

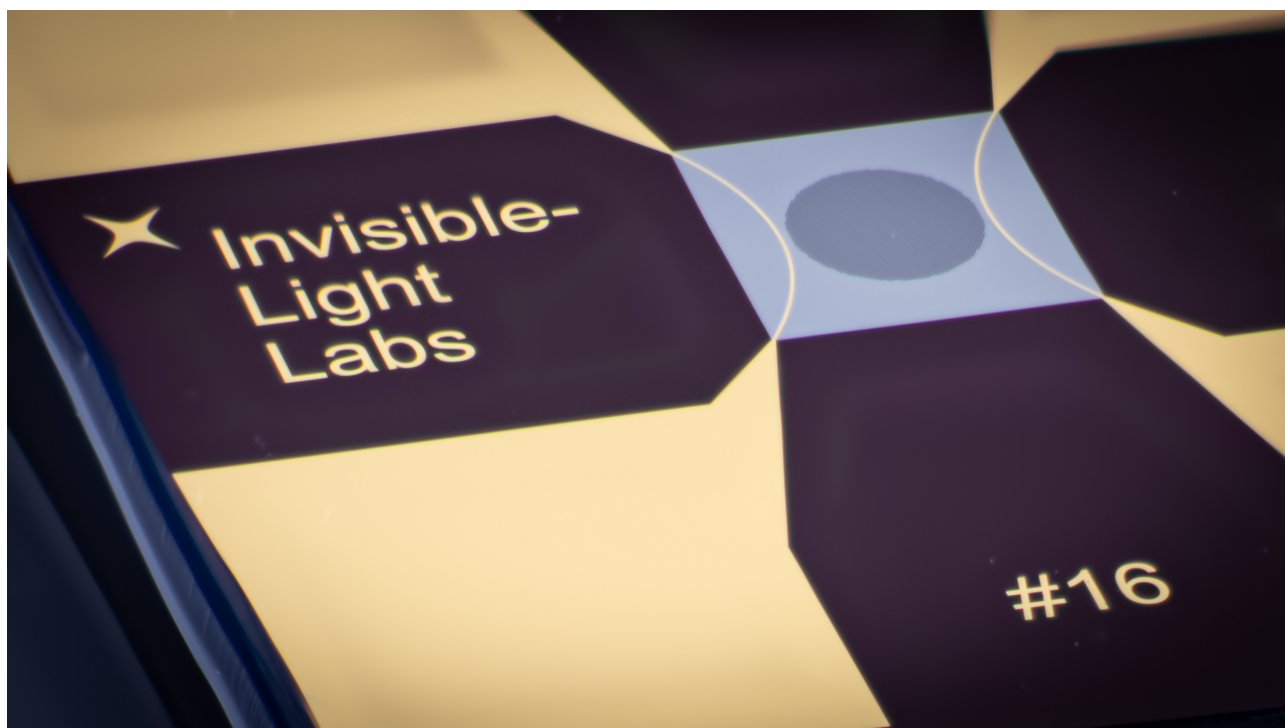
## SAMPLE COLLECTION & HANDLING GUIDE

Follow the instructions in this guide to get started with the handling and sample collection of EMILIE™ nanomechanical sampling and sensing chips.

November 4, 2025

---

This guide presents an overview of diverse sample collection techniques that can be used to deposit your sample on the EMILIE™ nanomechanical sampling and sensing chips ("EMILIE™ chips"). The general handling of the EMILIE™ chips as well as the procedures for sample collection via aerosol and drop casting methods are described in the following sections.

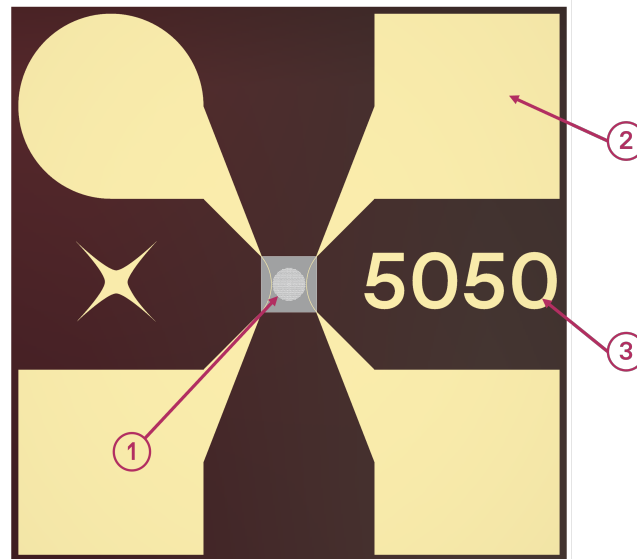


## CONTENTS

|          |                                                                             |          |
|----------|-----------------------------------------------------------------------------|----------|
| <b>1</b> | <b>The EMILIE™ nanomechanical sampling and sensing chip</b>                 | <b>3</b> |
| 1.1      | Detection limit and optimal mass load . . . . .                             | 4        |
| <b>2</b> | <b>EMILIE™ chip handling</b>                                                | <b>4</b> |
| 2.1      | Chips delivered in PVC boxes . . . . .                                      | 5        |
| 2.2      | Chips delivered in a Gel-Box™ . . . . .                                     | 5        |
| 2.3      | Chips delivered in capsules . . . . .                                       | 5        |
| <b>3</b> | <b>Sample collection strategies</b>                                         | <b>6</b> |
| 3.1      | Drop casting methods . . . . .                                              | 7        |
| 3.1.1    | Drop Casting Accessory . . . . .                                            | 8        |
| 3.1.2    | Preparation of the Drop Casting Accessory . . . . .                         | 9        |
| 3.1.3    | Drop casting procedure . . . . .                                            | 9        |
| 3.1.4    | Drop casting procedure with Drop Casting Accessory . . . . .                | 10       |
| 3.2      | Spin casting procedure . . . . .                                            | 11       |
| 3.3      | Aerosol methods for airborne nanoparticles . . . . .                        | 12       |
| 3.3.1    | The Aerosol Flow Adapter . . . . .                                          | 12       |
| 3.3.2    | Particle selection by impaction . . . . .                                   | 13       |
| 3.3.3    | Particle selection with a scanning mobility analyser . . . . .              | 14       |
| 3.3.3.1  | Particle counter . . . . .                                                  | 15       |
| 3.3.3.2  | Automated sample collection solutions . . . . .                             | 16       |
| 3.3.4    | Sample collection time estimation . . . . .                                 | 16       |
| 3.3.5    | Aerosol sample collection procedure with the Aerosol Flow Adapter . . . . . | 16       |
| 3.4      | Aerosol methods for liquid nanoparticle dispersions . . . . .               | 18       |
| 3.4.1    | Procedure for sample collection from liquid dispersions . . . . .           | 18       |

