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Research Paper

# Bias Testing of Mechanical Sampling System Based on Total Moisture and Ash Analysis

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***ABSTRACT :*** *As one of the energy sources utilized in various industries, coal quality is determined by its maceral composition, mineral matter, and coalification rank. Generally, coal quality can be assessed through proximate and ultimate analyses conducted in laboratories. Coal samples can be collected from various locations, such as moving streams or stationary setups, using either manual or mechanical systems. In this study, a bias test was conducted on a mechanical sampling system. Bias is detected as the difference between test results and accepted reference values. The acceptability of reference values is established through simultaneous coal sampling using both the reference method and the system under testing. Bias testing is required for new installations, system relocations, system upgrades or modifications, and system maintenance. The procedure involves several stages, including pre-bias inspection, bias inspection, sample analysis, statistical analysis, and data interpretation. A total of 30 paired samples were analyzed, with the parameters measured being total moisture and ash content. These parameters were mathematically processed to calculate the Hotelling's* $T^2$ *value, which was then compared to the* $T0^2$ *value to determine the presence or absence of bias. The results of the study indicated that the* $T^2$ *value was 4.425, while the* $T0^2$ *value for* $n = 30$ *and* $p = 2$ *was 6.885. This demonstrates that no bias was detected in the mechanical sampling system, as* $T^2 < T_0^2$*.*

***Keywords:*** *Ash; Bias; Mechanical sampling system; Reference value; Total moisture.*

## 1. INTRODUCTION

Coal is one of the most critical energy resources globally. The processing of coal is essential to produce high-quality coal that meets the demands of modern energy and industrial applications [1]. Coal is a combustible black or brownish-black sedimentary rock with a high amount of carbon and hydrocarbons. Coal is classified as a nonrenewable energy source because it takes millions of years to form. Coal contains the energy stored by plants that lived hundreds of millions of years ago in swampy forests [2]. The transformation of precursor materials into coal involves several critical stages, including peatification and geochemical coalification, which integrate biological, physical, and chemical processes. These stages produce coal with distinct levels of maturity and carbonization [3].

As a key energy source for industrial applications, Coal is considered one of the most complex geological materials. In addition to organic matter, which is valued for its energy potential, all coals contain varying amounts of water, minerals, and other inorganic constituents [4]. The quality of coal is primarily determined by its maceral, mineral matter content, and coalification rank [5]. The utilization of coal remains dominant as a primary energy source, particularly for power generation needs, with significant emphasis on domestic applications such as power plants (*PLTU*) and industrial sectors [6]. Typically, coal quality is evaluated through proximate and ultimate analyses performed in laboratory settings. Statement of [7] stated that hat each coal rank classification [8] possesses distinct quality characteristics, which are critical in determining its suitability for various chemical process applications.

Coal sampling is crucial for obtaining test samples that are subsequently analyzed to generate results for the coal sample [9]. Coal samples can be collected from various locations, including moving streams [10] or stationary sources, using either manual or mechanical methods.

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In this study, a bias test was performed on a mechanical sampling system, whether the equipment is suitable for operation or not. Bias is identified as the discrepancy between the test results and the accepted reference values. The acceptability of these reference values is determined by simultaneously sampling coal using both the reference method and the system under test (in this case, the mechanical sampling system). That essential aspect of the bias test is the cross belt (primary sampler), which tends to leave residual material on the belt conveyor. This indicates that the sample is intended to be collected cleanly [11]. Apart from identifying differences between test results and reference values, bias testing is also necessary in specific cases such as the installation of a new sampling system, relocation of the sampling system, or following any upgrades or modifications. The reference method used was the stopped belt, while the system under evaluation was the mechanical sampling system [9]. Moreover, according to [12], coal exhibits a high degree of heterogeneity, necessitating that sampling must ensure equal probability [13]. This approach eliminates the possibility of sample selection bias in coal and guarantees the representativeness of the entire population [14]. This approach is based on the ISO 13909-8:2016 standard, "Hard Coal and Coke – Mechanical Sampling – Part 8: Methods of Testing for Bias" [15]. The samples collected were subsequently analyzed in the laboratory for moisture and ash content. Total moisture represents the overall water content in coal, measured gravimetrically by determining the mass difference before and after the heating process [16][17]. Ash content refers to the residual inorganic substances present in coal, originating from inherent impurities formed during coalification or introduced during mining processes. Coal itself does not contain ash but comprises organic compounds that produce ash as a result of combustion [17]. All testing procedures followed ISO (International Organization for Standardization) methods.

