

SUPPORTING INFORMATION

TriazineTriamine derived Porous Organic Polymer supported Copper Nanoparticles (Cu-NPs@TzTa-POP): An efficient catalyst for synthesis of N-methylated products *via* CO₂ fixation and primary carbamates from alcohols and urea

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Materials

All chemicals were purchased from commercially available sources and used as received without further purification. Solvents were distilled and dried through standard methods before use.

Characterization Techniques

Fourier-transform infrared spectroscopy was carried out on a Perkin-Elmer FTIR 783 spectrophotometer using KBr pellets. Bruker D8 Advance X-ray diffractometer using Cu-K_α radiation ($\lambda = 1.5418 \text{ \AA}$) operating at 40 kV and 40 mA was utilized to record powder X-ray diffraction (PXRD) data of samples. Thermal stability of samples was monitored by Mettler Toledo TGA/DTA 851 instrument. Bruker AMX- 400 instrument was operated for ¹H NMR spectra. To measure the metal loading in catalyst (both fresh and used catalyst) AAS analysis was performed on Spectra- AA 240 (Agilent Technologies). UV-Vis absorption spectra of the Bpx-POP and catalyst were recorded on SHIMADZU, UV-2600 UV-Vis spectrometer with a standard 1 cm x 1 cm cuvette. Transmission Electron Microscope (TEM) [JEOL JEM 2100] was used to obtain the morphological information of the sample. The N₂ adsorption-desorption analysis of catalyst sample was conducted by using a BET Surface Analyzer [QUANTACHROME ASIQC602-5]. X-ray photoelectron spectroscopy was executed by using an Omicron Nanotechnology GmbH XPS machine.

Experimental Section:

Synthesis of Triazine Triamine based Porous Organic Polymer (TzTa-POP)

A solution of 0.01 mol 1,3,5-Triazine-2,4,6-triamine (melamine) in 20 ml dioxane was added to a solution of 0.04 mol of paraformaldehyde in 20 ml dioxane. The total reaction mixture was kept in ice to make the temperature around 5°C. In this mixture, 2 drops of 1M NaOH was added carefully and allowed to stir for 90 minutes. After the time period, 0.033 mol of 1,3,5-trihydroxybenzene in 20 ml dioxane was added and the reaction temperature was

increased to 80°C and allowed to stir for another 6h. The precipitation of this solution was centrifuged and washed thoroughly with water and acetone. Finally, the precipitate was dried under vacuum chamber for several days to get a powder like polymer (TzTa-Pop).

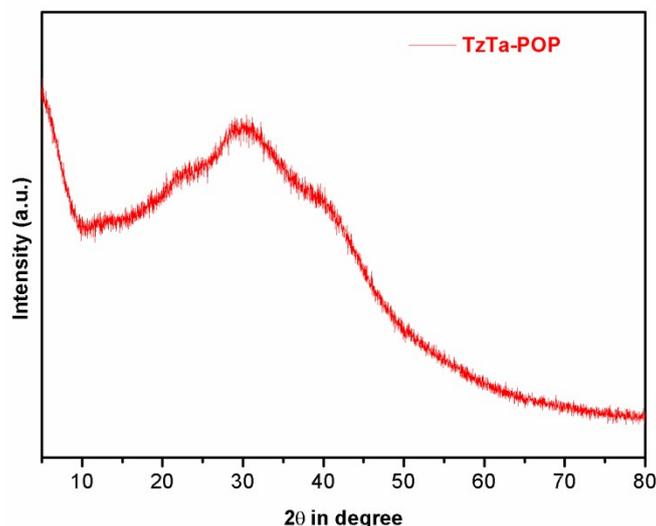


Figure S1: PXRD spectra of TzTa-POP.

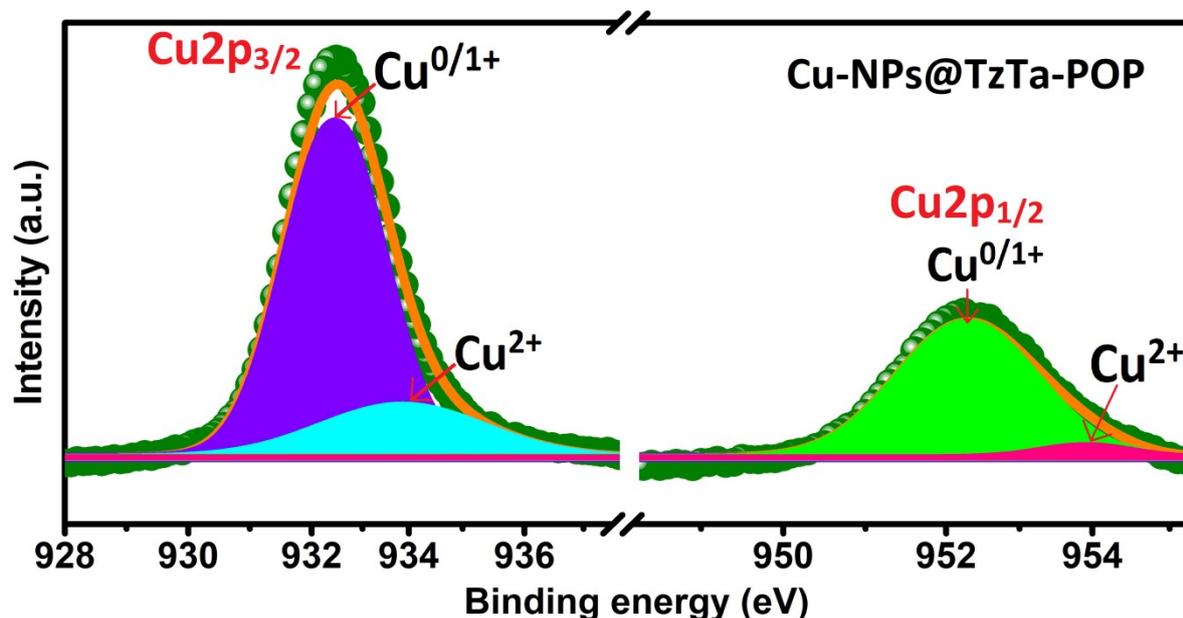


Fig. S2: Deconvoluted XPS spectrum of Cu 2p of Cu-NPs@TzTa-POP.

Figure S2 shows the detailed XPS of Cu 2p in Cu-NPs@TzTa-POP manifesting two recognizable peaks at binding energies of 932.4 and 952.2 eV, referring to the main Cu 2p_{3/2} and Cu 2p_{1/2}, respectively. These core levels could be resolved into two, Cu (0)/Cu (I) as a major and Cu(II) as minor species. In the Cu 2p spectrum, no satellite peaks were detected to somehow validate the purity of metallic Cu/Cu (II). We can attribute particular components comprising the line of Cu 2p_{3/2} to the states Cu⁰ and Cu⁺. Having regard to the XPS results

demonstrating the coexistence of Cu^{0/1+} as a major species and Cu²⁺ as a minor species on the surface of the as-obtained POP, one is willing to concede that such combined species of various oxidation states appear on the surface of the material. The formation of Cu NPs was assessed by XRD technique, which supports the synthesis of Cu NPs owing to the absence of Cu(I) diffraction peaks (i.e. Cu₂O). We can conclude that the analysis of XRD and XPS precisely indicates the chemical integrity of Cu NPs decorated POP.

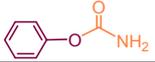
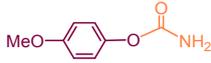
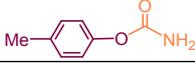
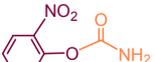
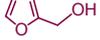
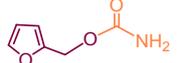
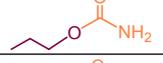
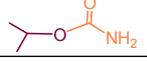
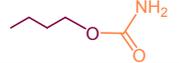
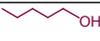
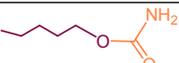
Table S1: Data for different runs of the catalytic N-methylation reaction^a

Amines	Product	Run 1 Conversion (isolated yield of product)	Run 2 Conversion (isolated yield of product)	Run 3 Conversion (isolated yield of product)	Mean value of conversion and (isolated yield of product)
		100 (98)	100 (96)	100 (95)	100 (96.3)
		100 (97)	100 (94)	100 (95)	100 (95.3)
		95 (92)	93 (90)	95 (92)	94.3 (91.3)
		100 (96)	100 (93)	100 (92)	100 (93.7)
		67 (64)	65 (60)	63 (60)	65 (61.3)
		100 (95)	100 (95)	100 (93)	100 (94.3)
		100 (94)	100 (90)	100 (92)	100 (92.0)

^aReaction conditions: substrate (5 mmol), PMHS (2 equiv.), Cu-NPs@TzTa-POP cataly at (20 mg, 0.015 mmol based on copper metal), CO₂ (1 atm), MeCN (5 ml), 80°C.

Table S2: Data for different runs of the catalytic primary carbamates formation reaction^a

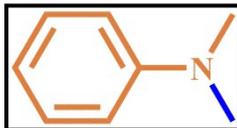
Alcohol	Product	Run 1 isolated yield of product	Run 2 isolated yield of product	Run 3 isolated yield of product	Mean value of isolated yield of product
		98	96	96	96.7
		98	97	95	96.7
		92	92	91	91.7
		85	80	82	82.3

		78	76	77	77.0
		82	80	83	81.7
		80	78	77	78.3
		56	55	58	56.3
		98	95	94	95.7
		72	74	72	72.7
		92	90	93	91.7
		90	88	87	88.3
		95	94	92	93.7
		98	94	95	95.7

^aReaction conditions: alcohols (5 mmol), urea (4 mmol), Cu-NPs@TzTa-POP catalyst (20 mg, 0.015 mmol based on copper metal), 1,4-dioxane (4 ml), 80°C, 4 h.

¹H NMR data of N-methylated products

N,N-dimethylaniline (2a)¹



¹H NMR (400 MHz, CDCl₃) δ 3.143 (s, 6H), 6.957-6.989 (m, 3H), 7.467-7.507 (t, *J*=8.0 Hz, 2H) ppm.

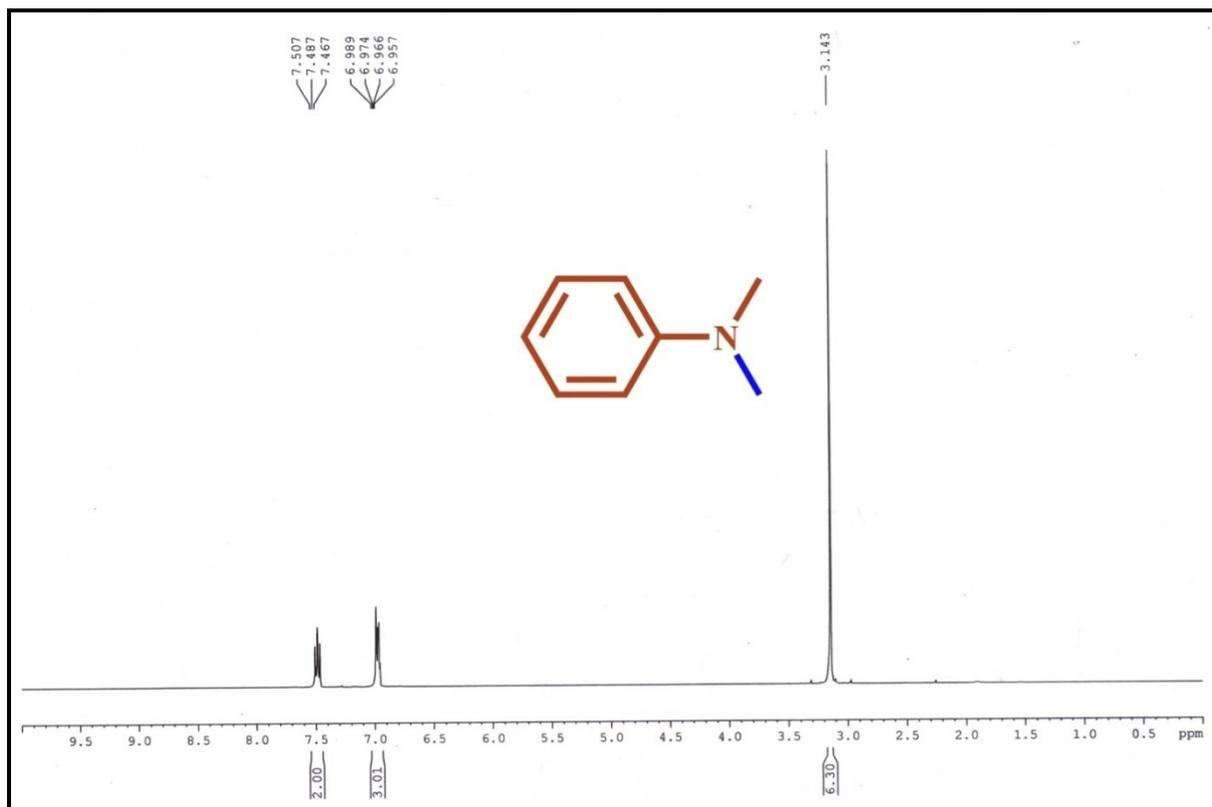
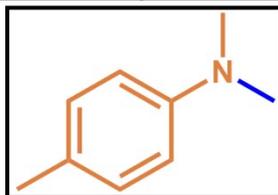


Figure S3: ¹H NMR spectra of N,N-dimethylaniline (**2a**)

*N,N,4-trimethylaniline (2b)*¹



¹H NMR (400 MHz, CDCl₃) δ 2.254 (s, 3H), 2.967 (s, 6H), 6.680-6.701 (d, *J*=8.4Hz, 2H), 7.045-7.065 (d, *J*=8.0 Hz, 2H) ppm.

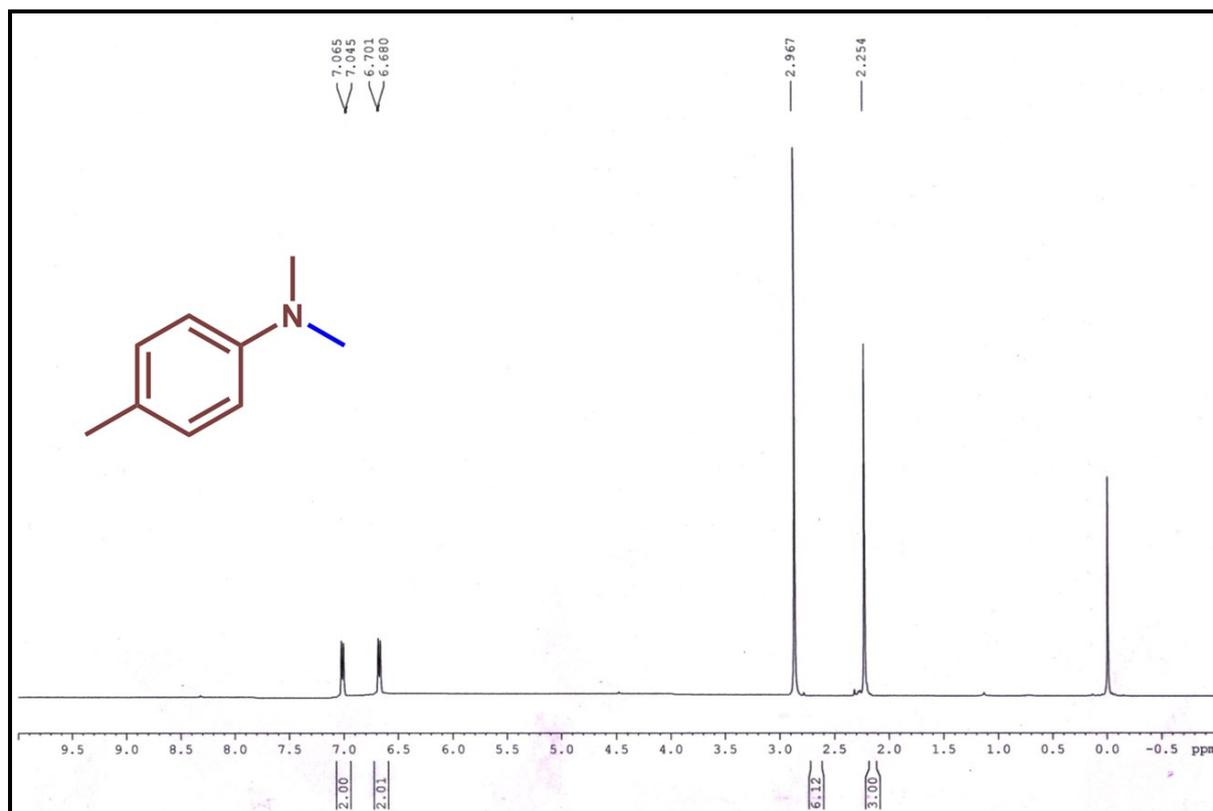
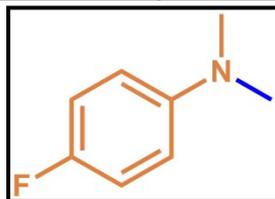


Figure S4: ¹H NMR spectra of *N,N,4-trimethylaniline* (2b).

