A Review on Steam Coal –Sampling & Preparation
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Abstract: Coal have been classified into many types, grades, ranks etc. mostly on the basis of physico-chemical parameters, utility and commercial aspects. It is interesting to note that all the classification cited do not refer to Steam Coal. The entire range of all chemical properties are covered under the term steam coal, from lignite to anthracite. Coal is a biochemical sedimentary rock with very high heterogeneity in chemistry both organic as well inorganic components, different maceral constituents, diverse physical properties. Steam coal if blended to meet the commercial criteria, adds more complexity. The sampling of such heterogeneous mass is very critical. A review of the available methods of sampling and its possibility of true application by the end user has been reviewed.

Key Words: Coal, Classification, Sampling, preparation and test methods, Indian Standards (IS), ISO, ASTM

I. Introduction
Coal is a combustible black or dark brown sedimentary rock, fossil fuel and is formed from the decomposition of organic materials originally accumulated in swamps and peat bogs that have been subjected to geologic heat and pressure over millions of years, composed mostly of carbon and hydrocarbons. The degree of change undergone by a coal as it matures from peat to anthracite is known as coalification. The transformation of vegetable matter into peat and coal is commonly regarded as proceeding as two steps, called as biochemical and physicochemical stage of coalification [1] respectively. The results of this process, i.e. the type of peat and coal formed, depend on the phytogenic input and the environmental conditions under which it is transformed into peat. Different biological, chemical and physical constraints result in different peat types which during the subsequent physicochemical coalification are transformed into different coal types without losing their paleo-environmental signature. Coalification is a dehydrogenation process with a reaction rate slower by many orders of magnitude than that of carbonization. Coalification has an important bearing on coal's physical and chemical properties and is referred to as the ‘rank’ of the coal. Ranking is determined by the degree of transformation of the original plant material to carbon. The ranks of coals, from those with the least carbon to those with the most carbon, are lignite, sub-bituminous, bituminous and anthracite.

