

## METHODS OF ANALYSIS OF COAL IN INDIA.

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There is probably no subject so pregnant with possibilities of dissension and acrimonious dispute than this hardy annual of methods of analysis of coal. The reason is obvious. We still, after several decades of intensive research on coal, do not know much about it. And when trying to analyse a substance whose chemical composition is still a mystery it is obvious that we cannot yet with confidence say that such and such a chemical procedure will give precisely such and such information about the substance. We have, therefore, to fall back upon agreed-upon methods of investigation and, further, we have to impose upon ourselves this and that restriction as to conditions of each experimental test, otherwise discrepant results obtained by different workers would merely be perpetual sources of disagreement and misunderstanding.

The rest of the technical and scientific world has long since agreed upon the necessity for adopting this attitude. India alone, it would appear, has not yet fallen into line and at this important stage in the development of industry in India, and especially at this stage in the history of mining and utilisation of coal in India, it is particularly unfortunate that it should still be necessary to reiterate the warning given by the author and others during the past ten years. Scientific research is progressing by leaps and bounds in this country and Fuel and Safety in Mines Research is at last coming into its own. The tragedy of the position is that for want of agreement upon this most vital and fundamental matter, the adoption of and adherence to standard methods of analysis of coal, probably more than 90 per cent of the work done on coal in this country during the past decade is of little scientific value. Years of otherwise valuable research work have been spent unprofitably and most of the investigational work that has been done will have to be repeated.

This is a subject that many have written on in recent years. Probably the most important authentic recent reference in the literature in India is the paper by Brodie in the *Records of the Geological Survey of India (Rec. LXIII, 1930, Part 2, 189)* with the accompanying note by Fermor. Its importance lies not in the value of the methods outlined but in the fact that the methods described are not those approved by the scientific and technical world. The laboratory in which the methods referred to by Brodie is that in which are analysed the coals of our two most important coalfields and it is on the results of these analyses that coals are graded in India. Further, it is largely on the grading thus achieved that the prosperity of the coal industry depends. As no authoritative statement appears to have been issued to the contrary it is assumed that these methods are still in use and the consequences appear to

the author sufficiently important, both from the scientific standpoint and from the point of view of the industry, to justify examination.

It is probably hardly necessary to state that analyses of coal are of two kinds, ultimate analyses furnishing information regarding the percentages of elements such as carbon, hydrogen, oxygen, sulphur, etc., in any coal and so-called 'proximate' analyses, indicating the proportions of what, for want of a better concise term, we may perhaps call the different components of a coal, the moisture, carbonaceous volatile matter, 'fixed carbon' and ash. In this *Symposium* it is assumed that all taking part are quite familiar with the meanings and full significance of those terms. We here are concerned with aspects of the matter that particularly affect us in India, with such questions as 'What differences are there between the methods used by various investigators?' and 'What justification, if any, is there for departing from what is generally adopted elsewhere throughout the world?'

It is sufficient if we examine the methods at present accepted as standard and certain known examples of departures from those methods.

Fortunately the scope of our enquiry can be limited. So little work has so far been done on ultimate analysis of coal that even if it had not been done according to accepted methods little harm would have resulted. But the methods that are suited for carrying out ultimate analyses are in any case standard, or more or less standard, methods of chemical analysis throughout the world. There is little risk, therefore, of the results of such ultimate analysis as have been done being inaccurate on account of the methods employed.

It is when we come to proximate analysis that we encounter a different state of affairs. And it is one the importance of which it is extremely difficult to impress upon anyone who has not himself performed such operations as are involved, still less anyone who is not himself a fuel technologist or even a scientifically trained person. The various 'components' are dealt with below, after reference to Preparation of Sample.

#### PREPARATION OF SAMPLE.

Perhaps a preliminary reference to Collection of Sample should be incorporated here. It is remarkable how this fundamental preliminary is improperly attended to and what extraordinarily stupid mistakes can be made when submitting samples for analysis. It is still no unusual experience for the author to be presented in his laboratory with a single large lump of coal with the request to have it analysed for the purpose of judging the quality of a seam of coal. It is needless to point out that in such a case it is almost unquestionably a waste of time even to carry out the analysis. The mining public and buyers and sellers of coal still require a certain degree of education in this matter. It must be admitted, of course, that extremely stringent conditions regarding the taking of a perfectly representative sample need not always be observed. There is such a thing as exercising discretion.

