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Determination of Synthesized 1-Phenyl Naphthaoic Acid Lignan (PNAL) By Using Analytical Techniques HPLC

Rajdip Utane[1]* Ritesh Kohale[1] Sujata Deo[2]

[1]Department of Chemistry, Sant Gadge Maharaj Mahavidyalaya, Hingna. Nagpur, Maharashtra, India
[2]Department of Chemistry, Govt. Institute of Science, Nagpur, Maharashtra, India

ABSTRACT

Determination of 1-phenyl naphthalene and lignans by analytical techniques are used High Performance liquid chromatography.1-phenyl naphthalene has been synthesis via friedel craft acylation and Perkin–Oglialoro reaction followed by cyclization reaction. The key precursor use for synthesis of foresaid product by β -benzoyl propionic acid (β-BPA) through friedel craft acetylating reaction by mixture of succinic anhydride, benzene and its derivative with zeolite at streamline time to obtain blended accumulation followed by work-up with cold acid-water (1:1) treatment. The obtained accumulation distillation eliminates benzene liquor and obtained the crude mass. It was dissolved in aqueous solution of sodium carbonate (1:10) and acidification by hydrochloric acid to form crude β-benzoyl propionic acid (β-BPA) and their derivatives , perkin acid synthesize by two steps in which butenolides are prepared by β-BPA and aryl aldehyde using weak base catalyst pyridine and followed by cleavage of lactone ring methanolic base hydrolysis to form perkin acid. The perkin acid undergoes cyclization using zeolite gives 1-phenyl naphthalene. All the compounds are determined by HPLC.

I. INTRODUCTION

Among various analytical methods for standardization of Indian herbal medicines. High performance liquid chromatography (HPLC) is the most popular one, due to its versality, precision and relativity low cost. HPLC is one of the most useful analytical techniques because it is easy to learn and use. HPLC has been employed to analyze several components in a medicinal preparations composed of several crude drug. One of the main advantages of HPLC is that many detectors can be coupled with it, such as UV, MS and NMR, etc. by which detection of more constituents can be done.

In recent years, coulumtric electrode array detector (HPLC-CEAD) and charge aerosol detector (CAD) have been also introduced to the analysis of herbal formulations. HPLC method with various detectors has been developed for qualitative and quantitative analysis of various phyto constituents such as isolation and identification of synthetic compounds as lignan. So, HPLC is highly versatile chromatographic method which cans separate a wide variety of chemical constituents in almost all mixture [1-2].

Liquid chromatography though troublesome than gas chromatography, has the main advantage of operating at low temperatures and can be used with advantages for separation of substances as proteins, nucleosides which are thermo labile.

In conventional liquid chromatography, a dilute solution of a sample is passed through vertical column packed with solid particle. Thus, liquid is passed

through vertical column under gravitational flow. This is passed with slow speed and especially if the packing granules were small enough to give efficient separation, then the delivery under gravity decrease even upto a few drops per minute[3-5].

The obvious way to increase the flow rate and get efficient separation is to force the liquid by a positive displacement pump or by gas pressure. This versatile can be achieved by making certain modifications in column and by using smaller, diameter and smaller surface area of column particles and by using other suitable packing structure.

Thus HPLC is high resolution and high speed liquid chromatography. It has beenseveral times more resolving power than open column liquid chromatography. Hence it is used for speedy resolution of complex mixtures.

II. METHODS AND MATERIAL

2.1 Materials:

All synthesized compound a Derivatives 1–phenyl naphthalenes and extracted component by pet. Ether from *Cleistahnthus collinus.*

2.2 Methods (HPLC)

The HPLC system used for analysis consisted of Waters, auto sampler, UV detector with Data ace software for data acquisition and processing. The chromatographic analysis was performed on thermo C18 with bonded phase octadecylsilane column (250 x 4.60 mm, 5 µ particle size) with isocratic condition at ambient room temperature. The analysis was performed at flow rate 1.0 ml per min consist of mobile phase Methanol: Water (70:30) quantification was achieved with UV detection at 230 nm. Mobile phase Retention time of aryl naphthalenes were found to be 2.215 ± 0.3 min. and . Calibrated analytical balance of mettle toledo and digital pH meter of eutech instruments pH tutor was used for analysis purpose. The chromatographic conditions are summarized in table 1.

