

## SHORT COMMUNICATION OPEN ACCESS

# Thermal Degradation of PFAS in Different Scales: Measurement of Hydrogen Fluoride (HF)

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## ABSTRACT

Approximately 13% of the waste generated in Germany (2023) is processed in municipal solid waste incinerators (MSWI). MSWI in Europe operate under strict regulatory requirements defined by the Industrial Emissions Directive (IED), resulting in very low overall emissions, including PCDD/F. A more recent and widely discussed group of pollutants are per- and polyfluoroalkyl substances (PFAS), which are thermally degraded to hydrogen fluoride (HF), carbon dioxide (CO<sub>2</sub>) and other thermodynamic end point compounds depending on elemental composition under complete combustion conditions. This study investigates the completeness of PFAS degradation in laboratory- and pilot-scale incinerators. The conversion of PFAS to HF is assessed using several analytical techniques for detecting fluorine-containing species in flue gas, liquids and solids, and their impact on establishing a consistent fluorine balance (F-balance). Depending on the analytical method employed, the achieved F-balance ranges from 59 to 100 wt% when ‘products of incomplete combustion’ (PICs) are not considered.

## 1 | Introduction

Per- and polyfluoroalkyl substances (PFAS) comprise a group of more than 14 000 synthetic organic compounds [1]. According to the Organisation for Economic Co-operation and Development (OECD) definition, PFAS contain at least one fully fluorinated methyl or methylene group [2]. A well-known example of a polymeric PFAS is polytetrafluoroethylene (PTFE). In recent years, however, research has increasingly focused on lower molecular-weight PFAS, such as perfluorooctanoic acid (PFOA) and perfluorooctane sulfonic acid (PFOS) [3–5]. PFAS have been used extensively across industrial sectors and consumer products

due to their water-, oil- and dirt-repellent behaviour and their exceptional chemical and thermal stability. At the same time, their persistence in the environment and in biological systems leads to bioaccumulation, meaning that several PFAS are now recognized as potentially harmful to both human health and the environment [6–8].

The effectiveness of thermal waste treatment for degrading PFAS has been demonstrated in several studies [9–12]. Under sufficiently high temperatures and appropriate combustion conditions, complete destruction occurs, resulting in negligible PFAS emissions [10, 13]. Nevertheless, the underlying degradation

**Abbreviations:** BRENDA, Brennkammer mit Dampfkessel (Engl. combustion chamber with steam boiler); FTIR, Fourier-transform infrared; HF, hydrogen fluoride; IC, ion chromatography; IED, Industrial Emissions Directive; ISE, ion selective electrode; ITC, Institute of Technical Chemistry; KLEAA, Karlsruher Laboranlage zur Ermittlung des Abbrandverhaltens von Abfällen (Engl. Karlsruhe laboratory plant for determination of the combustion behaviour of waste); LCV, low calorific value; LoQ, limit of quantification, quantification limit; OECD, Organisation for Economic Co-operation and Development; PCC, post-combustion chamber; PFAS, per- and polyfluoroalkyl substances; PFOA, perfluorooctanoic acid; PFOS, perfluorooctane sulfonic acid; PTFE, polytetrafluoroethylene; PIC, products of incomplete combustion; TDLS, tunable diode laser spectroscopy; WCs, wood chips.

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pathways and their completeness are not yet fully understood and require further investigation, as highlighted in a recent review by Wang et al. [14]. Furthermore, hydrogen fluoride (HF)—although historically considered a primary indicator of PFAS mineralization—is now rarely used as such. However, HF measurement and fluorine mass balancing remain valuable tools for assessing PFAS conversion.

Both HF and certain short-chain PFAS can be detected by infrared spectroscopy due to their (induced) dipole moments. Fourier-transform infrared (FTIR) spectroscopy is widely used for qualitative and quantitative assessments [15]. In combustion systems, however, the high water content of the flue gas and the presence of interfering species substantially elevate the limits of detection and quantification. Tunable diode laser spectroscopy (TDLS), in contrast, is less susceptible to cross-sensitivities but typically requires greater installation effort.

The aim of this study is to compare two analytical techniques—FTIR and TDLS—for HF determination in flue gases. To support the establishment of fluorine mass balances, fluorides in aqueous samples are analysed by ion chromatography (IC) and photometry, whereas solid residues are characterized using pyrohydrolysis.

