Article

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Convenient Synthesis of 14-Aryl-14-H-dibenzo[*a,j*]xanthenes Catalyzed by Acyclic Brønsted Acidic Ionic Liquid $[H–NMP]^+[HSO_4]^-$ under Microwave Irradiation

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Efficient method for direct preparation of 14-aryl-14-H-dibenzo[*a,j*]xanthenes through condensation of β-naphthol with various aromatic aldehydes in the presence of the catalytic amount of $[H–NMP]^+[HSO_4]^-$ under microwave irradiation was described. This method has the advantages such as; very easy reaction workup, absolute separation of catalyst from the reaction mixture and smooth recyclability of catalyst. In this reaction 14-aryl-14-H-dibenzo[*a,j*]xanthenes were obtained as desired products in excellent yields and short reaction times via green and one-pot procedure.

Keywords: β-Naphthol; Aromatic aldehyde; Ionic liquid; Microwave irradiation; Xanthene.

INTRODUCTION

Microwave-assisted organic synthesis (MAOS) has been known since 1986.[1] This "non-conventional" synthetic method has shown broad applications as a very convenient way to accelerate the course of many organic reactions, producing high yields and short reaction times, lower quantities of side products and easier work-up and formation of cleaner products. MAOS is considered as a "green" technology, principally since many organic reactions can be carrying out in solvent-free conditions.[2-3] Xanthenes especially benzoxanthenes, has emerged as a powerful tool in organic synthesis due to their wide range of useful pharmacological activity such as antibacterial,[4] anti-inflammatory,[5] antiviral properties.[6] Furthermore, these heterocyclic compounds are used as photodynamic therapy[7] and also utilized for antagonism of the paralyzing action of zoxazolamine. Benzoxanthenes are used as dyes,[8] laser technology,[9] and in fluorescent materials for visualization of biomolecules.[10] These compounds have been synthesized by many procedures. Some of the important methods are include, trapping of benzynes by phenol,[11] intermolecular phenyl carbonyl coupling reactions of aldehydes and β-naphthol,[12] cyclodehydrations,[13] carbon monoxide.[14] Furthermore, the reaction of aryloxymagnesium halides with triethylorthoformate,[15] intra molecular phenyl carbonyl coupling reactions of benzaldehydes and acetophenones,[16] cyclization of polycyclic aryltriflate esters,[17] and 2-tetralone.[18]

The most common synthesis of xanthenes are included the preparation of 14-H-dibenzo[*a,j*]xanthene has been reported by condensation of β-naphthol and aldehydes in the presence of *p*-toluenesulfonic acid,[19,20] molecular iodine,[21] $K_5CoW_{12}O_{40}.3H_2O$/silica-gel/MW,[22] LiBr/MW,[23] amberlyst-15,[24] α-iodoacetates from alkenes/ammonium acetate/I_2,[25] cation exchange resins[26] as catalyst isonitriles,[27] silicasulfuric acid.[28] The synthesis of heterocycles under microwave irradiation has attracted much attention in synthetic organic chemistry. However, many of these methods suffer a drawback, including prolonged reaction times, low yield, harsh reaction conditions, toxic organic solvents and excess amount of catalysts.

In this research, we hope to develop a simple, efficient method for synthesis of 14-aryl-14-H-dibenzo[*a,j*]xanthenes by treatment of β-naphthol and aromatic aldehydes in the presence of an ionic liquid as homogeneous catalyst under mild conditions.

RESULTS AND DISCUSSION

In this research, in order to optimization of reaction conditions, it was carried out the reaction of β-naphthol and 3-nitrobezaldehyde in a 2:1 ratio as a model substrate in the presence of various catalytic amounts of $[H–NMP]^+[HSO_4]^-$ as a homogeneous catalyst under microwave irradiation at different powers (Scheme I).

The obtained results from the reaction of determining the optimum power are presented in Table 1. As it can be seen in this reaction, the best results were obtained using power of 450 W. After optimization of the different powers, the reaction of β-naphthol with several aldehydes was carried out in various solvents. The best results were obtained using ethanol solution (Table 2, entry 4).

