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Research Paper

Sensitivity of PFAS-derived Fluorine Metrics to Target-List Composition, LoQ Distributions, and Non-Detect Handling in Fluoropolymer Combustion at a Pilot-Scale Waste Incineration Plant

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ABSTRACT

Targeted analytical methods are widely used to quantify per- and polyfluoroalkyl substances (PFAS) in combustion flue-gas. However, interpretation of PFAS emission data is challenged by high shares of non-detects (NDs), heterogeneous limits of quantification (LoQs), and extensive target analyte lists. This study evaluates the influence of ND-treatment, target-list composition and LoQ distributions on PFAS-derived fluorine metrics using two pilot-scale incineration datasets.

Both datasets exhibit high ND-shares and analyte-specific LoQs spanning several orders of magnitude. Current analytical target lists appear highly sensitive to the ND-substitution approach. However, sensitivity analyses demonstrated that this apparent sensitivity was governed by a small number of analytes exhibiting elevated LoQs. Excluding these analytes reduced variability among ND-substitution approaches by approximately 95 % in one dataset and by more than 99.9 % in the other, largely independent of the selected descriptive statistical parameter.

Target-list reduction also decreased variability but provided little additional improvement. Consequently, the apparent influence of target-list composition was found to be largely attributable to the inclusion of a small number of analytes with exceptionally high LoQs.

The findings indicate that apparent sensitivities to ND-treatment are governed primarily by analyte-specific LoQ distributions, and, to a lesser extent, by target-list composition. Robust assessment of PFAS-derived metrics therefore primarily requires critical evaluation of analytes exhibiting elevated LoQs, transparent handling of NDs and the application of lower- and upper-bound estimates. Future method development should prioritize harmonized LoQs, refinement of target lists, and inclusion of volatile PFAS and non-target analysis to improve PFAS emission assessment.

1. Introduction

Per- and polyfluoroalkyl substances (PFAS) constitute a large and highly diverse class of more than 10,000 fluorinated chemicals (US EPA, 2022) that are widely used in industrial and commercial applications due to their thermal and chemical properties (Gaines, 2023; Glüge et al., 2020; Habib et al., 2024). They include fluoropolymers (FPs) and a broad range of non-polymeric PFAS, including perfluoroalkyl carboxylic acids (PFCAs), perfluoroalkyl sulfonic acids (PFSAs) and various fluorinated replacement chemicals (Berkowitz et al., 2025; Chen et al., 2026; Li et al., 2022).

The widespread use of PFAS inevitably generates PFAS-containing waste streams requiring suitable end-of-life treatment (Chen et al., 2026). Municipal waste incineration plants (MWI) and hazardous waste incineration plants (HWI) are generally considered effective for PFAS destruction, provided that sufficiently high temperatures and residence times are maintained (Ross et al., 2018; Vecitis et al., 2009; Wother et al., 2026). Thermochemical studies demonstrate that complete mineralization of PFAS requires overcoming high C–F bond energies and may yield transient radicals such as CF_2 and CF_3, which can recombine or form volatile products of incomplete combustion (PICs) (Krug et al., 2022; Tsang et al., 1998).

PFAS in flue gas are typically quantified via targeted analytical methods. The U.S. EPA Other Test Method 45 (OTM-45) covers 49 polar,

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semi-volatile, and condensable PFAS (Berkowitz et al., 2025) and is widely applied for stack testing. Various adaptations are used in practice, including modified OTM-45 sampling trains and national extensions such as LUC/VI/003 (Hofman et al., 2025a; VITO, 2024). Complementary methods include OTM-50 for 30 volatile analytes, including known PICs such as fluoromethane (Jackson et al., 2025), and the developing OTM-55 for non-polar, semi-volatile PFAS (Jackson, 2024).

The relevance of individual PFAS classes within current target lists remains an active area of discussion. Thermochemical studies suggest that small fluorinated compounds and volatile PICs, including CH_3F, CHF_3, CF_2O and CF_4, are among the most plausible products under typical combustion conditions, whereas many long-chain PFAS included in current target methods are considered thermally unstable are therefore expected to occur less frequently in combustion emissions (Krug et al., 2022; Tsang et al., 1998).

This aligns with observations that flue-gas, residues and leachates from waste-incineration processes predominantly contain short-chain PFCAs and PFSAs, while many targeted analytes either remain undetected or occur at trace levels (Björklund et al., 2024; Chen et al., 2026; Hofman et al., 2025b; Liu et al., 2021; Shields et al., 2023; Solo-Gabriele et al., 2020; Weitz et al., 2024).

In contrast to flue-gas analysis, PFAS monitoring in aqueous matrices is well established and frequently complemented by non-target analysis (NTA), providing broader insight into PFAS occurrence and precursor transformation (Gehrenkemper et al., 2021; Güzel, 2026, 2025; Johansson et al., 2024; Zahra et al., 2025). NTA is increasingly explored for air and flue-gas applications, with various studies showing that it can identify PFAS outside current target lists and significantly improve fluorine-based assessments (Place et al., 2024; Reis de Carvalho et al., 2026; Shields et al., 2023).

A major challenge in PFAS-emission assessment is the treatment of data below the limit of quantification (LoQ), commonly referred to as censored-data or non-detects (NDs). In this context, 'detect' is often used as a synonym for 'quantifiable'. While the limit of detection (LoD) represents the lowest concentration distinguishable from background noise, the LoQ defines the lowest concentration that can be quantified with acceptable accuracy (Wenzl et al., 2016). When defined by signal-to-noise ratios (S/N), the LoD typically corresponds to $S/N = 3$, and the LoQ to $S/N = 10$ (FDA, 2023).

A distinction is required between instrumental, method, and procedural LoQs, which reflect the sensitivity of the instrument alone, the full analytical workflow, and the entire sampling process, respectively (Appendix A: Supplementary information, Figure SI 1). Consequently, LoQs can vary by several orders of magnitude among analytes, matrices, and analytical methods. Reported analyte-specific LoQ ratios range from approximately 2 to 1000 for PFAS combustion studies, highlighting the substantial differences in analytical sensitivity that currently exist among target analytes (Appendix A: Supplementary information, Table SI 1).

At the same time, PFAS flue-gas datasets are commonly characterized by high shares of NDs with only a limited number of analytes being regularly quantified (Aleksandrov et al., 2019; Gehrmann et al., 2024; Hofman et al., 2025b; Shields et al., 2023). The combination of heterogeneous analyte-specific LoQs and ND-dominated datasets may therefore become a major source of uncertainty when PFAS-derived fluorine concentrations and destruction metrics are calculated.

These challenges become particularly relevant when PFAS concentrations must be aggregated, because NDs are typically reported as "< LoQ", whereas subsequent calculations require explicit assumptions regarding their treatment.

In addition, the choice of descriptive statistical parameters can lead to widely different outcomes depending on the share of NDs and the magnitude of the LoQs. Arithmetic mean values are generally more sensitive to extreme values and surrogate concentrations assigned to NDs, whereas median values and the interquartile ranges (IQRs) are often considered more robust for skewed distributions or large shares of NDs.

Although several advanced ND-estimation methods are available, they typically require approximately 25–50 quantified observations and additional assumptions regarding the underlying data distribution to provide reliable estimates, even with moderate levels of censoring, whereas robust parametric approaches may remain applicable at ND shares of up to 70–80 % (Baccarelli et al., 2005; Groat, 2002; Helsel, 2011; Huybrechts et al., 2002; Hwang et al., 2023). Because PFAS flue-gas datasets typically contain few quantified observations, highly variable analyte-specific LoQs, and large shares of NDs, these methods are often difficult to apply. Consequently, simple substitution approaches assigning surrogate values such as 0, LoQ/2, LoQ/$\sqrt{2}$, or LoQ to all NDs remain widely used (DIN, 2014; Hornung and Reed, 1990; Ministry of the Environment of Japan, 2001; Rupp et al., 2025; Salangad et al., 2025; Wang et al., 2020; Yu et al., 2025) despite their potential to introduce bias (Baccarelli et al., 2005; Hwang et al., 2023; Zhao and Frey, 2006).

