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ORIGINAL PAPER

Ammonium acetate as a catalyst and/or reactant in the reaction of dimedone, aromatic aldehyde, and malononitrile: synthesis of tetrahydrobenzo[*b*]pyrans and hexahydroquinolines

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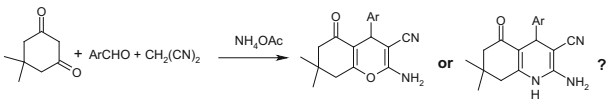
Abstract The reaction of aromatic aldehydes, dimedone, and malononitrile in the presence of ammonium acetate has afforded tetrahydrobenzo[*b*]pyrans instead of polyhydroquinolines under both grinding and reflux conditions; thus ammonium acetate acts as a catalyst for this transformation instead of a reactant. Polyhydroquinoline derivatives were also synthesized via a one-pot condensation of aromatic aldehydes, malononitrile and an enaminone, which was prepared by the reaction of dimedone and ammonium acetate, in refluxing ethanol in high yields.

Graphical abstract



Keywords Multi-component reactions · Tetrahydrobenzo[*b*]pyrans · Polyhydroquinolines · Grinding · Ammonium acetate

Introduction

Over the past decades, multi-component reactions (MCRs) have proved to be very powerful and efficient bond-forming tools in organic combinatorial and medicinal chemistry in the context of green chemistry [1–7]. Many important heterocycle syntheses are multicomponent reactions. Recently, the syntheses of 4*H*-chromene and quinoline derivatives have attracted great interest due to biological and pharmacological activities. The 4*H*-chromene derivatives show various pharmacological properties such as spasmolytic, diuretic, anticoagulant, anticancer, and antianaphylactic activities [8–12]. Furthermore these compounds can be employed as pigments, and also they constitute the structural unit of a series of natural products [13, 14].

Quinoline derivatives are a class of important compounds, which are widely recognized to have antitumor, antibacterial, anticancer, and antimalarial activities [15–18]. Tetrahydrobenzo[*b*]pyrans (4*H*-chromene derivatives) are structurally similar to biologically active polyhydroquinolines. Due to this similarity, in ammonium acetate-mediated reaction of aldehydes and active methylene compounds competitive synthesis of tetrahydrobenzo[*b*]pyrans instead of polyhydroquinolines could be achieved. Multi-component synthesis of polyhydroquinoline derivatives **I** from aldehydes, dimedone, ethyl acetoacetate, and ammonium acetate have been reported using TMSCl [19], ionic liquids [20, 21], polymers [22, 23] and $Yb(OTf)_3$ [24, 25], promotion of microwave [26], CAN [27], solar thermal energy [28], potassium dodecatungsto cobaltate trihydrate [29], Baker's yeast [30], organocatalyst [31, 32], $K_7[PW_{11}CoO_{40}]$ [33], montmorrilonite K10 [34], sulfamic acid [35], and FeF_3 [36]. Synthesis of polyhydroquinolines **I** from aldehydes, dimedone, ethyl acetoacetate, and ammonium acetate has also been reported under aqueous media without any catalyst [37].

Conflicting results have been obtained by replacing ethyl acetoacetate with malononitrile. Synthesis of 2-amino-3-cyanopolyhydroquinolines **II** have been reported via a multi-component reaction of aldehydes,

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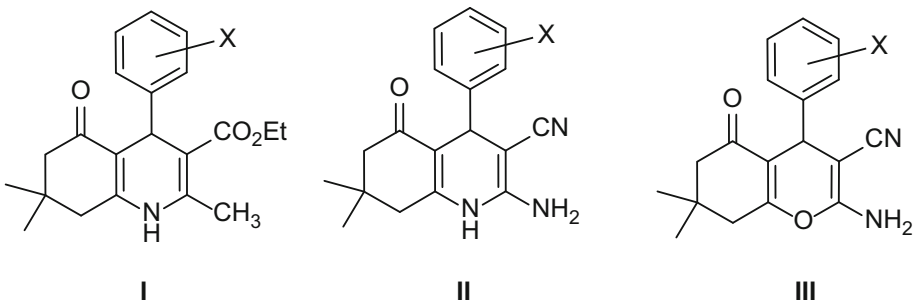


