

# An environmentally benign protocol: catalyst-free Michael addition of aromatic amines to $\alpha,\beta$ -unsaturated ketones in glycerol

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**Abstract** Glycerol was used as a reaction medium as well as promoter for aza-Michael addition of aromatic amines to electronic deficient  $\alpha,\beta$ -unsaturated ketones. Various aromatic amines can react smoothly with chalcone, 2-cyclohexen-1-one, 2-cyclopenten-1-one, and ethyl vinyl ketone to achieve good to excellent yields in the absence of any catalyst.

**Keywords** Glycerol · Aza-Michael addition · Catalyst free · Recyclability

## Introduction

Crude glycerol, generated by the biodiesel industry as a by-product, has attracted much attention due to its good solubility, low cost, non-volatility and high boiling point, non-toxicity, and that it is readily available. Thus, synthesis of new compounds using glycerol as starting material has been studied by many chemists [1, 2]. However, the direct utilization of glycerol as a reaction solvent in organic synthesis was developed slowly. Wolfson et al. [3, 4] reported that Pd-catalyzed Heck C–C coupling and Suzuki reaction could proceed smoothly in glycerol with the advantage of readily product isolation. Jérôme's group [5] observed that glycerol can be used as a promising medium for ring opening of epoxide with amine under catalyst-free conditions. He et al. [6] depicted that some condensation reactions of aldehydes that are conventionally carried out using acid catalysts could be

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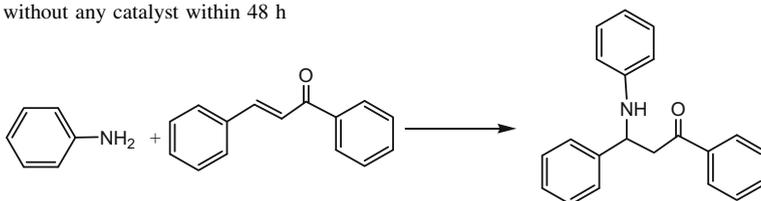
performed in catalyst-free conditions in glycerol, which is beneficial for promoting the electrophilic activity of aldehydes.

Aza-Michael addition has been regarded as one of the widely used reactions for carbon–nitrogen bond formation in modern organic chemistry. Conjugate addition of various amines to  $\alpha,\beta$ -unsaturated carbonyl compounds can readily afford  $\beta$ -amino carbonyl products, which represent a particularly attractive approach, providing rapid access to anticancer agents, antibiotics, and other drugs [7–9]. Recently, some efficient and green methodologies for aza-Michael addition of aliphatic amines to electron-deficient olefins have been developed, which include silica gel in acetonitrile [10],  $\beta$ -cyclodextrin in water [11], PEG (MW 400) and 2-hydroxyethylammonium formate using as reaction medium as well as reaction catalyst [12, 13], boric acid in water [14], and even using water as solvent without a catalyst [15]. The reports with regard to aza-Michael reaction of aromatic amines were relatively scarce for their inertness compared to aliphatic amines [16, 17]. With our continuous efforts for the development of green and highly efficient protocols for Michael addition [18–21] and encouraged by the successful applications of glycerol in organic reactions, we herein report glycerol as a solvent for aza-Michael addition of aromatic amines to electronic deficient  $\alpha,\beta$ -unsaturated ketones in the absence of a catalyst.

## Results and discussion

Firstly, the reaction of aniline and chalcone was selected as a model. In an initial screening of solvents, while traditional organic solvents such as dichloromethane, toluene, acetonitrile, and methanol showed not to be effective for the reaction (Table 1, entries 1–4), glycerol provided the final product with a promising value of yield (Table 1, entry 8). Moreover, some green reaction medium such as water, ionic liquids were attempted in the model reaction as solvents and some moderate yields were obtained (Table 1, entries 5–7). Finally, glycerol was the optimal one furnishing the best product yield, and was selected for subsequent experiments.

