JOC The Journal of Organic Chemistry

Article

pubs.acs.org/joc

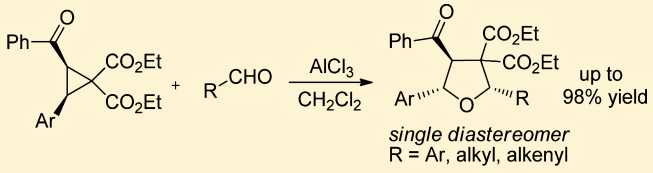
cis-2,3-Disubstituted Cyclopropane 1,1-Diesters in [3 + 2] Annulations with Aldehydes: Highly Diastereoselective Construction of Densely Substituted Tetrahydrofurans

Gaosheng Yang,* Yongxian Sun, Yue Shen, Zhuo Chai,* Shuangliu Zhou, Jiang Chu, and Jun Chai

Key Laboratory of Functionalized Molecular Solids, Ministry of Education, Anhui Key Laboratory of Molecule-Based Materials, Institute of Organic Chemistry, College of Chemistry and Materials Science, Anhui Normal University, Wuhu, Anhui 241000, China

Supporting Information

ABSTRACT: A series of *cis*-2,3-disubstituted cyclopropane 1,1-diesters were examined in the $AlCl_3$-promoted [3 + 2]-annulations with aldehydes. In this reaction, these *cis*-cyclopropanes displayed reactivities starkly different from their *trans* counterparts in terms of the high chemical yields (up to 98%) and provided the desired annulation products with excellent diastereomeric purity. This protocol provides a facile and highly stereoselective way to construct synthetically useful pentasubstituted tetrahydrofurans not easily accessible using other methods.



INTRODUCTION

Known as donor−acceptor (D−A) cyclopropanes, cyclopropane 1,1-diesters have been extensively utilized as three-carbon synthons in a number of annulations, providing various heterocyclic compounds.[1−3] In particular, the elegant work from Johnson's group on the annulations of these cyclopropanes with aldehydes demonstrated the simplicity and high efficiency of this type of chemistry in the stereoselective construction of valuable densely substituted tetrahydrofurans.[2a−g] In addition to aldehydes, many other substrates such as ketones, imines, and nitrones have also been successfully used as reaction partner to provide various useful heterocyclic compounds.[3]

Despite extensive studies on the use of diverse reaction partners, the variation on the structures of cyclopropane 1,1-diesters themselves has been less studied as compared to aldehydes. Inspired by Johnson's works, our group has examined the reactivity of 2,3-disubstituted cyclopropane 1,1-diesters *trans*-**1a** in this type of reaction (Scheme 1).[4] Compared to the commonly used 2-substituted cyclopropane 1,1-diesters, we found that *trans*-**1a** displayed a unique product-forming dependence on the substrate aldehydes and reaction temperature. In addition, the use of this type of cyclopropanes allowed for the stereoselective synthesis of otherwise difficult to obtain pentasubstituted tetrahydrofurans. Intrigued by these results, we became interested in the utilization of *cis*-**1a**[5] in this type of annulations. We assumed that the *cis*-**1a** was less stable thermodynamically and might be more reactive in the annulation process to give higher yields than the *trans*-**1a**. Furthermore, it would be more interesting to compare the stereochemical results obtained with the *cis*-**1a** and *trans*-**1a**, which might be instrumental in the understanding of the mechanism of the annulation process. Herein, we described our investigation on the reactivity of *cis*-**1a** in the annulation with aldehydes.

RESULTS AND DISCUSSION

Our study commenced with the model reaction between *cis*-**1a** and benzaldehyde **2a** (Table 1). To our surprise, under the promotion of $AlCl_3$, the best conditions are identical for *trans*-**1a** in the otherwise same transformation (Table 1, entry 1).[4] Notably, the stereochemical results obtained with *cis*-**1a** were compelling: only one stereoisomer of the desired annulation product **5aa** was obtained. This is in sharp contrast to the results with *trans*-**1a**, in which three stereoisomers were usually present and the major products were **3** or **4** (Scheme 1). In addition, besides the known open-chained byproduct **6a**, a new byproduct **7a** was also identified in this reaction. Reducing the amount of the aldehyde **2a**, varying the loading amount of $AlCl_3$ or raising the reaction temperature all led to inferior results (Table 1, entries 2−8). Moreover, several other Lewis acids were also tested for their efficiency as promoter in this reaction and similar results were generally obtained compared to those obtained with *trans*-**1a** (Table 1, entries 9−13): while the use of $Sn(OTf)_2$ and $Cu(OTf)_2$ could lead to better product ratios with significantly decreased yields, the use of $Mg(OTf)_2$ or $Yb(OTf)_3$ led to no reaction. To summarize, the optimal reaction conditions were selected as 5 equiv of aldehydes, 50 mol % of $AlCl_3$ in CH_2Cl_2 at 0 °C.

Subsequently, the scope of this reaction with regard to different aldehydes was examined with *cis*-**1a** under the optimal reaction conditions (Table 2). For most substituted benzaldehydes, the desired products **5** were obtained in good to

Received: March 16, 2013
Published: May 1, 2013

   dx.doi.org/10.1021/jo400554a | J. Org. Chem. 2013, 78, 5393−5400

Scheme 1. Diastereoselective Annulations of 2,3-Disubstituted Cyclopropane 1,1-Diesters with Aromatic Aldehydes Promoted by $AlCl_3$

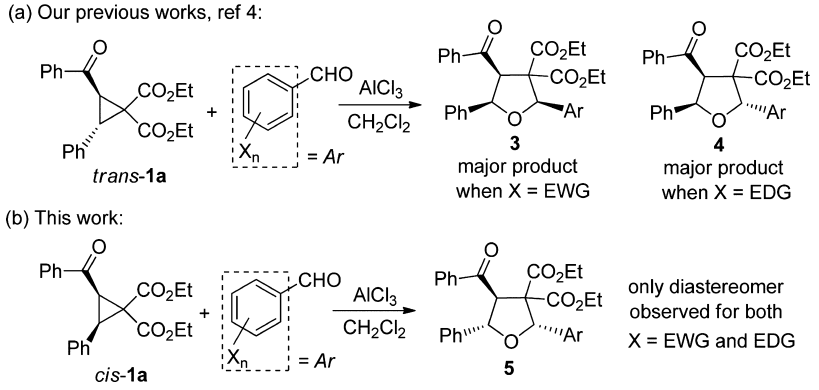
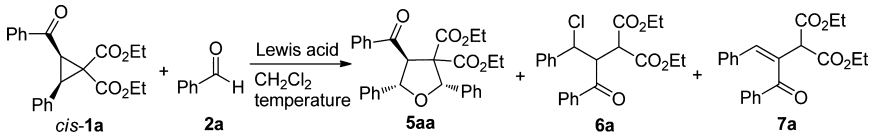


Table 1. Optimization of Reaction Conditions for the Annulation of *cis*-1a and Benzaldehyde[a]



entry	**2a** (equiv)	Lewis acid (equiv)	temp (°C)	time (h)	product/yield[b] (%)	product ratio[c] **5aa**:**6a**:**7a**
1	5.0	$AlCl_3$/0.5	0	19	**5aa**/91	94:4:2
2	5.0	$AlCl_3$/0.3	0	40	**5aa**/87	99:0:1
3	3.0	$AlCl_3$/0.5	0	42	**5aa**/75	99:0:1
4	5.0	$AlCl_3$/1.0	0	6	**5aa**/84	95:0:5
5	1.2	$AlCl_3$/1.0	0	7	**5aa**/67 **7a**/20	76:0:24
6	5.0	$AlCl_3$/0.5	0	6	**5aa**/72	92:8:0
7	5.0	$AlCl_3$/0.5	30	2	**5aa**/77	90:5:5
8	5.0	$AlCl_3$/0.5	30	15	**5aa**/88	90:5:5
9	5.0	$Sn(OTf)_2$/0.5	30	27	**5aa**/40	100:0:0
10	5.0	$Cu(OTf)_2$/0.5	30	27	**5aa**/7	100:0:0
11	5.0	$Mg(OTf)_2$/0.5	30	27	NR	
12	5.0	$Al(OTf)_3$/0.5	30	27	**5aa**/19	100:0:0
13	5.0	$Yb(OTf)_3$/0.5	30	27	NR	

[a]The reaction was conducted with 0.3 mmol of *cis*-**1a**. [b]Isolated yield of the pure product. [c]Determined by ^1H NMR analysis.

