Note

Synthesis of Alkynes from Vinyl Triflates Using Tetrabutylammonium Fluoride

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A convenient method for the preparation of alkynes and alkynyl esters from ketones and *β*-keto esters is described which involves the formation of vinyl triflates, followed by elimination with tetrabutylammonium fluoride trihydrate, to give alkynes. Unlike established elimination methods, the method requires neither a strong base nor anhydrous conditions.

Key words tetrabutylammonium fluoride; vinyl triflate; elimination; alkyne

Tetrabutylammonium fluoride (TBAF) is widely used for organic synthesis in a range of fluoride-assisted reactions, such as deprotection of silyl ethers, desilylation, and fluorination. TBAF can also act as a mild base[1] in a variety of base-catalyzed, aldol-type condensation reactions[2–4] and Michael-type reactions.[5–7] We have reported that TBAF can be employed as a base in the anti-elimination reaction of vinyl bromides to obtain the corresponding alkynes.[8,9] The salient feature of this elimination is that dehydrobromination is efficiently brought about by the commercially available hydrated TBAF (TBAF·$3H_2O$); further, in contrast to conventional methods, which require strong bases such as *t*-BuOK,[10–12] alkali hydrides,[13–15] and alkali metal amides[16,17] as well as strictly anhydrous conditions, our moisture-insensitive protocol is very practical.

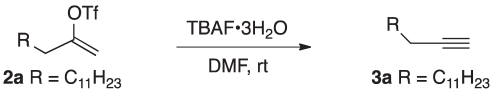
In this work, we have extended the scope of the hydrated TBAF-induced elimination reaction to vinyl triflates **2**, which are more reactive than vinyl bromides and can be readily prepared from ketones **1** (Chart 1). Several different procedures for conversion of vinyl triflates to alkynes have been reported; however, as with vinyl bromides, most of the triflates require strong bases and anhydrous conditions,[18–24] or prolonged heating with weak bases at elevated temperatures.[25–27] The TBAF-induced elimination of vinyl triflates is rare in the literature, and only two examples of its use were found.[28,29]

Vinyl triflates **2** were prepared from the corresponding ketones **1** in 50–80% yields by the established procedure using lithium diisopropylamide (LDA) and *N*-(2-pyridiyl)triflimide[18,30] under kinetically controlled conditions. Initial experiments were performed using vinyl triflate **2a** to establish reaction conditions (Table 1). Treatment of **2a** with 1.1 equiv of TBAF·$3H_2O$ in *N*,*N*-dimethylformamide (DMF) at room temperature resulted in incomplete conversion of **2a** to terminal acetylene **3a** after 22 h, and 11% of the starting material was recovered (Table 1, entry 1). Reaction with 2.0 equiv of the base led to complete elimination, resulting in 91% yield of the product (Table 1, entry 2). Further increase in the amount of base to 3.0 equiv drastically shortened the reaction time from 18 h to 10 min while maintaining the high yield (Table 1, entry 3). These optimized reaction conditions were adopted for further study.

The elimination reactions of acyclic vinyl triflates **2b**–**2d** are usually completed in 20 min to give the corresponding terminal alkynes in good yields (Table 2, entries 1 to 3). Macrocyclic (*Z*)-vinyl triflate **2e**[18,31] also undergoes elimination, but requires a longer reaction time (1 h) to afford alkyne **3e**. This reaction also affords the corresponding allene in 7% yield, which may arise from the *E*-isomer of the starting material (Table 2, entry 4). Notably, the LDA-induced elimination reaction of a 10 : 1 *E*/*Z* mixture of **2e** has been reported to afford a 93 : 7 mixture of the alkyne and allene in 95% yield, while an 11 : 1 *E*/*Z* mixture of a vinyl phosphate derivative corresponding to **2e** affords the allene in 75% yield.[32]

