

Development and Validation of UV Spectrophotometry Method for the Estimation of Dapsone Bulk and Pharmaceutical Dosage Form

Mrs. Priyanka Patel

Sardar Patel College of Pharmacy

Abstract - The selected drug Dapsone Dosage form is Anti leprosy drug. It is used for the treatment of the Leprosy. An extensive literature surveys reveals that various analytical method were reported for the estimation of Dapsone Dosage form in pharmaceutical dosage form. So, it was thought to developed simple, precise, accurate and specific UV Spectrophotometric method for the estimation of Dapsone Dosage form in pharmaceutical dosage form and statistically validate all the method for different parameter like precision, linearity, robustness, LOD, LOQ etc, as per ICH guideline. UV Spectrophotometric method was developed in methanol as a solvent. UV spectrophotometric method was found to be simple, accurate, and precise according to acceptance criteria given in ICH guideline.

Key Words: Dapsone Dosage form, UV Spectroscopy

1. INTRODUCTION (Size 11, Times New roman)

Dapsone is a sulfone derivative used for the treatment of leprosy, dermatitis herpetiformis, acne, and other skin infections. It acts by inhibiting bacterial growth and reducing inflammation. Chemically, Dapsone is known as 4,4'-diaminodiphenyl sulfone. It is a white or slightly yellow crystalline powder, practically insoluble in water and soluble in methanol. Literature survey reveals that several analytical methods such as HPLC, RP-HPLC, HPTLC, and UV spectrophotometric methods have been reported for estimation of Dapsone in bulk and pharmaceutical dosage forms. However, limited spectroscopic methods are available using methanol as solvent for routine analysis of Dapsone in pharmaceutical formulations.

2 MATERIALS AND METHODS:

Reagents and chemicals:

Dapsone tablet formulation was purchased from local pharmacy. methanol, Ethanol, acetone and octanol was purchased from Loba chemie Pvt. Ltd, Mum. Distilled water and all other chemicals used were analytical grade. A double-beam Shimadzu-1700 UV- Visible spectrophotometer, with spectral bandwidth of 2nm, along with of ± 0.5 nm wavelength accuracy for all the weighing throughout the research work, weighing balance Shimadzu AUX220 was used.

Stock Solution (Standard) of Dapson: Accurately weighed quantity of Dapsone 10 mg. transferred to 10 ml volumetric flask, dissolved and diluted up to mark with methanol. stock solution having strength of 1000 μ g/ml was prepared. Aliquot of 1 ml from above standard stock solution transferred to 10ml of volumetric flask diluted up to the mark with methanol. stock solution having strength of 100 μ g/ml was prepared.

Spectral characteristics of Dapson: solution of Dapsone (10 μ g/ml) was prepared by relevant dilution of standard solution and scanned at 10 μ g/ml to 50 μ g/ml

Preparation of Calibration curve: Appropriate volume of aliquots from working standard solution was transferred to different volumetric flasks of 10 ml capacity. The volume was adjusted to the mark with the methanol to obtain concentration of 10-50 μ g/ml. and measured absorbance at 220nm. Plotted the graph of absorbance versus respective concentration of Dapsone

Method Validation3 :

The method was validated with respect to LOD, Accuracy, linearity, LOQ and Precision as per the ICH Q2 (R1) guidelines.

Linearity:

The standard curve was obtained with six different concentrations of reference standard solution in range of 10-50 μ g/ml for the HPTLC method. All the values were performed in triplicate. The linearity was evaluated by linear regression analysis, which be situated to calculate the coefficient of correlation and slope11 .

Precision:

Precision was checked by taking five replicates of 30 μ g/ml for repeatability. For inter-day and intra-day (30 μ g/ml) where analysed for three times for consecutive three days and three thrice a day respectively. For Intermediate Precision: (Ruggedness) six repeats of 30 μ g/ml were analysed by different analysts. The data were analysed at 296nm. The percentage Relative Standard Deviation (% R.S.D.) of the analytical response were calculated12,13,14 .

Robustness

Robustness shows the reliability of an analyte with respect to deliberate variation in method parameters. Robustness test was performed with deliberate small changes in detection wave-length.

3. Results and Discussion

SOLUBILITY

Table 1: solubility

Sr. No	Solvent	Solubility
1.	Water	Sparingly Soluble
2.	Methanol	Soluble
3.	Chloroform	Insoluble
4.	Ethyl acetate	Sparingly soluble
5.	Methylene chloride	insoluble
6.	Ethanol	soluble

Linearity:

Linearity for different concentration of drug was performed and r2 value was noticed to be **0.999** for Dapsone.

