ISSN 1070-3632, Russian Journal of General Chemistry, 2017, Vol. 87, No. 12, pp. 2922–2929. © Pleiades Publishing, Ltd., 2017.

One-Pot Synthesis of 1,4-Dihydropyridine Derivatives Catalyzed by Silica-Coated Magnetic $NiFe_2O_4$ Nanoparticles-Supported $H_{14}[NaP_5W_{30}O_{110}]$[1]

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Received April 20, 2017

Abstract—Silica-coated magnetic $NiFe_2O_4$ nanoparticles-supported $H_{14}[NaP_5W_{30}O_{110}]$ ($NiFe_2O_4$@SiO_2-$H_{14}[NaP_5W_{30}O_{110}]$) was successfully synthesized and shown to be a versatile and highly efficient heterogeneous catalyst for one-pot multicomponent synthesis of 1,4-dihydropyridine derivatives under solvent-free condition. The synthesized catalyst can be magnetically recovered and reused four times without significant loss in catalytic efficiency.

Keywords: $NiFe_2O_4$@SiO_2-$H_{14}[NaP_5W_{30}O_{110}]$, 1,4-dihydropyridines, magnetically recyclable catalyst

DOI: 10.1134/S1070363217120325

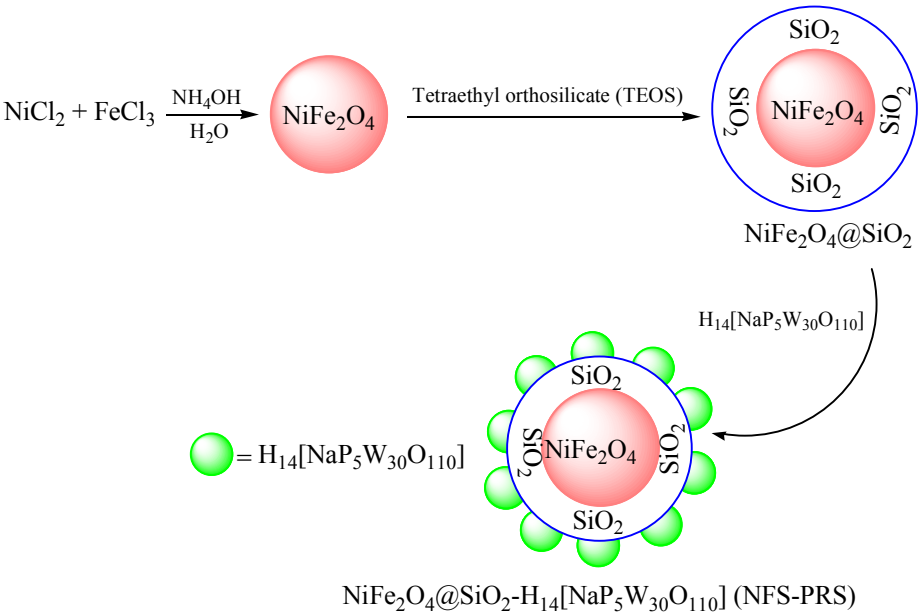
Multicomponent reactions (MCRs) are widely used in heterocyclic synthesis due to their advantages such as atom economy and bond-forming efficiency both of which belong to the green chemistry principles [1]. These reactions can also be used in the synthesis of 1,4-dihydropyridines which have attracted significant attention due to their biological activity, such as antitumor, vasodilator, bronchodilator, antiatherosclerotic, and geroprotective [2]. Furthermore, 1,4-dihydropyridine derivatives are analogs of NADH coenzymes [3] which can be used for the treatment of Alzheimer's disease and tumor therapy [4]. Some cardiovascular agent such as nifepidine, nicardipine, and amlodipine are dihydropyridine derivatives important for the treatment of hypertension [5].

Polyhydroquinolines, 1,8-dioxohexahydroacridines, and 2,5-dioxo-1,2,3,4,5,6,7,8-octahydroquinolines have shown various biological activities. Polyhydroquinolines are used in the synthesis of valuable drugs, includng those for the treatment of angina pectoris and cardiovascular diseases [6, 7]. Acridine derivatives containing a 1,4-showed a positive ionotropic effect which improves the entry of calcium to the intracellular space [8], as well as antimalarial [9], antitumor [10], antibacterial [11], antitubercular [12], and acetylcholinesterase inhibitory activities [13]. Acridine derivatives were also used in the synthesis of conjugates with peptides, proteins, and nucleic acids, which demonstrated DNA-binding properties [14].