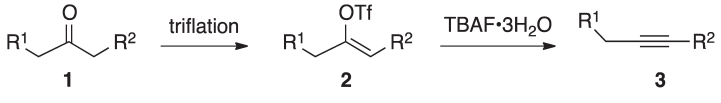
The success of this reaction prompted us to extend the procedure to vinyl triflates **5** derived from *β*-keto esters **4** (Table 3). The transformation of *β*-keto esters to conjugated alkynyl esters *via* a vinyl triflate has been reported by Brummond *et al.*[18] and Maity and Lepore.[23] Although their procedure is useful for 2-alkynoate synthesis, it requires anhydrous conditions and the use of LDA or lithium bis(trimethylsilyl)amide (LiHMDS) as the base. Fleming and Ramarao have demonstrated a decarboxylative elimination of triflic acid from vinyl triflate **5** to form terminal acetylenes in the presence of

Table 1. Elimination Reaction of **2a** Mediated by TBAF·$3H_2O$



2a R = $C_{11}H_{23}$ **3a** R = $C_{11}H_{23}$

Entry	TBAF·$3H_2O$ (equiv)	Time	Yield (%)
1	1.1	2 h	78 (+11% of sm)
2	2.0	18 h	91
3	3.0	10 min	92



1 **2** **3**

Chart 1. Preparation of Alkynes from Ketones *via* Vinyl Triflates

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Table 2. Elimination Reaction of Vinyl Triflates **2b–e** with TBAF·$3H_2O$

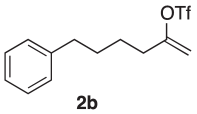
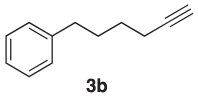
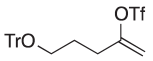
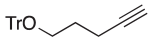
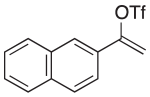
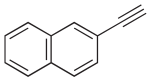
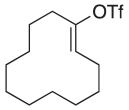
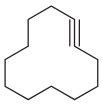
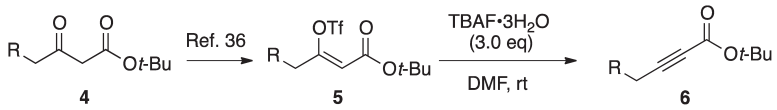
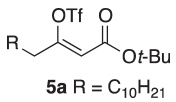
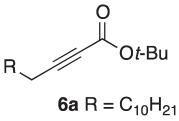
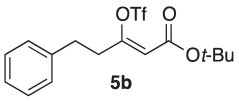
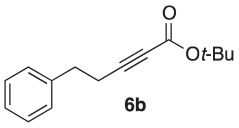
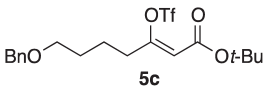
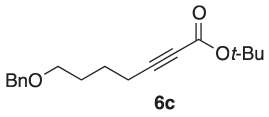
Entry	Vinyl triflate	Time (min)	Product	Yield (%)
1	 **2b**	20	 **3b**	94
2	 **2c**	20	 **3c**	95
3	 **2d**	15	 **3d**	76
4	 **2e** E : Z = 8 : 92	60	 **3e**	84 (+7% allene)

Table 3. Elimination Reaction of (*Z*)-3-Triflyloxy-2-enoates **5a–c** with TBAF·$3H_2O$



Entry	Vinyl triflate	Time	Product	Yield (%)
1	 **5a** R = $C_{10}H_{21}$	20	 **6a** R = $C_{10}H_{21}$	93
2	 **5b**	10	 **6b**	93
3	 **5c**	25	 **6c**	90

trifluoroacetic acid, followed by heating with K_2CO_3 in acetone.[33,34] Hence, investigation of the reaction of vinyl triflates **5** with hydrated TBAF could provide a convenient alternative to established methods of alkynoate synthesis.

