



User Guide for Differential Scanning Calorimeter (DSC)

Introduction: (Principles of the Test)

The Differential Scanning Calorimeter (DSC) is one of the best-selling products of Azmon Polymer Sepahan. The device is equipped with test control and management software designed to perform various tests in compliance with standards. This manual provides a step-by-step explanation of the software to facilitate easy learning.

In the **Oxidation Induction Time (OIT)** test, the time and temperature at which the antioxidant stabilizer in the sample prevents oxidation are measured. The sample is subjected to isothermal conditions at a specified temperature or a fixed heating rate in an oxygen or air atmosphere. The oxidation induction time or temperature indicates the stability level of the material under test. Performing the test at higher temperatures reduces the oxidation induction time, while faster heating rates increase the oxidation induction temperature. Additionally, results depend on factors such as the sample's surface area exposed to oxidation, thickness, and weight. It is important to note that samples tested in pure oxygen yield lower oxidation times and temperatures than those tested under standard atmospheric conditions.

In OIT and DSC tests, the sample is heated at a specified rate while placed in a thermal furnace. Simultaneously, temperature and energy variation curves are plotted, allowing for the analysis of the reactions that occur. In the DSC test, the sample is heated from ambient temperature at a specific rate, and its behavior is recorded as a DSC curve using the software. This allows thermal analysis of the sample and processes such as melting, degradation, and others, based on temperature and energy levels.

Section 1: Differential Scanning Calorimeter (DSC)

OIT Test: A Key Test for Polymer Materials

The **Oxidation Induction Time (OIT)** test is a crucial test for polymer materials. The purpose of this test is to determine the oxidation induction time. The detailed and technical instructions for the OIT test are provided in the national standard ISIRI 7186-6. OIT is a significant parameter influencing the quality and longevity of the material and the final product.

OIT Test Procedure:

A sample of approximately 15 ± 2 mg of material, which can be selected from different parts of the produced product or raw materials, is placed in the DSC sensor. The device's temperature is raised from ambient temperature to 200°C at a fixed rate, and nitrogen is introduced into the system during the heating process to neutralize the environment. Once the temperature stabilizes at 200°C , the nitrogen flow is stopped, and oxygen is introduced into the system. From the moment oxygen enters the system, the sample should be able to withstand oxidation for a defined period (e.g., 20 minutes) according to the relevant standard without undergoing oxidation.

OIT Test Standards:

The **ISIRI 7186-6** standard defines the procedure for determining the oxidation induction time and temperature of polymer materials using differential scanning calorimetry (DSC). This standard applies to polyolefin resins containing stabilizers, both as raw materials and final products. It may also be applicable to other plastics. The standard's title is "Plastics – Differential Scanning Calorimetry (DSC), Determination of Oxidation Induction Time (OIT Isothermal) and Oxidation Induction Temperature (OIT Dynamic)."

Objective and Scope of Application:

The purpose of this standard is to determine the oxidation induction time and temperature of polymer materials using differential scanning calorimetry. This standard applies to polyolefin resins containing stabilizing agents, both as raw materials and final products, and may also apply to other plastics.

Oxidation Induction Time:

This refers to the relative resistance of stabilized materials against oxidative degradation. The oxidation induction time is determined by measuring the heat during the period when the sample is placed under an oxygen or air atmosphere at a fixed temperature and atmospheric pressure, observing the start of exothermic oxidation. The result is expressed in minutes.

Oxidation Induction Temperature:

This refers to the relative resistance of stabilized materials against oxidative degradation. The oxidation induction temperature is determined by measuring the heat at the temperature at which the material begins to oxidize under an oxygen or air atmosphere and at atmospheric pressure. The result is expressed in degrees Celsius.

Principles of the Test:

The time and temperature at which the antioxidant stabilizer in the sample prevents oxidation are measured, while the sample is maintained under isothermal conditions at a fixed temperature or fixed heating rate in an oxygen or air atmosphere. The oxidation induction time or temperature evaluates the stability of the material under test.

