



REGULAR ARTICLE

Synthesis of meso-substituted corroles and porphyrins using iodine as a catalyst

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Abstract. Different types of corroles and porphyrins are synthesized from substituted aldehydes and pyrrole. The current synthetic method involves iodine as catalyst and proceeds at room temperature itself. By varying the amounts of reactants (i.e., pyrrole and aldehydes), the corrole and porphyrins were obtained in good to excellent yields. These products were characterized by $^1\text{H-NMR}$, UV-visible, and HRMS techniques. The reaction approach utilizes the readily available pyrrole and substituted aldehydes as starting materials and makes this reaction highly attractive in diversity-oriented synthesis.

Keywords. Pyrrole; aldehydes; iodine; corrole; corrin; porphyrin.

1. Introduction

Porphyrins are the class of tetrapyrrolic macrocycles comprising of four pyrrole rings that are linked through the meso-carbon atoms at meso-positions.¹ These are comprised of 18π electronic conjugated system in which 18π electrons are delocalized over 20 atoms.² It also contains 22π electrons, but their 18π electrons form a planar structure and are responsible for their aromaticity. Metalloporphyrin ring is common in a variety of important biological system where it is responsible for the activity of that system.³ Porphyrins are colored in nature and are characterized by their characteristic UV-visible absorption pattern. Recently, the porphyrins and metalloporphyrins were used in photodynamic therapy,⁴ solar cells,⁵ chemical sensors,⁶ catalysts,⁷ dyes and pigments,⁸ liquid crystals⁹, etc. Corroles are fully aromatic tetra-pyrrolic organic macrocyclic compounds that are analogues of porphyrins.¹⁰ It is a 19-carbon containing compound (one less than porphyrin), which possesses 18π conjugated electronic system.¹¹ Similar to other porphyrins and their analogues, corroles are aromatic in nature.¹² However, corrole's skeleton possesses a direct pyrrole–pyrrole linkage¹³ at each meso-carbon

atom of porphyrin skeleton. Moreover, in solutions, corroles are present in acid–base equilibria and lead to the formation of cationic or anionic species by reacting with acid or base. The presence of one imine-like and three pyrrole-like nitrogen donor atoms make them more acidic (stronger acid) than their porphyrin and corrin analogues.¹⁴

Due to the alteration of ligands structure, the transition metal complexes of corroles show very different properties than the analogous metalloporphyrins. As compared to the diprotonic porphyrins, corroles behave as tetradentate trianionic ligands toward transition metal ions.¹⁵ Thus, it helps them to stabilize the unusually high oxidation states, such as cobalt(IV),¹⁶ iron(VI),¹⁷ and cobalt(V).¹⁸ Currently, metallated corroles are used in biochemical applications such as sensor, antioxidant therapy, solar cells, diabetic therapy, anti-cancer agents,¹⁹ as a catalyst for different oxidation reactions²⁰ and generally used as a potential sensitizer in photodynamic cancer therapy.²¹

First corrole was synthesized in 1965 by Johnsen *et al.*²² following which, corroles were synthesized by various methods with the help of pyrrole and aldehydes²³ under different reaction conditions such as Al_2O_3 (in microwave),^{24–26} AcOH (by refluxing),²⁷ TFA (with

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solvent CH_2Cl_2),²⁸ HCl (with solvent, H_2O and MeOH),²⁹ neat TFA,³⁰ etc. These products are oxidized by 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ) or *p*-chloronil. Recently, corroles are also synthesized in microwave at high temperature in which iodine is used instead of acid.³¹ Furthermore, corroles are also synthesized under solvent-free conditions using electron deficient aldehydes.³² These methods required high temperature and strong organic acids. During the synthesis of corroles by pyrrole and aldehydes via these methods, porphyrins are obtained as a major product and corroles as byproduct.³³ Therefore, there is a need to develop an efficient method for the synthesis of corroles.

