



A Review on Steam Coal Analysis -Moisture

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Abstract: Many methods have been developed for determining the moisture content of coal. In this paper discussion is done on the practical applicability, inherent limitations and omissions in laboratory practices of IS, ISO and ASTM test methods for moisture estimation in steam coal. Also highlighted the necessary precautions in a laboratory while conducting the moisture test. Emphasis also was given on the measurement uncertainty budget.

Key Words: Coal, test methods, Total moisture, Inherent moisture, Equilibrium moisture, Indian Standards

I. Introduction

Coal contains bio-chemical energy that is converted at a power plant into electric energy with determined efficiency. The efficiency is dependent, among others, on coal quality parameters, since the efficiency of the boiler is mainly a function of gross calorific value in coal. Moisture may be held in coals in several distinct forms: superficial free water; capillary condensed water; sorbed water, water associated with polar groups and cations; and water released only by chemical decomposition of organic or inorganic matter [1]. The surface moisture results from water held on the surface of coal particles or macerals. Hygroscopic moisture is caused by water held by capillary action within the micro-fractures of coal. Decomposition moisture is due to water held within the coal's decomposed organic compounds. Mineral moisture is a result of water which comprises part of the crystal structure of hydrous silicates, especially clays minerals. Moisture re-adsorption capability of the dried coal sample is dependent on coal particle size, relative humidity of the atmosphere and the temperature at which the coal was dried. Moisture is usually determined as total moisture, calculated as the loss of weight between the untreated and analyzed samples. One of the variables influencing the performance of a coal-fired power plant is the moisture content of the raw coal which is directly proportional to its calorific value. There are many moisture concepts in coal analysis and its impact on power plant performance as stated below -

- Surface moisture: water held on the surface of coal particles. Surface moisture values are really estimates, never accurate [2]. These are obtained by subtracting equilibrium moisture from total moisture. However, there is no sharp dividing line between inherent moisture and surface moisture. The measurement of inherent moisture depends on the fact that its vapor pressure is less than that of surface moisture. Surface moisture is that which occurs on the surface of the coal particles in excess of the inherent moisture. It is also called free water or extraneous water. Its vapor pressure is that of water for the existing temperature. SM is an added quantity and can vary in any range. gives rise to difficulties in handling (transfer and flow ability) of coal with severe capacity reduction of all equipment in the coal plant ranging from crushers to conveyors.
- It is commonly thought that inherent moisture is contained in the pores and capillaries of coal. However, these pores and capillaries may vary in diameter and size to such an extent that the water in the larger capillaries has a vapor pressure approaching that of surface moisture. Thus, moisture in the larger pores behaves like surface moisture and is "lost" during the equilibrium moisture test. Since pore size increases as rank decreases low-rank coals are more problematic regarding equilibrium moisture [2]. Theoretically, inherent moisture is that which a coal can hold when in equilibrium with an atmosphere of 100 percent relative humidity. IM or equilibrated moisture is a non-controllable parameter and affects the coal combustion process. Inherent or equilibrium moisture is used for calculating moist mineral-matter-free calorific values for the rank classification of high-volatile bituminous coals. It is also used for estimating free or surface moisture, since total moisture is equal to the sum of the inherent moisture and the free moisture. The inherent moisture value is also referred to as bed moisture, since it is considered to be the moisture of the coal as it occurs in the unexposed seam, where the relative humidity is 100 %. As mentioned above, equilibrium moisture provides a fairly accurate estimate of the inherent moisture in high-rank coals but the same is not true for low-rank coals where the equilibrium moisture is usually less than the inherent moisture. The theory is that the inherent moisture is held in the capillaries of the coal substance

and the vapor pressure of the moisture is somewhat less than that of water on a plane surface. More energy is therefore required to expel this moisture than to evaporate water from the surface of the coal particles.

