International Journal of Drug Delivery 7 (2015) 233-240
http://www.arjournals.org/index.php/ijdd/index

Original Research Article

ISSN: 0975-0215

Method development for determination of water content from various materials by spectrophotometry and it's validation

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Abstract

A simple, sensitive and accurate UV-Spectrophotometric method for the estimation of water in various organic solvent (pyridine, methanol and DMSO) and its validation by spectrophotometry has been developed and compared with Karlfischer method. standard solution of water in Pyridine , methanol and DMSO was scanned over the range of 800-400 nm in spectrum mode of spectrophotometer at medium scanning speed using U.V spectrophotometer 1600 lambda max of water was found was found to be 450nm, 420nm and 438 nm in pyridine, methanol and DMSO. Three standard solution of water obeyed the Beer-Lambert's law and were found to be linear over the concentration range of 1-5 μg/ml. validation of method was done by spectroscopy., Percentage recoveries for pyridine, methanol and DMSO were found to be 99.58,99.71 and 99.92.

Keywords: Water, pyridine, methanol and DMSO,UV-Spectrophotometric method

Introduction

Analytical chemistry deals with quantitative analysis of composition of substances and complex materials in various matrices, by measuring a physical or chemical property of a distinctive constituent of the components of interest. Analytical methods are classified according to the property of the analyte measured.[1] The pharmaceutical analysis is one of the most important fields in analytical chemistry. Modern analytical chemistry is dominated by instrumental analysis. There are so many different types of instruments used today that, it can seem like a confusing array of acronyms rather than a unified field of study. [2] The analytical methods should be accurate as possible. [3] Analytical methods are classified into chemical and instrumental method. instrumental methods are used in pharmaceutical analysis, of which important methods are separation techniques, spectrometric techniques and other analytical techniques. [4] Among the instrumental analytical techniques the UV visible spectrophotometric methods are comparatively, simpler, sensitive, rapid and may be adapted for the analysis of binary mixtures without prior separation. [5]

Analytical method validation

According to the ICH, typical analytical performance characteristics that's should be considered in the validation methods are Accuracy, Precision, Specificity, Limit of quntitation, Limit of detection, Linearity and range, Ruggedness, Robustness. [6,7]

Water

Although water covers most of the earth's surface, we could put ourselves at grave risk if we took it for granted in fact, the availability of pure water may be destined to become the dominant resource issue of the 21st century, much as was oil in the 20th century.

To deal with water in any given system, we may need to view the quantity that is presenting the context of a required optimum amount or a minimum acceptable level.

Proper perspective can only be provided by accurate measurement of the amount or concentration of water present. The subject of this brief monograph concerns an important method of measurement - the Karl Fischer titration method.

The method of measurement of water- Karl Fischer titration method Essentially all real samples contain some moisture For solid samples, water theoretically can be present in six different modes: Adsorbed, Absorbed, Imbibed, Occluded, Hydrated, Water of chemical combination. [8,]

Other methods for determining water are as follows

A. Chemical methods: Include those based on reactions with acetyl chloride, acid anhydrides, lead tetra acetate, tertiary-butyl-*o*-vanadate, magnesium nitride, or calcium carbide.

B. Gravimetric methods: Include use of oven drying, desiccation, thermogravimetry, freeze drying, absorption, or condensation.

C. Thermal methods: Thermal conductivity, reaction, and differential thermal analysis methods.

D. Spectral methods: Visible, ultraviolet, infrared, NMR, mass spec, microwave, and X-ray spectroscopy (use of infrared methods to determine moisture content via dew point measurement).

E. Separation method: Measurement of water after separation by distillation, centrifugation, extraction, or chromatographically.
F. Physical methods: Determination based upon measurement of turbidity, density, refractive index, vapor pressure, volume of gas liberated by reaction of water with various substances, dew point, freezing point, piezoelectric effect by absorbed mass of water, and other physical phenomena or properties influenced by moisture content.[1]
Literature survey [9-12] revealed that no methods are developed for determination of water content from various material by spectrophotometry and it's validation. So here an attempt has been made to develop simple, accurate, sensitive, rapid and economic method for determination of water content from methanol, pyridine and DMSO by Spectrophotometry .

