# A Simple and Efficient Preparation of Propargylic β-Keto Esters through Transesterification

Christophe Mottet, Olivier Hamelin, Geraldine Garavel, Jean-Pierre Deprés,* and Andrew E. Greene

*LEDSS, Université Joseph Fourier, Bâtiment Chimie Recherche, 301 rue de la Chimie, BP 53, 38041 Grenoble Cedex 9, France*

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In connection with a program in our laboratory on bakkenolide synthesis,[1] we needed to prepare a propargylic β-keto ester intermediate. Propargylic β-keto esters have generally been synthesized by using diketene- or dioxinone-based methods,[2] but these procedures were ill-suited and a direct transesterification protocol was sought. To our surprise, in view of the number of methods that were available for the often-effected transesterification of β-keto esters,[3] we were unable to find any pertinent examples with propargylic alcohols,[4] even though several of the reported methods were effective with allylic alcohols.[3d−f]

Transesterification of β-keto esters with propargylic alcohols, we have discovered, in general is not trivial. Conventional acid-[5] or base-moderated[6] transesterification reactions with propargylic alcohols provided in most cases low yields of the propargylic β-keto esters; furthermore, the Taber procedure,[3c] as well as a modified version,[3d] produced considerable tarring. Fortunately, however, a mild and broadly applicable transesterification procedure, inspired by the work of Bader and collaborators,[3b] has been found for the efficient preparation of propargylic β-keto esters. The procedure involves equilibrium displacement *without catalysis* and is effective with methyl, ethyl, and *tert*-butyl β-keto esters, C-2 substituted or not, and with primary and secondary propargylic alcohols[7] (Table 1). Mechanistic studies[3e] have suggested that the transformation most likely proceeds via a ketene intermediate.

Witzeman[3e] has reported that transesterification of *tert*-butyl acetoacetate is considerably faster than the more commonly used methyl and ethyl esters. In our work, however, the *tert*-butyl β-keto esters were found to be only slightly more reactive than the methyl and ethyl esters. β-Keto esters unalkylated at C-2 (Table 1, entries 1−3) were with propargyl alcohol, as expected,[3c,d] substantially more reactive (≤24 h) than those monoakylated at C-2. 2-Cyclopentanonecarboxylate derivatives were also transformed quite rapidly (Table 1, entries 9 and 10). Transesterifications with secondary propargylic alcohols were found to be similar in rate to that with propargyl alcohol with the same β-keto ester (Table 1, entries 11 and 12 vs 5). *Finally, it is important to point out that this procedure is not at all limited to propargylic alcohols. With a variety of alcohols excellent results are obtained, generally much superior to those found in the literature (Table 2).*[3] For example, ethyl acetoacetate with benzyl alcohol and menthol (Table 2, entries 2 and 3) afforded the expected β-keto esters in purified yields of 96% and 90%, respectively, and the β-keto ester **1d**, a more difficult substrate due to C-2 substitution, with 4-pentyn-2-ol gave the anticipated product in 82% purified yield (Table 2, entry 4).

The transesterification procedure is extremely simple experimentally: a mixture of the β-keto ester and the alcohol in toluene is merely heated to reflux, with a short tube in place of the usual condenser. The equilibrium is thus shifted due to the loss of the relatively volatile methyl, ethyl, or *tert*-butyl alcohol from the reaction mixture.[8] Although the reaction times, not unexpectedly, can be lengthy with certain C-2 alkylated substrates, the reactions are nonetheless typically clean and high-yielding under these mild, neutral conditions. It is expected that the method will find general application for the preparation of these useful compounds.[9]

## Experimental Section

**General Procedure.** In a 100-mL flask fitted with a 10-cm tube, a stirred mixture of the β-keto ester (5.0 mmol) and the alcohol (1.2−5.0 equiv) in toluene (35 mL) was heated so the toluene refluxed halfway up the tube (120−130 °C, bath temperature), until no starting material remained ($^1$H NMR, 12 h to 12 days). If propargyl alcohol (bp 114−115 °C) was used it

(1) Hamelin, O.; Deprés, J.-P.; Greene, A. E.; Tinant, B.; Declerq, J.-P. *J. Am. Chem. Soc.* **1996**, *118*, 9992−9993. Hamelin, O.; Deprés, J.-P.; Heidenhain, S.; Greene, A. E. *Nat. Prod. Lett.* **1997**, *10*, 99−103 and references therein.