4-fluoro-N-methylaniline (2c)²



¹H NMR (400 MHz, CDCl₃) δ 2.892 (s, 6H), 6.703-6.724 (d, *J*=8.4 Hz, 2H), 7.200-7.220 (d, *J*=8.0 Hz, 2H) ppm.

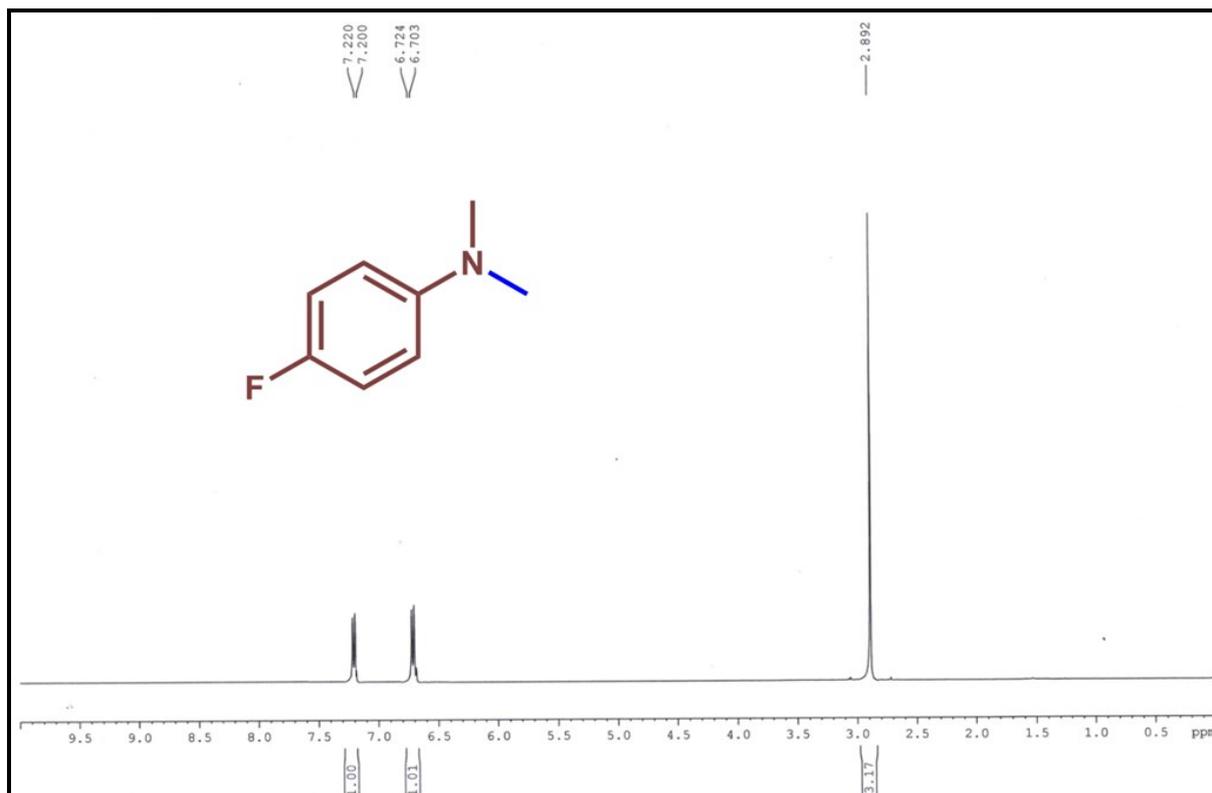
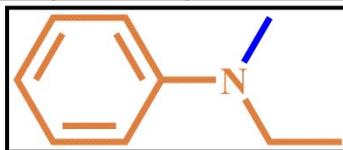


Figure S5: ¹H NMR spectra of 4-fluoro-N-methylaniline (**2c**).

N-ethyl-*N*-methylaniline (**2d**)¹



¹H NMR (400 MHz, CDCl₃) δ 1.096-1.132 (t, *J* = 7.2 Hz, 3H), 2.904 (s, 3H), 3.420-3.473 (m, 2H), 6.771-6.807 (t, *J* = 7.2 Hz, 3H), 7.249-7.287 (t, *J* = 7.2 Hz, 2H).

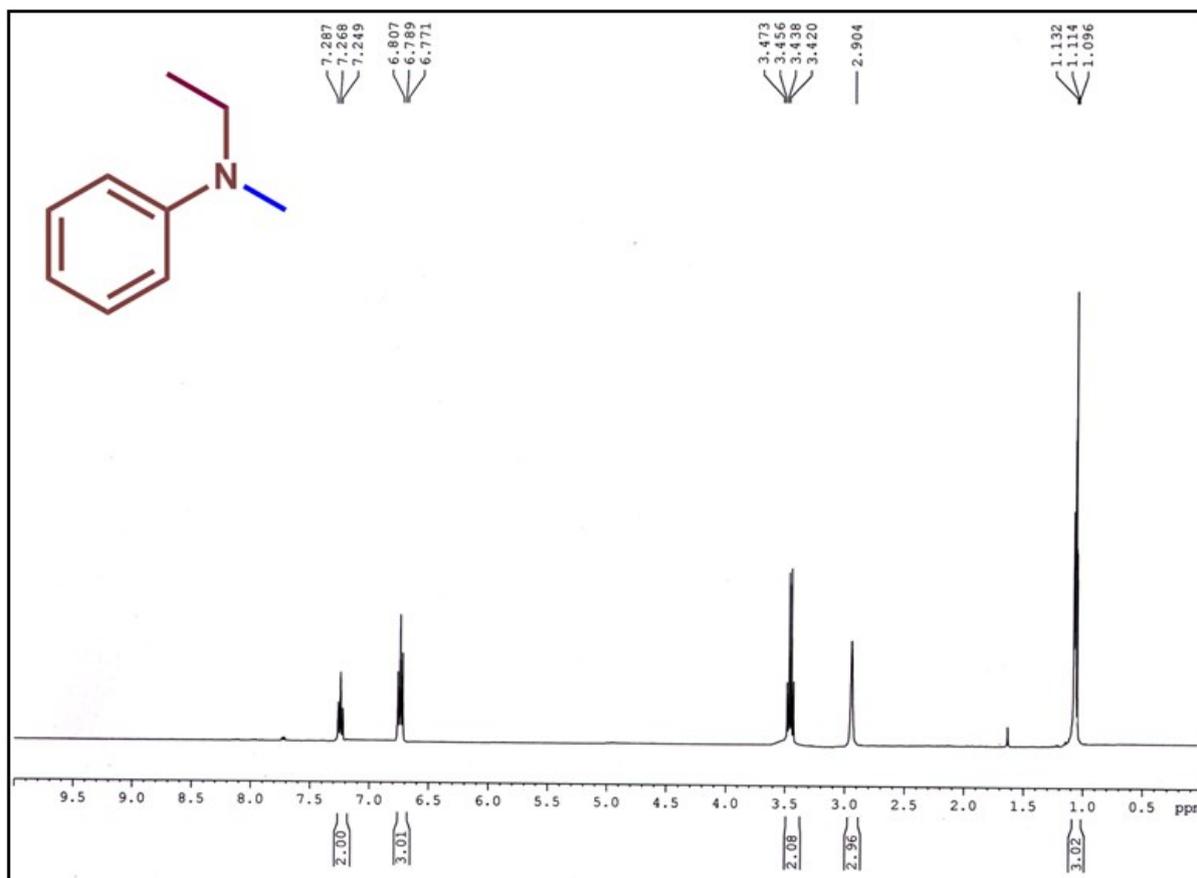
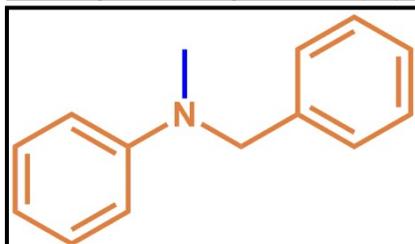


Figure S6: ¹H NMR spectra of *N*-ethyl-*N*-methylaniline (**2d**).

N-benzyl-*N*-methylaniline (**2e**)¹



¹H NMR (400 MHz, CDCl₃) δ 3.105 (s, 3H), 4.531 (s, 2H), 6.679-6.715 (t, *J* = 7.2 Hz, 3H), 7.198-7.337 (m, 7H).

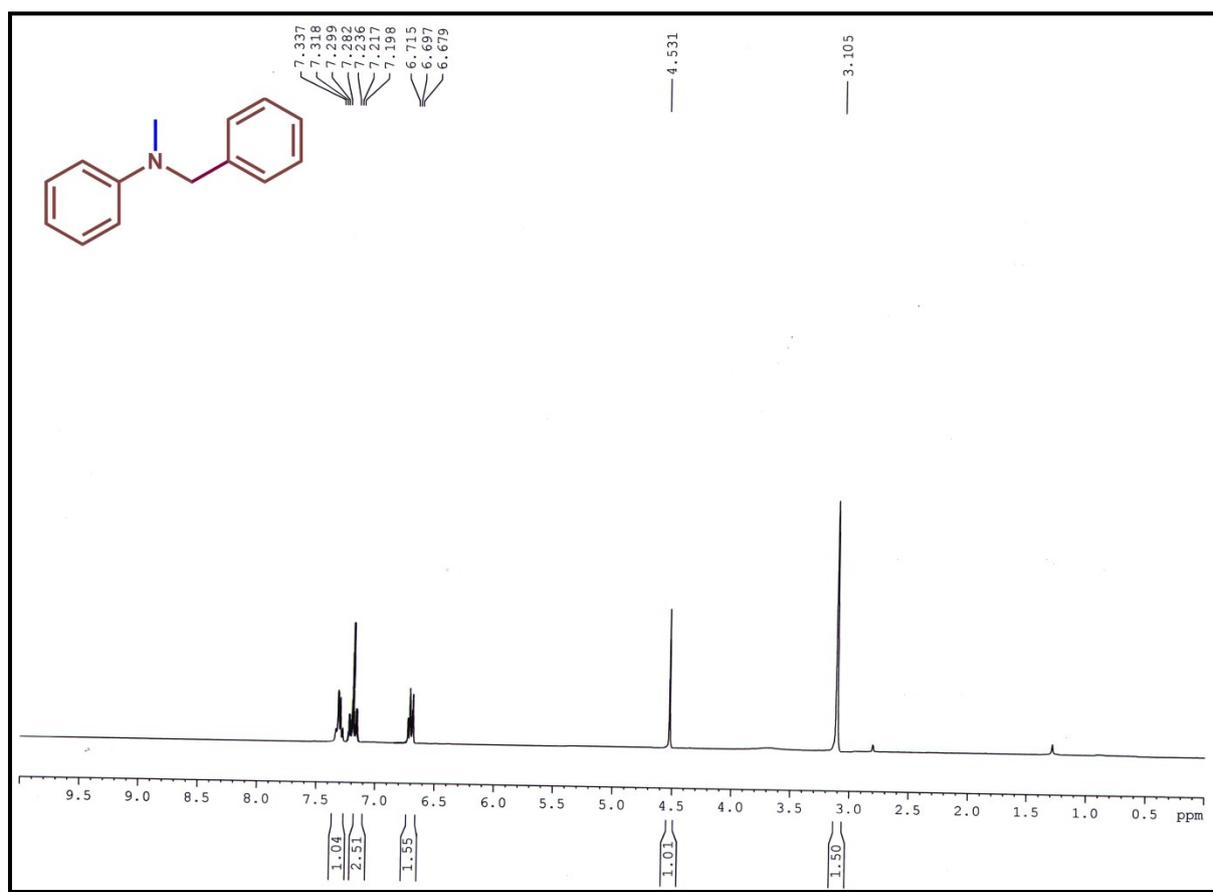
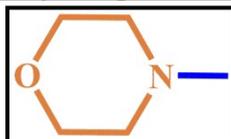


Figure S7: ¹H NMR spectra of *N*-benzyl-*N*-methylaniline (**2e**).

4-methylmorpholine (2f)¹



¹H NMR (400 MHz, CDCl₃) δ 2.207 (s, 3H), 2.321 (s, 4H), 3.614-3.637 (t, *J*=4.4 Hz, 4H) ppm.

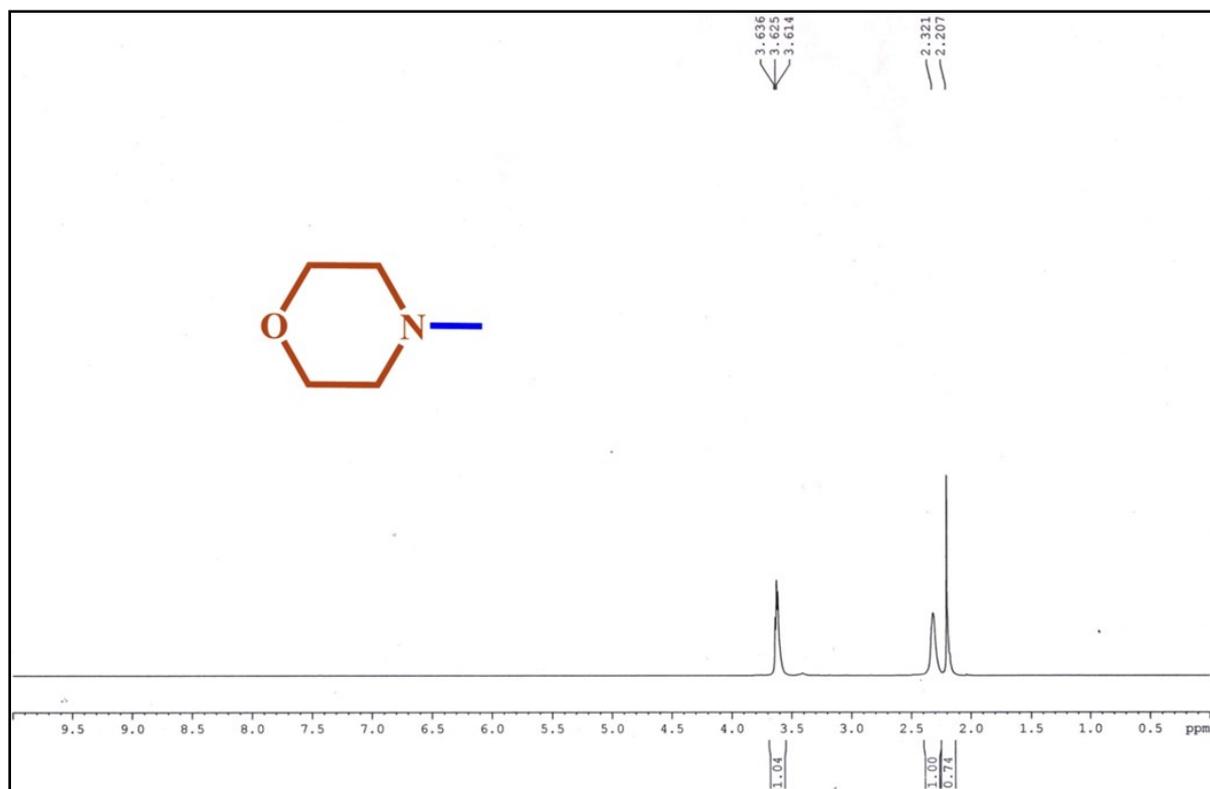
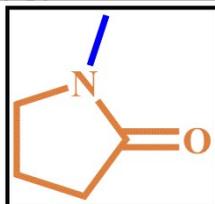


Figure S8: ¹H NMR spectra of 4-methylmorpholine (2f).

1-methylpyrrolidin-2-one (2g)



$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 2.000-2.087 (m, 2H), 2.350-2.400 (m, 2H), 2.847-2.857 (d, $J=4.0$ Hz, 3H), 3.391-3.437 (m, 2H) ppm.

