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Indian Standard

METHODS FOR SAMPLING OF COAL AND COKE PART I SAMPLING OF COAL Section 1 Manual Sampling

(Revised)

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BUREAU OF INDIAN STANDARDS MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG NEW DELHI 110002

February 1965

Indian Standard

METHODS FOR SAMPLING OF COAL AND COKE

PART I SAMPLING OF COAL

Section 1 Manual Sampling

(Revised)

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Indian Standard

METHODS FOR SAMPLING OF COAL AND COKE

PART I SAMPLING OF COAL

Section 1 Manual Sampling

(Revised)

0. FOREWORD

0.1 This Indian Standard (Part I) (Revised) was adopted by the Indian Standards Institution on 20 July 1964, after the draft finalized by the Solid Mineral Fuels Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was originally issued in 1953 with a view to prescribing the procedures for obtaining representative samples of coal and coke for test purposes. However, in view of the fact that at that time experiments on Indian coal under Indian conditions had just been initiated, it was issued as a tentative Indian Standard. The present revision incorporates the information obtained from the extensive investigations since carried out and the experience gained during the last eleven years.

0.3 The present revision differs from the earlier version in several important respects.

0.3.1 For purposes of sampling, Indian coals had originally been divided into five size groups, but the Sectional Committee responsible for the preparation of this standard felt that the division of coal into so many size groups was rather stringent. Hence, it decided that for the purpose of sampling, coal should be divided into the following three size groups only (see Note), which would generally be adequate in practice:

Name Run-of-mine coal Coal, large

23 to 0 cm 15 to 5 cm (the upper limit not exceeding 23 cm) 5 to 0 cm

Nominal Size

Coal, small

NOTE — The size groups given in 0.3.1 are meant only for convenience in sampling. As regards the size grading of coal, the various sizes as specified in IS: 437-1956^o shall apply.

*Size grading of coal and coke for marketing (revised). (Second revision of this standard also issued in 1965.)

0.3.2 Depending upon the ash content, the Indian coal had originally been divided into two ash groups, namely, with ash less than 10 percent and with ash 10 percent or more. But since the ash content of most of the Indian coals was found to be generally more than 10 percent, and since the variation in the quality of Indian coal does not depend on ash content, it was felt that the division of coal into two ash groups was not necessary and that it may be dispensed with. Accordingly, this standard lays down the sampling procedures for coal without any regard to its ash content.

0.3.3 The earlier version had specified the minimum number of increments and the minimum weight of the gross sample for an accuracy of +10 percent of the value sought with 95 percent probability. These recommendations had been made on the basis of results obtained for American coals. The experiments conducted on Indian coals, however, revealed that the average and the standard deviation for the ash percentage were not correlated, implying thereby that the standard deviation does not increase or decrease with the average ash in any well-defined manner. This finding made it imperative that the number of increments and the weight of the gross sample for Indian coals be determined differently. Accordingly, the optimum weight for a gross sample was first determined by trying out different sample weights and adjudging their optimal nature by taking the minimum standard deviation criterion. That is, the weight of the gross sample for which the observed standard deviation was found to be minimum was deemed to be the minimum weight for drawing samples in practice. The weight of the increment was then determined separately and thus the number of increments to be taken for constituting the gross sample became known.

0.3.4 Regarding the sampling procedure recommended in the earlier version it was also felt that this procedure did not provide any means for checking whether the expected accuracy (of ± 10 percent of the value sought with 95 percent probability) was being maintained in the course of sampling or not. This checking becomes essential as the errors introduced due to uncertainties at the various stages of reduction of a gross sample might, in effect, render the confidence limits obtained for the true average ash content inapplicable. To obviate this difficulty, it was considered desirable to always test more than one gross sample so that the test results themselves may furnish an estimate of the sampling error which can be utilized in setting the confidence limits.

0.3.4.1 Accordingly, this standard recommends the division of a lot into a number of sub-lots (see Table 1) depending upon the weight of the lot and then drawing a representative gross sample from each of the sub-lots separately. The gross samples after suitable reduction are to be tested individually for determination of important characteristics of coal and compositely for others. The standard also lays down a proper procedure for valid interpretation of the test results so as to evaluate the average

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level of quality as also the extent of variation in the average for a lot with 95 percent confidence.