After optimization of the reaction conditions, in order

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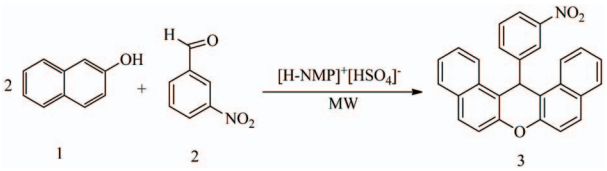
Scheme I Reaction of β-naphthol and 3-nitrobezaldehyde



to develop this protocol the reaction of β-naphthol with several aldehydes was carried out in according to the general experimental procedure. The corresponding products are summarized in Table 3. As it can be seen in this Table, the best activity of aromatic aldehyde was shown in the presence of an electron-withdrawing substituent (such as $–NO_2$) in the para position. The presence of electron-donating substituent (such as –OH) was decreased both the reaction rate and the yield of product (Table 3, entry 11).

In this study, 14-aryl-14-H-dibenzo[*a,j*]xanthenes as products in a new method were prepared using the model reaction of β-naphthol and various aromatic aldehyhes in the presence of the homogeneous catalytic amount of $[H–NMP]^+[HSO_4]^-$ (20 mol %) at 450 W microwave irradiation in ethanol. After the separation of the product, the catalyst as a by-product is removable by washing with water, and easily recycled to catalyze the preparation of 14-aryl-14-H-dibenzo[*a,j*]xanthenes with excellent yields.

The reusability of catalyst was studied through the completely separation of solid products (**3g**) with water. Then, the water containing Brønsted acidic ionic liquid (BAILs is soluble in water) was evaporated under vacuum and the catalyst was recycled for several times without any decrease in catalytic activity; the yields ranged from 95% to 86% (Fig. 1).

The structures of products were confirmed by spectroscopic and physical data such as; IR, ^{1}H NMR, ^{13}C NMR

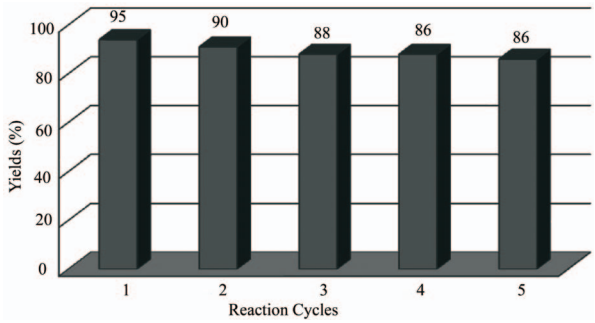


Fig. 1. Reusability of the catalyst in the synthesis of **3g**.

Table 1. The synthesis of **3g** under microwave at different powers in ethanol

Entry	Power	Time (min)	Yield[a] (%)
1	100	20	30
2	180	10	50
3	300	6	65
4	450	4	95

[a] Isolated yields.

Table 2. The synthesis of **3g** in different solvents under microwave irradiation (450 W)

Entry	Solvent	Time (min)	Yield[a] (%)
1	H_2O	11	10
2	CH_2Cl_2	9	50
3	DMF	8	30
4	EtOH	4	95

[a] Isolated yields.

Table 3. One-pot synthesis of various 14-aryl-14-H-dibenzo[*a,j*]xanthenes using the catalytic amount of $[H–NMP]^+[HSO_4]^-$ as catalyst under microwave irradiation at 450 W

Entry	Aldehyde	Product	Time (min)	Yield[a]
1	C_6H_5	**3a**	4	93
2	*o*–Cl.C_6H_4	**3b**	5	87
3	*p*–Cl.C_6H_4	**3c**	3.5	96
4	*2,4*–Cl_2.C_6H_4	**3d**	4.5	92
5	*2,3*–Cl_2.C_6H_4	**3e**	4	90
6	*o*–NO_2.C_6H_4	**3f**	4	86
7	*m*–NO_2.C_6H_4	**3g**	3.5	95
8	*p*–NO_2.C_6H_4	**3h**	3	97
9	*4*–Cl–*3*–NO_2.C_6H_3	**3i**	3.5	95
10	*m*–OH.C_6H_4	**3j**	6	90
11	*p*–OH.C_6H_4	**3k**	7	78
12	*m*–Me.C_6H_4	**3l**	5.5	88
13	*p*–Me.C_6H_4	**3m**	5.5	85
14	*m*–OMe.C_6H_4	**3n**	6.5	86
15	*p*–OMe.C_6H_4	**3o**	6	83