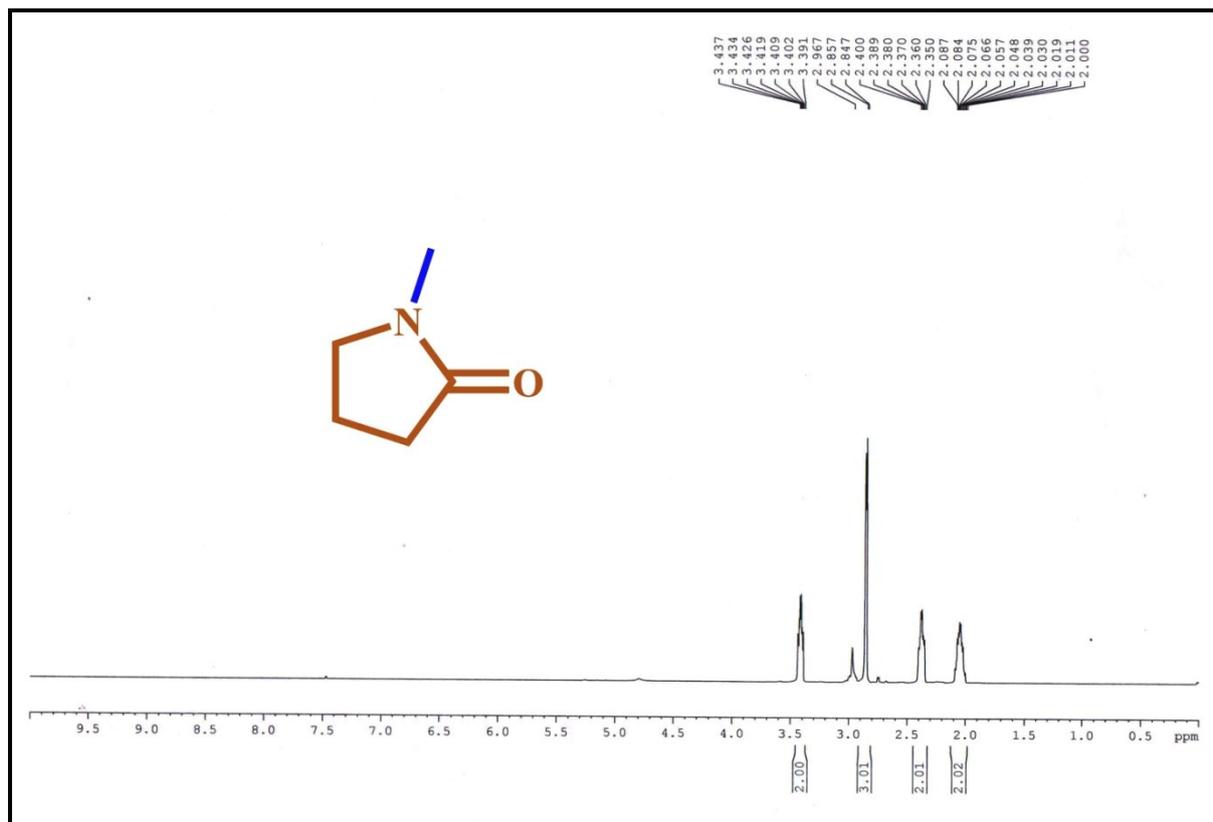
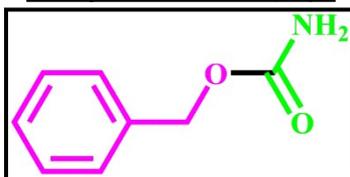


Figure S9: $^1\text{H NMR}$ spectra of 1-methylpyrrolidin-2-one (**2g**).

¹H NMR and FTIR data of carbamate products

Benzyl carbamate (4a)³



FTIR (KBr, ν , cm^{-1}): 3422-3332 (-NH stretching), 1694 ($>\text{C}=\text{O}$ stretching), 1610 (-NH bending), 1346 (-CN stretching), 1068 (C-O stretching).

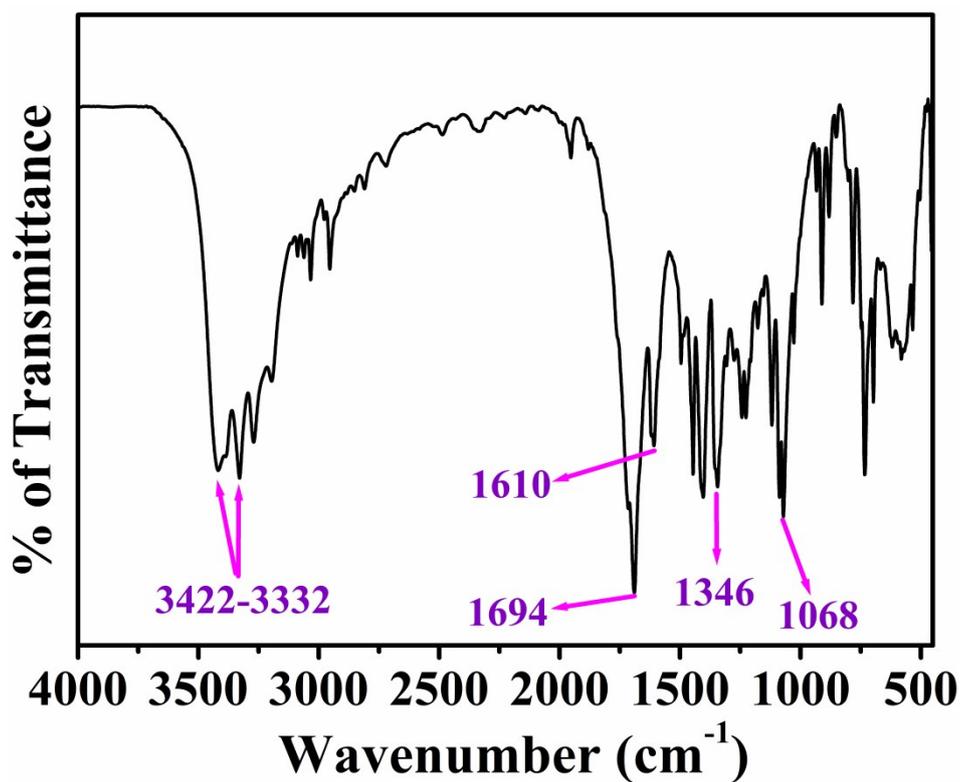


Figure S10: FTIR spectra of Benzyl carbamate (4a).

^1H NMR (400 MHz, CDCl_3) δ 4.913 (brs, NH_2), 5.108 (s, 2H), 7.391-7.455 (m, 3H), 7.541-7.559 (d, $J=7.2$ Hz, 2H) ppm.

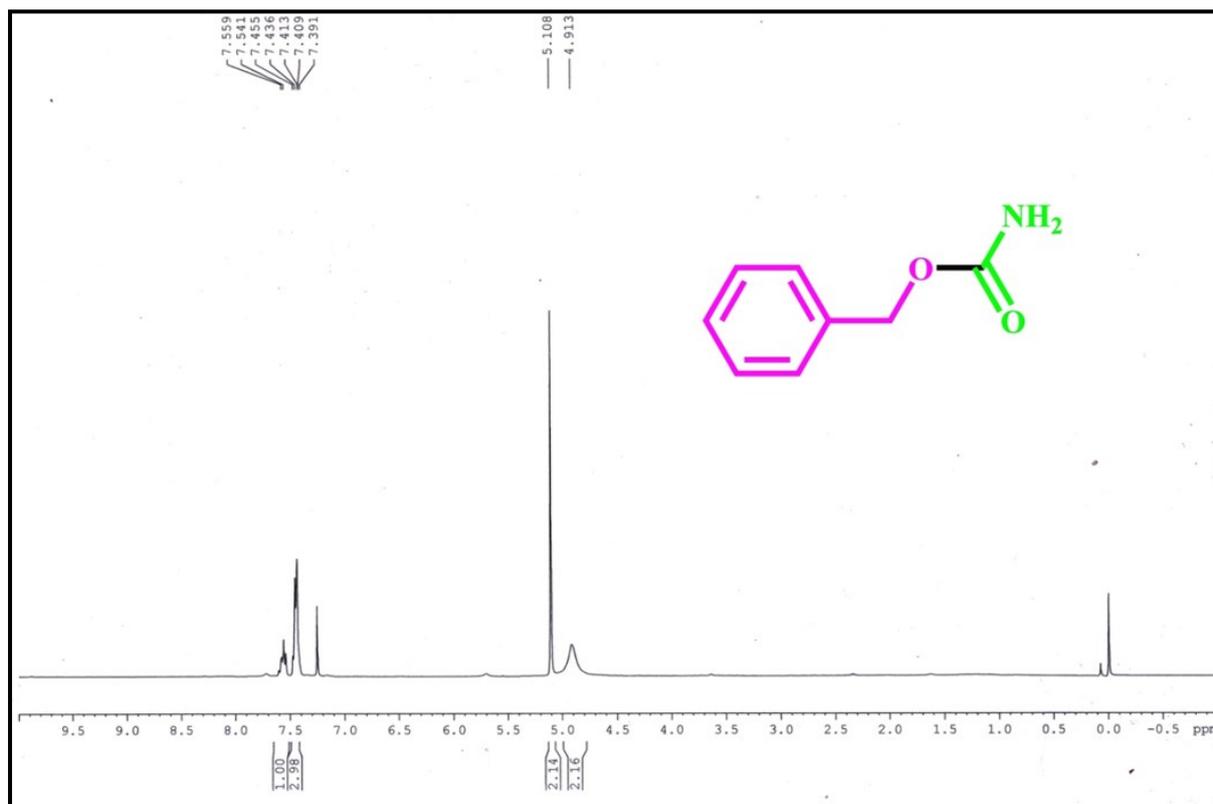
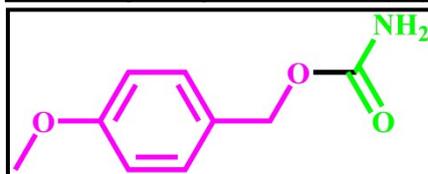


Figure S11: ^1H NMR spectra of Benzyl carbamate (**4a**).

4-methoxybenzyl carbamate (4b)



FTIR (KBr, ν , cm^{-1}): 3364 (-NH stretching), 1713 ($>\text{C}=\text{O}$ stretching), 1616 (-NH bending), 1307 (-CN stretching), 1036 (C-O stretching).

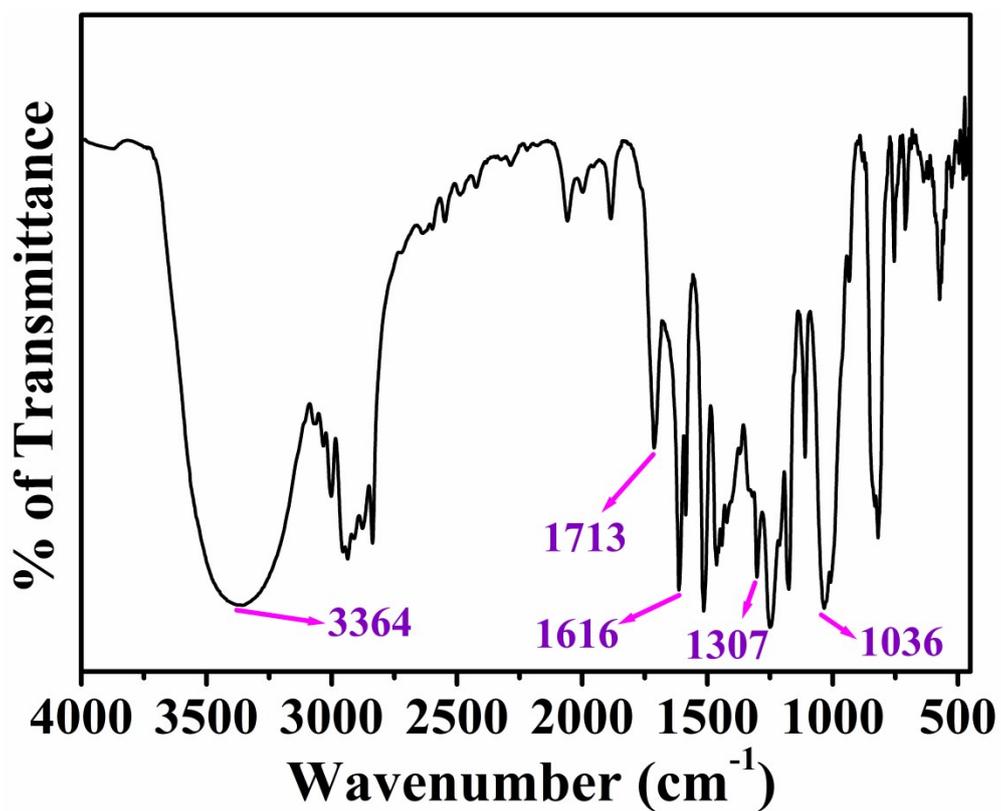


Figure S12: FTIR spectra of 4-methoxybenzyl carbamate (**4b**).

^1H NMR (400 MHz, CDCl_3) δ 3.735 (s, 3H), 5.099 (s, 2H), 6.820-6.841 (d, $J=8.4$ Hz, 2H), 7.192-7.213 (d, $J=8.4$ Hz, 2H), 7.605 (brs, NH_2) ppm.

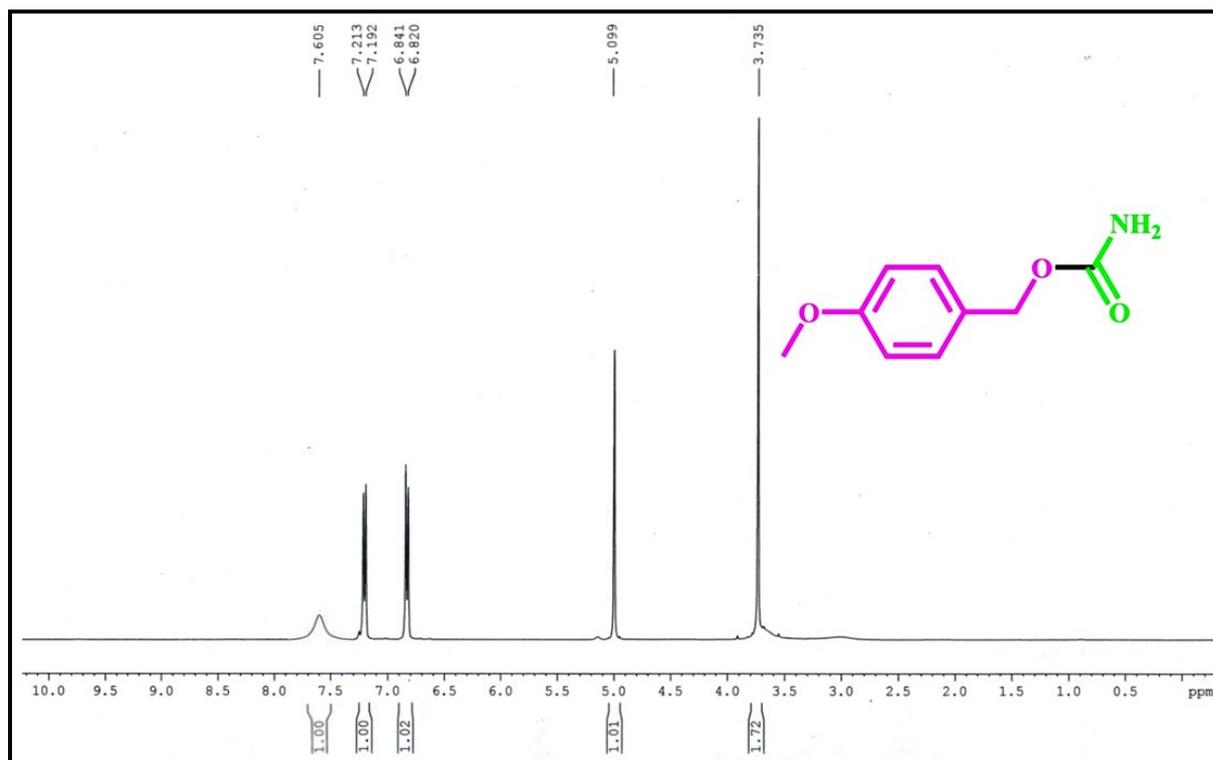
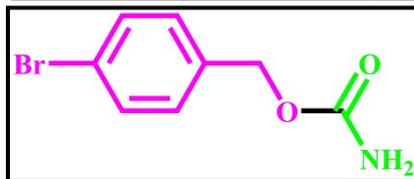


Figure S13: ^1H NMR spectra of 4-methoxybenzyl carbamate (**4b**).

4-bromobenzyl carbamate (4c)



FTIR (KBr, ν , cm^{-1}): 3442 (-NH stretching), 1707 ($>\text{C}=\text{O}$ stretching), 1610 (-NH bending), 1346 (-CN stretching), 1062 (C-O stretching).

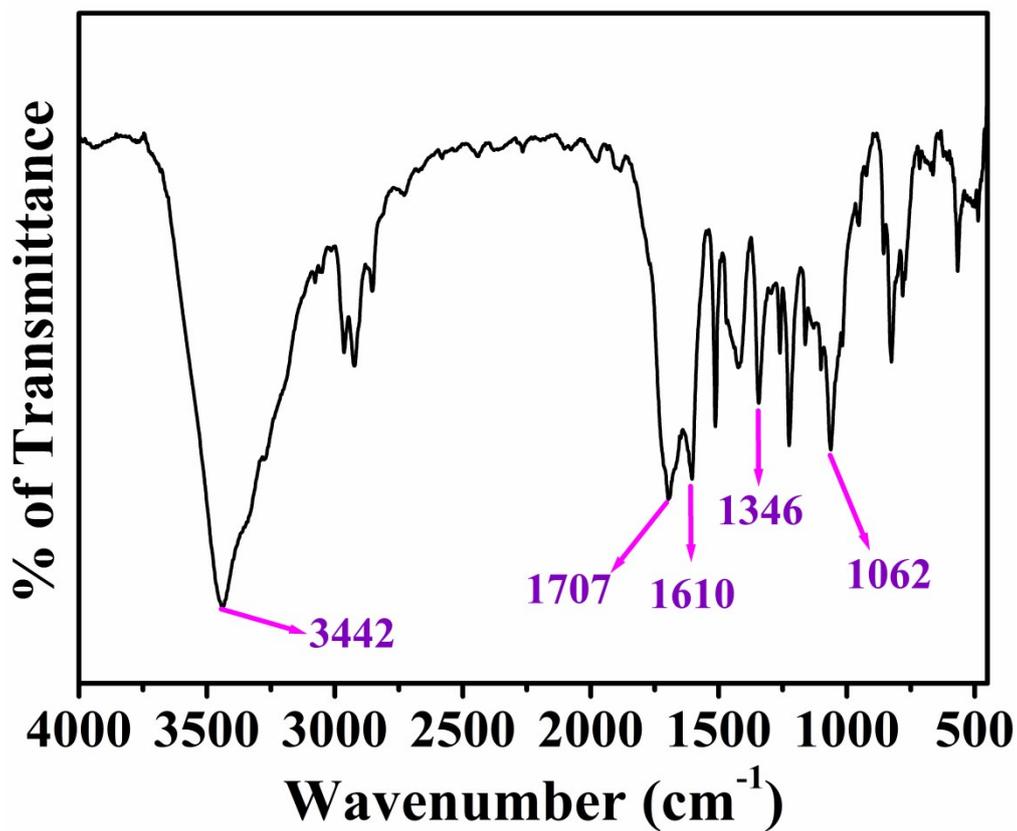


Figure S14: FTIR spectra of 4-bromobenzyl carbamate (**4c**).

