

## Inter-laboratory correlation exercise on a light-duty diesel passenger vehicle to verify nano-particle emission characteristics by Korea particle measurement program<sup>†</sup>

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### Abstract

The Light Duty Inter-Laboratory Correlation Exercise (ILCE) final report, performed with the ‘PMP Golden Vehicle’ at nine laboratories in the EU, Korea and Japan to demonstrate repeatability and reproducibility of the particle number concentration emissions measurement techniques proposed by the Particle Measurement Program (PMP), was released in 2007. The ILCE was conducted by the Korea Particle Measurement Program (KPMP) with a domestic diesel passenger vehicle equipped with a diesel particulate filter (DPF) between three certification laboratories and the research center of an automotive manufacturer to meet future regulations (EURO 5 and EURO 6) of particle number concentration for light-duty vehicles in early 2008. This research focused on measuring the particulate matter emission (particle number and mass) levels of a representative light-duty diesel passenger vehicle during new European driving cycle (NEDC) mode to analyze the repeatability and reproducibility between laboratories in Korea. From the ILCE test results in Korea, the mean total particle number concentration levels ranged from  $5.43\text{E}+10$  #/km to  $1.58\text{E}+11$  #/km and  $0.0003$  g/km to  $0.0036$  g/km for particle mass. Repeatability between participating laboratories ranged from 32% to 66% for particle number, 11% to 70% for particle mass; the reproducibility level was 46% for particle number, and 66% for particle mass emission.

**Keywords:** PMP; KPMP; Inter-laboratory correlation exercise; Particulate matter; Repeatability; Reproducibility

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### 1. Introduction

Diesel-powered engines have strong points by which engine power output, fuel economy, and greater durability than spark ignition engines are increased. In addition, they can reduce emissions such as hydrocarbons and carbon dioxide [1-3].

In addition, diesel engines are widely used in heavy-duty trucks, buses, and engine generators as they have merits in performance and emissions. Despite these advantages, the smoke emissions and particulate matter (PM) from diesel engines are mainly looked badly upon by general society and environmental researchers [4-7].

From the health perspective, PM emitted from diesel engines affects adverse health effects. Many studies have found that the PM of the atmosphere is an important factor with respect to mortality and morbid-

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ity [8-11].

Particulate matter concentration levels from state-of-the-art diesel engines tend to have exhaust emissions much lower than older engines. This is a result of improvements in engine design, quality of combustion, and atomization of fuel by high pressure injection as well as improved fuel and oil formulations. However, many studies have shown that the reduction in particle mass for diesel engines is not consistently accompanied by decreases in total particle number concentration [12-16].

To alleviate these problems, the international Particle Measurement Program (PMP) has progressed into a collaborative program operating under the auspices of the UNECE/GRPE Group with the heightened involvement of the governments of the UK, Germany, Sweden, France and lately, also Japan and Korea. Its function is focused on the development of a new approach to the measurement of PM in vehicle exhaust emissions, which may be used to replace or complement the existing mass based system used for regulatory purposes [17-19]. The activity of the PMP is to recommend to the regulatory authorities a new measurement system for application during the type approval testing of light-duty vehicles (LDV) and heavy-duty engines for particulate emissions. Nine laboratories participated in the international (ILCE) program for LDV, commencing in November 2004 and finally completed in June 2006 for the setting up of the regulatory procedures [20-22].

The Korea PMP (KPMP) and the National Institute of Environmental Research (NIER) reported the measurement and evaluation results of particle emission from the golden passenger vehicle recently. In this context, ILCE for a diesel passenger vehicle in Korea was performed at several laboratories using the established PM measurement facilities. The four participants in the ILCE were classified into two categories, three of the participants were vehicle emission certification laboratories and the other was the research center of an automotive manufacturer. A reference diesel passenger vehicle similar to the Peugeot 407 was selected by the KPMP and named as the 'Korea Golden Vehicle': a 2.0-liter diesel vehicle meeting EURO 4 emission regulations. In addition, a golden particle measurement system (GPMS) was transferred to each of the laboratories to provide repeatability and reproducibility data under the domestic emission evaluation environment.