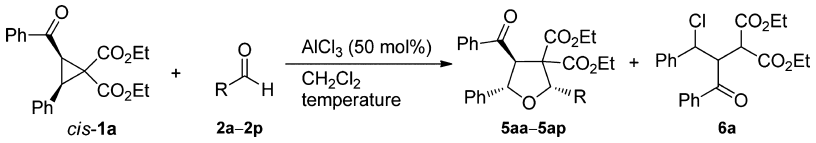
excellent yields as a single diastereomer, regardless of the electron nature or positions of the substituents on the benzene ring. Notably, in most cases, the chemical yields of this type of annulation products were significantly higher than those of the reactions of *trans*-**1a**.[4] Aldehydes **2c** and **2e** bearing strongly electron-withdrawing substituents were not suitable for the reaction, giving the open-chained chlorinated byproduct **6a** as the major product (Table 2, entries 3 and 5). Similar to previous observations, aldehydes having strongly electron-donating substituents required a higher reaction temperature (Table 2, entries 8−11 and 14), probably due to the competitive coordination of $AlCl_3$ between these aldehydes and the D−A cyclopropanes.[4] In addition, the α,β-unsaturated cinnamaldehyde **2o** participated in the reaction as well, providing the desired product in excellent yield (Table 2, entry 15). Moreover, alkyl aldehyde **2p** also underwent the reaction smoothly, albeit with a longer reaction time and a moderate yield (Table 2, entry 16). The relative configurations of product **5** were determined by NOESY analysis and the structure of **5ac** was further confirmed by X-ray crystallographic analysis.[6]

The scope of this annulation was further examined by varying the substituents on the Ar^1 group in the cyclopropanes *cis*-**1** (Table 3). The use of these *cis* cyclopropanes demonstrated a great advantage over their *trans* counterparts in terms of the chemical yields of the desired annulation products. Except for an extreme case, where both Ar^1 (*cis*-**1c**) and Ar^2 (aldehyde **2b**) are electron-deficient (Table 3, entry 6), the desired pentasubstituted tetrahydrofurans **5** were obtained in good to excellent yields as single diastereomers. It is worth mentioning that while reactions of *cis*-**1e** proceeded well to deliver the desired annulation products (Table 3, entries 13−16), the reactions of *trans*-**1e** have been found to give a γ-lactone byproduct, which was formed by an intramolecular attack of one of the oxygen atom in the ester group.[7]

The stereochemical outcome of this reaction could be explained by a mechanistic model similar to that originally proposed by Johnson and co-workers[2d] (Scheme 2). The catalyst $AlCl_3$ activated cyclopropane **1a** via coordination with the diester groups to form an intermediate (**I**). The ensuing nucleophilic attack of the aldehydes **2** would form a zwitterion (**II**). After a bond rotation, an intramolecular nucleophilic attack of the carbanion would close the ring to form the desired

dx.doi.org/10.1021/jo400554a | J. Org. Chem. 2013, 78, 5393−5400

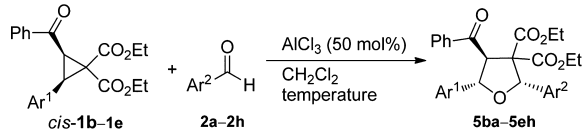
Table 2. Aldehyde Scope in $AlCl_3$-Promoted Annulation of *cis*-1a.[a]



entry	R	temp (°C)	time (h)	product/yield[b] (%)
1	C_6H_5 (**2a**)	0	19	**5aa**/91
2	4-ClC_6H_4 (**2b**)	0	20	**5ab**/87
3	2,4-$Cl_2C_6H_3$ (**2c**)	0	20	**5ac**/25
				6a/51
4	4-BrC_6H_4 (**2d**)	0	22	**5ad**/81
5	4-$NO_2C_6H_4$ (**2e**)	0	38	**6a**/71
6	4-MeC_6H_4 (**2f**)	0	18	**5af**/91
7	4-$MeOC_6H_4$ (**2g**)	30	21	**5ag**/93
8	3,4-$(MeO)_2C_6H_3$ (**2h**)	30	19	**5ah**/91
9	4-$BnOC_6H_4$ (**2i**)	30	21	**5ai**/90
10	3-MeO-4-$BnOC_6H_3$ (**2j**)	30	20	**5aj**/91
11	3-MeO-4-$AcOC_6H_3$ (**2k**)	30	20	**5ak**/85
12	4-$TsOC_6H_4$ (**2l**)	0	22	**5al**/89
13	3-MeO-4-$TsOC_6H_3$ (**2m**)	0	23	**5am**/77
14	3,4,5-$(MeO)_3C_6H_2$ (**2n**)	30	18	**5an**/89
15	(*E*)-PhCH=CH (**2o**)	0	28	**5ao**/94
16	$CH_3CH_2CH_2$ (**2p**)	0	42	**5ap**/63

[a]The reaction was conducted with 0.3 mmol of *cis*-**1a**. [b]Isolated yield.

Table 3. Cyclopropane Scope in the $AlCl_3$-Promoted Annulations[a]



entry	Ar^1	Ar^2	temp (°C)	time (h)	product/yield[b] (%)
1	4-ClC_6H_4 (*cis*-**1b**)	C_6H_5 (**2a**)	0	18	**5ba**/96
2	4-ClC_6H_4 (*cis*-**1b**)	4-ClC_6H_4 (**2b**)	0	19	**5bb**/91
3	4-ClC_6H_4 (*cis*-**1b**)	4-$MeOC_6H_4$ (**2g**)	30	40	**5bg**/98
4	4-ClC_6H_4 (*cis*-**1b**)	3,4-$(MeO)_2C_6H_3$ (**2h**)	30	18	**5bh**/85
5	4-$NO_2C_6H_4$ (*cis*-**1c**)	C_6H_5 (**2a**)	0	96	**5ca**/81
6	4-$NO_2C_6H_4$ (*cis*-**1c**)	4-ClC_6H_4 (**2b**)	0	110	**5cb**/27
7	4-$NO_2C_6H_4$ (*cis*-**1c**)	4-$MeOC_6H_4$ (**2g**)	30	93	**5cg**/82
8	4-$NO_2C_6H_4$ (*cis*-**1c**)	3,4-$(MeO)_2C_6H_3$ (**2h**)	30	110	**5ch**/82
9	4-$MeOC_6H_4$ (*cis*-**1d**)	C_6H_5 (**2a**)	0	16	**5da**/87
10	4-$MeOC_6H_4$ (*cis*-**1d**)	4-ClC_6H_4 (**2b**)	0	70	**5db**/82
11	4-$MeOC_6H_4$ (*cis*-**1d**)	4-$MeOC_6H_4$ (**2g**)	30	38	**5dg**/81
12	4-$MeOC_6H_4$ (*cis*-**1d**)	3,4-$(MeO)_2C_6H_3$ (**2h**)	30	14	**5dh**/90
13	3,4-$(MeO)_2C_6H_3$ (*cis*-**1e**)	C_6H_5 (**2a**)	0	46	**5ea**/86
14	3,4-$(MeO)_2C_6H_3$ (*cis*-**1e**)	4-ClC_6H_4 (**2b**)	0	40	**5eb**/87
15	3,4-$(MeO)_2C_6H_3$ (*cis*-**1e**)	4-$MeOC_6H_4$ (**2g**)	30	47	**5eg**/83
16	3,4-$(MeO)_2C_6H_3$ (*cis*-**1e**)	3,4-$(MeO)_2C_6H_3$ (**2h**)	30	40	**5eh**/91

[a]The reaction was conducted with 0.3 mmol of *cis*-**1**; the relative configuration of **5** was determined by NOESY analysis. [b]Isolated yield.

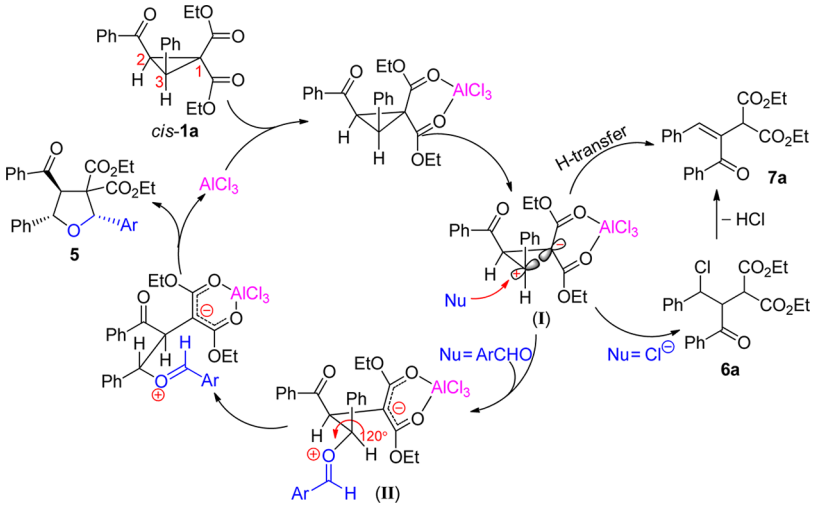
product **5**. On the other hand, the intermediate (**I**) may also receive the nucleophilic attack by chloride ion to give the byproduct **6a**, or undergo proton transfer processes to give the other byproduct **7a**, which might also be derived from the decholorination of **6a**.

CONCLUSION

In conclusion, we have applied *cis*-2,3-disubstituted cyclopropane 1,1-diesters to the [3 + 2]-annulations with aldehydes under the promotion of $AlCl_3$. Compared to their *trans* counterparts previously studied, the use of these *cis* cyclopropanes in the annulation led to drastically different results: the desired polysubstituted tetrahydrofuran products were obtained in higher yields, and more importantly, as single diastereomers different from those obtained with the corresponding *trans* cyclopropanes. Such results highlighted the significant impact of the substitution type of the D−A cyclopropanes on the efficiency and stereoselectivity of the [3 + 2]-annulation.

dx.doi.org/10.1021/jo400554a | J. Org. Chem. 2013, 78, 5393−5400

Scheme 2. Possible Mechanism for the [3 + 2] Annulation



EXPERIMENTAL SECTION

General Information. All the annulations of cyclopropane-1,1-diesters with aromatic aldehydes were carried out with flame-dried Schlenk-type glassware using a Schlenk line. All of the *cis*-2,3-disubstituted cyclopropane 1,1-diesters used were synthesized according to a reference.[5] Aluminum trichloride was purified by sublimation of the commercial product under vacuum (residual gas N_2) at 170 °C.[8] All other reagents were purchased from commercial suppliers and purified by standard techniques. Flash column chromatography was performed using silica gel (200−400 mesh). For thin-layer chromatography (TLC), silica gel plates (HSGF 254) were used and compounds were visualized by irradiation with UV light. ^{1}H NMR (300 MHz) and ^{13}C NMR (75 MHz) spectra were recorded at 300 MHz NMR spectrometer in $CDCl_3$. All chemical shifts (δ) are given in ppm relative to TMS (δ = 0 ppm) as internal standard. Data are reported as follows: chemical shift, multiplicity, coupling constants and integration. Melting points were uncorrected. IR spectra were reported in frequency of absorption (cm^{-1}). High-resolution mass spectral (HRMS) data were obtained with an ionization mode of ESI and a TOF analyzer.

