Procedural and Methodological Example of Gravimetric Measurement of Pollutant Particles in the Environment using Sampling Devices

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Abstract: *This paper deals, using a demonstrative example, with the methodological procedure of gravimetric measurement of the polluting particles, according to the standard SR EN 12341/2014, for the real evaluation of the 10 μm particle in the biphasic suspensions discharged into the environment. The procedure can be used to compare a non-certified measuring instrument with a reference instrument by verifying that the conditions for the difference between the particle-specific concentration of the particulate immissions (<± 10 μg / m*^{3}*) and their concentration in the environment (< 100 μg / m*^{3}*). The mathematical processing of the comparative results through the regression equation must verify that a correlation coefficient value is obtained to validate the fulfillment of the standard requirements.*

Keywords*: Particles, pollutants, immissions, regression, correlation.*

1. Introduction

Atmospheric pollution has become a major issue for today's society, and efforts to breathe clean air are of great importance. In day-to-day life, the introduction into the atmosphere, directly or indirectly, of substances or energy that is harmful to human health and / or the quality of the environment is air pollution. This causes large societal damages due to the serious socio-economic consequences that can affect human health and / or environmental quality.

As a result of the very large influence of the anthropogenic sources with major potential on air pollution (large combustion plants - IMA, transport, petrochemical industry, construction materials industry, etc.), the pollutants in suspension were noted, with PM (Particulate Matter) representing solid substances in the form of powders, which means an amount of substance present in a given volume. They are a category of air pollutants that greatly affect human health because they include harmful substances such as nitrates, sulphates, organic carbon, elemental carbon, dust, etc. in their composition [1].

Because pollutant particles can come from very different sources, they lead to a number of specific morphological, chemical and physical properties, and their distribution according to their dimensions is an important parameter that influences their behavior. Thus, PM10 pollutants, as defined by EPA (Environmental Protection Agency, USA), include more than 50% of particles collected by a 10 μm diameter sampler and the specific shape penetration curve [1, 2].

In recent years, government agencies in many European countries have monitored particulate matter PM10, respectively the total concentration of all particles smaller than 10 μm in diameter, which corresponds to all fine particle areas plus the smallest classes of coarse particles, all these being called, particles that can be inhaled. Pollutants with the PM2.5 index, which includes all fine particles with diameters less than 2,5 μm (inhalable particles), are also monitored. The new term of superfine particles is applied to particles with very small diameters, typically less than 0.05 μm (50 nm) and PM0.05 [3], respectively.

Therefore, it is imperative to step up efforts in order to monitor and control environmental pollution by using advanced methods and techniques to ensure increased quality indicators for all environmental factors, in accordance with applicable norms, standards and regulations.

2. Description of the method of gravimetric measurement of the powders content coming from gaseous pollutant emissions

The gravimetric measurement principle consists in the analysis of a representative volume of gaseous effluent extracted under isokinetic conditions from from an outlet channel. As schematically shown in Fig. 1, several simultaneous and successive thermotechnical determinations are made. Thus, the flow conditions (flow, static and dynamic pressure, humidity, temperature, etc.) must be known and a strategy correlated with the concrete conditions regarding the selection of the sampling section and the representative points [4, 5].

