# 8.14 Coal Analyzers


## Types:
Thermogravimetry (TG), oxygen combustion bomb, total sulfur analysis, x-ray fluorescence (XRF), atomic absorption spectrophotometry (AA), coal slurry analyzers, prompt gamma neutron activation analyzers (PGNAAs), and the pulsed neutron type

## Precision of Measurements:
Heating value, ±200 to 300 BTU/lb; ash content, ±0.05 wt%; and moisture content, ±0.05 wt% moisture

## Partial List of Suppliers:
- Fisher Scientific ([www.fisher.co.uk](http://www.fisher.co.uk))
- Gammametrics/Thermo Electron ([www.gammametrics.com](http://www.gammametrics.com))
- Kanawha Scales ([www.kanawahascales.com](http://www.kanawahascales.com))
- Leco Corp. ([www.leco.com](http://www.leco.com))
- Parr Instrument Co. ([www.parrinst.com](http://www.parrinst.com))
- Science Applications Inc. ([www.saic.com](http://www.saic.com))

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## INTRODUCTION

Coal is widely used as a source of power and heat by the chemical, paper, cement, and metal industries. In the U.S., there are some 900 coal preparation plants and coal-fired power plants, and some 100 coal analyzers have already been installed in them. One key consideration in operating coal-burning facilities is the control of SO₂ emissions to the atmosphere from coal-fired power plants. Because the most economical method of reducing the sulfur content of coal is through the blending of various coals, on-line coal analyzers are often needed. The characteristics of coal are monitored for environmental protection, quality assurance, and process control.

This section will describe the instrumental methods for:

1. Proximate analysis: Used to establish the rank of coals, to show the ratio of combustible to incombustible constituents, to provide the basis for buying and selling, to evaluate the benefits, or for other purposes
2. Gross calorific value: Provides the basis for buying and selling
3. Sulfur analysis: Used in coal preparation and in the determination of potential sulfur emissions from coal combustion or conversion processes, or in the determination of coal quality against contract specifications

The various methods for the on-line monitoring of coal streams are described in the paragraphs that follow.

## THERMOGRAVIMETRY

Due to the recent addition of microcomputer control and dedicated data reduction, the thermogravimetry (TG) technique has become popular for routine approximate analysis of coal and coal products. The TG unit performs a multistep analytical sequence automatically and unattended.

The sample is loaded into a two-arm furnace tube ([Figure 8.14a](#)) and is sequentially dried and burned; the residue is then weighed. The tube allows the active gas (air or oxygen) to enter near the top of the furnace rather than through the balance mechanism. The low-mass furnace provides both rapid heating rates and a short cooldown time. The microcomputer controller provides automatic switching between the purge gas and the active gas.

### Bituminous Coal Analysis

[Figure 8.14b](#) shows a typical proximate analysis of a bituminous coal using the automated TG system. The furnace is heated to 230°F (110°C) at a rate of 140°F/min (60°C/min) and is held isothermally for 5 min while water is vaporized off from the coal sample. The furnace is then heated at a rate...
of 176°F/min (80°C/min) to 1742°F (950°C) and held for 7 min while nitrogen is flowing through the TG, until all volatile matter is expelled from the sample. After the nitrogen purge, the purge gas is switched to either air or oxygen and the fixed carbon content of the char is oxidized, leaving the ash content as the residue.

The values from these determinations are read directly in weight percent from the chart recorder. The air or oxygen purge is switched back to nitrogen, and the system automatically cools back to load temperature. The elapsed time of the proximate analysis program and cooling of the tube back to load temperature totals 30 min.

**GROSS CALORIFIC VALUE**

Gross calorific value is determined by burning a weighed sample of coal. A calibrated isoperibol oxygen bomb calorimeter is used for that purpose under controlled conditions (Figure 8.14c). The bucket, which holds the oxygen bomb, provides good circulation and rapid thermal equilibrium for the bomb. Thermal jacketing is provided by a circulating water system, which maintains cooling water flow around the bucket. A microprocessor control system monitors and controls the jacket temperature, fires the bomb, and monitors the temperature in the bucket. The test continues until the controller determines that equilibrium has been reached.

A microcomputer uses the sample weight and temperature data—applying correction for acid, sulfur, fuse, and any added combustion aids—to calculate the gross calorific value.

