

**REACTIONS OF DIAZOCOMPOUNDS WITH CARBONYL COMPOUNDS MEDIATED BY DIORGANYL
TELLURIDE AND CATALYTIC AMOUNT OF CuI COMPOUNDS: CONVERSION OF ALDEHYDES
TO ALKENES VIA HIGHLY STABILIZED AND STABILIZED TELLURONIUM YLIDES***

Zhang-Lin Zhou, Yao-Zeng Huang,* and Li-Lan Shi
Laboratory of Organometallic Chemistry
Shanghai Institute of Organic Chemistry, Academia Sinica,
345 Lingling Lu, Shanghai 200032, China

(Received in China 20 April 1993)

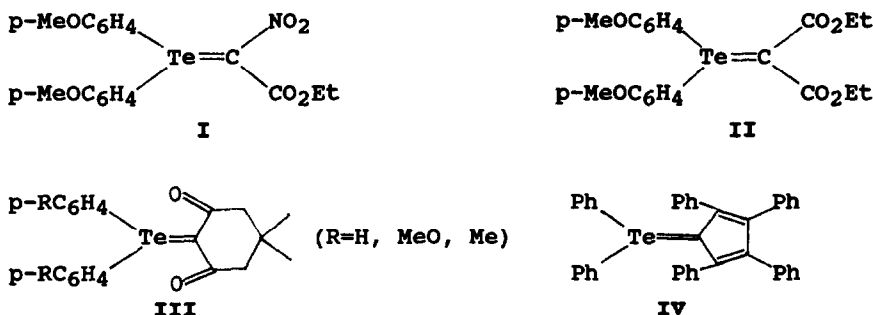
Abstract: A one-pot reaction of diorganyl telluride, diazo-compounds including dimethyl diazomalonate and ethyl diazoacetate, carbonyl compound and catalytic amount of Cu(I)I afforded the olefination products in high yields. The reaction is proposed to be through diorganyltelluronium bis(methoxycarbonyl)methylide and (ethoxycarbonyl)methylide respectively.

Introduction

There have been much interests in other heteroatom ylides for organic synthesis since the birth of the Wittig reaction in 1953.[1] It is well-known that the reaction of the non-stabilized sulfonium and selenonium ylides with carbonyl compounds to produce epoxides in high yields.[2] However, the stabilized sulfonium ylides are inert towards carbonyl compounds except for few extremely reactive carbonyl partners.[3] With the development of sulfonium and selenium ylides, the application of several stabilized and semi-stabilized telluronium ylides in organic synthesis has been described.[4] In our previous papers, we reported that various telluronium salts including precursors of non-stabilized,[5] semi-stabilized[6] and stabilized[7] telluronium ylides, after being treated with alkyl or aryl-lithium reagents, reacted with carbonyl compounds to give secondary alcohols, instead of alkenes or epoxides, the products expected by analogy with the reactions of other heteroatom ylides. However, we found that diphenyltelluronium methylide — a non-stabilized telluronium ylide generated in situ from methyldiphenyltelluronium tetraphenylborate with LiTMP (lithium 2,2,6,6-tetramethylpiperidine) reacted with carbonyl compounds to form substituted oxiranes.[8] Although the synthesis of some highly stabilized phenyl-ligand telluronium ylides with two strong electron-withdrawing substituents such as I, II, III, and IV (Scheme 1) has been described in the literature.[9] However, to the best of our

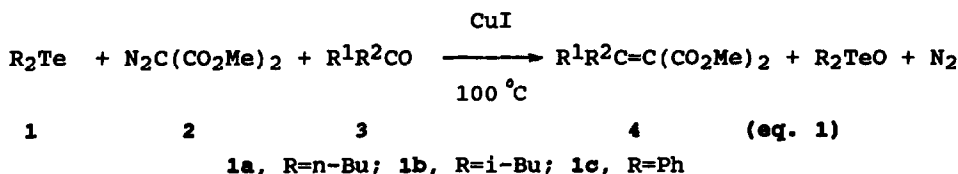
knowledge, no report concerning their reactivities towards carbonyl compounds has appeared. Maybe these telluronium ylides are inert toward carbonyl compounds.

