The Analysis Report on the Results of Laboratory Comparison of Combustion Performance of Large Plastic plates

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Abstract: Interlaboratory comparison is one of the important technical methods for internal quality control of laboratories. By participating in effective interlaboratory comparison activities and obtaining satisfactory results, the laboratory and customers can increase the trust of the laboratory. In the third quarter of 2018, the polymer material technology center organized and implemented a laboratory comparison of the flammability of plastic slabs. The comparison was made from the basic overview, sample preparation, comparison test, and analysis and test data. All the 6 laboratories participating in the comparison have obtained satisfactory comparison results, and only the laboratory data of code 005 is suspicious. The results of this interlaboratory comparison are reported below.

1. Introduction

In a car accident, a fire caused by a collision or spontaneous combustion poses a great threat to the safety of the occupants. In addition to the car's fuel tank, car interior materials are the second major factor affecting the severity of the fire. Many countries in the world have included the combustion characteristics of automotive interior materials in regulatory projects. At the same time, in order to improve the quality of their products, major automobile manufacturers have formulated their own corporate standards based on regulations.

The purpose of this inter-laboratory comparison plan is to verify the testing capabilities of the plastics combustion performance of participating laboratories, and to provide the laboratories with an objective basis for evaluating and demonstrating the reliability of their data.

Through this inter-laboratory comparison plan, it can objectively and accurately reflect the testing level of plastic combustion performance of participating laboratories, and can help the laboratory find problems in daily testing and improve the testing level of the laboratory.

2. Comparison Test Method

The method used in this comparison test is blind sample detection, that is, the same batch of samples is used for identification, and sample homogeneity screening is performed in the early stage. Random samples are taken from this and distributed to each laboratory for testing. The same testing method is required at a specified time.[1,2] Inspection. In order to ensure the consistency of the comparison test, a guideline for the combustion test of plastic slabs was formulated, which stipulated that each laboratory should strictly test in accordance with the requirements, and perform the test and report the results within the specified time.

3. Test Items and Requirements

The interlaboratory comparison requires the laboratory to determine the combustion characteristics of the standard samples in accordance with the test conditions specified in DBL 5307-2018 "Flame Retardant Performance-Requirements and Test Specifications for Automotive Interior Materials". Each laboratory performs tests and reports the results within the prescribed time in accordance with the standard requirements and results report requirements.

4. Description of the Sample Situation

This plan provides 1 set for each laboratory (5 samples in each group).

The sample is a PP plastic spline (sample size 356mm x 100mm x 3.2mm), packed in a plastic bag, and the sample is prepared by high-temperature injection molding. The sample is tested for uniformity before sending, and the results show that the sample is uniform good. See Appendix B for sample preparation reports and homogeneity test results.

5. Confidentiality Requirements

For the sake of confidentiality, this inter laboratory comparison assigns a code to each participating laboratory. In this report, where the results of testing and assessment of ability to participate in the laboratory are described, they are represented by codes.

6. Principles of Statistical Analysis Design and Capability Evaluation

The statistical analysis of the test results of this interlaboratory comparison is processed using Robust technology, that is, the median value of robust statistics is used as the specified value, and the standardized interquartile range (IQR) is the measure of variability (target standard deviation). To calculate the Z-scores for different test results. For the test results of this interlaboratory comparison, calculate the Z ratio score (Z value) according to the following formula:

$$Z = \frac{x - X}{\sigma} \tag{1}$$

6.1 The Formula: X- laboratory Test Results

X—specified value;

σ—Variability measure (target standard deviation).

The statistics involved in the calculation of the results of the interlaboratory comparison are the number of results, the median, the standardized interquartile range (IQR), the robust coefficient of variation (CV), the minimum, maximum, and range. Wait. For the meaning of each statistic and related calculation methods, refer to CNAS GL02: 2014 "Guidelines for Statistical Processing and Capability Evaluation of Proficiency Verification Results".

This time, the results of the laboratory were evaluated with Z scores, namely:

｜Z｜≤2 is the satisfactory result;

2<｜Z｜ <3 is the problematic result;

｜Z｜≥3 is the unsatisfactory result (outlier).

In order to clearly show the results of the laboratory-to-laboratory comparisons, the Z values are arranged in order of size as a histogram (Figure 1), and each bar is marked with the code of the laboratory. From the histogram, each laboratory can compare its results with other participating laboratories to understand the level of its results in this interlaboratory comparison.

6.2 Statistical Processing Results and Ability Evaluation

6.2.1 Summary of statistical parameters of average burning rate

A total of 7 laboratories submitted the test results of this project. The statistics of the results are shown in Table 1. The combustion rate results and evaluations of each laboratory are shown in Appendix A. The classification and statistics of the average combustion rate Z distribution are shown in Figure 1.