## 2 | Materials and Methods

Two pilot-scale incineration facilities were used to compare the analytical approaches for fluorine determination. The Brennkammer mit Dampfkessel (BRENDA) facility [11,12], which consists of a rotary kiln followed by a post-combustion chamber, is equipped with a complete flue-gas cleaning train, including a spray dryer, fabric filter, acidic and neutral scrubbers, and selective catalytic reduction for  $\text{NO}_x$  removal in accordance with the Industrial Emissions Directive (IED) requirements [16]. In BRENDA, wood chips (WCs) were mixed up with a fluoropolymer mixture with a fluorine content of 0.3 wt%.

To simulate waste combustion under conditions comparable to a grate-fired system, a fixed-bed reactor, Karlsruher Laboranlage zur Ermittlung des Abbrandverhaltens von Abfällen (KLEAA), was employed. Although the BRENDA plant has been described in detail in the referenced publications, the focus of the present study lies on the fixed-bed reactor KLEAA and the applied analytical methodologies. In KLEAA PFOA was mixed with WCs.

### 2.1 | Fixed-Bed Reactor KLEAA

The fixed-bed reactor KLEAA has a fuel bed volume of 10 L and is electrically heated to ensure an isothermal combustion chamber. The combustion behaviour can simulate the burnout characteristics of municipal solid waste incinerators (MSWI) based on grate technology [17,18]. In addition to the fixed bed, the KLEAA reactor system comprises a combustion chamber, a post-combustion chamber (PCC), and a downstream flue-gas cleaning section (Figure 1).

After the fuel is placed into the fuel pot, the combustion chamber is positioned beneath the electrically heated furnace. Depending on the fuel composition and the process conditions, the release of volatile components begins once the fuel surface has been sufficiently heated. These volatiles ignite in the presence of the primary air supply, initiating combustion (Phase I, defined as the ignition phase). Between the ignition phase and the subsequent char burnout stage (Phase III), a quasi-stationary combustion zone (Phase II) typically develops, during which the fuel mass decreases at an approximately uniform rate. Detailed descriptions of the combustion characteristics in the KLEAA system can be found in [17,18].

The KLEAA reactor can be operated in compliance with the requirements of the IED 2010/75/EU [16], including minimum residence time and temperature specifications for the PCC.

### 2.2 | Fluorine Measurement

During combustion processes, fluorine is predominantly bound in chemically stable forms, such as calcium fluoride (fluorspar) or as hydrogen fluoride (HF). When HF is captured in aqueous scrubbers, it is converted into fluoride ions. Depending on the specific fluorine species present, different analytical techniques are required for accurate quantification.

In the flue gas, hydrogen fluoride can be quantified by FTIR spectroscopy due to its permanent dipole moment. Alternatively, HF can be measured using TDLS. Both FTIR and TDLS are molecular spectroscopic methods; however, their operating principles and spectral selectivities differ significantly.

The FTIR system used in this study (CX 400, Gasetm Technologies GmbH) covers a wavenumber range of 90–4250  $\text{cm}^{-1}$ , enabling simultaneous detection of a wide range of flue-gas components. In contrast, the TDLS (GM700-2 laser gas analyser, Endress+Hauser) is configured to monitor HF absorption at a specific wavenumber of 7825  $\text{cm}^{-1}$ . As a result, TDLS offers substantially higher selectivity and is considerably less prone to cross-sensitivities from other gas constituents. The limit of quantification (LoQ) for HF with TDLS is 25  $\text{mg m}^{-3}$ , and the method is not affected by high water vapour concentrations, unlike FTIR.

FTIR, however, provides the advantage of detecting certain PFAS (e.g.,  $\text{CF}_4$ ) and other products of incomplete combustion (PICs), such as  $\text{COF}_2$ . Nevertheless, FTIR measurements may be influenced by spectral interferences from overlapping absorption bands of other compounds. For HF determination, the FTIR exhibits an LoQ of 1.5  $\text{mg m}^{-3}$ . In the KLEAA system, the FTIR was connected to the PCC via a heated sampling line equipped with a filter, enabling continuous monitoring of typical flue-gas constituents such as  $\text{H}_2\text{O}$ ,  $\text{CO}_2$ ,  $\text{CO}$ ,  $\text{NO}_x$ ,  $\text{SO}_2$  and HF. Due to this integrated filter, FTIR is less susceptible to particle interference compared to TDLS. However, if calcium-containing dust particles accumulate on the filter, they may bind HF (as a hypothesis) from the gas and thus reduce the measurement result.

