

Supporting Information for

A Stereoselective Total Synthesis of 7,8-*O*-isopropylidene

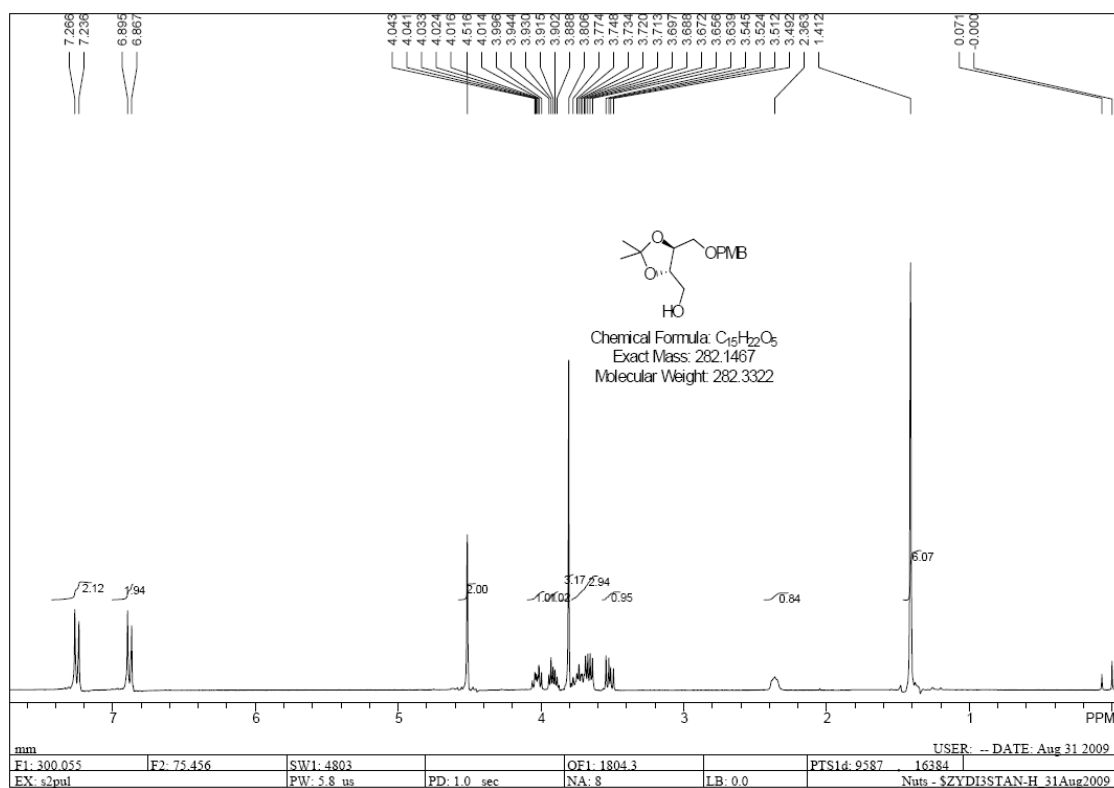
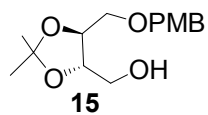
Iriomoteolide-3a

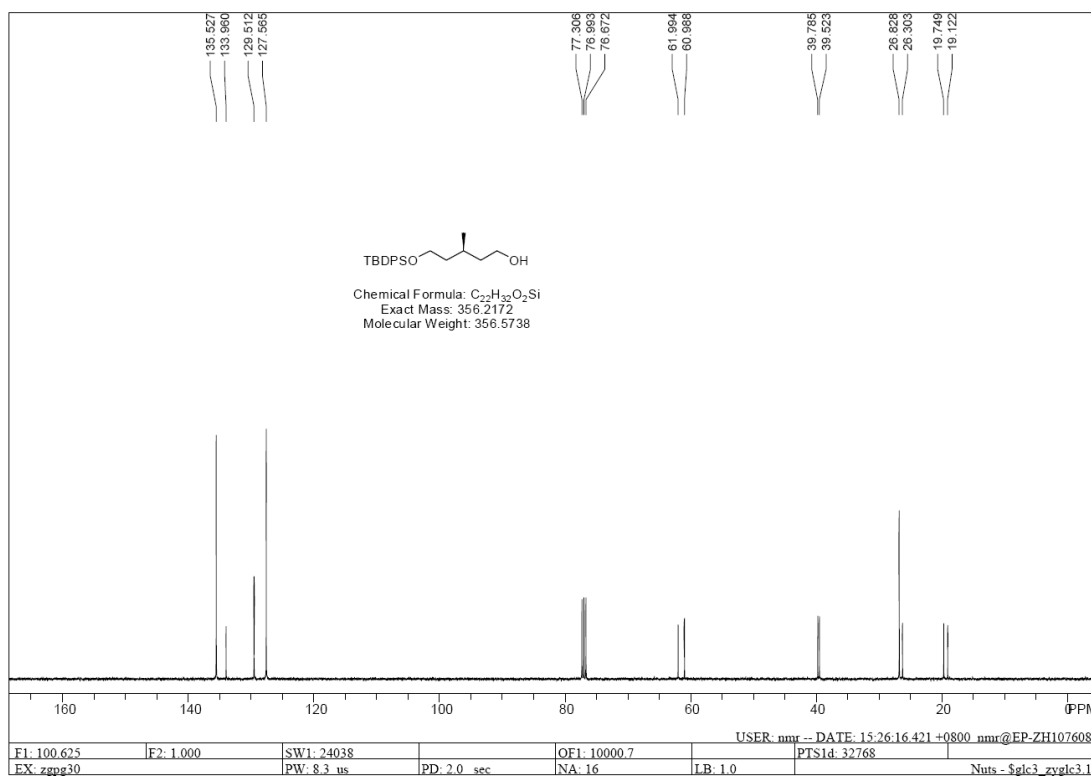
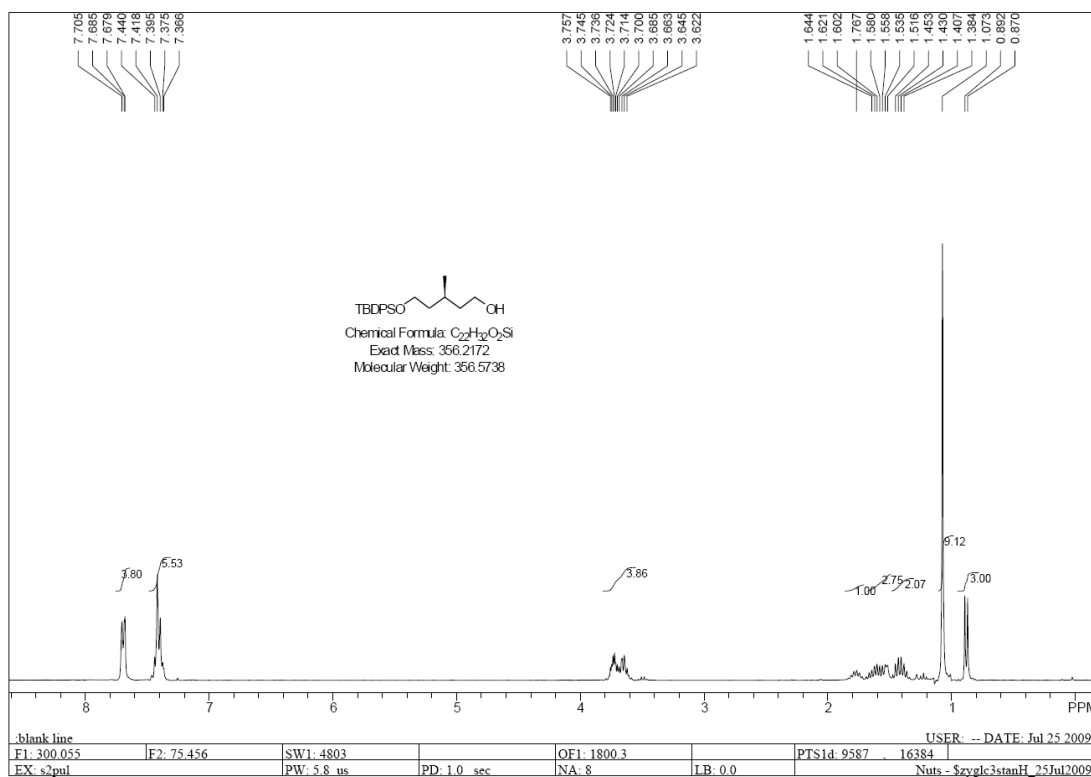
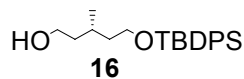
Yao Zhang, Lisheng Deng, Gang Zhao*

