

Comparison of Tests for Equivalence of Methods for Measuring PM₁₀ Dust in Ambient Air

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Abstract

Our paper presents the motives for conducting the equivalence tests and the reasons of discrepancies between the results of PM₁₀ dust concentration obtained via the reference method and the methods based on 'on-site' measurement. The reference and non-reference methods of measurement of PM₁₀ dust concentration in air have been discussed. Field tests for equivalence, following the Polish standard and the European Union (EU) guidelines, carried out by the ARMAAG Foundation in the Tri-City region, have been analyzed. Correction factors with respect to the 'on-site' samplers, obtained in various measurement stations in the EU member countries, also are presented.

Keywords: PM₁₀ dust, field test, correction factors, Eberline sampler, TEOM sampler

Introduction

Protection of air against pollution is one of the most important elements of European ecological policy. There are numerous directives and legal regulations in this field [1]. One of the most significant pollutants constituting a serious hazard for human health is dust contained in ambient air. Numerous epidemiological investigations reveal a statistical relationship between elevated dust concentration in air and adverse health impact, including respiratory and cardiovascular diseases [2-4]. Dust consists of a mixture of solid and liquid particles suspended in air, being a combination of organic and inorganic substances. It can be emitted from both natural sources (soil and rock erosion, sea spray) and anthropogenic sources (combustion of fuels, emission by various means of transport). Chemical composition of dust depends on its origin and it can contain: simple anions (sulphates, nitrates, chlorides, silicates, and car-

bonates), simple cations (ammonium, sodium, potassium, calcium, magnesium, and aluminium ones), heavy metals (arsenic, cadmium, lead, nickel, chromium, vanadium, and adsorbed organic compounds) and polycyclic aromatic hydrocarbons (benzo(a)pirene, benzo(a)anthracene, etc.) [5, 6]. Dust is classified according to the size of its particles. Air pollution standards usually refer to the dust, the aerodynamic diameter of which does not exceed 10 µm termed PM₁₀, to the dust of equivalent diameter 2.5 µm named PM_{2.5}, and to the dust of diameter not exceeding 1 µm abbreviated PM₁.

Annual investigations and evaluations of air quality [7] (performed by the Provincial Environmental Protection Inspectorate in Gdańsk and by other provincial environmental protection inspectorates) show that in practice it is only suspended dust that constitutes a serious problem as far as Polish and European quality standards are concerned. In southern Europe (Italy [8, 9], Cyprus [10], Greece [11], Spain [12]), in central Europe (Switzerland [13], Austria [14]) or northern Europe (Great Britain [15], Ireland [16])

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PM₁₀ dust concentrations exceed admissible annual limit 40 µg/m³, as well as the admissible 24 hour limit.

50 µg/m³ (exceeding the admissible 24 hour limit) is allowed to occur only 35 times a year. The European Union has set the standards concerning admissible dust concentrations in ambient air and has obliged the member countries to undertake effective actions in case the threshold concentration of dust is exceeded. The scope of these actions depends on the results of air quality monitoring. Thus, the legal regulations enforce consistent and objective measurements of dust concentration in ambient air. The legal system in Poland, following the EU regulations, requires ambient air quality control to be carried out annually. The evaluation is performed by the Provincial Environmental Protection Inspectorate according to Articles 89 and 90 of the Environmental Protection Law [17]. Analysis of the monitoring results allows identification of the zones where air quality is unsatisfactory. The Provincial Environmental Protection Inspectorate provides the provincial marshal with this information, and if the air quality standards are exceeded the Marshal launches the air quality protection programs. In the zones of high concentration of pollutants it is required that high-quality measurements are carried out in the stationary stations employing the flowmeters working according to the reference method or the ones having the certificate of equivalence with the reference method [18].