The present study investigates how target-list composition, analyte-specific LoQ distributions, ND-substitution approaches and descriptive statistical parameters influence the PFAS-derived fluorine metric Reduction Rate for Products of Incomplete Destruction (R2PIC) using data from two PFAS combustion studies conducted at the BRENDA pilot plant at Karlsruhe Institute of Technology (KIT). Particular emphasis is placed on identifying the factors responsible for the apparent sensitivity of PFAS-derived fluorine metrics to ND treatment, particularly the relative influence of elevated-LoQ analytes and broader target-list composition. The findings provide methodological guidance for PFAS emission assessment and identify priorities for future method development, including LoQ harmonization and target-list refinement.

2. Current handling of NDs, LoQs and PFAS-metrics in studies

When PFAS are incinerated at pilot- or industrial-scale, a range of metrics is used to evaluate destruction or mineralization, including the destruction and removal efficiency (DRE), the destruction efficiency (DE), and the R2PIC. While DE and DRE describe the degradation of individual PFAS, they do not directly account for the formation of fluorinated transformation products and therefore should not be interpreted as measures of complete fluorine mineralization. Likewise, R2PIC reflects the relative reduction of analytically targeted fluorinated PICs and therefore provides an indicator of incomplete PFAS degradation rather than a direct measure of total fluorine conversion or complete mineralization to hydrogen fluoride (HF), which is generally considered the dominant end-product of complete PFAS mineralization. The detailed calculation of these metrics is described elsewhere (Basel Convention, 2025; Gehrmann et al., 2024; Troxler et al., 2025; van Caneghem et al., 2025).

A study conducted at a HWI quantified 37 and 45 PFAS, respectively, and substituted all NDs with 'zero' (Hofman et al., 2025b), elsewhere an additional calculation was performed using LoQ/2 (Hofman et al., 2023). In the latter study, summed PFAS concentrations differed by 65 – 143 % depending solely on the selected ND-substitution approach, illustrating the potential influence of post-processing decisions on PFAS emission data.

A subsequent investigation at this facility based on three background measurements and one spiking test, reported DRE values between 99.99961 % and 99.999998 % for eight persistent organic pollutant PFAS (POP-PFAS) using mean concentrations and LoQ/2 substitution for NDs (van Caneghem et al., 2025). Similarly, another HWI study reported DRE values between 99.9452 % and 99.9999 % for nine spiked PFAS and corresponding DE values between 99.9753 % and 99.9992 %, substituting NDs with the MQL (Troxler et al., 2025).

Studies examining PFAS in untreated flue gas, stack emissions, and residues from MWIs performed three measurements per setting and estimated concentrations between the LoD and LoQ while assigning NDs

as ‘zero’ (Björklund et al., 2024, 2023). A similar approach was used in a pyrolysis study evaluating PFAS removal from multiple waste streams, where NDs were substituted with ‘zero’ and values between the LoD and LoQ with LoQ/2 (Sørmo et al., 2023).

In a Swedish industrial-scale MWI study assessing 13 PFAS in residues, NDs were substituted using the MQL and mean values were used for evaluation (Johansson et al., 2024). Another investigation reported a destruction rate of 99.999891 % for liquid PFOS in an industrial HWI, based on one background and one spiking measurement, substituting NDs with the LoQ (Ministry of the Environment of Japan, 2013).

In a pilot-scale combustor, DE values for eight PFCAs and PFSAs ranged from 45.7362 % to > 99.9999 % depending on temperature and analyte; with NDs substituted by the LoD based on one measurement per temperature (Shields et al., 2023). A pilot-scale HWI study substituted NDs with LoQ/2 to calculate mean PFAS concentrations in flue gas with and without PTFE addition, based on at least five baseline and spiking measurements under different post-combustion chamber (PCC) conditions (Aleksandrov et al., 2019). A subsequent study at the same plant used median values based on three background and three spiking measurements per PCC setting to report R2PIC values > 99.99 % when NDs were set to ‘zero’ and > 99.9 % when LoQ/2 was applied (Gehrmann et al., 2024).

Across studies, the reported destruction, degradation, or mineralization metrics differ considerably in their analytical scope, the analyte-specific LoQs, the treatment of NDs and the choice of descriptive statistical parameters. Notably, several studies report target lists containing analytes with LoQ ratios spanning approximately three orders of magnitude (Appendix A: Supplementary information, Table SI 1). Such heterogeneity creates the potential for a small number of analytes to dominate calculated fluorine loads and derived metrics, particularly in ND-dominated datasets.

Furthermore, many destruction metrics are reported with four or even six decimal places (Ministry of the Environment of Japan, 2013; Shields et al., 2023; Troxler et al., 2025; van Caneghem et al., 2025). Such reporting is often motivated by regulatory frameworks, where destruction and removal efficiencies of 99.999 % or 99.9999 % may be required for specific applications (Basel Convention, 2025).

Given the substantial influence of target-list composition, analyte-specific LoQs, matrix effects, sampling-related uncertainty, temporal process variability and ND-treatment assumptions on PFAS quantification, particularly in datasets dominated by NDs, the apparent precision implied by reporting destruction metrics to four or more decimal places frequently exceeds the accuracy realistically achievable in pilot- and industrial-scale combustion studies.

Consequently, such levels of numerical precision may reflect the mathematical outcome of data-processing assumptions rather than experimentally verifiable differences in PFAS destruction performance. This complicates cross-study comparisons and underscores the need for transparent uncertainty reporting when interpreting PFAS destruction metrics.

3. Origin of the experimental data: The BRENDA studies

Two comprehensive studies on fluoropolymer combustion were conducted at the BRENDA pilot-scale waste incineration facility. The plant is equipped with a rotary kiln, a downstream PCC, a boiler, and a flue-gas cleaning system operated in accordance with European emission control requirements. These components enable well-controlled experimental conditions that closely resemble those of industrial-scale municipal and hazardous waste incinerators.

The first study, further referred to as ‘study I’, investigated whether PFAS are released during the combustion of PTFE. To distinguish combustion-related emissions from plant background, experiments were conducted alternately with and without PTFE feed. Two PCC settings differing in temperature and gas residence time were examined to assess the influence of afterburning conditions. Flue-gas samples were analyzed for 31 PFAS using a method based on U.S. EPA Method 5 (Aleksandrov et al., 2019).

The subsequent ‘study II’ evaluated the thermal degradation of a mixture of fluoropolymers and included an expanded analysis of liquid and solid residues in addition to the flue gas. In total, 41 PFAS were quantified across all process streams. Two PCC settings were again compared, and the R2PIC was determined based on the measured concentrations of organic fluorine attributable to PICs. Flue-gas sampling and analysis followed OTM-45, complemented by an additional canister sampling position downstream of the filter to quantify six volatile C_1-C_4 perfluorocarbons (PFCs) (Gehrmann et al., 2024).

Because R2PIC is based on fluorine associated with analytically targeted products of incomplete combustion (PICs), it is essential to clearly define which species are considered as such. Analogous to carbon monoxide in hydrocarbon combustion, PICs represent fluorine-containing products that form when PFAS are not fully mineralized. Under conditions of complete thermal degradation and sufficient hydrogen availability, HF is expected to be the dominant fluorine-containing end product. In contrast, incomplete combustion may generate a range of fluorinated PICs, including small fluorinated substances such as COF_2 and SiF_4 and volatile C_1–C_2 PFAS (Silsby et al., 2026; Strandberg et al., 2025; van Caneghem et al., 2025). Consequently, R2PIC provides an indicator of the reduction of fluorine associated with analytically targeted fluorinated PICs and can therefore be used to assess the extent of incomplete PFAS degradation. However, it does not represent either a complete fluorine mass balance or the total conversion of fluorine to HF.

