



km. Notably, both laboratory and field measurements revealed minor signals of traditional toxic brake pad constituents such as Cu and Sb, suggesting a shift toward alternative materials in modern brake pad formulations. Our results emphasize the need to integrate BWP into air quality monitoring and regulatory frameworks, particularly using chemically specific tracers to better understand their environmental and health impacts.

## 1. Introduction

As regulatory efforts led to significant reductions in tailpipe emissions from internal combustion engines (Giechaskiel et al., 2014), attention has increasingly shifted toward non-exhaust vehicle-related particulate emissions (NEE). These emissions, which include brake wear, tire wear, road surface abrasion, and resuspended road dust, have emerged as dominant contributors to urban particulate matter (PM), especially in the PM<sub>10</sub> and PM<sub>2.5</sub> size fractions (Grigoratos and Martini, 2014). Recent studies have also demonstrated that NEE particles can be in the ultrafine size range (UFP, particle diameter < 100 nm) (Neukirchen et al., 2025). Among various NEEs, brake wear particles (BWP) have gained growing concern due to their small size and chemically complex composition. These particles are generated through the mechanical abrasion and thermal degradation of brake pad and disc materials, which have been reported to account for up to 16–55 % of PM<sub>10</sub> emissions in urban environments (Piscitello et al., 2021). As the transition to electric vehicles reduces tailpipe emissions, the relative contribution of NEEs to the total road traffic-related emissions is expected to increase, particularly due to heavier vehicle weight (Wagner et al., 2024).

Fine particulate matter from BWP consists of a heterogeneous mixture of metals (e.g. Fe, Cu, Zn, Sn, Sb), carbonaceous material, oxidized organics (Grigoratos and Martini, 2014). Due to their chemical composition and small size, which enables them to remain suspended in air and to be inhaled deeply into the respiratory tract, they have been considered to induce oxidative stress as well as pro-inflammatory effects (Gasser et al., 2009). Recent toxicological studies have also shown that BWP can induce not only oxidative stress and inflammation responses, but also DNA damage in mammalian models (Forest and Pourchez, 2023). Furthermore, BWP are often rich in transition metals such as Fe and Cu, which have been associated with cytotoxicity as well as pulmonary inflammation (Barosova et al., 2018). In addition, BWP have been found to be a potential threat to the environment, acting as major contributors to atmospheric Cu and to the contamination of ocean waters (Rossetol, 2005; Hulskotte et al., 2007). Despite various risks, BWP are not yet specifically subject to dedicated emission regulation in most countries. Upcoming standards such as EURO 7 aim to reduce BWP releases by introducing limit values for brake PM emissions, with proposed thresholds of 7 mg/km for light-duty vehicles and 3 mg/km for electric vehicles (Dornoff and Rodríguez, 2024).

To understand the physical and chemical characteristics of BWP, previous studies have investigated both laboratory and real-world measurements. Laboratory dynamometer studies have shown that driving cycle, brake temperature, and drag strongly influence BWP emissions. Wahlström et al. (2017) conducted a detailed laboratory study using a pin-on-disc tribometer to investigate the wear and airborne particle emissions from various disc brake contact material combinations, emphasizing that targeted material designs, especially disc surface engineering, can substantially lower NEE under controlled laboratory conditions. Farwick zum Hagen et al. (2019a) found that UFP emissions occurred only when critical disc temperatures of 140–170 °C were exceeded, while 34 % of the PM mass might be attributed to the brake drag even without active braking. Liu et al. (2023) demonstrated both laboratory and field measurement of BWP in a tunnel using a single-particle mass spectrometer (SPMS). While tunnel particles were directly sampled into the SPMS, laboratory-generated BWP were collected and resuspended in a flask and subsequently analyzed by SPMS. Durif et al. (2025) recently applied a pin-on-disc tribometer

