Are Available Test Methods for the Determination of Carboxyls in Mainstream Cigarette Smoke Fit for the Analysis of Cigars?

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Introduction

In May 2016, the U.S. Food and Drug Administration (FDA) issued a final rule to deem cigars to be subject to the Federal Food, Drug, and Cosmetic Act, as amended by the Family Smoking Prevention and Tobacco Control Act. As part of this regulation, FDA will require manufacturers to report the quantities of Harmful and Potentially Harmful Constituents (HPHCs) in cigar filler and smoke. Standardized methods do exist for the analysis of carboxyls in cigarette smoke such as CORESTA Recommended Method No. 74 (CRM 74), which was based on Health Canada method T-104 and is the basis of ISO/CD 21160:2017. Determination of selected carboxyls in the mainstream smoke of cigarettes – Method using High Performance Liquid Chromatography; however, these methods have not been shown to be fit for purpose for cigar smoke analysis.

Objectives

- Determine if the carbonyl–2,4-dinitrophenylhydrazine derivative (prepared according to CRM 74, T-104, and ISO/CD 21160:2017) is stable for the duration of cigar smoke collection.
- Determine if the instrumental analysis procedure described in CRM 74 is suitable for cigar smoke analysis.

NPHN Stability Experiment

Smoke Machine Parameters

- Smoke regime: 55 mL puff volume, 5 s duration, 30 s interval
- Impinger setup**: Two tandem impingers with 35 mL each of 2,4-dinitrophenylhydrazine (DNPH) trapping solution
- Sampling volume: 1 mL
- Injection volume: 2 µL
- UV Detector: 365 nm

Analytical Method

- Instrument: Waters Acquity UPLC
- Column: Acquity UPLC BEH Shield RP18, 2.1 mm x 100 mm, 1.7 µm particle
- Mobile Phase A: Water (18 MΩ cm)
- Mobile Phase B: Acetonitrile
- Injection volume: 2 µL
- UV Detector: 365 nm

Experimental Design

Two types of cigars were chosen for the study:
1. Black & Mild Straight (traditional dark air cured cigar blend)
2. Black & Mild Wine Plastic tip (flavored cigar blend)

DNPH Stability Results

Results: Black & Mild Straight (traditional dark air cured cigar blend)

- Acetaldehyde yields
  - Time (minutes): 0.00 10:01 9:01 6:01 1:01 0:00
  - Amount (µg/mL): 0.00 0.20 0.40 0.60 0.80 1.00

- Crotonaldehyde yields
  - Time (minutes): 0.00 10:01 9:01 6:01 1:01 0:00
  - Amount (µg/mL): 0.00 0.20 0.40 0.60 0.80 1.00

Conclusions

1. Time studies evaluating the stability of the carbonyl-DNPH derivatives revealed no stability issues over a one-hour period for the two cigar brands tested. This indicates that the sample collection portion of the cigarette methods (CRM 74, T-104, and ISO/CD 21160:2017) may be fit for the collection of cigar smoke.
2. The level of cigar smoke interferences cause a significant bias in the accuracy of target carbonyl compounds by HPLC-UV which is used in CRM74, therefore CRM 74 under current method conditions is not fit for purpose for the analysis of cigars.
3. UPLC-UV offers shorter run times and better selectivity than HPLC-UV, but it also suffers from the same matrix interference issues as the HPLC-UV method.
4. The UPLC-MS method has a shorter run time and minimal matrix interference and may be better suited for the accurate reporting of carbonyl yields in cigar smoke.

References