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Study into the loss of material from filters used for collecting particulate matter during stack emissions monitoring

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ABSTRACT

This report presents the results of a study carried out by NPL for the Environment Agency of England and Wales to investigate issues related to the measurement of particulate emissions from stacks. The study involved a series of laboratory tests on the performance of a range of filters and filter holders, in particular attempting to assess possible losses from filters during their use. The study was used to develop proposed guidance to help improve particulate emissions measurements.

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Approved on behalf of the Managing Director, NPL
by Martyn Sene, Director, *Quality of Life Division*

Executive Summary

This report covers work carried out for the Environment Agency of England and Wales (EA) to investigate filter performance. The project involved laboratory testing of a range of filters and filter holders used for particulate emissions monitoring. Previous work carried out by NPL under a DTI Joint Industry Project in collaboration with the Source Testing Association (STA) had identified potential losses from filter papers used in particulate sampling.

In order to investigate these potential losses NPL undertook a study to measure losses from filters. A number of instrument suppliers provided example filter holders and filter papers, and these were tested using air drawn from the NPL stack simulator containing humidified stack gases with no particulates. The aim was to study losses from the filters, in the absence of particulate matter. Significant losses were observed in all filter holders tested and in all filters, in the range of 0.3 to 1.4 mg. Flow rate did not appear to affect the mass loss, within the statistical variability of the measurements. Significant losses appeared to be a result of the handling of the filters, particularly due to placing them into and out of the filter holders. Filters produced using a new laser-cutting technique appear to lose material due to mechanical damage caused during installation in the filter holder.

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1 Introduction

This project involved a comprehensive laboratory study of the performance of filters and filter holders used in the monitoring of industrial emissions.

Previous work carried out identified issues with particulate sampling in the UK, associated with losses from the filter papers used to collect the sampled particulate matter. The work programme was therefore designed to clarify some of the issues raised in previous work. [1]

Recent work has raised concerns over the use of the reference method for low dust monitoring (EN 13284-1). The work programme has taken account of:

- a joint industry programme study carried out by NPL and the STA which identified filter loss issues; [JIP Project 280206]
- reports from manufacturers on poor performance of EN 13284-1 when used to calibrate particulate monitors; [reported in minutes of STA quality group - Document No QT2018-06]
- some recent filter loss measurements made by CES; [Document No QT2018-06]
- and the general experience of STA members, including stack teams and operators. [Document No QT2018-06]

In summary this work indicates that there are observable losses from filters when used in accordance with common practice in the UK. In particular there appears to be an initial loss of material from filters, of the order of 0.5 mg, consistent with a loss of loose filter material. Initial investigations suggest that this is removed with a relatively short conditioning period (5 minutes). A loss of 0.5 mg should be compared to a typical filter mass gain for a low dust measurement of between 1 to 5 mg of material.

Further concerns relate to the design and performance of the backing plates used to support the filters during sampling. Discussions with filter manufacturers have reinforced these concerns.

In addition filter handling would appear to be a major source of uncertainty in particulate monitoring, often leading to structural damage to the filters and the potential for further loss of material from the filters.

2 Background

Stack gas sampling for particulate monitoring is carried out using an extractive sampling train. The filter can either be mounted in-stack or out of stack. The tests in this project have concentrated on in-stack filters and filter holders. There are a number of different filter types and filter holder designs in use in the UK.

Measurements of particulates in emissions form an important part of the regulation of pollution from industry. A number of European Directives mandate such

measurements, including the Large Combustion Plant Directive [2] and the Waste Incineration Directive [3]. These Directives place data quality objectives on the reporting of emissions. CEN, the European Standards body, have produced standard reference methods (SRMs) that provide a means to achieve these objectives.

Monitoring of stack emissions is undertaken with continuous emissions monitors (CEMs) installed permanently on the stack and by periodic stack sampling carried out by emissions monitoring teams. Where continuous measurements are required the measurements are undertaken within a data quality control framework. This is defined within the European standard EN 14181 and EN 13284 Part 2 [4]. The overall approach consists of a series of quality assurance levels as follows:

QAL 1 : assessment of the CEM by laboratory and field type approval

QAL 2 : calibration of an installed CEM by parallel measurements with an SRM

QAL 3 : continuous assessment of the CEM by regular zero and span checks

AST : annual surveillance tests which involve further tests against an SRM

As can be seen, the use of periodic SRM measurements is integral to the quality assurance of the CEMs. These provide calibration and traceability of the continuous monitoring data.

