

## Development of a single particle ICP-MS method using a high efficiency sample introduction system (APEX™) for the characterisation of titanium dioxide nanoparticles in foodstuffs

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This work reports the development of a novel method based on single particle inductively coupled plasma-triple quadrupole mass spectrometry (ICP-QQQMS or ICP-MS/MS) using a high efficiency sample introduction system (HESIS) such as APEX™ for the characterisation of TiO<sub>2</sub> (nano)particles (NP<sub>s</sub>) in food samples. The use of the mass shift detection mode (O<sub>2</sub>+H<sub>2</sub> as a mixture reaction gas) avoids the isobaric interferences of <sup>48</sup>Ca isotope hence allowing accurate characterisation of TiO<sub>2</sub> NPs in complex matrices such the foodstuffs.

APEX™ Ω proved to be an excellent approach to avoid matrix effects from food samples and provided significantly low limits of detection in size (LOD<sub>size</sub>) compared to the conventional introduction system. In order to avoid the saturation of the detector when detecting large TiO<sub>2</sub> particles, the analyses were carried out using the forced analogue detection mode and excellent characterisation of TiO<sub>2</sub> NPs in terms of size and concentration was achieved, with LOD<sub>size</sub> of 12 nm.

The new method was applied to the analysis of a selection of foodstuffs, such as candies, sauce, cheese, cakes, etc. purchased in Spain before the ban of the use of TiO<sub>2</sub> based additive in the European Union. In all cases, NP<sub>s</sub> recoveries were assessed based on the comparison with the total Ti level that was also measured by ICP-MS in the same samples. This confirms the feasibility of this novel approach using an APEX™ Ω for the accurate characterisation of TiO<sub>2</sub> NPs in complex matrices such as the foodstuffs and hence its application to real-life studies.

**Keywords:** TiO<sub>2</sub> nanoparticles, total Ti, single particle ICP-MS/MS, high efficiency sample introduction, foodstuffs system.

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