4. Method

Measurements based on OTM-45, OTM-50 or adapted methodologies require logistical effort and are associated with substantial costs. Consequently, the number of feasible measurements is typically limited, resulting in small datasets (see chapter 2).

In both studies, three to six individual measurements with added fluoropolymers and flue gas samples collected downstream of the boiler were performed for each PCC setting and form the basis of the subsequent calculations. These numbers are comparable to, or exceed, most of those published in pilot- and industrial scale PFAS-combustion studies.

However, as described in the Introduction, the datasets remain too limited for the reliable application of advanced ND-estimation methods. Therefore, substitution-based approaches represent the most practical option for subsequent calculations. To evaluate the influence of ND-treatment on R2PIC, four commonly used substitution approaches were applied, in which the concentration c of NDs was set to 0, LoQ/2, $LoQ/\sqrt{2}$ and LoQ, respectively.

Approaches 1 and 4 correspond to lower- and upper-bound scenarios and are widely used to bracket the uncertainty associated with NDs. This practice provides optimistic (lower-bound) and conservative (upper-bound) estimates of the investigated metric and is frequently applied in exposure and risk-assessment studies (EFSA, 2010; EPA, 2000; FAO, WHO, EFSA, 2011; Robichaud et al., 2009). The resulting values should therefore be interpreted explicitly as bounding estimates rather than point estimates of the true concentration or degradation performance.

In addition to R2PIC, the PFAS-derived fluorine concentrations ($\mu g_{F,PFAS}/m^3_{N,dry}$) were calculated by summing the molar fluorine contributions of all quantified and substituted PFAS relative to the dry flue-gas volume. These concentrations represent only the fluorine associated with the PFAS included in the analytical scope and do not account for inorganic fluorine species, particularly HF.

To evaluate the influence of methodological choices beyond ND treatment, two sensitivity analyses were performed for both studies. First, analytes exhibiting substantially elevated LoQs relative to the remaining target compounds were excluded. This analysis was intended to distinguish LoQ-related effects from those associated with the ND-substitution approach itself.

Second, calculations were repeated using a reduced target list consisting only of PFCAs up to PFOA and PFSAs up to PFOS. All other PFAS were excluded irrespective of whether they were quantified or reported as NDs. This simplified, literature-based scenario was derived from PFAS groups most frequently reported in combustion studies and was used solely to assess the influence of target-list composition. It should not be interpreted as a thermochemical classification of PFAS combustion products.

It is important to note that the two datasets are not intended for direct quantitative comparison because they differ in experimental design, analytical scope, target-list composition and analyte-specific LoQ distribution. Instead, they were analyzed independently to evaluate whether similar methodological effects can be observed across different PFAS combustion studies.

5. Results and discussion

5.1. Current analytical practice: limitations of PFAS combustion datasets

The influence of descriptive statistical parameters on data interpretation is illustrated using PFOA-derived fluorine concentrations from 'study I' (Fig. 1), the only analyte quantified above the LoQ in all samples.

In Setting 1, the concentration range is narrow and mean and median nearly coincide, indicating the absence of influential outliers. Setting 2 exhibits a systematic shift toward lower concentrations, reflected in a reduced median and IQR. A single elevated maximum value in Setting 2 suggests contamination or analytical artefacts, resulting in a marked divergence between mean and median.

This illustrates the sensitivity of arithmetic means to isolated observations and demonstrates that median values are generally more robust in the presence of outliers. At first glance, the choice of descriptive statistical parameter therefore appears to influence the interpretation of PFAS combustion datasets.

The limitations imposed by PFAS-combustion datasets become apparent when the share of quantified analytes is considered (Fig. 2). In 'study I', only PFOA exceeded the LoQ in a sufficient number of samples to permit robust statistics such as medians and IQRs. Most PFAS were quantified only sporadically, primarily short-chain PFCAs, whereas other analytes remained consistently below the LoQ.

Under these conditions, descriptive statistical parameters rapidly lose interpretative value. When substitution approaches are applied, the resulting estimates become increasingly influenced by the surrogate values assigned to NDs. Consequently, PFAS-derived fluorine metrics may be governed less by measured concentrations than by the treatment of values reported below the LoQ.

The results of 'study II' further confirm this pattern (Appendix A: Supplementary information, Figure SI 2). Only PFOS and 6:2 FTS exceeded the LoQ once in setting 1 and 2, respectively, while all remaining analytes were reported as NDs. Thus, despite the larger analytical scope, the effective amount of quantified information available for statistical evaluation was even lower than in 'study I'.

These findings are consistent with observations from other PFAS combustion studies. A comparison of quantification frequencies reported in the literature (Fig. 3) shows that only a limited number of PFAS are quantified consistently across studies. PFBA, PFOA and PFBS exhibit the highest quantification shares, PFOS and HFPO-DA also show comparatively high quantification shares when included in the analytical scope. In contrast, most other PFAS classes are either not analyzed or remain entirely below the LoQ.

The consistently low quantification shares observed in both the BRENDA studies and published combustion investigations indicate that ND-dominated datasets represent the current analytical reality rather than an exception. Consequently, the selection of ND-substitution approaches becomes an important methodological decision whenever PFAS-derived fluorine concentrations or metrics are calculated. At the same time, the consistently high share of NDs suggests that analyte-specific LoQs may exert a substantial influence on the resulting values.

To improve the reliability of PFAS emission assessments, detailed reporting of analytical sensitivity is therefore essential. In particular, a clear distinction between analytes reported below the LoD and those reported between the LoD and LoQ, transparent reporting of quantification shares, and explicit documentation of ND-treatment procedures are required to ensure comparability between studies. Reporting substances exclusively observed below the LoD separately from quantified analytes may further reduce the risk of artificially inflating fluorine loads through substitution-based calculations.

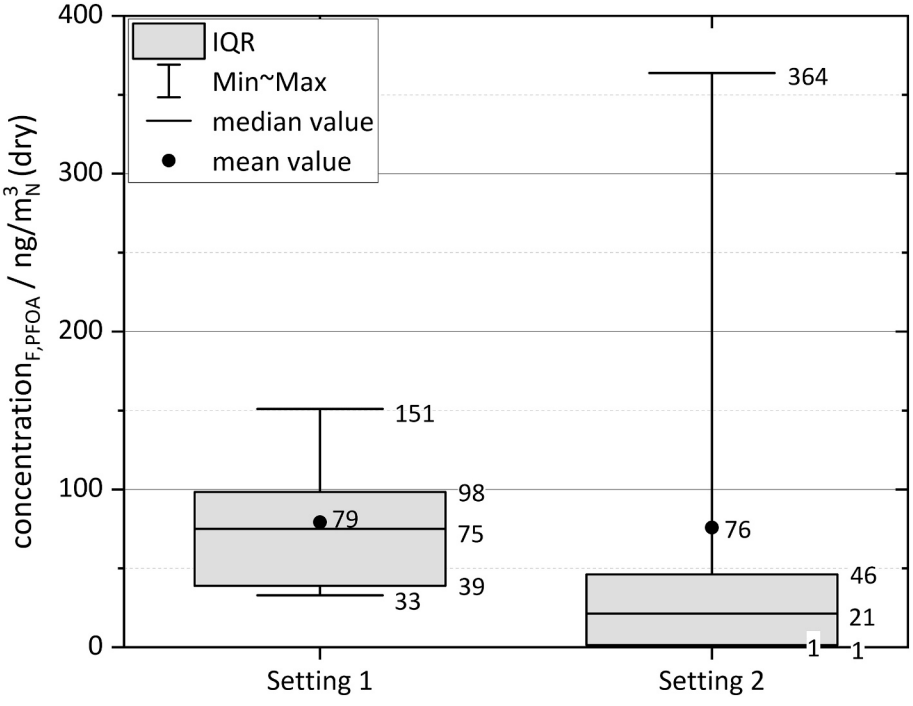

Fig. 1. Box plots of PFOA-derived fluorine concentration for the two PCC settings of 'study I'.

