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Method Verification of Carbon and Sulfur Determination in Ferronickel Tapping Samples Using HCS-801 in Accordance with GB/T 20123

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Abstract: Reliable determination of carbon (C) and sulfur (S) in tapping ferronickel is critical for metallurgical quality control and compliance with industrial specifications. Given the complex metallic matrix of ferronickel, verification of analytical performance is essential before routine application. This study verifies the GB/T 20123 standard method for the simultaneous determination of carbon and sulfur in tapping ferronickel using a High Carbon Sulfur (HCS-801) combustion–infrared analyzer. Method performance was assessed using accuracy, precision (repeatability), linearity, limit of detection (LOD), and limit of quantification (LOQ), in accordance with internationally accepted validation principles. Evaluation of recovery testing yielded 97.82 % for carbon and 99.04 % for sulfur, both within acceptable recovery ranges. Precision expressed as %RSD was 1.27% for carbon and 0.70% for sulfur, complying with the Horwitz criterion (% RSD < ½ CV Horwitz) and the general acceptance limit of 2 %. Excellent linearity was obtained across the calibration range, with correlation coefficients (r) of 0.998 for carbon and 0.9984 for sulfur. LOD and LOQ values, calculated based on the standard deviation of response and slope of calibration curves, were 0.0557% and 0.1855% for carbon, and 0.0123 % and 0.0409 % for sulfur, respectively. The results demonstrate that the GB/T 20123 method, when implemented with the HCS-801 analyzer, provides reliable analytical performance for carbon and sulfur determination in tapped ferronickel and is suitable for routine industrial applications.

Keywords: Ferronickel; Carbon and sulfur determination; Method verification; Combustion-infrared spectroscopy; Analytical performance.

1. Introduction

Carbon (C) and sulfur (S) are critical elements in ferroalloy materials, including ferronickel. Carbon plays a decisive role in determining mechanical properties such as hardness and shear strength, whereas sulfur, often considered an impurity element, can adversely affect ductility and increase susceptibility to cracking in steels and their alloys [1]. Therefore, the accurate determination of C and S contents in ferronickel samples is a crucial requirement for quality control in metallurgical industries [2].

High-temperature combustion followed by infrared (IR) absorption detection is one of the most widely applied analytical techniques for determining carbon and sulfur in metallic alloys and ferroalloys. In this method, the sample is combusted in an oxygen-rich atmosphere, converting carbon to CO_2 and sulfur to SO_2. These gases are subsequently quantified using infrared detectors, enabling accurate and simultaneous determination of the target elements [3].

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National and international standards, including the Gui Biao Tui Jian (GB/T) 20123 standard of the People's Republic of China, establish validated procedures for the determination of total carbon and sulfur in metallic materials using infrared combustion methods. Such standards define essential analytical performance characteristics, accuracy, precision, linearity, limit of detection (LOD), and limit of quantification (LOQ) to ensure the reliability of generated data [4]. However, the implementation of this method for industrial materials such as tapping ferronickel, a primary product of ferronickel smelting, remains relatively underreported, particularly in terms of comprehensive method verification. Method verification, encompassing systematic evaluation of performance parameters such as accuracy, precision, linearity, LOD, and LOQ, is essential to demonstrate fitness for purpose in quality control and laboratory accreditation frameworks [5].

Previous studies have highlighted the importance of analytical method verification as a foundation for establishing reliable testing procedures [6]. For instance, verification studies using spectrophotometric techniques for sodium determination in nickel ore according to GB/T standards demonstrated acceptable precision, linearity, and detection limits under laboratory conditions [7]. Similar investigations involving elemental analysis in environmental and mineral matrices (e.g., Mn in drinking water and Zn in sulfide concentrates) further emphasize the necessity of evaluating LOD, LOQ, and other performance parameters to ensure scientific defensibility of analytical results [8].

Despite these advancements, scientific literature specifically addressing the comprehensive verification of carbon and sulfur determination in tapping ferronickel using a High Carbon Sulfur (HCS) 801 instrument in accordance with GB/T 20123 remains limited. Existing studies often focus primarily on instrumental principles or partial validation parameters rather than conducting full performance verification aligned with internationally recognized analytical standards [9]. Furthermore, ferronickel matrices typically contain relatively high carbon concentrations compared to ores or concentrates, which may influence analytical linearity, repeatability, and detection capability, thereby necessitating dedicated verification studies [10].