Scheme 1



Results and Discussion

In our continuous studies on the synthetic application of telluronium ylides, we found that dibutyltelluronium bis(methoxycarbonyl)methylide, and diphenyltelluronium bis(methoxycarbonyl)methylide - two highly stabilized telluronium ylides, generated in situ from the reaction of dimethyl diazomalonate with dibutyl telluride and diphenyl telluride in the presence of copper(I) iodide respectively, reacted with carbonyl compound to afford α,β -unsaturated malonic esters in high yields (equation 1).



Thus dibutyl telluride (1.2mmol) was injected into a mixture of the carbonyl compound(1.0mmol), dimethyl diazomalonate(1.2mmol) and copper(I) iodide (0.3mmol) without solvent under nitrogen. The mixture was stirred at 100 °C for several hours. After the reaction was completed (monitored by ^1H NMR), flash chromatography on silica gel gave the pure product in high yield. The results are shown in Table 1(entry 1-5). It is noteworthy that diisobutyl telluride and diphenyl telluride can also mediate the same olefination reaction (entry 6-12).

The reaction is proposed to be through highly stabilized telluronium ylide with two strong electron-withdrawing substituents. In the absence

of organotellurium reagent, olefination did not take place at all. Di-organyl tellurides can react with the carbene from the dimethyl diazomalonate to generate the highly stabilized telluronium ylides. Moreover, these ylides are reactive towards carbonyl compounds due to its more nucleophilicity than corresponding sulfur and selenium counterparts (as shown in Scheme 2). Because the alkyl ligand is an electron-donating group compared with the phenyl ligand, thus the alkyl-ligand telluronium ylide is more reactive than phenyl-ligand one, the yield of the alkene product 4 (entry 1-9) is higher than that of the latter reaction (entry 10-12).

Table 1 Synthesis of the alkene 4 via dimethyl diazomalonate^a

Entry	R ₂ Te(1)	R ¹	R ²	Product 4	Yield(%) ^b
1	1a	C ₆ H ₅	H	4a	91
2	1a	p-ClC ₆ H ₄	H	4b	95
3	1a	p-CH ₃ C ₆ H ₄	H	4c	90
4	1a	p-PhCH=CH	H	4d	80
5	1a	2-thiophenyl	H	4e	93
6	1b	p-CH ₃ OC ₆ H ₄	H	4f	86
7	1b	i-Bu	H	4g	88
8	1b	-(CH ₂) ₅ -		4h	60
9	1c	p-ClC ₆ H ₄	H	4b	67
10	1c	p-NO ₂ C ₆ H ₄	H	4i	70
11	1c	2-Pyridyl	H	4j	54
12	1c	Cyclohexyl	H	4k	55

a All the products were confirmed by ¹H NMR and IR spectra; New compounds gave satisfactory microanalysis as shown in experimental part.

b Isolated yield by flash chromatography based on aldehyde.

Attempts to isolate these highly stabilized telluronium ylides by chromatography failed. However, ¹H NMR shows the exhibition of these highly stabilized telluronium ylides. After heating a mixture of an equivalent of diphenyl telluride, dimethyl diazomalonate and a catalytic amount of copper(I) iodide for three hours, ¹H NMR exhibits at δ = 3.26ppm (Me), corresponding to that of triphenylarsonium bis(methoxycarbonyl) methylide.[10] It is known that, on the ¹H NMR spectra, the ester protons of dimethyl diazomalonate appear at δ = 3.80ppm and those of dimethyl malonate show at δ = 3.65ppm. To the crude mixture, an equivalent of p-chlorobenzaldehyde was added, the olefination product was also produced (50%).

This shows that the ylide may be sensitive to air.

