Studies on Spectral Interference of Neodymium on Analytes in Trace Metallic Impurity Analysis of Neodymium Matrix Using CCD-based ICP-AES

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INTRODUCTION

Lanthanides are electron-rich rare earth elements (REEs) and form an overlap contour of spectral lines which is the main source of error in atomic emission spectroscopy (1-5). REEs are widely used in areas of industry, agriculture, and modern technology, e.g., as a growth promoter fertilizers in agriculture, as a catalysts in oil refining, as additives in new ceramic materials and special steels, etc. (6-9). Neodymium is the raw material used in high-strength permanent magnets (Nd-B-Fe), making it less expensive than samarium-cobalt permanent magnets (10). Many investigators have used inductively coupled plasma atomic emission spectrometry (ICP-AES) as the powerful technique to determine REEs in geological samples (11-16). Capacitive coupled device (CCD)-based ICP-AES is a highly sensitive method with a long dynamic range (17-18), has the capability to determine even trace amounts of the analytes (19-20), and has been used for over a decade (21-22). It also proved to be a better method than other earlier methods like neutron activation analysis (23-24) and isotope dilution mass spectrometry (25). A high temperature plasma (6000–8000 K) is used as the excitation source which minimizes matrix effects, prevents the formation of polyatomic species during the excitation process, which in turn produces adequate sensitivity and increases the detection limits for many elements (26). Before the

ABSTRACT

Due to the emission-rich spectra of all rare earth elements, they are expected to interfere in the determination of analytes at trace levels, especially when any of them is present as the matrix. The present paper deals with the study on the spectral interference of neodymium on 28 different analytes, including some rare earth elements (Ag, B, Ba, Ca, Cd, Co, Cu, Fe, Mg, Na, Sr, Zn, Gd, Lu, Pb, In, Ce, La, Pr, Ga, Ni, Al, Ti, Dy, Bi, Li, Mn, and Cr). This involves identification of the least interfered analytical lines of these elements, determination of correction factors, the tolerance level of Nd, and the analytical performance of all the analytical lines of the elements in the presence of a Nd matrix. Since the extent of the spectral interference depends on the concentration of the interfering elements, it necessitates the development of ICP-AES-based methods for the determination of Nd which revealed that the Nd 401.225-nm line is the best analytical line for Nd.

Based on the above study, a simple and rapid methodology was developed for the determination of these analytes at trace levels in the presence of a Nd matrix without any chemical separation. The method was validated using synthetic samples prepared from commercially available standard reference materials of the individual elemental solutions.

advancements of multichannel detectors, photomultiplier tubes (PMT) were used as the detector with the major drawback of it being a single-channel detector which leads to a drastic waste of information, even when several detectors are set up in the polychromator. The multichannel detectors used with commercially available ICP-AES systems based on charge transfer technology, such as the 2-D CCD detector or an association of linear CCD arrays, currently provide acquisition of the entire UV-visible spectrum: full flexibility in analytical line selection, use of several lines of the same element to extend the dynamic range, use of a large number of lines of the same element to improve accuracy and to verify possible matrix effects or spectral interferences, qualitative analysis and fast diagnostic. In addition to true simultaneous measurements, it offers speed of analysis, time correlation between lines of different elements to improve repeatability by internal standardization, and time correlation between line and adjacent background intensities to improve the limit of detection and the limit of quantitation (27-30).

Using the CCD-based ICP-AES instrument, the spectral interference of neodymium was studied for 28 analytes: Ag, B, Ba, Ca, Cd, Co, Cu, Fe, Mg, Na, Sr, Zn, Gd, Lu, Pb, In, Ce, La, Pr, Ga, Ni, Al, Ti, Dy, Bi, Li, Mn, and Cr. A simple and rapid method was developed for the trace level determination of these analytes in the presence of a Nd matrix since they are difficult to separate from Nd due to their similar chemistry.

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EXPERIMENTAL

Instrumentation and Operating Conditions

The analysis was carried out using an ARCOS inductively coupled atomic emission spectrometer (ICP-AES) (Arcos Industries, Germany), with argon plasma as the excitation source and a capacitive coupled device (CCD) as the detector system. The instrumental specifications and optimized operating conditions are summarized in Table I (30). The cross-sectional view of the detector system is given elsewhere (29). The resolution of the present instrument was documented as 0.01 nm for the wavelength range of 130–450 nm, while

for 450–800 nm it was 0.02 nm. The resolution of the instrument plays an important role to resolve two closely spaced lines. The above resolution quoted by the manufacturer is one of the highest resolutions reported for CCD-based ICP-AES. The instrument with higher resolution can serve the purpose only when the lines are not resolved due to optical reasons only. But most of the time, even using the instrument with the highest possible resolution cannot resolve the overlapped emission lines. This case becomes more dominant when the interest is to determine the interfered analytes at trace levels in the presence of an interfering matrix.

