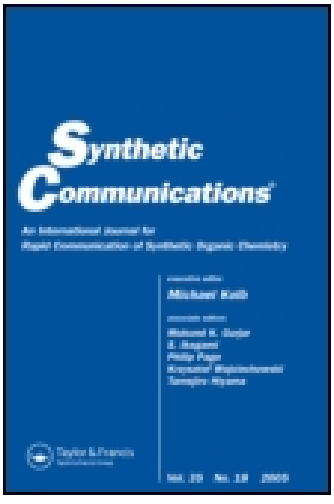

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Synthesis of 3-Aryl-2-benzoylbenzofuran Derivatives using Manganese(III) Acetate-Mediated Addition of Dimedon to Chalcone Derivatives

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Synthesis of 3-Aryl-2-benzoylbenzofuran Derivatives using Manganese(III) Acetate–Mediated Addition of Dimedon to Chalcone Derivatives

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Abstract: 3-Aryl-2-benzoylbenzofurans were synthesized by the reaction of α-carboradical produced from dimedon by oxidizing with manganese(III) acetate in acetic acid and the chalcone derivatives.

Keywords: 3-Aryl-2-benzoylbenzofurans, chalcones, dimedon, manganese(III) acetate

Furans and their derivatives are widely present in nature[1] and are some of the most important heterocyclic compounds[2] for the total synthesis of the complicated naturally occurring metabolites in organic chemistry.[3] Furthermore, polyfunctionalized furans are versatile and convenient synthetic starting materials for preparation of a variety of heterocyclic compounds.[4]

It is well known that C-C bonds are formed during the transition-metal-salt-mediated oxidative addition of 1,3-dicarbonyl compounds to unsaturated systems. In particular, $Mn(OAc)_3$ is effectively used in the synthesis of furans[5] and dihydrofurans.[6] Manganese(III) acetate acts as a one-electron oxidant and is used to form radicals at the α-position of carbonyls and

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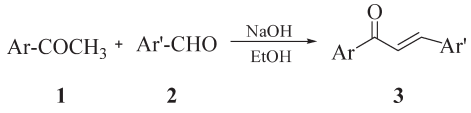


1 **2** **3**

Scheme 1.

particularly 1,3-dicarbonyls, which can react with electron-rich alkenes to form new C-C bonds.[7] $Mn(OAc)_3$-prompted addition of 1,3-dicarbonyl compounds to alkenes,[8] steric hindered alkenes,[9] bicyclic alkenes,[10] alkines,[11] imines,[12] and α,β-unsaturated amides[13] have been reported. In this study, we report the $Mn(OAc)_3$-mediated addition of dimedon as a 1,3-dicarbonyl compound to chalcone derivatives, which are electron-poor alkenes.

The chalcone derivatives **3a–i**, the starting material for the synthesis of **5a–i**, were synthesized by the condensation of corresponding benzaldhyde and acetophenone derivatives in basic medium (Scheme 1). All chalcone derivatives **3a–i** are well known in the literature.[14–16]

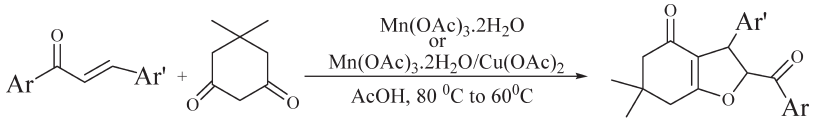
The reaction of dimedon with chalcones **3a–i** having different functional groups in the presence of $Mn(OAc)_3$ or $Mn(OAc)_3/Cu(OAc)_2$ mixture resulted in the formation of 3-aryl-2-benzoylbezofuran derivatives **5a–i** via oxidative free-radical cyclization (Scheme 2). As shown in Table 1, dimedon did not react with the amino-chalcone **5f** as it react with the other chalcone derivatives with lower yields in the presence of the $Mn(OAc)_3/Cu(OAc)_2$ mixture. The experimental results are given in Table 1.

This reaction formed only the desired heterocyclic compounds in one step, and no other by-products were isolated. Accordingly, we suggest that the present reaction is a convenient synthetic method of preparing 3-aryl-2-benzoylbenzofurans.

