

## FAST MEASUREMENT OF ASH CONTENT IN COAL WASHING PLANT

Ma Yonghe (马永和), Luo Xiangdong (罗向东) and Yang Dazhan (杨大战)

*(The Technical Physics Institute, Heilongjiang Academy of Science, Harbin 150010, China)*

(Received September 1992)

### ABSTRACT

The coal filter cake is a product of fine coal after floatation which has an ash content of 7—13 %, water content of  $30 \pm 2$  %, and a particle size of less than 1 mm. The ash content was measured by the intensity of the single backscattered gamma-ray, and its accuracy is mainly dependent on the energy of the gamma-ray. The  $^{238}\text{Pu}$  low energy photon source is selected in this work. The energy of its gamma-ray is 15 keV, which can result not only in the best sensitivity, but also in the lowest contribution to the environment radiation. The root mean square deviation of the ash measurement is  $\pm 0.33$  % ( $\pm 1\sigma$ ).

**Keywords:** Coal filter cake   Gamma ray backscattering   Ash content

### 1 INTRODUCTION

The measurement of the ash content in coal by nuclear techniques has a history of more than thirty years abroad. The methods of X-ray or low energy gamma ray backscattering were mainly used before the 1970's. The methods are suitable for the small particles and slow divergent stream system. Since the 1980's, the Australian researchers have developed the double gamma ray transmission method and high energy gamma ray annihilation method, being suitable for large particles and fast divergent stream system as well as direct on-line measurement. These are the two popular ash content measurement methods at present. Rather lately, in China, low energy backscattering method was first used in the divergent system. The method of double gamma ray transmission has also been being investigated.

Both instantaneous and average ash contents can be obtained by the on-line ash content measurement method, having a very fast response and can providing reliable information on the automatic control. The static ash content measurement method, compared with the on-line one, has some advantages: the instruments used feature in low cost and simple structure, being easy to use, and flexible in the installation spot, with no need to reform the field conditions and the processing technology.

The characteristics of the filter cake (coal powder) are small particle sizes, with

the upper limit of  $\sim 1$  mm, uniform particle distribution, and low ash content of about 7–13 %. The ash constituents are steady with little change, but the water content is high ( $30 \pm 2$  %). Based on these features, the low energy gamma ray backscattering method is used, and sensitive to low ash content. Its most sensitive region of the gamma radiation energy is 15 keV<sup>[1]</sup>.  $^{238}\text{Pu}$  is the ideal low-energy photon source. In order to suit the use to industrial environment, the Na(Tl) crystal scintillation detector is used to detect the backscattered gamma rays.

## 2 PRINCIPLE OF ASH MEASUREMENT

It is approved<sup>[2]</sup> that the ash content  $A$  has a linear relation of negative slope with the backscattered low energy gamma ray intensity  $I$ .

$$A = a + b \cdot I \quad (1)$$

where  $a$  and  $b$  are constants,  $A$  is presented in percent with two digits in the integral part and two digits in the decimal part,  $I$  is the accumulated counts of the detected backscattered gamma rays in the preset time  $t_0$ , with the order of four digits. In order to display the ash content with digitals directly, a certain measurement time interval  $t$  should be determined. The counts are accumulated within this time interval so as to let the value of the mathematical expression equal to the ash content value of the sample measured. Time has been considered in Eq. (1). If  $n$  is the count rate and the time used to accumulate counts  $I$  is  $t_0$ , then,

$$I = n \cdot t_0 \quad (2)$$

$$\text{let} \quad B \equiv a \quad (3)$$

$$t = b \cdot t_0 \quad (4)$$

Considering that the slope of the linear function is negative Eq. (1) can be rewritten as:

$$A = B - nt \quad (5)$$

This is a practical mathematical expression, in which  $t$  is the measurement time in seconds, its geometric meaning is the slope.  $B$  is the minuend or the preset counts, its geometric meaning is the offset. The count rate  $n$  is an independent variable which varies with the ash contents.  $B$  and  $t$  are calibration constants, which can be determined through experiments.

Eq. (5) is the fundamental basis for the design of the instrument, the basic function of which is the collection of the backscattered signals and the mathematical operations as in Eq. (5).

## 3 MEASUREMENT INSTRUMENT

The measurement instrument consists of the main unit and the detecting device.

