Facile Catalyzed Preparation of 14-Aryl- or Alkyl-14-*H*-dibenzo[a,j]xanthenes by Dodecylphosphonic Acid and Dodecylsulfamic Acid: Environmentally Benign Methods

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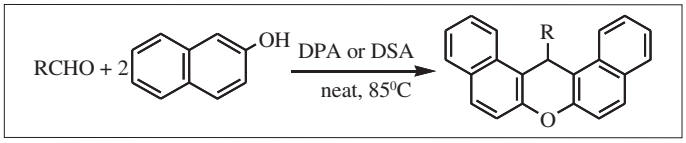

Under mild- and solvent-free conditions, one-pot preparation of 14-alkyl- or 14-aryl-14-H-dibenzo[a,j] xanthenes could proceed in the presence of catalytic amounts of dodecylphosphonic acid (DPA) and dodecylsulfamic acid (DSA) in good to excellent yields. In these reactions, DPA and DSA are recovered by straightforward work-ups.

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INTRODUCTION

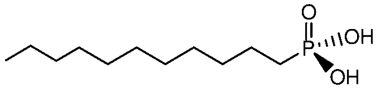
Benzoxanthenes are flat rigid structures, which have been used as a linker for peptide synthesis [1] and in unnatural amino acids and related pharmaceutical precursors [2]. Furthermore, because of their useful spectroscopic properties, they are used as dyes [3] in laser technologies [4] and in fluorescent materials for visualization of biomolecules [5]. Many synthetic methods exist for the synthesis of benzoxanthenes, such as the cyclocondensation reaction of 2-hydroxyaromatic aldehydes and 2-tetralone [6], the reaction of benzaldehyde and acetophenone [7], and the condensation of β-naphthol with alkyl or aryl aldehydes. The latter synthetic method can be promoted by many Brønsted acid catalysts such as H_2SO_4 [8], *p*-toluenesulfonic acid [9], sulfamic acid [10], methanesulfonic acid [11], H_3PO_4 or $HClO_4$ at 0°C in acetic acid [12], HCl [13], heterogonous solid acids such as Montmorillonite K10 [14], Indion-130 [15], and Amberlyst-15 [16], heteropolyacid [17], cellulose sulfuric acid [18], [MIMPS]HSO_4 [19], BSA [20], and $NaHSO_4/SiO_2$ [21]. However, many of these procedures have significant drawbacks such as low yields, long reaction times, difficulties in work-up, use of an excess of reagents/ catalysts, harsh reaction conditions, and use of toxic organic solvents. Thus, the development of more efficient procedures for the synthesis of benzoxanthenes is still needed. In recent years, the use of solid acidic catalysts has offered important advantages in organic synthesis, such as environmental compatibility, ease of handling, and low cost. Dodecylphosphonic acid (DPA) (Scheme 1) is a solid Brønsted acid that recently has been used as efficient Brønsted acid catalyst for Friedlander synthesis of poly substituted quinolines and Biginelli condensation [22]. In connection with our interest to introduce DPA abilities as a new catalyst in organic chemistry, we report here its application for preparation of 14-alkyl- or aryl-14-H-diben-zo[a,j] xanthenes from aldehydes and β-naphthol. Furthermore, we also wish to introduce dodecylsulfamic acid (DSA) (Scheme 1) as another effective catalyst for this conversion.

RESULTS AND DISCUSSION

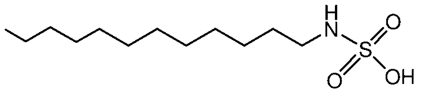
This article describes two favorite methods for the synthesis of benzoxanthenes catalyzed by DPA and DSA (Scheme 2).

Our initial experiments were carried out using 4-nitrobenzaldehyde, as an electrophile, and β-naphthol in the presence of 10 mol % DPA in different solvents such as water, acetonitrile, and toluene. According to the results in Table 1, in water and acetonitrile (entries 1 and 2, Table1), the reaction did not proceed at all even in reflux conditions. However, in toluene with higher boiling point, the desired product was produced only in 43%. The best isolated yield of the corresponding benzoxanthene was obtained in the mixture of 4-nitrobenzaldehyde, β-naphthol and DPA in 1:2:0.1 molar ratios in solvent-free condition for 60 min at 85°C (entry 4, Table1). A lower amount of DPA, 5 mol %, led to a similar yield in longer reaction time but a higher amount of the catalyst, 20 mol %, did not show any improvement in the yield or the reaction time (entries 5 and 6, Table1). The same reaction did not produce 14-(4-

Scheme 1



dodecylphosphonic acid



dodecylsulfamic acid

nitrophenyl)-14-H-dibenzoxanthene at room temperature (entry 8, Table1). Increasing the temperature to 125°C lowered the yield of reaction to 50% (entry 9, Table1). In the absence of catalyst, DPA, the product was formed only in 25% yield at 85–90°C after 6 h (entry 7, Table1).