Scheme 2

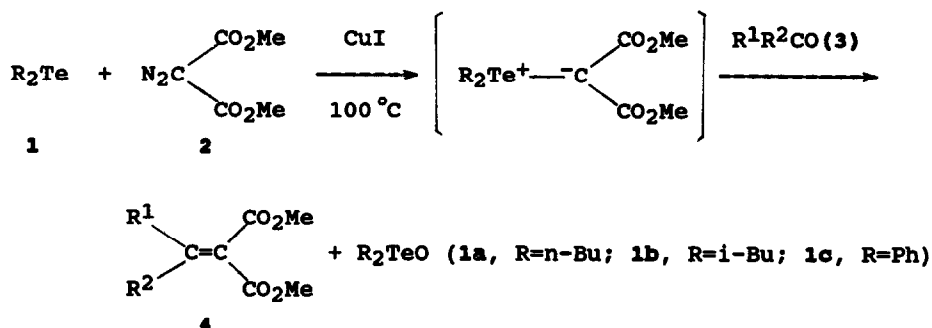


Table 2 Reaction Results from Various Catalysts^a

Entry	Catalyst	Amount (equiv.)	T (°C)/t (hr)	Yield (%) ^b
1	-C	0	110/10	0
2	active Cu ^{0d}	0.3	100/6	68
3	CuSO ₄	0.3	100/6	65
4	CuBr ₂	0.3	100/6	69
5	Cu(OAc) ₂	0.3	100/6	59
6	CuCl ₂	0.3	100/6	68
7	CuCl	0.3	100/5	90
8	Cu ₂ O	0.3	100/5	92
9	CuI	0.3	100/5	95

a. A mixture of n-Bu₂Te(1.2mmol), N₂C(CO₂Me)₂(1.2mmol), p-C₆H₄CHO (1.0mmol) and catalyst(0.3mmol) was heated under N₂ for certain hours.

b. Yields determined by ^1H NMR.

c. No catalyst was used in this entry.

d. Active Cu^0 was prepared in situ from the reaction of Zn powder with CuSO_4 .

Lloyd et al. reported that bis(hexfluoroacetylacetonato)copper(II) was effective for the preparation of diphenyltelluronium ylide.[9b] We found that copper(I) iodide was the most effective catalyst for the preparation of diorganyltelluronium bis(methoxycarbonyl)methylide. This finding is coincident with the reported result that CuI was the most effective catalyst for the decomposition of diazo compounds.[11] The efficiency of various catalysts for the reaction is shown in Table 2.

It is noteworthy that other trapping reagents such as tributylarsine, triphenylarsine, tributylstibine, and triphenylstibine could also mediate the same reaction in our hands. However, tributylphosphine, triphenylphosphine, dimethyl sulfide, and diethyl sulfide could not promote this olefination reaction. (as shown in Table 3)

Table 3 Reaction Results of p-Chlorobenzaldehyde with Dimethyl Diazomalonate Mediated by Various R₃M or R₂M ^a

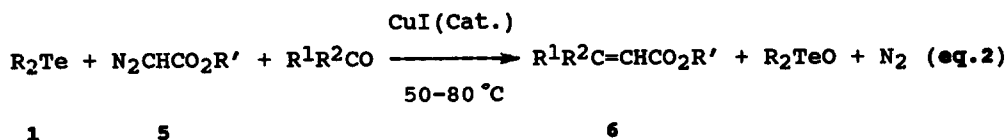
Entry	R ₃ M or R ₂ M	T (°C)/t(h)	Yield(%) ^b
1	-C	110/10	0
2	Bu ₃ As	100/6	93
3	Ph ₃ As	100/6	65
4	Bu ₃ Sb	100/6	95
5	Ph ₃ Sb	100/8	71
6	Bu ₃ P	100/8	0
7	Ph ₃ P	100/8	0
8	Et ₂ S	100/8	0
9	Me ₂ S	100/8	0

a. N₂C(CO₂Me)₂/R₃M or R₂M/p-ClC₆H₄CHO/CuI=1.2:1.2:1.0:0.3

b. Yields determined by ¹H NMR.

c. No R₃M or R₂M was used in this entry.

Similarly, the reaction of ethyl or methyl diazoacetate with carbonyl compounds mediated by diorganyl telluride also resulted in olefination in high yields (equation 2). The results are shown in Table 4. It is noteworthy that, when aldehydes were used as substrates, the reaction gave the olefins in E configuration. The reaction is also proposed to be through the corresponding telluronium ylide (Scheme 3)



1a, R=n-Bu; 1b, R=i-Bu; 1c, R=Ph; R'=Et, Me

The reaction is of wide scope. The aldehydes may be aromatic, alicyclic or heterocyclic. They may contain a double bond. A double bond conjugated with a carbonyl group does not interfere the reaction, the attack being at the carbonyl carbon.

