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Article

A Total Synthesis of (−)-Strychnine Using Photoredox Catalysis

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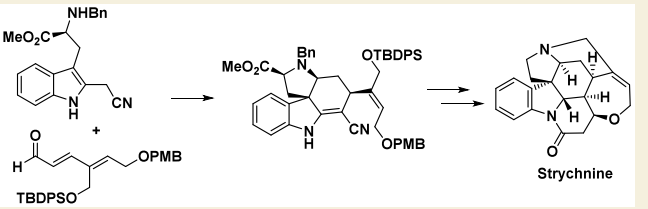
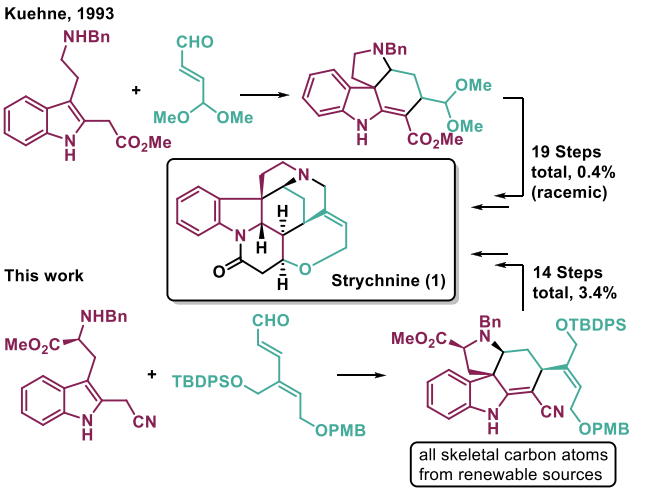
ABSTRACT: A concise route toward (−)-strychnine is presented. Key steps include the photoredox-catalytic C2-cyanomethylation of Boc-L-Trp-OMe and a condensation-electrocyclization cascade linking two fragments convergently to obtain an advanced intermediate. A photochemical decarboxylation provided convenient access to strychnofluorine, which could be transformed through the Wieland−Gumlich aldehyde to strychnine. All building blocks can be traced back to renewable sources.



KEYWORDS: *Alkaloids, Photoredox catalysis, Total synthesis, Cascade cyclization, Radical reactions*

Since its landmark total synthesis by Woodward in 1954,[1] strychnine (**1**) holds a special place in synthetic organic chemistry. Although applications of strychnine (**1**) in medicine, as a pharmacological tool compound or as a rodenticide, are rather limited at present,[2] the molecule still represents a formidable synthetic target. The highly compact polycyclic structure and numerous stereocenters of this natural product motivated numerous groups to propose and develop routes based on innovative approaches.[3−35] In more recent total syntheses of strychnine, the efficiency and practicality of novel methodologies have been evaluated by including them in the total synthesis of this complex molecule.[9,28,32,33,35] Inspired by the possibilities of photoredox chemistry and the option of cascade reactions that could construct several stereocenters in a single transformation, we devised an enantioselective route toward (−)-strychnine (**1**) based on these two key transformations. Retrosynthetically, the natural product was traced back to Wieland−Gumlich aldehyde (**2**), a frequently employed synthetic intermediate. The latter could be obtained from a 6−5−6−5 tetracyclic precursor **3** through functional group manipulations and the latter could be assembled from the two main precursors **4** and **5** through a condensation sigmatropic rearrangement cascade first employed by Kuehne in his 1993 strychnine synthesis (Scheme 1). The inclusion of a side chain containing all carbon atoms of the eastern half of **1** in the cascade cyclization should result in a reduced overall step count and in a more convergent strategy, since the elongation of the formyl group on C-15 into the C-18 to C-21 eastern unit is avoided. The indole building block **4** could be readily available from L-tryptophan (**8**) through photochemically induced C2 indole alkylation. We hypothesized that a nitrile could be a good precursor to the aldehyde functionality in **2**. The dienal **5** could be conveniently generated by the Heck reaction to vinyl iodide **6** available from butynediol **7** (Scheme 2).

Scheme 1. Previous Strategies for the Total Synthesis of Strychnine



To obtain indole building block **4**, the photoredox-catalyzed C2 cyanomethylation of Bn-L-Trp-OMe was attempted under various conditions using catalysts containing or devoid of transition metals.[36−40] The reactivity of the intermediate acetonitrile radical, paired with the nucleophilicity of the benzyl protected amine, resulted in side reactions, catalyst deactivation, and poor yields. We therefore utilized the Boc protected tryptophan methyl ester **9** and found that with

