**New Silyl Ether Reagents for the Absolute Stereochemical Determination of 2º Alcohols.**

R. Thomas Williamson*, Ana C. Barrios Sosa, Abhijit Mitra, Pamela J. Seaton, Douglas B. Weibel, Frank C.Schroeder, Jerrold Meinwald, and Frank E. Koehn.

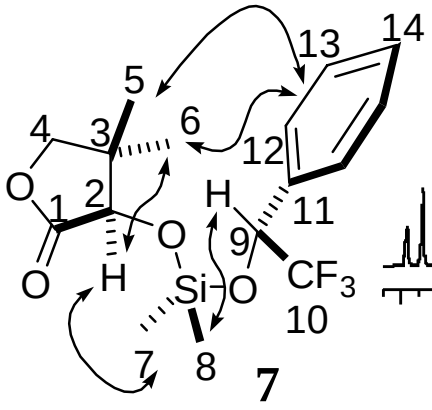
## Standard Derivitization Procedure:

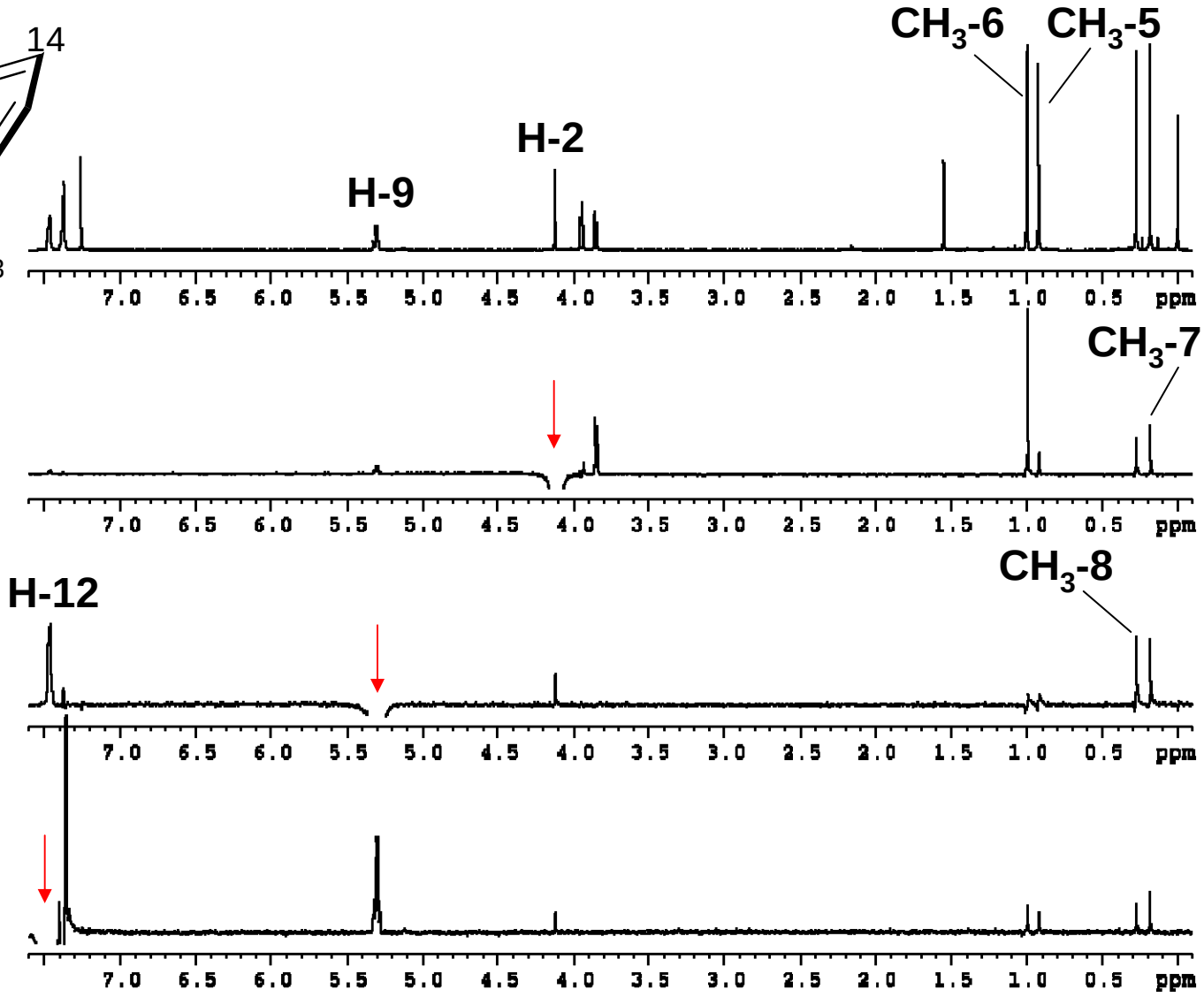
To a 5mm NMR tube containing 750 μL of $CDCl_3$ was added *R*- or *S*-(trifluoromethyl)benzyl alcohol (10 μL, 74.8 μmol). Dichlorodimethylsilane (180 μL, 1.48 mmol) was then added via syringe, followed by the addition of triethylamine (20 μL, 150 μmol). The mixture was shaken and allowed to react at room temperature for approximately 10 minutes. The solvent in the NMR tube was evaporated under vacuum until dry to give a white solid residue. The residue was then dissolved in 500 μL of $CDCl_3$ and a solution of the alcohol (18.5 μmol) in 150 μL of $CDCl_3$ was added to the NMR tube via syringe, followed by the addition of triethylamine (10 μL, 75 μmol). The mixture was shaken and allowed to react at room temperature for 20 minutes. The product was purified by eluting the mixture through a 3 cc silica gel solid phase extraction cartridge with $CDCl_3$ followed by an ethyl acetate/hexanes solvent system. In some cases, the products were further purified by HPLC using a semi-preparative silica column (10 x 300 mm) and an ethyl acetate/hexanes solvent system. Pure products were stored at –4 º C and were found to be stable as a solid or in $CDCl_3$ for several months.

