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$AsPh_3$-catalyzed ylide cyclopropanation for the synthesis of trisubstituted vinylcyclopropane derivatives

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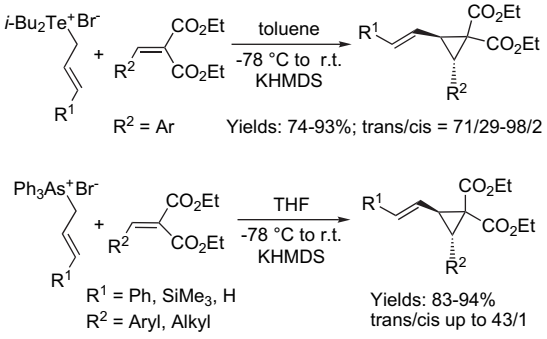
ABSTRACT

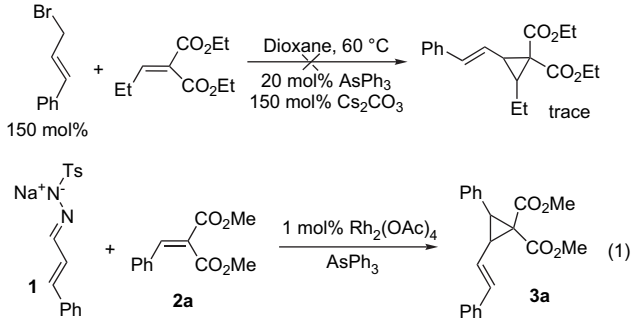
Under mild conditions, the reaction of alkylidene, arylidene, and heteroarylidene malonates with tosylhydrazone salts in the presence of catalytic amount of $Rh_2(OAc)_4$ and triphenylarsine affords *trans*-2,3-disubstituted cyclopropane 1,1-dicarboxylic esters in high yields and high diastereoselectivities.

1. Introduction

Vinylcyclopropane 1,1-dicarboxylic esters are very useful synthetic intermediates in the creation of cyclopentane skeleton and heterocyclic compounds.[1–4] In our study on ylide chemistry,[5] we recently developed a reaction of telluronium ylide with arylidene malonates for the preparation of such compounds.[6] Later on, we extended the substrate scope and found both arylidene malonates and alkylidene malonates were good for this cyclopropanation by employing arsonium ylides (Scheme 1).[7] However, stoichiometric amount of ylides are required in both aforementioned reactions. Attempts to develop the catalytic reaction of ethylidene malonate with cinnamylbromide in the presence of substoichiometric triphenylarsine[8] failed (Scheme 2). In this case, only a trace amount of the desired product was observed and propylidene malonate was



Scheme 1. Ylide cyclopropanation for the synthesis of cyclopropanes.



Scheme 2. Attempts on catalytic ylide cyclopropanation.

decomposed when the reaction time was prolonged probably because the reaction of triphenylarsine with cinnamylbromide was sluggish. Aggarwal et al. developed an elegant strategy to generate ylide by employing tosylhydrazone salt and successfully applied it to ylide reactions.[9] We envisioned that 2-phenylvinyl tosylhydrazone salt can be used for the formation of arsonium cinnamylide in the presence of metal catalyst, which allows to realize a catalytic version of the ylide cyclopropanation as shown in Eq. 1 (Scheme 2). In this paper, we wish to report our efforts on this subject.

2. Result and discussion

Initially, a mixture of tosylhydrazone salt **1** and dimethyl 2-benzylidenemalonate **2a** in dioxane was stirred at 40 °C in the presence of catalytic amounts of $Rh_2(OAc)_4$ (1 mol %), $AsPh_3$ (20 mol %), and benzyltriethylammonium chloride (20 mol %) for 8 h, affording the desired cyclopropane **3a** in 46% yield. Encouraged by this result, we screened several solvents and found that solvents strongly influenced the yield of the desired product. As shown in Table 1, both ether solvents such as THF, dioxane and arene solvents

