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Research Article

OXIDATION OF ALCOHOLS BY USING 2, 4, 6- TRIS [(4-DICHLOROIODO) PHENOXY)]- 1,3,5-TRIAZINE, AS A RECYCLABLE HYPERVALENT IODINE(III) REAGENT

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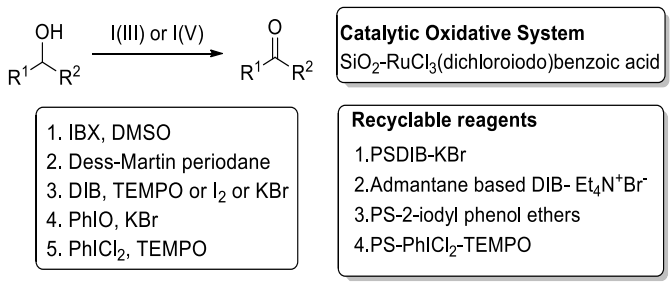
ABSTRACT

A clean and efficient method for the oxidation of alcohols by using recyclable nonpolymeric 2,4,6-*tris* [(4-dichloroiodo) phenoxy)]-1,3,5-triazine **2** and TEMPO as the co-oxidant is demonstrated. Near insolubility of the co-product triiodide 1 in methanol plays the crucial role in its easy regain and reuse for recyclability of the reagent 2, 4, 6-tris [(4-dichloroiodo) phenoxy)]-1,3,5-triazine 2.

Keywords: Oxidation, Alcohol, Hypervalent iodine reagent, (dichloroiodo)benzene, 1,3,5-triazine.

1. INTRODUCTION

The oxidation of alcohols to carbonyl compounds is a very basic and well-explored transformation in organic synthesis and numerous methods utilizing various reagents have been reported [1-2]. In synthetic chemistry, selective methods for oxidation of alcohols are highly searched. In order to differentiate amongst the variety of functional groups, these selective methods are very essential. This particular demand is fulfilled by the utilization of hypervalent iodine (III) and iodine(V) reagents as "leading reagents" for the oxidation of alcohols [3-5]. Dominance of hypervalent iodine reagents is due to its mild nature, easy access, reactivities similar to transition metal oxidants but with less toxicity [6-10]. (Scheme 1).



Scheme 1: Hypervalent iodine reagents used for the oxidation of alcohol

Various hypervalent iodine (III) and (V) reagents are reported for the oxidation of alcohols. Dess-Martin periodane (DMP) and *o*-iodoxybenzoic acid (IBX) [11-12], DMP with a base functionalized thiosulfate resin [13] is used for oxidation of reaction. Owing to the explosiveness of pentavalent iodine reagents [14-16] along with solubility issues, the oxidation of alcohols using trivalent iodine reagents have been long desired.

The trivalent iodine reagents normally show low reactivity towards alcohols. Therefore, iodine (III) reagents DIB, PhIO, BTI and $PhICl_2$ normally requires additives such as KBr, I_2, Et_4N+Br-, TEMPO, Pyridine, KNO_2, $RuCl_2(PPh_3)_3$, and chromium (III) (salen) complexes for effecting the oxidation of alcohols [3-5, 11, 12, 17-20].

In recent years the "*Green and clean*" approach for organic synthetic reactions is highly appreciated for ecological concern and use of recyclable reagents is emerging as best alternative to meet this purpose. Oxidation of alcohol by using various recyclable reagents like polymer supported hypervalent iodine reagents, DIB-TEMPO, Iodyl phenyl ethers [21-35], fluorine-tagged hypervalent iodine (III) reagents [36-38], ionic liquid-supported iodoarene-TEMPO bifunctional catalyst [39-40] are also demonstrated.