Many classifications of coal are available in literature. Any classification of coal should be scientific and systematic and should take into account the fundamental characters of coal. For example, (a) Classification by visual characters and classification based on their source of genesis was the initial way, which was changed by ultimate analysis: Regnault-Grüner-Brosquet System [2]. (b)Syler’s classification [3] the complete system was published in 1899. His classification divided coal into 7 carbon planes and 2 hydrogen planes, (c) Grout and Ralston classification: In 1907 Grout [4] plotted C (carbon), H(hydrogen), and O(oxygen) contents of American coal on a tri-axial diagram. The plot separated cannel coal (high H) from ordinary coal. Classification was based on dry-ash free analysis including fixed carbon and total carbon. (d) In1915 Ralston [5] extended the study and found coal of equal volatile matter (isovols) and equal calorific value (isocals) can be represented by straight lines in the triangle. (e) Frazer’s classification: in 1877, [6] he used fuel ratio to classify coal as given: Coals of lower rank than bituminous were not considered. Study was on only Pennsylvania coal. He divided four coal type depending on fuel ratio (FR) as anthracite 100-12 2, semi anthracite 8-12 3, semi bituminous 5-8 4. Bituminous 0-5. (f) Campbell classification: [7] also based on fuel ratio but all coals below 5 FR were taken as bituminous coal. In 1926, he combined fuel ratio to different characteristics for distinguishing lower rank coal. He divided four coal type depending on fuel ratio as anthracite 10-50 2. semi anthracite 5-10 3. semi bituminous 2.5-5 4. bituminous <2.5. Classification involving both proximate analysis and calorific value: (g) Parr’s classification [8]: considered volatile carbon, total carbon, inert volatile matter and gross coal index (C+ available H+ Sulphur). The basis was volatile carbon*100/total carbon and gave a new classification in 1928 as below - anthracite 0-8 15000-16500, semi anthracite 8-12 15000-16500, bituminous A 12-24 15000-16500, bituminous B 25-50 15000-16500, bituminous C 30-55 14000-15000, bituminous D 35-60 12500-14000, lignite 35-60 11000-12500 and peat 35-80 9000-110000. (h) A.S.T.M. Classification [9]: it classifies coal to 4 broad classes based on fixed carbon and calorific value (BTU) on dry mineral matter free basis. Applicable only to
vitrinite rich coal and excludes southern Gondwanaland coal. Gross heating value found on a moist and mineral matter free basis. Moist refers to the natural inherent water contained (MJ/kg X 430.11= Btu/lb.). Coals containing 69 wt. % or more fixed carbon on a dry mmf basis are ranked according to their fixed carbon content regardless of their gross heating value. (i) Classification by National Coal Board [10]: specifically designed for commercial use which is rank based. Uses 3-digit code to identify main class, class and subclass to which a coal belongs. Applicable to vitrinite rich coal, volatile matter in dmmf basis and Gray-King coke type values are considered. (j) International Classification of hard coal [11]: mainly for anthracites and bituminous coal but covers fairly kinds. Uses 14-digit code that defines 8 parameters namely: 1- vitrinite reflectance, 2- inertinite content, 3- exinite/limpetinite content, 4- caking property-free swelling index (FSI), 5-VM (volatile matter), 6-ash, 7- S (Sulphur) and 8- gross calorific value. (k) Classification of Indian Coal [12]: scientific coding of Indian coal has 3 basic parameters and 1 supplementary parameter: 1st digit (1-9) corresponds to calorific value (dmmf), 2nd (0-9) one volatile matter (dmmf), 3rd (0-5) one coke type, and 4th (1-6) one: maximum thickness of plastic layer for coking coal and M (moisture) % for non-caking. (l) Grading of Indian Coal [13]: For grading of non-coking coal useful heat value is used, which is calculated by HU= 8900-138(ash + moisture) kcal/kg, which excludes coal from North East India. (m) ISO Standard 11760, Classification of coals [14], was published in 2005. This classification system divides coals into three primary categories, low rank, medium rank, and high rank. The parameters used to classify the coals into the primary ranks and subcategories are vitrinite reflectance, vitrinite content, moisture, and ash yield.

Coal have been classified into many types, grades, ranks etc. mostly on the basis of physico-chemical parameters, utility and commercial aspects. **It is interesting to note that all the classification quoted do not refer to Steam Coal.** Steam coal, intermediate in rank between subbituminous coal and anthracite according to the coal classification used in the United States and Canada for boilers. In Britain bituminous coal is commonly called “steam coal,” and in Germany the term Steinkohle (“rock coal”) is used. While coking (metallurgical) coal and steam (thermal) coal have similar geologic origins, their commercial markets and industrial uses are vastly different. However, the fundamental difference between two types of coal is in their caking property. Non-coking coal (usually referred to as thermal coal) cannot form cake when heated in absence of air; whereas, when coking coal is heated in absence of air above 900 degrees (ash fusion temperature) the constituents start fusing and form a large chucky mass, knows as coke. A coking coal if not suitable for metallurgical purpose due to any physico-chemical constraint can be treated as steam coal and can be used as a blend. **So the entire range of all chemical properties are covered under the term steam coal, from lignite to anthracite.**

Coal is a biochemical sedimentary rock with very high heterogeneity in chemistry both organic as well inorganic components, different maceral constituents, diverse physical properties. Steam coal if blended to meet the commercial criteria, adds more complexity to the physico-chemical properties. The sampling of such heterogeneous mass is very critical. Many published literatures and standards are available for sampling of coal either addresses hard coal or lignite coal, which are not essentially followed by the industry. The purpose of this paper is to examine ways steam coals are sampled and prepared by the end user laboratory, identify the gap in standard & actual practice and what measures to close the opening existing out of inherent shortcomings or need a change in sampling methods, which can improve the quality of the coal it burns for electric generation, thereby gaining both environmental and economic benefits.

