PRIMARY COAL - ANALYTICAL NEEDS

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<u>Abstract</u> - Coal differs from other minerals in that its properties vary throughout the coalification series lignite - bituminous coal anthracite. The properties of the various ranks of coals are important in deciding the application of the coals in industry and the analytical needs for primary coal are those determinations which enable the correct assessment of quality and consistency to be made and enhance the utilization in modern industry and environmental conservation. The analytical needs cover three sets of parameters associated with chemical quality, physical nature and condition, and rank fundamental properties.

INTRODUCTION

"Analytical Chemistry" as involved in the evaluation and utilization of coal has not, until recent years, been of a very high degree of sophistication. The relatively simple testing procedures were inherent in the very nature of the material and its uses. Coal was regarded as something to burn, or at best carbonize, and provided it could be used satisfactorily in these two technologies, little attention was paid to either the scientific studies of combustion or carbonization, or of the material being consumed. It was not realised, at least in the United Kingdom, that coal was a valuable wasting asset and conservation should have been practised. The decrease in easily worked deposits, together with the decline in availability of the better quality coals has resulted in a need for the development of blending techniques - blending practices need controls and this is best done on an analysis basis. Blending is now extensively practiced in the preparation of coals for carbonization and this can involve blending of several different classes and/or grades of coals to produce blends which, on carbonization will yield cokes comparable with those hitherto produced by the carbonization of prime coking coals originating from single sources. The same need for blending applies to coal for combustion processes, but here the blending is usually of different qualities of the same class of coal - the need for accurate control is not so great as in coking technology.

The environmental problems associated with the use of coal have only recently been highlighted - the use of coal was probably the main cause of atmospheric pollution, when such pollution was first recognized or at least acknowledged. Pollution resulting from the presence of sulphur in coal has been recognized for a number of years and orders to proscribe the use of coals containing excessive amounts of sulphur, especially for power generation have been made in various countries; the sulphur content being limited unless secondary steps are taken to cleanse the combustion gases from sulphur products. Pollution resulting from the presence of trace elements is becoming more and more important. Trace elements can have a considerable effect upon the environment when they are released into the atmosphere in the quantities possible when using very large tonnages of coal at one power station. This possible pollution is common to all mineral fuels - oil as well as coal.

The presence of trace elements in iron and steel processes based upon coal and coke is also being recognized as detrimental to both the environment and the products or operations such effects are being enhanced by virtue of "recycling" which is being more and more practised in the iron and steel industry.

Coal Material

In the geological sense coal is a rock and not a mineral. In practice coal is considered a mineral despite its non-compliance with the classical definition due to Dana (Ref.1) "A mineral is an inorganic homogeneous substance with a definite chemical composition". Coal is actually an organic sedimentary rock; the physical, chemical and technological properties of which depend upon its composition and degree of metamorphism. According to Fox (Ref.2) "Coal is a compact stratified mass of metamorphosed plants which have in part suffered arrested decay to varying degrees of completeness". Coal therefore differs from other minerals in that its composition and properties are controlled to some degree by the nature of the original plants but more so by the conditions that existed during and subsequent to their deposition. Coalification, the changes responsible for the continuum "Peat - lignite - bituminous coal - anthracite" results from the effects of strata pressure and moderately elevated temperatures acting over long periods of geological time.

The foregoing simplified description of the origin of coal will serve to explain the principal variations that occur in the vast reserves of coal in the world's known deposits. Such variations form the basis of numerous commercial and scientific coal classifications - the classification of any coal being dependent upon chemical and fundamental properties. The major variables may be summarized as follows :-

<u>Types or variety</u>. Differences are due primarily to differences in the original plant debris. These are not important in the context of "primary coal - its evaluation and uses".

Rank. Differences represent the degree of coalification.

Grade. Differences refer to the purity, primarily with respect to ash and sulphur contents.

