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ARTICLE

Highly efficient and stable peracid for rapid and selective oxidation of aliphatic amine to oxime

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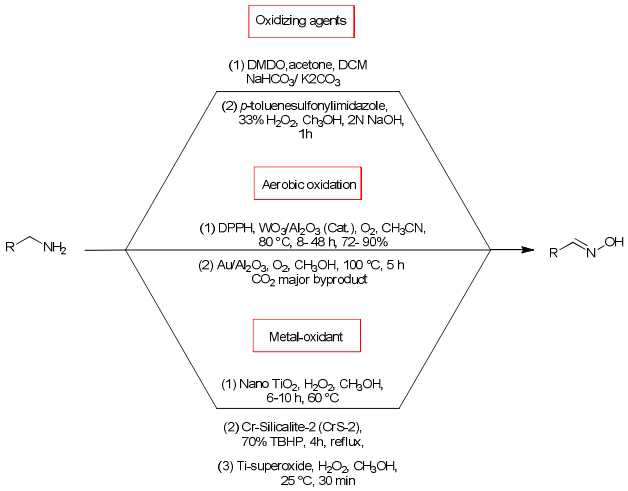
A novel, transition-metal free, rapid approach for selective oxidation of aliphatic and benzylic amine to oxime is described. The dodecanebis(peroxoic acid)-DMF combination efficiently oxidize various aliphatic amines at 50 °C temperature to give 100% conversion in 20 min with high oxime selectivity. The peroxy acid used here show exceptional stability at room temperature and non-shock sensitive in nature, which was confirmed by differential scanning colorimetric (DSC) analysis.

Keywords: Aliphatic amine, Benzylic amine, Dodecanebis(peroxic acid), Oxidation, Oxime

Introduction

Selective oxidation of aliphatic amines to oxime is very important but still challenging task in organic synthesis due to sensitive nature of aliphatic amines. Oximes are well recognized intermediates for the synthesis of polymers, medicines, heterocycles and fine chemicals. It can be easily transferred into various important derivatives such as amide,[1–3] nitrile,[4–6] nitro compounds,[7,8] nitrone[9] and also used for the synthesis of azoheterocycles.[10,11] Various oxidizing systems have been employed for oxidation of aliphatic amines which utilizes oxidants such as dimethyldioxirane,[12–14] sodium perborate[15] and sulfonic peracid,[16] oxidant-metal systems such as H_2O_2-nano TiO_2,[17] H_2O_2-titanium superoxide,[18] H_2O_2.oxidoperoxidotungsten (VI) complexes,[19] H_2O_2-SeO_2,[20] H_2O_2-with various Mo catalysts,[21–23] H_2O_2 –titanium silicate (TS-1),[24,25] and TBHP-chromium silicate[26]. Though in many cases protocol offered good oxime selectivity, reaction requires excess quantity of oxidant with longer time for completion and often the reaction result in to poor conversion. Emmon[27] has reported formation of oxime when oxidation of aliphatic amines was performed using anhydrous Peracetic acid. The prototrophic rearrangement of nitroso alkane intermediates results into formation of oxime which reduces the yield of nitro compound. Whereas Gilbert et. Al.[28] have reported that such rearrangement was not observed when they studied similar reaction using *m*-CPBA in various halogenated solvents.

On other hand the use of molecular oxygen as a source of oxidant in the presence of various transition metal catalysts such as gold-titania,[29] DPPH and WO_3/Al_2O_3[30,31], $InCl_3$ with TEMPO and acetaldoxime[32] offers various advantages such as inexpensive source of oxidant, minimum waste generation, reusability of catalyst. Though this aerobic oxidation is greener route for the oxime synthesis, it suffers various disadvantages such as, it involves costly transition metal complexes, reaction need to carry out at high temperature (up to 100- 120 °C) for 10 to 16 h under oxygen atmosphere. This system need further improvements in order to reduce the reaction time as well as the cost of catalyst employed. Thus, finding the scope for improvement, we focused our attention to develop an efficient protocol for oxidation of aliphatic amines which will give high selectivity in shorter time with high yield. As a continuation of our studies on exploration of effectiveness of long chain diperoxy acids in oxidation reactions,[33] here we have demonstrated the study of another diperoxy acid, dodecanebis(peroxoic acid) for the selective oxidation of aliphatic amine to oxime. It is solid in nature, stable at room temperature, easy to handle and possesses very good oxidation capacity. We have observed that these diperoxy acids are selective in action, work under mild conditions with easy separation of desired products.