0.3.4.2 For obtaining reliable conclusions, it is recommended that coal may be sampled when it is in motion, that is, from conveyers or during loading or unloading. For this purpose the sampling procedure as laid down in **3**, **4** and **5** shall be followed. If, however, it is desired to sample the coal when it is stationary as, for example, from a stock pile or a loaded wagon, the procedures as laid down in **6**, **7** and **8** may be followed. It may, however, be mentioned that the representativeness of the samples drawn in this manner and hence the reliability of the conclusions is not likely to be assured.

0.4 Careful consideration was given to the various conditions under which coal may be available or supplied, for example, coal reserves, wagons, stock piles, conveyers, ships, etc. However, taking into account the many variations in the sampling conditions as also from the commercial point of view, the Committee responsible for the preparation of this standard felt that the sampling procedures should be prescribed only for conveyers, wagons, ships, stock piles and seams.

0.5 In drawing up the tables for the minimum weight of gross samples, consideration has been given to the fact that the distribution of impurities varies more in some coals than in others. The recommendations made in Tables 2 and 3 for the increment sizes and the total weight of the gross samples are based on the variation of ash percentage in coal. But since this is the most variable characteristic of coal, the recommended quantities of samples are expected to be adequate for the determination of other characteristics also.

0.6 This standard as originally issued had also included the sampling procedure for coke. The investigations on coke are, however, still in a preliminary stage and it will take quite sometime before sufficient information is collected. In the meanwhile, there exists a great and expeditious demand for the sampling procedure for coal. The committee responsible for the preparation of this standard, therefore, decided to first publish the methods for sampling of coal as Part I of this standard. Part II, dealing with the sampling of coke, will be issued later on as and when the investigations on coke would be over. In the meantime, through an amendment all references for coal have been deleted from IS: 436-1953*. Sampling of Coke (Part II)-9f. IS: 436-1953* has been redesignated as IS: 436 (Part II)-1953* 'Methods for sampling of coal and coke, Part II Sampling of coke'.

0.7 In preparing this standard considerable assistance was derived from the statistically designed investigations carried out at the various collieries in the country by the Central Fuel Research Institute, Jealgora, Dhanbad.

*Since revised.

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0.8 This standard forms one of a series of Indian Standards on coal and coke. Other standards in the series are:

*IS: 437-1956 Size grading of coal and coke for marketing (revised)

†IS: 439-1953 Hard coke

†IS: 770-1960 General classification of coal

- [‡]IS: 1350-1959 Methods of test for coal and coke proximate analysis, total sulphur and calorific value
 - IS: 1351-1959 Methods of test for coal and coke ultimate analysis
 - IS: 1352-1959 Methods of test for coal and coke special impurities
 - IS: 1353-1959 Methods of test for coal carbonization caking index, swelling properties and gray-king assay (L.T.) coke types
- FIS: 1354-1959 Methods of test for coke special tests
 - IS: 1355-1959 Methods of test for ash of coal and coke

0.9 For the determination of the various sizes of coal, as well as for the reduction purposes, sieves conforming to IS: 460-1962‡ shall be used. Where these sieves are not available, other equivalent standard sieves as judged by the aperture may be used. For corresponding sizes of British Sieves and Sieves of the American Society for Testing and Materials, reference may be made to IS: 460-1962§.

0.10 Metric system has been adopted in India and all quantities and dimensions in this standard have been given only in this system.

0.11 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: 2-1960 ||.

1. SCOPE

1.1 This standard prescribes the methods for sampling of coal from (a) conveyers, (b) wagons, (c) ships, (d) stock piles and (c) seams. It also lays down a procedure for reporting the quality of the material sampled.

2. TERMINOLOGY

2.0 For the purpose of this standard, the following definitions shall apply.

2.1 Coal, Large — Coal with nominal size 15 to 5 cm, the upper limit not exceeding 23 cm.

'†Since revised.

^{*}Second revision issued in 1965.

[‡] Since revised and split into various parts.

Specification for test sieves (revised).

[#]Rules for rounding off numerical values (revised).

2.2 Coal, Run-of-Mine — Unscreened coal containing all sizes, mainly 23 to 0 cm.

2.3 Coal, Small — Coal with nominal size 5 to 0 cm.

2.4 Composite Sample (for the Lot) — The quantity of coal obtained by mixing together equal quantities of coal from each of the laboratory samples representing the sub-lots into which the lot has been divided.

2.5 Gross Sample — Sample as collected from a sub-lot, that is, the quantity of coal obtained by aggregating together all the increments drawn from the same sub-lot.