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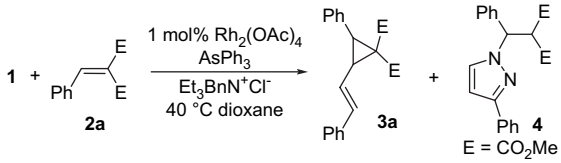
Table 1
Effects of reaction conditions for catalytic cyclopropanation[a]



Entry	Solvent (mL)[b]	Ph_3As (mol %)	PTC (mol %)	Conversion (%, **3a**/**4**)[c]
1	Toluene (6)	20	20	41(14:27)
2	BTF (6)	20	20	55(35:20)
3	THF (6)	20	20	73(30:43)
4	CH_3CN (6)	20	20	68(18:50)
5	DCE (6)	20	20	10(7:3)
6	$CHCl_2CHCl_2$ (6)	20	20	—
7	$CHCl_3$ (6)	20	20	12(12:0)
8	Dioxane (6)	20	20	77(46:31)
9	Dioxane (1)	20	20	72(60:12)
10[d]	Dioxane (1)	—	20	40(0:40)
11	Dioxane (1)	100	20	100(100:0)
12	Dioxane (1)	50	20	100(100:0)
13	Dioxane (1)	20	10	78(74:4)
14	Dioxane (0.8)	20	10	80(78:2)
15[e]	Dioxane (0.5)	20	10	100(100:0)

[a] Reactions were performed at 40 °C in dioxane with tosylhydrazone salt **1** (150 mg, 0.45 mmol).
[b] Volume of solvent.
[c] Conversion based on **2a** by ^1H NMR.
[d] In the absence of $Rh_2(OAc)_4$ and stirring for 24 h.
[e] Isolated yield was 86%.

gave good conversions (entries 1–3 and 8). In halogenated solvents, this reaction did not proceed well (entries 5–7). The optimal one was dioxane (Table 1, entries 1–8). In all cases screened, byproduct **4** was isolated and was confirmed by X-ray crystallographic analysis (Fig. 1). It is envisaged that **4** was formed by a rearrangement of cinnamyl diazo compound generated from tosylhydrazone salt **1**, followed by a Michael addition to dimethyl 2-benzylidenemalonate (Scheme 3).[10] Further study showed that increasing the loading of $AsPh_3$ from 20 to 50 mol % or to 100 mol % led to a complete conversion of **2a** into the desired cyclopropanation product **3a** and byproduct **4** was not formed (entries 9, 11, and 12).

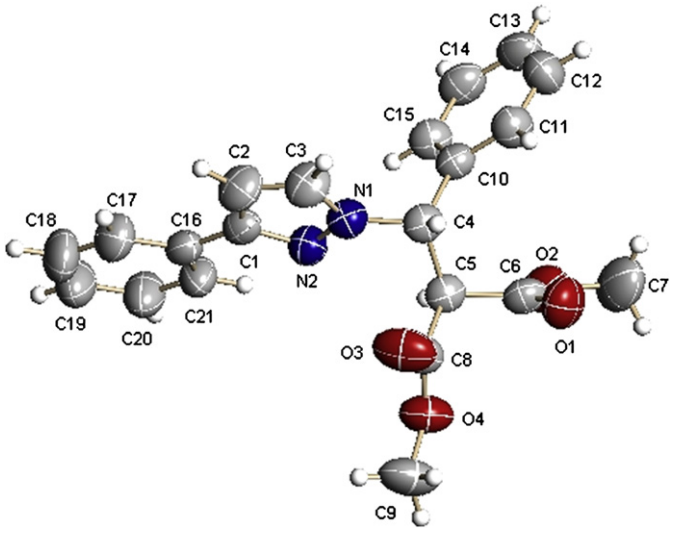
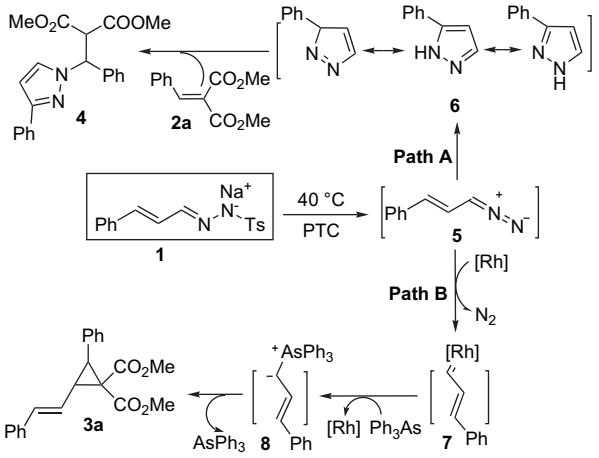


