

# Research and Reviews: Journal of Pharmaceutical Quality Assurance

## Novel Kinetic Spectrophotometric Method Using Sodium 1,2-Naphthoquinone-4-Sulphonate for Determination of Desloratadine in Pharmaceutical Formulations

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### Research Article

Received Date: 30/05/2015

Accepted Date: 22/06/2015

Published Date: 28/06/2015

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**Keywords:** Desloratadine, Kinetic spectrophotometry, Sodium 1,2-naphthoquinone-4-sulfonate(NQS), Pharmaceutical dosage forms.

#### ABSTRACT

New, sensitive, accurate and inexpensive kinetic spectrophotometric method was developed and validated for the determination of desloratadine (DES) in pure form and pharmaceutical preparations. The method is based on the formation of colored product between DES and sodium 1,2-naphthoquinone-4-sulphonate (NQS). The method involves the reaction of DES with NQS in presence of borax of pH 9 to form colored product with  $\lambda_{\text{max}}$  at 485 nm. The fixed time method (15 min) was adopted for constructing the calibration curve. The linearity range was found to be 2.50-37.50  $\mu\text{g/mL}$ . The limit of detection was 0.039  $\mu\text{g/mL}$ . Molar absorptivity for the method was found to be  $0.59 \times 10^4 \text{ L/mol cm}$ . The determination of DES by the fixed concentration and rate constant methods is feasible with the calibration equations obtained, but the fixed time method proves to be more applicable. Statistical treatment of the experimental results indicates that the method is precise and accurate. The developed method was found to be selective, accurate and precise. The method was successfully applied for the routine quality control analysis of DES in raw material and commercial available dosage forms.

### INTRODUCTION

Desloratadine is a non sedative, long acting antihistamine with selective peripheral H1 receptor antagonistic activity. Chemically desloratadine is 8-chloro-6,11-dihydro-11-(4-piperidinylidene)-5H-benzocyclohepta[1,2-b]pyridine. Desloratadine is slightly soluble in water, but highly soluble in methanol and propylene glycol <sup>[1]</sup>. Several methods have been described for the quantitative determination of desloratadine by high-performance liquid chromatography in different pharmaceutical preparations, either alone <sup>[2-6]</sup> or with other active ingredients <sup>[6,7]</sup>. HPTLC <sup>[8,9]</sup> and capillary electrophoresis <sup>[10]</sup> methods have been developed for the analysis of desloratadine in pharmaceutical preparations. Spectrophotometric techniques have been used for determination of desloratadine in tablets including ultraviolet spectrophotometric methods <sup>[11-13]</sup>, visible spectrophotometric methods used eosin <sup>[14]</sup>, 7,7,8,8-tetracyanoquinodimethane (TCNQ,  $\alpha$ -acceptor) <sup>[15]</sup>, 4-chloro-7-nitrobenzo-2-oxa-1,3-diazole (NBD-Cl) and 2,4-dinitrofluorobenzene (DNFB) <sup>[16]</sup> and extractive spectrophotometric method <sup>[17]</sup>. Also spectrofluorimetric methods were reported for determination of desloratadine <sup>[16,18]</sup>.

Kinetic methods have certain advantages in pharmaceutical analysis regarding selectivity and elimination of additive interferences, which affect direct spectrophotometric methods. The literature is still poor in analytical assay methods based on kinetics for the determination of desloratadine in dosage forms. Some specific advantages that the kinetic methods possess

are as follows: simple and fast methods because some experimental steps such as filtration, extraction, etc. are avoided prior to absorbance measurements, high selectivity since they involve the measurement of the absorbance as a function of reaction time instead of measuring the concrete absorbance value, other active compounds present in the commercial dosage forms may not interfere if they are resisting the chemical reaction conditions established for the proposed kinetic method and colored and/or turbid sample background may possibly not interfere with the determination process <sup>[19,20]</sup>.

