An Efficient One-Pot Three-Component Synthesis of Tetrahydrofuro[3,4-b]quinoline-1,8(3H,4H)-dione Derivatives Catalyzed by L-Proline
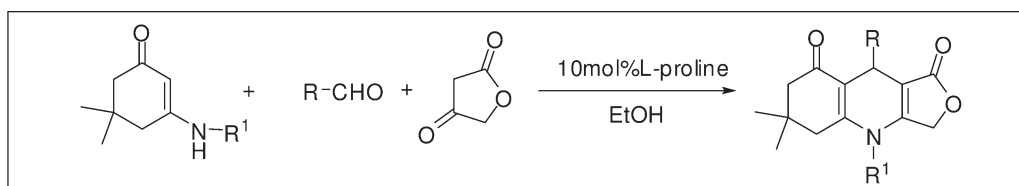
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L-Proline is found to be an efficient catalyst for the synthesis of tetrahydrofuro[3,4-b]quinoline-1,8(3H,4H)-dione derivatives. This protocol is novel and has the advantages of mild condition, high yield, and easy operation.
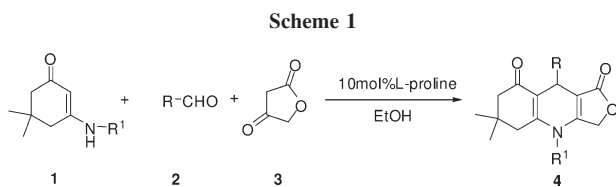
INTRODUCTION

The replacement of current chemical process with more environmentally benign alternatives is an increasingly attractive goal in organic synthesis. Recently, organic reaction catalyzed by small organic molecules has been studied more and more extensively. A lot of small organic molecules such as cinchona alkaloids, L-proline, and its derivatives have been used in various transformations with excellent yields [1–3]. Direct catalytic asymmetric aldol [4,5], Mannich [6,7], Michael [8,9], Diels-Alder [10,11], α-amination reactions, and Knoevenagel-type reaction [12] using L-proline as a catalyst have also been reported. Recently, L-proline and its derivatives have been used in multicomponent unsymmetrical Biginelli [13,14] and Hantzsch reactions[15]. More recently, we have reported the synthesis of furo[3-,4′:5,6]pyrido[2,3-c]pyrazole derivatives catalyzed by organocatalysts [16] and 3,3′-benzylidenebis[4-hydroxy-6-methylpyridin-2(1H)-one] derivatives catalyzed by L-proline [17].

1,4-Dihydropyridine compounds are well known as calcium channel modulators and have emerged as one of the most important classes of drugs for the treatment of cardiovascular diseases [18,19]. 1,4-Dihydropyridine derivatives possess a variety of biological activities such as vasodilator, bronchodilator, antitumor, and antidiabetic activity [20–23]. Extensive studies have revealed that these compounds exhibit various medicinal functions such as neuroprotectant, platelet antiaggregatory activity, cerebral antischemic activity in the treatment of Alzheimer's disease, and chemosensitizer in tumor therapy [24–27]. These examples clearly indicate the remarkable potential of novel dihydropyridine derivatives as a source of valuable drug candidates. Among these compounds, furo[3,4-b]quinoline-1,8(3H,4H)-dione (podophyllotoxin) derivatives, which inhibits microtubule assembly as an antitumor ligand, have attracted attention in recent decade [28–30]. Extensive structural modifications have been performed to obtain more potent and less toxic anticancer agents [31–33]. For example, Takeya and coworkers [34,35] reported the synthesis of aza-podophyllotoxin *via* condensation, cyclization, and reduction. This method was less efficient. Tratrat *et al*. [36] also reported the synthesis of aza-podophyllotoxin by one-pot reaction of aldehyde, tetronic acid, and aniline in refluxing ethanol with the limitation that aniline must be substituted in the metaposition by electron-donating groups. Tu *et al*. [37] also reported a three-component reaction for one-pot synthesis of 4-aza-podophyllotoxin derivatives in water under microwave irradiation conditions, with the wide applicable scope of aldehydes and anilines. The main drawback of this method is the need of a special reaction instrument and high temperature.

RESULTS AND DISCUSSION

As a consequence of our interest in organic synthesis catalyzed by L-proline, we describe a mild and highly efficient protocol for the synthesis of tetrahydrofuro[3,4-

Scheme 1

b]quinoline-1,8(3*H*,4*H*)-dione derivatives *via* three-component reaction of 5,5-dimethyl-3-aminocyclohex-2-enone **1**, aldehyde **2**, and tetronic acid **3** in EtOH (2 mL) catalyzed by 10 mol % L-proline (Scheme 1).

