Final Report of the Proficiency Testing in Vehicles Emissions 11th Round Otto Vehicles (RHE)





PROGRAMA DE ENSAIOS DE PROFICIÊNCIA DO INMETRO

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1. Introduction

By means of scientific studies, we are increasingly looking for solutions to assess air pollution that has been increasing the occurrence of respiratory diseases and decreasing the population quality of life. Motor vehicles are considered to be potential agents causing this type of pollution worldwide. Otto cycle cars are the vast majority of our private vehicle fleet. In the specific case of Brazil, these cars are Flex, that is, they can be fueled with gasoline and ethanol. Specifically in this search to improve measurements, we evaluated the emission measurements for the Hydrous Ethanol Reference Fuel (RHE) and, in addition, we sought to evaluate new parameters with their formulas being developed in the period of this PT round, which was the case of NMOG.

Due to regulatory and accreditation bodies requirements there is an increasing need to perform better measurement of pollutant gases. Besides that, due to constant emissions limits reductions, measurement methods should adequate to new needs. Pollutants analysis is one of the most delicate items of a vehicle or engine emission test. In this sense, the execution of Proficiency Testing (PT) in vehicles emissions aims to evaluate the performance of laboratories in determining the amount of compounds present in vehicle emissions, providing subsidies for the identification and solution of analytical problems and contributing to the harmonization of measurement results in the country, besides being a tool for data generation that can support the preparation of new regulations.

The objectives of this PT Scheme were:

- 1) To determine the performance of laboratories for the proposed tests;
- 2) To monitor the ongoing performance of the analytical vehicle emissions laboratories;
- To increase the confidence of the measuring emission process of the vehicle emission laboratories;
- 4) To improve continuously the measurement techniques of vehicle emissions laboratories.

2. Materials and Methods

2.1. Test Item

The test item was a vehicle supplied by FCA (FIAT Chrysler Automóveis Brasil) company with the following characteristics: Cronos Precision Model, marsala red color, identification code 8AP359A23JU000072, 1.8 etorq motor 16V flex and automatic 6-speed transmission.

Each participant laboratory used the Hydrous Ethanol Reference Fuel (RHE) as established in the PT protocol.

2.2. Metodology

In this round, the following tests were evaluated, according to the current versions of the respective documents:

Urban Cycle	ABNT NBR 6601:2012 Standard: THC, CH4, NMHC, CO, CO ₂ ; NO _x ABNT NBR 7024:2017 Standard: Urban Autonomy
Road Cycle	ABNT NBR 7024:2017 Standard: THC, CO, CO ₂ , Road Autonomy and Combined Autonomy
Aldehydes	ABNT NBR 12026:2016 Standard
Unburned Ethanol	ABNT NBR 15598:2016 Standard
Idling Speed CO	ABNT NBR 10972:2010 Standard
NMOG	Minutes of the 226 th meeting of the technical commission of emissions laboratory accreditation – item 3.2.2.4.

The laboratories reproduced the deceleration curve in dynamometer informed by CETESB. Laboratories drained the fuel of the tank to refuel with 30 L at minimum and to perform all the tests planned in this PT. Participants should follow the test flow chart presented in figure 1 when performing the tests and preferably start the tests at 25 °C temperature, aiming minimizing cold start effects in results.





Participants sent, obligatorily, **3** (three) measurements for each parameter, with exception to the parameters the protocol consider optional, otherwise, their results would not be evaluated. The results

of FCA (FIAT Chrysler Automóveis Brasil) to be considered were those referred to the tests performed in the beginning of the cycle (Y_1) .

3. Test Item Integrity

FCA (FIAT Chrysler Automóveis Brasil) laboratory performed stability tests in the beginning and at the end do the cycle – first analysis (Y_1) and second analysis (Y_2). It was verified if there were significant statistical differences between measurements of the 9 components of urban cycle: THC, NMHC, CH₄, CO, CO₂, NO_x, aldehydes, unburned ethanol and urban autonomy.

The Wilcoxon test was used to verify the hypothesis that two dependent data sets were extracted from the same population. This test should be used to the detriment of the paired t-test, when it is not possible to assume the normality of both data sets or in cases where there are small samples. In the Wilcoxon test, the original values are replaced by posts as follows:

Before	<i>x</i> 1	<i>x</i> ₂	 x_N
After	<i>y</i> 1	\mathcal{Y}_2	 y_N
d _i	<i>d</i> 1	d_2	 d_N
$ d_i $	$ d_1 $	$ d_2 $	 $ d_N $
post	Ν	2	 1
Source: Dimci/	Dimat/Lafia		

Table 1 - Statistics and p-values

In which $d_i = y_i - x_i$. The posts are obtained from absolute values of d_i . The hypotheses to be tested are:

 $\begin{cases} H_0 \end{pmatrix}$ no difference between the both sets of data H_1 there are differences between both sets of data

The test statistic is defined below:

 V_s = sum of the positively signaled posts

If $V_s \le c_1$ or $V_s \ge c_2$, H_0 is rejected, in which c_1 and c_2 are such that $P(V_s \le c_1 | H_0) = \frac{\alpha}{2}$ and $P(V_s \ge c_2 | H_0) = \frac{\alpha}{2}$.

Figures 2 to 10 refer to the boxplot for the test item evaluation.









Figure 10 – Total Aldehydes Boxplot





As noted, for the Unburned Ethanol component, there was no intersection of the boxes and there is a gap between the initial and final measurements, while in the others there appear to be minor discrepancies. The small number of repetitions (true replicates) makes inspection more difficult.

Then, the stability hypothesis was tested between such measurements, first analysis (Y_1) and second analysis (Y_2), using the Wilcoxon test, also known as the Mann-Whitney test. Table 2 below summarizes the result of the application for the data set between the two samples at the 5% significance level.