With the most appropriate reaction conditions in hand, the substrate scope was next explored with different aromatic amines as well as various  $\alpha,\beta$ -unsaturated ketones. The results are summarized in Table 2. Aromatic amines bearing with electron-donating substituents such as MeO and Me can react smoothly with chalcone, affording excellent product yields within 2 days at 150 °C in glycerol (Table 2, entries 1–3) while amine bearing with electron withdrawn group such as NO<sub>2</sub> almost gave no adduct (Table 2, entry 4). Cyclic ketones 2-cyclopenten-1-one, 2-cyclohexen-1-one were tested as Michael acceptors to react with various arylamines. 2-Cyclohexen-1-one can proceed readily to afford the desired products in excellent yields at 100 °C (Table 2, entries 7–10). However, lower reactivity was observed when 2-cyclopenten-1-one was used as acceptor, probably due to low reactivity of the five-membered ring (Table 2, entries 5–6). 4-Methyl substituted aromatic amine provided a higher yield than that with 2-methyl substitution (Table 2, entries 7–8), demonstrating the negative effect of steric hindrance on the reaction processes. Moreover, acyclic ethyl vinyl ketone (EVK) was also

**Table 1** Results of the reaction between aniline (1 mmol) and chalcone (1 mmol) in various solvents (1 mL) without any catalyst within 48 h

Entry	Solvents	Reaction temperature (°C)	Yields <sup>a</sup> (%)
1	Toluene	Reflux	44
2	CH <sub>2</sub> Cl <sub>2</sub>	Reflux	6
3	CH <sub>3</sub> OH	Reflux	38
4	CH <sub>3</sub> CN	Reflux	35
5	Water	Reflux	41
6	[Bmim]BF <sub>4</sub>	150	58
7	[Bmim]PF <sub>6</sub>	150	53
8	Glycerol	150	95

<sup>a</sup> Isolated yields

investigated. Gratifying, glycerol was very suitable for the reaction of EVK with aromatic amines and 93–98 % product yields were achieved within 12 h of reaction times (Table 2, entries 11–14). Finally, the reactions when other deficient carbonyl compounds were employed as acceptors were tested to react with aniline in glycerol at 150 °C. To our disappointment, almost no corresponding products were detected (Table 2, entries 15–16).

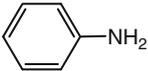
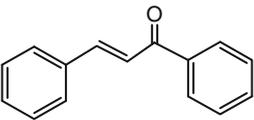
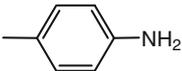
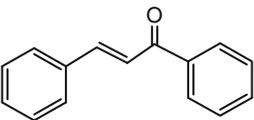
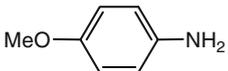
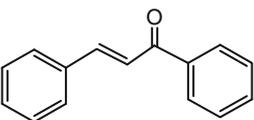
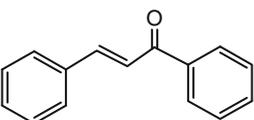
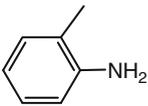
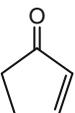
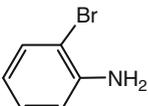
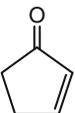
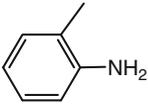
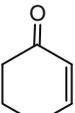
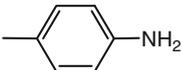
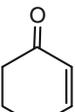
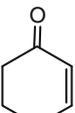
The recyclability of glycerol was then studied using the reaction of aniline and chalcone as a model. The results are shown in Table 3. Upon the completion of the reaction, the reaction solution was extracted with ethyl acetate and purified by flash chromatography. The addition product was identified by <sup>1</sup>H NMR, <sup>13</sup>C NMR, and mass spectra. The residual liquid was directly reused for subsequent reactions. As shown in Table 3, the recovered glycerol could be reused five times without an obvious loss of reactivity.

