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ARTICLE TYPE

A Highly efficient and recyclable molybdate sulfuric acid (MSA) catalyst for the synthesis of dimethyl (2,3-dihydro-1H-inden-6-ylamino)(substituted) methylphosphonates under microwave irradiation

MudumalaVeeranarayanaReddy,^aGangireddy Chandra SekharReddy,^bYeon Tae Jeong^{a*}

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An efficient green synthesis of dimethyl (2,3-dihydro-1H-inden-6-ylamino)(substituted) methylphosphonates has been achieved under solvent-free conditions by the reaction of 2,3-dihydro-1H-inden-5-amine, aldehydes and ethyl dimethyl phosphonate by microwave irradiation in the presence of molybdate sulfuric acid (MSA) as catalyst. High product yields in less reaction time, easy isolation of products, use of reusability of solid catalysts and environmentally benign reaction conditions are its advantages.

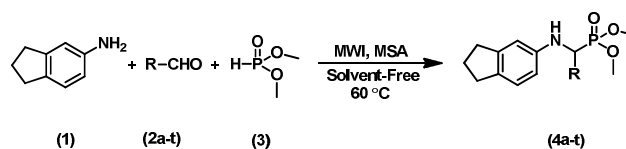
Organophosphorus Compounds (OPCs) have received considerable attention because of their pivotal roles in various biological activities.¹ Of these, the aminophosphonates have been shown to be effective herbicides, fungicides, insecticides, HIV protease, enzyme inhibitors, plant growth regulators, neuroprotective anti-coagulating agents, antibacterial, anticancer, cytotoxic, anti-thrombotic agents, and calcium channel modulators.² In view of this a number of synthetic modifications have been reported³ either solid or polymer supported with or without catalyst under either solvent or solvent free thermal/ultrasound/ microwaves reaction conditions. However, these methods suffer from draw backs, such as high reaction temperatures, long reaction times, need of expensive catalysts, and unsatisfactory yields. Consequently, investigations are still going for a better method for the synthesis of organophosphonates.

Now-a-days use of green chemical process is gaining importance in global chemical industry. Currently, multi-component one-pot syntheses under microwave-assisted organic synthesis are accepted widely as environmental benign efficient synthetic methodology.⁴

^bDepartment of Chemistry, Sri Venkateswara College of Engineering, Karakambadi Road, Tirupati – 517 507, India.

Moreover microwave promoted solvent-free heterogeneous reactions⁵ are particularly welcome due to their essential advantages such as enhanced reaction rates, cleaner products, manipulative simplicity, and improved product selectivity. In the recent years, heterogeneous catalysts have found increased application in organic synthesis⁶ as they are efficient, easily recovered and recycled. In this context molybdate sulfuric acid (MSA) being an efficient proton source was found synthetically useful in organic reactions.⁷ It has many advantages over conventional acid catalysts, such as ease of handling, stability, less cost, easy recyclability due to insolubility in most of the organic solvents. Thus, it has been selected as a solid heterogeneous alternative to sulfuric acid.

As part of our continuing research to develop a green reaction methodology by microwave irradiation (MWI) under solvent-free heterogeneous organic synthesis^{5f} literature survey revealed that there are no reports on MWI mediated solvent-free MAS catalysed synthesis for organophosphonates. Herein, we report a facile solvent-free one-pot neat synthesis of dimethyl (2,3-dihydro-1H-inden-6-ylamino)(substituted) methylphosphonates via three-component Kabachnik-Fields coupling reaction of 2,3-dihydro-1H-inden-5-amine, various aldehydes and dimethyl phosphonate in the presence of catalytic amount of MSA (5 mol% (w.r.t. reactants)) under microwave irradiation at 60 °C (Scheme 1).



Scheme 1: Synthesis of dimethyl (2,3-dihydro-1H-inden-6-ylamino) (substituted) methyl phosphonates.

