The Analysis of Titanium Nanoparticles in artificial and natural sweeteners by the Agilent 8900 QQQ-ICPMS

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Introduction

The health effects of titanium nanoparticles in the ecosystem ad thus biological system are being more widely documented with the nanoparticles ability to cross the human cell membrane. Titanium nanoparticles are used in a wide range of applications such as paints, toothpastes, sunscreens, as well as food products such as "natural" and artificial sweeteners.

There are various techniques to analyze titanium nanoparticles from Field Flow Fractionation, Xray Diffraction, but the quickest and most sensitive technique is via SP-ICP-MS. The Agilent 8900 ICP-MS with MSMS technology provides superior sensitivity and banground that allows for accurate and low level detection of these nanoparticles.

FIG 1. The Agilent 8800 ICP-QQQ-MS in Wilmington, DE

The USFDA allows food grade titanium dioxide without notification on nutrition labels up to 1%. The issue comes with the term NANO, and its size <100nm. The study of nanoparticles and their long term health effects are still in its infancy, however it is known that nanoparticles can cause respiratory issues and possible cell passage.

In 2015 Dunkin Donuts started the removal of titanium nanoparticles from its sweeteners on its donuts, namely the powdered sugar containing ones, despite the concrete effects of human toxicology knowledge.

Titanium nanoparticles are not only found in sweeteners as this poster focuses on, but they are also found in sunscreen, mayonnaise, gum, and soy milk. With its purposes varying from providing color, texture, or its properties to not melt, or dissolve in water.

This is why it is important to able to analyze titanium nanoparticles to the lowest particle size possible. The 8900 accomplishes this by providing the maximum sensitivity and unrivaled interference removal that is needed for the analysis of titanium. Titanium suffers from interferences from S, Ca, P, and Si. The 8900 uses MSMS technology to do selective reactive chemistry to remove both the isotopic and isobaric interferences that Titanium suffers.

Experimental

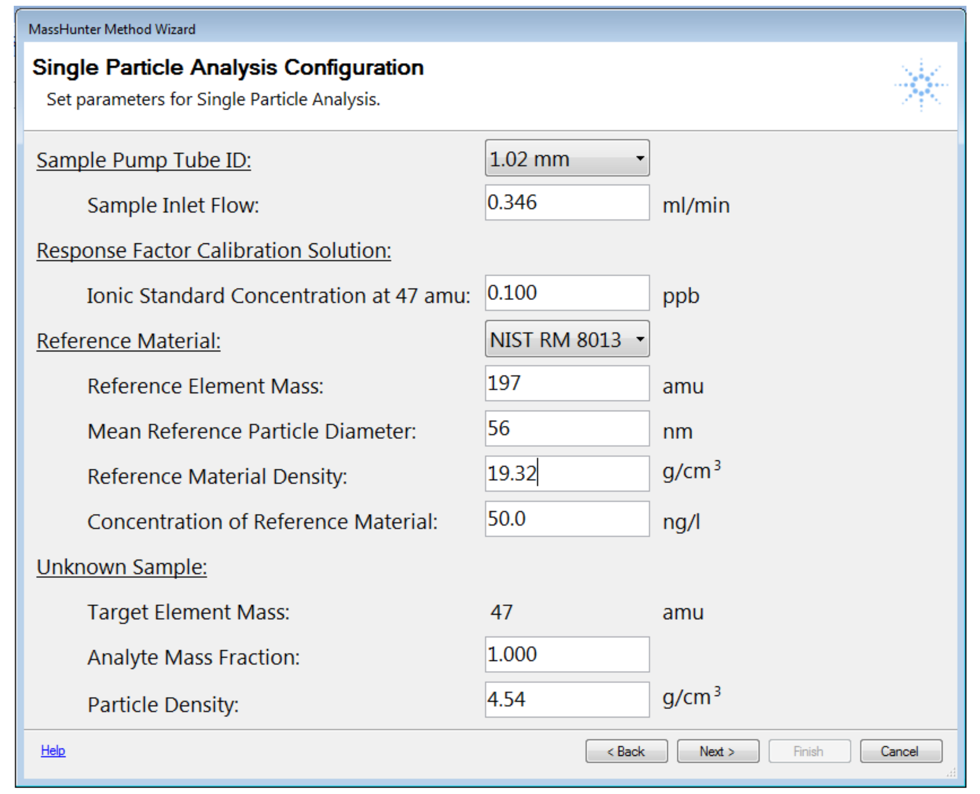
The Agilent 8900 QQQ-ICPMS with its ability to do MS/MS technology was used to analyze Titanium nanoparticles in several commercially available artificial and natural sweeteners. The 8900 was equipped with a standard Agilent quartz sample introduction system equipped with a quartz 1.0 mm injector. The 8900 also used an Agilent quartz concentric nebulizer.

