vitesse

Vitesse Note **NT10**

Very low mass isotope data collection with the Nu Vitesse, measurement of microplastic particles

Keywords:
Microplastics
Single particle multielement ICP-MS
Very low mass performance

Introduction

Time-of-flight ICP-MS is recognised as a simultaneous elemental analysis technique but the intrinsic qualities of a time-of-flight mass spectrum mean that sensitivity for very light masses is much lower than for heavier masses. The use of a multipole device in the reaction cell to remove interferences also introduces a mass bias in the instrument response which is partially overcome in the Nu Vitesse by the use of an axial field gradient across the reaction cell. The pressurising of the cell and required RF field needed to obtain broad band transmission across the mass range generally means that sensitivities below ^{23}Na are sometimes too low to be useful in full mass range spectra. This note will show how very low mass data can be obtained with specific tuning conditions and how this benefits certain applications such as the analysis of microplastics.

Optimising for microplastics

The first consideration for microplastics is that the standard spray-chambers used in all ICP-MS are designed to remove large droplets in the nebulised aerosol and consequently, losses in the sample introduction can be high and may interfere with accurate size distributions due to size dependencies. For microplastics, it was found more beneficial to use a sample introduction system developed for single cell ICP-MS where a single pass chamber and specialised nebuliser are used as shown in figure 1.

Ion optics of the Vitesse were then optimised to improve the transmission of very low masses by reducing the gas flows into the reaction cell so only the minimal amount needed to remove the argon interferences was used and that the scattering of very low mass ions was drastically reduced. By also then reducing the RF amplitude of the voltage applied to the reaction cell multipole, the transmission of very low mass ions was increased.

Figure 1: Glass expansion single cell sample introduction system attached to the Vitesse.

The reaction cell gas flows and axial field were then tuned to obtain narrow carbon mass peak widths from individual microplastics for the subsequent single particle work flow. Figure 2 shows the ability of Vitesse to distinguish 1 µm polystyrene particles from the ^{12}C background when using 80 µs dwell times.

