Standard and verification regulation of asphalt wax content measuring instrument

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Abstract. Asphalt wax content measuring instrument is a key equipment used to measure the wax content in petroleum asphalt. It is mainly used to measure the wax content of road asphalt, and is widely used in transportation industry and petroleum industry. This project focuses on the research of asphalt wax content meter, investigates the practicability of the current measurement verification regulation, excavates the deep-seated reasons in the process of metrological verification, corrects the deficiencies existing in the existing measurement verification regulations, and adds new requirements in line with the construction of transportation power, so as to make the measurement verification regulations meet the requirements of current engineering construction. Based on the above reasons, the research on asphalt wax content meter is carried out. According to the existing measurement verification regulations, combined with the characteristics of equipment value and the requirements of transportation power, it can guide the production of such equipment, improve the quality, and guide the test and detection personnel have a clear understanding of the status of the equipment in the daily detection process. The measurement verification regulations can ensure the wax content of asphalt in the construction process of high-grade highway. It has a positive effect on promoting the construction of transportation power.

1. Introduction
With the rapid development of my country’s national economy, my country will increase investment in the construction of basic industries, especially the construction of road and railway transportation, which will increase the demand for asphalt materials [1]. The development of the domestic asphalt market and the improvement of asphalt product quality will receive more attention, and the market share of domestic high-grade highway heavy-duty asphalt products will also increase. Wax content is a key indicator that affects the performance of road petroleum asphalt [2]. Many studies have shown that the level of asphalt wax content has a great impact on asphalt mixture pavement, such as softening at high temperature, hardening and brittleness at low temperature, and the presence of wax easily peeling off asphalt and aggregate, Wax wrapped on the surface of the stone reduces the anti-skid performance of the asphalt mixture pavement [3].

The determination of petroleum asphalt wax content is a relatively complicated problem, and there are many test methods. Currently, there are: German DIN52015 distillation method, French NF-T66, IR method, Iatroscan method, etc. The cracking distillation method is widely used in China, and the wax content determination method specified in T0615 in the latest SH/T 0425-2003 "Petroleum Asphalt Wax Content Determination Method" and JTG-E20 "Highway Engineering Asphalt and Asphalt Mixture Test Regulations" also uses distillation method [4]. However, in the actual analysis of the wax content, it was found that the test error was large, the accuracy of the determination result was...
not high, and the test repeatability was also poor. The reason for this problem is that the asphalt itself is a relatively complex mixture [5]. The most important thing is that the test only has a test method. For asphalt wax content testing equipment, there are neither industry standards nor verification procedures, which leads to the asphalt wax content analyzer. The quality of the equipment is uneven, and the instability of the equipment leads to huge errors in the test, which brings great trouble to the determination of the asphalt wax content. The verification procedure is the key to guarantee the value of the asphalt wax content meter [6]. There is an urgent need to formulate equipment verification and standardize the first verification, follow-up verification, and in-use inspections, so as to ensure the accuracy of the test results [7].

2. Research Overview at Home and Abroad

2.1. Research on The Wax Content of Petroleum Asphalt
The domestic measurement of the wax content of asphalt mostly adopts the distillation method, which is the content of the wax crystallized under the specified solvent and low temperature after the oil is distilled by the distillation method, expressed as a mass percentage. Foreign methods for determining wax content include: steaming method, sulfuric acid method, and composition analysis method [8]. At present, there is no national standard for the determination of petroleum asphalt wax content in my country, and most of them adopt the SH/T 0425-1992 "Petroleum Asphalt Wax Determination Method" recommended by Sinopec Corporation. This method is equivalent to the German industrial method DIN and the Japanese JPN method, reflecting the technological level of advanced countries. This method is more conditioned and requires harsher operation. During the test, if you do not carefully follow the operating procedures and deviate from the test conditions, the accuracy of the measurement results will be significantly affected.