2.6 Increment — The quantity of coal taken by a single operation of the sampling implement.

2.7 Laboratory Sample — The quantity of coal obtained by reducing a gross sample (see 2.5) by following a specified procedure for laboratory testing.

2.8 Lot - The quantity of coal offered for disposal at one time,

2.9 Moisture Sample — A sample to be used exclusively for the purpose of determining total moisture.

2.10 Sample Division — The process whereby a part of the sample is retained and the remainder rejected.

2.11 Sample Reduction — The process of crushing or grinding the sample to reduce the particle size and of mixing and sample-dividing in successive stages.

2.12 Sub-lot — The quantity of coal in each of the groups into which a lot is divided for the purpose of sampling; a lot may consist of two or more sub-lots.

3. SAMPLING FROM CONVEYERS

3.1 Sub-lots — For the purpose of sampling, a lot, while it is being discharged over a conveyer, shall be divided into a number of sub-lots of approximately equal weight as specified in Table 1.

TABLE 1 NUMBER OF SU	B-LOTS/GROSS SAMPLES
(Clauses 0.3	3.4.1 and 3.1)
-WEIGHT OF THE LOT (METEIC TONNES)	NO. OF SUB-LOTS/GROSS SAMPLES
Up to 500	2
501 ,, 1 000	3
1 001 " 2 000	4 4 -
2 001 ,, 3 000	5
Over -3 000	6

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3.1.1 A representative gross sample shall be drawn from each of the sublots and shall be kept separately. Thus, there will be as many gross samples as the number of sub-lots into which the lot has been divided.

3.2 The number of increments to be taken from a sub-lot for drawing the gross sample shall be governed by the weight of the gross sample and the weight of the increment as specified in Table 2 for various size-groups of coal. This number shall be evenly distributed over the sub-lot. The increments shall be drawn with the help of a suitable shovel (see Fig. 1) at regular intervals.

TABLE 2 WEIGHT OF GROSS SAMPLE AND NUMBER OF INCREMENTS FOR CONVEYERS					
Sl No.	SAMPLE	Run-of- Mine Coal	COAL, LARGE	COAL, Small	
(1)	(2)	(3)	(4)	(5)	
i)	Weight of gross sample, Min	350 kg	175 kg	75 kg	
ii)	Weight of increment (approx)	5 kg	5 kg	5 kg	
iii)	Number of increments, Min	70	35	15	

3.2.1 The increments shall preferably be taken from the full cross-section and thickness of the stream in one operation. When coal is in motion, the most reliable means of obtaining such increments is to sample at a point where the coal discharges from the belts. The best possible increment is one which cuts accross entirely a falling stream of the material by means of a suitable receptacle passed from one side of the stream to the other without allowing the receptacle to overflow. If the whole of the stream cannot be covered by one increment without overfilling the receptacle, the stream should be sampled systematically by taking material from all portions.

3.2.2 If it is not possible to sample satisfactorily at a point of discharge, the increments may be drawn from the moving belt itself. In this case, the increments shall be collected from the centre and the left and right sides of the belt along the same width. To ensure that very small material is also correctly obtained in the sample, the scoop should sweep the bottom of the conveyer.

3.2.3 If it is practicable to stop the belt periodically, increments may be collected from the whole cross-section of the stream by sweeping the whole of the coal lying between the sides of a suitable frame placed across the belt. The frame should be inserted in the coal until it is in contact with the belt across its full width.

NOTE — Before collecting the increments, the speed of the conveyer and the quantity of material passing a certain point in a given time shall be ascertained so that an appropriate spacing of the increments may be arranged over the whole of the lot.



FIG. 1 DIMENSIONS OF SHOVEL (CAPACITY 5 kg AND 7 kg APPROX)

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3.2.4 In case automatic samplers are available they may be utilized for drawing increments from a conveyer belt. The setting of such-machines shall be carefully adjusted to ensure that the whole thickness of the stream is taken.

3.2.5 The material collected from all the increments in a sub-lot shall be mixed together and shall constitute a gross sample.

4. SAMPLING FROM WAGONS DURING LOADING OR UNLOADING

4.1 Sub-lets — For the purpose of sampling, all the wagons in a lot shall be divided into a suitable number of sub-lots of approximately equalweight in accordance with the requirements of Table 1.