TABLE I
Specifications and Operating Conditions of ICP-AES

Instrumental Specifications	
Optical design	Paschen-Runge mounting, circular design
Focal length	750 mm
Grating	Holographic
Groove density	1800 grooves/mm (1), 3600 grooves/mm (2)
Wave length range	130–800 nm
Entrance slit width	15 microns
Resolution (FWHM)	0.01 nm from 130–450 nm 0.02 nm from 450–800 nm
Thermal regulation	Controlled to 30 ± 1 °C
Frequency	27.12 MHz
Pump	Dual channel peristaltic pump (12 roller)
Detector	Linear arrays of CCD (3648 pixels/array)
Nebulizer	Cross flow nebulizer
ICP torch	Demountable, radial viewing
Operating Conditions	
Coolant flow	13 L/min
Auxiliary flow	0.8 L/min
Nebulizer gas flow	0.8 L/min
Total time of measurement	28 s
Pump speed	30 rpm
Nebulizer pressure	3 bars
Pressure on manometer	6 bars
RF power output	1.2 kW
Reflected power	< 10 W
Input power	230 V AC

Standard Solutions and Reagents

Standard solutions for all of the elements were prepared from the CertiPUR® ICP standard reference material solution of individual elements (E-Merck, Darmstadt, Germany). Suprapur® HNO₃ (E-Merck, Darmstadt, Germany) and quartz double-distilled water were used throughout the study. Multi-point standardizations were carried out using 0.5 M HNO₃ as the lower standard and 0.05–1000 mg/L of the corresponding elemental standard as the higher standard for each line after proper peak search, auto-attenuation, etc. For the analysis of each sample, 10 replicate measurements were carried out, while for the determination of the detection limits and sensitivity, 25 replicate measurements were made.

Methods

The method chosen for evaluation of the correction factor, contribution due to Nd in each different channel of each analyte, tolerance level along with the analytical performance (detection limit, sensitivity, linear dynamic range, and precision for each analyte) was studied by preparing a series of Nd solutions (commercially available standard reference material elemental solution from E-Merck, Darmstadt, Germany) of varying concentrations from 0.05 to 1000 mg/L. Using the idea of dependence of correction factor on concentration of the interfering element (Nd), it becomes easy to directly determine the interferent in the presence of other analytes by ICP-AES. The analytical performance for each identified line of Nd was evaluated.

The developed methodology for the direct determination of trace amounts of 28 analytes was validated using synthetic samples without chemical separation.

Fig. 1. Emission profiles of different analytical lines of Nd: (a) 401.225 nm, (b) 406.109 nm, (c) 417.731 nm, and (d) 430.358 nm.

RESULTS AND DISCUSSION

Determination of Neodymium by ICP-AES

For evaluating the contribution of Nd to different analytes, it is required that the correct amount be estimated in the sample. The correction factor which quantifies the contribution of Nd also depends on the concentration of Nd. In this study, an ICP-AES method was developed for the direct determination of Nd along with different analytes including some rare earths. Based on the analytical performance, four lines of Nd were selected: 401.225 nm, 406.109 nm, 417.731 nm, and 430.358 nm (see Figure 1 a-d). The calibration curves for these lines were established as shown in Figure 2. The detection limit of these four Nd lines followed the trend:

Fig. 2. Calibration curves for different analytical lines of Nd.

401.225 nm < 406.109 nm < 430.358 nm < 417.731 nm, while for sensitivity it followed the trend: 401.225 nm > 430.358 nm > 406.109 nm > 417.731 nm. The slope of the calibration curves was considered as the sensitivity of the

analytical lines, whereas the detection limits were calculated as the concentration equivalent to the intensity of the blank ($\bar{x}$) + 5 times the relative standard deviation for the blank (5σ). Synthetic samples containing Nd in the con-

centration range 0.05–1000 mg/L were analyzed using these analytical lines. The results shown in Table II revealed that all lines of Nd can be suitably used for the determination of Nd up to 1000 mg/L. Beyond 1000 mg/L, the experiment was not carried out because of the choking problem of the nebulizer system.

Spectral Interference of Nd on Other Analytes

Nd (ground state electronic configuration [Xe] 4f⁴, 5d⁰, 6s², and ⁵I₄), like other members of the lanthanide series, has emission line-rich spectra due to its multi-electronic nature and hence, may interfere in the analysis of other analytes most of which do not possess emission-rich spectra and, therefore, have limited lines for the analysis. The best lines for each analyte were chosen on the basis of spectral interference and analytical performance (detection limit, sensitivity, linear dynamic range, and precision).

Effect on Bi, Li, Mn, and Cr

Table III reveals the systematic spectral interference study of Nd on different analytical lines of Bi, Li, Mn, and Cr along with their analytical performance in the presence of a Nd matrix.

Bismuth (Bi)

Among the five lines of Bi (306.772 nm, 190.241 nm, 206.170 nm, 222.825 nm, and 223.061 nm), the 306.772-nm line was highly sensitive and was not interfered by Nd; hence, it can be selected for the determination of Bi in a Nd matrix.

Lithium (Li)

For the 670.780-nm line of Li, the detection limit was too low in comparison to the other three lines (274.118 nm, 323.261 nm, 460.289 nm), while the sensitivity was far too high and was the line free from interference of Nd matrix.

TABLE II
Determination of Nd by ICP-AES

Nd (mg/L)	401.225 nm (mg/L)	430.358 nm (mg/L)	406.109 nm (mg/L)	417.731 nm (mg/L)
1	0.98 ± 0.03	1.07 ± 0.05	0.97 ± 0.03	0.99 ± 0.03
5	5.10 ± 0.08	4.94 ± 0.07	5.09 ± 0.08	5.11 ± 0.08
10	10.1 ± 0.4	10.4 ± 0.4	9.5 ± 0.6	9.3 ± 0.7
20	18.9 ± 0.9	20.3 ± 0.9	20.9 ± 0.9	18.2 ± 0.9
50	49 ± 2	47 ± 3	53 ± 2	55 ± 3
100	97 ± 4	99 ± 5	104 ± 4	103 ± 4
500	503 ± 6	497 ± 7	494 ± 6	497 ± 8
1000	995 ± 9	991 ± 11	995 ± 10	1010 ± 9
D.L (mg/L).	0.004	0.057	0.034	0.06
Sensitivity (counts/mg/L)	2961	2954	1983	1745
Linear Dynamic Range (mg/L)	0.004–1000	0.057–1000	0.034–1000	0.06–1000

Manganese (Mn)

The detection limit for the five lines of Mn follow the trend: 257.611 nm < 403.076 nm < 259.373 nm < 260.569 nm < 294.921 nm. The 257.611-nm line was not interfered by Nd and showed high sensitivity; thus, it was selected for the determination of Mn in a Nd matrix.