### II. Sampling Location

**An estimation of the true value of the desired parameters of a bulk material, to a certain degree confidence, through analysis on a few grams of test sample is definitely a daunting problem.** Location at which the sample is collected is crucial for its representation to the original coal. Coal can be sampled at various locations in a power plant which depends on the objective of the sampling followed by accessibility to coal and coal handling plant layout and method of sampling. It can be done to understand “Incoming Quality” from the delivery vehicles, conveyer belt, feeder to silo or bunker or stockpile, discharge from crusher; or to know the “As fired” quality by taking samples from silo out let, the pulverizer outlet etc. Both the qualities are important for the plant to monitor the plant efficiency with respect to feed quality irrespective of the blend or storage changes. Most of the power plant use blend of different sources and qualities of coal. It’s not only the chemistry of coal, the physical and mineral matter constituents of coal also play an important role in selecting the sampling point. As far as any standard goes, the sampling location is not stringent although it is essential. All the standards should have been strict on this aspect as it plays a major role in deciding the representativeness of the coal if it is sampled for incoming quality where external agency is involved and commercial aspects are obvious. Even a perfect analytical procedure cannot rectify the problems created by faulty sample collection. A good sampling plan will ensure that the samples obtained will, on average, closely represent the bulk composition of the coal being measured. In addition, the sample must be collected and handled in a such way that its chemical composition does not change by the time it is analyzed. Finally, the sampling must be done with the requirements of the analytical method in mind. It is always good to know as much about the sampling site as possible, especially about the sources of the errors being investigated, and the mechanisms for their isolation. Another important consideration in sampling is the physical environment like weather conditions, air temperature, humidity, wind
speed, rainfall, from both source as well as the point of sampling etc. Normally any sampling scheme is supposed to conform to relevant national or international standards. However, due to technical, cost and time constraints, very often some modifications are made in the method of sampling jointly by the seller and the purchaser. It is a known fact that about 80% of the total variances involved at the different stages of sample collection, preparation and analysis comes from errors during its collection only. The sampling plan describes the overall aims and objectives; it includes specific and practical instructions on what is going to be sampled, how it will be sampled, at what frequency, what the sample will be analyzed for and by whom. An appropriate sampling plan provides transparency to all users and will not only improve the reliability of the results and the level of assurance; it may also help to reduce costs for analyses and verification.

Here are few criteria that must be followed when sampling to ensure the overall precision and accuracy of the results:

- Who is responsible for each step?
- Where and when are samples taken?
- How are the samples taken? E.g. it might be necessary to first clean the system where residues from previous samples might still be contained, etc.
- Which instruments are used, if relevant? Describe automatic sampling equipment, but also describe the tools for manual sampling. It might also be important how samples can be picked out from sufficiently deep in a pile of several meter height.
- How will the identity of the samples be ensured?
- How are the samples stored (dry, cool, dark, inert atmosphere, etc.)?
- How and when are increments combined?
- When are the samples analyzed, are remaining samples stored after analysis, etc.?
- Ensure that the sample is representative of the bulk material, which means all parts of the material being sampled must have an equal probability of being collected and becoming part of the final sample for analysis,
- Make sure that the sample does not undergo any chemical or physical changes after completion of the sampling procedure and during the storage prior to analysis
- The sample size must be adequate
- Choosing appropriate sampling locations also depends on whether coal is received in a batch or a continuous process.
- A sampling point must be reasonably accessible to be an effective location. Sampling cannot be performed from a location that cannot be reached. It should be recognized that the best sampling point may not be accessible and that sampling will need to be performed at the next best point of accessibility. Accessibility and safety are related in that a sampling point may be physically accessible, but sampling from that location may present a risk of injury. Once chosen a sampling location, identify the potential risks associated with that location, take the appropriate safety precautions, and provide protective equipment.
- Sampling point has an impact on the granulometry of actual vs collected sample
- Chute and belt sampling is most economical and representative
- It is always desirable to have a second sampling point for cross verification
- Assess variability in coal to be sampled: Variability can be distinguished between
  - Spatial variability - This term refers to the heterogeneity of a material depending on the location, e.g. the heterogeneity within one single batch
  - Temporal variability - This term takes into account changes of properties over time,
- Based on historical data, any variation in the analytical values for the respective fuel or material does not exceed 1/3 of the uncertainty value to which the operator has to adhere with regard to the activity data determination of the relevant fuel or material