Sodium 1,2-naphthoquinone-4-sulphonate (NQS) has been used as a chromogenic reagent for the spectrophotometric determination of many pharmaceutical amines. It is a popular spectrophotometric reagent due to its efficient reactivity with both primary and secondary amines, and high reaction rate <sup>[21-28]</sup>. NQS proved to be a useful and sensitive analytical derivatizing agent for spectrophotometric analysis of pharmaceuticals bearing a primary or secondary amino group, however the use of NQS for spectrophotometric determination of desloratadine was not reported yet. Therefore, there is a need for another kinetic approach to estimate desloratadine in bulk and pharmaceutical formulations. The objective of the present study was to develop a precise, accurate and validated kinetic spectrophotometric method based on the reaction of NQS with amino group of desloratadine molecule to form orange compound.

## MATERIALS AND METHODS

### Instrumentation

UV-VIS spectrophotometer from Jasco model V-530 (Japan) with 1 cm quartz cells was used under the following operating conditions: scan speed medium (400 nm/min), scan range 400-700 nm and slit width 2 nm. Spectra were automatically obtained by Jasco system software. A pH meter Consort C830 (Belgium) equipped with combined glass pH electrode and a Thornton sonicator (model T-14) were also used.

### Materials

All chemicals used were of analytical reagent grade. Sodium 1,2-naphthoquinone-4-sulphonate (NQS) from Aldrich Chemical Co., St. Louis (USA), borax from BDH (England) and methanol was purchased from Merck (Germany). Doubly distilled water was used in all experiments. Working reference standard of desloratadine (DES),  $C_{19}H_{19}ClN_2=310.82$  g/mol, was supplied by CADILA Healthcare Limited (India). Its purity was found to be 99.74%. Claramix tablets (Medico Co., Syria), Newloratadine (BPI, Syria) and Newlora (Shepha, Syria) were purchased from commercial sources in the local market, labeled to contain 5 mg desloratadine per tablet.

### Solutions

**NQS (0.2%, w/v):** Aqueous solution of 0.2% w/v NQS was prepared by dissolving the appropriate weight of NQS in water. The solution was freshly prepared and protected from light during use.

**Buffer solution pH 9.0:** Borax solution ( $5 \times 10^{-3}$  M) was prepared by dissolving 0.192 g borax in 100 mL water and adjusting to pH 9.0. Other buffer solutions were also prepared according to literature methods <sup>[29]</sup>.

**DES Stock solution:** A stock solution of DES was prepared by dissolving the appropriate weight of DES in methanol to obtain 0.5 mg/mL. This stock solution was further diluted with water to obtain working solutions in the range of 2.50-37.50  $\mu$ g/mL.

**Sample solution:** Twenty tablets containing DES were weighed and finely powdered. An accurately weighed portion of the powder equivalent to 12.5 mg of DES was dissolved in a 15 mL of methanol and mixed for about 15 min. The solution was then filtered and the filtrate was diluted in a 25 mL volumetric flask to the volume with methanol to achieve a concentration of 0.5 mg/mL. The general procedure was then followed in the concentration range of the method.

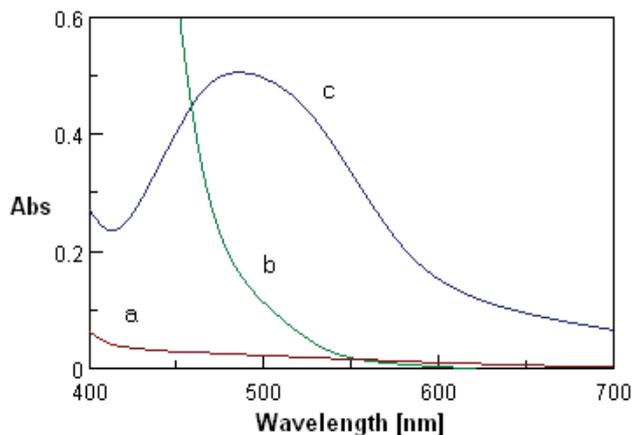
**Procedure for fixed time method:** Aliquots of 2.50-37.50  $\mu$ g/mL of standard DES solution (0.05-0.75 mL, 0.5 mg/mL) were transferred into a series of 10 mL calibrated volumetric flasks. Then 1.25 mL of 0.2% NQS solution and 0.2 mL of borax solution (pH 9.0) were added. The volume was made up to the mark with water and the absorbance was measured after 15 min at 485 nm against the reagent blank. Calibration curve was prepared and used to calculate the limit of detection (LOD) and limit of quantitation (LOQ). Concentrations of DES in the tablet samples were determined from the calibration curve or from the respective regression equation.

