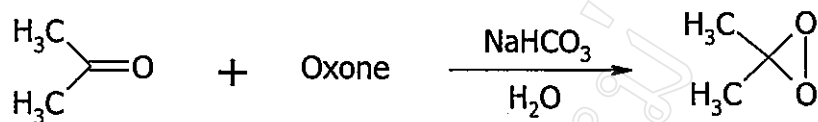


Appendix

I. Synthesis of Dimethyldioxirane



Procedure

Caution! Dimethyldioxirane is a volatile peroxide and should be treated as such. The preparation and all reactions of the dioxirane should be carried out in a hood.

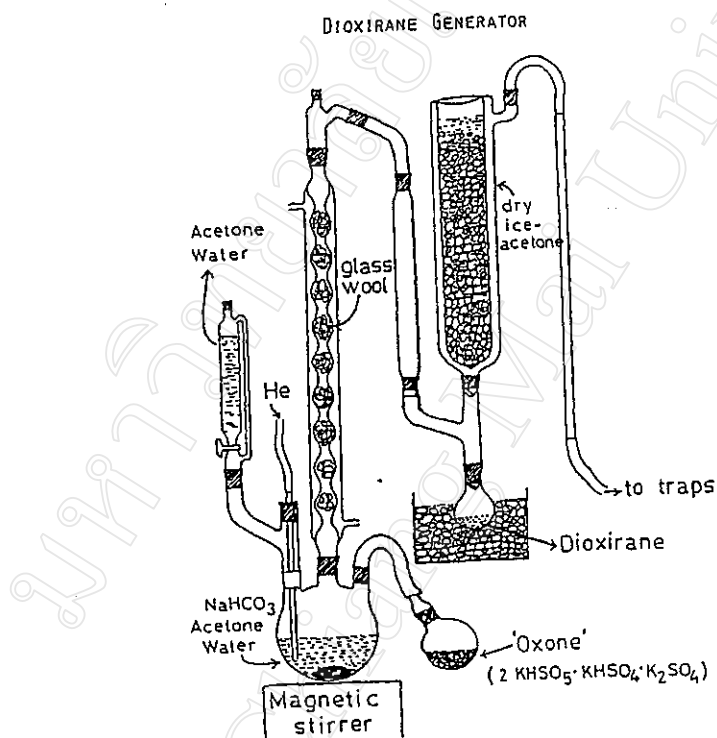
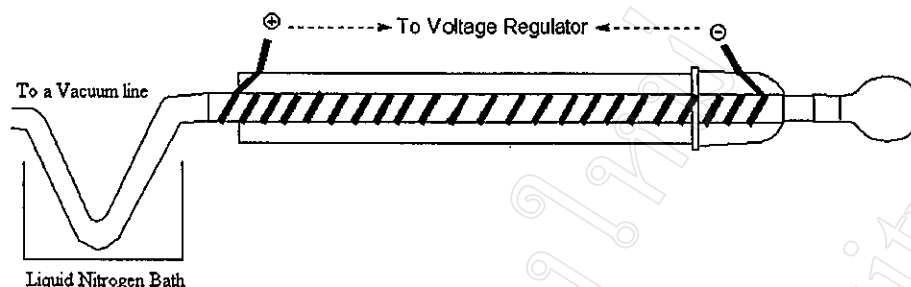


