

Supporting information

Task-specific acidic ionic liquid [CmMIm]Cl-catalyzed one-pot multicomponent synthesis of β -aminocarbonyl compounds at room temperature

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SI-1. General Information:

All reagents and solvents used in this study were purchased from trusted sources such as Merck, Sigma-Aldrich, TCI Chemicals, SD Fine, and HIMEDIA. Solvents used in this work were freshly distilled before use to maintain the required level of purity, whereas the remaining chemicals were employed as received without further purification. Deionized water was used wherever applicable during the experimental work. All reactions were carried out under normal laboratory atmospheric conditions using clean, oven-dried glassware, and no inert atmosphere was applied. The progress of the reaction was monitored by thin-layer chromatography (TLC) on Merck silica gel 60F254 plates (0.25 mm), with a mixture of ethyl acetate and petroleum ether. Fourier-transform infrared (FT-IR) spectrum of [CmMIm]Cl was recorded on a Bruker ALPHA-II. The ¹H and ¹³C NMR spectra of the synthesized compounds were recorded by a Bruker AVANCE NEO 500 MHz and 125 MHz spectrometer, respectively, using CDCl₃ or DMSO-d₆ as solvent.

SI-2. Method for the preparation of [CmMIm]Cl

An equimolar mixture of N-methyl imidazole (10 mmol) and 2-chloro acetic acid (10 mmol) was stirred at 40°C for 2 hours in acetonitrile (2 mL) till the solution became viscous. After that, the viscous liquid was washed thoroughly with ethyl acetate (3 × 2 mL) to remove the unreacted starting materials and dried under reduced pressure to obtain pure viscous [CmMIm]Cl.

SI-3. Representative experimental procedure for [CmMIm]Cl-catalyzed synthesis of 1,3-diphenyl-3-(phenylamino)propan-1-one (**1a**) via Mannich Reaction:

A mixture of benzaldehyde (1 mmol, 106 mg), aniline (1 mmol, 93.13 mg), acetophenone (1 mmol, 120.4 mg), and [CmMIm]Cl (50 mg, 28 mol%), was stirred at room temperature for 1.5 hours. The progress of the reaction was monitored by TLC (using ethyl acetate-pet ether as solvent in 1:4 ratio). After the completion of the reaction, the product was separated from the reaction mixture by adding 10 mL of ethyl acetate. The solid product, 1,3-diphenyl-3-(phenylamino)propan-1-one (**1a**), was then recrystallized using hot ethanol. (yield = 95%, M.P. = 168-170). The formation of the desired product was further confirmed by ¹H NMR and ¹³C NMR spectroscopic studies.

SI-4-. FTIR spectrum of fresh [CmMIm]Cl

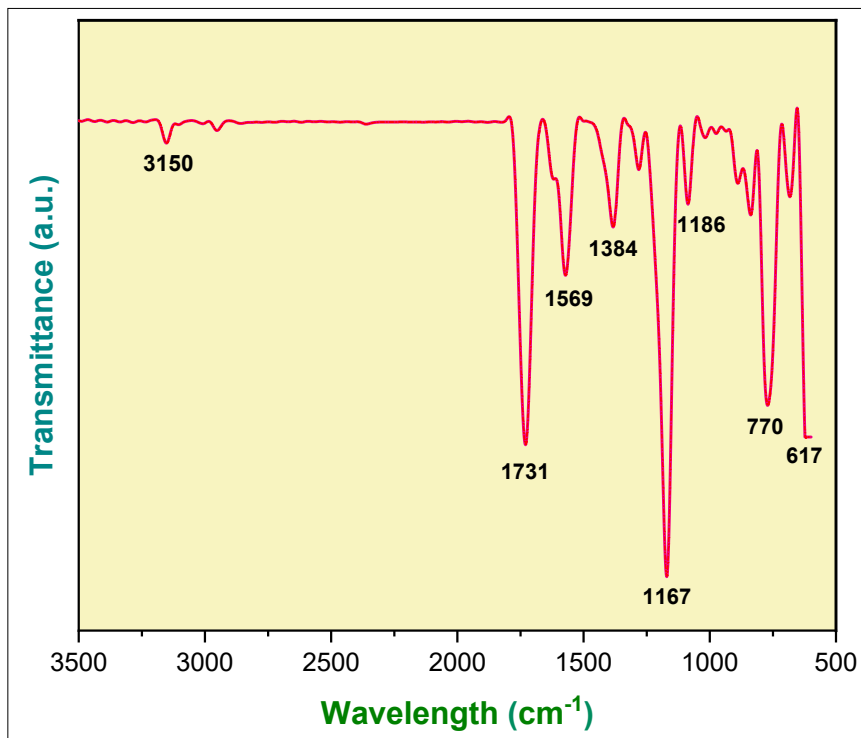


Fig S1- FTIR spectrum of fresh [CmMIm]Cl

SI-5-. FTIR spectrum of reused [CmMIm]Cl

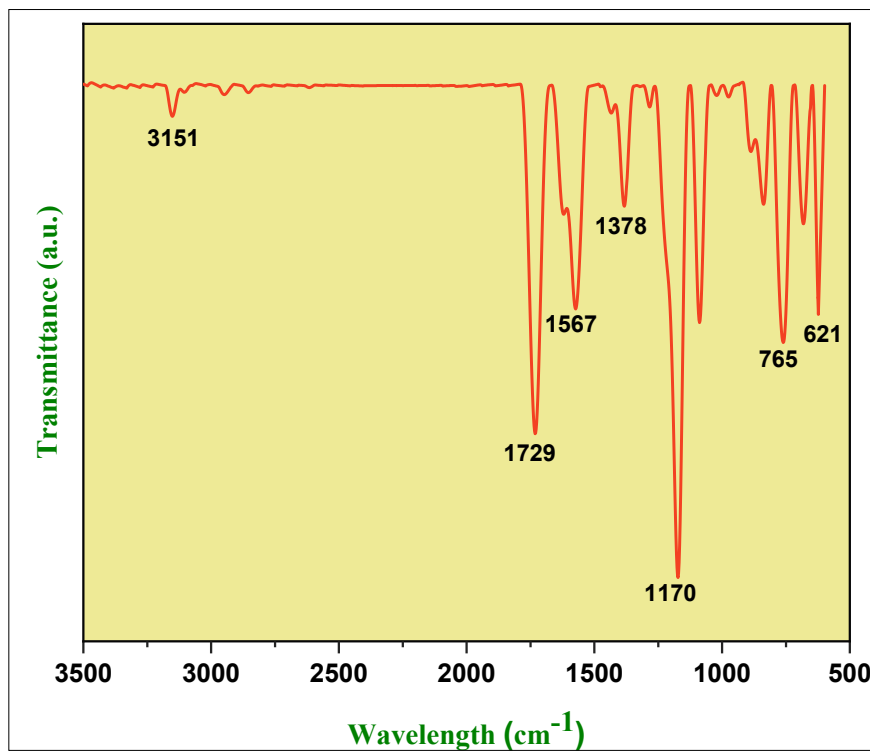


Fig S2- FTIR spectrum of reused [CmMIm]Cl

SI-6- Copies of ^1H NMR and ^{13}C NMR spectrum of compounds

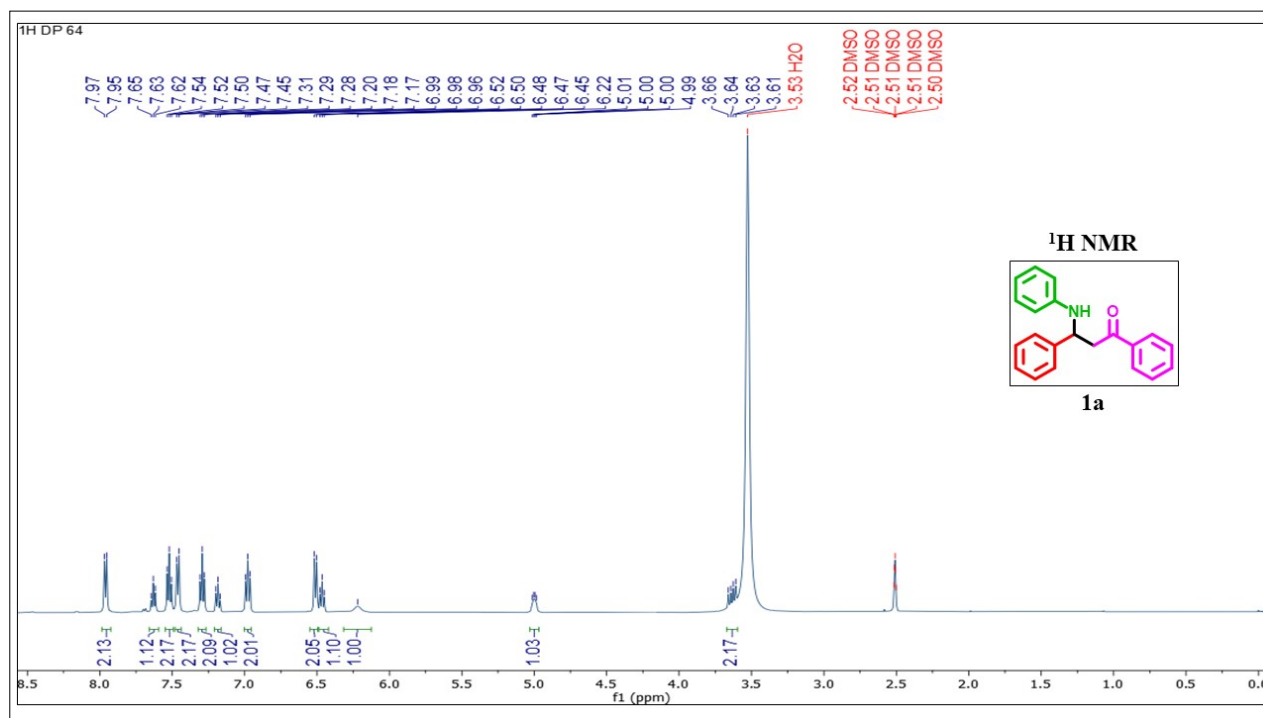


Fig S3- ^1H NMR spectra of 1,3-diphenyl-3-(phenylamino)propan-1-one (**1a**)

