

PYROPHORIC LEAD AS A NEW AGENT OF WURTZ-LIKE REACTIONS

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The limited synthetic value of the Wurtz and Wurtz-Fittig reactions is well known. One of the reasons restricting the applicability of these methods is the too high reactivity of sodium, which prevents the use of halides other than those of hydrocarbons and ethers, and gives rise to various undesired side reactions.

The coupling of the carbon residues of two molecules of an organic halide can be achieved, according to our present work, by the use of pyrophoric lead. Such lead is conveniently prepared in the reaction of lead oxide with furfural vapours at 290° / 1, 2 /. Pyrophoric lead may be purified by washing it with benzene, alcohol and water, and stored under the same solvents. Immediate use, however, without any further purification is often advantageous.

In our experiments the organic halide to be coupled was dissolved in a non-halide solvent of b.p. below 80°, such as

benzene and alcohol, or in water, containing the pyrophoric lead, and the reaction was effected by shaking the mixture at room temperature, or by refluxing.

In this way, as examples, the following syntheses have been achieved: n-octane from n-butyl bromide / in 44 % yield /; diphenylethane from benzyl chloride / 51 % /; diacetyl from acetyl chloride / 53 % /; benzil from benzoyl chloride /56 %/; succinic acid from chloroacetic acid / 49 % /; adipic acid from 3-chloropropionic acid / 47 % /; and the dehalogenated dimer from Chlorpromazine / 16 % /. Though the reaction to obtain biphenyl from chlorobenzene failed, the corresponding dinitrodiphenyl derivatives could be prepared from o-, m- and p-nitrochlorobenzene /in 25%, 36%, and 31% yield, respectively/.

These examples illustrate the scope of applicability of pyrophoric lead in organic syntheses. The yields given above have been obtained in preliminary experiments.

Further work is in progress to find the optimum conditions of each reaction, to explore the use and limitations of this Wurtz-like synthesis, and to elucidate the similarity or differences in its mechanism as compared with the use of sodium.

REFERENCES

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