Recently, our research group had developed newer efficient methodologies for the synthesis of different types of porphyrinoids such as porphyrins,³⁴ calix[4]pyrroles,³⁵ porphodimethene,³⁶ calixphyrins,³⁷ etc. We had also successfully synthesized different core-modified porphyrinogens, 5-porphomethenes, 5,10-porphodimethenes, 5,15-porphodimethenes and porphotrimethenes.³⁸ In continuation of our ongoing research, we have developed a newer efficient and simple method for the synthesis of porphyrinoids and their analogues. Here, we have reported the iodine-catalyzed synthesis of corroles and porphyrins from pyrrole and aldehydes in dichloromethane for 5–10 min. This reaction does not require high temperature but proceeds at room temperature itself. The easily assessable 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ) is used as an oxidant in the current reaction. This method does not require organic acids, extremely toxic chemicals, or costly catalysts for the synthesis of corroles.

2. Experimental

Pyrrole, substituted benzaldehydes, iodine, DDQ and solvents (like acetonitrile, methanol, dichloromethane (CH_2Cl_2) and chloroform (CHCl_3)) were purchased from Spectrochem and these analytic grade solvents were dried before their use. To confirm the formation of compounds, JEOL 400 MHz $^1\text{H-NMR}$ spectrometer was utilized, and tetramethylsilane (TMS) was taken as an internal standard solvent. The UV-visible spectrum was recorded by Perkin-Elmer LAMBDA 950 high-performance UV-visible spectrophotometer.

2.1 General procedure for the synthesis of A3-corrole (3a–f)

For the synthesis of corrole, a round-bottom flask with N_2 saturated dichloromethane (10 mL) was used in which

substituted benzaldehydes (1 mmol) and freshly distilled pyrrole (2 mmol) were added and stirred at room temperature. To this reaction mixture, instead of acid, iodine was added (as a catalytic amount) and the color change of the reaction mixture was observed. Further, the round-bottom flask was covered with foil paper and continued stirring for 5–10 min. Progress of the reaction was monitored with the help of thin layer chromatography (TLC) (i.e., if no reactant remains). Following which, DDQ (1 mmol) was added and the stirring was continued for another 5 min. The product (corrole) was separated by basic alumina column chromatography with hexane/chloroform solution. The compound was solid in nature with purple color and corroles were obtained in high yield with 1:2 molar ratio in the process.

2.2 General procedure for the synthesis of porphyrin (4a–f)

This synthetic procedure for the synthesis of porphyrin is similar to the above process. The only difference between this and the above process is the equimolar ratio of reactants (substituted benzaldehydes and pyrrole) and solvent quantity. In this process, substituted benzaldehydes (1 mmol) and pyrrole (1 mmol) were used in equimolar ratio 1:1 with 20 mL chloroform.

2.3 5,10,15-triphenylcorrole (TPC) (3a)

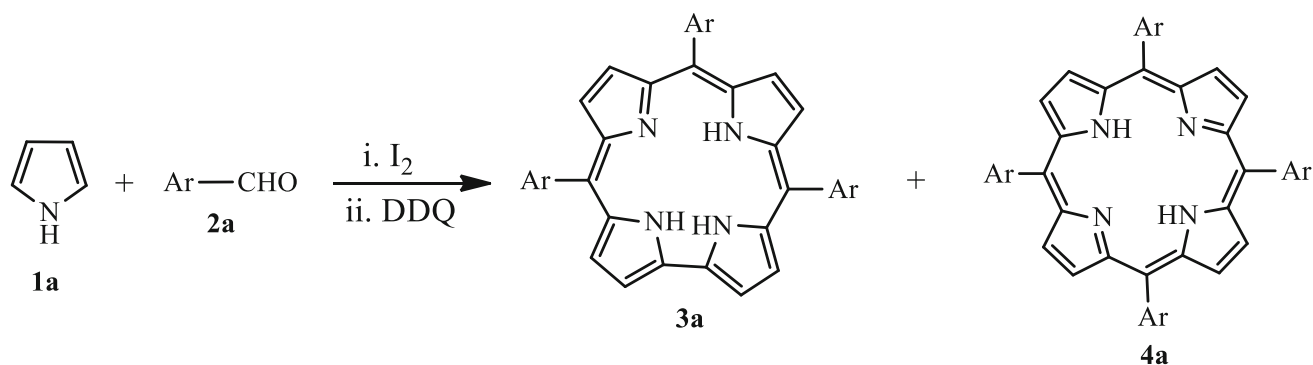
UV-Vis (CHCl_3): λ_{max} ($\log \epsilon$) = 415.10 (1.12), 523.89 (0.08), 575.23 (0.17), 615.17 (0.13), 646.45 nm (0.11). $^1\text{H-NMR}$ (CDCl_3 , 400 MHz): δ = 8.83 (s, 4H), 8.50 (s, 4H), 8.33 (d, 4H), 8.15 (d, 2H), 7.85–7.70 (m, 9H), –2.90 (s, 3H). MS (ESI): m/z = calculated ($\text{C}_{37}\text{H}_{26}\text{N}_4$), 526.63; observed ($\text{M}+\text{H}$)⁺ 527.59.