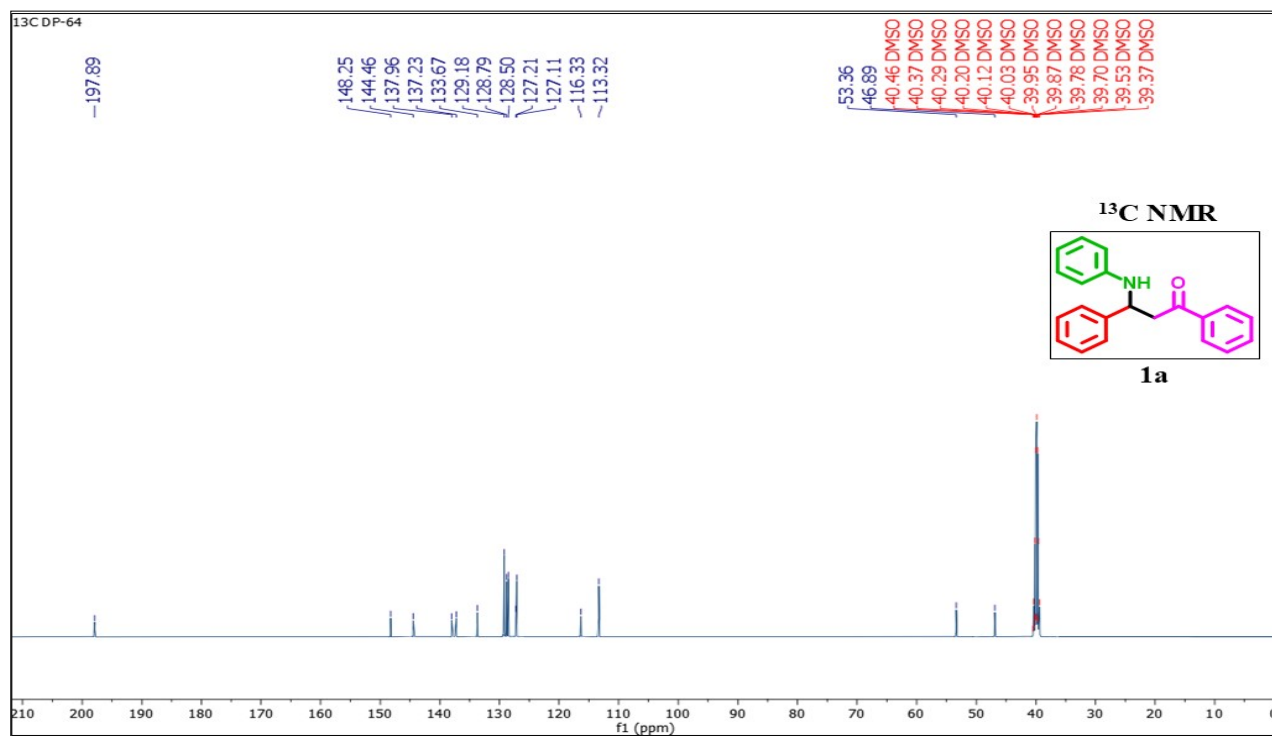


Fig S4-¹³C NMR spectrum of 1,3-diphenyl-3-(phenylamino)propan-1-one (**1a**)

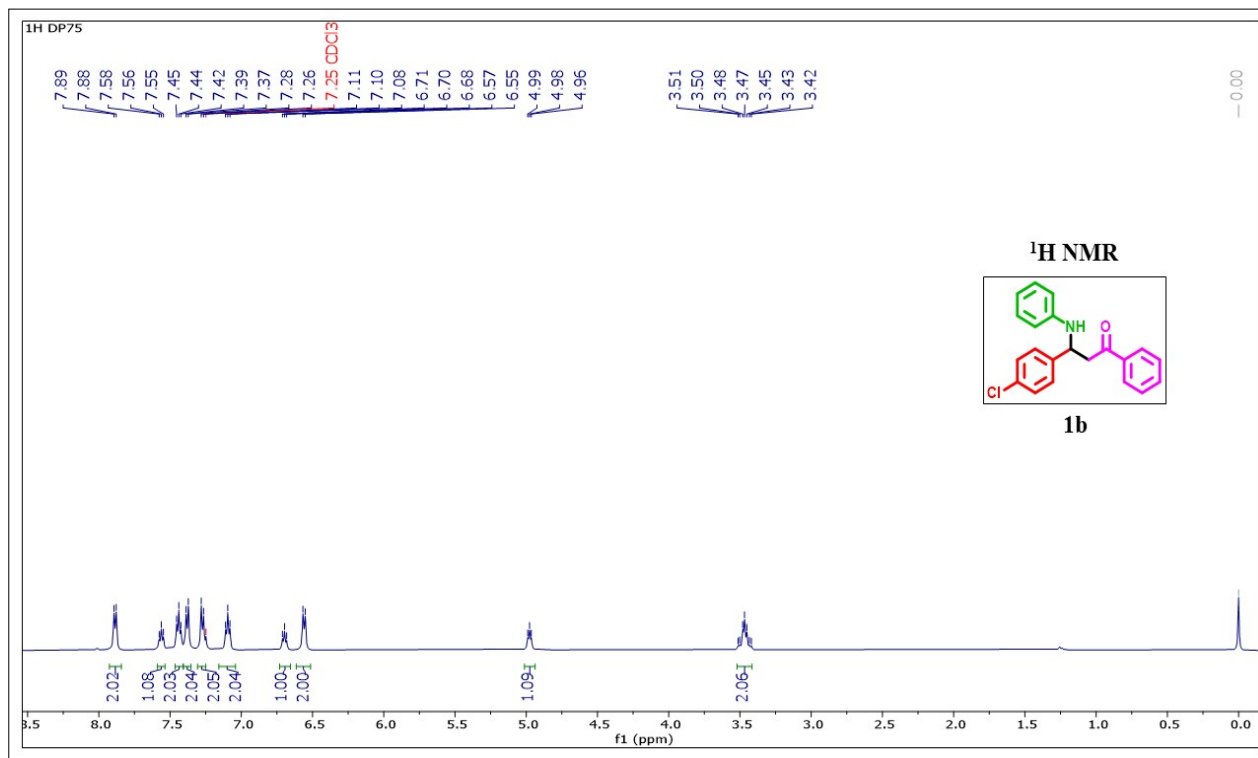


Fig S5- ¹H NMR spectra of 3-(4-chlorophenyl)-1-phenyl-3-(phenylamino)propan-1-one (**1b**)

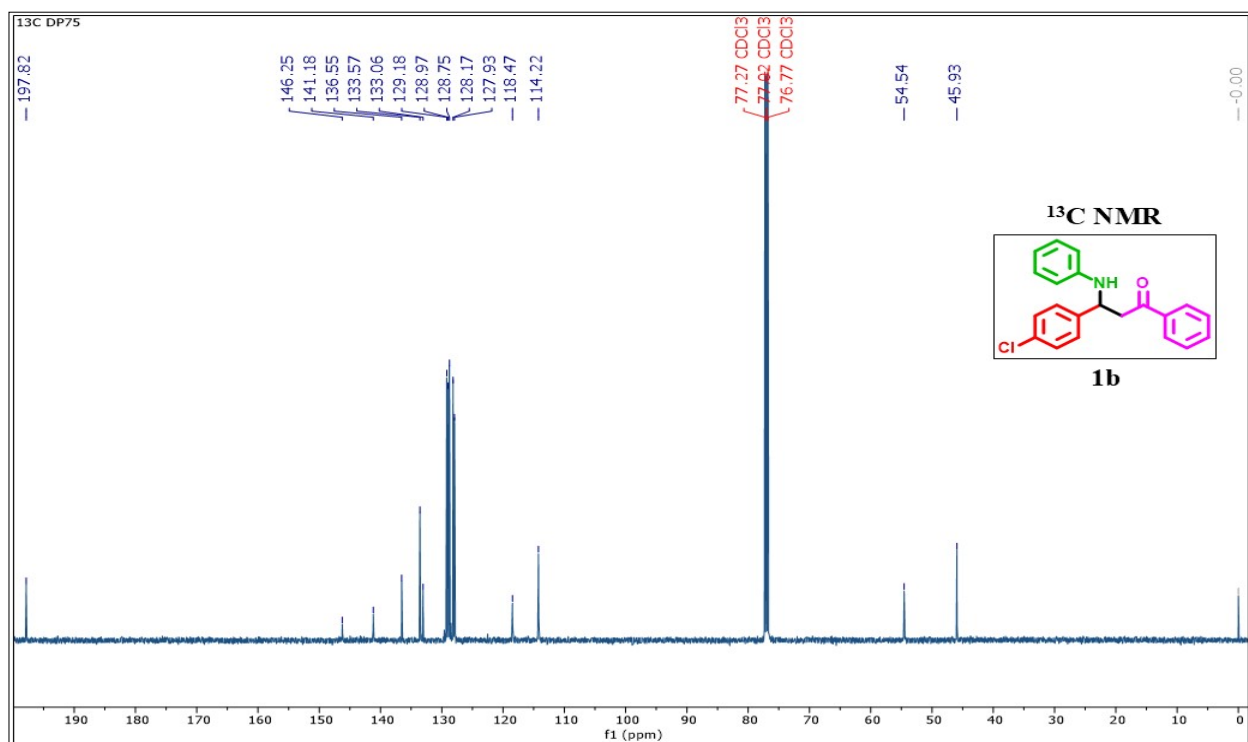


Fig S6-¹³C NMR spectrum of 3-(4-chlorophenyl)-1-phenyl-3-(phenylamino)propan-1-one (**1b**)