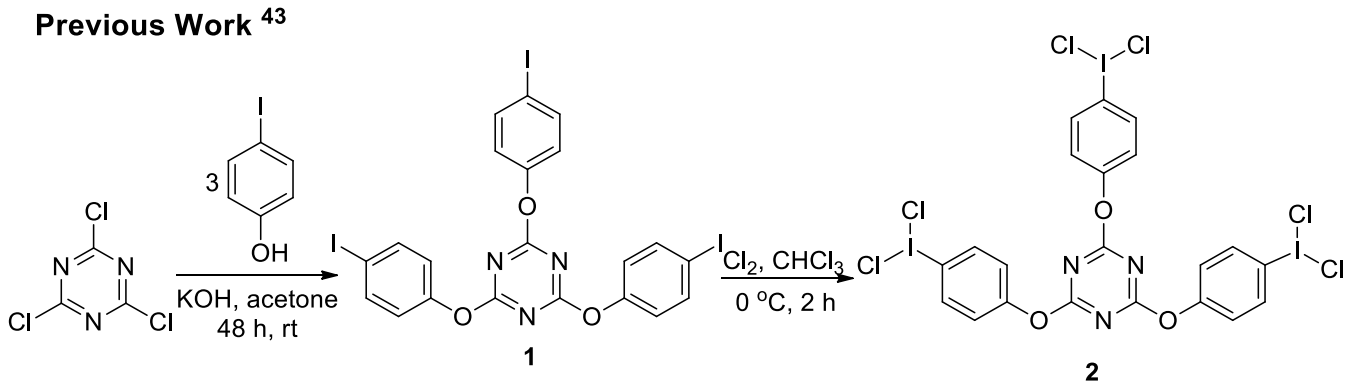
In the literature survey, recyclable catalytic oxidative system SiO_2-$RuCl_3$/3 (dichloroiodo) benzoic acid in water has been reported for the oxidation of alcohol, both SiO_2-$RuCl_3$ catalyst as well as the reduced form 3-iodobenzoic acid can be recycled easily [41]. The recyclable polymer supported dichloroiodoarene has been synthesized by Zhdankin *et. al.* and employed for

the oxidation of alcohols using catalytic quantity of TEMPO [31].

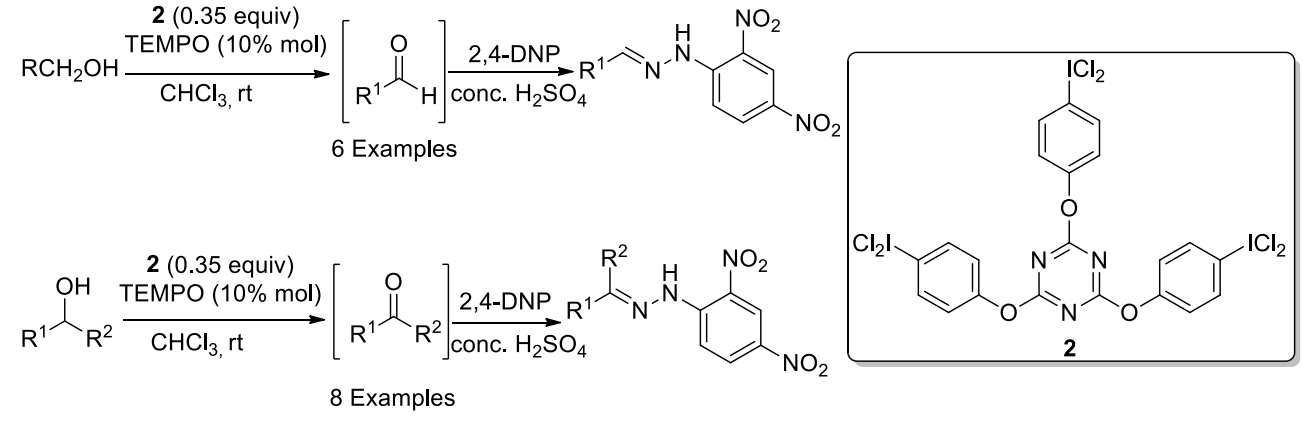
Inspired by the discovery of recyclable hypervalent iodine reagents based on admantane [35] and tetraphenyl tetraiodide [42] we reported the synthesis of hypervalent iodine (III) reagent 2,4,6-*tris* [(4-dichloroiodo) phenoxy)] -1,3,5-triazine (**2**) our previous work [43] from its "polyiodide" derivative 2,4,6-*tris* (4-iodophe-noxy)-1,3,5 -triazine [44-46] by chlorination.

