

ISSN 2314-5609 Nuclear Sciences Scientific Journal 13, 138-151 2024 https://nssi.journals.ekb.eg

### DERIVATIVE SPECTROPHOTOMETRIC DETERMINATION OF Ce (III), La (III) AND Sm (III), USING CHROME AZUROL S (CAS) DYE IN INDUSTRIAL PHOSPHOGYPSUM

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#### ABSTRACT

The simplest method of improving selectivity is to derivatively alter the spectrum; this procedure reduces spectral interference and, as a result, improves the assay's selectivity. It has been shown that derivative improves selectivity when first, second, third, and fourth derivatives are tried. Digital data derivatization is a well-known method for extracting useful signals from noisy data. Without requiring extraction or separation, the research study refers that application of higher order derivative spectrophotometry enables the simultaneous identification of cerium, lanthanum, and samarium rare earth elements. The established procedure is straightforward, sensitive, and effective. Using this technique can find out the concentrations of Ce, La, and Sm species in rare earth oxides obtained from Egyptian industrial phosphogypsum. The complexes' development and measured were best impacted by studying wavelength, pH, surfactant, molar ratio, initial chrome azurol S (CAS) dye concentration, and finally the linear calibration graphs in the 0.25 to 12  $\mu$ g/ml range for Sm, Ce, and La. The molar absorptivity ( $\epsilon$ ) at the wavelength  $\lambda$ max of the maximum absorbance of the colored species was 0.092, 0.1 and 0.11, relative standard deviation (RSD%) was 1.868, 0.026, and 0.051 and percentage error was 0.98, 0.53 and 0.51 % for Sm, Ce and La respectively.

Keywords: Derivative spectrophotometric, Ce, La, Sm, Chrome azurol S (CAS) and phosphogypsum.

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#### **1. INTRODUCTION**

There has long been interest in the determination of individual rare earth elements in their mixtures, based on the absorption bands of their 4f electron transitions [1-5]. It is very challenging to identify lanthanides in rare earth oxides with high purity whereas, the absorption spectra of all lanthanides are similar [6]. The application of derivative spectrometry to measure lanthanide in highly pure rare earth

oxides is growing [7]. A selective method is described for the determination of (Nd) with methyl thymol blue and cetylpyridinium chloride as a cationic surfactant in rare earth concentrate from laterite deposits in south fourth eastern Sinai using derivative spectrophotometry [8]. Bhayavathy V. investigated a third derivative of (Sm) based on the sensitization of Sm-methyl thymol complex.[9]. A highly sensitive and green method using a non-selective reagent, 2-(5bromo-2-pyridylazo)-5-diethylaminophenol (5-Br-PADAP), in the presence of a cationic surfactant, cetylpyridinium chloride, has been elaborated for simple and rapid trace level determination of Cd (II) ions by derivative spectrophotometry. [10-11]. Praseodymium forms a Pr (LMFX)3 complex with lomefloxacin, the absorption spectra of the complex has been investigated by applying conventional and derivative spectrophotometric methods, the second derivative spectra, the sensitivity is 7.4 times higher for Pr than in the normal method (zero derivative spectra) [12]. A number of metallochromic reagents have been described for the spectrophotometric determination of praseodymium (Pr). These include Xylenol Orange [13], Chrome Azurol S [14], Methyl Thymol Blue [15], Eriochrome Black T [16], Pyrogallol Red [17] and 4-(2-pyridylazo) resorcinol [18]. However, these procedures are not selective as a number of analytes, in addition to other rare earth elements and aluminum, interfere in the determination of trace amounts of praseodymium. Some of these interferents can be eliminated by prior conditioning of the sample, but the masking of other rare earth elements (REEs) and aluminum is not possible. Another approach to eliminating the interference due to other rare earths is to utilize higher order derivative techniques [19]. High pure individual separation of Pr (III) from this concentrate was achieved using a cationic Dowex 50W-X8 ion exchange resin. An EDTA solution 0.015 M<sup>-</sup> <sup>1</sup> was used for the complete recovery of Ce (IV) by oxidation at 200 °C and separation of Pr (III) from REEs concentrate via ion exchange. Simple perchlorate method at  $\lambda_{max}$ 444 nm using fourth derivative spectrum for Pr (III) was used [20]. Samarium, europium, gadolinium in concentrate REEs and generated from Egyptian monazite upgraded to approximately 97% by physical methods are determined using fourth derivative spectrophotometry; the results are in good agreement with that of the ICP-OES method [21]. Phosphogysum (PG) is an industrial residue from processing phosphate rock using the wet acid process to produce phosphoric acid  $(H_3PO_4)$ and Phosphogypsum, PG consists principally of calcium sulphate but also contains a high level of impurities, such as phosphates, fluorides, and sulphates, naturally occurring radionuclides, heavy metals and other trace elements. On the other hand, rare earth elements (REEs) have a great impact on the development of new technologies, e.g., strategic economy sectors [22-24]. PG contains an overall amount of REEs in the interval of 0.343-0.637% by mass and does not show radioactivity level which would exclude it from construction purposes.

