

MINOR RESEARCH PROJECT

“Synthesis and characterization of Aryl-14H-dibenzo xanthenes under solvent free Conditions.”

Final report submitted

To

The joint secretary and Head
University grants commission
Western Regional Office (WRO)

Ganeshkhind,

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Submitted By

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MARCH 2014

CERTIFICATE

This is to certify that the final report on UGC Minor research project entitled "Synthesis and characterization of Aryl-14H-dibenzo xanthenes under solvent free Conditions." is a record of bonafide research work carried out by Mr. Salunke S.T. Assistant Professor of Chemistry, Tuljaram Chaturchand College, Baramati, Maharashtra. A copy of the final report of Minor Research Project has been kept in the library of College and an executive summary of the report has been posted on the website of the College.




Principal

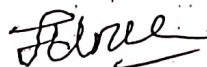
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Declaration

I hereby declare that the project report entitled "**Synthesis and characterization of Aryl-14H-dibenzo xanthenes under solvent free Conditions.**" Completed and written by me under the financial support of UGC, Pune University, Pune at Tuljaram Chaturchand College, Baramati has not been previously published or formed the basis of any degree, diploma, research project or any other similar title.

Place: Baramati

Date: 30/3/2014



Principal Investigator

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Last but not the least, I wish to extend my deep sence of gratefulness to all my family members for their support, this helped me to do my research work successfully.



Salunke S.T.

Place: Baramati

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Abstracts

Xanthenes and benzoxanthene have enormous biologically active heterocycles. There are numbers of publications showing antibacterial²⁸, antiviral²⁹ and anti-inflammatory³⁰ activities as well as in photodynamic therapy³¹ and antagonists of the paralyzing action of zoxazolamines³².

Here we report, procedure for the synthesis of 14-aryl or alkyl-14H dibenzo (a, j) xanthenes derivative have been synthesized by one-pot condensation of various aldehydes with 2-naphthol in presence of chlorosulphonic acid as a catalyst under micro-wave irradiation and solvent free Conditions. The present approach offers several application of simple work-up, shorter reaction time, excellent yields, low cost and economic availability of the catalyst.

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**PROFORMA FOR SUBMISSION OF INFORMATION AT THE TIME OF
SENDING THE**

FINAL REPORT OF THE WORK DONE ON THE PROJECT

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10. OBJECTIVES OF THE PROJECT : The main Objective of the present proposal is to construct the molecules like dibenzo xanthenes by one-pot multi-component condensation reaction and such products of biological significance which will pave the way in devising a new methodology in the synthesis of complex natural products.
11. WHETHER OBJECTIVES WERE ACHIEVED: Yes, Attached in Project Report.
12. ACHIEVEMENTS FROM THE PROJECT: Attached in project report.
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14. CONTRIBUTION TO THE SOCIETY : Attached in Project Report.
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16. NO. OF PUBLICATIONS OUT OF THE PROJECT : In process.


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Index

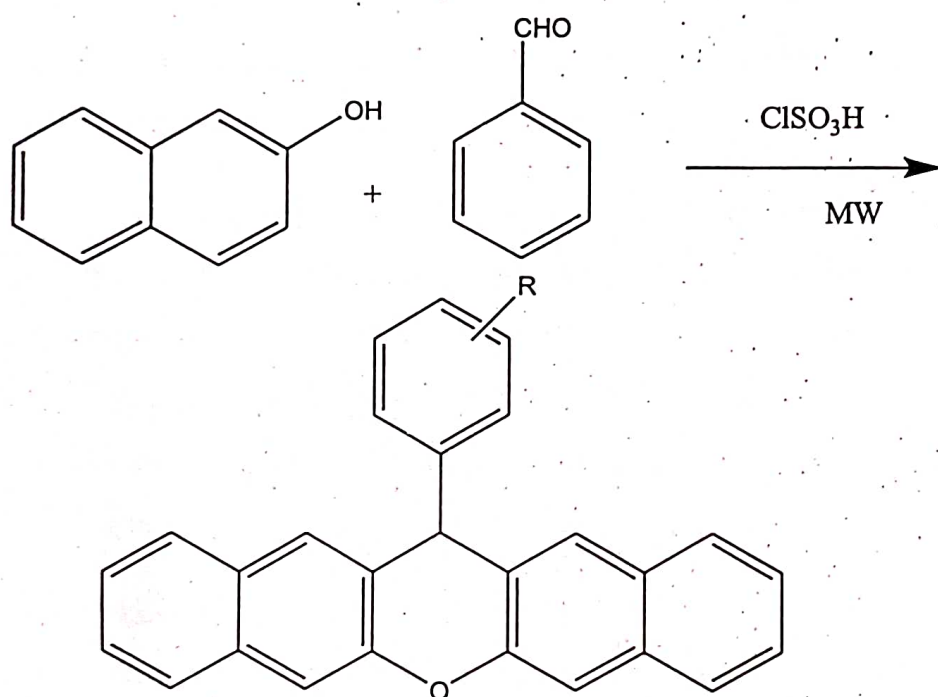
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1. Introduction:-

A heterocyclic compound forms the largest class of organic chemistry in recent years and is of immense important biologically and industrially. Nitrogen, sulphur, and oxygen containing five or six member Heterocyclic compounds are stable aromatic compounds, which are used in industries as varied as pharmaceuticals, paints and cosmetics etc. Xanthenes and benzoxanthene are very important class of heterocyclic compounds that are formed in widely used biologically active natural products and synthetic compounds of medicinal interest.