^1H NMR (400 MHz, CDCl_3) δ 4.770 (brs, $-\text{NH}_2$), 5.064 (s, 2H), 7.026-7.069 (m, 2H), 7.329-7.364 (m, 2H) ppm.

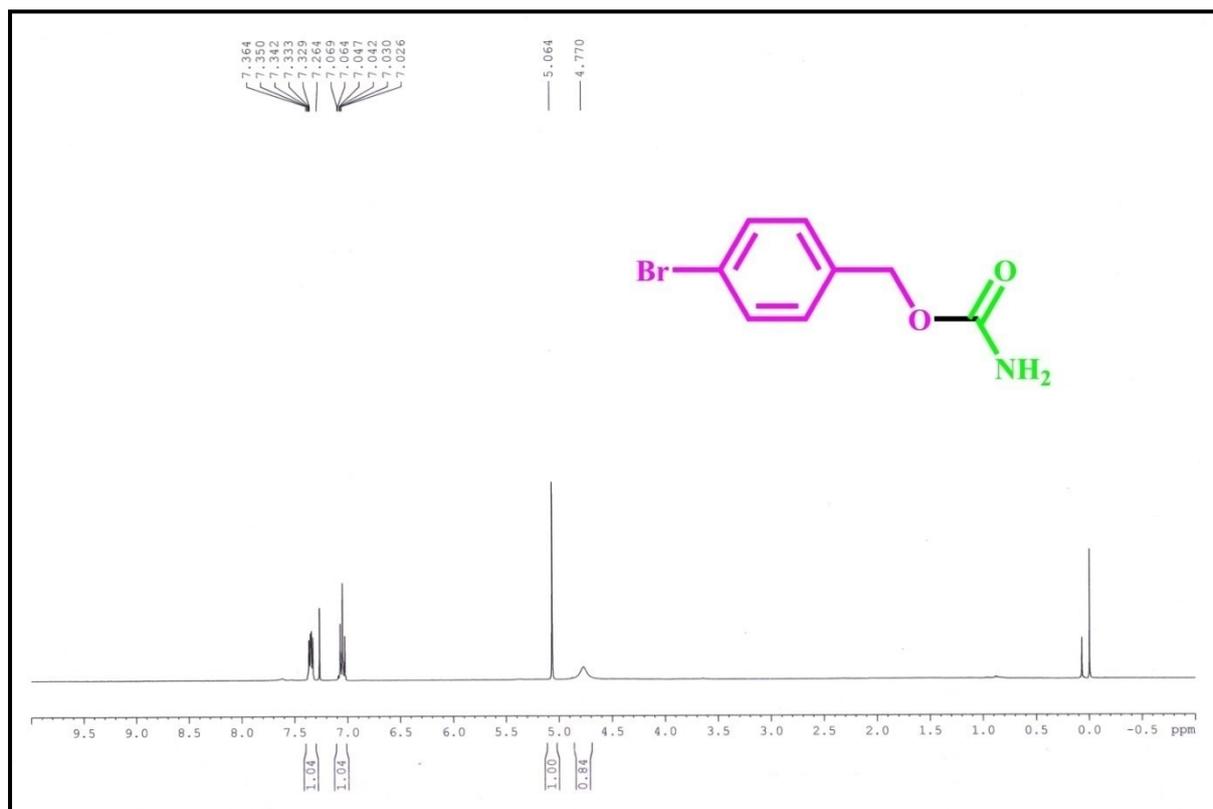
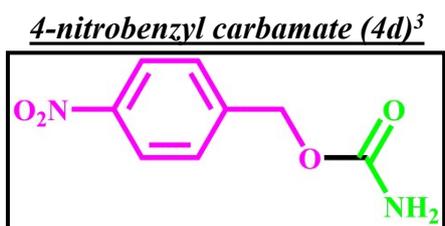


Figure S15: ^1H NMR spectra of 4-bromobenzyl carbamate (**4c**).



FTIR (KBr, ν , cm^{-1}): 3526 (-NH stretching), 1604 ($>\text{C}=\text{O}$ stretching), 1513 (-NH bending), 1339 (-CN stretching), 1056 (C-O stretching).

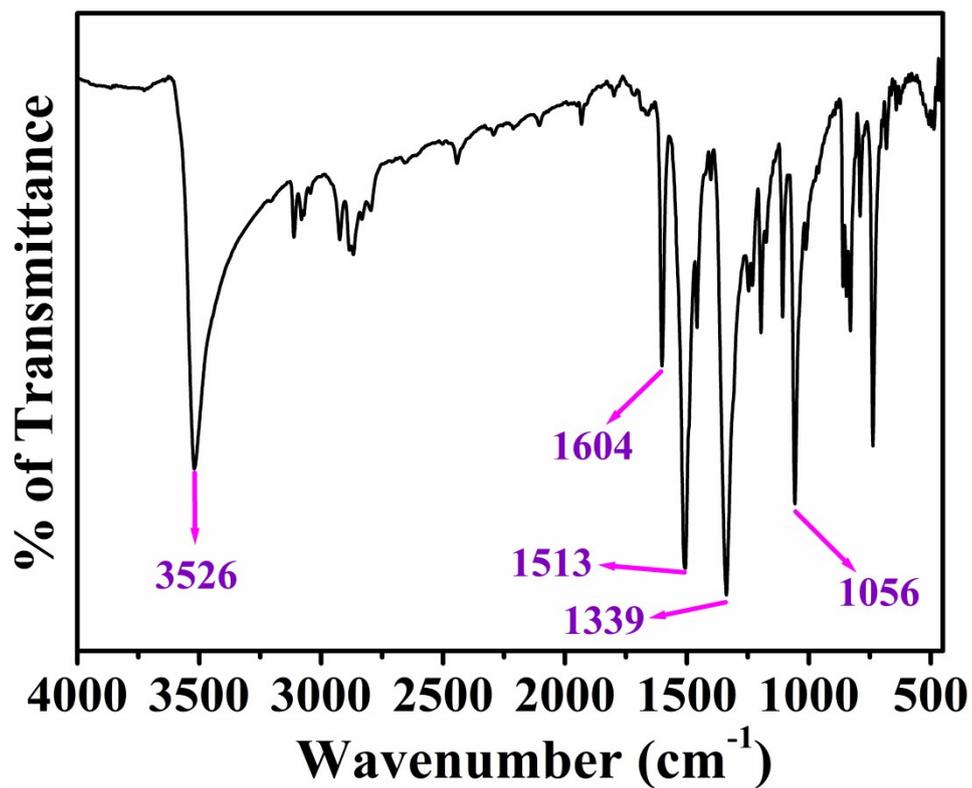


Figure S16: FTIR spectra of 4-nitrobenzyl carbamate (4d).

^1H NMR (400 MHz, CDCl_3) δ 4.834 (s, 2H), 6.673 (brs, $-\text{NH}_2$), 7.520-7.541 (d, $J=8.4$ Hz, 2H), 8.191-8.213 (d, $J=8.8$ Hz, 2H) ppm.

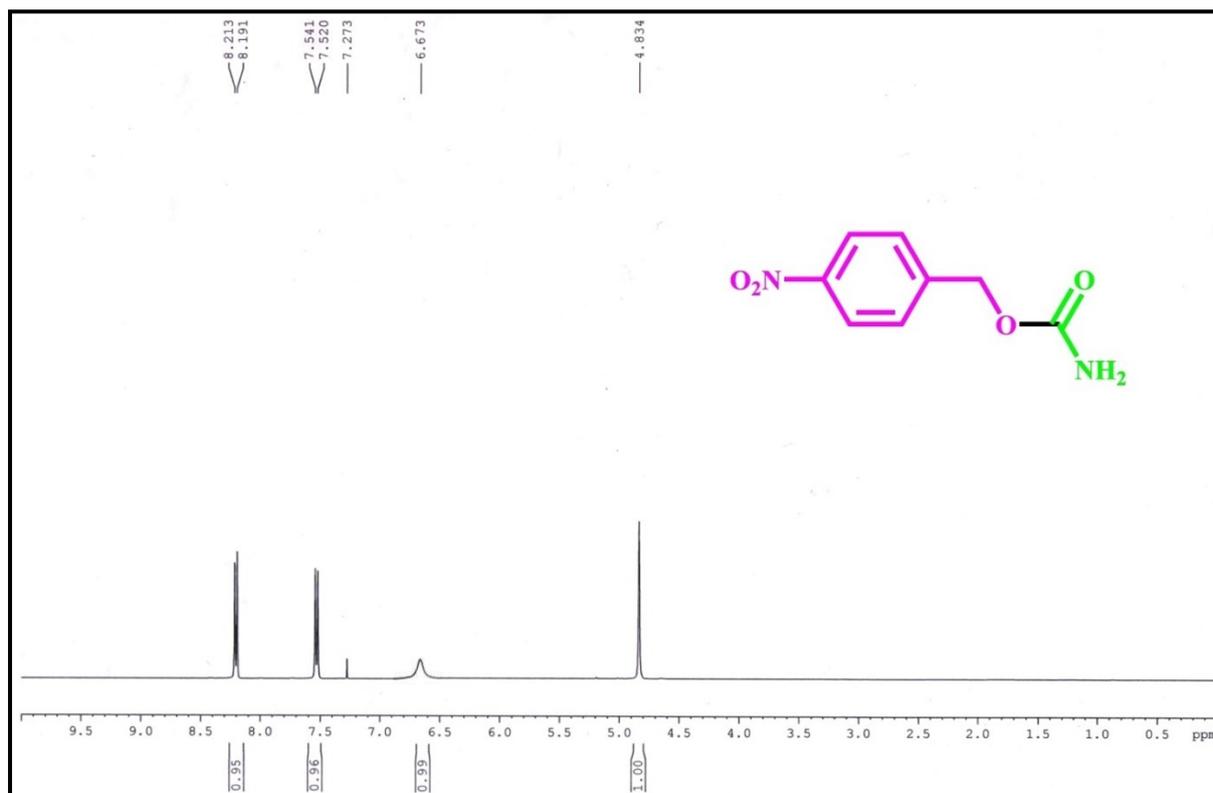
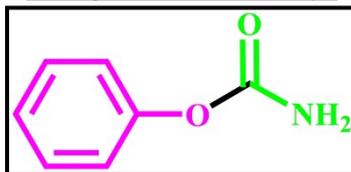


Figure S17: ^1H NMR spectra of 4-nitrobenzyl carbamate (**4d**).

*Phenyl carbamate (4e)*³



FTIR (KBr, ν , cm^{-1}): 3422-3339 (-NH stretching), 1707 ($>\text{C}=\text{O}$ stretching), 1616 (-NH bending), 1384 (-CN stretching), 1211 (C-O stretching).

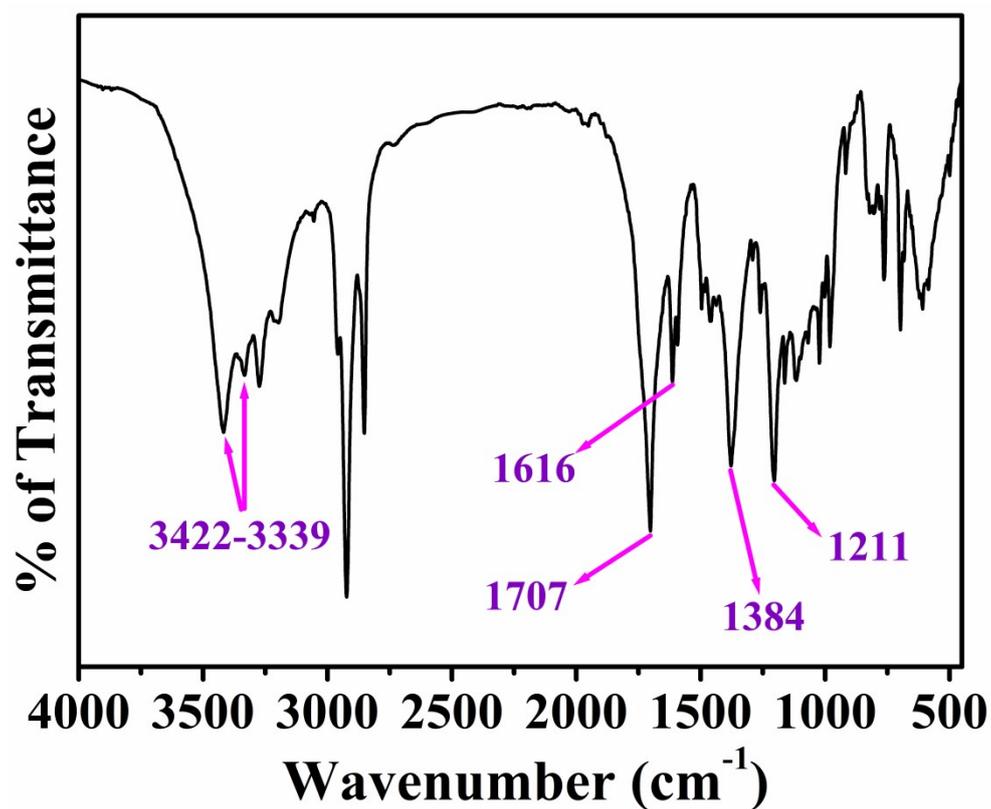


Figure S18: FTIR spectra of Phenyl carbamate (4e).

$^1\text{H NMR}$ (400 MHz, CDCl_3) δ 5.053 (brs, $-\text{NH}_2$), 7.058-7.100 (t, $J=8.4$ Hz, 2H), 7.132-7.190 (m, 1H), 7.258-7.324 (m, 2H) ppm.

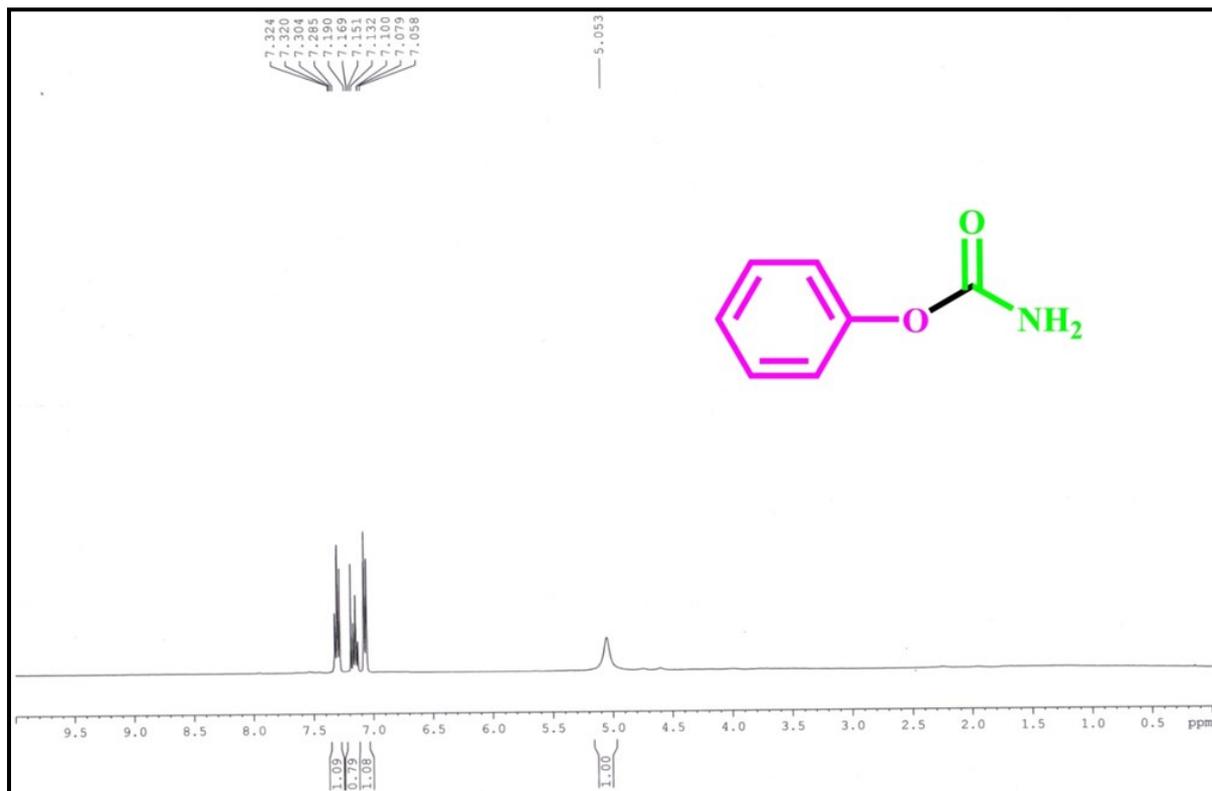
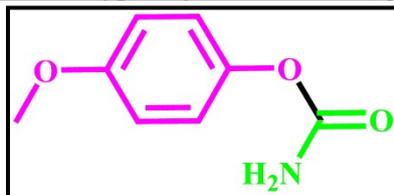


Figure S19: $^1\text{H NMR}$ spectra of Phenyl carbamate (4e).