Due to biological importance of 1,4-dihydropyridine derivatives, many classical methods have been reported for their synthesis [15]. Some other synthetic procedures have recently been proposed for the synthesis of polyhydroquinolines, 1,8-dioxohexahydroacridines, and 2,5-dioxo-1,2,3,4,5,6,7,8-octahydroquinolines in presence of various catalysts such as Co_3O_4-CNTs [16], Fe_3O_4@chitosan [17], SBA-15/SO_3H [18], poly(AMPS-co-AA) [19], 1,3-di(bromo or chloro)-5,5-dimethylhydantoin [20], urea [21], anilines or ammonium acetate [22] in the presence of *p*-dodecylbenzenesulfonic acid (DBSA) [23], Amberlyst-15 [24], ammonium chloride, $Zn(OAc)_2 \cdot H_2O$, or L-proline [25], ZnO nanoparticles [26], nano-Fe_3O_4 [27], 1-methylimidazolium trifluoroacetate ([Hmim]TFA) [28], nanoporous silica-supported ionic liquid (SBA-IL) [29], silica-based sulfonic acid [30] under microwave irradiation [31], $HClO_4$–SiO_2 [32], and ceric ammonium nitrate (CAN) [33].

[1] The text was submitted by the authors in English.

Scheme 1.



Despite obvious advantages, the above synthetic methods also suffer from some limitations such as low yields, difficult preparations of catalysts, and long reaction time. In order to overcome these restrictions, in continuation of our previous studies on organic reactions [34] we have become interested in using a nanocatalyst with a supported heteropolyacid for the synthesis of polyhydroquinolines, 1,8-dioxohexahydroacridines, and 2,5-dioxo-1,2,3,4,5,6,7,8-octahydroquinolines.

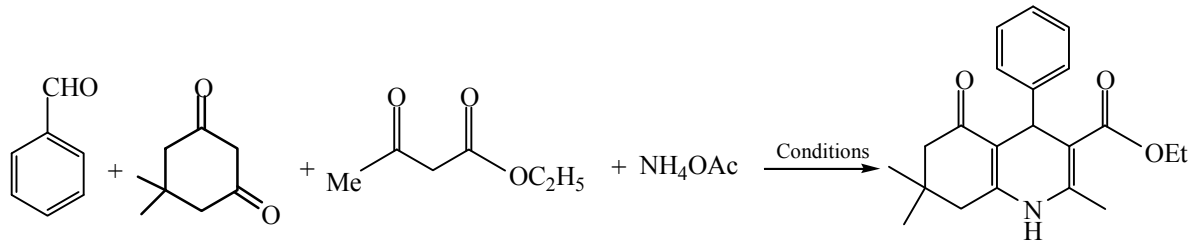
Nanocatalysts are the best alternatives to usual catalysts because they have a large surface-to-volume ratio which increases their catalytic activity. Also, their selectivity and stability can be improved by changing the size, shape, and composition. Nanocatalysts can be recovered from the reaction mixture by filtration or centrifugation, though these separation methods could lead to loss of nanocatalyst [35]. The best solution for this problem is the use of magnetic nanoparticles which have attracted great attentions due to their wide application in biological researches [36]. Magnetic nanoparticles (MNPs) can act as supports to immobilize the catalyst which can be easily separated from the reaction mixture using an external magnet [37]. Preyssler heteropolyacid $H_{14}NaP_5W_{30}O_{120}$ (HPA) has remarkable properties like high thermal and hydrolytic stability, acidic protons, high solubility in polar solvents, low surface area, and safety. We utilized $NiFe_2O_4$ as magnetic source because of its remarkable properties including high saturation, high Curie temperature, magnetization, and relatively high permeability.

The catalyst $NiFe_2O_4@SiO_2$-$H_{14}[NaP_5W_{30}O_{110}]$ (NFS-PRS) was synthesized as shown in Scheme 1 and was characterized by FT-IR, scanning electron microscopy, transmission electron microscopy, X-ray diffraction, energy-dispersive X-ray spectroscopy, and vibrating sample magnetometry (the data are available from the authors as supporting information).