General Procedure for the Annulation Reaction. To a solution of *cis*-2,3-disubstituted cyclopropane-1,1-diesters (*cis*-**1**) (0.3 mmol) and aldehydes (1.5 mmol) in 10.0 mL of dichloromethane was added $AlCl_3$ (0.15 mmol) at 0 or 30 °C. The reaction mixture was stirred at 0 or 30 °C and monitored by TLC. Upon completion, the reaction mixture was passed through a small plug of silica, eluting with 20 mL of CH_2Cl_2, and the solvent was removed under vacuum. ^{1}H NMR analyses of the unpurified products gave the diastereomeric ratios. The crude products were purified by flash chromatography to give the pure products.

Diethyl *r*-4-Benzoyl-*t*-2,*t*-5-diphenyltetrahydrofuran-3,3-dicarboxylate (5aa).[4] Purified by column chromatography (petroleum ether/ethyl acetate = 10/1) to afford a white solid in 91% yield (129 mg). Mp: 106−107 °C. IR (KBr, cm^{-1}): ν 1751, 1720, 1678, 1597, 1582, 1495, 1472, 1450, 1082, 1055, 1030, 770, 750, 696. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.85−7.79 (m, 2H), 7.65−7.60 (m, 2H), 7.54−7.47 (m, 3H), 7.40−7.24 (m, 8H), 6.01 (s, 1H), 5.31 (d, J = 8.9 Hz, 1H), 5.23 (d, J = 8.9 Hz, 1H), 4.04 (dq, J = 7.2, 3.5 Hz, 1H), 3.86 (dq, J = 7.2, 3.6 Hz, 1H), 3.79 (dq, J = 7.2, 3.5 Hz, 1H), 3.32 (dq, J = 7.2, 3.5 Hz, 1H), 0.82 (t, J = 7.2 Hz, 3H), 0.70 (t, J = 7.2 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 199.4, 168.9, 168.0, 138.7, 137.5, 136.9, 133.6, 128.7, 128.62, 128.59, 128.5, 128.3, 127.9, 127.3, 126.7, 85.5, 85.1, 70.8, 62.0, 61.5, 59.6, 13.2. HRMS (ESI-TOF): calcd for $C_{29}H_{29}O_6$ ([M + H]$^+$) 473.1964, found 473.1961.

Diethyl *r*-4-Benzoyl-*t*-2-(4-chlorophenyl)-*t*-5-phenyltetrahydrofuran-3,3-dicarboxylate (5ab). Purified by column chromatography (petroleum ether/ethyl acetate =10/1) to afford a white solid in 87% yield (133 mg). Mp: 112−113 °C. IR (KBr, cm^{-1}): ν 1755, 1732, 1678, 1597, 1580, 1491, 1448, 1090, 1070, 1016, 856, 760, 698. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.85−7.76 (m, 2H), 7.64−7.46 (m, 5H), 7.42−7.28 (m, 7H), 5.96 (s, 1H), 5.29 (d, J = 8.7 Hz, 1H), 5.22 (d, J = 8.8 Hz, 1H), 4.10−3.97 (m, 1H), 3.93−3.76 (m, 2H), 3.50−3.36 (m, 1H), 0.85−0.72 (m, 6H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 199.3, 168.7, 167.9, 138.5, 137.4, 135.4, 134.1, 133.6, 128.7, 128.6, 128.1, 126.7, 85.3, 84.7, 70.6, 62.1, 61.6, 59.5, 13.3, 13.2. HRMS (ESI-TOF): calcd for $C_{29}H_{28}O_6Cl$ ([M + H]$^+$) 507.1574, found 507.1569.

Diethyl *r*-4-Benzoyl-*t*-2-(2,4-dichlorophenyl)-*t*-5-phenyltetrahydrofuran-3,3-dicarboxylate (5ac). Purified by column chromatography (petroleum ether/ethyl acetate = 20/1) to afford a white solid in 25% yield (41 mg). Mp: 128−129 °C. IR (KBr, cm^{-1}): ν 1757, 1726, 1670, 1595, 1580, 1474, 1448, 1098, 1072, 1040, 891, 864, 843, 766, 698. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.99−7.93 (m, 2H), 7.62 (d, J = 8.5 Hz, 1H), 7.54−7.22 (m, 10H), 6.55 (s, 1H), 5.45 (d, J = 10.3 Hz, 1H), 5.12 (d, J = 10.3 Hz, 1H), 4.15 (dq, J = 7.1, 3.6 Hz, 1H), 3.91 (dq, J = 7.2, 3.5 Hz, 1H), 3.82 (dq, J = 7.1, 3.5 Hz, 1H), 3.36 (dq, J = 7.2, 3.5 Hz, 1H), 0.93 (t, J = 7.1 Hz, 3H), 0.86 (t, J = 7.2 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 197.6, 168.5, 167.3, 137.5, 137.4, 135.0, 134.8, 134.7, 133.6, 130.2, 129.0, 128.8, 128.7, 128.6, 128.5, 126.9, 126.5, 83.8, 81.2, 70.4, 62.0, 59.4, 13.4, 13.3. HRMS (ESI-TOF): calcd for $C_{29}H_{27}O_6Cl_2$ ([M + H]$^+$) 541.1184, found 541.1181.

Diethyl *r*-4-Benzoyl-*t*-2-(4-bromophenyl)-*t*-5-phenyltetrahydrofuran-3,3-dicarboxylate (5ad). Purified by column chromatography (petroleum ether/ethyl acetate = 10/1) to afford a white solid in 81% yield (134 mg). Mp: 113−114 °C. IR (KBr, cm^{-1}): ν 1753, 1730, 1666, 1595, 1489, 1448, 1097, 1068, 1032, 860, 767, 702. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.84−7.77 (m, 2H), 7.57−7.45 (m, 7H), 7.40−7.27 (m, 5H), 5.94 (s, 1H), 5.28 (d, J = 8.8 Hz, 1H), 5.22 (d, J = 8.8 Hz, 1H), 4.03 (dq, J = 7.2, 3.6 Hz, 1H), 3.92−3.77 (m, 2H), 3.44 (dq, J = 7.1, 3.5 Hz, 1H), 0.82 (t, J = 7.2 Hz, 3H), 0.77 (t, J = 7.1 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 199.3, 168.7, 167.9, 138.5, 137.4, 136.0, 133.6, 131.0, 129.0, 128.7, 128.6, 126.8, 122.3, 85.3, 84.8, 70.6, 62.1, 61.6, 59.5, 13.3, 13.2. HRMS (ESI-TOF): calcd for $C_{29}H_{28}O_6Br$ ([M + H]$^+$) 551.1069, found 551.1066.

Diethyl *r*-4-Benzoyl-*t*-5-phenyl-*t*-2-(4-tolyl)tetrahydrofuran-3,3-dicarboxylate (5af). Purified by column chromatography (petroleum ether/ethyl acetate = 10/1) to afford a white solid in 91% yield (133 mg). Mp: 125−126 °C. IR (KBr, cm^{-1}): ν 1753, 1719, 1670, 1595, 1516, 1448, 1061, 766, 702, 687, 654. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.82 (d, J = 7.5 Hz, 2H), 7.59−7.43 (m, 5H), 7.41−7.22 (m, 5H), 7.15 (d, J = 8.1 Hz, 2H), 5.97 (s, 1H), 5.29 (d, J = 9.0 Hz, 1H), 5.20 (d, J = 8.7 Hz, 1H), 4.11−3.95 (m, 1H), 3.93−3.71 (m, 2H), 3.45−3.29 (m, 1H), 2.34 (s, 3H), 0.82 (t, J = 7.2 Hz, 3H), 0.72 (t, J = 7.2 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 199.4, 168.9, 168.1, 138.8, 138.0, 137.6, 133.9, 133.5, 128.7, 128.6, 128.5, 128.4, 127.1, 126.7, 85.5, 85.0, 70.7, 61.9, 61.4, 59.6, 21.2, 13.24, 13.17.

dx.doi.org/10.1021/jo400554a | J. Org. Chem. 2013, 78, 5393−5400

HRMS (ESI-TOF): calcd for $C_{30}H_{31}O_6$ ([M + H]$^+$) 487.2120, found 487.2110.

