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Research Article

UTILITY OF REDOX REACTION FOR SIMPLE SPECTROPHOTOMETRIC METHOD FOR THE DETERMINATION OF DESLORATADINE IN DOSAGE FORMS

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ABSTRACT

A Precise, accurate, rapid, simple, and sensitive spectrophotometric procedures for the determination of desloratadine (DSL) in pure sample and in dosage forms has been developed. The procedures are based on oxidation of the drug by ceric ammonium sulphate and/or *N*-bromosuccinimide in acidic medium and determination of the excess oxidant by measuring the decrease in absorbance for two different dyes; amaranth (AM) and methyl red (MR), at a suitable λ_{max}. Regression analysis of Beer's plots showed good correlation in the concentration ranges 2.0-20 µg mL^{-1}. The apparent molar absorbtivity, Sandell sensitivity, detection and quantitation limits were calculated. For more accurate results, Ringbom optimum concentration ranges were 4.0-16 µg mL^{-1}. The validity of the proposed procedure was tested by analyzing in bulk and in dosage forms containing DSL. Statistical analysis of the results reflects that the proposed methods are precise, accurate and easily applicable for the determination of DSL in pure form and in pharmaceutical dosage forms compared with the Official method.

INTRODUCTION

Desloratadine (DSL), 4-(8-chloro-5,6-dihydro-11H-benzo-[5,6]cyclohepta [1,2b] pyridin-11-ylidene)-1-piperidine, is the descarboethoxy form of loratadine) (Fig. 1), is a selective peripheral H1 receptor antagonist, devoid of any substantial effect on the central and autonomic nervous systems [1,2]. Desloratadine exhibits qualitatively similar pharmacodynamic activity with a relative oral potency in animals, two to three-fold greater than its parent analogue loratadine, probably due to a higher affinity for histamine H1 human receptors [3]. Nevertheless, the development of drugs with increased potency will continue to challenge the analytical chemist to lower the limit of quantitation (LOQ).

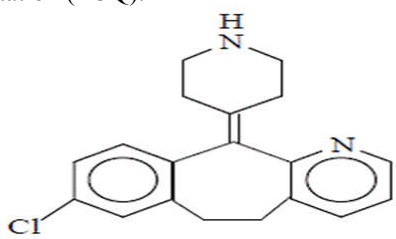

Figure 1 Chemical structure of Desloratadine

Several analytical methods have been reported for the determination of DSL in biological samples and applied in pharmacokinetic studies.

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These methods include gas chromatography with nitrogen phosphorous detection [4], liquid chromatography with fluorescence detection [5,6] ultraviolet detection [7], or mass spectrometric detection [8-11] However, DSL was determined in pharmaceutical preparations using a liquid chromatographic method with UV detection [12-14] and capillary isotachophoresis [15]. As DSL has no functional group that enables absorption in the visible region, we decided to analyze the drug through redox reactions.

In this approach, two different labeling agents, namely; Ceric ammonium sulphate (CAS) and N-bromosuccinamide (NBS), have been used in redox reactions based on their ocidation with the free secondary amino group in the piperidine ring of DSL. The proposed methods are highly specific for desloratadine in the presence of the parent drug (loratadine). Both reagents are well known to oxidize with primary and secondary amines and the excess of oxidants were determined with amaranth and methyl red. In the present work, the two colored reaction products obtained with AM and MR were measured spectrophotometrically. The four developed procedures were applied for the determination of DSL in tablets without any interference from the excipients. A low LOQ is a common requirement to support a clinical development program and when this is coupled with a simple procedure for sample preparation and feasible analytical tool, they would be the requirements of an optimized analytical method. Although the previously reported methods that were applied in pharmacokinetic studies of desloratadine possess the required low LOQ, they lack the simple sample preparation

and method feasibility that were obtained in our spectrophotometric methods where a low LOQ of 12 μg mL^{-1} was attained which allowed the analytical determination of DSL in spiked and real human plasma. The proposed procedure was used when modern and expensive apparatus such as GLC, HPLC and HPTLC are not present.