To evaluate primary coal it is essential to consider both analytical and testing requirements and to understand correct sampling procedures. This latter need is of paramount importance for the correct use of coal in industry. As a mineral, coal is valuable because of its basic structure and composition. It is essentially carbonaceous with varying degrees of contamination by inorganic substances, the amounts of which play an important role in coal utilization. The degree of metamorphoses of the coal-material, i.e. the rank of the coal, considerably affects the fundamental properties associated with behaviour on heating. It is these latter fundamental properties that are important in deciding the technology adopted to put coal to its most valuable use in industry - always provided the chemical quality is acceptable. In the modern industrial world coal is used for combustion, carbonization and, of comparatively recent date, conversion. Recent, in that conversion is in effect being revived or revised, after having been tried and found wanting in the 1920's, the procedures and economics then available were unable to compete with oil. The ultimate use of coal in industry depends to a very large extent upon the rank of the coal. All coals can be burned or used in combustion technology, as they all contain carbon and hydrogen. They can all be used in conversion processes, some easier than others, but only certain ranks can be used to produce cokes by carbonization.

Evaluation of Coal

In the United Kingdom the basis of any evaluation and classification is the analytical data necessary to complete the information asked for in the pro-forma shown in Fig. 1 and Fig. 2. Coal quality as it affects original assessment and subsequent industrial utilization covers three different sets of parameters -

- a) Chemical quality ash, sulphur, moisture and volatile matter contents
- b) Physical nature and condition moisture, size, hardness and handleability
- c) Rank fundamental properties petrology, plasticity and fluidity properties on heating.

The analytical and testing techniques to assess coal quality are relatively simple and most countries have issued standard methods for such analyses.

<u>Chemical quality</u>. The efficient operation of large capacity coal-consuming plants requires frequent and rapid checking of coal qualities "as received". A detailed analysis of a coal is the key to a meaningful specification and it is important to ensure consistent quality for both commerical and technical reasons.

<u>Moisture</u>. The natural bed-moisture, or inherent moisture, of coal varies greatly with rank, the lower the rank the higher the moisture content. As low rank coals are used more in combustion technology than are the higher rank coals, the extent of the inherent moisture is important in power production. Most bituminous coals are mechanically cleaned before use, usually by a wet process, this wet-washing results in an appreciable free-moisture content. Surface moisture of coals can vary considerably and for various technical reasons this surface or free-moisture is important in the handling of coal. Free-moisture affects handleability, flow from bunkers and grinding and pulverization procedures where applicable. It can also interfere with the efficient operation of coke-oven batteries both by affecting the oven schedule and increasing gas consumption.

<u>Ash.</u> This is the incombustible mineral-matter left after a coal burns completely or which, in the coking process, is concentrated in the coke. This mineral matter which varies in amount, composition and fusibility occurs in three distinct forms. Inherent ash which is finely divided and is evenly distributed throughout the coal substance - it is actually part of the coal substance; shale bands and inorganic material, e.g., pyrites which are present as discrete inclusions in the coal seam; and adventitious dirt which contaminates the coal during mining operations. Coal preparation is done to remove the free and



SECTION/BORCHOLE

Area Scientific Department

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P = Persistent horizontal parting

APU 28 39

	VANE TEMPEST: G.60 FACE	
	I″-0: FI. I.60 22.10.75	
ANALYSIS	0.0	
Ash (db) %	9.8	
Volatile Matter (db) %	30.6	
	39.5	
Sulphur (db) %	2.22	
B.S. Swelling No.	(<i>1</i> 2	
Gray - King	6.7	
Code N.C.B. E.C.E	502	
Cod Type Analysis 0.6-0.7	4	
(Vol: per cent) 0.7-0.8	24	
0.8-0.9	57	
Max Ro 0.9-1.0	15	
(1.518 546 nm) 1.0-1.1		
1.1-1.2		
.2-1.3		
1.4-1.5		
1.5-1.6		
1.6~1.7		
1.7-1.8		
1.8-1.9	0.97	
MEAN RO MAX.		
Maceral Analysis (Vol.per cent)		
Vitrinite	71.5	
Exinite	7.8	
	16.9	
Pyrites	1.3	
MODIFIED KOPPERS OVEN TEST	60	60
	55	55
CUARCE		
CHARGE	50	50
	45	45
Weight 45g		
Nominal Depth Scin		40
CARBONISING CONDITIONS	35	<u>35</u>
Rate of Rise (300°c-600°c)3°c/mm		
Time at 600°c 100 min	قى	3 30
Head Load (-8ft) 500a/sa cm	25	ي 25
		÷ 20
		5
	ξ ιο	
	o f 1997	0
	300 375 450 525 600 20 40 60 80 10	-15-
	Base of Charge Time at 600°(Temp (°C) (min)	Case of Charge Time at 600°C Temp(°C) (min)
GIESELER PLASTOMETER TEST		
Softening Point °C		· · · · · · · · · · · · · · · · · · ·
Initial Fluid Point °C		
Max Fluid Point °C		
Final Fluid Point °C		
Solidification <u>°C</u>	N.D	
Plastic Range °C		
Fluid Range C		
	ет	
AUDIBERT-ARNO DILATOMETER TE	345	
T2 Inflation Tomperature °C	396	· · · · · · · · · · · · · · · · · · ·
T3 Tomp of max dilation °C	330	
	440	
IVIAX. CONTRACTION %	<u> </u>	
Max. dilatation %		· · · · · · · · · · · · · · · · · · ·
	1.035	
HARDGROVE INDEX	N.D.	

