The process of production certified reference materials for silicon industry

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Silicon is the element of significant importance – including the production of electronic devices, solar panels and metallurgical alloys. Silicon materials producers must keep strict quality control of their raw materials and products. Modern and innovative silicon production line requires high quality analytical information about chemical composition – for this purpose instrumental methods are commonly used. Correctness of the results obtained with these methods must be verified using materials of well-know composition, traceable to SI units, confirmed by the certificate. These materials are called certified reference materials (CRM).

In response to the market needs, ELKEM (Norway) – the world's leading silicon producer, together with ŁUKASIEWICZ – Institute of Non-Ferrous Metals (Poland) - an experienced CRMs producer, started the SILREF project, which consist of production of 8 CRMs for silicon material: metal silicon, ferrosilicon and microsilica.

Development of new CRMs consist of several stages: production of material with planned composition, homogenization via grinding, sieving, and mixing, homogeneity and stability testing, characterisation, and certification. Sampling is part of almost every one of these steps. The choice of the right sampling scheme has a significant impact on the final quality of the produced CRMs. Homogeneity of powder type CRMs and its estimated uncertainty makes important contribution to the total uncertainty of the reference values.

After homogenization and dividing the material into the 100 mL jars, 200 to 700 single units were obtained - depending on the material. In this case it is important to determine the homogeneity between the units and inside them. This was performed in accordance to the rules of ISO Guide 35:2017 "Reference materials — Guidance for characterization and assessment of homogeneity and stability". 10 random single units from every material was selected. Then, three samples from different place were taken from each unit. All parameters planned for certification (element concentrations, loss of ignition etc.) require analysis for homogeneity, stability and quantitative determination. The obtained data were used for the statistical evaluation of homogeneity based on the ANOVA test. The determined homogeneity was taken then for further calculations of total uncertainty.

The determination of homogeneity, stability and characteristics is accompanied by a large number of analytical test and statistical calculations that lead to determination of the final reference's composition values with their expanded uncertainty.

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