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ORIGINAL PAPER

Sulfated polyborate: mild, efficient and eco-friendly catalyst for the synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones

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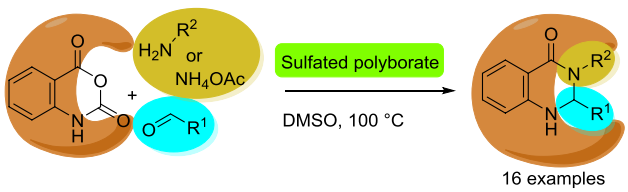
Abstract An efficient, inexpensive and recyclable sulfated polyborate catalyst was applied in a three-component, one-pot cyclocondensation of isatoic anhydride, aldehydes and ammonium acetate/amines to afford the corresponding 2,3-dihydroquinazolin-4(1*H*)-ones. The key advantages of the present method are high yields, short reaction time, easy workup, recyclability of catalyst and ability to tolerate a variety of functional groups which gives economical as well as ecological rewards.

Graphical Abstract



Keywords Sulfated polyborate · 2,3-Dihydroquinazolin-4(1*H*)-ones · Isatoic anhydride · Recyclable catalyst

Introduction

It has been more than a century since the initial studies on synthesis of quinazoline derivatives were published [1]. Quinazoline and its derivatives are gaining importance in organic and medicinal chemistry. Quinazolines are an important class of fused heterocycles with a wide range of pharmacological and biological activities such as anticancer [2], antitumor [3], antitremor [4], antiatherosclerotic [5], antimicrobial [6], antitubercular [7], antifungal [8], antimalarial [9], antiinflammatory and analgesic [10], choleretic and antifibrillatory [11], angiotensin-II receptor antagonists [12], monoamine oxidase inhibitors [13], potent and selective ALK5 inhibitors [14]. These compounds also have antihypertensive [15], sedative and tranquilizer properties [16]. In particular, there are several drugs containing 2,3-dihydroquinazolin-4(1*H*)-one scaffold commercially available such as quinethazone and fenquizone; diuretics [17, 18]; and evodiamine, a novel anticancer agent [19] (Fig. 1).

In the synthesis of quinazolinone compounds, widely used reagents are 2-aminobenzoic acid or its derivatives, 2-aminobenzamide, 2-aminobenzonitrile, isatoic anhydride, 2-carbomethoxyphenylisocyanate, *N*-arylnitrilium salts and 3,1-benzoxazinones [20–22]. Other methods involve the reaction of anthranilic acid and the substituted imidate, cycloaddition of anthranilic acid iminoketene to methylbutyrolactam via sulfonamide anhydride. However, use of isatoic anhydride is common due to its rapid reaction with low side product formation [7, 21, 23].

There are many reports on the synthesis of 2,3-dihydroquinazolin-4(1*H*)-one using various catalysts such as sulfuric acid–silica gel [24], potassium alum [25], Montmorillonite K-10 [26], *p*-TSA [27], 1-*n*-butyl-3-methylimidazolium tetrafluoroborate [28], iodine [29], gallium trifluoromethanesulfonate [30], aluminum tris (dihydrogen phosphate) [31], $SrCl_2{\cdot}6H_2O$ [32], 2,2,2-trifluoro ethanol [33], sodium lauryl sulfate/tartaric acid [34], L-proline [35], copper(II) benzene-sulfonate hexahydrate [36],

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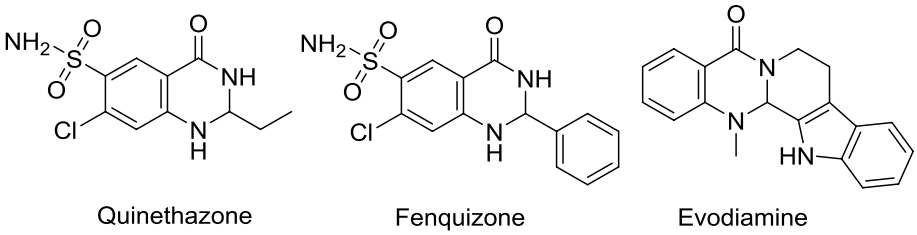


