



IJRASET

International Journal For Research in
Applied Science and Engineering Technology



INTERNATIONAL JOURNAL FOR RESEARCH

IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY

Volume: 6 Issue: 1 Month of publication: January 2018

DOI: <http://doi.org/10.22214/ijraset.2018.1154>

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Microwave Assisted Ecofriendly Halogenation of Organic Compounds by Hypervalent Cr (VI) Reagents with Alkali Metal Halides in Acid Free Media

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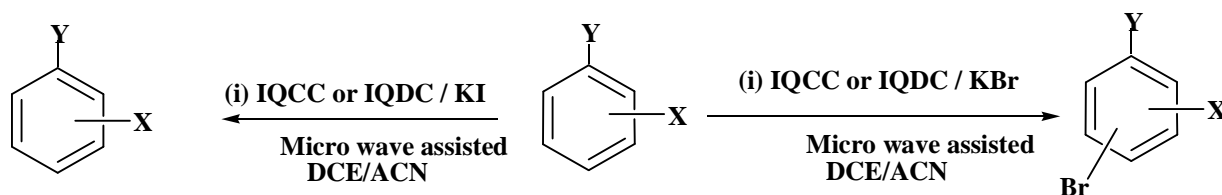
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GRAPHICAL ABSTRACT



Where Y= OH, NH₂ and NHCOCH₃; X = electron donating or withdrawing group

Abstract: An efficient and green chemical protocol for the synthesis of halo organic compounds by the action of Isoquinolinium Chlorochromate (IQCC), Isoquinolinium dichromate (IQDC) and alkali metal halides in acid free media is been described in the present work. In This procedure, which involved in situ generation of the active halogen in presence of alkali halides? and smooth high yield halogenations obtained.

Keywords: Green Chemistry; Halogenation; Synthesis Isoquinolinium chlorochromate (IQCC), Isoquinolinium dichromate (IQDC) Alkali Metal Halides (KBr,, ,KI), KHSO₄

I. INTRODUCTION

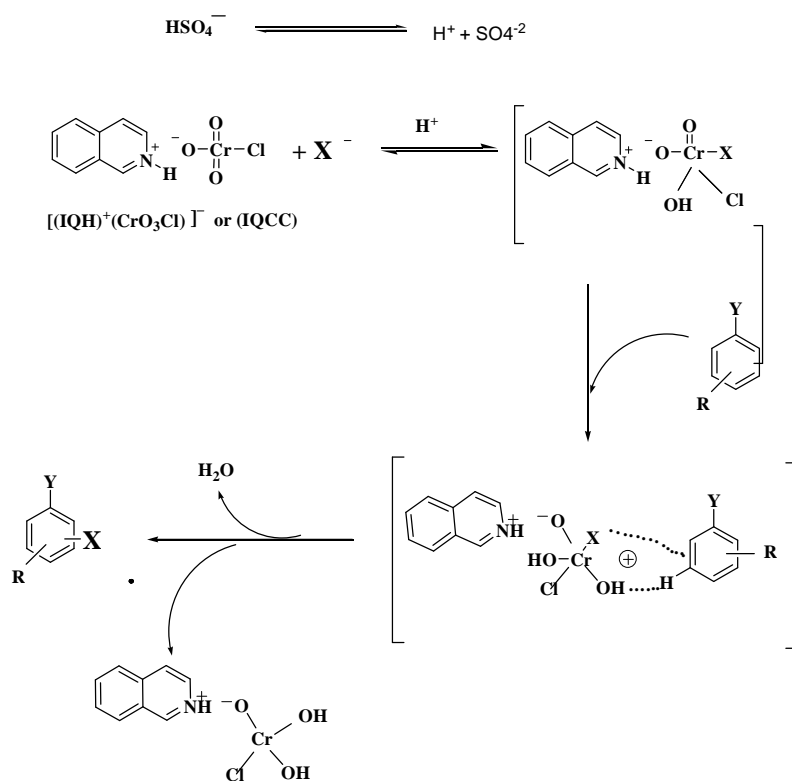
Halogenation of aromatic compounds is an area of evergreen interest to chemists among electrophilic aromatic substitution (EAS) reactions because organic halides can be converted efficiently into other functional groups by simple chemical transformations [1-3]. Numerous industrially valuable products such as pesticides, insecticides, herbicides, pharmaceutically and medicinally active molecules and other newer materials carry halogen functionality. Traditionally, aromatic iodo compounds and bromo compounds are prepared by reaction with elemental halogen in the presence corrosive medium For instance, in bromination method, molecular bromine or brominating mixture (a mixture of KBr and KBrO₃) and mineral acid are essential components for the generation of bromonium ion. . In H₂O₂-HBr, the classical direct bromine-tion of aromatic compounds using HBr is highly toxic, corrosive and as harmful as of molecular bromine to the environment. Also NBS-H₂SO₄-H₂O system has been reported for the bromination of benzene and toluene.