In recent year's multicomponents condensation reaction strategy, for the rapid and efficient generation of molecules because the produced are formed in a single step, which is carried out without isolation of any intermediate, this reduced time, save energy, saving money and raw material. Multicomponents condensation reaction by virtue of their convergence,, yields, production, ease of execution and high yields have attracted consideration attention in the fields of combinatorial chemistry, The first multicomponents reaction was investigated by Strecker in 1850 for the synthesis of amino acids. In the past decades, there have founds in tremendous application in three and four component reaction and great efforts are continually being made to develop new multicomponents reaction

In the present proposal, there is a need for developments of an alternative route to synthesis of Aryl 14H-dibenzoxanthene derivative. In this context we investigated the possibility of synthesizing of Aryl 14H-dibenzoxanthene derivatives through one-pot multicomponents condensation reaction of 2-naphthol with aryl aldehydes using chlorosulphonic acid catalyst under microwave irradiation under solvents free condition. The reaction is as-



The advantages of microwave irradiation in combinatorial chemistry become powerful tool in accelerating the pace of library synthesis. Microwave oven is most popularly apply in synthesis because the reaction proceed smoothly, low cost, readily available and atom economy for this reaction.

2. Literature survey:

In recent years, the application of microwave energy to accelerate organic reactions has gained popularity. Report on the use of microwave irradiation as a thermal source to carry out organic reactions. This advantageous over conventional methods due to shorter reaction times, dry media (thus avoiding the use of harmful solvents), cleaner reactions, easy work up, and minimization of thermal decomposition products. Using this environmentally benign protocol, several three-component reactions have been reported.

Naik et al [1] have studied the catalytic application of sulfated tin oxide for the synthesis of structurally diverse beta-acetamidoketones and Aryl 14H-dibenzoxanthene under microwave irradiation as a source of energy. The author also reported that the solid acid catalyst is found to be highly active for the synthesis of compounds in short reaction time with good yields and high purity of the products.

Shen et al [2] have reported the solvent free synthesis of xanthenediones and acridinediones catalyzed by methane sulfonic acid under thermal heating condition. The author also mentioned that the structure of synthesized compounds was confirmed by NMR, C-NMR and Mass spectra.

Shaterian et al [3] have reported Aluminum hydrogen sulfate as an efficient and heterogeneous catalyst for preparation of Aryl 14H-dibenzoxanthene derivatives under thermal and solvent free condition. The reactions were carried out at oil bath with short reaction time and produce high yields.

Shitole et al [4] have suggested an alternative method by using PEG-400 offers a convenient, non-toxic, thermally stable, inexpensive and recyclable reaction medium for synthesis of tetrahydrobenzoxanthene-11-one. They also reported the recyclability of the solvent makes the development of a green strategy and the reaction economically and potentially viable for commercial application.

Gui-yun Fu et al [5] have investigated a simple and convenient procedure for the synthesis of Aryl or Alkyl-14H-dibenzoxanthene using refundable $\text{HBF}_4\text{-SiO}_2$ catalyst under thermal and solvent free condition. This methodology offers very attractive features such as reduced reaction time higher yields, economic viability of catalyst and mild nature of silica-supported fluoroboric acid.

Kumar et al [6] have reported a novel one-pot synthesis of Aryl -14H-dibenzoxanthenes catalyzed by SelectfluorTM as catalyst under solvent free conditions.

Hazarkhani et al [7] have investigated N-bromosuccinimide as a mild catalyst for the efficient synthesis of dihydropyrimidinones using microwave irradiation as a source of energy. The advantages are the use of very cheap and safe reagent, nearly neutral reaction conditions, high yields and shorter reaction times under microwave irradiation. N, N-Dimethylacetamide Is a more superior solvent than EtOH, owing to the higher solubility of the substrate and the excellent energy transfer property.

Pasha et al. [8] have reported Zinc chloride-catalyzed one-pot synthesis of 3, 4-dihydropyrimidin-2(1H)-ones/thiones by] using microwave irradiation and solvent free condition. The same work has also reported [9] a cobalt (II) acetate-catalyzed, microwave-assisted Biginelli reaction in the absence of solvent.