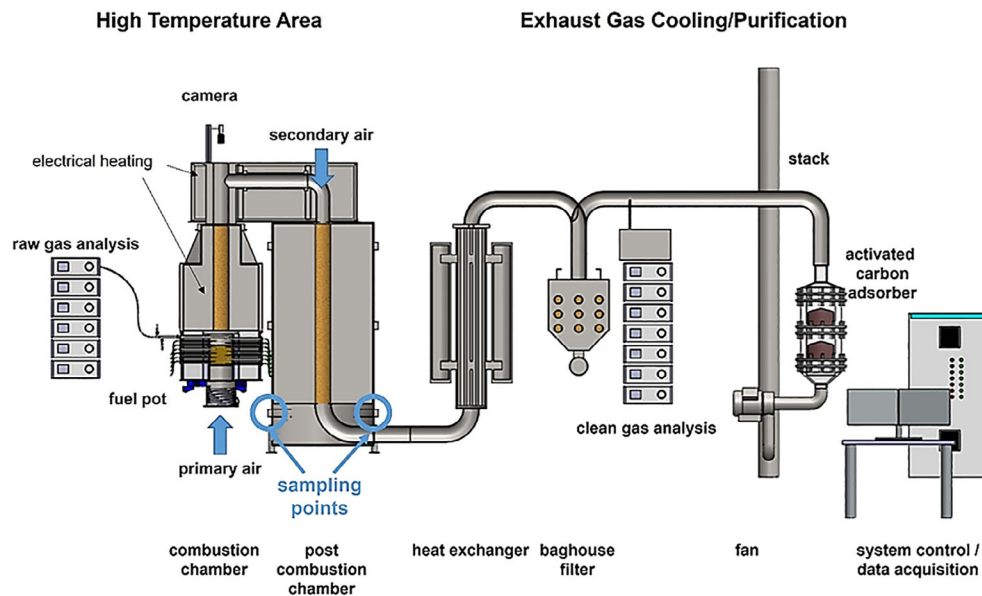
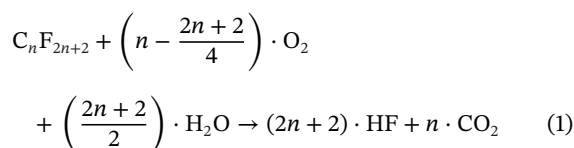


FIGURE 1 | Scheme of KLEAA fixed-bed reactor.

For complete combustion of polymeric or non-polymeric PFAS, the following reaction equation is applicable:



The stoichiometric reaction equation varies depending on the specific PFAS subgroup. Consequently, when investigating the thermal degradation of PFAS, quantifying HF in the flue gas is essential to assess the extent of fluorine mineralization.

For comparison of the fluorine content across all experiments and to enable the closure of fluorine mass balances, the mass fraction of fluorine related to the fuel,  $w_{Fuel}$ , is calculated using the following equation:

$$w_{Fuel} = \frac{m_{F,output}}{m_{Fuel}} \quad [mg \, kg_{Fuel}^{-1}] \quad (2)$$

The total mass of fluorine in the output,  $m_{F,output}$ , is determined from HF in the flue gas. The fluorine content in ashes is considered separately when including the ashes in the overall fluorine mass balance.

The fluorine recovery rate (RR) is defined as the extent to which the fluorine mass balance can be closed. The corresponding relationship is expressed in the following equation:

$$RR = \frac{m_{F,output}}{m_{F,input}} \cdot 100 \quad [\%] \quad (3)$$

The determination of inorganic fluorine in liquid samples as fluoride can be carried out using either IC [19] or a photometric method [20]. Both techniques were applied for the analysis of fluoride in scrubber water samples from the BRENDA facility.

The IC provides a measurement range for fluoride ions from  $0.5 \, mg \, L^{-1}$  up to  $150 \, mg \, L^{-1}$ . The measurement uncertainty of the instrument is between  $\pm 2$  and  $3 \, mg \, L^{-1}$  and increases with each dilution step. For photometric analysis, a rapid test was used, offering a measurement range between  $0.1$  and  $1.5 \, mg \, L^{-1}$ , which likewise requires appropriate dilution of the sample.

