

# Facile New Synthesis of Coniferyl and Sinapyl Alcohols

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## Introduction

Synthesis of coniferyl and sinapyl alcohols on large and small scale has become important during recent years due to the usefulness of these lignin monomers in preparing synthetic lignins and lignin model compounds. In the past, more or less selective 1,2-reduction of ethyl ferulate and ethyl sinapate with lithium aluminum hydride, sodium bis(2-methoxyethyl)aluminum hydride or diisobutyl aluminum hydride (DIBAL-H) has been used. The problem with all these reactions is the moderate yields, the necessity of handling air-sensitive reagents under protective gases, and the varying amount of contaminating 1,4-reduction products. Here we describe a simple, quick and efficient synthesis of the two lignin monomers by sodium borohydride reduction of coniferyl and sinapyl aldehydes that have recently become commercially available. Although coniferyl alcohol is now available commercially, this procedure produces a cleaner and cheaper product.

## Method

Coniferyl or sinapyl aldehyde (0.24 - 0.28 mmol) was dissolved in ethyl acetate (10 mL). Sodium borohydride (2 eq) was added and the mixture stirred at room temperature for 1 h. During that time a yellow precipitate formed. The mixture was then poured into water and washed once with saturated ammonium chloride solution. The organic layer was separated and dried over magnesium sulfate. Evaporation of the solvent in vacuo gave coniferyl or sinapyl alcohol as a slightly yellow oil in 94-98% yield.

The products were immediately characterized by NMR spectroscopy and showed no detectable 1,4-reduction products. A large-scale reduction performed on 5 g of coniferyl aldehyde required a

reaction time of 6.5 hours but gave an equivalently clean product, which was crystallized from methylene chloride/petroleum ether to give pale yellow crystals.

## Discussion and Conclusions

Sodium borohydride reduction of coniferyl or sinapyl aldehydes in ethyl acetate at room temperature offers an efficient method for preparing large or small amounts of clean coniferyl or sinapyl alcohols. The advantages of this method compared to the ones used to date are: the yields of the desired compounds are very high; the reaction products need no further purification and can therefore be used directly in following reactions; the starting materials (coniferyl and sinapyl aldehyde) are commercially available; there is no need to protect the phenolic hydroxy group as in some of the other preparation methods; and, most importantly, no 1,4-reduction products are formed. With the simplicity of this procedure and the relative inertness of the reducing agent (sodium borohydride), even a non-chemist can run the reaction and prepare quality lignin monomers.

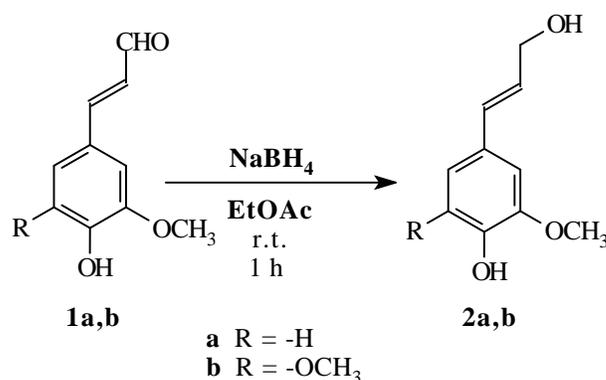


Figure 1. NaBH<sub>4</sub> reduction of coniferyl and sinapyl aldehydes.