

T_g determination using DSC

Testing methods

The methods to be used should be

1. with DSC, as described below (“Innventia”)
2. your in-house DSC method and/or
3. your in-house DTA method

The results from 2 and 3 should be reported along with a description of your method.

Procedure

Sample preparation

A sample of 1-3mg of lignin sample is placed in a hermetic aluminum pan. The pans used at Innventia has a diameter of ~5mm. Adapt the sample size to your pan size, if different. The lignin powder should cover the bottom of the pan. A hole is produced by piercing the lid with a pair of tweezers (∅ ~0.5mm). The sample should cover the bottom of the pan to ensure good contact. The sample is weighed after piercing of the lid. The method used is modulated reversed calorimetry where the temperature will be increased by oscillation of ±3°C every 60 seconds.

The analysis performed at Innventia is made with TA Instrument Q100 according to:

Drying cycle

1. Ramp 1°C/min up to 105°C.
2. Isothermal at 105°C for 20 min
3. Quench to 20°C
4. Isothermal at 20°C for 10 minutes

Test cycle

5. Ramp 3°C/min up to 250°C
6. End of test

Evaluation

The glass transition temperature can be defined in different ways (Mazurin 2007). In literature, both the onset temperature and the inflection point temperature is used to report T_g. This represents point A and B as shown in **figure 1**.

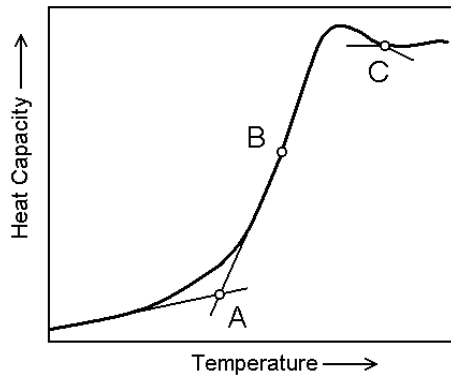


Figure 1: A, B the onset temperature and the inflection point, respectively.

Universal analysis 2000 (version 4.5A) has been used for T_g determination. In this software T_g is defined as point B, **the inflection point which is the one you should report to us.**

T_d determination using TGA

Procedure

The sample size as for DSC analysis should be used. T_d , defined as the temperature where **95% remains of the initial dry sample, should be reported.**

1. Drying at 105°C during 20 minutes
2. Ramp 15°C/min up to 350°C

Report which instrument that was used and any deviation from the above procedure.

General comment

Please note that both analysis (T_g and TD determination) should be performed in inert atmosphere, e.g. under nitrogen. For statistical calculations, please report $n=5$ for each analysis performed.