Table 1: Optimized condition during HPLC analysis of synthetically prepared1-Phenyl naphthalene from Cleistanthus. collinus

HPLC system	Waters
Softaware	Data Ace
Detector	UV-Visible
Pump	Waters 715
Stationary phase	µ
Mobile Phase	Methanol: Water (70:30% v/v)
Injection volume	20 µl
Column	Thermo C 18
Column length	250 nm
Column temperature	RT
Particle size packing	5 µ

Table 1. Blank preparation: Diluent used as blank

2.2.1 Stationary phase (Adsorbents)

HPLC separations are based on the surface interactions and depend on the types of the adsorption sites. Modern HPLC adsorbents are small rigid porous particles with high surface area. Main adsorbent parameters are Particle size: 3 to 10 um and Particle size distribution: as narrow as possible, usually within 10% of the mean.

2.2.2 Instrumentation of HPLC system

In order to attain reasonable high flow rates and yet keep particle size of packing very low (3-10 um), pumping pressures of several hundred atmosphreres (2000-8000 psi are required.

2.2.3 Mobile phase reservoir and solvent treatment systems

A modern HPLC apparatus is equipped with one or more glass or stainless steel reservoirs, each of which contain 500ml or more of solvent. The reservoirs are often equipped with a means of removing dissolved gases O2 and N2 that interface by forming bubbles in the columns and detector systems. These bubbles cause band spreading; in addition they interface with performance of the detector. Degassers may consist of A vacuum pumping system, Distillation system, Device for heating and stirring the solvents and

Device for sparring in which the dissolved gases are swept out of solution by fine bubbles of an inert gas of low solubility.

2.2.4 Pumps:

The pumps are used to pass mobile phase through the column at high pressure and at controlled flow rate. In addition to this pumps used in HPLC should have the following features. The generation of pressures upto 60000psi. Flow rates ranging from 0.1 to 10 ml/ min. Flow control and flow reproducibility of +_ 0.5 %.

2.2.5 Sample injectors

Often the limiting factor in the precision of liquid chromatographic measurements lies in the reproducibility with which samples can be introduced into the column packing. It must be noted that overlapping of the sample causes band boarding.

2.2.6 Syringe injection

This is the earliest and simplest technique. Hence the sample is injected through a self sealing elastomeric septum and the syringes are designed to withstand pressures a self sealing elastomeric septum and the syringes are designed to withstand pressures upto 1500 psi.

2.2.7 Liquid chromatographic column

They are usually constructed smooth bore stainless steel tubing or heavy-walled glass tubing. If prepared from heavy walled glass tubing, then pressure is restricted to lower than 600psi. Size length 25 to 100cm and internal diameter 2 to 6 mm.

2.3 Synthesis of 1-phenyl naphthalene

Take a mixture of1 gm of α-arylidine, β-Benzoyl Propionic acid (1 mmol), 1 g of activated zeolite H-beta (1 mmol) and 10 ml of ethanol (10 mmol) as a reaction solvent in 250 ml round bottom flask . Stir the mixture vigorously (using magnetic stirrer) by keeping reflux at 1200 C for an appropriate time as reaction mixture was cooled at room temperature and diluted with diethyl ether (3 x 10 ml) to precipitate of zeolite H-Beta easy separation. The combined organic layers were dried over anhydrous Na2SO4. The solvent was removed and the residue was column chromatograms using petroleum

ether: ethyl acetate (2:3) as the eluent, to obtain pure compound (1 to 5) shown in scheme 1 and table 2.