MATERIALS

Apparatus

A pH-meter Orion-Research Model 601 A/Digital Ionalyzer was used for adjusting the pH of solution. All the absorption spectral measurement were made using JASCO v-530 (UV-VIS) spectrophotometer (Japan), with scanning speed 400 nm min^{-1} and band width 2.0 nm, equipped with 10 mm matched quartz cells.

Reagents and standard solutions

All chemicals used were of analytical or pharmacopoeia grade purity and water was bidistilled. Standard DSL was obtained from Delta Pharma Company, Egypt, its potency was 99.99 ± 0.39%. Stock DSL solution (100 μg ml^{-1}) was prepared by dissolving 0.01 g in water and adjusted to 100 ml with bidistilled water. Working solutions of lower concentration were prepared by serial dilutions.

Aqueous solutions of 2.0×10^{-3} M AM and/or MR (Sigma Aldrich), were prepared by dissolving an appropriate weight in 100 mlL bidistilled water.

A stock solution of 1.0 M H_2SO_4 was prepared by adding 5.4 mL of concentrated acid (Merck, Darmstadt, Germany, 98%, Sp. Gr. 1.84) to bidistilled water, cooled to room temperature, transfer to 100 mL with measuring flask, diluted to the mark and standardized as recorded [16].

A stock solution of 100 μg mL^{-1} NBS (Sigma-Aldrish) was freshly prepared by dissolving about 0.01 g of NBS in least amount of warm bidistilled water in a 100 mL measuring flask and then diluted to the mark with bidistilled water and standardized [17]. The solution was kept in an amber colored bottle and was diluted appropriately to get 100 μg mL^{-1} NBS for use in all methods. The NBS solution was stored in a refrigerator when not in use. A 1.0% w/v KBr solution was also prepared by dissolving 1.0 g of KBr in 100 mL water.

A stock solution of 2×10^{-3} M cerium (IV) ammonium sulphate (CAS) (E-Merk, Darmstadt, Germany) was freshly prepared by dissolving appropriate weight of CAS in the least amount of H_2SO_4 (2.0 mol L^{-1}) then completed to the mark in a 100 mL calibrated flask with the same acid and kept in a dark bottle and a refrigerator when not in use.

Recommended procedure using CAS

Different aliquots (0.2-2.0 mL) of a standard 100 μg mL^{-1} DSL solution using AM procedure, were transferred into a series of 10 mL calibrated flasks followed by adding 2.0 mL of 1.0 M H_2SO_4 and 2.0 mL of (2×10^{-3} M) CAS solution. The flasks were stoppered and the contents were mixed well and the flasks were kept aside for 5.0 min at 60 ± 1.0 ^{o}C with occasional shaking. Finally, 1.0 mL of 2×10^{-3} M MR, dye solution was added to each flask and mixed well, and then the volume was diluted to the mark with bidistilled water. The decrease in color intensity of dyes were measured after 2.0 min against reagent blank solution treated similarly omitting the drug, at their corresponding λ_{max} 546 nm. The concentration of unknown was determined in each case from calibration graph which obtained by plotting the concentration of DSL against absorbance.

Recommended procedures using NBS

Different aliquots (0.1-1.4 mL), of a standard 100 μg mL^{-1} DSL solution using amaranth, and methyl red methods, respectively, were transferred into a series of 10 mL calibrated flasks by means of a micro burette. To each flask 1.25 mL each of 5.0 M HCl; 2.0 mL of NBS solution (100 μg mL^{-1}) and 1.0 mL of 1.0% (w/v) KBr were added successively. The flasks were stoppered, content mixed and the flasks were kept aside for 5.0 min with occasional shaking. Finally, 0.7 and 1.0 mL of 2×10^{-3} M amaranth and methyl red solution, respectively, were added to each flask and mixed well and then the volume was diluted to the mark with water. The absorbance of each solution was measured at 556, and 560 nm for amaranth, and methyl red methods, respectively, after 2.0 min against a reagent blank.