First, the synthesis of compound **4d** was chosen as a model to find an appropriate solvent. The 10 mol % L-proline catalyzed reaction of 3-(4-fluorophenylamino)-5,5-dimethylcyclohex-2-enone **1d**, 4-methoxybenzaldehyde **2d**, and tetronic acid **3** was examined using EtOH, DMF, CH_3CN, HOAc, $CHCl_3$, and H_2O as solvents, respectively. The results are summarized in Table 1. It can be seen from Table 1 that the reaction using EtOH as solvent resulted in the excellent yield and shortest time. Therefore, EtOH was chosen as the solvent of this reaction.

To optimize the reaction temperature, the synthesis of compound **4d** was carried out in EtOH at r.t, 40, 60, and 80°C, respectively. The results are shown in Table 2. We found that the reaction time was shortest and yield highest at 80°C. Therefore, the most reaction temperature should be 80°C.

The catalytic efficiency of other organocatalysts in this reaction was also studied. In all cases, 10 mol % of the catalyst was used, and the reaction was carried out in refluxing ethanol. As shown in Table 3, the catalytic effects of **5a**, **5b**, and **5c** were similar, whereas **5d**, **5e**, and **5f** gave lower yields. The presence of a secondary nitrogen and carboxylic acid at 2-position in the catalysts led to good yields of products, whereas DL-phenylglycine (**5d**) with a primary amino group, and (+)-cinchonine (**5e**), cinchonidine (**5f**) with a tertiary amino group, respectively, gave lower yields. These results indicate that the presence of secondary nitrogen may be essential for better catalytic activity. Replacing CH_2 (**5a**) by S (**5b**) and with CHOH (**5c**) did not affect yields

Table 2

Temperature optimization for synthesis of 4d in EtOH catalyzed by 10 mol % L-proline.

Entries	Reaction temperature (°C)	Reaction time (h)	Isolated yield (%)
1	r.t.	5	30
2	40	4	59
3	60	3	72
4	80	1.5	95

significantly. Therefore, it was concluded that L-proline was the best among the six catalysts in this study.

To optimize the catalyst loading, 5, 10, 15, and 20 mol % of L-proline was tested, respectively. The results are summarized in Table 4. A 10-mol % loading of L-proline was sufficient to push the reaction forward, and 5 mol % of L-proline was not enough. Higher amounts of L-proline did not lead to significant change in the reaction yields.

Under these optimized reaction conditions (2 mL EtOH, 80°C, 10 mol % L-proline), a series of tetrahydrofuro[3,4-*b*]quinoline-1,8(3*H*,4*H*)-dione derivatives (**4**) were synthesized. The results are summarized in Table 5.

As shown in Table 5, this protocol can be applied not only to the aromatic aldehydes with either electron-withdrawing groups (such as nitro and halide groups) or electron-donating groups (such as hydroxyl and alkoxyl groups) but also to aliphatic aldehydes under the same conditions. The substituted groups on nitrogen atom may be aromatic and aliphatic. Therefore, this reaction can be applied in a wide range.
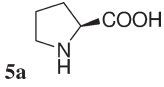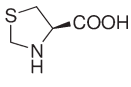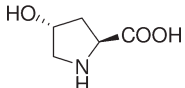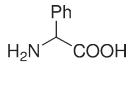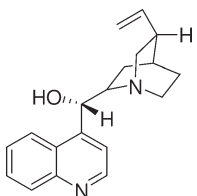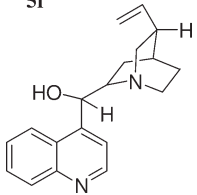
In conclusion, we have developed a novel three-component reaction of 5,5-dimethyl-3-aminocyclohex-2-enone, aldehyde, and tetronic acid in EtOH (2 mL) catalyzed by 10 mol % L-proline for the synthesis of tetrahydrofuro[3,4-*b*]quinoline-1,8(3*H*,4*H*)-dione derivatives. This new method has the advantages of good yields, convenient procedure and the use of environmentally friendly catalyst and solvent.

EXPERIMENTAL

Melting points are uncorrected. Infrared spectra were recorded on a Varian 1000 FTIR spectrometer in KBr with absorption in cm^{-1}. ^{1}H NMR spectra were recorded on a Varian Invoa-400 MHz superconductive NMR spectrometer as DMSO-d_6 solution. *J* values are in Hz. Chemical shifts are expressed in δ downfield from internal tetramethylsilane. Mass spectra were obtained using TOF-MS instrument.