**TOTAL SULFUR ANALYSIS**

An ASTM method for determining sulfur in coal uses the washings from the oxygen bomb calorimeter. Sulfur is precipitated as barium sulfate from the washings. The precipitate is filtered, ashed, and weighed. An automatic titrimetric system is also available for rapid sulfur determination using the oxygen bomb washings. The washings are titrated with a lead perchlorate solution to obtain a lead precipitate. The titration takes place in a nonaqueous medium to ensure complete precipitation and a sharp end point with a lead ion-selective electrode.
Other procedures use high-temperature tube furnace combustion methods for rapid determination of sulfur in coal and coke using automated equipment. The instrumental analysis provides a reliable and rapid method for determining sulfur contents of coal or coke.

Figure 8.14d illustrates the high-temperature combustion method of sulfur detection using infrared absorption detection procedures. The sample is burned in a tube furnace at a minimum operating temperature of 1350°C (2462°F) in a stream of oxygen to oxidize the sulfur.

Moisture and particulate matter are removed from the gas by traps filled with magnesium perchlorate. Sulfur dioxide is measured, using an infrared absorption detector. Sulfur dioxide absorbs infrared energy at a precisely known wavelength within the infrared spectrum. Total sulfur as sulfur dioxide is determined on a continuous basis.

**ASH ANALYSIS**

The major or minor elements in coal ash can be determined using x-ray fluorescence (XRF) techniques. The ash is fused with lithium tetraborate or other suitable flux and either ground or pressed into a glass disk. After that, the pellet or disk is irradiated, using an x-ray beam of short wavelength (high energy).

The characteristic x-rays of the atom that are emitted or fluoresced upon receiving the primary rays are dispersed, and their intensities are measured at selected wavelengths by sensitive detectors. Detector output is converted into concentration by computerized data-handling equipment. All elements are determined and are reported as oxides. They include Fe, Ca, K, Al, Si, P, Mg, Ti, and Na.

These major and minor elements can also be determined by atomic absorption spectrophotometry (AA). See Section 8.22 for a more detailed description of XRF and AA.

**ON-LINE MONITORS**

Figure 8.14e shows a schematic of a continuous monitor of moisture, ash, and BTU in coal. In this system, a microwave analyzer measures the moisture content of the coal, without requiring physical contact with the solids. (See Section 8.34 for an explanation of the principles of a microwave absorption hygrometer.)
The fingerprint of a given type of coal is its distinctive gamma spectrogram. This is produced by the detection and counting of photons released from atomic nuclei in the coal as it passes over a small source of neutron emissions. The precision of the measurements of heating value, ash, and moisture content varies with the type of coal, typically 200 to 300 BTU/lb, 0.05 wt% ash, and 0.05 wt% moisture.

Gamma-Based Analyzers

The Harwell spectrometer is used in the prompt gamma neutron activation analysis (PGNAA) in-line coal analyzer. This package also measures coal density and moisture content. This multiple detector package also includes a feed hopper to direct the coal into the analyzer and a discharge conveyor, which takes away the coal under variable speed control. The system also includes an RS422–RS232 interface and remote displays for reporting the readings made by the analyzer.

An analyzer is also available for determining the ash and solids contents of coal–water mixtures or coal slurries. This analysis is made by the use of three probes, which are immersed into the coal slurry stream. The ash probe uses a source of low-energy x-rays to measure not only the ash content, but also the concentration of iron. The density probe used gamma rays to detect the percent solids (slurry density) of the mixture. The low-intensity neutron source in the aeration probe detects the amount of air in the slurry, so that the density measurement can be corrected for aeration effects.

Recent Developments

Western Kentucky University is developing a prototype coal analyzer operated with microsecond-wide 14-MeV neutron pulses and containing several gamma ray detectors. This analyzer measures the density and sulfur content of coal along with its BTU, moisture, and volatile matter content. This pulsed fast/thermal neutron analyzer is self-calibrating and provides improved accuracy in the determination of elements such as carbon, oxygen, and sodium. If you want to follow this developmental effort, refer to Reference 3.

References

1. www.kanawhascales.com/CoalScan9500/9500.htm
2. www.gammametrics.com/gmm2/GMMcsa.html
3. A prototype elemental coal analyzer based on pulsed neutrons, www.wku.edu/Dept/Academic/Ogden/Phyast/API/research/coal.htm

Bibliography

ASTM standards on coal testing, sampling, and analysis, American Society for Testing and Materials, West Conshohocken, PA, www.astm.org/
“Reading the Composition of Coal,” EPRI Journal, July/August 1980.