Norz — The object of dividing a lot into a number of sub-lots is only to facilitate the drawing of a representative gross sample rather than to indicate its physical division.

4.1.1 One gross sample shall be drawn from each of the sub-lots so that there are as many gross samples as indicated in Table 1.

4.2 In order to get a representative gross sample, coal shall be sampled as far as possible in steady motion during loading or unloading of the wagons.

4.2.1 A minimum of 25 percent of the wagons shall be selected at random from the sub-lot and to ensure the randomness of selection the procedure as given in Appendix A may be followed. The number of increments to be taken from the selected wagons and the weights of the increments and the gross sample shall be in accordance with Table 3. The increments shall be evenly distributed over the selected wagons, with a view to determining the necessary number of increments that should be collected from each of the selected wagons of the sub-lot for making up the gross sample. These increments shall be drawn with the help of a suitable scoop or shovel (see Fig. 1 and 2), depending upon the size of the coal (see Table 3), at regular intervals at the time of loading or unloading of the wagons.

Norz — In case bigger lumps are encountered which are not accommodated in the scoop or shovel, they shall be collected and crushed separately. The corresponding increments shall then be drawn from the crushed material.

TABLE 3	WEIGHT OF GROSS SAMPLE AND NUMBER OF
	INCREMENTS FOR WAGONS

Sl No.	SAMPLE	RUN-OB- MINE COAL	Coal, Large	COAL, Small	
(1)	(2)	(3)	(4)	(5)	
i) Weight of gross sample, Minii) Weight of increment (approx)		350 kg	175 kg	75 kg 5 kg	
		7 kg	7 kg		
iii) Numt	er of increments, Min	50	25	15	
	-			••	



All dimensions in centimetres.

FIG. 2 SAMPLING SCOOP (CAPACITY 5 kg and 7 kg)

4.2.2 The material collected from the selected wagons in a sub-lot shall constitute a gross sample.

5. SAMPLING FROM SHIPS DURING LOADING OR UNLOADING

5.1 Sub-lots — For the purpose of sampling, the entire quantity of coal in a ship shall be divided into a suitable number of sub-lots of approximately equal weight as specified in Table 1.

5.1.1 A gross sample shall be drawn from each of the sub-lots and shall be kept separately so that there will be as many gross samples as the number of sub-lots into which the lot has been divided.

5.2 Sampling of coal from ships shall be carried out, as far as practicable, when coal is in motion. If it is taken on a conveyer, the gross sample shall be collected as per the procedure laid down in 3. If not, the gross samples may be drawn during loading or unloading of the ship. For this purpose, the number of increments to be taken shall be governed by the weight of the gross sample and the weight of increment as specified in Table 3 for various size groups of coal.

6. SAMPLING FROM STOCK PILE

6.1 Sub-lots — For the purpose of sampling, the quantity of coal in stock pile shall be divided into a suitable number of sub-lots

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approximately equal weight as specified in Table 1 by suitably marking the lines of demarcation on the surface of the lot.

6.1.1 The surface of each sub-lot shall be levelled and one point for approximately every 250 metric tonnes of material in the sub-lot shall be chosen at random. A gross sample shall then be taken from each of the sub-lots as per the procedure given in **6.1.2** and **6.1.3**.

6.1.2 In case the height of the stock pile is not more than 1.5 m, the naterial shall be collected at every selected point by taking the whole section of coal from top to bottom over the area of a circle of 30 cm diameter. For doing so, coal from the surface up to a depth of approximately 50 cm shall be collected at first. The bottom of the hole so formed shall then be covered by a plate and the coal lying on the sides shall be removed up to that plate so that when the hole is dug further (to collect further samples), the coal from the sides may not fill up the hole by falling down. This procedure is repeated till the bottom is reached.

NOTE — If the quantity of the material collected in the above manner is less than that specified in Table 3, additional quantity of material shall be collected from other point(s) chosen for the purpose.

6.1.3 In case the height of the stock pile is more than 1.5 m, the sample shall be collected at every selected point by taking the material over an area of a circle of 30 cm diameter and up to a depth of 1.5 m. The manner for taking out the sample shall be as described in 6.1.2.

NOTE — Pipe sampling may be resorted to for coals of small size which have been closely screened or sized so that the material is approaching uniformity and is in any case not more than 4 cm top size. The equipment and the procedure to be used for pipe sampling is described in Appendix B.