- Hygroscopic moisture: water held by capillary action within the micro fractures of the coal.
- Mineral moisture: water which comprises part of the crystal structure of hydrous silicates such as clays. Moisture in coal consists of inherent moisture (IM) and surface moisture (SM). There is no simple and reliable method of determining the water of hydration of mineral matter. The average value of 8 % of the ash is used as the value for water of hydration of mineral matter in coals in the United States. This value is acceptable, although it is an average of values that range from 2–3 % up to 15–30 %. Water of hydration values are used to correct ash to the form of hydrated minerals in mineral matter calculations.
- Then total moisture (TM) is a sum of IM and SM. Total moisture in coal is used to determine the amount of drying that is needed to reach a given moisture requirement and to determine the amount of dustproofing and freeze-proofing agents to add. In coking processes, coals with a high moisture content requires more heat for vaporization of the moisture, which leads to longer coking cycles and decreased production. The total moisture of the coal used must be accurately known to allow for proper charging of the coke ovens and overall control of the coking process. Hence TM is affected by the criticality of SM. This brings down the GCV of coal (thermal content of coal)
- Equilibrated Moisture means the moisture content, as determined after equilibrating at 60% relative humidity (RH) and 40 °C as per the relevant provisions (relating to determination of equilibrated moisture at 60% RH and 40 degrees Celsius) of BIS 1350 of 1959 [3]). The moisture content of air-dried coal varies and depends upon the temperature and relative humidity of the air to which it is exposed. As such it is necessary to determine moisture content of different samples of coal under standard conditions. For this purpose, the coal is ground to pass 212-micron IS sieve and equilibrated in an atmosphere of 60 percent relative humidity and 40 °C. The moisture determined under these conditions shall be taken as reference moisture for all purposes [4].
- Moisture in coal equilibrated at 96 percent relative humidity and 40 °C- This is also termed as 'Near Saturation Moisture' or 'Bed Moisture'. It is exclusive of free or visible water and is determined after equilibrating coal in an atmosphere of 96 percent relative humidity and 40°C. This is a measure of the moisture holding capacity of a coal [4].
- Free water or visible water- Free water or visible water is that quantity of water which is physically adhering to coal. In essence, this is that quantity of water which is in excess of the moisture holding capacity of a coal. [4].
- Moisture in air-dried laboratory analysis sample of coal and coke - It is the moisture in coal which has been air-dried under the laboratory atmospheric condition prior to analysis and is determined as a part of proximate analysis and also whenever portions of sample are weighed later for other analysis and tests. If all the portions for analysis are weighed under approximately the same conditions of humidity, one determination of moisture will suffice, but a check is desirable with high moisture coals [4].
- Decomposition moisture is water held within the coal's decomposed organic compounds, produced from the organic portion of the coal through thermal decomposition, which has been shown to be negligible up to a temperature of approximately 200 °C to 225° C. It is not commonly dealt with in ordinary coal analysis because the temperatures for expelling moisture are below these values.

The moisture impact on power plant performance as stated below-

- There are three cost effects of moisture in coal:
 - Increase in operation costs due to decreased boiler efficiency and decreased overall unit efficiency (increase in heat rate)
 - Increase in operation and maintenance costs attributed to handling of wet coal.
 - Decrease purchase cost of coal due to higher moisture and hence lower GCV
- While internal moisture affects the coal combustion process, external (mechanical) moisture gives rise to difficulties in handling (transfer and flow ability) of coal with severe capacity reduction of all equipment in the coal handling plant ranging from crushers to conveyors.
- External moisture also creates combustion difficulties by creating thermal lag during the combustion process.
- Retardation of coal flow through the systems results in capacity reduction. When the surface moisture of coal exceeds 6%, it becomes sticky in addition to the stickiness created by the clay content of the mineral matter leading to severe capacity restriction in the tipplers, conveyors, crushers, bunkers and mills. The effective flowable coal through bunkers gets restricted to only 20% of the bunker volume in its center.

- Even though the bunker level may be full, only 30% of the bunker capacity can be utilized due to bonding of coal at the bunker periphery and flow is only through rat hole in the bunker center. Choke up on full level can be quite difficult to release.
- The pulverized coal throughput is largely affected by the amount of inherent and surface moisture present in the coal. Due to climatic variations, there is change in "as received" to "as fired" moisture in ROM coal which needs correction for optimizing the coal feeding.
- The presence of high levels of moisture results in higher transportation costs, greater energy requirements, increased off-gas volume, lower efficiencies, increased maintenance costs and increased friability of the coal, which interferes with separation, blending and pneumatic transportation.
- High fuel moisture results in fuel handling problems, and it affects heat rate, mass rate (tonnage) of emissions, and the consumption of water needed for evaporative cooling.
- Fly ash from steam coal may have high electrical resistivity, making it difficult to collect in electrostatic precipitators.
- Mineral matter in lignite is largely organically bound and inseparable by standard washing techniques.
- The high sodium content contributes to boiler fouling and slagging problems.
- The quartz content in lignite steam coal accelerates erosion of furnace burners.

