

# Analysis of ethylene oxide and vinyl chloride in Platform 1 Aerosol

PRODUCT TESTING LABORATORY AND GOVERNANCE

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## 1. ANALYSIS OF ETHYLENE OXIDE AND VINYL CHLORIDE IN PLATFORM 1 AEROSOL

### 1.1. Abstract

The aerosols are generated on a linear smoking machine and collected using Cambridge filter pad (CFP) followed by two impingers filled with 5 mL of toluene. After the smoking process, the CFP is discarded and the contents of the two impingers are combined and delivered to the analytical laboratory for addition of internal standard and analysis.

The extracts are analyzed by Gas Chromatography with Mass Spectrometry detection (GC-MS) using a column Agilent Pora Plot U 25 m x 0.25 mm ID x 8  $\mu$ m film thickness.

Results are expressed as  $\mu$ g/item for P1.

### 1.1. Applicability

The method described is used to determine ethylene oxide (EO) and vinyl chloride (VC) in aerosol from Platform 1 (P1) under Health Canada (HC) and International Organization for Standardization (ISO) smoking conditions, as well as under alternative smoking regimens.

### 1.1. Reagents

- Toluene
- Ethylene oxide, certified
- Vinyl chloride, certified
- 1-2 propylene oxide-d6, stabilized with hydroquinone

### 1.2. Aerosol generation

P1 items are conditioned in climatized chamber for at least 48 hours at target conditions of  $22 \pm 1^\circ\text{C}$  and relative humidity of  $60 \pm 3\%$  before used for aerosol generation.

Cambridge filters are conditioned for at least 12 hours at target conditions of  $22 \pm 1^\circ\text{C}$  and relative humidity of  $60 \pm 3\%$  before used for aerosol generation.

The aerosol samples are generated on a linear smoking machine under ISO and HC smoking regimens and collected using a Cambridge filter pad and two impingers filled with 5 mL of toluene. The two impingers are immersed into a dry ice-isopropanol bath at  $-75 \pm 3^\circ\text{C}$ . The collection conditions for the different smoking regimes are summarized in [Table 1](#).

At the end of the smoking process, the glass fiber Cambridge filter pad is discarded and the contents of the two impingers are combined and mixed into a Filtrona tube.

Four replicates for each sample are generated. Two blanks are smoked each smoking day to ensure that no contamination is carried out. The first blank is smoked before the first aerosol collection, while the second one is smoked at the end of the smoking day.

**Table 1: Aerosol Collection Condition**

Regimen	Accumulation number	Puff number	Regimen Condition [puff volume/Puff duration/Puff Interval] [ml/s/s]
ISO	7	6	35/2/60
HC	5	12	55/2/30

### **1.3. Samples preparation**

Aerosol extracts generated by the aerosol collection laboratory do not need any further preparation step. Sample vials are prepared by mixing 1000 µL of the aerosol extracts with 100 µL of internal standard solution.

### **1.4. Internal standard solution**

The internal standard solution consists in a solution of deuterated propylene oxide (PO-d6) in toluene.

### **1.5. Calibration solutions preparation**

Individual 25 µg/mL ethylene oxide and 1 µg/mL vinyl chloride are prepared by dilution of the certified reference materials in toluene.

Seven standard (STD) solutions are prepared by dilution of the individual ethylene oxide and vinyl chloride stock solutions in toluene. The ranges of concentrations cover the ranges relevant for analysis and is provided in

**Table 2.**

The corresponding vials are prepared by mixing 1000  $\mu\text{L}$  of standard solution with 100  $\mu\text{L}$  of internal standard solution.

**Table 2:** Typical concentrations of ethylene oxide and vinyl chloride calibration standards

	Ethylene oxide.	Vinyl chloride
Level	( $\mu\text{g/mL}$ )	( $\text{ng/mL}$ )
1	0.05	2
2	0.15	8
3	0.50	15
4	1.00	21
5	2.00	27
6	3.50	34
7	5.00	40

The standard level 5 is also used as quality check.