2.2. Research on Measuring Method of Asphalt Wax Content
Wax content is a very important indicator in road petroleum asphalt. The presence of wax in asphalt is extremely unfavorable for heavy traffic roads with strict requirements. The methods commonly used in various countries for the determination of wax content in petroleum pitch are: German DIN52015 distillation method, French NF-T66, IR method, Iatroscan method, ASTM American standards, etc. The cracking distillation method is widely used in China, including the latest SH/T0425-2003 and JTG-E20. Among them, the T0615~2011 asphalt wax content test standard is analyzed, the factors affecting the accuracy of the measurement results are analyzed and some solutions are proposed. The analysis results show that during the operation of the pyrolysis distillation method, the pyrolysis distillation effluent rate, distillate sampling volume, freezing washing and filtering, etc., are the main factors affecting the determination results [9].

Since the determination of the wax content in asphalt by the pyrolysis method is a very complicated test, before the test analysis, first ensure that the test environment is clean and tidy, and the items used for the test must be prepared in an orderly manner. The weighing balance must meet the accuracy standard and place it. In accordance with the laboratory's specifications, the asphalt wax content analyzer and the vacuum drying oven must be regularly calibrated, and can be used only after the calibration is qualified to ensure the accuracy and reliability of the analysis data.

Pyrolysis distillation is a very important link in the determination of wax content. The quality and quantity of the obtained distillate directly affect the accuracy of the analysis results. Among them, the influence of heating distillation equipment, the influence of cracking rate, and the influence of distillate sampling volume have become key factors [10].

3. Research on Technical Index of Verification Regulation
This article is in accordance with the requirements of JJJF 1002-2010 "Rules for the Compilation of National Metrological Verification Regulations" to formulate the verification regulations for the asphalt wax content measuring instrument. The content and format are consistent with JJJF 1002. The
specific content of the regulations includes scope, cited documents, terminology, overview, measurement performance requirements, general technical requirements, measurement instrument verification control (including verification conditions, verification items, verification methods, and verification results Processing and verification cycle).

3.1. Study the Technical Indicators of the Asphalt Wax Content Measuring Instrument
The measurement technical indicators of the asphalt wax content measuring instrument are the basis and important basis for the design of measurement standards. Study the working principle, input and output of the asphalt wax content measuring instrument testing equipment, according to the characteristics of the instrument, study the measurement technical indicators of the asphalt wax content measuring instrument, comprehensively regulate the parameters of the equipment, and propose the corresponding test methods and control Indicators. This project conducted in-depth research on this part of the content, summarized and summarized on the basis of experimental data, and found a suitable method to complete the formulation of the regulations and standards.

3.2. Cracking distillation system
According to investigations and experiments, the indicators of the distillation flask are defined as follows. The diameter of the pyrolysis distillation flask is 85.7mm±3.0mm, the diameter of the bottle mouth is 16.0mm±1.0mm, and the inner diameter of the distillation tube is 10.0mm±0.5mm. When the vertical adjustable high temperature furnace maintains the temperature at 550℃, the error does not exceed ±10℃.

Regarding the measurement research of the distillation flask, the research team found during the institutional investigation that some organizations believed that the pyrolysis distillation device, that is, the distillation flask and the vertical adjustable high-temperature furnace were not equipment for measuring wax content, but a distillation pyrolysis equipment. After investigation, the research team believes that it should be added to the regulations mainly because the SH/T 0425 petroleum asphalt wax content determination method and the JTG E20 highway engineering asphalt and asphalt mixture test regulations both give the technical requirements for distillation and cracking of this part of the equipment. If the regulations do not have this part, it would have a great impact on the application of the equipment.

According to the research of the research group, it is believed that the indicators of the distillation flask should be the pyrolysis distillation flask diameter of 85.7mm±3.0mm, the mouth diameter of 16.0mm±1.0mm, and the inner diameter of the distillation tube of 10.0mm±0.5mm. Due to the JTG E20 highway engineering asphalt and asphalt mixture test regulations, some indicators are not consistent with the actual due to printing or definition errors, such as the total height of the flask of 120mm and the bottle mouth length of 135mm conflict. In addition, the center position of the elbow and the length of the flask center 73mm and the dimensions of the condenser tube are difficult to measure, as shown in Figure 1.

