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IMPLEMENTATION OF QUALITY CONTROL ON SAMPLED SOIL WITH TRIP AND FIELD BLANK

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Abstract

The quality control of the sampled soil is monitored by Trip and Field Blanks according to the ISO 18400-106:2017 standard. Except these blanks, before terrain sampling, it is necessary to form so called Zero Blank, which is analyzed element by element, and on which soil samples are to be analyzed. Returning from the terrain, Trip and Field Blanks are to be analyzed as well. Soil sampling was conducted on the following places: Kostolac, Prahovo and Coka Korugu. After every sampling on this place the quality control was implemented by Field and Trip Blank, as well as by duplicated control samples. Soil sampling on this terrain was carried out every three months. Samples were tested on Cu (ppm) and presented on R-control maps, that also implicate on the absence of the cross contamination in the process of sampling, transport, preparation of the chemical analysis. All this is confirmed by the analysis results which are below the detection limit and alert limit.

Keywords: soil sampling, quality control, Field and Trip blanks, R- control maps

INTRODUCTION

Soil is a complex heterogeneous matrix composed of mineral and organic solid matter, liquid and gaseous components and living organisms. It presents the important part of the urban ecosystem which directly or indirectly has impact on life quality and people's health. Industrial and urban development of human society among many advantages and benefits, results in environmental pollution and soil pollution as well. So, it is of the great importance to do the soil control and analysis more frequently.

Sampling is the beginning and the basis, on which depends the accuracy of the output results of chemical analysis of sampled soil. Mining and Metallurgy Institute has established procedures and standards for quality control.

For the blank forming can be taken any kind of soil, only thing to be known is the zero chemical soil composition so called zero blank. After soil sampling, the chemical composition of trip and field blanks are compared with the chemical composition of the zero blank.

If there are no significant deviations between the blanks and the zero blank, it can be concluded that no cross contamination occurred during the sampling and transport, which guarantees the accuracy of soil sampled chemical analysis [1].

SAMPLING QUALITY CONTROL

Sampling quality control has 3 main goals: Monitoring and detecting errors which may occur in the process of sampling, sampling error control and to present the sampling variability [2].

Precondition for accuracy and reproducibility of analytical results is sampling quality control that includes the following:

- Representativeness of the sampled material;
- Prevention of contamination during the terrain sampling, transport to the laboratory, storage and preservation of samples and preparation for chemical analysis.

Inadequate sampling can lead to the unexpected effects, for example, to the change of chemical composition of samples, which can bring into question the accuracy of chemical analysis inputs. In order to prevent the unwanted effects, in the Mining and Metallurgy Institute laboratory, the quality control is done by:

- Using field and trip blanks;
- Taking double control samples by each sampling in order to check sampling precision.

They provide information which ideally rejects all mistakes of possible sources of contamination, sampling inconsistency and check of applied analytical technics.

ZERO FIELDS AND TRIP BLANKS

For blank forming can be used soil of different chemical and physical characteristics. It is recommended to use unpolluted areas. Parameters on which zero, trip and field blanks are analyzed must be known before the coming on the terrain, they must be the same as the parameters where the sampled soil is to be analyzed [2–4].

Trip blanks are used to detect prospective mutual contamination of blanks during the transport and are also used to confirm or eliminate possible cross contaminations of samples. The sample must go through the entire process, from transport to the laboratory, preparation on the ring mill and chemical analysis.

Trip blank is analyzed on chemical elements, on which the soil samples will be analyzed, before going to the terrain. The sample must go through the entire process, from the field sampling, transport to the laboratory, preparation on the ring mill and chemical analysis. This way confirms or eliminates possible inconsistency in entire process, considering transport than preparation and chemical analysis at the end.

Comparative analysis of field and trip blanks with zero blanks, determines possible contamination of sampled soil, proper sampling method with previously established sampling plan or excludes the possibility for negative outputs.

CHEMICAL ANALYSIS AND SAMPLE PREPARATION

All samples including, control samples, field and trip blanks are transported to the laboratory for preparation and are stored according to the ISO18400 Soil quality-sampling-part105: Packaging, transport, storage and preservation of samples standard [4,5].

