Some Remarks on the Coal Testing Laboratory as Related to the Modern Coal Industry

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Of the many interesting lines of specialized or applied analytical work, coal analysis is one which is becoming more and more important. This is evidenced by the fact that coal companies are now establishing laboratories for the analysis of their products. The increase in the number of such laboratories may be attributed to two major causes, the increase of coal preparation due to keen competition, which preparation requires control analyses, and to the growing tendency for the public, both industrial and domestic to buy fuel according to analytical specifications. Industry is coming to realize that errors in analytical results may be responsible for losses of thousands of dollars in the course of a year's time. In the case of one large company an error of approximately 1½ per cent in the calorific value of their coal resulted in penalties which amounted to about $18,000 in 8 months' time. Thus we see the importance of this line of applied analysis and the necessity of making such analyses as accurately as possible.

The sampling and analysis of coal is complicated by its nonhomogeneous character. Coal is made up of organic material which has undergone varying degrees of coalification. To this are added varying amounts of minerals such as calcite, pyrite, and clay minerals together with varying amounts of moisture. To satisfactorily sample and analyze such a heterogeneous material, therefore, offers its difficulties. Morrow and Proctor[1] have shown that individual lumps of coal ½—1 inch which were picked by hand from the same stream of coal varied in ash content from a minimum of about 1½ per cent to a maximum of about 66 per cent. This serves to emphasize the extreme danger in accepting an analysis of a single lump or a small quantity of coal as being representative of a larger lot of coal.

Sampling of coal must satisfy two requirements, the securing of a satisfactory gross sample and the preparation of this gross sample for analysis. In order that the gross sample shall be representative of the whole lot of the coal its size will vary with the nature of the product being sampled. For example, larger gross samples are necessary for large sized coals. Furthermore, different means of obtaining gross samples are necessary depending on whether face, car, or preparation

[1] "Variables in Coal Sampling" by J. B. Morrow and C. P. Proctor, A. I. M. E. Technical Publication No. 645, Table 3, page 11.

plant samples are to be taken. At present there are standard procedures outlined by the American Society for Testing Materials[2] for obtaining face and car samples, but as yet no standard methods for sampling prepared coals have been adopted. The increased amount of coal preparation has therefore created a great need today for standard methods of sampling these products.

Once a satisfactory gross sample is obtained the problem of crushing and quartering this down to a laboratory sample presents itself. This laboratory sample usually consists of about two pounds of ¼-inch coal which is further crushed and quartered in the laboratory to about 60 grams of -60 mesh coal for the actual analysis. Previous to this final grinding, however, the sample should be dried for some time, preferably overnight, in an oven through which a current of air is kept circulating at approximately 37° C. This drying brings about the loss of moisture which would readily be lost at room temperature and thus prevents troublesome loss of moisture in the later handling of the sample. Furthermore it provides a drier sample for the final grinding which sample can be readily ground without troublesome caking. This air dry loss is carefully recorded and is taken into account in the final results.

Coming now to the analysis proper we have a 60-gram sample of -60 mesh coal from which part of the moisture has been driven by air drying. The most common determinations made commercially are the proximate analysis including determinations for moisture, volatile matter, ash and fixed carbon by difference. To these may be added determinations for total sulfur, calorific value and ash softening temperature. For research studies and certain classification studies ultimate analyses may be made including determinations of carbon, hydrogen, nitrogen and oxygen. In some cases varieties of sulfur are determined including sulfate, pyritic and organic sulfur. Mineral carbon dioxide is sometimes determined and ash analyses are less frequently made. Certain physical tests are used in certain studies such as shatter tests, agglutinating tests, accelerated weathering tests, etc.

The majority of methods available for use in coal analyis are of the empirical type, that is, those in which all details such as time, temperature and kind and dimensions of apparatus are carefully specified. Since this is true it has been necessary to adopt standard procedures and to insist that all laboratories adhere closely to these standard methods in order that their results shall be comparable. The methods commonly in use are those adopted as standard by the American Society for Testing Materials[3]. In order to emphasize the necessity of close adherence to the conditions specified for these determinations let us consider briefly a few determinations. The determination of moisture in coal offers a good example of the absolute necessity of closely adhering to a standard procedure. It is possible to drive out different amounts

[2] Standard Method of Sampling Coal, A. S. T. M. Designation D 21-16.
[3] "Laboratory Sampling and Analysis of Coal and Coke." D 271-33 American Society for Testing Materials.

of moisture from coal by heating all the way from room temperature up to as high as 1000° C. It has therefore been necessary to set up an empirical procedure by which results for this determination may be duplicated. The moisture value is determined by heating a sample in a double walled oven whose temperature is regulated at 105° C by a boiling solution between the walls and through which oven air is passed at a rate to insure renewing the air two to four times per minute. Any deviation from these specifications will result in different values for moisture. In some cases ordinary drying ovens have been used for this determination. Such ovens do not fulfil the two most important specifications for such equipment, which are that the oven shall have a minimum of air space and shall be uniformly heated. An oven with square corners permits the formation of air pockets which are impossible to sweep properly, and heating units in the bottom of the oven do not effect uniform temperatures throughout.

The determination of volatile matter in coal is another very good example of the empirical nature of coal analysis. The amount of volatile matter lost upon heating a coal sample depends upon the temperature at which it is heated, the time for which it is heated, the type of equipment in which it is heated, etc. It is therefore necessary to very carefully adhere to the standard procedure which has been outlined for this determination.

Not only is it necessary to adhere carefully to standard procedures for the two determinations which have been cited above, but also it is true for other determinations in coal analysis. For instance, in the determination of ash softening temperature it is possible to vary the value obtained as much as 400°F. simply by varying the atmosphere in the furnace from reducing to oxidizing.

The requirements of control, marketing and classification analyses are somewhat different. Control analyses usually must be rapid, in many cases permitting sampling and analysis of many samples each day. Marketing analyses should be more accurate but not so complete as classification analyses where accuracy and completeness are essential. In all cases where possible standard equipment and procedures should be carefully adhered to. In cases where it is necessary to use rapid procedures the relation of results so obtained to results obtained by standard procedures should be determined.

We have shown briefly some of the difficulties which the coal analyst encounters. One must therefore realize the extreme importance of proper sampling, careful standardization, close adherence to standard procedures and acquisition of good technique in such a laboratory. And by no means has the last word been written in coal analysis. Contrary to the idea of some that there is no analytical research left to be done, there is a great need in coal analysis as well as in other specialized lines for new methods and for simplification of existing procedures to eliminate errors as much as possible.