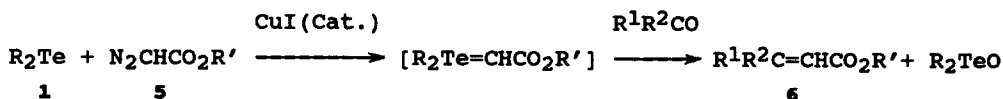
Table 4 Olefination of Carbonyl Compounds with Ethyl or Methyl Diazoacetate Mediated by Diorganyl Telluride^a

Entry	R ₂ Te	R'	R ¹	R ²	Product 6	Yield(%) ^b
1	1a	Et	C ₆ H ₅	H	6a	96
2	1a	Et	p-ClC ₆ H ₄	H	6b	95
3	1a	Et	PhCH=CH	H	6c	95
4	1a	Et	-(CH ₂) ₅ -	H	6d	80
5	1b	Et	C ₆ H ₅	H	6a	92
6	1c	Et	p-ClC ₆ H ₄	H	6b	86
7	1a	Me	C ₆ H ₅	H	6e	85
8	1a	Me	p-MeC ₆ H ₄	H	6f	90
9	1a	Me	2-Pyridyl	H	6g	86
10	1a	Me	Cyclohexyl	H	6h	82
11	1b	Me	C ₆ H ₅	H	6e	90
12	1c	Me	p-ClC ₆ H ₄	H	6i	90

a. All the products were characterized by ¹H NMR, IR and MS.

b. Isolated yields based on carbonyl compounds.

Scheme 3



This one-pot reaction is simple and proceeds without base in high yields. Furthermore, the diazo-compounds used are easily accessible and safe to handle. In this sense, this reaction is the simplified version of normal Wittig reaction which involves three steps: preparation of phosphonium salts, its base treatment to ylide, and reaction of the ylides with carbonyl compounds. It is of particular interest in that diorganyltelluronium ylides bearing two strong electron-withdrawing groups are still reactive towards aldehydes. These results illustrate that diorganyltelluronium ylide is more reactive than the analogues of phosphonium and arsonium ones.

Experimental

Melting points and boiling points were uncorrected. Proton nuclear

magnetic resonance (^1H NMR) spectra were recorded on Varian Model EM-360A or XL-200 spectrometer in CCl_4 solution using TMS as an internal standard other than noted. Infrared (IR) spectra were recorded on an IR-440 grating spectrophotometer. Mass spectral data were obtained with electron ionization (EI) on a Finnigan 4021 spectrometer.

Materials: Dibutyl telluride, [12] diisobutyl telluride, [12] diphenyl telluride, [13] tributylarsine, [14] tributylstibine, [15] triphenylstibine, [16] dimethyl diazomalonate [11] and ethyl or methyl diazoacetate [17] were prepared according to the methods given in the literature methods.

Reactions of dimethyl diazomalonate with aldehydes mediated by diorganyl telluride: General Procedure: Diorganyl telluride (1.2mmol) was injected into a mixture of the carbonyl compound (1.0mmol), dimethyl diazomalonate (1.2mmol) and copper(I) iodide (0.3mmol) without solvent under nitrogen. The mixture was stirred at 100°C for several hours. After the reaction was completed (monitored by ^1H NMR), flash chromatography on silica gel gave the pure product in high yields.

Dimethyl benzylidene malonate (4a): Yield, 91%; b.p. $168\text{--}171^\circ\text{C}/10\text{mmHg}$; [18] ^1H NMR(CCl_4/TMS , 60MHz) δ 3.70(s, 3H), 3.73(s, 3H), 7.29(s, 5H), 7.53(s, 1H); IR(neat) 1730(vs), 1630(s), 1260(s), 1220(s), 1080(s), 830(m), 770(s), 690(s) cm^{-1} .