(2) For examples of diketene-based preparations, see: Lacey, R. N. *J. Chem. Soc.* **1954**, 827−832. Sturzenegger, A.; Zelauskus, J.; Ofner, A. *J. Org. Chem.* **1963**, *28*, 920−922. Kato, T.; Chiba, T. *Chem. Pharm. Bull.* **1975**, *23*, 2263−2267. Kinder, F. R.; Padwa, A. *Tetrahedron Lett.* **1990**, *31*, 6835−6838. Padwa, A.; Dean, D. C.; Fairfax, D. J.; Xu, S. L. *J. Org. Chem.* **1993**, *58*, 4646−4655. For examples of dioxinone-based preparations, see: Cruciani, P.; Stammler, R.; Aubert, C.; Malacria, M. *J. Org. Chem.* **1996**, *61*, 2699−2708. Weingarten, M. D.; Padwa, A. *Synlett*, **1997**, 189−190. For a method based on Rh(II)-catalyzed cyclization of 2-alkynyl 2-diazo-3-oxobutanoates, see: Padwa, A.; Kinder, F. R. *J. Org. Chem.* **1993**, *58*, 21−28.

(3) (a) For a general review on transesterification, see: Otera, J. *Chem. Rev.* **1993**, 1449−1470. (b) Bader, A. R.; Cummings, L. O.; Vogel, H. A. *J. Am. Chem. Soc.* **1951**, *73*, 4195−4197. Bader, A. R.; Vogel, H. A. *Ibid.* **1952**, *74*, 3992−3994. (c) Taber, D. F.; Amedio, J. C., Jr.; Patel, Y. K. *J. Org. Chem.* **1985**, *50*, 3618−3619. (d) Gilbert, J. C.; Kelly, T. A. *J. Org. Chem.* **1988**, *53*, 449−450. (e) Witzeman, J. S.; Nottingham, W. D. *J. Org. Chem.* **1991**, *56*, 1713−1718. (f) Balaji, B. S.; Sasidharan, M.; Kumar, R.; Chanda, B. *J. Chem. Soc., Chem. Commun.* **1996**, 707−708.

(4) (a) For a high-temperature aluminum isopropoxide-based procedure, see: Kugatova-Shemyakina, G. P.; Kazlauskas, D. A. *Bull. Soc. Acad. Sci. USSR, Div. Chem. Sci.* **1966**, 262−269 and 480−485. McAndrew, B. A.; Riezebos, G. *J. Chem. Soc., Perkin Trans. 1*, **1972**, 367−369. (b) The β-keto ester **3b** has since been synthesized in 51% yield by clay-catalyzed transesterification. This procedure also gave β-keto ester **3m** from methyl acetoacetate and benzyl alcohol in 86% yield but failed to give **3n** in the case of methyl acetoacetate and menthol: Ponde, D. E.; Deshpande, V. H.; Bulbule, V. J.; Sudalai, A.; Gajare, A. S. *J. Org. Chem.* **1998**, *63*, 1058−1063.

(5) The β-keto ester was refluxed in toluene in the presence of a catalytic amount of *p*-toluenesulfonic acid and with slow distillation. While this procedure was initially used in the synthesis of 9-acetoxyfukinanolide,[1] the method described in this paper has since been found to be far superior.

(6) The β-keto ester in propargyl alcohol was treated with 3−5 equiv of base (LiH, NaH, or $K_2CO_3$).