combined with a real-time proton-transfer reaction time-of-flight mass spectrometer, highlighting the importance of gaseous emissions in addition to particulate mass in understanding NEE. The release of VOCs from brake pads was also demonstrated by Patel et al. (2024), showing that pyrolysis of phenolic resin binders can undergo photooxidation and form secondary particulate matter in oxidation flow reactors. Hence, BWP contribute not only to primary PM but can also lead to secondary aerosol formation. Complementary field work has emphasized the role of real driving dynamics. Al Wasif-Ruiz et al. (2025) recently measured BWP directly from a passenger vehicle during on-road braking events using a mobile laboratory. The study shows that high UFP concentrations are observed during harsh braking events, which can be attributed to brake pads and brake discs-based particles according to elemental analysis. Farwick zum Hagen et al. (2019b) also conducted on-road measurements using a cone-shaped collector mounted on the outer rim on a closed test track, highlighting the relevance of pad aging in terms of lower UFP emissions and confirming the significance of real-world driving dynamics on emission profiles. While these previous studies have provided important insights into freshly emitted BWP, including their physical and chemical characteristics and toxicological effects, comparatively fewer investigations have focused on resuspended BWP, which can persist on road surfaces and can be transported over longer distances. According to Harrison et al. (2012), resuspended road dust can contribute substantially to the coarse particle mass fraction, accounting for around 38 % at a heavily trafficked roadside site in London. In the absence of precipitation and under low-wind conditions such resuspended particles can persist on the road surface for extended periods and are thus available for continuous re-entrainment by passing vehicles. However, due to the difficulties of separation owing to the complex mixture of atmospheric particles and different emission sources, specific source apportionment of airborne PM with resuspended particles remains a key challenge in urban environments, particularly in traffic hotspots. While exhaust emissions can often be assigned by several independent markers such as NO<sub>x</sub>, CO, black carbon through established measurement techniques, NEE generally require a detailed breakdown of the chemical particle composition. SPMS has emerged as a powerful technique for this purpose, offering molecular-level chemical identification and sizing of individual particles with high temporal resolution. A recent study by Passig et al. (2022) showed its capability to differentiate between various combustion sources including biomass burning, diesel traffic, marine emissions based on simultaneous detection of inorganic and organic constituents. This capability enables real-time source apportionment, effectively overcoming limitations of conventional bulk filter analysis and making it particularly suitable for separating complex urban mixtures where brake wear, tire wear, and road dust frequently overlap.

In this study, we present a comparative analysis of BWP under two distinct scenarios. First, BWP were generated under controlled brake dynamometer conditions, closely following those defined by the GTR24 and the Worldwide harmonized Light Vehicle Test Procedure (WLTP) brake cycle. Airborne particles were directly measured using SPMS and other particle characterization instruments to obtain a comprehensive profile of their chemical and physical properties. These were compared using single-particle scanning electron microscope combined with energy dispersive X-ray spectroscopy (SEM-EDX) analysis of filter samples. Second, ambient particles were measured in close proximity to a highway tunnel entrance, where frequent heavy braking is expected due to a reduced speed limit, and at an urban background station to assess the potential atmospheric transport of these particles.

By investigating BWP characteristics under standardized, reproducible conditions, we minimized the influence of potentially disturbing factors such as meteorological variations and overlapping with other traffic emissions. This approach allowed us to develop distinctive spectral fingerprints for BWP validated under real-world conditions and to contribute to the growing needs for source-resolved air quality data, which is crucial for improving health risk assessments, urban planning strategies, and future regulatory frameworks.

## 2. Material and methods

### 2.1. Laboratory measurements

#### 2.1.1. Brake dyno setup and test cycle procedure

Brake particle measurements were carried out on a custom-built dynamometer designed in accordance with the technical requirements specified in GTR (Global Technical Regulation) No.24 as defined by the particle measurement program (UNECE, 2024). The setup incorporates a constant volume sampler (CVS) and isokinetic sampling probes to maintain stable and representative airflow conditions. The brake hardware, including the rotor, caliper, and pad materials, as well as the pre-conditioning and operational procedures, were identical to those described by Neukirchen et al. (2025). To emulate on-road braking behavior, the Worldwide harmonized Light vehicles Test Procedure (WLTP) brake cycle was applied. All experiments used original equipment manufacturer (OEM)-supplied 330 mm  $\times$  24 mm vented braked discs and caliper suitable for a 17-inch wheel assembly. Low-metallic (LM) brake pads were installed for the measurements. The brake disc temperature was monitored by thermoelements (positioned according to GTR24 specifications) at a data acquisition rate of 10 Hz.

The WLTP cycle consists of 303 individual braking events distributed over 10 trips. In compliance with GTR24, at least five complete WLTP cycles were performed to pre-condition new discs and pads prior to data collection. The pre-conditioning process ensures the formation of a stable friction layer on the disc surface and stabilizes the friction coefficient, which is critical for achieving representative and repeatable brake wear particle emissions and braking behavior (Grigoratos and Martini, 2015). During a cycle, the temperature of the disc was monitored until it dropped below 40 °C before a new trip was started, which is defined as soak time. Cooling air flow rates were adjusted in line with GTR24 by determining the ratio of wheel load to disc mass and verifying brake temperatures. The CVS flow was set to 540 m<sup>3</sup>/h. To ensure high repeatability of the emission rates and particle characteristics, two measurements without soak time and three measurements with soak time were conducted under identical conditions. The variation in terms of the total particle number and size distribution between these conditions was minimal. An overview of the variation between the experiments is given in Tables S1 and S2.