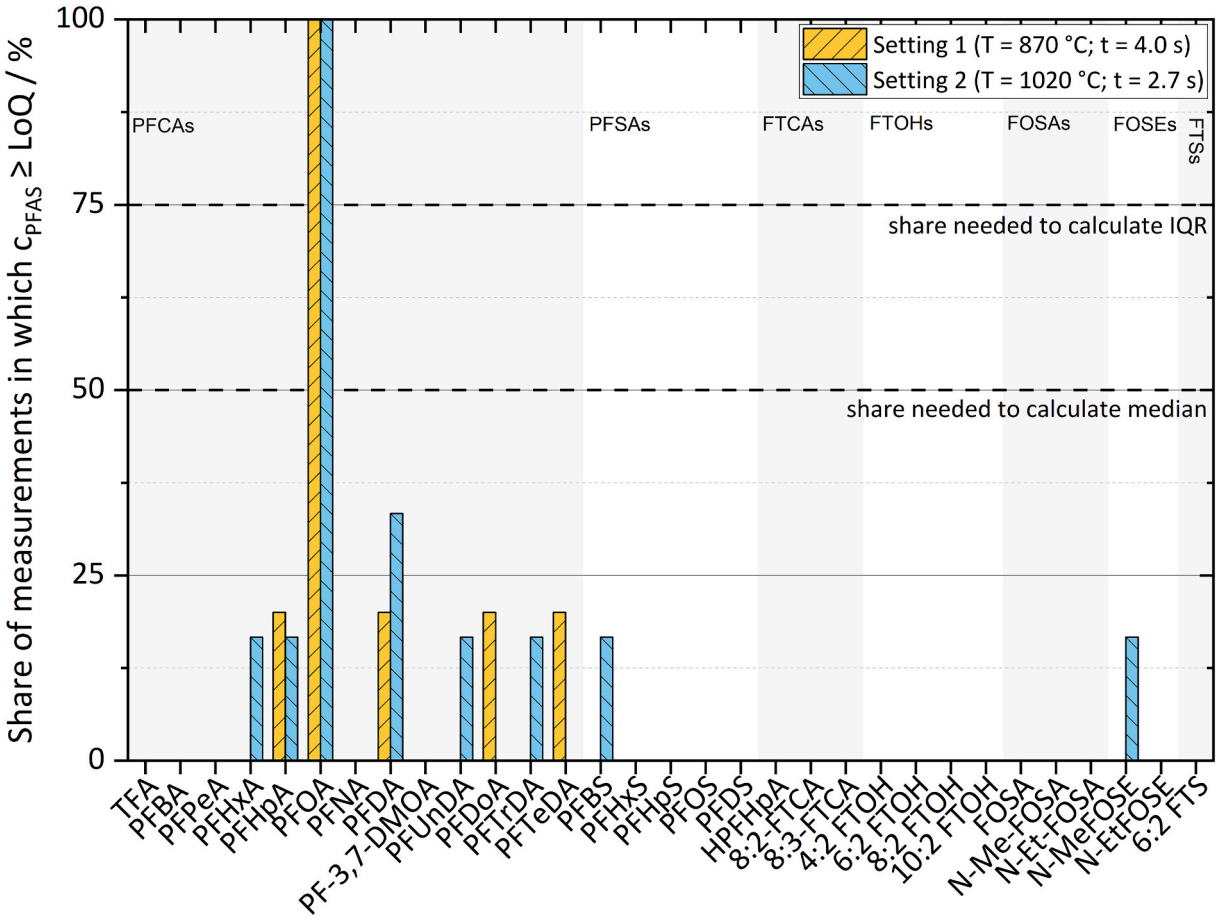

Fig. 2. Share of quantified PFAS for both PCC settings of the 'study I'. Dotted lines indicate the minimum shares required to calculate the median value and the IQR.

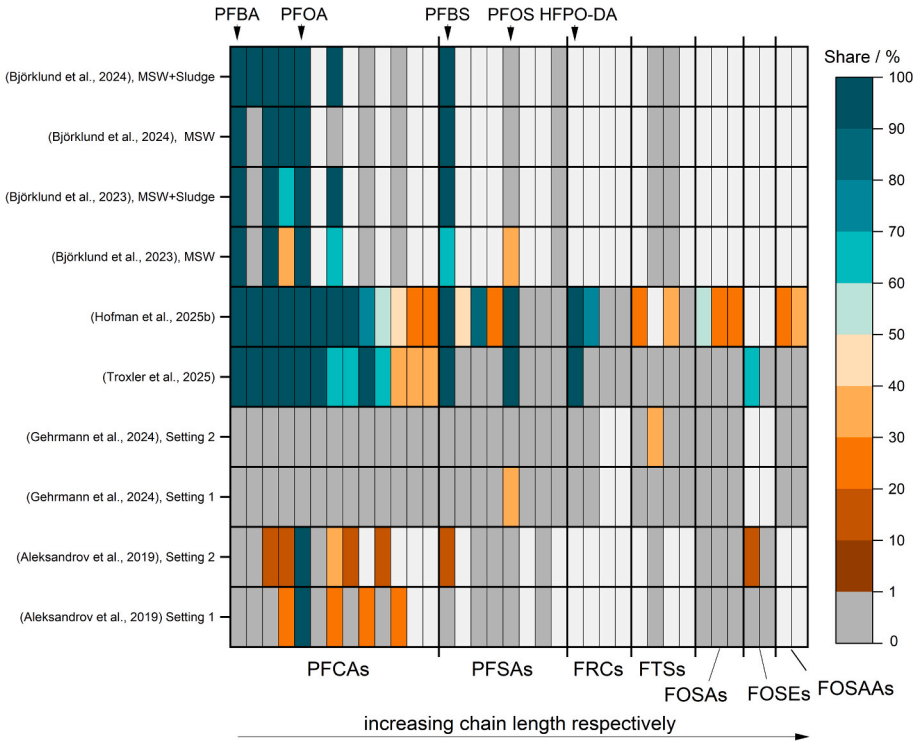

Fig. 3. Share of quantified PFAS (OTM-45 targets) across published incineration studies. Light grey fields indicate substances where not included in the analytical scope.

The limited number of quantifiable PFAS further raises questions regarding the composition of current analytical target lists. Although OTM-45 and related methods provide broad analytical coverage, combustion studies repeatedly report detectable concentrations for only a small subset of targeted analytes. This observation suggests that factors beyond ND-substitution, particularly target-list composition and analyte-specific LoQs, may substantially influence PFAS-derived fluorine metrics. These factors are therefore examined in more detail in the following sections.

5.2. Apparent influence of ND substitution on fluorine metrics

The influence of ND-substitution approaches on PFAS-derived fluorine concentrations and corresponding R2PIC values was first evaluated using the original datasets from both studies.

The effect of ND treatment and the choice of selected descriptive statistical parameter on PFAS-derived fluorine concentrations and R2PIC values is illustrated in Fig. 4 for both PCC settings of 'study I'.

When all NDs are substituted with 'zero' (Approach 1), calculated PFAS-derived fluorine concentrations reflect only quantifiable analytes and the resulting R2PIC values approach 100.000 %, irrespective of the descriptive statistical parameter applied.

As the substitution value increases from 0 to LoQ/2, LoQ/$\sqrt{2}$, and finally the full LoQ, calculated PFAS-derived fluorine concentrations increase substantially and corresponding R2PIC values decrease. Depending on PCC setting, ND-substitution approach and descriptive statistical parameter, R2PIC values range from approximately 99.807 % to 100.000 %.

