

Application Bulletin



Of interest for:
Plants for the manufacture and processing of plastics

No. 205/1 e

Determination of the thermal stability of PVC and related copolymers

Summary	<p>The determination of the thermal stability of PVC in conformity with DIN 53381, Part 1, and ISO 182:1970 is described using the 679 Rancimat. The instrument allows the fully automatic and simultaneous determination of the stability and induction time.</p> <p>The test is suitable for monitoring of the manufacture and processing, for a delivery check, for the characterisation and comparison of PVC products, as well as for testing the effectiveness of heat stabilizers in molded PVC materials.</p>
Apparatus	<ul style="list-style-type: none">▶ 2.679.001X Rancimat
Reagents	<ul style="list-style-type: none">▶ Distilled or deionized water▶ RBS 25-concentrate; Fluka No. 83460 (for cleaning the reaction vessels)
Sample preparation	<ul style="list-style-type: none">▶ Powdery samples can be used directly.▶ Foils and other solid products are disintegrated. The border length of the disintegrated parts shall not be bigger than 2 mm.
Method	<ul style="list-style-type: none">▶ The Rancimat is calibrated at 200°C as described in the instructions for use.▶ Evaluation mode 1 (induction time) and 2 (time for $\Delta\kappa = 50 \mu\text{S}/\text{cm}$), a measuring range of 100 μS and a chart speed of 10 cm/h are selected.▶ Dist. or deion. water (60 ... 70 mL) is added to the absorption vessels and the measuring cells installed ensuring freedom from air bubbles and coverage of the top lateral holes (rotate vessel gently). The measuring cells are then closed and their value entered in the instrument under cell constants.▶ The reaction vessels are preheated in the heating block.▶ Sample (0.5 g) is now added to each reaction vessel, the air flow and the absorption vessels (air flow 7 L/h) immediately attached and the analysis started with "GO".
Results	<ul style="list-style-type: none">▶ The induction time t_i is the time needed to reach the break point in the curve (Evaluation 1).▶ The stability time t_{st} is the time at which, under the conditions described above, a certain amount of HCl has been split off ($\Delta\kappa = 50 \mu\text{S}/\text{cm}$) (Evaluation 2).
Remarks	<ul style="list-style-type: none">▶ Duplicate determinations should be performed.▶ The reaction vessels can be cleaned as follows: They are heated at 80 ... 100°C for about 1 h with 2 mL RBS concentrate and then rinsed with hot water. After rinsing with dist. water, they are dried.
Literature	<ul style="list-style-type: none">▶ DIN 53 381 Part 1 <i>Bestimmung der Thermostabilität von Polyvinylchlorid (PVC). Dehydrochlorierungsverfahren.</i>▶ ISO 182:1970 <i>Plastics – Determination of the thermal stability of polyvinyl chloride and related copolymers and their compounds by splitting off of hydrogen chloride.</i>

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Curve examples

Determination of the thermal stability of three different PVC foils with conditions and result block.

- Positions 1, 2: Sample 1
- Positions 3, 4: Sample 2
- Positions 5, 6: Sample 3

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METROHM 679 RANCIMAT          METHOD 0
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RESULTS
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ch  smp1. ident  eval.1  eval.2  eval.3
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1  1.1          1.37 h  1.42 h  0.9 uS/cm
2  1.2          1.35 h  1.38 h  1.1 uS/cm
3  2.1          1.55 h  1.58 h  1.4 uS/cm
4  2.2          1.55 h  1.58 h  1.0 uS/cm
5  3.1          1.87 h  1.84 h  0.2 uS/cm
6  3.2          1.75 h  1.78 h  0.0.2 uS/cm

eval.1:  induction time
eval.2:  time at delta K = 50 uS/cm
eval.3:  delta K at t = 1 h

DATE 89-04-20          TIME 15:35

PARAMETERS
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temperature            200 Cel
temperature correction  0.0 Cel
conductivity range     100 uS/cm
evaluation modes       1/2/3
  eval.2: delta K      50 uS/cm
  eval.3: delta t      .1 h
delay time              0 h
paper feed              10 cm/h
cell constants: channel 1 0.77 /cm
                   channel 2 0.77 /cm
                   channel 3 0.79 /cm
                   channel 4 0.81 /cm
                   channel 5 0.80 /cm
                   channel 6 0.81 /cm
measuring time         30 h
end mode: EP stop      ON
                   heater stop ON
                   air stop  OFF
    
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