By Gladys Berchtold

Change comes to coal laboratories

THE DECADE of the seventies sparked a huge upheaval in the coal industry. Public awareness of the danger of dependence on an imported energy supply, and the realization that a substantial shift to coal would be essential to economic growth made the once-shunned fuel a more attractive prospect to the investor. Hundreds of new mines were opened and the capacity of existing mines increased.

The coal laboratory, a tiny microcosm bobbing in the wake of the coal industry, has undergone vast changes in attempting to cope with the increased demands for quantity, speed, and diversity in coal testing. Not only is the laboratory faced with a quantitative increase, but today's consumer of coal laboratory services makes more sophisticated analytical requests, requires quicker turnaround, and requests assistance with a plethora of environmental protection problems.

In the history of earth's search for fuel, the coal laboratory—in fact, the whole coal industry—is of relatively recent origin. Although mining and the use of coal as a fuel began in the twelfth century in Great Britain, annual production did not reach seven million tons until 1750. By the turn of the twentieth century, coal was being produced on a world-wide scale and the coal testing laboratory was born. These laboratories were destined to grow and multiply for several reasons. There is an abundance of coal in the earth's crust and it has wide geographic dispersion. As the use of coal continued to increase, the fledgling laboratory was assured a continuing supply of samples.

Growth was constant, but in the early years it was very slow. Investigations were undertaken to understand the origin of coal, and various classification systems began to be developed. Agreed-upon ways of conducting tests had to be formulated by groups composed of buyer, seller, and other interested parties.

A body of knowledge slowly

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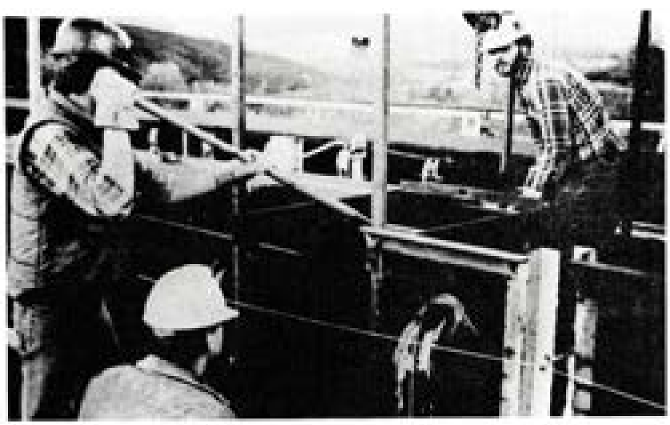
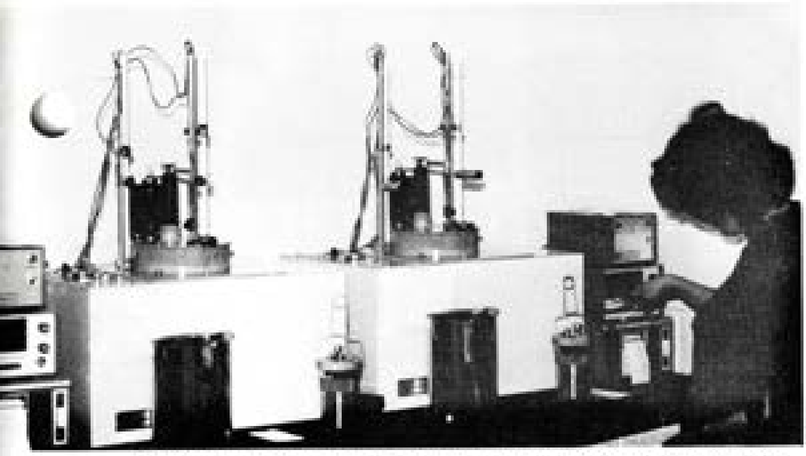
Above, crushing the gross sample and subsequent subdivision by riffling.



Left, taking a stopped-belt sampling. This type of sampling is accorded the highest reliability by ASTM D-2234.



Modern Parr calorimeters with digital readouts and printouts have greatly increased accuracy in calorific measurement.

Preparation of the oxygen bomb in BTU determination—a far cry from bombs of earlier days.

developed on the quality of various coal seams and the coal characteristics of individual mines. The work of coal chemists is a process of garnering information from chemical and physical tests performed in the laboratory and using this information to describe, classify, and predict the performance of coal under varying physical and chemical conditions.

In the early years of coal testing, most of the testing instruments used had been designed for other purposes and needed to be adapted for use in the coal laboratory. Occasionally a new instrument specifically designed for measurement of a particular coal characteristic would be introduced, but coal laboratories of the early 1900s were not characterized by great sophistication. Because of the heterogeneity of coal, tests were highly empirical. In fact, the determination of sulfur content was the only real wet chemical test run by most laboratories. Other common determinations, such as moisture, ash, and volatile matter, were made by physical testing methods.