Preparation includes the following steps: sample drying at 40°C, or on the air, exemption of representative samples for particle size analysis by aerometric, exemption of representative samples for chemical analysis and grinding the sample in the ring mill on the particle of 85-90% -75 μ. The prepared soil samples, in ring mill, are dissolved in the automatized system for digestion "Vulcan-Questron Technologies", with the addition of 3 different acids (HCl, HNO_3 and $HClO_4$) and the process of digestion is done in Teflon container on the temperature of 200°C.

Measured soil amount is 0.25 g and after the dissolving, the samples are moved to the normal container and the final volume of the dissolved sample was 25 mL. The prepared samples are further tested for copper content by ICP MS method.

An Agilent SPS 4 autosampler was used to deliver the samples. Standard Ni-cones were used. The operating conditions are shown below.

Multi-element standard solution for ICP MS, concentration 10 μg/ml, Calibration Standard#2, Ultra Scientific (Al, As, Ba, Be, Bi, Cd, Ca, Ce, Cr, Co, Cu, Ga, In, Fe, Pb, Li, Mg, Mn, Ni, K, Rb, Se, Na, Sr, Tl, U, V, Zn) was used to prepare the external calibration curve (from 1 to 200 μg/L). All solutions were prepared using Milli-Q® water (18 $M\Omega \cdot cm^{-1}$). To prevent contamination, all glassware and plasticware were acid-washed before use.

All the blanks undergo the same way of preparation, and are analyzed with all samples on the same element after coming from terrain. Exceptions are zero blanks whose composition must be known before going on the terrain. For each location are formed zero, trip and field blanks.

RESULT AND DISCUSION

Soil sampling is conducted according to the criteria which is applied in the Republic of Serbia, and are in accordance with standard norms [5–8].

In Tables 1, 2 and 3 are presented rezults of analyzed blanks for the given sampling periods. Results of analyzed control samples are presented in Table 4 and Table 5 presents limits of R-chart, on the basic calculated values of relative range.

According to the values of relative range, the control limits of R-chart are calculated for control samples, central lines, warning limits, detection limits, and presented on R-charts. From R-charts can be clearly seen that there is no outliery-the values that bounced off from the calculated limits [1,8].

Looking at the results of chemical analysis on copper for indicated period we can come to possitive constatation , according to the cross contamination during the transport, preparation and chemical analysis at the end, which leads to the conclusion that the sample contamination during the entire process is excluded and any inconsistency in the entire process from sampling to the the preparation and chemical analysis did not occur.

By comparative analyse of the sample, trip and fild blanks, zero blank we come to the minimal deviations, which can be regarded as negligible.

Table 1 Comparative chemical analysis zero blank with field and transport blank, Cu content (ppm) - mean of two measurements, sampling site of Ćoka Korugu

Month of sampling / Sample name	January	March	Jun	September	December
Zero blank	34.0	33.9	33.6	33.6	34.0
Trip blank	34.4	34.3	34.5	34.7	34.4
Field blank	34.1	34.2	34.1	34.3	34.1

Table 2 Comparative chemical analysis zero blank with field and transport blank, Cu content (ppm) - mean of two measurements, sampling site of Kostolac

Month of sampling / Sample name	January	March	Jun	September	December
Zero blank	44.7	44.6	44.7	45.0	44.9
Trip blank	45.0	45.1	44.2	44.7	44.0
Field blank	44.4	45.0	44.0	45.4	45.5

Table 3 Comparative chemical analysis zero blank with field and transport blank, Cu content (ppm) - mean of two measurements, sampling site of Prahovo

Month of sampling / Sample of name	January	March	Jun	September	December
Zero blank	51.5	51.8	51.8	51.0	51.6
Trip blank	49.9	51.2	49.6	50.0	50.5
Field blank	50.0	52.2	49.9	50.5	51.5

Table 4 Chemical analysis of control samples on Cu content (ppm), sampling sites of Ćoka Korugu Kostolac, Prahovo

Month of sampling / Sample of name	January	March	Jun	September	December
KK1ĆK	52.9	53.2	54.0	53.4	53.5
KK2ĆK	51.7	52.2	51.8	53.0	51.3
KK1K	64.3	65.2	63.6	65.1	64.5
KK2K	64.7	65.3	64.1	66.5	65.7
KK1P	70.0	71,1	68,9	71.3	73.5
KK2P	71.6	73.3	70.0	69.2	73.8

Table 5 Results of R-chart , CL- center line, CL, WL alert limit, and AL detection limits, ppm

