Direct Determination of Trace Elements in ZrO_2 By D.C. Arc-Carrier Distillation Technique Using CCD-based Spectrometer

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INTRODUCTION

Zirconium is one of the key elements used in the nuclear and chemical industries. In the chemical industry, zirconium oxide is used as a catalyst and catalyst support. Having good chemical stability and excellent electric conduction, ZrO_2 -based ceramics have been widely applied in manufacturing piezoelectric components, capacitors, solid electrolyte fuel batteries, etc. (1,2). In the nuclear industry, zirconium oxide is used as a starting material for the fabrication of clad material such as zircaloy and also for the fabrication of metallic fuel alloys such as U-Zr, U-Al-Zr, U-Pu-Zr, etc. The role of Zr in manufacturing various core components in a nuclear reactor is mainly due to its properties: resistance to embrittlement, very low thermal neutron absorption cross section, stability under elevated temperature and irradiation conditions, compatibility with fuel pellets, and good heat transfer characteristics.

In view of this, it is required to have a stringent chemical quality control of ZrO_2 similar to other nuclear-grade materials. Because the impurities in high purity ZrO_2 powder strongly influence its properties, it is of great importance to determine these elements at trace levels. Trace metal assay by direct current (D.C.) arc carrier distillation using a direct reading spectrometer (DRS) with photomultiplier tube (PMT) detectors for various matrices has already been reported by our laboratory (3-5).

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ABSTRACT

A method for the direct determination of trace elements at concentrations as low as sub-ppm and ppm levels in a ZrO_2 matrix has been developed by direct current (D.C.) arc carrier distillation, employing a charge coupled device (CCD)-based spectrometer using a 5% carrier combination of AgCl-SrF_2 (4:1). The CCD-based spectrometer was used to examine the analytical performance of a wider number of analytical lines and the determination of more analytes in comparison to earlier reports with a direct reading spectrometer (DRS) having a photomultiplier tube or photographic plate for detection.

The analytical method developed in the present work has the potential to simultaneously determine 24 elements (Al, B, Be, Ca, Cd, Co, Cr, Cu, Fe, In, Li, Mg, Mn, Mo, Na, Ni, Pb, Sb, Si, Sn, V, Ti, W, and Zn) in a ZrO_2 matrix with low limits of quantification ranging from 0.05-20 ppm. In the absence of a certified reference material, the method was validated by analyzing synthetically prepared samples. The relative standard deviation for the analytes was found to be better than 25% for analyte concentrations in the intermediate range.

Atomic emission spectrometric (AES) methods using spark and inductively coupled plasma (ICP) as excitation sources are reported in the literature for the trace metal assay of ZrO_2 (6-10). In general, the trace elemental assay of ZrO_2 by the AES method has been carried out

by correcting for the spectra of the Zr atoms and thereby improving the limit of detection (11-14). In particular, Chen et al. (15) reported the determination of rare earth impurities (La, Pr, Eu, Gd, Ho, and Yb) at trace levels in a ZrO_2 matrix. The removal of Zr was facilitated by converting it to its volatile fluoride to minimize the spectral interference from Zr, followed by ICP-AES analysis. Gordon et al. (6) reported the determination of impurities (Fe, Si, Al, Cu, Cr, W, Mo, Ni, Mn, Mg, Ti, Pb, and Hf) in zirconium and hafnium using a spark excitation source based on a photographic technique, while Easterday (7) reported the determination of Al, Fe, Co, Ti, Sn, V, Ni, Si, Mg, Mn, B, Cr, Cu, and Cd in ZrO_2 using spark excitation source based on a direct reading spectrometer. The determination of trace elements in ZrO_2 by an electrothermal atomic absorption spectrometric method has also been reported by Krivan et al. (16) for Cd, Co, Cr, Cu, Fe, Li, Mn, Ni, and Zn.

Application of the carrier distillation technique for the selective release of trace metallic impurities from refractory samples, such as U_3O_8 (17), PuO_2 (18), and ThO_2 (4), are well established. There is only one report on the use of a charge coupled device (CCD)-based spectrometer with D.C. arc excitation source for trace metal analysis in a matrix of boron carbide (19). However, the advantages of using a CCD-based spectrometer with a D.C. arc excitation source for trace metal analysis are not fully explored. Thus, the present work mainly focuses on the development of an analytical method using a

CCD-based spectrometer for trace metallic constituents in ZrO_2 by D.C. arc carrier distillation. The method was validated by the analysis of several synthetic samples, prepared in-house.

