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Instrumental Testing of Quasi-Binary Ore Materials by the Gamma-Albedo Method

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Abstract. It is noted that the traditional (standard) method of testing ore materials, due to its high labor intensity and low representativeness, cannot serve as the basis for controlling the quality of ores during their extraction and processing. The proposed spectrometric modification of the gamma-albedo method allows eliminating labor-intensive operations of ore grinding, preparing analytical samples (~0.1 mm) and direct chemical analysis for iron. Studies of the spectral distribution of scattered gamma radiation from the Se-75 radioisotope source (~220 keV) on ores with different iron content revealed critical energies and zones of their displacement depending on the ore quality and the energy of scattered gamma radiation. The critical energies found in the spectral distribution of gamma radiation albedo allowed selecting the optimal energy intervals in the hard (above the critical energy) and soft (below the critical energy) regions of the spectrum from the point of view of the maximum contrast of the ratio Ψ value of the scattered gamma radiation intensities measured in the selected energy intervals, with changing the iron concentration. In the course of testing the proposed modification of the method of monitoring the iron content in partially prepared samples weighing ~ 60 kg, with the size of 40 mm, there was achieved the mean square error of 1.32% abs. Reliability of the achieved accuracy is confirmed by the comparative value of the relative mean square deviation of the ratio Ψ value of intensities calculated on the basis of multiple measurements of a partially prepared sample.

Keywords: instrumental testing, gamma radiation albedo, critical energies, standard testing method, ore heterogeneity error, method sensitivity, partially prepared samples

Introduction

The accuracy and reliability of instrumental testing are usually assessed by comparing its data with the results of traditional (standard) testing, the final stage of which is most often the chemical analysis of iron ore material for iron content. With this approach, the results of standard testing are usually accepted as true. Hence, the actions of researchers are aimed at minimizing random discrepancies between the instrumental method and the data of chemical analysis, taking into account the tolerances adopted by the relevant standards [1-2]. However, these tolerances apply only to the final stage of testing.

The standard testing method is characterized by high labor intensity and relatively low representativeness. This is due to the fact that the testing process involves performing lengthy operations: selection of primary (spot) samples from the sampled batch of ore; crushing the combined sample to the size of 40 mm, averaging, reducing and crushing to 10 mm and 3 mm; averaging, reducing and crushing to the size of 0.16 mm; direct chemical analysis of analytical samples for iron content.

The ore heterogeneity creates the problem of its representativeness, i.e. correspondence of the iron concentration in the selected sample and the batch being tested. The error in representativeness of testing (the ore heterogeneity) is the error caused by the fact that the iron content in the sample subjected to direct analysis can differ from its content in the batch being tested.

A significant contribution to the error in determining the iron content in the existing iron ore testing regulated by the Interstate Standard is made by the error of representativeness, which is formed when selecting primary samples from the controlled batch of material [1]. This error is caused by segregation phenomena and statistical fluctuations in the number of iron-containing mineral inclusions in the primary sample. To minimize the effect of segregation, it is envisaged to form a combined sample from the number of selected primary samples. To minimize the effect of statistical fluctuation, it is envisaged to select point samples with the mass selected depending on the size.

Studies have shown that the greatest contribution to the overall [3] error of standard testing is made by the selection of primary samples. The selection error $\sigma_{\text{О}}$ characterizes the difference between the iron content in the controlled lot and the combined sample.

In the process of crushing the initial combined sample and subsequent preparation of a sample with the size of up to 40 mm, an error $\sigma_{\text{П}}$ occurs, which characterizes the difference between the iron content in the combined sample and the prepared sample with the size of up to 40 mm.

At subsequent stages of testing (grinding, averaging, reduction and preparation of samples of analytical size, an error $\sigma_{\text{И}}$ occurs, which characterizes the difference between the iron content in the sample prepared up to 40 mm and samples of the analytical size.

At the final stage of standard testing, the error $\sigma_{\text{Х}}$ occurs, which characterizes the difference between the results of chemical analysis of analytical samples for iron content.

Considering the independence of errors at each stage of testing and the additivity of variances of individual components, the total testing error σ can be represented in the form:

$$\sigma = \sqrt{\sigma_S^2 + \sigma_P^2 + \sigma_G^2 + \sigma_C^2}\ , \tag{1}$$

where σ_S^2 is the variance that characterizes the sampling error;

σ_P^2 is the variance that characterizes the error of preparing a sample at the stage of crushing;

σ_G^2 is the variance that characterizes the rro at the stage of grinding, reducing and preparing analytical samples;

σ_C^2 is the variance that characterizes the error of chemical analysis.

