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1-Methyl-3-(propyl-3-sulfonic acid)imidazolium triflate supported on magnetic nanoparticles: an efficient and reusable catalyst for synthesis of mono- and bisisobenzofuran-1(3*H*)-ones under solvent-free conditions

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An efficient procedure for the synthesis of isobenzofuran-1(3*H*)-one derivatives in the presence of $[HSO_3PMIM]OTf-SiO_2@MNPs$ as a highly recyclable catalyst under solvent-free thermal conditions and MW irradiation is reported.

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ARTICLE TYPE

1-Methyl-3-(propyl-3-sulfonic acid)imidazolium triflate supported on magnetic nanoparticles: an efficient and reusable catalyst for synthesis of mono- and bis-isobenzofuran-1(3*H*)-ones under solvent-free conditions

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 $\label{eq:heat} 1-Methyl-3-(propyl-3-sulfonic acid) imidazolium triflate supported on magnetic nanoparticles ([HSO_3PMIM]OTf-SiO_2@MNPs) was prepared by immobilization of [HSO_3PMIM]OTf onto the surface of silica-coated Fe_3O_4 nanoparticles and characterized by X-ray diffraction (XRD), transmission electron microscopy (TEM), vibrating sample magnetometery (VSM), and FT-IR techniques. Efficient synthesis$

¹⁵ of mono- and bis-isobenzofuran-1(*3H*)-ones was performed in the presence of this catalyst under thermal conditions and MW irradiation. The catalyst could be easily separated by an external magnet and reused six times under thermal conditions and MW irradiation without significant loss of its activity.

Introduction

During the recent years, ionic liquids (ILs) as environmentally-²⁰ friendly reaction media or catalysts, have received increasing interest due to their distinctive physicochemical properties such as high thermal stability, negligible vapor pressure, nonflammability, and the ability to dissolve many organic and inorganic materials.¹⁻³ In spite of extensive applications in ²⁵ organic reactions, they suffer from disadvantages such as difficult

- ²⁵ organic reactions, they suffer from disadvantages such as difficult product separation, catalyst recovery and the use of large amounts of ILs. Such disadvantages limit their usefulness in large scale operation and lead to serious economical and environmental problems. However, these problems can be solved by ³⁰ immobilization ILs on solid supports such as organic polymers.
- silica, ZrO_2 , TiO_2 and Al_2O_3 and mesoporous MCM-41.⁴⁻¹⁰ These heterogeneous IL catalysts have many advantages over their unsupported counterpart, such as separation, reusability, and the ability to provide practical conveniences in a continuous system.
- ³⁵ In addition, magnetic nanoparticles (MNPs) which have found many applications in the fields of biotechnology and biomedicine¹¹⁻¹⁴ have emerged as viable alternatives to the conventional heterogeneous supports.¹⁵⁻²⁵ The magnetic separation technology offers many advantages over conventional 40 filtration and other purification methods in which the catalysts
- can be simply and efficiently recovered from reaction media

Department of Chemistry, Catalysis Division, University of Isfahan, Isfahan 81746-73441, Iran. Fax: +98-031-36689732; Tel: +98-031-3793270. with the external magnetic field.^{23,24,26-31} This can be considered ⁵⁰ as a green technology that avoids the consequences brought about by filtration steps.

Isobenzofuran-1(3*H*)-ones are an important class of compounds possess a wide range of biological properties such as antibacterial, anti-convulsant, anti-HIV, anti-asthmatic, anti-tumor, anti-platelet activities³²⁻³⁷ as well as anesthesia prolongation, and PGF_{2α} inhibitory properties.³⁸⁻⁴¹ Because of the applications of these compounds in medicine and in the synthesis of natural products,^{42,43} several acidic and basic catalysts such as trifluoroacetic acid (TFA),⁴⁴ trifluoromethanesulfonic acid (HOTf),⁴⁵ Montmorillonite K-10,⁴⁶ NaOH,⁴⁷ KOH,⁴⁸ KF-Al₂O₃,⁴⁹ silica-supported preyssler nanoparticles (H₁₄[NaP₅W₃₀O₁₁₀]/nano-SiO₂)⁵⁰ and ZrOCl₂·8H₂O⁵¹ have been reported for their synthesis.

In continuation of our research on the use of efficient catalytic systems in the synthesis of fine chemicals,⁵²⁻⁵⁷ herein, we wish to report a convenient method for the synthesis of mono- and bis-



Scheme 1 Synthesis of isobenzofuran-1(3*H*)-ones catalyzed by [HSO₃PMIM]OTf-SiO₂@MNPs.

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isobenzofuran-1(3*H*)-ones in the presence of 1-methyl-3-(propyl-3-sulfonic acid)imidazolium triflate supported on magnetic s nanoparticles ([HSO₃PMIM]OTf-SiO₂@MNPs) as a highly reusable catalyst under thermal conditions and MW irradiation (Scheme 1). To the best of our knowledge, this is the first report on the application of MNPs supported IL catalyst in the synthesis of these important heterocyclic compounds.