Procedure for pharmaceutical formulations (tablets)

The contents of twenty tablets of each drug were weighed accurately and ground into a fine powder. An accurate weight of the powdered tablets equivalent to 20 mg DSL was dissolved in bidistilled water with shaking for 5.0 min and filtered using a Whatman No. 42 filter paper. The filtrate was diluted to the mark with bidistilled water for DSL in a 100 mL measuring flask to give 200 μg mL^{-1} stock solution of DSL for analysis by spectrophotometric methods. A convenient aliquot was then subjected to analysis by the spectrophotometric procedures described above. Determine the nominal content of the tablets using the corresponding regression equation of the appropriate calibration graph.

RESULTS AND DISCUSSION

Absorption spectra

Many dyes are irreversibly destroyed to colorless species by oxidizing agents in acid medium [18]. The proposed spectrophotometric procedures are based on the reaction between DSL and measured excess of CAS and/or NBS, and subsequent determination of the latter by reacting it with a fixed amount of amaranth, and methyl red dye and measuring the absorbance at their λ_{max}. These procedures make use of the bleaching action of CAS and/or NBS on the dyes, the decolorization being caused by the oxidative destruction of the dyes. DSL when added in increasing concentrations to a fixed concentration of CAS and/or NBS consumes the latter and there will be a concomitant decrease in the concentration of oxidant. When a fixed concentration of either dye is added to decreasing concentrations of CAS and/or, NBS a concomitant increase in the concentration of dye is obtained. Consequently, a proportional increase in the absorbance at the respective λ_{max} is observed with increasing concentrations of DSL.

Chemistry of the reactions

Because of its high oxidation potential and excellent solution stability, cerium(IV) ammonium sulphate, has been widely used as an effective analytical reagent in spectrophotometric methods for the determination of many pharmaceutical compounds [19-23]. NBS is a strong oxidizing or brominating agent and perhaps the most important positive bromine containing organic compound used to determine many pharmaceutical compounds [24-28]. It is also used for the

specific purpose of brominating alkenes at the allylic position [29].

The analytical reactions involved two steps; the first one was concerned with the oxidation of the investigated drugs with a known excess amount of oxidant in acid medium. The second step involved the determination of the excess residual of oxidant via its reaction with a fixed amount of both amaranth, or methyl red dyes and measuring the absorbance at the respective λ_{max}. The tentative reaction scheme of spectrophotometric procedures is shown in the following equations. In all procedures, the absorbance increased linearly with increasing concentration of drugs. The latter procedures make use of the bleaching action of CAS and/or NBS on dyes, the discoloration being caused by the oxidative destruction of the dye.

DSL + known excess of NBS(IV) + H^+ ⟶ oxidation product of DSL + NBS(III)

Unreacted Ce(IV) + known excess AM ⟶ oxidation product of dye + AM

Selection of acid type and concentration

The reaction between DSL with CAS and/or NBS were performed in different acid media CH_3-COOH, HCl, HNO_3, and H_2SO_4 solutions. Better results were suitable in sulfuric hydrochloric acid medium using CAS, whereas hydrochloric acid medium was the optimum on using NBS. The effect of H_2SO_4 and /or HCl concentration on the redox reaction between DSL and CAS and/or NBS were studied by varying the concentration of acid keeping the concentrations of CAS and/or NBS and drug fixed. The reaction was found to be rapid yielding a constant absorbance with maximum sensitivity and stability when the HCl concentration was 5.0 M and maintained in the range of 0.25−3.0 mL of HCl (5.0 M) in a total volume of 10 mL. The results indicated that, at 1.0-1.5 mL of HCl (5.0 M), there were almost same absorbance values were obtained in the presence of DSL, the absorbance values obtained were constant and were almost the same as those of the reagent blank. At the acid volumes less than 1.0 mL, reaction led to go slower and incomplete. Therefore, 1.25 mL of HCl (5.0 M) was used though out the study for both drug.