Material and Methods

Instrumentation

UV–Visible Spectrophotometer (UV spectrophotometer 1601, Shimadzu with 1 cm matched quartz cuvettes were used for all absorbance measurements. Electronic Weighing Balance (Shimadzu Analytical Balance) was used.

Chemicals and reagents used

Pyridine, methanol and DMSO, 2,4,6 trinitroresorcinol (At laboratory level synthesized derivative)

Preparation of standard solution of water in Pyridine

Placed about 8ml Dry Pyridine in 10ml of volumetric flask and added 0.1013g water and 0.001g of reagent (2,4,6-trinitroresorcinol) and makeup the volume up to 10 ml using dry pyridine and λ_{max} is found to be 450nm.

Preparation of Calibration Curve of Water in Pyridine

From the 'Std Stock water' (10 µg/ml) solution 1.0, 2.0, 3.0, 4.0 & 5.0 ml of aliquot was pipetted out in a series of 10 ml volumetric flasks. The volume was made up to the mark with Pyridine to obtain the concentration of 1, 2, 3, 4 and 5 µg/ml. Absorbance of the above solutions (1-5 µg/ml) was measured at 450.0 nm and a calibration curve was constructed by plotting absorbance vs. concentration.

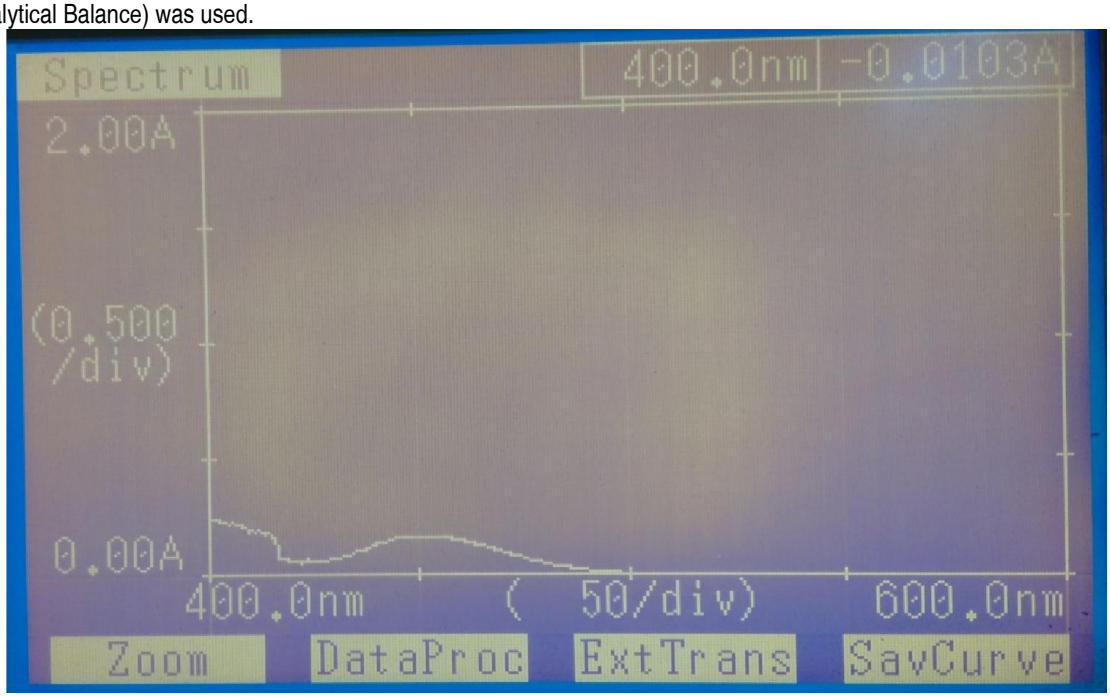

Figure. 1 Spectrum of water in Pyridine

Table 1 : Percentage of water in Pyridine in developed method and KF method

Sr. No.	Concentration (µg/ml)	Absorbance λ_{max} = 450nm	% water determination by developed method	% water determination by KF method
1	0.0004	0.4482	99.86	99.91
2	0.0004	0.4478	99.68	99.75
3	0.0004	0.4470	99.68	99.77
4	0.0004	0.4472	99.73	99.82
5	0.0004	0.4468	99.64	99.98
		Mean	99.71	99.84
		±S.D.	0.0769	0.0870
		R.S.D	0.0007	0.0008
		C.V	0.0771	0.0871

