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A mild and efficient H$_2$O$_2$ oxygenation of *N*-heteroaromatic compounds to the amine *N*-oxides and KI deoxygenation back to the tertiary amine with hexaphenyloxodiphosphonium triflate

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Abstract

A mild and efficient method for the oxidation of *N*-heteroaromatic compounds to the corresponding *N*-oxides using H$_2$O$_2$ in the presence of hexaphenyloxodiphosphnium triflate (Hendrickson reagent) in EtOH at room temperature was reported. This methodology presented relatively fast and selective reactions to afford the *N*-oxides in good yields. The reverse reactions, deoxygenation reactions, were also carried out under the same reaction conditions by KI to produce the tertiary amines.

Keywords Oxidation · *N*-Heteroaromatic compound · *N*-Oxide · Hydrogen peroxide · Hexaphenyloxodiphosphonium triflate

Introduction

The tertiary amine *N*-oxides are not only important as intermediates in the functionalization and structural modification of amines which are not accessible by other methods, but also serve as reagents for oxidation, protecting groups, and ligand metal complexes [1–4]. A variety of catalytic methods including O$_2$/Co(II)Schiff base complex [5], O$_2$/RuCl$_3$ [6], activated H$_2$O$_2$ such as H$_2$O$_2$/flavin [7, 8], H$_2$O$_2$/methyltrioxorhenium [9–11], H$_2$O$_2$/TS-1 [12], H$_2$O$_2$/Mg-Al layered double hydroxide-WO$_4^{2-}$ [13], H$_2$O$_2$/tungsten sulfide [14], H$_2$O$_2$/manganese tetrakis(2,6-dichlorophenyl)porphyrin [15], H$_2$O$_2$/heteropolyperoxo-tungstates [16], H$_2$O$_2$/selenium [17] and molybdenum [18] oxides, H$_2$O$_2$/V$_x$Si$_{4x}$O$_{6.4x}$ [19], as well as stoichiometric methods such as oxidation process using activated H$_2$O$_2$ [20], Caro's acid [21, 22], dioxiranes [23–25], α-azo hydroperoxides [26], peracids [27], oxaziridine [28], and magnesium monoperphthalate [29], have been reported for oxidation of tertiary amines into the corresponding tertiary amine *N*-oxides. Among these oxidants, O$_2$ and H$_2$O$_2$ are highly attractive since they are environmentally acceptable and cheap. Moreover, H$_2$O$_2$ produces no toxic waste products in contrast to many other oxidants. H$_2$O$_2$ is also easy to handle and has enough available oxygen for oxidation reactions compared to most other oxidants. In an ongoing study within our group on the application of Tf$_2$O/ H$_2$O$_2$ system on oxidation of sulfides to sulfoxides [30], we decided to investigate the applicability of hydrogen peroxide/Tf$_2$O or H$_2$O$_2$/Hendrickson reagent systems for oxidation of the amines (Scheme 1).