Fig. 1 The general structures of polyhydroquinolines **I**, **II**, and tetrahydrobenzo[*b*]pyrans **III**

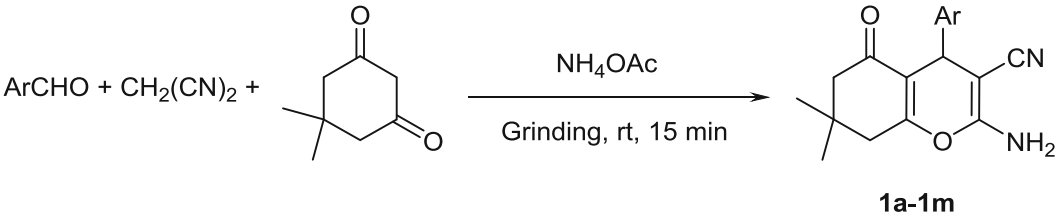
dimedone, malononitrile, and ammonium acetate in refluxing acetic acid [38], under solvent free conditions and microwave irradiation [39], and under grinding [40]. Although Kumar et al. [40] have reported the multi-component reaction of aldehydes, dimedone, malononitrile, and ammonium acetate for the synthesis of polyhydroquinolines **II** under grinding, Undale et al. [41] have obtained the corresponding tetrahydrobenzo[*b*]pyrans **III** instead of polyhydroquinolines **II** from the reaction of aldehydes, dimedone, malononitrile, and ammonium acetate in ethanol (Fig. 1). Also Patil et al. [42] have reported one-pot four-component sequential synthesis of hexahydroquinoline derivatives in aqueous media via enaminone intermediates. In continuation of our interest in multi-component reactions [43–46] and to combinatorial library synthesis of polyhydroquinoline derivatives **II**, we performed the multi-component reaction of aromatic aldehydes, dimedone, malononitrile, and ammonium acetate at room temperature under grinding, but unlike the Kumar's report, we obtained tetrahydrobenzo[*b*]pyran derivatives **III** instead of polyhydroquinolines **II**. We also performed the sequential reaction according the Patil et al. procedure [42], but tetrahydrobenzo[*b*]pyran derivatives **III** were obtained. Thus herein we wish to clarify the role of ammonium acetate as a catalyst or a reactant and to report the green synthesis of polyhydroquinoline derivatives **II** by a sequential two-stage reaction of dimedone, ammonium acetate, malononitrile, and arylaldehydes.

Results and discussion

We initially examined the one-pot reaction of benzaldehyde (1 equiv.), dimedone (1 equiv.), malononitrile (1 equiv.), and ammonium acetate (1.5 equiv.) under solvent-free conditions at room temperature under grinding. All reactants were taken in a mortar, mixed thoroughly, and ground well at room temperature for 15 min. However, after work-up and purification, the ^{1}H NMR spectrum of the obtained product revealed the formation of corresponding tetrahydrobenzo[*b*]pyran derivative **1a** instead of polyhydroquinoline derivative **2a** despite using an excess amount of ammonium acetate. The reaction conditions were then applied to a range of aldehyde substrates. The results with different aldehydes are depicted in Table 1. Aldehydes with both electron withdrawing and electron donating substituents were well tolerated. It is evident that ammonium acetate acts as a catalyst instead of a reactant for the present transformation. Then we performed the multi-component reaction of aldehydes, dimedone, malononitrile, and ammonium acetate using ethanol as a solvent both at room temperature and under reflux conditions, and the same results were obtained. The reaction is proposed to proceed through a mechanism shown in Scheme 1. We also examined three-component reaction of benzaldehyde, dimedone, and malononitrile, in the absence of ammonium acetate, under grinding at room temperature. No reaction was occurred. Furthermore, in using 20 mol% of ammonium acetate, tetrahydrobenzo[*b*]pyran derivative **1a** was obtained in 30 % yield and most of the reactants remained unreacted.

