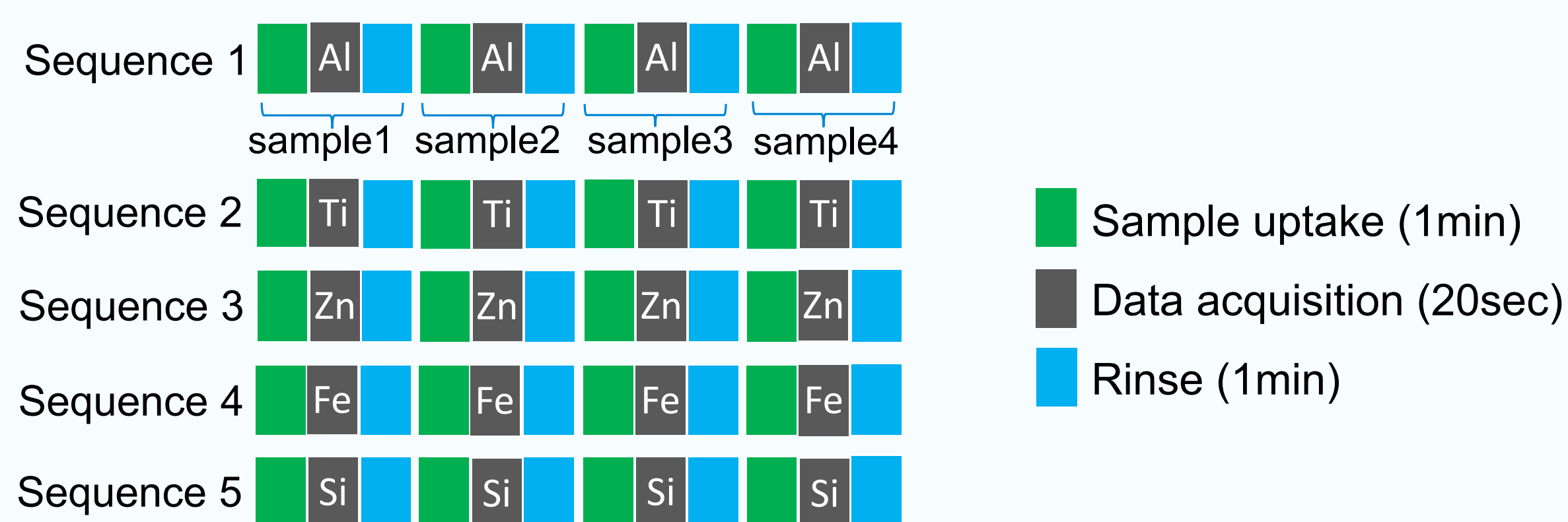


Introduction

- **spICP-MS (single particle ICP-MS)** is a powerful tool to measure metal-containing nanoparticles since it provides the particle concentration, particle size, particle distribution and dissolved element concentration simultaneously
- **Multi-elemental analysis** of spICP-MS is now being required for understanding which elements the NPs in a sample are composed of, and thereby gaining the information of the NPs' origin
- We recently developed a **Fast Time Program Analysis (FTPA) function** to rapidly measure multiple elements sequentially by spICP-MS mode within a single analysis
- FTPA function can reduce measurement time, simplify the multielement analysis
- In this study, Al, Ti, Zn and Si in sunscreens and swimming pool water samples were measured using the FTPA function and the application of the method to real samples is discussed

Example: measurement of 4 samples for 5 elements

Conventional method (1 element per sequence)



FTPA function (multielement screening)



16 uptakes and 16 rinses can be eliminated = 32 min shorter at the maximum (assuming no wait time between each element measurement)

Experimental

Reference Material and Samples

Au NP: NIST 8013 (nominal 60nm diameter) used for measuring the nebulization efficiency

Sunscreens: purchased in a local store in Tokyo

Swimming pool water: collected from swimming pools in Tokyo area

Sample Preparation

Sunscreens: All samples were diluted 50x with 1% Triton X-100 in de-ionized water, followed by 100,000x with 0.01% Triton X-100 to make working solutions. The ingredient information of Al, Ti, Zn and Si provided by sunscreen manufactures is shown in Table 1.