The reagent 2,4,6-*tris* [(4-dichloroiodo)phenoxy)]-1,3,5-triazine **2** could be efficiently applied for the chlorination reaction of various activated arenes, olefin, and 1,3-diketone in CH_2Cl_2 subsequently, the same reagent has also been applied to the oxidative synthesis of 1,3,4-oxadiazoles and 1,2,4-thiadiazoles under mild conditions in excellent yields in CH_3CN [43].

Herein, as part of our continuing research, we wish to report the extended application of **2** as a recyclable non-polymeric dichloroiodoarenes for the oxidation of alcohols using 2,2,6,6-tetramethylpiperidine 1-oxy (TEMPO) as the catalyst (Scheme 3).



Scheme 2: Preparation of 2,4,6-*tris*[(4-iodo)phenoxy)]-1,3,5-triazine 1 and 2,4,6-*tris*[(4-dichloroiodo) phenoxy)]-1,3,5-triazine 2 [43]



Scheme 3: Oxidation of alcohols by using 2,4,6-*tris*[(4-dichloroiodo)phenoxy)]-1,3,5-triazine

Insolubility of the co-product triiodide 1 in methanol is the basis of recyclability of the reagent 2. After completion of the reaction, solvent ($CHCl_3$) was evaporated under reduced pressure followed by addition of methanol, the reduced form of reagent 2 *i.e.* tri-iodide 1 was precipitated which can be recovered quantitatively without any loss or degradation by simple filtration and washing with small amounts of methanol. Triiodide 1 can be further re-oxidised to reagent 2 subsequently by chlorination at 0 °C, while the filtrate

was concentrated and treated with 2, 4 DNP and conc. H_2SO_4 to afford the DNP derivative of carbonyl compounds in excellent yields.

2. MATERIAL AND METHODS

2.1. Optimization Study

Initially, a model reaction of benzyl alcohol with reagent **2** (0.33 equiv) as the oxidant in $CHCl_3$ (5mL) was carried out at room temperature. Gratifyingly, the desired benzaldehyde was obtained as the major product in 43% yield. It is reported that catalytic amount of TEMPO could improve the reaction efficiency [17-19] we were pleased to find that the yield of the desired product could be increased to 89 % with the addition of 0.1 equiv of TEMPO (Table 1, entry2). On slightly increasing the amount of reagent to 0.35 equivalents along with 0.1 equivalents we got the optimized condition with 91% yield (Table 1, entry3). Reaction proceeds efficiently in chloroform solvent.

With the optimized reaction conditions in hand, the present oxidative method was applied to a series of alcohols as shown in Table 2.

Table. 1: Optimization of oxidation of alcohol by using recyclable reagent 2

S. No	Alcohol	Solvent	Equiv. of Reagent 2	Equiv. of TEMPO	Yield
1	$PhCH_2OH$	$CHCl_3$	0.33	-	43%
2	$PhCH_2OH$	$CHCl_3$	0.33	0.1 equiv	89%
3	**$PhCH_2OH$**	**$CHCl_3$**	**0.35**	**0.1 equiv**	**91%**
4	$PhCH_2OH$	$CHCl_3$	0.4	0.1 equiv.	91%
5	$PhCH_2OH$	$CHCl_3$	0.35	0.2 equiv	91%
6	$PhCH_2OH$	CH_2Cl_2	0.35	0.1 equiv	89%

2.2. Synthesis of 2,4,6-*Tris*(4-dichloroiodophenoxy)-1,3,5-triazine(2)

2,4,6-*Tris* (4-dichloroiodophenoxy)-1,3,5-triazine (2) is prepared as per the procedures given in our previous work [43].

2.3. Experimental procedure for oxidation of alcohol

To a stirred solution of appropriate alcohol (2mmol, 1equiv) in $CHCl_3$ (5 mL), 2,4,6-*tris*[(4-dichloroiodo) phenoxy]-1,3,5-triazine (0.7 mmol, 0.35 equiv) and TEMPO (0.2 mmol, 0.1 equiv) was added and the reaction mixture was then stirred at room temperature for 1-4 hrs. The progress of reaction was monitored by TLC. When the substrate was consumed, the chloroform was evaporated under reduced pressure. Methanol was added to the reaction mass, and the white precipitate was isolated by filtration. Several wash of small amount of methanol was given and air dried to give 2,4,6-*tris*(4-iodophenoxy)-1,3,5-triazine in 93% yield. The filtrate of the above reaction mixture was concentrated to volume of 3-5 ml. To this filtrate, 2,4-DNP (5 mL) (solution prepared by standard method: 3g 2,4-DNP + 15 mL conc. H_2SO_4 + 70 mL EtOH + 20 mL H_2O) was added. The residue was again filtered and washed thrice with rectified spirit to afford the DNP derivative of carbonyl compounds in excellent yields.