**Procedure for stoichiometric ratio:** The reaction stoichiometry between the studied drug and NQS has been determined spectrophotometrically by applying molar ratio and continuous variation methods. In the former method, equimolar solutions of DES and NQS ( $1 \times 10^{-4}$  M) were used. Different aliquots of NQS were added to fixed aliquots of drug solution -total volume 10 mL and the absorbance was measured at 485 nm against the reagent blank treated similarly. While in the latter method, a series of DES-NQS solutions was kept at 2.0 mL (0:2, 0.2:1.8, 0.4:1.6..... 2:0) where  $C_{DES} + C_{NQS} = 5 \times 10^{-4}$  M. The reagent was mixed in various proportions and then diluted to volume in a 10 mL calibrated flask with chloroform. The absorbance of the resulting solutions were measured at 485 nm against the reagent blank treated similarly.

## RESULTS AND DISCUSSION

### Absorption spectra

DES was found to react with NQS in an alkaline medium at 25°C producing an orange colored product which absorbs maximally at 485 nm, while the reagent blank (NQS), in alkaline medium, has practically negligible absorbance at 485 nm and DES exhibits no maximum absorption peak in the range 400-700 nm (**Figure 1**).



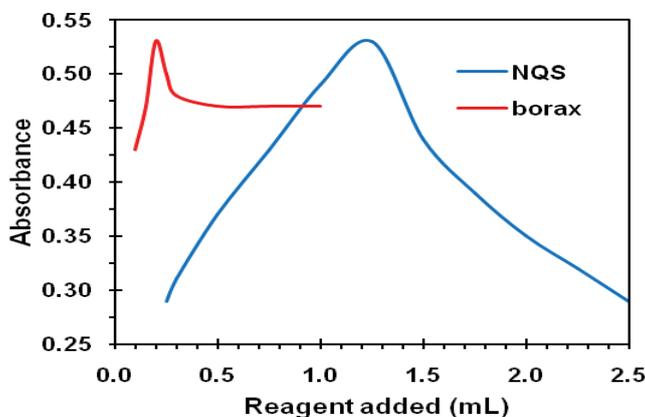
**Figure 1.** Absorption spectra of (a) DES solution (25 µg/mL) against methanol, (b) reagent blank against water and (c) DES-NQS system against reagent blank ( $C_{DES} = 25 \mu\text{g/mL} + 1.25 \text{ mL of } 0.2\% \text{ NQS} + 0.2 \text{ mL of borax } 5 \times 10^{-3} \text{ M}$  in 10 mL final volume); reaction time: 15 min.

It was found that the formation of the colored product increased with time, therefore it was deemed useful to elaborate a kinetically based method for the determination of DES. The absorbance of the chromogen remains stable for at least 15 hours. This allowed the processing of large batches of samples and their comfortable measurements with convenience. This increased the convenience of the methods as well as made it applicable for large number of samples. The following paragraphs describe the conditions under which the reaction of DES with NQS fulfills the requirements necessary for its spectrophotometric analysis.

### Optimization of reaction variables

In order to establish optimum experimental conditions, necessary for rapid and quantitative formation of colored product with maximum stability and sensitivity, the effect of various parameters such as volumes of NQS, addition of alkaline medium, reaction time and the stability of colored product were studied at room temperature.

