

# ***Determination of Standard Sample Composition When Developing Standard Samples of Establishment***

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## ***Abstract***

The paper dwells upon developing Standard Samples of Establishment (SSE) used for the recalibration in optical emission spectrometry. Elemental composition was determined for a line of State Standard Reference Samples (SSRS) of steel by atomic absorption, atomic emission, and infrared spectroscopy. The instruments used for the development, i.e., optical emission spectrometer, flame atomic absorption spectrometer, inductively coupled plasma atomic emission spectrometer, carbon and sulfur analyzer, as well as metal sample spectral speciation techniques and regulatory documents are listed. An algorithm for building a future SSE line for Research Centre JSC RPA CNIITMASH is described.

## ***Introduction, Aims and Tasks***

The RPA CNIITMASH JSC's chemical and spectral analysis laboratory tests steels and alloys used in the manufacture of nuclear power plant structures in Russia and the CIS countries to define their elemental composition. Most samples provided are tested using the express technique - the optical emission spectrometer. This instrument features a factory calibration built in the software. This facilitates the analysis and reduces its time, while a new problem arises: the matrix composition of samples, as well as the factory calibration and laboratory measurement conditions, differ. In this case, a single-point recalibration is performed.

### ***Heading 1***

#### **Objectives:**

- select the SSRS for the SSE development and pre-evaluate its composition,
- determine the metrological characteristics of the future SSE line samples using flame atomic absorption, inductively coupled plasma atomic emission, and infrared spectroscopy techniques,
- study the SSRS into SSE conversion stages and related technical documents.



Figure 1. SSRS No. 49-v line (future SSEs)

	C	Si	Mn	Cr	Ni	Cu	Nb
«491-v»	0.20	0.14	5.80	19.3	7.09	0.47	0.043
«492-v»	0.14	0.26	7.90	16.8	5.40	0.26	0.045
«493-v»	0.18	0.50	9.49	13.8	4.25	0.17	0.046
«494-v»	0.15	0.97	11.00	11.8	3.18	0.14	0.048

Table 1 - Preliminary concentration data (%) for the line No. «49-B» samples

## **Materials and Equipment**

- Since documents have been lost and cannot be restored (and the available SSRS validity has expired), SSRS No. 49 (USSR - the 1970s) has been selected

- High-purity argon (99.998 % min)

- Oxygen (95 % pure min)

- FOUNDRY-MASTER SMART UVR optical emission spectrometer

- OPTIMA 2100 DV inductively coupled plasma atomic emission spectrometer,

- Perkin Elmer Model 403 atomic absorption spectrometer,

- LECO CS-244 carbon and sulfur analyzer

## **Results and Discussion**

The analysis of line samples on the FOUNDRY-MASTER SMART UVR optical emission spectrometer has shown that this line corresponds to the steel grades 10Kh13G12BS2N2 and 10Kh13G12BS2N2D2.

The SSRS No. "49-v" line is suitable to solve the most frequently occurring analytical tasks (Table 1), i.e., recalibration and adding calibration points for Mn, Ni, and Cr elements since to date, the spectrometer database lacks the following concentration ranges: for Mn - 5-10 %, for Ni - 3-7 %.

SSE	C	Si	Mn	Cr	Ni	Cu	S
«491-v»	0.198	0.156	6.29	18.84	6.99	0.394	0.006
«492-v»	0.193	0.297	8.42	16.11	5.46	0.228	0.011
«493-v»	0.193	0.539	10.43	13.47	4.31	0.142	0.009
«494-v»	0.212	0.902	12.35	11.56	3.24	0.119	0.007

Table 2 – Metrological Characteristics Obtained for the SSE 49-v Line - Mass Fraction of Elements, in Percent

SSE	C	Si	Mn	Cr	Ni	Cu	S
«491-v»	0.016	0.007	0.467	1.40	0.519	0.018	0.0012
«492-v»	0.015	0.014	0.626	1.20	0.406	0.010	0.0011
«493-v»	0.016	0.025	0.778	1.00	0.322	0.009	0.0012
«494-v»	0.017	0.041	0.917	0.86	0.243	0.007	0.0002

Table 3 - Expanded Uncertainty of the Values Obtained with a Confidence Factor of 0.95, in Percent

## ***Conclusion***

The research outcomes:

- an SSRS specimen line was selected to develop the SSE line,
- specimens were analyzed by ICP AES, AAS, and IR spectroscopy, and metrological characteristics were determined for the future SSE line,
- the SSE development stages were defined.

It is planned to further issue an SSE line with its own datasheet, marking, and number in the Register of Standard Specimens of Establishment.

***Thank you for your attention!***