

TAR AND NICOTINE YIELD VARIATION IN THE SMOKE OF SOME COMMERCIAL CIGARETTE BRANDS

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ABSTRACT

The aim of this study was to evaluate medium-term variations associated with cigarette manufacturing by determining tar, nicotine and carbon monoxide yields in best-selling commercial cigarettes selected to represent a range of design parameters, including cigarette format and nicotine level. Average mainstream smoke yields for twelve commercial brands and the monitor reference cigarette CM 8, smoked under the ISO regimen, were determined using recommended standard analytical methods.

For King size cigarettes, RSD % increased from brands with higher nicotine yields to those with lower nicotine yields, whereas low-nicotine slim cigarettes generally exhibited lower variability in constituent yields compared to King size cigarettes.

Good repeatability for tar, nicotine and carbon monoxide was achieved, confirming the stability of the smoking process and the analytical performance of the reference cigarette CM 8. Overall, the higher variability observed among commercial cigarettes was due to actual differences between individual cigarette batches rather than an inadequate measurement method.

Key words: cigarette; mainstream smoke; nicotine; tar; variation, Pearson correlation.

АПСТРАКТ

Целта на оваа студија беше да се проценат среднорочните варијации поврзани со производството на цигари преку одредување на приносите на катран, никотин и јаглород моноксид. За таа цел беа анализирани најпродаваните комерцијални цигари избрани да претставуваат неколку параметри на дизајнот, вклучувајќи го форматот на цигарата и нивото на никотин.

Просечните приноси во главната струја на чадот од дванаесет комерцијални брендови и референтната цигара CM 8, пушени според ISO режимот, беа одредени со користење на препорачани стандардни методи.

Кај King size цигарите, RSD % се зголемуваше од брендовите со повисоки кон оние со пониски приноси на никотин, додека Slim цигарите со низок принос на никотин покажаа пониска варијабилност во споредба со King size цигарите.

Постигната е добра повторливост на мерењата за катран, никотин и јаглород моноксид, што ја потврдува стабилноста на процесот на пушење и аналитичките перформанси на референтната цигара CM 8. Резултатите укажуваат дека повисоката варијабилност кај комерцијалните цигари произлегува од реални разлики помеѓу поединечните серии, а не од применетиот метод на мерење.

Клучни зборови: цигара; главна струја на чадот; никотин; катран; варијација, Пирсонова корелација

INTRODUCTION

Commercial cigarette smoking is the most common form of tobacco use worldwide. Cigarette consumption in 2025 shows a global decline, with the World Health Organization (WHO) projecting about 15.4% of the world's population smoking, down from 27% in 2000 (Statista, 2025).

Conventional cigarettes burn tobacco and produce smoke that contains a mixture of potentially harmful constituents such as carbon monoxide, nicotine, heavy metals and other constituents. Therefore, regulatory pressure for the control of cigarettes is being reinforced globally.

The constituents of cigarette smoke, nicotine-free dry particulate matter (NFDPM) or “tar”, nicotine and carbon monoxide, are subject to control and monitoring and have received continued attention in recent years, especially when their levels are compared with those in newer tobacco products, such as e-cigarettes and heated tobacco products.

Directive 2001/37/EC of the European Parliament and later Directive 2014/40/EU aim to approximate the laws, regulations and administrative provisions of the Member States concerning the maximum tar, nicotine and carbon monoxide yields of cigarettes; the warnings regarding health and other information to appear on unit packets and measures relating to the ingredients in tobacco products.

Many governmental authorities have introduced regulations on smoke constituents and require manufacturers to print on the pack the yields per cigarette of tar (T), nicotine (N) and carbon monoxide (CO), or to set yield limits that must not be exceeded. A principal requirement for the implementation of regulatory controls is the capability to measure substances of regulatory interest at relevant levels in a reproducible manner.

The Republic of Macedonia is one of the 168

countries worldwide that ratified the World Health Organization Framework Convention on Tobacco Control in 2006. The country followed the recommendations of the Directive and implemented them in the Law on Tobacco and Tobacco Products and its amendments. (Law on Tobacco and Tobacco Products). As regards measurement methods, tests are carried out on the basis of standards specified in the Law on Tobacco, Tobacco Products and Related Products.