Chromium (Cr)

Even though the detection limit and counts for the five lines of Cr (205.618 nm, 267.716 nm, 283.563 nm, 284.325 nm, and 284.984 nm) were found to be good and with reasonable %RSD values, the 284.325 nm line was the only one not to be interfered by Nd; thus, this line was selected for further studies.

Effects on Ag, B, Ba, Ca, Cd, Co, Cu, Fe, Mg, Na, Sr, Zn, Gd, and Lu

All of these analytes show slight interference from Nd (see Table IV). Their analytical performance is listed in Table V. Some of the important observations from the above tables are as follows:

Silver (Ag)

The four lines selected for Ag determination were 338.289 nm, 328.068 nm, 243.779 nm, and 224.641 nm. Among these, the least interfered line was 328.068 nm. The detection limit follows the trend: 328.068 nm < 338.289 nm < 243.779 nm < 224.641 nm, while sensitivity follows just the reverse trend. From these results it can be interpreted that the 328.068-nm line is best for the determination of Ag in a Nd matrix.

Boron (B)

The contribution from Nd for the four lines of B follows the trend: 208.959 nm < 249.773 nm < 182.641 nm < 249.677 nm. The 208.959-nm line was found to be highly sensitive and hence, from both the spectral interference and analytical performance results, this line was best for B determination in the presence of Nd.

Barium (Ba)

The sensitivity for the three lines of Ba followed the trend: 455.404 nm > 230.424 nm > 233.527 nm. The 455.404-nm line was least interfered by Nd. Therefore, this

TABLE III
Spectral Interference of Nd on Bi, Li, Mn and Cr and
the Analytical Performance of Different Analytical Lines of These Analytes

Elements	Analytical Lines (nm)	Spectral Interference from Nd			Analytical Performance in Presence of Nd			
		Contribution from 1000 mg/L Nd	Correction Factor	Tolerance Level (mg/L)	D.L. (mg/L)	Sensitivity (counts/mg/L)	Linear Dynamic Range (mg/L)	%RSD
Bi	223.061	1.7	1.7×10^{-3}	10	0.3	346	0.3-900	3.5
	190.241	11.3	1.1×10^{-2}	50	1.3	39	1.3-600	4.2
	306.772	*	-	1000	0.34	1521	0.34-1000	2.2
	222.825	15.5	1.6×10^{-2}	10	1.5	48	1.5-500	4.8
	206.170	4.6	4.6×10^{-3}	10	0.44	76	0.44-800	3.9
Li	670.78	*	-	1000	0.001	200525	0.001-1000	1.0
	460.289	5.2	5.2×10^{-3}	10	0.18	201	0.18-700	2.5
	323.261	17.4	1.7×10^{-2}	0.05	0.047	199	0.047-800	2.8
	274.118	10.4	1.0×10^{-2}	10	0.81	41	0.81-200	5.0
Mn	257.611	*	-	1000	0.0016	12854	0.0016-1000	1.1
	259.373	0.1	1.0×10^{-4}	500	0.0037	10246	0.0037-1000	1.3
	260.569	0.17	1.7×10^{-4}	200	0.01	5858	0.01-950	1.5
	294.921	2.1	2.1×10^{-3}	10	0.042	1832	0.042-900	2.2
	403.076	2.1	2.1×10^{-3}	10	0.002	9246	0.002-1000	1.4
Cr	267.716	0.57	5.7×10^{-4}	10	0.069	660	0.069-800	3.3
	205.618	0.092	9.2×10^{-5}	500	0.0053	4588	0.0053-1000	2.2
	283.563	1.4	1.4×10^{-3}	10	0.01	3603	0.01-1000	2.6
	284.325	*	-	1000	0.079	1559	0.079-1000	3.3
	284.984	1	1.0×10^{-3}	5	0.018	512	0.018-750	4.1

*refers no interference.

line was chosen for Ba determination in the presence of Nd.

Calcium (Ca)

Six lines were selected for the determination of Ca: 183.801 nm, 315.887 nm, 317.933 nm, 393.366 nm, 396.847 nm, and 422.673 nm. The detection limit followed the trend: 393.366 nm < 396.847 nm < 422.673 nm < 317.933 nm < 315.887 nm < 183.801 nm, while the sensitivity followed just the reverse trend. The contribution by Nd for the 393.366-nm line was found to be less than the others and hence, it was chosen best for further study of Ca in a Nd matrix.

Cadmium (Cd)

The contribution by Nd for the four lines of Cd followed the trend: 226.502 nm < 214.438 nm < 228.802 nm < 361.051 nm. The detection limit for the 226.502-nm line was lower and the sensitivity was higher than for the other lines. Thus, it was chosen for Cd determination in the presence of Nd.

Cobalt (Co)

The detection limit and contribution of Nd for the four lines of Co were found to follow the trend: 238.892 nm < 237.862 nm < 228.616 nm < 230.786 nm, respectively. The sensitivity for these were found to be just reversed, thus the spectral interference and analytical performance favors the Co 238.892 nm line for its analysis in the Nd matrix.

Copper (Cu)

The detection limit trend for the five lines of Cu was: 324.754 nm < 327.396 nm < 224.7 nm < 219.958 nm < 219.226 nm, respectively. The 324.754 nm line was found to be highly sensitive and least interfered in the presence of Nd and thus was chosen as the best line for Cu in a Nd matrix.

