Chemoselective Transfer Hydrogenation of $\alpha,\beta$-Unsaturated Ketones Catalyzed by Iridium Complexes

Yaping Xia
Lu Ouyang
Jianhua Liao
Xiao Yang
Renshi Luo*

School of Pharmacy, Gannan Medical University, Ganzhou, 341000, Jiangxi Province, P. R. of China
eMail luorenshi2010@163.com

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Abstract  Efficient chemoselective transfer hydrogenation of the C=C bond of $\alpha,\beta$-unsaturated ketones has been developed, using the iridium complexes containing pyridine-imidazolidinyl ligands as catalysts and formic acid as a hydrogen source. In comparison with organic solvents or $\text{H}_2\text{O}$ as solvent, the mixed solvents of $\text{H}_2\text{O}$ and $\text{MeOH}$ are critical for a high catalytic chemoselective transformation. This chemoselective transfer hydrogenation can be carried out in air, which is operationally simple, allowing a wide variety of $\alpha,\beta$-unsaturated substrates with different functional groups (electron-donating and electron-withdrawing substituents) leading to chemoselective transfer hydrogenation in excellent yields. The practical application of this protocol is demonstrated by a gram-scale transformation.

Key words  transfer hydrogenation, iridium complex, $\alpha,\beta$-unsaturated ketones, formic acid, chemoselective reduction

Saturated carbonyl compounds are ubiquitous, and many pharmaceutically active molecules contain 1,3-diaryl ketones (Figure 1).1

![Figure 1](https://example.com/image)

Figure 1  Examples of pharmaceutically active molecules containing 1,3-diaryl ketones

Among the methodologies for the synthesis of saturated carbonyl compounds, one of the best ways is the selective reduction of carbon–carbon double bonds on $\alpha,\beta$-unsaturated carbonyl compounds.2 However, chemoselective reduction of carbon–carbon double bonds of $\alpha,\beta$-unsaturated carbonyl compounds wherein the carbon–oxygen double bond is not affected,3 is a challenge with a significant role in organic synthesis.4

Traditional transformation of chemoselective reduction of C=C bonds of $\alpha,\beta$-unsaturated carbonyl compounds include hydrogenation with hydrogen over a Pd/C catalyst.5 It is well known that transition metals are not only good electron donors, but also electron acceptors due to the availability of vacant d-orbitals that possess specific electronic and spatial effects when coordinated with organic ligands.6 Therefore, much effort has been devoted to the development of highly chemoselective reduction of C=C bonds for $\alpha,\beta$-unsaturated carbonyl compounds catalyzed by transition-metal catalysts, such as Pd,7 Rh,8 Ru,9 Ni,10 Co,11 and Fe (Scheme 1a).12 At the same time, nontransition-metal hydrides such as Sn, Se, Te, B,13 and others14 have
also been employed for the selective reduction of C=C bonds in α,β-unsaturated carbonyl compounds. Furthermore, enzymic reduction is receiving increasing attention due to the potential for high chemoselectivity. However, most of transition metals and their metal complexes are expensive, and the methodology is difficult to realize at industrial scale. Meanwhile, hydrogen is usually employed as the reductant, often under high pressures. \(^{15}\) Besides these drawbacks, other disadvantages of these methodologies may include harsh reaction conditions, long reaction time, low yields, and low functional group selectivity, limiting their applications in organic synthesis. Therefore, more general, practical, mild, and efficient methods for the selective reduction of the C=C bond in α,β-unsaturated carbonyl compounds without affecting the C=O bond remain highly desirable.

Transfer hydrogenation is a well-established and efficient protocol that has the advantage of not requiring special equipment or hydrogen gas. Recently, our group developed iridium complex catalyzed transfer hydrogenation of C=O and C=N bonds by using formic acid or formate as the hydride source. \(^{16}\) As far as we know, reports on iridium complex catalyzed transfer hydrogenation of C=C bonds remain limited. \(^{17}\) Therefore, we examined the iridium complex catalyzed chemoselective transfer hydrogenation of the C=C bond of α,β-unsaturated carbonyl compounds by

