Chemical Analysis of Potassium Chloride by Double Pulse Laser Induced Breakdown Spectroscopy using LEA-S500 Elemental Analyzer

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This paper reports the development of method of quantitative analysis of potassium chloride, obtained by floatation or halurgy methods, using a commercialized laser-induced breakdown spectroscopy instrument, model LEA-S500 (JV" SOLAR TII") in the course of control of technological process.

Determination of chemical composition of potassium chloride samples, obtained by halurgy method.

On the first stage, calibration of the instrument has been performed according to samples, prepared from chemically pure reagents. These samples have been prepared by mixing the materials, then grinding them, using the Fritch Pulverisette7 mill (4 g of mixture; 10 min) with following pressing in tablets, using 8-ton effort laboratory press. The calibration ranges of the instrument in mass fraction, % are as follow: KCl – 94-99.1; NaCl – 0.97-5.31; $CaCl_2.6H_2O$ – 0.074-0.79; $MgCl_2.6H_2O$ – 0.077-0.69.

11 samples with the composition that varies within the calibration range have been selected after the preliminary analysis of dozens of the probes. 11 samples have been certified on four of the above indicated compounds with high accuracy by multiple measurements. On the second stage, calibration of the instrument has been performed according to eleven selected samples without grinding, but with pressing in tablets using 8-ton effort laboratory press. Calibrations have been achieved with linear regression coefficients, on the first stage: 0.96-0.99, on the second stage: 0.965 – 0.995. Preparation of samples to be analyzed has been carried on in the same way as calibration samples preparation. Time of analysis is 7 min (sample preparation included). Composition determination accuracy of KCl is ± 0.12 % (at acceptable probability P=0.95).

Determination of chemical composition of potassium chloride samples, obtained by floating method.

On the first stage, calibration of the instrument has been performed according to samples, prepared from chemically pure reagents. Samples have been prepared by mixing materials and grinding them using Fritch Pulverisette7 mill (4g of mixture;10-20min) with following pressing in tablets using 8-tons effort laboratory press. The calibration ranges of the instrument in mass fraction ,% are as follow: KCl – 94-99.1; NaCl – 0.73-4.55; $CaSO_4$ – 0.080-1.00; $MgCl_2$ – 0.025-0.17; Fe_2O_3 – 0.054-0.34; Al_2O_3 – 0.017-0.18; SiO_2 – 0.032-0.46. After preliminary analysis of dozens of probes 12 samples with composition, varying within the calibration range, have been selected. The selected 12 samples have been certified on seven compounds with high accuracy. On the second stage, calibration of the instrument has been performed on 12 samples in similar way of grinding and pressing. Calibrations were achieved with linear regression coefficients 0.96 - 0.99 on the first and second stages. Preparation of samples to be analyzed has been carried on in the similar way as preparation of calibration samples. Time of analysis is 15 min (sample preparation included). Composition determination accuracy of KCl is ± 0.14 % (at acceptable probability P=0.95).