EXPERIMENTAL

Instruments

Melting points were measured on an Electrothermal 9100 apparatus. IR spectra (KBr disc or $CHCl_3$) were recorded on a Jasco FT/IR-430



3 **4** **5**

Scheme 2.

Table 1. Synthesis of 3-aryl-2-benzoylbenzofuran derivatives by Mn(OAc)3- or Mn(OAc)3/Cu(OAc)2- promoted oxidative addition of dimedon to chalcone derivatives

			Time (min)			Isolated yield (%)		
Entry	Ar	Ar[a]	A[a]	B[b]	Products	A′	B[b]	Mp (°C)
1	4-ClPh	Ph	10	15	**5a**	45	37	129–131
2	4-BrPh	Ph	20	25	**5b**	47	39	127–129
3	4-OHPh	Ph	15	15	**5c**	41	38	187–190
4	4-CH_3Ph	Ph	30	45	**5d**	66	60	—
5	4-CH_3OPh	Ph	15	15	**5e**	72	68	—
6	4-NH_2Ph	Ph	15	—	**5f**	62	—	—
7	Ph	4-CH_3OPh	15	5	**5g**	59	45	109–111
8	Ph	4-ClPh	5	15	**5h**	57	44	117–119
9	Ph	4-CH_3Ph	5	15	**5i**	45	36	113–115

[a]Method A: $Mn(OAc)_3$ (2 eq).
[b]Method B: $Mn(OAc)_3$ (2 eq), $Cu(OAc)_2$ (2eq).

spectrometer. ^1H and ^{13}C NMR spectra were recorded on a Varian EM 360 L instrument. TMS (δ 0.00) was the internal standard for ^1H NMR and $CDCl_3$ (δ 77.0) for ^{13}C NMR spectroscopy; *J* values are given in hertz. The multiplicities of the signals in the ^1H NMR spectra are abbreviated by s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), br (broad), and combinations thereof. Elemental analyses were obtained from a Leco CHNS 932 elemental analyzer.

Reagent

Manganese(III) acetate, copper(II) acetate, acetic acid, and dimedon were used as commercial products of the highest reagent grade.

All column chromatographies were performed on silica gel (60–230 mesh, Merck).

General Procedure for Synthesis of 3-Aryl-2-benzoylbenzofurans

A solution of manganese(III) acetate (1.9 g, 7.2 mmol) in 15 mL of glacial acetic acid was heated under a nitrogen atmosphere at 80 °C until a it dissolved. After $Mn(OAc)_3$ was dissolved completely, the solution was cooled down to 60 °C. A solution of dimedon (1 g, 7.2 mmol) and chalcone derivative (1 g, 3.6 mmol) in 5 mL of acetic acid or dimedon (1 g, 7.2 mmol),

chalcone derivative (1 g, 3.6 mmol), and $Cu(OAc)_2$ (1.43 g, 7.2 mmol) in 10 mL of acetic acid was added to this mixture. The reaction was finished when the dark brown color of the solution disappeared. Acetic acid was evaporated under reduced pressure. The water was added to the residue and extracted with EtOAc (3 × 20 mL). The combined organic phases were neutralized with saturated $NaHCO_3$ solution, dried over anhydrous Na_2SO_4, and evaporated. Crude products were purified by column chromatography on silica gel or preparative thin-layer chromatography (TLC) (20 × 20 cm plates, 2 mm thick) using *n*-hexane/EtOAc (9:1) as eluent. The products were crystallized in *n*-hexane/EtOAc (9:1).

Data

2-(4-Chlorobenzoyl)-6,6-dimethyl-3-phenyl-2,3,6,7-tetrahydrobenzofuran-4(5H)-one (**5a**)