(7) With tertiary propargylic alcohols the yields are low, presumably because of the Carroll rearrangement of the β-keto ester products (Carroll, M. F. *J. Chem. Soc.* **1940**, 704−706; **1941**, 507−511. Kimel, W.; Cope, A. C. *J. Am. Chem. Soc.* **1943**, *65*, 1992−1998) in addition to other side reactions.

(8) In the case of propargyl alcohol, however, an excess is necessary to compensate for its volatility (bp 114−115 °C).

(9) We have found, for example, that a range of propargylic β-keto esters undergo smooth β-methylene-γ-butyrolactonization in the presence of manganese(III) acetate (manuscript in preparation).

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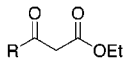
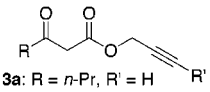
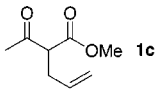
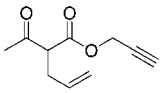
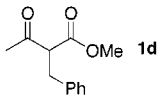
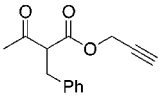
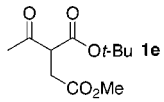
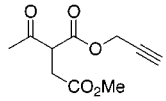
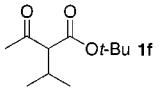
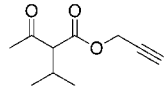
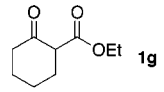
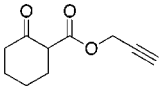
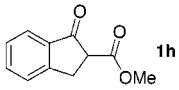
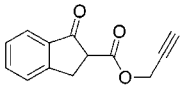
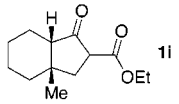
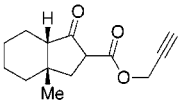
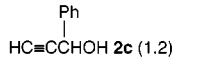
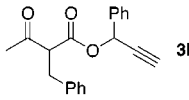
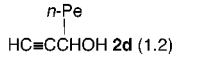
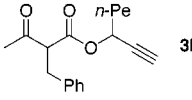
**Table 1. Propargylic β-Keto Esters by Transesterification without Catalyst**

| Entry | β-Keto ester | Propargylic alcohol (equiv) | Time (day) | Propargylic β-keto ester | Yield(%)[a] |
|---|---|---|---|---|---|
| |  | | |  | |
| 1 | **1a**: R = *n*-Pr | HC≡CCH$_2$OH **2a** (5.0) | 1 | **3a**: R = *n*-Pr, R′ = H | 91 |
| 2 | **1a** | MeC≡CCH$_2$OH **2b** (1.5) | 1 | **3b**: R = *n*-Pr, R′ = Me | 94 |
| 3 | **1b**: R = Ph | **2a** (5.0) | 1 | **3c**: R = Ph, R′ = H[b] | 96 |
| 4 |  **1c** | **2a** | 10 |  **3d** | 70 |
| 5 |  **1d** | **2a** | 12 |  **3e** | 69 |
| 6 |  **1e** | **2a** | 6 |  **3f** | 55 |
| 7 |  **1f** | **2a** | 12 |  **3g** | 79 |
| 8 |  **1g** | **2a** | 4 |  **3h**[c] | 59 |
| 9 |  **1h** | **2a** | 0.8 |  **3i** | 86 |
| 10 |  **1i** | **2a** | 1 |  **3j**[d] | 90 |
| 11 | **1d** |  **2c** (1.2) | 10 |  **3k**[e] | 85 |
| 12 | **1d** |  **2d** (1.2) | 10 |  **3l**[e] | 83 |

[a] Yields are for chromatographically and spectroscopically homogeneous, analytically pure material. [b] Reference 4b. [c] Mostly enolic. [d] 3:1 Diastereomeric ratio. [e] 1:1 Diastereomeric ratio.

was necessary to add an additional 2.5 equiv of the alcohol every 2 days. After being cooled to room temperature, the reaction mixture was directly concentrated under reduced pressure (or with propargyl alcohol worked up conventionally) to afford the crude product, which was purified by column chromatography on silica gel with increasing amounts of ethyl acetate or diethyl ether in hexane.