EXPERIMENTAL

Instrumentation

A Spectro Arcos (USA) direct reader spectrometer coupled with a D.C. arc and an ICP source was used. The unit is equipped with linear arrays of CCD detectors arranged in a Paschen-Runge mount having a focal length of 750 mm, fitted with three holographic gratings, two of them with 3600 grooves/mm and one with 1800 grooves/mm. This arrangement enables a wavelength coverage of 130–770 nm in the first order. Also, the spectrometer provides a resolution of 0.01 nm in the 130–340 nm region and 0.02 nm in the wavelength regions greater than 340 nm.

Reagents and Standard Solutions

SpecPure® grade oxides of individual elements (AgCl supplied by SPEX Industries, USA, and high purity pre-analyzed ZrO_2 procured from NFC Hyderabad) which served as a base material were used for preparation of the standards.

Electrodes

Lower electrode (anode): Standard carrier distillation-type electrode, ASTM designation E-130-66 type S-2 on a type S-1 pedestal.

Upper electrode (cathode): Preformed pointed electrode, ASTM designation E-130-66 type C-1.

Preparation of Standards and Samples

The required detection limits for trace analysis in nuclear materials can vary drastically from sub ppm for elements having high neutron absorption cross-section (such as B,

Cd, etc.) to a few hundreds ppm for elements such as Fe, Cr, Ni, W, etc. Hence, standards also need to be prepared accordingly using a wide linear dynamic range. In cases where the standards increase in concentration from low to high, it was observed that the total impurity content in the higher standards reached a level marginally lower than that of the carrier. Thus, other impurities can also affect the analyte intensity by influencing electrode temperature and arc parameters such as arc temperature and electron number density. Hence, it was decided to prepare the standards in such a manner that the concentrations for half of the analytes increased from standard-1 to standard-8, while for the other half, it increased from standard-8 to standard-1, resulting in almost uniform concentration levels of impurities in the set of standards. This necessitated preparation of two master impurity mixtures of MIM-1 and MIM-2. Appropriate amounts of the elements (most of them are in oxide form) were mixed with ZrO_2 to make the two different master impurity mixtures. MIM-1 contained the elements B, Be, Sn, Mn, Cu, Na, Ni, Ca, Cr, Fe, Zn, Mo, and Si, while MIM-2 contained Cd, Li, In, Sb, Mg, V, Co, Pb, Ti, Al, and W. AgCl, SrF_2 , and Pd in the form of $(\text{NH}_4)_2\text{PdCl}_4$ were ground together in 4:1:0.003 proportions to make a homogeneous carrier-internal standard mixture. The choice and proportion of the carrier combination was based on our earlier reported work on the choice of suitable carriers and their proportion in ZrO_2 (20). Martel (21) reported that the intensity ratios for different elements remain independent of particle size when the particle size is less than 75 μm , and this can be ensured by grinding all the mixtures for at least 30 minutes. Thus, all grindings were carried out for at least 30 minutes to achieve a homogeneous mixture and particles having a size less than 75 μm . A set of eight standards hav-

ing intermediate concentrations of trace elements in the analytical range, each in 5-g lots, was prepared by thoroughly grinding the graded amounts of MIM-1 and MIM-2, an amount corresponding to 5% of carrier-internal standard mixture, and the balance amount of ZrO_2 . The two mixtures, MIM-1 and MIM-2, were added in reverse direction to their respective standards. Similarly, a blank standard was prepared by grinding an appropriate amount of 5% of carrier-internal standard mixture and the balance amount of ZrO_2 .

Procedure

For each element, a minimum of three sensitive or prominent lines were chosen for arcing. Prior to arcing, the sample/standard of a known amount (100 mg) was weighed and transferred to each carrier distillation electrode. Subsequently, the electrodes were heated under an infrared lamp to remove possible traces of moisture. This was followed by tapping the electrodes for even distribution of the charge. A vent hole was made vertically through the center of the charge for smooth release of the gases generated during arcing in the electrode. To optimize the current setting during arcing, standard-4 was arced and the intensity of the elements was monitored at different settings (10, 12.5, 15, and 18 amps). The value of 12.5 A was found to be optimum beyond which the intensity was found to be saturated. All measurements were carried out in quadruplicate for each standard at the optimized conditions. Analytical data in the form of intensity ratio (I.R.), corresponding to the ratio of intensity of the concerned analytical line with that of Pd (363.470 nm), was followed throughout the investigations. Calibration data obtained after arcing the nine standards were fitted to get appropriate working curves by plotting intensity ratio (I.R.) vs.

concentration for all analyte lines of interest. Using the standardized parameters, several synthetic samples were analyzed to obtain their elemental concentrations. Details of the experimental conditions are given in Table I.

