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Three-step synthesis of flavanostilbenes with a 2-cyclohepten-1-one core by Cu-mediated [5 + 2] cycloaddition/decarboxylation cascade

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ABSTRACT

The first synthesis of flavanostilbenes with a 2-cyclohepten-1-one core was carried out by applying an effective strategy in three steps from abundant polymerized flavanol resources. A key regio- and stereoselective Cu-mediated [5 + 2] cycloaddition/decarboxylation cascade was explored and applied without the use of protecting groups, and water as an environmentally friendly solvent contributed to the cascade. The intramolecular [5 + 2] cycloaddition mechanism, involving oxidation and dearomatization of the flavanol unit as a diene, was proposed and supported by the synthesis of the intermediate. The regioselectivity of the cyclization was found to be dependent on the substitution effects of the stilbene units by the exploration of substrate scope.

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As two important groups of polyphenolic compounds, flavanols and stilbenes are largely present in plants as monomers, oligomers and polymers [1–5]. They react as renewable and sustainable sources in the synthesis of polyphenol polymers with fused or bridged ring systems. Flavanostilbenes constitute a class of natural polyphenols with diverse scaffolds, and are formed by adduction of flavanols and polyhydroxystilbene units [6–8]. Recently, some flavanostilbenes have been isolated and identified, such as cajanusflavanols A–C [9], rhamnoneuronal D–N [10] and polygolflavanol A [11]. These flavanostilbenes exhibited potent biological activities, such as antioxidant, antidiabetic, antitumor and anti-inflammatory activities, and are therefore highly attractive targets for synthesis [10–13]. Jezonocinol C (**1**) is a flavanostilbene isolated from the bark of *Picea jezoensis* var. *jezoensis* by Tanaka and coworkers in 2007, and it features a unique 2-cycloheptenone subunit. It was shown to have radical-scavenging and antitumor initiating activities [14,15]. Since its isolation, further systemic biological activities *in vitro* and *in vivo* have remained ambiguous because of the scarcity of natural samples. The unprecedented 7/6/5/6 fused ring system inspired us to investigate the synthesis of this family of compounds.

Structurally, the 2-cyclohepten-1-one core and contiguous hexavalent carbon stereocenters are synthetic challenges [16,17]. Hence, a biomimetic strategy through a stereoselective route is highly desirable to generate **1** and its analogs. Tanaka and coworkers speculated that **1** is biosynthesized *via* the radical coupling of catechin and piceatannol, resulting in formation of the C6-C8″ and C10-C7″ bonds (Fig. 1a). Next, bridged ring opened, followed by decarboxylation to obtain **4** with a 2-cycloheptenone core and a second radical coupling between C4-C2″. However, given the common dimerization in polyphenol units, the two-step radical coupling is not a compelling pathway. Based on our earlier work on the synthesis of flavanostilbenes [18], the C4-C2′ bond was connected first. The cycloheptenone core of **1** reminded us of the potential that the cyclization possessed a C4-C2′ linkage and a [5 + 2] cycloaddition.

[5 + 2] Cycloadditions have been applied in the total synthesis of natural products with complex bridged ring systems [19–22]. Natural bridged-ring products have been successfully synthesized *via* this type of cycloaddition to date (*e.g.*, α-pipitzol, perezoperezone, and epicolactone) [23–27]. Hence, our proposed biosynthesis commences with nucleophilic substitution between C-4 of catechin and C-2″ of piceatannol to form intermediate **5** (Fig. 1b). Ring A in **5** undergoes intramolecular [5 + 2] cycloaddition with a piceatannol unit to provide **6**, followed by decarboxylation to afford **1**. Herein, flavanostilbenes with a cycloheptenone system were synthesized in only three steps without complex protection/deprotection of the phenolic hydroxy groups, and the reaction mechanism of [5 + 2] cycloaddition was clarified (Scheme 1).