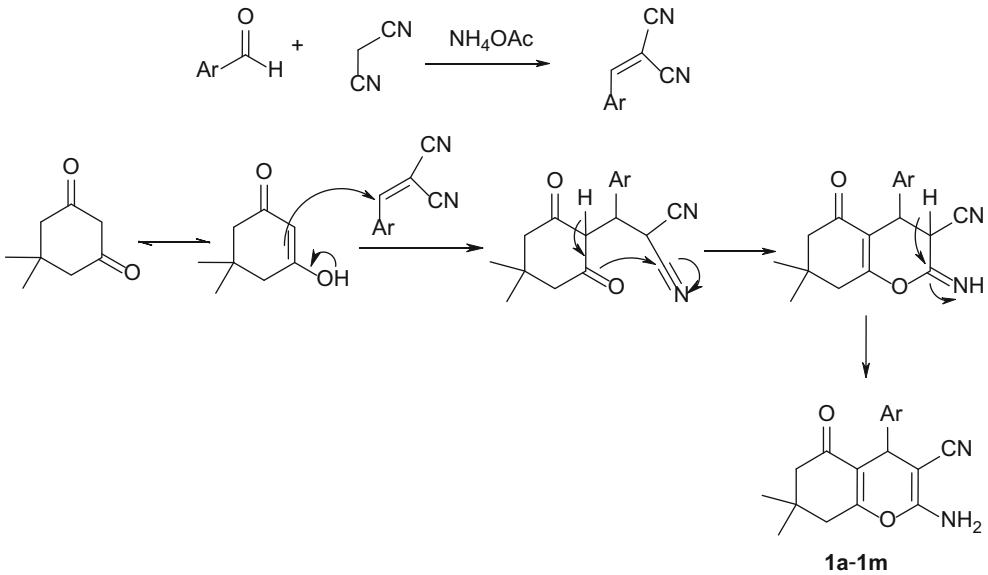
Therefore, the synthesis of polyhydroquinoline derivatives **2a**–**g** was performed using a two-stage process. Treatment of dimedone (2 mmol) and ammonium acetate (3 mmol) in dry toluene in the presence of 4 Å molecular sieves under reflux for 2 h furnished 3-amino-5,5-dimethyl-2-cyclohexen-1-one (**3**) in 90 % yield. Then the mixture of 3-amino-5,5-dimethyl-2-cyclohexen-1-one (1 mmol), benzaldehyde (1 mmol), and malononitrile (1 mmol) in 5 cm^3 ethanol was refluxed until completion of reaction as indicated by TLC (22 h). After recrystallization of the precipitated solid from ethanol, 2-amino-7,7-dimethyl-5-oxo-4-phenyl-1,4,5,6,7,8-hexahydroquinoline-3-carbonitrile (**2a**) was obtained in 75 % yield. The reaction was also performed in the presence of ammonium acetate and imidazole as catalysts. Addition of ammonium acetate or imidazole caused the acceleration of reaction and

Table 1 Ammonium acetate-mediated reaction of aromatic aldehydes, dimedone, and malononitrile at room temperature under grinding



Entry	Ar	Product	Yield/%	M.p./°C
1	C_6H_5	**1a**	90	228–230 (228–230 [47])
2	2-$O_2NC_6H_4$	**1b**	78	230–232 (233–234 [8])
3	3-$O_2NC_6H_4$	**1c**	92	211–212 (210–211 [47])
4	4-$O_2NC_6H_4$	**1d**	94	179–180 (176–178 [47])
5	2-$MeOC_6H_4$	**1e**	81	199–201
6	3-$MeOC_6H_4$	**1f**	92	199–201 (207–209 [49])
7	2-ClC_6H_4	**1g**	94	211–213 (208–210 [47])
8	3-ClC_6H_4	**1h**	80	222–224 (222–224 [48])
9	2-BrC_6H_4	**1i**	98	154–156
10	4-BrC_6H_4	**1j**	85	216–218 (206–208 [49])
11	2-MeC_6H_4	**1k**	85	205–207
12	3-MeC_6H_4	**1l**	91	202–204
13	4-MeC_6H_4	**1m**	88	218–220 (218–220 [48])

Scheme 1



the reduction of reaction time to 14 h and 45 min, respectively. So, the optimized conditions (refluxing in ethanol in the presence of 20 mol% imidazole) were then applied to a series of aldehyde substrates (Scheme 2; Table 2).

