An aerosol sampler for single particle analysis

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Abstract
The micro inertial impactor (MINI), a miniaturized cascade impactor designed especially for the collection of atmospheric aerosol for electron-microscopic single particle analysis is described. The small instrument size and the low air volume flows allow versatile operation in conjunction with other instruments or standalone. The split design of the single stages allows easy adaptation of the substrate holders to different types of substrate, as well as a quick change of substrate type. The necessary construction details are given in this work.

Introduction
The electron-microscopic single particle analysis has been gradually becoming a common technique for the analysis of atmospheric aerosol since the 1960s (e. g., McMurry, 2000 and references therein). Prior to the analysis, the aerosol is collected on a suitable substrate. Depending on the particle size, collection is mainly performed by filter sampling, impaction, or electrostatic and thermophoretic precipitation.

The collection of samples for individual particle analysis differs from those of for example bulk-chemical methods. The particles need to be isolated on the substrate, which should provide as much contrast to the particles as possible. Depending on the method of analysis, this can be achieved by differences between the particles and the substrate in chemical composition or morphological structure (different degrees of smoothness). It is of advantage, if the substrate is electrically conductive as it may be analyzed without further preparation (i. e. sputtering of conductive materials onto the sample, which may interfere with the X-ray analysis).

For a wide particle size range important for atmospheric processes, sampling by cascade impactors provides advantages such as defined deposition areas with gradually changing deposition density, very low air volume flow rates, which allows a miniaturized design and a robust operation. Although the first impactors were already developed in the 1860s and significant progress was made in the 1960s and 1970s (Marple, 2004), still new impactors are developed (Berner et al., 1979; Demokritou et al., 2004; Gómez-Moreno and Fernández de la Mora, 1996; Kwon et al., 2003; Marple et al., 2003; Marple et al., 1991; Raabe et al., 1988).

In this work, a micro inertial impactor (MINI) is described, a cascade impactor with single round nozzles designed for a versatile operation for the collection of single particle analysis. During the development most attention was paid to the small instrument size, ease of use, interchangeability of different substrates, and a size range of particle collection suitable for the electron-microscopic single particle analysis.

Sampler
One of the first instruments optimized for low volume flow rates was described by Mercer et al. (1970). The MINI follows this design, but for ease of use each single stage is split into a nozzle and a substrate holder. The MINI outer shell consists of a tube with interior and exterior diameters of 10 mm and 18 mm, respectively and an inner length of 40 mm. It features o-ring fittings at the top and bottom to seal the threads, which are right-handed on top and left-handed on bottom to avoid wrong insertion under field conditions. Stainless steel was chosen as construction material for the outer shell as well as for the stages. For collection in areas where steel may occur as aerosol particles, brass is a suitable alternative.

The split design of the single stages (see Fig. 1) allows the use of nozzles and substrate holders in any combination. O-

