challenges the representativeness of single-point grab samples and explains apparent “method disagreement” when samples are collected at different locations.

Opposite concentration patterns in simultaneously collected samples (e.g., Site B: OTM-45 elevated 30 times over EN 1948:1; Site C: EN 1948:1 elevated 86 times over OTM-45), combined with field blank validation, reveal fundamental limitations of single-point grab sampling for stratified emission sources.

The spatial stratification mechanism operates as follows: incomplete mixing in the stack creates concentration gradients, recent combustion events produce localised “plumes,” and different probe positions sample different zones. Which probe samples an elevated zone versus background is random, creating an apparent “method disagreement” that is actually a spatial sampling artefact.

For regulatory monitoring, single grab samples are problematic for compliance due to spatial variability. Multiple probe locations are required, in accordance with EPA multi-point traverse requirements. Co-located probes are necessary for a valid method comparison. Statistical frameworks must accommodate spatial and temporal variability. Continuous monitoring would be ideal, but it is not feasible with current PFAS methods.

Our findings also align with broader evidence that legacy halogenated substances in products are not always managed in line with their regulatory status. Ghasemi et al. (2025) concluded that POP-containing products and materials frequently appear in waste streams where their POP status is not recognised, increasing the risk of uncontrolled dispersion rather than controlled destruction. For PFAS and ozone-depleting substances in waste-to-energy feedstocks, this implies that stack measurements alone are not sufficient; upstream product identification, more detailed guidance for construction and demolition waste, and stricter control of mixed combustible fractions are also needed to ensure that legacy materials reach appropriate treatment routes.

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A Appendices

Table 5. Estimated field and laboratory time requirements per sample for each PFAS method (OTM-45, EN 1948:1 and OTM-50), including sampling, extraction and analysis.

Activity	OTM-45	Modified EN 1948:1	OTM-50
Field Operations			
Setup and equilibration	2–3 h + 0.5–1 h heating	1–2 h	0.5–1 h
Sampling duration	By design	By design	By design
Recovery and breakdown	2–3 h	1–1.5 h	0.1–0.2 h
Total field time	6–10 h	4.5–7.5 h	2.5–5 h
Laboratory Processing			
Extraction	8+ h (overnight)	16 h (8+8 h Soxhlet)	None
SPE cleanup	2–3 h	2–3 h	None
Concentration	2–4 h	2–3 h	None
Sample equilibration	—	—	12 h (passive)
Instrumental analysis	2–3 h (LC-MS/MS)	2–3 h (LC-MS/MS)	0.75–1 h (GC-MS)
Total active lab time	12–18 h	22–25 h	0.75–1 h
Total elapsed time	**~2–3 days**	**~3–4 days**	**~1 day**

Table 6. Chemical inventory for each method, where the OTM-45 includes the adjusted method where di-chloromethane was excluded from the method due to work safety and environmental concerns.

Chemical Category	OTM-45 (IVL Protocol)	Modified EN 1948:1	OTM-50
Methanol (flammable)	200–400 mL field recovery + 360 mL per XAD-2 module	400 mL Soxhlet extraction	Minimal (cleaning only)
Methanolic NH_4OH (5% v/v)	200–400 mL field recovery	0.1% NH_4OH (SPE, ~8 mL)	None
NaOH solution (0.1 M)	None	200 mL impinger	None
Acetic acid	pH adjustment (~10–20 mL)	pH adjustment (~10–20 mL)	None
Ultrapure water	SPE procedures	300 mL impinger + SPE	15 mL per impinger (when needed)
Dichloromethane (DCM)	Eliminated	Not used	Not used
Primary Hazards	Flammability (methanol); alkalinity (NH_4OH vapors)	Flammability (methanol); corrosivity (NaOH)	Minimal chemical hazards

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