Reference and Non-Reference Methods for Measurement of PM₁₀ Dust

In Poland the reference method for measurement of PM₁₀ dust concentrations in ambient air is defined by the Regulation of the Minister of the Environment of 17th December 2008 [19]. Accordingly, the reference method is the manual gravimetric method described in the standard PN-EN 12341. The method provides dust concentration averaged over 24 hours, but the results are not available on-line (a filter with trapped dust is stabilized in laboratory at temperature and humidity specified in the standard). Due to various reasons there is also a need for the measurement providing dust concentration averaged over a shorter time period (for example 1 hour) with the results available online. Such a mode is named a continuous 'on-site' measurement. Commonly applied continuous samplers are: Eberline sampler (the measurement method is based on absorption of beta radiation) and TEOM sampler (the measurement method consists in change of frequency of an oscillatory balance). Because of a relatively small number of the reference samplers in the measurement stations (economic reasons), other instruments are utilized in PM₁₀ measurements. That is why air quality control with respect to PM₁₀ dust is also performed using automatic methods, not listed as the reference ones. Accuracy and stability of such measurements depend on numerous factors. Changes of dust mass are caused by physico-chemical and chemical processes such as:

Table 1. Correction factors for Eberline and TEOM samplers obtained from the selected stations disseminating information about air quality via the AirBase network.

Country	Eberline sampler	TEOM sampler
Austria	1.18-1.3	1.16-1.3
Belgium	1.37	1.47
Estonia	1.15	-
Germany	1.0-1.3	1.0-1.3
Denmark	-	1.23-1.36
Spain	0.84-1.3	1.0-1.3
Hungary	1.0-1.31	-
Italy	-	1.3
Lithuania	1.3	-
Luxemburg	-	1.2
Netherlands	1.33	-
Norway	-	1.1
Portugal	1.11-1.18	1.1-1.2
Switzerland	-	1.12-1.3
Sweden	-	1.1-1.3
Slovakia	-	1.3
Great Britain	-	1.3

- water vapour desorption
- absorption of water vapour from air (permanent binding of dust with water – hydration)
- chemical decomposition of the substances contained in dust via the process of evaporation
- chemical reactions leading to the formation of solid substances (for example formation of salts)

All these processes are dependent on temperature, pressure, sun aberration, humidity and concentration of pollutants. In the equipment for 'on-site' measurement the air sucked in and the filter with trapped dust are heated to constant, higher than ambient temperature. Such an approach usually results in a loss of dust mass, yielding the results underrated as compared to the reference method. The difference may reach even 47% and depends on dust composition, site of measurement, metrological parameters, season, and measurement method. In order to assure an equivalence of the results obtained via the reference method and the 'on-site' method, there are some correction actions taken that employ correction equations or correction factors. Table 1 presents the values of correction factors for both Eberline and TEOM samplers obtained from selected EU member countries disseminating the information about air quality via the AirBase network [20-22].

The most commonly recognized reason of underestimation of dust concentration using the TEOM sampler as compared to the gravimetric reference method [23-26] is a loss

of volatile and semi-volatile substances. Volatile organic compounds and inorganic compounds such as ammonium nitrate are mentioned here. Another source of the differences between the TEOM sampler and gravimetric method are the phenomena connected with water vapour sorption on dust particles and chemical or physicochemical binding of water with the components of dust. It is suggested [27-29] that the TEOM samplers heat the air stream flowing through the microbalance filter, causing evaporation of water from dust particles. An opposite situation is in the case of quartz filter of the reference sampler, which retains dust particles together with entrapped water; then upon conditioning in the laboratory under 50% relative humidity (RH) conditions, no complete water desorption due to a hysteresis effect in case of selected inorganic salts occurs. The amount of water combined with PM₁₀ will be dependent on dust composition and relative humidity of air. This amount will vary depending on the season and time, and on characteristics of local and regional sources of pollution. Moreover, it has been proved that adsorption of organic vapours on the developed surface of the quartz filter may lead to significant differences between results obtained using the TEOM sampler and gravimetric approach. Green et al. [30] put an emphasis on the differences in the TEOM results depending on pressure and temperature (standard or ambient). In Great Britain the correction factor 1.3 has been used for the TEOM sampler independent of the season. In the case of the Eberline sampler the differences in PM₁₀ concentration as compared to the gravimetric method are smaller than an analogous difference between the TEOM sampler and gravimetric test. The most frequently reported reason for a difference between the gravimetric method and Eberline sampler is relative humidity of air [31, 32]. In this case the Eberline sampler may yield higher concentrations than the gravimetric method. In the case of moderate RH and temperatures the Eberline device underestimates PM₁₀ concentration as compared to the gravimetric method. Comparative measurements in air quality monitoring are of substantial importance as far as determination of the correction factors is concerned. Incorrect measurements of dust concentration can result in a dual way. On one hand, if the results are underrated, a population will be exposed to deteriorating environmental quality affecting its health. On the other hand, overrated results can stimulate the launch of unnecessary, costly correction programs.

