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CHEMICAL FORCE DEGRADATION ASSAY METHOD EVALUATION OF CAMEL MILK IN MARKETED (ADVIK) DRY POWDER DOSAGE FORM

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ABSTRACT

After a series of experimental attempts, it is possible to accomplish separation under optimal circumstances. A stationary phase column, such as the Hypersil BDS C18 (100 mm x 2.1 mm, 1.7 m), was the best choice since it generated symmetrical peaks with great resolution and sensitivity. The flow rate was kept constant at 0.5 mL min-1, indicating acceptable resolution. The PDA detector response of Camel Milk Dry powder was investigated, and the optimal wavelength with the maximum sensitivity was discovered to be 230 nm. At 0.5mL/min, a combination of two solutions, Chloroform and Methanol in the ratio of 30:70 percent v/v," was found to be an acceptable mobile phase for separation of Camel Milk Dry powder. The temperature in the column was kept at room temperature.

KEYWORDS: Camel Milk Dry powder, Chloroform and Methanol.

INTRODUCTION

Camel Milk has the potential to be a lucrative source of raw material for many dairy powder manufacturers, and it is becoming increasingly popular. The majority of observers have focused their attention on the composition of Camel Milk powder, as well as its capacity solidity and capabilities. Other Milk powders, however, such as camel Milk powder, are a cause of extreme instability. In fact, because of its therapeutic and nutritional properties, camel milk is the most often consumed milk in dry and semi-arid regions across the globe.

Validation of Analytical Methods (USP/ICH)

Method validation, according to the United States Pharmacopeia (USP), is performed to ensure that an analytical methodology is accurate, specific, reproducible, and rugged over the specified range that an analyte will be analyzed. Regulated laboratories must perform method validation in order to be in compliance with FDA regulations. In a 1987 guideline (Guideline for Submitting Samples and Analytical Data for Methods Validation), the FDA designated the specifications in the current edition of the USP as those legally recognized when determining compliance with the Federal Food, Drug and Cosmetic Act can be referred to as the "eight steps of method validation"

EXPERIMENTAL METHODOLOGY

Method Validation

The analytical procedure refers to the way of performing the analysis. It should describe in detail the steps necessary to perform each analytical test. This may include but is not limited to: the sample, the reference standard and the reagents preparations, use of the apparatus, generation of the calibration curve, use of the formulae for the calculation, etc. The described method extensively validated in terms of specificity, system suitability, linearity, accuracy, precision, limit of detection, limit of quantification and robustness.

- **Forced degradation studies of our selected pharmaceutical drugs**

In order to establish the analytical method for a stability indicating method, the drugs are subjected to various stress conditions to conduct forced degradation studies. Stress studies were carried out under the conditions of acid/base hydrolysis, oxidation, reduction, in accordance with ICH Q1A (R2). Several trials with different severity of each stressed condition are to be conducted, so that upto 10-30% degradation is to be achieved.

Preparation of Standard Stock Solution
Preparation of Diluent
After a series of experimental attempts, it is possible to accomplish separation under optimal circumstances. A stationary phase column, such as the Hypersil BDS C18 (100 mm x 2.1 mm, 1.7 m), was the best choice since it generated symmetrical peaks with great resolution and sensitivity. The flow rate was kept constant at 0.5 mL min-1, indicating acceptable resolution. The PDA detector response of Camel Milk Dry powder was investigated, and the optimal wavelength with the maximum sensitivity was discovered to be 230 nm.

At 0.5mL/min, a combination of two solutions, Chloroform and Methanol in the ratio of 30:70 percent v/v," was found to be an acceptable mobile phase for separation of Camel Milk Dry powder. The temperature in the column was kept at room temperature.

