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Regiospecific Ring Opening of Some Methyl- and Phenyl-Substituted 1,1,2-Trihalocyclopropanes to Acetylenic Acetals or Ketals by Variation of Reaction Conditions

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Regiospecific ring opening of six title compounds has been achieved under various reaction conditions. With dry sodium ethoxide in dry THF as reagent acetylenic diethyl ketals are obtained in 66–81% isolated yield. With DBU in dry ethanol, the corresponding acetylenic diethyl acetals are formed and isolated in 50–67% yield. Finally, treatment of the substrates with 50% aqueous sodium hydroxide in the presence of 2-propanol, triethylbenzylammonium chloride and dichloromethane gave acetylenic diisopropyl acetals, in 82% yield at the best. By-product formation was negligible in most cases.

We have earlier reported that treatment of substituted 1,1,2-trihalocyclopropanes (**1**) with 50% aqueous sodium hydroxide in the presence of triethylbenzylammonium chloride (TEBA), dichloromethane and ethanol gave mixtures of acetylenic diethyl ketals (**2**, $R \neq H$)[1–3] and diethyl acetals (**3**).[1,3] Interestingly, under the standard reaction conditions, viz., 8 equivalents of base and 4 equivalents of ethanol,[1] the selectivity of the reaction (the **2**/**3** ratio) and the total product yield (**2**+**3**) were sensitive to the halogen substituents in such a way that high selectivity (**2**/**3** >9) was only accomplished in reactions giving moderate yields (**2**+**3** $\leqslant 50\%$). These findings raised a challenging question: would it be possible to achieve high selectivity *and* high yields by variation of the reaction conditions? Subsequent investigations showed that this could be realized; the results of our studies are reported here.

Results and discussion

The first indication that the selectivity could be improved was noticed when 1,1,2-tribromo-2-methylcyclopropane (**1a**) was allowed to react under phase-transfer conditions in the presence of 35 equivalents of ethanol and with an ethanol/base ratio of 35. The products, viz., 3,3-diethoxy-1-butyne (**2a**) and 1,1-diethoxy-2-butyne (**3a**), were identical with those obtained under our standard conditions, but the larger amount of ethanol caused the acetal/ketal ratio (**3**/**2**) to increase from 1.0 to 4.0. This clearly suggested that *acetal* formation is favoured by *ethanol addition* to the intermediate cyclopropene[1] involved in the transformation, whereas the *ketal* is predominantly formed by *ethoxide attack* on the same intermediate. Consequently, two measures would be expected to improve the regioselectivity of the reaction: if ring opening is carried out using a mixture of ethoxide and an appropriate, non-reactive solvent as reagent, formation of ketal should be favoured, and if the same reaction is performed under phase-transfer conditions in the presence of an alcohol, which is *less acidic* than ethanol and whose alkoxide is *less nucleophilic* than ethoxide, formation of acetylenic acetal should become more favourable. Reactions encompassing both these measures were investigated and, indeed, our aspirations were fulfilled.

The study was performed with six 1,1,2-trihalocyclopropanes (**1a–f**, Scheme 1), prepared by addition of dihalocarbene to the corresponding haloalkenes according to the literature.[1] In order to facilitate *alkoxide* attack the substrates were reacted with a suspension of sodium ethoxide, prepared from sodium and *extremely dry* ethanol, in dry tetrahydrofuran (THF). Under these conditions **1a–f** reacted cleanly and gave acetylenic diethyl ketals **2** in good to excellent isolated yields on a multigram scale (see Table 1). Neither of the trihalocyclopropanes gave even traces of the corresponding acetals, and

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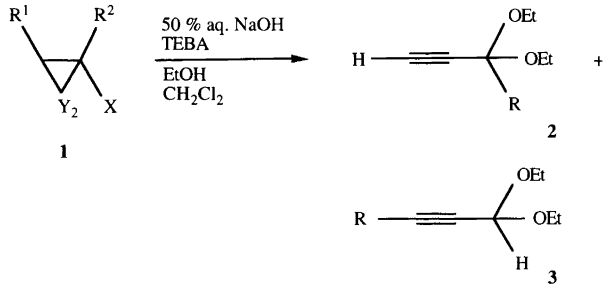


