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Microwave-Assisted Organocatalytic Synthesis of Tetrahydroquinolines *via* Hydride Transfer and Cyclization

You Kyung Kwon, Young Ku Kang, and Dae Young Kim*

*Department of Chemistry, Soonchunhyang University, Asan, Chungnam 336-745, Korea. *E-mail: dyoung@sch.ac.kr*
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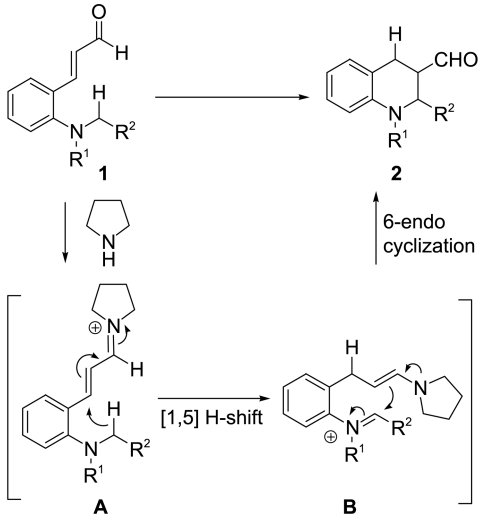
Tetrahydroquinoline derivatives have attracted considerable attention from organic and medicinal chemists primarily because they display a wide range of physiological activities.[1,2] Several derivatives have been found to elicit potent biological responses leading to analgesic, antiallergenic, anticonvulsant, and antifertility and NMDA antagonist activities.[3] Traditional methodologies for the formation of tetrahydroquinoline derivatives mainly include the Povarov reaction[4] and reduction of quinolines.[5] Recently, we have reported the synthesis of tetrahydroquinolines through organocatalytic C-H bond functionalization *via* tandem 1,5-hydride transfer and ring closure.[6] Despite significant advances in this field, one of the critical shortcomings of many organocatalysis reactions reported to date include long reaction times.

Over the past two decades, the application of microwave irradiation towards the acceleration of a wide range of organic and inorganic reactions, has received considerable attention.[7] The main benefit of performing reactions under microwave (MW) irradiation condition is the significant rate-enhancements, cleaner reaction profiles, and the higher yields that can frequently be observed. Recently, microwave-assisted synthesis of pyrido-fused heterocycles applying the *tert*-amino effect was reported.[8] To the best of our knowledge, there are no examples of microwave-assisted organocatalytic synthesis of tetrahydroquinolines *via* hydride transfer and cyclization.

As part of the research program related to the development of synthetic methods for the catalytic carbon-carbon bond formations,[9] we recently reported the organocatalytic conjugate addition reaction to α,β-unsaturated carbonyl compounds[10] and the other Michael acceptors.[11] Herein, we wish to describe the organocatalytic synthesis of tetrahydroquinoline derivatives *via* 1,5-hydride transfer/ring closure sequences under microwave (MW) irradiation condition. We have reported the use of organocatalysts for the transformation of **1** to **2** *via* imminium ion **A** and enamine **B** (Scheme 1).[6]

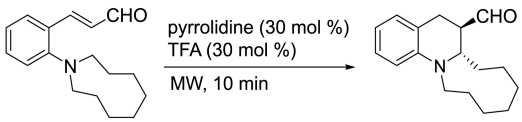
To determine suitable reaction conditions for the organocatalytic intramolecular redox reaction, we initially investigated reaction of *o*-(*N*-azonanyl) cinnamaldehyde **1a** catalyzed by pyrrolidine (30 mol %) and TFA (30 mol %) in various solvents under constant power of MW. The results of



Scheme 1. Organocatalytic intramolecular redox reaction.

representative selection of microwave-assisted intramolecular redox reaction are summarized in Table 1. Concerning the solvent, the use of acetonitrile gave the best results, whereas the use of 1,2-dichloroethane, toluene, and ethanol

Table 1. Optimization of reaction conditions

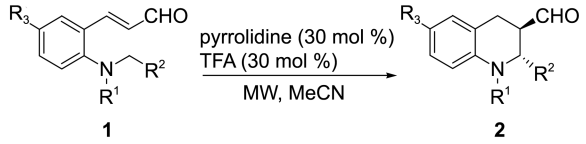


1a 2a

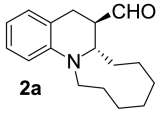
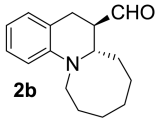
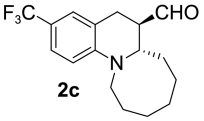
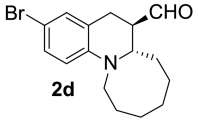
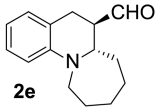
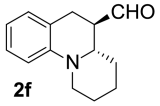
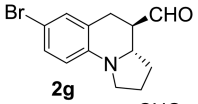
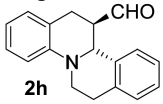
Entry	Solvent	Power (W)[a]	Yield (%)[b]	dr (%)[c]
1	DCE	200	25	64:36
2	PhMe	200	30	69:31
3	EtOH	200	42	67:33
4	MeCN	200	92	62:38
5	MeCN	150	92	70:30
6	MeCN	100	92	72:28
7	MeCN	50	93	77:23
8	MeCN	30	75	75:25