7. SAMPLING FROM LOADED WAGONS

7.1 Sub-lots — For the purpose of sampling, the loaded wagons in a lot shall be divided into a suitable number of sub-lots of approximately equal weight in accordance with Table 1.

7.1.1 One gross sample shall be drawn from each of the sub-lots, so that there are as many gross samples as indicated in Table 1.

7.2 The gross sample shall be collected from a sub-lot according to the method described in 6.1.2. For this purpose a minimum of 25 percent of the wagons shall be selected from the sub-lot at random (see Appendix A) and one point shall be located at random on the coal surface of each of the selected wagons. At every selected point a sample shall be collected by taking the whole section of coal from top to bottom over an area of 30 cm diameter as described in 6.1.2.

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8. SAMPLING FROM LOADED SHIPS

8.1 Sub-lots — For the purpose of sampling, the quantity of coal in a loaded ship shall be divided into a suitable number of sub-lots of approximately equal weight as specified in Table 1.

8.1.1 A gross sample shall be drawn from each of the sub-lots according to the method given in 6.1.1 to 6.1.3 and shall be kept separately so that there will be as many gross samples as the number of sub-lots into which the lot has been divided.

9. SAMPLING OF COAL SEAM in situ

9.0 Sampling of coal *in situ* gives a measure of the quality of coal to be mined. The analysis of this sample, however, will seldom give the same results as those of a run-of-mine sample.

9.1 The section of seam to be sampled shall be exposed from the roof to the floor. The exposed surface shall be as smooth as possible so that a rectangular channel may be cut. It may often be necessary to penetrate into the seam for a few decimetres to avoid taking weathered coal especially if the 'face' has been standing exposed for some time. The seam sample shall be taken in a channel representing the entire cross-section of the scam having the dimensions of 30×10 cm, that is, 30 cm in width and 10 cm in depth. For this purpose, two parallel lines, 30 cm apart end at right angles to the bedding planes of the seam shall be marked by a chalked string on the smooth, freshly exposed surface of the seam. Obvious dirt bands exceeding 10 cm in thickness shall be excluded. The channel between the marked chalk lines in the seam shall be cut to a depth of 10 cm and the coal sample collected on a clean strong cloth or tarpaulin placed immediately at the bottom so that the chances of pieces flying off during excavation of coal are minimised. The total height of the channel shall be measured and noted. The excluded dirt bands shall, if required, be separately collected and analysed.

10. REDUCTION OF GROSS SAMPLE

10.1 Run-of-Mine Coal — The gross sample shall be crushed to 5 cm, preferably by mechanical means, mixed thoroughly and quartered. Two opposite quarters shall be retained and the rest rejected. The retained material shall be further mixed together, halved and one-half retained. Thus, ultimately one quarter of the original gross sample is retained and the rest rejected. The material so obtained shall be crushed to 12.5 mm by a jaw crusher and then to 3.35 mm by a palmac type of reduction mill (see Fig. 3). The crushed material shall be reduced either by coning and quartering or by riffling (see Appendix C) till 2 kg of sample is obtained.

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10.1.1 Laboratory Sample — The sample as reduced under 10.1 shall be inally ground to pass through 212-micron IS Sieve (see also C-4). From the ground material, 1.5 kg shall be taken which shall constitute the laboratory sample.

10.1.1.1 Each laboratory sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee. The samples shall be kept in glass or polyethylene containers and shall be sealed and marked properly.

10.2 Large Coal — The gross sample shall be crushed to 5 cm, preferably by mechanical means, mixed thorougly and then quartered. Two quarters shall be retained and the rest rejected. The material so obtained shall be crushed to 12.5 mm by a jaw crusher and then to 3.35 mm by a palmac type of reduction mill (see Fig. 4). The crushed material shall be reduced either by coning and quartering or by riffling (see Appendix C) till 2 kg of sample is obtained.

10.2.1 Laboratory Sample — From the sample as reduced under 10.2, the laboratory sample shall be prepared as under 10.1.1 and 10.1.1.1.

10.3 Small Coal — The gross sample shall be crushed to 3.35 mm in two steps, namely, through 12.5 mm by a jaw crusher and finally through 3.35 mm by a palmac type of reduction mill (see Fig. 5). The crushed material shall be reduced either by coning and quartering or by riffling (see Appendix C) till 2 kg of the sample is obtained.