Methodology of Equivalence Determination

According to the Directive of European Parliament and European Council of 21st May 2008 concerning air quality and clean air for Europe, member countries are allowed to apply another measurement method provided that they are able to demonstrate its equivalence with the reference method. There are two ways to perform the equivalence tests:

- laboratory test – necessary only in the case when the non-reference method is a modification of the reference method (estimation of different components of uncertainty sources)

- field test – the non-reference method is verified ‘side by side’ with the reference method

The second form of the equivalence test will be discussed here in more detail as the aim of this paper is estimation of the correction equations or correction factors for the devices operating in on-line mode, which differ significantly from the reference method as far as the determination of PM₁₀ dust concentration in air is concerned.

According to the standard PN-EN 12341, the criteria for acceptance of a candidate sampler as equivalent to the reference sampler are as follows:

- at least 40 ‘side-by-side’ measurements
- application of regression for a set of ‘side-by-side’ measurements
- linear equation $Y=a+bX$ obtained via regression should be in the area of two-sided envelope of acceptance $Y=X\pm 10 \mu\text{g}/\text{m}^3$
- coefficient of determination of obtained linear equation should be $R^2\geq 0.95$

In a non-standard guide [33], the European Union recommends a procedure based on estimation of uncertainty of results obtained using a candidate sampler. The criteria for acceptance of the candidate sampler as equivalent to the reference sampler are as follows:

- 4 campaigns a year, each with 40 ‘side by side’ measurements
- orthogonal regression applied to each set of measurements separately and then again to one global measurement set
- inclination coefficient of the straight ‘b’ does not differ significantly from unity, coefficient of axis interception ‘a’ does not differ significantly from zero
- elimination of gross errors using Grubbs test
- difference in expanded relative uncertainty between results for the candidate sampler and the reference sampler should be less than 25%

Unlike the standard PN-EN 12341, the guide proposed by the European Union allows members not only to decide if a particular candidate sampler fulfils the reference conditions, but also makes it possible to determine the correction equation or correction factor. Such an approach was employed by Gehrig et al. to determine the correction equations for the Thermo ESM Andersen automatic sampler [26, 34]. The standard does not anticipate such a possibility; it only defines whether the candidate sampler is/is not in conformity with the reference conditions.

Field Test for Equivalence

The Agency of Regional Air Quality Monitoring in the Gdańsk Metropolitan Area performed the measurements of suspended PM₁₀ dust concentrations in the AM9 station between 7th February 2007 and 9th February 2008. There were reference sampler HVS Digital and two automatic dust samplers (candidate samplers) installed: Eberline (absorption of β particles) and TEOM (oscillatory balance). The aim of the measurements was comparison of data on suspended PM₁₀ dust concentrations obtained using the

gravimetric reference method and on-site dust samplers. Based on these results, the authors made an attempt to decide if the candidate samplers fulfil the conditions of equivalence and to estimate the correction equations or correction factors for both samplers. Due to the comparative character of both presented methodologies, comparison of the results obtained from different samplers was limited to summer (from 1st May 2007 to 31st August 2007) and winter heating season (from 31st October 2007 to 31st January 2008). The AM9 station is located in Gdynia beyond direct impact of communication routes, big sources of emission, and where individual heating employs gas or oil heaters. This station is representative for the region characterized by a stable level of PM₁₀ dust concentration. Volumetric flow rate of air through the samplers was as follows: 700 l/min for HVS, 16.7 l/min for Eberline and 3 l/min for TEOM. The volumes of air passed through all the devices were related to standard conditions, that is to 20°C and 1 atm. Calibration of the automatic samplers was carried out twice a year (flow and zero level). In the case of the HVS sampler only flow was calibrated (the sampler was new and possessed calibration certificates).

Principle of Operation of Measurement Devices

Eberline Sampler

The measurement using beta particles absorption method consists in passing the air sucked in through a filtering tape, which is moved in a step mode. The amount of dust deposited on the filter is determined by measurement of gradual dumping of beta radiation penetrating the filter. A source of the radiation is a synthetic sample exhibiting suitable activity (isotopes of C₁₄ or Kr₈₅), whereas a Geiger-Muller meter or ionization chamber constitutes a detector. A degree of radiation absorption is compared before and after or before and during trapping of dust. In this way the cumulative mass of deposited particles is measured.