**Effect of NQS concentration:** The effect of NQS concentration on the reaction was studied at room temperature ( $25 \pm 5^\circ\text{C}$ ). The reaction of DES with NQS was dependent on the concentration of NQS reagent. So, the reagent concentration in solution was studied by varying the NQS volume of 0.2% (w/v) NQS, while the DES concentration was maintained constant at 25 µg/mL. The study revealed that the reaction was dependent on NQS reagent. The absorbance of the reaction solution increased as the NQS concentration increased, and the highest absorption intensity was attained when the volume of NQS was 1.25 mL of 0.2% (w/v) NQS. Increasing the volume of NQS leads to decrease in the absorbance; this may be due to the high background absorbance of the reagent (**Figure 2**).



**Figure 2.** Effect of volume of NQS 0.2% in the presence of 0.2 mL of borax  $5 \times 10^{-3} \text{ M}$  and borax  $5 \times 10^{-3} \text{ M}$  in the presence of 1.25 mL of NQS 0.2% on the formation of colored product DES (25 µg/mL)-NQS, temperature:  $25 \pm 0.5^\circ\text{C}$ ; reaction time: 15 min.

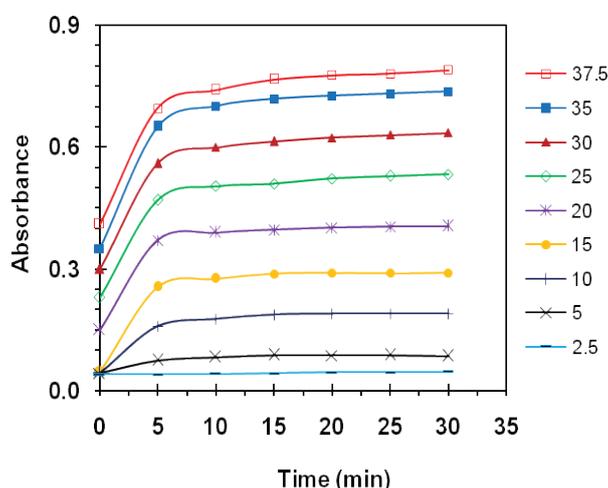
**Effect of buffer:** To generate the nucleophile from DES and activate the nucleophilic substitution reaction, alkaline medium was necessary, since the results revealed that DES does not react with NQS in acidic media. Different buffers were tested: phosphate, Britton, KCl-NaOH, borate, borax, sodium bicarbonate and sodium hydroxide solution, all prepared as aqueous

solution of a concentration range of  $3\text{-}40 \times 10^{-3}$  M. Best results were obtained in the case of higher concentrations of borax solution  $5 \times 10^{-3}$  M (Figure 2). With other bases, precipitation of white colloid occurred upon diluting the reaction solution with organic solvent, high blank readings, non reproducible results, and/or weak sensitivity were observed. Study for optimization of borax concentration revealed that the optimum volume of borax was 0.20 mL of  $5 \times 10^{-3}$  M borax (Figure 2). At this value, the amino group of DES facilitates the nucleophilic substitution reaction. At higher concentrations of borax, the absorbance of solution obviously decreased. This was attributed probably to the increase in the amount of hydroxide ion that holds back the condensation reaction between DES and NQS.

**Effect of temperature:** The effect of temperature on the reaction of DES with NQS in alkaline medium was studied at different values (20-75 °C) by continuous monitoring of the absorbance at 485 nm. It was found that the reaction with NQS was not affected by increasing the temperature, and the reaction at laboratory ambient temperature ( $25 \pm 5$  °C) went to completion within 15 min. The results revealed that increasing the temperature had negative effect on the absorption values of the reaction solution. This was probably attributed to the instability of the DES-NQS derivative. Under the aforementioned optimum conditions, increase absorbance values were observed from the beginning of the experiment up to 15 min. After this time and up to 60 min, absorbance suffered a slight increase, reaching values up to 0.5% higher than those observed after 15 min of the reaction. In view of these results, all measurements were carried out after 15 min of mixing of the reagent in order to make the method faster.

### Quantitation methods

Under the above described optimum conditions, the absorbance-time curves for the reactions of DES with NQS in alkaline medium were generated (Figure 3).