[a]Application of constant power. [b]Combined yield of both diastereomers. [c]Diastereomeric ratio is determined by ^{1}H NMR spectroscopic analysis.

Table 2. Microwave-assisted organocatalytic synthesis of tetrahydroquinolines



1 **2**

Entry	Power (W)[a]	Time (min)	Product, **2**	Yield (%)[b]	dr (%)[c]
1	50	10	 **2a**	93	77:23
2	50	10	 **2b**	95	75:25
3	50	15	 **2c**	90	75:25
4	50	10	 **2d**	93	77:23
5	50	30	 **2e**	95	88:12
6	200	120	 **2f**	78	78:22
7	200	150	 **2g**	72	78:22
8	50	20	 **2h**	97	57:43

[a]Application of constant power. [b]Combined yield of both diastereomers. [c]Diastereomeric ratio is determined by ^{1}H NMR spectroscopic analysis.

led to lower yields (Table 1, entries 1-4). The present catalytic system tolerates MW power loading down to 50 W without compromising both yield and diastereoselectivity (Table 1, entries 4-8).

To examine the generality of this protocol, we have examined several *o*-dialkylamino-substituted cinnamaldehydes **1a-1h** to confirm the efficiency of MW activation for this intramolecular tandem reaction *via* 1,5-hydride transfer and 6-endo cyclization. The corresponding cinnamaldehyde **1a-1e** containing seven- to nine-membered amine and tetrahydroisoquiniline donors readily afford to the corresponding products with high yields with 30 min under MW power of 50 W (Table 2, entries 1-5 and 8). Pyrrolidine- and piperidine-substituted cinnamaldehydes **1f-1g** were also converted to the corresponding products **2f-2g** under MW power of 200 W (Table 2, entries 6-8). The relative configuration of major diastereomer of **2** was established by comparison of the ^{1}H-NMR spectral data with previously reported data.[6]

In conclusion, we have developed microwave-assisted organocatalytic synthesis of tetrahydroquinolines *via* hydride transfer and cyclization. The synthetically useful ring-fused tetrahydroquinoline derivatives were obtained in high yields with short reaction time. Further investigations for organocatalytic hydride transfer are ongoing and will be reported in due course.

Experimental Section

General. All commercial reagents and solvents were used without purification. TLC analyses were carried out on precoated silica gel plates with F_{254} indicator. Visualization was accomplished by UV light (254 nm), I_2, *p*-anisaldehyde, ninhydrin, and phosphomolybdic acid solution as an indicator. Purification of reaction products was carried out by flash chromatography using E. Merck silica gel 60 (230-400 mesh). ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker AC 200 (200 MHz for ^{1}H, 50 MHz for ^{13}C). Chemical shift values (d) are reported in ppm relative to Me_4Si (δ 0.0 ppm). Microwave reactions were performed on CEM discovery microwave.

Typical Procedure. To a stirred solution of (*E*)-3-(2-(azonan-1-yl)cinnamaldehyde (**1a**, 77.2 mg, 0.3 mmol) and TFA (6.9 μL 0.09 mmol) in acetonitrile (3 mL) was added pyrrolidine (7.5 μL, 0.09 mmol) into a 10 mL flask equipped with a condenser. The reaction mixture was placed in the microwave cavity and exposed to microwave irradiation for 10 min at 80 °C using irradiation power of 50 W. On cooling to room temperature, the mixture was diluted with EtOAc, washed with water. The organic layer was dried over anhydrous $MgSO_4$, filtered, concentrated, and purified by flash column chromatography to give the desired product (**2a**, 93%, 71.8 mg).