Wang et al. [10] have studied a microwave-assisted, solvent-free condition for the synthesis of 3,4-dihydropyrimidinones by using poly (ethylene glycol)-bound sulfonic acid as a catalyst.

Rajitha et al. [11] have reported synthesis of benzofuran Mannich bases under solvent less p-toluenesulfonic acid or phase-transfer catalytic conditions in a domestic microwave oven. Thermal effects in the organ catalytic asymmetric Mannich reaction have been synthesized by Rodriguez et al [12] and found that under microwave irradiation, with only 0.5 mol% of catalyst (proline), products with up to 98% ee have been obtained after a shorter reaction time between cyclohexanone, formaldehyde, and substituted anilines.

Salehi and Guo [13] have investigated the synthesis of 1, 4-dihydropyridines in water using tetrabutylammonium bromide as a phase-transfer catalyst under microwave irradiation one pot condensation reaction of aldehydes, ethyl/methyl acetoacetates, and ammonium acetate. The reactions were completed in 3–10 mins. They also observed that the same reactions carried out without microwaves, even at 100°C did not result in significant yields in 10 mins. Terephthalaldehyde, a dialdehyde, has also been used as precursor for the by functional compounds containing two units.

Sivamurugan et al. [14] have reported A Lewis acid Zn [(L) proline] 2 catalyzed one-pot synthesis of Hantzsch 1,4-dihydropyridines by using microwave irradiation under solvent-less conditions.

Mu et al. [15] have described A solvent-free and catalyst-free, microwave-assisted method for the synthesis of α -aminophosphonates by the reaction of an aldehyde, an amine, and a dimethyl phosphate. The reactions conducted using pivalaldehyde, p-toluidine or 2,6-dimethylaniline resulted in much lower yields (53% and 40%) when compared to the reactions using other substrates.

Kidwai et al. [16] have found the synthesis of thiadiazolopyrimidin-2-thiones in dry media under microwave irradiation and solvent-free synthetic route for the synthesis of tetrahydroacridinones has been reported [17] by the same group via the microwave-assisted multicomponents condensation reaction of an aldehyde, dimedone and a primary aromatic amine. The most excellent microwave technique was established to be the "neat reaction" skill without the use of any solvent or inorganic solid supports. The reactions were finished within 5 mins with 82–87% yields, when compared to the conventional solution phase technique with longer reaction times (hours) and moderately lower yields. The other microwave solvent-free methods deliberate concerned the use of inorganic solid supports (basic and neutral alumina). Out of these two supports, basic alumina gave improved results than the neutral alumina. The same collected works has also advanced [18] a new way for the synthesis of acridine and quinazoline derivatives via the microwave assisted multicomponents condensation reaction of an aldehyde, dimedone, and ammonium acetate/thiourea performed on thin layer chromatography plates.

Yadav and Kapoor [19] have investigated one-pot synthesis of 4-aminobenzoxazinone derivatives by montmorillonite K-10 clay-supported reactions of substituted salicylaldehyde, N-substituted urea's (or carbonates'), and ammonium acetate (or an amine) under microwave-irradiation and solvent free condition

Dabiri et al. [20] have introduced one-pot condensation reaction of 4(3H)-quinazolinone derivatives by the reaction of isatoic anhydride, primary amines, and ortho esters in the presence of catalytic amounts of p-toluenesulfonic acid under microwave-irradiation and solvent free condition

Sridharan et al. [21] have investigated for the three-component stereo selective synthesis of tetrasubstituted isoxazolidines under microwave-irradiation and solvent free condition

Shelke et al [22] have studied solvent free, simple and general method for the synthesis of aryl 14H-dibenzo [a, j] xanthenes by using Chlorosulphonic acid catalyst under microwave irradiation. Its advantages are as follows: the catalyst is inexpensive and easily available, reaction time shorter and excellent yields are obtained. The same work has also reported for the synthesis of 14H-dibenzo[a,j]xanthene by condensation of β -naphthol and aldehydes in the presence of molecular iodine,[23] sulfamic acid[24], silica sulfuric acid[25], Amberlyst-15[26] and cation-exchange resins[27] as catalyst under solvent free conditions.

Above moiety we have different biological activities that why we decide to synthesis of aryl 14H-dibenzo [a, j] xanthenes. Different scientist synthesize by routine method but we synthesize by using microwave due to the use of this method we get better yield and also time required is also less.