For Setting 1, the maximum difference among substitution approaches is approximately 0.167 %-points, while for Setting 2 differences approach 0.193 %-points. By comparison, differences between minimum and maximum values within individual substitution approaches are smaller and generally remain below approximately 0.08 %-points. These findings suggest that, when current analytical target lists are evaluated, the choice of ND-substitution approach appears to exert a stronger influence on calculated fluorine metrics than the selected descriptive statistical parameter.

A similar pattern is observed in 'study II' (Fig. 5). When NDs are substituted with 'zero', fluorine concentrations remain in the low $ng_{F,PFAS}/m^3_{N,dry}$ range and the R2PIC reaches 100.00 %. Increasing the surrogate concentration assigned to NDs results in progressively higher fluorine concentrations and correspondingly lower R2PIC values.

For setting 1, R2PIC values ranged between 99.840 % and 100.000 %, corresponding to maximum differences of 0.160 %-points among substitution approaches. For setting 2, the maximum differences are 0.129 %-points. In contrast to 'study I', differences associated with the selected descriptive statistical parameter are negligible. This observation indicates that the influence of descriptive statistical parameters decreases substantially when only a few quantified observations are available and differences associated with ND treatment appear to dominate the resulting metrics.

Overall, both studies suggest that fluorine-based PFAS metrics are highly sensitive to the selected ND-substitution approach when evaluated using current analytical target lists, whereas the influence of descriptive statistical parameters appears comparatively small.

Comparable effects have been documented in other PFAS flue-gas

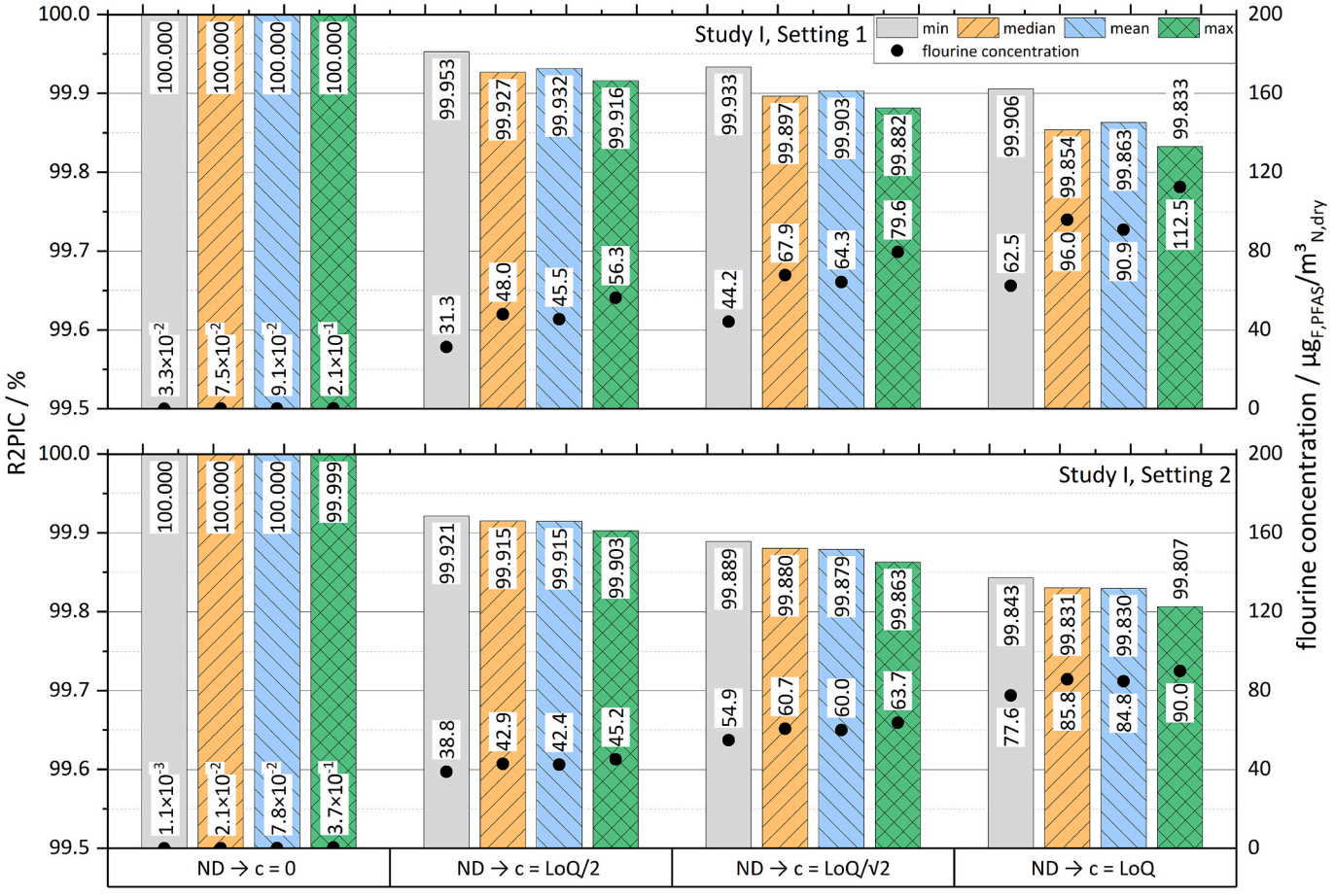

Fig. 4. Influence of ND-substitution approaches on PFAS-derived fluorine concentrations and R2PIC values based on the original datasets downstream of the boiler for 'study I' (Setting 1 top; Setting 2 bottom).