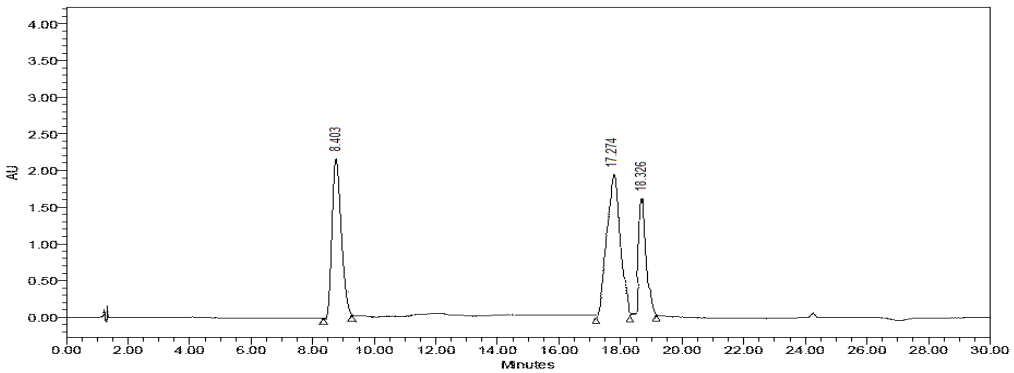
Preparation of internal standard solution
Weighed accurately about 10 mg of Camel Milk Dry powder working standard and transfer to 100 ml volumetric flask, add 50 ml of mobile phase and sonicate to dissolve it completely and then volume was made up to the mark with mobile phase to get 100 µg/ml of standard stock solution of working standard. Then it was ultrasonicated for 10 minutes and filtered through 0.20 µ membrane filter.

Preparation of Camel Milk Dry powder standard solution
Weighed accurately about 10 mg of Camel Milk Dry powder and transfer to 100 ml volumetric flask, add 50 ml of mobile phase and sonicate to dissolve it completely and then volume was made up to the mark with mobile phase to get 100 µg/ml of standard stock solution of working standard. Then it was ultrasonicated for 10 minutes and filtered through 0.20 µ membrane filter.

- ***Camel Milk Dry powder***

Camel Milk Dry powder	
System	UPLC
Stationary Phase	C18 column
"Mobile Phase"	"Chloroform and Methanol in the ratio of 30:70%v/v"
Diluents	Methanol
Injection volume	5µl
Temperature	Ambient
Flow rate	0.5 ml/min
UV detection	230nm
Retention Time	Lactoferrin – 17.274 mins; 18.236 mins Casein – 8.403 mins
Inference	"High column pressure were observed"

Trial 1 of Camel Milk Dry powder in UPLC System



Chromatogram of standard preparation of Camel Milk Dry powder (Chloroform and Methanol in the ratio of 30:70%v/v)

- **Validation of Related Substance Studies for Camel Milk**

Accuracy Procedure: The accuracy of an analytical procedure expresses the closeness of agreement between the value which is accepted either as a conventional true value or an accepted reference value and the value found. This is sometimes termed trueness. The accuracy of the method was evaluated in triplicate at three concentration levels, 50%, 100% and 150% of the target test concentration. The percentages of recoveries were calculated.

"***Accuracy 50%:*** "From the prepared stock solution 0.2 mL solution was transferred to a 10 mL volumetric flask and diluted to the mark with mobile phase to obtain a working sample solution of Camel Milk (2 µg/mL)."

"***Accuracy 100%:*** From the prepared stock solution 0.4 mL solution was transferred to a 10 mL volumetric flask and diluted to the mark with mobile phase to obtain a working sample solution of Camel Milk (4 µg/mL)."

"***Accuracy 150%:*** From the prepared stock solution 0.6 mL solution was transferred to a 10 mL volumetric flask and diluted to the mark with mobile phase to obtain a working sample solution of Camel Milk (6 µg/mL)."

Camel Milk						
Level %	**Amount added (µg/ml)**	**Amount found (µg/ml)**	**% Recovery**	**Mean recovery (%)**	**Std.Dev**	**% RSD**
50	02.13	02.11	99.06	99.46%	0.27004	0.27%
100	04.13	04.09	99.51			
150	06.14	06.13	99.83			

Accuracy

System Precision

"The parameters, retention time (RT), theoretical plates (N), tailing factor (T), peak asymmetry (As) and repeatability were evaluated at a concentration of 4 µg/mL (Camel Milk)."

System Precision

Parameters	**Camel Milk**
Retention time (min) ± % RSD	17.385 ± 0.06 ; 18.364 ± 0.06
Theoretical plates ± % RSD	4833.37 ± 0.50; 6506.99 ± 0.50
Asymmetry ± % RSD	1.03 ± 0.05; 1.04 ± 0.05
Repeatability (% RSD)	0.46; 0.47

Method Precision

The precision of an analytical procedure expresses the closeness of agreement (degree of scatter) between a series of measurements obtained from multiple sampling of the same homogeneous sample under the prescribed conditions.

Acceptance Criteria: %RSD is nmt 2%

""**Procedure:** Precision was investigated using the sample preparation procedure for six consecutive replicates of sample of concentration 4 µg/mL for Camel Milk."