TEOM Sampler

This device allows measurement of dust concentration by passing an air sample through a filter oscillating with characteristic frequency. The trapped dust increases mass of the system, thus decreasing frequency of the oscillations. Concentration of dust in air is determined taking calibration frequency, dust mass, and sample volume into account.

HVS Digitel

A high-volume (up to 1000 dm³/min) sampler sucks the air in via a blower; dust particles suspended in air are adequately accelerated in a separator in order to feed a filtration chamber with the particles of a diameter up to 10 µm. A quartz filter stops dust particles and clean air is directed into the sampler's outlet. The filters are exchanged automatically every 24 hours until all the spare filters loaded into a feed-

Table 2. Results of 'side by side' measurements of PM₁₀ dust obtained using the reference and candidate samplers localized in AM9 station.

Parameters	Reference sampler	Candidate sampler Eberline	Candidate sampler TEOM
Number of measurement days in summer	123		
Number of valid results (days) in summer	102		
Average dust concentration in summer (µg/m ³)	16.6	15.6	17.0
Number of measurement days in heating season	123		
Number of valid results (days) in heating season	101		
Average dust concentration in heating season (µg/m ³)	27.3	22.8	21.3

er are used. As the sampler turns off, new filters are inserted and the filters with dust are transferred to a laboratory in order to determine mass increment. Having the volume of air passed through the filter and the mass of dust trapped in the filter, it is possible to calculate PM₁₀ dust concentration. A microprocessor module placed inside the sampler enables recalculation of the air passed during 24 h from the actual conditions (temperature, pressure) into the reference conditions, namely 20°C and pressure of 1013.25 hPa.

Results

Table 2 gathers data obtained from the measurements performed in the summer and heating seasons.

The characteristics presented in Figs. 1A, 1B and 2A, 2B were determined by applying regression to the set of 'side by side' data (least squares method) obtained from the reference and candidate samplers and by applying a two-sided envelope of acceptance according to the standard PN-EN 12341.

Table 3 presents analysis of the acceptance criteria of equivalence between the candidate and reference samplers following the standard PN-EN 12341.

The courses shown in Figs. 3A, 3B and 4A, 4B were determined by applying regression to the set of 'side by side' data (orthogonal regression) obtained from the reference and candidate samplers and by following the instructions contained in the EU guide [33].

Table 4 presents analysis of the acceptance criteria of equivalence between the candidate and reference samplers according to the EU guide [33].

In case direct reference requirements are not satisfied, this test [33] anticipates a possibility to fulfil the requirements of indirect reference via implementation of the correction equations. Then the candidate sampler is rejected as not complying with the reference requirements when it is

Table 3. Acceptance criteria of equivalence between the candidate and reference samplers according to the standard PN-EN 12341.

Candidate sampler	Location of functional dependence within area of two-sided envelope of acceptance	Coefficient of determination R^2	Fulfilment of reference requirements
Eberline (summer)	yes	0.71	no
Eberline (heating season)	no	0.93	no
TEOM (summer)	yes	0.68	no
TEOM (heating season)	no	0.92	no