For both scrubbers, 24 IC measurements were conducted with dilution factors ranging from 0 (undiluted) to a maximum of 20. The average value obtained from these measurements was subsequently verified photometrically to assess whether the magnitude of the IC-derived concentrations was realistic. The corresponding datasets, along with detailed descriptions of both analytical methods, are provided in the [Supporting Information](#).

To convert fluorine species contained in solid samples into a measurable form, pyrohydrolysis was applied, followed by potentiometric acid–base titration to adjust the pH of the resulting solution. Fundamental information on the pyrohydrolysis procedure is given in [21].

## 2.3 | Fuel Specifications and Operational Parameters

The elemental composition and the low calorific value (LCV) of the WCs incinerated in the fixed-bed reactor are presented in Table 1.

The total fluorine content of the WCs was below the LoQ of the analytical method, which was  $50 \, mg \, kg^{-1}$ .

PFOA was obtained from Sigma-Aldrich Chemie GmbH (CAS No. 335-67-1) with a stated purity of 95%. For preparing mixtures of PFOA and WCs with fluorine mass fractions between 0.01 and 0.03 wt%, approximately 0.4 g of PFOA was used as the initial mass.

**TABLE 1** | Main elements of the wood chips.

| As received      | Unit                   | Wood chips (WCs) |
|------------------|------------------------|------------------|
| Water            | wt%                    | 37.3             |
| Ash              |                        | 0.7              |
| Volatile         |                        | 52.5             |
| C <sub>fix</sub> |                        | 9.4              |
| C                |                        | 31.4             |
| H                |                        | 3.7              |
| N                |                        | 0.2              |
| S                |                        | 0.013            |
| Cl               |                        | 0.007            |
| O (by diff.)     |                        | 26.6             |
| LCV              | (MJ kg <sup>-1</sup> ) | 10.7             |

Abbreviation: LCV, low calorific value.

The operating parameters for the air supply in the KLEAA reactor were kept constant at 11 m<sup>3</sup> h<sup>-1</sup> for the primary air and 13 m<sup>3</sup> h<sup>-1</sup> for the secondary air. To investigate PFAS degradation and fluorine mineralization, the temperature in the PCC was reduced from 850°C to 750°C while maintaining a constant residence time of 2 s. Each experiment was performed in triplicate to ensure statistical robustness.

### 3 | Results

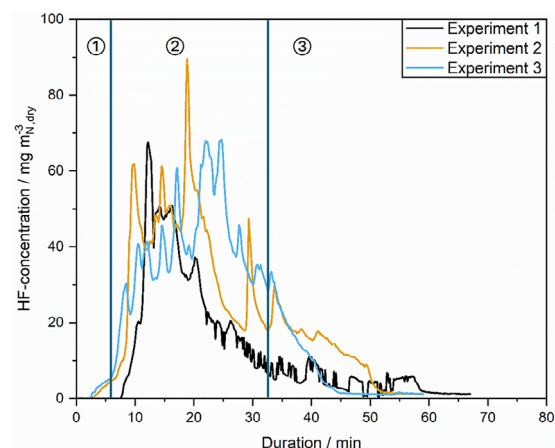
First, results obtained in the fixed-bed reactor KLEAA are discussed, followed by a comparison with the results achieved upon incineration experiment via BRENDA.

#### 3.1 | Fluorine Measurement at the Fixed-Bed Reactor KLEAA

To determine the RR of fluorine, a series of combustion tests were conducted, using pure WCs, as well as WCs mixed up with PFOA. The fluorine concentration in the mixture was calculated to be about 0.03 wt%.

Figure 2 shows the HF concentration for three combustion experiments in which WCs were mixed with PFOA. HF was measured by FTIR at the outlet of the PCC.

The combustion process can be divided into three characteristic phases. The first phase (1) corresponds to the release of volatile components: The fuel is heated by the surrounding walls of the combustion chamber (see Figure 1), leading to drying, devolatilization, and subsequent ignition of the volatiles in the presence of primary-air oxygen. The second phase (2) represents the quasi-stationary combustion zone, during which the majority of the fuel mass is converted under relatively stable conditions. The final phase (3) is the char burnout in which the remaining solid carbon is oxidized.