Scheme 1. Previous reports.

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In the present work, we have developed a novel, transition metal free, rapid and efficient route for selective oxidation of aliphatic amines to oxime using dodecanebis(peroxoic acid). The protocol developed was found to be effective for benzylic, alicyclic as well as acyclic amines to give oxime in higher yield and shorter period compared to previously reported methods (**scheme 1**)

Result and Discussion

Initially we performed the shock sensitivity and stability study of this peroxy acid as most of the commonly used per acids are shock sensitive and degrade at room temperature.[34] The non-shock sensitive nature of dodecanebis(peroxoic acid) was confirmed by Differential Scanning Colorimetric (DSC) analysis.

The shock sensitivity and explosion propagation calculation was done by using Yoshida's correlation equation.[35] According to Yoshida's correlation equation "if the value for Shock sensitivity (SS) or Explosion Propagation (EP) is ≥ 0.00, then the material is predicated to be shock sensitive or demonstrate explosive propagating properties, respectively." The value of energy of exotherm (Qdsc = 1478 j/g = 0.0003530 calories/g) and onset temperature of exotherm (Tdsc = 100.61 °C) was obtained from the DSC graph (Figure 1 supporting data). These values were substituted in the Yoshida's equation which gives value for Shock Sensitivity (SS) -3.7490 and Explosion Propagation (EP) -0.7993 (Figure and detail calculations are given in the supporting data).

After shock sensitivity analysis, we examined stability of dodecanebis(peroxoic acid) by keeping one sample of this diperoxy acid at room temperature in the self-sealing bag. The active oxygen content (AOC) of this sample was checked after every 15 days by Iodometric titration. The result obtained are summarized in the table 1.

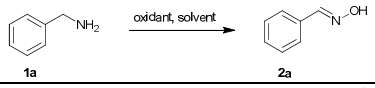
Table 1. Stability study of dodecanebis(peroxoic acid) at room temperature

Entry	Days	% AOC
1	Initial	11.20
2	After 15 days	11.18
3	After 30 days	11.20
4	After 45 days	11.17

From AOC data, it was clear that, dodecanebis(peroxoic acid) retain its active oxygen content till 45 days at room temperature. Thus from above studies, it was clear that, dodecanebis(peroxoic acid) is non-shock sensitive in nature and stable at room temperature.

We initiated our work with benzyl amine 1a as the probe substrate and dodecanebis(peroxoic acid) as an oxidant at 50 °C From the molar ratio study of dodecanebis(peroxoic acid) it was clear that, 1 equiv. of oxidant was sufficient to give maximum oxime selectivity of 90% (Table 2, entry 10). (Table 2). Different solvents were studied to screen the preferred solvent which can offer high oxime selectivity (Table 2, entries 1-10). It was found that, amongst the solvent studied DMF offered desired product 2a in 94% yield with 90% oxime selectivity with 6% benzaldehyde and 4% benzonitrile in 20 min (Table 2, entry 10). Whereas in all other solvents though conversion was 100% but oxime selectivity was observed in the 56-79% range (Table 2 entries 1-9). It was also observed that, oxime selectivity increases with temperature. As we increased temperature from room temperature (30 °C) to 50 °C oxime selectivity was also increased from 53% to 90% (Table 2 entries 10-12). Further rise in the temperature does not show any influence on the oxime selectivity (Table 2, entry 13).

