TA Instruments

Thermal Analysis Application Brief

Proximate Analysis of Coal and Coke

Number TA-129

Summary

Proximate analysis is an important quality control parameter for coal and coke. Thermogravimetric Analysis (TGA) which measures weight changes in a material with temperature provides a convenient method for performing a proximate analysis usually in less than 20 minutes.

INTRODUCTION

Proximate analysis covers the determination of moisture, volatile matter, fixed carbon, and ash in coals and cokes, and is used to establish the rank of coals, to show the ratio of combustible to incombustible constituents, or to provide the basis for buying/selling, and otherwise evaluating coal for various purposes. Thus, it is important to both the suppliers and users of coal to have a rapid, accurate, and reliable procedure for obtaining the proximate analysis.

Traditionally, the various proximate analysis determinations involve heating the sample to constant weight under ASTM specified conditions. These determinations, however, are time consuming and require a significant amount of laboratory equipment. An alternative method for proximate analysis is thermogravimetric analysis (TGA). Although this thermal analysis technique also involves heating the sample to constant weight under specified conditions, it does, because of smaller sample sizes and rapid temperature and atmosphere control, substantially reduce the analysis time as well as the equipment necessary for proximate analysis.

Experimental

In TGA, the sample contained in the pan of a sensitive analytical balance is heated under a flowing atmosphere (inert or reactive) and the weight change is recorded. To obtain a complete proximate analysis in a single TGA experiment, the system is programmed to hold initially at 200°C in nitrogen, then to jump to 900°C and hold for a specified period of time in nitrogen before switching to oxygen. Recent advances in TGA instrumentation have made proximate analysis even easier to perform. These include:

- Automated gas switching - with a Gas Switching Accessory, the purge gas change at 900°C can be built into the multisegment experiment method so that the Thermal Analysis Controller automatically makes the switch.

- Hi-Res™ TGA and the "abort" segment - historically the time that the TGA remained at a specific set of conditions to ensure that the associated weight loss was complete was operator - selected based on experience. In fact, the time was usually set to allow a "safety" margin which ensured that the weight loss was complete. With the new TGA "abort" segment, the Thermal Analyst Controller can be programmed to automatically proceed to the next experimental step when a weight loss is complete.

In this study, five coal samples representing the major classes of coal were evaluated. 40-45 mg samples were used, and nitrogen and oxygen purge gases at 60cc/min were introduced at appropriate times in the experiment.

Results

Figure 1 illustrates the typical TGA profile obtained. The three weight loss steps correspond to moisture, volatiles and

fixed carbon respectively. The remaining weight at 900°C in oxygen is ash. A summary of the results obtained is shown in Tables 1 & 2. Also shown in the tables are the ASTM - determined values for comparison.

PROXIMATE ANALYSIS OF COAL BY TGA COMPARISON TO ASTM METHODS
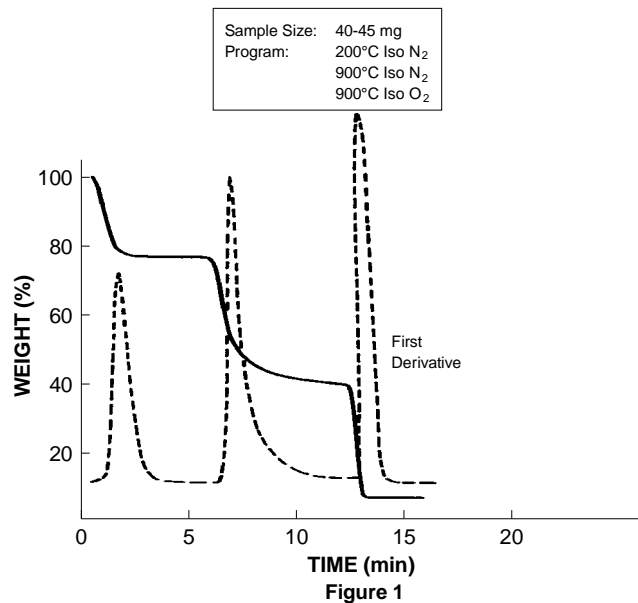
	Wt. %			
Coal Sample	Moisture (ASTM)	Moisture (TGA)	Ash Content (ASTM)	Ash Content (TGA)
Lignite	38.8	38.4	6.1	5.9
Sub-Bituminous	21.8	20.7	8.6	8.0
Bituminous	3.0	2.7	12.0	12.1
Bituminous	0.7	1.4	7.0	5.6
Anthracite	3.1	2.1	10.0	10.6

TABLE 1

PROXIMATE ANALYSIS OF COAL

Sample Size: 40-45 mg
Program: 200°C Iso N_2
900°C Iso N_2
900°C Iso O_2

First Derivative

TIME (min)
Figure 1

PROXIMATE ANALYSIS OF COAL BY TGA COMPARISON TO ASTM METHODS

	Wt. %			
Coal Sample	Volatiles (ASTM)	Volatiles (TGA)	Fixed Carbon (ASTM)	Fixed Carbon (TGA)
Linite	25.7	27.1	29.4	28.6
Sub-bituminous	32.3	32.6	37.3	38.7
Bituminous	34.1	32.0	50.8	53.2
Bituminous	41.7	41.3	50.6	51.6
Anthracite	5.1	5.7	81.8	81.6

TABLE 2

This general TGA procedure is also useful for the proximate analysis of cokes. Since the two quantities of primary interest to the users of coke are the fixed carbon and ash contents (the percent moisture and volatiles are usually low), the procedure used for coal is modified slightly to accommodate this fact. Using only the isothermal temperature of 900°C and varying the atmosphere from N_2 to O_2, a two step weight loss curve is obtained. The first weight loss corresponds to moisture and volatiles, the second to fixed carbon and the residue is ash. A complete proximate analysis for coke can be obtained in less than ten minutes.