RESULT AND DISCUSSION

For optimization of the current, the intensity ratio (I.R) vs. current was plotted for the elements. All elements behave similarly and the typical behavior of intensity as a function of arc current is shown in Figure 1a and Figure 1b for B (249.77 nm line) and V (292.40 nm line), respectively. With an increase in current from 10 A to 12.5 A, the intensity was found to increase drastically. Subsequently, the increase was marginal and later showed saturation. Based on these results, it was decided to use 12.5 A as the current setting for further studies.

A CCD-based direct reading spectrometer is known to provide a wider choice of analytical lines and analytes in comparison to a photomultiplier tube (PMT)-based polychromator. Further, the CCD array contains detectors of micron size dimensions, while even miniaturized PMTs are much larger in dimension, thus a CCD-based spec-

TABLE I
Experimental Parameters
for the Determination of
Common Metals by D.C. Arc-AES

Standard/sample charge on electrode	100 mg
Arc current (D.C.)	12.5 A
Arc gap	4 mm
Pre-burn time	10 s
Exposure time	30 s

trimeter has the capability of using closely spaced analytical lines. In order to exploit these advantages of a CCD-based spectrometer, a large number of analytical lines were chosen for each analyte investigated (a minimum of three analytical lines per analyte) to evaluate their analytical performance. Final selection of the analytical lines for each analyte was based on the criteria such as a large analytical range, lower level of detection, sensitivity, and freedom from spectral interference from concomitant analytes and matrix. In a few cases, the most sensitive analytical lines of the analyte were not selected because of either Zr/plasma interference or concomitant analyte interference.

With a concentration of analyte elements grouped in two sub-groups having reverse gradation in

the set of standards, it is possible to understand the occurrence of concomitant analyte interference, in particular for the cases where interfered and interfering elements belong to two different sub-groups. If the spectral interference arises from the analytes belonging to separate sub-groups at lowest standards for the interfered analyte, the concentration of the interfering element is highest. With an increase in concentration of the analyte, the effect goes to reducing due to a decreasing concentration of the interfering element. In the matrix blank, all elements are almost absent depending on the purity of the matrix. Analytical lines can also have spectral interference from either small amounts of the Zr matrix being carried away in the plasma or from molecular bands such as C₂, CO, CN, etc., arising from the graphite electrodes used for generation of the arc plasma in an air atmosphere. In such a case, the interference will be observed in the complete set of standards, including the blank standard.

Fig. 1. Intensity ratio (I.R) vs. current curve for B 249.77 nm (1a) and V 292.40 nm (1b).

Zr/Arc Plasma Interference

Figure 2(a) shows I.R. variation with the concentration observed for the analytical lines of Mg at 280.270 nm and 285.213 nm. As seen from the figure, good gradation in I.R. with increasing concentration is observed for the analytical lines at 280.270 nm and 279.553 nm. Further, the I.R. value for the standard "blank" is well below that of the lowest standard containing Mg at 2.5 ppm. These observations suggest that there is no significant blank contribution of Mg in high purity ZrO_2 . However, almost no gradation is observed for the analytical line at 285.213 nm with a very high I.R. value for the "blank" standard, indicating that the analytical line is probably suffering from spectral interference from either Zr/plasma. As the emission line for Zr at 285.197 nm is reported, it is

inferred that the 285.213 nm analytical line of Mg is interfered by Zr. Figure 2(b) illustrates a similar behavior for Ca, where observations of no gradation and high I.R. for the blank standard were noted for the analytical lines at 396.847 nm and 393.366 nm. Good gradation and a negligible I.R. value for the standard blank was observed for the Ca analytical line at 317.933 nm. These observations suggest the existence of Zr/plasma interference in the former analytical lines of Ca. A similar behavior was observed for Pb (357.273 nm), Ni (341.476 nm), and Ti (323.452 nm) in comparison to their corresponding other analytical lines for each of these elements. This suggests an occurrence of Zr interference due to the presence of the Zr emission lines of 357.247 nm, 341.466 nm, and 323.412 nm, respectively.