Based on these considerations, this study aims to perform a comprehensive methodological verification of carbon and sulfur determination in tapping ferronickel using the HCS 801 instrument in accordance with GB/T 20123. The evaluation focuses on accuracy, precision, linearity, LOD, and LOQ to ensure compliance with established analytical performance criteria. The findings are expected to strengthen the scientific basis for routine industrial analysis and contribute to enhancing quality assurance practices in ferronickel production laboratories.

2. Research and Methodology

2.1 Materials

The materials used in this study included industrial tapping ferronickel samples, ceramic crucibles, Certified Reference Materials (CRMs), accelerator catalysts (SES A and SES C), and high-purity oxygen gas. The instruments employed were a High Carbon Sulfur (HCS) 801 analyzer, an analytical balance, metal tongs, a catalyst spoon, and a furnace.

2.2 Experimental Procedure

The procedure for verifying the analytical method for carbon and sulfur determination in tapping ferronickel samples using the HCS 801 instrument in accordance with GB/T 20123 is described as follows.

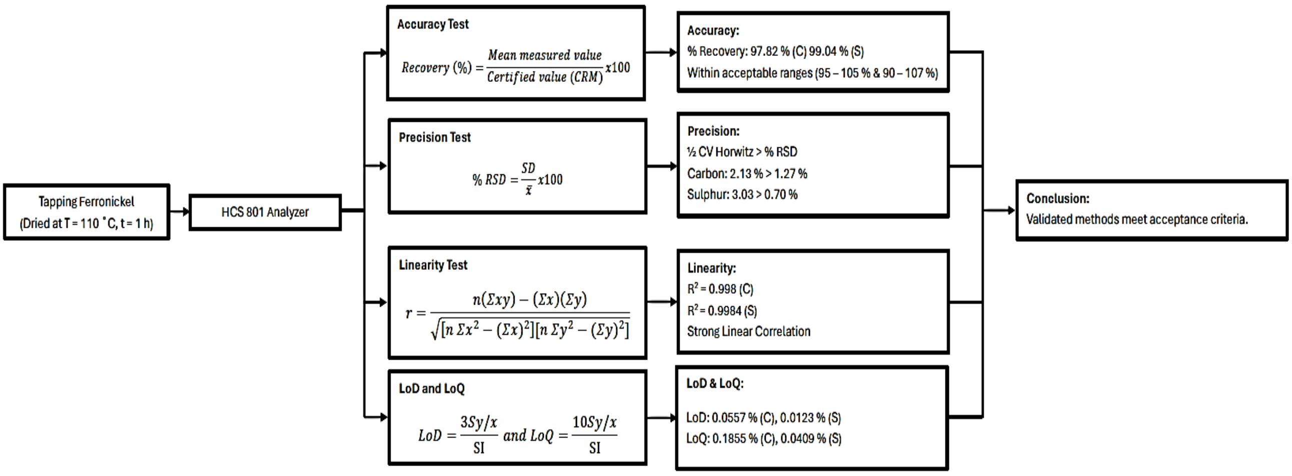

Figure 1. Graphical Abstract. Method verification workflow for carbon and sulfur determination in ferronickel tapping samples using HCS-801 based on GB/T 20123.

2.2.1 Sample Preparation

The samples were dried in an oven at 110 °C for 1 hour to remove moisture. Ceramic crucibles were pre-ignited at 1000 °C for 4 hours to eliminate residual humidity and potential contaminants. An appropriate amount of accelerator (SES A and SES C) was then added to the sample to ensure complete combustion during analysis [11]. Subsequently, the crucible containing the prepared sample was placed into the induction furnace of the HCS 801 analyzer. The sample was combusted at high temperature under a stream of high-purity oxygen to oxidize carbon to CO_2 and sulfur to SO_2. The resulting gases were passed through an infrared detection cell, where their concentrations were quantitatively measured. Based on the detected CO_2 and SO_2 signals, the carbon and sulfur contents in the sample were accurately determined.