Method Precision

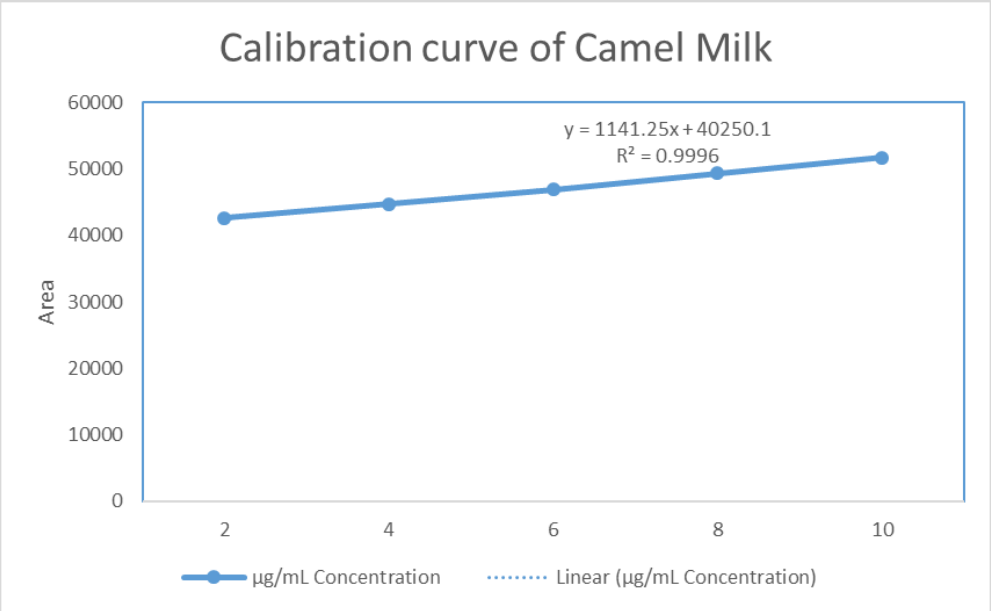
Replicate	***Camel Milk***		
S.No.	**Concentration Taken (µg/ml)**	**Area**	**%LC**
1	04.00	45748	99.99%
2		45731	99.96%
3		45767	99.93%
4		45679	99.86%
5		45692	99.81%
6		45752	99.76%
Average			99.88%
Std.Dev			0.090055
% RSD			0.09%
Standard weight			4mg
Standard potency			99.60%

Linearity

Camel Milk		
Linearity level	**Concentration in µg/mL**	**Area**
1	2 µg/mL	45767
2	4 µg/mL	50343
3	6 µg/mL	54920
4	8 µg/mL	59497
5	10 µg/mL	64073
Correlation co-efficient	0.9996	

Slope	1141.25
Intercept	40250.1

Linearity



Calibration Curve of Camel Milk

Robustness

Robustness Studies			
Parameter	**Value**	**Peak Area**	**% RSD**
Flow Rate	**Low**	45781	0.11%
	Actual	45767	
	Plus	45787	
Temperature	**Low**	45780	0.67%
	Actual	45773	
	Plus	45770	
Wavelength	**Low**	45769	0.07%
	Actual	45782	
	Plus	45789	

Robustness

Ruggedness

““**Intraday precision (Repeatability)**: Intraday Precision was performed and % RSD for Camel Milk was 0.11%.”

“**Inter day precision:** Inter day precision was performed with 24 hrs time lag and the %RSD Obtained for Camel Milk was 0.15%.”

Camel Milk			
Ruggedness			
Parameter	**Peak Area**	**% RSD**	**%LC**
Intraday precision	45796	0.46%	98.93%
	45801		99.10%
	45793		99.79%
Inter day precision	45850	0.47%	98.92%
	45816		99.09%
	45834		99.81%
Instrument:1 Acquity UPLC	45823	0.42%	99.52%
	45789		99.69%

Waters, 2695H	45797		98.90%
Instrument:2 Agilent Technologies, 1290	45836	0.41%	99.53%
	45791		99.67%
	45795		98.91%
Average			**99.23%**
Std.Dev			**0.3687**
%RSD			**0.37%**

Ruggedness

LOD and LOQ

LOD

LOD=3.3(SD of intercept/Slope)

Total numbers: 5

SE of Intercept: 1614.63

SD of Intercept: 724.04

LOD= 3.3*(724.04/ 1141.25)

