Exploration of CCD-based ICP-AES for Studying Spectral Interferences of Uranium on Other Analytes

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ABSTRACT

Even though inductively coupled plasma atomic emission spectrometry (ICP-AES) is a simultaneous multi-elemental analytical technique with improved precision and detection limits and is routinely used for the determination of trace metallic constituents in nuclear fuels and associated materials, it suffers from severe spectral interferences. Due to the multi-electronic nature, uranium has line-rich emission spectra which interfere with the determination of trace metallic constituents in a uranium matrix. Therefore, chemical separation of uranium, followed by the determination of trace metallic impurities in the raffinate by ICP-AES, is generally the adopted procedure by the nuclear industry. Since good resolution is one of the governing factors for reducing spectral interferences, the analysis depends on the resolution of the instrument and the availability of alternative analytical lines for

minimizing these spectral interferences. Since a charge-coupled detector (CCD) consists of an array of pixels, it has more options in choosing alternative analytical lines in comparison to the photomultiplier tube-based ICP-AES using a polychromator. On the other hand, a monochromator has the option of choosing alternative lines but, unfortunately, requires a great deal of time to scan all the elements and also requires a large sample size. Therefore, an attempt was made to study the spectral interferences of uranium on different analytical lines for 16 analytes (Ag, B, Ca, Cd, Co, Cr, Cu, Dy, Eu, Fe, Gd, Mn, Na, Ni, Sm, Zn) together with evaluating the correction factors (wherever necessary) using the CCD-based ICP-AES technique. The sensitivity and detection limits of the analytical channels of the elements in the presence of a uranium matrix were calculated. The present study also identified suitable analytical lines of uranium and measured their detection limits.

mation of analytes due to its spectral interferences. Thus, for the determination of trace metallic constituents in a uranium matrix, it either has to be separated physically from the trace metals using the d.c. arc carrier distillation technique or chemical separation, followed by ICP-AES analysis of the raffinate.

The d.c. arc technique, even though the contamination from sample handling is less, suffers from poor precision (10-13). With ICP-AES using chemical separation of the major matrix components, the chances of contamination during sample preparation is high but the detection limits and precision are much better. In ICP-AES it is also required to get suitable amounts of extractant and feed conditioning so that there is preferential extraction of the major matrices without loss of the analytes even at trace levels (2,3,7,8,14-21). Therefore, it is required to develop an ICP-AES-based method where sample handling is minimized and the analytical performance of the method (precision, detection limits, linear dynamic range, sensitivity, etc.) is superior.

In the present study, a charge-coupled device (CCD) detector was used to study the spectral interferences of uranium on other analytes. Due to the design of array pixels in the CCD-based detector, the choice of alternative interference-free analytical lines is similar to a photomultiplier-based detector system. Suitable analytical lines for 16 analytes were chosen and the tolerance limit of uranium on different analytical channels of the analytes were determined, together with the

INTRODUCTION

To achieve the desired burn-up and maximize the life span of the fuel in the nuclear reactor, it has to go through stringent physical and chemical quality control and quality assurance measurements at each step of the fuel fabrication. Trace metallic impurity analysis is one of the prime steps for chemical quality control of nuclear fuels and associated materials (1-21). Due to

the high neutron absorption cross-section of rare earth elements, they have stringent specification limits, while the presence of other elements beyond the specification limits changes the metallurgical properties of fuel materials. Uranium metal and in oxide form is one of the key materials used in the nuclear industry. For that reason, it is required to develop an analytical method for the determination of trace metallic constituents in a uranium matrix. Due to its line-rich emission spectra, the presence of uranium can lead to the over-esti-

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detection limits and the sensitivity of the analytical lines in the presence of the uranium matrix.

