

Nitrite Spectroquant® Test Kits Equivalency Checklist

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Introduction

The method flexibility allowed in the EPA rules 40 CFR part 136.6 [1] lay out the requirements a modified analytical method must meet to be considered equivalent to a promulgated analytical method. These requirements are explained in detail in a memo authored by Richard Redding [2]:

The Spectroquant® Test Kits are covered under the method flexibility allowed in the EPA rules 40 CFR part 136.6 [1] The rule lays out the requirements a modified analytical method must meet to be considered equivalent to a promulgated analytical method. These requirements are explained in detail in a memo authored by Richard Redding [2]:

The March 12th Methods Update Rule promulgated 136.6 which allows the regulated community more flexibility that includes:

- 1. Changes in equipment operating parameters such as minor changes in the monitoring wavelength of a colorimeter*
- 2. Adjusting sample sizes or changing extraction solvents to optimize method performance in meeting regulatory requirements*
- 3. Minor changes in reagents used where the underlying reaction and principles remain virtually the same:*
 - a. Changes in complexing reagent provided that the change does not produce interferences.*
 - b. Changes in reactants provided that the change does not produce interference.*

The method equivalency report for Spectroquant® Test Kits 1.14547, 1.14776 and 1.00609 will directly compare this kit with the allowed method modifications listed in Richard Reading's memo. The MDL results along with the IDC, LRB and LFB and % RSD all show good agreement with the Quality Control parameters that is expected in 40 CFR part 136.7 [1].

This equivalency checklist will directly compare the Spectroquant® nitrite test kits with the simple method modifications listed in Richard Reading's memo and allow a laboratory to establish method equivalency for their analyses and reporting to both users of the results and regulators.

Method Summary from Spectroquant® Equivalency Report

The report "*Equivalency of Spectroquant® Nitrite Method Azo Dye Nitrite Test Kits Nitrite Method Iron Redox Test Kit Reactions and Photometry*" [3] provides the single laboratory equivalency data required by EPA.[2] The significant areas covered in the report that summarize the areas that equivalency must be identified are listed in Table 1 below.

Table 1: Method Equivalency Summary	
Spectroquant® Change Test Kit 1.00609, 1.14547, 1.14776	136.6 Requirement
Wavelength change to new absorbance maxima	<ul style="list-style-type: none"> • Accuracy, Precision, MRL and MDL from the experimental data were acceptable.
Changes in complexing reagent. Changes in reactants.	<ul style="list-style-type: none"> • No interference is seen in the LFB, LFM, and LFMD • Accuracy, Precision, MRL and MDL from the experimental data were acceptable.
Adjusting sample sizes to amounts listed for the test kit.	<ul style="list-style-type: none"> • No interference is seen in the LFB, LFM, and LFMD • Accuracy, Precision, MRL and MDL from the experimental data were acceptable.

The “*Equivalency of Spectroquant® Nitrite Method Azo Dye Nitrite Test Kits Nitrite Method Iron Redox Test Kit Reactions and Photometry*” [3] report has provided the literature review information, EPA 40 CFR part 136.6 requirements and experimental data to support the equivalency of the current Spectroquant® nitrite test kits with the EPA current promulgated nitrite methods listed in the 40 CFR part 136.

Conclusion

In conclusion, these changes in the Spectroquant® test kits (or future test kits with the same equivalent chemistry) produce an equivalent set of tests to the current EPA promulgated nitrite methods listed in the 40 CFR part 136.

Current Spectroquant® Nitrite Test Kits of Merck KGaA, Darmstadt, Germany listed in this report are:

1. 1.00609
2. 1.14547
3. 1.14776

In Table 2 below, Method Equivalency of Standard Methods for the Examination of Water and Wastewater Nitrite Method to Spectroquant® Nitrite Test Kits, approved Standard Methods for the Examination of Water and Wastewater nitrite method requirements listed in the Reding memo and 40 CFR part 136.7 is compared to the Spectroquant test kits. This checklist should be kept onsite at the laboratory so as to be provided to any user of the laboratory results or as needed for regulatory audits/requirements.

Copies of the “*Equivalency of Spectroquant® Nitrite Method Azo Dye Nitrite Test Kits Nitrite Method Iron Redox Test Kit Reactions and Photometry*” [3]” report or the “*Nitrite Spectroquant® Analytical Test Kit Method*” can be obtained from <http://www.emdmillipore.com/USEPA>

Table 2: Method Equivalency of Standard Methods for the Examination of Water and Wastewater Nitrite Method to Spectroquant® Nitrite Test Kits

