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Feasibility of Using Isokinetic Sampling Techniques to Extract a Representative Sample from Processes in the United Kingdom

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Abstract: The requirement to monitor and control particulate emissions from industrial processes using continuous emission monitoring systems (CEMS) has significantly increased over recent years. Under current legislation, CEMS equipment requires calibration against the standard reference method (SRM) using isokinetic sampling and gravimetric analysis under controlled conditions as detailed through BS EN 13284-1 "Stationary source emissions–Determination of low range mass concentration of dust. Manual gravimetric method". This process includes pumping a known volume of gas through a filter, which is weighed before and after sampling, and the total mass of dust per m^3 can then be calculated to output results in mg/m^3. As tougher legislation is introduced and stringent emission limit values (ELVs) are imposed on emissions processes in the United Kingdom (UK), the calibration of CEMS is increasingly more difficult due to the reliability of the SRM at low concentrations. The accuracy of results from the SRM and therefore CEMS equipment must be questioned when the uncertainty of measurement is higher than process ELVs. This research analyses data taken from an industrial survey and 21 UK processes where the standard reference method, in accordance with the procedure in BS EN 13284-1 has been used for particulate measurement. Investigating the reliability of isokinetic sampling when used as a method to extract a representative sample from a stack process when used in conjunction with innovative, alternative methods of sample analysis. In processes with particulate emissions <5 mg/m^3, 80.7% of the total sample was collected in the rinse, and for processes >5 mg/m^3, 56.4% of the sample was collected in the rinse. The data does not suggest any correlation between any of the measured parameters and the percentage of particulate in the rinse, including the stack velocity, isokinetic percentage, sample volume, and total mass concentration.

Keywords: isokinetic sampling; gravimetric analysis; standard reference method; continuous emission monitoring system; particulate deposit; measurement accuracy

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1. Introduction

The impact of particulate matter emissions cannot be ignored; there are serious health [1,2] and economic effects to be considered [3]. Understanding particulate emissions accurately is imperative to implementing appropriate control and reduction measures to create a cleaner environment [4], as well as ensuring compliance with emissions limits imposed on plants and processes. As explained by Nicklin and Darabkhani [5], there is an immediate requirement for innovative development in the measurement of dust particulate emissions.

Operators of plant processes across the UK have legislative requirements to ensure that emissions monitoring equipment is operating to give reliable and repeatable results. In the UK, the responsibilities under BS EN 14181 "Stationary source emissions—Quality assurance of automated measuring systems" [6] for the client, are detailed in guidance notes issued by the Environmental Agency (EA) [7]; for the installation of compliant equipment termed Quality Assurance Level (QAL) 1, the in-situ calibration of the equipment through

an accredited test called QAL 2 and ongoing quality assurance tests based on zero and span equipment checks called QAL 3, as detailed in Figure 1. These QAL checks ensure that the legislative requirements for monitoring particulate emissions are met, and the burden lies with the operator of the equipment.

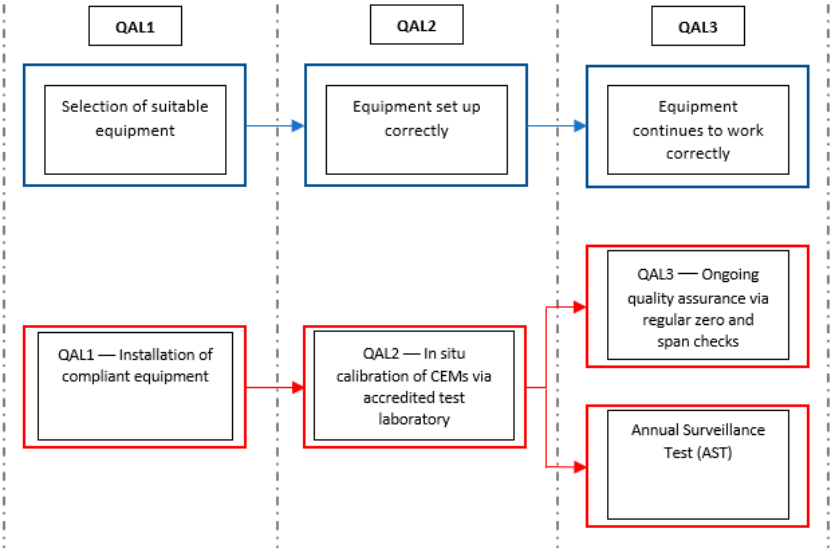

Figure 1. Operator equipment requirements.

As explained by Jahnke [8], particulate CEMS equipment is calibrated against the SRM and a calibration factor is applied to equipment, whereas with gas CEMS, calibration gases can be used as a reference to ensure calibration of equipment. The calibration gas should be traceable to recognised international standards and is used to establish a known analyser response to a certified gas component concentration. This methodology is not possible when considering the calibration of particulate CEMS, as no viable particulate mass concentration standard exists due to the complex nature of particulate emissions [5]. When processes are in operation, the characteristics of fuel can change. These small changes in fuel can cause the particulate emissions profile to differ significantly as detailed in a study by Kasurinen [9].