Colorless crystals; mp 129–131 °C; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 8.01$–7.96 (brd, 2H, $J = 8.7$ Hz, ArH, AA′ part of AA′BB′ system), 7.44–7.38 (m, 5H, ArH, BB′ part of AA′BB′ system and 3H), 7.29–7.25 (m, 2H, ArH), 5.94–5.92 (d, 1H, $J = 5.8$ Hz, H2, A part of AB system), 4.99–4.97 (ddd as "dt", 1H, $J_{3,2} = 5.8$, $J_{3,7} = 1.3$ Hz, H3, B part AB system), 2.58–2.52 (dd, 1H, $J_{7a,b} = 17.6$, $J_{7a,3} = 0.9$ Hz, H7a), 2.51–2.44 (dd, 1H, $J_{7b,a} = 17.6$, $J_{7b,3} = 1.7$ Hz, H7b), 2.34–2.28 (d, 1H, $J_{5a,b} = 16.2$ Hz, H5a), 2.27–2.22 (d, 1H, $J_{5b,a} = 16.2$ Hz, H5b), 1.20 (s, 3H, CH_3), 1.19 (s, 3H, CH_3) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 198.04$, 193.54, 177.44, 140.27, 139.41, 134.54, 130.65, 129.15, 128.85, 125.69, 112.63, 90.03, 54.63, 50.95, 37.92, 34.46, 28.63, 28.61; IR (KBr disc) cm^{-1}: 3062, 3031, 2958, 2871, 1681, 1637, 1589, 1398, 1213, 1091, 1002, 794, 759, 698. Anal. calcd. for $C_{23}H_{21}ClO_3$: C, 72.53; H, 5.56. Found: C, 72.77; H, 5.61.

2-(4-Bromobenzoyl)-6,6-dimethyl-3-phenyl-2,3,6,7-tetrahydrobenzofuran-4(5H)-one (**5b**)

Colorless crystals; mp 127–129 °C; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 7.93$–7.88 (d, 2H, $J = 8.6$ Hz, ArH, AA′ part of AA′BB′ system), 7.62–7.57 (d, 2H, $J = 8.6$ Hz, ArH, BB′ part of AA′BB′ system), 7.45–7.37 (m, 3H, ArH), 7.39–7.25 (m, 2H, ArH), 5.94–5.92 (d, 1H, $J = 5.8$ Hz, H2, A part of AB system), 4.98–4.96 (brd, 1H, $J = 5.8$ Hz, H3, B part AB system), 2.58–2.52 (dd, 1H, $J_{7a,b} = 17.7$, $J_{7a,3} = 1.1$ Hz, H7a), 2.51–2.44 (dd, 1H, $J_{7b,a} = 17.7$, $J_{7b,3} = 1.7$ Hz, H7b), 2.34–2.28 (d, 1H, $J_{5a,b} = 16.2$ Hz, H5a), 2.27–2.21 (d, 1H, $J_{5b,a} = 16.2$ Hz, H5b), 1.20 (s, 3H, CH_3), 1.19 (s, 3H, CH_3) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 198.26$, 193.54, 177.44, 139.39, 135.00 131.85, 130.73, 129.16, 125.68, 112.61, 90.02, 54.62, 50.95, 37.93, 34.46, 28.63, 28.61; IR (KBr disc) cm^{-1}: 3062, 3035, 2958, 2927,

2857, 1679, 1637, 1583, 1396, 1211, 1070, 1001, 757, 698. Anal. calcd. for $C_{23}H_{21}BrO_3$: C, 64.95; H, 4.98. Found: C, 64.96; H, 5.02.

2-(4-Hydroxybenzoyl)-6,6-dimethyl-3-phenyl-2,3,6,7-tetrahydrobenzofuran-4(5H)-one (**5c**)

Colorless crystals; mp 187–190 °C; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 9.21–8.67$ (brs, 1H, OH), 7.81–7.78 (brd, 2H, $J = 8.8$ Hz, ArH, AA′ part of AA′XX′ system), 7.45–7.36 (m, 3H, ArH), 7.30–7.25 (m, 2H, ArH), 6.67–6.64 (brd, 2H, $J = 8.8$ Hz, XX′ part of AA′XX′ system), 5.97–5.95 (d, 1H, $J = 5.5$ Hz, H2, A part of AB system), 5.00–4.98 (d, 1H, $J = 5.5$ Hz, H3, B part AB system), 2.63–2.57 (dd, 1H, $J_{7a,b} = 16.9$, $J_{7a,3} = 0.8$ Hz, H7a), 2.54–2.48 (dd, 1H, $J_{7b,a} = 16.9$, $J_{7b,3} = 1.3$ Hz, H7b), 2.42–2.37 (d, 1H, $J_{5a,b} = 16.4$ Hz, H5a), 2.34–2.29 (d, 1H, $J_{5b,a} = 16.4$ Hz, H5b), 1.23 (s, 3H, CH_3), 1.21 (s, 3H, CH_3) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 196.99$, 195.70, 179.88, 162.35, 139.33, 131.71, 129.13, 129.08, 127.96, 125.67, 115.51, 113.06, 90.93, 53.45, 50.77, 38.03, 34.67, 28.58 (2C); IR (KBr disc) cm^{-1}: 3249, 3066, 3031, 2960, 2890, 2815, 1617, 1602, 1579, 1400, 1284, 1226, 1170, 759, 736, 700. Anal. calcd. for $C_{23}H_{22}O_4$: C, 76.22; H, 6.12. Found: C, 76.67; H, 6.51.