Conclusion

Based on the obtained experimental results, in the multi-component reaction of aromatic aldehydes, dimedone, malononitrile, and ammonium acetate under both grinding

Scheme 2

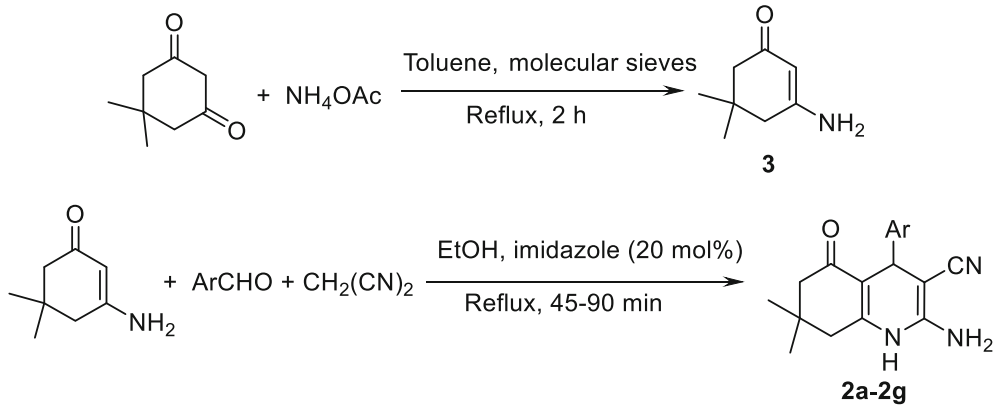


Table 2 The two-stage synthesis of polyhydroquinoline derivatives **2a**–**2g** in refluxing ethanol in the presence of catalytic amount of imidazole

Entry	Ar	Product	Time/min	Yield/%	M.p./°C
1	C_6H_5	**2a**	45	75	288–290 (280–281 [39])
2	3-$O_2NC_6H_4$	**2b**	45	87	281–282 (282–283 [39])
3	4-$MeOC_6H_4$	**2c**	90	89	288–289 (288–289 [39])
4	2-BrC_6H_4	**2d**	60	70	275–276
5	4-BrC_6H_4	**2e**	90	90	292–294 (295–296 [39])
6	2-MeC_6H_4	**2f**	75	80	276–279
7	4-MeC_6H_4	**2g**	65	88	>300 (>300 [39])

and reflux conditions, ammonium acetate acts as an efficient catalyst instead of reactant and led to the formation of tetrahydrobenzo[*b*]pyrans instead of polyhydroquinolines.

Experimental

General procedure for the synthesis of tetrahydrobenzo[b]pyrans 1a–1m

A mixture of 1.1 cm^3 benzaldehyde (10 mmol), 0.66 g malononitrile (10 mmol), 1.4 g dimedone (10 mmol), and 1.15 g ammonium acetate (15 mmol) was thoroughly mixed in a mortar by grinding until the completion of reaction as indicated by TLC (15 min). The precipitate was filtered, washed with water, and recrystallized from ethanol gave compound **1a** as white crystals (2.21 g, 90 %).

2-Amino-5,6,7,8-tetrahydro-4-(2-methoxyphenyl)-7,7-dimethyl-5-oxo-4H-chromene-3-carbonitrile
(**1e**, $C_{19}H_{20}N_2O_3$)
M.p.: 199–201 °C; IR (KBr): $\bar{\nu}$ = 3395 (m), 3330 (m), 3220 (m), 2964 (m), 2189 (s), 1685 (s), 1654 (s), 1606 (s), 1494 (s), 1371 (s), 1251 (s), 1212 (s), 1037 (s) cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.07 (s, 3H, CH_3), 1.14 (s, 3H, CH_3), 2.18–2.29 (m, 2H, CH_2), 2.46 (s, 2H, CH_2), 3.85 (s, 3H, OCH_3), 4.48 (s, 2H, NH_2), 4.73 (s, 1H, C(4)-H), 6.87–6.91 (m, 2H, ArH), 7.11 (d, J = 8.0 Hz, 1H, ArH), 7.20 (t, J = 8.0 Hz, 1H, ArH) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 26.67, 29.35, 32.52, 32.61, 50.67, 55.58, 61.81, 111.25, 112.45, 119.10, 120.45, 128.26, 128.62, 131.63, 157.17, 157.53, 157.74, 194.47 ppm.

