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**भारतीय मानक मसौदा**

**पेट्रोलियम और इसके उत्पाद — परीक्षण की पद्धतियाँ**

**भाग 4: प्रयोगशाला विश्लेषण के लिए पेट्रोलियम कोक का नमूना तैयार करना**

**[द्वितीय पुनरीक्षण]**

*Draft Indian Standard*

**PETROLEUM AND ITS PRODUCTS — METHODS OF TEST**

**PART 4 SAMPLING OF PETROLEUM COKE FOR LABORATORY ANALYSIS**

*[Second Revision]*

(ICS 75.080)

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Methods of Sampling and Test for Petroleum and related  
Products of Natural or Synthetic Origin (excluding  
bitumen) Sectional Committee PCD 01

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Last date for receipt of comment is  
**02 December 2024**

**FOREWORD**

*(Formal clause will be added later)*

Reduction & division of petroleum coke is necessary to produce appropriate analytical samples to determine different physical and chemical properties in the laboratory. Mechanical sampling from moving streams is the desired method for sampling. However, often infrastructure required for mechanical sampling are not available. The fundamental requirements of sampling are that all particles of the coke in the lot are accessible to the sampling instrument and that each individual particle has an equal probability of being selected and included in the sample. In case of manual sampling conditions are often far from ideal. The methods described in this standard are proposed to obtain the most representative sample that can be achieved. Manual sampling should only be applied if no possibility for mechanical sampling exists.

IS 1447 "Methods of sampling of petroleum and its products" was initially published in 1966, based on the overseas publications IP Part IV 1962 Ed. 2 'Methods of sampling' issued by "The Institute of Petroleum, London, ASTM D 270 Sampling and petroleum products and ASTM D 1265 Sampling of liquefied petroleum (LP) Gases issued by the American Society for Testing and Materials, respectively. Later with the advent of techniques, the Committee decided to revise the

standard dividing it into parts. Accordingly, first revision of the standard was taken up in 1989 and published based on ASTM D 346-78 and ASTM D 2013-86.

The second revision has been brought out to keep pace with the latest technological developments and international practices. In this revision following major changes have been made:

- a) Reduction of sample quantity to the desired level Coning and Quartering method has been introduced along with existing riffle divider.
- b) IS 1448 [P: 132] has been incorporated for determination of moisture content in raw and calcined petroleum coke. The moisture determined by this test method shall be used for calculating other analytical results on a moisture-free basis. The percentage of moisture determined by this test method shall also be combined with dry moisture loss to determine the total moisture in the sample. Total moisture shall then be used for calculating other analytical results on an 'as-received' basis.

In the preparation of this standard, considerable assistance has been derived from the following standards:

ASTM D 346/346M -11 Standard Practice for Collection and Preparation of Coke Samples for Laboratory Analysis

ASTM D 2013/D2013M-18 Standard Practice for Preparing Coal Samples for Analysis

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2: 2022 'Rules for rounding off numerical values (*second revision*)'.

## **1 SCOPE**

**1.1** This standard prescribes the method of test for the collection and preparation of petroleum coke samples for laboratory analysis.

## **2 REFERENCES**

The following Indian Standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
IS 436 (Part 1/Sec 1):2024	Methods for sampling of coal and coke Part 1 sampling of coal, section 1 manual sampling
IS 1448 Part 132: 2018	Methods of Test for Petroleum and its Products: Part 132 Determination of Moisture Content in Raw and Calcined Petroleum Coke

## **3 TERMINOLOGY**

### **3.1 Gross Samples**

The original sample representing one lot of petroleum coke, that is, composed of a number of increments on which size reduction or division has not been performed.

### **3.2 Bulk Samples**

A representative portion (R.P.) of the gross sample prepared for ease of handling. When the gross sample is very large, it is desirable to reduce it to a manageable amount for further subdivision of the R.P. The bulk sample should not exceed approximately 90 kg and should not be less than approximately 25 kg. If the gross sample is less than about 90 kg a bulk sample is not needed.

