C-THERM TECHNOLOGIES

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Operating Procedure for the TCi™: Small Volume Test Kit for Thermal Conductivity Testing

Document No: TH060073-OP

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1 Scope

This document provides instructions on testing minimal volumes of powder and liquids using the TCi Small Volume Test Kit (SVTK).

2 Background

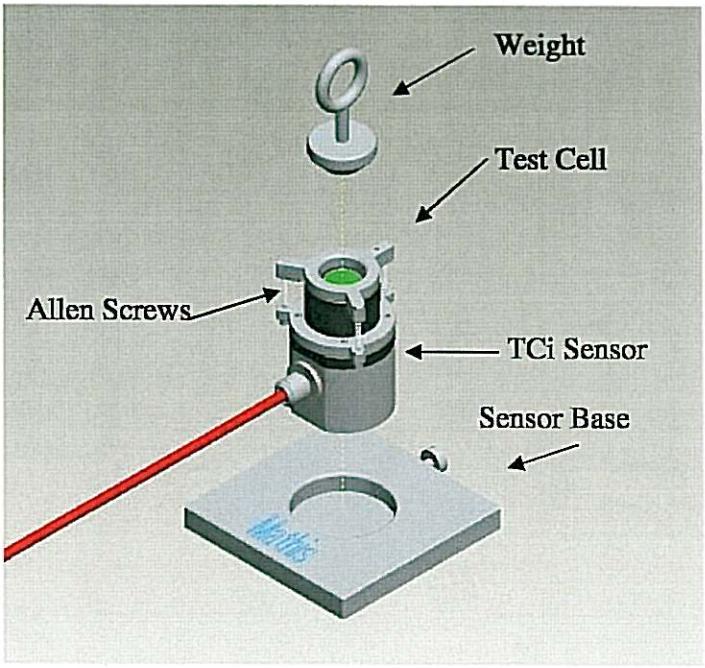
The SVTK was developed for testing minimal volumes of fluid material in partnership with the US Navy. Reducing the volume of sample material required for an effective thermal conductivity measurement is extremely important in the testing of energetic materials whereby larger samples pose a significant safety concern. The use of the accessory has also been applied widely in the testing of various materials that are doped with extremely expensive fillers (gold, diamond, silver, nano-materials, etc..) that are in limited supply.

3 Equipment: SVTK for use with C-Therm TCi™

SVTK P/N: TH060073, including additional parts not pictured:

- One 1/8 (tsp) measuring spoon, P/N: TH130051
- One 1/4 (tsp) measuring spoon, P/N: TH130052
- One 1"Quick Clamp Cap, P/N: TH040103
- Twenty VWR weighing dishes (41 mm × 41 mm × 8 mm), P/N: TH130022
- One Stainless Spatula (4"), P/N: TH130021
- 3/32" Allen wrench
- Five O-Ring RTV Seals

Note: TCi sensor (with base) is sold separately.



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4 Test Procedure

Before beginning powder or liquids testing, users should ensure that all parts of the apparatus are clean and free of debris or defect. The test cell collar should be securely attached to the TCi sensor with the provided screws. If testing energetic materials it is strongly recommended that all of the provided grounding straps be properly connected to limit the risk of electrostatic discharges.

4.1 Powder Testing

1. Fill the 1/8 tsp spoon (0.63 mL) with the powder to be investigated, as shown in Figure 1A.
2. Level off the excess powder by scraping off the excess with a spatula by making a horizontal movement, as shown in Figure 1B. The spatula should be applied on the side of handle and moved across to the opposite side of the teaspoon to scrape off the excess powder. Care must be taken to prevent compaction of the powder in the teaspoon (e.g. vibrations, rearranging powder with spatula, and tapping on the teaspoon).

Figure 1A & B – Powder Sampling

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3. The powder remaining in the teaspoon is the specimen. This material is transferred to the weighing dish.
4. Repeat the above steps three times (3X) for a total volume of specimen of approximately 3/8 (tsp) or 1.8 mL.
5. Taking a sample measurement
6. Carefully pour the specimen onto the test cell from the weighing dish, as shown in Figure 2 below. It is important to avoid tapping the weighing dish when pouring the sample. Ensure the active area (green area) of the sensor is fully covered by the powder. Avoid tamping or otherwise disturbing the sample.

Figure 2 – Sample Placement

7. Place the weight onto the sample so that it seats on the rim of the test cell as shown in Figure 3. No extra force is required, as the powder should completely fill in the enclosed volume.

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Figure 3 –Placement of Weight

8. Monitor the sensor temperature via the TCi software until it is stable and the sensor, sample and environment have all reached a state of thermal equilibrium.
9. Initiate the test sequence within the TCi software.

Note: It is recommended five measurements be conducted for each specimen improving the statistical accuracy of the measurement. Before testing the next specimen, follow the cleaning procedure described in Section 4.2.