A suitable analytical method was also developed for the determination of uranium by ICP-AES. Four different analytical lines of uranium (385.958 nm, 409.014 nm, 367.007 nm, 279.394 nm) were identified and calibration curves established up to 1000 µg/mL of uranium. The detection limits and sensitivity of the above-mentioned analytical lines of uranium were evaluated. It was found that the detection limits of 409.014 nm and 385.958 nm were comparable (0.09 µg/mL and 0.02 µg/mL, respectively), while those of 367.007 nm and 279.394 nm were a magnitude of order higher (0.129 µg/mL and 0.341 µg/mL, respectively).

EXPERIMENTAL

Instrumentation

The analytical experiments were carried out using a computer-controlled, high resolution, simultaneous atomic emission spectrometer (Spectro Arcos, Germany) with ICP and DC arc as the excitation sources and a charge-coupled device (CCD) as the detector. The instrumental specifications and experimental parameters are summarized in Table I. The detector system, consisting of linear arrays of the CCD detectors (3648 pixels/array), thermally stabilized together with an optical system, provides the ability to choose alternative analytical lines. In Figure 1, the cross-sectional view of the detector system is shown.

Reagents and Standard Solutions

Rare earth element (REE) and other common impurity standard solutions were prepared from CertiPUR® ICP multi-standard solutions (E. Merck, Darmstadt, Germany) by proper dilution with 0.5 M HNO₃. Suprapur® HNO₃ and

TABLE I
Instrumental Operating Conditions of ICP-AES

Instrumental Specifications	
Optical design	Paschen-Runge mounting, circular design
Grating	Holographic
Groove density	1800 grooves/mm (1), 3600 grooves/mm (2)
Wavelength range	130–800 nm
Entrance slit width	15 microns
Resolution (FWHM)	0.01 nm from 130–450nm 0.02 nm from 450–800nm
Thermal regulation	Controlled to 30 ± 1 °C
Frequency	27.12 MHz
Pump	Dual channel peristaltic pump
Detector	Linear arrays of CCD (3648 pixels/array)
Nebulizer	Concentric nebulizer with cyclonic spray chamber
ICP torch	Demountable, radial view
Operating Conditions	
Coolant flow	6 L/min
Auxiliary flow	0.6 L/min
Total measurement time	28 s
Pump speed	30 rpm
RF power out put	0.8–1.5 kW
Input power	230 V AC

Fig. 1. Cross-sectional view of the array of pixels in CCD-based detector.

quartz double-distilled water (E. Merck, Darmstadt, Germany) were used for all of the experiments. Multi-point standardization was carried out using 0.5M HNO₃ for the blank and 0.05–1000 µg/mL of the corresponding elemental standard for each analytical line after proper peak search. For the preparation of the uranium stock solution, high purity uranium oxide was used. From the stock solutions, different concentrations of U in the range of 0.05–1000 µg/mL were prepared using proper dilutions with 0.5M HNO₃. Blank correction was carried out using 0.5M HNO₃.

To understand the spectral interference of uranium on other analytes, pure uranium solutions of different concentrations were used, and the contributions at each analytical channel were monitored.

RESULTS AND DISCUSSION

ICP-AES Determination of Uranium

Uranium is a multi-electronic element having line-rich emission spectra. Due to the availability of large numbers of emission lines, the sensitivity of the lines was found to be less and the detection limits poorer with a PMT-based detection system. Exploring the superior analytical performances of CCD-based detector systems, an analytical method was developed for the determination of uranium by ICP-AES. The study includes identification of suitable analytical lines. Four emission lines of uranium were chosen (385.958 nm, 409.014 nm, 367.007 nm, and 279.394 nm), and Table II lists the determination of uranium using these analytical lines. The calibration curves were established using U- blank, U-10 µg/mL, U-20 µg/mL, and U-50 µg/mL solutions. The solutions of known concentrations of U (i.e., 500 µg/mL, 1000 µg/mL, etc.)

were analyzed using the developed methodology and established calibration curves. The solution containing 1000 µg/mL of U and using the developed method showed a value of 1000 µg/mL with a relative standard deviation of 5%. It can, therefore, be concluded that up to 1000 µg/mL the calibration curves maintain linearity. The sensitivity and detection limits of uranium using the above-mentioned analytical lines were evaluated using the slopes of the calibration curves and the concentrations equivalent to the intensity equal to the average of the blank value (15 readings), plus three times the standard deviation of the above measurements, i.e., $x + 3\sigma$. The detection limit of uranium using the 385.958 nm emission line was found to be the best (0.02 µg/mL), followed by 409.014 nm, while the detection limits using the 367.007 nm and 279.394 nm lines were an order of magnitude poorer as shown in Table III.

