

A New Perspective on Hydrogen Chloride Scavenging at High Temperatures for Reducing Smoke Acidity of PVC Cables in Fires.

V: Comparison between EN 60754-1 and EN 60754-2

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1) Materials

Table S1 shows the commercial additives in Tables 1-4 of the paper, S2 the test apparatuses, and S3 the test methods.

Table S1

Inovyn 271 PC: PVC S K70 produced by Inovyn. https://www.ineos.com/businesses/inovyn/
Diplast N: Di Iso Nonyl Phthalate produced by Polynt S.p.A.. https://www.polynt.com/it/
Reaflex EP/6: Epoxidized Soy Bean Oil (ESBO) in the product portfolio of Reagens. https://www.reagens-group.com/
Arenox A10: Pentaerythritol tetrakis(3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate) in the product portfolio of Reagens. https://www.reagens-group.com/
RI004: Antimony trioxide from Quimialmel: https://quimialmel.it/
Riochim: Ground Calcium Carbonate produced by Umbriafiller: https://umbriafiller.com/
Winnofil S: Precipitated Calcium Carbonate in the product portfolio of Imerys: https://www.imerys.com/
AS-1B, AS-6B, and AS-0B: Acid scavengers at high temperatures acting in the condensed phase from Reagens S.p.A. https://www.reagens-group.com/
Aparyl 40 CD: Synthetic aluminum tri hydroxide, produced by Nabaltec. https://nabaltec.de/en/
Ecopypren 3.5: Brucite in the product portfolio of Europiren. https://europiren.com/it/catalog/ecopypren/
Kisuma 5 A: Synthetic magnesium hydroxide produced by Kisuma. https://www.kisuma.com/
Cabosil H5: Fumed silica produced by Cabot. https://www.cabotcorp.com/
RPK B-NT/8014: anti-pinking additive produced by Reagens S.p.A.. https://www.reagens-group.com/

Table S2

Test apparatus	Producer	Model	Additional Info's
Plasticorder	Brabender	Plastograph EC	50 CC, chamber, 160 °C per 10 minutes.
Halogen Acid Gas test apparatus	SA Associates	Standard model	Porcelain combustion boats.
Multimeter	Mettler Toledo	S213 standard kit	
Conductivity electrode	Mettler Toledo	S213 standard kit	Reference thermocouple adjusting temperature fluctuation.
pH electrode	Mettler Toledo	S213 standard kit	Reference thermocouple adjusting temperature fluctuation.
Ion Exchange Deionizer	Culligan Pharma System 20		

Table S3

Technical standard	Measurement	Temperature	Note
EN 60754-2	pH and conductivity	Isothermal at 950 °C +/- 5 °C	The general method, according to the 2014 version.
Internal method 2	pH and conductivity	Isothermal at 500 °C +/- 5 °C	The general method, according to the 2014 version.
Internal method 3	pH and conductivity	23°C to 800 °C +/- 10 °C in 40 min +/- 5 min 800 °C +/- 10 °C per 20 min +/- 1 min	EN 60754-2 carried out with the thermal profile of EN 60754-1

2) Sample preparation

PVC compounds in Tables 1-4 of the article have been prepared by weighing the stabilizers' ingredients on a 0.001 g readability balance. PVC, plasticizers, fillers, flame retardants, and acid scavengers have been weighed on a 0.1 g readability balance. All solid ingredients have been introduced into a laboratory turbo mixer. Liquids such as ESBO and DINP have been added when the temperature reaches 80°C, and stabilizers at 90°C. The mixing has been stopped at 105°C, and the dry blend dropped into PE bags and stored for 24 hours at 23°C to allow for proper "maturation" of the dry blends. 60 g of the dry blend have been processed in a Plasticorder Brabender (the model in Table S2) for 10 minutes at 160°C, 30 rpm. Test specimens for EN 60754-2, internal methods 2 and 3, are derived directly from the kneaders.