Table 1. Sunscreen ingredients according to the manufactures

	Al	Ti	Zn	Si
Sunscreen A	Al(OH) ₃	TiO ₂	-	SiO ₂
Sunscreen B	-	-	-	-
Sunscreen C	Al(OH) ₃	TiO ₂	ZnO	cyclopentasiloxane

Swimming pool water: measured by ICPMS without any sample preparations.

Instrument and Conditions

- Agilent 7900 ICP-MS and MassHunter's Single Particle Application module were used for data collection and analysis
- ²⁷Al, ⁴⁷Ti, ⁶⁶Zn and ²⁸Si were measured using FTPA function
- No gas mode: ²⁷Al, ⁴⁷Ti, ⁶⁶Zn
H₂ mode: ²⁸Si
- Standard quartz sample introduction systems were used with a 1.0 mm injector diameter torch
- General analytical conditions are shown in Table 2

Table 2. Operational conditions

Parameter	Value
RF power	1550 w
Sampling depth	8.0 mm
Carrier gas	0.87 L/min
Sample uptake rate	0.35 mL/min
Spray chamber temp.	2 °C
Dwell time	0.1 ms
Settling time	0 ms
H ₂ flow at H ₂ mode	5.0 mL/min
Data acquisition time	20 s/element

Results and Discussion

Time resolved data of NPs in sunscreen samples

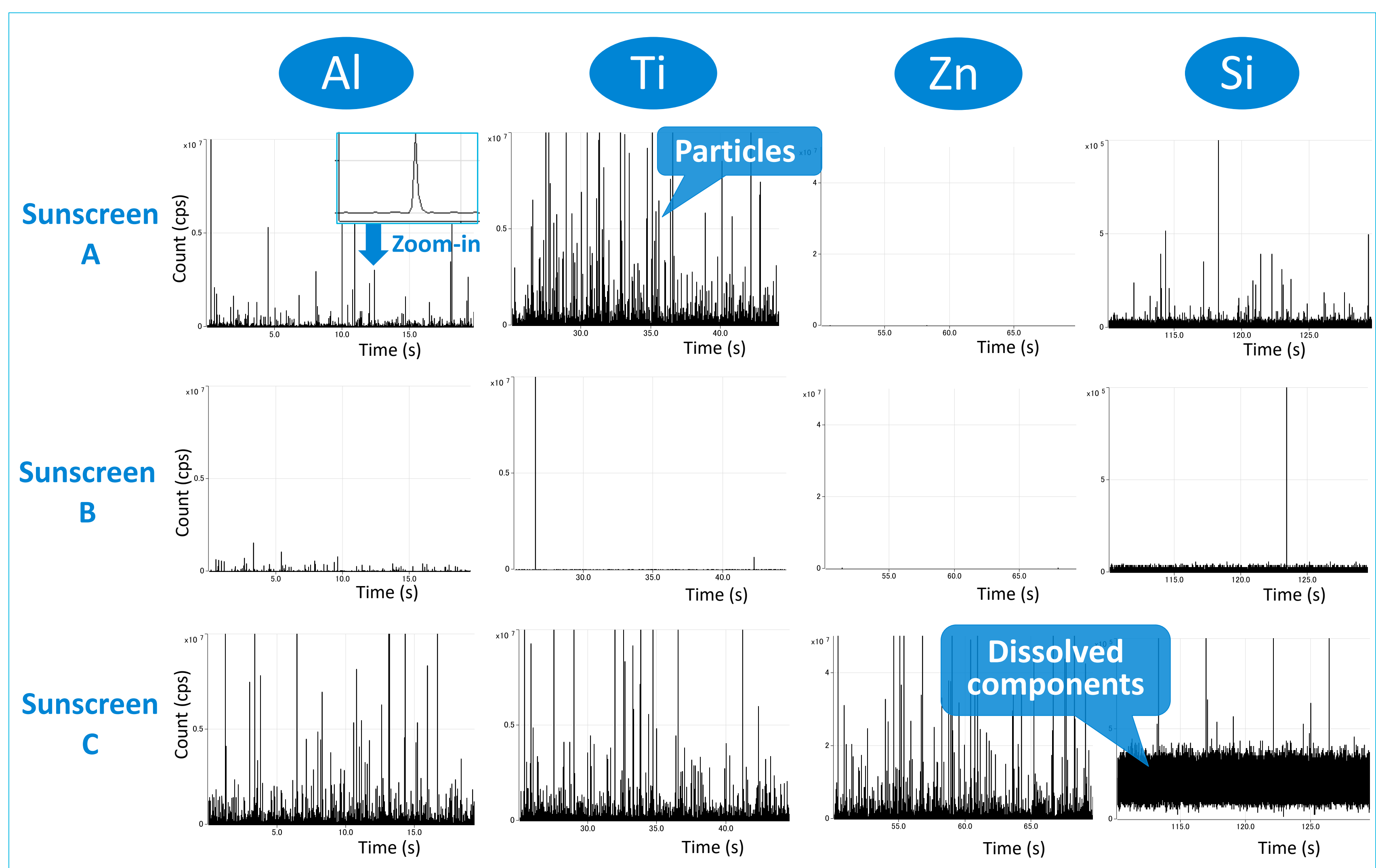


Figure 1. Time resolved data of NPs in sunscreens

- As shown in Figure 1, sharp peaks from metal containing NPs were detected in sunscreen A and C, while no NPs were observed in sunscreen B. This agreed with the ingredient information according to the manufactures (Table 1).
- The high and continuous background of Si in sunscreen C means that dissolved Si components exist in the sample. This is likely cyclopentasiloxane, which is one of the main ingredients for cosmetic products and has a unique fluidity that makes it easily spreadable.
- Figure 2 shows the quantitative results for Al, Ti, Zn and Si. The spICP-MS with FTAP function indicates its ability to give qualitative and quantitative information without a time-consuming acid digestion of sunscreen samples.