The inter-laboratory exercise was performed ac-

ording to the inter-lab guide whose procedure included both filter based particulate mass measurements and real-time particle number measurements of the LDV under the transient conditions of the new European driving cycle (NEDC) on a chassis dynamometer [23]. Regulated gaseous emissions were also measured in parallel with particle emissions using established regulatory measurement facilities under each laboratory condition. The purpose of this research focuses on the evaluation of PM emission characteristics, repeatability, and reproducibility between representative participating laboratories under the KPMP in preparation for the future LDV particle number emission standard which will be proposed by the international PMP from EURO 5 (number:  $6.0E+11$  #/km, mass: 0.0045 g/km).

## 2. Experimental apparatus and test procedures

### 2.1 Vehicle, driving cycle, and preconditioning

The test vehicle for ILCE was supplied to the KPMP by one of the vehicle manufacturers in Korea as shown in Fig. 1. The vehicle had a turbocharged 2.0-liter common-rail direct injection (CRDI) diesel engine with an oxidation catalyst and diesel particulate filter (DPF), meeting EURO 4 emission regulations. The detailed technical information of the vehicle is summarized in Table 1. To minimize fuel variations during test periods, diesel fuel whose properties were similar to the PMP Phase 3 reference fuel was supplied from the same filling station using one batch preparation. Its specifications are given in Table 2.

The vehicle driving cycle was the NEDC, which includes four ECE segments repeated without inter-



Fig. 1. Test vehicle in the ILCE test.

ruption, followed by one extra urban driving cycle (EUDC) segment at high speed driving modes of the maximum 120 km/h. The road load coefficient was provided to each laboratory and was confirmed by coast-down test.

Preconditioning on the vehicle was similar to standard diesel emission test ( $3 \times$  EUDC); however, 20 minutes of steady cruising speed about 120 km/h proceeded for the purpose of eliminating the contaminated materials in the exhaust and sampling system and for reducing variability during the inter-lab tests. To monitor the soot loading levels in the DPF, differential pressure sensor signal before and after the DPF was continuously measured.

Meanwhile, engine control logic for regeneration function was restrained by the manufacturer to avoid the possibility of active regeneration during the test. Two of the test facilities, laboratory C and D, experienced high DPF soot loading that showed both high PM emissions and high differential pressure signal; hence, an additional 130 km/h for 30 minutes on the chassis dynamometer was performed before the vehicle preconditioning.

The vehicle emission measurement over NEDC was repeated at least 3 times at each laboratory to establish the statistical data set on the PM and regulated emissions variability.

Table 1. Vehicle descriptions in ILCE test.

Vehicle/Emission category	Diesel with DPF/EURO 4
Number of cylinders	4
Aspiration	Turbocharged
Fuel supply system	Common rail DI
Capacity (cc)	1,991
Inertia (kg)	1,700
Weight empty (kg)	1,581
Transmission	4 speed automatic
Aftertreatment	CPF (DOC+DPF)

Table 2. Test fuel specifications.

Property items	ULSD Fuel
Cetane number	55.9
Distillation temperature (°C) : 90 vol %	337.0
Density@15°C (kg/m <sup>3</sup> )	826.9
Ignition point (°C)	55
Ash content (weight%)	0.007
Sulphur content (mg/kg)	6
10% residual carbon (weight%)	0.02
Lubrication@60°C (μm)	336

## 2.2 Particle mass and number emission measuring equipments

A full flow constant volume sampler (CVS) exhaust dilution tunnel system was used for measuring the particle emission characteristics. The flow rate of dilute exhaust gas through the CVS tunnel of each test laboratory ranged from 12 ~ 20 m<sup>3</sup>/min (A: 12 m<sup>3</sup>/min, B: 20 m<sup>3</sup>/min, C: 16 m<sup>3</sup>/min, D: 15 m<sup>3</sup>/min) at standard reference conditions. The dilution air used for the primary dilution of the exhaust in the CVS tunnel was first passed through a primary high efficiency particulate air (HEPA) filter type, charcoal scrubbed and then passed through a secondary HEPA filter type.

