

Modified Transient Plane Source (MTPS): Theory of Operation

The C-Therm TCi Thermal Conductivity Analyzer is based on the Modified Transient Plane Source (MTPS) technique. It employs a one-sided, interfacial heat sensor that applies a momentary constant heat source to the sample. Thermal conductivity and thermal effusivity are measured directly, providing a detailed overview of the thermal characteristics of the sample. The following paper details the further principles to the operation, including a detailed discussion of the calibration of the measurement.

NOTE: Calibration is typically provided by C-Therm Technologies Ltd. Users are unencumbered by the responsibility to calibrate the instrument as the sensors are provided in a “Plug & Play” format with the necessary calibration data stored already in a sensor ID chip embedded within the sensor. The following calibration details are provided for information purposes only and are NOT provided as instructions.

Sensor temperature calibration

This section explains the relationship between the sensor’s change in temperature, ΔT , its resistance ΔR and its voltage drop ΔV , and how to calculate ΔT from TCR (temperature coefficient of resistivity) calibration parameters.

The TCR calibration is calibration of sensor resistance versus temperature, and is given in this equation (assuming perfect linearity):

$$R(T) = R_0 + A \cdot T \quad (1)$$

Where:

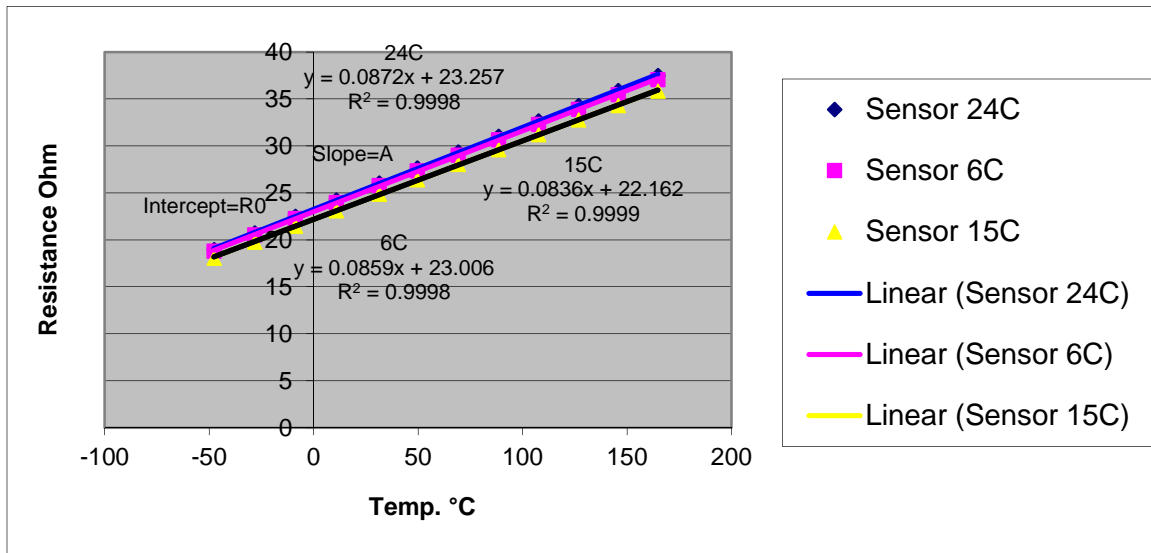
$R(T)$ = resistance of sensor at a given temperature (Ohm)

R_0 = resistance of sensor at 0°C (Ohm)

T = temperature (°C)

A = slope (Ohm/°C)

Example of TCR calibration:



Range of A ~0.20 – 0.30 Ohm/°C depending on sensor design
Range of R_0 ~60 – 70 Ohm

The slope A is equal to:

$$A = R_0 \cdot TCR \quad (2)$$

Where: TCR = Temperature Coefficient of Resistivity, assumed to be constant over the measured temperature range.

Notes:

1. The slope depends on sensor resistance in general, and on R_0 in particular. Therefore, even if 2 sensors have the same TCR , their temperature calibration lines will still have different slopes if their resistance at a given temperature is not the same.
2. The higher slope, the more sensitive the sensor is. In other words, higher TCR and higher sensor resistance will provide higher sensitivity.