Figure 1. Molecular structure of compound **4**.

In the absence of triphenylarsine, no desired product was produced. This observation is consistent with the mechanism as shown in Scheme 3, since the arrangement of diazo compound **5** to **6** is a competing reaction with the generation of metal carbene **7**.

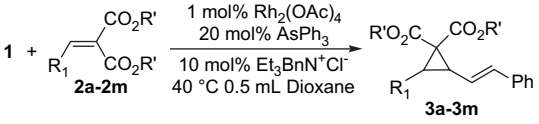


Scheme 3. A possible mechanism for the formation of byproduct **4**.

According to this mechanistic insight and the aforementioned observation, we envisioned that the loading reduction of the phase-transfer catalyst and increasing the concentration of the reactants might inhibit partially the formation of compound **4** and improve the yield of the desired cyclopropane. As shown in Table 1, decreasing the amount of PTC from 20 to 10 mol % reduced the byproduct yield from 12% to 4% (Table 1, entry 13). Under this condition, increasing the concentration of reactants and As_3Ph by reduction of dioxane from 1.0 to 0.8 mL and furthermore to 0.5 mL resulted in a clean reaction (Table 1, entries 14 and 15). In the case of 0.5 mL of dioxane used, cyclopropane **3a** could be obtained in 86% isolated yield and no byproduct **4** was isolated.

Under the optimized conditions, the scope and limitation of the current cyclopropanation was investigated by employing various alkylidene, arylidene, and heteroarylidene malonate derivatives as substrates. The results were summarized in Table 2. The ester groups of the Michael acceptors had almost no effects on the diastereoselectivity. For example, both methyl and ethyl benzylidenemalonates gave the corresponding vinylcyclopropanes with the same diastereoselectivity (entries 1 and 2, Table 2). In the case of arylidene malonates, both electron-donating and electron-withdrawing substituents on the benzene ring were well tolerated and

Table 2
Catalytic cyclopropanation of alkylidene and arylidene malonates[a]



Entry	Substrate	R_1	R′	**3** (yield, %)[b]	(trans/cis)[c]
1	**2a**	Ph	Me	**3a** (86)	88:12
2	**2b**	Ph	Et	**3b** (79)	88:12
3	**2c**	*p*-Br-C_6H_4	Et	**3c** (91)	89:11
4	**2d**	*o*-Br-C_6H_4	Et	**3d** (86)	86:14
5	**2e**	2,4-Cl_2-C_6H_3	Me	**3e** (78)	89:11
6	**2f**	*p*-NO_2-C_6H_4	Me	**3f** (87)	89:11
7	**2g**	*p*-Me-C_6H_4	Me	**3g** (98)	88:12
8	**2h**	1-Naphthyl	Et	**3h** (85)	92:8
9	**2i**	2-Furyl	Me	**3i** (90)	88:11
10	**2j**	2-Pyridyl	Me	**3j** (89)	95:5
11	**2k**	Et	Et	**3k** (42)	90:10
12	**2l**	*i*-Pr	Et	**3l** (63)	75:25
13	**2m**	*i*-Bu	Me	**3m** (76)	84:16

[a] Ph_3As (18 mg, 0.06 mmol), **2a**–**2m** (0.30 mmol), dioxane (0.5 mL), $Rh_2(OAc)_4$ (1.5 mg, 0.003 mmol), benzyltriethylammonium chloride (7 mg, 0.03 mmol), and tosylhydrazone salt **1** (145 mg, 0.45 mmol).
[b] Isolated yield.
[c] Determined by ^1H NMR.

