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#### Measurement Capabilities and Equipment at the Building Material Characterization Laboratory of Aalborg University (2024)

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# Measurement Capabilities and Equipment at the Building Material Characterization Laboratory of Aalborg University (2024)

**Hicham Johra** 



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## Measurement Capabilities and Equipment at the Building Material Characterization Laboratory of Aalborg University (2024)

by

Hicham Johra

November 2023

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#### 1. Foreword

The aim of this short communication report is to provide synthetic information concerning the measurement capabilities and equipment of the Building Material Characterization Laboratory of Aalborg University - Department of the Built Environment [1]. This laboratory uses state-of-the-art equipment to determine the thermophysical properties and the heat-air-moisture transport characteristics of materials, especially construction materials.

## **2. Physical Properties**

#### 2.1. Density, Mass and Volume

The bulk density of material at room temperature and atmospheric pressure can rapidly be measured by assessing the volume (dimensions) of a sample with a precision ruler, a caliper or a micrometer, and the mass of that sample with a precision scale. For a very accurate measurement of the density of small-size samples (few millimeters or centimeters), one can use a specific experimental setup mounted on a precision scale and based on the Archimedes' principle of buoyancy [2].

- Precision caliper "Mitutoyo": measurement up to 300 mm; resolution 0.01 mm.
- Precision **micrometer** "Mitutoyo": measurement up to 25 mm; resolution 0.001 mm.
- Precision scale "Sartorius Entris": measurement up to 2200 g; resolution 0.01 g.
- Precision scale "Sartorius Entris" with protection glass enclosure: measurement up to 220 g; resolution 0.0001 g.
- Precision scale "Sartorius Cubis": measurement up to 5200 g; resolution 0.1 g.
- "Sartorius" kit for small-size sample density measurement with Archimedes' principle of buoyancy: sample diameter up to 40 mm.



**Figure 1:** Measurement of dimensions and volume with a precision caliper (left); accurate measurement of mass with a precision scale [3] (center); accurate measurement of the density of small-size samples with the Archimedes' principle of buoyancy on a precision scale [2] (right).

## 3. Heat Transport and Dynamics Properties

#### **3.1. Thermal Conductivity**

The thermal conductivity of various types of material or entire construction elements can be measured with the appropriate equipment. The choice of the equipment mainly depends on the size of the sample to be tested: Small-size samples are tested with the Laser Flash Analysis Apparatus, medium-size samples are tested with the Guarded Hot Plate Apparatus [4], large full-scale construction elements are tested with the Guarded Hot Box, and very large full-scale construction elements are tested with the Big Guarded Hot Box. The smaller the sample the faster the measurement.

- Laser Flash Analysis Apparatus LFA 447 [5] (see Figure 2):
  - $\circ$  Thin sample of uniform thickness with smooth surfaces (no fibers or large pores structures).
  - o Powder, liquid, slurry, layered material, graded material, anisotropic material.
  - Thermal conductivity range 0.1 2000 W/m.K.
  - Temperature range 10 300 °C.
  - Round sample of diameter 6 mm\*; 8 mm\*; 10 mm\*, 12.7 mm\*, 25.4 mm\*. Thickness
    0.5 3 mm.
    \*can be diameter down to 1.2 mm less than prescribed diameter.
  - Scanning mode local measurement of large sample: round diameter 40 mm; square 50 x 50 mm. Resolution of 0.1 mm.
  - Measurement speed: ~ 3 min/measurement.



Figure 2: Laser Flash Analysis Apparatus LFA 447 (Netzsch Gerätebau GmbH) [5].

- **Guarded Hot Plate** Apparatus EP500 [4][6] (see *Figure 3*):
  - Cubical sample of uniform thickness with flat surfaces (compression of sample is controlled).
  - Thermal conductivity range 0.005 2 W/m.K.
  - $\circ$  Temperature range 10 40 °C.
  - $\circ$  Compression pressure range 0.05 2.5 kN/m² (5000 25000 Pa).
  - Sample dimension : 15 x 15 cm for conductive material, 50 x 50 cm for insulating material.
  - Sample thickness range 1 12 cm.

•

- Measurement uncertainty always below 1 % and mostly below 0.5 %.
- Measurement speed: ~ 5 hours/measurement.

	Einplatten-Wärmeleitfähigkeitsmessgerät X-Meter EP500
	459/57 N
-	

• Figure 3: The Guarded Hot Plate Apparatus EP500 from Lambda-Messtechnik GmbH Dresden [6].

