

This article was downloaded by:

On: 27 January 2011

Access details: *Access Details: Free Access*

Publisher *Taylor & Francis*

Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



## Organic Preparations and Procedures International

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t902189982>

### SYNTHESIS OF 4-ISOPROPYL-2,6-DINITROCHLOROBENZENE

Eddie V. P. Tao<sup>a</sup>; C. F. Christie Jr.<sup>a</sup>

<sup>a</sup> The Lilly Research Laboratories, Eli Lilly and Company, Indianapolis, Indiana

**To cite this Article** Tao, Eddie V. P. and Christie Jr., C. F.(1972) 'SYNTHESIS OF 4-ISOPROPYL-2,6-DINITROCHLOROBENZENE', *Organic Preparations and Procedures International*, 4: 2, 73 – 74

**To link to this Article:** DOI: 10.1080/00304947209458265

**URL:** <http://dx.doi.org/10.1080/00304947209458265>

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: <http://www.informaworld.com/terms-and-conditions-of-access.pdf>

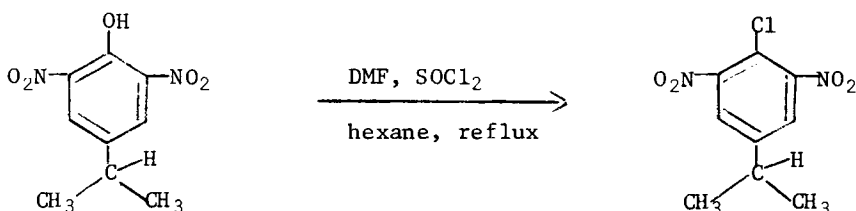
This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

SYNTHESIS OF 4-ISOPROPYL-2,6-DINITROCHLOROBENZENE

Eddie V. P. Tao and C. F. Christie, Jr.

The Lilly Research Laboratories  
Eli Lilly and Company  
Indianapolis, Indiana 46206



We would like to bring to your attention a convenient and efficient procedure for the preparation of 4-isopropyl-2,6-dinitrochlorobenzene by the treatment of 4-isopropyl-2,6-dinitrophenol with thionyl chloride and dimethylformamide. This method corresponds essentially to the one reported previously;<sup>1,2</sup> however, the yield and the quality of the product have been improved greatly by employing hexane as a solvent in the reaction. This modification provides a pure crystalline material directly from the reaction mixture without any purification and could also be utilized as a general synthesis for 4-alkyl-2,6-dinitrochlorobenzenes.

EXPERIMENTAL

4-Isopropyl-2,6-dinitrochlorobenzene.-Dimethylformamide (40 ml.) was added to a suspension of 4-isopropyl-2,6-dinitrophenol (452 g., 2 moles)

E. V. P. TAO AND C. F. CHRISTIE, JR.

in hexane (400 ml.). The reaction mixture was heated to 40-50°C, and thionyl chloride (333 g., 2.8 moles) was added dropwise over a period of 45 minutes. The reaction mixture was refluxed for 5 hours and cooled. Dilute sodium bicarbonate solution (5%) was added to neutralize the reaction mixture. The resulting mixture was stirred for thirty minutes and the product removed by filtration, washed with water, then hexane and air dried to yield 456 g. (93%) of 4-isopropyl-2,6-dinitrochlorobenzene, m.p. 61.5-64°. Anal. Calculated for C<sub>9</sub>H<sub>9</sub>ClN<sub>2</sub>O<sub>4</sub>: C, 44.58; H, 3.67. Found: C, 44.58; H, 3.81. The spectroscopic data are in accordance with the assigned structure. NMR (DMSO-<sub>d</sub><sub>6</sub>) δ 1.32 (d,6, J = 6.9 Hz, CH<sub>3</sub>), 3.21 (m,1, J = 6.9 Hz, CH), 8.37 (s,2, aromatic protons); mass spectrum M<sup>+</sup> 244, one chlorine atom.

#### REFERENCES

1. I. Matsumoto: *Yakugaku Zasshi* 85, 544 (1965).
2. C. S. Giam and R. C. Hall, *Organic Preparations and Procedures Int.*, 3, 155 (1971), and references cited therein.

(Received March 3, 1972; in revised form April 20, 1972)