#### 2. EXPERIMENTAL

#### 2.1. Instruments:

The Shimadzu (uv-1601) double beam UVvisible spectrophotometer, made in Japan, was utilized. The spectrophotometer has a wavelength range of 190 to 1100 nm, with a resolution of 2 nm, and a wavelength accuracy of  $\pm$ 0.5 nm. A NEL 980 pH meter was employed to measure the pH of the solutions under investigation. Three successive buffer solutions (4, 7, and 10) were used for the calibration of the pH meter.

#### 2.2. Reagents:

The present study employed analyticalgrade chemicals and reagents for its investigations. All of the solutions were prepared using double-distilled water. Sodium hydroxide, ammonium hydroxide, sodium tetraborate, hydrochloric acid, sulphoric acid, nitric acid, perchloric acid, chrome azurol s, studied solutions of Ce, La and Sm (Fluka). A 0.2% (w/v) of the dye solution was prepared by dissolving 0.2 g of (CAS) with double distilled water in 100 ml volumetric flask. Buffer solution prepared by dissolving 0.4 g of NaOH and 1.0061 g of sodium tetraborate in distilled water in 100 ml volumetric flask and complete to the mark.

#### 2.3. Characteristics of the used dye

The utilized dye, namely chrome azurol S (CAS), with chemical formula (C<sub>23</sub>H<sub>13</sub>C<sub>12</sub>Na<sub>3</sub>O<sub>9</sub>S) and with the following structural formula:



Among the benefits of CAS, an organic colorimetric dye, are its high molar absorptivity, selectivity of the complex formations with metal ions, and stable complex formation constants with metal ions. The structural formula of the dye under investigation makes it rather evident that more than one functional group is capable of combining with lanthanum, cerium, and samarium to produce a complex: sulphonate, carboxylate, and hydroxide.

#### **3. RESULTS AND DISCUSSION**

### **3.1.** Formation of chrome azurol S dye and complexes

The absorption spectrum of chrome azurol S dye alone as well as the absorption spectra of Sm, Ce and La -dye complexes against water as a blank in the visible regions between 400 nm- 700 nm Figure (1).

Inspection of the data revealed that the maximum absorbance value for the dye was at 450 nm while for the three complexes were at 473.5, 474 and 476 nm for Sm, Ce and La respectively.



Figure (1): Absorption spectra of chrome azurol S (CAS) dye against water and Sm, Ce and La –dye complexes against reagent blank.

### **3.2.** Influence of various acidic media on the CAS dye complexes of Sm, Ce, and La

Different concentrations from perchloric, hydrochloric and nitric acids were studied (the absorbance by use sulphoric acid media is not noticeable) in order to examine the impact of these acid media concentrations (pH) on complex formation. An aliquot of 1 ml from each of Sm, Ce, and La  $(10^{-3} \text{ M})$ and 1 ml chrome azurol S (CAS) dye ( $10^{-3}$ M) was kept constant in a 10 ml volumetric flask along with various pH concentrations from stock acid solution 1,  $10^{-2}$ , and  $10^{-4}$  M, and the contents were completed to the mark in order to determine the ideal acidic pH medium. Between 400 and 550 nm was the measurement range for the absorbance. The data obtained regarding the impact of varying acidic media (pH values) on the complex absorbance was summarized in Table (1). It showed that the complexes of Sm, Ce, and La formed in  $10^{-4}$ Μ hydrochloric acid media. with high absorbance values measured at pH 4.3.