### III. Sampling Method

The sampling of coal, whether performed manually or mechanically must extract a quantity of coal much similar than the original lot but with proportionately the same quality. But coal quality is not always uniform, and variability makes it difficult for representativeness. Preliminary to any laboratory testing of coal, it is imperative that a representative sample be obtained; otherwise, the most carefully conducted analysis is meaningless. Reliable sampling of a complex mixture such as coal is difficult and handling and preparation of the sample for analysis presents further problems. Variations in coal handling facilities make it practically impossible to publish a set of rules that would apply to every manual sampling situation. The proper collection of the sample involves an understanding and consideration of the minimum number and weight of increments, the particle size distribution of the coal, the physical character and variability of the constituents of coal, and the desired precision. The selection of a sampling method depends upon factors such as the sampling purpose, accuracy desired, accessibility of the site and technical, economic and time constraints. Taking a representative sample
manually from material that is stationary involves very great difficulties and almost invariably can only be realised in a limited manner. Manual sampling techniques, although subject to errors associated with human discretion, may be avoided to effectively collect samples of definable quantity. Automatic sampling system, once designed, installed and tested for a specific plant application will surely produce representative samples in terms of quality as well as cost.

The sampling error is by far the largest component of the total error in the analysis of coal samples. Generally, 80% of the total error is due to some aspect of sampling [15]. Sampling errors are two types – Random and systematic. Random error covers – isolated changes in process, coal heterogeneity, sample quantity etc. But interestingly random errors move both positive and negative direction around mean value and mostly averages out. However, systematic error mainly caused by inaccuracies in the sampling mechanism. This is known as sampling bias. Every end user of coal should consider the sampling bias, which can be done by a statistical comparison of the data from coal collected by the end user lab method and ASTM Stop-belt technique [16]. Hardly, any such data has been generated by the power plant lab to substantiate the bias in the sampling. This should have been a practice with frequent interval for comparison and establishing the sampling bias and it must be an essential part of the standards with defined frequency for comparison. The sampling personnel should also record coal feed rate, sampling speed, number increment, number of increment per averaging period, sampler dimension, total lot size, maintenance breakdown history.

III.A INDIAN STANDARD (IS)

Manual Sampling

Indian Standards IS: 436 (Part I/Section 1)-1964 (Reaffirmed 2013) [17] for manual sampling, specifies in paragraph 0.3.4.2: “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”.

The IS method covers - sampling from conveyers, sampling from wagons during loading or unloading: (These increments shall be drawn with the help of a suitable scoop or shovel, depending upon the size of the coal, at regular intervals at the time of loading or unloading of the wagons, covering at least 25% of 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), sampling from ships during loading or unloading, sampling from stock pile (the surface of each sub-lot shall be levelled and one point for approximately every 250 metric tons of material in the sub-lot shall be chosen at random. 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. Rarely this sampling procedure is being followed in totality. The reasons are best known to the institutions carrying out the job. Whatever the possible explanations, if a standard method cannot be practiced in totality, we should think the way out for the alternative solutions and amend the standards accordingly, instead of placing a standard name sake in paper.

Mechanical sampling


III.B ISO STANDARD

Manual Sampling

ISO 18283 [21] defines the basic terms used in manual sampling of hard coal and coke and describes the general principles of sampling. It specifies procedures and requirements for establishing a manual sampling scheme, methods of manual sampling, sampling equipment, handling and storage of samples, sample preparation and a sampling report. This International Standard applies to manual sampling from fuels in movement. Guidelines for manual sampling from fuels in stationary situations are given in Annex B, but this method of sampling does not provide a representative test sample and the sampling report shall state this. ISO 18283 does not include sampling of brown coals and lignites, which is described in ISO 5069-1:1983 [25] and ISO 5069-2 [26], nor sampling from coal seams, which is given in ISO 14180[27].

