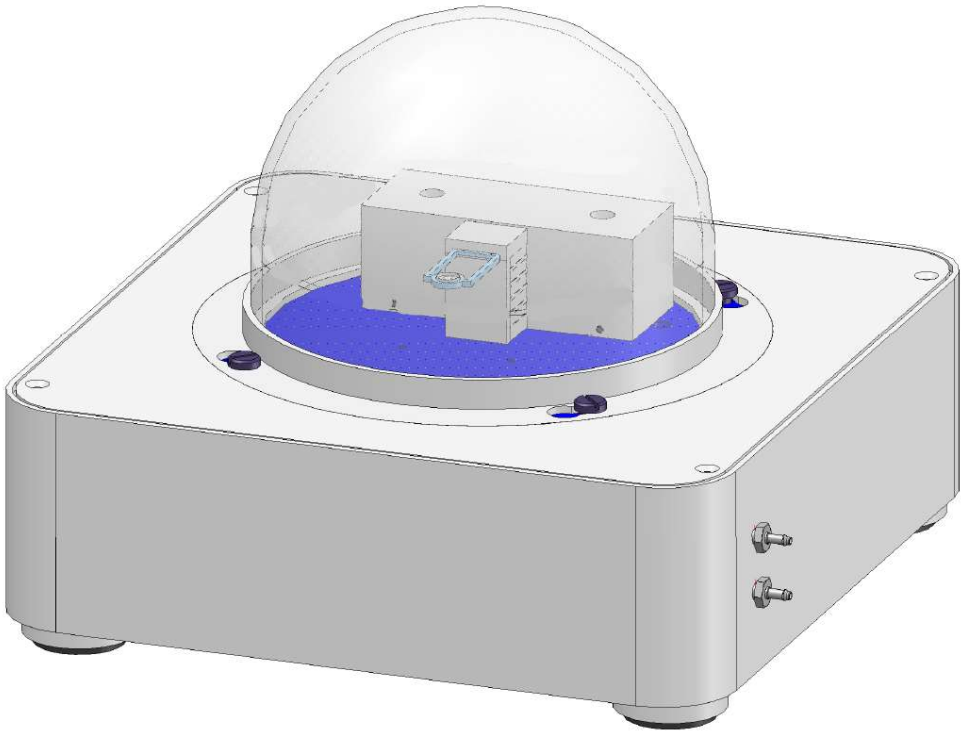
How To

Measure OIT/OOT

with Chip DSC



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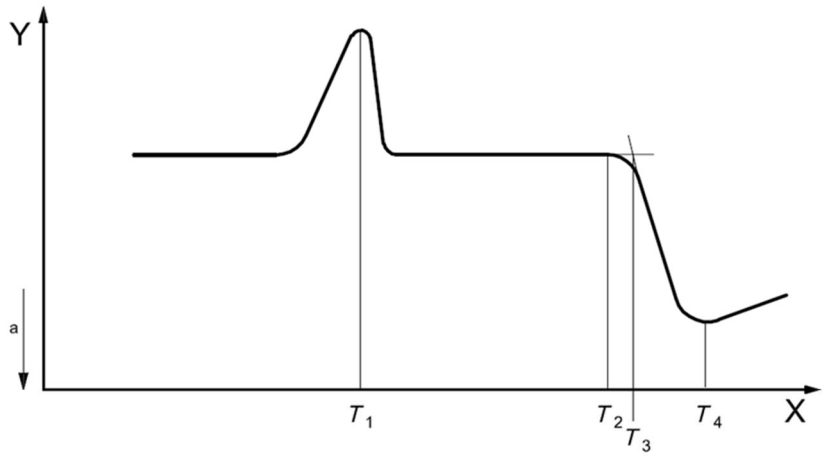
1. General information

This manual is a short description of how to measure Oxidation Onset Temperature (OOT) and Oxidation Induction Time (OIT) with Chip DSC. These measurements are used to evaluate the oxidation stability of plastic samples and the specifics are covered in more detail in the ISO 11357-6. Generally speaking, the OOT approach measures the temperature when oxidation starts to occur at a specific heating rate, whereas the QIT approach measures the time when oxidations starts to occur at an isothermal condition. The Chip DSC is well suited for both of these types of investigations.

2. Oxidation Onset Temperature (OOT)

The OOT test (sometimes also called dynamic OIT – Oxidation Induction Temperature) determines the substance-specific temperature at which the oxidation of a sample begins. This method is dynamic so it is associated with a defined heating rate. The oxidizing agent is contained in the atmosphere of the measuring cell at all times. The sample is heated at 10 K / min or 20 K/min in accordance with ISO 11357-6. After the start of the exothermic oxidation, the measurement is stopped. To determine the specific temperature, a tangent is fitted in the course of the baseline and the rising edge of the oxidation and the point of intersection is determined. During the measurement, the gas flow should be set in such a way that the gas in the measuring cell is exchanged sufficiently quickly but that no great noise can be seen on the measurement signal.



