





## Defining metrological characteristics of the method for measuring nitrogen oxides (no, nox) contents in gaseous phase of heated tobacco products

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**Abstract.** Results of international comparative interlaboratory tests on defining contents of nitrogen oxides (NO, NO<sub>x</sub>) in gaseous phase of aerosol of heated tobacco products are presented in the article. The objects of study were samples of heated tobacco intended for use with electric tobacco heating systems of the IQOS, Glo and Ploom brands. The determination of nitrogen oxides in the gas phase of aerosol from heated tobacco products was carried out using a technique developed in the laboratory of chemistry and quality control of the Federal State Budget Scientific Research Institute "All Russian research institute of tobacco, makhorka and tobacco products". The aim of interlaboratory comparative tests is calculation of intra- and interlaboratory variability (repeatability and reproducibility). Mandel h and k statistics were utilized for statistical compatibility analysis. Analysis of interlaboratory compatibility was carried by z - index, statistical identifying of outliers was carried by Cochran's and Grubbs' criteria. Also, repeatability and reproducibility were calculated. Statistical analysis of interlaboratory comparative tests results proved high level of validity of obtained data. Establishing metrological characteristics is a key aspect of ensuring the reliability and applicability of the methodology. Interlaboratory comparative tests for nitrogen oxides defining are the first time carried in Russia and are actual for innovative products from heated tobacco.

**Keywords:** nitrogen oxides, nicotine containing products, heated tobacco, electric systems for heating tobacco, aerosol, repeatability, reproducibility.

### Introduction

Popularity of innovative types of nicotine containing products (NCP) including electric systems for heating tobacco (eSHT) is increasing during last years. Electrically heated tobacco products have become increasingly popular since the introduction of the Tobacco Heating System 2.2 (THS 2.2, marketed as IQOS<sup>®</sup>) on the market in 2014. Previous studies have shown that the environmental aerosols emitted by electrically heated tobacco products have a substantially lower impact on indoor environments than the environmental tobacco smoke generated from cigarettes [1-11]. eSHT are utilized for inhaling nicotine containing aerosol, which consists of liquid particles dispensed in gaseous phase, formed by heating tobacco without its burning and smoldering. Heated tobacco products are declared in the market as safer alternative to smoking traditional cigarettes due to absence of burning or smoldering processes [12].

Nitrogen oxide and more dangerous dioxide contains in the smoke of tobacco products and in gaseous phase of heated tobacco products' aerosol. Nitrogen oxides are classified as substances 2 class (NO<sub>x</sub>) and class 3 (NO) toxic hazard, as negatively

affecting the body a person, causing damage to the lungs. Airway intake of nitrogen oxides enhances the absorption of nicotine [13, 14].

As eSHT is a new type of product, standard methods for defining nitrogen oxides contents in their aerosol are not approved.

Method for defining carbon monoxide (CO) in gaseous phase of nicotine containing aerosol is presented in paragraph 6 GOST R 57458–2017 [15] which content is an indicator proving absence of burning and smoldering processes. Except for carbon monoxide method, elaborated in chemistry and quality control laboratory of Federal State Budget Scientific Research Institute "All Russian research institute of tobacco, makhorka and tobacco products", for defining nitrogen oxides (NO, NO<sub>x</sub>) contents in gaseous phase of aerosol of heated tobacco products can be utilized as extra proof of absence of these processes. Interlaboratory comparative tests (ICT) were carried for defining metrological characteristics of this method.

Federal State Budget Scientific Research Institute "All Russian research institute of tobacco, makhorka and tobacco products" was the coordinator of these ICT.

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Aim of interlaboratory comparative tests was defining nitrogen oxides NO, NO<sub>x</sub> contents, and also defining intra – and interlaboratory variability for defining nitrogen oxides (NO, NO<sub>x</sub>) contents in gaseous phase of aerosol.