Component	p-value
CO (g/km)	0.596
CO ₂ (g/km)	0.377
THC (g/km)	0.216
CH4 (g/km)	0.476
NO _x (g/km)	0.212
NMHC (g/km)	0.216
Total Aldehydes (g/km)	0.596
Unburned Ethanol (g/km)	0.052
Urban Autonomy (km/L)	0.377
Source: Dimci/Dimgt/Lafig	

Table 2 - Statistics and p-values for the Wilcoxon test

All results were the same, with p-value greater than 0.05. Thereby, it can be assured that, to a level of confidence of 95 %, there are no difference statistically significant between the mean and the sample data can be considered as coming from the same population. Thus, the vehicle maintained its integrity during the performance of this Proficiency Test.

Due to data confidentiality, once FCA (FIAT Chrysler Automóveis Brasil) is also participant of this PT, these results were not presented.

4. Statistical Analysis of Participants' Results

4.1. *z*-Score

For the participants' results evaluation, it was followed one of ABNT NBR ISO/IEC 17043:2011 criteria, *z*-score (distance measurement related of the laboratory measurement result in relation to the PT designated value), that was calculated according to equation 1.

$$z_i = \frac{x_i - X}{\hat{\sigma}} \tag{1}$$

Where:

 x_i is the mean measurement result of the ith participant;

X is the PT designated value;

 $\hat{\sigma}$ is the standard deviation for the proficiency testing, that in this round was established as described in ISO 13528:2015 standard, that is, a robust standard deviation based on participants' results.

The interpretation of *z*-score is presented as follows:

 $|z| \le 2,0$ - indicates "satisfactory" performance and generates no signal;

2,0 < |z| < 3,0 – indicates "questionable" performance and generates a warning signal;

 $|z| \ge 3,0$ - indicates "unsatisfactory" performance and generates an action signal.

4.2. Results Preview and Outliers Detection: Boxplot (Specific for NMOG measurement)

The boxplot is a graph used to preview the data set distribution. It consists of five measures: 1st quartile (Q1), 3rd quartile (Q3), median (Q2), minimum value (IL) and maximum value (SL). In addition, the boxplot can be used to evaluate outliers. Thus, the minimum and maximum values are calculated according to the equations below:

The diagnosis for detecting outliers is given by the following rule:

If the obtained value> SL or the obtained value <IL, the obtained value is considered an outlier; If IL \leq the value obtained \leq SL, the value obtained is not considered an outlier.

5. Assigned Values

According to available procedures for the establishment of designated values by ABNT NBR ISO/IEC 17043:2011, the designated values of this PT were calculated by statistical methods described in 7.7 item of ISO 13528:2015 standard, that is, consensus values from participant results.

ISO 13528:2015 standard describes the robust analysis involving employment of the A algorithm for the calculation of designated value and standard deviation. The robust statistical techniques are used to minimize the influence that extreme results can have on estimates of mean and standard deviation.

Initially, all values object of the analysis (values sent by participants) were put in ascending order. Next, robust values and standard deviation of these data were denoted by (x^*) and (s^*) . Initial values of (x^*) and (s^*) were calculated according to equations below:

$\mathbf{x}^* = \mathbf{x}_i \text{ median} \tag{4}$

$$s^* = 1,483 \text{ x median } |x_i - x^*|$$
 (5)

 $(x^*) e(s^*)$ values were updated as follows. It was calculated: $\delta = 15e^*$

 $\delta = 1,5s^{\star} \tag{6}$

For each x_i (i = 1, 2, ..., p), it was calculated:

$$x_{i}^{*} = \begin{cases} x^{*} - \delta, & \text{if } x_{i} < x^{*} - \delta \\ x^{*} + \delta, & \text{if } x_{i} > x^{*} + \delta \\ x_{i}, & \text{otherwise} \end{cases}$$
(7)

new values of $(x^*) e(s^*)$ should be calculated from the equations:

$$x^* = \sum x_i^* / p \tag{8}$$

$$s^{*} = 1,134\sqrt{\sum \left(x_{i}^{*} - x^{*}\right)^{2} / (p-1)}$$
(9)

Where the summation is over *i*.

The robust estimation (x^*) and (s^*) can be obtained by an iterative calculation, i.e., by updating the values of (x^*) and (s^*) several times using the modified data, until the process converges. Convergence may be assumed when there is no change from one iteration to the next in the third significant figure of the robust standard deviation and of the equivalent figure in the robust average.

The results out of 2 standard deviation intervals after the robust average and robust standard deviation calculation were considered as outliers and new assigned value as well new robust standard deviation results were calculated, removing those outliers.

Tables 3, 4, 5 and 6 present the assigned values and the robust standard deviation for all parameters, including all PT participants, as well as the new robust average and standard deviation values after removal of the outlier results.

Where the obtained value is the result of each parameter measurement per participant.

Each participant in this report is identified by the last numeric characters of its identification code in tables, graphs and texts.

Parameter	Assigned Value	Standard deviation	Outliers (Participants)	Recalculated Assigned value	Recalculated standard deviation
CO (g/km)	2.613	0.230	-	-	-
CO ₂ (g/km)	153.9	4.2	-	-	-
THC (g/km) 0.350		0.020	-	-	-
NO _x (g/km)	0.388	0.035	-	-	-
NMHC (g/km)	0.320	0.019	-	-	-
CH4 (g/km)	0.032	0.003	-	-	-
Total Aldehydes (g/km)	0.07885	0.01625	-	-	-
Unburned Ethanol (g/km)	0.46996	0.09402	-	-	-
Urban Autonomy (km/L)	9.06	0.24	-	-	-

Table 3 – Assigned Values and standard deviations of the PT – urban cycle emissions.

Table 4 - Assigned Values and standard deviations of the PT – road cycle emissions.

