

# QUANTITATIVE ANALYSIS OF NITRITE AND NITRATE IN TOBACCO, TOBACCO PRODUCTS, AND TOBACCO DERIVED PRODUCTS WITH (b) (4)

## TABLE OF CONTENTS

<b>PURPOSE</b> .....	<b>3</b>
<b>APPLIES TO</b> .....	<b>3</b>
<b>GENERAL INFORMATION</b> .....	<b>3</b>
PRINCIPLE OF THE METHOD .....	3
METHOD SCOPE, MEASUREMENT RANGE AND MEASUREMENT UNCERTAINTY .....	4
SCOPE .....	4
MEASUREMENT RANGE .....	4
MEASUREMENT UNCERTAINTY .....	4
LITERATURE REFERENCES .....	5
<b>RISK ASSESSMENT AND SAFETY INSTRUCTIONS</b> .....	<b>5</b>
RISK ASSESSMENT .....	5
SUMMARISED RISK ASSESSMENT: .....	5
RISK AND SAFETY PHRASES .....	6
<b>EQUIPMENT</b> .....	<b>10</b>
APPARATUS .....	10
OTHER EQUIPMENT .....	10
CHEMICALS, REAGENTS AND SOLVENTS .....	11
CHECK SAMPLES .....	11
PREPARATION OF STOCK AND STANDARD SOLUTIONS .....	11
GENERAL INFORMATION .....	11
STANDARD COMPARISONS .....	11
PREPARATION OF STOCK SOLUTIONS .....	12
PREPARATION OF REAGENT SOLUTIONS .....	12
ACTIVATION OF Cd COLUMN .....	15
REACTIVATION OF Cd COLUMN OR ACTIVATION OF NEW Cd COLUMN .....	15
CHECK OF Cd COLUMN REDUCTION CAPACITY .....	15
<b>SAMPLE HANDLING</b> .....	<b>16</b>
SAMPLE STORAGE AND PREPARATION .....	16
SAMPLE AMOUNT .....	16
<b>ANALYSIS</b> .....	<b>16</b>
CALIBRATION AND INSTRUMENT VERIFICATION .....	16

SAMPLE STABILITY.....	17
PROCEDURE FOR ANALYSIS .....	17
GENERAL INFORMATION .....	17
SAMPLE PREPARATION .....	17
<b>DOCUMENTATION .....</b>	<b>17</b>
RAW DATA BINDER.....	17
LOG BOOK.....	17
METHOD BINDER.....	18
<b>DATA.....</b>	<b>18</b>
	18
	18
	19
	19
	19
	19
	20
	20
<b>REVISION HISTORY .....</b>	<b>20</b>
<b>PERSON RESPONSIBLE.....</b>	<b>21</b>
<b>VALIDATION.....</b>	<b>22</b>
VALIDATION REPORT .....	22
SPECIFICITY .....	23
REPEATABILITY .....	25
PRECISION WITHIN THE LABORATORY .....	26
INTERLABORATORY COMPARISONS .....	26
ACCURACY .....	27
BIAS FROM ACCURACY.....	28
EXTRACTION YIELD (RECOVERY).....	29
LIMIT OF DETECTION (LOD) AND LIMIT OF QUANTITATION (LOQ).....	31
LINEARITY .....	31
ROBUSTNESS.....	32
MEASUREMENT RANGE .....	32
MEASUREMENT UNCERTAINTY .....	32
(b) (4) .....	33
<b>CONCLUSION.....</b>	<b>34</b>
<b>APPENDICES .....</b>	<b>34</b>
<b>APPENDIX 1. (b) (4) .....</b>	<b>35</b>
<b>APPENDIX 2. (b) (4) .....</b>	<b>38</b>

## Purpose

To determine the concentration of nitrate and nitrite in tobacco, tobacco products, and tobacco derived products (also called Purified products) with (b) (4).  
(b) (4).

## Applies to

APS

## General information

### Principle of the method

The concentrations of (b) (4) and (b) (4) are measured for the analysis. A calibration is run for each analysis timepoint.

The obtained concentrations are specified after conversion as nitrite and nitrate.


Nitrite/nitrate is extracted from tobacco/tobacco products and Tobacco derived products using (b) (4).

(b) (4)

Number of analyses/person/week is about (b) (4).

