Final Report of the Proficiency Testing in Vehicles Emissions 12th Round ECE Cycle Vehicles – Gasoline (Mercosur)





PROGRAMA DE ENSAIOS DE PROFICIÊNCIA DO INMETRO

PROFICIENCY TESTING IN VEHICLES EMISSIONS 12th ROUND – ECE CYCLE VEHICLES – GASOLINE (MERCOSUR)

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1. Introduction

The automotive industry is of great relevance to the Brazilian economy and, in the search to keep expanding the markets, undergoes a constant adaptation to rules and regulations to obtain the acceptance of its products. In this chain formed by vehicle manufacturers and systemists, this search is constant.

Mercosur is an important market for the Brazilian automotive industry and it is very important expanding its participation in this segment. To pursue this objective, a specific Proficiency Test (PT) was carried out through European Regulations R83-06 and R101. This fact shows the search for new challenges even in times of global health crisis.

Regardless of the crisis, this industry is looking for new and important changes. Thus, we seek to understand this transformation scenario that has been designed for the sector. Finally, in the face of a challenging future, it is suggested that the automotive industry can be inspired to build a strategy to modernize its productive and technological insertion within Latin America.

The performance of the PT for vehicles emissions aims to assess the performance of laboratories in determining the amount of compounds present in such emissions, providing subsidies for the identification and solution of analytical problems. Also contributing to the harmonization of measurement results to evaluate the performance, in addition to being a tool for generating data that can support the preparation of new insertions in the most diverse markets.

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 provided by Toyota do Brasil with the following characteristics: Corolla Altis model, white color, Chassis 9BRBUWHE3F0001001, 1.8 Gasoline engine and 6-speed manual transmission.

Each participating laboratory used as fuel: Gasoline Euro V - code RF-02-08, as defined in the protocol.

2.2. Metodology

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

ECE Cycle	European Regulation R83-06: THC, NMHC, CH ₄ , CO, CO ₂ , NO _x and THC+NO _x European Regulation R101: Consumption
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The laboratories reproduced the deceleration curve in dynamometer informed by the emissions laboratory of Toyota do Brasil company. 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 Toyota do Brasil Company to be considered were those referred to the tests performed in the beginning of the cycle (Y_1).

3. Test Item Integrity

Toyota do Brasil laboratory performed stability tests in the beginning, in the middle and at the end of the cycle – first analysis (Y_1), and second analysis (Y_2) and third analysis (Y_3). It was verified if there were significant statistical differences between measurements of the 8 components: THC, NMHC, CH₄, CO, CO₂, NO_x, THC+NO_x and Consumption.

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:

Table 1 - Statistics and p-values						
Before	x_1	x_2		x_N		
After	y_1	\mathcal{Y}_2		\mathcal{Y}_N		
d _i	d ₁	d_2	•••	d_N		
$ d_i $	$ d_1 $	$ d_2 $		$ d_N $		
post	Ν	2		1		
Source: Dimci/Dimqt/Lafig						

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 $V_s \ge c_2$, H₀ is rejected, in which c_1 and c_2 are such that $P(V_s \le c_1 | H_0) = \frac{\alpha}{2}$ and $P(V_s \ge c_2 | H_0) = \frac{\alpha}{2}$.

Thus, the hypothesis of stability between such measurements was tested using the Wilcoxon test, also known as the Mann-Whitney test. Table 2 below summarizes the result of applying the two samples at the 5% significance level.

Component	p-value
CO (g/km)	0.733
CO ₂ (g/km)	0.051
CH ₄ (g/km)	0.066
THC (g/km)	0.061

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

p-value
0.058
0.061
0.039
0.079

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. However, the vehicle presented problems for the parameter THC + NO_x (g / km) changing its integrity during the realization of this PT.

Due to data confidentiality, once Toyota do 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.

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:

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

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:

$$x^{*} = \sum x_{i}^{*} / p$$
(8)
$$s^{*} = 1,134 \sqrt{\sum (x_{i}^{*} - x^{*})^{2} / (p - 1)}$$
(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.

	Assigned	Standard	Outliers	Recalculated	Recalculated
Parameter	Value	deviation	(Participants)	Assigned value	standard deviation
CO (g/km)	0.407	0.042	-	-	-
CO ₂ (g/km)	155.1	4.4	-	-	-
CH ₄ (g/km)	0.003	0.001	-	-	-
THC (g/km)	0.032	0.003	-	-	-
NO _x (g/km)	0.015	0.002	-	-	-
NMHC (g/km)	0.028	0.003	-	-	-
THC + NO _x (g/km)	0.047	0.005	-	-	-
Consumo (L/100 km)	6.72	0.30	19	6.67	0.25

T 11 0 1 7 7 1 1 . C .1

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. Emissions

Figures 2 to 9 graphically present the means and robust standard deviation of the reported emissions results by the participants for each analyzed parameter.





