

Analytical methods for detection and size determination of micro- and nanoscale plastic debris in water

MOTIVATION AND AIM

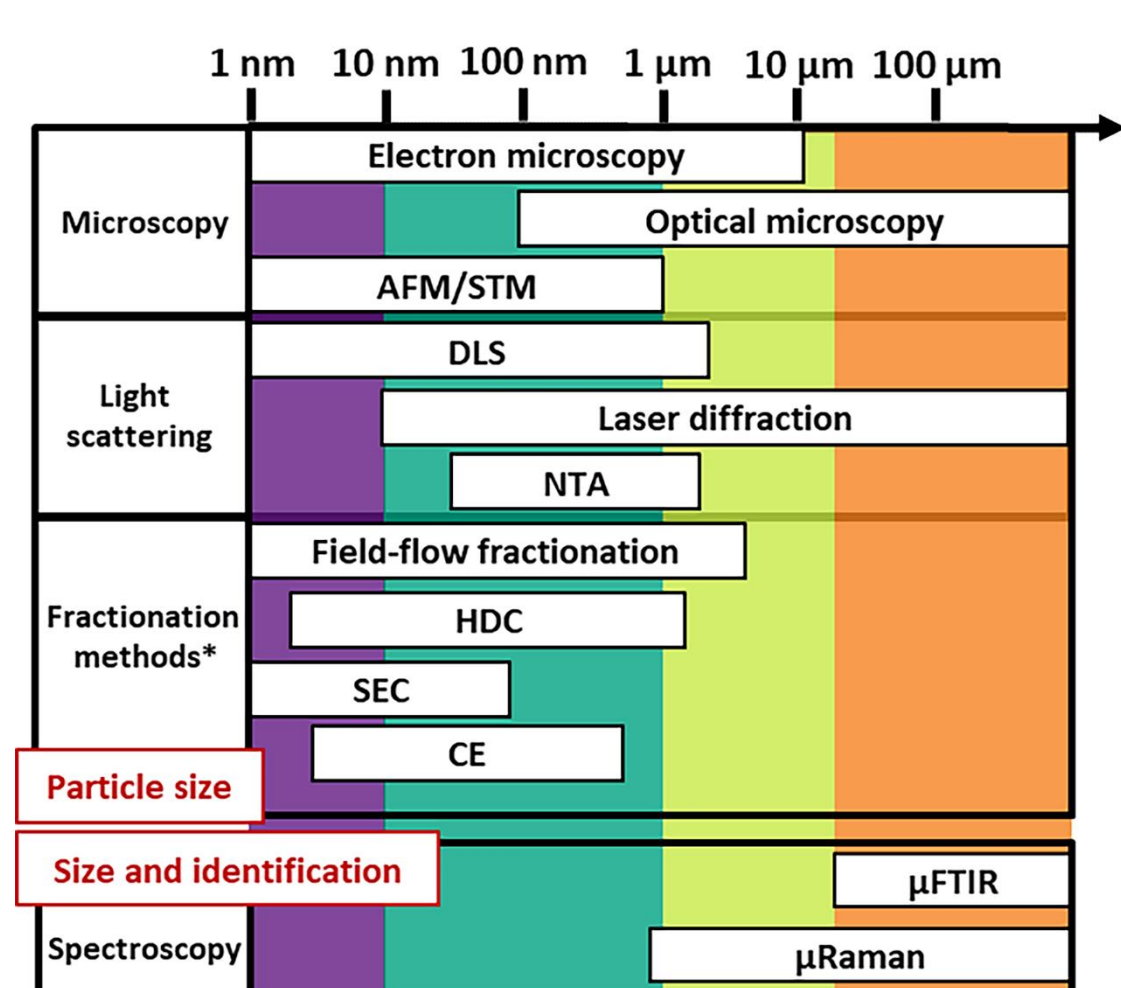


Figure 1. Comparison of mainstream analytical techniques to characterize MPs and NPs in terms of particle size and/or composition. (Velimirovic et al., 2021).

- Development of analytical methods for the characterization (particle size determination, chemical identification, and quantification) of the low μm -range microplastic (MPs; 1-10 μm) and nanoscale plastic (NPs; 1-1000 nm) debris in environmental matrices is a quickly emerging scientific field and has gained considerable attention, not only within the scientific community, but also on the part of policy makers and the general public.
- However, due to the limited sensitivity of the current state of the art monitoring techniques (Figure 1), detection of low μm -range MPs and NPs in different environmental compartments and especially in water is one of the biggest challenges for their monitoring, source identification and, ultimately, risk assessment.
- The main aim of this study is to give an overview of different complementary analytical methodologies showing considerable promise for the particle size determination, chemical identification, and quantification of MPs and NPs.