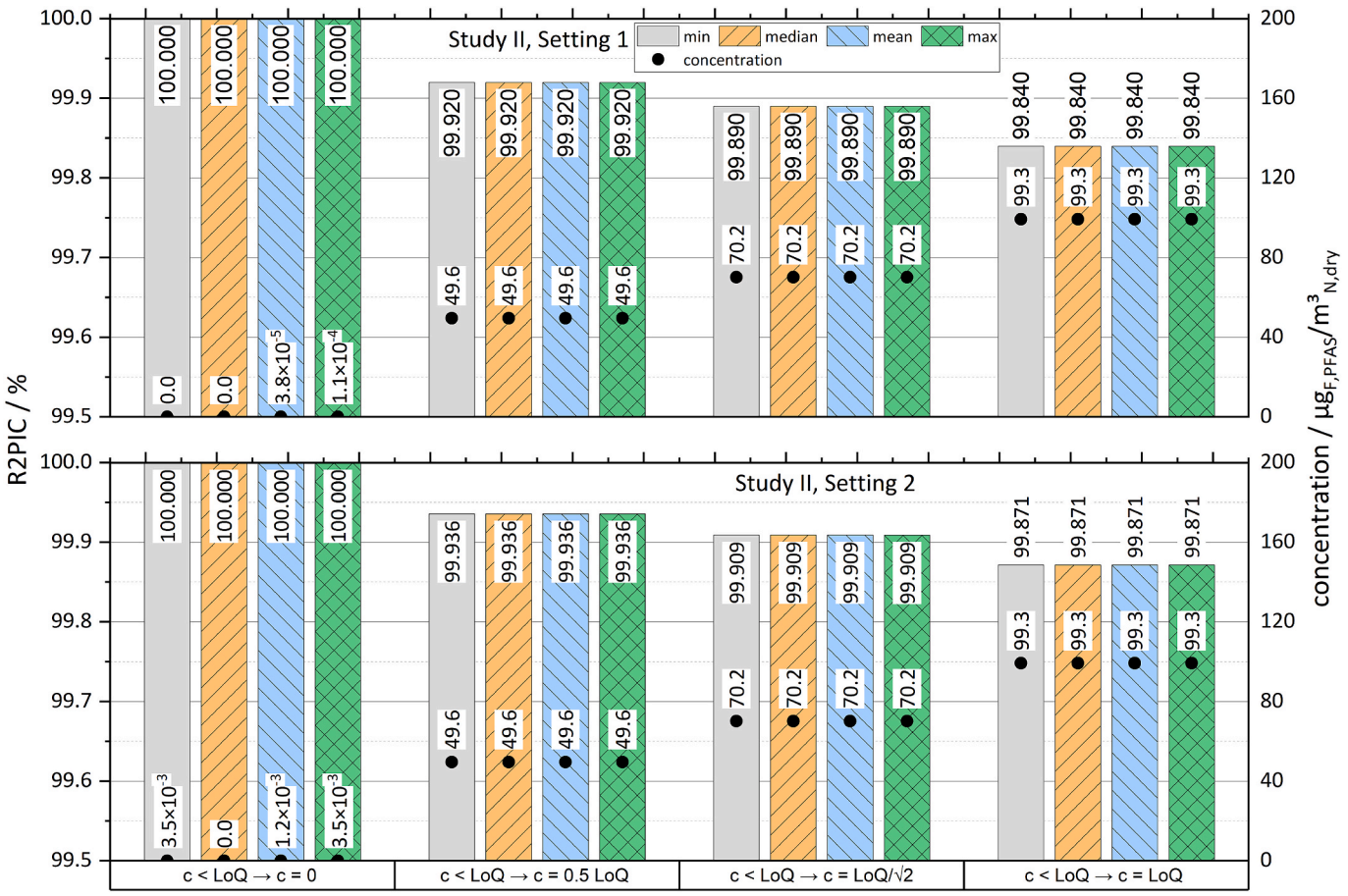

Fig. 5. Influence of ND-substitution approaches on PFAS-derived fluorine concentrations and R2PIC values based on the original datasets downstream of the boiler for 'study II' (Setting 1 top; Setting 2 bottom).

investigations. In one study, the summed concentration of 45 OTM-45 PFAS nearly doubled from 21–30 ng/Nm^3(dry) to 51–55 ng/Nm^3(dry) when NDs were substituted using LoQ/2 rather than 'zero' (Hofman et al., 2023). In another study, DE and DRE differed in the second or third decimal place when NDs in process streams were substituted with 'zero' or MDLs (Troxler et al., 2025).

Although numerically small, such deviations may nevertheless become relevant when evaluating performance. Under stringent performance criteria, differences arising from methodological choices may affect the interpretation of calculated PFAS-derived fluorine metrics despite occurring only in the second or third decimal place.

At the same time, interpretation of these observations requires caution. Both datasets contain high shares of NDs, analyte-specific LoQs spanning multiple orders of magnitude, and analytical target lists dominated by compounds that are rarely or never quantified. Consequently, the apparent sensitivity to ND treatment observed here may not originate solely from the substitution approaches themselves but may instead reflect characteristics of the underlying analytical datasets.

Several alternative explanations are possible. First, a small number of analytes exhibiting disproportionately elevated LoQs may dominate calculated fluorine concentrations. Second, the composition of the analytical target list itself may amplify the influence of ND treatment if many analytes are consistently reported as NDs. Third, because R2PIC is calculated from aggregate fluorine concentrations, observed sensitivities may differ from those expected for substance-specific metrics such as DE and DRE.

The following sections therefore examine whether the apparent influence of ND treatment is primarily a consequence of the substitution approaches themselves or whether it is driven by analyte-specific LoQ distributions and target-list composition.

5.3. *Influence of analytes exhibiting elevated LoQs*

To investigate the factors underlying the apparent sensitivity of PFAS-derived fluorine metrics to ND treatment, the contribution of individual analytes to the calculated fluorine concentrations was assessed. Particular emphasis was placed on analytes exhibiting elevated LoQs.

The influence of elevated-LoQ analytes is illustrated in Fig. 6 for Setting 1 of 'study I'. Seven of 31 analytes exhibit elevated LoQs and collectively account for approximately 95 % of the calculated PFAS-derived fluorine concentration. With more than 84 %, the calculated fluorine concentration is dominated by the four FTOHs, whose LoQs are up to two orders of magnitude higher than most other PFAS. Three additional short-chain substances with elevated LoQs (TFA, PFBA and PHFHpA) account for approximately 11 %.

This effect is even more pronounced in 'study II' (Fig. 7). Six PFCs, all reported as NDs, exhibit LoQs approximately three orders of magnitude higher than those of the remaining target analytes. Consequently, these six compounds alone contribute approximately 99.9 % of the calculated PFAS-derived fluorine concentration when substitution approaches were applied (left panel). The quantified PFAS therefore contributed only marginally to the calculated fluorine concentrations and corresponding R2PIC values.

For the remaining PFAS in 'study II' (middle and right panel), short-chain PFCAs with elevated LoQs dominated the fluorine contribution. TFA and PFBA together account for approximately 94 % of this PFAS

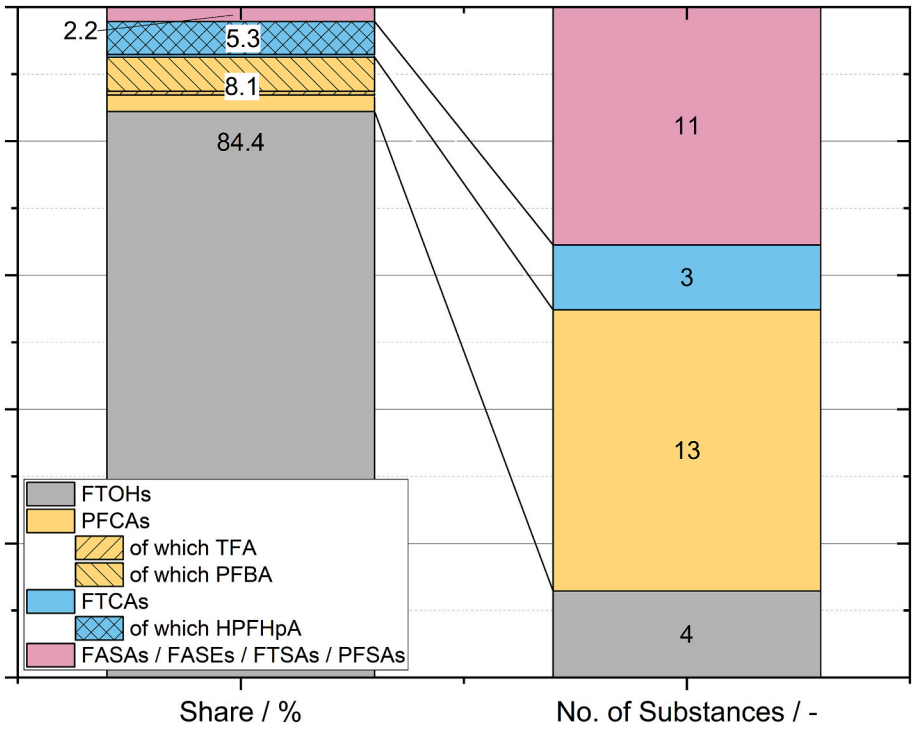

Fig. 6. Share of different PFAS on the calculated PFAS-derived fluorine concentration including the corresponding number of substances for PCC setting 1 of 'study I'.

