SYNTHESIS OF NEW 1, 2-DIPHENYL-4, 5-DIHYDRO-3H-3-BENZAZEPINES

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Abstract – 1,2-Diphenyl-4,5-dihydro-3H-3-benzazepine derivatives (2a-d) were synthesized via cyclization reaction of N-[2-(2-iodophenyl)ethyl]-α-phenylphenacylamines (5a-c) and (5e) with n-C4H9Li, followed by dehydration of the cyclization products, 1,2-diphenyl-1-hydroxy-3-methyl-2,3,4,5-tetrahydro-1H-3-benzazepines (4a-d) with trifluoromethanesulfonic acid.

INTRODUCTION
Tamoxifen is a well established estrogen antagonist and one of the most used anti-breast cancer drug.1,2 This triarylethylene compound is today the drug of choice for palliative therapy of advanced breast cancer.3 However, side effects including endometrial carcinoma are also sometimes observed as a major adverse consequence of drug treatment.4

In order to overcome these defects, in recent years, much attention has been paid to the design of novel alternate scaffolds for estrogen antagonists such as 4,5-diphenyl-2,3-dihydro-1-benzoxepins,5 3,4-diphenyl-quinolines and isoquinolines,6 1,2-diphenyl-1,2,3,4-tetrahydroisoquinolines,7 2-phenyl-1-phenyloxynaphthalenes,8 and 2,3-diphenylindenes.9

We have reported the synthesis and biological evaluation of 3,4-diphenyl-2-methyl-1,2-dihydroisoquinoline (1a)10 and the 7-phenolic compound (1b).11 Compounds (1a,b) were found to have nearly equipotent anti-proliferative activities to that of tamoxifen against human mammary carcinoma MCF-7 cell line.

On the basis of these facts, 1,2-diphenyl-4,5-dihydro-3H-3-benzazepine (2a) having a novel structure is an interesting compound in the biological and synthetic points of view (Figure 1). We now report a convenient synthesis of 2a and the substituted compounds (2b-d) on the 1-phenyl group.
RESULTS AND DISCUSSION

In our previous papers, we reported the synthesis of 3,4-diphenyl-1,2-dihydroisoquinolines (1a,b)\textsuperscript{10,11} by acidic dehydration of the corresponding 3,4-diphenyl-4-hydroxy-1,2,3,4-tetrahydroisoquinolines, which were prepared by intramolecular Barbier reaction of N-(2-iodobenzyl)phenacylamines with n-butyllithium (n-C\textsubscript{4}H\textsubscript{9}Li). Recently, we reported\textsuperscript{12} the synthesis of the phenolic derivatives of 1-hydroxy-1-phenyl-2,3,4,5-tetrahydro-1\textit{H}-3-benzazepine (3) by cyclization of N-[2-(2-iodophenyl)-...
ethyl]phenacylamines with \( n-C_5H_5Li \). Thus, we carried out the synthesis of 4,5-dihydro-1,2-diphenyl-3-methyl-3H-3-benzazepines (2a-d) by dehydration of 1,2-diphenyl-1-hydroxy-3-methyl-2,3,4,5-tetrahydro-1H-3-benzazepines (4a-d) with trifluoromethanesulfonic acid (CF\(_3\)SO\(_3\)H) as shown in Scheme 1. The compounds 4a-c were synthesized by intramolecular Barbier reaction of the key intermediates, N-[2-(2-iodophenyl)ethyl]-\( \alpha \)-phenylphenacylamines (5a-c) with \( n-C_5H_5Li \) in 33-43% yields. The singlet signal (\( \delta \) 4.65) of C2-H in the \( ^1H \)-NMR spectrum of 4a showed a single diastereomer, which may be formed due to the steric hindrance between an \( \alpha \)-phenyl group and a phenyl group of the phenacylamine in 5a. Compounds (5a-c) were prepared by condensation of N-methyl-2-(2-iodophenyl)ethylamine (6) with \( \alpha \)-phenylphenacyl bromides (7a-c) in the presence of propylene oxide in high yields. The bromides (7a),\(^{13}\) (7b),\(^{10}\) and (7c) were obtained by bromination of benzyl phenyl ketones (10a-c) with benzyltrimethylammonium tribromide (BTMA Br\(_3\)) according to the method reported by us.\(^{10}\) The ketone (10c) was prepared by Friedel-Crafts reaction of veratrole (8c) with phenylacetylene chloride (9).

