Using the KD2 Pro to measure thermal properties of fluids

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Overview

The KD2 Pro uses the transient heated needle to measure thermal properties of solid and fluid media. With this technique, a heat pulse is applied to a needle, and the temperature response with time is monitored either at the heated needle or at an adjacent needle both during and after the heat pulse. The nature of the temperature response is a result of the thermal properties of the material. When measuring thermal properties we wish to measure only the heat transfer resulting from conduction. In low viscosity fluids, heat transfer by convection can be much greater than heat transfer by conduction. Hence, accurate measurement of thermal properties of fluids requires that convective heat exchange be negligible.

Convective heat exchange in fluids can be broken down into two categories: forced and free convection. Forced convection occurs when the fluid is agitated or mixed by mechanical forces. Free convection may occur when a body of higher or lower temperature is inserted into a fluid. The temperature difference between the body and fluid create density gradients in the fluid, and these density gradients cause the fluid to mix. Certain steps can be taken to minimize both forced and free convective heat exchange.

Preventing forced convection

To eliminate forced convection, the fluid sample and the sensor must be absolutely still during the measurement. Even minute vibrations in the sample are often enough to compromise the thermal properties measurement. Some common sources of vibrations found in the laboratory that must be avoided include:

- Vibration from Heating, Ventilating, and Air Conditioning (HVAC) systems
- Vibration from computer fans that are near the measurement apparatus
- Vibration from people moving around the lab
- Vibration from other laboratory equipment

If sources of vibration cannot be eliminated in the laboratory, it may be necessary to make the measurement on an optical table or other vibration isolation device to prevent errors from forced convection. Another common strategy is to configure the KD2 Pro in auto mode and make measurements overnight after turning off the HVAC system and any other lab equipment that might cause vibrations.

Preventing free convection

Steps should also be taken to eliminate free convection. The heat transfer by free convection is described by

$$g_H = \frac{0.54\ \hat{\rho}\ D_H \left(\frac{g\ d^3 \Delta T}{T\ \nu\ D_H} \right)^{1/4}}{d}$$

where g_H is the heat conductance (mol m^{-2} s^{-1}),   is the molar density of the fluid (mol/m^3), DH is the thermal diffusivity of the fluid (m^2/s), g is gravitational acceleration (m/s^2), d is the characteristic dimension of the object placed in the fluid (m), ΔT is the temperature difference between the bulk fluid and the object inserted into it, T is temperature (K), and ν is the kinematic viscosity of the fluid (m^2/s).

Examining eq. 1, we see that the heat conductance is inversely related to the characteristic dimension (d) of the probe inserted into the fluid. The characteristic dimension of an object is a function of the shape of the object and the direction of fluid flow over that object. For this discussion, the needle probe can be accurately described as a cylinder. For a cylinder with its axis parallel to the fluid flow, the characteristic dimension is its length. For a cylinder with its axis perpendicular to the fluid flow, the characteristic dimension is its diameter. When considering a heated probe inserted into a cooler fluid, the fluid flow near the probe from free convection will be upward, as the warmer, less dense material near the probe is forced upwards by forces of gravity working on the surrounding, denser material. So, if a heated needle is inserted into a fluid horizontally, the fluid flow around the needle will be perpendicular to the axis of the needle, and the characteristic dimension will then be the diameter of the needle (1.27 mm for the KS-1 or 1.8 mm for the TR-1). If the needle is inserted into a fluid vertically, the fluid flow will be parallel to the axis of the needle, and the characteristic dimension is the length of the needle (60 mm for the KS-1 or 100 mm for the TR-1). Keeping in mind that the heat conductance by free convection is inversely proportional to the characteristic dimension, inserting the needle into the fluid vertically will greatly reduce convective heat transfer, and result in more accurate measurement of thermal conductivity.

Again from eq. 1, we discover that the heat conductance by free convection is proportional to the temperature difference between the fluid and the object inserted into it. Hence, free convection can be decreased by limiting the heating of the needle. The KD2 Pro KS-1 (60 mm single needle) sensor is specifically designed for use in fluids, and provides a very small heat pulse that can measure thermal conductivity of most fluids without causing free convection. The KD2 Pro TR-1 (100 mm single needle) and SH-1 (dual needle) sensors create a substantial heat pulse, and should never be used in low viscosity fluids (see Table 1). When using the KS-1 sensor in low viscosity fluids (such as water or aqueous solutions), the read time should be set to the minimum allowed 1 minute duration to further minimize the temperature difference between the fluid and the needle.

