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Analysis of nitrogen oxides in Platform 1 Aerosol

PRODUCT TESTING LABORATORY AND GOVERNANCE

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1. ANALYSIS OF NITROGEN OXIDES IN PLATFORM 1 AEROSOL

1.1. Abstract

The objective of this method is to quantify the nitrogen monoxide (NO) and the sum of nitrogen dioxides (NO_2) and nitrogen monoxide (NO_x) in Platform 1 (P1) aerosol. The aerosol is generated on a Cerulean SM450 linear smoking machine and collected using a cambridge filter pad (CFP) for TSNA analysis followed by a Tedlar bag to collect the gases for the nitrogen oxides determination. The Tedlar bags are then connected to an EcoPhysics CLD811 instrument which detect the NO/NO_x by chemiluminescence in ppm. To convert the results into [µg/stick], an equation based on the ideal gas law is used.

1.1. Applicability

The method described is used to determine nitrogen oxides in mainstream aerosol from Platform 1 (P1) under Health Canada (HC) smoking regimen.

1.1. Reagents

- Ethanol for cleaning the filter holders
- Nitrogen quality 50
- 10 and 30 ppm certified nitrogen monoxide standard gases with a relative uncertainty of ≤ 2 %.

1.2. Aerosol generation

P1 items are conditioned in climatized chamber for at least 48 hours at target conditions of 22 ± 1°C and relative humidity of 60 ± 3% before used for aerosol generation.

Cambridge filters are conditioned for at least 12 hours at target conditions of 22 ± 1°C and relative humidity of 60 ± 3% before used for aerosol generation.

The instrument CLD811 is calibrated with pure nitrogen and 30 ppm nitrogen oxide certified gas. The calibration is as well verified with 10 ppm nitrogen oxide certified gas.

The Tedlar bags are purged by alterning pure nitrogen and vacuum before the aerosol collection.

The aerosol samples are generated on a linear smoking machine (Cerulean SM450) under HC smoking regimen and collected using Tedlar bag after passing through a cambridge filter pad retaining the total particulate matter (TPM). The collection conditions are summarized in Table **1**. There is no clearing puff in between the accumulations, but there are 5 clearing puffs at the end of the plan.

At the end of the smoking process, while the Cambridge pad is kept for the TSNA analysis, NO/NO_x are quantified in the gas collected with the Tedlar bags. Four replicates for each sample are generated.

Table 1: Aerosol Collection Condition

Regimen	Accumulation number	Puff number	Regimen Condition [puff volume/Puff duration/Puff Interval] [ml/s/s]
HC	5	12	55/2/30

1.3. Instrumental Conditions

The samples are analyzed by EcoPhysics cheminluminescence detector following tables below:

Table 2: Instrumental conditions for determination of nitrogen oxides

Channel A	NO
Channel B	NO_x
Range	1-40 ppm
Filter	Fast
Compression	5
Purge Time	30 seconds
Measurement Time	60 secondes

1.4. Results processing

The value in ppm is converted in µg/stick by using Equation 1 based on the ideal gas law.

Equation 1 Conversion the results from ppm to µg/stick

$$NO_{\mu g/item} = \frac{M_{NO} \cdot [NO_{ppm}]/acc \cdot (puffs \cdot acc + cl) \cdot v \cdot 273.15 \cdot p}{22.414 \cdot 10^3 \cdot (273.15 + t) \cdot 1013.25}$$

$$NOx_{\mu g/item} = NO_{\mu g/item} + \frac{M_{NO_2} \cdot [NOx_{ppm} - NO_{ppm}]/acc \cdot (puffs \cdot acc + cl) \cdot v \cdot 273.15 \cdot p}{22.414 \cdot 10^3 \cdot (273.15 + t) \cdot 1013.25}$$

$[NO_{ppm}]$: NO concentration read on the analyzer [ppm]
$[NOx_{ppm}]$: NOx concentration read on the analyzer [ppm]
acc : Number of accumulations per replicate
cl : Number of clearing puffs after run
M : Molecular weight (NO: 30.006 g/mol et NO_2: 46.006 g/mol)
p : Atmospheric pressure [hPa]
puffs : Number of puffs per accumulation
t : Laboratory temperature [°C]
v : Puff volume [55 ml]

1.5. Limit of Detection (LOD) / Lower Limit of Quantitation (LLOQ)

The LOD and LLOQ were both calculated in terms of the standard deviation of blank measurement (3 measurements of pure nitrogen on 4 different days).

$$LOD = 3 \times SD_{level1}$$

$$LLOQ = 10 \times SD_{level1}$$

Detailed results are provided in Table 3.

Table 3: Limits of Detection and Quantitation (HC regimens)

Matrix	Compound	HC					
		LOD		LLOQ		ULOQ	
		(ppm)	(µg/stick)*	(ppm)	(µg/stick)*	(ppm)	(µg/stick)*
P1	NO	0.060	0.053	1.00	0.87	40	35
	NO_x	0.100	0.087	1.00	0.87	40	35

ULOQ = upper limit of quantitation (highest calibration level)

* When aerosol collected at 1 atm and 22°C.

1.6. Repeatability limit (r) and Intermediate precision limit (IP)

$$r = 2 \cdot \sqrt{2} \cdot s_r$$

$$IP = 2 \cdot \sqrt{2} \cdot s_{IP}$$

s_r is the standard deviation of repeatability.

s_{IP} is the standard deviation of intermedidate precision.

Repeatability limit and intermediate precision limit are determined during four different days by varying smoking machines and operators.

Table 4: Repeatability r and Intermediate precision IP (HC regimen)

Matrix	**Compound**	**HC**		
		r	**IP**	**Mean**
		(µg/item)	(µg/item)	(µg/item)
P1	NO	0.841	1.189	12.6
	NO_x	0.893	1.025	12.9