10.3.1 Laboratory Sample — From the sample as reduced under 10.3, the laboratory sample shall be prepared as under 10.1.1 and 10.1.1.1.

10.4 Ash Fusion Sample — Following the procedure laid down under 10.1, reduce the gross sample in successive stages and take an additional 1 kg sample, passing 12.5 mm IS Sieve for the determination of ash fusion. If, however, it is desired still further to avoid any contamination whatsoever with iron, take separate quantities of about 5 kg of the coarsely broken coal (12.5 mm and below) for the test. Do not grind the sample further in iron mills or vessels. Grind the ash obtained by incineration in an agate mortar to pass 75-micron IS Sieve.

10.5 Moisture Sample — In view of the fact that mixing and crushing or grinding of coal has been shown by experiment to lead, in certain cases, to considerable loss of moisture and since it is usually necessary to know the moisture content of the original coal, collect, as prescribed under 10.5.3, two or three special moisture samples, as required, from the gross sample.

10.5.1 Where the moisture samples have to be reduced it is preferable to use machinery which crushes rather than machinery which grinds.

10.5.2 It is essential in all operations, especially if the coal has been partially or totally air-dried, that it should not be exposed for any length

REDUCTION OF GROSS SAMPLES



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of time to a current of air or to an atmosphere of appreciably different temperature or humidity.

10.5.3 At the first stage of reduction of the gross sample to 12.5 mm (see 10.1) collect 5 kg of sample (see Note below) by not less than 10 approximately equal increments. while the coal is being discharged from the jaw crusher or after it has been collected in a heap. Reduce this sample to as near to 1 kg as possible and put the sample so reduced in an air-tight tin and seal it without delay.

Nore — If it has been found necessary to air-dry the coal before crushing and it is required to know the excess moisture, a similar 5 kg sample (or more) of the original wet coal should be taken in a dry container subsequently to be used for the determination of excess moisture by air-drying.

11. NUMBER OF TESTS

11.1 The laboratory samples representing the various sub-lots in a lot shall be tested individually for the important characteristics like ash percentage, moisture, etc. For the remaining characteristics like ash fusion, a composite sample prepared by mixing equal quantities (by weight) of the material from each of the laboratory samples shall be tested.

12. REPORTING OF TEST RESULTS

12.1 For those characteristics where a composite sample has been tested, only one test result shall be available and that result shall be reported as the value of the characteristic for the lot.

12.2 When only two laboratory samples have been analysed individually from a lot, the average of the two available test results shall be reported as the value of characteristic for the lot sampled. The individual test results shall also be reported to give an indication of the range of variation in quality.

12.3 When three or more laboratory samples have been tested individually, the average (X) and the range (R) of the test results shall be calculated as follows for assessing the average level and the extent of variation in the average:

If, for any characteristic, X_1 , X_2 X_n are the results of analysing *n* laboratory samples, then

Average
$$(X) = \frac{X_1 + X_2 + \dots + X_n}{n}$$

Range (R) = the difference between the maximum and the minimum of the test results.

12.3.1 The average level of the characteristic in the lot shall be reported as equal to X.

12.3.2 The limits for variation of the average quality in the lot shall be reported as $X \pm hR$, where h is a constant, the value of which depends upon the number of laboratory samples analysed (see Table 4), implying that the average quality of the material in the lot varies between these limits.

TABLE 4 VALUES OF THE CONSTANT, k				
NUMBER OF LABORATORY Samples Analysed, n	VALUE OF THE CONSTANT, h			
3	1•30			
4	0.72			
5	0.21			
6	0•40			

12.3.3 In case any of the test results deviates considerably from the others, it may be desirable to determine whether such a result may be rejected or whether it may be accepted as part of the normal variation expected. The procedure given in Appendix D may be followed for this purpose. If the suspect test result appears to be rejected, the average and the range shall be re-calculated from the remaining test results and appropriate inference drawn as in 12.3.2.

APPENDIX A

(Clauses 4.2.1 and 7.2)

RANDOM SELECTION OF WAGONS

A-1. GENERAL

A-1.1 To ensure the randomness of selection of wagons from a sub-lot, the use of random number tables as described in A-2 is recommended.

