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REACTION OF ENOL ETHERS WITH ZINC CARBENOID REAGENTS. CYCLOPROPANATION AND SUBSEQUENT ISOMERIZATION TO ALLYLIC ETHERS

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Summary

The reactions of enol ethers (ROC=C, R = alkyl, silyl) with zinc carbenoid reagents were found to give allylic ethers in several cases along with the expected cyclopropyl ethers. The ratio of these two products was highly dependent on the concentration of the reaction mixture. Thus, the selective formation of each product was conveniently attained by merely changing the amounts of the solvent used. Zinc iodide, a by-product of the reaction, plays a key role in the present cyclopropyl to allylic rearrangement. Ring-opened ionic intermediates are proposed.

Introduction

In the past decade, alkoxycyclopropanes [1] and siloxycyclopropanes [2] have found wide application in organic synthesis. A convenient method for the preparation of these compounds is cyclopropanation of enol ethers by carbenoid reagents. In the course of our study on the synthesis of cyclopropanols, we tried the Simmons–Smith reaction [3] of some 1-siloxycycloalkenes [4,5,6,7]. We found that the reaction often gave two products, the normally expected siloxycyclopropanes and isomerized 2-methylene-siloxyalkanes. This led us to a detailed investigation of the carbenoid reaction, both published and unpublished [8]. The primary objective of our study was to establish the reaction conditions for obtaining clean cyclopropanation products. The conditions for the selective preparation of the unusual products were also determined, providing a convenient route to 2-methylenecycloalkanols, after hydrolysis.

Results and discussion

1. The Simmons–Smith reaction of 1-ethoxycyclohexene (1e). A time course study

By the Simmons–Smith reaction of 1-ethoxycyclohexene (**1e**) (eq. 1), two prod-

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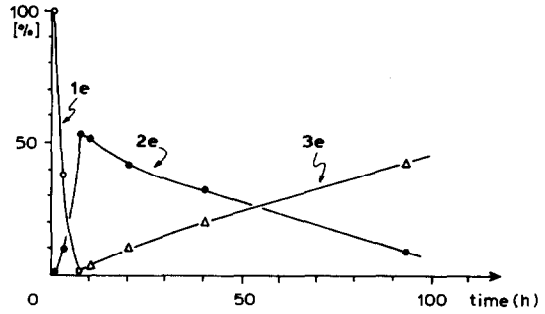
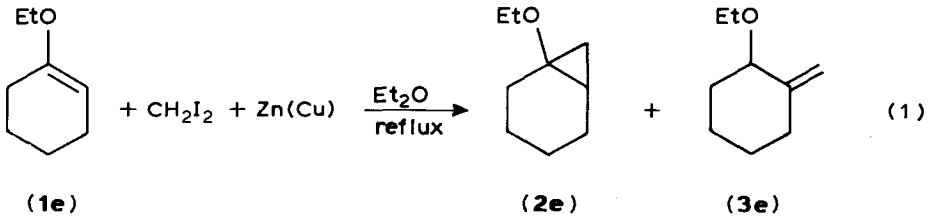
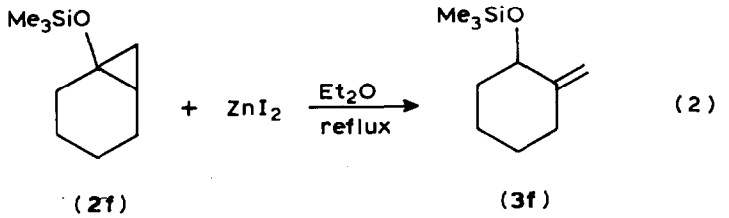


Fig. 1. Time course of the Simmons–Smith reaction of **1e**. (**1e**, 0.08 mol; zinc/copper couple, 0.16 mol; methylene iodide 0.12 mol); ether, 40 ml; reflux for x h).



ucts were obtained, 1-ethoxybicyclo[4.1.0]heptane (**2e**) and 2-methylene-ethoxycyclohexane (**3e**). In order to ascertain the process of the formation of the allylic isomer **3e**, a time course study was undertaken. The allylic isomer **3e** was produced via further isomerization of ethoxycyclopropane **2e** initially formed (Fig. 1). Zinc iodide, which was concomitantly produced with the consumption of zinc carbenoid, was suspected to be a key species.