The obtained catalyst was used first in the synthesis of hexahydroquinoline **4a** as model reaction (Scheme 2, Table 1). For this purpose, 0.02 g of the catalyst was added to a mixture of benzaldehyde (1 mmol), ammonium acetate (5 mmol), ethyl acetoacetate (1 mmol), and dimedone (1 mmol), and the mixture was heated at 100–130°C under solvent-free conditions (Table 1, entry nos. 1–4). The best results were obtained at 120°C (entry no. 3). We then tested different amounts of the catalyst (entry nos. 5, 6) and found that the optimum amount of the catalyst was 0.02 g (run no. 3). In addition, the reaction was carried out in different solvents under reflux, but the results were unsatisfactory (entry nos. 7, 8).

The scope and generality of this four-component one-pot Hantzsch synthesis of polyhydroquinoline derivatives was demonstrated with different 1,3-dicarbonyl compounds and aldehydes (Scheme 3, Table 2). All reactions proceeded efficiently within 10–60 min at 120°C under solvent-free condition to

Table 1. Optimization of the synthesis of hexahydroquinoline **4a** (Scheme 1)



1 **2** **3** **4a**

Entry no.	Catalyst, g	Conditions	Reaction time, min	Yield,[a] %
1	NFS-PRS, 0.02 g	Solvent-free/100°C	10	72
2	NFS-PRS, 0.02 g	Solvent-free/110°C	10	80
3	NFS-PRS, 0.02 g	Solvent-free/120°C	10	94
4	NFS-PRS, 0.02 g	Solvent-free/130°C	10	92
5	NFS-PRS, 0.01 g	Solvent-free/120°C	20	82
6	NFS-PRS, 0.03 g	Solvent-free/120°C	10	94
7	NFS-PRS, 0.02 g	Ethanol/reflux	60	68
8	NFS-PRS, 0.02 g	Methanol/reflux	60	65

[a] Isolated yield.

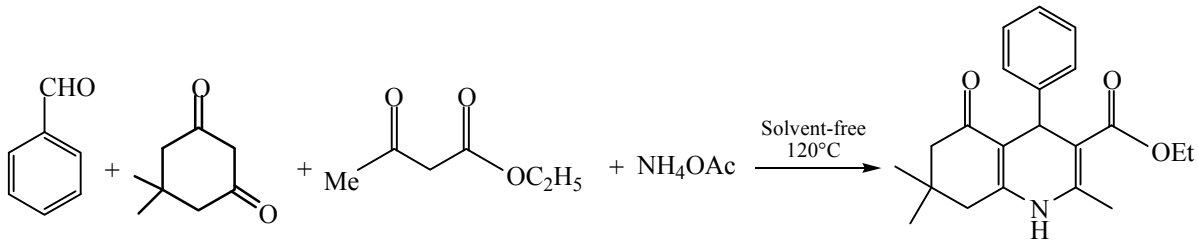
afford the corresponding polyhydroquinoline derivatives in good yields (77–96%).

We also used NFS-PRS to catalyze the synthesis of 1,8-dioxodecahydroacridines under analogous conditions. The catalyst was added to a mixture of an aromatic aldehyde (1 mmol), 5,5-dimethylcyclohexane-1,3-dione or cyclohexane-1,3-dione (2 mmol), and ammonium acetate (5 mmol). The progress of the reaction was monitored by TLC; after reaction completion, the catalyst was easily separated by an external magnet. We then used different substituted anilines as a source of nitrogen instead of ammonium acetate (Table 2). Anilines containing electron-donating groups (R^3 = H, 4-Me, 4-MeO) smoothly reacted under the given conditions to afford the corresponding 10-substituted 1,8-dioxodecahydroacridines in 75–92% yield, whereas anilines with electron-withdrawing groups failed to react.