Dimethyl 4-chlorobenzylidene malonate (4b): Yield, 95%; m.p. $55\text{--}56^\circ\text{C}$; [19] ^1H NMR(CCl_4/TMS , 60MHz) δ 3.77(s, 3H), 3.80(s, 3H), 7.33(s, 4H), 7.60(s, 1H); IR(KCl) 1720(vs), 1630(s), 1260(vs), 1220(s), 1060(s), 840(m), 820(s) cm^{-1} .

Dimethyl 4-methylbenzylidene malonate (4c): Yield, 90%; oil; [20] ^1H NMR(CCl_4/TMS , 60MHz) δ 2.33(s, 3H), 3.77(s, 6H), 7.18(q, 4H), 7.60(s, 1H); IR(neat) 1725(vs), 1630(s), 1260(s), 1220(s), 1085(s), 840(m), 815(s) cm^{-1} .

Dimethyl cinnamylidene malonate (4d): Yield, 80%; m.p. $65\text{--}66^\circ\text{C}$; [21] ^1H NMR(CCl_4/TMS , 60MHz) δ 3.70(s, 3H), 3.76(s, 3H), 6.90–7.60(m, 8H); IR(KCl) 1710(s), 1650(s), 1580(s), 1280(s), 1150(s), 1060(s), 750(s), 690(s) cm^{-1} .

Dimethyl 2-thiophenylmethylidene malonate (4e): Yield, 93%; m.p. $44\text{--}45^\circ\text{C}$; ^1H NMR(CCl_4/TMS , 60MHz) δ 3.77(s, 3H), 3.84(s, 3H), 7.00(dd, $J=4.0, 4.0\text{Hz}$, 1H), 7.31(d, $J=4.0\text{Hz}$, 1H), 7.45(d, $J=4.0\text{Hz}$, 1H), 7.55(d, $J=4.0\text{Hz}$, 1H); EIMS m/z (rel. intensity) 227(M^+1 , 22), 226(M^+ , 43), 195(44), 169(5), 108(22), 83(11), 43(100); IR(KCl) 1730(vs), 1630(s), 1260(s), 1060(s), 860(s), 840(s) cm^{-1} . Anal. Found: C, 52.96; H, 4.45; Calcd for $\text{C}_{10}\text{H}_{10}\text{O}_4\text{S}$, C, 53.09; H, 4.46.

Dimethyl 4-methoxybenzylidene malonate (4f): Yield, 86%; oil; [22] ^1H NMR(CCl_4/TMS , 60MHz) δ 3.72(s, 3H), 3.75(s, 3H), 3.78(s, 3H), 6.83(d, $J=8\text{Hz}$, 2H), 7.34(d, $J=8\text{Hz}$, 2H), 7.58(s, 1H); IR(neat) 1720(vs), 1625(m), 1255(s), 1200(s), 830(s) cm^{-1} .

Dimethyl isopentylidene malonate(4g): Yield, 88%; b.p. 110°C/2mmHg; ^1H NMR(CCl_4/TMS , 60MHz) δ 0.96(d, $J=6\text{Hz}$, 6H), 1.36–1.94(m, 1H), 2.12(dd, $J_1=6\text{Hz}$, $J_2=7\text{Hz}$, 2H), 3.72(s, 6H), 6.85(t, $J=7\text{Hz}$, 1H); EIMS m/z (rel. intensity) 201 (M^++1 , 100), 169(16), 157(6), 99(19); IR(neat) 1730(vs), 1640(s), 1260(s), 1150(s), 1060(s), 840(m) cm^{-1} . Anal. Found: C, 59.95; H, 8.19. Calcd for $\text{C}_{10}\text{H}_{16}\text{O}_4$: C, 59.98; H, 8.05.

Dimethyl cyclohexylidene malonate(4h): Yield, 60%; bp 130°C/5mmHg; ^1H NMR(CCl_4/TMS , 60MHz) δ 1.70(m, 6H), 2.50(m, 4H), 3.70(s, 6H); EIMS m/z (rel. intensity) 213(M^++1 , 4), 181(22), 153(14), 125(100), 97(49); IR(neat) 1730(vs), 1640(m) cm^{-1} . Anal. Found: C, 62.18; H, 7.88; Calcd for $\text{C}_{11}\text{H}_{16}\text{O}_4$, C, 62.25; H, 7.60.

