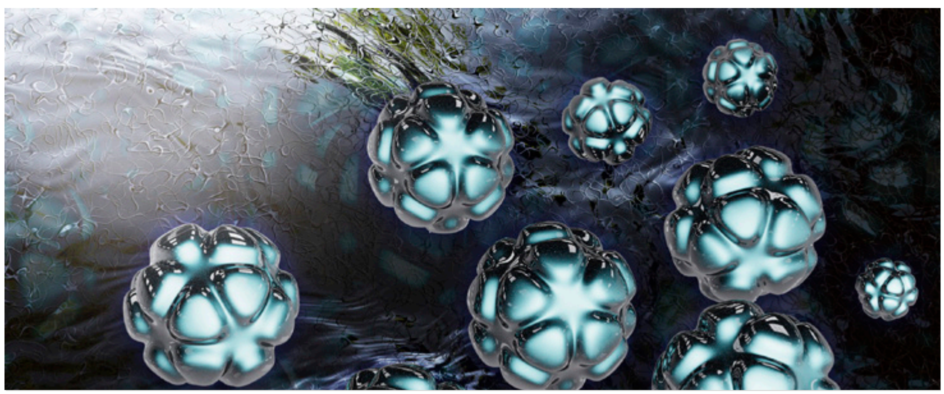
Application Overview

Analysis of 10 nm Gold Nanoparticles using the Agilent 7800 ICP-MS



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Introduction

High sensitivity and low background noise are critical for the detection of small nanoparticles by ICP-MS. The signal generated by the ionization of a nanoparticle decreases as the cube root of the diameter. This means detection of very small particles, such as 10 nm Au particles (NIST 8011), requires ICP-MS instrumentation with significantly higher sensitivity than is required for the more typical NIST reference materials such as NIST 8012 (30 nm) and 8013 (60 nm).

In this study, we demonstrate that the Agilent 7800 ICP-MS can easily achieve the signal-to-noise required to measure 10 nm Au nanoparticles. This is achieved without the need for complex reactive cell gases or customized tuning conditions. The standard operating conditions used could easily be applied to nanoparticles composed of other elements such as Ag.

Experimental

An Agilent 7800 ICP-MS was used for all measurements. The instrument was fitted with the standard MicroMist concentric nebulizer and double pass quartz spray chamber, a 1.5 mm ID quartz torch (optional on the 7800) was used.

Fast TRA (FTRA) capability—optional on the 7800—permitted the use of 100 µs integration times with no settling time. This allowed clear definition of individual nanoparticle peak shapes.

The instrument was operated in no-gas mode (Octopole Reaction System (ORS) cell unpressurized) as Au has no common polyatomic interferences and no-gas mode provides the simplest, most sensitive ORS conditions for routine samples that do not require polyatomic interference removal.

The instrument was tuned to maximize the sensitivity for Au by simply adjusting the plasma and lens parameters for best sensitivity at *m/z* 197 with ~2% oxides. This yielded a signal for the 0.5 ppb Au ionic standard of 116,319 cps (232,638 cps/ppb). Blank counts were 10.12 cps, resulting in a background equivalent concentration of 43.5 ppq.

The ICP-MS MassHunter Single Nanoparticle Application Module was used to automatically create the method including all acquisition and data analysis parameters as well as the recommended sample list.

The sequence of samples consisted of:

1. A blank and 0.5 ppb ionic calibration standard, used to determine the instrument response factor for Au.
2. NIST 8012 (30 nm Au), used by ICP-MS MassHunter to automatically calculate the nebulization efficiency.
3. NIST 8011 (10 nm Au) reference particle size standard.
4. Mixture of NIST 8011 and 8012, analyzed as unknown samples.

The ICP-MS MassHunter Single Nanoparticle Application Module automatically calculated the relevant results for the standards and samples. The results were displayed in the familiar "batch-at-a-glance" table and detailed graphical format for each sample.

Results

Figure 1 shows that the Au signal in NIST 8011 was clearly detected and that the mean size and most frequent size were both 10 nm. This is in good agreement with the certified value.

