

METROLOGY for HYDROGEN VEHICLES

REPORT:

D3: Good practice guide for the handling, transporting and weighing of filters from particulate sampling in gaseous hydrogen

EMPIR



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1 - Introduction

1.1 Task objectives under MetroHyVe

The aim of this task was to develop laboratory traceability for performing offline mass measurements of particulates collected on filters provided by Hydrogen Refuelling Stations (HRSs). A new method was developed to traceably determine the mass concentrations of particles on a filter collected in a hydrogen stream. The effect of ambient conditions at refueling stations during filter preparations was examined. These were used to validate the commonly used procedure for measuring particle mass concentration at the HRS. Tests were performed to assess the suitability of current methods, and the results were used to develop this guide with a particular focus on traceability, accuracy and good laboratory practice.

1.2 Scope of the Good Practice Guide

Particulate contamination can be found in hydrogen and may be introduced during of the hydrogen gas or from the degradation of transportation and storage equipment [3], [4], [5], [6]. These particles can be destructive to fuel cells so efforts must be made to monitor, fully characterize and ultimately eliminate them. A traceable, reliable and repeatable method for the determination of the mass concentration of the particulates in a volume of gas is required. Filters have long been used to collect and measure particulate matter in the area of ambient air monitoring and can, therefore, be considered as a reliable method to measure the particulate matter in a gaseous medium. This Good Practice Guide is intended to be used as a helpful document for those wishing to set up their own measurement system for the determination of particulates in hydrogen. This guide will highlight the considerations, quality control and procedures required for weighing particulates sampled from gaseous hydrogen, from a filter weighing methodology. It will also describe a method for the accurate measurement of the collected particulate mass on filters traditionally used for ambient air monitoring. It meets the requirements for filter storage; conditioning and weighing that are laid out in EN 12341:2014 [7]. It also meets the detection limits stipulated in Grade D of ISO 14687[1] which provides the technical specifications for hydrogen intended for fuel cell vehicles.

1.3 Particulate contamination causes and issues

The concentrations of impurities in fuel grade hydrogen gas must be tightly controlled and carefully monitored to ensure the optimum operation of vehicles which utilize hydrogen as a fuel. The limits for various impurities have been set by ISO 14687 Grade D, with a limit of 1 mg m⁻³ set for particles [1]. To date, there are no online, real-time measurement techniques for the measurement of the particulate content of a hydrogen gas stream. The standard method is to

collect particles on a pre-weighed filter and determine the mass of particles in the gas stream by reweighing after sampling [2].

Particles in the hydrogen gas can originate from a number of different sources, potentially related to the initial fuel source, production methods and storage methods [3], [4], [5], [6]. Contamination of the initial fuel source used for the production of hydrogen gas, with compounds such as water, ammonia, sulphur and formic acid [3] can lead to the formation of acids or salts within the gas stream or on the surfaces of storage units or fuel cell components [4], [5], [6]. These impurities must be removed in order to reduce the potential degradation of the fuel cell and potentially the corrosion of storage containers which could potentially introduce metallic particles into the gas stream [4]. The production process also has the potential to introduce particles into the fuel gas directly. For example, the pyrolysis and gasification of biomass to produce hydrogen gas can lead to tar formation, this could potentially lead to carbonaceous particles occurring in the gas stream [6].

In order to minimize the concentrations of particles in the hydrogen gas stream, rigorous decontamination of the initial fuel source and post generation hydrogen must be carried out. The selection of appropriate storage and transportation methods could also potentially minimize post generation particle contamination. Particle contamination of hydrogen fuel can potentially have serious operational, financial and health repercussions. Decreased performance of the fuel cell or the degradation of the fuel cell components, could lead to significant environmental and public health issues in terms of emissions from the vehicle. Rectifying these issues in the fuel cell represents a significant financial consequence as a result of contaminated fuel.

2 – Filter weighing procedure

2.1 Considerations

These considerations originate from NPL's process in creating a weighing system and procedure in accordance with the standard EN12341:2014 [7].

