Protection of functional groups plays an important role in multistep organic synthesis. In many preparations of delicate organic compounds, some specific parts of their molecules cannot survive the reagents deployed or chemical environments of different reactions. Thus, these parts or groups must be protected. In this work, selective protection of 1,2-diols was achieved in water as a solvent by using a commonly known water-stable coupling reagent, 4- (4,6- dimethoxy -1,3, 5- triazin -2-yl) -4 - methylmorpholinium chloride (DMTMM), as a coupling reagent in the presence of a catalytic amount of 4-dimethylaminopyridine (DMAP) with good to excellent results.

Various catalysts were initially screened, with dialkyltin catalysts being found most suitable for this process. In addition, a series of bases were evaluated, and weak inorganic bases such as K2C03, CS_2C0_3 and Na2C03 emerged as the best for the reaction For selective monoprotection by benzoylation, benzoyl chloride or benzoic acid was alternatively used; The study also surveyed various benzoyl chloride derivatives with electron-withdrawing and electron-donating groups at *meta*, *para* and *ortho* positions as alternative reagents for selective monoprotection of 1,2-diols with benzoyl chloride derivatives with substituent at *meta* and *para* positions emerging as excellent protecting groups. Other protections groups like 2-thenoyl chloride and phenyl acetyl chloride have been surveyed. The work was concluded with the method being successfully applied to selectively benzoylate some symmetric or asymmetric cyclic and acyclic 1,2-diol and 1,3-diols. The structures of different monobenzoylated compounds were elucidated by proton and carbon nuclear magnetic resonance (NMR), infra red (IR) spectroscopy and mass spectrometry (MS).