

Asian Journal of Research in Chemistry and Pharmaceutical Sciences

Journal home page: www.ajrcps.com

<https://doi.org/10.36673/AJRCPS.2020.v08.i01.A06>



UV SPECTROPHOTOMETRIC METHOD DEVELOPMENT AND VALIDATION OF DAPSONE IN BULK AND GEL FORMULATION

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ABSTRACT

Objective: A new, simple, sensitive, precise and reproducible UV spectroscopic method was developed for the estimation of Dapsone in bulk and gel formulation. **Methods:** The UV spectrum of Dapsone in methanol: water (30:70) showed λ max at 254nm. Beer's law is valid in the concentration range of 3-15 μ g/ml. This method was validated for linearity, accuracy, precision, ruggedness and robustness. **Results:** The method has demonstrated excellent linearity over the range of 3-15 μ g/ml with regression equation $y = 0.065x + 0.0353$ and regression correlation coefficient $r^2 = 0.9991$. Moreover, the method was found to be highly sensitive with LOD (0.519 μ g/ml) and LOQ (1.729 μ g/ml). **Conclusion:** Depending on results the given method can be successfully applied for assay of dapsone in gel formulation.

KEYWORDS

Dapsone, UV spectroscopy, Method development, Validation, Methanol: Water (30:70) and Dapsone gel.

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INTRODUCTON

Acne is caused by the effects of hormones on the pilosebaceous unit consisting of a hair follicle and sebaceous gland. The normal skin follicles are blocked by the overgrowth of bacteria¹.

Dapsone is used to treat dermatitis, malaria, and leprosy. Dapsone inhibits the synthesis of dihydrofolic acid by competing with para-aminobenzoate for the active site of dihydropteroate synthetase, thus resembling the action of sulphonamide. It also inhibits the myeloperoxidase-H₂O₂-halide-mediated cytotoxic system in polymorphonucleocytes. Myeloperoxidase covert hydrogen peroxide into hypochlorous acid (HOCl)

is the most potent oxidant generated by neutrophils, and can cause significant tissue damage during inflammation. Dapsone arrests myeloperoxidase in an inactive intermediate form, reversibly inhibiting the enzyme. This prevent accumulation of hypochlorous acid, and reduces tissue damage during inflammation. The mechanism of topical dapsone in the treatment of acne vulgaris may result from a combination of both anti-microbial and anti-inflammatory effects^{2,3}.

The Chemical name of Dapsone is (4, 4'-diaminodiphenylsulfone). The molecular formula of Dapsone is C₁₂H₁₂N₂O₂S and molecular weight is 248.302gm/mol. Dapsone is white powder and has melting point 175^o-176^oC. It is freely soluble in alcohol, acetone and methanol. The aim of this study is to give a new, simple, sensitive, precise and reproducible UV spectroscopic method was developed for the estimation of Dapsone in bulk and gel formulation^{4,5}.

MATERIAL AND METHODS

Materials

Dapsone was take as gift sample from Aadhar life sciences Pvt. Ltd. Chincholi, Solapur. Distilled water and Methanol were taken of analytical grade.

Instruments

Analytical balance (Aczet CY224C), Sonicator (Labman), UV-Visible spectrophotometer (Labman UV-1900).

Experimental

Preparation of standard stock solution

Accurately weighed 10mg of dapsone transferred to 100ml volumetric flask. It was dissolved in methanol: water (30:70) and sonicated for 10minutes. The volume was made up to mark with same diluent to obtain final strength.

Procedure for plotting calibration curve

For calibration curve in a series of 10ml volumetric flasks, 0.3-1.5ml of standard solution was pipetted out separately. The volume was completed to the mark using methanol: water (30:70). The absorbance was measured at wavelength 254nm against blank solution.

Analysis of Dapsone in Gel formulation

10 mg equivalent Dapsone gel was weighed and transferred to the 100ml volumetric flask and dissolved in methanol: water (30:70) as a solvent. After that sonicated for 10min and vortex for 5min. 0.8ml of above solution was pipetted out and transferred to the 10ml volumetric flask and make up the volume up to the mark with same solvents and analysed at 254nm. Calculate the % purity of dapsone.

RESULTS AND DISCUSSION

The absorption spectrum shows λ max of Dapsone at 254nm.

The proposed method was validated according to ICH Q28 R1 guidelines for validation of analytical procedure⁶.

Linearity

Five different concentrations of Dapsone were prepared and analysed at wavelength 254nm. The regression coefficient was found to be 0.9991. The absorbance was found in limit i.e. 0-2. Hence the analyzed parameter was found to be validated (Table No.1).

Accuracy

The concentration 3, 6, 9 μ g/ml was taken as 50, 100, 150% and % recovery was found to be in range 99%-101%. Hence the parameter was found to be validated.

Range

Range is an interval between highest and lowest concentration limit of the analyte i.e. 3-15 μ g/ml.

Precision

In precision intra-day and inter-day precision were performed at concentration 9(μ g/ml). The obtained results were found within limit i.e. less than 2% RSD.