Several methods for the triflation of *β*-keto esters, in which the stereochemical outcome of the reaction strongly depends on the triflating reagent and solvent, have been reported.[35] Our previous study has shown that the TBAF-induced elimination proceeds *via* an *anti*-transition state.[9] Therefore, the requisite substrates should be (*Z*)-vinyl triflates **5**. These were prepared stereoselectively according to the procedure reported by Pale and colleagues.[36] Elimination reactions of **5a–c** with 3.0 equiv of TBAF·$3H_2O$ were carried out in DMF at room temperature, and the results are summarized in Table 3. As expected, hydrated TBAF promoted the elimination reaction of **5** within 30 min to give conjugated alkyne esters **6a–6c** in high yields. The overall transformation was similar to that achieved in the one-pot reaction reported by Maity and Lepore.[23] in which *β*-keto esters were treated with LiHMDS and triflic anhydride (Tf_2O) at −78°C. However, the reaction conditions described here are very mild.

In conclusion, we have demonstrated an efficient method for the synthesis of terminal alkynes and 2-alkynyl esters from vinyl triflates using hydrated TBAF to promote elimination, in which TBAF serves as a mild base to eliminate triflic acid from vinyl triflates at room temperature. This method is a robust and moisture-insensitive alternative to existing methods of alkyne synthesis from ketones and *β*-keto esters.

Experimental

General All air- and moisture-sensitive reactions were carried out under an argon atmosphere in dry, freshly distilled solvents. The term "dried" refers to the drying of an organic solution over $MgSO_4$, followed by filtration. Flash chromatog-

raphy was carried out with silica gel (spherical, neutral, particle size 40–50 μm). IR spectra were recorded in $CHCl_3$ solution on a JASCO FTIR-420 spectrometer. ^{1}H- and ^{13}C-NMR spectra were recorded on a JEOL A-400 spectrometer. Chemical shifts are reported in ppm relative to an internal TMS standard (δ 0.00 ppm) for ^{1}H-NMR spectra, and to the solvent signals (δ 77.0 ppm for $CDCl_3$) for ^{13}C-NMR spectra. High-resolution electron-impact mass spectra (HR-EI-MS) were recorded on JEOL JMS-700 mass spectrometer.

General Procedure for Triflation of Ketones[30] A solution of ketone **1** (4.0 mmol, 1.0 equiv) in tetrahydrofuran (THF) (10 mL) was added dropwise to a stirring solution of LDA (1.5 equiv) in THF (3 mL) at −80°C. After stirring at −80°C for 2 h, a solution of *N*-(2-pyridyl)triflimide (1.2 equiv) in THF (9 mL) was added. The reaction mixture was stirred for 1 h at the same temperature and then allowed to warm to 0°C, followed by stirring for 2 h. The reaction was quenched with saturated aqueous NH_4Cl solution, and the reaction mixture was extracted with diethyl ether. The extract was washed with water and brine, dried, and concentrated under reduced pressure. Purification of the residue by flash chromatography (1–5% ethyl acetate in hexane) afforded vinyl triflate **2** in 50–85% yield.

General Procedure for Triflation of β-Keto Esters[36] To a suspension of β-keto ester **4** (3.0 mmol, 1.0 equiv) and LiOTf (2.0 equiv) in CH_2Cl_2 (30 mL) at 0°C was added Et_3N (1.1 equiv). After stirring at 0°C for 20 min, Tf_2O (1.1 equiv) was added, and the reaction mixture was stirred for 1 h at this temperature. The reaction was quenched with saturated aqueous NH_4Cl solution, and the reaction mixture was extracted with CH_2Cl_2. The extract was washed with water and brine, dried, and concentrated under reduced pressure. Purification of the residue by flash chromatography (5–15% ethyl acetate in hexane) afforded vinyl triflate **5** in 65–82% yield.

General Procedure for Elimination Reaction of Vinyl Triflates with TBAF·H_2O To a solution of vinyl triflate **2** (0.5 mmol) in DMF (2.5 mL) was added a 1 M solution of TBAF·$3H_2O$ in DMF (1.5 mmol). The reaction mixture was stirred for 15–60 min and extracted with diethyl ether. The organic phase was washed with water and brine, dried, and concentrated under reduced pressure. Purification of the residue by flash chromatography (hexane or 1–5% ethyl acetate in hexane) afforded alkynes **3**.

