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Investigation of the uncommon basic properties of $[Ln(W_5O_{18})_2]^{9-}$ (Ln = La, Ce, Nd, Gd, Tb) by changing central lanthanoids in the syntheses of pyrazolopyranopyrimidines

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ABSTRACT

Five Lindqvist-type lanthanopolyoxometalates ($[Ln(W_5O_{18})_2]^{9-}$, Ln = La, Ce, Nd, Gd, Tb) complexes were prepared and fully characterized. Their basic catalytic activity was manipulated in the successful and high yielding synthesis of a series of pyrazolopyranopyrimidines *via* a four-component reaction, involving ethyl acetoacetate, hydrazine hydrate, benzaldehydes, and barbituric acid under ultrasonic irradiation. The catalytic activity of these catalysts with different Ln were compared under the same optimized reaction conditions in term of yields of obtained products, in shorter reaction time, indicating the superiority of the of the catalytic activity of ($[Tb(W_5O_{18})_2]^{9-}$- in the aforementioned Multicomponent reaction (MCR).

1. Introduction

The targeted use of green and efficient catalysts for various organic reactions has been attracting a great deal of attention. Polyoxometalates (POMs), a well-known class of anionic metal–oxygen nanoclusters, are valuable inorganic materials for this goal [1]. POM compounds have several advantages as catalysts, which make them economically and environmentally attractive. Unrivaled and outstanding catalytic behaviors of POMs are mainly based on their special properties such as size, electron- and proton-transfer/storage abilities, high thermal stability, inherent resistance to oxidative decomposition and tunable redox and acid-base properties because of their multiple active sites, including protons, metal ions, and oxygen atoms [2–5]. Because of their ability to behave as inorganic ligands, POMs can combine with highly oxyphilic lanthanoid (Ln) ions to form a number of significant compounds, which create a new subgroup of POMs, namely lanthanopolyoxometalates (LnPOMs) [6]. As it is well-known, lanthanoid species have been used as catalysts to affect a number of organic transformations. Therefore, LnPOMs can merge the merits of both components, resulting in, this combination is expected to lead to the desirable catalytic activity. It has been shown that Lindqvist-type of LnPOMs ($[Ln(W_5O_{18})_2]^{9-}$ (abbreviated as LnW_{10}) have interesting catalytic activities towards the selective oxidation of primary alcohols to aldehydes, epoxidation of alkenes and the desulfurization of benzothiophene derivatives [7–9]. The structure of the LnW_{10} polyanion family consists of two monolacunary Lindqvist fragments $[W_5O_{18}]^{6-}$ encapsulating a central lanthanoid ion which exhibits square-antiprismatic coordination (Fig. 1).

These highly charged anions have electron-rich oxygen atoms on their surfaces. The highly negative charge on oxygen atoms makes these species relatively basic. In fact, the oxo ligands at the vacant sites are basic enough to attack the electrophiles. Thus, LnW_{10} has rarely been used as basic catalysts in which, their basicity can be adjusted. However, in spite of its basic properties, the applications of LnPOM anions as a basic catalyst in organic transformation have been largely overlooked.

Nowadays, molecular architecting becomes a favorable task of chemists to develop the construction of organic compounds having complex structures starting from simple starting materials,

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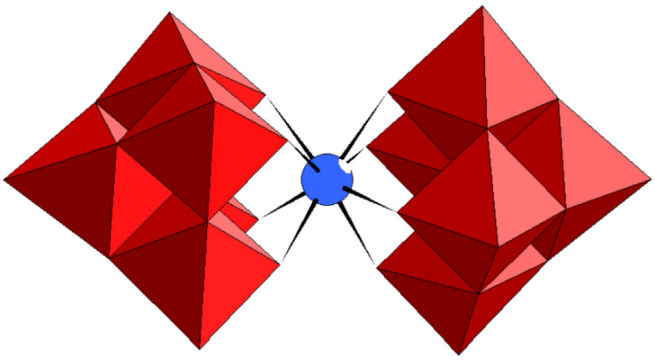


Fig. 1. Representation of $[Ln(W_5O_{18})_2]^{9-}$. Red octahedra: WO_6, blue ball: lanthanoid ion.

considering the eco-friendly protocols to respect the concept of green chemistry as well as economic concerns for the reaction being feasible, if they possibly can be applicable at large scale production. Nowadays, multicomponent reactions (MCRs) meet these requirements for the facile construction of new and complex molecules, in one pot fashion, under green conditions [10,11].