Preparation of standard solution of water in Methanol

Placed about 8ml Dry Methanol in 10ml of volumetric flask and added 0.1013g water and 0.001g of reagent (2,4,6-trinitroresorcinol) and makeup the volume up to 10 ml using dry methanol and λ_{max} is found to be 420nm.

Preparation of Calibration Curve of Water in Methanol

From the 'Std Stock water' (10µg/ml) solution 1.0, 2.0, 3.0, 4.0 & 5.0 ml ml of aliquot was pipetted out in a series of 10 ml volumetric flasks. The volume was made up to the mark with methanol to obtain the concentration of 1, 2, 3, 4 and 5 µg/ml. Absorbance of the above solutions (1-5 µg/ml) was measured at 420.0 nm and a calibration curve was constructed by plotting absorbance vs. concentration.

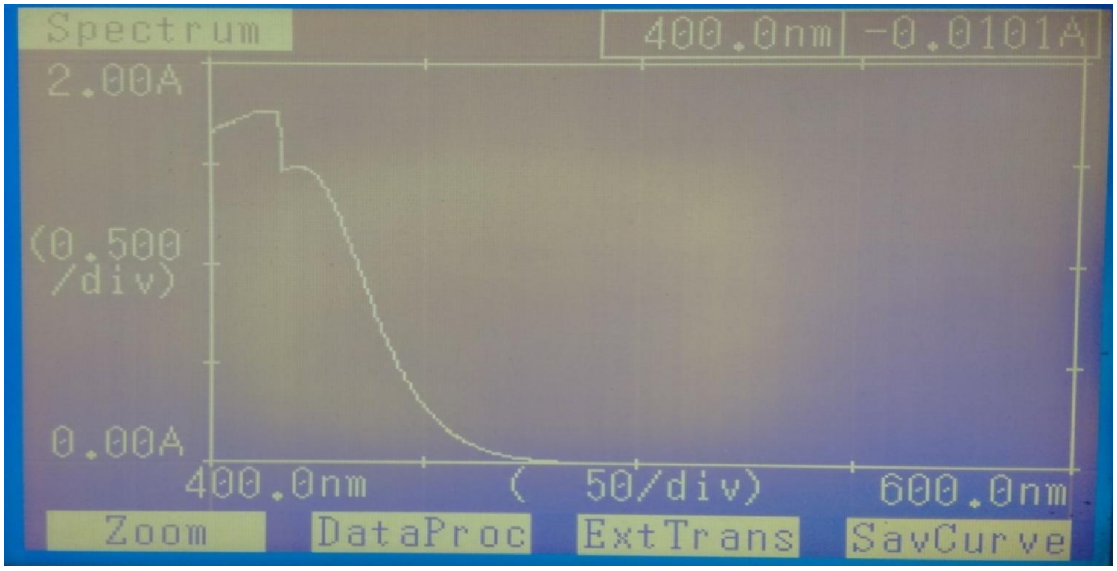

Figure. 2 Spectrum of water in Methanol

Table 2 : Percentage of water in Methanol in developed method and KF method

Sr. No.	Concentration (µg/ml)	Absorbance λ_{max} = 420nm	% water determination by developed method	% water determination by KF method
1	0.0002	0.8968	99.85	99.95
2	0.0002	0.8978	99.96	99.98
3	0.0002	0.8972	99.89	99.96
4	0.0002	0.8976	99.94	99.91
5	0.0002	0.8970	99.87	99.97
		Mean	99.90	99.95
		±S.D.	0.0932	0.0547
		R.S.D	0.0009	0.0005
		C.V	0.0932	0.0547

Preparation of standard solution of water in DMSO

laced about 8ml Dry Dimethyl Sulfoxide in 10ml of volumetric flask and added 0.1013g water and 0.001g of reagent (2,4,6-trinitroresorcinol) and make up the volume up to10 ml using dimethyl sulphoxide and λ_{max} is found to be 438nm.