Standard Desilylation Procedure:

To a solution of the silyl diether (1.5 μmol) in 500 μL of $CDCl_3$ was added tetrabutylammonium fluoride on silica gel (3 – 5 equivalents) and the mixture was shaken at room temperature for approximately 2 hrs. The reaction mixture was then filtered through a cotton plug and the free alcohol was purified by eluting the filtrate through a 3 cc silica gel solid phase extraction cartridge using a solvent system of ethyl acetate/hexanes.

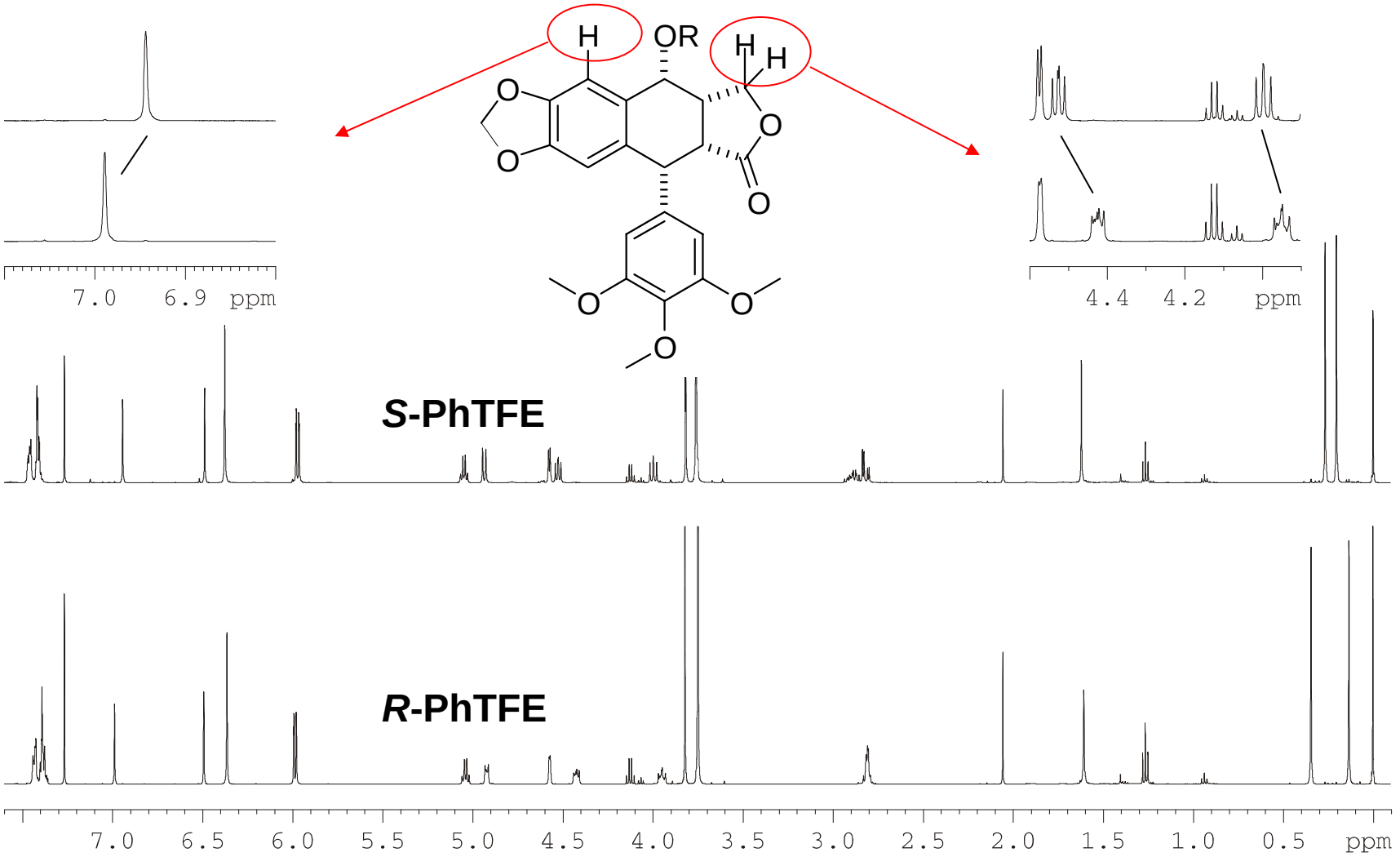
# Pantolactone DPFGSE 1D-NOE



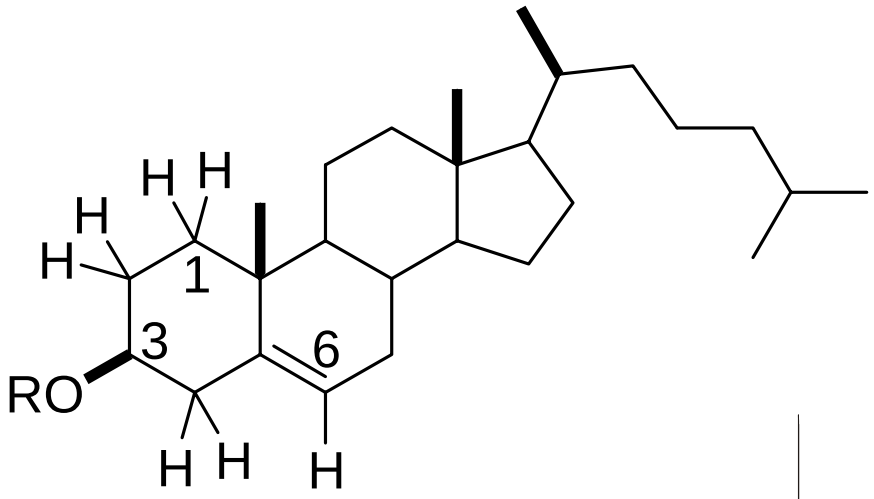



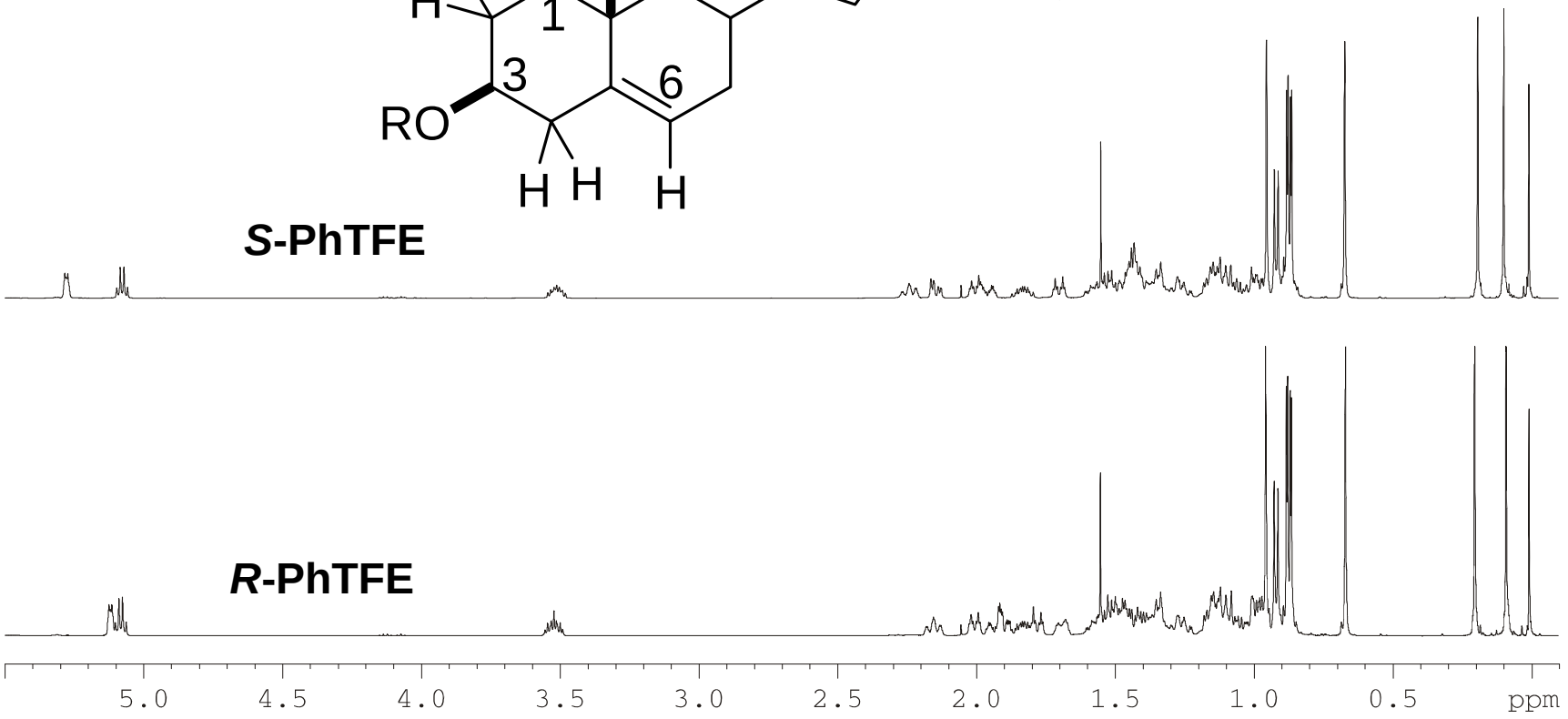

Red arrow indicates site of selective excitation.