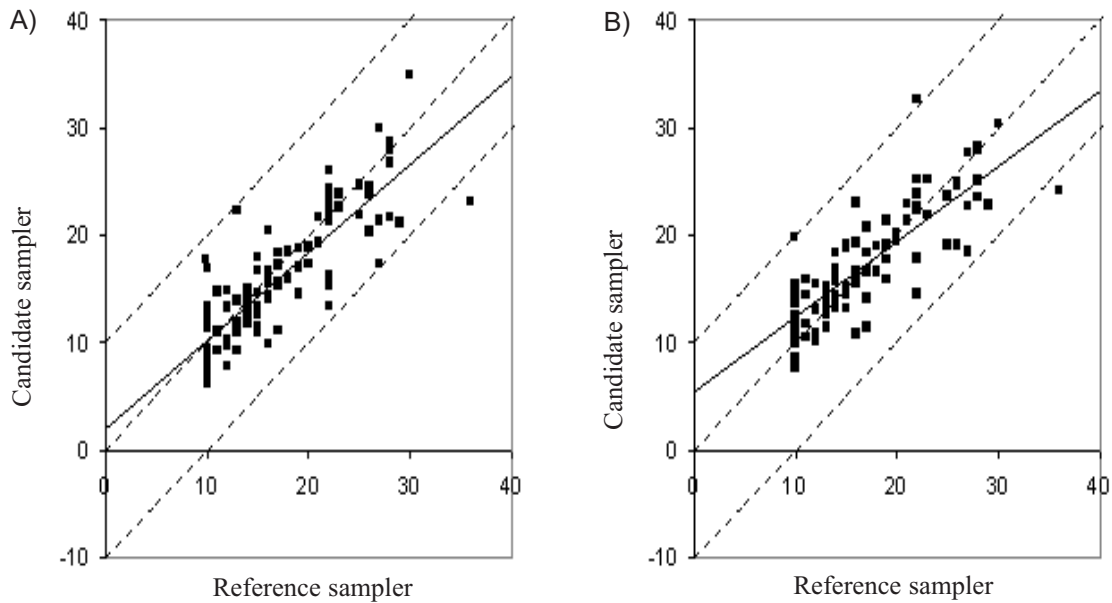


Fig. 1. A) Functional dependence of PM₁₀ dust concentration (linear regression) determined in summer using reference method and beta particles absorption method. B) Functional dependence of PM₁₀ dust concentration (linear regression) determined in summer using reference method and oscillatory balance method.

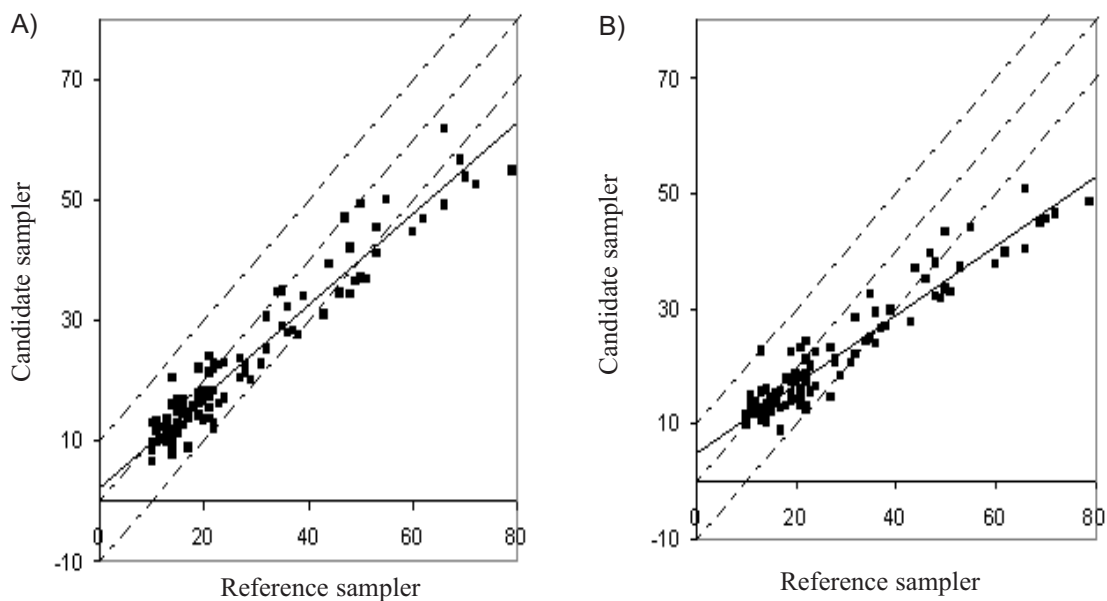


Fig. 2. A) Functional dependence of PM₁₀ dust concentration (linear regression) determined in heating season using reference method and beta particles absorption method. B) Functional dependence of PM₁₀ dust concentration (linear regression) determined in heating season using reference method and oscillatory balance method.

Table 4. Acceptance criteria of equivalence between the candidate and reference samplers according to the EU guide [33].