Fig. 1 Pharmacologically active drug molecules

$C_3H_9AlO_6S_3{\cdot}4H_2O$ [37], thiamine chloride hydrochloride [38], tetrabutylammonium bromide [39], indium sesquioxide [40], 2-pyrrolidon-1-ium hydrogen sulfate [41], H_3PO_4/Al_2O_3 [42], PFPAT [43], Fe_3O_4/SBA-15 [44], hydroxyapatite nanoparticles [45], lactic acid [46], etc. However, many of the methods suffer from various disadvantages such as unsatisfactory yields, longer reaction time, extractive product isolation with toxic organic solvents, use of expensive, metal-based, toxic/corrosive catalysts, which limits their use due to environmental issues. Thus, the development of a safe, environmentally benign, mild, efficient and high yielding rapid reaction procedure using cost effective and recyclable catalyst would be valuable.

Pursuit of green, convenient and practical catalytic methods for the current interest of organic synthesis and commercial process; recently we have introduced sulfated polyborate catalyst and demonstrated its efficiency for catalyzing various organic transformations [47, 55–59]. Its mild acidity, easy preparation, eco-friendliness and reusability have encouraged us to investigate its potential to catalyze many other useful reactions. Therefore, in continuation of our previous study, in this paper, we explored the application of sulfated polyborate as a highly effective catalyst for the preparation of 2,3-dihydroquinazolin-4(1*H*)-ones (Scheme 1).

Experimental

Melting points of all the compounds were recorded by Analab ThermoCal melting point apparatus in the open capillary tube and are uncorrected. The FTIR spectra (KBr) were recorded on Shimadzu FTIRAffinity-1 Fourier Transform Infrared spectrophotometer. ^{1}H NMR spectra were recorded on MR400 Agilent Technology NMR spectrometer using tetramethylsilane (TMS) as an internal standard and DMSO-d_6/$CDCl_3$ as solvent. Chemicals and solvents used were of LR grade and purchased from SD fine, Avra Synthesis and Spectrochem and used without purification. The purity determination of the starting materials and reaction monitoring was accomplished by thin-layer chromatography (TLC) on Merck silica gel G F_{254} plates.

Preparation of sulfated polyborate

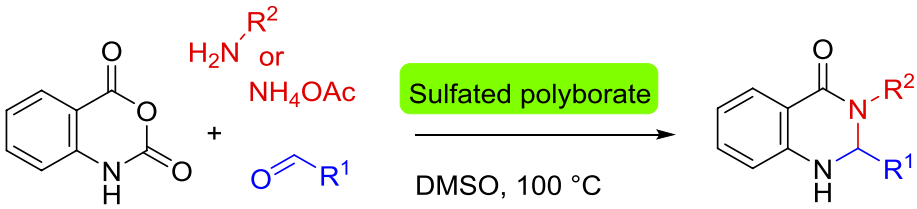
The sulfated polyborate catalyst was prepared following procedure reported in the literature [47].

General procedure for the synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones

To a DMSO solution of isatoic anhydride (2 mmol), aldehydes (2 mmol) and ammonium acetate (2.4 mmol))/amines (2 mmol) was added sulfated polyborate (10 wt%). The mixture was heated at 100 °C in an oil bath. The reaction was monitored by thin layer chromatography. After completion of the reaction, the mixture was cooled to room temperature and quenched by water. Solid was filtered at vacuum pump, washed with water (3 × 5 mL), dried under vacuum and recrystallized from ethanol to afford the pure products. The products obtained were known compounds and were identified by melting point, FTIR and ^{1}H NMR spectroscopy. The spectral data were compared with those in the literature.

