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Preparation and use of a new organic Reagent of 4-(Furan-2-yldiazenyl)-3-hydroxy naphthalene-1-sulfonic acid (FDHNSA) In An Analytical study of some Metal ions and Drugs, such as salbutamol (SAL) and methyldopa (MDP).

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ABSTRACT

This study includes the synthesis of organic reagent 4-(Furan-2-yldiazenyl)-3-hydroxy naphthalene -l-sulfonic acid (FDHNSA) by reacting a diazonium Salt (4-amino-3-hydroxy naphthalene-1-sulfonic aciel) with Furan in alkaline ethanol solution. The prepared reagent was identified by using (UV-Vis), (FT.IR), (H^1NMR) techniques, it was λ_{max}=412 nm for the reagent (FD HNSA) in ethanol. The melting point of reagent (FDANSA) is found to be (129-130)°C. This study tried to find a quick, easy and inexpensive spectral way to detection two kind of drugs, Methyldopa (MDP) and Salbutamol (SAL) through the reaction the drugs with anew regent 4-(furan-2-yldiazenyl)-3-hydroxynaphthalene-1-sulfonic acid (FDHNSA) in alkaline medium. The results a highest absorption for (SAL) with FDHNSA at λ_{max}=485 nm, while the λ_{max}= 348 nm for SAL in water, the highest absorption for the Free reagent (FDH NSA) λ_{max}= 412 nm in ethanol. This study was conducted to find the optimized Conditions to estimate, the drug (SAL) with the new Reagent (FDHNSA), It was show the Optimized Concentration reagent was equal to (0.023M) and (2.5ml), The volume of drug (SAL) was equal to (1.5ml). The value of pH was equal (11). Added to that the effect of temperature and time were Conducted, a higher absorbance was obtained when temperature At (35°C), while at that best time it was obtained at (15min) for (SAL). The Calibration curve for the durg (SAL) was Constructed after establish the optime conditions. The Calibration Curve was constructed and subsequently the Beer's Low was obeyed within the Concentration range of SAL (0.75 – 12 $\mu g.ml^{-1}$). The Molar absorptivity (ε) Value was equal $1.5802 \times 10^{+4} L.mole^{-1}.cm^{-1}$. The Sandell's sensitivity value was equal 0.0365 $\mu g.ml^{-1}$. The R.SD% value was equal. 1.230% with a correlation coefficient 0.9995. The stoichiometry of the reaction between the drug (SAL) and reagent (FDHNSA) was proposed with continuous variation and mode ratio methode was (1:1) ,(SAL:FDHNSA). Finally, interference was studied. The results reveal no effect on the determination of the drug This methods was success fully applied for the determination of the drugs and in pharmaceutical compounds. All materials used in the search are from the company B. D. H, Fluka, Riedel-Dheang seelz-Hannover, and Merk.

Keywords: organic reagent, FDHNSA, SAL

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INTRODUCTION

1- Azo dyes: Aso pigments from (60-70)% in all dyes (1), the reason for their designation is the presence of the azo group (-N≐N-) hybrization sp2 and related to the aromatic system (2), in order to prepare azo properties, we should choose amine first, which is the process of aldidze, then the reaction (3), the azo group may be associated with many different groups (R-N=N-R). These are dyes and their metal complexes are very low in dispersion to nitrogen and hydrocarbons, such as aromatics, which are called aromatic dyes, which are characterized by their high stability due to the high stability of these compounds. The color intensity is high due to the resonance phenomenon found in these aromatic compounds (4), one example is the simplest compounds of azo benzene.

Azo compounds are one important organic compound in many fields such as pharmaceuticals and cosmetics (5,6). The use of dates back to the prehistoric are and all sources used in this peried are natural sources (7,8), at the beginning of the 20th century, hatural dyes were replaced with industrial dyes and the researcher was able (William Perkin) 1856 from the preparation of the purple aniline dye (9,10), it considered the first cast trade mission until the year 1970 was called a dye Brown the composition of the compound as an average of pigments in clouds Yellow, orange, Red; Purple, Blue, Each compound is different depending on the initial material and the breadth of the additives if the system containing the na specific wavelength particles(11), azo dyes are important in the field organic synthesis as reagents in measuring chromatography(12), and are used in medical fields(13), azo dyes are used in chemistry analysis corrective processes such as or gage and reddish pigment as wea as used in the wall in dusty, wood and silk (14).

