A New Synthesis of 3-Isochromanone Derivatives Based on the Reaction of *o*-Acylbenzyllithiums with Ethyl Chloroformate

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Synopsis. A new method for the preparation of 3-isochromanone derivatives is reported. The method consists of the ethoxycarbonylation of *o*-acylbenzyllithiums with ethyl chloroformate and the subsequent $NaBH_4$ reduction of the resulting *o*-acylphenylacetic acid derivatives.

1,4-Dihydro-3*H*-2-benzopyran-3-ones (3-isochromanones) have recently attracted much attention because of their significant usefulness in organic synthesis. For example, pyrolysis of these derivatives provides a convenient method for the generation of *o*-quinodimethanes.[1] 3-Isochromanones have also been used as precursors in heterocyclic syntheses.[2] Several methods for synthesizing these compounds have been reported: e. g. 1) Baeyer–Villiger reaction of 2-indanones,[3] 2) *o*-hydroxyalkylation of phenylacetic acid derivative,[4] 3) tandem electrocyclic-sigmatropic reaction of benzocyclobutenes,[5] and 4) reaction of bromoarenes with α-lithio nitriles under aryne-forming conditions followed by acidic hydrolysis.[6] We report here, as the results of our continuing studies in exploring utilities of *o*-acylbenzyllithiums,[7] a new efficient method to synthesize 3-isochromanones carrying substituents at the 1- and/or 4-positions, in which the key step involves ethoxycarbonylation of these lithiated intermediates with ethyl chloroformate. There have been few reports concerning the preparation of 1,4-disubstituted 3-isochromanones prior to this report.

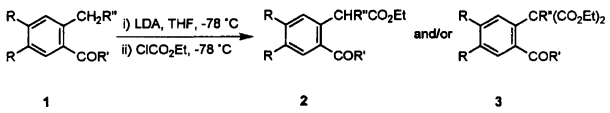
2-Alkylphenyl ketones **1** for the present experiments were readily available commercially (**1c**) or by the reported methods (**1a**,[7] **1b**,[7] and **1f**[8]). Lithiation of **1c** followed by treatment with iodomethane gave 2-ethylphenyl phenyl ketone (**1d**). 2-Methoxyphenyl 2-methylphenyl ketone (**1e**) was prepared by the reaction of 2-methylphenylmagnesium bromide with 2-methoxybenzaldehyde followed by oxidation of the resulting alcohol with pyridinium chlorochromate (PCC).

Shown in Eq. 1 is the ethoxycarbonylation process. Thus, treatment of 2-alkylphenyl ketones **1** with lithium diisopropylamide (LDA) in tetrahydrofuran at −78 °C generated 2-acylbenzyllithiums, which were allowed to react with ethyl chloroformate. As shown in Entries a—e, Table 1, the ethoxycarbonylated products **2** were obtained in 39—48% yields, although the bis(ethoxycarbonyl)ated products **3** were produced at the same time in the cases of Entries a, c, and e in 23—27% yields. Only the bis(ethoxycarbonyl)ated product **3f** was obtained in 63% yield from the reaction of 2-(3,4-dimethoxybenzoyl)-4,5-dimethoxybenzyllithium and ethyl chloroformate (Entry f).

Table 1. Results of the Ethoxycarbonylation of 2-Alkylphenyl Ketones **1**

Entry	R	R′	R″	Yield/%[a]	
				2	**3**
a	H	*t*-Bu	H	48	23
b	H	*t*-Bu	Me	40	—
c	H	C_6H_5	H	39	27
d	H	C_6H_5	Me	39	—
e	H	2-$MeOC_6H_4$	H	43	25
f	MeO	3,4-$(MeO)_2C_6H_3$	H	—	63

a) Based on isolated products.

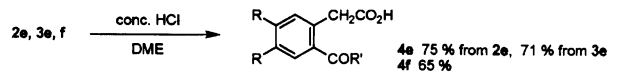


1 2 3

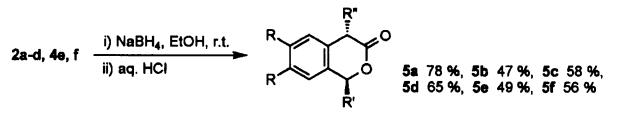
(1)