### **3.3 Analytical Sample**

The quantity of petroleum coke sample delivered to the laboratory for further preparation and analysis of a small R.P. of the gross sample or bulk sample specifically designed for a particular analytical test.

### **3.4 Sub-sample**

An unbiased portion taken from the sample at any point during sample reduction or division, or both, to be used for a special purpose.

### **3.5 Description of Riffle Divider**

**3.5.1** A manual sample divider which divides the sample of petroleum coke into two parts of approximately equal mass. Typical riffle bucket and riffle stands are shown in Fig. 1.

**3.5.2** It is preferable that feed chutes and enclosed riffles are used for sample preparation. The slope of feed chutes and riffles shall be at least 60 degree for free flow of moist material.

### **3.6 Coning and Quartering method**

Coning and quartering is a manual method to reduce the sample quantity up to the desired level. The petroleum coke sample shall be crushed to the desired size fraction and shall be heaped into the shape of a cone by pouring one portion (shovel/scoop) of the material after another at the apex of the cone till the entire sample has been coned. The material shall be allowed to slide down the sides of the cone only under the influence of gravity.

Flatten the cone evenly so that it forms a low circular pile. Cut the pile into four quarters along two diameters which intersect at right angles. Retain one pair of opposite quarters and reject the other. Repeat till the size of the retained sample is reduced to the required mass (*see* IS 436 Part 1/Sec 1).

### **3.7 Air-Drying**

A process of partial drying of coke to bring its moisture near to equilibrium with the atmosphere in the room in which further reduction and division of the sample is to take place.

### **3.8 Moisture Sample**

A representative portion (R.P.) of the gross sample used to check the 'As received' moisture in a lot (shipment) of raw petroleum coke. The gross sample and all R.P.'s of the gross sample used to obtain the analytical moisture sample shall be protected from changes in composition due to exposure to rain, snow, wind, sun, contact with absorbent materials, and extremes of temperature (not less than ambient climate or greater than 45 °C). The circulation of air through equipment shall be reduced to a minimum. Samples in which moisture content is important shall be protected from excessive air flow and then shall be stored in moisture tight containers.

## **4 COLLECTION OF GROSS SAMPLE**

**4.1** Sample coke while it is being loaded into or unloaded from railway wagon, ships or trucks or when discharged from supply bins, belt conveyors or other coke conveying equipment. Mechanical sampling devices are more reliable compared to manual intervention. However, when it is not possible, manual sampling may be done. Sampling shall be done close to the point of interest as possible.

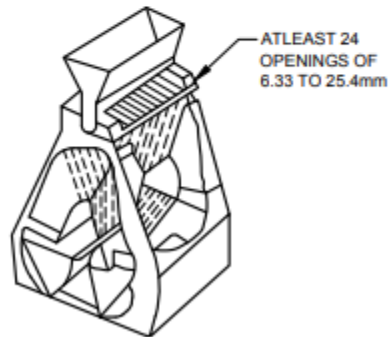
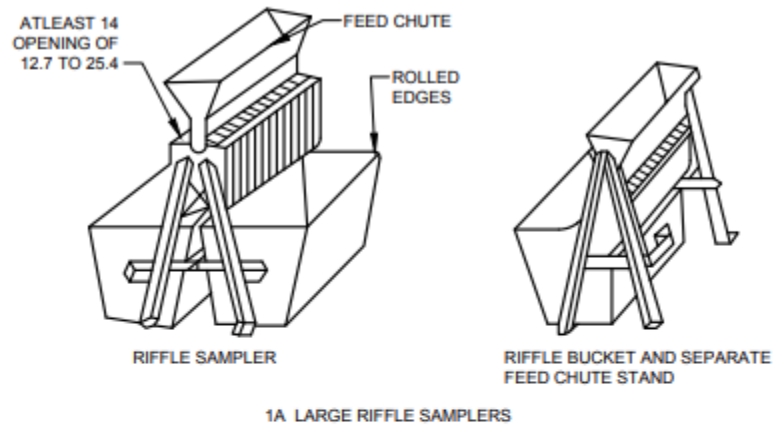
**4.2** When taking the sample, use a pail or a specially built receptacle which may be swung completely across the flowing stream in a brief interval, so as to take all of the stream for a fixed interval. Under no circumstances should the sampling receptacle be allowed to overflow or spill.

