



WORKING FOR A HEALTHY FUTURE

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Guidance for collection of relevant particle size distribution data of workplace aerosols- Cascade Impactor Measurements

Araceli Sánchez Jiménez, Karen S Galea, Robert J Aitken

RESEARCH CONSULTING SERVICES

Multi-disciplinary specialists in Occupational and Environmental Health and Hygiene

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ABBREVIATIONS AND DEFINITIONS

Aerodynamic diameter	The aerodynamic diameter of a particle is the diameter of a unit-density sphere that has the same settling velocity as the particle in question. It is used to compare particles of different sizes, shapes and densities.
APS	Aerosol particle sizer
CI	Cascade Impactor
DMA	Differential mobility analyser
DMEL	Derived Minimal Effect Level
DNEL	Derived No Effect Level
D ₅₀	Particle diameter corresponding to 50% sampling efficiency
DRI	Direct reading instrument
GSD	Geometric Standard Deviation
GSP	Gesamtstaubprobenahme an der Person (developed by BGIA in Germany and known in EU as the Conical Inhalable sampler, CIS).
Inhalable particles	Inhalable fraction represents particles that enter the respiratory system via the nose or mouth (D ₅₀ = 100 µm).
MMAD	Mass Median Aerodynamic diameter: The MMAD is a statistically derived figure for a particle sample: for instance, an MMAD of 5 µm means that 50 % of the total sample mass will be present in particles having aerodynamic diameters less than 5 µm, and that 50 % of the total sample mass will be present in particles having an aerodynamic diameter larger than 5 µm.
PTFE	polytetrafluoroethylene
PSD	Particle Size Distribution
Respirable particles	The portion of inhalable particles that enter the deepest part of the lung, the non-ciliated alveoli (D ₅₀ = 4.25 µm).
Thoracic particles	The portion of the inhalable particles that pass the larynx and penetrate into the conducting airways (trachea, bifurcations) and the bronchial region of the lung (D ₅₀ = 11.64 µm).
TOF	Time of Flight

EXECUTIVE SUMMARY

Information on the particle size distribution (PSD) of workplace nickel aerosols is necessary to make the comparison of Derived No Effect Level (DNELs) and Derived Minimal Effect level (DMEL) to workplace exposures more realistic. The DNEL and DMEL are required under the REACH (Registration, Evaluation and Authorization) regulations for all substances handled or manufactured above certain tonnage. The DNELs and DMEL are based on animal studies and therefore have to be extrapolated to personal workplace exposures. However, animal inhalation studies use smaller size aerosols (2-3 μm) compared to typical Mass Median Aerodynamic Diameter (MMAD) diameter found for Ni aerosols ($\geq 50 \mu\text{m}$ MMAD). The smaller the aerosol the deeper it travels into the respiratory tract and the higher risk it poses. Therefore the DNELs and the DMELs will not be directly comparable to nickel workplace exposures. An alternative is to collect and use PSD data representative of the nickel aerosols however the current availability of this data throughout the industry is limited. There is therefore a need to promote the collection of such information.

Currently there is not a standard method to measure PSD of airborne particles in occupational settings. There are two types of instruments available that assess PSD, direct reading instruments (DRI) and those which require laboratory analysis e.g. gravimetric methods.

DRI use particle properties to measure their size and number concentration and calculate the mass by estimating particle density and shape. These types of instruments can allow real time measurement of the PSD. However, because of their size they do not allow collection of personal exposure measurements and collection of PSD of nickel compounds specifically is not possible.

Cascade impactors (CI) use filters to collect the aerosol. Particles are size-separated in the different filters for collection of PSD. Nickel can then be chemically extracted from the individual filters so the PSD of nickel compounds can be calculated. Conventional CIs have upper size limits of 20 μm . However, the inlets can be modified for collection of the PSD of the inhalable fraction as was done for the modified Marple CI and modified Andersen C. Both impactors allow collection of personal measurements.

Collection of personal measurements is fundamental to accurately determine the PSD of aerosols in the breathing zone. Particle size is a dynamic property which changes in space and time and therefore area measurements are unlikely to represent the PSD of the aerosol when it reaches the breathing zone.

Based on the above considerations modified CIs are regarded as the most suitable instruments for personal measurements of PSD of total aerosol and nickel compounds.

1 INTRODUCTION AND AIMS

The Institute of Occupational Medicine (IOM) was approached by the Nickel Institute to develop protocols to assist their members in the collection of nickel aerosol particle size distribution measurements (PSD) in their production workplaces.

This report provides a summary of the methods that are currently available to measure workplace PSD and describes the respective advantages and limitations of the methods.

2 METHODOLOGY

IOM experts on PSD methodologies convened to identify and discuss the common current PSD methodologies which may be applied to the industrial settings of interest. These discussions were supplemented with targeted literature reviews on the electronic databases Science Direct and PubMed:

- **Science Direct** (<http://www.sciencedirect.com>) – this database contains over 25% of the worlds science, technology and medicine full text and bibliographic information. Over 2,000 journals from Elsevier covering nearly 7 million articles and over 60 million abstracts from all fields of science are included in the database.
- **PubMed** (<http://www.ncbi.nlm.nih.gov/>) - this is a service of the US National Library of Medicine (NLM). PubMed provides access to bibliographic information that includes MEDLINE. MEDLINE covers the fields of medicine, nursing, dentistry, veterinary medicine, the health care system, and the preclinical sciences. MEDLINE contains bibliographic citations and author abstracts from more than 4,800 biomedical journals published in the United States and 70 other countries. The database contains over 12 million citations dating back to the mid 1960s.

PSD sampling product manufacturers such as TSI, GRIMM, Thermo Scientific, Palas were also consulted for relevant product information.

