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JOURNAL OF THE AMERICAN CHEMICAL SOCIETY



Communication

# Domino Michael/Mannich/N-Alkylation Route to the Tetrahydrocarbazole Framework of Aspidosperma Alkaloids: Concise Total Syntheses of (-)-Aspidospermidine, (-)-Tabersonine, and (-)-Vincadifformine

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*J. Am. Chem. Soc.*, **Just Accepted Manuscript** • DOI: 10.1021/ja408114u • Publication Date (Web): 23 Aug 2013

# Domino Michael/Mannich/N-Alkylation Route to the Tetrahydrocarbazole Framework of Aspidosperma Alkaloids: Concise Total Syntheses of (–)-Aspidospermidine, (–)-Tabersonine, and (–)-Vincadifformine

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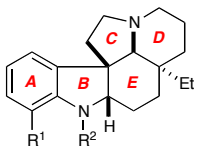
*Supporting Information Placeholder*

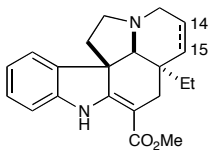
**ABSTRACT:** We report a novel, asymmetric domino Michael/Mannich/*N*-alkylation sequence for the rapid assembly of the tetrahydrocarbazole (ABE) framework of *Aspidosperma* alkaloids. This method was utilized in the concise total syntheses of classical targets (–)-aspidospermidine (**1b**), (–)-tabersonine (**2**), and (–)-vincadifformine (**3**) in 10–11 steps. Additional key steps include (1) ring-closing metathesis to prepare the D-ring and (2) the Bosch–Rubiralta spirocyclization to prepare the C-ring.

Monoterpene indole alkaloids of the *Aspidosperma* class, which include over 250 unique members, are fascinating natural products endowed with an irresistible combination of architectural complexity and pharmacological activity.[1] Moreover, these intriguing molecules have greatly benefited both organic chemistry and medicine. The structures of four classical members of the *Aspidosperma* family, namely (–)-aspidospermine (**1a**),[2] (–)-aspidospermidine (**1b**),[3] (–)-tabersonine (**2**),[4] and (–)-vincadifformine (**3**),[5] are shown in Figure 1. Of these, aspidospermidine (**1b**) is the most representative insofar as it possesses the hallmark ABCDE pentacyclic framework and common structural denominator amongst the *Aspidosperma* alkaloids. Accordingly, targets **1**-**4** have stimulated considerable interest in the synthetic community dating back to Stork's elegant, stereoselective total synthesis of **1a** in 1963 and continues unabated to this day.[2a]

**Figure 1.** Structures of (–)-aspidospermine (**1a**), (–)-aspidospermidine (**1b**), (–)-tabersonine (**2**), and (–)-vincadifformine (**3**).