## 2. RESEARCH METHODOLOGY

### 2.1. Pre-Bias Test

This stage involved a general condition check of the mechanical sampling system, referring to the ISO 13909-8:2016 standard, "Hard Coal and Coke – Mechanical Sampling – Part 8: Methods of Testing for Bias" [15]. The checks included several components, such as the primary sampler, belt feeder, secondary sampler, crusher, loading hopper, and collection bucket. Additionally, the weight of the coal sample discharged from the secondary sampler was also measured.

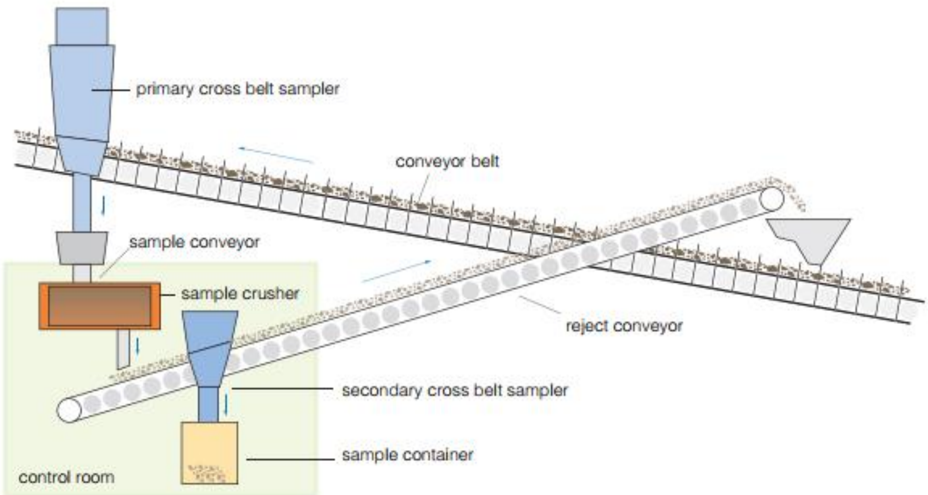


**Figure 1.** Mechanical Sampling System

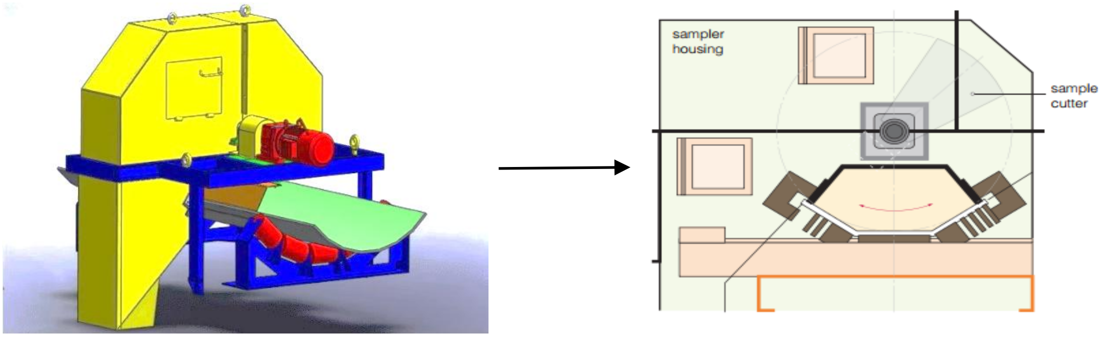


**Figure 2.** Primary Sampler

### 2.2. Bias Test

Samples taken during this bias inspection consist of the reference sample and the test sample. The standard reference used for this stage is the same as the one applied during the pre-bias test phase. The reference sampling method used is stopped-belt sampling. This method involves taking sample increments from the cross-section of the conveyor belt by halting the conveyor at specific intervals. When coal sampling is performed correctly and accurately, the increments in this method can be considered bias-free. The stopped-belt increments must be taken using a sampling frame that precisely matches the width of the conveyor belt and is at least three times the size of the top-size coal through the conveyor. Test samples refer to samples obtained from the mechanical sampling system being tested. The method used for sampling is the moving stream sampling technique. The test samples are collected using a moving mechanical sampler, which then cuts across the cross-section of the flow on the conveyor belt. In this stage, the total number of coal samples taken is 30 pairs, consisting of 30 reference samples and 30 test samples