Table 1: Statistics of average combustion rate results

Number of results	Median (mm/min)	Standardization IQR(mm/min)	Stabilize CV(%)	Minimum value (mm/min)	Maximum value (mm/min)	Poor range (mm/min)
7	22.37	3.86	0.17	13.00	26.54	13.54

6.2.2 The Z ratio score of the average burning speed of each laboratory
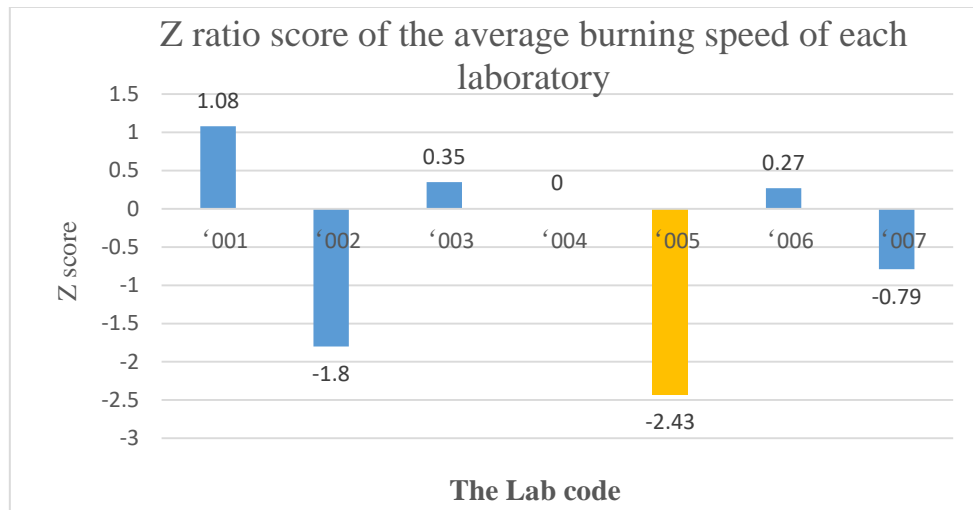

Figure 1: Classified statistics of Z-distribution of average combustion rate

7. Technical Analysis and Recommendations

7.1 The Combustion Rate Statistics of Each Laboratory are as Follows:

The Burning speed (V), ratio of burning distance L to burning time T, mm / min

Burning speed $V = 60\frac{L}{T}$ [mm/min]

In the formula:

Burning distance, mm

time taken for burning distance L, s
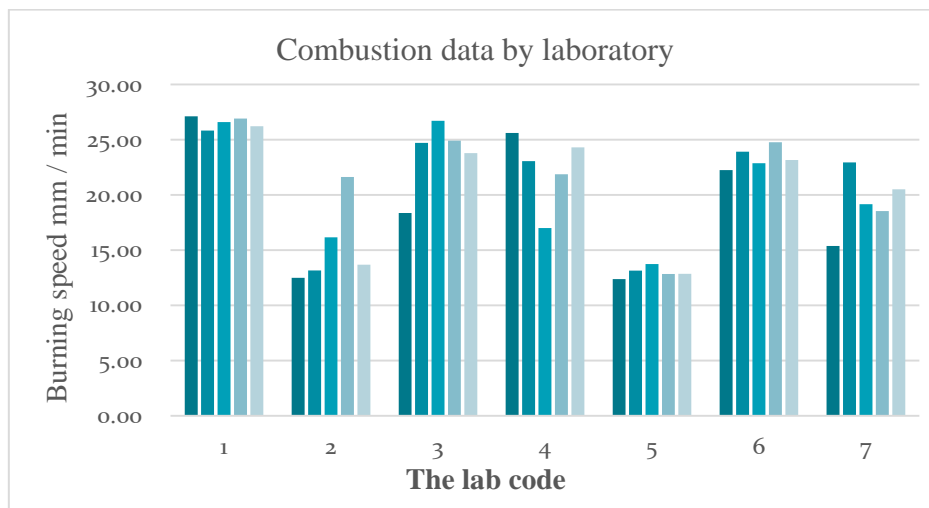
7.2 Combustion Data from the Laboratory

Figure 2 Combustion data of each laboratory

Observing the combustion data of each laboratory can be seen:

(1) Code 005 laboratory ($2 < |Z| < 3$) in which the problem occurred. Observing The results of the parallel sample data in this laboratory, it was found that the burning speed was generally low. It is recommended that the laboratory pay attention to find the cause of the problem.

(2) There are many influencing factors on the results of the combustion test, such as wind speed control (using an anemometer to measure the vertical wind speed at 100mm before and after the burner at a fixed position is 0.10 ~ 0.30m / s), and the heat value of the gas source is about 35 ~ 38MJ / m3 of flammable gas, pre-treatment conditions (23 ° C, 50% RH, 48hr), and whether to use

barbed wire, engineer's operation methods such as whether the sample is placed horizontally, when conducting a series of tests or repeated tests, ensure the next time Before the test, the maximum temperature in the combustion chamber and the sample holder should not exceed 30 ° C. Even within the tolerances specified by the standard, some differences can still occur.[3] The laboratories for ｜Z｜ <2 are: 001, 002, 003, 004, 006, 007. Results from these laboratories can be considered satisfactory.