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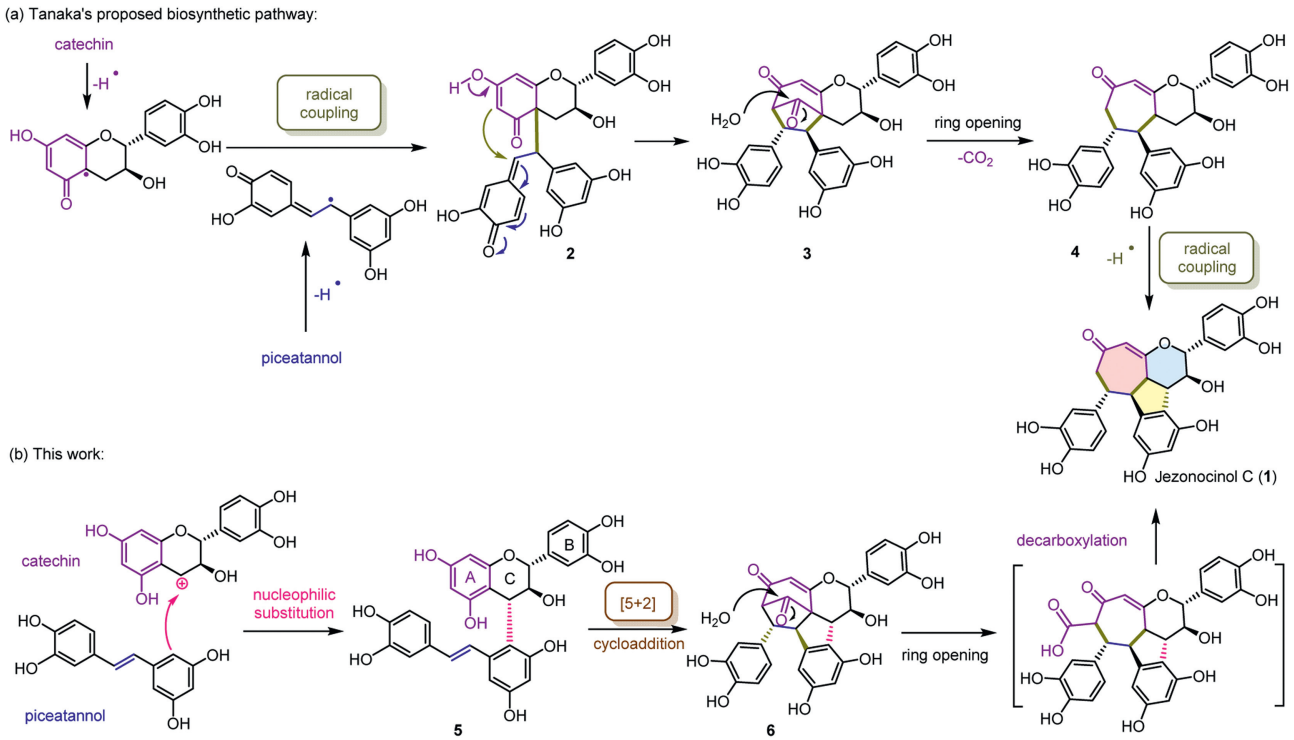
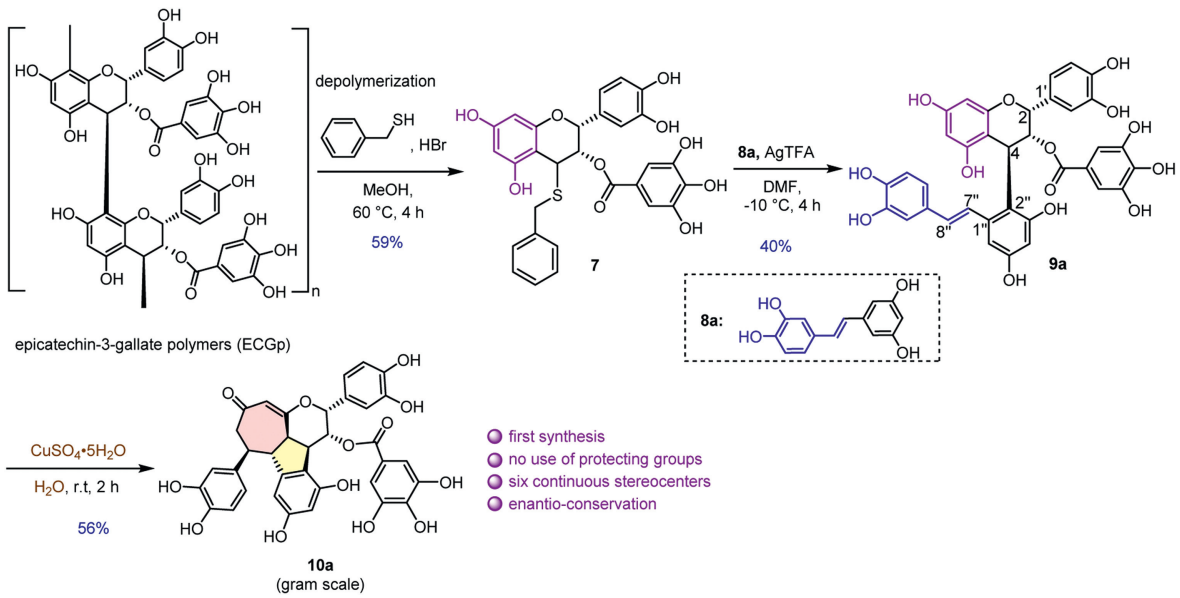