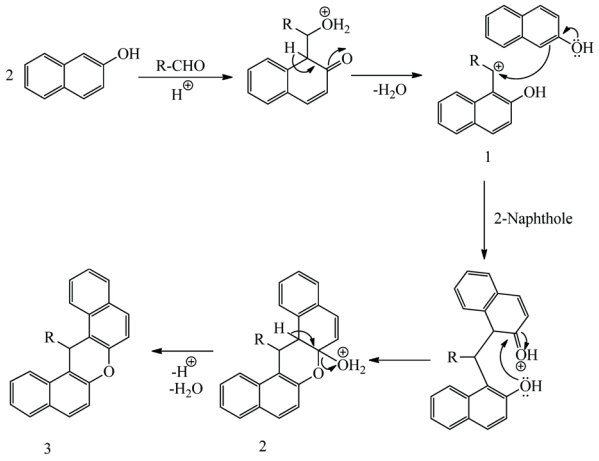
[a] Isolated yields.

and UV–vis. The infrared spectra of the 14-(phenyl)-14-H-dibenzo[*a,j*]xanthene exhibit a band at 1248 cm^{-1} assigned to υ (C–O). In the ^{1}H NMR spectra the signal around δ = 6.98-8.41 ppm is assigned to the protons of the aromatic rings υ (CH=CH), the signal of the aliphatic rings υ (CH) is shown at 6.5 ppm and the ^{13}C NMR spectrum, the signal around δ = 117.34-148.74 ppm is assigned to the carbons of the aromatic rings (CH=CH) and the signal of the aliphatic rings υ (CH) is shown at 38.07 ppm.

The proposed reaction mechanism

A plausible mechanism in the formation of 14-aryl-14-H-dibenzo[*a,j*]xanthenes is presented in Scheme II. As it can be seen, the reaction likely proceeds via initial formation of carbocation (1). Then, the oxonium species (2) is formed in the reaction with β-naphthol, which undergoes dehydration to afford the desired product (3).

Scheme II Proposed reaction mechanism



CONCLUSIONS

In this paper, we have described the synthesis of 14-aryl-14-H-dibenzo[*a,j*]xanthenes using β-naphthol with substituted aromatic aldehydes. This reaction was performed in the presence of catalytic amount of [H–NMP]$^+$[HSO_4]$^-$ as an efficient, inexpensive, non-toxic, easy work-up and reusable catalyst make the present procedure eco-friendly and economically acceptable.

EXPERIMENTAL

Materials. All commercially available reagents were used without further purification and purchased from the Merck Chemical Company in high purity. The used solvents were purified by standard procedure.

Apparatus. IR spectra were obtained as KBr pellets on a Perkin–Elmer 781 spectrophotometer and on an impact 400 Nicolet FT–IR spectrophotometer. ^{1}H NMR and ^{13}C NMR were recorded in $CDCl_3$ solvents on a Bruker DRX-400 spectrometer with tetramethylsilane as internal reference. Uv–vis spectra were obtained with a Perkin–Elmer 550 was recorded in $CDCl_3$ solvents. Melting points were obtained with a Yanagimoto micro melting point apparatus are uncorrected. Microwave irradiations were carried out in microwave oven specially designed for organic synthesis (Milestone LAVIS 1000 Basic Microwave). The purity determination of the substrates and reaction monitoring were accomplished by TLC on silica-gel polygram SILG/UV 254 plates (from Merck Company).