To accurately measure the mass concentration of particulate emissions from a combustion process using the SRM, the extracted sample must be representative of the emissions present. The general measurement concept of the SRM is to pump a known volume of the flue gas through a filter and weigh the filter in controlled conditions before and after sampling. The flue gas is sampled isokinetically, that is, at the same velocity as the gas in the stack or duct. The total mass of dust collected on the filter, and therefore per m^3 of gas, can then be calculated to give meaningful results in mg/m^3 format. In research presented by Antonsson [10], the performance characteristics of the SRM are investigated, detailing two contributions for underreporting in isokinetic sampling: the first being the widespread practise of 5% super-isokinetic suction velocity and the second being the geometry design of nozzles used. These two factors can account for a 13% underestimation of particulate emissions. Research conducted by the National Physical Laboratory (NPL) [11] suggests that significant losses from filters can be seen, potentially up to 50%, making this a major consideration when investigating the accuracy of SRMs. Developments have been made to create alternative techniques for determination of particulate on filter media, detailed in a study by Garland [12], and losses associated with particulate losses at the probe itself, detailed in a study by Sreenath [13]. This research investigates the distribution of particulate in the rinse procedure by analysing parameters collected during testing in accordance with BS EN 13284-1 [14] to determine the feasibility of isokinetic sampling to obtain a representative sample. Similar methods to the European SRM are used throughout the world, including ISO 9096:2017 "Stationary source emissions–Manual determination of mass con-

centration of particulate matter", and the United States (US) Environmental Protection Agency (EPA) Method 5 [15]. All standards use a very similar process, of which the findings of this study are relevant for the development and improvement of the methodology.

This research continues the investigation into the accuracy and reliability of isokinetic sampling and gravimetric analysis of particulate emissions—a process known as the standard reference method. An analysis of particulate emissions data from 21 UK sites was undertaken. This was used to assess the filter and rinse data to identify the distribution of particulate in the filter and sample train to determine possible areas of improvement to minimise losses and increase accuracy for operators and designers of the SRM process. The information presented in this research would aid the application of the SRM for stack emissions testers to help increase accuracy and understand where improvements can be made whilst highlighting areas for the development of the methodology through further research. Only a sample of the total emissions is extracted from the stack when using the SRM technique; therefore, it is imperative that the extracted sample is representative of the emissions as a whole.

2. Legislation

The field of emissions monitoring is highly regulated. The objective for accurate measurement for most operators and processes is to ensure compliance with increased legislative requirements. As technologies become available and CEMS are developed, process control can also be monitored. Emissions legislation is putting tighter restrictions on plant processes to reduce harmful emissions, as detailed in a study by Paola [16]. These low levels of particulate emissions are observed in the 21 sites tested in this project, with 62% of sites reporting a result <5 mg/m^3. It should be noted that in BS EN 13284-1 [14], the SRM is only validated for dust concentrations of >5 mg/m^3.

The legislation that is relevant to an application is generally determined by the size and operation of the process. In the UK, the EA are the governing body responsible for not only ensuring compliance with legislation, but also guidance and advice to maintain compliance. Whilst the legislation gives general guidance and emissions limits, in the UK, the EA are responsible for issuing permits detailing the application specific ELVs. Emissions from combustion processes are highly dependent upon the characteristics of the fuel used, particularly dust particulate emissions [9]. There are European Standards for emissions covering the requirements for particulate monitoring and measurement including the requirements of measuring points in a stack, requirements for the SRM to determine dust concentration, the calibration of CEMS, the characteristics of measurement equipment and the QAL requirements as detailed in Figure 1, to ensure equipment performs during its lifetime, and the handling of environmental data. This research project focusses on the SRM as a reliable method to extract a representative sample from a process, as per the methodology detailed in BS EN 13284-1.

In the UK and Europe, BS EN 13284-1 [14] details the SRM for particulate measurement in ducted gaseous streams >50 mg/m^3, although this technique can be, and is, used at higher concentrations. BS EN 13284-2 "Stationary source emissions—Determination of low range mass concentration of dust. Quality assurance of automated measuring systems" [17] derived from BS EN 14181, details the quality assurance procedures related to CEMS for the measurement of dust particulate in order to meet the uncertainty requirements on measured values detailed in relevant regulations.

In addition to the above-mentioned legislation, the most relevant European standards relating to dust particulate can be categorised into three sections including monitoring methods, instrument performance and test specifications, and quality assurance. Monitoring methods are covered in BS EN 13284-1, EN ISO 16911 "Stationary source emissions–Manual and automatic determination of velocity and volumetric flow in ducts" [18,19], and BS EN 15259 "Measurement of Stationary Source Emissions–Requirements for measurement sections and sites and for the measurement objective, plan and report" [20]. Instrument performance and test specifications are covered in BS EN 15267 "Certification of Automated

Measuring Systems", parts one, two, and three [21–23] and these regulations cover all requirements for certification of CEMS. Quality assurance is covered in BS EN 14181 and BS EN ISO/IEC 17025 "General requirements for the competence of testing and calibration laboratories" [24]. The Medium Combustion Plant Directive (MCPD) [25], applies to medium combustion plants in a thermal size input range of ≥1 to <50 MWth and applies to around 143,000 plants across Europe [26]. The majority of combustion plants are in the scope of the MCPD, including boilers, engines, and turbines excluding large combustion plants regulated under the Industrial Emissions Directive (IED) [27]. The IED [28] establishes the main principal for the permitting and control of large industrial installations, focusing on providing the best available techniques to achieve a high level of environmental protection considering both costs and benefits. There is considerable legislation covering all aspect of emissions monitoring, but all legislation influencing the measurement of particulate emissions references the SRM detailed in BS EN 13284-1. This research investigates the feasibility of using isokinetic sampling to obtain a representative sample, therefore specifically focusing on the methodology detailed in BS EN 13284-1.

In European Legislation, ELVs are expressed in mg/m^3 and mass/volume of dust particulate in a flow of gas, generally given on a dry basis at a reference O_2 concentration. For this reason, CEMS report an output in this format. As detailed in work covered by Nicklin and Darabkhani [5], CEMS use measuring principals that do not directly measure the particulate in a gas stream, but instead measure a function of a parameter of the dust emissions in the flow of gas. The output in mg/m^3 must be determined for each system for each application, due to changes in the characteristics of the particulate emissions; therefore, it is imperative that the SRM is representative of the actual emissions.

3. Process and Methodology

This section will detail the research and project methodology, and the process of the isokinetic sampling for the SRM to the requirements of BS EN 13284.

