TK04 Application Note

Testing fragments and powder

Principle

The thermal conductivity of loose materials like fragments or powder is composed of the thermal conductivities of the solid constituent and of the pore filling (air). As the thermal conductivity of air is extremely low, the resulting thermal conductivity value depends largely on the ratio of solid material to pore volume. The pore volume in loose materials is not well defined, but may change significantly when shaking the sample, or each time a new sample of the same material is filled into the container.

Hence the thermal conductivity of such samples cannot be tested directly. Measuring results could neither be reproduced nor would they permit conclusions about the thermal conductivity of the solid material.

In order to avoid these problems, the air between the solid grains is replaced by a fluid with known thermal conductivity. The fluid share in the sample is determined by weighing before and after adding the fluid. The so-called **matrix thermal conductivity** (i.e. the thermal conductivity of a non-porous solid consisting of the solid material share) can then be calculated from the measured thermal conductivity of the mixture, the shares of fluid and solid material in the sample and the known thermal conductivity of the fluid. The method is called 2-phase test because the sample consists of 2 phases (fluid and solid).

$$TC_{mixture} = (TC_{fluid})^{v} * (TC_{powder})^{1-v}$$

$TC_{mixture}$	thermal conductivity of the mixture
TC_{fluid}	known thermal conductivity of the fluid
TC_{powder}	matrix thermal conductivity of the powder
v	volume share of fluid in the sample

Suitable fluids

2-phase tests require a fluid which does neither dissolve the solid sample material nor reacts chemically with it. The fluid's thermal conductivity must be known accurately. The method is valid if the thermal conductivity of the dry sample material is not larger than 15 times the thermal conductivity of the fluid. As the thermal conductivity of water at room temperature is approx. 0.6 $Wm^{-1}K^{-1}$ it can be used for water-insoluble sample materials up to 9 $Wm^{-1}K^{-1}$. The method subsequently described for water is the same for other fluids.

Recommended tools and resources

For 2-phase tests a cylindrical sample container is available as an option whose inner diameter exactly matches the size of the Standard HLQ probe. Additionally we recommend to use the lever press with pressure limiter for removing the excess fluid and to guarantee good contact between the probe and the sample mixture.

A Vernier caliper is recommended for length measurements. Additionally a scale with a measuring range of at least 3000g is required, accuracy ± 1g or better.

In the customer download section of our website we offer a free spreadsheet file for OpenOffice, LibreOffice or Microsoft Excel which guides you step by step through sample preparation, measuring and evaluation. As the file includes the density and the thermal conductivity values of water for the complete temperature range supported by TK04, results are calculated automatically when using water as a fluid.

Preparation of sample material

The sample may not contain coarse or sharp-edged parts larger than approx. 1 mm, larger grains must be reduced in size. The solid material must be completely dry before mixing it with the fluid. Residual moisture will falsify the result.

We recommend to bring both the solid material and the fluid to the laboratory the day before starting measurements so they can adapt to ambient temperature.

Determining the volume share of fluid in the sample

The volume share of water in the sample is calculated from the volume of water divided by the total sample volume.

The sample volume is calculated from the inner diameter of the sample container (88 mm) and the fill height. To determine the fill height, put the probe into the empty sample container and measure the distance between the upper side of the probe and the upper edge of the container (see step 01 below). After finishing the thermal conductivity test, measure this distance again, this time with the sample material and the probe on top (see step 09 below). The difference between the two distances is the fill height.

Determine the water weight by weighing the sample container before and after adding the water. Then divide the weight by the known density of water to calculate the corresponding water volume. The temperature-dependent density characteristics of water is included in the worksheet we offer for download (see above). The sample temperature at the time of measuring can either be displayed using the TK04 measuring program, or retrieved directly from the data file (*.dwl), see step 11 below.

Measuring the thermal conductivity of the mixture

Measure the thermal conductivity of the mixture using TK04. As convection may occur in wet samples, the heating power should be chosen as low as possible (Power Control approx. 2.0).

Thermal conductivity of water at test temperature

The thermal conductivity of water is temperature dependent. The worksheet we offer for download (see above) includes the thermal conductivity values of water for the complete TK04 temperature range. The correct value is chosen automatically when entering the measuring temperature into the designated input field.

Calculating the thermal conductivity of the solid phase

The thermal conductivity of the solid fraction is calculated from the thermal conductivities of the mixture and of the fluid phase and from the volume share of fluid using the equation given above. The result is the thermal conductivity which a homogeneous, non-porous solid sample piece consisting of the solid material share would have (so-called matrix thermal conductivity). When using the TeKa worksheet (see above) the result is calculated automatically.

Step by step instructions

A best practice as used in the TeKa lab is described below. All measuring and weighing results are entered into the **Form** worksheet of the spreadsheet file provided by Teka which automatically does all calculations. For documentation purposes you can print the **Protocol** worksheet. When using another fluid than water the input fields for density and thermal conductivity of the fluid (both at measuring temperature) must be filled in manually.

