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In(OTf)$_3$-Catalyzed Cascade Cyclization for Construction of Oxatricyclic Compounds

Wenli Bao,[†] Yezi Tao,[†] Jiangqun Cheng,[†] Junrong Huang,[†] Jingming Cao,[†] Mengxun Zhang,[†] Weijian Ye,[†] Bo Wang,[†] Yang Li,[†] Lizhi Zhu,[†,‡] and Chi-Sing Lee*[,†,§,⊥]

[†]Laboratory of Chemical Genomics, School of Chemical Biology and Biotechnology, Peking University Shenzhen Graduate School, Shenzhen University Town, Xili, Shenzhen 518055, China

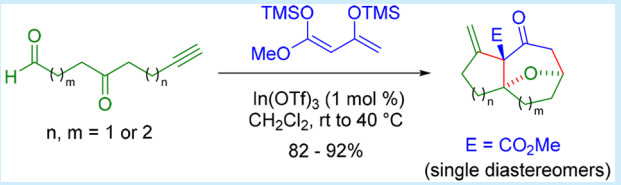
[‡]Institute of Translational Medicine, Shenzhen Second People's Hospital, The First Affiliated Hospital of Shenzhen University, Health Science Center, Shenzhen 518035, China

[§]Institute of Research and Continuing Edition (Shenzhen), Hong Kong Baptist University, Industrialization Complex Building, Shenzhen Virtual University Park, Shenzhen 518000, China

[⊥]Department of Chemistry, Hong Kong Baptist University, Kowloon Tong, Hong Kong SAR

Supporting Information

ABSTRACT: A highly diastereoselective cascade cyclization reaction has been developed for establishing a series of oxatricyclic compounds using Chan's diene and simple keto alkynal substrates with only 1 mol % of In(OTf)$_3$ as the catalyst in 82−92% yields. The potential utility of this synthetic strategy has been demonstrated in model studies for the construction the core structures of 1α,8α:4α,5α-diepoxy-4,5-dihydroosmitopsin and cortistatin A.



8-Oxabicyclo[3.2.1]octane (**I**) is a common structural motif found in polycyclic natural products.[1−4] When a five- or six-membered ring was fused at its C1,2-position, **I** became 12-oxatricyclo[7.2.1.0^{1,6}]dodecone (**II**) or 11-oxatricyclo-[6.2.1.0^{1,5}]undecane (**III**), respectively (Figure 1). These fused ring systems appeared widely among bioactive natural products, such as cortistatin A[1] and mollfoliagein E[2] (**II**-bearing natural products) (Figure 1) and corianlactone[3] and 1α,8α:4α,5α-diepoxy-4,5-dihydroosmitopsin (proposed structure)[4] (**III**-bearing natural products). Cortistatin A, isolated from the sponge *Corticium simplex* by Kobayashi's group in 2006, is particularly a promising lead for further study due to its highly potent and selective antiproliferative activity against human umbilical vein endothelial cells (HUVECs, IC$_{50}$ = 1.8 nM).[1] Due to its unique array of structural features and remarkable activities against HUVECs,[5] this natural product has attracted tremendous amounts of attention from the scientific community, including six total and semisyntheses,[6] five formal syntheses,[7] and more than 10 model studies for the core synthesis.[8] The 12-oxatricyclo[7.2.1.0^{1,6}]dodecone (**II**) fused ring system of cortistatin A has been established by a variety of synthetic strategies,[6−8] such as cycloetherification, oxa-Michael addition, aldol condensation, aza-Prins reaction, [4 + 3] cycloaddition, radical cyclization, ene-yne metathesis, rearrangement reaction, and oxidative and alkylative dearomatizations. For the construction of the 11-oxatricyclo[6.2.1.0^{1,5}]undecane (**III**) fused ring systems,[9] different synthetic strategies, such as [4 + 3] cycloaddition, air oxidation of furan, [3 + 2] cycloaddition, transannular etherification, oxonium ylide rearrangement, and radical cyclization, have been employed.

