# STANDARD TESTING METHODS FOR BITUMEN AND WAX

**Bitumen is a heterogeneous crude oil by-product that is composed of complex hydrocarbon chains. It is commonly used for asphalt roads and roofing due to its hydrophobic qualities, adhesion, hardness, and general versatility [1]. Bitumen needs to be put through various quality assurance tests to confirm its suitability for different functions, most of which are established through the ASTM, that search for specific properties that arise from excess waste products like oil, water, and waxes; Waxes, specifically, have an ambivalent impact on bitumen depending on its intended function. In this paper, the various ASTM testing methods curated for bitumen and waxes will be outlined, as well as their significance in industry use.**

Distillation tests can be used to separate water or oil residue from bituminous material to determine its purity and inherent market value. Bitumen with a high-water content is inherently less desirable, especially when used for asphalt, as water causes the bitumen to foam when heated above 100 °C [2]. This can result in a cracked and undesirable appearance and reduced resilience of the asphalt. The acceptable amount of water in bitumen is 0.2% by weight to reduce this likelihood [3, 4].

To successfully test and ensure that this threshold is maintained, distillation through a Dean and Stark apparatus, as outlined in the ASTM D95, is utilized. This test method determines the volume of water in petroleum products in the range of 0% to 25% through distillation [4]. The Dean and Stark apparatus connects to a metal still and a condenser to separate water from bitumen. The apparatus is an addition to a reflux condenser that condenses the solvent and water into a separate trap as opposed to the still. The higher density of the water in the water-immiscible solvent will result in two distinct layers forming that can be separated into a beaker. As the volume of the solvent increases towards the top of the trap, the solvent will begin to overflow back into the still, thus maintaining solvent volumes while extracting the water [4].

The ASTM procedure for carrying out this test consists of performing standard reflux by mixing the bitumen sample and aromatic solvent and heating until reflux begins. Then, as the two distinct water and solvent layers begin to appear in the Dean-Stark apparatus, wait until all the water is condensed from the still. This can be determined by continuing reflux for 5 minutes after the water level in the Dean-Stark apparatus, has stabilized. Once water levels become stabilized, the volume of the water collected in the trap must be observed and plugged into the following formula:

$$\frac{\text{(volume in water trap,mL) - (water in solvent blank,mL)}}{\text{Volume in test sample,mL}} \text{ *100 [5].}$$

The precision of this test, as is consistent with most ASTM tests, is dependent on repeatability and reproducibility. Repeatability is defined as the difference between successive tests obtained by the same operator under the same lab conditions, and reproducibility is the difference between independent tests of identical test material conducted in different labs [4] [5]. For entirely precise results to be achieved, multiple iterations of this experiment must be undergone with a level of error within acceptable ASTM D95 standards.

Koehler sells the Dean-Stark apparatus (K31800) equipped with everything necessary for reflux, including a metal still, gasket, O-ring seal, a ring burner, a 400 mL condenser, 10 mL and 25 mL traps, and mounting equipment. Additionally, Koehler sells an Aluminum alloy still (K31900), which can be used for oil and residue determination in emulsified asphalt. This is a further method of quality control that can be used to determine the oil and residue content of emulsified asphalt after distillation. The procedure includes standard distillation as per ASTM D244, in which the water content is separated from the asphalt leaving only the residue. A higher residue content indicates a higher concentration of asphalt binder which can enhance the adhesion and binding properties of the emulsion, whereas a lower residue content may result in a thinner asphalt film which could impact the asphalt's effectiveness [6].

Another important characteristic in bitumen testing is the softening point through the ring and ball test. This test determines when asphalt goes from semi-solid to liquid which is needed to test the proper climate for asphalt; asphalt with a lower softening point is not suitable for hotter climates, whereas asphalt with a higher softening point is more suitable for cooler climates [7]. The test is conducted by setting two balls on top of brass rings filled with hardened asphalt into a water bath that rises at 5 °C intervals [8]. Watch as the balls elongate the asphalt and note at what temperature the balls touch the bottom of the ball-centering apparatus. Koehler provides both an automatic and manual ring and ball apparatus, with the automatic having laser sensor detection for when the ball drops, a touch screen interface, and an integrated stirrer that makes the test easier and more exact. Koehler's K95100 Automatic Softening Point Apparatus is shown in Figure 1.

*Figure 1*

*K95100 Automatic Softening Point Apparatus*

The Ductility test involves pulling the bitumen apart while immersed in water to gauge its ductility. The Bitumen sample is attached to a rectangular machine with a water basin and is pulled apart at 5 cm/min; the time at which the bitumen snaps is noted. After the ductility test is complete, the sample sits for an hour and is moved back so that the two ends are just touching. Elastic recovery can be calculated through the following formula:

$$\frac{\text{Original elongation - elongation after completion}}{\text{of original elongation}} \text{ [9].}$$

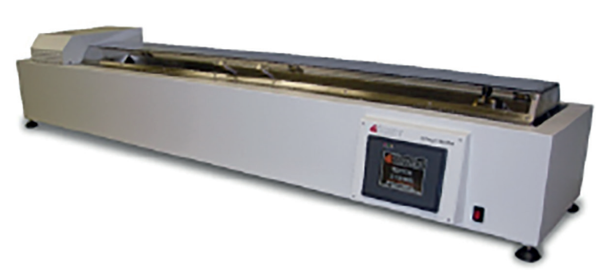

*Figure 2*

*K80050 Ductility Tester*

These tests measure the adhesive property of the bitumen as well as its ability to stretch. Concrete expands during the day and contracts at night, so the bitumen will easily crack without proper ductility and elastic recovery [10]. The elastic recovery test determines the recovery of the AR binder after being stretched. A higher elastic recovery correlates to reduced fragility and cracking [10]. Koehler's Semi-Automatic Standard Ductility Testing Machine features a maximum travel length of 150 cm, a preprogrammed ductility and recovery test method, a 6" LCD touch screen, and three sample testing stations.