2.1 Work Programme

A programme of tests was devised to assess the filter and filter holder losses. These tests were designed to investigate potential losses from filters which had been observed in the previous studies. The experimental configuration for the tests is shown in Figure 1. An example of the test configuration is shown in Figure 2. Conditioned stack gas was drawn into the filters from the NPL stack testing facility, Figure 3. This facility is able to generate a range of stack gases, with typical gas concentrations, humidity and flow rates. The gas was clean and did not contain particulate matter. The purpose of the tests was to determine if losses occurred due to material being removed from the filters during sampling.

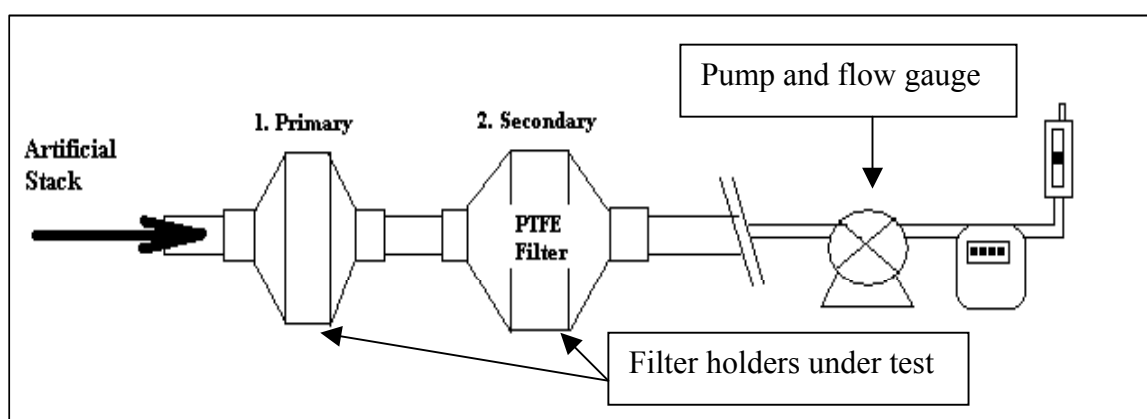


Figure 1. Experimental configuration used during filter tests.



Figure 2. Example of the configuration of a filter holder under test (in this case an Apex filter holder in line with a Tecora filter holder, with a PTFE filter).



Figure 3. The NPL stack simulator.

The laboratory tests involved assessment of the losses from glass fibre and quartz filters from four filter holders, with different filter backing, together with an assessment of the efficiency of a proposed filter pre-conditioning approach.

Filter holders tested were:

- JS Holdings, with a coarse backing (JS Coarse)
- JS Holdings with a fine mesh backing (JS Fine)
- AGL Air Testing (AGL)
- Westech Instrument Services (Westech)
- TCR Tecora (Tecora)

The following types of filters were tested:

- Quartz Fibre filters from Whatman – (Fibre Type 1)

- Quartz Fibre filters from Anachem – (Fibre Type 2)
- Laser cut Quartz fibre filters from Whatman – (Laser Cut)

The standard fibre filters from Whatman and Anachem are stamp cut. The laser cut production technique provides a cleaner cut with, presumably less mechanical damage to the filter. However, as will be seen, the laser cutting method appears to produce a fused rim to the filter, which potentially causes specific losses from these types of filters. Manufacturers have indicated that the use of laser cutting will become the preferred technique in the future, which means this type of filter is likely to become more common.

Before each test the filters were weighed in accordance with the procedure in EN 13284-1 [4]. This procedure involves placing the filters in an oven at 180 degC, allowing to cool in a desiccator, prior to weighing. The filter is weighed at 1, 2 and 3 minute intervals, so that the weight can be extrapolated to time zero, using linear regression.

The filters were then carefully mounted in the filter holders, and tested according to the test plan.

At least two filters were used as weighing blanks during each experiment. These were conditioned in the same way as the filters under test, but remained in the dessicator during the testing period and were re-conditioned and weighed after the tests. Differences in the weighing blanks pre and post testing were examined, as they can be used to normalise the weighing results. However, no significant changes were observed. After each experiment the test filters were re-conditioned (in the oven and desiccator) and re-weighed in accordance with the procedure in EN 13284-1. Filters were handled carefully during the tests, under laboratory conditions, to avoid losses during the weighing procedure itself.