Parameter	Assigned Standard Value deviation		Outliers (Participants)	Recalculated Assigned value	Recalculated standard deviation	
CO (g/km)	1.141	0.089	-	-	-	

Parameter	Assigned Value	Standard deviation	Outliers (Participants)	Recalculated Assigned value	Recalculated standard deviation
CO ₂ (g/km)	91.4	2.7	-	-	-
THC (g/km)	0.129	0.006	-	-	-
Road Autonomy (km/L)	15.35	0.49	-	-	-
Combined Autonomy (km/L)	11.11	0.31	-	-	-

Table 5 - Assigned Values and standard deviations of the PT – idling Speed CO.

Parameter	Assigned Value	Standard deviation	Outliers (Participants)	Recalculated Assigned value	Recalculated standard deviation	
Idling speed CO %vol	0.1839	0.0398	-	-	-	

Source: Dimci/Dimqt/Lafiq

Table 6 - Assigned Values and standard deviations of the PT – NMOG.

Parameter	Assigned Value	Standard deviation	Outliers (Participants)	Recalculated Assigned value	Recalculated standard deviation
NMOG (g/km)	0.328	0.025	-	-	-

Source: Dimci/Dimqt/Lafiq

6. Results Dispersion

In the presented graphs for all tested parameters, a continuous black line represents the assigned value. The blue and red lines, respectively, are representations of Ref \pm 1s and Ref \pm 2s, where "Ref" is the assigned value (robust average) and "s" is the robust standard deviation.

6.1. Urban Cycle Emissions

Figures 11 to 19 graphically present the means and robust standard deviation of the reported urban cycle emission results by the participants for each analyzed parameter.



Figure 11 - Scatter plot of the participants' measurement results for CO – urban cycle.

Figure 12 - Scatter plot of the participants' measurement results for CO₂ – urban cycle.





Figure 13 - Scatter plot of the participants' measurement results for THC – urban cycle.

Figure 14 - Scatter plot of the participants' measurement results for NMHC – urban cycle.



Source: Dimci/Dimqt/Lafiq



Figura 15 - Scatter plot of the participants' measurement results for NO_x – urban cycle.

Figure 16 - Scatter plot of the participants' measurement results for CH₄ – urban cycle.



Source: Dimci/Dimqt/Lafiq



Figure 17 - Scatter plot of the participants' measurement results for Total Aldehydes- urban cycle.

Source: Dimci/Dimqt/Lafiq

Figure 18 - Scatter plot of the participants' measurement results for Unburned Ethanol – urban cycle.



Source: Dimci/Dimqt/Lafiq



Figure 19 - Scatter plot of the participants' measurement results for Urban Autonomy – urban cycle.

Through the graphics, it can be seen that:

CO (g/km): 13 participants reported results within the Ref \pm 1s interval. Participants 57, 54, 93, 84, 67, 7 and 90 reported results within the Ref \pm 2s interval and participant 24 presented the greatest dispersion.

CO₂ (g/km): 16 participants reported results within the Ref ± 1s interval. Participants 3 and 85 reported results within the Ref \pm 2s interval, participants 7 and 64 presented results out of Ref \pm 2s interval and participant 80 presented the greatest dispersion.

THC (g/km): 15 participants reported results within the Ref \pm 1s interval. Participants 85, 3, 41 and 88 reported results within the Ref \pm 2s interval, participant 7 presented results out of Ref \pm 2s interval and participants 64 and 84 presented the greatest dispersions.

NMHC (g/km): 15 participants reported results within the Ref ± 1s interval. Participants 85, 3, 41 and 88 reported results within the Ref \pm 2s interval, participant 7 presented results out of Ref \pm 2s interval and participants 64 and 84 presented the greatest dispersions.

NO_x (g/km): 14 participants reported results within the Ref ± 1s interval. Participants 85, 67, 57, 3 and 80 reported results within the Ref \pm 2s interval, participant 41 presented results out of Ref \pm 2s interval and participants 64 and 41 presented the greatest dispersions.

CH₄ (g/km): 14 participants reported results within the Ref \pm 1s interval. Participants 85, 12, 3, 54 and 90 reported results within the Ref \pm 2s interval, participant 7 presented results out of Ref \pm 2s interval and participants 64 and 60 presented the greatest dispersions.

Total Aldehydes (g/km): 14 participants reported results within the Ref \pm 1s interval. Participants 24, 54, 30 and 90 reported results within the Ref \pm 2s interval, participant 61 and 85 presented results out of Ref \pm 2s interval and participant 88 presented the greatest dispersion.

Unburned Ethanol (g/km): 14 participants reported results within the Ref \pm 1s interval. Participants 54, 90, 61 and 7 reported results within the Ref \pm 2s interval, participant 80 and 67 presented results out of Ref \pm 2s interval and participant 88 presented the greatest dispersion.

Urban Autonomy (km/L): 14 participants reported results within the Ref \pm 1s interval. Participants 85, 12, 3 e 54 reported results within the Ref \pm 2s interval, participants 7 and 64 presented results out of Ref \pm 2s interval and participant 80 presented the greatest dispersion.

6.2. Road Cycle Emissions

Figures 20 to 24 graphically present the means and robust standard deviation of the reported road cycle emissions results by the participants for each analyzed parameter.



Figure 20 - Scatter plot of the participants' measurement results for CO - road cycle.

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Source: Dimci/Dimqt/Lafiq

Figure 22 - Scatter plot of the participants' measurement results for THC – road cycle.



Source: Dimci/Dimqt/Lafiq





Figure 24 - Scatter plot of the participants' measurement results for Combined Autonomy- road cycle.



Through the graphics, it can be seen that:

CO (g/km): 15 participants reported results within the Ref \pm 1s interval. Participants 57, 90, 84, 7 and 67 reported results within the Ref \pm 2s interval and participant 24 presented the greatest dispersion.

CO₂ (g/km): 15 participants reported results within the Ref \pm 1s interval. Participants 12, 3, 61 and 7 reported results within the Ref \pm 2s interval and participant 85 presented results out of Ref \pm 2s interval.

