Mild and efficient oxidation of 2-pyrazolines and isoxazolines by *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxalane–NH₄Cl–HOAc in water–MeCN

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Abstract An efficient method has been developed for oxidative aromatization of 2-pyrazolines and isoxazolines into their corresponding 2-pyrazoles and isoxazoles under mild conditions. In this method, *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxalane–NH₄Cl–HOAc was used as a novel and effective oxidant in water–MeCN at room temperature with good yields.

Keywords *trans*-3,5-Dihydroperoxy-3,5-dimethyl-1,2-dioxalane · 2-Pyrazolines · Isoxazolines · Pyrazoles · Isoxazoles · Ammonium chloride

Introduction

Isoxazolines and pyrazolines are interesting five-membered heterocyclic compounds because of their biological and pharmacological importance, for example as anti-cancer [1], anti-viral [2], anti-inflammatory [3], anti-diabetic [4], anti-bacterial [5], antimicrobial, and antifungal agents [6]. Several isoxazoles and pyrazoles have agrochemical herbicidal and soil fungicidal activity and act as pesticides and insecticides [7, 8]. Isoxazoles and pyrazoles are also useful synthetic intermediates capable of undergoing a variety of transformations and transition-metal catalyzed cross-coupling reactions, for example Heck, Stille, Suzuki, Sonogashira, and Negishi coupling [9]. Isoxazoles and pyrazoles are also in demand because of their importance both as synthetic intermediates [10] and as pharmacological agents [11]. Isoxazoles and 2-pyrazoles can be easily obtained by oxidation of isoxazolines [12–17] and 2-pyrazolines [18], respectively. Several methods have been reported for oxidation of isoxazolines and 2-pyrazolines including Pd–C–acetic acid [19], carbon-activated oxygen [20], cobalt soap of fatty acids [21], lead tetraacetate [22],

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Scheme 1 Oxidative aromatization of 2-pyrazolines and isoxazolines to the corresponding products

mercury or lead oxide [23], manganese dioxide [24], potassium permanganate [24, 25], silver nitrate [26], iodobenzene diacetate [27], zirconium nitrate [28], nickel peroxide [29], chromite [16], *N*-bromosuccinimide (NBS) [30], manganese triacetate [31], sodium bicarbonate–dimethylformamide [32], 2,3-dichloro-5,6-dicyanobenzoquinone (DDQ) [33], sodium carbonate–methanol [10], and tetrakis pyridine nickel(II) dichromate [34]. However, many of these methods have such disadvantages as long reaction times, low yields, and toxicity because of the elements present in the reagents used.

Recently, *gem*-dihydroperoxides have been synthesized and have been of interest as new and effective oxidants in organic synthesis [17, 35–41]. We have synthesized *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxalane and used it as a new, powerful, solid and effective oxidant in organic synthesis [42–46]. So, in continuation of our interest in the application of *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxalane, we report the use of *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxalane–NH₄Cl–HOAc for in situ generation of Cl⁺ from NH₄Cl for catalysis of oxidative aromatization of 2-pyrazolines and isoxazolines (Scheme 1).

Experimental

Caution

Although we did not encounter any problem with *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxolane, it is potentially explosive and should be handled with precautions; all reactions should be carried out behind a safety shield inside a fume hood and transition metal salts or heating should be avoided.

General

The materials were purchased from Merck and Fluka and were used without additional purification. All reactions were monitored by thin-layer chromatography (TLC) on silica gel F254 plates. Melting points were measured in open capillary tubes by use of an Electrothermal 9100 apparatus. Nuclear magnetic resonance spectra were recorded on a Jeol FX 90Q spectrometer with tetramethylsilane (TMS) as internal standard. IR spectra were recorded on a Perkin Elmer GX FT IR spectrometer (KBr pellets). 2-Pyrazolines and isoxazolines used in this work were

Scheme 2 Synthesis of *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxalane (**1**)

all prepared as reported elsewhere [11, 14–16]. 2-Pyrazoles and isoxazoles were characterized by comparison of their melting points and IR, ^1H NMR, and ^{13}C NMR spectra with reported data.