As reported by other authors [6], glycerol may play a role as a promoter as well as a reaction medium for the aza-Michael addition of aromatic amines to electron-deficient  $\alpha,\beta$ -unsaturated ketones. We speculated that strong solubility of glycerol can make the Michael reaction a homogenous process, which increases the reactivity. Secondly, glycerol as a hydrogen donor can form stabilized hydrogen bonds with the carbonyl of the ketone.

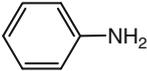
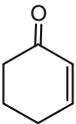
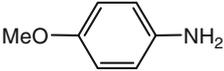
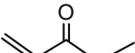
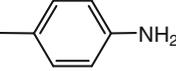
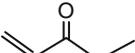
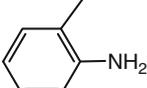
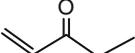
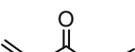
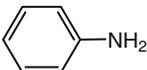
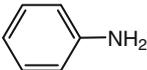
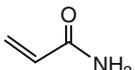
## Conclusions

In conclusion, glycerol was proven to be an effective medium for promoting an aza-Michael reaction between substituted aromatic amines and  $\alpha,\beta$ -unsaturated ketones. In glycerol, various aromatic amines can react smoothly with electron-deficient

**Table 2** Results of aza-Michael reaction between various substituted aromatic amines and  $\alpha,\beta$ -unsaturated ketones

Entry	Michael donors	Acceptors	Reaction time (h)	Reaction temperature (°C)	Isolated yields
1			48	150	95
2			36	150	92
3			36	150	97
4			96	150	NR
5			48	100	68
6			48	100	75
7			24	100	83
8			24	100	92
9			24	100	85

**Table 2** continued

Entry	Michael donors	Acceptors	Reaction time (h)	Reaction temperature (°C)	Isolated yields
10			24	100	90
11			12	100	98
12			12	100	95
13			12	100	96
14			12	100	93
15			96	150	NR
16			96	150	NR

Reaction conditions: aromatic amines (1 mmol),  $\alpha,\beta$ -unsaturated ketones (1 mmol), glycerol (1 mL), catalyst-free conditions

**Table 3** Recyclability of glycerol for reaction of aniline and chalcone

Cycle	Reaction time (h)	Isolated yield (%)
1	48	96
2	48	94
3	48	92
4	60	98
5	60	95

Reaction conditions: aniline (1 mmol), chalcone (1 mmol), glycerol (1 mL), catalyst-free conditions and 150 °C

olefins to afford desired products in the absence of any catalyst. Considering the high environmental compatibility and sustainability and catalytic activity of glycerol, the extension of its application to organic reactions are underway in our laboratory.

## Experimental

All chemicals were purchased from Aldrich or Fluka.  $^1\text{H}$  and  $^{13}\text{C}$  NMR were recorded on a Bruker Avance DPX 400 spectrometer at 400 and 100 MHz in  $\text{CDCl}_3$  and  $\text{DMSO}-d_6$ , respectively. Chemical shifts were reported in parts per million ( $\delta$ ), relative to the internal standard of tetramethylsilane (TMS). Melting points were determined using a YRT-3 apparatus and were not corrected. Mass spectrometry data were obtained on Bruker Esquire-LC for electro-spray (MS-ES) measurements. Elemental analysis was carried out on a Carlo Erba 1160. All reactions were monitored by thin-layer chromatography (TLC). Flash chromatography was performed on silica gel (100–200 mesh). All Michael adducts were purified through column chromatography and were characterized by NMR analysis, melting points, and MS.

General procedure for catalyst-free aza-Michael reaction of aliphatic amines with  $\alpha,\beta$ -unsaturated compounds in glycerol

To a mixture of the aromatic amine (1 mmol) and Michael acceptor (1 mmol) in a 10-mL flask equipped with a magnetic stirrer was added glycerol (1 mL). The reaction mixture was stirred at rational temperature for the desired time until the disappearance of the starting material, monitored by TLC. Upon completion of the reaction, the mixture was extracted with ethyl acetate several times. The combined organic phase was concentrated through vacuum evaporation and the resulting crude product was purified by silica-column chromatography to give the desired product. These products are in good agreement with spectra data of the literature. The recovered glycerol was then reused in subsequent reactions.