This was ascertained from the results of the separate experiments: treatment of siloxycyclopropane **2f** with zinc iodide, prepared in situ from zinc/copper couple and iodine, afforded isomerized **3f** from similar concentrations of reactants (eq. 2).



*2. The Simmons–Smith reaction of (1-methyl-siloxymethylene)cyclopentane (**1m**). High dependencey of product distribution on the concentration of reactants in the reaction mixture*

The Simmons–Smith reaction of (1-methyl-siloxymethylene)cyclopentane (**1m**) (eq. 3) also afforded two products, 1-methyl-siloxyspiro[4.2]heptane (**2m**) and its

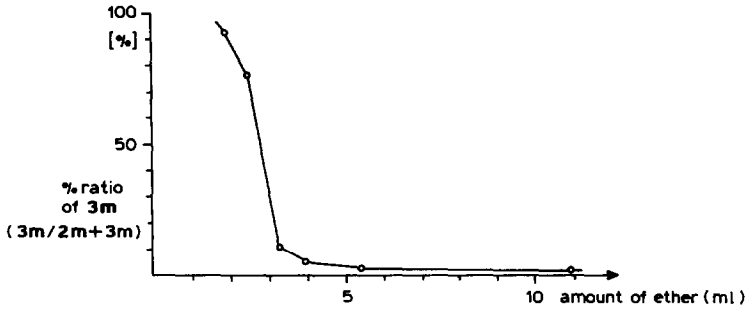
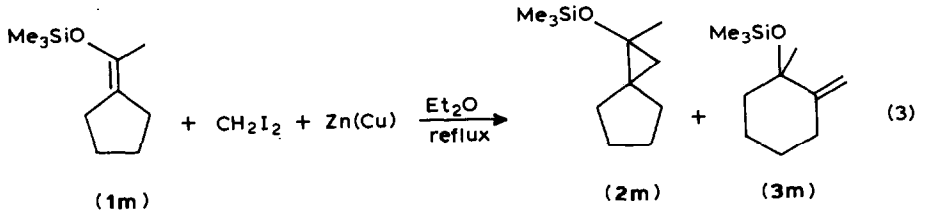


Fig. 2. The Simmons–Smith reaction of **1m** in several concentrations (**1m**, 5 mmol; zinc/copper couple, 16 mmol; methylene iodide, 8 mmol; ether, x ml; 40 h.



isomerized product **3m**. With this reaction system, we studied the effect of concentration of reactants in the reaction mixture on product distribution. The results are shown in Fig. 2 and in Table 1. Interestingly, a dramatic change in product distribution was observed when the amount of solvent was varied; more than 4 ml of ether for 5 mmol of **1m** gave predominantly **2m**, and 2 ml of ether afforded **3m** with high selectivity. The observed result that product ratio is markedly dependent on the amount of solvent present led us to attempt the zinc carbenoid reaction under both 'dilute' and 'concentrated' conditions.

3. Controlled synthesis of cyclopropyl ethers and allylic ethers

For preparing each product selectively, changing the amounts of the solvent ether gave three types of reaction conditions (A, B, and C): A corresponded to 'dilute' conditions in which 110 ml of ether was used for 50 mmol of substrate **1**, 80 mmol of methylene iodide, and 160 mmol of zinc/copper couple, while B and C corresponded to 'concentrated' and 'highly concentrated' conditions in which 40 ml and 25 ml of ether, respectively, were used. The results obtained from various enol ethers **1** are shown in Table 1. In most cases the expected cyclopropanated products **2** were obtained with good to exclusive selectivity under A. Thus, the reaction carried out under such reaction conditions ensures selective formation of cyclopropanated products.

