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RESEARCH ARTICLE

HPLC VALIDATION ONASPIRIN AND DIPYRIDAMOLEIN THE DRUG PRODUCTS

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ABSTRACT

This paper tries to recognize the substantiation through high performance liquid chromatography (HPLC) on the Dipyridamole and Aspirin, the separation was achieved on aX-Terra RP-18 5µm, 50 mm x 4.6 mm using a mobile phase consisting of 0.1% Ortho phosphoric acid and Acetonitrile in the ratio of 75:25 at a flow rate of 1.0 ml per minute. The detection was made at 227nm. The retention time of Aspirin and Dipyridamole were 1.5 and 2.8 minutes respectively. The proposed method was validated as per the ICH and USP guidelines. The method was found linear over the range of 4-80 µg per ml for Dipyridamole and 0.5-10 µg per ml for Aspirin.The experimental are peak purity and thresholdAspirin and Dipyridamolepeakswere met acceptance criteria.

Key words: Dipyridamole, Aspirin, HPLC

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INTRODUCTION

Dipyridamole is a medication that inhibits blood clot formation when given chronically and causes blood vessel dilation when given at high doses over a short time. Dipyridamole has two known effects, acting via different mechanisms of action such as (i) Dipyridamole inhibits the phosphodiesterase enzymes that normally break down cAMP (increasing cellular cAMP levels and blocking the platelet aggregation response to ADP) and/or cGMP; and (ii) Dipyridamole inhibits the cellular reuptake of adenosine into platelets, red blood cells, and endothelial cells, leading to increased extracellular concentrations of adenosine.[i]

Dipyridamole

Formula: $C_{24}H_{40}N_8O_4$ Molar Mass: 504.626 g/mol: Aspirin, also known as acetylsalicylic acid (ASA), is a medication used to treat pain, fever, or inflammation. Specific inflammatory conditions in which aspirin is used include Kawasaki disease, pericarditis, and rheumatic fever. Aspirin given shortly after a heart attack decreases the risk of death. Aspirin is also used long-term to help prevent further heart attacks, ischaemic strokes, and blood clots in people at high risk. It may also decrease the risk of certain types of cancer, particularly colorectal cancer.

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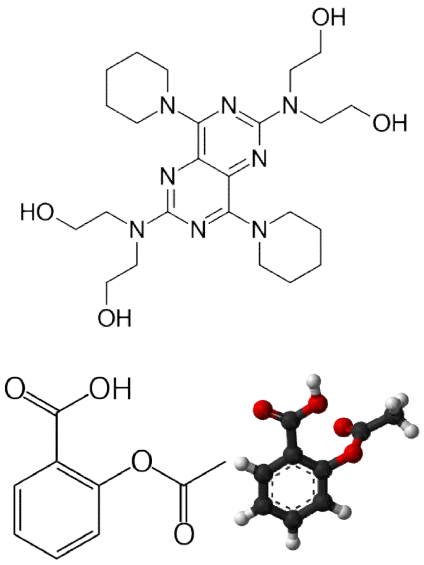
For pain or fever, effects typically begin within 30 minutes. Aspirin is a nonsteroidal anti-inflammatory drug (NSAID) and works similar to other NSAIDs but also suppresses the normal functioning of platelets.[ii]

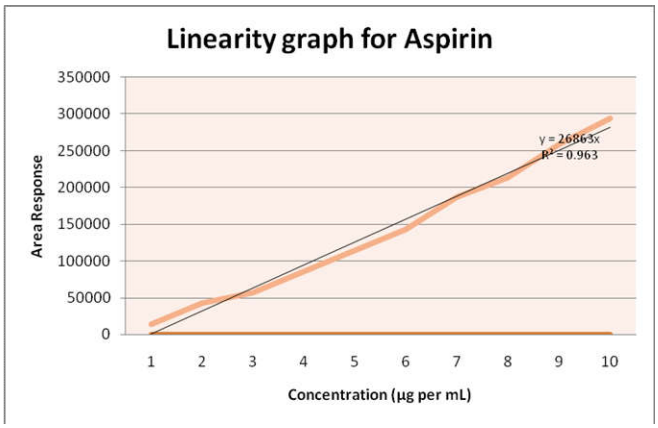
Aspirin

Formula: $C_9H_8O_4$ Molar Mass: 180.158 g/mol

Testing Method: On the basis of Preparation of buffer the content is diluted 1mL of ortho phosphoric acid to l000mL with milli-Q grade water. Filter this buffer through 0.45 µm Nylon 66 membrane filters. The preparation of mobile phase has been mixed under buffer and acetonitrile in the ratio of 75: 25 and degass in a sonicator for l0min. The diluent is Methanol. The preparation of Dipyridamole Standard Stock Solution is done by transfer accurately about 50 mg of dipyridamole working standard / reference standard, into a 50 mL volumetric flask, add about 30 mL of Methanol and sonicate to dissolve. Dilute to volume with Methanol and mix well. and the same type of preparation of aspirin standard stock solutionbytransferring accurately about 50 mg of aspirin working standard / reference standard, accurately weighed into a 200 mL volumetric flask, add about 150 mL of Methanol and sonicate dissolve. Dilute to volume with methanol and mix well with pipette 2 mL of dipyridamole standard stock solution and lmL of aspirin standard stock into a 50 mL volumetric flask and dilute to volume with methanol and mix well. Weighed and crushed not less than ten tablets into fine powder.



