Technical Assistance
HYDROLOGY PROJECT

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Training module # WQ - 46

How to Measure Silicate

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with
HALCROW, TAHAL, CES, ORG & JPS

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1. Module context

This module introduces importance of silicate in natural waters, and measurement of silicate in water samples by ammonium molybdate spectrophotometric method.

While designing a training course, the relationship between this module and the others, would be maintained by keeping them close together in the syllabus and place them in a logical sequence. The actual selection of the topics and the depth of training would, of course, depend on the training needs of the participants, i.e. their knowledge level and skills performance upon the start of the course.

No.	Module title	Code	Objectives
1.	Basic water quality concepts	**WQ - 01**	• Discuss the common water quality parameters • List important water quality issues
2.	Basic chemistry concepts	**WQ - 02**	• Convert units from one to another • Discuss the basic concepts of quantitative chemistry • Report analytical results with the correct number of significant digits.
3.	The need for Good Laboratory Practice	**WQ – 03**	• Follow approved general laboratory procedures
4.	How to prepare standard solutions	**WQ – 04**	• Select different types of glassware • Use an analytical balance and maintain it • Prepare standard solutions
5.	Basic Aquatic Chemistry concept	**WQ – 24**	• Calculate ion concentrations from ionisation constants
6.	Major ions in water	**WQ - 28**	• Know the major ions in water and air sources • Understand the significance of major ion concentrations
7.	Absorption Spectroscopy	**WQ - 34**	• Understand the principle of absorption spectroscopy • Explain the use of absorption spectroscopy for chemical analyses

2. Module profile

Title	:	How to Measure Silicate
Target group	:	HIS function(s): Q2, Q3, Q5, Q6
Duration	:	1 theoretical session of 30 min, plus 1 Practical Laboratory session of 120 min, plus 1 Report writing session of 30 min.
Objectives	:	After the training the participants will be able to*:* • Understand the relevance of silicate to water quality • Analyse water samples for silicate
Key concepts	:	• Molybdosilicate method for silicate
Training methods	:	Lecture, Laboratory Analytical Exercise, Report preparation
Training tools required	:	Board, flipchart, OHS, Complete Laboratory Facilities for silicate Analysis
Handouts	:	As provided in this module including SAP for Analysis of Silicate
Further reading and references	:	• Chemistry for environmental engineers - C. N. Sawyer, P. L. McCarty & G. F. Parkin, McGraw - Hill, Inc., 1994 • Standard methods for the examination of water and wastewaters, AWWA, 19th edition, 1995

3. Session plan

No	Activities	Time	Tools
1	***Preparations*** • Prepare reagents required for analysis according to SAP • Prepare samples as follows: A – Groundwaer B and C – Using distilled water and silica stock solution prepare samples containing 0.8 and 3 mg/L silica		
2	***Introduction***: • Introduce the session • Ask the question: "Why do we need to measure silicate?" • Talk about the occurrence of silicate in the environment	10 min	List answers on flip chart OHS
3	***Ammonium Molybdate Spectrophotometric Method – Background:*** • Discuss the ammonium molybdate method in terms of its chemistry, the theory of spectrophotometry and potential interferences	20 min	OHS
4	***Practical Session***: • Allow participants to conduct the silicate analysis according to SAP: **Refer to SAP for silicate** • Stress the need to write up material as the analysis is proceeding • Be available to guide participants and answer questions	120 min	OHS
5	***Report Writing:*** • Allow participants to complete their reports • Give the 'correct' answers to the silicate analyses • Discuss results	30 min	Use flip chart

4. Overhead/flipchart master

OHS format guidelines

Type of text	Style	Setting
Headings:	OHS-Title	Arial 30-36, with bottom border line (not: underline)
Text:	OHS-lev1 OHS-lev2	Arial 24-26, maximum two levels
Case:		Sentence case. Avoid full text in UPPERCASE.
Italics:		Use occasionally and in a consistent way
Listings:	OHS-lev1 OHS-lev1-Numbered	Big bullets. Numbers for definite series of steps. Avoid roman numbers and letters.
Colours:		None, as these get lost in photocopying and some colours do not reproduce at all.
Formulas/Equations	OHS-Equation	Use of a table will ease horizontal alignment over more lines (columns) Use equation editor for advanced formatting only

