METHODS OF TESTING VULCANIZED RUBBER

PART B14

DETERMINATION OF CARBON BLACK

B.S. 903 : Part B14 : 1964

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This standard makes reference to the following British Standards:

B.S. 148.	Insulating oil for transformers and switchgear.
B.S. 410.	Test sieves.
B.S. 1752.	Laboratory sintered or fritted filters.

The following B.S.I. references relate to the work on this standard:
Committee references, RUC/10 and RUC/10/5
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BRITISH STANDARD

METHODS OF TESTING VULCANIZED RUBBER

Part B14. Determination of Carbon Black

FOREWORD

This British Standard Method has been prepared under the authority of the Rubber Industry Standards Committee, and replaces Part 7 of B.S. 903 : 1950.

In the revision of B.S. 903 the methods are being published as separate parts, and chemical methods are distinguished by the letter B.

SECTION 1. INTRODUCTION

The method of determining carbon black in natural rubber vulcanizates which has appeared in previous editions of B.S. 903 has been used satisfactorily with natural rubber over many years, but it is not applicable to rubbers more resistant to acid degradation. The method given in Section Two included here is thought to be of more general application. It is, therefore, recommended as a general method to supersede the older method of acid degradation even for natural rubber vulcanizates. However, until experience with the new method is more widespread, the older method has been included in Section Four, but its application is normally limited to natural rubber.

The second method, given in Section Three, depends on clean pyrolysis of the polymer component of the vulcanizate and is generally applicable only to hydrocarbon rubbers, although acrylate rubbers also pyrolyse without carbonizing. It is included here because it can conveniently be run as part of a general method in which the rubber as well as carbon black is to be determined provided calcium carbonate is absent. If determinations of both polymer and carbon black are required then the preferred method is the pyrolysis method providing that the polymer pyrolyses substantially without carbonization.

All three methods are semi-micro in scale and in framing them it has been assumed that the analyst is familiar with the techniques of semi-micro analysis.

SECTION 2. DISSOLUTION METHOD

2.1. Summary of method. The rubber is degraded with t-butyl hydroperoxide in p-dichlorobenzene adding, in the case of acrylonitrile copolymers, a few millilitres of nitrobenzene to hasten the process. Mineral oil is added to prevent the liberated carbon black from adhering to the glass vessels. After the polymer has decomposed the solution is diluted with a suitable solvent depending on the nature of the polymer. The insoluble material is collected on a kieselguhr filter pad prepared in a filter tube, and treated with nitric acid to remove soluble inorganic substances and partly degraded polymer. After heating in nitrogen at about 340°C to remove most of the substances absorbed on the black, the filter tube is cooled and weighed.

If acid insoluble silicates are absent the carbon black is burnt off in a muffle furnace and the filter tube is weighed again to give the amount of carbon black by difference.

If acid insoluble silicates are present the filter tube is heated again under nitrogen but at a temperature of 800–900°C and the liberated water is weighed after absorption in magnesium perchlorate. The small amount of water in the kieselguhr is also corrected for by a blank determination. Finally the carbon black is burnt off in a muffle furnace and the filter tube weighed again to give the total loss by difference. A correction in respect of the water content of the silicates and kieselguhr is applied and thus the carbon black content is obtained.

2.2. Reagents. All reagents shall be of recognized analytical reagent quality unless otherwise specified.

p-Dichlorobenzene
Nitrobenzene
t-Butyl hydroperoxide
Mineral oil in accordance with B.S. 148*

Diluting solvent:

Acetone for acrylate rubber

Benzene for polyisoprene (natural or synthetic), polybutadiene, styrene butadiene rubber, acrylonitrile butadiene rubber or polychloroprene

Light petroleum, boiling range 40–60°C, for butyl rubber

Kieselguhr
Silica wool
Nitric acid, dilute: 3 vol concentrated nitric acid ($d = 1{\cdot}42$) diluted with 1 vol water

Acetone
Nitrogen, dry and free from oxygen†
Magnesium perchlorate, retained on a 710 micron B.S. test sieve and passing a 1200 micron B.S. test sieve‡ (14–22 mesh).

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* B.S. 148, ' Insulating oil for transformers and switchgear '. ' DIALA B ' marketed by Shell Refining and Marketing Ltd. has been shown to be satisfactory.

† Nitrogen sold commercially as free from oxygen may require further purification.

‡ B.S. 410, ' Test sieves '.