In addition, the following websites of recognised organizations for standard methods were consulted:

- National Institute for Occupational Safety and Health (NIOSH). URL: <http://www.cdc.gov/niosh/>
- Health and Safety Executive (HSE). URL: <http://www.hse.gov.uk/>
- US Environmental Protection Agency (USEPA). URL: <http://www.epa.gov/>
- American Society for Testing and materials (ASTM). URL: <http://www.astm.org/>
- International Standard Organisation (ISO). URL: <http://www.iso.org/iso/home.htm>

3 INTRODUCTION TO PSD SAMPLING

Health related aerosol size fractions are expressed as curves that relate the probability of aerosol penetration as a function of the aerodynamic size. The British Medical Research Council (BMRC) definition of the respirable aerosol fraction (those particles with a median aerodynamic diameter of 5 μm collected with a 50 % efficiency) was the first recognized internationally (Orenstein, 1960). In 1989, new criteria for aerosol

fractions were proposed by Soderholm (1989) and International collaboration lead to the agreement on the definitions of health-related aerosol fractions in the workplace, defined as inhalable, thoracic and respirable (CEN, 1992; ISO, 1995; ACGIH, 1995):

- Inhalable: the mass fraction of particles which can be inhaled by nose or mouth. The inhalable curve has 100% penetration for particles < 100 µm, dropping to 50% for 100 µm. Since at the time of development of the inhalable convention there were no experimental data on inhalable fraction of particles with an aerodynamic diameter of > 100 µm, particles > 100 µm are not included in the convention).
- Thoracic: the mass fraction of particles that reach the larynx. The thoracic curve has a median aerodynamic diameter of 11.64 µm and geometric standard deviation (GSD) of 1.5.
- Respirable: the mass fraction of particles that reach the alveoli. The respirable curve has a median aerodynamic diameter of 4.25 µm and GSD of 1.5.

Each curve can be interpreted as the sampling criterion to be achieved by an aerosol sampling instrument, in order to measure the corresponding aerosol fraction.

Following international agreement on these sampling criteria samplers have been developed and existing samplers evaluated, which have penetration characteristics that meet the CEN/ISO/ACGIH criteria. Examples include the IOM (Mark and Vincent 1986) and the Gesamtstaubprobenahme an der Person (GSP) (Kenny et al. 1997) samplers for the inhalable fraction and various cyclones for the respirable fraction. Several multi-fraction samplers have also been developed including a variant of the IOM sampler (Vincent et al 1993,). However, such samplers do not in themselves facilitate the elaboration of the complete size distribution of the sampled aerosol.

Instruments which collect and measure the entire PSD do however allow calculation of the three health related particle fractions (by integration across the corresponding channels) and also provide information on the MMAD.

Several principles can be used to separate airborne particles according to their aerodynamic diameter based on the particles properties. The particle property used for separation is important when comparing results from different instruments and the main principles are summarised below.

3.1 INERTIAL CLASSIFICATION SEPARATORS

These separators use inertia and gravitational forces to separate dust particles according to their size. Larger particles are separated from the air stream with the remaining particles being collected on a filter. Inertial separators include cascade impactors, cyclones, centrifuges, impingers and elutriators; however only cascade impactors which combine (usually) a series of collection stages are designed specifically to determine PSD.

3.2 LIGHT-SCATTERING DEVICES

These devices measure the scattered light by the particles and calculate the particle size based on a default calibration curve which is obtained for a specific dust (usually Arizona dust). As the refractive index of the particles measured in the occupational

setting may differ from the particles used for the calibration, the measured size does not represent the real size. The limit of detection for these devices is approximately 0.1 μm since the particle diameter must be larger than the wavelength of the incident light (Cohen and Hering, 1995). Examples of common light scattering devices include the GRIMM aerosol sampler.

3.3 TIME OF FLIGHT MASS SPECTROMETER

Time of Flight (TOF) techniques accelerate particles through a nozzle to obtain different velocities. The aerodynamic size of a particle determines its rate of acceleration, with larger particles accelerating more slowly due to increased inertia. At the nozzle exit, particles pass through two laser beams, one by one. The TOF between the two laser beams is measured for each particle. Because TOF sizing accounts for particle shape and is unaffected by index of refraction or MIE scattering (scattering of electromagnetic radiation by spherical particles), it is superior to sizing by light scattering methods alone (TSI, 2004). Assumptions about particle density and shape allow conversion of the size distribution to mass concentration. However, density varies with particle size and composition (Seinfeld and Pandis, 1998). The default density is $1\text{g}/\text{cm}^3$ and particles are assumed to be spherical (TSI, 2004)

3.4 CONDENSATION NUCLEUS COUNTERS

The condensation nucleus counter (CNC) or condensation particle counter (CPC) are used to measure particles that are too small to be measured directly by light-scattering instrumentation ($<0.1\ \mu\text{m}$). In these instruments, particles are grown to light-scattering sizes by exposure to a supersaturated vapour. Supersaturation is achieved by saturating the aerosol at an elevated temperature then cooling it in a condenser. Particles traversing the condenser increase in diameter by orders of magnitude reaching light-scattering sizes by the time they enter the optical detection cell following the condenser. There, light scattered by individual particles is measured by a photodetector and recorded as a voltage pulse. CNC and CPC can be connected to an electrical analyser (see section 3.5) to determine the PSD.

3.5 ELECTRICAL AEROSOL ANALYSERS

Electrical methods impart a charge to the particles. Size classification is made on the basis of the particle electrical mobility. The charged particles enter an electrical field, known as the mobility analyser, where particles are separated according to their size. The cut-off points are determined by the applied voltage. By varying this voltage and measuring the corresponding current carried sensor, a voltage-current curve is generated.

The latest development in electrical measurement techniques uses a differential mobility analyser (DMA). This type of analyser uses a bipolar charger rather than unipolar charger, which results in a better size resolution (Cohen and Hering, 1995).

This method can be used if the electric mobility of particles is a monotonic function of the particle size (i.e. each particle size has a constant electric mobility).

These devices measure in the nano size range and therefore are not suitable for measuring Ni aerosols in the μm scale. Examples of common electrical aerosol analysers include the Fast Mobility Particle Sizer (FMPS), size range (5.6-560 nm).

4 GENERAL CONSIDERATIONS WHEN DEVELOPING A SAMPLING STRATEGY FOR THE COLLECTION OF PSD

The same general principles which apply to measurement of inhalable or respirable dust also apply when sampling for PSD determination. Specific considerations that should be observed are highlighted below:

- Personal sampling is recommended rather than area/fix point sampling: Particle size is a dynamic property that changes in space. After emission particles can grow by aggregation or by condensation, or shrink by evaporation and therefore changes in the PSD occur. This means that the PSD of aerosols after emission will not be the same after certain time or in an area away from the emission source. Therefore area/static measurements may not represent exposure of the workforce.
- A sufficient number of samples are needed to allow representative exposure assessment. The sample size is dependant on the purpose of the assessment (e.g. compliance with OEL).
- Sampling duration should be similar to that of the working shift, since PSD can change in time due to changes in the process or worker activity, or because of particle aggregation, condensation or deposition
- Instruments for PSD are generally designed to measure dry particles. Care on the interpretation of the results have to be taken when sampling liquid aerosols
- Specific requirements must be considered depending on the instrument used. For example when sampling with cascade impactors the sampling time should be long enough so the mass of dust (or nickel) collected in all impactor stages is over the limit of detection. It could happen than for certain task/activities the typical working times are too short to allow collection of enough material on the filter.