An important preliminary to preparation for the actual analysis is the removal of surplus moisture. The Fuel Research Board and most other official authorities specify that the sample weighed out for analysis shall be air-dried. Now air-drying is admittedly an uncertain procedure, especially in a tropical climate, and yet it is conceivable that we can agree upon a method to suit India. Drying inside a laboratory for 24 hours by laying the large sample out in a layer on a bench would appear generally to be suitable, except during the wettest period of the monsoon. During the cold weather and the hot season the author finds it sufficient to lay the large sample out on a verandah (on a sheet of clean paper), gently stirring it about from time to time especially if it is visibly wet. Obviously the amount of moisture still adhering will depend to a certain extent upon the degree of humidity and the atmospheric temperature on the day on which the sample is dried. This would appear to be a useful line of research for a beginner in this work.

Apart from selecting a method for preliminary drying it is important also to note that the amount of loosely-held moisture thus lost may have to be determined. Agreement between the coal supplier and the analyst is essential in this matter.

The method adopted by the United States Steel Corporation for air-drying of visibly wet samples might well be adopted for Indian conditions. It consists of passing over the sample a continuous stream of air, at a temperature of 10° to 15° Centigrade above room temperature, until the sample is of approximately constant weight. But it also applies a maximum temperature of 35°C. (95°F.) which would not always suit Indian conditions. Perhaps it may prove necessary to discontinue attempting in India to do accurate scientific fuel analysis in certain seasons.

Apart from the matter of air-drying we come to powdering the gross sample. The Fuel Research Board specifies 60-mesh powdered coal. This is practically universally prescribed and should be adopted in India. The crushing and sifting, of course, should be done as expeditiously as possible to avoid continual loss of moisture in the process.

#### MOISTURE.

Here again we come to a determination on which it is absolutely essential that we agree. The standard method prescribed consists in heating one gramme of the 60-mesh air-dried coal for one hour in an air oven at a temperature of between 105°C. and 110°C. Now mere heating in an air oven at the prescribed temperature is not sufficient. The air in the air oven must be changed several times during the heating. In well-equipped laboratories specially designed air ovens are available for this purpose. Where such luxuries are not available the author suggests gently opening the door of the air oven till it is as wide open as possible and then closing it again, choosing a time when the temperature has risen to 110° or nearly so. Experience shows that consistent results can thus be attained without any further elaboration of

apparatus. When the author is carrying out a large series of analyses at a time he relies upon the simple expedient of opening the oven door at approximately 10-minute intervals, using a laboratory alarm clock when available. When the laboratory alarm is in use for other purposes he times the opening of the oven door by the time taken for determining volatile matter in other samples. By this means, maintaining a temperature of about 110°C. in the oven, the moisture-laden air at that temperature is periodically swept out and replaced by fresh, comparatively dry air which soon attains the temperature of about 105°.

In determining moisture it must be remembered that the layer of coal should be evenly spread over the dish. The Fuel Research Board specifies a layer not more than 0.3 gm. per square centimetre of exposed surface. The Government Test House method described in the paper referred to above allows for taking 'about 2 gm. in a weighing bottle'. Presumably a fairly thick layer of coal results and it would appear as if the moisture-laden layer of air immediately above the heated coal does not have a reasonable opportunity of escaping, and the remaining moisture in the coal underneath is not likely to be fully driven off.

This is a matter of great importance in India as the grading of coals depends to a large extent upon the amount of moisture thus determined. In this respect the Government Test House (if it still adheres to the methods described by Brodie in the paper referred to) differs from practically all other experimenters and in consequence the grading of coals in India by the Indian Coal Grading Board cannot, in the case of high-moisture coals, be that calculated by any experimenter using standard methods.

This is a matter on which agreement should be aimed at.

#### VOLATILE MATTER.

In determining the amount of volatile matter other than moisture, the standard method prescribed a platinum crucible with tight-fitting lid, the crucible of particular dimensions, the using of exactly one gramme of 60-mesh air-dried coal and heating for 7 minutes to 925° ± 25°C. Platinum crucibles are expensive and many analysts may have to be content with porcelain crucibles and Bunsen burners. It is true that results of value to the industry for most practical purposes may be obtained by the cruder method. The standard method does not specify the use of an electric furnace, but points out that where an electric furnace is available porcelain or silica crucibles may be used. For accurate work, for grading and for all research work the platinum crucible must be used if no electric furnace is available, and there must be some accurate method of ascertaining that the correct temperature range has been attained.

The author uses at present an electric muffle furnace always maintained at about 950°C. The frequent opening of the door causes the temperature to fall and in practice the range 925° ± 25°C. is attained with little difficulty. Ash determinations are not carried out in this furnace. Where an electric furnace

is not available some sort of shield is necessary to ensure that the sides and top of the crucible are more or less uniformly heated. If one of the approved Davies type cannot be made up, a useful expedient is to surround the crucible, when it is on the pipeclay triangle, with teased-out stringy asbestos. This acts as an excellent radiator of the heat of the burner flame and a light layer of the same material on the lid completes the furnace effect.

