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UNIVERSITY OF PRETORIA
YUNIBESITHI YA PRETORIA

Trace element analysis in precious metals using Time Resolved Emission Spectroscopy

by

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SYNOPSIS

Determining the purity of precious metals has received considerable scientific attention, mainly because of the high intrinsic value of these precious metals.
These metals are extremely useful as catalysts in the chemical and petroleum industry. They have been used as exhaust catalysts and as conductors in the electrical industry, in medical and dental applications as well as in the jewellery industry.

Two methods of analysis are used to determine the purity of metals; the direct and indirect method of analysis. During the direct method of analysis, the actual percentage of metal is directly determined. Techniques used are the Fire Assay, gravimetrical wet chemical techniques. However, these techniques are not suitable for characterising individual precious metals and impurities.

Indirect methods, based on spectroscopy, are well suited for the analysis of high purity metals. Using these techniques, the various impurities and their concentrations are determined and the purity of the metal is determined by difference. It is therefore

important that all impurities are determined with the highest degree of precision and accuracy, as this influences the ultimate purity and quality of the product.

A method was developed to determine the impurities in Ruthenium. The limits of detection obtained were in some instances improved up to a factor of ten times, compared to the current techniques used for the determination of the impurities. Elements such as Cd, Se, As and Cr which were previously reported as not detected, due to the difficulty of detecting them at levels below 10–15 ppm, are now measurable using the spark spectrometer. Making use of time resolved spectroscopy has in general increased the sensitivity by a factor of five to ten times, even when compared to normal spark analysis as used for Alloys.

The technique used in this study in the analysis of impurities in precious metals is known as SAFT (Spark Analysis For Traces). SAFT involves the use of time resolved spectroscopy, which is able to reduce background and thus improve signal to background ratios of spectral lines. It makes use of the phenomenon of "atomic afterglow". By switching the photomultiplier detector to observe (measure) only the afterglow, the background radiation can be separated from the atomic radiation.

The technique is very sensitive and analyses of purities at 99.995 % are possible. However, the greatest drawback of the method when used for precious metals is the lack of commercially available standard samples for low concentration of impurity.

Spoorelementanalise vir platinum metale met behulp van tydontledings Emissiespektroskopie

deur

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SAMEVATTING

Suiwerheidbepaling van edelmetale geniet baie wetenskaplike aandag hoofsaaklik as gevolg van hulle hoë intrinsieke waarde. Hierdie metale is baie waardevol en word as katalisators in die chemise en petrochemiese industrie bebruik, asook uitlaat-systeemkatalisatore en as geleiers in die elektriese industrie, mediese- en tandheelkunde en juweliersindustrieë.

Daar is twee tipes analise vir die bepaling van suiwerheid van metale: 'n direkte en indirekte metode van analise. Die direkte metode van analise, bepaal die persentasie waarde van die metaal op 'n direkte wyse. Hierdie metodes sluit in, traditionele essaieering, gravimetriese en nat chemiese tegnieke. Hierdie tegnieke is nie geskik vir karakterisering van individuele edelmetale en onsuiwerhede nie.

Indirekte metodes is gebaseer op spektroskopie en is geskik vir die hoë suiwerheid analise van metale. Hierdie tegnieke bepaal die konsentrasie van die onsuiwerhede. Die suiwerheid van die metal word bepaal deur die verskil. Om hierdie rede is dit baie belangrik dat al die onsuiwerhede bepaal word met die hoogste graad van presisie en akkuraatheid, omdat dit 'n invloed sal hê op die uiteindelike suiwerheid en kwaliteit van die produk.

Die alternatiewe metode is ontwikkel om die onsuiwerhede in Ruthenium te bepaal. Die deteksie limiete in sommige gevalle het met 'n faktor tot tien verbeter, invergelyking met huidige tegnieke wat gebruik word vir die bepaling van onsuiwerhede. Elemente soos Cd, Se, As en Cr was voorheen as onbepaald geraporteer omdat deteksie limiete laer as 10–15 ppm nie moontlik was nie, maar nou wel moontlik is met vonkanaliese.

Die tegniek gebruik vir die analiese van onsuiwerhede in die edelmetale is bekend as SAFT " Spark Analysis For Traces" (Vonkanaliese vir spoorelemente). SAFT maak gebruik van tydontledingsspektroskopie, wat agtergrond kan verminder en daardeur die sein tot agtergrond verhouding van spektrale lyne verbeter.

Dit maak gebruik van die atoomgloei. Deur die fotovermenigvuldigerdetektor te skakel om slegs die atoomgloei waar te neem kan die agtergronduitstraling geskei word van die atoom uitstraling. Die sensitiwiteit is 'n faktor van vyf tot tien keer beter as met normale vonkanalise.

Die tegniek is baie sensitief en analise met suiwerhede van 99.995 % is moontlik, alhoewel die grootste nadeel van die metode vir edelmetale is die gebrek aan kommersieële beskikbare standarde met laë konsentrasie onsuiwerhede in.

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