Representative spectral data

2-phenyl-2,3-dihydroquinazolin-4(1H)-one **(4a)** *(Table 3, entry 1)* ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (d, J = 7.6 Hz, 1H), 7.57 (d, J = 3.2 Hz, 2H), 7.46–7.38 (m,



Scheme 1 Sulfated polyborate catalyzed synthesis of 2,3-dihydroquinazoline-4(1*H*)-ones

3H), 7.32 (t, J = 7.6 Hz, 1H), 6.88 (t, J = 7.6 Hz, 1H), 6.65 (d, J = 8.1 Hz, 1H), 5.88 (s, 1H), 5.85 (s, 1H), 4.38 (s, 1H).

2-(4-methoxyphenyl)-2,3-dihydroquinazolin-4(1H)-one **(4b)** *(Table 3, entry 2)* ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.16 (s, 1H), 7.58 (d, J = 7.4 Hz, 1H), 7.39 (d, J = 8.0 Hz, 2H), 7.21 (t, J = 7.4 Hz, 1H), 6.99 (s, 1H), 6.92 (d, J = 7.8 Hz, 2H), 6.71 (d, J = 8.0 Hz, 1H), 6.64 (t, J = 7.4 Hz, 1H), 5.68 (s, 1H), 3.72 (s, 3H).

2-(4-bromophenyl)-2,3-dihydroquinazolin-4(1H)-one **(4c)** *(Table 3, entry 3)* ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.31 (s, 1H), 7.57 (d, J = 7.2 Hz, 3H), 7.41 (d, J = 8.1 Hz, 2H), 7.22 (t, J = 7.6 Hz, 1H), 7.12 (s, 1H), 6.71 (d, J = 8.0 Hz, 1H), 6.65 (t, J = 7.3 Hz, 1H), 5.72 (s, 1H).

2-(4-chlorophenyl)-2,3-dihydroquinazolin-4(1H)-one **(4d)** *(Table 3, entry 4)* ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.31 (s, 1H), 7.58 (d, J = 7.6 Hz, 1H), 7.48 (d, J = 8.3 Hz, 2H), 7.43 (d, J = 8.3 Hz, 2H), 7.22 (t, J = 7.6 Hz, 1H), 7.12 (s, 1H), 6.71 (d, J = 7.8 Hz, 1H), 6.65 (t, J = 7.4 Hz, 1H), 5.74 (s, 1H).

2-(4-fluorophenyl)-2,3-dihydroquinazolin-4(1H)-one **(4e)** *(Table 3, entry 5)* ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.23 (s, 1H), 7.56 (d, J = 7.4 Hz, 1H), 7.49 (dd, J = 8.2, 5.7 Hz, 2H), 7.22–7.15 (m, 3H), 7.05 (s, 1H), 6.70 (d, J = 8.1 Hz, 1H), 6.63 (t, J = 7.4 Hz, 1H), 5.72 (s, 1H).

2-(p-tolyl)-2,3-dihydroquinazolin-4(1H)-one **(4f)** *(Table 3, entry 6)* ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.20 (s, 1H), 7.57 (d, J = 7.7 Hz, 1H), 7.34 (d, J = 7.6 Hz, 2H), 7.24–7.12 (m, 3H), 7.03 (s, 1H), 6.71 (d, J = 8.1 Hz, 1H), 6.64 (t, J = 7.5 Hz, 1H), 5.68 (s, 1H), 2.27 (s, 3H).

2-(4-(dimethylamino)phenyl)-2,3-dihydroquinazolin-4(1H)-one **(4g)** *(Table 3, entry 7)* ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.05 (s, 1H), 7.57 (d, J = 7.7 Hz, 1H), 7.27 (d, J = 8.4 Hz, 2H), 7.20 (t, J = 7.6 Hz, 1H), 6.89 (s, 1H), 6.69 (d, J = 6.5 Hz, 3H), 6.63 (t, J = 7.4 Hz, 1H), 5.60 (s, 1H), 2.85 (s, 6H).