A-2. RANDOM NUMBERS

A-2.1 Two sets of random numbers are given in Table 5. The first set of random numbers shall be used if the number of wagons in the sub-lot is less than or equal to 100, whereas the second set shall be used if the number of wagons in the sub-lot is more than 100 but less than 1 000. Having selected the set, any one numeral shall be chosen from it at random. Starting from the selected numeral and continuing on with the numerals in any direction, right or left, up or down, the succeeding numerals shall be copied

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out one by one till the number of numerals copied out is equal to the number of wagons to be chosen. The numerals which are greater than the size of the sub-lot or which have already occurred shall be omitted. The numerals noted down in this manner shall then be arranged in the ascending order of magnitude.

A-2.2 Starting from any wagon in the sub-lot and counting them in one order, the wagons corresponding to the numerals already noted down shall be selected for drawing gross samples.

		TAB	LE 5 R	ANDOM	NUMB	ER TAB	LES		
				(Clau	se A-2.1)				
			First Se	et of Rar	udom Nu	mbers			
81	74	67	95	70	56	51	54	50	53
61	37	42	62	93	96	34	18	22	89
52	07	16	29	39	04	71	14	76	- 78
43	80	77	25	72	49	86	03	83	45
65	32	27	40	63	57	97	84	82	87
21	58	11	23	80	10	30	01	100	44
31	90	55	86	13	3 6	24	91	19	64
73	98	20	05	68	46	69	85	94	59
33	15	35	26	79	92	38	12	41	17
75	66	99	09	06	47	48	60	28	02
		Se	cond Se	t of Ra	ndom N	umbers			
288	302	957	018	109	053	044	058	849	285
898	732	965	943	462	554	146	318	313	540
090	553	340	096	870	654	605	967	968	085
370	252	657	094	698	056	813	728	351	266
619	151	079	473	763	886	097	893	506	662
573	866	835	785	689	529	992	283	964	416
304	855	222	564	247	726	626	370	569	002
759	996	232	804	271	605	536	173	607	504
020	357	975	079	547	746	272	659	500	487
039	821	904	130	633	750	57 9	419	722	753
395	463	995	067	695	681	279	885	746	493
121	7.32	123	413	978	8 82	953	243	007	380
876	871	606	733	298	482	384	482	801	332
.197	628	957	646	`776	436	936	237	913	581
376	581	715	933	367	497	659	196	884	954
227	967	689	687	405	412	088	466	685	099
305	216	474	185	280	576	273	320	270	564
226	712	640	231	789	872	395	896	237	514
238	786	906	967	245	315	316	029	024	692
036	461	087	973	762	208	505	849	449	440

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Example:

Suppose there are 60 wagons in a lot of approximately 1 200 tonnes. According to Table 1, this would require the division of the lot into 4 sub-lots, each consisting of 15 wagons of approximately 300 tonnes. Considering any particular sub-lot, if 25 percent of the wagons (see 4.2.1) have to be selected at random for drawing the gross sample, it shall imply that 4 wagons have to be selected out of 15 in the sub-lot.

A-2.3 Taking the first set of random numbers as given in Table 5, suppose, further that numeral 07 occurring in the third row under second column is chosen at random. Proceeding further in any direction, say right, and omitting the numerals which are greater than 15, the numerals encountered are 07, 04, 14 and 08. Arranging them in one order, the sequence 04, 07, 08 and 14 is obtained. It then follows that the 4th, 7th, 8th and 14th wagon counted from the beginning of the sub-lot shall be selected for drawing the gross sample.

APPENDIX B

(Clause 6.1.3)

EQUIPMENT AND PROCEDURE FOR PIPE SAMPLING

B-1. EQUIPMENT — The equipment (*see* Fig. 6) required for pipe sampling shall consist of a sampling pipe 120 cm long, 10 cm inside diameter and 0.5 cm wall thickness fitted with a 10 cm collar at the top and a separate steel beater. The pipe shall be tapered from 10 cm to 7.5 cm diameter within the bottom 15 cm and provided with 18 teeth at the bottom end which shall be bent slightly inwards. There are two holes in the collar, diametrically opposite to each other and of 2 cm diameter through which a 60-cm long rod of a slightly smaller diameter passes.

B-2. DRAWING OF SAMPLE — For drawing a sample, the sampling pipe shall be driven through the coal from the top downwards by means of blows with a 3-kg hammer on the beater inserted in the collar. If necessary, a rod shall be inserted through the holes in the collar to rotate the pipe between the blows to prevent jamming. After 115 cm of the pape has been inserted in the coal bed, the hammering shall be stopped. To withdraw the sample, the rod shall be inserted through the two holes in the collar and the pipe then pulled out with the help of the rod. The sample shall be collected on a clean strong cloth or tarpaulin, tapping the pipe, if necessary.

