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**Badania nad metodami rozdzielania niobu i tantalu  
oraz opracowaniem metod oznaczania tantalu  
w związkach niobu**

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## Summary

Results of studies on search for optimal conditions for determination of tantalum in niobium and its compounds using the ICP-OES method were presented. The procedure allowing to convert different types of niobium samples in identical form to be analysed in non-fluoride solutions without previous separation of Ta(V) from Nb(V) matrix was developed. This method involves digestion of the sample in the mixture of concentrated mineral acids usually with the addition of HF, precipitation of slightly soluble niobium acids (tantalum) from the obtained solution and converting them into soluble complexes. The application of oxalates complexes of Nb(V) and Ta(V) containing the controlled addition of H<sub>2</sub>O<sub>2</sub> and quartz elements in spectrometer ICP-OES ensures a better transport of the sample and better excitation of tantalum (as compared to fluoride solutions) and higher sensitivity of the method necessary for the determination of low content of tantalum in niobium or niobium compounds ( $10^{-3}\%$  Ta) with relatively high precision and accuracy. The advantage of this method is the possibility of using the standardised set of matrix reference solutions for all types of analysed niobium samples. Solutions of purified niobium used for this purpose were obtained simultaneously during studies on efficiency of existing and new separation methods for these two elements, especially in systems containing high initial ratio of niobium to tantalum ( $m_{\text{Nb}}^0 : m_{\text{Ta}}^0 > 10\,000$ ). The developed procedure of typical extraction of fluoride complexes Nb(V) and Ta(V) using ketone MIBK and of re-extraction of Ta(V) 4% (NH<sub>4</sub>)<sub>2</sub>C<sub>2</sub>O<sub>4</sub> proved to be efficient for their separation in the scope of initial concentrations ( $10 < m_{\text{Nb}}^0 : m_{\text{Ta}}^0 \leq 1\,000\,000$ ) provided the concentration of HF was constantly controlled. For optimal acidity conditions, this method allows to obtain high recoveries of Ta(V) to oxalate phase with a small spread of values around average value (RSD  $\leq 5\%$  with the exception of  $m_{\text{Nb}}^0 : m_{\text{Ta}}^0 = 1000\,000 : 1$ ) and practically independent of the ratio of niobium to tantalum. The determined extraction efficiency of Ta(V) allows to recommend this procedure for initial concentration of tantalum before its determination in niobium and its compounds at the level of  $10^{-4} \div 10^{-3}\%$  Ta (as compared to Nb). The solution of problems resulting from generation of large amounts of waste formed during extraction is the application of solvent-impregnated sorbents. Using this extraction chromatography method with the application of MIBK impregnated active carbons, a new and very efficient system for separation of trace amounts Ta(V) from Nb(V) was developed. The applied sorbent is inexpensive, easy to prepare and selective as compared to tantalum for systems containing fluoride complexes Nb(V) and Ta(V). Studies carried out under dynamic conditions confirmed the possibility of obtaining high purity niobium solutions (0,027 ppm Ta as compared to Nb) with good niobium efficiency 88% from solutions with weight ratio  $m_{\text{Nb}}^0 : m_{\text{Ta}}^0 = 1000 : 1$ , whereas the achieved purge factor was a 35000 which makes the developed method appropriate for deep purification of niobium compounds.