3.1. Research Methodology

Further to research undertaken by Antonsson [10] detailing the effect of nozzle design and placement on the representativeness of the sample, the sample train is of particular interest to this research project. This research analyses data collected through isokinetic testing on 21 sites in the UK across a range of processes including incineration, energy recovery, spray booths, energy production, and manufacturing. All testing was undertaken on industrial processes operating as expected with no known errors, such as any abatement failure, nor during any cleaning process which may influence results. The expected emissions values were not known before commencing any testing, and the characteristics of the particulate was not known. No additional analyses other than those completed under the SRM was undertaken throughout this research. All presented results are corrected for standard conditions and are representative of those used for the purposes of emissions reporting. The sample train is designed to be the transport system for the emissions sample and is therefore imperative to the accuracy of the final particulate measured. The typical equipment used for the isokinetic sampling and particulate capture is detailed in Figure 2.

The process of emissions monitoring is a sampling exercise with the fundamental requirement to ensure that the sampled gas is representative of the actual emissions of the bulk gas stream. An accredited laboratory or test facility to BS EN ISO/IEC 17025 [24] is required to undertake isokinetic sampling to BS EN 13284-1 for the purposes of particulate CEMS calibration. The general principal of measurement using the SRM is to sample a known volume of flue gas through a filter. The sampling must be undertaken isokinetically, where the suction at the nozzle is at the same velocity as the flue gasses in the duct or stack. By weighing the filter before and after sampling, the total particulate per volume of gas can be determined [29]. By undertaking the SRM in accordance with the process detailed in BS EN 13284-1, the results were analysed to determine the feasibility of achieving a representative sample from a plant process. A total of 21 sites were selected for testing

across a range processes, all of which require annual validation of particulate CEMS via the SRM. All tests undertaken in this research are to the same standard of that required for the calibration of CEMS and samples were taken from existing sample ports suitably sited for extractive surveillance testing. The data obtained and recorded for the purposes of analysis include the stack velocity, percentage isokinetic, sample volume, particulate concentration, filter mass, and rinse mass.

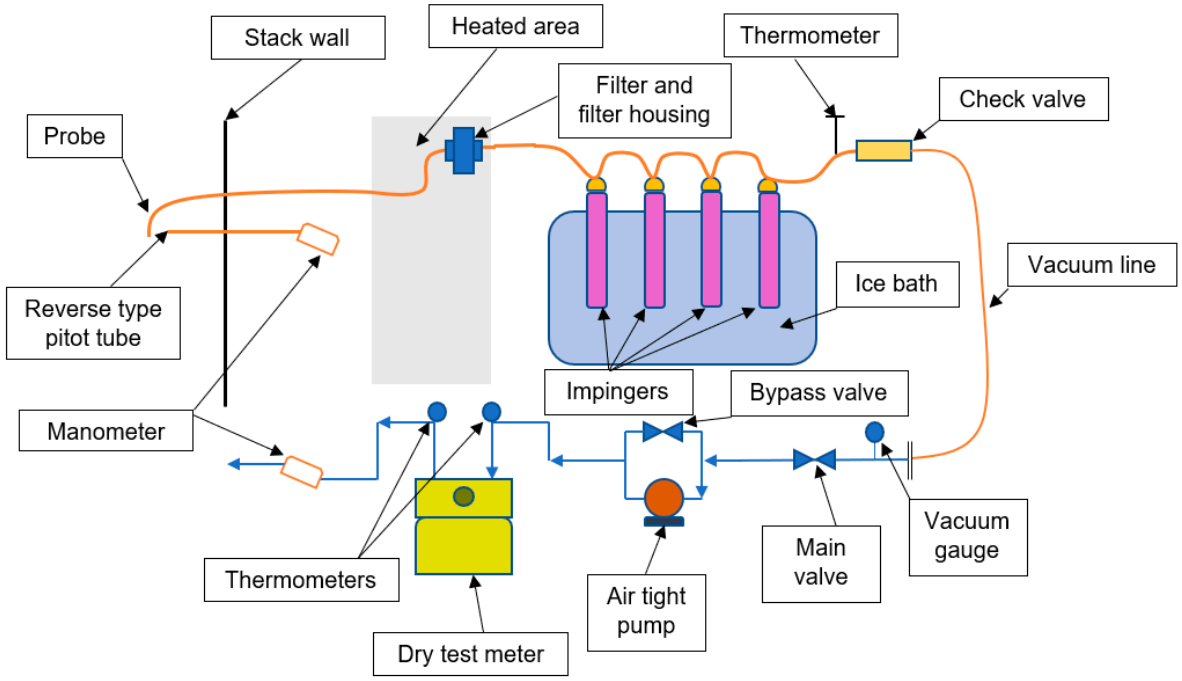

Figure 2. Typical isokinetic sampling equipment schematic [26].

3.2. Experimental Methodology

An overview of the SRM procedure detailed in BS EN 13284-1 and followed through this research is detailed in this section. The process begins with the preparation of equipment and pre-site tasks. This includes the handling of filters, which can be a major source of uncertainty, especially in low concentration measurements where only a small quantity of particulate will be collected on the filter. Filters are installed into their own housing for transportation to the test laboratory. To select the most suitable nozzle diameter, a number of tests are required. These "pre-measurements" include determining the waste gas concentration of oxygen, CO_2, and water vapor; the density of the waste gas; the number and location of measurement points in accordance with BS EN 15259 [20]; the temperature and velocity of gas at the measurement points; and a check for deviations of gas flow regarding duct axis. Leak testing of the equipment is required before any testing begins to ensure no external source will influence the results. The sample train is preheated to the selected temperature depending upon the stack temperature and inserted into the duct. The nozzle must face directly into the gas flow, avoiding contact with any part of the duct. A minimum of 30 min sample time is required. The probe is relocated during testing to cover each section required without pause in sampling. Isokinetic conditions must be maintained throughout the whole process. During sampling, the temperature and volume of gas at each point must be recorded, and during post sampling, a visual inspection of the filter is undertaken. All components to be weighed are enclosed in an electrostatic free container for transportation. In addition to the dust component captured on the filter, there may be dust deposits in the sample train. BS EN 13284-1 [14] states that the deposits found can often be 10–30% of the total dust when sampling from waste incinerators at around 5 mg/m^3. There is currently no known way to keep this negligible and therefore the system requires to be rinsed down to collect these deposits. The particulate collected from the

system rinse is added to the particulate collected on the filter for a total particulate value. The total mass of particulate/volume of gas can be calculated via the known volume of gas and known total mass of particulate.