Spectral Interferences of Uranium on the Analytes

Interferences may originate at various steps of the ICP-AES sample introduction and excitation process, which convert the sample into atomic radiation. Among various kinds of interferences, spectral interference is significant as far as ICP-AES is concerned. Usually for the determination of a particular analyte at the trace levels in a sample, the most sensitive line is cho-

TABLE II
Determination of Uranium by ICP-AES

Actual U Conc.	U Concentration 385.958 nm	U Concentration 409.041 nm	U Concentration 367.007 nm	U Concentration 279.394 nm
(mg/L)	(mg/L)	(mg/L)	(mg/L)	(mg/L)
Blank	BDL	BDL	BDL	BDL
0.05	0.06 ± 0.001	BDL	BDL	BDL
0.1	0.12 ± 0.002	0.13 ± 0.002	0.14 ± 0.001	BDL
0.5	0.5 ± 0.01	0.54 ± 0.004	0.5 ± 0.004	0.52 ± 0.005
1	1.16 ± 0.03	1.18 ± 0.04	1.11 ± 0.04	1.12 ± 0.03
2	2.07 ± 0.01	2.02 ± 0.03	2.09 ± 0.03	2.12 ± 0.07
5	5.3 ± 0.04	5.2 ± 0.07	5.1 ± 0.07	5.02 ± 0.03
10	10.1 ± 0.03	10.1 ± 0.04	10.3 ± 0.03	10.3 ± 0.2
20	20.2 ± 0.3	20.6 ± 0.3	20.8 ± 0.4	20.2 ± 0.6
50	50.3 ± 0.9	51.5 ± 0.4	50.6 ± 0.6	51.1 ± 1
100	101 ± 2	100 ± 1	102 ± 3	101 ± 7
200	204 ± 5	208 ± 5	201 ± 6	207 ± 5
500	501 ± 3	503 ± 9	502 ± 8	500 ± 11
1000	1016 ± 15	1009 ± 11	1019 ± 21	1004 ± 14

BDL = below detection limit.

TABLE III
Determination of Detection Limits of Different Analytical Lines of U

Analytical Line (nm)	Detection Limit (mg/L)
279.394	0.341
367.007	0.1219
385.958	0.02
409.014	0.09

sen, while if the particular line is overlapped by some other interfering line, an alternate line needs to be selected which may be less sensitive and have poorer detection limits. Improvements in the resolution of the instrument may help to resolve the lines only if spectral overlap is responsible for the interference. If overlap of the lines is from the line-broadening process occurring in the plasma, improvements in instrumental resolution will be insufficient. Therefore, alternate emission lines need to be investigated for their analytical applicability. In some cases it might be that only one analytical line is suitable for analytical purposes. In such cases, appropriate correction factors need to be evaluated.

In the present study, due to the advancement of detector systems, i.e., array of pixels of CCD detectors, the option of choosing an analytical line is better compared to PMT-based detector systems. A detailed study was carried out to choose appropriate emission lines for the trace determination of 16 trace metallic impurities in the presence of a uranium matrix.

Since B, Cd, the rare earth elements, and Ag have stringent specification limits, careful control of these constituents at each step of fuel fabrication is essential. Therefore, it is required to study the spectral interferences of U on the analytical lines of these elements to avoid their overestimation, which is detrimental in qualifying the life span of the fuel rods in the reactor.