Candidate sampler	Statistical significance of 'b' coefficient of orthogonal regression	Statistical significance of 'a' coefficient of orthogonal regression	Expanded uncertainty [%]	Fulfilment of reference requirements
Eberline (summer)	not significant	not significant	14.3	yes
Eberline (heating season)	significant	significant	40.0	no (correction of results)
TEOM (summer)	significant	significant	24.2	yes (correction of results)
TEOM (heating season)	significant	significant	59.7	no (correction of results)

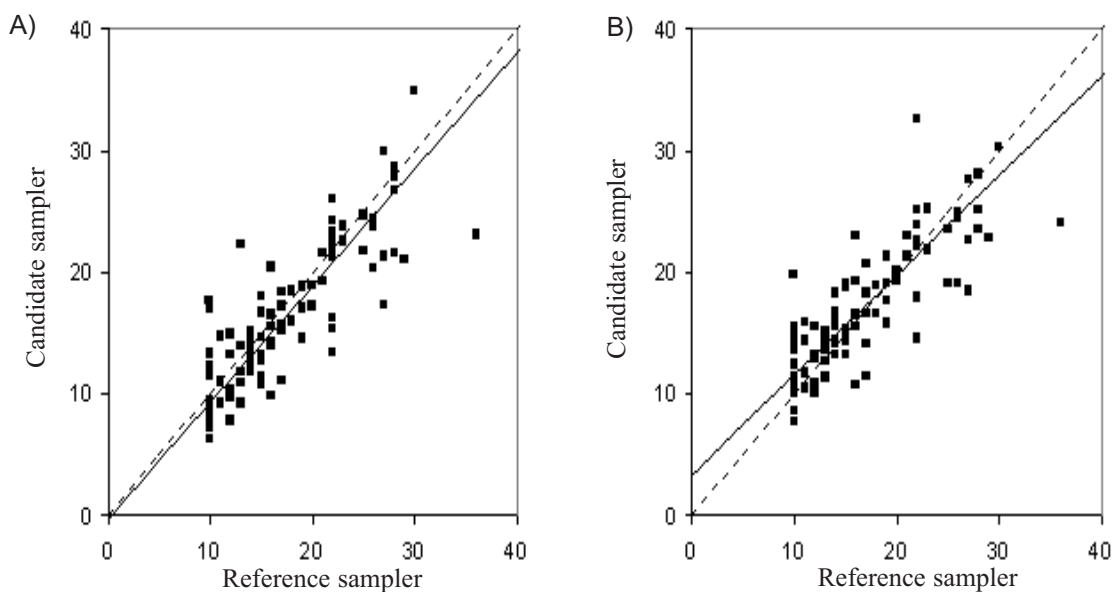


Fig. 3. A) Functional dependence of PM_{10} dust concentration (orthogonal regression) determined in summer using reference method and beta particles absorption method. B) Functional dependence of PM_{10} dust concentration (orthogonal regression) determined in summer using reference method and oscillatory balance method.