**Prop-2-ynyl 3-oxohexanoate (3a):** chromatography, ethyl acetate/hexane 8:92−10:90; IR 3282, 2130, 1751, 1717 cm$^{-1}$; $^1$H NMR $\delta$ 4.74 (d, $J$ = 2.5 Hz, 2 H), 3.50 (s 2 H), 2.53 (t, $J$ = 7.3 Hz, 2 H), 2.52 (t, $J$ = 2.5 Hz, 1 H), 1.64 (pseudo sext, $J$ = 7.3 Hz, 2 H), 0.93 (t, $J$ = 7.3 Hz, 3 H); $^{13}$C NMR $\delta$ 202.2, 166.5, 77.1, 75.0, 52.6, 48.9, 44.9, 16.9, 13.5; mass spectrum (EI) $m/z$ 168 (M$^+$, 2.9), 71 (100), 39 (HC=C=CH$_2$, 53). Anal. Calcd for $C_9H_{12}O_3$: C, 64.27; H, 7.19. Found: C, 64.01; H, 7.14.

**But-2-ynyl 3-oxohexanoate (3b):** chromatography, ethyl acetate/hexane 6:94−7.5:92.5; IR 2244, 1748, 1718 cm$^{-1}$; $^1$H NMR $\delta$ 4.70 (q, $J$ = 2.4 Hz, 2 H), 3.47 (s, 2 H), 2.53 (t, $J$ = 7.2 Hz, 2 H), 1.85 (t, $J$ = 2.0 Hz, 3 H), 1.63 (pseudo sext, $J$ = 7.4 Hz, 2 H), 0.93 (t, $J$ = 7.4 Hz, 3 H). $^{13}$C NMR $\delta$ 202.3, 166.7, 83.7, 72.7, 53.5, 52.2, 49.0, 16.9, 13.5, 3.6; mass spectrum (EI) $m/z$ 182 (M$^+$, 1.4), 71 (66), 53 (HC=C=CCH$_3$, 100). Anal. Calcd for $C_{10}H_{14}O_3$: C, 65.91; H, 7.74. Found: C, 65.46; H,7.79.

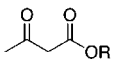
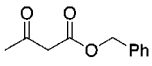
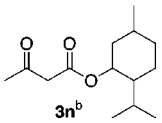
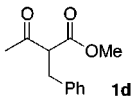
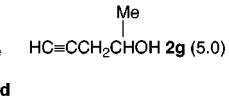
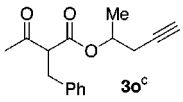
**Prop-2-ynyl 3-phenyl-3-oxopropanoate (3c):**[4b] chromatography, ethyl acetate/hexane 10:90−12:88; IR 3290, 2131, 1747, 1687 cm$^{-1}$; $^1$H NMR $\delta$ 8.0−7.6 (m, 5H), 4.81 (d, $J$ = 2.4 Hz, 2 H), 4.05 (s, 2 H), 2.49 (t, $J$ = 2.2 Hz, 1 H); $^{13}$C NMR $\delta$ 191.9, 166.7, 133.9, 128.8, 128.6, 128.5, 75.4, 52.8, 45.5.