3. RESULTS AND DISCUSSION

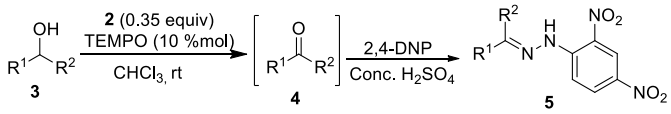
3.1. Oxidation of alcohols

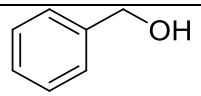
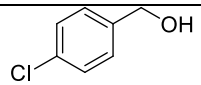
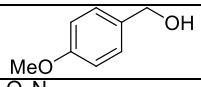
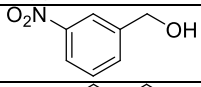
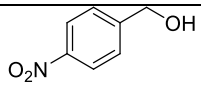
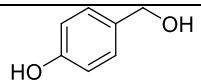
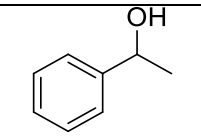
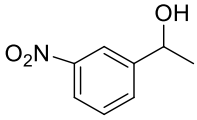
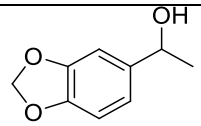
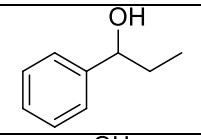
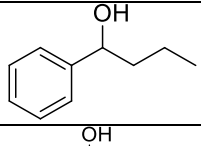
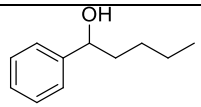
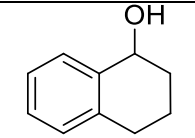
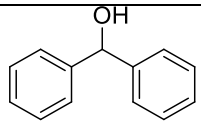
Oxidation of a series of alcohols to corresponding alcohols was carried out by applying optimised condition as shown in Table 2. Benzylic alcohols bearing either electron-rich or electron-deficient substituents on the aromatic ring all underwent the reactions smoothly to afford the corresponding aldehydes in good yields. No over oxidation of aldehydes to acids was observed in the case of the oxidation of primary alcohols. Secondary alcohols also underwent oxidation process smoothly to corresponding ketones. All the products thus formed are converted into its 2, 4-dinitrophenyl hydrazone derivatives. The oxidation is very clean and complete within 3-4 hours at room temperature. In all cases, the reactions proceeded readily at room temperature with high efficiency.

3.2. Mechanism

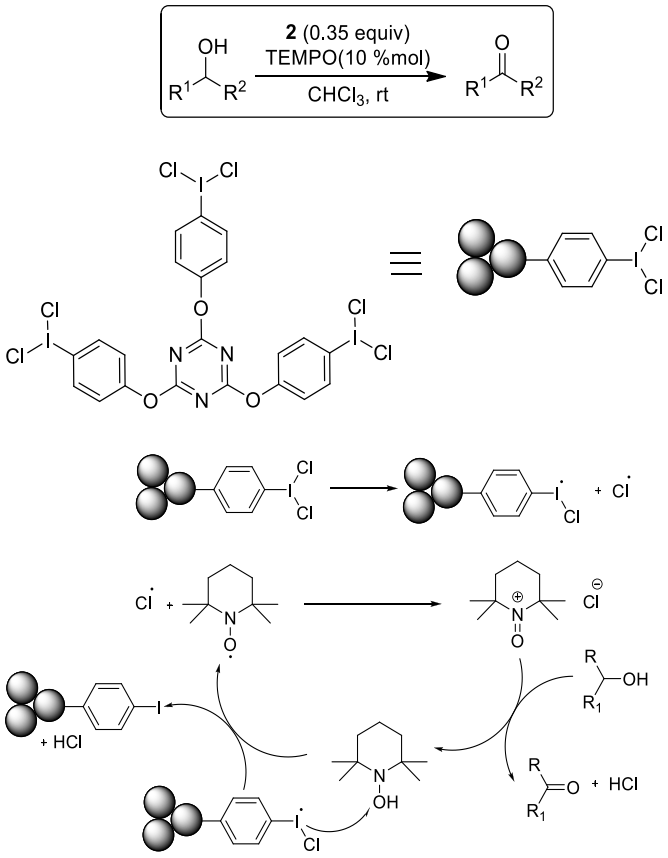
Plausible mechanism of reaction is assumed to be similar to the mechanistic path as predicted in previous report [20]. The formation of chlorine atom from $ArICl_2$ takes place on heating or light irradiation [47-49]. Oxidation of TEMPO to oxoammonium salt takes place by chlorine atom [50-51]. Alcohol is then, oxidized by this oxoammounium salt providing carbonyl compound and the salt reduces to a hydroxylamine [32, 52-59] which is further oxidized to TEMPO by second chlorine atom (Scheme 4).