The keto esters **2a—d** were then transformed into 3-isochromanone derivatives **5a—d** by reduction with sodium borohydride in ethanol at room temperature (Eq. 3). In order to examine the stereochemistry of compound **5b** and **d** NOE measurements of the ^1H NMR were performed. Thus, irradiation of the signal at $\delta=3.78$ due to 4-H of **5b** resulted in an enhancement of the area of the signal at $\delta=1.03$ due to 1-*t*-Bu (6.3%). This result indicates that compound **5b** had a trans stereochemistry. The stereochemistry of **5d** was deduced to be trans, since an enhancement of the area of the signal at $\delta=1.70$ due to 4-Me (3.3%) when the signal at $\delta=6.31$ due to 1-H was irradiated. The reduction of keto ester **2e** under the same conditions as the cases of keto ester **2a—d** did not proceed because of decreasing reactivity of the keto group, which may be attributable to the presence of the methoxy-substituent, and heating of the reaction mixture gave the complicated reaction mixture. This problem, however, was overcome by first transforming this keto ester to keto acid (Eq. 2), which was then cyclized to 3-isochromanone (Eq. 3). Thus, keto ester **2e** was treated with concentrated hydrochloric acid in 1,2-dimethoxyethane (DME) overnight at room temperature to afford keto carboxylic acid **4e**, which was produced by a similar treatment of keto diester **3e** as a result of decarboxylation. The hydrolysis of keto ester **3f** was sluggish under the same conditions as above and considerable amount of the starting material was remained. How-

ever, prolonged reaction time (3 days) gave the desired product **4f** in good yield. The keto carboxylic acids **4e** and **f** thus obtained were then treated with sodium borohydride in ethanol to give the corresponding 3-isochromanone derivatives **5e** and **f** after acidification with concentrated hydrochloric acid (Eq. 3).



(2)



(3)

Since the present method for the preparation of 3-isochromanone derivatives starting with readily available 2-alkylphenyl ketones is operationally simple, it may have potential in the synthesis of compounds of this class, especially 1,4-disubstituted derivatives which are hard to obtain by conventional methods.

Experimental

General Melting points were recorded with a Laboratory Devices MEL-TEMP II melting point apparatus and uncorrected. IR spectra were recorded with a Perkin–Elmer 1600 Series FT IR spectrometer. The ^{1}H NMR spectra were taken on JEOL JNM-PMX 60 (60 MHz) or JEOL JNM-GX270 FT NMR (270 MHz) spectrometers using Me_4Si as a standard. The high- and low-resolution mass spectra were determined with a JEOL JMS-GX 300 spectrometer. TLC was performed with Merck silica gel 60 GF_{254}. All of the solvents used were dried over appropriate drying agents and distilled under argon prior to use. Compounds **1a**,[7] **1b**,[7] and **1f**[8] were prepared according to the appropriate reported procedures.

2-Ethylphenyl Phenyl Ketone (1d). To a stirred solution of LDA (20 mmol) in THF (40 ml) at −78 °C, which was generated by the standard method, was added dropwise 2-methylphenyl phenyl ketone (**1c**) (2.0 g, 10 mmol). After the mixture was stirred for 10 min at the same temperature, iodomethane (2.8 g, 20 mmol) was added to the resulting red solution of (2-benzoyl)benzylithium. The red color of the carbanion was disappeared gradually (ca. 2 h), and then the resulting mixture was quenched by the addition of iced aq NH_4Cl and extracted Et_2O three times. The extract was washed with brine, dried over anhyd $MgSO_4$ and evaporated. Separation of the residue by chromatography on SiO_2 (1 : 10 EtOAc–hexane) gave a crude oily product, which was further purified by distillation, affording pure **1d** (1.0 g, 48%): bp 180 °C (bath temp)/5 Torr# (lit,[9] bp 165—166 °C/11 Torr); IR (neat) 1667 cm^{-1}; ^{1}H NMR (60 MHz, $CDCl_3$) δ=1.14 (3H, t, J=7.3 Hz), 2.65 (2H, q, J=7.3 Hz), 7.1—7.5 (7H, m), and 7.6—7.8 (2H, m).

