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Spectrophotometric determination 3-(benzyliden amino)-6-methyl-2-thioxo 2,5-dihydro pyrimidine-4(3H)-one (LS) by zero order method

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Abstract : The objective of this work rapid, accurate, precise, simple, specific and expeditious spectrophotometric method was used for determination of Schiff bases 3-(benzyliden amino)-6-methyl-2-thioxo 2,5-dihydro pyrimidine-4(3H)-one (LS).It was prepared and characterized using (FT-IR, UV) spectroscopy. The quantitative determination of (LS)was carried out using the zero order derivative values measurement of light absorption in UV region. The UV spectra of Schiff bases 3-(benzyliden amino)-6-methyl-2-thioxo 2,5-dihydro pyrimidine-4(3H)-one (LS) showed that maximum absorbance of light was observed at (316) nm and linearity was observed in the concentration range of(4-12)ppm with correlation coefficient 0.997.

Keyword : 3-(benzyliden amino)-6-methyl-2-thioxo 2,5-dihydro pyrimidine-4(3H)-one(LS), Zero-order method, Spectrophotometric.

Introduction

Pyrimidine is the parent6-membered heterocyclic ring of nitrogen and carbon group of composed compounds[1] that have been explored for developing pharmaceutically important molecules[2,6]. They are present during nature in various forms and the building blocks of abundant natural compounds from antibiotics to vitamins and liposacharides. The most usually recognized pyrimidine is the bases of RNA and DNA[3,5], the most abundant being uracil, thymine or cytosine[4]. The aim of this research is to progress economic, precise, rapid, , accurate and simple zero-order spectrophotometric method for determination of 3-(benzyliden amino)-6-methyl-2-thioxo 2,5-dihydro pyrimidine-4(3H)-one(LS).

Apparatus and Instrument

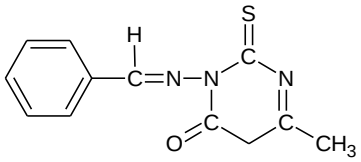
A Double beam UV-Visible spectrophotometer (Shimadzu) (Japan) having two matched1 cm quartz cel llight path was used for experiment. The weight was taken on Analytical balance.

Experimental

1-Preparation of 3-(benzyliden amino)-6-methyl-2-thioxo 2,5-dihydro pyrimidine-4(3H)-one (LS).

Methanol solution of thiosemicarbazide (0.03mol,0.87gr) was mixed with benzaldhyde (0.01mol,1.09gr) and ethyl acetoacetate (0.02mol, 1.4gr). The resulting reaction mixture was refluxed on a

steam bath for (8 hrs). The product (LS) was filtered, washed with cold water, dried and crystallized from methanol[7].fig(1).



Fig(1)Chemical structure of3-(benzyliden amino)-6-methyl-2-thioxo 2,5-dihydro pyrimidine-4(3H)-one (LS)

2-Preparations of the standard solution.

