

## THE USE OF SPC METHOD FOR EVALUATION OF THE CHEMICAL COMPOSITION OF CHOSEN STEEL

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### Abstract

In the paper, the quality research of the steel bars produced by chosen Polish metallurgical enterprise was presented. The chemical composition of the steel grade 1006 produced in this enterprise was determined. Using the principles of Statistical Process Control (SPC), measurement uncertainty based on validated research methods and current spectral analysis of the control samples were also determined. Thanks to the use of this method it will be possible to take preventive action to eliminate errors during the technological process.

**Keywords:** Steel bars, Statistical Process Control, homogeneous of chemical composition

### 1. INTRODUCTION

The requirements imposed by the recipients on the materials produced in the research metallurgical plant cause manufacturers' care for internal quality management systems. This has led to a change in the production technology of its products. Innovations also concerned laboratories evaluating these products. Statistical process control (SPC) methods have become an important part of the quality management [1-3]. The process should take advantage of all the potential evaluation and development. On the basis of the control results, it is possible to introduce the inspection of the corrective and preventive actions. They are reflected in the standards, procedures, and instructions. The basic concept from the point of view of SPC methodology is the variability generated by various process factors such as man, technology, tools, materials [4-5].

The spectroscope is one of the tools used to evaluate materials. Thanks to it, it is possible to determine up to several dozen elements in the research material. The chemical composition of the produced material affects the quality of the final products [6-7]. For the chosen metallurgical plant, methodology for the determination of the chemical composition of melted material samples of low content of silicon (Si) using the SPC principles, was presented. Chemical composition of the research material was defined, uncertainty of measurement based on validated research methods and current spectral analysis of control samples were determined.

### 2. CHARACTERISTICS OF THE METALLURGICAL PLANT

The research metallurgical plant (ZM) started its business several decades ago. In recent years it has undergone tremendous transformation. Thanks to the made investments, it has become a modern and competitive enterprise. Introduction of new technologies that take into account customer needs and natural environmental requirements are a priority for the enterprise's management [8].

### 3. QUALITY ASSESSMENT OF THE PRODUCED MATERIAL

1006 grade steel produced in the ZM was chosen for the research. The analysis of how to make the melt according to the technological instructions was conducted. There were no objections. In order to make a

spectral control sample with low content of Si, a control piece from a cast material on Continuous Casting line was taken and was subjected to forming process in the rolling mill. The diameter of the bar was 50 mm, and its chemical composition was determined during technological process. The sample was cut into 60 mm specimens and properly marked. Then samples were prepared for analysis. From the prepared group of samples, 5 samples were chosen at random. Homogeneity (chemical composition % w.) and standard deviation of these samples were determined (**Table 1**). The chemical composition was checked at two parts of the sample and the average composition (denotation /1, /2) was determined.

**Table 1** Chemical composition (% w), average and standard deviation of the research samples [own study]

No of sample	C	Si	Mn	S	P	Cr	Ni	Cu
1/1	0.057	0.0798	0.323	0.0131	0.0125	0.0549	0.0667	0.2227
1/2	0.0559	0.0796	0.327	0.0132	0.0123	0.0557	0.0669	0.2225
Average 1	0.0565	0.0797	0.325	0.0132	0.0124	0.0553	0.0668	0.2226
2/1	0.0557	0.0802	0.328	0.0147	0.0132	0.0546	0.0678	0.2217
2/2	0.0561	0.0798	0.324	0.0142	0.013	0.0548	0.067	0.2221
Average 2	0.0559	0.0800	0.326	0.01445	0.0131	0.0547	0.067	0.2219
3/1	0.0564	0.0798	0.325	0.0135	0.0125	0.0549	0.0668	0.221
3/2	0.0558	0.0802	0.323	0.0131	0.0121	0.0558	0.0665	0.219
Average 3	0.0561	0.0800	0.324	0.0133	0.0123	0.0554	0.0667	0.2200
4/1	0.0567	0.08	0.324	0.013	0.0122	0.0552	0.0667	0.2229
4/2	0.0569	0.0796	0.326	0.0132	0.012	0.0554	0.0669	0.2215
Average 4	0.0568	0.0798	0.325	0.0131	0.0121	0.0553	0.0668	0.2222
5/1	0.0561	0.0798	0.325	0.0132	0.0116	0.0551	0.0663	0.228
5/2	0.0558	0.08	0.321	0.0129	0.0125	0.0556	0.0669	0.217
Average 5	0.0560	0.0799	0.323	0.0131	0.0121	0.0554	0.0666	0.2225
Average	0.057	0.0798	0.323	0.0131	0.0125	0.0549	0.0667	0.2227
Standard deviation	0.0005	0.00032	0.00156	0.00050	0.00051	0.000248	0.000787	0.00098

The next step was to verify the hypothesis of variance from the obtained results with use of the Fisher-Snedecor test. For this purpose, each sample was tested in 10 samples of determination of chemical elements. In **Table 2** the results of average content of each element from this determination of chemical elements, standard deviations and variances for the research samples were presented.