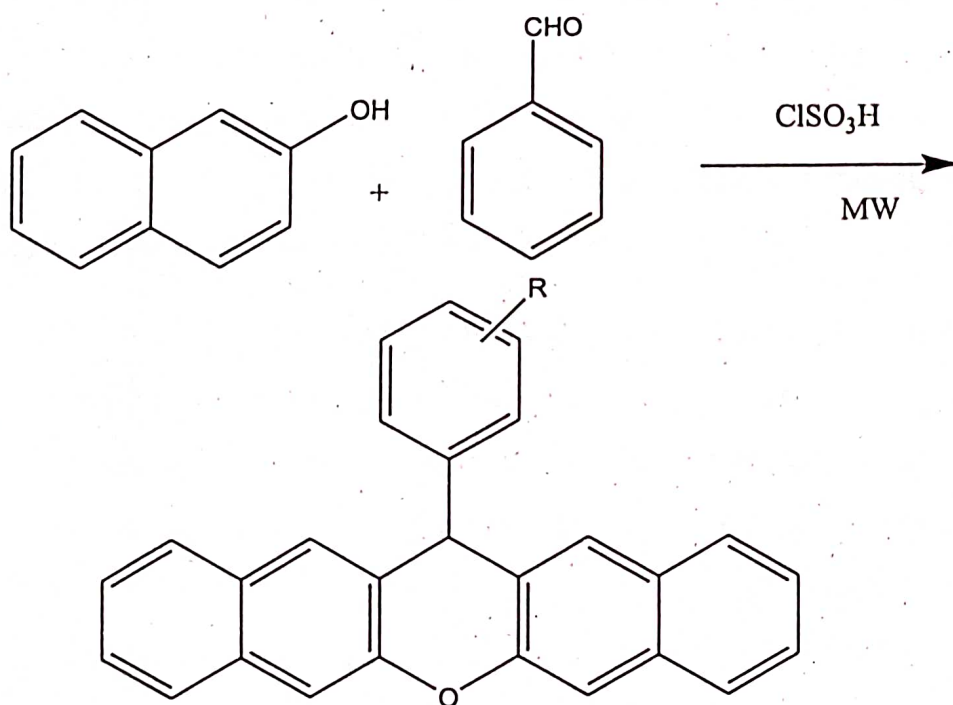
3. Experimental section:

All the organic chemicals and solvents were obtained from commercial sources (Sd fine, Loba) and used without further purification. Melting points were taken on a Viggo melting point apparatus and are uncorrected. Purity of the compounds was checked by TLC on silica gel coated aluminum plate in pet ether and ethyl acetate as solvent. The absorbance maxima (wavelength) were recorded on shimadzu UV visible spectrophotometer, $^1\text{H-NMR}$ was recorded on 300MHz using DMSO-d_6

A conventional household microwave oven operating at 900W was used for irradiation. All the products are known compounds and are characterized by comparing their physical and spectral characteristics with those reported in literature.

4. Procedure for synthesis of xanthenes:-

In the present work, a mixture of Para-nitro benzaldehyde (1 mmol), β -naphthol (2 mmol) and Chlorosulphonic acid (0.02 mmol) as a catalyst in a borosil beaker (50 ml) was exposed to microwave irradiation for 60 second. The reaction mixture was homogenized with the help of glass rod and the reaction was completed within two successive irradiation. The reactions were monitored by TLC. After completion of the reaction, crude reaction mixture, was poured on crushed ice. Thus obtained solid was filtered, dried, recrystallized from ethanol. The compounds are characterized by comparison of physical constant and ^1H NMR and IR spectra with those described in the literature.



5. Results and Discussion:

The objective of present research work is to provide simple and efficient methodologies for the synthesis of Aryl-14 H- dibenzo (a) xanthenes derivatives from aromatic aldehyde and 2-naphthol in presence of chlorosulphonic acid as a catalyst under solvent free conditions.

The reaction proceeded smoothly under microwave assisted solvent free condition to offer excellent yields.

Initially, we carried out the reaction of 2-naphthol and 4-nitro benzaldehyde in the presence of chlorosulphonic acid catalyst under microwave irradiation using different reaction condition. [Table] Excellent yield results were found when the reaction was carried out neat. Similarly the mole ratio of chlorosulphonic acid was studied and results are in table. It was found that the amount of ClSO_3H affects the yield of the desired product, with 0.02 mmole of ClSO_3H being optimum Table-1.

The reaction was performed with substituted benzaldehyde containing electron -donating as well as electron with- drawing groups, but electron donating group present on the benzaldehyde are generally more reactive than their corresponding electron- withdrawing group and give desired yield at shorter reaction time with excellent yield. [Table 1] This studied show clearly indicates that the synthesis of benzo xanthene is strongly affected by the electronic factors. The condensation reaction of ketones with 2-naphthol was not occurred such showed in the literature.

To show the merit of the present work is quite simple and convenient to synthesize aryl-14 H- dibenzo xanthenes (2,3) in excellent yield and high purity that precludes the use of toxic solvent and hazardous conventional minerals etc. encouraged by these results, this method is advantages in term of short reaction time, quantitative yield of the products.

Table: - 1. Synthesis of 14-aryl 14H-dibenzo xanthenes derivatives in presence of chlorosulphonic acid as catalyst from β -naphthol and aromatic aldehydes under microwave irradiation and solvent-free conditions.