1

1	R^1	R^2	X	Y
a	H	Me	Br	Br
b	Me	H	Br	Br
c	H	Me	Br	Cl
d	H	Me	Cl	Cl
e	H	Ph	Br	Br
f	H	Ph	Br	Cl

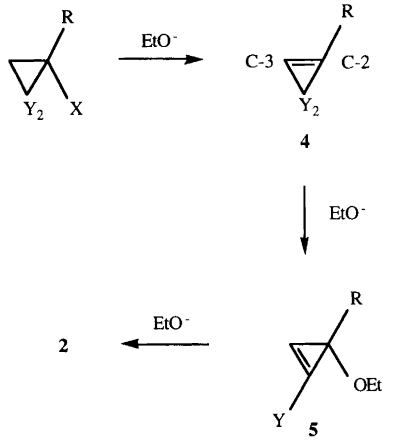
2a, **3a**; R = Me
2b, **3b**; R = Ph

Scheme 1.

Table 1. Acetylenic diethyl ketals (**2**) obtained by reacting trihalocyclopropanes (**1**) with sodium ethoxide, from sodium and absolutely dry ethanol, in dry THF at room temperature.

Starting material	Product	Yield (%)
1a	**2a**	79
1b	**2a**	76
1c	**2a**	72
1d	**2a**	66
1e	**2b**	81
1f	**2b**	76

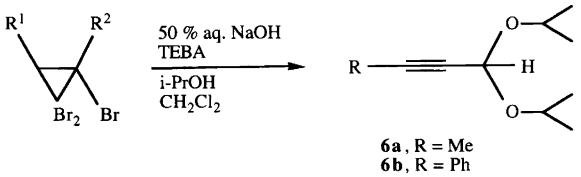
in no cases were functionalized alkenes[1] detected. Consequently, it is evident that the double bond in cyclopropene **4**, which is formed from **1** as an intermediate during the reaction,[1] is attacked *regiospecifically at the most substituted carbon atom* (C-2) by ethoxide and affords cyclopropene **5**, which suffers ring opening by attack of another ethoxide ion at the carbon atom attacked in the first step and affords **2** (Scheme 2). However, when sodium ethoxide, prepared from sodium and ethanol contaminated with a little water (1–2%),



Scheme 2.

was used, IR and NMR spectra of the crude product mixtures proved that traces (less than 3%) of α,β-unsaturated aldehydes were formed when **1c**, **1d**, **1e** and **1f** were reacted. The aldehydes were not isolated, but by comparison with authentic spectra[1] it was concluded that 3-ethoxy-2-methyl-2-propenal was formed from **1c** and **1d** and 3-ethoxy-2-phenyl-2-propenal was obtained from **1e** and **1f**. Both 2-propenal derivatives are most likely formed as discussed previously.[1] It is also noteworthy that application of several commercial samples of so-called dry sodium ethoxide in dry THF led to considerably less efficient and/or less regiospecific reactions than those reported in Table 1. These results indicate that the ethoxide reactivity is influenced by the presence of sodium hydroxide.

In order to favour *alcohol* attack on intermediate **4** trihalides **1** were first reacted under our standard phase-transfer conditions in the presence of 2-propanol. Replacement of ethanol with 2-propanol, which is less acidic than the former, lowers the alkoxide concentration during the reaction and makes alkoxide attack relative to alcohol attack on **4** less likely. This should favour formation of acetal **3** at the expense of the corresponding ketal **2**. Such a change did indeed take place, but it was surprising to find that the ketal formation was completely suppressed by what was seemingly a minor change. The results of this change turned out to be substrate dependent. Thus, all the 1,1,2-tribromocyclopropanes (**1a**, **1b** and **1e**) afforded the corresponding 1,1-di-isopropoxy-2-alkyne (**6**) in good yield (Scheme 3 and Table 2). The 1,1-dichloro-2-halocyclopropanes (**1c**, **1d** and **1f**), on the other hand, gave considerable amounts of several by-products. This was particularly the case with **1c** and



6a, R = Me
6b, R = Ph

Scheme 3.

Table 2. Acetylenic diisopropyl acetals (**6**) obtained by reacting 1,1,2-trihalocyclopropanes (**1**) with 50% aqueous sodium hydroxide in the presence of 2-propanol, dichloromethane, and triethylbenzylammonium chloride.