Tridec-1-en-2-yl Trifluoromethanesulfonate (2a) ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 5.09 (1H, d, J=3.4 Hz), 4.92 (1H, d, J=3.4 Hz), 2.33 (2H, t, J=7.3 Hz), 1.55 (2H, m), 1.26 (16H, m), 0.88 (3H, t, J=6.8 Hz). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 157.1, 118.4 (q, J=318 Hz), 103.9, 33.8, 31.9, 29.5, 29.43, 29.41, 29.3, 29.2, 28.6, 25.9, 22.7, 14.0. IR ($CHCl_3$) cm^{-1}: 1670, 1416, 1140. HR-EI-MS *m/z*: 330.1462 (Calcd for $C_{14}H_{25}F_3O_3S$: 330.1476).

6-Phenylhex-1-en-2-yl Trifluoromethanesulfonate (2b) ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.32–7.11 (5H, m), 5.09 (1H, d, J=3.4 Hz), 4.90 (1H, dt, J=3.4, 1.0 Hz), 2.64 (2H, m), 2.36 (2H, t, J=6.8 Hz), 1.79–1.55 (4H, m). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 156.7, 142.2, 128.4, 128.3, 125.7, 118.6 (q, J=317 Hz), 104.2, 35.4, 34.9, 30.3, 26.0. IR ($CHCl_3$) cm^{-1}: 1671, 1604, 1498, 1452, 1142. HR-EI-MS *m/z*: 308.0688 (Calcd for $C_{13}H_{15}F_3O_3S$: 308.0694).

5-(Trityloxy)pent-1-en-2-yl Trifluoromethanesulfonate (2c) ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.47–7.22 (15H, m), 5.04 (1H, d, J=3.4 Hz), 4.86 (1H, d, J=3.4 Hz), 3.13 (2H, t, J=6.8 Hz), 2.46 (2H, t, J=7.3 Hz), 1.83 (2H, m). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 156.5, 144.1, 128.6, 127.8, 127.0, 118.8 (q, J=318 Hz), 104.4, 86.5, 61.7, 30.9, 26.6. IR ($CHCl_3$) cm^{-1}: 1671, 1491, 1448, 1415, 1145. HR-EI-MS *m/z*: 476.1296 (Calcd for $C_{25}H_{23}F_3O_4S$: 476.1269).

1-(Naphthalen-2-yl)vinyl Trifluoromethanesulfonate (2d) ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 8.03 (1H, d, J=1.9 Hz), 7.92–7.83 (3H, m), 7.61–7.52 (3H, m), 5.73 (1H, d, J=3.9 Hz), 5.47 (1H, d, J=3.9 Hz). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 153.5, 133.85, 132.81, 129.2, 128.8, 128.7, 127.7, 127.5, 127.0, 125.2, 122.1, 118.8 (q, J=318 Hz), 104.5. IR ($CHCl_3$) cm^{-1}: 1672, 1595, 1410, 1140. HR-EI-MS *m/z*: 302.0246 (Calcd for $C_{13}H_9F_3O_3S$: 302.0224).

(*Z*)-Cyclododec-1-en-1-yl Trifluoromethanesulfonate (2e)[18,31] ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 5.37 (1H, t, J=8.3 Hz), 2.41 (2H, t, J=6.3 Hz), 2,25 (2H, m), 1.55–1.25 (16H). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 184.6, 123.8, 118.4 (q, J=319 Hz), 33.2, 26.2, 25.7, 25.4, 25.3, 25.2, 24.5, 24.4, 24.2, 22.9. IR ($CHCl_3$) cm^{-1}: 1692, 1411, 1221, 1142. HR-EI-MS *m/z*: 314.1160 (Calcd for $C_{13}H_{21}F_3O_3S$: 314.1163). ^{1}H-NMR data of the contaminated *E*-isomer (8%) δ: 5.48 (1H, t, J=8.3 Hz), 2.45 (2H, t, J=6.8 Hz), 2.15 (2H, dt, J=8.3, 6.8 Hz), 1.70–1.25 (16H).

