

PHOTOCHEMICAL CYCLIZATION OF EPOXY-ALCOHOL
A NEW SYNTHESIS OF SUGIRESINOL DIMETHYL ETHER

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Photochemical cyclization of 1,3-bis(4-methoxyphenyl)-4,5-epoxy-1-pentanol gave sugiresinol dimethyl ether in good yield.

It is known that the photochemical alcoholysis of oxiranes gives alkoxy-alcohols in good yield.^{1,2} Tokumaru¹ reported that the photochemical alcoholysis of cyclohexene oxide was initiated by the photoexcitation of the oxirane group. In the photolysis of propylene oxide, Gritter and his co-workers³ reported the C-1-O to C-2-O cleavage ratio is about 25:1. In order to find a new synthetic route to 3-hydroxytetrahydropyran, the photochemical cyclization of epoxy-alcohol⁴, 1,3-bis(4-methoxyphenyl)-4,5-epoxy-1-pentanol (I), was examined. This paper describes a new synthesis of sugiresinol dimethyl ether (II)⁵ via photochemical cyclization.

Reaction of 4,4'-dimethoxychalcone with vinylmagnesium bromide in

the presence of cuprous chloride gave 1,3-bis(4-methoxyphenyl)-4-penten-1-one (III)⁶ in 91% yield, which was reduced with sodium borohydride to give a stereoisomeric mixture of 1,3-bis(4-methoxyphenyl)-4-penten-1-ol (IV) in 85% yield. Treatment of IV with *m*-chloroperbenzoic acid gave a stereoisomeric mixture of I in 61% yield. Irradiation of I in Pyrex tube without solvent with a 450 W high pressure mercury lamp at 35-40° for 15 hr gave a crude material (II), which was treated with acetic anhydride and pyridine to give the acetate (V), mp 120.5-121.5°, in 51% yield (from I). Reduction of V with lithium aluminum hydride gave II, mp 121-122°, in 62% yield. This product (II) was identified by comparison with the natural sugiresinol dimethyl ether.



