Proficiency Testing for Determination of Ductility of Asphalt by Iteration Robust Statistic Technique

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Abstract. In order to investigate and improve the detection level of ductility of asphalt of laboratories, proficiency testing provider PT0031 (CNAS) has organized a proficiency testing program of ductility of asphalt of CNCA, and 87 laboratories from 21 provinces/cities/municipals and Taiwan took part in this PT. This work introduced the implementation process of proficiency testing for determination of ductility of asphalt, including the sample preparation, homogeneity and stability test, and the results of statistics of iteration robust statistic technique and analysis, summarized and analyzed different test standards widely used in the laboratories, and put forward the technological suggestions for the improvement of test quality of ductility. Satisfactory results were obtained by 85 laboratories, amounting to 97.7% of the total participating laboratories.

1. Introduction
Asphalt is a very important material in national economic construction and technological development and has a wide range of used. In 2018, the apparent consumption of China's asphalt market is about 29 million tons, of which domestic asphalt production is more than 24 million tons, and the import volume is about 5 million tons. It is mainly used as infrastructure materials, raw materials and fuels, and its application range is transportation, construction, agriculture, water conservancy engineering, industry, civil and other departments, roads and buildings for waterproof treatment, coating the roof, manufacturing linoleum, sleepers, electric pole anti-corrosion, steel pipe waterproof thin layer, etc. [1].

The ductility of asphalt is obtained by stretching the ends of the standard test mold until the length of the fracture at a specified speed and temperature. The ductility is related to the low-temperature cracking performance of the pavement, and is the final index for evaluating asphalt plasticity [2–4]. Therefore, it is of great significance to accurately determine the ductility of asphalt. At present, JTG E20-2011[5] and GB/T 4508-2010[6] are commonly used for the determination of asphalt ductility. The ability to verify the asphalt ductility is less active. This work takes the verification of the ability of asphalt ductility as an example to identify the differences between the results of asphalt ductility measurement by each laboratory, increase the comparability of the results between the laboratories, and improve the detection level of the laboratory. Through this program, the participating laboratories can not only understand their own testing capabilities, but also find gaps with similar laboratories, and promote the laboratory to further improve the testing technology and laboratory management level. Also provide the information for Certification and Accreditation Administration of the People's Republic of China (CNCA) and China National Accreditation Service for Conformity Assessment (CNAS) to regulate the accredited laboratories in this field [7].
2. Experimental

2.1. Instrumentation and Reagents
DD3-150 asphalt ductility tester (Petrotest, Germany), T 6060 Heraeus oven (Thermo Fisher Scientific, American), FP51 temperature control circulator (JULABO, Germany). The test water is water filtered through a Milli-Q pure water system.

2.2. Test Sample
(1) Sample Design: The test sample is an asphalt sample (approx. 200g, solid at room temperature) contained in an iron can. Each laboratory will receive a bottle of asphalt, which can be tested directly.

(2) Sample Preparation and Release: The asphalt sample in the stainless steel drum was sealed and heated at 130 °C and stirred uniformly, and then packed into 180 cans of samples, each of which was about 200 g. The iron cans containing the samples were wrapped in a bubble film, placed in a hard courier box, and distributed to each laboratory by courier.

Participate in the laboratory and receive the "Operation Guide", "Confirmation Form for Receiving Status of Tested Items" and "Report Report". The samples received should be kept in a sealed state under normal temperature conditions.

(3) Homogeneity Test: From the prepared samples, 10 bottles of samples were selected for homogeneity detection randomly. Ductility was tested according to JTG E20-2011. The single factor analysis of variance of F test and Ss≤ 0.3σcriterion will be used according to the CNAS-GL003:2018[8] for statistical processing of test results.

(4) Stability Test: 6 samples were selected for stability test randomly. T test method for assessing the stability of samples according to the CNAS-GL003, the results of each test and stability test of homogeneity data were compared, if \( t \) value is less than the significance level \( \alpha \) (usually \( \alpha=0.05 \)) degree of freedom \( (n_1+n_2-2) \) the critical value of \( t \alpha(n_1+n_2-2) \), the average value between the average and the stability test of homogeneity there was no significant difference, the sample is stable for proficiency test.

2.3. Test Method
This capability certification is recommended in accordance with JTG E20-2011 or other technical equivalent or equivalent standards.