Figure I. Dioxirane Generator

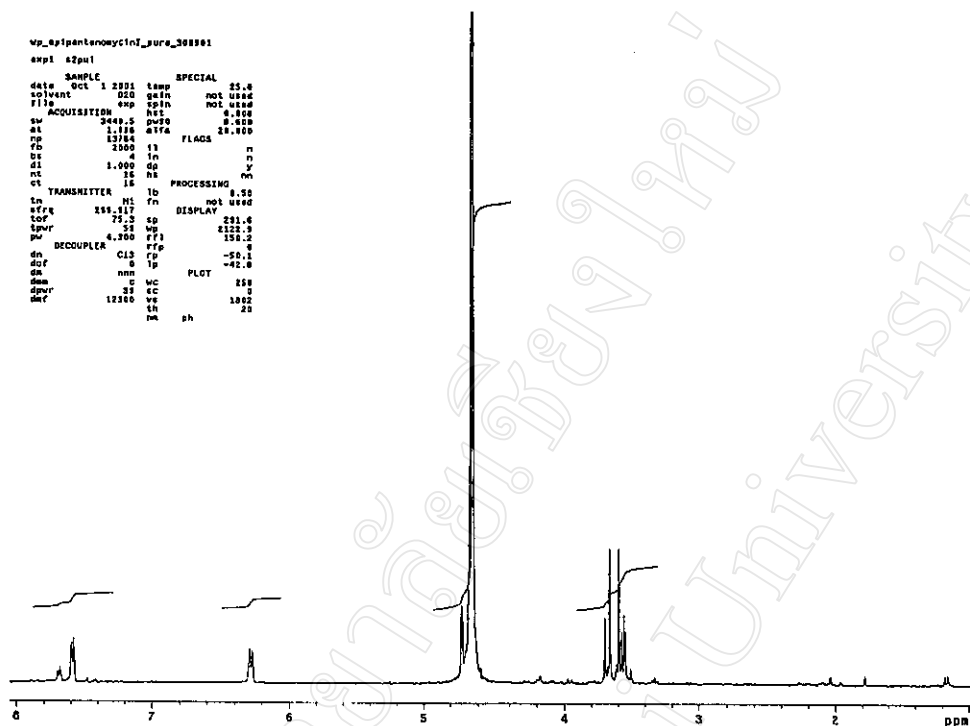
A 2-L, three-necked, round-bottomed flask containing a mixture of water (80 mL), acetone (50 mL, 0.68 mol), and sodium bicarbonate (69 g), is equipped with a magnetic stirring bar and a pressure equalizing addition funnel containing water (60 mL) and acetone (60 mL, 0.82 mol). A solid addition flask containing Oxone (180 g, 0.29 mol) is attached to the reaction vessel via a rubber tube. An air condenser (20 cm length) loosely packed with glass wool is attached to the reaction vessel. The outlet of the air condenser is connected to a 75 x 350-mm Dewar condenser filled with dry ice-acetone that is connected to a receiving flask (100 mL) cooled in a dry ice-acetone bath. The receiving flask is also connected in series to a second dry ice-acetone cold trap, a trap containing a potassium iodide solution, and a drying tube. A gas inlet tube is connected to the reaction flask and a stream of nitrogen gas is bubbled through the reaction mixture. The Oxone is added in portions (10-15 g) while the acetone-water mixture is simultaneously added dropwise. The reaction mixture is stirred vigorously throughout the addition of reagent (ca. 30 min). A yellow solution of dimethyldioxirane in acetone collects in the receiving flask. Vigorous stirring is continued for an additional 15 min while a slight vacuum (ca. 30 mm, water aspirator) is applied to the cold trap. The yellow dioxirane solution (62-76 mL) is dried over sodium sulfate (Na_2SO_4), filtered and stored in the freezer (-25°C) over Na_2SO_4 .