On the other hand, reaction of enol silyl ethers **1c**, **1f**, and **1g**, under B resulted in the exclusive formation of the respective allylic ethers **3c**, **3f**, and **3g**. Although the 7-membered ring enol silyl ether **1l** afforded siloxycyclopropane **2l** as the major product even under B, the predominant formation of isomerized **3l** was attained by the use of 'highly concentrated' conditions (C). The Simmons–Smith reaction of 5- and 6-membered ring enol alkyl ethers, **1a**, **1b**, **1d**, and **1e**, under these conditions (B, C) also gave corresponding allylic isomers, although the isomerization did not

(*Continued on p. 284*)

TABLE 1

THE SIMMONS–SMITH REACTION OF ENOL ETHER 1.

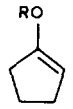
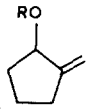
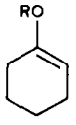
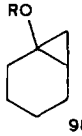
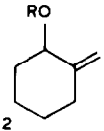
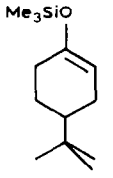
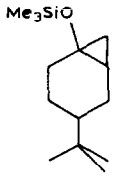
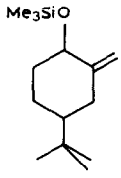
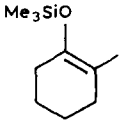
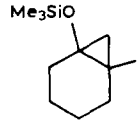
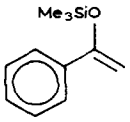
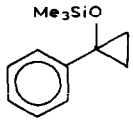
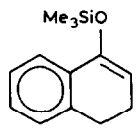
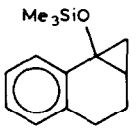
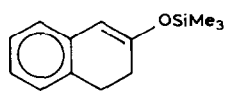
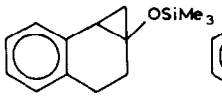
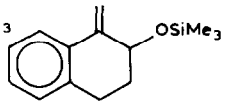
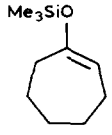
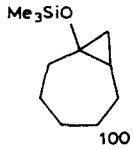
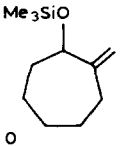
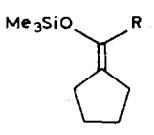
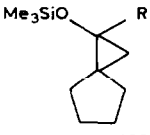
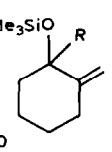
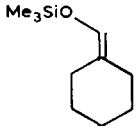
Enol ether (1)	Conditions[a]	Product and ratio (2 : 3)[b]	Total yield [%][c]
			
	A	99 1	73
(1a, R = Me;	B	87 13	60
	C	29 71	62
	A	86 14	69
1b, R = Et;	B	46 54	87
	C	4 96	63
	A	97 3	76
1c, R = Me_3Si)	B	0 100	71
		 	
	A	98 2	72
(1d, R = Me;	B	83 17	59
	C	2 98	66
	A	86 14	69
1e, R = Et;	B	44 56	87
	C	2 98	79
	A	92 8	71
1f, R = Me_3Si)	B	0 100	68
		 	
(1g)	A	91 9	80[d]
	B	0 100	71[d]
			
	A	100	73
(1h)	B	100	78
	C	100	70
			
	A	100	43
(1i)	B	100	40
	C	100	51

TABLE 1 (continued)

				—	
(1j)	A	100			65
	B	100			58
	C	100			63
					
(1k)	A	99	1		64
	B	82	18		66
	C	52	48		56
					
(1l)	A	100	0		61
	B	99	1		77
	C	16	84		65
					
(1m, R = Me)[f]	A	100	0		78[e]
	A'[g]	99	1		65[e]
	B	97	3		80[e]
	B'[h]	91	9		82[e]
	C	25	75		77[e]
	C'[i]	8	92		66[e]
(1n, R = H)[f]	A	100	0		87[e]
	B	99	1		73[e]
	C	80	20		73[e]
	C''[j]	15	85		80[e]
				—	
(1o)	A	100			58[e]
	B	100			66[e]
	C	100			86[e]

[a] See text. [b] Determined by GLC. [c] Isolated yield unless noted. [d] A mixture of 1:1 stereoisomers. [e] GLC yield. [f] The reaction was carried out in one tenth (5 mmol) reaction scale. [g] Ether: 5.5 ml. [h] Ether: 3.2 ml. [i] Ether: 2 ml. [j] Ether: 0.5 ml.