Tridec-1-yne (3a) ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 2.18 (2H, dt, J=7.3, 2.7 Hz), 1.93 (1H, t, J=2.7 Hz), 1.52 (2H, m), 1.40–1.25 (16H, m), 0.88 (3H, t, J=6.8 Hz). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 84.8, 68.0, 31.9, 29.7, 29.6, 29.5, 29.3, 29.1, 28.7, 28.5, 22.7, 18.4, 14.1. IR ($CHCl_3$) cm^{-1}: 3307, 2109, 1467. HR-EI-MS *m/z*: 180.1875 (Calcd for $C_{13}H_{24}$: 180.1878).

Hex-5-yn-1-ylbenzene (3b) ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.30–7.16 (5H, m), 2.63 (2H, t, J=7.3 Hz), 2.20 (2H, dt, J=6.8, 2.4 Hz), 1.94 (1H, t, J=2.4 Hz), 1.75 (2H, m), 1.58 (2H, m). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 142.2, 128.4, 128.3, 125.7, 84.4, 68.3, 35.4, 30.4, 28.0, 18.3. IR ($CHCl_3$) cm^{-1}: 3308, 2118, 1604, 1496, 1454. HR-EI-MS *m/z*: 158.1083 (Calcd for $C_{12}H_{14}$: 158.1096).

(Pent-4-yn-1-yloxy)triphenylmethane (3c) ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.48–7.20 (15H, m), 3.16 (2H, t, J=5.9 Hz), 2.34 (2H, t, J=7.3 Hz), 1.82 (2H, m), 1.88 (1H, t, J=2.4 Hz). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 144.2, 128.7, 127.7, 126.9, 86.5, 84.1, 68.4, 61.9, 29.2, 20.2. IR ($CHCl_3$) cm^{-1}: 3308, 2117, 1597, 1491, 1448. HR-EI-MS *m/z*: 326.1667 (Calcd for $C_{24}H_{22}O$: 326.1671).

2-Ethynylnaphthalene (3d) ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 8.03 (1H, s), 7.85–7.78 (3H, m), 7.54–7.48 (3H, m), 3.15 (1H, s). The ^{1}H-NMR spectrum of **3d** was identical with a reference sample of commercially available 2-ethynylnaphthalene.

Cyclododecyne (3e) ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 2,20–2.17 (4H, m), 1.58–1.51 (8H, m), 1.47–1.40 (8H, m). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 81.6, 25.7, 25.5, 24.9, 24.6, 18.5. IR ($CHCl_3$) cm^{-1}: 1461, 1447, 1322. HR-EI-MS *m/z*: 164.1531 (Calcd for $C_{12}H_{20}$: 164.1565).

1,2-Cyclododecadiene (Allene) ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 4.92–4.84 (2H, m), 2.17–2.02 (4H, m), 1.53–1.37 (10H, m), 1.27–1.05 (4H, m). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 206.6, 88.9, 27.0, 26.8, 26.1, 22.8, 21.6. IR ($CHCl_3$) cm^{-1}: 1956, 1460, 1443, 1328, 1270. HR-EI-MS *m/z*: 164.1548 (Calcd for $C_{12}H_{20}$: 164.1565).

(*Z*)-*tert*-Butyl 3-((Trifluoromethanesulfonyl)oxy)tetradec-2-enoate (5a) ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 5.66 (1H, s), 2.34 (2H, t, J=7.3 Hz), 1.51 (2H, m), 1.50 (9H, s), 1.31–1.25 (16H, m), 0.88 (3H, t, J=6.8 Hz). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 161.8, 157.6, 118.3 (q, J=319 Hz), 113.3, 82.3, 34.2, 31.9,

29.54, 29.51, 29.33, 29.28, 29.1, 28.6, 27.9 (3×C), 25.8, 22.7, 14.1. IR ($CHCl_3$) cm^{-1}: 1721, 1676, 1428, 1149. HR-EI-MS *m/z*: 374.1397 (Calcd for $C_{15}H_{25}F_3O_5S$ $[M-C_4H_8]^+$: 374.1375).

