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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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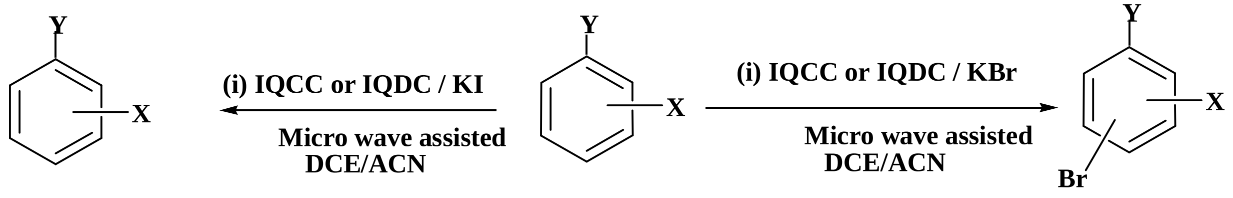
GRAPHICAL ABSTRACT



**Where Y= OH, $NH_2$ and $NHCOCH_3$ ;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), $KHSO_4$***

## I. INTRODUCTION

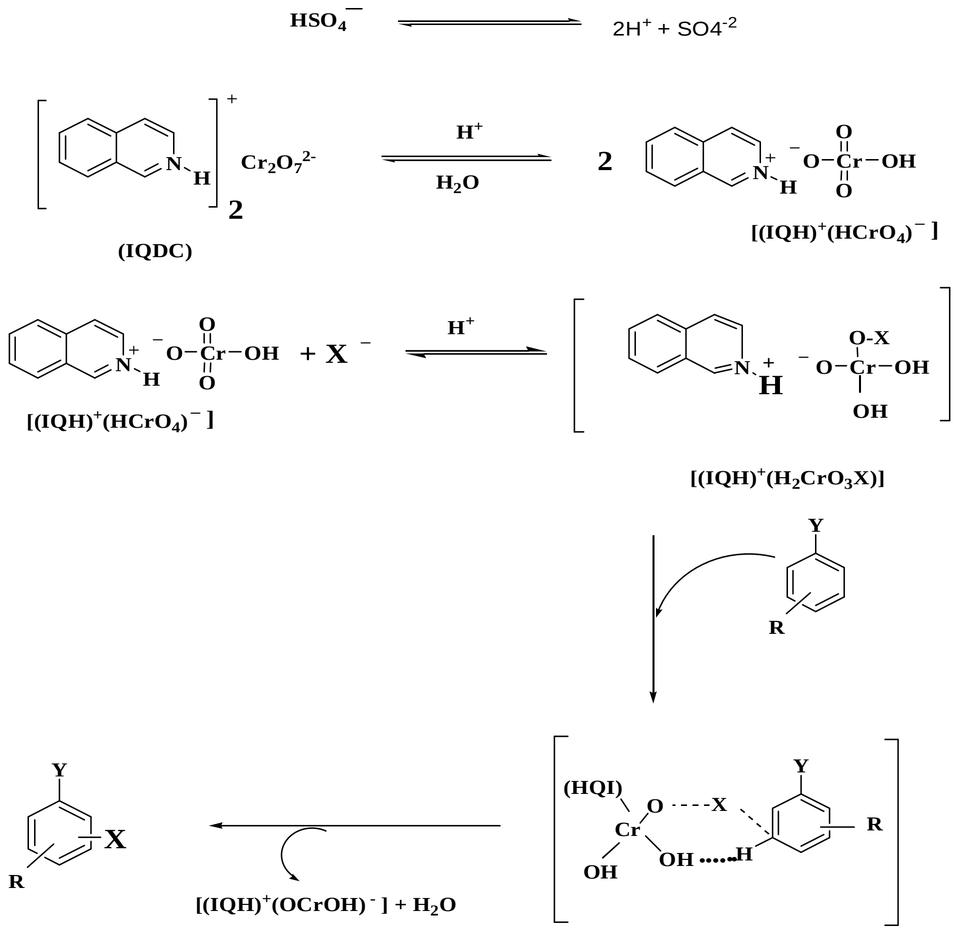
Halogenation of aromatic compounds is an area of evergreen interest to chemists among electrophilic aromatic substitution (EAS) reactions because organic halides can be converted efficiently into other functional groups by simple chemical transformations [1-3]. Numerous industrially valuable products 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 $KBrO_3$) and mineral acid are essential components for the generation of bromonium ion. . In $H_2O_2$-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-$H_2SO_4$-$H_2O$ 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 (bromine/iodine) several methods have been tried in the past several years. Oxyhalogenation protocols were among such efforts through which smooth halogenation could be achieved. Use of "onium halo chromates and dichromates" was among the few most effective oxidizing and oxyhalogenation reagents [13]. This could be probably due to the mildness, stability, selectivity, ease of handling, simple work-up, and regeneration of the reagent used in the work. On the other hand, during the past few decades, enormous interest has been paid for the development of environmentally safe and economically viable methods in organic synthesis in the lines of Green Chemistry [14]. One such effort is the drive for the development of Microwave assisted eco friendly halogenations trigger/accelerate the progress of reactions. Earlier reviews and publications [15- 21] in this field prove the importance of ultrasonically assisted organic synthesis and also highlight that this technique is not only simple; but also satisfies both economical and environmental demands. Present investigation is aimed at exploring the possibility of Isoquinolinium chlorochromate (IQCC) and Isoquinolinium dichromate (IQDC) as catalysts to achieve oxidative bromination/iodination of certain aromatic compounds with various organic compounds.