Some experiments prescribe removing the lid of the crucible and burning off the 'coke' layer formed thereon. That should not be done. There are other precautions to be observed, but it will probably be agreed that they are not materially affected more by Indian conditions than by those in other countries and, accordingly, they are not discussed here.

#### FIXED CARBON.

As this 'component' is determined arithmetically it will not be discussed here.

#### ASH.

Owing to the important chemical changes that may take place in the mineral matter present in a coal in the course of burning it, the temperature to which it is heated may greatly affect the weight of residue obtained. In order to minimise discrepancies due to this cause the standard method prescribes a temperature of from 750° to 800°C. The sample is heated for one hour (or till constant in weight) in an oxidising atmosphere. The author uses a muffle furnace kept at a temperature of about 780°C. The Government Test House method described in the paper referred to above prescribes a temperature of 1000°C. The Test House method referred to also uses, for this determination, the coke obtained from the volatile matter determination. Although coke is usually rather more difficult to burn to ash than coal the use of the coke obviates the difficulty that arises in the case of coals liable to mechanical loss through the coal splitting up on initial heating. If the extra time required for using the coke can be spared, therefore, the procedure is a useful one. The temperature prescribed, however, is much too high and is liable to give inaccurate results in those instances where kaolin, gypsum, some carbonates, common salt, etc. (liable to decomposition or volatilisation at the higher temperature) are present. Whether there is much risk of error from this cause in Indian coals is not known at present. This would be an interesting and important line of research.

#### DETERMINATION OF CALORIFIC VALUE.

Almost the only important statement to make in this connection is that no calorific value that has been determined otherwise than in a first-class bomb calorimeter should be given serious attention. Other methods of ascertaining approximate calorific values have their uses. The author himself has devised a formula for calculating calorific value from the data of the

proximate analysis and it has proved extremely useful as a check. But such calculation methods should not take the place of the use of the bomb, and other cruder apparatus such as the Lewis-Thomson calorimeter are only of use for purposes of rough comparison.

#### METHOD OF REPORTING ANALYSES.

There are several ways in which the results of a Proximate Analysis can be recorded. For example the amount of loosely-held moisture lost during air-drying may have to be recorded and that should be kept in mind prior to preparing the sample for the laboratory. Then the amount of moisture found by drying the 60-mesh air-dried coal in the air oven at between 105°C. and 110°C. may be shown in the total analysis, or a separate analytical statement regarding the moisture-free coal may be given, but in the latter event there must be some manner of showing what amount of moisture was present. Normally it is better to give a double or triple statement showing the analysis of the coal: (a) as received, (b) as 'air-dried sample', and (c) moisture-free.

Then it is frequently desirable to indicate the composition of the 'pure coal', *i.e.* the combustible substance originally present in the coal. A statement of analysis on such a basis is described as being given 'on the dry, ash-free basis'. The above are the most usual types of statement used or asked for. We have in India yet another type of statement, the one used officially by the Indian Coal Grading Board. In effect it is to some extent a compromise between the standard 'air-dried' sample method of recording and an 'oven-dried' sample method. In the single statement recording the results of the analysis the amount of moisture is shown (as determined in the air oven) and then the remaining items of the orthodox proximate analysis are shown, but they (without the moisture at the head of the statement) total 100. Including the moisture shown in the analysis the total, of course, would come to more than 100.

The calorific value is, in the standard method, shown for the coal according to the method of recording the results of the proximate analysis. For example if the coal has been air-dried the calorific value of the air-dried coal is shown. If a separate statement of the proximate analysis of the moisture-free coal or the 'dry, ash-free' coal is given then the calorific value as actually determined is re-calculated on the same basis and shown in the adjusted statement.

The reason for referring in such detail to this matter is that on account of the lack of agreement in India as to the method to be adopted to show the results of proximate analyses there are serious discrepancies in interpreting them.

Fortunately the discrepancy is not a serious one in the cases of the majority of Jharia Barakar coals, whose moisture content is seldom more than one per cent (though the error referred to below exists even in those cases). But in the case of high volatile coals there cannot but be reasons for misconception when critically examining the statements of analysis recorded by the Coal

Grading Board method, and those of us who are concerned in the winning, preparation and utilisation of coal in India should be fully alive to the possibilities of error.

A high-volatile coal, for example, usually, in India, contains a high percentage of moisture that would be driven off in the air-oven. A high-moisture high volatile Raniganj coal, containing about 7% of moisture, may be taken for the purpose of illustration. The statement of analysis of such a coal according to the several possible methods is given below and will repay close examination.

There are two important effects that operate in opposite directions, when judging the value of a coal by examining the analysis as reported according to standard methods and according to our official practice in India. The first is that the Indian official practice shows a high-volatile coal as having a higher calorific value than it really has, in the example given the calorific value being recorded as 6,458 calories per gramme, whereas the calorific value of the coal 'as received' is only 5,841 calories and that of the air-dried coal only 6,000.

