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ULTRASOUND-ASSISTED HYDROGENATION AND HYDROGENOLYSIS OF BETA-LACTAMS

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Abstract:
Catalytic transfer hydrogenation reaction was conducted on substituted β-lactams to accomplish synthesis of numerous open chain amides using ultrasound. The reduction of alkene and hydrogenolysis of functional groups were completed rapidly at about 50°C with 10% Pd/C and ammonium formate. β-Lactams were cleaved through hydrogenolysis of the N-C_4 bond with 10% Pd/C in good yield. These techniques described here for ultrasound-assisted hydrogenation reactions are simple, rapid and efficient.

Key words: Hydrogenolysis, Hydrogenation, β-Lactams, Sonochemistry

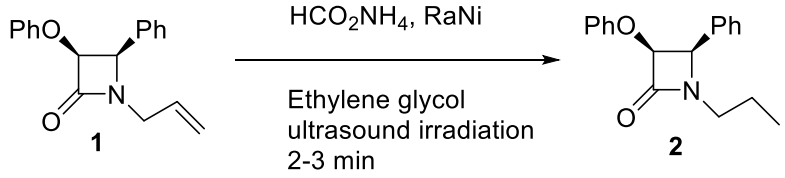
Introduction:

Catalytic hydrogenation [I] has a key role in the synthesis of organic compounds. Hydrogen gas from a cylinder connected to an appropriate valve system is required for hydrogenation experiment. The reaction flask must be free from oxygen. We report here sonochemical hydrogenation and hydrogenolysis methods that are applied on substituted β-lactams. Depending upon the functional groups, unsaturated groups in β-lactams are hydrogenated or hydrogenolysis are occurred.

Results and Discussion:

Catalytic transfer hydrogenation (CTH) is a crucial process [II, III]. This is a simple method in which one of the donors (cyclohexane [IV], hydrazine [V], formic acid [VI], ammonium formate [VII], cyclohexadiene [VIII], aphosphinic acid [IX], sodium hypophosphite[X]) is used. Ethyl alcohol is generally used as the solvent for CTH [XI].
The application of ultrasound in pharmacy, chemistry, biotechnology, and environmental engineering is documented [XII-XIV]. We have observed that catalytic transfer hydrogenation can be performed very rapidly using ultrasound technique [XV]. In this paper, the scope of this method is discussed using diverse β-lactams. The alkenyl β-lactam **1** was reduced to **2** using ammonium formate and Raney Ni using ultrasound very rapidly (Scheme 1).

Scheme 1



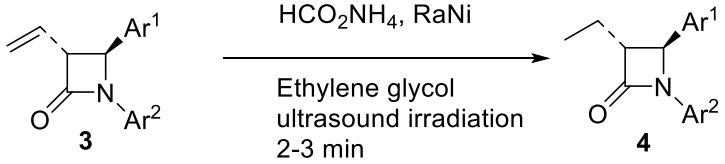
A polar solvent with a high boiling point is required to perform this reaction. Ethylene glycol (bp 198°C) or 1,3-propanediol (bp 210-212°C) can be used under ultrasound irradiation. Hydrazine hydrate was used as the hydrogen donor for this reaction but ammonium formate was proved to be the best reagent.

Most of our studies on CTH reactions were conducted with Pd/C (10%) catalysts. A few experiments were performed with Ra/Ni catalyst.

Reduction and Hydrogenolysis of β-Lactams. Ammonium formate-induced reduction in the presence of 10% Pd/C demonstrated that oleic and linoleic acids (on a 1-2 g scale), were completely reduced to stearic acid in only 2 min at about 50-60°C. This success was prompted to undertake an examination of CTH experiments of various types.

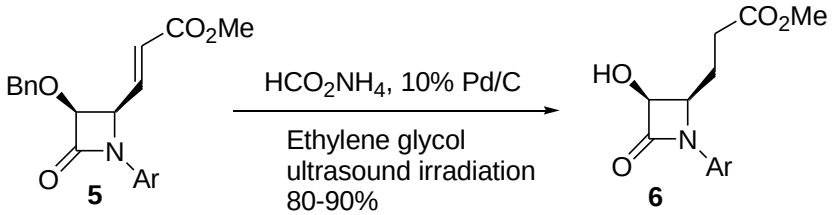
The ultrasound-assisted CTH method was applied to 3-alkenyl beta-lactams using commercially available Raney nickel catalyst. The reduction products were obtained in about 80% **3** to **4** (Scheme 2). The β-lactam **5** underwent selective hydrogenolysis of the *O*-benzyl group in the presence of 10% Pd/C as the catalyst and reduction of the unsaturated ester to a saturated ester side chain to give the β-lactam **6** (Scheme 3).