2.4 5,10,15-Tris(pentafluorophenyl)corrole (PFC) (3b)

UV-Vis (CHCl_3): λ_{max} ($\log \epsilon$) = 407.75 (0.87), 561.98 (0.15), 604.65 nm (0.09), $^1\text{H-NMR}$ (CDCl_3 , 400 MHz): δ = 9.13–9.12 (d, 2H), 8.78–8.77 (d, 2H), 8.60–8.58 (d, 4H), –2.83 (s, 3H). MS (ESI): m/z = calculated ($\text{C}_{37}\text{H}_{11}\text{F}_{15}\text{N}_4$), 796.49; observed ($\text{M}+\text{H}$)⁺ 797.08.

2.5 5,10,15-Tris(2,6-dichlorophenyl)corrole (DPC) (3c)

UV-Vis (CHCl_3): λ_{max} ($\log \epsilon$) = 409.82 (0.85), 422.03 (0.79), 566.09 (0.10), 605.30 (0.04), 633.09 nm (0.02). $^1\text{H-NMR}$ (CDCl_3 , 400 MHz): δ = 8.92 (d, 2H), 8.51 (d, 2H), 8.36 (m, 4H), 7.74 (m, 6H), 7.35 (m, 3H), –1.72 (s, 3H);

Table 1. Optimization of reaction conditions for the iodine-catalyzed corrole (**3a**) and porphyrin (**4a**) synthesis from pyrrole (**1a**) and benzaldehyde (**2a**).

Pyrrole/aldehyde ratio ^a		Solvent	Yield of corrole (3a) ^b (%)	Yield of porphyrin (4a) ^b (%)
1	2:1	CH₂Cl₂	27	6
2	2:1	CHCl ₃	25	6
3	2:1	MeOH	10	4
4	2:1	Acetonitrile	25	6
5	2:1	MeOH + CH ₂ Cl ₂	22	5
6	2:1	THF	2	<1
7	1:1	CH₂Cl₂	0	24
8	1:1	CHCl ₃	0	22
9	1:1	MeOH	0	9
10	1:1	Acetonitrile	0	22
11	1:1	MeOH + CH ₂ Cl ₂	0	15
12	1:1	THF	0	1

The accelerate catalytic system which is further applied in table 2 to synthesize more derivatives are in bold.

^aReaction proceeds at room temperature.

^bIsolated yield.

MS (ESI): m/z = calculated (C₃₇H₂₀N₄Cl₆), 729.98; observed (M+H)⁺ 730.96.

2.6 5,10,15-Tris(4-chlorophenyl)corrole (4-ClPC) (**3d**)

UV-Vis (CHCl₃): λ_{max} (log ε) = 416.24 (0.68), 514.67 (0.05), 575.25 (0.13), 616.87 (0.09), 647.89 (0.07). ¹H-NMR (CDCl₃, 400 MHz) δ = 8.99 (d, 2H), 8.85 (d, 2H), 8.27 (m, 4H), 8.13 (d, 2H), 7.76 (m, 6H), -2.91 (s, 3H); MS (ESI): m/z = calculated (C₃₇H₂₃N₄Cl₃), 628.10; observed (M+H)⁺ 629.12.