II. Methods of Moisture estimation in Steam Coal

Many methods have been developed for determining the moisture content of coal. Most of these methods can be included in the following categories: [5,6,7]

- a) Thermal drying method
- b) Desiccator method
- c) Distillation method
- d) Extraction and solution method
- e) Chemical method
- f) Electrical method
- g) Nuclear method
- h) Microwave absorption
- i) Thermogravimetric Analysis (TGA)

Most common tests for moisture involve a thermal drying procedure, [8]. usually at a temperature a few degrees above the boiling point of water; the moisture released upon heating is measured either directly or indirectly. Thermal drying includes both drying in conventional ovens and microwave ovens, where the moisture is lost through vaporization following heating. The direct method involves the gain in weight of a weighing tube packed with desiccant through which the gases evolved from heating a coal sample are passed. This is probably the more accurate method, since only water is absorbed by the tube, while other evolved gases, such as methane, are not. The indirect method is more often used, primarily because it is easier to do. The moisture is taken as the weight loss of a coal sample upon heating in various atmospheres. If the coal is susceptible to oxidation, as are some low-rank coals with a high moisture content, the heating can be done in an inert atmosphere. The drying of most high-rank coals in air is accepted practice.

After thermal drying methods, distillation methods are the next most commonly used [9]. In these procedures, coal is heated in a liquid that has a boiling point higher than that of water and is immiscible with it. Xylene, toluene, or a petroleum fraction of a selected boiling range, are the liquids normally used. The distilled vapors are condensed in a graduated tube, and the volume of water is measured after the two liquids separate. Distillation methods are considered particularly advantageous for low-rank coals, since air is excluded from the coal, which minimizes the error due to oxidation. This is also a direct method of measuring moisture, and consequently there is no error due to the loss of other gases.

A non-thermal method of determining moisture involves the use of an extraction procedure in which the coal is shaken with a solvent that extracts the water from the coal. The degree of change in some physical property of the solvent, such as density, is then used as a measure of the water extracted.

Desiccator methods [7,10] for determining moisture involve the determination of the loss in weight of a coal sample in the presence of a desiccant. Either a normal or reduced pressure vacuum desiccator may be used, but the drying is carried out at room temperature.

Chemical methods [11] used for determining moisture include the application of the Karl Fischer method of determining water content, and the reaction of quicklime with water in coal and the subsequent measurement of the heat generated by the reaction.

Electrical methods of measuring coal moisture involve the determination of the capacitances or the resistances of quantities of coal. These methods have been used by industry, particularly for moving streams of coal. Low-resolution proton nuclear magnetic resonance spectroscopy is a rapid and accurate technique for measuring moisture in coal and total solids in aqueous coal slurries now appears possible [12]

At the Morgantown (W. Va.) Coal Research Center [13], a nuclear method for continuous measurement of moisture in coal was developed. This method is based on the thermalization of fast neutrons by hydrogen in the water and organic matter of coal. Neutrons from a small radioisotope source penetrate the coal, are scattered by hydrogen and measured by a thermal neutron detector. The number of thermal neutrons counted can be directly correlated with the moisture content of coal. Moisture meter based on neutron thermalization depends on many variables, any or all of which can affect the sensitivity of the meter. These factors include those related to the nuclear aspect--type and size of neutron flux and source, type of detecting device, and background count--and those related to the coal being tested--rank, particle size, and ash content. A survey was initiated to eliminate the relatively insignificant factors and to ascertain the magnitude of the major effects. Such information was necessary to fully evaluate the technique and to establish design criteria.

A simple meter for measuring the moisture content in coal utilizes the phase shift in a guided microwave train caused by the presence of moisture. The phase shift is compensated by moving the position of a slide screw tuner by a micrometer movement. The moisture content can be read off a large rotary scale attached to the micrometer or displayed through a mechanically coupled sliding potentiometer. The zero setting and calibration were found to be independent of the coal matrix composition. Sensitivity and correlation with standard moisture values are good for 0–4% moisture content. A regular calibration curve from 0–18% moisture has been established, and direct rapid measurement of moisture in coal is thus possible [14]. The effective diffusion coefficients of moisture increased with both increasing particle size and microwave power, whereas the activation energy of the diffusion process decreased with increasing particle size. Measurements were carried out on different types of coal with moisture contents ranging between 5 and 15 wt.% and sizes from fine powder to 12 mm particles. The measurements are obtained almost immediately with r.m.s. errors ≤ 0.5 wt%. [15].

