Metrohm

Application Bulletin 205/2 e

Thermostability of PVC and related chlorinated polymers

Branch

Plastics; polymers

Keywords

Thermostability; thermal stability; 895; PVC; polyvinyl chloride; chlorinated polymers; sample preparation; branch 6

Summary

The determination of the thermostability of PVC (also called dehydrochlorination test or DHC test) characterizes the stability of polyvinyl chloride or related chlorinated polymers against heat exposure.

During the test, nitrogen is passing over the sample in the reaction vessel at constant elevated temperature. When the sample decomposes, hydrochloric acid gas is formed which is transported into the measuring vessel by the nitrogen stream and absorbed in the measuring solution (deionized water). The continuously recorded electrical conductivity of the measuring solution increases due to the absorption of the HCl gas. Thus, its appearance can be detected. The time until the conductivity has changed by a predefined value is called stability time and characterizes the thermostability of the PVC material.

The determination of the thermostability of PVC is described according to DIN 53381 part 1 or ISO 182 part 3 using the 895 Professional PVC Thermomat. The test is suitable for monitoring of the manufacture and processing, for a delivery check, for the characterization and comparison of PVC products as well as for testing the effectiveness of heat stabilizers in molded PVC materials.

This Application Bulletin provides a detailed description including necessary sample preparation steps.

Samples

Polyvinyl chloride (PVC) or related chlorinated polymers

Instruments

895 Professional PVC Thermomat	2.895.0010
Equipment for the determination of the temperature correction	6.5616.100
Nitrogen, Φ < 99.995%, pressure 1.5 bar	
Auxiliary instruments for sample preparation	
Laboratory balance (resolution ± 0.01 g)	

Reagents

- Deionized water (ISO 3696 Type II)

Sample preparation

Samples with small particle size

Samples in powder or particle form (max. edge length 2 mm) can be used directly.

Foils or other larger products

PVC foils and other solid products have to be ground or cut into pieces. The edge length of the ground particles should be maximum 2 mm.

Analysis

Preparation of the PVC Thermomat

The heating block is heated up to the respective temperature.

Preparation of the measuring vessel

The measuring vessel is filled with 60 mL deionized water and placed on the PVC Thermomat together with the measuring vessel cover.

Preparation of the reaction vessel

For each determination, a new reaction vessel is used. To remove particles (e.g., from the cardboard box) the reaction vessel is air-cleaned inside and outside by a sharp stream of nitrogen. Then, the sample is weighed directly into the reaction vessel. The samples should only cover the bottom of the reaction vessel. A sample size of 0.5 ± 0.05 g is used. The reaction vessel is closed with a reaction vessel cover assembled with a gas inlet tube. Ensure that the gas inlet tube always ends over the surface of the plastic particles.

Determination

Before the determination can be started, the temperature of the heating block has to be stable. The two tubings between PVC Thermomat and reaction vessel and between reaction vessel and measuring vessel are connected. Then, the reaction vessel is placed in the heating block and the measurement is started immediately.

Parameters

Sample size	0.5 ± 0.05 g
Measuring solution	60 mL
Temperature	170 … 210 °C
Gas flow	7 L/h
Evaluation	Stability time
Conductivity change	50 µS/cm

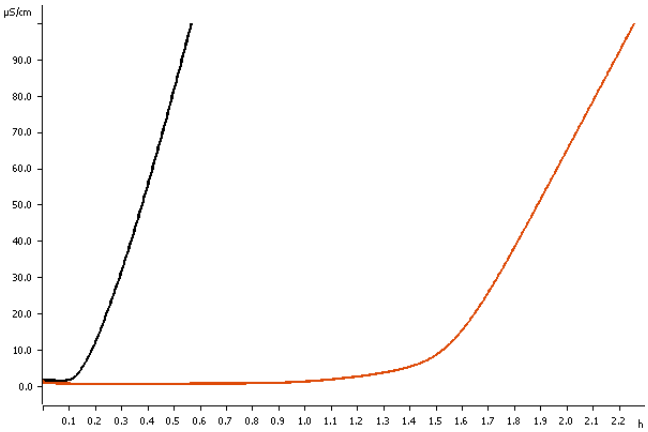
The measuring temperature depends on the thermostability of the sample. Usually temperatures between 170 and 210 °C are appropriate. 50 to 220 °C are possible. Most tests are carried out between 180 °C and 200 °C (lower stability – lower temperature). The rule of thumb is: a temperature increase of 10 °C lowers the stability time by a factor of two.