To consider the effect which the length of alkyl group has on the reactivity of phosphonic acid group, we prepared four alkyl phosphonic acids [22] with different lengths and then their effects were studied on the synthesis of 14-phenyl-14-H-dibenzo[a,j]xanthene as a model compound in solvent-free conditions at 85°C (Scheme 3). The results showed that dodecyl alkyl chain produced the most pronounced effect on the reactivity of phosphonic acid (entry 4, Table 2).

Then, to assess the generality of this method, various aromatic aldehydes were reacted with β-naphthol under the optimized reaction conditions (Scheme 4, Table 3, method A).

According to the results obtained, which have been shown in Table 3, aromatic aldehydes bearing electron-donating and electron-withdrawing groups were reacted in moderate to high yields although *para*- and *meta*-substituted aromatic aldehydes were transformed to the desired products in higher yields than those with *ortho*-substitutions.

A literature survey showed that there are few reports about the preparation of benzoxanthenes from the reaction between aliphatic aldehydes and β-naphthol [9,11,15,19,23]. Therefore, we decided to use aliphatic aldehydes for this purpose. Phenylacetaldehyde and 3-phenylpropanal were chosen as aliphatic aldehydes and investigated under the same conditions for the preparation of the related xanthenes. The reactions were occurred successfully but the yields of xanthenes were low in comparison to the aromatic aldehydes (entries 14 and 15, Table 3).

When we looked closely at the experiments during the progress of reactions, we observed that some of the aldehydes evaporated or sublimated from the reaction mixtures and, therefore, were not in the desirable position to collide with β-naphthol and DPA to initiate the reaction. For overcoming this problem, we used 1.2:2:0.1 (instead of 1:2:0.1) molar ratio of aldehyde, β-naphthol, and DPA, respectively. This enhancement in the amount of the aldehyde raised the yield of reaction for 4-nitrobenzaldehyde and 3-phenylpropionaldehyde from 80% and 60% to 95% and 93%, respectively (entries 16 and 17, Table 3).

In summary, this study showed that DPA is efficient catalyst for the simple, easy, and rapid preparation of a wide range of substituted benzoxanthenes under solvent-free conditions. Furthermore, this method offers several advantages such as high conversion, short reaction times, clean reaction profiles, simple experimental and work up procedures. Finally, recovery of DPA by centrifuge method is another advantage for this method.

Mechanistically, the reaction proceeds probably via condensation of 1 mol of aldehyde with 2 mol of β-naphthol and then by intramolecular elimination of water from two hydroxyl groups to form the corresponding dibenzoxanthene as has been suggested earlier [26] (Scheme 5).

Regarding to the effect of alkyl length on the reactivity of phosphonic acid group and also ability of sulfamic acid in the synthesis of xanthenes [10], we decided to prepare DSA and examine its reactivity, as a catalyst, in the formation of benzoxanthenes. Therefore, DSA was

Scheme 2

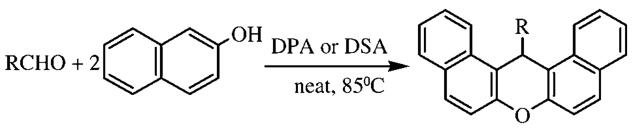


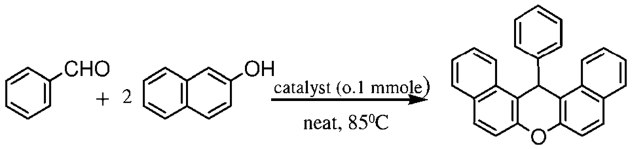
Table 1

Investigated conditions for preparation of 14-(4-nitrophenyl)-14-H-dibenzo[a, j]xanthene using of DPA as catalyst.