Cigarette manufacturers use standardized machine-smoking methods to determine the amounts of tar, nicotine, and carbon monoxide, which are then printed on the packaging. Independent or designated laboratories then conduct specific tests to verify the accuracy of these values. They must be accredited for testing according to the ISO/IEC 17025:2017 standard.

Yields of tar, nicotine and carbon monoxide are measured on standardized smoking machines. The most widely referenced machine-smoking regime (ISO 3308) and measurement methods (ISO 4387, ISO 8454, ISO 3400 and ISO 10315) for these yields have been published by the International Organization for Standardization (ISO) (Wright, 2015).

Many studies have estimated the variability of various smoke analyte measurements within a single laboratory (Rickert and Wright, 2002) and across several laboratories (Purkis and Intorp, 2014; Teillet et al., 2013). These studies, using reference cigarettes, concluded that measurements of tar, nicotine and carbon monoxide are generally variable within and between laboratories, and that measurements are more variable between laboratories than within a single laboratory.

On the other hand, fewer studies document how tar, nicotine and carbon monoxide levels within a given commercial cigarette might vary over time (Eldridge et al., 2015).

Variations in levels can be expected between product batches due to changes in both the tobacco sources for the blend and fluctuations in the cigarette manufacturing process. Design modifications in products also occur from time to time, which may impact emission levels and constituents' yields (Agnew-Heard et al., 2016).

Medium-term variation, in the context of cigarettes, primarily refers to the 3- to 12-month batch-to-batch consistency and variability of cigarette design features and smoke constituent yields within a specific brand. This is a technical term used in the

tobacco industry and by regulatory authorities to monitor product quality and consistency over time.

Given the large number of cigarette brands available on the market that have the same declared nicotine yield, the question arises as to how variations in cigarette design features can actually account for medium-term variations in nicotine, tar, and carbon monoxide. The current study addresses this question using commercially available brands produced by domestic cigarette factories as a case study.

MATERIAL AND METHODS

Cigarettes

The twenty commercial cigarette brands were sampled from samples submitted for official control, from domestic cigarette factories six times every two months from 2024 to 2025, and analyzed for tar, nicotine and carbon monoxide (TNCO) in the Laboratory for quality control of tobacco and tobacco products - L03, Scientific tobacco Institute Prilep, North Macedonia.

The cigarette samples from a single month were generally analyzed in a single analytical run, which included a sample of the reference monitor cigarette CORESTA CM 8 (Körber Technologies GmbH, Germany).

Two formats of cigarettes were pointed out in

the study:

1. King size cigarettes:
Brand A, B, C higher nicotine yields (declared 10-0.8-10 mg/cig. TNCO),
Brand D, E, F low nicotine yields (declared 6-0.5-6 mg/cig. TNCO),
Brand G, H, I very low nicotine yields (declared 4-0.4-4 mg/cig. TNCO)
2. Slim cigarettes:
Brand J, K, L low nicotine yields (declared 6-0.5-6 mg/cig. TNCO).

The design characteristics for the cigarettes tested are summarized in Table 1.

Table 1. Cigarettes characteristics

Characteristic	Range
Filter construction	Filtered
Filter types	Acetate
Filter ventilation	40–56 %
Cigarette length	82–96.7 mm
Cigarette diameter	5.0–7.0 mm
Cigarette paper permeability	65-82 CU
Tobacco weight	0.59–0.86 g/cig.

Analysis of Tar, Nicotine and Carbon Monoxide (TNCO)

Sample cigarettes and the reference monitor cigarette were conditioned as described in ISO 3402 (ISO, 2023). Filter ventilation, circumference, cigarette weight and tobacco weight were measured as basic physical properties.

Cigarettes were smoked in a 20-port rotary smoking machine (RM20CS, Borgwaldt, Hamburg, Germany) as described in detail in ISO 4387 (ISO, 2019), using 20 cigarettes per test in triplicate for each result. The ISO protocol used a 35 ± 0.25 mL puff volume, 2 ± 0.05 s puff duration and 60 ± 0.5 s puff interval (ISO 3308).

Total particulate matter (TPM) was trapped on a glass fiber filter as specified in ISO 4387. The mass of the particulate matter was determined gravimetrically. Nicotine collected on the glass fiber filter was extracted using isopropyl alcohol. After extraction from the filter pads, nicotine yields were determined by UV Vis spectrophotometer (SP-8001 Metertech) as described in ISO 3400 (ISO, 1997). Carbon monoxide was measured in the vapor phase

of the cigarette smoke with a calibrated non-dispersive infrared (NDIR) gas analyzer (C-24, Borgwaldt, Hamburg, Germany) as described in ISO 8454 (ISO, 2024).