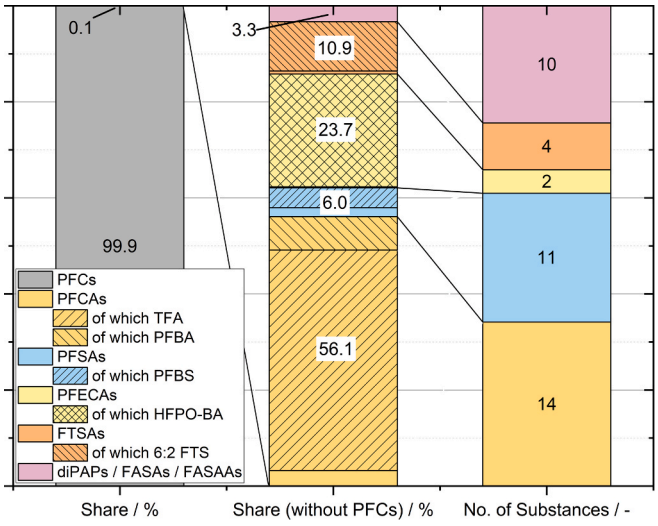

Fig. 7. Share of different PFAS on the calculated PFAS-derived fluorine concentration including the corresponding number of substances for PCC setting 2 of 'study II'.

class. Similar patterns are observed across PFSAs, PFECAs and FTSAs, where individual analytes account for 69 – 99 % within each class.

In all cases, the dominant contributors correspond to analytes exhibiting substantially elevated LoQs compared to the other analytes, typically by factors of 20 – 30 and in some cases by more than two orders of magnitude.

Comparable analytical challenges have been reported in other PFAS combustion studies. In a recent industrial-scale intercomparison, semi-volatile PFAS measured using OTM-45 or EN 1948-1 consistently appeared below about 1 ng/m^3_N, whereas volatile fluorinated species targeted by OTM-50 occurred at concentrations between 100 and 1000 µg/m^3_N, corresponding to differences of six to nine orders of magnitude in method-specific sensitivity (Strandberg et al., 2025). Similar variability was observed in Belgian OTM-45 applications, where many PFAS occurred exclusively as NDs due to method- and matrix-dependent limitations (Hofman et al., 2025b).

A review of published PFAS combustion studies further confirms that heterogeneous analyte-specific LoQs are common. Reported LoQ ratios range from approximately 2 to 1000 within individual analytical methods and target lists (Appendix A: Supplementary information, Table SI 1). Elevated reporting limits (LoD and LoQ) have been observed particularly for long-chain PFCAs, PFSAs and diPAPs (Björklund et al., 2024, 2023), while other studies document high LoQs across a broad range of PFAS, including long-chain FSAs, various PFCAs and multiple precursor compounds (Sørmo et al., 2023). These differences cannot be attributed solely to PFAS class or chain length but arise from matrix effects, adsorption behavior, ionization efficiency, method performance and inter-laboratory variability (Hofman et al., 2025a; Strandberg et al., 2025).

The observed LoQ-effects are further amplified by the matrix-dependent distribution of PFAS within the OTM-45 sampling train. OTM-45 generates four distinct extract fractions, reflecting heterogeneous partitioning of PFAS according to volatility, polarity and adsorption behavior (Berkowitz et al., 2025). Such matrix-dependent PFAS retention has been observed in both pilot and industrial scale combustion studies (Aleksandrov et al., 2019; Hofman et al., 2025b; Strandberg et al., 2025; Troxler et al., 2025) and contributes to substantial analyte-specific differences in analytical sensitivity.

Overall, these results demonstrate that a small number of analytes exhibiting disproportionately elevated LoQs can dominate PFAS-derived fluorine concentrations and thereby strongly influence fluorine-based metrics. Consequently, the apparent sensitivity of PFAS-derived fluorine metrics to ND treatment cannot be evaluated independently of the underlying LoQ distributions and may largely reflect the influence of elevated-LoQ analytes rather than the substitution approach itself.

5.4. Sensitivity analysis: Elevated LoQs versus target-list composition

To distinguish between the apparent influence of ND-substitution approaches and the potential effects of analyte-specific LoQ distributions and target-list composition, a series of sensitivity analyses was performed for both studies. Detailed information on the calculations can be found in Appendix A: Supplementary information, Chapter 5.4.

In 'study I', removal of the seven analytes exhibiting elevated LoQs reduces variability among ND-substitution approaches by approximately 95 % across both PCC settings (Fig. 8). For Setting 1, variability decreases from 0.094 to 0.167 %-points in the original dataset to 0.005–0.008 %-points after exclusion of the elevated-LoQ analytes. Similar reductions can be observed for Setting 2, where variability decreases from 0.157 to 0.193 %-points to 0.008–0.010 %-points (Appendix A: Supplementary information, Table SI 2).

Restricting the dataset to short-chain PFCAs and PFSAs also substantially reduces variability. However, the resulting reduction (approximately 93 %) is only marginally smaller than that achieved by removing the elevated-LoQ analytes alone. This suggests that most of the apparent target-list effect originates from a small number of analytes exhibiting elevated LoQs rather than from the analytical scope as a whole.

An even more pronounced pattern is observed in 'study II' (Fig. 9). The original dataset exhibits variability of approximately 0.169 %-points irrespective of the descriptive statistical parameter used. Removal of the six PFCs reduces variability by approximately 99.96 %, leaving only 0.00007–0.00008 %-points. Additional exclusion of the remaining elevated-LoQ analytes reduces variability further to approximately 0.00001 %-points, corresponding to only 0.01 % of the original variability. Subsequent restriction of the target list produces virtually no further reduction. Comparable results are obtained for Setting 2 (Appendix A: Supplementary information, Table SI 3).

Importantly, the reductions observed in both studies are nearly identical for all descriptive statistical parameters. The sensitivity analyses therefore affect calculated fluorine metrics largely independently of the selected descriptive statistical parameter.

The magnitude of observed reductions in variability closely reflects the contribution of the excluded analytes to the calculated PFAS-derived fluorine concentrations described in the previous section. Because R2PIC is calculated directly from aggregate PFAS-derived fluorine concentrations, the contribution of an analyte to the substitution-derived fluorine load is directly reflected in its influence on the resulting R2PIC variability. Consequently, analytes accounting for approximately 95 % and 99.9 % of the calculated fluorine concentrations in 'study I' and 'study II', respectively, also explain most of the apparent sensitivity to ND-substitution approaches.

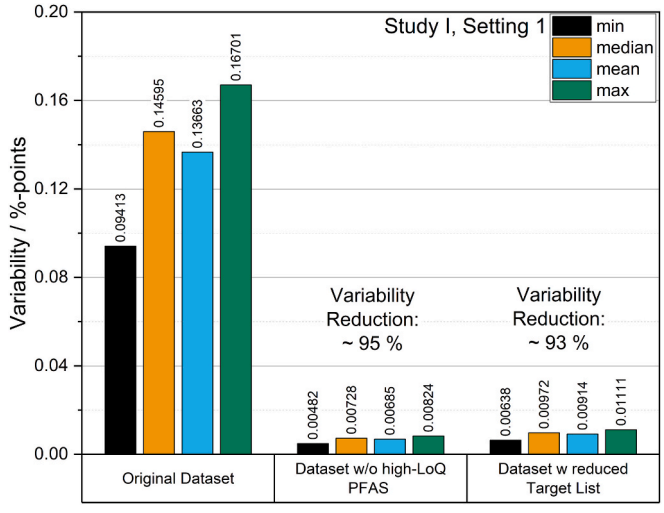

Fig. 8. Variability of R2PIC among ND-substitution approaches for the original dataset and successive sensitivity analyses (Study I, Setting 1).

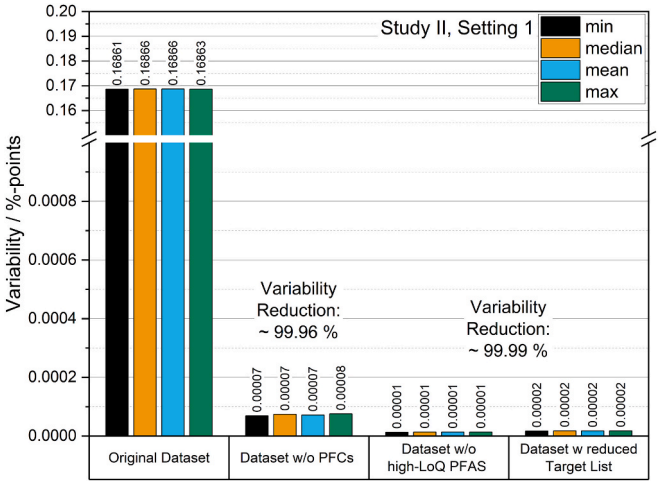

Fig. 9. Variability of R2PIC among ND-substitution approaches for the original dataset and successive sensitivity analyses (Study II, Setting 1).