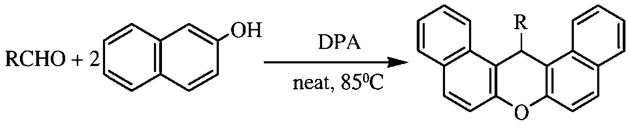
Entry	Solvent	Catalyst	Temp.	Time (h)	Yield[a,b] (%)
1	H_2O	DPA (10 mol %)	Reflux	6	N.R
2	CH_3CN	DPA (10 mol %)	Reflux	6	N.R
3	Toluene	DPA (10 mol %)	Reflux	6	43
4	Solvent-free	DPA (10 mol %)	85°C	1	80
5	Solvent-free	DPA (20 mol %)	85°C	1	82
6	Solvent-free	DPA (5 mol %)	85°C	6	80
7	Solvent-free	–	85°C	6	25
8	Solvent-free	DPA (10 mol %)	R.T.	6	N.R.
9	Solvent-free	DPA (10 mol %)	125°C	1	50

[a]Isolated yields.
[b]The equivalent ratio of aldehyde/ b-naphthol is 1/2 in all experiments.

Scheme 3



Scheme 4



prepared according to the literature procedure [27] and then the reaction of 4-nitrobenzaldehyde with β-naphthol was chosen as a model for finding the best conditions. The different conditions, as shown in Table 4, were applied and the solvent free condition at 85°C with 1:2:0.1 molar ratios among 4-nitrobenzaldehyde, β-naphthol, and DSA (entry 4, Table 4) were selected as the best condition for the rest of study.

With optimized condition in hand, we studied the behavior of a variety of aldehydes including aromatic aldehydes with electron-donating and electron-withdrawing substitutions in meta, *ortho* and *para* positions and also aliphatic aldehydes in the presence of DSA under the same conditions. The results obtained from this study were shown in Table 3, as method B.

Comparison between efficiency of the both catalysts, DPA and DSA, indicated that DSA catalyzes generally more effective the reaction between aromatic and aliphatic aldehydes with β-naphthol. Therefore, the corresponding benzoxanthenes are formed in higher yields and shorter times, except 2-methoxybenzaldehyde, which does not produce the corresponding product. This can be probably related partly to the more acidity strength of DSA ($pK_a < 3$) than DPA ($pK_a1 = 3$ and $pK_a2 = 8$) [28].

CONCLUSIONS

These studies showed that DPA and DSA, as nonvolatile heterogenous Brønsted acid catalysts, are efficient catalysts for one-pot preparation of a wide range of 14-substituted dibenzoxanthenes under solvent-free conditions. Furthermore, these methods offer several additional advantages such as recovery of DPA and DSA, high conversion, short reaction times, clean reaction profiles, simple experimental and work up procedures.

A comparison among these catalysts, DPA and DSA, and the other Brønsted acid catalysts or reagents reported in the literature for the synthesis of 14-(4-nitrophenyl)-14H-dibenzo[a,j]xanthenes *via* the reaction of 4-nitrobenzaldehye with β-naphthol, Scheme 6, shows clearly that DPA and DSA promote the reaction more efficiently than the most of the other catalysts , which are presented in Table 5, and gives comparable results with the some of them such as silica sulfuric acid.

Table 2

Effect of alkyl length on the reactivity of phosphonic acid group.

Entry	Catalyst[a]	Time (min)	Yield[b] (%)
1	$C_4H_9PO(OH)_2$	75	25
2	$C_8H_{17}PO(OH)_2$	75	65
3	$C_{10}H_{21}PO(OH)_2$	30	65
4	$C_{12}H_{25}PO(OH)_2$	30	70

[a]These acids are commercially available and have been prepared according known procedure [22].
[b]Isolated yields.

EXPERIMENTAL

NMR spectra were recorded on a Bruker Avance DPX-250 (^{1}H-NMR 250 MHz and ^{13}C-NMR 63 MHz). IR spectra were obtained using a Shimadzu FT-IR 8300 spectrophotometer. Mass spectra were determined on a Shimadzu GCMS-QP 1000 EX instruments at 70 or 20 ev and CHN data were reported on a Flash EA instrument. Melting points were determined in open capillary tubes in a Büchi-545 circulating oil melting point apparatus. Material purchased from Fluka, Aldrich and Merck Chemical Companies. DPA and DSA was prepared by the literature methods [28] (also, these acids are commercially available).