Performing the test at higher temperatures reduces the oxidation induction time, and faster heating rates increase the oxidation induction temperature. The oxidation induction time and temperature are also dependent on the surface area of the sample exposed to oxidation. It should be noted that samples tested in pure oxygen will yield lower oxidation times and temperatures compared to samples tested under normal atmospheric conditions.

Note: The oxidation induction time or temperature can indicate the effectiveness of antioxidants in the sample. Careful interpretation of data is necessary since the kinetics of the oxidation reaction depend on temperature and the inherent properties of the additives in the sample. For instance, results from oxidation induction time and temperature tests are often used for selecting optimal resin formulations. Volatile antioxidants or variations in the activation energy of oxidation reactions may result in lower oxidation induction times and temperatures, whereas at the final product's operating temperature, they may provide optimal results.

Test Setup:

The sample and reference material are heated at a constant rate in an inert gas environment (nitrogen). When the temperature reaches a specific value, the inert gas atmosphere is replaced with oxygen or air (at the same flow rate). The sample is then kept at a constant temperature to observe the oxidation reaction in the oxidation curve. The induction period is the time between the introduction of oxygen or air into the system and the start of oxidation. The end of the induction period is marked by a significant increase in the heat released from the sample, which can be determined using differential scanning calorimetry. The oxidation induction time is determined according to section 9-6-1.

For dynamic oxidation induction temperature, the sample is heated in oxygen or air, and the start of the oxidation reaction is observed in the oxidation curve. The oxidation induction temperature is the temperature at which the oxidation reaction begins. This temperature is determined according to section 9-6-2.

Device Specifications:

The DSC device must be capable of reaching high temperatures (up to at least 500°C) and should have stable temperature control at the test temperature during the test period (usually 60 minutes). For high-accuracy equipment, an isothermal stability of $\pm 0.1^\circ\text{C}$ is recommended.

Sample Holder and Calibration:

Samples should be placed in open or vented closed containers to prevent unwanted contact with the surrounding atmosphere. It is recommended to use aluminum crucibles, but crucibles made from other materials may also be used based on mutual agreement. The container material may significantly affect test results, including any catalytic effects.

Gas Flow Calibration:

For gas flow calibration, a flow measurement tool such as a rotameter or soap-film flowmeter should be used in conjunction with a gas flow regulator. The flowmeter should be calibrated using a positive displacement tool.

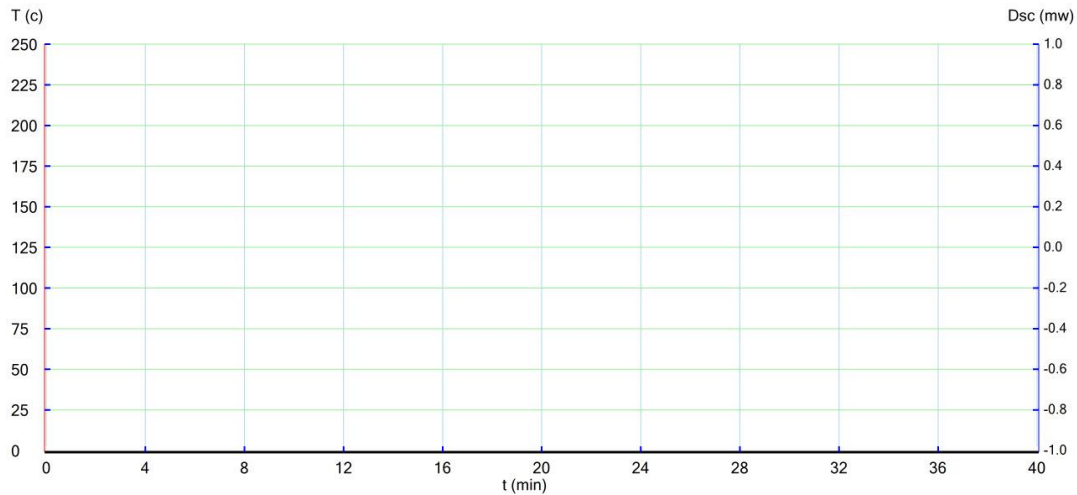
The oxygen used should be of high purity (99.5%) and completely dry. Similarly, nitrogen should also be of high purity (99.5%) and dry.