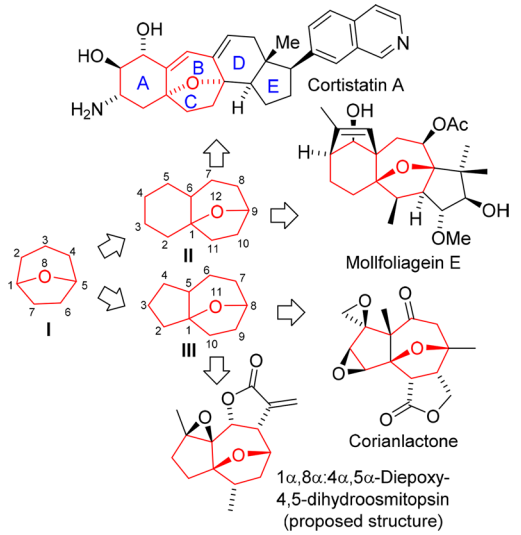


Figure 1. Examples of natural products bearing 12-oxatricyclo-[7.2.1.0^{1,6}]dodecone (**II**) and 11-oxatricyclo[6.2.1.0^{1,5}]undecane (**III**).

Our group is particularly interested in developing cascade reactions for establishing 1-oxadecalin-containing natural products, including phomactin A and cortistatin A. We have

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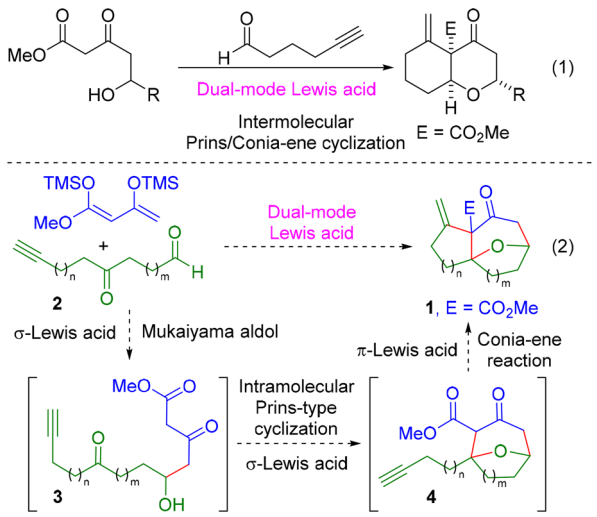
previously reported a dual-mode Lewis-acid-mediated intermolecular Prins/Conia-ene cascade cyclization for construction of the 1-oxadeclin fused ring system (Scheme 1, eq 1) and

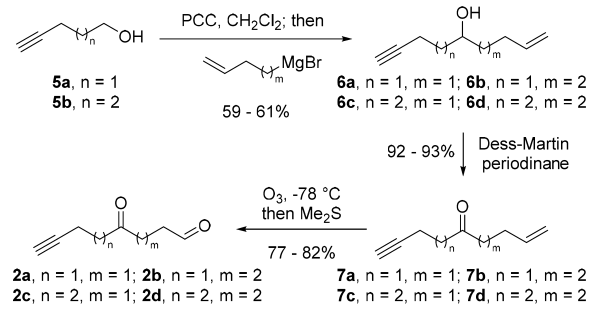
Scheme 1. Our Previous Work (1) and Strategy of This Work (2)



accomplished a formal synthesis of phomactin A using this synthetic strategy.[10] Herein, we report a two-component cascade cyclization for establishing a series of oxatricyclic compounds (**1a**–**d**) and demonstrated their potential utilities for establishing the core structures of 1α,8α:4α,5α-diepoxy-4,5-dihydroosmitopsin and cortistatin A.

A dual-mode Lewis acid that can induce reaction upon both σ- and π-binding should be able to catalyze the Mukaiyama aldol reaction between Chan's diene[11] and aldehyde **2** and the in situ intramolecular Prins/Conia-ene cascade reaction of **3**, affording tricyclic products (**1**) in a single step (Scheme 1, eq 2). To search for the appropriate conditions for this two-component cascade cyclization reaction, cyclization precursors **2a**–**d** were readily prepared from commercially available alkynols **5a** and **5b** (Scheme 2). PCC oxidation of **5a** and **5b** followed by Grignard

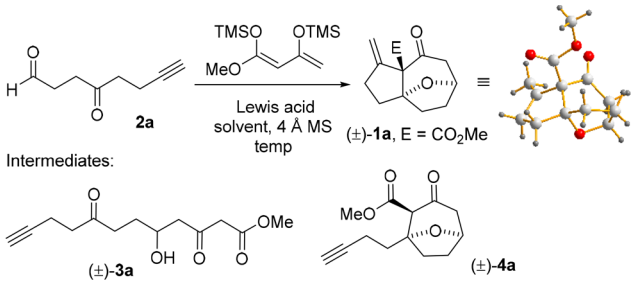
Scheme 2. Synthesis of Cyclization Precursors 2a and 2b



addition resulted in **6a**–**d**.[12] After oxidation of the resulting alcohol to ketones, ozonolysis of the terminal alkenes of **7a**–**d** afforded cyclization precursors **2a**–**d**.