The phenolic 1-hydroxy-1,2-diphenyl-3-benzazepine (4d) was obtained by deprotection of the \( t \)-butyldimethylsilyl (TBDMS) group of compound (4e) with tetrabutylammonium fluoride (TBAF). Compound (4e) was prepared in the same way as 4a-c as follows. Protection of the phenolic group of benzyl phenyl ketone (10d) with \( t \)-butyldimethylsilyl chloride (TBDMSCl) and then bromination of the product (10e) with BTMA Br\(_3\) gave an \( \alpha \)-phenylphenacyl bromide (7e). The condensation of 7e with 6 afforded a key intermediate (5e), which was treated with \( n-C_5H_5Li \) to give 4e in 50% yield.

It is interesting to note that the yields (33-50%) of 1,2-diphenyl-1-hydroxy-3-benzazepines (4) in the cyclization reaction of N-[2-(2-iodophenyl)ethyl]-\( \alpha \)-phenylphenacylamines (5) with \( n-C_5H_5Li \) in this study are higher than those (15-32%) of the phenolic derivatives protected with TBDMS of 1-hydroxy-1-phenyl-3-benzazepine (3) in cyclization of N-[2-(2-iodophenyl)ethyl]phenacylamines reported in our previous paper.\(^{12}\) The higher yields of 4 may be attributed to the restriction of conformational freedom by the \( \alpha \)-phenyl group in 5.

In conclusion, a cyclization reaction of N-[2-(2-iodophenyl)ethyl]-\( \alpha \)-phenylphenacylamines (5) with \( n-C_5H_5Li \), followed by dehydration reaction of the products (4) in this study provides an applicable method for the preparation of 1,2-diphenyl-4,5-dihydro-3H-3-benzazepine derivatives (2).

**EXPERIMENTAL**

**General** All melting points are given as uncorrected values. High-resolution mass (HR-MS) spectra were recorded on a JEOL JMS-D 300 spectrometer. \( ^1H \)-NMR spectra were recorded on a JEOL JNM-FX 200 spectrometer with TMS as a standard.

**Benzyl 3,4-Dimethoxyphenyl Ketone (10c)** \( AlCl_3 \) (3.00 g, 22.5 mmol) was added to a mixture
of veratrole (8c) (6.31 mL, 49.5 mmol) and phenylacetyl chloride (9) (1.98 mL, 15 mmol) for 10 min and the mixture was stirred for 30 min at rt. The mixture was poured into a solution of 36 % HCl (5 mL) and ice-cold H₂O (50 mL) and the mixture was extracted with CHCl₃ (100 mL x 3). The extract was washed with brine (50 mL), dried over MgSO₄, and evaporated to give a pale brown oil. This was subjected to column chromatography on SiO₂ with n-hexane-AcOEt (5 : 1) to afford 10c as colorless needles (from n-hexane) (3.57 g, 92.9 %), mp 76-78°C. ¹H-NMR (CDCl₃) δ: 7.66 (1H, d, J=8.4 Hz), 7.55 (1H, s), 7.29 (5H, m), 6.88 (1H, d, J=8.4 Hz), 4.24 (2H, s), 3.93 (3H, s), 3.90 (3H, s). HR-MS m/z: Calcd for C₁₀H₁₆O₃: 256.1100 (M⁺). Found: 256.1077. Anal. Calcd for C₁₆H₁₆O₃: C, 74.98; H, 7.20. Found: C, 74.98; H, 7.29.

**Benzyl 4-(t-Butyldimethylsilyloxy)phenyl Ketone (10e)**

A mixture of 10d (0.76 g, 3.59 mmol), TBDMSCl (0.82 g, 5.38 mmol), and imidazole (0.54 g, 8.97 mmol) in dry CH₂Cl₂ (20 mL) was stirred for 2 h at rt. The mixture was washed with brine (50 mL x 3), dried over MgSO₄, and evaporated to give 10e as colorless plates (from EtOH) (1.13 g, 96.4 %), mp 97.5-99°C. ¹H-NMR (CDCl₃) δ: 7.93 (2H, d, J=8.7 Hz), 7.27 (5H, m), 6.86 (2H, d, J=8.7 Hz), 4.22 (2H, s), 0.98 (9H, s), 0.23 (6H, s). HR-MS m/z: Calcd for C₂₀H₂₆O₂Si: 326.1702 (M⁺). Found: 326.1703. Anal. Calcd for C₂₀H₂₆O₂Si: C, 73.57; H, 8.03. Found: C, 73.27; H, 7.99.