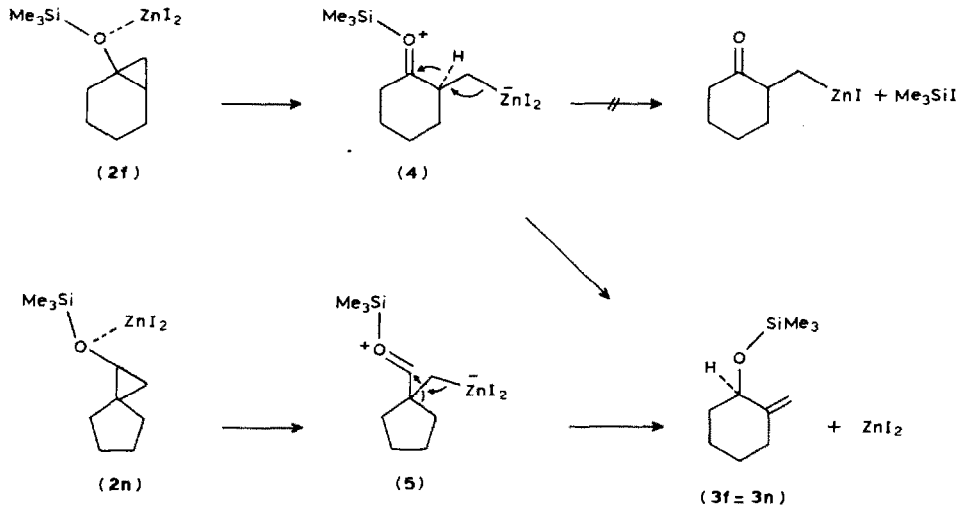
proceed as efficiently as for the siloxy analogues *. Ring cleavage was restricted to the peripheral carbon in each case, and ring-opened isomers due to central bond cleavage were not detected at all.

No isomerization took place in the case of **1h**, and this is probably due to the relative difficulty of the carbon migration compared to hydrogen migration. However, employment of the more strained system, which gave the spiro[4.2]-adducts **2m** and **2n** in the initial Simmons–Smith reaction, led to ring-enlarged **3m** and **3n**, respectively, after isomerization with skeletal rearrangement. It seems likely that ring strain is the driving force, thus, the attempted isomerization of less-strained **2i** and **2o** was unsuccessful. No allylic isomer was, however, obtained in the Simmons–Smith reaction of **1j**, although it did give moderately strained **2j** (vide infra).

4. The role of zinc iodide in the cyclopropyl to allylic isomerization

Under the usual Simmons–Smith reaction conditions zinc iodide is completely solvated by coordinated ether molecules. The notable ease of isomerization, observed in the experiments under higher concentrations (see Fig. 1), may indicate the importance of coordinated species such as **2f** or **2n**, which are the result of replacing one of the coordinated ether molecules with siloxycyclopropanes [10,11]. The fact that in less electron-donating solvents, such as benzene, isomerization proceeded more smoothly (vide infra, Table 2) might lend support to this hypothesis. The possible pathway to allylic isomers involving ionic intermediates is illustrated in Scheme 1 **.

There are two possible reaction courses that the proposed ionic intermediate **4** or **5** can follow: (i) 1,2-migration to the oxonium cation to allylic isomers, and (ii)



SCHEME 1

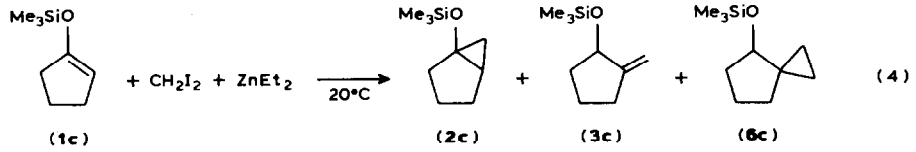
* The Simmons–Smith reaction of enol ether **1d** has been published [9].

