



## Bioinspired synthesis of cochlearol B and ganocin A

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### ABSTRACT

Described here is a divergent, biosynthetically inspired synthesis of cochlearol B and ganocin A. Key steps of the synthesis include the chromene unit construction through a biomimetic acid-catalyzed [4 + 2] ring cyclization. A photochemical [2 + 2] cycloaddition was featured to construct the cyclobutane core of cochlearol B. Different skeletal rearrangements of cochlearol B afforded ganocin A, that one of them was Lewis acid mediated epoxide rearrangement and another was DDQ induced cyclobutane formed tetrahydrofuran ring. The described syntheses not only achieved these natural products in an efficient manner, but also provided insight into the biosynthetic relationship between the two different skeletons.

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Recently, Cheng and Qiu reported the isolation of cochlearols A (**1**), B (**2**) and ganocins A-C (**3-5**) (Fig. 1) [1,2] from fungus ganoderma cochlear, a white rot fungus mainly distributed in tropical and subtropical areas of East Asia, which is used in traditional Chinese medicine for various diseases for centuries [3–6]. In addition to their unique motifs, these novel phenolic meroterpenoids containing multiple quaternary carbon centers and tetrasubstituted olefin fragments, have aroused the interest of both synthetic and pharmaceutical chemists owing to the synthetic challenges in their unique polycyclic skeleton structures, as well as the potential drug-gability in their potent anti-AChE activity and against chronic kidney disease activity [7–11]. Biological studies showed that (–)-cochlearol B (**2**) is a strong inhibitor of p-Smads, indicating renoprotective activities in TGF- $\beta$ 1 induced rat renal proximal tubular cells [1].

Biosynthetic studies have revealed ganocin A (**3**) that is derived from fornicin C (**6**) (Fig. 2A). In a forward manner, fornicin C (**6**), which bears a conjugated diene moiety, could undergo a hetero Diels-Alder reaction to yield chromene **7**. As a key intermediate in the biosynthesis of chromene **7** could further divert into various polycyclic compounds through free radical reactions [2]. In 1996, the Weyerstahl group reported that the italicene epoxides **9a** or **9b** rearranged with diluted HCl to the italicene ethers (epoxy-acorenes). They assume that due to additional ring strain in the tetracyclic epoxide **9**, fission of the cyclobutane ring takes place

and synchronously the oxirane is opened to give the intermediate **11**. Ring closure, which is simplified by the close neighborhood of the hydroxyl group and the double bond (as the Dreiding model and molecular modelling show), and elimination of water give the tetrahydrofuran **13** (Fig. 2B) [12]. Based on a concise intramolecular hetero-Diels-Alder reaction, Zhao and co-workers accomplished the divergent total synthesis of ganocins A-C in 2020 [13]. Later on, based on an oxidative cyclization and subsequent intramolecular [2 + 2] photo-cycloaddition strategy, Sugita's group accomplished a concise total synthesis of cochlearol B [14]. Schindler and co-workers achieved an enantioselective visible-light-mediated [2 + 2] photocyclo-addition closed the cyclobutane and formed the central bicyclo-[3.2.0]heptane core and then the asymmetric total synthesis of (+)-cochlearol B in the same year [15]. Recently, Hao and co-workers reported their bioinspired synthesis of cochlearol B and ganocins A-C [16].

Our long-standing interests in the biomimetic total synthesis of meroterpenoids led us to synthesize cochlearol B and ganocin A [17–19]. The proposed biosynthesis is summarised in Scheme 1, different with Qiu's point, that incorporates some changes as highlighted in red or blue. We envisioned that the tetrahydrofuran ring of ganocin A could be constructed by Lewis acid-mediated cyclization of tertiary alcohol **16** which derived from epoxide **17**. Epoxide **17** would be formed by epoxidation of cochlearol B. Cochlearol B was envisaged as arising from the photochemical [2 + 2] cycloaddition of 2*H*-chromene **19** and subsequent allylic oxidation [20–22]. 2*H*-Chromene **19** could be achieved through an intramolecular hetero-[4 + 2] Diels-Alder (IMDA) reaction involving