# 1 THE EMILIE™ NANOMECHANICAL SAMPLING AND SENSING CHIP

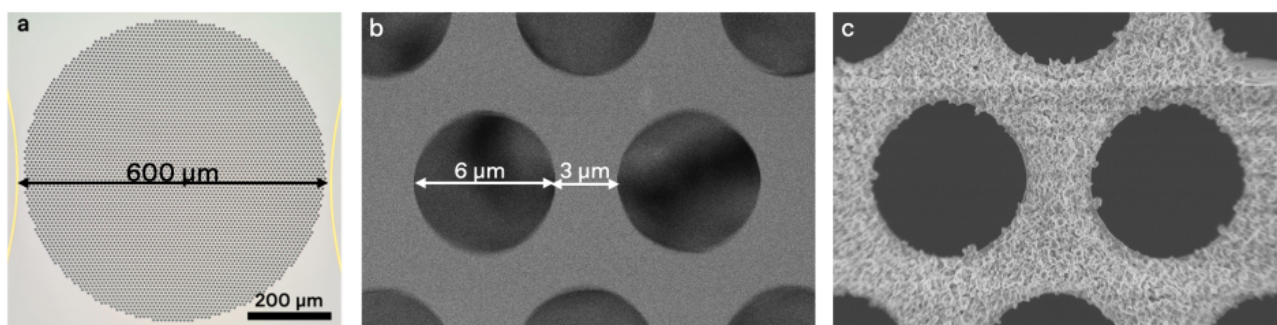
The EMILIE™ nanomechanical sampling and sensing chip, or EMILIE™ chip, is the heart of EMILIE™. The chip is both a sample holder and a highly sensitive detection element for NEMS-FTIR spectroscopy. Fabricated in a state-of-the-art clean room environment, the EMILIE™ chip features a highly transparent 50 nm-thick perforated silicon nitride membrane and two pairs of gold electrodes for signal transduction. The EMILIE™ nanomechanical sampling and sensing chip features a perforated area with an outer diameter of 600  $\mu\text{m}$ , each perforation has a 6  $\mu\text{m}$  diameter, and a 3  $\mu\text{m}$  pitch between adjacent perforations, as shown in Figure 2(a-b). The perforated area allows efficient collection of nanoparticles via impaction. In addition, samples can be drop casted and spin casted on the EMILIE™ chips. Figure 2(c) shows that sample collection on the EMILIE™ chip is not limited by the perforation size, but that it is possible to sample nanoparticles with diameters much smaller than that of the perforations. The EMILIE™ chip is designed to be disposable due to the high sensitivity of the method and the very high risk of cross-contamination when reusing chips.



**Figure 1:** Illustration of the EMILIE™ nanomechanical sampling and sensing chip highlighting its main components: (1) silicon nitride membrane, (2) gold electrodes, (3) individual chip number.

A schematic illustration highlighting the main components of the EMILIE™ chip is shown in Figure 1. It features:

1. A thin nanoelectromechanical membrane made of silicon nitride.
2. A pair of gold electrodes. One electrode is for driving the resonator, and the other is for readout.
3. A unique number identifying each chip within its batch.



**Figure 2:** Micrograph of an EMILIE™ chip's nanoelectromechanical membrane showing (a) the perforated membrane, (b) the perforations, and (c) the perforations after collecting 54 nm nanoplastic particles via nebulization

## 1.1 Detection limit and optimal mass load

As with other IR spectroscopy techniques, the detection limit of an analyte depends on its molecular attenuation coefficient. Table 1 summarizes the detection limit and optimal mass loading for the sample polystyrene as an example.

| Parameter               | Value               |
|-------------------------|---------------------|
| LOD Absorbance          | 40 $\mu$ AU         |
| LOD Polystyrene         | 350 pg <sup>1</sup> |
| Dynamic mass load range | 0.1 ng - 200 ng     |
| Optimal mass load       | 30 ng               |

**Table 1:** Detection limit and optimal mass load for polystyrene.

## 2 EMILIE™ CHIP HANDLING

EMILIE™ nanomechanical sampling and sensing chips are shipped in either a storage container consisting of a polyvinyl chloride (PVC) bottom and a polyoxymethylen copolymer (POM-C) lid (Figure 3), a Gel-Box™ (Figure 4), or individually packaged in capsules (Figure 5).



Store the EMILIE™ chips in their original packaging or in similar protective containers specifically designed for microelectronics to shield them from the environment.



Use high-precision tweezers equipped with carbon-fiber tips to manipulate the EMILIE™ chips. Narrow tips and metal tweezers may cause damage to the silicon.

<sup>1</sup>Evaluated for the asymmetric bending vibration of CH<sub>2</sub> groups of polystyrene located at  $\nu(\delta) = 1452 \text{ cm}^{-1}$ , featuring a molecular attenuation coefficient of  $\approx \alpha_{10_v} = 510$ .

## 2.1 Chips delivered in PVC boxes

The top side of the box is marked by a label, make sure it is facing up when opening the box. Slide the box out of the sleeve and remove the top cover. An EMILIE™ chip can be picked up by gently placing the tweezers on the outer edges of the chip, aligned with the center of the chip, carefully applying pressure and lifting the chip as shown in Figure 3. Keep the box closed when not in use to avoid contamination of the EMILIE™ chips and container.

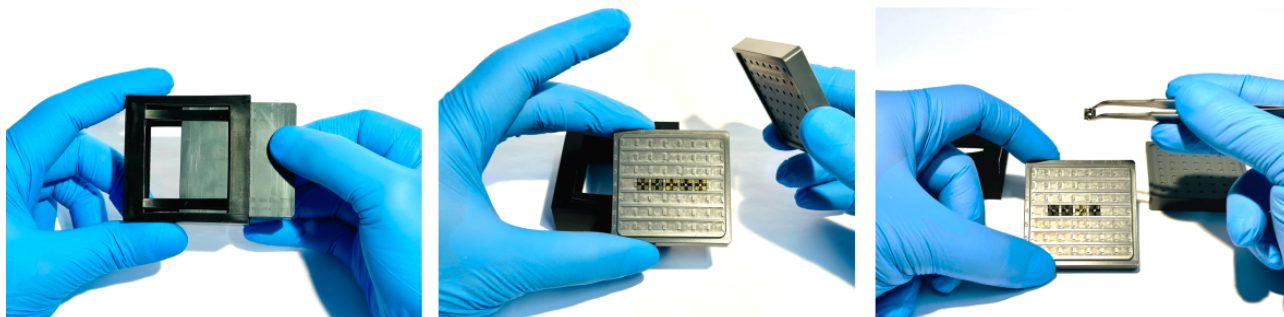


Figure 3: Handling of an EMILIE™ chip delivered in plastic container.

## 2.2 Chips delivered in a Gel-Box™

The bed of Gel-Box™ containers is adhesive. To release the EMILIE™ chip from the gel, gently place the tweezers on the outer edges of the chip, aligned with the center of the chip, carefully apply pressure to the edges and rotate the box while keeping the chip firmly in place. This will help break the adhesion without damage; do not try to lift the chip straight off the adhesive. The EMILIE™ chip can now be lifted from the Gel-Box™ as shown in Figure 4. Keep the box closed when not in use to avoid contamination of the EMILIE™ chips and container.

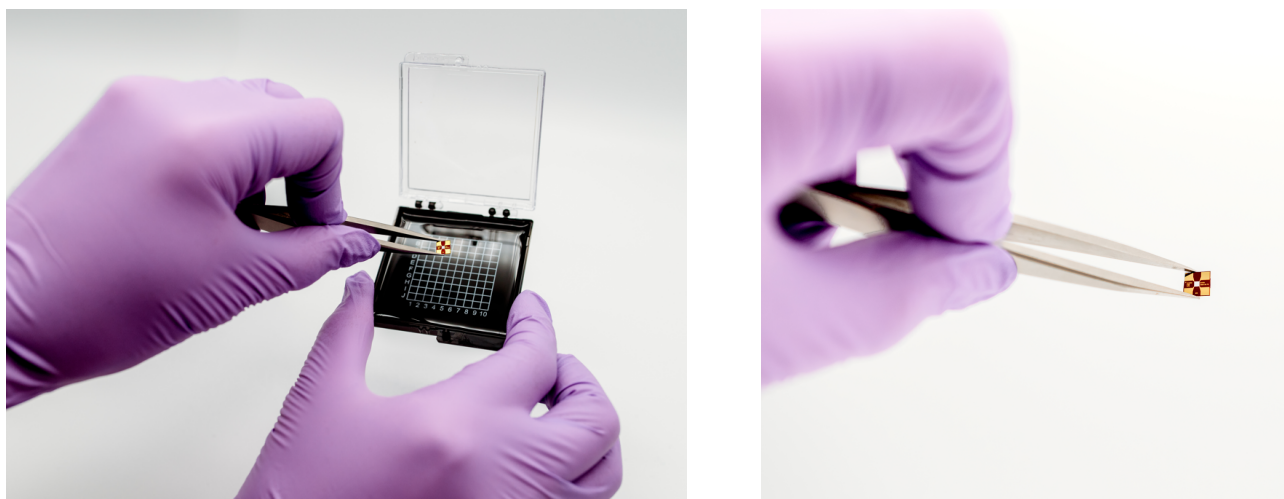


Figure 4: Handling of an EMILIE™ chip delivered in a Gel-Box™.

