Technical Assistance
HYDROLOGY PROJECT

World Bank & Government of The Netherlands funded

Training module # WQ - 39

How to measure Ammonia Nirogen: Phenate Method

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DHV Consultants BV & DELFT HYDRAULICS

with
HALCROW, TAHAL, CES, ORG & JPS

Table of contents

1. Module context

This module describes the laboratory procedure for measurement of ammonia nitrogen by phenate method. Modules in which prior training is recommended to complete this module successfully and other available, related modules in this category are listed in the table below:

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.	Basic ecology concepts	**WQ - 26**	• Explain how energy flows through an aquatic ecosystem • Explain how nutrients are cycled in the environment • Explain the causes and problems of eutrophication
4.	Surface water quality planning concepts	**WQ - 27**	• Understand principles of WQ monitoring and assessment • Know of simple data analysis methods
5.	Use of ion selective probes	**WQ - 33**	• Precautions required in use of ion selective electrodes
6.	Absorption spectroscopy	**WQ - 34**	• Understand theory and applications of absorption spectroscopy
7.	How to measure Oxidised Nitrogen: Cd reduction method and UV Spectrophotometric methods	**WQ - 37**	• Measure oxidised nitrogen by Cd-reduction and UV spectrophotometric methods • Appreciate limitations of the UV method
8.	How to measure Ammonia and Organic Nitrogen: Kjeldahl method	**WQ - 38**	• Understand the relevance of nitrogen to water quality • Know how to make analysis of ammonia and organic nitrogen by Kjeldahl method.

2. Module profile

Title	:	How to measure Ammonia Nirogen: Phenate Method
Target group	:	HIS function(s): Q2, Q3,Q5, Q6
Duration	:	One lecture session 15 min, One laboratory session 90 min One concluding session 15 min
Objectives	:	After the training the participants will be able to*:* • measure ammonia by Phenate Method
Key concepts	:	• Phenate Method
Training methods	:	Lecture, Laboratory
Training tools required	:	Board, flipchart, OHS, chemical laboratory, spectrophotometer
Handouts	:	As provided in this module
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*** • Reagents as described in SAP • Samples: A – Tap water B – Sample A 100 mL + 20 μg NH_3-N C – Sample A 100 mL + 50 μg NH_3-N		
2	***Introduction***: • Describe significance of nitrogen in water • Describe briefly the Phenate method • Ask participants to read the SAP	15 min	OHS
3	***Laboratory*** • Divide the class in groups of 2 to 3 persons • Provide the stock ammonia standard solution and ask each group to prepare atleast 4 standards • While the participants are preparing standards, demonstrate the working of the spectrophotometer to each group separately. • Ask the participants to complete the experiment	120 min	Laboratory
4	***Report and wrap up*** • Ask the participants to prepare their reports • Discuss results	30 min	

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

Significance of Nitrogen in water

- Eutrophication
  - *explosive growth in algae*
  - *decay of algal biomas and oxygen depletion*
- Nitrification reaction
  - *consumption of oxygen*
- Health problem
- Related to sewage pollution

Determination of Ammonia Nitrogen

- Distillation and titration
  - *Kjeldahl method*
- Nesslerization
  - *oldest method*
  - *environmental hazards*
- Phenate method
  - *recommended under HP*

Phenate Method

- Ammonia reacts with phenol to form indophenol in presence of alkali and oxidising agent
- Sodium nitroprusside acts as catalyst
- The developed blue colour absorbs light of 640 nm wavelength

Interference

- Ca^{++} and Mg^{++}
  - *form precipitate at high pH*
  - *complex with citrate*
- Turbidity
  - *remove by distillation or filtration*
- H_2S
  - *remove by aeration after acidifying sample*

Experiment

- Determine NH_3-N in samples by Phenate Method

Sample	Source	Expected conc. mg/L
A	Tap water	0 – 0.1
B	Sample A 100 mL + 20 μg NH_3-N	0.2
C	Sample A 100 mL + 50 μg NH_3-N	0.5

- Calibration standards: 0.1, 0.3, 0.5 and 0.7 mg NH_3-N/L

Report

- Aim of the experiment
- Results including calibration standard curve
- Comment on results and quality of samples

5. Evaluation sheets

6. Handout

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- Results including calibration standard curve
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Add copy of Main text in chapter 8, for all participants.

7. Additional handout

8. Main text

Contents

How to measure Ammonia Nirogen: Phenate Method

1. Introduction

Various aspects of occurrence of different forms of nitrogen, organic, ammonia, nitrite and nitrate in water, were dicussed in modules # 26 – 'Basic Ecology concepts' and # 27 – 'How to measure Ammonia and organic nitrogen. Briefly these are:

- fertilization leading to eutrophication and accompanying problems of decomposition of plant matter, oxygen depletion, odours, etc.
- nitrification and deoxygenation
- health related problems: methaemoglobinemia
- indicator of organic pollution, such as, sewage

Determination of ammonia nitrogen by distillation and titration was also described in module # 38.

Ammonia can also be measured directly colorimetrically. Ammonia combines with alkaline solution of potassium mercuric iodide (Nessler's reagent) to form a yellowish brown colloidal dispersion, the colour intensity of which is directly proportional to the amount of ammonia originally present. This is one of the older colorimetric procedure.

Due to the possible harmful environmental effects from the use of mercury in Nesslerization reaction, an alternative phenate method is described in this module which is recommended under HP.

2. Phenate Method

The method involves addition of phenol solution together with hypochlorite and nitroprusside catalyst to the sample. Ammonia reacts to form indophenol, which has an intense blue colour. The colour abosorbs light of 640 nm wavelength. The colour intensity is affected by the age of the reagents; therefore atleast two calibration standards are run with each measurement.

Calcium and manganese in sample may interfere by precipitating at high pH. Complexing with citrate removes the interference. Turbidity in sample should be removed by distillation or filtration. Hdrogen sulphide, if present, can be removed by aeration after acidifying the sample to pH 3.

3. Aim

To determine the concentration of ammonia nitrogen in water samples by Phenate method.

4. Method

a) Read the SAP for determination of ammonia nitrogen by Phenate method.
b) Collect samples for analysis. The source of sample and expected ammonia concentrations are given below

Sample	Source	Expected conc. mg/L
A	Tap water	0 – 0.1
B	Sample A 100 mL + 20 μg NH_3-N	0.2
C	Sample A 100 mL + 50 μg NH_3-N	0.5

c) Prepare calibration standards containing 0.1, 0.3, 0.5 and 0.7 mg NH_3-N/L
d) Develop colour in the samples and the standards according to SAP.

5. Observation and calculations

a) Fill in the following table as you proceed with the test

Sample	**Absorbance at 640 nm**
0.1 mg NH_3 – N/L standard	
0.3 mg NH_3 – N/L standard	
0.5 mg NH_3 – N/L standard	
0.7 mg NH_3 – N/L standard	
Sample A	
Sample B	
Sample C	

b) Use the values of the standard solutions in the table to plot a calibration curve of mg NH_3 – N/L vs absorbance.
c) Read the ammonia content of each sample from the calibration curve.

6. Report

Write your report addressing the following

- The aim of the experiment
- The results obtained by you
- Comment on the results if they are different from the expected values
- Comment on quality of samples in terms of ammonia contents