1. Introduction

Measurements of particulate matter emission from diesel engines create the heaviest problems during determination of emissions of harmful components of exhaust gases. The presently used experimental procedure is determined by the EPA [1], ISO [2] and ECE – R49 regulations accepted internationally more than 10 years ago. This procedure requires special measuring equipment with the exhaust diluting tunnel, which simulates the real conditions of mixing exhaust gases with surrounding air. Particulate matter emission is determined gravimetrically by thorough weighing of particles filtered from the mixture sampled from the tunnel. The above-mentioned regulations determine various features of the design of measuring equipment as well as the conditions of the measurement. However, many important details of the experimental procedure have not been accurately defined or are left free of choice. This reduces the possibility of comparing the results obtained in various centers and diminishes their repeatability and objectivity [4, 5, 6, 7, 8, 9].

These effects are the more pronounced, and the smaller are the measured emissions, e.g. during the investigations aimed for a significant reduction of emission as expected in the next future. The potential uncertainty of the measurement results may make it difficult to find correct conclusions during engine testing for particulate matter emissions (e.g. for homologation) or for research.

2. Measuring equipment and conditions of the measurements

The scheme of the measuring equipment used in investigations is presented in Fig.1. This equipment makes it possible to determine all the values and parameters necessary for measurements of the particulate matter emissions per hour or unit particulate emissions from engines, according to the ISO no CD 8178 – 1 or ECE – R49 standards. CO₂ concentrations were determined using the IR analyser (NDIR) and NOx (nitrogen oxides) excess was determined using the chemiluminescence instrument (CLA).

The tests were carried out on a 4-cylinder-loaded diesel engine with a direct injection system and compression ratio of 15.2. Its power was equal to 70 kW at 2600 RPM. Temperature of mixed...
gases in the tunnel did not exceed 52 °C. The sampled mixture flew through two serial PALL filters of 47 mm in diameter. These filters were weighted using the SARTORIUS microbalance with accuracy of 0.001 mg. All the measurements were fivefold.

The ECE-R49 experimental protocol was used and the particulate matter emission was calculated using the following formula:

\[
PM = m_f \cdot \frac{SR}{V_f} \cdot \frac{V_{sp}}{H_{11080} / H_{5007}} \quad [g/h]
\]

where:
- \(m_f\) - the mass of particles settled in the filters [g],
- \(SR\) - the exhaust gas dilution ratio in a tunnel, defined as a relation of the volume of the exhaust gas – air mix flowing in a tunnel to the exhaust gas volume, taken from the engine exhaust, contained in it,
- \(V_f\) - the volume of the exhaust gas – air mix directed from the diluting tunnel onto a particle filter [m³],
- \(V_{sp}\) - the exhaust gas flow in an engine exhaust [m³/h].

3. The effect of the determination method of the exhaust gases dilution ratio in the tunnel

The ECE-R49 protocol allows using several methods of determining the dilution ratio \(SR\) without any qualitative distinction between them. The result of particulate matter emission measurements may, however, depend on the chosen method. To evaluate the effect of the method, three methods from among those allowed by the ECE-R49 protocol were chosen for comparison:

I - the method based on measurements of \(CO_2\) concentration in exhaust gases in the diluting tunnel and in the sucked air;

II - the method based on the carbon balance and measurement of \(CO_2\) concentration in diluted exhaust gases in the diluting tunnel and in the sucked air; and

III - the method based on measurement of \(NO_x\) (nitrogen oxides) concentration in the engine exhaust gases in the diluting tunnel and in the sucked air.

Designations:

1 - measuring orifice plate to measure the exhaust gas and air flow; 2 - tunnel exhaust pipe; 3 - sampling probe to extract exhaust gas and air mix samples from the tunnel; 4 - pump to extract gas samples; 5 - \(CO_2\) analyser; 6 - \(NO_x\) analyser; 7 - exhaust gas dilution tunnel; 8 - mixing orifice plate to mix exhaust gas with air; 9 - exhaust gas inlet into the tunnel; 10 - adjustable diluting air intake; 11 - heat exchanger; 12 - microbalance to weigh particulate matter in filters; 13 - adjustable throttle; 14 - set of filters; 15 - diluting air chemical filter; 16 - rotameter; 17 - vacuum pump; 18 - laboratory gasometer; 19 - diluting air dust filter; 20 - fan; 21 - laminar flowmeter; 22 - three-way valve; 23 - adjustable exhaust gas throttle; 24 - \(CO_2\) analyser; 25 - \(NO_x\) analyser; 26 - stop-watch; 27 - fuel meter; 28 - three-way valve; 29 - Askania micromanometer; 30 - sampling probe to take samples of exhaust gas flowing into the tunnel; 31 - brake; 32 - tested Diesel engine; 33 - fuel tank

