

A Simple and Convenient Method for Iodination of Some Aromatic Compounds by Pyridinium Iodochloride

Dr.Sandeep V.Khansole
Department of chemistry,Indira Gandhi Sr.College,
CIDCO,Nanded (MS) 431603

ABSTRACT.

Pyridinium iodochloride can be used for iodination of some aromatic compound. The pyridinium iodochloride has been found to be an efficient solid iodinating reagent with no hazardous effect and it can be handled safely. The advantages of this method include no catalyst, mild conditions, simple operation and short reaction time with excellent yield.

Key words: *pyridinium iodochloride, iodination, simple, ecofriendly method, efficient reagent.*

1. Introduction

In recent years, direct iodination methods have been intensively developed using iodine donating systems, such as iodine-tetrabutylammonium peroxydisulfate¹, BuLi-F₃CCH₂I², iodine-F-TEDA-BF₄³, iodine-iodine pentaoxide⁴, iodine-mercury salt⁵, and NaOCl-NaI⁶. In our publication we reported the iodination of aromatic compound by using iodine and iodic acid as an iodinating reagent^{7,8}.

However some of the existing methods have associated environmental hazards with respect to handling and storage of molecular iodine, strongly acidic conditions, less expensive and complex catalyst, toxic metallic compounds and rare oxidizing reagents that are difficult to prepare. Therefore there is a need for simple, less expensive and safer method for iodination of some selective aromatic compounds. We report here iodination of several aromatic compounds by using pyridinium iodochloride as iodinating agent.

2. Experimental section:

2.1 General procedure:

Melting points were determined in an open capillary tube and are uncorrected. The purity of compound has been checked by TLC. IR spectra were recorded in KBr on Perkin Elmer spectrometer. ¹H NMR were recorded in CDCl₃ on Gemini 200 NMR spectrometer using THS as an internal standard. Elemental analysis were carried out on Carlo Erba 1108 analyzer.

2.2 Preparation of pyridinium iodochloride

To a solution of pyridine (7.9 g, 0.1 mol) in acetic acid a solution of iodine monochloride (16g, 0.1 mol) in acetic acid was added slowly at 0°C with continuous stirring. The pale yellow solid obtained was filtered, dried and recrystallised by ethyl alcohol. The purity of reagent was checked by thin layer chromatography.

2.3 Procedure for iodination of aromatic compounds by pyridinium iodochloride

Aromatic compound (1 mmol) and pyridinium iodochloride (0.24 g, 1 mmol) were dissolved in methanol (15 ml) and refluxed on water bath for 2hr. The content was poured on ice cold water; the solid that got separated was filtered and recrystallised from ethanol. By using two equivalents of pyridinium iodochloride and one equivalent of substrate diiodinated product were obtained.

3. Result and discussion

To illustrate the action of proposed reagent we selected several reactive aromatic substrate like phenols, anilines, aldehydes and ketones. The reaction was carried out by heating aromatic compounds with pyridinium iodochloride on water bath for one hour using methanol as a solvent.

The isolated product indicated a regioselective iodination had occurred at the electron rich ortho or para position. When the ortho position was blocked, iodination only took place at para positions.

Iodination of aromatic compound with pyridinium iodochloride

Entry	Iodinated product	yield	Melting point	
			Found	Lit
1	4-IC ₆ H ₄ NH ₂	85	63	63 ⁹
2	2,4-I ₂ C ₆ H ₃ NH ₂	78	95	96 ⁹
3	2-Cl-4-IC ₆ H ₃ NH ₂	85	71	72 ⁹
4	3-I C ₆ H ₄ CHO	81	55	54 ¹⁰
5	3-I C ₆ H ₄ CO NH ₂	88	189	188 ¹⁰
6	3I- C ₆ H ₄ COOH	85	186	186 ¹⁰
7	3IC ₆ H ₄ NO ₂	79	36	37 ¹⁰

4. Conclusion

In summary, a simple and convenient method for the iodination of aromatic compound has been developed. The advantages include no catalyst, mild conditions, simple operation and short reaction time with excellent yield. Since the reagent is solid it can be easily weighted and has no hazardous effect.

5. Acknowledgement

Author wish to thank WRO UGC, Pune for the financial assistance under MRP

REFERENCES

- 1) S.G. Yang, Y.H. Kim Tetrahedron Lett., (1990) 40,6051
- 2) I.J. Blackmore, A.N. Boa, E.J. Murkay, M. Dannis, S. Woodward, Tetrahedron Lett., (1999) 40,6671
- 3) Z. Marko, I. Jerney, S. Stogam, Tetrahedron Lett., (19970) 38,6305.
- 4) L.C. Brazil, C.J. Cuttler, J. Org. chem., (1994) 59, 6233
- 5) A. Bachki, J. Foabelo, M. Yus, Tetrahedron, (1994) 50, 5139.
- 6) K.J. Edger, S.N. Fallling J. Org. Chem., (1990) 55,5287.
- 7) B.R. Patil, S.R. Bhusare, R.P. Pawar, Y.B. Vibhute, Tetrhedron Lett., (2005) 46,7179.
- 8) B.R. Patil, S.R. Bhusare, R.P. Pawar, Y.B. Vibhute, Arkivoc, (2006) 1,104.
- 9) M. Sosnowski, L. Skulski Molecule, (2002) 7,867-870
- 10) L. Kraszkiwicz, M. Sosnowski, L. Skulski, Synthesis (2006) 7, 1195-1199