2.3. Apparatus.

Dissolution assembly consisting of:

a. Flat-bottomed wide-necked 150 ml flask, with lip and ground glass joint.

b. Glass column about 25 cm long and about 3 cm in diameter with ground joints.

c. PTFE sleeve interposed between *a* and *b*.

d. Short Liebig condenser with ground joint.

Silica filter tube, consisting of a 10 mm sintered silica filter disk porosity Grade 3 (B.S. 1752*) fused into one end of a silica tube 10 cm long. The tube may be widened to 15 mm at its upper end to facilitate the transfer of solutions.

Electric tube furnace with means for advancing and withdrawing the filter tube.

Muffle furnace.

Combined electric hot plate and magnetic stirrer. The glass-covered stirrer bar should be short enough to lie flat on the base of the dissolution flask.

Absorption tube, packed with magnesium perchlorate and closed with taps at both ends.

2.4. Procedure. Cut the thinly sheeted sample into narrow strips and weigh about 0·1 g to the nearest 0·0002 g.

Place 20 g p-dichlorobenzene in the flask and insert a small glass-covered iron stirrer bar for magnetic stirring. Cover the ground glass joint of the glass column with the PTFE sleeve, fit to the flask, and fit the Liebig condenser to the column. Mount this assembly on the hot plate. Heat the flask until the p-dichlorobenzene is melted, add the test portion (and 5 ml of nitrobenzene if the polymer is acrylontrile butadiene rubber), and boil gently for half to one hour, stirring slowly.

Add, over a period of about 10 minutes, 5 ml t-butyl hydroperoxide down the condenser, continue the boiling for a further two hours, then add 5 ml of mineral oil and continue boiling until decomposition of the polymer is complete. Cool the flask to about 60°C and dilute with 30 ml of the appropriate diluting solvent.

Place 0·15 g of kieselguhr into the filter tube, followed by a 5 mm layer of silica wool. Condition the tube by washing with hot dilute nitric acid, followed by water, and then igniting in the muffle furnace at 800–900°C for 20–30 min. Fit the filter tube into the neck of a Buchner flask and half fill the tube with the

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* B.S. 1752, ' Laboratory sintered or fritted filters '.

diluting solvent. Apply gentle suction and slowly pour the contents of the dissolution flask into the tube. Wash the flask with 30–50 ml of the diluting solvent and transfer to the filter tube without allowing the tube to become dry. Finally, wash the flask with acetone into the filter tube.

Empty the Buchner flask, wash the dissolution flask with portions of hot dilute nitric acid totalling 20 ml and add to the filter tube. Then wash the filter tube with a further 20 ml of hot dilute nitric acid. Wash the dissolution flask with three 5 ml portions of water (using a policeman to dislodge the black), and pass through the filter tube. Again empty the Buchner flask and then wash the dissolution flask with acetone passing the washings through the filter tube and removing the last traces of black with the policeman. Dry the filter tube in an oven at 100–140°C.

Place the dry filter tube, open end first, into the end of the combustion tube of the furnace heated to about 340°C, close the tube with an entry fitting and pass oxygen-free nitrogen through it at approximately 200 ml/min. After 5 min reduce the rate of gas flow to approximately 100 ml/min, then push the filter tube into the hot zone and heat for 30 min. Return the filter tube to the end of the combustion tube*, cool with nitrogen still passing, remove from the combustion tube, complete the cooling in a desiccator and weigh at room temperature (W_1 grammes).

2.4.1. *Acid insoluble silicates absent.* Place the filter tube in the muffle furnace at 800–900°C and ignite until all traces of carbon are removed. Cool in a desiccator and weigh (W_2 grammes).

Calculate the carbon black content from the formula:

$$\text{Carbon black, per cent} = \frac{(W_1 - W_2)}{W} \times 100$$

where W is the weight of the test portion in grammes.

2.4.2. *Acid insoluble silicates present.* Replace the combustion tube of the furnace with a silica tube having a reduced exit to which an absorption tube may be attached.

Heat the tube to 800–900°C and adjust the flow of nitrogen to 20–30 ml/min. Attach the absorption tube packed with magnesium perchlorate, pass nitrogen through the system for 15 min, detach the absorption tube, immediately close the taps and weigh the tube when at room temperature. This is the conditioning process for the absorption tube.

