Synthesis of New 2,3-Dihydro-2-phenyl-4-quinolone Derivatives;
Aza Analogs of Flavanone

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Flavonoids are natural polyphenol compounds of plant origin and exhibit various biological activities such as antiinflammatory, anti-oxidant, and anti-tumor activities.[1-3] It has been previously reported that various plant flavonoids possess the inhibitory activity on cyclooxygenase/lipoxygenase.[4-11]

Based on the results from the structure-activity relationships of Hesperetin and some other naturally occurring flavanone such as Taxifolin (Figure 2) and in order to improve the antiinflammatory activities of Hesperetin, we

designed aza analogs of flavanones that were replaced the oxygen atom of B ring of flavanone to the nitrogen atom (Figure 1). In this work, we report the synthesis of the title compound **3**, novel 2,3-dihydro-2-phenyl-4-quinolone derivatives as a potential candidate for NSAIDs (non-steriodal antiinflammatory drugs).

A series of 2,3-dihydro-2-phenyl-4-quinolones **3a-g** has been synthesized using acid-catalyzed one-pot reaction (Scheme 1). Quinolones **3a-g** were prepared through cyclization of the condensation product **2a-g** that were formed by heating of arylamines and ethyl benzoylacetate in toluene. Similarly, the 6 (7 or 8)-substituted 2,3-dihydro-2-phenylquinolones were prepared from the para(ortho or meta)-substituted aniline. The reaction mechanism of the formation of the final product involves the nucleophilic dehydration and following cyclization between arylamines and ethyl benzoylacetate (EBA). Nucleophilic dehydration, the condensation was undertaken with *p*-toluene sulfonic acid at 90-110 °C in toluene for 2-6 hours over the dean-
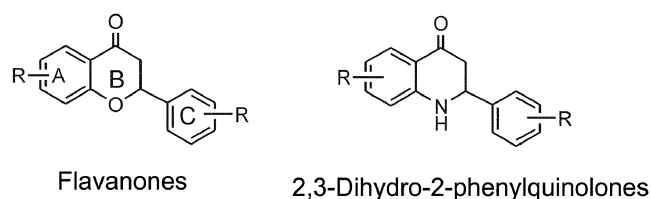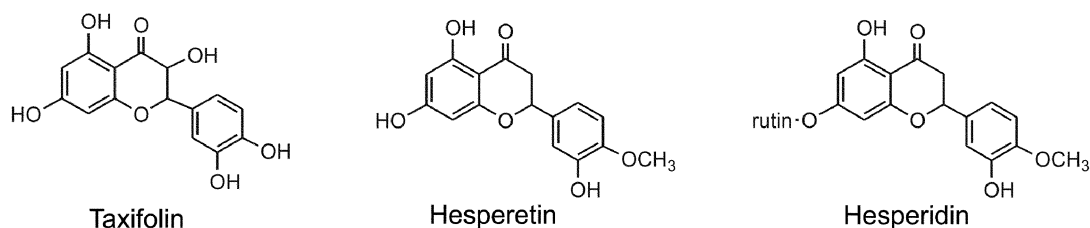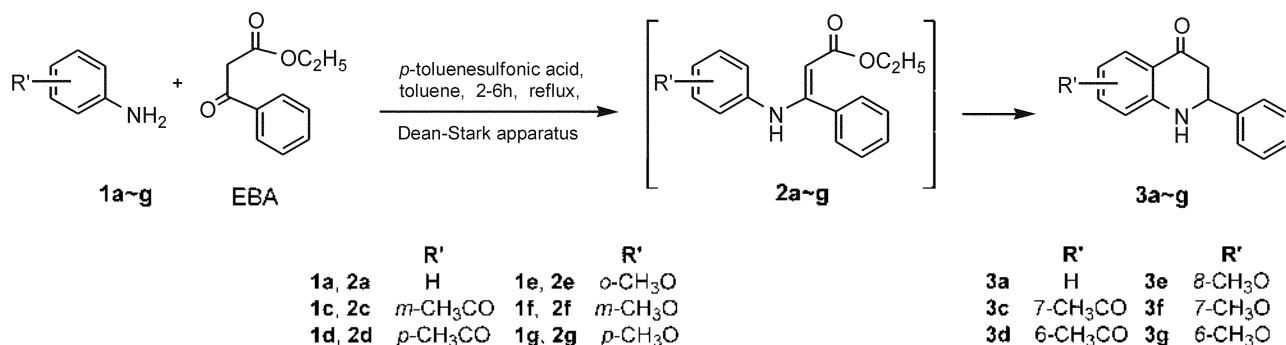

Flavanones   2,3-Dihydro-2-phenylquinolones

Figure 1. Structure of flavanones.