4-methoxyphenyl carbamate (4f)⁴



FTIR (KBr, v, cm^{-1}): 3396 (-NH stretching), 1733 ($>\text{C}=\text{O}$ stretching), 1610 (-NH bending), 1365 (-CN stretching), 1043 (C-O stretching).

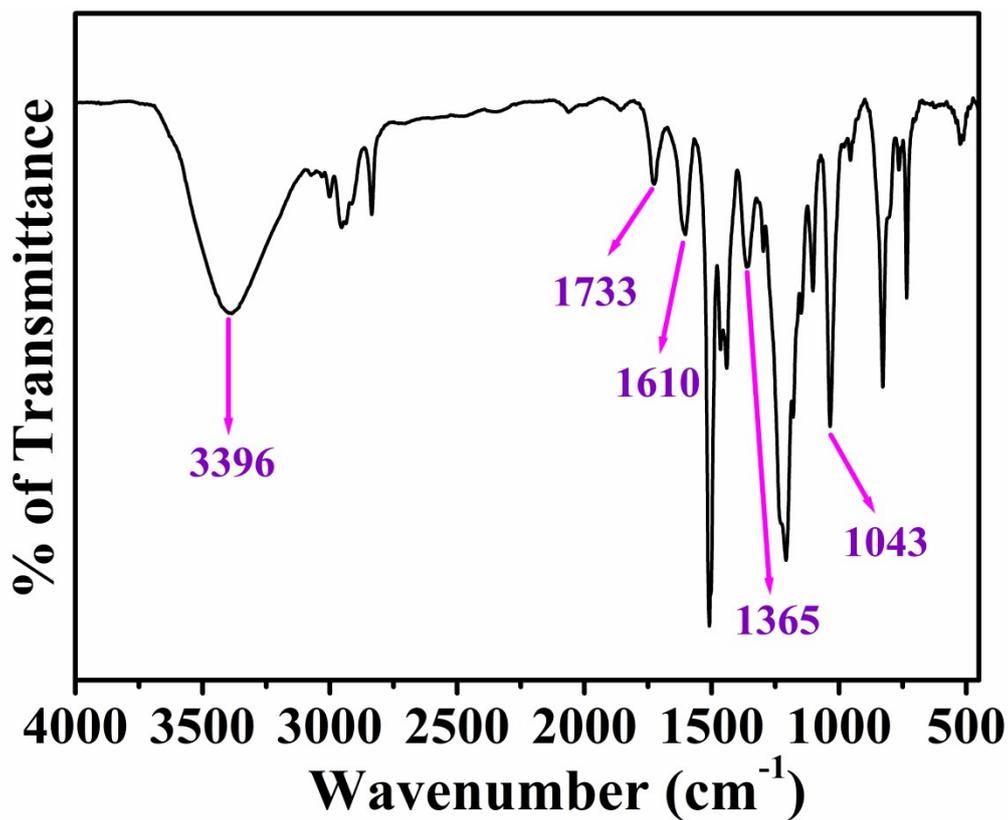


Figure S20: FTIR spectra of 4-methoxyphenyl carbamate (4f).

^1H NMR (400 MHz, CDCl_3) δ 3.750 (s, 3H), 5.750 (brs, $-\text{NH}_2$), 6.852-7.021 (m, 4H) ppm.

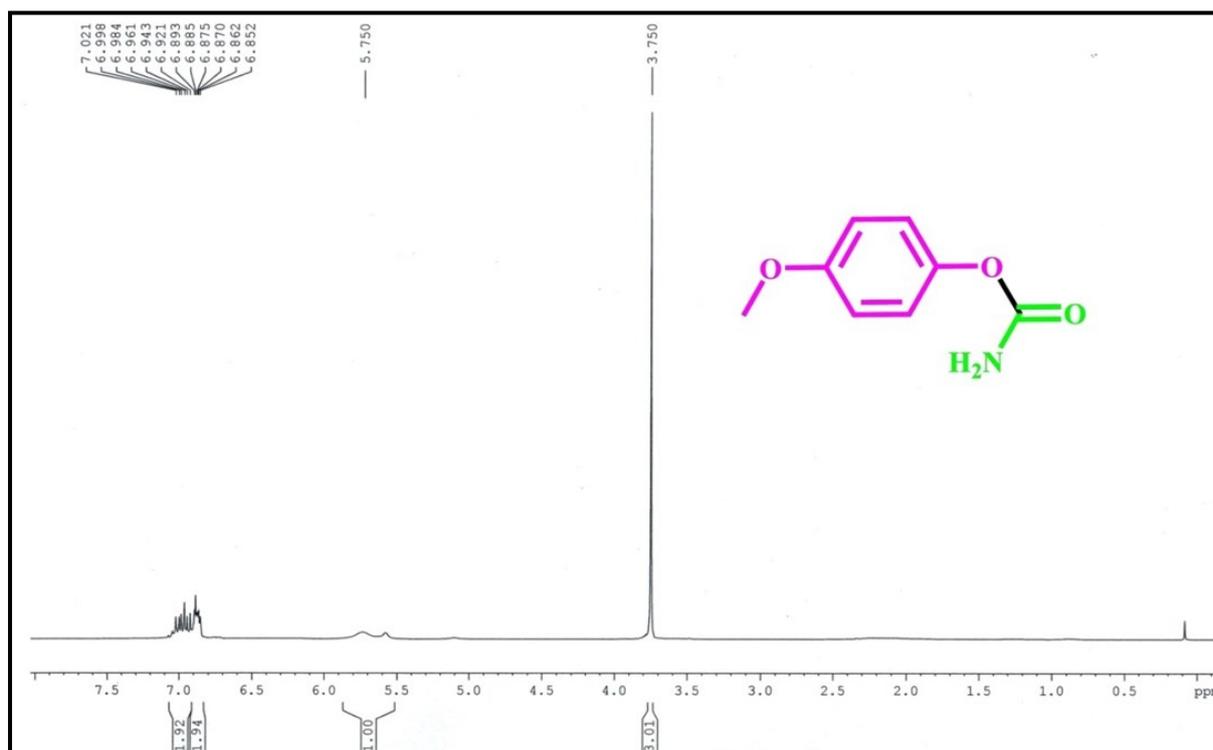
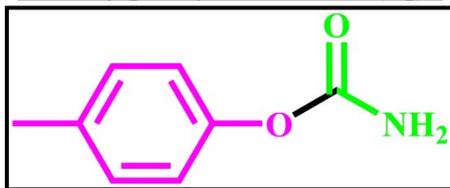


Figure S21: ^1H NMR spectra of 4-methoxyphenyl carbamate (**4f**).

4-methylphenyl carbamate (4g)⁴



FTIR (KBr, ν , cm^{-1}): 3390-3339 (-NH stretching), 1700 ($>\text{C}=\text{O}$ stretching), 1610 (-NH bending), 1365 (-CN stretching), 1216 (C-O stretching).

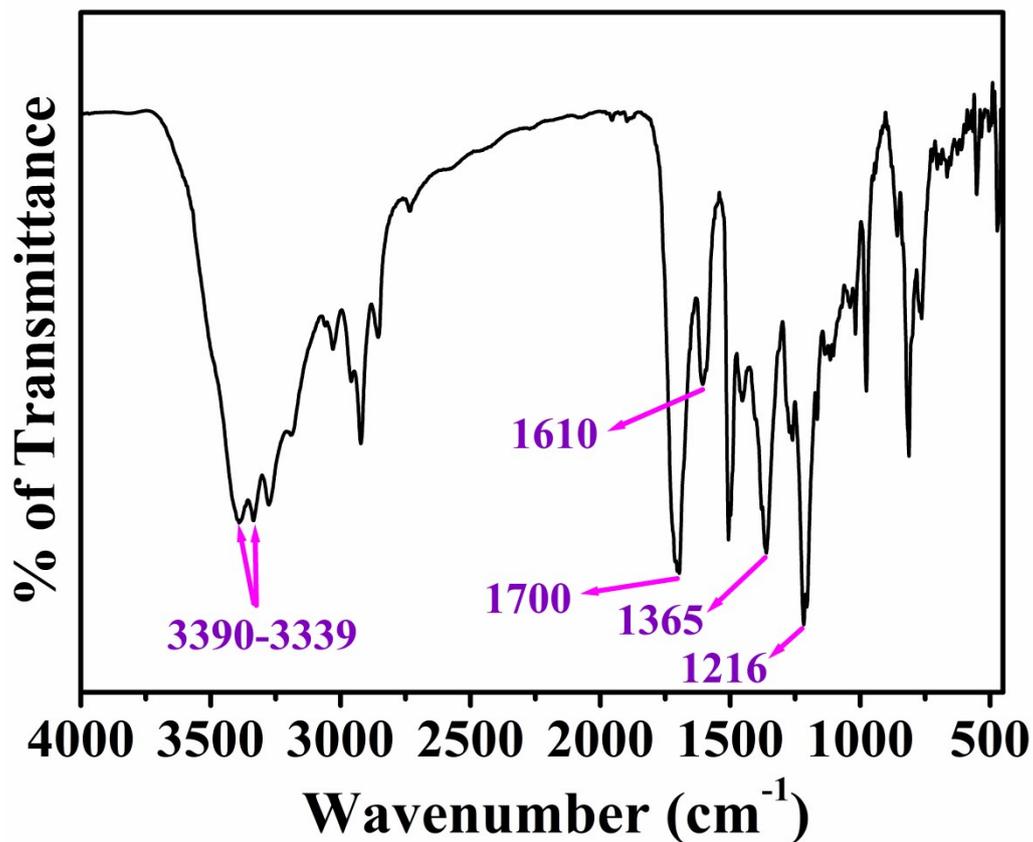


Figure S22: FTIR spectra of 4-methylphenyl carbamate (4g).

^1H NMR (400 MHz, CDCl_3) δ 2.244-2.262 (t, $J=3.6$ Hz, 3H), 4.957 (s, 1H), 6.996-7.021 (t, $J=8$ Hz, m, 2H), 7.055-7.092 (m, 2H) ppm.

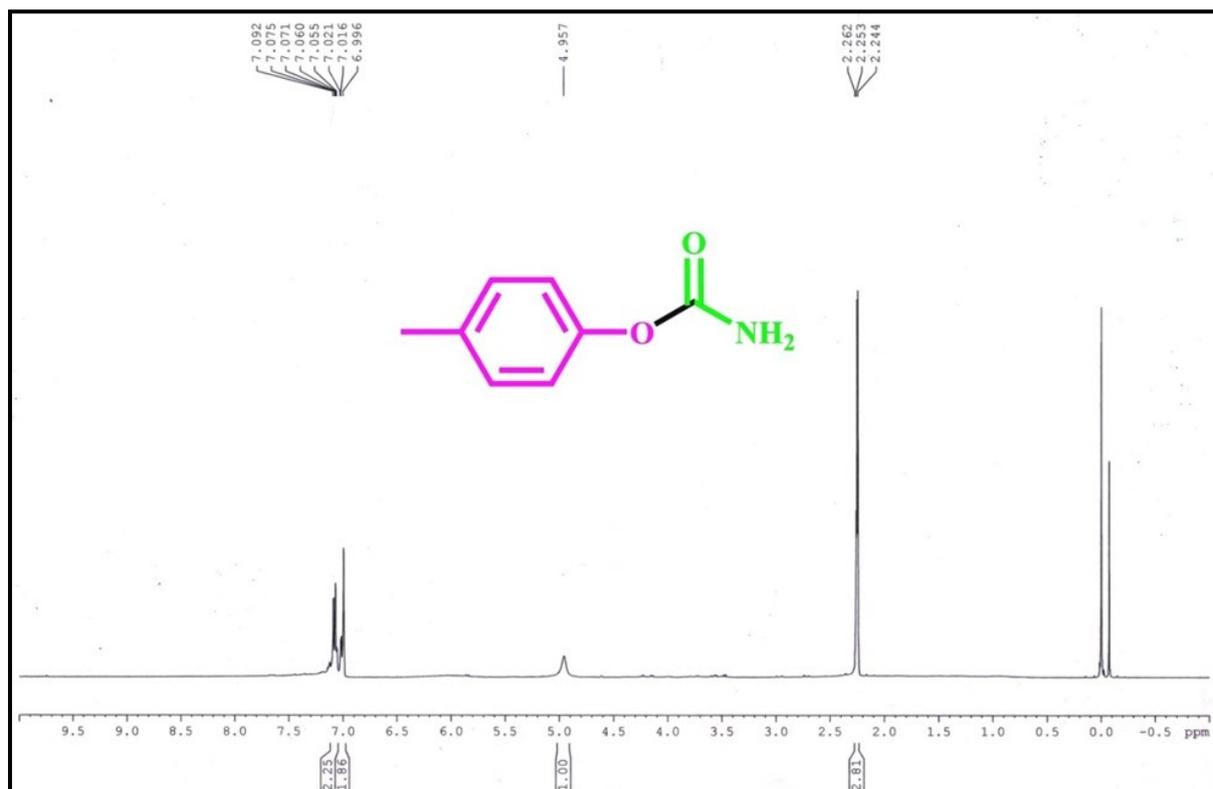
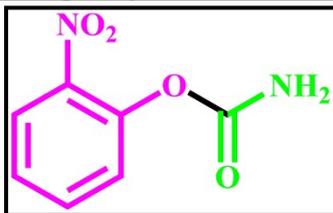


Figure S23: ^1H NMR spectra of 4-methylphenyl carbamate (**4g**).

2-nitrophenyl carbamate (4h)



FTIR (KBr, ν , cm^{-1}): 3255 (-NH stretching), 1745 ($>\text{C}=\text{O}$ stretching), 1616 (-NH bending), 1262 (-CN stretching), 1067 (C-O stretching).

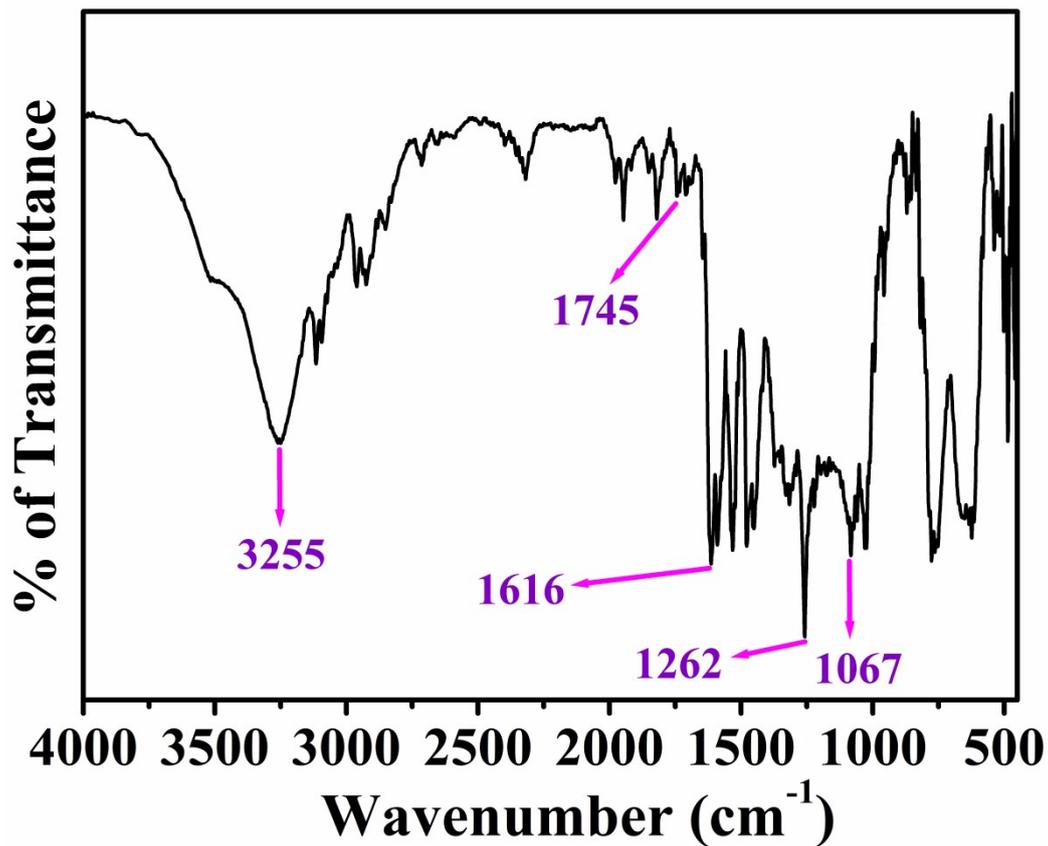


Figure S24: FTIR spectra of 2-nitrophenyl carbamate (4h).