The final tests are various methods of testing the aging of bituminous materials. These tests strive to simulate how the bitumen will react under common environmental stressors over some time. The Rolling Thin Film Oven (RSTO) and the Thin Film Oven Test (TFOT) test the aging of bitumen short-term during the production and paving period. The RSTO places vials of bitumen in a rotating oven at 325 °C to quantify the loss of volatiles and viscosity increase that can occur during the manufacturing process [11]. The bitumen can then be put through the property tests listed previously to note any changes in its performance. TFOT uses a similar mechanism to achieve the same result, it simply emits the rotating aspect of the RSTO and heats the bitumen directly. These devices comply with ASTM D2872 and ASTM D6 standards, respectively. Another aging device, the pressure aging vessel (PAV), uses pressurized air and elevated temperatures to simulate the accelerated aging of asphalt binders after several years. This device is useful to estimate how bitumen will fare after years of actual use and takes out the hassle associated with traditional aging tests.

Waxes are petroleum products that consist of long hydrocarbon chains that can be grouped into three general categories: paraffin, microcrystalline, and petrolatum. Waxes can have negative or positive impacts when found in bitumen, as they tend to decrease the strength of the asphalt binder, increase cracking at low temperatures, and reduce the viscosity at high temperatures. The properties of wax make it ideal for anti-corrosion additives for lubricants and anti-settling agents for fuel oils and paints [15, 16]. For this reason, it is important to test waxes in various ways.

The melting point of petroleum wax can be determined by cooling its molten wax in an air bath and waiting for it to solidify. As per ASTM D87, the wax is put into the melting point apparatus and cooled in an air bath surrounded by a water bath with periodic readings of its temperature being taken. This forms the cooling curve of the wax, which eventually plateaus when the phase changes from liquid to solid. The determination of the melting point of wax is necessary to determine its ability to hold shape and withstand heat so its proper usage can be determined. It is also important in the case of contamination, as the lower melting point of waxes in bitumen can negatively impact the quality of the bitumen. Koehler's Wax melting point apparatus, shown in Figure 3, has a nickel-plated air and water bath assembly and supports three test tubes vertically for multiple independent trials.

*Figure 3*
*K17500 Wax Melting Point Apparatus*

Finally, the Wax Appearance Point apparatus is used to determine the presence of wax in fuel at low temperatures. The apparatus cools the fuel in a water bath until a wax layer is formed and reported to the nearest 0.2 °C [18]. This device is another important component in determining unwanted waste in fuels, as wax crystals can hinder the performance of the fuel in machinery [18].

In summary, the quality and suitability of bitumen, a versatile crude oil by-product used in diverse applications like asphalt roads and roofing, are ensured through a comprehensive array of ASTM testing methods. Distillation tests, exemplified by the Dean and Stark apparatus, ascertain water content within acceptable limits, which is vital to prevent foaming and cracking during heating. Softening point evaluations through ring and ball tests guide climate-specific usage, while ductility and elastic recovery analyses gauge adhesive properties and stretch capabilities to avert cracking. Aging assessments like RSTO, TFOT, and PAV simulate real-world stresses over time, enabling performance prediction. Waxes, which negatively impact bitumen, undergo scrutiny via melting point determination and Wax Appearance Point apparatus to prevent detrimental influences on bitumen quality and fuel performance. Collectively, these tests safeguard bitumen's effectiveness, longevity, and applicability across construction and infrastructure domains.

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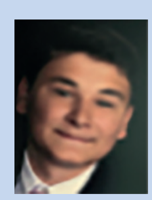
## Authors

**Dr. Raj Shah** is a Director at Koehler Instrument Company in New York, where he has worked for the last 28 years. He is an elected Fellow by his peers at IChemE, CMI, STLE, AIC, NLGI, INSTMC, Institute of Physics, The Energy Institute and The Royal Society of Chemistry. An ASTM Eagle award recipient, Dr. Shah recently coedited the bestseller, "Fuels and Lubricants handbook", details of which are available at ASTM's Long-Awaited Fuels and Lubricants Handbook 2nd Edition Now Available (https://bit.ly/3u2e6GY). He earned his doctorate in Chemical Engineering from The Pennsylvania State University and is a Fellow from The Chartered Management Institute, London. Dr. Shah is also a Chartered Scientist with the Science Council, a Chartered Petroleum Engineer with the Energy Institute and a Chartered Engineer with the Engineering council, UK. Dr. Shah was recently granted the honourific of "Eminent engineer" with Tau beta Pi, the largest engineering society in the USA. He is on the Advisory board of directors at Farmingdale university (Mechanical Technology) , Auburn Univ ( Tribology ), SUNY, Farmingdale, (Engineering Management) and State university of NY, Stony Brook ( Chemical engineering/ Material Science and engineering). An Adjunct Professor at the State University of New York, Stony Brook, in the Department of Material Science and Chemical engineering, Raj also has over 575 publications and has been active in the energy industry for over 3 decades. More information on Raj can be found at https://bit.ly/3QvfaLX

Contact: rshah@koehlerinstrument.com

**Mr. William Streiber and Ms. Gabriele Gentile** are part of a thriving internship program at Koehler Instrument company in Holtsville, and are students of Chemical Engineering at Stony Brook university, Long Island, NY where Dr. Shah is the current chair of the external advisory board of directors.

*Gabriele Gentile*



*William Streiber*

**Author Contact Details**

**Dr. Raj Shah, Koehler Instrument Company • Holtsvile, NY11742 USA • Email: rshah@koehlerinstrument.com • Web: www.koehlerinstrument.com**