2-(2-chlorophenyl)-2,3-dihydroquinazolin-4(1H)-one **(4h)** *(Table 3, entry 8)* ^{1}H NMR (400 MHz, DMSO-d_6) δ 8.19 (s, 1H), 7.63 (d, J = 6.1 Hz, 2H), 7.53–7.44 (m, 1H), 7.41–7.33 (m, 2H), 7.23 (t, J = 7.6 Hz, 1H), 6.99 (s, 1H), 6.73 (d, J = 8.2 Hz, 1H), 6.69 (t, J = 7.5 Hz, 1H), 6.11 (s, 1H).

2-(2-hydroxyphenyl)-2,3-dihydroquinazolin-4(1H)-one **(4i)** *(Table 3, entry 9)* ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.82 (s, 1H), 7.91 (s, 1H), 7.59 (d, J = 7.8 Hz, 1H), 7.31 (d, J = 7.6 Hz, 1H), 7.19 (t, J = 7.6 Hz, 1H), 7.12 (t, J = 7.7 Hz, 1H), 6.83 (d, J = 8.1 Hz, 1H), 6.79–6.72 (m, 3H), 6.63 (t, J = 7.5 Hz, 1H), 5.97 (s, 1H).

2-cyclohexyl-2,3-dihydroquinazolin-4(1H)-one **(4j)** *(Table 3, entry 10)* ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.85 (s, 1H), 7.52 (d, J = 7.7 Hz, 1H), 7.17 (t, J = 7.6 Hz, 1H), 6.71 (d, J = 8.1 Hz, 1H), 6.58 (t, J = 7.3 Hz, 1H), 6.53 (s, 1H), 4.42 (s, 1H), 1.69–1.52 (m, 6H), 1.10–1.09 (m, 5H).

1′H-spiro[cyclohexane-1,2′-quinazolin]-4′(3′H)-one **(4k)** *(Table 3, entry 11)* ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.87 (s, 1H), 7.52 (d, *J* = 7.4 Hz, 1H), 7.17 (t, *J* = 7.3 Hz, 1H), 6.77 (d, *J* = 8.0 Hz, 1H), 6.63–6.50 (m, 2H), 1. 75–1.65 (m, 2H), 1.63–1.42 m, 6H), 1.43–1.34 (m, 1H), 1.28–1.17 (m, 1H).

2,3-diphenyl-2,3-dihydroquinazolin-4(1H)-one **(4l)** *(Table 3, entry 12)* ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.69 (d, J = 7.8 Hz, 1H), 7.63 (s, 1H), 7.35 (d, J = 7.9 Hz, 2H), 7.34–7.18 (m, 8H), 7.16 (t, J = 6.7 Hz, 1H), 6.73 (d, J = 8.1 Hz, 1H), 6.69 (t, J = 7.5 Hz, 1H), 6.26 (s, 1H).

3-(4-bromophenyl)-2-phenyl-2,3-dihydroquinazolin-4(1H)-one **(4m)** *(Table 3, entry 13)* ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.67 (d, J = 7.6 Hz, 1H), 7.61 (s, 1H), 7.46 (d, J = 8.7 Hz, 2H), 7.32 (d, J = 7.0 Hz, 2H), 7.29–7.20 (m, 4H), 7.17 (d, J = 8.7 Hz, 2H), 6.75–6.64 (m, 2H), 6.26 (s, 1H).

3-(4-chlorophenyl)-2-phenyl-2,3-dihydroquinazolin-4(1H)-one **(4n)** *(Table 3, entry 14)* ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.85–6.89 (m, 12H), 6.66 (d, J = 14.5 Hz, 2H), 6.24 (s, 1H).