** Bond breaking and 1,2-shift may take place simultaneously.

desilylation to afford β-iodozinc substituted ketones [12]. The latter process seems quite unsuitable because of the expected facile reverse reaction or unfavorable formation of trimethylsilyl iodide, a combination of hard acid and soft base. The importance of the ionic intermediates may be supported by the fact that the α-cation- or β-cation-stabilizing substituents [13] enhanced the isomerization (see the results of conditions B for **1d**, **1e**, and **1f** or conditions C for **1m** and **1n**). However, the results of **1j** which afforded no allylic isomer in spite of moderate ring strain seem rather peculiar from this viewpoint. Steric congestion between the siloxy group and peri-hydrogen associated with the required inversion of the configuration of siloxy carbon may prevent isomerization. There have been some other examples of unusual Simmons–Smith reaction yielding ring-opened products [14,15,16] probably induced by zinc iodide, which promotes isomerization of highly strained molecules [17,18,19]. The present rearrangement of cyclopropyl to allylic may be added to these precedents as one of a relatively low-strained class of isomerization. The attached RO group may well compensate for the lack of strain release during the reaction.

5. The reaction of enol silyl ethers with diethylzinc / methylene iodide

The modified Simmons–Smith reaction using diethylzinc/methylene iodide produces ethylzinc iodide as the first by-product after the transfer of methylene group, and this by-product is believed to exist in equilibrium with diethylzinc and zinc iodide [20]. Since diethylzinc operates as a divalent species, a molar ratio of less than one of diethylzinc to methylene iodide should produce zinc iodide as a by-product. Thus, we again tested the possibility of the formation of allylic isomers in this system by using enol silyl ether **1c** as a starting substrate (eq. 4).



Interestingly, the reaction afforded three products. **2c**, **3c**, and **6c**. The formation of spiro ether **6c** is due to further cyclopropanation of allylic isomer **3c**. For the selective preparation of cyclopropanated product **2c**, an increase in the amount of diethylzinc (3 molar excess) was rather effective for this particular zinc carbenoid. The use of the similar 'dilute' conditions with ether (10 ml of solvent for 5 mmol of

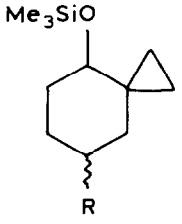
TABLE 2

THE REACTION OF **1c** WITH DIETHYLZINC-METHYLENE IODIDE [a]

Solvent	Ratio of Products [b] (**2c** : **3c** : **6c**)			Total yield [%] [b]
anisole	48	48	4	78
benzene	44	46	10	75
pentane	98	2	0	81
ether	99	1	0	70

[a] Reaction conditions: **1c**, 5 mmol; methylene iodide, 6 mmol; diethylzinc, (3.8 mmol); solvent, 10 ml; 20°C, 40 h. [b] Determined by GLC.

substrate) proved to be valid only for cyclopropanation (see Table 2). Interestingly, for the selective preparation of cyclopropyl ethers, a hydrocarbon solvent such as pentane caused the zinc iodide produced to aggregate immediately and so lose its activity [21]. In an aromatic solvent, such as benzene or anisole, isomerization by zinc iodide proceeded fairly smoothly even under 'dilute' conditions. Since the second cyclopropanation was fast, we failed to obtain allylic products in good selectivity in the diethylzinc/CH_2I_2 system. We carried out the one-pot conversion of **1c** to spiro ether **6c** via three consecutive reactions (cyclopropanation, isomerization, cyclopropanation) in benzene. Good yields were achieved with a large excess of zinc carbenoid reagent. The procedure was also proven to be applicable to six-membered **1f** and **1g**, which afforded good yields of spiro ethers **6f** and **6g**, respectively [21,22] *.