Fig. 1. Schematic of equipment details for determination the particulate matter emission in diesel engine exhaust
The comparative calculations were based on the investigations carried out under the following conditions of engine work:
1. idle work at 600 RPM,
2. maximum torque at 1600 RPM,
3. external power at 2100 RPM ($N_e = 79$ kW),
4. rated conditions of engine work ($n = 2600$ RPM, $N_e = 71$ kW).

The mixture volume sampled from the tunnel onto the filters $V_f$ was not varied and was equal to 200 dm$^3$. Various settings of the tunnel for the particular conditions of the engine work were chosen in such a way as to obtain the determined exhaust dilution ratios within the range of $SR = 8$ to 17 for all 3 methods used.

Fig. 2 presents the results of SR calculations using the above-presented methods. Under the same conditions of engine work, all applied methods should give theoretically the same values of SR (tunnel settings are invariable for a given revolutions). The arithmetic mean indicates that the highest SR values are obtained using the I-st method and the lowest – using the III-rd one. This regularity is not valid for $n = 2600$ RPM only, but in this case the scattering of results is highest.

Fig. 2. Dilution ratio SR determined by means of methods I, II and III under different engine running conditions and fivefold measurement repetition

$I$ - Standard deviation; $•$ - Arithmetic average

The presented data shows that the general scattering of SR values is similar for all methods and conditions of engine work. Only in the case of the III-rd method and the maximal revolutions a more significant scattering of the results was obtained.

Fig. 3 presents the values of the relative error, made during determination of the dilution ratio under the particular conditions of engine work.

Generally, under all conditions of the engine work the III-rd method gave the highest error. The relative SR error is the highest at the idle run and maximal revolutions. An increase of the SR error with the engine revolutions under load may be clearly observed.

Fig. 4 presents the results of measurements and calculations of the particulate matter emission $PM$, for various methods of determination (I, II and III) and for various conditions of engine work.

$PM$ values, their standard deviations and the arithmetic mean are also shown on the histograms of 5 subsequent measurements (their deviations). As it may be expected the lowest $PM$ values were found for small engine revolutions and the idle run, irrespective of the method of SR determination. Parallel to the higher SR values obtained using the I-st method the particulate matter emission is the highest. The similar regularity may be observed for the smallest SR and $PM$ values. It may indicate that the dilution ratio of exhaust gases in the tunnel significantly affects calculation of $PM$ values from the used formula.

The value of the $PM$ relative error, presented in Fig. 5 seems to be very interesting.

The highest relative error, of the order of 30 – 50 % was observed for $PM$ determinations at small revolutions and idle run of the engines. It may be related to the lower repeatability of combustion and lower particulate matter emission, leading to higher scatter of
the smaller mass of particles accumulated on the measuring filters. This result is, however, not fully consistent with the data presented in Fig. 3, where the SR relative error was not significantly dependent on particular conditions of engine work. This fact may be explained by taking into consideration the formula used in PM calculations. One should also remember that $V_f$ was invariable during measurements, while scattering of $V_{sp}$, the flow intensity of exhaust gases was relatively low. We may assume that both parameters do not significantly affect the measured PM value. For this reason the influence of the disturbance of the SR determination method on results of PM evaluation may be also caused by the scattering of mass of the particulate matter $m_f$ settled on filters. The relative error of the mass settled on filters, determined under various conditions of engine work is presented in Fig. 6.

It may be seen that scattering of the measured particle masses is highest at idle run of the engine, when the relative error reaches 40%. Under these conditions the particulate matter emission per hour is small, so at $V_f = \text{const.}$ the mass of particles accumulated on the measuring filters is also smaller than under other conditions of engine work. This leads to the increase of the scatter of both values, the measured mass and particulate matter emission. When the engine is loaded, this error is much smaller, but it increases with engine revolutions.