all gave the desired products in high yields with good selectivities (entries 3–7), suggesting that the electronic effects only influenced slightly the reaction. Heteroarylidene malonates **2i** and **2j** were also examined and high yields were obtained with high diastereoselectivities (entries 9 and 10). Alkylidene malonates gave moderate to good diastereoselectivities but the yields strongly depended on structure of the alkyl group (entries 11–13). For example, propylene malonate only afforded the desired product in 42% yield. However, *iso*-butylene and *iso*-pentylene malonates were used, the yields were increased to 63% and 76%, respectively (entries 12 and 13). Thus, this method provided a convenient way to the synthesis of vinylcyclopropane 1,1-dicarboxylic esters.

3. Conclusion

In summary, a mild catalytic ylide cyclopropanation for the preparation of vinylcyclopropane 1,1-dicarboxylic esters from arylidene and alkylidene malonates has been developed. Compared with our previous report, the loading of $AsPh_3$ was reduced from stoichiometric amount to 20 mol % for the first time. The readily available starting material, good selectivities, and high yields make this protocol potentially useful in organic synthesis. Current investigations are focused upon asymmetric variants of this reaction, and results will be reported in due course.

4. Experimental

4.1. General procedure for the catalytic cyclopropanation (2a as a substrate)

A mixture of triphenylarsine (18 mg, 0.06 mmol), rhodium(II) acetate dimer (1.5 mg, 0.003 mmol), benzyltriethylammonium chloride (7 mg, 0.03 mmol), dimethyl 2-benzylidenemalonate **2a** (66 mg, 0.30 mmol), and tosylhydrazone sodium salt **1** (145 mg, 0.45 mmol) in anhydrous dioxane (0.5 mL) was stirred vigorously at room temperature for 10 min, then heated at 40 °C for 8–24 h (turn to orange). After the reaction was complete, it was quenched by addition of ethyl acetate (10 mL) and filtered rapidly through a glass funnel with a thin layer of silica gel (ethyl acetate as a eluent, 50 mL). The filtrate was concentrated and the residue was subjected to ^{1}H NMR analysis to determine the ratio of isomers. The pure product **3a** was obtained by flash chromatography on silica gel.

4.2. Dimethyl 2-(phenyl(3-phenyl-1*H*-pyrazol-1-yl)methyl) malonate (4)

Mp (hexane) 99–101 °C; ^{1}H NMR (300 MHz, $CDCl_3$): 7.84–7.80 (m, 2H), 7.51–7.25 (m, 9H), 6.50 (d, *J*=2.4 Hz, 1H), 5.93 (d, *J*=11.1 Hz, 1H), 4.91 (d, *J*=11.1 Hz, 1H), 3.66 (s, 3H), 3.51 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): 167.1, 166.8, 151.1, 137.0, 133.5, 131.0, 128.7, 128.6, 128.5, 127.7, 127.5, 125.5, 103.0, 64.3, 57.2, 52.9, 52.7; MS (EI) *m*/*z* (rel intensity) 364 (M^+, 4), 57 (100), 43 (76), 71 (66), 85 (48), 55 (48), 41 (45), 69 (44), 83 (27); IR (thin film)/cm^{-1}: 2953 (m), 1756 (s), 1741 (s), 1500 (m), 1457 (m), 1435 (m), 1317 (m), 1265 (m), 753 (m), 696 (m). Anal. Calcd for $C_{21}H_{20}N_2O_4$: C, 69.22; H, 5.53; N, 7.69. Found: C, 69.11; H, 5.51; N, 7.49.

4.3. Dimethyl *trans*-2-phenyl-3-styrylclopropane-1,1-dicarboxylate (3a)[7]

^{1}H NMR (300 MHz, $CDCl_3$): 7.40–7.18 (m, 10H), 6.79 (d, *J*=15.9 Hz, 1H), 6.03 (dd, *J*=15.6, 8.4 Hz, 1H), 3.78 (s, 3H), 3.50 (d, *J*=8.4 Hz, 1H), 3.44 (s, 3H), 3.34 (t, *J*=8.4 Hz, 1H).

