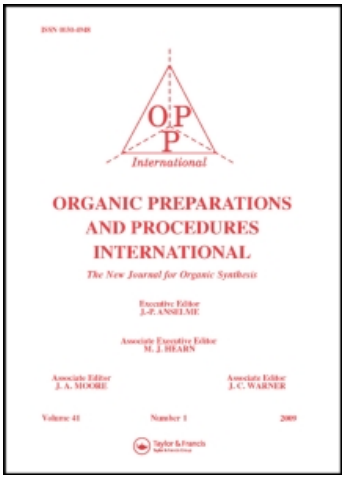

Organic Preparations and Procedures International
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t902189982

CATALYTIC SYNTHESIS OF METAL-FREE PHTHALOCYANINE

M. V. Twigg[a]
[a] Erindale College, University of Toronto, Toronto, Canada

To cite this Article Twigg, M. V.(1971) 'CATALYTIC SYNTHESIS OF METAL-FREE PHTHALOCYANINE', Organic Preparations and Procedures International, 3: 2, 97 — 98
To link to this Article: DOI: 10.1080/00304947109356043
URL: http://dx.doi.org/10.1080/00304947109356043

ORGANIC PREPARATIONS AND PROCEDURES INT. 3(2), 97-98 (1971)

CATALYTIC SYNTHESIS OF METAL-FREE PHTHALOCYANINE

M. V. Twigg
Erindale College, University of Toronto, Toronto 181, Canada

$$4C_6H_4(CN)_2 + \text{cyclohexylamine} \longrightarrow (C_8H_4N_2)_4H_2$$

The usual[1] two-stage synthesis of metal-free phthalocyanine involves the preparation of an easily hydrolysed metal phthalocyanine followed by its acid hydrolysis. The convenient single-stage synthesis described below produces a good yield of pure metal-free phthalocyanine directly from phthalonitrile and a catalytic quantity of cyclohexylamine in a high boiling solvent, thus avoiding the need for the preparation of a metal-containing intermediate. The method was evolved from a report[2] in the patent literature.

EXPERIMENTAL

A mixture of phthalonitrile (15.2g), cyclohexylamine (1 ml) and 1-methylnaphthalene (38 ml) is heated to about 10° below the boiling point (about 230°), under a reflux condenser. The clear liquid becomes brown, then green and purple crystals of product form. After heating for 10 hr., the mixture is cooled and the mat of crystalline product filtered off and washed well with the following solvents: benzene (4 x 100 ml) (brown washings), acetone (4 x 100 ml) (brown washings becoming

colourless), and ether (2 x 100 ml). Yield 10.1g (67%), found: C, 74.8; H, 3.4; N, 22.0%. Calculated for $C_{32}H_{18}N_8$: C, 74.7; H, 3.5; N, 21.8%. Higher yields can be obtained by using less solvent but the product is not so crystalline. 1-Chloronaphthalene, o-dichlorobenzene and other high boiling solvents may be substituted for 1-methylnaphthalene although yields are usually slightly lower.

REFERENCES

1. F.H. Moser and A.L. Thomas, "Phthalocyanine Compounds", chapter 3, Reinhold Publishing Corporation, New York, 1963.

2. W.L. Rintelman, U.S. Patent, 2485167, (1949); CA, 44, 10336i, (1950).

(Received February 5, 1971)