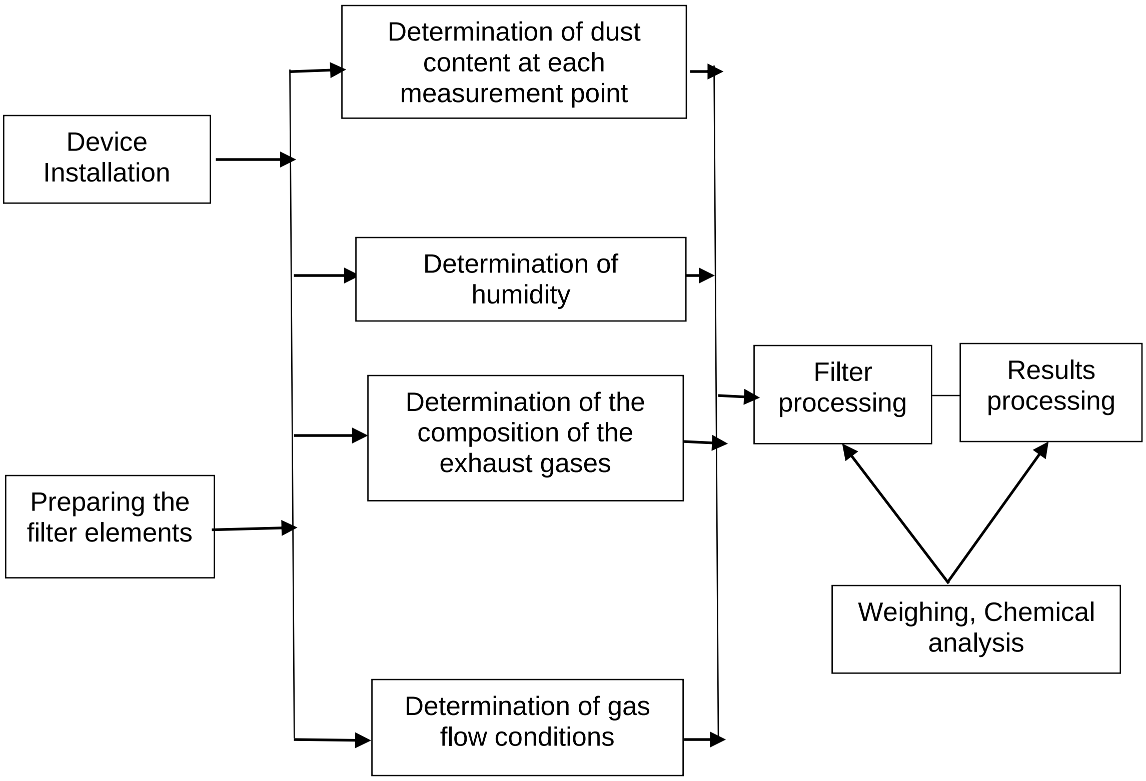

Fig. 1. Scheme of procedures necessary for the experimental determination of the dust content of gaseous pollutant emissions [5].

The installation with which the gravimetric measurement of the amount of dust pollutants from industrial gaseous emissions is carried out through the chimneys (exhaust channels) thereof is complex and basically consists of several independent components [6, 7].

After the installation, it is brought to the operating temperature (120^{o}C), the sealing is checked, operation followed by starting the suction pump. Then, the gas intake through the bypass system. Throughout the measurement, the flow parameters of the effluent are monitored and the data of the measured quantities are periodically noted. At the end of the measurement, the gas suction is switched off, after which the filter element is removed and stored in a specially prepared container. Next, the preparation of filter cartridges or flat filters is followed [8 - 10].

The measurement of the mass of the filter elements (before and after use) is performed with a precision electronic scale, in µg. The filter cartridges used may also be subjected to chemical analysis to determine the component chemical elements. The overall measurement of the particle emission is done over a representative period for the analyzed phenomenon, so the tested facility must be well known by the authorized personnel performing the experiment. It is defined as the individual measurement, the average value of the sum of the point measurements made on the axis or in the grid [8-10].

3. Determination of PM10 powders from the environment using sampling devices

At present, the national regulations represented by Law no. 104/2011 on ambient air quality limits the concentration of particulate matter PM10 from ambient to the daily limit value of 50 µg / m^3 over 24 hours [12].

As defined above, PM 10 represents the mass of particulate matter with a diameter of less than or equal to 10 µm. PM 10 inhalable dusts are harmful to human health because they can be deposited on the upper respiratory tract, causing severe traumatic lesions and illnesses.

Using the model of separator (analyzer) of 10 µm of dust in the environment, according to the requirements of SR EN 12341/2014, we aim to validate it for the current use in long-term determinations of PM10 powders in the environment.

3.1. Description of the sampling device

In order to determine PM 10 powder imissions, the procedure for experimenting with a sampling device, designated MVS6, is considered in accordance with the requirements of SR EN 12341/2014 „Air quality. Standardized gravimetric measurement method for determining the mass fraction of PM10 or PM2.5 of the suspended particles ”.

The MSV6 sample is used for the determination of particle immission (PM 10 fractions) in the surrounding air, the principle of determining the concentration of powders in the atmosphere being the determination of their mass, using the gravimetric method. The method complies with the requirements of Law no. 104/2011, as well as with international law and is used as a reference method in the technique of measuring suspended particles.

The particle sampling plant PM 10 in Fig. 2, a) and b) belongs to the company SVEN LECKEL INGENIEURBURG GMBH [12] and consists of the MSV6 analyzer (consisting of the separator, filter port, filter and separator support), the air meter, the vacuum pump and the connection hoses.

