Uncertainty of PM_{10} concentration measurement on the example of an optical measuring device

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Abstract. The interest in the natural environment, in particular the ambient air, has been growing. For this reason, growing interest in measuring of concentration of pollutants in the air. To the reduction of costs and time of obtained results, increasingly are used non-reference, alternative methods and devices. However, it is necessary to ensure equivalence of results obtained by alternative methods with the results of the reference method.
One of the equivalence issues is the assessment of the measurements uncertainty of the alternative method in relation to the reference method. This uncertainty is understood as the probability of obtaining a measurement result which differs from the real (reference) result by a predetermined value. Uncertainty can be caused by many reasons: imperfect implementation of the definition of the measurand, incomplete knowledge of the influence of external factors, heterogeneity of the analyzed phenomenon, errors of the measuring device and others. The paper presents methods for testing the uncertainty of measurements used in the study of equivalence and also proposed extending the test procedure with equivalence models, different from the linear regression models.

1 Introduction

The interest in the quality of the natural environment, in particular the surrounding air, is constantly growing. Information on this subject is interested in government institutions, local governments, but also ordinary people are increasingly looking for such information. They want to know what the state of air is, whether it improves or worsens, which affects the low air quality and whether it can counteract it. Is the air safe for them? They are interested in the influence of the presence of pollutants on the quality of life, health or well-being. One of the most commonly described in the context of air pollution is particulate matter (PM). They may be of anthropogenic origin, like fuel combustion, communication emissions, industrial emissions or natural (arising from erosion, evaporation or natural physicochemical transformations) [1-5]. The chemical composition of the particulate matter can vary greatly and depends largely on where it is located. Their impact on health and life is described in the literature. Many publications indicate the impact of high concentrations of particular matter

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on the increase in the incidence of lung and upper respiratory tract illness, heart disease, allergy and general malaise. They also indicate an increased mortality caused by a very poor air condition [6-8].

To answer these questions, we need to effectively and correctly measure airborne contaminants. Both their concentrations and their composition. We want to get precise, place-specific and short-time information. The measurements include new locations, the density of the measurement points is increasing. The measurement results are not only found in scientific publications but are also easily available online on the Internet and on television. Of course, this is done with the emphasis on reducing the costs of such activities.

The best and proper method of measuring the concentration of particulate matter is the reference gravimetric method, also called the manual method. It is used wherever the measurement precision is of the utmost importance. Unfortunately, it is an expensive and quite complicated method. For this reason, it can only be used to a limited extent by professional institutions. Currently, in Poland, there are approximately 180 PM_{10} measurement points using the gravimetric method and approximately 70 samplers of $PM_{2,5}$. Most likely, their number will not change significantly in the near future [9-11].

An alternative to the reference method is use of devices using other methods of measuring the concentrations. These devices can use light scattering (optical method), a tapered element oscillating microbalance (TEOM), or method based on absorption of beta radiation. They are usually much smaller, cheaper and don't require expensive and frequent service [12, 13]. The use of alternative methods for measuring concentrations may result in that there is the possibility of obtaining different results than using the reference method. In the case of different measurement methods, it is not possible to obtain identical results, because the measurement process is influenced by many factors that affect different methods in different ways. This applies to every measurement process. In this situation, it becomes crucial to demonstrate that the alternative method of measuring the concentrations of particulate matter produces results similar to the results of the reference method. This process is called the equivalence test of devices. Obtaining a confirmation of equivalence guarantees that the results of the alternative (candidate) method are really close to the reference values [13-15].

According to the methodology contained in the "*Guide to the demonstration of equivalence…*" the test of equivalence consists in showing that in different weather conditions, two devices give similar results, and large differences are rare and not systematic. Therefore, it is required that measurements are made in minimum 4 comparisons containing dozens of observations (at least 40 correct observations), at different times of the year and in different locations (minimum 2 locations). Measurements are made by the reference method (RM), and at least two candidate devices (CM), working exactly under the same conditions, i.e. located in close proximity, face-to-face. The data obtained in this way will be analyzed in terms of the size and nature of the differences between measurements. The most important concept in this analysis is measurement uncertainty [16].

2 Uncertainty of PM concentrations measurement

The uncertainty of measurements is a concept that occurs practically in every activity related to the measurement of any values, both physical and any other. It results from the lack of certainty regarding the accuracy of measurements, the equality of results obtained in the measurement with real values.