Deoxygenation of amine *N*-oxides to amines is an important transformation in organic synthesis, industrial systems and biological processes. Tertiary amine *N*-oxides can be reduced back to the corresponding amines. A number of methods involving metals have been used to execute this transformation [31–40]. Most of these methods suffer from harsh reaction conditions, low yields, incompatibility with other functional groups, low activity, need for high temperatures and difficult work-up procedures. Thus, the exploration of simple, mild and efficient method for deoxygenation of amine *N*-oxides to reduce back to the corresponding amines is of great interest in organic synthesis. We herein disclose that the amine *N*-oxides can be deoxygenated using Hendrickson reagent/KI system in ethanol under mild reaction conditions (Scheme 2).
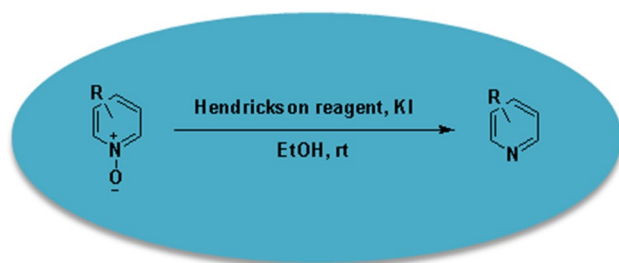
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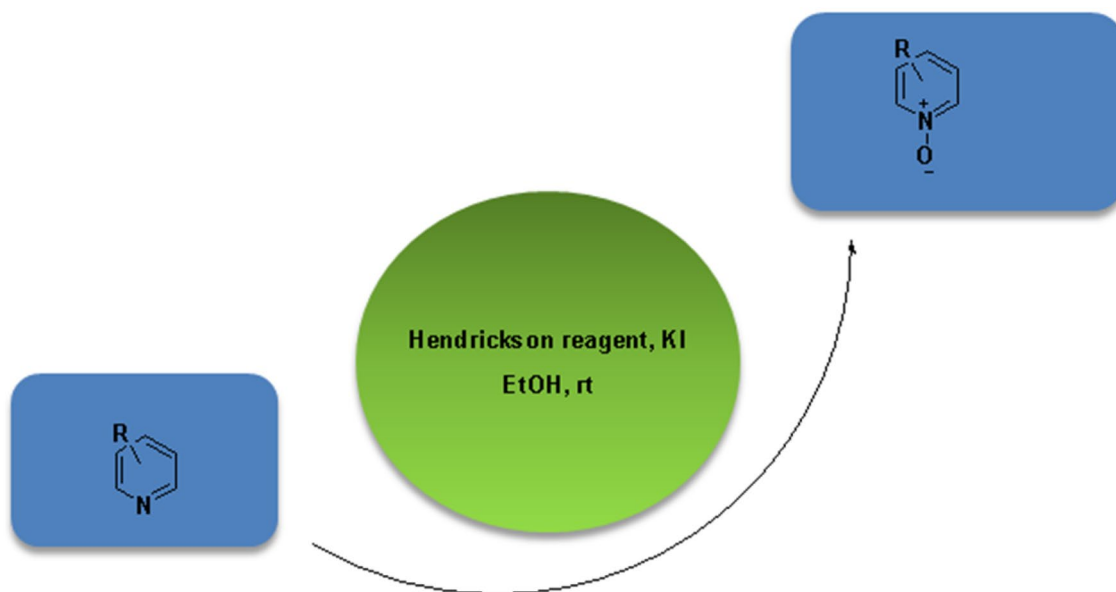
Scheme 1 Oxidation of pyridines with H_2O_2/Hendrickson reagent system

Scheme 2 Deoxygenation of pyridine-N-oxide derivatives with KI/Hendrickson reagent system

Experimental

Chemicals and materials

All the chemicals were obtained from Merck and used as received. The products are characterized by a comparison of their spectral (^{1}H NMR, ^{13}C NMR) data with those reported in the literatures. All yields refer to isolated products. NMR spectra were recorded on BruckerAV400 NMR spectrometer in DMSO-d_6 using as an internal standard.

Typical procedure for the oxidation of pyridine

In a 25-mL flask to a solution of Hendrickson reagent (1 mmol, 0.839 g) in 3 mL of ethanol at room temperature, H_2O_2 (4 mmol, 0.4 mL) was added, and the mixture allowed stirring for 15 min. Then, pyridine (1 mmol,

0.16 mL) was added to the mixture, and the solution was stirred again for appropriate time (Table 2). Upon completion of the reaction, the solvent was removed under reduced pressure, and then the saturated sodium bicarbonate solution (10 mL) was added. The product was extracted with chloroform (3×5 mL), washed with water, and dried over anhydrous $MgSO_4$. The filtrate was evaporated and the crude product was purified by silica gel column chromatography using ethyl acetate/n-hexane (3:7) as eluent to afford pyridine-N-oxide (0.076 g, 80%).

Typical procedure for the deoxygenation of pyridine-N-oxide

To a 25-mL flask containing a stirred solution of pyridine-N-oxide (1 mmol, 0.095 g) in EtOH (3 mL), Hendrickson reagent (1 mmol, 0.839 g), and KI (2 mmol, 0.336 g) were added. The mixture was stirred magnetically at room temperature, and monitored by TLC. Upon completion of the reaction, the solvent was evaporated, and then saturated sodium bicarbonate solution (10 mL) was added. The product was extracted with chloroform (3×5 mL), and dried over anhydrous $MgSO_4$. The filtrate was evaporated, and pyridine (0.059 g, 75%) was obtained as the sole product.