The effect of H_2SO_4 concentration on the redox reaction between DSL and CAS was studied by varying the concentration of acid keeping the concentrations of CAS and drug fixed. The reaction was found to be rapid yielding a constant absorbance with maximum sensitivity and stability when the H_2SO_4 concentration was 1.0 M and maintained in the range of 0.5−3.0 mL of H_2SO_4 (1.0 M) in a total volume of 10 mL. The results indicated that, at 2.0-2.5 mL of H_2SO_4 (1.0 M), there were almost same absorbance values were obtained in the presence of DSL, the absorbance values obtained were constant and were almost the same as those of the reagent blank. At the acid volumes less than 1.5 mL, reaction led to go slower and incomplete. Therefore, 2.0 mL of 1.0 M H_2SO_4 was used though out the study for both drug.

Effect of CAS and/or NBS concentration

To investigate the optimum concentration of CAS and/or, NBS different concentrations were treated in the range of 0.25−3.0 mL with a fixed concentration dyes in H_2SO_4 and/or HCl medium and the absorbance was measured at the optimum wavelength. It was found that maximum color intensity of the products was achieved with 2.0 mL of CAS (2×10^{-3} M), whereas NBS for the higher absorbance is obtained with 2.0 mL of 100 µg mL^{-1} were selected for all further studies due to high concordant results obtained (Fig. 1).

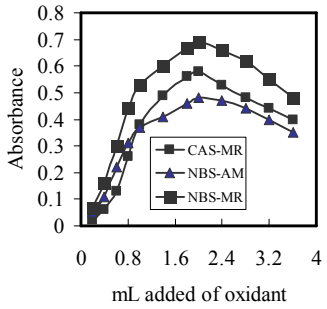

Figure 1 Effect of of volume of CAS 2×10^{-3} M and NBS (100 µg mL^{-1}) on the oxidation product of DSL using AM and MR dyes in H_2SO_4 and HCl media, respectively

Effect of KBr concentration

The effect of KBr concentration was studied in the range of 0.5-2.5 mL. 1.0 mL of 1.0% (w/v) KBr was chosen as an optimum volume to accelerate the oxidation process on using NBS, whereas it has no effect on using CAS.

Effect of dye concentration

The effect of amaranth, or methyl red concentration on the intensity of the color developed was carried out to obtain the optimum concentration of dyes that produces the maximum and reproducible color intensity by reducing the residual of CAS and/or NBS. The effect of dye concentration was studied in the range of 0.25-3.0 mL of each dye (2×10^{-3} M). It was found that maximum color intensity of the oxidation products was achieved with 1.0 and 0.7 mL of 2×10^{-3} M of AM for both CAS and NBS, respectively, whereas 1.0 mL of 2×10^{-3} M MR was used for NBS (Figure 2).

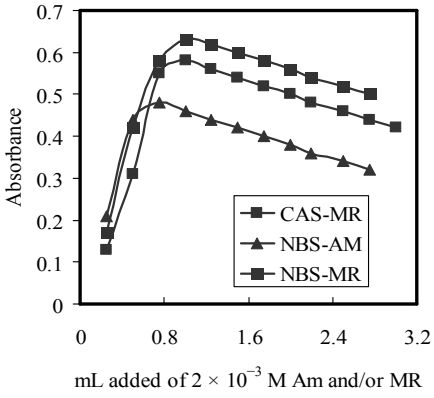

Fig. 2 Effect of volume of dyes (2×10^{-3} M) of the oxidation product of DSL using CAS and/or NBS and dyes at the other optimum cinditions

Effect of temperature and mixing time

The effect of temperature was studied by heating a series of sample and blank solutions at different temperatures ranging from 25 to 60 °C in water bath. It was found that raising the temperature does not accelerate the oxidation process and does not give reproducible results on using NBS as oxidant, so maximum color intensity was obtained at room temperature (25 ± 2 °C). On using CAS the redox reaction increase by increasing temperature upto 60 ± 1.0 °C. The effect of mixing time required completing oxidation of the studied drugs and for reducing the excess oxidant was studied by measuring the absorbance of sample solution against blank solution prepared similarly at various time intervals 2.0-20 min. It was found that

the contact times gave constant and reproducible absorbance values at 5.0 min at room temperature (25 ± 2 °C) for using NBS and (60 ± 1.0 °C) using CAS. The time required for complete oxidation of the drug is not critical and any delay up to 15 min in the determination of unreacted CAS and/or NBS had no effect on the absorbance. A 2.0 min standing time was found necessary for the complete bleaching of the dye color by the residual CAS and/or NBS was found necessary for complete reduction of residual CAS and/or NBS by the examined dyes and the absorbance of the unreacted dye was stable for at least 6.0 h.