^1H NMR (400 MHz, CDCl_3) δ 6.978-7.017 (t, $J=7.6$ Hz, 1H), 7.151-7.172 (d, $J=8.4$ Hz, 1H), 7.570-7.612 (m, 1H), 8.096-8.120 (m, 1H), 10.515 (brs, $-\text{NH}_2$) ppm.

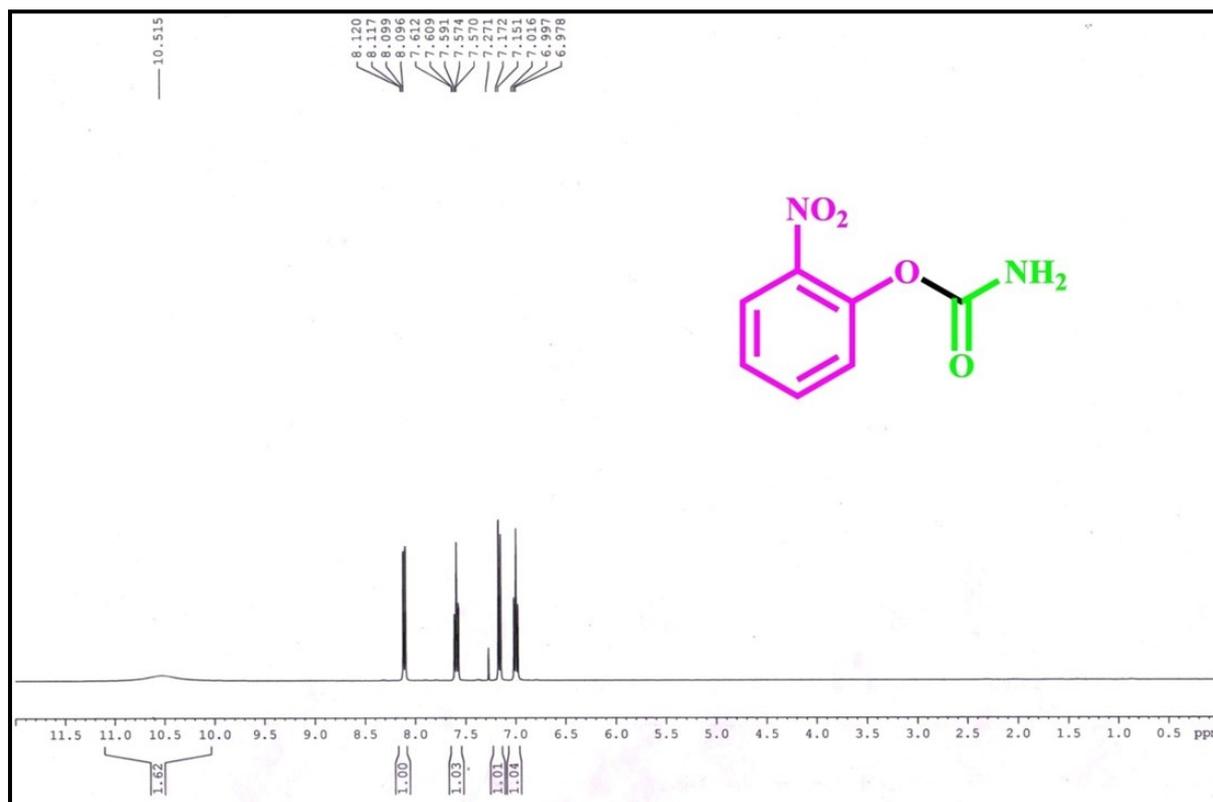
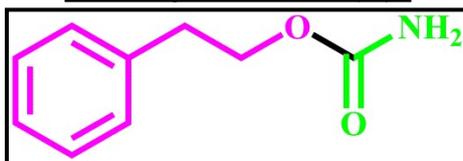


Figure S25: ^1H NMR spectra of 2-nitrophenyl carbamate (**4h**).

*Phenethyl carbamate (4i)*³



FTIR (KBr, ν , cm^{-1}): 3358 (-NH stretching), 1719 ($>\text{C}=\text{O}$ stretching), 1610 (-NH bending), 1346 (-CN stretching), 1075 (C-O stretching).

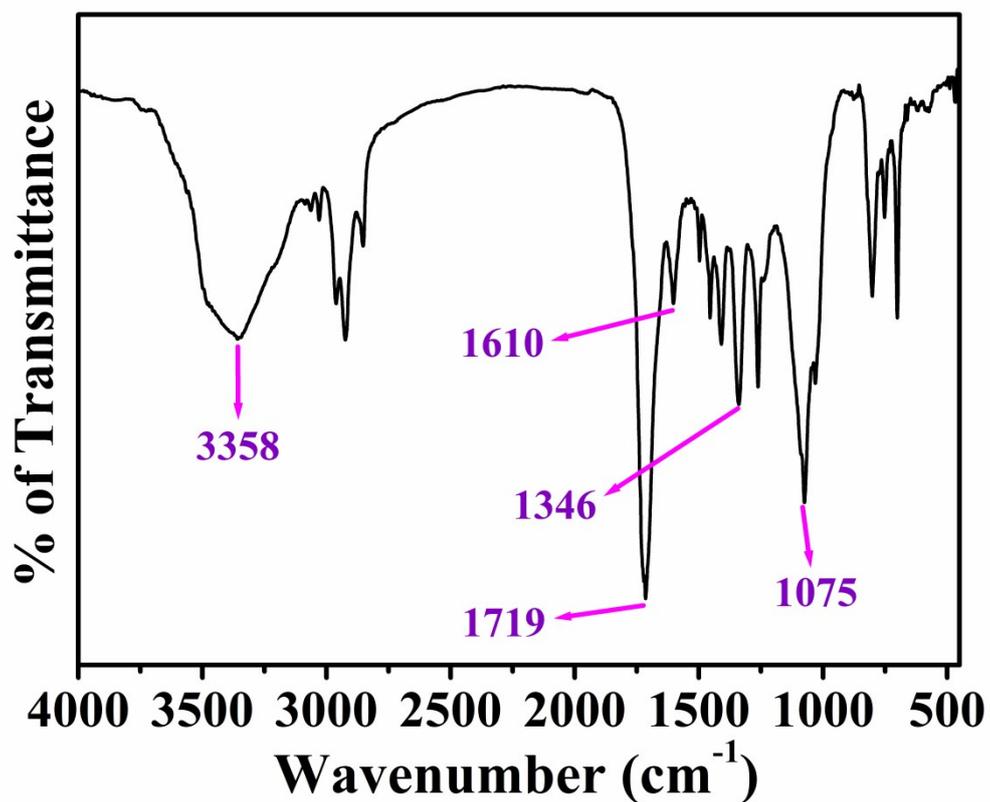


Figure S26: FTIR spectra of Phenethyl carbamate (4i).

^1H NMR (400 MHz, CDCl_3) δ 2.921-2.955 (m, $J=6.8$ Hz, 2H), 4.267-4.302 (t, $J=6.8$ Hz, 2H), 4.684 (brs, $-\text{NH}_2$), 7.210-7.314 (m, 5H) ppm.

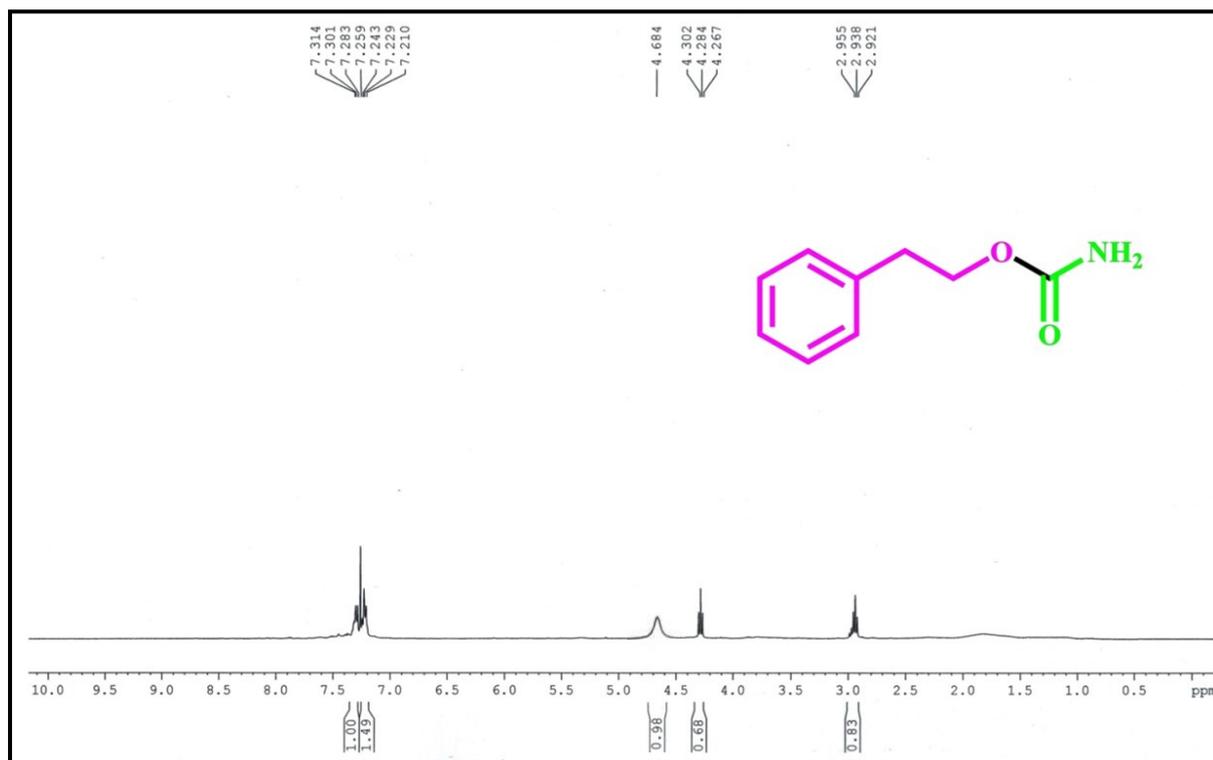
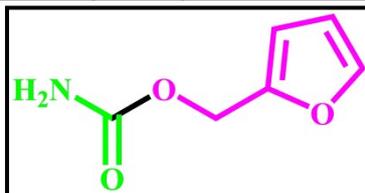


Figure S27: ^1H NMR spectra of Phenethyl carbamate (**4i**).

Furan-2-ylmethyl carbamate (4j)



FTIR (KBr, ν , cm^{-1}): 3474-3364 (-NH stretching), 1719 ($>\text{C}=\text{O}$ stretching), 1604 (-NH bending), 1333 (-CN stretching), 1023 (C-O stretching).

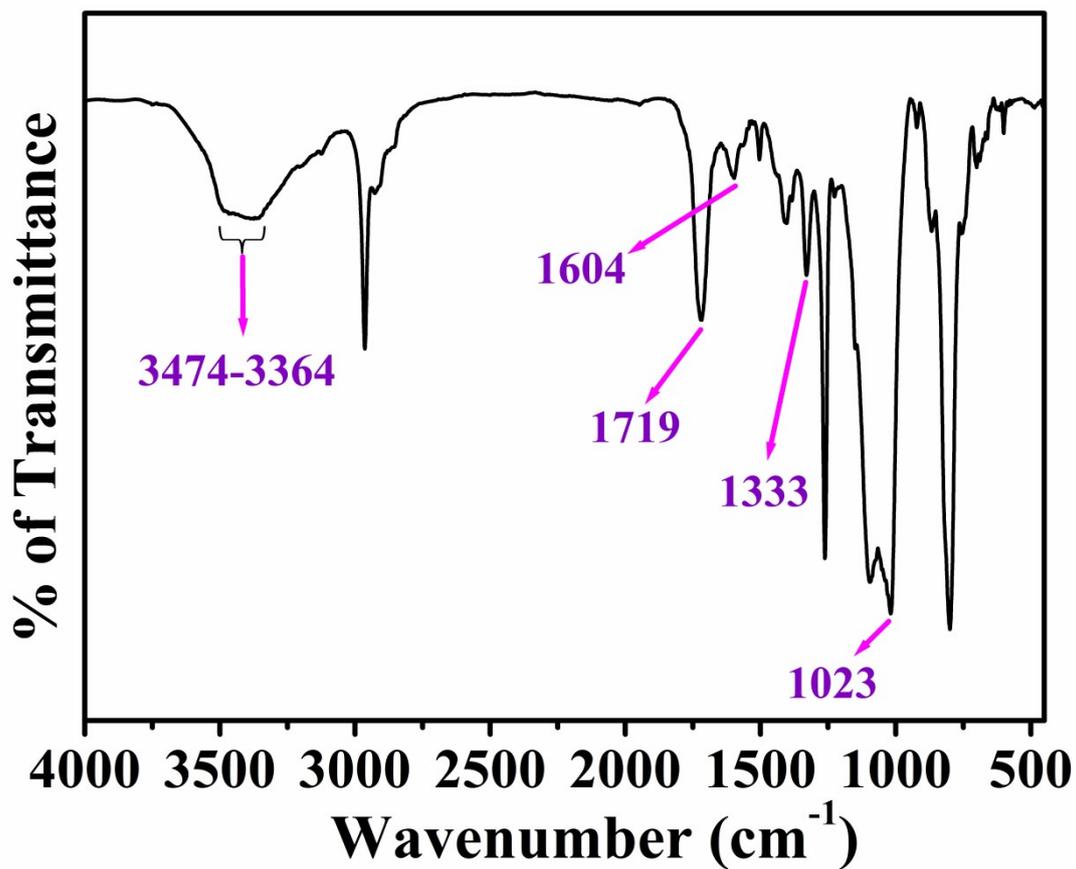


Figure S28: FTIR spectra of Furan-2-ylmethyl carbamate (4j).

^1H NMR (400 MHz, CDCl_3) δ 4.555 (brs, $-\text{NH}_2$), 5.049 (s, 2H), 6.350-6.364 (t, $J=2.8$ Hz, 2H), 7.417 (s, 1H) ppm.

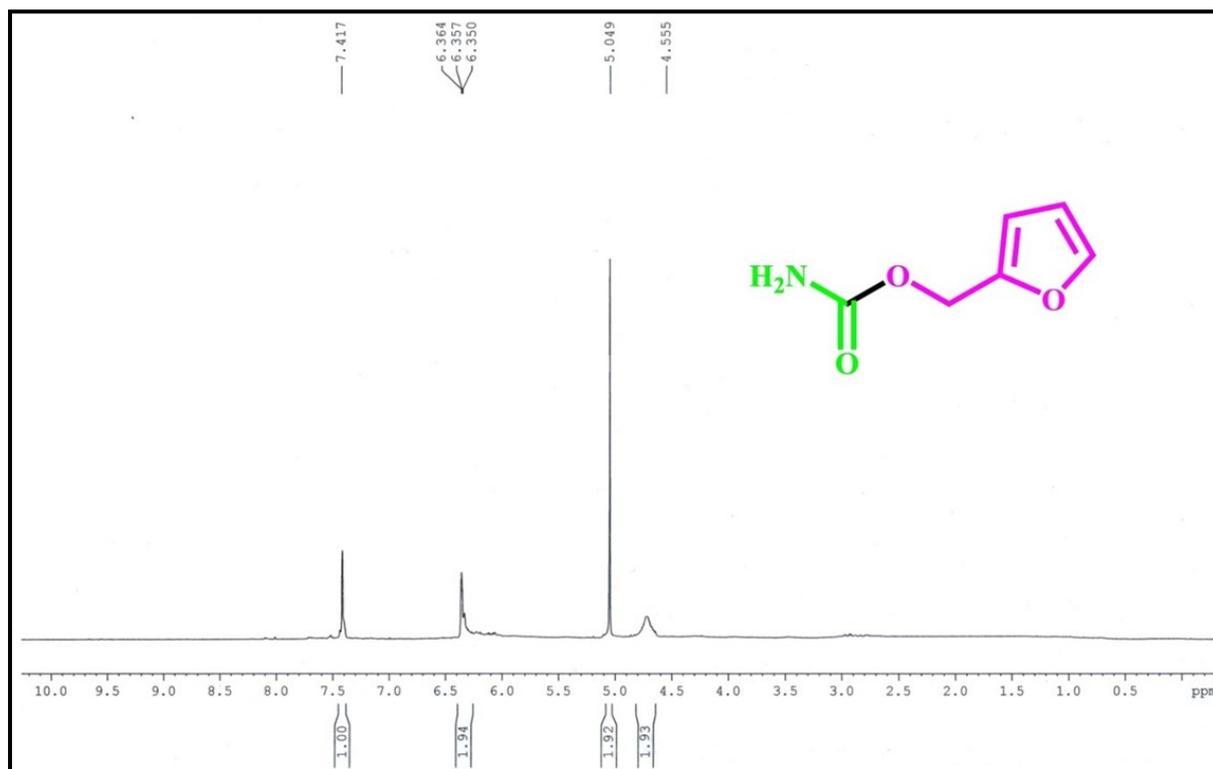
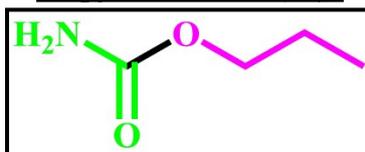


Figure S29: ^1H NMR spectra of Furan-2-ylmethyl carbamate (**4j**).