(2-Methoxyphenyl)(2-methylphenyl)methanol. To a stirred solution of 2-methylphenylmagnesium bromide at 0 °C, which was prepared in situ from magnesium turning (1.2 g, 50 mG) and 1-bromo-2-methylbenzene (7.9 g, 46 mmol) Et_2O (35 ml), was added dropwise a solution of 2-methoxybenzaldehyde (4.3 g, 31 mmol) in Et_2O (20 ml) over a period of 20 min. After the mixture was stirred overnight at room temperature it was quenched by the addition of iced 10% aq H_2SO_4. The organic phase was separated and the aqueous phase was extracted with Et_2O twice. The combined extract was washed with brine and dried over anhyd $MgSO_4$. The solvent was evaporated to give a solid residue which could be recrystallized from petroleum ether to afford (2-methyoxyphenyl)(2-methylphenyl)methanol (4.7 g, 66%): mp 61—62 °C; IR (KBr disk) 3190 cm^{-1}; ^{1}H NMR (60 MHz, $CDCl_3$) δ=2.20 (3H, s), 2.24 (1H, s), 3.86 (3H, s), 6.17 (1H, s), and 6.7—7.5 (8H, m); MS m/z (%) 228 (M^+, 71), 213 (95), and 119 (100). Found: C, 78.66; H, 6.96%. Calcd for $C_{15}H_{16}O_2$: C, 78.92; H, 7.06%.

2-Methoxyphenyl 2-Methylphenyl Ketone (1e). A mixture of the alcohol obtained above (4.7 g, 21 mmol) and PCC (14 g, 64 mmol) in CH_2Cl_2 (500 ml) containing 25 g of Celite 545 was stirred overnight at room temperature. After filtering the resulting mixture the filtrate was washed with 5% aq HCl and dried over anhyd $MgSO_4$. The solvent was removed in vacuo and the residue was subjected to a short column chromatography on SiO_2 (CH_2Cl_2). After concentration of the eluent, the residue was purified by recrystallization from hexane, affording **1e** (3.3 g, 70%): mp 71—72 °C; IR (KBr disk) 1657 cm^{-1}; ^{1}H NMR (60 MHz, $CDCl_3$) δ=2.47 (3H, s), 3.60 (3H, s), and 6.75—7.6 (8H, m); MS m/z (%) 226 (M^+, 33) and 225 (100). Found: C, 79.51; H, 6.19%. Calcd for $C_{15}H_{14}O_2$: C, 79.62; H, 6.24%.

Ethyl 2-[2-(2,2-Dimethylpropanoyl)phenyl]acetate (2a). Typical Procedure for the Ethoxycarbonylation of 2-Alkylphenyl Ketones 1. To a stirred solution of 2-(2,2-dimethylpropanoyl)benzylithium (1.5 mmol) in THF (7 ml) at −78 °C, which was generated by the previously reported method,[7] was added ethyl chloroformate (163 mg, 1.5 mmol). The characteristic red color of the carbanion was disappeated immediately, and then the resulting mixture was poured into iced aq NH_4Cl and extracted with Et_2O three times. The combined extract was washed with brine, dried over anhyd $MgSO_4$, and concentrated in vacuo to give a residue which was purified by PLC on SiO_2 (1 : 3 EtOAc–hexane) to afford **2a** (179 mg, 48%) and diethyl 2-[2-(2,2-dimethylpropanoyl)phenyl]propanedionate (**3a**) (110 mg, 23%).

2a: R_f 0.58; IR (neat) 1736 and 1684 cm^{-1}; ^{1}H NMR (270 MHz, $CDCl_3$) δ=1.25 (3H, t, J=7.3 Hz), 1.38 (9H, s), 3.64 (2H, s), 4.15 (2H, q, J=7.3 Hz), and 7.25—7.45 (4H, m); MS m/z (%) 249 (M+1, 0.13), 203 (7.8), 191 (64), and 135 (100). Found: C, 72.80; H, 8.33%. Calcd for $C_{15}H_{20}O_3$: C, 72.55; H, 8.12%.

3a: R_f 0.50; IR(neat) 1752, 1725, and 1685 cm^{-1}; ^{1}H NMR (60 MHz, $CDCl_3$) δ=1.25 (6H, t, J=7.0 Hz), 1.28 (9H, s), 4.21 (4H, q, J=7.0 Hz), 4.58 (1H, s), and 7.25—7.6 (4H, m); MS m/z (%) 321 (M+1, 9.7), 263 (32), and 217 (100). Found: C, 67.55; H, 7.78%. Calcd for $C_{18}H_{24}O_5$: C, 67.48; H, 7.55%.

Physical properties as well as spectral and analytical data of products listed in Table 1 are as follow.