Taxifolin   Hesperetin   Hesperidin

Figure 2. Structure of flavanones with antiinflammatory activity.

	R'		R'			R'		R'
1a, 2a	H	1e, 2e	*o*-CH₃O		3a	H	3e	*8*-CH₃O
1c, 2c	*m*-CH₃CO	1f, 2f	*m*-CH₃O		3c	7-CH₃CO	3f	*7*-CH₃O
1d, 2d	*p*-CH₃CO	1g, 2g	*p*-CH₃O		3d	6-CH₃CO	3g	6-CH₃O

Scheme 1. Synthesis of 2,3-Dihydro-2-phenylquinolone Derivatives **3a-g** and Isolation of the intermediate **2a-g**.

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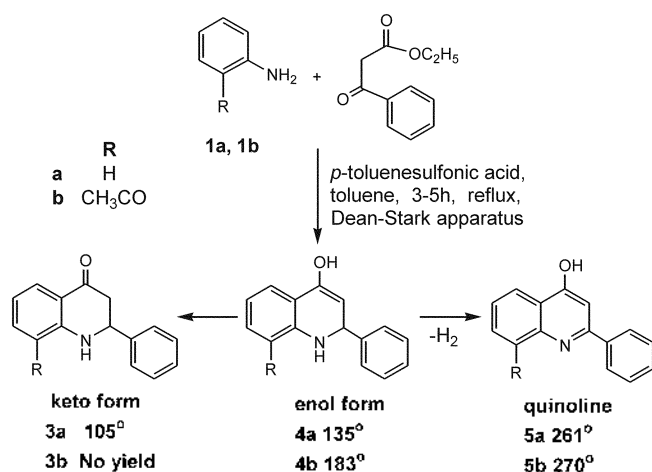
Scheme 2. Tautomerism of 2,3-Dihydro-2-phenylquinolone Derivatives **3a** and Dehydrogenation of **4b**.

stark apparatus for the ester **2**. The ester **2** was continuously converted to the 2-phenylquinolones **3** with removal of ethanol and dihydrogenation. All synthetic process from arylamines and ethyl benzoylacetate to quinolones **3a-g** could be carried out in one-pot without isolation of intermediates.

Even though synthetic pathways for 2-phenyl-4-quinolones were developed by Li *et al.*,[12] Kuo *et al.*,[13] Watterson *et al.*[14] and Xia *et al.*,[15] the synthesis of 2,3-dihydro-2-phenyl-4-quinolones has not been reported until now. We applied the synthetic method of one-pot operations for dihydroquinolone by Cho *et al.*[16] We synthesized the 2,3-dihydro-2-phenyl-4-quinolones using the reaction between ethyl benzoylacetate and arylamines.

The moiety that were introduced to aromatic nucleus of quinolones **3a-g** were nonsubstituted, 7-acetyl, 6-acetyl, 8-methoxy, 7-methoxy and 6-methoxy. Even though we efforted to complete cyclization in several methods, **2a-g** were always isolated in reaction mixture. In the acid-catalytic dehydration, ethyl benzoylacetate was protonated and followed to transfer of electron. A molecule of water eliminated from nucleophillic attraction of aniline, C-N bond was formed for esters **2a-g**. Starting material nearly disappeared after about 1 hour after starting of reaction.

Even though esters **2a-g** were converted to **3** and **4**, they were left in reaction mixture in state of chemical equibrium (Scheme 2). Prolonged reaction time could not make into a complection of reaction, but resulted in a complicated reaction mixture. For the additional cyclization, ester **2a-g** were protonated at 2 and 3-position of ring, converted with **3** and **4** by eliminating ethanol.

Although enols **4a, b** were isolated and charaterized, we found that **4a** was transformed to quinolone **3a** in nonpolar solvent such as CDCl₃. Enol **4a** was stable in state of solid, but changed to keto-form **3a** as hydrogen transfer in solvent. Enol-form was transformed to more stable keto-form in the course of time. In the synthesis for **3b**, the product was not keto-form **3b** but enol-form **4b**. Enol **4b** was not transformed

into enol **3b** in solvent, but dehydrogenation at 1,2-position of quinolone ring took place during the process of reaction and chromatographic (SiO₂) isolation gave quinoline compound **5a-b** (Scheme 2).