Table 2. Optimization of reaction parameters[a]



1a 2a

Entry	Parameters	Oxidant equiv.	Oxime selectivity[b] (%)	Isolated yield (%)
Solvent Study				
1	Toluene	1	56	48
2	Acetone	1	70	62
3	Ethyl acetate	1	79	72
4	Dichloromethane	1	70	68
5	Chloroform	1	74	71
6	Acetonitrile	1	76	66
7	Ethanol	1	59	51
8	Methanol	1	62	56
9	Water	1	64	52
10	DMF	1	90	94
Temperature study				
11[c]	DMF	1	53	51
12[d]	DMF	1	78	73
13[e]	DMF	1	91	92
Oxidant molar ration study				
14[f]	DMF	0.75	76	80
15[g]	DMF	1.25	91	93
Comparison with other oxidant				
16	50% H_2O_2	2	32	41
17	Oxone	2	44	53
18	70% TBHP	2	38	29
19	*m*-CPBA	2	43	55
20	Sodium perborate	2	45	36
21	Potasium peroxydisulphate	2	30	23
22	Hexanebis(peroxoic acid)	1	84	88
23	Nonanebis(peroxoic acid)	1	89	90
24	No oxidant	-	No reaction	-

[a]Reaction condition: **1a** (1 equiv. 0.93 mmol); Dodecanebis(peroxoic acid) (1 equiv., 0.93 mmol); solvent (4 mL); Temperature 50-55 °C; Time 20 min.[b]Conversion determined by GC using internal standard; [c]Reaction kept at room temperature for 3 h. [d]Reaction kept at 40 °C for 3 h; [e]Reaction kept at 60 °C for 30 min.; [f]Dodecanebis(peroxoic acid) 0.75 equiv. reaction kept for 3 h; [g]Dodecanebis(peroxoic acid) 1.25 equiv. reaction kept for 20 min.

Reduction in oxidants molar ratio (0.75) reduces the oxime selectivity to 76% (Table 2, entry 14), on the other hand, increasing the oxidant molar ratio to 1.25 did not show any

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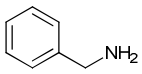
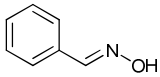
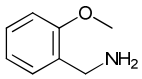
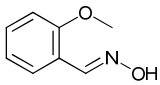
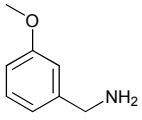
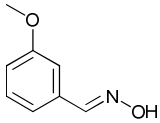
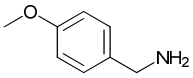
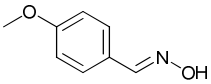
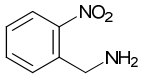
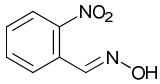
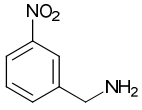
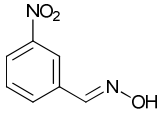
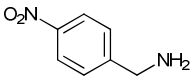
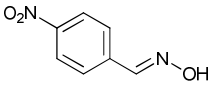
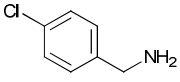
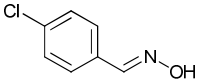
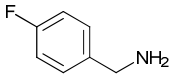
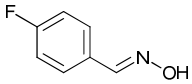
enhancement in the oxime selectivity (Table 2, entry 15). When we performed oxidation of benzyl amine with other commonly used oxidizing agents under optimized conditions, poor oxime selectivity was observed (Table 2, entries 16-21). On other hand, oxidation using other two diperoxy acids such as hexanebis(peroxoic acid) and nonanebis(peroxoic acid) also demonstrated good selectivity towards oxime formation (Table 2, entries 22-23). No reaction occurred in absence of oxidizing agent (Table 2, entry 24).

With the optimized conditions in hand, we sought to explore the substrate scope. The results of the oxidation of various benzyl amine derivatives by dodecanebis(peroxoic acid) in DMF are summarized in Table 3. It was observed that a variety of benzyl amines can be converted into desired oxime in good yields. The reaction time and yield of the desired product was not found to be dependent of the substituent present on the amine functionality. The present protocol can tolerate various electron donating groups such as $–OCH_3$, $-C(CH_3)_3$, $-OCH_2O-$,3,4-dimethoxy (Table 3, entries 2-4, 11, 13, 15) as well as electron withdrawing groups such as $–NO_2$, -CN, -X (-Cl, -F) (Table 3, entries 5-8, 9, 10).