/LR:

adventitious dirt. In all cases ash is deleterious to efficiency. In power production it incurs expense to have it removed from the combustion chamber; it can also cause fuel-bed problems by clinkering or slagging. In the blast-furnace the coke ash affects furnace operations and efficiency; it requires "carbon" to melt and be removed as slag. Normal conventional methods of determining moisture and ash contents, requiring as they do sampling and sample preparation before laboratory analysis, are not suitable for continuous measurement and are hence not applicable to control systems. The analytical trend in these areas, moisture and ash determinations, is towards automated analysis. It is intended that the rapid methods should be capable of being used "on stream" and should therefore be suitable for control purposes and control systems where blending and mixing is being done. Such control systems will lead to consistency in quality for both combustion and carbonization coals.

The continuous determination of moisture is based upon indirect measurements related to the effect of moisture content on electrical properties such as electrical resistance, absorption of micro-waves and capacitance variation. Two of these methods, micro-wave absorption and change in capacitance, have been shown to be satisfactory for the continuous determination of moisture on coal preparation and coking plans in Durham Area, National Coal Board, United Kingdom (Refs. 3 and 4).

With both methods there is a rank effect due to the varying inherent moisture content, it is considered that the inherent moisture, being "absorbed" does not behave in exactly the same way as free or surface moisture. This rank effect necessitates calibration curves to be prepared for each application. Accuracies obtained in the laboratory for the United Kingdom range of coals are in Table 1.

<u>Coal Rank</u> U.K.	Classification E.C.E.	Moisture Range(%)		e Range(%)	<u>Standard Deviati</u> Micro-wave method	<u>on(% Moisture)</u> Capacitance method	
202	200/211	1.0	-	16.3	0.62	0.72	
301	435	0.4	-	12.2	0.48	0.46	
401	635	2.3	-	17.1	0.35	0.61	
501/502	634	4.2	-	17.6	0.59	0.78	
602	633	2.2	-	20.6	0.70	0.57	
702	622/732	5.4	-	18.4	0.71	0.75	
802	721/731	17.8	-	22.8	-	0.38	
902	711	14.6	-	29.4	-	0.68	

TABLE 1. Coal rank and moisture content for rapid methods of analysis

The rapid determination of ash content can be done by using methods based upon the absorption of quantum radiation. The relationship between ash content and absorption of such radiation stems from the fact that the absorption is proportional to the atomic numbers of the elements present. The elements of the inorganic constituents which go to make up the ash have higher atomic numbers than the lighter elements (carbon, oxygen and hydrogen) which make up the combustible substance; hence the greater the ash content the more the attenuation. A number of methods involving radiometric techniques have been proposed and practical experience of such methods has been successful in the United Kingdom (Refs. 5 and 6). The actual absorption is not easy to measure, necessitating constant density of the samples being irradiated so most of the methods measure the back-scatter which is proportional to the absorption. The sources of the radiation are usually radioactive isotopes although the Cendrex, the first successful ash monitor, used X-rays (Ref. 5).

Results for the rapid analysis of ash content obtained in the British investigations are in Table 2 (Refs. 7 and 8).

