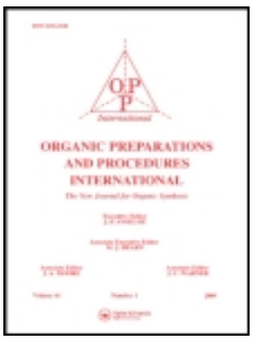

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Organic Preparations and Procedures International

The New Journal for Organic Synthesis

ISSN: 0030-4948 (Print) 1945-5453 (Online) Journal homepage: http://www.tandfonline.com/loi/uopp20

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**To cite this article:** Shohreh Ameli, Abolghasem Davoodnia & Mehdi Pordel (2016) Rapid One-pot Aspartic Acid-promoted Synthesis of Tetrahydrobenzo[b]pyrans in Water, Organic Preparations and Procedures International, 48:4, 328-336, DOI: 10.1080/00304948.2016.1194127

**To link to this article:** http://dx.doi.org/10.1080/00304948.2016.1194127

Published online: 20 Jul 2016.

*Organic Preparations and Procedures International*, 48:328–336, 2016

ISSN: 0030-4948 print / 1945-5453 online
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# Rapid One-pot Aspartic Acid-promoted Synthesis of Tetrahydrobenzo[*b*]pyrans in Water

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One-pot multi-component reactions (MCRs) have proven to be superior to conventional linear type processes that usually suffer from complex isolation procedures, produce significant amounts of waste materials, and low yields of the products.[1–3] These MCRs may be useful for drug design, discovery and synthesis of natural products because of their simplicity, efficiency, and high selectivity.[4,5] They consist of two or more steps which are carried out without isolation of any intermediate. They also provide a rapid and efficient approach to organic transformations including the preparation of polyfunctionalized heterocycles.[6–8] Still, great efforts are being made to develop new MCRs and improve known ones such as the synthesis of polyfunctionalized 4*H*-pyrans, especially tetrahydrobenzo[*b*]pyrans.

Pyrans are a large group of heterocycles with diverse and interesting biological activities. These compounds are reported to possess anti-bacterial, anti-cancer, spasmolytic, anti-coagulant, anti-anaphylactin, diuretic and anti-anaphylactic activities.[9–14] A number of them have also been employed as photo-active materials[15] and constitute the structural unit of a series of natural products.[16] In addition, they can be utilized as cognitive enhancers for the treatment of neuro-degenerative disease, including Alzheimer, AIDS, Huntington, Parkinson, and Down's syndrome diseases.[17] The most straightforward procedure for the synthesis of tetrahydrobenzo[*b*]pyrans is the one-pot three-component cyclocondensation of dimedone with an aromatic aldehyde and malononitrile in the presence of Lewis and Brønsted acid catalysts such as rare-earth perfluorooctanoate [RE $(PFO)_3$],[18] $I_2$,[19] nano-ZnO,[20] tetra(*n*-butyl)ammonium bromide (TBAB),[21] piperazine under solvent-free ball-milling conditions, [22] $PhB(OH)_2$,[23] $KF/Al_2O_3$ under ultrasound irradiation,[24] nanomixed metal oxides,[25] ionic liquid [bmim][$BF_4$],[26] NaBr under microwave irradiation,[27] hexadecyltrimethyl ammonium bromide (HTMAB),[28] 1,4-diazabicyclo[2.2.2]octane (DABCO),[29] basic ionic liquid,[30] carbon-based solid acid (CBSA),[31] 2,2,2-trifluoroethanol (TFE),[32] and $Na_2SeO_4$.[33] While each of these methods has its own advantage, many suffer from limitations such as prolonged reaction times, the use of relatively expensive catalysts, required utilization of organic solvents, un-satisfactory yields, and tedious isolation procedures. Thus the discovery of a new and efficient catalyst with high catalytic activity, short reaction times, recyclability, and simple reaction work-up for the preparation of tetrahydrobenzo[*b*]pyrans is of great interest.

Received October 19, 2015; in final form February 3, 2016.
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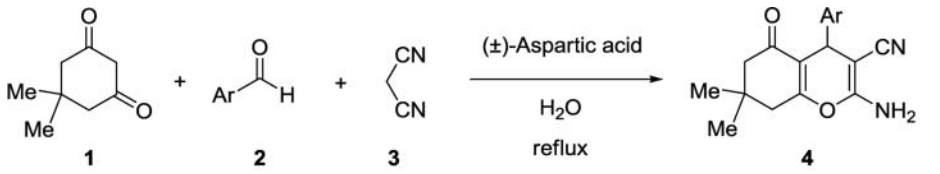
The low cost, availability, the absence of inflammability, of explosive, mutagenic, and carcinogenic properties are some of the economic and environmental benefits associated with water as solvent in a number of reactions.[34,35] Furthermore, the network of hydrogen bonds, the large surface tension, the high cohesive energy, specific heat capacity, and polarity are some of the valuable properties of water that can dramatically influence the course of reactions performed in this medium.[36–38] Our interest in heterocycles,[39–44] and in the development of environmentally friendly methods for the synthesis of organic compounds led us to study the employment of re-usable catalysts.[45–54] We now report our results for an efficient synthesis of tetrahydrobenzo [*b*]pyrans **4** in racemic form *via* a one-pot, three-component reaction of dimedone (**1**), aromatic aldehydes (**2**), and malononitrile (**3**) using racemic aspartic acid ((±)-aspartic acid) as a novel organocatalyst (*Scheme 1*).