Acid	pH ranges									
10 <sup>-4</sup> M	6	5.5	5	4.8	4.6	4.5	4.3	4.2	4	3.9
HClO <sub>4</sub>										
Sm abs.	0.351	0.508	0.644	0.698	0.602	0.704	0.737	0.699	0.521	0.405
Ce abs.	0.251	0.408	0.584	0.628	0.665	0.696	0.754	0.744	0.602	0.423
La abs	0.251	0.308	0.484	0.598	0.602	0.684	0.718	0.699	0.504	0.498
HCl										
Sm abs.	0.396	0.609	0.658	0.666	0.669	0.701	0.770	0.697	0.620	0.548
Ce abs.	0.396	0.509	0.581	0.666	0.679	0.715	0.829	0.716	0.676	0.566
La abs	0.396	0.609	0.658	0.676	0.709	0.771	0.798	0.676	0.578	0.382
HNO <sub>3</sub>										
Sm abs.	0.319	0.428	0.579	0.609	0.624	0.697	0.757	0.701	0.663	0.512
Ce abs.	0.319	0.442	0.579	0.609	0.664	0.725	0.744	0.710	0.600	0.523
La abs.	0.319	0.349	0.486	0.508	0.605	0.653	0.708	0.629	0.552	0.363

Table (1): Influence of pH ranges in diverse acidic environments on complexes absorbance.

### **3.3. Influence of optimum buffer for metal- dye complexes**

As mentioned before the selected optimum pH was 4.3 for optimal metal- CAS complex formation. Various buffer solutions were prepared in order to investigate this impact. It was found that the optimum buffer with the highest absorbance values of the Sm, Ce and La- dye complexes result from using 2M sodium acetate and 2M acetic acid in a manner to optimize the pH value (4.3)studied in different media for HClO<sub>4</sub>, HCl and HNO<sub>3</sub> for complexes. In Figure (2) it was recognized that the maximum absorbance value observed with addition of 0.5 mL from buffer pH 4.3.

## **3.4.** Effect of chrome azurol S dye concentration on the absorbance of the complexes

The concentration of chrome azurol s dye should be optimized since lesser or higher

amount than necessary, would cause Beer's deviation from law in the construction of calibration curve. For this purpose, different volumes of 10<sup>-3</sup> M CAS dye were added to a series of 10 ml volumetric flasks volume containing 1 ml of 10<sup>-3</sup>M (Sm, Ce and La) where the pH was adjusted at the optimized value. The volume was completed to 10 ml and the absorbance of each solution was then measured. Figure (3) shows that, the maximum absorbance of the complexes occurs at a concentration of  $1.5 \times 10^{-3}$  M CAS dye at pH value (4.3).

### **3.5. Effect of duration time on the stability of the complexes**

After optimizing the formation of metal- dye complexes, duration of the stability of complexes was studied. All optimized additions were added. The absorbance was measured instantly and after a period of time.



Figure (2): Effect of buffer solution of different pH values on absorbance of metal- CAS complexes formation.



Figure (3): Effect of CAS dye concentration on the absorbance of Sm, Ce and La complexes.

The data is recorded in Table (2) showed that the complexes were formed instantly and stable for 30 minutes.

Table (2): Effect of duration time on the stability of the complexes.

Time		Absorbance	2
(min)	Sm	Ce	La
Zero	0.960	0.990	0.999
5	0.962	0.990	0.998
10	0.962	0.992	0.998
15	0.960	0.992	0.999
20	0.962	0.990	0.999
25	0.962	0.992	0.999
30	0.962	0.990	0.997
35	0.932	0.971	0.958
40	0.902	0.940	0.939

### **3.6.** Stoichiometry of the "metal - CAS" complex

The molar ratio between metal (Sm, Ce or La) and the dye was estimated using the continuous variation method.

#### 3.6.1. Continuous variation method

In this method [25, 26], the molar ratio between the dye and the metal ion is continuously varied by changing their molarities while maintaining the total moles in both constituent's constant. The current study examined the dye-to-metal ratios using the continuous variation approach. The molarities of the two components (metal and dye) were varied using the continuous variation approach, while the total number of moles of both components remained unchanged. The optimum molar ratio showed that the ratio between CAS dye [L] and metal [M] at high absorbance [L]/[M]=1/1.5 as shown in Figure (4).



Figure (4): Continuous variation method for estimating the molar ratio between Sm, Ce and Ladye complexes.

#### **3.6.2.** Construction of calibration curves

Following a thorough examination of the pertinent variables influencing the chrome azurol S dye spectrophotometric determination of Sm, Ce and La, it was necessary to determine the concentration ranges of Sm, Ce and La it was necessary to

determine the minimum and maximum detection limits for complex formation of the studied metals which no further and complexes were formed hence determined. These two limits could be identified through the construction of calibration curves for the complexes. The spectra provide linear calibration graphs in the range from 0.25 to 12  $\mu$ g ml<sup>-1</sup> for Sm, Ce and La respectively as shown in Figures (5, 6 and 7).