Thermogravimetric Analysis (TGA) which measures weight changes in a material with temperature provides a convenient method for performing a proximate analysis usually in less than 20 minutes. Although the thermal analysis technique also involves heating the sample to constant weight under specified conditions, it does, because of smaller sample sizes and rapid temperature and atmosphere control, substantially reduce the analysis time as well as the equipment necessary for proximate analysis. To obtain a complete proximate analysis in a single TGA

experiment, the system is programmed to hold initially at $107 \pm 3^\circ\text{C}$, $200 \pm 5^\circ\text{C}$ in nitrogen, then to jump to 900°C and hold for a specified period of time in nitrogen before switching to oxygen. But this can be done with laboratory sample which is crushed and screened. Therefore, the moisture information we get from TGA would be residual moisture or moisture in analyzed sample, not total moisture. For moisture determinations, heat the weighed test

specimens in crucibles without the covers at $107 \pm 3^\circ\text{C}$. Use a drying gas flow rate of 0.4 to 1.4 furnace volume changes per minute. [16]. Program the instrument to terminate the test when the test specimens and crucibles have reached a constant mass. Alternatively, program the instrument to allow for moisture determination by heating the test specimens for 1h [17]. The value below which the absolute difference between two test results of separate and consecutive test determinations, carried out on the same sample in the same laboratory by the same operator using the same apparatus on samples taken at random from a single quantity of homogeneous 250 μm is 0.21-0.29% for TM and reproducibility between two labs is 0.69-0.79% with the coal having moisture in the range of 1.3-21.6%. However, it is not clear for steam coals with $>22\%$ TM what would be repeatability and reproducibility values.

A standard test method is a method for a physical test, chemical test, or statistical test; with definitive procedure that produces a reliable and unbiased accurate test result. Using a standard test method, perhaps published by a respected standards organization, is a good place to start. A well-written test method is important. However, even more important is choosing a method of measuring the correct property or characteristic. Not all tests and measurements are equally useful: usually a test result is used to predict or imply suitability for a certain purpose. Many such published standard test methods available for measurement of moisture in coal. In this paper we will be discussing on the practical applicability and inherent limitations and omissions of IS, ISO and ASTM test methods for moisture estimation in steam coal.

IS: 1350 (Part 1) – 1984 [4]

A. Indirect method: A known mass of the material is dried and the loss of mass calculated as moisture. The following are the indirect methods:

- I. Drying in air (one stage): two methods
 - a. Method I (at 200 °C): This method is not applicable to coal
 - b. Method II: This method applies to laboratory samples of coal crushed to pass 212-micron IS sieve. The method is also used in determining the moisture for coal equilibrated at 60 percent relative humidity
 - Requires Ventilated drying oven: Airflow rate is not defined. Excess airflow likely to oxidize the coal.
 - The temperature inside the oven is to be measured at what point is also critical in an electric oven. The oven specification is also not clear in the standard, whether hot air, nitrogen atmosphere, volume, temperature sensitivity and control etc.
 - The range of weighing balance is also not specified. The test will be conducted for 1.5 gm of sample, so need to specify the balance accuracy
 - II. Drying in air (two stage) -air-drying followed by oven-drying –
 - a. Stage I: special moisture sample of 1 kg of coal crushed (12.5 mm). weigh the sample and the container to the nearest 0.5 g. Allow the material in the tray to air-dry at atmospheric temperature in a well ventilated place free from dust. Take the drying to be complete when the change in mass during an hour is less than 0.1 percent of the sample. The standard is not specific on -
 - Temperature and humidity of sample preparation room, which has significance on test results ignored in standard
 - In 1 kg coal in the weighing balance of 0.5 gm of least count means 5% error is acceptable in the standard.
 - Prolonged atmospheric exposure of lignite coal may generate erroneous result
 - b. Stage II: Crush the air-dried material to pass 2.90 mm IS sieve. Spread uniformly in the weighing vessel about 10 g of the crushed material and weigh. Heat the uncovered vessel in the drying oven at a temperature of $108 \pm 2^{\circ}\text{C}$ until there is no further loss in mass. The standard is not specific on -
 - Gain or loss of moisture during sample preparation
 - Weighing balance least count
 - Oven atmosphere control
 - Presence of excess air in oven
 - III. Drying in a minimum-free-space oven (for coke): not specified for coal
 - What is the reason this method cannot be used for coal?
 - The volume of excess space is limited and atmosphere is nitrogen, would be more suitable for steam coal testing
 - Temperature can be reduced to $108 \pm 2^{\circ}\text{C}$
 - Need to specify the balance sensitivity, should be $\pm 0.01\%$ or ± 0.001 gm least count
- B. Direct method: This method applies to the analysis sample of 500 g of coal, crushed to pass 212-micron IS sieve, delivered in an air-tight container. This method is recommended where the material is particularly liable to oxidation. A known mass of the material is heated in a glass tube in a current of dry nitrogen. The gas is passed through weighed moisture-absorption tubes. This method is applicable when free water is absent in coal. The sample is air dried crushed sample, not as received sample. So this moisture is not total moisture but residual moisture after air dried loss is done.
- C. Moisture at 60 Percent Relative Humidity and 40 °C: Two methods
- I. Reference Method: Test done with laboratory sample of coal, ground to pass 212-micron IS sieve. Testing requires more than 48 hrs. time. Impact of humidity of atmosphere have significant impact. Measurement of 60% humidity in test zone is difficult. Again the test results are referring to the residual moisture as the sample is air dried.
 - II. Rapid Method: Test done with laboratory sample of coal, ground to pass 212-micron IS sieve. Testing requires less time. Test result denotes to the residual moisture not total moisture of coal.
- D. Moisture at 96 percent Relative Humidity at 40°C: Here also laboratory coal sample is used. The sample is equilibrated for >48 hours. And testing was done. This measures equilibrated moisture.
- E. Free Water or Visible Water: is calculated with a formula by using the TM measured by two stage method and equilibrated moisture.