Measurement uncertainty is a non-negative parameter related to the measurement result, characterizing the dispersion of values that can be reasonably attributed to the actual value [17]. It can be understood as the interval in which measured values are found with satisfactory probability [18]. The occurrence of uncertainty of measurements can have many reasons. The measurement uncertainty may be influenced by: inaccuracy or inadequate accuracy class of

the measuring device, imperfect of the observer's senses, inexact or incomplete definition of the measured value, incomplete implementation of the definition of measured value, ignorance of the impact of the environment and its changes on measurement, inaccurate data values from external sources and many other. The multiplicity of causes indicates that the measurement is always inaccurate and in each case there will be measurement uncertainties.

When measuring concentrations by various methods, the uncertainty may be caused by errors in the functioning of both devices (tested device and reference device), the influence of the environment, different aggregation of results, or various implementations of the definition of measured pollution in devices. The dimension of uncertainty is of great importance in evaluation of the obtained measurement results. In extreme cases, with high uncertainty, individual results may be unreliable, which may result in the decision of the necessity to improve functioning of the method or its unsuitability.

The aim of the analysis of the uncertainty of the candidate device in relation to the reference method is to establish the limits of confidence intervals in which the results obtained with the candidate method will be found with determined probability. Determining the chance that the results obtained by the candidate method would not differ significantly from the results obtained using the reference method. If the probability obtained in this way is higher than assumed for a given confidence interval, or with a predetermined probability, the interval containing results will have a smaller spread from the postulated W_{acc}, the candidate method can be considered as the equivalent reference method. Of course, meeting the relevant assumptions regarding obtaining measurements of pollution concentrations.

The procedure for testing of devices and methods uncertainty is part of the methodology for testing the equivalence of devices for measuring concentrations of pollutants in the air, described in European Union regulations [16]. For this purpose, it is used the measurements of concentrations of particulate matter obtained using the reference method and two candidate devices. All devices should be in close proximity. The test procedure consists of five stages:

1. Test of the measurement uncertainty between the candidate devices.
2. Test of the measurement uncertainty of candidate devices in relation to the reference method.
3. Construction of a regression model between the results of the reference method and results from candidate devices. If the uncertainty has to high value, or regression doesn't meet all assumptions (is inappropriate) measurements of the candidate device must be corrected (calibrated).
4. On the basis of the regression model, if necessary, is built measurements calibrating function.
5. After calibrating the measurements, the uncertainty test procedure is repeated using the new regression model. Negative result indicates the need to modify the candidate method. A positive result confirms the equivalence of methods, but imposes an obligation to continue monitoring and improving the method.

In the first place it is examined whether candidate devices measure the same phenomenon and in the same way. This is done by calculating the uncertainty of measurements between candidate devices (Between sampler uncertainty), using the equation:

$$u_{BS}^2 = \frac{\sum_{i=1}^{N}(y_{i,1}-y_{i,2})}{2n} \quad (1)$$

where: $y_{i,1}$ i $y_{i,2}$ are measurements from two candidate devices. Between sampler uncertainty value, calculated for PM_{10} or PM_{25}, lower or equal to 6.25 $(\mu g/m^3)^2$ indicates slight differences between the results obtained by both measures. This means that both devices measure in the same way and the results of their measurements are comparable [16].

Research methods of candidate uncertainty based on the expanded relative uncertainty of device. It is based on the uncertainty of all identifiable and described factors affecting the

quality of the measurements obtained. In this case, these are uncertainties resulting from: variability of the reference method, regression variability combining the results obtained with both methods and possible variation of the calibration regression. The assumption is also, that the regression between the reference method (x_i) and the candidate method (y_i) has a linear form and is an identity function (the results obtained using the reference and candidate methods should be the same, if insignificantly small errors are omitted). Therefore, the estimation of coefficients in the linear regression model:

$$y = a + bx \tag{2}$$

should have values a=0 i b=1. A deviation from this assumption causes uncertainty which should also be included in the structure of expanded uncertainty. The construction of regression model (2) is used orthogonal regression. This is due to the uncertainty of measurements obtained by both, the candidate and reference methods. The estimator of the measurement uncertainty is a standard deviation of a sample or the product of this deviation and values from the Student's t-distribution for the selected significance level [19-22].