Figure (5): Calibration curve of spectrophotometric determination of Sm.



Figure (6): Calibration curve of spectrophotometric determination of Ce.



Figure (7): Calibration curve of spectrophotometric determination of La.

#### **3.7. Effect of interference**

Some elements which can be correlated with samarium. cerium and lanthanum in phosphogypsum had to be tested for interference effects. Generally, some of the common cations and anions which are expected to be associated with samarium, cerium and lanthanum are; Ca<sup>2+</sup>, Fe<sup>3+</sup>, Al<sup>3+</sup>, Si<sup>4+</sup>, P<sub>2</sub>O<sub>5</sub>, SO<sub>4</sub><sup>2-</sup> and total REEs. These cations and anions may interfere in the Sm, Ce and La spectrophotometric estimation; thus, they have been chosen to study their interference effects.

Table (3): Effect of foreign ions on the determination of Sm, Ce and La with equal concentrations of 1  $\mu g$   $mL^{-1}$ 

Foreign ions	Tolerance limits (ppm)
Ca <sup>2+</sup>	7
Fe <sup>3+</sup>	5
Al <sup>3+</sup>	13
Si <sup>4+</sup>	4.5
$P_2O_5$	6
Total REEs	5

According to the composition of the concentrates of the investigated samples, the of interference effect accompanying elements on determining Sm, Ce and La with CAS dye were studied. Then a series of known concentration mixture of Sm, Ce and La were prepared with a series of different concentrations for each interfering element, with a constant concentration of Sm, Ce and La (1  $\mu$ g ml<sup>-1</sup>). The standard solution concentrations of Sm, Ce and La only were measured at the optimized conditions against reagent blank at  $\lambda_{max}$  465 nm. The prepared solutions of Sm, Ce and La with a series of different concentrations of the studied interfering elements were measured at the same optimized conditions. The obtained results indicated that the tolerance concentrations of other elements mixed with Sm, Ce and La should not exceed above certain concentration values. otherwise it was causing a systematic error on their determination using conventional

spectrophotometry. For determination of Sm, Ce and La in presence of each other or precedent cations and anions, derivative spectrophotometric technique was used. Derivatisation of spectra is the easiest approach for increasing selectivity; this process allows for the overcoming of spectral interferences and, as a result, increases assay selectivity. Digital data derivatisation is a well-known method for extracting useful signals from noised data [21].

# **3.8.** Selection of the type of derivative for the spectrophotometric determination of the complexes

The simplest way to boost selectivity is to derivatively modify the spectrum; this process can be used to reduce spectral interferences and, in turn, raise assay selectivity. Derivatization of digital data is a well-known technique for separating valuable signals from noisy data; selectivity is observed to increase with first derivative when first, second, third, and fourth derivatives are evaluated. From the first derivative spectrum Figure (8) it was found that, there are significant peaks for Sm, Ce and La at 315, 350 and 425 nm respectively.

## **3.9.** Construction of calibration curves using derivative spectrophotometry

After the detailed study of the relevant affecting the optimization of factors complex formation of Sm, Ce and La with CAS dye, it was found necessary to determine the concentration ranges of Sm, Ce and La above and below which no further complexes could be formed and hence determined. These two limits could be identified through the construction of calibration curves for the complexes. This spectrophotometric determination must be carried out within the concentration range which obeys Beer's law. The first derivative spectra provide linear calibration graphs in the range from 0.25 to 12  $\mu$ g ml<sup>-1</sup> for Sm, Ce and La respectively after which plateau is formed as shown in Figures. (9, 10 and 11).



Figure (8): Absorption spectra of a mixture Sm, Ce and La using conventional spectrophotometry method and First derivative of Sm, Ce and La respectively using CAS dye.



Figure (9): Calibration curve using 1<sup>st</sup>. Order of spectrophotometric determination of Sm.



Figure (10): Calibration curve using 1<sup>st</sup>. Order of spectrophotometric determination of Ce.



Figure (11): Calibration curve using 1<sup>st</sup>. Order of spectrophotometric determination of La.

#### 4. Evaluation of the method

The current method examined by the reference sample JMn-1. The outcome is shown in Table (4), where it is evident that the method is largely acceptable.

#### 5. Application

The proposed method has been used for derivative spectrophotometric estimation of Sm, Ce and La from rare earth oxides obtained from phosphogypsum. As shown from flow sheet in Figure (12) [28].

Figure (13) is an EDAX chart of individual rare earth oxides obtained from industrial phosphogypsum.