## 2.3 Chips delivered in capsules



Avoid exposure of the capsule to water, high humidity or other solvents.

To remove an EMILIE™ chip from a capsule (Figure 5(left)), remove the cap and gently squeeze the capsule containing the EMILIE™ chip to widen its mouth along the width of the chip as shown in Figure 5(center). Using high-precision tweezers, apply gentle pressure on the area of the chip with markings, while staying clear of the gold electrodes and the central nanomechanical membrane. Place the EMILIE™ chip on a clean surface, such as lint-free wipes, change the grip of the tweezers to the edges of the chip and proceed with your manipulations.

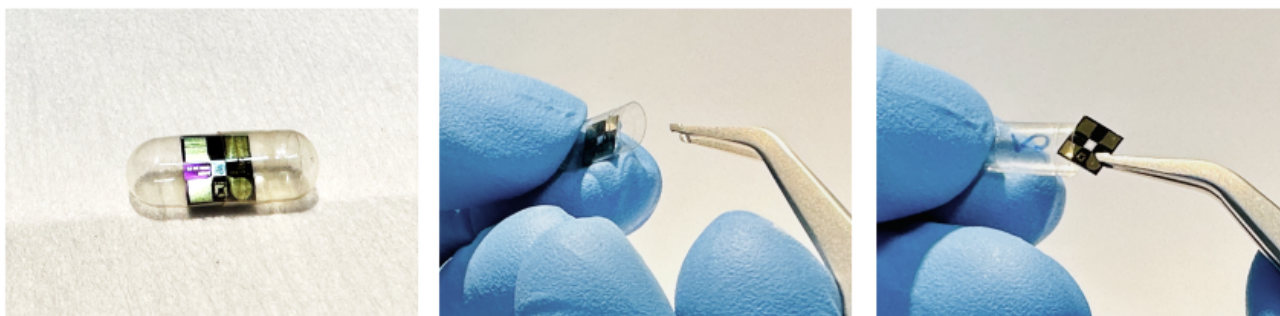


Figure 5: Handling of an EMILIE™ chip delivered in a capsule.

### 3 SAMPLE COLLECTION STRATEGIES

To analyze a sample with EMILIE™, the sample has to be deposited on the EMILIE™ chip. Several strategies are available to achieve this, depending on the requirements.

- Standard aerosol and nebulization techniques can be used for aerosols and dispersed nanoparticle solutions.
- Drop casting can be used for dispersions and solutions.
- Spin casting can be used for thin films, dispersions, and solutions.

In this guide, the focus is on the techniques illustrated in Figure 6.



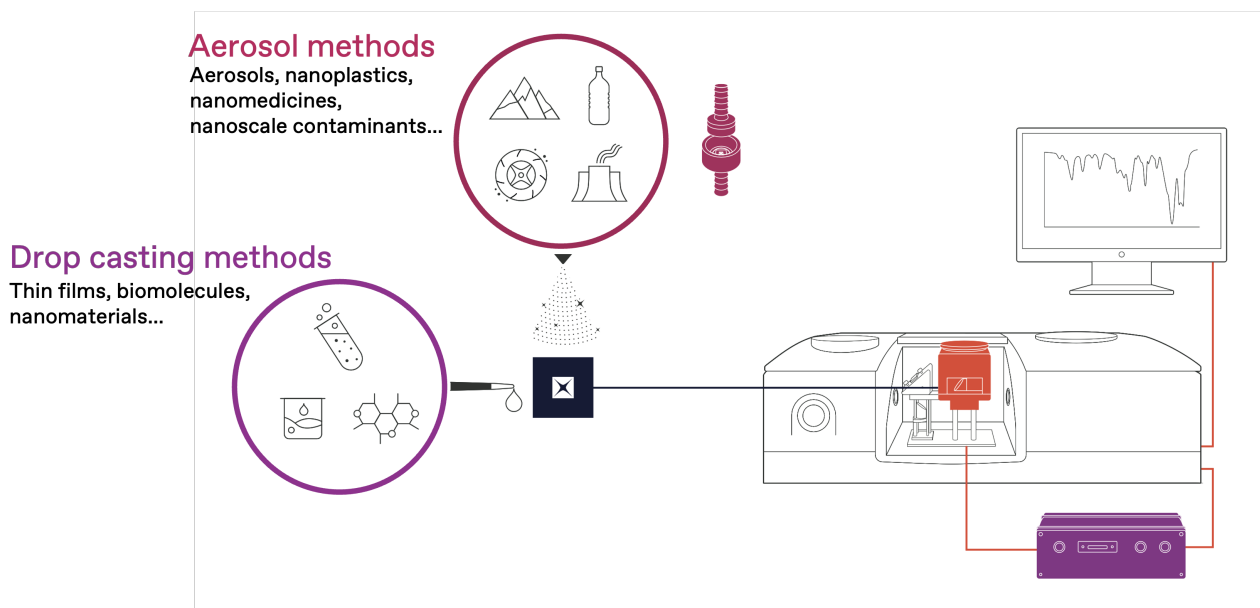
Avoid procedures that may harm the mechanical integrity or break the thin nanomechanical membrane in the center of the EMILIE™ chips.



Avoid overloading the EMILIE™ chip by keeping the dried analyte mass under 200 ng.



Due to the presence of a vacuum during measurements, EMILIE™ chips are not suitable for the analysis of volatile analytes. Some semi-volatile analytes can be retained on the chip by cooling the chip continuously with the integrated Peltier element.



**Figure 6:** Sample deposition on the surface of the EMILIE™ chip via aerosol or drop casting methods prior to NEMS-FTIR analysis



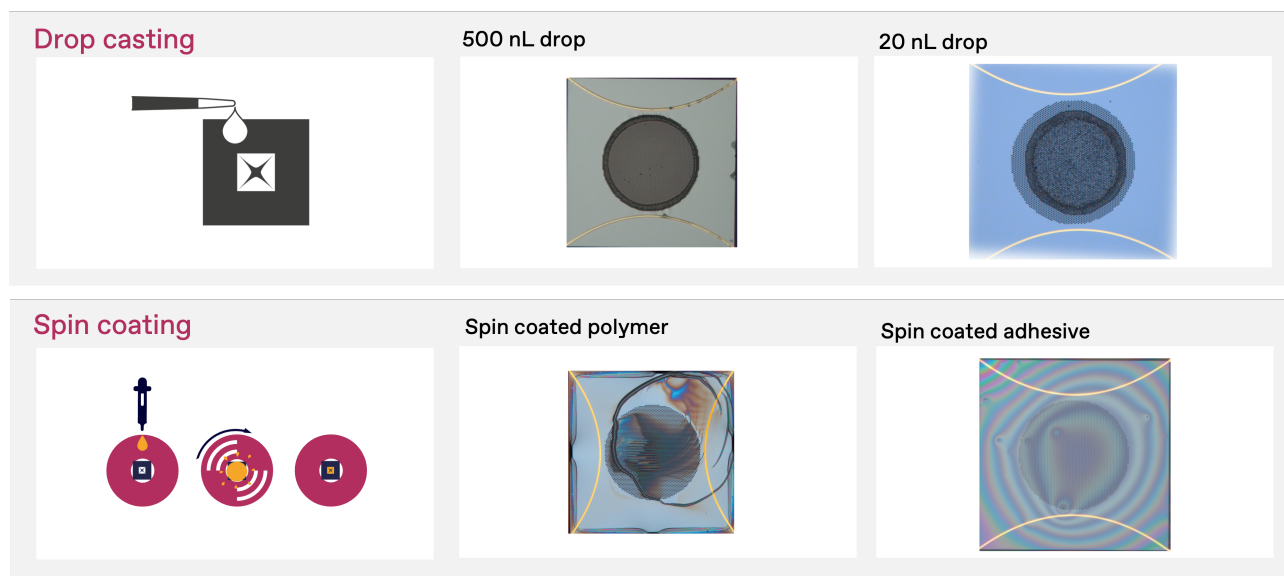
EMILIE™ chips are not suitable for the analysis of thick or rigid polymer films which affect the tensile stress of the thin nanomechanical membrane at the center of the chips.

