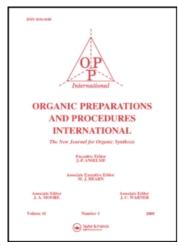
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# A CONVENIENT PROCEDURE FOR THE PREPARATION OF 2-BROMO-1-PHENYLETHANOL

S. S. Bhosale<sup>a</sup>; P. L. Joshi<sup>a</sup>; A. S. Rae<sup>ab</sup>

 $^{\rm a}$  National Chemical Laboratory, Pune, INDIA  $^{\rm b}$  Indian Institute of Chemical Technology, Hyderabad, INDIA

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# A CONVENIENT PROCEDURE FOR THE PREPARATION OF 2-BROMO-1-PHENYLETHANOL<sup>§</sup>

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.<sup>1-3</sup> It can be converted to the antibiotic chloramphenicol *via* β-bromostyrene.<sup>4-6</sup> In connection with some synthetic studies, large amounts of 1 were needed and we have found the normally used method,<sup>2,7</sup> 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<sup>1</sup> based on the reaction of styrene with Br<sub>2</sub>-KBr-H<sub>2</sub>O 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;<sup>8</sup> the dibromide 2 has been prepared quantitatively from styrene according to a known method.<sup>9</sup>

### **EXPERIMENTAL SECTION**

IR spectra were recorded on a Perkin-Elmer 599B infrared spectrophotometer. <sup>1</sup>H 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), 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<sub>2</sub>SO<sub>4</sub>). 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<sup>7</sup> bp. 110-111°/2 mm). <sup>1</sup>H NMR (CCl<sub>4</sub>):  $\delta$  3.17 (1H, s, OH disappears after D<sub>2</sub>O exchange), 3.40 (2H, m, -CH<sub>2</sub>Br), 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.<sup>7</sup>

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

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## 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).
- O. Cervinka, V. Dudek, A. Fabryova, J. Kolar, J. Lukac, J. Simon and M. Viktorin, Collect. Czech., Chem. Commun., 54, 2748 (1989).
- B. G. Hazra, V. S. Pore, S. P. Maybhate, M. V. Natekar and A. S. Rao, Synth. Commun., 19, 1763 (1989).
- 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).

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#### A CONVENIENT PREPARATION OF 4-CYCLOPROPYLPHENOL

Submitted by (05/26/92)

Bruce W. Horrom and H. Mazdiyasni\*

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

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