2.7 5,10,15-Tris(4-bromophenyl)corrole (4-BrPC) (**3e**)

UV-Vis (CHCl₃): λ_{max} (log ε) = 417.96 (1.10), 515.81 (0.05), 577.75 (0.13), 615 (0.09), 648.65 (0.08) nm. ¹H-NMR (CDCl₃, 400 MHz) δ = 9.02 (d, 2H), 8.86 (d, 4H) 4 Hz), 8.63 (m, 4H), 8.27 (d, 4H), 7.92–8.16 (m, 6H), -2.80 (s, 3H).

2.8 5,10,15,20-Tetraphenylporphyrin (TPP) (**4a**)

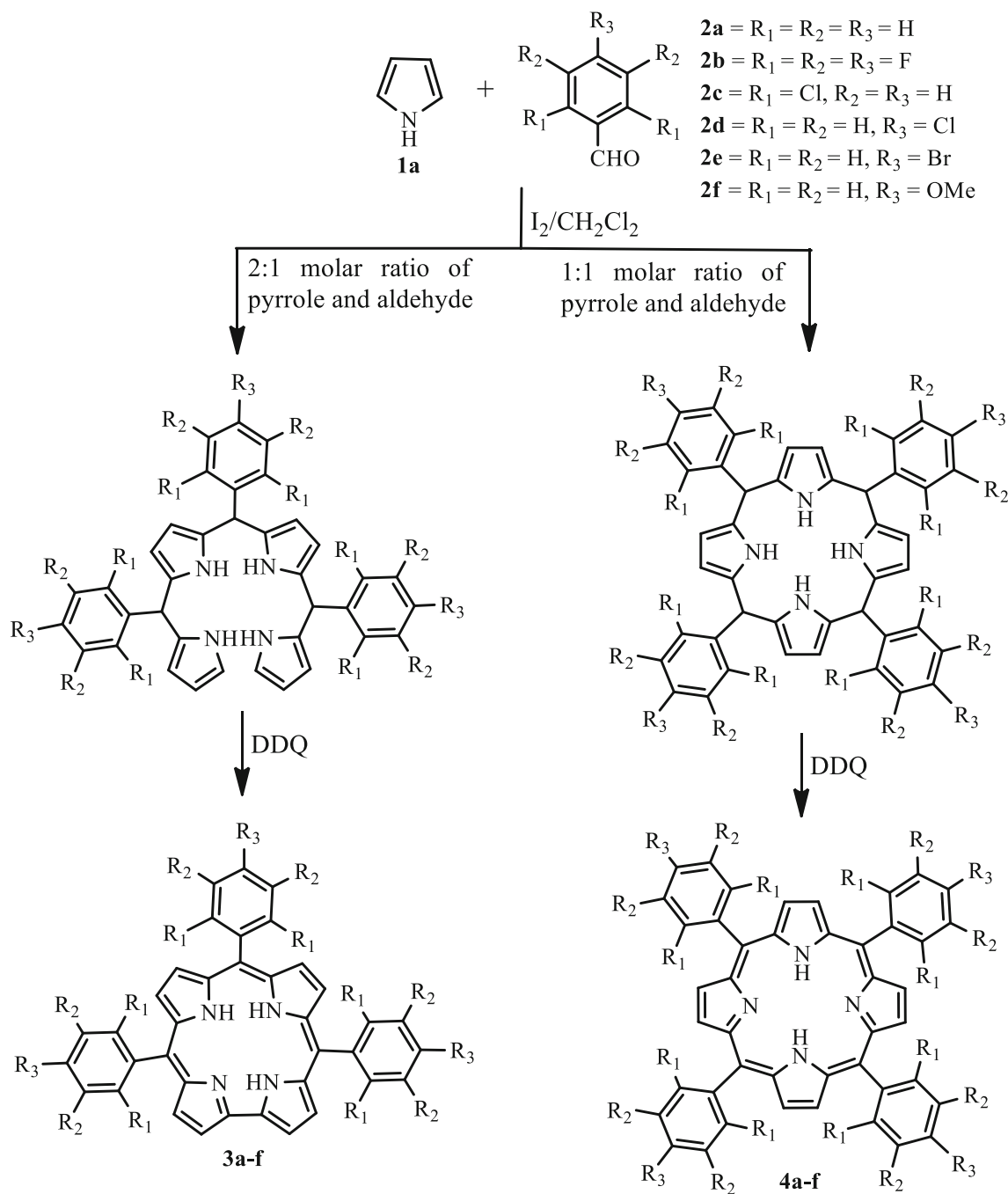
UV-Vis (CHCl₃): λ_{max} (log ε) = 415.86 (1.74), 515.11 (0.06), 549.88 (-0.01), 590.12 (-0.02), 648.63 (-0.01) nm, ¹H-NMR (CDCl₃, 400 MHz): δ = 8.84 (s, 8H), 8.22–8.20 (d, 8H), 7.77–7.72 (d, 12H), -2.78 (s, 2H).

