Carbon black used in rubber products—Specification

Part 5: Fast extrusion furnace carbon black (FEF)—N550 Grade
TECHNICAL COMMITTEE REPRESENTATION

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Part 5: Fast extrusion furnace carbon black (FEF)— N550 Grade
Foreword

This Part of KS 2053 has been prepared by the Technical Committee on Rubber and rubber products under the direction of the Plastics and ceramics Industry Standards Committee, and it is in accordance with the procedures of the Bureau.

This Part of KS 2053 gives requirements and test methods for fast extrusion furnace carbon black, N550 type used in rubber products. It has been given the nomenclature N550 by the American Society for Testing and Materials (ASTM).

The first character in the nomenclature system for rubber-grade carbon blacks is a letter indicating the effect of the carbon black on the cure rate of a typical rubber compound containing the black. The letter “N” is used to indicate a normal curing rate typical of furnace blacks that have received no special modification to alter their influence on the rate of cure of rubber.

The letter “S” is used for channel blacks or for furnace blacks that have been modified to effectively reduce the curing rate of rubber. Channel blacks characteristically impart a slower rate of cure to rubber compounds. Thus, the letter “S” designates a slow cure rate. Blacks may vary considerably in “curing rate” within each of the two letter classifications.

The second character in the nomenclature system is a digit to designate the average surface area of the carbon black as measured by nitrogen surface area. The third and fourth characters in this system are arbitrarily assigned digits.

The major portion of carbon black consumed by the rubber industry is used to improve the physical properties and life expectancy of rubber products.

During the development of this standard, reference was made to the following documents:


IS 7498:1985, Indian Standard Methods of sampling and test for carbon black


Acknowledgement is hereby made for the assistance received from these sources.
Carbon black used in rubber products—Specification

Part 5: Fast extrusion furnace carbon black (FEF)—N550 Grade

1 Scope

This Part of KS 2053 prescribes the requirements and test methods for fast extrusion furnace carbon black, N550 grade used in rubber products.

2 Normative references

The following Standards contains provisions which, through reference in this text, constitute provisions of this Kenya Standard. For undated references, the latest edition of the normative document referred to applies.

KS 06-877 Method of test for rubber compounding ingredients - Carbon black - Determination of iodine absorption number titrimetric method

KS ISO 4656-1 Rubber compounding ingredients - Carbon black - Determination of dibutylphthalate absorption - Part 1: Method using absorptometer

KS ISO 1306 Rubber compounding ingredients - Carbon black (pelletized) - Determination of pour density

KS ISO 1437 Rubber compounding ingredients - Carbon black - Determination of sieve residue.

KS ISO 1126 Rubber compounding ingredients - Carbon black - Determination of loss on heating.

KS ISO 1125 Rubber compounding ingredients - Carbon black - Determination of ash.

KS ISO 1435 Rubber compounding ingredients - Carbon black (pelletized) - Determination of fines content.

KS ISO 1124 Rubber compounding ingredients - Carbon black - Shipment sampling procedures

KS 06-898 Specification for determination of tensile stress-strain properties of vulcanized rubber

3 Requirements

3.1 Grade

The carbon black shall be of the N550 grade.

3.2 Material

The carbon black shall be free from foreign matter such as wood, metal, fibres and any visible impurities when examined visually.

3.3 Pelletization

The carbon black shall be delivered in pelletized form. Pellet hardness shall be controlled to such a degree that satisfactory dispersion is obtained on its being compounded using standard mixing equipments as desired by the purchaser.
3.4 Compounding

When compounded and tested as specified Annex A, the difference in tensile strength and 300 percent modulus properties of vulcanizates containing the carbon black as compared to Industry Reference Black (IRB) No.7 shall be as specified in Table 1.

Table 1 — Difference in Physical Properties of Vulcanizates Containing N550 carbon black from IRB No.7

<table>
<thead>
<tr>
<th>SI No.</th>
<th>Carbon black type</th>
<th>Cure Condition</th>
<th>Tensile Strength, (Min MPa*)</th>
<th>300 Percent Modulus (MPa*)</th>
</tr>
</thead>
<tbody>
<tr>
<td>i)</td>
<td>N550</td>
<td>30 min at 145 °C</td>
<td>-5.71</td>
<td>-2.95 to +0.05</td>
</tr>
</tbody>
</table>

*1 MPa = 10.2 kgf/cm² approx.

3.5 Physical and chemical characteristics

The carbon black shall comply with the requirements given in Table 2.