3) Measurements of the main properties

3.1) pH and conductivity

The data have been reported in Tables 7-15 of the article.

3.3.1) EN 60754-2 procedures

The tube furnace (SA Associates, Table S2) has a touch screen temperature controller through which the heating regime of EN 60754-2 has been selected (isothermal at 950 °C). The final temperature has been checked and adjusted with a calibrated external thermocouple. The airflow has been set at 300 ml/min +/- 10 ml/min, according to the quartz tube dimensions, as EN 60754-2 requested. Before the first run, a preliminary bubbling test in the devices was done to verify that all connections were well-tightened and that no HCl leak could affect the measurements. The kneaders have been conditioned for 24 h at 23°C. Slices with similar dimensions for all runs (approximately 1 mm x 1 mm) have been derived from them. The slices have been weighed into the combustion boat and arranged in the tube furnace when the temperature was stable at 950 +/- 5 °C.

Smokes have been collected in two bubbling devices containing double deionized water produced by Ion Exchange Deionizer (Table S2) of the quality required by EN 60754-2 for 30 minutes. The two quotes have been collected in a 1 L volumetric flask, ensuring all connectors and bubbling devices were well-cleaned to recover as much HCl as possible.

pH and conductivity have been measured simultaneously by inserting the electrodes in two different vessels. pH and conductivity measures are taken at 25 °C +/- 1 with the following procedure: the multimeter has been calibrated with standard solutions before each measurement: pH at two points (4.01 and 7.00) and conductivity at 1 point at 141.3 µS/mm. The solutions closest to the measured value have been

chosen as correction standards, and the measurements are corrected accordingly through a correction factor. pH and conductivity electrodes have a reference thermocouple that adjusts the temperature fluctuation. pH and conductivity are measured, and two replicates give the mean value, standard deviation (SD), and coefficient of variation (CV).

3.3.2) Internal method 2 procedures

Internal method 2 follows the procedures described in paragraph 3.3.1, setting the test temperature at 500 °C +/- 5 °C.

3.3.3) Internal method 3 procedures

The tube furnace (SA Associates, Table S2) has a touch screen temperature controller through which the heating regime of EN 60754-1 ² has been selected (Table S3). The check of the temperature regime has been done following this procedure. An empty combustion boat is introduced in the tube furnace through the sample carrier. The airflow is set between 290 and 310 ml/min according to the quartz tube geometry. The thermocouple is inserted in the center of the tube furnace, the initial ramp is chosen, the heater is started, and the time is followed with a stopwatch. The ramp is chosen for reaching 800 °C +/- 10 °C in 40 min +/- 5 min and for keeping an isothermal condition of 800 °C +/- 10 °C per 20 min +/- 1 min. The heating rate is adjusted accordingly if temperatures and times exceed the above ranges. The conductivity of the water in the bubblers is checked to verify the possibility of contamination from previous tests.

After determining the heating regime and the cleaning status of the quartz tube, the sample is weight in the combustion boat (1.000 g +/- 0.001 g of material) and introduced in the tube furnace at room temperature through the sample carrier. The heater is switched on, and the stopwatch checks the ramp. After 1 hour, the connectors are opened, and the water from the bubbling devices and washing procedures is collected in a 1 L volumetric flask filled to the mark. pH and conductivity are measured (as described in paragraph 3.3.1), and two replicates give mean value, standard deviation (SD), and coefficient of variation (CV).

References

1. EN 60754-2:2014/A1:2020; Test on Gases Evolved during Combustion of Materials from Cables—Part 2: Determination of Acidity (by pH Measurement) and Conductivity. CENELEC: Brussels, Belgium, 2020. Available online: <https://my.ceinorme.it/home.html> (accessed on 10 June 2023).
2. EN 60754-1; Test on Gases Evolved during Combustion of Materials from Cables—Part 1: Determination of the Halogen Acid Gas Content. CENELEC: Brussels, Belgium, 2014. Available online: <https://my.ceinorme.it/home.html> (accessed on 1 August 2022).