Thus, the algorithm for selecting primary samples and subsequent stages of sample preparation should ensure that the concentration of iron C_{Fe} in the combined sample corresponds to its value in the sampled ore batch with an error not exceeding the error permitted by the standard.

The standard sampling method according to SS [1] regulates the minimum mass of the selected primary samples and their number depending on the mass of the sampled ore batch, its size and the magnitude of the variation in the quality of the ore by iron content. An important point is the selection of primary samples. Experience shows that, in general, the sampling error depends to a greater extent not on the mass of the selected primary sample, but on the number of samples that make up the combined sample. In general, the iron content in the combined sample will differ from the true iron content in the sampled ore batch.

The standard deviation of the iron concentration in point samples from the iron concentration in the sampled batch of ore can be taken as the parameter characterizing the sampling error σ_S due to heterogeneity. The sampling error was experimentally determined based on the magnitude of the variation in quality (iron concentration) in the selected point samples:

$$\sigma_0 = \sqrt{\sum_{i=1}^{n} \frac{\bar{C} - C_i}{n-1}}\ , \tag{2}$$

where $\bar{C}$ is the mean value of C_{Fe} вin point samples;

n is the number of point samples.

For sufficiently large values of n, the value of $\bar{C}$ will approach the true concentration of iron in the analyzed batch of ore. The value of quality variation for iron ores of the Lisakovsk deposit (Kazakhstan) estimated according to SS [4], was 2.42%. Hence, the number of point samples n, depending on the specified sampling error, is found using the formula:

$$n = (k \cdot \sigma / \beta_0)^2, \tag{3}$$

where k is the coefficient equal to 2 with 95% -th probability;

σ is the standard deviation of the iron content in primary samples;

β_0 is the specified sampling error, %.

The specified sampling error β_0 changing within the range of (0.90-0.37) depending on the mass of the sampled ore batch, means that on average in 95% of cases the iron content in the combined sample will not differ from its content in the sampled batch by more than β_0 percent.

When sampling an ore batch weighing up to 500 tons and the quality variation value equal to 2.42%, the optimal sampling parameters were found: the minimum mass of spot samples 8 kg; the minimum number of spot samples 29; the mass of the combined sample 232 kg.

In the process of sample preparation, along with crushing and grinding, mixing and reducing the sample is a mandatory operation. SS [1] regulates the minimum required sample mass m after reduction depending on the size and quality variation of the ore

$$m = k_1 \cdot d_{max}^2, \tag{4}$$

where k_1 is the coefficient that depends on the quality variation;

d_{max} is the size of the maximum piece, mm. The size of the maximum piece is taken as the size of the sieve opening on which no more than 5% of the material remains after sifting.

With the selected k_1= 0.1 (ore of the great quality variation) and the size to 40 mm, the needed sample mass made 160 kg.

The standard deviation of C_{Fe} in samples crushed to 40 mm from the iron concentration in the combined sample characterizes the error of ore heterogeneity at this stage of sample preparation. This error $\sigma_{П}$ estimated by the discrepancy in the iron concentration in samples of this size class, was 1.26%.

A similar approach was used to estimate the heterogeneity at the subsequent stage of testing (grinding, mixing, reducing and preparing samples of analytical size (~0.16 mm)). The error at this stage of sample preparation $\sigma_{И}$ was 0.84%.

The error of the final stage of testing that characterizes the discrepancy in the results of chemical analysis of analytical samples, was 0.5%.

Thus, the total error of the standard sampling method, estimated according to expression (1) taking into account individual components ($\sigma_{о} = 2.42\%, \sigma_{П} = 1.26\%, \sigma_{И} = 0.84\%, \sigma_{Х} = 0.5\%$), made 2.9% abs.

2. Research methods

The problems of ensuring the necessary accuracy and reliability of traditional testing methods are especially relevant in connection with the intense development of instrumental nuclear-physical methods of quality control of various raw materials [5-8]. In this case, the metrological characteristics of instrumental methods are usually assessed by a simple comparison of the results of instrumental and chemical analyses, unreasonably neglecting significant errors at the labor-intensive stages of traditional testing. In this regard, the error of the standard testing method should be considered as an acceptable limit of the accuracy of instrumental methods. Therefore, the accuracy and reliability of nuclear testing methods cannot be assessed by direct comparing their data with the results of chemical analysis.