Preparation of trans-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxalane [46]

Silica sulfuric acid (SSA) (100 mg) was added to a stirred solution of acetylacetone (100 mg, 1 mmol) in CH_3CN (4 ml), and stirring of the reaction mixture was continued for 5 min at room temperature. Aqueous 30 % H_2O_2 (5 mmol) was then added to the reaction mixture and stirring was continued for 30 min at room temperature. After completion of the reaction (monitored by TLC) the resulting mixture was filtered and the residue was washed with EtOAc (2×5 ml) to separate the solid catalyst. The combined filtrates were diluted with water (5 ml) and extracted with EtOAc (3×5 ml). The organic layer was separated, dried over anhydrous Mg_2SO_4 and evaporated under reduced pressure to give almost pure white crystalline product **1**. (Scheme 2).

General experimental procedure for oxidative aromatization of 2-pyrazolines and isoxazolines

A mixture of 2-pyrazolines (or isoxazolines) (1 mmol), NH_4Cl (0.1 mmol, 0.0054 g), and acetic acid (0.05 mmol, 0.029 ml) was prepared in water (3 ml) and MeCN (2 ml). *trans*-3,5-Dihydroperoxy-3,5-dimethyl-1,2-dioxalane (0.3 mmol, 0.05 g) was then added, and the mixture was stirred for an appropriate time at room temperature. The progress of the reaction was followed by TLC. After completion of the reaction, 1 ml Na_2SO_3 (2 M) was added and the reaction was stirred for 10 min. Water (15 ml) was then added to precipitate the products, which are insoluble in water. Precipitates were isolated by filtration and dried for obtain pure products.

Results and discussion

For aromatization of 2-pyrazolines several oxidants have been used. Many of these involve use of toxic metals, and the reaction conditions are often harsh. In addition, the yields obtained by use of these methods are usually poor to moderate. Oxidation of isoxazoline is difficult, because of the undesirable ring opening that may occur. In this paper we report a mild, effective, and more environmentally benign method for oxidative aromatization of isoxazolines and 2-pyrazolines at room temperature in high yields. It is notable that no by-products were observed from ring-opening (Scheme 1).

Table 1 Optimization of the reaction conditions for oxidation of 1,3,5-triphenyl-1*H*-pyrazole

Entry	Oxidant (1) (mmol)	NH ₄ Cl (mmol)	HOAc (mmol)	Solvent	Time (h)	Yield (%)
1	0.3	0.8	–	MeCN	2	35
2	0.3	0.5	–	MeCN	2	44
3	0.3	0.2	–	MeCN	1.5	62
4	0.3	0.1	–	MeCN	1.5	75
5	0.3	0.05	–	MeCN	3	70
6	0.3	0.1	0.05	MeCN	0.6	94
7	0.3	0.1	0.05	CHCl ₃	1.2	40
8	0.3	0.1	0.05	H ₂ O	1.2	53
9	0.3	0.1	0.05	THF	0.6	87
10	0.3	0.1	0.05	H ₂ O–MeCN	0.4	96
11	0.1	0.1	0.05	H ₂ O–MeCN	1.3	85
12	0.2	0.1	0.05	H ₂ O–MeCN	1.1	90
13	0.5	0.1	0.05	H ₂ O–MeCN	0.3	94
14	1	0.1	0.05	H ₂ O–MeCN	0.3	72

Reactants: 1,3,5-triphenyl-1*H*-pyrazole (1 mmol), solvent (5 ml) (for H₂O–MeCN: 3 ml H₂O + 2 ml MeCN)

Scheme 3 Suggested mechanism for oxidative aromatization of 2-pyrazolines and isoxazolines

trans-3,5-Dihydroperoxy-3,5-dimethyl-1,2-dioxalane is synthesized by an easy procedure from available and inexpensive compounds (Scheme 2) and is a solid and powerful oxidant which overcomes the defects of hydrogen peroxide (for example problems of weighing, weak power in organic solvents, need for catalyst, etc.). We have used *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxalane for in-situ generation of Cl⁺ as an effective electrophile from NH₄Cl.

