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Silicon assisted diversified reaction of a β-silylmethylene malonate with dimethylsulfoxonium methylide

Pintu K. Kundu, Rekha Singh, Sunil K. Ghosh *

Bio-Organic Division, Bhabha Atomic Research Centre, Trombay, Mumbai 400 085, India

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ABSTRACT

Reaction of dimethylsulfoxonium methylide with a β-silylmethylene malonate gives diversified products, cyclopropane, cyclobutane or allyl and homoallyl silanes depending upon the stoichiometry of the reactants and reaction conditions. Contrary to this, reaction of a β-arylmethylene malonate gives only the cyclopropane product. The product(s) formed are unique and the silicon group played a crucial role either by assisting and/or by participating in the process.

1. Introduction

The unique properties of silicon [1,2] have led to its wide utilization in organic chemistry ranging from protecting functional groups [3], forming temporary tether [4–6] and masking hydroxyl group [7,8], to highly controlled and selective organic reactions [1,2,9]. The stabilization of either an electron deficient center such as a carbocation at the β-position (β-effect) [10–12] or a carbanion at the α-position (α-effect) [13] with respect to a silicon group is the central theme of organosilicon chemistry, applied to organic synthesis. The former effect is clearly demonstrated by the enhanced rates of unimolecular solvolysis [14] of β-(trimethylsilyl) esters compared to the corresponding silicon-free analogues. The regio- and stereo-selectivity of electrophilic substitution reactions [9,15] of allyl-, vinyl- and aryl-silanes have been shown to be controlled by the β-effect. This stabilizing effect also plays an important role in directing the regioselectivity in various organic reactions. Some recent examples include Baeyer–Villiger oxidation [16], Bamford–Stevens reaction [17], Beckmann fragmentation [18,19], Curtius reaction [20], Norrish types I and II cleavages [21,22], palladium-catalyzed nucleophilic substitution [23], Nazarov cyclizations [24,25], decarboxylation reactions [26] and stereospecific 1,2-silyl shifts [27,28]. The propensity of β-elimination of organosilicon compounds was first demonstrated by Peterson [29,30] for olefin synthesis. The starting β-silyl alcohols were conveniently prepared by the reaction of a stabilized α-silyl carbanion with carbonyl compounds. Besides, silicon has unique affinity for fluorine and oxygen, which makes the silicon chemistry highly selective as exemplified in Brook rearrangement [31–33] and cross-coupling reactions [34–38].

Sulfonium and sulfoxonium ylides [39–43] are extensively used in organic chemistry to achieve the stepwise insertion of a methylene or a substituted methylene across the double bond of a carbonyl, an imine, or an electrophilic olefin to yield an oxirane, an aziridine, or a cyclopropane, respectively. Besides their difference in stability, the outcomes of many reactions with these ylides are similar, although, some notable differences in their reactivity and selectivity are also reported [44]. Recently, we and others have shown that dimethylsulfonium methylide (DIMSY) (**1**) when used in excess, or in the presence of a base can act as an equivalent of a carbenoid to produce interesting product(s) when reacted with various Michael acceptors [45–52], activated dienes [53], carbonyl compounds, imines and epoxides [54–57]. For example, when DIMSY was reacted with β-silylmethylene or β-arylmethylene malonates **2**, cyclopropane derivatives **3** or olefins **4** were obtained [45–52] depending upon the quantities of the ylide and base used (Scheme 1).

We were therefore interested to know if excess dimethylsulfoxonium methylide (DIMSOY) (**5**) would react in similar fashion with β-silylmethylene malonate **2a** (R = $SiMe_2Ph$, E = CO_2Et). Interestingly, when excess DIMSOY, generated from the reaction of trimethylsulfoxonium iodide and sodium hydride in dimethyl sulfoxide (DMSO) was reacted with the β-silylmethylene malonate **2a** at room temperature, the expected cyclopropane **3a** was formed albeit in moderate yield, associated with two unusual products viz. cyclobutane **6a** and the allylated malonate **7** (Scheme 2). However, when stoichiometric quantity of the ylide **5** was used, besides

* Corresponding author. Tel.: +91 22 25595012; fax: +91 22 255055151.
E-mail address: ghsunil@barc.gov.in (S.K. Ghosh).

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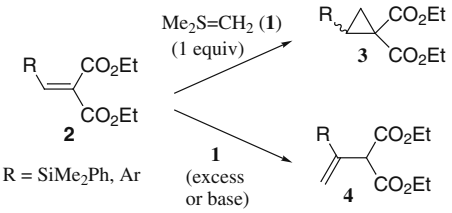


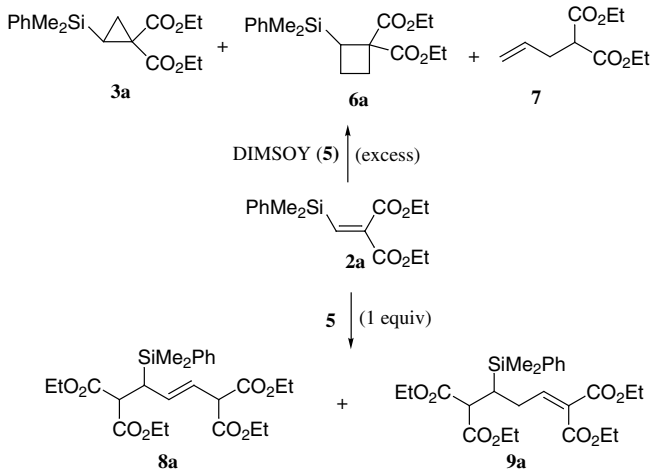
Scheme 1. Reaction of some Michael acceptors with ylide **1** under different conditions.

cyclopropane **3a**, a pair of new products, an allylsilane **8a** and its regioisomeric homoallylsilane **9a** were formed (Scheme 2). The diversified products formed under different conditions therefore challenged us to formulate conditions for individual products. We report, herein, the conditions developed for two interesting groups of products, cyclobutane and allyl/homoallylsilane with very high selectivity merely by adjusting the stoichiometry of the ylide and reaction conditions.