### **1.6. Instrumental Conditions**

The samples are analyzed by Gas Chromatography (GC) with mass spectrometry detection (MS) following tables below:

**Table 3:** Instrumental conditions for determination of ethylene oxide and vinyl chloride

Column	Agilent Pora Plot U 25 m x 0.25 mm ID x 8 µm film thickness
Oven temperature	45°C, 10 min 4°C/min→95°C, hold time 0 min 15°C/min→135°C, hold time 3 min 30°C/min→190°C, hold time 20 min
Run time	41 min
Injection mode	Splitless, 1 min
Injector temperature	190 °C
Injection volume	2 µL
Carrier gas flow	2.0 mL/min
Gas	Helium
Transfer line temperature	180 °C
Source temperature	180 °C
Quadrupole temperature	150 °C
Detector source	Electron Impact

### 1.7. Testing procedure

The following typical analytical sequence, is used for the determination of ethylene oxide and vinyl chloride:

- Calibration curve (STD 1 to 7)
- Solvent (toluene)
- 2 smoked blanks
- Samples
- After every 5 samples, inject a quality check (STD level 5)
- Always end an analytical sequence with a quality check

### 1.8. Verification of results

#### 1.8.1. Calibration curve verification

A calibration curve is used to quantify the unknown samples using the response ratio of analyte to the internal standard. The peak area ratio is applied to generate the curves. The linear



regression is calculated automatically by the instrument software Mass Hunter. Specific information about regressions are provided in [Table 4](#):

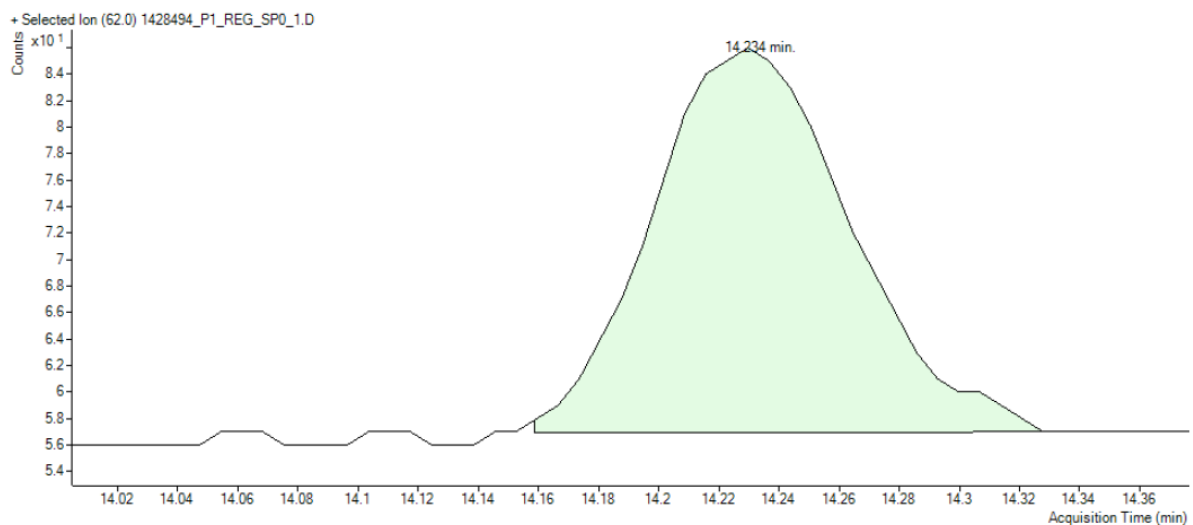
**Table 4:** Parameters used for calibration curves

Compound	Internal standard	Regression type	Weighting factor	R <sup>2</sup>
ethylene oxide (EO)	1,2-propylene oxide-d6	linear	1/x	≥ 0.995
vinyl chloride (VC)	1,2-propylene oxide-d6	linear	1/x	≥ 0.995

### 1.8.2. Quality check

The validity of the calibration is continuously verified during the batch analysis by ensuring that the injected quality check (STD level 5) is within ±15% of its theoretical value for ethylene oxide and ±10% for vinyl chloride.