Instrument Conditions	
Scan Mode	MS/MS
RF Power (W)	1500
Sample Depth (mm)	8.0
Spray Chamber (Co)	2.0
Carrier Gas Flow (mL/min)	0.65
KED (V)	-5
O2 Mode Flow Percentage	30%

FIG 2. 8900 Instrumental Conditions

The analysis was done by diluting the samples 0.05g to 50 mL (1000:1) then analyzed by size against a NIST 8013 (60nm) nanoparticle standard diluted to about 50 ppt.

An ionic standard of titanium was made to determine the ionic titanium concentration of 100 ppt. This was all set up in the MassHunter Software using the "Start Up" Autotune conditions and the instrument batch configured for nanoparticle analysis using the Agilent MassHunter software's Method Wizard feature for nanoparticles



MassHunter Method Wizard

Single Particle Analysis Configuration

Set parameters for Single Particle Analysis.

Sample Pump Tube ID:	1.02 mm	
Sample Inlet Flow:	0.346	ml/min
Response Factor Calibration Solution:		
Ionic Standard Concentration at 47 amu:	0.100	ppb
Reference Material:	NIST RM 8013	
Reference Element Mass:	197	amu
Mean Reference Particle Diameter:	56	nm
Reference Material Density:	19.32	g/cm^3
Concentration of Reference Material:	50.0	ng/l
Unknown Sample:		
Target Element Mass:	47	amu
Analyte Mass Fraction:	1.000	
Particle Density:	4.54	g/cm^3

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FIG 3. Mass Hunter Conditions using Method Wizard

Results and Discussion

Titanium Nanoparticles of Various Sweeteners

The figures below displays some of the contrast of titanium nanoparticles in some sweeteners