Ethyl 2-[2-(2,2-Dimethylpropanoyl)phenyl]propanoate (2b): R_f 0.37 (1 : 10 EtOAc–hexane); IR (neat) 1737 and 1688 cm^{-1}; ^{1}H NMR (60 MHz, $CDCl_3$) δ=1.18

1 Torr=133.322 Pa.

(3H, t, J=7.0 Hz), 1.29 (9H, s), 1.44 (3H, d, J=7.0 Hz), 3.53 (1H, q, J=7.0 Hz), 4.11 (2H, q, J=7.0 Hz), and 7.15—7.45 (4H, m); MS m/z (%) 262 (M^+, 3.5), 217 (15), 205 (90), and 149 (100). Found: C, 73.11; H, 8.52%. Calcd for $C_{16}H_{22}O_3$: C, 73.25; H, 8.45%.

Ethyl 2-(2-Benzoylphenyl)acetate (2c): R_f 0.50 (1:3 EtOAc–hexane); IR (neat) 1735 and 1663 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ=1.11 (3H, t, J=7.3 Hz), 3.88 (2H, s), 4.02 (2H, q, J=7.3 Hz), 7.05—7.65 (7H, m), and 7.7—7.85 (2H, m); MS m/z (%) 268 (M^+, 12), 239 (22), 223 (23), and 194 (100). Found: C, 76.19; H, 6.10%. Calcd for $C_{17}H_{16}O_3$: C, 76.10; H, 6.01%.

Diethyl 2-(2-Benzoylphenyl)propanedioate (3c): R_f 0.36 (1:3 EtOAc–hexane); IR (neat) 1751, 1736, and 1664 cm^{-1}; ^1H NMR (60 MHz, $CDCl_3$) δ=1.21 (6H, t, J=7.3 Hz), 4.19 (4H, q, J=7.3 Hz), 5.09 (1H, s), and 7.15—7.9 (9H, m); MS m/z (%) 340 (M^+, 3.2), 294 (8.7), and 266 (100). Found: C, 70.66; H, 5.96%. Calcd for $C_{20}H_{20}O_5$: C, 70.57; H, 5.92%.

Ethyl 2-(2-Benzoylphenyl)propanoate (2d): R_f 0.24 (1:10 EtOAc–hexane); IR (neat) 1732 and 1666 cm^{-1}; ^1H NMR (60 MHz, $CDCl_3$) δ=1.06 (3H, t, J=7.3 Hz), 1.45 (3H, d, J=7.0 Hz), 3.87 and 3.93 (combined 3H, 2q, J=7.0 and 7.3 Hz), 7.1—7.5 (7H, m), and 7.7—7.9 (2H, m); MS m/z (%) 282 (M^+, 5.5), 236 (22), and 208 (100). Found: m/z 282.1255. Calcd for $C_{18}H_{18}O_3$: M, 282.1256.

Ethyl 2-[2-(2-Methoxybenzoyl)phenyl]acetate (2e): R_f 0.17 (1:5 EtOAc–hexane); IR (neat) 1731 and 1667 cm^{-1}; ^1H NMR (60 MHz, CCl_4) δ=1.26 (3H, t, J=7.4 Hz), 3.77 (3H, s), 4.03 (2H, s), 4.22 (2H, q, J=7.4 Hz), and 6.9—7.7 (8H, m); MS m/z (%) 298 (M^+, 6.8), 269 (26), 225 (60), and 224 (100). Found: m/z 298.1202. Calcd for $C_{18}H_{18}O_4$: M, 298.1205.

Diethyl 2-[2-(2-Methoxybenzoyl)phenyl]propanedioate (3e): R_f 0.10 (1:5 EtOAc–hexane); IR (neat), 1738, 1731, and 1667 cm^{-1}; ^1H NMR (60 MHz, CCl_4) δ= 1.31 (6H, t, J=7.4 Hz), 3.74 (3H, s), 4.36 (2H, q, J=7.4 Hz), 5.50 (1H, s), and 7.0—7.8 (4H, m); MS m/z (%) 370 (M^+, 6.8), 325 (18), and 297 (100). Found; m/z 370.1405. Calcd for $C_{21}H_{22}O_6$: M, 370.1416.

The ethoxycarbonylation of **1f** was carried out by the use of 3 molar amounts each of LDA and ethyl chloroformate to give the best result.

Diethyl 2-[2-(3,4-Dimethoxybenzoyl)-4,5-dimethoxyphenyl]propanedioate (3f): R_f 0.23 (1:2 EtOAc–hexane); IR 1746, 1731, and 1644 cm^{-1}; ^1H NMR (60 MHz, CCl_4) δ=1.22 (6H, t, J=7.4 Hz), 3.76 (3H, s), 3.86 (3H, s), 3.93 (3H, s), 4.12 (4H, q, J=7.4 Hz), 4.83 (1H, s), and 6.6—7.3 (5H, m); MS m/z (%) 460 (M^+, 6.1) and 386 (100). Found: m/z 460.1747. Calcd for $C_{24}H_{28}O_9$: M, 460.1733.