5 DIRECT READING INSTRUMENTS TO MEASURE PSD

DRI for real-time aerosol measurements are available to cover particles in the size range of 0.4 to 105 μm .

Most of the DRI potentially useful for measurement of PSD are light-scattering devices. Examples include Promo, Promo 3000 and Fidas manufacture by PALAS. These instruments measured in the size range from 0.2 to 105 μm . However their performance has not been documented in the peer-reviewed literature. For example, information on the entry characteristics of the instrument and explanation of how particles as large as 105 μm can reach the optic volume without impacting on the walls of the instrument are important issues which have to be considered. (Further information on this has been requested to the manufacturer).

The GRIMM aerosol sampler manufactured by Aerosol Grimm Technology seems to have certain advantages compare to the others (e.g. smaller size, performance documented in literature review). This instrument is discussed in more detail in section 7.1.

Two instruments have been selected as the most appropriate for PSD measurements based on:

- Measure size distribution of particles in the micron size range
- Application in occupational settings
- Performance documented in peer-reviewed scientific literature.

5.1 GRIMM AEROSOL SPECTROMETER

There are two models available, the Grimm 1.108 and Grimm 1.109, both of which are optical particle counters.

The GRIMM 1.108 measures particulates as particle counts (particles/m³) or mass distribution (µg/m³) from 0.3 to 20 µm in 16 channels ((0.23) - 0.3- 0.4- 0.5- 0.65- 0.8- 1.0- 1.6- 2- 3- 4- 5- 7.5- 10- 15- 20 µm).

The model 1.109 provides similar information over a slightly wider size range at double the resolution (0.2-20 µm in 31 channels (0.25- 0.28- 0.3- 0.35- 0.4- 0.45- 0.5- 0.58- 0.65- 0.7- 0.8- 1- 1.3- 1.6- 2- 2.5- 3- 3.5- 4- 5- 6.5- 7.5- 8.5- 10- 12.5- 15- 17.5- 20- 25- 30- 32 µm).

In addition, all particles can be collected after optical detection on an integrated 47mm polytetrafluoroethylene (PTFE) filter inside of the spectrometer for further investigation e.g. speciation analysis (water soluble, sulfidic, oxidic and metallic nickel), microscopically or gravimetric, therefore offering the possibility of more targeted analysis for key aerosols of interest.



Figure 1 Photograph of the Grimm aerosol spectrometer Mode 1.108 (courtesy of Grimm Aerosol Technology, <http://www.grimm-aerosol.com>)

The GRIMM 1.108 and 1.0109 offer the following advantages:

- Relatively robust.
- Cheaper than APS (£9000 approximately, APS cost is £28,700)

- Can also collect a physical aerosol sample at the same time for (chemical) analyses of the aerosol (e.g. Ni speciation).
- Provide direct measurements of inhalable, thoracic and alveoli mass fractions as well as PM₁₀, PM_{2.5} and PM₁.
- Do not need to connect to lap top. Collects and stores data directly onto the device which can be downloaded at a later date.
- Can operate with batteries.
- Portable (1.7 kg plus battery 0.7 Kg)

The limitations and disadvantages of the GRIMM include:

- Fewer channels than the APS (15 channels for the PAS 1.108; 30 channels for model 1.109 compared to 52 with the APS)
 - Based on estimated density.
- Light scattering signal
- Complex to use
- Requires annual servicing and calibration (estimated cost £800)

5.2 AERODYNAMIC PARTICLE SIZER (APS)

The APS uses time-of-flight (TOF) particle sizing technology. Measurements are recorded for 52 channels (<0.523 to 19.81µm).

Aerodynamic Particle Sizer® (APS™) are trade marks of TSI Incorporated. The most recent model, 3321 (Figure 1) provides high-resolution, real-time aerodynamic measurements of particles from 0.5 to 20 µm. This model uses a patented, double-crest optical system to detect the occurrence of particle coincidence (when more than one particle is in the detection area) and to identify poor signals near the instrument's lower detection threshold, providing a higher accuracy on the calculations of mass-weighted distribution and higher aerodynamic sizing resolution with respect to the previous model (3320).



Figure 2 Photograph of the APS Model 3321 (TSI)

The Model 3321 can handle a concentration of 1,000 particles/cm³ with the coincidence error (two or more particles in the optical path) being less than 10%. If the concentration is too high, an Aerosol Diluter can be used to dilute the aerosol before it enters the APS spectrometer.

An example of data output from the APS is shown in Figure 3.