Dimethyl 4-nitrobenzylidene malonate (4i): Yield, 70%; m.p. 136–137°C; [23] ^1H NMR(CDCl_3/TMS , 200MHz) δ 3.67(s, 3H), 3.71(s, 3H), 7.48(d, $J=12\text{Hz}$, 2H), 8.13(d, $J=12\text{Hz}$, 2H), 8.21(s, 1H); IR(KCl) 1720(s), 1700(s), 1625(m), 1340(s), 1200(s), 935(m), 850(m) cm^{-1} .

Dimethyl 2-pyridylmethylidene malonate(4j): Yield, 54%; oil; [20] ^1H NMR(CCl_4/TMS , 60MHz) δ 3.76(m, 6H), 7.23–7.56(m, 4H), 8.55(d, $J=2\text{Hz}$, 1H); IR(film) 1735(vs), 1645(m), 1345(s), 1210(s), 930(m), 845(m) cm^{-1} .

Dimethyl cyclohexylmethylidene malonate(4k): Yield, 55%; b.p. 120–125°C/2mmHg; [24] ^1H NMR(CCl_4/TMS , 60MHz) δ 0.90–1.60(m, 10H), 1.90–2.10(m, 1H), 3.60(s, 3H), 3.65(s, 3H), 6.65(d, $J=10\text{Hz}$, 1H); IR(neat) 1740(s), 1720(s), 1640(m) cm^{-1} .

Reactions of Methyl or Ethyl Diazoacetate with Aldehydes and Diorganyl Telluride: General procedure: Diorganyl telluride(1.2mmol) was injected into a mixture of aldehyde(1.0mmol), methyl or ethyl diazoacetate(1.2mmol) and copper(I) iodide(0.15mmol) under nitrogen. The mixture was stirred at 50°C for several hours. After the reaction was completed (monitored by ^1H NMR), flash chromatography on silica gel gave the pure product.

Ethyl 3-phenyl-(2E)-propenoate(6a): Yield, 96%; b.p. 122°C/5mmHg; [25] ^1H NMR(CCl_4/TMS , 60MHz) δ 1.23(t, $J=7\text{Hz}$, 3H), 4.05(q, $J=7\text{Hz}$, 2H), 6.17(d, $J=16\text{Hz}$, 1H), 7.22(m, 5H), 7.44(d, $J=16\text{Hz}$, 1H); IR(neat) 1710(vs), 1640(s), 1260(s), 1160(s), 860(s), 765(s), 680(s) cm^{-1} .

Ethyl 3-(p-chlorophenyl)-(2E)-propenoate(6b): Yield, 95%; oil; [4a] ^1H NMR(CCl_4/TMS , 60MHz) δ 1.15(t, $J=7\text{Hz}$, 3H), 4.25(q, $J=7\text{Hz}$, 2H), 6.50(d, $J=16\text{Hz}$, 1H), 7.56(m, 5H); IR(neat) 1720(s), 1650(m), 1410(s), 1120(m), 825(s) cm^{-1} .

Ethyl 5-phenyl-(2E,4E)-pentadienoate(6c): Yield, 95%; oil; [26] ^1H NMR(CCl_4/TMS , 60MHz) δ 1.18(t, $J=6\text{Hz}$, 3H), 4.08(q, $J=6\text{Hz}$, 2H), 5.80(d, $J=15\text{Hz}$, 1H), 6.70(m, 2H), 6.97–7.40(m, 6H); IR(neat) 1715(s), 1630(s), 1600(m), 1580(w), 1240(s), 840(m), 760(m) cm^{-1} .

Ethyl cyclohexylideneacetate (6d): Yield, 80%; b.p. 102°C/10mmHg; [27]

^1H NMR(CCl_4/TMS , 60MHz) δ 1.20(t, J=6Hz, 3H), 1.55(brs, 6H), 2.10(brs, 2H), 2.70(m, 2H), 4.00(q, J=6Hz, 2H), 5.40(s, 1H); IR(neat) 2910(s), 2850(s), 1720(s), 1650(s), 1260(s), 1040(s), 850(s) cm^{-1} .

