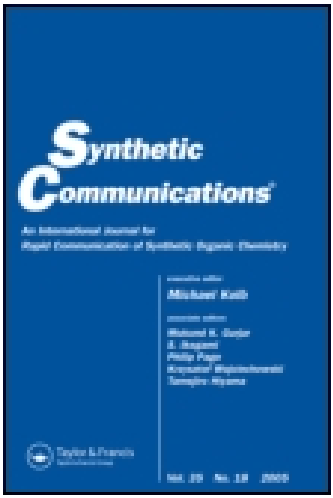

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An Improved Synthesis of Dialkylcyclopropenones

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AN IMPROVED SYNTHESIS OF DIALKYLCYCLOPROPENONES

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Abstract: Dialkylcyclopropenones are formed in high yields when alkynes are treated with chloroform-butyllithium in THF at - 78 °C followed by acidic hydrolysis of the dichlorocyclopropene intermediates at low temperatures.

Cyclopropenones undergo several interesting reactions and they may be useful starting materials for a variety of compounds.[1-4] The cyclopropenone moiety is also found in natural products,[5,6] for instance the antibiotic Penitricin,[5] and it has been incorporated in synthetically prepared bioactive compounds.[7,8] In our study of indolizinol derivatives with antioxidant properties, we prepared 2,3-diphenylindolizines by cyclization of commercially available diphenylcyclopropenone with appropriate (di)azines.[9] When we wanted to extend this study to include indolizines formed from other cyclopropenones, we found that few efficient syntheses of dialkylcyclopropenones have been published.

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Modified Favorskii reactions on α,α'-dibromoketones gives dialkylcyclopropenones in moderate yields.[10-12] Dialkylcyclopropenones have been prepared via metallation of a cyclopropenone acetal,[13] or from alkenes via cyclopropenium cations.[12] However, both strategies involves quite a few steps. Another route to cyclopropenones is addition of dichlorocarbeniod reagents to alkynes followed by hydrolysis. When suitable alkynes are readily available, the latter method may seem very attractive, but the use of dichlorocarbenoids generated from sodium trichloroacetate,[15-17] chloroform and sodium hydroxide under PTC-conditions,[18] chloroform and potassium *tert*-butoxide,[19-21] or bromotrichloromethane and methyllithium[22,23] generally results in low yields of the desired cyclopropenones and the formation of several by-products, especially when one or two methylene groups were situated next to the triple bond. These problems were largely overcome when dichlorocyclopropenes were formed by reaction of an alkyne with $LiCCl_3$ in THF (generated *in situ* from *n*-BuLi and $CHCl_3$),[24] but in this study ynones were the major product after hydrolysis of the dichlorocyclopropene intermediates and the isolated yields of cyclopropenones were rather modest. It appears from the literature that the cyclopropenone : ynone ratio may be dependent on the conditions used for hydrolysis of dichlorocyclopropenes, when the reaction is quenched with water or diluted aqueous base, substantial amounts of ynones may be formed,[18,24,25] but acidic hydrolysis seems to favour cyclopropenone formation.[24-26]

We have prepared dialkylcyclopropenones **3** in one step from the alkynes **1** by combining the use of the mild carbenoid reagent $LiCCl_3 \cdot 3THF$ with acidic hydrolysis at low temperatures of the dichlorocyclopropene intermediates **2** (Method A, Scheme 1, Table 1).

Scheme 1

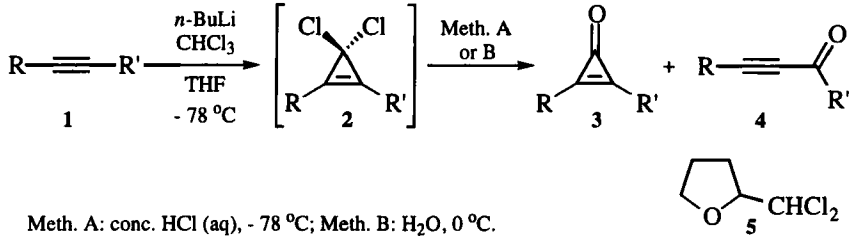

Table 1. Synthesis of dialkylcyclopropenones **3**.