(*Z*)-*tert*-Butyl 5-Phenyl-3-((trifluoromethanesulfonyl)oxy)-pent-2-enoate (5b) ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.34–7.16 (5H, m), 5.66 (1H, t, J=1.0 Hz), 2.88 (2H, t, J=7.3 Hz), 2.64 (2H, t, J=7.3 Hz), 1.49 (9H, s). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 161.6, 156.2, 138.8, 128.8, 128.2, 126.8, 118.4 (q, J=318 Hz), 114.6, 82.5, 36.2, 32.2, 27.9 (3×C). IR ($CHCl_3$) cm^{-1}: 1722, 1676, 1497, 1456, 1428, 1221, 1149. HR-EI-MS *m/z*: 324.0245 (Calcd for $C_{12}H_{11}F_3O_5S$ $[M-C_4H_8]^+$: 324.0279).

(*Z*)-*tert*-Butyl 7-(Benzyloxy)-3-((trifluoromethanesulfonyl)-oxy)hept-2-enoate (5c) ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.36–7.24 (5H, m), 5.66 (1H, s), 3.86 (2H, t, J=5.3 Hz), 2.36 (2H, m), 1.67–1.51 (4H, m), 1.50 (9H, s). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 161.8, 156.4, 138.3, 128.3, 127.6, 127.5, 118.3 (q, J=318 Hz), 113.2, 82.3, 72.9, 69.9, 34.2, 31.8, 28.0 (3×C), 24.6. IR ($CHCl_3$) cm^{-1}: 1722, 1676, 1496, 1451, 1425, 1230, 1147. HR-EI-MS *m/z*: 382.0667 (Calcd for $C_{15}H_{17}F_3O_6S$ $[M-C_4H_8]^+$: 382.0698).

***tert*-Butyl Tetradec-2-ynoate (6a)** ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 2.29 (2H, t, J=7.3 Hz), 1.56 (2H, m), 1.49 (9H, s), 1.47–1.26 (16H, m), 0.88 (3H, t, J=6.8 Hz). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 153.0, 87.1, 82.9, 74.4, 31.9, 29.6 (2×C), 29.4, 29.3, 29.0, 28.9, 28.0 (3×C), 27.6, 22.7, 18.6, 14.1; IR ($CHCl_3$) cm^{-1}: 2235, 1699, 1457, 1370, 1284, 1159. HR-EI-MS *m/z*: 280.2416 (Calcd for $C_{18}H_{32}O_2$: 280.2402).

***tert*-Butyl 5-Phenylpent-2-ynoate (6b)** ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.32–7.19 (5H, m), 2.89 (2H, t, J=7.3 Hz), 2.58 (2H, t, J=7.3 Hz), 1.49 (9H, s). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 152.8, 139.7, 128.5, 128.3, 126.5, 85.8, 83.0, 74.9, 33.9, 28.0 (3×C), 20.8. IR ($CHCl_3$) cm^{-1}: 2239, 1699, 1603, 1455, 1370, 1285, 1159, 1074. HR-EI-MS *m/z*: 230.1316 (Calcd for $C_{15}H_{18}O_2$: 230.1307).

***tert*-Butyl 7-(Benzyloxy)hept-2-ynoate (6c)** ^{1}H-NMR (400 MHz, $CDCl_3$) δ: 7.37–7.27 (5H, m), 4.50 (2H, s), 3.49 (2H, t, J=5.9 Hz), 2.33 (2H, t, J=6.8 Hz), 1.77–1.65 (4H, m), 1.49 (9H, s). ^{13}C-NMR (100 MHz, $CDCl_3$) δ: 152.9, 138.4, 128.5, 128.4, 127.6, 127.5, 86.6, 82.9, 74.6, 72.9, 69.5, 28.8, 28.0 (3×C), 24.4, 18.4. IR ($CHCl_3$) cm^{-1}: 2235, 1699, 1495, 1477, 1454, 1370, 1283, 1159, 1079. HR-EI-MS *m/z*: 232.1112 (Calcd for $C_{14}H_{16}O_3$ $[M-C_4H_8]^+$: 232.1099).

Conflict of Interest The authors declare no conflict of interest.

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