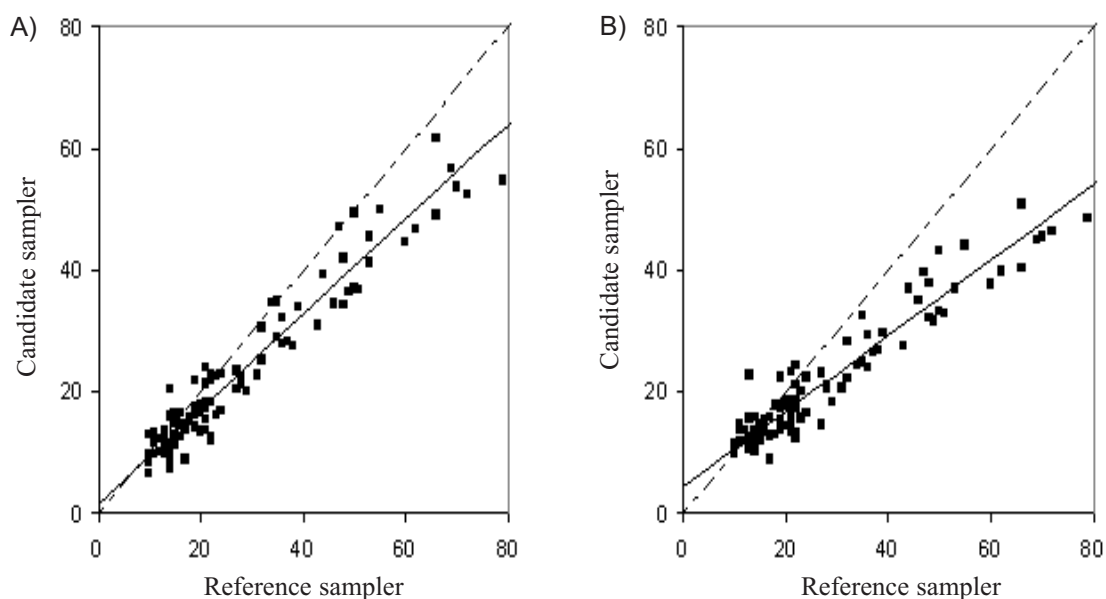


Fig. 4. A) Functional dependence of PM_{10} dust concentration (orthogonal regression) determined in heating season using reference method and beta particles absorption method. B) Functional dependence of PM_{10} dust concentration (orthogonal regression) determined in heating season using reference method and oscillatory balance method.

Table 5. Acceptance criteria of equivalence between the candidate and reference samplers according to the EU guide [33], including correction of the results.

Candidate sampler	Expanded uncertainty prior to correction [%]	Expanded uncertainty after correction [%]	Correction equation
Eberline (heating season)	40.0	17.0	$C_R = 1.28C_{NR} - 2.00$
TEOM (summer)	24.2	17.3	$C_R = 1.22C_{NR} - 4.09$
TEOM (heating season)	59.7	19.4	$C_R = 1.61C_{NR} - 7.16$

C_R is the concentration of PM_{10} dust obtained from reference sampler, C_{NR} is the concentration of PM_{10} dust obtained from candidate sampler.

not feasible to elaborate the equation, yielding expanded uncertainty of the candidate sampler lower than 25%. Table 5 presents correction of the results in order to estimate the correction equation according to the EU guide [33].

Conclusions

Reliable application of an on-site method for measurement of PM_{10} dust and dissemination of the information about concentration of dust in air requires suitable equivalence tests in order to verify compliance of the method used with the reference one. The authors of this paper carried out two independent equivalence tests and arrived at the conclusion that in the case of the automatic samplers, the PN-EN 12341 standard sets too strict requirements and is not an appropriate tool to define equivalence of the automatic samplers. It is confirmed by the information contained on page 4 of the standard saying that it is not suitable for the automatic samplers. Following the standard PN-EN 12341, it was shown that none of the candidate samplers fulfilled the reference requirements, both in summer and heating seasons. Following the EU guide [33], it was revealed that in summer the Eberline sampler fulfilled all the requirements and was equivalent to the reference sampler. Eberline sampler in heating season and TEOM sampler in both summer as well as heating seasons fulfil the reference requirements after application of the correction equations. The results obtained are in accordance with the expectations that the correction factors are lower for the Eberline sampler than for the TEOM sampler. Moreover, higher correction factors can be observed for winter (heating) season than for summer. It is due to difference in temperature of ambient air and the air flowing through the measurement ducts of automatic samplers. According to literature in summer, when this difference is the smallest, there is a slight difference in indications of the automatic samplers and the gravimetric method. In winter this difference takes the highest values, which is reflected in big varieties of slopes of the calibration curves (there is PM_{10} mass loss in the automatic samplers due to volatility of dust components). Another factor aggravating the phenomenon can be related to volumetric flow rate of air. In the case of significant volume of flowing air the accuracy of measurement increases, and the flow rates in the Eberline and TEOM samplers differ 5 times. There is one more aspect connected with recalculation of

volume of sampled air into standard conditions (this practice has been abandoned since 2009), which may generate additional differences in results. Independent of a number of parameters influencing obtained differences, a convenient and rational approach is implementation of seasonal factors or correction equations. They will account for metrological parameters as well as for time-space distribution of pollutants.

The Agency of Regional Air Quality Monitoring in the Gdańsk Metropolitan Area is one of only a few institutions in Poland that performs field equivalence tests providing valuable information about dust concentration and its dependence on various external parameters. High levels of these equivalence tests as well as cooperation with the provincial environmental protection inspectorates and the Main Environmental Protection Inspectorate should result in elaboration of a common, unified program of equivalence tests having defined criteria and conditions. Unification of these rules will make it easier to conduct the equivalence tests in Poland and will comply with the EU recommendation for the member countries to apply the approach to verify the equivalence of a particular method utilized with the reference method.

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