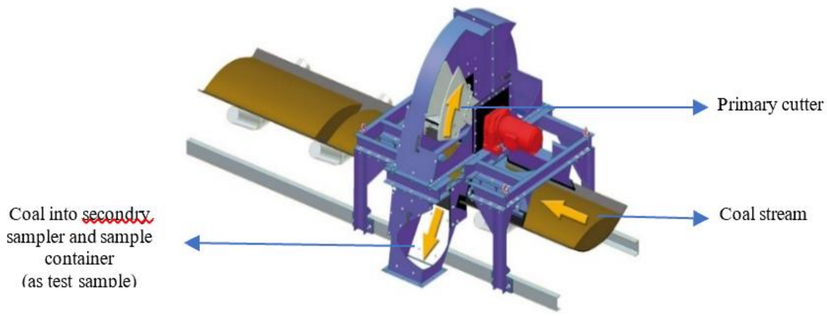


**Figure 3.** Sampling Used Mechanical Sampler (Primary Sampler)

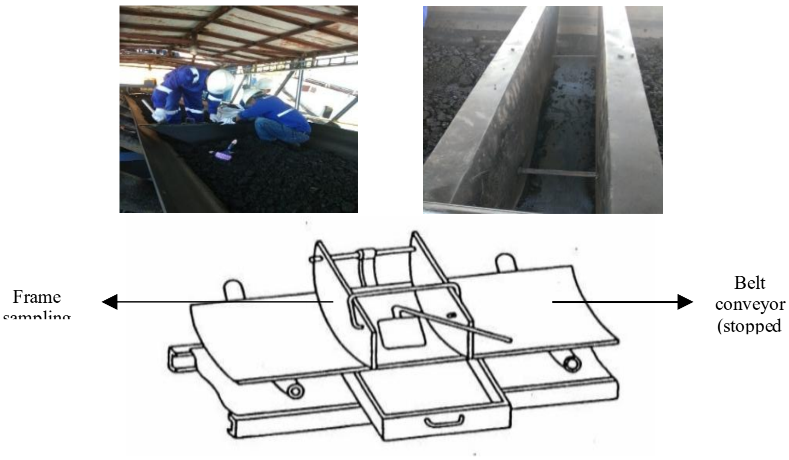


**Figure 4.** Sampling Manual Used Stopped Belt with Frame

### 2.3. Sample Analysis

The standard parameters to be included are moisture and ash (ash on a dry basis) [18]. The as received weight and air dried basis of all reference sample increments and test sample increments have been determined. Preparation and analysis of total moisture (percentage as received basis, "ar"), moisture in analysis (percentage air dry basis, "adb") and ash (percentage dry basis, "db") are conducted in accordance with ISO standards:

**Table 1.** Standard parameters dan standard method

| Parameter | Standard Method |
|---|---|
| Preparation [19] | ISO 13909 *Part* 4 : 2016 |
| Total moisture (% ar) [20] | ISO 589:2008 |
| Moisture in analysis (% adb) [21] | ISO 11722:2013 |
| Ash (% db) [22] | ISO 1171:2010 |

### 2.4. Statistical Analysis and Interpretation of Total Moisture and Ash

#### 2.4.1. Statistical Procedure for Outlier Identification

The formula used to calculate the value of C is:

$$C = \frac{d^2 max}{\sum_{i=1}^{i=n} di^2} \qquad (1)$$

C : Cochran's Criteria, critical value
dmax : the highest absolute value in the set of difference
n : number of pairs in the set

If the value of Cochran's Criteria (C) calculated is greater than the table value (appendix 2), dmax is identified as outlier.