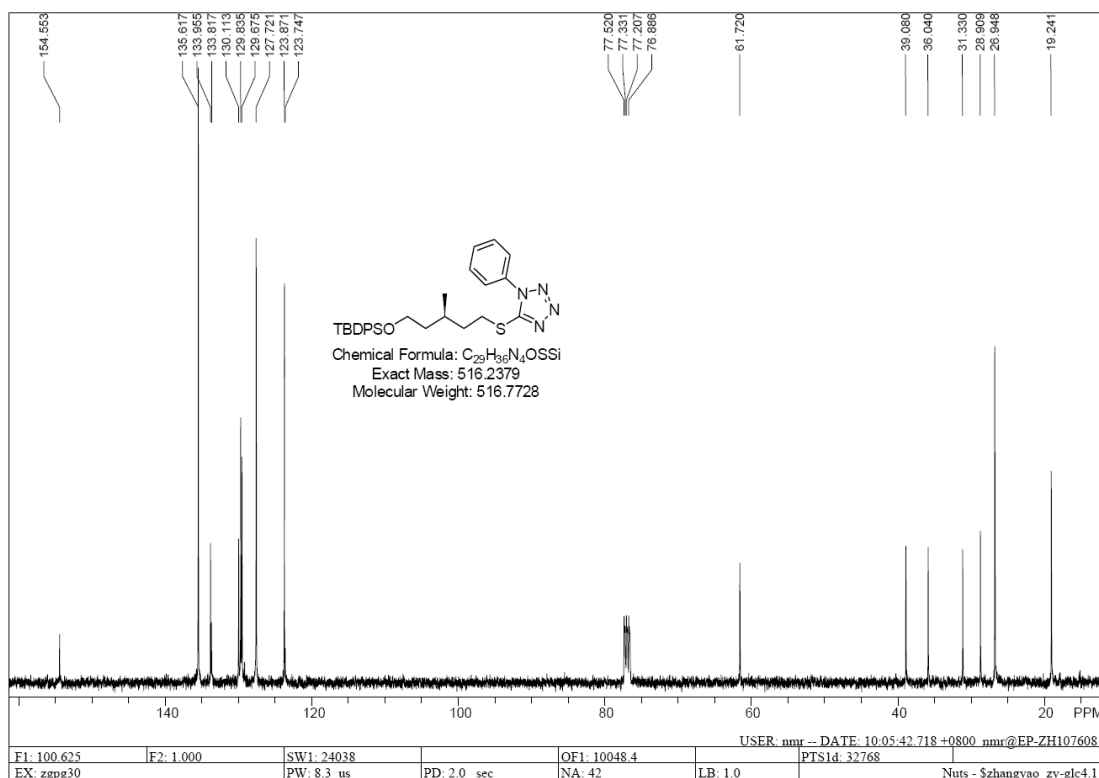
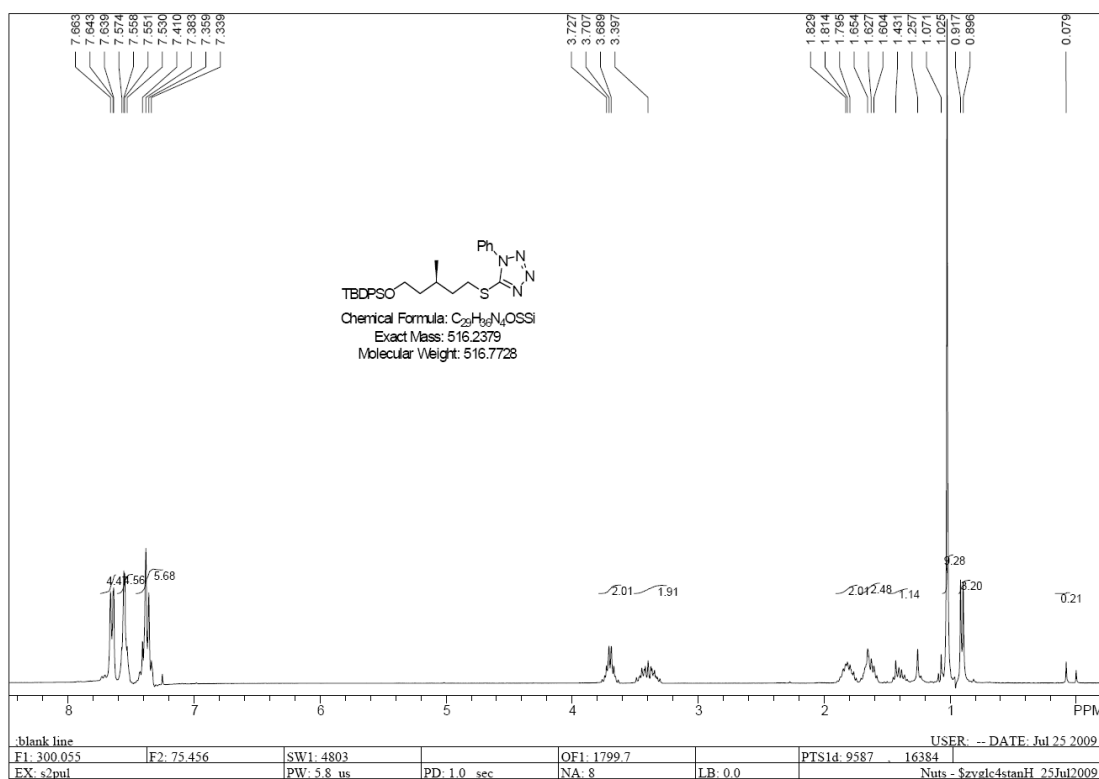
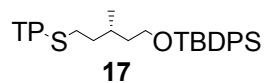
Laboratory of Modern Synthetic Organic Chemistry, Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, 345 LingLing Road, Shanghai 200032, China.

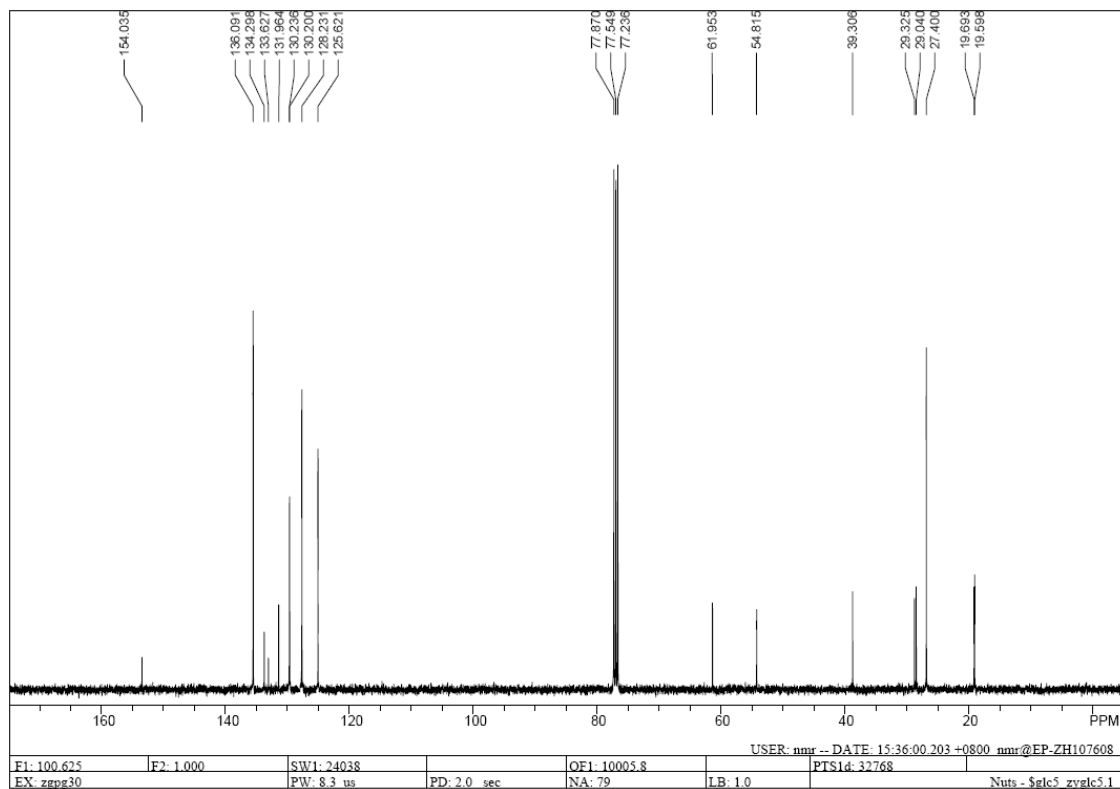
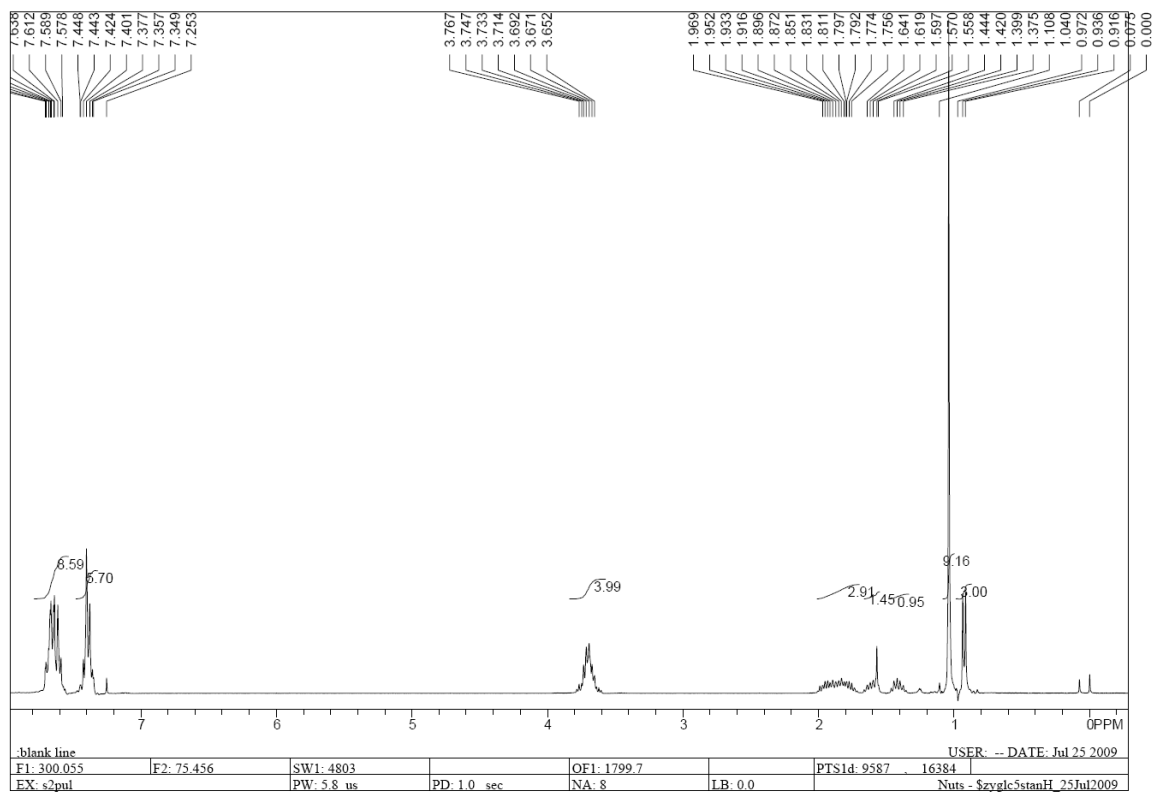
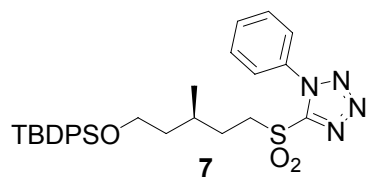
General information	S2
Spectra of the corresponding compounds	S2-S39
¹ H NMR for 15	S 3
¹ H NMR & ¹³ C NMR for 16 .	S 3
¹ H NMR & ¹³ C NMR for 17	S 5
¹ H NMR & ¹³ C NMR for 7	S 6
¹ H NMR & ¹³ C NMR for 18	S 7
¹ H NMR & ¹³ C NMR for 19	S 8
¹ H NMR & ¹³ C NMR for 20	S 9
¹ H NMR & ¹³ C NMR for 21	S 10
¹ H NMR & ¹³ C NMR for 2	S 11
¹ H NMR for 12	S 12
¹ H NMR & ¹³ C NMR for 24	S 13
¹ H NMR & ¹³ C NMR for 26	S 14
¹ H NMR & ¹³ C NMR for 8	S 15
¹ H NMR & ¹³ C NMR for 27	S 16
¹ H NMR & ¹³ C NMR for 28	S 17
¹ H NMR & ¹³ C NMR for 29	S 18
¹ H NMR & ¹³ C NMR for 30	S 19
¹ H NMR & ¹³ C NMR for 31	S 20
¹ H NMR & ¹³ C NMR for 3	S 21
¹ H NMR & ¹³ C NMR for 4	S 22
¹ H NMR & ¹³ C NMR for 33	S 23
¹ H NMR & ¹³ C NMR for 34	S 24
¹ H NMR & ¹³ C NMR for 34	S 25
¹ H NMR & ¹³ C NMR for 7,8- <i>O</i> -isopropylidene Iriomoteolide-3a(1)	S 26
HPLC spectrum for 23	S 27