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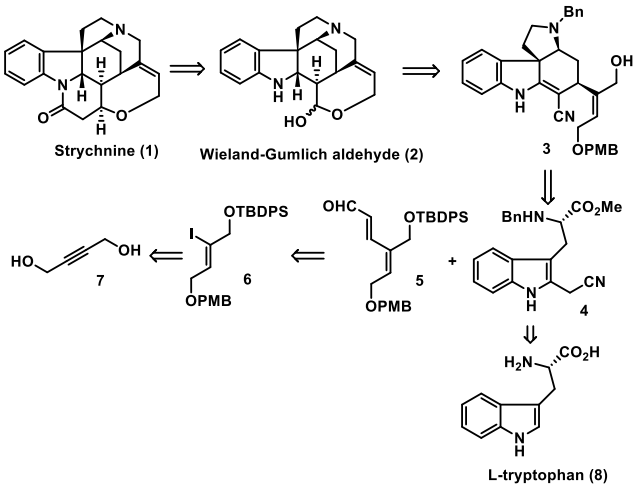
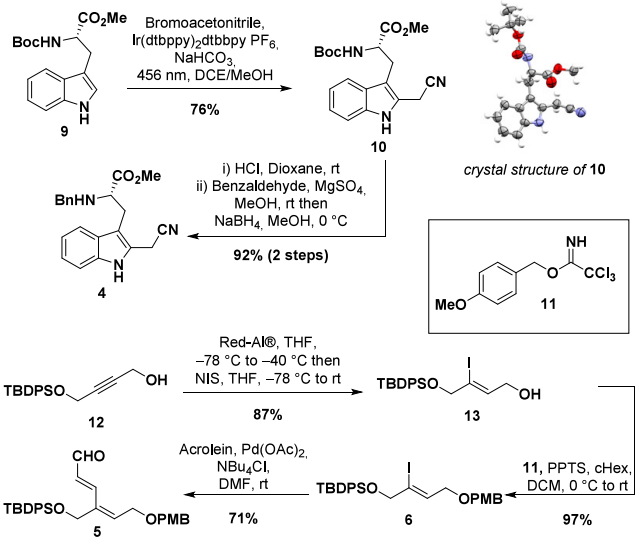
Scheme 2. Retrosynthetic Analysis of (−)-Strychnine (1)



Scheme 3. Preparation of the Indole and Unsaturated Aldehyde Building Blocks 4 and 5

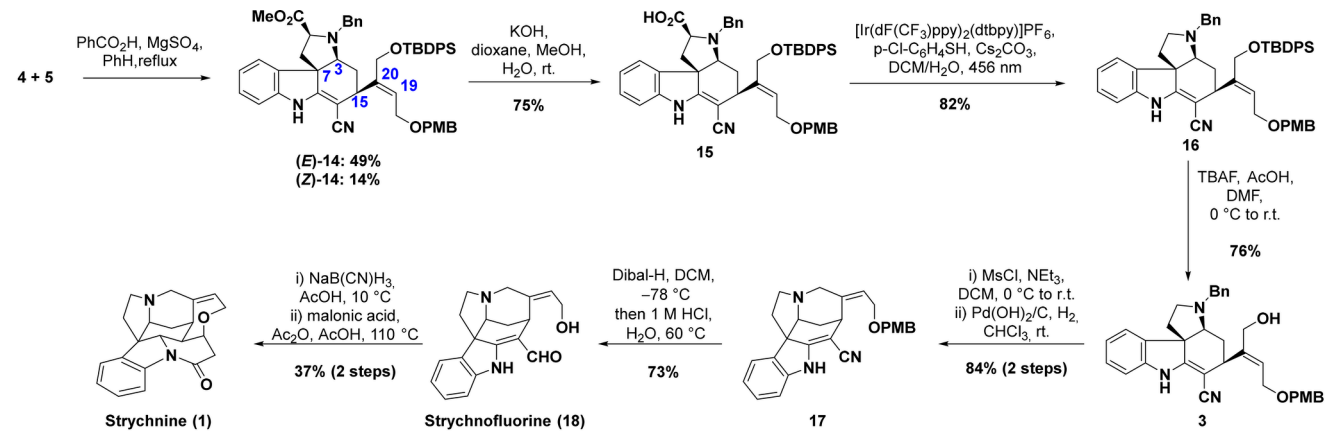


$[Ir(dtbppy)_2(dtbpy)]PF_6$ and bromoacetonitrile under blue light irradiation cyanomethylation proceeded smoothly and delivered compound **10** in 76% yield on multigram scale. The reaction was also scalable to decagram amounts in one batch and utilizes only 0.1% of the iridium-catalyst. Deprotection and reductive amination with benzaldehyde produced the first key component **4** in 92% yield (67% from L-tryptophan (**8**) without loss of stereochemical integrity).[41] For the second building block, TBDPS protected butynediol **12** was converted to the corresponding vinyl iodide **13** by treatment with Red-Al at cryogenic temperatures and subsequent trapping with NIS,[42,43] followed by PMB protection to **6** in 97% yield. The synthesis of the second building block was finalized by ligand free Heck reaction[44] of iodoolefin **6** with acrolein affording dienal **5** in 71% (Scheme 3).