#### 2.4.2. Calculation of Confidence Interval and Determining Bias

##### 2.4.2.1. Calculation of Paired Difference and Sample Variances and Covariances of the Difference

Assume that parameters of testing are moisture and ash, formula of calculation of average moisture difference ($\bar{d}m$) and average ash difference ($\bar{d}a$) is :

$$\bar{d}_m = \frac{1}{n} \sum_{n=1}^{n} d_{mi} \qquad (2)$$

$$\bar{d}_a = \frac{1}{n} \sum_{n=1}^{n} d_{ai} \qquad (3)$$

Calculated the sample variances $V_{mm}$ dan $V_{aa}$ of the moisture and ash difference, using the formula below :

$$V_{mm} = \frac{1}{n-1} \sum_{i=1}^{n} (dmi - \bar{d}m)^2 \qquad (4)$$

$$V_{aa} = \frac{1}{n-1} \sum_{i=1}^{n} (dai - \bar{d}a)^2 \qquad (5)$$

Sample covariances for moisture and ash difference, using the formula below :

$$V_{ma} = \frac{1}{n-1} \sum_{i=1}^{n} (dmi - \bar{d}m)(dai - \bar{d}a) \qquad (6)$$

#### 2.4.3. Determination of Bias

Compare the value of $T^2$ calculated from the bias test data to the value that shown in table appendix 3. If the calculation of value $T^2$ is greater than the table value, it's concluded that the system is bias, if the calculation of value $T^2$ is less than the table value, no bias detected. For 2 parameters (p =2), using the formula below :

$$T^2 = \frac{n}{(Vmm\ Vaa - Vma^2)} \left[ (\bar{d}a)^2 Vmm - 2\bar{d}m\,\bar{d}a\ Vma + (\bar{d}m)^2\ Vaa \right] \qquad (7)$$

## 3. RESULTS AND DISCUSSION

In this study, each pair of test and reference samples was subjected to an initial weighing to determine sample mass, followed by analysis of total moisture (% ar) and ash content (% db) parameters. The analytical results were statistically processed and interpreted to evaluate the bias within the mechanical sampling system.