Five laboratories: Russia, Germany, Austria, Italy, Switzerland took part in comparative tests for defining NO, NO<sub>x</sub> in heated tobacco. All laboratories carried tests according to regulative documents for carrying tests.

### Materials and Methods

Three types of electric systems for heating tobacco (Ploom, model S; IQOS, model 2.4; Glo Hyper, model G401) and six samples of heated tobacco were utilized in the tests. Each participant of tests received 2 blocks (400 articles of heated tobacco) and seven devices ESHT of each trademark. Each sample received its own code (A, B, C, D, E, F).

Conditioning samples of heated tobacco was carried directly in consumers' pack (not less than 48 hours, but not more than 10 days), according to GOST ISO 3402–2003 [16].

Samples that were taken from open pack were stored not more than 4 hours in closed containers. After expiring this time samples of heated tobacco were not utilized [17].

Ambient air characteristics during conditioning and testing were maintained according to standard [16].

Protocol for collection aerosol was utilized according to ISO 20778:2018 standard [18].

Collection gaseous phase of aerosols of studied samples in testing laboratories was carried by linear smoking machines "Cerulean SM 450" and "Borgwaldt LM 20E".

### Results

Presence of incompatible with all others results (outliers) was utilized for defining precision values (repeatability and reproducibility) for the method of defining nitrogen oxides (NO, NO<sub>x</sub>) in gaseous phase of heated tobacco. Decisions for excluding results were made after carrying analysis of intra – and interlaboratory compatibility and statistical testing of outliers.

Mandel's h and k statistics were utilized for graphic analysis of compatibility, which allow defining intralaboratory (statistics k) and interlaboratory (statistics h) compatibilities. Mandel's statistics were carried for each laboratory and sample of heated tobacco under GOST R ISO 5725–2–2002 [19]. Results analysis indicates that values from different laboratories differs, but not exceeds 1% level of significance for h statistics. However exceeding 5% level of significance for h statistics for nitrogen oxide NO<sub>x</sub> defining was noticed for laboratories

"a" (samples C, D, F) and "b" (samples A and B). Exceeding critical level for k statistics for nitrogen oxide NO<sub>x</sub> defining was noticed for laboratory "c" for sample D. Significant variance of intralaboratory results on nitrogen oxide NO defining was noticed for laboratory "c" (sample D), on nitrogen oxide NO<sub>x</sub> defining – for laboratories "b" (sample A) and "c" (sample C).

Interlaboratory results compatibility was defined by using Z-index, which was calculated for each defined value and for each laboratory (R 50.2.011, 2005) [20].

Diagrams analysis indicates that value of Z-index for all participants who defined nitrogen oxides not exceeds 2. In this case results prove satisfactory quality of ICT carried for defined values.

Statistic quality evaluation of experimental data was carried by Cochran's and Grubbs' criteria, according to GOST R ISO 5725–2–2002 [19]. Also harmonized flow-chart for exclusion abnormal results recommended by International Union of Pure and Applied Chemistry (IUPAC) was utilized [21].

Comparison of results for Cochran's and Grubbs' criteria for all defined values for heated tobacco products allows identifying suspicious (quasi outliers) and abnormal (statistical outliers) results. According to data analysis abnormal results are absent. Quasi outlier for nitrogen oxide NO<sub>x</sub> defining under Cochran's criterion was noticed only for sample D (laboratory "c") and under Grubbs' criterion – for two minimum extreme values (laboratories "a" and "f").

Next stage of carried tests analysis is calculation repeatability (r) and reproducibility (R) for each defined parameter. Calculated, according to (GOST R ISO 5725–2–2002), values of general mean values, repeatability and reproducibility for each component of aerosol of heated tobacco and for each sample are presented in tables 1–2. Comparison of mean values of defined parameters (nitrogen oxides) with r and R values allowed calculation empirical equations and defining correlation coefficients.

Table 1.