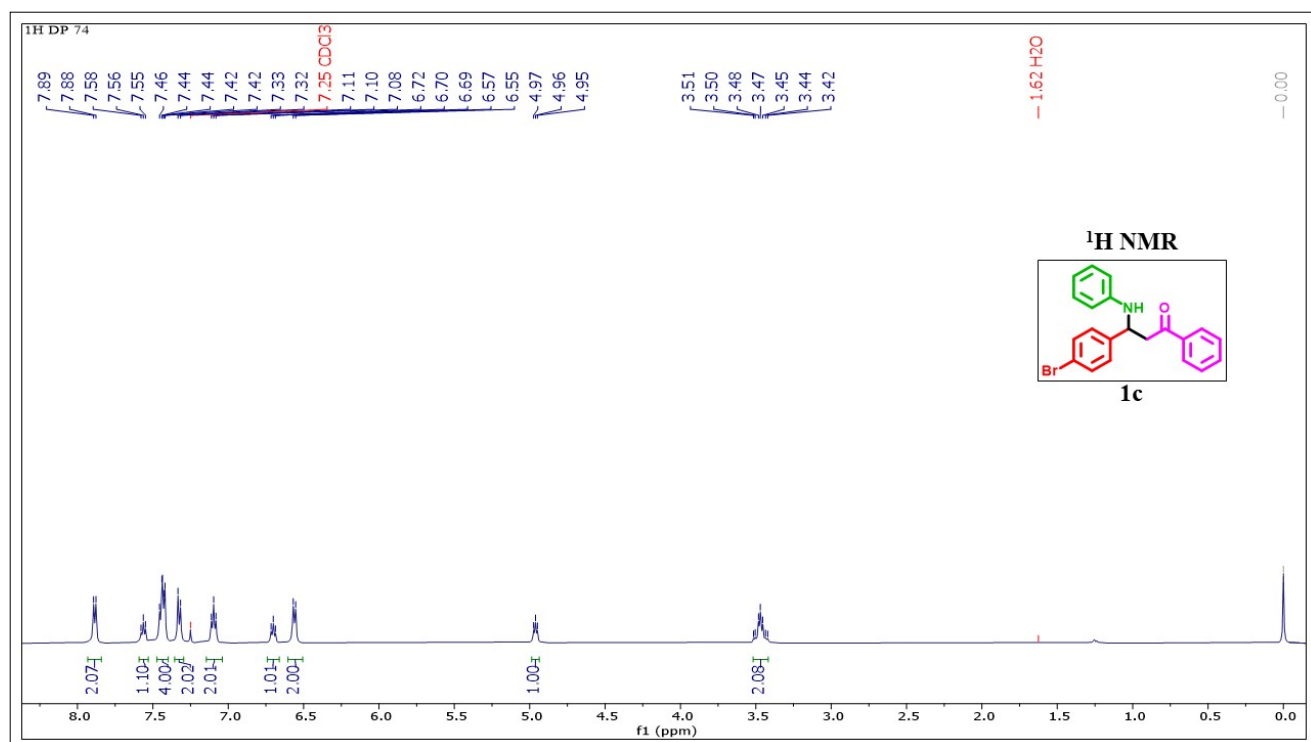


Fig S7- ¹H NMR spectra of 3-(4-bromophenyl)-1-phenyl-3-(phenylamino)propan-1-one (**1c**)

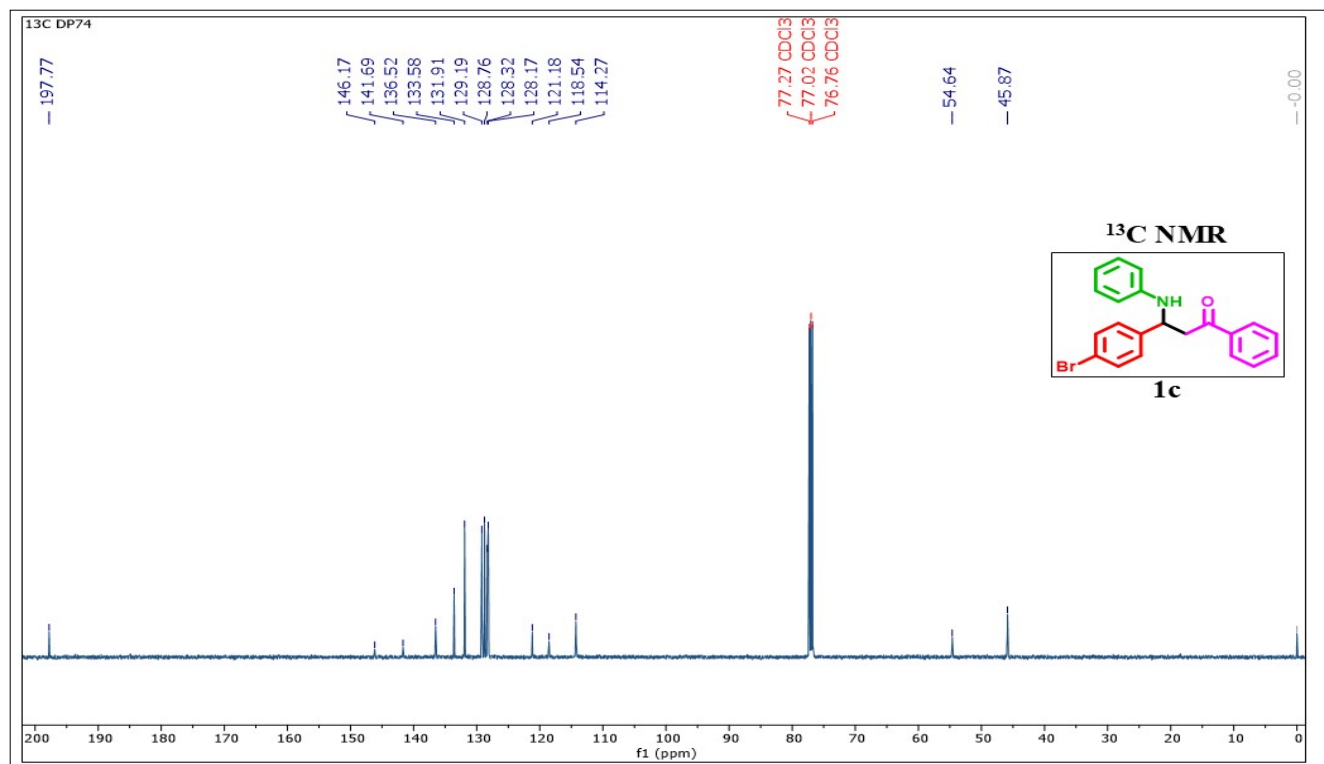


Fig S8-¹³C NMR spectrum of 3-(4-bromophenyl)-1-phenyl-3-(phenylamino)propan-1-one (**1c**)