^aDepartment of Image Science and Engineering, Pukyong National University, Busan, Korea, 608-737, *Corresponding author. Tel.: +82-51-629-6411; fax: +82-51-629-6408; e-mail: ytjeong@pknu.ac.kr

To develop optimal reaction conditions, we carried out the reaction between 2,3-dihydro-1*H*-inden-5-amine (**1**), 4-chlorobenzaldehyde (**2a**) and ethyl dimethyl phosphonate (**3**) as a model. It was investigated by utilizing different catalysts under both conventional and microwave conditions without solvent. Catalysts such as Y(OAc)₃, H₂O, GaBr₃, GaCl₃, and NbCl₅ either completely impede the reaction or diminish the yield of the product (Table 1, entries 1–4). Due to numerous advantages with heterogeneous solid acid catalysed reactions, the reaction was performed with STA, FeCl₃·SiO₂, PS-PTSA, InF₃, and K-10 and obtained moderate product yields (Table 1, entries 5–9). Subsequently the reaction was performed in the presence MSA, to obtain the desired product (**4a**) in high yields (Table 1, entry 10).

When studied the required amount of MSA catalyst for maximum efficiency it was found that 5 mol % of MSA (Table 1, entry 10) was sufficient to get optimum product yield. Excessive amount of catalyst did not increase the yields considerably (Table 1).

The effect of solvent on the model reaction was studied in both conventional and microwave conditions using 5 mol% of MSA in different solvents and without solvent. The reaction was also studied at varying temperature (40, 60 and 80 °C). The optimum conversion of reactants to product was achieved under solvent-free conditions at 60°C.

The recyclability of the MSA catalyst was also established by running the same model reaction in three cycles with recovered MSA and obtained **4a** in 97, 94, 91 and 87% product yield. This proved that efficiency of the catalyst can be used for multiple usage purpose without much loss of its efficiency (Table 1, entry 10). From these all establishments (Table 1) concluded that 5 mol% of MSA, solvent-free and 60 °C MWI are optimized reaction conditions for the synthesis of α-aminophosphonates (**4a-t**).

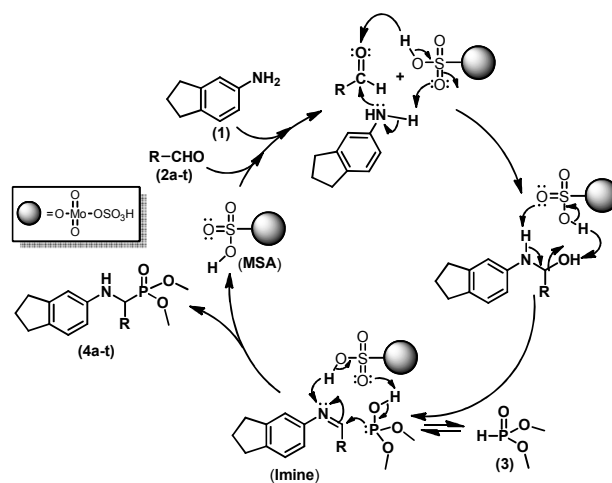
Under these optimized set of experimental reaction conditions the condensation of dimethyl phosphonate (**3**) with different aromatic aldehydes (**2a-t**) and 2,3-dihydro-1*H*-inden-5-amine (**1**) was carried out and obtained (2,3-dihydro-1*H*-inden-6-ylamino)(substituted) methyl phosphonates (**4a-t**) in good to excellent yields without the formation of any side products. This catalyst worked excellently for aromatic aldehydes bearing both electron-donating and electron-withdrawing substituents. Heteroaromatic aldehydes such as thiophene-2-carboxaldehyde, and 2-pyridinecarboxaldehyde also participated in the reaction and gave higher product yields.