Probe type	Viscous fluids (glycerol, castor oil)	Low viscosity fluids (water, aqueous solutions)	Stabilized water
KS-1	Best	Best[1]	Best
TR-1	Ok	NO	Ok
SH-1	NO	NO	Ok

[1] In low viscosity fluids, the read time of the KS-1 should be set to the minimum allowed 1 minute and the needle should be oriented vertically.

Table 1. Suitability of KD2 Pro probes for various fluids

Further examination of eq. 1 shows us that the heat conductance is inversely proportional to the kinematic viscosity of the fluid. So, thermal conductivity measurements in more viscous (thicker) fluids are less affected by free convection. More viscous fluids such as castor oil ($\nu = 1.0 \times 10^{-3}$ m^2/s @ 20C) and glycerol ($\nu = 7.4 \times 10^{-4}$ m^2/s @ 20C) are unaffected by free convection during the thermal conductivity measurement, and are easy to measure with the KS-1 needle without further precautions. However, in low viscosity fluids such as water ($\nu = 8.9 \times 10^{-7}$ m^2/s @ 20C) free convection is difficult to prevent. With careful experimental technique, it is possible to measure the thermal conductivity of water and aqueous solutions with the KS-1 sensor, but fluids with viscosities lower than that of water cannot be measured accurately. The viscosities of fluids are inversely proportional to the fluid temperature. With careful experimental technique, researchers have been able to measure the thermal conductivity of aqueous solutions up to about 50 C with the KS-1 sensor.

Low viscosity fluids can often be stabilized with thickeners to increase viscosity and prevent convection. In fact, it is commonplace in the scientific literature for researchers to calibrate thermal properties probes in water stabilized with agar (e.g. Ochsner et. al., 2003, Campbell et. al., 1991). Even the KD2 Pro SH-1 and TR-1 sensors can accurately measure thermal properties of stabilized water. Pure agar powder can be obtained from a variety of sources. Two suppliers that generally have agar powder in stock are:

VWR Scientific	Alfa Aesar
Agar powder USP, 100g	Agar powder
CAS: 9002-18-0	CAS: 9002-18-0
Item # IC10026280	Item #A10752
$20 /100g	$15 /100g

Agar powder should be brought to a boil in the aqueous solution to be stabilized, and stirred until all of the powder is dissolved. In general, a mixing ratio of 5 g agar powder per 1 L of solution is sufficient to adequately stabilize the solution. However, the necessary concentration is somewhat dependent on the chemical properties of the solution. After the agar – water mixture returns to room temperature it should have the consistency of Jell-O™. If the mixture is not adequately stabilized, add additional agar powder and again bring to a boil and stir until all agar powder has dissolved.

Liquid Sample Temperature Control

Often it is desirable to control the temperature of the liquid sample above or below ambient temperature during thermal properties measurement. It is important that the act of heating or cooling the sample doesn't cause forced or free convection as mentioned above. There are several things that should be avoided when measuring thermal properties of heated or cooled samples.

- Do not heat the sample from the bottom (e.g. on a hot plate). The temperature gradient from the heating will cause free convection.
- Do not make measurements in a conventional refrigerator or freezer. Conventional cooling devices have very large cyclical temperature cycles which can cause excessive sample temperature drift and poor measurements. Vibrations from the compressor will also cause forced convection in the sample.
- Do not measure the thermal properties of the sample while it is in a circulating water bath. The vibrations from the water bath pump and from the circulating water will cause forced convection in the sample.

According to several researchers who use the KD2 Pro with liquid samples, the best method for controlling temperature of liquid samples is as follows.

1. Heat or cool the sample (with sensor inserted) in a water bath.
2. Once the sample temperature has equilibrated to the desired water bath temperature, turn the water bath off.
3. Allow enough time for the water bath to become absolutely still, then make the measurement.

Summary

- Only measure the thermal properties of fluids with the KS-1 (small single needle) sensor.
- Ensure that there is no shaking, mixing, or vibration of the fluid during or immediately before the measurement.
- Vertical insertion of the probe into the fluid will minimize errors from free convection.
- Configure the KD2 Pro to the minimum allowed read time of 1 minute.
- Less viscous fluids are more subject to errors from free convection so stabilized solutions are sometimes used.
- The KD2 Pro can measure thermal conductivity of water and aqueous solutions up to about 50 C, and cannot measure thermal conductivity of fluids that are less viscous than water.

References

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