From all the test methods described by IS 1350 (part1), certain limitations in terms of accuracy and precision levels are observed, which have been discussed under each method. The IS method is not accounting the errors due to measurement uncertainty. The repeatability and reproducibility of the test for steam coal with higher moisture level is also not initiated, taking various laboratories into the task, which would have been very valuable.

ISO STANDARD

ISO 589:2008 - Hard coal -- Determination of total moisture [18]: This International Standard describes two methods for determination of the total moisture content of hard coals, a two-stage method and a single-stage method. For either method there is a choice between drying in air and drying in a nitrogen atmosphere. Depending on the coal rank, there may be systematic differences between the results obtained by drying in the different atmospheres on subsamples of the same sample. Drying in a nitrogen atmosphere is suitable for all hard coals, while drying in air is only suitable for hard coals not susceptible to oxidation. The term "not susceptible to oxidation" cannot be defined easily. Usually, high-rank coals such as anthracites are not oxidized under the conditions described in this International Standard. For all other types of coal, this has to be verified by experiments. It is very clear that steam coal which is low rank lignite coals or a blend are not appropriate for testing in air drying due to their susceptibility to oxidation. ISO 589: ensures samples are dried between 105 °C – 110 °C in a drying oven featuring nitrogen blow possibility with flow equal to about 15x capacity of the oven per hour. ISO 11722 [19] Solid mineral fuels – Hard coal – This standard covers determination of moisture in the general analysis test sample by drying in nitrogen.

ISO 5068-1&2:2007: Brown coals and lignites -- Determination of moisture content -- Part 1 & 2: Indirect gravimetric method for total moisture [20], specifies two basic procedures:

- A. A single-stage method which can be used for determination of the total moisture content of the special moisture sample or of a moisture sample taken from a common sample (in accordance with ISO 5069-2 to pass a 3.15 mm sieve and divide to 500 g). A sample, prepared using a closed mill, is dried to constant mass at a temperature of 105 °C to 110 °C in an atmosphere of nitrogen, and the total moisture content is calculated from the loss in mass of the sample.
- B. A two-stage method which can be used for determination of the residual moisture content and, if required, also the total moisture content of a special moisture sample or of a moisture sample taken from a common sample. The single-stage method consists in drying the sample to constant mass at a temperature of 105 to 110 °C under specified conditions. The two stage method contains additionally drying at temperatures not exceeding 40 °C.
 - I. ADL or Air Dried Loss: A sample is coarsely ground (ISO 5069-2 [21] to pass a 20 mm sieve and divide to 2 kg) and is then allowed to dry, either at ambient temperature or at a higher temperature not exceeding 40 °C, to reach equilibrium with the atmosphere.
 - II. Residual moisture or RM: The sample is further crushed (grain sizes between 3 mm and 20 mm and, in any case, not less than 500 g) and then dried to a constant mass at a temperature of 105 °C to 110 °C in an atmosphere of nitrogen.
 - III. The total moisture content is calculated from the losses in mass during the two drying stages ADL and RM.