Typical procedure for the synthesis of tetrahydrofuro[3,4-*b*]quinoline-1,8(3H,4H)-diones 4. A mixture of 5,5-dimethyl-3-aminocyclohex-2-enone **1** (1 mmol), aldehyde **2** (1 mmol), and tetronic acid **3** (2 mmol) was suspended in EtOH (2 mL) using 10 mol % L-proline (0.0115 g) as catalyst and

Table 1

Solvent optimization for the synthesis of 4d catalyzed by 10 mol % L-proline.

Entries	Solvents	Temperature (°C)	Time (h)	Yield (%)
1	EtOH	80	1.5	95
2	DMF	100	1.5	78
3	CH_3CN	70	3	79
4	HOAc	100	2.5	78
5	$CHCl_3$	65	2.5	82
6	H_2O	100	3	60

Table 3

Catalytic efficiency of different organocatalysts over the reaction synthesizing 4d[a]

Entries	Catalysts	Reaction time (h)	Isolated yield (%)
1	None	2	68
2	5a	1.5	95
3	5b 5c	1.5	90
4		1.5	87
5	5d 5e	2	63
6	5f	2	51
7		2	49

[a] Reaction conditions: 3-(4-fluorophenylamino)-5,5-dimethylcyclo-hex-2-enone (1 mmol), 4-methoxybenzaldehyde (1 mmol), and tetronic acid (1 mmol), catalyst 10 mol %, EtOH, 80°C

stirred at 80°C for 1.5–3 h. The reaction was monitored by TLC. After completion of the reaction, the reaction mixture was allowed to cool to room temperature and was poured into the water. The precipitate was collected by suction and recrystallized from ethanol to give products **4** with high purity.

Spectroscopy data. *4-(4-Fluorophenyl)-6,6-dimethyl-9-p-tolyl-5,6,7,9-tetrahydrofuro[3,4-b]quinoline-1,8(3H,4H)-dione (4a).* M.p. 281–283°C; ^{1}H NMR (DMSO-d_6, δ, 400 ppm): 0.86 (3H, s, CH$_3$), 0.94 (3H, s, CH$_3$), 1.99–2.08 (2H, m, CH$_2$),

Table 4

The effect about amounts of ʟ-proline for the synthesis of 4d in ethanol at 80°C.

Entries	Amounts of ʟ-proline (mol %)	Time (h)	Yield (%)
1	0	3	61
2	5	2	83
3	10	1.5	95
4	15	1.5	95
5	20	1.5	96

2.18–2.23 (2H, m, CH$_2$), 2.26 (3H, s, CH$_3$), 4.51 (1H, d, *J* = 16.4 Hz, CH$_2$), 4.57 (1H, d, *J* = 16.4 Hz, CH$_2$), 4.73 (1H, s, CH), 7.09 (2H, d, *J* = 7.6 Hz, ArH), 7.22 (2H, d, *J* = 7.6 Hz, ArH), 7.44 (2H, t, *J* = 8.0 Hz, ArH), 7.62–7.65 (2H, m, ArH); IR (KBr, ν, cm^{-1}): 3070, 2981, 2880, 1751, 1687, 1578, 1510, 1366, 1223; HRMS [Found: *m/z* 417.1738 (M$^+$), Calcd for C$_{26}$H$_{24}$NO$_3$F: M, 417.1740].

9-(4-Chlorophenyl)-4-(4-fluorophenyl)-6,6-dimethyl-5,6,7,9-tetrahydrofuro[3,4-b]quinoline-1,8(3H,4H)-dione (4b). M.p. 297–299°C; ^{1}H NMR (DMSO-d_6, δ, ppm): 0.85 (3H, s, CH$_3$), 0.93 (3H, s, CH$_3$), 2.03–2.10 (2H, m, CH$_2$), 2.19–2.25 (2H, m, CH$_2$), 4.52 (1H, d, *J* = 16.4Hz, CH$_2$), 4.59 (1H, d, *J* = 16.4 Hz, CH$_2$), 4.78 (1H, s, CH), 7.35 (4H, dd, J_1 = 8.4 Hz, J_2 = 15.2 Hz, ArH), 7.44 (2H, s, ArH), 7.65 (2H, s, ArH); IR (KBr, ν, cm^{-1}): 3298, 3062, 2960, 2882, 1754, 1689, 1580, 1510, 1367, 1224; HRMS [Found: *m/z* 437.1184 (M$^+$), Calcd for C$_{25}$H$_{21}$NO$_3$F^{35}Cl: M, 437.1194].