2.2.1 Accuracy Test

Accuracy refers to the closeness of agreement between the measured value and the true value, reflecting the degree of analyte recovery during measurement [12]. This test was conducted to evaluate the agreement between experimental results and certified values using a carbon CRM (0.74%) and a sulfur CRM (0.071%). Accuracy data analysis used standard sample analysis (CRM) data, tested using the HCS 801 instrument five times. Data analysis was carried out using equations [13]:

$$Recovery\ (\%) = \frac{Mean\ measured\ value}{\text{Certified value (CRM)}}\ x\ 100\% \tag{1}$$

2.2.2 Precision Test

Precision is defined as the degree of agreement among individual test results when the analytical procedure is applied repeatedly to the same sample under specified conditions [14]. The precision test was performed using analytical results obtained from tapping ferronickel samples analyzed with the HCS 801 instrument at closely spaced time intervals (every 2 minutes). Data analysis was conducted using the following equations [15][16]:

$$SD = \sqrt{\frac{\Sigma(x-\bar{x})}{n-1}} \tag{2}$$

$$\%\ RSD = \frac{\text{SD}}{\bar{x}}\ x\ 100\% \tag{3}$$

Where:
SD = Standard Deviation
x = individual test result
x̄ = mean value
n = number of observations
RSD = Relative Standard Deviation

The calculated %RSD values were compared with the Horwitz coefficient of variation (CV Horwitz) to determine precision acceptability [17]. The Horwitz equation is expressed as:

$$CV\ Horwitz = 2^{(1-0.5\log c)} \tag{4}$$

where *c* represents the analyte concentration expressed as a mass fraction.

For repeatability conditions, the acceptance criterion for precision is:

$$\%RSD < \frac{1}{2}(2^{(1-0.5\log c)}) \tag{5}$$

2.2.3 Linearity Test

Linearity refers to the ability of an analytical method to produce results that are directly proportional to the analyte concentration within a specified range [18]. Linearity for sulfur determination was evaluated using calibration curves constructed from six CRMs with known concentrations. The carbon concentration levels were 0.115%, 0.303%, 0.533%, 0.74%, 0.90%, and 1.10%, while sulfur concentrations were 0.034%, 0.071%, 0.129%, 0.180%, 0.213%, and 0.283%. Each CRM was analyzed in five replicate measurements using the HCS 801 instrument. The calibration curve generated a linear regression equation (*y* = *a* + *bx*) and a coefficient of determination (R^2). The slope (*b*), intercept (*a*), and correlation coefficient (*r*) were calculated using least squares regression according to the following equations [19]:

$$a = \frac{(\Sigma y)(\Sigma x^2)-(\Sigma x)(\Sigma xy)}{n\,\Sigma x^2-(\Sigma x^2)} \tag{6}$$

$$a = \frac{n(\Sigma xy)-(\Sigma x)(\Sigma y)}{n\,\Sigma x^2-(\Sigma x^2)} \tag{7}$$

$$r = \frac{n(\Sigma xy)-(\Sigma x)(\Sigma y)}{\sqrt{[n\,\Sigma x^2-(\Sigma x)^2][n\,\Sigma y^2-(\Sigma y)^2]}} \tag{8}$$

where:
n = number of data points
X = independent variable (concentration)
Y = dependent variable (instrument response)

2.2.3 Limit of Detection (LOD) and Limit of Quantification (LOQ)

The Limit of Detection (LOD) is defined as the lowest concentration of analyte that can be detected but not necessarily quantified with acceptable precision and accuracy [20]. In contrast, the Limit of Quantification (LOQ) represents the lowest analyte concentration that can be quantitatively determined with acceptable precision and accuracy [21]. LOD and LOQ were statistically calculated from the linear regression of the calibration curve obtained from five replicate analyses of standard samples using the HCS 801 instrument. The calculations were based on the following equations [22]:

1) Residual standard deviation (Sy/x):

$$Sy/x = \frac{\sqrt{\Sigma(y-yi)^2}}{\mathrm{n-2}} \tag{9}$$

2) LoD:

$$LoD = \frac{3Sy/x}{\mathrm{SI}} \tag{10)}$$

3) LoQ:

$$LoQ = \frac{10Sy/x}{\mathrm{SI}} \tag{11}$$

Where: Sy/x = Residual Standard Deviation
y = observed value
yi = the result of substituting the value of x into the equation yi = a + bx
n = number of data points
SI = slope of the calibration curve

2.3 Product characterization

All samples used in the method validation were analyzed using the High Carbon Sulfur (HCS) 801 instrument. The HCS 801 operates based on the combustion-infrared (IR) absorption principle for the simultaneous determination of carbon and sulfur content. In this method, the sample is combusted at high temperature in a stream of high-purity oxygen in the presence of an accelerator to ensure complete oxidation. Carbon and sulfur in the sample are quantitatively converted into carbon dioxide (CO_2) and sulfur dioxide (SO_2), respectively. The generated gases are transported through infrared detection cells, where CO_2 and SO_2 selectively absorb infrared radiation at their characteristic wavelengths. The reduction in infrared intensity detected is directly proportional to the concentration of each gas, enabling quantitative determination of carbon and sulfur contents based on calibrated response factors [23].