**3,4-Dimethoxy-α-phenlyphenacyl Bromide (7e)**

A mixture of 10c (2.00 g, 7.80 mmol) in CH₂Cl₂-CH₃OH (5:2) (35 mL). The mixture was refluxed for 5 h and evaporated in vacuo. H₂O (80 mL) was added to the residue and the mixture was extracted with CHCl₃ (70 mL x 3). The extract was washed with H₂O, dried over MgSO₄, and evaporated in vacuo to give a pale brown oil. This was purified by column chromatography on SiO₂ with CHCl₃ to afford 7c as pale yellow plates (from n-hexane) (1.24 g, 47.3%), mp 117-118°C. ¹H-NMR (CDCl₃) δ: 7.61 (1H, d, J=8.4 Hz), 7.55 (1H, s), 7.53 (2H, d, J=7.5 Hz), 7.35 (3H, m), 6.85 (1H, d, J=8.4 Hz), 6.38 (1H, s), 3.93 (3H, s), 3.90 (3H, s). HR-MS m/z: Calcd for C₁₀H₁₆Br: 334.0205 (M⁺). Found: 334.0216. Anal. Calcd for C₁₀H₁₆Br: C, 57.33; H, 4.51. Found: C, 57.20; H, 4.61.

**4-(t-Butyldimethylsilyloxy)-α-phenlyphenacyl Bromide (7e)**

In the same way as 10c, compound (10e) (1.00 g, 3.06 mmol) was treated with BTMA Br₃ (1.43 g, 3.68 mmol) to give 7e as a pale yellow oil (0.54 g, 43.3 %). ¹H-NMR (CDCl₃) δ: 7.91 (2H, d, J=9.0 Hz), 7.53 (2H, d, J=7.5 Hz), 7.35 (3H, m), 6.85 (1H, d, J=9.0 Hz), 6.34 (1H, s), 0.97 (9H, s), 0.22 (6H, s). HR-MS m/z: Calcd for C₁₀H₁₆Br: 404.0807 (M⁺). Found: 404.0766.

**N-[2-(2-Iodophenyl)ethyl]-N-methyl-α-phenlyphenacylamine (5a)**

A solution of 6 (1.622 g, 6.19 mmol), α-phenlyphenacyl bromide (7a) (1.42 g, 5.15 mmol), and propylene oxide (3 mL) in dioxane (40 mL) was heated at 110°C for 1.5 h. The mixture was evaporated and H₂O (50 mL) was added to the residue. The mixture was basified with 25 % NaOH and extracted with CHCl₃ (50 mL x 3). The extract was washed with H₂O, dried over MgSO₄, and evaporated to give a pale brown oil. This was subjected to column chromatography on SiO₂ with CHCl₃-acetone (100:1) to afford 5a as a pale yellow oil (2.04 g, 87.0 %). ¹H-NMR (CDCl₃) δ: 7.99 (2H, d, J=7.2 Hz), 7.74 (1H, d J=7.8 Hz), 7.47-7.36 (5H, m), 7.28 (3H,
N-[2-(2-Iodophenyl)ethyl]-4-methoxy-N-methyl-α-phenylphenacylamine (5b)  
Reaction of 6 (1.32 g, 5.03 mmol) with 7b (1.28 g, 4.19 mmol) and propylene oxide (2.57 mL) in dioxane (20 mL) gave 5b as a pale yellow oil (2.10 g, 98.0 %). 1H-NMR (CDCl3) δ: 7.01 (2H, d, J=8.7 Hz), 7.73 (7H, m), 8.01 (2H, d, J=8.7 Hz), 7.38-7.25 (5H, m), 7.20 (1H, t-like, J=7.5 Hz), 7.09 (1H, d, J=7.5 Hz), 6.84 (2H, d, J=8.7 Hz), 5.13 (1H, s), 3.81 (3H, s), 2.93-2.61 (4H, m), 2.48 (3H, s). HR-MS m/z: Calcd for C32H25NO2I: 516.1026. Found: 516.1026.

