

Device for X-ray spectral absorption analysis with use of acoustic monochromator

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Abstract. The method for X-ray elemental analysis and the device for its realization, based on the X-ray absorption spectroscopy technique, are proposed. Estimations of the method sensitivity have been carried out by means of numerical simulation. The simulation results indicate that the sensitivity of the proposed method for elemental analysis is not less than 10^{-4} .

Nowadays X-ray fluorescence analysis (XRF) [1] is the most common method of the X-ray elemental analysis. This technique is well known, simple and widely used for variety of applications. There are some problems, though, which are still impossible to solve by XRF method. One of them is ultrapure elemental analysis, during which radiated fluorescence is almost completely absorbed by investigated object. Variety of special methods of sampling, such as grinding or evaporation, is used to address this issue. Nevertheless these ways have some significant disadvantages: they are quite difficult to implement, time-consuming and the most important one - lead to destruction of an object.

Another solution, which has no aforementioned drawbacks, can be obtained by analyzing X-ray absorption degree at different wavelengths instead of XRF analysis for determination of elemental composition. Methods of absorption analysis are widely used in the X-ray absorption spectroscopy area and primarily in such kind of investigation as EXAFS (extended X-ray absorption fine structure) and XANES (X-ray absorption near edge structure) [2-3], but they can be used for elemental analysis. In contrast to XRF, where the information is carried by secondary radiation, in absorption analysis the information carrier is primary radiation, passing through the object and partly absorbed depending on radiation energy and elemental composition of the object. The presence of any admixture is found by the presence of intensity leap in the measured spectrum corresponding to the absorption edge of admixture element. The quantitative evaluation of the content of admixture can be obtained from the value of the leap when passing through the absorption edge. Obviously, the leap can be distinguished in a spectrum if its value is more than value of the statistical error. Thus the main limiting factor for the sensitivity of this method is intensity of the primary radiation beam and correspondingly the main task is producing the X-ray beams with satisfactory intensity.

There are two main methods of the X-ray energy spectra measurements in absorption analysis: energy dispersive and wave dispersive. In the first approach X-ray detectors with spectral resolution are used for energy dependence registration. In the second one, registration of energy spectral information is based on the use of monochromatic X-ray. Monochromatic line is isolated from continuous spectrum by means of using special devices (monochromators) before or after passing through the object and after that only this line is detected. Both of these approaches have their advantages and disadvantages. For example, energy dispersive method is easier and allows to register information across the whole energy range at the same time, without changing of the experimental



setup parameters. However, sensitivity of this method to the scattered X-ray impact and significant limit of the speed according to relative low count rate of the modern energy dispersive detectors represent the main limitations. Wave dispersive method does not have these drawbacks and allow to achieve better resolution (equal to a few eV). The main disadvantage of this approach is significant loss in X-ray energy line intensity during monochromatization due to only partial reflection of radiation energy line to the direction of the diffraction and additionally absorption in the monochromator material. The last problem can be solved, though, using “pumping” and “transparency” effects [4-5], which are observed in crystal monochromators with super lattice initialized by external influences.

The “acoustic monochromator” device based on “pumping” effect was proposed by the authors in previous works [6] to produce more intensive monochromatic X-ray beams. Acoustic monochromator is a device consisting of crystal quartz plate with excited acoustic wave field. Production of monochromatic X-ray beam with certain characteristics which can be controlled by the changing of acoustic wave field parameters has a potential to reduce dramatically the beam loss. In this work the authors propose the device for X-ray spectral absorption analysis based on the use of X-ray tube and acoustic monochromator.

Functional scheme of the device is shown in figure 1. The device for X-ray spectral absorption analysis includes: (1) X-ray source based on X-ray tubes, (2) double-slit collimator, (3) acoustic monochromator, placed in the first holder of a goniometer (4), and absorber (5). The source, collimator, monochromator and absorber are placed at the one optical axis. An object under investigation (6) and an X-ray detector (7) are positioned at the second goniometer holder behind acoustic monochromator in the diffraction direction. Acoustic monochromator is connected with electrical signal amplifier (9) which enhances amplitude of the signal from electromagnetic waves generator (8). The X-ray source, goniometer, electromagnetic waves generator and detector are connected with a computer by means of control drivers. The goniometer holders with the set acoustic monochromator, object and detector can be turned and energy of monochromatic X-ray passing through the object can be changed accordingly. At the same time detector registers spectrometric information, observation of which is the main purpose of the experiment.