ISO Standard clearly specifies Oven requirement such as: nitrogen-flushed, capable of being controlled by a temperature of 105 °C to 110 °C and with the additional provision for passing a current of dry nitrogen through it at a flow rate about 15 times the oven volume per hour. Insert samples only where the temperature is known to be 105 °C to 110 °C. Balance is sensitive to 0.01 g and to 0.001 g. The results of duplicate determinations, carried out in the same laboratory by the same operator using the same apparatus on the sample within short intervals of time i.e. repeatability shall not differ by more than $\pm 0.5\%$ for TM and $\pm 0.4\%$ for RM. The mean of the results of duplicate determinations, carried out in each of two laboratories on representative test portions taken from the same sample, shall not differ by more than $\pm 1.2\%$ for TM and no specification for RM. ISO test method also specifies that the test report should refer identification of the product tested; reference of the method used; and result & the method of expression used.

ASTM Method

Some of the methods of coal analysis are empirical and require strict adherence to specified conditions, such as particle size, temperature, time and rate of heating, and so on. The establishment of uniform specifications that are recognized as standards and supported by authoritative organizations is essential. Committee D05 on Coal and

Coke of the American Society for Testing and Materials (now ASTM International), one of the largest standards development and delivery systems in the world, has the responsibility of developing standard procedures for

coal analysis. Most of the coal tests that ASTM has standardized were written around higher ranked bituminous and anthracite coals. The tight pore structures of these coals limited the amount of inherent moisture they could hold. Typically, these high ranked coals have EQ moistures of 1-10. The first step of determining coal quality in the lab is to air-dry the sample to near equilibrium with the laboratory humidity levels. This is done to minimize any impact on lab results of additional drying or absorption of water from the air. In high rank coals, the moisture lost in the air-drying step is near equivalent to the surface moisture. The residual moisture is that moisture that is still locked up in the coal after air-drying. The higher ranked coals that ASTM standards were based on possess this well-defined split between the air-dried or surface moisture and the residual or near EQ moisture. This is not the case for low rank or blended coals like steam coal. The sponge-like or wood like nature of steam coals make the split between surface moisture and inherent moisture a rather fuzzy line. In practice, the various forms of moisture in coal are described according to the manner in which they are measured by some prescribed standard method. As described in ASTM D 121 [22] the moisture forms routinely determined for coals are inherent moisture, free or surface moisture, total moisture, air-dry loss moisture, residual or air-dried moisture, and as-received moisture [23].

ASTM D 2961 [24] - Single-Stage Total Moisture Less than 15 % in Coal Reduced to 2.36 mm (No. 8 Sieve) Topsize. Moisture in the 2.36 mm top-size sample is determined by heating the test portion (minimum of 125 g) evenly dispersed (1 in. maximum depth) in a shallow pan at 104–110 °C for 1½ hours. After weighing, the sample is reheated and reweighed at ½-h intervals until the weight loss is ± 0.05 %. Materials subjected to this test shall not be used in the determination of other test parameters. It is recognized that the conditions of the test can increase the potential for significant oxidation effects on some coals. If the oxidation potential is of concern, the use of this single-stage method shall involve prior agreement between the parties involved. This method was formally referred to as a “limited purpose” method or an “industrial” method. This test method is not to be construed as a substitute for the referee standard test method for total moisture, which is ASTM D 3302 [25].

ASTM D 3173 [26] - Moisture in the Analysis Sample of Coal and Coke, laboratory sample must be reduced to Number 60 (250- μ m) size and divided, and an analysis by heating for 1 h in dry air atmosphere. It is used for calculating other analytical results to a dry basis. When used in conjunction with the air drying loss as determined in accordance with Method D2013 [27] or Practice D346 [28], each analytical result can be calculated to an as-received basis

ASTM D 3302 [25] - Total Moisture in Coal from air-dried No 8 top size coal, residual moisture is determined by heating at 104–110 °C for 1½ h.

Usually the first moisture value to be obtained on a coal sample is the air-dry loss moisture. This moisture loss occurs during an attempt to bring the coal sample into equilibrium with the atmosphere in the sample preparation room. Temperatures used for air drying vary over a wide range. The ASTM specifications call for air drying on a drying floor at room temperature or in a drying oven at temperatures 10–15 °C above room temperature, with a maximum of 40 °C. The air-dry loss (ADL) moisture as a percentage of the total moisture in coal is variable. It may vary from 25–90 % of the total moisture for different samples and may vary widely for coals of the same rank. It has been used incorrectly in some instances as a measure of the surface or free moisture. The use of the air-dry loss moisture value by itself has no real significance in the characterization of coals.