Methyl 3-phenyl-(2E)-propenoate(6e): Yield, 85%; b.p. 113°C/5mmHg; [28] ^1H NMR(CCl_4/TMS , 60MHz) δ 3.65(s, 3H), 6.25(d, J=16Hz, 1H), 7.25(m, 6H); EIMS m/z (rel. intensity) 162(M^+ , 57), 131(100), 103(55), 77(39); IR(neat) 1720(s), 1695(s), 1320(s), 1270(s), 1180(s), 980(s), 760(s) cm^{-1} .

Methyl 3-(p-methylphenyl)-(2E)-propenoate(6f): Yield, 90%; m.p. 54.5-55.5°C; [29] ^1H NMR(CCl_4/TMS , 60MHz) δ 2.37(s, 3H), 3.72(s, 3H), 6.16, 6.43(d, J=16Hz, 1H), 7.05-7.73(m, 5H); EIMS m/z (rel. intensity) 192(M^+ , 77), 161(100), 133(41), 118(13), 89(21); IR(KCl) 1710(vs), 1590(s), 160(s), 960(s) cm^{-1} .

Methyl 3-(2-pyridyl)-(2E)-propenoate(6g): Yield, 86%; [30] ^1H NMR(CCl_4/TMS , 60MHz) δ 3.75(s, 3H), 6.38(d, J=16Hz, 1H), 6.86-7.90(m, 4H), 8.57(d, J=4Hz, 1H); IR(KCl) 1710(vs), 1610(m), 1250(s), 950(s) cm^{-1} .

Methyl 3-cyclohexyl-(2E)-propenoate(6h): Yield, 82%; b.p. 88-90°C/15mmHg; [31] ^1H NMR(CCl_4/TMS , 60MHz) δ 1.16-1.80(m, 11H), 3.68(s, 3H), 5.15, 5.36(d, J=16Hz, 1H), 7.10(m, 1H); IR(KCl) 1710(vs), 1640(m), 1320(m), 1170(m), 1000(s), 820(s) cm^{-1} .

Methyl 3-(p-chlorophenyl)-(2E)-propenoate(6i): Yield, 90%; m.p. 70-72°C; 29 ^1H NMR(CCl_4/TMS , 60MHz) δ 3.65(s, 3H), 6.07, 6.34(d, J=16Hz, 1H), 7.30(m, 5H); EIMS m/z (rel. intensity) 197(M^+ +1, 30), 196(M^+ , 47), 165(100), 137(25), 102(35); IR(KCl) 1720(s), 1650(m), 1405(s), 1125(m), 820(s) cm^{-1} .

Acknowledgment: Financial support from the National Natural Science Foundation of China and Academia Sinica is gratefully acknowledged.

References and notes:

This paper is the 102nd report on the synthetic application of elemento-organic compounds of 15th and 16th groups.

- [1] Wittig, G.; Geissler, G. *Liebigs Ann. Chem.* **1953**, *44*, 580.
- [2] (a) Trost, B.M.; Melvin, L.S., Jr. "Sulfur Ylides", Academia Press, New York, 1975. (b) Dumont, W.; Bayet, P.; Krief, A. *Angew. Chem., Internat. Ed. Engl.* **1974**, *13*, 274.
- [3] (a) Payne, G.B. *J. Org. Chem.* **1968**, *33*, 3517. (b) Trost, B.M.; Arndt, H. C. *J. Org. Chem.* **1973**, *38*, 3140.
- [4] (a) Osuka, A.; Mori, Y.; Shimizu, H.; Suzuki, H. *Tetrahedron Lett.* **1983**, *24*, 2599. (b) Osuka, A.; Suzuki, H. *Tetrahedron Lett.* **1983**, *24*, 5109. (c) Huang, X.; Xie, L.H.; Wu, H. *J. Org. Chem.* **1988**, *53*, 4862. (d) Zhou, Z.L.; Sun, Y.S.; Shi, L.L.; Huang, Y.Z. *J. Chem. Soc., Chem. Commun.* **1990**, 1439. (e) Zhou, Z.L.; Shi, L.L.; Huang, Y.Z. *Tetrahedron Lett.* **1990**, *31*, 7657.