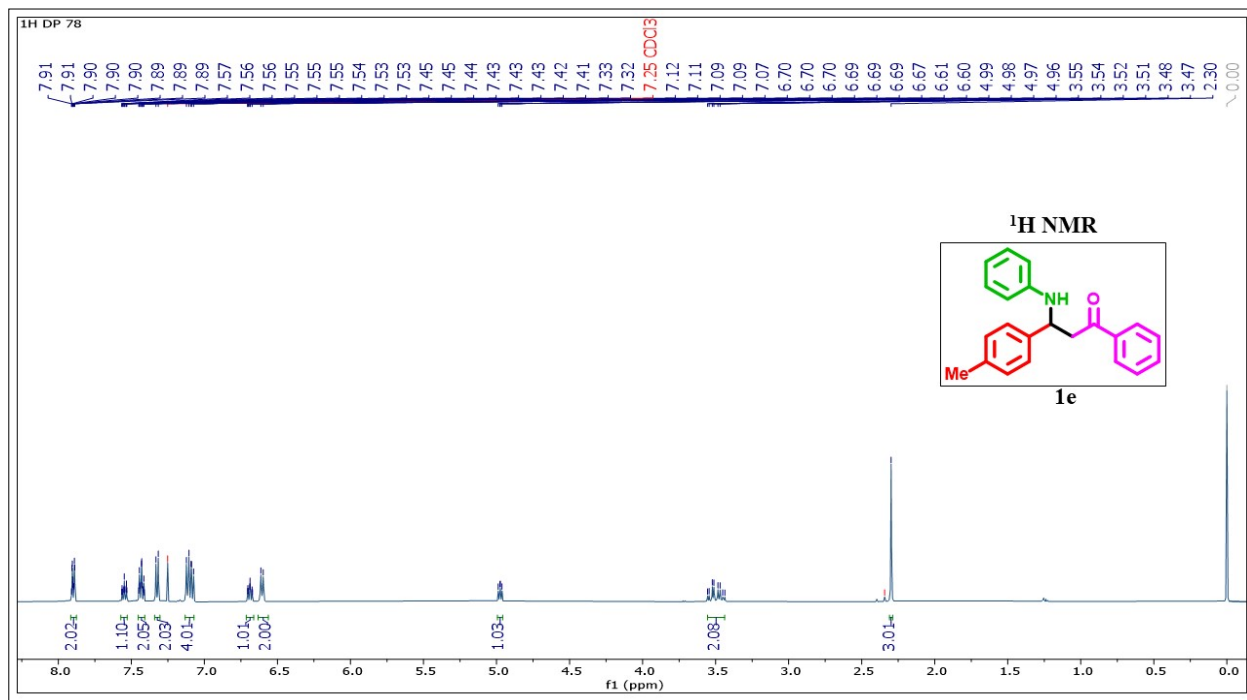


Fig S9- ^1H NMR spectra of 1-phenyl-3-(phenylamino)-3-(p-tolyl)propan-1-one (**1e**)

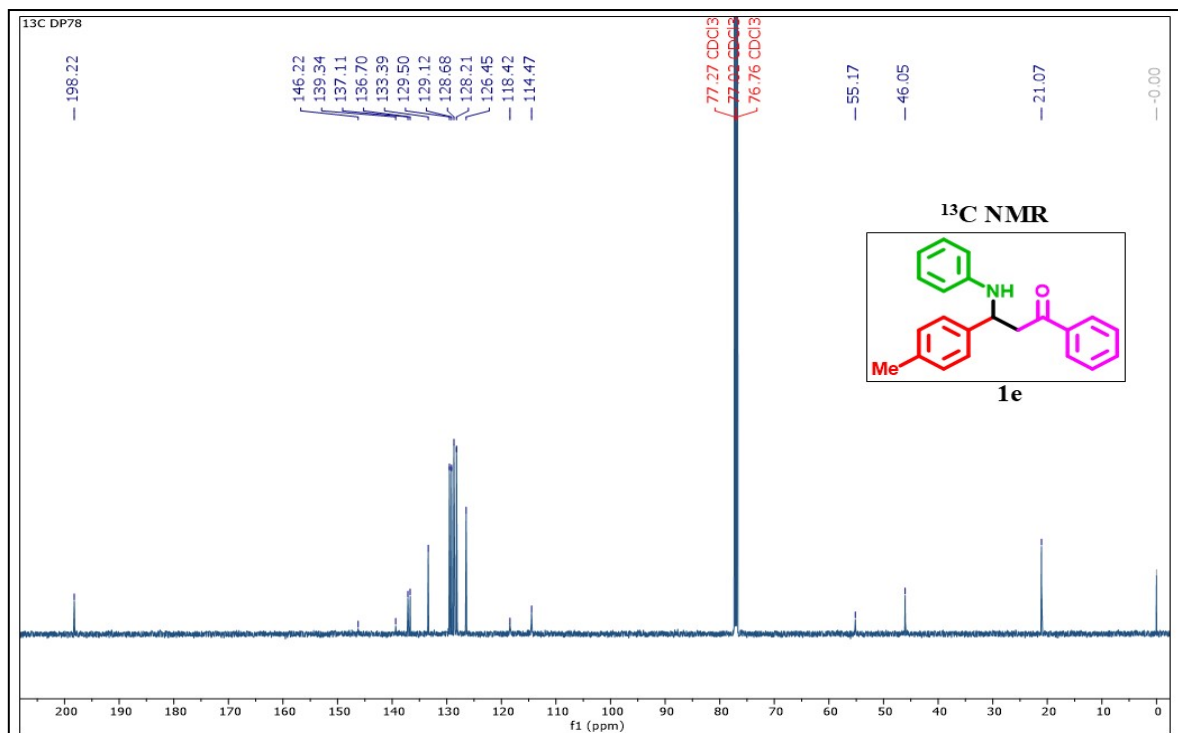


Fig S10-¹³C NMR spectrum of 1-phenyl-3-(phenylamino)-3-(p-tolyl)propan-1-one (**1e**)

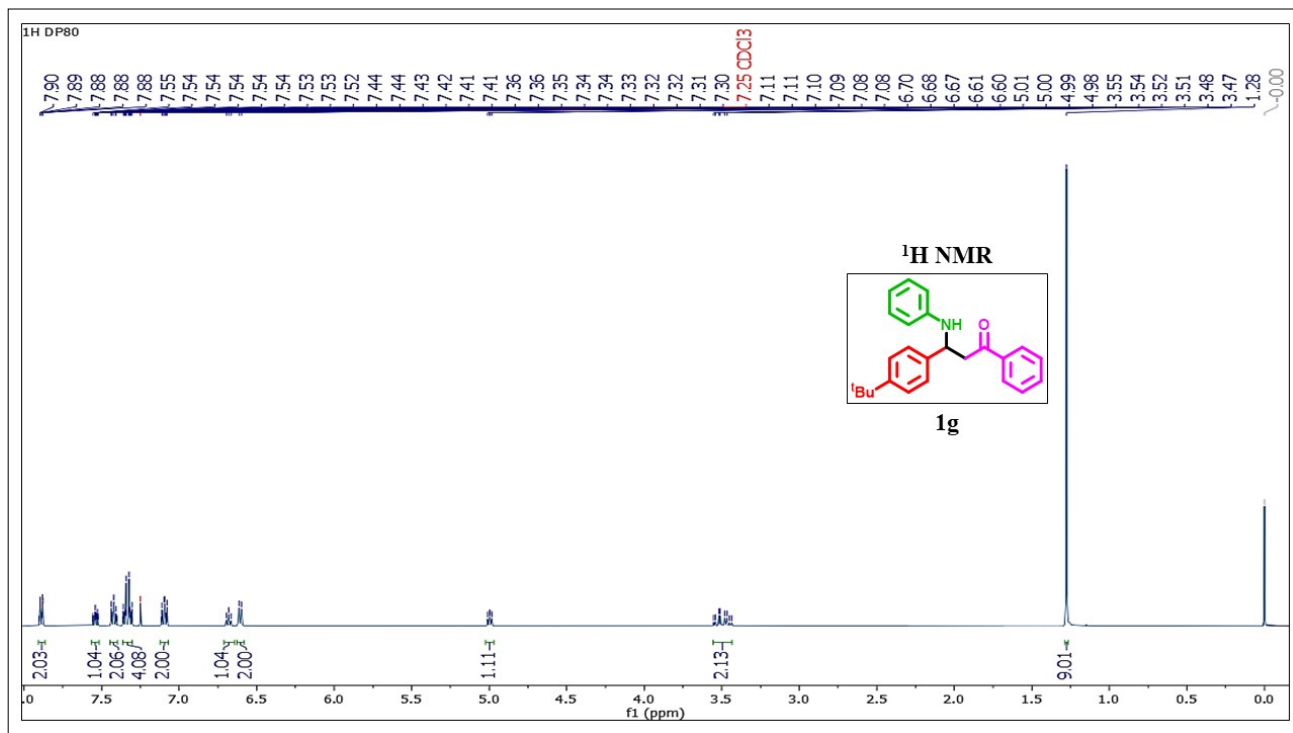


Fig S11- ¹H NMR spectra of 3-(4-(tert-butyl)phenyl)-1-phenyl-3-(phenylamino)propan-1-one (**1g**)