Initially, the amounts of oxidant, NH₄Cl, and solvent were optimized for oxidation of 1,3,5-triphenyl-1*H*-pyrazole (entry 1, Table 1). To obtain the results

Table 2 Oxidative aromatization of 2-pyrazolines (1a–p) and isoxazolines (1q–y) to the corresponding pyrazoles (2a–p) and isoxazoles (2q–y) with *trans*-3,5-dihydroperoxy-3,5-dimethyl-1,2-dioxalane–NH₄Cl–HOAc at rt

Substrate ^a	Product	X	R ¹	R ²	Time (h)	Yield (%) ^b	Mp (°C)	
							Found	Reported
1a	2a	NPh	C ₆ H ₅	C ₆ H ₅	0.4	96	136–138	133–134 [47]
1b	2b	NPh	C ₆ H ₅	4-NO ₂ C ₆ H ₄	2.7	91	142–144	144–146 [47]
1c	2c	NPh	C ₆ H ₅	4-CH ₃ OC ₆ H ₄	2.6	90	78–80	79–80 [47]
1d	2d	NPh	C ₆ H ₅	4-ClC ₆ H ₄	3.1	92	113–115	114–115 [48]
1e	2e	NPh	C ₆ H ₅	3-ClC ₆ H ₄	3	91	96–98	93–95 [48]
1f	2f	NPh	4-MeOC ₆ H ₄	C ₆ H ₅	4.2	85	76–78	77–79 [48]
1g	2g	NPh	2-Naphthyl	4-ClC ₆ H ₄	3	89	131–133	130–133 [49]
1h	2h	NPh	2-Naphthyl	2-MeC ₆ H ₄	3.6	89	146–148	148–150 [50]
1i	2i	NPh	2-Naphthyl	2-ClC ₆ H ₄	3.7	87	70–72	67–70 [50]
1j	2j	NPh	2-Thienyl	4-ClC ₆ H ₄	2.8	96	130–132	135–138 [51]
1k	2k	NPh	3-Thienyl	4-ClC ₆ H ₄	3.1	94	146–148	145–148 [51]
1l	2l	NPh	3-Thienyl	4-Me ₂ NC ₆ H ₄	3.7	90	118–120	120–123 [51]
1m	2m	NPh	2-Naphthyl	2-MeC ₆ H ₄	3.1	89	148–150	148–150 [47]
1n	2n	NPh	4-CH ₃ C ₆ H ₄	3-CH ₃ C ₆ H ₄	3.2	91	96–98	94–96 [47]
1o	2o	NPh	4-CH ₃ OC ₆ H ₄	2-MeC ₆ H ₄	3.5	85	71–73	70–71 [47]
1p	2p	NPh	4-CH ₃ C ₆ H ₄	2-Furyl	3.8	84	97–99	96–98 [47]
1q	2q	O	1-Naphthyl	2-ClC ₆ H ₄	5	91	66–68	64–65 [52]
1r	2r	O	2-Naphthyl	2-ClC ₆ H ₄	5	86	91–93	94–95 [52]
1s	2s	O	2-Naphthyl	2-Thienyl	5.3	83	117–119	118–120 [52]
1t	2t	O	2-Naphthyl	4-MeOC ₆ H ₄	5.5	84	106–108	107–109 [52]
1u	2u	O	2-Naphthyl	3-ClC ₆ H ₄	5.4	78	91–93	90–92 [52]
1v	2v	O	2-Naphthyl	2-Styryl	5	94	164–166	165–167 [52]
1w	2w	O	2-Thienyl	4-ClC ₆ H ₄	5.2	91	192–194	192–194 [52]
1x	2x	O	2-Furyl	4-ClC ₆ H ₄	6	80	122–124	124–125 [52]
1y	2y	O	2-Pyrrolyl	4-ClC ₆ H ₄	5	93	188–190	185–187 [52]