Table 3 Substrate scope[a]

Entry	Substrate	Product	Isolated yield (%)	M.P./B.P. (°C) Observed	Reported
1	 1a	 2a	94	35-36[b]	35[36]
2	 1b	 2b	92	87-88[b]	88-90[37]
3	 1c	 2c	89	40-41[b]	39-40[38]
4	 1d	 2d	91	62-64[b]	63-66[39]
5	 1e	 2e	88	96-98[b]	97[40]
6	 1f	 2f	90	121-122[b]	121[40]
7	 1g	 2g	86	125-127[b]	126-128[41]
8	 1h	 2h	86	105-106[b]	106-108[42]
9	 1i	 2i	90	85-87[b]	85-86[43]

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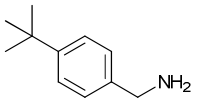
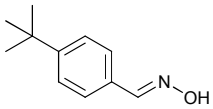
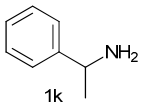
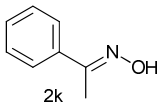
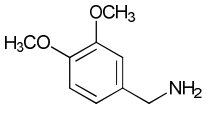
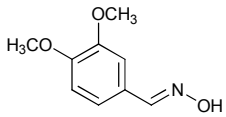
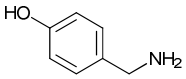
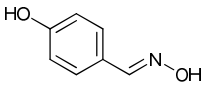
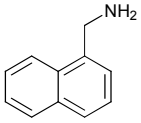
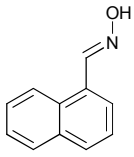
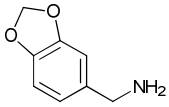
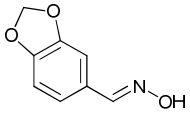
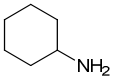
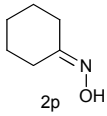
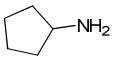
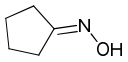
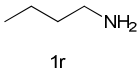
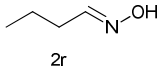
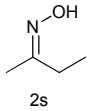
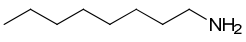
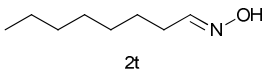
10	 1j	 2j	88	105-107[b]	-
11	 1k	 2k	85	58-60[b]	58-60[44]
12	 1l	 2l	90	92-94[b]	94-95[45]
13	 1m	 2m	87	92-94[b]	92-93[46]
14	 1n	 2n	84	95-97[b]	96[47]
15	 1o	 2o	90	105-107[b]	106-108[48]
16	 1p	 2p	91	87-88[b]	87-89[49]
17	 1q	 2q	92	55-56[b]	55-57[49]
18	 1r	 2r	82	150-152[c]	152[50]
19	 1s	 2s	80	151-153[c]	150-152[51]
20	 1t	 2t	82	58-60[b]	60[36]

[a]Reaction conditions: **1** (1 equiv.); Dodecanebis(peroxoic acid) (1 equiv.), DMF 5 mL; Temperature 50-55 °C, Time 20 min. [b]Melting Point, [c]Boiling Point.

Further we applied optimized reaction for oxidation of alicyclic and alkyl amine derivatives. The oxidation smoothly proceeded to give corresponding oxime in good yield (Table 3, entries 16-20). Typically maximum yield was obtained for cyclohexyl and cyclopentyl amine (Table 3, entries 16, 17) as compared to acyclic aliphatic amines.

Conclusion

In summary, we have successfully demonstrated that the oxidant, dodecanebis(peroxoic acid) effectively oxidizes various benzylic and aliphatic amines into oxime with good yield in significantly less time. The use of mild conditions, easy

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handling of oxidant, notably in less quantity than previously described reports and broad substrate scope have made the protocol useful for organic synthesis. Further studies for the utilization of dodecanebis(peroxoic acid) in various oxidation reactions is underway in our group.