10 Results and Discussion

The acidic IL supported on magnetic nanoparticles ([HSO₃PMIM]OTf-SiO₂@MNPs) was prepared according to the procedure shown in scheme 2. The ionic liquid [HSO₃PMIM]OTf was prepared by quaternization of 1-methyl-3*H*-imidazole with 15 1,3-propanesultone, followed by treatment with HOTf. The magnetite nanoparticles (MNPs) was easily prepared *via* the coprecipitation method described by Massart⁵⁸ and protected with a layer of silica to prevent aggregation.⁵⁹ The coating process was performed by ultrasonic suspending of MNPs in ethanol and ²⁰ added tetraethoxysilane (TEOS) to form a silica shell under basic conditions through a sol-gel method. Ultimately, a mixture of MNPs and [HSO₃PMIM]OTf in dichloromethane was dispersed by sonication for 5 h.

The prepared catalyst was characterized by X-ray diffraction ²⁵ (XRD), transmission electron microscopy (TEM), vibrating sample magnetometery (VSM) and FT-IR techniques. The XRD

- patterns of Fe_3O_4 magnetic nanoparticles and silica-coated Fe_3O_4 magnetic nanoparticles are shown in Fig. 1. These patterns show the characteristic peaks and relative intensity which are well-³⁰ matched with those of standard Fe_3O_4 nanoparticles (JCPDS file
- No. 19-0629). Moreover, the broad peak from $2\theta = 20^{\circ}$ to 30° is consistent with an amorphous silica phase in the shell of the silica-coated Fe₃O₄ nanoparticles (Fig. 1a).^{60,61} The TEM image of [HSO₃PMIM]OTf-SiO₂@MNPs showed dark nano-Fe₃O₄
- ³⁵ cores surrounded by grey silica shells (Fig 2a). The TEM image of the reused catalyst reveals its stability during the reaction (Fig 2b). The histogram of size distribution shows that the average diameter of nanoparticles is about 8-12 nm (Fig. 2c).
- The magnetic properties of MNPs, $SiO_2@MNPs$ and ⁴⁰ [HSO₃PMIM]OTf-SiO₂@MNPs were measured at room temperature from -10000 to +10000 Oe. The magnetization



Fig. 1 X-Ray diffraction pattern of: a) SiO₂@MNPs and b) standard magnetite pattern (JCPDS No. 19-0629).





Fig. 2 TEM images of: a) [HSO₃PMIM]OTf-SiO₂@MNPs; b) recovered [HSO₃PMIM]OTf-SiO₂@MNPs and c) particle size distribution results for [HSO₃PMIM]OTf-SiO₂@MNPs.



Fig. 3 The VSM result for: a) MNPs; b) SiO₂@MNPs and c) [HSO₃PMIM]OTf-SiO₂@MNPs.

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5 Fig. 4 FT-IR spectra of: a) SiO₂@MNPs; b) [HSO₃PMIM]OTf, c) [HSO₃PMIM]OTf-SiO₂@MNPs and d) the 6-times reused catalyst.

curves for these samples are shown in Fig. 3. The saturation magnetization (Ms) values of samples are 70, 35 and 20 emu.g⁻¹, respectively. The decreasing of Ms values from MNPs to ¹⁰ [HSO₃PMIM]OTf-SiO₂@MNPs could be attributed to the coating of iron oxide magnetic nanoparticles with silica and IL. Furthermore, the magnetization curves of the magnetic

Table 1 Optimization of the reaction conditions for the synthesis of isobenzofuran-1(3H)-ones

5			[HSO ₃ -PMI s Tr	MJOTF-SiO ₂ @MNPs Solvent-free eermal or MW		
	Entry	Method	T (°C)	Catalyst	Time	Yield $(\%)^a$
				amount (mg)		
	1	Thermal	100	0	2.5 h	0
	2 ^b	Thermal	100	25	2.5 h	8
	3 ^c	Thermal	100	-	2.5 h	69
	4	Thermal	100	15	2.5 h	65
	5	Thermal	100	20	2.5 h	82
	6	Thermal	100	25	2.5 h	95
	7	Thermal	100	30	2.5 h	95
	8	Thermal	80	25	2.5 h	72
	9	Thermal	90	25	2.5 h	83
	10	Thermal	110	25	2.5 h	95
	11	MW (550 W)	100	25	10 min	80
	12	MW (600 W)	100	25	10 min	95
	13	MW (650 W)	100	25	10 min	95

^{*a*} Isolated yield. ^{*b*} Reaction was performed in the presence of SiO₂@MNPs. ^{*c*} Reaction was performed in the presence of 0.01 mmol [HSO₃PMIM]OTf.

nanoparticles display no hysteresis before and after ²⁰ functionalization. These results clearly demonstrate their superparamagnetic characteristics,⁵⁸ which further reinforce by easy separation of these magnetic nanoparticles with an external magnetic field.