Mechanical sampling

Mechanical sampling of hard coal and coke is covered in ISO 13909 [24] (all parts). In India very limited use of mechanical sampler is in practice by power plants or suppliers.

III.C ASTM STANDARD

Part of Committee D05 on Coal and Coke, the subcommittee’s six task groups develop and maintain guides, practices, and test methods for sampling of coal and coke derived from coal. For coal sampling ASTM has got following standard test methods -
- D2234/D2234M: Practice for Collection of a Gross Sample of Coal (followed by D2013 Practice for Preparing Coal Samples for Analysis) [28]
- D 4916: Standard Practice for Mechanical Auger Sampling; [29]
- D 6543: Standard Guide to the Evaluation of Measurements Made by On-Line Coal Analyzers [31]; and
- D 6609: Standard Guide for Part-Stream Sampling of Coal. [32]
- D7256/D7256M: Practice for Mechanical Collection and Within-System Preparation of a Gross Sample of Coal from Moving Streams [33]. The new standard ASTM D 7256/7256M is a combination of several previous sampling standards and sample preparation ASTM Standard D 2013—Method of Preparing Coal Samples for Analysis [34]
- D 7430: Standard Practice for Mechanical Sampling of Coal [35]

Historically, the U S Steel Corporation advise removing full cross sectional cut from a stopped belt conveyor or moving stream, but discourage sampling from railroad cars and other stationary sources [36]. The accuracy of sampling procedure can be checked by running a bias test between stopped belt cut and stationary manual sampling. Both automated and manual sampling can be done at various sampling locations as below with a predefined sampling scheme:

- Sampling from moving streams
  - Sampling from a falling stream
  - Sampling from a moving belt
  - Stopped belt sampling

- Sampling from stationary coal
  - Sampling from stockpiles
  - Sampling from wagons, barges and ships

| Table 1 Number and weight of increments for general purpose sampling procedure (ASTM D2234/2234M) |
|---------------------------------|-----|-----|-----|
| Top size, mm                   | 16  | 50  | 150 |
| Mechanically cleaned coal†     |     |     |     |
| Number of increments           | 15  | 15  | 15  |
| Minimum weight of increments, kg | 1   | 3   | 7   |
| Raw (uncleaned) coal†          |     |     |     |
| Number of increments           | 35  | 35  | 35  |
| Minimum weight of increments, kg | 1   | 3   | 7   |

For coal above 150 mm top size, the sampling procedure should be mutually agreed upon in advance by all parties concerned† The fundamental requirements of sampling have been explained in OAR/EPA [37]. If there is any doubt as to the condition of the preparation of the coal (for example, mechanically cleaned coal or raw coal) the number of increments for raw coal shall apply. Similarly, although a coal has been mechanically cleaned, it may still show great variation because of being a blend of two different portions of one seam or a blend of two or many different seams. In such cases, the number of increments should be as specified for raw (uncleaned) coal. Sampling should be carried out by systematically sampling either on a time--basis or on a mass--basis, or by stratified random sampling. The interval between primary increments (Table 1) depends on the size of the sub-lot and the number of primary increments in the sample, and should be determined in accordance with relevant standards. This interval should not be changed during the sampling of the sub-lot. Following are the points to be considered in the selection of a sampling method which depends upon factors such as the sampling purpose, accuracy desired, accessibility of the site and technical, economic and time constraints. Precision is the closeness of the data to the true value in given conditions as indicated by the reproducibility of the unbiased results. Sampling precision depends on variability of coal, number of samples from a lot, number of increments comprising each sample, and mass of sample relative to the nominal top size. The testing laboratory should have the supporting data of its precision.