Effect of sequence of addition

The optimum sequence of addition was drug-acid-oxidant and then dye. Other sequences gave lower absorbance values under the same conditions.

Method validation

The proposed procedures have been validated for linearity, sensitivity, accuracy, precision, selectivity and recovery. Linearity and sensitivity under the optimum conditions a linear correlation was found between absorbance at λ_{max} and the concentration of DSL in the ranges of 2.0−20 μg mL^{-1}. The calibration graph is described by the equation:

$$A = a + b\,C \quad (1)$$

Where A= absorbance, a= intercept, b= slope and C= concentration in μg mL^{-1}, obtained by the method of least squares. Correlation coefficient, intercept and slope of the calibration data are summarized in Table 1. For accurate determination, Ringbom concentration range [30] was calculated by plotting log concentration of drug in μg mL^{-1} against transmittance % from which the linear portion of the curve gives an accurate range of microdetermination of NVH and represented in Table 1.

Table I Analytical and regression parameters of proposed oxidation spectrophotometric methods for determination of DSL

Parameters	CAS	NBS	
	MR	AM	MR
λ_{max}, (nm)	546	546	526
Beer's law limits, μg mL^{-1}	2.0-12	2.0-12	2.0-20
Ringboom limits, μg mL^{-1}	4.0-10	4.0-10	4.0-16
Molar absorptivity, x 10^4 L mol^{-1} cm^{-1}	2.0956	2.0956	0.7922
Sandell sensitivity, ng cm^{-2}	26.8	26.8	49.15
Regression equation[a]			
Intercept (a)	0.0056	- 0.006	0.004
SD of intercept (S_a)	0.009	0.012	0.017
Slope (b)	0.0358	0.0322	0.0279
SD of slope (S_b)	0.018	0.067	0.039
Correlation coefficient, (r)	0.9993	0.9996	0.9988
Mean ± SD	100.81±1.06	101.21 ± 1.6	99.73 ± 1.25
RSD%	1.05	1. 20	1.31
RE%	1.10	1.32	1.36
Limit of detection, μg mL^{-1}	0.59	0.52	1. 85
Limit of quantification, μg mL^{-1}	1.97	1.97	1.93
Calculated *t*-value[b]	1.03	1.40	1.68
Calculated *F*-value[b]	3.58	3.44	3.79

[a] $A = a + bC$, where C is the concentration in μg mL^{-1}, A is the absorbance units, a is the intercept, b is the slope.
[b] The theoretical values of t and F are 2.57 and 5.05, respectively at confidence limit at 95% confidence level and five degrees of freedom (p= 0.05).

Sensitivity parameters such as apparent molar absorptivity and Sandell's sensitivity values, as well as the limits of detection and quantification, were calculated as per the current ICH guidelines [31] and illustrated in Table 1. The high molar absorptivity and lower Sandell sensitivity values reflect the good and high sensitivity of the proposed methods. The validity of the proposed methods was evaluated by statistical analysis [32] between the results achieved from the proposed methods and that of the reported method.

Regarding the calculated Student's t-test and variance ratio F-test (Table 1), there is no significant difference between the proposed and reported method [6,7] regarding accuracy and precision. The limits of detection (LOD) and quantification (LOQ) were calculated according to the same guidelines using the formulas [31, 32]:

$$LOD = 3.3\,\sigma/s \text{ and } LOQ = 10\,\sigma/s \quad (2)$$

Where σ is the standard deviation of two reagent blank determinations, and s is the slope of the calibration curve.

Accuracy and precision

In order to evaluate the precision of the proposed methods, solutions containing four different concentrations of DSL were prepared and analyzed in six replicates. The analytical results obtained from this investigation are summarized in Table 2.