Further characterization of the catalyst was performed by FT-IR 25 spectroscopy. The FT-IR spectra of blank SiO₂@MNPs, [HSO₃PMIM]OTf and [HSO₃PMIM]OTf-SiO₂@MNPs are presented in Fig. 4. All these samples show broad bands at around 3424 and 1630 cm⁻¹, that are assigned to the Si-OH group and adsorbed water, respectively, in which band at 1630 ³⁰ cm⁻¹ is overlapped with band at 1655 cm⁻¹ (Fig. 4b and 4c). The spectra of the SiO₂@MNPs and [HSO₃PMIM]OTf-SiO₂@MNPs exhibit the characteristic bands at about 1099, 948 and 484 cm⁻¹, which are attributed to the stretching vibrations of Si-O-Si and Fe-O, respectively (Fig. 4a and 4c). Furthermore, in the FT-IR of [HSO₃PMIM]OTf and [HSO₃PMIM]OTf-35 spectra

SiO₂@MNPs, the typical bands at around 1655 cm⁻¹ (C=N), 1572 cm⁻¹ (C=C), 1428 cm⁻¹ (C-H), 1179 and 1030 cm⁻¹ (S=O) were observed (Fig. 4b and 4c). In addition, the characteristic band at 1247 cm⁻¹ is attributed to the CF₃ group. These results confirm ⁴⁰ that IL has been successfully supported on the surface of SiO₂@MNPs (Fig. 4c).

Synthesis of isobenzofuran-1(3*H*)-ones catalyzed by [HSO₃PMIM]OTf-SiO₂@MNPs

The condensation of phthalaldehydic acid (1 mmol) with 45 acetophenone (1 mmol) was chosen as a model reaction for the optimization of parameters such as the amount of catalyst, temperature, and MW power. The results are summarized in Table 1. Initially, the reaction was examined in the absence of the catalyst at 100 °C; no desired product was obtained under this conditions 50 even after 2.5 h. Then, the reaction was performed in the presence of SiO₂@MNPs and [HSO₃PMIM]OTf and the corresponding product was obtained in 8% and 69% yields, respectively. In order to find the optimum amount of [HSO₃PMIM]OTf-SiO₂@MNPs catalyst, the reaction was performed with different amounts of the catalyst (15, 20, 55 25, and 30 mg) under solvent-free conditions at 100 °C. The best yield

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^a Reaction conditions: phthalaldehydic acid (1 mmol), acetophenone (1
 ^s mmol), catalyst (25 mg), Thermal (100 °C) or MW (600 W, 100 °C).
 ^b Isolated yield.

Со ₂ н сно +		D ₃ -PMIMJOTF-SiO₂@MNPs o Solvent⊱free Thermal or MW	-0-(° 5a	° + ↓ 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0
1 mmol	1 mmol	Thermal (3.5 h)	80%	0%
		MW (15 min)	95%	0%
3 mmol	1 mmol	Thermal (3.5 h)	10%	85%
		MW (15 min)	5%	90%
Schomo	Salactiva	synthesis of mono	and his sic	benzofuran 1(3H)

Scheme 3 Selective synthesis of mono- and bis-siobenzofuran-1(3*H*)ones catalyzed by [HSO₃PMIM]OTf-SiO₂@MNPs.

¹⁰ of the desired product was achieved with 25 mg of [HSO₃PMIM]OTf-SiO₂@MNPs. Using lower amount of catalyst resulted in lower yield, while higher amount did not affect the reaction time and yield. To evaluate the influence of temperature, the model reaction was performed in the range of 80-110 °C. It ¹⁵ was found that 100 °C was the optimal temperature and the reaction was incomplete at lower temperature. To find the optimized MW power, the model reaction was carried out in the presence of 25 mg catalyst at 550, 600 and 650 W, and the desired product was obtained in 80%, 95% and 95% yields, ²⁰ respectively. Therefore, 600 W and 100 °C was selected as the optimum power and temperature.

The scope of this protocol was then extended by using a variety of acetophenones to synthesize isobenzofuran-1(3H)-ones. The results are summarized in Table 2. Unsubstituted acetophenone 25 and acetophenones with electron-withdrawing or electrondonating substituents reacted very well with phthalaldehydic acid in the presence of [HSO₃PMIM]OTf-SiO₂@MNPs catalyst under thermal conditions, affording good to excellent yields of the corresponding isobenzofuran-1(3H)-one derivatives (Table 2, 30 entries 1-12). Acid-sensitive ketones such as 2-acetylthiophene and 4-acetylpyridine were also reacted efficiently to give the desired products in high yields (Table 2, entries 13 and 14.). In order to investigate the effect of MW, the reaction of acetophenones with phthalaldehydic acid was performed under 35 the optimized microwave irradiation. Under these conditions, the desired isobenzofuran-1(3H)-ones were obtained in 65-98% yields in very short reaction times (4-12 min) (Table 2). These results clearly demonstrated that the yields were slightly improved, but the reaction times were significantly reduced under 40 microwave irradiation.