Limit of Detection (LOD)

The limit of detection was found to be 0.51 μ g/ml (Table No.6).

Limit of Quantification (LOQ)

The limit of quantification was found to be 1.72 μ g/ml (Table No.6).

Ruggedness

The change in analyst with same concentration and environmental condition didn't affect the results.

Robustness

The change in wavelength (254nm and 257nm) and concentration (5µg/ml) didn't affect the results.

Assay

The assay was performed by using Acnedap gel 5% at concentration 8µg/ml. The % purity was found to be 99.2%.

Table No.1: Results of Linearity

S.No	Concentration(µg/ml)	Absorbance
1	3	0.221
2	6	0.431
3	9	0.625
4	12	0.824
5	15	0.999

Table No.2: Optimization parameters of Dapsone

S.No	Parameters	Method values
1	Maximum Wavelength	254nm
2	Beer's Law	3-15µg/ml
3	Correlation Coefficient (r ²)	0.9991
4	Regression Equation	y = 0.065x + 0.0353
5	Slope (m)	0.065
6	Intercept (c)	0.0353

Table No.3: Results of Accuracy

S.No	Name of Drug	Recovery Level in %	Concentration	Amount Recovered	% recovery with SD
1	Dapsone	50	3µg/ml	3.05	100.05±0.25
		100	6 µg/ml	4.03	99.03±0.7
		150	9 µg/ml	5.04	100.06±0.04

Table No.4: Results of Intra-day Precision

S.No	Concentration	Absorbance
1	9(µg/ml)	0.625
2		0.624
3		0.626
4		0.622
5		0.625
6		0.626
7	SD	0.001507
8	%RSD	0.241%

Table No.5: Results of Inter-day precision

S.No	Concentration	Absorbance (Day 1)	Absorbance (Day 2)
1	9(µg/ml)	0.625	0.627
2		0.624	0.625
3		0.626	0.629
4		0.622	0.624
5		0.625	0.627
6		0.626	0.626
7	SD	0.001507	0.001751
8	%RSD	0.2428%	0.2795%

Table No.6: Results of LOD and LOQ

LOD	0.51 µg/ml
LOQ	1.72µg/ml

Table No.7: Results of Ruggedness

S.No	Concentration	Absorbance (Analyst 1)	Absorbance (Analyst 2)
1	5µg/ml	0.385	0.386
		0.387	0.385
		0.384	0.387
		0.388	0.386
		0.385	0.385
		0.386	0.385
2	Average	0.385833	0.385667
3	SD	0.001472	0.000816

Table No.8: Results of Robustness

Wavelength	254nm	257nm
Concentration	5 µg/ml	5µg/ml
Absorbance	0.385	0.384
	0.387	0.387
	0.384	0.383
	0.388	0.382
	0.385	0.387
	0.386	0.384
Average	0.385833	0.3845
SD	0.001472	0.002074

Table No.9: Results of Assay

S.No	Formulation	Labeled Amount	Amount obtained	% purity
1	Acnedap Gel	5%	0.492%	99.2%