9-(4-Bromophenyl)-4-(4-fluorophenyl)-6,6-dimethyl-5,6,7,9-tetrahydrofuro[3,4-b]quinoline-1,8(3H,4H)-dione (4c). M.p. 283–285°C; ^{1}H NMR (DMSO-d_6, δ, ppm): 0.86 (3H, s, CH$_3$), 0.93 (3H, s, CH$_3$), 2.03–2.10 (2H, m, CH$_2$), 2.19–2.25 (2H, m, CH$_2$), 4.52 (1H, d, *J* = 16.4 Hz, CH$_2$), 4.59 (1H, d, *J* = 16.4 Hz, CH$_2$), 4.76 (1H, s, CH), 7.31 (2H, d, *J* = 8.4 Hz, ArH), 7.44–7.48 (4H, m, ArH), 7.66 (2H, s, ArH); IR (KBr, ν, cm^{-1}): 3081, 2960, 2881, 1755, 1690, 1578, 1510, 1367, 1224; HRMS [Found: *m/z* 481.0692 (M$^+$), Calcd for C$_{25}$H$_{21}$NO$_3$F^{79}Br: M, 481.0689].

Table 5

Synthesis of *N*-substituted furo[3,4-*b*]quinoline derivatives catalyzed by 10 mol% ʟ-proline in ethanol at 80°C.

Products	R^1	R	Time (h)	Yield (%)
4a	4-FC$_6$H$_4$	4-CH$_3$C$_6$H$_4$	2	92
4b	4-FC$_6$H$_4$	4-ClC$_6$H$_4$	2	91
4c	4-FC$_6$H$_4$	4-BrC$_6$H$_4$	1.5	93
4d	4-FC$_6$H$_4$	4-CH$_3$OC$_6$H$_4$	1.5	95
4e	4-CH$_3$C$_6$H$_4$	3,4,5-(CH$_3$O)$_3$C$_6$H$_2$	3	91
4f	4-CH$_3$C$_6$H$_4$	4-CH$_3$C$_6$H$_4$	1.5	95
4g	4-CH$_3$C$_6$H$_4$	4-CH$_3$OC$_6$H$_4$	2	94
4h	4-CH$_3$C$_6$H$_4$	*n*-Propyl	3	91
4i	3-Cl-4-CH$_3$C$_6$H$_3$	4-BrC$_6$H$_4$	2	94
4j	*n*-Butyl	4-HOC$_6$H$_4$	3	92
4k	*n*-Butyl	4-BrC$_6$H$_4$	2.5	93

4-(4-Fluorophenyl)-9-(4-methoxyphenyl)-6,6-dimethyl-5,6,7,9-tetrahydrofuro[3,4-b]quinoline-1,8(3H,4H)-dione (4d). M.p. 266–268°C; ^{1}H NMR (DMSO-d_6, δ, ppm): 0.86 (3H, s, CH$_3$), 0.93 (3H, s, CH$_3$), 2.01–2.05 (2H, m, CH$_2$), 2.19–2.25 (2H, m, CH$_2$), 3.72 (3H, s, OCH$_3$), 4.51 (1H, d, J = 15.6 Hz, CH$_2$), 4.58 (1H, d, J = 16.0 Hz, CH$_2$), 4.72 (1H, s, CH), 6.84 (2H, d, J = 8.4 Hz, ArH), 7.25 (2H, d, J = 8.4 Hz, ArH), 7.42–7.46 (2H, m, ArH), 7.62–7.65 (2H, m, ArH); IR (KBr, ν, cm^{-1}): 3070, 2962, 2888, 1751, 1685, 1578, 1510, 1365, 1223; HRMS [Found: m/z 433.1708 (M$^+$), Calcd for C$_{26}$H$_{24}$NO$_4$F: M, 433.1689].