Selected spectra for the known products

Pyridine-1-oxide (Table 2, entry 1) (2a): colorless solid; ^{1}H NMR (400 MHZ, DMSO-d_6): δ 7.31–7.44 (m, 3H), 8.21 (d,

2H) ppm; ^{13}C NMR (100 MHZ, DMSO-d_6): δ 125.67, 127.15, 139.30 ppm.

4-Methylpyridine-1-oxide (Table 2, entry 2) (2b): orange solid; ^{1}H NMR (400 MHZ, DMSO-d_6): δ 2.32 (s, 3H, CH$_3$), 7.99 (d, 2H), 8.73 (d, 2H) ppm; ^{13}C NMR (100 MHZ, DMSO-d_6): δ 18.12, 125.15, 134.55, 147.30 ppm.

3-Methylpyridine-1-oxide (Table 2, entry 4) (2d): colorless oil; ^{1}H NMR (400 MHZ, DMSO-d_6): δ 2.22 (s, 3H, CH$_3$), 7.27 (dd, 1H), 8.04 (d, 1H), 8.07–8.13 (m, 2H) ppm; ^{13}C NMR (100 MHZ, DMSO-d_6): δ 17.99, 126.32, 126.77, 136.51, 137.34, 139.01 ppm.

2,4,6-Trimethylpyridine-1-oxide (Table 2, entry 5) (2e): brown solid; ^{1}H NMR (400 MHZ, DMSO-d_6): δ 2.27 (s, 9H, 3CH$_3$), 7.22 (s, 1H), 8.09 (s, 1H) ; ^{13}C NMR (100 MHZ, DMSO-d_6): δ 18.66, 19.94, 127.50, 136.36, 138.54 ppm.

4-*N,N*-Dimethylaminopyridine-1-oxide (Table 2, entry 6) (2f): yellow solid; ^{1}H NMR (400 MHZ, DMSO-d_6): δ 2.95 (s, 6, 2CH$_3$), 6.58 (d, 2H), 8.10 (d, 2H) ppm; ^{13}C NMR (100 MHZ, DMSO-d_6): δ 40.57, 107.17, 149.73, 154.42 ppm.

2,4-Dimethylpyridine-1-oxide (Table 2, entry 7) (2 g): brown solid; ^{1}H NMR (400 MHZ, DMSO-d_6): δ 2.26 (s, 6H, 2CH$_3$), 6.98 (d, 1H), 7.14 (s, 1H) 7.63 (d, 1H) ppm; ^{13}C NMR (100 MHZ, DMSO-d_6): δ 20.90, 21.58, 127.01, 128.75, 134.10, 143.92, 145.37 ppm.

Quinoline-1-oxide (Table 2, entry 8) (2 h): brown solid; ^{1}H NMR (400 MHZ, DMSO-d_6): δ 7.45 (d, 1H), 7.48–7.73 (m, 2H), 7.83 (d, 1H), 8.10 (d, 1H), 8.32–8.58 (m, 2H) ppm; ^{13}C NMR (100 MHZ, DMSO-d6): δ 119.32, 122.39, 125.60, 129.11, 129.21, 130.78, 135.74, 141.29 ppm.

Quinolin-8-ol-1-oxide (Table 2, entry 9) (2i): brown solid; ^{1}H NMR (400 MHZ, DMSO-d_6): δ 3.33 (s, 1H, OH), 6.98 (d, 1H), 7.08–8.32 (m, 3H), 8.52 (d, 1H), 8.84 (d, 1H) ppm; ^{13}C NMR (100 MHZ, DMSO-d_6): δ 111.77, 114.45, 117.52, 122.20, 132.53, 135.84, 136.58, 148.56, 153.79 ppm.

2-Methylquinoline-1-oxide (Table 2, entry 10) (2j): brown solid; ^{1}H NMR (400 MHZ, DMSO-d_6): δ 2.56 (s, 3H, CH$_3$), 7.65 (d, 1H), 7.75 (t, 1H), 7.78–7.81 (m, 2H), 7.82 (d, 1H), 7.99 (t, 1H); ^{13}C NMR (100 MHZ, DMSO-d_6): δ 18.66, 119.12, 123.91, 124.43, 128.13, 128.93, 129.45, 130.51, 141.25, 145.34 ppm. Elemental analysis: calcd (%) for C$_{10}$H$_9$NO: C, 75.47; H, 5.66; N, 8.80; found: C, 75.55; H, 5.72; N, 8.63.