General procedure for the synthesis of [H–NMP]$^+$[HSO_4]$^-$. 1-Methyl-2-pyrolidone (0.2 mol) was charged into a 250 mL three necked flask with a magnetic stirrer. Then equimolar concentrated sulfuric acid (98 wt %) was added drop wise slowly into the flask at 80 °C for 12 h. The mixture was washed by diethyl ether three times to remove non-ionic residues and dried in the vacuum by a rotary evaporator to obtain the viscous clear [H–NMP]$^+$[HSO_4]$^-$.[29]

General procedure for the synthesis of 14-aryl-14-H-dibenzo[*a,j*]xanthenes. To a mixture of an aldehyde (1 mmol), β-naphthol (2 mmol, 0.288 g), [H–NMP]$^+$[HSO_4]$^-$ (20 mol %), EtOH as solvent was added and the mixture was inserted in a microwave oven and heated at 450 W for the appropriate time (Table 1) to yield the desired products. The progress of the reactions was monitored by TLC (ethyl acetate/petroleum ether 3/7).

14-(Phenyl)-14H-dibenzo[*a,j*]xanthene (3a). Pale yellow solid, m.p = 183-184 °C, (m.p = 182-183 °C),[30] Uv–vis (CH_2Cl_2) λ*max*: 244, 230; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 6.5 (s, 1H, CH), 6.98-7.01 (t, 1H, *J* = 7.6 Hz, Ar), 7.13-7.17 (t, 2H, *J* = 7.6 Hz, Ar), 7.40-7.43 (t, 2H, *J* = 7.6 Hz, Ar), 7.48-7.54 (m, 4H, Ar), 7.56-7.60 (t, 2H, *J* = 7.2 Hz, Ar), 7.92-7.81 (d, 2H, *J* = 8.8 Hz, Ar), 7.82-7.84 (d, 2H, *J* = 8.0 Hz, Ar), 8.39-8.41 (d, 2H, *J* = 8.8 Hz, Ar); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 148.74, 145.03, 131,48, 131.07, 128.88, 128.82, 128.5, 128.29, 126.81, 126.41, 124.26, 122.72, 118.04, 117.34, 38.07; IR (KBr) υ (cm^{-1}): 3061, 1624, 1592, 1513, 1458, 1410, 1248, 1078, 962, 805, 744. **14-(2-Chlorophenyl)-14H-dibenzo[*a,j*]xanthene (3b).** White solid, m.p = 212-213 °C, (m.p = 214-215 °C)[30], UV–vis (CH_2Cl_2) λ*max:* 246, 230; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 6.81 (s, 1H, CH), 6.92 (m, 2H, Ar), 7.40-7.44 (m, 3H, Ar), 7.48-7.51 (d, 2H, *J* = 8.8 Hz, Ar), 7.61-7.64 (m, 5H, Ar), 8.74-8.76 (d, 2H, *J* = 8.4 Hz, Ar); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 148.95, 143.59, 131.83, 130.91, 130.15, 129.61, 129.06, 128.67, 127.95, 127.88, 126.94, 124.45, 123.49, 118.12, 118.01, 34.65; IR (KBr) υ (cm^{-1}): 3059, 1625, 1592, 1461, 1400, 1243, 1032, 959, 808. **14-(4-Chlorophenyl)-14H-dibenzo[*a,j*]xanthene (3c)**. Yellow solid, m.p = 289-290 °C, (m.p = 287-288 °C),[30] UV–vis (CH_2Cl_2) λ*max*: 244, 230; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 6.48 (s, 1H, CH), 7.10-7.12 (d, 2H, *J* = 8.4 Hz, Ar), 7. 41-7.47 (m, 4H, Ar), 7.48-7.50 (d, 2H, *J* = 8.8 Hz, Ar), 7.57-7.61 (t, 2H, *J* = 7.6 Hz, Ar), 7.80-7.82 (d, 2H, *J* = 8.8 Hz, Ar), 7.84-7.86 (d, 2H, *J* = 8.0 Hz, Ar), 8.32-8.34 (d, 2H, *J* = 8.4 Hz, Ar); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 148.51, 145.01, 131.39, 131.31, 131.20, 130.18, 129.83, 129.19, 128.91, 127.60, 125.14, 123.77, 118.21, 117.50, 39.87; IR (KBr) υ (cm^{-1}): 3067, 1624,