6,6-Dimethyl-4-p-tolyl-9-(3,4,5-trimethoxyphenyl)-5,6,7,9-tetrahydrofuro[3,4-b]quinoline-1,8(3H,4H)-dione (4e). M.p. 221–223°C; ^{1}H NMR (DMSO-d_6, δ, ppm): 0.93 (6H, d, J = 4.8 Hz, 2 × CH$_3$), 2.02–2.13 (2H, m, CH$_2$), 2.23–2.28 (2H, m, CH$_2$), 2.40 (3H, s, CH$_3$), 3.62 (3H, s, OCH$_3$), 3.77 (6H, s, 2 × OCH$_3$), 4.50 (1H, d, J = 16.4 Hz, CH$_2$), 4.55 (1H, d, J = 16.4 Hz, CH$_2$), 4.72 (1H, s, CH), 6.56 (2H, s, ArH), 7.40 (4H, s, ArH); IR (KBr, ν, cm^{-1}): 2962, 1750, 1679, 1589, 1511, 1422, 1367, 1227, 1124, 1001; HRMS [Found: m/z 489.2156 (M$^+$), Calcd for C$_{29}$H$_{31}$NO$_6$: M, 489.2151].

6,6-Dimethyl-4,9-dip-tolyl-5,6,7,9-tetrahydrofuro[3, 4-b]quinoline-1,8(3H,4H)-dione (4f). M.p. 263–264°C; ^{1}H NMR (DMSO-d_6, δ, ppm): 0.83 (3H, s, CH$_3$), 0.91 (3H, s, CH$_3$), 2.00–2.06 (2H, m, CH$_2$), 2.18–2.22 (2H, m, CH$_2$), 2.25 (3H, s, CH$_3$), 2.40 (3H, s, CH$_3$), 4.48 (1H, d, J = 16.0 Hz, CH$_2$), 4.56 (1H, d, J = 15.6 Hz, CH$_2$), 4.71 (1H, s, CH), 7.08 (2H, d, J = 8.0 Hz, ArH), 7.20 (2H, d, J = 8.0 Hz, ArH), 7.40 (4H, d, J = 6.0 Hz, ArH); IR (KBr, ν, cm^{-1}): 2957, 2932, 1754, 1682, 1577, 1510, 1369, 1274; HRMS [Found: m/z 413.2010 (M$^+$), Calcd for C$_{27}$H$_{27}$NO$_3$: M, 413.1991].

9-(4-Methoxyphenyl)-6,6-dimethyl-4-p-tolyl-5,6,7,9-tetrahydrofuro[3,4-b]quinoline-1,8(3H,4H)-dione (4g). M.p. 254–256°C (Lit 256–257°C[38]); ^{1}H NMR (DMSO-d_6, δ, ppm): 0.83 (3H, s, CH$_3$), 0.91 (3H, s, CH$_3$), 1.99–2.06 (2H, m, CH$_2$), 2.18–2.25 (2H, m, CH$_2$), 2.40 (3H, s, CH$_3$), 3.71 (3H, s, OCH$_3$), 4.48 (1H, d, J = 16.4 Hz, CH$_2$), 4.56 (1H, d, J = 16.0 Hz, CH$_2$), 4.70 (1H, s, CH), 6.84 (2H, d, J = 8.8 Hz, ArH), 7.23 (2H, d, J = 8.4 Hz, ArH), 7.40 (4H, d, J = 6.0 Hz, ArH); IR (KBr, ν, cm^{-1}): 2956, 2937, 1753, 1681, 1579, 1509, 1371, 1244, 1026; HRMS [Found: m/z 429.1926 (M$^+$), Calcd for C$_{27}$H$_{27}$NO$_4$: M, 429.1940].

6,6-Dimethyl-9-propyl-4-p-tolyl-5,6,7,9-tetrahydrofuro[3,4-b]-quinoline-1,8(3H,4H)-dione (4h). M.p. 213–215°C; ^{1}H NMR (DMSO-d_6, δ, ppm): 0.87 (3H, t, J = 7.6 Hz, CH$_3$), 0.93 (6H, d, J = 6.0Hz, 2 × CH$_3$), 1.26–1.33 (2H, m, CH$_2$), 1.43–1.55 (2H, m, CH$_2$), 1.92 (1H, d, J = 17.6 Hz, CH$_2$), 2.15 (1H, d, J = 17.2 Hz, CH$_2$), 2.19–1.29 (2H, m, CH$_2$), 2.39 (3H, s, CH$_3$), 3.77 (1H, t, J = 4.4 Hz, CH), 4.43 (1H, d, J = 16.4 Hz, CH$_2$), 4.52 (1H, d, J = 16.0 Hz, CH$_2$), 7.27–7.29 (1H, m, ArH), 7.36 (3H, d, J = 8.0 Hz, ArH); IR (KBr, ν, cm^{-1}): 3256, 2982, 2869, 1745, 1679, 1575, 1511, 1422, 1373, 1232; HRMS [Found: m/z 365.1973 (M$^+$), Calcd for C$_{23}$H$_{27}$NO$_3$: M, 365.1991].