**Prop-2-ynyl 2-allyl-3-oxobutanoate (3d):** chromatography, ether/hexane 10:90−12:88; IR 3290, 2130, 1750, 1718 cm$^1$; $^1$H NMR $\delta$ 5.86−5.62 (m, 1 H), 5.16−5.04 (m, 2 H), 4.74 (d, $J$ = 2.5 Hz, 2 H), 3.58 (t, $J$ = 7.4 Hz, 1H), 2.66−2.56 (m, 2 H), 2.51 (t, $J$ = 2.5 Hz, 1 H), 2.26 (s, 3 H); $^{13}$C NMR $\delta$ 201.7, 166.5, 133.9, 117.8, 77.0, 75.5, 58.9, 52.7, 32.1, 29.2; mass spectrum (EI) $m/z$ 181 (M$^+$ + 1, 2.7), 180 (M$^+$, 2), 43 (100), 39 (HC=C=CH$_2$, 71). Anal. Calcd for $C_{10}H_{12}O_3$: C, 66.65; H, 6.71. Found: C, 66.43; H, 6.49.

**Prop-2-ynyl 2-benzyl-3-oxobutanoate (3e):** chromatography, ethyl acetate/hexane 10:90−12:88; IR 3287, 2128, 1745, 1720 cm$^{-1}$; $^1$H NMR $\delta$ 7.32−7.12 (m, 5 H), 4.67 (pseudo t, $J$ = 2.5 Hz, 2 H), 3.82 (pseudo t, $J$ = 7.6 Hz, 1 H), 3.17 (pseudo d, $J$ = 7.6 Hz, 2 H), 2.47 (pseudo t, $J$ = 2.5 Hz, 1 H), 2.18 (s, 3H); $^{13}$C NMR $\delta$ 201.7, 168.3, 137.8, 128.8, 128.6, 126.8, 76.9, 75.5, 60.9, 52.7, 33.9, 29.6; mass spectrum (EI) $m/z$ 230 (M$^+$, 4.7), 187 (61), 43 (100), 39 (HC=C=CH$_2$, 23). Anal. Calcd for $C_{14}H_{14}O_3$: C, 73.02; H, 6.13. Found: C, 73.24; H, 6.20.

**Methyl prop-2-ynyl 2-acylbutanedioate (3f):** chromatography, ethyl acetate 17:83−20:80; IR 3282, 2130, 1730, 1720 cm$^{-1}$; $^1$H NMR $\delta$ 4.76 (d, $J$ = 2.4 Hz, 1 H), 4.75 (d, $J$ = 2.4 Hz, 1 H), 4.03 (X of ABX, dd, $J$ = 7.8, 6.6 Hz, 1 H), 3.69 (s, 3 H),

**Table 2. Transesterification of β-Keto Esters with Various Alcohols**

| Entry | Substrate | Alcohol (equiv) | Time (day) | Product | Yield (%)[a] |
|---|---|---|---|---|---|
| |  | | |  | |
| 1 | **1j**: R = O*t*-Bu | $PhCH_2OH$ **2e** (1.2) | 0.5 | **3m** | 96 |
| 2 | **1k**: R = Et | **2e** (1.2) | 0.7 | **3m**[b] | 96 |
| 3 | **1k** | Menthol **2f** (1.2) | 0.5 |  **3n**[b] | 90 |
| 4 |  **1d** |  **2g** (5.0) | 12 |  **3o**[c] | 82 |

[a] Yields are for chromatographically and spectroscopically homogeneous, analytically pure material. [b] References 3f, 4b. [c] 1: 1 Diastereomeric ratio.

2.99 (A of ABX, dd, $J$ = 18.0, 7.8 Hz, 1 H), 2.85 (B of ABX, dd, $J$ = 18.0, 6.6 Hz, 1 H), 2.54 (pseudo t, $J$ = 2.4 Hz, 1 H), 2.38 (s, 3 H); $^{13}$C NMR $\delta$ 200.9, 171.6, 167.7, 75.7, 54.4, 53.1, 52.1, 32.0, 29.9; mass spectrum (CI) $m/z$ 230 ($M^+$ + 18, 100), 213 ($M^+$ + 1, 78). Anal. Calcd for $C_{10}H_{12}O_5$: C, 56.60; H, 5.70. Found: C, 56.31; H, 5.84.