3.3. Survey Methodology

For additional analysis and understanding in this project, a survey was conducted as a systematic method to obtain perspective from users and operators of SRM equipment. The focus of the survey was to target personnel who work to the methodology set out in BS EN 13284-1, to determine the current position of the industry. Jones [30] explains that to obtain a representative sample it is important to consider who will be targeted, and therefore the participants' industry statuses were confirmed for all participants. An online research tool was developed, similar to that used by Sherman [31] to collect data from participants with the total number of answers for the survey being 732.

3.4. Analysis Methodology

Data collection for analysis was through the SRM and included recording of the stack velocity, isokinetic percentage, sample volume, particulate concentration, filter mass, and rinse mass. The objective of this study is to determine the feasibility of extracting a representative sample using the SRM. The behaviour and influence of the measured parameters were analysed to determine and define any correlation affecting the representativeness of an extracted sample. Analysis procedures in this research include visual examination of data patterns with graphs and visual aids. Through scatter graphs, an inspection of the distribution of the isokinetic percentage, stack velocity, and sample volume against the percentage of particulate in the rinse was carried out for an inspection of data patterns. Using the same dataset, the Pearson correlation coefficient was calculated to measure the strength of the relationship between the two variables, using the following formula.

$$r = \frac{\Sigma(x_i - \overline{x})(y_i - \overline{y})}{\sqrt{\Sigma(x_i - \overline{x})^2 \, \Sigma(y_i - \overline{y})^2}} \quad (1)$$

where:
r = correlation coefficient;
x_i = values of the x variable in a sample;
$\overline{x}$ = mean of the values of the x variable;
y_i = values of the y variable in a sample;
y_i = mean of the values of the y variable.

Using the value calculated for r, the correlation strength can be assessed to determine the influence of parameters. A comparison was made through a visual scatter graph and the Pearson correlation coefficient methods to compare the effects of the total mass concentration of particulate and the percentage of the particulate found in the rinse. A mean average for all results with and without filter losses were calculated for stack velocity, isokinetic percentage, sample volume, percentage of particulate in rinse, and the total mass concentration. This allowed for analysis of samples recorded with and without filter material losses. Survey analysis was undertaken to determine the perceptions of operators of SRM equipment, looking for trends in results using NVivo software to aid organisation of the data. Thematic analysis was then undertaken on the data by coding topic areas and identifying prevalent themes.

4. Results and Discussion

This section will detail results from 21 UK sites detailed in Figure 3, using the SRM detailed in BS EN 13284-1 to measure dust particulate mass concentration with a view to analyse data on the feasibility of extracting a representative sample. With legislation driving levels of particulate emissions lower than ever before, the reliability of equipment is questionable at these low levels, especially at levels <10 mg/m^3. There is a requirement to measure the mass of particulate emissions in a gas stream directly, eliminating

the issues surrounding particulate parameter driven measurements encountered with continuous monitoring. Methodologies including triboelectric, electrodynamic, laser, and optical measurements require site calibration due to the vast inconsistencies in particulate characteristics. It is not possible to create a standard "calibrated particulate" due to the variations in particle size, composition, shape, colour, and refractive index. Using the SRM as a technique to take a sample of particulate for a given period, the purposes of calibration of CEMS is well used in the emissions monitoring industry and is the best available technique for processes emitting >10 mg/m^3. There is a particular focus in this research to determine the feasibility of using the isokinetic sampling methodology to achieve a representative sample for the purposes of integration of alternative analysis methods. When the methodology is used as a complete system and a rinse is part of the procedure, the losses associated with particulate deposits collected in the sample train can be minimised. When the isokinetic sampling system is used with a view to analyse the sample for use with alternative methods, the losses of particulate in the sample train must be considered.

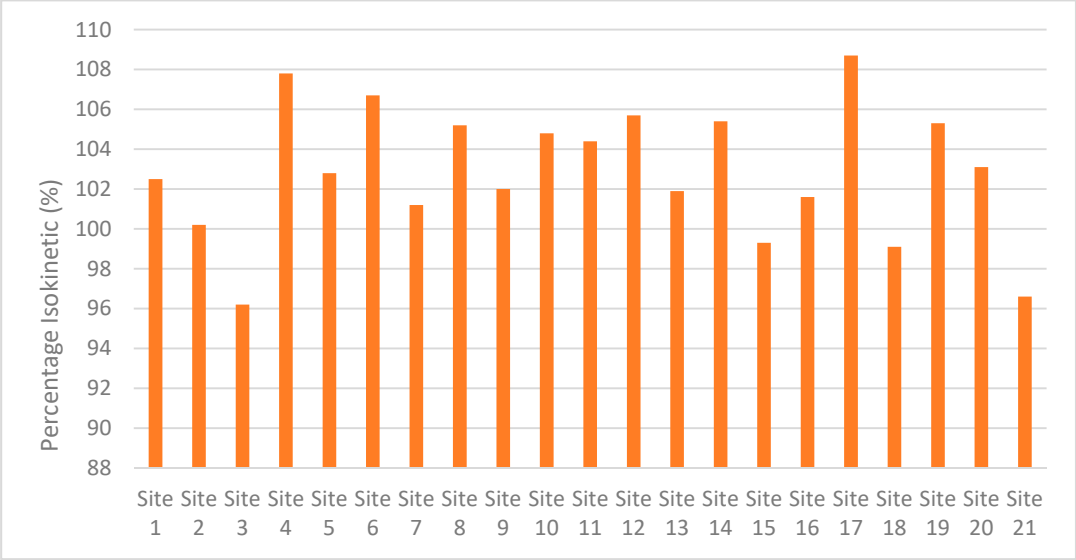

Figure 3. Isokinetic percentage for each sample.