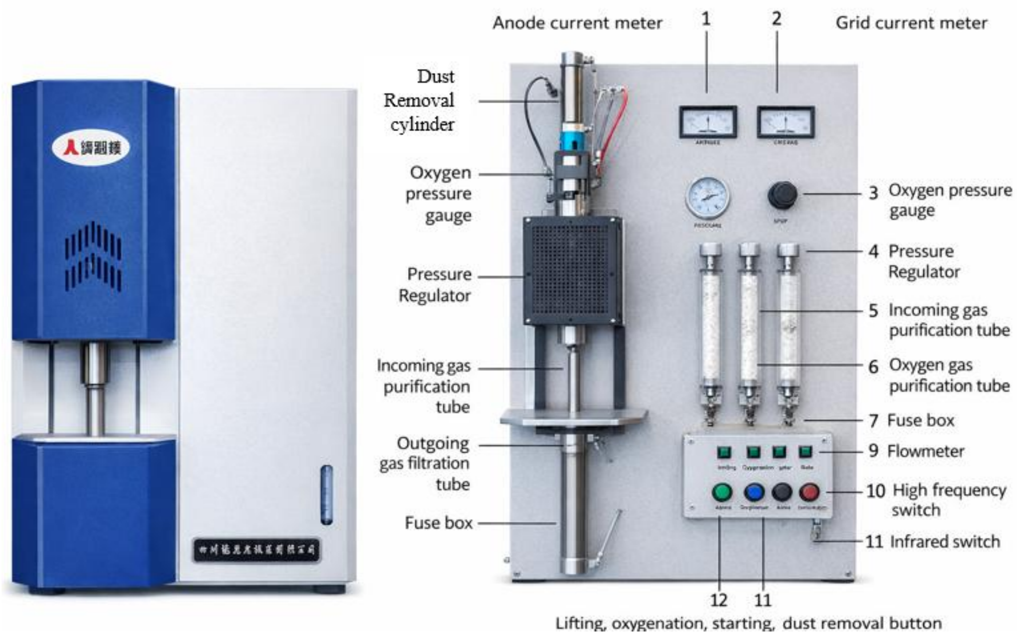

Figure 1. Schematic diagram of the High Carbon Sulfur (HCS) 801 analyzer and its main components.

3. Results and Discussion

3.1 Accuracy

Accuracy was evaluated by analyzing the Certified Reference Material (CRM) GBW(E)010421 in five replicate measurements. The obtained results were compared with the certified concentration values stated in the CRM certificate.

Table 1. Analytical results for accuracy testing

Replicate	Measured Carbon Concentration	Measured Sulfur Concentration
1	0.7317	0.0715
2	0.7111	0.0703
3	0.7287	0.0708
4	0.7259	0.07
5	0.7219	0.069
Mean Value	0.72386	0.07032
Certified CRM Concentration	0.74	0.071
% Recovery	97.82	99.04

Based on Table 1, the accuracy test for carbon using a CRM with a certified concentration of 0.74% yielded a recovery value of 97.82%. This result indicates that the accuracy is acceptable, as the analyte concentration falls within the range of 0.1% < A < 1%, for which the acceptable recovery range is 95%–105% [24]. Similarly, the accuracy test for sulfur using a CRM with a certified concentration of 0.071% resulted in a recovery value of 99.04%. This demonstrates that the accuracy is acceptable, as the analyte concentration is within the specified range and the recovery falls within the acceptable limits of 90%–107% [24]. Recovery values exceeding 100% are acceptable as long as they are within tolerance limits, as they reflect the random variation and measurement uncertainty inherent in analytical procedures. This is in line with the principle that accuracy is a combination of random and systematic errors, within which 95%–105% of positive readings can be accommodated without compromising the validity of the method [25]. From the results obtained, it can be concluded that the analytical method shows good accuracy.

3.2 Precision

Precision in this study was determined by measuring the sample seven times using the HCS 801 instrument. The objective of precision testing was to evaluate the closeness of agreement among the seven replicate measurements. Precision is considered satisfactory when repeated measurements yield results that do not differ significantly from one another. It was assessed by calculating the standard deviation (SD) of the replicate measurements, dividing it by the mean value, and multiplying by 100 to obtain the percent Relative Standard Deviation (%RSD) [24]. The obtained %RSD values were then compared with ½ CV Horwitz to evaluate acceptability [26]. All measurements were performed under repeatability conditions.