Entry	R-	R'-	Meth.	**3** : **4**[a]	Yield (%) **3**[b]	Yield (%) **4**[b]
1	CH_3CH_2-	CH_3CH_2-	A	93 : 7	90, **3a**	—[c]
2	CH_3CH_2-	CH_3CH_2-	B	48 : 52	29, **3a**	33, **4a**
3	$CH_3(CH_2)_2$-	$CH_3(CH_2)_2$-	A	95 : 5	67, **3b**	—[c]
4	$CH_3(CH_2)_2$-	$CH_3(CH_2)_2$-	B	37 : 63	26, **3b**	41, **4b**
5	CH_3-	$CH_3(CH_2)_4$-	A	—[d]	55, **3c**	—[c]
6	CH_3-	$CH_3(CH_2)_4$-	B	33 : 67[e]	16, **3c**	34, **4c**[e]
7	-$(CH_2)_6$-		A	—[d]	63, **3d**	—[c]
8	-$(CH_2)_6$-		B	43 : 57	21, **3d**	28, **4d**
9	-$(CH_2)_{10}$-		A	73 : 27	60, **3e**	25, **4e**
10	-$(CH_2)_{10}$-		B	32 : 68	23, **3e**	54, **4e**
11	-$(CH_2)_{13}$-		A	80 : 20	50, **3f**	12, **4f**
12	-$(CH_2)_{13}$-		B	32 : 68	23, **3f**	48, **4f**

[a] From ^{1}H NMR of the crude product. [b] Yield of isolated product. [c] Not isolated. [d] **4** not detectable by ^{1}H NMR of the crude product. [e] A ca. 1:1 mixture of the isomeric ynones **4c** and **4c'**.

When the reaction was quenched according to Method A, the yields of the cyclopropenones **3** are generally high and only minor amounts of ynones **4** could be detected.

On the other hand, when the reaction mixture was allowed to reach 0 °C and was quenched by addition of water (Meth. B) essentially as reported by Gleiter,[24] more ynone **4** than cyclopropenone **3** was formed. Furthermore, in meth. B the reaction temperature was allowed to reach ca 0 °C before quenching, and the THF-derivative **5** was formed by reaction of the carbenoid with THF.[27] Separation of the ynones **4** and compound **5** was in some instances rather tedious. Compound **5** could hardly be detected when the hydrolysis was performed according to Meth. A.

Experimental

The ^{1}H NMR spectra were recorded at 300 MHz with a Bruker Avance DPX 300 instrument, or at 200 MHz with a Bruker Avance DPX 200 instrument. The ^{13}C NMR spectra were recorded at 75 or 50 MHz using spectrometers mentioned above. Chemical shifts (δ) are given in ppm downfield from tetramethylsilane. Mass spectra were recorded with a VG Prospec instrument at 70 eV ionising voltage and are presented as *m/z* (% rel. int.). Elemental analyses were performed by Ilse Beetz Mikroanalytisches Laboratorium, Kronach, Germany. Melting points are uncorrected. Flash chromatography was performed on neutral alumina, activity III. THF was distilled from Na-benzophenone and chloroform was distilled from CaH_2. Commercially available alkynes were redistilled prior to use. *n*-Butyllithium (ca. 1.6 M in hexane) was titratet with diphenylacetic acid.[28] Cyclooctyne,[29] cyclododecyne[30] and cyclopentadecyne[30] were prepared according to literature procedures. All other reagents were commercially available and were used as received.

General Procedure - Method A. *n*-Butyllithium (2.2 mmol) was added dropwise over a period of 60 min. to a stirring solution of chloroform (0.20 ml, 2.5 mmol) and alkyne **1** (1.0 mmol) in THF (20 ml) under N_2-atm. at -78 °C. The resulting mixture was stirred for an additional 4 h at -78 °C before conc. hydrochloric acid (1 ml) was added dropwise over 10 min. The cooling bath was removed and the mixture was stirred for 10 min. without external cooling, before water (20 ml) was added. The mixture was extracted with dichloromethane (5 × 20 ml) and the combined organic extracts were dried ($MgSO_4$) and evaporated *in vacuo*. The products were isolated by flash chromatography.