*N*-(3-oxo-1,3-diphenylpropyl)aniline (Table 2, entry 1)

White solid, mp 173–175 °C (lit. [23], 172–174 °C);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.92 (d, 2H,  $J$  = 8.0 Hz, ArH), 7.56 (t, 1H,  $J$  = 7.2 Hz, ArH), 7.46–7.43 (m, 4H, ArH), 7.33 (t, 2H,  $J$  = 7.6 Hz, ArH), 7.24 (t, 1H,  $J$  = 7.6 Hz, ArH), 7.10 (t, 2H,  $J$  = 8.0 Hz, ArH), 6.67 (t, 1H,  $J$  = 7.2 Hz, ArH), 6.57 (d, 2H,  $J$  = 8.0 Hz, ArH), 5.02 (brs, 1H, NH), 4.58–4.57 (m, 1H, CH), 3.55–3.40 (m, 2H,  $\text{CH}_2$ );  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 198.2, 146.9, 142.9, 136.5, 133.4, 129.0, 128.8, 128.7, 128.2, 127.3, 126.3, 117.7, 113.7, 54.6, 46.3; ESI-MS ( $m/z$ ): 302 (M+1).

*N*-(3-oxo-1,3-diphenylpropyl)-4-methylaniline (Table 2, entry 2)

White solid, mp 170–172 °C (lit. [23], 170–171 °C);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.94–7.89 (m, 2H, ArH), 7.55 (t, 1H,  $J$  = 7.2 Hz, ArH), 7.43–7.42 (m, 4H, ArH), 7.32–7.22 (m, 4H, ArH), 6.89 (d, 2H,  $J$  = 7.2 Hz, ArH), 6.47 (d, 2H,  $J$  = 7.2 Hz, ArH), 4.96 (brs, 1H, NH), 4.43–4.41 (m, 1H, CH), 3.52–3.37 (m, 2H,  $\text{CH}_2$ ), 2.17 (s, 3H,  $\text{CH}_3$ );  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 198.3, 144.7, 143.1, 136.7, 133.4, 129.6, 129.0, 128.8, 128.7, 128.2, 127.3, 126.4, 114.0, 55.0, 46.3, 20.3; ESI-MS ( $m/z$ ): 316 (M+1).

*N*-(3-oxo-1,3-diphenylpropyl)-4-methoxyaniline (Table 2, entry 3)

White solid, mp 142–143 °C (lit. [24], 140–144 °C); <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ = 7.90 (d, 2H, *J* = 7.6 Hz, ArH), 7.55 (t, 1H, *J* = 7.2 Hz, ArH), 7.43–7.42 (m, 4H, ArH), 7.31 (t, 2H, *J* = 7.2 Hz, ArH), 7.22 (t, 1H, *J* = 7.2 Hz, ArH), 6.67 (d, 2H, *J* = 8.4 Hz, ArH), 6.52 (d, 2H, *J* = 8.4 Hz, ArH), 4.92 (brs, 1H, NH), 4.23 (m, 1H, CH), 3.68 (s, 3H, CH<sub>3</sub>), 3.50–3.36 (m, 2H, CH<sub>2</sub>); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ = 198.3, 152.3, 143.2, 141.2, 136.7, 133.4, 128.8, 128.7, 128.2, 127.3, 126.4, 115.3, 114.7, 55.6, 46.4, 39.9; ESI-MS (*m/z*): 332 (M+1).

*3*-(*o*-Toluidinyl)-cyclopentan-1-one (Table 2, entry 5)

Pale yellow oil; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ = 7.16–7.02 (m, 2H, ArH), 6.73–6.65 (m, 2H, ArH), 4.19–4.10 (m, 1H, CH), 3.57 (brs, 1H, NH), 2.77–2.68 (m, 2H, CH<sub>2</sub>), 2.51–1.97 (m, 4H, 2 × CH<sub>2</sub>), 2.44–2.19 (m, 2H, CH<sub>2</sub>); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ = 217.1, 144.7, 134.5, 130.3, 127.0, 117.6, 110.4; ESI-MS (*m/z*): 190 (M+1).