**Fig. 6. Relative error of the particulate mass $m_f$ collected on the filters at fivefold measurement repetition. Measuring conditions as given on Fig. 4**

Based on the above-presented results of measurement one may state that the measured value of particulate matter emission depends clearly on the method used for determining the exhaust dilution ratio. Each of these methods bears different levels of experimental and calculation errors, dependent on the conditions of engine work. Thus, comparisons of the results of particulate matter emission determinations obtained in various centres (laboratories) should be limited to the same method of SR determination. The preferable method seems to be the 1-st method, based on measurements of $CO_2$ concentrations, when the calculated values of PM, the particulate matter emissions are highest at the lowest scatter of their values.

**4. The effect of the dilution ratio in the diluting tunnel**

The ratio of exhaust gas dilution is one of the parameters that may be generally freely set during the measurement of the particulate matter emission measurements down to the value of $SR = 4$ [2]. The theory states that changes of the dilution ratio under fixed engine work conditions should not affect the measured value of the particulate matter emission. The investigations of the influence of this parameter on the result of measurements of particulate matter emissions were carried out during the engine work under the maximal torque $M_{\text{max}} = 420 \text{ [Nm]}$ and revolutions of 1600 RPM. The dilution ratio SR was determined using the 1-st method based on determining the carbon dioxide $CO_2$ concentration. Measurements were performed in two series of experiments of different choice of the gas mixture volume sampled from the diluting tunnel onto the filters $V_f$.

In the first series the same volume of gases $V_f = \text{const.} = 200 \text{ dm}^3$ was sampled from the tunnel at various exhaust-gas dilution ratios. The dilution ratio SR fluctuated roughly between 5 to 21 and was equal to 4.95; 10.1; 15.7 and 21.6 (average values for 5 measurements). The following values of SR were achieved by changing the volumes of exhaust gas and air supplied in the diluting tunnel. The results of measurements are shown in Fig. 7 together with the average standard deviation for 5 repetitions.

**Fig. 7. The effect of the exhaust gas dilution ratio SR on the particulate matter emission per hour PM, at fixed engine settings and constant volume of gases sampled onto the filters $V_f$.**

I – Standard deviation; • – Arithmetic average

**Fig. 8. The effect of dilution of the exhaust gases on the relative error of determination of the particulate matter emission $\Delta PM$, at fixed engine settings and invariable volume of gases sampled onto the filters $V_f$.**
The obtained results do not allow drawing any substantial conclusions concerning the effect of dilution ratio SR on the measured values of the particulate matter emission. Both Fig. 7 and 8 show that with increasing dilution of exhaust gases the relative error of PM measurements increases strongly, which may make it difficult to draw conclusions.

As it was assumed for this series the volume of gases sucked off the filters was maintained constant for all SR values. Under such conditions the increase in SR caused a mass of particles deposited on the filters was obviously diminishing. This relationship is shown in Fig. 9. Within the SR changes between about 5 and 21, the loading of filters with particles diminishes fourfold. In consequence the relative error of PM determination (equivalent to the error of determining the mass on the filters) increases as the dilution ratio SR (presented in Fig. 8) increases. This seriously enhances the uncertainty of conclusions, which might be determined based on the investigations carried out at \( V_f = \text{const.} \)

For these reasons another series of measurements was carried out in such a way that the volume of the gas mixture sampled from the diluting tunnel onto the filters was changing along with variations of SR. The volume \( V_f \) was varied with SR so as to obtain a similar mass of particles on the filters independent of the dilution ratio. These masses varied within the limits of 0.8 – 1.0 mg. As it may be seen in Fig. 10, the range of the variations in particle masses did not exceed 20 %. The exhaust-gas-dilution ratio was similarly changing as in the first series of measurements, i.e. within the limits of 3 – 21. Its values were equal to: 3.2, 8.5; 15.5 and 20.3, respectively. Under such conditions the volume taken onto the filters varied between about 50 to 400 dm\(^3\) (Fig. 10). The results of the particle matter emissions measurements carried out under these conditions are presented in Fig. 11.

