IMPLEMENTATION OF QUALITY CONTROL ON SOIL SAMPLING

Zorica Sovrlić[1]
Daniela Urošević[2]
Ivan Svrkota[3]

DOI: https://doi.org/10.31410/ITEMA.2019.67

Abstract: *Adequate soil sampling with the implementation of quality control guaranties high accuracy outputs of chemical analysis of the sampled soil. Mining and Metallurgy Institute Bor, as accredited scientific institution, sampling the soil from various locations according to the previously established sampling plan and to the standards ISO 18400-101 and 180400-104. The standards define the sampling scheme. Control of compulsory monitoring of sampling quality is done by ISO 18400-106 standard. Monitoring the quality control of the sampled soil, means formation of Trip blanks and Field blanks. Developed quality control plan monitors the status of the mentioned blanks, which considers the initial chemical composition of the zero blank, its way of packing and transport to the place where sampling occurs, preservation and storage as well as chemical analysis on specific elements. During 2019, Mining and Metallurgy Institute conducted quality control after every sampling on 6 locations. Comparative chemical analysis of the blanks after the sampling, started with zero blank, resulted in non-existence of cross contamination of the sampled soil. Conclusion is that the sampling was implemented according to the given procedures and standards.*

Keywords: *Quality Control, Soil Sampling, Blank Samples.*

1. INTRODUCTION

Soil is a complex heterogeneous matrix composed of mineral and organic solid matter, liquid and gaseous components and living organisms. (Alloway,1995.) 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. Soil samples for the analysis can be taken in disturbed or in undisturbed condition, depending which parameters are to be examined. Soil sampling is performed according to the criteria applicable in the Republic of Serbia, and to the following standards:

- ISO 18400 Soil quality- Sampling/part 101; Frame work for the preparation and application of the sampling plan, 2007.
- ISO 18400 Soil quality-sampling- part 102; selection and application of sampling techniques, 2017.
- ISO 18400 soil quality- sampling –part 104; strategies, 2018.Ways of taking disturbed and undisturbed field samples are defined in ISO 18400-104 standard:
- Systematic unaligned sampling pattern
- Circular grid
- Stratified random sampling

[1] Mining and Metallurgy Institute Bor, Zeleni bulevar 35, 19210 Bor, Serbia
[2] Mining and Metallurgy Institute Bor, Zeleni bulevar 35, 19210 Bor, Serbia
[3] Mining and Metallurgy Institute Bor, Zeleni bulevar 35, 19210 Bor, Serbia

Transport, storage, preservation and preparation of samples for the analysis are defined by standards ISO18400 Soil Quality-Sampling-Part105; Packaging, transport, storage and preservation of samples.

2. 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.

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

- Representativeness of the sampled material
- Prevention of contamination and unwanted changes or changes made on the samples during terrain sampling, pre-treatment of the samples if it is necessary, transport, storage and preservation of samples
- Adjustment to all protective measures on the field

Laboratory preparation for sampling quality control occurs by using field and trip blanks, gathering control samples of every field sampling (double samples) in order to check the sampling precision.

3. TRIP AND FIELD BLANKS

Trip blanks are used to detect prospective mutual contamination of blanks during the transport. Sample container or any other agent for sample storage, identical to the one used for samples, must be filled with soil or any other material that resembles the soil. The parameters that are to be analyzed must be presented before the container fills. Container or the agent for sample storage closes and together with samples is transported back to laboratory. Blank is than at the same time analyzed with gathered samples.

Trip blank is analyzed on chemical elements on which the soil samples will be analyzed, before going to the terrain, and packaging in container for preserving blanks and after the transport to the laboratory. This way confirms or eliminates the possible cross contamination of samples during the transport.

Field blanks are similar to trip blanks but are used in order to reveal inconsistency in entire process- from the sample taking to the laboratory analysis. 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.