Fig. 1. (a) Tanaka's proposed biogenesis of **1**. (b) Our proposed biogenesis of **1**.



Scheme 1. Biomimmetic synthesis of flavanostilbenes **10a** with a 2-cyclohepten-1-one skeleton.

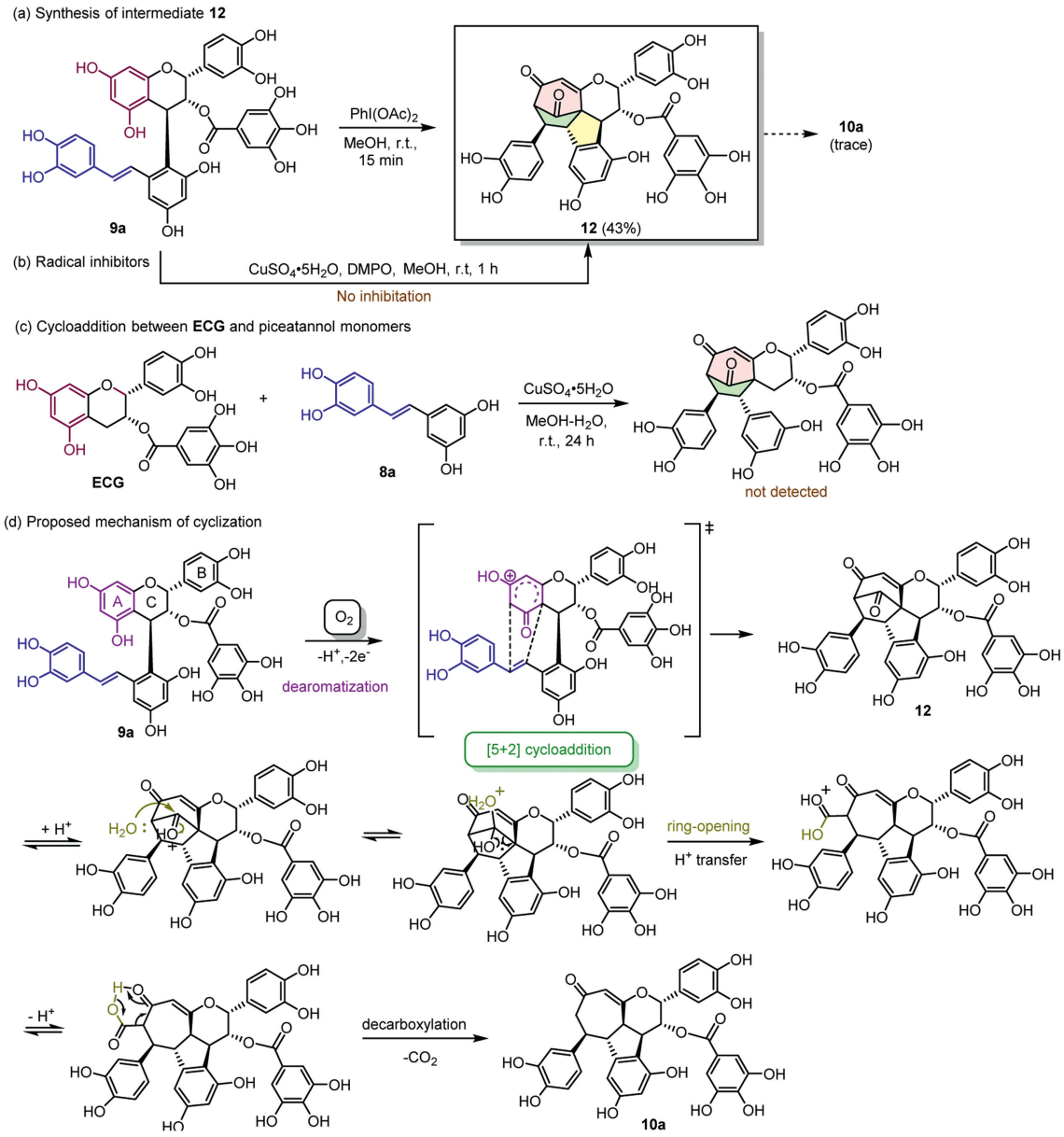
To promote atom economy, our synthetic strategy initially focused on the non-use of protecting groups. Considering that the biomimetic substrate of **1** is limited by the need for sufficient purified catechin units, we selected (-)-epicatechin-3-gallate (ECG) as a facile unit from natural sources [28,29]. We then investigated nucleophilic substitution to generate key intermediate **9a**. An abundance of ECG polymers (ECGp) was extracted from *Rhodiola crenulate* [30,31], and can be used as ECG substrates *via* depolymerization [32,33]. Benzyl mercaptan was employed to react with ECGp and HBr in MeOH [34], affording 4-benzylthio-substituted ECG monomers **7**. In our previous work, silver(I)-mediated stereoselective nucleophilic substitution of **7** at C-4 has been applied [18]. Delightfully, by direct treatment of **7** with piceatannol in the same condition of silver trifluoroacetate (AgTFA), the key intermediate **9a** was obtained in 40% yield, and the absolute configuration of C-4 was assigned as 4*R*, similar to our report [18].