Scheme 2



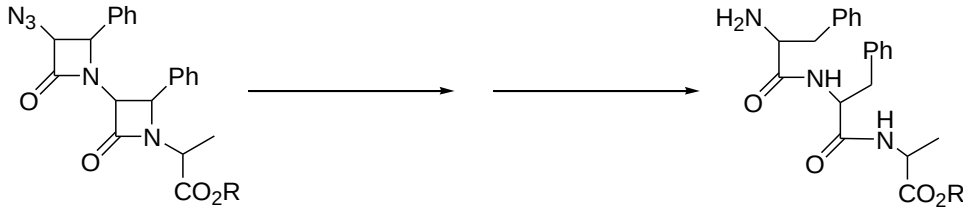
a Ar = Ph, Ar^1 = Ph
b Ar = PMP, Ar^1 = Ph
c Ar = PMP, Ar^1 = 2-furyl
PMP = *p*-methoxyphenyl

Scheme 3



a Ar = PMP
b Ar = Bn
Bn = CH_2Ph

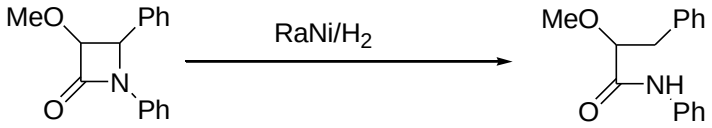
Scheme 4



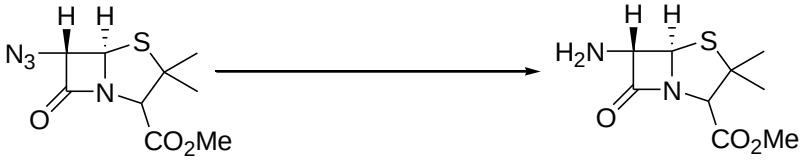
Using catalytic hydrogenation, Ojima et al. [XVI] demonstrated a cleavage of the N-C_4 bonds in 4-phenyl-2-azetidinones to produce phenylalanine derivatives (for example, see Scheme-4). It was [XVII] observed that, in the presence of a large excess of Raney nickel and hydrogen, 3-methoxy-1,4-diphenyl-2-azetidinone underwent β-lactam ring rupture to provide the anilide of α-methoxy-β-phenylpropionic acid (Scheme-5). Mild catalytic hydrogenation (5-10% Pd/C catalyst, room temperature) was the method for the reduction of α-azido-β-lactams to α-amino-β-lactams (Scheme-6).

Ultrasound-assisted catalytic transfer hydrogenolysis of 4-phenyl-2-azetidinones was performed at 50-60°C using 10% Pd/C as the catalyst. Rapid scission of the N-C4 bond was observed. The *N*-benzyl group of the β-lactam **9** was not hydrogenolyzed, but the *O*-Bn group at C-3 was converted to an OH group. Alkenes (**11**, **13**, and **15)** were reduced to alkyl groups (Scheme 7). The reduction product was obtained in high yield and in a few minutes. It was useful to note that under these conditions Ra–Ni did not cleave the β-lactam ring in **3** (see Scheme-2).