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<th>Table 2 Preference Order of coal sampling procedures and methods</th>
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<td>Sampling procedure</td>
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<td>Stopped Belt Cut</td>
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<td>Full stream cut</td>
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<td>Stationary Sampling</td>
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**Manual Sampling** – top size, increment design, plan and lot system, frequency of sampling, how to collect a sample from segregated lot, how to access the center point of stock otherwise its peripheral sample, dimension of sampling tool should be sufficient to allow the largest particle to pass freely into it, the first stage of sampling known as primary increments is the collection of an adequate number of coal portions from positions distributed...
over the entire lot, minimum mass of the gross sample relative to the nominal top size, randomness of check and of course the knowledge, experience and safety of sampler. Manual method of wagon top sampling of large sized coals is not only difficult but also violates some of the fundamental principles of sampling. As per requirement samples are to be drawn from the full depth of the wagons which is impossible to be collected manually. Furthermore, due to size segregation the samples collected from the wagon top does not satisfy the criteria of representativeness of the whole samples. Since the ash and mineral matter distribution in the different size fractions of coal or blended steam coal is not homogeneous, results from the samples which do not reflect the true size distribution of the lot are likely to be biased. More importantly, sample collection by a shovel from the top or bottom discharge is a function of human discretion and not governed by the probability rule. Wagon sampling when practiced in other parts of the globe is done on smaller and uniform sized coals, generally washed and blended, and preferably by auto-mechanical auger systems, not by manual methods which is less prominent in Indian context and possibly the major contributor of deviation in test results among the laboratories (Table 2). Coal samples can also be taken from a moving conveyor belt. Manual sampling from stationary coal such as a coal storage pile or railcars is, sometimes, necessary but this is problematic and less representative. Some standards, such as ISO 13909-1 [24] stipulate that manual sampling should not be exercised in cases where the nominal coal top size is above 63 mm or when the coal flow rate is greater than 100 tons per hour. Certainly, for a manual sampling, when the belt stands still, the calculation takes into consideration the length of the conveyor belt on which the material is sampled.

**Mechanical Sampling** – material flow rate, sampling speed, particle size distribution, sample cutter speed and dimension, in addition to top size and segregation, lot size and shape, sampling variance, location of sampler etc.

### IV. Sample preparation

The distribution of mineral matter in coal presents problems for the crushing, grinding, and uniform mixing at each step of the sampling procedure. The various densities of the materials found in coal can easily cause their segregation, especially if there is a wide range of particle sizes. Crushing and grinding coal, or both, from a large particle to a very small particle in one operation tends to produce a wide range of particle sizes and a high concentration of very fine particles. The crushing, grinding, and pulverizing should involve a reasonable number of steps, considering the starting particle size and nature of the coal. The samples obtained by quartering on the cement floor were consistently higher in ash than those obtained from the sampling machine, but when the quartering was done on oil cloth the agreement was very close. This would indicate that the high ash in the quartering samples was due to floor dust but more samples should be obtained by quartering on oil cloth before a definite conclusion can be reached. Care to be taken for – feed size, type of crusher, crusher capacity and uniformity in feed rate and product size, step size reduction with screening and atmospheric equilibration, crusher cleanliness to avoid contamination, proper storage of crushed product to avoid any atmospheric loss, room cleanliness, air flow, room temperature & humidity fluctuations etc. Any such variable, which can impact the physico-chemical properties of coal should be monitored and recorded in the lab and its influence is to be quantified as an error or uncertainty in measurement. To minimize the moisture problem, all standard methods include, when necessary, an air-drying stage in the preparation of the analysis sample so that subsequent handling and analysis will be made on a relatively stable laboratory sample with reference to gain or loss of moisture from or to the laboratory atmosphere. The distribution of mineral matter in coal presents problems for the crushing, grinding, and uniform mixing at each step of the sampling procedure. The various densities of the materials found in coal can easily cause their segregation, especially if there is a wide range of particle sizes. Too many handling steps will increase the exposure of the coal to air and increase the chance of moisture changes and oxidation in low rank coals.