Enols **4a, b** and keto-form **3a** were defined by ¹H, ¹³C-NMR, IR spectra and GC-MS. In the ¹H-NMR, 11.02 ppm of OH peak of enol-form was disappeared in keto-form. In ¹³C-NMR spectra of the enol- and keto-form, C-O peak of enol-form appeared at 168.20 ppm, while C=O peak of keto-form showed at the 196.50 ppm. Even though the carbonyl band of keto-form was clearly shown at about 1689 cm⁻¹ on IR spectra, was not shown in case of enol-form.

Enol-form **4b** was not transformed into keto-form **3b** but into quinoline **5b** because of a strong steric hindrance between 8-acetoxy moiety and NH of B ring and a weak conjugation effect. Keto-forms **3c** (6-position) and **3d** (7-position) were produced in this condition since they do not have steric hindrance. The 8-methoxy compound **3e** was synthesized on less steric hindrance with NH of B ring because methoxy moiety was less bulky than acetoxy moiety. The reason for conversion of unsubstituted compound **4a** into **3a** in solvent such as CDCl₃ is that keto-form is more stable than enol-form. Also, a part of **4a** was transformed into quinoline **5a** due to conjugation effect. We thought that keto-form was more stable than enol-form because most of reaction was processed to yield keto-form **3c-g**. We could not find enol-form in reaction of methoxy compound because electron donating methoxy group facilitate cyclization and additional transfer of proton to make keto-form.

Identification of products **2a-g** and **3a-g** were defined by spectra of NMR, IR and GC-MS. The NH peak of **3a-g** was appeared at 9.2 ppm, a coupling of CH peak at 7.12 ppm and CH₂ peak at 4.11 ppm was observed.

Finally, 2,3-dihydro-2-phenyl-4-quinolones **3a, 3c-g** were synthesized through the condensation and cyclization of related amines and ethyl benzoylacetate. Esters **2a-g** were isolated as intermediates through one-pot reaction (Scheme 1).

Experimental Section

Chemicals were supplied by Aldrich, Sigma, Merck, and Tokyo Kasei. Melting points were determined in open capillary tubes on a Büchi 535 melting point apparatus and uncorrected. The NMR spectra were recorded using Bruker 300 MHz NMR spectrometer. Chemical shift values were reported in parts per million on the scale in deuteriochloroform or dimethyl-d₆ sulfoxide with tetramethylsilane as the internal standard. The NMR spin multiplicities were indicated by the symbols: s (singlet), d (doublet), t (triplet), q (quartet) and m (multiplet). IR spectra were recorded on a Perkin-Elmer 16F PC FT-IR spectrometer using NaCl discs and pellets. The Mass fragmentations were recorded using Agilent 6890 GC and 5973 MS.

Isolation of intermediate 2a-g; 3-phenyl-3-anilinopropenoic acid ethyl ester (2a). mp 72.5 °C TLC [*n*-hexane : ethyl acetate (3 : 1)] Rf 0.73. ¹H NMR (CDCl₃) δ 10.30 (s, 1H, NH), 7.35-7.24 (m, 5H, aromatic), 7.08 (t, *J* = 7.5 Hz,

2H, aromatic), 6.91 (t, *J* = 7.4 Hz, 1H, aromatic), 6.65 (d, *J* = 7.6 Hz, 2H, aromatic), 4.99 (s, 1H, CH), 4.20 (q, *J* = 7.1 Hz, 2H, CH$_2$), 1.31 (t, *J* = 7.1 Hz, 3H, CH$_3$). ^{13}C NMR (CDCl$_3$) δ 170.10 (COO), 159.04, 140.35, 135.95, 129.42, 128.59, 128.41, 128.20, 122.17, 91.14 (CH), 59.30 (CH$_2$), 14.52 (CH$_3$). IR (NaCl, cm^{-1}) 3259 (NH), 2979 (aromatic), 1657 (CO).