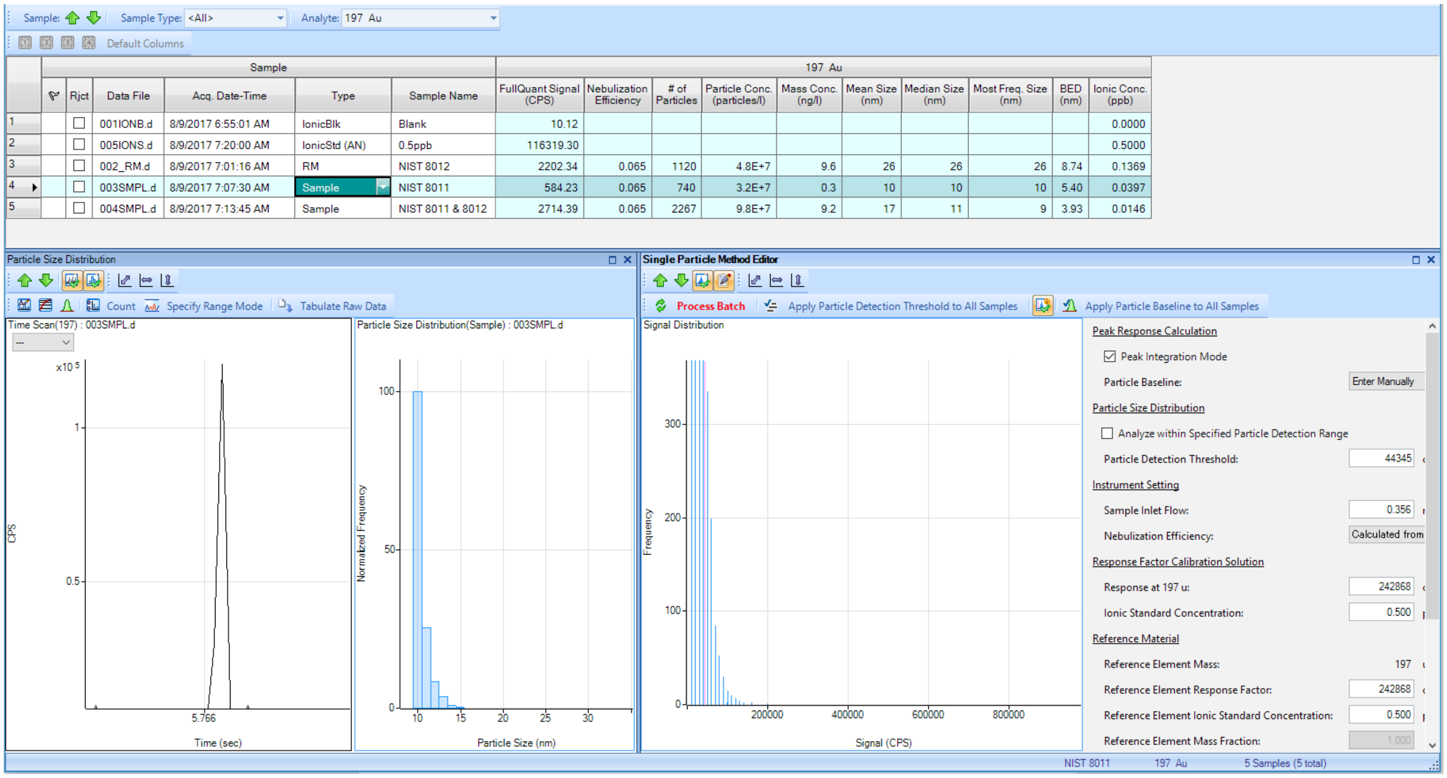
The background equivalent diameter (BED), which is the theoretical lowest detectable particle size in the particular sample, was ~ 5 nm for NIST 8011. This indicates that the 10 nm determination is well above the background.

Figure 2 depicts the graphical results for the NIST 8011 sample. It shows a "peak" in the TRA chart for a single particle (left) as well as the signal distribution (right) and particle size distribution (center), clearly centered around 10 nm.

The particle detection threshold was automatically calculated from the baseline noise using a sophisticated smoothing algorithm. This enabled separation of the small particle signals from the baseline noise. The value is indicated by the vertical red bar in the signal distribution plot, which can be manually adjusted if necessary.

Sample						197 Au									
	Rjct	Data File	Acq. Date-Time	Type	Sample Name	FullQuant Signal (CPS)	Nebulization Efficiency	# of Particles	Particle Conc. (particles/l)	Mass Conc. (ng/l)	Mean Size (nm)	Median Size (nm)	Most Freq. Size (nm)	BED (nm)	Ionic Conc. (ppb)
	☐	001IONB.d	8/9/2017 6:55:01 AM	IonicBlk	Blank	10.12									0.0000
	☐	005IONS.d	8/9/2017 7:20:00 AM	IonicStd (AN)	0.5ppb	116319.30									0.5000
	☐	002_RM.d	8/9/2017 7:01:16 AM	RM	NIST 8012	2202.34	0.065	1120	4.8E+7	9.6	26	26	26	8.74	0.1369
	☐	003SMPL.d	8/9/2017 7:07:30 AM	Sample	NIST 8011	584.23	0.065	740	3.2E+7	0.3	10	10	10	5.40	0.0397

Figure 1. Tabular results – ionic calibration standards, NIST 8012 reference material and NIST 8011 10 nm Au, analyzed as an unknown sample.