**4.3** Increments shall be collected regularly and systematically and with such frequency, so that the entire quantity of the coke sampled shall be proportionately represented in the gross sample. The standard gross sample should approach the quantities given in Table 1.

**Table 1 Mass of the Gross Sample for Tonne of Coke**  
(Clause 4.3)

<b>SI. No.</b>	<b>Tonnage of Coke to be Sampled (Ton)</b>	<b>Minimum Sample Required Gross Sample (kg)</b>
<b>(1)</b>	<b>(2)</b>	<b>(3)</b>
i)	1000 (ship load)	500
ii)	500	250
iii)	50	25

**4.4** For quantities up to 500 tonnes it is recommended that one gross sample represents the lot. A separate gross sample may be taken for each 500 tonnes or fraction thereof. After reduction of each of the gross sample to a smaller particle size the resulting smaller samples (reduced particles) may be composited, blended and divided. To composite (blend) to make one gross sample, the size of the top size particle shall be no larger than the particle size listed in Table 2 for mass of individual gross samples.



NOTE - May be constructed either closed or open type, closed type preferred.

FIG. 1 SAMPLE DIVIDERS RIFFLE

**4.5** All gross samples taken from shipment of petroleum coke shall contain at least 25 kg.

**4.6** The gross sample of 25 kg will serve as the bulk sample.

## **5 SIZE OF INCREMENTS**

**5.1** When increments are collected from the surface of a loaded rail wagon or truck shipment, the gross sample shall consist of nine increments of approximate equal quantity. When increments are taken from piles, conveyor belts, barges, etc. the gross sample shall consist of not less than 25 nor more than 50 increments of approximately equal quantity.

NOTE - Sample collected from the surface of coke in piles, pins, cars, ships or barges are, in general, unreliable because of size segregation and should not be used for determining conformance to specifications unless the purchaser and seller agree. If it is necessary to collect a sample of coke from the surface of a loaded railroad car, locate the nine points as shown in Fig. 2. The diameter of the hole at the point of sample collection shall be at least three times that of the largest piece in the shipment.

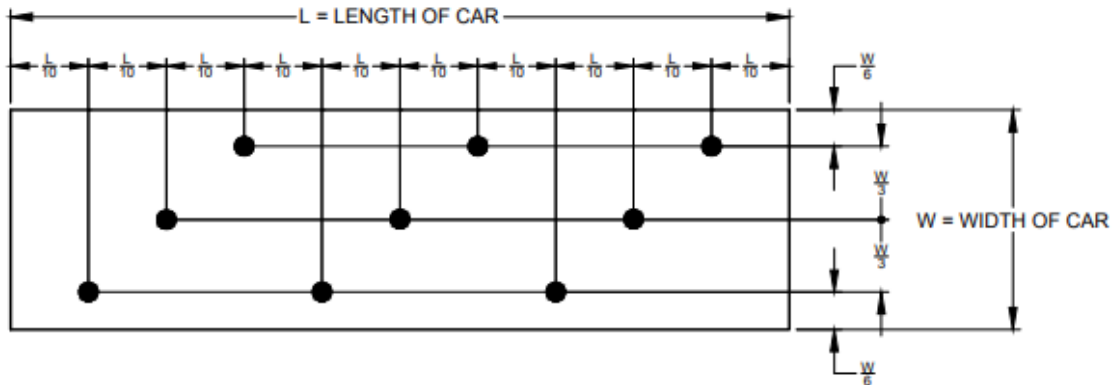


FIG. 2 LOCATION OF SAMPLING POINTS FROM EXPOSED SURFACE OF CAR

## 6 PREPARATION OF COKE SAMPLE FOR LABORATORY ANALYSIS

### 6.1 Gross Sample

**6.1.1** The preparation of the gross sample shall be done by trained and experienced personnel and should be performed in an enclosed space, roofed, cool and free from excessive air movements.