2. Results and discussion

2.1. Preparation of cyclobutane derivative

Cyclobutanes have been known for more than a century but their use as synthetic intermediates has gained popularity very recently [58–61]. The reactivity of cyclobutane derivatives is due to their inherent ring strain. The regioselectivity of bond cleavage in cyclobutane is decided by the ring substituents, reagents and conditions. Although cyclobutane systems are more commonly prepared by cycloaddition, cyclopropane ring expansion and ring contraction reactions, synthesis of suitably functionalized derivatives is still challenging. We, therefore, first aimed to establish the optimized conditions for the preparation of the silicon functionalized cyclobutane derivative **6a**. To this end, we carried out the reactions of **2a** with DIMSOY, generated from trimethylsulfoxonium iodide under different conditions and the results are presented in presented in Table 1. The reaction of **2a** with 2 equiv. of DIMSOY, generated using sodium hydride as base in DMSO at 20 °C provided the cyclopropane **3a** associated with a significant amount of cyclobutane **6a** and the allylated malonate **7** in moderate yield (Table 1, entry 1). Further increase in the DIMSOY quantity or changing the temperature did not improve the yield, but led



Scheme 2. Reaction of β-silylmethylene malonate **2a** with ylide **5** under different conditions.

Table 1
Optimization of conditions for the synthesis of cyclobutane **6a**.

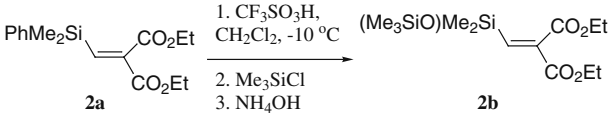
Entry	$Me_3S(O)I$ (equiv.)	Base (equiv.)	Solvent	**3a:6a:7**[a]	% Yield of **3a+6a**[b]
1	2	NaH (2)	DMSO	24:41:35	34[c]
2	2.5	NaH (2.5)	DMSO	49:2:49	nd[d,e]
3	2.5	NaH (2.5)	DMSO	28:4:68	nd[d,f]
4	2	LiOBu-*t* (2)	DMSO	17:83:0	60
5	2.5	LiOBu-*t* (2.5)	DMSO	13:87:0	71
6	3	LiOBu-*t* (3)	DMSO	12:88:0	69
7	2.5	LiOBu-*t* (2.5)	DMF	13:87:0	69
8	2.5	LiOBu-*t* (2.5)	THF	68:32:0	19
9	2.5	NaOBu-*t* (2.5)	DMSO	43:57:0	61
10	2.5	KOBu-*t* (2.5)	DMSO	59:41:0	60
11	2.5	LiOBu-*t* (2.5)	DMSO	12:88:0	73

[a] Ratios determined by ^{1}H NMR from the crude product.
[b] All the reactions (except entries 2 and 3) were performed at 20 °C.
[c] 20% of **7** was also isolated.
[d] Yield not determined.
[e] Reaction was performed at −10 °C.
[f] Reaction was performed at 50 °C.

to substantial formation of the undesired byproduct **7** at the expense of the target cyclobutane **6a** (Table 1, entries 2 and 3). Changing the base to LiOBu-*t* (generated from *n*-butyl lithium and *tert*-butanol) in DMSO improved the product yield with overwhelming preference for the cyclobutane **6a**. Amongst the solvents studied, *N*,*N*-dimethyl formamide (DMF) showed comparable yields and selectivity. The cation in the base had a dramatic influence on the selectivity of the formation of cyclobutane **6a** over cyclopropane **3a**, and increased in the order Li > Na > K. Hence, the best condition was to use 2.5 equiv. each of LiOBu-*t* and trimethylsulfoxonium iodide with respect to malonate **2a** in DMSO at 20 °C, which gave cyclobutane **6a** and cyclopropane **3a** (**3a**:**6a** = 13/87) in 71% isolated yield (Table 1, entry 5). Increasing the amount of ylide **5** beyond 2.5 equiv. neither enhance the yield nor selectivity (Table 1, entry 6). The effect of counter ion in the trimethylsulfoxonium salt did not have much impact since the reaction with trimethylsulfoxonium chloride proceeded with similar yield and selectivity (Table 1, entry 11).

To see the generality of this synthetic strategy, we prepared two more silylmethylene malonates **2b**,**c**. The dimethyl(trimethylsilyloxy)silyl substituted methylene malonate **2b** was prepared in 58% yield from **2a** by a selective protiodephenylation of the phenyldimethylsilyl group with trifluromethanesulfonic acid in dichloromethane followed by mixed disiloxane formation by adding excess trimethylsilyl chloride to the reaction mixture and then quenching with ammonium hydroxide (Scheme 3). For the preparation of di-*tert*-butyloxy(phenyl)silyl substituted methylene malonate **2c**, we adopted a procedure similar to the preparation of **2a**. Therefore, the known di-*tert*-butyloxy(phenyl)silyl lithium was prepared from the corresponding silyl chloride [62,63], and reacted with diethyl ethoxymethylene malonate in THF to give **2c** in moderate yield (Scheme 4).

When **2b** was reacted with DIMSOY, generated under the optimized conditions (Table 1, entry 5), a mixture of cyclopropane **3b** and cyclobutane **6b** were formed in moderate yield and selectivity (**3b**:**6b** = 1/2) (Scheme 5). Under these conditions, silylmethylene

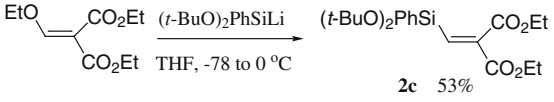


Scheme 3. Preparation of silylmethylene malonate **2b**.