Having obtained satisfactory results, we continued to study the reaction using dimedone, Meldrum's acid, various aldehydes, and ammonium acetate for one-pot synthesis of 2,5-dioxo-1,2,3,4,5,6,7,8-octahydroquinolines under solvent-free conditions at 120°C with $NiFe_2O_4@SiO_2$-$H_{14}[NaP_5W_{30}O_{110}]$ (NFS-PRS) as catalyst (0.02 g); the corresponding octahydroquinolines **9a**–**9g** were thus obtained in good to high yields (Scheme 3, Table 2).

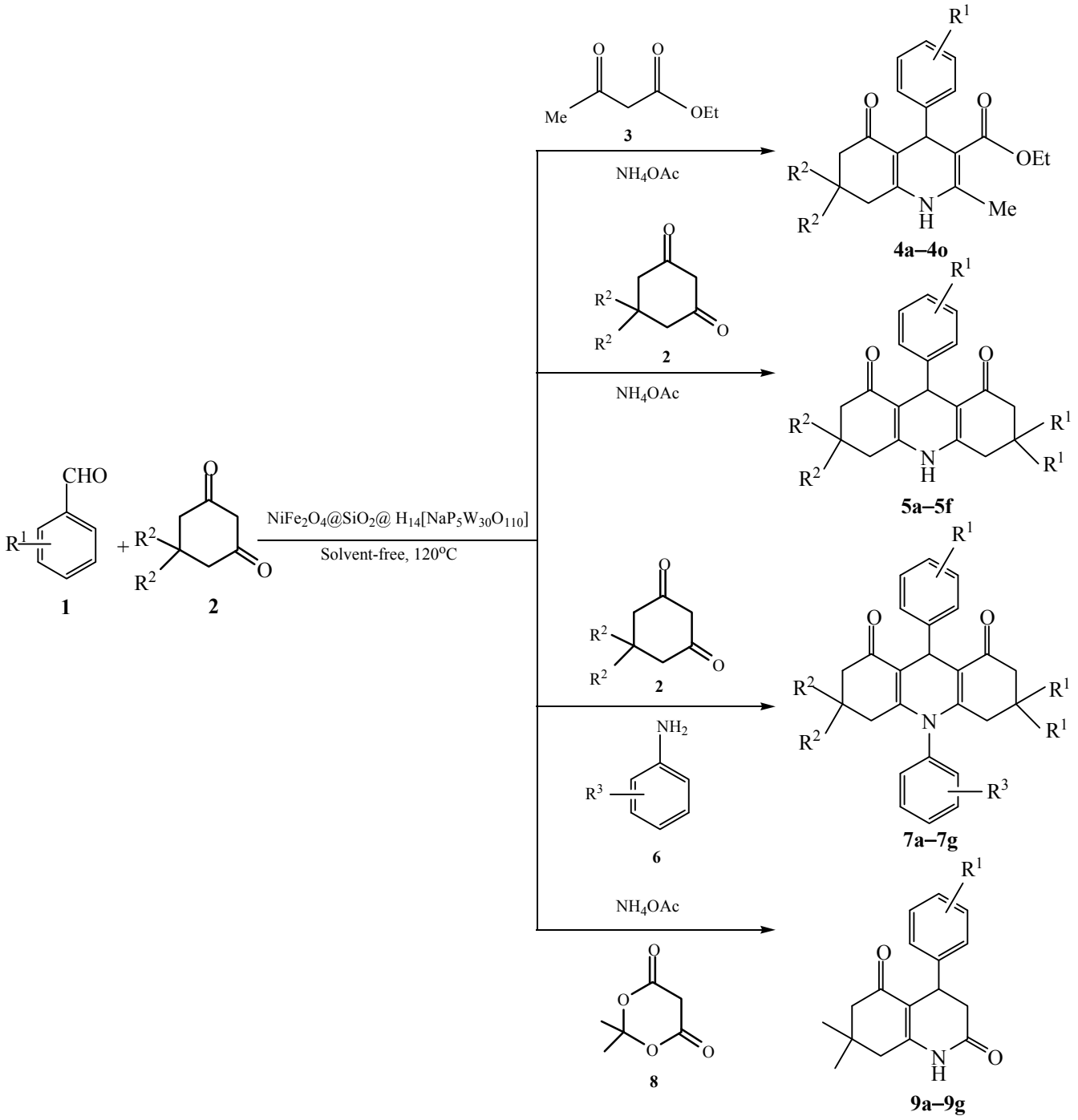
The main advantage of the proposed protocol is simple separation of the catalyst from the reaction mixture. In all cases, the reaction procedure was clean,

Scheme 2.



