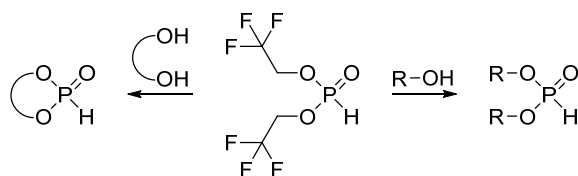


MICROWAVE ASSISTED SYNTHESIS OF H-PHOSPHONATES

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H-phosphonates have the pronounced capability to act as nucleophiles as well as electrophiles, thereby making them versatile reagents in organic synthesis. The synthesis of H-phosphonates is typically accomplished by direct substitution of phosphorus trichloride, either in a tert-butanolysis reaction, with excess alcohol or in the presence of a base [1]. During our investigations we noticed that the reaction with PCl_3 , especially for H-phosphonates with a dioxaphospholane scaffold, does not proceed in satisfactory yield and purity. In regard to this we developed a method utilizing bis-(2,2,2-trifluoroethyl) phosphite (BTFEP) in a transesterification reaction under microwave irradiation.



This method enables the convenient and quick synthesis of various cyclic H-phosphonates and dialkyl phosphites by simple alcoholysis under non-inert and additive-free conditions. Reaction times in the realm of 30 min and 1 mmol scale makes it ideal for screening purposes in a laboratory environment. This method permits the economic usage of alcohol and opens a pathway to a facile synthesis for this type of phosphorus species.

[1] For an Overview, see: Sobkowski, M.; Kraszewski, A.; Stawinski, J. Recent Advances in H-Phosphonate Chemistry. Part 1. H-Phosphonate Esters: Synthesis and Basic Reactions. In *Phosphorus Chemistry II: Synthetic Methods*; Montchamp, J.-L., Ed.; Springer International Publishing: Cham, 2015; pp 137–177.