Typical procedure for the preparation of benzoxanthenes in the presence of DPA (method A). A mixture of the aldehyde (1 mmol), β-naphthol (2 mmol), and DPA (0.1 mmol) was stirred at 85°C for the appropriate time (Table 3). After completion of the reaction, which was determined by TLC monitoring, and cooling of the mixture to room temperature, H_2O (10 mL) and CH_2Cl_2 (10 mL) were added to the reaction mixture and then the mixture was centrifuged. The catalyst, which was formed as a solid phase between aqueous and organic layer, was recovered by filtration. Then, organic phase was separated from the filtrate and dried over anhydrous $CaCl_2$. The crude product was obtained by evaporation of CH_2Cl_2 and then purified by recrystalization from ethanol.

Typical procedure for the preparation of benzoxanthenes in the presence of DSA (method B). A mixture of the aldehyde (1 mmol), β-naphthol (2 mmol), and DSA (0.05 mmol) was stirred at 85°C for the appropriate time (Table 3). Completion of the reaction was monitored by TLC. After cooling the reaction mixture to room temperature, acetone (10 mL) was added and the catalyst was precipitated. The catalyst was recovered from the mixture by filtration. Evaporation of acetone under reduced pressure and then recrystalization of the crude product from ethanol afforded the pure product.

All the products, except entries 7, 12, 14 and 15 in Table 3, were characterized by comparison of their IR, ^{1}H-NMR, ^{13}C-NMR, mass spectral, and physical data with the authentic

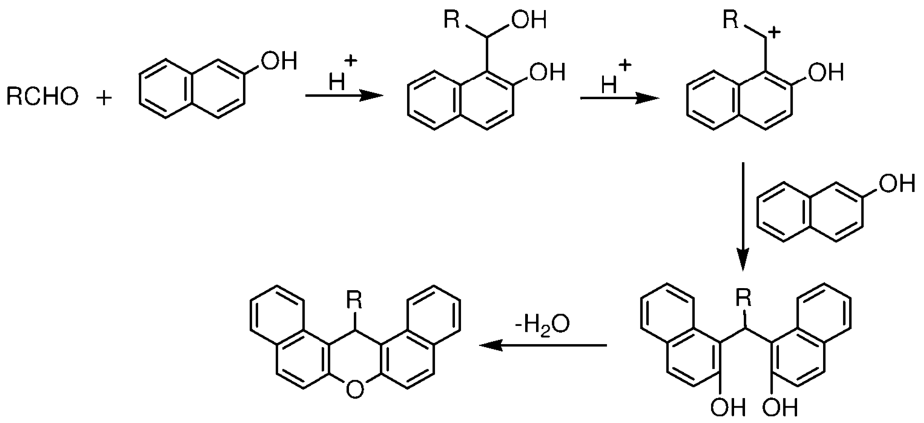
Table 3

Synthesis of 14-alkyl- or aryl- 14-*H*-dibenzo[a, j]xanthenes in the presence of a catalytic amount of DPA and DSA.

Entry	R-	Time (min) Method A/ Method B	Yield (%)[a] Method A/ Method B	Mp (°C) Found	Mp (°C) Reported
1	C_6H_5	30/31	70/66[c]	189	183 [8]
2	4-Cl—C_6H_4	36/19	70/86[c]	291	287 [9]
3	2-Cl—C_6H_4	24/16	60/63[c]	215	215 [9]
4	2-O_2N—C_6H_4	83/35	71/80[c]	213	214 [24]
5	4-O_2N—C_6H_4	60/15	80/90[c]	315	312 [8]
6	3-O_2N—C_6H_4	39/31	80/82[c]	216	213 [8]
7	2-Br—C_6H_4	53/54	60/81[c]	204–206	—
8	4-Br—C_6H_4	45/45	96/97	296	297 [11]
9	4-CH_3O—C_6H_4	77/99	65/77[c]	205	204 [9]
10	2-CH_3O—C_6H_4	65/50	74/–[b]	256	258 [10]
11	4-$(CH_3)_2CH$—C_6H_4	38/14	68/80[c]	153	151–153 [11]
12	3-CH_3—C_6H_4	67/73	74/57[c]	198.5	—
13	4-HO—C_6H_4	84/90	84/52[c]	140	140 [25]
14	$PhCH_2$	20/105	50/74[c]	159	—
15	$PhCH_2CH_2$	50/32	60/83[c]	164	—
16	4-O_2N—C_6H_4	60/–	95[d]/–	315	312 [8]
17	$PhCH_2CH_2$	60/–	93[d]/–	164	—

[a]Isolated yields.
[b]The reaction mixture was polymerized.
[c]The equivalent ratio of aldehyde/ β-naphthol is 1/2.
[d]The equivalent ratio of aldehyde/ β-naphthol is 1.2/2.