Method B. *n*-Butyllithium (2.2 mmol) was added dropwise over a period of 60 min. to a stirring solution of chloroform (0.20 ml, 2.5 mmol) and alkyne **1** (1.0 mmol) in THF (20 ml) under N_2-atm. at -78 °C. The resulting mixture was stirred for an additional 4 h at -78 °C before the cooling bath was removed and the mixture was stirred for 10 min. without external cooling. Water (20 ml) was added and the mixture was extracted and the products were purified as described under Method A.

2,3-Diethyl-2-cyclopropen-1-one (3a). The product was isolated by flash chromatography on Al_2O_3 eluting with CH_2Cl_2. Yield 99 mg (90 %) oil from method A and 33 mg (29 %) from method B. ^1H NMR ($CDCl_3$, 300 MHz): δ 1.22 (t, *J* 7.5 Hz, 6 H, 2 × CH_3), 2.52 (q, *J* 7.5 Hz, 4 H, 2 × CH_2). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 11.4 (CH_3), 20.1 (CH_2), 159.9 (C=O), 162.7 (C=C). MS (EI): 110 (12, M^+), 82 (35), 67 (100), 53 (32), 41 (65). Hrms: Found 110.0728, $C_7H_{10}O$ requires 110.0732.

2,3-Dipropyl-2-cyclopropen-1-one (3b). The product was isolated by flash chromatography on Al_2O_3 eluting with CH_2Cl_2. Yield 92 mg (67 %) oil from method A and 36 mg (26 %) from method B. ^1H NMR ($CDCl_3$, 300 MHz): δ 0.97 (t, *J* 7.2 Hz, 6 H, 2 × CH_3), 1.6-1.7 (m, 4 H, 2 × CH_2), 2.52 (t, *J* 7.2 Hz, 4 H, 2

× CH_2). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 13.7 (CH_3), 19.7 and 28.2 (CH_2), 160.0 (C=O), 160.6 (C=C). MS (EI): 138 (23, M^+), 81 (70), 67 (100), 53 (49), 41 (59). NMR data are in accordance with those reported in the literature.[14]

2-Methyl-3-pentyl-2-cyclopropen-1-one (3c). The product was isolated by flash chromatography on Al_2O_3 eluting with CH_2Cl_2. Yield 77 mg (55 %) oil from method A and 22 mg (16 %) from method B. ^1H NMR ($CDCl_3$, 300 MHz): δ 0.87 (t, *J* 7.0 Hz, 3 H, CH_3), 1.3-1.4 (m, 4 H, 2 × CH_2), 1.66 (m, 2 H, CH_2), 2.21 (s, 3 H, CH_3), 2.55 (t, *J* 7.4 Hz, 2 H, CH_2). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 11.0 and 13.6 (CH_3), 22.0, 25.5, 26.0 and 31.1 (CH_2), 156.7 and 159.5 (C=/C=O), 161.3 (C=). MS (EI): 138 (6, M^+), 123 (5), 110 (25), 109 (22), 95 (78), 81 (83), 67 (71), 54 (79), 41 (100). Hrms: Found 138.1050, $C_9H_{14}O$ requires 138.1045.

Bicyclo[6.1.0]non-1(8)-en-9-one (3d). The product was isolated by flash chromatography on Al_2O_3 eluting with CH_2Cl_2. Yield 79 mg (63 %) oil from method A and 26 mg (21 %) from method B. In both reactions 0.92 mmol alkyne was used. ^1H NMR ($CDCl_3$, 300 MHz): δ 1.5-1.6 (m, 4 H, 2 × CH_2), 1.7 (m, 4 H, 2 × CH_2), 2.5 (m, 4 H, 2 × CH_2). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 21.8, 25.6 and 25.9 (CH_2), 157.6 (C=O) 161.9 (C=C). MS (EI): 136 (14, M^+), 93 (55), 79 (100), 77 (53). NMR data are in accordance with those reported in the literature.[24]

Bicyclo[10.1.0]tridec-1(12)-en-13-one (3e). The product was isolated by flash chromatography on Al_2O_3 eluting with CH_2Cl_2. Yield 114 mg (60 %) oil from method A and 42 mg (23 %) from method B. 0.94 mmol alkyne was used in Meth. B. ^1H NMR ($CDCl_3$, 200 MHz): δ 1.1-1.3 (m, 12 H, 6 × CH_2), 1.61 (m, 4 H, 2 × CH_2), 2.55 (t, *J* 6.3 Hz, 4 H, 2 × CH_2). ^{13}C NMR ($CDCl_3$, 50 MHz): δ 23.7, 24.0, 24.2, 24.5 and 26.1 (CH_2), 160.4 (C=O), 161.1 (C=C). MS (EI): 192 (4, M^+), 121 (39), 95 (37), 93 (50), 81 (70), 79 (79), 67 (100), 55 (55). Hrms: Found 193.1601, $C_{13}H_{20}O$ requires 193.1592.