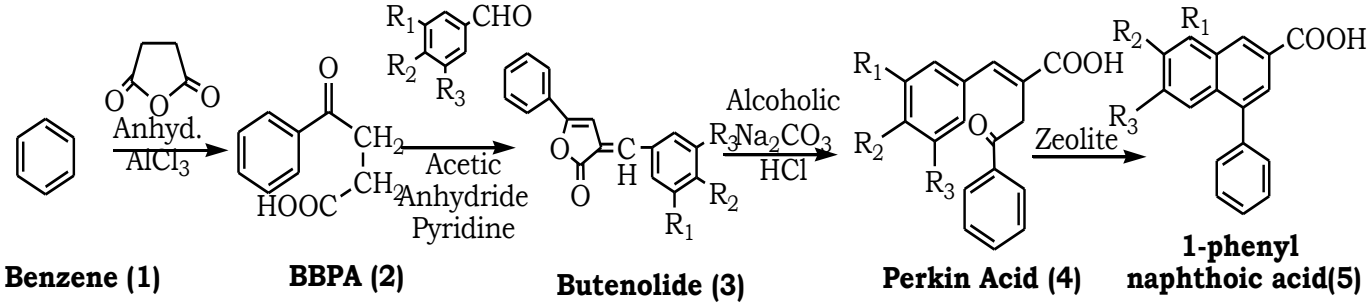

Figure 1 : Graphical Schematic Representation of Synthesis of 1-Phenyl naphthalene

2.4 Preparation of sample solution Synthetically prepared intermediates of 1-phenyl naphthalene

To prepare a stock solution for assay intermediates were weight and mixed. The average weight was determined and then they were finally powdered. An aliquot of powder equivalent to 1 mg per ml of intermediates, this add diluents to dissolve the substance by ultrasonication for 10 minutes and diluted with diluents and converted to 1 ug per ml. the resulting solution was stirred for 1 hour after that centrifuged at 1000 rpm for 10 minutes. Upper supernatant solution was used for further analysis.

These entire compounds of 1mmol β-Benzoyl Propionic acid, α-Arylidene γ -phenyl δ-β-γ-Butenolide, α-arylidine, β-Benzoyl Propionic acid and

1-phenyl naphthalene's subsidiaries are prepared by dissolving with 10 mmol of methanol. All these sample solution are examined by HPLC.

III. RESULTS AND DISCUSSION

Spectrophotometric analysis of 1-phenyl naphthoic acid

Take a mixture of 1 mmol of α-arylidine, β-benzoyl propionic acid (4a), 0.5mmol of activated zeolite H-beta (1mmol) and 10 mmol ofethanol (10 ml). 1-phenyl 7-methoxy naphthalene-3- carboxylic acid has showed molar extinction coefficient (logϵ) are 293.1 (1.82), 450.4 (0.68), HPLC chromatogram of RT values 2.679 as shown in fig. 4.22 and 4.26 . 1-phenyl 6-methoxy 7-Hydroxy naphthalene-3- carboxylic acid has showed UV max and molar extinction coefficient (logϵ) are 215.29 (1.56), 264.72 (1.23), 411.19 (2.23) , HPLC chromatogram of RT values 2.6798 as shown in fig. 2 and 5. 1-phenyl naphthalene-3- carboxylic acid has showed UV max and molar extinction coefficient (logϵ) are 238.4 (0.830), 330.8(330.8), HPLC chromatogram of RT values 2.679 respectively, as shown in fig. 4.24 and 4.28.

Table 2 : UV-visible Spectrophotometric of 1-Phenyl Naphthoic acid

SN	α-Arylidene β-benzoyl propionic acid	Derivatives of 1-Phenyl Naphthoic acid (5)	Molecular formula	HPLC (Rt values)
1	α-Anisalidene β-benzoyl propionic acid (4a)	1-phenyl 7-methoxy naphthalene-3- carboxylic acid (5a)	$C_{18}H_{14}O_3$	2.627
2	α-Varatralidene β-benzoyl propionicacid (4b)	1-phenyl 6 Hydroxy naphthalene-3- carboxylic acid (5b)	$C_{17}H_{14}O_4$	-
3	α-Salicalidene β-benzoyl propionic acid (4c)	1-phenyl 6,7-dimethoxy naphthalene-3- carboxylic acid (5c)	$C_{19}H_{18}O_5$	2.609
4	α-Vanilidene β-benzoyl propionic acid (4d)	1-phenyl 6-methoxy 7-Hydroxy naphthalene-3-carboxylic acid (5d)	$C_{18}H_{16}O_5$	2.678
5	α-Benzylidene β-benzoyl propionic acid (4e)	1-phenyl naphthalene-3-carboxylic acid (5e)	$C_{17}H_{14}O_3$	2.679

DataAce Company Pvt. Ltd.