Iron (Fe)

Though the Fe 239.562 nm line (out of ten lines: 259.941 nm, 238.204 nm, 239.562 nm, 244.451 nm, 261.187 nm, 262.567 nm, 262.829 nm, 275.573 nm, 241.331 nm, and 373.486 nm) was not the best but showed acceptable analytical performance, it was also least interfered by Nd. Hence, it is considered best for Fe determination in the presence of Nd.

TABLE IV
Spectral Interference of Nd on Ag, B, Ba, Ca, Cd, Co, Cu, Fe, Mg, Na, Sr, Zn, Gd, and Lu Analytes

Elements	Analytical Lines (nm)	Contribution from 1000 mg/L of Nd	Correction Factor	Tolerance Level (mg/L)	Elements	Analytical Lines (nm)	Contribution from 1000 mg/L of Nd	Correction Factor	Tolerance Level (mg/L)
Ag	338.289	1.5	1.5×10^{-3}	10	Fe	259.941	1.3	1.3×10^{-3}	10
	328.068	0.34	3.4×10^{-4}	50		238.204	0.81	8.1×10^{-4}	10
	224.641	20.3	2.0×10^{-2}	10		239.562	0.31	3.1×10^{-4}	50
	243.779	2.1	2.1×10^{-3}	10		244.451	4.4	4.4×10^{-3}	10
B	249.773	0.31	3.1×10^{-4}	50	Na	261.187	0.95	9.5×10^{-4}	10
	249.677	0.52	5.2×10^{-4}	10		262.567	10.4	1.0×10^{-2}	10
	182.641	0.46	4.6×10^{-4}	50		262.829	2.6	2.6×10^{-3}	10
	208.959	0.10	1.0×10^{-4}	500		275.573	1.9	1.9×10^{-3}	10
Ca	396.847	0.98	9.8×10^{-4}	10	Sr	241.331	3.9	3.9×10^{-3}	10
	393.366	0.32	3.2×10^{-4}	200		373.486	#	-	-
	317.933	0.96	9.6×10^{-4}	10		589.592	0.24	2.4×10^{-4}	200
	315.887	2.0	2.0×10^{-3}	200		588.995	0.14	1.4×10^{-4}	500
Cd	183.801	10.0	1.0×10^{-2}	50	Zn	330.237	#	-	-
	422.673	5.6	5.6×10^{-3}	10		330.298	#	-	-
	214.438	0.16	1.6×10^{-4}	500		407.771	0.34	3.4×10^{-4}	200
	226.502	0.13	1.3×10^{-4}	500		421.552	0.09	9.0×10^{-5}	500
Co	228.802	0.34	3.4×10^{-4}	10	Gd	460.733	15.7	1.6×10^{-2}	1
	361.051	#	-	-		213.856	0.09	9.0×10^{-5}	500
	228.616	0.81	8.1×10^{-4}	10		206.200	2.6	2.6×10^{-3}	10
	238.892	0.33	3.3×10^{-4}	10		202.613	0.53	5.3×10^{-4}	50
Cu	230.786	1.1	1.1×10^{-3}	10	Lu	334.502	#	-	-
	237.862	0.44	4.4×10^{-4}	10		342.247	0.36	3.6×10^{-4}	50
	324.754	0.22	2.2×10^{-4}	200		335.047	8.2	8.2×10^{-3}	5
	327.396	2.5	2.5×10^{-3}	10		336.223	0.69	6.9×10^{-4}	200
Mg	224.700	0.42	4.2×10^{-4}	50	Ba	335.862	5.6	5.6×10^{-3}	0.05
	219.226	1.2	1.2×10^{-3}	10		261.542	0.14	1.4×10^{-4}	500
	219.958	0.78	7.8×10^{-4}	10		291.139	0.24	2.4×10^{-4}	10
	279.553	0.10	1.0×10^{-4}	200		307.760	0.63	6.3×10^{-4}	5
	280.270	0.12	1.2×10^{-4}	200		219.554	0.47	4.7×10^{-4}	10
	285.213	0.14	1.4×10^{-4}	200		455.404	0.34	3.4×10^{-4}	10
	279.079	1.3	1.3×10^{-3}	10		233.527	1.5	1.5×10^{-3}	1
	202.647	2.7	2.7×10^{-3}	0.05		230.424	0.83	8.3×10^{-4}	10

Note: #significant interference and the line should therefore be rejected.

Magnesium (Mg)

The contribution of Nd to the five lines of Mg followed the trend: 279.553 nm < 280.270 nm < 285.213 nm < 279.079 nm < 202.647 nm, respectively. The analytical performance result for 279.553 nm line of Mg was far better than the other lines and hence,

seemed to be the best line for Mg determination in the Nd matrix.

Sodium (Na)

Of the four lines of Na, 330.237 nm, 330.298 nm, 588.995 nm, and 589.592 nm, the last two lines slightly interfered, and the 588.995 nm line least interfered. The sensi-

tivity of the lines followed the trend 588.995 nm > 589.592 nm > 330.237 nm > 330.298 nm, respectively, where the detection limit for the 588.995 nm and the 589.592 nm line was almost similar. Thus, considering all factors, the 588.995 nm line seems to be the best line for Na in a Nd matrix.

