

DETERMINATION OF RHENIUM IN EXTRACTANTS BY INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROMETRY

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Extraction methods having high selectivity are widely used in technology of rhenium. Traditional methods of rhenium determination in extracts based on the analysis of aqueous solutions and on the calculation of its content using difference of concentration in initial and finished solutions. Direct determination of rhenium in extracts is connected usually with the sample preparation by caking with oxidized agents. This process lead to losses of rhenium [1]. Direct determination of rhenium by the neutron activation and the gamma-activation methods is characterised by high expences and the use of special devices.

The aim of paper is the elaboration of technique of direct determination of rhenium in extractants and in the active phase (immobilised extractant) of impregnates and tvexes by using of inductively coupled plasma atomic emission spectrometry (ICP-AES). The ICP-AES analysis is very fast, and gives accurate, precise results. This method is mainly used for analysis of aqueous solutions, thus the preparation of impregnates and tvexes samples concludes the washout of extractant by acetone [2]. However, acetone has high volatility at room temperature. By this reason direct analysis of acetone solutions using standard devices is difficult to be realized due to the quenching of the plasma, the possible changes in content of samples and standards in the course of analysis. The distillation is used for the separation of acetone from extractant.

The small volume of organic phase obtained by the washout and the distillation was diluted by organic solvent – tributylphosphate (TBP). Tributylphosphate is properly introduced into the plasma due to its physico-chemical properties (saturation vapour pressure, density, viscosity, and surface tension). The matrix effects stemmed by radiation of lines CN, CO etc. are poorly expressed. TBP is properly mixed with extractants, and it can hold different metals. The standards are conveniently prepared by using TBP.

The primary standard contained 1g/l Re is prepared by dissolving of weighed sample of metallic rhenium (50 mg) in 3 ml TBP after prior saturation by concentrated nitric acid (ratio is equal to 1:1). The time of contact is 15 min. The prepared solution was diluted by pure TBP in the flask of 50 ml. The standards were prepared by dilution of the primary standard by pure TBP. The standards containing 0.1, 0.05, 0.02, 0.005, 0.002, 0.0002 g/l Re have been prepared.

A Jobin Yvon JY-38 Inductively Coupled Plasma Atomic Emission Spectrometer was used for all the measurements. The operating conditions used are listed in Table 1.

Table 1. Operating conditions for ICP-AES

Forward power	1300 kW
Intermediate plasma gas flow rate	0.6 l*min ⁻¹
Outer gas flow rate	14 l*min ⁻¹
Nebulizer	Concentric, glass
Aerosol carrier gas flow rate	0,3 l· min ⁻¹
Spray chamber	Scott, glass
Torch	Quartz Fassel type
Observation height above load coil	15 mm

The results of analytical possibility estimation for different lines of rhenium are presented in Table 2.

Table 2. Spectral wavelengths, detection limits of Re in TBP

Spectral wavelength	Detection limit, ppb
Re II 213.904	76
Re II 214.274	129
Re II 221.426	16
Re II 227.525	12

The testation of absence of matrix influence caused by different nature and content of organic phase was implemented by using model samples by the method “added-found”. The model samples were prepared as standards except that some sample organic phase identical to content used by impregnation and saturated by acids extractant was added to them. The results of processing of data obtained show that there is no significant discrepancy between proposed and determining concentration of rhenium. This technique makes possible determination of rhenium in extractants in the range 0.2-100 mg/g, RSD=0.8-1.7%.

References

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