The mean values and relative standard deviations (RSD%) of smoke constituents for each cigarette brand were calculated based on six replicate measurements per analytical run. Pearson's correlation coefficients were also computed using the same six averaged data points per run to assess the strength of linear relationships between the investigated parameters. The coefficient of determination is the percentage of the response variable variation that is explained by a linear model. The significance of the association of the sample correlation coefficient r was determined using a p-value.

All statistical analyses were performed using the Data Analysis ToolPak. Finally, mean compound yields of each cigarette brand were compared with values provided by manufacturers on the cigarette packs.

RESULTS AND DISCUSSION

The influence of measurement over the variations in the yields of the constituents in smoke

Occurrence of medium-term variability often leads to systematic differences in the yields of smoke constituents from the same cigarette measured in the same laboratory using the same analytical methods, but at different points in time (3-12 month). It is due to a number of factors, most notably variability in the smoking and analytical process, and variability in the product.

Average mainstream tar, nicotine and carbon monoxide yields for each brand in this study, the monitor reference cigarette CM 8 and the percent relative standard deviation (RSD %) are reported in Table 2. The relative standard

deviations (RSD) of tar, nicotine and carbon monoxide yields are below 5% across all brands and both formats.

For measurements in the King size format, RSD increases from high-nicotine brands (Brand A, B, and C, higher nicotine yields) to low-nicotine brands (Brand G, H, and L, very low nicotine yields).

King size and slim cigarettes differ in physical dimensions (circumference/length), affecting tobacco amount and smoke delivery, but the actual nicotine yield is not always drastically different. Overall, the variation in compounds levels in slim

cigarettes (Brands J, K and L, low nicotine yields) was generally lower compared to King size cigarettes. Batch-to-batch constituent variability is generally greater for

commercial cigarettes manufactured within one year compared to a single batch of CM 8 reference cigarettes.

Table 2 Average tar, nicotine and carbon monoxide yields and relative standard deviation

Cigarette brand	Determined yield (mg/cig.)					
	Tar		Nicotine		Carbon monoxide	
	Mean	RSD %	Mean	RSD %	Mean	RSD %
King size cigarettes						
Brand A	10.90	3.55	0.89	3.84	9.94	2.71
Brand B	11.20	2.22	0.81	3.23	10.32	3.87
Brand C	10.88	2.30	0.88	3.65	10.69	2.15
Brand D	5.90	3.66	0.51	3.98	6.14	2.99
Brand E	6.45	3.37	0.52	3.42	6.38	3.10
Brand F	6.23	2.44	0.49	4.76	6.17	2.08
Brand G	4.04	3.77	0.42	3.89	4.10	3.52
Brand H	4.19	3.80	0.41	4.74	4.18	3.51
Brand I	3.90	3.32	0.39	3.46	4.28	2.49
Slim cigarettes						
Brand J	5.84	1.38	0.51	2.21	5.88	2.89
Brand K	5.72	2.84	0.51	2.53	6.08	2.64
Brand L	6.07	1.98	0.48	3.68	5.89	2.52
Reference monitor cigarette						
CM8	14.21	0.77	1.64	0.68	13.51	0.72

ISO standard 8243 provides confidence intervals for sampling over a period of time which have been estimated for tar and nicotine at a level of 95%, and ± 20 % for carbon monoxide. Sampling over a period of time is used for ongoing appraisals of a product over a longer duration, such as several months, and is recommended wherever possible due to variations in manufacturing.

Intra-laboratory repeatability

Cigarettes CM 8 were the reference monitor cigarettes used to check the stability of the measurement and intra-laboratory measurement repeatability.

The average values of tar and nicotine for the reference cigarette CM 8 smoked under the

The differences between the average yields of tar, nicotine and carbon monoxide for each brand in this study and the on-package declared values were compared using the confidence intervals over a period of time. It can be concluded that across all brands and formats, there were no differences within the range of the confidence intervals for tar, nicotine and carbon monoxide.

ISO smoking regime corresponded to the CORESTA certificate content. Moreover, the relative standard deviations (RSD %) of tar and nicotine in reference cigarettes were $< 1\%$ and were statistically lower than the values for commercially available cigarettes

($p < 0.05$).