Table 2 — Physical and chemical characteristics

<table>
<thead>
<tr>
<th>SL NO.</th>
<th>Characteristics</th>
<th>Requirement</th>
<th>Test Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>i)</td>
<td>Iodine adsorption, mg of iodine/g of carbon black</td>
<td>38 to 48</td>
<td>KS 06-877</td>
</tr>
<tr>
<td>ii)</td>
<td>Dibutyl phthalate absorption, ml/100 g of carbon black</td>
<td>115 to 125</td>
<td>KS ISO 4656-1</td>
</tr>
<tr>
<td>iii)</td>
<td>Pour density, g/l</td>
<td>320 to 380</td>
<td>KS ISO 1306</td>
</tr>
<tr>
<td>iv)</td>
<td>Sieve residue, % m/m, max</td>
<td>0.100 0</td>
<td>KS ISO 1437</td>
</tr>
<tr>
<td>v)</td>
<td>Loss on heating, % m/m, max</td>
<td>1.5</td>
<td>KS ISO 1126</td>
</tr>
<tr>
<td>vi)</td>
<td>Ash content, % m/m, max</td>
<td>0.75</td>
<td>KS ISO 1125</td>
</tr>
<tr>
<td>vii)</td>
<td>Staining tendency</td>
<td>No staining</td>
<td>Annex B</td>
</tr>
<tr>
<td>viii)</td>
<td>Fines content, % m/m, max</td>
<td>15.0</td>
<td>KS ISO 1435</td>
</tr>
<tr>
<td>ix)</td>
<td>Discoloration of toluene, percent transmission, Min</td>
<td>80</td>
<td>Annex C</td>
</tr>
</tbody>
</table>
4. Packing and Marking

4.1 Packing

The carbon black shall be supplied in paper bags or any suitable package that shall ensure protection of the carbon black from damage or deterioration during transportation, storage and use. The bags shall be shaped to facilitate stacking of pallets.

4.2 Marking

Each package shall be clearly and indelibly marked with the following:

a) Name of the material;

b) Grade;

c) Colour of the material;

d) Manufacturer’s name and address (with the country of origin);

e) Net mass of the material;

f) Batch or lot number;

g) Month and year of manufacture.

5 Sampling

The sampling of carbon black shall be done in accordance with KS ISO 1124

5.1 Criteria for Conformity

The lot shall be declared as conforming to the requirements of the specification if all the test results of each of the individual samples satisfy the corresponding requirements.

Annex A
Schedule for compounding and testing for physical evaluation of carbon black

A-1  General

This procedure involves the incorporation of the black to be tested in rubber along with necessary auxiliary agents, to permit vulcanisation, followed by testing. Along with each test black, a corresponding stock containing the Industry Reference Black(IRB) No. 7 is included. The difference between the properties obtained on the reference black is simply a device to cancel the inevitable variations in test results which are due to minor variations between laboratories in equipment, materials, procedure and ambient conditions.

A-2  Standard Formula

The standard formula for testing carbon black is given below.

<table>
<thead>
<tr>
<th>Material</th>
<th>Parts by mass</th>
</tr>
</thead>
<tbody>
<tr>
<td>Natural rubber grade 5 (conforming to KS ISO 2000)</td>
<td>100</td>
</tr>
<tr>
<td>Zinc Oxide (for the rubber industry)</td>
<td>5</td>
</tr>
<tr>
<td>Stearic acid (for the rubber industry)</td>
<td>3</td>
</tr>
<tr>
<td>Dibenzothiazyl disulphide (for the rubber industry)</td>
<td>0.6</td>
</tr>
<tr>
<td>Sulphur (for the rubber industry)</td>
<td>2.5</td>
</tr>
<tr>
<td>Carbon black N550</td>
<td>50</td>
</tr>
</tbody>
</table>

A-3  Mixing Method

A-3.1  The method of mixing is given in A-3.1.1 to A-3.1.11.

A-3.1.1 Use a two roll laboratory mill having 150 mm outside diameter and 250 to 280 mm working distance between the guides. The roll speed ratio should be 1 to 1.4. Adjust and maintain roll temperature at 70 ± 5° C and set mill opening at 1.4 mm.

A-3.1.2 The carbon black shall be conditioned before weighing by heating in an oven at 100 to 110 ºC for 1 hour.

A-3.1.3 weigh the ingredients for a batch size which is 4 times of the parts by mass in given in A-2

A-3.1.4 Add rubber and band on front roll, make two 3/4 cuts from each side on the mill (time 2.0 minutes).

A-3.1.5 Add stearic acid and cut once each way (time 2.5 minutes).

A-3.1.6 Add zinc oxide, sulphur and accelerator and cut twice each way (time 2 minutes).

A-3.1.7 Add carbon black. Open mill gradually to maintain approximately constant bank. Cut three times each way after all black is incorporated (time 7.5 minutes). Add carbon from the mill pan until all the black is incorporated.

Note: Along with each test black, a corresponding stock containing the Industry Reference Black(IRB) No. 7 is included. The difference between the properties obtained on the reference black is simply a device to cancel the inevitable variations in test results which are due to minor variations between laboratories in equipment, materials, procedure and ambient conditions.
A-3.1.8 Cut stock, roll and weigh (time 1 minute). If the mass of mixed batch is beyond the tolerance of ± 0.6 percent of the total mass of all ingredients, reject the batch.

A-3.1.9 Pass end-wise six times at 0.8 mm opening, the sheet off at 2.2 mm finished gauge (time 2.5 minutes), cool on metal table top, and prepare specimen for cure.