Sr. No.	Entry	time	Power	yield	catalyst	m.p.
1	Ph-CHO	2.00	720 W	93 %	0.02 mmole	182-184
2	P-NO ₂ - C ₆ H ₆ CHO	2.30	720 W	94 %	0.02 mmole	306-308
3	P-Chloro- C ₆ H ₆ CHO	2.00	720 W	94 %	0.02 mmole	287-289
4	m-Nitro- C ₆ H ₆ CHO	2.00	720 W	95 %	0.02 mmole	209-210
5.	P-OH C ₆ H ₆ CHO	2.30	720 W	92%	0.02 mmole	141
6.	O-Nitro- C ₆ H ₆ CHO	2.30	720 W	94%	0.02 mmole	292
7.	P-OCH ₃ C ₆ H ₆ CHO	3.00	720 W	89%	0.02 mmole	199

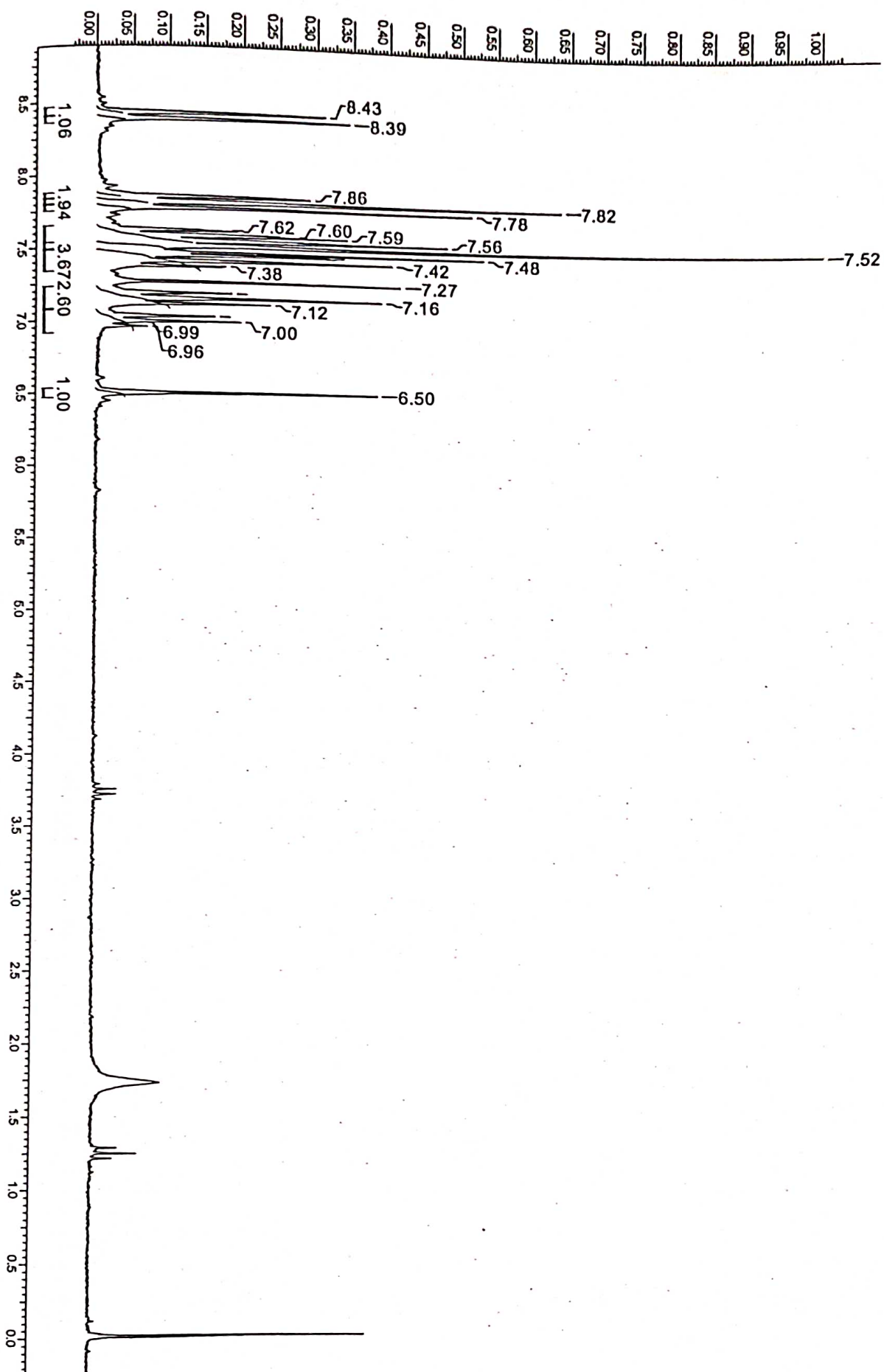
Yields refer to pure isolated products. All known products have been reported previously in the literature and were characterized by comparison of IR and NMR spectra.