During testing a second filter holder was used in-line to collect material lost from the filter under test. Conditioned PTFE filters were used for this. A series of pre-tests were carried out to check that the PTFE filters did not exhibit losses. No losses were observed with the PTFE filters. The masses collected on these filters during the subsequent test programme was used as a check that material really was being lost from the filters under test. PTFE filters are not normally used for stack emissions monitoring because they cannot be used at high temperatures. Concerns have also been expressed that they can become clogged with high dust loading.

The results from any filters which showed physical damage, such as tears, were not included in the study.

The Apex filter holder is US EPA Method 5 compliant. The JS Holdings filter holder was designed to meet the requirements of the CEN standard BS EN 13284-1.

The Apex and TCR Tecora filter holders used plates with holes drilled in them as the backing support for the filters (Figure 4). The JS Holdings filter holder employed a wire-style filter support.

The filters were conditioned and weighed prior to testing according to the requirements of BS EN 13284:1. The standard test configuration was then to place the filters in the holders and to draw air from the test rig through the filters at 30 l/min for approximately 15 minutes. The filters were then removed, and conditioned and weighed again in accordance with BS EN 13284:1. Reference filters were also weighed before and after each test.



*Figure 4, Examples of filter holders tested
Top left, Apex, Top right, TCR Tecora, Bottom left, JS Holdings.*

2.1.1 Preparation

Before each test the filters were conditioned (by placing in an oven and desiccator prior to weighing) and pre-weighed according to EN 13284-1. Following weighing they were mounted in the filter holders.

After each test the filters were re-conditioned and re-weighed in accordance with EN 13284-1.

Filters were handled under laboratory conditions, to avoid losses during the weighing procedure itself. Weighing was carried out in accordance with EN 13284-1, in a dedicated weighing room. A Mettler AT261 balance was used. Reference weights and reference filters were weighed before each set of tests.

2.1.2 Filter loss with different holders / filters

Five tests were carried out using each type of filter on each type of filter holder. Air was drawn through the filters at typical flow rates for UK sampling (25 l/min) for a period of 15 minutes.

2.1.3 Effect of continued flow

A selected filter holder / filter type combination was tested for a period of 30 minutes to assess whether losses are correlated with sampling time, i.e. whether there appeared to be further loss from the filters with extended test time.

2.1.4 Effect of weighing procedure

A selected filter holder / filter type combination was oven-dried and pre-weighed and tested for a further 10 minutes. This aimed to assess whether the pre-weighing and conditioning procedure, in particular the oven conditioning, resulted in filter degradation leading to further loss of material when subjected to an air flow.

2.1.5 Effect of higher flow rate

A set of filters was prepared and pre-weighed, and then tested using a higher test flow rate (45 l/min).

2.1.6 Effect of filter handling

A series of tests were carried out to assess the effect of filter handling, and in particular the effect of placing a filter paper into a holder and then removing it. It had been proposed to perform this test using only one filter holder and filter type. However, following the initial testing, and the indication that the laser cut filters may have suffered from significant losses due to mechanical damage, these tests were carried out using different filter holders and filter types.

2.1.7 Test of pre-conditioning procedure

A set of filters were preconditioned by passing air through them prior to the pre-weighing procedure. It had been initially proposed that these tests would be carried out on a single filter type and filter holder. However, due to the variability seen in the performance of the filters following the first phase of testing, it was decided to extend this test to include all filter types and filter holders.

The pre-conditioning phase consisted of placing the filters in a filter holder and drawing ambient laboratory air through them for a minimum of 5 minutes at a flow rate of nominally 30 l/min. In order to reduce the possibility of contamination from material in the air, a second filter holder containing a PTFE filter (which has been demonstrated not to lose material) was placed upstream of the filter. This procedure was carried out before the pre-weighing procedure for the filters.

These filters were then tested for losses in the same manner as above, by following the EN 13284-1 pre-weighing procedure, then running the filters for 15 minutes at 30 l/min and then following the EN 13284-1 post-weighing procedure.