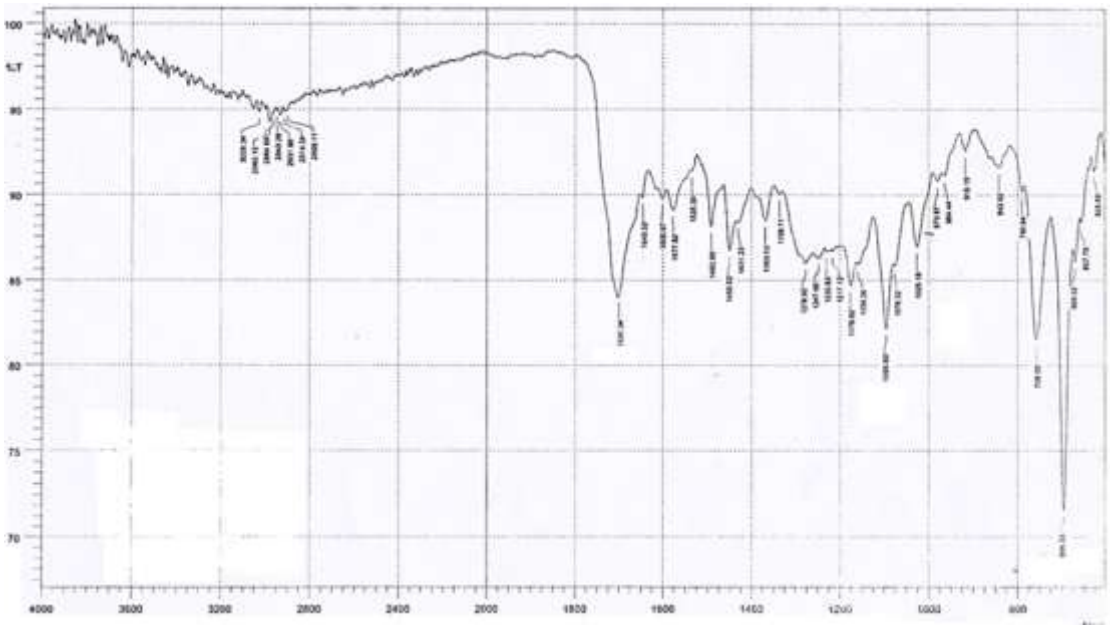
The stock standard solution of (LS) was prepared in methanol to a concentration of 1000 ppm. Working series standards solution (4-12 ppm) for 3-(benzyliden amino)-6-methyl-2-thioxo 2,5-dihydro pyrimidine-4(3H)-one (LS) were prepared by diluting stock solutions with methanol .A calibration graph was constructed in the(4-12 ppm) of (LS) (n = 5).

Result and discuses

Infrared Spectroscopic Study

before subjecting(LS) studies identification functional group was carried out using FT-IR spectrophotometer in the range of (400-4000)cm. good informations about the (LS) The characteristic frequencies of the(LS) showed in Fig.2 .one absorption bond at (1699)cm^{-1}toυC=O and two absorption bands at (1645,1095)cm^{-1}, can be attributed to

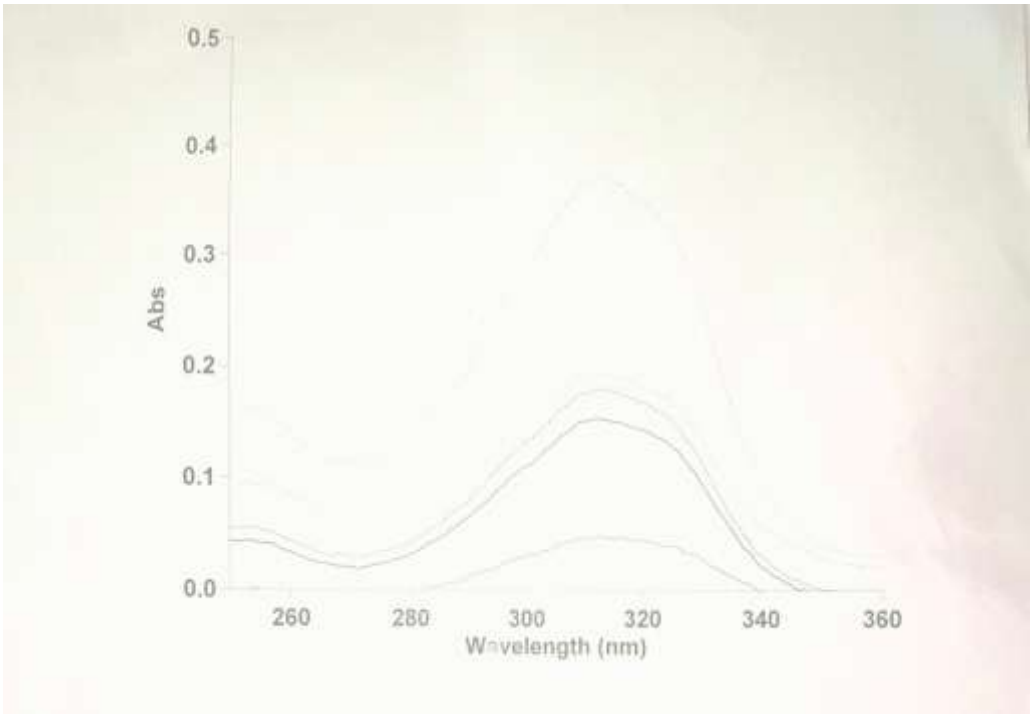
υC=Nand υC=S groups respectively prepared was identified using FT-IR Fig(2).