Nitrogen oxide NO,  $\mu\text{g} / 100 \text{ cm}^3$

Sample code	Mean value m,	Repeatability (r)	Reproducibility (R)
A	0.495	0.045	0.150
B	0.469	0.032	0.117
C	1.875	0.172	0.565
D	1.745	0.174	0.488
E	2.962	0.309	1.067
F	2.763	0.305	0.953

Source: Compiled by authors

Table 2.  
Nitrogen oxide NO<sub>x</sub>, µg / 100 cm<sup>3</sup>

Sample code	Mean value m,	Repeatability (r)	Reproducibility (R)
A	0.565	0.042	0.314
B	0.539	0.030	0.284
C	2.073	0.196	0.266
D	1.918	0.170	0.245
E	3.459	0.319	0.664
F	3.192	0.281	0.623

Source: Compiled by authors

It was discovered linear correlation with high correlation coefficients between repeatability and reproducibility and mean value of nitrogen oxides (NO, NO<sub>x</sub>) contents in gaseous phase of aerosol of heated tobacco.

### Discussion

Carrying calculations of metrological characteristics (repeatability and reproducibility) of method for defining nitrogen oxides (NO, NO<sub>x</sub>) in gaseous phase of aerosol of heated tobacco was the aim of the research.

Statistical analysis of defined data noticed absence of statistical outliers for defined values.

It was discovered, that increasing of mean value leads to repeatability and reproducibility increasing.

At present moment, there is no information about carrying such interlaboratory comparative tests for defining nitrogen oxides contents in aerosol of heated tobacco.

### Conclusion

As the result of carried ICT metrological characteristics for the method of defining nitrogen oxides in gaseous phase of aerosol of heated tobacco products were obtained. This proves satisfactory quality of participating laboratories. The obtained values will be utilized for evaluation intra – and interlaboratory convergence of results on defining nitrogen oxide NO and nitrogen oxides NO<sub>x</sub> in gaseous phase of aerosol of heated tobacco products.

Work for including method of defining nitrogen oxides (NO, NO<sub>x</sub>) in gaseous phase of heated tobacco products into GOST R 57458–2017 “Heated tobacco. General technical specifications” for proving absence of burning or smoldering process during utilizing ESHT is now in process.

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#### Conflict of interest

The authors declare no conflict of interest.

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## Установление метрологических характеристик метода при определении оксидов азота (но, нох) в газовой фазе аэрозоля изделий из табака нагреваемого

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**Аннотация.** В статье приводятся результаты проведения международных межлабораторных сравнительных испытаний изделий из табака нагреваемого по определению оксидов азота (NO, NOx) в газовой фазе аэрозоля. Объектами изучения являлись образцы табака нагреваемого, предназначенного для использования с электрическими системами нагревания табака торговых марок IQOS, Glo и Ploom. Определение оксидов азота в газовой фазе аэрозоля изделий из табака нагреваемого проводили с помощью методики, разработанной в лаборатории химии и контроля качества Федерального государственного бюджетного научно-исследовательского учреждения "Всероссийский научно-исследовательский институт табака, махорки и табачных изделий". Цель межлабораторных сравнительных испытаний – расчет внутри – и межлабораторной вариальности (повторяемости и воспроизводимости). При статистической обработке результатов использовали анализ совместимости по статистикам Манделя h и k, анализ межлабораторной совместимости с помощью z – индекса, статистическое тестирование выбросов по критериям Кохрена и Граббса, и провели расчет повторяемости и воспроизводимости. Статистическая обработка результатов межлабораторных сравнительных испытаний подтвердила высокий уровень достоверности полученных данных. Установление метрологических характеристик является ключевым аспектом обеспечения надежности и применимости методики. Межлабораторные сравнительные испытания по определению оксидов азота были проведены впервые в России и являются актуальными для инновационной продукции изделий из табака нагреваемого.

**Ключевые слова:** оксиды азота, никотинсодержащая продукция, табак нагреваемый, электрические системы нагревания табака, аэрозоль, повторяемость, воспроизводимость.

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