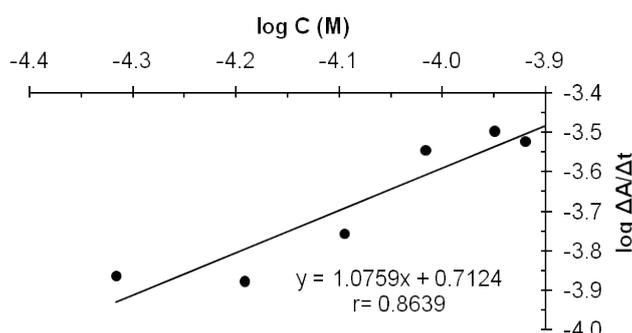
**Figure 3.** Absorbance-time curve for the reaction of DES with NQS in alkaline medium;  $C_{DES} = 2.5\text{-}37.5$   $\mu\text{g/mL}$ .

This was used as the basis for a useful kinetic method for the determination of DES. The initial rate, rate constant, variable time (fixed concentration or fixed absorbance) and fixed time methods<sup>[30,31]</sup> were tested and the most suitable analytical methods were chosen regarding the applicability, sensitivity, the values of the intercept and correlation coefficient (*r*).

**Initial rate method:** The initial rate of reaction would follow a pseudo order rate constant and obeyed the following rate equation:

$$v = \Delta A / \Delta t = k' C^n$$

Where *v* is the reaction rate, *A* is the absorbance, *t* is the measuring time, *k'* is the pseudo order rate constant, *C* is the concentration of the drug mol/L and *n* is the order of the reaction. A calibration curve was constructed by plotting the logarithm of the initial rate of reaction ( $\log v$ ) versus logarithm of drug concentration ( $\log C$ ) which showed a linear relationship over the concentration range of 15.0-37.5  $\mu\text{g/mL}$  (Figure 4).



**Figure 4.** Calibration plot of logarithm rate of the reaction against logarithm molar concentration of DES for initial rate method.

The logarithmic form of the above equation is written as follows:

$$\log v = \log \Delta A/\Delta t = \log k' + n \log C$$

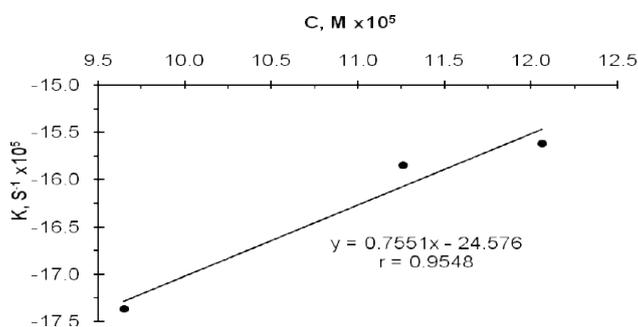
$$\log v = \log \Delta A/\Delta t = 0.7124 + 1.0759 \log C_{DES} \quad (r = 0.8639)$$

Thus,  $k' = 5.16 \text{ 1/S}$ , and the reaction is the first order ( $n = 1.0759$ ) with respect to DES concentration.

**Rate constant method:** Graphs of log absorbance versus time for DES concentration in the range of 30.0-37.5  $\mu\text{g/mL}$  ( $9.65 \times 10^{-5}$ - $12.06 \times 10^{-5} \text{ M}$ ) were plotted and all appeared to be rectilinear. Pseudo order rate constant ( $k'$ ) corresponding to different DES concentrations were calculated from the slopes multiplied by -2.303 and are presented in (Table 1) and (Figure 5). Regression of  $C$  versus  $k'$  gave the following equation:  $k' = 0.7551 C - 0.00024$  ( $r = 0.9548$ ).