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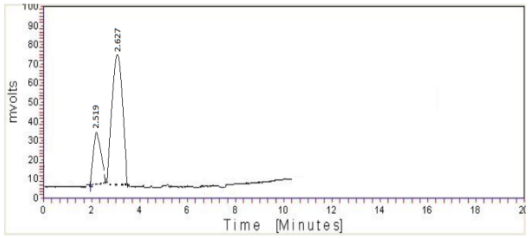

Result-A Table

Peak No	Retn.Time	Area	Height	Area %	Height %
1	2.519	30.857	36.63	42.02	33.31
2	2.627	42.575	73.37	57.98	66.69
Total		73.432	110.00	100	100

Figure 2 :HPLC chromatograms of 1-phenyl 7-methoxy naphthalene-3- carboxylic acid(5a)

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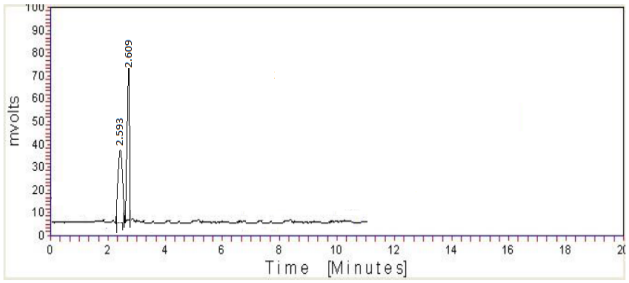

Result-A Table

Peak No	Retn.Time	Area	Height	Area %	Height %
1	2.593	30.821	36.617	41.997	33.407
2	2.609	42.569	72.980	58.003	66.593
Total		73.390	109.597	100	100

Figure 3 :HPLC chromatograms of 1-phenyl 6 , 7-dimethoxy naphthalene-3-carboxylic acid (5c)

DataAce Company Pvt. Ltd.

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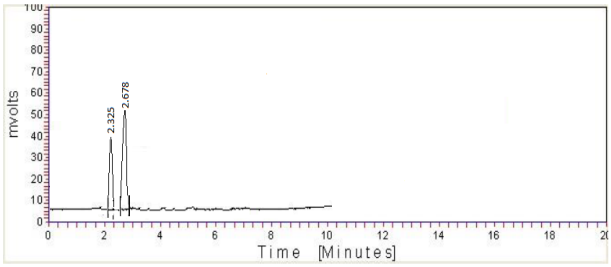

Result-A Table

Peak No	Retn.Time	Area	Height	Area %	Height %
1	2.325	30.592	40.736	41.301	43.313
2	2.678	43.466	53.314	58.699	56.687
Total		74.058	94.050	100	100

Figure 4 :HPLC chromatograms of 1-phenyl 6-methoxy 7-Hydroxy naphthalene-3- carboxylic acid (5d)

DataAce Company Pvt. Ltd.

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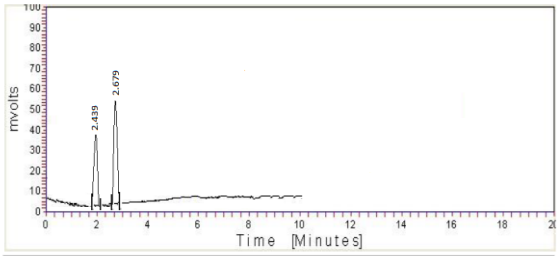

Result-A Table

Peak No	Retn.Time	Area	Height	Area %	Height %
1	2.439	30.589	40.835	41.302	43.346
2	2.679	43.474	53.374	58.698	56.654
Total		74.063	94.209	100	100

Figure 4 :HPLC chromatograms of 1-phenyl naphthalene-3- carboxylic acid (5e)

IV.CONCLUSION

In above synthesized of 1-phenyl naphthalene from benzene through stable intermediate as β-benzoyl propionic acid, α-Anisalidene γ -phenyl δ- β -γ-Butenolide, α-Arylidene β-benzoyl propionic acid and are identified and 1-Phenyl Naphthoic acid characterized by UV-visible spectrophotometric. A HPLC method has been developed for the determination of 1-phenyl naphthalene lignans. The developed method was simple rapid, linear, accurate, precise and specific. Results from the detection experiments showed that the method is reliable and accurate therefore it can be successfully applied for the routine quality control analysis of 1-phenyl naphthalene lignan.

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