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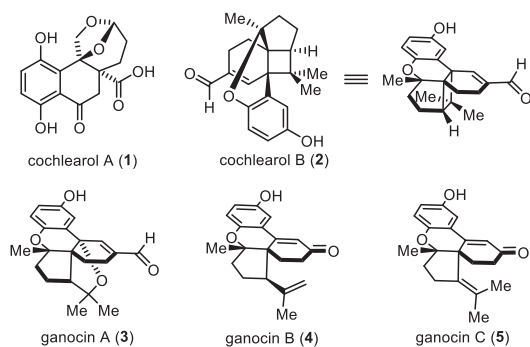


Fig. 1. Representative ganoderma meroterpenoids.

an *o*-QM intermediate **20** [23–29]. The latter would be assembled in a convergent manner from the two readily available building blocks. The intermediate **20** could be synthesized by the coupling between bromide **21** and nerolidol. Herein we report the details of our effort in realizing such a novel synthetic strategy to complete a concise and divergent total synthesis of cochlearol B (**2**) and ganocin A (**3**).

Our synthesis commenced with the Heck coupling of cheap and commercially available bromide **23** and nerolidol **22** (Scheme 2). Alcohol **24** could be rapidly established through palladium acetate catalysis with the reactant was treated at 100 °C for 2 h. With the intermediate **24** in hand, the stage was set for the key Lewis acid catalyzed hetero Diels–Alder reaction. As described in our previous work, unwanted benzopyran was formed as major product under strong acidic conditions and high temperatures [19]. To our delight, a high yield of **25** and its diastereomers (*d.r.* = 2:1) were observed when the reaction was done at low temperature with catalyzed amounts of TsOH to treat **24**. The screening of conditions proved that low temperature and weak acid were important to the hetero Diels–Alder reaction for construction of **25** [30]. Active property of the aryl allylic hydroxyl results in the instability of alcohol **24** at room temperature. During the purification of **24**, benzopyran byproduct was formed, reducing the overall yield of two-step reaction. Interestingly, by using one-pot method, that was adding equivalent trifluoroacetic acid to react with the crude product of **24**, key tricyclic product **25** could be obtained with a yield of 58% over 2 steps.