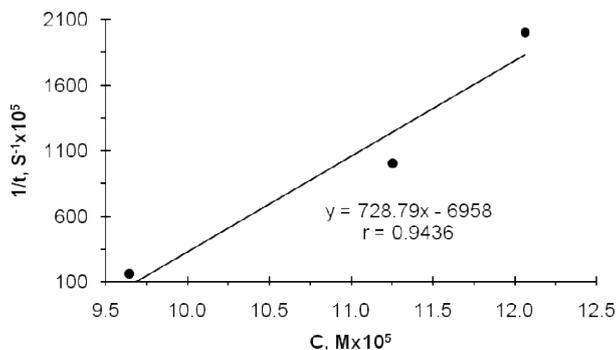
**Table 1.** Values of rate constant  $k'$  and reciprocal time taken at fixed absorbance (0.60) for different rates of variable concentration of DES.

$C_{DES} \text{ (M)}$	$k' \text{ (1/S)}$	$1/t \text{ (S}^{-1}\text{)}$
$9.65 \times 10^{-5}$	$-1.73 \times 10^{-4}$	$16 \times 10^{-4}$
$11.26 \times 10^{-5}$	$-1.58 \times 10^{-4}$	$100 \times 10^{-4}$
$12.06 \times 10^{-5}$	$-1.56 \times 10^{-4}$	$200 \times 10^{-4}$



**Figure 5.** Calibration plot of DES for rate constant method.

**Variable time (fixed absorbance) method:** Reaction rate data were recorded for different DES concentrations in the range 30.0-37.5  $\mu\text{g/mL}$ . The time was measured in the seconds at fixed value of the absorbance 0.60 (Table 1). The reciprocal of time ( $1/t$ ) versus the initial concentration of DES was plotted (Figure 6) and the following equation of calibration graph was obtained:



**Figure 6:** Calibration plot of DES for fixed absorbance method

$$1/t = 728.79 C - 0.06958 \quad (r = 0.9436)$$

The range of DES concentrations giving the most satisfactory results was limited 30.0-37.5  $\mu\text{g/mL}$  ( $9.65 \times 10^{-5}$ - $12.06 \times 10^{-5} \text{ M}$ ).

**Fixed time method:** At preselected fixed time, the absorbance of orange colored solution containing varying amounts of DES was measured at 25 °C and 485 nm. Calibration graphs were constructed by plotting the absorbance against the initial concentration of DES at fixed time 0-30 min. The regression equations, correlation coefficients and linear ranges are given in (Table 2).

**Table 2.** Calibration equations at different fixed time over the range of 2.50-37.50  $\mu\text{g/mL}$  ( $0.80 \times 10^{-5}$ - $12.06 \times 10^{-5} \text{ M}$ ) for DES.

Time (min)	Calibration equation	Correlation coefficient
0	$A = 0.0153C - 0.0141$	0.9917
5	$A = 0.0194C - 0.0180$	0.9964
10	$A = 0.0204C - 0.0185$	0.9992
15	$A = 0.0210C - 0.0187$	0.9996
20	$A = 0.0213C - 0.0203$	0.9994

25	A = 0.0215C-0.0210	0.9992
30	A = 0.0217C-0.0231	0.9991

A, absorbance; C, concentration

Correlation coefficient, intercept and slope values for the calibration data calculated using the least squares method [32]. It is clear that, both the slopes and intercepts increase with time. The most acceptable values of the correlation coefficient and more reaction products (indicated by higher absorbance readings) were obtained for a fixed time of 15 min, which was, therefore chosen as the most suitable time interval for measurements (**Table 3**). After optimizing the reaction conditions, the fixed time method was applied to the determination of DES in pure form and in pharmaceutical formulation over the concentration range of 2.50-37.50  $\mu\text{g/mL}$ . The minimum level at which the investigated compound can be reliably detected (limit of detection, LOD) and quantified (limit of quantitation, LOQ) was determined experimentally for fixed time (15 min) method. The LOD was expressed as the concentration of drug that generated a response to three times of the signal to-noise (S/N) ratio, and the LOQ was 10 times of the S/N ratio. The LOD of DES attained as defined by IUPAC [33],  $\text{LOD}_{(k=3)} = k \times S_a/b$  (where  $b$  is the slope of the calibration curve and  $S_a$  is the standard deviation of the intercept), was found to be 0.039  $\mu\text{g/mL}$ . The LOQ was also attained according to the IUPAC definition,  $\text{LOQ}_{(k=10)} = k \times S_a/b$ , and was found to be 0.16  $\mu\text{g mL}^{-1}$ .