Experimental

Chemicals and Instruments

Common reagent grade chemicals were purchased from Spectrochem, Sigma Aldrich and Sd fine chemicals. N,N-dimethylformamide used was AR grade "DRY" DMF purchased from Sd fine chemicals. All melting points are uncorrected and are presented in degree Celsius. The ^{1}H NMR spectroscopic data were recorded with Agilent 500 MHz, Bruker 400 MHz, spectrometer with $CDCl_3$ and DMSO-d_6 as a solvent and chemical shifts are expressed in δ ppm using TMS as an internal standard. GC analysis were carried out with Thermo scientific, column-TR-1, 30mX0.25mm, IDX0.25um film, FID detector and sample size 0.1 l µl. GC/MS was performed on a GCMS-QP 2010 instrument. The DSC analysis was done using a DSC Q100 V9.9 Build 303 (Universal V4.5A TA Instrument), ramp 10.00 °C/min to 300.00 °C, flow rate: 50.0 mL/min.

Preparation of the dodecanebis(peroxoic acid)[52]

In 250 mL three neak round bottom flask equipped with mercury sealed stirrer, 40 g 95% sulphuric acid was placed to which 10 g of dodecanedioic acid was added under continuous stirring at room temperature till it get completely dissolved. The reaction mass was cooled to 15 °C using ice water mixture. To this, 9.2 g 65% hydrogen peroxide was drop wise added under continuous stirring in 40 to 45 min with maintaining the temperature between 15 to 20 °C. The reaction mass was stirred for further 5 h at 15 to 20 °C. The reaction mass was cooled to 0 °C and 50 mL half saturated aqueous ammonium sulphate (35 g/ 100 mL water) was added. The white slurry obtained was stirred for further 30 min and then filtered and washed with cold half saturated ammonium sulphate solution. The white colour solid mass obtained was dried under vacuum at room temperature to give dry white solid powder (11.7 g, 85%). The active oxygen content of final product was determined by Iodometric titration (Reported 11.30%, Obtained 11.20%).

General procedure for the oxidation of benzylamine

In 50 mL two neak round bottom flask , 5 mL DMF was placed to which 100 µl of benzylamine (0.933 mmol, 1 equiv.) was added at room temperature. The above mixture was stirred for 5 min. To this 240 mg of dodecanebis(peroxoic acid) (0.933 mmol, 1 equiv.) was added in 2 min with constant stirring. The reaction mass was heated to 50-55 °C. The progress of reaction was monitored by TLC. After completion, the reaction was quenched by adding 5 mL saturated sodium bicarbonate solution and stirred for 10 min. Further 15 mL water and 10 mL ethyl acetate was added and product was extracted in ethyl acetate. The aqueous layer was further extracted in ethyl acetate (3 X 5 mL). All organic layers were combined and washed with water. The organic layer was dried over anhydrous sodium sulphate and concentrated to give crude product which was further purified by flash chromatography using hexane: ethyl acetate system.

Spectral data for the representative compounds:

1. **Benzaldehyde oxime (2a):** white solid (0.106 g, 94%); ^{1}H-NMR (500 MHz, $CDCl_3$): δ 8.78 (s, 1H), 8.19 (s, 1H), 7.61-7.59 (m, 2H), 7.42-7.43 (m, 3H); ESI-MS m/z 121$[M]^+$.

2. **2-Methoxybenzaldehyde oxime (2b):** white solid (0.101 g, 92%); ^{1}H-NMR (400 MHz, DMSO): δ 11.19 (s, 1H), 8.29 (s, 1H), 7.65 (dd, *J* = 7.7, 1.4 Hz, 1H), 7.41-7.31 (m, 1H), 7.06 (d, *J* = 8.4 Hz, 1H), 6.95 (t, *J* = 7.5 Hz, 1H), 3.82 (s, 3H); ESI-MS m/z 151 $[M]^+$.

3. **3-Methoxybenzaldehyde oxime (2c):** white solid (0.098 g, 89%); ^{1}H-NMR (400 MHz, DMSO): δ 11.16 (s, 1H), 8.10 (s, 1H), 7.25 (t, *J* = 7.5 Hz, 1H), 7.13 (d, *J* = 8.6 Hz, 2H), 6.87 (d, *J* = 7.5 Hz, 1H), 3.73 (s, 3H); ESI-MS m/z 151 $[M]^+$.

