# HOW TO PAVE THE WAY FOR EASIER BITUMEN ANALYSIS

**Schwechat is a community located in Lower Austria, to the south-east of Vienna. Mainly known for its airport connecting the eastern part of Austria to the rest of the world and for the Schwechat refinery, Schwechat has another significant institution to offer: the I.M.U. ("Institut für Mineralölprodukte und Umweltanalytik"), an independent testing laboratory and consulting office specialized in petrochemical and environmental analysis.**

It is no mere coincidence that I.M.U. settled down in Schwechat: the refinery is its main customer. Founded in 1996, I.M.U. employs around 50 people who cover all relevant analysis parameters for the requested sample analyses. In the well-equipped laboratories of I.M.U.'s seven specialized departments, fossil fuels, renewable raw materials, lubricants, petrochemical products as well as sediments, eluents, and microbiological samples are analyzed. Most analyses are performed according to standards and norms such as ASTM, EN, IP, ISO and AASHTO, but I.M.U. is also always ready to test new equipment and evaluate new procedures. One of the most requested samples to be analyzed is bitumen – or asphalt, as it is also called in some areas.

## What "bitumen" means to the world

Many different standards for analyzing relevant bitumen parameters are valid worldwide such as ASTM and ISO standards. Other standards, such as BS and DIN, are linked to certain countries. The nomenclature may vary in different standards on the same topic: In Europe a differentiation is usually made between the expressions "bitumen" and "asphalt". Americans tend to use the term "asphalt" for both materials.

Whatever the denotation, this substance is a highly sought-after building material worldwide because of its stability against environmental influences, mechanical and chemical stability, thermoplastic behavior and workability.

Bitumen, according to DIN 55946 and DIN EN 12597, is a dark high-molecular hydrocarbon mixture, its consistency ranging from semi-solid to glassy hard. Bitumen acts as a binder, while asphalt is a mixture of bitumen and crushed stone, mainly to be used for paving roads.

Bitumen is not only used for road construction, but also for roof tiles, in the protection of buildings against humidity, as a dampening coating for vehicles and dividing walls, as floor cover for frequently walked-on areas and for many other applications.

Due to its many uses, the demand for bitumen is high, and the specifications vary with respect to its intended use. No wonder that the daily number of bitumen determinations in the I.M.U. laboratories is so high.

Several tests are performed to characterize the bitumen samples such as the Ring-and-Ball test and the Fraass breaking point or tests with a dynamic shear rheometer because viscosity, rheological properties and the softening point are among the interesting characteristics. Orders of bitumen are usually delivered by volume, but the bitumen price is based on weight. Thus, the volume needs to be converted into weight by means of the bitumen's density. For that reason, another regular test on the daily to-do list is the determination of the density of bitumen.

## The classical way: Density measurement of bitumen with a pycnometer

Density measurements on bitumen have to be carried out with a pycnometer according to EN 15326 titled "Bitumen and bituminous binders - Measurement of density and specific gravity - Capillary-stoppered pycnometer method", the equivalent to ASTM D70. This determination method requires a lot of experience, skill, and time, not only for the measurement itself, but also for the cleaning procedure after each measurement. Samples are usually submitted to the laboratory in tin cans, and have to be brought to elevated temperatures to liquefy the bitumen so it can be transferred into the pycnometer according to a standard which clearly defines the duration and temperature for heating the sample.

The hot container is grabbed with a rubber glove. Sample is poured into the pycnometer to around three quarters of its capacity through its neck which is only about 2 centimeters wide – without splashing and without the bitumen touching the inner walls above the final filling level. Considering the possibility that in the hot, black liquid an air bubble could get trapped that

***Figure 1.*** *A hot bitumen sample is injected into the heated DMA 4200 M measuring cell*

cannot be seen, the norm requires double determinations. If the two results are not in agreement, a third determination has to be performed. After a determination is finished – this takes up to three hours –, the pycnometer has to be cleaned of any residues, which requires a considerable volume of solvent, e.g. toluene, and protective equipment.

"It takes more time and effort to clean the pycnometer than to perform the measurement," states Natascha Schultze, leading employee at the I.M.U. laboratory. "After the measurement I need to place the pycnometer in the oven for some time to soften the sample, and while pouring it out afterwards it hardens again. Then the pycnometer is soaked in toluene. The rest is removed manually by mechanical cleaning."

Fortunately, there is more than one way to determine the density. Several ASTM standards (ASTM D4052, D5002, D7777) specify the determination of density, relative density, and API gravity of all kinds of petroleum products with the oscillating U-tube method. The question arose: why couldn't this easier way of density measurement also be applicable to bitumen samples?

**Figure 2.** *Ms. Schultze handles the DMA 4200 M density meter effortlessly*

## As easy as it looks

A few months ago, I.M.U. acquired a DMA 4200 M density meter from Anton Paar GmbH in Graz, Austria. DMA 4200 M complies with the "Standard Test Methods ASTM D4052 Standard Test Method for Density, Relative Density, and API Gravity of Liquids" and "D5002 Standard Test Method for Density and Relative Density of Crude Oils" and therefore appeared to be a promising solution for determining the density of bitumen, especially for research purposes, but also as an eagerly awaited standardized determination method for bitumen as a fast, reliable, and user-friendly alternative to the pycnometer method.

With this density meter a measurement only takes around 12 minutes. Considering the number of bitumen samples per day and the potential of savings for future determinations it is not surprising that this technology appealed to Ms. Schultze. "When I get a chance I love to try out new technologies," states Ms. Schultze. "The DMA 4200 M density meter is very user-friendly. We not only save time and cumbersome cleaning steps, but we also get the information we need right away without needing to re-calculate the result – which is required with the pycnometer - as DMA 4200 M automatically converts the result obtained at the measuring temperature to the density at 60 °F / 15.6 °C or 77 °F / 25 °C as required by the standard."