<table>
<thead>
<tr>
<th>Entry</th>
<th>Catalyst</th>
<th>Hydrogen donor</th>
<th>Solvent</th>
<th>Yield (%)(^{b})</th>
</tr>
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<tbody>
<tr>
<td>1</td>
<td>TC-1</td>
<td>HCOOH</td>
<td>H(_2)O</td>
<td>60</td>
</tr>
<tr>
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<td>HCOOH</td>
<td>H(_2)O</td>
<td>45</td>
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<tr>
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<td>HCOOH</td>
<td>H(_2)O</td>
<td>49</td>
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<tr>
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<td>HCOOH</td>
<td>H(_2)O</td>
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<tr>
<td>5</td>
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<td>HCOOH</td>
<td>H(_2)O</td>
<td>52</td>
</tr>
<tr>
<td>6</td>
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<td>HCOOH</td>
<td>H(_2)O</td>
<td>57</td>
</tr>
<tr>
<td>7</td>
<td>TC-1</td>
<td>HCOONa</td>
<td>H(_2)O</td>
<td>48</td>
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<tr>
<td>8(^c)</td>
<td>TC-1</td>
<td>HCOOH/Et(_3)N</td>
<td>H(_2)O</td>
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<tr>
<td>9(^d)</td>
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<td>HCOOH</td>
<td>H(_2)O</td>
<td>63</td>
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<tr>
<td>10</td>
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<td>HCOOH</td>
<td>DMF</td>
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<td>11</td>
<td>TC-1</td>
<td>HCOOH</td>
<td>toluene</td>
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<tr>
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<td>HCOOH</td>
<td>CH(_2)Cl(_2)</td>
<td>57</td>
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<td>HCOOH</td>
<td>MeOH</td>
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<tr>
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<td>H(_2)O</td>
<td>69</td>
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<tr>
<td>17(^f)</td>
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<tr>
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<td>H(_2)O/MeOH</td>
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<td>H(_2)O/MeOH</td>
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<td>H(_2)O</td>
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<tr>
<td>21(^j)</td>
<td>TC-1</td>
<td>HCOOH</td>
<td>–</td>
<td>5</td>
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</tbody>
</table>

\(^{a}\) Reaction conditions: 1a (0.5 mmol), solvent (2 mL), catalyst (1 mol%), hydrogen donor (10 equiv) at room temperature under air for 12 h.

\(^{b}\) Determined by GC–MS using dodecane as the internal standard. The number in parentheses is the isolated yield.

\(^{c}\) The reaction was carried out with 5.0 equiv of HCOOH, 2.0 equiv of Et\(_3\)N.

\(^{d}\) The reaction was carried out under N\(_2\) atmosphere.

\(^{e}\) The reaction was carried out at 80 °C.

\(^{f}\) CH\(_2\)Cl\(_2\) (1 mL) was added in the reaction.

\(^{g}\) MeOH (1.0 mL) was added in the reaction.

\(^{h}\) MeOH (2 mL) was added in the reaction.

\(^{i}\) 2 equiv (n-Bu)\(_4\)NBr was added in the reaction.

\(^{j}\) This reaction in only formic acid.
adjusting the structures of the catalysts and hydrogen source. To achieve this goal, two problems needed to be settled: the effective catalytic system to realize C=C bond reduction and the suppression of side reactions. Herein, we describe an efficient and practical iridium complex catalyzed chemoselective transfer hydrogenation of the C=C bond of α,β-unsaturated carbonyl compounds.

In the initial attempts at selective reduction of α,β-unsaturated ketones, chalcone was used as model substrate, iridium complexes as catalysts, and HCOOH as hydrogen source at room temperature under air (Table 1). Interestingly, the desired product 3aa was afforded in a yield of 60% with the TC-1 catalyst (entry 1). To explore better catalytic system, several other of Tang’s catalysts with different substituted functional groups were also screened (entries 2–6). Disappointedly, lower catalytic activities were obtained. As different hydride sources have important effects on transfer hydrogenation, other candidates were employed in this catalytic system. However, lower yields were obtained by using HCOONa and HCOOH/NEt3 as hydride sources (entries 7 and 8). We also performed the reaction under N2 under standard conditions, but this showed no obvious improvement (entry 9). During our study, we observed that the substrate did not dissolve in the water. Therefore, a screening of organic solvents was performed. As shown (Table 1, entries 10–15), only moderate yields were achieved in organic solvents. However, at the same time, we also found that the solubility of 1a in organic solvents was different. For example, MeOH and CH2Cl2 can dissolve 1a completely, while it did not dissolve in DMF. Furthermore, when the reaction was performed in water, a white suspension was observed on the water surface, which was characterized by NMR spectroscopy and found to be unreacted starting material 1a. A higher reaction temperature led to slightly better conversion (entry 16). In our previous study, we knew that the catalysts had the features of excellent water solubility. Based on our previous research and above results, we envisaged that mixed solvents could help to improve catalytic activity. With this in mind, mixed solvents were next tested. To our satisfaction, high yields of 3aa were achieved in a mixture of H2O and MeOH under the standard conditions (entries 17–19). A phase-transfer catalyst such as quaternary ammonium salt ([n-Bu4N]+[Br]) was used in just water, but only 12% of desired product was detected (entry 20). When using formic acid as hydrogen source and solvent, only a 5% yield of 3aa was obtained (entry 21).