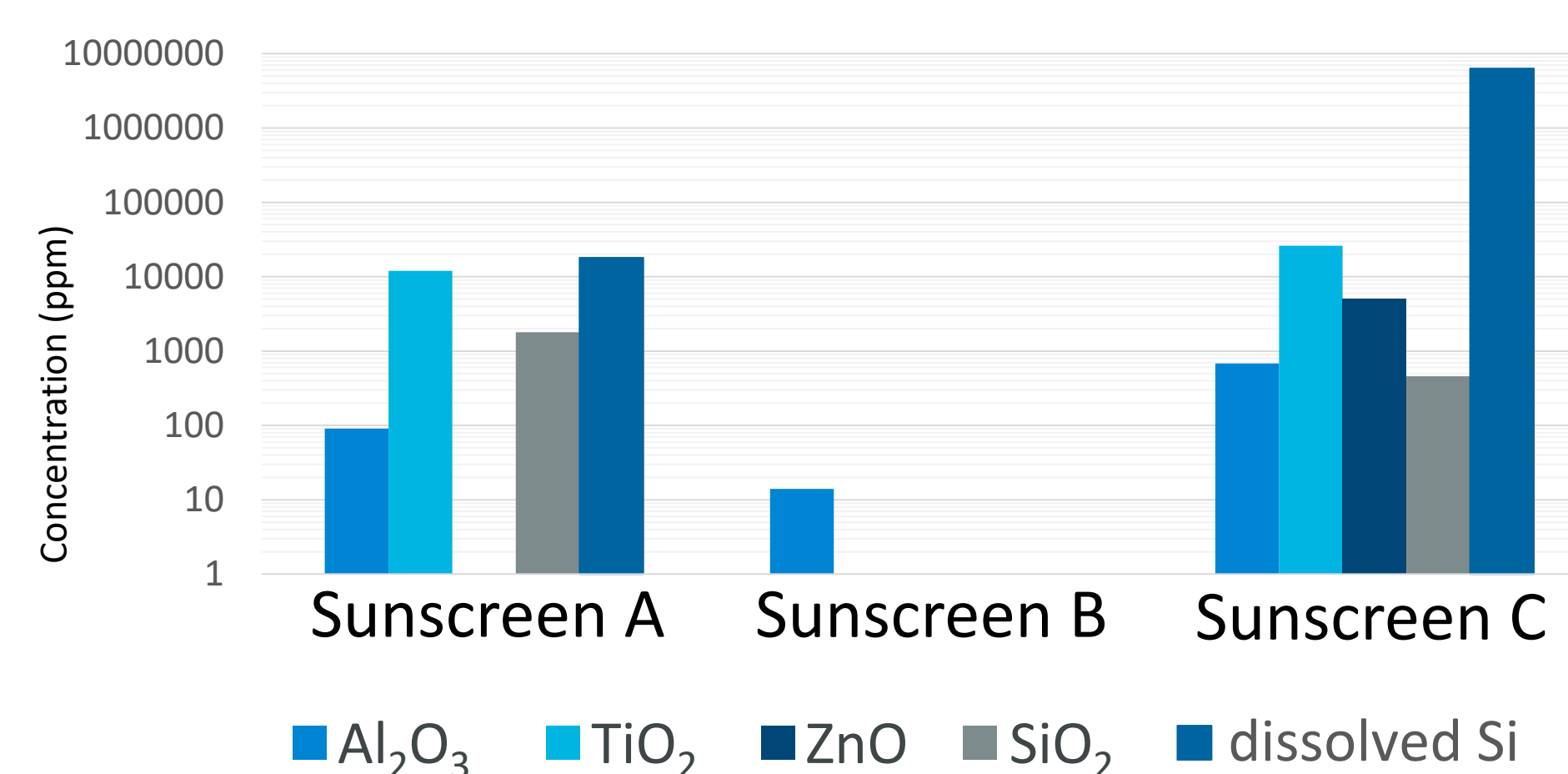


Figure 2. quantitative result in sunscreens

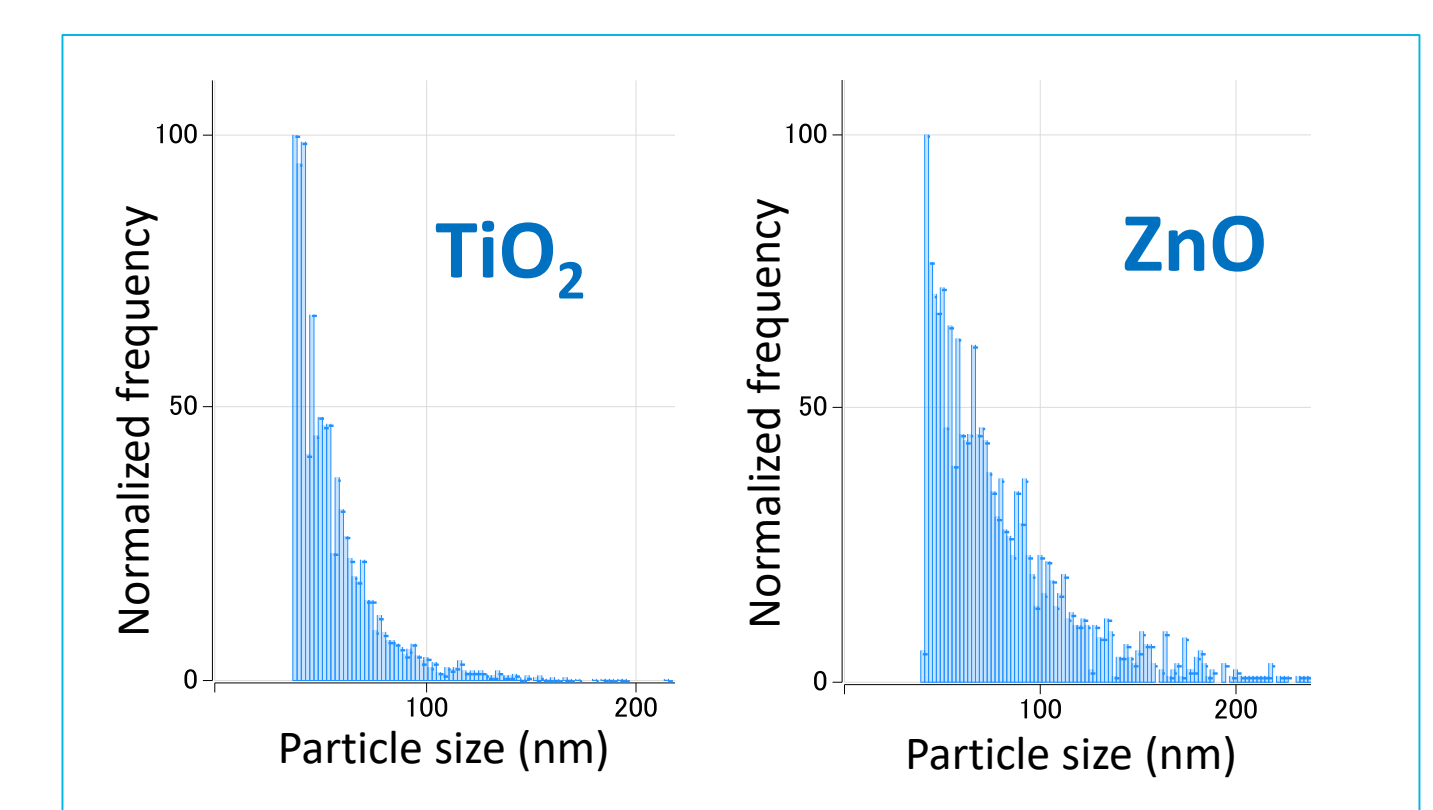


Figure 3. Particle size distribution of sunscreen C

Nanoparticles in swimming pool

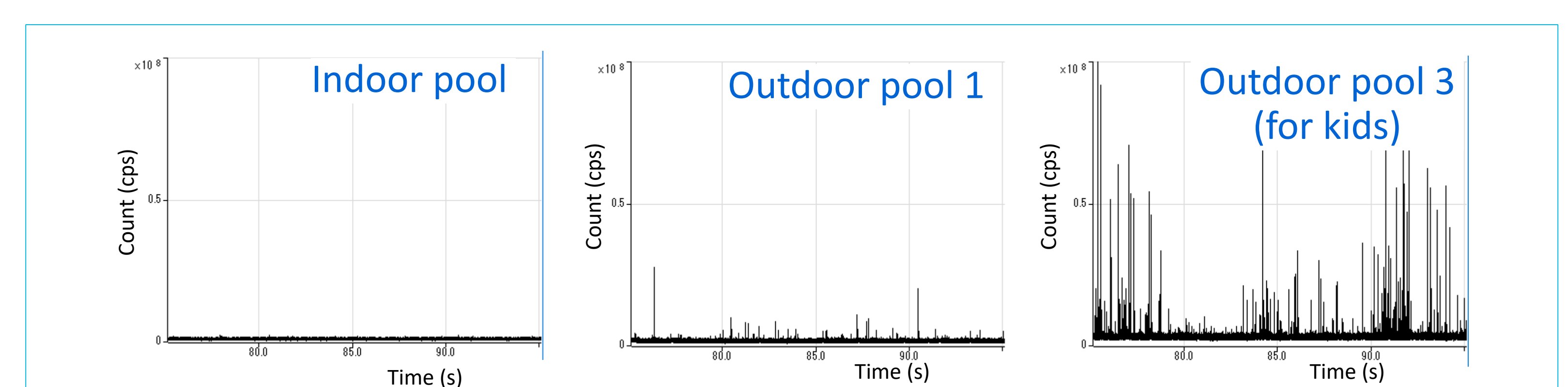


Figure 4. Time resolved data for Zn

Table 2. particle numbers in swimming pool waters

	Detected particle number (particles/20s)		Particle concentration (particles/L)	
	TiO ₂	ZnO	TiO ₂	ZnO
Pure water	1	2	1.3 x 10 ⁵	2.6 x 10 ⁵
Indoor pool	30	7	3.9 x 10 ⁶	9.1 x 10 ⁵
Outdoor pool 1	105	122	1.4 x 10 ⁷	1.6 x 10 ⁷
Outdoor pool 2	187	181	2.4 x 10 ⁷	2.3 x 10 ⁷
Outdoor pool 3 (for kids)	395	390	5.1 x 10 ⁷	5.1 x 10 ⁷

Conclusions

FTPA function with spICP-MS module in MassHunter

- provides particle concentration, particle size, particle distribution and dissolved element concentration for multiple elements in a single analysis
- simplifies the analytical method and shortens the sample run time
- can be used for a real sample analysis without a time-consuming sample preparation

References

1. V. Nischwitz and H. Goenaga-Infante, J. Anal. At. Spectrom., 2012, 27(7), 1084-1092.
2. P. Lu, S. Huang, Y. Chen, L. Chiueh and D. Y. Shih, J. Food and Drug Anal., 2015, 23, 587-594.