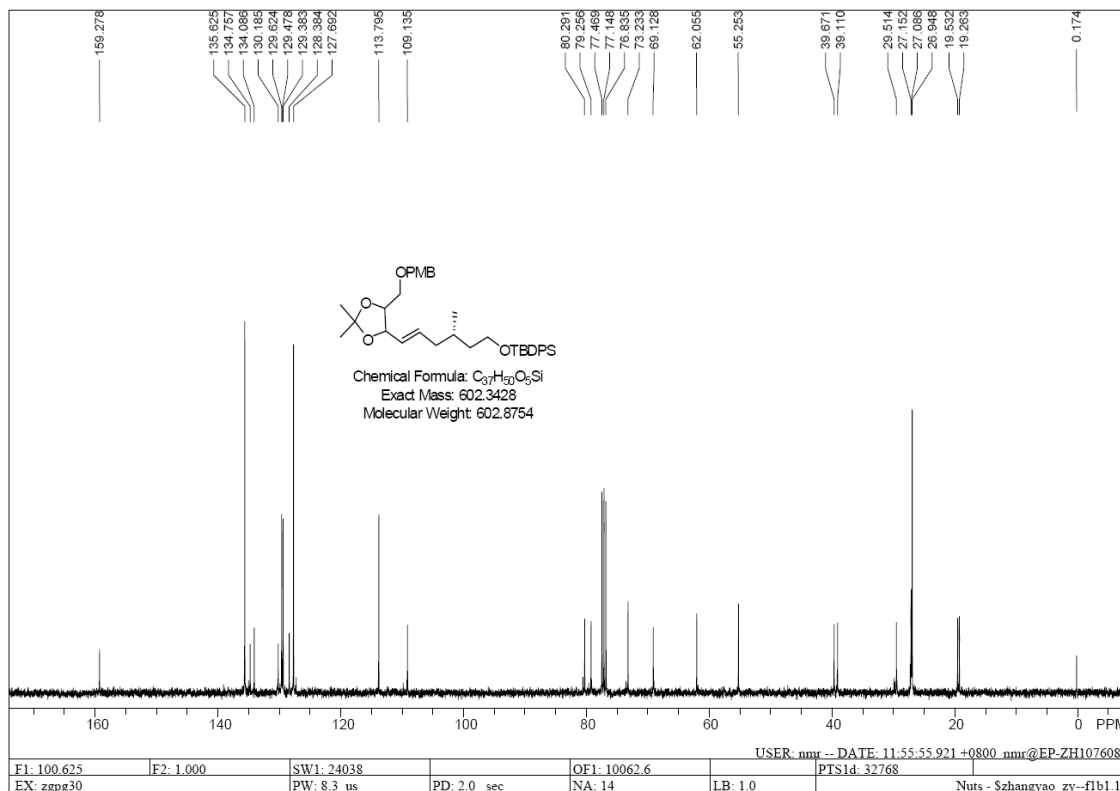
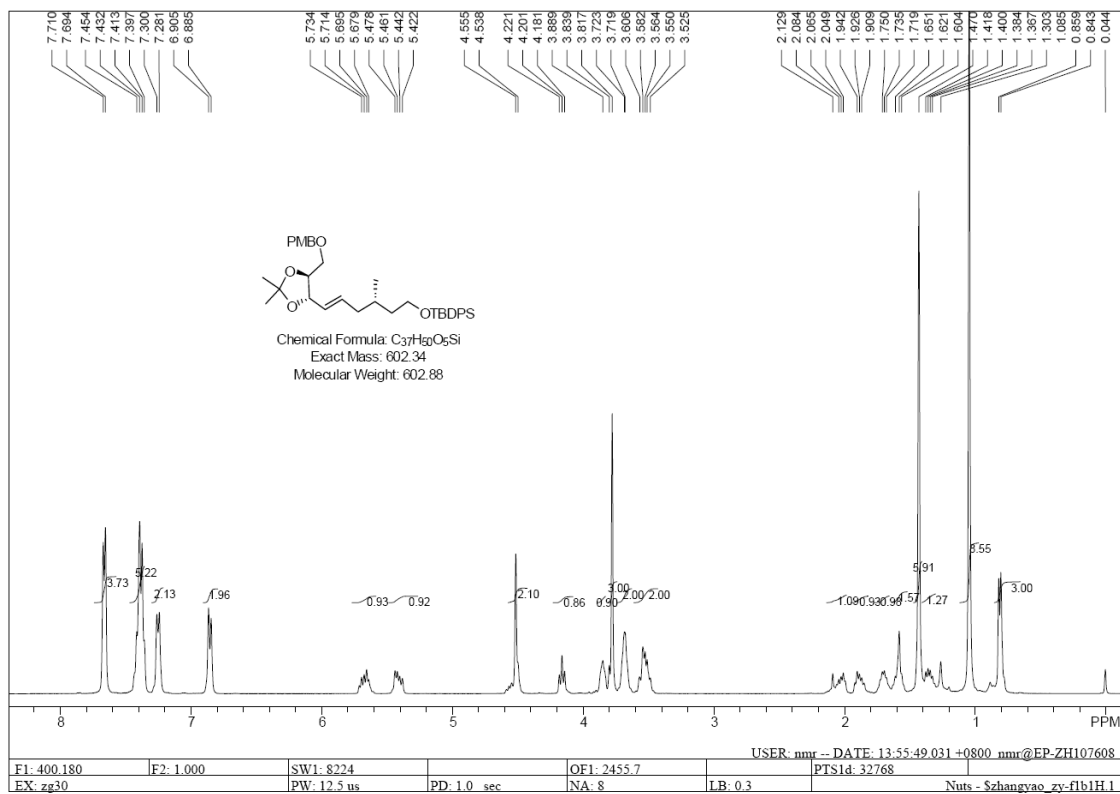
General information: All solvents were distilled prior to use except where noted. All reactions sensitive to moisture or oxygen were conducted under an atmosphere of nitrogen or argon using flame-dried or oven-dried (300 °C) glassware. Crushed 4Å molecular sieves were activated by flame-drying immediately prior to use. Tetrahydrofuran (THF), diethyl ether (Et₂O) and toluene (PhCH₃) were purified by sodium/benzophenone. Dichloromethane (CH₂Cl₂), N, N-diisopropylethylamine (DIPEA), diisopropylamine, pyridine, triethylamine (TEA) and boron trifluoride etherate were distilled from calcium hydride before use. Dimethylsulfoxide (DMSO) and dimethylformamide (DMF) were distilled from calcium hydride under reduced pressure and stored over 4Å molecular sieves until needed. Reactions were monitored by thin-layer chromatography (TLC) carried out on 0.25 mm silica gel plates visualized with UV light and/or by staining with ethanolic phosphomolybdic acid (PMA). Flash column chromatography was performed on silica gel H (10-40 μ). NMR spectra were recorded on 300 or 500 MHz instruments. Chemical shifts (δ) are given in ppm relative to TMS, coupling constants (*J*) in Hz. IR spectra were recorded on a spectrometer. Optical rotations were taken. Mass spectra (MS) were measured with a spectrometer. High resolution mass spectra (HRMS) were recorded on a mass spectrometer. Elemental analyses were performed. Melting point were measured on a digital melting-point apparatus.