4. **4-Methoxybenzaldehyde oxime (2d):** white solid (0.10 g, 91%); ^{1}H-NMR (400 MHz, DMSO): δ 10.93 (s, 1H), 8.05 (s, 1H), 7.50 (d, *J* = 8.8 Hz, 2H), 6.93 (d, *J* = 8.8 Hz, 2H), 3.75 (s, 3H); ESI-MS m/z 151 $[M]^+$.

5. **3-Nitrobenzaldehyde oxime (2f):** white solid (0.098 g, 90%); ^{1}H-NMR (500 MHz, $CDCl_3$): δ 8.451-8.444 (t, *J* = 2, 1H), 8.256-8.233 (dd, *J* = 8.5, 2.5, 1H), 8.215 (s, 1H), 7.926-7.913 (dd, *J* = 8.0, 1.5, 1H), 7.736 (s, 1H), 7.603-7.572 (t, *J* = 8, 7.5, 1H); ESI-MS m/z 166 $[M]^+$.

6. **4-Nitrobenzaldehyde oxime (2g):** light yellow solid (0.094 g, 86%); ^{1}H-NMR (500 MHz, DMSO): δ 11.84 (s, 1H), 8.31 (s, 1H), 8.28 - 8.23 (m, 2H), 7.88 - 7.83 (m, 2H); ESI-MS m/z 166 $[M]^+$.

7. **4-Chlorobenzaldehyde oxime (2h):** white solid (0.094 g, 86%); ^{1}H-NMR (500 MHz, $CDCl_3$): δ 8.12 (s, 1H), 8.03 (s, 1H), 7.534-7.525 (d, *J* = 8.4, 2H), 7.385-7.359 (d, *J* = 8.55, 2H); ESI-MS m/z 155 $[M]^+$.

8. **4-Fluorobenzaldehyde oxime (2i):** white solid (0.10 g, 90%); ^{1}H-NMR (500 MHz, $CDCl_3$): δ 8.127 (s, 1H), 7.856 (s, 1H), 7.589-7.560 (dd, *J* = 5.4, 5.5, 2H), 7.106 – 7.072 (t, *J* = 8.7, 8.65, 2H); ESI-MS m/z 139 $[M]^+$.

9. **Acetophenone oxime (2K):** white solid (0.095 g, 85%); ^{1}H-NMR (500 MHz, $CDCl_3$): δ 8.984 (s, 1H), 7.650-7.630 (q, 2H); 7.408-7.393 (q, 3H), 2.319 (s, 3H); ESI-MS m/z 135 $[M]^+$.

10. **4-Hydroxybenzaldehyde oxime (2m):** white solid (0.097 g, 87%); ^{1}H-NMR (500 MHz, DMSO): δ 10.824 (s, 1),

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9.737 (s, 1H), 7.986 (s, 1H), 7.396-7.379 (d, J = 8.55, 2H), 6.765-6.748 (d, J = 8.55, 2H); ESI-MS m/z 137 $[M]^+$.

11. 1-Naphthaldehyde oxime (2n): white solid (0.091, 84%); ^{1}H-NMR (500 MHz, DMSO): δ 11.51 (s, 1H), 8.80 (s, 1H), 8.68 (d, J =8.45 Hz, 1H), 7.95 (t, J =7.5, 7.65 Hz, 2H), 7.81 (d, J = 6.6 Hz, 1H), 7.63-7.49 (m, 3H); ESI-MS m/z 171 $[M]^+$.

12. Cyclohexanone oxime (2p): white solid (0.104 g, 91%); ^{1}H-NMR (500 MHz, $CDCl_3$): δ 8.546 (s, 1H), 2.522-2.497 (t, 2H), 2.228-2.203 (t, J = 6.0, 2H), 1.699-1.584 (m, J = 6.05, 6H); ESI-MS m/z 113 $[M]^+$.

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Notes and references

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