With the successful synthesis of **9a**, its cyclization was attempted. In view of the application of Fe(III) catalysts in dimerization and their commercial availability, $FeCl_3 \cdot 6H_2O$ was tested first [35,36]. To our delight, despite the low yield, cycloheptenone product **10a** was separated as the main product in MeOH-H_2O (1:1, v/v) in 22 h (Table 1, entry 2). We then evaluated the ability of other transition metals to increase the yield. From this screening, it was found that $CuCl_2 \cdot 2H_2O$ (0.5 equiv.) as the additive in MeOH-H_2O

(1:1, v/v) afforded **10a** in 23% yield in 2 h (entry 3). The higher yield and shorter reaction time indicated that Cu(II) as the additive outperformed Fe(III) in this cyclization [37]. Subsequent tests focused on different types of solvents. Fortunately, water as a solvent contributed to the formation of **10a** in 56% yield (entry 4), and decomposition reaction occurred in nonaqueous solvents such as acetonitrile to afford monomers like piceatannol (entries 1, 6 and 7). Under the above optimized conditions, Fe(III) could increase the yield only up to 43% and required a longer time (entry 13). Some other transition-metal catalysts (*e.g.*, $Mn(OAc)_2$, $FeCl_2$, and CuBr) were also attempted, but no better promotion of the reaction was discovered (entries 8–12). Additionally, no significant improvement was observed by changing other reaction parameters, such as light, temperature, and concentration (Table S1 in Supporting information). With the optimal reaction conditions in hand, we achieved the biomimetic synthesis of **10a** on gram scale (Scheme 1).