The mass of particulate material emitted by the reference vehicle was measured using a system comprising of a particulate mass sampling and filter holder assembly. A cyclone as a pre-classifier (50% cut point at 2.5 μm) was used to measure particle number. Laboratory D was not equipped with the cyclone for particle sampling in the CVS tunnel. The number of particles was determined using the golden particle measurement system (GPMS) which was recommended by the PMP and supplied by laboratory A. The volatile particle remover (VPR) provided heated dilution, thermal conditioning of the sample aerosol, and secondary dilution for cooling and freezing of the sample evolution prior to entry into the particle number counter (PNC). In this manner, the particle sampling system was required to draw a sample from the CVS, size classify it, transfer it to a diluter, and condition the sample, so that only solid particles were measured and passed suitable concentration of those particles to the counter.

Fig. 2 shows the schematic diagram of the GPMS. The volatile particle remover comprised a primary particle number diluter (PND<sub>1</sub>), an evaporation tube (ET) and a secondary PND. The PND<sub>1</sub> is a rotating disc diluter (MD19-2E) with hot dilution set at 150°C and HEPA filtered dilution air. After the first diluter, the sample was further divided into two flows. The flow was conducted to the ET held at a constant temperature of 300°C. The VPR was designed to achieve greater than 99% reduction of 30 nm C<sub>40</sub> (tetracontane) particles and greater than 80% solid particle penetration at 30, 50 and 100 nm particle diameter sizes. The PNC\_GOLD was TSI 3010 condensation particle counter (CPC) with the lower cut-off modified to 23 nm by the manufacturer [23–25].

Furthermore, the engine exhaust particle sizer (EEPS) was also used in laboratory A to analyze the particle size distribution during the vehicle emission test. The regulated gaseous emissions were measured during all the tests in accordance with the current R83 [17]. A Horiba MEXA-7200D instrument was used for HC, CO, NO<sub>x</sub>, and CO<sub>2</sub> measurements at laboratory A, C, and D, while a Pierburg AMA-2000 emission analyzer was used at laboratory B.

Total hydrocarbon emissions were measured by heated flame ionization detector (FID); CO and CO<sub>2</sub> were determined by non-dispersive infra-red (NDIR) and NO<sub>x</sub> were measured using a chemiluminescence analyzer. To avoid the possibility of particle generation in the coupling tube between tailpipe and CVS by the high exhaust temperature, the muffler size was modified for metal to metal contact and a Teflon tube was applied to the CVS transfer line.

### 2.3 Statistical analysis for Lab to Lab variation

When the data sets on particle number concentration and mass of NEDC mode were acquired, statistical evaluation was performed to find the similarity and difference of the emission results between the laboratories [18]. The experiments in a laboratory that measure the emissions of  $n$  NEDC cycles of a vehicle comprise the sample (where  $x_1, x_2, \dots, x_n$  are the results). The sample has a mean value ( $x_m$ ) and a standard deviation ( $s$ ). Deviation ( $s^2$ ) is the square of the standard deviation  $s$ . These can be calculated as follows;

$$x_m = \frac{x_1 + x_2 + \dots + x_n}{n} = \frac{\sum_{i=1}^n x_i}{n} \quad (1)$$

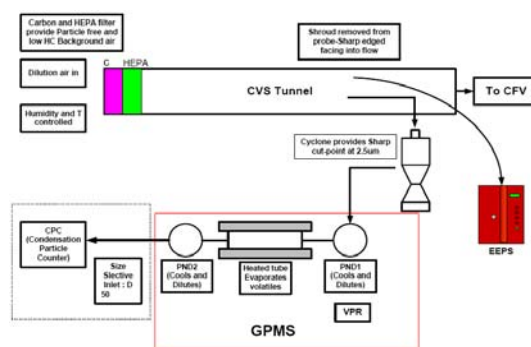


Fig. 2. Schematic diagram of GPMS.

