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Supplementary information

Environmentally benign and effective syntheses of N-substituted carbamates via alcoholysis of disubstituted ureas over TiO_2/SiO_2 catalyst*

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Methyl cyclohexylcarbamate:

White solid, m.p.=73-74 ^{o}C (the melting point of methyl cyclohexylcarbamate in the single-crystal form was also reported in reference [1])

GC-MS, *m/z*: M^{+}=157 (17%), M^{+}-43=114 (100%), M^{+}-15=142 (7%); ^{1}H NMR ($CDCl_3$) : 1.1 (p, 3H), 1.4 (p, 2H), 1.6(p, 3H), 1.99 (p, 2H), 3.4 (m, 1H), 3.6 (m, 3H), 4.5 (m, 1H); ^{13}C NMR (CD_3COCD_3), δ: 25.74, 26.28, 33.89, 50.72, 51.45,156.81 ppm.

Ethyl cyclohexylcarbamate:

White solid, m.p.=53-54 ^{o}C

GC-MS, *m/z*: M^{+}=171 (18%), M^{+}-43=128 (100%), M^{+}-29=142 (28%); ^{1}H NMR ($CDCl_3$) : 1.1 (p, 3H), 1.2 (p, 3H), 1.3 (p, 2H), 1.5(p, 1H), 1.6 (p, 2H), 1.99 (p, 2H), 3.4 (m, 1H), 4.0 (m, 2H), 4.5 (m, 1H); ^{13}C NMR (CD_3COCD_3), δ: 15.01, 25.74, 26.29, 33.89, 50.57, 60.24, 156.38 ppm.

Butyl cyclohexylcarbamate:

White solid, m.p.=52-53 ^{o}C

GC-MS, *m/z*: M^+=199 (16%), M^+-43=156 (100%), M^+-57=142 (44%); ^{1}H NMR ($CDCl_3$) : 1.1 (p, 3H), 1.2 (p, 3H), 1.3 (p, 4H), 1.5(p, 3H), 1.6 (p, 2H), 1.99 (p, 2H), 3.4 (m, 1H), 4.0 (m, 2H), 4.5 (m, 1H); ^{13}C NMR (CD_3COCD_3), δ: 13.98, 19.73, 25.73, 26.32, 32.02, 33.91, 50.61, 64.19, 156.51 ppm.

EXPERIMENTAL

The melting point was measured by a SGW X-4 microscopic melting point instrument with a digital thermometer. GC-MS analysis was conducted on a HP 6890/5973 GC-MS with chemstation containing a NIST Mass Spectral Database. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker AMX 400 MHz spectrometer, and the chemical shifts were reported relative to TMS.

REFERENCE

1 Methyl N-cyclohexylcarbamate, Chen Ying, Zhang Haitao, Zhou Lina, Acta Cryst. (2006), E62, o3757-o3758.