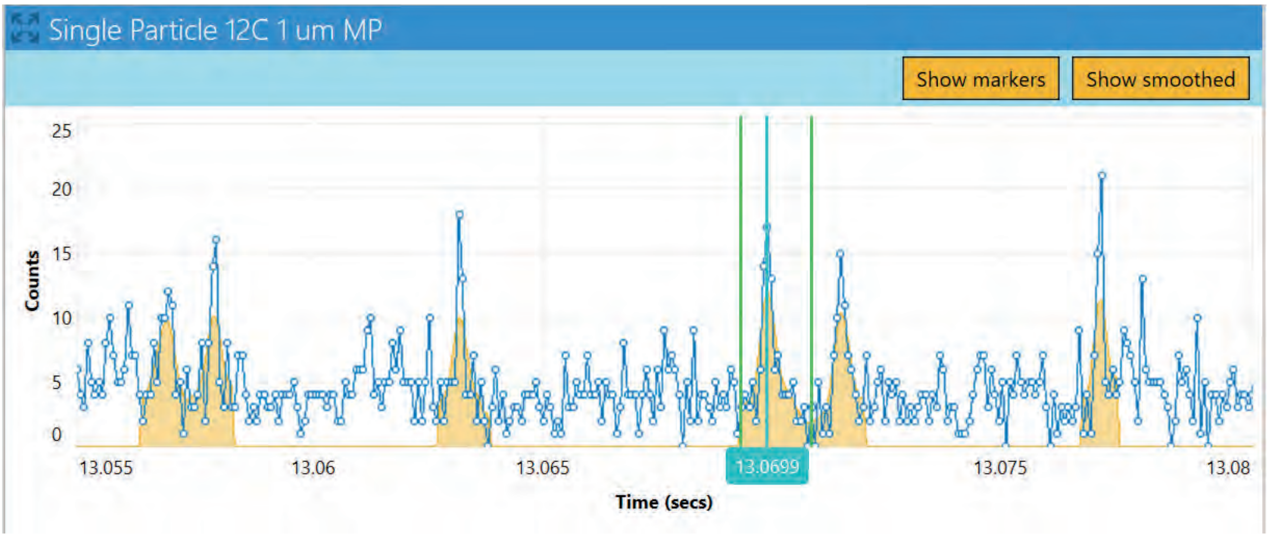


Figure 2: Chromatogram for ^{12}C showing the traces for individual particles collected with 80µs spectra. The data smoothing and peak search algorithms allows 1 µm microplastic signals to be differentiated from the baseline noise.

Characterising microplastic materials

All nano and micro materials have a more or less polydisperse size distribution and may be constituted of various isotopes across the mass range. Characterising particles for calibration and reporting