Concentration (µg/ml)	Absorbance Mean	SD	%RSD
10	0.1026	0.0015	1.47
20	0.1942	0.0024	1.28
30	0.3078	0.0033	1.08
40	0.416	0.0062	1.49
50	0.5216	0.010	1.96

Table 3: Statistical data for Dapsone

PARAMETER	Dapsone Dosage form
Linearity	10-50 µg/ml
Linearity Equation	$y = 0.0106x - 0.0095$
Slope	0.0106x
Intercept	0.0095
Correlation Coefficient (r2)	0.999

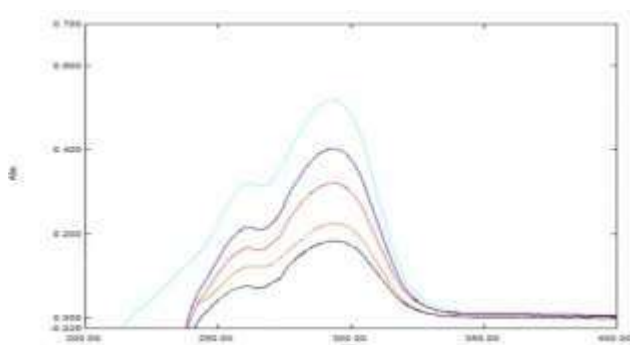


Figure 1 Absorption spectrum of standard solution of Dapsone in methanol at 296nm

Acceptance criteria

The correlation coefficient value should not be less than 0.998 over the working range.

Conclusion

The correlation coefficient value was found to be 0.9988. The absorbance obtained were directly proportion to the concentration of analyte in the sample. The method can, therefore be termed as linear in the specified range.

Precision:

Repeatability:

Precision was performed and % RSD was observed as 1.01

Table 4: repeatability of Dapsone at 296nm

Sr. No.	Concentration (µg/ml)	Absorbance of
1	30	0.312
2	30	0.301
3	30	0.299
4	30	0.304
5	30	0.31
6	30	0.312
	Mean	0.312
	SD	0.0057
	%RSD	1.87

Acceptance criteria

%RSD of six preparations for method precision should not be more than 2.0%.

Conclusion

The results obtained lies well within the acceptance criteria. The method can therefore be termed as precise.

Intra-day and Inter-day precision: The % RSD was instituted to be >2% for inter-day or intraday

Table 5: Intra- day of Dapsone at 296nm

Sr. No.	Conc. (µg/ml)	Mean	SD	%RSD
1	10	0.108	0.0015	1.40
2	30	0.198	0.0030	1.51
3	50	0.311	0.0025	0.80

5	LOQ	3.44
6	ROBUSTNESS	Robust

Table 6: Inter-day of Dapsone at 2296nm

Sr. No.	Conc. (µg/ml)	Mean	SD	%RSD
1	10	0.119	0.0015	1.28
2	30	0.190	0.0015	0.80
3	50	0.316	0.0041	1.31

Limits of Detection and Limit of Quantitation

Table 7: LOD & LOQ of Dapsone

Parameters	Concentration(µg/ml)
LOD	1.136
LOQ	3.444

Acceptance criteria

LOQ should be 3 times higher than LOD.

Conclusion

The result obtained was the acceptance criteria

Summary of Validation Parameter

Sr. No.	Parameter	Dapsone Dosage form
1	LINEARITY	
	Range (µg/ml)	10-50µg/ml
	Linearity Equation	$y = 0.0106x - 0.0095$
	Slope	0.0106x
	Intercept	0.0095
	Correlation Coefficient (r2)	0.999
2	Accuracy	99.12 – 101.98
3	PRECISION (Percent RSD)	
	Repeatability	1.87
	Intra-day	0.80 – 1.51
	inter-day	0.80 – 1.31
4	LOD	1.13

CONCLUSIONS:

- The UV Spectrophotometric method was developed and validated for the estimation of **Dapsone**.
- Review of literature for various analytical techniques has been performed. According to literature, aim of present work was summarized.
- UV Spectrophotometric method method was developed as per ICH guidelines for estimation of **Dapsone**.
- Method was found to be precise, and robust according to acceptance criteria and with low level of LOD and LOQ. So, it can conclude that current work satisfies all necessary requirement for method development and can be used routinely for estimation of **Dapsone** from Tablet dosage forms

Reference

1. Settle FA. Handbook of Instrumental Techniques for Analytical Chemistry; 1st Edn; Prentice Hall Inc., New Jersey, 1997, 98
2. British pharmacopoeia commission Dapsone British pharmacopoeia, volume 3 2010
3. De, Anjan, Suddhasattya Dey, Prasanna Kumar Pradhan, Falguni Chaudhari, and Madhavi Patel. "Estimation of dapsone in bulk & dosage form by uv spectroscopic method." *American Journal of Pharm Research* 4, no. 01 (2014).
4. De, A., Dey, S., Pradhan, P.K., Chaudhari, F. and Patel, M., 2014. Estimation of dapsone in bulk & dosage form by uv spectroscopic method. *American Journal of Pharm Research*, 4(01).
5. Dapsone, Drug Bank: <https://go.drugbank.com/drugs/DB00250>
6. Dapsone, Wikipedia: <https://en.wikipedia.org/wiki/Dapsone>
7. Dapsone, PubChem: <https://pubchem.ncbi.nlm.nih.gov/compound/Dapsone>