THC (g/km): 16 participants reported results within the Ref \pm 1s interval. Participants 3, 41, 88 and 7 reported results within the Ref \pm 2s interval and participant 64 and 19 presented the greatest dispersions.

Road Autonomy (km/L): 14 participants reported results within the Ref \pm 1s interval. Participants 7, 54, 30 and 12 reported results within the Ref \pm 2s interval and participants 85 and 64 presented results out of Ref \pm 2s interval.

Combined Autonomy (km/L): 14 participants reported results within the Ref \pm 1s interval. Participants 54, 30 and 12 reported results within the Ref \pm 2s interval and participants 85, 64 and 7 presented results out of Ref \pm 2s interval.

6.3. Idling Speed CO

Figure 25 graphically present the means and robust standard deviation of the reported results by the participants for the Idling Speed CO (% vol) parameter.



Figure 25 – Scatter plot of the participants' measurement results for Idling Speed CO.

Through the graphics, it can be seen that:

Idling Speed CO (%vol): 12 participants reported results within the Ref \pm 1s interval. Participants 61, 93, 64, 12 and 57 reported results within the Ref \pm 2s interval and participants 30 and 84 presented results out of the Ref \pm 2s interval. Participant 61 presented the greatest dispersion.

6.4. NMOG

Figure 26 graphically present the means and robust standard deviation of the reported results by the participants for the NMOG.



Figure 26 – Scatter plot of the participants' measurement results for NMOG (g/km).

Through the graphics, it can be seen that:

NMOG (g/km): 12 participants reported results within the Ref \pm 1s interval. Participants 63, 53, 32 and 95 reported results within the Ref \pm 2s interval and participant 73 presented results out of Ref \pm 2s interval. Participant 34 presented the greatest dispersion.

7. Participants' Results

Measurement results reported by participants in this PT are presented in sections 7.1 to 7.3.

Each participant in this report is identified by the last numeric characters of its identification code in tables, graphs and texts.

7.1. Average and Standard Deviations Results

7.1.1. Urban Cycle Emissions

Tables 7 and 8 present the replicates average and standard deviations of each participant, for urban cycle emission data by participants for each analyzed parameter.

Note: All decimal places were considered for calculations, but the values in all tables were rounded to the same number of decimal places as requested in the results form.

Code	de CO (g/km)		CO2 (g/km)		T] (g/	THC (g/km)		NMHC (g/km)		Ox xm)
	Average	Standard deviation	Average	Standard deviation	Average	Standard deviation	Average	Standard deviation	Average	Standard deviation
3	2.397	0.017	146.7	1.1	0.327	0.004	0.300	0.004	0.341	0.007
7	2.970	0.075	167.5	0.5	0.418	0.003	0.378	0.001	0.374	0.013
12	2.455	0.081	150.3	1.2	0.340	0.002	0.314	0.001	0.423	0.005
19	2.611	0.040	150.4	1.2	0.350	0.010	0.319	0.008	0.371	0.016
24	2.832	0.230	157.3	1.0	0.367	0.004	0.337	0.003	0.412	0.021
30	2.664	0.109	150.3	0.9	0.354	0.006	0.324	0.005	0.384	0.008
35	2.588	0.057	153.6	0.4	0.346	0.009	0.318	0.008	0.388	0.006
41	2.553	0.067	151.1	0.5	0.373	0.002	0.344	0.002	0.467	0.022
54	2.352	0.039	150.3	0.4	0.331	0.004	0.302	0.005	0.396	0.005
57	2.320	0.084	151.4	0.1	0.366	0.005	0.334	0.005	0.342	0.006
60	2.637	0.111	154.4	0.2	0.345	0.007	0.311	0.004	0.390	0.003
61	2.463	0.047	150.4	0.7	0.344	0.008	0.313	0.005	0.411	0.015
64	2.692	0.080	163.0	1.9	0.341	0.014	0.309	0.012	0.399	0.020
67	2.916	0.067	153.9	0.4	0.355	0.004	0.322	0.004	0.347	0.009
80	2.623	0.036	155.8	2.5	0.337	0.009	0.305	0.008	0.335	0.014
84	2.853	0.050	154.3	0.4	0.342	0.015	0.313	0.013	0.374	0.003
85	2.473	0.048	162.1	0.4	0.314	0.009	0.288	0.009	0.453	0.015
88	2.541	0.108	156.2	0.9	0.381	0.007	0.349	0.007	0.396	0.006
90	3.001	0.107	156.4	1.0	0.370	0.007	0.337	0.006	0.400	0.003
93	2.367	0.037	153.3	0.4	0.331	0.010	0.304	0.008	0.402	0.005

Table 7 – Average and standard deviation of participants for CO, CO₂, THC, NMHC and NO_x (g/km) parameters – urban cycle.

Table 8 – Average and standard deviation of participants for CH₄ (g/km), Total Aldehydes (g/km), Unburned Ethanol (g/km) and Urban Autonomy (km/L) parameters – urban cycle.

Code	CH4 (g/km)		Total Aldehydes (g/km)		Unbu Ethano	urned l (g/km)	Urban Autonomy (km/L)	
coue	Average	Standard deviation	Average	Standard deviation	Average	Standard deviation	Average	Standard deviation
3	0.029	0.001	0.08889	0.00176	0.47195	0.01793	9.33	0.07
7	0.040	0.003	0.07411	0.00409	0.62378	0.01198	8.31	0.03
12	0.028	0.001	0.08833	0.00133	0.51138	0.00372	9.31	0.09
19	0.032	0.002	0.08243	0.01064	0.42598	0.02558	9.20	0.07
24	0.032	0.002	0.05212	0.00162	0.51317	0.01136	8.88	0.06
30	0.033	0.001	0.06203	0.00109	0.53384	0.00830	9.29	0.07