Concomitant Interference

Figure 2(c) shows the variation of interference ratio (I.R.) with concentration, observed for Sb at 231.147 nm and 252.852 nm. This figure shows good gradation of I.R. with an increase in concentration at 231.147 nm and that the I.R. value for the standard "blank" is well below that of the lowest standard for Sb. This indicated that there is no significant blank contribution of Sb in high purity ZrO_2 . However, it was observed that for the Sb analytical line at 252.852 nm, even though the I.R. for the blank standard was lowest, a reverse trend in I.R. versus concentration was observed. This was followed by an increase in I.R. with increasing concentrations from 40 ppm to 200 ppm. The observed trend in I.R. as a function of concentration

Fig. 2. Intensity ratio (I.R.) vs. concentration (ppm) curve for Mg (2a), Ca (2b), and Sb (2c).

indicates the possibility of a concomitant spectral interference from the element whose concentration has a reverse gradation. It was noted from the MIT (Massachusetts Institute of Technology, MA, USA) wavelength tables (22) that Sb (252.852 nm) suffers a spectral interference from Si (252.851 nm) and in the standards prepared in the present work. The Si concentration decreases with increasing Sb concentrations, which can explain the observed changes in the I.R. with concentration. A similar behavior of a possible spectral interference from the concomitant analytes was found for only a few other elements. The interfered analytical lines, elements, and interfering elements with their wavelength position are included in Table II.

Table III shows the data for 24 elements regarding their suitable analytical lines, estimation range, and lower limit of detection. As

TABLE II
Mutual Spectral Interference in Concomitant Analytes

Element	Analytical Line (nm)	Interfering Line (nm)
Al	309.271	Na 309.273
Cd	226.502	Ni 226.446
	361.051	Ni 361.046 and Mn 361.030
Fe	238.204	In 238.263
In	325.609	Mn 325.614
Mn	260.569	Ti 260.515
Si	288.158	Al 288.145
Sn	270.651	W 270.658 and Al 270.644
Ti	336.121	Ni 336.156
V	292.402	Na 292.439
Zn	213.856	Co 213.897
	202.613	W 202.608

seen from this table, the lower limit of detection for B and Be is as low as 0.05 ppm, while for other elements the best lower limit of detection is in the range of 0.2-20.0 ppm. Table IV lists the analytical results of the earlier reports (6,7) using the photographic plate method, the direct reading spectrometric method with PMT detection, and the current CCD-based spectrometric method. It can be seen that for the majority of the elements a lower detection limit is achieved with the present CCD-based detection method in comparison to the PMT method.

The reproducibility of the working curves for the analyte elements of interest was investigated by repeated arcing of the set of standards on different dates. In general it was observed that there was a very small deviation in these calibration curves within the different sets of arcings for a given analytical line. Figures 3a-3f show the calibration curves for B at 249.77 nm, Fe at 275.57 nm, Pb at 261.41 nm, In at 451.13 nm, Co at 228.616 nm, and Cr at 267.716 nm where the I.R. is plotted against the concentration range of the analyte. These findings suggest that the day-to-day

TABLE III
Estimation Ranges and Detection Limits of Analytes

Element	Analytical Line (nm)	Conc. Range (ppm)	Lower DL (ppm)	Element	Analytical Line (nm)	Conc. Range (ppm)	Lower DL (ppm)
Al	308.215	5-500	5	B	249.773	0.05-10	0.05
	309.271				249.677		
Be	313.042	0.05-10	0.05	Ca	317.933	2.5-250	2.5
	249.473	0.2-10	0.2	Co	228.616	2.5-250	2.5
Cd	214.438	0.2-10	0.2		230.786		
	228.802	0.5-10	0.5	Cu	219.958	1-200	1
Cr	205.618	2.5-250	2.5	Fe	261.187	5-1000	5
	283.563				275.573		
In	451.131	1-100	1	Li	323.261	1-200	1
Mg	280.27	2.5-250	2.5	Mn	257.611	1-200	1
Mo	281.615	5-1000	5		259.373		
	204.664			Na	589.592	4-200	4
Ni	221.648	2.5-250	2.5	Pb	261.418	2.5-250	2.5
	231.604			Sn	242.170	0.5-100	0.5
Sb	217.581	1-100	1		242.949		
	231.147			Si	288.158	20-4000	20
V	311.071	2.5-250	2.5		251.612	80-2000	80
	292.464			Ti	334.941	20-500	20
Zn	334.502	5-1000	5	W	240.558	20-4000	20
	206.200	5-1000	5				

DL= Detection Limit.