2.9 5,10,15,20-Tetra(pentafluorophenyl)porphyrin (PFP) (**4b**)

UV-Vis (CHCl₃): λ_{max} (log ε) = 412.60 (0.78), 506.82 (0.01), 586.02 (0.003), 655.05 nm (0.001). ¹H-NMR (CDCl₃, 400 MHz): δ = 8.95 (s, 8 H), -2.90 (s, 2 H).

2.10 5,10,15,20-Tetra(2,6-dichlorophenyl)porphyrin (DCP) (**4c**)

UV-Vis (CHCl₃): λ_{max} (log ε) = 418.29 (1.35), 512.65 (0.04), 555.10 (0.02), 592.37 (0.003), 658.20 (0.002) nm, ¹H-NMR (CDCl₃, 400 MHz); δ = 8.69 (s, 8 H), 7.82–7.78 (m, 8 H), 7.73–7.68 (m, 4 H), -2.65 (s, 2 H).



Scheme 1. Iodine-catalyzed synthesis of substituted corroles (**3a-f**) and porphyrins (**4a-f**) from pyrrole (**1a**) and aldehydes (**2a-f**) in dichloromethane under nitrogen atmosphere at room temperature.

2.11 5,10,15,20-Tetra(4-chlorophenyl)porphyrin (4-CIPP) (**4d**)

UV-Vis (CHCl_3): λ_{max} ($\log \epsilon$) = 419.02 (1.44), 515.16 (0.07), 550.22 (0.03), 590.18 (0.02), 647.73 (0.02) nm, $^1\text{H-NMR}$ (CDCl_3 , 400 MHz): δ = 8.83 (s, 8H), 8.13–8.10 (d, 8H), 7.74–7.72 (d, 8H), –2.89 (s, 2H).

2.12 5,10,15,20-Tetra(4-bromophenyl)porphyrin (4-BrPP) (**4e**)

UV-Vis (CHCl_3): λ_{max} ($\log \epsilon$) = 421.53 (1.60), 515.22 (0.08), 550.33 (0.04), 590.36 (0.03), 647.20 (0.02) nm, $^1\text{H-NMR}$ (CDCl_3 , 400 MHz): δ = 8.82 (s, 8H), 8.06–8.04 (d, 8H), 7.89–7.87 (d, 8H), –2.91 (s, 2H).

Table 2. Synthesis of substituted corroles (**3a–f**) and substituted porphyrins (**4a–f**) from pyrrole (**1a**) and aldehydes (**2a–f**) in dichloromethane under nitrogen atmosphere.

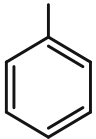
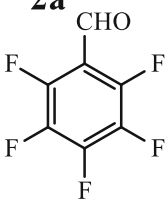
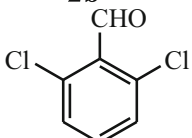
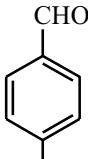
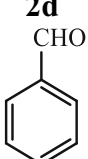
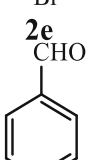
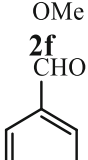
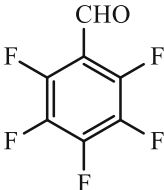
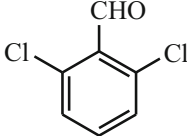
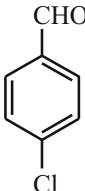
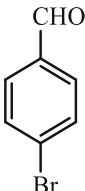
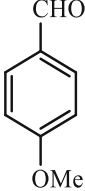
	Pyrrole	Aldehyde	Product	Yield (%)	Reference
1 ^a	1a	CHO 	3a	27	29, 32
2 ^a	1a	2a CHO 	3b	35	32
3 ^a	1a	2b CHO 	3c	31	29, 32
4 ^a	1a	2c CHO 	3d	30	32
5 ^a	1a	2d CHO 	3e	28	29
6 ^a	1a	2e CHO 	3f	–	
7 ^b	1a	2f CHO 	4a	24	31, 41