Code	C (g/I	H4 km)	Total A (g/)	Total Aldehydes Unburn (g/km) Ethanol (g		urned l (g/km)	ned Urban Autonom (g/km) (km/L)	
coue	Average	Standard deviation	Average	Standard deviation	Average	Standard deviation	Average	Standard deviation
35	0.030	0.001	0.09268	0.01080	0.42943	0.03751	9.11	0.03
41	0.031	0.000	0.07991	0.00115	0.50542	0.02635	9.25	0.03
54	0.029	0.001	0.05725	0.00724	0.33017	0.04202	9.33	0.02
57	0.032	0.002	0.09012	0.00207	0.41922	0.00887	9.22	0.01
60	0.034	0.004	0.07294	0.00793	0.48370	0.02417	9.02	0.01
61	0.034	0.003	0.03073	0.00554	0.36567	0.02763	9.21	0.04
64	0.034	0.004	0.08412	0.00867	0.38434	0.02288	8.40	0.09
67	0.033	0.001	0.09010	0.00952	0.69738	0.11183	9.02	0.03
80	0.032	0.003	0.08910	0.00282	0.68190	0.02669	8.94	0.14
84	0.031	0.002	0.07735	0.00124	0.43842	0.04446	9.02	0.03
85	0.027	0.001	0.12419	0.00576	0.52535	0.04145	8.62	0.02
88	0.033	0.002	0.06610	0.02018	0.42658	0.17134	8.96	0.06
90	0.036	0.002	0.09698	0.00310	0.36507	0.03448	8.91	0.06
93	0.031	0.002	0.07241	0.00772	0.43656	0.01677	9.12	0.03

7.1.2. Road Cycle Emissions

Tables 9 and 10 present the replicates average and standard deviations of each participant for CO (g/km), CO₂ (g/km), THC (g/km), Road Autonomy (km/L) and Combined Autonomy (km/L) parameters.

 Table 9– Average and standard deviation of participants for CO (g/km), CO₂ (g/km), THC (g/km) and Road Autonomy (km/L) parameters – road cycle.

Code	(g/	20 km)	CO ₂ (g/km)		THC (g/km)		Road Autonomy (km/L)	
couc	Average Standard deviation		Average	Standard deviation	Average	Standard deviation	Average	Standard deviation
3	1.122	0.030	88.6	0.4	0.121	0.000	15.58	0.09
7	1.270	0.024	95.6	0.7	0.140	0.001	14.70	0.11
12	1.058	0.014	87.7	0.7	0.128	0.002	16.10	0.12
19	1.113	0.007	89.1	0.8	0.131	0.006	15.68	0.12
24	1.217	0.081	92.5	1.3	0.133	0.002	15.25	0.23
30	1.139	0.009	88.9	0.9	0.125	0.001	15.87	0.16
35	1.117	0.029	92.0	0.7	0.134	0.002	15.35	0.11
41	1.118	0.019	91.7	0.1	0.139	0.001	15.39	0.03
54	1.060	0.015	89.1	0.2	0.125	0.000	15.87	0.04
57	1.010	0.008	90.0	0.4	0.135	0.002	15.66	0.07
60	1.167	0.033	90.9	0.2	0.125	0.002	15.46	0.02

Code	(g/	CO km)	CO ₂ (g/km)		THC (g/km)		Road Autonomy (km/L)	
coue	Average	Standard deviation	Average	Standard deviation	Average	Standard deviation	Average	Standard deviation
61	1.052	0.012	88.6	0.4	0.127	0.001	15.79	0.07
64	1.100	0.035	91.5	1.8	0.123	0.006	14.24	0.28
67	1.305	0.039	91.8	0.1	0.124	0.001	15.26	0.03
80	1.152	0.012	94.1	1.3	0.127	0.001	14.94	0.21
84	1.245	0.034	93.3	0.7	0.124	0.001	15.07	0.10
85	1.205	0.023	105.8	0.9	0.129	0.000	13.33	0.10
88	1.082	0.009	94.0	0.4	0.139	0.001	15.04	0.06
90	1.244	0.038	93.7	0.3	0.125	0.001	15.06	0.06
93	1.071	0.010	90.1	0.5	0.126	0.001	15.63	0.08
Source: Dime	ci/Dimqt/Lafi	q						

Code	Combined Autonomy (km/L)			
	Average	Standard deviation		
3	11.39	0.07		
7	10.33	0.04		
12	11.49	0.09		
19	11.30	0.07		
24	10.93	0.09		
30	11.42	0.09		
35	11.15	0.04		
41	11.28	0.03		
54	11.46	0.03		
57	11.32	0.01		
60	11.10	0.01		
61	11.33	0.05		
64	10.30	0.14		
67	11.05	0.02		
80	10.91	0.17		
84	11.01	0.04		
85	10.25	0.02		
88	10.95	0.05		
90	10.92	0.06		
93	11.22	0.02		

7.1.3. Idling Speed CO (%vol.)

Table 11 presents the replicates average and standard deviations of each participant for Idling Speed CO (%vol.) parameter.

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0.0173
0.0021
0.0053
0.1080
0.0200
).0067
).0058
0.0385
0.0023
0.0115
0.0063
).0119

Table 11- Average and standard deviation of participants for Idling Speed CO (%vol.) parameter.

Source: Dimci/Dimqt/Lafiq

7.1.4. NMOG (g/km)

Table 12 presents the replicates average and standard deviations of each participant for NMOG (g/km) parameter.

Table 12 – Average and standard deviation of participants for NMOG (g/km) parameter.