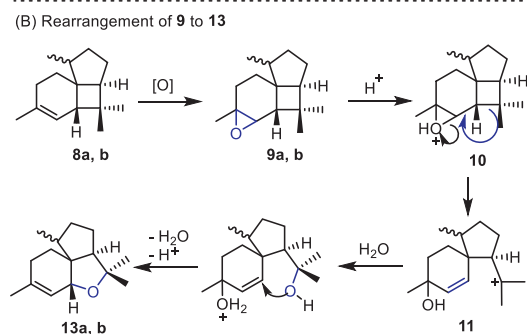
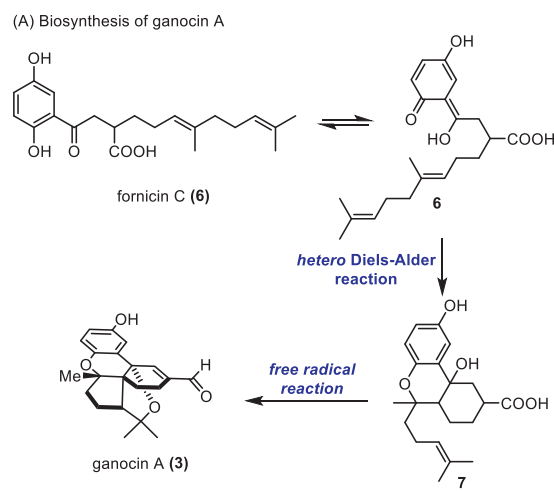
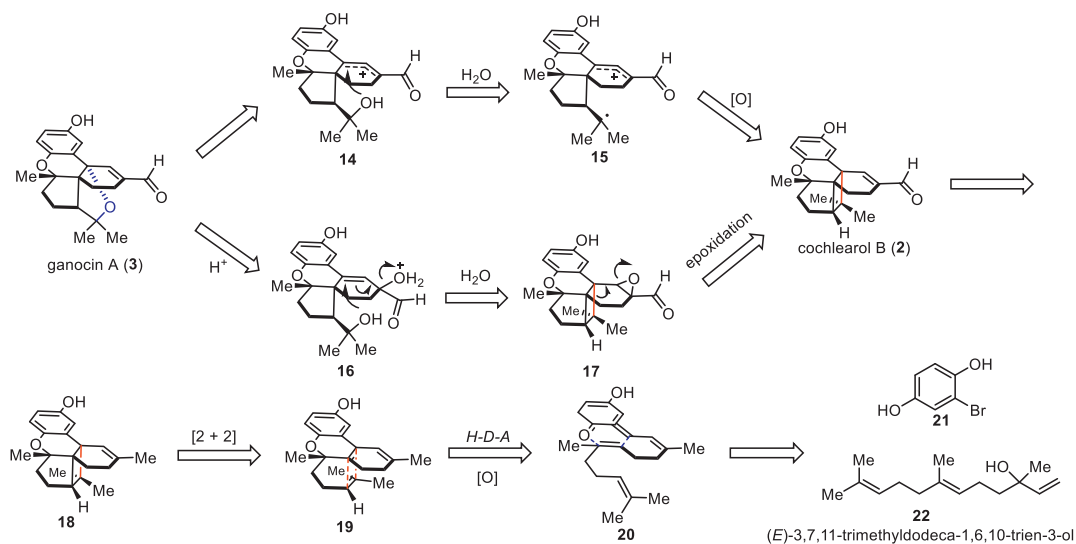
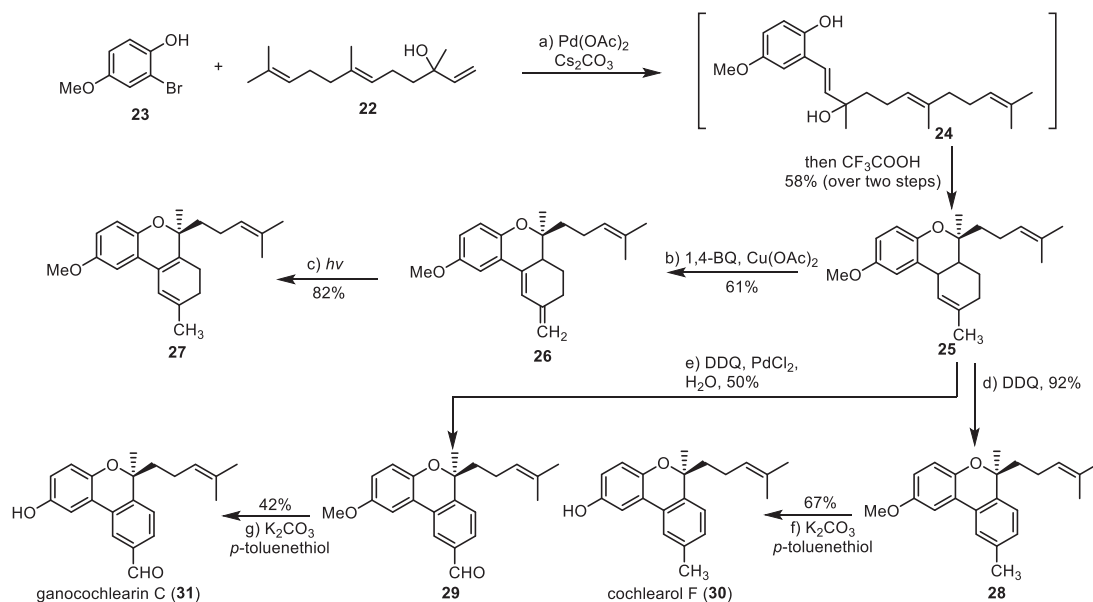


Fig. 2. (A) Biosynthesis of ganocin A. (B) Italicene epoxides **9a** or **9b** rearranged to ethers **13a** or **13b**.

With chromene **25** in hand, we tried selective dehydrogenation to provide *2H*-chromene **27**. In 2022, Loewinger and co-workers reported the preparation of dihydro-carmabinol by the oxidation of  $\Delta^9$ -tetrahydrocarmabinol with 3,5-di-*tert*-butyl-*o*-benzoquinone [31]. 2010, Fu and co-workers synthesized allyl aldehydes from allyl aromatics by Pd(II) catalysis and DDQ oxidation of allyl C-H [32]. Inspired by them, we screened different quinones to dehydrogenate **25**. It was observed that treatment of **25** with DDQ at room temperature provided only aromatized product **28**. Then



Scheme 1. Retrosynthetic analysis of cochlearol B and ganocin A.