- All filters are weighed on high-accuracy single-pan balances in a temperature and relative humidity-controlled environment.
- The range of application for this procedure is from 25 µg to 8 mg of collected particulate, which represents measured concentrations in the order of 1 µg.m⁻³ dependent on exposure time and flow rate.
- The absolute combined filter and particulate mass must not exceed 300 mg. The procedure is suitable for the weighing of filters of any material, with diameters of either 37 mm or 47 mm. The procedure has been developed at NPL using a single pan balance,

Mettler XP2U (0.1 µg resolution). This balance is calibrated by NPL's mass section on an annual basis. ISO 14687 [1] states that the recommended gravimetric method has a detection limit of 5 µg/Kg. Assuming sampling at 700 Bar pressure this equates to 5 µg/25L or 0.2 µg/L. At a resolution of 0.1 µg for the balance this criterion is met at a sample size of 0.5 L. Such a small sample size, however, is not recommended, as the larger the sample size the more representative of the particulate concentration of the stored hydrogen due to its homogeneity. Also, additional uncertainty components, examined later, will reduce the effective resolution of the measurement. This simple calculation, however, is enough to show that this is an appropriate method following ISO 14687 [1].

- To prevent the loss of volatile and semi-volatile species, all sampled filters shall be stored at a temperature of ≤ 23 °C, in accordance with EN12341:2014 [7].
- Static electrical charge is known to have a potentially significant effect on the weighing of filters, especially those made of PTFE. Trials have shown that the use of the Faraday cage weighing pan reduces this effect to within allowable tolerances. If this were not the case it would be apparent from repeat weighings and a failure to reach a stable weight. The standard weighing pan has shown to be acceptable for Emfab and glass fibre filters. However, fibre filters are not recommended as discussed later in this document.

2.2 Equipment

The equipment used in this practice are as follows:

2.2.1 Autohandler

The design of the autohandler (Figure 1) built by MTL (Measurement Technology Laboratories) allows for the circulation of air around the top and bottom surfaces for all artefacts to be weighed, ensuring that they are fully conditioned at the desired temperature and humidity. Sample filters, calibration weights and quality control filters are delivered from their storage positions to the balance via a barcode reader for artefact identification. The autohandler should be serviced on a regular basis to ensure that performance is not compromised. This service interval will depend on usage but will be annually during typical regular use. For users looking for a currently available system similar to that at NPL, the AH500E System from MTL would be an option.

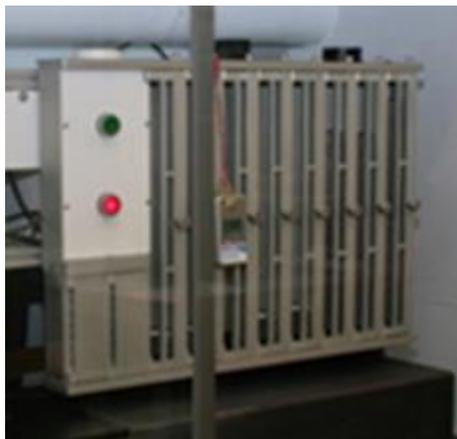


Figure 1: Autohandler used for filter weighing at NPL

2.2.2 Balance

A balance with a resolution of $0.1 \mu\text{g}$ should be used (e.g. Mettler Toledo XP2U) and a MTL designed Faraday cage style weighing pan (Figure 2) can be used to reduce the effects of static charges. Effect of static is negligible for Emfab filters but needs to be considered for PTFE/PFA filters and to a lesser extent quartz filters.