4. CONTROL SAMPLES

As it is already mentioned, in order to implement the quality control, the double samples are taken on the terrain to show the quality of sampling on previously defined points for sampling. They provide information that ideally rejects all errors of possible contamination sources, inconsistencies in sampling and check the applied analytical techniques.

In order to gain insight in sampling quality, r-control maps are established (map of duplicate samples). In r-control map are input the controlled values, differences of the compatible results of the repeated analysis of control samples.

5. RESULTS AND DISCUSION

The following text will present the results of the analyzed chemical parameters of the zero blank, trip and field blank, control samples, processed in R-control maps. Sampling location, analysis of the double soil samples on previously defined points of sampling, for the period from April to May will not be presented in this project. Location on which the soil monitoring occurs–once per month, is presented as Location 1, and is situated in Eastern Serbia. According to the previously established sampling plan, all the necessary requests for sampling are defined, on the mentioned location, figure 1 and figure 2.

Figure 1.
Site Sampling at Site 1

Figure 2.
Sampling quality control, field blank

Table 1 shows the results of analyzed chemical parameters of zero, field and trip blank. (month April). Results of the analyzed control samples (April -November) are presented in table 2. Table 3 presents the relative range and average value of measuring. Table 4 presents control limits of all R–charts (control maps). Chemical analysis of examined parameter, Tables 1 and 2. on control soil samples, zero, trip and field blank, was done in duplicate. The difference of the obtained values is put in R –chart for each examined parameter and presented on charts 1-8. According to the duplicate differences of the analyzed parameters(R- range), the control limits of R- charts are calculated central line, warning limit and the limit of action are also presented in R- charts , it can be clearly seen that there is no outlier -the values that jump out from the calculated limits. It is of the great importance to mention that the blank analysis is done after every sampling and compared with zero blank.

From Tables of analyzed blank, Table 1, on the same chemical elements as well as on the control samples, we can conclude that the contamination of samples during the transport is excluded and no inconsistency in the entire process from sampling to chemical analysis. By comparative analysis of the examined element, trip and field blanks, marked as 5Te-4 and 6Te-4, with zero blank, mostly comes to concurrence of analyzed parameters which are done before they went on the terrain and after they came back to laboratory.

Table 1.
Comparative chemical analysis of zero-blank from field and transport blank

Sample name	Element, ppm					
	Cu	Be	Zn	Co	Pb	V
0-Blank	33.3	1.3	82.3	15.5	20.6	101.6
	33.6	1.2	82.5	15.3	20.5	101.1
5Te-4	34.4	1.3	83.6	15.6	20.6	102.1
	34.3	1.2	83.8	15.3	20.6	101.9
6Tr-4	34.1	1.3	82.9	15.5	20.5	101.2
	34.7	1.3	83.0	15.4	20.6	101.2

Table 2.
Chemical analysis of control samples

Sample name	Element, ppm					
	Cu	Be	Zn	Co	Pb	V
1K-1	53.0	1.2	74.6	10.6	23.0	109.8
1K-2	52.9	1.1	74.3	10.4	23.5	109.6
2K-1	54.5	1.2	72.3	13.8	22.7	105.6
2K-2	54.4	1.1	72.4	13.5	22.8	105.2
3K-1	76.3	1.2	89.6	12.9	26.5	125.3
3K-2	76.4	1.3	88.9	12.9	26.2	124.9
4K-1	67.4	1.1	73.8	14.2	26.6	113.3
4K-2	67.8	1.2	74.5	14.1	26.7	113.8
5K-1	117.2	1.1	79.3	21.2	19.9	116.8
5K-2	117.4	1.2	78.4	21.6	20.0	115.9
6K-1	123.5	0.98	80.5	20.6	21.6	223.8
6K-2	124.0	0.97	80.4	20.5	21.8	222.6
7K-1	142.6	1.0	87.2	18.6	19.9	142.2
7K-2	142.1	0.97	87.4	18.5	21.2	141.1
8K-1	144.1	1.2	78.6	13.1	22.5	135.7
8K-2	144.2	1.2	77.9	12.9	22.7	135.2