[2,2′-Bipyridine]-1,1′-dioxide (Table 2, entry 11) (2 k): violet solid; ^{1}H NMR (400 MHZ, DMSO-d_6): δ 7.45–7.90 (m, 3H), 7.96 (t, 1H), 8.01 (d, 1H), 8.10 (d, 1H), 8.74 (t, 2H) ppm; ^{13}C NMR (100 MHZ, DMSO-d_6): δ 124.87, 125.36, 125.81, 126.65, 127.96, 136.72, 140.95, 146.60, 149.89, 150.02 ppm.

Results and discussion

Hexaphenyloxodiphosphonium triflate, known as Hendrickson reagent, was prepared by exothermic reaction of Tf$_2$O with Ph$_3$PO in CH$_2$Cl$_2$ [41–43]. This salt was indicated to

be powerful dehydrating agent and promoter, and promising reagent for some organic reactions [44–53]. At the beginning of these investigations, pyridine (1a) was selected as the model substrate to establish the oxygenation reaction with H$_2$O$_2$ in the presence of Tf$_2$O as a promoter in EtOH at room temperature. It was found that the reaction failed to proceed. Thus, Hendrickson reagent was used as a promoter under the same reaction conditions, and it was found that pyridine-N-oxide (2a) was obtained in 80% yield, and confirmed by its ^{1}H NMR spectrum (Table 1, entry 1). The effect of other solvents such as methylene chloride, chloroform, n-hexane, acetonitrile, ethyl acetate, acetone and water was examined under the same reaction conditions and the desired product was obtained in lower yields (0–35%) (Table 1, entries 4–10).

Chloroform, dichloromethane and *n*-hexane as solvents appeared to afford the worst results due to their low ability to solve the promoter reagent affecting the reaction. The optimum conditions for the amounts of Hendrickson reagent and H$_2$O$_2$ were 1 equi, and 4 equiv, respectively. Using less than 1 equiv of the reagent or 4 equiv of H$_2$O$_2$ in the reaction led to an incomplete reaction. Application of 2 equiv of Hendrickson reagent gave no significant increment based on the yield of the product. It was found that temperature increasing of the reaction shows no significant improvement in respect to the yield. Therefore, it seems that the optimum conditions for the reaction are pyridine (1 equiv), H$_2$O$_2$ (4 equiv) and Hendrickson reagent (1 equiv) in ethanol (3 mL) at room temperature. In the absence of promoter, after 48 h,

Table 1 Effect of solvent, amount of promoter and H$_2$O$_2$ on synthesis of pyridine-N-oxide

Entry	Solvent	Promoter (mmol)	H$_2$O$_2$ (mmol)	Time (min)	Yield (%)[a]
1	EtOH	1	4	70	80
2	EtOH	0.5	4	100	50
3	EtOH	2	4	70	80
4	EtOH	1	2	110	67
5	EtOH	1	6	65	80
6	H$_2$O	1	4	120	35
7	CH$_2$Cl$_2$	1	4	120	0
8	MeCN	1	4	120	20
9	CHCl$_3$	1	4	120	0
10	*n*-Hexane	1	4	120	0
11	Ethylacetate	1	4	120	35
12	Acetone	1	4	120	30

Pyridine (1 mmol), H$_2$O$_2$ (4 mmol), Hendrickson reagent (1 mmol) in EtOH at room temperature