TABLE IV
Comparison of the Analytical Results
With Earlier Work Using Different Method

Elements	Estimation Range (ppm)		
	Present Work	Photographic Plate Method (6)	Direct Reading Spectrometric Method (7)
Al	5-500	20-500	20-250
B	0.05-10	0.1-20	0.2-0.8
Be	0.05-10	Not reported	Not reported
Ca	2.5-250	10-100	Not reported
Cd	0.2-10	0.1-10	0.1-0.5
Co	2.5-250	1.0-100	5-100
Cu	1-200	1-200	5-8000
Fe	5-1000	100-1000	200-2000
In	1-100	Not reported	Not reported
Li	1-200	10-50	Not reported
Mg	2.5-250	8-200	10-40
Mn	1-200	2-200	5-25
Mo	5-1000	Not reported	Not reported
Na	4-200	Not reported	Not reported
Ni	2.5-250	10-500	20-1000
Pb	2.5-250	10-200	Not reported
Sb	1-100	Not reported	Not reported
Sn	0.5-100	10-200	10-20
Si	20-4000	10-1000	40-300
Ti	20-500	20-200	20-100
V	2.5-250	20-300	20-50
W	20-4000	10-1000	Not reported
Zn	5-1000	Not reported	Not reported

variation can be taken care of using a two-point standardization. Since a certified reference material for the analytes investigated in the present work was not readily available, a synthetic spiked sample was prepared and used for method validation. Table V shows the results obtained for the analysis of the synthetic sample. The results were found to be in good agreement with the expected values; in addition, there is little deviation in the result for both sets of arcings, which confirms reproducibility of the results.

CONCLUSION

An analytical method was developed with a charge coupled device (CCD)-based spectrometer for the regular determination of 24 elements (Al, B, Be, Ca, Cd, Co, Cr, Cu, Fe, In, Li, Mg, Mn, Mo, Na, Ni, Pb, Sb, Si, Sn, Ti, V, W, and Zn) in a refractory ZrO_2 matrix using the direct current (D.C.) arc carrier distillation technique resulting in a RSD better than 25%. The investigations have shown that the CCD-based spectrometer can be easily adapted for trace analysis with detection limits in the sub-ppm/ppm range of 0.05–20.0 ppm. It

enabled the simultaneous measurements of the closely placed analytical lines such as 249.473 nm for Be, and 249.677 nm and 249.773 nm for B. It also enabled monitoring a wider choice of analytical lines which helps to identify the best analytical lines having freedom from spectral interference from the matrix/concomitant elements. The analytical method developed in the present work was validated by carrying out spiked synthetic sample analysis.

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Fig. 3. Calibration curves of B (3a), Fe (3b), Pb (3c), In (3d), Co (3e), and Cr (3f).

TABLE V
Analysis of Synthetic Samples

Element	Analytical Line (nm)	Amount Added (ppm)	Amount Determined (ppm)		Element	Analytical Line (nm)	Amount Added (ppm)	Amount Determined (ppm)	
			Set of Arcing 1st	2nd				Set of Arcing 1st	2nd
Al	308.215	100	165	155	B	249.773	0.5	0.64	0.56
	309.271		176	157		249.677		0.44	0.47
Be	313.042	0.5	0.49	0.57	Ca	317.933	25	19.67	17.20
	249.473		0.51	0.47		228.616		46.13	45.90
Cd	214.438	1	0.98	1.18	Co	230.786	50	45.10	40.92
	228.802		1.21	1.26		219.958		9.30	7.81
Cr	205.618	25	22.84	23.45	In	451.131	10	11.05	11.32
	283.563		24.52	19.89		323.261		17.99	16.45
Fe	275.573	50	38.32	33.51	Mg	280.270	50	53.21	46.62
	261.187		38.94	32.72		281.615		46.22	42.88
Mn	257.611	10	16.85	10.41	Mo	204.664	50	35.49	31.04
	259.373		14.18	10.91		589.592		30.54	31.13
Ni	221.648	25	20.26	21.56	Na	589.592	20	30.54	31.13
	231.604		22.28	19.39		589.592		30.54	31.13
Sb	217.581	20	19.20	18.28	Pb	261.418	50	48.75	50.5
	231.147		16.73	15.64		242.170		4.83	4.19
Ti	334.941	100	168.33	198.77	Sn	242.949	5	5.11	3.85
	311.071		40.18	39.48		242.949		5.11	3.85
V	292.464	50	53.55	48.41	Si	288.158	200	253.17	249.83
						251.612		269.33	292.66
					W	240.558	400	459.04	470.10
						334.502		53.70	51.68
					Zn	206.200	50	59.39	62.59

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