*3*-(2-Bromoanilinyl)-cyclopentan-1-one (Table 2, entry 6)

Pale yellow oil; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ = 7.45–7.39 (m, 1H, ArH), 7.22–7.17 (m, 1H, ArH), 6.67–6.60 (m, 2H, ArH), 4.37 (brs, 1H, NH), 4.14 (m, 1H, CH), 2.77–2.69 (m, 2H, CH<sub>2</sub>), 2.53–2.45 (m, 2H, CH<sub>2</sub>), 2.44–2.19 (m, 2H, CH<sub>2</sub>); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ = 216.4, 143.7, 132.6, 128.5, 118.5, 111.8, 110.1, 51.1, 45.8, 36.6, 29.7; ESI-MS (*m/z*): 255 (M+1).

*3*-(*o*-Toluidinyl)-cyclohexan-1-one (Table 2, entry 7)

Pale yellow oil; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ = 7.14–7.06 (m, 2H, ArH), 6.70–6.66 (m, 2H, ArH), 6.62–6.60 (m, 1H, ArH), 3.44 (brs, 1H, NH), 2.89–2.85 (m, 1H, CH), 2.42–2.28 (m, 4H, 2 × CH<sub>2</sub>), 2.12 (s, 3H, CH<sub>3</sub>), 2.08–2.01 (m, 2H, CH<sub>2</sub>), 1.82–1.70 (m, 2H, CH<sub>2</sub>); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ = 209.7, 144.1, 130.4, 127.1, 122.0, 117.3, 110.2, 52.0, 48.6, 41.1, 38.0, 31.0, 22.1, 17.5; ESI-MS (*m/z*): 204 (M+1).

*3*-(*p*-Toluidinyl)-cyclohexan-1-one (Table 2, entry 8)

Pale yellow oil; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ = 6.98 (d, 2H, *J* = 8 Hz, ArH), 6.51 (d, 2H, *J* = 8 Hz, ArH), 3.73 (brs, 1H, NH), 2.82–2.78 (m, 1H, CH), 2.43–2.24 (m, 4H, 2 × CH<sub>2</sub>), 2.16–2.03 (m, 2H, CH<sub>2</sub>), 1.72–1.67 (m, 2H, CH<sub>2</sub>); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ = 210.1, 144.2, 130.1, 127.4, 113.8, 52.9, 48.9, 41.4, 31.3, 22.4, 20.6; ESI-MS (*m/z*): 204 (M+1).

*3*-(4-Chloroanilinyl)-cyclohexan-1-one (Table 2, entry 9)

Pale yellow oil; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ = 7.11 (d, 2H, *J* = 8 Hz, ArH), 6.50 (d, 2H, *J* = 8 Hz, ArH), 3.74 (brs, 1H, NH), 2.83–2.81 (m, 1H, CH), 2.43–2.23

(m, 4H, 2 × CH<sub>2</sub>), 2.20–2.00 (m, 2H, CH<sub>2</sub>), 1.77–1.62 (m, 2H, CH<sub>2</sub>); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ = 209.6, 145.1, 129.5, 122.6, 114.6, 52.6, 48.5, 41.3, 31.1, 22.3; ESI-MS (*m/z*): 224 (M+1).

*3-(Aniliny)-cyclohexan-1-one* (Table 2, entry 10)

Pale yellow oil; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ = 7.19–7.13 (m, 3H, ArH), 6.77–6.69 (m, 2H, ArH), 3.78 (brs, 1H, NH), 2.83 (m, 1H, CH), 2.43–2.15 (m, 4H, 2 × CH<sub>2</sub>), 2.09–1.99 (m, 2H, CH<sub>2</sub>), 1.79–1.64 (m, 2H, CH<sub>2</sub>); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ = 209.4, 146.0, 129.1, 117.6, 113.0, 52.0, 48.2, 40.8, 30.7, 21.8; ESI-MS (*m/z*): 190 (M+1).