Figure 3 - Scatter plot of the participants' measurement results for CO₂.





Figure 4 - Scatter plot of the participants' measurement results for CH₄.

Figure 5 - Scatter plot of the participants' measurement results for THC.



Source: Dimci/Dimqt/Lafiq



Figura 6 - Scatter plot of the participants' measurement results for NO_x.

Figure 7 - Scatter plot of the participants' measurement results for NMHC.



Source: Dimci/Dimqt/Lafiq



Figure 8 - Scatter plot of the participants' measurement results for THC+NO_x.

Figure 9 - Scatter plot of the participants' measurement results for Consumption.



Through the graphics, it can be seen that:

CO (g/km): 11 participants reported results within the Ref \pm 1s interval. Participants 21 and 24 reported results within the Ref \pm 2s interval and participants 02, 06, 16, 19 and 25 presented the greatest dispersion.

CO₂ (g/km): 10 participants reported results within the Ref \pm 1s interval. Participant 25 reported results within the Ref \pm 2s interval and participants 02 and 16 presented results out of Ref \pm 2s interval.

CH4 (g/km): 10 participants reported results within the Ref \pm 1s interval. Participant 16 reported results within the Ref \pm 2s interval, participants 02 and 21 presented results out of Ref \pm 2s interval and participants 02, 07, 19, 21 e 25 presented the greatest dispersions.

THC (g/km): 7 participants reported results within the Ref \pm 1s interval. Participants 06, 07, 17 and 24 reported results within the Ref \pm 2s interval, participants 02 and 25 presented results out of Ref \pm 2s interval and participant 25 presented the greatest dispersion.

NO_x (g/km): 9 participants reported results within the Ref \pm 1s interval. Participants 10, 17 and 19 reported results within the Ref \pm 2s interval, participant 12 presented results out of Ref \pm 2s interval and participant 25 presented the greatest dispersion.

NMHC (g/km): 8 participants reported results within the Ref \pm 1s interval. Participants 02, 06, 07 and 16 reported results within the Ref \pm 2s interval, participant 25 presented results out of Ref \pm 2s interval and the greatest dispersion.

THC + NO_x (g/km): 9 participants reported results within the Ref \pm 1s interval. Participants 02, 06, 17 and 25 reported results within the Ref \pm 2s interval, and participant 25 presented the greatest dispersion.

Consumption (L/100 km): 9 participants reported results within the Ref \pm 1s interval. Participants 02, 10 and 16 reported results within the Ref \pm 2s interval and participant 19 presented results out of Ref \pm 2s interval.

7. Participants' Results

Measurement results reported by participants in this PT are presented in sections 7.1 and 7.2.

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. Emissions

Tables 4 and 5 present the replicates average and standard deviations of each participant for each emissions analyzed parameter.

Code	C (g/	CO km)	C (g/	O2 km)	C (g/	H4 km)	T] (g/	HC km)
Coue	Average	Standard deviation	Average	Standard deviation	Average	Standard deviation	Average	Standard deviation
2	0.439	0.057	162.7	0.7	0.004	0.001	0.037	0.003
6	0.383	0.069	157.3	0.8	0.003	0.000	0.028	0.002
7	0.449	0.034	152.6	0.1	0.003	0.000	0.034	0.002
10	0.416	0.015	151.4	1.3	0.003	0.000	0.032	0.001
12	0.386	0.023	152.9	0.7	0.003	0.000	0.030	0.002
15	0.383	0.028	153.1	0.7	0.003	0.000	0.030	0.001
16	0.394	0.050	164.6	1.9	0.004	0.000	0.031	0.000
17	0.374	0.014	150.7	1.2	0.003	0.000	0.028	0.001
19	0.436	0.062	157.3	0.3	0.003	0.000	0.032	0.002
21	0.475	0.034	151.2	0.2	0.005	0.005	0.032	0.001
24	0.339	0.015	153.9	0.8	0.003	0.007	0.028	0.001
25	0.442	0.054	159.2	0.4	0.004	0.008	0.042	0.005
38	0.377	0.044	152.9	0.2	0.003	0.014	0.032	0.002

Table 4 – Average and standard deviation of participants for CO, CO₂, CH₄ and THC (g/km) parameters.