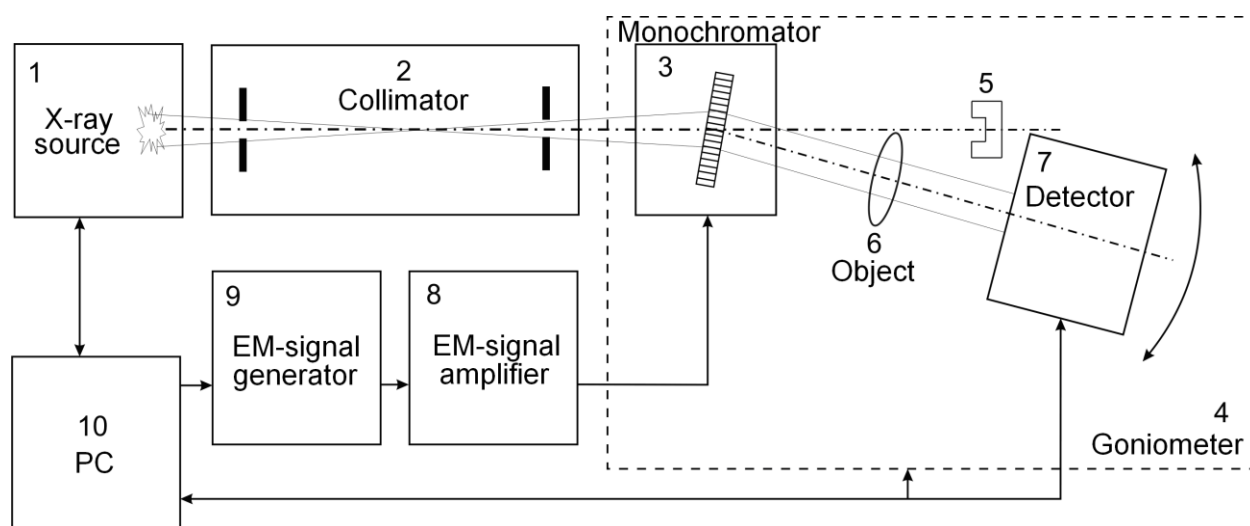


Figure1. Functional scheme of the device for X-ray spectral absorption analysis.

The aim of this work is to describe the sensitivity of the proposed method and evaluate value of admixture which can be distinguished in an experiment.

The estimations were carried out by means of the developed numerical model of the interaction between X-ray and investigated object material. Model was developed by using GEANT4 toolkit [7],

which based on application of Monte Carlo method. Samples of SiO_2 matrixes with different values of Cr admixture concentration were chosen as the objects for simulated experiment. The samples were irradiated by X-ray beam with the energy close to Cr absorption K-edge. A detector with the transverse dimensions equal to 2×2 mm was placed at the distance equal to 20 mm at the beam axis. The detector registered all passed photons with energy resolution equal to 10 eV. Simulation of the X-ray spectra passed through the samples was carried out for samples with different admixture concentrations. These spectra were compared with spectrum after pure SiO_2 sample to find the leap of intensity.

X-ray spectra passed through the sample for admixture with mass fractions equal to $5 \cdot 10^{-2}$, 10^{-2} , 10^{-3} , 10^{-4} , 10^{-5} and 10^{-6} are shown in the figure 2. Simulation was carried out for 1 keV bandwidth from 5.5 keV to 6.5 keV according to the Cr absorption K-edge energy value, which equals to 5.97 eV. Number of initial photons histories was equal to 10^9 for all energy ranges or 10^7 per 10 eV channel. Figure 2a includes spectra for admixture mass fractions not less than 10^{-4} for all considered energy range while figure 2b - for shorter one for mass fractions not more than 10^{-3} . The minimal admixture concentration, which could be distinguished for that number of histories, equalled to 10^{-3} as can be seen from simulated spectra. Spectra for lower concentrations were almost equal to the X-ray spectrum passed through the pure SiO_2 sample.

