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Note d'application

ACQUITY UPLC™ I-Class with Xevo™ TQ-S micro for Rapid Analysis of 13 N-Nitrosamines

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Abstract

Waters™ ACQUITY UPLC I-Class System with Xevo TQ-S micro was used for the analysis of 13 volatile or nonvolatile N-nitrosamines statutorily required to be detected. The method was characterized by good selectivity, high sensitivity, rapid analysis, and good repeatability, and exhibited excellent response even with oral fluid matrix. We have established a method package containing complete LC conditions and MS conditions in Quanpedia database

Benefits

- ACQUITY UPLC I-Class System ultra performance liquid chromatography is an effective tool for isolating compounds, with increased sample throughput and a reduction of solvent usage due to reduced run times
- Owing to the good sensitivity of Xevo TQ-S micro, the signal-to-noise (S/N) ratio of nitrosamine assay remained excellent even when the sensitivity was 0.1 ng/mL

Introduction

N-nitrosamines are a class of compounds that have been shown to exhibit carcinogenic and mutagenic (or genotoxic) effects in animals at several different tissue sites and by several different routes of exposure. EU Directive 2009/48/EC pertains to the safety of toys, finger paints, and elastomeric materials likely to be placed in the mouth of children. This legislation also limits the cumulative presence of N-nitrosamines to less than 10 µg/kg in total. The legislation applies to any toys or equipment containing elastomeric materials such as soothers and bottle teats aimed at children.

In this application note, Waters™ ACQUITY UPLC I-Class System with Xevo TQ-S micro was used for the analysis of 13 volatile or nonvolatile N-nitrosamines statutorily required to be detected. The method was characterized by good selectivity, high sensitivity, rapid analysis, and good repeatability, and exhibited excellent response even with oral fluid matrix. We have established a method package containing complete LC conditions and MS conditions in Quanpedia database. In real-life applications, the package can be readily imported into the system for direct use.

Experimental

LC Conditions

Run time:	12 min
Column:	ACQUITY UPLC HSS T3 1.7 µm, 2.1 x 100 mm
Column temperature:	40 °C
Mobile phase A:	2 mmol/L ammonium acetate in water
Mobile phase B:	Methanol

Flow rate:	0.4 mL/min

MS Conditions

Ionization parameters and transition pairs were optimized automatically using the IntelliStart™ function of MassLynx Software. In this instance, the automatic tuning parameters feature of IntelliStart was used to determine optimum ionization parameters such as cone voltages for parent ions and collision gas energies for MRM transitions. The resulted method was integrated into Quanpedia for convenient and practical use through just one click.