II. Flash vacuum pyrolysis (FVP)

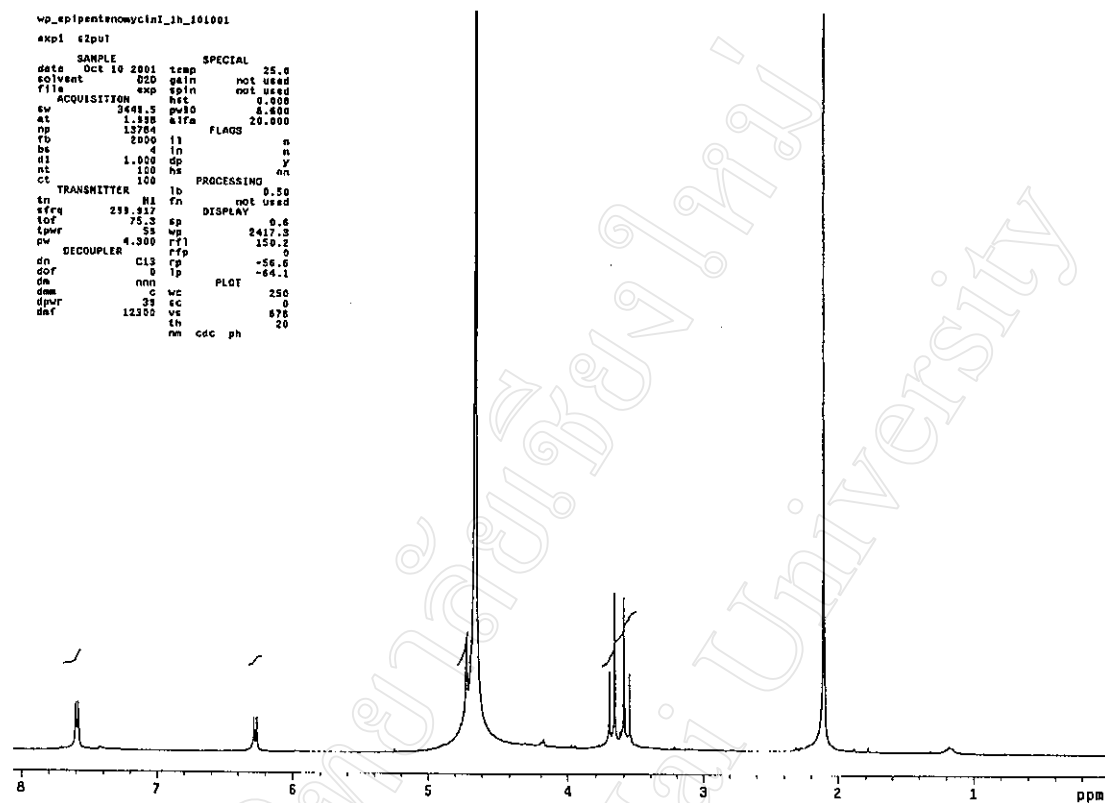


The FVP 400-450 °C, 0.05 mm apparatus was based on the design reported by Thebtaranonth. The compounds to be pyrolysed was placed in a 10-25 ml round-bottom flask connected to a heating column (40 cm) packed with glass chips and wrapped with a heating coil enclosed in a glass jacket. A U-tube attached to a vacuum line was connected to the end of the heating column and immersed in a liquid nitrogen bath. The temperature of heating column (400-450 °C) was controlled by a voltage regulator. A hotgun(or free flame) was used to control the temperature of strating material in the flask. After the reaction, the trap was opened, and the products were washed out with pure DCM.