It is confirmed that all results are within the isokinetic rate of 95–110% (Figure 3), as required by BS EN 13284-1. The isokinetic rate is the relationship between the ratio of gas velocity in the entry nozzle to the gas velocity in the duct, determined by the following expression, shown as a percentage:

$$V_n / V_d \quad (2)$$

where;
V_n = gas velocity in the entry nozzle;
V_d = gas velocity in the duct.

BS EN 13284-1 specifies a generous acceptable tolerance of between 95–110% isokinetic rate. While all results in this study are within these parameters, a study completed by Antonsson [10] states that any deviations from the ideal isokinetic condition will have an impact on the accuracy of results. The BS EN 13284-1 standard references work completed by Belyaev [32] to justify claims that the SRM can be used to obtain a representative sample. Antonsson [10] highlights critical differences between the work presented by Belyaev [32] and the procedure stated in the BS EN 13284-1 standard, including the limits for ratio of suction velocity at the nozzle to the free gas flow. A super-isokinetic sample will have a negative impact on the result and render the process in accurate.

The effects of both the isokinetic percentage of the sample and the stack velocity to the percentage of particulate in the rinse are shown in Figure 4. The horizontal axis

corresponds to the percentage of particulate in the rinse and is expressed as a percentage (%). BS EN 13284-1 [14] states directly that when the isokinetic percentage rate of the sample and the isokinetic rate are one, the stack gas and gas entering the nozzle are at the same velocity and a representative sample can be taken. However, Antonsson [10] has found that any probe inserted into a particulate laden flow of gas influences the representativeness of the sample even at ideal isokinetic conditions. When using the SRM, the isokinetic percentage parameter is required to confirm that the velocity of the stack gas and gas entering the sample nozzle are within required parameters given to reduce any influence of flow velocity. It is evident from the sites investigated and results presented, that through this research there is a wide spread of data. Calculating the Pearson correlation coefficient gives an r value of 0.081, which is a positive but very weak correlation between the data. This weak relationship suggests that the isokinetic percentage of the sample has no influence on the percentage of particulate in the rinse. The stack velocity is measured as part of the process to monitor the percentage of the sample isokinetic and ensure that the velocity of the gas in the stack and entering the probe are as close as practically possible, on site, during testing. The effects of the stack velocity expressed in m/s on the percentage of particulate in the rinse, and therefore on the distribution in the sampling equipment is shown in Figure 4. The calculated Pearson correlation coefficient gives an r value of −0.248, which is a negative but weak relationship. From the 21 sites tested, there is no evidence to suggest that there is any correlation between the velocity of the gas in the stack and the distribution of particulate between the filter and rinse.

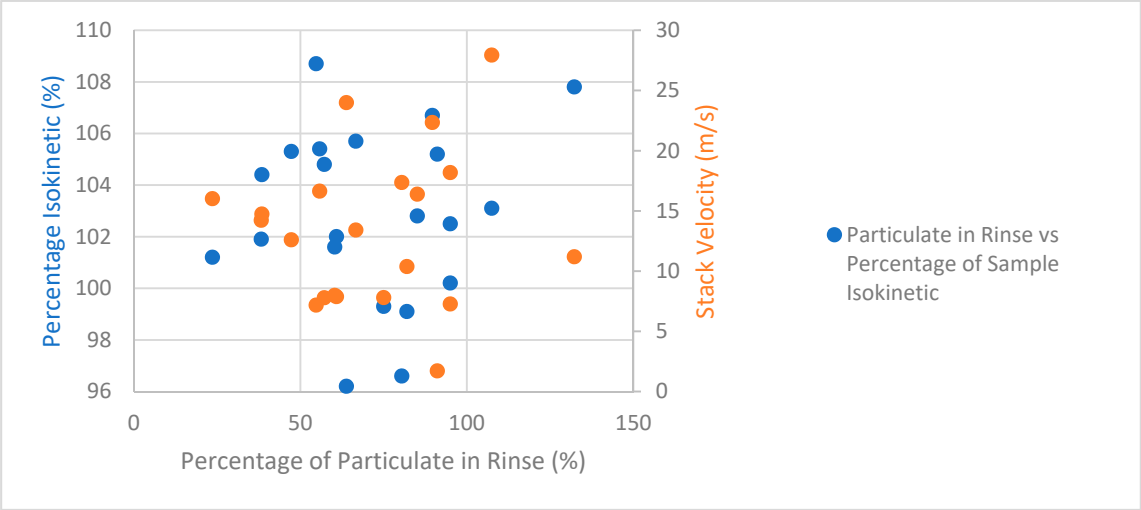

Figure 4. Isokinetic percentage of each process detailing the percentage of particulate in the rinse.

The sample volume is a required measured parameter used to determine the mass concentration to output in reportable format of mg/m^3. The 21 sites analysed through this research shown in Figure 5 all expectedly have varying emissions profiles, including particulate emissions with a sample volume range between 0.35 m^3 and 1.416 m^3. Any variation in the measured volume of gas will have significant impact on the accuracy and reported particulate emissions value. The relationship of sample volume, expressed in m^3 and percentage of particulate in the rinse, is detailed in Figure 5, and analysis was undertaken using the Pearson correlation coefficient with an r value of 0.0248 showing a positive but very weak relationship. There is no evidence to suggest any relationship between the sample of gas analysed and the percentage of particulate found in the rinse. For the purposes of accurate analysis when referencing the percentage of particulate in the rinse, some data represent >100% of the sample captured in the rinse, which can be attributed to particulate filter losses. When reporting for legislative purposes, the negative value of filter mass is not considered. In this research, the losses of the filter are added to

the mass of the rinse, creating a negative recorded mass for the filter when compared to the pre-weighed mass so that the influence of filter losses can be considered.