(**6f**, R = H;
6g, R = t-Bu)

Experimental

Boiling points are uncorrected. Infrared spectra were recorded on a Shimadzu IR-400 instrument as neat liquids. ^1H NMR (100 MHz) were recorded on a JEOL JNM-PS-100 spectrometer using TMS as internal standard in CCl_4. Mass spectra were obtained on a Hitachi mass spectrometer Model RMU-6E. Elemental compositions were determined by elemental analyses or by high resolution mass spectra for gas-chromatographically pure (100%) samples. GLC data were obtained with a Shimadzu GC-3BF equipped with a flame ionizing detector. Unless otherwise specified, a stainless steel column (3 mm × 3.8 m) was used, packed with 5% OV-1 on Uniport KS or 10% SE-30 on Chromosorb W. When necessary, pure samples were obtained by preparative GLC with a Varian Aerograph Model 90-P. The preparation of enol alkyl ethers was by the method of Wohl [23] and enol silyl ethers were obtained by the method of House [24]. Anhydrous diethyl ether was distilled over Na/Pb alloy prior to use. Zinc/copper couple was made in situ from zinc and cuprous chloride by Rawson's method [25].

A time course study of the Simmons–Smith reaction of 1-ethoxycyclohexene (1c)

To a stirred suspension of zinc/copper couple (10.4 g, 0.16 g-atom) in anhydrous ether (40 ml) were added **1e** (10.24 g, 0.08 mol), and *p*-tolyl methyl ether (19.52 g, 0.16 mol) to serve as the internal standard for GLC analysis. Methylene iodide (32.16 g, 0.12 mol) was added, and the mixture heated under reflux. Small aliquots

(*Continued on p. 289*)

* Application of this one-pot conversion to 6-methyl-siloxycyclohexene has been recently published [22].

TABLE 3

SPECTRAL DATA OF PRODUCTS 2, 3, AND 6 [a]

Product	b.p. (°C/mmHg)	IR (film) (cm^{-1})	MS $m/e(M^+)$	^1H NMR(CCl_4) (ppm/TMS)
2a	74/114	3020 (cyclopropane)	112	0.50(dd, 1H) 0.70(dd, 1H) 0.90–2.10(m, 7H) 3.25(s, 3H)
2b	74–75/75	3020	126	0.45(dd, 1H) 0.70(dd, 1H) 0.90–2.10(m, 7H) 1.12(t, 3H) 3.45(q, 2H)
2c	66–68/13	3030	170	0.09(s, 9H) 0.33–2.05(m, 9H)
2d	104/134	3030	126	0.03(dd, 1H) 0.17(dd, 1H) 0.50–2.30(m, 9H)
2e	59/18	3030	140	0.14(dd, 1H) 0.75(dd, 1H) 0.75–2.20(m, 9H) 1.19(t, 3H); 3.39(q, 2H)
2f	70–71/24	3030	184	0.07(s, 9H) 0.00–2.40(m, 11H)
2g [b]	120–123/17	3040	240	0.06(s, 9H) 0.00–2.40(m, 19H; incl. 0.79, s, 0.81, s)
2h	72–75/7	3040	198	0.09(s, 9H) 0.35(s, 2H) 0.85–2.10(m, 11H)
2i	90–95/10	3010, 3050	206	0.02(2, 9H) 0.86–1.09(m, 4H) 7.20(br.s, 5H)
2j	139–143/16	3027, 3061	232	0.09(s, 9H) 0.75–2.79(m, 7H) 6.74–7.68(m, 4H)
2k	82–86/0.2	3020, 3050	232	0.13(s, 9H) 0.97–1.27(m, 2H) 1.77–2.74(m, 5H) 6.76–7.15(m, 4H)
2l	97–99/16	3020	198	0.10(s, 9H) 0.15–2.45(m, 13H)
2m	74/14	3020	198	0.12(2, 9H) 0.28–0.60(m, 2H) 1.33(s, 3H) 1.30–2.04(m, 8H)
2n	63–65/13	3020	184	0.08(s, 9H) 0.24–0.64(m, 2H) 0.88–1.88(m, 8H) 2.94–3.08(dd, 1H)
2o	93–95/23	3100	198	0.10(s, 9H) 0.15–0.42(m, 2H) 1.00–1.50(m, 10H) 2.92(dd, 1H)