Another noteworthy advantage of this catalytic system lies in the selective reaction of 1,4-diacetylbenzene with phthalaldehydic acid. Using a 1:1 molar ratio of 1,4-diacetylbenzene to phthalaldehydic acid, only one acetyl group reacted selectively ⁴⁵ and the corresponding mono-isobenzofuran-1(3*H*)-one **5a** was obtained in 80% and 95% yields under thermal conditions and MW irradiation, respectively. Whereas, with a 1:3 molar ratio of 1,4-diacetylbenzene to phthalaldehydic acid, both acetyl groups reacted and the corresponding bis-isobenzofuran-1(3H)- one **5b** was obtained in 85% and 90% yields, under the above mentioned conditions (Scheme 3). To the best of our knowledge, this is the

s first report on the selective synthesis of mono- and bisisobenzofuran-1(3H)-one, which shows the efficiency and applicability of this catalytic system.

A plausible mechanism for the formation of isobenzofuran-

1(3*H*)-one is proposed in Scheme 4. Initially, [HSO₃PMIM]OTf-¹⁰ SiO₂@MNPs activates the formyl group of phthalaldehydic acid and also catalyzes the keto-enol tautomerization of acetophenone to give **A** and **B**, respectively. Nucleophilic attack of **B** to **A** furnishes the intermediate **C**. The intermediate **C** undergoes intramolecular nucleophilic cyclization in the presence of the ¹⁵ catalyst giving the adduct **D**. Finally, dehydration of **D** in the presence of the catalyst produces the desired isobenzofuran-

1(3H)-one **3** and releases the catalyst for the next run.



Scheme 4 Proposed mechanism for the synthesis of isobenzofuran-1(3*H*)-ones catalyzed by [HSO₃PMIM]OTf-SiO₂@MNP

Recyclability of the catalyst

- ²⁵ Finally, we turned our attention to the possibility of recycling the catalyst, since the recovery and reuse of the catalyst are highly preferable for a green process. In this respect, the reusability of the catalyst was investigated in the reaction of phthalaldehydic acid with acetophenone under thermal conditions
- ³⁰ and MW irradiation. At the end of the reaction, the mixture was cooled to room temperature and chloroform (15 mL) was added. The catalyst was easily separated by a permanent magnet, dried and reused for subsequent reactions. The experimental results suggest that there is no appreciable loss in catalytic activity even
- ³⁵ after sixth recycle (Fig. 5). The comparison of FT-IR spectra of fresh and reused catalyst suggests the stability of the present catalyst during the reaction (Fig. 4d).

Conclusions

In conclusion, we have developed a convenient, efficient, and ⁴⁰ environmentally benign procedure for the preparation of isobenzofuran-1(3*H*)-one derivatives in the presence of [HSO₃PMIM]OTf-SiO₂@MNPs as a highly recyclable catalyst under solvent-free thermal conditions and MW irradiation. Easy work-up, high yields, short reaction times, high atom-economy, ⁴⁵ environmental acceptability, and use of easy recoverable and reusable catalyst are the significant advantages of the present protocol in the synthesis of these important compounds.



Fig. 5 Recycling experiment of [HSO₃PMIM]OTf-SiO₂@MNPs ⁵⁰ under thermal conditions (red) and MW irradiation (green).

Experimental

General information

The chemicals used in this work were purchased from Fluka and Merck chemical companies. Melting points were determined with 55 a Stuart Scientific SMP2 apparatus. FT-IR spectra were recorded on a Nicolet-Impact 400D spectrophotometer. ¹H NMR (400 and 500 MHz) and ¹³C NMR (100 and 125 MHz) spectra were recorded on a Bruker Avance spectrometer using CDCl₃ and DMSO-d₆ as solvent. X-Ray diffraction (XRD) images were $_{60}$ obtained from a Bruker XRD D8 Advance instrument with Co K_a radiation at 40 kV. The transmission electron microscopy (TEM) was carried out on a Philips CM10 transmission electron microscope operating at 200 kV. The magnetic measurements were performed with a vibrating sample magnetometer (VSM) at 65 Meghnatis Daghigh Kavir Co. The microwave system used in these experiments includes the following items: Micro-SYNTH labstation, equipped with a glass door, a dual magnetron system with pyramid shaped diffuser, 1000 W delivered power, exhaust system, magnetic stirrer, 'quality pressure' sensor for flammable 70 organic solvents, and a ATCFO fiber optic system for automatic temperature control.