3-Phenyl-3-(3-acetoxyanilino)propenoic acid ethyl ester (2c), oil TLC [*n*-hexane : ethyl acetate (3 : 2)] Rf 0.57. ^{1}H NMR (CDCl$_3$) δ 10.40 (s, 1H, NH), 7.56 (s, 1H, aromatic), 7.32 (m, 5H, aromatic), 7.20 (s, 1H, aromatic), 7.13 (t, 1H, aromatic), 6.81 (d, *J* = 7.4 Hz, 2H, aromatic), 5.05 (s, 1H, CH), 4.20 (q, *J* = 7.1 Hz, 2H, CH$_2$), 3.32 (s, 2H, COCH$_3$), 1.30 (t, *J* = 7.1 Hz, 3H, CH$_3$). ^{13}C NMR (CDCl$_3$) δ 196.79 (C=O), 170.33 (COO), 158.77, 141.19, 137.88, 131.60, 130.07, 129.26, 129.13, 129.04, 128.90, 126.47, 122.77, 122.02, 92.80 (CH), 60.68 (CH$_2$), 26.79 (CH$_3$) 14.58 (CH$_3$). IR (pellet, cm^{-1}) 3367 (NH), 2980 (aromatic), 1740 (CO), 1664 (CO).

3-Phenyl-3-(4-acetoxyanilino)propenoic acid ethyl ester (2d), oil TLC [*n*-hexane : ethyl acetate (3 : 1)] Rf 0.44. ^{1}H NMR (CDCl$_3$) δ 10.73 (s, 1H, NH), 7.68 (d, *J* = 7.4 Hz, 2H, aromatic), 7.34 (m, 5H, aromatic), 6.64 (d, *J* = 7.4 Hz, 2H, aromatic), 5.11 (s, 1H, CH), 4.23 (q, *J* = 7.1 Hz, 2H, CH$_2$), 2.46 (s, 2H, COCH$_3$), 1.30 (t, *J* = 7.1 Hz, 3H, CH$_3$). ^{13}C NMR (CDCl$_3$) δ 196.69 (C=O), 169.77 (COO), 155.44, 144.98, 133.73, 129.80, 129.39, 128.77, 128.49, 120.24, 94.28 (CH), 61.37 (CH$_2$), 29.28 (CH$_3$) 14.44 (CH$_3$). IR (pellet, cm^{-1}) 3367 (NH), 2980 (aromatic), 1740 (CO), 1664 (CO).

3-Phenyl-3-(2-methoxyanilino)propenoic acid ethyl ester (2e), mp 105 °C TLC [*n*-hexane : ethyl acetate (2 : 1)] Rf 0.63. ^{1}H NMR (CDCl$_3$) δ 10.27 (s, 1H, NH), 7.36-7.27 (m, 5H, aromatic), 6.83 (m, 2H, aromatic), 6.49 (t, *J* = 7.2 Hz, 1H, aromatic), 6.20 (d, *J* = 7.2 Hz, 1H, aromatic), 4.99 (s, 1H, CH), 4.21 (q, *J* = 7.2 Hz, 2H, CH$_2$), 3.88 (s, 3H, OCH$_3$), 1.30 (t, *J* = 7.2 Hz, 3H, CH$_3$). ^{13}C NMR (CDCl$_3$) δ 170.25 (COO), 158.79, 150.89, 136.78, 130.01, 129.78, 128.79, 128.39, 123.29, 122.19, 120.27, 110.91, 91.99 (CH), 59.65 (OCH$_2$), 56.11 (OCH$_3$) 14.98 (CH$_3$).

3-Phenyl-3-(3-methoxyanilino)propenoic acid ethyl ester (2f), oil TLC [n-hexane : ethyl acetate (2 : 1)] Rf 0.59. ^{1}H NMR (CDCl$_3$) δ 10.29 (s, 1H, NH), 7.37-7.28 (m, 5H, aromatic), 6.96 (t, *J* = 8.1 Hz, 1H, aromatic), 6.26 (t, *J* = 8.1 Hz, 1H, aromatic), 6.16 (d, *J* = 8.1 Hz, 1H, aromatic), 4.99 (s, 1H, CH), 4.20 (q, *J* = 7.2 Hz, 2H, CH$_2$), 3.53 (s, 3H, OCH$_3$), 1.31 (t, *J* = 7.2 Hz, 3H, CH$_3$). ^{13}C NMR (CDCl$_3$) δ 170.46 (COO), 160.14, 159.28, 141.95, 136.50, 129.85, 129.68, 128.86, 128.53, 114.82, 109.44, 107.83, 91.78 (CH), 59.75 (OCH$_2$), 55.37 (OCH$_3$), 14.92 (CH$_3$).