**Table 3.** Analytical characteristics of the fixed time (15 min) method.

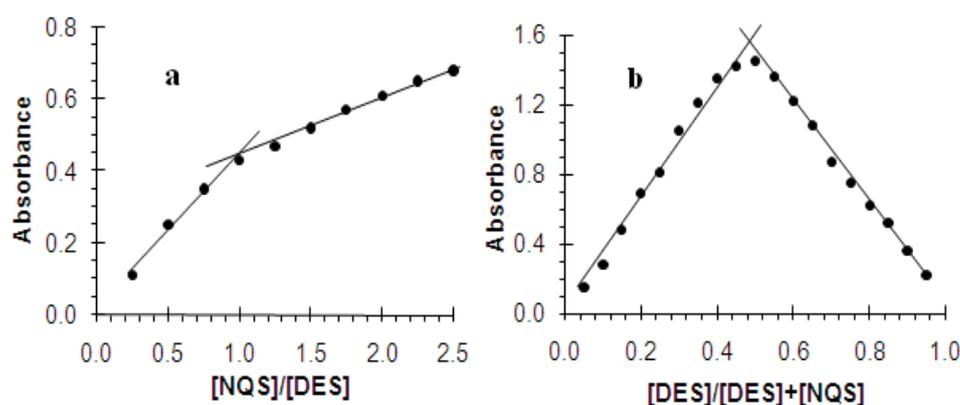
Parameters	DES
$\lambda_{\text{max}}$ (nm)	485
Beer's law range ( $\mu\text{g/mL}$ )	2.50-37.50
Ringbom optimum concentration range ( $\mu\text{g/mL}$ )	7.50-30.00
LOD ( $\mu\text{g/mL}$ )	0.039
LOQ ( $\mu\text{g/mL}$ )	0.16
Molar absorptivity (L/mol cm)	$0.59 \times 10^4$
Stability (hrs)	15
Logarithmic formation constant	6.10
Sandell's sensitivity ( $\mu\text{g/cm}^2$ per 0.001 absorbance unit)	0.10
Regression equation <sup>a</sup>	A = 0.021C-0.0187
Correlation coefficient, $r$	0.9996

<sup>a</sup>  $A = mC + b$ , where  $A$  is the absorbance and  $C$  is the concentration in  $\mu\text{g/mL}$ .

Sandell's index represents the number of micrograms or nanograms of the determinant per milliliter of a solution having an absorbance of 0.002 for the cell path length of 1 cm and is a suitable parameter for expressing and comparing the sensitivity of developed spectrophotometric method. For more accurate analysis, Ringbom optimum concentration range was calculated [34]. (Table 3) shows the analytical parameters for the determination of DES using the proposed method.

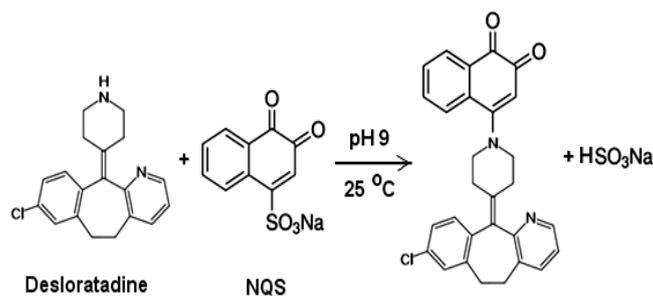
### Stoichiometry of the reaction

Under the optimum conditions, the stoichiometry of the reaction between DES and NQS was investigated by continuous variation and mole-ratio methods [35]. The stoichiometric ratio between NQS and DES was found to be 1:1 (**Figure 7**).



**Figure 7.** (a) Mole-ratio method of DES-NQS complex ( $C_{\text{DES}} = 1 \times 10^{-4}$  M) and (b) continuous variation method of DES-NQS system,  $C_{\text{DES}} + C_{\text{NQS}} = 5 \times 10^{-4}$  M.