C(1)	NMOG (g/km)			
Coalgo	Average	Standard deviation		
6	0.350	0.003		
29	0.346	0.004		
32	0.356	0.005		
34	0.341	0.041		
44	0.321	0.002		

	NMOG (g/km)				
Código	Average	Standard deviation			
47	0.314	0.009			
48	0.339	0.017			
52 0.334		0.002			
53	0.297	0.002			
55	0.352	0.007			
58	0.332	0.013			
63	0.285	0.014			
68	0.323	0.011			
70	0.312	0.017			
73	73 0.261				
95	95 0.356 0.01				
99	0.319	0.003			
Source: Dimci/Dimqt/Lafiq					

7.2. *z*-Score

7.2.1. Urban Cycle Emissions – *z*-score

For the performance evaluation of the participants, *z*-score values were calculated, after the exclusion of the outlier results, using the robust average and robust standard deviation of the results for each urban cycle emission parameter. Tables 13 and 14 and figures 27 to 35 present these results.

	CO (g/km)	CO ₂ (g/km)	THC (g/km)	NMHC (g/km)	NO _x (g/km)
Code	z-score	z-score	z-score	z-score	z-score
3	-0.94	-1.69	-1.14	-1.04	-1.36
7	1.55	3.21	3.36	3.09	-0.42
12	-0.69	-0.84	-0.50	-0.30	0.99
19	-0.01	-0.83	0.00	-0.06	-0.49
24	0.95	0.80	0.84	0.89	0.69
30	0.22	-0.85	0.21	0.24	-0.13
35	-0.11	-0.07	-0.20	-0.09	0.00
41	-0.26	-0.66	1.14	1.30	2.28
54	-1.13	-0.84	-0.96	-0.92	0.23
57	-1.27	-0.58	0.79	0.77	-1.35
60	0.11	0.12	-0.23	-0.44	0.05
61	-0.65	-0.82	-0.28	-0.35	0.65
64	0.34	2.15	-0.45	-0.57	0.30
67	1.32	0.00	0.26	0.12	-1.20
80	0.04	0.46	-0.64	-0.76	-1.54
84	1.05	0.10	-0.40	-0.35	-0.40
85	-0.61	1.94	-1.80	-1.69	1.88
88	-0.31	0.55	1.52	1.53	0.21
90	1.69	0.59	1.00	0.90	0.33

Table 13 – z-score values for the CO, CO₂, THC, NMHC and NO_x parameters– urban cycle.

Cada	CO (g/km)	CO ₂ (g/km)	THC (g/km)	NMHC (g/km)	NO _x (g/km)
Code	z-score	z-score	z-score	z-score	z-score
93	-1.07	-0.13	-0.94	-0.85	0.41

* Satisfactory result * Questionable result

* Unsatisfactory result

Table 14 – z-score values for the CH4 (g/km), Total Aldehydes (g/km), Unburned Ethanol (g/km) and Urban Autonomy (km/L) – urban cycle.

Code	CH4(g/km)	Total Aldehydes (g/km)	Unburned Ethanol (g/km)	Urban Autonomy (km/L)
	z-score	z-score	z-score	z-score
3	-1.24	0.62	0.02	1.12
7	3.02	-0.29	1.64	-3.14
12	-1.37	0.58	0.44	1.05
19	0.18	0.22	-0.47	0.57
24	0.18	-1.64	0.46	-0.76
30	0.31	-1.03	0.68	0.97
35	-0.59	0.85	-0.43	0.19
41	-0.35	0.07	0.38	0.80
54	-1.11	-1.33	-1.49	1.14
57	0.18	0.69	-0.54	0.68
60	0.83	-0.36	0.15	-0.18
61	0.96	-2.96	-1.11	0.62
64	0.70	0.32	-0.91	-2.75
67	0.57	0.69	2.42	-0.19
80	-0.08	0.63	2.25	-0.51
84	-0.46	-0.09	-0.34	-0.19
85	-1.88	2.79	0.59	-1.84
88	0.31	-0.78	-0.46	-0.44
90	1.43	1.12	-1.12	-0.63
93	-0.46	-0.40	-0.36	0.23

Source: Dimci/Dimqt/Lafiq

* Satisfactory result

* Questionable result * Unsatisfactory result



Figure 27 – *z*-score graph for CO – urban cycle.

Source: Dimci/Dimqt/Lafiq



Figure 28 - z-score graph for CO₂ – urban cycle.



Figure 29 – *z*-score graph for THC – urban cycle.



Figure 30 – *z*-score graph for NMHC – urban cycle.



Figure 31 - z-score graph for NO_x – urban cycle.



Figure 32 - z-score graph for CH₄ – urban cycle.



Figure 33 – *z*-score graph for Total Aldehydes– urban cycle.



Figure 34 – *z*-score graph for Unburned Ethanol – urban cycle.



Figure 35 – z-score graph for Urban Autonomy– urban cycle.

Source: Dimci/Dimqt/Lafiq

Through *z*-score graphic analysis, it can be seen that:

CO (g/km): 20 participants presented satisfactory results;

CO₂ (g/km): 18 participants presented satisfactory results, participant 64 presented questionable result and participant 7 presented unsatisfactory result;

THC (g/km): 19 participants presented satisfactory results and participant 7 presented unsatisfactory result;

NMHC (g/km): 19 participants presented satisfactory results and participant 7 presented unsatisfactory result;

 NO_x (g/km): 19 participants presented satisfactory results and participant 41 presented questionable result;

CH4 (g/km): 19 participants presented satisfactory results and participant 7 presented unsatisfactory result;

Total Aldehydes (g/km): 18 participants presented satisfactory results, participants 61 and 85 presented questionable results;

Unburned Ethanol (g/km): 18 participants presented satisfactory results, participants 80 and 67 presented questionable results;

Urban Autonomy (km/L): 18 participants presented satisfactory results, participant 64 presented questionable result and participant 7 presented unsatisfactory result.

7.2.2. Road Cycle Emissions – *z*-score

For the performance evaluation of the participants, *z*-score values were calculated, after the exclusion of the outlier results, using the robust average and robust standard deviation of the results for each road cycle emission parameter. Table 15 and figures 36 to 40 present these results.