Residual moisture, or as-determined moisture, is used to calculate other measured analytical values to the dry basis. Residual moisture alone has no significance in the characterization of coals.

The sum of residual moisture and air-dry loss moisture is equal to the total moisture, is that which exists at the site, at the time, and under the conditions it is sampled. It applies to coals as mined, processed, shipped, or utilized in normal commercial operations. Coal-water slurries, sludges, or pulverized products under 0.5-mm diameter sieve size are exceptions. Total moisture applies to coals of all ranks. Total moisture is used for calculating other measured quantities to the as received basis (ARB) and as dry basis (ADB). Total moisture is important in assessing and controlling the commercial processing of coals.

ASTM D 5142 [29]- Proximate Analysis of the Analysis Sample of Coal and Coke by Instrumental Procedures, laboratory sample must be reduced to Number 60 (250- μ m) size and divided, and an analysis to a constant weight at 104–110 °C in nitrogen atmosphere which reduces the chance for oxidation of the sample. This standard is being withdrawn to be replaced by ASTM D 7582 [30].

ASTM D 1412 [31] - In this method, a sample is brought into equilibrium in a partially evacuated desiccator with an atmosphere of 96–97 % relative humidity at 30 °C. The amount of moisture in the coal under these conditions is determined by weight loss upon heating. While equilibrium moisture provides a fairly accurate estimate of the inherent moisture in high rank coals, the same is not true for low-rank coals where the equilibrium moisture is usually less than the inherent moisture. The chemical and physical nature of the low-rank coals, as compared to higher-rank coals, and differences in pore size distribution and resulting capillary action are just some of the factors affecting the measured equilibrium moisture in low-rank coals. Even though longer equilibration times are used for low-rank coals, equilibrium moisture values are still often less than the inherent moisture. The banded constituents—vitrain, clarain, durain, and fusain—that occur in coal vary considerably in the amount of moisture they hold at various relative humidities. One constituent, fusain, holds relatively little moisture below 90 % relative humidity. This is another reason for using a high relative humidity in the determination of the equilibrium moisture of coal. Inherent or equilibrium moisture is used for calculating moist mineral-matter-free calorific values for the rank classification of high-volatile bituminous coals. It is also used for estimating free or surface moisture, since total moisture is equal to the sum of the inherent moisture and the free moisture. The inherent moisture value is also referred to as bed moisture, since it is considered to be the moisture of the coal as it occurs in the unexposed seam, where the relative humidity is 100 %.

III. Precautions:

Coal being a very heterogeneous mass by nature, is most difficult material to be sampled because of varying composition from combustible to non-combustible in a single seam. This change occurs with depth, length and breadth of the seam in the same mine. Quality control thus becomes a stupendous task for coal preparation personnel. Whilst the total moisture and residual moisture of a given coal are related to coal rank there are many factors which determine the actual values including local mining conditions, the presence of extraneous mineral matter such as clays, and the treatment of the coal post mining. Therefore, the moisture characteristics of a given coal (total, residual and free moisture) are semi-empirical parameters and the values for a given coal may vary according to the test method used for its determination.

The sensitivity of the balance (scale) that is being used to weigh the samples must be to within a minimum of 0.1% of the weight of the sample being tested (for example, if samples being tested are expected to be about 100 grams in weight when dry, the scale should be able to read to at least 0.1 gram (or a tenth of a gram) – this would be the minimum sensitivity allowable, but a somewhat greater sensitivity, such as a sensitivity of 0.01 gram (or a hundredth of a gram) would be preferred in this situation.