(3) There are 001, 003, 005, 006 laboratories with more stable and uniform combustion results. The rest of the laboratory data fluctuates greatly, and you need to pay attention to the consistency and standardization of the operation.

8. Conclusion

Through this interlaboratory comparison test, the combustion test capabilities of each laboratory were determined. Through analysis of comparison results, find out the differences between laboratories. The laboratory results of codes 001, 002, 003, 004, 006, 007 are satisfactory. 005 Laboratory combustion results are suspicious. [4,5] In addition, the laboratory combustion data of codes 001, 003, 005, and 006 are stable and uniform, and the laboratory combustion data of codes 002, 004, and 007 fluctuate greatly. There are many factors that affect the results of the combustion test, such as wind speed control, air source heating value, pretreatment conditions, whether to use barbed wire, and engineer's operating methods. It is hoped that the laboratories will pay attention to every detail to improve the accuracy of the test.

9. Appendix

9.1 Combustion Test Results and Evaluation of Results

Table 2. Appendix A-1: Combustion Test Results and Results Evaluation

The Lab code	The Burning speed, mm/min								Z Value (average burning speed)
	1	2	3	4	5	Average burning speed	Statistical burning speed	Burning speed	
001	27.12	25.83	26.60	26.92	26.23	26.54	28.10	27.12	1.08
002	12.50	13.16	16.17	21.63	13.69	15.43	26.63	21.63	-1.80
003	18.37	24.72	26.71	24.92	23.79	23.70	33.19	26.71	0.35
004	25.61	23.06	17.00	21.87	24.31	22.37	32.30	25.61	0
005	12.38	13.15	13.74	12.85	12.86	13.00	14.49	13.74	-2.43※
006	22.25	23.92	22.88	24.78	23.16	23.40	26.33	24.78	0.27
007	15.38	22.95	19.17	18.54	20.51	19.31	27.63	22.95	-0.79

Note: The results marked with "§" are not satisfactory; while the results marked with "※" are problematic;

9.2 Appendix B Sample Preparation and Sample Uniformity Evaluation Report

9.2.1 Appendix B-1 Sample Preparation Report

The sample used in this interlaboratory comparison was prepared from PP with the grade of B-M02 (J340) as the sample preparation raw material.
The sample preparation is made by injection molding. The Haitian HTF160X1 injection molding machine is used. According to GB / T 17037.1-1997 "Preparation of injection molding samples of thermoplastic materials. Part 1: General principles and preparation of multi-purpose samples and strip samples" General conditions, as well as other important process parameters of injection molding obtained from empirical verification. After adjusting the injection molding process

conditions, start injection in the full cycle mode, discard the first 10 molds, start sample collection from the 11th mold, and use this as the first mold to collect the samples. A total of 53 samples were prepared in this laboratory comparison. One sample from every 10 large plates is used as a sample for homogeneity testing, and they are arranged according to the sequence number.

9.2.2 Appendix B-2 Sample homogeneity report

Due to the molecular orientation in the injection-molded sample, a maturation time of at least 7 days is required to eliminate the internal stress caused by the factor orientation and stabilize the sample performance. The samples for this interlaboratory comparison were aged for 7 days after injection molding. After being adjusted for more than 48 hours, according to the standard requirements, ATLAS HMV combustion chamber was used for testing. The test results are shown in Table B-2.

Table3. Appendix B-2 Test results of uniformity of combustion samples

Sample No.	Burning length [mm]	Burning duration [s]	Burning rate [mm/min]	Rating
1-#	254	525	29.0	BR < 100
2-#	254	562	27.1	BR < 100
3-#	254	547	27.9	BR < 100
4-#	254	549	27.8	BR < 100
5-#	254	535	28.5	BR < 100
Bmax	[mm/min]		29.0	
Bstat	[mm/min]		30.22	
Bave	[mm/min]		28.06	
standard deviationσ	/		0.72	
0.3NIQR	/		1.158	

It can be seen from the above table that the standard deviation of the combustion rate test results of the five samples is less than 0.3 times the NIQR of this interlaboratory comparison, indicating that the samples are uniform and can meet the requirements.

References

[1] CNAS-GL02: 2014 Guidelines for Statistical Processing and Capability Evaluation of Proficiency Test Results

[2] CNAS-GL03: 2006 Guidelines for evaluating homogeneity and stability of proficiency testing samples

[3] DBL 5307-2018 Flame retardant properties–Interior trim parts Requirements and test specifications

[4] GB/T 17037.1-1997 Preparation of injection-molded specimens of thermoplastic materials Part 1: General principles and preparation of multi-purpose specimens and strip specimens

[5] GB 8410-2006 combustion characteristics of automotive interior materials