A-3.1.10 Total mixing time is about 17.5 minutes.

A-3.1.11 Condition the stock for 1 to 24 hours at 27± 2 °C and cut out suitable slabs for vulcanisation.

A-4 Vulcanization

The test pieces are vulcanised for 30 minutes at 145 °C in a standard 4-cavity mould which gives sheets of dimensions 150 x 150 x 2 mm. The curing press shall be capable of exerting a minimum pressure of 3.5 MN/m $^2$ (approx 35kg/cm$^2$) on the cavity areas of the mould during vulcanisation. After vulcanisation, the sheets shall be cooled immediately in water. The time interval between curing and testing shall be a minimum of 1 hour and maximum of 72 hours.

A-5 Testing

A.5.1 The vulcanised sheets prepared from the test sample and those prepared from a corresponding stock containing the Industry Reference Black(IRB) No. 7 are tested for 300 percent modulus and tensile strength in accordance with KS 06-898*.

A.5.2 Calculation for Difference in Physical Properties of Vulcanizates Containing N550 carbon black from IRB No.7

A.5.2.1 Calculation for Difference in 300 percent modulus

Difference in 300 percent modulus = $M_1 - M_2$

Where

$M_1 = 300$ percent modulus of the sheet containing carbon black test sample

$M_2 = 300$ percent modulus of the sheet containing Industry Reference Black(IRB) No.7.

A.5.2.2 Calculation for Difference in tensile strength

Difference in tensile strength = $T_1 - T_2$

$T_1 = $ tensile strength of the sheet containing carbon black test sample

$T_2 = $ tensile strength of the sheet containing Industry Reference Black(IRB) No.7.

Annex B
Staining Test

B.1 Principle
In this test the colour of toluene extract of carbon black is visually compared with that of pure toluene. It is a measure of the staining tendency.

B.2 Apparatus - two matched Nessler cylinders.

B.3 Reagent - toluene.

B.4 Procedure

B.4.1 Into a Nessler cylinder, weigh 1 g of sample to the nearest 0.01 g. Add 10 ml of toluene and shake for 1 minute. Allow to settle and compare the colour of the supernatant layer with that of toluene alone in the other Nessler cylinder, by viewing against a white background through the side.

B.4.2 The sample shall be deemed to have no staining tendency if the supernatant liquid is of the same colour as that of the pure toluene.
To determine the degree of discolouration of toluene by carbon black by means of a spectrophotometer. This method is not applicable to high extract thermal type blacks.

**C.2 Apparatus**

**C.2.1 Spectrophotometer** - 20 nm maximum spectral band pass capable of measuring transmittance in the 425 ± 5 nm range.

**C.2.2 Absorption Cells** - With an optical light path of 10 mm (light path of the toluene and not the outside of the cell) for the spectrophotometer.

**C.2.3 Analytical Balance** - Sensitivity 0.01 mg.

**C.2.4 Oven** - Gravity convection type capable of temperature regulation of ± 1°C at 105°C.

**C.2.5 Filter Paper** - Whatman No. 41 or equivalent, diameter 150 mm.

**C.2.6 Mechanical Shaker** - Capable of 240 strokes/minute.

**C.2.7 Mortar and Pestle**

**C.2.8 Glass Filtering funnel**

**C.2.9 Beaker**

**C.2.10 Erlenmayer Flask**

**C.3 Reagent**

**C.3.1 Toluene** - Analytical grade,

**C.4 Procedure**

**C.4.1 Standardization of Apparatus** - Clean the absorption cell with the lens tissue. Allow the spectrophotometer to warm up at least 10 minutes before standardization. Rinse the cell twice with clean toluene, wipe the outside surface with lens tissue. Adjust the transmission value to 100 percent on the spectrophotometer, using the wave length of 425 nm.

**C.4.2 Crush pelleted samples using mortar and pestle or equivalent. Dry an adequate amount of crushed carbon black sample at 105°C for 60 minutes using the oven.**

NOTE - An infra-red lamp must not be used for drying sample as it could vaporize some of the extractible materials.

**C.4.3 Allow sample to cool to room temperature in a closed container. Weigh 2.00 ± 0.01 g of sample and transfer to a 125-ems Erlenmeyer flask. Add 20 cm³ of toluene to the sample in the flask and stopper. Without delay, begin shaking the mixture in the mechanical shaker for 60 seconds. Immediately pour as much of the mixture as possible into the glass funnel with filter paper which has previously been prepared and inserted into a 125-ems Erlenmeyer flask. Check standardization of spectrophotometer at 425 nm. Using the same cell as used to standardize before testing, rinse the cell twice with the same filtrate to be tested.**

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Fill the absorption cell with the filtrate and determine the percentage transmission on the spectrophotometer at 425 nm. If necessary larger quantities of sample and toluene may be used, keeping the ratio 10 ml of toluene per gram of back unchanged, to a maximum of 5 g/50 ml of toluene.