4.4. Diethyl *trans*-2-phenyl-3-styrylcyclopropane-1,1-dicarboxylate (3b)[6]

^{1}H NMR (300 MHz, $CDCl_3$): 7.40–7.19 (m, 10H), 6.78 (d, *J*=15.9 Hz, 1H), 6.04 (dd, *J*=15.9, 8.7 Hz, 1H), 4.30–4.16 (m, 2H), 4.00–3.82 (m, 2H), 3.49 (d, *J*=7.8 Hz, 1H), 3.32 (t, *J*=9.0 Hz, 1H), 1.24 (t, *J*=7.2 Hz, 3H), 0.92 (t, *J*=7.2 Hz, 3H).

4.5. Diethyl *trans*-2-(4-bromophenyl)-3-styrylcyclopropane-1,1-dicarboxylate (3c)[7]

Mp 71–72 °C; ^{1}H NMR (300 MHz, $CDCl_3$): 7.41 (d, *J*=8.7 Hz, 2H), 7.34–7.25 (m, 5H), 7.15 (d, *J*=8.7 Hz, 2H), 6.79 (d, *J*=15.9 Hz, 1H), 6.01 (dd, *J*=15.9, 8.7 Hz, 1H), 4.36–4.10 (m, 2H), 4.04–3.84 (m, 2H), 3.42 (d, *J*=8.1 Hz, 1H), 3.28 (t, *J*=8.1 Hz, 1H), 1.25 (t, *J*=7.2 Hz, 3H), 1.00 (t, *J*=6.9 Hz, 3H).

4.6. Diethyl *trans*-2-(2-bromophenyl)-3-styrylcyclopropane-1,1-dicarboxylate (3d)

^{1}H NMR (300 MHz, $CDCl_3$): 7.55 (d, *J*=7.5 Hz, 1H), 7.37–7.09 (m, 8H), 6.80 (d, *J*=16.2 Hz, 1H), 6.05 (dd, *J*=15.6, 8.7 Hz, 1H), 4.33–4.20 (m, 2H), 3.97–3.89 (m, 2H), 3.57 (d, *J*=8.1 Hz, 1H), 3.37 (t, *J*=8.7 Hz, 1H), 1.25 (t, *J*=7.2 Hz, 3H), 0.94 (t, *J*=7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): 207.4, 166.9, 166.3, 136.5, 134.2, 132.5, 129.8, 128.9, 128.5, 127.6, 126.9, 126.3, 126.1, 123.6, 61.7, 61.4, 44.0, 37.8, 33.9, 14.2, 13.7; IR (thin film)/cm^{-1}: 2980 (m), 1724 (s), 1369 (m), 1288 (m), 1210 (m), 1097 (m), 1026 (m), 963 (m), 749 (m), 693 (m); HRMS (ESI) calcd for $C_{23}H_{23}O_4BrNa$ $(M+Na)^+$ 465.0672; found 465.0672.

4.7. Dimethyl *trans*-2-(2,4-dichlorophenyl)-3-styrylcyclopropane-1,1-dicarboxylate (3e)

^{1}H NMR (300 MHz, $CDCl_3$): 7.39–7.13 (m, 8H), 6.80 (d, *J*=15.9 Hz, 1H), 5.99 (dd, *J*=15.9, 8.4 Hz, 1H), 3.80 (s, 3H), 3.54 (d, *J*=8.7 Hz, 4H), 3.52 (s, 3H), 3.32 (dd, *J*=16.5, 8.7 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): 167.3, 166.7, 136.5, 136.3, 134.6, 133.9, 131.1, 130.4, 129.1, 128.6, 127.8, 126.7, 126.2, 123.0, 52.9, 52.6, 43.6, 35.1, 34.2; IR (thin film)/cm^{-1}: 2947 (m), 1731 (s), 1436 (m), 1294 (m), 1221 (m), 1098 (m), 753 (m), 694 (m), 563 (m); HRMS (ESI) calcd for $C_{21}H_{18}O_4Cl_2Na$ $(M+Na)^+$ 427.0479; found 427.0474.