The principle of dust collection PM 10 from the ambient air with the mentioned installation takes into account the following: with the aid of the vacuum pump 7, the dust air is sucked in through the separator 1 with which the particles with dimensions larger than 10 µm are retained. The breathable particles below 10 µm are retained on the filter 3. The volume of air sucked through the filter is determined by means of the meter 6. The connection between the components of the system is made with the elastic hoses 5. The flow regulation Q = 6 m^3 / h is made by means of the valve 8. The separator has the role of selecting PM 10 particles from ambient air, according to the standard SR EN 12341/2014. In designing and constructing the separator, the basic dimensional elements of the respective standard were considered.

a. b.

Fig. 2. Particle sampling plant PM10 - MVS6 [12].

Circulation path through the air sampler sucked from the environment and the particle retainer MSV6 PM 10, shown in Fig. 3, and the overview of the apparatus, in Fig. 4.

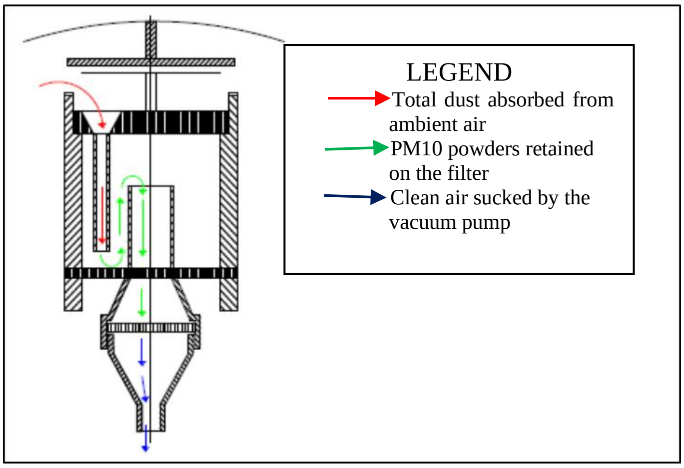

Fig. 3. Trajectory of the suction air from the environment through the MSV6 sampler [12]

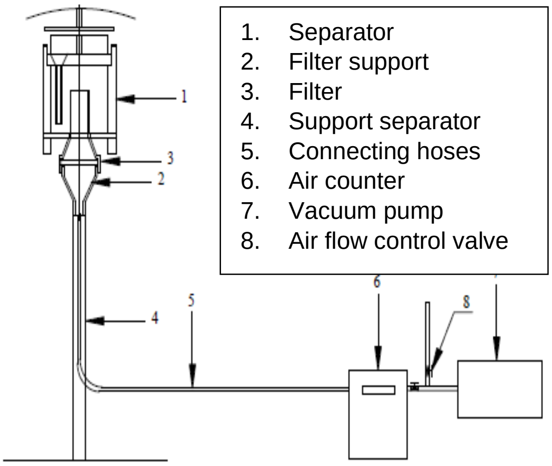

Fig. 4. Overview of the MSV6 sampler [12]

The separator consists of 8 suction tubes, with which the large particles are selected by the impact phenomenon. Particles aspirated from the ambient air stream are retained on the filter.

The impact velocity of the particles at the outlet of the suction tubes with diameter Ø 6.5 mm, results from the fundamental relation of the flow of a fluid through a pipe (Q = w • S), respectively (1):

$$w = \frac{Q}{S} = \frac{Q}{8 \times \frac{\pi \cdot d^2}{4}} \tag{1}$$

and the particle velocity is obtained: w = 6.28 m / s.

For the determination of the gravimetric concentration PM10 particles, glass fiber filters are used at which the mass is measured before and after sampling. The air meter together with the vacuum pump is metrologically checked and has a calibration certificate.

The gravimetric (C) concentration of PM 10 particles is determined by the relationship (2):

$$C = \frac{G_2 - G_1}{V} \ [\text{mg/m}^3] \tag{2}$$

where: G_2, G_1 represents the final and initial mass of the filter [mg]; V – inlet air volume [m^3].

3.2. Description of the validation method of the sampling device

According to SR EN 12341/2014 for the real evaluation of PM 10 powders from the total dust in suspension, 50% particles of 10 µm must be found in the separator and 50% on the filter.