[a]Isolated yield
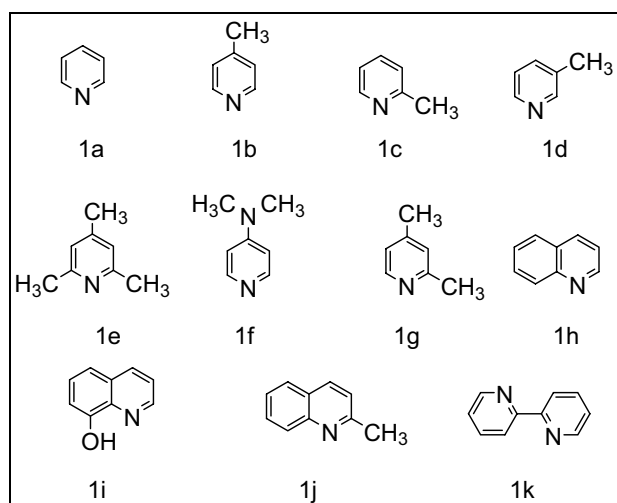
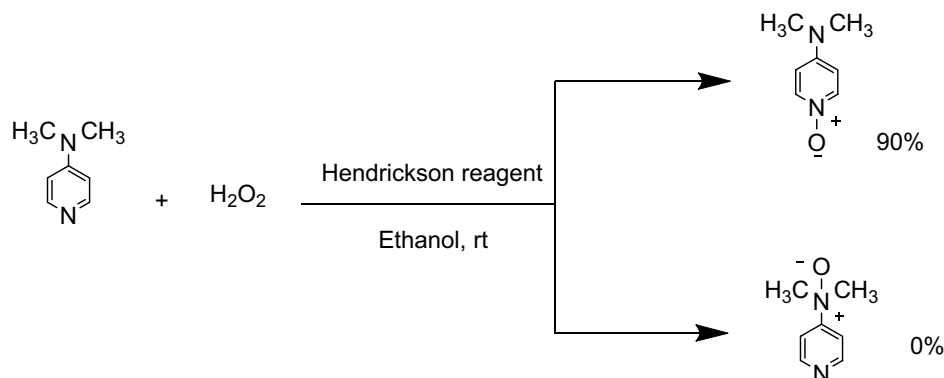**Scheme 3** Scope of *N*-heteroaromatic compounds

Table 2 Oxidation of *N*-heteroaromatic compounds to the corresponding *N*-oxides using H_2O_2/Hendrickson reagent system

Entry	Product	Time (min)	Yield (%)[a]
1	2a	70	80
2	2b	60	92
3	2c	60	95
4	2d	60	88
5	2e	50	98
6	2f	45	90
7	2g	70	82
8	2h	60	90
9	2i	50	95
10	2j	60	90
11	2k	70	85

[a]Isolated yield

the product efficiency was only 10%. No product was also obtained when H_2O_2 was not present in the reaction.

Having confirmed the key role of Hendrickson reagent in promoting the H_2O_2-mediated oxygenation of the amines, we examined the scope and generality of the reaction using various *N*-heteroaromatic compounds (**1a**–**1k**, Scheme 3; Table 2). The pyridines with electron-releasing groups reacted with shorter reaction times and good yields, while oxidation of pyridines with electron-withdrawing groups such as 4-pyridine carboxaldehyde and pyridine-3-carboxylic acid was not successful. The oxidation of 2,4,6-trimethylpyridine (**1e**) was carried out in 98% yield (Table 2, entry 5). The oxidation of 2-methyl pyridine (**1c**), 3-methyl pyridine (**1d**) and 4-methyl pyridine (**1b**) under the present reaction conditions occurred in 95, 88, and 92% yields, respectively (Table 2, entries 3, 4, and 2).

The oxidation of 4-*N,N*-dimethylaminopyridine was chemoselectively led to 4-*N,N*-dimethylaminopyridine-1-oxide in 70% yield and no pyridyldimethyl amine *N*-oxide obtained as indicated in Scheme 4. It seems that lone pair electrons on nitrogen of alkyl amine with SP^3 hybridization are less available for oxygen due to steric effect compared with nitrogen of pyridine which has SP^2 hybridization.

In addition, the oxidation of quinoline (**1g**), 8-hydroxyquinoline (**1i**) and 2-methylquinoline (**1j**) with H_2O_2 occurred in 82, 95, and 90% yields, respectively (Table 2, entries 7, 9, and 10). Bipyridine oxidation was carried out and its both nitrogens were oxidized (Scheme 5).

The reaction was found to be general for production of most heteroaromatic *N*-oxides but not for aliphatic amine *N*-oxides under the same reaction conditions. For example, our attempts to oxidize triethylamine under the same reaction conditions failed, and apparently the oxidizing system was not strong enough to oxidize these amines.

To show the merit of this method, the results of this oxygenation reaction by H_2O_2/Hendrickson reagent system were compared with the other reported results in the literature for pyridine (Table 3). The results show that this method is comparable to the other methods in terms of reaction time, temperature and yield.