3,4-Dimethoxy-N-[2-(2-Iodophenyl)ethyl]-N-methyl-α-phenylphenacylamine (5c)  
Reaction of 6 (1.03 g, 3.93 mmol) with 6c (0.80 g, 2.93 mmol) and propylene oxide (2.01 mL) in dioxane (28 mL) gave 5c as an amorphous (1.18 g, 99.4 %). 1H-NMR (CDCl3) δ: 7.73 (1H, d, J=8.4 Hz), 7.70 (1H, d J=8.6 Hz), 7.57 (1H, d, J=1.8 Hz), 7.40-7.23 (5H, m), 7.20 (1H, dd, J=7.5 and 7.7 Hz), 7.09 (1H, d, J=7.7 Hz), 6.84 (2H, t-like, J=7.5 Hz), 6.79 (1H, d, J=8.4 Hz), 5.15 (1H, s), 3.90 (3H, s), 3.88 (3H, s), 2.97-2.61 (4H, m), 2.49 (3H, s). HR-MS m/z: Calcd for C32H25NO2I: 516.1031 (M+H). Found: 516.1026.

4-t-Butyldimethylsilyloxy-N-[2-(2-Iodophenyl)ethyl]-N-methyl-α-phenylphenacylamine (5e)  
Reaction of 6 (0.426 g, 1.63 mmol) with 7e (0.52 g, 1.29 mmol) and propylene oxide (0.85 mL) in dioxane (6 mL) gave 5e as a pale yellow oil (0.628 g, 83.3 %). 1H-NMR (CDCl3) δ: 7.94 (2H, d, J=8.7 Hz), 7.73 (1H, d, J=7.8 Hz), 7.38 (5H, m), 7.20 (1H, t-like, J=7.5 Hz), 7.08 (1H, d, J=7.8 Hz), 6.84 (2H, t-like, J=7.5 Hz), 6.77 (2H, d, J=8.7 Hz), 5.12 (1H, s), 2.96-2.62 (4H, m), 2.47 (3H, s), 0.96 (9H, s), 0.20 (6H, s). HR-MS m/z: Calcd for C32H36NO2ISi: 585.1560. Found: 585.1537.

1-Hydroxy-3-methyl-1,2-diphenyl-2,3,4,5-tetrahydro-1H-3-benzazepine (4a)  
n-C3H7Li (3.13 mL of 1.53 M solution in n-hexane, 4.79 mmol) were added to a solution of 5a (1.452 g, 3.19 mmol) in dry THF (30 mL) under argon atmosphere at -78°C. The mixture was stirred for 30 min at rt. H2O (30 mL) was added and the mixture was extracted with ether (50 mL x 3). The extract was dried over MgSO4 and evaporated to give a pale brawn oil. This was subjected to column chromatography on SiO2 with n-hexane-AcOEt (10:1) to afford 4a as a pale yellow oil (0.450 g, 42.8 %). 1H-NMR (CDCl3) δ: 7.48 (1H, d, J=7.5 Hz), 7.40 (2H, d, J=7.2 Hz), 7.30-7.09 (9H, m), 6.78 (2H, d, J=7.7 Hz), 4.65 (1H, s), 3.01-2.61 (4H, m), 2.18 (3H, s). HR-MS m/z: Calcd for C29H23NO: 329.1780 (M+). Found: 329.1770.

1-Hydroxy-3-benzazepines (4b,c) and (4e) were prepared in the same way as 4a.

1-Hydroxy-1-(4-methoxyphenyl)-3-methyl-2-phenyl-2,3,4,5-tetrahydro-1H-3-benzazepine (4b)  
Compound (5b) (0.50 g, 1.03 mmol) was reacted with n-C3H7Li (1.04 mL of 1.53 M solution in n-hexane, 1.58 mmol) in dry THF (8 mL). The crude product was purified by column chromatography on SiO2 with n-hexane-AcOEt (5:1) to give 4b as a pale yellow oil (0.123 g, 33.2 %). 1H-NMR (CDCl3) δ: 7.52 (1H, d,
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\( J=7.5 \text{ Hz} \), 7.33 (2H, d, \( J=8.7 \text{ Hz} \)), 7.21-7.10 (6H, m), 6.83 (2H, d, \( J=8.7 \text{ Hz} \)), 6.75 (2H, d, \( J=7.2 \text{ Hz} \)), 4.62 (1H, s), 3.78 (3H, s), 3.01-2.59 (4H, m), 2.18 (3H, s). HR-MS m/z: Calcd for \( \text{C}_2\text{H}_{15}\text{NO}_2: 359.1886 \) (M'). Found: 359.1888.