1 **2** **3** **4a**

Scheme 3.



For R^1, R^2, and R^3, see Table 2.

safe, simple, and consistent with the green chemistry principles. The reusability and recovery of NFS-PRS was investigated in the synthesis of **4a** and **5b**. After completion of the reaction, the catalyst was separated from the reaction mixture using an external magnet, dried at 100°C for 2 h, and reused in the same reaction. In four successive runs, the yields of **4a** and **5b** were 94, 93, 90, and 90% and 95, 92, 92, and 90%, respectively.

Table 2. One-pot synthesis of polyhydroquinolines, 1,8-dioxodecahydroacridines, and 2,5-dioxo-1,2,3,4,5,6,7,8-octahydroquinolines in the presence of $NiFe_2O_4@SiO_2$-$H_{14}[NaP_5W_{30}O_{110}]$

Compound no.	R^1	R^2	R^3	Reaction time, min	Yield,[a] %	mp, °C (lit. data)
4a	H	Me	–	10	94	200–202 (200–202) [19]
4b	4-O_2N	Me	–	30	90	242–244 (245–247) [16]
4c	2-Cl	Me	–	35	89	207–209 (210–211) [16]
4d	2,4-Cl_2	Me	–	40	90	240–242 (245–246) [17]
4e	2-MeO	Me	–	25	85	190–192 (193–195) [17]
4f	4-Cl	Me	–	30	92	240–242 (242–244) [17]
4g	4-Br	Me	–	20	90	249–251 (252–253) [32]
4h	2-Thienyl	Me	–	40	90	226–228 (224–226) [33]
4i	3-O_2N	Me	–	30	88	171–173 (175–176) [17]
4j	4-Me	Me	–	10	90	252–254 (257–259) [17]
4k	3-MeO	Me	–	20	87	201–203 (202–204) [20]
4l	H	H	–	10	82	240–242 (240–241) [20]
4m	4-O_2N	H	–	10	87	200–202 (196–198) [17]
4n	4-Cl	H	–	10	92	237–239 (236) [17]
4o	4-Me	H	–	5	89	241–243 (240–241) [20]
5a	H	Me	–	30	85	190–192 (190–192) [22]
5b	4-Br	Me	–	25	95	244–245 (241–243) [25]
5c	4-Cl	Me	–	20	93	298–300 (299–301) [25]
5d	4-O_2N	Me	–	60	92	284–286 (286–288) [25]
5e	H	H	–	10	82	280–282 (279–281) [20]
5f	4-Cl	H	–	15	94	264–266 (268–270) [20]
7a	H	Me	4-Me	35	92	256–258 (260–262) [27]
7b	H	Me	4-MeO	20	89	210–212 (215–216) [27]
7c	4-Me	Me	4-Me	25	75	288–290 (293–294) [23]
7d	4-MeO	Me	4-Me	15	81	281–283 (285–287) [23]
7e	H	Me	H	20	92	252–254 (254–255) [27]
7f	H	Me	4-MeO	20	89	212–214 (215–216) [27]
7g	3-O_2N	Me	4-Me	25	77	280–283 (285–287) [23]
9a	H	Me	–	20	80	213–215 (210–212) [30]
9b	4-Br	Me	–	10	96	190–192 (192–194) [30]
9c	2,4-Cl	Me	–	15	95	180–182 (184–186) [30]
9d	4-Cl	Me	–	5	95	186–188 (188–190) [31]
9e	3-O_2N	Me	–	10	96	189–191 (190–192) [30]
9f	4-MeO	Me	–	15	92	212–214 (213–215) [31]
9g	2,3-$(MeO)_2$	Me	–	25	84	236–238 (234–240) [30]

[a] Isolated yield.

In summary, we have proposed a novel procedure for the synthesis of 1,4-dihydropyridine derivatives in the presence of a heterogeneous magnetic nanocatalyst under solvent-free conditions. The proposed procedure is advantageous due to its environmental and economical efficiency, short reaction time, experimental simplicity, generality, easy work-up, and reusability of the catalyst.