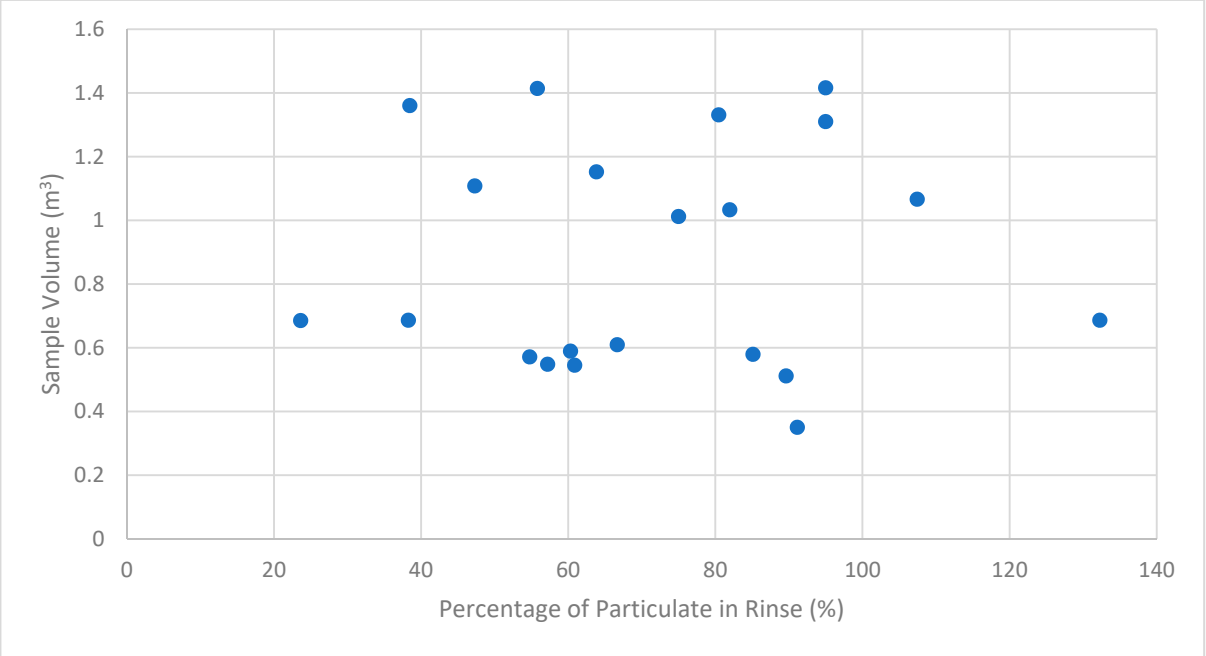

Figure 5. Sample volume vs. percentage of particulate in rinse.

Throughout the testing of the 21 sites, analysis shows that losses are experienced during the pre- and post-sampling filter measurement; therefore, losses of filter media are experienced. A study completed by the National Physical Laboratory (NPL) [11] details that losses on filters specifically for BS EN 13284-1 can potentially be up to 50% of the mass of particulate collected when sampling. Careful handling of filters is required at all stages of testing with the design of filters having little impact on losses experienced. Figure 6 shows a comparison of tested parameters from samples with and without filter losses to determine how these factors influence the losses in the filter and therefore the reliability of the sampling equipment. The data were split into two categories, those with negative mass readings suggesting filter losses, and those without, showing a positive mass reading. A mean average for the tested parameters in each category was used for analysis and comparison. The average stack velocity across all results is 16.2 m/s, whilst the average for sites with filter losses is 19.6 m/s and the average for sites without filter losses is 12.8 m/s. Whilst the data from the sites tested show that an increase in velocity increases filter losses, further specific research on filter losses is recommended for reliable analysis. The mean average for the percentage of the sample that is isokinetic is 104.05%, whilst that for sites with filter losses is 105.5% and that for sites without losses is 102.6%. The sample volume taken shows no influence on the losses in the filter as the average with and without filter losses is 0.9 m^3. In a study by the NPL [11], they found that the flow rate has little influence on the losses in filters and that the greatest losses are due to the handling and measuring processes. As previously mentioned, the presented results will show >100% of particulate in some instances where there are filter losses; therefore, as expected the average of particulate in the rinse where there are filter losses is 119.9% and the average without losses is 65%, showing that the >100% results are due to negative filter mass results and exaggerated rinse mass results. The mass concentration is lower for samples where there are filter losses, with an average of 1.9 mg/m^3, while the same measurement is 30.6 mg/m^3 where there are no filter losses. As there are no data to suggest any strong influence from the stack velocity, isokinetic percentage, and sample volume on the filter loss, the results suggest that filter losses could be present

in all samples, but the higher concentration found on the filter is higher than the losses experienced, which maintains a positive value for the filter mass. In a study by Elder [33], the efficiency of glass-fiber filters similar to those used in the SRM procedure in this research are around 99.9% effective with particle size having little influence. For processes with particulate emissions <5 mg/m^3, 80.7% of the sample was collected in the rinse and for processes >5 mg/m^3, 56.4% of the sample was collected. Whilst the delicate handling of the filter and accurate processing and measurement is vital to achieving good quality results, there is a significant percentage of particulate collected within the rinse that cannot be neglected.