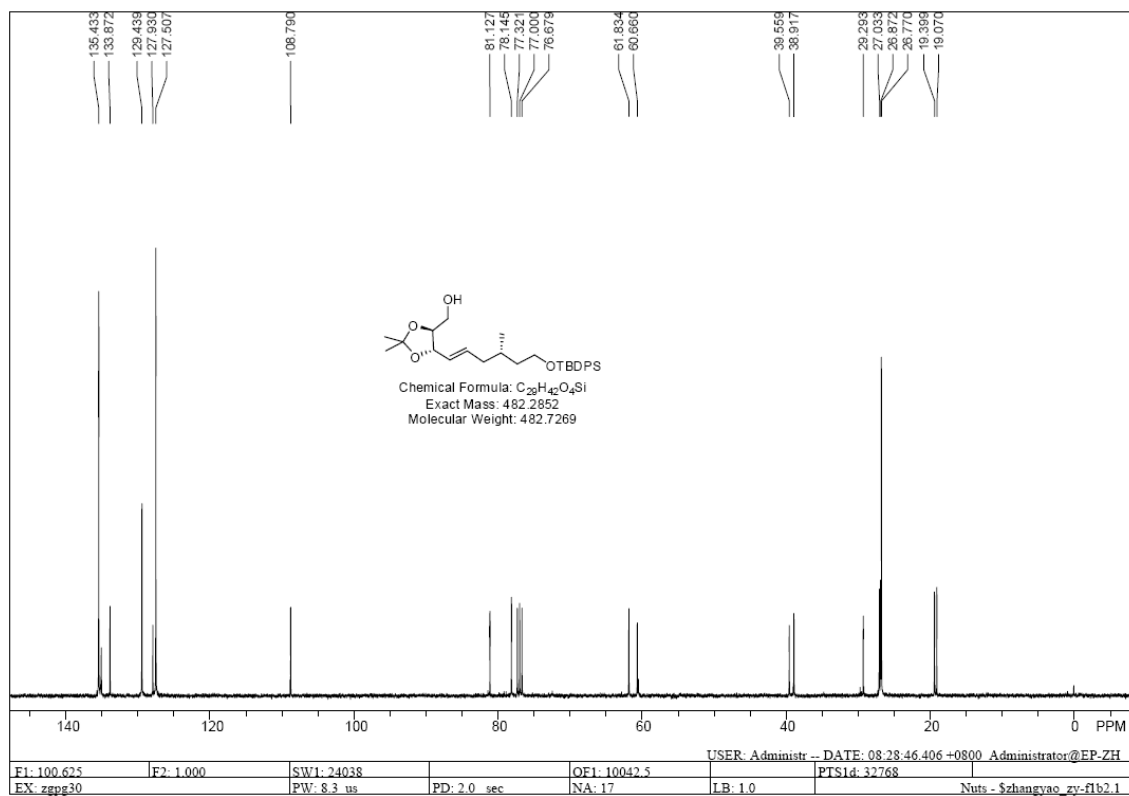
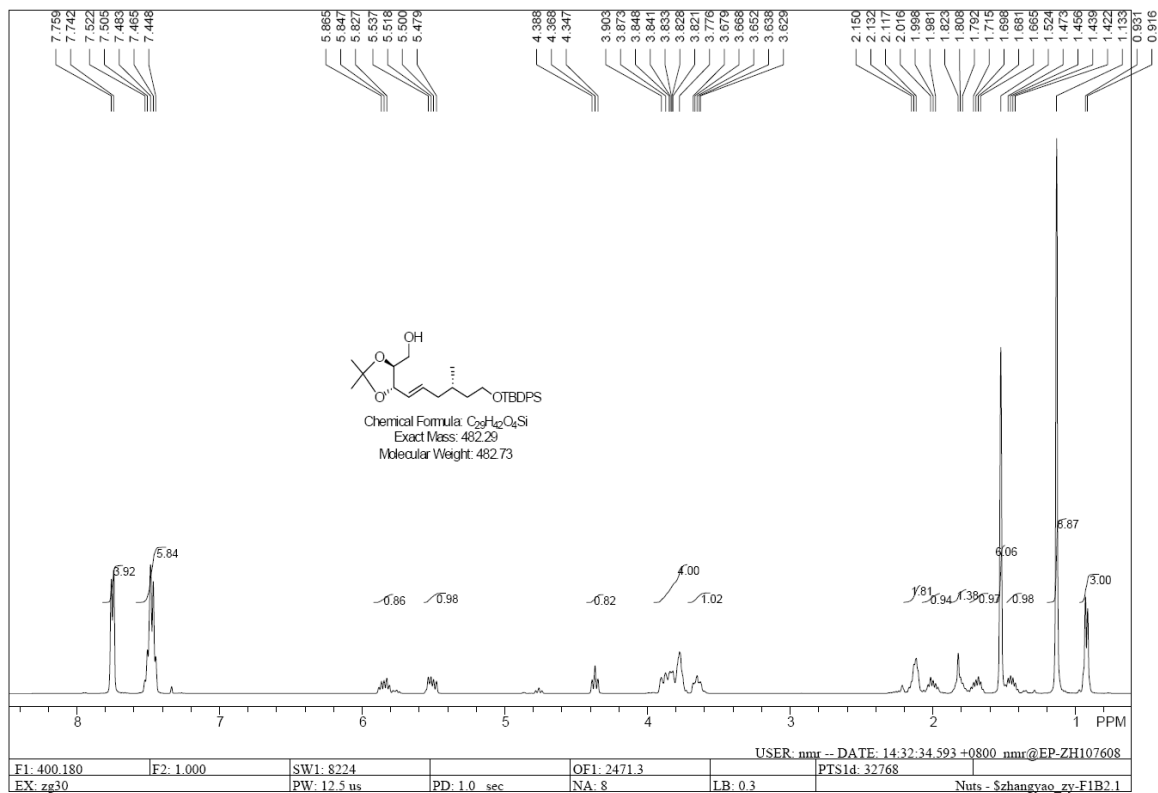
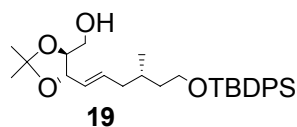