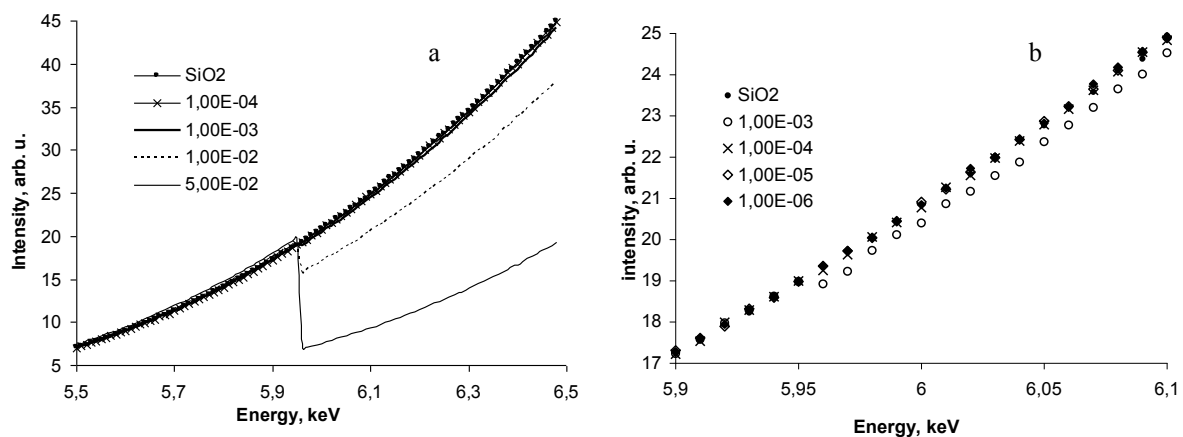


Figure 2. X-ray intensity distributions in the experiment, after passing through the samples with different admixture concentration.

It should be noted that “differential” spectra are more suitable for this kind of experiments. These spectra represent differences between intensities of X-rays passed through the pure sample and admixture contained one. Such spectra for mass fraction equal to 10^{-3} and 10^{-4} are shown in figure 3a. It is clearly seen from these spectra, that concentration 10^{-3} is distinguishable (a specific “leap” is present in the spectrum at the absorption edge), while concentration 10^{-4} is not seen (a spectrum only contains “noises”, caused by a statistical error). The last proposition, though, is right for announced histories number. Rise of the initial X-ray intensity which passes through the samples can allow to distinguish lower concentrations.

The parameters of the model were slightly modified to distinguish 10^{-4} concentration in the experiment. At first, total number of particle histories was increased up to 10^{10} . Besides, simulation was carried out in the immediate vicinity of the absorption edge and width of simulated region was equal to 90 eV. All of that allows to increase particle histories per channel number up to 10^9 keeping the acceptable amount of time spending for simulation. Differential spectrum for admixture mass fraction equals to 10^{-4} in this simulation is shown in figure 3b. Well distinguished step at the absorption edge region is clearly visible and consequently for this value of the initial X-ray intensity the concentration equal to 10^{-4} can be successfully found in the sample. Thus, the proposed method of

elemental analysis provides sensitivity not less than 10^{-4} . In the same time, it should be noted that further rising of the initial X-ray beam intensity might allow to find much lower concentration.