**6.1.2** In handling the gross sample the entire quantity of petroleum coke thus being sampled should be represented proportionately in the sub-sample.

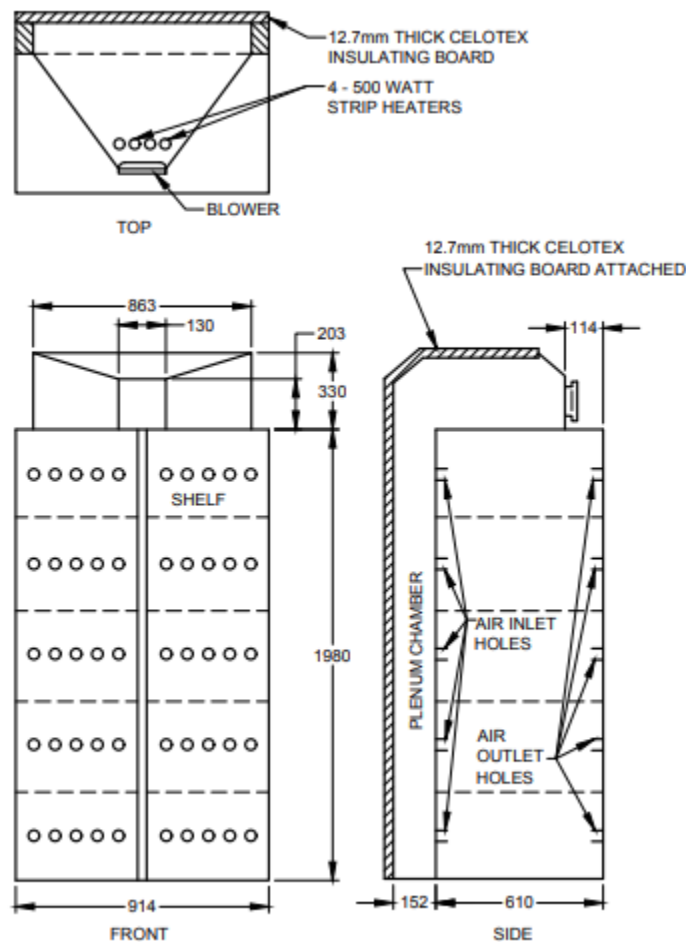
### 6.2. Air Drying of the Gross Sample

**6.2.1** When the gross sample contains excessive moisture which will hamper the riffing operation for the moisture R.P., the gross sample shall be weighed and air dried and weighed again recording the percent, mass loss. In case of excessive moisture in the sample, coning and quartering method is recommended to get required R.P. sample. However, for wet sample necessary air drying is required for smoothly passing the samples through crushing/grinding circuit.

**6.2.2** For air-drying the gross sample either of the following methods is suitable.

**6.2.2.1** Drying pans shall be made of non-corroding metal and of sufficient size so that the sample can be spread to a depth of preferably not more than 25 mm, with the sides of the pan not exceeding 38 mm in height. The room should be free of dust and excessive air currents to avoid contamination.

**6.2.2.2** Air-drying ovens shall be used to pass slightly heated air over the sample. The oven shall be capable of maintaining a temperature 10 °C to 15 °C above room temperature, with a maximum temperature of 45 °C. Air volume changes should occur at a rate of 1 to 4 per minute to avoid excessive drying. A typical oven is shown in Fig. 3.



All dimensions in millimetres.

FIG. 3 AIR-DRYING OVEN

**6.2.2.3** A drying floor may be used to dry the gross sample. A smooth clean floor in a room free from dust and excessive air current is suitable.

**6.2.2.4** Continue air-drying until the loss in mass of the total gross sample is not more than 0.1 percent per h. stirring at intervals will reduce the air-drying time. Avoid excessive drying time.