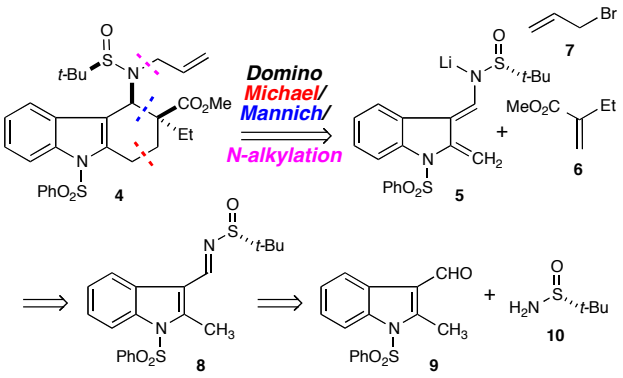

**1a:** (–)-aspidospermine ($R^1$=OMe; $R^2$=Ac)
**1b:** (–)-aspidospermidine ($R^1$=$R^2$=H)



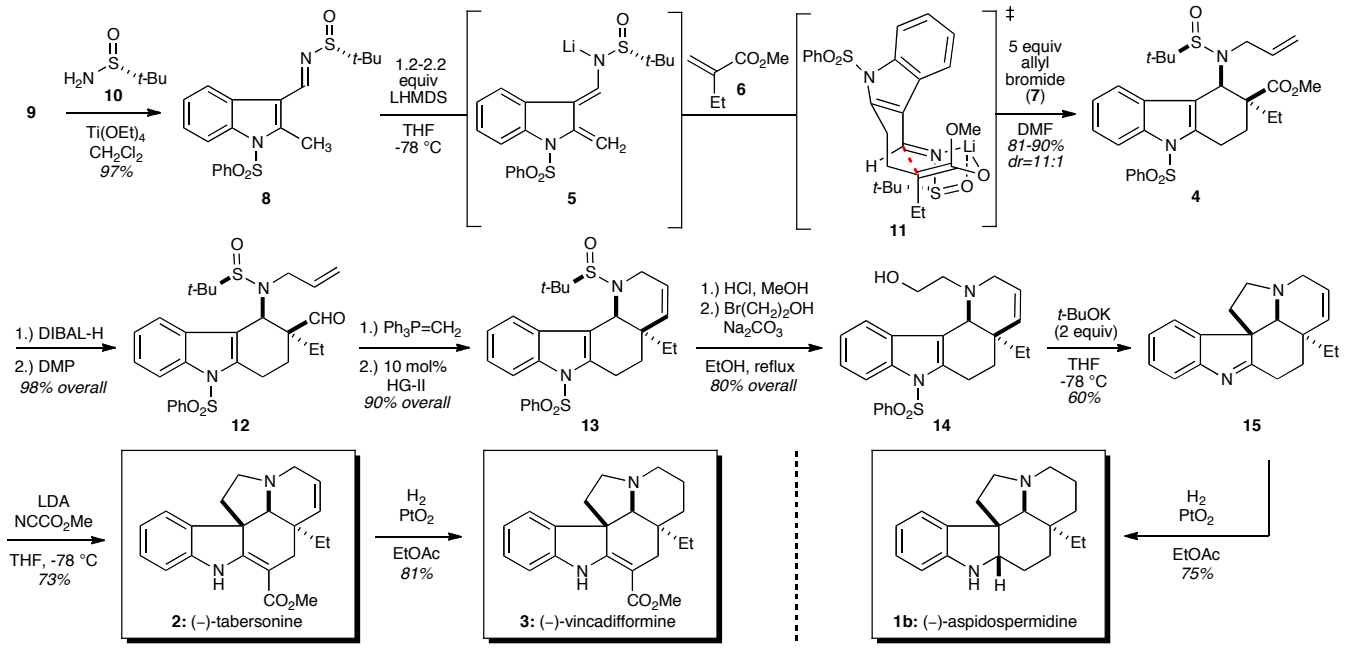

**2:** (–)-tabersonine, $\Delta^{14,15}$
**3:** (–)-vincadifformine

Although we have been engaged in the total synthesis of complex indole alkaloids of the *Strychnos* class[6] and most recently rearranged *Aspidosperma* alkaloids,[7] our methods were ill suited for preparing targets such as **1**-**4**. We were, nonetheless, intrigued by two disparate bodies of work whose merger, if successful, would offer facile, concise access to appropriately functionalized ABE tetrahydrocarbazole cores of **1**-**4** with satisfactory control over relative and absolute stereochemistry. Specifically, we were inspired by (1) Magnus's elegant and step-efficient indole-2,3-quinodimethane approach to *Aspidosperma* and *Kospia* alkaloids;[8,3h] and, (2) Ellman's clever use of metalloenamines derived from *tert*-butanesulfinylimines as asymmetric nucleophiles.[9] Retrosynthetically, we reasoned that tetrahydrocarbazole **4** could be obtained in one operation by means of a domino Michael/Mannich sequence (Scheme 1) from the reaction of azadienolate **5** and methyl ethacrylate (**6**).[10] The efficiency of the operation could be enhanced in the forward sense by productive trapping the *N*-sulfinylanion intermediate with allyl bromide (**7**). Azadienolate **5** would in turn be derived from *N*-sulfinylimine **8**, which was readily accessible from commercial **9** and (*R*)-*N*-*tert*-butanesulfinamide (**10**).[9]

**Scheme 1.** Retrosynthetic analysis of tetrahydrocarbazole **4** via novel Domino Michael/Mannich/*N*-alkylation route.



**Scheme 2.** Total syntheses of (–)-aspidospermidine (**1b**), (–)-tabersonine (**2**), and (–)-vincadifformine (**3**).