Month of sampling	January			March			Jun			September			December		
Sample of name	CL	WL	AL	CL	WL	AL	CL	WL	AL	CL	WL	AL	CL	WL	AL
KK1ČK	0.22	0.552535	0.718901	0.22	0.552535	0.718901	0.22	0.552535	0.718901	0.22	0.552535	0.718901	0.22	0.552535	0.718901
KK2ČK	0.18	0.452074	0.588191	0.18	0.452074	0.588191	0.18	0.452074	0.588191	0.18	0.452074	0.588191	0.18	0.452074	0.588191
KK1K	0.24	0.602766	0.784255	0.24	0.602766	0.784255	0.24	0.602766	0.784255	0.24	0.602766	0.784255	0.24	0.602766	0.784255
KK2K	0.22	0.552535	0.718901	0.22	0.552535	0.718901	0.22	0.552535	0.718901	0.22	0.552535	0.718901	0.22	0.552535	0.718901
KK1P	0.21	0.505625	0.662143	0.21	0.505625	0.662143	0.21	0.505625	0.662143	0.21	0.505625	0.662143	0.21	0.505625	0.662143
KK2P	0.3	0.753457	0.980319	0.3	0.753457	0.980319	0.3	0.753457	0.980319	0.3	0.753457	0.980319	0.3	0.753457	0.980319