**Table 2** Average content of elements after the determination of chemical elements, standard deviations and variances [own study]

No of sample	C	Si	Mn	S	P	Cr	Ni	Cu
Average 1	0.0564	0.0800	0.3625	0.0134	0.0123	0.0554	0.0669	0.2225
Standard deviation	0.00074	0.000387	0.001398	0.000371	0.000327	0.00025	0.00060	0.00071
Variance	2.03E-07	1.49E-07	1.96E-06	1.37E-07	1.07E-07	6.32E-08	3.56E-07	5E-07
Average 2	0.0558	0.0797	0.3247	0.0132	0.0122	0.0554	0.0666	0.02218
Standard deviation.	0.00045	0.000345	0.0013375	0.000420	0.000423	0.00032	0.00052	0.00114
Variance	5.27E-07	1.19E-07	1.79E-06	1.76E-07	1.87E-07	1.05E-07	2.67E-07	1.29E-06

No of sample (continue)	C	Si	Mn	S	P	Cr	Ni	Cu
Average 3	0.0562	0.0800	0.32522	0.0132	0.0122	0.0553	0.0666	0.2221
Standard deviation.	0.00073	0.000371	0.001932	0.000414	0.000492	0.00033	0.00048	0.00074
Variance	2.34E-08	1.38E-07	3.73E-06	1.71E-07	2.42E-07	1.09E-07	2.33E-07	5.44E-07
Average 4	0.0563	0.0800	0.326	0.0133	0.0122	0.0554	0.0666	0.2221
Standard deviation	0.00048	0.000294	0.0011547	0.00044	0.00498	0.00021	0.00046	0.00129
Variance	2.96E-07	8.62E-08	1.33E-06	1.94E-07	2.48E-07	4.23E-08	2.1E-07	1.66E-06
Average 5	0.0560	0.0800	0.326	0.0130	0.0120	0.0555	0.0668	0.2227
Standard deviation	0.00054	0.000314	0.0011005	0.000346	0.000432	0.00024	0.00041	0.00106
Variance	2.03E-07	9.88E-08	1.21E-067	1.2E-07	1.86E-07	5.79E-08	1.71E-07	1.12E-06
Minimum variance	2.03E-09	8.62E-08	1.21E-06	1.2E-07	1.07E-07	4.23E-08	1.71E-07	5E-07

A comparison of the calculated results of the Fisher-Snedecor test for five research samples (**Table 3**) was performed. The calculated values were compared with the  $F_{kr}$  value in each research case.

**Table 3** Fisher- Snedecor test results for individual samples [own study]

No of sample	C	Si	Mn	S	P	Cr	Ni	Cu
1	2.695	1.733	1.615	1.149	1.000	1.493	2.078	1.000
2	1.001	1.378	1.477	1.473	1.742	2.480	1.559	2.578
3	2.599	1.599	3.083	1.432	2.260	2.583	1.361	1.089
4	1.154	1.000	1.101	1.622	2.311	1.000	1.227	3.11
5	1.459	1.146	1.000	1.000	1.737	1.367	1.000	2.244

For ten samples from the determination of chemical elements, their comparison with the critical value  $F_{kr} = 3.63$  with probability of 95% was conducted. Because  $F_{obl.}$  (**Table 3**)  $< F_{kr}$ , it was assumed that there were no differences between variances. Thus, the research samples showed homogeneity.

In **Table 4** the minimum and maximum content of individual elements in the research samples were presented. For these values, the minimum and maximum variance was calculated. Then the T value according to the T-Student test was calculated.