III. The ^1H -NMR spectrum of a mixture of epipentenomycin I and pentenomycin I (D_2O)

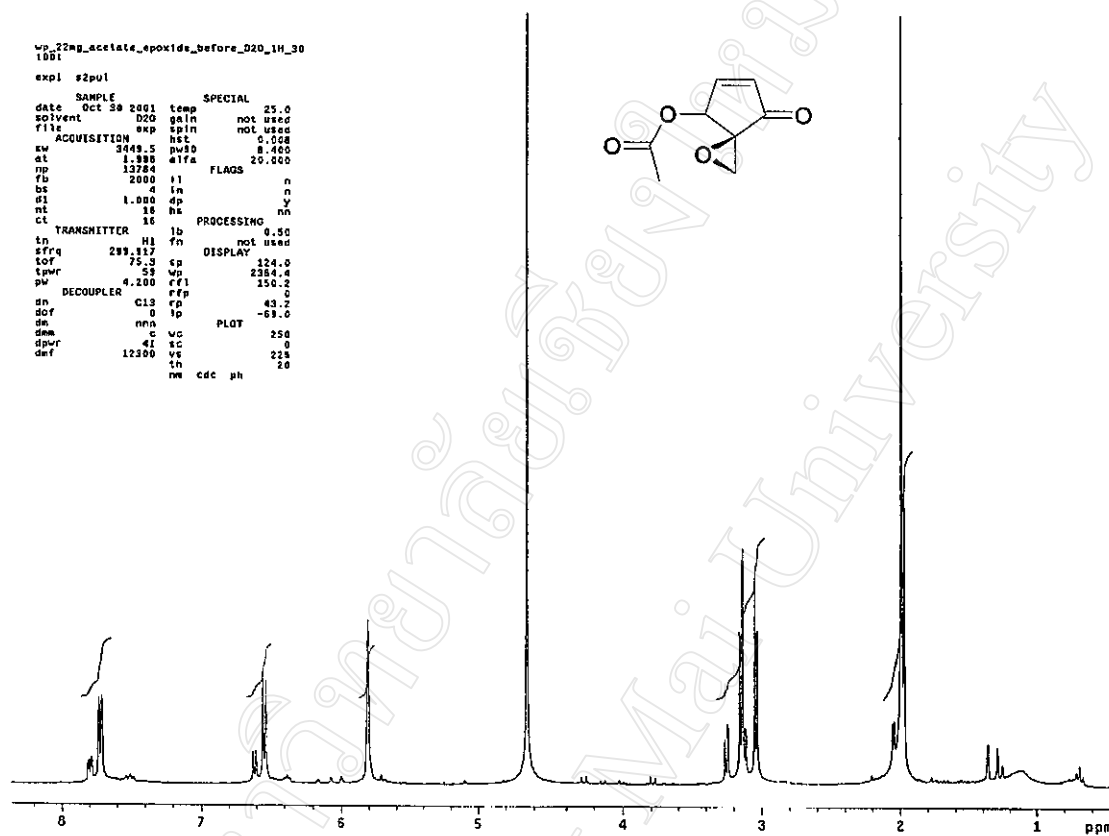


IV. The ^1H -NMR spectrum of epipentenomycin I (D_2O)

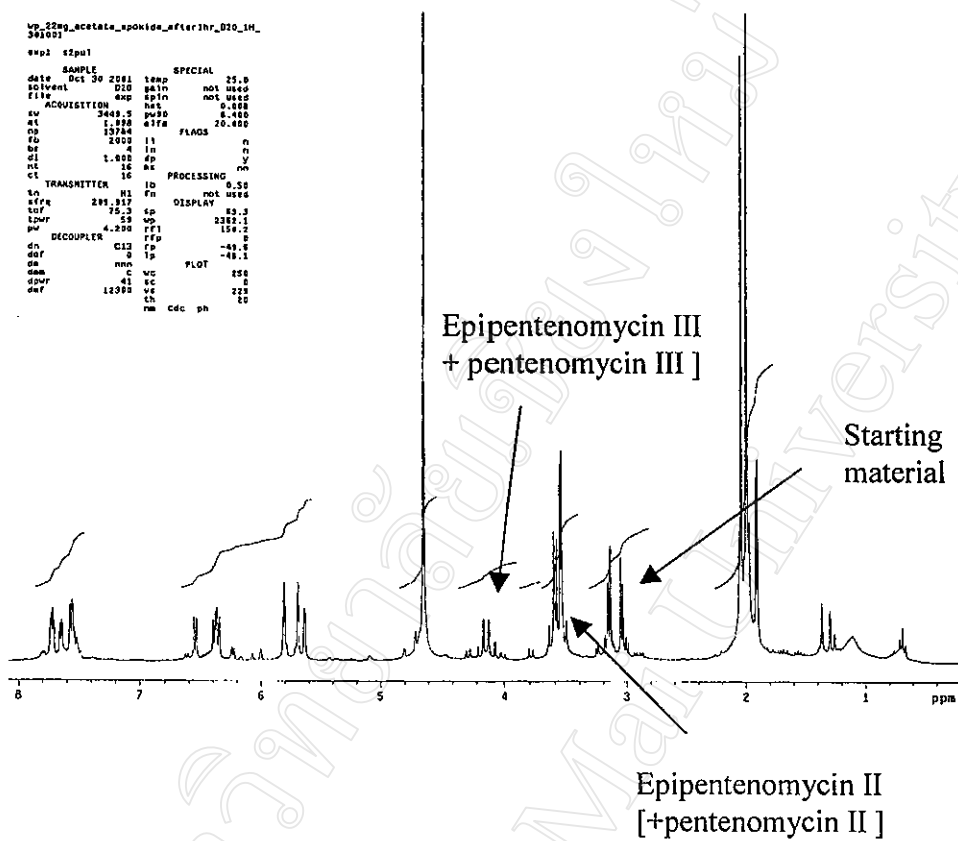


V. The conversion of pentenomycin II and epipentenomycin II to pentenomycin III and epipentenomycin III monitored by the ^1H -NMR spectroscopy