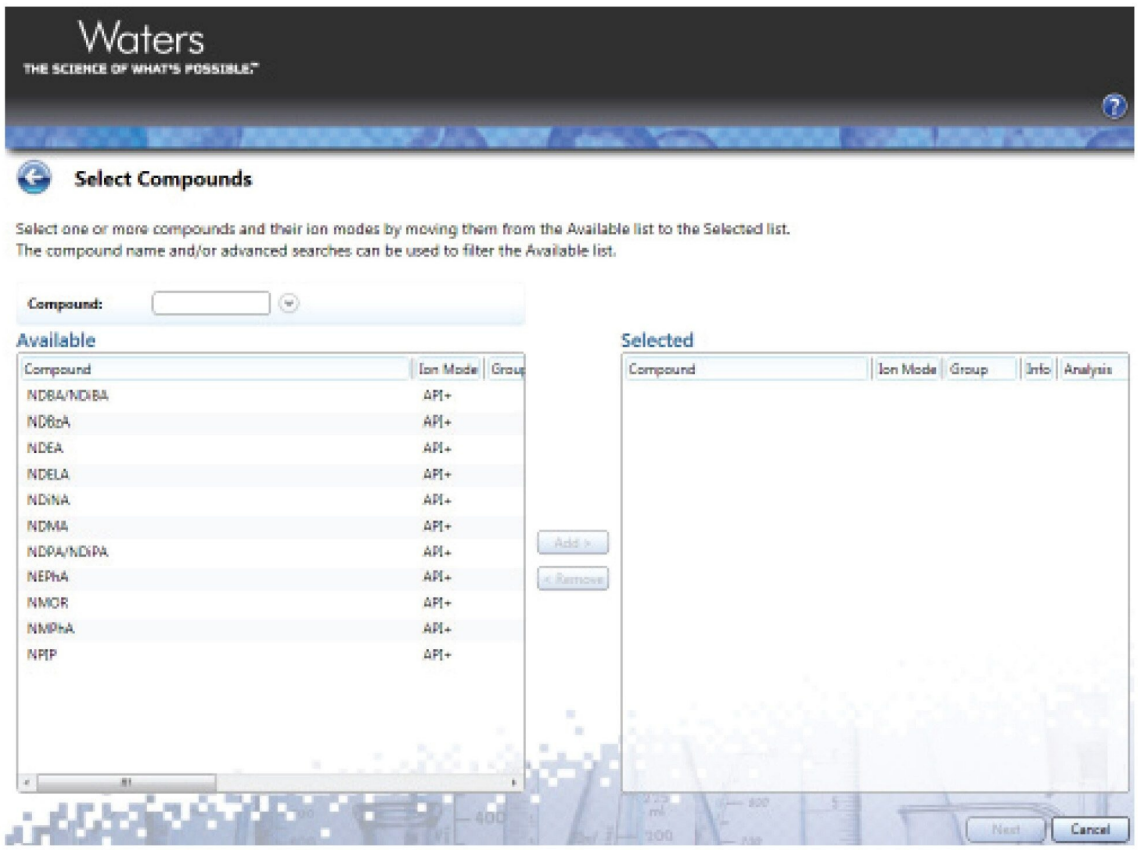

Figure 1. 13 N-nitrosamines monitored in Quanpedia database.

Results and Discussion

Repeatability

The low-dispersion flow paths of ACQUITY UPLC I-Class combined with the rigid, small particle sub-2 µm particle column guaranteed the stability of experimental data. 6 replicate injections were performed, peak areas were calculated by MassLynx Software, and their RSD% were less than 3.0%.

Compound	%RSD (n=6)	Compound	%RSD (n=6)
NDELA	1.37	NMPhA	1.79
NDMA	1.18	NEPhA	1.96
NDEA	0.58	NDBA	1.56
NPIP	1.12	NDiBA	2.12
NMOR	0.77	NDBzA	1.67
NDPA	0.47	NDiNA	1.1
NDiPA	2.19		

Table 1. Repeatability (RSD%) of consecutive injections of 13 N-nitrosamines.

Sensitivity

The sensitivity of 13 N-nitrosamines listed in Table 1 was optimized using MRM method in APCI positive ion mode, and the results are shown in Figure 2 below. When the concentration of target analyte was 0.1 ng/mL, the S/N ratios of 13 N-nitrosamines were still greater than 10.