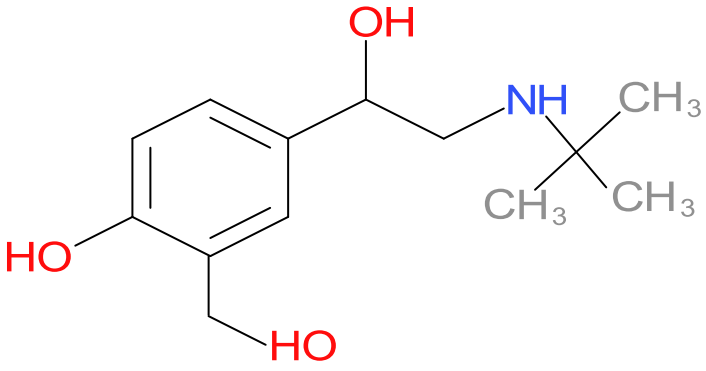
In the field of drugs, these compounds have been used as drugs because they have inhibitory wall of bacterial (15), and these germs germicidal colon and bacteria causing chronic bowel disease (16,17), and cancer (18), the article was used prontosil itison azo anti-cancer drug and ulcer(19) classification of azo compounds are classified according to the azo compounds founds in the knees, such as mono clonal, bifocal or train gulor (20), or can be classified into two main categories depending on the rings associated with the two groups of the two.

- Hormo cyclic azo compounds.
- Hetro cyclic azo compounds.

Salbutamol (SAL)

Salbutamol is a bronchodilator in the epinephrine family, which relaxes the muscles surrounding the bronchi, and is used in respiratory distress and in chronic asthma, reducing premature births [21].

Salbutamol is available on the market in tablets, inhalers and syrup, preferably not for use by pregnant and breastfeeding women unless consulted by a doctor, salbutamol has the following formula [22].



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Salbutamol (C13H21NO3)

Molar mass= 239.311 g/mole

RS – 4 – [2 – (terd – butyl amino) – 1 – hydroxyl ethyl] – 2 – (hydroxyl methyl) phenole.

EXPERIMENTAL

Apparatus:

FTIR spectrophotometer (FTIR-8400s) from Shimadzu company, UV-vis. spectrophotometer single Beam UV-vis- (uv-1650pc).

Spectrophotometer from Shimadzu company, UV-vis. -spectrophotometer Biotech, UV-vis. Spectrophotometer (uv-9200), pH meter instrument, pH meter HANN Instrument, Melting point instrument electro thermal malting point 9300-u-k, Electrical balance B1210s, Water bath:- GFL 1083, Molar conductivity meter:- Cand- 720 (WTW), C.H.N analysis Euro vector instrument & software, Atomic absorption spectrophotometer Atomic Absorption 2600, H^1NMR Spectro photometer (400MHZ) Fourier Trans formation Bruker.

Preparation of Salbutamol stock solution (100 $\mu g.ml^{-1}$)

A 0.1000 gm amount of pure SAL was dissolved in distilled water and the solution was made up to volume of 100ml in Volumetric flask with the same solvent. To obtain (SAL) working solution (100 $\mu g.ml^{-1}$) a 10ml volume of the stock solution was transferred into a 100ml volumetric flask and made up to the mark with distilled water.

Preparation of organic reagent [23]

4-(Furan –2– yldiazenyl) –3– hydroxy naphthalene –1– sulfonic acid (FDHNSA)

It was melted (0.004mole) from (4-amino-3-hydroxy naphthalene -1- Sulfonic acid) in (10ml) from ethanol and place the mixture under Cooling (0 - 5°C), the add (1ml) from HCl with stirring and then add (0.004 mole) from $NaNO_2$ dissolved in (10ml) from water at (0-5°C), then leave the mixture for (5-10Min) period to complete to dialysis process, then add this solution (0.004mole) from Furan dissolved in (20ml) from ethanol and same degree warm (0-5°C) , then add to mixture (5ml) from Noah with concentration (0.1M), the leave the mixture under the cooling from the next day.