2-Amino-4-(2-bromophenyl)-5,6,7,8-tetrahydro-7,7-dimethyl-5-oxo-4H-chromene-3-carbonitrile
(**1i**, $C_{18}H_{17}BrN_2O_2$)
M.p.: 154–156 °C; IR (KBr): $\bar{\nu}$ = 3465 (m), 3327 (m), 3196 (m), 2958 (m), 2872 (m), 2197 (s), 1676 (s), 1603 (s), 1366 (s), 1213 (s), 1038 (s) cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.09 (s, 3H, CH_3), 1.13 (s, 3H, CH_3), 2.18-2.28 (m, 2H, CH_2), 2.48 (s, 2H, CH_2), 4.70 (s, 2H, NH_2), 4.92 (s, 1H, C(4)-H), 7.07 (dt, J = 8.0, 2.0 Hz, 1H, ArH), 7.18 (d, J = 8.0 Hz, 1H, ArH), 7.26 (dt, J = 8.0, 2.0 Hz, 1H, ArH), 7.54 (d, J = 8.0 Hz, 1H, ArH) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 26.92, 29.56, 32.42, 36.60, 50.67, 60.27, 111.92, 117.38, 122.45, 126.96, 127.62, 128.73, 132.03, 141.89, 156.53, 156.74, 194.67 ppm.

2-Amino-5,6,7,8-tetrahydro-7,7-dimethyl-4-(2-methylphenyl)-5-oxo-4H-chromene-3-carbonitrile
(**1k**, $C_{19}H_{20}N_2O_2$)
M.p.: 205–207 °C; IR (KBr): $\bar{\nu}$ = 3374 (m), 3142 (m), 2958 (m), 2187 (s), 1684 (s), 1668 (s), 1608 (m), 1364 (s), 1216 (s), 1039 (s) $cm^{-1;}$ ^1H NMR (400 MHz, $CDCl_3$): δ = 1.08 (s, 3H, CH_3), 1.14 (s, 3H, CH_3), 2.20–2.25 (AB quartet, J = 20.0 Hz, 2H, CH_2), 2.49 (s, 2H, CH_2), 2.60 (s, 3H, CH_3), 4.57 (s, 2H, NH_2), 4.70 (s, 1H, C(4)-H), 6.97-7.15 (m, 4H, ArH) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 19.27, 26.84, 29.46, 32.54, 32.70, 50.76, 61.68, 112.52, 119.12, 126.50, 126.86, 127.85, 130.37, 135.32, 142.48, 156.62, 157.44, 194.74 ppm.

2-Amino-5,6,7,8-tetrahydro-7,7-dimethyl-4-(3-methylphenyl)-5-oxo-4H-chromene-3-carbonitrile
(**1l**, $C_{19}H_{20}N_2O_2$)
M.p.: 202–204 °C; IR (KBr): $\bar{\nu}$ = 3349 (m), 3177 (s), 2964 (m), 2191 (s), 1683 (s), 1656 (s), 1604 (s), 1371 (s), 1214 (s), 1036 (s), cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 1.08 (s, 3H, CH_3), 1.14 (s, 3H, CH_3), 2.29 (s, 2H, CH_2), 2.34 (s, 3H, CH_3), 2.48 (s, 2H, CH_2), 4.38 (s, 1H, C(4)-H), 4.55 (s, 2H, NH_2), 7.02-7.04 (m, 3H, ArH), 7.17–7.21 (m, 1H, ArH) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 21.45, 26.74, 29.61, 32.63, 36.33, 49.96, 62.52, 113.12, 118.82, 124.62, 127.46, 128.25, 128.37, 138.05, 143.64, 156.62, 157.05, 194.67 ppm.

3-Amino-5,5-dimethyl-2-cyclohexen-1-one (**3**)
A mixture of 0.28 g dimedone (2 mmol) and 0.23 g ammonium acetate (3 mmol) in 5 cm^3 dry toluene in the presence of 4 Å molecular sieves was refluxed for 2 h. After completion of the reaction as indicated by TLC, the reaction mixture was cooled to room temperature and the precipitate was filtered and then purified by recrystallization from dry toluene. 3-Amino-5,5-dimethyl-2-cyclohexen-1-one (**3**) was obtained as needles (0.25 g, 90 %). M.p.: 163–165 °C (Ref. [50] 163.5–164 °C).