Preparation of Calibration Curve of Water in DMSO

From the 'Std Stock water' (10µg/ml) solution 1.0, 2.0, 3.0, 4.0 & 5.0 ml ml of aliquot was pipetted out in a series of 10 ml volumetric flasks. The volume was made up to the mark with DMSO to obtain the concentration of 1, 2, 3, 4 and 5 µg/ml. Absorbance of the above solutions (1-5 µg/ml) was measured at 438.0 nm and a calibration curve was constructed by plotting absorbance vs. concentration.

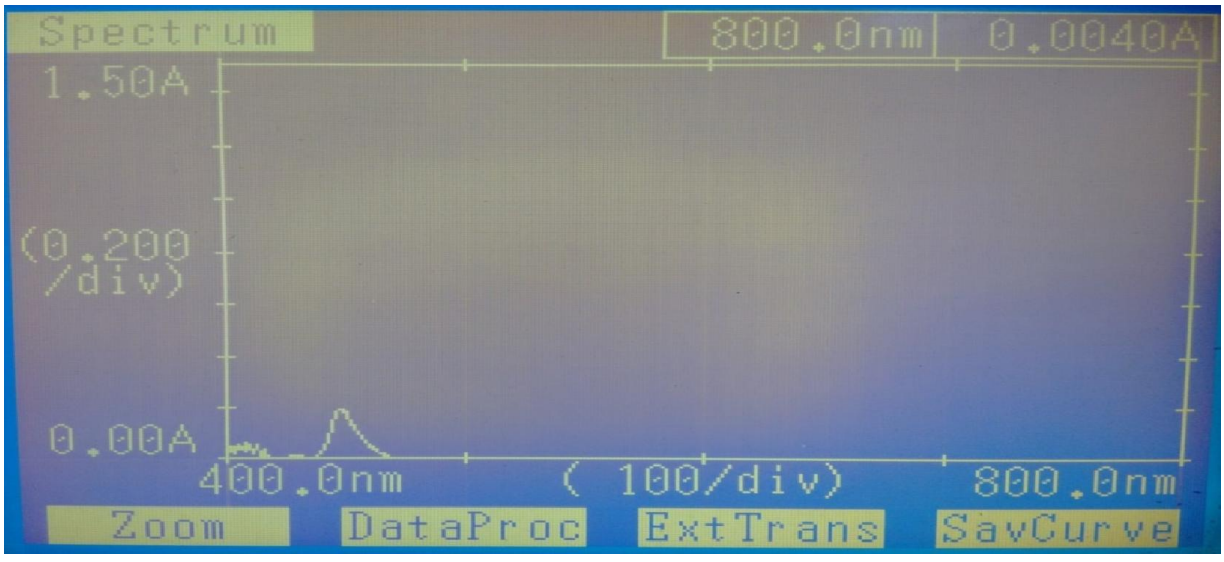

Figure. 3: Spectrum of water in DMSO

Table 3 : Percentageof water in Dimethyl sulfoxide in developed method and KF method :

Sr. No.	Concentration (µg/ml)	Absorbance λ_{max} = 438nm	% water determination by developed method	% water determination by KF method
1	0.0002	0.4386	99.86	99.89
2	0.0002	0.4390	99.95	99.99
3	0.0002	0.4388	99.90	99.91
4	0.0002	0.4382	99.77	99.97
5	0.0002	0.4384	99.81	99.93
		Mean	99.85	99.93
		±S.D.	0.0657	0.0379
		R.S.D	0.0006	0.0003
		C.V	0.0658	0.0379

Validation of Spectrophotometric Method

The method was validated by various parameters as recommended by ICH Guidelines10-11.

(a) Accuracy

To study the accuracy, % recoveries has to be calculated, recovery studies were carried out by standard addition method by adding the known amount of Water to the std sample solution at three different concentration levels i.e. 80%, 100%, and 120% of assay concentration and percentage recoveries were calculated. All solutions were scanned over the spectrum mode in the range of 800 nm to 400 nm, the absorbance at 450,420 and 438 nm are taken into calculation and likewise from these data obtained, % recoveries were calculated.