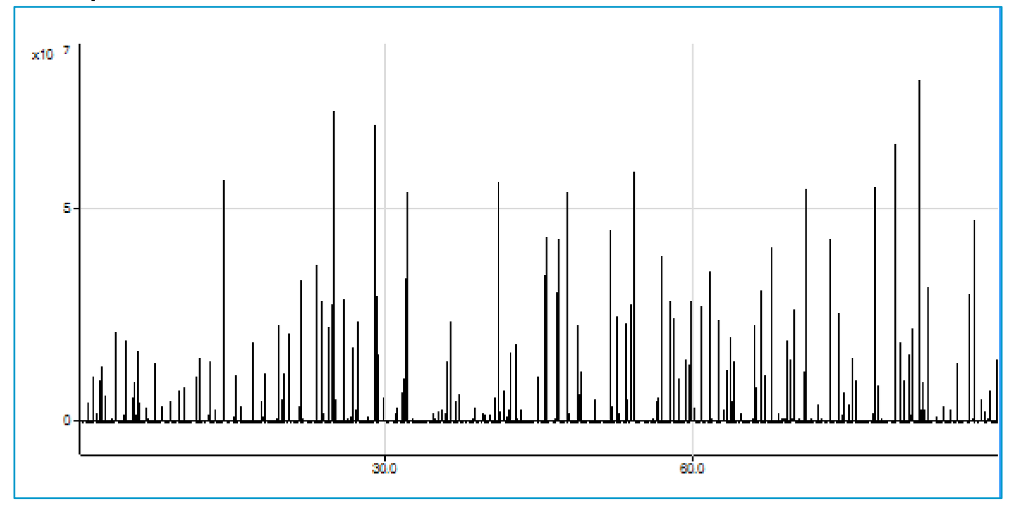

FIG 4. Splenda

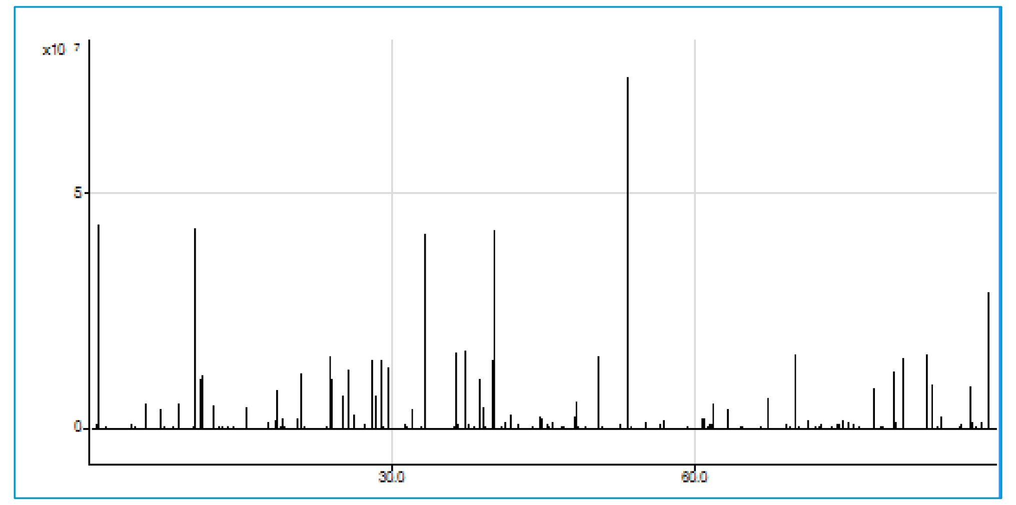

FIG 5. Cane Sugar Non Organic

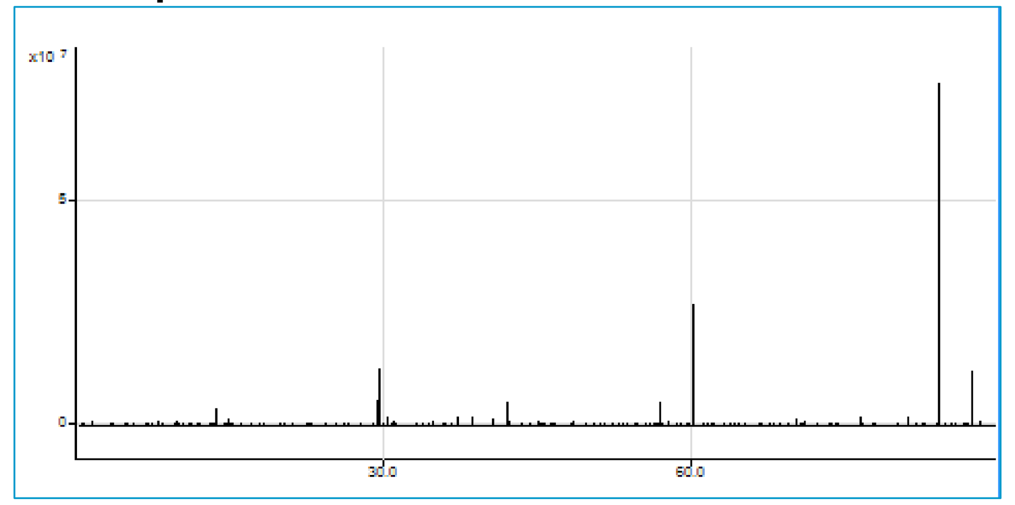

FIG 6 Sweet & Low