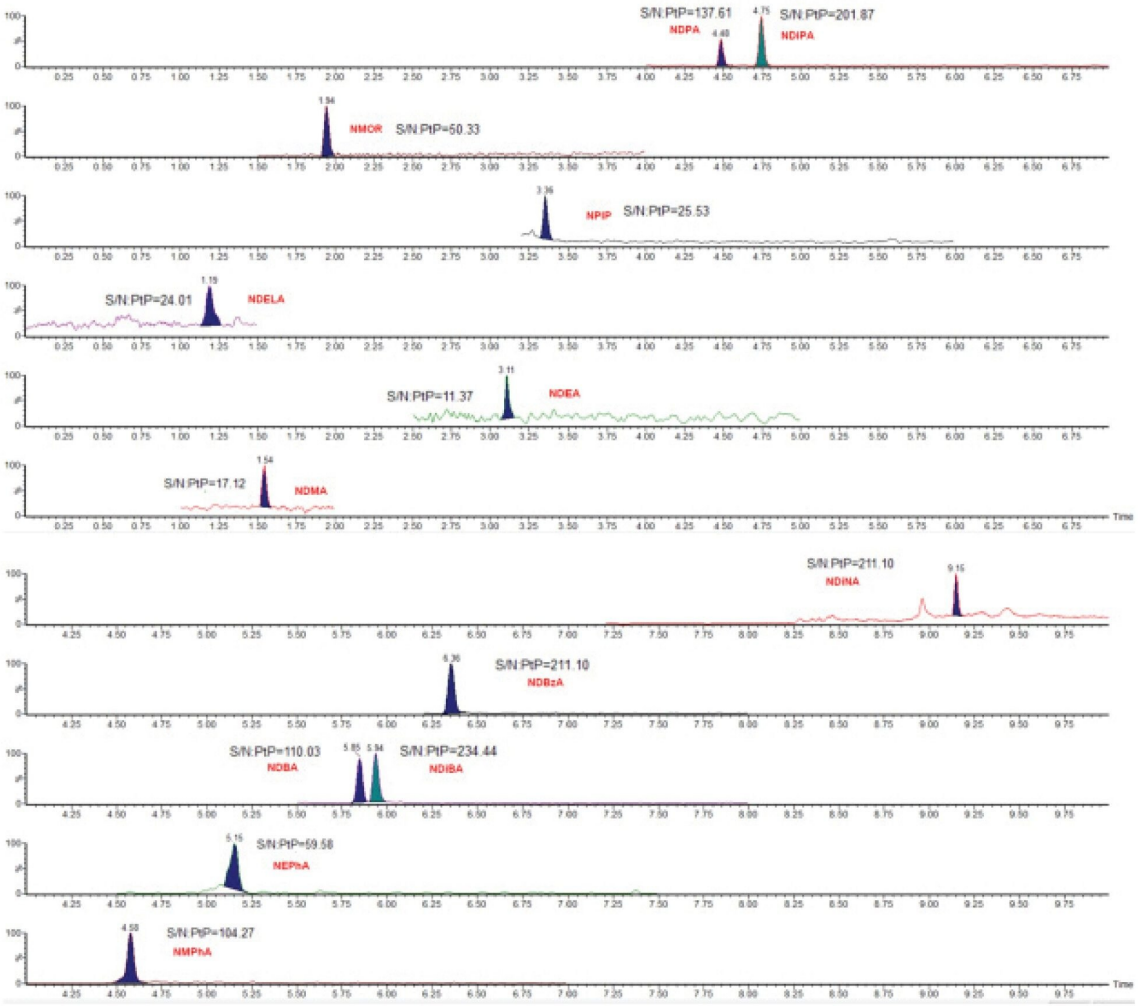

Figure 2. Chromatograms and S/N ratios of 13 N-nitrosamines.

Linearity

Seven-point calibration curves at 0.1 ppb, 0.2 ppb, 0.5 ppb, 1.0 ppb, 5.0 ppb, 10.0 ppb, and 20.0 ppb were established, and for all the compounds under analysis, the correlation coefficient R^2 of the calibration curve was >0.995, indicating good linearity and reliable quantitative results. The calibration curve equation and correlation coefficient R^2 of the 13 compounds are shown in Table 2.

Compound	Linear equation	Correlation coefficient R^2	Compound	Linear equation	Correlation coefficient R^2
NDELA	y=1175x+96	0.996	NMPhA	y=10678x+139	0.999
NDMA	y=311x+4	0.999	NEPhA	y=4282x-153	0.998
NDEA	y=1658x+16	0.999	NDBA	y=8969x-6	0.999
NPIP	y=6829x+104	0.999	NDiBA	y=6608x+38	0.999
NMOR	y=2809x+41	0.999	NDBzA	y=9527x-44	0.998
NDPA	y=10799x+183	0.999	NDiNA	y=573x-120	0.996
NDiPA	y=9471x+88	0.999			

Table 2. Seven-point calibration curve equations and correlation coefficient R^2 of the 13 nitrosamines.

Matrix Effect

According to the regulation, responses of the 13 N-nitrosamines in real simulated oral fluid matrix were studied using this protocol. As shown in Figure 3 below, this method exhibited excellent responses and S/N ratios in matrix environment, indicating good suitability for real sample assays.