3-Phenyl-3-(4-methoxyanilino)propenoic acid ethyl ester (2g), mp 115 °C TLC [*n*-hexane : ethyl acetate (2 : 1)] Rf 0.6. ^{1}H NMR (CDCl$_3$) δ 10.22 (s, 1H, NH), 7.36-7.28 (m, 5H, aromatic), 6.62 (m, 4H, aromatic), 4.93 (s, 1H, CH), 4.21 (q, *J* = 7.1 Hz, 2H, CH$_2$), 3.69 (s, 3H, OCH$_3$), 1.33 (t, *J* = 7.1 Hz, 3H, CH$_3$). ^{13}C NMR (CDCl$_3$) δ 170.29 (COO), 159.88, 155.78, 136.01, 133.46, 129.22, 128.34, 128.28,

124.24, 113.84, 89.51 (CH), 59.16 (OCH$_2$), 55.30 (OCH$_3$) 14.55 (CH$_3$), IR (NaCl, cm^{-1}) 3255 (NH), 2976 (aromatic), 1734 (CO), 1652 (CO).

General procedure for the synthesis of 3a, 3c-g; 2,3-Dihydro-2-phenyl-4-quinolone (3a). Ethyl benzoylacetate (1.72 mL, 0.01 mol), *p*-toluenesulfonic acid (19.2 mg, 0.1 mmol) and aniline (1 mL, 0.011 mol) were refluxed for about 4 h in anhydrous toluene (30 mL) over the Dean-stark apparatus until the starting material disappeared. The reaction mixture was allowed to cool to room temperature, concentrated under reduced pressure to remove the toluene. The residue was extracted with ethyl acetate (30 mL) and water (15 mL) and the organic layer was washed with 5% NaHCO$_3$ (15 mL), water (15 mL × 2). The extracts were dried over anhydrous Na$_2$SO$_4$, and concentrated under a reduced pressure. The residue was purified by column chromatography to give compound 3a as a high viscous oil. The oil was crystallized from methanol.

Yield: 31.3%, mp 105 °C TLC [*n*-hexane : ethyl acetate (3 : 1)] Rf 0.27. ^{1}H NMR (CDCl$_3$) δ 9.29 (s, 1H, NH), 8.04 (d, *J* = 7.2 Hz, 2H, aromatic), 7.48 (m, 6H, aromatic), 7.33 (t, *J* = 7.2 Hz, 2H, aromatic), 7.12 (t, *J* = 7.2 Hz, 1H, CH), 4.11 (s, 2H, CH$_2$). ^{13}C NMR (CDCl$_3$) δ 196.50 (C=O), 163.74, 137.56, 136.07, 134.37, 129.00, 128.62, 124.57, 120.20 (CH), 45.52 (CH$_2$). IR (pellet cm^{-1}) 3307 (NH), 3063 (aromatic), 1689 (C=O). GC-MS: m/z 93.10 (100.00), 66.10 (30.79), 65.10 (15.44), 92.20 (12.31), 94.10 (7.13).

7-Acetoxy-2,3-dihydro-2-phenyl-4-quinolone (3c). Yield: 43.9%, mp 151 °C TLC [*n*-hexane : ethyl acetate (3 : 2)] Rf 0.31. ^{1}H NMR (CDCl$_3$) δ 9.60 (s, 1H, NH), 8.44 (m, 2H, aromatic), 8.10 (m, 1H, aromatic), 7.58 (m, 3H, aromatic), 7.49 (m, 2H, CH), 4.12 (s, 2H, CH$_2$), 2.55 (s, 3H, CH$_3$). ^{13}C NMR (CDCl$_3$) δ 198.89 (C=O), 196.32 (C=O), 165.05, 138.09, 137.78, 136.34, 134.72, 129.69, 129.34, 128.89, 125.20, 124.71, 120.16, 46.25 (CH$_2$), 27.46 (CH$_3$). IR (pellet cm^{-1}) 3345 (NH), 1681, 1656 (C=O). GC-MS: m/z 222.20 (100.00), 237.20 (54.07), 223.10 (17.30), 91.10 (12.96), 238.20 (9.58).