2.1.8 Effect of standard leak test

An additional test was added during the testing programme, to check whether the standard leak test procedure used during stack sampling has an effect on the filters. In this test a set of filters were weighed and then placed in the filter holder and a leak test

simulated. This imparts a pressure pulse on the filter, potentially causing further material loss.

2.2 Results of filter testing

The following sections present the results of the filter testing.

Filter loss with different holders / filters

The following table (Table 1) summarises the results of the series of tests on different filter and filter holder combinations. Each result is the average change in mass (mg) for sets of five filters all tested in the same way.

Filter holder	Filter	Average Mass Change mg
AGL	Fibre Type 1	-0.21
AGL	Laser Cut	-0.29
AGL	Fibre Type 2	-0.25
AVERAGE AGL		-0.25
JSH FINE	Fibre Type 1	-0.09
JSH FINE	Laser Cut	-0.53
JSH FINE	Fibre Type 2	-0.29
AVERAGE JSH FINE		-0.30
WESTECH	Fibre Type 1	-0.27
WESTECH	Laser Cut	-0.28
WESTECH	Fibre Type 2	-0.07
AVERAGE WESTECH		-0.21
TECORA	Fibre Type 1	-0.39
TECORA	Laser Cut	-1.44
TECORA	Fibre Type 2	-0.27
AVERAGE TECORA		-0.70
JSH COARSE	Fibre Type 1	-0.23
JSH COARSE	Laser Cut	-0.23
JSH COARSE	Fibre Type 2	-0.29
AVERAGE JSH COARSE		-0.25
Average Fibre Type 1		-0.24
Average Laser Cut		-0.55
Average Fibre Type 2		-0.23

Table 1, Average results of sets of different filters and filter holders

The maximum losses were observed with the laser cut filters with the Tecora filter holder. Examination of the laser cut filters showed a raised edge presumably due to the effect of the laser melting the filter material. It is proposed that the reason the laser

filters in general showed high losses is due to mechanical damage to the fused edge, when the filter is clamped in the holder. The clamping ring of the Tecora filter holder design may exacerbate this effect, by pressing on the filter edge.

Effect of continued flow.

No statistical difference in the losses were observed between the short and long periods of testing (15 and 30 minutes) carried out using Fibre Type 1 and 2 filters in the Tecora and JS Holdings coarse filter holders. This provides evidence that, within the variability of the observed losses, the loss of material from the filters is **not continuous**, and this supports the use of a short pre-blow to remove loose material from the filters.

Effect of higher flow rate

A set of five fibre filters were tested for 15 minutes with a flow rate of 45 l/min in a Tecora filter holder. No statistical difference in the losses were observed between tests at a flow rate of 30 l/min and 45 l/min.

Effect of weighing procedure

Filters, which were retested (for 15 minutes 25 l/min flow), after having been weighed according to the weighing scheme, were observed to still exhibit losses, although not in every case. This implies that the weighing process may in some cases cause structural effects to the filters causing more material to be loosened and subsequently lost. It is unclear whether this is due to the oven heating or the mechanical handling of the filters – though the mechanical handling tests indicate that potentially the losses with laser cut filters could have been caused by mechanical loss only.

It is also possible that the orientation of the filters in the filter holders could explain the losses. If the filters have been placed in the filter holders in a different orientation for the second test after weighing (i.e. with the faces reversed), this could in principle allow material which was on the front of the filter for the first test to have subsequently been lost during the second test. This would explain why in some tests losses were observed and in others they were not.

Effect of filter handling

Table 2 presents the results from a series of tests to examine the effect of filter handling, by placing the filters into and out of the filter holders, without passing any flow through them. In general, small losses were observed. However, the laser cut filters exhibit losses when used in the filter holders which firmly clamped the edges of the filter. It is possible that the fused lip of the filter, caused by the laser cutting process, was crushed by the filter clamp. It was also observed that this loss was not seen as a positive gain on the secondary filter. This can be explained by the crushed filter material being held in the filter holder, and only being lost from the filter when the filter is removed prior to weighing.