Based on the observation stoichiometric ratio and the presence of only one center (N-H group) in DES molecule that is available for the substitution reaction, the reaction pathway was postulated to be proceeded as shown in (**Scheme 1**).



**Scheme 1.** Structures of DES and its reaction product with NQS.

## Analytical Method Validation

The accuracy and precision of the proposed method were carried out by six determinations at five different concentrations. Percentage relative standard deviation (RSD%) as precision and percentage relative error (Er %) as accuracy of the suggested method were calculated. The standard deviations, relative standard deviations, standard error and recoveries applying the fixed time method were listed in **(Table 4)**. These results of accuracy and precision show that the proposed method has good repeatability and reproducibility.

**Table 4.** Accuracy and precision for the determination of DES by the proposed method.

Taken	C <sub>DES</sub> <sup>a</sup> µg/mL		SD	Er (%)	RSD (%)	Recovery (%)
	Found <sup>a</sup>					
5.00	5.03		0.09	0.59	1.79	100.60
10.00	10.10		0.13	0.99	1.29	101.00
20.00	19.96		0.16	-0.20	0.80	99.80
30.00	30.14		0.15	0.46	0.50	100.47
35.00	35.09		0.18	0.26	0.51	100.26

<sup>a</sup>Average of six determinations

## Application to the pharmaceutical dosage forms

The proposed method was applied for determination of DES in its pharmaceutical formulations. The results of analysis of the commercial dosage forms are shown in **(Table 5)**.

**Table 5.** Determination of DES in tablets by the proposed and official methods.

Formulation	% Found $\pm$ SD <sup>a</sup>	
	Proposed method	Official method
Claramix <sup>b</sup> (5 mg/tablet)	100.30 $\pm$ 0.66	100.20 $\pm$ 0.96
	$t=0.95$	$t=1.90$
	$F=0.47$	
Newloratadine <sup>c</sup> (5mg/tablet)	101.35 $\pm$ 1.65	101.02 $\pm$ 1.60
	$t=1.82$	$t=2.09$
	$F=1.06$	
Newlora <sup>d</sup> (5mg/tablet)	100.30 $\pm$ 1.25	100.08 $\pm$ 0.74
	$t=0.56$	$t=2.16$
	$F=2.85$	

<sup>a</sup> Five independent analyses. Theoretical values for  $t$  and  $F$ -values at five degree of freedom and 95% confidence limit are  $t = 2.776$  and  $F = 6.26$ . <sup>b</sup> Supplied by Medico Co., <sup>c</sup> supplied by BPI and <sup>d</sup> supplied by Shepha, Syria.

The average percent recoveries obtained, indicated good accuracy of the method. The result obtained for the analysis of the studied drug in drug formulations employed was compared with those obtained with the official method **(Table 5)**<sup>[29]</sup>. The Student  $t$ -test and  $F$ -test values of 95% confidence level for five degrees of freedom<sup>[32]</sup> did not exceed the theoretical values indicating no significant difference between the accuracy and the precision of the two methods. The proposed method has the advantages of being virtually free from interferences by excipients such as glucose, lactose, and starch or from common degradation products.

## CONCLUSION

The developed kinetic spectrophotometric method for the determination of DES was sensitive, accurate and precise and hence can be used for the routine analysis of DES in bulk and pharmaceutical formulations with a limit of detection of 0.039 µg/mL. The method was based on the coupling reaction of DES with NQS. The fixed time method for the proposed kinetic spectrophotometric method can be easily applied to the determination of DES in its pure form and tablets. The proposed method is compared with the previously reported methods in terms of accuracy and precision. The proposed method is more selective and higher sensitivity than the sophisticated spectrophotometric techniques and similar reported methods and has a wider range of

linearity. The sample recoveries from all formulations were in good agreement with their respective label claims, which suggested non-interference of formulations excipients in the estimation. Therefore, the proposed method can be further applied for routine quality control laboratories, as alternatives for the existing methods.

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