The handling of iodine and liquid bromine is difficult due to their hazardous nature while special equipment and care are needed for the transfer of these materials in large scale. Moreover, halogenation of aromatic substrates with elemental halogens involves a substitution reaction with the formation of high toxic, corrosive acids as by product after completion of the reaction, large amount of acid waste is drained through the outlets of laboratories and industries. This acid waste became root cause for environmental pollution. Besides, molecular bromine is hazardous, which also pollutes the environment. In oxyhalogenation, hydrogen halide (HX), generated *in situ* could be used as a halogen source. In order to prevent acid-waste and direct use of molecular halogen

Scheme-2

It is believed that the active "Protonated IQCC" species is more selective than chromic acid/chromyl chloride.

Scheme-2: IQCC triggered halogenation mechanism.



It is also of interest that there is not much difference in the rates of bromination and iodination as could be seen from the data presented in tables 1 and 2. The observed rate accelerations in ultrasonically assisted reactions could be explained due to cavitation, a physical process that creates, enlarges, and 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 ^[15-20].

III. EXPERIMENTAL DETAILS

A. Materials

All chemicals used were synthesis-grade reagents and procured from Merck, SD Fine or Avra Chemicals.

B. Isoquinolinium Dichromate (iqdc)

Isoquinoline (30 ml, 0.25mol) is added in small portions to the aqueous solution of Chromium tri oxide (30.0 g or 0.30 mol 30 mL water) in a beaker at chilled temperature in an ice bath. The reaction mixture was stirred for about 30 min. The resultant solution was diluted with 100 ml of acetone and cooled again to -10°C. The orange crystalline isoquinolinium dichromate [(C₉H₇NH⁺)₂Cr₂O₇²⁻] formed in the beaker is filtered, washed with acetone, recrystallized from hot water and dried in vacuum for about 2 h. Melting point of the compound (169-170°C), is verified with literature reports

C. Isoquinolinium Chlorochromate (IQCC)

The method of preparation of IQCC is mostly similar to IQDC. Isoquinoline (30 ml, 0.25mol) is added in small portions to the aqueous solution of Chromium trioxide (30.0 g or 0.30 mol 30 mL water) and few mL of 6M HCl in a beaker in an ice bath under constant stirred conditions for about 30 min. The resultant solution was diluted with 100 ml of acetone and cooled again to -10°C. Dark brown crystalline isoquinolinium Chlorochromate (IQCC) formed in the beaker is filtered, washed with acetone, recrystallized from hot water and dried in vacuum for about 2 h. Observed melting point of the compound (169-170°C), which is by and large in the range of literature reports [25].

D. General Procedure for Microwave assisted halogenation reactions

The general method for Microwave assisted bromination and iodination reaction is almost similar to conventional reaction as mentioned above. A cent molar (0.01mol) organic substrate (Phenols, Anilines or Acetanilides), potassium halide (KBr), (0.001 mol), about 50 mg of dilute KHSO_4 and hypervalent Cr (VI) reagent (IQCC or IQDC) were suspended in about 30 ml solvent (DCE or ACN), and silica gel was added covered with a lid after the mixer was placed in the microwave apparatus, operating at a frequency of 2.45 ghz with continuous irradiation power from 0 t 300 w . . The progress of the reaction was monitored by TLC. After the completion of reaction, the organic layer was diluted with dichloro-methane (10 ml), and separated from aqueous layer. It was then washed with (3 × 5 ml) water, separated and finally dried over sodium sulphate. The anhydrous dichloro- me-thane layer was concentrated in reduced pressure to afford the crude product, which was subjected to column chromatography (silica gel, 100 - 200 mesh) using EtOAc-hexane (1:9) as fluent to obtain the pure product.

. The yields of the products have been compiled in tables 1 and 2.