**FIGURE 2** | HF concentration over time in three experiments with wood chips and PFOA.

In the initial phase (1) of the experiments, only minimal HF concentrations are detected during devolatilization prior to ignition. Once ignition occurs, a substantial increase in the measured HF concentration is observed in all experiments, indicating effective conversion of fluorine from PFOA into HF. During the quasi-stationary combustion phase (2), HF release remains largely consistent, although several concentration peaks occur. These fluctuations suggest that the PFOA–WC mixture and its subsequent combustion are heterogeneous, likely due to insufficient premixing before the fuel is inserted into the fuel pot.

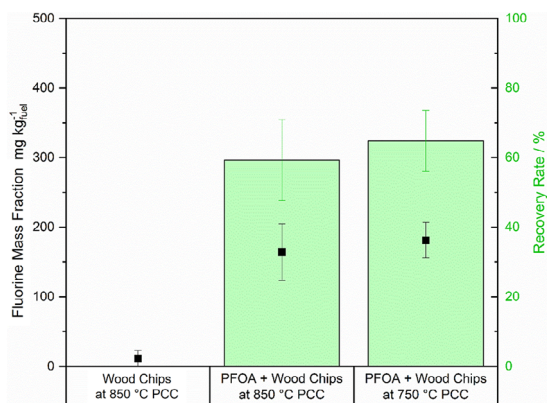
Toward the end of the experiment (Phase 3), the HF concentration gradually decreases but does not return to zero—even after more than 65 min of measurement. This behaviour reflects a ‘sponge-like’ response of the system, where HF desorbs slowly into the gas phase over an extended period. The underlying chemical mechanisms have not yet been fully clarified. This phenomenon indicates an inertial memory effect, which may generate low blank values in subsequent experiments. In the present study, at least 24 h elapsed between experiments, and no plant-related blank value was detected via FTIR prior to ignition. If a blank was present, it remained below the FTIR detection limit (1.5 mg m<sup>-3</sup>).

Additional experiments were performed to determine the intrinsic blank value of the WCs. As previously described, a fluorine-related RR was calculated for all experiments involving PFOA according to Equation (3). Figure 3 presents the calculated fluorine content and RR for each experimental configuration.

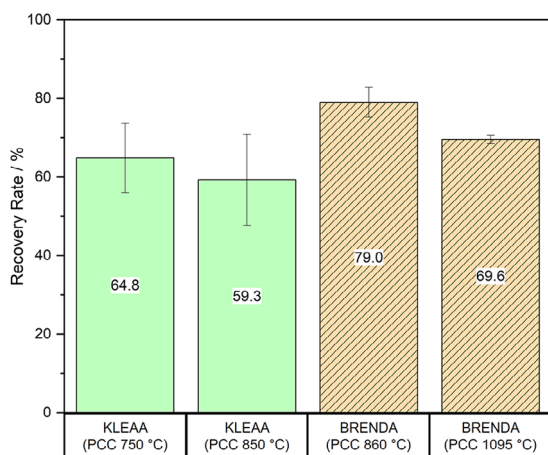
The fluorine mass fraction was calculated according to Equation (2), based on HF concentrations measured in the flue gas via FTIR. Owing to the low ash content of the WCs (0.7 wt%), the contribution of fluorine potentially bound in mineral phases, such as CaF<sub>2</sub>, was considered negligible.

It should be noted that the HF background originating from WC combustion corresponds to a low blank value of 11.4 mg kg<sup>-1</sup>. This value is in good agreement with the analytical result obtained in the laboratory (<50 mg kg<sup>-1</sup>). The relatively high standard deviation of over 100% highlights the considerable uncertainty associated with HF measurements at such low concentrations,





**FIGURE 3** | Left axis (squares): fluorine mass fraction of the fuel; right axis (bars): recovery rate (RR). PCC, post-combustion chamber; PFOA, perfluorooctanoic acid.