The latter consists of measurement stand, detector, radiation source, sample container, sample holder, etc. The detector, radiation source, and the sample container are arranged in the centrally symmetrical way. The detector is a Model FJ-374A Na(Tl) scintillation detector, mounted in the measurement stand with its vertical position adjustable. The radiation source is a  $^{238}\text{Pu}$  low energy photon source, the intensity is  $8.92 \times 10^8$  Bq (25 mCi). The radiation source is mounted under the window of the detector, with its active surface downward, with the aid of a steel shielding ring, which acts both as a device to hold the source in position and as a protection shield. The coal sample is packed into the sample container. It is better to keep the bulk density of the sample constant and the surface smooth. The sample is then put on the sample holder beneath the radiation source.

The whole electronic circuit is put in a T8 standard module, with all the operation and display components on the front panel and all the cable connections to the detector on the rear panel. The high voltage supply and the amplifier are composed by discrete components, and the remaining digital circuits by CMOS integrated circuits.

## 4 GEOMETRIC CONDITIONS FOR MEASUREMENT

### 4.1 The thickness of the sample

The intensity of the backscattered gamma rays ( $I$ ) increases as the mass thickness of the coal sample ( $m$ ) increases, up to saturation. We assume that there is not any change happened to the photon energy during the backscattering process, i.e. the mass absorption coefficient ( $\mu$ ) is the same for both the incident and scattered gamma rays, then we have:  $I \sim [1 - e^{-\mu m}]$ , where  $m = \rho d$ ,  $\rho$  is the bulk density of the coal layer,  $d$  is the thickness of the coal layer. When the thickness of the coal layer increases to a certain point, the backscattered intensity is going to be saturated. This thickness is called the saturated backscattered thickness. In order to eliminate the effect on the intensity of the backscattered gamma rays by the change in the thickness of the coal sample, the thickness of the coal layer should not be less than the saturated thickness. In order to obtain the saturated thickness, we let  $\mu m = 3 \sim 5$ . In the extreme case of zero ash content and when  $^{238}\text{Pu}$  radiation source is used, it can be calculated that the mass absorption coefficient  $\mu$  of the fine coal with zero ash content and 30 % water content is  $1.0564^{[3]}$ . Therefore, the saturated mass thickness of the coal layer can be calculated as  $m = 4.73 \text{ g/cm}^2$ . In the experiments, based on the standard sample preparation method, the average bulk density of the filter cake of fine coal is  $\rho = 1.19 \text{ g/cm}^3$ . If the thickness of the coal layer is 4.5 cm, the mass thickness will be  $5.35 \text{ g/cm}^2$ , which is greater than the saturated.

### 4.2 The distance between the detector/radiation source and the surface of the sample

Experiment shows that the intensity of the backscattered gamma rays increases

with the increase of the distance  $D$  between the detector/radiation source and the surface of the sample, up to a maximum, then decreases slowly. The change of the bulk density of the coal layer has the same effect as this change. In practice, the distance is selected which results in the maximum intensity.

## 5 CALIBRATION AND MEASUREMENT

The nuclear measurement method is a relative one. The instrument can be used for measurement only after it is calibrated, i.e. the values of the constants in the calibration function is determined. The calibration of the ash content is based on the production practice. The counting measurements were done for the same time interval on some production samples, to obtain the backscattered intensities  $I_i$ , and on the other hand, these samples will be tested with the conventional methods to obtain the corresponding ash content  $A_i$ . Therefore, an array of  $(A_i, I_i)$ ,  $i=1,2,3,\dots,N$ , can be obtained, which shows the relations of the ash contents with the backscattered intensities, where  $N$  is the number of samples for calibration. The values of calibration constants  $B$  and  $t$  can be obtained by graphic method or analytical method based on Eq. (2-4). The preset count wheel and preset timing wheel on the front panel can be set according to the calibration constants  $B$  and  $t$  respectively, and the sample to be measured is put on the sample holder. Then, the measurement will begin when the stop, clear, preset, and start buttons are pushed in turn. The counter starts working to perform the subtracting operation, beginning from the minusend (preset counts)  $B$ , until the completion of measuring time interval  $t$ . The residual counts is displayed as the ash content.

The accuracy of the measurement is dependent on the accuracy of the calibration. For linear functions, in principle the calibration can be performed with two coal samples of different ash contents. However, taking into account the error in ash content test as well as the human factors, there exists the uncertainty in the result of the ash content tests. Besides, the uncertainty in the recorded backscattered intensity in equal interval also comes from the fluctuation of water content, bulk density of the sample and the statistical fluctuations. Based on the law of statistics and according to practice, the calibration should be done on enough number of samples. The constants so determined will be accurate and have good representation. At the same time, the coverage range of the ash content must be considered.