Single particle inductively coupled plasma mass spectrometry for MPs size determination and quantification

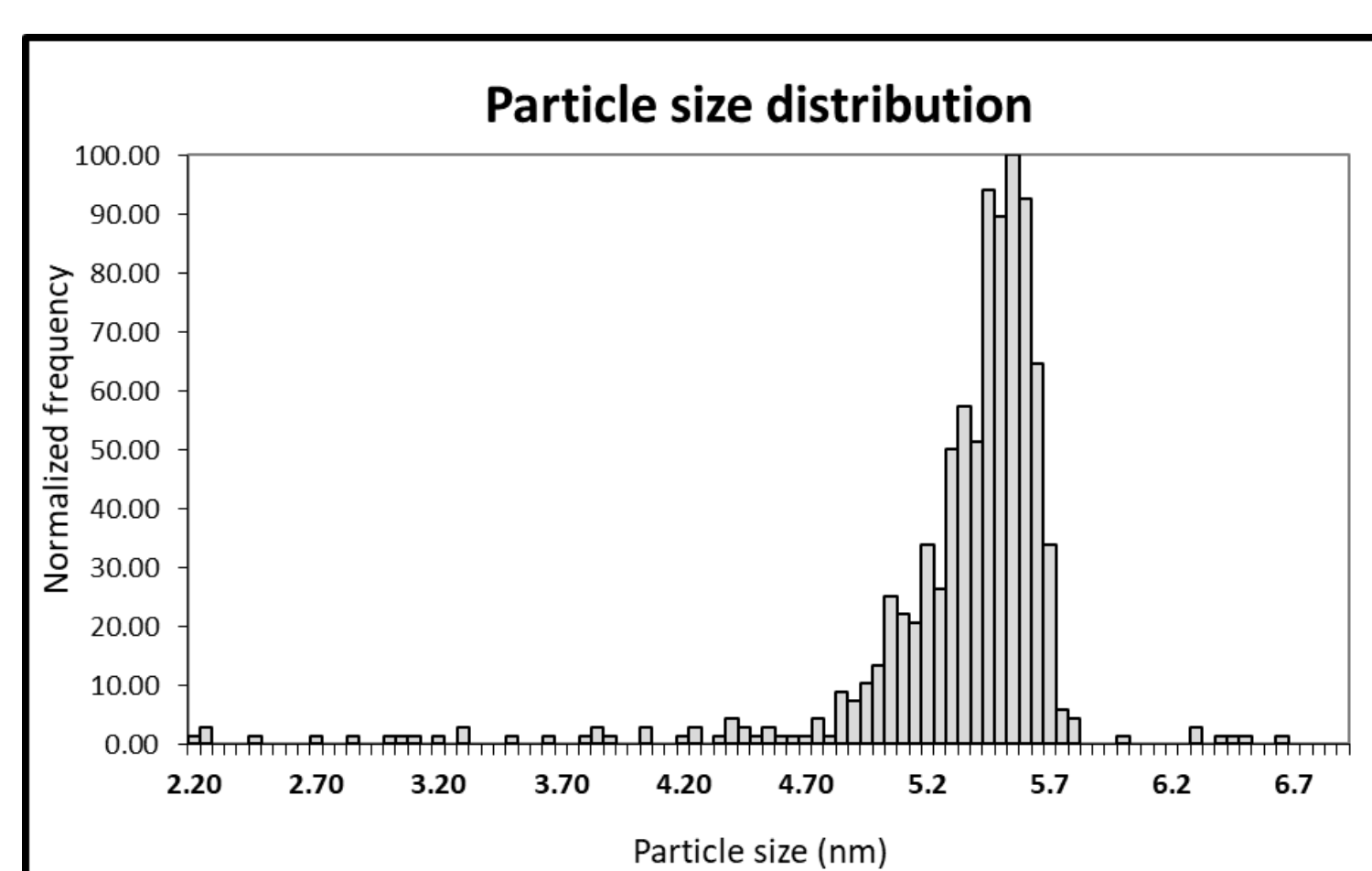
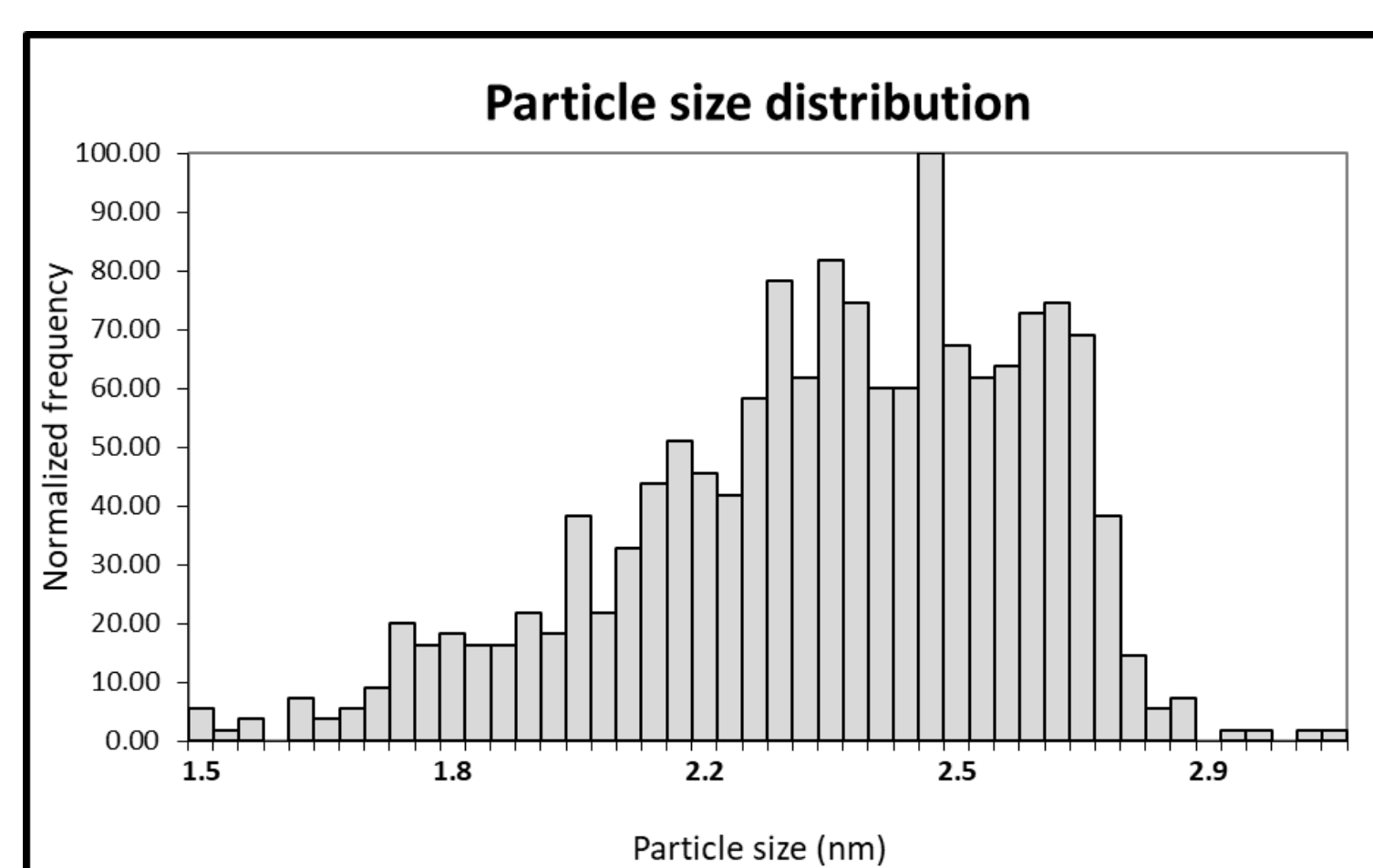