Scheme 5



Scheme 6

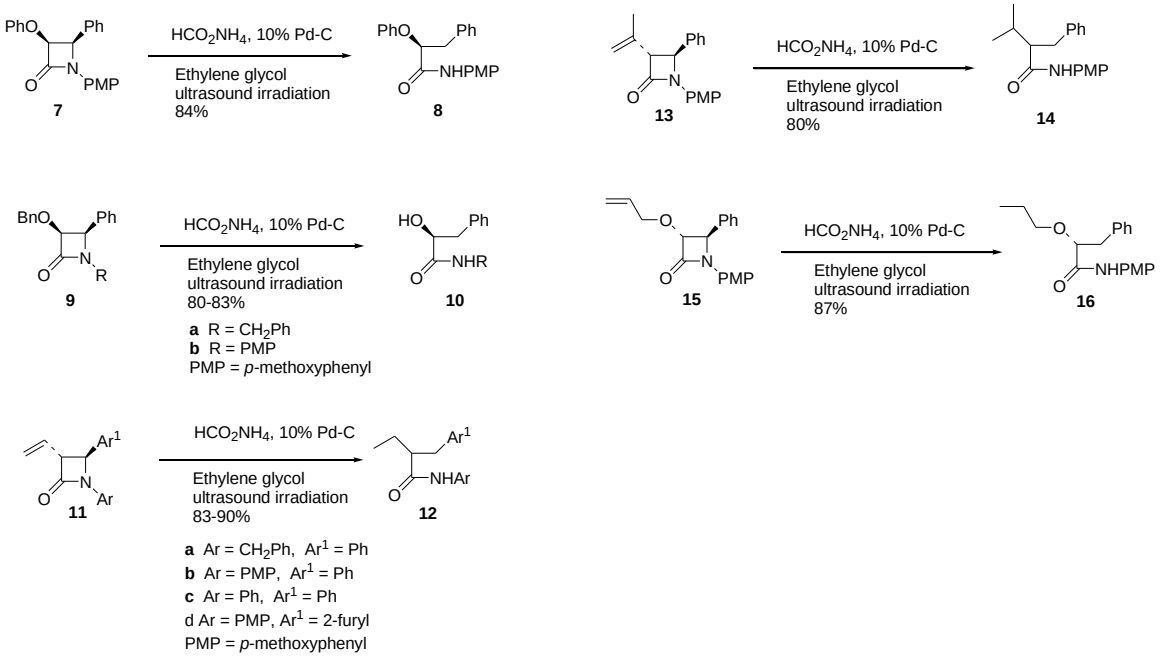


The hydrogenation of 4-styryl-2-azetidinones (**17** and **19**) showed an interesting result (Scheme 8). The alkene groups were reduced but partial β-lactam ring scission was observed under the conditions used with Pd/C catalyst. Therefore, the two products (**18a, b)**, the saturated β-lactam and the open chain amide, were formed 6:4 ratio.

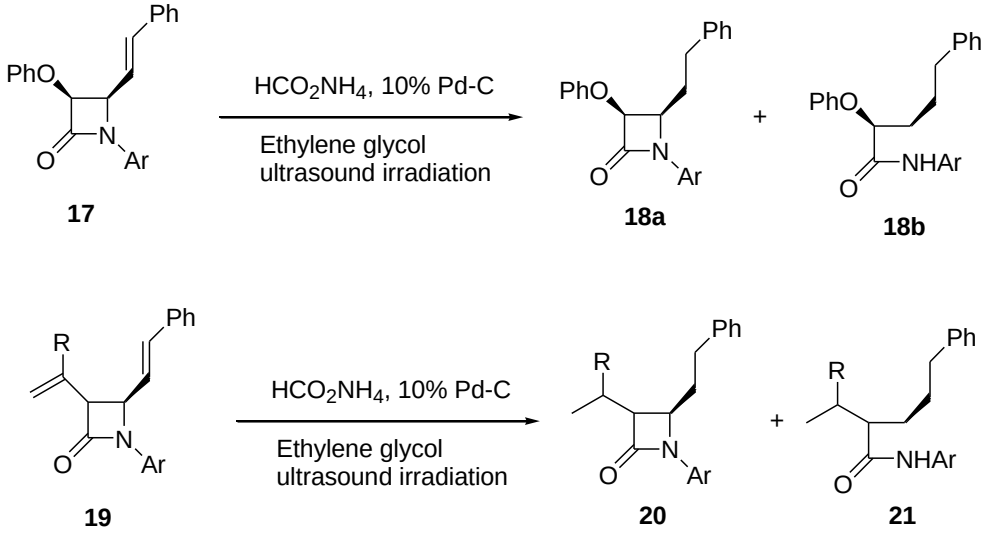
A procedure was developed [XIX] for resolving hydroxy-β-lactams (e.g., **23**) by the formation of two diastereomeric glycosides by Ferrier reaction involving a glucal (e.g., **22**). The determination of the stereochemistry of the glycosidic bond in these compounds was essential. CTH reaction using 10% Pd/C was performed on **24a** and found to be successful. The unsaturated bond in the sugar moiety was hydrogenated without ring fission of the β-lactam to give **25a**. Allylic deacetoxylation was also occurred to give **25b**. Ultrasound-induced CTH method of **26** produced a reduced and deacetylated product **27**.