At present, standard testing is mainly used at Kazakhstan mining and metallurgical enterprises, the final stage of which is chemical analysis. In some cases, chemical analysis is successfully replaced by nuclear-physical methods.

The X-ray fluorescence method that consists in measuring the X-ray fluorescence of the elements being determined is widely used for the analysis of quasi-binary materials [9-10].

Various modifications of this method are characterized by relatively high sensitivity and sufficient accuracy of quantitative analysis of finely ground material of analytical size (~0.1 mm). X-ray fluorescence of iron is used to determine the pyrite content in coals.

The fundamental feature of the new modification of the method is selection of optimal energy parameters in the area of analytical lines of sulfur, iron and scattered gamma radiation. The capabilities of the X-ray fluorescence method are limited to the analysis of finely ground samples. Labor-intensive sample preparation processes and low representativeness of analytical samples are retained.

The value of the gamma radiation albedo is widely used in the practice of studying various metals and quasi-binary materials [8-11]. Depending on the energy of the primary and registered scattered gamma radiation and the probe parameters, enhanced metrological characteristics of the gamma-albedo method are achieved. The novelty of the two-probe study of complex substances consists in measuring the energy spectra of scattered gamma radiation at different probe lengths, finding critical energies corresponding to the maximum in the albedo spectra of gamma radiation and optimal energy intervals that ensure maximum sensitivity to the effective atomic number of the substance being studied.

A distinctive feature of the gamma annihilation method is registration of annihilation radiation arising from the effect of electron-positron pair formation. The proportionality of the macro cross-section of pair formation from the atomic number (effective atomic number for substances) is the basis for the analysis of quasi-binary materials, in particular coals [12]. To increase sensitivity, an improved technique has been developed that is distinguished by the choice of the optimal probe length for annihilation gamma radiation and the optimal energy range for scattered gamma radiation. The gamma annihilation method can be successfully applied in the analysis of quasi-binary materials containing heavy elements.

The neutron activation method is widely used for elemental analysis in materials science and mining and geological studies due to its high sensitivity and selectivity of the analysis due to the possibility of taking into account competing nuclear reactions, the choice of time and energy parameters [13]. The high labor intensity of the multi-stage activation method and the low representativeness of the analysis (activation sample 1-2 grams) serve as constraints.

Modifications of the neutron-gamma method of inelastic scattering of fast neutrons and radiative capture of thermal neutrons are used for elemental analysis of various materials [14]. A method for instrumental quality control of solid fuel has been developed, based on spectrometry of neutron-gamma radiation of inelastic scattering of fast neutrons by carbon and radiative capture of thermal neutrons by mineral components (ash-forming elements) [15]. An inversion alternating nature of the dependence of sensitivity to the heat of combustion of fuel on the layer thickness has been established. Optimized selection of boundary values of the layer thickness and instrumental signals allows achieving satisfactory accuracy of quality control of coarse-dispersed fuel.

The advantage of the method is the relatively high depth of research, which allows for non-destructive analysis of solid bulk materials in large masses with the ability to compensate for the disturbing influence of neutron-moderating and absorbing properties of the analyzed materials.

The aim of the study is to improve the existing system of standard testing of iron ores in terms of reducing labor intensity, increasing the efficiency and accuracy of obtaining representative information of the quality of iron ores in large masses. Labor intensity is reduced by eliminating a number of standard testing operations (crushing, grinding, reducing, and preparing analytical-size samples). Increased accuracy and representativeness are achieved by determining the iron concentration in partially prepared large-mass samples using the deep gamma-albedo method that provides the increased sensitivity to iron due to optimization of the scattered gamma-radiation spectrometry principle.

Due to its relative simplicity, the gamma-albedo method is widely used in geological and geophysical studies and in the mining and metallurgical industry. The intensity of scattered gamma radiation, which is the main metrological characteristic of this method, generally depends on the effective atomic number of the medium, the energy of the primary gamma radiation, and the geometric conditions of measurement. The choice of primary gamma radiation energy is made depending on the task at hand and the object of testing (powder sample, broken rock mass, natural occurrence conditions) from the point of view of ensuring maximum sensitivity to iron, maximum depth (representativeness) of the study and minimum effect of the granulometric composition.

Depending on the material and granulometric composition of the ores being sampled, stability of the relationship between the effective atomic number and the iron content, the range of its variation and the density of the ore, various methodological approaches and modifications of the gamma-albedo method are used, taking into account the features of inversion dependences and multiple scattering of polyenergetic gamma radiation, angular and linear characteristics of probe devices.