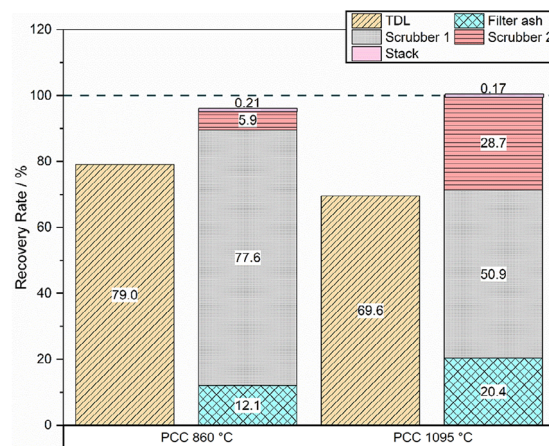


**FIGURE 4** | Fluorine recovery rates, based on HF, analysed with FTIR (KLEAA, green bars) and TDL (BRENDAA, hatched bars) each at two different temperatures 750 °C, 850 °C (KLEAA) and 860 °C, 1095 °C (BRENDAA), respectively. BRENDAA, Brennkammer mit Dampfkessel; KLEAA, Karlsruher Laboranlage zur Ermittlung des Abbrandverhaltens von Abfällen; PCC, post-combustion chamber.

which can be attributed to the proximity of the FTIR detection limit.

The experiments show that the combustion of WCs containing PFOA achieves comparable fluorine RRs at PCC temperatures of 850 °C (59.3%) and 750 °C (64.8%). The corresponding standard deviations remain below 20%. HF formation during combustion—representing the main product of complete PFAS mineralization—is largely governed by volatile release during the primary combustion phases (1–3), whereas the influence of the post-combustion stage appears comparatively minor.

These results fall within the range reported by Gehrman et al. [11] and Aleksandrov et al. [12], who observed RRs between 55.9% and 79.0% (see Figure 4). Nonetheless, the RRs determined here are somewhat lower than expected. This discrepancy can be explained by the measurement technique: FTIR is more susceptible to spectral interferences from other flue-gas species. In addition, particle separation in the upstream filter may have



**FIGURE 5** | Recovery rates analysed with TDL and F-IC, respectively, photometry at BRENDAA facility at two different temperatures: 860 °C and 1095 °C, respectively. PCC, post-combustion chamber; TDL, Tunable diode laser spectroscopy.

contributed to lower measured HF levels, as adsorption of hydrogen fluoride onto retained particles would reduce the amount available for detection via FTIR.

### 3.2 | Evaluation of Inorganic Fluorine in All Output Phases of BRENDAA

For HF measurements, the TDLS was applied in the pilot-scale downstream of the boiler. Figure 4 compares the RRs from FTIR measurements in KLEAA with TDL in BRENDAA.

This indicates that the two plants have comparable fluorine RRs. For the gas phase, online spectroscopic analysis using FTIR or TDL appears to be an appropriate method. Additionally, analysing liquid and solid residues enabled closing the fluorine mass balance up to 100% (see Figure 5).

The TDL is installed at BRENDAA downstream of the boiler and upstream of the flue gas treatment system, which comprises a spray dryer, a fabric filter and two scrubbers. As shown in Figure 4, this set-up allows determination of the majority of the fluorine introduced with the PFAS (first bar), but not 100%.

Therefore, an analysis of the filter ash and scrubber water downstream of the TDL measurement is advisable in order to achieve a more complete fluorine mass balance (second bar in Figure 5).

The left bar in Figure 5 represents the online TDL measurement, whereas the right stacked bar shows the corresponding analysis of the solid and liquid residues, including the flue-gas measurement at the stack. HF in the stack gas was quantified via IC on the basis of a 24-h sampling period in which HF was absorbed in water.

The fluorine concentrations in the scrubber solutions were determined twice by IC and once by photometry to validate the analytical consistency. Table S2 demonstrates good agreement between IC and photometric data for the ‘PCC 860 °C’ condition. With increasing fluorine concentrations in the scrubber water,

the deviations become more pronounced, particularly at higher dilution factors. This increases uncertainty in the fluorine balance; nevertheless, RRs of up to 100% were achieved at elevated temperatures. A large fraction of inorganic fluorine is detected in the gas phase as HF. TDLS measurements are influenced by particle loading, as particle scattering reduces the detected signal. Deposits on the optical windows may further impair measurement accuracy and thus recovery.