**Prop-2-ynyl 2-isopropyl-3-oxobutanoate (3g):** chromatography, ether/hexane 8:92−10:90; IR 3282, 2130, 1746, 1717 cm$^{-1}$; $^1$H NMR $\delta$ 4.73 (d, $J$ = 2.5 Hz, 2 H), 3.25 (d, $J$ = 9.4 Hz, 1 H), 2.51 (t, $J$ = 2.5 Hz, 1 H), 2.54−2.34 (m, 1 H), 2.24 (s, 3 H), 1.00 (d, $J$ = 6.7, 3 H), 0.95 (d, $J$ = 6.7 Hz, 3H); $^{13}$C NMR $\delta$ 202.4, 168.4, 75.3, 67.1, 52.4, 29.3, 28.8, 20.5, 20.3; mass spectrum (CI) $m/z$ 200 ($M^+$ + 18), 183 ($M^+$ + 1, 100). Anal. Calcd for $C_{10}H_{14}O_3$: C, 65.91; H, 7.74. Found: C, 66.31; H, 7.47.

**Prop-2-ynyl cyclohexanone-2-carboxylate (3h):** chromatography, ether/hexane 6:94; IR 3290, 2130, 1752, 1711, 1655, 1616 cm$^{-1}$; $^1$H NMR (enolic form) $\delta$ 11.96 (s, 1 H), 4.77 (d, $J$ = 2.5 Hz, 2 H), 2.50 (t, $J$ = 2.5 Hz, 1 H), 2.34−2.20 (m, 4 H), 1.76−1.50 (m, 4 H); $^{13}$C NMR (enolic form) $\delta$ 173.3, 171.7, 97.3, 77.9, 74.8, 51.6, 29.2, 22.3, 21.8; mass spectrum (CI) $m/z$ 198 ($M^+$ + 18, 4), 181 ($M^+$ + 1, 10). Anal. Calcd for $C_{10}H_{12}O_3$: C, 66.65; H, 6.71. Found: C, 66.59; H, 6.84.

**Prop-2-ynyl 1-indanone-2-carboxylate (3i):** 25% enolic form; chromatography, ethyl acetate/hexane 15:85−20:80; mp 44−45 °C; IR 3290, 2130, 1748, 1713 cm$^{-1}$; $^1$H NMR (only ketone form) $\delta$ 7.80−7.35 (m, 4 H), 4.78 (pseudo t, $J$ = 2.3 Hz, 2 H), 3.78 (X of ABX, dd, $J$ = 8.2, 4.3 Hz, 1 H), 3.58 (B of ABX, dd, $J$ = 17.3, 4.3 Hz, 1 H), 3.40 (A of ABX, dd, $J$ = 17.3, 8.2 Hz, 1 H), 2.51, (pseudo t, $J$ = 2.5 Hz, 1 H); $^{13}$C NMR (only ketone form) $\delta$ 198.7, 168.4, 153.4, 135.5, 127.8, 126.5, 124.7, 75.4, 75.1, 52.9, 30.2; mass spectrum (EI) $m/z$ 214 ($M^+$, 22), 130 (93), 71 (100), 39 (HC≡C═$CH_2$, 40). Anal. Calcd for $C_{13}H_{10}O_3$: C, 72.89; H, 4.71. Found: C, 72.77; H, 4.80.