Weighed the blend equivalent to 200 mg of dipyridamole or 25 mg of aspirin in to 500ml of volumetric flask. Add about 150 ml of diluent kept on rotary shaker for 15minutes and added 150ml of diluent and sonicated for 30 minutes with intermediate shaking.



Graph 1. Linearity for Aspirin

The experimental resulting of tailing factor of Aspirin is 1.1 with 0.3 % RSD of Peak Area and tailing factor of Dipyridamole is 1.3 with 0.2 % RSD of Peak Area. Both of these tests are under the acceptance criteria of NMT 2.0%. The specificity of blank interference experiment is resulting that with the sample of purity angle of 0.56 and 0.385 of Aspirin and Dipyridamole respectively got its purity threshold as respectively with 1.26 and 0.921. Hence these angle and threshold are higher than the standard level.

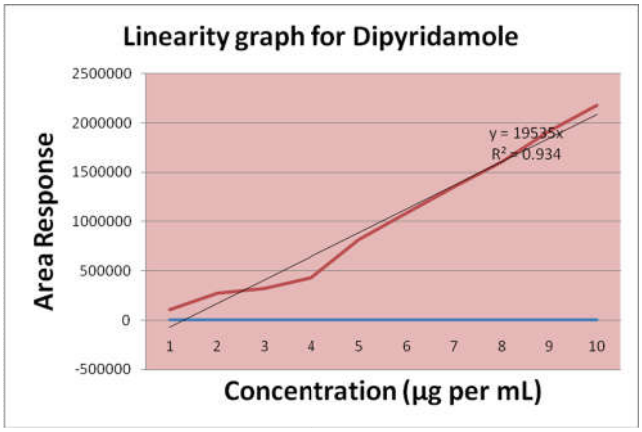
System Suitability	Parameter	Result		Acceptance Criteria
		Aspirin	Dipyridamole	
	Tailing Factor	1.1	1.3	NMT 2.0%
	%RSD of Peak Area	0.3	0.2	NMT 2.0%

Standard and Sample Peak Purity	Injection	Purity Angle		Purity Threshold		Peak Purity
		Aspirin	Dipyridamole	Aspirin	Dipyridamole	
	Standard	0.65	0.337	1.24	0.896	Pass
	Sample	0.56	0.385	1.26	0.921	Pass

Linearity	Sl.No.	Aspirin		Dipyridamole	
		Concentration (µg per mL)	Area	Concentration (µg per mL)	Area
	1	0.5	14286	4	108726
	2	1.5	42865	10	272015
	3	2	57285	12	325426
	4	3	85809	16	431235
	5	4	114829	30	815287
	6	5	143180	40	1088983
	7	6.5	187286	50	1351847
	8	7.5	213986	60	1602157
	9	9	258725	70	1912720
	10	10	294235	80	2175276
	Slope		29099.1	Slope	27137.5
	R		0.9998	R	0.99991

Dilute to volume with diluent and mix. Centrifuge above solution with lid at 4000 RPM for about 5 minutes. Pipette 5 mL of the above solution into a 50 mL volumetric flask and dilute to volume with diluents and mix.The liquid chromatography is equipped with UV visible detector at 227nm Column: X-Terra RP-18 5µm, 50 mm x 4.6 mm or equivalent; Column temperature is Ambient; Flow rate is 1.0 mL/min; Injection volume is 10 µL; and the Run time is 5Minutes.

Validating: The validation is on the basis of system suitability and specificity. A standard solution of Aspirin and Dipyridamole working standard was prepared and injected five times into the HPLC system. Peak area of Aspirin/Dipyridamole peak, the average peak area for 5 replicate injections, its %RSD and the USP tailing factor were calculated.



Graph 2. Linearity for Dipyridamole

Therefore, the result is passing for the both separation and validation of Aspirin/Dipyridamole as per the peak purity.

Linearity Experimental: Linearity of detector response for Aspirin/Dipyridamole was established by analyzing serial dilutions of a stock solution of the working standard. Five concentrations ranging from 50% to 150% of the test concentration were prepared and analyzed. The final concentration of each solution in mg per mL was plotted against peak area response. Slope, intercept and correlation coefficient (R) were calculated.

Conclusion

A rapid, simple, precise and cost effective and RP-HPLC method has been developed and validated for the simultaneous determination of Aspirin and Dipyridamole in pharmaceutical formulations. Separation of both Aspirin and Dipyridamole was achieved within 5 minutes with required resolution, accuracy and precision thus enabling the utility of the method for routine analysis. Chromatographic separation was achieved on a X-Terra RP-18 5µm, 50 mm x 4.6 mm using a mobile phase consisting of 0.1% Ortho phosphoric acid and Acetonitrile in the ratio of 75:25 at a flow rate of 1.0 ml per minute. The detection was made at 227nm. The retention time of Aspirin and Dipyridamole were 1.5 and 2.8 minutes respectively. The method was found linear over the range of 4-80 µg per ml for Dipyridamole and 0.5-10 µg per ml for Aspirin. The proposed method was validated as per the ICH and USP guidelines.

REFERENCES

[i]Brown DG, Wilkerson EC, Love WE (March 2015). "A review of traditional and novel oral anticoagulant and antiplatelet therapy for dermatologists and dermatologic surgeons". Journal of the American Academy of Dermatology.**72** (3): 524–34.

[ii]*https://web.archive.org/web/20170425142242/https://www.drugs.com/monograph/aspirin.html*

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