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Intrigued by this simple and efficient procedure for the selective reduction of 1a, we then explored the substrate scope under the optimized transfer hydrogenation conditions (Scheme 2). In general, electron-donating and electron-withdrawing substituents on the phenyl ring (β to carbonyl group) and benzoyl rings are well tolerated and furnished the desired products in good yields (Scheme 2). For example, the substrate with a phenyl ring β to the carbonyl group contains substituents such as p-methyl, methoxy, chloride, fluoride, and bromide provided good to excellent yields of the corresponding products under standard conditions (3ab–ah). Notably, substrates with heterocyclic rings such as furyl, thiophenyl, and naphthyl also reacted smoothly and gave the desired products in high yields (3ai,aj).

To explore the utility of this iridium-catalyzed chemoselective transfer hydrogenation further, we also examined substrates with different substituents on the benzoyl ring. A variety of chalcones with halogen substituents on the benzoyl ring were selectively reduced in good yields (3ba–bc). Substrates with strongly electron-drawing groups on the benzoyl ring, such as trifluoromethyl and nitryl, were selectively reduced to give the desired substituted ketones in yields of 80% and 81%, respectively (3bd,bf). Substrates possessing methyl and methoxy groups on the benzoyl ring also reacted under the optimized conditions (3bg,bh). Of note, heterocyclic acyl substrates were also observed to be well-tolerated under the standard conditions, giving 3bi and 3bj in 87% and 81% yields, respectively. In addition, the sterically hindered 1-naphthyl substrate (2bk) led to the successful synthesis of 3bk. In addition, the doubly unsaturated substrate 2bl with a β-aryl and β-cyclohexenyl substituent gave 3bl efficiently, which demonstrates that unsaturated double bonds contiguous with a β-aryl group can also be reduced selectively. In keeping with this

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observation, when dibenzylideneacetone was employed as the substrate, the corresponding doubly reduced product 3bm could be obtained in a yield of 76%.

In order to verify the practical synthetic application of this chemoselective transfer hydrogenation reduction of α,β-unsaturated ketones, a scaled-up experiment was conducted. When 1a (10 mmol) was carried out under above established conditions, 3aa was isolated by flash chromatography in a yield of 88% (Scheme 3).

Scheme 3 Gram-scale experiment

Alcohols are usually employed as the proton source for transfer hydrogenation reduction and are preferred as a convenient, economical, and environmentally relatively benign choice. In this catalytic system, the hydride and proton sources are derived from formic acid and methanol. To gain more insight into this catalytic system, deuterium-labeling experiments were performed (Scheme 4). By using D₂O as solvent, the ratio of H/D at C2 was found to be 61:39 and C1 was not deuterated. The deuterium incorporation at C2 may be caused by H–D exchange between [Ir]–H and D₂O. However, the outcome was quite different when DCOOD was employed. By using DCOOD instead of HCOOH under standard reaction conditions, product 3aa was afforded in 90% yield with a C1 ratio of H/D of 44:56, with no deuteration at C2.

Scheme 4 Mechanistic studies

In conclusion, we have developed an iridium-catalyzed chemoselective transfer hydrogenation of the C=C bond of chalcones to prepare 1,4-diaryl ketones in good to excellent yields by using formic acid as hydrogen source. The broad substrates scope, simple operation, and high chemoselectivity are the attractive features of this transformation. Further investigations as well as exploration of asymmetric transfer hydrogenation are in progress.

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Supporting Information

Supporting information for this article is available online at https://doi.org/10.1055/s-0040-1706022.

References and Notes

Procedure for the Preparation of 3

To a 2.5 mL dried Schlenk tube was added the a,b-unsaturated ketone (2, 0.5 mmol), Ir catalyst (1.0 mol %), HCOOH (10.0 equiv), water (2.0 mL), and MeOH (2.0 mL) successively. The mixture was stirred at room temperature for 12 h under air. After reaction was complete, the mixture was diluted with H2O (15.0 mL), neutralized with saturatedaq. NaHCO3, and extracted with EtOAc (3 × 10.0 mL). The combined organic layers were washed with brine (3 × 10.0 mL), and dried over anhydrous MgSO4. After filtration and removal of the EtOAc under vacuum, the crude product was purified by column chromatography on silica gel, eluting with hexane or petroleum ether/ethyl acetate (10:1 to 50:1) to achieve the desired products.