01 - Determining the inner height of the empty sample container
Put the probe into the empty sample container and measure the distance from the upper side of the probe to the upper edge of the container with a Vernier caliper.

Enter the result into the input field labeled **Height (empty) - H1**.

02 - Filling in the solid phase
Fill the dry sample material into the sample container. The total sample volume and weight should be as large as possible in order to keep the relative errors caused by volume determination and weighing small, which means that you should fill the container up to 1 or 2 cm below the upper edge.

03 - Determining the dry weight
Weigh the filled sample container together with the probe (do not place the probe on top of the sample yet).

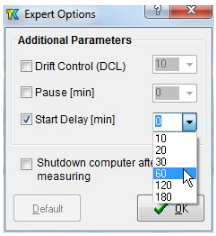
Enter the result into the input field
Weight (dry sample + probe + container) - W_{dry}.

04 - Filling in the fluid phase
Add water in small amounts and stir with a spatula until the sample is completely saturated. We recommend to place a container with water and the solid sample material in the laboratory the day before measuring starts in order to let the sample components adapt to measuring temperature.

05 - Removing air bubbles
Let the sample settle for a while and shake the container if necessary to remove all remaining air from the sample.

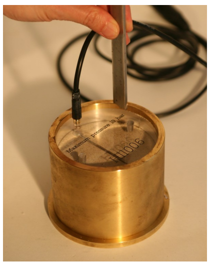
06 - Placing the probe on the sample
Place the probe on top of the sample and push it down cautiously until all air has escaped through the lateral notches in the probe body (slightly tilt the container if necessary). Trapped air will affect the contact between sample and probe.

07 - Removing excess fluid
Use the lever press to press the probe to the sample material. Move the lever slowly and do not apply more pressure than necessary to remove excess fluid and establish good contact. The needle embedded in the underside of the probe body must be in contact with the sample material on its whole length.



08 - Measuring thermal conductivity
As sample preparation disturbs thermal equilibrium, usually a waiting time is required before starting measurements. Use the *Start Delay* parameter of the TK04 measuring program to configure a waiting time (see chap. 3.2.4 of the TK04 Manual). We recommend to use a *Start Delay* of 60 minutes.

Enter the result of the thermal conductivity test into the input field labeled **Result of TC measurement - $\mathbf{TC_{mixture}}$**.



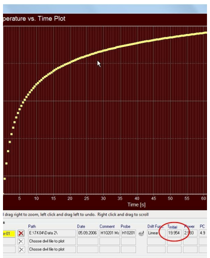
09 - Determining sample height
After finishing the thermal conductivity measurements, open the press, remove the sample container and measure the distance from the upper side of the probe to the upper edge of he container. If the probe protrudes from the container, add a leading minus sign to the distance value.

Enter the value into the input field labeled **Height (with sample) - H2**.

10 - Determining the wet weight

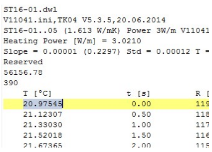
Cautiously remove any remaining fluid from the upper side of the probe. Then weigh the container including the probe.

Enter the result into the input field
Weight (moist sample + probe + container) - $W_{saturated}$.



11 - Determing sample temperature

The sample temperature during the measurements is required for determining the temperature dependent values of density and thermal conductivity of water. You can use the Heating Curves plot of the TK04 measuring software (see chap. 3.5 of the TK04 Manual) to display the start temperature $T_{initial}$ of a finished measurement. In measuring series the sample temperature may vary slightly between single measurements, we recommend to use the undisturbed start temperature at the beginning of the first measurement.



Or use a file viewer or editor to open the dwl file of the first measurement and note down or copy the temperature T at time t=0.00 s. The dwl files are located in the data folder you have chosen when configuring the measurements.

Enter the temperature value into the input field labeled
Sample temperature (at start time) - $T_{initial}$.

12 - Calculating the result

If the measuring and weighing results from all previous steps are correctly entered into the worksheet, the thermal conductivity of the solid phase is automatically calculated using the equation given above.

Accuracy

The accuracy of a 2-phase test is determined by the accuracy of the thermal conductivity measurement, the accuracy of volume determination and by the weighing error. Hence the result of a 2-phase test is always less accurate than that of a direct thermal conductivity measurement.

The influence of weighing and of length measurement errors on the total error is nonlinear. Their influence depends on the precise sample and fluid amounts and on the thermal conductivities of solid and fluid phases. In order to estimate the error, vary the values for sample height or weight in a completely filled-in worksheet for 2-phase tests and check how much thermal conductivity results are changed.

The total error is composed of the relative errors of volume and weight determination (not the absolute ones). Hence accuracy can be improved by choosing sample volume and weight as large as possible, i.e. by filling the sample container as high as possible.