- Guarded Hot Box (see Figure 4):
  - Large full-scale vertical construction element.
  - $\circ$  Square element with dimensions ranging from 1 x 1 m to 2 x 2 m.
  - Thickness of test element up to 70 cm.
  - Temperature range -20 80 °C.
  - Measurement speed: ~ 1 day/measurement.
- **Big Guarded Hot Box** [8] (see *Figure 5*):
  - Very large full-scale vertical construction element.
  - Square element with dimensions 4 x 4 m.
  - Thickness of test element up to 1 m.
  - Temperature range -20 80 °C.
  - Measurement speed: ~ 2 day/measurement.



*Figure 4:* The Guarded Hot Box setup at Aalborg University, Department of the Built Environment.



Figure 5: The Big Guarded Hot Box setup at Aalborg University, Department of the Built Environment [8].

#### **3.2. Specific Heat Capacity**

Very small samples can characterized with a Differential Scanning Calorimetry (DSC) Apparatus. The specific heat capacity of small samples can be measured with the Laser Flash Analysis Apparatus LFA 447 [5]. It is also possible to measure the local specific heat capacity of a larger sample with the scanning mode of the Laser Flash Analysis Apparatus LFA 447. The specific heat capacity of medium-size samples can be measured with a modified Guarded Hot Plate apparatus operating in dynamic mode.

- Laser Flash Analysis Apparatus LFA 447 [5] (see Figure 6):
  - $\circ$  Thin sample of uniform thickness with smooth surfaces (no fibers or large pores structures).
  - Powder, liquid, slurry, layered material, graded material, anisotropic material.
  - Temperature range 10 300 °C.
  - Round sample of diameter 6 mm\*; 8 mm\*; 10 mm\*, 12.7 mm\*, 25.4 mm\*. Thickness
    0.5 3 mm.
    \*can be diameter down to 1.2 mm less than prescribed diameter.
  - Scanning mode local measurement of large sample: round diameter 40 mm; square 50 x 50 mm. Resolution of 0.1 mm.
  - Measurement uncertainty: 5 %.
  - Measurement speed: ~ 3 min/measurement.



Figure 6: Laser Flash Analysis Apparatus LFA 447 (Netzsch Gerätebau GmbH) [5].

- **Guarded Hot Plate** Apparatus EP500 modified dynamic version [4][6] (see *Figure 7*):
  - Cubical sample of uniform thickness with flat surfaces (compression of sample is controlled).
  - $\circ$  Temperature range 10 40 °C.
  - $\circ$  Compression pressure range 0.05 2.5 kN/m<sup>2</sup> (5000 25000 Pa).
  - Sample dimension : 15 x 15 cm for conductive material, 50 x 50 cm for insulating material.
  - $\circ$  Sample thickness range 1 12 cm.



Figure 7: The Guarded Hot Plate Apparatus EP500 from Lambda-Messtechnik GmbH Dresden [6].

- **Differential Scanning Calorimetry** (DSC) Apparatus Netzsch Jupiter STA 449 F5 and Jupiter STA 449 F3 (see *Figure 8*):
  - Temperature range 100 900 °C (F5); -150 600 °C (F3 with liquid nitrogen).
  - Sample dimension: a few grams.
  - Sample conditioning: Compressed air or nitrogen gas (F5); helium or nitrogen gas (F3); sample placed in aluminum crucible.
  - Measurement uncertainty: 2 %.
  - Measurement speed: 6h/sample (3 scans: empty crucible, standard sapphire, test sample; with autosampler).



Figure 8: Differential Scanning Calorimetry Apparatus Jupiter STA 449 F3 (Netzsch Gerätebau GmbH) [5].

#### **3.3. Thermal Diffusivity**

The thermal diffusivity of small samples can be measured with the Laser Flash Analysis Apparatus LFA 447 [5][7]. It is also possible to measure the local specific heat capacity of a larger sample with the scanning mode.

- Laser Flash Analysis Apparatus LFA 447 [6][8] (see Figure 9):
  - Thin sample of uniform thickness with smooth surfaces (no fibers or large pores structures).
  - Powder, liquid, slurry, layered material, graded material, anisotropic material.
  - $\circ$  Thermal diffusivity range 0.01 1000 mm<sup>2</sup>/sec.
  - Temperature range 10 300 °C.
  - Round sample of diameter 6 mm\*; 8 mm\*; 10 mm\*, 12.7 mm\*, 25.4 mm\*. Thickness
    0.5 3 mm.
    \*can be diameter down to 1.2 mm less than prescribed diameter.
  - Scanning mode local measurement of large sample: round diameter 40 mm; square 50 x 50 mm. Resolution of 0.1 mm.
  - Measurement uncertainty: 3 %.
  - Measurement speed: ~ 3 min/measurement.



Figure 9: Laser Flash Analysis Apparatus LFA 447 (Netzsch Gerätebau GmbH) [6].