Calorimetry was crude. Old timers in the business still tell horror stories about the four-foot spanner wrench needed to seal lead gaskets on early combustion bombs. Applying sufficient force to prevent oxygen leakage from the bomb required exertion of the full strength of a muscular man. Opening the bomb after combustion was even more difficult. Oxygen leakage was not the only problem in coal calorimetry. Various schemes were tested to isolate the heat rise attributable to coal combustion from the effects of the bomb's surroundings, and many years were spent before an adequate solution was found. Early calorimetry required the services of a very dedicated operator to accomplish reliable results.

The gas fusion furnaces were loud and smelly. The optical pyrometers used to read temperatures were bulky and unhandy. Reading the melting point of fusion cones was accomplished by visual means and sometimes resulted in a welder's flash burn to the operator's eye. Analytical balances were the swing-arm type, needing careful usage and constant maintenance.

Today, there are basically four types of coal laboratories. Producer laboratories are owned and operated for control purposes by the coal producer, just as consumer laboratories are by the coal consumer. Independent laboratories have no vested interest in the coal industry in any way other than the rendering of coal testing services. Research laboratories are devoted to research in the field of coal industry development and coal usage.

Many laboratories combine a control function with research capabilities. In these laboratories, many different approaches have been taken in the attempt to bring order to the complexity that is coal. The proximate analysis is the most useful approach for commercial purposes. Coal is generally bought and sold on the basis of a group of empirical tests called a proximate analysis. These tests include moisture, ash, volatile matter, and fixed carbon. The first three values are determined by laboratory tests, and fixed carbon is calculated by subtracting the sums of these values from 100%. Two or three other tests (sulfur content, calorific value, and fusion temperature of ash) are of vital importance in commerce also.

Another type of chemical test is the elementary or ultimate analysis. In this test the gaseous products of coal's complete combustion are analyzed to determine the amounts of hydrogen and carbon present. Sulfur, nitrogen, and ash are determined in the material as a whole, and a calculation is made for oxygen by subtraction from 100%.

A knowledge of elemental associations in coal can provide valuable information for coal benefication activities and is useful in assessing the extent of the harm to the environment of coal combustion. So, today, coal is analyzed for a number of major and minor or trace elements.

Another type of investigative approach is the study of the fundamental physical constituents of coal. The source of coal ash is the inorganic matter present in coal. This matter occurs predominately as discrete particles unevenly distributed in coal but, also, as chemical elements combined with organic matter and in dissolved material in the pore water.

Coal is studied by megascopic observation of coal beds in the field or samples thereof and as an aggregate of botanical entities through microscopic means using optical microscopy, scanning electron microscopy, and x-ray deflection. This approach is called coal microscopy—or more commonly coal petrography—and is useful in solving problems encountered in blending processes and also in utilizing various types of coals for coke production.

Many sophisticated studies involving modern instrumentation are being conducted on coal, and the catalog of testing services offered by coal laboratories grows daily. From the laboratory the coal producer can expect to receive daily information on coal quality before shipment, enabling one to make decisions about price and environmental impact.

General laboratory view showing two fusion furnaces and laboratory hood.

Various coals can be blended to build a more desirable or more valuable product. Decisions about future mining operations can be based on information gathered from the analysis of core drills and coal washing operations can be regulated based on coal washability data.

The consumer also gains valuable information from the laboratory involving protection from boiler fouling, and advance knowledge necessary for coke formation and steel quality decisions. Environmental regulations require that the impact of coal upon the ecosphere be known before the coal is burned. Many businesses are affected by coal and rely on the information emanating from the coal laboratory; immense sums of money are expended on coal laboratory data.

Big or small, complex or simple, captive or independent, all coal laboratories have common problems stemming from the fact that the raw material which they test is one of the most heterogeneous substances on earth. Coal is a black-brown combustible rock of organic origin. Just as every human is an individual unit, as no two trees are the same, or even as no two leaves on a tree are identical, coal exhibits the complexity expected from a consideration of its origin.

Coal is formed from plant remains, no two plants of which were identical. It was formed by accumulation methods that were perhaps similar in each time period and location but never identical. These accumulated plant remains were probably partly decomposed—again a process not characterized by uniformity—before they were subjected to various conditions of sedimentary burial. The final steps, those of increasing temperature and pressure, could not have been identical even in the same coal bed, much less in various locations in the world throughout geological history. It is little wonder then that coal exhibits a wide range of properties.

Coal's heterogeneous nature dictates that the early tests were preponderatingly empirical and based upon economic necessity. The same heterogeneity increases the problems associated with sample taking. The greater the degree of variability of a material, the more difficult it becomes to obtain a representative sample of the material.

The coal laboratory is faced with the task of sampling one of the most difficult of all natural materials to sample. The *ASTM Book of Standards*, Part 26, contains standard methods for collection of a gross sample of coal. Standard D2234 recognizes four conditions of increment collection, listed in the order of descending reliability: stopped belt cut, full stream cut, part stream cut, and stationary coal sampling.

Unfortunately, the least reliable condition, stationary sampling, is most often the only feasible way to collect a sample after coal has been loaded into cars and, then, only the surface coal is available to the sampler's increment collection device.

Great care must be taken in the choice of sampling equipment, careful attention must be given to increment size and placement as per Table II of Standard D2234, and knowledge of the physical character of the coal is needed. Persons entrusted with the task of coal sample collection must be well acquainted with the principles in D2234 and in D2013, Standard Method of Preparing Coal Samples for Analysis.