#### 2.1.2. Online measurements and filter sampling

During testing, the particle instruments are configured and located as follows: An optical particle spectrometer (Aerosol Particle Size Spectrometer LAP 322, Topas GmbH, Germany) was placed inside the brake test bench cell and measured the particle number size distribution from 0.3  $\mu$ m to 2.5  $\mu$ m at 0.5 Hz. Samples for SEM-EDX measurements were drawn onto 47 mm polycarbonate (PC) track-etched membrane filters (Whatman, Nuclepore, 2  $\mu$ m pore-size, Germany) which were loaded in the PM<sub>10</sub> and PM<sub>2.5</sub> filter holders. A Q150T ES Plus sputter coater (Quorum technologies, UK) was used to coat PC filters with 10–20 nm of a conductive carbon layer using a woven carbon fiber thread (density 1.55 g/m, Quorum technologies, UK) in pulsed cord evaporation mode. All other instruments were placed outside of the test bench cell and were individually connected via stainless steel tubes and conductive sampling lines. The aerosol flow was diluted at a ratio of 1:25 using a portable dilution system (Dekati eDiluter Pro, Dekati Ltd., Finland). The dilution air was supplied by a zero-air generator (AADCO 717 15, Tisch

Environmental Inc., USA) to eliminate background contamination. A condensation particle counter (CPC5420, Grimm Aerosol Technik GmbH, Germany) measured the total particle number concentration (TPNC). Size-resolved particle number distributions were obtained via a separate, undiluted sampling line using a Fast Aerosol Sizer (DMS500 Mk II, Cambustion Ltd., UK), which covered a size range from 5 nm to 1  $\mu$ m at a 1 Hz time resolution. Chemical and size-resolved single particle analysis was performed in real time with a single-particle mass spectrometer (SPMS, Photonion GmbH, Germany). The operational principles and setup parameters of the SPMS are detailed in previous studies (Pratt and Prather, 2012; Schade et al., 2019; Passig and Zimmermann, 2021). In brief, aerosol particles are transferred into a vacuum chamber via an aerodynamic lens (Wang et al., 2005) and passed through a pair of continuous-wave Nd:YAG lasers (50 mW, 532 nm). The particles scatter light upon interaction with the lasers, allowing their detection by photomultipliers and enabling estimation of their aerodynamic diameter via velocimetric sizing. Subsequently, the particles enter the ionization stage, where a series of laser pulses hit each particle. Initially, a CO<sub>2</sub> infrared laser pulse (10.6  $\mu$ m, GAM Laser Inc., USA) thermally desorbs surface-bound species such as polycyclic aromatic hydrocarbons (PAHs). These desorbed species are then subjected to resonance-enhanced multiphoton ionization (REMPI) with an unfocused krypton fluoride (KrF) excimer laser (248 nm,  $\sim$ 3 MW/cm<sup>2</sup>, MLase GmbH, Germany). The same laser beam is subsequently reflected and focused via a concave mirror to intensities  $\sim$ 2 GW/cm<sup>2</sup>, inducing laser desorption ionization (LDI) of the remaining particle core. Positive and negative ions are extracted into two time-of-flight mass spectrometers, generating bipolar mass spectra from which individual particle composition can be determined with unit-mass resolution. Spectra with less than 3 peaks were regarded as not valid. Before each measurement campaign, the SPMS was calibrated using a certified diesel particulate matter standard (SRM 2975, NIST, USA) to ensure reproducible ionization performance and accurate mass calibration throughout the measurements. It is noteworthy that the instruments used in this study classify particles according to different physical principles. The SPMS measures particle aerodynamic diameter, while the DMS and SMPS report electrical mobility diameter. In contrast, OPS and EDM determine scattered light equivalent diameter. Because these sizing metrics are not directly equivalent, the given particle diameters should not be conflated.

#### 2.1.3. SEM-EDX analysis

12 mm-diameter samples from the loaded carbon-coated PC filters were cut and adhered to SEM pin stubs using conductive high purity EDX-suitable double-sided carbon adhesive pads (Spectro-Tabs, Plano GmbH, Germany). Carbon cement (Micro to Nano BV, EM-Tec C38, Netherland) was used to seal the edges of the stub to ensure the grounding path from the sample to the stub. Prepared SEM samples were stored in a desiccator under vacuum for 24 h to ensure the removal of volatile compounds. A Gemini SEM 360 (Carl Zeiss, Germany) field emission SEM equipped with InLens and SE2 detectors was used to obtain SEM micrographs at acceleration voltages of 2–2.5 kV. An Ultim Max 40 EDX detector (Oxford Instruments, UK) equipped with a thin polymer detector window and a silicon drift detector allowed for the measurement of low Z elements (Z > 6). To minimize the piercing of the electron beam through the particles, acceleration voltages of 12 kV and 5 kV were used to analyze particles with a geometric diameter of >200 nm and < 200 nm, respectively (Neukirchen et al., 2025).