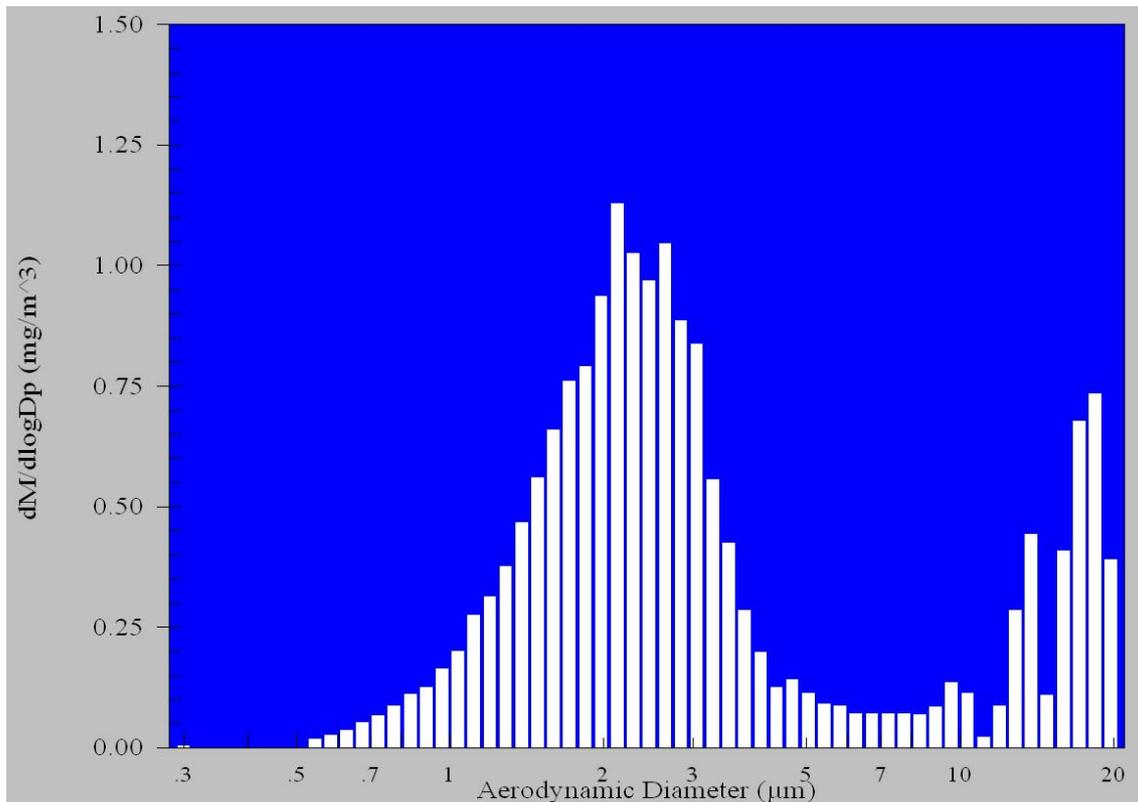


Figure 3 Example of data output from the APS.

Actual price of the APS 3321 is £28,700. From January 1st 2010 the price will be £30,710 with 2 years service agreement and extended warranty for £4,745 (it is possible to purchase 2 of these to give the 4 years extension and 4 service and calibration, (personal communication, TSI, 2009).

The TSI APS (model 3321) offers the following advantages:

- Real-time measurements.
- Rapid collection of measurements allowing for the ability to sample in different areas over one day.
- Data is easy to derive and manipulate to derive MMAD, GSD and can be used to obtain estimates of the respirable, thoracic etc aerosol fractions (following further data manipulation).
- No laboratory analysis costs, no consumables required.
- Performance is well documented in the peer-reviewed literature (Volchens and Peters, 2005; Peters et al. 2006).

The limitations and disadvantages of using the APS include:

- Usable but robustness in occupational setting questionable.
- Expensive to purchase (£28,700) and annual service costs (£4,745 for two years).
- Requires power supply.

- Measures all aerosol present, not specific to nickel substances / compounds.
- Not a routine occupational hygiene instrument; requires some basic level of expertise to use instrument and data analysis software.
- Requires calibration before use.
- Based on estimated density
- Upper and lower concentration limits (Peters et al. 2006)
 - Upper limit 20 microns, performance in upper size range variable
 - Lower limit 0.5 microns, performance at this lower range is poor
- Needs to be connected to lap top to log data continuously.
- Need a relatively safe place to install equipment and computer.
- Not portable (10 kg)

6 GRAVIMETRIC INSTRUMENTS TO MEASURE NICKEL PSD

There are currently no available methods available for real-time measurement of PSD for metals in airborne particles. This type of analysis requires collection of particles on a filter, from which nickel is extracted and quantified. If particles are size-separated in different filters, collection of PSD of nickel compounds is then possible.

Cascade impactors (CI) operate on this principle. A CI utilizes the inertial characteristics of a particle moving in a gas stream to separate the aerosol into different single fractions. Multi-stage impactors consist of a number of separated plates arranged in parallel with each containing an inlet nozzle, collection plate, and outlet orifice. The diameter of the orifice, the distance between the orifice and the collection plate and the flow rate for each stage are such that a particle greater than a certain size will be separated from the airstream in which it travels, and deposited on the plate. Smaller particles, having less inertia, will remain in the air stream and be carried past the plate. The larger the orifice size and the distance to the plate, the greater is the minimum size sampled. The filters on each plate are weighed separately before and after sampling. Flow rates must be carefully controlled.

Prior to sampling, collection substrates and the back-up filter are weighed. After sampling, the substrates and filters are re-weighed. Weight increase on each substrate is the mass of the particles for that impactor stage. The total weight of particles on all stages and final filter is added and the percent particle mass in each size range is calculated.

CIs do not provide immediate data on the aerosols PSD. Raw data of particle mass collected in the different stages have to be converted to the desired continuous PSD. The most common approach involves the application of software with PSD parameters. The algorithms can also be entered in an Excel spread sheet.

Particle sizes are expressed as MMAD and GSD.

The advantages of the CIs compared to DRIs include:

- The collection of a physical sample allowing chemical determination and speciation.
- Can be used as a personal sampling device as well as static sampler.
- Facilitates long term averaging.
- Less specialist knowledge is required to carry out the sample collection.

The disadvantages of CI instrumentation include:

- The cut-off points are dependant of the flow-rate, plugged nozzles and differences in the nozzle diameter. Differences between nozzle cut-off points are inevitable within manufacturing tolerance. Therefore certain amount of the aerosol lies within regions of uncertainty.
- Need for a mathematical algorithm to reconstruct the size distribution from the mass results obtained from each impactor stage. Curve fitting protocols always default to log normal distribution but the actual distribution of the aerosol may be different leading to error in the calculated PSD.
- CI requires an external pump.
- Does not give real time measurements.
- Complex to handle: it requires a clean environment to load and upload the filters.
- Laboratory analysis is required.
- Data needs to be input into appropriate software tools to obtain PSD.

6.1 MARPLE CASCADE IMPACTOR

The Marple Series 290 Personal Cascade Impactor is designed for personal measurements, establishing the aerodynamic PSD from 0.4 to 21 μm . Samplers come in various configurations - 2-stage, 4-stage, 6-stage and 8-stage. Cut-off points for the 8-stage configuration are $>21 \mu\text{m}$ and above, 15, 10, 6.5, 3.5, 1, 0.7, 0.4 and final filter.

Sampled air enters the inlet cowl and accelerates through six radial slots in the first impactor stage. The cowl eliminates ashes and debris from the sampler. Particles larger than the cut-point of the first stage impact on the pre-cut collection substrate. The air flows through the narrower slots in the second impactor stage and smaller particles impact on the second collection substrate. This process continues to the final filter. The width of the radial slots is constant for each stage but smaller for each succeeding stage. The jet velocity is higher for each succeeding stage, and smaller particles eventually acquire sufficient momentum to impact on the collection substrates. After the last impactor stage, remaining fine particles are collected on the built-in 34mm filter.

The impactor inlet is attached to a personal pump that should operate at 2 L/min. The impactors include brackets to attach it to the lapel or pocket.

Problems associated with the Marple CI include particle bounce, losses on the impaction surface (Vincent, 2007) and lack of clearly defined aspiration efficiency of the sampling inlet (Wu and Vincent, 2007). However, collection substrates loaded into the impactors may be greased to prevent losses due to particle bounce. A variety of filters can be used depending on the purpose of the analysis (e.g. Mylar, stainless steel, glass fiber).