1591, 1514, 1585, 1431, 1243, 1085, 961, 808, 743. **14-(2,4-Dichlorophenyl)-14H-dibenzo[*a,j*]xanthene (3d).** Pale yellow solid, m.p = 229-230 °C, (m.p = 227-228 °C),[30] UV–vis (CH_2Cl_2) λ*max*: 248, 230; ^{1}H NMR ($CDCl_3$, 400 MHz δ: 6.77 (s, 1H, CH), 6.90-6.92 (d, 1H, Ar), 7.28-7.32 (m, 2H, Ar), 7.43-7.50 (m, 4H, Ar), 7.61-7.65 (t, 2H, *J* = 7.6 Hz, Ar), 7.81-7.85 (t, 4H, *J* = 9.0 Hz, Ar), 8.65-8.67 (d, 2H, *J* = 8.0 Hz, Ar); ^{13}C NMR/ ($CDCl_3$, 100 MHz) δ: 148.9, 142.25, 132.8, 132.71, 132.6, 130.92, 130.59, 129.3, 128.78, 128.41, 127.06, 124.57, 123.17, 118.11, 117.46, 34.24; IR (KBr) υ (cm^{-1}): 3061, 1622, 1591, 1514, 1464, 1430, 1401, 1247, 1103, 1072, 960, 864, 836, 807, 743. **14-(2,3-Dichlorophenyl)-14H-dibenzo[*a,j*]xanthene (3e).** White solid, m.p = 250-253 °C, UV–vis (CH_2Cl_2) λ*max*: 248, 230; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 6.85 (s, 1H, CH), 6.87-6.89 (d, 1H, *J* = 8.0 Hz, Ar); 7.09-7.11 (d, 2H, *J* = 8.8 Hz, Ar), 7.32-7.35 (d, 1H, *J* = 9.0 Hz, Ar), 7.44-7.51 (m, 4H, Ar), 7.62-7.85 (t, 4H, *J* = 9.2 Hz, Ar), 8.69-8.67 (d, 2H, *J* = 8.40 Hz, Ar); IR (KBr) υ (cm^{-1}): 3057, 1625, 1593, 1514, 1459, 1405, 1401, 1251, 1107, 1070, 962, 869, 813, 807, 746. **14-(2-Nitrophenyl)-14H-dibenzo[*a,j*]xanthene (3f).** Yellow solid, m.p = 213-214 °C, (m.p = 214-215 °C),[30] UV–vis (CH_2Cl_2) λ*max*: 244, 230; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 7.08 (t, 1H, *J* = 8.0 Hz, CH), 7.23-7.27 (t, 1H, *J* = 7.2 Hz, Ar), 7.42-7.46 (t, 2H, *J* = 7.2 Hz, Ar), 7.42-7.49 (m, 3H, Ar), 7.57-7.63 (m, 3H, Ar), 7.81-7.84 (m, 4H, Ar), 8.53-8.55 (d, 2H, *J* = *8*.4 Hz, Ar); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 149.4, 147.03, 140.87, 134.14, 132.26, 131.72, 130.97, 129.47, 128.73, 127.59, 127.41, 124,92, 124,67, 122.58, 118.03, 117.58; IR (KBr) υ (cm^{-1}): 3057, 1625, 1593, 1500, 1397, 1346, 1305, 817, 750. **14-(3-Nitrophenyl)-14H-dibenzo[*a,j*]xanthene (3g).** Yellow solid, m.p = 212-213 °C, (m.p = 210-211 °C),[30] UV–vis (CH_2Cl_2) λmax: 246, 228; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 6.62 (s, 1H, CH), 7.28-7.32 (t, 1H, *J* = 7.6 Hz, Ar), 7.43-7.47 (t, 2H, *J* = 7.6 Hz, Ar), 7.51-7.53 (d, 2H, *J* = 8.8 Hz, Ar), 7.60-7.64 (t, 2H, *J* = 7.2 Hz, Ar), 7.81-7.87 (m, 6H, Ar), 8.30-8.32 (d, 2H, *J* = 8.4 Hz, Ar), 8.42 (s, 1H, Ar); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 148.77, 148.21, 146.94, 134.27, 131.04, 129.57, 129.49, 129.07, 127.25, 124.59, 122.71, 122.04, 121.69, 118.13, 115.87, 37.71; IR (KBr) υ (cm^{-1}): 3075, 1592, 1527, 1500, 1397, 1346, 1252, 1080, 958, 812, 748. **14-(4-Nitrophenyl)-14H-dibenzo[*a,j*]xanthene (3h).