Method or Quality Control Requirement	Standard Methods for the Examination of Water and Wastewater 4500-NO₂ (B) 1997,2011	Spectroquant® Nitrite Test Kits 1.14547 1.14776	Spectroquant® Nitrite Test Kits 1.00609
1. Analyte Detected	NO ₂ or NO ₂ -N	NO ₂ or NO ₂ -N	NO ₂ or NO ₂ -N
2. Matrix Tested	Water	Water	Water
3. Detection Method	Visible Absorbance Maxima	Visible Absorbance Maxima	Visible Absorbance Maxima
4. Wavelength Maxima (nm)	537	542	450
Analytical Reagents Required			
5. pH Adjustment Chemical	1 N HCl or 1 N NaOH	1 N H ₂ SO ₄ or 1 N NaOH	1 N H ₂ SO ₄ or 1 N NaOH
6. Color Reagent	Phosphoric Acid Sulfanilamide N-(1-naphthyl)-ethylenediamine dihydrochloride	Sulfanilic acid* N-(1-naphthyl)-ethylenediamine dihydrochloride <i>* Note: acid provides pH adjustment and same reactive group as Sulfanilamide.</i>	Iron (2) ethylene diammonium sulfate
Quality Control Requirements from 40 CFR part 136.7			

Table 2: Method Equivalency of Standard Methods for the Examination of Water and Wastewater Nitrite Method to Spectroquant® Nitrite Test Kits

Method or Quality Control Requirement	Standard Methods for the Examination of Water and Wastewater 4500-NO₂ (B) 1997,2011	Spectroquant® Nitrite Test Kits 1.14547 1.14776	Spectroquant® Nitrite Test Kits 1.00609
1. Demonstration of Capability (DOC),	Required in 4020	Required in Spectroquant® Nitrite Method	Required in Spectroquant® Nitrite Method
2. Method Detection Limit (MDL),	Required in 4020	Required in Spectroquant® Nitrite Method Single Laboratory Results in: Spectroquant® Equivalency Report	Required in Spectroquant® Nitrite Method Single Laboratory Results in: Spectroquant® Equivalency Report
3. Reagent blank (also referred to as method blank),	Required in 4020	Required in Spectroquant® Nitrite Method Single Laboratory Results in: Spectroquant® Equivalency Report	Required in Spectroquant® Nitrite Method Single Laboratory Results in: Spectroquant® Equivalency Report
4. Laboratory fortified blank (LFB, also referred to as a spiked blank, or laboratory control sample (LCS)),	Required in 4020	Required in Spectroquant® Nitrite Method Single Laboratory Results in: Spectroquant® Equivalency Report	Required in Spectroquant® Nitrite Method Single Laboratory Results in: Spectroquant® Equivalency Report

Table 2: Method Equivalency of Standard Methods for the Examination of Water and Wastewater Nitrite Method to Spectroquant® Nitrite Test Kits

Method or Quality Control Requirement	Standard Methods for the Examination of Water and Wastewater 4500-NO₂ (B) 1997,2011	Spectroquant® Nitrite Test Kits 1.14547 1.14776	Spectroquant® Nitrite Test Kits 1.00609
5. Matrix spike (MS), matrix spike duplicate (MSD), or laboratory fortified blank duplicate (LFBD) for suspected difficult matrices,	Required in 4020	Required in Spectroquant® Nitrite Method Single Laboratory Results in: Spectroquant® Equivalency Report	Required in Spectroquant® Nitrite Method Single Laboratory Results in: Spectroquant® Equivalency Report
6. Internal standard/s, surrogate standard/s (for organic analysis) or tracer (for radiochemistry),	Not Required	Not Required	Not Required
7. Calibration (initial and continuing),	Required in 4020	Required in Spectroquant® Nitrite Method Single Laboratory Results in: Spectroquant® Equivalency Report	Required in Spectroquant® Nitrite Method Single Laboratory Results in: Spectroquant® Equivalency Report
8. Control charts (or other trend analyses of quality control results), and	Required in 4020	Required in Spectroquant® Nitrite Method	Required in Spectroquant® Nitrite Method

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Method or Quality Control Requirement	Standard Methods for the Examination of Water and Wastewater 4500-NO₂ (B) 1997,2011	Spectroquant® Nitrite Test Kits 1.14547 1.14776	Spectroquant® Nitrite Test Kits 1.00609
9. Corrective action (root cause analyses),	Required in 4020	Required in Spectroquant® Nitrite Method	Required in Spectroquant® Nitrite Method
10. Specific frequency of QC checks,	Required in 4020	Required in Spectroquant® Nitrite Method	Required in Spectroquant® Nitrite Method
11. QC acceptance criteria,	Required in 4020	Required in Spectroquant® Nitrite Method	Required in Spectroquant® Nitrite Method
12. Definitions of a batch (preparation and analytical)	Required in 4020	Required in Spectroquant® Nitrite Method	Required in Spectroquant® Nitrite Method

References

1. EPA, *Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Analysis and Sampling Procedures*. 2012. p. 29758-29846.
2. Reding, R., *Flexibility to Modify CWA Methods*, E. Engineering & Analytical Support Branch, OST, Editor. 2007, EPA.
3. Askew, E.F., *Equivalency of Spectroquant® Nitrite Method Azo Dye Nitrite Test Kits Nitrite Method Iron Redox Test Kit Reactions and Photometry*. 2016.