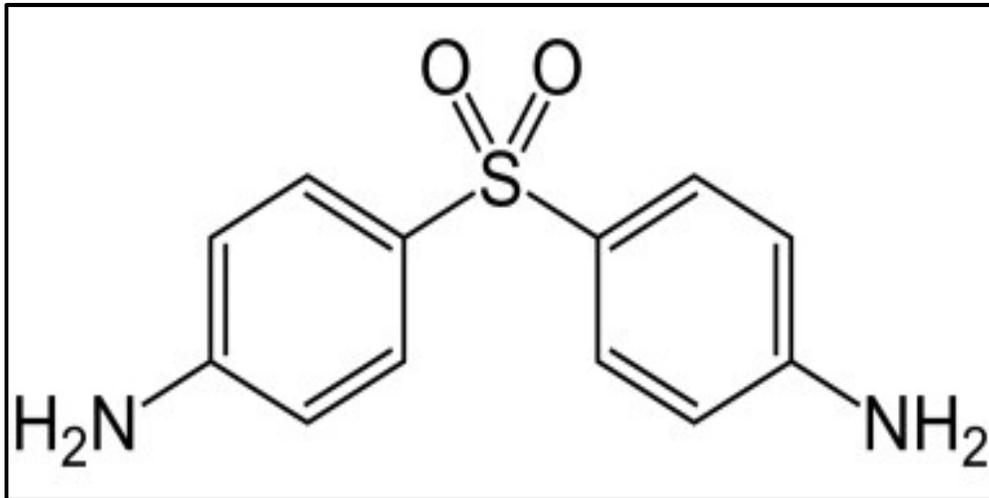


Figure No.1: Structure of Dapsone

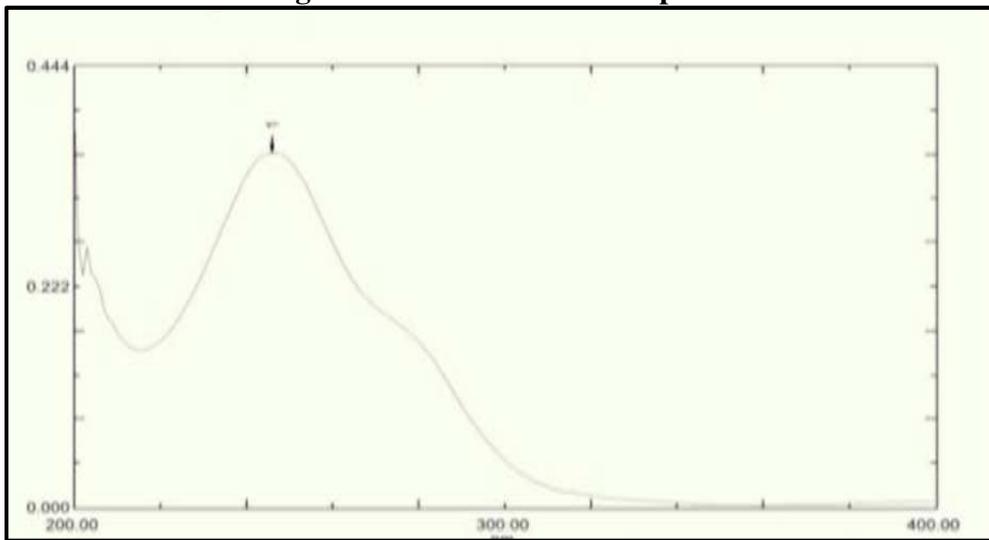


Figure No.2: UV spectrum of Dapsone

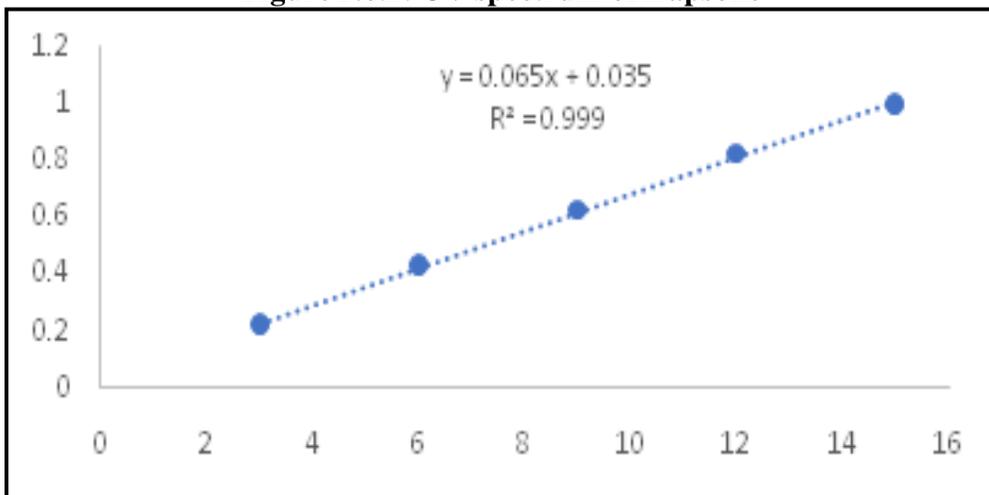


Figure No.3: Calibration curve for Dapsone (Concentration Vs Absorbance)

CONCLUSION

An analytical UV spectrophotometric method was developed and validated thoroughly for quantitative determination of Dapsone in pure drug and gel. The presented method was found to be simple, precise, accurate, rugged, reproducible and gives an acceptable recovery of the analyte, which can be directly easily applied to the analysis of pharmaceutical gel formulation of Dapsone.

ACKNOWLEDGEMENT

Authors are thankful to the Aadhar life science Private Ltd., Chincholi, Solapur, for providing the necessary facilities.

CONFLICT OF INTEREST

We declare that we have no conflict of interest.

BIBLIOGRAPHY

1. Mahore J, Suryawanshi S, Shirolkar S, Deshkar S. Enhancement of Percutaneous Delivery of Dapsone by Microemulsion Gel, *J Young Pharm*, 9(4), 2007, 507-512.
2. Seshadri V, Manohari P, Kunchithapatham N. Formulation and evaluation of Dapsone topical gel 7.5% w/w, *World J Pharm. Research*, 7(5), 2018, 1281-1299.
3. Indian Pharmacopoeia 2007, 376.
4. Williams D, Lemke T. Foye's Principles of Medicinal Chemistry, *Philadelphia: Lippincott Williams and Wilkins, Baltimore: Wolters and Kulwer Business*, 7th Edition, 2013, 1192.
5. <https://www.drugbank.ca/drugs/DB00250>.
6. ICH Q2 (R1) validation of analytical procedures: text and methodology, *International Conference on Harmonization*, 1996, 1-15.

Please cite this article in press as: Smeeta naganath Patil et al. UV spectrophotometric method development and validation of Dapsone in bulk and Gel formulation, *Asian Journal of Research in Chemistry and Pharmaceutical Sciences*, 8(1), 2020, 34-39.