Table 4. Data of recovery study

Sr. No.	Amount of water added (µg/mL)			Amount of water recovered (µg/mL)			% of water recovered		
	In Pyridine	In Methanol	In DMSO	In Pyridine	In Methanol	In DMSO			
80% Recovery									
1	4.0	4.0	4.0	3.97	3.97	3.99	99.83	99.43	99.94
2	4.0	4.0	4.0	3.97	3.95	3.99	99.39	99.86	99.87
3	4.0	4.0	4.0	3.97	3.99	3.99	98.94	99.85	99.96
100% Recovery									
1	5.0	5.0	5.0	5.00	4.98	4.99	100.14	99.75	99.98
2	5.0	5.0	5.0	5.01	4.98	5.00	100.21	99.74	100.00
3	5.0	5.0	5.0	5.00	4.98	4.99	100.17	99.79	99.96
120% Recovery									
1	6.0	6.0	6.0	6.00	5.99	5.99	100.13	99.77	99.97
2	6.0	6.0	6.0	6.01	5.99	5.99	100.19	99.75	99.93
3	6.0	6.0	6.0	6.00	5.99	5.99	100.07	99.73	99.96

Table 5 : Statistical data of recovery study

Water	Mean	± SD	CV
80% Recovery			
In Pyridine	99.58	0.4110	0.4127
In methanol	99.71	0.2003	0.2008
In DMSO	99.92	0.0387	0.0387
100% Recovery			
In Pyridine	100.17	0.0282	0.0281
In methanol	99.76	0.02	0.0200
In DMSO	99.98	0.0632	0.0632
120% Recovery			
In Pyridine	100.13	0.0489	0.0488
In methanol	99.75	0.0161	0.0161
In DMSO	99.95	0.0173	0.0173

(b) Precision

The precision of an analytical method was studied by performing Intermediate precision

(i) Intra-day Precision

Variation of results within the same day was analyzed. Intra-day precision was determined by measuring the standard solution of Water at three different time intervals on the same day.

(ii) Inter-day Precision

Variation of results between the days was analyzed. Inter-day precision was determined by measuring the standard solution of water three consecutive days.

Table 6.Data of Ruggedness study

Water	Parameter	Intermediate Precision		
		Interday	Intraday	Different Analyst
In Pyridine	Mean	99.80	99.98	99.1
	±SD	0.2206	0.0741	0.0447
	CV	0.2210	0.0741	0.0451
In methanol	Mean	99.79	99.86	99.87
	±SD	0.0648	0.01	0.0141
	CV	0.0649	0.0100	0.01411
In DMSO	Mean	99.82	99.99	99.98
	±SD	0.1557	0.0244	0.0325
	CV	0.1559	0.0244	0.03250

Results and Discussion

In this method, Water was found to be absorbing prominently at 450 nm (λmax) in Pyridine, 420nm in methanol and 438nm in DMSO are described in Fig.1,fig.2 & fig.3. The linearity of analytical method at five concentration levels was ranging from 1-5 µg/ml for Water in Pyridine, methanol and DMSO and are presented in Table 3. The regression equation of calibration curves were curves were y = 0.112x + 0.000 and y = 0.448x + 0.001, y = 0.219x + 2E-05 for water in pyridine, methanol and DMSO and are shown in figure 4, figure 5 and figure 6 respectively. The results show that an excellent correlation exists between response factor and concentration of water within the concentration range. The correlation coefficient (r2) was found to be 0.9994, 0.9992 and 0.9994 for water respectively. Thus these above data represents that simultaneous equation method obeyed Beer- Lambert's Law. The developed method was found to be accurate from % recovery studies, the results are shown in Table 4. The mean % Assay shown in Table 1, Table 2 and Table 3 was found to be 99.71%, 99.90% and 99.85 % for water it was obtained by comparing with the Karlfischer method The results obtained had satisfactorily fulfilled the criteria.

Conclusion

From the above result and discussion it is concluded that developed method is easy, economic, reproducible, less time consuming and precise as compared to Karl-fischer method.

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