1-(3,4-Dimethoxyphenyl)-1-hydroxy-3-methyl-2-phenyl-2,3,4,5-tetrahydro-1H-3-benzazepine (4c)

Compound (5c) (0.41 g, 0.80 mmol) was reacted with \( n-\text{C}_6\text{H}_5\text{Li} \) (0.78 mL of 1.53 M solution in \( n \)-hexane, 1.20 mmol) in dry THF (2.5 mL). The crude product was purified by column chromatography on SiO\( _2 \) with CHCl\(_3\)-acetone (20:1) to give 4c as a pale brown amorphous (0.108 g, 34.9 %). \( ^1\text{H}-\text{NMR} \) (CDCl\(_3\)) \( \delta \): 7.47 (1H, d, \( J=7.3 \text{ Hz} \)), 7.20-7.10 (6H, m), 6.99 (1H, d, \( J=2.0 \text{ Hz} \)), 6.91 (1H, dd, \( J=8.5 \) and 2.0 Hz), 6.79 (2H, d, \( J=7.2 \text{ Hz} \)), 6.78 (1H, d, \( J=8.5 \text{ Hz} \)), 4.58 (1H, s), 3.84 (3H, s), 3.78 (3H, s), 3.10-2.63 (4H, m), 2.19 (3H, s). HR-MS m/z: Calcd for \( \text{C}_{24}\text{H}_{25}\text{NO}_2: 389.2004 \) (M'). Found: 389.2004.

1-[4-(t-Butyldimethylsilyloxy)phenyl]-1-hydroxy-3-methyl-2-phenyl-2,3,4,5-tetrahydro-1H-3-benzazepine (4e)

Compound (5e) (0.48 g, 0.83 mmol) was reacted with \( n-\text{C}_6\text{H}_5\text{Li} \) (0.76 mL of 1.53 M solution in \( n \)-hexane, 1.16 mmol) in dry THF (2.5 mL). The crude product was purified by column chromatography on SiO\( _2 \) with \( n \)-hexane-AcOEt (15:1) to give 4e as a pale yellow oil (0.19 g, 50.0 %). \( ^1\text{H}-\text{NMR} \) (CDCl\(_3\)) \( \delta \): 7.49 (1H, d, \( J=7.5 \text{ Hz} \)), 7.40-7.09 (10H, m), 6.74 (2H, d, \( J=8.8 \text{ Hz} \)), 4.59 (1H, s), 3.00-2.83 (2H, m), 2.71-2.60 (2H, m), 2.17 (3H, s), 0.90 (9H, s), 0.17 (6H, s). HR-MS m/z: Calcd for \( \text{C}_{29}\text{H}_{35}\text{NO}_2\text{Si}: 459.2593 \) (M'). Found: 459.2579.

1-Hydroxy-1-(4-hydroxyphenyl)-3-methyl-2-phenyl-2,3,4,5-tetrahydro-1H-3-benzazepine (4d)

A solution of TBAF (1.20 mL of 1.0 M solution in THF, 1.20 mmol) in dry THF (1.2 mL) was added to a solution of 4e (0.184 g, 0.40 mmol) in dry THF (3 mL) under ice-cooling. The mixture was stirred for 30 min. H\(_2\)O (70 mL) was added and the mixture was extracted with CHCl\(_3\) (100 mL x 3). The extract was washed with \( \text{H}_2\text{O} \), dried over MgSO\(_4\), and evaporated to give a pale brown oil. This was purified by column chromatography on SiO\( _2 \) with CHCl\(_3\)-acetone (10:1) to give 4d as a pale yellow amorphous (0.081 g, 58.5 %). \( ^1\text{H}-\text{NMR} \) (CDCl\(_3\)) \( \delta \): 7.48 (1H, d, \( J=7.5 \text{ Hz} \)), 7.32-7.10 (8H, m), 6.75 (2H, d, \( J=7.5 \text{ Hz} \)), 6.63 (2H, d, \( J=8.3 \text{ Hz} \)), 4.62 (1H, s), 3.12-2.63 (4H, m), 2.16 (3H, s). HR-MS m/z: Calcd for \( \text{C}_{25}\text{H}_{27}\text{NO}_2: 345.1729 \) (M'). Found: 345.1729.