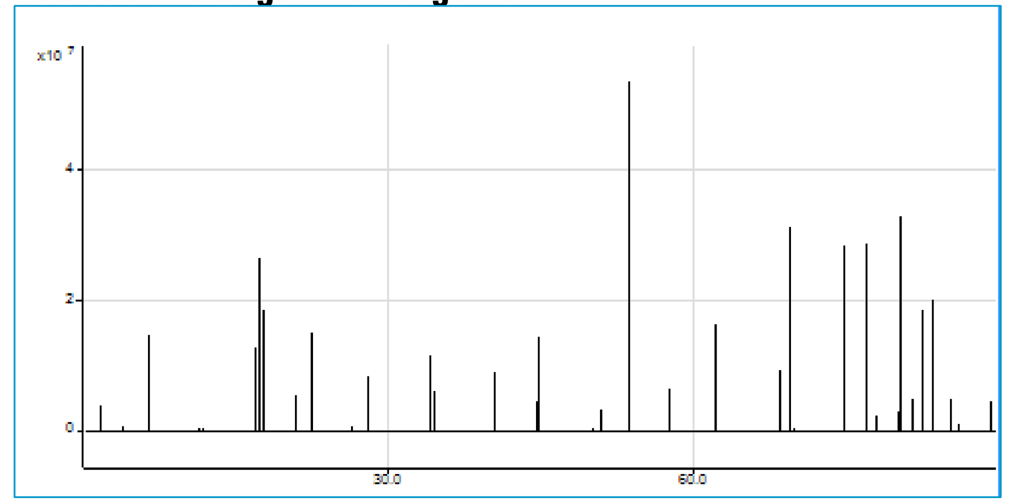

FIG 7. Organic Raw

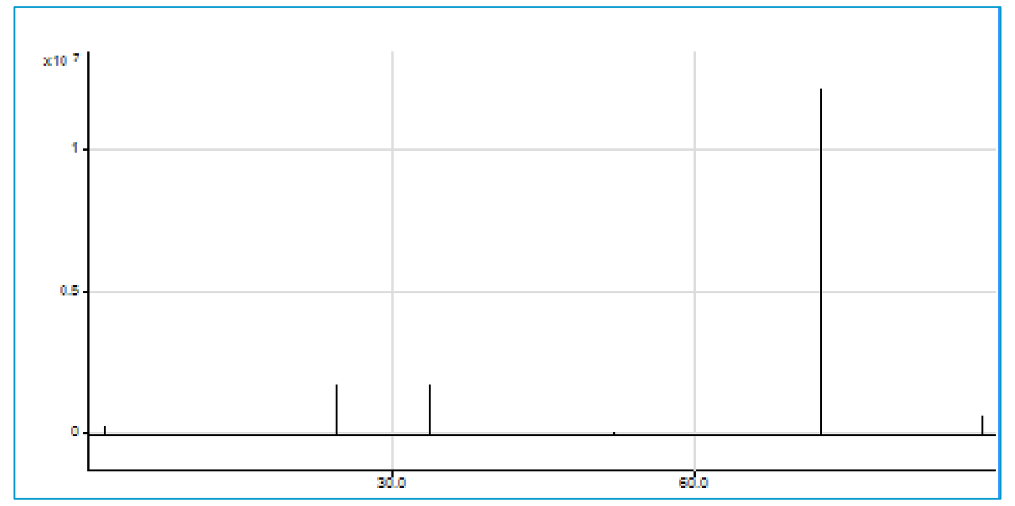

FIG 8. Truvia

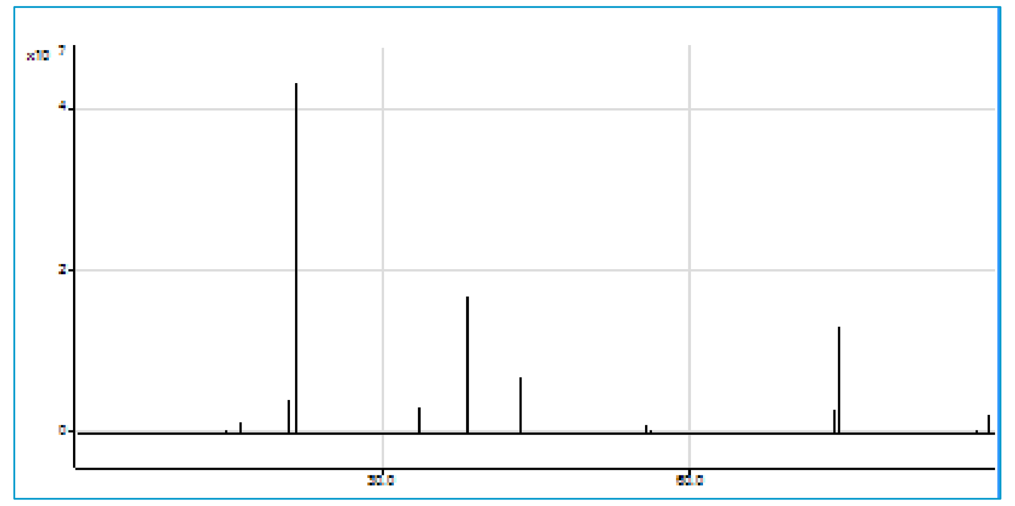

FIG 9 Stevia

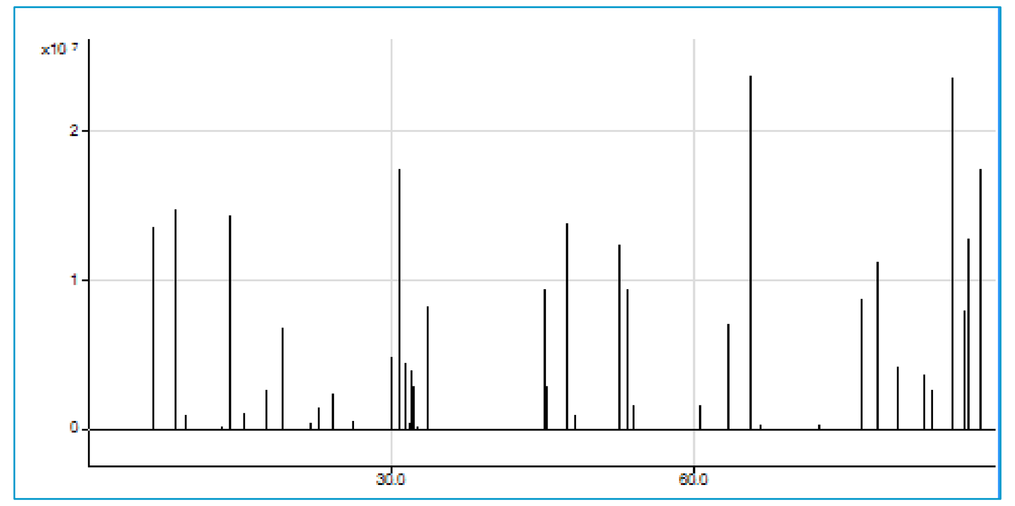

FIG 10. Equal