### 6.3 Bulk Sample

**6.3.1** The gross sample is now subdivided to yield a bulk sample for convenience in handling and for ultimately making the small analytical sample required. To obtain a bulk sample from a large gross sample, the top size particle shall not exceed the particle size listed in Table 2 below for the mass of the gross sample.



**Table 2 Sub-Division of Gross Sample**  
(Clauses 4.4, 6.3.1, 6.3.3, 6.3.5 and 6.4.3.3)

SI. No.	Mass of the Sample to be Divided Kg	Largest Size of Petroleum Coke in Sample Before Division mm
(1)	(2)	(3)
i)	450 and above	25.0
ii)	225	19.0
iii)	110	12.5
iv)	60	10.0
v)	30	6.7
vi)	15	4.75

**6.3.2** The bulk sample should not be exceeding about 90 kg and should not be less than 25 kg. If the gross sample needs to be further subdivided to obtain a bulk sample, this may be done using a mechanical sample reducer.

**6.3.3** The desired bulk sample may also be obtained by using a riffle or the coning quartering method after the gross sample has been sized to meet the requirement in Table 2.

**6.3.4** Air dry (*see* 6.2.2) the gross sample, if needed, so that it shall be fed properly through the large riffle (75 mm riffle). Calcined coke usually does not require air-drying. Air drying may not be required in case of coning and quartering method is applied.

**6.3.5** Reduce the gross sample to a top size to meet the requirements specified in Table 2.

**6.3.6** Determine the number of passes required in the riffling operation based on the total mass of the gross sample and the mass desired for the bulk sample.

**6.3.7** Pass the coke through the riffle from a feed scoop, feed bucket or riffle pan having a lip or opening extending the full length of the riffle. When using any of these containers to feed the riffle, spread the coke evenly in the container, raise the container, and hold it with its front edge resting on the top of the feed through. Then slowly tilt the container so that the coke flows in a uniform stream through the hopper, straight down over the centre of riffle, into each slot, then, into the pans. Under no circumstances should the sample be shoveled into the riffle or dribbled from a small mouthed container. Do not allow the coke to build up in or above the riffle slots. If the coke does not flow freely through the slots, shake or vibrate the riffle to facilitate the flow. The riffle used for this operation shall have a slot opening of at least 2 to 24 times greater than the top size particle of the petroleum coke being riffled.

**6.3.8** Similarly in case of coning and quartering method determine the number of cycles required to get the desired quantity of sample.

**6.3.9** The gross sample is now reduced to a bulk sample that shall be conveniently handled. The bulk sample is now ready for the preparation of the small analytical sample.

**6.4 Analytical Sample**

**6.4.1** The bulk sample shall now be handled in a way depending upon whether it is calcined or raw petroleum coke, to obtain the analytical sample. Two general schematics (I and II) are given as Fig. 4 and Fig. 5, one for raw petroleum coke and the other for calcined petroleum coke, illustrating the reduction of the bulk sample to a specific size portion for the analytical sample.