3-Methyl-1,2-diphenyl-4,5-dihydro-3H-3-benzazepine (2a)

\( \text{CF}_3\text{SO}_3\text{H} \) (0.162 ml, 1.82 mmol) was added to a solution of 4a (0.120 g, 0.37 mmol) in dry benzene (5 mL) and the mixture was refluxed for 30 min at 100°C. The reaction mixture was made basic with 5 % NaOH and extracted with CHCl\(_3\) (70 mL x 3). The extract was washed with \( \text{H}_2\text{O} \), dried over MgSO\(_4\), and evaporated in vacuo to give 2a as a pale yellow solid (0.089 g, 78.5 %). \( ^1\text{H}-\text{NMR} \) (CDCl\(_3\)) \( \delta \): 7.17 (1H, m), 7.00 (12H, m), 6.85 (1H, m), 3.49 (2H, m), 3.11 (2H, m), 2.42 (3H, s). HR-MS m/z: Calcd for \( \text{C}_{23}\text{H}_{21}\text{N}: 311.1674 \) (M'). Found: 311.1646.

1, 2-Diphenyl-3-benzazepines (2b-d) were prepared in the same way as 2a.

1-(4-Methoxyphenyl)-3-methyl-2-phenyl-4,5-dihydro-3H-3-benzazepine (2b)
(0.093 g, 0.26 mmol) was reacted with CF$_3$SO$_3$H (0.114 mL, 1.29 mmol) in benzene (5 mL) for 30 min at 100°C. The crude product was purified by column chromatography on SiO$_2$ with n-hexane-AcOEt (5:1) to give 2b as a pale yellow solid (0.033 g, 36.9%). $^1$H-NMR (CDCl$_3$) δ: 7.18 (1H, m), 7.09 (4H, m), 7.05-7.00 (3H, m), 6.91 (2H, d, J=8.5 Hz), 6.88 (1H, m), 6.59 (2H, d, J=8.5 Hz), 3.70 (3H, s), 3.49 (2H, m), 3.10 (2H, m), 2.41 (3H, s). HR-MS m/z: Calcd for C$_{24}$H$_{23}$NO: 341.1779 (M$^+$). Found: 341.1809.

1-(3,4-Dimethoxyphenyl)-3-methyl-2-phenyl-4,5-dihydro-3H-3-benzazepine (2c) Compound (4c) (0.041 g, 0.11 mmol) was reacted with CF$_3$SO$_3$H (0.082 mL, 0.55 mmol) in benzene (3.5 mL) for 30 min at 90°C. The crude product was purified by column chromatography on SiO$_2$ with n-hexane-AcOEt (10:1) to give 2c as a pale yellow solid (0.0153 g, 37.7%). $^1$H-NMR (CDCl$_3$) δ: 7.18 (1H, m), 7.10-7.01 (7H, m), 6.92 (1H, m), 6.58-6.52 (3H, m), 3.77 (3H, s), 3.65 (3H, s), 3.46 (2H, m), 3.08 (2H, m), 2.43 (3H, s). HR-MS m/z: Calcd for C$_{25}$H$_{25}$NO$_2$: 371.1886 (M$^+$). Found: 371.1858.

1-(4-Hydroxyphenyl)-3-methyl-2-phenyl-4,5-dihydro-3H-3-benzazepine (2d) Compound (4d) (0.017 g, 0.048 mmol) was reacted with CF$_3$SO$_3$H (0.021 mL, 0.24 mmol) in benzene (1.0 mL) for 30 min at 90°C. The crude product was purified by column chromatography on SiO$_2$ with n-hexane-AcOEt (5:1) to give 2d as a pale yellow oil (0.0083 g, 52.9%). $^1$H-NMR (CDCl$_3$) δ: 7.18 (1H, m), 7.10-6.70 (7H, m), 6.88 (1H, m), 6.85 (2H, d, J=8.5 Hz), 6.51 (2H, d, J=8.5 Hz), 3.48 (2H, m), 3.09 (2H, m), 2.40 (3H, s). HR-MS m/z: Calcd for C$_{23}$H$_{21}$NO: 327.1623 (M$^+$). Found: 327.1592.

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