In order to determine the dispersion of the particles (%) depending on their size in [µm], it is necessary to carry out dust from the ambient air with the MSV6 analyzer (having the technical characteristics: flow rate Q = 6 m^3 / h; pressure drop of the apparatus / installation: (147 - 196) · 10^5 MPa; electric vacuum pump 220 V, type ACOO with flow rate of 150 l / min; glass fiber filter, diameter Ø42 mm and pore size 0.8 µm), as well as preparation of the samples for reading the dimensions, respectively the dispersion of these particles under a Zeiss microscope. The powder taken from the separator is inserted into a clock bottle with alcohol water (25%), to prevent losses, and after homogenization, a drop is placed on the glass blade. After drying, cover the sample with a blade that is secured to the edge with a tape.

The prepared samples are analyzed under a microscope with a magnification power of about 1000 times. The values of the accumulated retentions and passes in / from the separator, respectively of the accumulated retentions on the filter, are presented in Table 1.

Table 1: The values of the accumulated particles retention and passage (%) in / from the separator and the accumulated particle retention (%) on the filter

Particle size PM x [µm]	The value of retensions (R_s) in separator q_i [%]	The value of cumulative retentions (R_{CS}) in separator Σq_i [%]	The value of cumulative passes from the separator (T_{CS}) 100 - Σq_i [%]	The value of retentions on the filter (R_F), q_i [%]	The value of cumulative retentions on the filter (R_{CF}), Σq_i [%]
20	12	12	88	0	0
19	11	33	67	0	0
18	10	43	57	0	0
17	9	52	48	0	0
16	8	60	40	0	0
15	7	67	33	0	0
14	6	73	27	0	0
13	5	78	22	0	0
12	4	82	18	0	0
11	3	85	15	0	0
10	2	87	13	2	2
9	2	89	11	2	4
8	2	91	9	2	6
7	2	93	7	3	9
6	2	95	5	3	12
5	2	97	3	6	18
4	1	98	2	8	26
3	1	99	1	12	38
2,5	1	100	0	26	64
1	0	100	0	36	100

4. Results and discussions

In order to determine the critical separation size, the graph of cumulative passage (TCS) from the separator [%] and the cumulative filter retention (RCF) of the powders, according to Fig. 5.

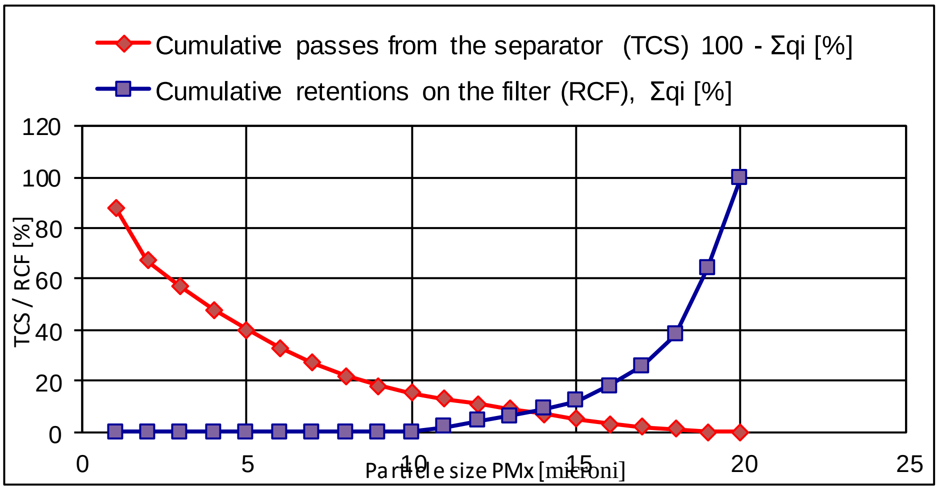

Fig. 5. The graph of the passage from the separator and the retention on the filter

The intersection of the two TCS and RCF curves is about 13 µm from the abscissa.
In another procedural stage comparative determinations are made with a reference device, namely the TEOM 1405, produced by the American company TEOM [11].
According to the standard of SR EN 12341/2014 (ch. 4.2, point B), for the comparison of a non-certified device with a reference device (certified), the following requirements must be met:

- the difference of the concentrations determined by the devices to admit values less than ± 10 µg / m^3, if the concentration in ambient environment Xi is less than 100 µg / m^3;
- the difference of the concentrations determined by the devices to admit values less than ± 10%, if the concentration in the ambient environment Xi is greater than 100 µg / m^3.