LOD= 3.3*(0.06344)

LOD= 0.20936(µg/ml)

LOQ

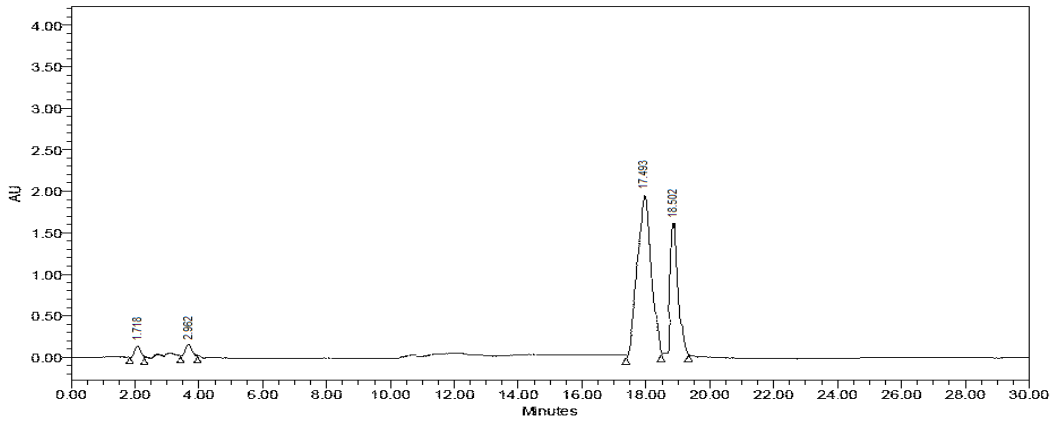
LOQ=10*(SD/S)

LOQ= 10*(724.04/ 1141.25)

LOQ= 0.6344(µg/ml)

ASSAY

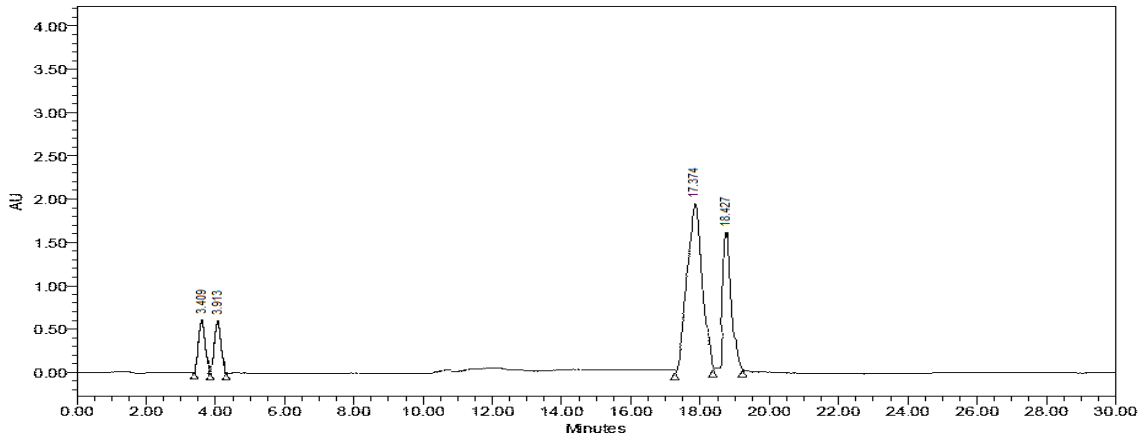
a. Acidic Degradation: "An accurate 10 ml of pure product sample solution was transferred to a clean and dry round bottom flask (RBF). 30 ml of 0.1 N HCl was added to it. It was refluxed in a water bath at 60°C for 6 hours. Product became soluble after reflux which was insoluble initially. Allowed to cool at room temperature. The sample was then neutralized using 2N NaOH solution and final volume of the sample was made up to 100ml with water to prepare 100ppm solution. It was injected into the UPLC system against a blank of mobile phase after optimizing the mobile phase composition, chromatogram was recorded and shown in Chromatogram."