2-[2-(2-Methoxybenzoyl)phenyl]acetic Acid (4e). A solution of **2e** (0.25 g, 0.85 mmol) in 1,2-dimethoxyethane (3 ml) and concd HCl (1 ml) was stirred overnight at room temperature. The resulting mixture was poured into water (15 ml) end extracted with Et_2O three times. The extract was washed with water and dried over anhyd $MgSO_4$. The solvent was evaporated and the resulting residue was recrystallized from hexane–Et_2O to give **4e** (0.17 g, 75%). Compound **4e** was obtained from **3e** by the same procedure.

4e: mp 150—151 °C; IR (KBr disk) 3500—2600, 1708, and 1654 cm^{-1}; ^1H NMR (60 MHz, $CDCl_3$) δ=3.77 (3H, s), 4.05 (2H, s), 7.0—7.75 (8H, m), and 8.3—8.5 (1H, br); MS m/z (%) 270 (M^+, 20), 225 (94), and 224 (100). Found: C, 70.81; H, 5.35%. Calcd for $C_{16}H_{14}O_4$: C, 71.10; H, 5.22%.

2-[2-(3,4-Dimethoxybenzoyl)-4,5-dimethoxyphenyl]acetic Acid (4f). The hydrolysis of **3f** for 3 d in a similar manner as above gave **4f**: R_f 0.15 (1:1 EtOAc–hexane); IR (neat) 3500—2600, 1718, and 1648 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ=3.70 (2H, s), 3.81 (3H, s), 3.97 (3H, s), 3.98 (3H, s), 6.90 (1H, d, J=8.4 Hz), 6.98 (1H, s), 7.03 (1H, m), 7.38 (1H, d, J=8.4 Hz), 7.51 (1H, s), and 8.5—9.0 (1H, br); MS m/z (%) 360 (M^+, 6.9), 342 (21), and 285 (100). Found: m/z 360.1209. Calcd for $C_{19}H_{20}O_7$: M, 360.1229.

1-*t*-Butyl-1,4-dihydro-3*H*-2-benzopyran-3-one (5a). General Procedure for the Conversion of Keto Esters 2 into 2-Isochromanone Derivatives 5. A mixture of **2a** (0.12 g, 0.5 mmol) and $NaBH_4$ (18 mg, 0.5 mmol) in EtOH (5 ml) was stirred overnight at room temperature, and then 5% aq HCl (10 ml) was added to it. The resulting mixture was concentrated under reduced pressure and extracted with Et_2O three times. The combined extract was washed with brine, dried over anhyd $MgSO_4$, and evaporated to give a residue, which was purified by PLC on SiO_2 to give **5a** (80 mg, 78%): R_f 0.31 (1:3 EtOAc–hexane); IR (neat) 1737 cm^{-1}; ^1H NMR (60 MHz, $CDCl_3$) δ=1.02 (9H, s), 3.60 (1H, d, J=23 Hz), 3.89 (1H, d, J=23 Hz), 5.11 (1H, s), and 7.05—7.35 (4H, m); MS m/z (%) 205 (M+1, 4.5), 204 (M^+, 4.0), 161 (9.5), and 148 (100). Found: C, 76.20; H, 7.91%. Calcd for $C_{13}H_{16}O_2$: C, 76.44; H, 7.90%.

***trans*-1-*t*-Butyl-1,4-dihydro-4-methyl-3*H*-2-benzopyran-3-one (5b):** R_f 0.20 (1:10 EtOAc–hexane); mp 78—80 °C (Et_2O–hexane); IR (nujol) 1721 cm^{-1}; ^1H NMR (60 MHz, $CDCl_3$) δ=1.03 (9H, s), 1.68 (3H, d, J=7.0 Hz), 3.78 (1H, q, J=7.0 Hz), 5.09 (1H, s), and 7.15—7.35 (4H, m); MS m/z (%) 218 (M^+, 2.0) and 162 (100). Found: m/z 218.1290. Calcd for $C_{14}H_{18}O_2$: M, 218.1307.

1, 4- Dihydro- 1- phenyl- 3*H*- 2- benzopyran- 3- one (5c):[10] R_f 0.29 (1:3 EtOAc–hexane); mp 76—77 °C (Et_2O–hexane) (lit,[10] 75 °C).