Which sample collection strategy to use depends mainly on the form of the analyte. With EMILIE™, it is possible to collect analytes in various forms, including aerosol, powder, dispersion, or solution:

- **Aerosol** (particle size from 10 nm to 3200 nm): Can be collected directly by the aerosol method. See section 3.3.
- **Powder**: Can be aerosolized and collected via the aerosol method (see section 3.3) or dispersed in a liquid and drop casted (see section 3.1), respectively.
- **Dispersion**: Can be nebulized and collected by the aerosol method (see section 3.4) or drop casted (see section 3.1) directly.
- **Solution**: Can be nebulized and collected by the aerosol method (see section 3.4), or drop casted (see section 3.1) directly.

### 3.1 Drop casting methods

Various types of semi-volatile and non-volatile samples can be collected on the EMILIE™ chip using drop casting methods, as illustrated in Figure 7. These methods include drop casting with a pipette or dispensing a nanodrop with a nanoliter droplet dispenser. For drop casting, Invisible-Light Labs offers a dedicated Drop Casting Accessory that ensures all the sample is collected at the center of the sensor chip, facilitating quantitative analysis. Additionally, it is possible to spin coat the drop casted solution to produce a thin film for analysis.

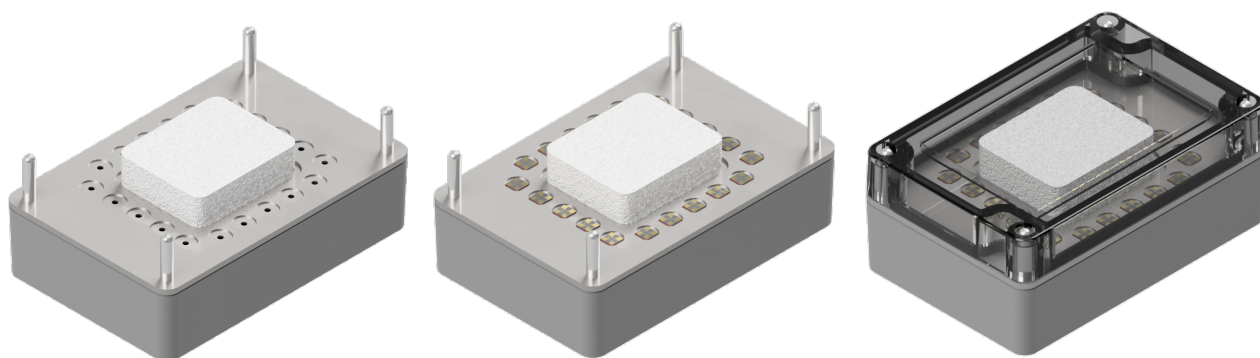


**Figure 7:** Solutions are easily deposited on the EMILIE™ chip via drop casting. Small droplets can dry immediately, while larger volumes are concentrated in the center with the Drop Casting Accessory. Further treatment such as spin coating can be further applied for samples such as polymers and adhesives.

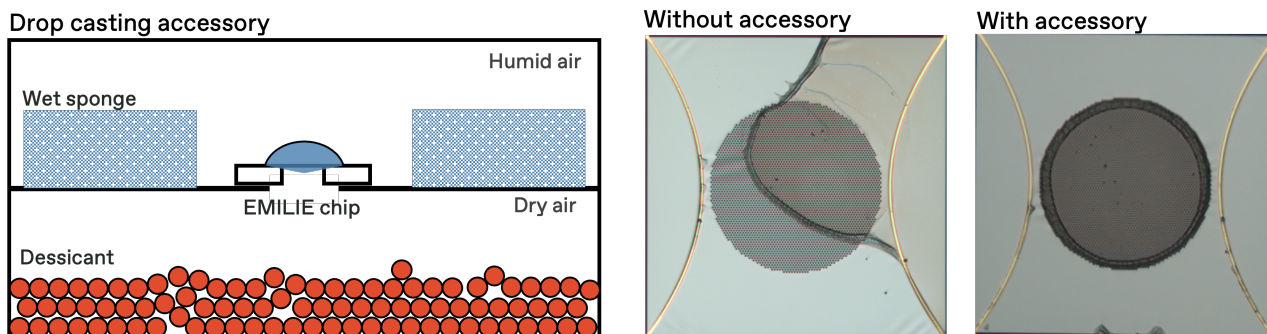
### 3.1.1 Drop Casting Accessory

One of the primary challenges associated with drop casting is the phenomenon known as the coffee ring effect (also known as “coffee stain effect”). When a droplet containing dissolved or dispersed analytes evaporates on a solid surface, particles are transported toward the contact line due to capillary flow, resulting in the accumulation of analytes at the periphery of the droplet. As evaporation progresses, the solvent evaporates more rapidly at the droplet’s edge, leading to the formation of a ring-like deposit of analytes, commonly referred to as the coffee ring.

The Drop Casting Accessory shown in Figure 8 mitigates this effect. The principle of operation shown in Figure 9 restricts the evaporation of solvent to within the perforated area of the EMILIE™ chip, ensuring that no analyte dries outside the perforated area, where the analyte’s signal would be lost. When the analyte is deposited in the center of the perforated area, the drop shrinks in volume and diameter until it is entirely within the perforated area and dries completely.



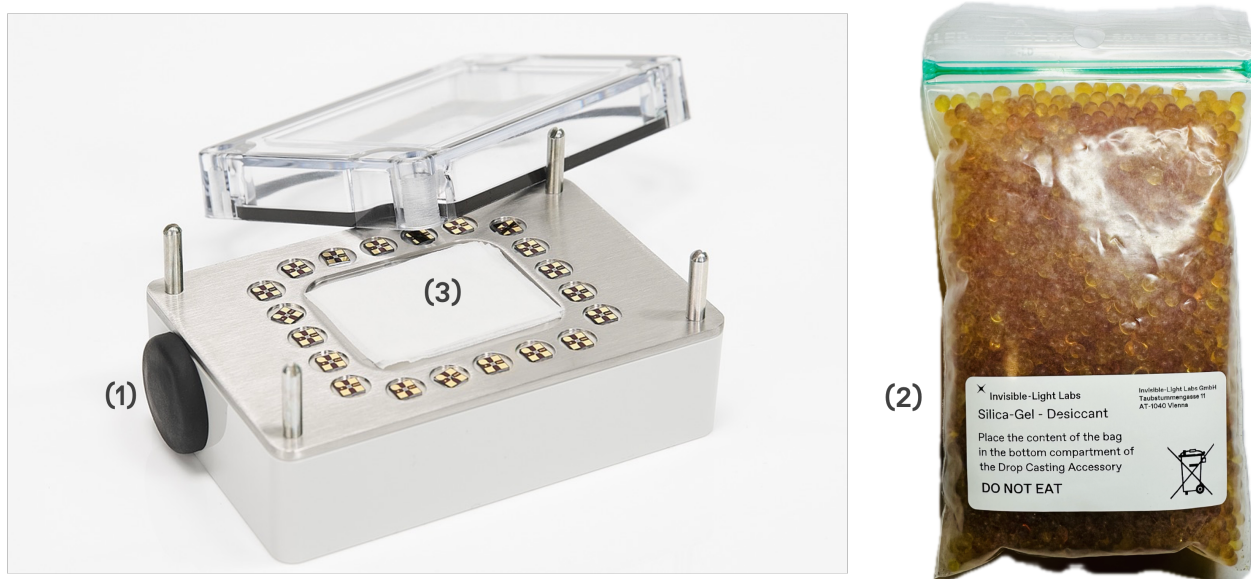
**Figure 8:** The Drop Casting Accessory facilitates drop casting of samples on up to 20 - EMILIE™ chips simultaneously.