Acidic Degradation

b. Basic Degradation

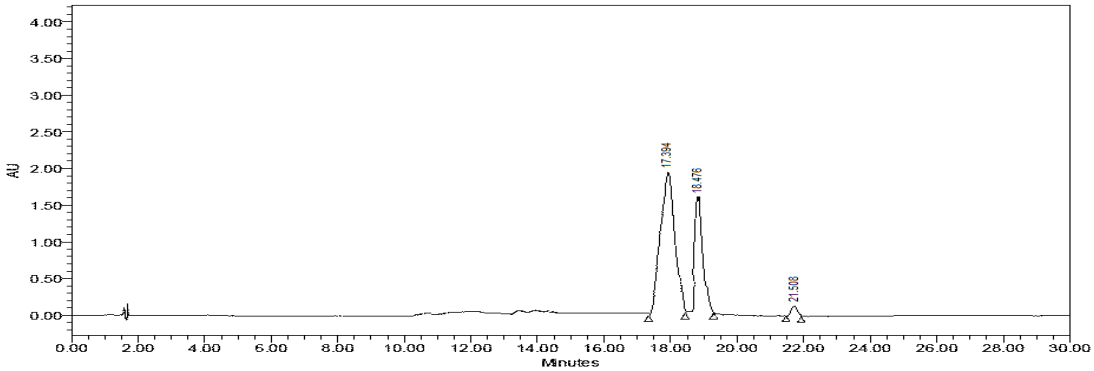
"An accurate 10 ml of pure drug sample solution was transferred to a clean and dry RBF. 30 ml of 0.1N NaOH was added to it. It was refluxed in a water bath at 60°C for 6 hours. Drug became soluble after reflux which was insoluble initially. It was allowed to cool at room temperature. The sample was then neutralized using 2N HCl solution and final volume of the sample was made up to 100ml with water to prepare 100ppm solution. It was injected into the UPLC system against a blank of mobile phase after optimizing the mobile phase composition, chromatogram was recorded and shown in Chromatogram."



Basic Degradation

c. Wet heat degradation

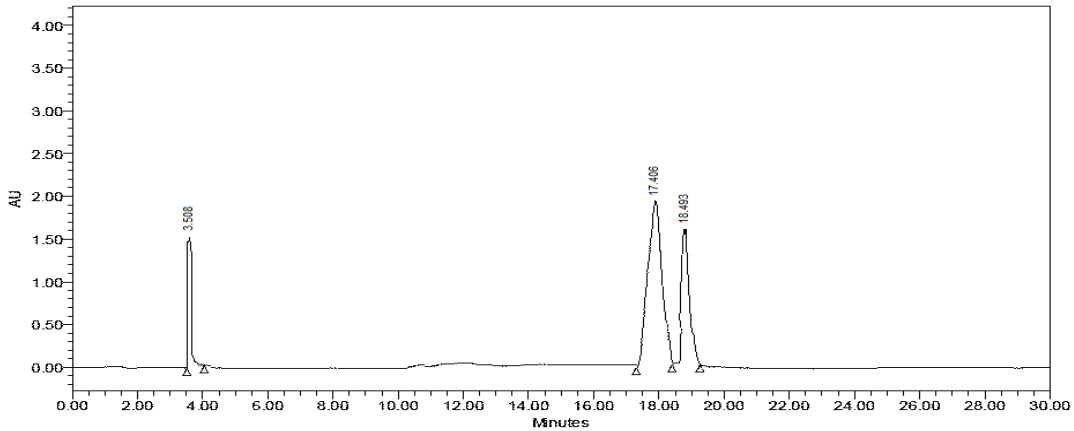
"Accurate 10 ml of pure drug sample was transferred to a clean and dry RBF. 30 ml of HPLC grade water was added to it. Then, it was refluxed in a water bath at 60°C for 6 hours uninterruptedly. After the completion of reflux, the drug became soluble and the mixture of drug and water was allowed to cool at room temperature. Final volume was made up to 100 ml with HPLC grade water to prepare 100 ppm solution. It was injected into the UPLC system against a blank of mobile phase after optimizing the mobile phase composition, chromatogram was recorded and shown in Chromatogram."