#### 4. Air transport properties

#### 4.1. Effective Air Permeability

The effective air permeability of medium-size samples of porous materials can be measured at room temperature with an air permeameter [10].

- Air permeameter [10] (see Figure 10):
  - Medium-size sample of porous materials uniform thickness.
  - Cubical samples: 50 x 50 cm (squared section); thickness 1 20 cm.
  - Room temperature and relative humidity (possible to condition sample in specific temperature and relative humidity environment inside climatic room).
  - o Measurement uncertainty: 2% 20% (3σ confidence interval).
  - Measurement speed: 1 hour/test sample.

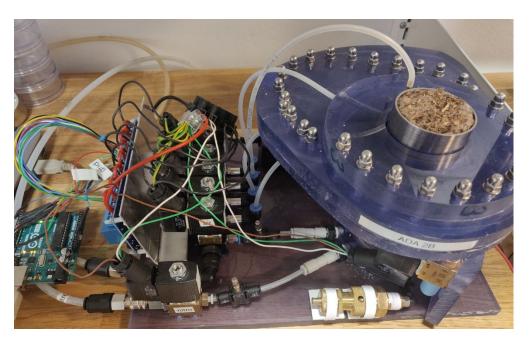


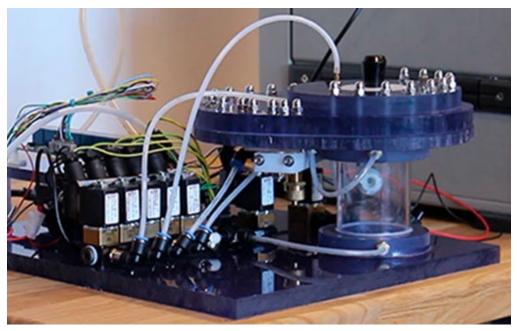
Figure 10: Air permeameter setup [10].

#### 4.2. Gas Diffusion Coefficient

The gas diffusion coefficient characterizes how permeable to gas is a porous material. It can be measured with the Oxygen Diffusion Apparatus ODA 20, an instrument that has developed at Aalborg University [14].

- **ODA 20** (see *Figure 11*):
  - Small-size cylindrical test sample: diameter 53 mm, length 50 mm.
  - Measurement speed: ~ 1-3 hour/measurement (10 samples at a time in parallel setups).
  - Room temperature and room relative humidity (possibility to control it with climatic room).





*Figure 11:* The oxygen diffusion apparatus ODA 20 for the measurement of the gas diffusion coefficient in porous materials [14].

## **5. Moisture Transport and Dynamics Properties**

#### 5.1. Hygrothermal Properties: Water Vapor Sorption and Desorption Capacity

The moisture transfer, retention and release capacity of materials can be determined by measuring water vapor sorption and desorption isotherms. This can be done by the AquaLab VSA [13] with both the static and the dynamic isotherms methods: Dynamic Vapour Sorption (DVS) and Dynamic Dew Point Isotherm (DDI). The VSA Apparatus is much faster and simpler than other conventional methods.

- AquaLab VSA apparatus [13] (see Figure 12):
  - Small-size test sample: mass range 500 5000 mg.
  - Sample holder volume 10 cm<sup>3</sup>.
  - Dynamic Vapour Sorption (DVS) and Dynamic Dew Point Isotherm (DDI).
  - Relative humidity range 3 95 %.
  - $\circ$  Temperature range 15 60 °C.
  - Measurement accuracy of relative humidity 0.5 %.
  - Temperature stability ± 0.1 °C.
  - Mass resolution 0.5 mg.
  - Measurement speed: ~ 2 days/full isotherm.



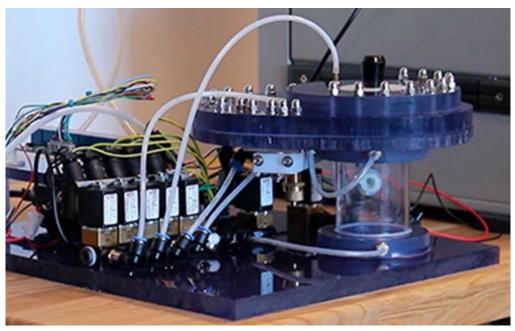
Figure 12: AquaLab VSA (METER Group) [13].

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- **ODA 20** (see *Figure 13*):
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  - Room temperature and room relative humidity (possibility to control it with climatic room).





*Figure 13:* The oxygen diffusion apparatus ODA 20 for the measurement of the gas diffusion coefficient in porous materials [14].

#### 5.3. Water Vapor Permeability

The water vapor permeability characterizes how permeable to water vapor is a porous material. It can be measured with the Wet/Dry Cup method with the help of a climatic chamber for temperature and relative humidity control. Dedicated 3D-printed sample holders have been designed at Aalborg University to perform the Wet/Dry Cup method accurately.