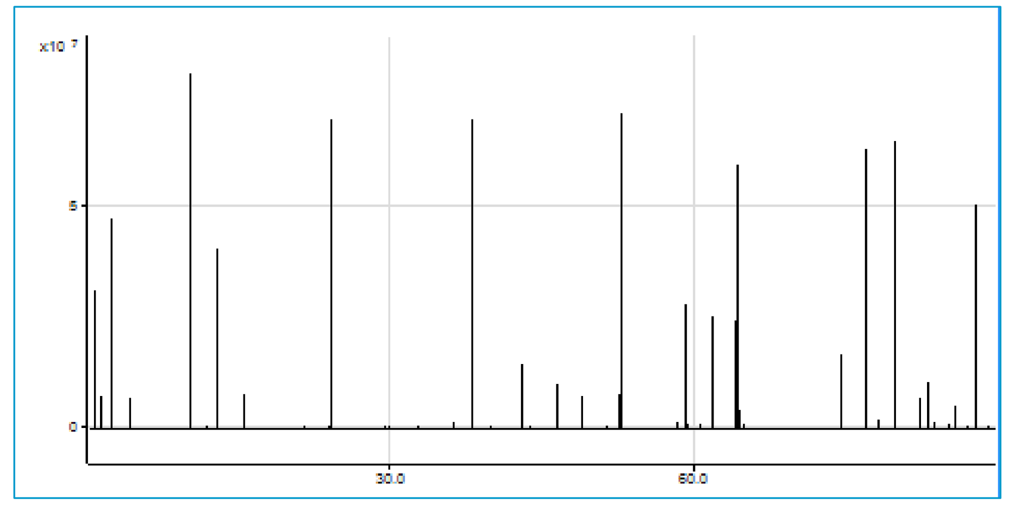

FIG 11. Granulated Sugar

Results and Discussion

Nanoparticle Size Distribution

The table below displays the size distribution of the NIST 8013 Gold (Au) standard (60nm)