Table IV summarizes the contribution of different U concentrations on the different analytical lines of critical elements (Ag, Cd, Eu, Sm, Gd, and Dy). In all cases, more than one analytical line was chosen. Since all the analytical lines of a particular element do not suffer from spectral interferences of U to the same extent, the analytical

line having lowest contribution from U can be taken as the most suitable analytical line of that particular element in the presence of the U matrix. Table IV shows that the contributions of U on different analytical lines of the analytes vary linearly with the concentration of U. The least interfered analytical lines of the critical elements in the presence of uranium are 338.289 nm for Ag, 182.641 nm for B, 214.438 nm for Cd, 420.505 nm for Eu, 336.223 nm for Gd, 428.079 nm for Sm, and 394.468 nm for Dy. Similarly, Table V summarizes the effect of U concentration on different analytical lines of the common contaminants. A linear increase of U contribution was observed for all the analytical channels with an increase in U concentration. From Table V the least interfered analytical lines of common metallics were as follows: 588.995 nm for Na, 393.366 nm for Ca, 238.204 nm for Fe, 284.325 nm for Cr, 269.569 nm for Mn, 221.648 nm for Ni, 224.700 nm for Cu, 228.616 nm for Co, and 213.856 nm for Zn.

The detection limits and the sensitivity for the above-mentioned analytical lines are listed in Table VI. The detection limits were calculated on the basis of the concentration corresponding to $\langle x \rangle + 3\sigma$, where $\langle x \rangle$ is the average value (15 readings) of the blank intensity, and σ is the standard deviation of the above measurements. The sensitivity was calculated based on the emission intensity counts per unit concentration (mg/L), which is the slope of the calibration curve. Best detection limits can be achieved in the presence of U by using the following analytical lines of the analytes: 338.289 nm for Ag, 182.641 nm for B, 422.673 nm for Ca, 206.200 nm for Zn, 228.616 nm for Co, 284.325 nm for Cr, 324.754 nm for Cu, 364.540 nm for Dy, 381.967 nm for Eu, 261.187 nm for Fe, 359.260 nm for Sm, 221.648 nm for Ni, 588.995 nm for Na, and 257.611 nm for Mn.

In some situations, the analytical line with lowest detection limit may not be the line with the least spectral interference from U. As indicated above, optimization would then be required to select the best analytical line of a particular element together with acceptable detection limits and minimum spectral interferences.

CONCLUSION

A systematic study was carried out to understand the spectral interferences of uranium on 16 analytes (Ag, B, Ca, Cd, Co, Cr, Cu, Dy, Eu, Fe, Gd, Mn, Na, Ni, Sm, Zn). For all the analytical lines, the tolerable limits of uranium were evaluated. The detection limits of the analytes were calculated in the presence of the uranium matrix. An analytical method was developed for the determination of uranium by inductively coupled plasma atomic emission spectrometry (ICP-AES), which includes the identification of the emission lines, establishing the calibration curves, and evaluating the sensitivity and detection limits of uranium. For uranium, the 385.958 nm and 409.014 nm emission lines were found to be equally sensitive with the detection limits of 0.02 $\mu\text{g/mL}$ and 0.09 $\mu\text{g/mL}$, respectively. Both lines can be used for the determination of uranium by ICP-AES.

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TABLE IV
Spectral Interference of U on the Critical Elements