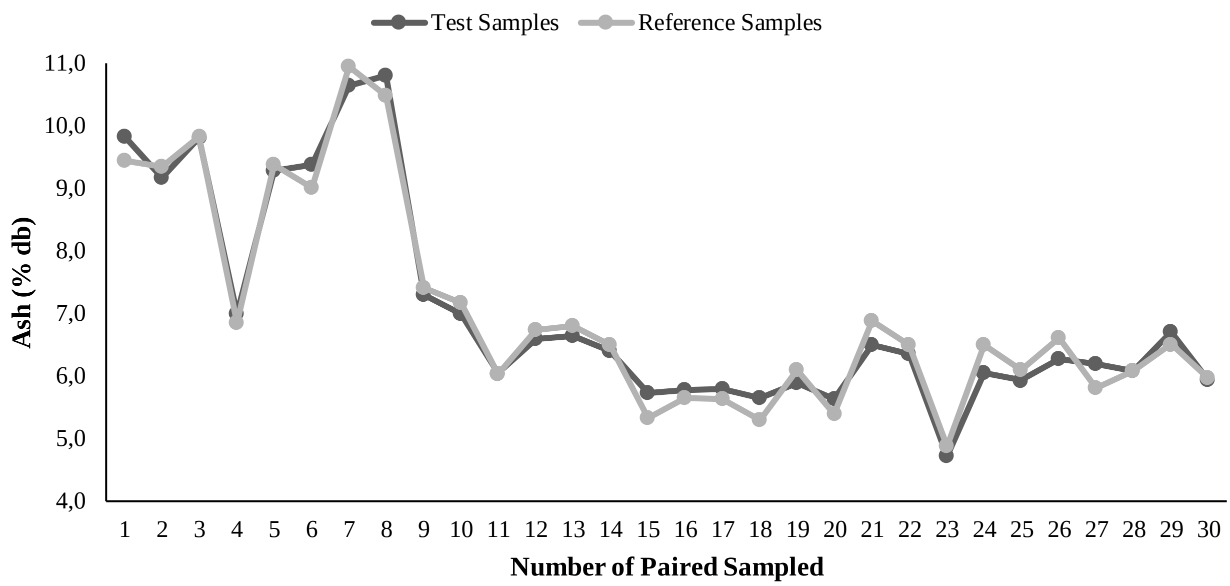


**Figure 5.** Analysis Result of Samples based on Parameter Total Moisture (% ar)

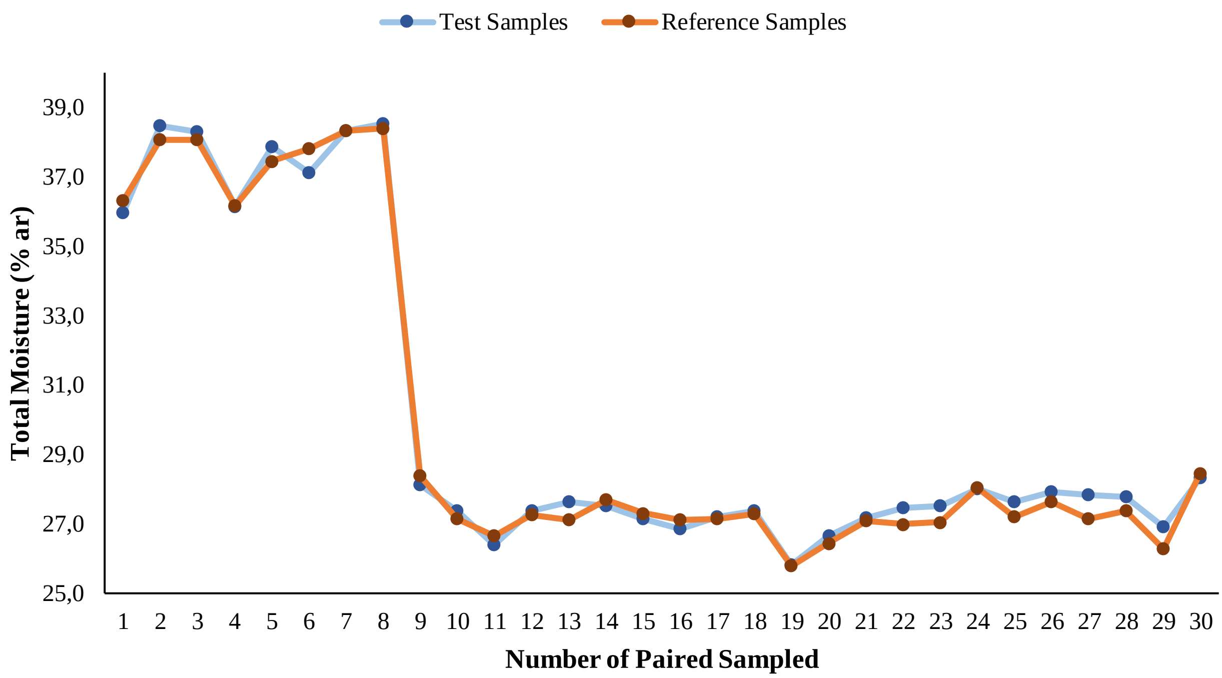


**Figure 6.** Analysis Result of Samples based on Parameter Ash (% db)

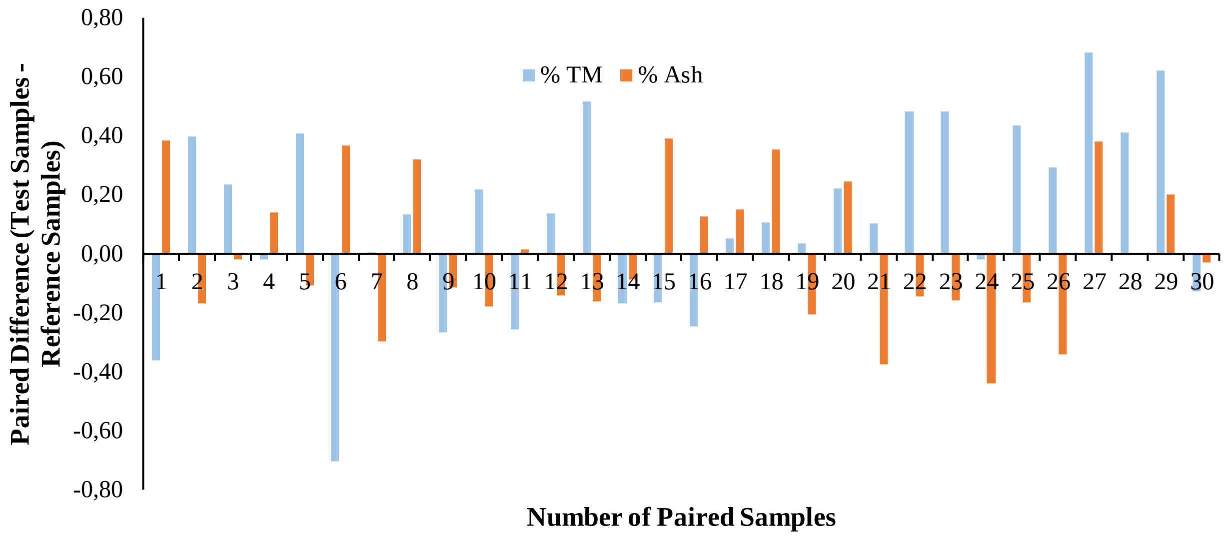


**Figure 7.** Paired difference (test – reference) for total moisture and ash

**Table 2.** Cochran's test for outlier (n = 30)

| Parameter | Total Moisture | Ash |
|---|---|---|
| Sum of squared values | 3.44 | 1.76 |
| Maximum squared differences | 0.49 | 0.19 |
| Rasio maximum (C) | 0.1424 | 0.1099 |
| Cochran's criteria (C) for n = 30 (see table appendix 1) | 0.363 | 0.363 |

**Table 3.** Calculation of paired difference and sample variances & covariances of difference total moisture and ash, determination of bias (n = 30 and p = 2)

| Calculation | Value |
|---|---|
| Average moisture differences | 0.1211 |
| Average ash differences | - 0.0019 |
| Sample variance for moisture differences | 0.1040 |
| Sample variance for ash difference | 0.0608 |
| Sample covariance for moisture and ash difference | - 0.0183 |
| $T^2$ | 4.425 |
| $T_0^2$ (n = 30, p = 2) | 6.885 |

Errors in sampling remain the largest component of total error, generally accounting for approximately 80% [9] of the total errors observed in sampling processes. These errors comprise both random errors and systematic errors. Systematic errors, also known as sampling bias, refer to recurrent inaccuracies often caused by the inaccuracy of the sampling mechanism, resulting in consistently higher or lower outcomes compared to an intrinsically unbiased reference sampling method. A sampling system with systematic errors or bias will consistently fail to represent the actual coal properties in a uniform manner, leading to inaccuracies in analytical results. Therefore, it is advisable to conduct periodic inspections of the sampling system to ensure its reliability.