## II. RESULTS AND DISCUSSION

The reactions afforded fairly good product yields. The products were characterized by IR, $^1$H-NMR, Mass spectra and physical data with authentic samples and found to be satisfactory. The yields of major products are compiled in Tables 1 and 2. It is observed that the halogenations of aromatic compounds is moderately slow under conventional conditions with three to four hours of reaction time for completion. But under microwave the reaction shown in **Scheme-1**. It is believed that the active "IQ bound chromic acid" species is more selective than chromic acid, because the reactions aromatic substrates underwent only mono halogenation through the formation of cyclic intermediate with aromatic compound. On the basis of similar magnitudes of rate and product yields (Tables 1 and 2), almost a similar mechanism of halogenation could be envisaged for IQCC (Isoquinolinium chlorochromate) triggered reactions through the formation of "Protonated IQCC" due the protonation of IQCC, supplemented by the dissociation of bisulphate ion in the pre dissociation step.

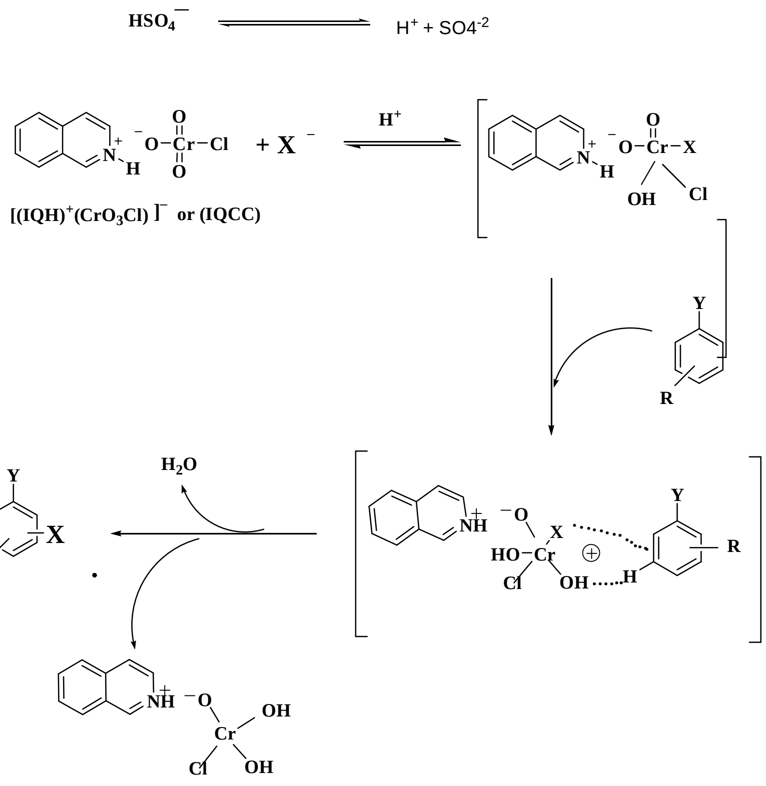
Scheme-1: IQDC triggered halogenation mechanism.



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 isoquinoliniumdi chromate [(C9H7NH+)2 Cr2O72-] 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 cons tan 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 1and 2.

Table -1

| S.No | Substrate | Product | Convensional KBr+IQCC | Convensional KBr+IQDC | Microwave Assisted KBr+IQCC | Microwave Assisted IQCC 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)ethanone | 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 | Convensional KI+IQCC | Convensional 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:* $^1$H NMR $\delta$ 5.3 (s, 1H, -O*H*), 6.72(d, 2H, Ar-*H*), 7.33 (d, 2H, Ar-*H*); m/z = 173
2) *2-Bromo-4-nitrophenol*: $^1$H NMR $\delta$6.16 (s, 1H, -O*H*), 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*: $^1$H NMR $\delta$ 5.7 (s, 1H, -O*H)*, 4.13 (s, 2H, -N$H_2^{)}$, 6.42-6.53 (m, 3H, Ar-*H)*; m/z = 188
4) *4-Bromoanisole*: $^1$H NMR $\delta$ 3.78 (s, 3H, -OC$H_3$), 7.33 (d, 2H, Ar-*H*), 6.79 (d, 2H, Ar-*H*); m/z = 187
5) *2-Bromo-4-methylanisole*: $^1$H NMR $\delta$ 2.27 (s, 3H, C$H_3$), 3.83 (s, 3H, -OC$H_3$), 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*: $^1$H NMR $\delta$ 10.80 (s, 1H, -O*H*), 9.83 (s, 1H, -C*H*O), 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*: $^1$H NMR $\delta$ 2.30 (s, 3*H*), 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*: $^1$H NMR $\delta$ 3.70 (brs, 2H, -N$H_2$), 6.54 (d, 2H, Ar-*H*), 7.24 (d, 2H, Ar-*H*); m/z = 172
9) *2-Bromo-4-Chloro phenol*: $^1$H NMR δ7.43 (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- Napthol*: $^1$H NMR δ5.10 (s, 1H, -OH), 6.6–8 (m, 6H, Ar-H); m/z = 223
11) *4-Iodo Phenol : $^1$H 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: $^1$H 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, O*H*); m/z:270
13) *4-Iodo-anisole: $^1$H NMR* δ3.79 (s, 3H, -OMe), 6.66 (d, 2H, Ar-H), 7.53 (d, 2H, Ar-H); m/z = 234
14) *4-Iodo toluene: $^1$H 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, -$CH_3$, 3H) 5.45 (br, s, -OH, 1H). ). ( m/z = 234