Chromene based molecules are well-renowned for their broad biological properties and are present in several bioactive pharmaceuticals and photochromic compounds. Furthermore, pyrimidine-fused heterocyclic derivatives are found in diverse bioactive skeletons such as, vitamin B1 (thiamine), and alloxan. Some bioactive pyrimidine and chromene-fused heterocyclic structures are displayed in Fig. 2 [12–15].

The pyranopyrazole scaffold is an attractive organic moiety in medicinal chemistry and provides a wide range of biological activity. Pyranopyrazole including organic structures may act as hypotensive [16], antitumor [17], hypoglycemic [18], and antidepressant agents [19]. Indeed, since the pyranopyrimidine scaffold, belongs to the pyrimidine family, and this family show a broad of biological activity, they also exhibit potency such being hepatoprotective [20], antitumor [21], antitubercular [22], and antimicrobial [23].

We are interested in heterocyclic chemistry. In the last two decades, our laboratory has been engaged in the synthesis of a wide range heterocyclic systems catalyzed by different heteropoly acids as strong and effective Brønsted acid with too many hydrogens such as Keggin, Wells-Dawson, and Preyssler [24]. We also reviewed the catalytic potency of HPAs and their polyoxometalate, from different points of view. We also recently reviewed the applications of inorganic-organic hybrid architectures based on polyoxometalates in catalyzed and photocatalyzed chemical transformations. Preservation of environment, and comply of the green chemistry principles has always been our research group concerns [25].

In this research we tried to expand our research interest, by manipulating of the catalytic activity of Lindqvist-type lanthanopolyoxometalates ($[Ln(W_5O_{18})_2]^{9-}$, Ln = La, Ce, Nd, Gd, Tb) complexes as general basic catalyst instead of different types of HPAs, as Brønsted strong acid with too many hydrogen toward the catalyzed synthesis of a biologically active heterocyclic compounds *via* MCRs [12].

Herein, we wish to report a novel, effective, high yielding and green strategy for the synthesis of series of pyrazolopyranopyrimidine derivatives in high yields *via* a one-pot, four components reaction, involving ethyl acetoacetate, hydrazine hydrate, benzaldehydes, and barbituric acid catalyzed by sodium salts of Lindqvist-type of LnPOMs (LnW_{10}; Ln = La, Ce, Nd, Gd, Tb) as the basic catalysts under ultrasonic irradiation (Scheme 1).

In addition, the catalytic activities of these catalysts, (five LnPOM salts with different central lanthanoid ions) were compared in term of the yields of the products and reaction times.

2. Experimental

2.1. General information

All chemicals were purchased from Merck or Aldrich and used without further purification. Melting points were determined using an electro thermal 9200 apparatus with the capillary tube method. FTIR spectra were recorded by using FTIR spectrometer Bruker Tensor 27. Metal content was measured by ICP on a Varian Vistapro analyzer. The progress and completion of the reactions were monitored by TLC. All products were known and identified by comparison of their physical data with those of already reported for authentic compounds and found being identical.

2.2. Preparation of LnW_{10} catalyst

Lanthanide-containing POMs with molecular formula $Na_9LnW_{10}O_{36}\bullet nH_2O$ (LnW_{10}, Ln = La, Ce, Nd, Gd, Tb) were synthesized and their structure were reconfirmed [7,26].

Synthesis of LnW_{10}: $Na_2WO_4 \cdot 2H_2O$ (10 mmol, 3.3 g) was dissolved in 20 ml of water and the pH of the solution was adjusted to 7.0–7.5 with acetic acid. After that, 0.8 ml lanthanoid nitrate solution containing 1 mmol lanthanoid nitrate was added dropwise to the above solution under continuous stirring and heated to 85 °C. The whole solution was cooled down in an ice water bath. The LnW_{10} were obtained after filtration and recrystallized three times from warm water.