**ASTM Method**

### IV.A Gross Sample

In ASTM gross sample is defined as a sample representing a quantity, or lot, of coal and is composed of a number of increments on which neither reduction nor division has been performed.

A lot is a discrete quantity of coal for which the overall quality to a particular precision needs to be determined. Minimal quantity of sample depends on top size, mineral characteristics, variation in the valuable component content, coal density, grain shape, content, uniformity and size of mineral matters. For quantities of coal up to approximately 1000 tons it is recommended that the one gross sample represent the lot. The number of increments to be taken for the gross sample depends on the type of coal being sampled (35 for raw coal and 15 for mechanically cleaned coal). The size of each increment depends on the top size (granulometry) of the coal being sampled. The ASTM general purpose sampling procedures are designed to give a precision such that if gross samples are taken repeatedly from a lot or consignment and one ash determination is made on the analysis sample from each gross sample, 95 out of 100 of these determinations will fall within ±10 % of the average of all determinations.
IV.B Laboratory Sample

Once a gross sample has been taken, it is reduced in both particle size and quantity to yield a laboratory sample. The particle size distribution, or nominal top size, of the laboratory sample depends on its intended use in the laboratory and the nature of the tests to be run. The minimum allowable weight of the sample at any stage of reduction depends on the size consist, the variability of the constituents sought, and the degree of precision desired.

IV.C Analysis Sample

The subsample is reduced to 100% through a number 60# (250 µm) sieve and then divided to not less than 50 g, which is called the analysis sample and is required for most ASTM laboratory tests.

Many problems may arise during the sampling and sample preparation processes [38], such as

- the loss or gain of moisture, due to uncontrolled lab atmosphere
- improper mixing of constituents, due granulometry & HGI (in blended coals), feed rate and crusher type
- improper crushing and grinding due to mineral matter, feed rate and crusher type etc.
- contamination of the sample by equipment improper cleaning
- oxidation of coal due to atmospheric exposure

As the crusher wear, the top size of the crushed product will grow and hammer/screen replacement should take place before this exceeds acceptable limits and clearance between wear plates and the rotor assembly can also influence the top size of the crushed product. The major sampling risk of poor crusher inspection & maintenance to the moisture integrity of the coal – either directly through excessive drying or indirectly through improper save sample sizing contributing to sample preparation error.

To minimize the moisture problem, all standard methods include, when necessary, an air-drying stage in the preparation of the analysis sample so that subsequent handling and analysis will be made on a relatively stable laboratory sample with reference to gain or loss of moisture from or to the laboratory atmosphere. [39]

Coal is susceptible to oxidation at room temperature, especially the low rank steam coal. Like moisture changes, such oxidation has to be considered in sampling, preparing, and storing samples. Comparison of moisture and ash-free MAF (Kcal/kg) values is often useful for evaluating suspected oxidation problems. Containers should be selected that will hold only the required amount of sample and leave a very minimum of air space. Even when such precautions are taken, the samples change very quickly, so the analysis of a sample should be carried out as soon as possible after it is received.