It may be clearly seen that within the applied limits of SR the variation of the average particle matter emission per hour \( PM \) practically does not depend on the exhaust-gas-dilution ratio in the tunnel \( SR \).

Investigations carried out in both series lead to hints important for the technique of measuring the particle matter emissions using the exhaust-gas-diluting tunnel. During the routine measurements of particulate matter emission one should pay attention to the proper choice of the volume of the mixture of gases \( V_f \) sampled onto the filters from the diluting tunnel and onto the loading of the filters with particles. The ratio of exhaust gases dilution usually may be set quite arbitrarily. In order to obtain a good accuracy of PM measurements, one should try to obtain similar loading of the measuring filters in the subsequent measurements and to gain a sufficient mass of particles on the filters. These conditions should create a criterion of the choice of the mixture sampled from the diluting tunnel, \( V_f \), at various ratios of exhaust dilution in the tunnel.
5. The effect of conditioning the filters applied in the particle matter emission measurements

One of the measurement conditions, which are not sufficiently precisely defined, is the preparation of filters, called their conditioning. According to the EPA [1], ISO [2] and ECE - R49 [3] standards these filters should be conditioned both before the application and before weighing in the pure state and after deposition of particles and before the second weighing. According to the ISO standard, before the investigation the pure filters should be conditioned for a period of time not shorter than one hour. In this time they should be kept in an air-conditioned chamber in a closed but unsealed Petri dish. After removal from the chamber the filter must be used within eight hours. Otherwise, the conditioning procedure must be repeated.

Conditioning the filter after depositing of particles (and before weighing) should be carried out for at least two hours but not longer than 80 hours. The ISO standard does not precisely state other conditions of the conditioning procedure. The EPA and ECE - R49 recommendations give more detailed data concerning both temperature of the procedure and humidity of air. According to the ECE - R49 protocol temperature in the conditioning chamber and during the weighing of filters should be kept within 20 ± 30 ± 6 °C, at relative humidity of air within 35 ± 55 ± 10 %. Duration of conditioning is consistent with the ISO recommendations described above.

From among those recommendations the most doubtful are the rather wide limits conditioning time. They vary in measurements of various authors, which makes any comparison less reliable.

For this reason the conditioning time was taken as a main variable in the investigations presented below.

As it was mentioned before in the investigations the PALL filters (EMFAB TX 40 HJ 20 WW), meeting the requirements of the ISO and ECE standards and widely used in measurements of particulate matter emissions by diesel engines were used in our studies.

At the beginning the changes in mass of two pure filters (new) during conditioning were measured before use in particle emission measurements. Fig. 12 presents variations in mass of these filters for different periods of conditioning.

As it may be seen, there is an increase of the mass of filters over the whole period of conditioning, up to about 100 hours; although, these changes of mass are not big and remain within several hundred parts of a milligram. These results show, however, that the filters are not completely non-hygroscopic. In such a situation, because of the precision of the measurements of particulate matter emission, it is very important to strictly follow the same protocol of conditioning (time, temperature and humidity of air) of both pure filters before use and loaded filters. Moreover, it seems that conditioning time should be close in both cases.

In the second series of investigations the loaded filters were taken into consideration. The influence of conditioning time on the mass of deposited particles and the particle emission per hour were determined.

Filters loaded with particles during the engine work under three different experiment conditions were studied:
- idle run of the engine, at \( n = 600 \) RPM,
- partial load of the engine, at \( n = 1600 \) RPM and 50 % of the maximal torque \( M_n = 210 \) Nm;
- full load of the engine, at \( n = 2600 \) RPM and \( M_n = 270 \) Nm (on the external characteristics of the engine).

Substantially different conditions of engine work were chosen because of the real possibility of different behavior of particles on the filters at various states of the engine work. For similar reasons for each of the above described states of the engine, measurements were carried out on the filters loaded with a “small mass” of particles of 0.1 ÷ 0.35 mg or a “big mass” of 0.9 ÷ 1.8 mg. These ranges correspond roughly to the mass of particles on filters from one test point in the 13-phase test acc. to ECE – R49 and the whole 13-phase test. Such data may be helpful in evaluation of the influence of the procedure used in the 13-phase test on the potential accuracy of the measurements of particle emission. It is important because the ECE – R49 protocol allows application of separate pairs of filters for each test point (all together 13 pairs of filters less loaded) or one pair of filters for the whole 13-phase test, much more loaded.