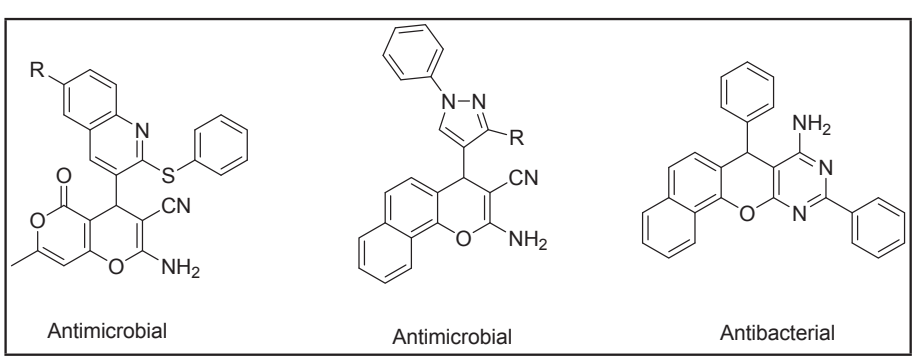
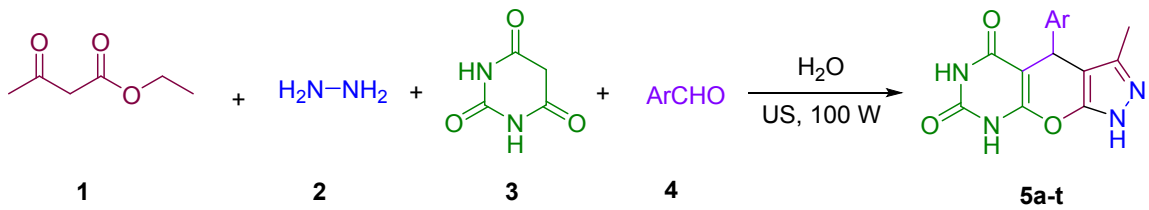


Fig. 2. Some bioactive pyrimidine and chromene-fused heterocyclic structures.



Scheme 1. Synthesis of pyrazolopyranopyrimidines *via* MCR in water.

2.3. Selected analytical data

$Na_9[LaW_{10}O_{36}]\cdot 28H_2O$. (Calcd.: La, 4.3; Na, 6.1; W, 56.3%. Anal. Found: La 4.2; Na, 6.2; W, 56.1).

$Na_9[CeW_{10}O_{36}]\cdot 38H_2O$. (Calcd.: Ce, 4.1; Na, 6.0; W, 53.3%. Anal. Found: Ce, 4.2; Na, 6.1; W, 53.2).

$Na_9[NdW_{10}O_{36}]\cdot 32H_2O$. (Calcd.: Nd, 4.3; Na, 6.2; W, 55.0%. Anal. Found: Nd, 4.2; Na, 6.1; W, 54.8).

$Na_9[GdW_{10}O_{36}]\cdot 30H_2O$. (Calcd.: Gd, 4.7; Na, 6.2; W, 55.4%. Anal. Found: Gd, 4.6; Na, 6.1; W, 55.3).

$Na_9[TbW_{10}O_{36}]\cdot 32H_2O$. (Calcd.: Tb, 4.7; Na, 6.2; W, 54.7%. Anal. Found: Tb, 4.6; Na, 6.0; W, 54.5).

2.4. Synthesis of 3-methyl-4-aryl-1,4-dihydropyrazolo[4′,3′:5,6]pyrano[2,3-d]pyrimidine-5,7(6H,8H)-diones: general procedure [27]

To a round bottomed flask containing ethyl acetate (1 mmol), hydrazine hydrate (1 mmol) was added dropwise, until a white deposit of 5-methyl-pyrrazole-2-one was obtained. Then barbituric acid (1 mmol), an appropriate substituted benzaldehyde (1 mmol), LnW_{10} (0.4 mmol) and water (5 ml) were added. The resulting mixture was placed under ultrasound irradiation at ambient temperature for indicated reaction time with power of 100 W (Table 2). After completion of the reaction (as monitored by TLC), the mixture was cooled and filtered. The precipitate was recrystallized from EtOH to give pure entitled compounds (Table 3).

