

# Silica Analyzer Calibration Verification: Ensuring Confidence in the Quality of Steam Cycle Water

## Introduction

When used together, the Hach® 5500sc Silica Analyzer [0.5-5000 µg/L SiO<sub>2</sub>] and Ultra-Low-Range (ULR) Silica Method 8282 lab analysis [3-1000 µg/L SiO<sub>2</sub>] ensure the most accurate and reliable silica monitoring results. The DR3900 spectrophotometer may be used to perform Method 8282, as well as to measure a host of other parameters. The 5500sc is an independent and self-calibrated system ready from startup with quality reagents delivered from one single source supplier. Additionally, the 5500sc may be calibrated and validated with external standards verified with the DR3900 and introduced via the "Grab Sample In" function. The "Grab Sample Out" function allows for easy online measurement verification with the DR3900. Single sourcing of reagents for each analysis eliminates variability due to reagent quality.

## Background

Both the 5500sc Silica Analyzer and DR3900 ULR Silica analysis use the sensitive heteropoly blue chemistry to measure low concentrations of silica for high purity steam cycle applications. First, any silica and phosphate ions in the sample are reacted with the molybdate ion to form phosphomolybdic and silicomolybdic acid complexes. The phosphomolybdic acid complexes are then destroyed by the addition of citric acid. Finally, the remaining silicomolybdic acid complexes are reduced with an amino naphthol sulfonic acid to form a blue colored heteropoly acid. The intensity of this blue species is proportional to the amount of silica in the original sample. Each of the reagents used for this analysis are identical for the 5500sc and DR3900.

Both the 5500sc and DR3900 implement a built-in calibration curve. However, it is critical that these calibrations be periodically verified to ensure that the curves are accurate for each specific instrument. Instrument response can drift over time due to regular wear and tear. While these issues are addressed through regular maintenance, it is recommended that verification be performed periodically to ensure that accuracy is maintained throughout the maintenance interval.

5500sc	DR3900
Reagent 1 Silica – 6774802	Molybdate 3 Reagent – 199532
Reagent 2 Silica – 6774902	Citric Acid Reagent – 2254232
Reagent 3 Silica – 6775202	Amino Acid F Dilution Solvent – 2353011
Reagent 3 Silica Powder – 6775355	Amino Acid F Reagent Powder – 2651155
Silica Standard – 6775002	Silica Standards – 2100817
Silica Reagent Set – 6783600	Silica Reagent Set – 2553500



DR3900 Spectrophotometer



5500sc Silica Analyzer

## Calibration Verification

Calibration verification can be performed by analyzing a known standard, or by analyzing a single sample with different instruments. Each of these techniques is easily performed with the 5500sc and DR3900. Known standards can be analyzed on the 5500sc through the "Grab Sample In" function. Known standards measured with the 5500sc and DR3900 show excellent accuracy and agreement between instruments (see Figure 1). If measured values do not correlate known concentrations, the measurement is inaccurate. The source of this inaccuracy must be identified and corrected. Follow troubleshooting instructions in the instrument manual and procedure.

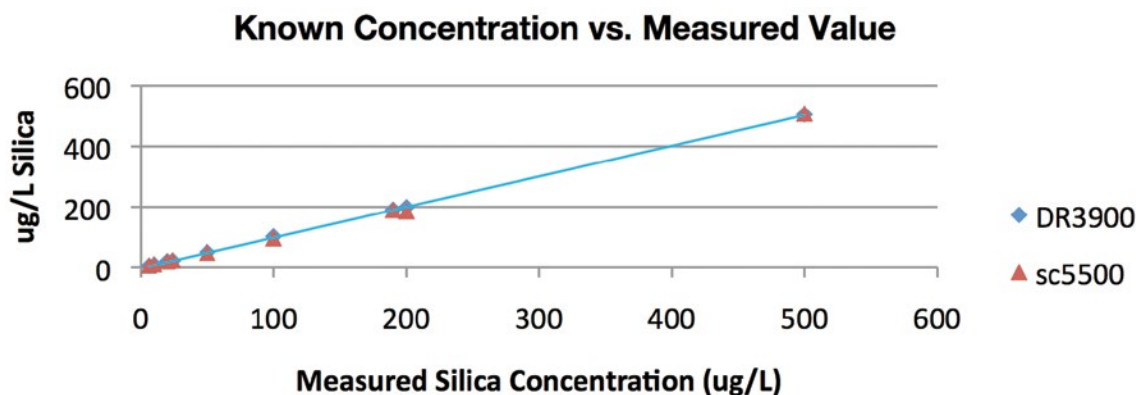


Figure 1: Known Standard Verification

Process samples can be verified with the 5500sc "Grab Sample Out" function. Known standards and process grab samples can both be analyzed with the DR3900 lab procedure. Comparison of measured results against known standards or between instruments will indicate maintenance issues or poor analytical technique. Standards and samples measured on each instrument show excellent agreement (see Figure 2). If inter-instrument measurements do not correlate, one of the measurements is inaccurate. The source of this inaccuracy must be identified and corrected. Follow troubleshooting instructions in the instrument manual and procedure.