Synthesis of 1-methyl-3-(propyl-3-sulfonic acid)imidazolium triflate supported on magnetic nanoparticles 75 ([HSO₃PMIM]OTf-SiO₂@MNPs)

Synthesis of 1-methyl-3-(propyl-3-sulfonic acid)imidazolium triflate ([HSO₃PMIM]OTf) ionic liquid. A mixture of 1-methyl-3*H*-imidazole (0.82 g, 10 mmol) and 1,3-propanesultone

(1.22 g, 10 mmol) was stirred magnetically for 72 h at room temperature. The resulting white solid was washed with diethyl ether (3×10 mL) and dried in vacuum to afford the desired zwitterion in 90% yield. Then, trifluoromethanesulfonic acid (1.5

- ⁵ g, 10 mmol) was added to this white solid zwitterion and the mixture was stirred for 2 h at 40 °C. The resulting crude material was washed repeatedly with toluene and diethyl ether and dried in vacuum to give 1-methyl-3-(propyl-3-sulfonic acid)imidazolium triflate ([HSO₃PMIM]OTf) ionic liquid in quantitative yield. ¹H
 ¹⁰ NMR (400 MHz, D₂O): δ 8.54 (s, 1H), 7.36 (dd, 1H, *J* = 15.2, 8.0 Hz), 7.26 (dd, 1H, *J* = 18.0, 6.8 Hz), 4.19 (t, 2H, *J* = 7.2 Hz), 3.73 (s, 3H), 2.77 (t, 2H, *J* = 7.2 Hz), 2.16 (q, 2H, *J* = 7.6 Hz). ¹³C
- (s, 511), 2.77 (t, 211, 3 7.2 112), 2.10 (d, 211, 3 7.0 112). C NMR (100 MHz, D₂O): δ 137.0, 135.8, 123.5, 121.9, 47.5, 47.0, 35.5, 24.8.
- 15 Synthesis of silica-coated Fe₃O₄ nanoparticles (SiO₂@MNPs). Magnetite nanoparticles (MNPs) were prepared according to the reported procedure.⁵⁸ In this manner, FeCl₃·6H₂O (11.0 g) and FeCl₂·4H₂O (4.0 g) were dissolved in 250 mL deionized water under N₂ with vigorous stirring at 85 °C,
 20 during which the pH of the solution adjusted to 9 using conc. NH₃·H₂O. The mixture was further stirred for 4 h and the resulting MNPs precipitates were washed with deionized water and ethanol until the pH reached 7. The black precipitate (MNPs) was collected with a permanent magnet. For coating of a layer of 25 silica on the surface of MNPs, the MNPs (2.0 g) in ethanol (400
- ml) were sonicated for 30 min at room temperature under N₂. Then, conc. NH₃·H₂O (12.0 mL) and TEOS (4.0 mL) was added sequentially, and the mixture was dispersed for 24 h under the above mentioned conditions. Finally, the black precipitate ³⁰ (SiO₂@MNPs) was collected using a permanent magnet, washed with ethanol three times and dried in a vacuum.

Synthesis of [HSO₃PMIM]OTf-SiO₂@MNPs. 5.0 g of SiO₂@MNPs was dispersed in 50 mL CH₂Cl₂ by sonication for 1 h. Then, a solution of [HSO₃PMIM]OTf (1.0 g) in CH₂Cl₂ (15

- ³⁵ mL) was added, and the mixture was sonicated for 5 h. Finally, the [HSO₃PMIM]OTf-SiO₂@MNPs catalyst was separated by a permanent magnet, washed with *n*-hexane (3×15 mL) and dried under vacuum. The loading of the catalyst was determined by elemental analysis to be 14% W.
- ⁴⁰ General procedure for the synthesis of isobenzofuran-1(3*H*)ones under thermal conditions and MW irradiation.

A mixture of phthaladehyic acid (1 mmol), acetophenone (1 mmol) and [HSO₃PMIM]OTf-SiO₂@MNPs (25.0 mg) was stirred at 100 °C or subjected to MW irradiation (600 W, 100 °C) for the

- ⁴⁵ appropriate time according to Table 2. The progress of the reaction was monitored by TLC (eluent: petroleum ether/ethyl acetate, 5:2). After completion of the reaction, the mixture was cooled to room temperature, chloroform (15 mL) was added and the catalyst was easily separated by a permanent magnet. The
- ⁵⁰ filtrate was evaporated and the crude product was purified by chromatography on silica gel (eluent: petroleum ether/ethyl acetate, 5:2) or by recrystallization from acetone and ethanol (1:2) to afford the pure product.

3-(2-Oxo-2-phenyl)isobenzofuran-1(3H)-one (3a): Mp: 143-

- ⁵⁵ 145 °C (Ref.⁴⁴ 145 °C). FT-IR (KBr, cm⁻¹): v_{max} 3053, 2930, 1629, 1466, 1365, 1335, 1098, 1045, 793. ¹H NMR (400 MHz, CDCl₃): δ 7.93-7.98 (m, 3H), 7.56-7.70 (m, 4H), 7.49-7.54 (m, 2H), 6.20 (t, 1H, J = 6.6 Hz), 3.80 (dd, 1H, J = 17.6, 5.6 Hz), 3.41 (dd, 1H, J = 17.6, 7.6 Hz).
- ⁶⁰ **3-[2-(4-Cyclohexylphenyl)-2-oxo-ethyl]isobenzofuran-1(3***H***)one (3b): Mp: 188-191 °C. FT-IR (KBr, cm⁻¹): v_{max} 3043, 2927, 2790, 1760, 1674, 1605, 1445, 1294, 1088, 972, 826, 760. ¹H NMR(400 MHz, CDCl₃): \delta 7.89-7.94 (m, 3H), 7.65-7.68 (m, 1H), 7.49-7.59 (m, 2H), 7.27-7.40 (m, 2H), 6.20 (t, 1H,** *J* **= 6.4 Hz),**