requires that the detection of particles is sufficient that the distribution to the lower and higher particle mass sides can be fully determined. The Vitesse is able to collect enough particle data in a few seconds to fully characterise distributions for calibrations. For a 1 µm polystyrene standard in deionised water over 3000 particles were determined in less than 30 seconds leading to the signal distribution shown in figure 3.

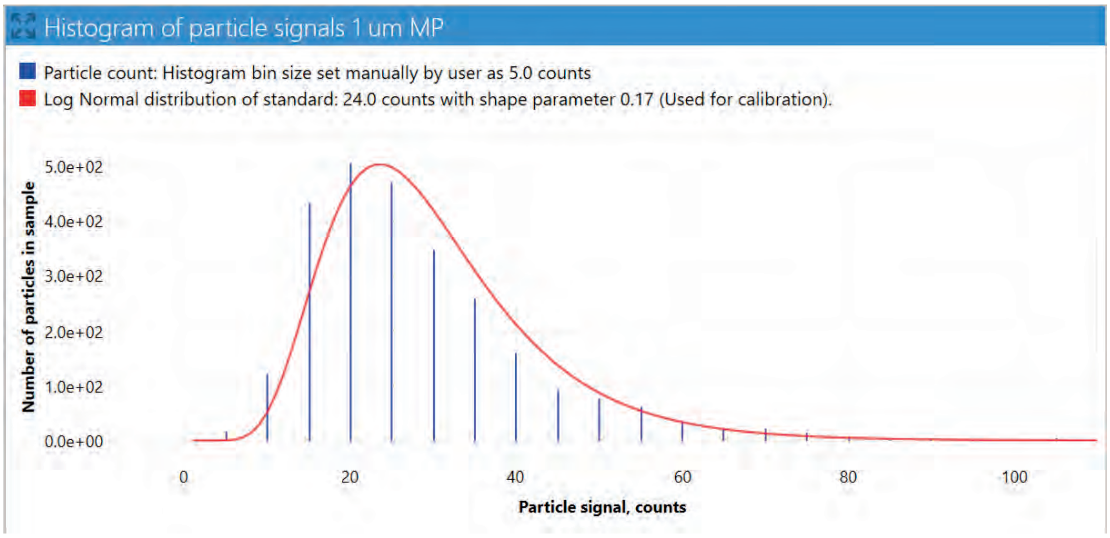
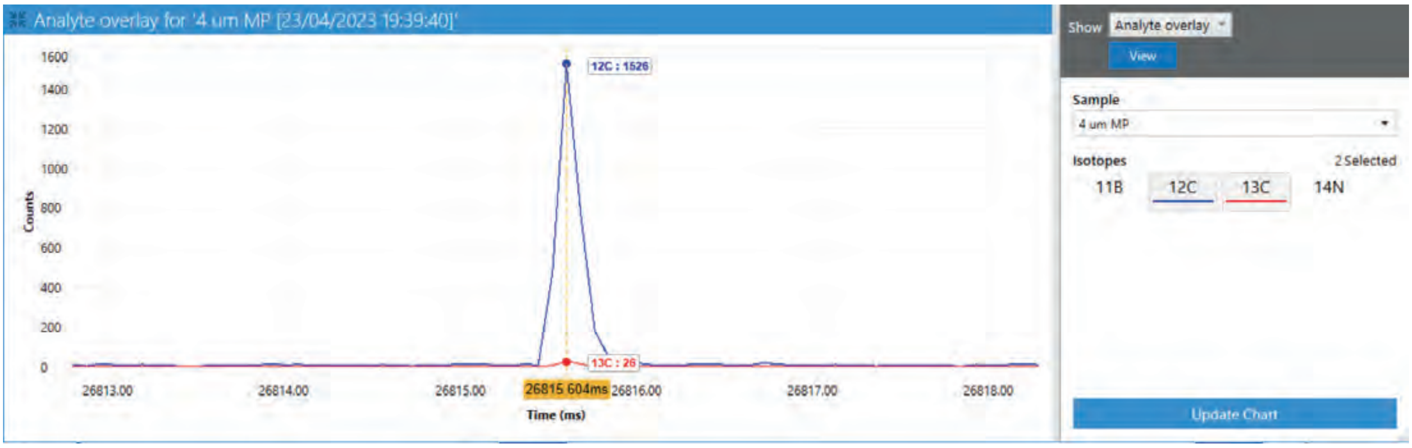