With the cyclization precursors prepared, the cascade cyclization of **2a** with Chan's diene was studied using a variety of Lewis acids. As shown in Table 1, using strong σ-Lewis acids, such as $TiCl_4$, in THF at −78 °C induced Mukaiyama aldol reaction and gave (±)-**3a** as the only product (Table 1, entry 1). However, extending the reaction time led to slow decomposition

Table 1. Cascade Cyclization of 2a and Chan's Diene[a]



entry	Lewis acid (equiv), solvent	temp (°C)	time	(±)-**1a**[b]
1	$TiCl_4$ (1), THF	−78	10 min	*e*
2	$TiCl_4$ (1), CH_2Cl_2	−78	10 min	*f*
3	$TiCl_4$ (1), CH_2Cl_2	−78	30 min	trace[g]
4	$TiCl_4$ (1), CH_2Cl_2	−78 to rt	18 h	58
5	$AuCl(PPh_3)/AgSbF_6$,[c] CH_2Cl_2	rt	5 min	*h*
6	$AuCl(PPh_3)/AgSbF_6$,[c] toluene	rt	20 min	43
7	$AuCl_3$ (0.01), CH_3CN	rt	1 min	*h*
8	$ZnBr_2$ (1), CH_2Cl_2	rt to 40	1 h	60
9	ZnI_2 (1), CH_2Cl_2	rt to 40	1 h	70
10	$Zn(OTf)_2$ (1), CH_2Cl_2	rt to 40	5 h	75
11	$Zn(OTf)_2$ (1), toluene	rt to 110	5 h	72
12	InX_3[d] (1), CH_2Cl_2	rt to 40	1 h	85–89
13	$In(OTf)_3$ (1), CH_2Cl_2	rt to 40	1 h	91
14	$In(OTf)_3$ (0.5) CH_2Cl_2	rt to 40	3 h	90
15	$In(OTf)_3$ (0.1) CH_2Cl_2	rt to 40	5 h	90
16	$In(OTf)_3$ (0.01) CH_2Cl_2	rt to 40	5 h	92

[a]General procedures were followed. [b]Isolated yield (%) of (±)-**1a** after silica gel column chromatography. [c]0.03 equiv of Lewis acid was used. [d]X = Cl, Br, or I. [e]Compound (±)-**3a** (55%) was isolated. [f]Compound (±)-**3a** (78%) was isolated. [g]Detected by LC-MS along with 89% of (±)-**4a**. [h]Decomposition of substrates.

of (±)-**3a**. Switching the solvent to CH_2Cl_2 at −78 °C resulted in a higher yield of (±)-**3a** (Table 1, entry 2). Surprisingly, extending the reaction time led to a trace amount of the expected cyclization product (±)-**1a** along with 89% of the Prins cyclization intermediate (±)-**4a** (Table 1, entry 3). With this encouraging result in hand, the effects of reaction temperature were studied, and we found that the cascade reaction proceeded smoothly at room temperature for 18 h, which afforded (±)-**1a** as a single diastereomer in 58% yield (Table 1, entry 4). The structure of (±)-**1a** was confirmed unambiguously by X-ray crystallography.[12] After the study using $TiCl_4$ as the σ-Lewis acid, several π-Lewis acids were examined. $AuCl(PPh_3)/AgSbF_6$ in CH_2Cl_2 resulted in rapid decomposition of the substrates (Table 1, entry 5). Interestingly, switching the solvent to toluene led to (±)-**1a** in 43% yield (Table 1, entry 6). However, several attempts at improving the yields by changing the reaction time and temperature failed due to the stability of the substrates toward the Au(I) catalyst. The substrates are also found to be highly unstable toward $AuCl_3$ in CH_3CN (Table 1, entry 7).