Then wash the precipitate several times with distilled water and leave to dry. As in the chart (1)

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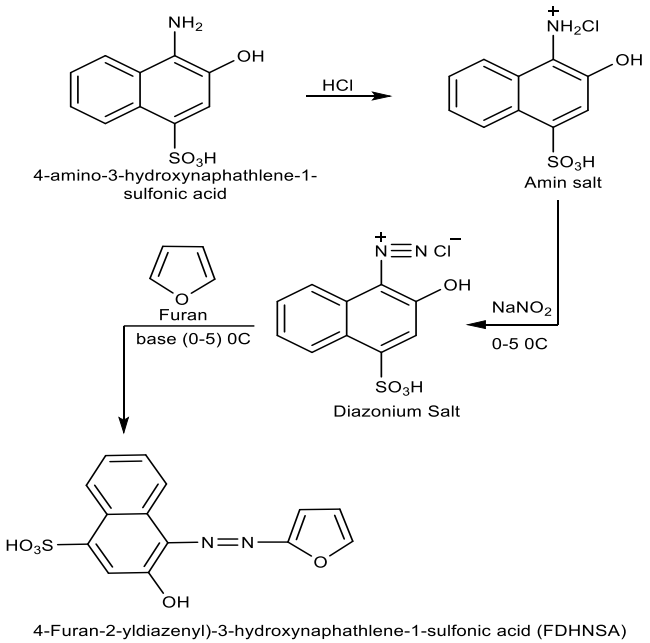

Chart (1) steps to prepare organic reagent (FDHNSA)

Hydrochloric acid solution (0.8m)

Prepared by dilution of concentration hydrochloric acid and standardized against sodium carbonate.

Sodium hydroxide solutions (0.1m)

A 0.4000gm of sodium hydroxide was dissolved in distilled water and made up to the 100m volumetric flask with the same solvent

Analysis of commercial dosage forms

Twenty tablets were accurately weighed and powdered. An amount to tablets equivalent to 100mg of the pure drug, woes dissolved in distilled water and tranferred into a 100mg calirated flask and completed to the mark with the same solvent. The flask with its contents was shaked well and filtered. Samples of 2-13$\mu g.ml^{-1}$ of (SAL) were taken and the meassivements were carried out as described ealier under general procedure.

Stoichiometry of the reaction

The stoichiometry of the reaction between each (SAL) and FDANSA was investigated under the recommended optimum Conditions and applying Job's method[24] volumes of (1-10 ml) of 0.023M portions of FDHNSA were coupled with corresponding 0.023M (SAL) solution to give a total volume (10ml). The results obtained (Fig.1) showed that (1:1) (SAL:FDHNSA) product was formed between (SAL) and FDHNSA the apparente stability constante was sateetation calculated by comparing the absorbance of solution containing stoichiometric amount of (SAL) and FDHNSA with that of a solution containing a five-fold excess of FDHNSA reagent. The stability constant of the product in water under the described experimental conditions was $2.4432 \times 10^{+4}\ L.mole^{-1}$.

RESULTS AND DISCUSSION

Absorption spectra

When a very diluted aqueous solution of SAL was mixed with FDHNSA reagent in alkaline medium, an intense yellow-orange azo dye formed immediately, wich became stable after (5min). The yellow-

orange product has a maximum absorption at λ_{max}=485nm Fig.2 , Fig.3 shows the spectra of product formed and of the reagent blank and the free reagent.

The maximum absorption at (485nm) was used in all subsequent experiments.

-Study of the optimum reaction Conditions

The effects of various parameters on the absorption intensity of the formed product were studied and the reaction conditions were optimized

Effects of reagent concentration.

When various concentration of FDHNSA solution were added to a fixed concentration of SAL, 2.5 ml of 0.023M FDHNSA solution was sufficient to develop the color to its full intensity and give minimum blank Value, 2.5 ml the absorbance of blank value increased, causing a decrease in the absorbance of sample. There for, 2.5 ml of 0.023M FDHNSA solution was found optimum and was used in all subsequent experiments.