Warning: Compressed gases require appropriate safety measures and proper handling. Oxygen is a very strong oxidizer and can accelerate combustion. Equipment containing oxygen or using oxygen should be kept away from oils and greases. Compressed air should be dry and free from oils and greases.

The DSC device requires the cessation of nitrogen flow and the introduction of air or oxygen to measure oxidation induction time. The delay between the gas cutoff and switching to oxygen or air should be minimized, ensuring that the time lost is under one minute for a flow rate of 50 ml/min.

Note: If the lost time is known, more accurate results can be obtained. One way to determine the lost time is to perform the test using an unstabilized material that oxidizes rapidly in the presence of oxygen. The oxidation induction time for this test will provide an opportunity to correct the OIT for subsequent measurements.

Section Two: Main Program Screen



1-2- Main Menu of the Program

The main menu of the program is located at the top of the screen and contains various menus and submenus for managing and controlling tests, analyzing graphs, displaying and recording output data, as well as settings options to conduct tests in accordance with standards and user requirements.



1-3- Quick Access Toolbar

The Quick Access Toolbar is located at the top, beneath the main menus, and contains tools that are frequently used. Detailed explanations of each tool are provided in section four of the manual.

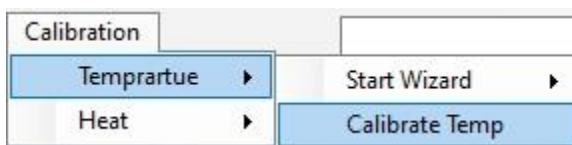
Section Three: Main Menu of the Program

Calibrating the Temperature

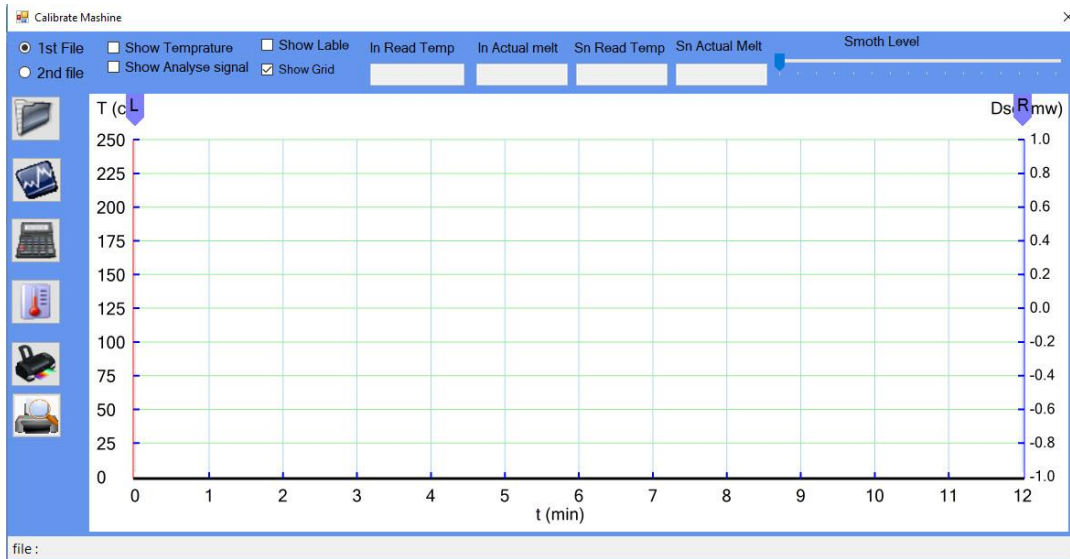
This device calibrates the temperature using a two-point method according to the standard. To do this, follow the calibration menu and obtain the two melting points. The software has a default tin and indium melting program, and you can also create and run a melting program for any other material by selecting the "Other" option.