With the nitrogen still flowing at a rate of 20–30 ml/min remove the entry fitting of the combustion tube, insert the filter tube and connect the absorption tube to the exit of the furnace. Open the taps of the absorption tube and immedi-

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* The filter tube may be brought back to its original position by means of a hook on the end of a silica rod engaging a loop of platinum wire attached to the filter tube.

ately close the combustion tube with the entry fitting. Push the filter tube into the hot zone (800–900°C) and heat for 20–30 min. Disconnect the absorption tube, immediately close the taps and weigh when the tube is at room temperature. The increase in weight is due to water in the silicates and the kieselguhr (C_1 grammes).

Withdraw the filter tube and cool in air to room temperature. Insert the filter tube into the furnace again and reconnect the conditioned absorption tube to the exit of the combustion tube. Open the taps of the absorption tube and immediately close the combustion tube with the entry fitting. Push the filter tube into the hot zone (800–900°C) and heat for 20–30 min. Disconnect the absorption tube, immediately close the taps and weigh when the tube is at room temperature. The second increase in weight is due to water absorbed by the kieselguhr (C_2 grammes) when standing at room temperature. The weight of water derived from the silicates is ($C_1 - C_2$) grammes.

Place the filter tube in the muffle furnace at 800–900°C and ignite until all traces of carbon are removed. Cool in a desiccator and weigh (W_2 grammes).

Calculate the carbon black content from the formula:

$$\text{Carbon black, per cent} = \frac{(W_1 - W_2) - (C_1 - C_2)}{W} \times 100$$

where W is the weight of the test portion in grammes.

NOTE. Carbon black which will not filter easily may usually be separated by high-speed centrifuge. In such cases transfer the degraded polymer solution and black from the dissolution flask to a silica centrifuge tube, centrifuge and decant the supernatant layer. Rinse the flask into the centrifuge tube with 5 or 6 portions of diluting solvent, stir with a glass rod and centrifuge after each 10 ml addition. Evaporate the residual solvent remaining after the last washing in a gentle current of air, then add 5 ml hot dilute nitric acid to the flask, swirl, and add to the centrifuge tube. Stir again with the glass rod and then centrifuge and decant the clear liquid. Repeat the acid treatment until the washings are colourless and then continue the washing sequence with three 5 ml portions of water. Finally, wash with acetone and use a policeman to dislodge particles of black from the glassware. After the last decantation dry the tube in a gentle current of air and then determine the black by heating first in purified and dried nitrogen and then by ignition as already described in Sections 2.4.1 and 2.4.2. The correction for moisture in kieselguhr will, of course, be unnecessary.

SECTION 3. PYROLYSIS METHOD

3.1. Summary of method. The polymer is removed by pyrolysis at a relatively low temperature in a stream of nitrogen, and the combustion boat containing the non-volatile residue is weighed. In the absence of acid insoluble silicates the carbon is burnt off in a muffle furnace and the boat weighed again to give the amount of carbon black by difference.

If acid insoluble silicates are present the combustion boat is heated again under nitrogen but at a temperature of 800–900°C and the liberated water is weighed after absorption in magnesium perchlorate. Finally, the carbon black is burnt off in a muffle furnace and the boat weighed to give the total loss by difference. A correction in respect of the water content of the silicates is applied and thus the carbon black content is obtained. The method is applicable to hydrocarbon rubbers comprising polyisoprene (natural and synthetic), polybutadiene, styrene butadiene rubber, butyl rubber and to acrylate rubber.

3.2. Reagents. All reagents shall be of recognized analytical reagent quality unless otherwise specified.

Nitrogen, dry and free from oxygen*.

Magnesium perchlorate, retained on a 710-micron B.S. test sieve and passing a 1200-micron test sieve† (14–22 mesh).

Xylene.

3.3. Apparatus.

Combustion boat, silica, with handle, 55–60 mm long.

Electric tube furnace, with means for advancing and withdrawing the combustion boat.

Combustion tube with reduced exit.

Vapour absorbing system consisting of:

a. A trap for readily condensible vapours.

b. A xylene trap.

Absorption tube packed with magnesium perchlorate and closed with taps at both ends.

3.4. Procedure. Weigh about 0·1 g of the sample to the nearest 0·0002 g and extract with acetone and then with chloroform as described in B.S. 903, Parts B2 and B4.

NOTE. Extraction with chloroform is not necessary if materials not completely soluble in acetone, such as bitumen, are absent.