Table 2. Results of Precision Testing for Carbon and Sulfur Determination

Replicate	Carbon analytical result	Sulfur analytical result
1	0.6694	0.062
2	0.6459	0.063
3	0.644	0.0631
4	0.6509	0.0634
5	0.6541	0.0631
6	0.6559	0.0629
7	0.6534	0.0629

Replicate	Carbon analytical result	Sulfur analytical result
Mean value	0.6534	0.0629
SD	0.0083	0.00044
%RSD	1.2707	0.6954
%CV Horwitz	4.2646	6.0655
1/2 %CV Horwitz	2.1323	3.0327

Based on Table 2, the standard deviation obtained for carbon was 0.0083, while for sulfur it was 0.00044. The calculated %RSD values were 1.2707% for carbon and 0.6954% for sulfur. A method is considered to have good precision when the %RSD value is lower than ½ CV Horwitz. The calculated CV Horwitz values were 4.2646% for carbon and 6.0655% for sulfur, while the corresponding ½ CV Horwitz values were 2.1323% and 3.0327%, respectively. Since the %RSD values for both carbon and sulfur are lower than the corresponding ½ CV Horwitz values, the analytical method demonstrates acceptable precision and meets the established acceptance criteria [26].

3.3 Linearity

Linearity for carbon determination was evaluated using calibration curves constructed from CRMs with known concentrations of 0.115%, 0.303%, 0.533%, 0.74%, 0.90%, and 1.10%. Similarly, linearity for sulfur determination was assessed using CRMs with certified concentrations of 0.034%, 0.071%, 0.129%, 0.180%, 0.213%, and 0.283%. The data used to construct the calibration curves are presented in Tables 3 and 4.

Table 3. Analytical results for sulfur linearity testing

Concentration	Test 1	Test 2	Test 3	Test 4	Test 5	Mean
0.034	0.033	0.0345	0.034	0.0335	0.03	0.0330
0.071	0.0715	0.0703	0.0708	0.07	0.069	0.0703
0.129	0.1269	0.1249	0.1246	0.1247	0.1239	0.1250
0.18	0.1742	0.1711	0.1729	0.1727	0.1793	0.1740
0.213	0.2155	0.2178	0.2185	0.2178	0.2155	0.2170
0.283	0.2871	0.2878	0.2869	0.2892	0.289	0.2880

Table 4. Analytical results for carbon linearity testing

Concentration	Test 1	Test 2	Test 3	Test 4	Test 5	Mean
0.115	0.1056	0.106	0.1068	0.1048	0.1076	0.1062
0.303	0.3191	0.3173	0.312	0.3156	0.3179	0.3164
0.533	0.5248	0.517	0.5199	0.5177	0.5296	0.5218
0.74	0.7317	0.7111	0.7287	0.7259	0.7219	0.7239
0.9	0.9313	0.933	0.921	0.9291	0.9359	0.9301
1.1	1.095	1.1115	1.105	1.0915	1.113	1.1032

Based on the standard data for carbon and sulfur presented in Tables 3 and 4, calibration curves were constructed to obtain linear regression equations in the form $y = ax + b$, where y represents the measured response for carbon or sulfur, a is the slope, and b is the intercept. The relationship between the standard concentrations and the measured results is illustrated in Figures 1 and 2.

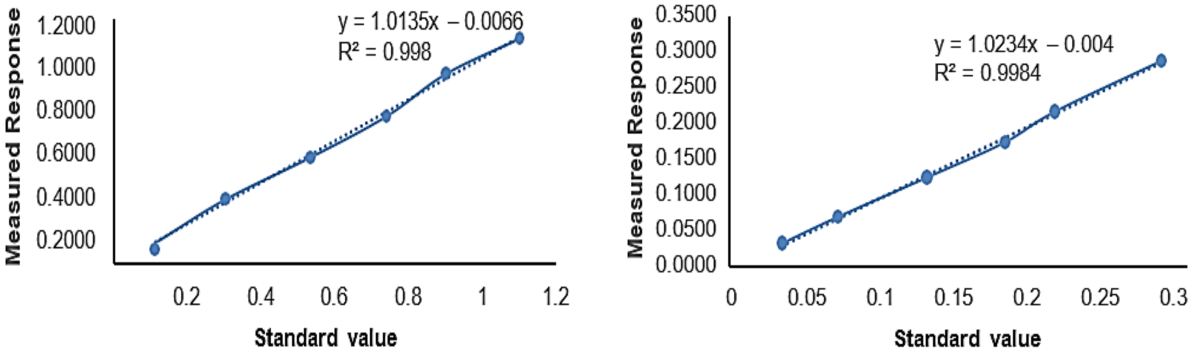

Figure 2. (a) Carbon standard calibration curve, (b) Sulfur standard calibration curve.

