ISO/FDIS 21766ISO 21766:2021 (E)

Tobacco and tobacco products — Determination of tobacco-specific nitrosamines in tobacco products — Method using LC-MS/MSTobacco and tobacco products — Determination of tobacco-specific nitrosamines in tobacco products — Method using LC-MS/MS

Contents

Foreword

Introduction

Foreword

Introduction

1 Scope

2 Normative references

3 Terms and definitions

4 Principle

5 Reagents

6 Apparatus

7 Preparation

7.1 Preparation of glassware
7.2 Preparation of solutions
7.2.1 Extraction solution, 100 mM ammonium acetate in water
7.2.2 HPLC Mobile Phase A: Water, resistivity ≥ 18,2 MΩ·cm at 25 °C
7.2.3 HPLC Mobile Phase B: 0,1 % acetic acid in methanol
7.3 Preparation of standards
7.3.1 General
7.3.2 Preparation of internal standard solutions
7.3.2.1 Stock solution
7.3.2.2 Combined secondary internal standard solution
7.3.2.3 Internal standard spiking solution
7.3.3 Preparation of calibration standard solutions
7.3.3.1 Stock solution
7.3.3.2 Mixed TSNA standard solution (I)
7.3.3.3 Mixed TSNA standard solution (II)
7.3.3.4 TSNA calibration standards

8 Sampling

8.1 General
8.2 Sample preparation
8.3 Sample extraction

9 Sample analysis

9.1 General
9.2 Suggested HPLC parameters
9.3 MS/MS parameters
9.3.1 General
9.3.2 Quantification and qualification transitions
9.4 System suitability

9.5 Calibration
9.6 Calculation

10 Repeatability and reproducibility

11 Test report

1 Scope

2 Normative references

3 Terms and definitions

4 Principle

5 Reagents

6 Apparatus

7 Preparation

7.1 Preparation of glassware
7.2 Preparation of solutions
7.2.1 Extraction solution, 100 mM ammonium acetate in water
7.2.2 HPLC Mobile Phase A: Water, resistivity ≥ 18,2 MΩ·cm at 25 °C
7.2.3 HPLC Mobile Phase B: 0,1 % acetic acid in methanol
7.3 Preparation of standards
7.3.1 General
7.3.2 Preparation of internal standard solutions
7.3.2.1 Stock solution
7.3.2.2 Combined secondary internal standard solution
7.3.2.3 Internal standard spiking solution
7.3.3 Preparation of calibration standard solutions
7.3.3.1 Stock solution
7.3.3.2 Mixed TSNA standard solution (I)
7.3.3.3 Mixed TSNA standard solution (II)
7.3.3.4 TSNA calibration standards

8 Sampling

8.1 General
8.2 Sample preparation
8.3 Sample extraction

9 Sample analysis

9.1 General
9.2 Suggested HPLC parameters
9.3 MS/MS parameters
9.3.1 General
9.3.2 Quantification and qualification transitions
9.4 System suitability
9.5 Calibration
9.6 Calculation

10 Repeatability and reproducibility

11 Test report

Annex A (informative) Sample clean-up using solid phase extraction (SPE)

A.1 Reagents and supplies
A.2 Reagent preparation
A.2.1 Wash No. 1: 4,5 % methanol and 0,5 % ammonium hydroxide in water
A.2.2 Wash No. 2: 0,01 % formic acid in water
A.2.3 Eluting solvent: 30 % water and 0,1 % acetic acid in methanol
A.3 SPE procedure

Annex B (informative) Examples of typical chromatograms

Annex A (informative) Sample clean-up using solid phase extraction (SPE)

A.1 Reagents and supplies
A.2 Reagent preparation
A.2.1 Wash No. 1: 4,5 % methanol and 0,5 % ammonium hydroxide in water
A.2.2 Wash No. 2: 0,01 % formic acid in water
A.2.3 Eluting solvent: 30 % water and 0,1 % acetic acid in methanol
A.3 SPE procedure

Annex B (informative) Examples of typical chromatograms

Page count: 0 16