EXPERIMENTAL

All reagents were purchased achieved from commercial sources. The melting points were measured by a Stuart BI Barnstead Electrothermal IA9200 apparatus and are uncorrected. The IR spectra were recorded in KBr on a Shimadzu 435-U-04 FT-IR spectrometer. The ^{1}H NMR spectra were obtained using a Bruker 300 MHz spectrometer in DMSO-d_6 or $CDCl_3$ with TMS as internal standard. The particle size and morphology of the nanocatalyst were studied using Philips CM-200 and Titan Krios transmission electron microscopes and Philips XL 30 and S-4160 scanning electron microscopes with gold coating; the particle size distribution was determined using a CORDOUAN Vasco3A laser particle size analyzer.

Silica-coated magnetic $NiFe_2O_4$ nanoparticles-supported $H_{14}[NaP_5W_{30}O_{110}]$ (NFS-PRS]. Magnetic $NiFe_2O_4$ nanoparticles were prepared by reaction between $NiCl_2$ and $FeCl_3$ in presence of H_2O and NH_4OH under alkaline conditions and were coated with silica by treatment with tetraethyl orthosilicate. In the next step, the obtained $NiFe_2O_4$@SiO_2 (NFS, 1 g) was dispersed in water (50 mL) and sonicated at room temperature for 15 min. A solution of $H_{14}[NaP_5W_{30}O_{110}]$ (0.75 g) in water (5 mL) was prepared and added dropwise to the suspension of NFS, and the mixture was stirred for 12 h at room temperature. The solvent was evaporated under reduced pressure, and the supported catalyst was collected by an external magnet, dried in a vacuum overnight, and calcined at 250°C for 2 h.

General procedure for the synthesis of polyhydroquinolines 4a–4o. A mixture of aldehyde **1** (1 mmol), dimedone or cyclohexane-1,3-dione (**2**, 1 mmol), ethyl acetoacetate (**3**, 1.2 mmol), ammonium acetate (5 mmol) and NFS-PRS (0.02 g) was heated on an oil bath at 120°C for a time indicated in Table 2). The progress of the reaction was monitored by TLC (*n*-hexane–ethyl acetate, 2 : 1). When the reaction was complete, 5 ml of hot ethanol (96%) was added, and the mixture was stirred for 2 min. The catalyst was separated from the reaction mixture by means of an external magnet. The crude product was poured into crushed ice, and the solid product was filtered off and recrystallized from ethanol (5 mL).

General procedure for the synthesis of 1,8-dioxodecahydroacridines 5a–5f and 7a–7g. A mixture of aldehyde **1** (1 mmol), dimedone or cyclohexane-1,3-dione (**2**, 2 mmol), ammonium acetate (5 mmol) or aromatic aniline **6** (1.2 mmol), and NFS-PRS (0.02 g) was heated on an oil bath at 120°C for a time indicated in Table 2. The subsequent procedure was the same as in the synthesis of **4a–4o**.

General procedure for the synthesis of 2,5-dioxo-1,2,3,4,5,6,7,8-octahydroquinolines 9a–9g. A mixture of aldehyde **1** (1 mmol), dimedone (2 mmol), Meldrum's acid (1 mmol), ammonium acetate (5 mmol), and NFS-PRS (0.02 g) was heated on an oil bath at 120°C for a time indicated in Table 2. The mixture was then treated as described above for the synthesis of **4a–4o**.

The products were identified by comparing their melting points with those reported in the literature (Table 2), as well as by spectral data. Spectroscopic data for some representative compounds are given below.

Ethyl 2,7,7-trimethyl-5-oxo-4-phenyl-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (4a). IR spectrum, ν, cm^{-1}: 3235, 3216, 3087, 1699, 1604, 1063, 697. ^{1}H NMR spectrum (300 MHz, DMSO-d_6), δ, ppm: 0.94 s (3H), 1.09 s (3H), 1.14 t (3H, J = 7.3 Hz), 2.13–2.34 m (4H), 2.37 s (3H), 4.05 q (2H, J = 7.3 Hz), 5.02 s (1H), 5.74 s (1H), 7.03–7.34 m (5H). ^{13}C NMR spectrum (75 MHz, DMSO-d_6), δ_C, ppm: 14.5, 19.4, 21.7, 27.9, 36.8, 37.7, 59.9, 106.3, 113.8, 126.8, 127.10, 128.4, 143.7, 147.4, 149.5, 167.7, 194.11.