(continued)

TABLE 3 (continued)

Product	b.p. (°C/mmHg)	IR (film) (cm^{-1})	MS $m/e(M^+)$	^1H NMR(CCl_4) (ppm/TMS)
3a	104/134	1650 (ν(C=C))	112	0.90–2.45(m, 6H) 3.24(s, 3H) 3.65–3.85(m, 1H) 4.95(brs, 2H)
3b	53–54/28	1650	124	1.15(t, 3H) 1.40–2.55(m, 6H) 3.28(dq, 2H) 3.85(m, 1H) 4.90(brs, 2H)
3c	60–61/18	1665	170	0.09(s, 9H) 1.34–2.47(m, 6H) 4.11–4.47(m, 1H) 4.72–5.41(m, 2H)
3d	72–73/48	1660	126	1.20–2.30(m, 8H) 3.15(s, 3H) 3.50(m, 1H) 4.72(brs, 2H)
3e	87–88/73	1658	140	0.90–2.55(m, 8H) 1.15(t, 3H) 3.32(dq, 2H) 3.60(m, 1H) 4.72(brs, 2H)
3f = 3n	60–65/10	1660	184	0.09(s, 9H) 0.97–2.74(m, 8H) 3.86–4.19(m, 1H) 4.62(s, 1H) 4.75(s, 1H)
3g [b]	108–115/6	1655	240	0.06(s, 9H) 0.88(s, 9H) 0.98–2.19(m, 7H) 4.11(m, 1H) 4.62(brs, 1H) 4.80(brs, 1H)
3k	82–83/0.35	1630	232	0.09(s, 9H) 1.70–3.14(m, 4H) 4.40(m, 1H) 5.16(brs, 1H) 5.42(brs, 1H) 6.70–7.20(m, 3H) 7.36–7.58(m, 1H)
3l	74–78/10	1650	198	0.07(s, 9H) 1.00–2.45(m, 10H) 4.10–4.35(m, 1H) 4.20–5.00(m, 2H)
3m	76/174	1642	198	0.09(s, 9H) 1.35(s, 3H) 1.55(c, 8H) 4.23(brs, 1H) 4.76(brs, 1H)
6c	80–82/19	3055	184	0.04(s, 9H) 0.00–0.86(m, 4H) 1.14–2.12(m, 6H) 3.63(dd, 1H)

TABLE 3 (continued)

Product	b.p. (°C/mmHg)	IR (film) (cm^{-1})	MS $m/e(M^+)$	^1H NMR(CCl_4) (ppm/TMS)
6f	74–76/8	3055	198	0.05(s, 9H) 0.00–0.50(m, 4H) 1.00–2.00(m, 8H) 3.14–3.36(m, 1H)
6g	78–79/0.4	3060	254	0.06, 0.08(s, s, 9H) 0.00–0.80(m, 4H) 0.84, 0.86(s, s, 9H) 2.88, 3.64(c, dd, 1H)

[a] Satisfactory microanalyses or high resolution mass spectrum data were obtained for all new compounds. [b] Data for a 1:1 mixture of stereo isomers.

of the mixture were occasionally withdrawn by syringe, treated with aqueous ammonium chloride, and analyzed by GLC (10% PEG 20M on Chromosorb W, 3 mm × 3 m). The results are shown in Fig. 1.

