Microwaves Make Hydroformylation a Rapid and Easy Process

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General experimental conditions:
In a typical experiment the starting alkene (0.64 mmol) was dissolved in toluene (4 mL). (Ph₃P)₃Rh(CO)H (0.013 mmol), XANTPHOS (0.05 mmol) and [bmim][BF₄] (187 µL) were added. The yellow solution obtained was submitted to pressurized syngas at 40 psi (2.7 Atm) and heated for 4 minutes at 110°C by microwave irradiation at 150 W (value previously settled on the Microwave oven. The flask was cooled and the internal gas released. Et₂O (10 mL) was added to the reaction mixture and washed twice with H₂O (5 mL x 2); the organic layers were washed with brine (5 mL) and dried on dry Na₂SO₄. After filtration and evaporation \textit{in vacuo} the yellow oil obtained was purified by flash chromatography.
Microwave report:

Sample ID Null

Method el1 08/03/2006 12.00.10

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Pressure and temperature window.
$^1$H NMR (200 MHz, CDCl$_3$) 0.85 (t, $J = 6.6$, 3H), 1.20 (m, 10H), 1.59 (m, 2H), 2.41 (m, 2H), 9.73 (s, 1H).
$^1$HNMR (200 MHz, CDCl$_3$) 1.42 (d, $J = 7.0$, 3H), 3.61 (q, $J = 7.0$, 1H), 7.21 (m, 3H), 7.33 (m, 2H), 9.67 (s, 1H).
$^{1}$H NMR (200 MHz, CDCl$_3$) 1.46 (d, $J = 7.2$, 3H), 2.34 (s, 3H), 3.56 (q, $J = 7.2$, 1H), 7.15 (d, $J = 6.8$, 2H), 7.26 (d, $J = 6.8$, 2H), 9.68 (s, 1H).
$^1$H NMR (200 MHz, CDCl$_3$) 1.95 (m, 2H), 2.45 (t, $J = 7.2$, 2H), 2.65 (t, $J = 7.2$, 2H), 7.25 (m, 5H), 9.86 (s, 1H).
$^{1}$H NMR (200 MHz, CDCl$_3$) 1.16 (t, $J$ = 7.1, 3H), 1.58 (m, 4H), 2.21 (m, 2H), 2.34 (m, 2H), 4.03 (q, $J$ = 7.1, 2H) 9.67 (s, 1H).
$^{1}$H NMR (200 MHz, CDCl$_3$) 1.33 (m, 12H), 1.55 (m, 4H) 1.82 (m, 2H), 2.39 (t, 2H), 3.62 (t, 2H), 9.74 (s, 1H).
$^1$H NMR (200 MHz, CDCl$_3$) 1.71 (m, 2H), 2.15 (m, 2H), 4.98 (m, 1H), 5.35 (m, 1H), 7.44 (m, 11H). $^{13}$C NMR (50 MHz, CDCl$_3$) 29.68, 39.40, 55.41, 195.10, 123.02, 125.49, 126.83, 128.39, 137.88, 152.99. m/z: ES/MS 302 [M+Na]$^+$. HRMS Calcd for C$_{19}$H$_{20}$N$_2$O$_7$S , 420.0991. Found 420.0989.
$1^H$ NMR (200 MHz, CDCl$_3$) 1.75 (m, 4H), 2.52 (m, 2H), 3.78 (m, 2H), 7.71 (m, 2H), 7.84 (m, 2H), 9.69 (s, 1H).
$^1$H NMR (200 MHz, CDCl$_3$): 1.72 (m, 4H), 2.34 (m, 4H), 2.83 (s, 3H), 4.99 (d, $J = 8.5$, 2H), 7.33 (m, 5H), 7.85 (m, 1H), 8.01 (m, 1H), 9.62 (s, 1H), 9.71 (s, 1H). $^{13}$C NMR (200 MHz, CDCl$_3$): 21.6, 24.55, 32.3, 33.2, 43.6, 48.5, 123.8, 125.1, 125.6, 125.91, 127.0, 128.2, 128.4, 131.7, 132.7, 133.8, 202.0. m/z (ES-MS) = 306 [M$^+$+Na] HRMS Calcd for C$_{18}$H$_{21}$NO$_2$ Exact Mass: 283.1572. Found 283.1569.
$^{1}H$ NMR (200 MHz, CDCl$_3$) 1.78 (m, 4H), 2.47 (m, 2H), 2.69, (m, 2H), 7.55 (m, 4H), 7.92 (m, 1H), 8.65 (m, 1H), 9.77 (s, 1H). $^{13}$C NMR (200 MHz, CDCl$_3$) 22.15, 25.71, 37.24, 44.1, 126.01, 126.53, 128.15, 128.95, 131.97, 152.04, 201.71. $m/z$: 257 [M]$^+$, 279 [M+Na]$^+$. HRMS Calcd for C$_{15}$H$_{16}$N$_2$O$_2$; 256.1212. Found 256.1210.
1^H NMR (200 MHz, CDCl₃) 1.42 (s, 9H), 1.71 (m, 2H), 2.05 (m, 2H), 4.86 (m, 1H), 5.37 (m, 1H), 7.03 (m, 1H), 7.17 (m, 5H). ^13C NMR (200 MHz, CDCl₃) 23.75, 29.15, 38.51, 57.82, 73.41, 108.67, 125.37, 126.53, 128.44, 136.96, 151.38. \( m/z \) (ES-MS): 282 [M+Na]^+. HRMS Calcd for C₁₆H₂₁NO₂, 259.1572. Found 259.1569.
$^1$H NMR (200 MHz, CDCl$_3$) 1.71 (m, 2H), 2.15 (m, 2H), 4.98 (m, 1H), 5.35 (m, 1H), 7.44 (m, 1H). $^{13}$C NMR (200 MHz, CDCl$_3$) 29.68, 39.40, 55.41, 195.10, 123.02, 125.49, 126.83, 128.39, 137.88, 152.99. m/z (ES-MS): 302 [M+Na]$^+$. HRMS Calcd for C$_{18}$H$_{17}$NO$_2$ Exact Mass: 279.1259 Found 279.1256.
$^1$H NMR (200 MHz, CDCl$_3$): 1.77 (m, 4H), 2.30 (m, 5H), 4.76 (d, $J = 8.5$, 2H), 7.30 (m, 5H), 7.85 (m, 1H), 8.00 (m, 1H), 9.62 (s, 1H), 9.76 (s, 1H). $^{13}$C NMR (200 MHz, CDCl$_3$): 21.8, 24.5, 34.3, 33.9, 49.7, 123.9, 125.0, 125.6, 125.9, 127.0, 128.2, 128.3, 131.9, 133.0, 133.8, 200.7. $m/z$: ES/MS 292 [M+Na]$^+$. Anal. Calcd for C$_{17}$H$_{19}$NO$_2$: C, 75.81; H, 7.11; N, 5.20. Found: C, 75.89; H, 7.09; N, 5.19.