SUMMARY

This study reports the stepwise synthesis of F_2-BODIPY compounds and their derivatives, aimed at developing fluorescent tertiary phosphines and phosphonium salts. These compounds have potential as precursors in organometallic chemistry, particularly for applications related to functional materials and complex molecular design. The F_2-BODIPY core compound (**3**) was obtained through a condensation reaction between 3-ethyl-2,4-dimethylpyrrole and 4-bromobenzaldehyde under a nitrogen atmosphere, catalyzed by trifluoroacetic acid, followed by oxidation with DDQ and complexation with $BF_3{\cdot}OEt_2$, yielding the product in 37% yield. The compound's identity was confirmed by NMR spectroscopy, indicated by a triplet signal at δ 0.78 ppm ($^{11}B\{^{19}F\}$, J = 33.4 Hz) and a quartet at δ –145.81 ppm ($^{19}F\{^{11}B\}$, J = 32.9 Hz), along with consistent ^{1}H and ^{13}C NMR data corresponding to the F_2-BODIPY structure. The compound was efficiently purified via hot extraction with hexane, eliminating the need for column chromatography.

Subsequently, compound **3** underwent a Pd-catalyzed reaction with diethyl phosphite to produce the phosphonate derivative (**4**), identified by a characteristic signal at δ 17.6 ppm in the $^{31}P\{^{1}H\}$ NMR spectrum and a doublet of doublets pattern at δ 7.88 and 7.37 ppm in the ^{1}H NMR spectrum due to coupling with the phosphorus nucleus. Transformation into Me_2-BODIPY phosphonate (**5**) was achieved through a Grignard reaction with methylmagnesium bromide, resulting in fluorine loss (confirmed by the disappearance of the $^{19}F\{^{1}H\}$ signal), the appearance of a new singlet at δ –0.74 ppm in the $^{11}B\{^{1}H\}$ NMR, and a methyl signal at δ 0.19 ppm in the ^{1}H NMR. Further reduction with $LiAlH_4$/TMSCl yielded the primary phosphine (**6**), marked by a characteristic signal at δ –122.3 ppm in the $^{31}P\{^{1}H\}$ NMR spectrum, showing a triplet of triplets with coupling constants 1J_PH = 202.7 Hz and 3J_PH = 7.1 Hz, indicating P–H bond formation.

The primary phosphine was then converted into a dichlorophosphine (6b), displaying chemical shifts at δ 159.5 ppm (in toluene) and δ 161.1 ppm (in THF) in the $^{31}P\{^{1}H\}$ NMR spectrum. A subsequent reaction with 4-methoxyphenylmagnesium bromide produced the tertiary phosphine (**7**), which showed a singlet at δ –9.8 ppm ($^{31}P\{^{1}H\}$ NMR) and a methoxy singlet at δ 3.71 ppm (^{1}H NMR), consistent with the proposed structure. The final step involved the synthesis of phosphonium salt (**8**) via reaction with α,α′-dibromo-p-xylene, producing a $^{31}P\{^{1}H\}$ signal at δ 23.9 ppm and characteristic ^{1}H NMR signals, including an aromatic doublet at δ 5.30 ppm and a methylene singlet at δ 4.40 ppm, confirming the formation of the phosphonium salt structure.

Keywords: BODIPY, Fluorescent, Tertiary Phosphine, Phosphonium Salt