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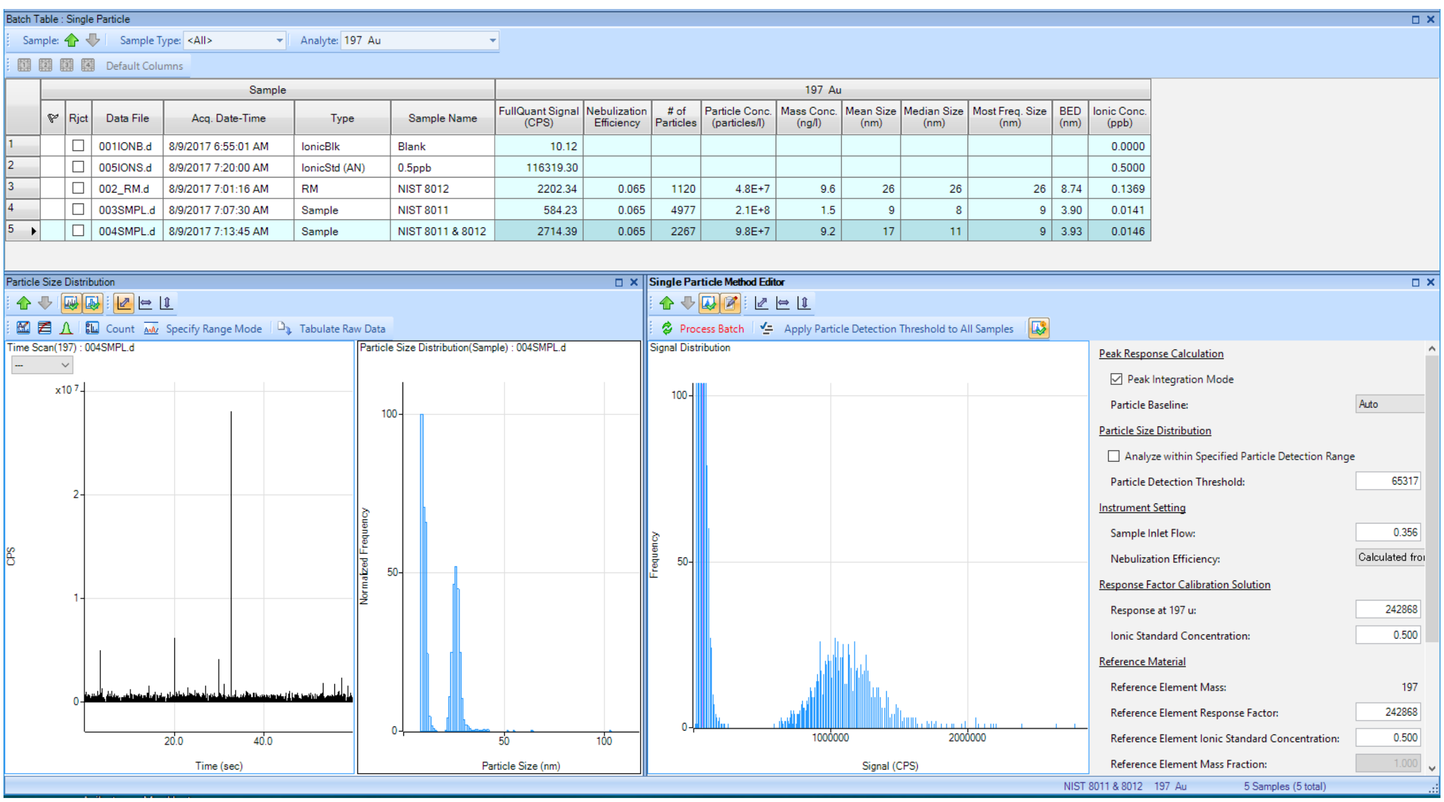
4		003SMPL.d	8/9/2017 7:07:30 AM	Sample	NIST 8011	584.23	0.065	740	3.2E+7	0.3	10	10	10	5.40	0.0397
5		004SMPL.d	8/9/2017 7:13:45 AM	Sample	NIST 8011 & 8012	2714.39	0.065	2267	9.8E+7	9.2	17	11	9	3.93	0.0146

Figure 2 The ICP-MS MassHunter nanoparticle data analysis pane, showing tabular results for entire sequence of samples and graphical results for selected sample–NIST 8011 10 nm Au.



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4		003SMPL.d	8/9/2017 7:07:30 AM	Sample	NIST 8011	584.23	0.065	4977	2.1E+8	1.5	9	8	9	3.90	0.0141
5		004SMPL.d	8/9/2017 7:13:45 AM	Sample	NIST 8011 & 8012	2714.39	0.065	2267	9.8E+7	9.2	17	11	9	3.93	0.0146

Figure 3. The data analysis pane, showing graphical results for the mixed NIST 8011 and 8012 reference materials containing 10 and 30 nm particles.

Figure 3 shows the same data analysis panel with the sample containing a mixture of 10 nm and 30 nm particles selected. The signal distribution and particle size distribution plots clearly show the results for both particle size populations, which are well resolved. The FTRA plot on the left shows several peaks of different heights (indicating different particle diameters) measured in the raw time resolved data.

Conclusions

A simple method for the detection and characterization of 10 nm Au nanoparticles and a mixture of 10 nm and 30 nm nanoparticles was created.

The method took advantage of the high sensitivity and ease of optimization of the Agilent 7800 ICP-MS, using no-gas mode coupled with ICP-MS MassHunter's simple, powerful single particle application module.

Developing the method using the Method Wizard required only a few mouse clicks, after which the short sequence was automatically analyzed.

Single particle analyses are very fast, typically only 60 seconds per sample. The entire sequence illustrated in this note—including calibration, determination of nebulization efficiency and sample analysis—took less than half an hour.

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