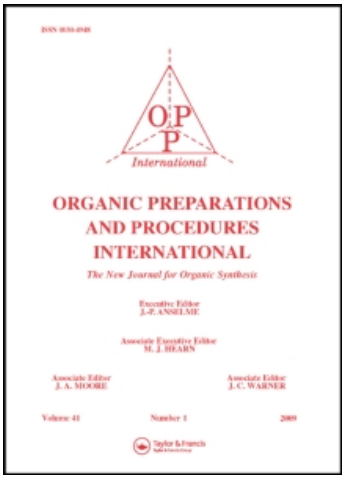

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AN IMPROVED METHOD FOR THE PREPARATION OF DIPHENYLACETYLENE

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Several methods have been reported for the preparation of diphenylacetylene(I).[1-3] The most recent procedure[1] involving the oxidation of benzil dihydrazone using cuprous chloride and oxygen in pyridine gives a high yield(96.6%) of I. However, the preparation of benzil dihydrazone requires 60 hours. The dehydrohalogenation of dibromostilbene in dimethylsulfoxide(DMSO) gave a 93% yield of I after recrystallization, in a total reaction time of about one hour and is stilbene-free.

$$PhCHBrCHBrPh + NaOCH_3 \longrightarrow Ph\text{-}C{\equiv}C\text{-}Ph \quad (I)$$

Reactions carried out with sodium ethoxide, potassium t-butoxide or potassium hydroxide as the base gave lower yields of I, which is often contaminated with trans-stilbene.

EXPERIMENTAL

Diphenylacatylene.- Into a 250ml. Erlenmeyer flask were added 10 g. (0.03 mole) of stilbene dibromide,[3] 10 g. (0.19 mole) of sodium methoxide

and 100 ml. of dry dimethylsulfoxide. The sodium methoxide had been prepared by treating sodium with excess genuine methanol and removing the excess methanol under vacuum. The dimethylsulfoxide was dried over molecular sieves. The Erlenmeyer flask was stoppered, placed in a 60° water bath and stirred with a magnetic stirrer for 1 hr. The reaction mixture was poured into 150 ml. of cold water and filtered. After air drying, 5.4 g. of slightly yellow diphenylacetylene was obtained. The product was dissolved in 15 ml. of hot methanol and the small amount of insoluble white solid was removed by gravity filtration. Upon thorough cooling, 5.0 g. (93%) of pure diphenylacetylene, mp. 60-61°, lit.[2] mp. 60-61°, was isolated. Its nmr spectrum showed it to be free of cis- or trans-stilbene.

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* To whom correspondence should be addressed.

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