The Marple CI is available commercially from New Star Environmental Inc. and Thermo Scientific Corporation for approximately £1000. Further details on sampling and PSD calculation are shown in section 10.

The advantages of the Marple CI include:

- Light sampler allowing for the collection of personal measurements.
- The small size allows collection in multiple locations.
- Can also be used as a static sampler
- A variety of substrates (e.g. Mylar, glass fibre, stainless steel filter) can be used for specific sampling analysis.
- It is marketed as occupational hygiene equipment.

The disadvantages include:

- It does not account for particles deposited between the stages.
- It needs long sample duration so the mass collected at each impactor stage is over the limit of detection of the analytical method used to determine the concentration of nickel.
- Care needs to be taken when transporting greased substrates.
- Laboratory analysis can be expensive due to the requirement for laboratory analysis of 4, 6 or 8 stages per sampler.
- Data needs to be entered into purpose built software / excel package.

6.2 MODIFIED-MARPLE CASCADE IMPACTOR

To overcome some of the limitations associated with the Marple impactor (e.g. clearly defined aspiration efficiency) the sampling aperture can be designed to correspond with the IOM head (Figure 4). The modified-Marple cascade impactor complies with the convention criteria for inhalable dust. However this version is not commercially available. The cost of the modification is approximately £80.



Figure 4 Original Marple cascade impactor (left) and modified-Marple cascade impactor with equivalent to the IOM head

Particles between 20-100 μm are collected on the top stage. PSD for these particles is estimated by extrapolation on the relationship of accumulated mass vs. aerodynamic diameter calculated for particles below 20 μm .

Advantages:

- Higher cut-off point than the conventional Marple CI allowing collection of the inhalable fraction.

Disadvantages:

- The inlet is not commercially available therefore it needs to be specially made. Estimated costs for the designed that correspond to the IOM head are £80 each. Details of an engineering company that can build the head are given in Table 1.

6.3 ANDERSEN CASCADE IMPACTOR (ACI)

The ACI consist of eight aluminium–alloy stages, which are held together by spring clamps and connected to a constant airflow. There are three versions covering three different size ranges (0.25-8.6 μm , which operates at 60L/min; 0.43-8.0 μm , which operates at 90 L/min and 0.4-9.0 μm which operates at 28.3 L/min. The cut-off points for the are (0.4- 0.7- 1.1- 2.1- 3.3- 4.7- 5.8- 9.0-.

The Andersen CI is also subject to particle bounce problems as the Marple CI (Kamilla et al. 2009) however again greased collection filters may be used to minimise these.

The Andersen CI has been widely used in pharmaceutical industry (Thermo Scientific, 2009). It is available commercially from Thermo-Andersen, Smyrna, GA.

Advantages:

- High volume sampler. Therefore does not require long sampling periods to guarantee sufficient mass collection at each stage for chemical determination and or speciation.

Disadvantages:

- Does not allow collection of personal measurements in contrast to the Marple CI due to its large size.
- Small particle size range (0.4-10 μm)

6.4 MODIFIED-ANDERSEN MARK II CASCADE IMPACTOR (M-ACI)

The modified-Andersen CI consists of a conventional Andersen CI with an additional top stage incorporating a plug of plastic porous foam collection media (Figure 5). This additional stage enables the upper end of the range of the instrument to be extended from about 10 μm to greater than 70 μm (Vincent et al. 2001). The instrument is designed for collection of liquid and solid aerosols (Thermo Scientific, 2009).

Data on the continuous PSD is achieved by calibration of the collection characteristics of the porous foam stage, together with the known calibration data for the impactor stages of the original instrument. The collection properties of the porous foam media used has been well characterized (Vincent et al. 1993, Chen et al. 1998).

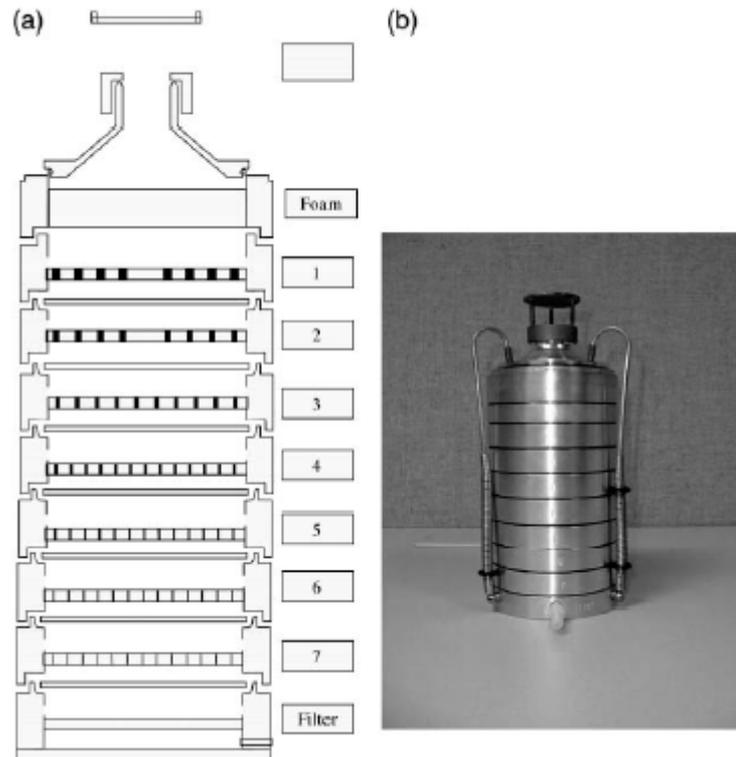


Figure 5 Schematic of the modified-Andersen Mark-II cascade impactor (a) and photograph of the assemble instrument (Vincent et al. 2001)

The advantages of the modified Andersen CI include:

- Higher cut-off point than the conventional ACI allowing for the collection of the inhalable fraction.
- Tested in a Nickel refinery for chemical speciation of nickel compounds (Vincent et al. 2001). No problems were raised in the study regarding the instruments performance.

The disadvantages of the modified Andersen CI include:

- It is not commercially available
- Also same disadvantages as others CI– cost of analysis, preparation of greased substrates and transportation of these.

7 DISCUSSION AND RECOMMENDATIONS

Table 1 provides a summary of the main characteristics (type of measurement, collection of physical sample for further analysis and estimated costs,) of instruments that allow collection of PSD of airborne particles.