Starting material	Product	Yield (%)
1a	**6a**	82
1b	**6a**	70
1c	[a]	[a]
1d	[a]	[a]
1e	**6b**	79
1f	**6b**[b]	65[b]

[a]The reaction mixture was complex and contained a number of products in addition to **6a**. No pure products were isolated. [b]In addition 3-isopropoxy-2-phenyl-2-propenal (**7**) was formed and isolated in 11% yield.

1d; these compounds afforded an intractable reaction mixture that contained only minor amounts of **6a** and from which no pure compound could easily be isolated in significant quantities. The reaction mixture from **1f** was less complex and allowed the isolation of two products, 3,3-diisopropoxy-1-phenyl-1-propyne (**6b**) in 65% yield and 3-isopropoxy-2-phenyl-2-propenal (**7**) in 11% yield. Both **6** and **7** are supposed to be formed as discussed previously for the ethoxy analogues.[1]

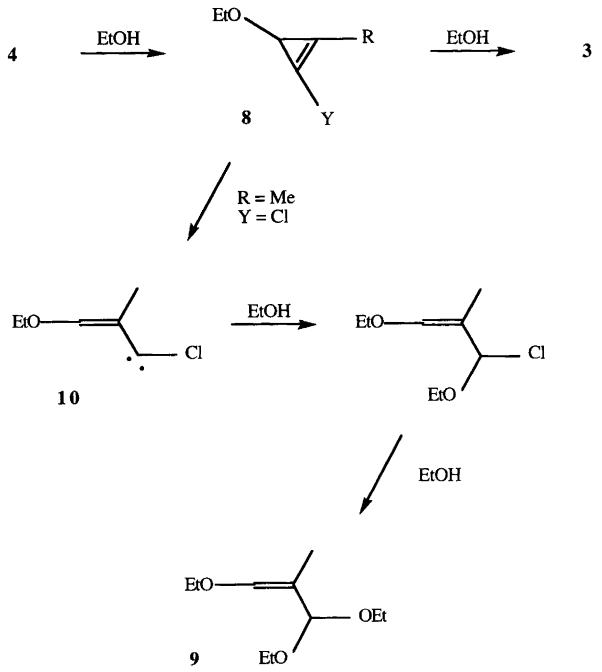
From the results presented above it was deemed impossible to achieve regiospecific ethanol addition to **4** and regiospecific formation of acetal **3** by modifying the phase-transfer reaction conditions. In order to favour *ethanol* addition to intermediate **4** the trihalides were, therefore, reacted with ethanol mixed with 1,5-diazabicyclo[4.3.0]non-5-ene (DBN) and 1,8-diazabicyclo-[5.4.0]undec-7-ene (DBU). The best results were obtained with the latter mixture. When **1** was refluxed in this mixture with 2 equivalents of DBU (relative to **1**) regiospecific ring opening and formation of acetal **3** were observed. Consequently, it is evident that *ethanol* attacks the double bond in **4** *regiospecifically at the least substituted carbon atom* (C-3) (see Scheme 2) and gives cyclopropene **8**, which is attacked by another ethanol molecule, at the carbon atom attacked in the first step, and suffers concomitant ring opening to **3**.

Acetals **3** were isolated in rather moderate yields (50–67%, Table 3) due to incomplete consumption of the starting material; this was the case even when a large excess of DBU was used. In neither case was any of corresponding ketals formed, but two compounds, viz., **1c** and **1d**, gave small amounts of 3-ethoxy-2-methyl-2-propenal diethyl acetal (**9**) as a by-product. Compounds similar to **9** have been obtained from other halogenated cyclopropanes under comparable conditions,[1] by a reaction sequence starting with a cyclopropene–vinylcarbene rearrangement.[4–11] Such a rearrangement may take place in our cases as well and convert cyclopropene **8** into vinylcarbene **10**, which affords **9** by a two-step transformation involving ethanol (Scheme 4). The alternative cyclopropene–vinylcarbene rearrangement of **8**, which has literature precedence,[12,13] would require an unprecedented and complex rearrangement to give **3** and is therefore regarded as unlikely.

Table 3. Acetylenic diethyl acetals (**3**) obtained by reacting trihalocyclopropanes (**1**) with DBU in refluxing absolute ethanol.

