

Ultrasensitive determination of silicate in process and boiler water

Photometric determination using the PMB method after decomposition in a thermoreactor

Introduction

Build-up of undesirable deposits (i.e. scale) on industrial equipment like pipes, boilers, and turbines, is a problem that must be addressed in order to maintain efficiency. One of the main causes of this type of buildup, especially at high pressure, is silicate, which is often dissolved in steam.¹ To minimize the need for time-consuming cleaning operations that interrupt operations, regular silicate determination in process and boiler water is necessary. Here, we describe the ultrasensitive quantification of silicate in process and boiler water using the Spectroquant® photometric system.

Experimental

Method

In sulfuric solution silicate ions react with molybdate ions to form a yellow heteropoly acid. This is reduced to silicomolybdenum blue that is determined photometrically. The method is analogous to APHA 4500-SiO₂ D+E, DIN 38 405-21, and ASTM D859-16.

Reagents and Instruments

Cat. No.	Product Description
Test Kits	
1.01813	Spectroquant® Silicate (Silicic Acid) Test Method: photometric 0.25 - 500.0 µg/l SiO ₂ 0.12 - 233.7 µg/l Si
1.01262	Ultrapur Water
1.09138	Sodium hydroxide solution 0.5 mol/L Titripur®
1.12310	Silicon standard solution traceable to SRM from NIST acidic, (NH ₄) ₂ SiF ₆ in H ₂ O 1000 mg/l Si Certipur®
Instruments	
1.73018	Spectroquant® UV/VIS Spectrophotometer Prove 600

Reagents and Instruments (continued)

Cat. No.	Product Description
Materials	
1.74011	Rectangular cell 100 mm
XX1102012	Plastic Syringes, 20 mL, Luer slip, polypropylene
SLGP033NS	Syringe filter holder Millex-GP Filter, 0.22 µm, PES 33 mm, non-sterile
	Preparation vessels: Volumetric flasks and sample beakers, plastic
	Precision pipettes with plastic tips for 200 µl, 1000 µl, and 20 ml

Analytical Approach

Sample preparation using Variant 1:

If the solution shows any signs of turbidity, filter the sample over the above-mentioned syringe filter holder into a clean and dry plastic vessel.

Conditioning of the working materials

If disposable plastic materials are not available (e.g. disposable syringes for piston pipettes; 50-mL centrifuge tubes), the materials should first be rinsed free of silicate with sodium hydroxide solution 0.5 mol/L and subsequently washed until pH-neutral with Ultrapur water.

Measurement procedure

Determine the SiO₂ content with the above-mentioned test kit. In deviation from the instructions for use for the test kit, to enhance the precision reagents Si-1 and Si-2 are not added by dropping, but instead using a pipette. For a sample volume of 20 mL, the corresponding volumes of 200 µL of reagent Si-1 and 200 µL of reagent Si-2 and 1.00 mL of reagent Si-3 are pipetted into the sample.

The measurement is carried out against a blank, prepared from Ultrapur water and the reagents in an analogous manner. The reaction times A and B remain unchanged. After measurement, the result can be read off from the instrument.

Results

Comparison of results of the Spectroquant® Silicate Test 1.01813 with graphite-tube AAS reference analysis

Sample	Spectroquant® Silicate Test 1.01813	GF-AAS
Concentration [µg/L SiO₂]		
Ultrapur water	< 0.25	< 1.93
Steam water from power plant	3.26	2.25
Boiler water from power plant	4.85	3.66
DI-water	0.29	< 1.93
Double-distilled water	0.77	< 1.93

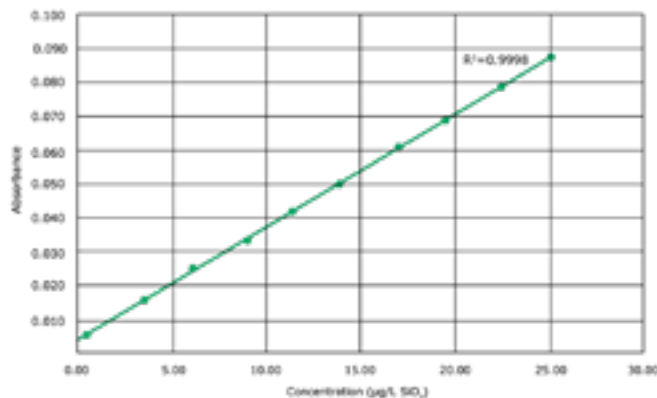
Standard addition with the Spectroquant® Silicate Test 1.01813

Sample	Addition (µg/L SiO ₂)	Recovered concentration (µg/L SiO ₂)
Ultrapur water	1.00	0.86
	5.00	6.25
	10.00	10.60
Steam water from power plant	1.00	1.83
	5.00	6.14
	10.00	11.01
Boiler water from power plant	1.00	1.24
	5.00	6.09
	10.00	10.20
DI-water	1.00	1.97
	5.00	5.74
	10.00	11.31
Double-distilled water	1.00	1.75
	5.00	7.40
	10.00	11.53

Note:

If the accuracy of the method is to be increased further, we recommend carrying out an own calibration for a limited measuring range. We recommend the performance of the calibration according to the ISO 8446-1 method. For this purpose, a user-defined method can be programmed on the Prove 600. For details see Prove Manual chapter 9.6 "User-defined Concentration Methods".

By recording a 10-point calibration curve for the measuring range 0.50 – 25.00 µg/L SiO₂ the following performance characteristics could be achieved:



Product Description	User-defined calibration 0.50 - 25.00 µg/L SiO ₂
Method standard deviation (µg/L)	+ 0.185
Method coefficient of variation (%)	+ 1.44
Confidence interval (P = 95 %) (µg/L)	+ 0.450

References

1. Bahadori A, Vuthaluru HB. Prediction of silica carry-over and solubility in steam of boilers using simple correlation. Applied Thermal Engineering. 2010;30(2-3):250-253. doi:10.1016/j.applthermaleng.2009.07.010

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