Table 1 Summary of the characteristics of PSD instruments

Instrument	Principle	Personal / static	Collection of PSD of nickel	Costs (£) (approx costs at Nov 2009)	Level of expertise ¹	Specificity		
						Size channels	Weight (kg)	Particle concentration (particles/m ³)
APS 3321	Time-of-flight	Static	No	28,700	Medium	0.5 to 20 µm In 52 size channels	10	- 0.001 to 1x 10 ⁹
Grimm 1.108	90° light scattering and filter-sampling	Static	No	9000	Low	0.3 to 20 µm in 16 size channels	2.4	0.1 to 100.000 1 to 2 x 10 ⁹
Grimm 1.109	90° light scattering and filter-sampling	Static	No	9000	Low	0.25 to 32 µm in 31 size channels	2.4	0.1 to 100.000 1 to 2 x 10 ⁹
Marple CI	Inertial forces	Personal & static	Yes	1500 (8 stage impactor) 1580	Low	0.4 to 21 µm. In 8 stages	0.3	-
Modified-Marple ² CI	Inertial forces	Personal & static	Yes	(8 stage impactor)	Low	0.4 to 100 µm	0.3	-
Andersen CI	Inertial forces	Static	Yes		Low	0.4 to 10 µm In 8 stages	pending	-
Modified-Andersen	Inertial forces	Personal & static	Yes		Low	0.4 -100 µm In 9 stages	pending	-

¹ As judged by authors; ² The modified head can be purchased from IBP engineering in the UK (Tel. +44 (0) 1506 884 287; Email: ian@ibpengineering.co.uk)

Details of companies that provide sampling services with these instruments are given in Table 2.

Table 2 Details of some companies that carry out sampling with PSD instruments.

Company	Instruments	Web site	Contact	Approximate price
IOM (Edinburgh)	Modified Marple CI	www.iom-world.org	martie.vantongeren@iom-world.org +44(01) 31 449 8097	£ 2500* (gravimetric & chemical analysis of 8 CI)
VITO (Belgium) Flemish institute for technological research	Andersen Mark III CI (area/static) (9-stages)	www.vito.be	nico.bleux@vito.be +32 (0)14 335358	
Genesis Environmental Limited (UK)	8-stage impactor	www.genesis-environmental.co.uk/	domalley@globalnet.co.uk	

*Price refers to gravimetric analysis of dust and do not include travelling and accommodation

Currently there is not a standard method to measure PSD of airborne particles in occupational settings. The ISO 21501-1:2009 (ISO, 2009) method specifies characteristics of a light scattering aerosol spectrometer (LSAS) which is used for measuring the size, number concentration and number/size distribution of particles suspended in a gas in the range of 0.06 to 45 µm. The light scattering technique described in this document is based upon single particle measurements. This means that at high concentrations, when two or more particles coincide in the optical path they will not be counted leading to underestimation of the real concentration.

Other international organisations consulted (e.g. ASTM) provide specification on PSD measurements in clean environments and dust control rooms, but not in occupational settings. The use of this equipment in occupational environments might not be appropriate as the concentrations in industrial settings are likely to be much higher than those in control rooms and the instrument might not be designed to cope with such high concentrations.

The main advantage of DRIs compared to filter-based methods relies on the real-time availability of the data, often at one minute resolution, allowing collection of multiple samples without further analysis. They are simple to operate and possibly less expensive in the long term as they do not require filters which need laboratory analysis.

However, these methods are not specific to nickel compounds. They assess the distribution of all the particles present in the air. Furthermore, greater uncertainty is associated with the measurement of mass by this approach as opposed to gravimetric measurements (Solomon and Sioutas, 2008), as assumptions on the density and shape of the measured particles have to be done to transform particle numbers into mass concentration. Additionally, they are heavy and don't allow collection of personal measurements. Thorpe and Walsh (2002) have also highlighted other limitations: calibration of the device can vary significantly depending on the physical properties of

the aerosol being measured. Therefore to obtain accurate measurements monitors should be calibrated using standard methods with the aerosol of interest; some devices can also be susceptible to contamination, particularly of the optics, which often results in a change of the monitor's response, with it not always being possible to determine the impact of this change; high acceleration forces can affect the size response as a function of particle density and shape; forces can also de-agglomerate particles causing them to appear smaller.

Recommendations on the basis of instrument performance are difficult. Peters et al. (2006) reported systematic differences in the number and mass concentration measurements collected with the APS (model 3321) and the GRIMM (model 1.108). Performance was assessed for dry particles (three sizes of monodisperse PSL spheres (0.83, 1.0 and 3.0 μm) and a polydisperse aerosol (Arizona test dust)). The GRIMM was able to detect particles with greater efficiency than the APS for particles smaller than 0.7 μm . Although number concentrations reported by the GRIMM were greater than the APS in monodisperse tests, they were lower than the APS in polydisperse tests for particles between 0.7 and 2 μm . Peters et al (2006) concluded that the experiments conducted in this work were insufficient to resolve these differences.

The Marple CI and the modified Andersen CI allow collection of personal measurements in contrast to DRI. This feature is of considerable importance when sampling PSD as the size of particles changes in time and space and therefore static measurements might not represent the real PSD of the aerosol in the breathing zone.

Comparison of the performance of the Marple and the Andersen CI were not found in the reviewed literature.

Based on the above information, the modified Marple CI or modified-Andersen CI are considered the most appropriate instruments for simultaneous characterization of the PSD of the total aerosol and nickel compounds (note that chemical extraction of nickel is required). Both instruments allow collection of personal measurements and have an upper size limit that allows examination of the PSD of the inhalable fraction.

8 COLLECTION AND ANALYSIS OF AIRBORNE PSD WITH A MARPLE CI

8.1 SAMPLING METHOD

The modified 8 stages-Marple Series 290 Personal Cascade Impactor comprises an IOM head as sampling inlet with a standard body of the Marple CI. This has 8 separated plates arranged in parallel with each containing six radial slots. The width of the radial slots is constant for each stage but smaller for each succeeding stage. Therefore the velocity is higher for each succeeding stage, and smaller particles eventually acquire sufficient momentum to impact on the collection substrates. Particles between 21-100 μm (and greater) are collected in the top stage (entry stage). The cut-off points of the successive plates are > 15, 10, 6.5, 3.5, 1, 0.7, 0.4 and final filter were remaining particles are collected on the built-in 34mm PVC filter (Figure 1).

Prior to sampling, collection substrates and the back-up filter are weighed. After sampling, the substrates and filters are re-weighed.