For platinum used in the sensor, the TCR is approximately $0.0037 \text{ }^\circ\text{C}^{-1}$, or 0.37% for each °C.

To calculate the surface temperature of the sensor from (1) we use:

$$T = \frac{R(T) - R_0}{A} \quad (3)$$

The resistance may be measured directly by the electronics (using very low current and short time to avoid sensor heating), or calculated from the initial voltage, V_0 , or from the applied power P (if applied power is the same for all sensors).

$$R = \frac{V_0}{I} \quad (4)$$

$$R = \frac{V_0^2}{P} \quad (5)$$

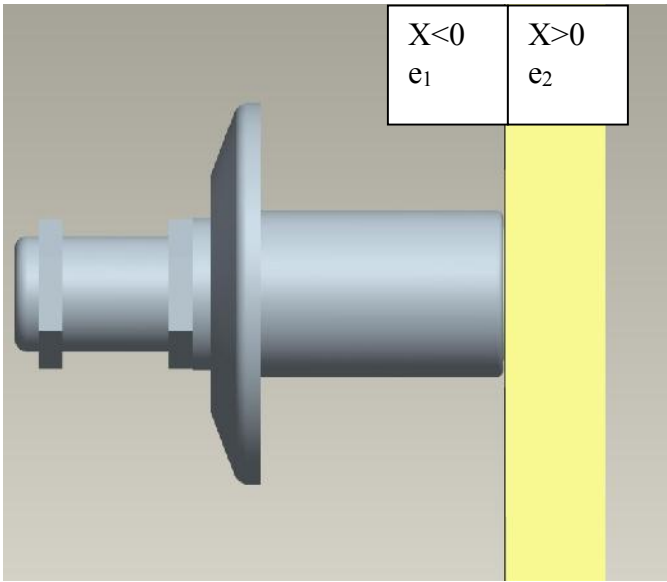
Where R is the measured sensor resistance at the said temperature, I is the current and P is the power.

Basic multi-point effusivity calibration

The heat equation with a constant supply of heat per sec per volume G' is given below:

$$\dots c_p \frac{\partial T}{\partial t} = \} \frac{\partial^2 T}{\partial x^2} + G' \quad (6)$$

Assume two semi-infinite media in contact with heat generated at the interface at a constant rate per unit area per unit time. Further assume that one medium is represented by the effusivity sensor, and the other medium is the tested material, and both are at the same temperature and in equilibrium after contact between them has been established. The solution of equation (6) follows these expressions:



$$\Delta T_1(x,t) = \frac{2G\sqrt{t}}{e_1 + e_2} \operatorname{ierfc} \frac{|x|}{2\sqrt{a_1 \cdot t}} \quad \text{for } x < 0, t > 0 \quad (7)$$

$$\Delta T_2(x,t) = \frac{2G\sqrt{t}}{e_1 + e_2} \operatorname{ierfc} \frac{x}{2\sqrt{a_2 \cdot t}} \quad \text{for } x \geq 0, t > 0 \quad (8)$$

Where: T = change in sensor surface temperature ($^{\circ}\text{C}$)
 G = power flux supplied to sensor (W/m^2)
 t = time measured from start of process (sec)

e_1 = equivalent effusivity of sensor $(\sqrt{\lambda_1 \cdot c_{p1} \cdot \dots_1}, \frac{\text{W}\sqrt{\text{s}}}{\text{m}^2\text{K}})$

e_2 = effusivity of measured material $(\sqrt{\lambda_2 \cdot c_{p2} \cdot \dots_2}, \frac{\text{W}\sqrt{\text{s}}}{\text{m}^2\text{K}})$

a_1 = equivalent diffusivity of sensor, m^2/s

a_2 = diffusivity of measured material, m^2/s

λ_1 = equivalent thermal conductivity of sensor, W/mK

λ_2 = thermal conductivity of measured material, W/mK

ρ_1 = equivalent density of sensor, Kg/m^3

ρ_2 = density of measured material, Kg/m^3

c_{p1} = equivalent specific heat capacity of sensor, J/kgK

c_{p2} = specific heat capacity of measured material, J/kgK

Assumption: Both sensor and measured material are in equilibrium and at the same temperature when the measurement starts.