If an uncertainty value is found to be inadequate, the values obtained with the candidate method must be corrected using the calibration function. In the methodology described in the *Guide to the demonstration of equivalence* ... [16], a simple calibration function is built based on the regression model (1) and is written in the formula:

$$y_{cal} = \frac{y-a}{b} \tag{3}$$

assuming the significance of both model (2) parameters. This model can be simpler when one of the parameters is statistically insignificant.

The final form of the combined uncertainty of the candidate method in relation to the reference method, based on the formula for the sum of variance, is described by the formula:

$$u_{CR}^2 = S_e^2 - u^2(x_i) + [a + (b-1) * LV]^2 \tag{4}$$

for uncalibrated measurements, and:

$$u_{CR,cal}^2 = S_e^2 - u^2(x_i) + [c + (d-1) * LV]^2 + [u^2(a) + u^2(b) * LV^2] \tag{5}$$

for calibrated measurements, where:

- S^2_e – variance of the regression model (2) error,
  $u^2(x_i)$ – uncertainty (variance) of the results of the reference method,
- c i d – estimators of coefficients of regression model after calibration,
- $[a + (b-1) * LV]^2$ and $[c + (d-1) * LV]^2$ – the maximum errors resulting from the derogation of regression models from assumptions about identity transformation,
- LV – daily limit value of concentration of tested particulate matter, in the case of PM_{10} PM_{10} LV=50 μg/m^3,
- $u(a)$, $u(b)$ – uncertainties (estimated standard error) of intercept and sloop, respectively. Analogously for parameters c and d in the model after calibration,
- $u^2(a) + u^2(b) * LV^2$ – calibration error. This expression may be partially reduced in the case of statistical non-significance of the parameters of the linear calibration model.

On the basis of the combined uncertainty (4) (5), is calculated the relative expanded uncertainty for the daily limit value of concentration. Finally it takes the form:

$$W_{CM} = k * \sqrt{\frac{u_{CR}^2}{LV}} \quad (6)$$

Extension parameter *k* is half of the spread of the confidence interval for a fixed level of significance *p*. In this case, it is the value in the *t*-Student's distribution *n*-1 degrees of freedom. Usually it is assumed approximate value *k*=2.

The candidate method is acceptable when the value of expanded uncertainty fulfills the condition:

$$W_{CM} \leq W_{acc} \quad (7)$$

The currently applicable regulations assume the limit value of expanded complex uncertainty at the level W_{acc}=25% [16].

The uncertainty analysis is performed for all observations in the sample after the removal of outlying data. This can be done using the procedure derived from the Grubb's test on the occurrence of outliers [23]. The test is repeated for the observation of two sets of data created by separating the original set into observations with PM_{10} concentrations greater than or equal and smaller to 30 μg/m^3. Positive results for each device separately, for all data sets, allow to determine the correct uncertainty of the candidate method.

The method described above allows to calculate the uncertainty of measuring the candidate method in relation to the reference method using a linear calibration function with one independent variable. However, there are situations in which there is a clear influence of other factors on the obtained measurements. The candidate method may function differently from the reference method at other temperatures, air humidity or wind power. The failure to recognize and disregard such factors is one of the reasons for the uncertainty of measurements. The influence of these factors on the measurement result can of course be linear, but it is not obligatory. In this situation, the function calibrating the results of measurements of the candidate method can be any function (it can have any functional form), in which independent variables are all factors significantly affecting the measurement value, and which can be effectively measured. However, it should be noted that, the form of the calibration function and the variables used in this function will have a significant impact on the value of the combined uncertainty of the candidate method.

The study used a linear calibration function in which the independent variables, in addition to the measurement of PM_{10} concentration, were: humidity, air temperature and wind force. As a result of the preliminary analysis, the last two factors turned out to be statistically insignificant and the final form of the modified calibration function was obtained:

$$y_{cal} = a_1 + b_1 y + e_1 H \quad (8)$$

where *H* is the air humidity, y_{cal} is the approximation of the values obtained by the reference method. For uncorrelated variables, the uncertainty square associated with the output value estimate is calculated by the formula:

$$u^2(y_i) = \sum_{i=1}^{k} c_i^2 * u^2(y_i) \quad (9)$$

where c_i are the sensitivity coefficients associated with the estimator of the model parameter, $u(y_i)$ the uncertainty of estimating the parameter for the variable y_i [2]. On the basis of formulas (5) i (9) the combined uncertainty after modified calibration was finally obtained in the form:

$$u_{CR,mcal}^2 = S_e^2 - u^2(x_i) + [c + (d-1) * LV]^2 + [u^2(a_1) + u^2(b_1) * LV^2 + u^2(e_1) * LH^2] \quad (10)$$

This form was used to verify the uncertainty of functioning of the candidate method on the basis of the results obtained in the field study.