Bias is identified as the deviation between the test results and the accepted reference values. These reference values are determined through simultaneous sampling of the test coal using both the reference method and the mechanical sampling system under evaluation. In addition to evaluating the deviation between test results and reference values, bias testing is a critical requirement in various scenarios, including the commissioning of newly installed sampling systems, the relocation of existing systems, and during system upgrades or modifications.

Bias test analysis is typically assessed using two primary parameters, moisture and ash. These parameters represent standard benchmarks commonly employed in bias testing procedures. Bias in ash (dry basis) is the most frequently observed and is predominantly attributed to inconsistencies in particle size distribution or coal particle segregation. In contrast, moisture bias may arise from a broader range of factors, including but not limited to particle size distribution errors, moisture loss during handling, or ventilation inefficiencies within the mechanical sampling system. This is also consistent with [11], which states that the testing of mechanical sampling systems is related to particle size. Furthermore, the study [23] highlights that the quality of coal combustion is also influenced by the total moisture content. An increase in total moisture reduces the caloric value . The research [23] found that not all mining locations have the same total moisture content. Several factors contribute to total moisture, such as handling, storage in stockpiles, and even the crushing process. Smaller coal particle sizes tend to have higher surface moisture, leading to an increase in total moisture. Additionally, coal handling plays a role in the potential formation of fines coal, which is correlated with ash content. A significant increase in fines coal formation is directly proportional to an increase in ash content, subsequently lowering the caloric value [4].