Propyl carbamate (4k)⁴



FTIR (KBr, ν , cm^{-1}): 3393 (-NH stretching), 1701 ($>\text{C}=\text{O}$ stretching), 1605 (-NH bending), 1326 (-CN stretching), 1151 (C-O stretching).

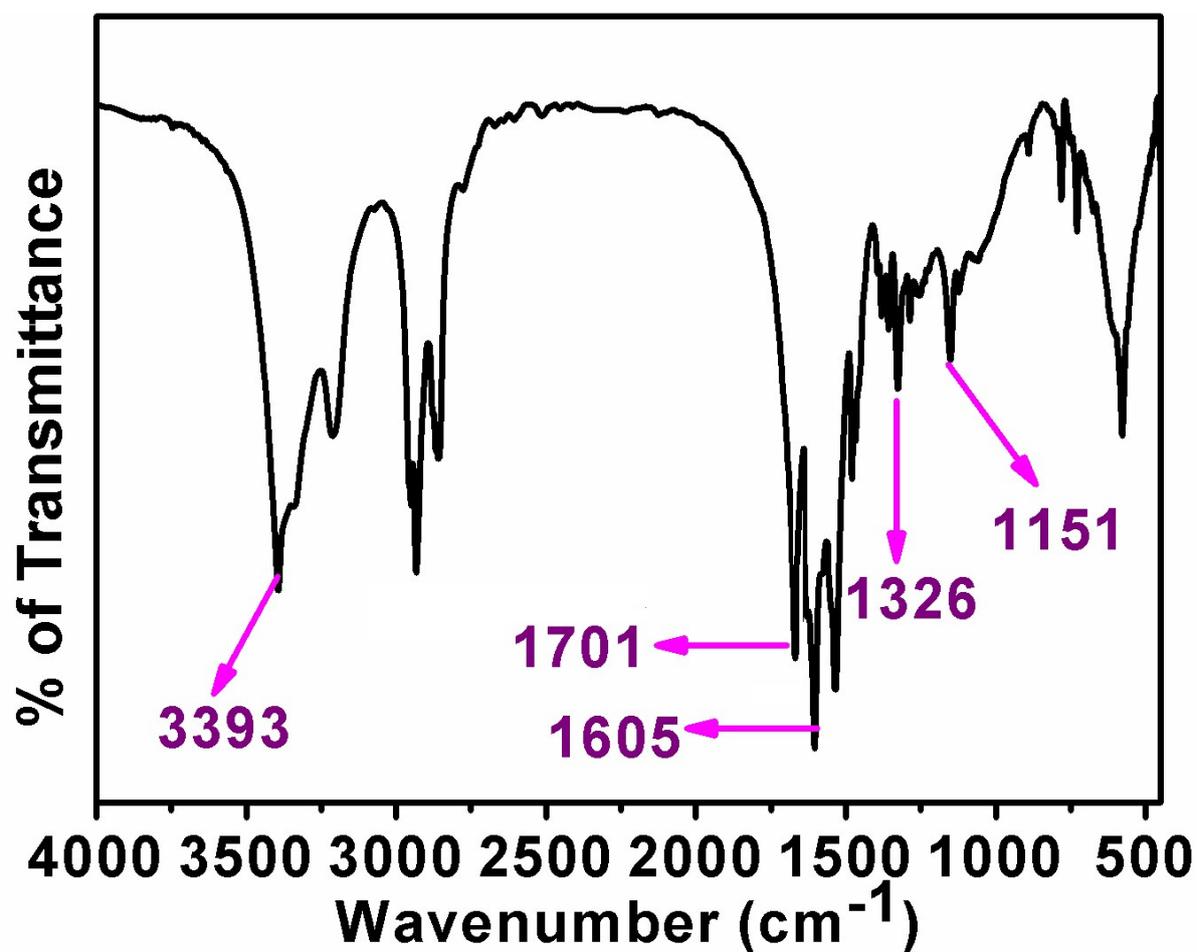


Figure S30: FTIR spectra of Propyl carbamate (4k).

^1H NMR (400 MHz, CDCl_3) δ 0.874-0.911 (t, $J=7.6$ Hz, 3H), 1.585-1.656 (m, 2H), 4.042-4.076 (t, $J=6.8$ Hz, 2H), 5.572 (brs, $-\text{NH}_2$) ppm.

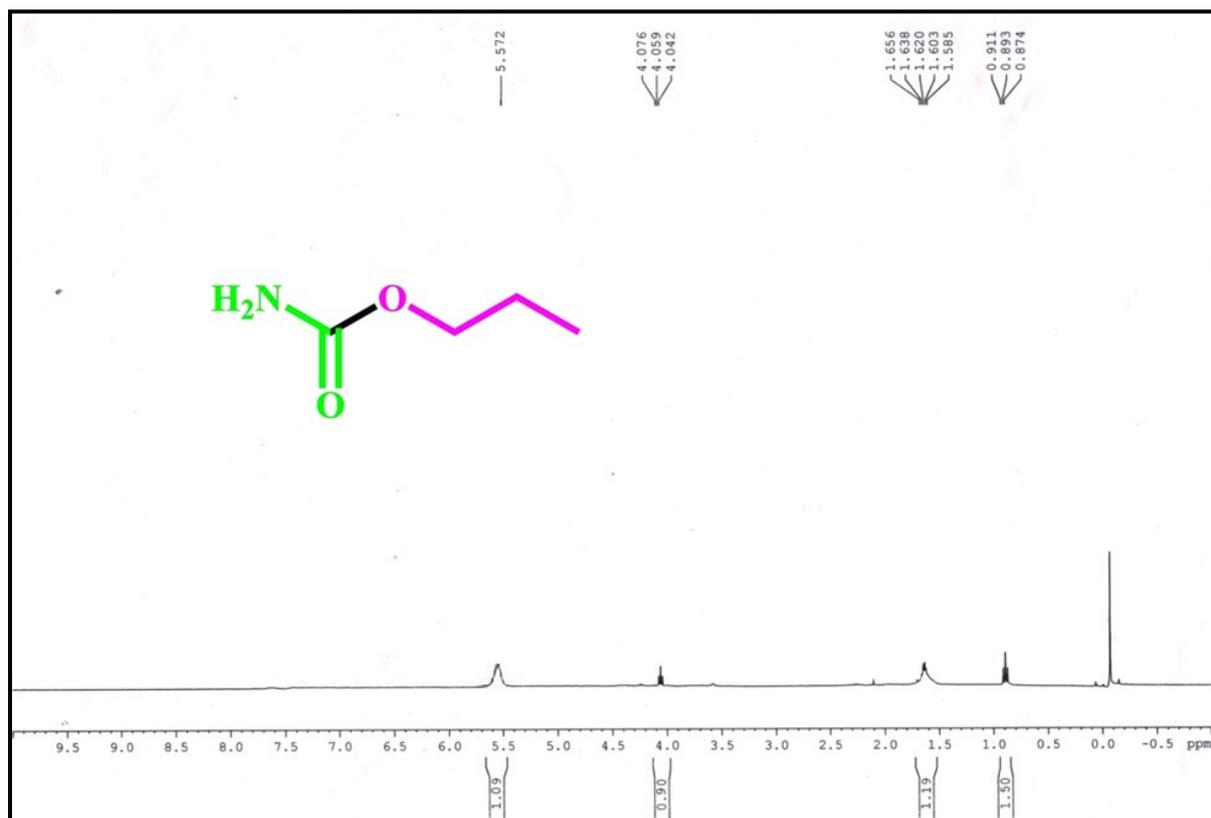


Figure S31: ^1H NMR spectra of Propyl carbamate (**4k**).

Isopropyl carbamate (4I)³



FTIR (KBr, ν , cm^{-1}): 3396-3345 (-NH stretching), 1719 ($>\text{C}=\text{O}$ stretching), 1668 (-NH bending), 1268 (-CN stretching), 1101-1023 (C-O stretching).

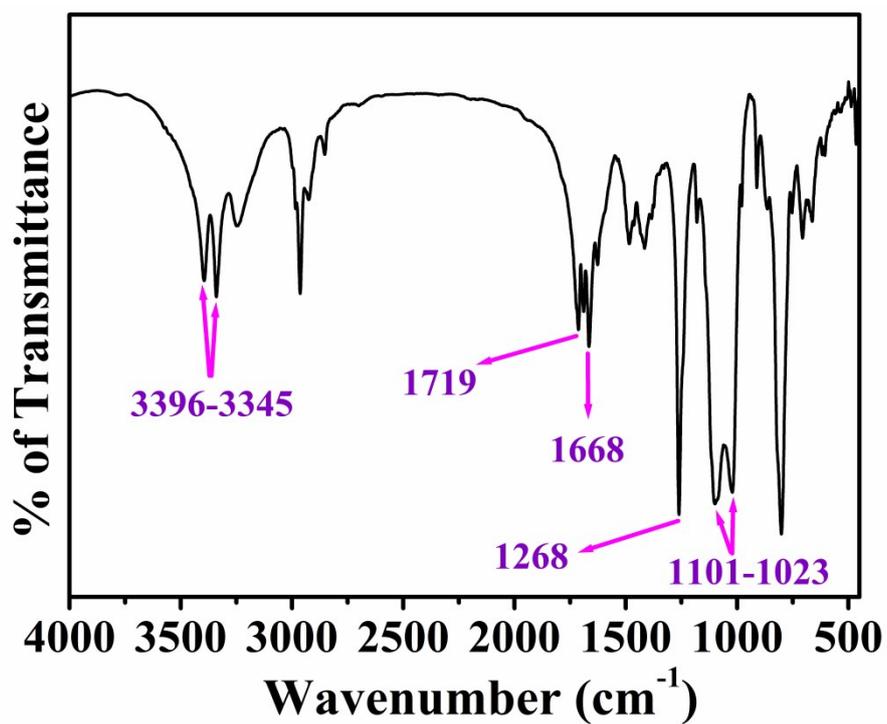


Figure S32: FTIR spectra of Isopropyl carbamate (4I).

^1H NMR (400 MHz, CDCl_3) δ 1.183-1.236 (m, 6H), 4.901-4.933 (m, 1H), 5.201 (brs, $-\text{NH}_2$) ppm.

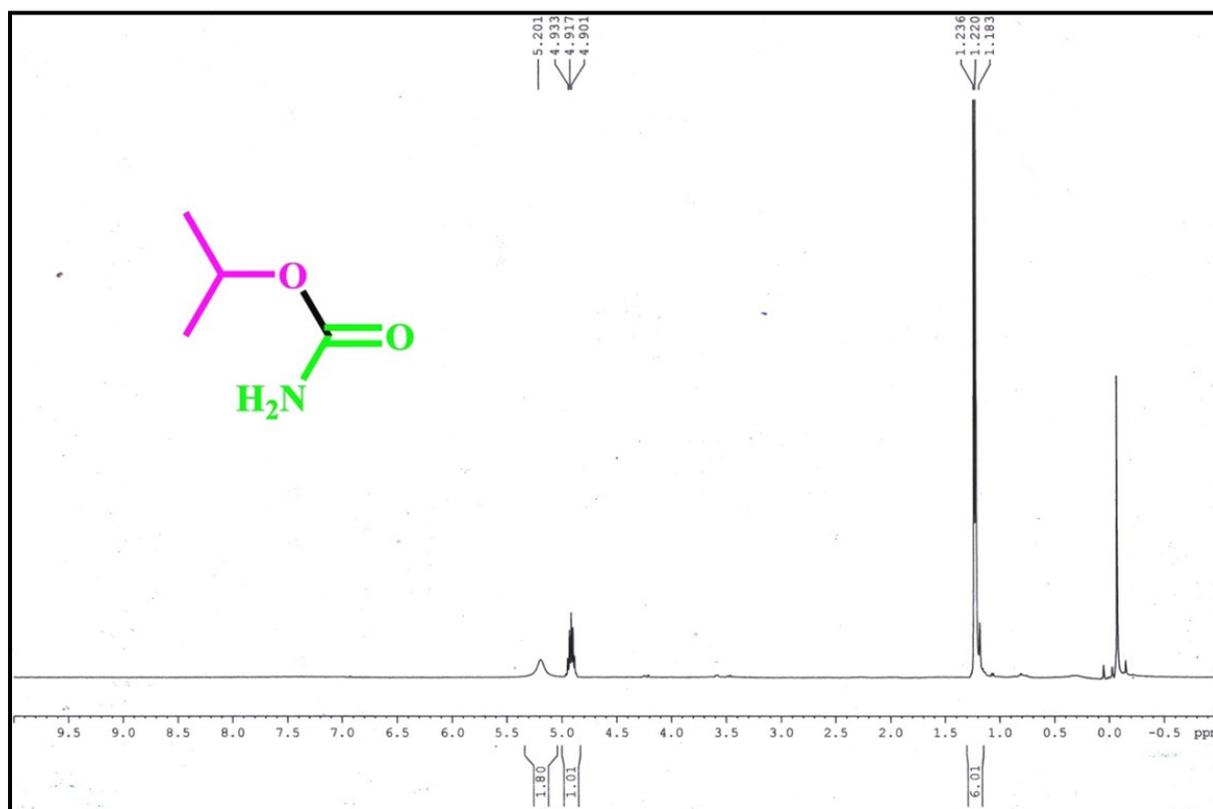
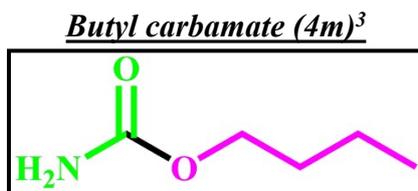


Figure S33: ^1H NMR spectra of Isopropyl carbamate (**4I**).



FTIR (KBr, ν , cm^{-1}): 3377-3339 (-NH stretching), 1733 ($>\text{C}=\text{O}$ stretching), 1604 (-NH bending), 1242 (-CN stretching), 1120 (C-O stretching).

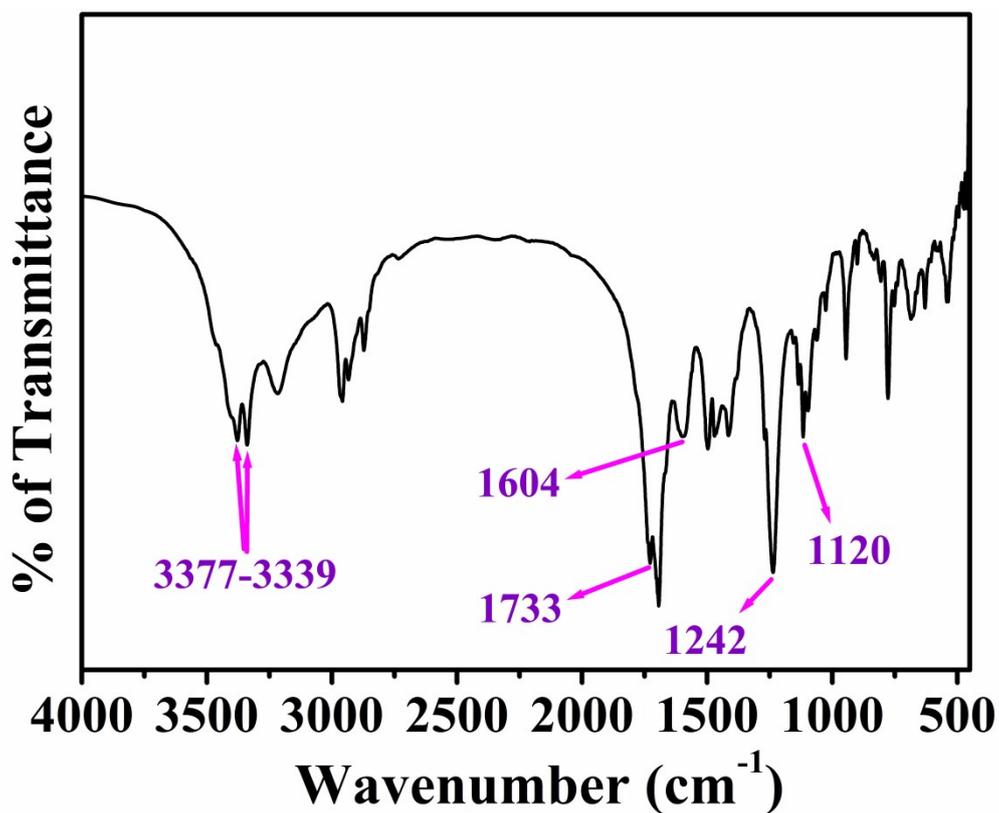


Figure S34: FTIR spectra of Butyl carbamate (**4m**).

