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Rate Enhancements Due to Ultrasound in Isoquinolinium Dichromate and Isoquinolinium Chlorochromate Catalyzed Chlorination of Aromatic Compounds in Presence of $KHSO_4$/KCl

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Chlorination of aromatic compounds underwent magnificent rate accelerations in isoquinolinium dichromate and isoquinolinium chlorochromate catalyzed chlorination of aromatic hydrocarbons in the presence of KCl and $KHSO_4$. Reaction times reduced highly significantly from 4-5 h in conventional protocol to 30-40 min under sonication, followed by high yields of monochloro derivatives as products with high regioselectivity.

Keywords: Chlorination, Aromatic compounds, Isoquinolinium chlorochromate, Isoquinolinium dichromate, Sonication.

INTRODUCTION

Like several other electrophilic aromatic substitution reactions, chlorination of aromatic compounds is also an evergreen and prominent synthetic protocol because of its uses in the synthesis of diversified aromatic compounds such as aromatic ethers and thioethers, amines, arylhydrazines, benzonitriles, fluoroaromatic, silylated aromatic hydrocarbons and many more functionalities [1-3]. Reagents such as molecular chlorine [4], sulfuryl chloride [5], alkyl and acyl hypochlorites [6], inorganic chlorides [7], *m*-CPBA/18-crown-6/KCl [8], $NaBO_3/Na_2WO_4$/KCl [9], H_2O_2/NH_4VO_3/KCl [10], N-chlorosuccinimide [11], benzyltrimethyl ammonium tetrachloroiodate ($BTMAICl_4$) [12], dichlorinemonoxide [13], $PhICl_2$ in trifluoroacetic acid [14], N-chloroamines [15], N-chloroamides and N-chlorosulfonamides [16]. Many of the reported methods have some limitations such as use of strong and non-selective chlorinating agents, toxic and expensive reagents, low yields and long reaction times. In some protocols, after completion of the reaction, large amount of acid waste is drained through the outlets of laboratories and industries. Oxychlorination methods were among some such protocols through which one can address few issues and also achieve smooth chlorination [17,18]. Onium halochromates and onium dichromates were among the few most effective reagents, which could be used for oxidation as well as oxyhalogenation of organic compounds [19]. Srinivasan *et al*. [20] synthesiszed isoquinolinium dichromate (a new oniumdichromate) which proved as a versatile reagent for selective oxidation of primary and secondary alcohols under mild conditions. It oxidized vicinal and non-vicinal diols to the corresponding α-hydroxy carbonyl compounds. Recently, we have accomplished the use of isoquinolinium dichromate and isoquinolinium chlorochromate as efficient catalysts to trigger oxidative bromination and iodination of aromatic hydrocarbons with KBr/KI and $KHSO_4$ under acid-free conditions. Reaction times reduced highly significantly under sonication, followed by corresponding monobromo derivatives in very good yield with high regioselectivity [21]. Encouraged by this protocol we have embarked on exploring the possibility of isoquinolinium chlorochromate (IQCC) and isoquinolinium dichromate (IQDC) as catalysts to achieve oxidative chlorination of aromatic compounds with various functional groups such as phenols, anilines and acetanilides. We also wish to explore the use of ultrasound to assist IQCC and IQDC mediated chlorination reactions with a view to accelerate chemical reactions for achieving better yields as well as improving the greenery of reaction protocols. Earlier reviews and publications in this field [22-32] proved the importance of ultrasonically assisted organic synthesis and also highlighted that sonication using ultrasound is not only simple, but also satisfies both economical and environmental demands, as recommended by Anastas and Warner in the green chemistry formulations [33].

EXPERIMENTAL

All chemicals used were of synthesis grade reagents and procured from Merck, S.D. Fine or Avra Chemicals. Isoquino-

linium dichromate (IQDC) and isoquinolinium chlorochromate (IQCC) were prepared as reported method [20,21].

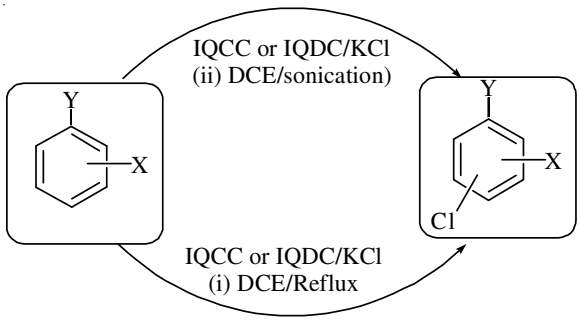
General procedure for conventional chlorination reactions: Organic compound (10 mmol) potassium chloride (KCl 10 mmol) and (1 mmol) Cr(VI) reagent (IQCC or IQDC) and solvent (aq. acetontrile or DCE) were taken in a clean round bottom flask. About 50 mg of $KHSO_4$ is also added to the contents of reaction flask and the reaction mixture is refluxed for about 4-5 h at 50-60 ºC till the completion reaction, as ascertained by thin layer chromatography (TLC). The reaction mixture is then treated with 5% sodium thiosulphate solution followed by the addition of ether. The aqueous layer was separated from the ether layer. The ether is evapourated from organic layer under vacuum, and purified with column chromatography using chloroform: *n*-hexane (9:1) as eluent to get pure chlorinated aromatic compound as product [2,3].