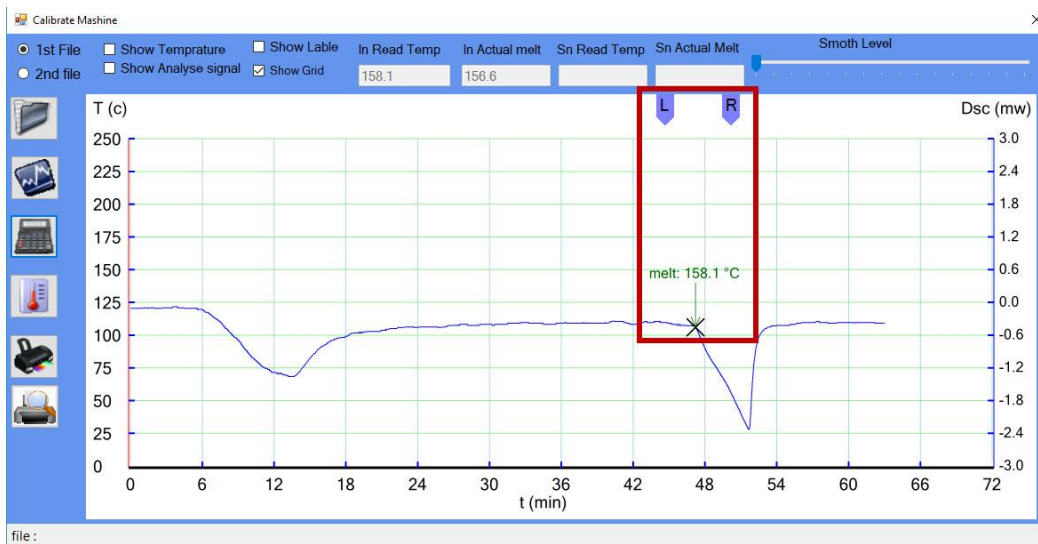
After the melting peaks are obtained, follow the path below.



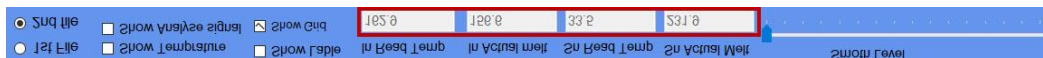
After selecting the option specified above, the following window will appear.



In this page, you need to load the two materials for which you have previously obtained the melting peaks. It is important to ensure that the melting temperature of the 1st file (Material 1) is lower than the melting temperature of the 2nd file (Material 2). When selecting each of these files, the actual melting temperature, which is the one provided in the material certificate and entered by the user in the start wizard, will be considered as the real melting temperature. To calculate the starting point of the melting peak, use the left and right sliding tools to position them before and after the start of this point on the diagram. Then, click on **Calculate melting point and view it on diagram** (the calculator icon) to perform the calculations.



After obtaining the melting points of both materials and filling in all four cells of the table, click on **Calibrate machine** (the thermometer icon) to calibrate the temperature.



Section Four: Quick Access Toolbar

3. Quick Access Toolbar

The Quick Access Toolbar includes the following tools for easy and efficient management of the testing process:

3-1. Open Diagram



Allows the user to open the temperature and energy graph for real-time monitoring and analysis of the test results.

3-2. Save Diagram



Enables saving the current diagram for future reference or analysis.

3-3. Stop Current Test



This option stops the ongoing test at any point during the experiment.

3-4. Alarm Check Connection



Activates an alarm to notify the user in case of any connectivity issues with the system or sensors.

3-5. Display Diagram Without Save Option and Start Test



Displays the diagram without the ability to save the data and begins a new test session.

3-6. OIT Test Settings



Allows configuration of test parameters for the Oxidative Induction Time (OIT) test, including temperature, flow rate, and time settings.

3-7. DSC Test Settings



Provides access to the settings for the Differential Scanning Calorimetry (DSC) test, allowing the user to customize the testing conditions such as heating rates and temperature range.

3-8. Exit Program



Closes the program safely, ensuring all data and test results are properly saved before exiting.