3 Results of measurement uncertainty test

The study used the results from measurements of particulate matter PM_{10} concentration, derived from two electronic mobile measuring devices (candidate method) and from the WIOŚ[1] station using a gravimetric measuring method (reference method), located in Rabka Zdrój, Poland. The study was conducted from March 13 to April 27, 2017. Candidate devices use the optical method.

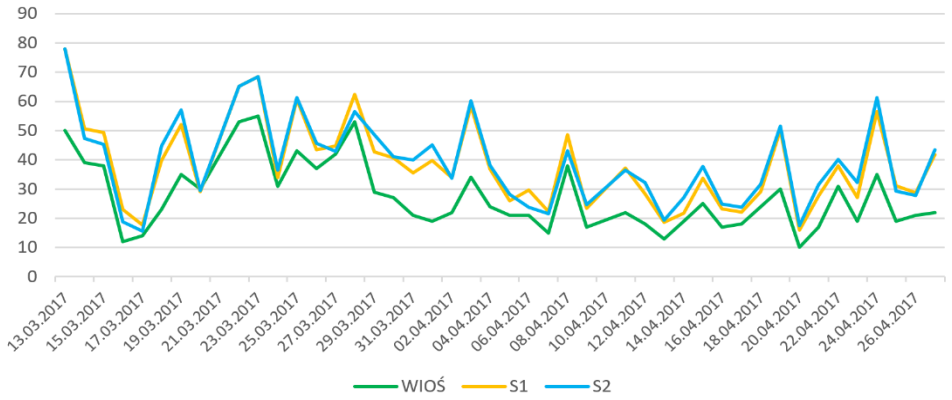

Fig. 1. PM_{10} concentrations in the WIOŚ station and the tested devices, March 13 to April 27, 2017.

One of the electronic devices was in close proximity to the WIOŚ measuring station, the other one a few meters away from it. The location of the second meter causes a systematic error. Therefore, based on this data, it is not possible to conduct a full equivalence study of the candidate method. Meters provide PM_{10} concentration data every hour, however, for comparison with the reference method, measurements were aggregated to daily data by calculating 24-hour averages. Obtained in this way 46 observations. Then, using the Grubbs test, outliers were removed, leaving $n = 44$ observations used later in the study. The values of obtained measurements of PM_{10} concentrations coming from both devices (S1 and S2) and the reference method (WIOŚ) are shown in Fig. 1.

On the basis of Fig. 1., a similar structure of all three series can be observed. However, there are noticeable differences between them. For the vast majority of observations, the concentration measurements obtained by the reference method are smaller than the values obtained by candidate methods.

First of all, the uncertainty of measurements between candidate devices (between sampler uncertainty) was tested. The analysis shows that this uncertainty met the assumptions, i.e. it was less than 6.25 for all required data sets (Table 1.). This indicates satisfactory reproducibility of the results obtained by both devices using the candidate method.

Table 1. Values of uncertainty between candidate devices.

Between sampler uncertainty	**all data**	**data ≥ 30 µg/m³**	**data < 30 µg/m³**
u_{BS}^2	5.283	5.513	4.918

[1] WIOŚ is a government institution whose task is to supervise the condition of the natural environment in Poland.

The results of the uncertainty analysis of S1 and S2 devices relative to the reference method are presented respectively Table 2 for device S1 and Table 3 for device S2. They contain parameters of regression models and uncertainty assessment of these parameters, simple calibration function and regression model after calibration together with the equivalence assessment of the device. They also include an extended calibration function along with an evaluation of the results obtained using it. The analyzes were performed assuming a confidence level 1-α=0.95, uncertainty of the reference method $u^2(x_i)$=0.67 and acceptable uncertainty of the measurement method W_{acc}=25%.