# Podophyllotoxin



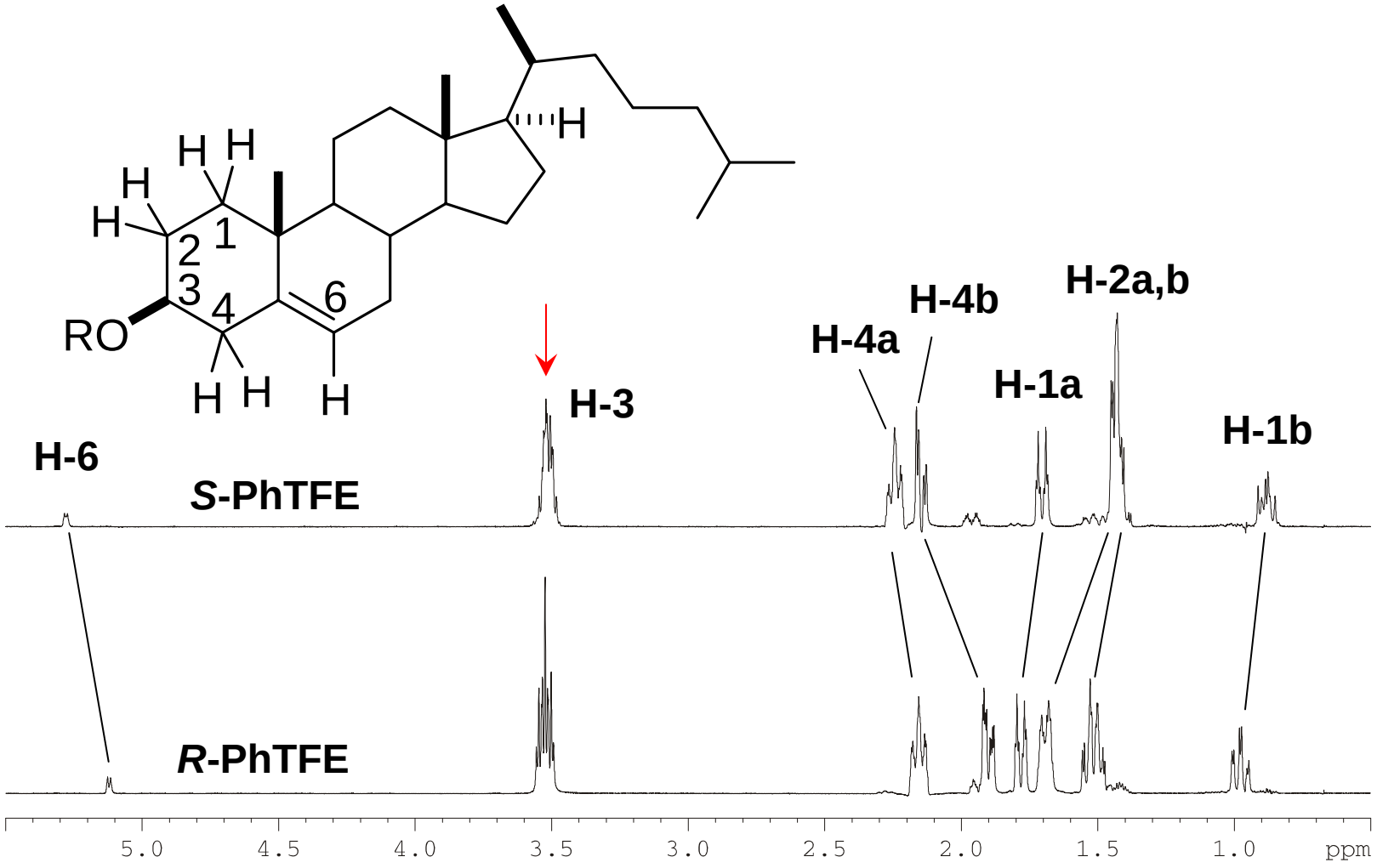
# Cholesterol



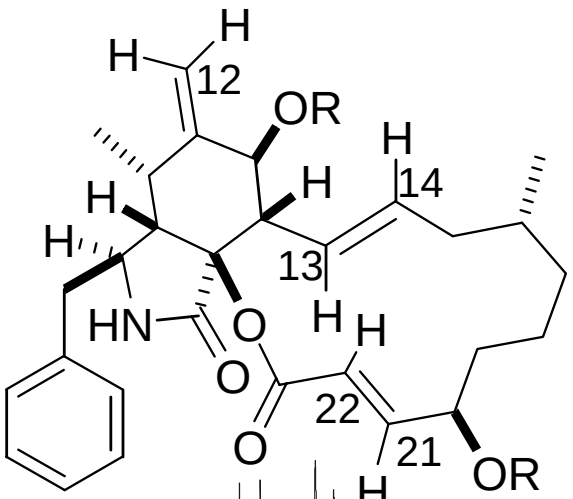



