OVERVIEW OF PM SAMPLING AND DILUTION METHODS

Summary and Key Messages of Action A1 Report

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Technical report on overview of PM sampling and dilution methods

Summary and Key messages of Action A1 report

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1 Introduction

Small-scale appliances for the solid fuels combustion at residential heating produce large amounts of gaseous and particulate emissions containing both organic and inorganic substances due to the poor combustion in such appliances. The use of biomass such as firewood and wood pellets for residential heating is very common in Europe and beyond. Biomass is a renewable energy source and it is considered climate friendly source of fuel as trees and forests act as the sink for carbon dioxide (CO_2) emissions. It can provide energy security of the regions where biomass is easily available. However, the combustion of biomass in the residential sector contributes significantly to the pollution of ambient air, in terms of fine particle emissions (1). In some European countries, the use of other solid fuels such as coal and anthracite are also accepted for residential heating which produce high particulate and organic emissions than the biomass(2). Despite the source, all solid fuels produce both the gaseous and particulate emissions which should be measured appropriately to estimate their emission factors and pollution level. If the correct measurement method is not applied, measured emission factors in or close to the high-temperature and high concentration exhaust stack may misrepresent and even miss the amount of gaseous or particulate emissions that actually enters the atmosphere (3). Sampling methods are baseline for reliable testing, because they affect both quality and quantity of particle sample that is collected or analyzed after sampling. In addition, there is a direct relationship between the particulate matter (PM) sampling methods and the emission components to be measured. To estimate and evaluate the important components of emissions, it is imperative that all released emissions from residential combustion sources are measured with appropriate sampling and measurement methods. The accurate PM emission measurement results are required also for the real calculation of emissions in the emission inventories for the modelling of climate effects of PM emissions, standardization groups (e.g. European committee for standardization - CEN) for developing new harmonized standards for emissions of air pollutants, the Construction product regulation (CPR) and/or Eco-design regulations for implementing measures, and environmental health researchers and organizations for the study of adverse effects of PM emissions to health.

2 Particulate matter sampling and dilution methods

At the moment, measured PM emission factors vary between member countries depending on the standard used (4). This makes difficult to understand the effects of residential combustion emissions on air quality (1,4–6). However, reliable methods exist, and are well known in the most European countries but these methods have not been evaluated and implemented in the harmonized way. There are several different standardized and non-standardized sampling methods for the measurement of combustion generated particles. The most standardized PM measurement methods are based on the hot flue gas method i.e. collecting particles directly from the hot exhaust in filters while some standard methods also collect the particles diluting the exhaust gases in dilution tunnel (DT), considering the measurement of condensable organic compounds. However, hot flue gas method cannot capture many organics that are still in gas phase due to the high temperature of exhaust in the stacks (7). Instead, dilution of combustion exhaust can capture most of such organics and provide better estimation of particulate emissions from the combustion (8–10). Then again, the appropriate measurement of PM emissions is dependent on the used sampling techniques, type of filters used, dilution and sampling conditions, and other variables of the measurement systems such as size of the nozzle and nozzle orientation, and sampling point considered etc. (1,6).

There have been several initiatives on the solutions for the issues associated with the sampling and dilution methods (e.g. inclusion of organic compounds) for the measurement residential combustion emissions in Europe. For instance; BeReal project coordinated by Technology and Support Centre in the Centre of Excellence for Renewable Resources (TFZ) (http://www.bereal-project.eu/). Another initiative was the EN-PME validation project which was intended to replace three existing methods and to make clear distinction between solid particles and volatile organic compounds (VOCs) with both components to measure separately (3). The project has developed a simple sampling probe which can collect combustion particles directly from the stack and it has been included in the European standard EN 16510 for residential solid fuel burning appliances (11). However, EN-PME method is not designed to measure particulate organic matter. Therefore, there is still a clear need for the harmonized measurement method to measure the residential emissions effectively and include all the solid particles and organic emissions. Similarly, an international expert workshop hosted by MSC-W in 2020 to create better understanding of the issues and possible approaches for dealing organic compounds concluded that there is a real need for the harmonized PM emission measurement methods to include condensable for residential combustion emissions in EU and dilution method is an option for this purpose (11).