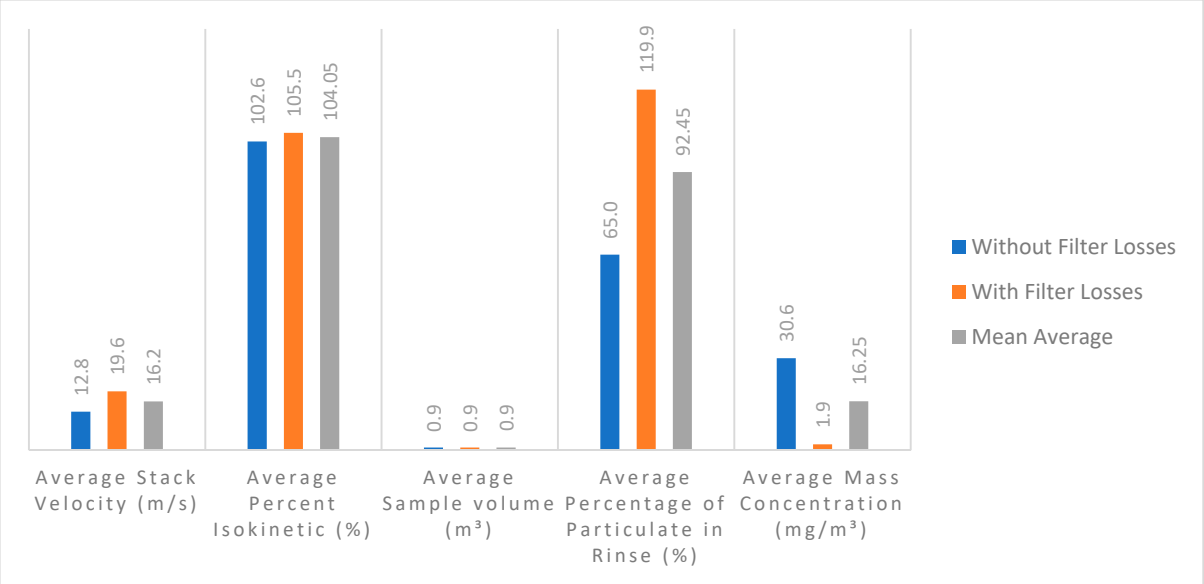

Figure 6. Comparison of an average of testing parameters for samples with and without filter losses.

The flow of gas emissions through a duct or similar is not constant, as detailed in a study by Malet [34]. Particulate generally follows the path of least resistance, with the greatest concentration generally found where velocity is highest and not near the stack walls [35]. Emissions from plant processes can vary significantly, for combustion processes, fuel has great influence on the emissions profile [9]. Conditions can be hot, wet, and corrosive, adding to challenges in accurate sampling. The range of particulate collected within the rinse across the 21 sites is from 23.6% to 132.3% with an average of 71.4%; therefore, consideration for the accurate collection of particulate in the rinse is imperative for accurate implementation of the SRM. In Elders [33] study on the deposition of particulate in the sample train, the highest quantities of particulate were found on the leading edge of the probe liner and at the bends. With such high quantities of particulate found in the sample train, and therefore the rinse, confirmation of careful implementation of the rinse procedure is required.

The concentration of particulate for results greater and less than 5 mg/m^3 are shown plotted against the percentage of the mass in the rinse on the horizontal axis in Figure 7. For sites with a mass concentration >5 mg/m^3 the r value is −0.078, showing a negative but weak relationship. For sites with a mass concentration of <5 mg/m^3 the r value is −0.1073, again showing a negative but weak relationship. The higher the concentration, the less percentage of particulate is captured in the rinse. Although this correlation is weak, it does still suggest, as with earlier findings, that the losses on the filter are due to the handling and measurement process. When a lower concentration of particulate is seen on the filter, the losses due to handling have a greater impact on the ratio of particulate on the filter to the rinse. More data on the losses associated with the handling of filters are required for statistical reliability.

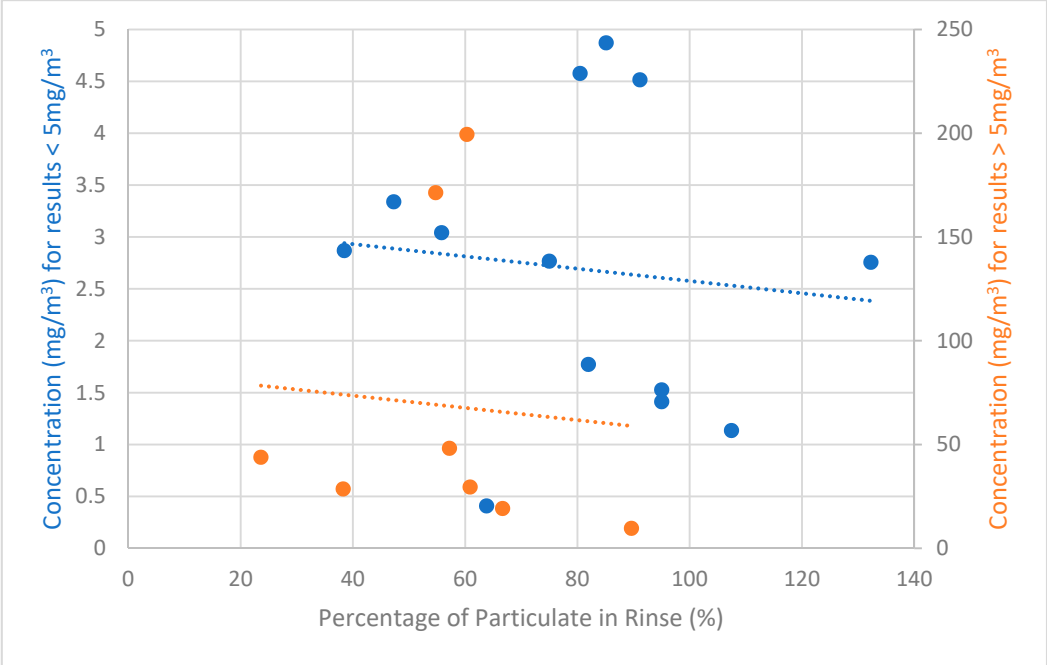

Figure 7. Percentage of mass in rinse (%) and concentration (mg/m^3).