Some monocyclic β-lactams were prepared with *exo*-alkene group at C-3 during the studies on carbapenem antibiotics [XV-b, XX]. The conjugated double bonds were reduced under ultrasound-induced CTH reaction. Because of the planar shape of the β-lactam ring and the bulk of the substituent at C-4, the hydrogenation was stereospecific. Therefore, only *cis* β-lactams were obtained because the catalyst surface was always placed *trans* to the large substituent at C-4.

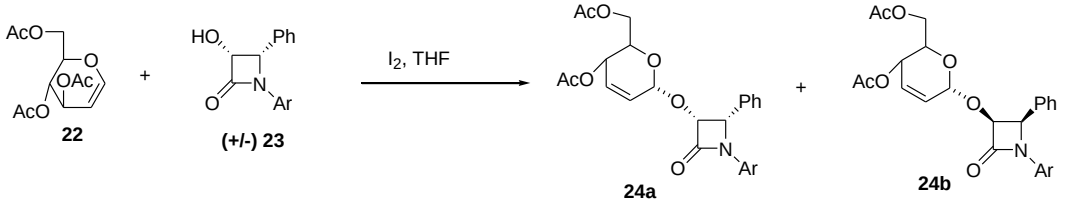
Scheme 7

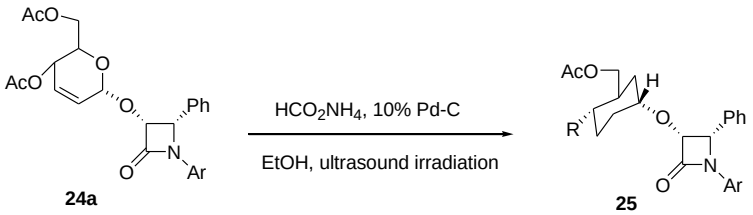


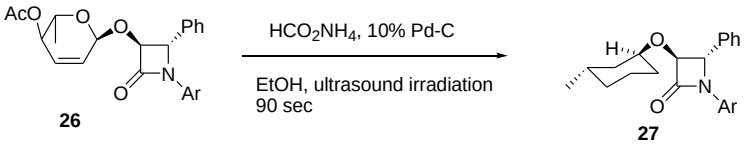
Scheme 8



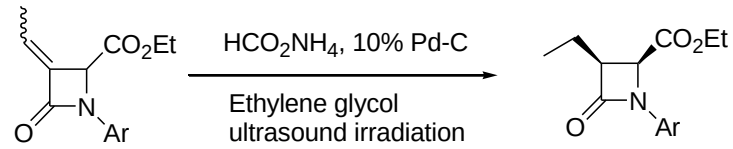
Scheme 9

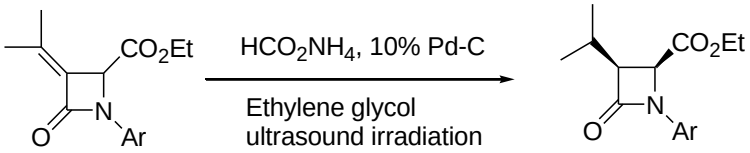




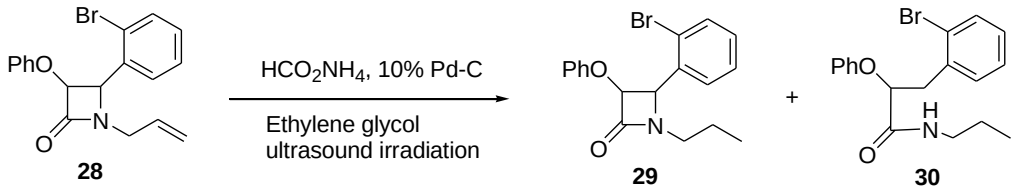


Scheme 10





Scheme 11



Experimental Section:

Melting points were taken with a Mel-temp apparatus. IR spectra were taken on a Perkin-Elmer instrument. NMR spectra were recorded on a Bruker spectrometer using TMS as a standard. Chemical ionization mass spectra were recorded on a Biospect instrument using CH_4. Thin-layer chromatography was conducted with Whatman plates. Micro Analyses were performed by Schwartzkopf Microanalytical Laboratory, NY.

