

Density Measurement of Bitumen or Asphalt

Relevant for: Petroleum Industry

Bituminous materials are residues of the distillation processes of coal or petroleum products. They are highly viscous or even solid at room temperature. For monitoring their quality, density measurement is used on a routine basis on the heated samples.

DMA 4200 M fulfills these high demands.



Figure 1: The DMA 4200 M density meter and syringe heating accessory

1 Introduction

Bituminous materials include bitumen (or asphalt, as it is called in some regions), pitch, tar and related substances. These substances, as well as certain crude oils, are highly viscous or even solid at room temperature, but melt at temperatures of 100 °C to 150 °C.

For quality control the density of these products is measured on a routine basis. However, to measure the density of such highly viscous samples, the samples have to be brought to elevated temperatures. DMA 4200 M operates at high temperatures and keeps the sample liquid during the measurement.

2 DMA 4200 M plus syringe heating accessory: ideal partners for this application

For improved sample filling, a syringe heating accessory (SHA) is available as an optional part. The syringe heating accessory heats the sample inlet and outlet to measuring temperature and thus prevents the sample from clogging in the measuring cell.

In combination with the syringe heating accessory, DMA 4200 M is suitable for use in the temperature range from -10 °C to +200 °C (+14 °F to +392 °F) under atmospheric pressure conditions.

3 The measurement procedure

Important: As this application involves working with hazardous materials at high temperatures, only trained and experienced personnel should perform these measurements.

Obey all safety regulations and wear protective clothing (safety goggles, gloves) against heat!

- Install DMA 4200 M including the syringe heating accessory according to the instruction manual.
- Place an appropriate waste vessel under the sample outlet of DMA 4200 M.
- Connect the PTFE Luer extension to the tip of a heat-resistant syringe.
- Insert the sample and a heat-resistant syringe with the attached PTFE Luer extension into an oven that is held at measuring temperature until the sample has a sufficiently liquid consistency for injecting it into the measuring cell.
- Before filling the pre-heated sample into the measuring cell, make sure that DMA 4200 M has reached the measuring temperature which is shown on the instrument screen if the respective

option of temperature display is activated in the software.

- Remove the sample and the syringe with the attached PTFE Luer extension from the oven.
- Dip the tip of the Luer extension into the liquid bitumen or asphalt sample and slowly pull back the plunger of the syringe to fill the syringe with sample.
- Disconnect the PTFE Luer extension from the syringe tip.
- Insert the syringe into the syringe heating attachment and connect the syringe to the Luer-lock adapter of DMA 4200 M.
- Tighten the connection by turning the syringe clockwise.
- Slowly push the plunger of the syringe until the sample emerges from the sample outlet tube.
- Press start to initiate the measurement.

It takes approximately 10 to 12 minutes to complete one measurement with the pre-heated liquid bitumen or asphalt sample, including cleaning.

4 How to clean the measuring cell of DMA 4200 M

Important: Organic solvents represent a fire hazard! Make sure all safety regulations are obeyed! Only use solvents with a flash point considerably higher than the operating temperature of DMA 4200 M. Always operate in a well-functioning fume hood!

- Remove the rest of the sample from the measuring cell by pushing air through the measuring cell using an empty syringe.
- Fill solvent into a new syringe.
- Push the solvent through the measuring cell. Typically, 25 mL – 50 mL of solvent are required.
- Dry the measuring cell with the integrated air pump according to the instruction manual.

5 Adjustment media

An adjustment can be performed with air ($\rho = 0.00080 \text{ g/cm}^3$ at $150 \text{ }^\circ\text{C}$) and a certified reference material (CRM) or other pure liquids with a high boiling point.

6 What standards are available for high temperatures?

Anton Paar's portfolio comprises three CRMs for the high temperature range of $100 \text{ }^\circ\text{C}$ to $150 \text{ }^\circ\text{C}$ (in $10 \text{ }^\circ\text{C}$ increments) with an uncertainty of 0.01% (approx.

0.00008 g/cm^3). **Table 1** summarizes some information on the densities. The exact values depend on the standard and are mentioned on the respective standard package.

Table 1: Density standards for DMA 4200 M

Mat.no.	Name	Density at $100 \text{ }^\circ\text{C}$ [g/cm ³]	Density at $150 \text{ }^\circ\text{C}$ [g/cm ³]
161221	S200HT	0.823xx	0.793xx
161222	S600HT	0.830xx	0.800xx
161223	N100HT	0.818xx	0.788xx

Further reference data of other pure substances can be found on <https://webbook.nist.gov/chemistry/fluid/>.

ASTM D8188 describes the determination of density and relative density of asphalt, semi-solid bituminous materials, and soft-tar pitch by use of a digital density meter based on the U-Tube measuring principle.

ASTM D4311 / D4311M is used to convert the measured density at elevated temperatures to the reporting temperature (e.g. $15 \text{ }^\circ\text{C}$ / $60 \text{ }^\circ\text{F}$).

7 Alternative procedures

ASTM D70 describes the density determination of bituminous materials using the pycnometer method. This method is unpopular because it is hazardous, very time-consuming and difficult to perform in terms of sample preparation and cleanup.

EN 15326 describes the measurement of density and specific gravity of bituminous materials using the pycnometer method. The measuring procedure is similar to the one described in ASTM D70. The only difference is the determination in duplicate with a maximum deviation of 1 kg/m^3 . If this deviation is exceeded, the measurement has to be repeated.

Measurements with a pycnometer take 1.5 to 2 hours. Preparation of the sample includes heating and pouring molten asphalt or tar into the pycnometer. The operator is at risk of severe burns in the event of spillage. Cleanup is extremely difficult since the sample, which has solidified during the measurement, has to be reheated and cleaned out with large quantities of solvent.

8 Benefits of measuring with DMA 4200 M

- Fast and reliable results
- Small sample volumes, easy cleaning
- Densities of other liquids can be measured with a repeatability of $\pm 0.00001 \text{ g/cm}^3$ under optimum conditions

- Reduced health risk for the operator
- Long working life construction of the instrument, virtually maintenance-free
- Tables and/or equations can be programmed into the instrument for the calculation of density-related results (API, concentrations, etc.)
- Connection to a PC or printer through USB interfaces possible

9 Literature

ASTM D70-09e1 Standard Test Method for Density of Semi-Solid Bituminous Materials (Pycnometer Method)

EN 15326:2009 Bitumen and bituminous binders. Measurement of density and specific gravity. Capillary-stoppered pycnometer method

ASTM D8188 – 18 Standard Test Method for Determination of Density and Relative Density of Asphalt, Semi-Solid Bituminous Materials, and Soft-Tar Pitch by Use of a Digital Density Meter (U-Tube)

ASTM D4311 / D4311 M Standard Practice for Determining Asphalt Volume Correction to a Base Temperature



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