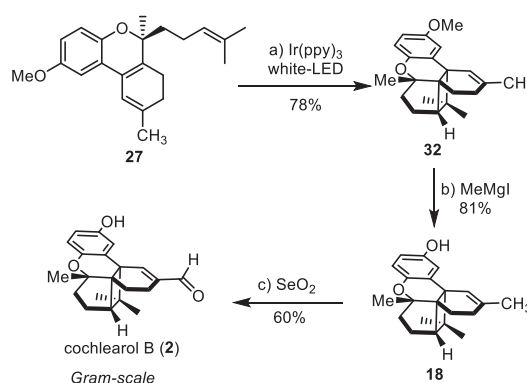


**Scheme 2.** Syntheses of **27**, cochlearol F (**30**) and ganocochlearin C (**31**). Reagents and conditions: (a) **23** (1.0 equiv.), nerolidol (1.3 equiv.), Pd(OAc)<sub>2</sub> (0.05 equiv.), Cs<sub>2</sub>CO<sub>3</sub> (2.5 equiv.), DMF, 100 °C, 2 h; then CF<sub>3</sub>COOH (2.0 equiv.), DCM, 0 °C, 1 h, 58% over two steps; (b) 1,4-BQ (2.5 equiv.), Cu(OAc)<sub>2</sub> (0.1 equiv.), DCE, 50 °C, 24 h, 61%; (c) Hg-lamp, DCM, r.t., 1 h, 82%; (d) DDQ (2.5 equiv.), DCM, r.t., 4 h, 92%; (e) DDQ (3.5 equiv.), PdCl<sub>2</sub> (0.1 equiv.), DCE, 80 °C, 2 h, 50%; (f) *p*-toluenethiol (2.0 equiv.), K<sub>2</sub>CO<sub>3</sub> (1.0 equiv.), DMF, 150 °C, 6 h, 67%; (g) *p*-toluenethiol (2.0 equiv.), K<sub>2</sub>CO<sub>3</sub> (1.0 equiv.), DMF, 160 °C, 8 h, 42%. DMF = *N,N*-dimethylformamide, DCM = dichloromethane, DCE = 1,2-dichloroethane, 1,4-BQ = 1,4-benzoquinone, DDQ = 2,3-dicyano-5,6-dichlorobenzoquinone.

**28** was successfully demethylated with *p*-toluenethiol and K<sub>2</sub>CO<sub>3</sub> in DMF at 150 °C, providing cochlearol F (**30**) in 67% yield. If palladium chloride was added to the above aromatization reaction and reacts at 80 °C, it could provide the product benzaldehyde **29**. We demethylated **29** with similar conditions, which get ganocochlearin C (**31**) was a moderate yield. The spectral properties of synthetic compounds **30** and **31** were consistent with those of natural products [33,34]. As described in the Supporting information, the chemical shift of cochlearol F C-5' ( $\delta_c$  136.9) was corrected by our work. Unfortunately, reducing DDQ and lowering the temperature does not provide 2*H*-chromene **27**. Given the strong oxidation of DDQ, we planned to use less oxidizing quinone as the dehydrogenation reagent. Olefin **26**, having a terminal double bond, was found after treatment of **25** with 1,4-benzoquinone at 50 °C. The catalytic amount of copper acetate can accelerate the completion of this reaction [35–39]. Inspired by Kalesse's total synthesis work at antroalobocin A, we were pleased to find that one hour of exposure to a high-pressure mercury lamp can provide a two-step 50% total yield of 2*H*-chromene **27** [40].

The stage was then set for the key steps, construction of the cyclobutane by intramolecular [2+2] photocycloaddition. Photocatalytic [2+2] reaction has been widely used in the synthesis of natural products [41–50]. When we added *fac*-tris(2-phenylpyridine)iridium as photocatalyst to the methanol solution of **27**, which can provide cyclobutene **32** after the irradiation of incandescent lamp (Scheme 3) [51]. At the same time, this reaction provided byproduct **33**, which was formed by Diels–Alder cycloaddition. However, efforts to optimize this transition were unable to overcome the formation of **33**, which has a formation rate of up to 11% (Table 1, entry 4). With **32** in hand, the following challenge was the demethylation. Compound **32** with a cyclobutane structure proved challenging through strong acid or nucleophilic demethylation conditions. We refer to the Schindler's method, Phenol **18** could be achieved by reacting with neat MeMgI [15,52]. Finally, cochlearol B (**2**) was successfully obtained by allylic oxidation of **18** using SeO<sub>2</sub> with a yield of 60%.

