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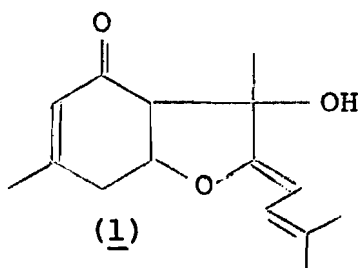
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## ABSTRACT

Angelikoreanol (1), a new sesquiterpene keto-alcohol, was first isolated from the ethereal extract of dried roots of Angelica koreana Max. (Korean Qianhu). The product was recrystallized from ethanol as colorless plates, m.p. 157-158°,  $(\alpha)_D + 132.2^\circ$  ( $C = 1.2$  in  $\text{CHCl}_3$ ). Its structure was assigned on the basis of chemical and spectroscopic data. In this project various approaches



to the total synthesis of angelikoreanol were investigated.

Methylation of orcinol monohydrate (9) with dimethyl sulfate and potassium carbonate in acetone gave a 94.0% yield of orcinol dimethylether (10). The conversion of (10) to 5-methylcyclohexan-1,3-dione (11) was effected by using Birch reduction in 42.7% yield. Orcinol monohydrate (9) could also be converted directly to (11) by catalytic hydrogenation over Adam's catalyst in 51.0% yield. In the next step the enolate anion of (11) was first generated in the presence of sodium methoxide and

then alkylated with ethyl bromoacetate in dimethylsulfoxide (DMSO) to give 2-(carboethoxymethyl)-5-methylcyclohexan-1,3-dione (8a) in 23.0% yield. The product (8a) appeared to be unstable and polymerized upon standing for a few days at room temperature. The structure of (8a) was established on the basis of its spectral data. An alternative route was also investigated. The first step of this route was the condensation of chloroacetonitrile with phloroglucinol (21a, R = OH) (Hoesch reaction) to give 2,4,6-trihydroxy- $\alpha$ -chloroacetophenone (22a, R = OH). This was followed by cyclisation with potassium acetate to afford a 51.3% yield of 4,6-dihydroxybenzofuran-3(2H)-one (23a, R = OH). Methylation of (23a) with dimethyl sulfate and potassium carbonate in 1,2-dimethoxyethane (DME) gave 4,6-dimethoxybenzofuran-3(2H)-one (24a, R = OMe) in 70.0% yield. The reaction of (24a) with ethylene glycol and p-toluenesulfonic acid in benzene afforded 3(2H)-ethylenedioxy-4,6-dimethoxybenzofuran (25a, R = OMe) in 36.0% yield. Compound (25a) was reduced by sodium in liquid ammonia to give three isomeric products (in 15.0%, 13.0% and 5.0% yields), the structures of which were established on the basis of their spectral data.