Based on the constructed calibration curves, the correlation coefficient and coefficient of determination were obtained, as presented in Table 5.

Table 5. Calibration curve parameters for carbon and sulfur standards

Component	Slope	Intercept	Coefficient of Determination (R^2)
CRM carbon	1.0135	0.0066	0.998
CRM sulfur	1.0234	0.004	0.9984

According to Table 5, the linear regression equation for carbon was obtained as $y=1,035x–0,0066$, with a coefficient of determination (R^2) of 0.998. For sulfur, the linear regression equation was $y = 1.0234x + 0.004$, with an R^2 value of 0.9984. The obtained coefficients of determination indicate a strong linear correlation between the standard concentrations and the measured responses. An R^2 value closer to 1 reflects a better fit of the regression model to the experimental data [24]. The high R^2 values for both carbon (0.998) and sulfur (0.9984) demonstrate an excellent linear relationship between the concentration variables and the corresponding analytical responses. Furthermore, according to Kuntari [27], A calibration curve is considered acceptable when the coefficient of determination is ≥ 0.997. Therefore, the results obtained in this study confirm that the method meets the required linearity criteria within the tested concentration ranges.

3.5 Limit of Detection (LOD) and Limit of Quantification (LOQ)

The LoD and LoQ values were determined from the sulfur and carbon standard calibration curves. The LoD represents the lowest concentration of an analyte that can be detected by the instrument, although not necessarily quantified with acceptable accuracy [28]. In contrast, the LOQ indicates the lowest concentration at which the analyte can be quantitatively determined with acceptable precision and accuracy [12]. Thus, LOD and LOQ are critical parameters in method verification, as they define the minimum detectable and quantifiable concentrations measurable by the instrument. These parameters were calculated using the linear regression equation ($y = a + bx$), where a is the intercept and b is the slope. The LOD was calculated as three times the residual standard deviation (Sy/x) divided by the slope, while the LOQ was calculated as ten times (Sy/x) divided by the slope. The results are presented in Tables 6 and 7.

Table 6. LoD and LoQ determination for carbon

Parameter	Value
Slope	1.0135
Intersep	0.0066
n	6
Sy/x	0.01880
LoD	0.0557
LoQ	0.1855

Based on Table 6, the residual standard deviation Sy/x or carbon was 0.01880. The calculated LoD for carbon was 0.0557 %, indicating that 0.0557 % is the lowest standard concentration detectable by the instrument. The LoQ value was 0.1855 %, representing the lowest concentration that can be quantitatively determined with acceptable analytical precision.

Table 7. LoD and LoQ determination for sulfur

Parameter	Value
Slope	1.0135
Intersep	0.004
n	6
Sy/x	0.0042
LoD	0.0123
LoQ	0.0409

For sulfur (Table 7), the residual standard deviation Sy/x was 0.0042. The calculated LOD was 0.0123%, indicating that this is the minimum detectable concentration. The LOQ value was 0.0409%, representing the lowest concentration that can be quantified with acceptable analytical performance. Based on these results, the LOD and LOQ values meet the acceptance criteria, as the method demonstrates adequate sensitivity, accuracy, and precision within the evaluated concentration ranges [29].

4. Conclusion

Based on the conducted study, it can be concluded that the accuracy values for carbon and sulfur, as indicated by the percentage recovery, met the acceptance criteria, with values of 97.8189% (acceptable range: 95 %-105 %) and 99.0422 % (acceptable range: 90 %-107 %), respectively. The precision values for carbon and sulfur, evaluated by comparing % RSD with ½ CV Horwitz, also met the acceptance criteria, where 2.1323 % > 1.2707 % for carbon and 3.0327 % > 0.6954 % for sulfur. The linearity assessment showed coefficients of determination (R^2) of 0.998 for carbon and 0.9984 for sulfur, indicating excellent linear relationships within the tested concentration ranges. The LoD values obtained from the carbon and sulfur standards were 0.0557 % and 0.0123 %, respectively, while the LoQ values were 0.1855% for carbon and 0.0409 % for sulfur. Therefore, it can be concluded that the method verification results, including accuracy, precision, linearity, LoD, and LoQ parameters, meet the established acceptance criteria.

Conflict of Interest: The authors declare no conflicts of interest.

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