

U6000AT+ Application Note

Measurement of Silicon in Methyl Isobutyl Ketone (MIBK) using Ultrasonic Nebulization with Membrane Desolvation and ICP-AES Detection

Silicone-based compounds are commonly used in the health care industry. The silicone content (as silicon) in these products may be monitored for quality control purposes. The use of organic solvents, such as methyl isobutyl ketone (MIBK) for extraction of the silicone-based materials presents a particular challenge for the measurement of Si by inductively coupled plasma atomic emission spectrometry (ICP-AES). The volatility of the organic solvents can cause loss of plasma, plasma instability, and carbon buildup on the ICP torch.

The CETAC U6000AT+ Ultrasonic Nebulizer with Membrane Desolvator removes most of the organic solvent during transport of Si to the ICP-AES, allowing for stable plasma operation. The CETAC BGX-100 Blend Gas Accessory is used to add a low-flow of oxygen (approx. 30 mL/min) between the U6000AT+ and the ICP-AES, thus preventing any buildup of carbon on the ICP torch.

Operating Conditions

ICP-AES: PerkinElmer Optima 3000XL
Plasma gas: 15 L/min
Auxiliary gas: 0.5 L/min
Nebulizer gas: 0.65 L/min
Integration time: 5.0 s

CETAC U6000AT+:
Heater temp: 140°C
Cooler temp: -10°C
Membrane oven temp: 160°C
Uptake rate: 1.0 mL/min
Inlet pump tubing: PharMed™

CETAC BGX-100 Blend Gas Accessory:
Oxygen rate: 30 mL/min

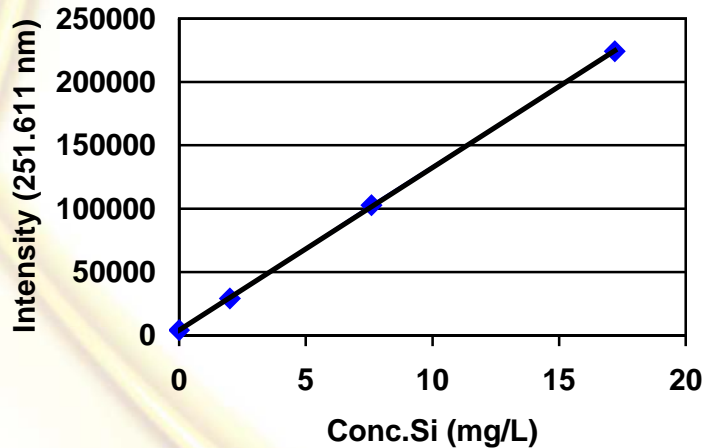


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Calibration

A calibration curve was obtained for three Si standards in MIBK: 2.0 mg/L, 7.6 mg/L, and 17.2 mg/L. Using the 251.611 nm Si line an excellent curve with a correlation coefficient of 0.99996 was obtained. Note that no internal standard was used.



Recovery and Detection Limit

The middle standard was then analyzed six times against the calibration curve. Between each run the U6000AT+ was rinsed for 3 minutes with MIBK. The table below shows the mean results for each analysis (three 5-s integrations).

<u>Run #</u>	<u>Measured Si (mg/L)</u>	<u>% Recovery</u>
1	7.4 +/- 0.2	97
2	7.6 +/- 0.3	100
3	7.6 +/- 0.4	100
4	7.7 +/- 0.2	101
5	7.4 +/- 0.4	97
6	7.6 +/- 0.4	100

The instrument detection limit (IDL) for Si at 251.611 nm was determined to be 0.01 mg/L or 10 ug/L.

Samples

A set of four samples (with spiked levels of Si) were then analyzed. Excellent agreement was obtained versus the spike values.

<u>Sample #</u>	<u>Known Si Conc. (mg/L)</u>	<u>Measured Si Conc. (mg/L)</u>
1	5.0	5.4 +/- 0.3
2	5.0	5.4 +/- 0.1
3	2.0	1.9 +/- 0.1
4	2.0	2.0 +/- 0.1