Table 5 – Average and standard deviation of participants for NO_x (g/km), NMHC (g/km), THC + NO_x (g/km) and Consumption (L/100 km).parameters.

NOx (g/km)		NMHC (g/km)		THC + NOx (g/km)		Consumption (L/100km)		
Couc	Average	Standard deviation	Average	Standard deviation	Average	Standard deviation	Average	Standard deviation
2	0.016	0.003	0.033	0.003	0.054	0.002	7.07	0.03
6	0.015	0.002	0.024	0.002	0.042	0.004	6.76	0.03
7	0.015	0.000	0.031	0.002	0.049	0.002	6.60	0.03
10	0.018	0.001	0.029	0.001	0.050	0.001	6.38	0.06
12	0.021	0.002	0.027	0.002	0.051	0.004	6.58	0.03
15	0.016	0.002	0.028	0.001	0.046	0.002	6.61	0.03
16	0.015	0.002	0.025	0.001	0.046	0.003	7.13	0.09
17	0.012	0.001	0.026	0.001	0.040	0.001	6.44	0.06
19	0.013	0.001	0.029	0.003	0.045	0.002	14.68	0.02
21	0.015	0.003	0.028	0.001	0.047	0.004	6.52	0.01
24	0.016	0.001	0.025	0.001	0.044	0.002	6.64	0.03

Code	N (g/I	Ox km)	NMHC (g/km)		THC + NOx (g/km)		Consumption (L/100km)	
coue	Average	Standard deviation	Average	Standard deviation	Average	Standard deviation	Average	Standard deviation
25	0.014	0.005	0.039	0.005	0.056	0.010	6.90	0.02
38	0.015	0.002	0.029	0.002	0.047	0.002	6.57	0.01

7.2. *z*-Score

7.2.1. 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 emissions parameter. Tables 6 and 7 and figures 10 to 17 present these results.

Table 6 – z-score values for the CO, CO₂, CH₄ and THC parameters with the unit of measurement (g / km) for all parameters.

C. J.	CO (g/km)	CO ₂ (g/km)	CH ₄ (g/km)	THC (g/km)
Code	z-score	z-score	z-score	z-score
2	0.74	2.31	2.27	2.44
6	-0.57	0.81	-0.56	-1.31
7	0.98	-0.50	0.36	1.20
10	0.21	-0.81	-0.56	0.48
12	-0.51	-0.40	-0.56	-0.40
15	-0.57	-0.35	-0.56	-0.40
16	-0.31	2.86	1.56	-0.02
17	-0.78	-1.02	-0.56	-1.05
19	0.69	0.83	0.26	0.45
21	1.62	-0.88	2.98	0.24
24	-1.63	-0.12	-0.56	-1.05
25	0.83	1.34	0.86	4.25
38	-0.71	-0.41	-0.56	0.37

Source: Dimci/Dimqt/Lafiq

* Satisfactory result

* Questionable result

* Unsatisfactory result

Table 7 – z-score values for the NO _x (g/km), NMHC (g/km), THC + NO _x (g/km) and Cor	nsumption
(L/100km) parameters.	

Code	NOx (g/km)	NMHC (g/km)	THC + NO _x (g/km)	Consumption (L/100 km)
Couc	z-score	z-score	z-score	z-score
2	0.85	2.03	1.34	1.57
6	-0.33	-1.26	-1.09	0.33
7	0.14	1.06	0.44	-0.31

Code	NO _x (g/km)	NMHC (g/km)	THC + NO _x (g/km)	Consumption (L/100 km)
	z-score	z-score	z-score	z-score
10	1.92	0.60	0.58	-1.18
12	4.38	-0.39	0.70	-0.37
15	0.85	0.10	-0.23	-0.24
16	0.14	-1.14	-0.37	1.82
17	-1.98	-0.64	-1.51	-0.95
19	-1.29	0.47	-0.41	31.95
21	-0.09	-0.02	-0.16	-0.63
24	0.61	-0.89	-0.66	-0.12
25	-0.56	4.08	1.91	0.89
38	-0.33	0.60	-0.16	-0.43

* Satisfactory result * Questionable result

* Unsatisfactory result



Figure 10 - z-score graph for CO measurement.



Figure 11 - z-score graph for CO₂ measurement.



Figure 12 - z-score graph for CH₄ measurement.

Source: Dimci/Dimqt/Lafiq



Figure 13 – z-score graph for THC measurement.



Figure 14 - z-score graph for NO_x measurement.



Figure 15 – z-score graph for NMHC measurement.