Diethyl *r*-4-Benzoyl-*t*-2-(4-anisyl)-*t*-5-phenyltetrahydrofuran-3,3-dicarboxylate (5ag). Purified by column chromatography (petroleum ether/ethyl acetate = 8/1) to afford a white solid in 93% yield (140 mg). Mp: 105−106 °C. IR (KBr, cm^{-1}): ν 1748, 1720, 1672, 1614, 1595, 1580, 1518, 1462, 1450, 1070, 820, 770, 702. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.85−7.78 (m, 2H), 7.58−7.46 (m, 5H), 7.41−7.26 (m, 5H), 6.91−6.85 (m, 2H), 5.96 (s, 1H), 5.28 (d, J = 8.9 Hz, 1H), 5.20 (d, J = 8.9 Hz, 1H), 4.03 (dq, J = 7.1, 3.6 Hz, 1H), 3.94−3.74 (m, 5H), 3.41 (dq, J = 7.2, 3.5 Hz, 1H), 0.82 (t, J = 7.2 Hz, 3H), 0.76 (t, J = 7.1 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 199.4, 169.0, 168.1, 159.7, 138.8, 137.6, 133.5, 129.1, 128.7, 128.60, 128.5, 126.7, 113.3, 85.3, 84.9, 70.6, 61.9, 61.4, 59.6, 13.3, 13.2. HRMS (ESI-TOF): calcd for $C_{30}H_{30}O_7Na$ ([M + Na]$^+$) 525.1889, found 525.1884.

Diethyl *r*-4-Benzoyl-*t*-2-(3,4-dimethoxyphenyl)-*t*-5-phenyltetrahydrofuran-3,3-dicarboxylate (5ah). Purified by column chromatography (petroleum ether/ethyl acetate = 5/1) to afford a white solid in 91% yield (146 mg). Mp: 150−151 °C. IR (KBr, cm^{-1}): ν 1755, 1720, 1676, 1595, 1516, 1452, 1092, 1045, 1028, 862, 806, 760, 702. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.86−7.78 (m, 2H), 7.55−7.46 (m, 3H), 7.42−7.25 (m, 5H), 7.23−7.14 (m, 2H), 6.86 (d, J = 8.3 Hz, 1H), 5.95 (s, 1H), 5.29 (d, J = 8.9 Hz, 1H), 5.21 (d, J = 8.9 Hz, 1H), 4.03 (dq, J = 7.2, 3.6 Hz, 1H), 3.92 (s, 3H), 3.91−3.79 (m, 5H), 3.42 (dq, J = 7.2, 3.5 Hz, 1H), 0.82 (t, J = 7.1 Hz, 3H), 0.78 (t, J = 7.2 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 199.4, 169.0, 168.1, 149.0, 148.4, 138.7, 137.5, 133.5, 129.4, 128.7, 128.60, 128.56, 128.5, 126.7, 119.7, 110.6, 110.5, 110.4, 110.3, 85.3, 84.9, 70.6, 62.0, 61.5, 59.5, 55.95, 55.93, 13.4, 13.2. HRMS (ESI-TOF): calcd for $C_{31}H_{32}O_8Na$ ([M + Na]$^+$) 555.1995, found 555.1994.

Diethyl *r*-4-Benzoyl-*t*-2-(4-benzyloxyphenyl)-*t*-5-phenyltetrahydrofuran-3,3-dicarboxylate (5ai). Purified by column chromatography (petroleum ether/ethyl acetate = 10/1) to afford a white solid in 90% yield (157 mg). Mp: 128−129 °C. IR (KBr, cm^{-1}): ν 1749, 1726, 1680, 1612, 1580, 1512, 1466, 1454, 1072, 1063, 1018, 864, 810, 762, 700. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.85−7.78 (m, 2H), 7.58−7.46 (m, 5H), 7.45−7.26 (m, 10H), 6.95 (d, J = 8.8 Hz, 2H), 5.95 (s, 1H), 5.28 (d, J = 8.9 Hz, 1H), 5.19 (d, J = 8.9 Hz, 1H), 5.08 (s, 2H), 4.03 (dq, J = 7.1, 3.6 Hz, 1H), 3.92−3.73 (m, 2H), 3.34 (dq, J = 7.2, 3.4 Hz, 1H), 0.81 (t, J = 7.2 Hz, 3H), 0.72 (t, J = 7.2 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 199.4, 169.0, 168.1, 158.7, 138.8, 137.6, 137.0, 133.5, 129.4, 128.7, 128.60, 128.5, 127.9, 127.4, 126.7, 114.4, 85.2, 85.0, 70.6, 69.9, 61.9, 61.4, 59.6, 13.3, 13.2. HRMS (ESI-TOF): calcd for $C_{36}H_{38}O_7N$ ([M + NH$_4$]$^+$) 596.2648, found 596.2646.

Diethyl *r*-4-Benzoyl-*t*-2-(4-benzyloxy-3-methoxyphenyl)-*t*-5-phenyltetrahydrofuran-3,3-dicarboxylate (5aj). Purified by column chromatography (petroleum ether/ethyl acetate = 8/1) to afford a white solid in 91% yield (166 mg). Mp: 134−135 °C. IR (KBr, cm^{-1}): ν 1759, 1728, 1670, 1593, 1518, 1466, 1448, 1061, 1036, 1009, 848, 746, 700. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.83−7.77 (m, 2H), 7.53−7.22 (m, 13H), 7.17 (d, J = 1.9 Hz, 1H), 7.09 (dd, J = 8.3, 1.9 Hz, 1H), 6.84 (d, J = 8.3 Hz, 1H), 5.91 (s, 1H), 5.26 (d, J = 8.9 Hz, 1H), 5.18 (d, J = 8.9 Hz, 1H), 5.16 (s, 2H), 4.06−3.95 (m, 1H), 3.92 (s, 3H), 3.89−3.72 (m, 2H), 3.30 (dq, J = 7.2, 3.5 Hz, 1H), 0.80 (t, J = 7.2 Hz, 3H), 0.70 (t, J = 7.2 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 199.4, 169.0, 168.1, 149.2, 148.0, 138.7, 137.6, 137.2, 133.5, 130.0, 128.7, 128.62, 128.59, 128.53, 128.49, 127.8, 127.2, 126.7, 119.6, 113.7, 111.1, 85.3, 85.0, 71.0, 70.6, 62.0, 61.5, 59.6, 56.1, 13.4, 13.2. HRMS (ESI-TOF): calcd for $C_{37}H_{40}O_8N$ ([M + NH$_4$]$^+$) 626.2753, found 626.2751.

Diethyl *r*-4-Benzoyl-*t*-2-(4-acetoxy-3-methoxyphenyl)-*t*-5-phenyltetrahydrofuran-3,3-dicarboxylate (5ak). Purified by column chromatography (petroleum ether/ethyl acetate = 10/1) to afford a white solid in 85% yield (143 mg). Mp: 161−162 °C. IR (KBr, cm^{-1}): ν 1767, 1728, 1678, 1607, 1597, 1580, 1512, 1462, 1448, 1072, 1034, 860, 823, 762, 698. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.84−7.78 (m, 2H), 7.55−7.45 (m, 3H), 7.40−7.19 (m, 7H), 7.00 (d, J = 8.1 Hz, 1H), 5.97 (s, 1H), 5.29 (d, J = 8.9 Hz, 1H), 5.23 (d, J = 8.9 Hz, 1H), 4.03 (dq, J = 7.2, 3.5 Hz, 1H), 3.93−3.78 (m, 5H), 3.40 (dq, J = 7.2, 3.5 Hz, 1H), 2.30 (s, 1H), 0.82 (t, J = 7.2 Hz, 3H), 0.81 (t, J = 7.2 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 199.4, 169.0, 168.9, 168.0, 150.7, 139.8, 138.6, 137.5, 135.7, 133.6, 128.70, 128.68, 128.63, 128.58, 126.8, 122.3, 119.5, 111.4, 85.2, 85.1, 70.7, 62.4, 61.5, 59.5, 56.0, 20.7, 13.4, 13.2. HRMS (ESI-TOF): calcd for $C_{32}H_{36}O_9N$ ([M + NH$_4$]$^+$) 578.2390, found 578.2382.

Diethyl *r*-4-Benzoyl-*t*-2-(4-tosyloxyphenyl)-*t*-5-phenyltetrahydrofuran-3,3-dicarboxylate (5al). Purified by column chromatography (petroleum ether/ethyl acetate = 10/1) to afford a white solid in 81% yield (156 mg). Mp: 128−129 °C. IR (KBr, cm^{-1}): ν 1730, 1682, 1595, 1500, 1446, 1090, 1070, 1016, 837, 762, 700. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.83−7.75 (m, 2H), 7.74−7.67 (m, 2H), 7.61−7.44 (m, 5H), 7.41−7.27 (m, 7H), 7.02−6.94 (m, 2H), 5.94 (s, 1H), 5.26 (d, J = 8.8 Hz, 1H), 5.19 (d, J = 8.8 Hz, 1H), 4.02 (dq, J = 7.2, 3.5 Hz, 1H), 3.91−3.74 (m, 2H), 3.31 (dq, J = 7.2, 3.5 Hz, 1H), 2.45 (s, 3H), 0.81 (t, J = 7.2 Hz, 3H), 0.76 (t, J = 7.2 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 199.3, 168.6, 167.9, 149.6, 145.4, 138.4, 137.4, 135.9, 133.6, 132.6, 129.8, 128.7, 128.63, 128.6, 128.5, 126.8, 121.8, 85.3, 84.7, 70.6, 62.1, 61.6, 59.5, 21.7, 13.4, 13.2. HRMS (ESI-TOF): calcd for $C_{36}H_{38}O_9NS$ ([M + NH$_4$]$^+$) 660.2267, found 660.2263.