- ⁶⁵ 3.78 (dd, 1H, *J* = 17.6, 5.6 Hz), 3.37 (dd, 1H, *J* = 17.6, 7.6 Hz), 2.58 (s, 1H), 1.86-1.95 (m, 4H), 1.78 (d, 1H, *J* = 12.4 Hz),1.41-1.58 (m, 4H), 1.28-1.36 (m, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 195.7, 170.2, 154.7, 149.9, 134.2, 134.1, 129.4, 128.4, 127.3, 126.0, 125.7, 122.9, 77.4, 44.8, 43.6, 34.1, 26.7, 26.0.
- ⁷⁰ **3-[2-(4-Methoxyphenyl)-2-oxoethyl]isobenzofuran-1(3***H***)-one
 (3c): Mp: 118-120 °C (Ref.⁴⁸ 120 °C). FT-IR (KBr, cm⁻¹): ν_{max} 3116, 2966, 1786, 1763, 1607, 1355, 1282, 1048, 940, 838, 751.
 ¹H NMR (500 MHz, CDCl₃): δ 7.92-7.96 (m, 3H), 7.65-7.68 (m, 1H), 7.53-7.59 (m, 2H), 6.96 (d, 2H, J = 8.8 Hz), 6.18 (t, 1H, J = 4.6 Hz), 200 (-2H), 251 (t) HW J = 5.5 Hz**
- ⁷⁵ 6.6 Hz), 3.89 (s, 3H), 3.74 (dd, 1H, J = 17.4, 5.7 Hz), 3.34 (dd, 1H, J = 17.3, 7.5 Hz).
- **3-[2-(4-Methylphenyl)-2-oxo-ethyl]isobenzofuran-1(3H)-one** (**3d**): Mp: 147-149 °C (Ref.⁵⁰ 149 °C). FT-IR (KBr, cm⁻¹): v_{max} 2903, 1762, 1673, 1604, 1296, 1082, 942, 800, 763. ¹H NMR (400 MHz (CDCI)): δ 7.00 (4. 11) μ = 7.00 7.05 7.05 (4. 11)
- ⁸⁰ (400 MHz, CDCl₃): δ 7.90 (d, 1H, J = 7.6),7.85-7.87 (m, 2H), 7.64-7.68 (m, 1H), 7.53-7.58 (m, 2H),7.27-7.29 (m, 2H), 6.18 (t, 1H, J = 6.5 Hz), 3.76 (dd, 1H, J = 17.5, 5.7 Hz), 3.37 (dd, 1H, J = 16.2, 7.5 Hz), 2.42 (s, 3H).

3-[2-(3,4-Dimethoxyphenyl)-2-oxo-ethyl]isobenzofuran-1(3H)-

- ⁸⁵ **one (3e):** Mp: 116-119 °C. FT-IR (KBr, cm⁻¹): v_{max} 2965, 1765, 1650, 1469, 1256, 1171, 1034, 941, 750. ¹H NMR (400 MHz, CDCl₃): δ 7.92 (d, 1 H, J = 7.6 Hz), 7.65-7.69 (m, 2H), 7.57-7-60 (m, 2H), 7.54 (d, 2H, J = 2.0 Hz), 6.18 (t, 1H, J = 6.6 Hz), 3.96 (s. 3H), 3.95 (s, 3H), 3.75 (dd, 1H, J = 17.2, 6.0 Hz), 3.37 (dd,
- ⁹⁰ 1H, J = 17.2, 7.2 Hz). ¹³C NMR (100 MHz, CDCl₃): δ 194.5, 170.2, 153.9, 149.8, 149.2, 134.3, 129.4, 129.3, 125.9, 125.7, 123.2, 122.9, 110.1, 110.0, 77.5, 56.2, 56.0, 43.2.