** Pale yellow solid, m.p = 308-309 °C, (m.p = 310-311 °C),[30] UV–vis (CH_2Cl_2) λ*max*: 248, 228; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 6.61 (s, 1H, CH), 7.43-7.47 (t, 2H, Ar), 7.50-7.53 (d, 2H, Ar), 7.59-7.63 (t, 2H, Ar), 7.68-7.70 (d, 2H, Ar), 7.84-7.89 (t, 4H, Ar), 8.00-8.02 (d, 2H, Ar), 8.28-8.30 (d, 2H, Ar); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 152.01, 148.77, 146.29, 131.06, 129.6, 129.07, 128.97, 127.19, 124.59, 123.87, 122.04, 118.07, 115.76, 37.87; IR (KBr) υ (cm^{-1}): 3068, 1593, 1515, 1341, 1243, 824, 745. **14-(4-Chloro-3-nitrophenyl)-14H-dibenzo[*a,j*]xanthene (3i).** Yellow solid, m.p = 232-234 °C, UV–vis (CH_2Cl_2) λmax: 246, 232; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 7.27 (s, 1H, CH), 7.29-7.31 (d, 1H, *J* = 8.4 Hz, Ar), 7.45-7.52 (m, 4H, Ar), 7.62-7.65 (m, 3H, Ar), 7.84-7.89 (t, 4H, *J* = 9.2 Hz, Ar), 8.03-8.04 (d, 1H, *J* = 2.0 Hz, Ar), 8.24-8.26 (d, 2H, *J* = 8.4 Hz, Ar); IR (KBr) υ (cm^{-1}): 3054, 1623, 1592, 1530, 1462, 1350, 1246, 952, 812, 742. **14-(4-Hydroxyphenyl)-14H-dibenzo[*a,j*]xanthene (3k).** Pink solid, m.p = 137-138 °C, (m.p = 138-140 °C),[32] ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 4.97 (br s, 1H, OH), 6.43 (s, 1H, CH), 6.55-8.36 (m, 16H, Ar); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 37.41, 115.70, 118.00, 118.39, 123.10, 124.62, 127.23, 129.11, 129.20, 129.78, 131.53, 131.81, 137.90, 149.11, 154.24; IR (KBr) υ (cm^{-1}): 3404,1592, 1511, 1401, 1250, 1242, 816. **14-(4-Methyl)-14H-dibenzo[*a,j*]xanthene (3m).** Pale yellow solid, m.p = 228-230 °C, (m.p = 227-228 °C),[30] UV–vis (CH_2Cl_2) λ*max*: 247, 232; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 6.46 (s, 1H, CH), 6.95-6.97 (d, 2H, *J* = 8.0 Hz, Ar), 7.39-7.43 (m, 4H, Ar), 7.47-7.50 (d, 2H, *J* = 9.2 Hz, Ar), 7.56-7.6 (t, 2H, *J* = 9.2 Hz, Ar), 7.78-7.80 (d, 2H, *J* = 8.8 Hz, Ar), 7.82-8.84 (d, 2H, *J* = 8.0 Hz, Ar), 8.39-8.41 (d, 2H, *J* = 8.8 Hz, Ar); ^{13}C NMR ($CDCl_3$, 100) δ: 20.91, 37.64, 117.46, 118.02, 122.72, 124.22, 126.77, 128.11, 128.77, 128.79, 129.19, 131.08, 131.46, 135.91, 142.14, 148.68; IR (KBr) υ (cm^{-1}): 3069, 1623, 1591, 1531, 1458, 1399, 1244, 961, 810, 741

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