Table 2: Oxidation of alcohols using reagent 2 and TEMPO



Entry	Alcohol (3)	Prod-uct (5)	Reac-tion time (h)	Yield of 5(%)[a]
1		**5a**	2	91
2		**5b**	2	93
3		**5c**	2.5	71
4		**5d**	3	76
5		**5e**	3	69
6		**5f**	4	77
7		**5g**	3.5	91
8		**5h**	2.5	83
9		**5i**	2	86
10		**5j**	2	93
11		**5k**	2.5	90
12		**5l**	2.5	87
13		**5m**	2.5	93
14		**5n**	3.5	84

[a]*Isolated yields*



Scheme 4: Plausible mechanism of the oxidation of Alcohols by using 2,4,6-*tris*[(4-dichloroiodo) phenoxy)]-1,3,5-triazine and TEMPO

The near insolubility of *co-produced* 2,4,6-*tris*[(4-iodo) phenoxy)]-1,3,5-triazine **1** in methanol paved the way for recyclability of 2,4,6-*tris* [(4-dichloroiodo) phenoxy)]-1,3,5-triazine **2**. Thus, after the completion of reaction, the reaction solvent ($CHCl_3$) was evaporated under reduced pressure followed by addition of methanol The resulting heterogeneous solution was then filtered, and the solid on the filter was washed several times with small amounts of methanol to recover **1** in nearly quantitatively yield (93-97%) which was subsequently subjected to the chlorination in $CHCl_3$ at 0°C to form **2**. The crude oxidation product present in the filtrate was then concentrated and treated with 2, 4 DNP and conc. H_2SO_4 to afford the DNP derivative of carbonyl compounds in excellent yields (Fig.1).

3.3. Spectral data

3.3.1. (E)-1-Benzylidene-2-(2,4-dinitrophenyl) hydrazine (5a)

Nature: Yellow solid; Yield: 77% (407 mg); m.p. 238-240°C (Lit.m.p. 241-242°C) [40]; IR (cm^{-1}): 3285,

3102, 1620, 1585, 1505, 1417, 1329, 1312, 1261, 1218, 1133, 1073, 950, 835, 763, 741, 722, 693, 645.

3.3.2. (E)-1-(4-Chlorobenzylidene)-2-(2,4-dinitrophenyl) hydrazine (5b)

Nature: Orange yellow solid; Yield: 93% (419 mg); m.p. 262-264°C (Lit.m.p. 265-266°C) [60]; IR (cm^{-1}): 3283, 3090, 1611, 1582, 1505, 1488, 1422, 1324, 1220, 1134, 1079, 1010, 896, 824, 741, 726, 612.

3.3.3. (E)-1-(2,4-Dinitrophenyl)-2-(4-methoxybenzylidene)hydrazine (5c)

Nature: Orange yellow solid; Yield: 71% (325 mg); m.p. 258-260°C (Lit. m.p. 256-257°C [40]; 258-259°C [60]); IR (cm^{-1}): 3273, 1625, 1604, 1585, 1501, 1416, 1334, 1309, 1267, 1250, 1211, 1163, 1133, 1081, 1018, 969, 914, 832, 742, 715, 643, 625.