With both building blocks **4** and **5** in hand, we utilized reaction conditions by Kuehne[11,45] to produce the tetracycle (*E*)-**14** in 49% yield and its double bond isomer (*Z*)-**14** in 14%, with full stereocontrol regarding positions 3, 7, and 15. Tetracycle (*E*)-**14** already comprises the full ABCE tetracyclic carbon framework and contains all necessary carbons to form the D- and F-rings of the final product. Attempts to further limit the production of undesired C19−C20 (*Z*)-isomer (*Z*)-**14** were unsuccessful. Based on control experiments, aldehyde **5** isomerizes to its (*Z*)-isomer by the addition of the indole through iminium formation and bond rotation. This process was more rapid than the subsequent cyclization cascade under all tested conditions, and the yield of (*Z*)-**14** reflects the efficiency of the chromatographic isolation rather than its formation in roughly equimolar amounts. Nevertheless, (*Z*)-**14** served as a perfect test substrate for further optimization studies (see the Supporting Information for details). The obtained conditions were transferred with minor changes to the desired (*E*)-isomer. Hydrolysis of the ester afforded acid **15** in 75% yield and photochemical decarboxylation under oxidative conditions gave compound **16** in 82% yield (Scheme 4).

In order to close the D-ring, desilylation, followed by activation with MsCl produced the quaternary ammonium salt, which was debenzylated utilizing $H_2/Pd(OH)_2$ in $CHCl_3$. The selection of solvent and reaction time control in this transformation was crucial, since polar solvents or increased reaction times led to double bond reduction or even allylic ether cleavage. Finally, conversion of enamino nitrile **17** to γ-amino aldehyde **18** was attempted. A sequence by Qin et al.[30]

Scheme 4. Completion of the Total Synthesis[a]



[a]Abbreviations: TBAF, tetrabutylammonium fluoride; Dibal-H, diisobutylaluminum hydride.

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which reduces the enamine and Pinner reaction could yield a known strychnine precursor. In our case, the reduction only gave unsatisfactory yields, and the Pinner conditions only resulted in PMB deprotection without conversion of the nitrile. Instead, reduction of the nitrile and acidic cleavage of the PMB protection group gave strychnofluorine (**18**) in 73% yield.

We also hypothesized that the hemiacetal formation during enamine reduction toward the Wieland−Gumlich aldehyde (**2**) could suppress formation of the undesired C-16 epimer. Strychnofluorine (**18**) was reduced with sodium cyanoborohydride in acetic acid, yielding the Wieland−Gumlich aldehyde, contaminated with the diol overreduction product, which was not purified and was instead directly converted to (−)-strychnine by the method of Robinson.[46]

Overall, we report a concise total synthesis of strychnine. Starting from L-tryptophan, 14 linear steps were required, and the overall yield amounted to 3.4%. All skeletal atoms of the final product can be traced back to xylochemicals or other renewable resources, including the fermentation product L-tryptophan: bromoacetonitrile (available from acetic acid),[47,48] butynediol (from biochar-derivable acetylene[49] and formaldehyde),[50] acrolein (glycerol dehydration),[51] and malonic acid (fermentation).[52] A combination of an optimized photochemical indole C2-cyanomethylation and Kuehne's cascade-Mannich electrocyclization with an advanced aldehyde building block reduced the overall step count of the synthesis, while the light-driven steps were superior to more classical approaches in terms of step count and yield. We hope this report will inspire further works in the field of alkaloid total synthesis.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/jacsau.5c01709.

Detailed experimental procedures, spectra, and X-ray data (PDF)

Accession Codes

Deposition Number 2481414 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via the joint Cambridge Crystallographic Data Centre (CCDC) and Fachinformationszentrum Karlsruhe Access Structures service.

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Author Contributions

#**Rainer Wiechert** and **Leander Geske** contributed equally. The manuscript was written through contributions of all authors./All authors have given approval to the final version of the manuscript. CRediT: **Rainer Wiechert** data curation, formal analysis, investigation, methodology, validation, writing - original draft; **Leander Geske** data curation, formal analysis, investigation, methodology; **Jasmin Hammes** formal analysis, investigation; **Dogus Tuncer** formal analysis, investigation; **Till Opatz** conceptualization, funding acquisition, methodology, project administration, resources, supervision, writing - original draft, writing - review & editing.

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Notes

The authors declare no competing financial interest.

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■ DEDICATION

Dedicated to Professor Albert (Al) Padwa, mentor and friend.

■ ABBREVIATIONS

DCE, 1,2-dichloroethane; rt, room temperature; Red-Al, sodium bis(2-methoxyethoxy)aluminum hydride; NIS, *N*-iodosuccinimide; PPTS, pyridinium 4-methylbenzenesulfonate; cHex, cyclohexane

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