Nors — If the coal consists of fines and is dry, it is occasionally necessary to spray some water round the pipe to moisten the coal. This will be found essential only in exceptionally dry weather.



FIG. 6 COAL SAMPLING PIPE AND BEATER

APPENDIX C

(Clauses 10.1, 10.1.1, 10.2 and 10.3)

REDUCTION OF GROSS SAMPLES

C-1. GENERAL PRECAUTIONS

C-1.1 The place set apart for the treatment of gross samples shall preferably be enclosed, roofed over, cool and free from draughts. Where this is not possible, precautions shall be taken against (a) loss of fine wind-borne sample, (b) contamination with moisture, and (c) contamination with foreign matter.

C-1.2 Select a hard and clean surface free of cracks for sample mixing, quartering and other operations. Do not let cinders, sand, chippings from the floor or any other foreign matter get into the sample.

C-2. CONING AND QUARTERING

C-2.1 The material which has been crushed to 3.35 mm (see 10.1) shall be heaped into the shape of a cone by pouring one scoopful 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.

C-2.2 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 weight of 2 kg.

C-3. RIFFLING

C-3.1 The material which has been crushed to 3.35 mm shall be dropped uniformly in the riffle. One half shall be retained and the other half rejected. This procedure shall be repeated several times till 2 kg of material is obtained.

C-4. GRINDING (FINE SIZE)

C-4.1 In grinding the sample to pass 212-micron IS Sieve (see 10.1.1), it has been found that unnecessarily fine grinding is harmful. The ground coal should have the following approximate particle size distribution:

a) Passing 212-micron IS Sieve and retained on 125-micron IS Sieve 33

35 percent

b) Passing 125-micron IS Sieve and retained on 63-micron IS Sieve

30 to 35 percent Remainder

c) Passing 63-micron IS Sieve

APPENDIX D

(*Clause* 12.3.3)

CRITERIA FOR REJECTION OF SUSPECT TEST RESULTS

D-1. GENERAL

D-1.1 It may sometimes happen that a test result is obtained which deviates considerably from the other test results and therefore arouses suspicion that it may have arisen from a mistake in sampling or sample preparation rather than in the course of normal variation. If the analyst knows that a mistake has occurred, the test result must be rejected irrespective of its magnitude. If, however, only a suspicion exists, it may be desirable to determine whether such a result may be rejected or whether it must be accepted as part of the normal variation expected.

D-2. CRITERIA

D-2.1 The procedure as given in **D-2.1.1** may be followed for determining the acceptability or rejectability of the suspect test result.

D-2.1.1 Arrange all the test results obtained by analysing n laboratory samples in the ascending order of magnitude and designate them as x_1, x_2, x_n , when x_1 is the smallest and x_n the largest test results. If x_n is suspect, calculate the value of K_1 from

$$K_1 = \frac{x_n - x_{n-1}}{x_n - x_1}$$

If x_1 is suspect, calculate the value of K_2 from

$$K_2 = \frac{x_2 - x_1}{x_n - x_1}$$

If the calculated value of K_1 or K_2 exceeds the corresponding one given in Table 6, the suspect test result shall be rejected, otherwise not.

The above procedure can also be applied repeatedly to remove the suspect test results other than the one first removed.

TABLE 6 VALUES OF CRITERION K_1 OR K_2			
SAMPLE SIZE	VALUE OF K1 OF K2		
<i>(n)</i>			
3	0.941		
4	0.765		
5	0.642		
6	0-560		

D-3. EXAMPLE

D-3.1 In an investigation on ash percentage of coal, the following test results are obtained:

28.9, 26.9, 26.8, 26.7, 26.1

It is required to find out whether 28.9, which deviates rather unduly from the others, could be considered to be a suspect.

The test results, when arranged in ascending order, are: 26.1, 26.7, 26.8, 26.9 and 28.9

According to the criterion given in D-2.1.1,

 $K_1 = \frac{28 \cdot 9 - 26 \cdot 9}{28 \cdot 9 - 26 \cdot 1} = \frac{2 \cdot 0}{2 \cdot 8} = 0.714$

Since this value of K_1 exceeds the corresponding tabulated value 0.642 (see Table 6) for sample size 5, the test result shall be rejected.