Table 2. (contd)

	Pyrrole	Aldehyde	Product	Yield (%)	Reference
8 ^b	1a	 2b	4b	34	39
9 ^b	1a	 2c	4c	31%	39
10 ^b	1a	 2d	4d	29	32, 41
11 ^b	1a	 2e	4e	28	40
12 ^b	1a	 2f	4f	20	41

^a2:1 Molar ratio of pyrrole (**1a**) and aldehydes (**2a–f**).

^b1:1 Molar ratio of pyrrole (**1a**) and aldehydes (**2a–f**).

3. Results and Discussion

To establish an efficient catalytic system for the synthesis of corroles (**3a**) and porphyrins (**4a**), pyrrole (**1a**) and aldehydes were selected as a model to screen the reaction parameters (Table 1). The amount of reactants and solvents play crucial role in the synthesis of corroles and porphyrins. When pyrrole (**1a**) and aldehydes (**2a**) were taken in a molar ratio of 2:1 and reaction was catalyzed by iodine (0.1 equivalent) and followed by oxidation with DDQ, corrole (**3a**) was a major product.

However, the molar ratio 1:1 of pyrrole (**1a**) and aldehydes (**2a**) gave porphyrin (**4a**) as a major product. Different types of solvent (dichloromethane, methanol, acetonitrile, chloroform, tetrahydrofuran (THF), and mixture of dichloromethane and methanol) are provided in Table 1. From Table 1, it is observed that in dichloromethane, both corroles (entry 1) and porphyrins (entry 7) were obtained in maximum yields. These reactions were performed at room temperature without any heating (in microwave and refluxing) and the progress of the reaction was monitored via thin layer chromatography (TLC).

Thus, from Table 1, it is clear that the molar ratio 2:1 of pyrrole (**1a**) and aldehyde (**2a**) in CH₂Cl₂ produced corrole (**3a**) as a major product, whereas the molar ratio 1:1 of pyrrole (**1a**) and aldehyde (**1b**) in CH₂Cl₂ produced porphyrin (**4a**) as a major product. Both these reactions (synthesis of corrole and porphyrin) were performed under nitrogen atmosphere and was completed within 5–10 min. After getting the suitable catalytic system in hand, we further broadened the scope of the catalytic system by employing different substrates (Scheme 1). The results of the reaction are summarized in Table 2.

Different types of electron withdrawing and electron donating aldehydes (**2b–f**) along with pyrrole (**1a**) were employed as substrates in Scheme 1, and it was observed that most of the electron deficient aldehyde (**2b**) gave maximum yield. Further, the reactions were completed within 5–10 min with electron deficient aldehydes, whereas with electron donating aldehyde, reaction was prolonged up to 15 min. With electron donating aldehydes, only porphyrins were obtained as the final product. All the products were characterized by their ¹H-NMR, UV-visible and HRMS spectrometry, and these characterization data are fully satisfied with literature data. These reactions were constantly carried out at 1 mmol scale, and similar yields were also observed with 10 mmol scale.

4. Conclusion

In this study, we have developed an efficient, simple and convenient method for the synthesis of different corroles and porphyrins from easily available substrates. The current reaction did not require high temperature and proceeds at room temperature itself. The reaction requires minimal (0.1 mmol) amount of iodine as a catalyst. Simply by changing the amounts of the reactants, corroles and porphyrins can be obtained in good yield. The shorter reaction time, room temperature, simple work-up procedure, higher product yield, and easily assessable starting materials make the method better than the other reported methods.

Supplementary Information (SI)

Supplementary data of this article contains experimental details, synthetic procedures and characterization data, available at www.ias.ac.in/chemsci.

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