5,6,6a,7,8,9,10,11,12,13-Decahydroazonino[1,2-a]quinoline-6-carbaldehyde (2a): 77:23 diastereomeric mixture. ^{1}H NMR (200 MHz, $CDCl_3$) δ 9.83 (s, 1H), 9.52 (s, 0.3H), 7.16-7.05 (m, 2.6H), 6.81-6.60 (m, 2.6H), 3.87-3.61 (m, 2.6H), 3.28-3.05 (m, 2.6H), 2.99-2.81 (m, 1.3H), 2.73-2.62 (m, 1H), 2.58-2.52 (m, 0.3H), 1.91-1.38 (m, 15.6H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 203.3, 203.1, 144.8 (x 2), 129.6, 129.4, 127.3 (x 2), 118.1, 116.9, 116.2 (x 2), 113.4 (x 2). 59.8, 58.7, 56.8, 56.7, 48.9, 47.9, 32.9, 28.7, 28.1, 27.5, 26.9, 26.6, 26.3, 26.1, 25.3, 25.1, 25.0, 24.6, 23.9, 22.5; ESI-HRMS: *m/z* calcd for $C_{17}H_{24}NO$ $[M+H]^+$: 258.1861; found 258.1858.

6,6a,7,8,9,10,11,12-Octahydro-5*H*-azocino[1,2-a]quinoline-6-carbaldehyde (2b): 75:25 diastereomeric mixture. ^{1}H NMR (200 MHz, $CDCl_3$) δ 9.82 (s, 0.3H), 9.50 (s, 1H), 7.24-7.02 (m, 2.6H), 6.74-6.53 (m, 2.6H), 3.86-3.75 (m, 2.6H), 3.28-3.13 (m, 1.3H), 3.08-3.05 (m, 2H), 2.98-2.81 (m, 0.6H), 2.82-2.75 (m, 0.3H), 2.55-2.49 (m, 1H), 2.00-1.20 (m, 13H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 203.1, 202.6, 143.8, 143.6, 129.6, 129.4, 127.4, 126.9, 118.5, 117.3, 115.5, 115.4, 111.5, 111.2, 58.3, 58.0, 53.3, 53.0, 48.9, 48.5, 33.7,

30.6, 27.7 (x 2), 26.8, 26.4, 26.1, 25.9 (x 2), 24.0, 23.3; ESI-HRMS: *m/z* calcd for $C_{16}H_{22}NO$ $[M+H]^+$: 244.1701.

3-(Trifluoromethyl)-6,6a,7,8,9,10,11,12-octahydro-5*H*-azocino[1,2-a]quinoline-6-carbaldehyde (2c): 75:25 diastereomeric mixture. ^{1}H NMR (200 MHz, $CDCl_3$) δ 9.83 (s, 0.3H), 9.50 (s, 1H), 7.34-7.26 (m, 2.6H), 6.65-6.60 (m, 0.3H), 6.58-6.53 (m, 1H), 4.00-3.80 (m, 2H), 3.80-3.73 (m, 0.6H), 3.33-3.20 (m, 1.6H), 3.18-3.05 (m, 2H), 3.04-2.95 (m, 0.6H), 2.80-2.70 (m, 0.3H), 2.62-2.56 (m, 1H), 2.00-1.25 (m, 13H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 202.0, 201.7, 146.2 (x 2), 126.4 (q, $J_{C\text{-}F}$ = 3.0 Hz) (x 2), 125.0 (q, $J_{C\text{-}F}$ = 268.2 Hz) (x 2), 124.6 (q, $J_{C\text{-}F}$ = 2.7 Hz) (x 2), 118.4, 117.1, 117.2 (q. $J_{C\text{-}F}$ = 32.4 Hz) (x 2), 110.8, 110.6, 58.5 (x 2), 53.8, 53.1, 48.6, 48.3, 33.9, 30.1, 27.6 (x 2), 26.3, (x 2) 25.9 (x 2), 25.6 (x 2), 23.7, 23.2; ESI-HRMS: *m/z* calcd for $C_{17}H_{21}F_3NO$ $[M+H]^+$: 312.1575; found 312.1571.

3-Bromo-6,6a,7,8,9,10,11,12-octahydro-5*H*-azocino[1,2-a]quinoline-6-carbaldehyde (2d): 77:23 diastereomeric mixture. ^{1}H NMR (200 MHz, $CDCl_3$) δ 9.81 (s, 0.3H), 9.49 (s, 1H), 7.22-7.09 (m, 2.6H), 65.7-6.49 (m, 0.3H), 64.6-6.39 (m, 1H), 3.93-3.79 (m, 2H), 3.78-3.65 (m, 0.6H), 3.27-3.14 (m, 1.3H), 3.07-3.05 (m, 2H), 2.98-2.81 (m, 0.6H), 2.82-2.75 (m, 0.3H), 2.56-2.51 (m, 1H), 2.00-1.20 (m, 13H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 202.3, 202.0, 142.7 (x 2), 131.9, 131.7, 130.0 (x 2), 120.7, 119.4, 113.0, 112.7, 107.0 (x 2), 58.2, 57.9, 53.3, 53.0, 48.6, 48.5, 33.7, 29.9, 27.6 (x 2), 26.4, 26.2, 25.9 (x 4), 23.6, 23.1; ESI-HRMS: *m/z* calcd for $C_{16}H_{21}BrNO$ $[M+H]^+$: 322.0807; found 322.0807.