The results of this series of investigations are shown in Figs. 13 - 14.

From the data presented in Figs. 13A and 13B it results that masses of particles accumulated on filters behave differently during conditioning of filters, depending on the origin of the particles, i.e. in dependence on the engine work conditions, under which they have been collected. It seems to be related to different chemical composition and structure of the particles deposited on the filters, for instance, a different ratio of the solid phase (soot) and volatile phase forming the particles.
In the case of idle run (curve 1), the mass of particles diminishes both for “small” and “big” loading of the filters. This data indicates that the most intensive changes of mass appear at about 80 hours of conditioning for idle run. The fastest loss of mass occurs in the first period of conditioning during the first 10-20 hours.

Also, the particles settled on the filters during the work of the partly-loaded engine (curve 2 on fig. 13 A and 13B) show the tendency to diminish their mass during conditioning. In this case the highest loss occurs during the first period of conditioning.

In the case of particles settled at full load of engine (curves 3 in figures) and “small” loading of the filter, there is a reverse phenomenon – the mass on the filter increases with time of conditioning. On the other hand, the mass of a “big” mass of particles practically does not change during filter conditioning.

The particulate matter emission per hour $PM \ [\text{g/h}]$, shows a similar variation (Fig. 14). Our investigations show that the measurements of the particulate matter emissions may be severely erroneous, depending on conditioning time of filters loaded with these particles. Undoubtedly, it decreases the possibility of comparing the results of particulate matter emission measurements carried out in various centres. A more precise definition of conditioning time by the rules of the standards of these measurements seems necessary.

Generally, the less mass accumulated on filters, the greater may be the error related to conditioning time. The presented results of measurements indicate other sources of the differences in results of particulate matter emission measurements carried out, for instance, using the 13-phase ECE - R49 test with one pair of the filters for the whole test or with 13 separate, less loaded filters (i.e. for each stage of the test). One should note here that clearly the highest error in the particle emission might occur for measurements carried out at idle engine work. Under such conditions the changes of particles mass during conditioning are, moreover, reverse than for other conditions of the engine work.
6. The influence of isokineticity of sampling gas from the diluting tunnel

Both the ISO 8178 and ECE - R49 standards do not give precise requirements in this respect. The character of gas flow at the inlet to the probe and the literature data [10] indicate that this question is not negligible for accuracy and comparability of the results of measurements.

The criterion of evaluating isokineticity by taking samples from the diluting tunnel is the ratio of flow rates of the mixture of air and exhaust in the tunnel \( V_t \) (m/s), to the flow rate of this mixture \( V_g \) (m/s) in probe sampling gas onto the measuring filters, i.e. \( V_t/V_g \). Theoretically, the ideal case of sampling from the diluting tunnel is the equality \( V_t = V_g \) (or \( V_t/V_g = 1 \)). In reality, meeting this condition is not always possible, for instance, because of changing flow rates during the 13-phase test acc. to the ECE-R49 standard. It may be a source of additional errors of exhaust particulate emission measurement. When \( V_t \neq V_g \), the concentration of particles at the probe inlet may be different from the average concentration in gas flowing in the tunnel. It is caused by various images of gas jet deviations at the edge of the probe, dependent on the \( V_t/V_g \) ratio. When \( V_t > V_g \), then at the probe inlet there proceeds “centrifugation” of solid particles. So, a more concentrated gas will flow to the filters, and the particles emission will be apparently higher. The reverse phenomenon occurs when \( V_t < V_g \).

The influence of sampling isokineticity on the value of the measured particle emission was determined for a probe in the form of a tube, the outlet of which was directly opposite to the flow of gas in the tunnel (see scheme in Fig. 15). It is one of the solutions recommended by ISO 8178. 5 values of the \( V_t/V_g \) ratio were chosen for studies: 0.5; 1.0; 1.5; 2.0 and 2.5. These values were achieved in two ways, either by:
1. change of the flow rate of gas in the diluting tunnel (choking of flow), or by:
2. use of probes, of two different internal inlet diameters \( ds \) (alternatively):
   - for \( V_t/V_g < 2 - d_s = 8 \text{ mm} \)
   - for \( V_t/V_g > 2 - d_s = 16 \text{ mm} \).