6,6-Dimethyl-2-(4-methylbenzoyl)-3-phenyl-2,3,6,7-tetrahydrobenzofuran-4(5H)-one (**5d**)

Viscous oil; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 7.94–7.91$ (d, 2H, $J = 8.2$ Hz, ArH, AA′ part of AA′BB′ system), 7.42–7.38 (m, 2H, ArH), 7.31–7.24 (m, 5H, BB′ part of AA′BB′ system and 3H), 5.91–5.89 (d, 1H, $J = 5.6$ Hz, H2, A part of AB system), 5.04–5.02 (d, 1H, $J = 5.6$ Hz, H3, B part AB system), 2.58–2.52 (dd, 1H, $J_{7a,b} = 17.6$, $J_{7a,3} = 0.9$ Hz, H7a), 2.51–2.44 (dd, 1H, $J_{7b,a} = 17.6$, $J_{7b,3} = 1.7$ Hz, H7b), 2.34–2.28 (d, 1H, $J_{5a,b} = 16.2$ Hz, H5a), 2.27–2.22 (d, 1H, $J_{5b,a} = 16.2$ Hz, H5b), 1.22 (s, 3H, CH_3), 1.19 (s, 3H, CH_3) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 198.47$, 193.63, 177.31, 144.64, 139.64 133.71, 129.37, 129.26, 129.08, 129.02, 125.77, 112.78, 90.10, 54.44, 50.98, 37.93, 34.46, 29.73, 28.67, 28.38; IR ($CHCl_3$) cm^{-1}: 3031, 2956, 2925, 2854, 1677, 1643, 1606, 1455, 1396, 1226, 1182, 757, 698. Anal. calcd. for $C_{24}H_{24}O_3$: C, 79.97; H, 6.71. Found: C, 79.96; H, 6.83.

2-(4-Methoxybenzoyl)-6,6-dimethyl-3-phenyl-2,3,6,7-tetrahydrobenzofuran-4(5H)-one (**5e**)

Viscous oil; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 8.04–7.98$ (brd, 2H, $J = 8.9$ Hz, ArH, AA′ part of AA′XX′ system), 7.44–7.37 (m, 3H, ArH), 7.31–7.26 (m, 2H, ArH), 6.93–6.90 (brd, 2H, $J = 8.9$ Hz, XX′ part of AA′XX′ system), 5.92–5.95 (d, 1H, $J = 5.6$ Hz, H2, A part of AB system),

5.01–4.99 (d, 1H, $J = 5.6$ Hz, H3, B part AB system), 3.84 (s, 3H, -OCH_3), 2.57–2.52 (dd, 1H, $J_{7a,b} = 17.1$, $J_{7a,3} = 0.8$ Hz, H7a), 2.50–2.43 (dd, 1H, $J_{7b,a} = 17.1$, $J_{7b,3} = 1.6$ Hz, H7b), 2.34–2.29 (d, 1H, $J_{5a,b} = 16.2$ Hz, H5a), 2.27–2.22 (d, 1H, $J_{5b,a} = 16.2$ Hz, H5b), 1.21 (s, 3H, CH_3), 1.18 (s, 3H, CH_3) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 197.20$, 193.67, 177.31, 164.05, 139.74, 131.67, 129.19, 129.07, 128.98, 125.74, 113.73, 112.76, 90.12, 55.48, 54.24, 51.00, 37.92, 34.44, 28.64, 28.63; IR ($CHCl_3$) cm^{-1}: 3062, 3031, 3008, 2958, 2927, 2857, 1639, 1598, 1573, 1396, 1263, 1224, 1172, 1029, 757, 700. Anal. calcd. for $C_{24}H_{24}O_4$: C, 76.57; H, 6.43. Found: C, 76.35; H, 6.45.