All the titled compounds are well characterized by analytical and spectral data. All the N-H protons resonated at δ 4.42-7.25. The two methoxy protons on phosphorus gave as two separate doublet signals at δ 3.36-3.62 and δ 3.76-3.86 due to interaction with phosphorus. This indicates that the two methoxy groups are magnetically different even though they are connected to the same phosphorus atom. It is also confirmed by CMR spectrum shown as two distinguished doublets at δ 53.5-55.9 and δ 51.5-54.2. The C-2 methyn proton present in the vicinity of N and P resonated at high frequency region as doublet at δ 5.57-4.68 due to strong coupling with phosphorus atom. On the other hand

CMR signal of this carbon also appears in lower field region at δ 24.32-27.25. Rest of the aromatic and other protons and carbons resonated at their expected frequency regions. The ³¹P NMR spectrum was also shown that strong evidence for aminophosphonates as singlet peak at δ 24.32-27.25.

The formation of high yields of the products (**4a-t**) is explained mechanistically in **Scheme 2**. The catalyst MSA appear to plays a key role initially as acid followed by as base in the reaction and helps formation of the imine intermediate. Subsequent nucleophilic attack of phosphite (**3**) on imine affords the product.

In summary, we have developed an efficient and green method for the synthesis dimethyl (2,3-dihydro-1*H*-inden-6-ylamino)(substituted)methylphosphonates (**4a-t**) by three-component neat Kabachnik-Fields reaction between 2,3-dihydro-1*H*-inden-5-amine (**1**), aldehydes (**2a-t**) and dimethyl phosphonate (**3**) using catalytic amount of MSA with MWI. The attractive features of this method are simple procedure, green reaction, reusability and high efficiency of the catalyst and easy workability.

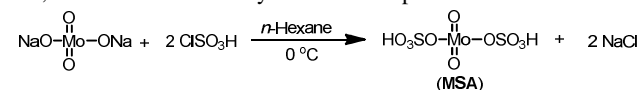


Scheme 2: Schematic presentation of MSA catalyst activity in the synthesis of **4a-t**

Experimental

Preparation of MSA

To dry *n*-hexane (25 mL) in a 100 mL round bottom flask equipped with overhead stirrer and kept in an ice bath was added a suspension of anhydrous sodium molybdate (20 mmol, 4.118 g). To this solution drop wise added chlorosulfonic acid (0.266 mL, 40 mmol) during 30 min and stirred for 1.5 h (**Scheme 3**). The reaction mixture was gradually poured into 25 mL of chilled distilled water with stirring. MSA was separated by filtration and it was washed 5-6 times with cold distilled water until its filtrate tests negative for chloride ions. It was dried at 120 °C for 5 h, and obtained in 91% yield as bluish powder.



Scheme 3: Synthesis of MSA

Synthesis of dimethyl (2,3-dihydro-1H-inden-6-ylamino)(4-chlorophenyl)methylphosphonate (4a)

A mixture of 2,3-dihydro-1H-inden-5-amine (**1**, 1 mmol, 133 mg), 4-chlorobenzaldehyde (**2a**, 1 mmol, 140 mg), dimethyl phosphonate (**3**, 1 mmol, 108 mg) and MSA (5 mol %, 16 mg) was taken in an open vessel in CATA-4R – Scientific Microwave oven and irradiated at 60 °C (140 W) at ambient pressure in solvent-free condition for 4 min. The reactions were followed by thin layer chromatography (TLC) using hexane/ethyl acetate as an eluent. After completion of the reaction, the mixture was

washed with ethyl acetate and filtered to recover the catalyst. The filtrate was evaporated, and the crude product was recrystallized from ethanol to afford pure dimethyl(2,3-dihydro-1H-inden-6-ylamino)(4-chlorophenyl)methylphosphonate (**4a**) in excellent yield. The MSA catalyst was reused by the way of addition of ethyl acetate to the reaction mixture and filtration followed by drying in air every time.