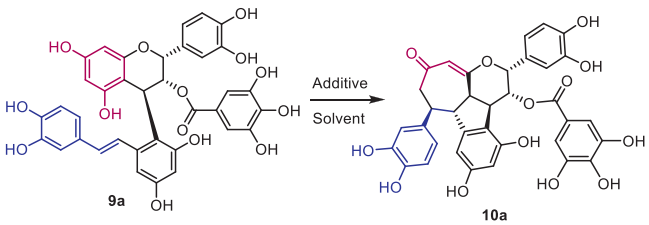
In Tanaka's work, the absolute configuration of **1** was not determined. In light of this, the remaining task was to verify the stereochemistry of the contiguous hexavalent carbons of **10a**. A rotating frame overhauser enhancement spectroscopy (ROESY) experiment was performed to determine the relative configuration of **10a**. The ROESY correlations of H-3/H-2, H-8″ and H-10/H-4, H-7″ revealed that H-2, H-3, and H-8″ were positioned on the same face. Given the correlations from H-10 to H-4 and H-7″ and the rigidity of the 5/6/7 fused-ring system, it was concluded that H-4, H-10, and H-7″ must all be in the same orientation. Therefore, together with the fact that the absolute configurations of C-2 and C-3 of (-)-epicatechins from natural products were 2*R* and 3*R*, the absolute configuration (2*R*, 3*R*, 4*R*, 10*R*, 7″*S*, 8″*R*) was unambiguously verified. The absolute configuration of **10a** was also confirmed by measurement of the electronic circular dichroism (ECD) spectrum and comparison with calculated ECD data by time-dependent density functional theory (TDDFT) performed at the CAM-B3LYP/6-31+g(d,p) level of theory (Fig. S1 in Supporting information).

Furthermore, the mechanism of cyclization of **9a** was investigated, and some control experiments were initially carried out. Interestingly, radical inhibitors such as 5,5-dimethyl-1-pyrroline *N*-oxide (DMPO) were first tested, but no inhibition was observed,



Scheme 2. Synthesis of intermediate **12** and mechanistic exploration of the cycloaddition.

Table 1
Optimization of the reaction conditions.[a]



Entry	Additive	Solvent	Time (h)	Yield (%)[b]
1	$FeCl_3·6H_2O$	MeOH	22	Trace
2	$FeCl_3·6H_2O$	MeOH-H_2O	22	16
3	$CuCl_2·2H_2O$	MeOH-H_2O	2	23
4	$CuCl_2·2H_2O$	H_2O	1	56
5	$CuSO_4·5H_2O$	H_2O	1	55
6	$CuCl_2$	MeCN	12	Decomp.
7	$CuCl_2$	THF	12	Decomp.
8	$FeCl_2$	H_2O	22	10
9	AgOAc	H_2O	42	27
10	$Mn(OAc)_2$	H_2O	22	Trace
11	$Ni(OAc)_2·4H_2O$	H_2O	22	Trace
12	CuBr	H_2O	22	Trace
13	$FeCl_3·6H_2O$[c]	H_2O	22	43

[a] Conditions: **9a** (1.0 equiv.), additive (0.5 equiv.), room temperature.
[b] Yields of isolated products.
[c] Conditions: **9a** (1.0 equiv.), additive (1.5 equiv.), room temperature.

precluding the possibility of a free radical mechanism (Scheme 2b and Table S3 in Supporting information) [38]. Notably, direct cycloaddition product **12** with a bicyclo[3.2.1]oct-3-ene-2,8-dione skeleton provided more convincing evidence. High-resolution mass spectrometry (HRMS) was successfully employed to characterize **12**, showing $[M+H]^+$ ions at *m/z* 685.1554. However, water-mediated fast decarboxylation at room temperature increased the difficulty of purification. To avoid decarboxylation, a hypervalent iodine reagent was utilized to replace the Fe(III) or Cu(II) additive