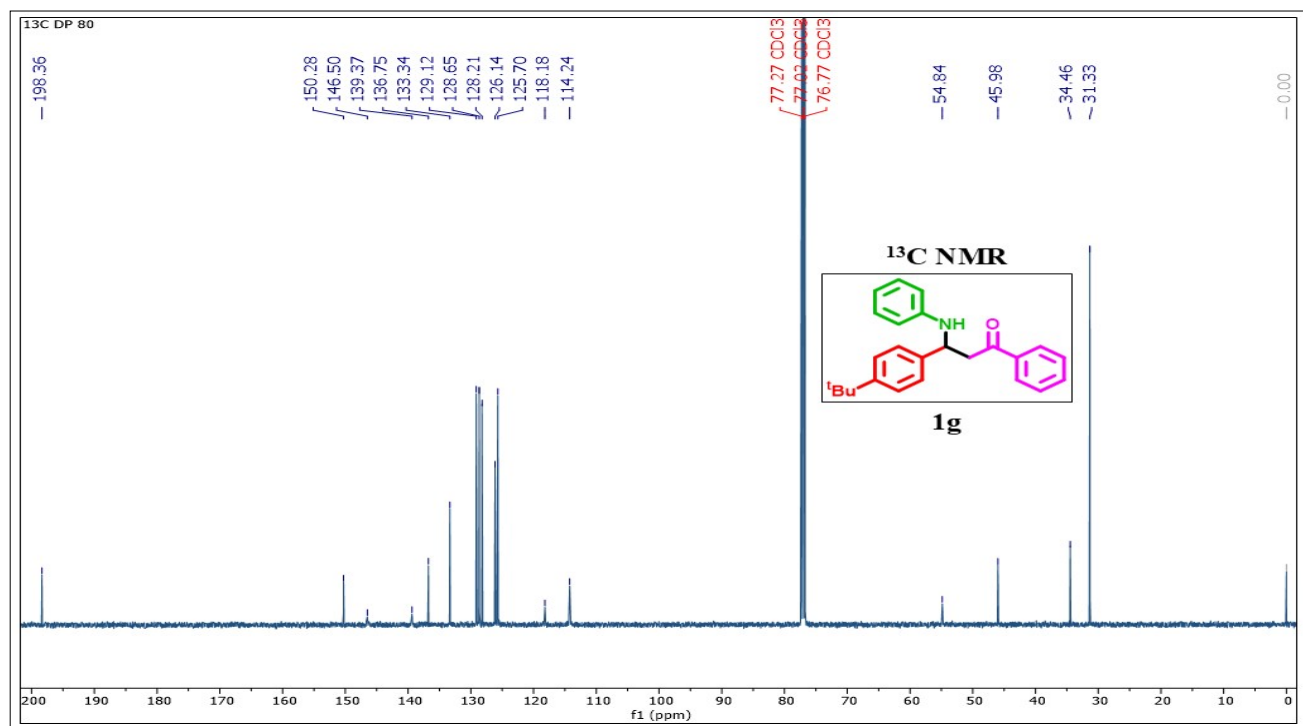


Fig S12-¹³C NMR spectrum of 3-(4-(tert-butyl)phenyl)-1-phenyl-3-(phenylamino)propan-1-one (**1g**)

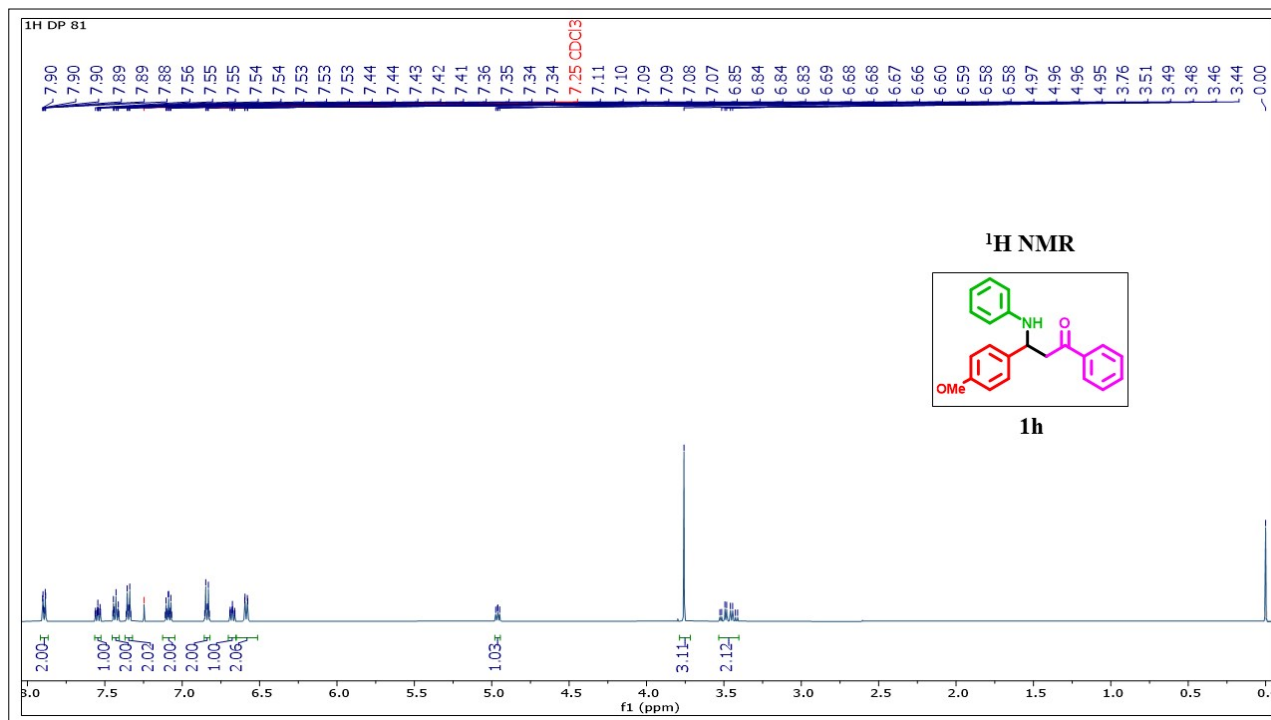


Fig S13- ¹H NMR spectra of 3-(4-methoxyphenyl)-1-phenyl-3-(phenylamino)propan-1-one (**1h**)

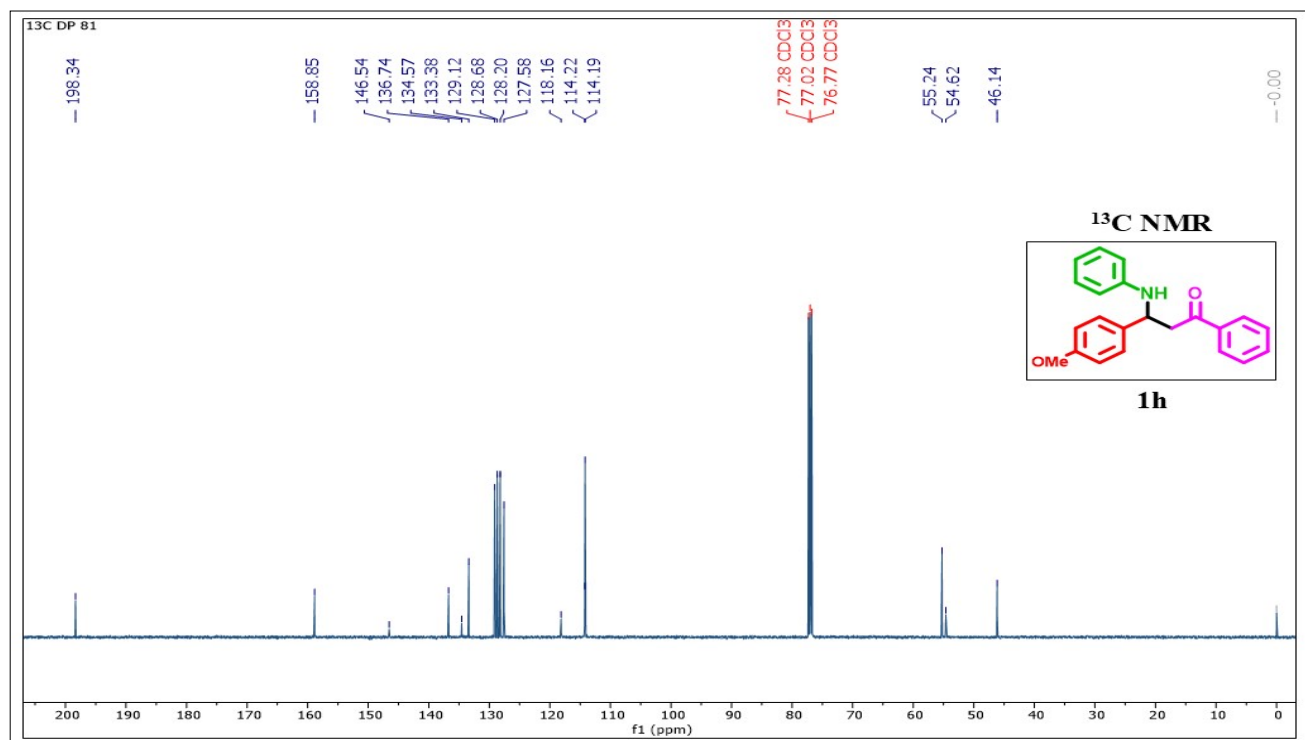


Fig S14-¹³C NMR spectrum of 3-(4-methoxyphenyl)-1-phenyl-3-(phenylamino)propan-1-one (**1h**)

