

	<p style="text-align: center;">Standard Operating Procedure</p> 	Effective Date: 20 June 2013	Version: 002
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SOP_Chem_007 Chemical Analysis_ Spectroquant Nitrite			Page #: 1 of 4

Standard Operation Procedure – Nitrite Test, Spectroquant, Cell Test (Cat. No. 1.09713)

1. Scope and Application,

- Test measures the nitrite concentration in the range 1.0 – 90 mg/l NO₂-N.

2. Summary

- In acidic solution nitrite ions react with iron(II) ethylenediammonium sulphate to form a yellow to green-brown iron(II) compound that is determined photometrically.

3. Apparatus and Glassware

- Spectroquant.
- Reaction cells.
- Pipette.

4. Interferences

Concentrations of foreign substances in mg/l or %					
Ag ⁺	1	Hg ²⁺	100	EDTA	1000
Ca ²⁺	1000	Mg ²⁺	1000	Reducing agents (ascorbic acid, sulfite)	10
Cd ²⁺	1000	Mn ²⁺	1000		
BO ₃ ²⁻	1000	Mo ⁶⁺	500	NaCl	20 %
CN ⁻	1000	NH ₄ ⁺	1000	NaNO ₃	20 %
Cr ³⁺	100	Pb ²⁺	1000	Na ₂ SO ₄	15 %
CrO ₄ ²⁻	100	PO ₄ ³⁻	1000		
Cu ²⁺	100	S ²⁻	10		
F ⁻	100	SiO ₃ ²⁻	1000		
Fe ³⁺	1	Zn ²⁺	1000		

CO_3^{2-}	100	Sn^{2+}	10	
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5. Collection, Preservation and Storage

- Collect faecal samples in 1L plastic buckets.
- Preferably, analyse samples immediately after sampling.
- Store samples at 4 °C or freeze dry samples.
- Preserve wastewater samples by acidifying with concentrated sulphuric acid to pH 2 and faecal samples by freeze drying or freezing.
- Determine $\text{NO}_2\text{-N}$ on well- homogenised samples.
- Samples containing more than 90.0 mg/l $\text{NO}_2\text{-N}$ must be diluted with distilled water.
- The pH must be within the range 1 - 12. Adjust, if necessary, with sulfuric acid.
- Filter turbid samples.

6. Safety Precautions

- Handle concentrated acid with care.
- Always use safety goggles, gloves and laboratory coat while working in laboratory.
- Wear face shield and protect hands from heat produced when contents of the vessels are mixed. After the analysis, clean bottles and beakers with clear water keep it for drying.
- Dispose the used gloves after completion of analysis.
- Clean the hands using antiseptic soap.
- Disinfect hands after washing with soap.
- Avoid spillage and contact with skin. In the latter case use copious washings with cold water and call for medical attention.

7. Sample Preparation –Faecal Sludge

1. Weigh out 2.0000g of well-mixed faecal sludge sample.
2. Blend the weighed sample with 500ml of distilled water in a 1L blender for 30 seconds on the highest speed.
3. Add 250ml distilled water and blend on highest speed until the sample is homogenised (this could range from 30 to 60 seconds).
4. Transfer the blended mixture into a 1L volumetric flask.
5. Add 200ml of blender washings into the flask and top up to 1L with distilled water.
6. Transfer the 1L solution to a plastic bottle and store at 4 °C.

8. Reagents

- Reagent $\text{NO}_2\text{-1K}$.
- Universal indicator strips pH 0-14.

- pH indicator strips, pH 0-6.
- Sulphuric acid 0.5 mol/l.

9. Calibration

- To check the photometric measurement system (test reagents, measurement device, handling) and the mode of working, use the nitrite solution 40 mg/l NO₂-N.
- Prepare a series of at least three standards, covering the desired range, and a blank by diluting suitable volumes of standard solutions. Prepare a calibration curve by plotting instrument response against standard concentration. Compute sample concentration by comparing sample response with the standard curve. Multiply answer by appropriate dilution factor. Report only those values that fall between the lowest and the highest calibration standards. Dilute and reanalyse amples exceeding the highest standard. Report results in mg/L.

10. Procedure

- Place 2 level blue micro spoons of reagent NO₂-1K in a reaction cell.
- Pipette 8.0 ml of pre-treated sample into the reaction cell, close the cell and shake until the reagent is completely dissolved.
- Leave to stand for exactly 20 minutes, then measure the sample in the photometer. Do not shake or swirl the cell before the measurement.

Notes on the measurement:

- For photometric measurement, the cells must be clean. Wipe, if necessary, with a clean dry paper towel.
- Measurement of turbid solutions yields false-high readings.
- The pH of the measurement solution must be within the range 1.2 - 1.6.
- The colour of the measurement solution remains stable for only a short time after the end of the reaction time stated above.

11. Waste Disposal

- Collect waste in a 2.5L bottle for collection from Waste Tech.

12. Data Quality

Measurement	10 – 150 mg/l N
Standard Deviation (mg/l N)	± 1.1
Confidence Interval (mg/l N)	± 3
Sensitivity (mg/l N)	

Accuracy (mg/l N)	
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13. References

<http://www.merckmillipore.com/ZA/en/products/analytcs-sample-prep/test-kits-and-photometric-methods/instrumental-test-systems-for-quantitative-analyses/photometric-measurements-spectroquant-system/spectroquant-tests/>

APPROVAL OF STANDARD OPERATING PROCEDURE

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