$$s^2 = \frac{\sum_{i=1}^n (x_i - x_m)^2}{n-1} \quad (2)$$

The standard deviation  $s$  is an index of how closely the individual data points cluster around the mean. This variability is due to random variations of the properties measured and to the fluctuations of some factors (such as measurement equipment, the operators and environmental conditions). When repeat tests were performed in the same laboratory within a short period of time and with these factors as constant as possible, then the variation is called '*within laboratory variability*'. The ratio of the standard deviation  $s$  over the average value ( $x_m$ ) is called the *Coefficient of Variance (CoV= $s/x_m$ )* and is referred to as the *repeatability* of the specific laboratory. The variability of the mean results from different laboratories is called the '*inter-laboratories variability*'. The ratio of the standard deviation of the mean results of the labs ( $\sigma_i$ ) over the average value ( $x_i$ ) is called the *Coefficient of Variance (CoV= $\sigma_i/x_i$ )* and is referred to as the *reproducibility*. An approach based on this has been used to establish the similarities and differences between sets of data from the same vehicle at different laboratories. Meanwhile, it should be noted here that the reproducibility of the particle number concentration is based on the use of the same instrumentation (GPMS) in all laboratories. From the 2007 final test results by PMP, the mean particle number results were from 1.00E+10 #/km to 2.50E+11 #/km and 0.0002 g/km to 0.0006 g/km for particle mass of the PMP Golden Vehicle. In addition, repeatability ranged from 12% to 72% for particle number, 12% to 66% for particle mass, and the reproducibility level was 31% for particle number, 35% for particle mass.

### 3. Experimental results and discussion

Each laboratory performed  $3 \times$  NEDC tests, including additional variation test at laboratory A for confirming the functional check on the DPF and various emissions at the end of ILCE time table. As mentioned previously, all the tests were preceded by 20-minute periods of operation at 120 km/h and  $3 \times$  EUDC for preconditioning. In addition, a minimum soak period of 6 hours was included between successive tests in accordance with R83.

Regulated gaseous emissions were measured at the

same time under the established emission measurement environment of each laboratory condition. Since DPF diesel vehicles emit low levels of PM, the tested vehicle was always the last to be conditioned in the test facility and had the emission test conducted first in each day to reduce the contamination of the CVS system.

**3.1 Comparison of time resolved particle concentration in NEDC mode**

Fig. 3 shows the three consecutive times resolved particle emission traces measured by CPC during NEDC in laboratory A. The levels of the PM number emissions show a close relationship with vehicle driving conditions, such as engine speed and load, engine coolant and catalyst temperatures, etc. Particle number concentrations of the DPF diesel vehicle were highest during the cold start phase whose level was in the E+3 #/cm<sup>3</sup> order. Particle emissions were gradually reduced after the first transient cycle and were maintained below E+1 #/cm<sup>3</sup> except during accelerating at the 120 km/h condition.

The order of total particle number ranged from 1.00E+11 #/km to 2.31E+11 #/km and its level increased over twofold with the soot loading in the DPF. The accumulated particle emission trace showed that almost all the particles were emitted within the second hill of the ECE 1 mode (1-195 s).

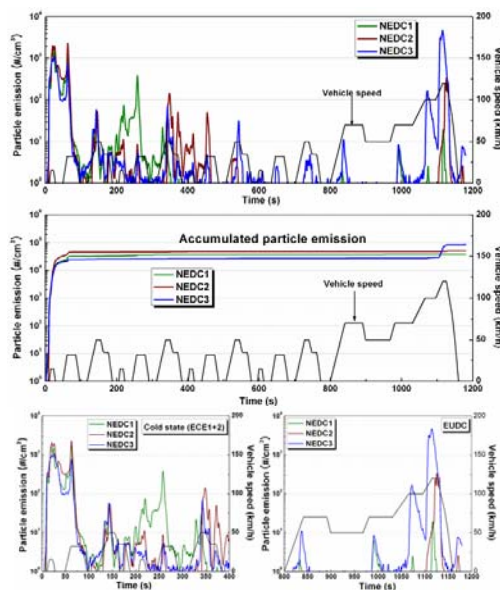


Fig. 3. Time resolved particle emission characteristics for NEDC mode at Laboratory A.

In the case of diesel fueled vehicles, aftertreatment system with DPF has the potential to reduce particle number and mass emission effectively. Fig. 4 shows nano-particle size distribution and number concentration with EEPS at laboratory A. This equipment was employed to analyze the PM in the range between 6 to 500 nm during time resolved cycle operation [26]. PM size distributions in an internal combustion engine are generally classified into nucleation, accumulation, and coarse modes, as distinguished by particle diameter. The nucleation and accumulation modes have particles with diameters less than about 50 nm and from 50 to 1000 nm respectively [28]. In the figure, the distinctive PM size distribution with the DPF equipped diesel vehicle was bi-modal, consisting of the nucleation and accumulation modes.