On the other hand, the same coal is shown by the official Indian method as having 14.60 per cent of ash whereas the coal as received has only 13.21 per cent of ash and the air-dried coal only 13.52.

In other words the official Indian method of reporting coal analyses accords to high-volatile moist coals a relatively better calorific value and a relatively higher ash content than it does to low-volatile coals. If the one effect cancelled or 'neutralised' the other no great harm would result, but that does not happen.

Apart from the unrealised possibility of these two effects tending to neutralise each other it is more important to note that the official Indian Statement or Report of Analysis does not readily indicate to the purchaser what he is buying.

STATEMENT OF ANALYSIS OF A HIGH-VOLATILE INDIAN COAL ACCORDING TO DIFFERENT METHODS OF REPORTING.

*Statements according to internationally-accepted standard methods compared with C.G.B. method.*

	Coal as received.	Air-dried coal.	Calculated to dry-ash-free basis.	Coal Grading Board method.
Moisture .. ..	9.55	7.40	....	7.40
Volatile matter less moisture .. ..	28.49	29.19	36.92	31.50
'Fixed carbon' .. ..	48.75	49.89	63.18	53.90
Ash .. ..	13.21	13.52	....	14.60
Colour of ash .. ..	Light reddish brown.			
Character of coke .. ..	Moderately coherent.			
Calorific value. (Calories over gramme) .. ..	5,841	6,000	7,060	6,458

It will probably be agreed by all fuel technologists, and it ought to be agreed by all sellers and purchasers of coal in India, that this anomalous position should be rectified.

Unfortunately, closely bound up with this matter of adopting standard methods of analysis and of reporting the results is the whole commercial system on which has been built up in recent years the stability of the Indian coal trade. There is naturally a fear, among mining interests, that any 'tampering' with the present system of grading of coals would endanger the trade. The author does not agree. In order to rectify the matter it would not be necessary either to abandon the present system (in general) of grading coals nor would a coal at present in any particular grade be placed in another grade. All that would be necessary would be to adhere to the existing grading and, in re-wording the specification according to which the grading is at present carried out, the arithmetical changes necessary would be calculated and applied to grading of coals submitted for grading in the future. Except in so far as a very few particular coals might be found to be graded too low or too high according to the present method of reporting, no material change would take place. And purchasers would be better able to compare one coal with another, which they cannot satisfactorily do at present.

#### OTHER DETERMINATIONS.

It is not considered necessary to refer in detail to the other components, such as sulphur and phosphorus, as are sometimes determined, as no particular difficulty arises that is peculiar to India. But reference may with advantage be made to one test the importance of which is growing, namely, the determination of the fusibility of coal ash. Comparatively few coal ash fusibilities have been determined so far and fuel technology laboratories that are to be fitted up in future in this country should be capable of making this determination. The author has a small amount of information on this subject, mostly of a confidential nature, but hopes shortly to be able to publish an authoritative statement regarding a large series of Indian coals. Pending the liberation of that information it is sufficient to indicate some temperatures likely to be recorded.

A selection of Raniganj coal ashes was found to soften at from 1140°C. to 1290°C., their fusion points being shown to be from 1250°C. to 1420°C. The corresponding figures for a small selection of Jharia coal ashes were 1200°C. (for softening point) and 1230°C. to 1255°C. (for fusion point). A mixture of Jharia coal ashes gave a softening point of 1100°C. and a fusion point (complete fluidity) of 1250°C. These determinations were made in a normal oxidising atmosphere. [Note. Later information gives a much wider range for coal of both those fields.]

It will be noted that, as might be expected, the mixture of Jharia coal ashes began to soften well below the softening point of individual samples from the same field.



There is probably only one other point that calls for special comment and that is the determination of oxygen as well as of 'fixed carbon'. The amount of oxygen in coal is usually determined by calculation, after taking into account the distribution of the various other elements and radicles known to be present in the coal; and the amount of fixed carbon is determined arithmetically 'by difference'. But it is impossible to say in what form the incombustible mineral matter that gives rise to the ash exists in the coal and it is definitely known that the amount of the ash may be, and usually is, less than the amount of mineral matter that produced it. An average of seven-eighths is frequently assumed, but the matter is an extremely complicated one and much research work, especially by the well-known American worker Parr, has been devoted to this subject. The average figure referred to means that a coal shown to have 14% of ash may really contain 16% of useless mineral matter. In consequence the figure shown in the Report of Analysis for Fixed Carbon is too high and, in consequence, the classification of the coal by the usual methods is incorrect. For similar reasons the determination of the amount of oxygen in Indian coals cannot yet be accurately carried out. This is an important field for the research worker.