Starting material	Product	Yield (%)
1a	**3a**	67
1b	**3a**	65
1c	**3a**[a]	58
1d	**3a**[a]	52
1e	**3b**	59
1f	**3b**	50

[a] In addition 3-ethoxy-2-methyl-2-propenal diethyl acetal (**9**) was formed and isolated in 11% yield.



Scheme 4.

As reported above several functionalized alkenes were obtained as by-products in some of our reactions. Their formation is supposed to involve the cyclopropene–vinylcarbene rearrangement. Since all the alkenes are 2-substituted 3-alkoxy-2-propenal derivatives their generation involves cyclopropene **8** (Scheme 4) and not **5** (Scheme 2). It therefore seems as if the latter intermediate does not undergo the cyclopropene–vinylcarbene rearrangement before other reactions take place. The reluctance of **5** to rearrange is in accordance with the results of similar reactions studied by Müller and Pautex,[4] but the reason for this unwillingness to react is not evident. Further studies will hopefully clear this problem.

Another interesting point is the observation that the products formed from cyclopropene **8** depend on the nature of the halogen Y. When Y = Br, acetal **3** is the only product formed, but when Y = Cl, a 3-alkoxy-2-propenal derivative is generally obtained in addition to **3**. One explanation for this difference might be that the former cyclopropene is attacked more readily by nucleophiles than is the latter. It is also conceivable that **8** rearranges more easily to the corresponding vinylcarbene when Y = Cl than when Y = Br, although Baird[14] observed that 1-bromo-2-chloro-3,3-dimethylcyclopropene gave bromo(1-chloro-2-methyl-1-propenyl)carbene and chloro(1-bromo-2-methyl-1-propenyl)carbene in a 1:1 ratio at room temperature. Further studies in this area are under way.

Experimental

General. IR spectra were recorded on a Perkin–Elmer 1310 infrared spectrophotometer with the compounds as

a liquid film between NaCl plates. The intensity of the absorptions is reported as weak (w), medium (m) and strong (s). NMR spectra were run on a Bruker Spectrospin AC 200 F spectrometer at 200 MHz for ^1H and 50 MHz for ^{13}C with $CDCl_3$ as the solvent and tetramethylsilane (TMS) as an internal reference. Chemical shifts are given in ppm downfield from TMS. GC analyses were performed on a Hewlett Packard 5720 A gas chromatograph equipped with FID and a 15% SP 2100 fused silica column. TLC analyses were carried out with silica gel on aluminium (Merck 5554) with hexane–ethyl acetate (EtOAc) 9:1 as eluent. Flash chromatography was performed on a column (2 cm × 70 cm) of silica gel (Merck 9385) using hexane, hexane–EtOAc, pentane and pentane–dichloromethane (DCM) as solvents.

Chemicals. Extremely dry ethanol was obtained by refluxing commercially available absolute ethanol in the presence of sodium metal and diethyl succinate as described in the literature.[15] Tetrahydrofuran (THF) was freshly distilled from sodium–benzophenone ketyl prior to use. All the 1,1,2-trihalocyclopropanes, used as starting materials, were synthesized as described in the literature.[1]

Reaction of **1** *with sodium ethoxide in dry THF: preparation of acetylenic diethyl ketals* (**2**). *General procedure.* To a mixture of sodium ethoxide (2.72 g, 40 mmol, prepared from an excess of extremely dry ethanol and 0.92 g (40 mmol) of sodium in dry THF (50 ml), kept under nitrogen, was added 1,1,2-trihalocyclopropane (10 mmol) in dry THF (25 ml). The resulting mixture was stirred vigorously at room temperature and monitored by GC and TLC. Water was added, the products were extracted with diethyl ether and worked up in the usual way. The products were isolated by flash chromatography using eluents as indicated. By applying this procedure the following ketals were synthesized.

3,3-Diethoxy-1-butyne (**2a**), from 1,1,2-tribromo-2-methylcyclopropane (**1a**, 2.93 g), a *cis/trans* mixture of 1,1,2-tribromo-3-methylcyclopropane (**1b**, 2.93 g), 2-bromo-1,1-dichloro-2-methylcyclopropane (**1c**, 2.03 g) and 1,1,2-trichloro-2-methylcyclopropane (**1d**, 1.59 g). The product was isolated by flash chromatography (pentane–DCM = 95:5); yield 1.12 g (79%) from **1a**, 1.08 g (76%) from **1b**, 1.03 g (72%) from **1c** and 0.94 g (66%) from **1d**. The spectroscopic data were in agreement with those reported in the literature.[1]