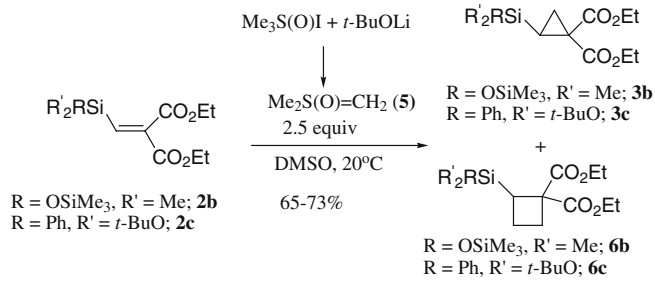
malonate **2c** also reacted with DIMSOY to give a mixture of cyclopropane **3c** and cyclobutane **6c**. In this case, there was a reversal of selectivity wherein cyclopropane **3c** was formed as the major product (**3c**:**6c** = 7/3) (Scheme 5). This suggested a crucial role of the silyl substituents on the course of the reaction.

To find the role played by the silicon group in these reactions, the arylidene malonate **2d**, was reacted with varying amounts of DIMSOY, generated from the corresponding iodide and base. In all the cases, no trace of cyclobutane or the dimerization products was observed. The cyclopropane **3d** was the sole product associated with the unreacted starting material in cases where substoichiometric quantity of ylide **5** was used (Scheme 6).

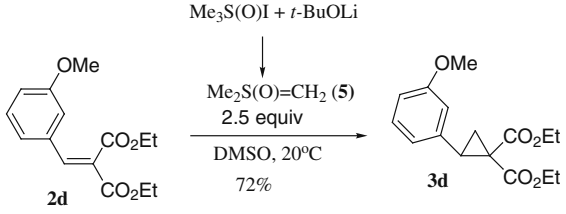
These results, unambiguously established the vital role of the silyl group in the malonates **2a–c** in governing the pathway of the reaction. Evidently, the cyclobutane **6a**, formed in the reaction of **2a** with excess DIMSOY was not produced via the cyclopropane intermediate. In separate experiments, cyclopropane **3a** was found to be unreactive to the ylide **5** even when treated in large excess. Although, cyclopropanes are known not to react with excess DIMSOY to give cyclobutanes, DIMSOY can add to epoxides and suitably N-protected aziridines under harsh conditions to give oxetanes [64–68] and azetidines [69,70] or other products [71–76], depending on the other functionalities present on the substrates. A plausible mechanism for the formation of cyclobutane products from silyl substituted methylene malonates **2** is delineated in Scheme 7. The nucleophilic addition of DIMSOY on the β-silylalkylidene malonate **2** produced the intermediate **10** which can give the cyclopropane **3** by loss of DMSO (path-a). As the silicon group is known to facilitate nucleophilic substitution β-to it, the other favorable pathway (path-b) was the nucleophilic



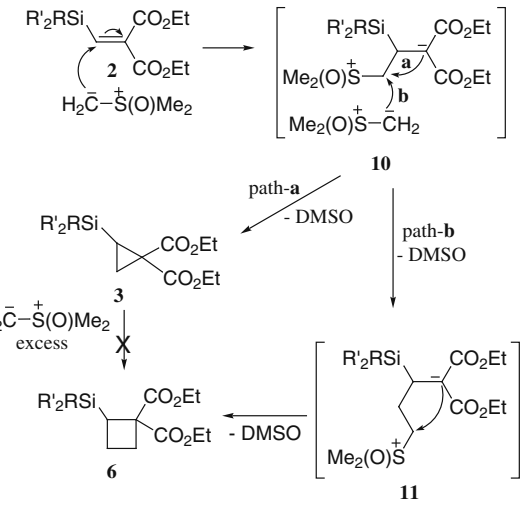
Scheme 4. Preparation of silylmethylene malonate **2c**.



Scheme 5. Reaction of β-silylmethylene malonates **2b** and **2c** with ylide **5** to give **6b** and **6c**.



Scheme 6. Reaction of β-arylmethylene malonate **2d** with ylide **5** to give cyclopropane **3d**.



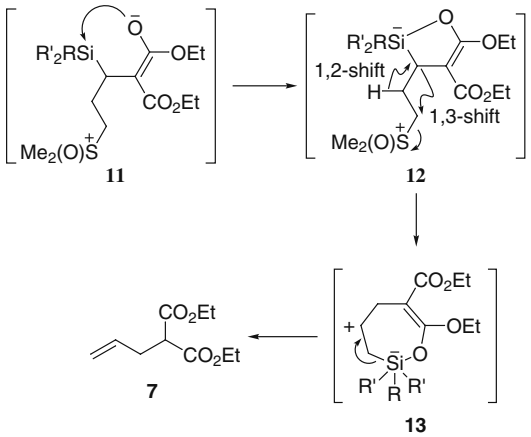
Scheme 7. Plausible mechanism for the formation of cyclobutane **6**.

displacement of DMSO on **10** by ylide **5** to give the intermediate **11**, which on intramolecular cyclization provided the cyclobutane **6** with expulsion of DMSO. As the silicon group becomes larger, path-b becomes unfavorable owing to the steric effect. This was clearly manifested in case of **2c** where cyclopropane **3c** was obtained as the major product.