**Figure 9:** In the Drop Casting Accessory (left), the EMILIE™ chip is placed at the interface between a humid and a dry atmosphere. Without the Drop Casting Accessory, a drop dries with a coffee ring over a larger surface area of the chip (center) whereas with the Drop Casting Accessory, the analyte is fully contained within the perforated area of the membrane (right).

### 3.1.2 Preparation of the Drop Casting Accessory

Before use, the bottom compartment of the Drop Casting Accessory must be filled with silica gel. This is done by removing the black plug as shown in Figure 10. Pre-cut lint-free wipes are placed in the dedicated space at the center of the drop casting platform to generate the necessary humidity gradient.



**Preparation of the Drop casting Accessory:** (1) The black plug can be removed to inspect or refill the desiccant (2). Pre-cut humidified lint-free wipes are placed in the dedicated space at the center of the drop casting platform (3).

**Figure 10:** Preparation of the Drop Casting Accessory .

### 3.1.3 Drop casting procedure

Different drop casting procedures can be used depending on the sample collection requirements. For larger volumes (500–1000 nl), the Drop Casting Accessory is recommended to concentrate the analyte at the center. Using a micropipette, carefully deposit a droplet of up to 1  $\mu$ l onto the

center of the EMILIE™ chip, ensuring that the pipette does not touch the membrane surface.

For smaller volumes with higher analyte concentrations, a contactless nanoliter dispenser can be employed. Depending on the device, volumes between 2 and 50 nL can be dispensed with high precision. Since the resulting droplet is much smaller than the membrane, it is important to align the dispenser so that the droplet is deposited in the center (perforated area) of the chip. This improves reproducibility.

#### 3.1.4 Drop casting procedure with Drop Casting Accessory

- Before drop casting, make sure the desiccant in the bottom compartment of the Drop Casting Accessory is dry and not saturated (dry desiccant is a bright orange).
- Prepare the Drop Casting Accessory by placing a small sponge or a few pre-cut lint-free wipes in the dedicated space at the center of the drop casting platform. To generate the humidity gradient between the top and bottom compartments of the drop casting accessory, deposit a few large drops of clean water on the lint-free wipes using a syringe or a micropipette.
- The drop casting platform features 20 chip indentations with through-holes covered by small metallic discs. Remove one of the small metal discs where you wish to deposit a chip for drop casting. When depositing sample and drying multiple chips simultaneously, remove as many metal covers as needed. To maximize drying efficiency, ensure that all remaining holes are covered.
- Open the container with your EMILIE™ chips and gently transfer the chip centrally over the uncovered hole on the drop casting accessory platform.
- Load the micropipette with a 500 nL aliquot of your sample. The droplet should be large enough to cover the entire perforated area of the membrane for higher reproducibility.
- Slowly approach the chip with the loaded micropipette tip and gently deposit the drop in the center of the chip. Avoid any direct contact between the micropipette tip and the EMILIE™ chip to prevent damage to the fragile nanomechanical membrane.
- After depositing the sample on the EMILIE™ chip, close the lid of the drop-casting accessory by sliding it on the guidance rods and allow the droplet to dry. A droplet of 500 nL will take approximately 30 minutes to dry.

As a guideline, a 1 µL droplet of DI water takes roughly 60 min to evaporate, while a droplet of 500 nL takes 30 min. If the process is much faster than expected and the drop does not fully dry in the center, the air in the top compartment is likely not saturated and the sponges need to be rehydrated. To provide a humidity rich environment in the beginning the inside of the lid can be wiped with a piece of moist sponge or lint-free tissue to cover the surface in small droplets that evaporate fast and saturate the environment. If the drop does not dry at all or takes a much longer time to do so, the desiccant might be saturated and must be replaced.



Cover unused chip indentations in the Drop Casting Accessory with the supplied metal discs for optimal efficiency.



When the Drop Casting Accessory is not in use, remove the sponge from the container to avoid saturating the desiccant.

## 3.2 Spin casting procedure

Follow this procedure to spin cast a thin film of polystyrene on the surface of an EMILIE™ chip.

### Sample preparation

- Prepare a 1.0 wt% polystyrene solution in Toluene.
- Using the parameters provided below produces a film with an approximate thickness of 17 nanometers. Due to the perforations, thicker layers may form in the center.
- To adjust the film thickness, modify the concentration of the polystyrene solution accordingly.

### Procedure

- **Caution:** Membranes may break under the pressure difference exerted by the vacuum chuck. To prevent this, place a clean glass slide (e.g., a 10×10 mm microscope slide) between the chuck and the chip. To ensure the chip stays in place during the process, apply a small droplet of isopropanol (20 µL) between the glass slide and the chip. The added surface tension will enhance adhesion and prevent slipping.
- Place the chip-microscope slide assembly in the center of rotation of the spin-coating apparatus.
- Drop cast an adequate volume of the analyte to ensure the entire chip is covered (typically 20 µL or more).
- Perform the spin casting. After completion, gently slide off the chip from the glass slide using tweezers. Removing the chip by pulling upwards may cause damage to the membrane due to under-pressure between the membrane and the microscope slide.

Table 2: spin casting parameters

| Parameter            | Value             |
|----------------------|-------------------|
| Drop cast volume     | 20 µL             |
| Rotation Speed       | 2000 rpm for 60 s |
| Acceleration         | 500 rpm/s         |
| Post-bake (optional) | 90 °C for 1 min   |

### 3.3 Aerosol methods for airborne nanoparticles



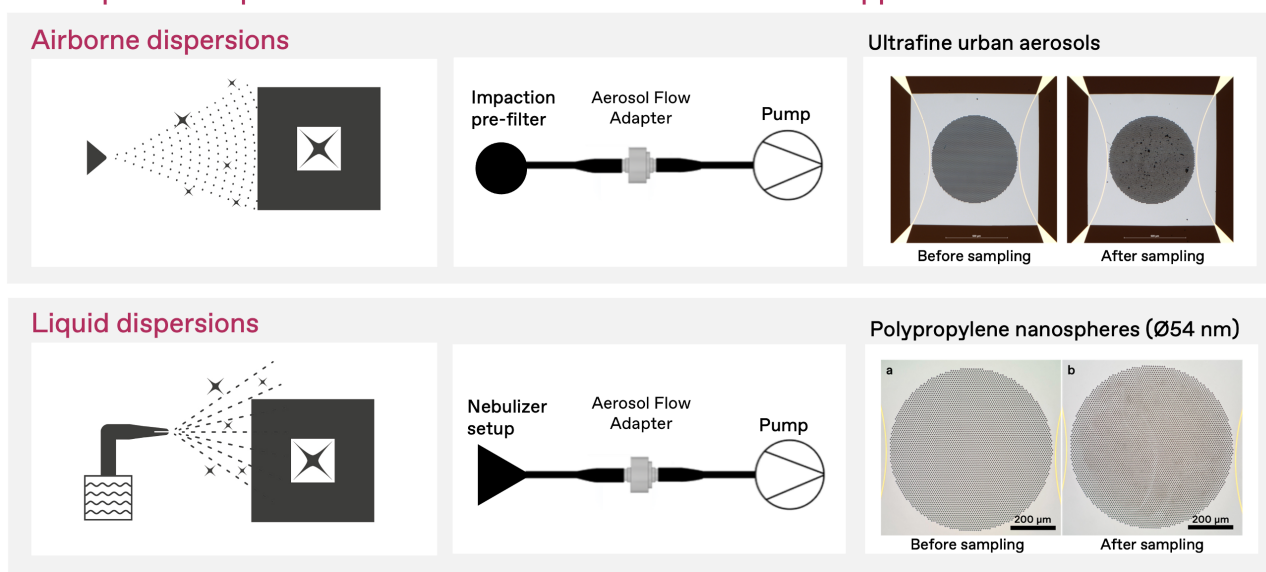
For optimal sample collection efficiency, the flow rate should be set to 1.0 L/min when collecting aerosols on the EMILIE™ chips. Higher flow rates can cause the membrane to break, and lower flow rates will result in a lower collection efficiency.