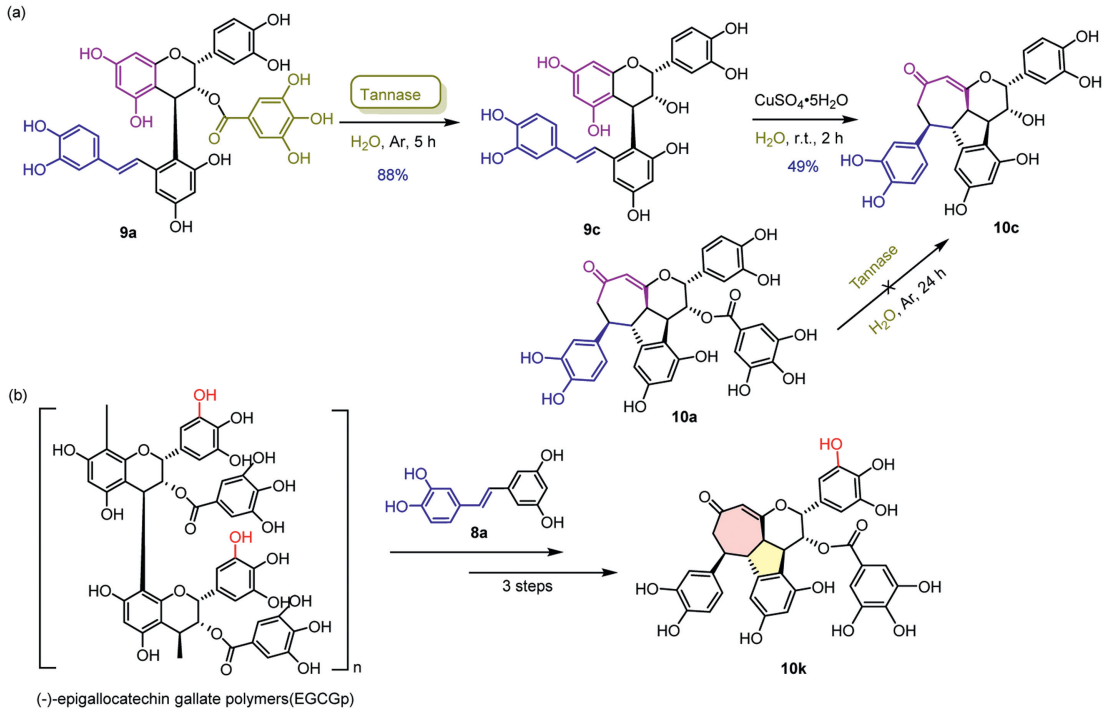
Table 2
Investigations of oxygen.[a]

Entry	Additive	Atmosphere	Time (h)	Yield (%)[b]
1	–	Ar	22	Trace
2	–	O_2	22	9
3	$CuSO_4·5H_2O$	Ar	22	Trace
4	–	Air	22	Trace
5	$CuSO_4·5H_2O$	Air	2	55

[a] Conditions: **9a** (0.1 mmol), additive (0.05 mmol), water (1 mL), room temperature.
[b] Yields of isolated products.

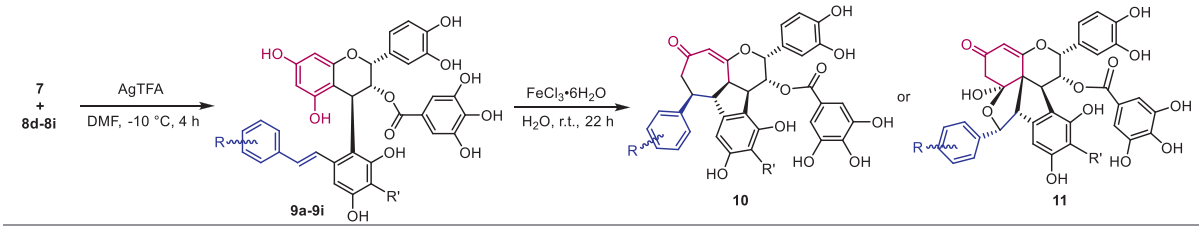
in a nonaqueous solvent [39]. Treating **9a** under the conditions of $PhI(OAc)_2$/MeOH produced **12** in 43% yield (Scheme 2a), and its structure was then determined by 1D and 2D NMR.