4.8. Dimethyl *trans*-2-(4-nitrophenyl)-3-styrylcyclopropane-1,1-dicarboxylate (3f)

^{1}H NMR (300 MHz, $CDCl_3$): 8.16 (d, *J*=8.4 Hz, 2H), 7.45–7.24 (m, 7H), 6.82 (d, *J*=16.5 Hz, 1H), 6.00 (dd, *J*=15.9, 8.7 Hz, 1H), 3.80 (s, 3H), 3.55 (d, *J*=8.1 Hz, 1H), 3.49 (s, 3H), 3.37 (t, *J*=8.4 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): 167.2, 166.2, 147.1, 142.1, 136.2, 134.8, 129.4, 128.6, 127.9, 126.2, 123.4, 122.6, 53.1, 52.8, 44.7, 36.1, 34.2; IR (thin film)/cm^{-1}: 2949 (m), 1731 (s), 1522 (m), 1347 (m), 1293 (m), 858 (m), 740 (m), 694 (m); HRMS (ESI) calcd for $C_{21}H_{19}NO_6Na$ $(M+Na)^+$ 404.1119; found 404.1105.

4.9. Dimethyl *trans*-2-styryl-3-*p*-tolylcyclopropane-1,1-dicarboxylate (3g)[7]

Mp 84–85 °C; ^{1}H NMR (300 MHz, $CDCl_3$): 7.39–7.19 (m, 5H), 7.13 (d, *J*=9.0 Hz, 2H), 7.08 (d, *J*=9.0 Hz, 2H), 6.78 (d, *J*=15.9 Hz, 1H), 6.02 (dd, *J*=15.9, 9.0 Hz, 1H), 3.77 (s, 3H), 3.49–3.42 (m, 4H), 3.33 (t, *J*=8.1 Hz, 1H), 2.31 (s, 3H).

4.10. Diethyl *trans*-2-(naphthalen-1-yl)-3-styryl-cyclopropane-1,1-dicarboxylate (3h)

^{1}H NMR (300 MHz, $CDCl_3$): 8.27 (d, *J*=8.1 Hz, 1H), 7.84–7.74 (m, 2H), 7.60–7.20 (m, 9H), 6.87 (d, *J*=16.2 Hz, 1H), 6.11 (dd, *J*=16.2,

8.7 Hz, 1H), 4.37–4.28 (m, 2H), 3.88 (d, J=8.1 Hz, 1H), 3.67–3.58 (m, 2H), 3.53 (t, J=8.4 Hz, 1H), 1.27 (t, J=7.2 Hz, 3H), 0.47 (t, J=7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): 167.7, 166.3, 136.6, 134.0, 133.3, 132.8, 130.6, 128.6, 128.2, 128.1, 127.6, 126.1, 126.0, 125.8, 125.0, 124.5, 124.1, 61.8, 61.0, 44.2, 34.9, 33.3, 14.2, 13.2; IR (thin film)/cm^{-1}: 2976 (m), 1721 (s), 1289 (m), 1198 (m), 793 (m), 776 (m), 752 (m), 735 (m), 689 (m), 694 (m), 640 (m); HRMS (ESI) calcd for $C_{27}H_{26}O_4Na$ $(M+Na)^+$ 437.1712; found 437.1723.

4.11. Dimethyl *trans*-2-(furan-2-yl)-3-styrylcyclopropane-1,1-dicarboxylate (3i)

^{1}H NMR (300 MHz, $CDCl_3$): 7.36–7.22 (m, 6H), 6.77 (d, J=15.6 Hz, 1H), 6.30 (dd, J=3.3, 2.1 Hz, 1H), 6.19 (d, J=3.3 Hz, 1H), 6.02 (dd, J=15.9, 8.7 Hz, 1H), 3.77 (s, 3H), 3.60 (s, 3H), 3.38 (d, J=7.8 Hz, 1H), 3.25 (t, J=8.1 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): 167.3, 166.7, 148.8, 142.1, 136.5, 134.5, 128.5, 127.7, 126.2, 122.8, 110.5, 107.8, 53.0, 52.8, 43.4, 33.9, 30.0; IR (thin film)/cm^{-1}: 2967 (m), 1732 (s), 1436 (m), 1289 (m), 1231 (m), 793 (m), 752 (m), 735 (m); HRMS (ESI) calcd for $C_{19}H_{18}O_5Na$ $(M+Na)^+$ 349.1042; found 349.1047.

