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# DETERMINATION ZIRCONIUM WITH THE SOLUTION OF 1 - (2-HYDROXY-1-NAPHTHOYAZO)-2-NAPHTHOL-4- SULFATE

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**Abstract:** New methods for spectrophotometric determination of zirconium have been developed. The 1 - (2-hydroxy-1-naphthoyazo)-2-naphthol-4-sulfate was selected as the analytical reagent. Influence of pH, time and amount of reagent in obtaining complex zirconium has been studied. The dependence of the analytical signal on the concentration of analytes is linear in the range from 2.0–100.0 µg.

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**Keywords:** zirconium, spectrophotometric determination.

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**Introduction.** Zirconium (Zr) is an element with wide application in modern technology. Among the applications of zirconium alloys, is highlighted the use in nuclear area to coat the structural material and also used in ceramic industry, refractories, glazes, enamels, foundry mold and abrasive grits. The main economic source of zirconium, its compounds and alloys is the Zirconium Silicate ( $ZrSiO_4$ ) known as Zircon, is the most abundant mineral zirconium and of great commercial importance[1]. Usually, for quantitative determination of Zr used molecular fluorescence spectrophotometry [2], atomic absorption spectroscopy (AAS), high performance liquid chromatography (HPLC) [3], X-ray fluorescence spectroscopy (XRF) [4] and UV-spectrophotometry [5-7]. For determination of zircon with UV-spectrophotometric methods widely organic reagents as alizarin S [5-6], arsenazo III [6,8], xylenol orange [5,7], 2,4-dinitrophenol-(6-azo-2)-1-naphthol-3,8-disulphonic acid (picramine e) [9], 4-(2-pyridylazo)resorcinol (PAR) [8], 2-(6-bromo-2-benzothiazoylazo)-5-diethylaminophenol and sodium lauryl sulphate [10], 2-(5-bromo-2-pyridylazo)-5-(diethylamino)phenol [11] have been used.

**Methodology & empirical analysis.** This study presents development of a new method for determination of Zr using the solution of 1 - (2-hydroxy-1-naphthoyazo)-2-naphthol-4-sulfate.

**Apparatus and reagents.** Absorption spectra were measured on an EMC-30PC-UV spectrophotometer. The pH control of solutions was carried out on a universal ion meter EV-74 and pH meter / mV / TEMP Meter P25 EcoMet (Korea).

For this work a standard Zr (IV) solution was prepared by dissolving  $ZrOCl_2 \cdot 8H_2O$  (0.39 g) in 0,1 M HCl (3 ml) and the solution was adjusted to the mark with bidistillate water in a volumetric flask (100 ml). 0.1% 1 - (2-hydroxy-1-naphthoyazo)-2-naphthol-4-sulfate (HNNS) were prepared by dissolving its 0.1 g sample in 100 ml of bidistilled water.