To determine suitable conditions for this transformation, we examined the reaction of dimedone (1 mmol), 4-chlorobenzaldehyde (1 mmol), and malononitrile




**a)** Ar = $C_6H_5$, **b)** Ar = 4-$ClC_6H_4$, **c)** Ar = 4-$BrC_6H_4$, **d)** Ar = 3-$BrC_6H_4$, **e)** Ar = 4-$FC_6H_4$, **f)** Ar = 4-$HOC_6H_4$, **g)** Ar = 3-$O_2NC_6H_4$, **h)** Ar = 4-$O_2NC_6H_4$, **i)** Ar = 4-$MeOC_6H_4$, **j)** Ar = 2-thienyl

**Scheme 1**

(1 mmol) as test reaction for the synthesis of compound **4b** in the absence and presence of (±)-aspartic acid as catalyst. The conditions were optimized in terms of various parameters such as the amount of catalyst, the effect of solvent and the influence of temperature. The results are summarized in *Table 1*. None or only trace amounts of the product **4b** was formed in the absence of the catalyst in refluxing $H_2O$ or EtOH and also under solvent-free conditions at high temperature (*Entries 1–3*) indicating that the catalyst is necessary for the reaction. *Table 1* indicates that under solvent-free conditions and from among the solvents tested such as $H_2O$, EtOH, MeOH, $CHCl_3$, MeCN and using varying amounts of the catalyst, the reaction proceeded more easily and gave the highest yield, using 10 mol% of (±)-aspartic acid in $H_2O$ at reflux temperature (*Entry 8*). No significant improvement in yields or reaction times was observed using a higher amount of the catalyst (*Entry 9*). All subsequent reactions were carried out in these optimized conditions.

To explore the generality of this method, we extended it to a range of aromatic aldehydes under the optimized reaction conditions. As illustrated in *Table 2*, in all cases the expected products were obtained in high yields and short reaction times. Melting points, TLC and the $^1$H NMR spectroscopic data were used to establish that only one product is formed in all cases with no undesirable side-products being present after purification. Facile separation of the products from the catalyst makes this method useful for the synthesis of tetrahydrobenzo[*b*]pyrans. Under the same conditions however, no reaction occurred when aliphatic aldehydes were used. The products **4** were obtained in racemic form and the structures were deduced from their spectral data and concordance of their mps with those reported (*Table 2*).

**Table 1**
Optimization of Reaction Conditions for the Synthesis of Compound **4b** Catalyzed by (±)-Aspartic Acid*

| Entry | Catalyst (mol%) | Solvent | T/°C | Time/min | Yield (%) |
|---|---|---|---|---|---|
| 1 | None | $H_2O$ | Reflux | 90 | Trace |
| 2 | None | EtOH | Reflux | 90 | Trace |
| 3 | None | Solvent-free | 120 | 100 | None |
| 4 | 1 | $H_2O$ | Reflux | 80 | 31 |
| 5 | 3 | $H_2O$ | Reflux | 65 | 46 |
| 6 | 5 | $H_2O$ | Reflux | 50 | 64 |
| 7 | 7 | $H_2O$ | Reflux | 30 | 78 |
| 8 | 10 | $H_2O$ | Reflux | 10 | 96 |
| 9 | 12 | $H_2O$ | Reflux | 15 | 97 |
| 10 | 10 | $H_2O$ | 80 | 20 | 77 |
| 11 | 10 | $H_2O$ | 60 | 35 | 63 |
| 12 | 10 | $H_2O$ | r.t. | 110 | 54 |
| 13 | 7 | EtOH | Reflux | 35 | 68 |
| 14 | 10 | EtOH | Reflux | 20 | 80 |
| 15 | 7 | MeOH | Reflux | 35 | 53 |
| 16 | 10 | MeOH | Reflux | 25 | 67 |
| 17 | 7 | $CHCl_3$ | Reflux | 50 | 31 |
| 18 | 10 | $CHCl_3$ | Reflux | 30 | 37 |
| 19 | 7 | MeCN | Reflux | 30 | 49 |
| 20 | 10 | MeCN | Reflux | 25 | 54 |
| 21 | 7 | solvent-free | 120 | 25 | 46 |
| 22 | 10 | solvent-free | 120 | 15 | 50 |