General procedure for the synthesis of polyhydroquinolines 2a–2g

Method A A mixture of 150 mg 3-nitrobenzaldehyde (1 mmol), 70 mg malononitrile (1 mmol), and 140 mg 3-amino-5,5-dimethyl-2-cyclohexen-1-one (**3**, 1 mmol) in 5 cm^3 ethanol was refluxed until completion of the reaction as indicated by TLC (22 h). The reaction mixture was cooled to room temperature and the precipitate was filtered and then purified by recrystallization from ethanol. 2-Amino-7,7-dimethyl-5-oxo-4-(3-nitrophenyl)-1,4,5,6,7,8-hexahydroquinoline-3-carbonitrile (**2b**) was obtained as yellow crystals (246 mg, 76 %).

Method B The reaction of 150 mg 3-nitrobenzaldehyde (1 mmol), 70 mg malononitrile (1 mmol), 140 mg 3-amino-5,5-dimethyl-2-cyclohexen-1-one (**3**, 1 mmol), and 10 mg imidazole (0.2 mmol) in 5 cm^3 ethanol was performed as above method. The reaction was completed in 45 min and **2b** obtained as yellow crystals (290 mg, 87 %).

2-Amino-7,7-dimethyl-5-oxo-4-(2-bromophenyl)-1,4,5,6,7,8-hexahydroquinoline-3-carbonitrile
(**2d**, $C_{18}H_{18}BrN_3O$)
M.p.: 275–276 °C; IR (KBr): $\bar{\nu}$ = 3442 (m), 3324 (m), 3215 (m), 2955 (m), 2184 (s), 1662 (s), 1630 (s), 1496 (s), 1370 (s), 1270 (s) cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6): δ = 0.96 (s, 3H, CH_3), 1.03 (s, 3H, CH_3), 1.96 (d, J = 16 Hz, 1H, CH_2), 2.17 (d, J = 16 Hz, 1H, CH_2), 2.38, 2.50 (AB quartet, J = 14 Hz, 2H, CH_2), 4.86 (s, 1H, C(4)-H), 5.74 (s, 2H, NH_2), 7.07 (t, J = 8.0 Hz, 1H, ArH), 7.14 (d, J = 8.0 Hz, 1H, ArH), 7.28 (t, J = 8.0 Hz, 1H, ArH), 7.49 (d, J = 8.0 Hz, 1H, ArH), 8.93 (s, 1H, NH) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 26.89, 29.52, 33.02, 36.36, 50.43, 60.43, 112.57, 117.25, 122.45, 127.06, 127.67, 128.68, 132.13, 141.64, 149.53, 158.27, 194.07 ppm.

2-Amino-7,7-dimethyl-4-(2-methylphenyl)-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carbonitrile
(**2f**, $C_{19}H_{21}N_3O$)
M.p.: 276–279 °C; IR (KBr): $\bar{\nu}$ = 3441 (m), 2961 (m), 2180 (s), 1664 (s), 1620 (s), 1497 (s), 1368 (s), 1271 (s) $cm^{-1;}$ ^1H NMR (400 MHz, DMSO-d_6): δ = 0.93 (s, 3H, CH_3), 1.03 (s, 3H, CH_3), 1.95 (d, J = 16.0 Hz, 1H, CH_2), 2.16 (d, J = 16.0 Hz, 1H, CH_2), 2.30 (d, J = 16.8 Hz, 1H, CH_2), 2.41 (d, J = 16.8 Hz, 1H, CH_2), 2.47 (s, 3H, CH_3), 4.60 (s, 1H, C(4)-H), 5.68 (s, 2H, NH_2), 6.96–7.10 (m, 4H, ArH), 8.84 (s, 1H,NH) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 19.84, 26.76, 29.53, 32.54, 32.73, 50.82, 60.98, 112.57, 118.82, 126.59, 126.87, 127.63, 129.87, 135.21, 142.28, 149.89, 159.24, 194.45 ppm.

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