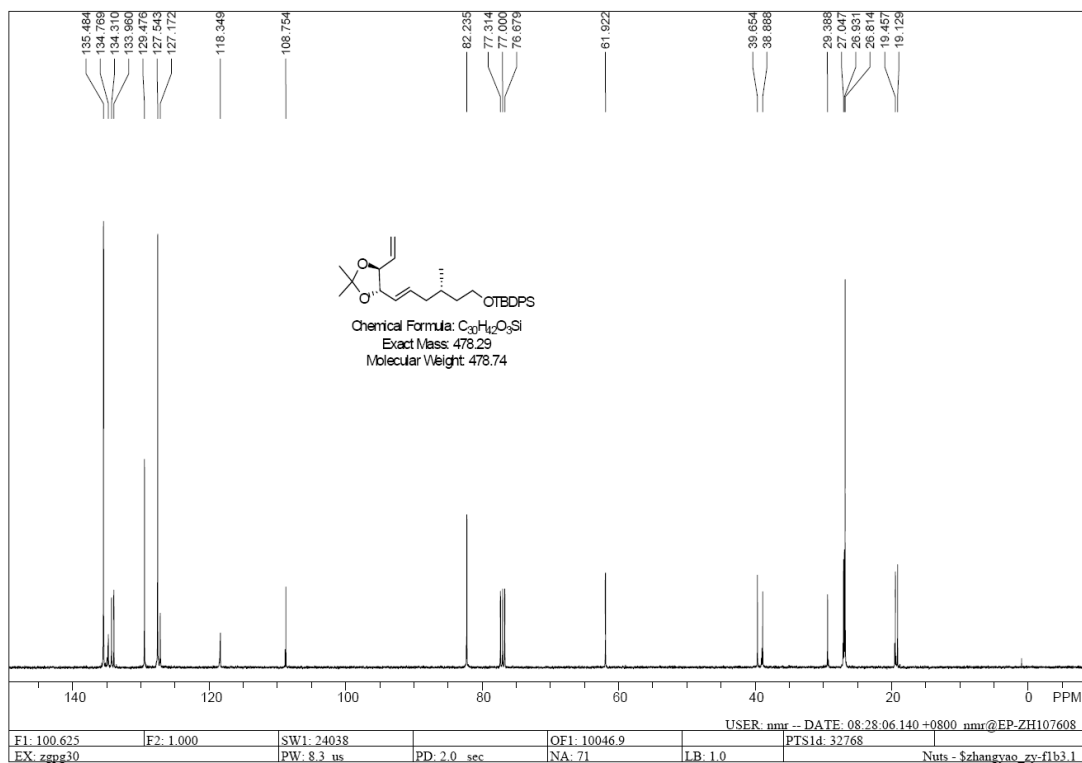
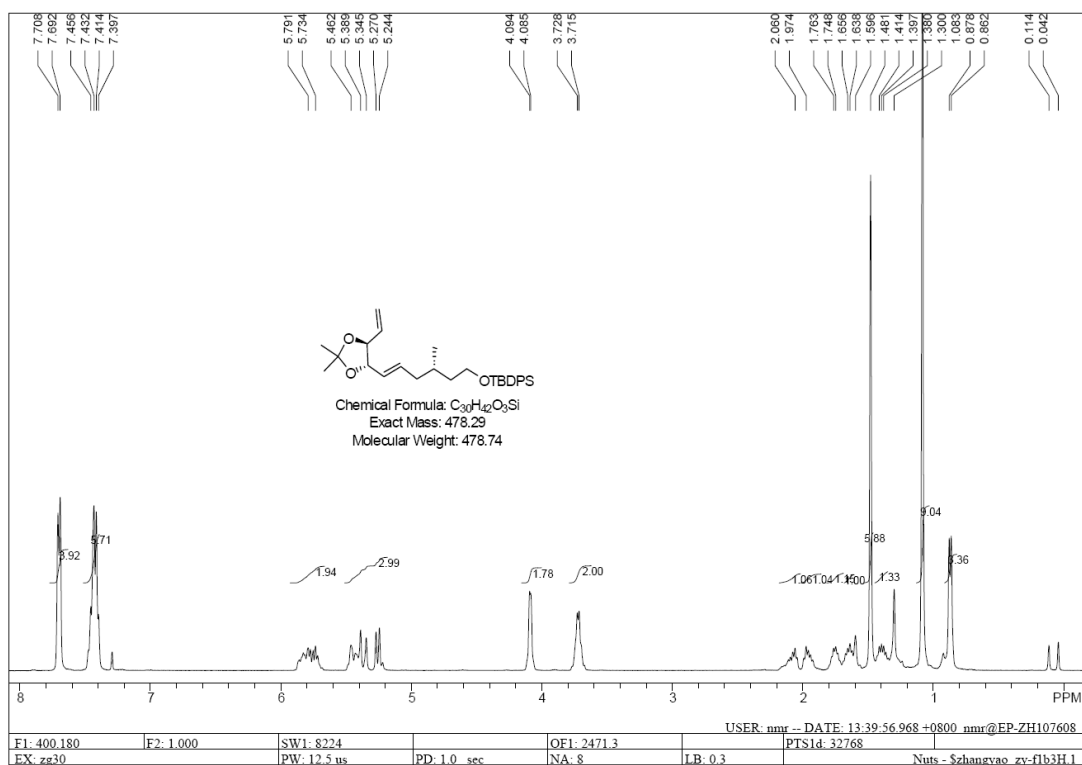
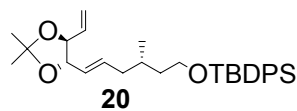


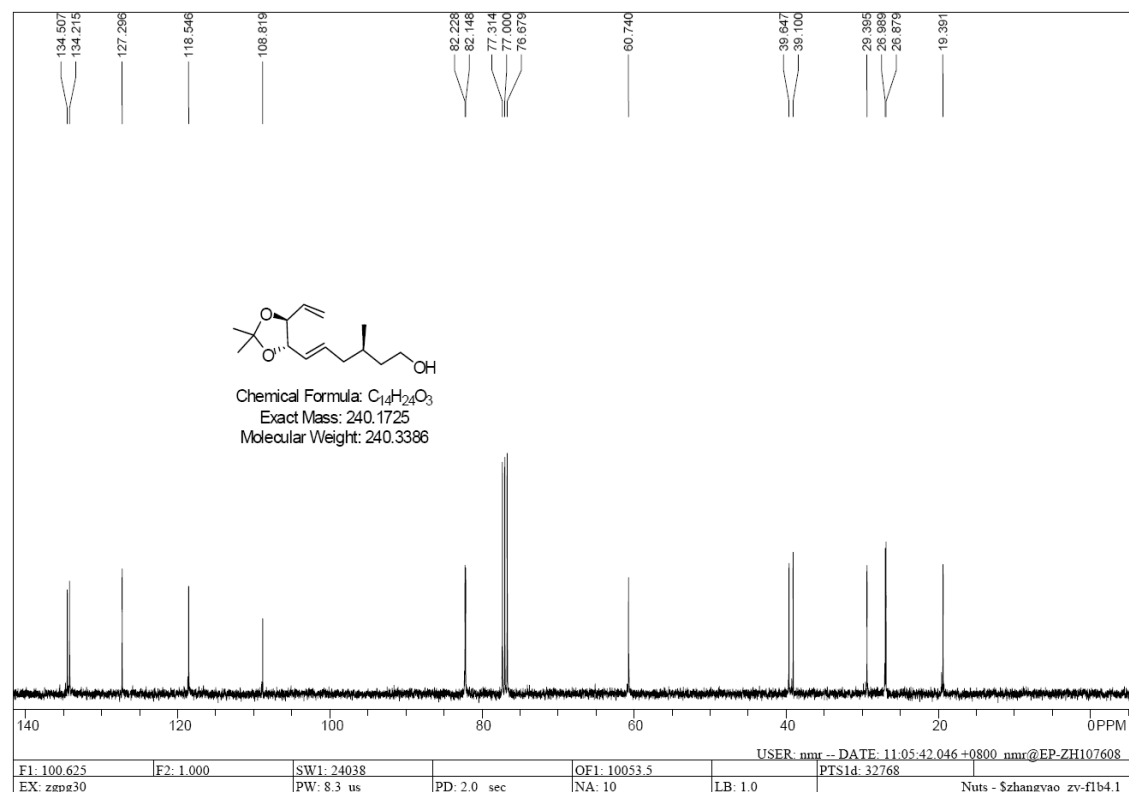
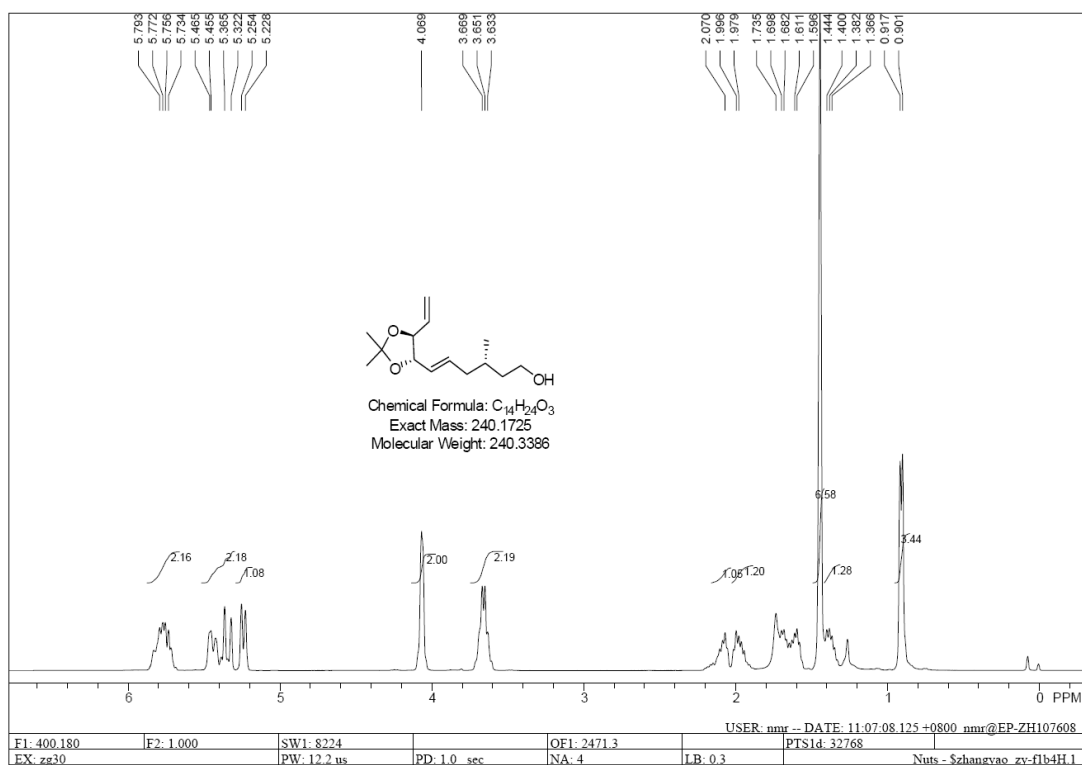
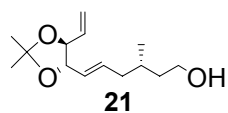