As vehicle speed shifted from a low state (ECE) to a high state (EUDC), more accumulation mode particles were emitted than the nucleation mode. However, nucleation mode particle concentration increased at a cold state (below 400 s) compared to EUDC mode. There are five basic mechanisms by which particles can be deposited into a filter, especially, nucleation mode particles are collected into the filter by diffusion. The filtration efficiency ( $E_D = 2Pe^{-2/3}$ ) due to diffusion is a function of the dimensionless Peclet number ( $Pe = d_p U_0 / D$ ), where  $Pe$  is inversely proportional to the diffusion coefficient ( $D$ ) which increases with temperature [29]. Therefore, nucleation mode particles are not deposited on the filter surface and emitted through porosity inside the DPF because the diffusion velocity of the particle which is proportional to the diffusion coefficient is dropped in case of insufficient temperature of DPF during acceleration at cold state.

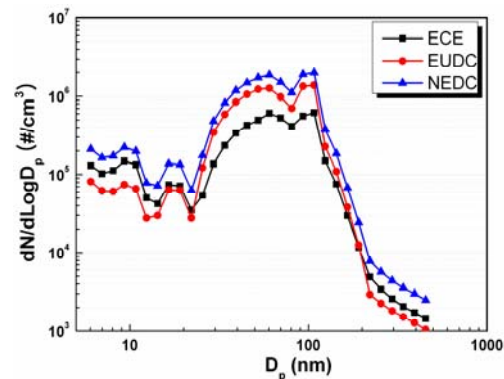


Fig. 4. Averaged particle size distribution of ECE, EUDC, and NEDC mode at Laboratory A.



During the high speed period of EUDC, volatile and sulfate particles formed under high temperature were removed by evaporation tube heated with 300°C but HC particles with greater molecular weight formed by engine oil combustion and rich operation were generated with self nucleation particle by cooling process into the CVS tunnel.

Much of the volatile particles, however, can be removed into the evaporation tube. Emitted solid particles are comprised of both elemental carbons from particulate which is incompletely oxidized and very low volatility HCs. The low volatility HCs may be derived from lubricant species both stored on the DPF through adsorption with carbon and evaporated during regeneration, or materials that slip through the DPF when filtration efficiency and exhaust temperature are low as well as condensed in the exhaust system. These are then released in response to high thermal temperature in the exhaust [18, 30].

Moreover, solid particles are formed in the combustion process and usually agglomerated; but, most are oxidized under very high temperature conditions.

The large concentration of the diesel DPF vehicle under the EUDC condition (especially, 120 km/h state) could be explained by the high temperature inside the particulate filter (measured temperature: 330°C at 120 km/h) causing the natural regeneration (partial oxidation of loaded soot) of particles during

high speed operating condition, such that the accumulation mode is emitted after the surface growth of particles [31, 32].

Figs. 5, 6, and 7 show the comparison of Lab B, C, and D's particle emission levels, respectively. There were two kinds of sampling system variations between the laboratories; one is the dilute flow rate of

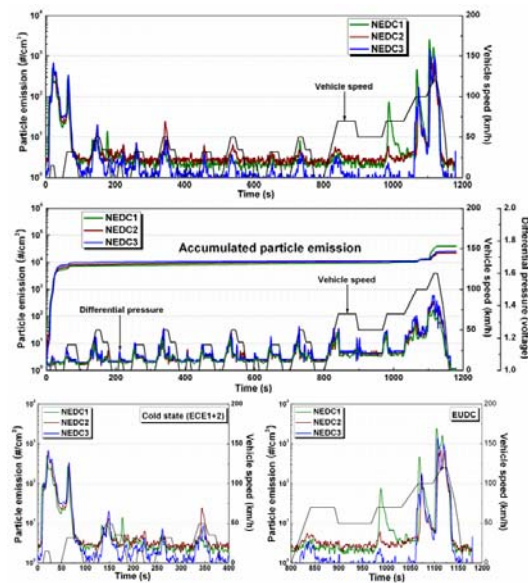


Fig. 5. Time resolved particle emission characteristics for NEDC mode at Laboratory B.