2-(4-Aminobenzoyl)-6,6-dimethyl-3-phenyl-2,3,6,7-tetrahydrobenzofuran-4(5H)-one (**5f**)

Yellow oil; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 7.86–7.84$ (d, 2H, $J = 8.5$ Hz, ArH, AA′ part of AA′XX′ system), 7.41–7.28 (m, 5H, ArH), 6.62–6.58 (d, 2H, $J = 8.5$ Hz, XX′ part of AA′XX′ system), 5.88–5.86 (d, 1H, $J = 5.5$ Hz, H2, A part of AB system), 4.95–4.93 (d, 1H, $J = 5.5$ Hz, H3, B part AB system), 2.58–2.52 (d, 1H, $J_{7a,b} = 17.8$, H7a), 2.49–2.43 (d, 1H, $J_{7b,a} = 17.8$ Hz, H7b), 2.35–2.30 (d, 1H, $J_{5a,b} = 16.3$ Hz, H5a), 2.27–2.22 (d, 1H, $J_{5b,a} = 16.3$ Hz, H5b), 1.21 (s, 3H, CH_3), 1.18 (s, 3H, CH_3) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 196.43$, 193.79, 177.26, 151.72, 139.89, 131.91, 129.01, 128.88, 126.51, 125.76, 113.70, 112.88, 90.21, 53.88, 51.06, 37.95, 34.45, 28.70, 28.62; IR ($CHCl_3$) cm^{-1}: 3367, 3064, 2956, 2927, 2857, 1662, 1592, 1562, 1440, 1299, 1218, 1176, 997, 784, 763. Anal. calcd. for $C_{23}H_{23}NO_3$: C, 76.43; H, 6.41; N, 3.88. Found: C, 76.77; H, 6.61; N, 4.18.

2-Benzoyl-3-(4-methoxyphenyl)-6,6-dimethyl-2,3,6,7-tetrahydrobenzofuran-4(5H)-one (**5g**)

Colorless crystals; mp 109–111 °C; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 8.01–7.97$ (dd, 2H, $J = 7.8$ Hz, ArH), 7.60–7.54 (tt, 1H, $J = 7.3$, 1.3 Hz, ArH), 7.46–7.41 (brt, 2H, $J = 7.8$ Hz, ArH), 7.26–7.22 (brd, 2H, $J = 8.6$ Hz, ArH, AA′ part of AA′BB′ system), 6.95–6.91 (brd, 2H, $J = 8.6$ Hz, BB′ part of AA′BB′ system), 5.82–5.80 (d, 1H, $J = 5.8$ Hz, H2, A part of AB system), 5.06–5.04 (d, 1H, $J = 5.8$ Hz, H3, B part AB system), 3.83 (s, 3H, -OCH_3), 2.55–2.49 (dd, 1H, $J_{7a,b} = 17.5$, $J_{7a,3} = 0.9$ Hz, H7a), 2.47–2.41 (dd, 1H, $J_{7b,a} = 17.5$, $J_{7b,3} = 1.7$ Hz, H7b), 2.36–2.30 (d, 1H, $J_{5a,b} = 16.2$ Hz, H5a), 2.28–2.23 (d, 1H, $J_{5b,a} = 16.2$ Hz, H5b), 1.22 (s, 3H, CH_3), 1.18 (s, 3H, CH_3) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 199.02$, 193.57, 177.18, 160.21, 136.26, 133.62, 131.50, 129.14, 128.55, 127.54, 114.44, 112.76, 90.12, 76.67, 55.37, 54.43, 50.96, 37.96, 34.46, 28.68, 28.61; IR (KBr disc) cm^{-1}: 3062, 3004, 2958, 2927, 2871, 1681, 1637, 1515, 1396, 1251, 1216, 1178, 1033, 794, 692. Anal. calcd. for $C_{24}H_{24}O_4$: C, 76.57; H, 6.43. Found: C, 76.35; H, 6.45.