6-Acetoxy-2,3-dihydro-2-phenyl-4-quinolone (3d). Yield: 67.4%, mp 164 °C TLC [*n*-hexane : ethyl acetate (2 : 1)] Rf 0.21. ^{1}H NMR (CDCl$_3$) δ 9.69 (s, 1H, NH), 8.03 (d, *J* = 6.9 Hz, 2H, aromatic), 7.99 (d, *J* = 6.9 Hz, 2H, aromatic), 7.68 (m, 3H, aromatic), 7.55 (t, *J* = 6.9 Hz, 2H, CH), 4.15 (s, 2H, CH$_2$), 2.55 (s, 3H, CH$_3$). ^{13}C NMR (CDCl$_3$) δ 197.01 (C=O), 196.42 (C=O), 164.08, 141.88, 135.87, 134.60, 133.13, 129.73, 129.06, 128.60, 119.38, 45.20 (CH$_2$), 26.46 (CH$_3$). IR (pellet cm^{-1}) 3435 (NH), 1687, 1663 (C=O). GC-MS: m/z 120.10 (100.00), 135.10 (51.28), 92.10 (37.80), 65.10 (21.62), 121.10 (8.26).

8-Metoxy-2,3-dihydro-2-phenyl-4-quinolone (3e). Yield: 41.6%, mp 87 °C TLC [*n*-hexane : ethyl acetate (2 : 1)] Rf 0.37. ^{1}H NMR (CDCl$_3$) δ 9.40 (s, 1H, NH), 8.37 (d, *J* = 8.1 Hz, 1H, aromatic), 7.96 (d, 2H, aromatic), 7.43-7.36 (m, 3H, aromatic), 7.01-6.80 (m, 3H, aromatic), 4.11 (s, 2H, CH$_2$), 3.82 (s, 3H, OCH$_3$). ^{13}C NMR (CDCl$_3$) δ 196.09 (C=O), 164.24 (CO), 148.76, 136.53, 134.52, 134.44, 129.24, 129.00, 128.88, 128.35, 127.90, 126.23, 124.51, 121.40,

120.51, 110.55, 56.22 (OCH₃), 46.99 (CH₂). IR (NaCl, cm⁻¹) 3291 (NH), 3089 (aromatic), 1693 (C=O). GC-MS: m/z 108.10 (100.00), 123.10 (99.66), 80.10 (70.35), 53.10 (12.48), 52.10 (8.69).

7-Metoxy-2,3-dihydro-2-phenyl-4-quinolone (3f). Yield: 55.3%. oil TLC [n-hexane : ethyl acetate (2 : 1)] Rf 0.32. ¹H NMR (CDCl₃) δ 9.26 (s, 1H, NH), 7.94 (d, J = 7.5 Hz, 2H, aromatic), 7.55 (t, J = 7.5 Hz, 1H, aromatic), 7.42 (t, J = 7.5 Hz, 2H, aromatic), 7.21 (s, 1H, aromatic), 7.13 (t, 1H, aromatic), 6.99 (d, J = 7.2 Hz, 1H, aromatic), 6.60 (dd, 1H, CH), 4.03 (d, J = 10.1 Hz, 2H, CH₂), 3.71 (s, 3H, OCH₃). ¹³C NMR (CDCl₃) δ 196.75 (C=O), 164.33, 160.48, 139.16, 136.38, 134.75, 130.08, 129.36, 129.00, 128.88, 128.45, 112.76, 110.86, 106.20, 55.70 (OCH₃), 46.02 (CH₂). GC-MS: m/z 73.10 (100.00), 60.10 (72.73), 55.10 (59.93), 129.10 (59.70), 57.10 (57.74).

6-Metoxy-2,3-dihydro-2-phenyl-4-quinolone (3g). Yield: 36.8%. mp 120 °C TLC [n-hexane : ethyl acetate (2 : 1)] Rf 0.26. ¹H NMR (CDCl₃) δ 9.24 (s, 1H, NH), 8.01 (d, J = 7.2 Hz, 2H, aromatic), 7.61 (m, 1H, aromatic), 7.50 (t, J = 7.2 Hz, 4H, aromatic), 6.86 (d, J = 7.2 Hz, 2H, aromatic), 4.09 (s, 2H, CH₂), 3.78 (s, 3H, OCH₃). ¹³C NMR (CDCl₃) δ 196.78 (C=O), 164.23, 156.93, 136.45, 134.66, 131.11, 129.33, 128.98, 128.85, 122.41, 114.49, 55.84 (OCH₃), 45.89 (CH₂). IR (NaCl, cm⁻¹) 3291 (NH), 3089 (aromatic), 1693 (C=O). GC-MS: m/z 108.10 (100.00), 123.10 (77.30), 80.10 (33.06), 53.10 (9.69), 109.10 (7.39).