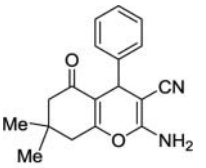
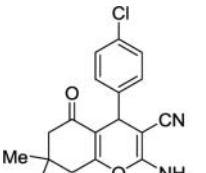
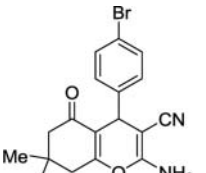
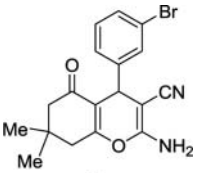
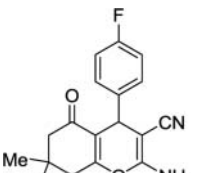
*Reaction conditions: dimedone **1** (1 mmol), 4-chlorobenzaldehyde **2b** (1 mmol), and malononitrile (1 mmol).

The applicability and efficiency of our catalyst was compared with some of the reported methods for the synthesis of tetrahydrobenzo[*b*]pyrans as shown in *Table 3*. Our very simple procedure gave higher yields in shorter times.

Since the stability and recyclability of catalysts are important factors in these methods, the recovery and catalytic activity of recycled (±)-aspartic acid was explored. After completion of the reaction, the product was collected, and washed with cold water. Evaporation of the combined filtrates to dryness under reduced pressure gave the solid catalyst which was dried at 60°C under vacuum for 1 h, and could be re-used for the same experiment without loss of activity over at least five catalytic cycles (93–96% yield of **4b**). The structure of (±)-aspartic acid catalyst is retained during those five cycles.

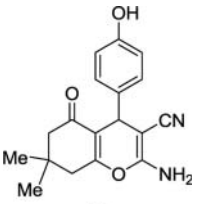
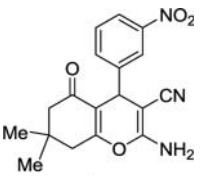
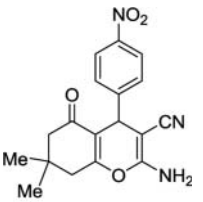
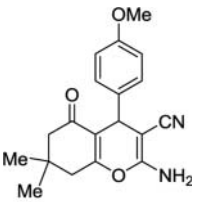
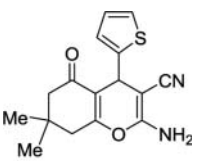
In conclusion, we have successfully developed a highly efficient and green method for the synthesis of tetrahydrobenzo[*b*]pyrans in racemic form by one-pot three-component cyclocondensation of dimedone, aromatic aldehydes, and malononitrile using inexpensive and readily available (±)-aspartic acid as catalyst. The reaction occur in water and furnishes the expected products in high yields. Short reaction times, simple isolation of the products, and usage of eco-friendly nontoxic catalyst and solvent are other features of this procedure. In addition the catalyst was easily recovered and used in multiple catalytic cycles.

**Table 2**
Synthesis of Tetrahydrobenzo[*b*]pyrans **4** using (±)-Aspartic Acid as Catalyst

| Entry | Ar | Product | Time (min) | Yield (%) | mp (°C) | lit. mp (° C) |
|---|---|---|---|---|---|---|
| 1 | $C_6H_5$ |  **4a** | 15 | 94 | 230–232 | 227–229[18] |
| 2 | 4-$ClC_6H_4$ |  **4b** | 10 | 96 | 212–213 | 213–215[22] |
| 3 | 4-$BrC_6H_4$ |  **4c** | 10 | 98 | 210–211 | 215[33] |
| 4 | 3-$BrC_6H_4$ |  **4d** | 13 | 95 | 231–233 | 230–232[31] |
| 5 | 4-$FC_6H_4$ |  **4e** | 5 | 89 | 188–189 | 189–191[22] |

(*Continued on next page*)

**Table 2**
Synthesis of Tetrahydrobenzo[*b*]pyrans **4** using (±)-Aspartic Acid as Catalyst
*(Continued)*

| Entry | Ar | Product | Time (min) | Yield (%) | mp (°C) | lit. mp (° C) |
|---|---|---|---|---|---|---|
| 6 | 4-$HOC_6H_4$ |  **4f** | 10 | 88 | 209–211 | 210–212[31] |
| 7 | 3-$O_2NC_6H_4$ |  **4g** | 7 | 88 | 214–215 | 210–212[23] |
| 8 | 4-$O_2NC_6H_4$ |  **4h** | 7 | 90 | 180–182 | 180–181[29] |
| 9 | 4-$MeOC_6H_4$ |  **4i** | 15 | 95 | 201–202 | 200–202[29] |
| 10 | 2-Thienyl |  **4j** | 15 | 89 | 225–227 | 224–226[26] |

Reaction conditions: dimedone **1** (1 mmol), an aromatic aldehyde **2** (1 mmol), malononitrile **3** (1 mmol), (±)-aspartic acid (10 mol% based on dimedone), $H_2O$ (5 ml), reflux.