Figure 1. Examples of incorrect printing definitions in test procedures and correct identification

In the SH/T 0425 petroleum asphalt wax content determination method, the situation is similar. The difference is that the nozzle of the condenser tube is sharp. Research was also carried out when this regulation was written, and it was found that both types of equipment in the highway industry exist. Therefore, the defined indicators can meet the requirements of the two test methods.
When the high temperature resistance furnace maintains the temperature at 550℃, the error does not exceed ±10℃. In the actual test, the heating capacity is mainly determined by adjusting the height, and some institutions adopt the heating method of blowtorch. The temperature accuracy requirements are not high. However, since two drops per second are required during distillation, the temperature fluctuation of the resistance furnace is required to be good to ensure that the temperature is stable and uniform. Therefore, the requirements of uniformity are mainly considered when writing this regulation. Distillation requires 5-8 minutes of initial distillation, the whole process is within 25 minutes, and the distillation process does not exceed 20 minutes, so this regulation sets the test time as 20 minutes.

As shown in Figure 2, the research team also used a thermal imaging camera to conduct experimental observations. During the distillation process, the temperature change is as shown in the figure below. It can be clearly seen that the heat is concentrated in the distillation flask during the distillation process, and the condensation effect of the condenser tube is very obvious. The control in the establishment process of the index of the distillation flask and the index of the resistance furnace is more effective and reasonable.

![Figure 2. Thermal imaging camera observes the temperature change of the distillation flask during the distillation](image)

3.3. Constant Temperature Cold Bath
According to investigations and experiments, the requirement of the constant temperature cold bath is to keep the cooling temperature at -20℃ and the error does not exceed ±0.5℃.

JTG E20 Highway Engineering Asphalt and Asphalt Mixture Test Regulations require that the minimum temperature of the equipment should reach -30℃, while the SH/T 0425 petroleum asphalt wax content determination method requires the minimum temperature of the equipment to reach -22℃. In the process of writing this regulation, it was found that the conventional compressor refrigeration is used, and the equipment temperature cannot reach -30℃. If it reaches -30℃, two compressors must be used, which greatly increases the procurement cost of each organization, and the test temperature is 20℃, if the constant temperature can be maintained at -20℃, -30℃ is not necessary.

The wax content test needs to ensure temperature uniformity and stability. In the test procedure, the test time is 15 minutes, and the verification procedure also takes 15 minutes. The research team found through experiments that the temperature of the test tank is still stable when the external temperature is high, as shown in Figure 3.

![Figure 3. Wax content experiment temperature uniformity and stability experiment](image)

3.4. Sand core filter funnel for cooling filter
The sand core filter funnel of the cooling filter is the key to the entire wax content detection equipment, and its accuracy is directly related to the measurement result of the wax content. After investigation and testing, the research team formulated the following indicators. The pore size limit of the sand core filter funnel is 10μm~16μm; the pore size uniformity of the sand core filter funnel is not less than...
10μm; the permeability of the sand core filter funnel (pressure difference 100pa) is not less than 1.0cm³/(cm³•min).

The SH/T 0425 petroleum asphalt wax content determination method stipulates that the filter plate pore size is 20-30 microns, and the JTG E20 highway engineering asphalt and asphalt mixture test regulations stipulate that it is 10-16 microns. According to the preparation instructions and test methods of E20, the P30 filter plate specified by SH/T 0425 has been deleted from the pore size, classification and grade of the GB/T 11415 laboratory sintered (porous) filter. At the same time, considering the application of my country in the highway industry, decided to adopt the E20 regulation, namely the P16 filter plate.