Avoid overloading the EMILIE™ chip by keeping your dry sample mass under 200 ng. A reduction in flow rate by 0.1 L/min is a good indication that the air flow through the chip pores is restricted by particles and that the loading limit of your chip is reached

An efficient technique to collect airborne nanoparticles is to collect them directly from an aerosol onto the EMILIE™ chip. An overview of the aerosol sample collection methods is presented in Figure 11. In these methods, the aerosol is pumped/pulled directed through the perforations of the EMILIE™ chip with the help of a vacuum pump. The airborne nanoparticles in the aerosol are collected on the EMILIE™ chip through an impaction process with a high efficiency of up to 60%. This method is adequate for airborne nanoparticles with a size as low as 10 nm and up to approximately 3200 nm.

**Nanoparticle capture size with the aerosol method:  $\varnothing 10\text{nm}$  – approx.  $3200\text{nm}$**



**Figure 11:** Analytes can be collected directly on the EMILIE™ chip using aerosol methods. This sample collection strategy is suitable for natural aerosols (airborne dispersions), nebulized liquid dispersions, solutions, and powders.

#### 3.3.1 The Aerosol Flow Adapter

The EMILIE™ Aerosol Flow Adapter, shown in Figure 12, provides a straightforward interface between the EMILIE™ chip and standard aerosol sample collection apparatus. The EMILIE™ chip

can be transferred from the Aerosol Flow Adapter to the EMILIE™ infrared analyzer without further sample processing (see Figure 13). The Aerosol Flow Adapter is available with either a slip or Luer Lock™ inlet. The outlet features a slip connector compatible with a 4 mm inner diameter tube.

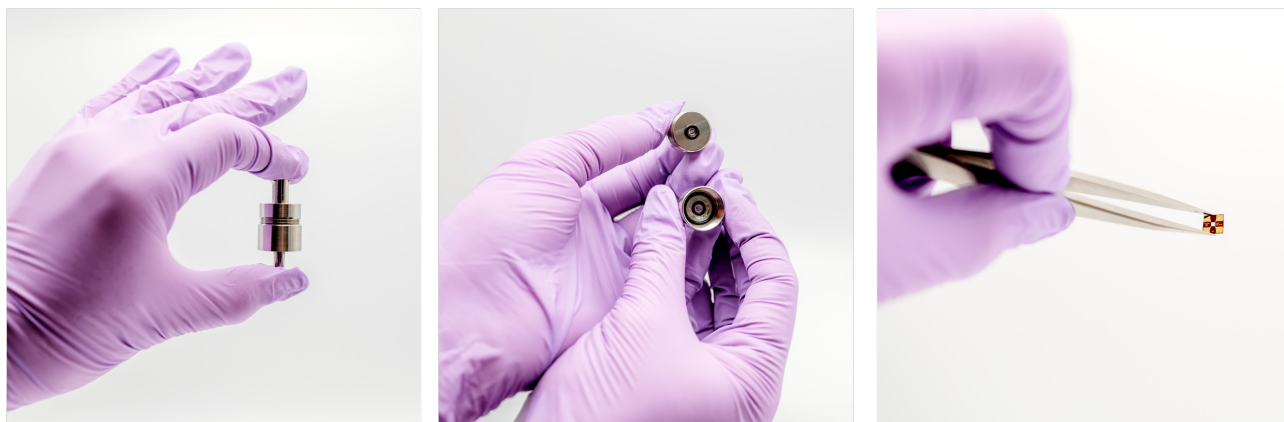


Figure 12: The Aerosol Flow Adapter .



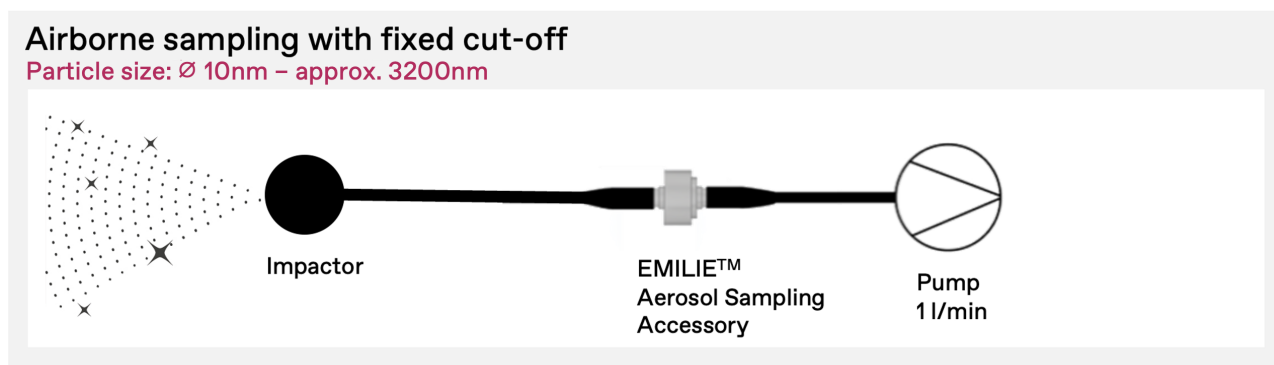
Figure 13: The EMILIE™ chip can be transferred from the Aerosol Flow Adapter to the EMILIE™ infrared analyser without further sample processing.

### 3.3.2 Particle selection by impaction

To avoid overloading the EMILIE™ chip, using the Aerosol Flow Adapter in combination with a particle size selection device upstream is recommended. Large particles can be removed effectively with an impactor featuring a suitable particle cut-off as shown in Figure 14.

A non-exhaustive list of compatible solutions that can be used to remove large particles is shown in Table 3.

For particle pre-selection with TSI's Mini-MOUDI™ (TSI GmbH), Invisible-Light Labs provides the Aerosol Impactor Adapter, designed specifically for use with Mini-MOUDI™ impactors. For the operation and particle pre-selection with a Mini-MOUDI™ we recommend using the Aerosol Impactor Adapter from Invisible-Light Labs, designed specifically for use with TSI's Mini-MOUDI™ impactors. The Aerosol Impactor Adapter accommodates two EMILIE™ chips for the simultaneous particle collection in duplicates. The Aerosol Impactor Adapter can be integrated after any



**Figure 14:** Recommended implementation of the Aerosol Flow Adapter in combination with an impactor pre-filter to remove large aerosol particles.

**Table 3:** Non-exhaustive list of impaction-based particle selection devices that can be used in conjunction with EMILIE™. \*Full compatibility to be determined for items marked by an asterisk

| Type                                   | Brand    | Model   |
|----------------------------------------|----------|---------|
| Mini-MOUDI™ 6 stages impactor (560 nm) | TSI GmbH | 135-6B  |
| Mini-MOUDI™ 8 stages impactor (180 nm) | TSI GmbH | 135-8B  |
| Mini-MOUDI™ 10 stages impactor (56 nm) | TSI GmbH | 135-10B |

cut-off stage of the Mini-MOUDI™ as shown in Figure 15. After loading the Aerosol Impactor Adapter with two chips, the desired stages for a specific particle size cut-off can be added on top. The Mini-MOUDI™ impactor needs to be operated at the nominal flow of 2.0 L/min.