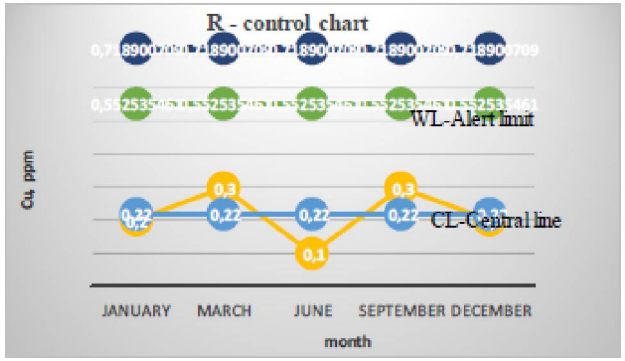

Figure 1 R chart of control sample KK1ČK

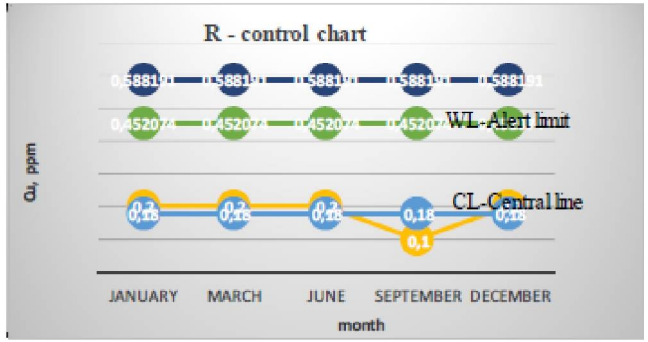

Figure 2 R chart of control sample KK2ČK

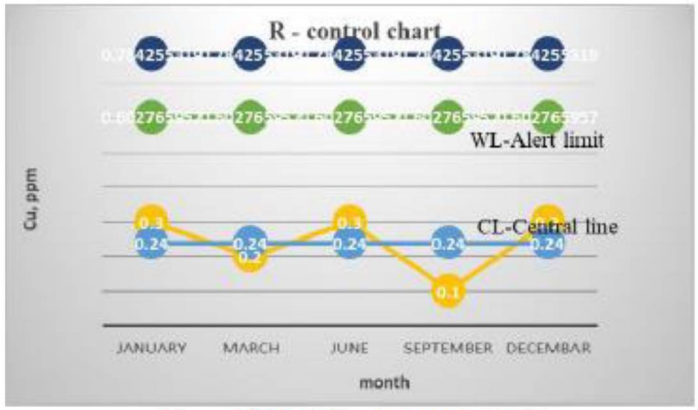

Figure 3 R chart of control sample KK1K

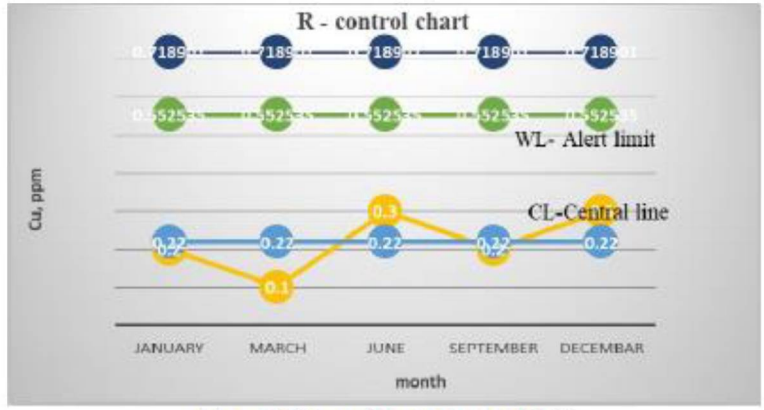

Figure 4 R chart of control sample KK2K

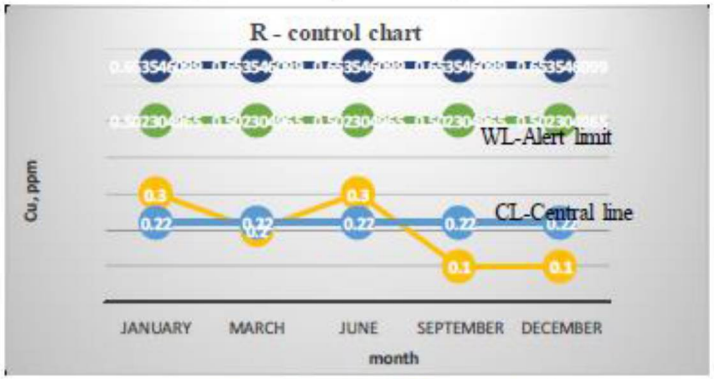

Figure 5 R chart of control sample KK1P

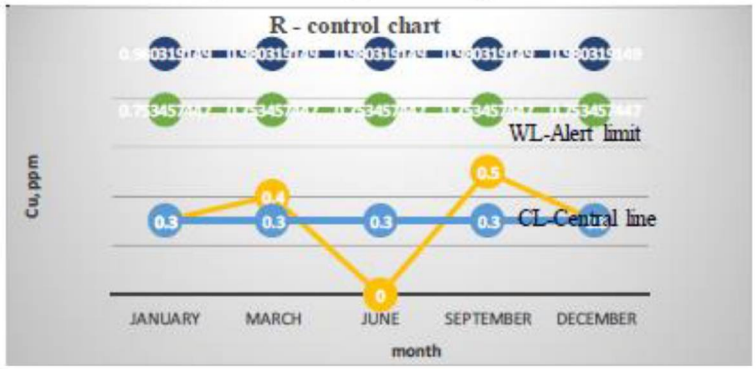

Figure 6 R chart of control sample KK2P

CONCLUSION

On the locations Coka Korugu, Kostolac i Prahovo, the soil sampling was done every 3 months according to the standards norm. Results of the analyzed parameter, copper, on the sampled soil are not part of this work, for justified reasons, except the copper content in blanks and control samples. The sampling and sampler control was done in laboratory in two ways:

- with field and transport blanks;

- and with taking control samples in duplicate.

Results of chemical analysis of control samples are presented on R-maps. R-control map is made of the range of two measurements and of the difference in results of two measurements. The value of the arithmetical range is used as a central line (CL). Standard deviation is used for calculation of control limits where the Al is limit of action and WL is warning limit.

Results from the R-control map present that all the results of analyzed parameters on blanks and control samples, go into range of measuring and are acceptable, and can be used for further statistic data, as well as the quality of sampling and measurements in specific period of time.

The double samples are used for monitoring the sampling quality and precision of analytical laboratory processes. Field and trip blanks illustrate the effects of managing the samples, they reveal the contaminations such as dust and atmospheric precipitation.

Examining and analyzing result from the Tables 1, 2 and 3 we can conclude that the deviation values of field and trip blanks are minor in comparison to the zero blank. The entire process of sampling, transport, to the preparation and analyze of the samples is conducted in adequate and accurate way relying on previously mentioned standards, relevant legislation and examining methods.

The conclusion is that the adequately conducted quality control on soil sampling on the terrain results in high accuracy of chemical analysis outputs. R-control charts of field, transport and zero blanks, because of the limited number of pages, were not presented in this work [8,9].

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