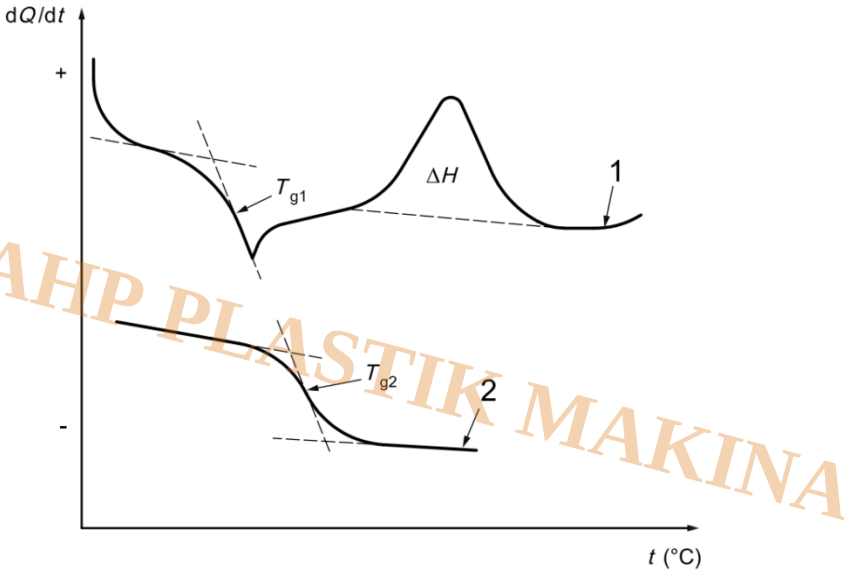
DIN EN ISO 21809-1 External Coatings for Buried or Submerged Pipelines/ Polyolefin coatings (3-layer PE and 3-layer PP) / Annex D Thermal Analysis / Testing Equipment

Description

D.1 General
Thermal analysis shall be used to characterize the uncured epoxy (powder, one-component liquid or two-component liquid) and the cured coating film.
Differential scanning calorimetry (DSC) shall be used. Reference can be made to ISO 11357-2 for a description of the general procedure and definitions. General handling and calibration shall be performed as in
ISO 11357-2 unless stated otherwise in this part of ISO 21809.
D.2 Equipment
D.2.1 Differential scanning calorimeter (DSC), with cooling accessory.
D.2.2 Balance, accurate to 0,1 mg.
D.2.3 Sample-encapsulating press.
D.2.4 Aluminium pans, with covers.
D.2.5 Nitrogen gas supply, dry, analytical grade

D.3 Procedure and measurement for epoxy
D.3.1 Procedure
For two-component epoxy liquid, taking into consideration the supplierâ€™s recommendations, accurately and separately homogenize each component before use and mix them together in the exact mixing ratio; then continue to homogenize completely the mix of base and hardener for about 5 min. The minimum quantity to be mixed is 100 g to avoid mixing mistakes.
For one-component epoxy liquid, taking into consideration the supplierâ€™s recommendations, completely
homogenize 100 g of the sample before use.
D.3.2 Measurement
Perform the following heating cycles, starting with run (a) as the conditioning run for powder epoxy only.
âŽ¯ Run (a): heat the sample from 25 Â°C Â± 5 Â°C to 70 Â°C Â± 5 Â°C at a rate of 20 Â°C/min, then immediately cool
the sample to 25 Â°C Â± 5 Â°C.
âŽ¯ Run (b): heat the sample from 25 Â°C Â± 5 Â°C to 275 Â°C Â± 5 Â°C at a rate of 20 Â°C/min, then immediately cool the sample to 25 Â°C Â± 5 Â°C. Hold for 3 min at 25 Â°C Â± 5 Â°C.
âŽ¯ Run (c): heat the sample from 25 Â°C Â± 5 Â°C to Tg + 40 Â°C (typically 150 Â°C) at a rate of 20 Â°C/min, then
immediately cool the sample to 25 Â°C Â± 5 Â°C.
For certain epoxies, different heating cycles can be required according to the instructions of the epoxy manufacturer.