After the coal sample has been taken by or under the supervision of a person qualified by experience and training for the responsibility and who has understanding of the variable conditions under which coal may be sampled, specific information about the immediate conditions, and a knowledge of the nature of the particular coal being sampled, the coal is ready for reduction. The reduction is from a gross size (requirement for uncleaned coal—210 pounds of 2-in. × 0) to a laboratory sample of 50 to 100 g of -60 mesh coal. The independent, quality control laboratory is often required to perform this preparation process on hundreds of samples daily, while constantly protecting the integrity of each individual sample.

The purchaser of coal testing services needs to take several factors into consideration when rating a laboratory: quality of work, confidentiality of results, speed, and price—in that order. Too often the order is reversed. Speed of service and low price are entirely irrelevant if the quality of the analytical work is questionable or if the results of the tests are made available to other interests. The uninformed buyer of analytical services is prone to take quality for granted and then expect both fast service and low price. The prime consideration in choosing a laboratory must be a judgment regarding the ability of the laboratory to produce analytical data that are close to the true value of the product tested. True value, of course, cannot be determined until the total volume of coal is consumed, but the measure of a laboratory's quality is how close it can come to the true value by sampling and analyzing a representative portion of the gross lot.

The sophisticated consumer of laboratory services will require the laboratory to demonstrate its capability. To be competitive, the modern coal laboratory must have a good quality control system. It should be able to prove that it provides a uniform, quality service in testing, inspection, analysis and, if applicable, research. It should be able to meet minimum specifications in three areas: personnel, equipment (including calibration and standardization), and evaluation of results.

A coal laboratory should be under the technical supervision of a professional scientist. It should be staffed by personnel who are qualified by training and experience to conduct tests and inspections, to perform calibrations and standardizations, and to certify that materials and products tested comply with the requirements of applicable standards.

Coal laboratories should have all the necessary test facilities for the work which they warrant they are able to perform and should be capable of making and repeating measurements and analyses accurate within the limits specified in the appropriate standards. Their standard reference materials should have direct traceability to the National Bureau of Standards or other appropriate agencies. The laboratory must be able to prove that it can give accurate, reliable, and repeatable results. This is best demonstrated by a good reference sample program, sometimes called a round robin program. The laboratory should be able to demonstrate by its records that it is capable of repeatability and accuracy in laboratory test results.

Fortunately, it is much easier today to access a laboratory, perhaps because of an increased interest in laboratory accreditation which has caused the development of a great many guidelines for use in judging the relative merits of laboratories. One program is that of the American Association for Laboratory Accreditation (AALA), which has developed criteria for accrediting several types of laboratories on the basis of such parameters as personnel qualification and experience, condition of test equipment, recordkeeping and quality control systems, and other necessary laboratory controls.

The careful consumer of laboratory services would be well advised to secure information on specifications to be used in judging laboratories from the American Council of Independent Laboratories, Inc., 1725 K Street, NW, Washington, DC 20006. ASTM committee D05 on coal and coke is also currently preparing a standard which will be used for the evaluation of testing laboratories.

A word should be said about the role of the private, for-profit, independent coal laboratory in the United States economy. These independent laboratories provide a valuable service to the coal industry, since they render reports objectively and without bias. The cornerstone of the structure of the independent laboratory is its impartiality and objectivity. The independent laboratory must report facts to clients in an unbiased way, comparable to that of a professional in medicine and law. The laboratory does not long remain in operation unless it maintains absolute independence, impartiality, and confidentiality, whether the client be an agent of government, a coal producer, a seller, or a coal consumer, or any other link in the chain of commerce and industry. The independent laboratory is a cornerstone of our free enterprise society. These firms help: 1) industry to improve the quality and the performance of its product; 2) consumers to choose wisely among a range of products; and 3) government agencies to obtain accurate and objective engineering and scientific information supporting their legislative responsibilities. Thus, a proper environment for growth of independent laboratories is in the national interest.

The coal laboratory of today faces a bright future. Many changes have been made since the early 1900s. Rapid strides have been made in instrumentation. Modern calorimetry requires little operator input. Electric fusion furnaces with automated television readout systems make ash fusion tests simpler and more accurate. Analytical balances are electronically controlled and have digital readouts. Computers have been developed to perform many laboratory functions without human assistance. Quality control and quality assurance programs are the norm instead of the exception. New frontiers, such as testing for the synfuels industry, are being explored. Accreditation by a nationally recognized organization (AALA) is a reality instead of a dream. The role of the independent laboratory has been firmly established as a functioning intermediary between buyers and sellers of coal.

Other things firmly resist change: coal is still heterogeneous, it is still black. Cleanliness in the laboratory is still accomplished with great difficulty and requires eternal vigilance. The coal laboratory worker still must be intelligent, very strong, and must love soap and water. The customer still wants analytical results the day before yesterday and wants them with ASTM standard accuracy. With all its problems, coal testing has survived its first 100 years—the period of infancy. The child now walks and looks forward to the day in which it will run.