^1H NMR (400 MHz, CDCl_3) δ 0.880-0.967 (m, 3H), 1.252-1.420 (m, 4H), 4.154-4.222 (m, 2H), 5.184 (brs, $-\text{NH}_2$) ppm.

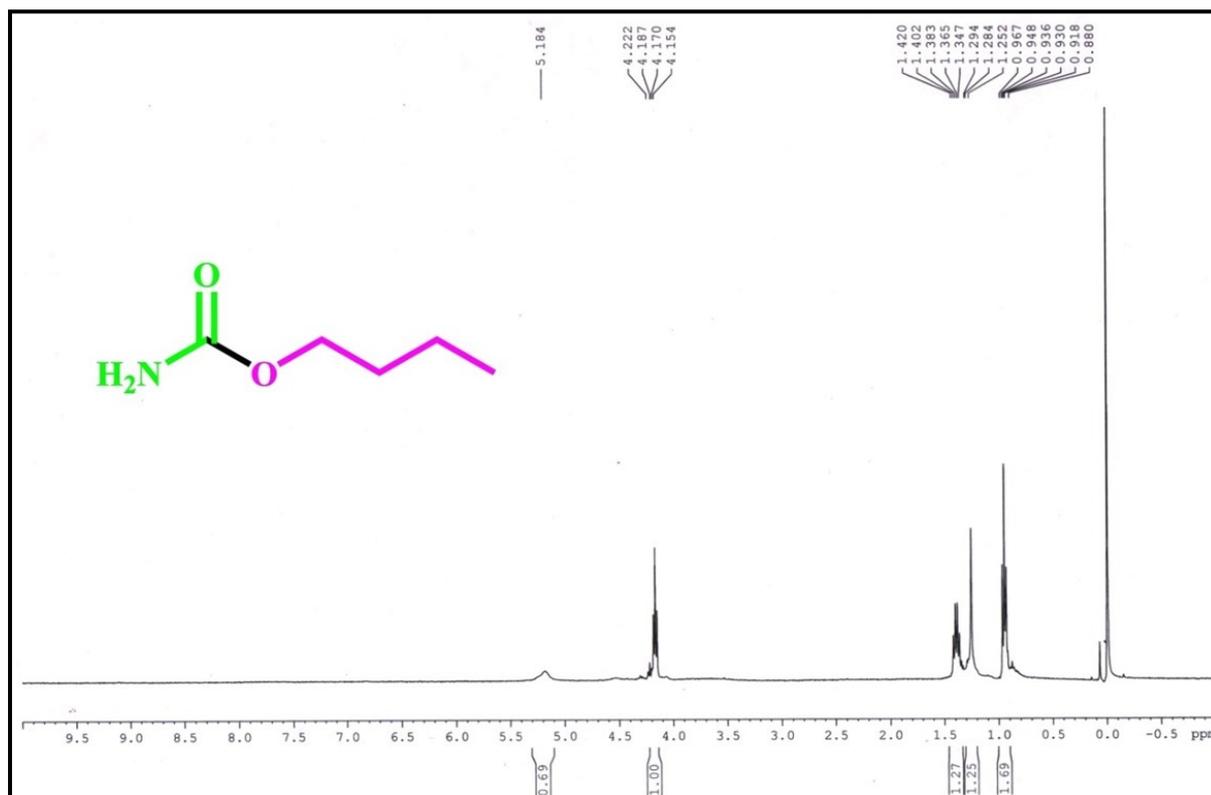
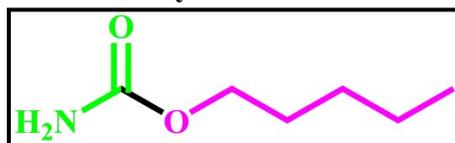


Figure S35: ^1H NMR spectra of Butyl carbamate (**4m**).

Pentyl carbamate



FTIR (KBr, ν , cm^{-1}): 3383-3345 (-NH stretching), 1694 ($>\text{C}=\text{O}$ stretching), 1604 (-NH bending), 1262 (-CN stretching), 1101-1030 (C-O stretching).

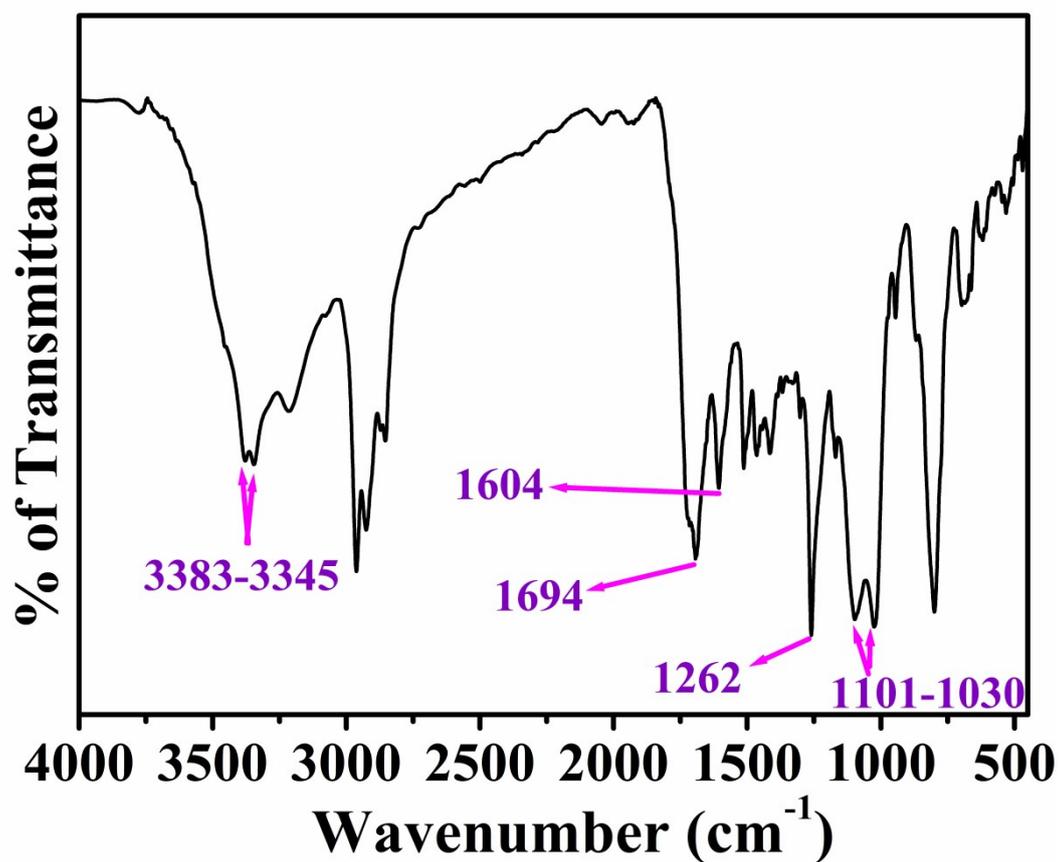


Figure S36: FTIR spectra of Pentyl carbamate (4n).

^1H NMR (400 MHz, CDCl_3) δ 1.246 (s, 3H), 1.500-1.565 (m, 4H), 1.671-1.705 (t, $J=6.8$ Hz, 2H), 4.071-4.105 (t, $J=6.8$ Hz, 2H), 5.585 (brs, $-\text{NH}_2$) ppm.

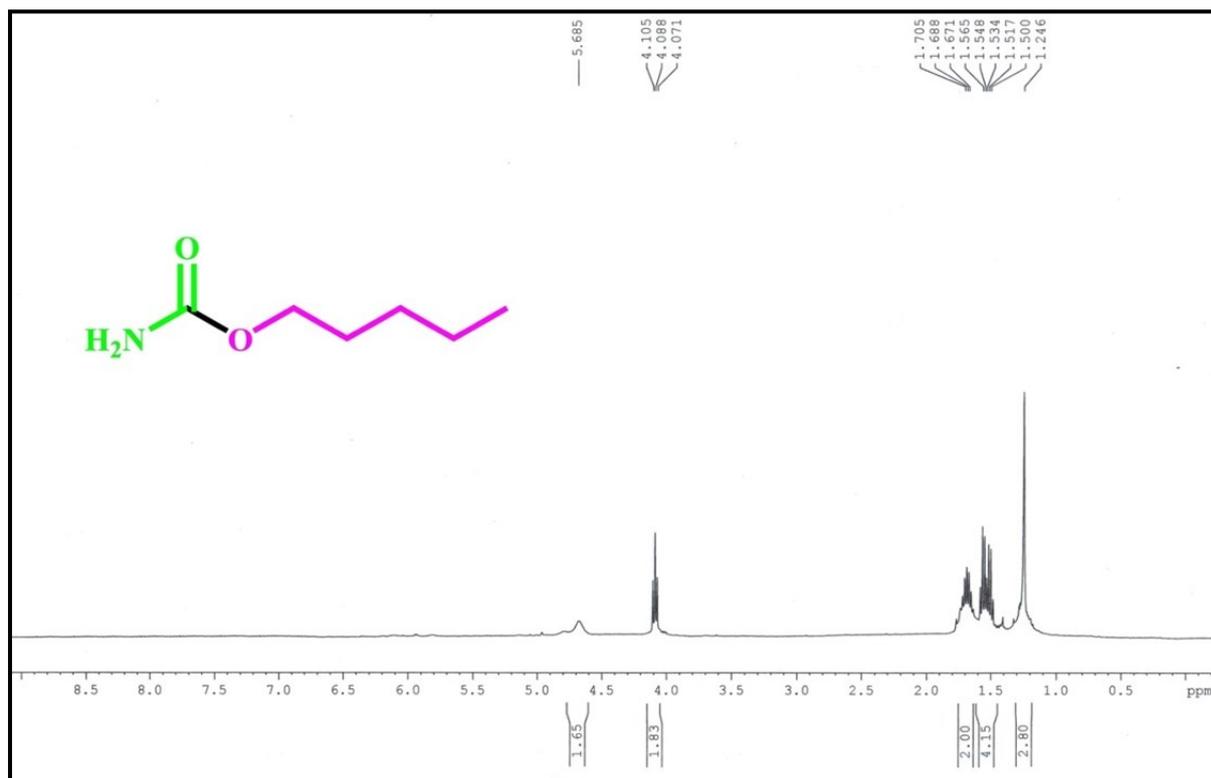


Figure S37: ^1H NMR spectra of Pentyl carbamate (**4n**).

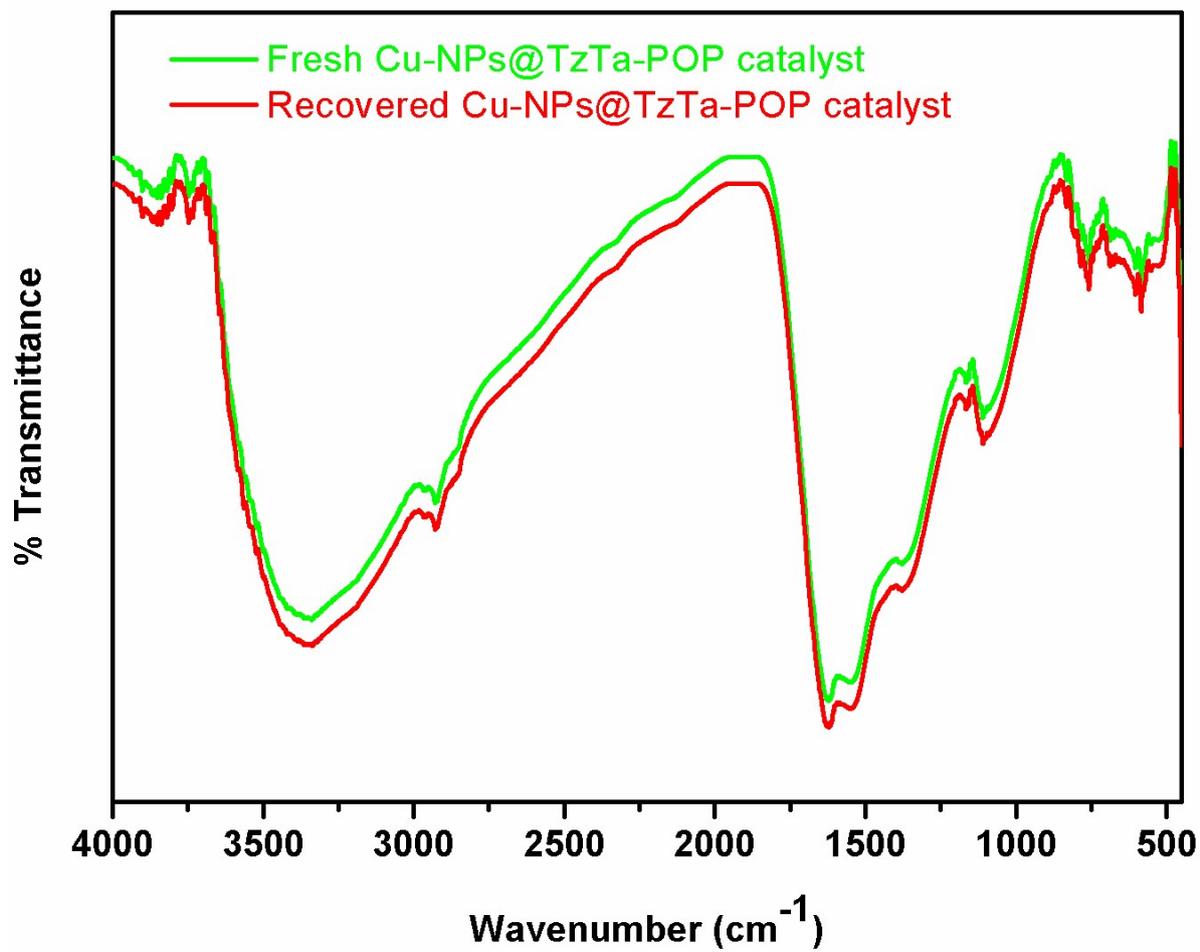


Figure S38: FTIR spectra of recycled Cu-NPs@TpTz-POP catalyst.

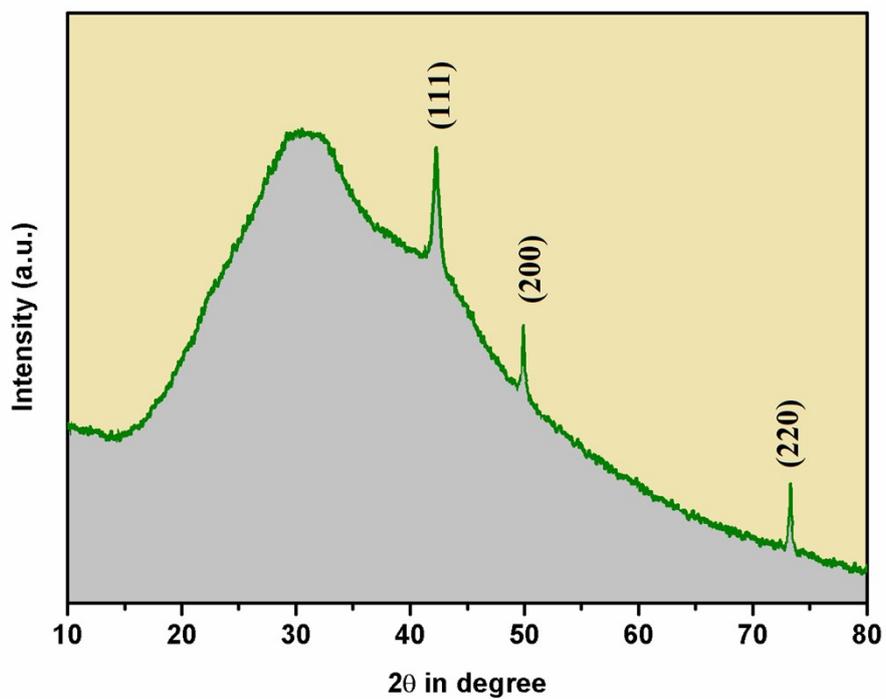


Figure S39: PXRD pattern of recycled Cu-NPs@TpTz-POP catalyst.

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- 2 P. Sarkar, A. H. Chowdhury, S. Riyajuddin, S. Biswas, K. Ghosh and S. M. Islam, *New J. Chem.*, 2020, **44**, 744-752.
- 3 P. Basu, T. K. Dey, A. Ghosh, S. Biswas, A. Khan and S. M. Islam, *New J. Chem.*, 2020, **44**, 2630-2643.
- 4 I. D. Inaloo, S. Majnooni and M. Esmaeilpour, *Eur. J. Org. Chem.*, 2018, 3481–3488.