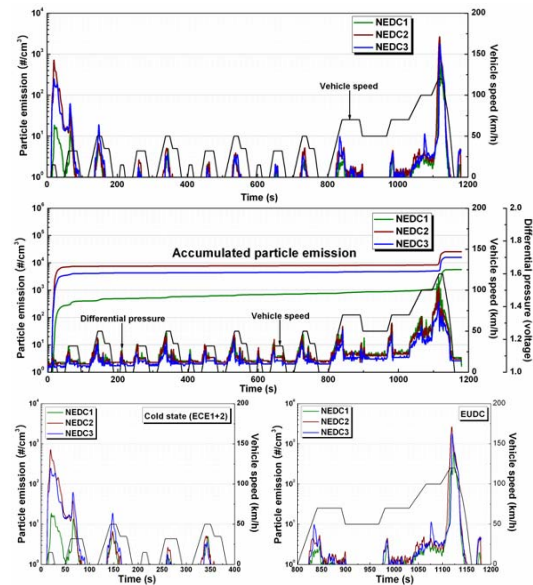


Fig. 6. Time resolved particle emission characteristics for NEDC mode at Laboratory C.

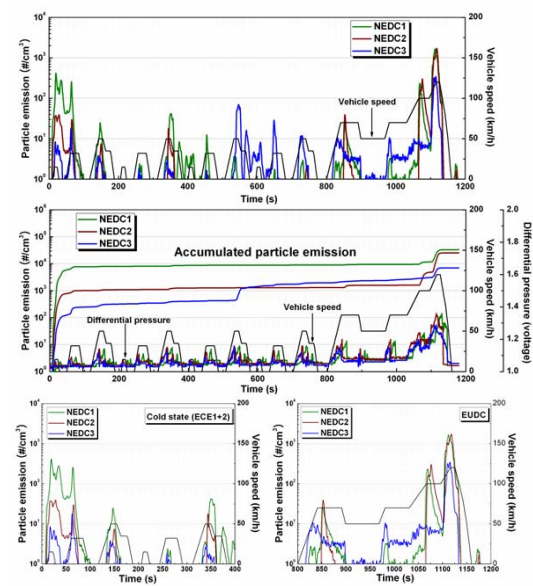


Fig. 7. Time resolved particle emission characteristics for NEDC mode at Laboratory D.

CVS and the other is the sampling system of the pre-classifier cyclone. The same vehicle possibly produced the significantly different results on reproducibility of the nano-particle measurement when tested in CVS systems operating at different dilution ratio, but the alternative sampling condition still needs refinement. The CVS flow rate recommended by PMP of 12 m<sup>3</sup>/min deviated from 15 m<sup>3</sup>/min to 20 m<sup>3</sup>/min in the laboratories. Moreover, the type-approval sampling system of the cyclone pre-classifier for limiting the large particle size over 2.5 μm was not installed in Lab D. Meanwhile, the background levels of particle numbers in the CVS were measured approximately 0.5–2 #/cm<sup>3</sup> which had influence on the particle number and mass characteristics.

However, it needs to be clarified that the use of a cyclone with full flow sampling would be optional within ILCE in the KPMP protocol.

From the cumulative particle emission levels at Lab A and B, whose order of magnitude was E+4 #/cm<sup>3</sup>, a similar particle number concentration of the last test in Lab A was measured in Lab B's first vehi-

cle operation. However, E+3 ~ E+4 #/cm<sup>3</sup> of particle number was distributed in Lab C and Lab D due to the additional 130 km/h steady cruising speed to promote natural soot regeneration in the DPF. The regeneration criteria during the preconditioning were assessed by the differential pressure signal installed in the DPF at 120 km/h of the EUDC segment.