Dry the extracted rubber in air at moderate temperature. When the furnace has attained a temperature of 550 ± 25°C, place the test portion in the combustion boat and then place the boat in the entrance of the combustion tube and close with the entry fitting. Connect the supply of oxygen-free nitrogen through the flow-meter to the entry fitting, and the vapour-absorbing system to the exit of the combustion tube. Pass nitrogen through the system at about 200 ml/min for 5 min, then reduce the rate of flow to 100 ml/min and move the

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* Nitrogen sold commercially as free from oxygen may require further purification.

† B.S. 410, ' Test sieves '.

boat slowly into the centre of the furnace over a period of about 5 min. Leave the boat in the hot zone of the furnace for a further 5 min. At the end of this period withdraw the boat to the cold part of the tube and allow to cool for 10 min, maintaining the flow of nitrogen. Then take out the boat, complete the cooling in a desiccator and weigh at room temperature (W_1 grammes).

3.4.1. *Acid insoluble silicates absent.* Place the boat in the muffle furnace at 800–900°C and ignite until all traces of carbon are removed. Cool in a desiccator and weigh (W_2 grammes).

Calculate the carbon black from the formula:

$$\text{Carbon black, per cent} = \frac{(W_1 - W_2)}{W} \times 100$$

where W is the weight of the test portion in grammes.

3.4.2. *Acid insoluble silicates present.* Replace the combustion tube with one having a reduced exit to which an absorption tube may be attached. Heat the furnace to 800–900°C and adjust the flow of nitrogen to 20–30 ml/min. Attach the absorption tube packed with magnesium perchlorate, pass nitrogen through the system for 15 min, detach the absorption tube and weigh when at room temperature. This is the conditioning process for the absorption tube.

With the nitrogen still flowing at a rate of 20–30 ml/min remove the entry fitting to the combustion tube, insert the boat and connect the absorption tube to the exit of the furnace. Open the taps of the absorption tube and immediately close the combustion tube with the entry fitting. Push the combustion boat into the hot zone at 800–900°C and heat for 20–30 min. Disconnect the absorption tube, immediately close the taps and weigh when the tube is at room temperature. The increase in weight is due to water in the silicates (C_1 grammes).

Place the combustion boat in the muffle furnace at 800–900°C and ignite until all traces of carbon are removed. Cool in a desiccator and weigh (W_2 grammes).

$$\text{Carbon black, per cent} = \frac{(W_1 - W_2) - C_1}{W}$$

where W is the weight of the test portion in grammes.

SECTION 4. NITRIC ACID METHOD

4.1. Summary of method. The rubber is heated with nitric acid of such concentration as to oxidize all the organic matter but not the free carbon. The insoluble residue of carbon (with any mineral matter) is separated by filtration on a kieselguhr pad prepared in a filter tube, and is treated with a mixture of acetone and chloroform to remove residual organic material. The filter tube and its contents are dried and then heated in nitrogen at about 340°C to remove most of the substances absorbed on the black. The filter tube is cooled and weighed.

If acid insoluble silicates are absent the carbon black is burnt off in a muffle furnace and the filter tube weighed again to give the amount of carbon black by difference.

If acid insoluble silicates are present the filter tube is heated again under nitrogen, but at a temperature of 800–900°C, and the liberated water is weighed after absorption in magnesium perchlorate. The black is burnt off in a muffle furnace and the filter tube weighed again to give the total loss by difference. A correction in respect of the water content of the silicates and kieselguhr is applied and thus the carbon black content is obtained. The small amount of water in the kieselguhr is also corrected for by a blank determination.

The method can be recommended only for polyisoprene, natural or synthetic.

4.2. Reagents. All reagents shall be of recognized analytical quality unless otherwise specified and demineralized water shall be used wherever water is specified.

Nitric acid, dilute: 3 vol. concentrated nitric acid ($d = 1{\cdot}42$) diluted with 1 vol. water.

Kieselguhr

Silica wool

Carbon tetrachloride

Nitric acid, concentrated: $d = 1{\cdot}42$

Acetone-chloroform mixture. Mix 2 vol. acetone with 1 vol. chloroform

Nitrogen dry and free from oxygen*

4.3. Apparatus.

Filter tube, silica, consisting of a 10 mm diameter sintered silica filtered disk, porosity Grade 3 (B.S. 1752†) fused in to one end of a silica tube 10 cm long. The tube may be widened to 15 mm diameter at its upper end to facilitate the transfer of solutions.

Muffle furnace.

Electric tube furnace with means for advancing and withdrawing the filter tube.

Combustion tube with reduced exit.

Absorption tube packed with dried magnesium perchlorate, 14–22 mesh, and closed with taps at both ends.