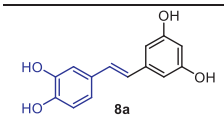
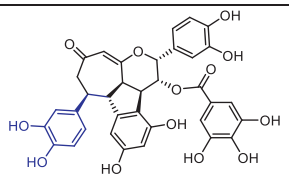
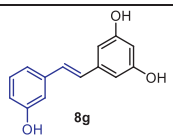
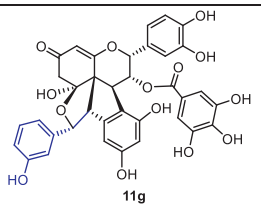
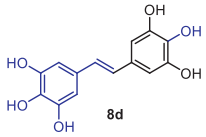
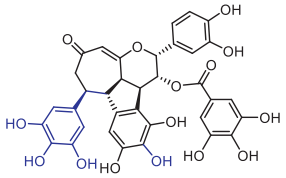
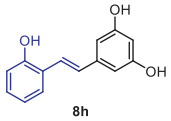
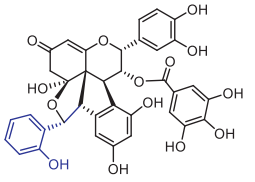
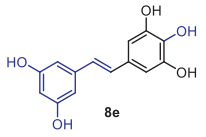
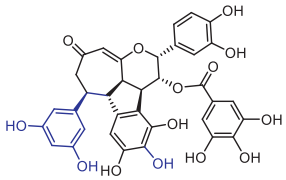
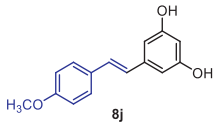
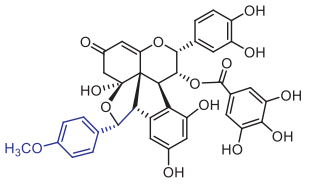
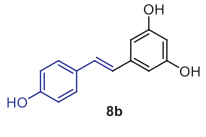
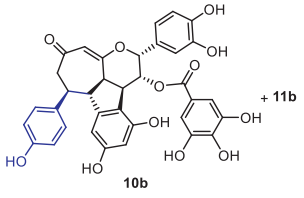
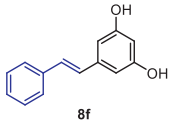
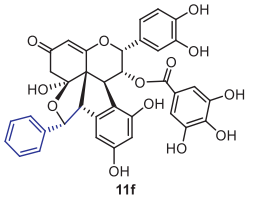
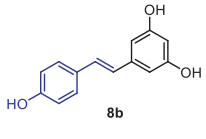
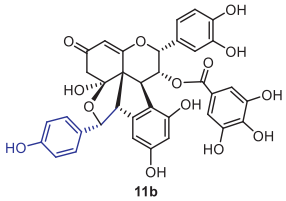
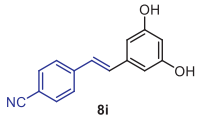
Subsequently, under the optimized conditions, we could not obtain **10a** in an oxygen-free atmosphere (Table 2, entry 3), and reaction in oxygen atmosphere afforded **10a** in 9% yield without the use of additive, indicating that oxidative dearomatization by oxygen was essential to cyclization and adding Cu(II) or Fe(III) accelerated the cycloaddition (Table 2, entry 2) [40,41]. Additionally, the use of Tris buffer (pH 7.5) and acetate buffer (pH 7.5) as weakly basic solvents led to the oxidation and decomposition of **9a**, eliminating the possibility of carbanion formation (Table S1 in Supporting information) [42]. When (-)-epicatechin gallate and piceatannol monomers were mixed, no [5 + 2] cycloaddition product was detected under the same conditions (Scheme 2c), indicating that the biosynthetic pathway of **10a** started with a connection between C4 and C2′ followed by cycloaddition. On the basis of the results of our control experiments, we proposed a mechanism of cyclization (Scheme 2d). The presence of oxidants leads to the dearomatization of ring A in the ECG units. Subsequently, dearomatized ring A undergoes intramolecular [5 + 2] cycloaddition with the double bond of the piceatannol unit. Finally, the unstable bridged-ring system of **12** undergoes ring-opening at C-10 by hydrolysis to relieve the steric hindrance, and decarboxylation of β-ketonic acid through a six-membered ring transition state with an intramolec-



Scheme 3. (a) Synthesis of a diastereoisomer of Jezonocinol C. (b) Synthesis of compound **10k**.

Table 3
Chemoselectivity and substrate scope.[a]



Stilbene	Product	Yield	Stilbene	Product	Yield
 8a	 10a	43%	 8g	 11g	30%
 8d	 10d	7% (two steps)	 8h	 11h	31%
 8e	 10e	11% (two steps)	 8j	 11j	35%
 8b	 + 11b 10b	**10b** (16%) **11b** (23%)	 8f	 11f	Trace
 8b	 11b	34%	 8i	No reaction	

[a] Conditions: $FeCl_3{\cdot}6H_2O$ (0.5 equiv.), H_2O, r.t., 22 h, isolated yield.
[b] Commercially available.
[c] Conditions: $FeCl_3{\cdot}6H_2O$ (0.5 equiv), H_2O, 40 °C, 22 h, isolated yield.

ular hydrogen bond gives **10a**. Water plays a key role in promoting the reversible [5 + 2] cycloaddition toward the final product and increasing the yield of product [43,44].