The diameter of the diluting tunnel was equal to 160 mm. The flow rate of gases \( V_t \) in the tunnel was determined by flow intensity measurement using the standard orifice. On the other hand, the flow rate in the probe was determined by measuring the time of taking a definite volume sample, determined by using a laboratory gas meter.

The results of these measurements are presented in Fig. 15. As it may be expected the greater the ratio of \( V_t/V_g \), the greater is the measured value of the particulate emission (at fixed engine settings).

This relationship is roughly linear. Thus, greater deviations from isokineticity of gas sampling from the tunnel may cause serious errors in the values of the particles emission. This data shows that the ratio of \( V_t/V_g \) for a particular probe should not exceed about 0.8 \div 1.2 (the shadowed region in Fig. 15), in order to limit the error derived from anisokineticity, e.g. to \( \pm 2 \% \).

7. Conclusions

1. The method of determining the dilution ratio \( \text{SR} \) in the diluting tunnel significantly affects the value of the calculated particle emission. The choice of this method is important for objectivity of particulate matter emission measurements. Our investigations show that the most objective results are obtained using the method based on the measurement of \( \text{CO} \) concentration.

2. To improve the accuracy of the particulate matter emission measurements using various dilution ratios in the tunnel, it is necessary to pay attention to accumulating masses of particles that should be similar and possibly large for subsequent measuring points (in the case of the ECE R49 test this note is applicable only to the method of many filters).

3. Depending on the engine work conditions, the mass of loaded filters changes during conditioning in a different way within 80 hours. To increase the accuracy and comparability of measurements, the more detailed and narrow limits of conditioning time loaded filters before weighing should be defined (for instance \( 5 \pm 0.25 \) hours).

4. To obtain objective results of the particle emission measurements one should assure the isokinetic conditions of gas sampling from the diluting tunnel. The measuring error of particle emission may be equal to \( \pm 2 \% \), when the isokineticity is kept within the limits of 0.8 \div 1.2 for the probe used in the presented investigations.
8. References

[1] "Code of Federal Regulations", U.S.A. Environmental Protection Agency. Gaseous and Particulate Exhaust Test Procedure. 1990, Vol. 40, Part 86.

[2] "RIC Engines - Exhaust emission measurement, Part 1. Test bed measurement of gaseous and particulate exhaust emissions from RIC Engines". Norma ISO, nr CD 8178 - 1, 1992.

[3] "Emission of diesel engines", U.N. Economic Commission for Europe ECE), Regulation No 49 - (z poprawkami i uzupełnieniami do r. 1992).

[4] BLACK, F., HIGH, L.: "Methodology for Determining Particulate and Gaseous Diesel Hydrocarbon Emissions". SAE Technical Paper 1979, Nr 79 0422.

[5] FRISCH, L. E., JOHNSON, J. H., LEDDY, D. G.: "Effect of Fuels and Dilation Ratio on Diesel Particulate Emissions", SAE – Transactions, 1979, Paper Nr 79 0417.

[6] LACH, G., WINCKLER, J.: "Specific Problems of Sampling and Measuring Diesel Exhaust Emissions". SAE – Transactions, 1988, nr 88 1763.

[7] WALDENMAIER, D. A., GRATZ, L. D., BAGLEY, S. T., JOHNSON, J. H., LEDDY, D. G.: "The Influence of Sampling Conditions on the Repeatability of Diesel Particulate and Vapor Phase Hydrocarbon and PAH Measurements": SAE – Transactions 1990, nr 90 0642.

[8] KITTELSON, D. B., JOHNSON, J. H.: "Variability in Particle Emission Measurements in the Heavy - Duty Transient Test", SAE – Transactions, 1991, nr 910738.

[9] BASTENHOF, D.: "Exhaust Gas Emission Measurements – A Contribution to a Realistic Approach", 21 Kongres CIMAC, 1995, Interlaken, ref. nr D43.

[10] LIPKEA, W. H., JOHNSON, J. H., VUC, C. T.: "The Physical and Chemical Character of Diesel Particulate Emission-Measurement Technique and Fundamental Considerations". SAE – Transactions, 1978, Paper 780108