In our previous study, Zn(II) and In(III) were found to be effective dual-mode Lewis acids for inducing the intermolecular Prins/Conia-ene cascade cyclization.[9] Because of this, a variety of Zn(II) and In(III) catalysts were investigated. $ZnBr_2$, ZnI_2, and $Zn(OTf)_2$ in CH_2Cl_2 at room temperature only provided intermediate (±)-**4a**. The in situ Conia-ene reaction proceeded smoothly by increasing the reaction temperature and gave (±)-**1a** in 60, 70, and 75% yields (Table 1, entries 8–10). Switching the solvent to toluene with $Zn(OTf)_2$ gave a similar

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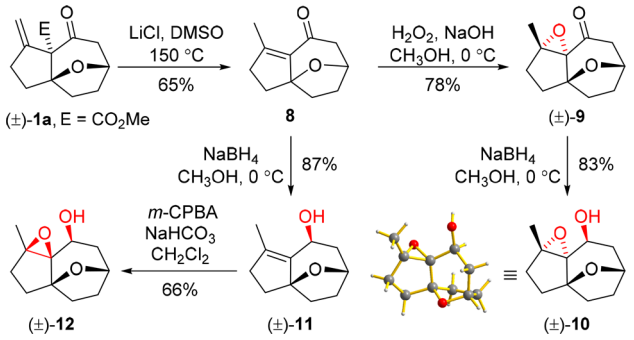
result (Table 1, entry 11). Using the same protocol for controlling the reaction temperature, InX_3 (X = Cl, Br, I or OTf) in CH_2Cl_2 gave better results than the Zn(II)-based Lewis acids (Table 1, entries 12 and 13). $In(OTf)_3/CH_2Cl_2$ was found to be the optimal dual-mode Lewis acid for the two-component cascade cyclization reaction, which provided 91% yield of (±)-**1a** in a single step. The effects of $In(OTf)_3$ loading was then studied (Table 1, entries 14−16). We found that the catalyst loading of $In(OTf)_3$ can be reduced to 1 mol % without affecting the efficiency of the reaction (Table 1, entry 16). The optimal condition for the cyclization only required 1 mol % of $In(OTf)_3$ in CH_2Cl_2 at room temperature for 10 min and then reflux for 5 h, which gave (±)-**1a** as a single diastereomer in 92%.

To investigate the potential utility of this synthetic strategy, the oxatricyclic fused ring system of (±)-**1a** was converted to the *syn*-diepoxy structure of 1*α*,8*α*:4*α*,5*α*-diepoxy-4,5-dihydroosmitopsin. Surprisingly, decarboxylation of (±)-**1a** under basic conditions led to decomposition of the substrate. Switching the condition to LiCl/DMSO at 150 °C afforded enone **8** in 65% yield (Scheme 3). Epoxidation of enone **8** using H_2O_2/NaOH at

Scheme 3. Diastereoselective Reduction and Epoxidation of Enone 8

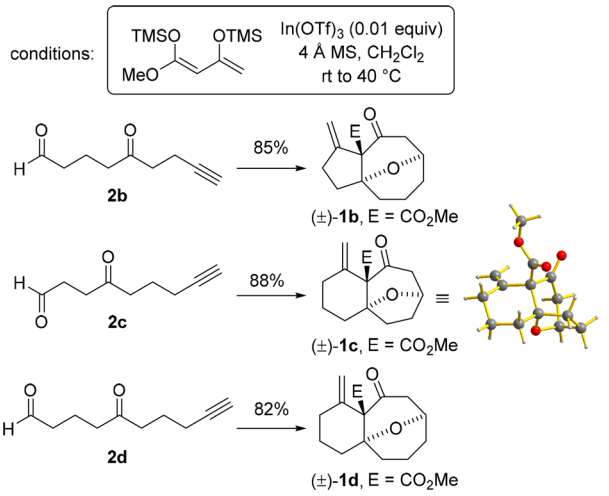


0 °C followed by $NaBH_4$ reduction of (±)-**9** at 0 °C afforded (±)-**10** as a single diastereomer. The structure and relative configurations of (±)-**10** were confirmed unambiguously by X-ray crystallography.[12] Unfortunately, the result indicated that epoxidation occurred in the concave face of enone **8** and gave the undesired epoxide diastereomer. To resolve this problem, the sequence of epoxidation and reduction was then switched. $NaBH_4$ reduction of enone **8** at 0 °C gave allylic alcohol (±)-**11** as a single diastereomer. The relative configurations of (±)-**11** were determined by NOESY.[13] Hydroxyl-directed epoxidation using *m*-CPBA afforded (±)-**12**, which was found to be a diastereomer of (±)-**10**, indicating a *syn*-epoxy structure.