TABLE V
Analytical Performance of Ag, B, Ba, Ca, Cd, Co, Cu, Fe, Mg, Na, Sr, Zn, Gd, and Lu in the Presence of Nd

Ele- ments	Analytical Lines (nm)	D.L. (mg/L)	Sensitivity (counts/ mg/L)	Linear Dynamic Range (mg/L)	(%) RSD	Ele- ments	Analytical Lines (nm)	D.L. (mg/L)	Sensitivity (counts/ mg/L)	Linear Dynamic Range (mg/L)	(%) RSD
Ag	224.641	1.6	38.6	1.6-800	5.0	Fe	238.204	0.093	800	0.093-900	4.2
	243.779	0.22	346	0.22-700	5.0		239.562	0.032	2063	0.032-1000	2.2
	328.068	0.012	13737	0.012-1000	2.1		241.331	0.27	229	0.27-600	4.5
	338.289	0.018	7156	0.018-1000	2.0		244.451	0.53	157	0.53-900	5.0
B	182.641	0.024	757	0.024-800	4.9		259.941	0.018	1689	0.0018-1000	3.2
	208.959	0.014	4309	0.014-1000	1.8		261.187	0.025	943	0.025-970	4.1
	249.677	0.025	1013	0.025-1000	2.3		262.567	0.29	136	0.29-750	4.9
	249.773	0.014	1587	0.014-1000	2.1		262.829	0.38	194	0.38-1000	4.8
Ca	183.801	0.53	29.6	0.53-900	5.0		275.573	0.14	293	0.14-900	4.5
	315.887	0.10	1114	0.10-800	4.3		373.486	0.052	2173	0.052-1000	2.1
	317.933	0.039	3613	0.039-950	3.1	Na	330.237	0.81	84.2	0.81-900	3.7
	393.366	0.0013	521209	0.0013-1000	1.1		330.298	1.7	52.1	1.7-950	5.0
Cd	396.847	0.0017	400267	0.0017-1000	1.1		588.995	0.016	53419	0.016-1000	1.3
	422.673	0.0041	25334	0.0041-1000	1.4		589.592	0.01	32488	0.01-1000	1.5
	214.438	0.0063	5244	0.0063-1000	2.3	Sr	407.771	0.0002	346709	0.0002-1000	0.8
	226.502	0.0048	11494	0.0048-1000	1.8		421.520	0.0006	262155	0.0006-1000	0.9
Co	228.802	0.025	1884	0.025-800	2.9		460.733	0.033	2307	0.033-1000	1.6
	361.051	0.17	318	0.17-500	3.7		460.733	0.033	2307	0.033-1000	1.6
	228.616	0.049	826	0.049-800	4.0	Zn	202.613	0.008	2058	0.008-1000	2.2
	230.786	0.14	658	0.14-900	4.6		206.200	0.21	157	0.21-500	3.9
Cu	237.862	0.043	1457	0.043-1000	3.9		213.856	0.001	18061	0.001-1000	2.0
	238.892	0.033	2001	0.033-1000	3.6		334.502	0.12	453	0.12-950	3.5
	219.226	0.12	377	0.12-750	3.2	Gd	335.047	0.007	6474	0.007-1000	2.3
	219.958	0.04	762	0.04-900	2.8		335.862	0.015	3729	0.015-1000	3.4
Mg	224.700	0.038	1859	0.038-750	2.3		336.223	0.027	4423	0.027-1000	3.0
	324.754	0.0026	52030	0.0026-1000	1.0		342.247	0.014	5073	0.014-1000	2.4
	327.396	0.0034	34031	0.0034-1000	1.1	Lu	219.554	0.045	911	0.045-800	4.3
	202.647	0.026	496	0.026-800	2.4		261.542	0.003	7454	0.003-1000	1.2
	279.079	0.23	390	0.23-900	3.4		291.139	0.021	2427	0.021-1000	2.8
	279.553	0.0005	653523	0.0005-1000	0.85		307.760	0.095	2856	0.095-1000	2.7
	280.270	0.0009	48536	0.0009-1000	0.94	Ba	230.424	0.012	793	0.012-950	3.8
	285.213	0.0046	15455	0.0046-1000	1.6		233.527	0.091	388	0.091-950	4.8
							455.404	0.028	2479	0.028-1000	1.6

Strontium (Sr)

The three lines chosen for the determination of Sr were 407.771 nm, 421.552 nm, and 460.733 nm. The 421.552 nm line interfered less and possesses good analytical performance; thus, the 421.552 nm line of Sr seems to be appropriate for its determination in a Nd matrix.

Zinc (Zn)

The detection limit trend for the four lines of Zn was: 213.856 nm < 202.613 nm < 334.502 nm < 206.200 nm, respectively. The 213.856 nm line was found to be much less interfered, has the highest sensitivity and lowest detection limit. Thus, the 213.856 nm line

was the best choice for Zn determination in presence of Nd.

Gadolinium (Gd)

The analytical performance of each of the four lines of Gd (335.047 nm, 335.862 nm, 336.223 nm, and 342.247 nm) seems to be close to each other with a reason-

TABLE VI
Spectral Interference of Nd on Ga, Ni, Al, Tl, and Dy and the Analytical Performance of Different Analytical Lines of These Analytes