Filter Holder	Filter Type	Average Filter Weight Difference
		mg
TECORA	Fibre Type 1	0.01
TECORA	Laser Cut	-0.43
TECORA	Fibre Type 2	-0.03
WESTECH	Fibre Type 1	-0.05
WESTECH	Laser Cut	0.07
WESTECH	Fibre Type 2	-0.02
AGL	Fibre Type 1	-0.07
AGL	Laser Cut	-0.14
AGL		0.03
JSH COARSE	Fibre Type 1	-0.01
JSH COARSE	Laser Cut	0.03
JSH COARSE	Fibre Type 2	0.09

Table 2, Results of tests on handling losses for sets of filters

Test of pre-conditioning procedure

The results of the tests on the pre-conditioning procedure are presented in Table 3. As described previously the pre-conditioning procedure was carried out by placing the filters in a filter holder and drawing 30 l/min of laboratory air through the filters for 5 minutes. These show that although small losses are still observed with the pre-conditioned filters, there are no further losses when the filter is run for longer. The unconditioned filters continued to lose material, though at a small level.

FILTER HOUSING: JSH COARSE		Pre Blown mg		No Pre Blow mg
LOSSES FROM	Fibre Type 1	-0.09	Fibre Type 1	0.03
RUNNING FILTER	Fibre Type 1	-0.11	Fibre Type 1	-0.06
FOR 15 MINS AT 25	Laser Cut	-0.17	Laser Cut	-0.14
L/MIN	Laser Cut	-0.15	Laser Cut	-0.29
LOSSES FROM	Fibre Type 1	0.01	Fibre Type 1	-0.11
RUNNING FILTER	Fibre Type 1	0.07	Fibre Type 1	-0.01
FOR A FURTHER 15	Laser Cut	0.00	Laser Cut	-0.04
MINS AT 25 L/MIN	Laser Cut	0.00	Laser Cut	-0.08

Overall losses	Fibre Type 1	-0.08	Fibre Type 1	-0.08
(sum of each stage)	Fibre Type 1	-0.04	Fibre Type 1	-0.06
	Laser Cut	-0.17	Laser Cut	-0.18
	Laser Cut	-0.15	Laser Cut	-0.37

Table 3, Test on the efficacy of the pre-conditioning of filters

Effect of standard leak test

The tests on the effect of the initial leak check have produced some surprising results, which are hard to explain (Table 4). The filters which had been pre-conditioned with the pre-blow appear to lose material during the leak test. The large loss from the laser filters may in part be due to the previously discussed possibility of mechanical damage to the filter edge. The unconditioned filters however, produced some currently unexplained results with a consistent weight gain. Further work is required to explain these results.

FILTER HOUSING: JSH COARSE			
Filter Type	Conditioned Filter		Unconditioned Filters
	Weight Difference		Weight Difference
	Mg		mg
Fibre Type 1	-0.17	Fibre Type 1	0.22
Fibre Type 1	-0.16	Fibre Type 1	0.24
Laser	-1.66	Fibre Type 1	0.34
Laser	-1.45	Laser	0.44
		Laser	0.19

Table 4, Losses from initial leak check procedure

3 Conclusions and Recommendations

The experimental investigation of filter losses identified significant losses from filters when clean dry air was drawn through them. These losses are potentially up to 50% of the mass of particulate collected when sampling low dust conditions (plant with dust concentrations of below 5 mg/m³).

Careful handling of filters is important. With all types of filter holders tested it was found that it was very easy to damage the filters when removing them from the support. Such damaged filters were not included in the reported results – the reported results are for filters which appeared to be intact and undamaged.

Extreme care is also required during the weighing procedure, even under conditions which meet all requirements of the relevant measurement standard (EN 13284-1), it was noted that operator actions could easily affect the weighing results. It is clear that stringent control of weighing procedures is necessary to achieve the weighing uncertainties appropriate for the measurement of low levels of particulate.

Laser cut filters appear to suffer from significant mechanical damage. Until this issue is addressed it is recommended that laser cut filters are not used.

The use of a 5 minute 30 l/min pre-blow conditioning appears to reduce the effect of filter material loss. An issue relating to ensuring the filters are installed in the filter housing in the same direction as they were pre-blown remains to be clarified.

There did not appear to be a significant impact due to the design of the filter holders, although there was some effect on the degree of mechanical loss.

A filter cartridge approach, in which a support structure is used to reduce the loss of material from the filter and reduce mechanical damage, is being developed by NPL. This aims to address a number of issues related to mechanical material loss, such as that associated with laser cut filters.

3.1 Acknowledgements

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