Although diethyl allylmalonate **7** can be readily prepared from diethyl malonate and allyl bromide, its formation in the reaction of silymethylene malonates **2** with excess DIMSOY is somewhat unusual. It is well established that cyclopropylmethyl halides, cyclobutyl halides and homoallyl halides are all in equilibrium in acid solutions and the mixture of products are often formed via delocalized cationic intermediate [77–87]. But, under present circumstances, this situation is unlikely. We believe that the silyl group plays an important role in this reaction also. We propose that the malonate **7** is also formed from the intermediate anion **11** as depicted in Scheme 8. The enolate form of **11** probably formed a pentacoordinated silicate [88,89] species **12** which facilitated a γ-silyl group assisted (neighboring group participation) [90,91] loss of DMSO to give a primary carbocation that initiated a 1,2-hydride shift induced 1,3-silyl shift to give a β-silicon stabilized cationic intermediate **13**. Loss of silyl group from it then yields **7**.

2.2. Preparation of allyl and homoallylsilanes

Next, we turned our attention towards the preparation of the functionalized allylsilane **8a**, obtained by the same reaction under

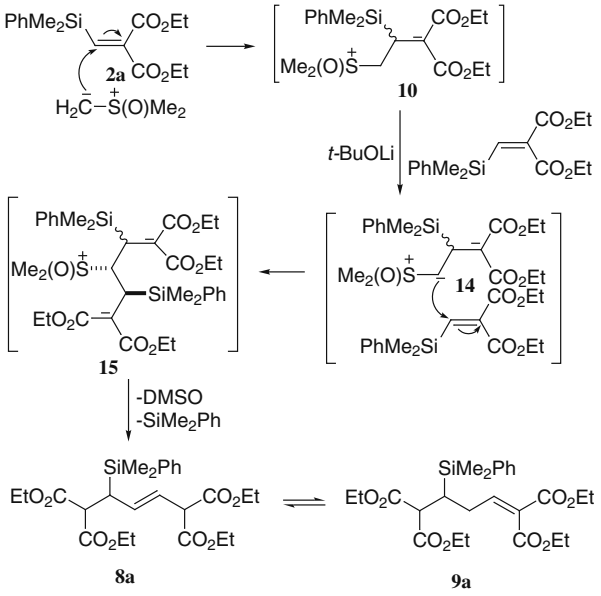


Scheme 8. Plausible mechanism for the formation of allylated malonate **7**.

Table 2
Optimization of conditions for the synthesis of olefins **8a** and **9a** from **2a** using 1.1 equiv. of *t*-BuOLi and 0.5 equiv. of trimethylsulfoxonium iodide.

Entry	Solvent (temp)	% Yield of **8a**+**9a**[a]	% Yield of **3a**[b]
1.	DMSO (5 °C)	55	13
2.	DMF (15 °C)	58	11
3.	DMF (5 °C)	65	12
4.	DMF (0 °C)	51	11
5.	NMP (5 °C)	67	11

[a] Refers to combined isolated yield.
[b] Product contaminated with 8–10% of **6a**.



Scheme 9. Plausible mechanism for the formation of allylsilane **8a** and homoallylsilane **9a**.

stoichiometric condition (Scheme 2). Allylsilanes are important intermediates in organic synthesis and their use in stereocontrolled organic reactions are well documented [9,92–96]. The homoallylsilane **9a** and the allylsilane **8a** are essentially labile olefin regioisomers, and are interconvertible under mild conditions. Their structures suggest that these products are formed from two molecules of malonate **2a**. Therefore, while optimization, the ylide quantity was reduced to half that of the malonate **2a**. A large number of reaction conditions were tried to optimize the formation of **8a** and/ or **9a** as presented in Table 2. The reactions were performed using 0.5 equiv. of DIMSOY generated from 0.5 equiv. of trimethylsulfoxonium iodide and 1.1 equiv. of *t*-BuOLi (with respect to **2a**) at a temperature range of 0–15 °C using different solvents like DMSO, DMF and *N*-methyl pyrrolidone (NMP). The best result was obtained by carrying out the reaction with 0.5 equiv. of ylide **5** in DMF or NMP at 5 °C (Table 2, entries 3 and 5).

The formation pathway of allylsilane from β-silylalkylidene malonate **2a** under substoichiometric amount of ylide and stoichiometric amount of base is exemplified in Scheme 9. The ylide **5** adds to malonate **2a** to give the adduct **10** from which the base abstracts a proton to generate a new ylide **14**. This, in turn, reacts with another molecule of malonate **2a** to give the intermediate **15**. A Peterson type olefination with elimination of $PhMe_2Si$ and $Me_2S(O)^+$ groups afforded the allylsilane **8a**. The homoallylsilane **9a** is then formed by base induced double bond isomerization of the allysilane.

3. Conclusion

In conclusion, we have illustrated for the first time the reaction of silymethylene malonates with DIMSOY, which produces diversified products depending upon the stoichiometry of the reactants and reaction conditions. The product(s) formed are unique, and the silicon group played crucial role either by assisting and/or by participating in the process. The cyclopropanes, cyclobutanes, allyl and homoallyl silanes, produced in this method have synthetic potentials as the chemistry of these classes of molecules is well established. In addition, the present work also exemplified the unique difference of reactivity of DIMSOY and DIMSY with silymethylene and /or arylmethylene malonates.