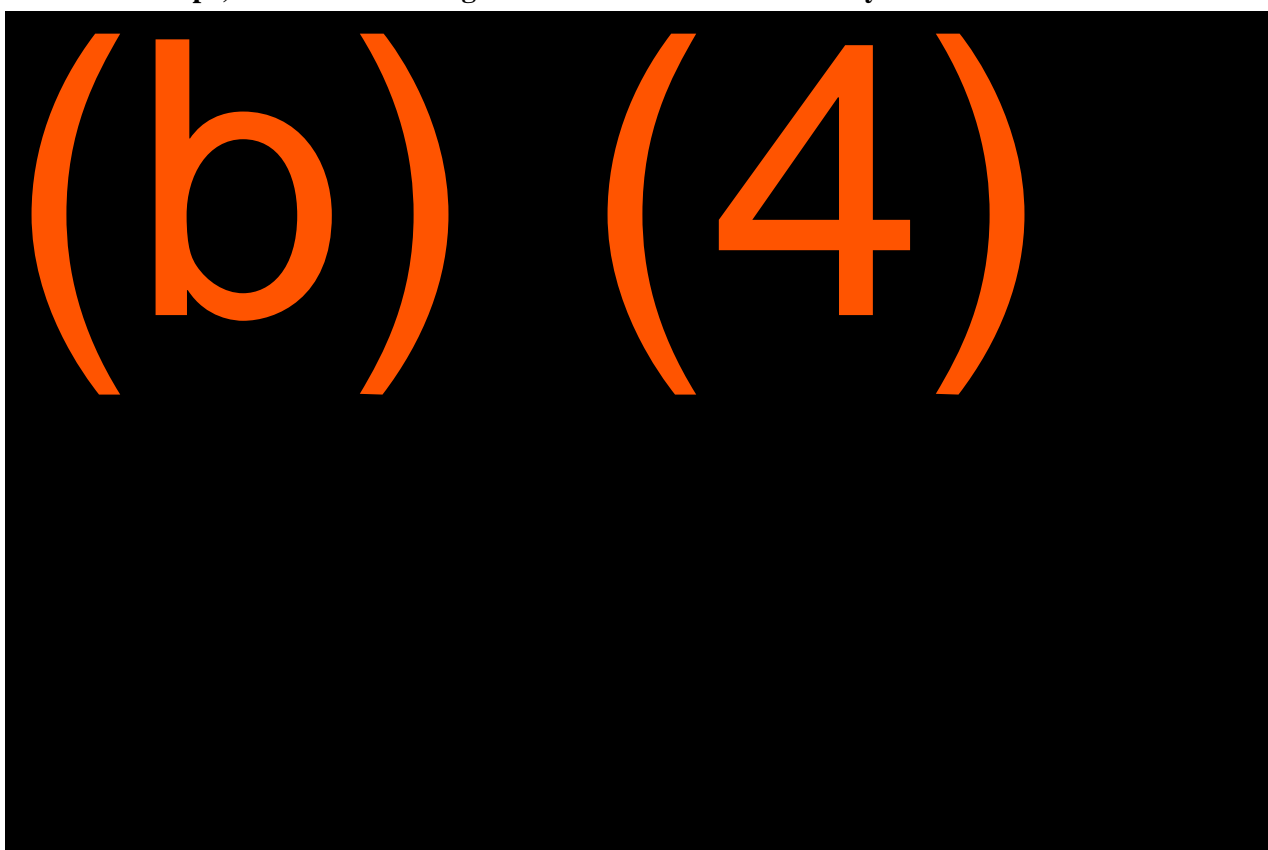
The method has been modified in compliance with ISO standard 13395 (15/07/1996) and CORESTA's recommended method N'36 (January 2015). The differences between the methods are listed in [Table 1](#).

**Table 1. The differences between the methods.**

	ISO standard 13395 (15/07/1996)	CORESTA recommended method N'36 (January 2015)	APS method: Quantitative analysis of nitrite and nitrate with CFA
Analyte			
Reduction of Nitrate to Nitrite			
Buffer solution			
Nitrate			
Gas for segmentation			
Wavelength detector			
Calibration solution			

Note: All reference documents and additional information stated “available upon request” are in Swedish. They are available upon request but need to be translated into English first.

**Method scope, measurement range and measurement uncertainty**



**Table 2. Combined relative measurement uncertainty**

(b) (4)

(b) (4)

**Literature references**

(b) (4)

**Risk assessment and safety instructions**

**Risk assessment**

Summarised risk assessment:

(b) (4)

### Risk and safety phrases

(b) (4)

(b) (4)

(b) (4)



(b) (4)

(b) (4)

## Equipment

### Apparatus

(b) (4)

(b) (4)

### Other equipment

(b) (4)

#### Chemicals, reagents and solvents

(b) (4)

#### Check samples

(b) (4)

#### Preparation of stock and standard solutions

##### General information

(b) (4)

##### Standard comparisons

(b) (4)

*Verification of standard*

(b) (4)

*Preparation of stock solutions*

(b) (4)

Table 3. (b) (4)

(b) (4)

*Preparation of reagent solutions*

(b) (4)

(b) (4)