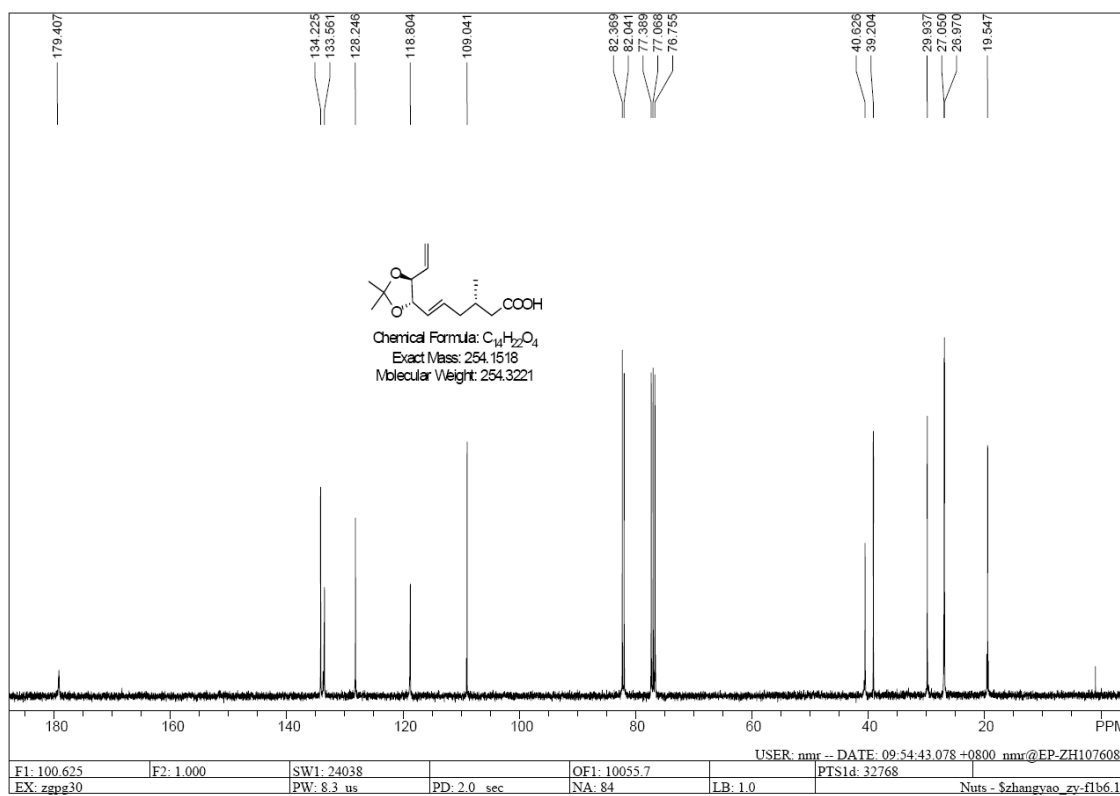
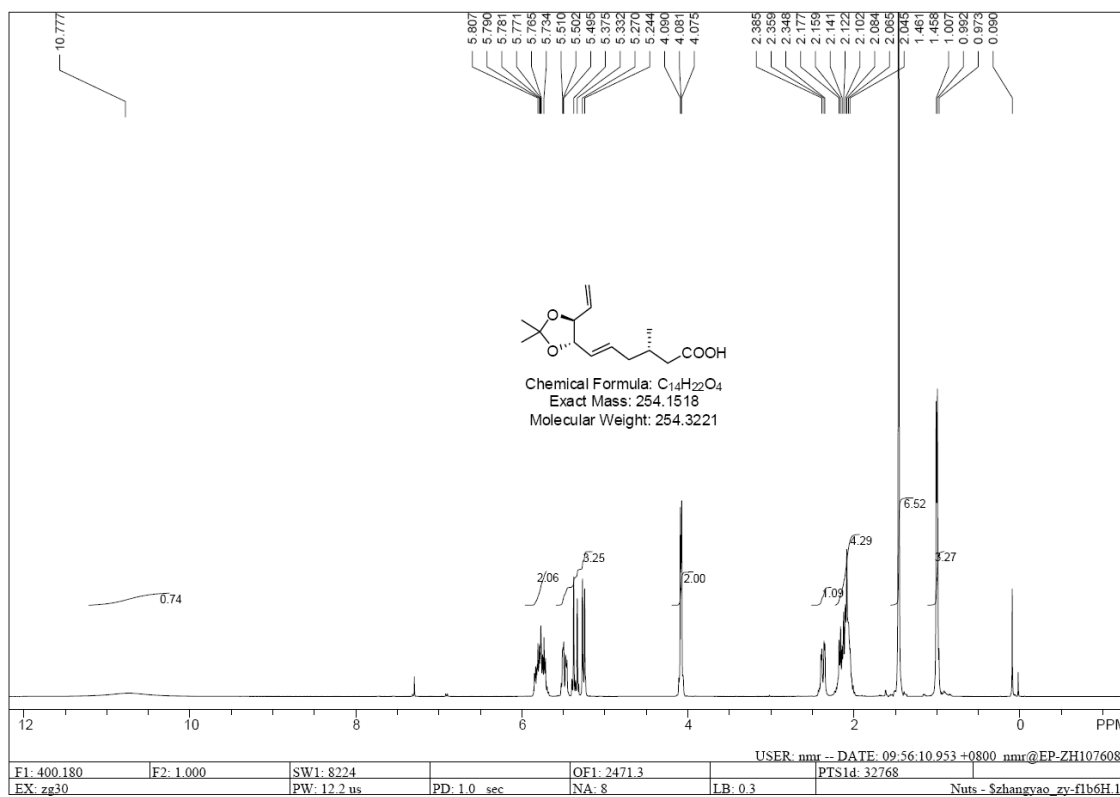
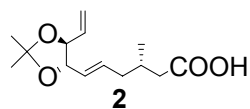


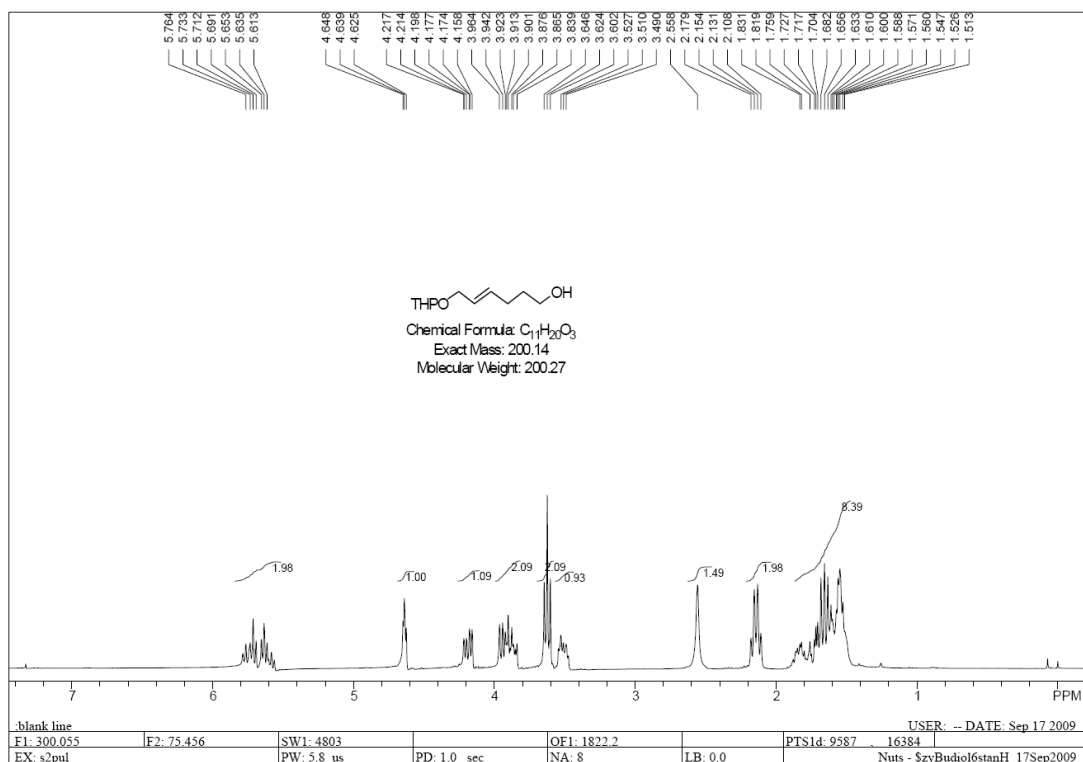
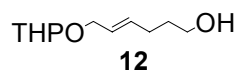