If no contact resistance exists at the interface, $T_1(x = 0, t) = T_2(x = 0, t)$ at all points with $x = 0$. For $x = 0$ equations (7) and (8) are reduced to:

$$\Delta T(x = 0, t) = \frac{2G\sqrt{t}}{e_1 + e_2} \cdot 0.5642 = \frac{1.1284G\sqrt{t}}{e_1 + e_2} \quad (9)$$

In the previous section we saw the connection between sensor temperature and resistance. The resistance change of the sensor is:

$$\Delta R(t) = R(t) - R(t = 0) = A \cdot \Delta T(x = 0, t) \quad (10)$$

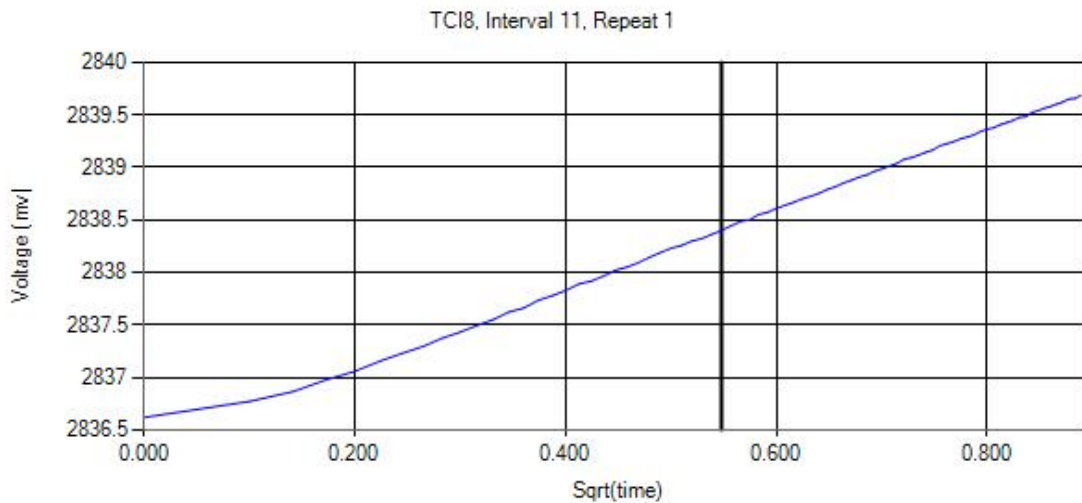
And the voltage change on the sensor is:

$$\Delta V(t) = I \cdot \Delta R(t) = I \cdot A \cdot \Delta T(x = 0, t) \quad (11)$$

Using equation (9) we can write:

$$\Delta V(t) = \frac{1.1284I \cdot A \cdot G\sqrt{t}}{e_1 + e_2} \quad (12)$$

Example of voltage versus \sqrt{t} measurement:



Equation (12) can be written (in the linear zone) as:

$$\Delta V = m\sqrt{t} \quad (13)$$

Where m is the slope,

$$m = \frac{1.1284I \cdot A \cdot G}{e_1 + e_2} \quad (V/\sqrt{s}) \quad (14)$$

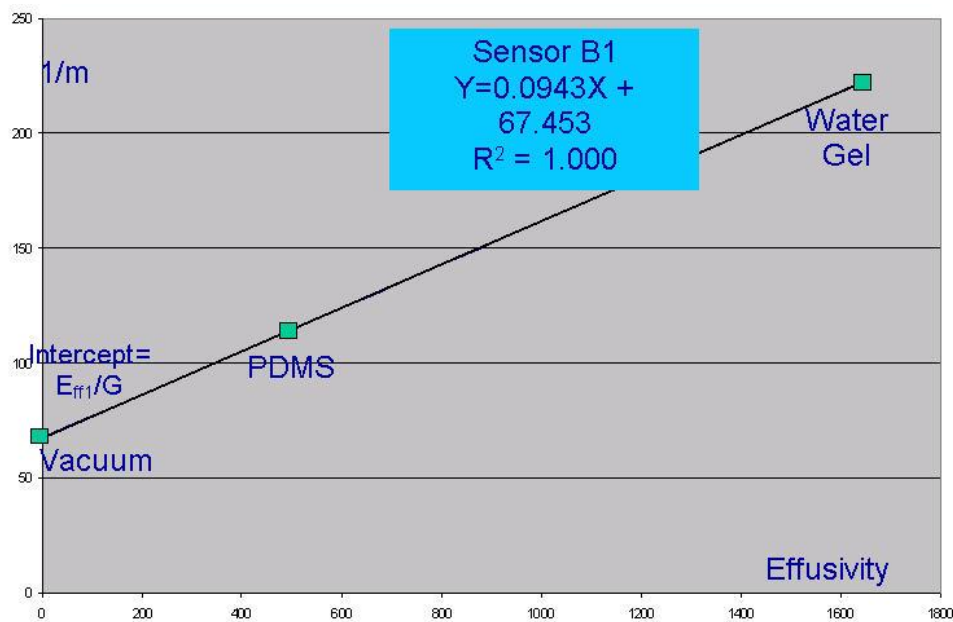
$$\frac{1}{m} = \frac{e_1 + e_2}{1.1284I \cdot A \cdot G} \quad (\sqrt{s}/V) \quad (15)$$