**Procedure**

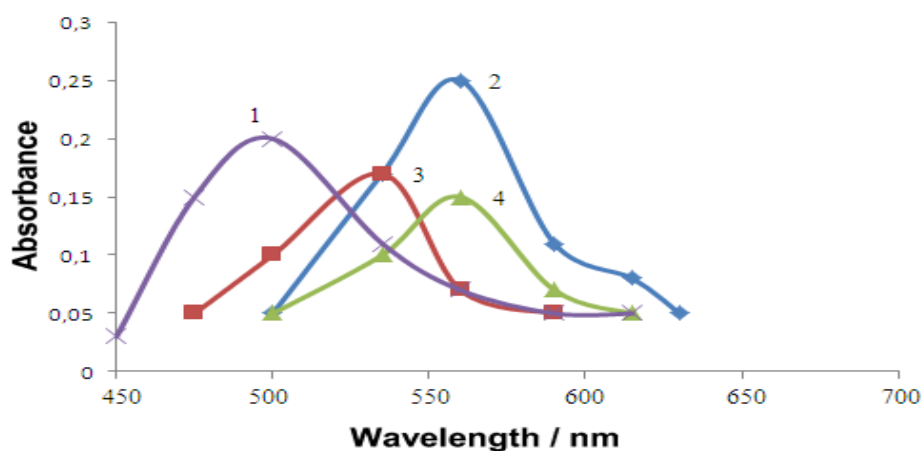
**General Procedure**

Into a 25 ml calibrated flask were transferred 1.0 ml  $1.0 \cdot 10^{-4} M$  Zr(IV). To this flask were added 0.1% HNNS (1 ml) and 2.0 ml 0.1 M HCl. This flask diluted to the mark with bidistillate water. Then, absorbance was measured immediately at 570 nm in a 1.0 cm cell against the reagent and water blanks.

**II. Results.** We know that, for determination of some metals - monoazo systems were successfully applied [12-15]. So sodium salt of HNNS as monoazo derivative was taken in this study. These reagents enhance stability of formed band with metals, because of high electron density between -OH groups, ortho to azo group and nitrogen atom of azo group. Thus, analytical properties of formed complex increase. Zr metal was chelated with HNNS ligand. In this work, the optimal conditions for obtaining chelate were studied.

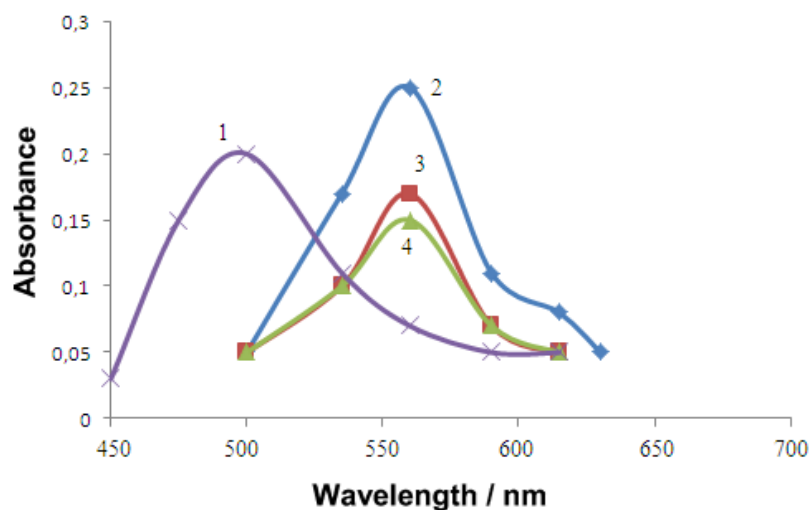
**Effect of Acids and pH**

This paper were studied the effect of various 0.1 M acids ( $HNO_3$ , HCl,  $HClO_4$ ) and HCl acid were found to be the best acid on the complexation of HNNS with Zr in equilibrium aqueous phase [fig.1].



**Figure 1.** Absorption spectra of complex formation of HNNS with Zr(IV) on various acids: 1-HNNS, 2- 0.1 M HCl, 3- 0.1 M  $HNO_3$ , 4- 0.1 M  $HClO_4$ .

After known the best acid on the complex formation, the effect of concentration of acid to the complexation of HNNS with Zr(IV) was studied and given in Fig. 2.