Bicyclo[13.1.0]hexadec-1(15)-en-16-one (3f). The product was isolated by flash chromatography on Al_2O_3 eluting with CH_2Cl_2. Yield 113 mg (50 %) from method A and 53 mg (23 %) from method B. M.p. 40-43 °C. (Found: C, 81.88; H, 11.31. Calc. for $C_{16}H_{26}O$: C, 81.99; H, 11.18 %). ^1H NMR ($CDCl_3$, 300 MHz): δ 1.1-1.3 (m, 18 H, 9 × CH_2), 1.63 (m, 2 × CH_2), 2.51 (t, *J* 6.8 Hz, 4 H, 2 × CH_2). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 24.2, 24.5, 24.77, 24.80, 25.0, 25.3 and 26.5 (CH_2), 159.2 (C=O), 160.3 (C=C). MS (EI): 234 (10, M^+), 135 (27), 121 (30), 109 (32), 95 (52), 93 (43), 81 (73), 79 (59), 67 (88), 41 (100).

4-Heptyn-3-one (4a). The product was isolated by flash chromatography on Al_2O_3 eluting with hexane-CH_2Cl_2 (10:1). Yield 38 mg (33 %) oil from method B using 1.1 mmol alkyne. ^1H NMR ($CDCl_3$, 300 MHz): δ 1.08 (t, *J* 7.4 Hz, 3 H, CH_3), 1.16 (t, *J* 7.5 Hz, 3 H, CH_3), 2.32 (q, *J* 7.5 Hz, 2 H, CH_2), 2.50 (q, *J* 7.4 Hz, 2 H, CH_2). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 7.9 (C-1), 12.5 (C-6), 12.7 (C-7), 38.6 (C-2), 79.9 (C-5), 95.0 (C-4), 188.7 (C=O). MS (EI): 110 (5, M^+), 82 (8), 81 (100), 53 (48). NMR data are in accordance with those reported in the literature.[31,32]

5-Nonyn-4-one (4b). The product was isolated by flash chromatography on Al_2O_3 eluting with hexane-CH_2Cl_2 (10:1). Yield 56 mg (41 %) oil from method B. ^1H NMR ($CDCl_3$, 300 MHz): δ 0.91 (t, *J* 7.4 Hz, 3 H, CH_3), 0.98 (t, *J* 7.4 Hz, 3 H, CH_3), 1.56 (m, 2 H, CH_2), 1.66 (m, 2 H, CH_2), 2.30 (t, *J* 7.0 Hz, 2 H, CH_2), 2.47 (t, *J* 7.3 Hz, 2 H, CH_2). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 13.4 and 13.5 (CH_3), 17.6, 20.8, 21.2 and 47.4 (CH_2), 81.0 and 94.0 (C≡C), 188.4 (C=O). MS (EI): 138 (1, M^+), 123 (10), 110 (27), 95 (100), 67 (14), 53 (20). ^1H NMR and MS data are in accordance with those reported in the literature.[33]

2-Nonyn-4-one (4c) and 3-nonyn-2-one (4c'). A ca. 1:1 mixture of the isomers **4c** and **4c'** was isolated by flash chromatography on Al_2O_3 eluting with

hexane-CH_2Cl_2 (10:1). Total yield 47 mg (34 %). ^{1}H NMR ($CDCl_3$, 300 MHz): δ 0.89-0.95 (m, 6 H, CH_3), 1.3-1.4 (m, 8 H, CH_2), 1.6-1.7 (m, 4 H, CH_2), 2.03 (s, 3 H, CH_3), 2.33 (s, 3 H, CH_3), 2.37 (t, *J* 7.1 Hz, 2 H, CH_2), 2.52 (t, *J* 7.5 Hz, 2 H, CH_2). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 4.4, 14.3 (2×C), 19.3, 22.5, 22.8, 24.1, 27.8, 31.4, 31.5, 33.1, 45.8, 80.2, 81.4, 89.7, 94.1, 184.9, 188.4. ^{1}H NMR resonances belonging to compound **4c'** are in accordance with those reported in the literature.[34]