There are examples of determining C_{Fe} in prepared samples of the 3 mm class by the gamma-albedo method. The error does not exceed 0.5% with the sample mass of about 1 kg. Gamma-albedo testing of railcars using hard gamma radiation Cs-137 (661 keV) is ineffective due to its low sensitivity to iron and low representativeness of testing. Considering the dimensions of the railcar, a significant mass of the ore being sampled is outside the effective control zone. Any instrumental testing method should be considered effective if its results apply to the entire volume of the ore being monitored.

The variety of factors influencing the metrological characteristics of testing actualizes the choice of the optimal energy of primary gamma radiation in terms of the maximum sensitivity to iron and the minimum sensitivity to interference. In this case, the interference is the size of the ore, the dispersion of which causes heterogeneity.

3. Results and discussion

Studies of the gamma radiation albedo have established that the dependence of the relative sensitivity of the method to the effective atomic number S_Z on the value of Z has an inversion character [8]. Moreover, the inversion region (maximum SZ) shifts to the region of increased values of the effective atomic number with an increase in the energy of primary gamma radiation. The error due to the heterogeneity of the sampled ore (size) depends in a complex way on the particle diameter (size variation), ore composition and the depth of the method [3]. In the general case, the error due to size variation has an inversion character from the energy of primary gamma radiation for a specific particle diameter. The maximum error is observed when the depth of the method (energy of gamma radiation, Z of the ore) is commensurate with the particle size.

Uncontrolled fluctuations in particle size introduce an error into the results of the method, which can be estimated from the expression:

$$\sigma = \left[\left(\frac{S_d}{S_{Fe}}\right)^2 \cdot D\right]^{1/2} \tag{5}$$

where S_d is the method sensitivity to the size;
S_{Fe} is the method sensitivity to iron;
D is the size variance.

Thus, to achieve the minimum error due to heterogeneity (size), the optimal energy of primary gamma radiation should be selected based on the minimum S_d/S_{Fe} ratio. These recommendations predetermined the possibility of express testing of iron ores in large masses using the gamma-albedo method.

The spectrum of scattered gamma radiation depends in a complex way on many parameters: the energy of the primary gamma radiation, the density and material composition of the sample object, and the linear-angular characteristics of the probe. A spectrometric version of the gamma-albedo method is known, which consists in measuring the intensity of scattered gamma radiation in two energy ranges of the spectrum and determining the value of Z or the concentration of the heavy component (ash content in coals, iron content in ores) based on the value of the ratio of the measured intensities in the selected spectrum ranges. The method, having sufficient noise immunity to density variations, is characterized by a relatively low sensitivity to iron. How to achieve an increased sensitivity of the method to Z (iron content) and the minimal effect of the size (dispersity) of the analyzed ore?

To minimize the disturbing effect of particle size, the energy of primary gamma radiation is selected, at which the depth of the method significantly exceeds the maximum particle size. The gamma-albedo method, implemented using a Se-75 radioisotope source (~220 keV), provides a research depth of about 14 cm, which is more than 3 times greater than the maximum particle size in the prepared samples.

A relatively low sensitivity of the spectrometric modification of the gamma-albedo method is explained by the choice of non-optimal measurement parameters, at which the same qualitative nature of the intensity change in the normalized spectral regions is observed with a change in the iron concentration in the ore.

There were studied the features of the spectral distribution of the albedo of polyenergetic Se-75 gamma radiation depending on the probe parameters and the iron concentration in the ore.

From the probe device, the collimated gamma-radiation flow is directed perpendicularly to the ore surface. Scattered gamma radiation is recorded using a 40x30 mm NaJ(Tl) scintillation detector and an AI-1024 multichannel analyzer. The objects of control are partially prepared iron ore samples of the 40 mm class, weighing ~ 60 kg, placed in a measuring cuvette measuring 50x30x16 cm, ensuring the absence of edge effects. The spectral distribution of scattered gamma radiation was studied on control and reference samples with an iron content of 29.7% and 49.3% at different values of the probe length L and the distance H from the probe to the ore surface.

Figure 1 shows the instrumental spectra of scattered gamma radiation from reference samples with different iron concentrations. The spectra were obtained with the parameters L and H, providing an inversion character in the behavior of the intensities of scattered gamma radiation in different energy ranges of the spectrum. The hard peak in the spectrum is mainly caused by single scattering of gamma quanta with the energy of 265 keV. The shift in the critical energy depending on the iron concentration in the ore is caused by changing the average scattering angle of the primary gamma radiation and intense photoelectric absorption of lower-energy gamma radiation with increasing the iron concentration. In the region above the critical energy, the increased intensity of scattered gamma radiation is due to increasing the electron density.