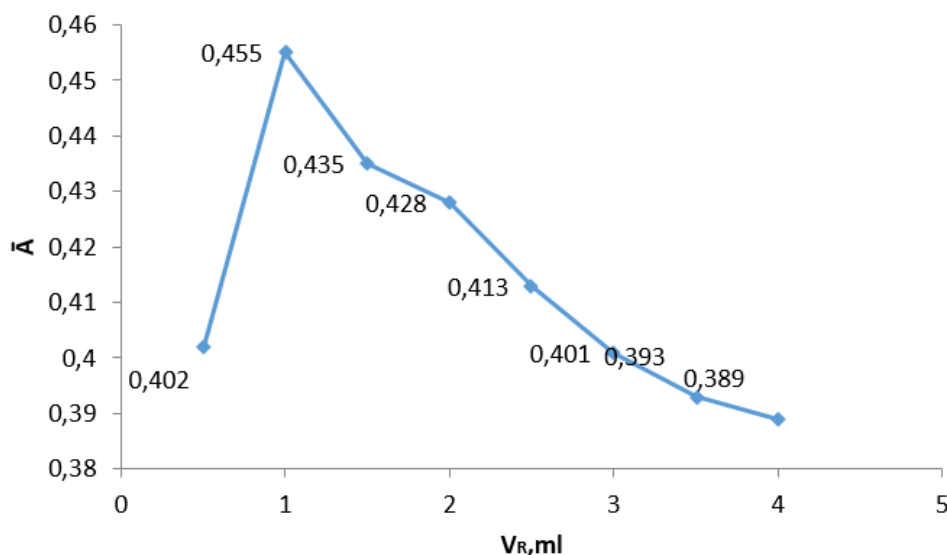


**Figure 2.** The effect of pH on the complexation of HNNS with Zr on various concentration of HCl acid`s: 1-HNNS, 2- 0,1 M, 3- 0,5 M, 4- 1 M.

In this Figure 2, we see that absorbance of complex gives high absorption peak at 570 nm in 0.1 M acidic medium. A 70 nm difference was observed between maximum absorbance wavelength of HNNS and Zr(IV) complex.

**Effect of HNNS amount**

For study effect of amount of ligand were taken fixed Zr-ion concentration, complex observed that at 1.0 ml 91.0 µg Zr metal and 0,5; 1,0; 1,50; 2,0; 2,50; 3,0; 3,50; 4,0 ml of 0.1 % HNNS.



**Figure 3.** Effect of the amount of reagent on the absorbance of the Zr(IV)-HNNS system.

From the results of the research, it can be observed that the optical density reached the highest value as a result of interaction with 1.0 ml solution containing 91 µg of

zirconium and 1.0 ml solution containing 0.1% reagent. A decrease in optical density was observed when the amount of reagent exceeded 1.0 ml.

**Effect of Time**

Complex formation reaction Zr(IV) and HNNS was instantaneous and absorbance remained stable for at least 180 seconds in a room temperature. And every 20 seconds the optical destiny was measured.

**Table 1.** Time dependence of optical density

T <sub>sec</sub>	20	40	60	80	100	120	130	150	160	170	180
$\bar{A}$	0,455	0,455	0,455	0,455	0,455	0,455	0,454	0,449	0,438	0,429	0,418

It was observed that the optical density of the complex remains unchanged for 120-130 seconds, and the value of the optical density changes in the following minutes.

**Calibration graph (Beer’s law and sensitivity).** In this paper was studied the effect of metal concentration over 0.01–100 mg l<sup>-1</sup>. For study of the measurement effect concentration of metal, were prepared four different sets (0.01–0.1, 0.1–1, 1–10, 10–100 µg) for convenience. The absorbance was linear for 2–100 µg of zirconium at 570 nm.

**Effect of Interference**

The influence of interfering species on the determination of Zr(IV) was studied under optimal conditions using the proposed method. The proposed method is more selective than the arsenazo III (Table - 1) method for determining Zr (IV) in the presence of Th<sup>4+</sup> and Mn<sup>2+</sup>.

**Table 2.** Comparison of effect of interfering ions on determination of 91 µg zirconium(IV) with HNNS.

Interfering ion (I)	Limiting mass ratio (Zr: Me)	
	Proposed method	Arsenazo III method
Al <sup>3+</sup>	1:25	1:1
Th <sup>4+</sup>	1:2	1:0.1
Pm <sup>3+</sup>	1:10	**
Sm <sup>3+</sup>	1:5	1:1
Gd <sup>3+</sup>	1:5	1:2
Ce <sup>3+</sup>	1:1	1:1

**III. Conclusion.** Thus, the developed method for determination of zirconium with HNNS on the spectrophotometric method is high accuracy at 0.1 M HCl, 1:1 volume ration, and room temperature condition in 120 seconds. The developed method by us for the determination of zirconium can be successfully applied to the analysis of alloys, minerals, as well as in the analysis of ores.

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