1,3-Diphenylprop-1-one (3aa)12

Yield 90% (94.5 mg), pale yellow oil. 1H NMR (400 MHz, CDCl3): δ = 7.80 (d, J = 7.8 Hz, 2 H), 7.59 (t, J = 7.8 Hz, 1 H), 7.49 (1, J = 7.7 Hz, 2 H), 7.37–7.22 (m, 5 H), 7.30–7.25 (m, 3 H), 7.23 (s, 3 H). 13C NMR (100 MHz, CDCl3): δ = 133.3, 129.3, 128.9, 128.8, 128.7, 127.8, 127.6, 127.5, 127.4, 126.9, 126.8, 126.7, 126.5, 125.3, 119.5.

1-Phenyl-3-(p-tolyl)prop-1-one (3ab)14

Yield 84% (119.5 mg), colorless oil. H NMR (400 MHz, CDCl3): δ = 7.91–7.85 (m, 2 H), 7.69–7.58 (m, 5 H), 7.07 (d, J = 8.6 Hz, 2 H), 6.05 (s, 1 H), 141.4, 139.5, 132.8, 128.8, 128.1, 126.9, 125.8, 39.8, 20.7, 15.1. ESI-HRMS: m/z calcd for C18H12O4Br [M + Br]$: 397.0134; found: 397.0135.

1-(4-Nitrophenyl)-3-phenylpropan-1-one (3bh)14

Yield 81% (103.3 mg), colorless oil. H NMR (400 MHz, CDCl3): δ = 8.32 (d, J = 8.8 Hz, 2 H), 8.11 (d, J = 8.8 Hz, 2 H), 7.36–7.30 (m, 2 H), 7.27 (dd, J = 11.1, 4.1 Hz, 3 H), 3.37 (t, J = 7.6 Hz, 2 H), 3.12 (t, J = 7.5 Hz, 2 H). 13C NMR (100 MHz, CDCl3): δ = 197.7, 150.3, 141.3, 140.6, 129.1, 128.7, 128.4, 128.9, 124.1, 119.0, 50.7, 40.3, 21.7.

1-(4-Methoxyphenyl)-3-phenylpropan-1-one (3bh)14

Yield 52% (110.4 mg), colorless oil. H NMR (400 MHz, CDCl3): δ = 7.22 (d, J = 7.7, 1.7 Hz, 1 H), 7.51–7.46 (m, 1 H), 7.28 (dd, J = 21.7, 7.4 Hz, 5 H), 7.06–6.97 (m, 2 H), 3.91 (s, 3 H), 3.37–3.31 (m, 2 H), 3.28–3.23 (m, 2 H). 13C NMR (100 MHz, CDCl3): δ = 201.8, 158.6, 141.8, 133.5, 130.4, 128.5, 128.4, 128.3, 125.9, 120.7, 111.5, 55.5, 45.5, 30.5.

1-(4-Methoxyphenyl)-3-phenylpropan-1-one (3bh)14

Yield 81% (94.0 mg), pale yellow oil. 1H NMR (400 MHz, CDCl3): δ = 7.72 (d, J = 3.8 Hz, 1 H), 7.65 (d, J = 4.9 Hz, 1 H), 7.36–7.27 (m, 4 H), 7.24 (t, J = 7.5 Hz, 1 H), 7.14 (t, J = 4.3 Hz, 1 H), 3.29–3.24 (m, 2 H), 3.13–3.08 (m, 2 H), 2.44 (s, 3 H). 13C NMR (100 MHz, CDCl3): δ = 198.9, 143.9, 141.4, 134.4, 129.3, 128.6, 128.5, 128.2, 126.1, 40.4, 30.3, 21.7.

1-(4-Hydroxyphenyl)-3-phenylpropan-1-one (3bh)14

Yield 88% (100.3 mg), colorless oil. H NMR (400 MHz, CDCl3): δ = 7.98–7.93 (m, 2 H), 7.43–7.34 (m, 5 H), 7.09 (t, J = 8.6 Hz, 2 H), 6.85 (d, J = 3.1 Hz, 1 H), 3.35–3.31 (m, 2 H), 3.28–3.23 (m, 2 H). 13C NMR (100 MHz, CDCl3): δ = 198.7, 154.8, 141.1, 136.8, 133.2, 128.6, 128.1, 110.3, 105.3, 36.9, 22.5.