Figure 2. Particle size distribution obtained for 2.5 μm and 5 μm polystyrene (PS) microspheres measured using ICP-MS operated in single-event mode via the monitoring of $^{13}\text{C}^+$.

- Individual plastic particles are observed as short transient signals or signal spikes upon ultra-fast continuous monitoring (with a detector dwell time of 100 μs) of the intensity at a mass-to-charge ratio (m/z) of 13 ($^{13}\text{C}^+$) using an ICP-MS unit (Nu AttoM ES, Nu Instruments, UK) operated in single-event mode (Figure 2) following the procedure reported for the first time by Bolea Fernandez et al., 2020.
- The results obtained for PS microspheres (2.5 and 5 μm) in terms of size (most frequently occurring integrated intensity of the signal distribution) compared well with the size as determined using electron microscopy.

Nanoparticle tracking analysis for NPs size determination and quantification

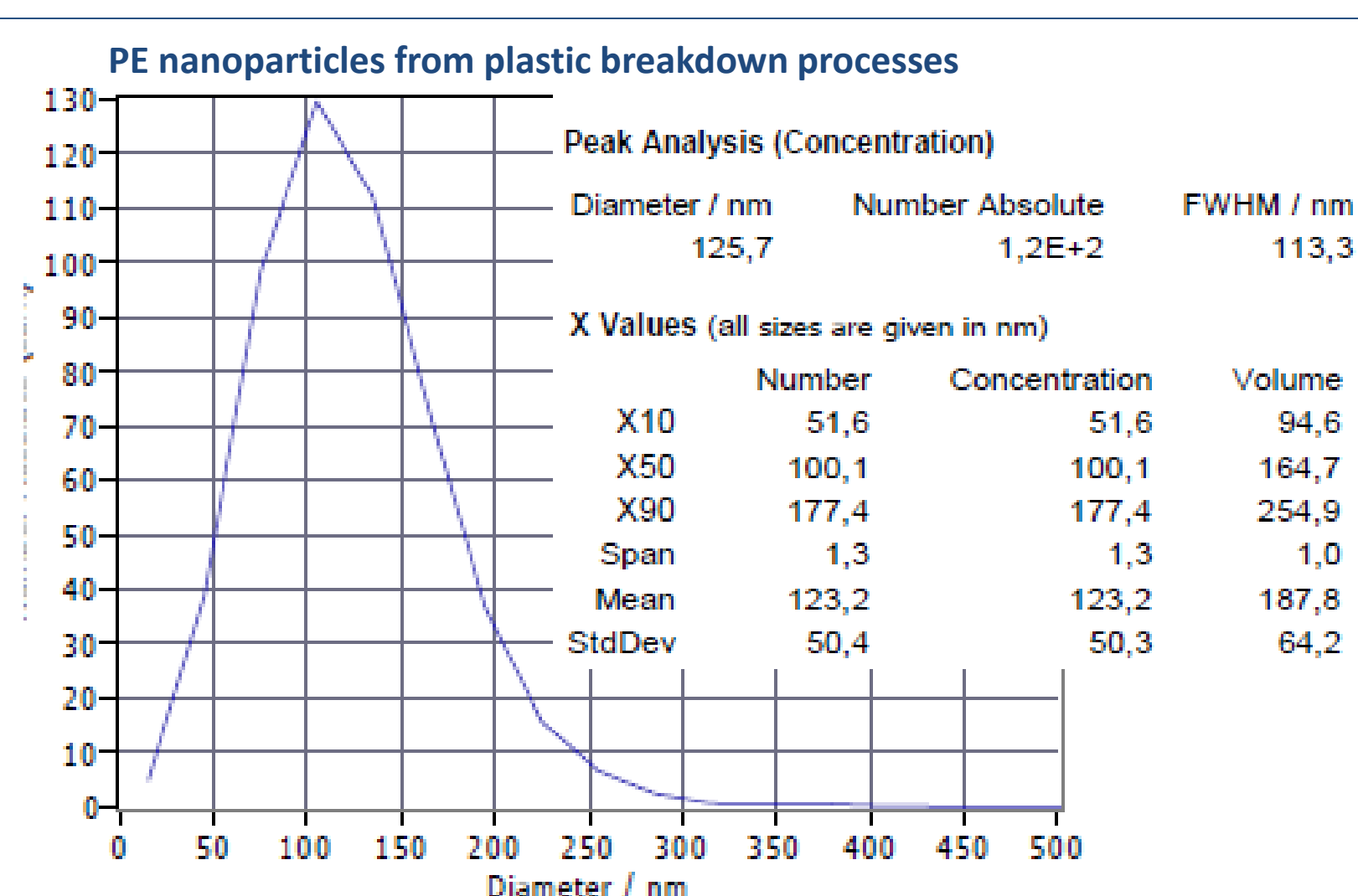
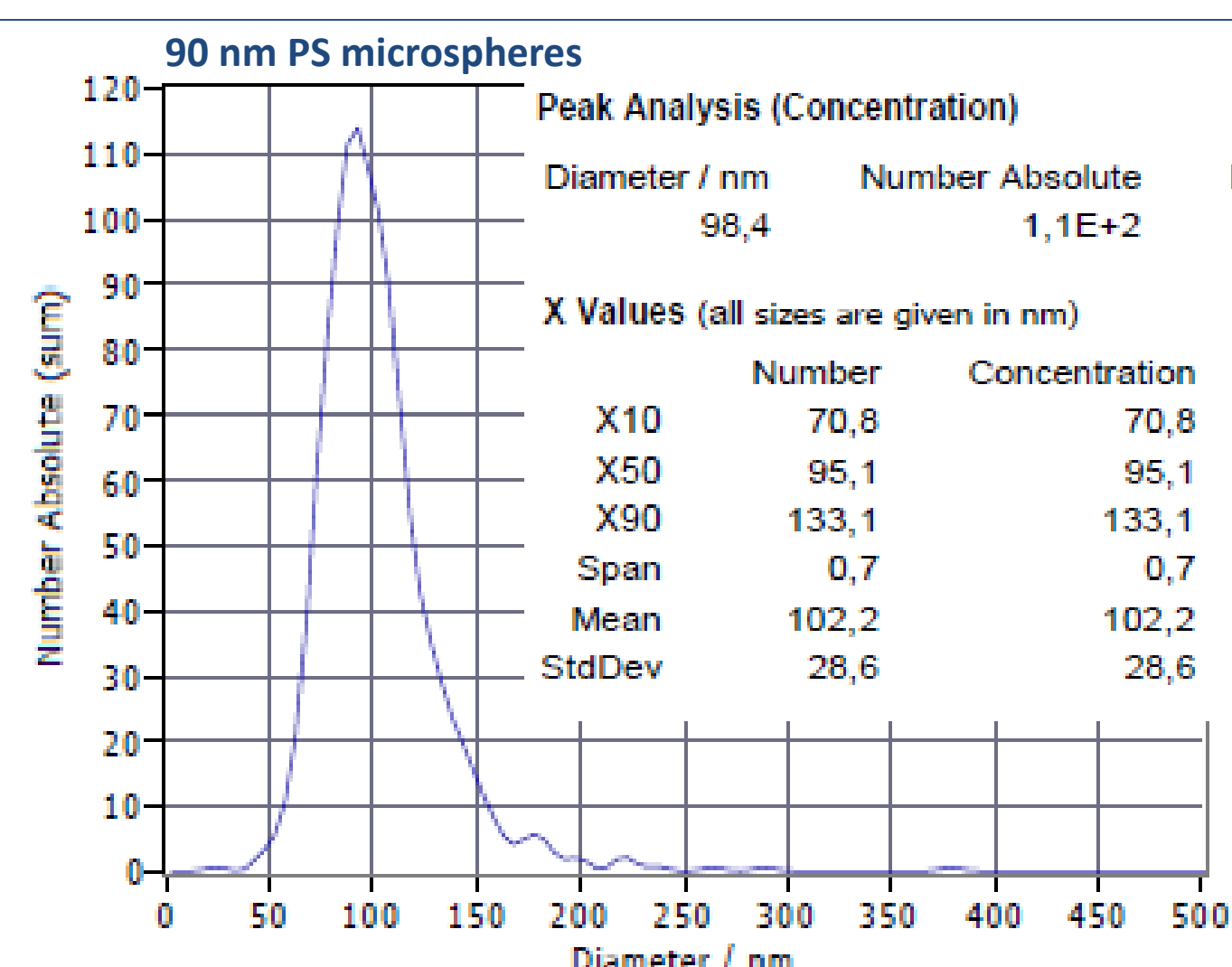