Effect of pH value

Preliminary results indicated that the presence of an al Kaline in the reaction mixture is essential for developing a more intense yellow-orange color. In this respect, sodium hydroxide, potassium hydroxide, sodium acetate ammonium hydroxide and sodium carbonate were examined, It was found that the best results were obtained with sodium hydroxide, therefore, sodium hydroxide was chosen and 1ml of 0.lm solution was added as optimum after the diazotized reagent as it gives a high sensitivity and minimum blank value. The best pH was equal (11) Fig. 4.

Effect of Reaction time

In spite of the rapid color development, the color intensity reached a maximum after (SAL) solution had been reacted with FDHNSA in alkaline medium was equal (15min) Fig.5.

Effect of Temperature

The effect of temperature was studied ant its effect on the stability of the resulting compounds, the best reactions temperature was 35°c fig.6.

Accuracy and precision

To determine the accuracy and precision of the methods, SAL was determined at of five determinations. The overall relative standard deviation and recoverie were summarized in Table (1). small relative standard deviation (less than 1.992) and a good recovery (101.7%) indicated high precision and accuracy of the method.

Calibration curve

Under the recommended conditions described above and mentioned in the general assay procedure, a linear calibration graph (Fig.7) for SAL was obtained which shows that Beer's low obeyed over the concentration range of (0.75-12 $\mu g.ml^{-1}$) with a correlation coefficient of 0.9992. The conditional molar absorpativity of the product formed with SAL was found to be $1.5802 \times 10^{+4} L.mole^{-1}.cm^{-1}$ with reference to the SAL and sandell's sensitivity was $0.0535 \mu g.ml^{-1}$ [Table 2].

Analytical Applications

The suggested method was applied to the quantitative determination of SAL in pharmaceutical formulation.

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Two types of tables containing SAL have been analyzed and they gave a good accuracy and precision as shown in [Table 3]. The proposed method was compared successfully with other standard method (spectroscopic methods).

CONCLUSION

A simple, rapid and sensitive spectrophotometric method has been developed for the determination of trace amount of SAL in aqueous solution based on reagent azo (FDHNSA) in the presence of sodium hydroxide. The proposed require temperature control [Table 4].

The method was applied successfully to pharmaceutical tablets containing (SAL). Easy to prepare the organic reagent by obtaining diazione salt for aromatic salts in normal Conditions.

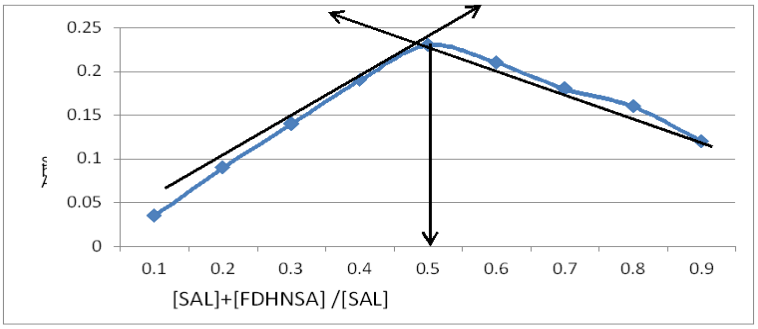
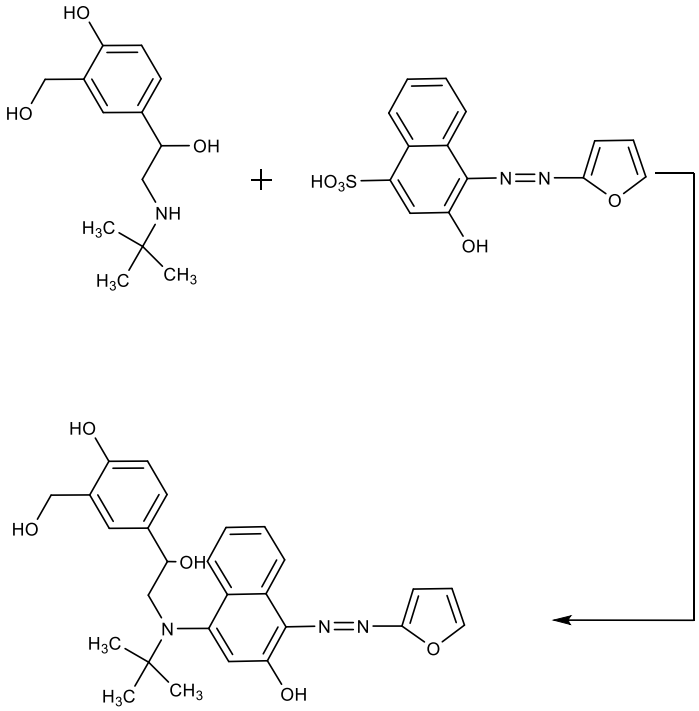