### 3.2 Comparison of repeatability and reproducibility for measured emissions between laboratories

The results of the mean particle number and mass emissions, as well as repeatability and reproducibility from the 'Korea Golden Vehicle', are presented in Fig. 8. The mean NEDC emissions of particle numbers from the Golden Vehicle ranged from 5.43E+10 #/km to 1.58E+11 #/km. The repeatability of the particle number emissions between laboratories ranged 32% to 66%, while reproducibility level was 46%. The mean particle mass ranged from 0.0003 g/km to 0.0036 g/km, repeatability for particle mass ranged from 11% to 70%, and reproducibility level was 66%.

The main difference between the tests in these laboratories was preconditioning. Lab C and D used 20-minute periods of operation at 120 km/h plus 3 × EUDC after driving 20 minutes at 130 km/h to remove the particles through natural regeneration during preconditioning. This effect is believed to relate to reduced particle concentration levels during cold start at the next test and can be confirmed with the particle number concentration levels during cold start shown in Figs. 6 and 7. The comparison of the particle number concentration levels and mass at each laboratory is summarized in Table 3.

The normalized gaseous emission levels from the test vehicle between laboratories are presented in Fig. 9. Mean total hydrocarbon emissions ranged from 0.003 g/km to 0.012 g/km with a mean value of 0.007 g/km. Repeatability for THC ranged from 9% to 24% and reproducibility level was 52%. As observed, the CO and NOx emissions from the test vehicle were also low and varied from laboratory to laboratory in the range 0.02 to ~0.05 g/km compared to the EURO 4 standards (CO: 0.5 g/km, NOx: 0.25 g/km).

The repeatability for NOx emissions was generally acceptable level, with *CoVs* generally below 10% and the reproducibility level at ≤ 10%. Mean carbon dioxide emissions were about 200 g/km and its level between laboratories was similar.

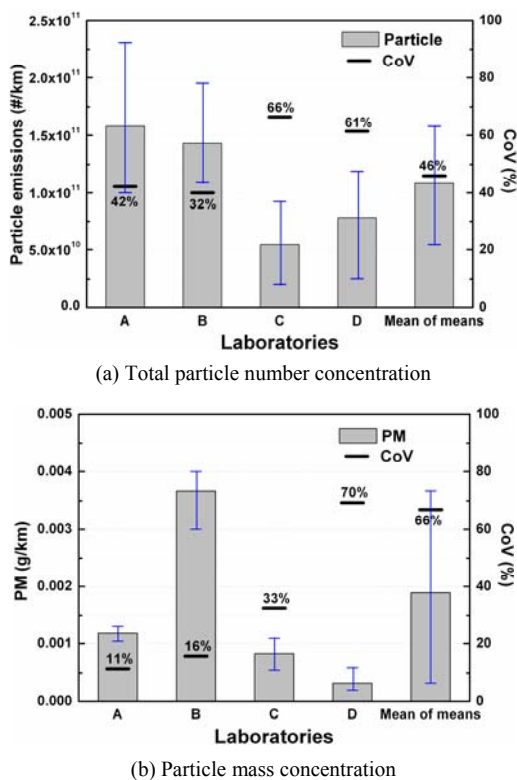


Fig. 8. Comparison of ILCE results on total particle number and mass emissions for NEDC mode.

Table 3. Comparison of particle number and mass emissions.

Laboratory	Particle number and mass emissions							
	NEDC1		NEDC2		NEDC3		Mean values and Standard deviations	
	Particle (#/km)	PM (g/km)	Particle (#/km)	PM (g/km)	Particle (#/km)	PM (g/km)	Particle (#/km)	PM (g/km)
A	1.00E+11	0.0010	1.43E+11	0.0010	2.31E+11	0.0010	1.58E+11±6.68E+10	0.0010±0.0001
B	1.95E+11	0.0040	1.09E+11	0.0040	1.25E+11	0.0030	1.43E+11±4.57E+10	0.0036±0.0006
C	2.04E+10	0.0010	9.23E+10	0.0010	5.04E+10	0.0010	5.43E+10±3.61E+10	0.0010±0.0003
D	1.19E+11	0.0005	9.02E+10	0.0002	2.53E+10	0.0002	7.81E+10±4.78E+10	0.0003±0.0002