## 2.2. Field measurement

Outdoor aerosol measurements were conducted at two different sites at the campus of the University of the Bundeswehr Munich, which is located in Neubiberg, southeast of the city of Munich, Germany. The first site is near a major highway with expected high traffic emissions. The second site is in a relatively unaffected background area to enable a comparative analysis of traffic-related emissions and urban background.













mass contribution of atmospheric BWP to PM<sub>2.5</sub>. Consequently, further research is needed to enhance both the detection sensitivity and the chemical differentiation of various particle types. Expanding real-time analysis methods to include other non-exhaust sources, such as tire wear particles, is also essential for a more complete understanding of traffic-related emissions.

#### 4. Conclusion

This study presents an approach that links laboratory studies with field measurements, focusing on the physical and chemical characteristics of brake wear particles. Our results were obtained by combining controlled laboratory dynamometer experiments with real-world highway and urban background measurements to identify suitable BWP spectral markers for source apportionment. Under standardized WLTP brake cycle conditions, single-particle mass spectrometer consistently identified three distinct particle groups: Ba-containing particles, K-rich particles mixed with organics, and carbonaceous particles. The particle groups and compositions were confirmed by SEM-EDX analysis of single particles sampled onto carbon-coated PC filters. These chemical compounds highlight that BWP is formed not only by mechanical abrasion but also by temperature-driven volatilization and condensation of organic constituents, leading to different chemical compositions and aerodynamic particle sizes for each group. The laboratory results show that Ba-containing particles had the dominant relative proportion of BWP followed by K-rich and carbonaceous particles. The suitability of the classified BWP markers was validated in field measurements. The detection of BWP signatures at a highway station confirmed the chemical resilience of Ba-containing particles, while K-rich and carbonaceous particles were less clearly identified due to their complex mixing and overlap with other chemical components. Measurements at the urban background indicate the chemical resilience and transport potential of Ba-containing particles. Despite atmospheric aging, these particles can be clearly assigned to BWP by pronounced Fe and Ba signals. Moreover, the laboratory and field data suggest a transition toward more environmentally and human-health friendly brake pad formulations, evidenced by less-prominence of Cu and Sb signals, which have been increasingly replaced by alternative elements such as Bi. Nevertheless, the study findings underscore the importance of incorporating BWP into broader regulatory frameworks, especially with respect to their chemical compositions and particle size range. Fine particulate matter metrics such as PM<sub>2.5</sub> and PM<sub>1</sub> which capture the alveolar and more health-relevant fraction, might be a better metric for quantifying BWP and non-exhaust traffic emissions. Given that BWP represent a significant portion of traffic-related particulate emissions, further research and the development of targeted emission reduction strategies are essential before more stringent regulations are implemented.

#### CRedit authorship contribution statement

**Seongho Jeong:** Visualization, Validation, Methodology, Investigation, Data curation, Conceptualization, Writing review & editing, Writing original draft. **Chi-Long Tang:** Visualization, Validation, Investigation, Data curation, Conceptualization, Writing review & editing. **Carsten Neukirchen:** Methodology, Investigation, Conceptualization, Writing review & editing. **Gary Sean Cooney:** Visualization, Validation, Investigation, Writing review & editing. **Julian Schade:** Visualization, Validation, Methodology, Investigation, Conceptualization, Writing review & editing. **Patrick Martens:** Investigation, Writing review & editing. **Michael Mäder:** Investigation. **Heinrich Ruser:** Investigation, Writing review & editing. **Thomas Adam:** Resources, Project administration, Funding acquisition, Conceptualization, Writing review & editing.

#### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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#### Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.scitotenv.2025.181277>.

#### Data availability

Data will be made available on request.

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## Glossary

- ART-2a*: Adaptive Resonance Theory  
*BWP*: Brake wear particle  
*CPC*: Condensation particle counter  
*CVS*: Constant Volume Sampler  
*GMD*: Geometric Mean Diameter  
*GSD*: Geometric Standard Deviation  
*GTR*: Global Technical Regulation  
*LDI*: Laser desorption ionization  
*LM*: Low metallic  
*NEE*: Non-exhaust emission  
*OEM*: Original Equipment Manufacturer  
*PAH*: Polycyclic aromatic hydrocarbon  
*PN*: Particle number  
*PM*: Particulate matter  
*REMPI*: Resonance enhanced multiphoton ionization  
*SEM-EDX*: Scanning electron microscopy energy dispersive X-ray spectroscopy  
*SMPS*: Scanning mobility particle sizer  
*SPMS*: Single-particle mass spectrometer  
*TOF*: Time of Flight  
*TPNC*: Total particle number concentration  
*UFP*: Ultrafine particle  
*UNECE*: United Nations Economic Commission for Europe  
*WLTP*: Worldwide harmonized Light Vehicle Test Procedure