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* Nitrogen sold commercially as free from oxygen may require further purification.

† B.S. 1752, 'Laboratory sintered or fritted filters'.

4.4. Procedure. Weigh about 0·1 g of the sample to the nearest 0·0002 g and extract with acetone and then with chloroform as described in B.S. 903, Parts B2 and B4.

NOTE. Extraction with chloroform is not necessary if materials not completely soluble in acetone, such as bitumen, are absent.

Dry the extracted rubber in air at moderate temperature and place in a beaker. Add cautiously 20 ml dilute nitric acid, promoting the reaction with gentle heat if necessary and then warm finally at 90–100°C for one hour. Add about 0·1 g of ignited kieselguhr and disperse it thoroughly by heating the mixture a few minutes longer. Cool the beaker and contents, add 10–20 ml of carbon tetrachloride and agitate thoroughly to mix with the nitric acid.

Place 0·2 g kieselguhr in the filter tube and cover it with a 5 mm layer of silica wool. Condition the tube by washing first with hot dilute nitric acid, then with water, and follow by igniting in a muffle furnace at 800–900°C for 20–30 min. Fit the filter tube into the neck of a Buchner flask and wet the pad with a few millilitres of concentrated nitric acid. Apply gentle suction and slowly pour the contents of the beaker into the filter tube. Rinse the beaker several times into the filter tube with hot dilute nitric acid, completing the transference finally with hot water and using a policeman if necessary to dislodge the black. When all the black is transferred to the filter tube wash with a boiling mixture of acetone and chloroform until the washings are colourless. (Do not allow the filter tube to become dry between additions of washing liquid.) Dry the filter tube in an oven at 100–140°C.

Place the dry filter tube, open end first, into the end of the combustion tube of the electric furnace heated to about 340°C, close the combustion tube with the entry fitting and pass oxygen-free nitrogen through the system at a rate of about 200 ml/min. After 5 min reduce the rate of gas flow to about 100 ml/min, then push the filter tube into the hot zone and heat for 30 min. Return the filter tube to the end of the combustion tube, cool with nitrogen still passing, remove from the combustion tube, complete the cooling in a desiccator and weigh at room temperature (W_1 grammes).

4.4.1. *Acid insoluble silicates absent.* Place the filter tube in the muffle furnace at 800–900°C and ignite until all traces of carbon are removed. Cool in a desiccator and weigh (W_2 grammes).

Calculate the carbon black content from the formula:

$$\text{Carbon black, per cent} = \frac{(W_1 - W_2)}{W} \times 100$$

where W is the weight of the test portion in grammes.

4.4.2. *Acid insoluble silicates present.* Replace the combustion tube with one having a reduced exit to which an absorption tube may be attached. Heat the furnace to 800–900°C and adjust the flow of nitrogen to 20–30 ml/min. Attach the absorption tube packed with magnesium perchlorate, pass nitrogen through the system for 15 min, detach the absorption tube, immediately close the taps and weigh the absorption tube when at room temperature. This is the conditioning process for the absorption tube.

With the nitrogen still flowing at a rate of 20–30 ml/min remove the entry fitting to the combustion tube, insert the filter tube and connect the absorption tube to the exit of the furnace. Open the taps of the absorption tube and immediately close the combustion tube with the entry fitting. Push the filter tube into the hot zone at 800–900°C and heat for 20–30 min. Disconnect the absorption tube, immediately close the taps and weigh when the tube is at room temperature. The increase in weight is due to the water in the silicates and kieselguhr (C_1 grammes).

Withdraw the filter tube and cool in the air to room temperature. Insert the filter tube into the furnace again and reconnect the absorption tube, which has been conditioned as explained above, to the exit end of the combustion tube. Open the taps on the absorption tube and heat the filter tube to 800–900°C for 20–30 min with nitrogen still passing at 25–30 ml/min. Disconnect the absorption tube, close the taps, cool and weigh. The second increase in weight is due to water absorbed by the kieselguhr (C_2 grammes). The weight of water derived from the silicates is therefore $(C_1 - C_2)$ grammes.

Transfer the filter tube to the muffle furnace at 800–900°C and ignite until all traces of carbon are removed. Cool in a desiccator and weigh (W_2 grammes).

Calculate the carbon black content from the formula:

$$\text{Carbon black, per cent} = \frac{(W_1 - W_2) - (C_1 - C_2) \times 100}{W}$$

where W is the weight of the test portion in grammes.

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