Figure 3: Distribution of integrated particle counts for a 1 µm polystyrene standard in deionised water.

The mass of a particle and therefore the signal generated at the detector increases with the cube of size so in order to fully characterise larger particles, it is critical to consider the effect of very high signals on the distribution profiles. A 4 µm polystyrene standard in deionised water will have nominally 64 times more mass meaning that the linear range of the detector needs to be sufficient to measure over 10M cps for very short transient periods as shown in figure 4. For larger particles, it was even possible to simultaneously measure ^{13}C in approximate relative abundance to the ^{12}C (~1.1%).



Show: Analyte overlay

Sample: 4 um MP

Isotopes: 2 Selected

Figure 4: Showing the measurement range of the Vitesse detector. For 80 µs per spectrum, a signal of 1500 counts at the peak maximum of some of the 4 µm particle event is equivalent to over 18M cps. The ^{13}C signal is measured in the same particle at near the expected abundance.

The range of the Vitesse allows a clear distribution to be determined for larger microplastics (figure 5) with the use of the attenuators making it possible to extend the measurement even further in a second run of the particle suspension should it be needed. Though it should be noted that the use of a microconcentric nebuliser for single cell sample introduction may cause blockages at significant larger particles sizes.

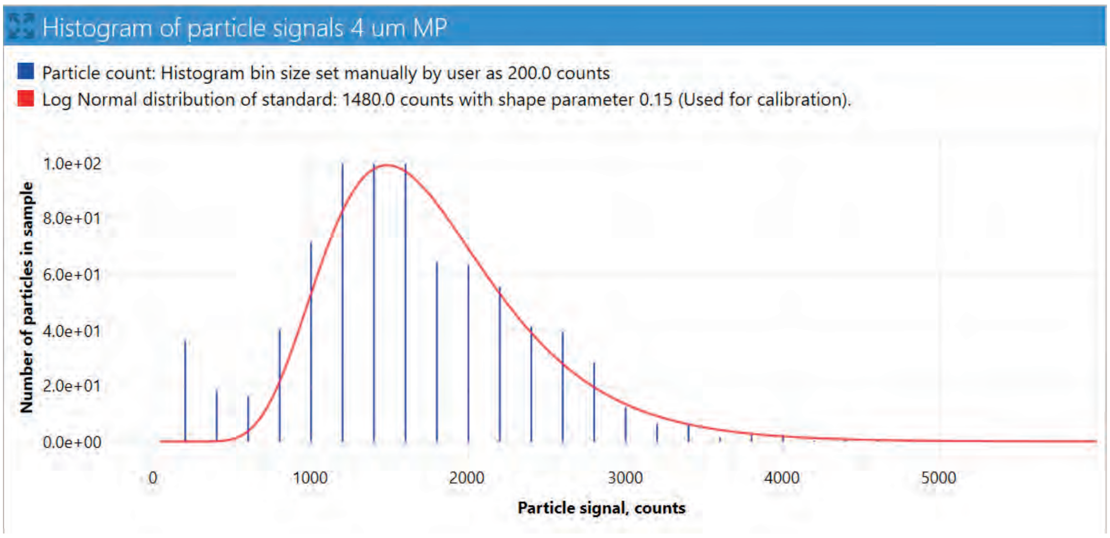


Figure 5: Distribution of integrated particle counts for a 4µm polystyrene standard in deionised water.

Once distributions have been established for standards, it is possible to see the linear range of measurement from Vitesse when viewing the calibration forced through the origin, figure 6.

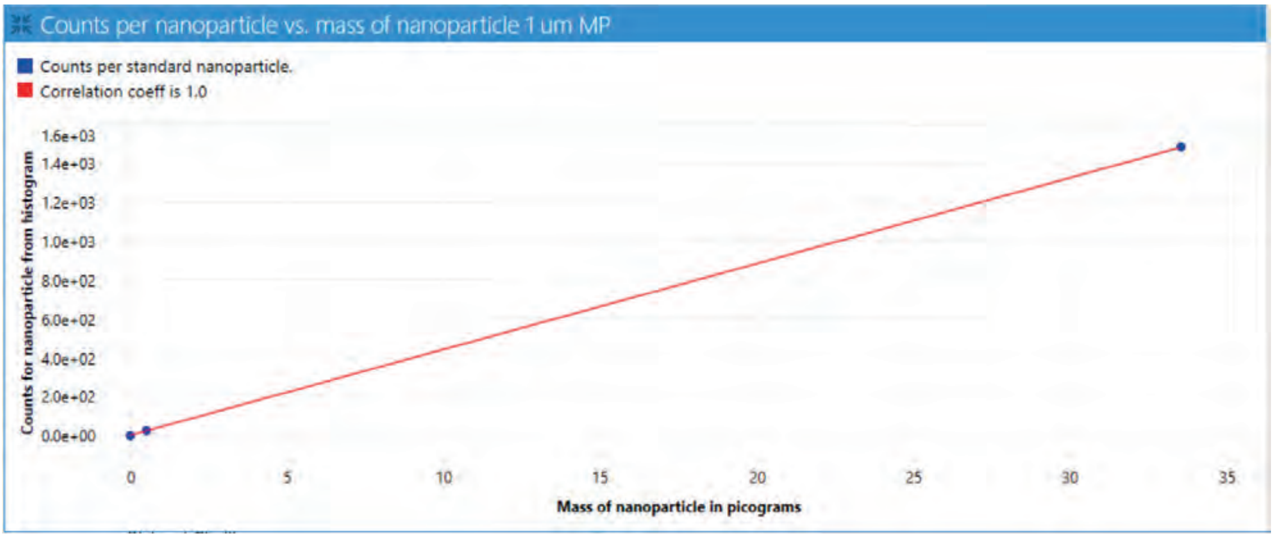


Figure 6: Calibration of particle signal with the mass of the particle covering the dynamic range of the Vitesse from the noise floor of the background ionic signal of the gas blank to the upper counting range of the detector.

Conclusion

It has been possible to collect data for carbon in microplastics and easily distinguish the signals for sub-micron particles from the ionic ^{12}C background. The detection system of the Vitesse has shown a large dynamic range allowing full characterisation of signals for both small and large particles for a linear calibration. The simultaneous measurement of ^{13}C in larger microplastic particles has shown the advantage of the Vitesse of being able to confirm that for the absence of relevant interferences such as Mg^{2+} ions.

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