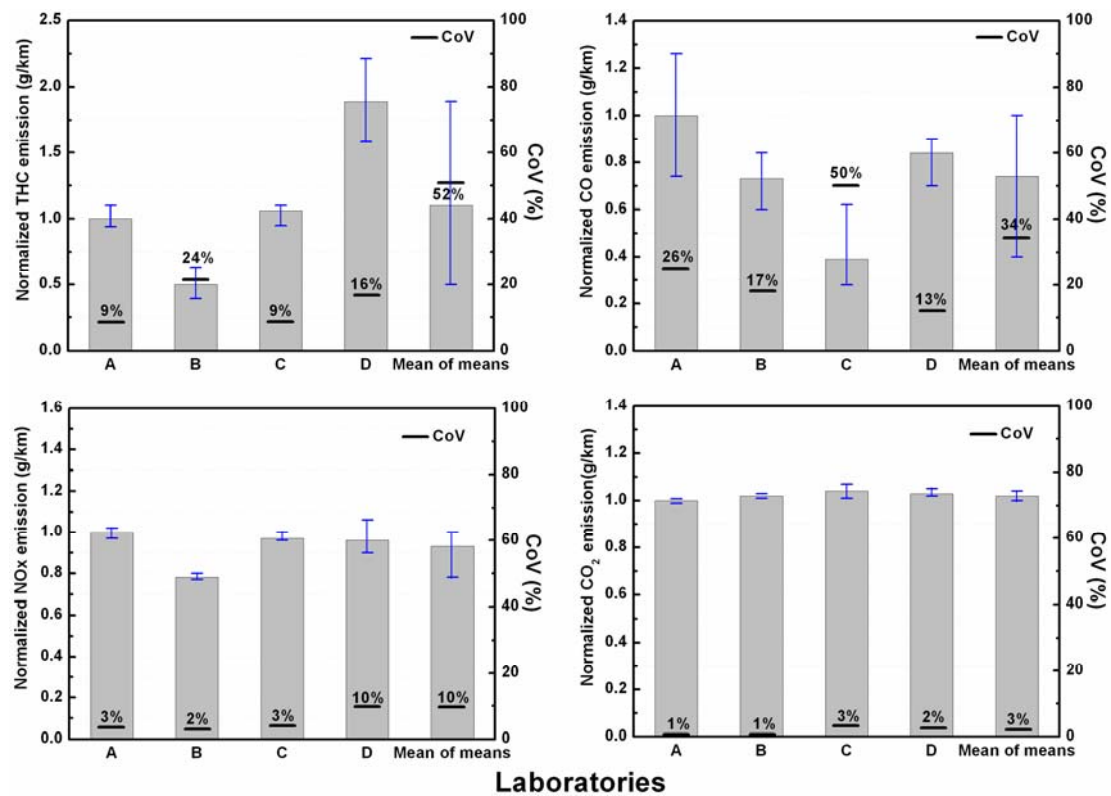


Fig. 9. Normalized gaseous emission levels at different laboratories.

Repeatability and reproducibility for CO<sub>2</sub> was reliable results with *CoV*s below 3%. In general, gaseous emissions reproducibility across the participating laboratories showed different levels due to the variation of averaged THC and CO emissions except for NO<sub>x</sub> and CO<sub>2</sub> emissions.

#### 4. Conclusions

The particle measurement systems of Lab B, C, and D were not exactly consistent with particle measurement system of Lab A. However, the Korean LDV particulate measuring project had been successfully conducted to acquire a comprehensive database on particle number concentration, size distribution, and



particle mass. Mean total particle number concentration levels ranged from  $5.43\text{E}+10$  #/km to  $1.58\text{E}+11$  #/km and  $0.0003$  g/km to  $0.0036$  g/km for particle mass within EURO 5 particle number regulation. Repeatability for particle number ranged from 32% to 68% and from 11% to 70% for particle mass at all the participating laboratories.

Reproducibility level was 46% for particle number and 66% for particle mass. Repeatability and reproducibility for gaseous emissions showed reliable results except for THC and CO. Compared with the PMP results, particle number and mass concentration levels accomplished in the Korea particle measurement project showed a similar trend. In conclusion, it is important to improve the standardization of particle number measurement system according to the proposed international PM analysis procedures.

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