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Apparatus	Isotope	Particle size (mm)	Coal grade	Ash Range (%)	Standard deviation of Ash (%)
Laboratory prototype	109 _{Cd}	13-0	Blended smalls	11-26	0.8
Laboratory prototype	109 _{Cd}	13-0	Untreated smalls	8-37	1.3
Laboratory prototype	238 _{Pu}	5-0	Untreated smalls	18–28	0.7
Laboratory prototype	238 _{Pu}	5-0	Untreated smalls	9-13	0.6
Commercial type	238 Pu	5-0	Coking smalls	3.5-5.0	0.15
Commercial type	238 Pu	5-0	Coking smalls	3.8-6.3	0.4
Commercial type	238 _{Pu}	5-0	Part treated smalls	17-14	0.6
Commercial type	238 _{Pu}	5-0	Part treated smalls	19 - 35	1.0

TABLE 2. Ash content and standard deviations from rapid ash installations

Such rapid methods for determining analytical needs are very important for blending systems aimed at producing consistent qualities in coals for combustion and/or carbonization purposes. The application to boiler fuels in particular being very important because, quite often, acceptable coals for power production can only be prepared by blending untreated, and hence relatively high ash coals, with treated coals. Ash monitors are therefore essential for controlling the blend within reasonable limits, and their development and installation should be rapidly pursued.

<u>Sulphur</u>. This can be present in coal from traces to over 5%. Its presence is detrimental to both combustion and carbonization coals. On combustion it produces sulphur dioxide, an environmental hazard and actually many coals are excluded from use in power stations by virtue of their sulphur content unless the products of combustion are cleaned before being discharged to atmosphere. This limitation of sulphur content and the need for cleaning of stack gases are particularly stringent in the United States of America. Processes to remove the harmful gases are expensive in both capital cost and running costs.

In the carbonization process sulphur is undesirable because most of the coal sulphur is retained in the coke. This sulphur has to be removed from the iron and steel-making processes by either modifying the blast-furnace burden with a consequent reduction in iron output or by external desulphurizing techniques which are both money and time-consuming. To comply with clean-air regulations and to optimize blast-furnace operations it is necessary to have reliable data on the sulphur content of coal or indeed of any other fuel.

Sulphur occurs in coal in three forms, pyrite, organic and sulphate sulphur. Some sulphur may be removed during coal preparation if present as relatively coarse pyrite, but finely divided pyrite and organic sulphur cannot be removed by present conventional coal preparation methods. Sulphate sulphur is usually less than 0.1% as calcium sulphate and is not considered important either as a possible source of atmospheric pollution or as a draw-back to efficient blast-furnace operation. Methods of further reducing or removing both pyrite and organic sulphur not removed by normal washing techniques are being studied. These possible techniques must of necessity involve fine grinding of the coal, by which token they must be costly in coal recovery even if successful to some degree in reducing the sulphur content. It is considered that a better approach to acceptable sulphur content for power raising would be one of the conversion processes being investigated which involve pyrolysis and hydrogenation and produce a clean fuel, either gas or char.

A method for the rapid and automatic determination of total sulphur in coal would be of great value in controlling blending operations where the sulphur content vary in the several components of the blend. Research and investigation are at present conducted to develop a radiometric technique similar to that for ash determination (Ref. 9).

An acceptable level for sulphur in coking coal blends for furnace coke production is about 1.5 - 1.60%; for foundry coke the sulphur content of the coal should not exceed 1.05%.

<u>Physical nature of coal</u>. The hardness or strength of the coal material is important in technology where size reduction is needed before utilization, e.g., pulverized fuel firing and charge preparation for carbonization. Strength or hardness in this context relates to the evaluation of coal as measured by the ease of grinding - this is sometimes referred to as friability or grindability. The standard test for grindability is the Hardgrove Test (Ref.10) which is an approved test of the American Standard for Testing Materials (A.S.T.M) (Ref.11). There is a relationship between the grindability of coal and coal rank or classification. The easiest coals to grind are the United Kingdom classifications 200-300-400, i.e., the coking coals, the lower ranks 500-600-700 are much more difficult to grind. Where blending is being practised the differences in grindability could lead to operational difficulties and adverse results unless steps are taken to ensure the correct grinding of the hardest coal in the blend. These practical difficulties and effects upon carbonization coals to ovens - the differences in grindability resulting from rank and petrographic differences being the basis for the Burstlein process.