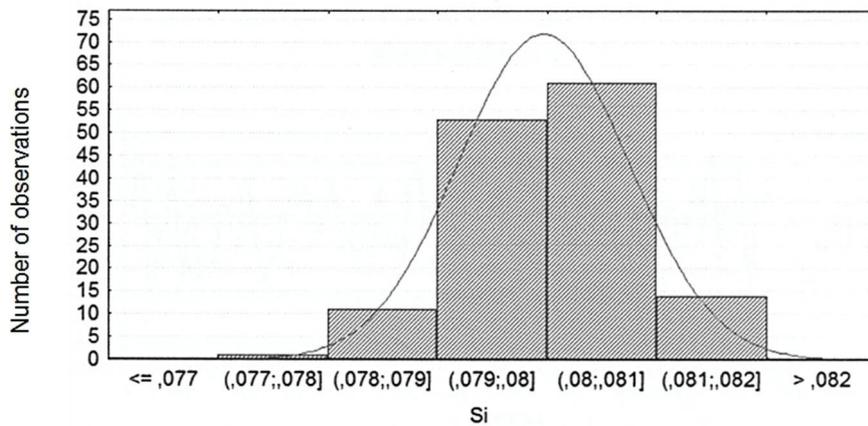
**Table 4** Calculation of T-Student test for individual elements [own study]

	C	Si	Mn	S	P	Cr	Ni	Cu
Minimum content, % w.	0.0558	0.0797	0.325	0.0130	0.0120	0.05526	0.06661	0.2218
Maximum content, % w.	0.0564	0.0800	0.326	0.0134	0.0123	0.05553	0.0669	0.2227
Variance for minimum	2.03E-07	8.62E-08	1.21E-07	1.2E-07	1.07E-07	4.23E-08	1.71E-07	5E-07
Variance for maximum	5.46E-07	1.49E-07	3.73E-07	1.93E-07	5.46E-07	1.09E-07	3.56E-07	1.66E-06
$T_{obl}$	1.973	2.150	2.133	2.033	1.433	2.192	1.263	1.94

For the 10 determined elements contained in the research samples, in the T-Student table the critical value  $T_{tab} = 2.62$  was found. On the basis of the conducted calculations it was concluded that  $T_{obl} < T_{tab}$ .

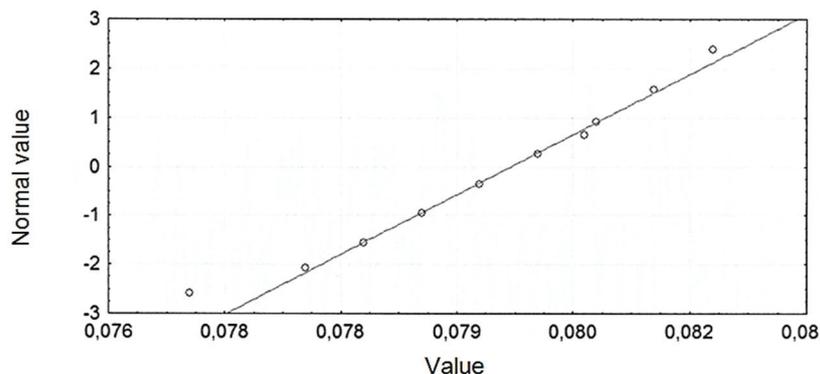
**Table 5** Class size distribution and cumulative population size for Si (silicon) [own study]

Range	Size	Cumulative size	Cumulative important, %	Frequency, %	Cumulative frequency, %
$0.0760 < x \leq 0.0770$	0	0	0.00	0.00	0.00
$0.0770 < x \leq 0.0780$	1	1	0.71	0.67	0.67
$0.0780 < x \leq 0.0790$	11	12	8.57	7.33	8.00
$0.0790 < x \leq 0.0800$	53	65	46.43	35.33	43.33
$0.0800 < x \leq 0.0810$	61	126	90.00	40.67	84.00
$0.0810 < x \leq 0.0820$	14	140	100.00	9.33	93.33
No data available	10	150		6.67	100.00