4. Experimental

4.1. Materials and methods

All reactions were performed in oven-dried (120 °C) or flame-dried glass apparatus under dry N_2 or argon atmosphere. Tetrahydrofuran (THF) was dried from sodium/benzophenone while *t*-BuOH, DMSO, DMF and NMP were dried from CaH_2 followed by storage over 4 Å molecular sieves. *n*-BuLi (1.5 M in hexane), $Me_3S(O)I$ and Me_3SI were purchased from Aldrich. Compounds **2a** and **2d** were prepared following our reported procedure [46]. The column chromatography was performed on silica gel (230–400 mesh). ^1H NMR and ^{13}C NMR spectra were recorded with a Bruker 200 and 500 MHz spectrometers. Spectra were referenced to residual chloroform (δ 7.25 ppm, ^1H; δ 77.00 ppm, ^{13}C). The mass spectra were recorded on a Shimadzu GC–MS 2010 mass spectrometer (EI 70 eV). High resolution mass spectra were recorded at 60–70 eV with a Waters Micromass Q-TOF spectrometer (ESI, Ar). Infrared spectra (IR) were recorded on a JASCO FT IR spectrophotometer in NaCl cells or in KBr discs. Peaks are reported in cm^{-1}. Melting points (mp) were determined on a Fischer John's melting point apparatus and are uncorrected. Gas chromatography (GC) studies were carried out using Younglin Acme 6000 M Gas Chromatograph fitted with a capillary column (WCOT Fused Silica, CP-SIL-5-CB, 50 m × 0.25 mm/0.39 mm, 0.25 μm; carrier: helium 1 mL/min).

4.2. Diethyl dimethyl(trimethylsilyloxy)silylmethylene malonate (*2b*)

Trifluoromethanesulfonic acid (4.3 mL, 49 mmol, 5 equiv.) was added to a stirred solution of **2a** (3 g, 9.8 mmol) in dry CH_2Cl_2 (15 mL) at −2 °C. After 10 min, chlorotrimethylsilane (12.5 mL, 98 mmol, 10 equiv.) was added to the reaction mixture. The reaction mixture was poured into ice-cold saturated NH_4OH solution (100 mL) and extracted with $CHCl_3$. The organic extract was dried over anhydrous sodium sulfate and evaporated under reduced pressure. The residue was purified by chromatography to give **2b** (1.8 g, 58%) as colorless oil. R_f = 0.58 (hexane/EtOAc, 95:5); IR (film, cm^{-1}): 2982, 2958, 2904, 1732, 1370, 1318, 1254, 1198, 1051, 844, 809, 754, 691; ^1H NMR (200 MHz, $CDCl_3$): δ −0.02 (s, 9H, Me_3SiO), 0.13 (s, 6H, Me_2Si), 1.18 (t, 3H, $^3J_{HH}$ = 6 Hz, OCH_2CH_3), 1.20 (t, 3H, $^3J_{HH}$ = 6 Hz, OCH_2CH_3), 4.13 (q, 2H, $^3J_{HH}$ = 6 Hz, OCH_2CH_3), 4.15 (q, 2H, $^3J_{HH}$ = 6 Hz, OCH_2CH_3), 6.99 (s, 1H, SiCH=C); ^{13}C NMR (50 MHz, $CDCl_3$): δ 0.46 (2C), 1.55 (3C), 13.73 (2C), 60.91, 61.11, 140.11, 149.36, 163.78, 165.5; ESI-MS: *m/z* (relative intensity) 341 (43, $[M+Na]^+$), 201(16), 155 (10), 83 (100); HRMS Calc. for $C_{13}H_{26}O_5Si_2Na$ [M+Na]: 341.1217. Found 341.1219.

4.3. Diethyl di-tert-butyloxy(phenyl)silylmethylene malonate (*2c*)

Di-*tert*-butyloxy(phenyl)silyl lithium was prepared following the procedure reported in the literature [63]. For this di-*tert*-butyl-

oxy(phenyl)silyl chloride [62] (8.5 g, 29.6 mmol) was added to a stirred suspension of lithium metal (830 mg, 0.118 g atom) in THF (30 cm^3) at room temperature under argon. After 40 min, the reaction mixture was cooled to 0 °C and stirred for 4 h. This deep red silyl lithium solution was slowly cannulated to a stirred solution of diethyl ethoxymethylene malonate (6.5 mL, 32.6 mmol, 1.1 equiv.) in THF (30 mL) at −78 °C over 15 min. After the addition was over, the reaction mixture was stirred for 30 min and the cold bath was removed. The reaction mixture was allowed to attain to room temperature (about 25 min) and poured into saturated ammonium chloride solution, extracted with Et_2O. The organic extract was washed with water and with brine, dried over anhydrous $MgSO_4$ and evaporated. The residue was purified by column chromatography on silica using hexane/EtOAc (95/5) to give the diester **2c** (6.6 g, 53%) as colorless oil. R_f = 0.62 (hexane/EtOAc, 5:95); IR (film, cm^{-1}): 3070, 2978, 1731, 1615, 1390, 1366, 1331, 1235, 1119, 1046, 899, 870, 830; ^{1}H NMR (200 MHz, $CDCl_3$): δ 1.13 (t, 3H, $^3J_{HH}$ = 7 Hz, OCH_2CH_3), 1.29 (t, 3H, $^3J_{HH}$ = 7 Hz, OCH_2CH_3), 1.29 (s, 18H, OBu-*t*), 3.89 (q, 2H, $^3J_{HH}$ = 6 Hz, OCH_2CH_3), 4.23 (q, 2H, $^3J_{HH}$ = 6 Hz, OCH_2CH_3), 7.12 (s, 1H, SiCH=C), 7.28–7.37 (m, 3H, Ar), 7.62–7.67 (m, 2H, Ar); ^{13}C NMR (50 MHz, $CDCl_3$): δ 13.43, 13.75, 31.62 (6C), 60.48, 61.24, 73.88 (2C), 127.22 (2C), 129.58, 134.38 (2C), 135.30, 140.99, 143.45, 163.44, 165.57; ESI-MS: *m/z* (relative intensity) 445 (17, $[M+Na]^+$), 349 (23), 265 (100), 219 (47), 147 (23); HRMS Calc. for $C_{22}H_{34}O_6SiNa$ [M+Na]: 445.2022. Found: 445.2026.