- Wet/Dry Cup method [according to EN ISO 12572 with silica gel as drying agent](see Figure 14):
  - Small-size cylindrical test sample: diameter 49 mm, length 51 mm (sides sealed with rubber sealant).
  - Measurement speed: ~ 2-10 weeks/measurement.
  - Temperature range 10 °C 95 °C (with climatic chamber VCL 7010 Vötsch Industrietechnik [11]).
  - Relative humidity range 10 98 % (with climatic chamber VCL 7010 Vötsch Industrietechnik [11]).



*Figure 14:* 3D-printed sample holder with bio-based material inside it for Wet/Dry Cup method (left); several Wet/Dry Cup method tests running in the climatic chamber VCL 7010 – Vötsch Industrietechnik [11] (right).

#### **5.3. Humidity Content**

The humidity content of a moist material is measured by continuously monitoring the mass loss of the test sample (with a precision scale) when it is drying in an oven (see *Figure 15*).

- Oven temperature range room temperature 300 °C.
- Interior volume 115 L.
- Measurement method speed: ~ 1 week.



Figure 15: Drying oven FP 115 (BINDER GmbH) [12].

#### References

- [1] Building Material Characterization Laboratory of Aalborg University, Department of the Built Environment, Aalborg, Denmark.
  - https://www.en.build.aau.dk/lab/building-material-characterization
- [2] Mettler Toledo, Laboratory weighing applications, Density measurement. https://www.mt.com/ch/en/home/applications/Laboratory\_weighing/density-measurement.html
- [3] Sartorius Lab Instruments GmbH & Co, Laboratory weight balances. https://www.sartorius.com/en/products/weighing/laboratory-balances
- [4] Hicham Johra. Description of the Guarded Hot Plate Method for thermal conductivity measurement with the EP500. DCE Lecture Notes No. 75. Department of Civil Engineering, Aalborg University, 2019. <u>https://vbn.aau.dk/ws/portalfiles/portal/317020205/Description\_of\_the\_Guarded\_Hot\_Plate\_Method\_for\_Thermal\_Conductivity\_Measurement\_with\_the\_EP500.pdf</u>
- [5] Netzsch Gerätebau GmbH. Product brochure STA 449 F5 Jupiter: <u>https://analyzing-testing.netzsch.com/en/products/simultaneous-thermogravimetry-differential-scanning-calorimetry-sta-tg-dsc/sta-449-f5-jupiter</u>
- [6] Netzsch Gerätebau GmbH. Operating Instructions Nano-Flash-Apparatus LFA 447, 2001.
- [7] http://www.lambda-messtechnik.de/en.html
- [8] Hicham Johra. Description of the laser flash analysis method for thermal diffusivity measurement with the LFA 447. DCE Lecture Notes No. 73. Department of Civil Engineering, Aalborg University, 2019. <u>https://vbn.aau.dk/ws/portalfiles/portal/312969074/Description\_of\_the\_Laser\_Flash\_Analysis\_Metho\_d\_for\_Thermal\_Diffusivity\_Measurement\_with\_the\_LFA\_447.pdf</u>
- [9] Martin Veit, Hicham Johra. Experimental Investigations of a Full-Scale Wall Element in a Large Guarded Hot Box Setup: Methodology Description. Technical Reports No. 304. Department of the Built Environment, Aalborg University, 2022. <u>https://doi.org/10.54337/aau488363266</u>
- [10]Hicham Johra. Air permeameter for porous building materials: Aalborg University prototype 2023. Lecture Notes No. 84. Department of the Built Environment, Aalborg University, 2023. <u>https://doi.org/10.54337/aau545266824</u>.
- [11]Vötsch 2009 VTL and VCL for Temperature and Climatic Tests URL. https://www.citrotek.dk/images/2009 laborhelfer E 06.pdf.
- [12]BINDER GmbH, Drying oven chamber FP 115. https://www.binder-world.com/en/products/drying-and-heating-chambers/series-fp-classicline/fp-115
- [13]Vapor Sorption Analyzer AquaLab. https://www.metergroup.com/food/products/vsa/
- [14]K.M. Frandsen, Y.I. Antonov, H. Johra, P. Møldrup, R.L. Jensen (2021). Experimental investigation of water vapor diffusivity in bio-based building materials by a novel measurement method. Proceedings of the Resilient Materials 4 Life (RM4L) 2020 International Conference. Cambridge, United Kingdom, 20-23 September 2021.

https://orca.cardiff.ac.uk/id/eprint/145287/1/RM4L2020%20Conference%20Proceedings%20-%20Online%20Version.pdf.

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