General procedure for ultrasonically assisted chlorination reactions: The general procedure for ultrasonically assisted chlorination reaction is largely similar to the preceeding conventional method. After mixing the reactants in a clean round bottom flask, is placed in an ultrasound assited sonicator and sonicated at room temperature till the reaction is completed (about 30-40 min). Progress of the reaction was monitored by TLC technique. Work up procedure after completion of the reaction mixture is similar to as described above.

RESULTS AND DISCUSSION

The chlorination reactions of aromatic compounds were conducted using isoquinolinium dichromate (IQDC)/KCl and isoquinolinium chlorochromate (IQCC)/KCl combinations in aqueous $KHSO_4$ under mineral acid free conditions. Described methods worked out well for an array of functionalities such as phenols, anilines or acetanilides (**Scheme-I**).

Each reaction is repeated at least half a dozen times and the reaction times are reproducible with an accuracy of ± 5 min. The chlorination of aromatic compounds required 4-5 h under conventional conditions at reflux temperatures, but under sonication the reaction times are drastically reduced to about 30 to 40 min followed by considerable yield enhancements. The products of these reactions were characterized by spectroscopic and physical data with authentic samples and found to be satisfactory with literature reports [2,3]. The yields of major products are given in Table-1.



Scheme-I: IQDC and IQCC triggered chlorination of aromatic compounds

Spectroscopic data of some synthesized compounds:

4-Chlorophenol: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24-7.17 (m, 2H, Ph), 6.77-6.74 (m, 2H, Ph), 5.27 (s, 1H, OH); HRMS (ESI-TOF) *m/z*: calcd. for C_6H_5OCl 127.0029, found 126.9956.

2-Methoxy-4-chlorophenol: ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.20 (s, 1H, OH), 6.95 (d, *J* = 2.0 Hz, 1H, Ph), 6.79-6.75 (m, 2H, Ph), 3.78 (s, 3H, OCH_3); HRMS (ESI-TOF) *m/z*: calcd. for $C_7H_6O_2Cl$ 157.0135, found 157.0056.

2-Chloro-4-methoxyphenol: ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.55 (s, 1H, OH), 6.94-6.90 (m, 2H, Ph), 6.76 (dd, *J* = 3.2, 8.8 Hz, 1H, Ph), 3.69 (s, 3H, OCH_3); HRMS (ESI-TOF) *m/z*: calcd. for $C_7H_6ClO_2$ 157.0135, found 157.0059.

4-Chloro-3-methylphenol: ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.51 (s, 1H, OH), 7.16 (d, *J* = 2.4 Hz, 1H, Ph), 6.74 (d, Hz, 1H, Ph), 6.62 (dd, , 8.8 Hz, 1H, Ph), 2.24 (s, 3H, CH_3); HRMS (ESI-TOF) *m/z*: calcd. for C_7H_6ClO 141.0185, found 141.0116.

TABLE-1
OXIDATIVE CHLORINATION OF AROMATIC HYDROCARBONS
Reaction time Conventional: 4-5 h; Sonication: 30-40 min

S. No.	Substrate	Product	Conventional yield (%)		Sonication yield (%)	
			KCl/IQCC	KCl/IQDC	KCl/IQCC	KCl/IQDC
1	Phenol	4-Chloro Phenol	70	68	71	70
2	*o*-Cresol	4-Chloro-2-methylphenol	65	60	66	62
3	*p*-Cresol	2-Chloro-4-methylphenol	65	69	69	68
4	*m*-Cresol	4-chloro-3-methylphenol	66	70	68	71
5	4-Chloro phenol	2,4-Dichloro phenol	55	60	59	63
6	2-Chloro phenol	2,4-Dichloro phenol	59	61	62	64
7	4-Bromo phenol	4-Bromo-2-chloro phenol	60	66	66	69
8	1,4-Dihydroxy benzene	2-Chloro benzene-1,4-diol	71	74	76	72
9	1-Naphthol	2-Chloro-1-naphthol	69	73	68	71
10	Benzaldehyde	3-Chloro benzaldehyde	70	73	71	73
11	1-(3-OH phenyl)ethanone	1-(3-OH-4-chloro phenyl)ethanone	62	66	63	65
12	4-NO_2 phenol	4-NO_2-2-chloro phenol	65	69	68	70
13	2-OH benzaldehyde	5-Chloro-2-OH-benzaldehyde	55	59	59	60
14	4-OH benzaldehyde	3-Chloro-4-OH-benzaldehyde	65	69	69	70
15	Toluene	2-Chlorotoluene	69	70	68	69
16	2-OH-benzoic acid	3-Chloro-6-OH- benzoic acid	69	70	71	70
17	2-OH-aniline	3-Chloro-4-OH-aniline	66	68	70	77
18	4-Cl-acetophenone	3,4-di-Cl-acetophenone	71	67	69	70