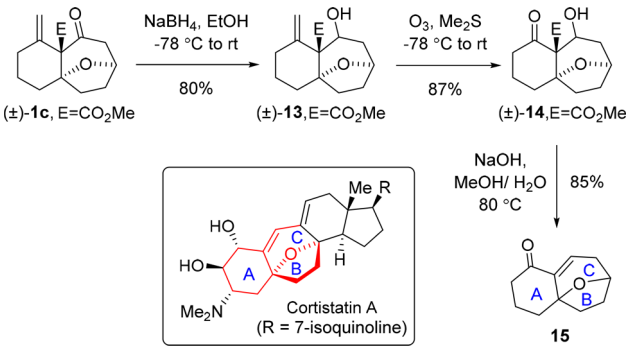
To study the scope of the substrates, cascade cyclizations of **2b−d** with Chan's diene were investigated. We found that all of the substrates underwent the cascade reaction smoothly under the optimized conditions and afforded 82−88% of the cyclized products (±)-**1b−d** with different ring sizes as a single diastereomer under the same reaction condition (Scheme 4). The structure of (±)-**1c** was confirmed unambiguously by X-ray crystallography, and the relative configurations of (±)-**1b** and (±)-**1d** were determined by NOESY.[13]

The potential utility of this cascade cyclization reaction for the synthesis of cortistatin A was also investigated by employing cyclic product (±)-**1c** as the substrate in a model study. As shown in Scheme 5, $NaBH_4$ reduction of the ketone moiety of (±)-**1c** gave good yields of (±)-**13** as a single diastereomer (the stereochemistry was not determined). After ozonolysis of the

Scheme 4. Scope of Substrates for the Cascade Cyclization



Scheme 5. Model Study of Cortistatin A



exocyclic alkene, basic hydrolysis of *β*-keto ester (±)-**14** afforded **15**, which contains the ABC tricyclic core of cortistatin A.

In summary, we have developed an $In(OTf)_3$-catalyzed two-component cascade cyclization of **2a−d** with Chan's diene and simple keto alkynal substrates. The condition of the cascade reaction was optimized by using 1 mol % of $In(OTf)_3$ in CH_2Cl_2 and provided very good yields of a series of oxatricyclic compounds **1a−d** (82−92%) diastereoselectively in a single operation. The utility of this reaction has been demonstrated in model studies by converting cyclized product (±)-**1a** to the *syn*-diepoxy structure of 1*α*,8*α*:4*α*,5*α*-diepoxy-4,5-dihydroosmitopsin and cyclized product (±)-**1c** to the ABC tricyclic core of cortistatin A in three simple transformations. We are currently exploring the utility of this synthetic strategy for synthesis of natural products that contain these oxatricyclic cores, such as cortistatin A and 1*α*,8*α*:4*α*,5*α*-diepoxy-4,5-dihydroosmitopsin.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acs.orglett.8b03461.

Experimental procedures, characterization details, and ^1H and ^{13}C NMR spectra of all new compounds (PDF)

Accession Codes

CCDC 1868304, 1868353, and 1880206 contain the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/

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cif, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.

AUTHOR INFORMATION

Corresponding Author

*E-mail: cslee-chem@hkbu.edu.hk.

ORCID

Chi-Sing Lee: 0000-0002-3564-8224

Notes

The authors declare no competing financial interest.

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REFERENCES

(1) Aoki, S.; Watanabe, Y.; Sanagawa, M.; Setiawan, A.; Kotoku, N.; Kobayashi, M. *J. Am. Chem. Soc.* **2006**, *128*, 3148.

(2) Zhou, J.; Liu, J.; Dang, T.; Zhou, H.; Zhang, H.; Yao, G. *Org. Lett.* **2018**, *20*, 2063.

(3) Shen, Y.-H.; Li, S.-H.; Li, R.-T.; Han, Q.-B.; Zhao, Q.-S.; Liang, L.; Sun, H.-D.; Lu, Y.; Cao, P.; Zheng, Q.-T. *Org. Lett.* **2004**, *6*, 1593.