In line with its current predominant applications [6], coal quality significantly affects operational efficiency, particularly in boiler power plants. This is consistent with the findings [24], which indicate that boiler efficiency, flow rate, and heat loss depend on the coal's heating value. Thus, with coal quality closely matching the source data, energy demands can be met more efficiently, leading to reduced production costs [25].

The collected samples, each weighing as recorded (refers to sampling weight records in Appendix 1), comprised 30 pairs and were subsequently analyzed in the laboratory based on the two aforementioned parameters. The analytical results are presented in Table 2.

The data were then used to calculate the differences between the test samples and reference samples for each parameter, resulting in data as shown in Table 3. Subsequently, the squared differences for each parameter were calculated and summed, yielding the total squared values for each parameter as shown in Table 4: total moisture of 3.44 and ash of 1.76. Additionally, the maximum squared difference for each parameter was determined, with the total moisture having a value of 0.49 and ash 0.19. To assess the presence of outliers for each parameter, a ratio was calculated by dividing the maximum squared difference by the total squared sum (as per Equation 1) for each parameter. These ratios were then compared to the values in the table provided in Appendix 2 for $n = 30$. The calculated ratios were 0.1424 for total moisture and 0.1099 for ash. The Cochran's criterion for $n = 30$ is 0.363. Based on this comparison, no outliers were identified, as the ratio values were below the Cochran's criterion threshold.

Further statistical analysis involved calculating the mean difference for each parameter (according to Equations 2 and 3), yielding values of 0.1211 for total moisture and 0.0019 for ash. Following this, the sample variance of the differences for each parameter was calculated (as per Equations 5 and 6), resulting in values of 0.1040 for total moisture and 0.0608 for ash. The next step in the analysis was the calculation of the sample covariance between the parameters total moisture and ash (according to Equation 6), which resulted in a value of -0.0183.

Based on the calculations performed above, the next step is the computation of Hotelling's $T^2$ for the two parameters ($p = 2$) (as per Equation 7). The resulting value obtained was 4.425. By comparing this value to the critical value $T_0^2$ from the table in Appendix 3 for $n = 30$ and $p = 2$, which is 6.885, it can be concluded that the computed $T^2$ value is smaller than the critical value $T_0^2$. This indicates that no bias was detected in the mechanical sampling system.

## 4. CONCLUSION

Based on the results and discussion obtained in this study, it can be concluded that the new installation of mechanical system sampling indicates that no bias was detected. This is evidenced by the calculation of $T^2 < T_0^2$. This also shown that the stopped belt method which assumed that bias free could serve as reference sample for the acceptane value of the test samples. Therefore, the mechanical sampling system is deemed suitable and capable for operation.

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## REFERENCES

[1] Irwandi D., Alim Mas'ud Z., Sutriah K., & Khotib M. (2016). Preparation and Characterization of Carbon Foam Derived from Fine Coal and Phenolic Resin. Indonesia Journal of Chemistry, 16(3), 243

[2] U.S. Energy Information Administration. Coal Explained. http://eia.gov accessed on December 5 2024

[3] PT. Bukit Asam. Terjadinya Batubara. https://ptba.co.id accessed on October 2 2024

[4] Finkelman R.B., Dai S., French D. (2019). The Importance of Minerals in Coal as The Hosts of Chemical Elements : A Review. International Journal of Coal Geology, 212(2019), 1

[5] Zahratun N., Marisa O., Desmawita. (2020). Analisis Kualitas Batubara di Pit dan Stockpile dengan Metoda Analisis Proksimat di PT. Surya Anugrah Sejahtera Kecamatan Rantau Pandan Kabupaten Bungo Provinsi Jambi. Mine Magazine, 1(2), 1

[6] Pratama Afin A., & Tamtomo Kiono B.F. (2021). Potensi Energi Batubara serta Pemanfaatan dan Teknologinya di Indonesia Tahun 2020 – 2050 : Gasifikasi Batubara. Jurnal Energi Baru & Terbarukan, 2(2), 115

[7] Arisanti R., Yusuf M., & Faisal M. (2017). Study of The Effect of Coal Quality Parameters on Gas Methane ($CH_4$) Emission in Coal Fire for Sustainable Environment. Indonesian Journal of Enviromental Management and Sustainability., 1(1), 19-20

[8] Kentucky Geological Survey. Coal Rank. http://uky.edu accessed on December 5 2024