Confusingly, resveratrol as a substitution alternative for piceatannol afforded **11b** as the major product instead of **10b**. Structure of **11b** has been reported in our previous work and features a hexahydrocyclopenta[*c*]furan skeleton [18]. The yield of **10b** could be increased only to 16% by heating under Fe(III)-mediated conditions (Table 3). This discovery prompted us to investigate the substrate scope and explain the difference of chemoselectivity between **9b** and **9a** in intramolecular cyclization. Notably, Fe(III) contributed to the formation of both **10b** and **11b**, and thus it was more appropriate to reflect the competitiveness of the cyclization. Analogs of resveratrol substituted by electron-donating or electron-withdrawing groups were synthesized *via* Arbuzov and Wittig-Horner reactions followed by demethylation (see Supporting information for details) [45,46]. The connection of **7** with these stilbenes afforded **9d∼9j**, which cyclized to generate products with a hexahydrocyclopenta[*c*]furan or 2-cycloheptenone core (Table 3).

After analyzing the association between the selectivity and structures of the substrates, it was presumed that stilbenes substituted by more electron-donating groups tended to provide cycloheptenone products **10**, in which **9d** and **9e** equipped with five or six hydroxy–substituted stilbenes even cyclized directly in the presence of water (Table 3). Less production of cycloheptenone products **10** was observed with a decrease in electron-donating

groups. For instance, resveratrol-substituted intermediate **9b** could only cyclize to afford **10b** as a minor product in low yield, despite the change in the substitution position of the 4′-OH moiety. In addition, intermediate **9i**, substituted with electron-withdrawing groups, afforded neither **10i** nor **11i** under the same reaction conditions. The reactivity difference of [5 + 2] cycloaddition might be explained by frontier molecular orbital (FMO) theory. Oxidation and dearomatization of ring A led to the electron-deficient diene (delocalized 4-π-electron). The HOMO energy of the dienophile was raised through the π-conjugated framework from electron-donating groups such as hydroxyl groups, and their frontier orbitals were therefore closer in energy and able to interact [47,48].

Finally, compound **10c**, a diastereoisomer of **1**, was obtained *via* a concise strategy of tannase-catalyzed hydrolysis (Scheme 3a) [49–51]. Hydrolysis of intermediate **9a** with tannase in water in an Ar atmosphere provided **9c** in 88% yield. Cyclization of **9c** under standard Cu(II)-mediated conditions afforded **10c** in 49% yield. Direct hydrolysis of **10a** could not be carried out because of its poor water solubility. In addition, we found that (-)-epigallocatechin gallate polymers (EGCGp) isolated from *Rhodiola kirilowii* could also react as the source of flavans, and cycloheptenone products **10k** with was synthesized (Scheme 3b).

In conclusion, flavanostilbenes with a 2-cyclohepten-1-one core have been synthesized on gram scale for the first time through oxidative dearomatization followed by a Cu-mediated [5 + 2] cycloaddition/decarboxylation cascade. The first intramolecular [5 + 2] cycloaddition of the phloroglucinol unit as the diene was reported. The new synthesis route features contiguous hexavalent carbon stereocenters constructed in three steps under mild conditions without sophisticated protection/deprotection of the phenolic hydroxy groups. The significant intermediate of [5 + 2] cycloaddition **12** was synthesized, and the mechanism of cyclization was proven. Water as a green solvent provides better chemoselectivity, shorter reaction time and higher yield. In addition, investigation of the substrates demonstrated that the substitution of stilbenes also affected the regioselectivity of the cyclization reaction.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

CRediT authorship contribution statement

Gangsheng Li: Writing – review & editing, Writing – original draft, Validation, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Xiang Yuan:** Validation, Supervision, Methodology, Formal analysis, Data curation. **Fu Liu:** Validation, Investigation, Formal analysis, Conceptualization. **Zhihua Liu:** Validation, Supervision. **Xujie Wang:** Validation, Methodology. **Yuanyuan Liu:** Validation, Formal analysis. **Yanmin Chen:** Methodology. **Tingting Wang:** Formal analysis. **Yanan Yang:** Supervision, Project administration, Funding acquisition, Conceptualization. **Peicheng Zhang:** Supervision, Project administration, Funding acquisition, Conceptualization.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.cclet.2024.109880.

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