4.4. Reaction of 2a with 2.5 equiv. of **5** using sodium hydride as base

Sodium hydride (96 mg, 50% in oil, 2 mmol, 2 equiv.) was made oil free by washing with dry hexane and dry DMSO (2 mL) was added into it. Trimethylsulfoxonium iodide (440 mg, 2 mmol, 2 equiv.) was added and the suspension was stirred under argon atmosphere for 30 min. The solution was cooled to 20 °C and a solution of **2a** (306 mg, 1 mmol, 1 equiv.) in dry DMSO (1 mL) was added slowly (10 min) to the reaction mixture. The reaction mixture was stirred at room temperature for 30 min and diluted with water (50 mL), extracted with 5% ethyl acetate in hexane (3 × 30 mL). The combined extract was washed with brine, dried over anhydrous magnesium sulfate and concentrated under reduced pressure. The residue was purified by column chromatography on silica using hexane/EtOAc (97/3) to give the pure cyclopropane **3a** [46] (42 mg, 13%), cyclobutane **6a** (70 mg, 21%) and malonate **7** [97] (40 mg, 20%). Compound **3a**: R_f = 0.49 (hexane/EtOAc, 95:5); IR (film, cm^{-1}): 1731, 1249, 1116; ^{1}H-NMR (200 MHz, $CDCl_3$): δ 0.25 (s, 3H, SiMe), 0.34 (s, 3H, SiMe), 1.10 (dd, 1H, $^3J_{HH}$ = 9.5, 11 Hz, SiCH), 1.18 (t, 3H, $^3J_{HH}$ = 7.1 Hz, OCH_2CH_3), 1.26 (t, 3H, $^3J_{HH}$ = 7.1 Hz, OCH_2CH_3), 1.41 (dd, 1H, $^2J_{HH}$ = 3.4 Hz, $^3J_{HH}$ = 9.5 Hz, CH_AH_BCHSi), 1.52 (dd, 1H, $^2J_{HH}$ = 3.4 Hz, $^3J_{HH}$ = 11 Hz, CH_AH_BCHSi), 3.80–4.30 (m, 4H, 2 × OCH_2Me), 7.30–7.75 (m, 5H, Ph); ^{13}C NMR (50 MHz, $CDCl_3$): δ −3.32, −3.16, 13.80, 14.00, 15.35, 18.38, 33.31, 61.16, 61.56, 127.71 (2C), 129.12, 133.78 (2C), 137.86, 169.07, 170.98; EIMS: *m/z* (relative intensity) 305 (100, M^+−Me), 275 (11), 243 (70.3), 231 (90), 215 (21), 187 (76), 169 (73), 159 (27), 135 (41), 105 (29). Compound **6a**: R_f = 0.45 (hexane/EtOAc, 95:5); IR (film, cm^{-1}): 3070, 2981, 2958, 2904, 1724, 1427, 1371, 1321, 1251, 1135, 1114, 1025, 836 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ 0.34 (s, 3H, SiMe), 0.35 (s, 3H, SiMe), 0.43 (dd, 1H, $^3J_{HH}$ = 11.5, 11.5 Hz, SiCH), 1.20 (dd, 1H, $^2J_{HH}$ = 3 Hz, $^3J_{HH}$ = 11.5 Hz, $SiCHCH_AH_B$), 1.25 (t, 3H, $^3J_{HH}$ = 6.5 Hz, OCH_2CH_3), 1.24–1.27 (m, 1H, CH_AH_BC), 1.28 (t, 3H, $^3J_{HH}$ = 6.5 Hz, OCH_2CH_3), 1.39 (dd, 1H, $^2J_{HH}$ = 3 Hz, $^3J_{HH}$ = 7 Hz, CH_AH_BC), 1.86–1.92 (m, 1H, $SiCHCH_AH_B$), 4.09–4.29 (m, 4H, 2 × OCH_2CH_3), 7.36 (s, 3H, Ar), 7.50–7.51 (m, 2H, Ar); ^{13}C NMR (50 MHz, $CDCl_3$): δ −3.41, −3.27, 13.80, 13.98, 14.81, 22.17, 24.75, 34.56, 60.92 (2C), 127.58 (2C), 128.88, 133.26 (2C), 137.86, 167.94, 170.14; ESI-MS: *m/z* (relative intensity) 357 (100, $[M+Na]^+$), 243 (6), 201 (16), 135 (10), 111 (6); HRMS Calc. for $C_{18}H_{22}O_4SiNa$ [M+Na]: 357.1498. Found 357.1505. Compound **7**: R_f = 0.58 (hexane/EtOAc, 95:5); IR (film, cm^{-1}): 3082, 2984, 2939, 1733, 1644, 1466, 1370, 1336, 1237, 1178, 1033, 916, 856, 733; ^{1}H NMR (200 MHz, $CDCl_3$): δ 1.25 (t, 6H, $^3J_{HH}$ = 7.2 Hz, 2 × OCH_2CH_3), 2.63 (t, 2H, $^3J_{HH}$ = 7.6 Hz, CH_2=$CHCH_2$), 3.41 (t, 1H, $^3J_{HH}$ = 7.6 Hz, $CH(CO_2Et)_2$), 4.18 (q, 4H, $^3J_{HH}$ = 7.2 Hz, 2 × OCH_2CH_3), 5.04 (d, 1H, $^3J_{HH}$ = 9.6 Hz, C=CH_AH_B), 5.10 (d, 1H, $^3J_{HH}$ = 16.8 Hz, C=CH_AH_B), 5.67–5.84 (m, 1H, H_2C=CH–); ^{13}C NMR (50 MHz, $CDCl_3$): δ 13.90 (2C), 32.65, 51.47, 61.21 (2C), 117.31, 133.94, 168.74 (2C).