An industrial survey was undertaken to analyse opinions of professionals working within the industry. All participants currently work within the emissions monitoring industry and use SRM equipment. The survey was used to determine the current position of the industry, taking the perspectives of experienced users of monitoring equipment to determine the feasibility of using the SRM to extract a representative sample. The survey was designed with 20 questions for all participants and included both open and closed format question. A total of 732 responses to the questions were recorded. The topics covered include the perceptions of the industry, the roles and experience of participants within the industry, details of the measurement of particulate emissions, requirements for monitoring, and issues/improvements with monitoring equipment. The analysis of results focuses upon the perceptions of participants regarding issues relating to monitoring equipment and thoughts regarding improvement. Current technology is not capable of the measurement of current emission limits, with participants stating that the practical limit of detection for the SRM is around 5 mg/m^3. Many processes are operating at lower mass concentrations than 5 mg/m^3, with 62% of processes analysed in this project reporting these low values. Poor application of equipment is seen in the field, which is said to result is low quality data. Contamination of the sample is highlighted as an issue with poor application, with better cleaning procedures suggested as a method to improve contamination issues. Performing the rinse procedure on site in harsh conditions can significantly influence the accuracy of results. When dealing with such low mass concentrations, the inconsistencies with the measurement technique can have considerable effects on accurately reporting results. It was noted that where there are high levels of ambient dust, the accuracy of results is negatively affected, suggesting that ambient dust contaminates the measured sample at the measurement stage. Using sealed filters was suggested as a methodology to overcome issues relating to damaged filters and filter losses. Whilst sealing a filter would reduce losses, the contamination issue still exists regarding the rinse procedure. It was suggested that sampling flow rates should be increased to increase the sample volume, although this would involve a change in design of the probe. As highlighted by Antonsson [10], the influence of any interruption of flow within the stack influences results up to 13%, and larger probes would only worsen these effects. BS EN 13284-1 is written as a guide to perform SRM measurements from stack processes. The equipment used to undertake these measurements is bulky and difficult to navigate the harsh conditions called upon by stack

emitting processes. Regardless of the quality of implementation, there are limitations to the SRM, the key point being its inability to accurately measure processes with emissions levels <5 mg/m^3. The process of employing the SRM introduces errors, influencing the quality and accuracy of results. Alternative methods of analysis are available, but the process of extracting a representative sample limits the reliability and potential of these new technologies. The SRM is a critical process to accurately measure particulate emissions from a plant process. The characteristics of dust particulate emissions, including the diameter, size distribution, color, shape, and chemical composition make the selection of equipment challenging. For instruments that measure a parameter of particulate in such unstable conditions, it is not possible for manufactures to calibrate instruments that can be installed on any process and give accurate representative results. The requirement of the SRM is to provide an unbiased, representative measurement of the particulate concentration in the stack.

5. Conclusions

This research project analysed data from an industrial survey and 21 UK sites with data obtained by implementing methodology available through BS EN 13284-1, known as the SRM, for the measurement of particulate for the purposes of calibrating CEMS. There are several issues surrounding the SRM, including the validation of measurements < 5 mg/m^3, introduction of losses through the harsh measurement environment, environmental influences (especially where high levels of ambient dust are present), the influence of the probe inside the stack (which can contribute to up to 13% overreporting), and difficulties with the rinse and clean procedure to obtain accurate results.

The data collected in this research show very weak relationships between any of the measured parameters required for SRM testing, and the percentage of particulate in the rinse including the stack velocity, isokinetic percentage, sample volume, and the total mass concentration. The issue with particulate deposits collected in the sample train limits the use of isokinetic sampling to obtain a representative sample for use with alternative analysis methods. Using alternative and new methods of measurement, especially when developing systems to be considered as a reference method, takes considerable collaboration and integration with policies and standards. This research has highlighted that there are potential problems with achieving a representative sample using the isokinetic process in its current form for use with new technologies to analyse an extracted sample.

There are possible applications where the procedure detailed in BS EN 13284-1 can be used with alternative measurement techniques. Where continuous analysis techniques are being developed to integrate with extractive isokinetic sampling techniques, the possibility of a full stack profile can be undertaken to output the percentage of particulate found at each point in the stack. The problem with particulate captured within the sample train is still present but by giving a percentage profile across a stack, valuable data can be obtained. When installing gas analysers for the purposes of emissions measurement, a stack profile can be obtained to identify the best location for CEMS to be installed. This is currently not possible with the SRM and is difficult with any other available techniques. Using this method would allow a full profile to be supplied for the purposes of accurate CEMS location, although this would not provide any quantitative data for particulate emissions as the issues with sample train capture are still present. This research shows that there are difficulties with the SRM in achieving a representative sample, but further research opportunities are highlighted for the future integration of these processes. Automating the filter measurement process to produce an online, real-time measurement would assist the users of measurement equipment, knowing when sufficient sample has been measured to achieve reliable results. This would not completely solve problems associated with the SRM procedure; however, some errors associated with the handling of filters could be eliminated. The process of the rinse procedure was highlighted in the survey as being difficult. Significant improvement is suggested to automate the manual technique currently used. Through the automation of a rinse procedure, many of these issues could be overcome,

including the ability to use the isokinetic sampling method integrated with an automated rinse as a system for extracting an accurate representative sample.

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Abbreviations

CEMS	Continuous Emission Monitoring Systems
SRM	Standard Reference Method
ELV	Emission Limit Value
UK	United Kingdom
BS	British Standard
EN	European Norm
QAL	Quality Assurance Level
MCPD	Medium Combustion Plant Directive
IED	Industrial Emissions Directive
CO_2	Carbon dioxide
ISO	International Organisation for Standardization
EPA	Environmental Protection Agency
US	United States
NPL	National Physical Laboratory

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