20

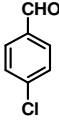
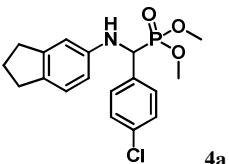
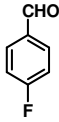
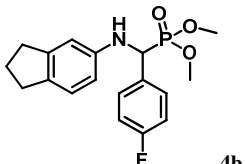
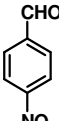
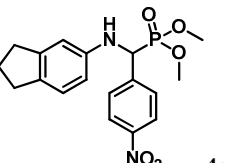
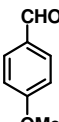
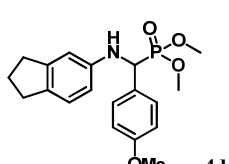
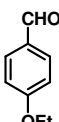
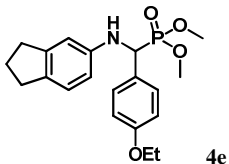
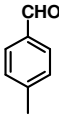
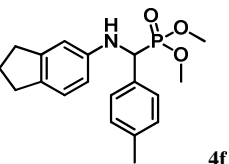
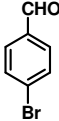
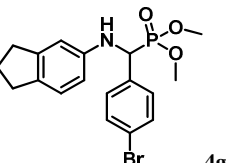
Table 1 Optimization of reaction conditions for the synthesis of **4a**^a

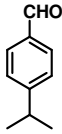
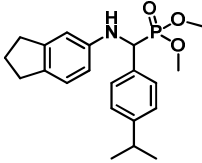

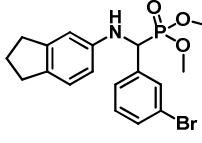

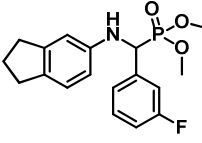

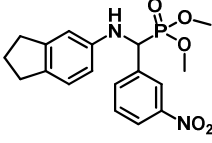

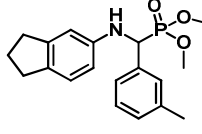

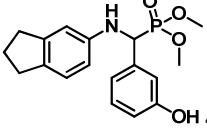

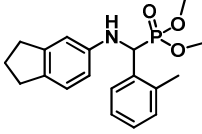

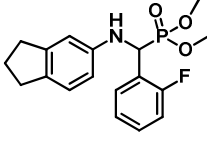
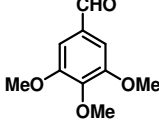
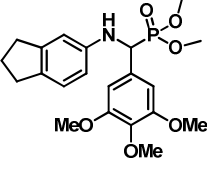
Entry	Catalyst (mol%)	Solvent	Temp (°C)	Conventional		Microwave	
				Time (min)	Yield ^b (%)	Time(min)	Yield ^b (%)
1	Y(OAc) ₃ .H ₂ O (5)	neat	60	110	30	35	45
2	GaBr ₃ (5)	neat	60	90	35	50	55
3	GaCl ₃ (5)	neat	60	100	30	60	52
4	NbCl ₅ (5)	neat	60	120	45	70	53
5	STA(5)	neat	60	70	75	20	85
6	FeCl ₃ .SiO ₂ (5)	neat	60	65	60	40	74
7	PS-PTSA (5)	neat	60	80	60	45	78
8	InF ₃ (5)	neat	60	75	65	35	75
9	K-10 (0.05 g)	neat	60	72	60	30	77
10 ^c	MSA (5)	neat	60	20	85	4	97, 94, 91, 87
11	MSA (2)	neat	60	40	65	20	75
12	MSA (3)	neat	60	30	70	10	85
13	MSA (8)	neat	60	20	85	4	97
14	MSA (5)	H ₂ O	60	40	60	15	86
15	MSA (5)	[bmim][Cl]	60	45	55	20	65
16	MSA (5)	Toluene	60	50	60	30	75
17	MSA (5)	Ethanol	60	45	75	15	90
18	MSA (5)	neat	40	35	68	18	80
19	MSA (5)	neat	80	20	85	4	97

^aReaction of 2,3-dihydro-1H-inden-5-amine (**1**, 1 mmol), 4-chlorobenzaldehyde (**2a**, 1 mmol), dimethyl phosphonate (**3**, 1 mmol); ^bIsolated yield; ^cCatalyst was reused four times.