*1-(4-Methoxyaniliny)-3-pentanone* (Table 2, entry 11)

White solid, mp 42–43 °C (lit. [22], 41–43 °C); <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ = 6.78 (d, 2H, *J* = 7.2 Hz, ArH), 6.66 (d, 2H, *J* = 7.2 Hz, ArH), 3.28 (s, 3H, OCH<sub>3</sub>), 3.36 (t, 2H, *J* = 6 Hz, CH<sub>2</sub>), 2.71 (t, 2H, *J* = 6 Hz, CH<sub>2</sub>), 2.42 (q, 2H, *J* = 7.2 Hz, CH<sub>2</sub>), 1.05 (t, 3H, *J* = 7.2 Hz, CH<sub>3</sub>); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ = 211.1, 152.1, 141.8, 114.7, 114.5, 55.6, 41.2, 39.5, 36.2, 7.6; ESI-MS (*m/z*): 208 (M+1).

*1-(p-Toluidiny)-3-pentanone* (Table 2, entry 12)

White solid, mp 64–65 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ = 6.98 (d, 2H, *J* = 8.0 Hz, ArH), 6.53 (d, 2H, *J* = 8.0 Hz, ArH), 3.88 (brs, 1H, NH), 3.39 (t, 2H, *J* = 6 Hz, CH<sub>2</sub>), 2.70 (t, 2H, *J* = 6 Hz, CH<sub>2</sub>), 2.43 (q, 2H, *J* = 7.2 Hz, CH<sub>2</sub>), 2.23 (s, 3H, CH<sub>3</sub>), 1.04 (t, 3H, *J* = 7.2 Hz, CH<sub>3</sub>); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ = 211.0, 145.3, 129.7, 126.8, 113.2, 41.2, 38.8, 36.2, 20.3, 7.6; ESI-MS (*m/z*): 192 (M+1).

*1-(o-Toluidiny)-3-pentanone* (Table 2, entry 13)

Pale yellow oil; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ = 7.13 (t, 2H, *J* = 8.0 Hz, ArH), 7.05 (d, 2H, *J* = 7.2 Hz, ArH), 6.68–6.61 (m, 2H, ArH), 3.94 (brs, 1H, NH), 3.47 (t, 2H, *J* = 6 Hz, CH<sub>2</sub>), 2.75 (t, 2H, *J* = 6 Hz, CH<sub>2</sub>), 2.43 (q, 2H, *J* = 7.2 Hz, CH<sub>2</sub>), 2.11 (s, 3H, CH<sub>3</sub>), 1.03 (t, 3H, *J* = 7.2 Hz, CH<sub>3</sub>); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ = 211.0, 145.6, 130.2, 127.0, 122.5, 117.0, 109.5, 41.1, 38.3, 36.3, 17.4, 7.6; ESI-MS (*m/z*): 192 (M+1).

*1-(4-Chloroaniliny)-3-pentanone* (Table 2, entry 14)

White solid, mp 74–76 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ = 7.12 (d, 2H, *J* = 8.8 Hz, ArH), 6.53 (d, 2H, *J* = 8.8 Hz, ArH), 4.07 (brs, 1H, NH), 3.39 (t, 2H, *J* = 6 Hz, CH<sub>2</sub>), 2.72 (t, 2H, *J* = 6 Hz, CH<sub>2</sub>), 2.45 (q, 2H, *J* = 7.2 Hz, CH<sub>2</sub>), 1.06 (t, 3H, *J* = 7.2 Hz, CH<sub>3</sub>); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ = 210.8, 146.2, 129.0, 121.9, 114.0, 40.9, 38.4, 36.37.6; ESI-MS (*m/z*): 212 (M+1).

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