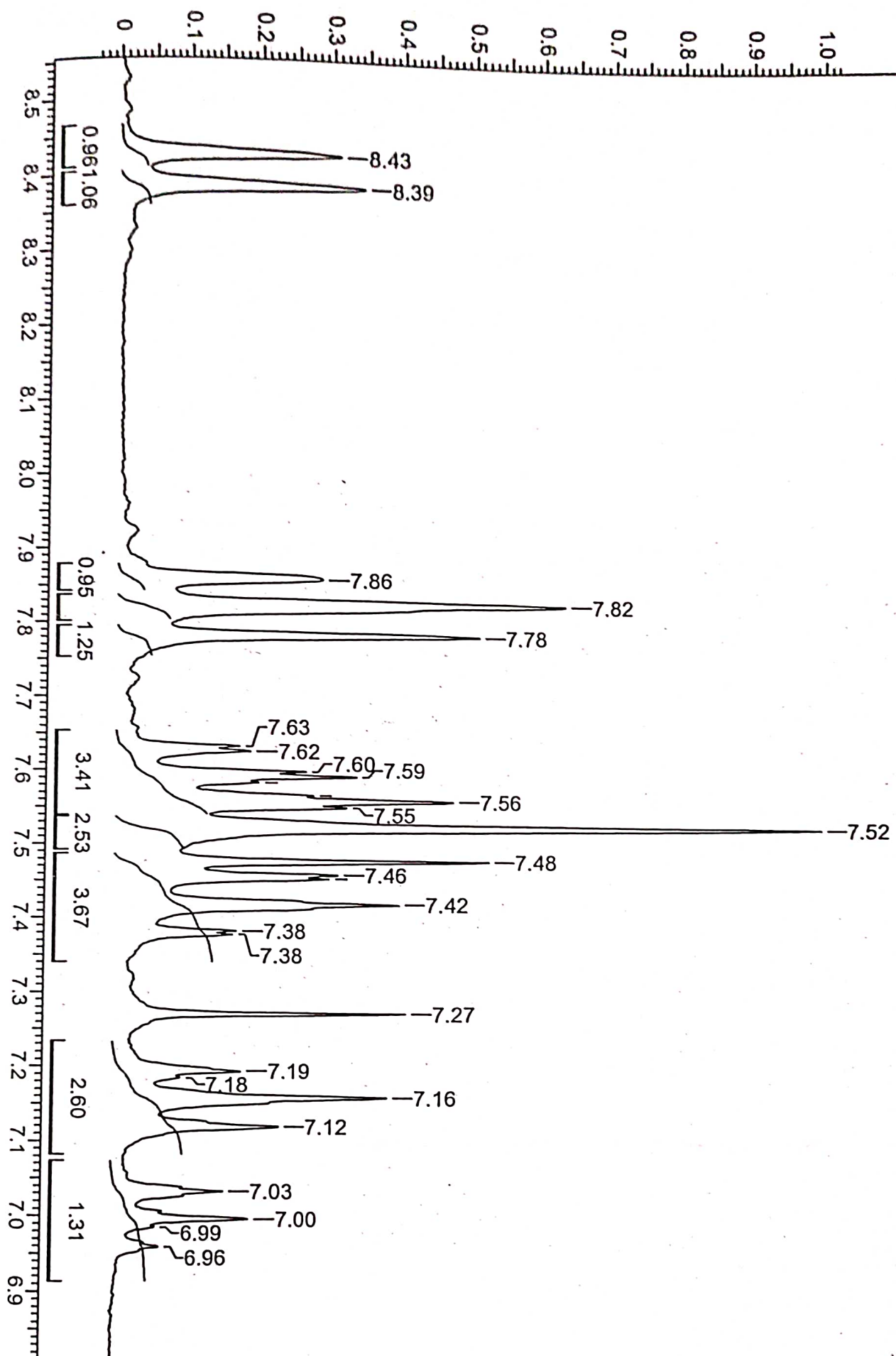
Following the success from the synthesis of aryl-14 H- dibenzo (2,3) xanthene by condensation aryl aldehyde with 2-Naphthol in presence of ClSO_3H as catalyst under microwave irradiation and solvent free condition. The spectral data of selected compounds are below.