Elements	Analytical Lines (nm)	Spectral Interference from Nd			Analytical Performance in Presence of Nd			
		Contribution from 1000 mg/L Nd	Correction Factor	Tolerance Level (mg/L)	D.L. (mg/L)	Sensitivity (counts/mg/L)	Linear Dynamic Range (mg/L)	%RSD
Ga	417.206	0.76	7.6×10^{-4}	10	0.03	7693	0.03-1000	1.1
	294.364	5.3	5.3×10^{-3}	0	0.03	864	0.03-1000	3.3
	287.424	11.6	1.2×10^{-2}	0	0.26	182	0.26-800	4.2
Ni	231.604	0.69	6.9×10^{-4}	10	0.01	853	0.01-850	3.6
	221.648	0.55	5.5×10^{-4}	5	0.13	848	0.13-900	3.2
	232.003	1.5	1.5×10^{-3}	10	0.07	704	0.07-800	3.6
	227.021	1.4	1.4×10^{-3}	10	0.05	154	0.05-650	4.6
	300.249	5.5	5.5×10^{-3}	10	0.11	642	0.11-900	3.9
	341.476	2	2.0×10^{-3}	10	0.01	4201	0.01-1000	1.7
	396.152	4.8	4.8×10^{-3}	10	0.01	10037	0.01-1000	1.0
Al	394.401	9.6	9.6×10^{-3}	0	0.02	3109	0.02-1000	1.6
	308.215	0.6	6.0×10^{-4}	10	0.04	1822	0.04-1000	1.5
	309.271	*	-	1000	1.9	3130	1.9-900	1.0
	190.864	0.69	6.9×10^{-4}	10	0.03	848	0.03-1000	2.1
Tl	276.787	#	-	-	0.83	27.2	0.83-600	5.0
	353.170	4	4.0×10^{-3}	10	0.02	14075	0.02-1000	1.0
Dy	394.468	5.1	5.1×10^{-3}	10	0.04	3884	0.04-1000	2.2
	340.780	0.44	4.4×10^{-4}	10	0.02	4300	0.02-1000	1.8
	353.602	8.1	8.1×10^{-3}	10	0.05	4040	0.05-1000	2.1
	364.540	2.8	2.8×10^{-3}	10	0.03	4864	0.03-1000	2.6

Note: *No interference, #Significant Interference.

able % RSD value. However, the 342.247 nm line was found to be least interfered by Nd and hence, was selected for the estimation of Gd in the presence of Nd.

Lutetium (Lu)

The contribution of Nd and the detection limit for the four lines of Lu followed the trend: 261.542 nm < 291.139 nm < 219.554 nm < 307.760 nm. Since the 261.542 nm line is more sensitive than the other lines, it was considered the best line for Lu estimation in a Nd matrix.

Effects on Ga, Ni, Al, Tl, and Dy

These analytes showed a slightly higher interference from Nd than the previously mentioned analytes. The results are listed in Table VI including the analytical performance.

Gallium (Ga)

Three lines (287.424 nm, 294.364 nm, and 417.206 nm) were chosen for Ga. It was found that the 294.364 nm and 417.206 nm lines have similar detection limits with reasonable %RSD values, and are definitely better than the 287.424 nm line. Since the 417.206 nm line interfered least and has a tolerance level of 10 mg/L, it was

considered the best line for Ga determination in a Nd matrix.

Nickel (Ni)

In the case of Ni, of the six lines (221.648 nm, 227.021 nm, 231.604 nm, 232.003 nm, 300.249 nm, and 341.476 nm), only the 221.648 nm and 231.604 nm lines have a closer and lesser value of contribution due to Nd. However, the detection limit of the 231.604 nm line was less and with a good %RSD value than with the 21.648 nm line. Thus, the 231.604 nm line, with a tolerance level of 10 mg/L, seems to be the best choice for analyzing Ni in the presence of Nd.

Aluminum (Al)

For Al, the 309.271 nm line (out of the four chosen lines of 308.215 nm, 309.271 nm, 394.401 nm, and 396.152 nm) was found not to be interfered by Nd. The detection limit of this line was very high, had a poor % RSD value, and was thus not considered. Since the 308.215 nm line was least interfered and has reasonable analytical performance, it was selected as the best line for Al in the presence of Nd.

Tellurium (Tl)

For Tl, the 190.864 nm and 276.787 nm lines were chosen. The analytical performance for the 190.864 nm line was superior over the 276.787 nm line, also has less contribution of Nd, and was therefore chosen to estimate Tl up to 10 mg/L concentration of Nd.

Dysprosium (Dy)

The five lines of Dy (340.780 nm, 353.170 nm, 353.602 nm, 364.540 nm, and 394.468 nm) have good detection limit with an appropriate %RSD value. The contribution by Nd followed the trend: 340.780 nm < 364.540 nm < 353.170 nm < 394.468 nm < 353.602 nm. Therefore, the 340.780 nm line, with a tolerance level of 10 mg/L, is considered the best line for Dy determination in the presence of Nd.

Effects on Pb, In, Ce, La, and Pr

The analytes listed in Table VII show their spectral interference and analytical performance which cause a higher interference in the presence of a Nd matrix than the previously discussed analytes.

Lead (Pb)

Four lines of Pb (220.353 nm, 261.418 nm, 283.305 nm, and 405.778 nm) were chosen for this study. The sensitivity of the 283.305 nm and 405.778 nm lines

was found to be similar to each other and better than the remaining two lines due to their low detection limit and less interference from Nd. Thus, the 283.305 nm line, with a tolerance level of 10 mg/L, was chosen as the best line for Pb determination in the presence of Nd.

Indium (In)

The detection limit trend for the four lines of In was found to be as follows: 230.606 nm < 325.609 nm < 303.936 nm < 451.131 nm. The 230.606 nm line, with a tolerance level of 10 mg/L, was found to be least interfered and was, therefore, chosen as the best line for In determination in a Nd matrix.

Cesium (Cs)

Among the five lines of Cs (393.373 nm, 413.380 nm, 413.765 nm, 418.660 nm, and 448.691 nm), the 413.380 nm line, with a tolerance level of 1 mg/L, and the 413.765 nm line, with a tolerance level of 10 mg/L, showed peculiar results. The 413.380 line has a better detection limit but a poor spectral interference, while the 413.765 nm line showed a completely reverse result. These two lines were found superior over the other three lines and thus, both of them were chosen as the best line for Cs determination in the presence of Nd.