Scheme 5



samples. Also, the analytical data of four new representative dibenzoxanthenes are given below.

14-(2-Bromophenyl)-14H-dibenzo[a,j]xanthene (entry 7, Table 3). White solid, m.p.204–206°C, IR (KBr, cm^{-1}): 3055, 1593, 1515, 1458, 1249, 964, 803, 733. ^{1}H-NMR (250MHz, $CDCl_3$): δ = 6.72 (s, 1H), 6.83 (m, 1H), 6.99 (m, 1H), 7.47 (m, 6H), 7.63 (m, 2H), 7.81 (t, *J* = 8.46 Hz, 4H), 8.90 (d, *J* = 8.5 Hz, 2H). ^{13}C-NMR (250MHz, $CDCl_3$): δ = 37.11, 117.93, 118.13, 120.72, 123.85, 124.21, 125.88, 126.55, 127.91, 128.28, 128.40, 128.68, 128.85, 130.68, 131.54, 131.83, 132.87, 145.20, 148.68. EIMS: *m/z* (%): 437 (M^+, 5.5), 436 (11.5), 282 (28.8), 281 (100.0), 86 (30.9), 57 (15.2). Anal. Calcd for $C_{27}H_{17}BrO$: C, 74.14; H, 3.89%. Found: C, 73.92; H, 3.80%.

Table 4

Investigated conditions for preparation of 14-(4-nitrophenyl)-14-*H*-dibenzo[a, j]xanthenes using of DSA as catalyst.

Entry	Solvent	Catalyst	Temp.	Time (h)	Yield[a] (%)
1	H_2O	DSA (10 mol %)	Reflux	6	N.R
2	CH_3CN	DSA (10 mol %)	Reflux	6	N.R
3	Toluene	DSA (10 mol %)	Reflux	3	60
4	Solvent-free	DSA (10 mol %)	85°C	0.25	90
5	Solvent-free	DPA (20 mol %)	85°C	1	85
6	Solvent-free	DPA (5 mol %)	85°C	3	55
8	Solvent-free	DSA (10 mol %)	R.T.	6	N.R.
9	Solvent-free	DSA (10 mol %)	100°C	2	80

[a]Isolated yields.

Scheme 6

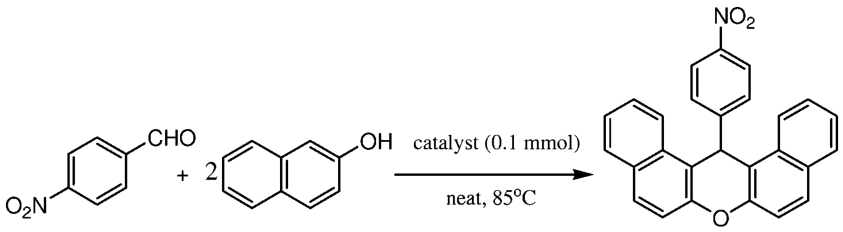


Table 5

Comparison among the efficiencies of various Brønsted acid catalysts used in the synthesis of 14-(4-nitrophenyl)-14-*H*-dibenzo[a, j]xanthene.

Catalyst	Solvent	Temp. (°C)	Time (h)	Yield (%)
$NH_4H_2PO_4/SiO_2$ [29]	H_2O	40	0.70	94
Montmorilonite K10 [14]	Solvent-free	120	2	80
$NaHSO_4$ [30]	Solvent-free	90	0.70	88
$NaHSO_4$ [21]	1,2-Dichloroethane	82—84	3	91
Amberlyst-15 [21]	1,2-Dichloroethane	82—84	3	90
Dowex-50w [31]	Solvent-free	100	2	84
Heteropoly acid [17]	Solvent-free	100	1	94
Silica sulfuric acid [32]	Solvent-free	80	1	94
Indion-130 [15]	Solvent-free	110	0.20	92
H_2SO_4 [8]	AcOH	80	73	60
Sulfamic acid [10]	Solvent-free	125	11	94
p-TSA [9]	Solvent-free	125	2.5	90
Cellulose sulfuric acid [18]	Solvent-free	110—115	2.0	90
$[MIMPS]HSO_4$ [19]	Solvent-free	100	0.1	94
DPA	Solvent-free	85	1	95
DSA	Solvent-free	85	0.25	90