		<u> </u>			
Code	CO (g/km)	CO ₂ (g/km)	THC (g/km)	Road Autonomy (km/L)	Combined Autonomy (km/L)
	z-score	z-score	z-score	z-score	z-score
3	-0.21	-1.05	-1.28	0.48	0.90
7	1.45	1.57	1.81	-1.31	-2.52
12	-0.93	-1.37	-0.18	1.54	1.25
19	-0.32	-0.87	0.43	0.68	0.61
24	0.85	0.39	0.70	-0.19	-0.57
30	-0.02	-0.94	-0.62	1.07	1.03
35	-0.27	0.20	0.81	0.02	0.12
41	-0.26	0.11	1.62	0.10	0.54
54	-0.90	-0.85	-0.65	1.07	1.13
57	-1.46	-0.53	0.98	0.63	0.68
60	0.29	-0.19	-0.62	0.23	-0.02
61	-1.00	-1.05	-0.23	0.89	0.73
64	-0.46	0.03	-0.90	-2.24	-2.61
67	1.84	0.15	-0.79	-0.18	-0.19
80	0.13	1.00	-0.29	-0.82	-0.64
84	1.17	0.69	-0.84	-0.55	-0.33
85	0.72	5.34	0.04	-4.10	-2.78
88	-0.66	0.95	1.75	-0.61	-0.50
90	1.16	0.84	-0.65	-0.58	-0.62
93	-0.78	-0.48	-0.40	0.58	0.37

Table 15 – *z*-score values for the CO, CO₂, THC, Road Autonomy and Combined Autonomy– road cycle parameters.

Source: Dimci/Dimqt/Lafiq

* Satisfactory result

* Questionable result

* Unsatisfactory result



Figure 36 – *z*-score graph for CO – road cycle.



Figure 37 - z-score graph for CO₂ – road cycle.

Source: Dimci/Dimqt/Lafiq

Source: Dimci/Dimqt/Lafiq



Figure 38 – *z*-score graph for THC – road cycle.



Figure 39 – *z*-score graph for Road Autonomy– road cycle.



Figure 40 – *z*-score graph for Combined Autonomy– road cycle.

Through *z*-score graphic analysis, it can be seen that:

CO (g/km): 20 participants presented satisfactory results;

CO₂ (g/km): 19 participants presented satisfactory results and participant 85 presented unsatisfactory result;

THC (g/km): 20 participants presented satisfactory results;

Road Autonomy (km/L): 18 participants presented satisfactory results, participant 64 presented questionable result and participant 85 presented unsatisfactory result.

Combined Autonomy (km/L): 17 participants presented satisfactory results, participants 85, 64 and 7 presented questionable results.

7.2.3. Idling Speed CO (% vol.) - z-score

For the performance evaluation of the participants, *z*-score values were calculated, after the exclusion of the outlier results, using the robust average and robust standard deviation of the results for Idling Speed CO (% vol.) parameter. Table 16 and figure 41 present these results.

Code	Idling Speed CO (g/km)	
Coue	z-score	
3	0.45	
12	1.04	
19	0.36	
24	-0.77	
30	-4.55	
35	0.40	
41	-0.17	
54	-0.35	
57	1.15	
60	0.22	
61	-1.39	
64	-1.20	
67	0.18	
80	0.49	
84	3.08	
85	0.30	
88	0.74	
90	-0.22	
93	-1.22	

Table 16 – z-score values for the Idling Speed CO (% vol.) parameter.

Source: Dimci/Dimqt/Lafiq * Satisfactory result

* Questionable result

* Unsatisfactory result



Figure 41 – *z*-score graph for Idling Speed CO.

Source. Diner Dinq/ Lang

Through *z*-score graphic analysis, it can be seen that:

Idling Speed CO (% vol.): 17 participants presented satisfactory results, participants 30 and 84 presented unsatisfactory results.

7.2.4. NMOG (g/km) - z-score

For the performance evaluation of the participants, *z*-score values were calculated, after the exclusion of the outlier results, using the robust average and robust standard deviation of the results for NMOG parameter. Table 17 and figure 42 present these results.

Code	NMOG (g/km)
	z-score
6	0.89
29	0.73
32	1.14
34	0.53
44	-0.26
47	-0.57
48	0.46
52	0.23
53	-1.24

Table 17 - z-score values for the NMOG (g/km) parameter.

Code	NMOG (g/km)
	z-score
55	0.98
58	0.18
63	-1.71
68	-0.21
70	-0.64
73	-2.69
95	1.14
99	-0.34

* Satisfactory result

* Questionable result * Unsatisfactory result





Through *z*-score graphic analysis, it can be seen that:

NMOG (g/km):16 participants presented satisfactory results, participant 73 presented questionable result.

7.3. Results Preview and Outliers Detection: Boxplot

Figure 43 graphically presents the boxplot of the reported results by participants for the NMOG parameter.



Figure 43 – Boxplot graphic of the participants results for NMOG.

Through the graphic, it can be seen that:

NMOG: 16 participants presented satisfactory results, participant 73 presented questionable result, that is, further away from the median. Participant 34 presented the greatest data dispersion.

8. Analysis Testimony

As established in the proficiency testing protocol, a Cetesb representative witnessed one of the PT three measurements at each participant laboratory. Cetesb sent a conclusion regarding its testimony result to the PT Coordination by email, indicating there was no occurrence of non-compliance, not being necessary to send the witnessed results reports to the PT Coordination.

It should be noted that, in case Cetesb registered the occurrence of non-compliance to the PT Coordination, the participant's results would be invalidated and, thus, its data would not figure in the PT report.

9. Confidenciality

Each participant was identified by an individual code which is only known by the participant and the PT coordination. As stated on the registration form, the identification of accredited laboratories and

laboratories in stage of accreditation will be forwarded for information of the General Accreditation Coordination (Cgcre). The participant received, by email, his own identification code corresponding to the participation in this PT. This code was used to identify the participant in the results registration form. The results may be used in studies and publications by Inmetro respecting the confidentiality of each participant.