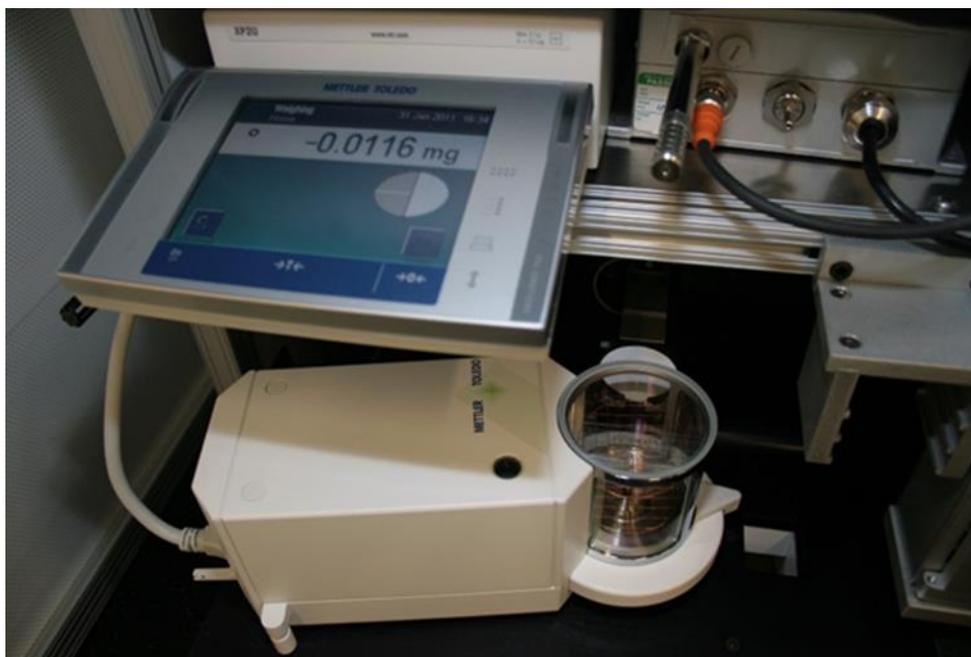


Figure 2: Mettler Toledo XP2U, balance used for filter weighing at NPL [8]

2.2.3 Temperature and Humidity Controlled Chamber

A climate-controlled chamber encloses the autohandler and shall be used for conditioning and weighing the filters (Figure 3). The temperature and the relative humidity shall be continuously monitored and controlled to $20\text{ °C} \pm 1\text{ °C}$ and $47.5\% \pm 2.5\%$ RH respectively, measured as an hourly mean value. A temperature and humidity logger is used to record these conditions during filter conditioning and weighing.

The interior of the chamber shall be cleaned after a break in regular use and quarterly during regular use to remove the build-up of waste particulate matter within the chamber. The chamber should be serviced annually during regular use to ensure that the temperature and humidity control is not compromised.



Figure 3: Chamber used for filter weighing at NPL

2.2.4 Filters

This procedure covers the weighing of filters made from the following materials: quartz, glass fibre, PTFE and PTFE coated glass fibre. Filter diameters can be either 37 mm or 47 mm. Other filter diameters can be weighed if suitable filter holders are used.

During the feasibility testing stage to determine whether the weighing procedure was suitable for hydrogen gas both porous PTFE (Figure 4) and fibrous PTFE filters (Figure 5) were used. It was

found that the fibrous filters were unsuitable as the hydrogen appeared to get in between the fibres causing the filters to swell. Originally, we believed that elevated pressures were to blame but the same swelling was seen when ambient conditions were used in the laboratory. This swelling had two effects firstly, the filters also curled up at the edges meaning that it was not possible to use the robot weigher. Secondly, this swelling degraded the structural integrity of the filter making handling very difficult with a high chance that some filter material could be lost.



Figure 4: (left) PTFE porous filters in their packaging case and (right) quartz fibre filters [9]

In Figure 5 the degradation of a PTFE glass fibre filter can be seen in the right image.



Figure 5: (left) PTFE glass fibre filter and (right) filter exposed to hydrogen

2.3 Filter weighing procedure

The weighing procedure here is designed to mitigate both long- and short-term drift in the balance being used. To this end both weights internal and external to the balance are used. Traceability to the SI unit the Kilogram is assured through an annual calibration in the NPL Mass and Measurement area. This traceability is key to ensure that the measurements taken using this procedure are comparable between laboratories, that data is defensible and that results meet accredited standards required by industry. The procedure for this annual calibration is not included here.

All weighings on the system are performed according to the following protocol:

1. Zero balance
2. Record empty pan weight, E_n
3. Place artefact (A) to be weighed on the balance pan
4. Record artefact weight, A_n
5. Remove artefact from balance pan
6. Record empty pan weight, E_{n+1}

Steps 2 to 6 can be repeated as many times as required to reduce the uncertainty in the measured weight of the artefact.

The weight of A is calculated from Equation 1:

$$A = \frac{\sum_1^n \left(A_n - \left(\frac{E_n + E_{n+1}}{2} \right) \right)}{n}$$

Equation 1

where:

- A = weight of artefact in mg
- A_n = weight of artefact at the nth weighing in mg
- E_n = empty pan weight before the A_n weighing in mg
- E_{n+1} = empty pan weight before the A_{n+1} weighing in mg
- n = number of weighings

For test weights $n = 1$, and for test filters and reference filters $n = 3$.