Wet heat Degradation

d. Oxidation with (3%) H_2O_2

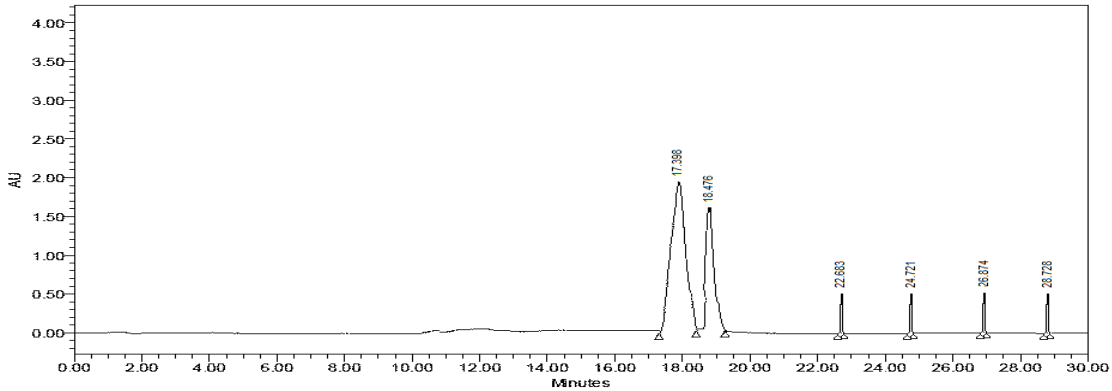
"Approximately 10 ml of pure drug sample was transferred in a clean and dry 100 ml volumetric flask. 30 ml of 3% H_2O_2 and a little methanol was added to it to make it soluble and then kept as such in dark for 24 hours. Final volume was made up to 100 ml using water to prepare 100 ppm solution. The above sample was injected into the UPLC system. The chromatogram was recorded and shown in Chromatogram."



Oxidation Degradation

Photolytic degradation

The photochemical stability of the drug was also studied by exposing the drug solution (4ml) to sunlight for 6 h. Twenty microlitres of the resultant solutions were injected onto column and the chromatograms were run as described.



Photolytic degradation

Nature of Stress	Degradation condition	Time(h)	Number of degradation products
Acidic	60°C	6	2
Basic	60°C	6	2
Oxidative	RT	6	1
Wet Heat	105°C	6	1
Photolytic	AT	6	4

Force Degradation

Calculation formula

$$\%\ Assay = \frac{AT}{AS} \times \frac{W1}{100} \times \frac{1}{25} \times \frac{100}{W2} \times \frac{25}{1} \times \frac{AW}{LC} \times P$$

Whereas,"

AT = Average area of test preparation, 26139"

AS = Average area of standard preparation, 28358"

W1 = Weight taken of reference standard (µg), 04.15"

W2 = Weight taken of test sample (µg), 04.25"

AW = Average weight of sample (µg), 3057"

LC = Label claim (µg), 3000"

P = Potency of reference standard (%), 99.98%"

$$\%\ Assay = \frac{AT}{AS} \times \frac{W1}{100} \times \frac{1}{25} \times \frac{100}{W2} \times \frac{25}{1} \times \frac{AW}{LC} \times P$$

Acidic Degradation

$$\%\ Assay = \frac{24721}{28358} \times \frac{04.15}{100} \times \frac{1}{25} \times \frac{100}{04.25} \times \frac{25}{1} \times \textbf{Error!} \times$$

99.98= 86.74%

Basic Degradation

$$\%\ Assay = \frac{23581}{28358} \times \frac{04.15}{100} \times \frac{1}{25} \times \frac{100}{04.25} \times \frac{25}{1} \times \textbf{Error!} \times$$

99.98= 82.73%

Oxidative Degradation

$$\%\ Assay = \frac{24357}{28358} \times \frac{04.15}{100} \times \frac{1}{25} \times \frac{100}{04.25} \times \frac{25}{1} \times \textbf{Error!} \times$$

99.98= 85.45%

Wet Heat

$$\%\ Assay = \frac{25832}{28358} \times \frac{04.15}{100} \times \frac{1}{25} \times \frac{100}{04.25} \times \frac{25}{1} \times \textbf{Error!} \times$$

99.98 = 90.63%

Photolytic

$$\%\ Assay = \frac{25874}{28396} \times \frac{04.15}{100} \times \frac{1}{25} \times \frac{100}{04.25} \times \frac{25}{1} \times \textbf{Error!} \times$$

99.98= 90.75%

CONCLUSION

A quick specific, accurate, exact, and delicate procedure was built up in order to determine the quantitative amounts of process-related contaminants and Camel Milk corruption items in pharmaceutical formulations. Following a stretch inquiry, the debasement items of Camel Milk were successfully separated from Camel Milk and its impurities, and the mass equalizations were determined to be adequate under all of the push conditions, demonstrating the method's ability to identify soundness. In terms of specificity, linearity, location and assessment restraint, accuracy, exactness, roughness, and vigour, the suggested approach was validated in understanding with the Universal Conference on undestanding standards.

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