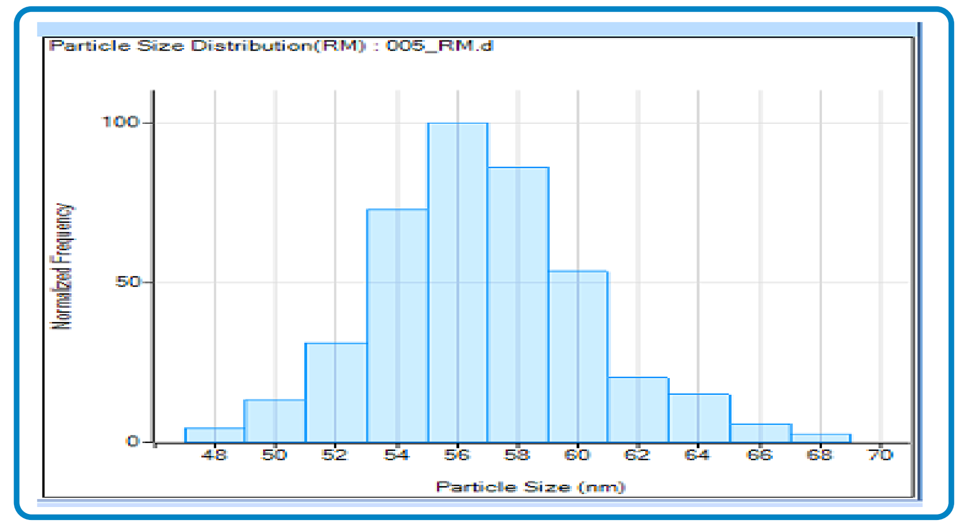

FIG 12. NIST 8013 60 nm Au Standard

The table below displays the numbers of titanium partcles seen in each sample analyzed over a 90 second time frame at 0.0001 sec scan speed. This is a an indicator on the number of particles in each solution.

Sample	# of Particles	BED (nm)	Median Size (nm)
Equal	1396	6.7	12.6
Splenda	4275	7.5	14.1
Sweet Low	333	18.7	33.3
Sugar	24465	8.5	16.4
Organic Raw Cane Sugar	1141	12.4	20.4
Confectionary 10x Sugar	1556	7.8	14.2
Truvia	338	6.0	12.6
Blue Agave	7376	8.7	19.9
Stevia	603	6.2	12.6
Cane Sugar Non Organic	45171	9.4	25.2
Splenda Naturals	144	5.7	14.4
Splenda Sugar Mix	12735	9.4	13.9
Coconut Palm	1542	31.7	66.3

FIG 13. DLs and BECs in ppt for B isotopes in 100% IPA

The data shows some interesting results, Sugar, Cane Sugar and the Splenda/Sugar Mix were the highest in TiNPs, With the Sweet Low, Truvia amd Splenda Naturals all being the lowest.

The fact that the Stevia leaf extracts (Stevia, Truvia, Splenda Naturals) were low in TiNP's is not surprising however the fact that Sugar (Highland Estates) and non Organic Cane sugar were the highest is surprising.