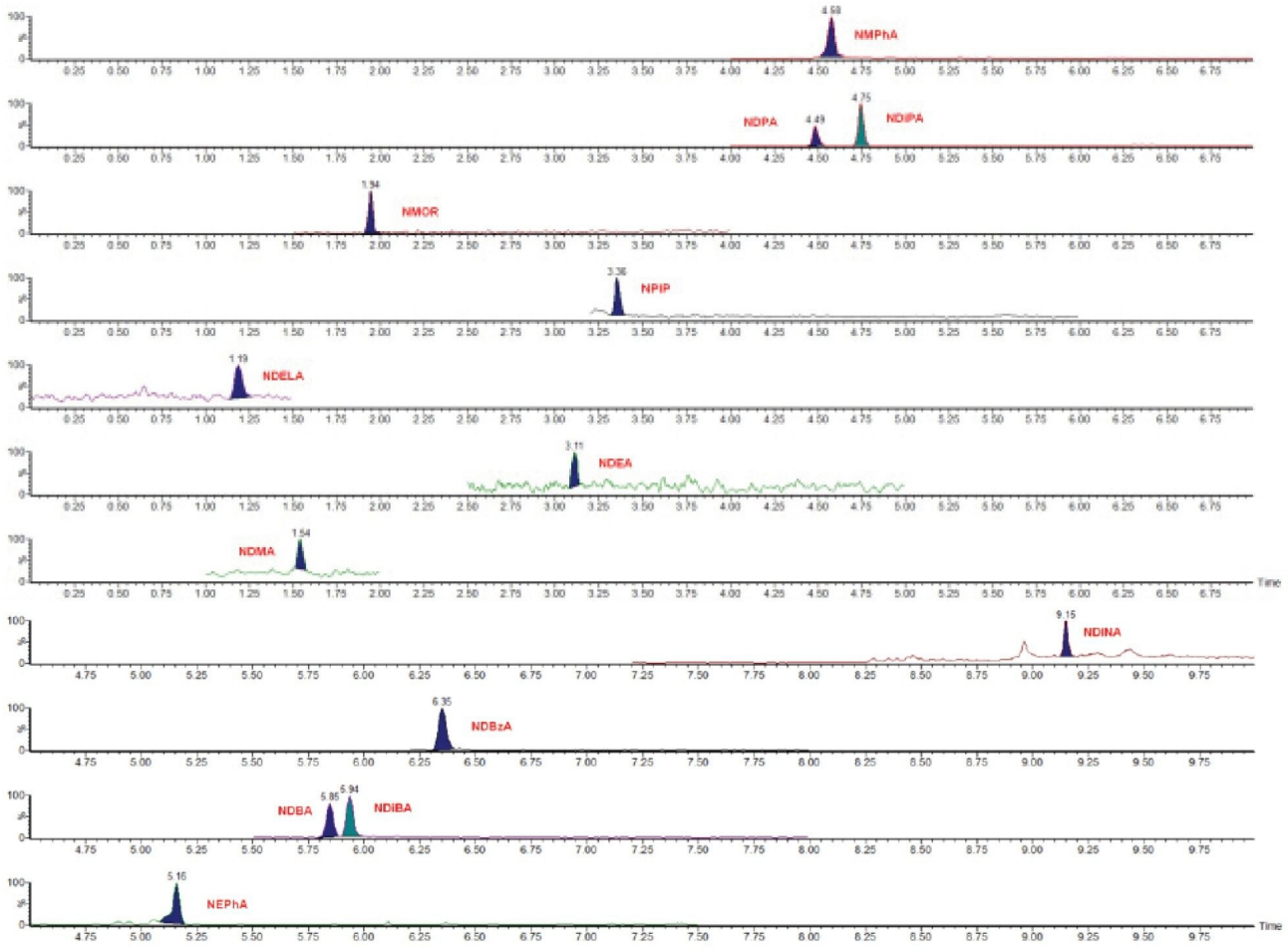

Figure 3. Chromatograms of the 13 N-nitrosamines at 0.1 ng/mL in matrix environment.