5,6,6a,7,8,9,10,11-Octahydroazepino[1,2-a]quinoline-6-carbaldehyde (2e): Major diastereoisomer. ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.53 (s, 1H), 7.06-7.01 (m, 2H), 6.53 (t , J = 8.5 Hz, 1H), 6.49 (d, J = 8.5 Hz, 1H), 3.85 (dd, J = 6.0 Hz, 3.0 Hz, 1H), 3.82-3.79 (m, 1H), 3.22-3.09 (m, 2H), 3.02 (dd, J = 8.0 Hz, 6.5 Hz, 1 H), 2.54-2.52 (m, 1H), 2.09-2.05 (m, 1H), 1.74-1.71 (m, 1H), 1.67-1.57 (m, 5H), 1.37-1.34 (m, 1H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 203.3, 143.8, 129.4, 127.4, 117.0, 115.6, 110.3, 58.2, 49.5, 47.8, 34.9, 26.5, 26.0, 25.8, 23.7; ESI-HRMS: *m/z* calcd for $C_{15}H_{20}NO$ $[M+H]^+$: 230.1545; found 230.1541.

2,3,4,4a,5,6-Hexahydro-1*H*-pyrido[1,2-a]quinoline-5-carbaldehyde (2f): Major diastereoisomer. ^{1}H NMR (200 MHz, $CDCl_3$) δ 9.63 (s, 1H), 7.21-7.00 (m, 2H), 6.78-6.57 (m, 2H), 4.01-3.80 (m, 1H), 3.50-3.42 (m, 1H), 3.19-2.50 (m, 3H), 2.64-2.55 (m, 1H), 2.00-1.30 (m, 6H), ^{13}C NMR (50 MHz, $CDCl_3$) δ 202.6, 143.9, 128.8, 127.6, 122.0, 117.5, 112.5, 56.4, 51.9, 48.3, 31.2, 25.9, 24.9, 24.0; ESI-HRMS: *m/z* calcd for $C_{14}H_{18}NO$ $[M+H]^+$: 216.1388; found 216.1393.

7-Bromo-1,2,3,3a,4,5-hexahydropyrrolo[1,2-a]quinoline-4-carbaldehyde (2g): Major diastereoisomer. ^{1}H NMR (200 MHz, $CDCl_3$) δ 9.90 (d, J = 1.8 Hz, 1H), 7.20-7.14 (m, 2H), 6.33-6.29 (m, 1H), 3.49-3.30 (m, 2H), 3.23-3.10 (m, 1H), 2.90 (d, J = 8.3 Hz, 2H), 2.43-2.31 (m, 2H), 2.30-1.96 (m, 2H), 1.70-1.53 (m, 1H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 202.0, 142.7, 130.9, 130.2, 116.4, 111.8, 107.3, 57.7, 49.8, 46.6, 31.5, 28.2, 23.9; ESI-HRMS: *m/z* calcd for $C_{13}H_{15}BrNO$ $[M+H]^+$: 280.0337; found 280.0263.

7,11b,12,13-Tetrahydro-6*H*-isoquinolino[2,1-a]quinoline-12-carbaldehyde (2h): Major diastereoisomer. ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.82 (s, 1H), 7.25-7.10 (m, 3H), 7.09-7.00 (m, 2H), 6.99-6.85 (m, 2H), 6.71-6.64 (m, 1H), 5.02 (d, J = 3.8 Hz, 1H), 4.20-4.04 (m, 1H), 3.49 (ddd, J = 13.5 Hz, 11.8 Hz, 4.0 Hz, 1H), 3.38-3.31 (m, 1H), 3.31-3.01 (m, 2H), 2.84 (dd, J = 16.2 Hz, 5.5 Hz, 1H), 2.65-2.59 (m, 1H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 202.6, 144.5, 136.7, 136.0, 129.3, 127.4, 126.9, 126.5, 124.3, 121.9, 118.4, 113.7 (one aromatic carbon missing due to overlapping), 56.9, 47.7, 46.3, 25.7, 24.3; ESI-HRMS: *m/z* calcd for $C_{18}H_{18}NO$ $[M+H]^+$: 264.1388; found 264.1391.

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