2.5. 3-Methyl-4-phenyl-1,4-dihydropyrazolo[4′,3′:5,6]pyrano[2,3-d]pyrimidine-5,7(6H,8H)-dione [27]

Mp: 216–218 °C; ^{1}H NMR (400 MHz, DMSO-d_6): 2.23 (s, 3H, CH_3), 5.43 (s, 1H, CH), 7.05 (d, J = 7.6 Hz, 2H, Ph), 7.11 (t, J = 7.0 Hz, 1H, Ph), 7.21 (t, J = 7.6 Hz, 2H, Ph), 10.18 (s, 2H, 2NH), 13.18 (br s, 1H, NH); ^{13}C NMR (100 MHz, DMSO-d_6): 161.0, 151.1, 144.1, 143.0, 128.3, 127.1, 125.8, 106.3, 91.7, 56.5, 30.9, 19.0, 10.5; IR (KBr): 3420 (br), 2889 (br), 2763 (br), 2360, 1678, 1631, 1587, 1540, 1469, 1360, 1309, 781 cm^{-1}.

2.6. 3-Methyl-4-(2-methoxyphenyl)-1,4-dihydropyrazolo[4′,3′:5,6]pyrano[2,3-d]pyrimidine-5,7(6H,8H)-dione [27]

Mp: 230–231 °C; ^{1}H NMR (400 MHz, DMSOd$_6$): 2.25 (s, 3H, CH_3), 3.69 (s, 3H, OCH_3), 5.64 (s, 1H, CH), 6.81 (m, 2H, Ph), 7.11 (t, J = 7.4 Hz, 1H, Ph), 7.34 (d, J = 7.2 Hz, 1H, Ph), 10.10 (s, 2H, 2NH), 12.92 (br s, 1H, NH); ^{13}C NMR (100 MHz, DMSO-d_6): 161.1, 157.1, 151.0, 144.2, 131.1, 129.0, 127.4, 120.0, 111.0, 106.5, 90.5, 61.0, 55.7, 31.8, 26.4, 10.7; IR (KBr): 3500, 3453, 2957 (br), 2837 (br), 1691, 1610, 1480, 1459, 1394, 1294, 759 cm^{-1}.

Table 2
Synthesis of 3-methyl-4-aryl-1,4-dihydropyrazolo[4′,3′:5,6]pyrano[2,3-d]pyrimidine-5,7(6H,8H)-diones by different LnW_{10} catalyst.

Entry	Catalyst	Solvent	Temp.	Time (min)	Yield (%)
1	TbW_{10}	H_2O	US (100 W)	8	99
2	GdW_{10}	H_2O	US (100 W)	10	95
3	NdW_{10}	H_2O	US (100 W)	12	97
4	LaW_{10}	H_2O	US (100 W)	15	95
5	CeW_{10}	H_2O	US (100 W)	18	92

2.7. 3-Methyl-4-(3-nitrophenyl)-1,4-dihydropyrazolo[4′,3′:5,6]pyrano[2,3-d]pyrimidine-5,7(6H,8H)-dione [27]

Mp: 265–266 °C; ^{1}H NMR (400 MHz, DMSO-d_6): 2.26 (s, 3H, CH_3), 5.53 (s, 1H, CH), 7.54 (m, 2H, Ph), 7.85 (s, 1H, Ph), 8.02 (m, 1H, Ph), 10.26 (s, 2H, 2NH), 13.44 (br s, 1H, NH); ^{13}C NMR (100 MHz, DMSO-d_6): 160.5, 151.1, 148.1, 145.9, 143.9, 134.3, 129.9, 121.6, 121.2, 105.2, 91.3, 58.1, 31.2, 19.5, 10.5; IR (KBr): 3598, 3469 (br), 3031 (br), 2920 (br), 1701, 1586, 1527, 1477, 1355, 1310, 810, 543 cm^{-1}.

2.8. 3-Methyl-4-(4-nitrophenyl)-1,4-dihydropyrazolo[4′,3′:5,6]pyrano[2,3-d]pyrimidine-5,7(6H,8H)-dione [27]

Mp: 231–233 °C; ^{1}H NMR (400 MHz, DMSO-d_6): 2.25 (s, 3H, CH_3), 5.51 (s, 1H, CH), 7.31 (d, J = 8.4 Hz, 2H, Ph), 8.11 (d, J = 8.8 Hz, 2H, Ph), 10.24 (s, 2H, 2NH), 13.26 (br s, 1H, NH); ^{13}C NMR (100 MHz, DMSO-d_6): 160.5, 151.7, 151.1, 146.0, 144.1, 128.5, 123.6, 105.3, 91.5, 60.2, 31.5, 14.5, 10.5; IR (KBr): 3148 (br), 3056 (br), 2901 (br), 1693, 1589, 1517, 1468, 1351, 1274, 832, 547 cm^{-1}.