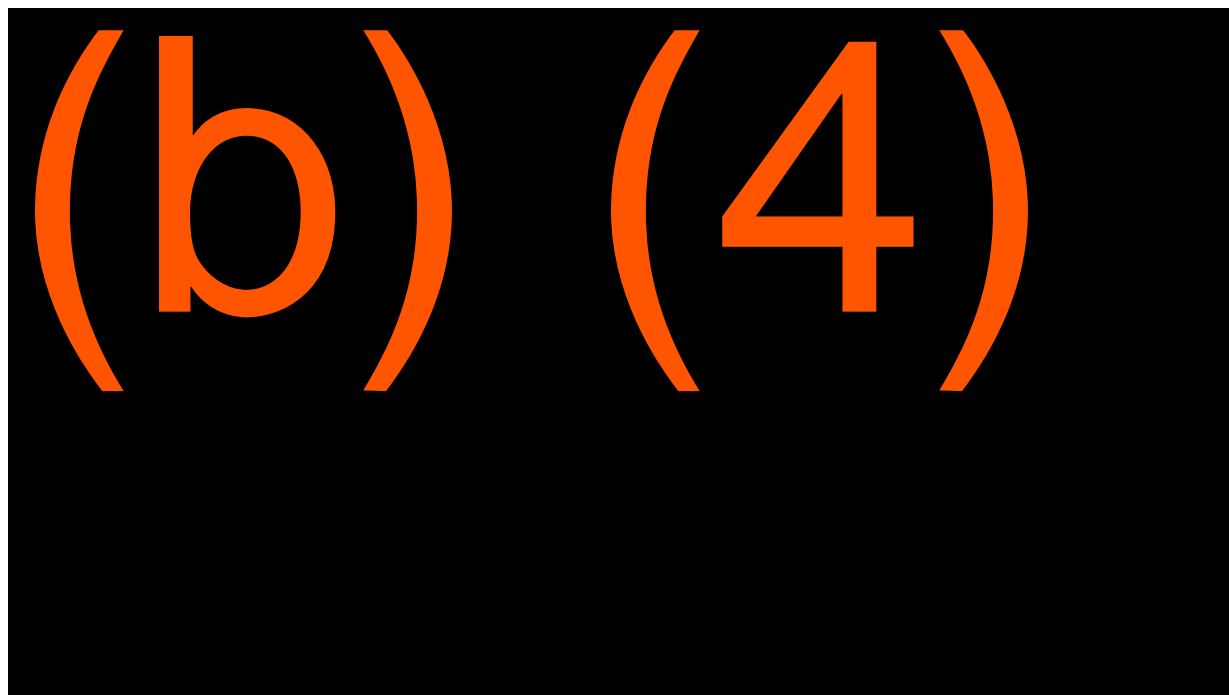
(b) (4)

(b) (4)

(b) (4)

**Activation of Cd column**

(b) (4)



## **Sample handling**

### **Sample storage and preparation**

The sample is stored and prepared in accordance with (b) (4) (available upon request).

### **Sample amount**

The minimum amount of sample for performing an analysis is (b) (4) grams.

## **Analysis**

### **Calibration and instrument verification**





### Sample stability

Prepared sample solution has a shelf life in a refrigerator of (b) (4).

### Procedure for analysis

(b) (4)

### Documentation

#### Raw data binder

(b) (4)

#### Log book

(b) (4)

**Method binder**

(b) (4)

**Data**

(b) (4)

(b) (4)

(b) (4)

**Revision history**

(b) (4)

(b) (4)

**Person responsible**

Director APS



Quality and Environmental  
Management System

Document Title

**Quantitative Analysis of Nitrite/Nitrate**

Part of Process

**Contract Analysis APS**

Document Type

**Method Description**

Valid From

31/05/2018

Page

22(42)

Approved By

(b) (6)

Document Publisher

(b) (6)

## Validation

### Validation report

(b) (4)

### Specificity

(b) (4)

(b) (4)



(b) (4)

(b) (4)

(b) (4)

**Accuracy**

(b) (4)

(b) (4)

**Bias from accuracy**

(b) (4)

**Extraction yield (Recovery)**

(b) (4)

(b) (4)

**Limit of detection (LOD) and limit of quantitation (LOQ)**

(b) (4)

**Linearity**

(b) (4)

(b) (4)

**Robustness**

(b) (4)

**Measurement range**

(b) (4)

**Measurement uncertainty**

(b) (4)



(b) (4)

(b) (4)

#### **Conclusion**

(b) (4)

#### **Appendices**

(b) (4)

Appendix 1. (b) (4)

(b) (4)

(b) (4)

(b) (4)

Appendix 2. (b) (4)

(b) (4)

(b) (4)

(b) (4)



(b) (4)

(b) (4)