

# Spectroquant<sup>®</sup> Nitrate Test Kits Equivalency Checklist

---

## Table of Contents

<b>Introduction</b> .....	<b>3</b>
<b>Method Summary from Spectroquant® Equivalency Report</b> .....	<b>3</b>
Table 1: Test Kit Method Equivalency Summary .....	4
<b>Conclusion</b> .....	<b>6</b>
Table 2: Method Equivalency of Standard Methods for the Examination of Water and Wastewater Nitrate Method to Spectroquant® Nitrate Test Kits .....	7
<b>References</b> .....	<b>13</b>

## 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<sup>®</sup> Test Kits: 1.14764, 1.14563, 1.09713, 1.00614, 1.14542, 1.14942, 1.14773, 1.14556 and 1.01842 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 12<sup>th</sup> 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<sup>®</sup> Test Kits 1.14764, 1.14563, 1.09713, 1.00614, 1.14542, 1.14942, 1.14773, 1.14556 and 1.01842 will directly compare these kits 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<sup>®</sup> nitrate 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<sup>®</sup> Equivalency Report

The report "*Method Equivalency Spectroquant<sup>®</sup> Nitrate 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.

<b>Table 1: Test Kit Method Equivalency Summary</b>		
<b>Spectroquant® Test Kit</b>	<b>Spectroquant® Change</b>	<b>136.6 Requirement</b>
1.14764, 1.14563, 1.09713, 1.00614, 1.14542, 1.14942, 1.14773, 1.14556	Nitronium Ion Formation and Electrophilic Substitution to Add a Nitro Group to 2,6 Xylenol, Resorcinol or a Substituted Benzoic Acid	<p>Changes Allowed Under Method Equivalency.</p> <ul style="list-style-type: none"> <li>• Changes in complexing reagent provided that the change does not produce interferences.</li> <li>• Changes in reactants provided that the change does not produce interference.</li> <li>• Accuracy, Precision, MRL and MDL from the experimental data were acceptable.</li> </ul>
1.14764, 1.14563, 1.09713, 1.00614, 1.14542, 1.14942, 1.14773, 1.14556	Absorbance Maxima Changed	<p>Changes Allowed Under Method Equivalency.</p> <ul style="list-style-type: none"> <li>• Changes in equipment operating parameters such as minor changes in the monitoring wavelength of a colorimeter</li> <li>• Accuracy, Precision, MRL and MDL from the experimental data were acceptable.</li> </ul>

<b>Table 1: Test Kit Method Equivalency Summary</b>		
<b>Spectroquant® Test Kit</b>	<b>Spectroquant® Change</b>	<b>136.6 Requirement</b>
1.00609	Azo Dye Reaction with Substituted Benzoic Acid	<p>Changes Allowed Under Method Equivalency.</p> <ul style="list-style-type: none"> <li>• Minor changes in reagents used where the underlying reaction and principles remain virtually the same:</li> <li>• Changes in complexing reagent provided that the change does not produce interferences.</li> <li>• Changes in reactants provided that the change does not produce interference.</li> <li>• Accuracy, Precision, MRL and MDL from the experimental data were acceptable.</li> </ul>
	Absorbance Maxima Changed	<p>Changes Allowed Under Method Equivalency.</p> <ul style="list-style-type: none"> <li>• Changes in equipment operating parameters such as minor changes in the monitoring wavelength of a colorimeter</li> <li>• Accuracy, Precision, MRL and MDL from the experimental data were acceptable.</li> </ul>

The *Method Equivalency Spectroquant® Nitrate 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® nitrate test kits with the EPA current promulgated nitrate methods listed in the 40 CFR part 136.

## Conclusion

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

Current Merck KGaA, Darmstadt, Germany, Spectroquant<sup>®</sup> Nitrate Test Kits listed in this report are:

1. 1.14764
2. 1.14563
3. 1.09713
4. 1.00614
5. 1.14542
6. 1.14942
7. 1.14773
8. 1.14556
9. 1.01842

In Table 2 below, Method Equivalency of Standard Methods for the Examination of Water and Wastewater Nitrate Method to Spectroquant<sup>®</sup> Nitrate Test Kits, approved Standard Methods for the Examination of Water and Wastewater nitrate method requirements listed in the Reding memo and 40 CFR part 136.7 is compared to the Spectroquant<sup>®</sup> 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 “*Spectroquant<sup>®</sup> Nitrate Test Kit Method*” [4] can be obtained from <http://www.emdmillipore.com/USEPA>

