EXPERIENCE IN USING A POLAROGRAPHIC METHOD IN THE ANALYSIS OF MINERAL RAW MATERIALS

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The Central Laboratory of the Southern Kazakhstan Geological Administration has been using the polarographic method on a large scale since 1949.

We decided to make a critical evaluation of the suitability of the existing method for the large-scale analysis of mineral raw materials, to develop new methods which would be sufficiently sensitive and accurate, and to find organizational measures which would give greater economies. These problems were solved by D. P. Shcherbov, O. N. Pashevkina, V. P. Bakarasova, and I. I. Sagalovich. In 1949-1950 more than 56,000 polarographic determinations were performed and in 1952-1955 more than 100,000.

In recent years the workers of the laboratory have published about 19 articles with a description of the methods which have been developed and a number of teaching seminars have been held.

In our laboratory lead, zinc, copper, cadmium, tin and fractions of the phase analysis of lead, zinc and copper are only analyzed by a polarographic method, with sampling of specimens containing >25% Cu and Pb by a chemical method.

The methods used are described below.

Tin in the form of cassiterite. After acid decomposition the residue was fused with sodium peroxide, leached with water, the silica was separated and the solution was polarographed using a hydrochloric acid supporting electrolyte (2:3) in the presence of hydrogen-reduced metallic iron within the limits −0.3 and −0.6 V.

Lead. After acid decomposition the whole sample (0.2 g) was polarographed using a hydrochloric acid (1:4) supporting electrolyte with the addition of iron powder or with a supporting electrolyte of calcium or sodium chlorides. The polarograph limits were −0.3 and −0.6 V.

Copper, cadmium and zinc. After acid decomposition the whole sample (0.2 g) was directly polarographed using a chloride-ammonia supporting electrolyte. The polarograph limits were: for copper −0.4 and −0.8 V, for cadmium −0.7 and −1.1 V, for zinc −1.3 and −1.6 V. Cadmium and zinc were determined in copper ores after cementation of the copper on lead plates.

The analytical work was organized in the following way. All samples of polymetallic ores arriving for analysis were subjected to semiquantitative spectrum analysis. The results were used to select "empty" samples and samples with a low content of the required elements (only samples containing not less than 0.1% Pb, 1.0% Zn, 0.01% Cd, 0.1% Cu and 0.05% Sn were analyzed). This preliminary selection avoided unnecessary expense and prevented the laboratory being overloaded with unnecessary analyses.

For conditional samples "sampling" was performed with an indication of the approximate content and the presence of interfering impurities. The most efficient methods were then selected for the determination, the weight of sample and the dilution. For simultaneous analysis the analysts were given two or three batches containing between 50 and 100 samples. The work was organized so that the difficult operations in the analysis of one batch coincided in time with the normal analytical operations for other batches. The determinations were performed on visual instruments with the results calculated by the method of "two readings."*

Before the polarography, daily determinations are made of the start and end of the wave, the volt-ampere curves are taken of the control specimen with the maximum content of the required element and several samples which are to be analyzed; these polarograms are used to check the position of the start and end of the wave and

* I. G. Grinman specifically designed the visual PPT-2 polarograph for work with this method; it is produced by the experimental workshop of the Energetics Institute, Academy of Sciences, Kazakh SSR.

the potentials of both points taken for the calculation of the results. The results of the determination are calculated using a special logarithmic slide rule. There is systematic strict control of the quality of the determinations by the crossed analysis of numbered samples and the inclusion in each analyzed batch of standard or control specimens. The results for the external control of the laboratory work are shown in the table.

Results of Control Determinations

Element being determined	Number of samples	Content in samples		Error		$\frac{MU}{MKh}$
		main Mkh	control MU	absolute D	relative $\frac{D}{MKh} \cdot 100$	
Pb	1	0.31	0.29	0.02	6.4	0.9
	24	1.83	1.80	0.10	5.5	0.9
	2	8.24	8.08	0.16	1.9	0.98
	1	0.34	0.32	0.02	5.9	0.97
Zn	19	2.13	2.01	0.12	5.6	0.9
Cu	29	0.25	0.26	0.01	4.0	1.0
	53	1.48	1.50	0.05	3.4	1.01
	111	2.29	2.29	0.07	3.0	1.0
Sn	216	0.11	0.11	0.009	15.6	1.0
Cd	87	0.06	0.06	0.005	10.8	1.0

In large-scale polarographic analyses the productivity for the determination of copper, cadmium, zinc, tin, and lead increased 3-4 fold compared with chemical methods; expenditure on reagents was reduced by a factor of 5-8.

The use of the polarographic method for determining rare and scattered elements in mineral raw material is held up due to the lack of high-sensitivity up-to-date electron polarographs. After the laboratory has been equipped with these instruments in 1962 the use of polarographic analysis will be considerably extended.

EXPERIENCE IN OPERATING A SPECTRUM ANALYSIS BASE LABORATORY

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In accordance with the recommendations of the Second Urals Conference on Spectroscopy in 1960 the Sverdlovsk Council of National Economy organized a spectrum analysis base laboratory at the Urals Research Institute for Ferrous Metals. It was to coordinate work in applied spectrum analysis, control the introduction of spectroscopic methods in industry, perform research and development work in order to improve and automate analytical control in industry and also to give scientific assistance to industrial laboratories. The laboratory cooperates with plants of the Sverdlovsk economic region and also with plants of the Chelyabinsk and Perm regions, Cherepovetsk and Novosibirsk Metallurgical Plants, the "Elektrostal'" plant, and the Kuznetsk Metallurgical Combine.

An important feature in the work of the laboratory is the publicity and introduction of the latest techniques.

In the technical information bulletin of the Council of National Economy, and in the regional and city newspapers there are articles on photoelectric methods of spectrum analysis, and on latest techniques. In 1961 an interfactory school was held to exchange the latest experience in the operation of spectrum laboratories at ferrous