Establishment of Quanpedia Database

This method has been added to Quanpedia database and can be directly invoked for use. Quanpedia employs a compound-centric database that may hold all UPLC chromatographic methods, MS acquisition methods, and TargetLynx quantitation information in one place. The users can easily export the LC method and MS method from the database for direct use, or they can add self-developed methods to the database for later use as needed. A brief procedure flowchart is shown in Figure 4:

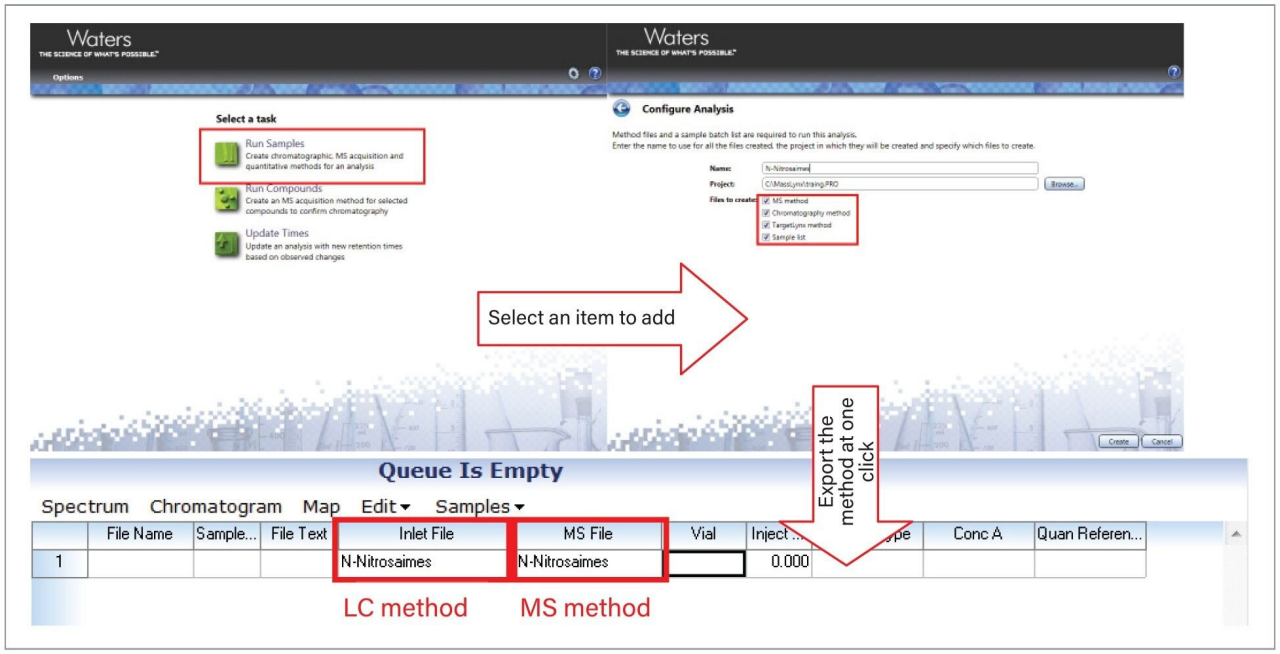

Figure 4. Brief procedure flowchart of Quanpedia database.

Conclusion

An analytical method for 13 N-nitrosamines statutorily required to be detected was established using ACQUITY UPLC I-Class Xevo TQ-S micro System, and the matrix effect was studied using real simulated oral fluid matrix. This method is simple and fast. The Quanpedia method package established in this study can be readily exported at just one click to provide complete LC method and MS method, making it easy to be directly used by the analysts.

Featured Products

ACQUITY UPLC I-Class PLUS System <https://www.waters.com/134613317>

Xevo TQ-S micro Triple Quadrupole Mass Spectrometry <https://www.waters.com/134798856>

MassLynx MS Software <https://www.waters.com/513662>

TargetLynx <https://www.waters.com/513791>

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