If e_2 is 0, i.e. sensor response is measured in vacuum, then:

$$\frac{1}{m}(\text{vacuum}) = \frac{e_1}{1.1284I \cdot A \cdot G} \quad (16)$$

The figure $(e_1/IA G)$ is a sensor/system figure of merit, and depends only on sensor characteristics and supplied power, and may be used for calibration.

Example of sensor effusivity calibration curve using vacuum and other materials is given below. The calibration line shows very good linearity.



The calibration line can be written as:

$$\frac{1}{m} = M \cdot e_2 + C \quad (17)$$

Where M is the slope of the effusivity calibration and is equal to:

$$M = \frac{1}{1.1284I \cdot A \cdot G} \quad (m^2 \cdot ^\circ C / W \cdot Amp \cdot \Omega) \quad (18)$$

And C is:

$$C = \frac{e_1}{1.1284I \cdot A \cdot G} \quad (\sqrt{s}/V) \quad (19)$$

C is the $1/m$ value for vacuum.

$$\frac{C}{M} = e_1 \quad (W \sqrt{s} / m^2 \cdot ^\circ C) \quad (20)$$

e_1 , sensor effusivity can be calculated from (20).

Material groups and power levels

In order to maintain a high accuracy and precision to the MTPS technique across a broad range of thermal conductivity and effusivity – the sensor's calibrations for both thermal properties are sub-divided into smaller ranges in bracketing the wider spectrum. Power and timing parameters are optimized for the group of materials. For example, a longer test time and lower power setting is optimal for testing insulation materials like foams in achieving a deeper depth of penetration to the measurement. Conversely, a shorter test time and higher power setting is optimal for higher-conductivity metals where the pulse travels very quickly through the material and a shorter test time provides for more flexibility in limiting the thickness requirements of the sample. Each group of materials has its own calibration curve.

Thermal conductivity (*k*) calibration

Calibration and measurements of *thermal conductivity* are based on same data acquired for effusivity. The same separation into material groups and power levels apply for *k* as well.

The algorithm to calibrate and calculate *k* is the *m**, which is outlined in US Patent 6,676,287 B1. It uses an iterative process to calibrate the sensor against measured materials with known thermal conductivity. The calibration process generates a value called *m**, such that:

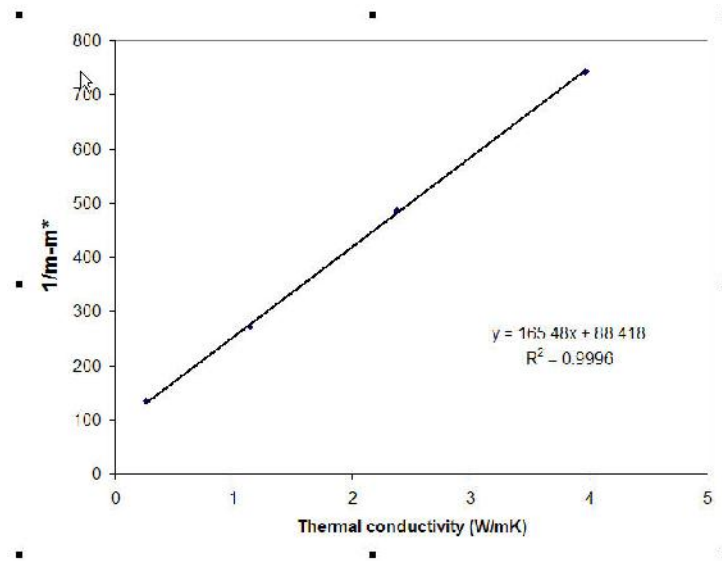
A typical *k* calibration has this form:

$$\frac{1}{m - m^*} = Slope \cdot k + Intercept \tag{24}$$

*m** is found by an iterative process during calibration. To calculate *k* use:

$$k = \frac{\frac{1}{m - m^*} - Intercept}{Slope} \tag{25}$$

Each group of materials has a different *k* calibration for room temperature. With automatic power correction and *C(T)* correction (see below), this calibration can be also used for different temperatures. The calibration curves for *k* are all linear. Example is given below.



Limitations

The method for measuring *thermal properties* requires different calibration curves for materials with different ranges of properties. Additionally, it is important to group liquids separately from solids. Measuring a liquid with a calibration made for solids, or vice versa, will generate an incorrect result.

In practice, the selection of appropriate calibration groups from those made available with the C-Therm TCi Thermal Conductivity Analyzer is intuitive and obvious with the help of the software which flags any reported values from the sensor outside of the calibration range for the material calibration group selected.

Error analysis

General

There are in general 2 types of errors (sometimes called uncertainty) – offset and variation. Offset errors affect the accuracy of the measurement while the variation affects the repeatability.

Errors in effusivity and conductivity measurements have three significant sources: quality of contact between sensor and tested material, errors from equipment and errors from calibration (offset only).

Contact

The quality of contact between the sensor surface and the tested material is critical to having accurate and repeatable measurements. Powders, liquids and creams naturally create a good contact with the sensor, but greater care must be taken with solids.

The impact of contact resistance becomes more significant in testing solids, the higher the thermal conductivity of the sample tested. In efforts to negate the contact resistance it is suggested to use a contact agent for solids greater than 0.12 W/mK. This approach to using a contact agent is consistent with other ASTM-methods for measuring thermal conductivity such as ASTM D5470.

Water is recommended in most cases in testing materials between 5°C and 70°C. It normally would create a uniform thin layer, and given its relative high conductivity, its contribution in thermal resistance will be small. The user must be careful to avoid any air bubbles, as those affect the heat transfer between the sensor and tested material.

Alternate contact agents suggested for porous materials that may absorb the water or for testing at higher temperatures include Wakefield Type 120 Thermal

Grease and glycol. Correction factors for all contact agents are provided for within the software automatically.

Note: the surface of the tested material must be smooth. The sample need not be polished, but a rough surface finish will add to the total error budget.

Equipment

Equipment errors may originate from variations in the current source due to changes in environment temperature, short term and long term drifts, change in sensor resistance (and hence supplied power) during the transient measurement and change in sensor resistance (and hence supplied power) due to initial sensor temperature. Additional errors may come from the voltage measurement circuitry.

To evaluate the error from the change in sensor resistance during a transient, assume a ~1 degree Celsius change in sensor temperature (~1.0 s transient) for the range of materials used with the system. The platinum wire of the sensor has a TCR of $0.0037\text{ }^{\circ}\text{C}^{-1}$. During the transient, the sensor resistance changes 0.37%, and the power supplied to the sensor changes by the same amount. However, since the calibration of the sensor is performed in exactly the same manner as the measurement of the tested material, this error is calibrated out for most practical cases, leaving a very small residual error of less than 0.1%.

As mentioned earlier, the power supplied to the sensor is automatically corrected at the beginning of each sampling, to the extent of the precision of the current source. Additionally, offset errors due to the effusivity of the sensor are corrected to a large extent, though at low and high temperatures there will be larger offset error than at room temperature, where calibration is done.

Calibration

The TCi calibration curves are based on various sets of materials, which were tested for conductivity and volumetric heat capacity by the optimal recommended ASTM methods for the thermal property range of material. The measured values of those materials are used for the TCi calibration curves. Since these values may have accuracy errors of a few percentage points, these errors are naturally transferred to the TCi sensor.

The overall accuracy of the TCi is estimated to be better than 5%. The variation is typically under 1% (%RSD calculated from at least 10 consecutive samples).