Silicon

- Next to oxygen in abundance in earth's crust
- Silica (SiO_2) in sand and quartz
- Silicate Minerals
- Causes scaling problems

Molybdosilicate Method

- Silica reacts with molybdate to give yellow colour
- Phosphate also undergoes similar reaction
  - *oxalic acid is added to destroy colour due to phosphate*
- Colour and turbidity of sample may also interfere
  - *run suitable blank for compensation*
- Read colour at 815 nm
- Dilute sample if silica > 2 mg/L

Experiment

- Aim:
  - *To determine silicate in different samples of water*
- Method:
  - *Collect samples A, B and C*
  - *Dilute sample if out of range*
  - *Use SAP*
- Report:
  - *Discuss observed silicate conc. in terms of water quality*

5. Evaluation sheets

6. Handout

Measurement of Silicate

- Next to oxygen in abundance in earth's crust
- Silica (SiO_2) in sand and quartz
- Silicate minerals
- Causes scaling problems

Molybdosilicate Method

- Silica reacts with molybdate to give yellow colour
- Phosphate also undergoes similar reaction
  - *oxalic acid is added to destroy colour due to phosphate.*
- Colour and turbidity of sample may also interfere
  - *Run suitable blank for compensation*
- Read colour at 815 nm.
- Dilute sample if silica > 2 mg/L.

Experiment

- **Aim**
  - *To determine silicate in different samples of water*

- **Method**
  - *Collect samples A, B and C*
  - *Dillute Sample if out of range*
  - *Use SAP*

- **Report**
  - *Discuss observed silicate conc. in terms of water quality.*

Add copy of Main text in chapter 8, for all participants.

7. Additional handout

These handouts are distributed during delivery and contain test questions, answers to questions, special worksheets, optional information, and other matters you would not like to be seen in the regular handouts.

It is a good practice to pre-punch these additional handouts, so the participants can easily insert them in the main handout folder.

8. Main text

Contents

How to Measure Silicate

1. Introduction

Silicon occurs as the oxide (silica) in quartz and sand and in the form of complex silicate minerals, particularly igneous rocks. Silica occurs in natural waters as suspended particles, in a colloidal or polymaric state and as silicilic acids as silicate ions. Its concentration ranges between 1 – to 30 – mg/L.

Silica forms scales in boilers, which is difficult to remove. Silica and silicate scales particularly formed on high pressure steam turbine blades are troublesome.

2. Molybdosilicate Method

Ammonium molybdate at low pH reacts with silica and any phosphate present to produce hetropoly acids giving a yellow colour. Oxalic acid is added to destroy the molybdo phosphoric acid. The intensity of the colour can be measured at 815 nm. In atleast one of its forms, the silica does not react with molybdate. To detect unreactive, silica the sample may be digested with sodium bicarbonate. The method can detect upto 0.2 mg/L silica. Samples containign more than 10 mg/L silica should be diluted.

Interference due to colour and turbidity may be compensated by running a parallel blank without addition of molybdate, which should be used to adjust the spectrophotometers for zero absorbance.

3. Experiment

Aim

a. To determine the concentration of silicate ion in a number of different samples by spectrophotometry

Method

a. Collect a sample from each of the buckets marked A, B and C.
b. Determine the silicate in each sample according to the Standard Analytical Procedure for silicate, ammonium molybdate spectrophotometric method.

Observations & calculations

a. Fill in the following table as you proceed with the method:

Standard or Sample	Absorbance at 815 nm (R)
0.4 mg/l Standard	
0.8 mg/l Standard	
1.2 mg/l Standard	
1.6 mg/l Standard	
2.0 mg/l Standard	
A	
B	
C	

b. Use the values of the standard solutions in the table to plot a graph of absorbance vs. silicate concentration.

c. Read the silicate concentration of the three samples from the standard curve.

Report

When writing your report the following aspects should be addressed:

- the aim of the investigation
- the results that you have produced
- the silicate concentration of the samples and what this could mean in terms of water quality