Table -1

S.No	Substrate	Product	Conventional KBr+IQCC	Conventional KBr+IQDC	Microwave Assisted KBr+IQCC	Microwave Assisted IQDC KBr+IQDC
1	Phenol	4- Bromo Phenol	65	66	78	69
2	o-cresol	4-brom-2-methylphenol	60	62	70	67
3	p-cresol	2bromo -4-methylphenol	69	67	75	62
4	m-cresol	4-bromo-3-methylphenol	69	66	68	62
5	4-chloro Phenol	4-chloro-2-bromoPhenol	68	61	66	68
6	2-chloro Phenol	2-chloro-4-bromoPhenol	59	62	69	62
7	4-bromo Phenol	4-bromo-2-bromoPhenol	65	69	69	75
8	1,4 dihydroxy benzene	2-bromobenzene-1,4-diol	66	68	66	63
9	1-Naphthol	2- bromo -1-Naphthol	69	67	66	66
10	benzaldehyde	3- bromobenz- aldehyde	65	70	71	72
11	1-(3-OH phenyl)ethano ne	1-(3-OH-4-bromo phenyl)ethanone	65	68	69	72
12	4-NO ₂ Phenol	4- NO ₂ -2- bromo Phenol	58	62	56	65
13	2-OH benzaldehyde	5-bromo-2-OH- benzaldehyde	64	64	66	67
14	4-OH benzaldehyde	3-bromo-2-OH- benz- aldehyde	69	70	68	72
15	Toluene	2- bromo -Toluene	61	64	61	69
16	2-OH- benzoic acid	3-bromo- 6-OH- benzoic acid	69	68	68	65
17	2-OH- aniline	3- bromo- 4-OH- aniline	59	66	52	62

Table -2

S.No	Substrate	Product	Conventional KI+IQCC	Conventional KI+IQDC	Microwave Assisted KI+IQCC	Microwave Assisted KI+IQDC
1	Phenol	4- Iodo Phenol	64	66	66	69
2	o-cresol	4- Iodo -2-methylphenol	62	68	69	60
3	p-cresol	2- Iodo -4-methylphenol	69	66	66	62
4	m-cresol	4- Iodo -3-methylphenol	70	68	66	60
5	4-chloro Phenol	4-chloro-2- Iodo Phenol	65	69	66	68
6	2-chloro Phenol	2-chloro-4- Iodo Phenol	69	65	66	70
7	4-Bromo Phenol	4- Bromo -2- Iodo Phenol	67	65	67	67
8	1-Naphthol	2- Iodo -1-Naphthol	69	68	68	67
9	benzaldehyde	3- Iodobenz- aldehyde	68	70	68	69
10	4-NO ₂ Phenol	4- NO ₂ -2- Iodo Phenol	69	62	69	65
11	2-OH benzaldehyde	5-Iodo- 2-OH-benz- aldehyde	67	64	65	70
12	4-OH benzaldehyde	3-Iodo- 2-OH-benz- aldehyde	68	70	65	66
13	Toluene	2- Iodo -Toluene	69	67	66	69
14	2-OH- benzoic acid	3- Iodo- 6-OH- benzoic acid	69	68	69	66
15	2-OH- aniline	3- Iodo- 4-OH- aniline	55	59	55	60