6. Spectral Analysis:

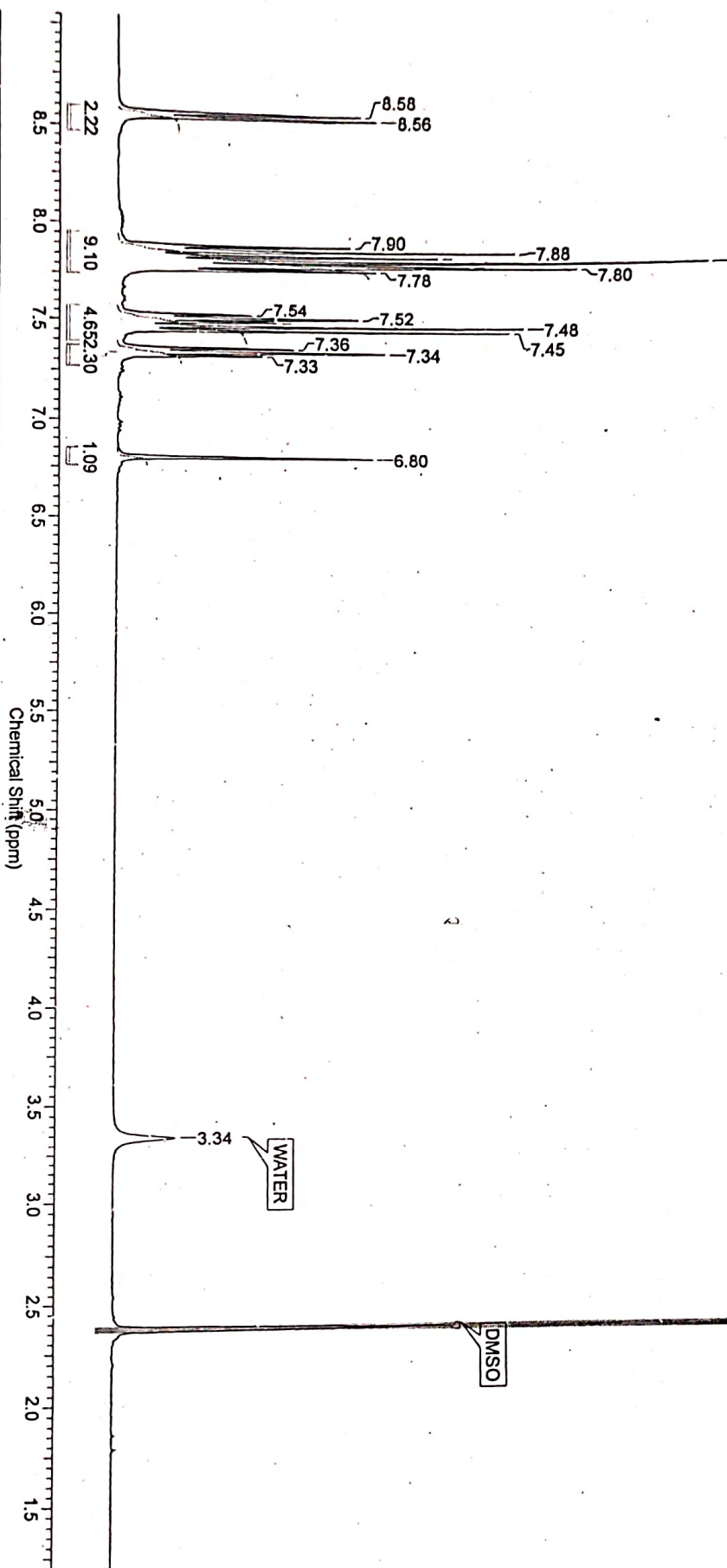
1. 14[4- nitro phenyl]-14 H dibenzo (b) xanthene.[Table No. 1 Entry-2]
Yellow solid M.P. 306-308 (lit. Value 310°C) IR [KBR] V_{\max} cm^{-1} 2930, 1594, 1517., ^1H NMR [300 MHz DMSO] δ 6.80 (s, 1H), δ 7.12 to 8.5 (m, 16 H).
- 2 14[4- Chlorophenyl]-14 H dibenzo (a) xanthene.[Table No. 1 Entry-3]
White solid, M.P. 287-289°C (lit. Value 289°C) IR [KBR] V_{\max} cm^{-1} 2925, 1590, 1484. ^1H NMR [300 MHz DMSO] δ 6.75 (s, 1H), δ 7.12 to 8.75 (m, 16 H).
3. 14-(4-Hydroxyphenyl)-14*H*-dibenzo [*a,j*]xanthene (Table 1, entry 5).
Pink solid: mp.141 oC IR (KBr, cm^{-1}): 3404, 1592, 1511, 1401, 1250, 1242, 816;
 ^1H NMR (300 MHz, CDCl_3): δ =4.97 (br s, 1H, OH), 6.42(s, 1H, CH), 6.56-8.36 (m, 16H, Ar-H).
4. 14-(2-Nitrophenyl)-14*H*-dibenzo [*a,j*]xanthene (Table 1, entry 6). Yellow solid: mp 292 oC.IR (KBr, cm^{-1}): 3400, 3058, 1593, 1523, 1350, 1240, 1142, 810, 748; ^1H NMR (300 MHz, CDCl_3): δ = 7.52 (s, 1H) 7.10-8.56 (m, 16H).