*16)* *4-iodo-2-nitrophenol*: 1H 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%; 1H NMR δ (300 MHz, CDCl3): 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%; 1H NMR δ (300 MHz, CDCl3): 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 then 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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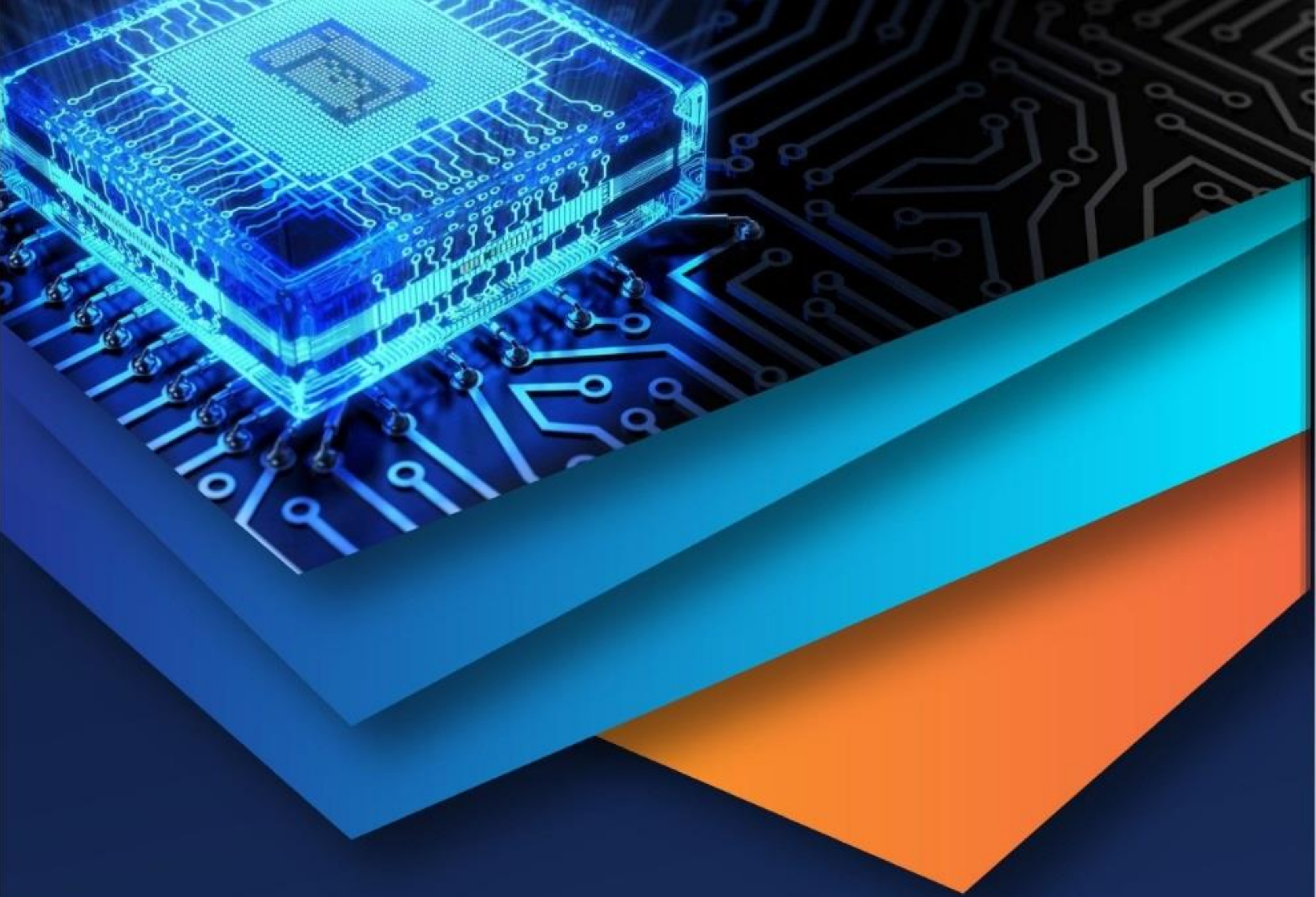

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