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ANALYSIS OF RARE AND SEMICONDUCTOR ELEMENTS

- USSR -

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[Following is a translation of an article by M. P. Volynets in Vestnik Akademii Nauk SSSR (Bulletin of the Academy of Sciences USSR), Vol. XXX, No 3, Moscow 1960, pages 101-103.]

The 21st session of the Communist Party of the Soviet Union planned a significant increase in the production of rare and semiconductor elements, heat-resistant alloys, anti-corrosives, and other materials required by the new technology. Methods of analytical control at all stages of production of these materials play an important role in the resolution of this problem. Taking this into account, the Gosplan (State Planning Commission) of the USSR, the State Scientific-Technical Committee of the Council of Ministers of the USSR, and the USSR Academy of Sciences organized a conference for rare and semiconductor elements which took place in Moscow from 7-11 December 1959.

The conference had as its goal the detailed consideration, systematization and crystallization of much factual material compiled in this field of research by the institutes of the academies of sciences of the USSR and the Union Republics, affiliated scientific-research institutes, institutes of higher learning and industrial laboratories. About a thousand representatives from 285 organizations and foreign guests participated in the work of the conference. One hundred and fifty reports examined the methods of analysis of rare and semiconductor elements: Li, Rb, Cs, Be, In, Ga, Tl, Sc, Y, rare earths, Si, Ge, Ti, Zr, Hf, Th, V, Nb, Ta, Mo, W, Se, Te, Re.

Presented before plenary sessions were the reports of A. P. Vinogradov concerning the basis of conditions for impurities in pure metals, and of N. P. Sazhin concerning the requirements of industry with respect to the degree of purity of materials. In these reports it was noted that the development of new branches of modren technology has specified the broad use of rare metals whose requirements for purity steadfastly increase. Although in 1941 methods for the determination of impurities in metals were completely acceptable with a sensitivity of 10^{-2}-10^{-3}%, in 1950 for the majority of pure metals impurities were determined to 10^{-4}%, and in the case of certain especially harmful impurities (B, Gd)- to 1×10^{-5} - 10^{-6}%. Especially stringent demands for the purity of metals are made in the manufacture of semiconductor devices (electronics, telemechanics, automation, etc.) For Ge and Si used in semiconductor devices, impurities of As, Cu, and Ni are harmful even in amounts of 1×10^{-7} -1×10^{-8}%. For thermonuclear energetics materials are required in which the allowable content of impurities

is not in excess of 1×10^{-9} - 1×10^{-10}%. In this connection analysts are faced with exceptionally complex tasks, related to the development of especially sensitive methods of analysis of pure products, reagents and various materials used in technological processes.

I. P. Alimarin shed light on prospects for increasing the sensitivity and accuracy of analytical methods. He noted that progress in the analytical chemistry of rare elements and the possibility of detecting impurities in them of less than 1×10^{-6}% is tightly bound to the employment of new instrumental methods of analysis. Feasible means of increasing the sensitivity of analytical methods (widely adopted as well as new) were outlined in detail in the report. Along with chemical methods of concentration of microimpurities, considerable attention must be allotted to physical methods of enrichment, in particular vaporization and sublimation at high temperatures in a vacuum and zone melting. Radioactive isotopes can be effectively employed in the control of the enriching process. R. L. Globus surveyed the present state and prospects of development in the chemical reagents industry.

In separate sessions devoted to questions touching upon the analytical chemistry of the individual rare and semiconductor elements, leading specialists in the given field came forth with summary reports (D. I. Ryabchikov, I. P. Alimarin, V. A. Nazarenko, N. S. Poluektov, A. I. Busev, V. L. Zolotavin et al.) The materials brought to light in these reports were crystallized and complemented by short reports and presentations dedicated to the detection of rare elements in ores, tailings, raw stocks and other products, and to the quantitative determination of various impurities in metals of high purity. In these presentations were outlined methods of spectroscopy, x-ray spectroscopy, flame-photometry, photometry, polarography, potentiometry, chromatography, radio-activity, and others.

The conference indicated the need for the perfection of highly sensitive, selective, rapid and simple methods of analysis, which would permit the detection of impurity in amounts of from 10^{-4} to 10^{-8}%. As most promising methods, according to the opinion of the conference, it is expedient to recommend the development of fluorescent, chemiluminescent, catalytic, and radio-active methods. (We must) expediently and in the future develop photometric methods, methods of flame photometry, emission spectrum, and x-ray spectrum analysis, isotopic dilution analysis and ultramicro-analysis. Of the number of methods employed in preliminary concentration, those used most successfully are co-precipitation, extraction, chromatography, ion exchange, electrolysis, sublimation and vaporization, and also zone melting.

In the sessions of the sections on physical and chemical methods of analysis general questions concerning instrumental means were studied. Along with a clarification of achievements in the field of physical methods introduced into practice in most recent years feasibilities for the employment of methods not yet adopted or of small abundance in analytical chemistry were considered. To their number belong the methods of nuclear and paramagnetic resonance, the method of mass spectrometry, and gamma spectrometry (N. M. Pomerantsev, M. S. Chupakhin, and I. K. Zadorozhnyy, V. I. Baranov and co-workers). A special survey was devoted to the present state of

x-ray spectrum analysis and the means of its adaptation in the field of the analysis of x-ray absorption spectra (E. Ye. Vaynshteyn). Methods were outlined in the report for x-ray fluorescent analysis, analysis of microvolumes of a substance, and the use of the fine structure of spectra for studying the physical state of atoms in condensed media.

In the examination of new methods of spectral analysis of raw minerals and materials of high purity it was indicated (A. K. Rusanov and V. V. Nedler), that the creation of more refined excitation sources for spectra is one of the most promising directions. In efficient combination with methods for preliminary physical and chemical concentration this direction allows an increase in sensitivity in the very near future of a minimum of one to two orders, and an increase of considerable measure in the accuracy and rapidity of spectral determinations.

As A. N. Frumkin emphasized in his introductory remarks, the most successfully exploitable directions for electrochemical analysis are in the use of polarographic maxima, solid electrodes and other devices. The present state and prospects for the adaptation of the newer electrochemical methods of analysis were the subject of a special report (O. L. Kabanov). In this section were elucidated the most promising methods of electrochemical analysis: preparatory enrichment in mercury drop electrode (S. I. Sinyakova and co-workers), oscillographic polarography (Ya. P. Gokhshtejn), alternating current polarography (S. B. Tsfasman and B. Ya. Kaplan), amperometric methods (Yu. I. Usatenko) and other methods.

The critical examination of all the material for the purpose of choosing the most rewarding methods of analysis of the rare and semiconductor elements appeared to be a long pressing problem, as was noted in the conference. The necessity of publishing a guidebook in the near future on the employment of better methods was recognized.

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