4-Hydroxy-2-hydro-2-phenyl-4-quinolone (4a). Yield: 45.5%. mp 135.5 °C TLC [n-hexane : ethyl acetate (3 : 1)] Rf 0.48. ¹H NMR (CDCl₃) δ 11.02 (s, 1H, OH), 7.51 (d, J = 7.5 Hz, 2H, aromatic), 7.28 (m, 4H, aromatic+NH), 7.03 (m, 2H, aromatic), 6.87 (t, J = 7.5 Hz, 1H, aromatic), 6.62 (d, J = 7.5 Hz, 2H, aromatic) 4.92 (s, 1H, CH). ¹³C NMR (CDCl₃) δ 168.20 (C-O), 157.67, 140.51, 138.49, 136.36, 129.26, 128.94, 128.56, 128.51, 128.11, 122.57, 121.97 (CH), 120.21. IR (pellet cm⁻¹) 3307 (NH), 3058 (aromatic), 1592. GC-MS: m/z 180.10 (100.00), 77.10 (49.5), 195.10 (47.99), 51.10 (17.14), 181.10 (14.01).

8-Acetoxy-4-hydroxy-2-hydro-2-phenyl-4-quinolone (4b). Yield: 38.6%. mp 183 °C TLC [n-hexane : ethyl acetate (1 : 1)] Rf 0.32. ¹H NMR (CDCl₃) δ 8.50 (s, 1H, OH), 8.07 (d, J = 7.2 Hz, 2H, aromatic), 7.58 (m, 2H, aromatic), 7.47 (m, 3H, aromatic), 7.22 (m, 3H, aromatic), 6.71 (d, J = 7.2 Hz, 1H, CH), 4.68 (s, 1H, CH), 1.63 (s, 3H, CH₃). ¹³C NMR (CDCl₃) δ 197.04 (C=O), 166.63 (C-O), 136.75, 134.20, 130.14, 129.39, 128.94, 128.62, 124.36, 124.17, 115.87, 72.48, 59.80, 27.38. IR (pellet cm⁻¹) 3310 (OH), 3058 (aromatic), 1705, 1646, 1595. GC-MS: m/z 120.10 (100.00), 135.10 (75.18), 92.10 (42.89), 65.10 (25.20), 121.10 (8.13).

4-Hydroxy-2-phenylquinoline (5a). Yield: 16%. mp 261 °C TLC [n-hexane : ethyl acetate (3 : 1)] Rf 0.1. ¹H NMR (DMSO-d₆) δ 11.75 (s, 1H, OH), 8.14 (d, J = 7.8 Hz, 1H, aromatic), 7.84 (m, 3H, aromatic), 7.80 (m, 1H, aromatic), 7.69 (m, 3H, aromatic), 7.22 (t, J = 7.8 Hz, 1H, aromatic) 6.39 (s, 1H, CH). ¹³C NMR (DMSO-d₆) δ 177.75 (C-O),

150.31, 140.63, 134.49, 131.53, 130.23, 128.79, 127.37, 124.98, 124.84, 123.13, 118.72, 107.65(CH).

8-Acetoxy-4-hydroxy-2-phenylquinoline (5b). Yield: 28%. mp 270 °C TLC [n-hexane : ethyl acetate (1 : 1)] Rf 0.23. ¹H NMR (DMSO-d₆) δ 12.02 (s, 1H, OH), 7.87 (dd, J = 7.5 Hz, 3H, aromatic), 7.66 (t, 1H, aromatic), 7.52 (m, 3H, aromatic+CH), 7.40 (d, J = 7.5 Hz, 1H, aromatic), 7.26 (t, J = 7.5 Hz, 1H, aromatic) 2.28 (s, 3H, CH₃). ¹³C NMR (DMSO-d₆) δ 195.50 (C=O), 159.71 (C-O), 144.39, 138.28, 136.41, 133.73, 130.83, 130.80, 128.81, 125.07, 122.10, 119.10, 115.72 (CH), 15.57. IR (pellet cm⁻¹) 3449 (OH), 2993 (aromatic), 1687 (C=O), 1640 (C-O).

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