3-[2-(2-Hydroxyphenyl)-2-oxo-ethyl]isobenzofuran-1(3*H***)one (3f**): Mp: 134-136 °C (Ref.⁴⁷ 134-136 °C). FT-IR (KBr, cm⁻¹):

⁹⁵ v_{max} 3038, 2920, 1773, 1634, 1443, 1297, 1078, 965, 759. ¹H NMR (500 MHz, CDCl₃): δ 12.03 (s, 1H), 7.94 (d, 1H, *J* = 7.6 Hz), 7.65-7.72 (m, 2H), 7.56-7.59 (m, 2H), 7.51-7.54 (m, 1H), 7.04 (dd, 1H, *J* = 8.4, 1.0 Hz), 6.90-6.93 (m, 1H), 6.17 (t, 1H, *J* = 6.5 Hz), 3.8 (dd, 1H, *J* = 17.6, 6.2 Hz), 3.45 (dd, 1H, *J* = 17.6, 6.8 I00 Hz)

3-[2-(4-Nitrophenyl)-2-oxo-ethyl]isobenzofuran-1(3*H***)-one (3g**): Mp: 208-210 °C (Ref.⁵¹ 210 °C). FT-IR (KBr, cm⁻¹): v_{max} 3104, 2909, 1748, 1689, 1603, 1456, 1320, 1270, 1085, 982, 854,

- 5104, 2909, 1748, 1689, 1603, 1436, 1520, 1270, 1083, 982, 834, 752. ¹H NMR (400 MHz, CDCl₃): δ 7.93 (d, 1H, J = 7.6), 7.86-105 7.90 (m, 2H), 7.66-7.74 (m, 3H), 7.55-7.58 (m, 2H), 6.16 (t, 1H, J
- = 6.2 Hz), 3.72 (dd, 1H, *J* = 17.0, 5.8 Hz), 3.36 (dd, 1H, *J* = 17.6, 6.8 Hz).

3-[2-(3-Chlorophenyl)-2-oxo-ethyl]isobenzofuran-1(3*H*)-one

- (3h): Mp: 142-144 °C. FT-IR (KBr, cm⁻¹): v_{max} 2927, 1754, 1634, 110 1423, 1227, 1163, 1097, 946, 799. ¹H NMR (400 MHz, CDCl₃): δ 7.76-7.84 (m, 4H), 7.61-7.65 (m, 1H), 7.47-7.55 (m, 2H), 7.37 (t, 1H, J = 6.4 Hz), 6.08 (t, 1H, J = 6.4 Hz), 3.64 (dd, 1H, J = 17.8, 6.6 Hz), 3.42 (dd, 1H, J = 17.8, 6.2 Hz). ¹³C NMR (100 MHz, CDCl₃): δ 194.7, 170.0, 149.4, 137.6, 135.0, 134.4, 133.6, 115 130.2, 129.5, 128.1, 126.4, 125.8, 125.6, 122.7, 76.9, 42.9.
- **3-[2-(4-Fluorophenyl)-2-oxo-ethyl]isobenzofuran-1(3H)-one** (**3i):** Mp: 132-134 °C (Ref.⁴⁸130-133 °C). FT-IR (KBr, cm⁻¹): v_{max} 3080, 2965, 2911, 1756, 1681, 1596, 1213, 973, 837. ¹H NMR (500 MHz, CDCl₃): δ 7.98-8.00 (m, 2H), 7.92 (d, 1H, *J* = 7.6 Hz),
- ¹²⁰ 7.67 (t, 1H, J = 7.5 Hz), 7.53-7.57 (m, 2H), 7.15 (t, 2H, J = 8.5), 6.16 (t, 1H, J = 6.5 Hz), 3.74 (dd, 1H, J = 17.5, 6.0 Hz), 3.38 (dd, 1H, J = 17.5, 7.0 Hz).

3-[2-(4-Chlorophenyl)-2-oxo-ethyl]isobenzofuran-1(3H)-one

(3j): Mp: 144-146 °C (Ref.⁴⁴ 146 °C). FT-IR (KBr, cm⁻¹): v_{max} 3070, 2924, 1753, 1680, 1590, 1217, 1085, 988, 821. ¹H NMR (500 MHz, CDCl₃): δ 7.89-7.92 (m, 3H), 7.67 (t, 1H, J = 7.5 Hz), 5 7.54-7.57 (m, 2H), 7.46 (d, 2H, J = 8.6 Hz), 6.16 (t, 1H, J = 6.5 Hz), 3.73 (dd, 1H, J = 17.5, 6.0 Hz), 3.38 (dd, 1H, J = 17.5, 7.0 Hz).

3-[2-(4-Bromophenyl)-2-oxo-ethyl]isobenzofuran-1(3H)-one

(3k): Mp: 146-148 °C. FT-IR (KBr, cm⁻¹): v_{max} 3040, 2965, 1760, 10 1681, 1583, 1405, 1290, 1215, 965, 756. ¹H NMR (500 MHz, CDCl₃): δ 7.92 (d, 1H, J = 7.8 Hz), 7.81-7.83 (m, 2H), 7.66-7.69 (m, 1H), 7.62-7.64 (m, 2H), 7.54-7.57 (m, 2H), 6.16 (t, 1H, J = 6.5 Hz), 3.72 (dd, 1H, J = 17.5, 6.0 Hz), 3.37 (dd, 1H, J = 17.5, 7.0 Hz).