Handling a bitumen sample with a DMA 4200 M is straightforward: once the sample is heated, bitumen is filled into a syringe as is shown in Figure 1, and injected into the measuring cell of the density meter. The measuring cell is also held at an elevated temperature which can be up to 200 °C. Even the fact that different samples require different temperatures to soften does not pose a problem: DMA 4200 M's software provides custom-tailored measuring conditions and allows users to create user functions which can be attributed to certain samples and recalled upon request.

## Density measurement easily withstands the comparison

To evaluate how precisely the results obtained with a pycnometer and a DMA 4200 M agree, Ms. Schultze dedicated a few weeks of parallel determinations to compare the traditional pycnometer results, carried out according to ASTM D70, with the results of the DMA 4200 M density meter. Ms. Schultze injected the heated sample with a syringe, as can be seen in Figure 2, into the density meter's measuring cell which was held at 120 °C. Once the density measurement was finished, the instrument automatically converted the density value obtained at 120 °C to the density value that corresponds to 15.6 °C, thus making the cooling of the sample or conversion with a table obsolete. This way, a lot of time is saved and no more additional working steps are required.

*Table 1. Density results obtained with a pycnometer and the DMA 4200 M density meter*

| | Pycnometer | | DMA 4200 M | | Difference |
|---|---|---|---|---|---|
| | Mean value [kg/m³] | Standard deviation [kg/m³] | Mean value [kg/m³] | Standard deviation [kg/m³] | [kg/m³] |
| **Sample 1** | 1042.34 | 0.499 | 1043.10 | 0.0524 | 0.758 |
| **Sample 2** | 1035.38 | 0.439 | 1036.38 | 0.0670 | 0.997 |
| **Sample 3** | 1029.08 | 0.422 | 1029.38 | 0.1031 | 0.297 |

In order to obtain comparison data, Ms. Schultze selected three different bitumen samples and measured the density of the samples at 15.6 °C (60 °F) with a pycnometer according to the ASTM standard, and with the DMA 4200 M density meter at 120 °C. DMA 4200 M automatically referred the obtained density value to the reference temperature of 15.6 °C. Repetitive determinations of each sample were carried out and the mean values and standard deviations calculated. All results are summarized in Table 1.

According to ASTM D70, results of two properly conducted tests by the same operator on the same material should not differ by more than 3.7 kg/m³ at 15.6 °C: the difference between the mean values of the results obtained with DMA 4200 M and the mean values obtained with the pycnometer is well below the standard's requirements. This suggests that the DMA 4200 M density meter is a suitable analysis method for bitumen samples and even more precise and efficient than a pycnometer.

The obtained comparison data are graphically displayed in Figure 3.

It can be seen from the graph that the results obtained with DMA 4200 M satisfactorily matched the pycnometer results. The repeatability shown with DMA 4200 M is much higher and easily meets the specification of the ASTM D70.

"I aim for a result accurate to the fourth decimal place," says Ms. Schultze. This requirement is met easily and effortlessly with DMA 4200 M.

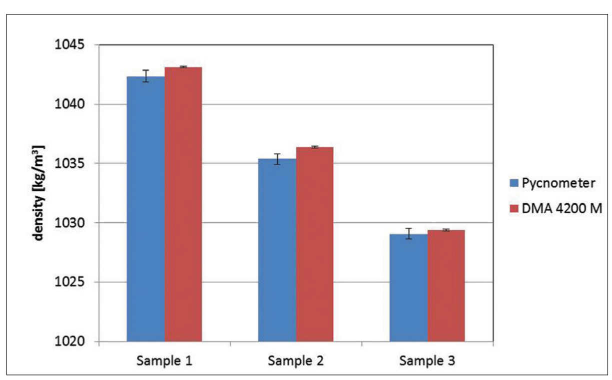


**Figure 3:** *Density results of pycnometer and DMA 4200 M measurements on 3 different bitumen samples (reporting temperature 60 °F)*

## DMA 4200 M makes itself useful

Over time, DMA 4200 M also proved useful for the quality analysis of many other samples commonly submitted to the I.M.U. laboratories such as crude oil, and for measurements on heavy intermediates from the production process.

Sometimes, especially in the winter months, the reduced number of samples for road paving leaves Ms. Schultze a little more time for other topics, such as testing new bitumen mixtures. "Sometimes we do not know a lot about these samples," says Natascha Schultze. "In this case I am very happy about the Hastelloy cell of DMA 4200 M as this material will not suffer any damage from unknown products."

With its expertise and modern equipment, I.M.U. is well prepared to cover the steadily growing number of samples and measuring parameters, and the expected increase of its customers' requirements on accuracy.

*The DMA 4200 M density meter in its assigned working environment*

## Infobox DMA 4200 M

DMA 4200 M is equipped with a measuring cell made of Hastelloy C276 and is based on the oscillating U-tube principle. The instrument is designed for measuring heavy samples, samples at pressures up to 500 bar, samples at temperatures up to 200 °C, and samples with viscosities up to 35,000 mPa.s. The required sample amount is 2 mL; the measuring temperature is achieved and maintained with the aid of a Peltier element.

DMA 4200 M is suitable for the quality control of heavy process samples and intermediate products, and for final product quality control of bituminous materials, LPG, heavy fuel oil (HFO), crude oil, and live crude oil. The fact that the instrument also copes with the quality control of bitumen, tar, and pitch in "heavy labs" as well as with R&D requests makes DMA 4200 M an asset for any petrochemical institution. Only small solvent volumes are needed for cleaning, which results in savings on cleaning time, a considerable reduction of cleaning agent, and less solvent waste to be disposed of.

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