The first step of any weighing procedure is to warm up the balance to prepare the balance for weighing. This is achieved by weighing the 100 mg test weight 10 times.

After the balance has been warmed up, and every four hours, the following quality assurance protocol is run using the internal masses within the balance and two external test weights.

1. Weigh 100 mg test weight
2. Perform internal balance calibration
3. Weigh 100 mg test weight
4. Weigh 200 mg test weight

Two reference filters of the same material as the test filters are weighed at the beginning of the weighing procedure. These reference filters are always kept within the climate control chamber and are used as an indication of the system performance. The difference between the weights of the check filter between weighing sessions shall not exceed the 40 µg for unsampled filters and 60 µg for sampled filters, criteria for weight difference for filter weights. These requirements come from EN12341 [7], gravimetric PM sampling.

2.4 Calculations

The unloaded or loaded filter weights are calculated according to Equation 2

$$m_{\text{unloaded}} \text{ or } m_{\text{loaded}} = \frac{(m_{t=0} + m_{t+24hr})}{2}$$

Equation 2

where:

m_{unloaded}	=	mass of unloaded filter (unsampled), µg
m_{loaded}	=	mass of particulate loaded filter (sampled), µg
$m_{t=0}$	=	mass measurement performed after 48 hrs of conditioning, µg
$m_{t=24}$	=	mass measurement performed after an additional 24 hrs of conditioning, µg

The collected mass on a filter is calculated according to Equation 3:

$$\text{Collected Mass} = m_{\text{loaded}} - m_{\text{unloaded}}$$

Equation 3

where:

<i>Collected Mass</i>	=	mass of particulate matter collected on the filter, µg.
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NPL weigh each filter 3 times each weighing session.

The particulate mass concentration is calculated according to the Equation 4:

$$\textit{Particulate concentration} = \frac{\textit{Collected Mass}}{\textit{Sample Volume}}$$

Equation 4

where:

Particulate Concentration = concentration of particles in a gas sampled, $\mu\text{g}\cdot\text{m}^{-3}$
Sample Volume = volume of gas sampled by the filter, m^3

2.5 Quality Assurance and Quality Control

- If the two weighings of an unsampled filter differ by more than 40 μg then that particular filter should be discarded.
- Unloaded filters may be stored for up to 2 months before sampling.
- If the two weighings of a sampled filter differ by more than 60 μg then the result for that particular filter will be considered invalid.
- Sampled filters will be stored at a temperature of ≤ 23 °C before they are conditioned for weighing. After weighing, the filters will be left in the autohandler until it is confirmed that the results meet difference in mass criteria over a 24-hour period stated in EN12341 (40 μg and 60 μg for unsampled and sampled filters respectively) and then stored in an air-conditioned laboratory.
- The maximum allowable change in humidity during conditioning and weighing is 5% Rh, as stated in EN12341.

2.6 Uncertainty

This section details the possible sources of uncertainty in this measurement and the potential magnitude of their contributions. It should be noted that all of the sources of uncertainty under EN12341 [7] are mentioned here. This is in effect the situation before physical mitigations are applied. Therefore, the $K=2 \pm 85.6$ μg uncertainty is a worst-case scenario for this measurement. For a set up like the one at NPL where a temperature and humidity conditioning process is in place the uncertainties are in the region of 8-9 μg dependent on the filter. As mentioned earlier ISO 14687 [1] states that the recommended gravimetric method has a detection limit of 5 $\mu\text{g}/\text{Kg}$. Assuming sampling at 700 Bar pressure this equates to 5 $\mu\text{g}/25\text{L}$ or 0.2 $\mu\text{g}/\text{L}$. The resolution of the balance is 0.1 μg but there is an uncertainty component of ± 9 μg . Therefore, if we take 10 μg as the real-World detection limit this criterion is met at a sample size of 50 L. The model equation, which represents the procedure for obtaining the desired output quantity from the input quantities, is shown in Equation 5:

$$PM = m_{loaded} - m_{unloaded}$$

Equation 5

where:

- PM = Particulate mass collected on the filter
- m_{loaded} = Mass of the filter after sampling
- $m_{unloaded}$ = Mass on the filter before sampling

It is possible to introduce significant errors in the weight of collected particulate matter because of the loss of filter or particulate material during handling and transport, or the addition of matter not collected during sampling. The procedures for handling, storage and transport are designed to control these factors. The uncertainty of this measurement is not included in the uncertainty budget below.