Fig(2) FT-IR of (LS)

2-Selection of Wavelength Range

The standard solution of(LS) (1000ppm) was scanned at different concentrations in the range of (190-400) nm and against methanol as a blank and a peak observed gives λmax was found to be (316) nm this show in fig(3).



Fig(3). Spectrum of obtaining calibration graph Zero – order spectrum of standard solution of3-(benzyliden amino)-6-methyl-2-thioxo 2,5-dihydro pyrimidine-4(3H)-one(LS)

3-Calibration Curve and Statistical Analysis

Linearity and Range five points calibration curve were obtained in a concentration range from (4-12)ppmfor3-(benzyliden amino)-6-methyl-2-thioxo 2,5-dihydro pyrimidine-4(3H)-one (LS) standard solution and recorded the one peak at (316) nm. The response of(LS) the was found to be linear in the investigation concentration range. The absorbance was plotted versus concentration fig.(4) and statistical parameters shown in table (1).

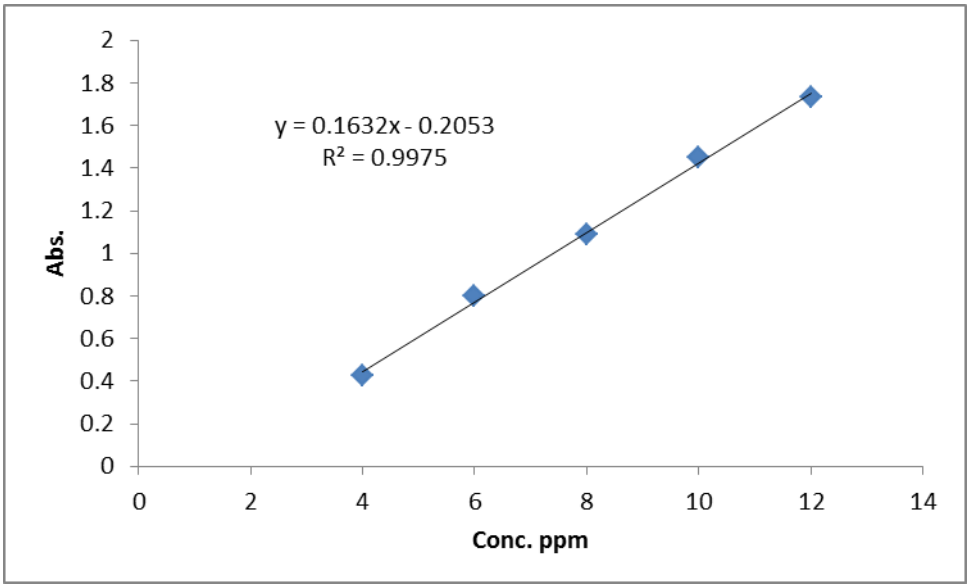


Fig 4: Calibration curve of zero-order spectra forfor3-(benzyliden amino)-6-methyl-2-thioxo 2,5-dihydro pyrimidine-4(3H)-one (LS) (4-12) ppm at peak = 316 nm

Table 1 : Statistical parameters obtain from the calibration curve for3-(benzyliden amino)-6-methyl-2-thioxo 2,5-dihydro pyrimidine-4(3H)-one (LS)

