

## Determination of the Oxidation Stability of Fats and Oils

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### Introduction

External influences such as UV radiation (light), temperature, atmospheric oxygen or chemical/biological media lead to premature aging in materials, resulting in a change in their chemical and physical properties. Proper aging inhibitors (stabilizers) slow down the aging process and prolong the induction period, i.e. the time span preceding the onset of thermo-oxidative decomposition (chain degradation, technical failure).

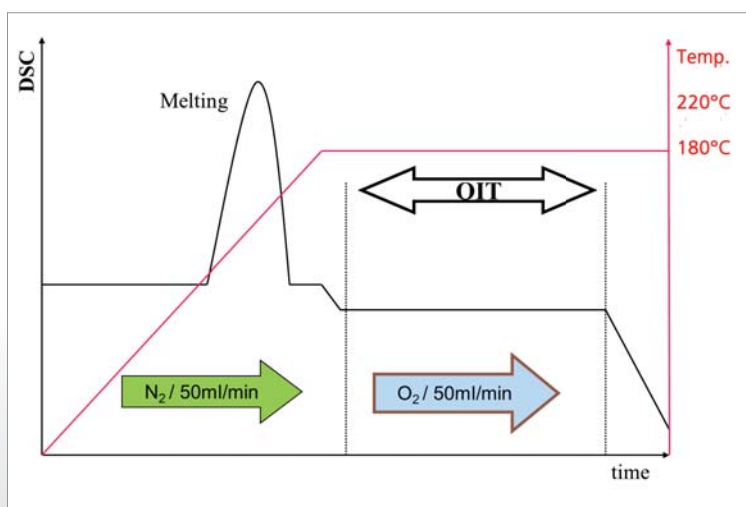
An important indicator of the oxidation stability of oils, fats, lubricants, fuels or plastics is the Oxidative-Induction Temperature or Oxidative-Induction Time (OIT) which can be determined by means of DSC in standardized procedures.

In practice, two different methods are used: dynamic and isothermal OIT tests. In the dynamic technique, the sample is heated at a defined constant heating rate under oxidizing conditions until the reaction begins. The corresponding Oxidative-Induction Temperature (also called Oxidation Onset Temperature, OOT) is the same as the extrapolated onset temperature of the exothermal DSC effect which occurs.



2 NETZSCH Pressure DSC 204 (max. pressure: 150 bar)

In isothermal OIT tests, the materials to be investigated are first heated under a protective gas, then held at a constant temperature for several minutes under protective gas to establish equilibrium, and subsequently exposed to an atmosphere of oxygen or air. The time span from the first contact with oxygen until the beginning of oxidation is called the Oxidative-Induction Time. This is shown in figure 1.



1 Determination of the Oxidative-Induction Time on a polyolefin as per ISO 11357-6

Many national and international standards – such as ASTM D3895 (polyethylene), DIN EN728 (plastic pipelines), ISO 11357-6 (plastics) and ASTM D525 (aircraft fuel) – give recommendations for sample preparation and proper selection of the measurement conditions.

### Oxidation Tests on Lubricating Oils and Greases

Oxidation tests on lubricating oils and greases are usually carried out using a high-pressure DSC instrument (see figure 2). A back-pressure is generated

## APPLICATIONNOTE Determination of the Oxidation Stability of Fats and Oils

– generally at 35 bar – in an attempt to prevent evaporation of the sample. In oxidation reactions, however, the oxygen not only serves for pressure generation, but also as a reaction partner. For this reason, both the pressure and the gas flow must be regulated with the utmost precision.

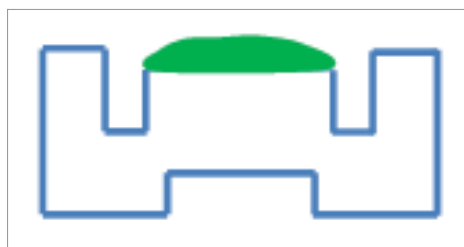
Determination of the oxidation stability is “surface-sensitive”. This means that the oil or grease film to be investigated should ideally exhibit a smooth, uniform surface in order to ensure high reproducibility of the test results. Very well suited for such investigations are SFI crucibles (SFI stands for Solid Fat Index; see diagram in figure 3), as recommended in ASTM D5483 for lubricating greases and ASTM D6186 for lubricating oils.

An example of these would be pan-shaped aluminum crucibles with an outer diameter of 6.7 mm and a volume of 85  $\mu\text{l}$  which can be shaped with a sealing tool (built into a standard crucible press – see figure 4).

In crucibles with a flat bottom, oils and fats often creep to the rim zones at higher temperatures. The effective surface of the sample which can interact with the surrounding atmosphere is thus reduced in size. This affects the OIT results (see figure 5). When the analysis is conducted in an open standard crucible (blue curve), the OIT time (extrapolated onset) amount to 64.6 min. In comparison, when analyzed in an SFI crucible (green curve), the OIT is shortened considerably (to 46.4 min) due to the larger effective surface.

### Summary

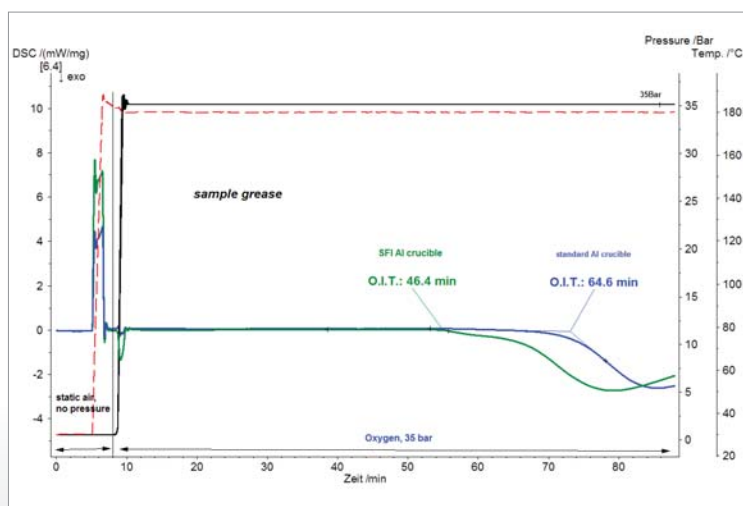
With the help of DSC-OIT tests, comparative conclusions on the stability of materials compared to thermo-oxidative attacks can be drawn. For the investigation of lubricating oils and greases, the NETZSCH High-Pressure DSC 204 in combination with SFI crucibles is ideally suited.



3 Diagram of an SFI crucible with sample (green)



4 Crucible press and insert (enlarged scaling)



5 Comparison of the OIT times of a grease analyzed in a standard aluminum crucible (blue) versus an SFI crucible (green); instrument: DSC 204; 35 bar oxygen