The agreement between the obtained and expected values showed that the method mentioned was accurate in the complex matrices for analysis of Sm, Ce and La. This means that both the reference and observed sample error percentage values using this spectrophotometric technique are small and similar to the approved values obtained by the ICP-OES. This states that the findings obtained are highly accurate and precision.

#### 5.1. Statistical Evaluation of the results

Statistical analysis by using appropriate statistical parameters is here in attempted to cast some light on the significance of the results.

#### 5.1.1. Standard error

Calculation of standard error is very important in the field of applied analytical chemistry as in Table (7). It is used for determination of error percentage for any developed method according to the following successive equations:

Standard deviation (S) =  $[(\sum X_1 - \overline{X})^2/N-1] \frac{1}{2}$ 

Standard error (S.E.) =  $S/N^{\frac{1}{2}}$ 

Error % = 100 (S.E. /  $\overline{X}$ )

RSD % = 100 (S /  $\overline{X}$ )

Where: X<sub>1</sub> measurement value.

 $\overline{X}$  mean of the measurement values.

N number of samples (number of measurements).

Table (4): Analytical results of Sm, Ce and La (ppm) in reference sample by the currently developed approach as opposed to ICP-OES

The present developed method			ICP	-OES met	hod	Certificate result [27]			
Sm	Ce	La	Sm	Ce	La	Sm	Ce	La	
27.3	27.3 270.5 121.5			271	121	30.2	277	122	

#### DERIVATIVE SPECTROPHOTOMETRIC DETERMINATION OF Ce (III), La (III) AND Sm (III)

Compound formula	P <sub>2</sub> O <sub>5</sub>	CaO	Fe <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	MgO	SiO <sub>2</sub>	Na <sub>2</sub> O	K <sub>2</sub> O	TiO <sub>2</sub>	F	$SO_3$	L.O. I
PG Conc. %	1.03	31.5	0.84	0.84	0.30	3.68	0.23	0.10	0.12	0.31	42.3	20.08

Table (5): Chemical analysis of Phosphgypsum.

Table (6): Analytical results of Sm, Ce and La in samples by the present developed method in comparison to ICP-OES method.

The p	resent deve	eloped	ICP-OES method			
	method					
Sm	Ce	La	Sm	Ce	La	
13.7 216 113.5			14	216.4	114	



Figure (12) Flow sheet for obtained rare earth oxides.



Figure (13): SEM-EDAX chart of individual rare earth oxides in PG.

Table (7): Statistical Evaluation of the results.

Element	Content	Mean	S	$S^2$	SE	Error %	RSD %
		x					
Sm	13.5						
	13.7	13.47	0.25	0.063	0.13	0.98	1.87
	13.7						
Ce	216						
	215.9	215.93	0.057	0.003	1.15	0.53	0.03
	215.9						
La	113.4						
	113.5	113.47	0.057	0.003	0.58	0.51	0.05
	113.5						

#### CONCLUSION

The current research utilizes higher order derivative spectrophotometry to identify rare elements phosphogypsum, earth in specifically samarium, cerium, and lanthanum. Without requiring extraction or separation, these elements can be determined through the straightforward, dependable, and sensitive method. The molar ratio, wavelength, pH, initial dye concentration, and linear calibration graphs linear calibration graphs in the range 0.25 to 12  $\mu$ g<sup>-1</sup> for Sm, Ce and La was obtained whereas relative standard deviation RSD% was 1.868, 0.026 and 0.051 percentage error 0.98 %, 0.53 % and 0.51 % respectively, and the lowest quantity of a substance that can be determined was 0.1 ppm for three elements.

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در اسات على تقدير السيريوم ، اللانثانوم والسماريوم بطريقة مشتقة القياس الضوئى في الجبس الفسفوري الصناعي باستخدام صبغة كروم ازورال اس

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يختص هذا البحث بدراسة امكانية تقدير عناصر السيريوم ، اللانثانوم والسماريوم باستخدام طريقة الاشتقاق اللونى مع صبغة كروم ازورال اس فى الجبس الفسفورى الصناعى وفى هذا الغرض تم دراسة العوامل المختلفة و المؤثره على عملية القياس مثل تركيز مكونات المتراكب و نسبة المولاريه و الوسط.

وعلى هذا تم دراسة استخدام طريقة الاشتقاق اللونى لتجنب تأثير تداخل العناصر الأخرى. وقد تم التأكد من ثبات الطريقه بتطبيقها على عينه من وقد أثبتت النتائج لمتوسط القياسات دقة الطريقة وسرعتها في القياس وقلة التكاليف.