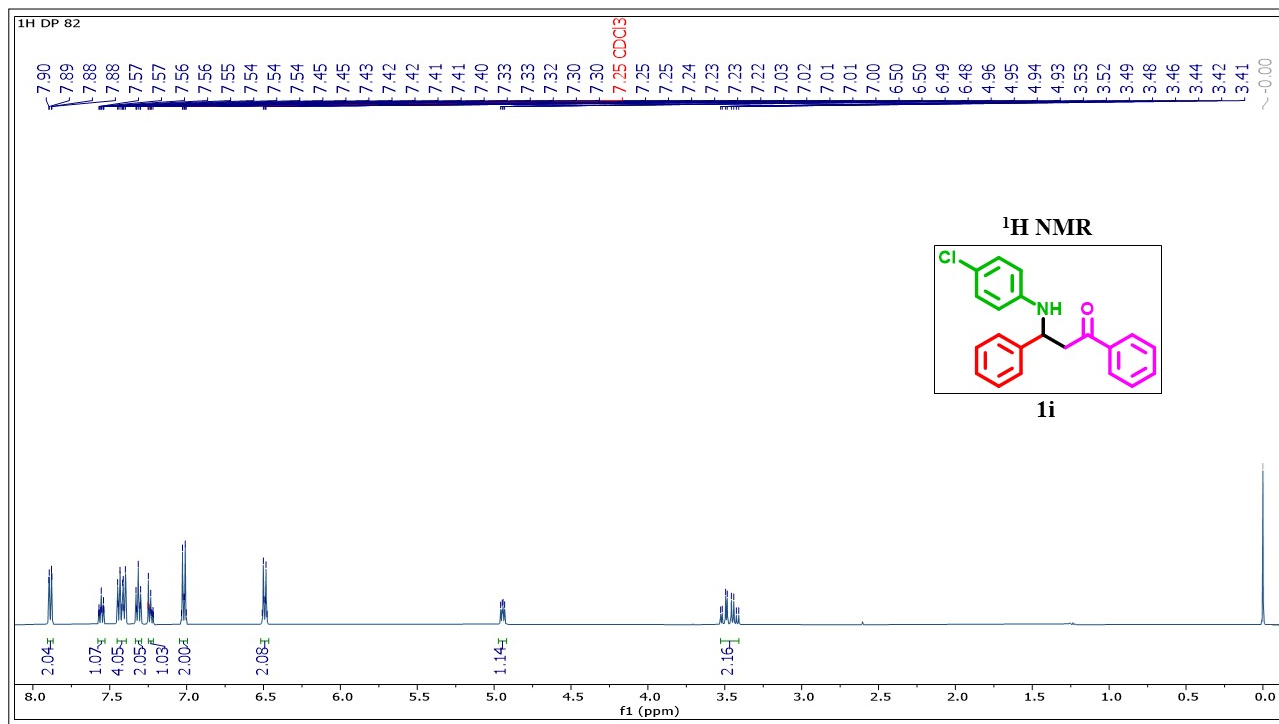


Fig S15- ¹H NMR spectra of 3-((4-chlorophenyl)amino)-1,3-diphenylpropan-1-one (**1i**)

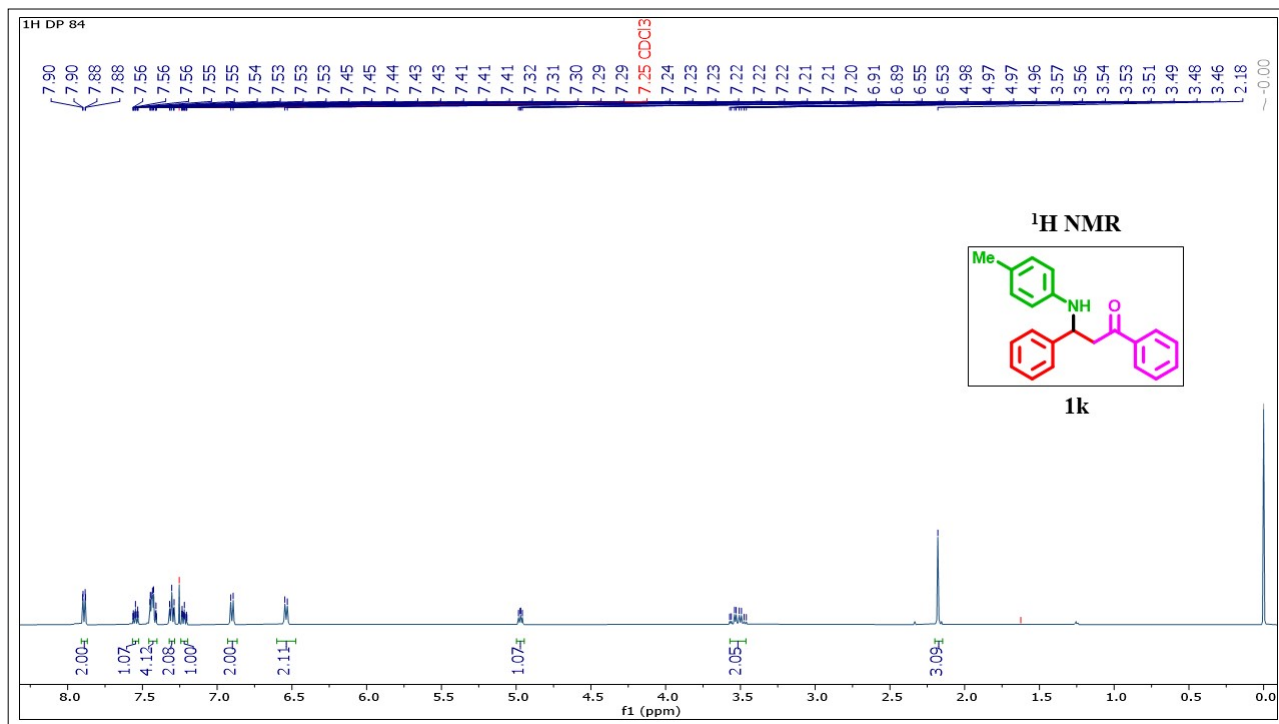


Fig S16- ¹H NMR spectra of 1,3-diphenyl-3-(p-tolylamino)propan-1-one (**1k**)

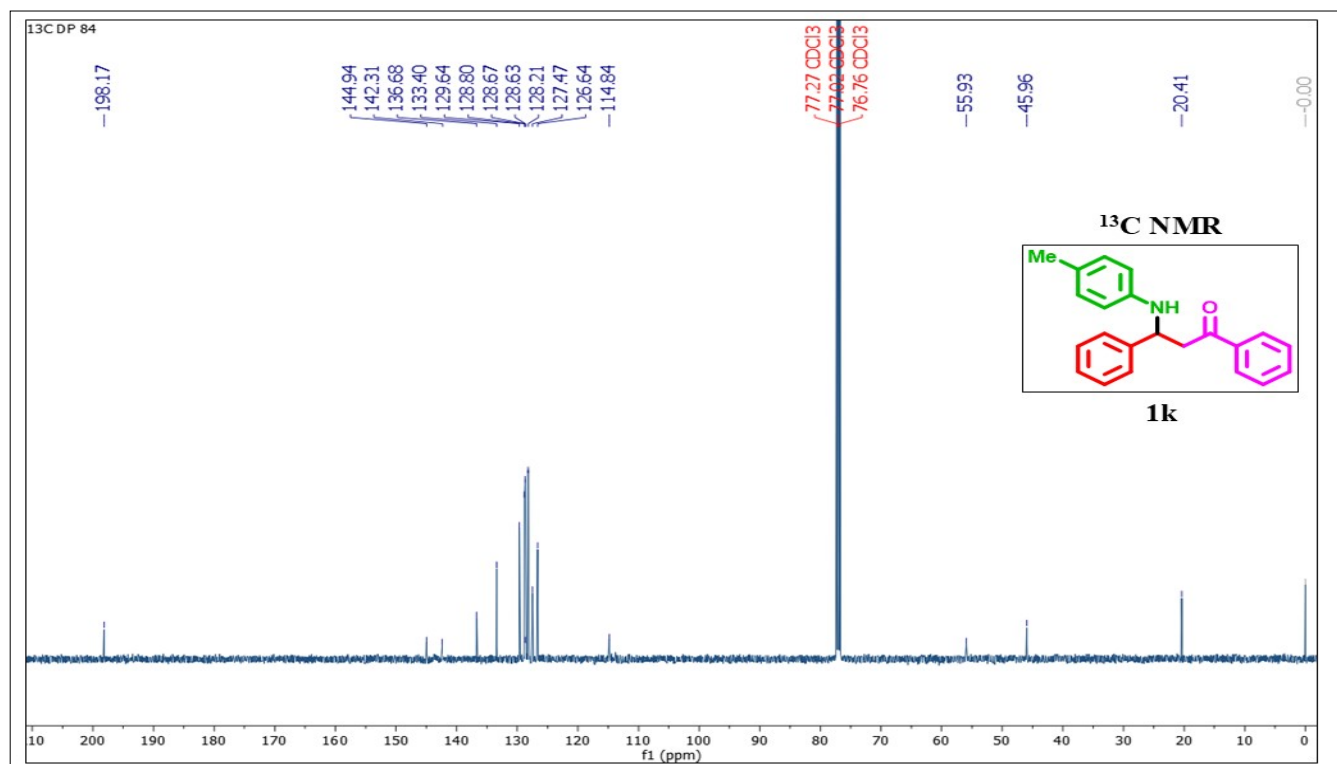


Fig S17-¹³C NMR spectrum of 1,3-diphenyl-3-(p-tolylamino)propan-1-one (**1k**)