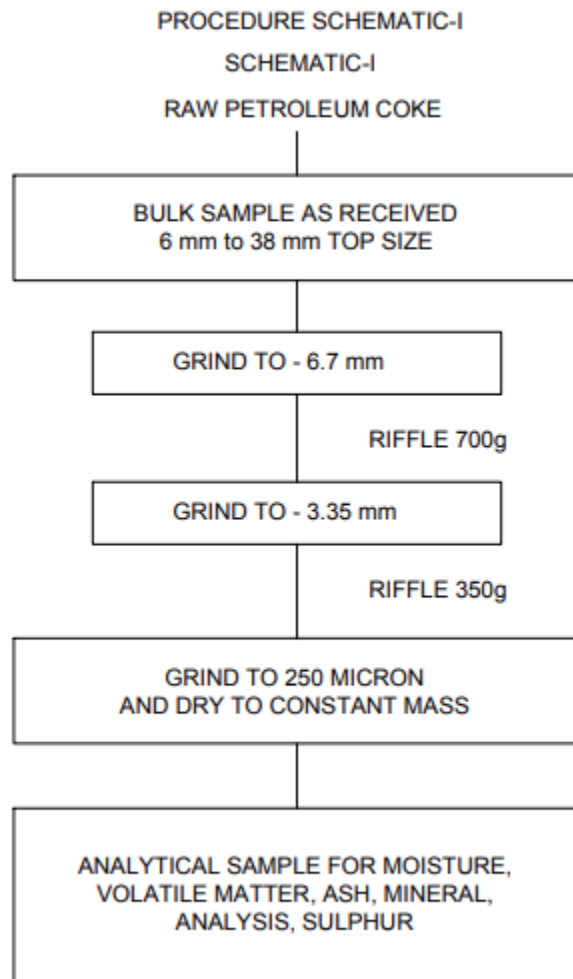


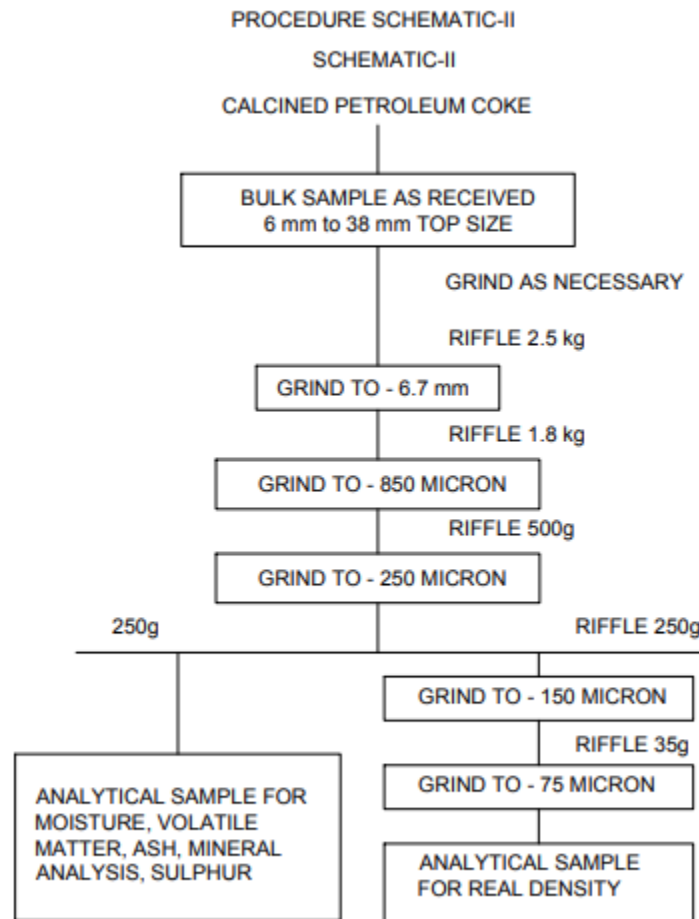
FIG. 4 PROCEDURE SCHEMATIC - I

**6.4.2 Analytical Sample from a Bulk Sample of Raw Coke**

**6.4.2.1** The bulk sample of raw petroleum coke will vary in mass from 25 kg to about 90 kg and will vary in top size particle from 6 mm to 38 mm, depending upon the coke and the mass of the gross sample from which the bulk sample obtained.

**6.4.2.2** Air dry (*see 6.2.2*) the bulk sample of raw coke, if needed, in order that it shall be properly ground and fed through riffles or use coning and quartering method.

**6.4.2.3** After the bulk sample has been air dried, grind the entire bulk sample to pass a 6.7 mm sieve using a jaw crusher. Do not over grind, keep a maximum amount of sample as coarse as possible.



**FIG. 5 PROCEDURE SCHEMATIC - II**

**6.4.2.4** Using a riffle, split the bulk sample to obtain a 700 g portion. Follow the procedure for riffling mentioned in **6.3.6** and **6.3.7**. Instead of riffling, the same amount of sample may be prepared using the coning and quartering method.