Diethyl *r*-4-Benzoyl-*t*-2-(3-methoxy-4-tosyloxyphenyl)-*t*-5-phenyltetrahydrofuran-3,3-dicarboxylate (5am). Purified by column chromatography (petroleum ether/ethyl acetate = 8/1) to afford a white solid in 77% yield (156 mg). Mp: 155−156 °C. IR (KBr, cm^{-1}): ν 1747, 1724, 1674, 1595, 1506, 1458, 1088, 1032, 841, 820, 752, 721. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.83−7.73 (m, 4H), 7.55−7.44 (m, 3H), 7.40−7.27 (m, 7H), 7.20−7.15 (m, 2H), 7.08 (d, J = 8.8 Hz, 1H), 5.92 (s, 1H), 5.27 (d, J = 8.8 Hz, 1H), 5.20 (d, J = 8.8 Hz, 1H), 4.02 (dq, J = 7.2, 3.6 Hz, 1H), 3.91−3.78 (m, 2H), 3.64 (s, 3H), 3.37 (dq, J = 7.2, 3.6 Hz, 1H), 2.45 (s, 3H), 0.85−0.77 (m, 6H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 199.3, 168.7, 167.9, 151.4, 145.0, 138.4, 137.4, 137.0, 133.6, 133.5, 129.5, 128.7, 128.6, 128.5, 126.8, 123.3, 119.4, 111.9, 85.3, 84.8, 70.6, 62.2, 61.6, 59.4, 55.8, 21.7, 13.5, 13.2. HRMS (ESI-TOF): calcd for $C_{37}H_{40}O_{10}NS$ ([M + NH$_4$]$^+$) 690.2372, found 690.2371.

Diethyl *r*-4-Benzoyl-*t*-2-(3,4,5-trimethoxyphenyl)-*t*-5-phenyltetrahydrofuran-3,3-dicarboxylate (5an). Purified by column chromatography (petroleum ether/ethyl acetate = 7/1) to afford a white solid in 89% yield (150 mg). Mp: 116−117 °C. IR (KBr, cm^{-1}): ν 1757, 1728, 1670, 1595, 1508, 1464, 1448, 1078, 1036, 1009, 826, 758, 702. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.85−7.78 (m, 2H), 7.55−7.45 (m, 3H), 7.40−7.27 (m, 5H), 6.88 (s, 2H), 5.92 (s, 1H), 5.29 (d, J = 8.9 Hz, 1H), 5.22 (d, J = 8.9 Hz, 1H), 4.09−3.98 (m, 1H), 3.94−3.79 (m, 11H), 3.54−3.42 (m, 1H), 0.87−0.76 (m, 6H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 199.3, 168.9, 168.0, 152.9, 138.6, 138.0, 137.5, 133.6, 132.4, 128.7, 128.6, 128.5, 126.7, 104.43, 104.36, 85.4, 85.0, 70.6, 62.1, 61.5, 60.8, 59.5, 56.2, 13.4, 13.2. HRMS (ESI-TOF): calcd for $C_{32}H_{38}O_9N$ ([M + NH$_4$]$^+$) 580.2546, found 580.2537.

Diethyl *r*-4-Benzoyl-*t*-5-phenyl-*t*-2-((*E*)-styryl)tetrahydrofuran-3,3-dicarboxylate (5ao). Purified by column chromatography (petroleum ether/ethyl acetate = 9/1) to afford a white solid in 94% yield (141 mg). Mp: 104−105 °C. IR (KBr, cm^{-1}): ν 1732, 1670, 1595, 1448, 1092, 970, 764, 702, 665. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.79 (d, J = 7.2 Hz, 2H), 7.57−7.18 (m, 13H), 6.90 (d, J = 16.1 Hz, 1H), 6.37 (dd, J = 16.0, 5.6 Hz, 1H), 5.50 (d, J = 4.8 Hz, 1H), 5.22 (d, J = 8.2 Hz, 1H), 5.11 (d, J = 8.3 Hz, 1H), 4.23−4.00 (m, 3H), 3.99−3.85 (m, 1H), 1.09 (t, J = 7.1 Hz, 3H), 0.90 (t, J = 7.1 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 199.5, 168.9, 167.8, 138.7, 137.4, 136.5, 133.6, 132.0, 128.7, 128.6, 128.5, 127.8, 126.8, 126.6, 124.0, 85.4, 83.9, 69.4, 62.2, 61.5, 59.2, 14.0, 13.4. HRMS (ESI-TOF): calcd for $C_{31}H_{31}O_6$ ([M + H]$^+$) 499.2120, found 499.2117.

Diethyl *r*-4-Benzoyl-*t*-5-phenyl-*t*-2-propyltetrahydrofuran-3,3-dicarboxylate (5ap). Purified by column chromatography (petroleum ether/ethyl acetate = 15/1) to afford a white solid in 63% yield (83 mg). Mp: 93−94 °C. IR (KBr, cm^{-1}): ν 1751, 1734, 1672, 1597, 1579, 1496, 1477, 1446, 1093, 1066, 1047, 763, 704, 653. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.79−7.68 (m, 2H), 7.56−7.46 (m, 1H), 7.44−7.22 (m, 7H), 5.10−4.96 (m, 2H), 4.78−4.67 (m, 1H), 4.37−4.21 (m, 2H), 4.10−3.96 (m, 1H), 3.94−3.80 (m, 1H), 1.96−

dx.doi.org/10.1021/jo400554a | J. Org. Chem. 2013, 78, 5393−5400

1.84 (m, 1H), 1.76−1.39 (m, 3H), 1.29 (t, J = 7.1 Hz, 3H), 0.99 (t, J = 7.1 Hz, 3H), 0.87 (t, J = 7.2 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 199.8, 169.4, 167.9, 138.8, 137.2, 133.2, 128.4, 128.3, 128.1, 126.6, 85.2, 84.2, 67.9, 61.9, 61.1, 59.5, 33.3, 20.1, 13.9, 13.8, 13.1. HRMS (ESI-TOF): calcd for $C_{26}H_{30}O_6Na$ ([M + Na]$^+$) 461.1940, found 461.1940.

Diethyl *r*-4-Benzoyl-*t*-5-(4-chlorophenyl)-*t*-2-phenyltetrahydrofuran-3,3-dicarboxylate (5ba). Purified by column chromatography (petroleum ether/ethyl acetate = 10/1) to afford a white solid in 96% yield (146 mg). Mp: 118−119 °C. IR (KBr, cm^{-1}): ν 1751, 1724, 1674, 1595, 1581, 1492, 1473, 1448, 1085, 1064, 1016, 862, 833, 758, 705, 686. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.92−7.78 (m, 2H), 7.67−7.50 (m, 3H), 7.49−7.27 (m, 9H), 5.98 (s, 1H), 5.28 (d, J = 8.7 Hz, 1H), 5.15 (d, J = 9.0 Hz, 1H), 4.10−3.96 (m, 1H), 3.92−3.72 (m, 2H), 3.40−3.25 (m, 1H), 0.81 (d, J = 7.2 Hz, 3H), 0.69 (d, J = 7.2 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 199.2, 168.8, 167.8, 137.4, 137.3, 136.7, 134.2, 133.7, 128.8, 128.7, 128.4, 128.1, 128.0, 127.2, 85.5, 84.3, 70.7, 62.0, 61.6, 59.5, 13.2. HRMS (ESI-TOF): calcd for $C_{29}H_{27}O_6ClNa$ ([M + Na]$^+$) 529.1394, found 529.1394.

Diethyl *r*-4-Benzoyl-*t*-2,*t*-5-bis(4-chlorophenyl)tetrahydrofuran-3,3-dicarboxylate (5bb). Purified by column chromatography (petroleum ether/ethyl acetate = 10/1) to afford a white solid in 91% yield (148 mg). Mp: 110−111 °C. IR (KBr, cm^{-1}): ν 1755, 1728, 1678, 1597, 1580, 1493, 1448, 1088, 1043, 1014, 858, 822, 814, 737, 702. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.84−7.78 (m, 2H), 7.60−7.50 (m, 3H), 7.46−7.27 (m, 8H), 5.94 (s, 1H), 5.26 (d, J = 8.7 Hz, 1H), 5.14 (d, J = 8.7 Hz, 1H), 4.02 (dq, J = 7.2, 3.5 Hz, 1H), 3.91−3.77 (m, 2H), 3.43 (dq, J = 7.2, 3.5 Hz, 1H), 0.80 (t, J = 7.2 Hz, 3H), 0.76 (t, J = 7.2 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 199.2, 168.6, 167.8, 137.3, 137.1, 135.2, 134.4, 134.3, 133.8, 128.9, 128.8, 128.7, 128.6, 128.1, 84.8, 84.5, 70.5, 62.2, 61.7, 59.4, 13.3, 13.2. HRMS (ESI-TOF): calcd for $C_{29}H_{27}O_6Cl_2$ ([M + H]$^+$) 541.1184, found 541.1180.