(4) Bohlmann, F.; Zdero, C.; Jakupovic, J.; Rourke, J. P. *Liebigs Ann. Chem.* **1985**, *1985*, 2342.

(5) (a) Aoki, S.; Watanabe, Y.; Tanabe, D.; Arai, M.; Suna, H.; Miyamoto, K.; Tsujibo, H.; Tsujikawa, K.; Yamamoto, H.; Kobayashi, M. *Bioorg. Med. Chem.* **2007**, *15*, 6758. (b) Cee, V. J.; Chen, D. Y.-K.; Lee, M. R.; Nicolaou, K. C. *Angew. Chem., Int. Ed.* **2009**, *48*, 8952.

(6) For total and semisyntheses of cortistatins, see: (a) Shenvi, R. A.; Guerrero, C. A.; Shi, J.; Li, C.-C.; Baran, P. S. *J. Am. Chem. Soc.* **2008**, *130*, 7241. (b) Nicolaou, K. C.; Sun, Y.-P.; Peng, X.-S.; Polet, D.; Chen, D. Y.-K. *Angew. Chem., Int. Ed.* **2008**, *47*, 7310. (c) Lee, H. M.; Nieto-Oberhuber, C.; Shair, M. D. *J. Am. Chem. Soc.* **2008**, *130*, 16864. (d) Nicolaou, K. C.; Peng, X.-S.; Sun, Y.-P.; Polet, D.; Zou, B.; Lim, C. S.; Chen, D. Y.-K. *J. Am. Chem. Soc.* **2009**, *131*, 10587. (e) Flyer, A. N.; Si, C.; Myers, A. G. *Nat. Chem.* **2010**, *2*, 886. (f) Yamashita, S.; Iso, K.; Kitajima, K.; Himuro, M.; Hirama, M. *J. Org. Chem.* **2011**, *76*, 2408. (g) Shi, J.; Manolikakes, G.; Yeh, C. H.; Guerrero, C. A.; Shenvi, R. A.; Shigehisa, H.; Baran, P. S. *J. Am. Chem. Soc.* **2011**, *133*, 8014. (h) Nilson, M. G.; Funk, R. L. *J. Am. Chem. Soc.* **2011**, *133*, 12451.

(7) For formal syntheses of cortistatins, see: (a) Yamashita, S.; Kitajima, K.; Iso, K.; Hirama, M. *Tetrahedron Lett.* **2009**, *50*, 3277. (b) Simmons, E. M.; Hardin-Narayan, A. R.; Guo, X.; Sarpong, R. *Tetrahedron* **2010**, *66*, 4696. (c) Fang, L.; Chen, Y.; Huang, J.; Liu, L.; Quan, J.; Li, C.-C.; Yang, Z. *J. Org. Chem.* **2011**, *76*, 2479. (d) Kuang, L.; Liu, L. L.; Chiu, P. *Chem. - Eur. J.* **2015**, *21*, 14287. (e) Fu, J.; Gu, Y.; Yuan, H.; Luo, T.; Liu, S.; Lan, Y.; Gong, J.; Yang, Z. *Nat. Commun.* **2015**, *6*, 8617.