4.5. Reaction of **2a** with 2.5 equiv. of **5** using lithium tert-butoxide as base

Dry *t*-BuOH (0.26 mL, 2.7 mmol) was added dropwise to *n*-BuLi (1.7 mL, 1.5 M solution in hexane, 2.5 mmol) under argon atmosphere. The solvent was removed under vacuum and the residue was dissolved in dry DMSO (2 mL). The solution was cooled to 20 °C and solid trimethylsulfoxonium iodide (550 mg, 2.5 mmol) was added into it. After 20 min, a solution of **2a** (306 mg, 1 mmol, 1 equiv.) in dry DMSO (1 mL) was added slowly (10 min) to the reaction mixture. The reaction mixture was stirred at room temperature for 30 min and diluted with water (50 mL), extracted with 5% ethyl acetate in hexane (3 × 30 mL). The combined extract was washed with brine, dried over anhydrous magnesium sulfate and concentrated under reduced pressure. The residue was purified by column chromatography on silica using hexane/EtOAc (97/3) to give the pure cyclopropane **3a** (25 mg, 8%) and cyclobutane **6a** (210 mg, 63%).

4.6. Reaction of **2b** with 2.5 equiv. of **5** using lithium tert-butoxide as base

Following the procedure described for the reaction of **2a** with ylide **5**, trimethylsulfoxonium iodide (550 mg, 2.5 mmol), *t*-BuOH (0.26 mL, 2.7 mmol), *n*-BuLi (1.7 mL, 1.5 M solution in hexane, 2.5 mmol) and **2b** (318 mg, 1 mmol, 1 equiv.) gave a mixture of cyclopropane **3b** and cyclobutane **6b** (220 mg, 65%; **3b:6b** = 1/2 by ^{1}H NMR) which could not be separated. Compounds **3b** and **6b** mixture: R_f = 0.47 (hexane/EtOAc, 95:5); IR (film, cm^{-1}): 2981, 2958, 2903, 1728, 1371, 1321, 1284, 1254, 1207, 1135, 1057, 843; ^{1}H NMR (200 MHz, $CDCl_3$): (recognizable peaks for **3b**) δ 0.84 (dd, 1H, $^3J_{HH}$ = 11.2, 9.4 Hz, SiCH); (recognizable peaks for **6b**) δ 0.97 (dd, 1H, $^2J_{HH}$ = 3.4 Hz, $^3J_{HH}$ = 14.4 Hz, $SiCHCH_AH_B$), 1.83–1.98 (m, 1H, $SiCHCH_AH_B$); EIGCMS (column: WCOT Fused Silica, CP-SIL-5-CB, 50 m × 0.25 mm/0.39 mm, 0.25 μm; carrier: helium 1 mL/min; temp.: 60 °C–2 min–10 °C/min–300 °C): t_R 7.34 min (**3b**) (35%); t_R 8.61 min (**6b**) (57%); *m/z* for **3b** (relative intensity): 317 (73, M−Me), 287 (20), 243 (100), 199 (34) 169 (80, 157 (35), 147 (27), 133 (40), 95 (14), 73 (23); *m/z* for **6b** (relative intensity): 346 $[1,M]^+$, 331 (20), 301 (12), 273 (19), 257 (8), 177 (15), 147 (100), 133 (29), 108 (39), 81 (44).

4.7. Reaction of **2c** with 2.5 equiv. of **5** using lithium tert-butoxide as base

Following the procedure described for the reaction of **2a** with ylide **5**, trimethylsulfoxonium iodide (550 mg, 2.5 mmol), *t*-BuOH (0.26 mL, 2.7 mmol), *n*-BuLi (1.7 mL, 1.5 M solution in hexane, 2.5 mmol) and **2c** (422 mg, 1 mmol, 1 equiv.) gave a mixture of cyclopropane **3c** and cyclobutane **6c** (322 mg, 73%; **3c:6c** = 7/3 by ^{1}H NMR) which could not be separated. Compounds **3c** and **6c** mixture: R_f = 0.65 (hexane/EtOAc, 95:5); IR (film, cm^{-1}): 2977, 2933, 1731, 1429, 1366, 1330, 1241, 1194, 1118, 1060, 702; ^{1}H NMR

(200 MHz, $CDCl_3$): (recognizable peaks for **3c**) δ 0.99 (dd, 1H, $^3J_{HH}$ = 9.6, 10.8 Hz, SiCH), 1.46 (dd, 1H, $^2J_{HH}$ = 3 Hz, $^3J_{HH}$ = 10.8 Hz, $SiCHCH_AH_B$), 1.61(dd, 1H, $^2J_{HH}$ = 3 Hz, $^3J_{HH}$ = 9.6 Hz, $SiCHCH_AH_B$); (recognizable peaks for **6c**) δ 0.44 (dd, 1H, $^3J_{HH}$ = 11.6, 14.6 Hz, SiCH), 1.96–2.12 (m, 1H, $SiCHCH_AH_B$); EIGCMS (column: WCOT Fused Silica, CP-SIL-5-CB, 50 m × 0.25 mm/0.39 mm, 0.25 μm; carrier: helium 1 mL/min; temp: 60 °C–2 min–10 °C/min–300 °C): t_R 12.91 min (**3c**) (66%); t_R 13.66 min (**6c**) (31%); *m/z* for **3c** (relative intensity): 363 (100, M−*t*-BuO), 360 (71), 335 (11), 307 (13), 279 (23), 247 (11), 233 (39), 219 (24), 189 (18), 173 (14), 161 (12), 140 (12), 139 (88); *m/z* for **6c** (relative intensity): 450 (2, M^+), 393 (6), 377 (8), 349 (5), 293 (4), 251 (5), 195 (15), 140 (13), 139 (100), 108 (4).