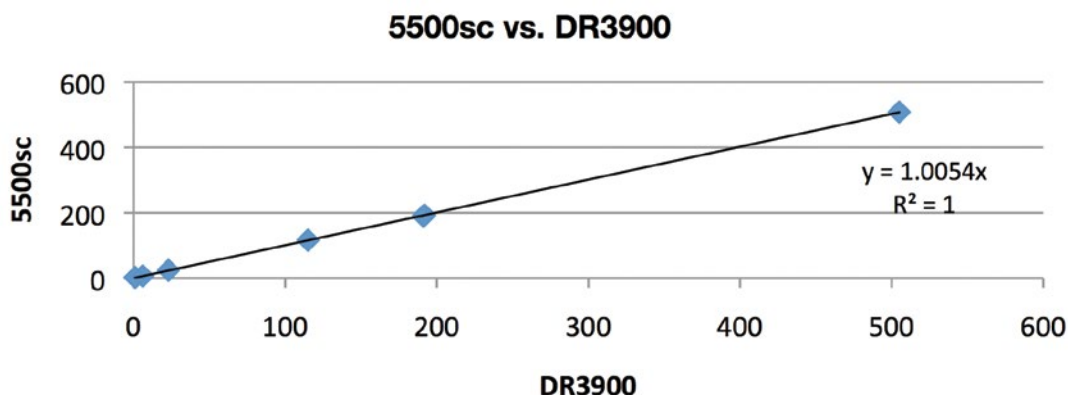


Figure 2: Inter-Instrument Verification

## Procedure

### 5500sc Known Standard Verification:

1. Access the "Grab Sample" menu from the home screen on the 5500sc.
  - a. Select "Grab Sample In."
  - b. Follow the on-screen instructions.
2. Remove the sample funnel from the analyzer and rinse it with the standard (see Figure 3).
3. Reinstall the funnel.
4. Pour 250-500 mL of standard into the funnel when prompted by the analyzer.
5. The measured value will display on the analyzer screen.
6. Grab sample results are stored in the Data Event Log.

### DR3900 Known Standard Verification:

1. Follow the written procedure using the grab sample.
2. The measured value will display on the spectrophotometer screen.
3. Sample results are stored in the Data Log

### 5500sc Process Sample Verification:

1. Access the "Grab Sample" menu from the home screen on the 5500sc.
  - a. Select "Grab Sample Out."
  - b. Follow the on-screen instructions.
2. Open the lower analyzer compartment.
3. Open the Grab Sample Out line by switching the valve open.
4. Rinse the sample container several times.
5. Use the sample for the DR3900 analysis.
6. The measured value will display on the analyzer screen.
7. Grab sample results are stored in the Data Event Log.

### DR3900 Process Sample Verification:

1. Follow the written procedure using the grab sample.
2. The measured value will display on the spectrophotometer screen.
3. Sample results are stored in the Data Log.

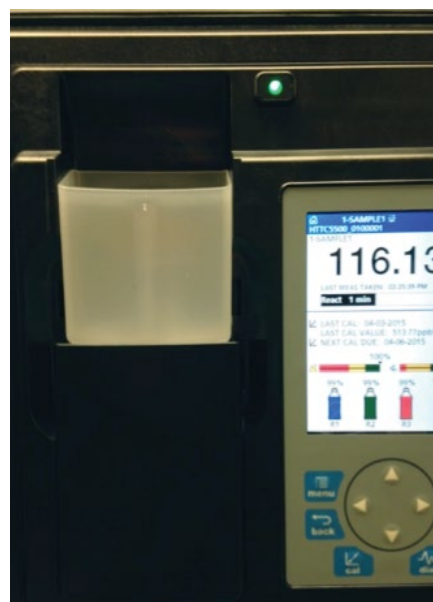


Figure 3: Grab sample funnel

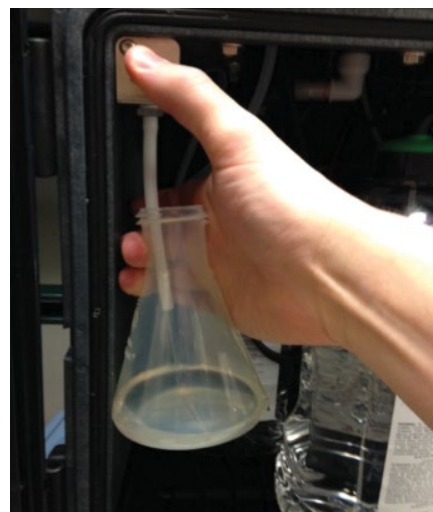


Figure 4: Grab sample outlet

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