In the industrial in-situ experiment, the filter cakes from the vacuum filtering machine were measured against the laboratory tests. For a single sample, the difference of the ash contents between the measurements by instrument and the laboratory test is called the deviation of the measurement, and is represented as  $d_i$ . The distribution of the measurement deviations on 165 samples is shown in Tab.1.

In order to evaluate the measurement error specification, statistical processing must be done to a large number of comparative measurements. From the point of view of mathematical and physical statistics,

the standard error or root mean square error ( $\sigma$ ) is often used. From the point of view of production control, the mean error of the absolute value ( $d$ ) is often used. It can be calculated from the data by comparative measurement on a single sample that:

$$\sigma = \pm [\sum d_i^2 / n]^{1/2} = \pm [18.2019/165]^{1/2} = \pm 0.33 \%,$$

$$d = \sum |d_i| / n = 43.47/165 = 0.26 \%.$$

**Table 1**  
**The distribution of the measurement deviations**

| Deviation / %     | 0 — ±0.30 | ±0.31 — ±0.50 | > ±0.51 |
|-------------------|-----------|---------------|---------|
| Number of samples | 105       | 38            | 22      |
| Percentage        | 63.64     | 23.03         | 13.33   |

## 6 SOURCES OF ERRORS

The deviation of the ash content measurement by instrument is determined by comparison with the conventional analytical method that is regarded as a standard method. The errors of measurements come from the following aspects:

a. The error  $\sigma_1$  in the conventional laboratory test; This error is specified as  $\sigma_1 = \pm 0.30 \%$  according to the national standard. But due to the effect of human factors, the error can be sometimes greater than this.

b. The error  $\sigma_2$  caused by the change of water content; The ash content ( $A$ ) has a linear relation with the mass absorption coefficient  $\mu$ . The change of composition in the coal will cause change in the mass absorption coefficient. Therefore, the error in the ash measurement can be calculated. The nominal water content of the filter cake is 30 %. In case of the fine coal after floatation<sup>[3]</sup>, for the coal of 7 % nominal ash content, if the water content increases by 2 %, the measurement error caused will be +0.17 %; For the coal of 10 % nominal ash content, will be +0.08 %.

c. The operational error  $\sigma_3$  in sample preparation; A homogeneously mixed coal sample is divided into four aliquots, on which the error content measurement is taken respectively. The error between the measured value and the average is  $\sigma_3 = \pm 0.10 \%$ .

d. The error  $\sigma_4$  caused by the statistic fluctuation and the shift of the instrument itself; The error caused by the instrument after a 60 h long-term stability test is equivalent to a change in ash content,  $\sigma_4 = \pm 0.10 \%$ .

The overall instrument measurement error caused by the above-mentioned independent sources is :

$$\sigma = \pm (\sum \sigma_i^2)^{1/2} = \pm 0.36 \%$$

It can be seen that the above analysis is correct and basically agrees with the measured root mean square error  $\pm 0.33 \%$ .

It should be noted that the error in the ash content measurement by instrument

mainly comes from the error in the traditional laboratory test. Even if the instrument is ideally stable, the error in the ash measurement by instrument will not be by no means lower than the error in the traditional laboratory test.

## 7 CONCLUSION

The root mean square deviation between the ash content measured by instrument and that by laboratory test is  $\pm 0.33\%$ . Such an accuracy is enough for the fast direction on the production processes. The combustion method for the fast ash measurement can be replaced by the instrument. The problem of fast ash measurement on the fine coal powder after floatation has been solved. The tedious processes such as sampling, baking, grounding, laboratory test, etc. can be omitted. The whole process of the traditional analysis takes about one to two hours for the analysis on a single sample, while the measurement by instrument takes less than five minutes from sampling to measurement, and only half a minute for measurement. In case of the ash measurement by instrument, the information can be feed back in time. The method is useful for improving the quality of the product and the recovery rate, and it is of important in economics.

## REFERENCES

- 1 Boyce I S, Clayton C G, Page D. Nuclear techniques and mineral resources. Vienna: IAEA, 1977; 135—165
- 2 Ma Y H, Xiao D Y, Luo X D. Nuclear Electronics and Detection Techniques (in Chinese), 1990; 10(6):331
- 3 Ma Y H, Zhou W Z, Weng F. Nuclear Electronics and Detection Techniques (in Chinese), 1989; 9(5):285