**Prop-2-ynyl 1-methylbicyclo[4.3.0]nonane-7-one-8-carboxylate (3j):** 3:1 mixture of diastereomers; chromatography, ethyl acetate/hexane 1:9; IR 3290, 2130, 1747, 1730 cm$^{-1}$; $^1$H NMR (major diastereomer) $\delta$ 4.73 (pseudo t, $J$ = 2.2 Hz, 2 H), 3.37 (pseudo t, $J$ = 9.8 Hz, 1 H), 2.49 (pseudo t, $J$ = 2.5 Hz, 1 H), 2.14−2.00 (m, 3 H), 1.6−1.2 (m, 8 H), 1.25 (s, 3 H); $^{13}$C NMR (major diastereomer) $\delta$ 211.4, 169.2, 75.3, 55.8, 52.7, 52.2, 39.4, 36.6, 34.2, 25.1, 22.4, 21.4, 20.7; mass spectrum (EI) $m/z$ 234 ($M^+$, 8), 219 (32), 81 (100), 39 (HC═C═$CH_2$, 86). Anal. Calcd for $C_{14}H_{18}O_3$: C, 71.77; H, 7.74. Found: C, 71.77; H, 7.84.

**1-Phenylprop-2-ynyl 2-benzyl-3-oxobutanoate (3k):** 1:1 mixture of diastereomers; chromatography, ethyl acetate/hexane 8:92−10:90; IR 3290, 2129, 1745, 1718 cm$^{-1}$; $^1$H NMR $\delta$ 7.5−7.1 (m, 10 H), 6.44 (pseudo t, $J$ = 6.5 Hz, 1 H), 3.16 (m, 2 H), 2.68 and 2.64 (2 d, $J$ = 2.2 Hz, 1 H), 2.18 and 2.08 (2 s, 3 H); mass spectrum (EI) $m/z$ 306 ($M^+$, 1.5), 115 (HC═C═CHPh, 97), 43 (100). Anal. Calcd for $C_{19}H_{24}O_3$: C, 78.41; H, 5.92. Found: C, 78.40; H, 5.78.

**1-Pentylprop-2-ynyl 2-benzyl-3-oxobutanoate (3l):** 1:1 mixture of diastereomers; chromatography, ethyl acetate/hexane 8:92−12:88; IR 3288, 2125, 1748, 1718 cm$^{-1}$; $^1$H NMR $\delta$ 7.35−7.10 (m, 5 H), 5.40−5.25 (m, 1 H), 3.86−3.70 (m, 1 H), 3.20−3.10 (m, 2 H), 2.44 (m, 1 H), 2.22 and 2.18 (2 s, 3 H), 1.80−1.55 (m, 2 H), 1.5−1.1 (m, 6 H), 0.95−0.78 (m, 3 H); mass spectrum (EI) $m/z$ 300 ($M^+$, 7.5), 149 (50), 43 (100). Anal. Calcd for $C_{19}H_{24}O_3$: C, 75.97; H, 8.05. Found: C, 75.84; H, 8.09.

**1-Methylbut-3-ynyl 2-benzyl-3-oxobutanoate (3o):** 1:1 mixture of diastereomers; chromatography, ethyl acetate/hexane 1:9; IR 3290, 2122, 1738, 1715 cm$^{-1}$; $^1$H NMR $\delta$ 7.35−7.15 (m, 5 H), 5.00 (q, $J$ = 6.2 Hz, 1 H), 3.79 and 3.78 (2 pseudo t, $J$ = 2.7 Hz, 1 H), 3.20−3.10 (m, 2 H), 2.41 and 2.35 (2 dd, $J$ = 5.9, 2.6 Hz, 2 H), 2.21 and 2.20 (2 s, 3 H), 1.99 and 1.95 (2 pseudo t, $J$ = 2.6 Hz, 1 H), 1.29 and 1.21 (2 d, $J$ = 6.3 Hz, 3 H); $^{13}$C NMR $\delta$ 202.1, 168.5, 138.0, 128.8, 128.5, 126.6, 79.3, 70.8, 69.7, 61.3, 33.8, 29.5, 25.3, 18.8, 18.7; mass spectrum (EI) $m/z$ (258, $M^+$, 7), 215 (51), 149 (70), 43 (100). Anal. Calcd for $C_{16}H_{18}O_3$: C, 74.39; H, 7.02. Found: C, 74.33; H, 7.06.

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