

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>

PREPARATION OF 2-ISOCYANATO-5-CHLOROBENZOYL CHLORIDE

B. K. Misra^a; Y. R. Rao^a; S. N. Mahapatra^a

^a Regional Research Laboratory, Bhubaneswar, INDIA

To cite this Article Misra, B. K. , Rao, Y. R. and Mahapatra, S. N.(1981) 'PREPARATION OF 2-ISOCYANATO-5-CHLOROBENZOYL CHLORIDE', *Organic Preparations and Procedures International*, 13: 5, 363 — 365

To link to this Article: DOI: 10.1080/00304948109356141

URL: <http://dx.doi.org/10.1080/00304948109356141>

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.

REFERENCES

- † Convenient Syntheses of Heterocycles. 4. Previous papers see reference 7.
- 1 R. C. Elderfield, "Heterocyclic Compounds", Vol. 5, p. 547, Wiley, New York, N. Y., 1957.
 - 2 A. I. Meyers, "Heterocycles in Organic Synthesis", p. 255, Wiley, New York, N. Y., 1974.
 - 3 (a) J. B. Jepson, A. Lawson, and V. D. Lawton, *J. Chem. Soc.*, 1791 (1955), no description on the yields; (b) H. Muxfeldt, J. Behling, G. Grethe, and W. Rogalski, *J. Am. Chem. Soc.*, **89**, 4991 (1967).
 - 4 H. T. Clarke, J. R. Johnson, and R. Robinson, "The Chemistry of Penicillin", p. 848, Princeton University Press, Princeton, N. J., 1949.
 - 5 H. Behringer and H. W. Stein, *Chem. Ber.*, **82**, 209 (1949).
 - 6 A. Kjaer, *Acta Chem. Scand.*, **4**, 1347 (1950).
 - 7 (a) N. Suzuki and Y. Izawa, *Tetrahedron Lett.*, 1863 (1974); *Bull. Chem. Soc. Japan*, **49**, 3155 (1976); (b) N. Suzuki, T. Yamabayashi, and Y. Izawa, *ibid.*, **49**, 353 (1976).

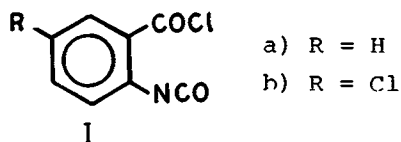
PREPARATION OF 2-ISOCYANATO-5-CHLOROBENZOYL CHLORIDE

Submitted by B. K. Misra, Y. R. Rao and S. N. Mahapatra*
(9/2/80)

Regional Research Laboratory
Bhubaneswar-751013, INDIA

In our studies^{1,2} on the reactions of 2-isocyanato benzoyl chlorides(I), we were required to prepare the 5-chloro-derivative(Ib). Its preparation (in 31% yield) from 6-chloro isatoic anhydride(IIb) by the action of thionyl chloride takes

three weeks time.³ We report here the preparation of 2-isocyanato-5-chlorobenzoyl chloride (Ib) in 75% yield by the chlorination of Ia following a modified procedure for the chlorination of phenyl isocyanate.⁴



EXPERIMENTAL

Into a stirred solution of 2-isocyanato benzoyl chloride (36.2 g; 0.2 M) in ethylene dichloride (110 g) at 25° was introduced a slow stream of dry chlorine for 5 min. Iodine (0.75 g in 30 g ethylene dichloride) was then added and chlorine addition (total 14.9 g; 5% excess) resumed during which period the temperature rose to 50-55°. After stirring for 2 hours at 25°, the solvent was evaporated and the residue treated with dry benzene (75 ml). The separated 6-chloro isatoic anhydride (1.64 g) was filtered out and benzene evaporated from the filtrate. The residue (45 g) on distillation under reduced pressure gave 2-isocyanato-5-chlorobenzoyl chloride at 115-118°/1.5 mm Hg, m.p. and m.m.p. 54-57°. Its IR spectrum was superimposable with that of an authentic sample prepared from Ib and PMR spectra of its reaction products² confirm the substitution pattern.

REFERENCES

1. B. K. Misra, Y. R. Rao and S. N. Mahapatra, *Indian J. Chem.*, **18B**, 19 (1979).
2. B. K. Misra, Y. R. Rao and S. N. Mahapatra, *Indian J. Chem.*, **19B**, 908 (1980).

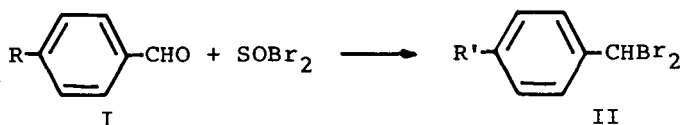
3. N. P. Peet and S. Sunder, *J. Org. Chem.*, 40, 1909 (1975).
4. F. M. Julien and H. T. Seifen, U.S.2,974,163 (1961); *Chem. Abstr.*, 55, 14381 (1961).

A NEW SYNTHESIS OF 4-BROMOMETHYLBENZAL BROMIDE
AND 1,4-bis(DIBROMOMETHYL)-BENZENE

Submitted by S. D. Saraf
(1/12/81)

Department of Chemistry
Kuwait University, KUWAIT

Although two different methods for the preparation of 4-bromomethylbenzal bromide IIa have been studied, a one-step synthesis of this compound in good yield has yet to be achieved. In one such method, *p*-xylene was brominated at 130° to yield a mixture of brominated products from which IIa was isolated in 23% yield.^{1,2} Drefahl and Plotner³ isolated the same product after treatment of *p*-tolualdehyde with phosphorous tri-bromide in carbon disulphide followed by bromination of the intermediate product, 4-methylbenzal bromide, at 140° in the presence of a powerful source of light.



a) R = CH₃ b) R = CHO

a) R' = CH₂Br b) R' = CHBr₂

The other compound 1,4-bis(dibromomethyl)benzene (IIb) has been synthesized by various research groups, but a high yield synthesis of this compound under ordinary conditions is yet to be realized. In one such method^{4,5} dry bromine was