E. Spectral data of certain compounds

- 1) 4-Bromophenol: ¹H NMR δ 5.3 (s, 1H, -OH), 6.72(d, 2H, Ar-H), 7.33 (d, 2H, Ar-H); m/z = 173
- 2) 2-Bromo-4-nitrophenol: ¹H NMR δ 6.16 (s, 1H, -OH), 7.8 (d, 1H, Ar-H), 8.07 (dd, 1H, Ar-H), 8.42 (d, 1H, Ar-H); m/z = 218
- 3) 2-Bromo-4-aminophenol: ¹H NMR δ 5.7 (s, 1H, -OH), 4.13 (s, 2H, -NH₂), 6.42-6.53 (m, 3H, Ar-H); m/z = 188
- 4) 4-Bromoanisole: ¹H NMR δ 3.78 (s, 3H, -OCH₃), 7.33 (d, 2H, Ar-H), 6.79 (d, 2H, Ar-H); m/z = 187
- 5) 2-Bromo-4-methylanisole: ¹H NMR δ 2.27 (s, 3H, CH₃), 3.83 (s, 3H, -OCH₃), 6.79 (d, 1H, Ar-H), 7.02 (dd, 1H, Ar-H), 7.34 (d, 1H, Ar-H). m/z = 201
- 6) 5-Bromo-2-hydroxy benzaldehyde: ¹H NMR δ 10.80 (s, 1H, -OH), 9.83 (s, 1H, -CHO), 7.66 (d, 1H, Ar-H), 7.51 (dd, 1H, Ar-H), 6.64 (d, 1H, Ar-H); m/z = 201
- 7) 2-Bromo-4-methylphenol: ¹H NMR δ 2.30 (s, 3H), 5.30 (s, 1H, -OH), 6.79 (d, 1H, Ar-H), 7.01 (d, 1H, Ar-H), 7.25 (d, 1H, Ar-H); m/z = 187
- 8) 4-Bromoaniline: ¹H NMR δ 3.70 (brs, 2H, -NH₂), 6.54 (d, 2H, Ar-H), 7.24 (d, 2H, Ar-H); m/z = 172
- 9) 2-Bromo-4-Chloro phenol: ¹H NMR δ 7.43 (d, 1H, Ar-H), 7.16 (dd, 1H, Ar-H), 6.94 (d, 1H, Ar-H), 5.49 (s, 1H, -OH); m/z = 207
- 10) 1-Bromo 2- Naphthol: ¹H NMR δ 5.10 (s, 1H, -OH), 6.6-8 (m, 6H, Ar-H); m/z = 223
- 11) 4-Iodo Phenol : ¹H NMR δ = 7.48 (d, J=7.98 Hz, Ar, 2H), 6.42 (d, J=7.98 Hz, Ar, 2H), 5.45 (br, s, -OH, 1H). (m/z = 220)
- 12) 2-iodo- 1-naphthol: ¹H NMR δ 7.93 (d, 1H, 8-H), 7.69 (d, 2H, 4-H and 5-H), 7.46 (t, 1H, 7-H), 7.26 (t, 1H, 6-H), 7.15 (d, 1H, 3-H) 5.24 (s, 1H, OH); m/z:270
- 13) 4-Iodo-anisole: ¹H NMR δ 3.79 (s, 3H, -OMe), 6.66 (d, 2H, Ar-H), 7.53 (d, 2H, Ar-H); m/z = 234
- 14) 4-Iodo toluene: ¹H NMR δ 2.3 (s, 3H, -Me), 6.9 (d, 2H, Ar-H), 7.55 (d, 2H, Ar-H); m/z = 218
- 15) 4-iodo-3-methylphenol: ¹H NMR δ = 7.46 (d, J=8.08 Hz, Ar, 1H), 6.7 (s, Ar, 1H), 6.38 (dd, J=2.9, 2.9 Hz, Ar, 1H), 2.35 (s, -CH₃, 3H) 5.45 (br, s, -OH, 1H). (m/z = 234)

- 16) 4-iodo-2-nitrophenol: $^1\text{H NMR}$ δ = 5.42 (s, -OH, 1H), 8.40 (s, Ar, 1H), 7.8 (dd, J=2.2, 2.4 Hz Ar, 1H), 6.74 (d, J=8.8 Hz, Ar, 1H). m/z = 265
- 17) 2-chloro-4-iodophenol: Isolated yield = 87%; $^1\text{H NMR}$ δ (300 MHz, CDCl_3): δ = 7.6 (d, J= 1.47Hz, Ar, 1H), 7.45 (dd, J=2.2, 1.5 Hz Ar, 1H), 6.78 (d, J=8.08 Hz, Ar, 1H), 5.5 (s, -OH, 1H).
- 18) 2-iodo-4-nitrophenol: Isolated yield = 64%; $^1\text{H NMR}$ δ (300 MHz, CDCl_3): δ = 8.61 (d, J= 1.8Hz Ar, 1H), 8.22 (dd, J= 2.26, 3.12 Hz Ar, 1H), 7.11 (d, J=9.06 Hz Ar, 1H).

IV. CONCLUSIONS

In summary, the author has successfully demonstrated halogenation reactions with eco friendly hypervalent Cr(VI) reagents Viz., Isoquinolinium Chlorochromate (IQCC), Isoquinolinium dichromate (IQDC) in presence of potassium halide (KBr/KI) under acid free (small amount of KHSO_4) conditions. Reaction times reduced from four (4) hours to less than 10 min. 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 halogenation reactions.

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