Table 3.
Relative range and mean of measurement, ppm

Sample name	Cu		Be		Zn		Co		Pb		V	
	R	Sr.vr.	R	Sr.vr.	R	Sr.vr.	R	Sr.vr.	R	Sr.vr.	R	Sr.vr.
0-Blank	0.3	33.45	0.1	1.25	0.2	82.4	0.2	15.4	0.1	20.55	0.5	101.35
5Te-4	0.1	34.35	0.1	1.25	0.2	83.7	0.3	15.45	0.0	20.60	0.2	102.0
6Tr-4	0.6	34.40	0.0	1.30	0.1	82.95	0.1	15.45	0.1	20.55	0.0	101.2
1K	0.1	52.95	0.1	1.15	0.3	74.45	0.2	10.5	0.5	23.25	0.2	109.7
2K	0.1	54.45	0.1	1.15	0.1	72.35	0.3	13.65	0.1	22.75	0.4	105.4
3K	0.1	76.35	0.1	1.25	0.7	89.25	0	12.9	0.3	26.35	0.4	125.1
4K	0.4	67.6	0.1	1.15	0.7	74.15	0.1	14.15	0.1	26.65	0.5	113.55
5K	0.2	117.3	0.1	1.15	0.9	78.85	0.4	21.4	0.1	19.95	0.9	116.35
6K	0.5	123.75	0.01	0.975	0.1	80.45	0.1	20.55	0.2	21.7	1.2	223.2
7K	0.5	142.35	0.03	0.985	0.2	87.3	0.1	18.55	1.3	20.55	1.1	141.65
8K	0.1	144.15	0	1.2	0.7	78.25	0.2	13	0.2	22.6	0.5	135.45

Table 4.
Results of R-Limits of control chart: CL central line, WL alert limit, and AL detection limits

Sample name	Cu			Be			Zn		
	CL	WL	AL	CL	WL	AL	CL	WL	AL
0-Blank	0.272	0.684	0.8911	0.067	0.1689	0.2198	0.381	0.958	1.2476
5Te-4	0.272	0.684	0.8911	0.067	0.1689	0.2198	0.381	0.958	1.2476
6Tr-4	0.272	0.684	0.8911	0.067	0.1689	0.2198	0.381	0.958	1.2476
1K	0.25	0.627	0.816	0.06	0.169	0.220	0.46	1.161	1.511
2K	0.25	0.627	0.816	0.06	0.169	0.220	0.46	1.161	1.511
3K	0.25	0.627	0.816	0.06	0.169	0.220	0.46	1.161	1.511
4K	0.25	0.627	0.816	0.06	0.169	0.220	0.46	1.161	1.511
5K	0.25	0.627	0.816	0.06	0.169	0.220	0.46	1.161	1.511
6K	0.25	0.627	0.816	0.06	0.169	0.220	0.46	1.161	1.511
7K	0.25	0.627	0.816	0.06	0.169	0.220	0.46	1.161	1.511
8K	0.25	0.627	0.816	0.06	0.169	0.220	0.46	1.161	1.511
	Co			Pb			V		
	CL	WL	AL	CL	WL	AL	CL	WL	AL
0-Blank	0.1818	0.456	0.5941	0.272	0.6849	0.891	0.5363	1.347	1.7526
5Te-4	0.1818	0.456	0.5941	0.272	0.6849	0.891	0.5363	1.347	1.7526
6Tr-4	0.1818	0.456	0.5941	0.272	0.6849	0.891	0.5363	1.347	1.7526
1K	0.175	0.439	0.571	0.35	0.879	1.143	0.65	1.632	2.124
2K	0.175	0.439	0.571	0.35	0.879	1.143	0.65	1.632	2.124
3K	0.175	0.439	0.571	0.35	0.879	1.143	0.65	1.632	2.124
4K	0.175	0.439	0.571	0.35	0.879	1.143	0.65	1.632	2.124
5K	0.175	0.439	0.571	0.35	0.879	1.143	0.65	1.632	2.124
6K	0.175	0.439	0.571	0.35	0.879	1.143	0.65	1.632	2.124
7K	0.175	0.439	0.571	0.35	0.879	1.143	0.65	1.632	2.124
8K	0.175	0.439	0.571	0.35	0.879	1.143	0.65	1.632	2.124