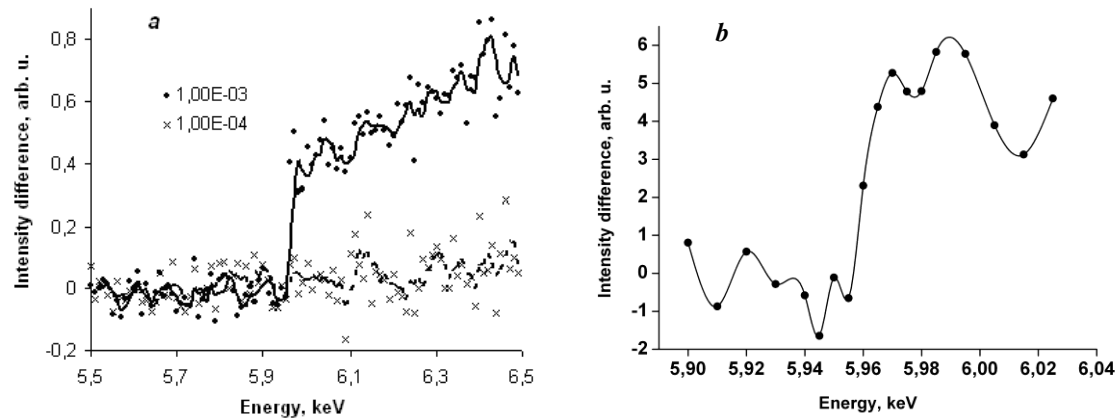


Figure 3. Differential spectra for SiO_2 sample contained Cr admixture. Number of the initial particles histories is equal to 10^7 (a) and 10^9 (b) per channel. Mass fraction is 10^{-3} and 10^{-4} (a), 10^{-4} (b).

With regard to the potential application of the presented device besides laboratory investigations environmental control can be an attractive area. For example, this device can be used for the analysis of water composition. The issue of the presence of harmful admixtures in sewage water or drinking water is very important now. To check the applicability of the proposed device for the control of water compositions simulations of radiation spectra passing through the sample consisting of a water layer containing different admixtures and through a layer of pure water were carried out. Elements from different areas of the periodic table which are the most common admixtures for natural water were chosen as admixtures. These elements were Ca for the lights, Fe for the mediums and Pb for the heavies. All of these elements had mass fractions equal to 10^{-4} . The region near to absorption K-edge was chosen for simulating for Ca and Fe admixtures while L-edge region was observed for Pb. Differential spectra are shown in figure 4 for all these cases. Presence of these admixtures with 10^{-4} mass fraction is well distinguished by proposed method as it can be seen from the figures.

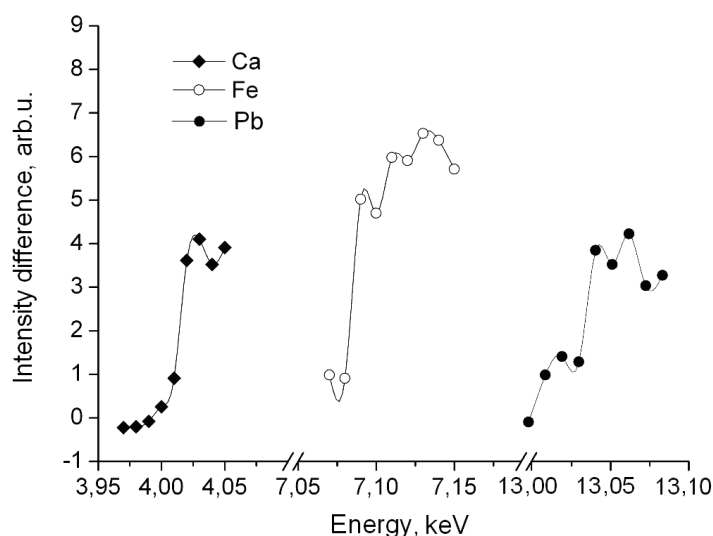


Figure 4. Differential spectra for water sample with Ca, Fe and Pb admixtures. Initial particles number is equal to 10^9 per channel.

Thus, the method for elemental analysis by different wavelength X-ray absorption in the object is proposed as well as device for X-ray spectral absorption analysis, which allows to realize this method. Developed numerical model of X-ray interaction with the object allows to estimate the proposed method sensitivities. The sensitivity is not less than 10^{-4} by Cr admixture in SiO_2 sample according to simulation results. Use of the device allows to analyze pure and ultrapure samples.

The estimations, carried out with the developed model for water samples consisting of a water layer, containing different admixtures, show retention of the method sensitivity at the same level for the wide range of admixtures – from Ca to Pb. This fact allows to conclude, that the proposed method can be applied in the analysis of air composition in ecological monitoring.

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