2.4. Statistical Design
The z score for a proficiency test result \( x \) is calculated as:

\[
z = \frac{(x - X)}{\sigma}
\]

where
\( x \) is laboratory test results, and
\( X \) is the assigned value, and
\( \sigma \) is the standard deviation for proficiency assessment.

The ability to verify the plan in accordance with the ISO 13528:2015 (E) [9], computing robust average algorithm to estimate the sample population mean, the average value is specified as a robust, robust standard deviation as the standard deviation for proficiency assessment. The main process of the algorithm A is as follows:

Denote the \( p \) items of data, sorted into increasing order, by:

\[x_1, x_2, ..., x_p\]

The robust mean values and robust standard deviations for these data are calculated as follows. The initial value of the calculation is as follows (MED means median value):

\[
x^* = \text{med} x_i \quad (i=1, 2, ..., p)
\]

\[
s^* = 1.483 \times \text{med}|x_i - x^*| \quad (i=1, 2, ..., p)
\]

According to the following steps, update and calculate:

\[
\delta = 1.5s^*
\]
For each \(i=1, 2, \ldots, p\), calculate

\[
x_i^* = \begin{cases} x^* - \delta, & \text{if } x_i < x^* - \delta \\ x^* + \delta, & \text{if } x_i < x^* + \delta \\ x_i, & \text{other}
\end{cases}
\]  \( (5) \)

Next, the new values are calculated:

\[
x^* = \frac{\sum x_i^*}{p}
\]  \( (6) \)

\[
s^* = 1.134 \sqrt{\frac{\sum (x_i^* - x^*)^2}{p-1}}
\]  \( (7) \)

So far, the first iteration was completed. The second iteration type (4) from beginning to end the formula (7), in turn, until the new robust average and robust convergence to the standard deviation of third decimal digits remained unchanged, and finally get the data robust average and robust standard deviation. The standard uncertainty of the robust mean is calculated as in formula\([10,11]\):

\[
u_x = 1.25 \times \frac{\sigma}{\sqrt{p}}
\]  \( (8) \)

Ability to validate the significance of each statistic and related computational methods see CNAS-GL002:2018\([12]\) standards. The conventional interpretation of \(z\) scores is as follows. A result that gives \(|z| \leq 2.0\) is considered to be acceptable. A result that gives \(2.0 < |z| < 3.0\) is considered to give a warning signal. A result that gives \(|z| \geq 3.0\) is considered to be unacceptable (or action signal).

3. Results And Discussion

3.1. Participation in Laboratory Distribution

A total of 87 laboratories submitted the test results of asphalt ductility, including China Railway System, Highway Engineering Industry, PetroChina Petrochemical System, Quality Supervision Department, Inspection and Quarantine System, Social Testing Agency and a small number of research institutes. The laboratories are distributed in 21 provinces, autonomous regions, municipalities directly under the Central Government.

3.2. Uniformity and Stability of the Sample

According to the method of uniformity in the 2.2 section in the same conditions for 2 times were calculated between mean square and mean square in the sample were 2.36, 1.36, F-measure computing statistics for 2.41, less than the critical value \(F_{0.05,9,10} = 3.02\); the calculation of statistics value \(S_s\) is 0.98, less than the critical value of \(0.3\sigma(8.5)\). It is showed that the sample was homogeneous at 0.05 significance levels.

Stability is calculated by 2.2 in section S before \(S\) before =1.31, \(S\) after =1.32, and the calculated statistic \(t=1.39\) is less than the critical value \(t_{0.05/2} = 2.23\). It showed that the sample was stable at 0.05 significance level.

3.3. Test Results and Mathematical Statistics

(1) Test Result: Test results were presented in 87 laboratories, and the results were shown in table 1.

