

DETERMINATION OF CHLORINE CONTENT OF HIGH FREQUENCY ADHESIVES**Application**

This procedure is used to determine the percentage of chlorine in PVC copolymer high frequency adhesives based on solid content.

The material under test is volatilized by heating in an oxygen stream and totally burnt in an oxyhydrogen flame. The combustion products are absorbed in a receiver.

Apparatus and Reagents Required**Combustion Apparatus**

For solid substances. Dr. Wickbold system (see sketch) Type 3.

Source: Heraeus - Schott Quarzschmelze GmbH
6450 Hanau
Germany

pH Meter

With glass electrode suitable for pH measurement. For the potentiometric titration a combined comparison electrode (Ag-AgCl) is required. (Reference solution KNO₃ saturated.)

Magnetic Agitator and Stirring Rods**Measuring Flasks**

100 mL

Beakers

250 mL

Burettes

25 mL

Fish Tail Burner**Vacuum Pump****Analytical Balance****Quartz Combustion Boats**

Date	Action	Revisions
2000 08 30	Revised	Editorial – no technical change A. Cockman
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Sodium Hydroxide Solution (NaOH)

1 Normal - for absorption.

Sulphuric Acid (H₂SO₄) 20 % by Volume

Prepared from 95 - 97 % sulphuric acid.

Silver Nitrate Solution (AgNO₃)

0.02 Normal.

Potassium Pyrosulphate (K₂S₂O₇)

Conditioning and Test Conditions

All test values indicated herein are based on material conditioned in a controlled atmosphere of 23 +/- 2 °C and 50 +/- 5 % relative humidity for not less than 24 h prior to testing and tested under the same conditions unless otherwise specified.

Procedure

1. Weigh a specimen of adhesive in a quartz boat to the nearest .1 mgf. The weight of the specimen used is based on the chlorine content of the adhesive and must be chosen as follows:

Approximately	25 mgf at 30 - 60 % Cl by weight
Approximately	50 mgf at 10 - 30 % Cl by weight
Minimum	100 mgf at 1 - 10 % Cl by weight
	0.5 - 3 g at below 1 % Cl by weight

If inorganic substances are present in the adhesive, potassium pyrosulphate must be added to the sample to act as a flux.

2. The quartz boat is inserted in the gasifying chamber (A), the chamber locked with the ground glass joint (E) and the cock (F) opened.
3. A 100 mL measuring flask is connected to the combustion apparatus. Open the cock between the combustion apparatus and the measuring flask.
4. Set under a vacuum of 150 - 300 mm mercury.
5. After opening of the cooling water valve, the burner (B) is ignited. The oxygen flow rate should be 1000 l/h and hydrogen flow rate should be 400 l/h. The length of the oxyhydrogen flame is to be adjusted to approximately 10 cm.
6. The burner (B) is inserted into the ground glass joint and the absorption solution (NaOH) valve opened. The drop sequence is adjusted to 3 - 4 drops/s for chlorine traces and to 7 - 8 drops/s for a higher chlorine content.