Overall, these results demonstrate that the apparent variability of R2PIC is governed primarily by a small number of analytes exhibiting elevated LoQs rather than by the ND-substitution approach itself. Likewise, the apparent influence of target-list composition largely reflects the inclusion of analytes associated with disproportionately high LoQs. Analytical target lists containing such compounds inherently amplify the effect of ND substitution because they dominate substitution-derived fluorine concentrations despite never being quantified.

5.5. Implications for PFAS destruction metrics and PFAS emission assessment

The sensitivity analyses demonstrates that the apparent influence of ND-substitution approaches on PFAS-derived fluorine metrics is largely attributable to a small number of analytes exhibiting disproportionately elevated LoQs. Once these analytes are removed, variability among substitution approaches decreases by approximately 95 % in 'study I' and by more than 99.9 % in 'study II'. Together with the wide range of analyte-specific LoQs reported in the literature, these findings indicate that elevated-LoQ analytes represent a major methodological driver of PFAS-derived fluorine metrics.

This has important implications for the interpretation of PFAS destruction metrics. While DE and DRE describe the reduction of individual PFAS, R2PIC reflects the reduction of PFAS-derived fluorine associated with analytically targeted PICs. Consequently, none of these metrics should be interpreted as a direct measure of complete fluorine mineralization. Furthermore, because DE and DRE are calculated for individual analytes, they may be more sensitive to outliers, small sample sizes, and ND treatment than fluorine-based aggregate metrics such as R2PIC. Aggregation of multiple PFAS into a fluorine-equivalent concentration reduces the influence of individual observations, particularly when no single analyte dominates the fluorine inventory. Consequently, descriptive statistical parameters may exert a greater influence on DE and DRE than observed for R2PIC in the present study.

The observed sensitivity of PFAS-derived fluorine metrics has direct implications for regulatory interpretation. Destruction metrics are frequently reported with four or more decimal places (Ministry of the Environment of Japan, 2013; Shields et al., 2023; Troxler et al., 2025; van Caneghem et al., 2025) and are often compared against stringent performance criteria (Basel Convention, 2025). As demonstrated in the present study, differences occurring in the second or third decimal place may be governed primarily by analytical factors, including analyte-

specific LoQ distributions, target-list composition, and the treatment of NDs, rather than by actual differences in PFAS destruction performance. Consequently, apparently small methodological differences may influence the interpretation of compliance-related results.

For this reason, transparent ND-handling is essential. The toxicological practice of reporting lower- and upper-bound estimates (Barone et al., 2021; Houlihan et al., 2021; Knutsen et al., 2018) provides a transparent framework for representing uncertainty associated with NDs and should be considered for PFAS emission assessments.

The results further highlight the importance of distinguishing between the LoD and the LoQ. Analytes reported below the LoD differ fundamentally from analytes detected between the LoD and LoQ. While the latter provide analytical information and may justify estimation or substitution procedures, assigning non-zero concentrations to compounds consistently reported below the LoD may artificially inflate PFAS-derived fluorine concentrations and metrics. Future studies should therefore report both LoDs and LoQs whenever possible and clearly distinguish between non-detected and non-quantified PFAS.

Finally, the present findings have implications for future method development. The strong influence of a small number of elevated-LoQ analytes suggests that analytical target lists should be evaluated not only with respect to analytical coverage but also to analytical sensitivity and practical relevance. Priority should be given to analytes that are repeatedly observed in combustion studies, while substances consistently reported below detection or quantification limits warrant critical review. Future PFAS emission assessments should place greater emphasis on volatile PFAS and fluorinated combustion products covered by complementary methods such as OTM-50 (Shields et al., 2023; Strandberg et al., 2025; Troxler et al., 2025). In addition, NTA should be increasingly integrated to improve the characterization of unidentified fluorinated transformation products and to support more comprehensive fluorine-based assessments (Baqar et al., 2025; Reis de Carvalho et al., 2026; Shields et al., 2023).

Overall, the results demonstrate that robust interpretation of PFAS destruction metrics requires transparent reporting of analyte-specific LoQ distributions and explicit treatment of NDs. Without such information, apparent differences in PFAS-derived metrics may reflect methodological artefacts rather than actual differences in PFAS destruction performance.

6. Conclusion

This study demonstrates that the calculation and interpretation of PFAS emissions from combustion systems are profoundly influenced by the treatment of NDs, analyte-specific LoQs, and analytical target-list composition using data from two pilot-scale fluoropolymer combustion studies.

Initial evaluation of the original datasets suggests a strong influence of ND-substitution approaches on PFAS-derived fluorine concentrations and R2PIC values. However, sensitivity analyses demonstrates that this apparent sensitivity is largely attributable to a small number of analytes exhibiting disproportionately elevated LoQs. In ‘study I’, removal of seven elevated-LoQ analytes reduced variability among substitution approaches by approximately 95 %, while restricting the dataset to a reduced target list decreased variability by approximately 93 %. In ‘study II’, six PFCs alone accounted for almost the entire observed variability, and their exclusion reduced variability by approximately 99.96 %. Similar reductions were observed for minimum, median, mean, and maximum values, indicating that the observed effects were largely independent of the selected descriptive statistical parameter.

The results highlight important limitations associated with the interpretation of PFAS destruction metrics. Although values such as R2PIC, DE, and DRE are frequently reported with four to six decimal places, the underlying analytical uncertainty associated with sampling, matrix effects, LoQs, ND treatment, and temporal process variability often exceeds the implied precision, particularly under industrial operating conditions. Consequently, differences in the fourth, fifth, or sixth decimal place should be interpreted primarily as mathematical outcomes of data processing rather than as directly measurable differences in PFAS destruction performance.

To improve robustness, transparency and comparability, PFAS-derived fluorine concentrations and destruction metrics should be reported as lower- and upper-bound estimates rather than as single numerical values with excessive apparent precision. This should be accompanied by explicit reporting of both LoDs and LoQs and by a clear distinction between analytes detected below the LoQ and analytes consistently reported below the LoD.

Future method development should prioritize harmonization and reduction of analyte-specific LoQs, critical evaluation of analytes exhibiting elevated LoQs, and refinement of target lists toward repeatedly observed and analytically relevant PFAS. Particular emphasis should be placed on short-chain and volatile fluorinated compounds, including those targeted by OTM-50, while non-target analysis should be increasingly integrated to capture transformation products and unidentified fluorinated species not covered by existing target methods.

Overall, this study demonstrates that analyte-specific LoQ distributions represent a major methodological driver of PFAS-derived fluorine metrics. Without transparent handling of NDs, harmonized analytical sensitivity, and uncertainty-aware reporting, PFAS destruction metrics risk reflecting methodological artefacts arising from analytical scope and data-processing assumptions rather than actual differences in PFAS destruction performance.

CRediT authorship contribution statement

Manuela Wexler: Writing – review & editing, Writing – original draft, Visualization, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Vanessa Nuredin:** Writing – review & editing, Visualization, Investigation, Formal analysis, Data curation, Conceptualization. **Hans-Joachim Gehrmann:** Writing – review & editing, Supervision, Project administration, Investigation, Conceptualization. **Dieter Stapf:** Writing – review & editing, Supervision, Project administration.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.wasman.2026.115836.

Data availability

Data are available in Chemosphere (https://doi.org/10.1016/j.chemosphere.2019.03.191; https://doi.org/10.1016/j.chemosphere.2024.143403)

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