The individual sources of uncertainty are summarised in Table 1.

Table 1: Table outlining the contributions towards the uncertainty budget and brief explanations on why these must be considered.

Contribution	Symbol	Maximum allowable uncertainty Component, μg	Why this needs to be considered
Mass $m_{loaded} - m_{unloaded}$	u_m		
Loss of semi-volatiles	u_{msv}	Zero by convention	(Zero by convention) It is known that substantial fractions of particulate mass can be composed of semi-volatile components, which can volatilise at any time between entry to the sample inlet and weighing of the loaded filter, at a rate predominantly determined by the temperature of the sampling system and the filter. Hence, some loss of semi-volatile material is expected when this procedure is followed and the loss may differ between the different location, day and PM values. This volatilisation loss is limited within the standard measurement method laid out in EN12341:2014, by the constraints on the sampling system components and on sample storage, transport and conditioning. However, the definition of PM for the purpose of EN12341:2014 incorporates these losses of semi-volatile material. Therefore, the average effect of

			<p>volatilisation losses on the measurement of PM is considered to be zero by convention, when the constraints set out in EN12341:2014 and this procedure are followed.</p> <p>EN12341:2014 specifies sampling should occur within 56 days of the pre-sampling weighing. If the same time period is used for the maximum period between sampling and post weighing then the maximum period between pre and post weighings is 4 months (112 days). If this time period is exceeded, then the mass measurement would not comply with EN12341 and would not be valid.</p>
Effect of humidity on filter	U_{mhf}	$\frac{40}{\sqrt{3}}$	<p>Extensive work on the effect of humidity on filter weight has been performed using the autohandler and conditioning chamber. This validation work on EN12341:2014 is summarised in Annex F of the standard. The humidity weight dependence of Emfab filters was found to be $0.7 \mu\text{g}.\%Rh^{-1}$ and the humidity weight dependence of quartz QMA filters to be $2.8 \mu\text{g}.\%Rh^{-1}$. The range of allowable conditioning humidity is 5% Rh, so the maximum expected filter weight change between pre and post sampling would be $3.5 \mu\text{g}$ for Emfab filters and $14 \mu\text{g}$ for quartz QMA filters. In reality, due to the tight relative humidity and temperature control of the climate chamber ($20 \text{ }^\circ\text{C} \pm 1 \text{ }^\circ\text{C}$ and $47.5\% \pm 2.5\% \text{ RH}$), this dependence, U_{mhf}, is less than $7 \mu\text{g}$ for quartz QMA filters and less than $1.8 \mu\text{g}$ for Emfab filters.</p>
Effect of filter drift	U_{mfd}	$\frac{135}{\sqrt{3}}$	<p>Both Emfab and Quartz QMA filters slowly drift in weight over prolonged periods of time when conditioned at stable temperatures and humidities. This is mainly due to the manufacturing conditions being different from the conditioning conditions. Previous work has shown that Emfab filters always gain weight over time at a rate of $0.3 \mu\text{g}$ per day. While quartz QMA sometimes gain or sometimes lose weight over time, tests have shown a typical drift in quartz QMA filter weight of $1.2 \mu\text{g}$ per day.</p>
Effect of humidity on particulate	U_{mhp}	$\frac{46}{\sqrt{3}}$	<p>The effect of this on the uncertainty of the measurement is quantified by the constraint on mass change at the separate weighings of loaded filters given above ($60 \mu\text{g}$). From tests on the effect of humidity on particulate mass and the maximum allowable change in humidity of 5% Rh, the</p>