Figure 1: Job's method for the (A) complex produced



Suggested structure product SAL with FDHNSA

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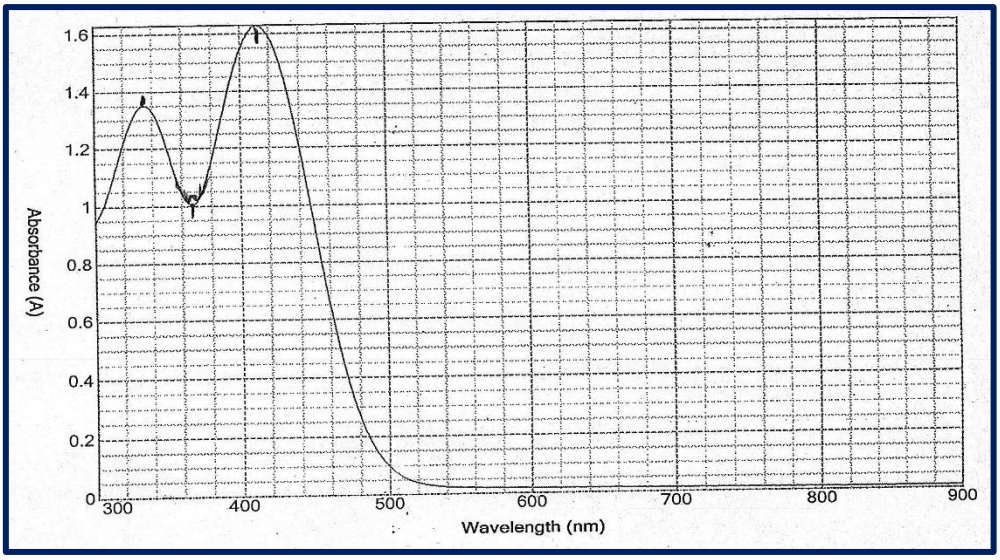