## 1.9. Example Chromatograms



**Figure 1:** Extracted ion chromatogram for vinyl chloride (62.0 amu) in P1 sample

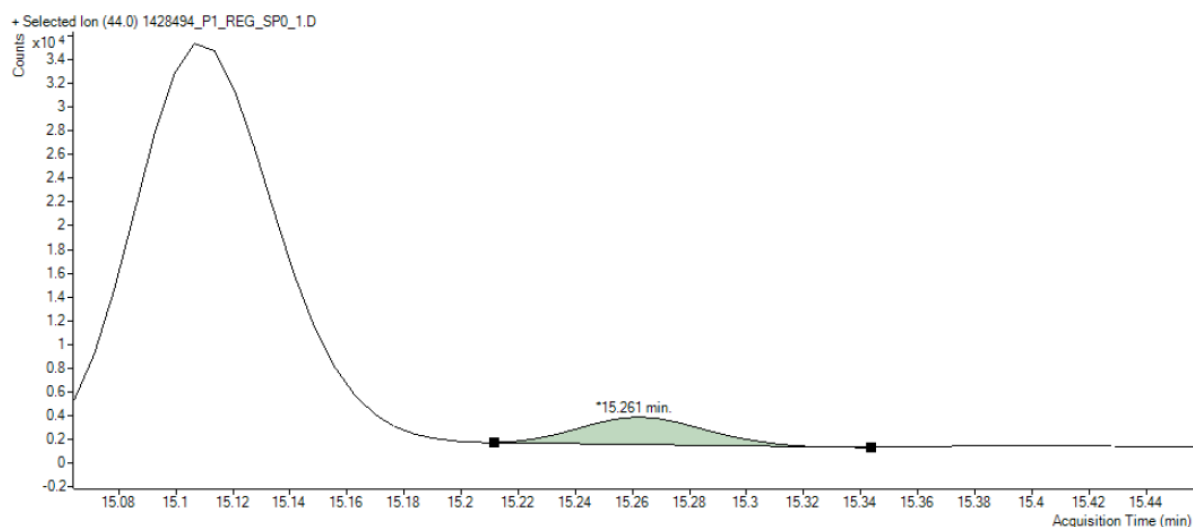


Figure 2: Extracted ion chromatogram for ethylene oxide (44.0 amu) in P1 sample

### 1.10. Limit of Detection (LOD) / Lower Limit of Quantitation (LLOQ)

The LOD and LLOQ were both calculated in terms of the standard deviation of five different measurements of the lowest calibration standard for all other analytes produced under intermediate precision conditions (five different preparations from at least two different operators and analyzed on five different days).

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

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

Level 1 is the lowest calibration level.

Detailed results are provided in [Table 5](#).

Table 5: Limits of Detection and Quantitation (HC and ISO regimens)

Matrix	Compound	HC regimen			ISO regimen		
		LOD [µg/item]	LLOQ [µg /item]	STD1 [µg /item]	LOD [µg/item]	LLOQ [µg /item]	STD1 [µg /item]
P1	ethylene oxide	6.42E-3	2.14E-2	1.00E-1	4.58E-3	1.53E-2	7.14E-2
	vinyl chloride	5.09E-4	1.70E-3	4.00E-3	3.64E-4	1.21E-3	2.86E-3

### 1.11. 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 intermediate precision.

Repeatability limit and intermediate precision limit are determined during four different days using different smoking machines and standard solutions preparation. Different operators are involved in the aerosol generation and analysis.

**Table 6:** Repeatability r and Intermediate precision IP for ISO and HC smoking regimens

Matrix	Compound	HC regimen			ISO regimen		
		r [µg/item]	IP [µg /item]	Mean [µg /item]	r [µg/item]	IP [µg /item]	Mean [µg /item]
P1	ethylene oxide	0.0475	0.0561	0.226	0.0417	0.105	0.192
	vinyl chloride	NA	NA	NA	NA	NA	NA

Repeatability limit r and intermediate precision are not reported at nominal content for vinyl chloride due to its low concentration level at nominal content.

### **1.12. NORMATIVE REFERENCES**

- ISO 3308:2000 – Routine analytical cigarette smoking machine – definitions and standard conditions
- ISO 3402:1999 – Tobacco and tobacco products – atmospheres for conditioning and testing