Different types of diluters have been tested during the wood combustion experiments in different health and environmental studies (5,12–18). Their results are varied depending on the types and capacities of these diluters as well as configuration of the test rigs. In addition, the studies have used different instrumentations for the measurement of diluted emissions and their results are also varied depending on the used instrumentations. Increased use of such new methods, protocols, and instrumentations should expand the reliability and comparability of data, and lead to much more realistic and comparable emission factors (EFs) for European residential combustion emissions (3). In addition, large-scale comparison of existing methods is required to explore a harmonized method for the measurement of residential combustion emissions in terms of reliability, cost, and usability.

3 Sampling methods in hot flue gas

In Europe (except Norway or the United Kingdom), the most commonly used standardized approach for PM measurement is the extraction of flue gas directly behind the combustion appliances (19). Generally, sampling in hot flue gas gives lower results in comparison to sampling from DT because organic substances in the gas phase penetrate the filter in hot flue gas methods, especially during poor combustion, and therefore these methods are not very suitable for determining real-life emissions.

There are several methods for sampling from hot flue gas such as EN-PME method, EN 16510-1:2018 - Heated filter, Dust measurement according to DIN^{+}, EPA Method 5H, VDI

2066:2021 Blatt 1 and EN 13284-1:2017 methods. In addition, SPC-IPA method also combines heated filter and impinger filled in with isopropanol collection. These methods vary in different approaches to nozzle orientation choices, probe length, filter holder temperature, start time and sampling length, choice of anisokinetic/isokinetic sampling, filter conditioning, considering of deposits in probe and others. This can lead to different results of the measurement.

4 Sampling from diluted flue gas

Several dilution methods offer good measurement results for wide range of emission components formed during residential combustion. However, the amount of PM emissions captured by a dilution method depend on the type of used diluters, residence time, mixing effects, flow control, sampling flow rates applied and operating conditions such as temperature limits, humidity, pressure/draft condition, and dilution ratio (DR), (1,7,20)Particle deposition and losses in the sampling line also affects the concentration of PM emissions(1,7,20) There is also a lack of knowledge or agreement on these sampling parameters to be applied. At the moment, full flow dilution tunnel (FFDT) has been used in several international standard methods (e.g. Norwegian standard, US EPA standards and Australian/New Zealander standard) for measuring residential combustion emissions which has numerous shortcomings (20–23). Several partial flow dilution sampling methods suitable for the measurement of small-scale combustion emissions are available and used mainly in the research-based studies (20,21,24).

Dilution methods vary between different studies and there is no comparable scientific studies available. Thus, it is hard to say which dilution sampling method is the best to measure residential combustion emissions. Currently, dilution sampling methods are not used in type testing in Europe except in Norway and the UK (19). For FFDT methods, these are inappropriate for a research purposes and field testing because the dilution ratio in the DT is low (meaning that concentrations are too high for specific sensitive instruments, such as for particle number measurement) and the DTs have large dimension with high construction and maintenance costs. In contrary, the most partial flow dilution systems are smaller, portable, less expensive and easy to use as compared to full flow dilution methods which are spacious, costly for maintenance and construction and unsuitable for field testing (20,24). These dilution systems can also be feasible for the dilution parameters such as temperature adjustment, flow control, and dilution ratios.

4.1 Table of comparison of dilution methods based on literature review

Methods	Disadvantages	Advantages
Full flow dilution tunnel or hood (EPA 5G, NS 3058-2) (US Environmental	The dilution setup is large, complex and costly and not applicable e.g., in field testing (20,24)	It enables quite accurate measurement as compared to hot filter method (20,24). If constant volume sampling (CVS) is used, also transient phases can be measured (e.g., cold start, on-off operation) (20,24).

Protection Agency, 2000).	Use of unfiltered laboratory air which may contain organic species (20,24) Dilution ratio in the dilution tunnel method is usually quite low (DR < 10) and secondary dilution might be needed (20,24). The dilution ratio may vary during one measurement and from one measurement to another (20,24)	
CEN TC295 WG5 standard draft: Full flow dilution tunnel (FFDT)	This is a proposed method and has not been tested for any experiments (20). There is not any explanation in the draft what is the scientific basis of this conversion equation.	Similar as EPA 5G Dilution Tunnel method; FFDT (20).
Partial flow dilution tunnel (ISO 8178-1)	A temperature correction is required (13,20). Difficult to adjust the dilution ratio (13,20).	Volume flow is constant (13,20). Modification to the dilution tunnel can be done as per requirement (13,20).
U.S. EPA Conditional test method (CTM 039)	Condensation of moisture in the mixing cone or residence chamber (20,25). Not useful for measuring stack gases containing water droplets (20,25). There can be losses in cyclones, venturi, mixing cone and residence chamber (20,25).	Dilution sampling at constant sampling rate procedures (20,25). No hood is required and is portable (20,25). Useful standard method for field measurement (20,25).
Modified full flow dilution tunnel (Boman et al. (20)	Dilution ratio may vary during one measurement and from one measurement to another in batch combustion (20,26).	Collect more condensable organics than EPA Method 5G (20,26). Hood is not needed, and the setup can be made portable quite easily (20,26).
Atmospheric wind tunnel (Kinsey et al. (14))	Wind tunnel is relatively expensive to construct, requires a	High amount of PM mass is captured (14,20).