Lanthanum (La)

The detection limit for the three lines of La (333.749 nm, 379.478 nm, and 408.672 nm) were found to be reasonable with an appropriate %RSD value. However, the contribution of Nd for these three lines followed the trend: 333.749 nm < 379.478 nm < 408.672 nm, respectively. Thus, the 333.749 nm line, with a tolerance level of 0.05 mg/L, can be considered the best line for La determination in the presence of Nd.

Praeseodymium (Pr)

The analytical performance results for the four lines of Pr (411.846 nm, 414.311 nm, 417.939 nm, and 422.535 nm) were found to be very close to each other. However, the contribution of Nd is least for the 422.535 nm line, with a tolerance level of 10 mg/L, and was chosen as the best line for Pr determination in a Nd matrix.

Determination of Analytes at Trace Levels in a Nd Matrix Without Chemical Separation Using Synthetic Samples

All of the analytes were divided into four groups according to their behavior in a Nd matrix and eight synthetic samples were prepared using standard reference material of the individual elemental solutions procured from E. Merck, Germany. The synthetic samples were fed into the plasma without any chemical separation and the analytes were determined using the most suitable analytical lines.

Composition of the Synthetic samples

- Synthetic sample 1 (S1): 0.1 mg/L of Bi, Li, Mn, Cr in 1000 mg/L of Nd
- Synthetic sample 2 (S2): 1 mg/L of Bi, Li, Mn, Cr in 1000 mg/L of Nd
- Synthetic sample 3 (S3): 0.6 mg/L of Ga, Ni, Al, Tl and Dy in 1000 mg/L of Nd
- Synthetic sample 4 (S4): 2 mg/L of Ga, Ni, Al, Tl and Dy in 1000 mg/L of Nd
- Synthetic sample 5 (S5): 2 mg/L of Pb, In, Pr and 5 mg/L of Cs, La in 1000 mg/L of Nd
- Synthetic sample 6 (S6): 5 mg/L of Pb, In, Pr and 10 mg/L of Cs, La in 1000 mg/L of Nd

TABLE VII
Spectral Interference of Nd on Pb, Ce, In, La and Pr Analytes and the Analytical Performance of Different Analytical Lines of These Analytes

Elements	Analytical Lines (nm)	Spectral Interference from Nd			Analytical Performance in Presence of Nd			
		Contribution from 1000 mg/L Nd	Correction Factor	Tolerance Level (mg/L)	D.L. (mg/L)	Sensitivity (counts/mg/L)	Linear Dynamic Range (mg/L)	%RSD
Pb	220.353	2	2.0×10^{-3}	10	0.12	275	0.12-850	3.8
	405.778	12.7	1.3×10^{-2}	1	0.13	777	0.13-950	2.2
	283.305	1.1	1.1×10^{-3}	10	0.068	719	0.068-950	1.7
	261.418	4.3	4.3×10^{-3}	0.05	0.28	99	0.28-700	4.4
Cs	418.660	8.7	8.7×10^{-3}	10	0.094	1899	0.094-900	3.2
	448.691	#	-	-	0.85	58.8	0.85-500	4.6
	413.765	4.3	4.3×10^{-3}	10	0.083	2312	0.083-1000	1.8
	413.380	6	6.0×10^{-3}	1	0.055	2314	0.055-1000	1.2
In	393.373	#	-	-	0.21	2216	0.21-1000	1.4
	230.606	1.3	1.3×10^{-3}	10	0.07	577	0.07-800	4.3
	325.609	#	-	-	0.074	1489	0.074-950	2.8
	451.131	#	-	-	8.4	31	8.4-900	3.6
La	303.936	12.7	1.3×10^{-2}	1	0.1	535	0.1-900	3.5
	408.672	12	1.2×10^{-2}	0.5	0.012	5525	0.5-1000	1.1
	333.749	5.7	5.7×10^{-3}	0.05	0.017	3927	0.017-1000	1.6
	379.478	6.6	6.6×10^{-3}	1	0.013	5142	0.013-1000	1.0
Pr	411.846	5.2	5.2×10^{-3}	10	0.033	3257	0.033-1000	1.1
	417.939	20	2.0×10^{-2}	0.05	0.038	3605	0.038-1000	1.2
	422.535	2.3	2.3×10^{-3}	10	0.034	2983	0.034-1000	1.4
	414.311	11.6	1.2×10^{-2}	1	0.019	4095	0.109-1000	1.0

Note: # significant interference.

• Synthetic sample 7 (S7):
0.2 mg/L of Ag, B, Ba, Ca, Cd, Co, Cu, Fe, Mg, Na, Sr, Zn, Gd and Lu in 1000 mg/L of Nd

• Synthetic sample 8 (S8):
2 mg/L of Ag, B, Ba, Ca, Cd, Co, Cu, Fe, Mg, Na, Sr, Zn, Gd and Lu in 1000 mg/L of Nd

The analysis of these samples revealed that 0.1 mg/L of Bi, Li, Mn, Cr; 0.6 mg/L of Ga, Ni, Al, Tl, and Dy; 0.2 mg/L of Ag, B, Ba, Ca, Cd, Co, Cu, Fe, Mg, Na, Sr, Zn, Gd, and Lu; 2 mg/L of Pb, In, Pr; and 5 mg/L of Cs and La can be determined in a Nd matrix without any chemical separation. The analytical results are summarized in Table VIII and were found to be satisfactory.