Diethyl *r*-4-Benzoyl-*t*-2-(4-anisyl)-*t*-5-(4-chlorophenyl)-tetrahydrofuran-3,3-dicarboxylate (5bg). Purified by column chromatography (petroleum ether/ethyl acetate = 7/1) to afford a white solid in 98% yield (158 mg). Mp: 132−133 °C. IR (KBr, cm^{-1}): ν 1749, 1722, 1672, 1612, 1597, 1517, 1494, 1448, 1078, 1045, 1014, 829, 817, 705, 684. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.82 (d, J = 7.5 Hz, 2H), 7.60−7.27 (m, 9H), 6.89 (d, J = 7.8 Hz, 2H), 5.94 (s, 1H), 5.25 (d, J = 8.7 Hz, 1H), 5.13 (d, J = 8.7 Hz, 1H), 4.10−3.95 (m, 1H), 3.93−3.75 (m, 5H), 3.49−3.32 (m, 1H), 0.91−0.70 (m, 6H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 199.2, 168.9, 167.9, 159.7, 137.4, 137.3, 134.2, 133.7, 128.81, 128.76, 128.7, 128.4, 128.1, 113.3, 85.3, 84.1, 70.5, 62.0, 61.5, 59.5, 55.3, 13.3, 13.2. HRMS (ESI-TOF): calcd for $C_{30}H_{29}O_7ClNa$ ([M + Na]$^+$) 559.1500, found 559.1500.

Diethyl *r*-4-Benzoyl-*t*-5-(4-chlorophenyl)-*t*-2-(3,4-dimethoxyphenyl)tetrahydrofuran-3,3-dicarboxylate (5bh). Purified by column chromatography (petroleum ether/ethyl acetate = 7/1) to afford a white solid in 85% yield (145 mg). Mp: 145−146 °C. IR (KBr, cm^{-1}): ν 1748, 1720, 1672, 1595, 1518, 1466, 1448, 1086, 1058, 1047, 1032, 864, 822, 810, 770, 706. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.85−7.80 (m, 2H), 7.57−7.50 (m, 1H), 7.45−7.35 (m, 4H), 7.32−7.27 (m, 2H), 7.20−7.13 (m, 2H), 6.86 (d, J = 8.2 Hz, 1H), 5.92 (s, 1H), 5.26 (d, J = 8.8 Hz, 1H), 5.14 (d, J = 8.8 Hz, 1H), 4.01 (dq, J = 7.2, 3.6 Hz, 1H), 3.97−3.79 (m, 8H), 3.42 (dq, J = 7.2, 3.5 Hz, 1H), 0.81 (t, J = 7.2 Hz, 3H), 0.77 (t, J = 7.2 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 199.2, 169.0, 167.9, 149.1, 148.6, 137.42, 137.37, 134.3, 133.7, 129.2, 128.9, 128.70, 128.68, 128.1, 119.7, 110.7, 110.5, 85.4, 84.2, 70.5, 62.1, 61.5, 59.5, 56.0, 13.4, 13.2. HRMS (ESI-TOF): calcd for $C_{31}H_{35}O_8NCl$ ([M + NH_4]$^+$) 584.2051, found 584.2043.

Diethyl *r*-4-Benzoyl-*t*-5-(4-nitrophenyl)-*t*-2-phenyltetrahydrofuran-3,3-dicarboxylate (5ca). Purified by column chromatography (petroleum ether/ethyl acetate = 5/1) to afford a white solid in 81% yield (126 mg). Mp: 165−166 °C. IR (KBr, cm^{-1}): ν 1751, 1726, 1672, 1595, 1521, 1494, 1473, 1450, 1091, 1074, 1037, 854, 748, 705, 671. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 8.18 (d, J = 7.8 Hz, 2H), 7.85 (d, J = 7.8 Hz, 2H), 7.69−7.51 (m, 5H), 7.46−7.30 (m, 5H), 6.03 (s, 1H), 5.43 (d, J = 8.7 Hz, 1H), 5.13 (d, J = 9.0 Hz, 1H), 4.10−3.94 (m, 1H), 3.91−3.73 (m, 2H), 3.42−3.26 (m, 1H), 0.80 (t, J = 7.2 Hz, 3H), 0.70 (t, J = 7.2 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 198.8, 168.6, 167.5, 147.9, 146.2, 137.2, 136.3, 134.0, 128.8, 128.7, 128.6, 128.1, 127.3, 127.2, 123.9, 85.7, 83.5, 70.6, 62.2, 61.7, 59.3, 13.2. HRMS (ESI-TOF): calcd for $C_{29}H_{28}NO_8$ ([M + H]$^+$) 518.1815, found 518.1815.

Diethyl *r*-4-Benzoyl-*t*-2-(4-chlorophenyl)-*t*-5-(4-nitrophenyl)tetrahydrofuran-3,3-dicarboxylate (5cb). Purified by column chromatography (petroleum ether/ethyl acetate = 5/1) to afford a white solid in 27% yield (45 mg). Mp: 89−90 °C. IR (KBr, cm^{-1}): ν 1751, 1728, 1678, 1597, 1581, 1521, 1490, 1473, 1448, 1087, 1035, 1014, 856, 813, 752, 705, 665. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 8.19 (d, J = 8.7 Hz, 2H), 7.83 (d, J = 7.2 Hz, 2H), 7.68−7.51 (m, 5H), 7.47−7.31 (m, 4H), 5.98 (s, 1H), 5.41 (d, J = 8.7 Hz, 1H), 5.11 (d, J = 8.7 Hz, 1H), 4.09−3.95 (m, 1H), 3.92−3.76 (m, 2H), 3.52−3.37 (m, 1H), 0.88−0.70 (m, 6H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 198.7, 168.4, 167.3, 147.9, 145.8, 137.0, 134.7, 134.4, 134.0, 128.8, 128.6, 128.5, 128.1, 127.2, 123.8, 84.9, 83.6, 70.4, 62.2, 61.7, 59.1, 13.2, 13.1. HRMS (ESI-TOF): calcd for $C_{29}H_{26}NO_8ClK$ ([M + K]$^+$) 590.0984, found 590.0979.

Diethyl *r*-4-Benzoyl-*t*-2-(4-anisyl)-*t*-5-(4-nitrophenyl)tetrahydrofuran-3,3-dicarboxylate (5cg). Purified by column chromatography (petroleum ether/ethyl acetate = 5/1) to afford a white solid in 82% yield (135 mg). Mp: 112−113 °C. IR (KBr, cm^{-1}): ν 1751, 1728, 1672, 1595, 1521, 1473, 1448, 1083, 1028, 854, 831, 732, 702, 665. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 8.18 (d, J = 8.7 Hz, 2H), 7.85 (d, J = 7.5 Hz, 2H), 7.71−7.50 (m, 5H), 7.47−7.36 (m, 2H), 6.91 (d, J = 8.8 Hz, 2H), 5.99 (s, 1H), 5.41 (d, J = 8.7 Hz, 1H), 5.10 (d, J = 8.8 Hz, 1H), 4.10−3.95 (m, 1H), 3.93−3.78 (m, 4H), 3.51−3.36 (m, 1H), 0.87−0.71 (m, 6H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 198.9, 168.8, 167.6, 159.9, 147.9, 146.2, 137.2, 134.0, 128.8, 128.7, 128.4, 127.3, 123.9, 113.4, 85.5, 83.4, 70.5, 62.2, 61.6, 59.3, 55.4, 13.3, 13.2. HRMS (ESI-TOF): calcd for $C_{30}H_{29}NO_9Na$ ([M + Na]$^+$) 570.1740, found 570.1742.

Diethyl *r*-4-Benzoyl-*t*-2-(3,4-dimethoxyphenyl)-*t*-5-(4-nitrophenyl)tetrahydrofuran-3,3-dicarboxylate (5ch). Purified by column chromatography (petroleum ether/ethyl acetate = 4/1) to afford a white solid in 82% yield (142 mg). Mp: 142−143 °C. IR (KBr, cm^{-1}): ν 1747, 1720, 1674, 1606, 1597, 1527, 1517, 1465, 1450, 1047, 1031, 1014, 856, 810, 768, 698, 655. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 8.19 (d, J = 8.6 Hz, 2H), 7.85 (d, J = 7.6 Hz, 2H), 7.70−7.52 (m, 3H), 7.49−7.36 (m, 2H), 7.23−7.11 (m, 2H), 6.88 (d, J = 8.2 Hz, 1H), 5.97 (s, 1H), 5.41 (d, J = 8.5 Hz, 1H), 5.12 (d, J = 8.6 Hz, 1H), 4.09−3.77 (m, 9H), 3.53−3.37 (m, 1H), 0.87−0.71 (m, 6H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 198.8, 168.8, 167.6, 149.2, 148.5, 147.9, 146.1, 137.1, 134.0, 128.8, 128.7, 127.3, 123.9, 119.6, 110.6, 110.4, 85.6, 83.4, 70.4, 62.2, 61.7, 59.3, 56.0, 13.4, 13.2. HRMS (ESI-TOF): calcd for $C_{31}H_{31}NO_{10}Na$ ([M + Na]$^+$) 600.1846, found 600.1846.