1-Phenyl-3-(thiophen-2-yl)propan-1-one (3a)17

Yield 88% (86.4 mg), pale yellow oil. H NMR (400 MHz, CDCl3): δ = 7.98–7.93 (m, 2 H), 7.43–7.34 (m, 5 H), 7.09 (t, J = 8.6 Hz, 2 H), 6.85 (d, J = 3.1 Hz, 1 H), 3.35–3.31 (m, 2 H), 3.28–3.23 (m, 2 H). 13C NMR (100 MHz, CDCl3): δ = 198.7, 154.8, 141.1, 136.8, 133.2, 128.6, 128.1, 110.3, 105.3, 36.9, 22.5.

1-Phenyl-3-(thiophen-2-yl)propan-1-one (3a)17

Yield 88% (86.4 mg), pale yellow oil. H NMR (400 MHz, CDCl3): δ = 7.98–7.93 (m, 2 H), 7.43–7.34 (m, 5 H), 7.09 (t, J = 8.6 Hz, 2 H), 6.85 (d, J = 3.1 Hz, 1 H), 3.35–3.31 (m, 2 H), 3.28–3.23 (m, 2 H). 13C NMR (100 MHz, CDCl3): δ = 198.7, 154.8, 141.1, 136.8, 133.2, 128.6, 128.1, 110.3, 105.3, 36.9, 22.5.

1-(4-Fluorophenyl)-3-phenylpropan-1-one (3a)14

Yield 88% (100.3 mg), colorless oil. H NMR (400 MHz, CDCl3): δ = 7.98–7.93 (m, 2 H), 7.32–7.18 (m, 5 H), 7.09 (t, J = 8.6 Hz, 2 H), 3.25 (t, J = 7.7 Hz, 2 H), 3.05 (t, J = 7.6 Hz, 2 H). 13C NMR (100 MHz, CDCl3): δ = 197.6, 165.7 (d, J = 253 Hz), 141.2, 133.3 (d, J = 2 Hz), 130.7 (d, J = 9 Hz), 128.6, 128.5 (d, J = 14 Hz), 126.2, 115.7 (d, J = 22 Hz), 40.4, 30.1. ESI-HRMS: m/z calcd for C18H12OF [M + H]+: 277.0928; found: 277.0930.

1-(4-Chlorophenyl)-3-phenylpropan-1-one (3b)14

Yield 84% (102.5 mg), colorless oil. H NMR (400 MHz, CDCl3): δ = 7.91–7.86 (m, 2 H), 7.42 (d, J = 8.5 Hz, 2 H), 7.31–7.20 (m, 5 H), 3.26 (dd, J = 10.0, 5.3 Hz, 2 H), 3.06 (dd, J = 10.0, 5.3 Hz, 2 H). 13C NMR (100 MHz, CDCl3): δ = 198.0, 141.1, 139.5, 132.2, 129.5, 128.9, 128.6, 128.4, 126.2, 40.4, 30.1.
(E)-5-Phenyl-1-(2,6,6-trimethylcyclohex-2-en-1-yl)pent-1-en-3-one (3bl)
Yield 78% (110.0 mg), colorless oil. \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta = 7.36–7.18\) (m, 7 H), 6.15 (d, \(J = 16.3 \text{ Hz}, 1 \text{ H})\), 2.96 (td, \(J = 14.1, 6.9 \text{ Hz}, 2 \text{ H})\), 2.08 (t, \(J = 6.0 \text{ Hz}, 2 \text{ H})\), 1.76 (s, 3 H), 1.65 (d, \(J = 2.8 \text{ Hz}, 1 \text{ H})\), 1.49 (d, \(J = 5.6 \text{ Hz}, 2 \text{ H})\), 1.07 (s, 6 H). \(^1\)C NMR (100 MHz, CDCl\(_3\)): \(\delta = 199.7, 142.5, 141.4, 136.2, 136.0, 130.5, 128.5, 128.4, 126.1, 42.2, 39.7, 34.1, 33.6, 30.4, 28.8, 21.8, 18.9\). ESI-HRMS \(m/z\) calcd for C\(_{20}\)H\(_{27}\)O [M + H\(^+\)]: 283.2062; found: 283.2064.

1,5-Diphenylpentan-3-one (3bm)\(^{22d}\)
Yield 76% (90.4 mg), colorless oil. \(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta = 7.31\) (t, \(J = 7.4 \text{ Hz}, 4 \text{ H})\), 7.22 (dd, \(J = 17.0, 7.3 \text{ Hz}, 6 \text{ H})\), 2.93 (t, \(J = 7.6 \text{ Hz}, 4 \text{ H})\), 2.75 (dd, \(J = 9.7, 5.5 \text{ Hz}, 4 \text{ H})\). \(^1\)C NMR (100 MHz, CDCl\(_3\)): \(\delta = 209.2, 141.0, 128.5, 128.3, 126.1, 44.5, 29.8\)