Wavelength nm	316
R^2	0.997
Linearity range(ppm)	4– 12
Equation	Y=0.163x-0.205
Slope, b	0.163
Intercept, a	0.205
Conf. limit for Slope b± t_{sb}	0.163±34.739
Conf. limit for Intercept a ± t_{sa}	0.205±5.515
Standard deviation of the slope, S_b	0.005
Standard deviation of intercept , S_a	0.04
Molar absorptivity Ɛ (L. mol^{-1}. cm^{-1})	$1.0441*10^{+3}$
Sandell's sensitivity (µg. cm^{-1})	0.02356

ANOVA: is the parsing of variance was also outstretch the linearity of calibration curve. F ratio for the 1 and 3 degree of break down is larger than the decisive value ($F_{1.3}$=10.13 at 95%CL), indicating that the predication based on the calibration curve is satisfactory table(2).

Table(2) ANOVA: analysis of variance of calibration curve

Sours		Sum of Squares Ss	df	Mean Square MS	F calculation
1	Regression	1.065	1	1.065	1206.810
	Residual	0.003	3	0.001	
	Total	1.067	4		

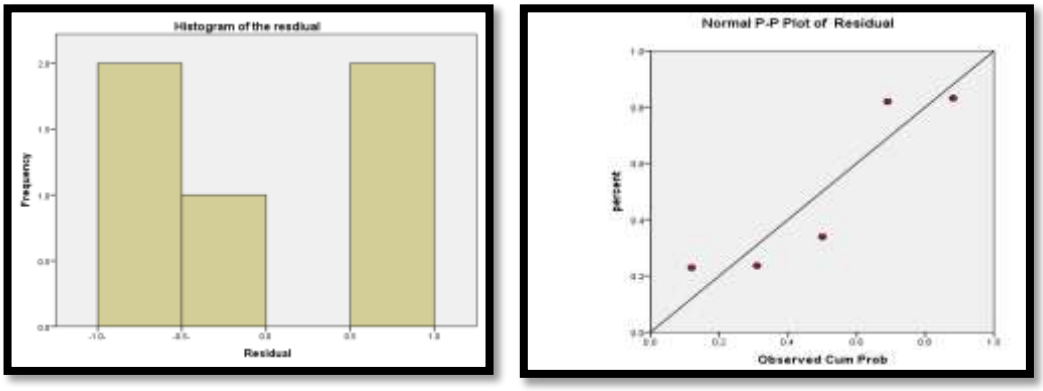


Fig 4: statistical analysis for calibration curve showing residual versus the amount of 3-(benzyliden amino)-6-methyl-2-thioxo 2,5-dihydro pyrimidine-4(3H)-one (LS)

Intermediate Precision (Interday and Intraday precision)

The prepared stock solution was subsequently diluted to get (4-12)ppm The resulting solutions absorbance was measured at wavelength of (316)nm using double beam UV spectrophotometer against blank (methanol). The experiments were repeated five times in a day to determine intraday precision and on five different days to determine inter day precision. The results of the summarized in table 3and4respectively.

Table 3: results from determination of intraday precision.

Conc.ppm	mean	SD	RSD%
4	0.4549	0.01925	4.2597
8	1.0868	0.00063	0.0579
12	1.7302	0.00148	0.0855

N=5

Table 4: results from determination of inter day precision.

Conc. ppm	mean	SD	RSD%
4	0.8946	0.00135	0.1509
8	1.2733	0.00067	0.0526
12	1.8005	0.00167	0.2071

SD= STANDAR deviation, SE= standard error of mean, RSD= relative standard of deviation, RSE = relative standard of error.

Conclusion

The aim of this search rapid, accurate, precise, simple, specific and expeditious spectrophotometric method was used for determination of Schiff bases 3-(benzyliden amino)-6-methyl-2-thioxo 2,5-dihydro pyrimidine-4(3H)-one (LS).A UV-Spectrophotometric method was developed for Safranal determination. Zero order derivative spectrophometric methods were developed for the determination of(LS) developed methods can be recommended for routine and quality control analysis of(LS)

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