Figure 2: Absorption spectra of reagent FDHNSA $\lambda_{max} = 412nm$

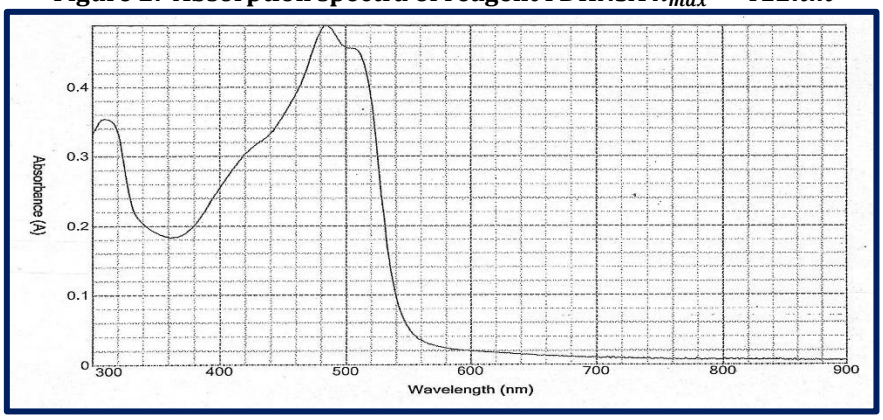

Figure 3: Absorption spectra of SAL with FDHNSA $\lambda_{max} = 485nm$

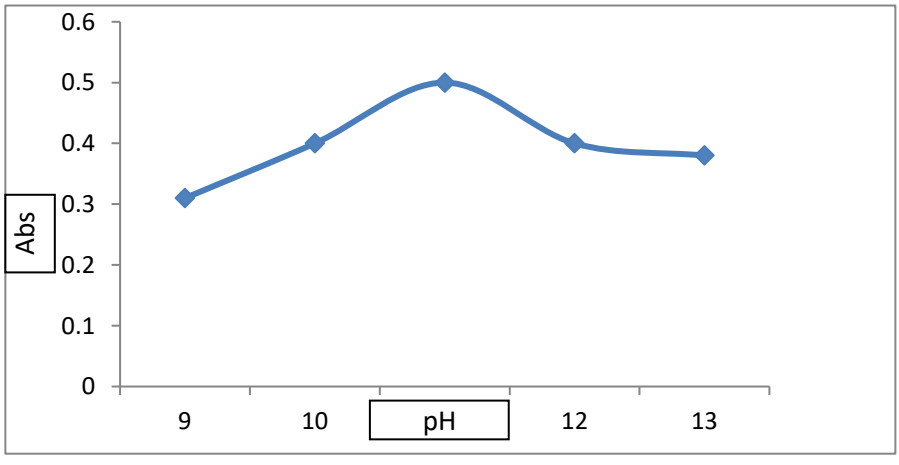

Figure 4: Effect of pH

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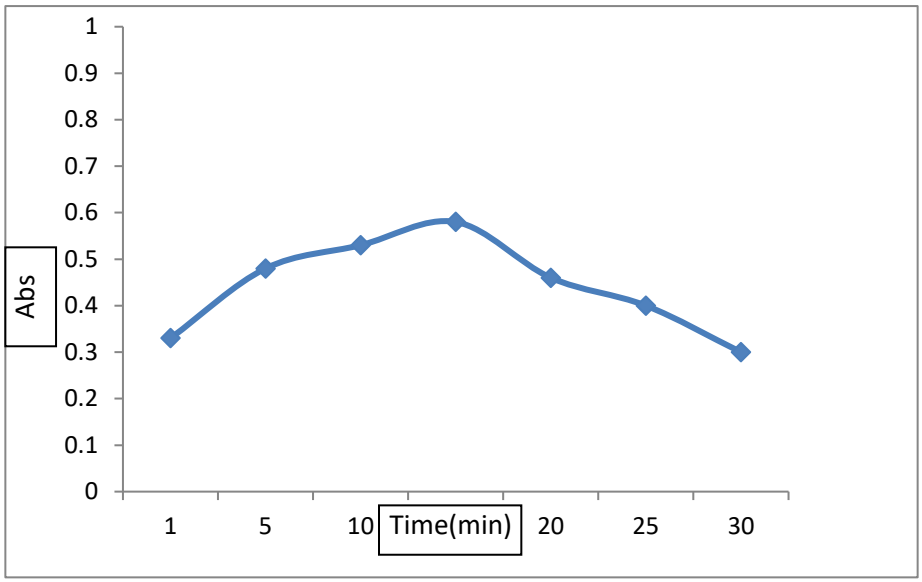