- [5] Shi, L.L.; Zhou, Z.L.; Huang, Y.Z. J. Chem. Soc., Perkin Trans. 1 **1990**, 2847.
- [6] Li, S.W.; Zhou, Z.L.; Huang, Y.Z.; Shi, L.L. J. Chem. Soc., Perkin Trans. 1 **1991**, 1099.
- [7] Zhou, Z.L.; Shi, L.L.; Huang, Y.Z. J. Chem. Soc., Perkin Trans. 1 **1991**, 1931.
- [8] Shi, L.L.; Zhou, Z.L.; Huang, Y.Z. Tetrahedron Lett. **1990**, 31, 4173.
- [9] (a) Freeman, B.H.; Lloyd, D.L.; Singer, M.I.C. Tetrahedron **1972**, 28, 343. (b) Glidewell, C.; Lloyd, D.; Metcalfe, S. Synthesis **1988**, 319. (c) Sadekov, I.D.; Usachev, A.I.; Maksimenko, A.A.; Minkin, V.I. Zh. Obshch. Khim. **1975**, 45, 2563. Chem. Abstr. **1976**, 84, 73780y; (d) Sadekov, I.D.; Usachev, A.I.; Bren, V.A.; Korobov, M.S.; Tseimakh, I.D.; Minkin, V.I.; Zh. Obshch. Khim. **1977**, 47, 2232. Chem. Abstr. **1978**, 88, 50417d. (e) Sadekov, I.D.; Usachev, A.I.; Maksimenko, A.A.; Minkin, A.I. Zh. Obshch. Khim. **1978**, 48, 934. Chem. Abstr. **1978**, 89, 108486q.
- [10] (a) Hood, J.N.C.; Lloyd, D.; Macdonald, W.A.; Shepferd, T.M. Tetrahedron **1982**, 38, 3355. (b) Chen, C.; Huang, Y.Z.; Shen, Y.C.; Liao, Y. Heteroatom Chem. **1990**, 1, 49.
- [11] Peace, B.W.; Carman, F.; Wulfman, D.S. Synthesis **1971**, 658.
- [12] Balfe, M.P.; Chaplin, C.A.; Philips, H. J. Chem. Soc. **1938**, 341.
- [13] Reichel, L.; Krischbaum, E. Ber. **1943**, 11, 1105.
- [14] Dyke, W.J.C.; Jones, W.J. J. Chem. Soc. **1930**, 2426.
- [15] Seifter, J. J. Am. Chem. Soc. **1939**, 61, 530.
- [16] Hiers, G.S. Org. Syn., Coll. Vol. 1, **1956**, 550.
- [17] Popp, F.D.; Catala, A. J. Org. Chem. **1961**, 26, 2738.
- [18] Knoevenagel, E. Chem. Ber. **1898**, 31, 2605.
- [19] Vogel, D.; Rondestvedt, C.S., Jr. J. Am. Chem. Soc. **1950**, 78, 3799.
- [20] Mildenberger, H.; Maier, J.; Sachse, B. Ger. Offen. **1980**, 2836945.
- [21] Liebermann, C. Ber. **1895**, 28, 1438.
- [22] American Cyanamide Co. Neth. Appl. 6503314. Chem. Abstr. **1966**, 64, P14365d.
- [23] Kohler, E.P.; Darling, S.F. J. Am. Chem. Soc. **1930**, 52, 424.
- [24] Verbe, R.; Kimpe, N.D.; Bagck, L.D.; Courtheyn, D.; Schamp, N. Bull. Soc. Chim. Belg. **1977**, 83, 215.
- [25] Buckingham, J. Dictionary of Organic Compounds **1982**, p-01516.
- [26] Auwers, K.V. J. Prakt. Chem. **1923**, 105, 377.
- [27] Auwers, K.V.; Ellinger, P. Liebigs Ann. Chem. **1912**, 387, 235.
- [28] Brunet, J.J. Tetrahedron Lett. **1981**, 22, 103.
- [29] Schiemehz, G.D.; Thobe, J. Chem. Ber. **1966**, 99, 2663.
- [30] Shi, L.L.; Wang, W.B.; Wang, Y.C.; Huang, Y.Z. J. Org. Chem. **1989**, 54, 2028.
- [31] Sahay, S.; Sircer, G. J. Chem. Soc. **1928**, 54.