**Table 3**
Comparison of the Efficiency of Various Catalysts for the Synthesis of Tetrahydrobenzo [*b*]pyrans

| | Conditions | | | | | |
|---|---|---|---|---|---|---|
| Catalyst | Solvent | T (°C) | Other | Time (min) | Yield (%) | *Lit.* |
| $[RE(PFO)_3]$ | EtOH | 60 | — | 240–480 | 80–90 | 18 |
| $I_2$ | DMSO | 120 | — | 180–240 | 80–92 | 19 |
| Nano ZnO | $EtOH/H_2O$ | r.t. | — | 180–240 | 79–91 | 20 |
| TBAB | EtOH | reflux | — | 20–140 | 87–95 | 21 |
| Piperazine | — | r.t. | Ball-milling at 20–25 Hz | 20–120 | 88–96 | 22 |
| $PhB(OH)_2$ | $EtOH/H_2O$ | reflux | — | 30 | 61–88 | 23 |
| $KF/Al_2O_3$ | EtOH | 27–34 | Ultrasound irradiation | 20–240 | 81–98 | 24 |
| Nano mixed metal oxides | EtOH | reflux | — | 35–45 | 90–94 | 25 |
| $[bmim][BF_4]$ | $[bmim][BF_4]$ | 80 | — | 30–120 | 85–95 | 26 |
| NaBr | — | 70–85 | Microwave irradiation | 10–15 | 80–95 | 27 |
| HTMAB | $H_2O$ | 80–95 | — | 180 | 59–95 | 28 |
| DABCO | $H_2O$ | reflux | — | 120 | 75–97 | 29 |
| Basic ionic liquid | $EtOH/H_2O$ | reflux | — | 5–240 | 50–94 | 30 |
| CBSA | EtOH | reflux | — | 10–20 | 85–98 | 31 |
| TFE | TFE | reflux | — | 300 | 80–95 | 32 |
| $Na_2SeO_4$ | $EtOH/H_2O$ | reflux | — | 40–210 | 80–98 | 33 |
| (±)-Aspartic acid | $H_2O$ | reflux | — | 5–15 | 88–98 | This work |



## Experimental Section

All reagents and chemicals were available commercially from Aldrich and Merck, and used without purification. Melting points were determined using a Stuart SMP3 melting point apparatus. The IR spectra were recorded as KBr pellets on a Tensor 27 Bruker spectrophotometer. The $^1H$ NMR (300 & 400 MHz) spectra were determined using Bruker 300 & 400 spectrometers in DMSO-$d_6$ as the solvent with TMS as the internal reference.

### *General Procedure for the Synthesis of Tetrahydrobenzo[*b*]pyrans (4)*

A mixture of dimedone (**1**, 0.14 g, 1 mmol), the aromatic aldehyde **2** (1 mmol), malononitrile (**3**, 0.07 g, 1 mmol), and (±)-aspartic acid (0.01 g, 0.1 mmol, 10 mol% based on dimedone) in water (5 ml) was heated under reflux for 5–15 min. During the procedure, the reaction was monitored by TLC on silica gel (*n*-hexane-ethyl acetate, 3:1). Upon completion of the reaction, the mixture was cooled to room temperature. The precipitated solid was collected, washed with cold water (10 ml × 3) and recrystallized from 96% ethanol (5 ml) to give products **4** in racemic form in high yields.

### *Larger Scale Preparation of 4b*

A mixture of dimedone (**1**, 2.8 g, 20 mmol), the aromatic aldehyde **2** (20 mmol), malononitrile (**3**, 1.4 g, 20 mmol), and (±)-aspartic acid (0.2 g, 2 mmol, 10 mol% based on dimedone) in water (100 ml) was heated under reflux for 5–15 min. The progress of the reaction was monitored by TLC (*n*-hexane-ethyl acetate, 3:1). Upon completion, the reaction mixture was cooled to room temperature. The precipitated solid was collected, washed with cold water (200 ml × 3) and recrystallized from 96% ethanol (100 ml) to give pure **4b** in racemic form (95% yield).

## Acknowledgements

The authors express their gratitude to the Islamic Azad University, Mashhad Branch for financial support.



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