			maximum change in particulate mass between the two weighings 24 hours apart is in the order of 46 µg. In reality due to the tight relative humidity and temperature control of the climate chamber (20 °C ± 1 °C and 47.5% ± 2.5% RH), this dependence is less than 10 µg.
Buoyancy	U_{mb}	$\frac{3}{\sqrt{3}}$	The density of the surrounding air, which causes a buoyancy effect on the weight of the filters, is predominantly determined by the air pressure and temperature. Of these only the temperature is controlled as part of the filter condition requirements. However, the expected effects of buoyancy changes can be calculated from physical principles.
Static	U_{ms}	Negligible	(negligible) Static electrical charge is known to have a potentially significant effect on the weighing of filters, especially those made of PTFE. However, the use of the MTL Faraday cage style weighing pan is considered to control this and the associated uncertainty is considered to be negligible. For Emfab and quartz filters the standard balance pan is considered to deliver negligible uncertainty for the effect of static electrical charge.
Balance calibration	U_{mc}	2.7	Yearly calibration of balances is suggested
Balance linearity	U_{mbl}	1	(negligible compared to the mass of particulates on loaded filter)
Balance span drift	U_{mbds}	1	An internal balance recalibration every 4 hours removes the effect of span drift. An external independent weighing a 100 mg weight before and after recalibration is used to monitor the effect of the internal recalibration. The effect of internal recalibration and span drift over 4 hours must be less than 1µg in both cases. Span drift is also monitored by weighing a 200 mg weight in the 4 hourly QA procedure.
Balance zero drift	U_{mbdz}	Negligible	(negligible) Zero drift is controlled by zeroing the balance before an artefact is weighed.
Balance repeatability	U_{mbr}	$\frac{1.0}{t_{(n=8)}}$	(negligible compared to the mass of particulates on loaded filter)

Combined standard uncertainty	u_m	85.6	
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3 - Handling and transport of filters

3.1 Handling

When handling filters and filter holders, clean tweezers and gloves must be used to prevent grease and other contaminants being introduced to the filters. Following the guidelines in EN 12341:2014 [7], weighing room conditions must be recorded at every weighing session. If any visual defects are observed on a filter it is immediately disposed of. The guidelines outline that blank filters should be loaded into filter holders in a 'clean environment' [7], however, repeated 1 hour exposures on a forecourt, yielded mass concentrations within the uncertainty of the measurement (less than $K = 2$, $\pm 17 \mu\text{g}$ for these filters) and so this contribution to any filter mass can be considered negligible (Table 1). Therefore, clean room conditions are generally not necessary for standard forecourt filter loading into filter holders for measuring.

Table 2: Filter weights after a 1-hour exposure on a HRS forecourt.

Filter	Mass difference	Measurement Uncertainty
Travel Blank 1	-3.4 μg	17 μg
Exposed Filter 1	-7.4 μg	17 μg
Exposed Filter 2	6.7 μg	17 μg
Travel Blank 2	-12.4 μg	17 μg

3.2 Storage

While being stored or during transport, the filters must be placed within filter sealed containers that prevent exposure. Insulation, such as basic cool boxes, should be used to aid in maintaining a temperature $\leq 23 \text{ }^\circ\text{C}$ during transport.

3.3 Blanks

Blanks are the simplest method to assess the quality of results and to account for factors outside of our control. For each stage of filter preparation, handling and measurement, a blank should be used. Two types of blanks are covered within EN 12341:2014 [7]:

Weighing room blanks – weights must be recorded at each weighing session alongside the sample filters. This ensures constant conditions within the weighing room.

Field blanks – These should be conditioned with the sample filters and weighed before transport and storage. These filters will then be conditioned after the sample filter has finished its collection and weighed again. This mass difference accounts for the mass change of the sampled filter under the same conditions. (note: this mass difference should be less than or equal to 60 µg to comply with the guidelines).

4 – Conclusions

This Good Practice Guide is intended to be used as helpful document for those wishing to set up their own measurement system for the determination of particulates in hydrogen. In line with the objectives set out in the MetroHyVe EMPIR project, this guide has highlighted the considerations, quality control and procedures required for weighing particulates collected from gaseous hydrogen from a filter weighing methodology. It has described a method for the accurate measurement of the collected particulate mass on filters, building on the expertise gained from the area of ambient air monitoring. It meets with the requirements for filter storage; conditioning and weighing that are laid out in the ambient air standard EN 12341:2014 [7]. Further to this it is compliant with the BS ISO 14687[1] standard for sampling from hydrogen. Due to the low particulate masses regulated in hydrogen it is clear that a measurement technique for this purpose must go beyond what is required in EN 12341:2014 [7]. By carefully controlling the temperature and humidity during the weighing process, it is possible to decrease the measurement uncertainty by a factor of 10. This guide has also shown experimentally that although care must be taken during filter exchanges, this is possible on site in an ambient environment without compromising the mass measurements. Finally, this guide has highlighted the importance of filter type and material for the weighing process.

5 – References

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