General Procedure for the synthesis of β-Lactams:

The synthesis of the β-lactams were reported in our earlier paper [XX-XXIII].

General Procedure for CTH Reaction. An ultrasound apparatus placed in a hood should be used. The reaction vessel should be a beaker or an Erlenmeyer flask of fairly large size. The

desired temperature of the solvent should be 50-60°C. The catalyst should be quickly introduced into the reaction vessel and covered with the solvent. The compound to be reduced is dissolved in the solvent (ethylene glycol or 1,3-propanediol) and then added to the reaction vessel. The hydrogen donor (such as ammonium formate) is added now. Ultrasound irradiation for the predetermined period of time should be applied. The irradiation with the ultrasound wave should be resumed for another 3-4 min. After completion of reaction monitored by TLC, careful decantation of the reaction mixture after cooling followed by the addition of glycol to the reaction vessel would preserve the catalyst for the next experiment.

After the hydrogenation the reaction mixture was cooled and then filtered. The filtrate was diluted with water and extracted with ethyl acetate, and the organic layer was washed with water. Evaporation of the solvent from the organic layer (dried over anhydrous Na_2SO_4) followed by crystallization gave the pure product an 80-90% yield. We have observed that the optimal ratio of the catalyst (10% Pd/C) to substrate is 0.3:1 by weight for each reducible group. Five equivalents of ammonium formate for each reducible group gave good results.

8: yield 83%; mp 125°C; IR (Nujol) 3310, 1630 cm^{-1}; ^1H NMR 7.78 (brs, 1H) 7.05 (m, 14H), 4.92 (dd, $J_1 = J_2 = 3.91$ Hz, 1H), 3.81 (s, 3H), 3.35 (d, $J_1 = J_2 = 14.16$ Hz, 2H); ^{13}C NMR 168.7, 157.2, 136.4, 129.8, 128.2, 126.3, 122.4, 122.1, 115.8, 114.2, 80.1, 55.49, 38.9; Anal. Calcd for $C_{22}H_{2l}NO_3$; C, 76.00; H, 6.05; N, 4.03. Found: C, 75.75; H,6.15; N, 3.98.

10a: yield 85%; mp 88°C; IR (Nujol) 3310, 1632 cm^{-1}; ^1H NMR 7.20-7.10 (m, 10H), 4.25-4.30 (m, 2H), 3.39 (brs, 1H), 3.10 (dd, 2H), 2.65 (dd, 1H); CIMS (NH_3 reagent gas) m/z 273 (M + 18)$^+$. Anal. Calcd for $C_{26}H_{17}NO_2$: C 75.29; H, 6,66; N, 5.49. Found: C, 75.15, H, 6.70; N, 5.60.

10b: yield 80%; mp 129°C; IR (Nujol) 3310, 1650 cm^{-1}; ^1H NMR 8.18 (brs, 1H), 7.50-6.82 (m, 9H), 4.4-4.5 (m, 1H), 3.80 (s, 3H), 3.41 (dd, $J_1 = 7.80$ Hz, $J_2 = 7.80$ Hz, 1H), 3.11 (dd, J_1=8.30 Hz, $J_2 = 14.10$ Hz, 1H), 2.65 (d, $J = 8.30$ Hz, 1H); CIMS (NH_3 reagent gas) m/z 289 (M + H)$^+$. Anal. Calcd for $C_{16}H_{17}$- NO_3: C, 66.43; H, 6.51; N, 4.48. Found C, 66.62; H, 5.74; N, 4.66.