4.8. Reaction of **2d** with 2.5 equiv. of **5** using lithium tert-butoxide as base

Following the procedure described for the reaction of **2a** with ylide **5**, trimethylsulfoxonium iodide (550 mg, 2.5 mmol), *t*-BuOH (0.26 mL, 2.7 mmol), *n*-BuLi (1.7 mL, 1.5 M solution in hexane, 2.5 mmol) and **2d** (278 mg, 1 mmol, 1 equiv.) gave diethyl 2-(3-methoxyphenyl) cyclopropane-1, 1-dicarboxylate **3d** [98] (210 mg, 72%). R_f = 0.44 (hexane/EtOAc, 9:1); IR (film, cm^{-1}): 2981, 2938, 2837, 1725, 1603, 1585, 1491, 1465, 1371, 1280, 1208, 1131, 1032, 991, 862; ^{1}H NMR (200 MHz, $CDCl_3$): δ 0.87 (t, 3H, $^3J_{HH}$ = 7 Hz, OCH_2CH_3), 1.26 (t, 3H, $^3J_{HH}$ = 7 Hz, OCH_2CH_3), 1.65 (dd, 1H, $^2J_{HH}$ = 5.2 Hz, $^3J_{HH}$ = 9.4 Hz, CH_AH_B), 2.11 (dd, 1H, $^2J_{HH}$ = 5.2 Hz, $^3J_{HH}$ = 8 Hz, CH_AH_B), 3.16 (dd, 1H, $^3J_{HH}$ = 8.6, 8.6 Hz, ArCH), 3.74 (s, 3H, ArOMe), 3.84 (q, 2H, $^3J_{HH}$ = 7 Hz, OCH_2CH_3), 4.14–4.29 (m, 2H, OCH_2CH_3), 6.72–6.77 (m, 3H, Ar), 7.10–7.18 (m, 1H, Ar); ^{13}C NMR (50 MHz, $CDCl_3$): δ 13.58, 13.95, 18.76, 32.00, 37.30, 55.05, 61.01, 61.57, 112.96, 114.03, 120.69, 128.97, 136.19, 159.31, 166.48, 169.73.

4.9. Reaction of **2a** with 0.5 equiv. of **5** using lithium tert-butoxide as base

Dry *t*-BuOH (0.1 mL, 1 mmol) was added dropwise to *n*-BuLi (0.67 mL, 1.5 M solution in hexane, 1 mmol) under argon atmosphere. The solvent was removed under vacuum and the residue was dissolved in dry *N*-methyl pyrrolidone (1 mL). The solution was cooled to 20 °C and solid trimethylsulfoxonium iodide (110 mg, 0.5 mmol) was added into it. After 20 minutes, this ylide solution was added dropwise to neat **2a** (306 mg, 1 mmol) over 15 min at 5 °C under argon atmosphere. The reaction mixture was allowed to attain to room temperature and stirred for 20 min, diluted with water (30 mL) and extracted with 10% ethyl acetate in hexane (3 × 30 mL). The combined extract was washed with brine, dried over anhydrous magnesium sulfate and concentrated under reduced pressure. The residue was purified by column chromatography on silica using hexane/EtOAc (96/4) to give the pure allylsilane **8a** (10 mg, 4%) and homoallylsilane **9a** (155 mg, 63%). A small amount of a mixture of cyclopropane **3a** and cyclobutane **6a** (36 mg, 11%) was also isolated. Compound **8a**: R_f = 0.34 (hexane/EtOAc, 95:5); ^{1}H NMR (200 MHz, $CDCl_3$): δ 0.32 (s, 6H, 2 × $SiMe_3$), 1.12–1.29 (m, 12 H, 4 × OCH_2CH_3), 2.60 (t, 1H, $^3J_{HH}$ = 9.4 Hz, SiCH), 3.42 (d,1H, $^3J_{HH}$ = 8.6 Hz, $CH(CO_2Et)_2$), 3.88–4.22 (m, 9H, 4 × OCH_2CH_3, $CH(CO_2Et)_2$), 5.54 (dd, 1 H, $^3J_{HH}$ = 8.6, 15.4 Hz, CH=CH), 5.72 (dd, 1H, $^3J_{HH}$ = 15.4, 10 Hz, CH=CH), 7.32–7.49 (m, 5H, Ar). Compound **9a**: R_f = 0.34 (hexane/EtOAc, 95:5); IR (film, cm^{-1}): 3070, 2982, 2938, 2906, 1725, 1644, 1446, 1371, 1254, 1153, 1111, 1028, 836, 817, 777, 737; ^{1}H NMR (200 MHz, $CDCl_3$): δ 0.33 (s, 3H, $SiMe_3$), 0.36 (s, 3H, $SiMe_3$), 1.17–1.32 (m, 12H, 4 × OCH_2CH_3), 1.88–1.98 (m, 1H, SiCH), 2.50–2.60 (m, 2H, $C=CHCH_2$), 3.43 (d, 1H, $^3J_{HH}$ = 5.2 Hz, $CH(CO_2Et)_2$), 4.02–4.28 (m, 8H, 4 × OCH_2CH_3), 6.84 (t, 1H, $^3J_{HH}$ = 7.6 Hz, C=CH), 7.33–7.53 (m, 5H, Ph); ^{13}C NMR (50 MHz, $CDCl_3$): δ −3.92, −3.41, 13.85 (2C), 13.99 (2C), 26.12, 28.30, 52.20, 61.08 (2C), 61.24 (2C), 127.71 (2C), 128.68, 129.17, 134.00 (2C), 137.27, 149.05, 163.65, 164.99, 169.10, 169.46; ESIMS: *m/z* (relative intensity) 515 (93, $[M+Na]^+$), 510 (33), 447 (33), 415 (10), 401 (37), 369 (100), 323 (6); HRMS Calc. for $C_{25}H_{36}O_8SiNa$ [M+Na]: 515.2077. Found: 515.2099.

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