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4.2 Cleaning

It is recommended that the sensor surface and test cell be cleaned using the following method before initiating the measurement of another specimen of a dissimilar powder. Otherwise, the sensor may be carefully wiped.

1. Pour out the contents of the specimen from the test cell. Place sensor upside down and remove the test cell by gradually unfastening the three screws in a sequenced manner. Use a 3/32" Allen wrench. (See Figure 4)
2. Remove sensor test cell and thoroughly clean the sensor surface and test cell using C-Therm's approved cleansers outlined in the user manual. The disassembled apparatus is shown in Figure 7.
3. To test again place the test cell on the sensor and place upside down in order to have easy access to the screws. Tighten gradually and in sequence until the test cell seats perfectly flat against the sensor-housing surface. Tighten the screws in the reverse sequence as utilized in step 2 above. Ensure that you do not apply excessive torque to the screws. Now you are ready to test again.

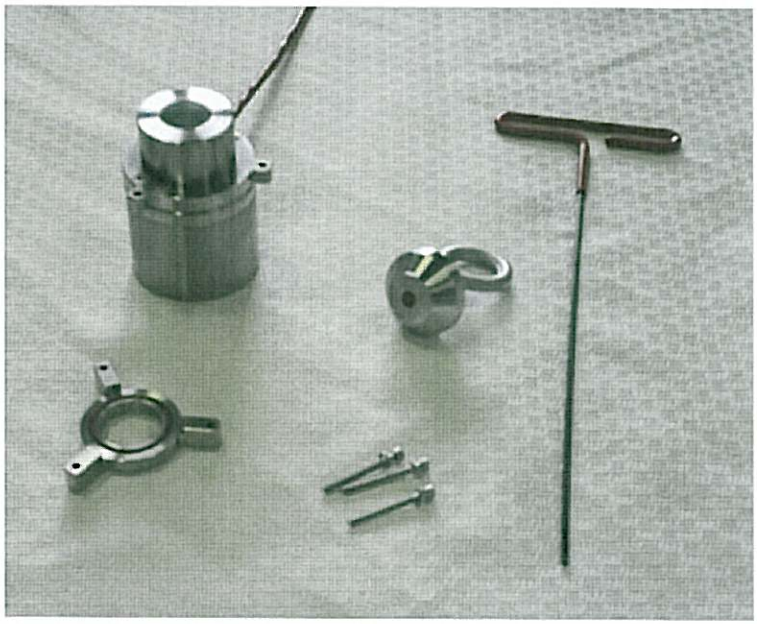

Figure 4 – Disassembled SVTK parts ready for cleaning

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4.3 Liquid Testing

1. Measure 1.25 ml (1/4 tsp) of liquid total volume of specimen.

Figure 5 – Dispense Sample

2. Transfer this volume directly to the test cell.

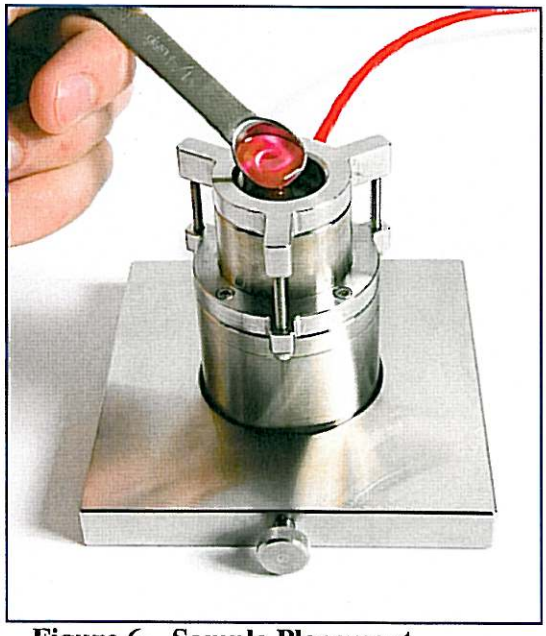

Figure 6 – Sample Placement

3. Place the quick clamp cap on the test cell. Use of the cap is optional – but will prevent any undesirable evaporation of liquid from the cell.
4. Monitor the sensor temperature via the TCi software until it is stable and the sensor, sample and environment have all reached a state of thermal equilibrium.
5. Initiate the test sequence within the TCi software.

Note: It is recommended five measurements be conducted for each specimen improving the statistical accuracy of the measurement. Before testing the next specimen, follow the cleaning procedure described in Section 4.2.

5 Method Performance Evaluation

For highly homogeneous powders and liquids, the RSD (relative standard deviation) of thermal conductivity measurements of several consecutive specimens of the same material should typically be less than 1%.

PDMS (DiMethyl PolySiloxane Silicone Fluid) is provided as a standard reference material with the TCi Thermal Conductivity Analyzer. Section 6.6 of the TCi Users Manual outlines how to perform a diagnostics accuracy check to verify the functionality of the instrument and the sensor calibration.