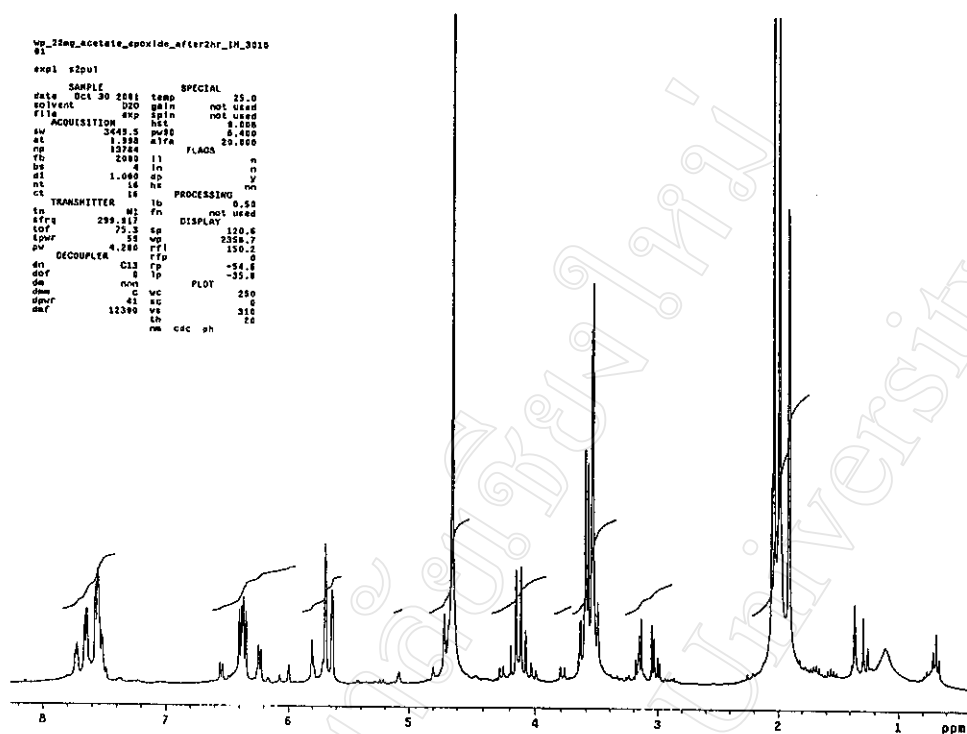
1. The ^1H -HMR of spiro epoxide (70) plus (71) before hydrolysis (D_2O)



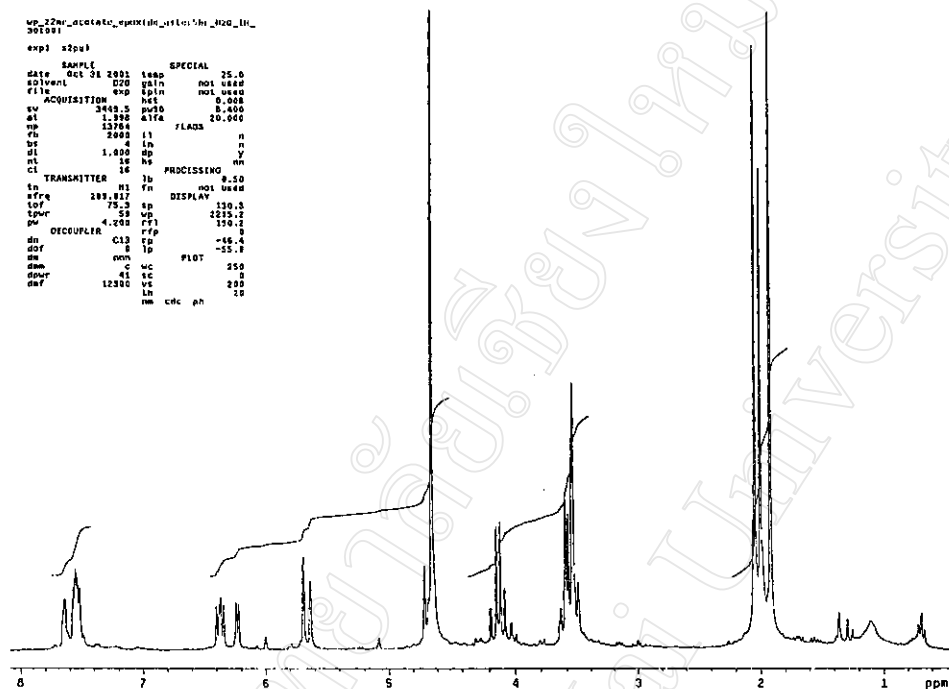
2. The ^1H -NMR spectrum after hydrolysis of (70) and (71) for 1 hour (D_2O)