U Conc.	B 249.773	B 249.677	B 182.641	Cd 214.438	Cd 226.502	Cd 361.051	Sm 359.260	Sm 428.079	Dy 353.170	Dy 394.468	Dy 364.540
mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L
blnk	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
0.05	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
0.1	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL
0.5	BDL	BDL	BDL	BDL	BDL	0.189	BDL	BDL	BDL	BDL	BDL
1	BDL	BDL	BDL	BDL	BDL	0.271	BDL	BDL	BDL	BDL	BDL
2	BDL	BDL	BDL	BDL	BDL	0.423	BDL	BDL	BDL	BDL	0.01
5	BDL	BDL	BDL	BDL	BDL	0.873	0.015	BDL	BDL	0.005	0.01
10	BDL	BDL	BDL	BDL	BDL	1.709	0.026	0.031	0.004	0.014	0.02
20	BDL	BDL	BDL	BDL	BDL	3.435	0.051	0.044	0.008	0.028	0.03
50	BDL	BDL	BDL	0.005	0.009	8.789	0.13	0.103	0.023	0.074	0.08
100	0.009	0.028	BDL	0.008	0.016	17.557	0.264	0.192	0.047	0.148	0.17
200	0.03	0.064	BDL	0.014	0.028	34.059	0.522	0.372	0.094	0.293	0.33
500	0.089	0.162	BDL	0.031	0.056	84.656	1.296	0.799	0.238	0.722	0.77
1000	0.188	0.343	0.014	0.052	0.116	167.646	2.439	1.622	0.443	1.384	1.54

U Conc.	Eu 381.967	Eu 393.048	Eu 420.505	Ag 328.068	Ag 338.289	Ag 224.641	Gd 335.047	Gd 336.223	Gd 335.862
mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L
blnk	BDL	BDL	0.002	< 0.001	< 0.002	< 0.123	BDL	BDL	BDL
0.05	BDL	BDL	0.002	< 0.001	< 0.003	< 0.112	BDL	BDL	BDL
0.1	BDL	BDL	0.002	< 0.002	< 0.002	< 0.140	BDL	BDL	BDL
0.5	BDL	BDL	0.002	< 0.002	0.004	< 0.135	BDL	BDL	BDL
1	BDL	BDL	0.002	< 0.001	< 0.004	< 0.162	BDL	BDL	BDL
2	BDL	BDL	0.002	< 0.003	0.004	< 0.143	BDL	BDL	BDL
5	0.001	BDL	0.003	0.006	0.008	< 0.147	0.008	0.007	0.008
10	0.003	BDL	0.004	0.011	0.014	< 0.176	0.016	0.011	0.016
20	0.005	BDL	0.005	0.021	0.023	< 0.240	0.03	0.019	0.036
50	0.012	0.017	0.01	0.055	0.056	0.35	0.075	0.044	0.095
100	0.024	0.05	0.017	0.109	0.109	0.613	0.146	0.083	0.191
200	0.047	0.11	0.031	0.217	0.211	1.062	0.289	0.162	0.384
500	0.107	0.3	0.073	0.523	0.518	2.346	0.709	0.393	0.945
1000	0.215	0.609	0.133	1.019	0.973	3.925	1.357	0.741	1.815

TABLE V
Spectral Intereference of U on Common Metallic Constituents

U Conc.	Cr 205.618	Cr 284.325	Mn 257.611	Mn 260.569	Ni 231.604	Ni 221.648	Ni 227.021	Cu 224.700	Cu 219.958
mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L
blnk	BDL	BDL	0.002	BDL	BDL	BDL	BDL	BDL	BDL
0.05	BDL	BDL	0.003	BDL	BDL	BDL	BDL	BDL	BDL
0.1	BDL	BDL	0.003	BDL	BDL	BDL	BDL	BDL	BDL
0.5	BDL	BDL	0.002	BDL	BDL	BDL	BDL	BDL	BDL
1	BDL	BDL	0.003	BDL	BDL	BDL	BDL	BDL	BDL
2	BDL	0.007	0.002	BDL	BDL	BDL	BDL	BDL	BDL
5	BDL	0.01	0.007	BDL	BDL	BDL	BDL	BDL	BDL
10	BDL	0.014	0.005	BDL	BDL	BDL	BDL	BDL	BDL
20	BDL	0.023	0.005	BDL	0.012	BDL	BDL	BDL	0.022
50	0.162	0.05	0.012	0.01	0.023	0.016	BDL	0.015	0.035
100	0.274	0.099	0.022	0.017	0.041	0.03	0.103	0.026	0.061
200	0.442	0.192	0.035	0.026	0.073	0.052	0.166	0.037	0.096
500	0.968	0.479	0.084	0.061	0.168	0.118	0.359	0.079	0.211
1000	1.66	0.899	0.163	0.123	0.33	0.227	0.612	0.141	0.357

