

# PHASE TRANSFER CATALYSIS THROUGH **"ORGANIC SYNTHESES" GLASSES**

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## Phase Transfer Catalysis (PTC) – general and extremely useful method

organic phase



- Method for performing ion neutral partner reactions
- Heterogeneous two phase (organic and inorganic) system, tetraalkylammonium salt as a catalyst
- Anionic species being continuously introduced into the organic phase
- Increases yield and speed of many reactions
- Allows to avoid use of solvents and strong bases

### DOI:10.15227/orgsyn.055.009

### Organic Syntheses, Coll. Vol. 6, p.897 (1988); Vol. 55, p.91 (1976)

PHASE-TRANSFER ALKYLATION OF NITRILES: 2 PHENYLBUTYRONITRILI



itted by M. Makosza1 and A. Jonczy ecked by Harold W. Wagner and Richard E. Benson

1. Procedure

lving henzene should be carried out in a well-ve

A 3-1., four-necked, round-bottomed flask equipped with a mechanical stirrer, a dropping funnel. and an efficient reflux condenser is charged with 540 ml. of 50% aque g, (253 ml., 2.20 moles) of phenylacetonitrile (Note 1), and 5.0 g, (0.022 mole) of chloride (Note 2). Stirring is begun, and 218 g. (150 ml., 2.00 moles) of e romide (Note 3) is added dropwise over a period of approximately 100 minutes at 28-35°. I necessary, the flask may be cooled with a cold-water bath to keep the temperature of the mixture at 28 After the addition of ethyl bromide is complete, stirring is continued for 2 hours, then the ture is increased to 40° for an additional 30 minutes. The reaction mixture is cooled to 25°, 21. (20.3 ml., 0.200 mole) of benzaldehyde (Note 4) is added, and stirring is continued for 1 hour. T k is immersed in a cold-water bath, and 750 ml. of water and 100 ml. of benzene are added. Th rated, and the aqueous phase is extracted with 200 ml. of benzene. The organic layers a mbined and washed successively with 200 ml. of water, 200 ml. of dilute hydrochloric acid (Note 5 and 200 ml. of water. The organic layer is dried over anhydrous magnesium sulfate, and the solvent moved by distillation under reduced pressure. The product is distilled through a Vigreux column giving 225-242 g. (78-84%) of 2-pheny utyronitrile, b.p. 102–104° (7 mm.),  $n_{\rm p}^{25}$ 

### The checkers used phenylacetonitrile obtained from Aldrich Chemical Company, Inc., and distilled before use. It may also be purified according to the directions given in Org. Synth., Coll. Vol. 1, 10 ium chloride is available from Fisher Scientific Company. The preparation this reagent is described in *Org. Synth.*, **Coll. Vol. 6**, 232 (1988). 3. Ethyl bromide (available from Fisher Scientific Company) was distilled before use. 4. Benzaldehyde (available from Fisher Scientific Company) was distilled before use. It is added at this oint to convert any unreacted phenylacetonitrile to the high-boiling α-phenylcinnan The acid solution was prepared by adding 1 volume of acid to 5 volumes of wate 6. The checkers obtained a forerun of 7–12 g of product having $n_D^{25}$ 1.5065–1.5066. 7. The $\alpha$ -phenylcinnamonitrile (Note 4) present in the distillation flask can be recovered. The residue is

broken up with 75 ml. of methanol, the mixture stirred and cooled, and the product recovered by filtration. Recrystallization from methanol gives 17–20 g. of crystalline material, m.p. 86–88°. The <sup>1</sup>H



- Publishes carefully checked procedures
- Preparation of compounds of general interest
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### **PTC in "Organic Syntheses"**

- Abstracts reviewed, considering conditions and character of the transformation