Key

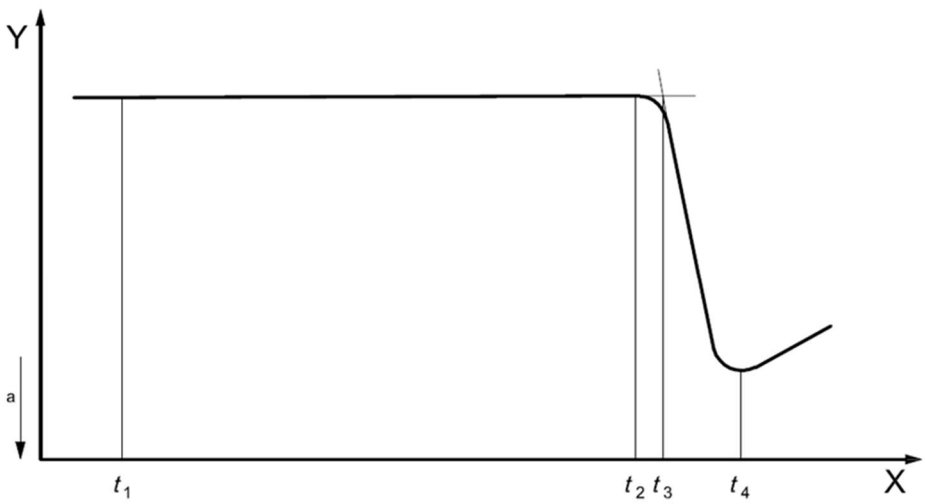
X temperature
Y heat flow rate

T_1 melting temperature of polymer
T_2 onset of oxidation
T_3 intercept point determined by tangent method (oxidation induction temperature)
T_4 temperature of oxidation peak

a Exothermic.

3. Oxidation Induction Time (OIT)

The OIT test (also called isothermal OIT test) determines the time after which a sample begins to oxidize. There is no oxidizing agent in the sample space before the actual measurement. In an inert atmosphere, the sample is heated up to a temperature below the OOT and kept isothermal. At a certain point in time, the gas control system adds the oxidizing agent (air or pure oxygen). The measurement is ended after the start of the oxidation. To determine the point in time of the OIT, a tangent is fitted to the baseline and the rising edge of the oxidation and the point of intersection is determined. During the measurement, the gas flow should be set in such a way that the gas in the measuring cell is exchanged sufficiently quickly but that no great noise can be seen on the measurement signal.



Key

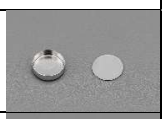
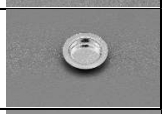
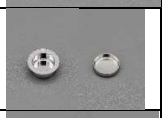
X time
Y heat flow rate

t_1 changeover to oxygen or air (time zero)
t_2 onset of oxidation
t_3 intercept point determined by tangent method (oxidation induction time)
t_4 time to oxidation peak

a Exothermic.

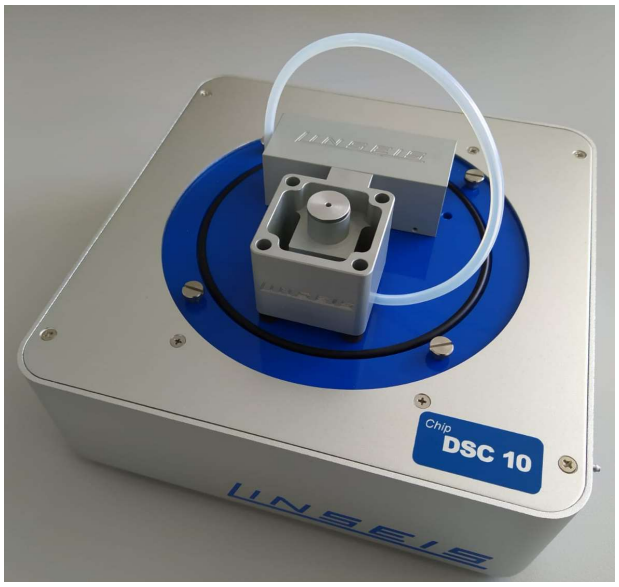
4. Crucibles

The ISO 11357-6 standard states that the samples should ideally be measured in an aluminum crucible. Since these fit perfectly into the temperature range of the Chip-DSC, there is a large selection of possible crucibles. In order to guarantee a good gas exchange, however, no lid may be used for OIT / OOT tests. A copper crucible can also be used to achieve a catalytic effect. In order to achieve comparable results, however, you should ensure that the crucible dimensions and material are comparable.

Name	Best.-Nr.	Description	Picture
Al Standard crucible 6x1,5mm 40µl	30293042	The most lightweight crucible and easy to use	
Al crimpable crucible 100µl	30293043	The biggest crucible for voluminous samples. Crimpable to measure with atmosphere	
Al crimpable crucible 40µl	30293045	The small crimpable crucible to measure with atmosphere	
Al crucible for foils with lid	30293050	Crimpable crucible to measure foils and powders. Special crimping tool is needed.	
Al crucible for small samples with lid	30293051 30293052	Crimpable crucible to measure small samples. Special crimping tool is needed	
Cu crucible	30293049	The copper crucible to measure Oxidation induction time and Oxidation onset temperature	

5. Gas Adapter

To make OIT / OOT measurements with a Chip-DSC 1 or Chip-DSC 10, it is possible to use an additional adapter. This leads the gas directly to the sample and, thanks to the significantly smaller sample space, enables a considerably faster gas exchange at the sample, which makes the results clearer. In addition, the additional shielding reduces interference. The block can simply be screwed onto the device.



6. Measure under high pressure

To show the oxidation more clearly, it is possible to measure under increased pressure. The oxidation reaction proceeds faster under high pressure, as more oxygen reaches the sample. Therefore the Oxidation Induction Time is shortened depending on the pressure. In order to make the results comparable, however, the reference material and the sample must be measured under identical conditions. With the help of a high pressure measurement, productivity and the quality of the measurement signal can be significantly increased. With increased pressure, however, there is also increased convection and higher thermal conductivity, which is why the measurement signal shows more noise and the maximum temperature that can be reached drops slightly.