D.4 Evaluation of results
D.4.1 Glass transition temperature
The glass transition temperature, Tg, is calculated at the point of inflection intersection (see Figure D.1). By evaluating run (b), the Tg of the uncured powder obtained is equal to Tg1. By evaluating run (c), the Tg of the cured material (powder and liquid) obtained is equal to Tg2.
D.4.2 Heat of reaction of the epoxy
The exothermic heat of reaction, â^†H, is obtained by integrating the exothermic peak of the DSC scan.



Key
1 run (b)
2 run (c)

Figure D.1 — Examples of thermal scans on epoxy powder

D.5 Procedures and measurement for coating sample
D.5.1 Sample preparation
D.5.1.1 Two-component epoxy liquid
Accurately and separately homogenize each component before use in accordance with the instructions of the manufacturer.
Mix them together in the exact ratio and completely homogenize the mix of base and hardener for about 5 min.
Apply the product at the thickness of 500 Âµm on an aluminium panel that has been completely degreased.
The thickness of the panel is about 1 mm.

Let the film cure for at least 2 h at ambient temperature. Put the panel in a ventilated oven for 15 min at 170 Â°C, then take it out and let it cool at ambient temperature.
After about 2 h, bend the aluminium panel and take off one or more scales of the film in order to get the necessary mass of material to put in the test capsule.
As an alternative, a representative sample of cured film may be taken directly from the pipe.
D.5.1.2 One-component epoxy liquid
Homogenize the sample before use in accordance with the instructions of the manufacturer.

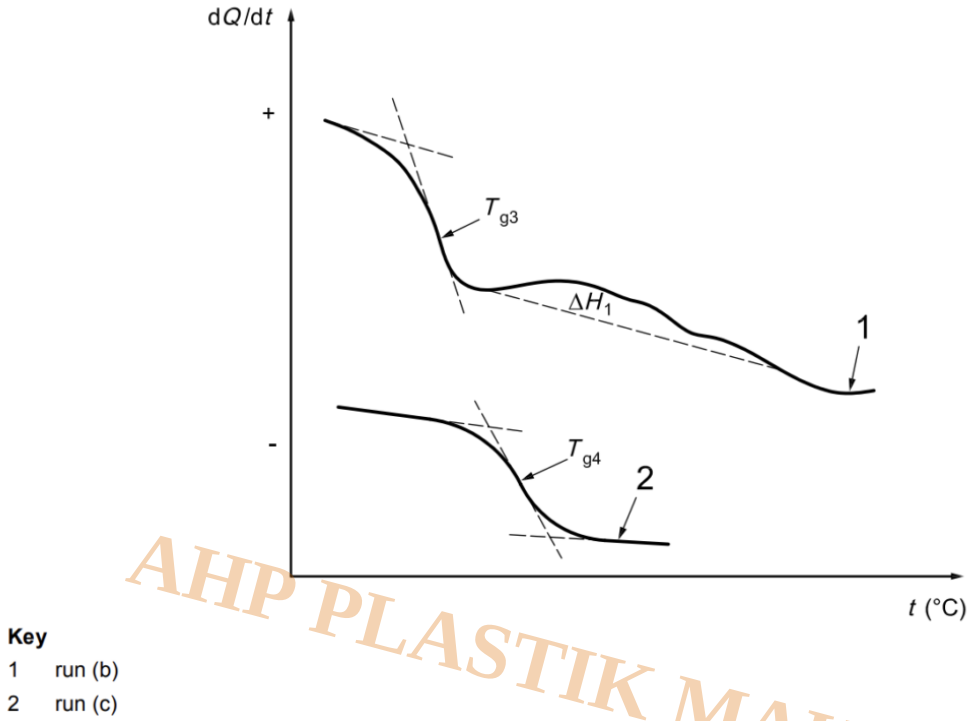
Apply the product at the thickness of 500 Âµm on an aluminium panel that has been completely degreased.
The thickness of the panel is about 1 mm. Let the film cure for at least 2 h at ambient temperature. Put the panel in ventilated oven for 15 min at 170 Â°C, then take it out and let it cool at ambient temperature.
After about 2 h, bend the aluminium panel and take off one or more scales of the film in order to get the necessary mass of material to put in the test capsule.
As an alternative, a representative sample of cured film may be taken directly from the pipe.

