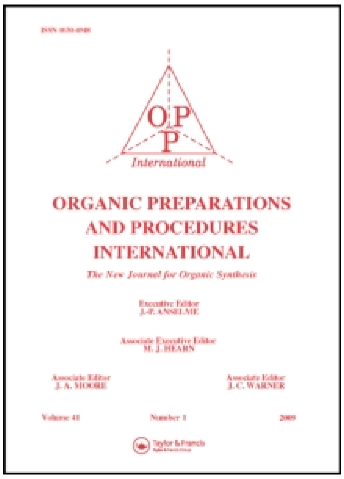

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t-BUTYL PHENYL CARBONATE

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6. After ca. 15 minutes a dark brown solid is formed on the top of the reaction mixture so that more vigorous stirring is required.
7. The yields are improved by using ethanol free chloroform. Although there has been no accident so far, the reaction should be performed behind a safety shield (Ag_2O, R-I, $CHCl_3$!) and in a well-ventilated fume hood (alkyl iodides are suspected to be carcinogenic).

t-BUTYL PHENYL CARBONATE

Submitted by John W. Scott* and David Parker
(9/4/79)

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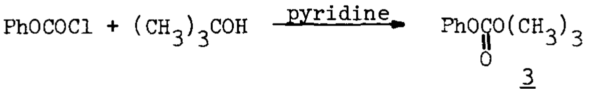
In contrast to a previous report [J. Am. Chem. Soc., 79, 98 (1957)], pyridine has been found to be an excellent base for the preparation of t-butyl phenyl carbonate (93-95%).



$$\mathrm{PhOCOCl} + (\mathrm{CH_3})_3\mathrm{COH} \xrightarrow{\text{pyridine}} \mathrm{PhO}\underset{\overset{\|}{\mathrm{O}}}{\mathrm{C}}\mathrm{O(CH_3)_3}$$

3

PROCEDURE

t-Butyl Phenyl Carbonate (3).- A stirred mixture of 1.103 ℓ. (868 g., 11.72 moles) of t-butyl alcohol (Matheson Coleman and Bell), 938 ml. (924 g., 11.65 moles) of pyridine (Fisher Scientific Company) and 1.75 ℓ. of methylene chloride was cooled with a tap water bath as 1.82 kg (11.62 moles) of phenyl chloroformate (PPG Industries, Inc.) was added at a rate such that the temperature remained ≤28° (5.5 hrs); small amounts of ice were added to the bath to help control the exotherm. The water bath was left in place as stirring was continued for an additional 3 hrs. The suspension was allowed to stand overnight and filtered to remove the pyridine hydrochloride, which was washed with methylene chloride. The combined filtrates were washed with H_2O and a total of 3 ℓ. of 5% HCl and dried over $MgSO_4$. The solvent was removed on a rotary evaporator at 35-40° and the residual liquid (2.258 kg) was distilled through a 12" Vigreux column. After removal of a small forerun, the main fraction, 2.115 kg (93.5% yield), was collected as a colorless liquid, bp. 71-74°/0.2 mm, n_D^{24} 1.481. The material was homogeneous to GC and no additional peaks were observed in its NMR spectrum.

AN IMPROVED SYNTHESIS OF 2,2'-DIPYRIDYL-1-OXIDE

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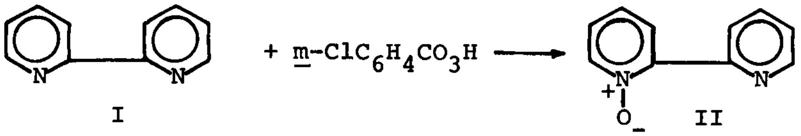
2,2'-Dipyridyl-1-oxide (II),[1,2] previously isolated as a brown hygroscopic solid,[2,3] from the oxidation of 2,2'-dipyridyl (I) with 30% hydrogen peroxide in acetic acid, can be obtained in 50-55% yield from the oxidation of I with m-chloroperbenzoic acid[4] in chloroform.



PROCEDURE

To a stirred ice-cold chloroform (15 ml.) solution of 2,2'-dipyridyl (1.99 g., 12.8 mmol) was added over a period of 2 hrs. a solution of 2.98 g. (1.72 mmol) of m-chloroperbenzoic acid (85% peracid) in 60 ml. of