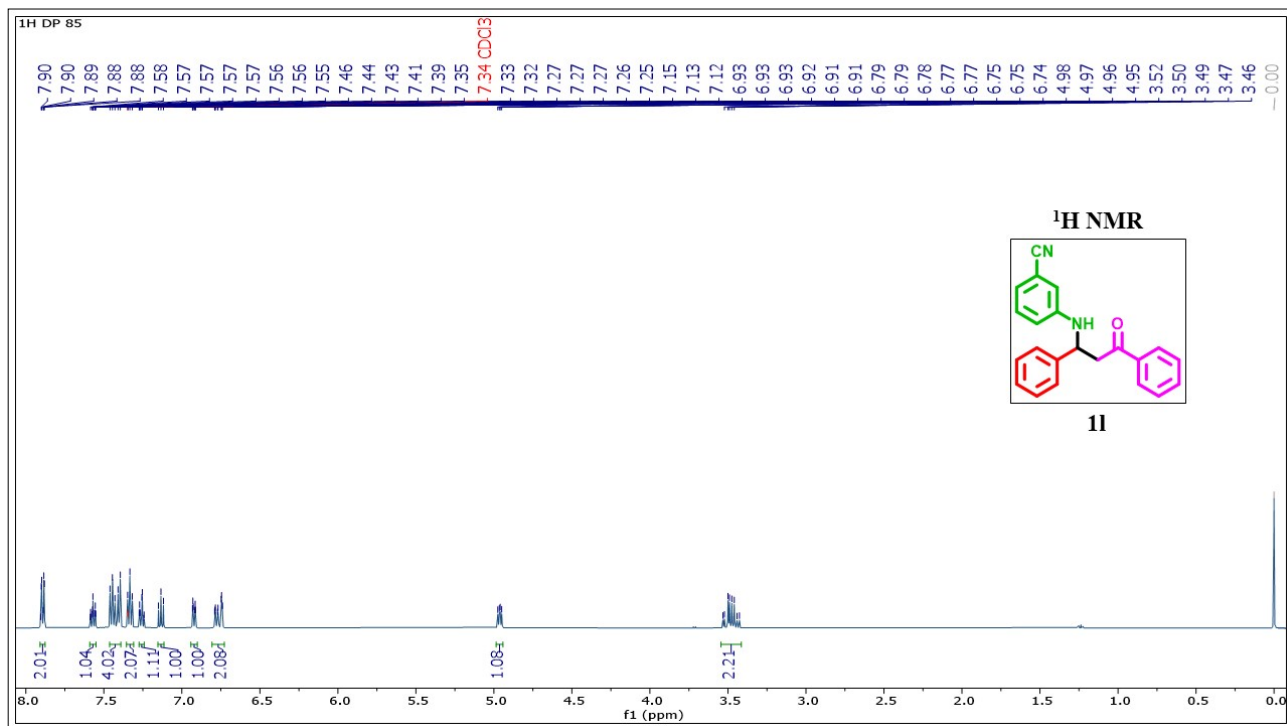


Fig S18- ¹H NMR spectra of 3-((3-oxo-1,3-diphenylpropyl)amino)benzonitrile (**11**)

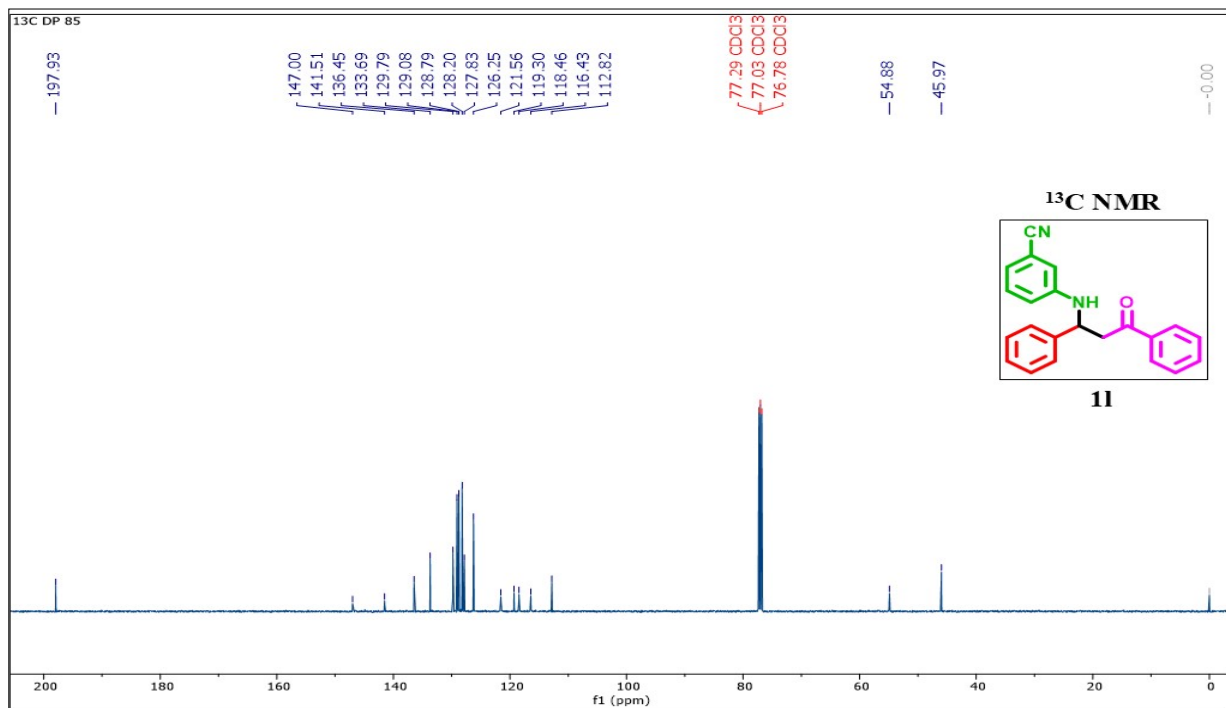


Fig S19-¹³C NMR spectrum of 3-((3-oxo-1,3-diphenylpropyl)amino)benzonitrile (**11**)

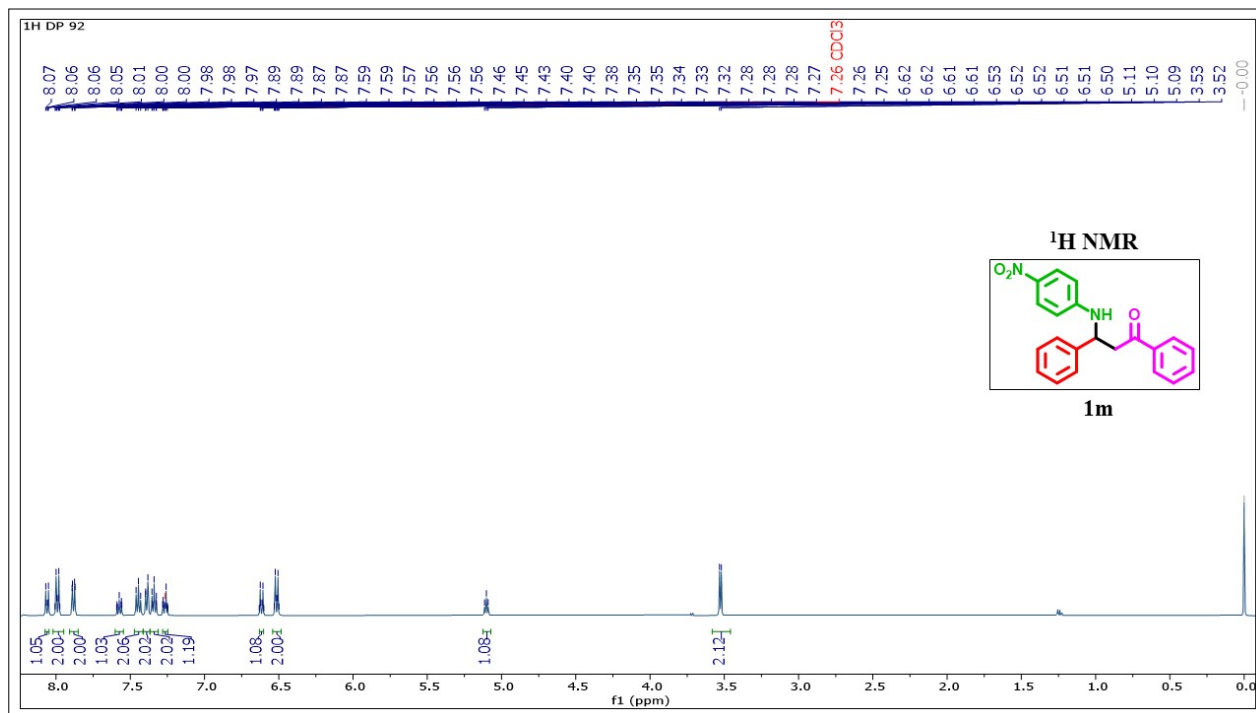


Fig S20- ¹H NMR spectra of 3-((4-nitrophenyl)amino)-1,3-diphenylpropan-1-one (**1m**)