3,3-Diethoxy-3-phenyl-1-propyne (**2b**), from 1,1,2-tribromo-2-phenylcyclopropane (**1e**, 3.55 g) and 2-bromo-1,1-dichloro-2-phenylcyclopropane (**1f**, 2.66 g). The product was isolated by flash chromatography (hexane–EtOAc = 9:1), yield 1.65 g (81%) from **1e** and 1.55 g (76%) from **1f**. The spectroscopic data were in agreement with those reported in the literature.[1]

Reaction of **1** *with 2-propanol under phase-transfer conditions: preparation of acetylenic diisopropyl acetals* (**6**). *General procedure.* To a solution of 1,1,2-trihalocyclopropane (20 mmol), triethylbenzylammonium chloride (TEBA) (0.2 g) and 2-propanol (4.8 g, 80 mmol) in dichloromethane (50 ml), kept at room temperature under nitrogen, was added 50% aqueous sodium hydroxide (12.8 g of the solution, 6.4 g of NaOH, 160 mmol). The resulting mixture was stirred vigorously at room temperature and monitored by GC and TLC. Water was added, and the products were extracted with diethyl ether and worked up in the usual way. The products were isolated by distillation or flash chromatography.

1,1-Diisopropoxy-2-butyne (**6a**), from 1,1,2-tribromo-2-methylcyclopropane (**1a**, 5.86 g) and a *cis/trans* mixture of 1,1,2-tribromo-3-methylcyclopropane (**1b**, 5.86 g). The product was isolated by distillation, b.p. 80–82 °C/20 mmHg, yield 2.80 g (82%) from **1a** and 2.40 g (70%) from **1b**. IR: 2960 (s), 2910 (s), 2290 (w), 2230 (m), 1945 (w), 1630 (w), 1455 (m), 1375 (s), 1315 (s), 1175 (s), 1145 (s), 1050 (s), 1015 (s) cm^{-1}. ^1H NMR: δ 1.17–1.22 (12 H, m), 1.88 (3 H, d, *J* 2.0 Hz), 4.09 (2 H, septet, *J* 6.2 Hz), 5.30 (1 H, q, *J* 2.0 Hz). ^{13}C NMR: δ 3.3 (CH_3), 22.2 (2 × CH_3), 22.9 (2 × CH_3), 67.2 (CH), 75.7 (C), 81.1 (C), 88.5 (CH).

The reaction mixtures from **1c** and **1d** were very complex (NMR) and contained a number of products according to GC analysis. Efforts to isolate pure samples of any of the products were in vain.

3,3-Diisopropoxy-1-phenyl-1-propyne (**6b**), from 1,1,2-tribromo-2-phenylcyclopropane (**1e**, 7.10 g) and 2-bromo-1,1-dichloro-2-phenylcyclopropane (**1f**, 5.32 g). The product was isolated by flash chromatography (hexane–EtOAc = 95:5), yield 3.67 g (79%) and 3.01 g (65%) from **1e** and **1f**, respectively. IR: 3040 (m), 3015 (m), 2960 (s), 2920 (s), 2180 (m), 1480 (m), 1440 (m), 1370 (s), 1310 (s), 1270 (s), 1170 (s), 1100 (s), 1070 (s), 1020 (s), 920 (m), 770 (m), 755 (s), 690 (s) cm^{-1}. ^1H NMR: δ 1.24 (12 H, t, *J* 6.2 Hz), 4.19 (2 H, septet, *J* 6.2 Hz), 5.56 (1 H, s), 7.27–7.49 (5 H, m). ^{13}C NMR: δ 22.3 (2 × CH_3), 23.1 (2 × CH_3), 67.7 (CH), 84.4 (C), 85.4 (C), 89.1 (CH), 121.9 (C), 128.0 (2 × CH), 128.5 (CH), 131.6 (2 × CH).