Table II Results of intra-day and inter-day accuracy and precision study for DSL obtained by the proposed methods.

Method	Taken (μg mL^{-1})	Recovery %	Precision RSD % [a]	Accuracy RE %	Confidence Limit [b]
Intra-day using Ce(IV)					
MR	3.0	99.40	0.79	-0.60	2.982 ± 0.025
	6.0	100.40	1.04	0.40	6.024 ±0.066
	9.0	99.70	1.52	-0.30	8.973 ± 0.143
Intra-day using NBS					
AM	3.0	99.10	1.10	-0.90	2.973 ± 0.034
	6.0	99.20	1.40	-0.80	5.952 ± 0.087
	9.0	99.60	1.60	-0.40	8.964 ± 0.151
MR	4.0	99.20	0.77	-0.80	3.968 ± 0.031
	8.0	100.20	0.98	0.20	8.016 ± 0.082
	12	100.50	1.76	0.50	12.06 ± 0.223
Inter-day using Ce(IV)					
MR	5.0	99.00	0.90	-1.0	4.95 ± 0.047
	10	99.30	1.20	-0.70	9.93 ± 0.125
	15	99.50	1.70	-0.50	14.93 ± 0.266
Inter-day using NBS					
AM	3.0	99.30	0.90	-0.70	2.979 ± 0.038
	6.0	99.50	1.36	-0.50	5.97 ± 0.085
	9.0	100.40	1.65	0.40	9.036 ± 0.156
MR	3.0	99.30	0.90	-0.70	2.979 ± 0.038
	6.0	99.50	1.36	-0.50	5.97 ± 0.085
	9.0	100.40	1.65	0.40	9.036 ± 0.156

[a] RSD %, percentage relative standard deviation; RE %, percentage relative error.
[b] Mean ± standard error.

Lower values of the relative standard deviation (RSD%) and percentage relative error (RE%) indicate the precision and accuracy of the proposed methods. The assay procedure was repeated six times, and RSD % values were obtained within the same day to evaluate repeatability (intra-day precision) and over five different days to evaluate intermediate precision (inter-day precision). For the same concentrations of drugs inter- and intra-day accuracy of the methods was also evaluated. The percentage recovery values with respect to found concentrations of each drug were evaluated to ascertain the accuracy of the methods. The recovery values close to 100 % as compiled in Table 2 shows that the proposed methods are very accurate.

Robustness and ruggedness

For the evaluation of method robustness, volume of HCl and/or H_2SO_4 was slightly altered (± 0.2 mL) and the reaction time (after adding NBS and/or CAS, time varied was 5.0 ± 2.0 min) were slightly varied deliberately in the proposed methods. The analysis was performed with altered conditions by taking three different concentrations of drugs and the methods were found to remain unaffected as shown by the RSD values in the ranges of 0.85−2.15% for DSL. procedures ruggedness was expressed as the RSD of the same procedure applied by two different analysts as well as using three different instruments (spectrophotometers). The inter-analysts RSD were in the ranges 0.95−2.50% DSL, whereas the inter-instruments RSD ranged from 0.85−2.65% DSL suggesting that the developed methods were rugged. The results are shown in Table 3.

Table III Results of method robustness and ruggedness (all values in RSD%) studies for DSL.

Methods	Nominal amount concentration (µg mL^{-1})	RSD%			
		Robustness		Ruggedness	
		Variable alerted [a,b]			
		Acid Volume (n=3)	Reaction Time (n=3)	Different analysts (n=3)	Different instruments (n=3)
		NBS[a]			
AM	3.0	1.05	0.90	1.15	1.05
MB	6.0	1.40	1.70	1.90	1.80
	9.0	2.10	2.25	2.40	2.30
	3.0	1.10	1.08	0.93	0.96
	6.0	1.60	2.10	1.60	1.87
	9.0	2.0	2.40	2.20	2.50
		CAS[b]			
MB	3.0	1.10	1.08	0.93	0.96
	6.0	1.60	2.10	1.60	1.87
	9.0	2.0	2.40	2.20	2.50

[a] Volume of (5.0 M) HCl is (1.25 ± 0.2 mL) and reaction time is (5.0 ± 2.0 min) (after adding NBS) were used.
[b] Volume of (1.0 M HCl is (2.0 ± 0.2 mL) and reaction time is (5.0 ± 2.0 min) (after adding CAS) were used.