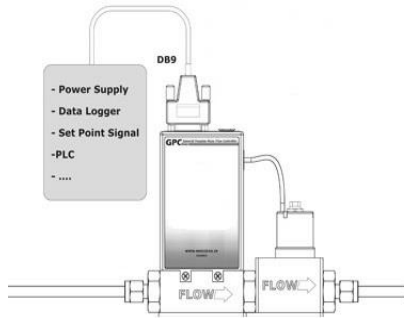
5. Performing the Test

OIT Test:

1. After ensuring that the gas valve used for the test is open and placing the sample in the device, as well as securing the chamber lids, use the OIT test icon on the main screen to enter the **Test Parameter** window.

*Do not forget to ensure that the **DB9 connector** (digital flow controller) is properly connected to the device and that its display is turned on before starting the test.*

In this flow controller, the larger number (the one displayed more prominently) indicates the output flow rate and the input to the device.



Test Parameter

file name

Sample Weight mgr

Constant temp °C Heat rate °C/Min

Gas 1 Flow 1 ml/Min

Gas 2 Flow 2 ml/Min

Operator Date

ComPort

- In this page, enter details such as the file name, sample type, sample weight, test temperature, heating rate, gases, gas flow rates, operators, test date, and more. Then, click **Next** to proceed to the **Temperature Plane Setting** page.

Temperature Plane Setting

OIT_PE.tpf

seg	Start.T	Final.T	Rate	Duration	Gas	Flow
1	0	200	20	10	N2	50.0
2	200	200	0	5	N2	50.0
3	200	200	0	150	O2	50.0

groupBox1

Start Temp (C)

Final Temp (C)

Rate (C/min)

Duration (min)

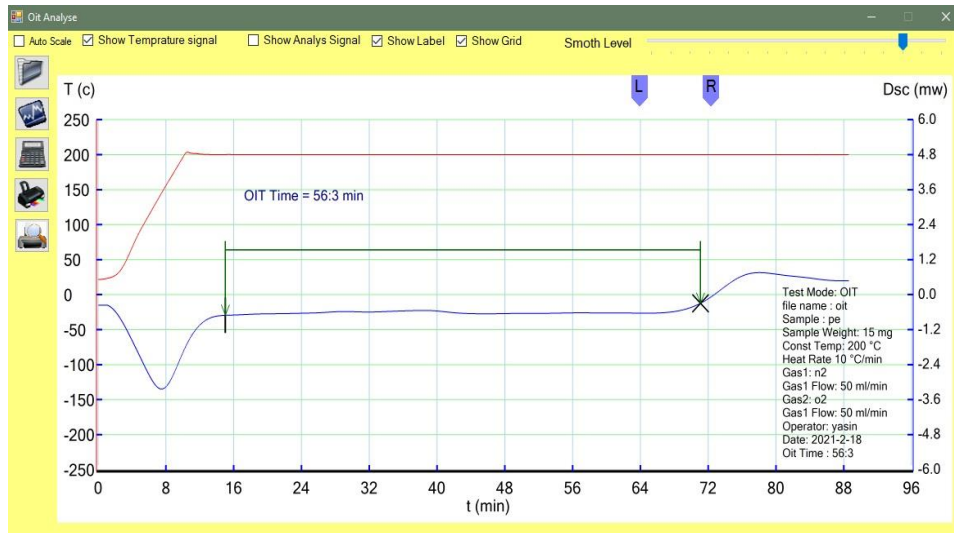
Gas

Gas flow

000.tpf
0_sn_calibre.tpf
In_Calibre.tpf
OIT_Fast.tpf
Oit_High.tpf
OIT_PE.tpf

file name:

- In this page, first, select your desired test program from the existing options or create and save a new program. To do this, after the page opens, enter a name in the **File Name** section, then define the program details in the **group box** and click **Add**. Finally, click **Save file** to save it. If you wish to modify an existing program, first click on it, then click **Load file** to view its steps. To modify any step, double-click the step number (seg) to highlight it in orange, and the details will appear on the left. Apply the necessary changes and click **Modify**, then save the changes by clicking **Save**.
4. After selecting and loading the program, click **Send Table** to return to the main program page, and the process will begin. The temperature curve and thermal behavior of the sample will be plotted simultaneously.
 5. When the test process is complete, the device will notify you with an alarm. You can save the test by clicking **Save as** in the **File** menu.
 6. Now, use the **Analyse** option and then click **oit calculate** to enter the related page.