Scheme 4 Oxidation of 4-dimethylaminopyridine

Scheme 5 Oxidation of bipyridine

Table 3 Comparison of pyridine-N-oxide formation under different conditions

Entry	Reagents	Temp (°C)	Time	Yield (%)	References
2	Lipase/glucose	rt	1 h	97.6	[54]
3	1,2-Diphenyl-1,1,2,2-tetrahydroperoxyethane	rt	3 min	98	[55]
3	Tungsten-loaded TiO_2/H_2O_2	rt	18 h	92	[56]
4	$V_xSi_{4x}O_{6.4x}$/H_2O_2	80	10 h	79	[19]
5	Hendrickson reagent/H_2O_2	rt	70 min	80	This work

Furthermore, we found that pyridine-N-oxide was successfully deoxygenated and converted to pyridine using Hendrickson reagent/KI system. In this case, we consider set of experiments to optimize the reaction conditions. Therefore, the reactions of pyridine-N-oxide with different amounts of the above system at room temperature using several solvents were examined. The results show that the optimum conditions are pyridine-*N*-oxide (1 mmol), KI (2 mmol), Hendrickson reagent (1 mmol) in EtOH (3 mL) at room temperature and pyridine obtained in 75% yield (Table 4).

The reactions of pyridine-N-oxide derivatives under the same reaction conditions were also studied and the corresponding pyridines were obtained as the products (Table 5).

As a result of the observations, the following mechanism indicated in Scheme 6 was proposed for H_2O_2 oxidation of pyridine promoted by Hendrickson reagent. The **A** intermediate was prepared via a nucleophilic substitution reaction between the promoter and H_2O_2 as a nucleophile. Replacement of triphenylphosphine oxide with the oxygen of the H_2O_2 generates intermediate **A**. This intermediate was used for successful oxidation of broad range of pyridine derivatives via an ordinary nucleophilic substitution reaction.

The intermediate **A** was isolated and confirmed by its NMR spectra. The peak of proton at 10.29 ppm observed as a singlet in the ^{1}H NMR spectrum ascribed to OH group of H_2O_2. The peaks at 7.25–8.83 ppm related to aromatic C–H

Table 4 Effect of solvent, amount of promoter, and KI on deoxygenation of pyridine-N-oxide

Entry	Solvent	Promoter (mmol)	KI (mmol)	Time (min)	Yield (%)[a]
1	EtOH	1	2	95	75
2	EtOH	0.5	2	100	44
3	EtOH	2	2	90	75
4	EtOH	1	1	120	46
5	EtOH	1	4	95	75
6	H_2O	1	2	120	25
7	CH_2Cl_2	1	2	120	0
8	MeCN	1	2	120	30
9	$CHCl_3$	1	2	120	0
10	*n*-Hexane	1	2	120	0
11	Ethylacetate	1	2	120	10

Pyridine-*N*-oxide (1 mmol), KI (2 mmol), Hendrickson reagent (1 mmol) in EtOH at room temperature

[a]Isolated yield

bonds. The ^{13}C NMR spectrum of intermediate **A** shows the presence of aromatic rings by the peaks of carbons at 126–127 ppm. These data indicate that the intermediate **A** was produced during the reaction. The presence of intermediate **A** might be confirmed by the observation peak at 3420 cm^{-1} in FT-IR related to the stretching vibrations of

Table 5 Deoxygenation of pyridine-N-oxide derivatives to the corresponding pyridines with KI/Hendrickson reagent system

| Entry	Product	Time (min)	Yield[a] (%)
1	1a	95	75
2	1b	70	89
3	1c	65	90
4	1d	90	80
5	1e	65	90
6	1f	60	95
7	1g	70	93
8	1h	75	85
9	1i	70	90
10	1j	90	85
11	1k	90	88

[a]Isolated yield

hydroxyl group. This peak in FT-IR spectrum of Hendrickson reagent was not observed (supplementary material).

Also, a possible mechanism shown in Scheme 7 for KI deoxygenation of pyridine-N-oxide, is promoted by Hendrickson reagent. Herein, this reagent is attacked by a nucleophilic oxygen of pyridine-N-oxide, and as the result, the triphenylphosphine oxide leaving group replaced with the oxygen of pyridine-N-oxide and produced intermediate **B**. Then iodide anion attacks to intermediate **B**, and the resultant iodinated species is in turn attacked by another iodide anion to give the deoxygenated pyridine.