3-Chloro-5-hydroxy benzaldehyde: ^{1}H NMR (400 MHz, DMSO-d_6) δ 10.93 (s, 1H), 10.23 (s, 1H), 7.60 (d, Hz, 1H, Ph), 7.54 (dd, , 8.8 Hz, 1H, Ph), 7.05 (d, Hz, 1H, Ph); HRMS (ESI-TOF) *m/z*: calcd. for $C_7H_4ClO_2$ 154.9978, found 154.9898.

4-Chloro-2-methylphenol: ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.52 (s, 1H, OH), 7.10 (d, Hz, 1H, Ph), 7.01 (dd, , 8.4 Hz, 1H, Ph), 6.78 (d, Hz, 1H, Ph), 2.11 (s, 3H, CH_3); HRMS (ESI-TOF) *m/z*: calcd. for C_7H_6OCl 141.0185, found 141.0107.

4-Chloro-2-nitroaniline: Yellow orange powder; ^{1}H NMR (400 MHz, DMSO) δ 7.90 (d, *J* = 2.42 , 1H, Ar), δ 7.28 (dd, *J* = 9.20, 2.24, 1H, Ar), δ 7.06 (d, *J* = 9.22, 1H, Ar) δ 7.55 (bs, 1H, NH_2) ppm; MS (APCI): calcd. for $C_6H_5N_2O_2Cl$ $[M]^+$ 172.57, found 172 $[M]^+$.

3-Chloro-4-hydroxybenzaldehyde: Light brown powder; ^{1}H NMR (400 MHz, DMSO) δ 9.78 (s, 1H, CHO), δ 7.81 (d, *J* = 1.80 Hz, 1H, Ar), δ 7.64 (dd, *J* = 8.40, 1.80 Hz, 1H, Ar), δ 7.12 (d, *J* = 8.32 Hz, 1H, Ar) ppm; MS: calcd. For $C_7H_5O_2Cl$ $[M]^+$ 156,5, found 155 $[M-1]^+$.

5-Chlorosalicylic acid: White crystals; ^{1}H NMR (400 MHz, DMSO) δ 9.05 (s, 1H, OH), δ 7.79 (d, *J* = 2.52 Hz, 1H, Ar), δ 7.38 (dd, *J* = 8.80, 2.52 Hz, 1H, Ar), δ 6.91 (d, *J* = 8.88 Hz, 1H, Ar) ppm; MS: calcd. for $C_7H_5O_3Cl$ [M]+ 172, found 171.

We have also tried to to explain the mechanism of IQDC/KCl mediated chlorination reaction. It is well established in earlier reports that in aquoues acidic media $K_2Cr_2O_7$ or Cr(VI) exists in several reactive forms, such as $HCrO_4^-$, H_2CrO_4, $[HCrO_3]^+$ and $HCrO_3B$ (where B = HSO_4^-, ClO_4^- or Cl^-) [34,35]. On the basis of fact that quinolinium dichromate (QDC) is related to $K_2Cr_2O_7$, we have formulated similar types of reactive species with quinolinium ion background [36]. Further, it is also of interest to note that isoquinolinium dichromate (IQDC) resembles the structure of quinolinium dichromate (QDC), following species could be considered according to the following equilibria:

$$(IQH)_2Cr_2O_7 + H_2O \rightleftharpoons 2[(IQH) + (HCrO_4)^-] \quad (1)$$

As the reactions are conducted in presence of aqueous $KHSO_4$ medium, it is more likely that the bisulfate ion dissociates to give (H^+) creating mild acid reaction conditions. Protons (H^+) thus released could be used to protonate IQDC, leading to the formation of active $[(IQH)^+(HCrO_4)^-]$ (IQ bound chromic acid) species.