(8) For recent reports of model studies of cortistatins, see: (a) Dai, M.; Danishefsky, S. J. *Tetrahedron Lett.* **2008**, *49*, 6610. (b) Dai, M.; Wang, Z.; Danishefsky, S. J. *Tetrahedron Lett.* **2008**, *49*, 6613. (c) Simmons, E. M.; Hardin, A. R.; Guo, X.; Sarpong, R. *Angew. Chem., Int. Ed.* **2008**, *47*, 6650. (d) Yamashita, S.; Iso, K.; Hirama, M. *Org. Lett.* **2008**, *10*, 3413. (e) Craft, D. T.; Gung, B. W. *Tetrahedron Lett.* **2008**, *49*, 5931. (f) Magnus, P.; Littich, R. *Org. Lett.* **2009**, *11*, 3938. (g) Liu, L.; Gao, Y.; Che, C.; Wu, N.; Wang, D. Z.; Li, C. C.; Yang, Z. *Chem. Commun.* **2009**, 662. (h) Frie, J. L.; Jeffrey, C. S.; Sorensen, E. *Org. Lett.* **2009**, *11*, 5394. (i) Yu, F.; Li, G.; Gao, P.; Gong, H.; Liu, Y.; Wu, Y.; Cheng, B.; Zhai, H. *Org. Lett.* **2010**, *12*, 5135. (j) Baumgartner, C.; Ma, S.; Liu, Q.; Stoltz, B. M. *Org. Biomol. Chem.* **2010**, *8*, 2915. (k) Liu, L. L.; Chiu, P. *Chem. Commun.* **2011**, *47*, 3416. (l) Kotoku, N.; Sumii, Y.; Kobayashi, M. *Org. Lett.* **2011**, *13*, 3514. (m) Wang, Z.; Dai, M. J.; Park, P. K.; Danishefsky, S. J. *Tetrahedron* **2011**, *67*, 10249. (n) Aquino, C.; Greszler, S. N.; Micalizio, G. C. *Org. Lett.* **2016**, *18*, 2624. (o) Chan, D.; Chen, Y.; Low, K.-H.; Chiu, P. *Chem. - Eur. J.* **2018**, *24*, 2375. For the construction of the CD ring, see: (p) Kotoku, N.; Sumii, Y.; Hayashi, T.; Kobayashi, M. *Tetrahedron Lett.* **2008**, *49*, 7078. For the formation of the seven-membered ring, see: (q) Kurti, L.; Czako, B.; Corey, E. *Org. Lett.* **2008**, *10*, 5247. For synthesis of D-ring cortistatin analogues, see: (r) Shi, J.; Shigehisa, H.; Guerrero, C. A.; Shenvi, R. A.; Li, C.-C.; Baran, P. S. *Angew. Chem., Int. Ed.* **2009**, *48*, 4328.

(9) (a) Kaiser, R.; Föhlisch, B. *Helv. Chim. Acta* **1990**, *73*, 1504. (b) Kreiselmeier, G.; Föhlisch, B. *Tetrahedron Lett.* **2000**, *41*, 1375. (c) Zhurakovskyi, O.; Ellis, S. R.; Thompson, A. L.; Robertson, J. *Org. Lett.* **2017**, *19*, 2174. (d) Li, Y.; Dai, M. *Angew. Chem., Int. Ed.* **2017**, *56*, 11624. (e) Anagnostaki, E. E.; Zografos, A. L. *Org. Lett.* **2013**, *15*, 152. (f) Kusama, H.; Ishida, K.; Funami, H.; Iwasawa, N. *Angew. Chem., Int. Ed.* **2008**, *47*, 4903. (g) Wang, L. F.; Shi, Z. F.; Cao, X. P.; Li, B. S.; An, P. *Chem. Commun.* **2014**, *50*, 8061. (h) Ishida, K.; Kusama, H.; Iwasawa, N. *J. Am. Chem. Soc.* **2010**, *132*, 8842. (i) Appendino, G.; Jakupovic, J.; Cravotto, G.; Biavatti-Weber, M. *Tetrahedron* **1997**, *53*, 4681. (j) Murphy, G. K.; West, F. G. *Org. Lett.* **2005**, *7*, 1801. (k) Wartenberg, F.-H.; Hellendahl, B.; Blechert, S. *Synlett* **1993**, *1993*, 539. (l) Wartenberg, F.-H.; Junga, H.; Blechert, S. *Tetrahedron Lett.* **1993**, *34*, 5251.

(10) (a) Peng, W.; Lee, C.-S. *Synlett* **2008**, *2008*, 142. (b) Huang, S.; Du, G.; Lee, C. S. *J. Org. Chem.* **2011**, *76*, 6534. (c) Du, G.; Bao, W.; Huang, J.; Huang, S.; Yue, H.; Yang, W.; Zhu, L.; Liang, Z.; Lee, C. S. *Org. Lett.* **2015**, *17*, 2062. (d) Huang, J.; Bao, W.; Huang, S.; Yang, W.; Lizhi, Z.; Du, G.; Lee, C.-S. *Org. Lett.* **2018**, *20*, 7466.

(11) Chan, T. H.; Brownbridge, P. *J. Chem. Soc., Chem. Commun.* **1979**, 578.

(12) van der Waals, A.; Keese, R. *J. Chem. Soc., Chem. Commun.* **1992**, 570.

(13) The NMR spectra of all new compounds are available in the Supporting Information.

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