12a: yield 83%; mp 113-114°C; IR (Nujol) 1645 cm^{-1}; ^1H NMR 7.12-6.70 (m, 9H), 3.74 (s, 3H), 2.81-2.7 (m, 2H), 2.20-2.12 (m, 1H), 1.73 (m, 2H), 0.92 (t, 3H); CIMS (NH_3 reagent gas) m/z 301 (M + 18)$^+$. Anal. Calcd for $C_{18}H_{21}NO_2$: C, 76.32; H, 7.42; N, 4.84. Found: C, 75.60; H, 7.69; N, 4.62.

12b: yield 75%; mp 106°C; IR (CH_2Cl_2) 1645 cm^{-1}; ^1H NMR 7.60 (s, 1H), 7.35-7.10 (m, 3H), 6.80 (d, $J = 6.61$ Hz, 2H), 6.22-6.32 (m, 1H), 6.00-6.05 (m, 1H), 3.75 (s, 3H), 3.10-2.67 (m, 2H), 2.59-2.45 (m, 1H), 1.86-1.44 (m, 2H), 0.98 (t, $J = 7.34$ Hz, 3H); CIMS (CH_4 gas) m/z 258 (M + H)$^+$. Anal. Calcd for $C_{16}H_{19}NO_2$: C, 74.68; H, 7.44; N, 5.44. Found C, 74.34; H, 7.28; N, 5.23.

14: yield 88%; IR (Nujol) 1640 cm^{-1}; ^1H NMR 7.14-7.05 (m, H), 6.61-6.60 (m, 2H), 3.65 (s, 3H), 2.84 (d, $J = 6.81$ Hz, 1H), 2.08-1.95 (m 2H), 1.04 (d, $J = 6.26$ Hz, 3H), 0.99 (d, $J = 6.18$ Hz, 3H); CIMS (CH_4 gas) m/z 258 (M + H)$^+$.

16: yield 88%; mp 70°C; IR (Nujol) 3345, 1735 cm^{-1}; ^1H NMR 8.26 (brs, 1H), 7.50-6.83 (m, 9H), 4.06 (dd, $J_1 = 3.5$ Hz, $J_2 = 7.57$ Hz, 1H), 3.84 (s, 3H), 3.40 (m, 2H), 3.06 (dd, $J_1 = 7.72$ Hz, $J_2 = 3.95$ Hz, 2H), 1.59-1.55 (m, 2H), 0.92 (t, 3H); CIMS (NH_3 reagent gas) m/z 314 (M + H)$^+$.

18a: yield 64%; mp 71°C; IR (Nujol) 1745 cm^{-1}; ^1H NMR 7.11-7.05 (m, 14H), 5.93 (d, $J = 4.9$ Hz, 1H), 4.72-4.65 (m, 1H), 3.80 (s, 3H), 2.65-2.56 (m, 2H), 1.83-1.75 (m, 2H); CIMS (NH_3 reagent gas) m/z 399 (M + 18)$^+$. Anal. Calcd for $C_{24}H_{23}NO_3$: C, 77.21; H, 6.16; N, 3.75. Found: C, 76.38; H, 6.51; N, 3.84.

18b: yield 35% oil; IR (CH_2Cl_2) 3310, 1650 cm^{-1}; ^1H NMR 7.40-7.15 (m, 13H), 6.82 (d, $J = 8.95$ Hz, 2H), 3.75 (s, 3H), 2.65 (t, $J = 7.19$ Hz, 1H), 2.31 (t, $J = 6.97$ Hz, 2H), 1.72-1.65 (m, 4H); CIMS (CH_4 reagent gas) m/z 376 (M + H)$^+$. Anal. Calcd for $C_{24}H_{25}NO_3$: C, 76.76; H, 6.71; N, 3.73. Found: C, 76.55; H, 6.58; N, 3.82.