	lot of roof area and is not portable (14,20). Questionable experimental results (14,20).	
Partial flow dilution tunnel – Caltech dilution sampler (Myers and Logan(27)**)**	Mixing is not complete at dilution ratios < 21 (20,27). Particle losses are higher at the inlet line and the venture(20,27). VOC results should be corrected. Background VOCs are deducted from the measured VOCs (20,27).	7-16 times as much organic aerosol as the heated filter portion of the EPA method 5H (20,27). Dilution ratio can be kept constant (20,27).
Portable partial flow dilution tunnels (Lipsky and Robinson, (28)**)**	Losses in the sampler inlet lines (20,28). Difficult to stabilize the exhaust flows before collecting the sample (20,28).	Portable system as the size of the dilution tunnel and associated flow control systems are reduced (20,28). Operates at a much lower flow rate of the exhaust than Caltech sampler (20,28). Residential time tank is not required (20,28).
Compact dilution sampler (CDS) (England et al., 2007). [Quite similar to U.S. EPA CTM-039]	Long residence time as compared to many other diluters (20,25). Adjustment to flow required during the measurement (20,25).	Rapid mixing of the raw sample with the dilution air (20,25). Works on the low concentration of stack sample (20,25). Presents uniform velocity profile and minimize wall losses (20,25). Useful for sampling semi-volatile species in the gas stream (20,25).
Rotating disk diluter (Hueglin et al. (29)**)**	Loss of larger (> 1 µm) particles due to impaction of particles into cavities (20,29). Long term stability is questionable due to the dilution ratios (20,29).	These are small and portable, low-cost and easy to use (20,29). Compatible with variety of sensitive particle sizers and counters (20,29). The dilution process occurs in a single step and on a fast time scale (20,29). The diluter can control and regulate flows and temperature (20,29).

Concentric tube diluters	High losses in dilution point as well as thermophoretic losses can be significant in the concentric tube diluters (20,30). Nucleation may occur during the measurement (20,30).. If the diluter is placed out stack, condensation of water or organic species on the particles probably occur (20,30).	Concentric tube diluters induce turbulent flow in the dilution point and ensure fast mixing (20,30).. Easy to clean and maintain, possesses local and remote-control operation (20,30).
Ejector diluter	Fast clogging of the ejector nozzle at high particle loadings (24,31). The calculation of the true residence time in the ejector dilution systems is not a straightforward and may require 3D CFD (24,31). Pressure fluctuations and easy contamination of orifice (24,31). The sensitivity of the DR on sample pressure and temperature variations (24,31).	No need for flow control devices or pumps (24,31). Stable operation and low maintenance costs (24,31). With the two-stage setup condensation and nucleation can be eliminated if desired and a sample can be taken directly from the flue gas duct (24,31). Sample can be taken directly from stack with two-stage set up (24,31). Constant dilution factor/ratio (24,31). It can perform the measurement up to 450 ^{0}C (24,31).
Perforated tube diluter	Induce vapor losses in the diluter and also thermophoretic and other particle losses occur (20,32). A complicated turbulence inside the diluter produces broad particle number size distributions (20,32). This diluter is not useful for the study where nucleation should be avoided (20,32). The diluter provides a direct contact between the undiluted sample and dilution air in turbulent mixing (20,32)	The dilution ratio can be adjusted continuously e.g. in the range of 2-40 by using sample flows of 0.5-10 lpm (20,32). Useful for the study related to nucleation, condensation, and agglomeration (20,32)