CI's do not provide immediate data on the aerosols PSD. Raw data of particle mass collected in the different stages has to be converted to the desired continuous PSD. The weight increase on each substrate gives the mass of the particles collected in that impactor stage. The total weight of particles on all stages and final filter is added and the percent particle mass in each size range is calculated. An Excel spreadsheet has been developed which allows data entry, fitting of cumulative size distribution curves and calculation of the MMAD and GSD. This is provided as Appendix 2. Data analysis is described in section 10.3.

8.2 PRACTICAL CONSIDERATIONS WHEN SAMPLING

The following practical steps are important when using the modified Marple CI. These have been taken primarily from the manufacturer's instructions (Marple Series 290, 2006).

1. Make sure all parts of the impactor are present: nine stages, final filter holder, two metal screws, rubber rings and impactor unit (Figure 1).

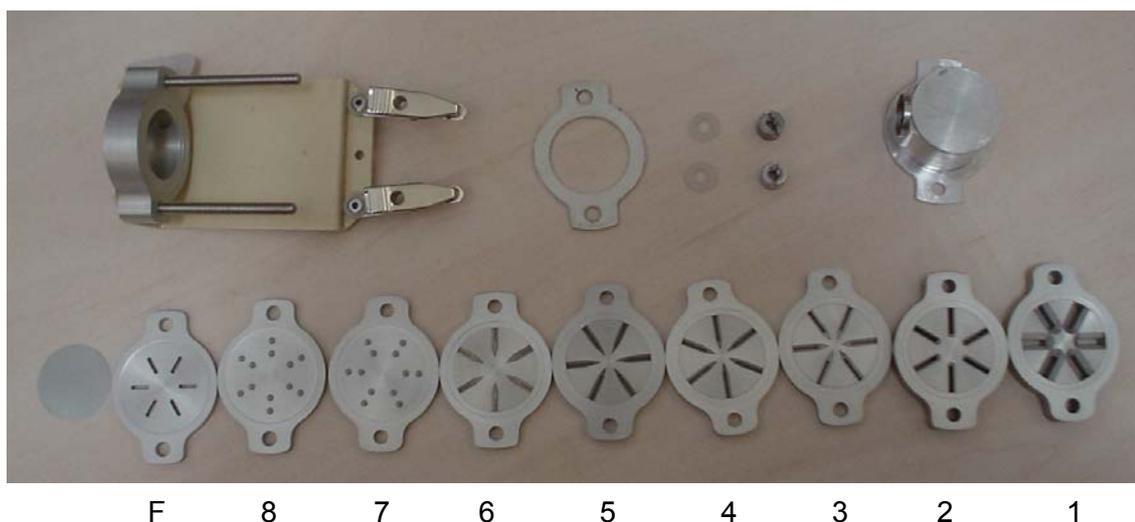


Figure 6 Marple Series 290 Personal Cascade Impactor. The particle cut-off of the stages decreased from right to left.

2. Clean all the impactor parts with detergent or alcohol. Make sure all the slots of the stages are free of dirt. Once cleaned, dry completely.
3. If using Mylar substrates or glass fibre filters grease the edge of the substrate with silicone taking care the slots are not covered. This should be done before weighing the filters and several hours before start the monitoring to allow solvent to evaporate completely. Greasing the substrates prevents losses due to particle bounce. The grease should be of appropriate stability to prevent it from flowing under the impaction plate. If the grease flows under the plate there is a risk for particles getting stuck to it, which would result in underestimation of the particle mass. Silicon grease or Vaseline are usually used (MIAC, 2008).

- The impactor stages are all labeled on the right edge. The filters should be kept in tin cassettes properly labeled, so as to know what filter correspond to each impactor stage. Take a note of what filter has been placed in each stage. An example of a sampling record sheet is given in Appendix 1. Start assembling the impactor by placing the final filter holder in the impactor unit. Then place the PVC filter, following by the stage labeled "F". By using tweezers, previously cleaned with alcohol, place the corresponding filter over stage F and then the impactor labeled "8". Place the corresponding filter over stage 8 making sure the perforations on the filter match the slots of the stage, then place impactor 7 and so son. Table 3 describes in detail the assembly process.

Table 3 Cascade Impactor assembly order

Filter holder
PVC filter (note number on sheet)
F plate
Substrate (note number on sheet - 8)
Impactor stage 8
Substrate (note number on sheet - 7)
Impactor stage 7
Substrate (note number on sheet - 6)
Impactor stage 6
Substrate (note number on sheet - 5)
Impactor stage 5
Substrate (note number on sheet - 4)
Impactor stage 4
Substrate (note number on sheet - 3)
Impactor stage 3
Substrate (note number on sheet - 2)
Impactor stage 2
Substrate (note number on sheet - 1)
Impactor stage 1
Rubber gasket
Entry cowl

Make sure all the impactor stages are aligned (i.e. the numbers are line up on the same side in ascending order). After putting the impactor stage 1 place the rubber ring followed by the inlet cowl and finally the small rubber rings and the metal bolts.

- Assemble the dust pump and adjust the flow rate to 2 L/min with a calibrated flow-meter.
- Label the cascade impactor.
- Attached the cascade impactor to the collar of the employee. Make sure the sampler inlet is not covered by the employee's coat.
- The following data should be recorded:
 - Initial and final flow rate. If possible also measured the flow rate during sampling, especially for long sampling periods (> 2hrs).
 - Time the pump has been turn on and off.

- Contextual information on the type of activities of the employee wearing the sampler.
9. To disassemble the impactor first remove the metal bolts and rubber rings. Remove the inlet cowl and impactor stage 1. With the help of clean tweezers take the filter and place it in the corresponding tin cassette. Then remove impactor 2 and so on.

8.3 CALCULATION OF THE MMAD AND GSD

8.3.1 Introduction

This section describes the formulae used for the calculation of the MMAD, GSD and the percentage of dust contained in the thoracic and respirable fractions. These formulas have been entered in an Excel spreadsheet which allows direct calculation of the PSD parameters (Appendix 2). Section 8.3.3 explains how to enter the data in the spreadsheet.

A description of the possible errors associated to the calculations is discussed in section 8.3.4.