For the device S1 (see table 2), the verification of the expanded uncertainty condition concerning all measurements indicated the need to correct the obtained results using the calibration function. The use of a simple calibration function (3) use gave positive results and the device passed the test successfully. A different situation occurs in the case of measurements with values above 30 μg/m^3. For this observation group, the value of the expanded uncertainty is too high and the test can't be passed. Unfortunately, both parameters of the regression model are irrelevantly different from 0 and 1, which indicates the inability to build a simple function that calibrates the results. This means that it is not possible to adjust the results to a level that allows this part of the uncertainty test to pass.

Table 2. The results of the measurement uncertainty test of the S1 device.

Feature	**all data**	**data ≥ 30 μg/m^3**	**data < 30 μg/m^3**
estimation of regression parameters	a=2.629 b=1.307*	a=9.444 b=1.136	a=8.476* b=0.880
the original regression model	y=2.629+1.307x	y=9.444+1.136x	y=8.476+0.880x
combined uncertainty u_{CR}	19.018	*17.448*	*3.752*
expanded relative uncertainty W_{CM}	0.761	*0.698*	*0.15*
uncertainty test result	**not accepted**	**not accepted**	**not accepted**
simple calibration function	y_{cal}=0.765y	y_{cal}=y	y_{cal}=y-*8.476*
estimation of regression parameters after calibration	c=2.651 d=0.977	a=9.444 b=1.136	c=0 d=0.880
regression model after calibration	y_{cal}=2.651+0.977x	y_{cal}=9.444+1.136x	y_{cal}=0+0.880x
combined uncertainty $u_{CR,cal}$	6.246	*17.448*	7.622
expanded relative uncertainty W_{CM}	0.2499	*0.698*	0.305
uncertainty test result	**accepted**	**not accepted**	**not accepted**
extended calibration function	y_{cal}=0.775y-0.034H	y_{cal}=0.869y-0.102H	y_{cal}=0.963y-0.068H
estimation of regression parameters after calibration	c=-0.302 d=0.998	c=-0.437 d=1.003	c=1.655 d=0.902
regression model after calibration	y_{cal}= -0.302+0.998x	y_{cal}= -0.437+1.003x	y_{cal}= 1.655+0.902x
combined uncertainty $u_{CR,mcal}$	4.943	5.812	7.275
expanded relative uncertainty W_{CM}	0.198	0.232	0.291
uncertainty test result	**accepted**	**accepted**	**not accepted**
* indicates parameters significantly different from: 0 for intercept parameters a i c, and 1 for slope parameters b i d, with significance level α=0.01.			

A similar situation occurs for samples with concentrations of PM_{10} less than or equal to 30 μg/m^3. The value of expanded relative uncertainty allows to pass the uncertainty test

without calibrating the results. Unfortunately, the relationship (regression) between measurements has a parameter *a* (intercept) significantly different from 0. This indicates a systematic shift of the obtained measurements. Unfortunately, the correction with a simple calibration function eliminates the problem of systematic error, but increases the uncertainty of measurements. The value of expanded uncertainty exceeds the acceptable value. In this part of the uncertainty test, the test is also failed.

The analysis of the uncertainty of candidate device was carried out again using a modified calibration function, based on linear regression dependent on the values obtained by the device and air humidity. The air temperature and wind force were statistically insignificant and were not used in the calibrating function. Both variables are uncorrelated with each other (values of correlation coefficients are statistically insignificant), therefore it is possible to use them to calculate combined uncertainty (9). In the calculations the value of the sensitivity coefficient for air humidity was assumed as LH=0.9.

The application of a multi-variable calibration function allows to adjust the results to a level where the test can pass both, for all observations, and for observations corresponding to high concentrations of particular matter. For low concentrations of PM_{10}, the test was negative, but the value of expanded uncertainty only slightly exceeds the permissible standard.

Table 3. The results of the measurement uncertainty test of the S2 device.