# Cholesterol

## DPFGSE 1D-TOCSY



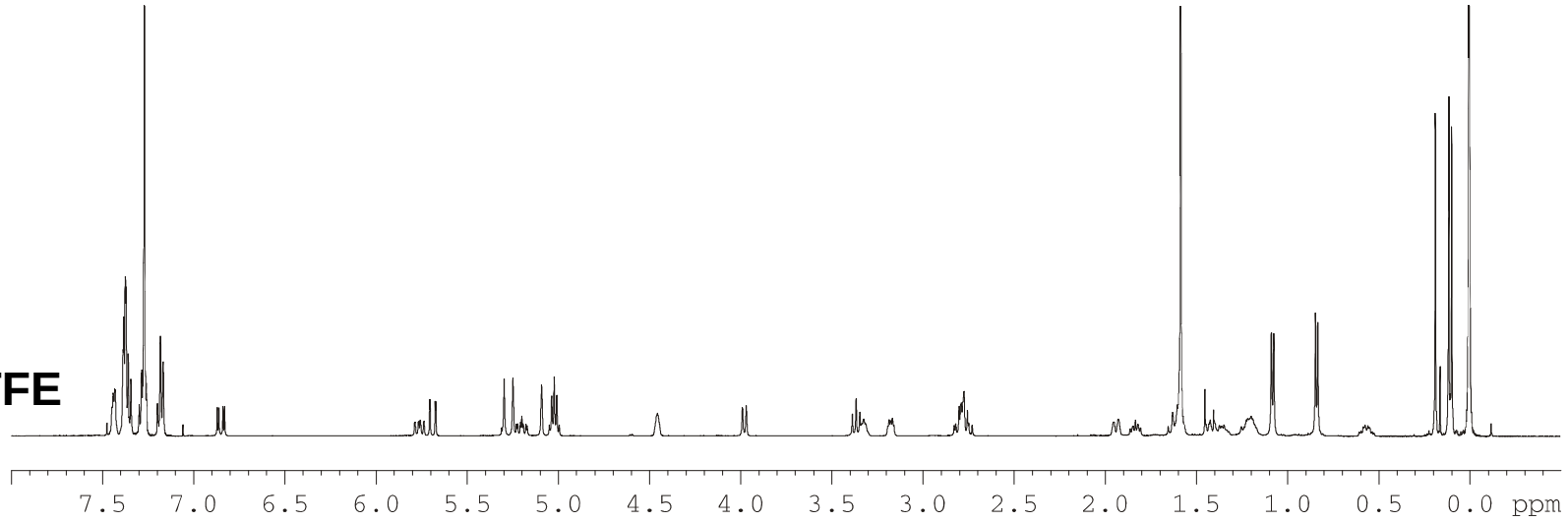
# Cytochalasin B




***S*-PhTFE**




***R*-PhTFE**



# Cytochalasin B