Conclusions

In summary, Hendrickson reagent can develop mild and efficiently the oxygenation of *N*-heteroaromatic compounds into the tertiary amine *N*-oxides using H_2O_2 in EtOH at room temperature. The oxidation of 4-dimethylaminopyridine was chemoselectively led to 4-dimethylaminopyridine-*N*-oxide in 70% yield and no pyridyldimethyl amine *N*-oxide obtained. Furthermore, KI deoxygenation of tertiary amine
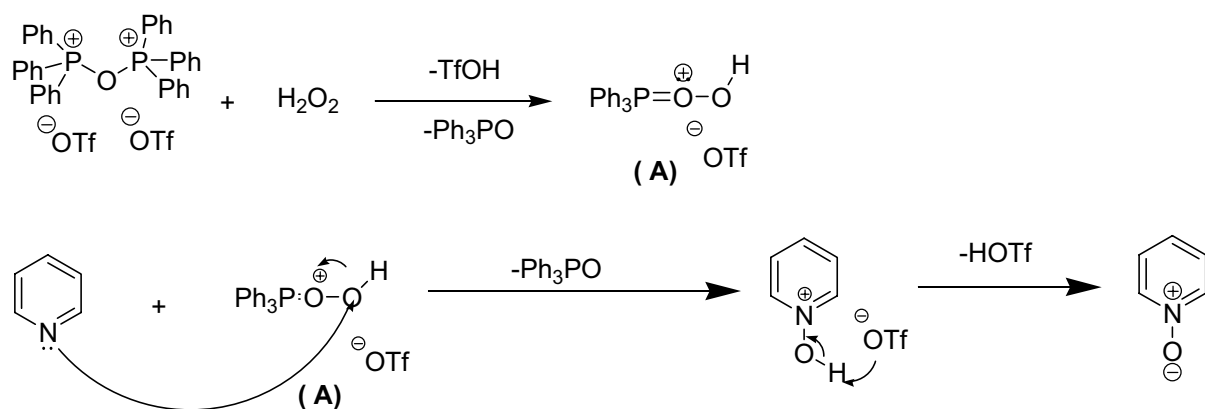
Scheme 6 Plausible mechanism for the oxygenation of pyridine

Scheme 7 Plausible mechanism for the deoxygenation of pyridine-N-oxide
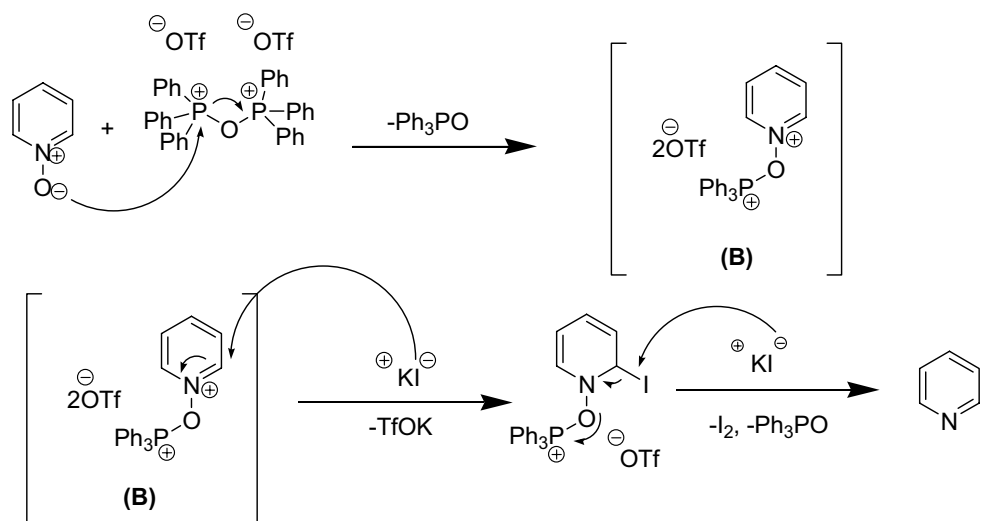
N-oxides into the corresponding *N*-heteroaromatic compounds was also efficient and conveniently promoted by this reagent in ethanol under mild reaction conditions. The results obtained in this study were comparable with previous reports in respect to overall efficiency.

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