2.9. 3-Methyl-4-(4-methylphenyl)-1,4-dihydropyrazolo[4′,3′:5,6]pyrano[2,3-d]pyrimidine-5,7(6H,8H)-dione [27]

Mp: 200–202 °C; ^{1}H NMR (400 MHz, DMSO-d_6): 2.22 (s, 6H, 2CH_3), 5.38 (s, 1H, CH), 6.92 (d, J = 8.0 Hz, 2H, Ph), 7.00 (d, J = 8.0 Hz, 2H, Ph), 10.17 (s, 2H, 2NH), 13.20 (br s, 1H, NH); ^{13}C NMR (100 MHz, DMSO-d_6): 161.1, 151.1, 144.0, 140.0, 134.6, 129.0, 127.1, 106.5, 91.8,

Table 1
Optimization of the reaction conditions for the model reaction involving hydrazine hydrate, ethyl acetoacetate, barbituric acid and benzaldehyde.

Entry	Catalyst	Solvent	Temp (°C)	Time (min)/Yield %	Cond.	Time (min)/Yield %
1	LaW_{10}	EtOH	reflux	45/89	US (100 W)	18/92
2	LaW_{10}	CH_3CN	reflux	50/88	US (100 W)	20/88
3	LaW_{10}	H_2O/EtOH	reflux	47/90	US (100 W)	15/90
4	LaW_{10}	H_2O	reflux	45/92	r.t	50/92
5	LaW_{10}	H_2O	reflux	42/94	US (100 W)	15/95
6	LaW_{10}	H_2O	reflux	41/94	US (80 W)	20/91

Table 3
Synthesis of pyrazopyrimidine derivatives *via* a four-component reaction catalyzed by TbW_{10}.

Entry	Ar	Product	Time (min)	Yield (%)	Mp (°C) Obs.	Mp (°C) Ref. [27]
1	C_6H_5	5a	8	99	216–218	218–219
2	2MeO–C_6H_4	5b	12	95	228–231	230–231
3	3MeO–C_6H_4	5c	10	95	219–222	221–222
4	3,4(MeO)$_2$–C_6H_3	5d	18	93	275–277	275–276
5	2OH–C_6H_4	5e	12	91	158–161	159–160
6	4Me–C_6H_4	5f	18	94	200–202	201–201
7	2F–C_6H_4	5g	12	95	221–223	223–224
8	3F–C_6H_4	5h	12	94	241–244	242–244
9	4F–C_6H_4	5i	8	97	236–238	237–238
10	2Cl–C_6H_4	5j	10	98	222–224	223–225
11	3Cl–C_6H_4	5k	12	92	246–248	246–247
12	4Cl–C_6H_4	5l	10	97	222–224	222–223
13	2,4-Cl$_2$-C_6H_3	5 m	18	98	232–234	233–234
14	2Br–C_6H_4	5n	12	94	250–253	250–251
15	3Br–C_6H_4	5°	15	91	128–129	128–129
16	4Br–C_6H_4	5p	8	98	210–212	211–212
17	2NO_2–C_6H_4	5q	12	94	208–210	208–209
18	3NO_2–C_6H_4	5r	18	93	265–266	266–267
19	4NO_2–C_6H_4	5s	8	94	231–233	233–234
20	1-Naphthaldehyde	5t	10	91	220–222	221–222

56.5, 30.6, 21.0, 19.0, 10.5; IR (KBr): 3422, 3121 (br), 3041 (br), 1687, 1579, 1516, 1469, 1359, 1271, 828, 789, 543 cm^{-1}.