2-phenyl-3-(p-tolyl)-2,3-dihydroquinazolin-4(1H)-one **(4o)** *(Table 3, entry 15)* ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.66 (d, J = 7.6 Hz, 1H), 7.53 (s, 1H), 7.31 (d, J = 7.2 Hz,

2H), 7.28–7.15 (m, 4H), 7.12–7.04 (m, 4H), 6.73–6.59 (m, 2H), 6.18 (s, 1H), 2.20 (s, 3H).

3-benzyl-2-phenyl-2,3-dihydroquinazolin-4(1H)-one **(4p)** *(Table 3, entry 16)* ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.66 (d, J = 7.5 Hz, 1H), 7.37–7.10 (m, 12H), 6.68–6.56 (m, 2H), 5.70 (d, J = 2.3 Hz, 1H), 5.28 (d, J = 15.4 Hz, 1H), 3.78 (d, J = 15.4 Hz, 1H).

3-phenethyl-2-phenyl-2,3-dihydroquinazolin-4(1H)-one **(4q)** *(Table 3, entry 17)* ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.61 (d, J = 7.6 Hz, 1H), 7.32–7.28 (m, 6H), 7.24–7.20 (m, 2H), 7.17–7.11 (m, 4H), 6.63–6.57 (m, 2H), 5.80 (s, 1H), 4.00–3.89 (m, 1H), 2.95–2.81 (m, 2H), 2.71–2.61 (m, 1H).

Results and discussion

For initial screening, the study was structured to investigate the suitability of sulfated polyborate as a catalyst at different reaction conditions. For the preliminary experiment, a mixture of benzaldehyde—a representative substrate, isatoic anhydride and ammonium acetate was used. The results are summarized in Table 1.

The effect of the catalyst loading on time and yields of the reaction was assessed. In absence of a catalyst, the reaction proceeded at 100 °C but took longer reaction time with a lower yield (Table 1, entry 1). An increase in the catalyst loading increased the product yield with significate reduction in the reaction time (Table 1, entries 2–5). The catalyst loading beyond 10 wt % was not advantageous (Table 1, entries 5 and 6). Hence, a 10 wt% catalyst loading was chosen for further study.

The effect of various solvents on time and yield of the reaction was ascertained (Table 1, entries 5 and 7–12). None of the solvents have shown advantage over DMSO. Hence, DMSO was regarded as a best solvent to carry out the further reactions.

Temperature played an important role in the synthesis of 2,3-dihydroquinazolin-4(1*H*)-one. The temperature effect was examined at ambient, 50, 80 and 100 °C using sulfated polyborate (Table 1, entries 5 and 13–15). The reaction proceeded at room temperature but took longer reaction time with a lower yield (Table 1, entry 13). An increase in the temperature to 100 °C resulted in significantly increased product yield in shorter reaction time (Table 1, entries 5, 14 and 15). Therefore, 100 °C was chosen as optimum temperature for the reaction.

Table 1 Results of optimization studies

Entry	Catalyst (wt%)	Solvent	Temperature (°C)	Time (min)	Yield[a] (%)
1.	0	DMSO	100 °C	180	69
2.	1	DMSO	100 °C	30	80
3.	2.5	DMSO	100 °C	15	85
4.	5	DMSO	100 °C	15	91
5.	10	DMSO	100 °C	15	97
6.	15	DMSO	100 °C	15	97
7.	10	Solvent free	100 °C	30	88
8.	10	EtOH	Reflux	30	82
9.	10	ACN	Reflux	30	90
10.	10	THF	Reflux	30	88
11.	10	Water	Reflux	30	Traces
12.	10	Toluene	Reflux	30	91
13.	10	DMSO	Rt	30	79
14.	10	DMSO	50 °C	30	86
15.	10	DMSO	80 °C	30	95

[a] Isolated yield

Various catalysts are reported for the synthesis of 2,3-dihydroquinazolin-4(1*H*)-one. Herein, in comparison with other acid catalysts, sulfated polyborate catalyst showed an advantage with respect to reaction conditions, workup procedure, time and yields (Table 2, entries 1–10).