20a: yield 42%; mp 73°C; IR (CH_2Cl_2): 1750 cm^{-1}; ^1H NMR 7.35-7.20 (m, 7H), 6.84 (d, *J* = 8.81 Hz, 2H), 4.12-4.14 (m, 1H), 3.75 (s, 3H), 3.40-3.18 (m, 1H), 2.85-2.67 (m, 2H), 2.3-1.67 (m, 4H), 1.18 (t, *J* = 7.41 Hz, 3H); CIMS (CH_4 gas) m/z 326 $(M + H)^+$. Anal. Calcd for $C_{20}H_{23}NO_2$: C, 77.64; H, 7.49; N, 4.52. Found: C, 77.86; H, 7.30; N, 4.67.
20b: yield 32%; mp 52°C; IR (CH_2Cl_2) 1742 cm^{-1}; ^1H NMR 7.32-7.24 (m, 7H), 6.81 (d, *J* = 6.7 Hz, 2H), 4.10 (dd, J_1 = 5.50 Hz, J_2 = 11.28 Hz, 1H), 3.81 (s, 3H), 3.12 (dd, J_1 = 5.60 Hz, J_2 = 10.05 Hz, lH), 2.81-2.64 (m, 2H), 2.29-1.84 (m, 3H), 1.29 (d, *J* = 6.55 Hz, 3H), 1.08 (d, 6.34 Hz); CIMS (CH_4 gas) m/z 324 $(M + H)^+$. Anal. Calcd for $C_{2l}H_{25}NO_2$: C, 77.98; H, 7.79; N, 4.33. Found: C, 77.70; H, 7.68; N, 4.31.
21a: yield 42%; mp 86°C; IR (CH_2Cl_2) 3310, 1650 cm^{-1}; ^1H NMR 7.75 (s, 1H), 7.45 (d, *J* = 8.9 Hz, 2H), 7.30-7.18 (m, 5H), 6.82 (d, *J* = 8.9 Hz, 2H), 3.78 (s, 3H), 2.65 (t, *J* = 6.77 Hz, 2H), 2.06-2.08 (m, 1H), 1.80-1.55 (m, 6H), 0.93 (t, *J* = 7.35 Hz, 3H); CIMS (CH_4 gas) m/z 312 $(M + H)^+$. Anal. Calcd for $C_{20}H_{25}NO_2$: C, 77.13; H, 8.09; N, 4.49. Found: C, 77.10; H, 8.27; N, 4.40.
21b: yield 38%; mp 122°C; IR (CH_2Cl_2) 3340, 1652 cm^{-1}; ^1H NMR 7.44 (d, *J* = 8.77 Hz, 2H), 7.28-7.15 (m, 6H), 6.86 (d, *J* = 9 Hz, 2H), 3.74 (s, 3H), 2.65 (t, *J* = 7.07 Hz, 2H), 1.94-1.55 (m, 6H), 0.99 (d, *J* = 5.73 Hz, 3H); CIMS (CH_4 gas) m/z 326 $(M + H)^+$. Anal. Calcd for $C_{21}H_{27}NO_2$: C, 77.49; H, 8.36; N, 4.30. Found: C, 77.67; H, 8.45; N, 4.45.
29: mp 109°C; IR (CH_2Cl_2) 1750 cm^{-1}; ^1H NMR 7.39-6.64 (m, 10H), 5.45 (d, *J* = 4.33 Hz, 1H), 4.95 (d, *J* = 4.30 Hz, 1H), 3.50-3.34 (m, 1H), 3.10-2.85 (m, 1H), 1.55-1.48 (m, 2H), 0.88 (t, *J* = 7.32 Hz, 3H); CIMS (CH_4 gas) m/z 282 $(M + H)^+$. Anal. Calcd for $C_{18}H_{19}NO_2$: C, 76.84; H, 6.80; N, 4.97. Found C, 76.77; H, 6.58; N, 4.99.
30: IR (CH_2Cl_2): 1645 cm^{-1}; ^1H NMR 7.38-6.53 (m, 11H), 4.85 (dd, J_1 = 6.83 Hz, J_2 = 10.80 Hz, 1H), 2.96-2.43 (m, 3H), 2.51-2.47 (m, 1H), 1.42-1.29 (m, 2H), 0.76 (t, *J* = 7.40 Hz, 3H).

Conclusions:

Ultrasound-assisted technique has been developed which was safe, rapid, and efficient for performing metal mediated catalytic hydrogenation/hydrogenolysis in simple glass apparatus (just beakers and flasks). All reactions were performed under ambient pressure in open systems using ultrasound irradiation helped to prevent any possible explosion. Hydrogen source was ammonium formate for this catalytic transfer hydrogenation method, which is inexpensive and easy to avoid hydrogen gas cylinders.

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