15 3-[2-(4-Iodophenyl)-2-oxo-ethyl]isobenzofuran-1(3H)-one

- (31): Mp: 174-175 °C. FT-IR (KBr, cm⁻¹): v_{max} 3085, 2903, 1758, 1678, 1457, 1295, 1076, 987, 765. ¹H NMR (500 MHz, CDCl₃): δ 7.93(d, 1H J = 7.7 Hz), 7.86-7.88 (m, 2H), 7.66-7.70 (m, 3H), 7.57 (t, 2H, J = 7.0 Hz), 6.16 (t, 1H, J = 6.3 Hz), 3.72 (dd, 1H, J
- $_{20} = 17.6, 5.8$ Hz), 3.37 (dd, 1H, J = 17.6, 6.8 Hz). 13 C NMR (125 MHz, CDCl₃): δ 195.3, 170.0, 149.6, 138.2, 135.5, 134.3, 129.5, 129.4, 125.9, 122.7, 102.1, 76.9, 74.0, 43.6.

3-(2-Oxo-2-thiophen-2-yl-ethyl)isobenzofuran-1(3H)-one

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- (3m): Mp: 132-135 °C. FT-IR (KBr, cm⁻¹): v_{max} 3084, 2926, ²⁵ 1767, 1654, 1519, 1291, 1067, 855, 754. ¹H NMR (400 MHz, CDCl₃): δ 7.94 (d, 1H, J = 7.6 Hz), 7.67-7.72 (m, 3H), 7-55-7.60 (m, 2H), 7.17 (s, 1H), 6.17 (t, 1H, J = 6.4 Hz), 3.70 (dd, 1H, J = 16.6, 6.0 Hz), 3.35 (dd, 1H, J = 16.8, 6.8 Hz). ¹³C NMR (100 MHz, Acetone- d_6): δ 189.7, 170.4, 150.8, 144.8, 135.6, 135.0, ³⁰ 134.3, 130.2, 129.4, 127.0, 125.9, 123.7, 77.8, 44.3.
- **3-[2-Oxo-2-(pyridine-4-yl)ethyl]isobenzofuran-1(3H)-one** (**3n):** Mp: 168-170 °C. FT-IR (KBr, cm⁻¹): v_{max} 3071, 2959, 2928, 2859, 1770, 1730, 1600, 1465, 1352, 1285, 1125, 1070, 916, 747. ¹H NMR (500 MHz, CDCl₃): δ 8.82-8.83 (m, 2H), 7.90 (d, 1H, J
- ³⁵ = 7.6 Hz), 7.66-7.73 (m, 3H), 7.54-7.57 (m, 2H), 6.14 (t, 1H, J = 5.0 Hz), 3.74 (dd, 1H, J = 17.9, 6.3 Hz), 3.43 (dd, 1H, J = 17.9, 6.5 Hz). ¹³C NMR (125MHz, CDCl₃): δ 195.7, 169.9, 151.2, 149.2, 141.8, 134.4, 128.7, 125.9, 125.8, 122.7, 120.9, 76.5, 43.9. **3-[2-(4-Acetylphenyl)-2-oxo-ethyl]isobenzofuran-1(3***H***)-one**
- ⁴⁰ (**5a**): Mp: 135-137 °C. FT-IR (KBr, cm⁻¹): v_{max} 2923, 1758, 1679, 1608, 1403, 1358, 1215, 970, 829, 755. ¹H NMR (400 MHz, CDCl₃): δ 7.60-7.65 (m, 4H), 7.50 (dd, 2H, *J* = 7.0, 2.2 Hz), 7.45-7.48 (m, 2H), 6.11 (t, 1H, *J* = 6.4 Hz), 3.72 (dd, 1H, *J* = 17.6, 6.0 Hz), 3.37 (dd, 1H, *J* = 17.6, 6.8 Hz), 2.59 (s, 3H). ¹³C NMR (100
- ⁴⁵ MHz, DMSO-*d*₆): δ 196.7, 196.3, 169.7, 149.7, 139.4, 134.3, 129.3, 128.5, 125.4, 124.9, 123.5, 122.8, 122.3, 76.9, 43.1, 27.0. **3,3'-(1,4-Phenylenebis(2-oxoethane-2,1-**

diyl))bis(isobenzofuran-1(3*H***)-one (5b):** Mp: 273-275 °C. FT-IR (KBr, cm⁻¹) v_{max} 3094, 1756, 1676, 1294, 1223, 1087, 969, 759,

⁵⁰ 690. ¹H NMR (400 MHz, DMSO- d_6): δ 8.08-8.18 (m, 4H), 7.86-7.94 (m, 3H), 7.75-7.83 (m, 4H), 7.61-7.69 (m, 1H), 6.14 (dd, 2H, *J* = 7.8, 3.8 Hz), 3.95 (dd, 2H, *J* = 18.4, 3.8 Hz), 3.81 (dd, 2H, *J* = 18.4, 8.4 Hz). ¹³C NMR (100 MHz, DMSO- d_6): δ 206.5, 196.4, 139.8, 138.4, 134.3, 129.3, 124.9, 122.9, 119.1, 117.4, ⁵⁵ 76.9, 30.6.

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