Another aspect of the physical nature of coals related to hardness but probably more so to the methods of mining, is the size consist of a product as prepared for use. The size consist affects the moisture content of washed coals and influences pulverization; it also affects handleability.

Fundamental rank properties

Rank refers to the position of the coal in the coalification series. The rank fundamental properties are more applicable to, and more important for, the evaluation of coking coals than other coals. The properties concerned are primarily associated with the changes which occur when the coal substance is heated. The fluid behaviour of the thermal decomposition products formed by some bituminous coals in the temperature range 300-600 °C is referred to as the plasticity of the coal. The measurable properties of the plastic systems of coals and coal blends are recognized as important basic knowledge for coal utilization. These plastic properties, fluidity and dilatation, are related to coal rank as defined by petrology. Coal petrography or petrology is the study of the organic materials which make up coal. The object of the study is to identify, in terms of reactive capabilities, each of the organic materials making up coal and to attempt to relate the reactive capabilities with the industrial application of the coal. Plasticity and petrology are closely connected, and evaluation of coal quality, as assessed by these properties, has developed along parallel lines.

Coal petrology as practised today consists of microscope studies of coal - it was originally conceived as a macro-study of coal. Over the past decade the microscope has been gainfully used to study the composition and structure of coal and to establish coal petrology as a scientific discipline in its own right. It has been developed to such an extent that claims have been made of its application to predict accurately the physical properties of cokes from the petrological analyses of the original coals (Ref. 12). Its application has been mostly confined to coals for carbonization and covers two main fields or properties of these coals - maceral analysis and vitrinite reflectance measurements.

The original petrological macro-studies of coal classified the coal entities as clarain. vitrain, fusain and durain - these were the Stopes nomenclature. Under the microscope the macerals or entities recognized are termed vitrinoids, exinoids and fusinoids or inertinites (V.E.I.). There are sub-groups within these major groups, but these further entities or microlithotypes are not of great value in coal technology - they are primarily of academic value. Generally the macerals (V.E.I.) can be divided into two classes according to their thermal behaviour - reactives which soften and re-solidify during heat treatment or carbonization and inerts which remain relatively unchanged. Vitrinoids are the principal constituents of British coals, as they are of most carboniferous coals. and reactive vitrinoids are the essential coke-forming materials in coking coals. The ratio of reactives to inerts in a coal is important in assessing coal quality, as this ratio affects the plastic properties - the ratio has been suggested as a method of predicting coke properties (Ref. 12). There is probably an optimum ratio of reactives to inerts for acceptable coke properties from some coals but it is now considered that this ratio or maceral composition is not adequate to assess quality in coking coals, and it does not apply to some coals, e.g., Australian coals, where the inertinite content can be 50%.

As petrographic and thermal behaviour studies developed and improved the knowledge of the nature of coal substances it became increasingly apparent that the definitions in the petrographic analysis of coal were not sufficiently discriminating. The maceral concept (V.E.I.) did not comprehend the degree of coalification. It has already been stated that rank or degree of coalification is important in determining fundamental properties, especially the plastic properties, and to a lesser degree the chemical composition - volatile matter - of the coal entities. It was deemed important to find a measurable physical property that could be used to define coal entities in terms of the stages of coalification or rank. Hoffman and Jenkner (Ref. 13) showed that vitrinite could be used as a key for determining rank; they developed methods based upon reflectance from polished

surfaces and also showed that the reflectance of vitrinite increased with rank or carbon content. Coal is anisotropic and the maximum reflectance of vitrinite is recognized as being the most accurate method for determining rank. When depicting or reporting petrological data for a coal it is usual to give the maceral count V.E.I. and to plot the maximum reflectance of the vitrinites as a histogram. There is at least one standard method of determining maceral composition and the maximum reflectance of vitrinite (Ref. 14) and the British Standards Institution is busy preparing a standard method.