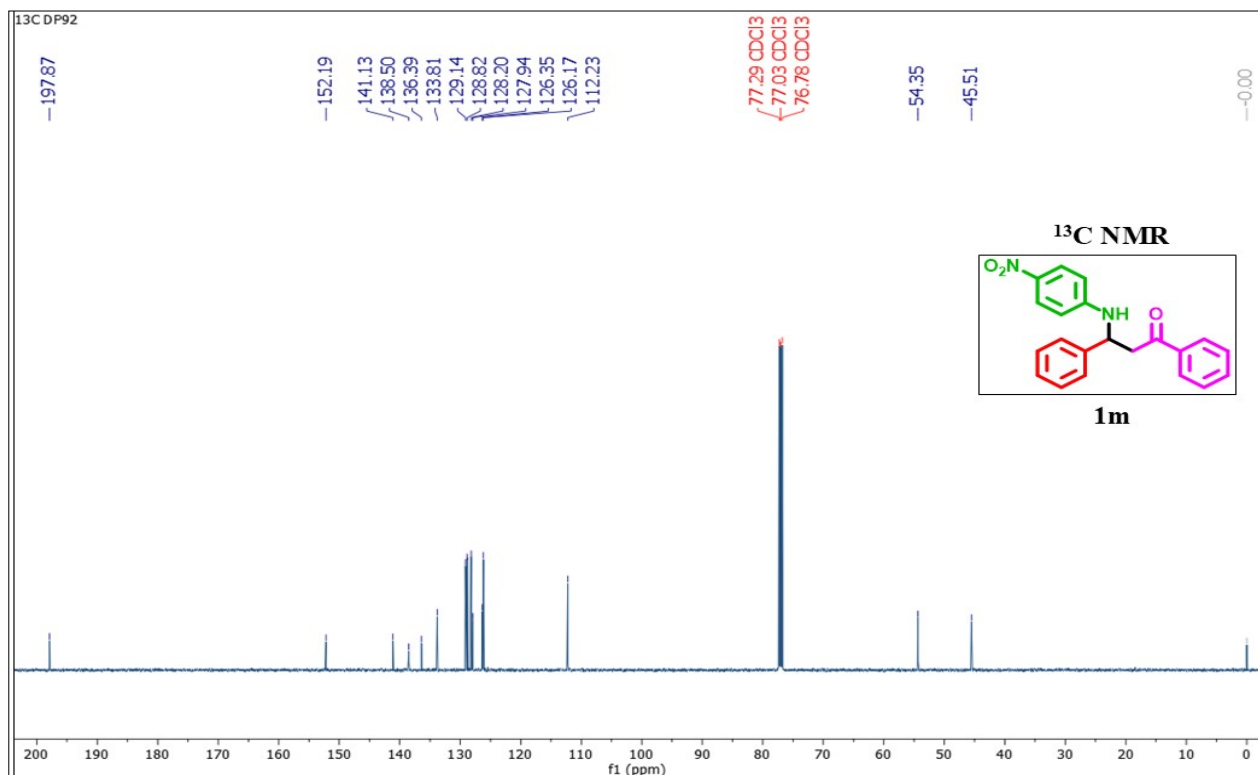


Fig S21-¹³C NMR spectrum of 3-((4-nitrophenyl)amino)-1,3-diphenylpropan-1-one (**1m**)

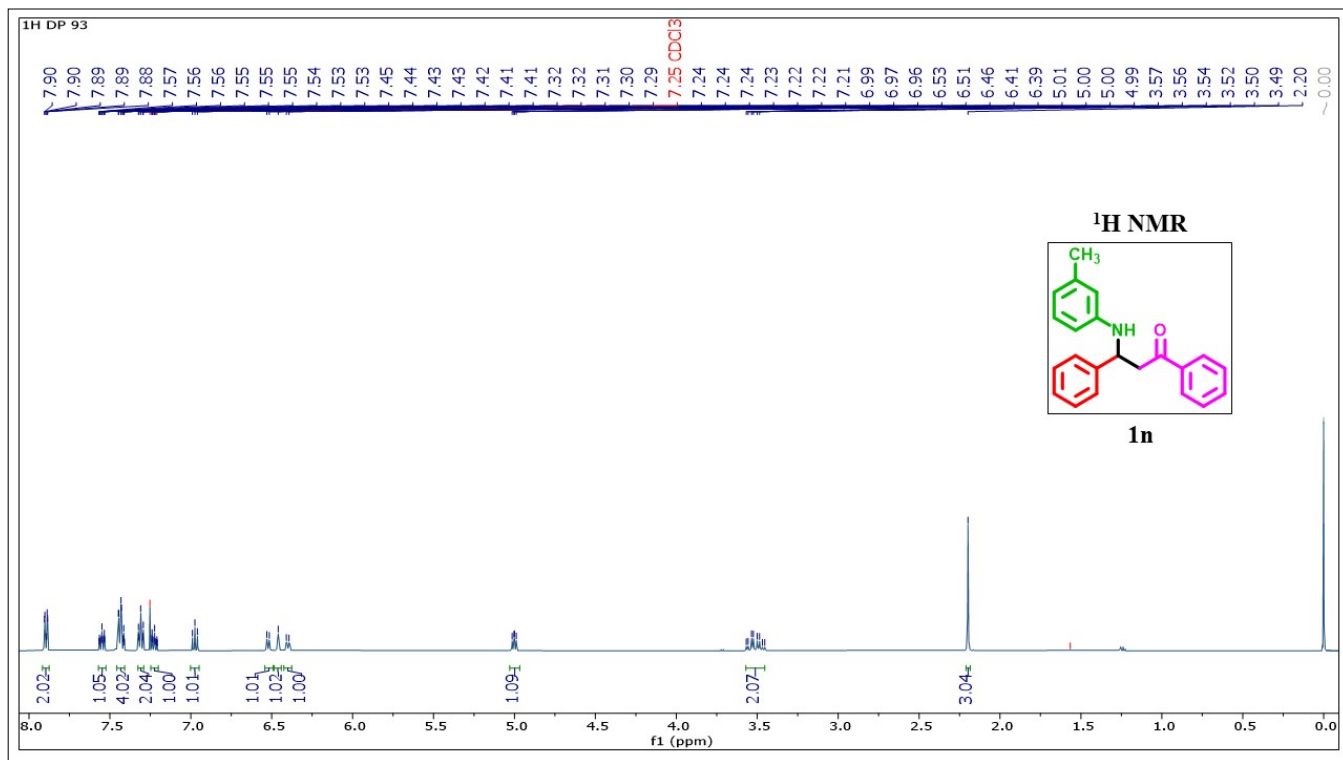


Fig S22- ¹H NMR spectra of 1,3-diphenyl-3-(m-tolylamino)propan-1-one (**1n**)

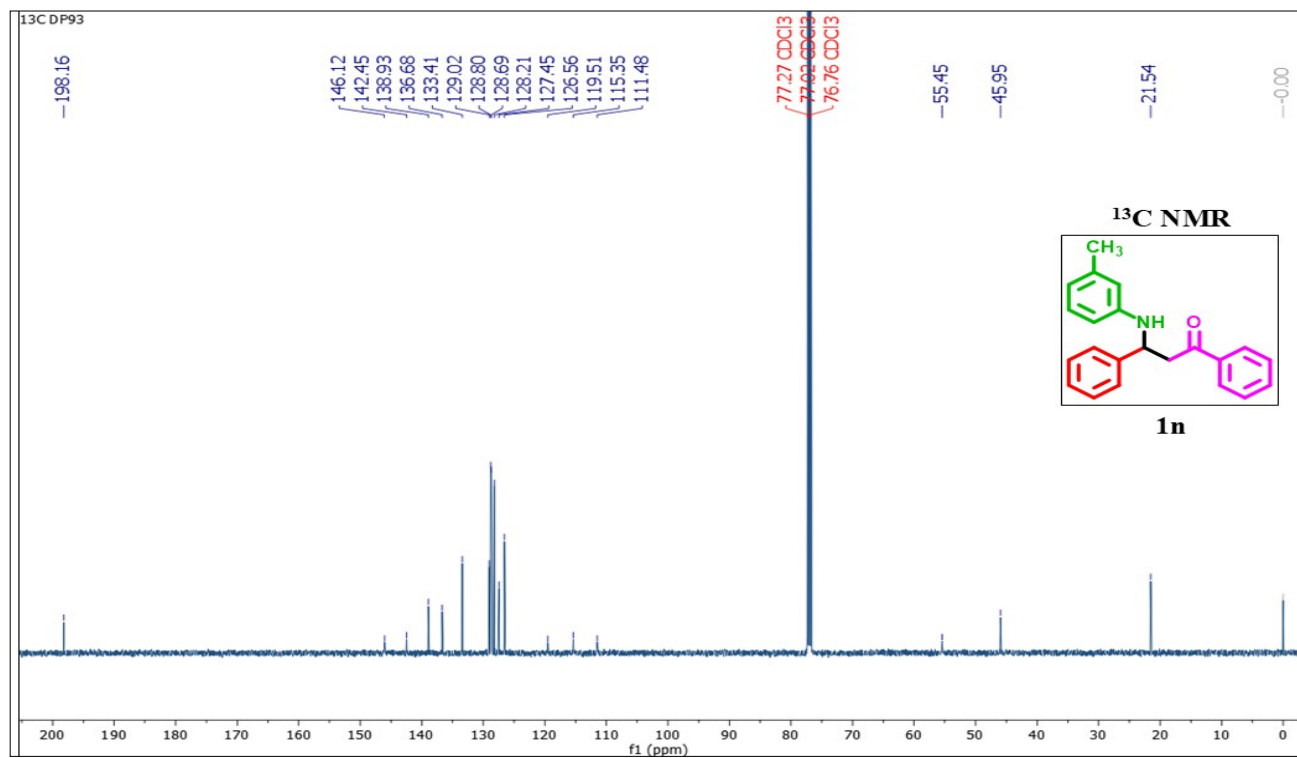


Fig S23-¹³C NMR spectrum of 1,3-diphenyl-3-(m-tolylamino)propan-1-one (**1n**)