^a 2-Pyrazolines and isoxazolines were prepared as reported in the literature [10, 12] and characterized by elemental analysis, and on the basis of their IR, ¹H NMR, and ¹³C NMR spectra and physical data

^b Isolated yields

listed in Table 1, water–MeCN was selected as solvent and the amounts of oxidant and NH₄Cl were optimized (entry 10, Table 1). It is notable that addition of a catalytic amount of glacial acetic acid improved the yields and the reaction time. The suggested mechanism is shown in Scheme 3. The accelerating effect of HOAc is a result of formation of ClOAc, which is more active than ClOH.

Results for several 2-pyrazolines and isoxazolines are shown in Table 2. 2-Pyrazolines and isoxazolines with aromatic substituents containing electron-withdrawing groups (entries 2b, 2d, 2e, 2g, 2i, 2j, 2k, 2q, 2r, 2u, 2w, 2x, and 2y, Table 2) and electron-releasing groups (entries 2c, 2f, 2h, 2l, 2m, 2n, 2o, 2p, 2t, and 2v, Table 2) were oxidized to corresponding pyrazoles and isoxazoles, respectively. In Table 3, the results obtained

Table 3 Comparison of this method with other procedures reported for synthesis of 1,3,5-triphenyl-1*H*-pyrazole

Entry	Oxidant	Condition	Solvent	Time (h)	Yield (%)	Ref
1	This method	rt	H ₂ O–MeCN	0.4	96	–
2	I ₂ O ₅ –KBr	rt	H ₂ O	3.5	98	[53]
3	4-(<i>p</i> -Chloro)phenyl-1,3,4-triazole-3,5-dione	rt	CH ₂ Cl ₂	0.25	70	[47]
4	NHPI–Co(OAc) ₂ –O ₂	Reflux	MeCN	4	99	[48]
5	<i>N,N'</i> -Dibromo- <i>N,N'</i> -1,2-ethanedithylbis(<i>p</i> -toluenesulfonamide)	rt	CCl ₄	0.25	82	[54]
6	1,3-Dibromo-5,5-dimethylhydantoin	rt	CCl ₄	0.5	90	[34]
7	Silica sulfuric acid-activated poly-1,3-dichloro-5-methyl-5-(4'-vinylphenyl)hydantoin	rt	EtOH	1	82	[55]
8	Silica-adsorbed benzyltriphenylphosphonium peroxymonosulfate	Reflux	MeCN	5	76	[56]
9	<i>N,N'</i> - <i>N,N'</i> -Tetrabromo-benzene-1,3-disulfonylamide (TBBDA)	rt	CCl ₄	0.3	92	[57]
10	1,3-Dichloro-5,5-dimethylhydantoin (DCH)	rt	AcOH	4	79	[58]
11	Tris(4-bromophenyl)aminium hexachloroantimonate	rt	CHCl ₃	0.5	91	[59]
12	Trichloroisocyanuric acid	rt	CCl ₄	0.75	85	[60]
13	Zr(NO ₃) ₄	rt	AcOH	0.25	85	[28]
14	Pd–C	80 °C	AcOH	6.5	86	[19]
15	I ₂	rt	AcOH	3–4	86	[61]
16	Molecular oxygen promoted by activated carbon	120 °C	AcOH	2.5	91	[62]

by use of this method for 1,3,5-triphenyl-1*H*-pyrazole (Table 2, entry 1) have been compared with those for other reported methods. Clearly this method improves yields and reaction time.

Conclusions

Aqueous work-up of products is an attractive aspect of green chemistry. In this method no toxic organic solvent was used and the acetylacetone probably produced, and NH_4Cl , are soluble in water and thus easily separated. Toxic metals and other toxic materials, for example molecular iodine, not required in this procedure. No by-products were observed. Therefore, this procedure is selective, effective, rapid, inexpensive, and almost clean. Purity of products is high, work-up is easy, and complex methods of purification are not necessary. Finally, the results of this method (reaction time and yields) are better than for other reported methods.

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