Diethyl *r*-4-Benzoyl-*t*-5-(4-anisyl)-*t*-2-phenyltetrahydrofuran-3,3-dicarboxylate (5da). Purified by column chromatography (petroleum ether/ethyl acetate = 15/1) to afford a white solid in 87% yield (131 mg). Mp: 109−110 °C. IR (KBr, cm^{-1}): ν 1753, 1728, 1674, 1614, 1598, 1583, 1446, 1085, 1064, 1033, 839, 758, 700, 671. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.82 (d, J = 7.5 Hz, 2H), 7.61 (d, J = 6.6 Hz, 2H), 7.55−7.27 (m, 8H), 6.85 (d, J = 8.7 Hz, 2H), 5.97 (s, 1H), 5.23 (s, 2H), 4.11−3.97 (m, 1H), 3.94−3.72 (m, 5H), 3.39−3.25 (m, 1H), 0.83 (t, J = 7.2 Hz, 3H), 0.70 (t, J = 7.2 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 199.5, 168.9, 168.1, 159.7, 137.6, 137.0, 133.5, 130.8, 128.7, 128.6, 128.3, 128.2, 127.9, 127.3, 114.0, 85.3, 84.9, 70.7, 61.9, 61.4, 59.6, 55.2, 13.3, 13.2. HRMS (ESI-TOF): calcd for $C_{30}H_{30}O_7Na$ ([M + Na]$^+$) 525.1889, found 525.1888.

Diethyl *r*-4-Benzoyl-*t*-5-(4-anisyl)-*t*-2-(4-chlorophenyl)-tetrahydrofuran-3,3-dicarboxylate (5db). Purified by column chromatography (petroleum ether/ethyl acetate = 10/1) to afford a white solid in 82% yield (132 mg). Mp: 107−108 °C. IR (KBr, cm^{-1}): ν 1749, 1724, 1678, 1614, 1595, 1516, 1448, 1056, 1031, 844, 815, 765, 692, 650. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.86−7.75 (m, 2H), 7.63−7.27 (m, 9H), 6.86 (d, J = 8.6 Hz, 2H), 5.92 (s, 1H), 5.21 (s, 2H), 4.11−3.96 (m, 1H), 3.93−3.81 (m, 2H), 3.78 (s, 3H), 3.49−3.35 (m, 1H), 0.82 (t, J = 7.1 Hz, 3H), 0.77 (t, J = 7.2 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 199.4, 168.8, 168.0, 159.8, 137.4, 135.5, 134.1, 133.6, 130.5, 128.7, 128.6, 128.3, 128.0, 114.0, 85.1, 84.6, 70.5, 62.1,

dx.doi.org/10.1021/jo400554a | J. Org. Chem. 2013, 78, 5393−5400

61.5, 59.5, 55.2, 13.2. HRMS (ESI-TOF): calcd for $C_{30}H_{29}O_7ClNa$ ([M + Na]$^+$) 559.1500, found 559.1500.

Diethyl *r*-4-Benzoyl-*t*-2,*t*-5-bis(4-anisyl)tetrahydrofuran-3,3-dicarboxylate (5dg). Purified by column chromatography (petroleum ether/ethyl acetate = 10/1) to afford a white solid in 81% yield (130 mg). Mp: 21−22 °C. IR (KBr, cm^{-1}): ν 1751, 1726, 1676, 1614, 1514, 1463, 1448, 1074, 1033, 852, 831, 812, 758, 690, 665. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.81 (d, J = 7.2 Hz, 2H), 7.61−7.32 (m, 7H), 6.94−6.80 (m, 4H), 5.92 (s, 1H), 5.21 (s, 2H), 4.10−3.96 (m, 1H), 3.93−3.72 (m, 8H), 3.47−3.34 (m, 1H), 0.82 (t, J = 7.0 Hz, 3H), 0.76 (t, J = 7.2 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 199.4, 169.0, 168.1, 159.6, 159.5, 137.4, 133.4, 130.7, 129.0, 128.6, 128.5, 128.3, 128.1, 113.9, 113.2, 85.0, 84.7, 70.5, 61.8, 61.3, 59.5, 55.2, 55.1, 13.24, 13.16. HRMS (ESI-TOF): calcd for $C_{31}H_{32}O_8K$ ([M + K]$^+$) 571.1733, found 571.1729.

Diethyl *r*-4-Benzoyl-*t*-5-(4-anisyl)-*t*-2-(3,4-dimethoxyphenyl)tetrahydrofuran-3,3-dicarboxylate (5dh). Purified by column chromatography (petroleum ether/ethyl acetate = 6/1) to afford a white solid in 90% yield (152 mg). Mp: 139−140 °C. IR (KBr, cm^{-1}): ν 1747, 1718, 1674, 1612, 1595, 1514, 1465, 1450, 1047, 1029, 864, 850, 812, 709, 692. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.88−7.78 (m, 2H), 7.57−7.33 (m, 5H), 7.23−7.12 (m, 2H), 6.92−6.81 (m, 3H), 5.91 (s, 1H), 5.21 (s, 2H), 4.09−3.97 (m, 1H), 3.96−3.80 (m, 8H), 3.78 (s, 3H), 3.49−3.36 (m, 1H), 0.82 (t, J = 7.2 Hz, 3H), 0.77 (t, J = 7.2 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 199.5, 169.1, 168.2, 159.7, 149.0, 148.5, 137.6, 133.5, 130.8, 129.5, 128.7, 128.6, 128.2, 119.8, 114.0, 110.6, 110.5, 85.2, 84.8, 70.6, 61.9, 61.4, 59.6, 55.98, 55.96, 55.2, 13.4, 13.2. HRMS (ESI-TOF): calcd for $C_{32}H_{34}O_9Na$ ([M + Na]$^+$) 585.2100, found 585.2100.

Diethyl *r*-4-Benzoyl-*t*-5-(3,4-dimethoxyphenyl)-*t*-2-phenyltetrahydrofuran-3,3-dicarboxylate (5ea). Purified by column chromatography (petroleum ether/ethyl acetate = 5/1) to afford a white solid in 86% yield (138 mg). Mp: 106−107 °C. IR (KBr, cm^{-1}): ν 1749, 1722, 1680, 1595, 1516, 1496, 1463, 1448, 1062, 1026, 862, 817, 756, 727, 702, 646. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.83 (d, J = 7.3 Hz, 2H), 7.62 (d, J = 6.6 Hz, 2H), 7.56−7.48 (m, 1H), 7.44−7.28 (m, 5H), 7.13−6.99 (m, 2H), 6.80 (d, J = 8.2 Hz, 1H), 5.97 (s, 1H), 5.23 (s, 2H), 4.11−3.98 (m, 1H), 3.95−3.72 (m, 8H), 3.41−3.25 (m, 1H), 0.84 (t, J = 7.0 Hz, 3H), 0.69 (t, J = 7.0 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 199.6, 168.9, 168.1, 149.1, 149.0, 137.6, 136.9, 133.6, 131.2, 128.7, 128.6, 128.3, 127.9, 127.2, 119.3, 111.0, 109.9, 85.4, 85.1, 70.6, 61.9, 91.4, 59.6, 55.85, 55.83, 13.3, 13.2. HRMS (ESI-TOF): calcd for $C_{31}H_{33}O_8$ ([M + H]$^+$) 533.2175, found 533.2173.

Diethyl *r*-4-Benzoyl-*t*-2-(4-chlorophenyl)-*t*-5-(3,4-dimethoxyphenyl)tetrahydrofuran-3,3-dicarboxylate (5eb). Purified by column chromatography (petroleum ether/ethyl acetate = 5/1) to afford a white solid in 87% yield (148 mg). Mp: 134−135 °C. IR (KBr, cm^{-1}): ν 1753, 1728, 1683, 1597, 1579, 1516, 1489, 1463, 1448, 1076, 1029, 869, 856, 812, 767, 752, 694, 669. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.87−7.77 (m, 2H), 7.64−7.49 (m, 3H), 7.44−7.30 (m, 4H), 7.13−7.01 (m, 2H), 6.81 (d, J = 8.2 Hz, 1H), 5.92 (s, 1H), 5.27−5.16 (m, 2H), 4.11−3.98 (m, 1H), 3.94−3.77 (m, 8H), 3.49−3.36 (m, 1H), 0.84 (t, J = 7.2 Hz, 3H), 0.76 (t, J = 7.1 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 199.6, 168.7, 167.9, 149.2, 149.0, 137.4, 135.4, 134.1, 133.6, 130.9, 128.65, 128.62, 128.0, 119.3, 111.0, 109.9, 85.3, 84.6, 70.4, 62.0, 61.5, 59.5, 55.85, 55.83, 13.3. HRMS (ESI-TOF) calcd for $C_{31}H_{31}O_8ClNa$ ([M + Na]$^+$) 589.1605, found 589.1600.

Diethyl *r*-4-Benzoyl-*t*-2-(4-anisyl)-*t*-5-(3,4-dimethoxyphenyl)tetrahydrofuran-3,3-dicarboxylate (5eg). Purified by column chromatography (petroleum ether/ethyl acetate = 5/1) to afford a white solid in 83% yield (140 mg). Mp: 105−106 °C. IR (KBr, cm^{-1}): ν 1751, 1722, 1685, 1614, 1595, 1463, 1074, 1028, 862, 815, 765, 698, 671. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.82 (d, J = 8.2 Hz, 2H), 7.60−7.47 (m, 3H), 7.44−7.32 (m, 2H), 7.13−6.99 (m, 2H), 6.88 (d, J = 8.5 Hz, 2H), 6.79 (d, J = 8.2 Hz, 1H), 5.93 (s, 1H), 5.20 (s, 2H), 4.11−3.97 (m, 1H), 3.94−3.75 (m, 11H), 3.47−3.33 (m, 1H), 0.84 (t, J = 7.0 Hz, 3H), 0.76 (t, J = 7.1 Hz, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 199.7, 169.0, 168.2, 159.6, 149.04, 148.97, 137.5, 133.5, 131.2, 129.0, 128.7, 128.6, 128.4, 119.2, 113.3, 110.9, 109.8, 85.2, 84.9, 70.4, 61.9, 61.4, 59.5, 55.83, 55.81, 55.3, 13.33, 13.28. HRMS (ESI-TOF): calcd for $C_{32}H_{34}O_9Na$ ([M + Na]$^+$) 585.2100, found 585.2100.