**6.4.2.5** Grind the 700 g sub-sample to pass a 3.35 mm sieve using a roll crusher. Do not over-grind. Use riffle or coning and quartering method to collect a sample of around 350 g.

**6.4.2.6** Using a roll crusher, grind the 350 g of sub-sample to pass through 250-micron IS Sieve. Do not over-grind. About one half of the sample at this point should be retained on a 125-micron IS Sieve.

**6.4.2.7** This prepared sample is now ready for analysis of moisture, volatile matter, ash content and other chemical analysis. The dried sample shall be stored in a closed container within a desiccator. Thoroughly mix the sample before extracting portion for analysis.

#### **6.4.3 *Analytical Sample from a Bulk Sample of Calcined Petroleum Coke***

**6.4.3.1** The bulk sample of calcined coke will vary in mass from 25 kg to about 90 kg and will vary in top particle size from 6 mm to 38 mm, depending upon the type of coke and the mass of the gross sample from which the bulk sample obtained.

**6.4.3.2** The bulk sample shall be air-dried if necessary in accordance with **6.2.2**. Very seldom does a calcined coke has to be air dried.

**6.4.3.3** The bulk sample of calcined petroleum coke shall be ground according to the mass and size indicated in Table 2 and then either riffled or reduced using the coning and quartering method into an approximately 2.5 kg portion

**6.4.3.4** The 2.5 kg portion of the bulk sample shall now be ground to pass a 6.7 mm sieve using a jaw crusher (do not over-grind) and then riffled or reduced using the coning and quartering method to collect approximately 1.8 kg.

**6.4.3.5** The 1.8 kg riffled portion of minus (-) 6.7 mm sample shall be further ground in a roll crusher to pass through an 850-micron IS Sieve. Do not over-grind the sample. Divide the sample to obtain about 500 g.

**6.4.3.6** Grind this 500 g sub-sample with a roll crusher to pass through a 250-micron IS Sieve and then divide into two parts.

**6.4.3.7** The first portion (250 g) of the sample shall be used for the analytical sample for determination of moisture, volatile matter, ash, and Sulphur, silicon and trace metal analysis.

**6.4.3.8** The second portion (250 g) of the sample shall be further ground to a 150-micron size and about 35 g shall be riffled out and ground in a ball mill or other suitable pulverizing equipment to pass through a 75-micron IS Sieve. This sample shall be used for the analytical sample for determination of real density test.

**6.4.3.9** Store the sample in closed containers and mix it thoroughly before extracting portions for analysis.

## **6.5 Operation of Grinding Equipment**

### **6.5.1 Jaw Crusher Operation**

- a) Every effort shall be made to grind the sample in a manner that prevents the introduction of contaminants. The jaw crusher shall be cleaned after each use.
- b) Set the jaw crusher to the desired top size particle by using the manganese steel jaw plates;
- c) Pass only + (mesh) sample through the jaw crusher, catching the sample below the jaw in a suitable container;
- d) Feed the jaw crusher slowly, never completely filling or overflowing the jaw compartment.

### **6.5.2 Roll Crusher Operation**

- a) Every effort shall be made to grind the sample in a manner that prevents the introduction of contaminants. It is good practice to avoid using the same sample preparation equipment for handling both types of petroleum coke (calcined and raw).
- b) When grinding a coke, set the rolls far enough apart to crush only the largest particles of sample (R.P.).
- c) Screen out all the minus (-) mesh of the desired size (particle), commonly known as scalping. Only the plus (+) mesh shall be crushed again.
- d) Turn on the roll crusher and pour the scalped sample very slowly through the rolls.
- e) Again, scalp off the plus (+) mesh size and blend of the minus (-) size with the minus (-) mesh size from (c) above. Only the plus (+) mesh size shall be ground through the roll crusher again.
- f) Repeat the crushing and scalping process until all material is in the desired minus (-) mesh size. Ensure that the final minus (-) mesh particles are well blended. Do not rush this operation.