2-Cyclononyn-1-one (4d). The product was isolated by flash chromatography on Al_2O_3 eluting with hexane-CH_2Cl_2 (10:1). Yield 35 mg (28 %) oil from method B using 0.92 mmol alkyne. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 1.7-1.9 (m, 8 H, 4 × CH_2), 2.3-2.5 (m, 4 H, 2 × CH_2). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 19.8, 22.7, 25.8, 27.2, 27.6 and 44.0 (CH_2), 85.0 and 114.0 (C≡C), 191.0 (C=O). MS (EI): 136 (3, M^+), 135 (6), 121 (19), 108 (31), 107 (27), 93 (33), 79 (100), 66 (44). NMR data are in accordance with those reported in the literature.[24]

2-Cyclotridecyn-1-one (4e). The product was isolated by flash chromatography on Al_2O_3 eluting with hexane-CH_2Cl_2 (10:1). Yield 47 mg (25 %) oil from method A and 98 mg (54 %) from method B. 0.94 mmol alkyne was used in Meth. B. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 1.3-1.4 (m, 10 H, 5 × CH_2), 1.5-1.6 (m, 4 H, 2 × CH_2), 1.76 (m, 2 H, CH_2), 2.37 (dd, *J* 1.0 Hz and 5.8 Hz, 2 H, CH_2), 2.43 (dd, *J* 1.3 Hz and 6.7 Hz, 2 H, CH_2). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 18.9, 24.2, 25.0, 25.5, 25.7, 25.7, 26.1, 26.3, 26.4 and 44.3 (CH_2), 81.1 and 96.0 (C≡C), 189.8 (C=O). MS (EI): 192 (19, M^+), 164 (8), 163 (16), 149 (38), 121 (48), 107 (73), 79 (100), 67 (60), 55 (80), 41 (99). ^{1}H NMR and MS data are in accordance with those reported in the literature.[35]

2-Cyclohexadecyn-1-one (4f). The product was isolated by flash chromatography on Al_2O_3 eluting with hexane-CH_2Cl_2 (10:1). Yield 28 mg (12 %)

oil from method A and 110 mg (48 %) from method B. (Found: C, 81.95; H, 11.17. Calc. for $C_{16}H_{26}O$: C, 81.99; H, 11.18 %). ^{1}H NMR ($CDCl_3$, 300 MHz): δ 1.3-1.4 (m, 16 H, 8 × CH_2), 1.4-1.6 (m, 4 H, 2 × CH_2), 1.7-1.8 (m, 2 H, 2 × CH_2), 2.42 (t, *J* 6.7 Hz, 2 H, CH_2), 2.44 (t, *J* 7.4 Hz, 2 H, CH_2). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 18.8, 24.7, 25.9, 25.9, 26.2, 26.6, 26.7, 26.9, 27.0, 27.1, 27.3, 27.5 and 45.1 (CH_2), 81.2 and 95.2 (C≡C), 189.3 (C=O). MS (EI): 234 (7, M^+), 205 (5), 191 (12), 177 (19), 163 (22), 110 (45), 95 (61), 79 (71), 41 (100).

(Dichloromethyl)tetrahydrofuran (5). This compound formed in various amounts whenever method B was employed. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 2.0-2.1 (m, 4 H, 2 × CH_2), 3.9-4.0 (m, 2 H, CH_2), 4.26 (m, 1 H, OCH), 5.69 (d, *J* 4.8 Hz, 1 H, $CHCl_2$). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 26.1 (CH_2), 27.8 (CH_2), 70.0 (CH_2O), 74.4 ($CHCl_2$), 82.9 (CHO). MS (EI): 155 (0.14, M^+), 71 (100), 53 (6), 43 (37), 42 (10), 41 (21). NMR and MS data are in accordance with those reported in the literature.[36]

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