Recovery studies

To ascertain the accuracy, reliability and validity of the proposed methods, recovery experiment was performed through standard addition technique. This study was performed by spiking three different levels of pure drugs (50, 100 and 150% of the level present in the tablet) to a fixed amount of drugs in tablet powder (pre-analysed) and the total concentration was found by the proposed methods.

Table IV Results of recovery experiments by standard addition method for the determination of DSL, in tablets using the proposed methods.

Samples	Taken drug in Tablet (µg mL^{-1})	Pure drug Added (µg mL^{-1})	AM		MR	
			found (µg mL^{-1})	Recovery [a] (%) ± SD	found (µg mL^{-1})	Recovery [a] (%) ± SD
Aerius tablets using NBS	4.0	2.0	5.976	99.60 ± 0.40	5.94	99.00 ± 1.40
		4.0	7.976	99.70 ± 0.72	8.056	100.70 ± 0.88
		6.0	10.02	100.20 ± 0.86	9.96	99.60 ± 1.10
		8.0	12.15	101.25 ± 0.89	11.90	99.17 ± 1.54
		10	14.20	101.43± 1.35	13.70	97.86 ±1.76
Aerius tablets using CAS	4.0	2.0			6.048	100.80 ± 0.50
		4.0			7.968	99.60 ± 0.76
		6.0			9.91	99.10 ± 1.25
		8.0			12.15	101.25 ± 0.59
		10			13.75	98.21 ± 1.52

[a] Average of six determinations.

The determination with each level was repeated three times and the percent recovery of the added standard was calculated. The results of this study presented in Table 4 revealed that the accuracy of the proposed methods was unaffected by the various excipients present in tablets which did not interfere in the assay.

Application of pharmaceutical formulations (tablets)

The proposed methods were applied to determine DSL in pharmaceutical formulations (tablets). The results in Table 5 showed that the methods are successful for the determination of DSL and that the excipients in the dosage forms do not interfere.

Table V Results of analysis of tablets by the proposed methods for the determination of DSL, and statistical comparison with the reference methods.

Samples	Recovery [a] (%) ± SD		Reported methods [7]
	Proposed Method using Ce(IV)		
	AM	MR	
	NBS		
Aerius tablets	99.20 ± 0.80	99.60±0.51	99.50 ± 1.18
t-value [b]	0.80	0.78	
F-value [b]	1.32	1.83	
	CAS		
Aerius tablets		99.50 ± 0.72	99.76±0.54
t-value [b]		0.64	
F-value [b]		1.77	

A statistical comparison of the results obtained from the assay of DSL by the proposed methods and the reported methods [6,7] for the same batch of material is presented in Table 5. The results agree well with the label claim and also were in agreement with the results obtained by the reported methods [6.7]. When the results were statistically compared with those of the reported methods by applying the Student's t-test for accuracy and F-test for precision, the calculated t-value and F-value at 95% confidence level did not exceed the tabulated values for five degrees of freedom [32]. Hence, no significant difference between the proposed methods and the reported methods at the 95 % confidence level with respect to accuracy and precision.

CONCLUSION

Two new, useful simple, rapid and cost-effective spectrophotometric methods have been developed for determination of DSL in bulk drugs and in its tablets using CAS and/or NBS as oxidizing agent and validated as per the current ICH guidelines. The present spectrophotometric methods are characterized by simplicity of operation, high selectivity, comparable sensitivity, low-cost instrument, they do not involve any critical experimental variable and are free from tedious and time-consuming extraction steps and use of organic solvents unlike many of the previous methods reported for DSL. The assay methods have some additional advantages involve less stringent control of experimental parameters such as the stability of the colored system, accuracy, reproducibility, time of analysis, temperature independence and cheaper chemicals. These advantages encourage the application of the proposed methods in routine quality control analysis of NVH in pure and dosage forms.

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