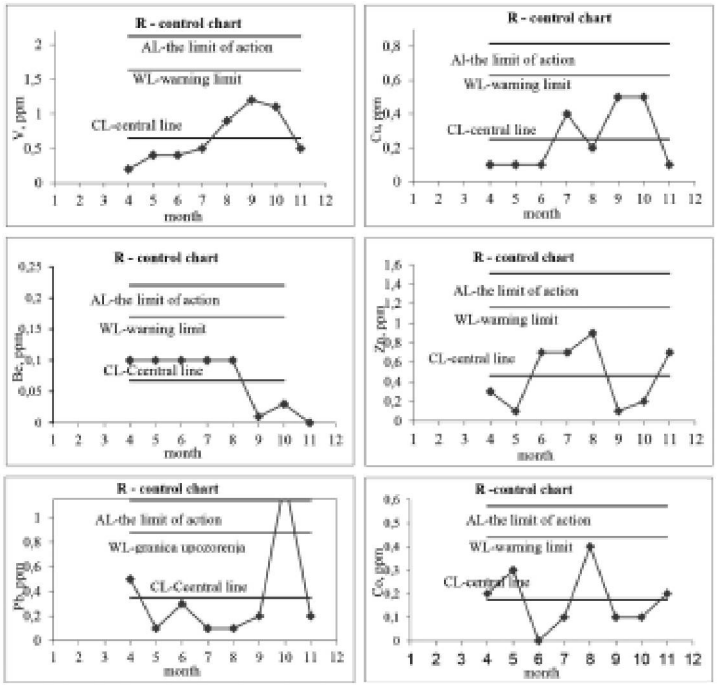

Figure 3. R control charts for V, Cu, Be, Zn, Pb, Co

6. CONCLUSION

On the location 1, the soil sampling was done every month according to the standards given above. Results of the analyzed parameters on the sampled soil are not part of this work, for justified reasons, except blanks and control samples. Their analysis is done according to the law, without any exception. The sampling and sampler control were done in laboratory in two ways:

- With blanks,
- and with taking control samples in duplicate.

Results of chemical analysis of control samples are presented on R-control chart, 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 sample, they reveal the contaminations such as dust and atmospheric precipitation. The conclusion is that the Laboratory for preparation in Mining and Metallurgy Institute, adequately conducts the quality control on soil sampling, that results in high accuracy of chemical analysis outputs.

ACKNOWLEDGMENT

The authors are grateful to the Ministry of Education, Science and Technological Development of Republic of Serbia for financial support for projects TR34005 „Development of advanced materials and technology for multifunctional application based on ecological knowledge", TR 33023 „Development of Technologies for Flotation Processing of Copper Ore and Precious Metals in Order to Achieve Better Technological Results"

REFERENCES

Standard SRPS ISO / IEC 17025, *General requirements for the competence of testing and calibration laboratories,* (2017).

Standard ISO 18400 Soil quality — Sampling —Part 101: *Framework for the preparation and application of a sampling plan*, (2017).

Standard ISO 18400 Soil quality — Sampling —Part 102: Selection and application of sampling techniques, (2017).

Standard ISO 18400 Soil quality — Sampling —Part 104: Strategies, (2018)

Law on Land Protection („SG RS", No. 112 of December 30, 2015).

The Regulation on the Limit Values of Polluting, Harmful and Hazardous Substances in Land (Official Gazette of RS, No. 30 of 20 April 2018)