V. Conclusion

In analyzing coal samples for their chemical composition, it is apparent that certain current standard test methods for sampling and sample preparation either require modification or they are not fully applied by the end users of coal. Obtaining a representative sample implies that every particle has a chance of being selected. A correct and representative sample requires that every particle in a lot be sampled is equally represented. A representative sample is collected by taking a definitive number increments, periodically throughout the entire coal lot being sampled. The number and weight of increments required for a desired degree of precision depends on the variability of the coal which increases with increasing impurities. The sampling of coal can take place from either stationary lots or from moving streams. Sampling from stationary lots is particularly problematic because in many cases it is not in compliance with the fundamental sampling principle stipulating that all parts of the lot being sampled must be accessible for physical sampling. Therefore, sampling from moving streams is preferred. The best location for sampling from a moving stream is at the discharge point of a conveyor belt or chute, that is a falling stream where the complete stream can be intersected at regular intervals. However, cross-belt cutters are now more popular and are widely used in the coal industry. Accurate coal sampling of it requires that you obtain all the various size gradients it contains in their proper proportion with each sample increment. While this is impossible to accomplish from a stockpile or from the top of a truck, railcar or barge, it can be accomplished from the surface of a conveyor belt. The stopped belt sampling, when properly executed, is considered as bias free and is recommended by several standards as a reference sampling method when carrying out a bias test procedure. This should have been a practice with frequent interval for comparison and establishing the sampling bias and it must be an essential part of the standards with defined frequency for comparison. Thus, the summary or the total error of sample preparation, excluding the sampling error, consists of the error of the sample division due to the inadequate homogenization during the treatment and error from insufficient number of pieces in the sample, i.e. the inadequate sample mass, as well as the error of the chemical analysis [8]. Since the error from the insufficient sample mass is included in the total error, and because the error resulting from the inaccuracy of the sample division is insuperable, the former error is not possible to determine. Still, a careful operation of the sample division during the homogenization can reduce the specific error to a constant value. Consequently, the variation of the total error from one set to another will be a result of the disproportion between the sample mass and the grain size in the mixture. All sampling systems should be checked for bias, because systematic errors may be introduced. These systematic errors generally are caused by a loss or gain in the mass of increments during collection or by cyclical variations of coal quality at time intervals coinciding with systematic sampling time. Bias testing is
discussed in certain standards, such as ISO/DIS 13909 Part 8 [24] for bias testing of mechanical samplers and in ASTM D4702 [30] with guidelines for inspecting cross-cut, sweep arm, and auger mechanical sampling systems. Coal analysis, and possibly in most new uses of coal, will require higher standards of quality control and accuracy than are currently quoted in many of the existing standard test methods and practices by the laboratories in India. These factors will become increasingly more important when economic decisions must be made based on the validity, i.e., Accuracy, Bias, Interlab Tolerance, Measurement Uncertainty of coal analysis data ISO’s Published Document — Guide to the Expression of Uncertainty in Measurement (1995) (known as GUM); published in 1993, reprinted with corrections in 1995) [40-42]. It’s worth to mention here that many of the coal testing laboratories are accredited to ISO 17025 "General Requirements for the Competence of Testing and Calibration Laboratories" [43], states that " where applicable, a statement on the estimated uncertainty of measurement; information on uncertainty is needed in test reports when it is relevant to the validity or application of the test results, when a customer's instruction so requires, or when the uncertainty affects compliance to a specification limit; ". It has been seen that the total cost of a sampling system in a coal handling plant is very low in comparison to the overall cost of the plant (less than 1%) is either not included in the system and if included is put out of commission for various reasons which could be easily over-come. Automatic samplers although slightly higher initial cost can save many hours of costly operator-time while producing reliable and representative sample. In most cases investment in sampling system will be still a fraction of what is expended on the analysis, but the analysis can only be as reliable as the sample delivered to the laboratory. Ultimately, all of us agree to the fact that sampling is not an art but processes and methods that characterize it neither depend on contingent social and ethical values, nor on the individual bias of a scientist but part of science.

VI. References

[10] The Coal Classification System Used by the National Coal Board. Revision of 1964, https://books.google.co.in/books/about/The_Coal_Classification_System_Used_by_the_National_Coal_Board.html?id=d8oAQwAACAAJ&redir_esc=y
[17] Indian Standards IS: 436 (Part I/Section 1)-1964 (Reaffirmed 2013)
[33] ASTM D7256/D7256M-08, Standard Practice for Mechanical Collection and Within-System Preparation of a Gross Sample of Coal from Moving Streams (Withdrawn 2008)
[34] ASTM D2013 / D2013M – 12, Standard Practice for Preparing Coal Samples for Analysis
[36] U.S. Steel Corporation, Sampling and Analysis of Coal and Coke, Pittsburgh, PA, 1929, 334pp


[42]. JCGM 106:2012, Evaluation of measurement data – The role of measurement uncertainty in conformity assessment

[43]. ISO 17025-2005, General requirements for the competence of testing and calibration laboratories