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

<b>Method or Quality Control Requirement</b>	<b>Standard Methods for the Examination of Water and Wastewater 4500-NO<sub>3</sub> (E) 2000,2011</b>	<b>Spectroquant® Nitrate Test Kits</b> 1.14764 1.14563 1.09713 1.00614	<b>Spectroquant® Nitrate Test Kits</b> 1.14542 1.14773	<b>Spectroquant® Nitrate Test Kits</b> 1.14942 1.14556	<b>Spectroquant® Nitrate Test Kits</b> 1.01842
1. Analyte Detected	NO <sub>3</sub> or NO <sub>3</sub> -N	NO <sub>3</sub> or NO <sub>3</sub> -N	NO <sub>3</sub> or NO <sub>3</sub> -N	NO <sub>3</sub> or NO <sub>3</sub> -N	NO <sub>3</sub> or NO <sub>3</sub> -N
2. Matrix Tested	Water	Water	Water	Water	Water
3. Detection Method	Visible Absorbance Maxima	Visible Absorbance Maxima	Visible Absorbance Maxima	Visible Absorbance Maxima	Visible Absorbance Maxima
4. Wavelength Maxima (nm)	537 nm	1.14764 337 nm 1.14563 334 nm 1.09713 357 nm 1.00614 345 nm	1.14542 520 nm 1.14773 516 nm	1.14942 499 nm 1.14556 504 nm	1.01842 500 nm
<b>Analytical Reagents Required</b>					
5. pH Adjustment Chemical	1 N HCl or 1 N NaOH	1 N H <sub>2</sub> SO <sub>4</sub> or 1 N NaOH	1 N H <sub>2</sub> SO <sub>4</sub> or 1 N NaOH	1 N HCl or 1 N NaOH	1 N H <sub>2</sub> SO <sub>4</sub> or 1 N NaOH

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

<b>Method or Quality Control Requirement</b>	<b>Standard Methods for the Examination of Water and Wastewater 4500-NO<sub>3</sub> (E) 2000,2011</b>	<b>Spectroquant® Nitrate Test Kits</b> 1.14764 1.14563 1.09713 1.00614	<b>Spectroquant® Nitrate Test Kits</b> 1.14542 1.14773	<b>Spectroquant® Nitrate Test Kits</b> 1.14942 1.14556	<b>Spectroquant® Nitrate Test Kits</b> 1.01842
6. Color Reagent	Phosphoric Acid Sulfanilamide N-(1-naphthyl)-ethylenediamine dihydrochloride	Nitronium Ion 2,6 Xylenol	Nitronium Ion Benzoic Acid Derivative	Nitronium Ion Resorcinol	Diazotized Sulfanilic Acid Benzoic Acid Derivative
<b>Quality Control Requirements from 40 CFR part 136.7</b>					
7. Demonstration of Capability (DOC),	Required in 4020	Required in Spectroquant®, Nitrate Method	Required in Spectroquant®, Nitrate Method	Required in Spectroquant®, Nitrate Method	Required in Spectroquant®, Nitrate Method
8. Method Detection Limit (MDL),	Required in 4020	Required in Spectroquant®, Nitrate Method Single Laboratory Results in: Spectroquant®, Nitrate Equivalency Report	Required in Spectroquant®, Nitrate Method Single Laboratory Results in: Spectroquant®, Nitrate Equivalency Report	Required in Spectroquant®, Nitrate Method Single Laboratory Results in: Spectroquant®, Nitrate Equivalency Report	Required in Spectroquant®, Nitrate Method Single Laboratory Results in: Spectroquant®, Nitrate Equivalency Report



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

<b>Method or Quality Control Requirement</b>	<b>Standard Methods for the Examination of Water and Wastewater 4500-NO<sub>3</sub> (E) 2000,2011</b>	<b>Spectroquant® Nitrate Test Kits</b> 1.14764 1.14563 1.09713 1.00614	<b>Spectroquant® Nitrate Test Kits</b> 1.14542 1.14773	<b>Spectroquant® Nitrate Test Kits</b> 1.14942 1.14556	<b>Spectroquant® Nitrate Test Kits</b> 1.01842
9. Reagent blank (also referred to as method blank),	Required in 4020	Required in Spectroquant®, Nitrate Method  Single Laboratory Results in: Spectroquant®, Nitrate Equivalency Report	Required in Spectroquant®, Nitrate Method  Single Laboratory Results in: Spectroquant®, Nitrate Equivalency Report	Required in Spectroquant®, Nitrate Method  Single Laboratory Results in: Spectroquant®, Nitrate Equivalency Report	Required in Spectroquant®, Nitrate Method  Single Laboratory Results in: Spectroquant®, Nitrate Equivalency Report
10. Laboratory fortified blank (LFB, also referred to as a spiked blank, or laboratory control sample (LCS)),	Required in 4020	Required in Spectroquant®, Nitrate Method  Single Laboratory Results in: Spectroquant®, Nitrate Equivalency Report	Required in Spectroquant®, Nitrate Method  Single Laboratory Results in: Spectroquant®, Nitrate Equivalency Report	Required in Spectroquant®, Nitrate Method  Single Laboratory Results in: Spectroquant®, Nitrate Equivalency Report	Required in Spectroquant®, Nitrate Method  Single Laboratory Results in: Spectroquant®, Nitrate Equivalency Report