4.12. Dimethyl *trans*-2-(pyridin-2-yl)-3-styrylcyclopropane-1,1-dicarboxylate (3j)

^{1}H NMR (300 MHz, $CDCl_3$): 8.46 (d, J=4.5 Hz, 1H), 7.64–7.56 (m, 1H), 7.37–7.09 (m, 7H), 6.80 (d, J=15.9 Hz, 1H), 6.17 (dd, J=16.2, 9.3 Hz, 1H), 3.77 (s, 3H), 3.57 (s, 3H), 3.50 (dd, J=16.2, 9 Hz, 1H), 3.41 (d, J=7.2 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): 167.8, 167.0, 154.9, 149.0, 136.7, 136.2, 134.2, 128.5, 127.5, 126.1, 123.9, 123.6, 121.9, 52.9, 52.5, 44.6, 37.9, 35.8; IR (thin film)/cm^{-1}: 2940 (m), 1732 (s), 1591 (m), 1435 (m), 1280 (m), 1231 (m), 1206 (m), 1111 (m), 755 (m), 694 (m), 563 (m); HRMS (ESI) calcd for $C_{20}H_{20}NO_4$ $(M+H)^+$ 338.1395; found 338.1387.

4.13. Diethyl *trans*-2-ethyl-3-styrylcyclopropane-1,1-dicarboxylate (3k)[7]

^{1}H NMR (300 MHz, $CDCl_3$): 7.32–7.19 (m, 5H), 6.63 (d, J=15.9 Hz, 1H), 5.89 (dd, J=15.9, 9.0 Hz, 1H), 4.29–4.10 (m, 4H), 2.58 (t, J=8.1 Hz, 1H), 2.13 (q, J=7.8 Hz, 1H), 1.51–1.40 (m, 2H), 1.28 (t, J=7.2 Hz, 3H), 1.21 (t, J=7.2 Hz, 3H), 1.00 (t, J=7.2 Hz, 3H).

4.14. Diethyl *trans*-2-isopropyl-3-styrylcyclopropane-1,1-dicarboxylate(3l)[7]

^{1}H NMR (300 MHz, $CDCl_3$): 7.34–7.14 (m, 5H), 6.63 (d, J=15.9 Hz, 1H), 5.86 (dd, J=15.9, 9 Hz, 1H), 4.34–4.06 (m, 4H), 2.62 (t, J=7.8 Hz, 1H), 1.95 (dd, J=10.5, 7.8 Hz, 1H), 1.40–1.18 (m, 7H), 1.05 (d, J=6.6 Hz, 3H), 0.98 (d, J=6.9 Hz, 3H).

4.15. Dimethyl *trans*-2-isobutyl-3-styrylcyclopropane-1,1-dicarboxylate (3m)[7]

^{1}H NMR (300 MHz, $CDCl_3$): 7.40–7.08 (m, 5H), 6.63 (d, J=15.9 Hz, 1H), 5.86 (dd, J=15.9, 9.0 Hz, 1H), 3.76 (s, 3H), 3.72 (s, 3H), 2.60 (t, J=8.1 Hz, 1H), 2.22–2.12 (m, 1H), 1.78–1.62 (m, 1H), 1.58–1.40 (m, 1H), 1.20–1.06 (m, 1H), 0.94 (t, J=6.9 Hz, 6H).

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Supplementary data

General synthetic procedures, characterization, and spectral data for new compounds, CIF for compound **4**. Supplementary data associated with this article can be found in the online version, at doi:10.1016/j.tet.2008.03.076.

References and notes

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