Porous tube diluter (Lyyränen et al. (21)	Nucleation is not favorable in the porous tube diluter, because mixing and cooling are not rapid (24). Controlling of flows in the system may be challenging due to unstable combustion process if porous tube is the only diluter used (20,24).	Good mixing due to its structure (20,21,24). The most preferable dilution system if the object is to prevent nucleation (20,24). Control of dilution temperature (20,24). Very low particle losses as compared to other diluters (24,32). DR adjustable.
Combination of porous tube diluter and ejector diluter (Lyyränen et al.(21)	At low dilution ratios (primary dilution ratio < 8), the nucleation mode is stronger than in the single stage porous tube dilution (21).	This combination can resolve many problems of the single diluters (20,21). Provides better results than combination of other diluters (8,20,21). Avoids or minimizes particle losses and water vapor condensation (8). The dilution ratio can be adjusted using the control valves (8). Applicable for very sensitive instruments and field measurements (8). The dilution setup functions well in the wintertime and also suitable for other residential fuels and environmental conditions (8).
Dilution Chamber (DC)	Measures slightly low concentration of particulate emissions than dilution tunnel method (23,33).	Provides good residence time (2-3 seconds), enough to produce the condensation of the most SVOCs (23,33). Plane filter can be used to collect the sample (23,33). Easy to control the parameters by an integrated software (23,33). Easy to construct, simple to use (23,33).
Laminar flow dilution system (Pagels et al. (34)	Limited information is available about laminar flow dilution technique (34).	The dilution ratio can be adjusted quickly using the control valves. Particle

	Not sure if this system is sustainable and resolve the problems of other diluters (34).	deposition in the capillary tube is minimal (34). The proportion of pressure drop and flow rate is steady which causes minimal deposition in the tube (34). Wide range of dilution ratio and short residence time can be achieved (34). Applicable for the sensitive instruments (34). Easy to construct and low cost (34).

5 Key Messages

Based on the literature review, it can be stated that there is a clear need for a harmonized particle sampling system for small-scale combustion emissions in EU countries which can collect both the solid and condensable fractions of particle emissions. Key points of the report are below:

- Several sampling and dilution methods and dilutors are available to measure particulate emissions from residential combustion and some of these (e.g. hot filter and dilution tunnel) methods are established also as standards in some countries around the world.
- All available methods have own advantages and disadvantages.
- Three dilution methods are more reliable than others for the measurement of particle emissions in real-life operation.
  - The combination of ejector and porous tube diluters (ED+PTD) system is considered as the most useful method, with which all the most important chemical components and physical parameters of PM emissions can be measured. The combined system is adjustable for different dilution ratios, has low particle losses, prevent nucleation and collect the most important emission components.
  - The second method is Dilution tunnel (DT) method which is used in many emission measurement standards at the moment. It provides more accurate measurements than hot filter methods but set-up is large to use in the field and does not provide high and constant dilution ratios.
  - The third method is dilution chamber (DC) method which seems to be more comfortable option than DT. It provides good residence time to produce enough SVOCs and easy to control the sampling and dilution parameters but measures slightly lower concentration of emissions than DT.
- The usefulness of these dilution methods is rated based on the target parameters of particle emissions to be measured. For instance, if it is needed to measure just solid PM including course particles, DT is the best option. If it is needed to measure both the condensable organic matter and specific compounds e.g. PM2.5, number concentrations and size distributions, Polycyclic aromatic hydrocarbons (PAHs), black carbon (BC)

and surface area concentrations, the combination of ED+PTD sampling method can be the best option. DC in another hand is also a feasible option for measuring solid and organic fractions (e.g. PAHs). However, with the partial flow dilution methods such as DC and ED+PTD systems, it is not possible to measure course particles because isokinetic sampling is difficult with these dilution sampling systems. For the DT, if is it needed to measure more parameters such as PM, additional dilution phase is required.

- Other reviewed methods have one or several disadvantages such as long residence time, large losses of sample, turbulence inside the diluter causing losses, clogging at nozzle of the diluter, difficulties to control sample flow and adjust the dilution ratio.
- To measure solid and condensable particulate matter, a combined hot flue gas filter with dilution can be an option. In this project, a combination of EN-PME-probe (with filter) and Porous Tube Dilutors (PTD) (with filter) will also be tested.

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Harmonizing reliable test procedures representing real-LIFE air pollution from solid fuel heating appliances - **Real-LIFE Emissions** project.

Project Partners

- University of Eastern Finland (UEF)
- Technical University in Ostrava (VSB)
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