Following are the list of precautions to be taken by the lab chemist -

- Particle size: the release of moisture is related to the particle size (surface area).
- Uniformity of coal collection and airtightness of sample container
- Size and shape of sample container (how much free space and exposure)
- Effect of different materials for the containers
- Moisture may evaporate from the coal in the sample container and condense on the walls of the container.
- Measure the change in temperature of crushing and grinding unit before and after operation
- Sample room temperature: Care should be taken to maintain the temperature consistent throughout. Any fluctuations in sampling room temperature the impact must be assessed and included the uncertainty of measurement
- Density of the coal layer: This is the ratio of weight of sample and surface area of sample spread. Keep this near to standard specification
- The ASTM specifications call for air drying on a drying floor at room temperature or in a drying oven at temperatures 10–15 °C above room temperature, with a maximum of 40 °C.
- Temperature of heating in Oven: find out the consistency in maintaining temperature and fluctuations at a fixed setup with time. Also calculate the impact of ± 1 °C temperature on the moisture. Use this as uncertainty expression component in the result calculation
- Duration of heating: Calculate the impact of ± 1 minute of time exposure on the TM results
- Rate of heating: Impact of rate of heating is significant on the weight loss.
- Oven atmosphere - Effect of nitrogen or air
- Rate of change of atmosphere inside oven
- How to maintain flow of dry air inside oven?
- Repeatability type (A) uncertainty covering Human factors and environment (housing and environmental conditions).
- Resolution at the testing range
- Non-linearity/interpolation error is taken into account if calibration curve or function is reported (usually taken from the manufacturer catalogue).

- Long-term stability is usually added by the user of the equipment after a period of using time
- It should be noted that moisture-holding capacity is not equivalent to total moisture, and that residual moisture is not equivalent to moisture in the analysis sample
- Some coals are so gassy that error due to loss of gas during drying is appreciable
- Composite sampling is not very reliable if too long a time elapses between the taking of the component increments
- The oven being used must be capable of maintaining temperatures of $103\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$ (or holding between $101\text{ }^{\circ}\text{C}$ to $105\text{ }^{\circ}\text{C}$) near the drying endpoint (this is the same as holding between about $214\text{ }^{\circ}\text{F}$ and $221\text{ }^{\circ}\text{F}$).
- Determining the moisture in the analysis by weight loss at $104\text{--}110\text{ }^{\circ}\text{C}$ presents several problems, especially if the current ASTM method D3173 [26] is used. experience, the ASTM method is unsuitable for low-rank coals because it does not use an inert gas as the purge gas and recommends too short a drying period. Oxidation -- gain in weight as the sample reacts with oxygen in the air -- can take place, and not all of the $104\text{--}110\text{ }^{\circ}\text{C}$ moisture is removed during the recommended one-hour drying time. In addition, with certain low-rank coals, decarboxylation during the drying period can result in weight loss. [32]
- The uncertainty analysis for a measurement - sometimes called the uncertainty budget of the measurement - should include a list of all sources of uncertainty together with the associated standard uncertainties of measurement and the methods of evaluating them [33]. For repeated measurements the number n of observations also has to be stated. For the sake of clarity, it is recommended to present the data relevant to this analysis in the form of a table. In this table all quantities should be referenced by a physical symbol or a short identifier. For each of them at least the estimate the associated standard uncertainty of measurement, the sensitivity coefficient and the different uncertainty contributions should be specified. The dimension of each of the quantities should also be stated with the numerical values.
- By comparison the experimentally obtained results with the exact values given in the reference material certificate, the method accuracy can be determined. Accuracy refers to a particular analytical result and hence represents a combination of systematic and random errors. Systematic error (bias) is the difference between the average value of a large number of measurement results and the exact value of CRM. Systematic errors can be eliminated by calibration.

IV. Conclusion

Standard Method preparing organization's role is to provide the coal industry unbiased methods periodically reviewed and updated, that are globally accepted for the use of commercial coal deals. The purpose is to determine accurate, precise, verifiable, measurements are part of the standards. Following the globally recognized standards by Indian Laboratories is mainly due to the international trade contractual agreement and statutory compliance from NABL (www.nabl.nic.in) that enforces the Indian industry to comply with BIS standards (by dual numbering of ISO standards) in lieu of which other standards like ASTM can be adopted. In addition, NIST Handbook 44 [34] regulates how weighing devices are selected, calibrated, tested and operated. Testing and operation of weighing devices are normally monitored and/or certified by State Agencies if large scale but for testing laboratory it's the lab to monitor and get certification by Calibration labs and use. The ISO standard deals with special precautions for moisture test samples. To be fair, the ASTM standard contains strong warnings about avoiding a test environment that could induce bias by reason of moisture losses. The ISO standard also says that "It is recommended that the complete bias test be carried out on a coal from a single source" for bias study. It is difficult to make sure in case of imported coals on the source and blending. The language of the ASTM standard lacks clarity and mandatory language thereby implying that uniformity is the governing criterion for selection of the test coal, while at the same time suggesting that selection of coals from different sources is good practice. The standard should contain mandatory language that the test coal shall challenge the capacity of the system to function without bias under whatever unfavorable operating conditions it is expected to encounter.

V. References

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