2-Benzoyl-3-(4-chlorophenyl)-6,6-dimethyl-2,3,6,7-tetrahydrobenzofuran-4(5H)-one (**5h**)

Colorless crystals; mp 117–119 °C; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 8.02–7.99$ (dd, 2H, $J = 7.1$ Hz, ArH), 7.61–7.55 (tt, 1H, $J = 7.3$, 1.2 Hz, ArH), 7.47–7.42 (brt, 2H, $J = 7.6$ Hz, ArH), 7.24–7.21 (brd, 2H, $J = 8.3$ Hz, ArH, AA′ part of AA′BB′ system), 7.20–7.17 (brd, 2H, $J = 8.3$ Hz, BB′ part of AA′BB′ system), 5.88–5.86 (d, 1H, $J = 5.8$ Hz, H2, A part of AB system), 5.04–4.98 (d, 1H, $J = 5.8$ Hz, H3, B part AB system), 2.57–2.51 (dd, 1H, $J_{7a,b} = 17.6$, $J_{7a,3} = 0.9$ Hz, H7a), 2.50–2.43 (dd, 1H, $J_{7b,a} = 17.6$, $J_{7b,3} = 1.7$ Hz, H7b), 2.34–2.28 (d, 1H, $J_{5a,b} = 16.2$ Hz, H5a), 2.27–2.22 (d, 1H, $J_{5b,a} = 16.2$ Hz, H5b), 1.21 (s, 3H, CH_3), 1.19 (s, 3H, CH_3) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 198.82$, 193.48, 177.11, 138.05, 136.17, 134.91, 133.81, 129.31, 129.15, 128.62, 127.15, 112.77, 89.26, 54.52, 50.94, 37.88, 34.47, 28.69, 28.56; IR (KBr disc) cm^{-1}: 3062, 3031, 2958, 2871, 1681, 1643, 1596, 1492, 1394, 1216, 794. Anal. calcd. for $C_{23}H_{21}ClO_3$: C, 72.53; H, 5.56. Found: C, 72.87; H, 5.64.

2-Benzoyl-6,6-dimethyl-3-p-tolyl-2,3,6,7-tetrahydrobenzofuran-4(5H)-one (**5i**)

Colorless crystals; mp 113–115 °C; ^{1}H NMR (300 MHz, $CDCl_3$): $\delta = 8.03–7.99$ (dd, 2H, $J = 7.8$ Hz, ArH), 7.62–7.57 (tt, 1H, $J = 7.3$, 1.2 Hz, ArH), 7.48–7.44 (brt, 2H, $J = 7.8$ Hz, ArH), 7.39–7.37 (brd, 2H, $J = 8.5$ Hz, ArH, AA′ part of AA′BB′ system), 7.25–7.19 (brd, 2H, $J = 8.5$ Hz, BB′ part of AA′BB′ system), 5.85–5.83 (d, 1H, $J = 5.6$ Hz, H2, A part of AB system), 5.06–5.04 (d, 1H, $J = 5.6$ Hz, H3, B part AB system), 2.57–2.51 (dd, 1H, $J_{7a,b} = 17.5$, $J_{7a,3} = 0.8$ Hz, H7a), 2.49–2.43 (dd, 1H, $J_{7b,a} = 17.5$, $J_{7b,3} = 1.7$ Hz, H7b), 2.36–2.30 (d, 1H, $J_{5a,b} = 16.2$ Hz, H5a), 2.29–2.23 (d, 1H, $J_{5b,a} = 16.2$ Hz, H5b), 1.22 (s, 3H, CH_3), 1.19 (s, 3H, CH_3) ppm; ^{13}C NMR (75 MHz, $CDCl_3$): $\delta = 199.10$, 193.57, 177.28, 139.10, 136.54, 136.28, 133.62, 129.76, 129.17, 128.54, 125.88, 112.80, 90.17, 54.48, 50.97, 37.95, 34.46, 28.69, 28.61, 21.27; IR (KBr disc) cm^{-1}: 3056, 3027, 2958, 2929, 2883, 1681, 1641, 1596, 1394, 1216, 1049, 790, 759, 692. Anal. calcd. for $C_{24}H_{24}O_3$: C, 79.97; H, 6.71. Found: C, 79.86; H, 6.83.

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