The BED (background equivalent diameter) is an indicator of the how low a size particle could be analyzed under the conditions analyzed. In this analysis almost all BED's were in the 6-10 nm range, this lends to the excellent sensitivity and background of the 8900. The samples that were higher than this probably could have used further dilution

The median size of almost all samples were between 12 and 20 nm for the titanium nanoparticles. Some examples of the distributions of these particles sizes are shown below in figures 14

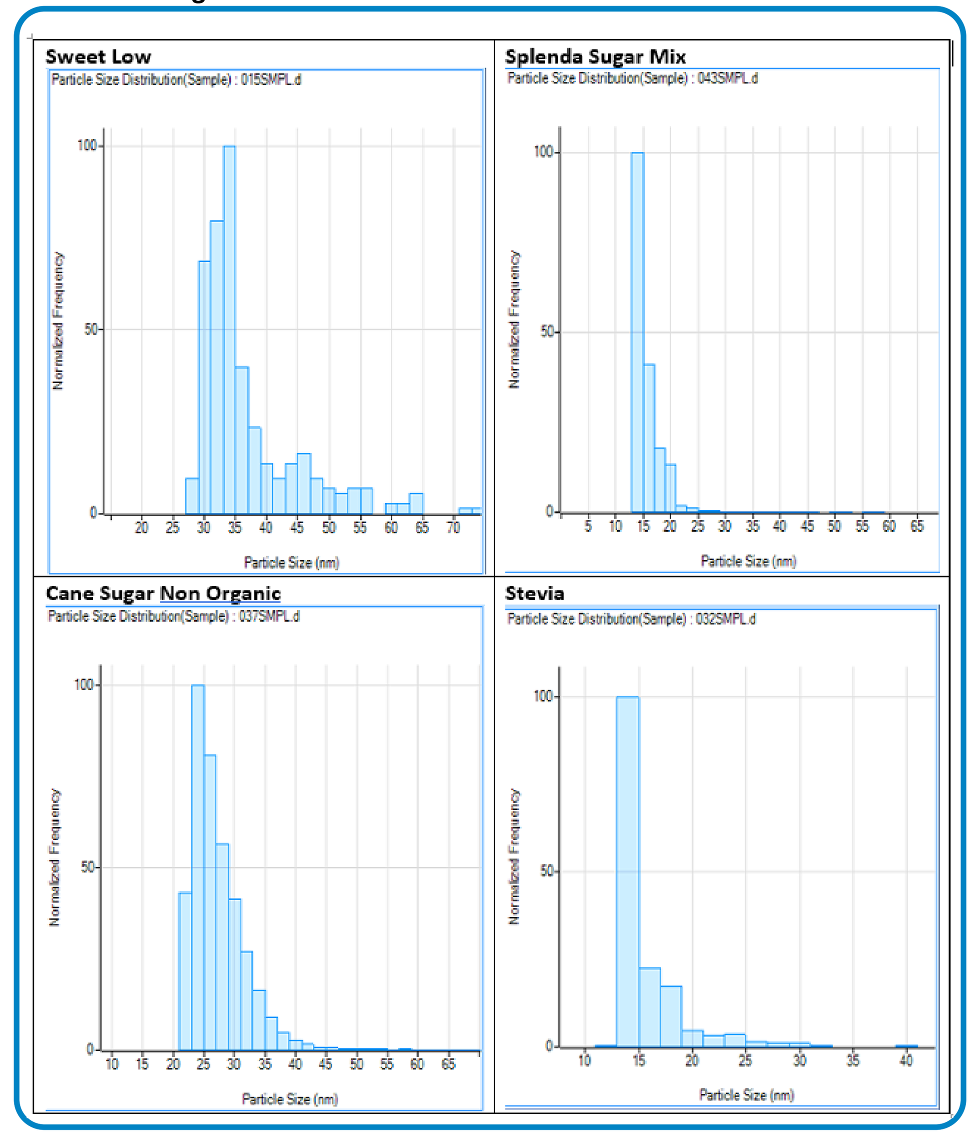

FIG 14. DLs and BECs in ppt for B isotopes in 100% IPA

Conclusions

The Agilent 8900 QQQ ICP-MS, operated in MS/MS mode, eliminates problematic spectral interferences that cannot be removed with typical single quad collision/reaction cell ICPMS's without using less abundant masses or other type of sample preparation. of organic solvents, thus getting better detection limits. This allows for the difficult element to be analyzed at very low BED's for what may be a growing field as more information on health effects become available over time