3.3.4. (E)-1-(2,4-Dinitrophenyl)-2-(3-nitrobenzylidene)hydrazine (5d)

Nature: Yellow solid; Yield: 76% (328 mg); m.p. 292-294°C (Lit. m.p. 293-294°C) [60] ; IR (cm^{-1}): 3281, 3079, 1698, 1613, 1584, 1504, 1418, 1326, 1314, 1276, 1199, 1090, 1053, 1031, 814, 763, 758, 676.

3.3.5. (E)-1-(2,4-Dinitrophenyl)-2-(4-nitrobenzylidene)hydrazine (5e)

Nature: Yellow solid; Yield: 79% (341 mg); m.p. 316-318°C (Lit. m.p. 320-321°C) [60]; IR (cm^{-1}): 3277, 3090, 1610, 1591, 1573, 1504, 1422, 1325, 1267, 1213, 1133, 1086, 1054, 897, 851, 833, 741, 723, 685, 619.

3.3.6. (E)-4-((2-(2,4-Dinitrophenyl) hydrazono) methyl) phenol (5f)

Nature: Red solid; Yield: 77% (375 mg); m.p. 184-186°C; ^{1}H NMR 400 MHz (DMSO-d6): δ 6.86 (d, J = 8.4 Hz, 2H), 7.62 (d, J = 7.2 Hz, 2H), 8.03 (d, J = 8.8 Hz, 1H), 8.32 (d, J = 10 Hz, 1H), 8.56 (s, 1H), 8.83 (s, 1H), 11.54 (bs, 1H); IR (cm^{-1}): 3263, 1605, 1583, 1488, 1416, 1301, 1267, 1200, 1131, 1048, 837, 738, 715, 651.

3.3.7. (E)-1-(2,4-Dinitrophenyl)-2-(1-phenylethylidene)hydrazine (5g)

Nature: Orange solid; Yield: 91% (447 mg); Mp 242-244°C (Lit. m.p. 247-248°C) [60]; IR (cm^{-1}): 3304, 3118, 1615, 1582, 1514, 1493, 1417, 1329, 1298, 1259, 1220, 1111, 1070, 1054, 925, 839, 759, 742, 719, 689, 659, 618.

3.3.8. (E)-1-(2,4-Dinitrophenyl)-2-(1-(3-nitrophenyl)ethylidene)hydrazine (5h):

Nature: Yellow solid; Yield: 83% (342 mg); m.p. 228-232°C (Lit. m.p. 233-234°C) [60]; ^{1}H NMR 400 MHz (DMSO-d6): δ 2.50 (s, 3H), 7.78 (t, J = 8 Hz, 1H), 8.10 (d, J = 9.6 Hz, 1H), 8.29-8.49 (m, 3H), 8.63 (s, 1H), 8.90 (s, J = 2.8 Hz, 1H), 11.12 (bs, 1H);IR (cm^{-1}): 3305, 3105, 1612, 1587, 1530, 1504, 1417, 1336, 1302, 1279, 1260, 1220, 1119, 1049, 921, 906, 878, 844, 831, 80, 783, 747, 736, 674, 620.

3.3.9. (E)-1-(1-(Benzo[d][1,3]dioxol-5-yl) ethylidene)-2-(2,4-initrophenyl)hydrazine (5i)

Nature: Red solid; Yield: 86% (356 mg); m.p. 250-252°C; ^{1}H NMR 400 MHz (DMSO-d6): δ 2.42 (s, 2H), 6.11 (s, 2H), 7.01 (d, J = 8 Hz, 1H), 7.44 (d, J = 8 Hz, 1H), 7.58 (s, 1H), 8.11 (d, J = 9.6 Hz, 1H), 8.391 (d, J = 7.2 Hz, 1H), 8.89 (d, J = 2.4 Hz, 1H), 11.08 (bs, 1H); IR (cm^{-1}): 3315, 3083, 1615, 1585, 1514, 1485, 1446, 1417, 1330, 1297, 1267, 1226, 1129, 1093, 1060, 1036, 935, 891, 841, 808, 742, 709, 664, 629.

3.3.10. (E)-1-(2,4-Dinitrophenyl)-2-(1-phenylpropylidene)hydrazine (5j)

Nature: Red solid; Yield: 93% (429 mg); m.p. 146-150°C; IR (cm^{-1}): 3322, 3113, 1617, 1582, 1531, 1509, 1477, 1411, 1363, 1304, 1246, 1214, 1130, 1103, 1052, 908, 844, 829, 766, 751, 738, 715, 685, 655.