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

<b>Method or Quality Control Requirement</b>	<b>Standard Methods for the Examination of Water and Wastewater 4500-NO<sub>3</sub> (E) 2000,2011</b>	<b>Spectroquant® Nitrate Test Kits</b> 1.14764 1.14563 1.09713 1.00614	<b>Spectroquant® Nitrate Test Kits</b> 1.14542 1.14773	<b>Spectroquant® Nitrate Test Kits</b> 1.14942 1.14556	<b>Spectroquant® Nitrate Test Kits</b> 1.01842
11. Matrix spike (MS), matrix spike duplicate (MSD), or laboratory fortified blank duplicate (LFBD) for suspected difficult matrices,	Required in 4020	Required in Spectroquant®, Nitrate Method  Single Laboratory Results in: Spectroquant®, Nitrate Equivalency Report	Required in Spectroquant®, Nitrate Method  Single Laboratory Results in: Spectroquant®, Nitrate Equivalency Report	Required in Spectroquant®, Nitrate Method  Single Laboratory Results in: Spectroquant®, Nitrate Equivalency Report	Required in Spectroquant®, Nitrate Method  Single Laboratory Results in: Spectroquant®, Nitrate Equivalency Report
12. Internal standard/s, surrogate standard/s (for organic analysis) or tracer (for radiochemistry),	Not Required	Not Required	Not Required	Not Required	Not Required

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

<b>Method or Quality Control Requirement</b>	<b>Standard Methods for the Examination of Water and Wastewater 4500-NO<sub>3</sub> (E) 2000,2011</b>	<b>Spectroquant® Nitrate Test Kits</b> 1.14764 1.14563 1.09713 1.00614	<b>Spectroquant® Nitrate Test Kits</b> 1.14542 1.14773	<b>Spectroquant® Nitrate Test Kits</b> 1.14942 1.14556	<b>Spectroquant® Nitrate Test Kits</b> 1.01842
13. Calibration (initial and continuing),	Required in 4020	Required in Spectroquant®, Nitrate Method  Single Laboratory Results in: Spectroquant®, Nitrate Equivalency Report	Required in Spectroquant®, Nitrate Method  Single Laboratory Results in: Spectroquant®, Nitrate Equivalency Report	Required in Spectroquant®, Nitrate Method  Single Laboratory Results in: Spectroquant®, Nitrate Equivalency Report	Required in Spectroquant®, Nitrate Method  Single Laboratory Results in: Spectroquant®, Nitrate Equivalency Report
14. Control charts (or other trend analyses of quality control results), and	Required in 4020	Required in Spectroquant®, Nitrate Method	Required in Spectroquant®, Nitrate Method	Required in Spectroquant®, Nitrate Method	Required in Spectroquant®, Nitrate Method
15. Corrective action (root cause analyses),	Required in 4020	Required in Spectroquant®, Nitrate Method	Required in Spectroquant®, Nitrate Method	Required in Spectroquant®, Nitrate Method	Required in Spectroquant®, Nitrate Method
16. Specific frequency of QC checks,	Required in 4020	Required in Spectroquant®, Nitrate Method	Required in Spectroquant®, Nitrate Method	Required in Spectroquant®, Nitrate Method	Required in Spectroquant®, Nitrate Method

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

<b>Method or Quality Control Requirement</b>	<b>Standard Methods for the Examination of Water and Wastewater 4500-NO<sub>3</sub> (E) 2000,2011</b>	<b>Spectroquant® Nitrate Test Kits</b> 1.14764 1.14563 1.09713 1.00614	<b>Spectroquant® Nitrate Test Kits</b> 1.14542 1.14773	<b>Spectroquant® Nitrate Test Kits</b> 1.14942 1.14556	<b>Spectroquant® Nitrate Test Kits</b> 1.01842
17. QC acceptance criteria,	Required in 4020	Required in Spectroquant®, Nitrate Method	Required in Spectroquant®, Nitrate Method	Required in Spectroquant®, Nitrate Method	Required in Spectroquant®, Nitrate Method
18. Definitions of a batch (preparation and analytical)	Required in 4020	Required in Spectroquant®, Nitrate Method	Required in Spectroquant®, Nitrate Method	Required in Spectroquant®, Nitrate Method	Required in Spectroquant®, Nitrate 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., *Method Equivalency Spectroquant<sup>®</sup> Nitrate Test Kit: Reactions and Photometry*. 2017.
4. Askew, E.F., *Spectroquant<sup>®</sup> Nitrate Test Kit Method* 2017.