$$HSO_4 \rightleftharpoons SO_4^{2-} + H^+ \quad (2)$$

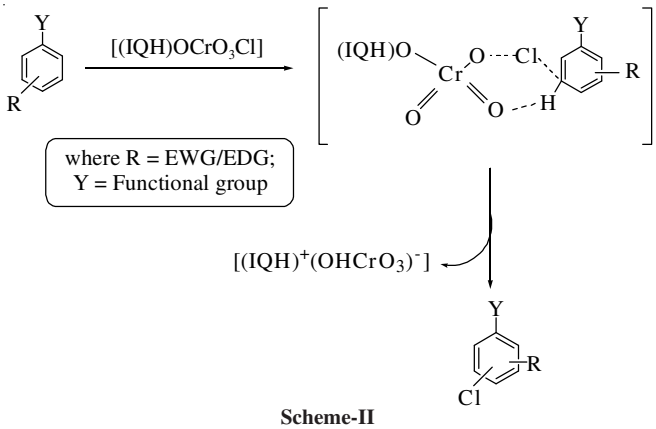
$$(IQH)^+(HCr_2O_4)^- + H^+ \rightleftharpoons [(IQH)CrO_3]^+ + H_2O \quad (3)$$

Active species thus formed may further react with chloride ion to afford $[(IQH)CrO_3Cl]$ species, since the reactions are conducted in excess KCl.

$$[(IQH)CrO_3)^+ + Cl^- \rightleftharpoons [(IQH)OCrO_2Cl] \quad (4)$$

Finally, aromatic substrates undergo mono chlorination through the formation of cyclic intermediate with aromatic compound as shown in **Scheme-II**.

On the other hand, the most plausible mechanism in IQCC triggered reactions could be proposed by formulating isoquinolinium chlorochromate (IQCC) in the lines of other onium halochromate species [37-40]. Since the reactions are studied in aqueous $KHSO_4$ medium, IQCC could exists as $(IQHOCrO_2Cl)$



Scheme-II

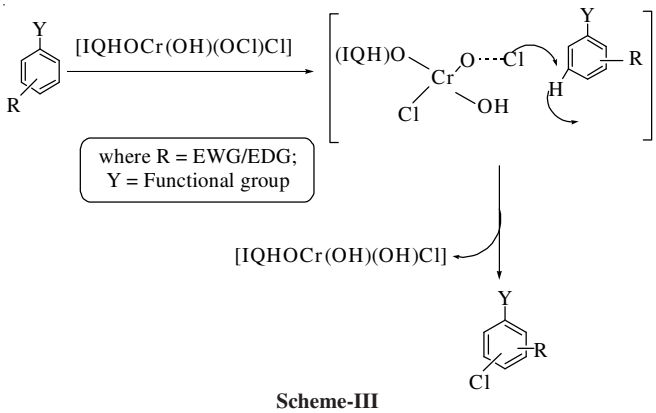
and its protonated form $(IQHOCr(OH)Cl)^+$, by capturing H^+ from the dissociated bisulfate species, according to the following equilibria:

$$(IQHOCrO_2Cl) + H^+ \rightleftharpoons (IQHOCr(OH)OCl)^+ \quad (5)$$

The protonated chromium(VI) species thus formed $(IQHOCr(OH)OCl)^+$ being a stronger electrophile, may further react with chloride ion to afford [IQHOCr(OH)(OCl)Cl] species, since the reactions are conducted in excess KCl.

$$[IQHOCr(OH)OCl]^+ + Cl^- \rightleftharpoons [IQHOCr(OH)(OCl)Cl] \quad (6)$$

Finally, aromatic substrates undergo mono chlorination through the attack of Cl^+ of [IQHOCr(OH)(OCl)Cl] on the aromatic ring as shown **Scheme-III**.



Scheme-III

The observed rate enhancements in ultrasonically assisted reactions could be explained due to cavitation, a physical process that creates, enlarges implodes gaseous and vaporous cavities in an ultrasonically assisted (irradiated) liquid. Cavitation induces very high local temperatures in the reaction mixture and enhances mass transfer [20-29].

Conclusion

In summary, isoquinolinium chlorochromate (IQCC) and isoquinolinium dichromate (IQDC) as cost-effective catalysts to trigger chlorination of aromatic compounds in presence of KCl and small amount of $KHSO_4$ is successfully explored. The use of mineral acid is completely avoided in these protocols. Reaction times drastically reduced from 4-5 h (in conventional) to 30-40 min under sonication. The reactions occurred under mild and under environmentally safe conditions with simple

work up at room temperature. Thus, it is believed that the developed protocols are one of the important contributions in the area of chlorination reactions.

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