A pronounced increase in raw-gas particulate concentration is observed when the PCC temperature is raised from 860°C to 1095°C. This effect is primarily caused by the reduced volumetric gas flow at constant residence time in the PCC, resulting in higher particulate loading both within the chamber and downstream of the boiler. Consequently, laser-based measurements show an apparently lower HF concentration because increased particle fractions attenuate the HF signal by scattering the emitted laser light.

At elevated temperatures, the fluorine content in the system increases substantially—from 12.1% to 20.4%—accompanied by a doubling of the filter-dust mass, whereas the fluorine concentration within the dust remains approximately constant. The increased quantity of dust is likely attributable to enhanced volatilization of fluoride-containing compounds such as  $\text{KHF}_2$  and  $\text{NH}_4\text{HF}_2$  in the high-temperature combustion zone, followed by re-sublimation during cooling in the boiler section.

When all liquid and solid streams are analysed, the fluorine mass balance can be fully closed. The highest fluorine fractions were found in the acid scrubber (77.6% and 50.9%). Because the scrubber waters were not replaced between experiments, hydrogen fluoride appears to have progressively shifted from the first toward the second scrubber. The fluorine content of the stack dust (0.21% and 0.17%) contributes only marginally to the total fluorine balance and RR.

It is important to note that the WCs used in KLEAA were also supplied to the BRENDA facility. The results clearly demonstrate that, even at low ash contents in the fuel, mineral-bound fluorine contributes substantially to closing the overall fluorine balance—particularly at higher combustion temperatures. This observation also explains the lower fluorine detection in the KLEAA experiments at 850°C compared to 750°C (see Figure 3, middle bar vs. right bar).

## 4 | Conclusion

HF measurements performed in the fixed-bed reactor demonstrated good agreement with the results obtained from the pilot-scale facility. Nevertheless, even for fuels with very low ash contents (e.g., <1 wt%), the mineral-bound fluorine fraction must be considered in the fluorine mass balance, as these minerals can contribute measurable amounts of fluorine.

Rapid estimates of fluorine conversion can be obtained using online techniques such as FTIR for small-scale investigations or TDLS for pilot- and full-scale operation. However, it must be kept in mind that a fully closed fluorine balance cannot be achieved with these methods alone. Water vapour, particulate matter

and dust deposits affect the optical path and can significantly influence the HF signal in both spectroscopic approaches. Therefore, verification of the fluorine balance requires complementary analyses: Fluorides in liquid streams must be quantified using IC or photometry, whereas fluorine in solid residues must be analysed via pyrohydrolysis.

A nearly closed fluorine balance does not permit conclusions regarding the concentrations or mass flows of PICs in the gas phase, wastewater or solid residues, as these occur at ng levels in flue-gas and µg levels in aqueous and solid matrices and therefore do not contribute significantly on a percentage basis.

For ash-rich fuels such as sewage sludge or municipal solid waste, thorough analysis of the solid residues is particularly important for establishing a reliable fluorine balance.

Continuous HF measurements using FTIR or TDLS nonetheless provide valuable insights into the operational behaviour of the combustion and flue-gas cleaning system—especially when memory or ‘sponge’ effects of fluorine-containing species occur within the plant. Such effects must be accounted for when interpreting the overall fluorine mass balance.

## Nomenclature

### Symbols

|          |                                    |
|----------|------------------------------------|
| C        | mass of carbon, kg                 |
| Cl       | mass of chlorine, kg               |
| F        | mass of fluorine, kg               |
| H        | mass of hydrogen, kg               |
| <i>m</i> | mass, kg                           |
| N        | mass of nitrogen, kg               |
| O        | mass of oxygen, kg                 |
| RR       | recovery rate, %                   |
| S        | mass of sulphur, kg                |
| <i>w</i> | mass fraction, mg kg <sup>-1</sup> |

### Sub- and Superscripts

|                            |                            |
|----------------------------|----------------------------|
| <i>F</i>                   | fluorine                   |
| <i>F</i> <sub>output</sub> | fluorine content in output |
| <i>F</i> <sub>input</sub>  | fluorine content in input  |
| Fuel                       | fuel                       |
| <i>N</i>                   | variable, normal           |

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## Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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## Supporting Information

Additional supporting information can be found online in the Supporting Information section.

This section includes additional references to primary literature relevant for this research [22–25].

**Supporting File:** cite70137-sup-0001-SupMat.docx.