Title of doctoral thesis: Research on the development of new certified reference materials for selected products of the silicon industry

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Abstract:

The aim of the implementation doctorate was to develop, produce and implement three certified reference materials (CRM): silica fume, silicon and magnesium ferrosilicon alloy (FSM). This dissertation describes the successive actions taken to achieve this goal. The theoretical part of the work reviews the literature on analytical methods used to determine the elemental composition of silicon materials and the market for silicon standards. The experimental part describes the successive stages of developing new CRMs. This included the preparation of candidate materials for CRMs by mixing, homogenizing and separating them into final units. The work also developed analytical procedures for preliminary, stability, homogeneity and characterization tests of materials intended for CRMs. Various analytical techniques were used, such as: ICP-OES, XRF and elemental analysis with infrared detection. At the same time, various methods of preparing silicon material samples were investigated: acid digestion in an open system, microwave digestion, fusion to form borate beads, and pressing to form pellets. The determined validation parameters confirmed the suitability of the developed methods for use in individual processes and for use in commercial analyses. Further studies allowed to determine the short- and long-term stability of all three materials, and the statistical evaluation of the results confirmed the negligible uncertainty resulting from these parameters. Homogeneity tests were also carried out, combined with the statistical evaluation of the results and the determination of the uncertainty resulting from the inhomogeneity of the materials, which met the expected criteria and confirmed the effectiveness of the homogenization operations. The next process - characterization - was based on the results obtained by a network of competent laboratories, using various analytical techniques. This research strategy led to the determination of the final certified values and the accompanying expanded uncertainties. As part of the activities aimed at supporting the XRF analysis of silicon materials, the possibility of using an internal standard addition for grain size corrections in the analysis of pellets was tested. A positive effect was observed on the precision of the calibration curves for silica fume and for the FSM alloy. For the FSM material, the possibility of using sample preparation in the form of a thin layer for the determination of Fe, Ca, Mg, Al and Mn was also tested. The spectrometer was calibrated based on synthetic standards, which is a great advantage in the absence of commercially available solid standards. Pyrometallurgical tests of calibration materials for silicon were also carried out, the results of which were unsatisfactory, but the methodology itself shows potential and will be developed in the coming years. The implementation aspect of the doctoral thesis included activities related to the introduction of new standards into the scope of ISO 17034 accreditation, promotional activities, among others in the international distribution network and development of the necessary documentation for the sale of new CRMs.

The work within the implementation doctorate was completed by implementing the developed CRMs into the commercial offer of Łukasiewicz-IMN for the international market and implementing the developed analytical procedures to provide silicon material analysis services on commercial rules.