To study the generality and scope, optimized reaction conditions were applied to various aromatic/aliphatic aldehydes and amines. All the substrate variants reacted well and afforded higher yields of 2,3-dihydroquinazolin-4(1*H*)-ones in shorter reaction time (Tables 3, 4). Various electron donating or electron withdrawing substituents at the *ortho* and *para* position of aromatic aldehydes have been examined. The nature of substitutions on aromatic aldehydes has no significant effect on the reaction time and yields (Table 3, entries 2–9). The protocol is also extended to an aliphatic aldehyde/ketone variants cyclohexane carboxaldehyde and cyclohexanone to get corresponding 2,3-dihydroquinazolin-4(1*H*)-ones in shorter reaction time in good yield (Table 3, entry 10 and 11).

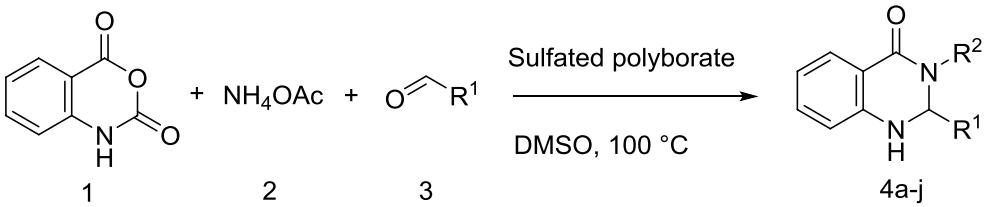
On the other side, the applicability of this protocol on aromatic and aliphatic amines was also examined using substituted aniline, benzylamine and phenylethylamine variants. All the amine variants were reacted well and afforded higher yields of the corresponding 2,3-dihydroquinazolin-4(1*H*)-ones in shorter reaction time (Table 4, entries 12–17).

Table 2 Efficiency of sulfated polyborate in comparison with literature-reported catalyst for the 2-phenyl-2,3-dihydroquinazolin-4(1*H*)-one

Entry	Catalyst	Conditions	Time (min)	Yield[a] (%)	References
1.	Sulfated polyborate	DMSO/100 °C	15	97	This work
2.	Copper(II) benzene-sulfonate hexahydrate	EtOH/water/reflux	30	93	[36]
3.	Sulfuric acid–silica gel	EtOH/reflux	180	92	[24]
4.	1-*n*-Butyl-3-methyl imidazolium tetrafluoro borate	Solvent free/70 °C	90	92	[28]
5.	$C_3H_9AlO_6S_3{\cdot}4H_2O$	EtOH/water/reflux	60	91	[37]
6.	Lactic acid	Solvent free/70 °C	30	90	[46]
7.	2-Pyrrolidon-1-ium hydrogen sulfate	Solvent free/80 °C	11	89	[41]
8.	Gallium trifluoro methanesulfonate	EtOH/70 °C	55	87	[30]
9.	Montmorillonite K-10	EtOH/reflux	240	85	[26]
10.	*p*-TSA	Water/reflux	60	84	[27]

[a] Isolated yield

Table 3 Synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones using a variety of aldehydes



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Entry	Aldehydes (R^1)	Products	Time (min)	Yield[a] (%)	Melting point (°C)	
					Obtained	Literature
1.	C_6H_5	**4a**	15	97	215–216	216–218 [48]
2.	4-CH_3O-C_6H_4	**4b**	15	95	190–191	189–191 [49]
3.	4-Br-C_6H_4	**4c**	15	93	195–196	196–198 [49]
4.	4-Cl-C_6H_4	**4d**	15	94	201–202	202–204 [49]
5.	4-F-C_6H_4	**4e**	15	94	195–196	194–196 [49]
6.	4-CH_3-C_6H_4	**4f**	15	93	227–228	230–232 [49]
7.	4-$(CH_3)_2N$-C_6H_4	**4g**	15	89	222–223	220–222 [50]
8.	2-Cl-C_6H_4	**4h**	15	90	206–208	206–208 [51]
9.	2-HO-C_6H_4	**4i**	20	88	209–210	210–211 [50]
10.	*c*-C_6H_{11}	**4j**	15	92	174–175	–
11.	*c*-C_5H_{10}	**4k**	20	93	227–228	228–230 [52]