25

5 **Table 2:** Microwave-assisted synthesis of dimethyl (2,3-dihydro-1*H*-inden-6-ylamino) (substituted) methyl phosphonates.^a

Entry	Aldehyde	Product	Time (min)	Yield ^b (%)	mp (°C)
1			4	97	136-138
2			5	96	122-124
3			7	94	146-148
4			5	96	109-111
5			5	97	88-90
6			6	95	90-92
7			9	92	125-127

8		 4h	10	90	80-82
9		 4i	8	91	98-100
10		 4j	7	94	98-100
11		 4k	6	92	123-125
12		 4l	5	92	96-98
13		 4m	7	93	145-147
14		 4n	5	93	113-115
15		 4o	6	94	97-99
16		 4p	8	93	148-150

17			6	91	121-123
18			8	92	88-90
19			8	91	114-116
20			10	91	101-103

^aReaction of 2,3-dihydro-1H-inden-5-amine (**1**, 1 mmol), aldehydes (**2a-t**, 1 mmol), dimethyl phosphonate (**3**, 1 mmol) catalysed by MSA under solvent-free microwave irradiation at 60 °C.; ^bIsolated yield.

Supporting Information

⁵ Analytical and spectral data and NMR spectra were provided as supplementary data for all Compounds.

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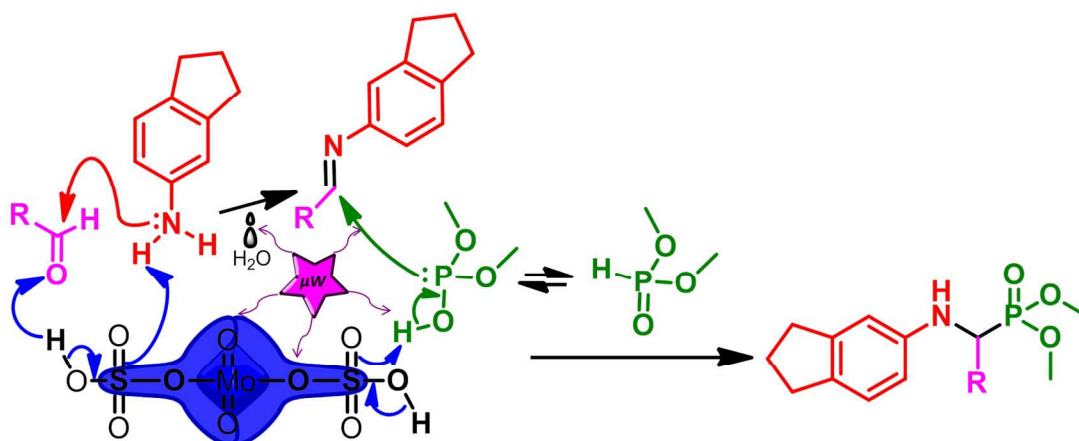
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A Highly efficient and recyclable Molybdate sulfuric acid (MSA) catalyst for the synthesis of dimethyl (2,3-dihydro-1*H*-inden-6-ylamino)(substituted) methylphosphonates under microwave irradiation

Mudumala Veerananarayana Reddy,^a Gangireddy Chandra Sekhar Reddy,^b Yeon Tae Jeong^{a*}

^aDepartment of Image Science and Engineering; Pukyong National University, Busan 608-737, Korea, Tel.: +82-51-629-6411; fax: +82-51-629- 6408; E-mail: ytjeong@pknu.ac.kr

^bDepartment of Chemistry, Sri Venkateswara College of Engineering, Karakambadi Road, Tirupati – 517 507, India.



The attractive features of this methodology are simple procedure, green reaction, reusability and high efficiency of the catalyst and easy workability.