As established in section 4.10.4 of ABNT ISO/IEC 17043:2011, in exceptional circumstances, a regulatory authority may require the results and the identification of the participants to the PT provider. If this occurs, the provider will notify the PT participants about this action.

10. Conclusions

This round once again sought to advance to improve the measurements of parameters already established and of new ones, as was the case of NMOG measurement in which the formula was defined within the course of this Proficiency Test. This research and methods development are essential to move forward continuously. In addition, we can conclude that the results have been quite satisfactory and their achievement has been of great importance for industry and society throughout these eleven rounds held within the Inmetro-AEA partnership.

This PT round involved a large number of variables and the testimony of a regulation body (Cetesb). This large numbers of variables in the vehicle emissions PT certainly have influence in the reported results. Therefore it is recommended that participants that showed questionable and unsatisfactory performance to critically evaluate their measurement methods.

Among 540 results within nine parameters in the urban cycle evaluated by the *z*-score, 93.33% indicated satisfactory performance, 3.88% indicated questionable performance in five different parameters and 2.77% indicated unsatisfactory performance in five parameters. Among 300 results within five parameters of the road cycle evaluated by the *z*-score, 94% indicated satisfactory performance, 4% indicated questionable performance in two different parameters and 2% indicated unsatisfactory performance in two parameters. Among 57 results for Idling Speed CO (g / km) evaluated by the *z*-score, 89.5% indicated satisfactory performance and 10.5% indicated unsatisfactory performance.

It should be emphasized the importance of different laboratory participation in a proficiency test scheme, since it constitutes an useful tool to monitor the procedures in routine analysis and to evaluate

the laboratory measurement results, enabling the improvement of the results quality and ensuring greater reliability to the measurements.

It is up to PT participant to carry out a critical analysis of the results, where the entire process and laboratory experience must be considered. Therefore, the continuous participation in a proficiency test can assure information to the participants about the measurement capability and it is of great importance for monitoring the validity of the results.

11. Participants

Twenty three participants were registered in the 11th round of the Proficiency Testing in Vehicles Emissions, but three participants did not send their results report because of equipment problems and informed it to the PT coordination. Thus, twenty participants remained.

The list of laboratories that sent results to this PT coordination is presented in Table 17. It is important to note that the numbering of laboratories in the table only indicates the number of PT participants; under no circumstances, it is associated to laboratory identification in presenting their results.

Institution		
1.	AVL South America Ltda. AVL South America	
2.	CAOA Montadora de Veículos Centro de Pesquisa e Eficiência Energética	
3.	Cetesb Companhia Ambiental do Estado de São Paulo Setor de Laboratório de Emissão Veicular - São Paulo	
4.	CPT Brasil Automotiva Ltda. Laboratório de Emissões Veiculares	
5.	FCA Fiat Chrysler Automóveis Brasil Ltda. Laboratório de Emissões e Consumo	
6.	Ford Motor Company Brasil Ltda. Laboratório de Emissões do Campo de Provas de Tatuí	
7.	General Motors do Brasil Ltda. Laboratório de Emissões do Campo de Provas de Cruz Alta	
8.	General Motors do Brasil Ltda. Laboratório de Emissões Veiculares GM SJC – Qualidade GPS	
9.	Honda Automóveis do Brasil Ltda. Laboratório de Emissões Honda Automóveis	
10.	Hyundai Motor Brasil Montadora de Automóveis Ltda. Laboratório de Emissões do R&D Center da Hyundai Motor Brasil	
11.	Instituto de Tecnologia para o Desenvolvimento LEME – Laboratório de Emissões Veiculares	
12.	Jaguar e Land Rover Brasil Industria e Comercio de Veículos Ltda. Jaguar Land Rover	
13.	Magneti Marelli Sistemas Automotivos Ind. e Comércio Ltda. Magneti Marelli	

Table 17 – Participants

Institution		
14.	Petróleo Brasileiro S.A. Laboratório de Ensaios Veiculares – CENPES	
15.	Peugeot Citroen do Brasil Automóveis LTDA	
16.	Renault do Brasil S/A. LEV - Laboratório de Emissões Veiculares	
17.	Robert Bosch Ltda. Laboratório de Emissões Veiculares - LEV	
18.	Toyota do Brasil Ltda. Laboratório de Emissões Toyota do Brasil - Indaiatuba	
19.	Umicore do Brasil Ltda. Umicore	
20.	Volkswagen do Brasil Indústria de Veículos Automotores Ltda. Laboratório de Emissões Veiculares da Volkswagen do Brasil	

Total participants: 20 participants.

12. References

- ABNT NBR ISO/IEC 17025:2017: Requisitos gerais para a competência de laboratórios de ensaio e calibração.
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- ISO 13528:2015 (E), "Statistical methods for use in proficiency testing by interlaboratory comparisons".
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- ABNT NBR 6601:2012: "Veículos rodoviários automotores leves Determinação de hidrocarbonetos, monóxido de carbono, óxidos de nitrogênio, dióxido de carbono e material particulado no gás de escapamento".
- ABNT NBR 12026:2016: Veículos rodoviários automotores leves- Determinação da emissão de aldeídos e cetonas contidos no gás de escapamento, por cromatografia líquida Método DNPH.
- ABNT NBR 15598:2016: Veículos rodoviários automotores leves- Determinação de etanol não queimado contido no gás de escapamento por cromatografia gasosa- Método de ensaio.
- ABNT NBR 10972:2010: Veículos rodoviários automotores leves Medição da concentração de monóxido de carbono no gás de escapamento em regime de marcha lenta- Ensaio de Laboratório.
- ABNT NBR 7024:2017: "Veículos rodoviários automotores leves Medição do consumo de combustível - Método de ensaio".
- ABNT NBR 8689:2012: "Veículos rodoviários automotores leves Combustíveis para ensaio Requisitos".



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