***trans*-1-Phenyl-1,4-dihydro-4-methyl-3*H*-2-benzopyran-3-one (5d):** R_f 0.12 (1:7 EtOAc–hexane); mp 117—118.5 °C (Et_2O–hexane); IR (KBr disk) 1746 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$) δ=1.70 (3H, d, J=6.9 Hz), 3.80 (1H, q, J=6.9 Hz), 6.31 (1H, s), 6.66 (1H, d, J=8.3 Hz), and 7.2—7.45 (8H, m); MS m/z (%) 238 (M^+, 8.3), 194 (43), and 179 (100). Found: C, 80.48; H, 5.70%. Calcd for $C_{16}H_{14}O_2$: C, 80.65; H, 5.92%.

1,4-Dihydro-1-(2-methoxyphenyl)-3*H*-2-benzopyran-3-one (5e). A mixture of **4d** (130 mg, 0.48 mmol) and $NaBH_4$ (36 mg, 0.96 mmol) in EtOH (10 ml) was stirred for 2 h at room temperature. After acidification of the resulting mixture with concd HCl, it was diluted with water (10 ml) and evaporated. The residue was extracted with Et_2O three times and the combined extract was dried over anhyd $MgSO_4$. Evaporation of the solvent gave a residue, which was purified by PLC on SiO_2 to afford **5e** (60 mg, 49%): R_f (1:2 EtOAc–hexane); mp 151—152 °C (hexane–$CHCl_3$); IR (KBr disk) 1746 cm^{-1}; ^1H NMR (60 MHz, $CDCl_3$) δ=3.81 (2H, s), 3.84 (3H, s), 6.79 (1H, s), and 6.85—7.3 (8H, m); MS m/z (%) 254 (M^+, 52), 226 (63), and 209 (100). Found: C, 75.32; H, 5.52%. Calcd for $C_{16}H_{14}O_3$: C, 75.58; H, 5.55%.

1-(3,4-Dimethoxyphenyl)-1,4-dihydro-6,7-dimethoxy-3*H*-2-benzopyran-3-one (5f). This compound was prepared in a similar manner as above. **5f**: R_f 0.63 (1:1

EtOAc–hexane); IR (neat) 1731 cm^{-1}; ^1H NMR (60 MHz, $CDCl_3$) δ=3.42 (2H, s), 3.69 (3H, s), 3.81 (9H, s), 6.42 (1H, s), and 6.6—6.9 (5H, m); MS m/z (%) 344 (M^+, 43) and 269 (100). Found: m/z 344.1255. Calcd for $C_{19}H_{20}O_6$: M, 344.1260.

References

1) a) R. J. Spangler, B. G. Beckmann, and J. H. Kim, *J. Org. Chem.*, **42**, 2989 (1977); b) K. G. Das, J. Afzal, B. G. Hazra, and B. M. Bhawal, *Synth. Commun.*, **13**, 787 (1983); c) G. B. Black, M. Sainsbury, and A. J. Majeed, *Tetrahedron*, **40**, 4383 (1984).

2) N. S. Narasimhan, R. S. Mali, and B. K. Kulkarni, *Tetrahedron*, **39**, 1975 (1983); J. C. Estevez, M. C. Villaverde, R. J. Estevez, J. A. Seijas, and L. Casted, *Can. J. Chem.*, **68**, 964 (1990); C. A. Busacca and R. E. Johnson, *Tetrahedron Lett.*, **33**, 165 (1992); Refs. 1b and 1c and references cited therein.

3) G. A. Swan, *J. Chem. Soc.*, **1949**, 1720.

4) R. S. Mali, S. D. Patil, and S. L. Patil, *Tetrahedron*, **42**, 2075 (1986).

5) K. Shishido, E. Shitara, K. Fukumoto, and T. Kametani, *J. Am. Chem. Soc.*, **107**, 5813 (1985).

6) S. P. Khanapure and E. R. Biehl, *J. Org. Chem.*, **55**, 1471 (1990).

7) K. Kobayashi, A. Konishi, Y. Kanno, and H. Suginome, *J. Chem. Soc., Perkin Trans. 1*, **1993**, 111.

8) T. Garofano and A. Oliverio, *Ann. Chim. (Rome)*, **47**, 260 (1957); *Chem. Abstr.*, **60**, 12869i (1957).

9) R. K. Hill and W. R. Schearer, *J. Org. Chem.*, **27**, 921 (1962).

10) A. T. Blomquist and C. G. Bottomley, *Justus Liebigs Ann. Chem.*, **653**, 67 (1962).