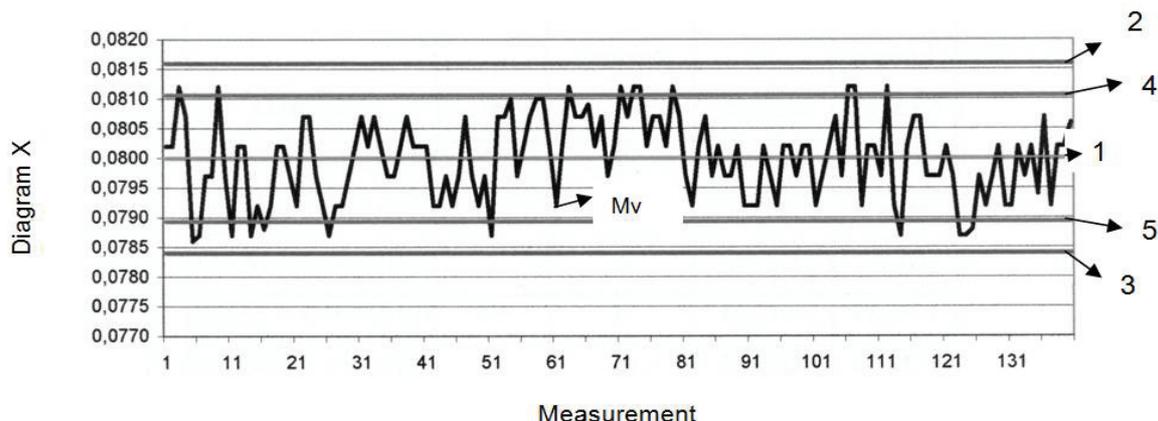


**Figure 1** Normal distribution of spectral analysis results for control samples of silicon of 1006 grade steel melt [own study]

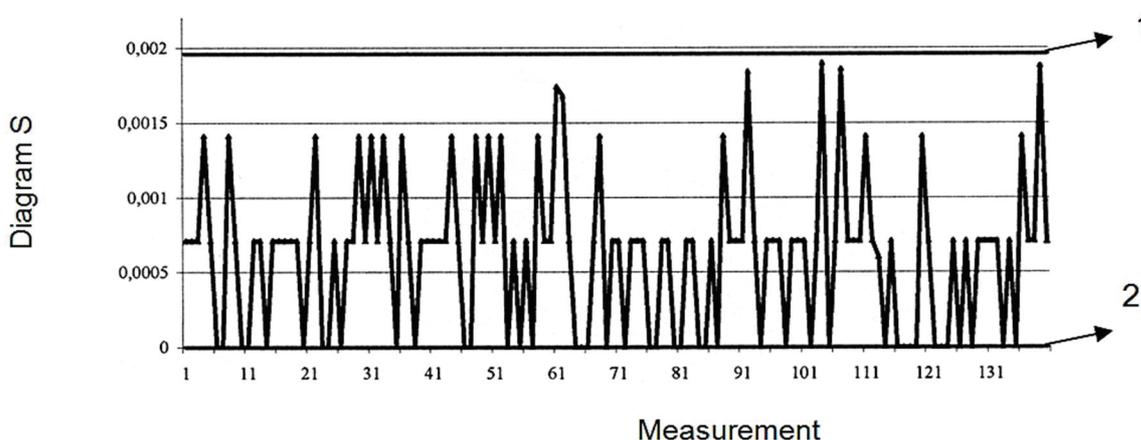
Subsequently, the research samples, which were controlled for homogeneity, were subjected to a standard analysis on an emission spectrometer. This research was performed in an external enterprise Y. The analysis was carried out under repeatability and reproducibility at a certain time. The results of the analysis were adjusted for the difference between the spectral results and the actual value. Average is the result of at least two time of determination of chemical elements with standard deviation lower or equal to the inter-laboratory acceptable standard deviation for the specified analytical range. At the same time histograms were created and normal probability plots for individual elements were developed. The control charts X and the control S for individual elements were constructed. In the paper results for silicon content in the research samples were presented (Table 5, Figures 1 - 4).



**Figure 2** Normal distribution of probability of spectral analysis for silicon of 1006 grade steel melt [own study]



**Figure 3** Control chart X of spectral analysis for silicon of 1006 grade steel melt  
1 - Central line; 2 - Upper control limit; 3 - Lower control limit; 4 - Upper warning limit;  
5 - Lower warning limit; Mv - Measurement points [own study]



**Figure 4** Control chart S of spectral analysis for silicon of 1006 grade steel melt  
1 - Upper control limit; 2 - Lower control limit [own study]

#### 4. CONCLUSION

An analysis of the control sample from the material used for the production of 1006 grade steel bars with low content of silicon on validated research methods [9-10] according to the SPC rules which are in force in the laboratory Y, was carried out. At the same time a verification of records from smelting technological process, from which samples were taken for the research, was carried out. The control covered all relevant elements of the whole production process. The effect of the paper was also to check the predispositions of individual employees to the process of sample preparation and analysis execution. This allows to modify technological instructions and actions included in it.

The conducted analysis made it possible to conclude that the research material was homogeneous in terms of chemical composition. This is evidenced by the conducted research and the basic results of statistical analysis. These results are the effect of the application of standards, procedures, technological instructions and principles of good laboratory practice.

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