Figure 16 - z-score graph for THC + NO_x measurement.



Figure 17 – z-score graph for Consumption measurement.

Source: Dimci/Dimqt/Lafiq

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

CO (g/km): 13 participants presented satisfactory results;

CO₂ (g/km): 11 participants presented satisfactory results, participants 02 and 16 presented questionable results;

CH₄ (g/km): 11 participants presented satisfactory results and participants 02 and 21 presented questionable results;

THC (g/km): 11 participants presented satisfactory results and participants 02 and 25 presented questionable results;

NO_x (**g/km**): 12 participants presented satisfactory results and participant 12 presented unsatisfactory result;

NMHC (g/km): 12 participants presented satisfactory results and participant 25 presented unsatisfactory result;

THC + NO_x (g/km): 13 participants presented satisfactory results;

Consumo (L/100 km): 12 participants presented satisfactory results and participant 19 presented unsatisfactory results;

8. 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.

9. Conclusions

This round once again sought to advance. The Brazilian automotive industry understands that expanding its participation in Mercosur is very important. For this, a specific proficiency test was carried out through European Regulations R83-06 and R101. This evaluation shows us the strength and commitment of this industry that fits and adapts to new rules and new situations, with the aim of always being competitive in new markets. 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 twelve rounds carried out within the Inmetro-AEA partnership.

Among the 312 results, in eight parameters, evaluated by the *z*-score, 90.38% indicated satisfactory performance, 5.76% indicated questionable performance in three different parameters and 3.86% indicated unsatisfactory performance in four parameters.

It should always be emphasized the importance of the participation of different laboratories in a proficiency test, since it is a useful tool to monitor the analysis procedures used in the routine and to evaluate the results of the laboratory measurements, enabling the improvement of the quality of the results and ensuring greater reliability to measurements.

It is up to the PT participant to perform a critical analysis of the results, and the entire process and laboratory experience must be considered. Therefore, participation in proficiency tests on an ongoing basis can guarantee information to participants about their measurement capability and is of great importance for monitoring the validity of their results.

10. Participants

Fourteen participants were registered in the 11th round of the Proficiency Testing in Vehicles Emissions, but one participant did not send its results report and informed it to the PT coordination. Thus, thirteen participants remained.

The list of laboratories that sent results to this PT coordination is presented in table 8. 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.	BRAZIL TRADING LTDA. Gandini Centro Tecnológico	
2.	CAOA Montadora de Veículos Ltda. Centro de Pesquisa e Eficiência Energética	
3.	FCA Fiat Chrysler Automóveis Brasil Ltda. LEC - Laboratório de Emissões e Consumo	
4.	Ford Motor Company Brasil Ltda. Laboratório de Emissões do Campo de Provas de Tatuí	
5.	General Motors do Brasil Laboratório de Emissões do Campo de Provas de Cruz Alta	
6.	General Motors do Brasil Ltda. Laboratório de Emissões Veiculares – Global Propulsion Systems	
7.	Honda Automóveis do Brasil Ltda. Laboratório de Emissões Honda Automóveis	
8.	Hyundai Motor Brasil Montadora de Automóveis Ltda. Laboratório de Emissões do R&D Center da Hyundai Motor Brasil	
9.	Instituto de Tecnologia para o Desenvolvimento LEME – Laboratório de Emissões Veiculares	
10.	Peugeot Citroen do Brasil Automóveis Ltda. Laboratório de Emissões	
11.	Renault do Brasil S/A LEV - Laboratório de Emissões Veiculares	
12.	Toyota do Brasil Ltda.	
13.	Volkswagen do Brasil Indústria de Veículos Automotores Ltda. Laboratório de Emissões Veiculares da Volkswagen do Brasil	

Total participants: 13 participants.

11. References

- ABNT NBR ISO/IEC 17025:2017: Requisitos gerais para a competência de laboratórios de ensaio e calibração.
- ABNT NBR ISO/IEC 17043:2011: Avaliação de Conformidade Requisitos Gerais para ensaios de proficiência.

- ISO 13528:2015 (E), "Statistical methods for use in proficiency testing by interlaboratory comparisons";
- Vocabulário Internacional de Metrologia: conceitos fundamentais e gerais e termos associados (VIM 2012). 1ª Edição Luso–Brasileira;
- Regulation No 83 of the Economic Commission for Europe of the United Nations (UN/ECE) Uniform provisions concerning the approval of vehicles with regard to the emission of pollutants according to engine fuel requirements;;
- UN Regulation No. 101 Carbon Dioxide Emissions and Fuel/Energy Consumption.



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