Coal petrology is an excellent means of checking coal quality as deduced from the rank classification based upon volatile matter and Gray-King coke type. Changes in the shape and distribution of the reflectance histogram provide irrefutable evidence of changes in coal quality, either of coals as produced or of mixtures subsequently prepared. Conventional laboratory analysis cannot approach this "detective ability".

Petrological studies are therefore important in assessment of coal qualities and a method of speeding up the microscope examination would be most useful. The draw-back to automation of the studies would be that the measurement of reflectance would relate to global reflectance and not only the vitrinite reflectance - the latter reflectance is considered the true measure of rank.

Coal plasticity. The reactives in the vitrinoid classes are responsible for the fluid behaviour when the coal substance is heated. The degree or extent of plasticity developed is responsible for the coking properties or otherwise of the coal. There are three standard methods of measuring the plastic properties of coal

- a) Giesler plastometer, which is a standard A.S.T.M. test
- b) Audibert-Arnu dilatometer, used in the Economic Commission of Europe (E.C.E.) classification system and which has been recommended by the International Organization for Standardization as the approved test
- c) Hoffman or Ruhr dilatometer which differs very slightly from the Audibert-Arnu test.

The fluidity and dilatation properties as measured by the above tests are rank dependent and exhibit maximum values in the plasticity-volatile matter curves. It is interesting to note that the A.S.T.M. Giesler fluidity test has been adopted by the Canadian, Australian and Japanese coal producers and users, whereas the dilatation tests, Audibert-Arnu or Ruhr dilatometers are favoured by the European countries. There is a growing need for standardization of these tests especially the Giesler plasticity test where the use of different applied torques can result in vastly different fluidity values.

Inorganic constituents of coals Recent years have seen extensive developments in the detection and determination of inorganic elements in both coal and coal ash. Some, like sulphur and phosphorus, have always been recognized as important because of their effect upon coal utilization. Sulphur is polluting in coals for power generation and it has deleterious effects upon blast-furnace practice when present in coke. Phosphorus is also important in that its presence can lead to deposits in super-heaters and furnace tubes, while it is detrimental to steel quality and more so to castings when present in foundry coke. Some metallurgical coke producers fix maximum contents of both sulphur and phosphorus in coke as follows :-

Furnace	coke	maximum	sulphur	1.30%	e.g.,	coal	sulphur	about	1.60%
Foundry	coke	maximum	sulphur	0.90%	e.g.,	coal	sulphur	about	1.05%
Foundry	coke	maximum	phosphorus	0 -01 %	e.g.,	in co	Dal 0.008	3%	

The alkali metals have always been recognized as undesirable in coals especially when associated with chlorine. Alkali attack on refractories occurs below the temperature at which potassium and sodium compounds volatilize. These elements also act as fluxes and can cause trouble in super-heaters and furnace-tubes, and in the blast-furnace can lead to tuyère and refractory failures. The effect on coke activity due to the presence of alkali metals is being investigated as a possible reason for enhanced carbon solution in the blastfurnace due to the reaction of coke with carbon dioxide, such solution being considered as factor in the strength, or lack of strength, in cokes in the tuyere zone.

An element which is assuming importance is zinc. Zinc is concentrated in the blast-furnace because the top is normally too cold for it to escape as a gas and the bottom is too hot to remove it as a liquid. Zinc deposits in the upper zones of the blast-furnace can lead to corrosion of refractories and to "scaffolding" with subsequent faulty furnace operation. The zinc content of coal and coke whilst very small, aggravates the problem of zinc in the blast-furnace which is primarily due to recycling of feedstock to both furnace and steel plants - an appreciable amount of zinc being introduced as galvanized scrap.

Other inorganic elements are being recognized as pollutants and intensive surveys are being made using modern analytical techniques of mass spectrometry, atomic absorption and Z-ray fluorescence (Ref 15). The large variations in reported values for many of the elements show a need for standard reference materials for evaluating methods and quality control. The exact import of many of these trace elements has yet to be determined especially their environmental significance.

CONCLUSION

As already stated the analytical needs for primary coal evaluation and quality control in utilization are relatively simple, but the importance of coal in modern technology is so great it is essential that the analytical needs and the methods of determining them are fully documented and approved. The growing export and import of coals warrants the international standardization of test procedures.

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