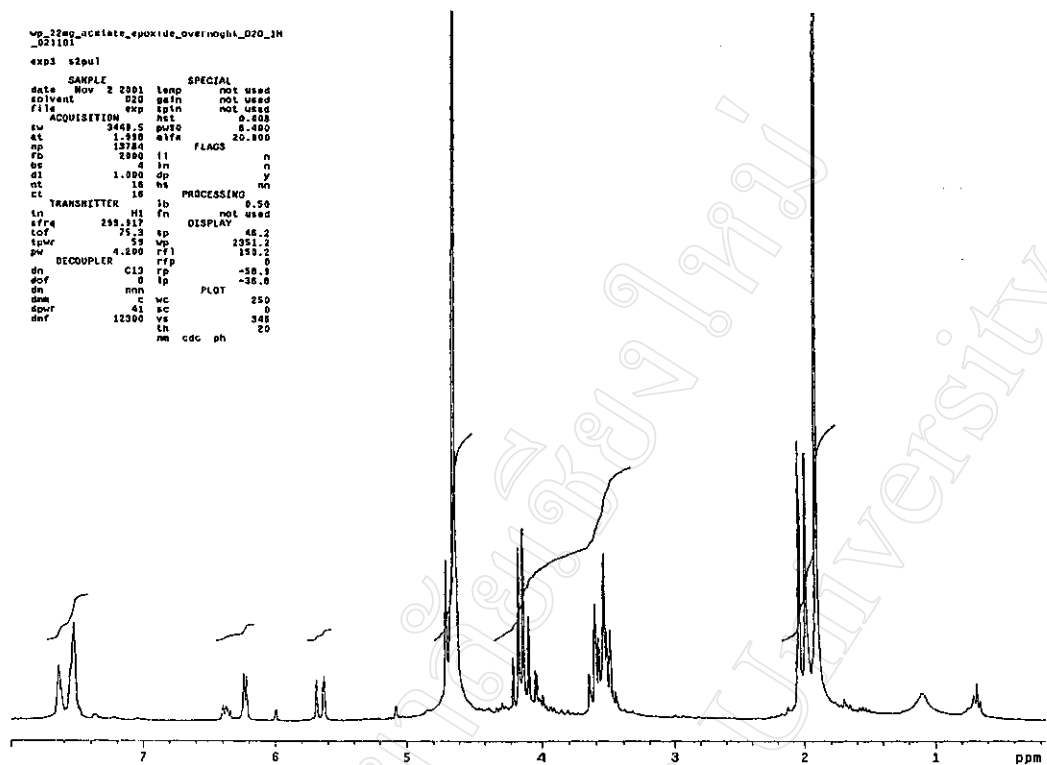
3. The ^1H -NMR after hydrolysis of (70) and (71) for 2 hours (D_2O)



4. The ^1H -NMR after hydrolysis of (70) and (71) for 5 hours (D_2O)



5. The ^1H -NMR after hydrolysis of (70) and (71) for 16 hours (D_2O)



VITA

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