To assess the variability of the analytical method within the laboratory, an intra-laboratory analysis was performed, which refers to the evaluation of the repeatability of a reference material (CM 8). To calculate the intra-laboratory variability, the RSD of tar, nicotine and carbon monoxide were taken. Intra-laboratory analysis refers to the evaluation of the repeatability of a reference

material (CM 8) and is used to assess the variability of an analytical method within a laboratory.

The overall intra-laboratory repeatability for tar was 0.158, for nicotine 0.353 and for carbon monoxide 0.173. Good repeatability for tar, nicotine, and carbon monoxide was demonstrated, as shown by comparison with the reference cigarette CM 8, confirming the stability of the smoking regime.

Pearson Correlation

Nicotine in cigarette smoke is generally related to tar yield in commercial cigarettes in a roughly linear manner (St. Charles et al., 2011). We used the Pearson correlation to measure the strength and direction of the linear relationship between these two constituents. Because of the small sample size (we only have 6 data points), there was a small chance that our data points would fall

in such a way that it appeared a correlation existed, even when it did not. In this case, we needed to consider whether the correlation was statistically significant and calculate the p-value alongside the Pearson coefficient.

Pearson's correlation coefficients relating tar (x) and nicotine (y) and p-values in the smoke for the twenty brands and CM 8 are shown in Table 3.

Table 3. Pearson's correlation coefficient (r) between tar and nicotine (Bold faced correlation coefficients are large positive relationship between x and y)

Cigarette brand	r	p -value
King size cigarettes		
Brand A	0.0783	0.8828
Brand B	0.1337	0.8006
Brand C	0.2918	0.5747
Brand D	0.1911	0.7168
Brand E	0.7234	0.1042
Brand F	-0.0991	0.8518
Brand G	0.7441	0.0899
Brand H	0.5816	0.2259
Brand I	-0.2203	0.6749
Slim cigarettes		
Brand J	0.1939	0.7127
Brand K	0.6835	0.1787
Brand L	-0.0157	0.9765
Reference monitor cigarette		
CM8	0.2243	0.6691

The number of brands with Pearson's correlation coefficients below $r = \pm 0.7$ between tar and nicotine was ten, as shown in Table 3. This means that most brands exhibited a moderate correlation. Only two brands had Pearson's correlation coefficients above 0.7.

The number of brands with positive Pearson's correlation coefficients was nine, and three had negative coefficients. A positive value indicates a positive relationship, where both variables increase together. A negative value indicates a negative relationship, where one variable increases as the other decreases.

By measuring the components in cigarette samples, we attempted to determine the probability that the observed difference between the measured variables (tar and nicotine) is due to chance, or to the existence of a real difference in the population. This probability is expressed by a *p*-value.

Regarding significance, all brands showed *p*-values higher than 0.05, meaning that the

correlations were not statistically significant. Differences in the nicotine yield that are statistically significant at a Type-I error rate of 5% may be observed and indicate that an effect exists, even though it may not actually be true.

The complex manufacturing process involves tolerances and slight fluctuations in process parameters, such as filter ventilation, paper porosity, or the inclusion of expanded tobacco, can impact the final smoke constituent yields.

In practice, it would be advantageous for the laboratory to control for variations in nicotine content between individual batches.

This study has some limitations, particularly in that it is not a fully comprehensive analysis of the Macedonian cigarette market. Cigarettes manufactured by domestic cigarette factories were analyzed, but imported cigarettes were included. Also, the small number of brands tested limited the power of the statistical analyses.

CONCLUSIONS

There is an expected variation in cigarette constituents between different manufacturing batches, although the variation for tar, nicotine and carbon monoxide within a specific brand is generally low. The percent of relative standard deviations (RSD) of tar, nicotine and carbon monoxide yields was below 5%, indicating low variability between analytical runs in cigarette testing over the specified time period of one year.

Greater variability was observed for commercial cigarettes collected over one year compared to the reference monitor

cigarette CM 8, which was manufactured at once.

Good repeatability for tar, nicotine and carbon monoxide was established, confirming the high accuracy of the reference monitor cigarette CM 8 and the smoking process.

These findings suggest that the higher variability in tar and nicotine yields between commercial cigarettes was due to actual differences between single cigarette, rather than method measurement uncertainties.

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