To calculate the oxidation induction time (OIT) for the sample, place the left tool (L) before the exothermic peak and the right tool (R) after the peak. Then click the **Calculate** icon (calculator). By checking the **Show Label** option, the test and sample information entered previously in the **Set Parameter** page will be displayed. You can click and drag the display cursor to place it at the desired location.

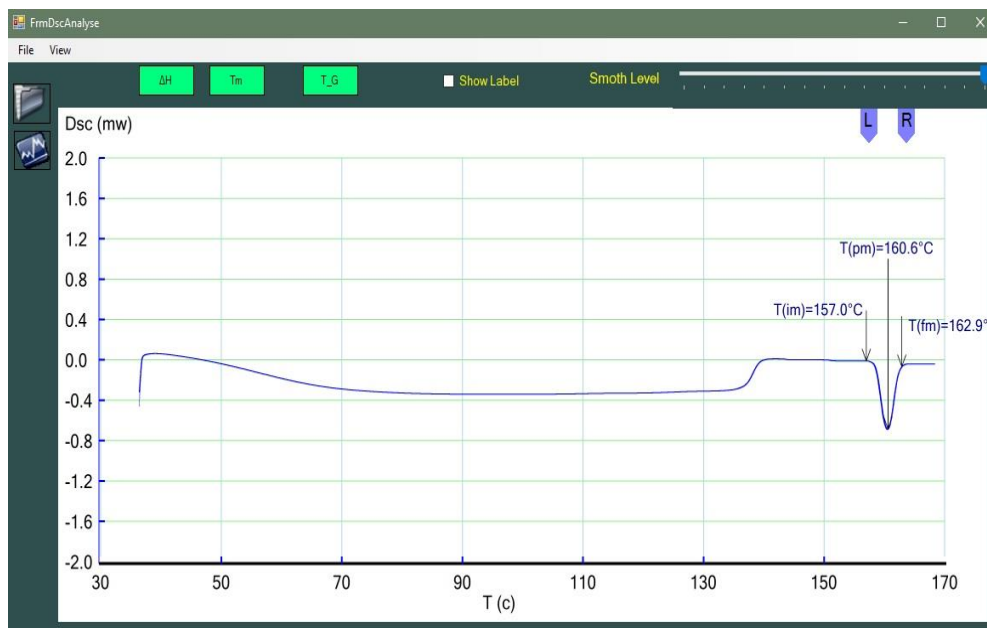
The **Show Grid** option allows you to grid the page, and the **Show Temperature Signal** and **Show Analyse Signal** options control the visibility of the temperature and thermal behavior curves of the sample.

To save the page, you can right-click anywhere on the curve and select **Export to PDF** or **Save Image** to store the test data. The print options are also available on this page.

DSC Test:

After ensuring the gas valve used in the test is open and placing the sample in the device, as well as closing the chamber lid, use the **DSC Test** icon from the main page to enter the **Test Parameter** window.

Follow steps 2-5 as described for the OIT test, then use the **DSC Calculate** option available in the **Analyse** section to enter the related page. By clicking the **Folder** icon at the top of the screen, you can open the test file that was completed.



To calculate the melting temperatures (T_m) and enthalpy of reaction (ΔH), place the left tool (L) before the peak and the right tool (R) after the peak, then click on the **Tm** or **ΔH** option.

After completing the tests (DSC, OIT) and saving the data, if you want to view the gas flow curve during the test, first open the test file from the main program page. Then, select the **Gas Flow** option from the tools above the curve. The behavior and changes in the gas flow will be visible on the corresponding axes, displayed in **ml/min**.