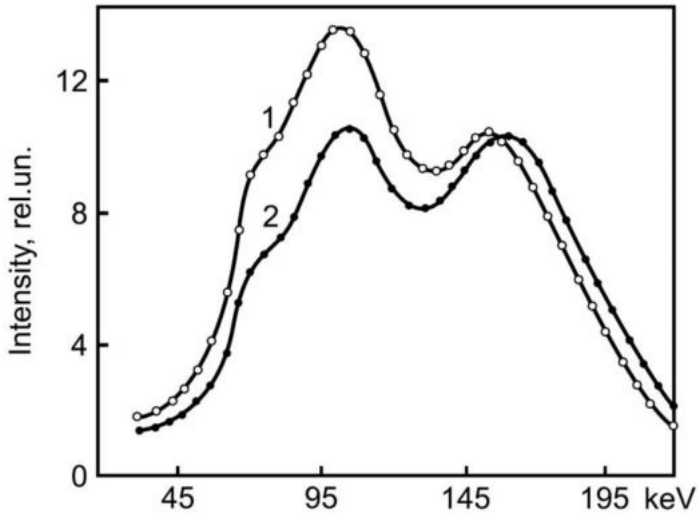

Fig.1. Spectra of gamma radiation albedo: 1 - C_{Fe} =30%; 2 - C_{Fe} = 50%

The inversion character in the behavior of the soft and hard components of the spectrum with changing the iron concentration is observed with the characteristics of the L and H probes (the average scattering angle changes), at which the critical energies, the maxima in the spectrum are in the energy intervals of (90-110) keV and (145-160) keV. This is explained by the predominant role of photoelectric absorption of gamma radiation with the energy less than ~100 keV for iron and the predominant role of Compton scattering for gamma radiation with the energy above ~150 keV.

By successive changing the parameters of the H, L probe, the energy spectra of the gamma radiation scattered from the reference samples were measured. By the machine processing of the measured spectra, the dependences of the relative sensitivity of S_{Fe} to iron on energy were constructed (Figure 2). The energy quantization step and the width of the energy window were chosen equal to 25 keV. The relative sensitivity S_{Fe} is estimated as the relative increment of the measured intensity for a single change in the iron content.

The dependence of the relative sensitivity to iron SFe on energy has an inversion alternating character. The maximum sensitivity is observed in the region of maximum intensity (critical energy). At energies above ~140 keV (depending on H) the sensitivity changes its sign. With increasing the iron content, the intensity of scattered gamma radiation increases. Moreover, with decreasing the air gap H, the region of zero sensitivity occurs at high values of the energy of scattered gamma radiation.

Based on the established patterns, the control parameters (probe parameters L and H, energy ranges of the recorded gamma radiation) were optimized, which made it possible to increase the sensitivity of the spectrometric modification of the gamma-albedo method of monitoring the iron content in ores [8].

The optimal energy intervals in the soft region of the spectrum (75-110) keV and the hard region of the spectrum (180-215) keV provide the maximum contrast of the ratio values of the intensities of scattered gamma radiation measured in the selected energy intervals of the spectrum to the change in C_{Fe}.

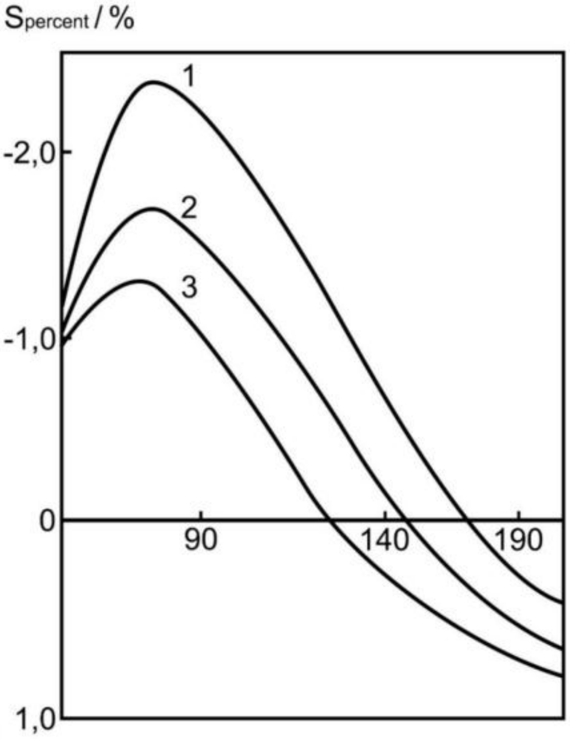

Fig.2. Sensitivity to iron dependштп on the energy of gamma radiation registered: 1 – H=2 cm; 2 – H=4 cm; 3 – H=7 cm