Results

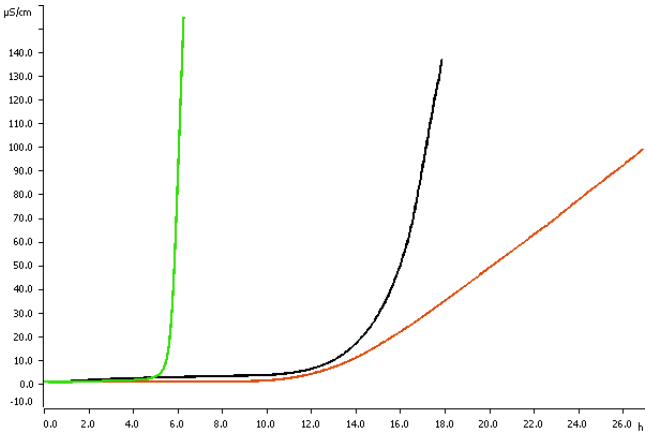
Sample	Temperature/°C	Stability time/h
PVC resin (pure)	180	0.39
PVC resin (blended)	180	1.90
PVC pellets 1	180	5.87
PVC pellets 2	180	16.0
PVC pellets 3	180	20.28
PVC foil	180	1.79

Examples

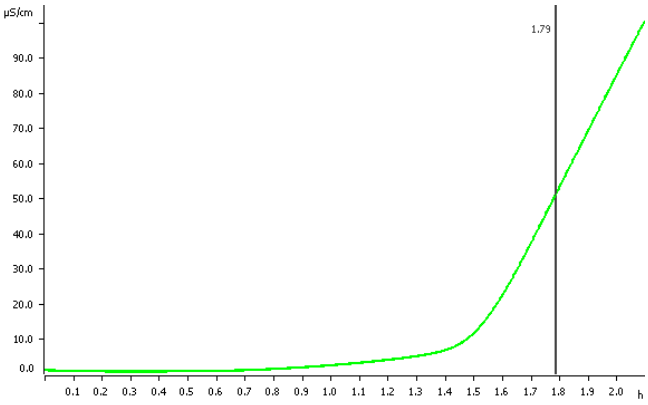
PVC resin pure (black) and blended (red)



PVC pellets (3 types)



PVC foil



Comments

- Any kind of contamination, particles, or scratches in the glass of the reaction vessel can catalyze reactions and thereby affect the result. Contaminations may deteriorate the reproducibility of the results or cause incorrect results. Therefore, it is recommended to use a new reaction vessel and gas inlet tube for each

determination and blow off particles by a sharp stream of nitrogen.

- The temperature is the most crucial parameter in the determination of the thermostability. Especially if results from different instruments should be compared it is essential to determine the «temperature correction» value correctly. For more information about the «temperature correction» and its determination, see the StabNet software tutorial and the 895 Professional PVC Thermomat instrument manual.
- The gas flow is relevant for the reliable transfer of the reaction products from the reaction vessel to the measuring vessel. Beyond that, there is no influence of the gas flow on the result as long as the cooling effect is compensated by the correct adjustment of the «temperature correction».
- The sample size can be a critical parameter for the direct determination of solid PVC samples. Since the air stream cannot mix the sample, no homogeneous temperature can be guaranteed in a larger volume. Hence, a small sample size that just covers the bottom of the reaction vessel should be preferred.
- Since for the stability time an absolute value for the conductivity has to be measured, the cell constant of each cell has to be determined. For details, refer to the instructions in the StabNet Tutorial.

References

- StabNet, Tutorial
- 895 Professional PVC Thermomat, Manual
- Hamsen K.-H., Jaksch R., Kolb T., Schäfer J., Marks D., Sittel J., Zahn A.: Comparison of Thermal Stability Measurement Processes for PVC, Kunststoffe international 2003/09 35-39.
- DIN 53381-1; Prüfung von Kunststoffen – Bestimmung der Thermostabilität von Polyvinylchlorid (PVC) – Dehydrochlorierungsverfahren
- ISO 182-3; Plastics – Determination of the tendency of compounds and products based on vinyl chloride homopolymers and copolymers to evolve hydrogen chloride and any other acidic products at elevated temperatures – Part 3: Conductometric method