| No. | Result | No. | Result | No. | Result | No. | Result | No. | Result |
|-----|--------|-----|--------|-----|--------|-----|--------|-----|--------|
| 01  | 34.5   | 19  | 20.8   | 37  | 29.2   | 55  | 24.3   | 73  | 12.3   |
| 02  | 16.4   | 20  | 31.8   | 38  | 46.1   | 56  | 29.0   | 74  | 21.1   |
| 03  | 27.9   | 21  | 33.0   | 39  | 28.4   | 57  | 22.9   | 75  | 23.0   |
| 04  | 30.3   | 22  | 23.2   | 40  | 21.1   | 58  | 16.1   | 76  | 28.5   |
| 05  | 30.4   | 23  | 35.2   | 41  | 34.0   | 59  | 18.6   | 77  | 17.6   |
| 06  | 25.3   | 24  | 26.8   | 42  | 17.6   | 60  | 27.6   | 78  | 38.7   |
| 07  | 32.8   | 25  | 22.9   | 43  | 31.9   | 61  | 21.2   | 79  | 33.5   |
| 08  | 24.9   | 26  | 15.0   | 44  | 29.6   | 62  | 23.7   | 80  | 33.1   |
| 09  | 34.6   | 27  | 24.4   | 45  | 26.7   | 63  | 32.0   | 81  | 24.9   |
Statistics the frequency of the laboratory results, grouped according to the result, and frequency histograms, as shown in figure 1.

![Histogram showing frequency distribution of testing results](image)

**Fig. 1 Histogram showing of frequency distribution of testing results**

Frequency distribution diagram shows that although there are individual extreme values, the overall data of this capability verification result appear to be in the middle and less at both ends.

(2) Mathematical Statistics of Test Results: The results show that the ability of statistical validation plan, there are 85 laboratories for the detection results with satisfactory results, accounting for 97.7% of the total number of detection results; 2 laboratories results for the problem, accounting for 2.3% of the total number of detection results. For statistical parameters, see table 2.

| Test item                      | parameters |
|-------------------------------|------------|
| Result number                 | 87         |
| Assigned value (cm)           | 26.1       |
| Robust standard deviation (cm)| 7.4        |
| Standard uncertainty (cm)     | 1.0        |
| Robust coefficient of variation (%) | 28.4     |
| Minimum (cm)                  | 12.3       |
| Maximum (cm)                  | 46.1       |
| Max-Min (cm)                  | 33.8       |

In order to clearly indicate the results of each laboratory's ability validation program, the Z ratio are arranged in order of size (Fig. 2). Each column is marked with the code of the laboratory. From the bar chart, it is easy for each laboratory to compare its results with the results of other participating laboratories to find out where the results are in the plan.
Technical Analysis

The laboratories participate in this proficiency test adopted three standard methods, JTG E20-2011, GB/T 4508-2010 and ASTM D113-17 [13], and 93.5% of the laboratories used JTG E20-2011 test standard.

For the determination of asphalt ductility, the above standards are basically consistent with the key equipment and material specifications such as the assembled mold, brass base plate, ductility tester and device combination, and the heating rate (5.0 cm/min±0.25 cm/min). The test and operating conditions are basically the same.

For the determination of asphalt ductility, there are some factors affecting the measurement results. The following points can be noted in the testing process for each laboratory: 1) The release agent should be applied to the inner surface of the bottom plate and the brass mold, and the assembled mold should be placed horizontally. Full contact with the bottom, in order to prevent inconsistency in thickness and influence, the assembly of the mold is very important for the molding of the sample.

2) The sample should be heated and melted to prevent local overheating, and pay attention to heating time to prevent overtime aged.

3) When asphalt pouring into the mold, pay attention to reciprocating from one end of the mold to the other end, injecting into the mold slowly, and higher than the mold slightly, control the speed to prevent unevenness, and pay attention to not allow air bubbles to mix in the filling.

4) According to the standard requirements, after the test piece is cooled in the air, and then placed in the water bath, the temperature of the water bath should be kept constant.

5) When the asphalt of the mold is scraped out with the hot knife and flushed with the mold, pay attention to the scraping. The method should be scraped from the middle of the mold to both sides, and the two sides should be smoothed.

6) The normal test should be drawn into a cone until the actual cross-sectional area is close to zero at the time of the fracture, otherwise it is redone.

4. Conclusion

The capability verification of asphalt ductility measurement was discussed. 87 laboratories from 21 provinces, cities, municipalities and Taiwan participated in this proficiency testing activity. The results were analyzed and evaluated based on robust statistical techniques. The satisfaction rate of asphalt ductility test results was 97.7%, and the robust coefficient of variation was 28.4%, which objectively reflected the detection level of each participating laboratory and promoted laboratory testing technology. And further improvement in the level of laboratory management. In particular, laboratories that are dissatisfied with the test results of this proficiency testing program should actively find the cause and take measures to ensure that the daily test results are accurate and reliable.

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