Feature	**all data**	**data ≥ 30 μg/m³**	**data < 30 μg/m³**
estimation of regression parameters	a=3.308 b=1.316*	a=11.454 b=1.103	a=8.009 b=0.890
the original regression model	y=3.308+1.316x	y=11.454+1.103x	y=8.009+0.890x
combined uncertainty u_{CR}	20.632	18.462	3.653
expanded relative uncertainty W_{CM}	0.825	0.738	0.146
uncertainty test result	**not accepted**	**not accepted**	**accepted**
simple calibration function	y_{cal}=0.760y	y_{cal}=y	-
estimation of regression parameters after calibration	c=3.595 d=0.960	a=11.454 b=1.103	-
regression model after calibration	y_{cal}=3.595+0.960x	y=11.454+1.103x	-
combined uncertainty $u_{CR,cal}$	7.686	18.462	-
expanded relative uncertainty W_{CM}	0.307	0.738	-
uncertainty test result	**not accepted**	**not accepted**	**-**
extended calibration function	y_{cal}=0.764y-0.039H	y_{cal}=0.852y-0.010H	-
estimation of regression parameters after calibration	c=0.268 d=0.973	c=2.082 d=0.931	-
regression model after calibration	y_{cal}=0.268+0.973x	y_{cal}=2.082+0.931x	-
combined uncertainty $u_{CR,mcal}$	6.225	7.972	-
expanded relative uncertainty W_{CM}	0.249	0.319	-
uncertainty test result	**accepted**	**not accepted**	**-**
* indicates parameters significantly different from: 0 for intercept parameters a i c. and 1 for slope parameters b i d. with significance level α=0.01.			

In the case of the S2 device (Table 3.). the test performed for all observations. both before and after a simple calibration. didn't give satisfactory results. For observations whose values are higher than 30 μg/m³. the uncertainty test gives a negative result. but it is not possible to build a simple calibration model (the calibration function is an identity function). For

observations less than or equal to 30 $\mu g/m^3$ measurement uncertainty test gave positive results.

The use of a modified calibration function allowed to pass the test for all observations. In the case of the observation of concentrations value greater than 30 $\mu g/m^3$ the test was negative, but with a relatively low value of expanded uncertainty.

The results obtained for both devices using a simple calibration function show that in selected data configurations. the devices have inadequate measurement uncertainty. This means that devices are not equivalent with the reference method. It can be improved with the help of an extended calibration function. The modified calibration function used improves the results in a way that allows to conclude that the obtained results are similar to the reference measurements.

4 Conclusions

On the market there is a growing number of measuring devices to ensure air quality testing methods other than the reference methods for type of pollution. In order to be able to use them efficiently. it is necessary to state the equivalence of these devices with reference methods. A significant part of the methodology of demonstrating the equivalence of methods is the analysis of the uncertainty of device measurements. Too high value of uncertainty makes it impossible to use the device in professional applications.

The uncertainty of a candidate measuring device is a composition of: the variability of the model describing the dependence of the reference method and the candidate method. and the deviation of this model from the identity function. the variability of the reference device. and the potential variability of the calibration function of the candidate device. The uncertainty function is described by the formula (5).

The commonly used methodology of demonstrating the equivalence of devices measuring air pollution uses as a calibration function a linear regression model based solely on device measurements. However. this approach may give unsatisfactory corrections to the measurement results. Due to the fact that the concentration of particulate matter in the air may be affected by temperature and humidity. the direction and force of the wind and other factors. it is proposed a calibrating function also uses these factors. The use of such a modified calibration function also forces changes in the calculation of measurement uncertainty. They depend on the number of used variables and the form of used function.

In the conducted study. the only important factor influencing the results of measurements of PM_{10} concentrations was air humidity. Using it in a linear calibration function (8) gave the desired results in most cases. The measurement uncertainty for this calibration function is calculated using formula (10). Its values were within the required standards for all observations. with the exception of the measurement groups for high concentrations of PM_{10} for the device S2 and low concentrations for the device S1. These results indicate a certain accidental inefficiency of the model. Perhaps it can be explained by relatively small samples. In the real equivalence study. it will be necessary to use much larger samples. which can solve the problem.

It should be added that it is possible to create an uncertainty function for almost every calibration function. Unfortunately you can't give one coherent model describing this uncertainty. Each calibration model requires its own analysis of uncertainty.

The obtained results indicate that in the devices measuring the PM_{10} particulate matrices. modified calibration functions can be effectively be used. The results obtained with this function can be much closer to the measurements obtained by the reference method. and measurement uncertainty may have a lower value. As result. it will be possible to demonstrate the equivalence of measurement methods and full use of electronic measuring devices for testing air quality.

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