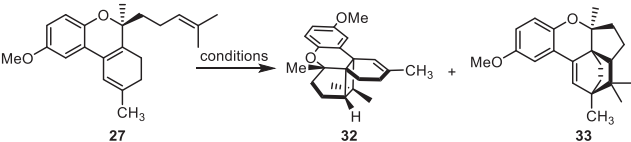
After completing the synthesis of cochlearol B, we turned our attention to ganocin A (**3**). In 2005, Kabuto reported an electron-



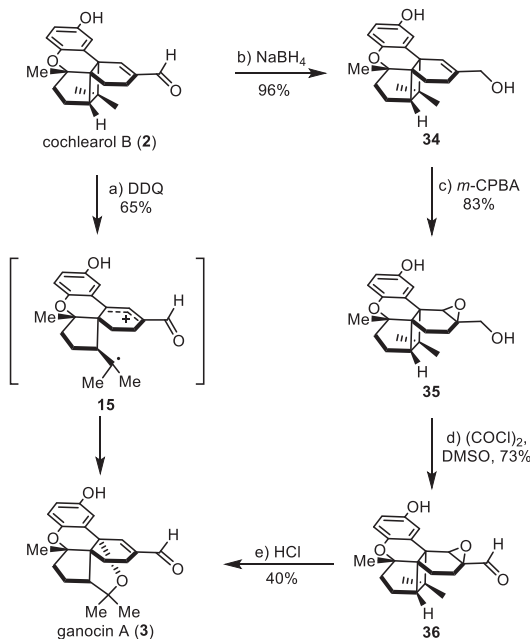
**Scheme 3.** Syntheses of cochlearol B (**2**). Reagents and conditions: (a) Ir(ppy)<sub>3</sub> (1% mol), white-LED, MeOH, r.t., 10 h, 78%; (b) MeMgI (20.0 equiv.), neat, 150 °C, 1 h, 81%; (c) SeO<sub>2</sub> (1.3 equiv.), 1,4-dioxane, reflux, 2 h, 60%. Ir(ppy)<sub>3</sub> = *fac*-Tris(2-phenylpyridine) iridium.

transfer reaction of 2,2-dianisyl-3,3-dimethyl-cyclobutanone in acetonitrile containing *p*-chloranil and water gave 2,2-dianisyl-5,5-dimethyldihydrofuran-3-one [53,54]. They speculated the reaction proceeded irreversibly via an oxatetramethylene-ethane radical cation derivative. Inspired by their work, we tried the electron-transfer reaction of cochlearol B (**2**) in different solvents and quinones (Scheme 4). Initial attempts to oxygenation rearrangement of cyclobutane cochlearol B (**2**) to generate tetrahydrofuran ganocin A (**3**) under previously reported *p*-chloranil and water conditions were unsuccessful. After an extensive screen of different quinones, we were pleased to find that treatment of cochlearol B with DDQ smoothly generated ganocin A with 38% yield (Table 2, entry 4) [55–57]. Different solvents and dosages optimization found that the desired ganocin A was obtained in 3.0 equiv. DDQ in MeCN/H<sub>2</sub>O mixed solution with 65% yield (Table 2, entry 5).

Next, we focused on the synthesis of ganocin A from rearrangement of epoxide **36** under Lewis acid. To our disappointment, the

**Table 1**  
Selective conditions for the [2 + 2] cycloaddition.<sup>a</sup>


Entry	Light	Catalyst	Solvent	Yield of (%) <sup>b</sup>	
				32	33
1	Hg-lamp	I <sup>c</sup>	<i>n</i> -Hexane	45	10
2	White-LED	I <sup>c</sup>	<i>n</i> -Hexane	44	12
3	Hg-lamp	II <sup>d</sup>	<i>n</i> -Hexane	70	13
4	White-LED	II <sup>d</sup>	MeOH	78	11
5	Blue-LED	II <sup>d</sup>	MeOH	20	35
6	White-LED	III <sup>e</sup>	MeOH	58	16
7	White-LED	IV <sup>f</sup>	MeOH	49	18
8	–	Fe(OTf) <sub>3</sub> <sup>g</sup>	MeOH	0	0