P16 filter plate adopts GB/T 11415 laboratory sintered (porous) filter pore size, classification and grade requirements, but this regulation has the following problems. a. It has been compiled for a long time, and some test methods have been difficult to use, b. This standard is mainly for filter plates, and specific devices are still required for sand core funnels used on highways. In the process of formulating the method, the project team also referred to GB/T 5250 permeable sintered metal material fluid permeability measurement and GB/T 5249 permeable sintered metal material bubble test pore diameter measurement related regulations, determined its main experiment method. According to the method requirements, the research team redesigned the calibration device, the device for measuring the aperture and the device for measuring the air permeability. And its corresponding requirements. The device for measuring the aperture is composed of an air inlet, a precision control valve, a sand core filter funnel, a pressure gauge and a drip bottle. The schematic diagram of the structure is shown in the figure 4.

Figure 4. Schematic diagram of measuring aperture device

The technical requirements of the device for measuring the aperture are: the range of the pressure gauge is not less than 50kPa, and the accuracy level is 0.1. The measured air permeability is composed of 1 regulator tube, 2 air inlets, 3 precision control valves, 4 flow meters, 5 sand core filter funnels and 6 pressure gauges. The schematic diagram of the structure is shown in Figure 5.

Figure 5. Schematic diagram of the device for measuring air permeability

The technical requirements of the device for measuring air permeability are: the range of the flowmeter should include (100~200) cm³/min, and the accuracy level is 0.5; the range of the pressure gauge is not less than 50kPa, and the accuracy level is 0.1.

4. Conclusion and outlook
In this project, the existing verification procedures of the asphalt wax content measuring instrument were studied, and the deficiencies of the existing procedures were modified. Some measurement parameters were added to meet the current engineering quality requirements, so that the use of
equipment can better meet the engineering inspection demand. Through this revision of the regulations and its use in the national transportation industry, it has won unanimous praise from engineering inspection agencies and has been generally recognized by the market. The issuance of the revised regulations for the verification of asphalt wax content measuring instrument will provide an effective operating basis for the measurement department of the transportation industry, and also provide a test basis for the stable and reliable performance of the asphalt wax content measuring instrument in the industry.

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Footnotes should be avoided whenever possible. If required they should be used only for brief notes that do not fit conveniently into the text.

References
[1] Ji Hongbin. Analysis of factors affecting the determination of petroleum asphalt wax content[J]. China Petroleum and Chemical Standards and Quality, 2018, v.38;No.477(19):55-56.
[2] Zhao Peng, An Changwei. Discussion on the determination method of road petroleum asphalt wax content in asphalt pavement[J]. Science and Technology Information, 2012, No.415(23):359.
[3] Li Conghao, Zhang Xiaolin. Discussion on the determination of petroleum asphalt wax content by distillation[J]. Petroleum Asphalt, 2008, No.92(02):56-58.
[4] Wang Chuanmin. Influencing factors and operation points for determination of wax content in petroleum asphalt[J]. Petroleum Asphalt, 2006, (01):71.
[5] Tao Zizheng, Yang Li, Mo Shuhong. Discussion on the determination of road petroleum asphalt wax content[J]. China Science and Technology Information, 2005, (12):174-175.
[6] Wang Chuanmin, Chang Yuyan, Cai Jinpo. Analysis of Influencing Factors on the Accuracy of Determination of Wax Content in Petroleum Asphalt[J]. Petroleum Asphalt, 2004, (02):43-45.
[7] Yao Liqun, Dong Zhaode. Research on the determination method of petroleum asphalt wax content[J]. Xinjiang Petroleum Science and Technology, 2001, (02):63-67.
[8] Sun Yanzhong. Discussion on the wax content in road petroleum asphalt[J]. Jilin Transportation Technology, 1994, No.54(03):48-49.
[9] Zhang Changxiang, Zhang Yuzhen, Guo Yansheng, Chen Jijun. On the wax content in petroleum asphalt (4)—The influence of wax content on the properties of asphalt[J]. Petroleum Asphalt, 1992, (01):48-50+27.
[10] Li Yongzhen, Zhang Hemeng, Zhuang Wei, Fan Liang. Discussion on Influencing Factors of Determination of Petroleum Asphalt Wax Content[J]. Petroleum Asphalt, 2015, v.29; No.133(01):69-72.