The synthesis began with the condensation of *N*-benezensulfonyl-2-methylindole-3-carboxaldehye (**9**), sulfinamide **10**, and $Ti(OEt)_4$ to afford *N*-sulfinylimine **8** in 97% yield (Scheme 2). Treatment of **8** with 1.2–2.2 equivalents of LHMDS in THF at -78 °C generated dienolate **5** via deprotonation of the acidic 2-methyl group. Addition of methyl ethacrylate (**6**)[11] triggered a Michael reaction whose enolate stereoselectively cyclized onto the regenerated *N*-sulfinylimine moiety in a Mannich fashion; trapping the resulting anion with 5 equivalents of allyl bromide in DMF furnished tetrahydrocarbazole **4** in 81-90% yield (dr=11:1).[12] We rationalized the relative and absolute stereocontrol in the domino Michael/Mannich sequence by means of transition state **11**, which is consistent with those posited by Ellman[13] and Davis.[14] To the best of our knowledge, this method represents the first (1) use of a vinylogue of an *N*-sulfinyl metalloenamide; (2) domino process wherein the *in situ*-generated nucleophile cyclizes onto an *N*-sulfinyl imine; and (3) intramolecular process wherein β-amino esters (i.e., Mannich bases) bearing α-quaternary stereocenters are prepared in both high yield and diastereoselectivity.

The next stage of the synthesis called for ring-closing metathesis (RCM) of the D-ring, a strategy employed by Rawal in the *Aspidosperma* series.[3ag,4j] To this end, we converted the methyl ester in **12** to a requisite terminal olefin via the intermediary aldehyde. This goal was best accomplished by sequential reduction to the alcohol with DIBAL-H and oxidation with the Dess–Martin periodinane (DMP) in 98% overall yield.[15] Wittig methylenation of **12** and ring-closing metathesis under the agency of 10 mol% Hoveyda–Grubbs 2nd generation catalyst (HG-II)[16] delivered ABDE tetracycle **13** in 90% overall yield.

We envisioned installing the C-ring with a step-efficient process discovered by Bosch and Rubiralta wherein an *N*-benzenesulfonyl protecting group on indole is transferred to an appropriately positioned primary hydroxyl group by the action of *t*-BuOK; spirocyclization of the ensuing indolyl anion at C3 with the benzenesulfonate ester establishes the C-ring.[17,18] Accordingly, removal of the *N*-sulfinyl group in **13** with HCl in MeOH and *N*-alkylation with 2-bromoethanol, $Na_2CO_3$ in refluxing EtOH gave substrate **14** in 80% overall yield. Addition of 2 equivalents of *t*-BuOK in THF at 0 °C effected the Bosch–Rubiralta spirocyclization to afford ABCDE pentacycle **15** in 60% yield.

Endgame for **1b**, **3**, and **4** commenced with indolenine **15**. Whereas previous reports had employed a two-step protocol (i.e., hydride reduction of the imine and metal-catalyzed hydrogenation of the D-ring olefin), we found hydrogenation of **15** over Adams's catalyst in EtOAc at rt delivered (–)-aspidospermidine (**1b**) in a single step (75% yield). The synthesis of **2** employed tactics first employed in Overman's elegant synthesis of classical *Strychnos* alkaloid akuammicine.[19] Specifically, treatment of **15** with LDA at -78 °C and quenching the intermediary azaenolate species with Mander's reagent[20] furnished (–)-tabersonine (**2**) in 73% yield. Hydrogenation of **2** over Adams's catalyst in EtOAc afforded (–)-vincadifformine (**3**) in 81% yield. Spectral data for **1a**, **2**, and **3** (e.g., $^1H$ and $^{13}C$ NMR, IR, optical rotation) were in complete agreement with those reported in the literature.[3,4,5]

In summary, we have completed concise, asymmetric total syntheses of classical *Aspidosperma* alkaloids (–)-aspidospermidine (**1b**, 10 steps, 27% overall yield), (–)-tabersonine (**2,** 10 steps, 26% overall yield), and (–)-vincadifformine (**3**, 11 steps, 22% overall yield) from commercial starting materials. Key steps include (1) a novel domino Michael/Mannich/*N*-alkylation sequence to access the tetrahydrocarbazole framework of the *Aspidosperma* alkaloids; (2) ring-closing metathesis to prepare the D-ring; and, (3) the Bosch–Rubiralta spirocyclization

to prepare the C-ring. We are currently exploring the scope of this novel process and applying it toward the total synthesis of other complex, bioactive alkaloids.

## ASSOCIATED CONTENT

**Supporting Information**. Experimental details and spectra. This material is available free of charge via the Internet at http://pubs.acs.org.

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### Notes

The authors declare no competing financial interest.

## ACKNOWLEDGMENT

This research was supported by the National Science Foundation (CHE-1111558). We thank Dr. Charles DeBrosse, Director of the NMR Facilities at Temple Chemistry, for kind assistance with NMR experiments. We thank Prof. Chris Schafmeister (Temple University) for access to LC-MS instrumentation. We thank Prof. David Dalton for carefully reading our manuscript, Dr. Richard Pederson (Materia, Inc.) for catalyst support, and the reviewers for helpful comments.

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