2.10. 3-Methyl-4-(4-chlorophenyl)-1,4-dihydropyrazolo[4′,3′:5,6]pyrano[2,3-d]pyrimidine-5,7(6H,8H)-dione [27]

Mp: 222–224 °C; ^{1}H NMR (400 MHz, DMSO-d_6): 2.08 (s, 3H, CH_3), 5.27 (s, 1H, CH), 7.06 (dd, J1 = 1.0 Hz, J2 = 8.6 Hz, 2H, Ph), 7.17 (d, J = 8.8 Hz, 2H, Ph), 9.41 (s, 2H, 2NH), 12.02 (s, 1H, NH); ^{13}C NMR (100 MHz, DMSO-d_6): 160.6, 151.2, 144.1, 142.0, 130.5, 129.1, 128.6, 105.9, 91.6, 55.9, 30.7, 19.1, 10.5; IR (KBr): 3130 (br), 1691, 1592, 1481, 1368, 1298, 837, 776, 551 cm^{-1}.

3. Results & discussion

3.1. Synthesis and characterization of catalysts

Five $Na_9[Ln(W_5O_{18})_2]\bullet nH_2O$ catalysts of this study were obtained by reaction of lanthanoid nitrate with hot aqueous sodium tungstate at pH 7.0–7.5 and characterized by FT-IR and ICP analyses. The four main characteristic vibration bands of LnW_{10} are as follows: 935-949 cm^{-1} ascribed to ν(W=O_d), (840–844) cm^{-1} assigned to ν(W-O_b-W), and attributed 784–787 and 705-706 cm^{-1} to ν(W-O_c-W) (O_b is the bridged oxygen of two octahedra sharing a corner, O_c is the bridged oxygen of two octahedra sharing an edge and O_d is the terminal oxygen) [26]. As a representative example, the FT-IR spectrum of GdW_{10} is represented in Fig. 3.

3.2. Catalytic activity

Primarily, for finding and securing the optimal conditions, the four components reaction of hydrazine hydrate, barbituric acid, ethyl acetoacetate, and benzaldehyde was selected as a model reaction. In the absence of the catalyst after 6 h, the corresponding product was obtained in 10% yield. Then to the purpose, diverse kind and amount of catalyst say, LaW_{10}, different solvents were examined at room temperature and the temperature was raised to reflux conditions. Having the optimized conditions, we contemplated that is worthwhile to conduct the reaction under unconventional but green source of energy. Thus, we performed the model reaction under ultrasonic irradiation, this model reaction was accomplished under reflux condition and the yield of product was compared with that of obtained by ultrasonic irradiation. Satisfyingly, under ultrasonic irradiation the corresponding product was obtained in higher yield and shorter reaction time. (18 min) (Table 1). Without the catalyst after 6 h, the product was obtained in 10% yield.

Under optimal reaction conditions, we next examined different LnW_{10} catalysts in the above -mentioned reaction (Table 2). The catalyst with heavier lanthanoid ions (Tb, Gd) were found to give better yields in comparison with those with lighter ones (La, Ce). It appears that the lanthanoid hetero-atom plays effective role in these catalysts. It is supposed that the different yield and required reaction times for the preparation of pyrazolopyranopyrimidine may be related to the ionic size of hetero-atom. By increasing the atomic number and decreasing of ionic size, the conversion required a shorter reaction time.

Under secured optimal reactions, other pyrazopyrimidine derivatives were synthesized using differently substituted benzaldehydes in the presence of TbW_{10} as catalyst. This protocol showed broad substrate scope. Irrespective of the substituent present on the aromatic ring of the benzaldehyde, the corresponding products were obtained in excellent yields, and all the reactions were complete within 8–18 min. The results are represented in Table 3.

3.3. Reaction mechanism

A possible reaction mechanism for the synthesis of pyrazolopyranopyrimidine is depicted in Scheme 2. The reaction can be initiated by the formation of 3-methyl-1H-pyrazol-5(4H)-one from ethyl acetoacetate and hydrazine which subsequently reacted with aldehyde to form intermediate I from Knoevenagel condensation in the presence of catalyst. Then, the generated intermediate undergoes reaction with barbituric acid to afford the intermediate II. The final product is obtained by ring closure and dehydration.

To demonstrate the suitability of the presented catalyst, its catalytic activity for the synthesis of the model compound was compared with those reported previously (Table 4). The reaction time and yield of product in the presence of this active catalyst and under ultrasonic conditions were better than those with catalysts tabulated in Table 4.