U conc	Co 228.616	Co 230.786	Zn 213.856	Zn 206.200	Na 589.592	Na 588.995	Ca 396.847	Ca 393.366	Fe 239.562	Fe 238.204
mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L
blnk	BDL	BDL	BDL	BDL	0.038	0.037	0.224	0.221	0.071	0.065
0.05	BDL	BDL	BDL	BDL	0.067	0.066	0.342	0.341	0.056	0.052
0.1	BDL	BDL	BDL	BDL	0.093	0.089	0.521	0.518	0.072	0.067
0.5	BDL	BDL	BDL	BDL	0.086	0.084	0.262	0.26	0.037	0.035
1	BDL	BDL	BDL	BDL	0.083	0.08	0.414	0.41	0.07	0.063
2	BDL	BDL	BDL	BDL	0.035	0.032	0.198	0.191	0.027	0.025
5	BDL	BDL	BDL	BDL	0.131	0.124	0.883	0.859	0.145	0.134
10	BDL	BDL	BDL	BDL	0.105	0.1	0.479	0.474	0.076	0.067
20	0.007	0.01	BDL	BDL	0.047	0.046	0.149	0.142	0.03	0.027
50	0.014	0.02	BDL	0.01	0.104	0.098	0.458	0.454	0.072	0.063
100	0.025	0.035	BDL	0.03	0.195	0.181	0.891	0.864	0.202	0.187
200	0.046	0.064	BDL	0.02	0.1	0.091	0.364	0.357	0.085	0.067
500	0.101	0.149	BDL	0.05	0.146	0.133	0.257	0.245	0.119	0.079
1000	0.205	0.293	0.032	0.09	0.306	0.277	0.516	0.491	0.24	0.177

TABLE VI
Determination of Sensitivity and Detection Limits of the Analytes

Element	Wavelength nm	Detection Limit mg/L	Sensitivity Counts mg/L	Element	Wavelength nm	Detection Limit mg/L	Sensitivity Counts mg/L
Ag	328.068	0.0036	5.93×10^{-6}	Eu	381.967	0.00034	1.95×10^{-6}
	338.289	0.0024	1.17×10^{-5}		420.505	0.0014	2.58×10^{-6}
B	182.641	0.0047	1.95×10^{-5}	Fe	239.562	0.00407	1.76×10^{-5}
	249.773	0.0051	1.255×10^{-5}		261.187	0.0025	2.59×10^{-5}
Ca	396.847	0.00147	2.07×10^{-7}	Na	588.995	0.0018	1.04×10^{-5}
	422.673	0.00133	1.44×10^{-5}		589.592	0.0025	1.92×10^{-5}
Cd	214.438	0.0036	1.29×10^{-5}	Mn	257.611	0.00069	2.39×10^{-6}
	226.502	0.00566	1.54×10^{-5}		259.373	0.00088	2.8×10^{-6}
Cr	283.563	0.005	1.42×10^{-5}	Ni	221.648	0.0077	2.02×10^{-5}
	284.325	0.0037	1.58×10^{-5}		231.604	0.0093	2.19×10^{-5}
Cu	324.754	0.0016	4.81×10^{-6}	Sm	359.26	0.0032	2.35×10^{-5}
	327.396	0.0048	8.81×10^{-6}		360.428	0.0045	6.18×10^{-5}
Dy	340.78	0.0016	2.35×10^{-5}	Co	228.616	0.0054	1.25×10^{-5}
	353.17	0.0016	4.67×10^{-6}		238.892	0.0056	1.49×10^{-5}
	364.54	0.0011	1.35×10^{-5}	Zn	202.613	0.016	9.5×10^{-6}
					206.2	0.006	4.18×10^{-5}

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