8.3.2 Calculations

For calculation of the MMAD, GSD and dust concentration in the different impactor's stages the following information is required:

- Sampling duration (t): length of time the pump is working (min).
- Flow rate (Q): the mean sampler flow rate is calculated as the average of the flow rates at the beginning (Q₁) and at the end of the sampling period (Q₂) (l/min).
- Pre-weight (W₁): weigh of the collection substrate prior to sampling (mg)
- Post-weight (W₂): weigh of the collection substrate after sampling (mg)

Sampling volume (V): the sampling volume is calculated from the flow rate (Q) and sampling duration (t) (Equation 1).

$$V \text{ (m}^3\text{)} = \frac{Qxt}{1000} \quad (1)$$

The weigh gains for each stage (W) is calculated as shown in (Equation 2).

$$W_i \text{ (mg)} = W_{i1} - W_{i2} \quad (2)$$

Where i= 1,2,3,4,5,6,7, 8, F

The concentration at each stage is calculated by dividing the weight of each stage (W_i) by the volume (V):

$$\Delta C_i \text{ (mg/m}^3\text{)} = \frac{W_i}{V} \quad (3)$$

The total dust (C_{tot}) is calculated by adding all the weigh gains for each stage and dividing by the total volume (V) (Equation 4).

$$C_{tot} \text{ (mg/m}^3\text{)} = \frac{\sum_{i=1}^n W_i}{V} \quad i = 1, 2, 3, 4, 5, 6, 7, 8, F \quad (4)$$

The percentage of mass at each stage is calculating by dividing the mass collected in each stage (W_i) by the total mass (W_{tot}), which is the sum of particle masses for all stages including the back-up filter (Equation 5)

$$\% \text{ mass in each stage (mg)} = \frac{W_i}{W_{tot}} \times 100 \quad (5)$$

The percentage of mass with a diameter less than that indicated by each stage is calculated as follow:

$$\% < D_p \text{ (stage } i\text{)} = 100 - \sum_{j=1}^i \% \text{ mass in stage } j \quad (6)$$

The differential particle size is calculated as follow:

$$\Delta \log_{10} D_p = \log_{10} D_{p_{i-1}} - \log_{10} D_{p_i} \quad (7)$$

D_{p_0} is the largest particle sampled. If unknown D_{p_0} is taken as 50 μm . By convention the diameter for the final stage (F) is take as half that of the stage 8.

The differential concentration for each particle size is therefore:

$$\Delta C_i \text{ (mg/m}^3 \log_{10} \mu\text{m)} = \frac{\Delta C_i}{\Delta \log_{10} D_p} \quad (8)$$

The GMD is calculated from equation 9:

$$\text{GMD } (\mu\text{m)} = \sqrt{D_{p_i} - D_{p_{i-1}}} \quad (9)$$

The MMAD is calculated from the 2-point interpolation of the data points representing the cumulative mass percent above and below 50%.

The GMD is calculated by dividing the MMAD by the D_p at which the cumulative percent of the mass is 16%...

$$GSD = \frac{MMAD}{D_p(16\%)} \quad (10)$$

The D_p corresponding to the 16% cumulative mass is calculated in the same way as the MMAD, from the 2-point interpolation of the data points representing the cumulative mass percent above and below 16%.

The percentage of mass in the thoracic and respirable fractions is calculated from the 2-point interpolation of the data points above and below 11.64 μm (thoracic) and 4.25 μm (respirable).

The 2-point interpolation method does not assume an underlying distribution of the particle sizes. Therefore, this method can be applied to any distribution.

The MMAD of the distribution is usually calculated assuming a log-normal distribution of the particle sizes mass. For example the USP (US Pharmacopeia) method recommends using the straight line of the normalized cumulative mass vs. particle diameter plotted on log-normal probability paper. If the distribution of the data is not log-normal the determination of MMAD will be biased to an unknown extent, depending on the deviation of the distribution from the assumed log-normality (Christophe et al. 2010).

8.3.3 Excel sheet for data processing

An Excel spreadsheet has been developed with all the calculations (Appendix 2). The file contains a section with instructions. The mass gains for each stage (W_i) are entered in the corresponding blue boxes. This allows calculation of the cumulative percent of mass below the indicated particle size. The user has to enter the cumulative percent mass values and the corresponding cut-off diameters above and below 50% and 16% in the corresponding blue boxes. This allows calculation of the MMAD and the GSD.

The method used for the calculation of the PSD parameters is a simple algebraic interpolation of the data points. This approach does not assume the distribution of the particle size mass is log-normal. The only assumption made is that the mass particle size distribution is uni-modal. To get a sense of the form of the particle size distribution the user can examine the shape of the curve in the graph $\Delta C / \Delta \log_{10} D_p$ vs. GMD (in the sheet: particle size distribution).

8.3.4 Possible sources of error

There are a number of features which could give rise to error

- The cut-off points are dependant on the flow-rate, the slot width and distance to the collection plate. Differences in the slot width and spacing to the collection plate are inevitable within manufacturing tolerance. Therefore certain amount of the aerosol falls within regions of uncertainty.

- Partial blockage of the slot can occur. This would have the effect of increasing the velocity through the slots and therefore increasing the collection efficiency for smaller particles.
- Particle bounce is a feature which is known to affect cascade impactors. Large particles travelling at high velocity can rebound on impact with the collection plate and be re-entrained in the airstream and carried to the next collection stage. At that point their velocity will be higher and the probability of bounce will be even greater. These particles can often reach the final collection filter. To some extent, this effect can be diminished by coating the collection substrates with grease although this adds to the difficulties in handling and analysis (Marple Series 290, 2006)
- The curve fitting used for the calculation of the MMAD and GSD described in the Excel spreadsheet defaults to unimodal distribution but the actual distribution of the aerosol may be different leading to error in the calculated MMAD. In some cases, for example if the actual distribution was bi-modal, this would lead to an underestimation of the MMAD of the largest mode.
- In situations where most of the mass is collected in the entry stage there would be a loss of accuracy of the calculation of MMAD. By using a data inversion technique assuming a bi-modal distribution, similar to that described by Vincent (1996) it would in principle be possible to evaluate bimodal distributions. However this technique is also subject to limitations. The Mercer-Morgan-Flodin method (MMF) and the Chapman-Richards (CR) methods use a computational algorithm to fit the cumulative percent mass to the observed data, without assuming an underlying distribution. These methods are described in Christophe et al. (2010).

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APPENDIX 1 SAMPLE RECORD SHEET FOR THE CASCADE IMPACTOR

8-stage CASCADE IMPACTOR SAMPLE RECORD SHEET			
Cascade Impactor no.:			
Pump number:			
Date:			
Location:			
Start time:		End time:	
Start flow rate:		End flow rate:	
Cascade Impactor levels		Filter code	
1			
2			
3			
4			
5			
6			
7			
8			
PVC filter			
Comments:			

APPENDIX 2 EXCEL SPREADSHEET FOR CALCULATION OF PSD FROM THE 8-STAGE MARPLE CI



Modified-8-stage
Marple CI_data_anal