9-(4-Bromophenyl)-4-(3-chloro-4-methylphenyl)-6,6-dimethyl-5,6,7,9-tetrahydrofuro[3,4-b]quinoline-1,8(3H,4H)-dione (4i). M.p. 272–274°C; ^{1}H NMR (DMSO-d_6, δ, ppm): 0.85 (3H, s, CH$_3$), 0.93 (3H, s, CH$_3$), 2.01–2.11 (2H, m, CH$_2$), 2.17–2.30 (2H, m, CH$_2$), 2.41 (3H, s, CH$_3$), 4.51–4.57 (1H, m, CH$_2$), 4.65 (1H, d, J = 16.4 Hz, CH$_2$), 4.73 (1H, s, CH), 7.31 (2H, dd, J_1 = 8.4 Hz, J_2 = 15.6 Hz, ArH), 7.43–7.48 (3H, m, ArH), 7.52–7.60 (1H, m, ArH), 7.73–7.83 (1H, m, ArH); IR (KBr, ν, cm^{-1}): 2959, 2933, 1749, 1683, 1583, 1494, 1366, 1270, 1194; HRMS [Found: m/z 511.0560 (M$^+$), Calcd for C$_{26}$H$_{23}$NO$_3$^{35}Cl^{79}Br: M, 511.0550].

4-Butyl-9-(4-hydroxyphenyl)-6,6-dimethyl-5,6,7,9-tetrahydrofuro[3,4-b]quinoline-1,8(3H,4H)-dione (4j). M.p. 237–238°C; ^{1}H NMR (DMSO-d_6, δ, ppm): 0.94–0.97 (6H, m, 2 × CH$_3$), 1.06 (3H, s, CH$_3$), 1.33–1.39 (2H, m, CH$_2$), 1.61–1.64 (2H, m, CH$_2$), 2.07 (1H, d, J = 16.0 Hz, CH$_2$), 2.21 (1H, d, J = 16.4 Hz, CH$_2$), 2.58 (1H, d, J = 17.6 Hz, CH$_2$), 2.66 (1H, d, J = 17.6 Hz, CH$_2$), 3.40–3.44 (1H, m, CH$_2$), 3.61–3.68 (1H, m, CH$_2$), 4.58 (1H, s, CH), 5.01 (1H, d, J = 16.0 Hz, CH$_2$), 5.09 (1H, d, J = 15.6 Hz, CH$_2$), 6.60 (2H, d, J = 8.0 Hz, ArH), 6.93 (2H, d, J = 8.4 Hz, ArH), 9.11 (1H, br., s, OH); IR (KBr, ν, cm^{-1}): 3473, 3158, 2955, 1750, 1686, 1583, 1543, 1511, 1428, 1392, 1220; HRMS [Found: m/z 381.1949 (M$^+$), Calcd for C$_{23}$H$_{27}$NO$_4$: M, 381.1940].

9-(4-Bromophenyl)-4-butyl-6,6-dimethyl-5,6,7,9-tetrahydrofuro[3,4-b]quinoline-1,8(3H,4H)-dione (4k). M.p. 249–250°C; ^{1}H NMR (DMSO-d_6, δ, ppm): 0.94–0.97 (6H, m, 2 × CH$_3$), 1.07 (3H, s, CH$_3$), 1.31–1.40 (2H, m, CH$_2$), 1.59–1.69 (2H, m, CH$_2$), 2.08 (1H, d, J = 16.0 Hz, CH$_2$), 2.22 (1H, d, J = 15.6 Hz, CH$_2$), 2.61 (1H, d, J = 17.6 Hz, CH$_2$), 2.68 (1H, d, J = 17.2 Hz, CH$_2$), 3.38–3.47 (1H, m, CH$_2$), 3.59–3.67 (1H, m, CH$_2$), 4.67 (1H, s, CH), 5.05 (1H, d, J = 16.0 Hz, CH$_2$), 5.11 (1H, d, J = 16.4 Hz, CH$_2$), 7.11 (2H, d, J = 8.4 Hz, ArH), 7.43 (2H, d, J = 8.4 Hz, ArH); IR (KBr, ν, cm^{-1}): 2956, 1755, 1675, 1643, 1568, 1429, 1368, 1219, 1147; HRMS [Found: m/z 443.1094 (M$^+$), Calcd for C$_{23}$H$_{26}$NO$_3$^{79}Br: M, 443.1096].

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