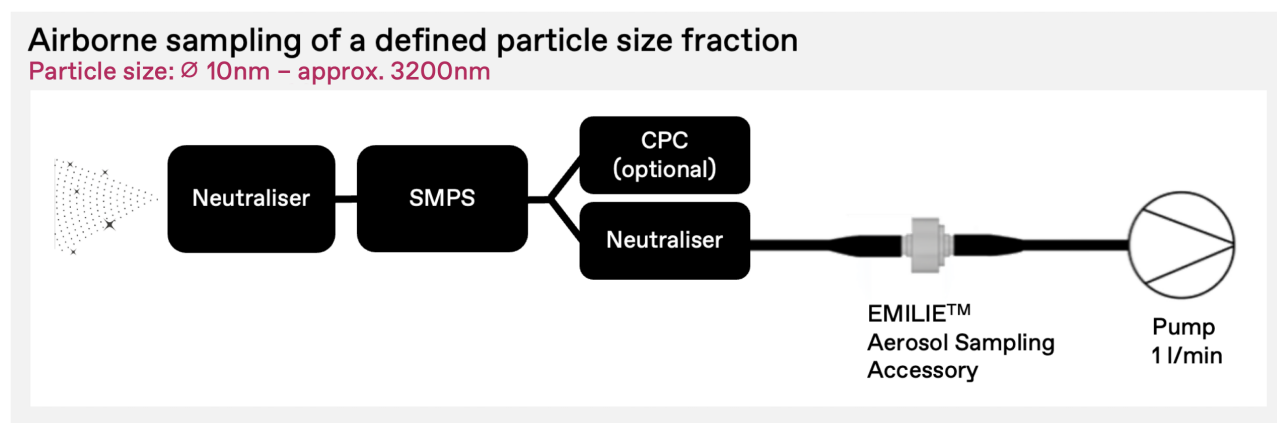


**Figure 15:** The Aerosol Impactor Adapter allows for sample collection on duplicate EMILIE™ chips simultaneously at any stage of a Mini-MOUDI™ impactor (TSI GmbH).

### 3.3.3 Particle selection with a scanning mobility analyser

A scanning mobility analyser can be used to collect an aerosol fraction with a defined particle size. Commonly, this is done with a Scanning Mobility Particle Sizer (SMPS) system in combination with a charge neutralizer and Condensation Particle Counter (CPC). Figure 16 shows the recommended implementation of the Aerosol Flow Adapter when used in combination with a particle sizing system.

To improve collection efficiency and avoid deflection of particles from the EMILIE™ chip, we recommend using electrostatic sizing techniques only in combination with a charge neutralizer. In combination with a CPC, as depicted in Figure 16, the particle count and size can be used to es-



**Figure 16:** Recommended implementation of Aerosol Flow Adapter in combination with a particle sizer system.

estimate the required collection time on the EMILIE™ chip. Table 4 shows a non-exhaustive list of commercially available sizing solutions suitable for use with the Aerosol Flow Adapter .

**Table 4:** List of particle sizing and neutralizer devices. \* Full compatibility to be determined for items marked by an asterisk

| Type                                  | Particle range (nm) | Brand       | Model      | Additional Notes                            |
|---------------------------------------|---------------------|-------------|------------|---------------------------------------------|
| SMPS                                  | 5 - 1000            | Brechtel    | 2100       | Adjustable flow 0.1 - 2.5 L/min             |
| SMPS                                  | 10 - 2000           | Brechtel    | 2102       | Adjustable flow 0.1 - 2.5 L/min             |
| SMPS*                                 | 10 - 500            | TSI         | 3938L50    | Adjustable flow 0.3 - 3 L/min               |
| SMPS*                                 | 4 - 400             | Airmodus    | MPSS       | Fixed flow 1.5 L/min (flow splitter needed) |
| Aerosol neutralizer (Soft X-ray)*     | Full range          | TSI         | 3088       | Fixed flow of 1.0 L/min                     |
| Aerosol neutralizer (63Ni radiation ) | Full range          | Durag Group | GRIMM 5523 | Fixed flow of 1.0 L/min                     |

**3.3.3.1 Particle counter** Particle concentration can be monitored with a particle counter to precisely control the sample mass deposited on the EMILIE™ chip . Table 5 provides a non-exhaustive list of particle counters suitable for determining particle concentration to estimate the sample collection time.

**Table 5:** List of particle counter devices.

| Type                                                                     | Brand    | Model         |
|--------------------------------------------------------------------------|----------|---------------|
| Particle counter (handheld device for size distribution & concentration) | Naneos   | Partector 2   |
| Wide-range Ambient Monitoring Scanning Mobility Particle Sizer (SMPS)    | TSI      | 3938W50-CEN10 |
| Scanning Electrical Mobility Spectrometer (SEMS)                         | Brechtel | Model 2100    |

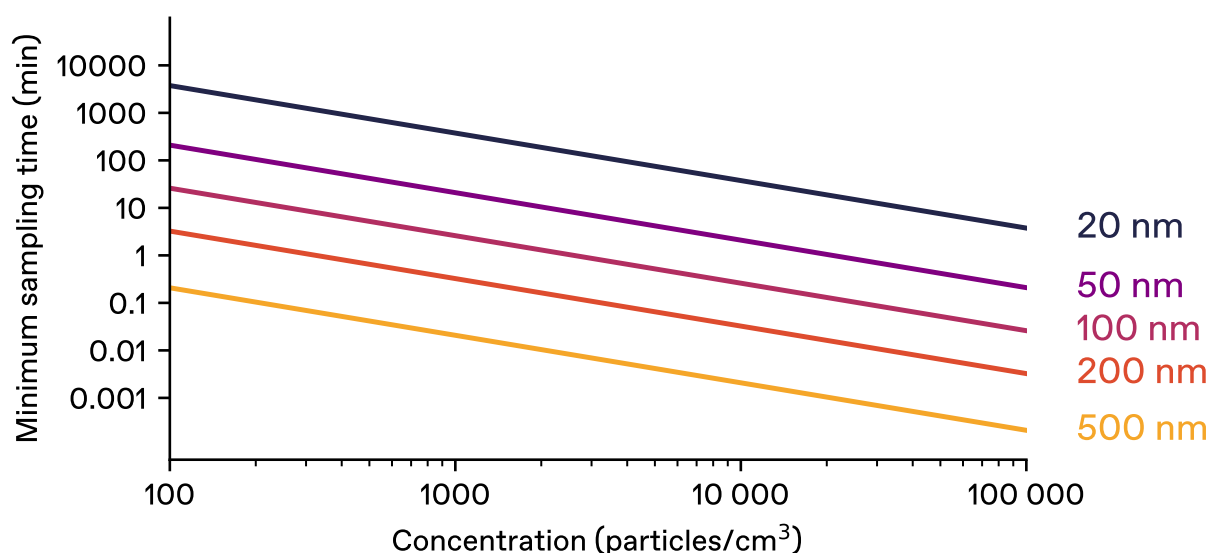
**Table 6:** Automated sample collection solution compatible with the Aerosol Flow Adapter

| Type                          | Brand    | Model      |
|-------------------------------|----------|------------|
| FILT 8-channel filter sampler | Brechtel | Model 9401 |

**3.3.3.2 Automated sample collection solutions** An automated sample collection solution can facilitate the remote sample collection on several EMILIE™ chips in the same experiment, such as the item listed in Table (6) in combination with the Aerosol Flow Adapter .

### 3.3.4 Sample collection time estimation

Figure 17 shows the estimated minimum sample collection time necessary to accumulate 1 ng material on the EMILIE™ chip assuming a particle density of 1000 kg/m<sup>3</sup>, a sample collection efficiency of 10% and a volume flow of 1 standard liter per minute (slpm). For optimal sample collection, we recommend measuring the particle concentration shortly before sampling using a particle counter. Table 7 provides suggested sample collection times for environmental aerosols based on various particle concentrations.