Figure 5: Effect of Reaction Time

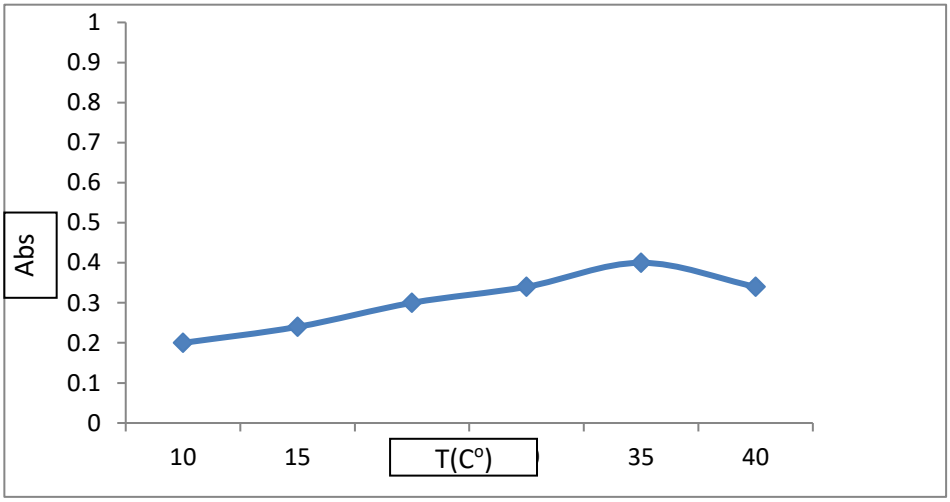

Figure 6: Effect of Temperature

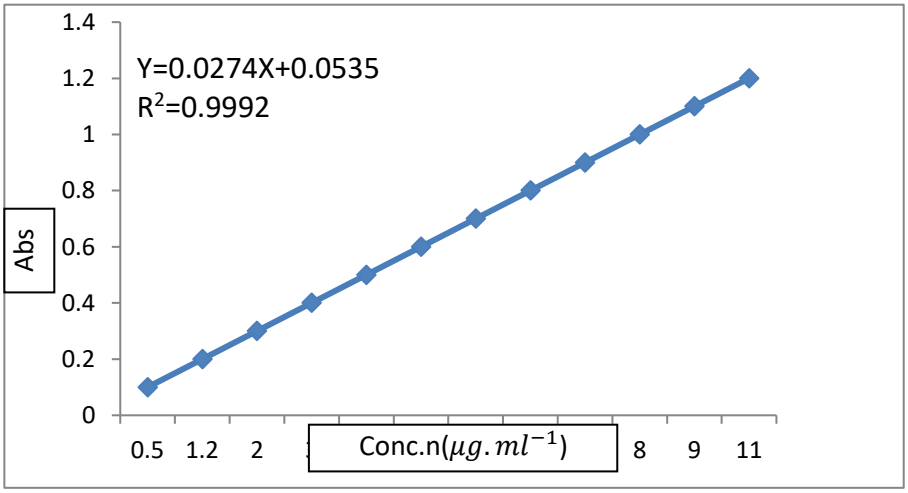

Figure 7: Calibration curve of (SAL) with FDHNSA

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Table: Accuracy and precision of the proposed method

Values	SAL
Wavelength (nm)	485
Con. C ($\mu g.ml^{-1}$)	3.5
$\bar{X}$	0.234
R.S.D%	1.230%
Error%	1.7%
DL($\mu g.ml^{-1}$	1.292
%Recovery	101.7%

Table 2: Analytical data obtained from proposed method

Parameter	SAL
Beer's low limit ($\mu g.ml^{-1}$)	0.75-12
Molar absorptivity ($L.mol^{-1}.cm^{-1}$)	$1.5802*10^4$
Sandell's sensitivity ($\mu g.cm^{-2}$)[25]	0.0365
Detection limit ($\mu g.ml^{-1}$)[26]	0.3656
Quantition limit ($\mu g.ml^{-1}$)[27]	0.8345
Correlation Cofficient (R)	0.9995
Determination Cofficient (R^2)	0.9992
Slop (b)	0.027
Intercept (a)	0.054

Table 3: Application of proposed method for the determination of SAL in preparations pharmaceutical

SAL Sample (Tablets)	Comc.mg/ml		Error%	Recovery%
	present	found		
Butadin (SDI)	5	4.880	+2.4	102.4
Butadin (Dijla)	0.5	0.504	-0.8	99.2

Table 4: comparison of the proposed method with some method for the determination of (SAL)

Application	Wavelength nm	Reagents used	linear range $\mu g.ml^{-1}$	drugs	R
Butadiene	620	Chloramine- T – N ,N –Dimethyl – p – phenylene dimine	10-40 LOD=0.3	Salbutamol	(28)
Butadiene	448	Diazotized – o –	0.6-14 LOD=0.4	Salbutamol sulfate	(29)
This study	433	NQS	0.75-12 LOD=0.5 LOQ=0.5311	Butadiene	-
This study	485	FDHNAS	0.75-12 LOD=0.36 LOQ=0.83	Butadiene	-

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