CHAPTER V

CONCLUSION

The synthesis route of [2-(1-propylbutyl)-1,3-dioxolan-4-yl]methyl sulfamate proceeded through 3 step: the formation of 2- propylpentanal, 2-(1-propylbutyl)-4-hydroxymethyl-1,3-dioxolane and [2-(1-propylbutyl)-1,3-dioxolan-4-yl]methyl sulfamate respectively.

2-Propylpentanal could be obtained from 2 different methods, e.g. either from the reaction of 4-heptylmagnesium chloride with triethyl orthoformate or propylmagnesium bromide with ethyl ethoxyacetate. The latter gave much higher yield of aldehyde than the former. The difficulty and incompleteness of the formation of 4-heptylmagnesium chloride may be responsible for the unsatisfying result of the former method.

The NMR-spectroscopic data indicated that the condensation of 2-propylpentanal with glycerol gave a mixture of 2-(1-propylbutyl)-4-hydroxymethyl-1,3-dioxolane and 2-(1-propylbutyl)-5-hydroxy-1,3-dioxane, each existed in *cis*-, and *trans*-forms, which could not be separated. An attempt to synthesize pure five-membered cyclic acetal derivative was performed from the condensation of glycerol

2-monobenzoate with 2-propylpentanal. However the attempt was not successful because the product obtained was still contaminated with a small amount of the six-membered cyclic acetal derivative.

The reaction of the condensation product of 2-propylpentanal and glycerol with sulfamoyl chloride which was generated from chlorosulfonyl isocyanate and formic acid gave the desired sulfamate derivative. The NMR-spectroscopic data showed that the sulfamate product was a mixture of *cis*-,*trans*-pair of [2-(1-propylbutyl)-1,3-dioxolan-4-yl]methyl sulfamate and one form of [2-(1-propylbutyl)-1,3-dioxan-5-yl]sulfamate which was proposed to be *cis*-form. The mixed sulfamate derivatives could not be separated as well.