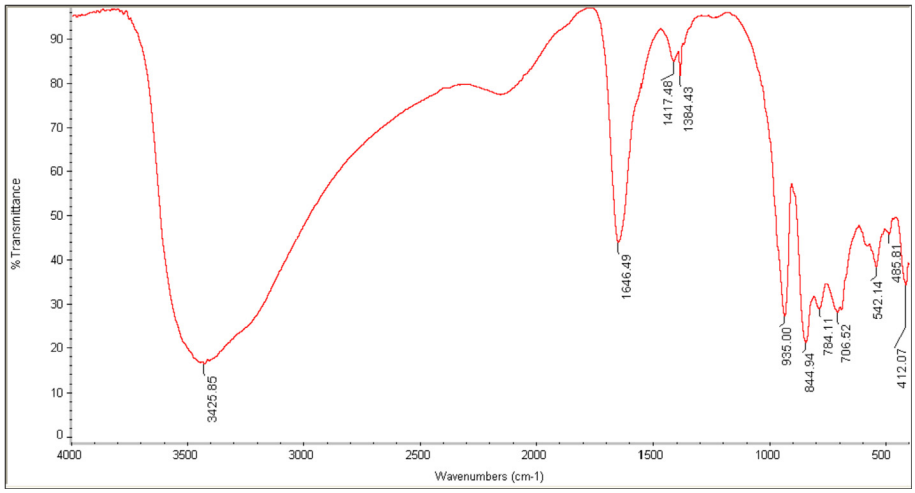
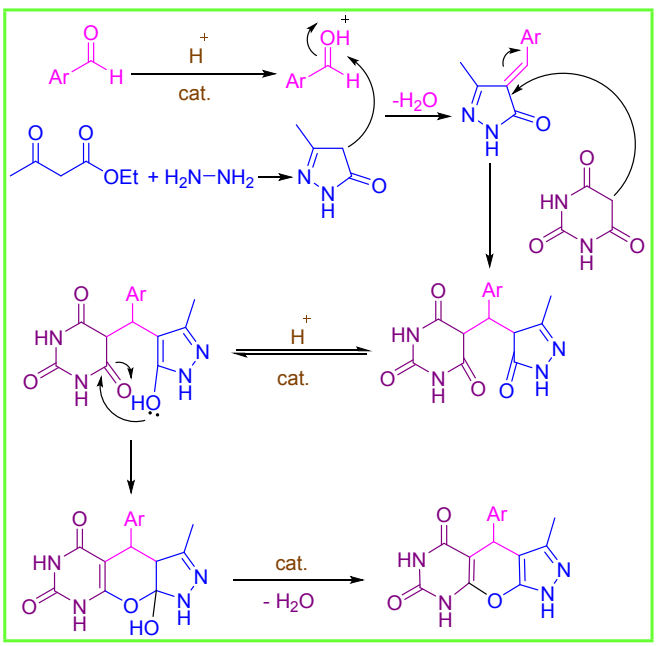


Fig. 3. FT-IR spectrum of GdW_{10} catalyst.



Scheme 2. Plausible mechanism for synthesis of pyrazolopyranopyrimidines.

Table 4
Comparison of the catalytic activity of active catalyst in ultrasonic conditions with those of reported catalysts.

Entry	Catalyst	Time (min)	Yield (%)	Ref.
1	Meglumine	15	95	[27]
2	TiO_2 NWs	60	95	[28]
3	OMWCNTs	70	94	[29]
4	Oleic acid	180	78	[30]
5	HPA-F-HNTs	35	96	[31]
6	LnW_{10}	8	99	This work

4. Conclusions

In summary, a series of $[Ln(W_5O_{18})_2]^{9-}$ (Ln = La, Ce, Nd, Gd, Tb) were prepared and identified by infrared spectroscopy and inductively coupled plasma. They were used as novel basic catalysts for the promotion of the MCR, including ethyl acetoacetate, hydrazine hydrate, benzaldehydes, and barbituric acid under ultrasonic irradiation to afford pyrazolopyranopyrimidines under ultrasound irradiation at room temperature to give corresponding pyrazolopyranopyrimidines in excellent yields within 8–45 min. The obtained results also showed the activity of catalysts increases when the size of the lanthanoid heteroatom decreased, basically in terms of yields of products and reaction times in order of Tb > Gd > Nd > La > Ce. Preservation of environment, and comply of the green chemistry principles has always been our research group concerns.

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