From the reaction mixture from **1f** a second product, isolated by flash chromatography with EtOAc as the eluent, was obtained as well, viz., 3-isopropoxy-2-phenyl-2-propenal (**7**), yield 0.40 g (11%). IR: 3030 (w), 2995 (w), 2955 (m), 2900 (w), 2705 (w), 2225 (m), 1652 (s), 1607 (s), 1585 (s), 1475 (m), 1430 (m), 1325 (m), 1245 (s), 1205 (s), 1165 (m), 1125 (m), 1075 (s), 1012 (m), 835 (m), 678 (m) cm^{-1}. ^1H NMR: δ 1.29 (6 H, d, *J* 6.2 Hz), 4.28 (1 H, septet, *J* 6.2 Hz), 7.06 (1 H, s), 7.15–7.45 (5 H, m), 9.27 (1 H, s). ^{13}C NMR: δ 22.2 (2 × CH_3), 79.7 (CH), 122.0 (C), 127.1 (CH), 127.6 (2 × CH), 129.0 (2 × CH), 130.7 (C), 167.5 (CH), 190.4 (C=O).

Reaction of **1** *with DBU in extremely dry ethanol: preparation of acetylenic diethyl acetals* (**3**). *General procedure.* To a solution of 1,1,2-trihalocyclopropane (10 mmol) in extremely dry ethanol (50 ml) kept under nitrogen was added 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU) (3.04 g, 20 mmol). The resulting mixture was refluxed with vigorous stirring and monitored by GC and TLC. The reaction was terminated after 24 h, although some unreacted starting material was still present. Water was added, the products were extracted with diethyl ether and worked up in the usual way. The products were isolated by either distillation or flash chromatography. By applying this procedure the following acetals were synthesized.

1,1-Diethoxy-2-butyne (**3a**), from 1,1,2-tribromo-2-methylcyclopropane (**1a**, 2.93 g), a *cis/trans* mixture of 1,1,2-tribromo-3-methylcyclopropane (**1b**, 2.93 g), 1-bromo-2,2-dichloro-1-methylcyclopropane (**1c**, 2.03 g) and 1,1,2-trichloro-2-methylcyclopropane (**1d**, 1.59 g).

The product was isolated by distillation when **1a** and **1b** were used, b.p. 62–64 °C/12 mmHg. The yield was 0.96 g (67%) from **1a** and 0.92 g (65%) from **1b**; recovered starting material amounted to 0.51 g (17%) and 0.45 g (15%) from **1a** and **1b**, respectively.

The product was isolated by flash chromatography when **1c** and **1d** were employed as starting materials, using pentane–DCM = 95 : 5 as the eluent. The yield was 0.82 g (58%) from **1c** and 0.74 g (52%) from **1d** whereas the amount of recovered starting material was 0.20 g in both cases, corresponding to 10% and 13% recovery, respectively. The spectroscopic data were in agreement with those reported in the literature.[1]

From the reaction mixtures from **1c** and **1d** a second product was obtained, viz., 3-ethoxy-2-methyl-2-propenal diethyl acetal (**9**), yield 0.19 g (10%) from **1c** and 0.29 g (15%) from **1d**. IR: 3100 (w), 3060 (w), 2980 (s), 2920 (s), 2870 (s), 1635 (m), 1435 (m), 1375 (s), 1345 (s), 1310 (s), 1255 (m), 1175 (s), 1115 (s), 1060 (s), 1005 (s), 935 (w), 875 (m), 840 (m), 810 (s), 780 (s), 710 (w) cm^{-1}. ^1H NMR: δ 1.13 (9 H, t, *J* 7.1 Hz), 1.70 (3 H, d, *J* 1.4 Hz), 3.44 (6 H, m), 4.68 (1 H, s), 6.21 (1 H, q, *J* 1.4 Hz). ^{13}C NMR: δ 12.3 (CH_3), 14.9 ($3 \times CH_3$), 61.2 ($3 \times CH_2$), 102.2 (CH), 117.6 (CH), 136.0 (C).

3,3-Diethoxy-1-phenyl-1-propyne (**3b**), from 1,1,2-tribromo-2-phenylcyclopropane (**1e**, 3.55 g) and 2-bromo-1,1-dichloro-2-phenylcyclopropane (**1f**, 2.66 g). The product was separated and isolated by flash chromatography. Unreacted starting material was recovered using hexane as the eluent, 1.0 g (28%) from **1e** and 0.46 g (17%) from **1f**, whereas **3b** was eluted with hexane–EtOAc = 90 : 10, yield 1.2 g (59%) and 1.02 g (50%) from **1e** and **1f**, respectively. The spectroscopic data were in agreement with those reported in the literature.[1]

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