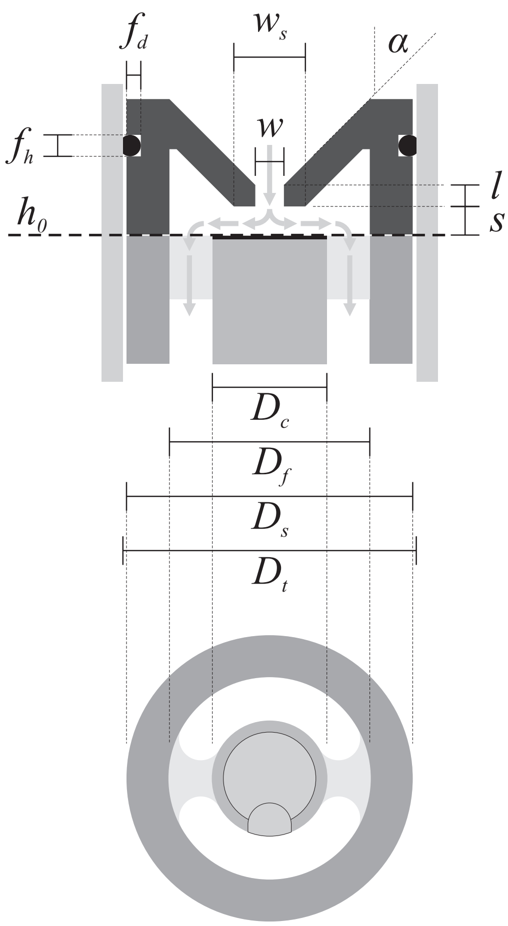

Fig. 1: Schematic drawing of a nozzle/TEM grid substrate holder set; measures and criteria are given in Table 1

criterion	value
s/w	1.3
l/w	1 for w > 0.5 mm, else 0.5 mm
w_s/s	3 for w > 0.5 mm, else 1.5 mm
α	45°
D_t	10.0 mm
D_s	9.95 mm
D_f	7.0 mm
D_c	4.0 mm
f_d	0.9 mm
f_h	1.2 mm
O-ring spec.	8 mm x 1 mm

Table 1: Design criteria and measures for the stages; see Fig. 1 for explanation

w, mm	d_{50}, μm	*Re*	*w*, mm	d_{50}, μm	*Re*
0.25	0.1	2900	0.70	1.0	950
0.30	0.2	2300	0.75	1.1	900
0.35	0.29	1900	0.80	1.2	800
0.40	0.38	1700	1.0	1.7	650
0.45	0.47	1500	1.3	2.6	500
0.50	0.56	1300	1.5	3.2	450
0.60	0.76	1100	2.0	5.0	350

Table 2: Nozzle diameters w, associated nominal 50 % cut-off sizes d_{50} and Reynolds Re numbers for the MINI at 20°C and 1000 hPa inlet pressure, using the smallest nozzle as critical orifice

rings around the nozzles ensure the sealing of the single stages and hold the stages in position through friction. The MINI tube can hold up to five single stages. Nozzles from 0.2 to 1.5 mm were constructed with a small interval. The nozzle construction followed the design criteria given by Newton et al. (1977). Specific relationships and measures are summarized in Table 1. Alternatively the MINI can be used in low-pressure mode. To achieve this, a drop in the pressure on the critical nozzle of significantly more than half of the inlet pressure is necessary. In the low pressure region behind the critical orifice, particles down to 30 nm can be collected. However, the maintenance of the necessary vacuum (typically 50 to 100 hPa at a strongly increased air volume flow) on the impactor outlet requires rather large vacuum pumps, contrary to the design concept of the MINI.

The flow rate is usually set by keeping one of the nozzles in critical condition. When the 0.25 mm nozzle is used as critical orifice, a flow of approximately 0.5 l/min is reached. Table 2 gives the nozzle diameters and the corresponding approximate cut-off sizes for this case (Raabe et al., 1988).

For each type of substrate the substrate holders need to be adapted to place the surface of the sampling substrate at the reference height h_0 (Fig. 1) with exactness better than the nozzle diameter *w* to yield the target cut-off size. The substrate holders feature magnets to securely hold substrates like transmission electron microscopy (TEM) grids or nickel plates, which are commonly used in electron microscopy. The maximum usable substrate size is about 5 mm in square. Fig. 2 gives examples of substrate holders designed for various substrates, which are commercially available for electron microscopy. Other substrates from beryllium and boron to silicon and copper as well as carbon adhesive have been successfully used in laboratory work as well as in ground-based and airborne field campaign measurements (e. g., Kandler et al., 2007; Petzold et al., 2009; Weinbruch et al., 2008).