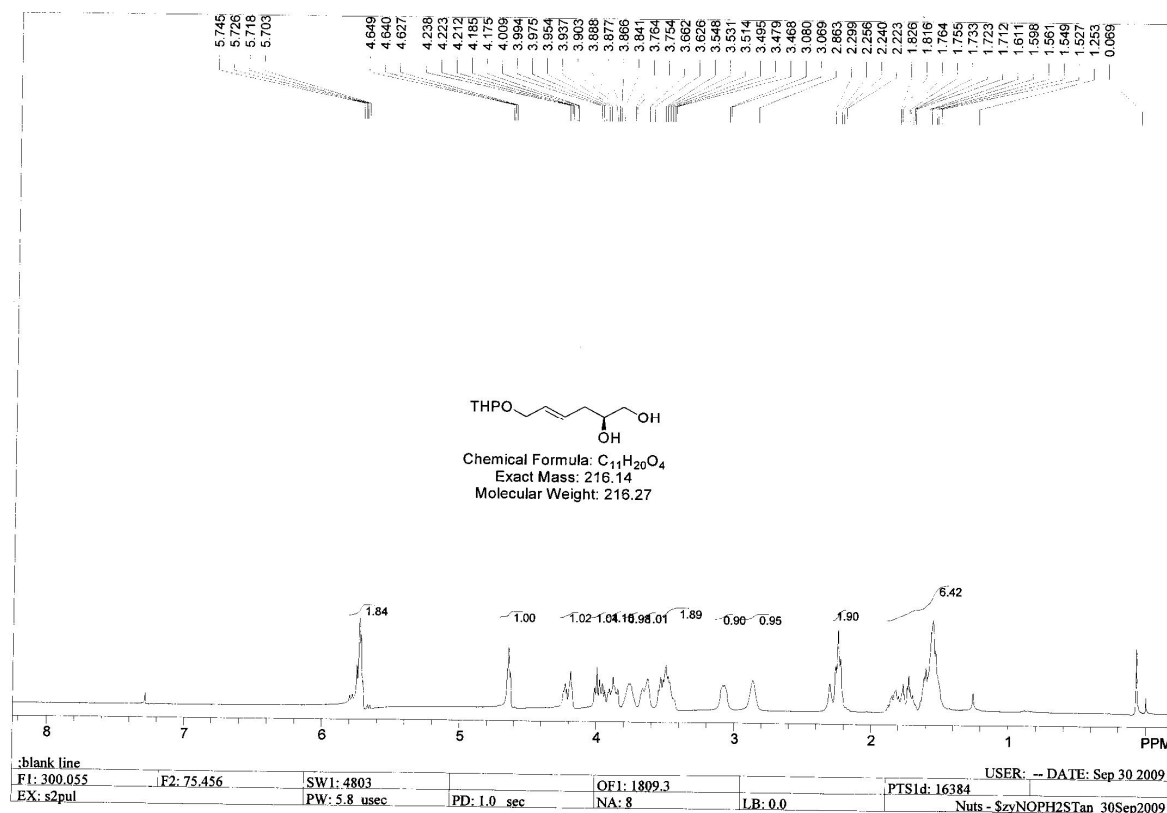
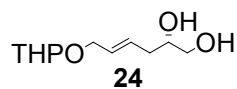


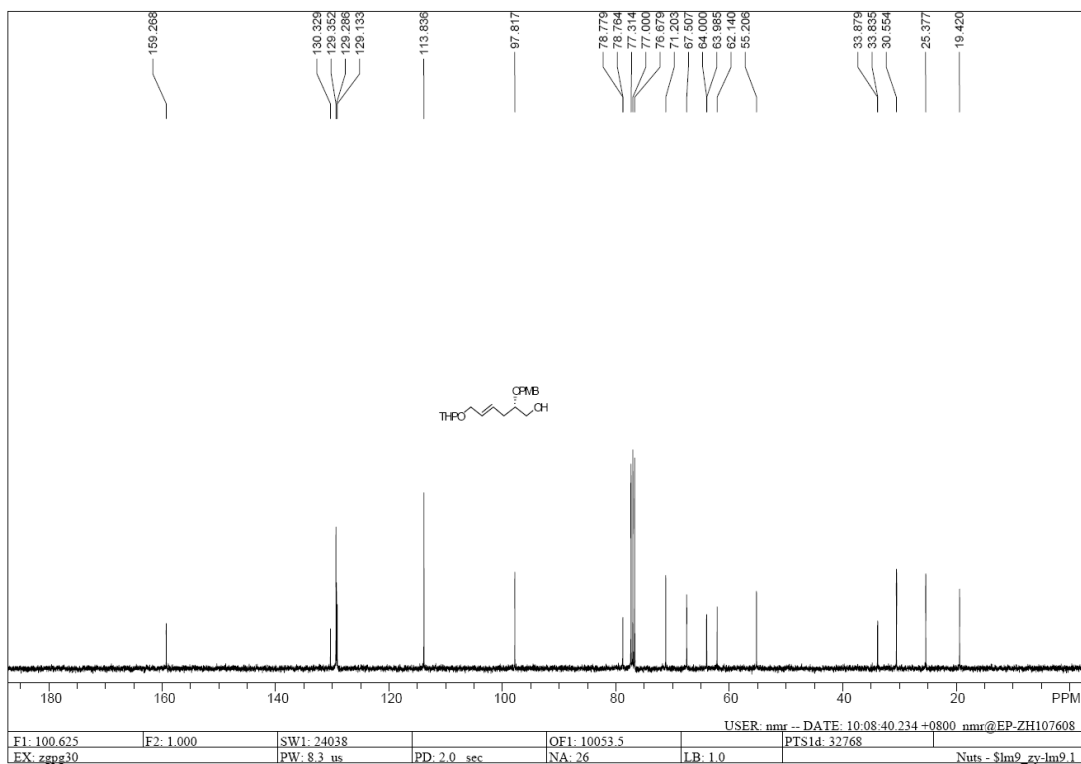
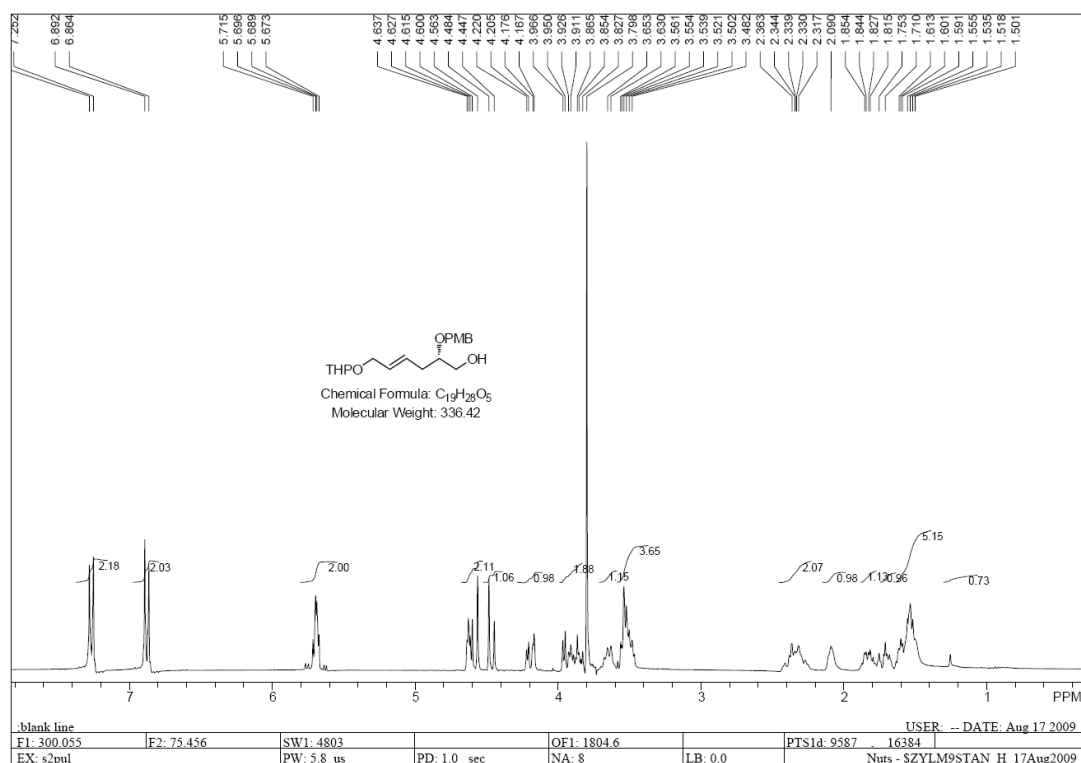
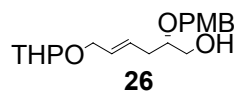