D.5.1.3 Epoxy powder
A representative sample of the cured film shall be taken directly from the pipe.
Weigh out 10 mg Â± 3 mg to an accuracy of 0,1 mg. The pan is sealed with the cover. Determine the final mass after sealing.
Place the sample and the reference sample in the DSC cell and purge with dry, nitrogen gas.
Samples taken from pipes that have been stored or buried shall be dried before testing.
D.5.2 Measurement
The following heating cycles shall be performed, starting with run (a) as the conditioning run for the powder samples only.
Liquid epoxy samples shall start with run (b).
âŽ¯ Run (a): heat the sample from 25 Â°C Â± 5 Â°C to 110 Â°C Â± 5 Â°C at a rate of 20 Â°C/min and hold for 1,5 min,
then cool the sample to 25 Â°C Â± 5 Â°C.
âŽ¯ Run (b): heat the sample from 25 Â°C Â± 5 Â°C to 275 Â°C Â± 5 Â°C at a rate of 20 Â°C/min, then cool the sample
to 25 Â°C Â± 5 Â°C. Hold for 3 min at 25 Â°C Â± 5 Â°C.
âŽ¯ Run (c): heat the sample from 25 Â°C Â± 5 Â°C to Tg + 40 Â°C (typically 150 Â°C) at a rate of 20 Â°C/min, then
cool the sample to 25 Â°C Â± 5 Â°C.
For certain epoxies, different heating cycles can be necessary according to the instructions of the epoxy manufacturer. Samples taken from pipes that have been stored or buried shall be dried before testing.

D.5.3 Evaluation of results
D.5.3.1 Glass transition temperature
The glass transition temperature, Tg, shall be calculated in the same way for the epoxy for run (b) and run (c) in Figure D.2 where Tg3 is Tg of run (b) in D.5.2 and Tg4 is Tg of run (c) in D.5.2, all expressed in degrees Celsius.

For coatings, determine âˆ†Tg, the change in Tg value, using Equation (D.1) and the Tg values defined in
paragraph 1.

$$\Delta T_g = T_{g4} - T_{g3}$$



Key
1 run (b)
2 run (c)

Figure D.2 — Examples of thermal scans on coating

D.5.3.2 Residual heat of reaction of cured coating
The exothermic heat of reaction, âˆ†H1, shall be obtained by integrating the exothermic peak of the DSC scan run (b) in Figure D.2.
In a fully cured coating film, no residual heat of reaction should be observed.
The degree of conversion, C, expressed as a percentage, can be calculated as given in Equation (D.2):

$$C = \frac{\Delta H - \Delta H_1}{\Delta H} \times 100$$

where

ΔH is the exothermic heat of reaction of the powder; run (b) in D.3.2

ΔH_1 is the exothermic heat of reaction of the powder; run (b) in D 5.2

D.6 Results and test report
Record the following information on uncured and cured material:
â˘ reference to this part of ISO 21809;
â˘ type of material and batch number;
â˘ date of test;
â˘ type of DSC equipment;
â˘ for the epoxy powder: Tg1, Tg2, âˆ†H;

âŽ¯ for the epoxy liquids: Tg2, âˆ†H;
âŽ¯ for the cured coating film: Tg3, Tg4, âˆ†Tg, âˆ†H1 and C.
Testing of production coating shall also require the pipe number or identification.

DSC Tester According to ISO 21809-1

- According to ASTM D3895 ISO11357 EN728 ASTM D3418
- Up to 300C
- Including standard Tin, Ind pellets for calibration checkout
- 400pcs of aluminium pans
- Software flash disk (windows based software including Report out to MS WORD)
- N2 and O2 rotameter mass flow controller
- Hoses and connectors
- Software also has data export to Advantage UA Analysis Software for analysis
- Temperature sensors for control & sample temperature
- Connection for Purge gas flow to the sample for 2 gas types
- Temperature accuracy 0.1 C
- Sensitivity: 3.6 to 4.0 Î¼V/mW
- Sensor Time constant: 2.5 s
- Calorimeter accuracy of Â± 2%
- Calorimetric precision of Â± 0.5% or better
- Two connections for purge gas-Inlet to the sample & 1 connection for cooling air
- 7/24 online distance support
- Flow controller are rotameter type (digital type is as option)

Category

1. Equipment for Standards
2. Standards