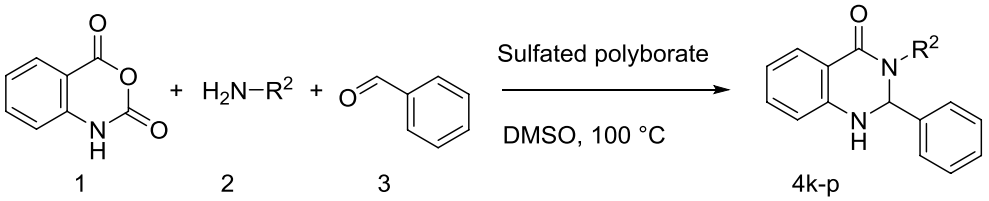
[a] Isolated yields

The reusability of the catalyst for the model reaction of isatoic anhydride, benzaldehyde and ammonium acetate in DMSO at 100 °C was evaluated. After completion of each cycle, the reaction was quenched with water and the product was filtered off. The water was evaporated from the filtrate in vacuum rotary evaporator to recover the catalyst quantitatively in DMSO. The recovered catalyst in DMSO was recycled for four times with no significant loss in the catalytic activity (Fig. 2).

Conclusion

In conclusion, the present procedure is a rapid, efficient and eco-friendly protocol for the synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones using various aldehydes, ammonium acetate/amines and isatoic anhydride under optimized conditions. Mild reaction conditions, easy of workup procedure, shorter reaction time, high yields and recyclability of the catalyst are the key features of this

Table 4 Synthesis of 2,3-dihydroquinazolin-4(1*H*)-ones using various amines



Entry	Amines (R^2)	Products	Time (min)	Yield[a] (%)	Melting point (°C)	
					Obtained	Literature
1.	C_6H_5	**4k**	20	94	203–204	203–204 [40]
2.	4-Br-C_6H_4	**4l**	20	90	125–127	228–229 [45]
3.	4-Cl-C_6H_4	**4m**	20	91	214–215	216–217 [45]
4.	4-CH_3-C_6H_4	**4n**	20	93	197–198	198–199 [45]
5.	$C_6H_5CH_2$	**4o**	15	90	128–129	128–130 [53]
6.	$C_6H_5CH_2CH_2$	**4p**	15	94	146–147	145–147 [54]

[a] Isolated yields

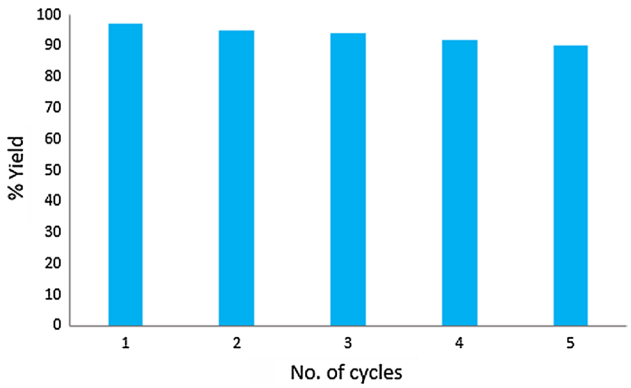


Fig. 2 Reusability of the catalyst

procedure. Moreover, this protocol has the ability to tolerate a wide variety of substituents along with enhanced product purity which promises economical as well as ecological rewards.

Supplementary information

Full experimental details and ^{1}H NMR spectra can be found in Electronic Supplementary Information section of this article's webpage.

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