3.3.11. (E)-1-(2,4-Dinitrophenyl)-2-(1-phenylbutylidene)hydrazine (5k)

Nature: Orange solid; Yield: 90% (393 mg); m.p. 158-160°C; ^{1}H NMR 400 MHz (DMSO-d6): δ 1.05 (t, J = 7.4 Hz, 3H), 1.55-1.77 (m, 2H), 2.90 (t, J = 7.8 Hz, 2H), 7.48 (s, 3H), 7.94-7.97 (m, 2H), 8.10 (d, J = 9.2 Hz, 1H), 8.41-8.43 (m, 1H), 8.90 (s, 1H), 11.28 (bs, 1H); IR (cm^{-1}): 3325, 3112, 1613, 1583, 1537, 1511, 1489, 1418, 1331, 1307, 1275, 1260, 1135, 1106, 1056, 910, 832, 789, 765, 741, 719, 696, 653.

3.3.12. (E)-1-(2,4-Dinitrophenyl)-2-(1-phenylpentylidene)hydrazine (5l)

Nature: Red solid; Yield: 87% (362 mg); m.p. 142-144°C; ^{1}H NMR 400 MHz (DMSO-d6): δ 0.95 (t, J = 7.4 Hz, 3H), 1.47-1.60 (m, 4H), 2.91 (t, J = 8 Hz, 2H), 7.48 (s, 3H), 7.93-7.96 (m, 2H), 8.10 (d, J = 9.2 Hz, 1H), 8.40-8.43 (m, 1H), 8.89 (d, J = 2.4 Hz, 1H), 11.26 (bs, 1H); IR (cm^{-1}): 3314, 2956, 2863, 1687,

1615, 1581, 1514, 1480, 1417, 1362, 1328, 1260, 1219, 1095, 915, 843, 824, 763, 719, 689, 653.

3.3.13. (E)-1-(3,4-Dihydronaphthalen-1(2H)-ylidene)-2-(2,4-dinitrophenyl)hydrazine (5m)

Nature: Red solid; Yield: 93% (409 mg); m.p. 240-242°C; ^{1}H NMR 400 MHz (DMSO-d6): δ 1.95 (t, J = 5.8 Hz, 2H), 2.79-2.86 (m, 4H), 7.26-7.38 (t, m, 3H), 8.14-8.21 (m, 2H), 8.41-8.44 (m, 1H), 8.90 (s, 1H), 11.14 (bs, 1H); IR (cm^{-1}): 3310, 3112, 2957, 1621, 1587, 1513, 1501, 1415, 1329, 1306, 1254, 1221, 1127, 1103, 1067, 909, 838, 759, 734, 706, 665, 633.

3.3.14. 1-(2,4-Dinitrophenyl)-2-(diphenylme-thylene) hydrazine (5n):

Nature: Orange solid; Yield: 84% (330 mg); m.p. 226-228°C; ^{1}H NMR 400 MHz (DMSO-d6): δ 7.47-7.49 (m, 5H), 7.65-7.71 (m, 5H), 8.25 (d, J = 9.6 Hz, 1H), 8.43-8.48 (m, 1H), 8.81 (s, 1H), 11.07 (bs, 1H); IR (cm^{-1}): 3288, 1611, 1580, 1501, 1442, 1417, 1362, 1306, 1260, 1211, 1125, 1083, 1053, 966, 906, 829, 774, 737, 694, 665, 631.

4. CONCLUSION

In summary, we have reported oxidation of alcohols to carbonyl compounds by using 2,4,6-*tris*[(4-dichloroiodo) phenoxy)]-1,3,5-triazine recyclable reagent. The products of oxidation reactions are conveniently separated from the co-produced 2,4,6-*tris*(4-iodophenoxy)-1,3,5-triazine **1** by simple filtration in methanol promoting the high recyclability and reuse of reagent. Oxidation products are further converted to its 2,4-dinitrophenyl hydrazone derivatives. Thus this protocol provides a clean, efficient and transition metal free procedure with high recyclability and good yields for oxidation of alcohols.

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