14-(3-Methylphenyl)-14H-dibenzo[a,j]xanthene (entry 12, Table 3). White solid, m.p.198.5°C, IR (KBr, cm^{-1}): 3036, 2912, 1593, 1512, 1431, 1249, 806, 748. ^{1}H-NMR (250MHz, $CDCl_3$): δ = 2.19 (s, 3H, —C$\underline{H}_3$), 6.47 (s, 1H), 6.84 (m, 1H), 7.09 (t, *J* = 7.6 Hz, 1H), 7.53 (m, 7H), 7.82 (m, 5H), 8.45 (d, *J* = 8.5 Hz, 2H). ^{13}C-NMR (63 MHz, $CDCl_3$): δ = 21.50, 37.98, 117.24, 117.84, 122.54, 124.00, 125.19, 126.53, 127.09, 127.94, 128.56, 128.78, 130.11, 130.85, 131.27, 137.92, 144.69, 148.47. EIMS: *m/z* (%): 372 (M^+, 15.3), 282 (100.0), 281 (90.4), 253.

14-Benzyl-14H-dibenzo[a,j]xanthene (entry 14, Table 3). Yellow solid, m.p.159°C, IR (KBr, cm^{-1}): 3028, 2931, 1589, 1454, 1246, 856, 741. ^{1}H-NMR (250MHz, $CDCl_3$): δ = 3.29 (d, *J* = 3.64 Hz, 2H, —C$\underline{H}_2$), 5.80 (s, 1H), 6.14 (m, 2H), 7.03–7.91 (m 13H), 8.26 (d, *J* = 8.23 Hz, 2H). ^{13}C-NMR (63MHz, $CDCl_3$): δ = 32.99, 41.29, 115.10, 117.20, 121.98, 123.82, 125.19, 126.47, 126.98, 128.12, 128.66, 129.54, 130.62, 131.10, 137.33, 149.87. EIMS: *m/z* (%): 372 (M^+, 0.1), 371 (0.2), 348 (0.6), 282 (26.8), 281 (100.0), 152 (12.2), 57 (2.0). Anal. Calcd for $C_{28}H_{20}O$: C, 90.32; H, 5.37%. Found: C, 89.95, H, 5.41%.

14-(2-Phenylethyl)-14H-dibenzo[a,j]xanthene (entry 15, Table 3). White solid, m.p.164°C, IR (KBr, cm^{-1}): 3039, 2921, 1591, 1450, 1238, 800. ^{1}H-NMR (250MHz, $CDCl_3$): δ = 2.41 (m, 4H), 5.68 (s, 1H), 6.28 (m, 2H), 7.07 (m, 3H), 7.48 (m, 4H), 7.63 (m, 2H), 7.83 (m, 4H), 8.29 (d, *J* = 8.45 Hz, 2H). ^{13}C-NMR (63 MHz, $CDCl_3$): δ = 30.94, 37.22, 115.88, 117.40, 122.16, 123.97, 125.28, 126.51, 127.78, 127.90, 128.22, 128.68, 130.86, 131.15, 141.61, 149.80. EIMS: *m/z* (%): 386 (M^+, 1.2), 282 (30.2), 281 (100.0), 252 (10.4), 57 (23.5). Anal. Calcd for $C_{29}H_{22}O$: C, 90.15; H, 5.69%. Found: C, 89.99; H, 5.69%.

14-(4-Nitrophenyl)-14H-dibenzo[a,j]xanthene (entry 16, Table 3). Light-brown solid, m.p.315°C (Lit. 312°C).[8] IR (KBr, cm^{-1}): 3066, 1589, 1512, 1332, 1238, 1138, 806, 741. 1H-NMR (250MHz, $CDCl_3$): δ = 6.60 (s, 1H), 7.52 (m, 8H), 7.84 (m, 4H), 7.87 (d, *J* =8.8 Hz, 2H), 8.30 (d, *J* =8.4 Hz, 2H). ^{13}C-NMR (63 MHz, $CDCl_3$): δ = 37.78, 111.16, 117.34, 117.84, 121.80, 123.63, 124.35, 126.95, 128.72, 128.83, 129.35, 130.84, 148.50, 149.48, 151.73. EIMS: *m/z* (%): 403 (M^+, 8.9), 374 (7.7), 282 (70.7), 281 (100.0), 253 (11.7), 122 (3.7), 111 (15.2), 83 (33.6), 57 (91.7). Anal. calcd. for $C_{27}H_{17}NO_3$: C, 80.40; H, 4.22; N, 3.47%. found: C, 80.16; H, 4.30; N, 3.24%.

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