**Figure 17:** Estimate of the minimum sample collection time as a function of the aerodynamic particle diameter and number concentration.

### 3.3.5 Aerosol sample collection procedure with the Aerosol Flow Adapter

Loading a EMILIE™ chip in the Aerosol Flow Adapter :

- Unscrew and open the Aerosol Flow Adapter . The lower part of the Aerosol Flow Adapter features a groove and O-ring for placing the EMILIE™ chip . The upper part features a flat surface and an O-ring to ensure a tight seal.
- Place the desired EMILIE™ chip in the groove of the lower part of the aerosol flow adapter, ensuring the gold electrodes and markings on the EMILIE™ chip are facing upwards.

**Table 7:** Initial sample collection time suggestions for environmental (indoor/outdoor) aerosols. The optimal sampling time will depend on specific experimental conditions.

| Particle concentration particle/cm <sup>3</sup> | Suggested sampling time (min) |
|-------------------------------------------------|-------------------------------|
| 1000                                            | 30                            |
| 2500                                            | 12                            |
| 5000                                            | 6                             |
| 10,000                                          | 4                             |
| 20,000                                          | 2                             |

- Screw in the counterpart of the Aerosol Flow Adapter . Make sure that the lower part remains upright while screwing the two parts together to prevent the chip from sliding out of the groove. Screw the top part until you feel resistance. Gently, continue to screw until hand-tight. Avoiding over-tightening. The Aerosol Flow Adapter can now be integrated into your airflow system.

#### Sample collection:



Do not exceed 1.0 L/min to avoid breaking the EMILIE™ chips . A sudden increase in flow rate indicates a broken chip. A reduction in flow rate by 0.1 L/min from the initial flow at the start of sample collection indicates a sufficiently loaded chip.



Note that when collecting samples with the Aerosol Impactor Adapter in a Mini-MOUDI™ impactor (TSI GmbH), the flow rate must be adjusted to 2.0 L/min. A lower flow rate will affect the particle cut-off and allow larger particles onto the stage.

- Start your particle counter if using one. Table 7 provides basic guidance on sample collection times based on the number of particles present in the environment.
- Start your sampling pump and set the flow rate to a maximum of 1.0 L/min.
- Collect particles for the desired time or until a reduction in flow rate by 0.1 L/min is observed (i.e. when the flow rate is reduced to 0.9 L/min if the initial flow rate was 1 L/min).
- Switch off the pump to end sample collection.

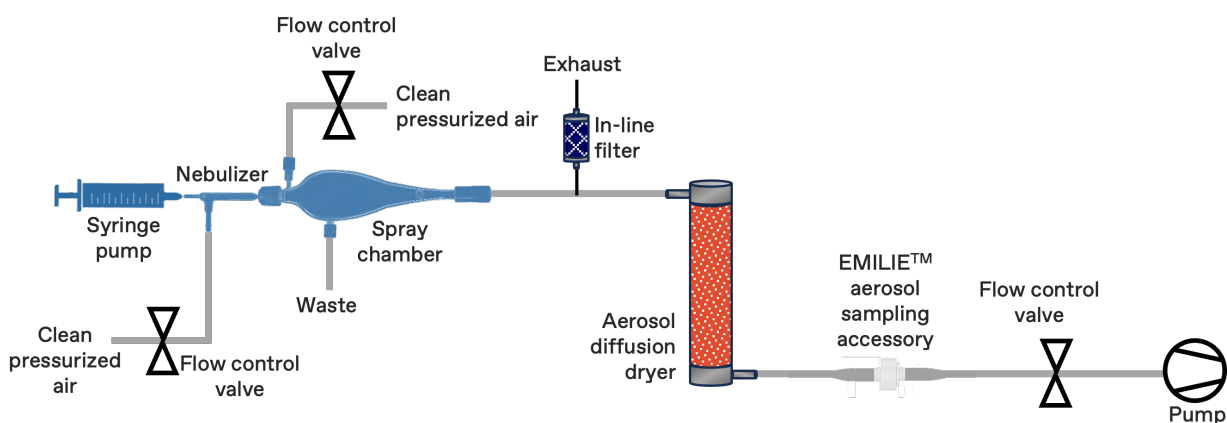
#### Unloading the EMILIE™ chips :

- Switch off the pump.
- Unscrew the Aerosol Flow Adapter to unload your EMILIE™ chip . Make sure the lower part remains upright during the unscrewing procedure to prevent the sampled chip from sliding out of the groove.

- Be aware that the EMILIE™ chip might remain stuck to the O-ring of the top part of the Aerosol Flow Adapter, or the O-ring to the EMILIE™ chip surface, especially if it was closed for a long time. In these events, hold the EMILIE™ chip from one side using tweezers and gently pull it off the O-ring.
- Place your EMILIE™ chip in an appropriate storage container and protected from contaminants until analysis with EMILIE™.

### 3.4 Aerosol methods for liquid nanoparticle dispersions

When collecting samples from a liquid dispersion or a solution, the Aerosol Flow Adapter can be seamlessly interfaced with a nebulizer and spray chamber apparatus, similar to those commonly used in elemental spectroscopy methods. An example is given in Figure 18.



**Figure 18:** Liquid dispersions are aerosolized via, for example, a concentric nebulizer and spray chamber, and analytes are collected on the EMILIE™ chip with the help of the Aerosol Flow Adapter.

#### 3.4.1 Procedure for sample collection from liquid dispersions

This section describes briefly the procedure for aerosol sample collection from a nanoparticle dispersion using the apparatus illustrated in Figure 18.

- Fill the syringe with the analyte dispersion and connect it to the capillary that leads to the nebulizer.
- Turn on the air supply and set the flow meters to the recommended values (see Table 8) before starting the syringe pump.
- Flush the system for approximately 10 minutes with the solution.
- Place a used but intact chip in the Aerosol Flow Adapter during flushing and adjust the flow rates to recommended values (see Table 8).

**Table 8:** Example parameters for aerosol sample collection from liquid dispersions (see Figure 18).

| Item                             | Recommended setting    |
|----------------------------------|------------------------|
| Syringe Pump flowrate            | 10 µL/min              |
| Nebulizer gas                    | 0.45-0.50 L/min        |
| Make-up flow (spray chamber)     | 0.45-0.50 L/min        |
| Flow rate before the pump        | approx. 0.90-1.0 L/min |
| Typical suspension concentration | approx. 20 µg/L        |



For the highest sample collection efficiency, the flow rate should be 1.0 L/min when collecting aerosols on the EMILIE™ chips. Higher flow rates cause the membrane to break and lower flow rates result in a lower collection efficiency.



The use of a vortex mixer or ultrasonic bath with the dispersion is recommended to produce homogeneous emulsions.

To ensure that the system flow rates are balanced, place the spray chamber waste line in a vial filled with water, and block the exhaust line completely (e.g. by blocking the exhaust with a gloved finger). One out of three scenarios are possible:

1. Air comes out of the waste line and creates bubbles in the water - this means that a larger amount of air enters the system than the pump at the end of the system extracts. Reduce the flow rates from the nebulizer and spray chamber, or increase the flow rate on the last control valve right before the pump making sure not to exceed the 1.0 L/min limit.
2. Water from the vial enters the waste line and goes up to the spray chamber - this means that more air leaves the system than enters. Increase the flow rates from the nebulizer and spray chamber, or decrease the flow rate on the last control valve right before the pump.
3. Water is at rest. The system is balanced and ready to use.

Once the flow rates have been adjusted and balanced - no further adjustments are necessary. Pre-concentration of very dilute suspensions can be achieved by adjusting the sample collection time accordingly. To stop sample collection, turn off the pumps and retrieve your loaded chip from the Aerosol Flow Adapter. Always rinse the system with a suitable media followed by distilled deionized water after use. Rinsing times depend on the analytes and matrices.