Figure 3. Size distribution of 90 nm PS microspheres and polyethylene nanoparticles using nanoparticle tracking analysis.

- The nanoparticle tracking analysis (ZetaView®, Particle Metrix GmbH, DE) is not only suitable to determine the size of individual plastic particles, but the presented method can also be used to directly determine particle concentrations. In the current setup PS and PE nanoparticles size and number concentrations were determined (Figure 3) and compared well with dynamic light scattering results.
- As such, the detection and quantification of nanoplastics in water samples is feasible.

Gel permeation chromatography coupled with UV detector for NPs size

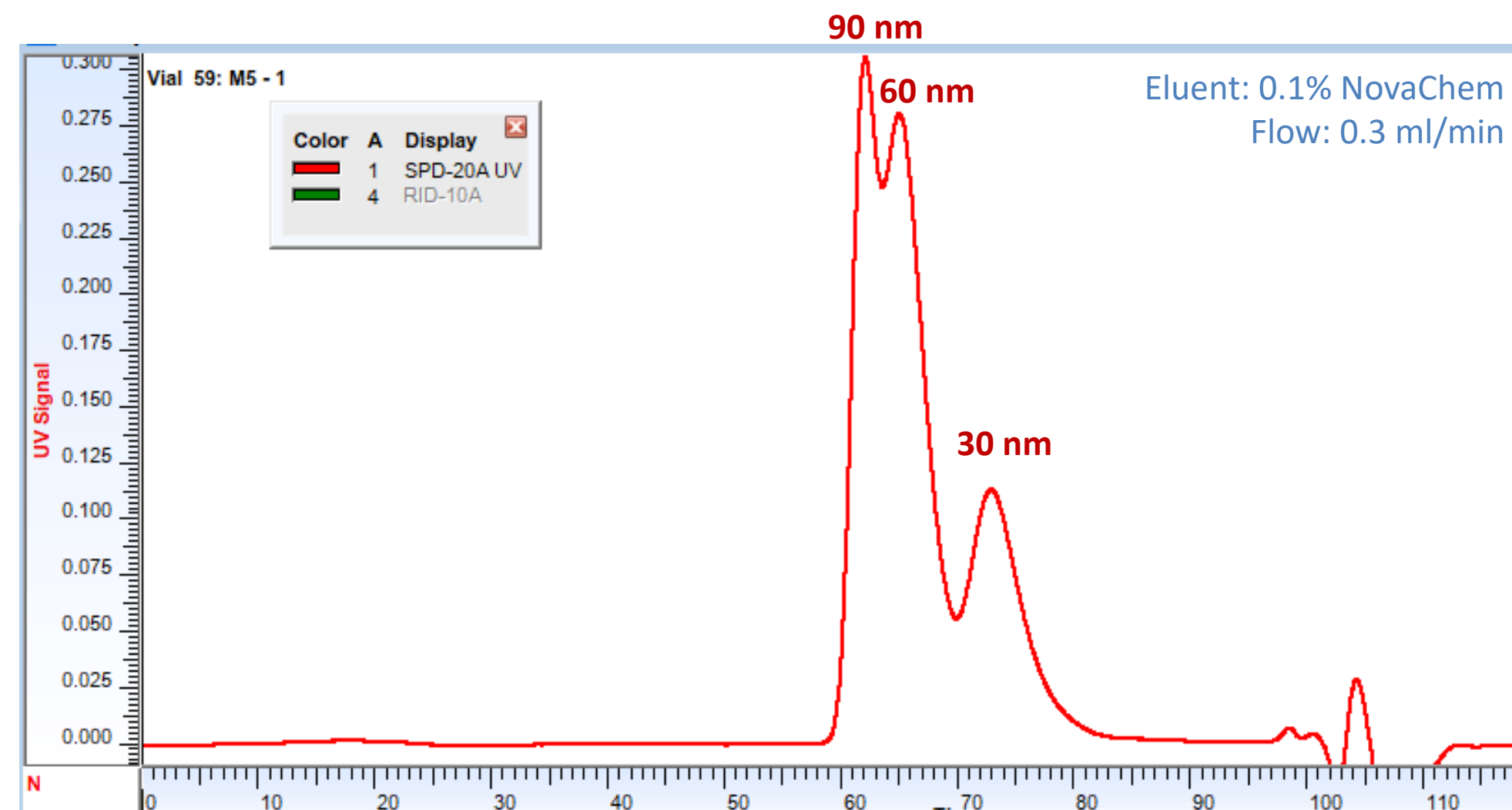


Figure 4. UV spectra of GPC elution fractions of 30, 60 and 90 nm PS particles.

- Use of a 2x 1000-Å-pore-size ultrahydrogel column and an aqueous mobile phase of 0.1% (v/v) NovaChem resulted in the satisfactory separation of 30, 60 and 90 nm PS particles.
- Using UV and refractive index detection this method provides simple and straightforward separation of the smallest fraction of NPs (< 100 nm).

CONCLUSIONS

- Further optimization is still required to fully exploit the potential of ICP-MS operated in single-event mode for the characterization of not only low- μm range MPs, but also NPs (< 1 μm).
- As the currently available monitoring techniques are typically limited to particles larger than 10 μm , nanoparticle tracking analysis and gel permeation chromatography present a first step toward the detection and size characterization of the NPs in water samples.

REFERENCES

Velimirovic, M. et al., Anal. Bioanal. Chem. 413, 7–15 (2021); Bolea Fernandez, E. et al., J. Anal. At. Spectrom. 35, 455–460 (2020).