*Reaction of 1-trimethylsiloxybicyclo[4.1.0]heptane (**2f**) with zinc iodide*

Zinc iodide was prepared in situ by treating zinc/copper couple (15 mmol) with iodine (15 mmol) in anhydrous ether (35, 15, or 7 ml). To this solution was added **2f** (2.26 g, 15 mmol), and the mixture was heated under reflux for 40 h with stirring. After the usual aqueous work up, the extract was dried over $MgSO_4$ and analyzed by GLC. When 35 ml of ether was used, the starting material, **2f**, was recovered almost unchanged, while the latter two 'concentrated' cases resulted in the formation of isomerized **3f** in 29 and 88% product ratio, respectively.

*General procedure for the Simmons–Smith reaction of enol ethers **1a–1o** leading to cyclopropyl ethers **2a–2o** under conditions A*

A mixture of zinc powder (10.4 g, 0.16 g-atom) and cuprous chloride (1.60 g, 0.016 mol) in anhydrous ether (110 ml: conditions A) was stirred under reflux for 2 h. Enol ether **1** (0.05 mol) and methylene iodide (0.08 mol) were then successively added dropwise, and the mixture stirred for 40 h under reflux. After cooling, the remaining zinc was filtered off and the filtrate washed twice with cold aqueous ammonium chloride (50 ml). The aqueous layer was extracted with two 20 ml portions of ether. The combined extracts were washed with aqueous sodium bicarbonate (50 ml) and water (50 ml), and then dried over magnesium sulfate. The solvent was removed under reduced pressure, and the resulting oil was distilled to give cyclopropyl ether (**2**) in good yield.

*General procedure for the Simmons–Smith reaction of enol ethers **1a–1o** under concentrated conditions B or C*

Procedures similar to those above were followed, except for decreasing the amounts of the ether, where either 40 ml (conditions B) or 25 ml (conditions C) of ether were used for the 0.05 mol (enol ether) scale experiments. As stated in the text, allylic ethers **3a, 3b, 3c, 3d, 3e, 3f, 3g, 3k, 3l, 3m,** and **3n(= 3f)** were obtained with good selectivity from corresponding enol ethers under these conditions.

General procedure for the cyclopropanation of enol silyl ethers by reaction with diethylzinc / methylene iodide

To a stirred solution of enol silyl ether **1** (**1c, 1f,** or **1l**) (5 mmol) and solvent

(pentane, 10 ml) was added diethylzinc (0.38 ml, 3.8 mmol) by syringe under a stream of nitrogen. Methylene iodide (1.61 g, 6 mmol) was then, added dropwise (caution exothermic!) and stirring was continued for 20 h at 15°C. The mixture was then subjected to aqueous work up (aq. NH_4Cl, aq. $NaHCO_3$, H_2O), and dried over $MgSO_4$. After removal of the solvent, **2** was obtained by vacuum distillation. Yields of **2**: **2c**, 81%; **2f**, 82%, **2l**, 75%. Use of ether instead of pentane also afforded very pure cyclopropanated products under these dilute conditions. Yields of **2**: **2c**, 70%; **2f**, 78%; **2l**, 79%.

General procedure for one-pot synthesis of 4-trimethylsiloxyspiro[n.2]alkanes ***6c, 6f, 6g***

To a stirred solution of enol silyl ether **1f** (0.85 g, 5 mmol), diethylzinc (1.0 ml, 10 mmol), and dry benzene (2 ml) was added methylene iodide (2.68 g, 10 mmol) during 20 min at 20°C. After further stirring for 20 h, additional methylene iodide (2.14 g, 8 mmol) was added in one portion (slightly exothermic, the temperature reached 40°C) to complete the reaction. The mixture was then stirred for another 2 h, diluted with ether (20 ml) and poured into cold aqueous ammonium chloride (50 ml). The aqueous layer was extracted twice with ether (2 × 10 ml). The combined ether extracts were washed with aq. sodium bicarbonate then water, and dried over $MgSO_4$. After removal of the solvent, the residue was distilled to give 0.65 g (66%) of 4-trimethylsiloxyspiro[5.2]octane (**6f**), (b.p. 83–85°C/20 mmHg). Similarly **6c** and **6g** were obtained from, **1c** and **1g** in 63% and 71% yields, respectively.

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