Diethyl *r*-4-Benzoyl-*t*-2,*t*-5-bis(3,4-dimethoxyphenyl)tetrahydrofuran-3,3-dicarboxylate (5eh).[4] Purified by column chromatography (petroleum ether/ethyl acetate = 4/1) to afford a white solid in 91% yield (162 mg). Mp: 159−160 °C. IR (KBr, cm^{-1}): ν 1753, 1720, 1678, 1593, 1518, 1466, 1450, 1051, 1026, 870, 851, 813, 764, 715, 696. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.82 (d, J = 7.9 Hz, 2H), 7.58−7.48 (m, 1H), 7.45−7.33 (m, 2H), 7.24−6.97 (m, 4H), 6.86 (d, J = 8.1 Hz, 1H), 6.80 (d, J = 8.3 Hz, 1H), 5.92 (s, 1H), 5.21 (s, 2H), 4.11−3.97 (m, 1H), 3.96−3.77 (m, 14H), 3.49−3.35 (m, 1H), 0.84 (t, J = 7.1 Hz, 3H), 0.77 (t, J = 7.1 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 199.6, 169.1, 168.1, 149.04, 148.99, 148.95, 148.4, 137.5, 133.6, 131.2, 129.4, 128.65, 128.62, 119.7, 119.2, 110.9, 110.6, 110.3, 109.8, 85.2, 84.9, 70.4, 61.9, 61.4, 59.5, 55.9, 55.8, 55.7, 13.4, 13.3. HRMS (ESI-TOF): calcd for $C_{33}H_{36}O_{10}Na$ ([M + Na]$^+$) 615.2206, found 615.2205.

Diethyl 2-(1-Benzoyl-2-chloro-2-phenylethyl)malonate (6a).[4] White solid. Mp: 104−105 °C. IR (KBr, cm^{-1}): ν 1740, 1719, 1678, 1597, 1495, 1452, 1022, 758, 696. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.94 (d, J = 7.5 Hz, 2H), 7.56−7.22 (m, 8H), 5.33 (d, J = 8.1 Hz, 1H), 4.989 (t, J = 8.1 Hz, 1H), 4.15−3.82 (m, 5H), 1.20−1.10 (m, 6H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 198.7, 167.6, 167.4, 138.0, 133.0, 128.9, 128.7, 128.6, 128.5, 128.3, 128.1, 62.1, 61.9, 53.9, 51.4, 13.8. HRMS (ESI-TOF): calcd for $C_{22}H_{24}ClO_5$ ([M + H]$^+$) 403.1312, found 403.1314. Anal. Calcd for $C_{22}H_{23}ClO_5$ (402.87): C, 65.59; H, 5.75. Found: C, 65.44; H, 5.78.

Diethyl 2-(1-Benzoyl-2-phenylvinyl)malonate (7a). Colorless oil. IR (neat, cm^{-1}): 1747, 1732, 1661, 1597, 1447, 1038, 945, 700. ν . ^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.85 (d, J = 7.8 Hz, 2H), 7.61−7.34 (m, 9H), 4.89 (s, 1H), 4.33−4.12(m, 4H), 1.25 (t, J = 6.7 Hz, 6H). ^{13}C NMR ($CDCl_3$, 75 MHz): δ 196.7, 167.8, 144.1, 137.6, 134.4, 134.3, 132.2, 129.9, 129.3, 129.0, 128.8, 128.3, 61.9, 51.5, 14.0. HRMS (ESI-TOF): calcd for $C_{22}H_{23}O_5$ ([M + H]$^+$) 367.1545, found 367.1550.

■ ASSOCIATED CONTENT

Ⓢ Supporting Information

X-ray structure of **5ac**; copies of ^{1}H and ^{13}C NMR spectra for **5aa**−**5eh**, **6a** and **7a**; copies of COSY and NOESY NMR spectra for **5aa**−**5eh** and **7a**; and crystal data of **5ac** in CIF format. This material is available free of charge via the Internet at http://pubs.acs.org.

■ AUTHOR INFORMATION

Corresponding Author

*Fax: 0086-553-3883517. E-mail: gshyang@mail.ahnu.edu.cn, chaizhuo@mail.ahnu.edu.cn.

Notes

The authors declare no competing financial interest.

■ ACKNOWLEDGMENTS

We gratefully appreciate the National Natural Science Foundation of China (NSFC Nos. 21172002, 20972001, and 21202001) for financial support.

■ REFERENCES

(1) Recent reviews: (a) Yu, M.; Pagenkopf, B. L. *Tetrahedron* **2005**, *61*, 321−347. (b) Carson, C. A.; Kerr, M. A. *Chem. Soc. Rev.* **2009**, *38*, 3051−3060. (c) Campbell, M. J.; Johnson, J. S.; Parsons, A. T.; Pohlhaus, P. D.; Sanders, S. D. *J. Org. Chem.* **2010**, *75*, 6317−6325. (d) Lebold, T. P.; Kerr, M. A. *Pure Appl. Chem.* **2010**, *82*, 1797−1812. (e) Mel'nikov, M. Ya.; Budynina, E. M.; Ivanovaa, O. A.; Trushkov, I. V. *Mendeleev Commun.* **2011**, *21*, 293−301.

(2) (a) Pohlhaus, P. D.; Johnson, J. S. *J. Org. Chem.* **2005**, *70*, 1057−1059. (b) Pohlhaus, P. D.; Johnson, J. S. *J. Am. Chem. Soc.* **2005**, *127*, 16014−16015. (c) Parsons, A. T.; Campbell, M. J.; Johnson, J. S. *Org.*

Lett. **2008**, *10*, 2541−2544. (d) Pohlhaus, P. D.; Sanders, S. D.; Parsons, A. T.; Li, W.; Johnson, J. S. *J. Am. Chem. Soc.* **2008**, *130*, 8642−8650. (e) Parsons, A. T.; Johnson, J. S. *J. Am. Chem. Soc.* **2009**, *131*, 3122−3123. (f) Sanders, S. D.; Ruiz-Olalla, A.; Johnson, J. S. *Chem. Commun.* **2009**, 5135−5137. (g) Smith, A. G.; Slade, M. C.; Johnson, J. S. *Org. Lett.* **2011**, *13*, 1996−1999. (h) Xing, S.; Pan, W.; Liu, C.; Ren, J.; Wang, Z. *Angew. Chem., Int. Ed.* **2010**, *49*, 3215−3218. (i) Karadeolian, A.; Kerr, M. A. *J. Org. Chem.* **2010**, *75*, 6830−6841. (j) Benfatti, F.; Nanteuil, F.; Waser, J. *Org. Lett.* **2012**, *14*, 386−389.

(3) For selected examples, see: (a) Gupta, A.; Yadav, V. K. *Tetrahedron Lett.* **2006**, *47*, 8043−8047. (b) Benfatti, F.; Nanteuil, F.; Waser, J. *Chem.—Eur. J.* **2012**, *18*, 4844−4849. (c) Carson, C. A.; Kerr, M. A. *J. Org. Chem.* **2005**, *70*, 8242−8244. (d) Lebold, T. P.; Kerr, M. A. *Org. Lett.* **2009**, *11*, 4354−4357. (e) Parsons, A. T.; Smith, A. G.; Neel, A. J.; Johnson, J. S. *J. Am. Chem. Soc.* **2010**, *132*, 9688−9692. (f) Tomilov, Y. V.; Novikov, R. A.; Nefedov, O. M. *Tetrahedron* **2010**, *66*, 9151−9158. (g) Young, I. S.; Kerr, M. A. *Angew. Chem., Int. Ed.* **2003**, *42*, 3023−3026. (h) Kang, Y.; Sun, X.; Tang, Y. *Angew. Chem., Int. Ed.* **2007**, *46*, 3918−3921. (i) Sapeta, K.; Kerr, M. A. *J. Org. Chem.* **2007**, *72*, 8597−8599. (j) Dias, D. A.; Kerr, M. A. *Org. Lett.* **2009**, *11*, 3694−3697.

(4) Yang, G.; Shen, Y.; Li, K.; Sun, Y.; Hua, Y. *J. Org. Chem.* **2011**, *76*, 229−233.

(5) Sun, Y.; Yang, G.; Shen, Y.; Hua, Z.; Chai, Z. *Tetrahedron* **2013**, *69*, 2733−2739.

(6) See the Supporting Information for an ORTEP structure and X-ray data for **5ac** (CCDC 928917). These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

(7) For examples of the formation of similar products in related reactions, see: (a) Wu, X.; Cao, W.; Zhang, H.; Chen, J.; Jiang, H.; Deng, H.; Shao, M.; Zhang, J.; Chen, H. *Tetrahedron* **2008**, *64*, 10331−10338. (b) Kolsaker, P.; Jensen, A. K. *Acta Chem. Scand., Ser. B* **1988**, *42*, 345−353 and ref 4..

(8) Hjuler, H. A.; Mahan, A.; Von Barner, J. H.; Bjerrun, N. J. *Inorg. Chem.* **1982**, *21*, 402−406.