CONCLUSION

A systematic study was performed on the spectral interference of Nd on 28 analytes involving identification of the least interfered analytical lines of these analytes, determination of their tolerance level, evaluation of the correction factors and analytical performance of these lines in the presence of a Nd matrix (i.e., detection limits, sensitivity, linear dynamic range, and %RSD). Their most suitable analytical lines for their determination in a Nd matrix were as follows: Ag 328.068 nm, B 208.959 nm, Ba 455.404 nm, Ca 393.366 nm, Cd 226.502 nm, Co 238.892 nm, Cu 324.754 nm, Fe 239.562 nm, Mg 279.553 nm, Na 588.995 nm, Sr 421.552 nm, Zn 213.856 nm,

Gd 342.247 nm, Lu 261.542 nm, Pb 283.305 nm, In 230.606 nm, Cs 413.380 nm and 413.765 nm, La 333.749 nm, Pr 422.535 nm, Ga 417.206 nm, Ni 231.604 nm, Al 308.215 nm, Tl 190.864 nm, Dy 340.780 nm, Bi 306.772 nm, Li 670.780 nm, Mn 257.611 nm, and Cr 284.325 nm.

An ICP-AES-based simultaneous determination of Nd revealed that though all of the chosen analytical lines, i.e., 401.225 nm, 406.109 nm, 417.731 nm, and 430.358 nm can be suitably used for the determination of Nd, the best line is Nd 401.225 nm. Using the above-mentioned most suitable lines of the analytes, they can be determined at trace levels in a Nd matrix without any chemical separation.

TABLE VIII
Determination of the Analytes (a) Bi, Li, Mn, Cr; (b) Ga, Ni, Al, Tl and Dy; (c) Pb, In, Ce, La, Pr;
and (d) Ag, B, Ba, Ca, Cd, Co, Cu, Fe, Mg, Na, Sr, Zn, Gd and Lu
in Nd Matrix Without Chemical Separation Using Synthetic Samples

Elements	Analytical Lines (nm)	S1		S2	
		Actual Concentration (mg/L)	Amount Estimated (mg/L)	Actual Concentration (mg/L)	Amount Estimated (mg/L)
Bi	306.772	0.1	0.097 ± 0.004	1	0.97 ± 0.05
Li	670.780	0.1	0.010 ± 0.005	1	0.94 ± 0.08
Mn	257.611	0.1	0.098 ± 0.005	1	1.07 ± 0.06
Cr	284.325	0.1	0.099 ± 0.006	1	1.04 ± 0.05

Elements	Analytical Lines (nm)	S3			S4		
		Actual Conc. (mg/L)	Amount Estimated Before Incorporating	Amount Estimated After Correction Factor (mg/L)	Actual Conc. (mg/L)	Amount Estimated Before Incorporating	Amount Estimated After Correction Factor (mg/L)
Ga	417.206	0.6	1.32 ± 0.04	0.56	2	2.80 ± 0.09	2.04
Ni	231.604	0.6	1.30 ± 0.05	0.61	2	2.70 ± 0.11	2.01
Al	308.215	0.6	1.18 ± 0.06	0.58	2	2.58 ± 0.13	1.98
Tl	190.864	0.6	1.30 ± 0.03	0.61	2	2.65 ± 0.07	1.96
Dy	340.780	0.6	1.09 ± 0.04	0.65	2	2.42 ± 0.08	1.98

Elements	Analytical Lines (nm)	S5			S6		
		Actual Conc. (mg/L)	Amount Estimated Before Incorporating	Amount Estimated After Correction Factor (mg/L)	Actual Conc. (mg/L)	Amount Estimated Before Incorporating	Amount Estimated After Correction Factor (mg/L)
Pb	283.305	2	3.0 ± 0.2	1.9	5	6.0 ± 0.3	4.9
In	230.606	2	3.5 ± 0.3	2.2	5	6.1 ± 0.2	4.8
Cs	413.380	5	9.4 ± 0.4	5.1	10	14.6 ± 0.6	10.3
	413.765	5	9.4 ± 0.4	4.8	10	15.0 ± 0.7	10.4
La	333.749	5	10.9 ± 0.5	5.8	10	15.5 ± 0.8	9.8
Pr	422.535	2	4.1 ± 0.2	1.8	5	7.2 ± 0.3	4.9

Elements	Analytical Lines (nm)	S7			S8		
		Actual Conc. (mg/L)	Amount Estimated Before Incorporating	Amount Estimated After Correction Factor (mg/L)	Actual Conc. (mg/L)	Amount Estimated Before Incorporating	Amount Estimated After Correction Factor (mg/L)
Ag	328.068	0.2	0.52 ± 0.02	0.18	2	2.36 ± 0.08	2.02
B	208.959	0.2	0.32 ± 0.01	0.22	2	2.00 ± 0.09	1.90
Ba	455.404	0.2	0.55 ± 0.02	0.21	2	2.35 ± 0.11	2.01
Ca	393.366	0.2	0.50 ± 0.03	0.18	2	2.30 ± 0.12	1.98
Cd	226.502	0.2	0.35 ± 0.01	0.22	2	2.09 ± 0.08	1.96
Co	238.892	0.2	0.52 ± 0.02	0.19	2	2.29 ± 0.07	1.96
Cu	324.754	0.2	0.45 ± 0.03	0.23	2	2.35 ± 0.09	2.03
Fe	239.562	0.2	0.50 ± 0.02	0.19	2	2.30 ± 0.08	1.99
Mg	279.553	0.2	0.30 ± 0.03	0.20	2	2.11 ± 0.09	2.01
Na	588.995	0.2	0.35 ± 0.03	0.21	2	2.05 ± 0.08	2.01
Sr	421.552	0.2	0.30 ± 0.02	0.21	2	2.11 ± 0.08	2.02
Zn	213.856	0.2	0.32 ± 0.02	0.23	2	2.15 ± 0.10	1.96
Gd	342.247	0.2	0.58 ± 0.01	0.22	2	2.34 ± 0.10	1.98
Lu	261.542	0.2	0.33 ± 0.01	0.19	2	2.08 ± 0.13	1.94

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