The sensitivity to iron of the spectrometric gamma-albedo method depending on the probe parameters L and H is shown in Figure 3. The optimal measurement parameters were selected: L=18 cm and H=2 cm. With the selected probe parameters and optimal energy intervals, the relative sensitivity of the proposed method to iron was 2.86%.

A relatively high sensitivity allowed providing the satisfactory accuracy of ore quality assessment by spectrometry of scattered gamma radiation with Se-75 source.

When testing and analyzing 18 partially prepared samples of iron ores of the 40 mm class and weighing about 60 kg in the range of iron concentration variation of 32-49%, the mean square error of iron determination of 1.32% abs. was achieved. The relative statistical error at the selected time of 7 minutes was less than 1%.

To assess reliability of the proposed instrumental testing method, additional studies of the heterogeneity of the sampled ore were carried out at the stage of sample preparation of the 40 mm class. For this purpose, the averaged partially prepared sample was divided into 4 parts by quartering. Each second part was placed in the measuring cuvette as a point sample and the method reading was measured: the ratio Ψ value of the intensities of scattered gamma radiation in the selected energy ranges of the spectrum. After each measurement, the sample was unloaded and reloaded. Multiple measurements were performed under identical conditions. The standard deviation of the measured intensity ratio Ψ can be taken as the parameter characterizing the error due to heterogeneity at a given stage of testing. The relative standard deviation of the ratio Ψ value for 20 control measurements was 4.12%, which is adequate to the standard error of 1.44%

abs. for iron. These results are in satisfactory agreement with the previously obtained error of 1.26% abs. at this stage of standard testing.

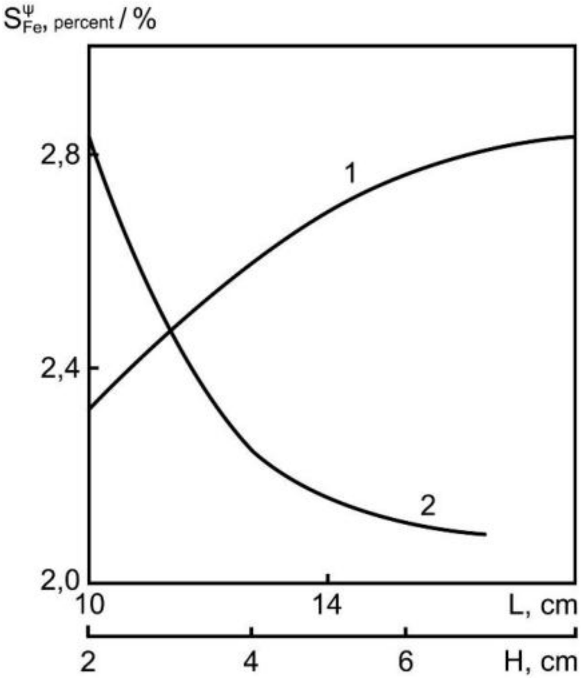

Fig. 3. - Spectrometric method sensitivity to iron dependшгп on geometrical parameters: 1 – on the probe length (H=2 cm); 2 – on H (L=18 cm)

Conclusion

A new spectrometric modification of the gamma-albedo method of monitoring the iron content in partially prepared samples weighing about 60 kg is proposed. It allows eliminating labor-intensive stages of grinding, preparation of analytical samples and direct chemical analysis. The established inversion, alternating-sign nature of sensitivity to iron from the energy of scattered gamma radiation allows optimizing the control parameters (energy intervals and probe characteristics), providing the maximum sensitivity to iron of the proposed modification of the method. In the process of laboratory testing the developed modification of the method of testing iron ores of the 40 mm class in the range of iron concentration variation of 32-49%, the mean square error in determining the iron concentration was 1.32% abs. Reliability of the obtained data on the accuracy of testing is confirmed by the results of an additional study of ore heterogeneity at the recommended stage of testing partially prepared samples. The proposed modification of the gamma-albedo method allows improving the standard testing system in terms of increasing reliability, representativeness and efficiency of the obtained information of the ore quality and reducing labor intensity by eliminating a number of testing stages.

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