To facilitate the assembly of the MINI it is recommended to create a rod with two different tips from a softer material (e. g., PTFE) to push the stages in and out. As the nozzles are susceptible to mechanical damage, an annular tip should be used with an outer diameter of 6.5 mm and an inner diameter of 4.5 mm. The other tip used for pushing into the inlet cone

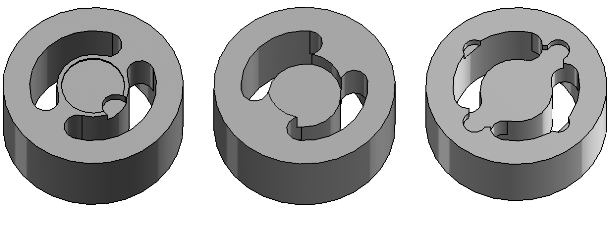

Fig. 2: Substrate holders for 3.05 mm diameter TEM grids with edge to prevent slipping (left), for 4 mm diameter metal discs with or without coating (center), and for 5 mm square silicon plates (right)

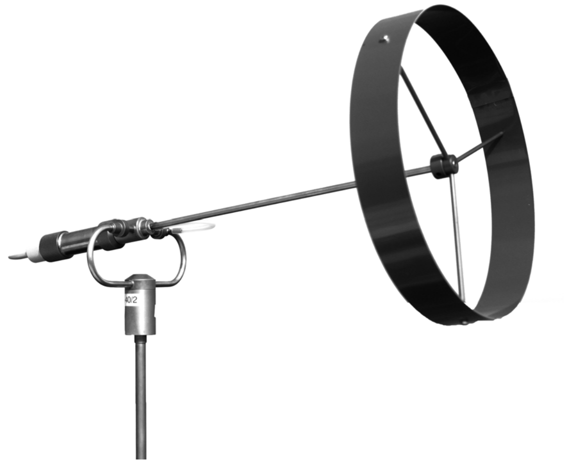

Fig. 3: MINI mounted on a three-dimensional annular-type wind vane for iso-axial sampling

of the nozzles should be spherical or tapered. In addition, a cap should be built which holds the stages safely while pushing them out of the impactor tube. An inner diameter of 11 mm and a length of 40 mm are recommended, as well as a screw mount fitting the impactor tube outlet. An end-to-end slot in the cap facilitates taking out the nozzles and substrate holders with tweezers.

Application

Depending on the application, the MINI can be used in-line with or inside of other sampling/measurements devices or stand-alone. However, to collect particles larger than 10 µm under typical atmospheric wind speeds, iso-axial behavior should be ensured. To achieve this, the MINI can be mounted on a three-dimensional wind vane and be equipped with an inlet nozzle (Fig. 3). This way, particles of up to 30 µm diameter can be collected.

The sampling time for single particle analysis depends on the aerosol size distribution and is thus strongly variable under typical atmospheric conditions. In regions with high aerosol load like street canyons usually a few seconds of sampling time are sufficient. In remote regions or the free troposphere, sampling times of one hour may be necessary. When the concentration levels of sub- and super-micron aerosol are extremely different, it may be advisable to make several collections with different nozzle sets and different sampling times to achieve a suitable particle density on the substrate.

Limitations

As all impaction samplers, the MINI applies high forces to the particles during impact. For soft material, this may lead to particle deformation (Wittmaack, 2002), which can be eased somewhat by using a soft collection surface (Kavouras and Koutrakis, 2001). If droplets are present in the aerosol, they may break up during impact. The advantage of a very low sampling volume and sampling area of the MINI can become a disadvantage, if large particles in low concentration are to be investigated. As for all cascade impactors and depending on the environmental situation, particle bounce-off and re-entrainment is a potential problem (Dzubay et al., 1976; John et al., 1991), which can, however, be minimized by the use of appropriate sampling substrates. The MINI is designed for the collection for single particle analysis, and thus its use for size distribution determination is not recommended.

Conclusion

The MINI has proven to be an easy to use tool to collect atmospheric aerosol particles for single particle analysis. Thanks to the split design of the single impaction stages, a wide variety of different substrates suitable for the use in electron microscopy can be used. The MINI is operated with a lower volume flow than most of the impactors available commercially, and it is one of the smallest available impactors. For these reasons, it is well suited when miniaturization is a demand, e. g. onboard of airborne sampling platforms like aircrafts and balloons or as a personal monitoring sampler.

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