<sup>a</sup> All reactions were conducted at ambient temperature.<sup>b</sup> Reaction yield determined by <sup>1</sup>H NMR analysis of the crude reaction mixture.<sup>c</sup> Catalyst I: CAS 2030437-90-0, 1 mol%.<sup>d</sup> Catalyst II: *fac*-tris(2-phenyl-pyridine)iridium, CAS 94928-86-6, 1 mol%.<sup>e</sup> Catalyst III: [Ru(bpz)<sub>3</sub>][PF<sub>6</sub>]<sub>2</sub>, CAS 80907-56-8, 1 mol%.<sup>f</sup> Catalyst IV: Tris(2,2'-bipyridine)ruthenium dichloride, CAS 14323-06-9, 1 mol%.<sup>g</sup> 10 mol%.**Scheme 4.** Syntheses of ganocin A (**3**). Reagents and conditions: (a) DDQ (3.0 equiv.), MeCN/H<sub>2</sub>O (2:1), r.t., 1.5 d, 65%; (b) NaBH<sub>4</sub> (1.05 equiv.), MeOH, 0 °C, 40 min, 96%; (c) *m*-CPBA (1.3 equiv.), DCM, r.t., 2 h, 83%; (d) (COCl)<sub>2</sub> (1.1 equiv.), DMSO (2.2 equiv.), DCM, -70 °C, 40 min, then Et<sub>3</sub>N (5.0 equiv.), 20 min, 74%; (e) 1 mol/L HCl (aq.), MeOH, r.t., 8 h, 40%. *m*-CPBA = 3-chloroperoxybenzoic acid, DMSO = dimethyl sulfoxide.

epoxidation of **2** to afford **36** was not feasible in our preliminary experiments. What was complex but feasible was that following a reduction of the aldehyde with NaBH<sub>4</sub>, epoxide **35** was achieved upon treatment with *m*-CPBA, then swern oxidation completed **36** in 59% yield over 3 steps. Ganocin A was not observed when AlCl<sub>3</sub> or BF<sub>3</sub>·Et<sub>2</sub>O were added to an anhydrous solution of **36**, which proved that the rearrangement requires water [12,57]. Unlike the original design, some Lewis acids, just as TsOH, gave the desired product **3**, along with a considerable amount of side product [11]. The use of HCl instead of other acids in MeOH/H<sub>2</sub>O provided 40% yield and with much fewer side reactions (Scheme 4).

**Table 2**  
Reaction optimization of rearrangement for ganocin A.<sup>a</sup>

Entry	Quinone	Equiv.	Solvent	Yield of <b>3</b> (%)
1	<i>p</i> -Chloranil	3.0	MeCN/H <sub>2</sub> O	0
2	1,4-BQ	3.0	MeCN/H <sub>2</sub> O	0
3	DTBQ	3.0	MeCN/H <sub>2</sub> O	0
4	DDQ	1.0	MeCN/H <sub>2</sub> O	38
5	DDQ	3.0	MeCN/H <sub>2</sub> O	65
6	DDQ	3.0	MeOH/H <sub>2</sub> O	29
7	DDQ	3.0	Acetone/H <sub>2</sub> O	31

<sup>a</sup> All reactions were conducted at ambient temperature. 1,4-BQ = 1,4-benzoquinone, DTBQ = 3,5-di-*tert*-butyl-*o*-benzoquinone, DDQ = 2,3-dicyano-5,6-dichlorobenzoquinone.

In summary, we have developed a biomimetic synthesis route of cochlearol B in 7 steps (12% overall yield) from commercially available **22** and **23**. The key steps were intramolecular hetero Diels–Alder reaction to accomplish tricyclic structure, and [2 + 2] photocycloaddition for the construction of 4/5/6/6/6 skeleton. Cochlearol F and ganocochlearin C have also been synthesized *via* aromatization by making use of DDQ. Skeletal rearrangement of cochlearol B under oxidizing with DDQ or treating epoxide by Lewis acid was conducted generating unique 5/5/6/6/6 skeleton of ganocin A. Our work indicates the biosynthetic relationship, which ganocin A could be converted from cochlearol B.

### 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.

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## Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.ccl.2023.109247.

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