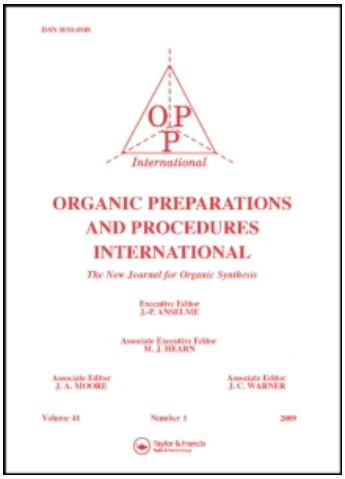


Organic Preparations and Procedures International
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t902189982

A CONVENIENT PROCEDURE FOR THE PREPARATION OF 2-BROMO-1-PHENYLETHANOL

S. S. Bhosale[a]; P. L. Joshi[a]; A. S. Rae[ab]
[a] National Chemical Laboratory, Pune, INDIA [b] Indian Institute of Chemical Technology, Hyderabad, INDIA

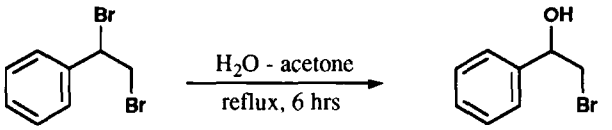
To cite this Article Bhosale, S. S. , Joshi, P. L. and Rae, A. S.(1992) 'A CONVENIENT PROCEDURE FOR THE PREPARATION OF 2-BROMO-1-PHENYLETHANOL', Organic Preparations and Procedures International, 24: 6, 695 — 696
To link to this Article: DOI: 10.1080/00304949209356251
URL: http://dx.doi.org/10.1080/00304949209356251

A CONVENIENT PROCEDURE FOR THE PREPARATION OF 2-BROMO-1-PHENYLETHANOL[§]

Submitted by (07/16/92) S. S. Bhosale, P. L. Joshi and A. S. Rao*†

National Chemical Laboratory, Pune 411008, INDIA

2-Bromo-1-phenylethanol (**1**) is a useful intermediate in organic synthesis.[1-3] It can be converted to the antibiotic chloramphenicol *via* β-bromostyrene.[4-6] In connection with some synthetic studies, large amounts of **1** were needed and we have found the normally used method,[2,7] employing reaction of styrene with N-bromosuccinimide in moist DMSO to be unsatisfactory for the preparation of **1**, since the yields are moderate; a different route[1] based on the reaction of styrene with Br_2-KBr-H_2O also furnishes the bromohydrin **1** in moderate yields. We now report that heating dibromide **2** in aqueous acetone results in selective solvolysis of the benzylic bromine to furnish the bromohydrin **1** in 93% yield;[8] the dibromide **2** has been prepared quantitatively from styrene according to a known method.[9]



2 **1**

EXPERIMENTAL SECTION

IR spectra were recorded on a Perkin-Elmer 599B infrared spectrophotometer. ^1H NMR spectra were obtained on a Varian T-60 instrument with TMS as internal standard. Bps are uncorrected.

2-Bromo-1-phenylethanol (1).- A mixture of dibromide **2** (5.5 g, 2.08 mmol),[9] acetone (30 mL) and water (140 mL) was heated under reflux for 6 hrs. Most of the acetone was removed on a rotary evaporator. The residue was diluted with water (100 mL) and extracted with ether (50 mL x 3). The combined ethereal extracts were washed with water (50 mL x 2) and dried (Na_2SO_4). The residue obtained after evaporation of solvent was distilled *in vacuo* to furnish 3.91 g (93% yield) of bromohydrin **1**, bp. 110-111°/2 mm, (lit[7] bp. 110-111°/2 mm). ^1H NMR (CCl_4): δ 3.17 (1H, s, OH disappears after D_2O exchange), 3.40 (2H, m, $-CH_2Br$), 4.77 (1H, dd, J = 4, 8Hz, -CHOH), 7.27 (5H, s, Ar-H). The identity of the solvolysis product was further confirmed by comparing its IR spectrum and TLC behavior with those of an authentic sample of **1**.[7]

We have also observed that the bromohydrin **1** can be prepared in 93% yield if acetone is replaced by methyl ethyl ketone or dioxane.

REFERENCES

§ NCL Communication No. 5435

† Present address: Indian Institute of Chemical Technology, Hyderabad 500007, INDIA

1. S. Arai, T. Takeuchi, M. Ishikawa, T. Takeuchi, M. Yamaozaki and M. Hida, *J. Chem. Soc., Perkin Trans. 1*, 481 (**1987**).

2. A. R. Katritzky, S. I. Bayyuk, N. Dennis, G. Musumarra and E. Wurthwein, *ibid.*, 2535 (**1979**).

3. J. Das, *Synth. Commun.*, **18**, 907 (1988).

4. S. Zee and S. Chou, *Proc. Nat. Sci. Council, Republic of China, Part B*, **7**, 394 (1983).

5. O. Cervinka, V. Dudek, A. Fabryova, J. Kolar, J. Lukac, J. Simon and M. Viktorin, *Collect. Czech., Chem. Commun.*, **54**, 2748 (1989).

6. B. G. Hazra, V. S. Pore, S. P. Maybhate, M. V. Natekar and A. S. Rao, *Synth. Commun.*, **19**, 1763 (1989).

7. D. R. Dalton, J. B. Hendrickson and D. Jones, *Chem. Commun.*, 591 (**1966**); A. W. Langman and D. R. Dalton, *Org. Synth.*, **59**, 16 (1980).

8. S. S. Bhosale, M. V. Natekar, P. L. Joshi, K. N. Dixit, A. S. Vaidya and A. S. Rao, *Indian Patent*, 166181 (1990); *C.A.*, **114**, 163712v (1991).

9. M. S. Newman, B. Dhawan, M. M. Hashem, V. K. Khanna and J. M. Springer, *J. Org. Chem.*, **41**, 3925 (1976).

****

A CONVENIENT PREPARATION OF 4-CYCLOPROPYLPHENOL

Submitted by Bruce W. Horrom and H. Mazdiyasni*
(05/26/92)

Process Research, Pharmaceutical Products Division
Abbott Laboratories, D-45L/AP10, Abbott Park, IL 60064

4-Cyclopropylphenol, an important intermediate[1] for the synthesis of agricultural and pharmaceutical products, has been obtained by a variety of reported methods[2,3] which however, are inefficient and low yielding. We now describe a variant of the Boissier[3] procedure which provides a facile and direct route to the title compound in an overall yield of 86% from 4-cyclopropylacetophenone with