[9] Qian Zu. (2014). Coal Sampling and Analysis Standards (p. 23-47). London : IEA Clean Coal Centre

[10] International Standard. (2016). ISO 13909-2:2016(E) Hard Coal and Coke - Mechanical Sampling Part 2 : Coal – Sampling from Moving Streams. The International Organization for Standardization. 2nd Ed. Geneva : ISO

[11] Lyman G., Nel M., Lombard F., Steinhaus R., & Bartlett H. (2010). Bias Test of Cross Belt Samplers. The Journal of Southern African Institute of Mining and Metallurgy. 110(6), 290-291

[12] Murdani Muchsin D. (2020). Pengaruh Non-Probability Sampling Batubara terhadap Parameter Dry Ash Antara Certificate of Analysis Loading dan Unloading. KILAT, 9(1),2-3

[13] Office of Air and Radiation. (2009). The Coal Sector Proposed Rule for Mandatory Reporting of Greenhouses Gasses. Technical Support Document (pp 23). USA : U.S. Enviromental Protection Agency

[14] Mwangi, S. W. (2017). Probability and Non-Probability Sampling. Asian Research Journal of Business Management, 4(4), 115

[15] International Standard. (2016). ISO 13909-8 Hard Coal and Coke – Mechanical Sampling – Part 8 : Methods of Testing for Bias. The International Organization for Standardization. 2nd Ed. Geneva : ISO

[16] Kusniawati E., Pratiwi E., Nanda Yonika S. (2023). Analisa Pengaruh Nilai Total Moisture terhadap Gross Calorivic Value pada Batubara Jenis X di PT Bukit Asam Tbk Unit Pelabuhan Tarahan. Journal Of Innovation Research And Knowledge, 2(8), 3125-3216

[17] Fadhili M. A., & Ansosry. (2019). Analisis Pengaruh Perubahan Nilai Total Moisture, Ash Content dan Total Sulphur terhadap Nilai Kalori Batubara Bb-50 di Tambang Banko Barat PT. Bukit Asam, Tbk. Tanjung Enim Sumatera Selatan. Jurnal Bina Tambang, 4(3), 56

[18] Rose, C.D. (2012). Evaluation of Precision Being Obtained by a Three Stage Sampling System for Coal. In Proceedings of Sampling of Sampling 2012 (pp. 10). Perth, Australia : The Australasian Institute of Mining and Metallurgy

[19] International Standard. (2016). ISO 13909-4:2016 Hard Coal and Coke - Mechanical Sampling Part 4 : Coal–Preparation of Test Samples. The International Organization for Standardization. 2$^{nd}$ Ed. Geneva : ISO

[20] International Standard. (2008). ISO 589:2008(E) Hard Coal – Determination of Total Moisture. The International Organization for Standardization. 4$^{th}$ Ed. Geneva : ISO

[21] International Standard. (2013). ISO 11722:2013 Solid Mineral Fuels – Hard Coal - Determination of Moisture in the General Analysis Test Sample by Drying in Nitrogen. The International Organization for Standardization. 2$^{nd}$ Ed. Geneva : ISO

[22] International Standard. (2010). ISO 1171:2024 Coal and Coke - Determination of Ash. The International Organization for Standardization. 5$^{th}$ Ed. Geneva : ISO

[23] Taringan Rivaldo R., NSP Tanggara D., Apui Ganang N. M., Putu Putrawiyanta I., Iashania Y. (2024). Analisis Pengaruh Inherent Moisture dan Total Moisture terhadap Nilai Kalori Batubara pada Stockpile PT. Berau Coal Kalimantan Timur. Jurnal Global Ilmiah, 1(11), 771-772

[24] Pratama Sandi R. (2022). Pengaruh Kualitas Batubara terhadap Efisiensi Boiler Tipe Cfb Unit 3 PLTU Jeranjang Lombok Barat dengan Metode ASME PTC 4. Jurnal Teknik Mesin, 10(3), 86-91

[25] Wulandari S., Sipahutar R., Bizzy I. (2023). Analisis Pengaruh Parameter Operasi terhadap Kinerja Sistem Energi Termal pada PLTU Tanjung Enim 3x10 MW. Sebatik, 27(1), 60