The values recorded correspond to concentrations less than 100 µg / m^3. The sampling height is 1.0 m above the ground. For testing, the reference device TEOM 1405 and the non-certified apparatus - MVS6 were considered.
Other equipment and materials used were: gas meter (0.06 - 6 m^3 / h); analytical balance (± 0.2 mg); vacuum pump (0.1 ÷ 120 l / min); filter funnel; desiccator; FM filters with pore diameter Ø0.8 µm; connecting hoses.
It should also be specified: the temperature of the environment with values between 8 ºC and 10 ºC; the relative humidity of the air between 50 - 80%.
The measurements were made in parallel with both devices, the distance between them being a minimum of 2 m. At each measurement, the operating rates for the TEOM 1405 and MVS6 respectively were determined.
The preparation of the filters for both devices must comply with the operational and system procedures of the industrial polluter on which the sampling is carried out. Certified filters were used for both devices. Simultaneous sampling was performed over a period of 24 hours. The results of the comparative measurements made are summarized in Table 2.

Table 2: Results of comparative measurements

Nr. crt.	x_i (TEOM 1405)	y_i (MVS6)
1	96	99
2	64	72
3	74	64
4	42	49
5	85	82
6	11	16
7	53	46
8	21	19
9	32	29

By processing the values in Table 2 with the help of graphical tools in Excel, we obtain the equation of the regression line (3), for estimating a linear model of connection between the results of comparative measurements, according to Fig. 6:

$$y = f(x) = 0.9633\,x + 1.7252 \quad (3)$$

where, x represents the concentrations determined with the reference device TEOM 1405, mg/m^3; y - concentrations determined with MVS6 [mg/m^3].

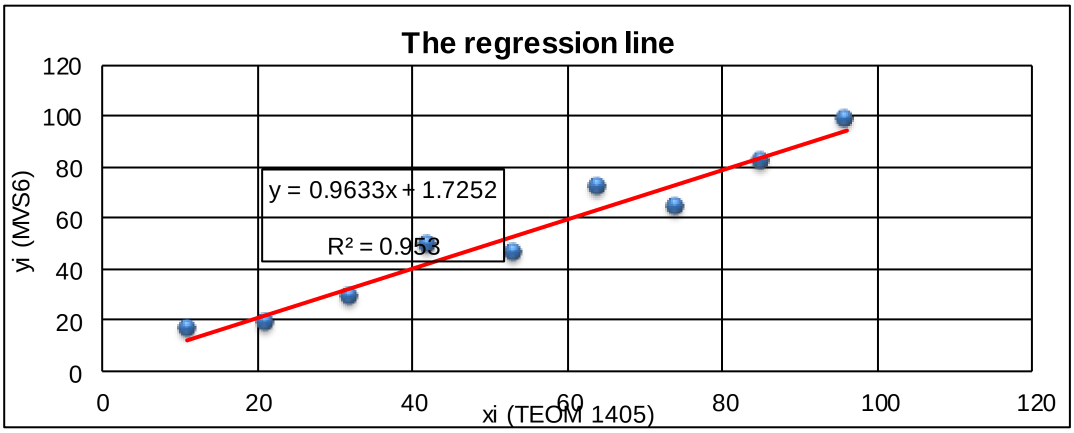

Fig. 6. Correlation between TEOM 1405 and MVS6 sampling devices.

The correlation coefficient (determination) d, results from Fig. 6:

$$d = R^2 \cdot 100 = 95.33\ \%.$$

According to SR EN 12341/2002, the correlation coefficient must be: R2 ≥ 0.95. But, since R2 = 0.9533 ≥ 0.95, it turns out that the requirement in the standard has been met.

5. Conclusions

In conclusion, the comparative determinations made between the TEOM 1405 reference device and the MVS6 analyzer are significantly correlated, respectively the value of the correlation coefficient validates the fulfilment of the requirements of the standard SR EN 12341/2014.

From here, the certification decision for the conformity of the MSV6 powder collection device is also used, in order to use it.

Therefore, the present work can constitute an application guide apt to lead to the possibility to use both for the assessment based on gravimetric measurement of the pollutant particles in the suspensions discharged in the environment and for the validation of the certification of any dust sampling devices compared to those considered as references or basis for meeting the technical-legislative standards and norms in force.

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