Ethyl 2,7,7-trimethyl-4-(3-nitrophenyl)-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (4i). IR spectrum, ν, cm^{-1}: 3307, 2965, 1693, 1615, 1169, 763. ^{1}H NMR spectrum (300 MHz, DMSO-d_6), δ, ppm: 0.96 s (3H), 1.04 s (3H), 1.22 t (3H, J = 7.3 Hz), 2.10–2.34 m (4H), 2.38 s (3H), 4.01 q (2H, J = 7.3 Hz), 4.96 s (1H), 6.32 s (1H), 6.74–7.38 m (4H). ^{13}C NMR spectrum (75 MHz, DMSO-d_6), δ_C, ppm: 14.21, 19.35, 21.3, 27.6, 33.4, 33.93, 59.8, 105.7, 112.7, 121.5, 122.11, 128.9, 134.11, 144.9, 148.6, 149.8, 151.3, 166.12, 196.3.

3,3,6,6-Tetramethyl-9-(4-nitrophenyl)-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione (5d). IR spec-

trum, ν, cm^{-1}: 3390, 3075, 2959, 1650, 1519, 1484, 1347, 1221, 1170. ^1H NMR spectrum (300 MHz, DMSO-d_6), δ, ppm 0.87 s (6H), 1.02 s (6H), 2.04 d (2H, J = 16.3 Hz), 2.15 d (2H, J = 16.3 Hz), 2.26 d (2H, J = 17.0 Hz), 2.34 d (2H, J = 17.0 Hz), 5.05 s (1H), 7.44 d (2H, J = 8.5 Hz), 7.98 d (2H, J = 6.9 Hz), 8.49 s (1H). ^{13}C NMR spectrum (75 MHz, DMSO-d_6), δ_C, ppm: 27.7, 29.12, 32.12, 34.11, 51.3, 112.9, 123.8, 129.7, 146.4, 149.11, 154.12, 195.9.

3,3,6,6-Tetramethyl-9,10-diphenyl-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione (7e). IR spectrum, ν, cm^{-1}: 3082, 3055, 2961, 2930, 2893, 2861, 1722, 1640, 1582, 1490, 1362, 1273, 1220, 1132, 1081, 1015, 917, 899, 842, 798, 738, 699, 680, 609, 587, 579, 532, 470. ^1H NMR spectrum (300 MHz, $CDCl_3$), δ, ppm: 7.56 d (3H, J = 6.4 Hz), 7.44 d (2H, J = 7.6 Hz), 7.25 d (4H, J = 7.2 Hz), 7.10 d.d (1H, J = 7.2, 6.8 Hz), 5.29 s (1H), 2.24–2.11 m (4H), 2.07 d (2H, J = 17.2 Hz), 1.82 d (2H, J = 17.6 Hz), 0.94 s (6H), 0.79 s (6H). ^{13}C NMR spectrum (75 MHz, $CDCl_3$), δ_C, ppm: 195.77, 162.21, 159.80, 149.68, 141.97, 141.94, 138.74, 129.91, 129.33, 129.16, 129.08, 115.33, 114.68, 114.47, 114.30, 49.99, 41.63, 32.21, 31.98, 29.53, 26.51.

4-(2,3-Dimethoxyphenyl)-7,7-dimethyl-4,6,7,8-tetrahydroquinoline-2,5(1*H*,3*H*)-dione (9g). IR spectrum, ν, cm^{-1}: 3220, 2939, 1616, 1485, 1385, 1290, 1222, 1069, 793. ^1H NMR spectrum (300 MHz, DMSO-d_6), δ, ppm: 10.11 s (1H), 6.92 m (2H), 6.50 d.d (1H), 4.46 d (1H), 3.82 s (3H), 3.79 s (3H), 2.94 d.d (1H), 2.47 d (2H), 2.23 d (1H), 2.19 q (2H), 1.07 s (3H), 1.04 s (3H).

ACKNOWLEDGMENTS

The authors thank Hakim Sabzevari University for financial support to this research.

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