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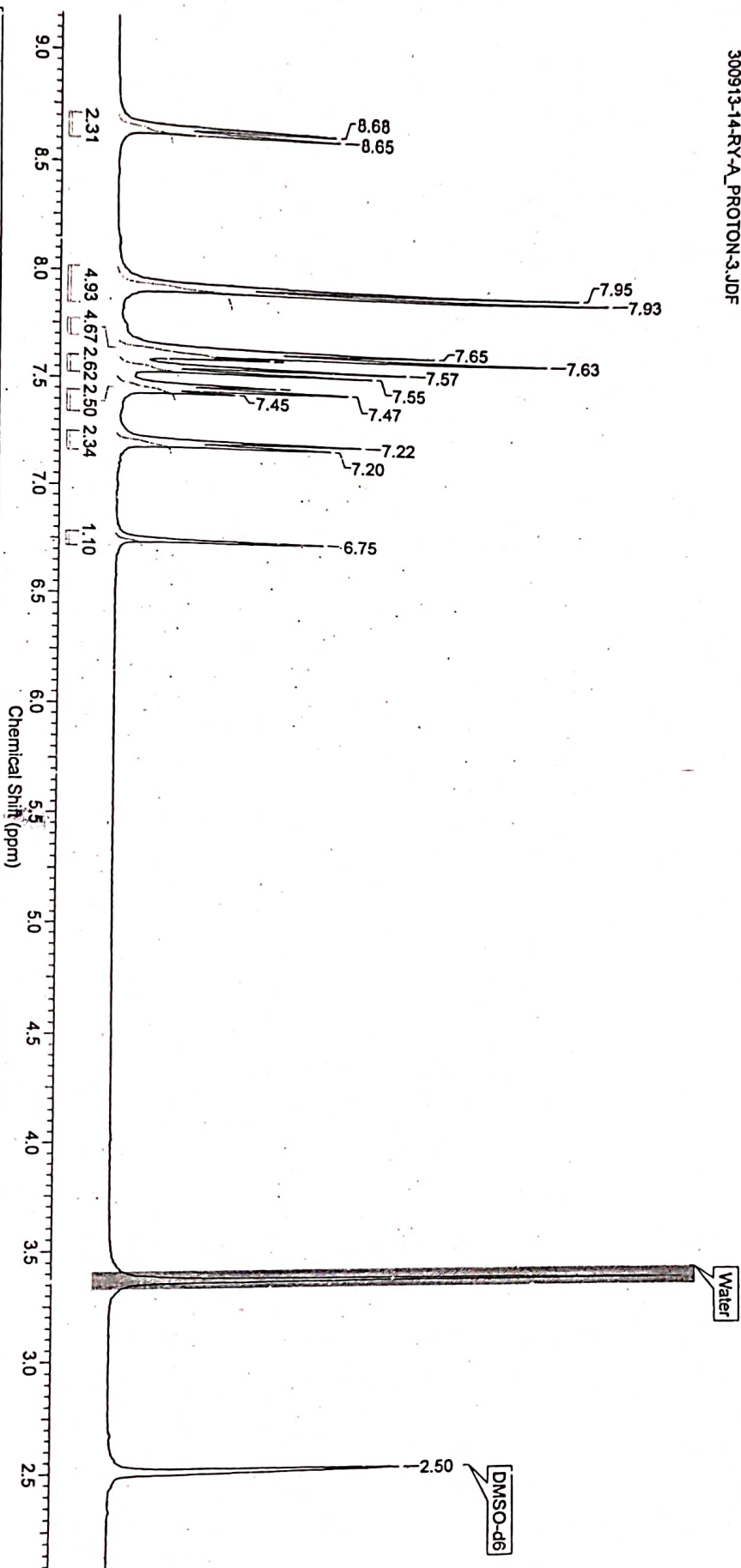




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2	3.34	WATER	1	hkbavireddi	Sun 10/27/2013 11:24:40 AM		



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2	[3.33 .. 3.41]	Water	1	hkbavireddi	Sun 10/27/2013 11:12:48 AM		

7. Conclusion:

In this present project we are reporting the most convenient way to synthesize the Xanthene derivatives in which microwave irradiation plays an important role for promoting condensation reaction of P-nitrobenzaldehyde and 2-naphthol. In conclusion, in this work we investigated a simple efficient and rapid method for the synthesis of aryl 14H-dibenzo [a, j] xanthenes under microwave irradiation method.

The application of microwave irradiation in combinatorial chemistry becomes potent device in accelerating the pace of library synthesis. Domestic microwave oven is very popularity used in organic synthesis because of its low cost and easily available, shorter reaction time and products are obtained in excellent yields. Furthermore this methodology also follows several principles of green chemistry.

8. Recommendations for future work:-

The application of the present method to biological sample should be feasible, in spite of the fact that further investigations are necessary.

9. Papers presented: -

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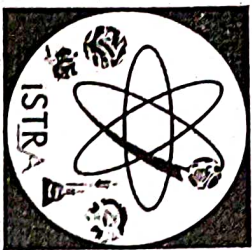
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