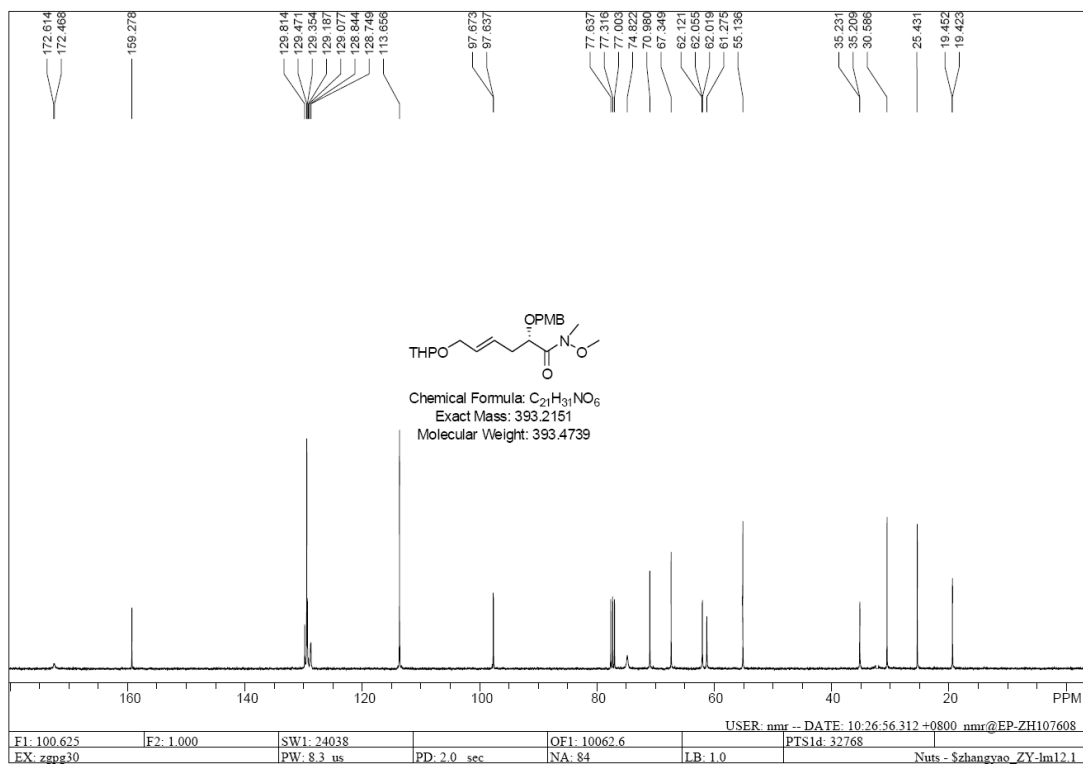
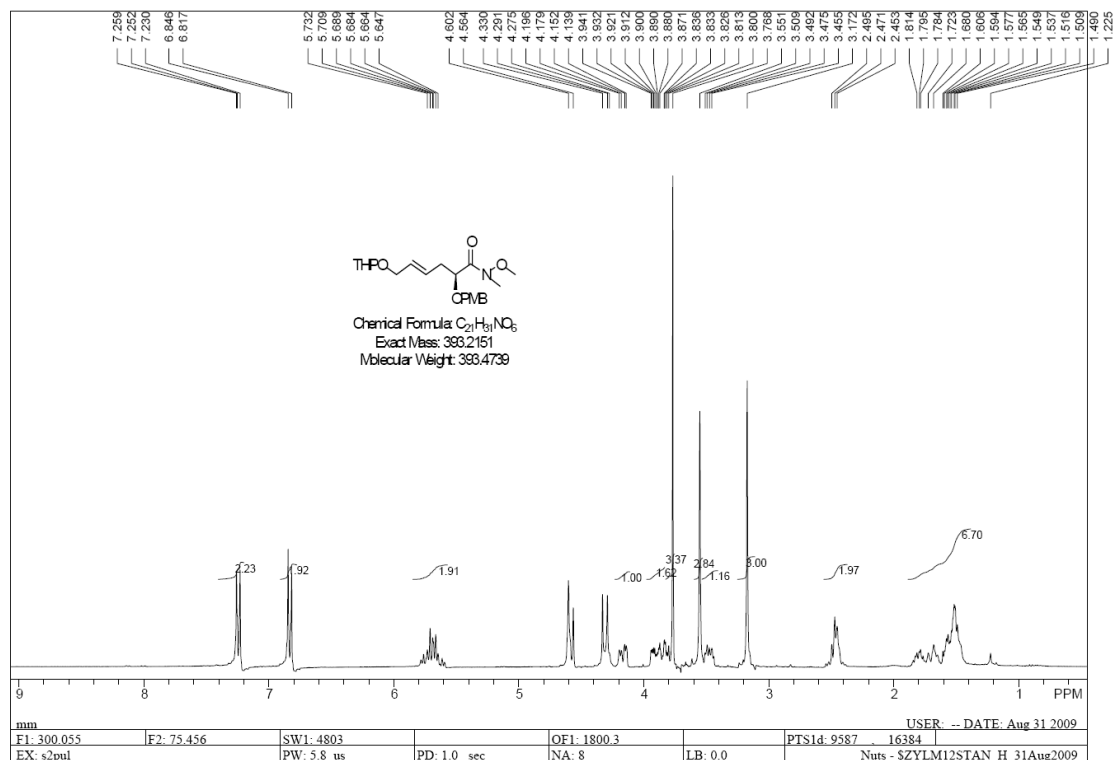
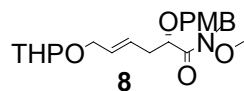


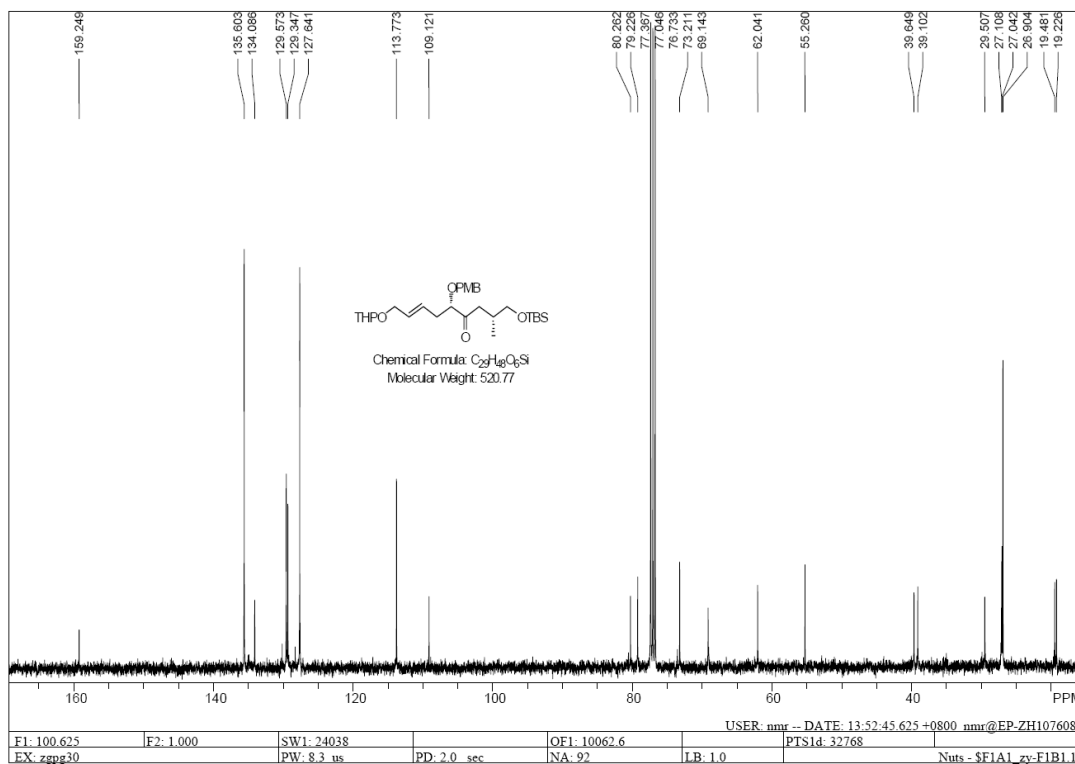
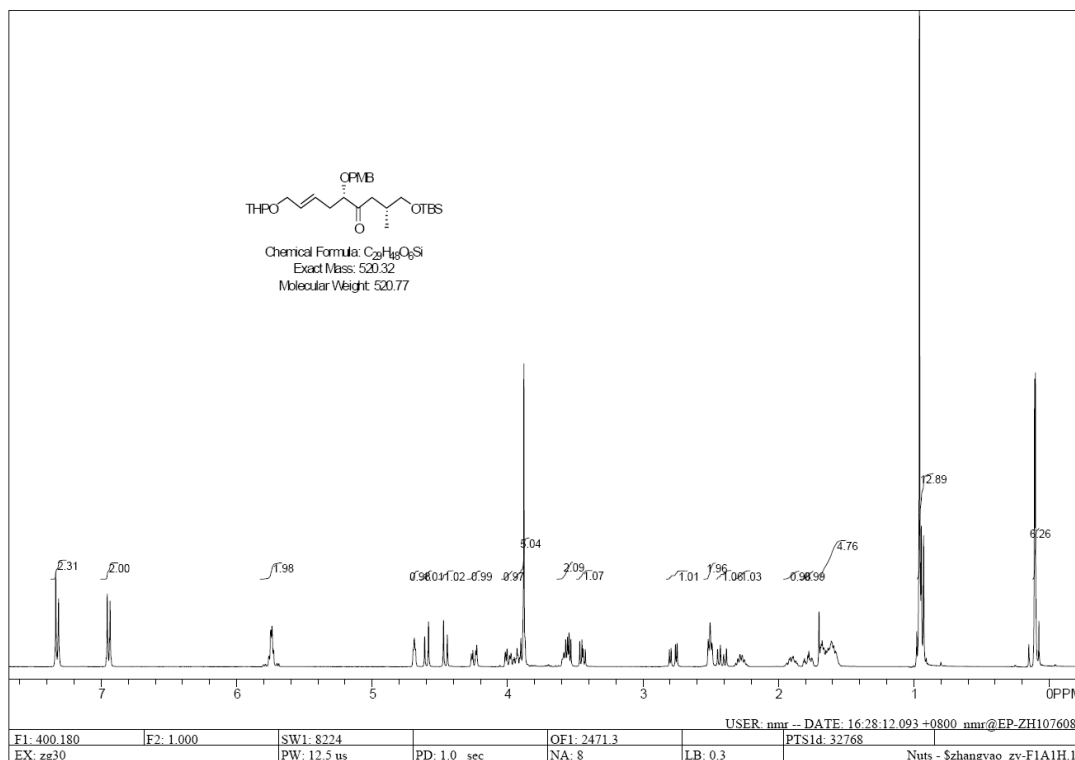
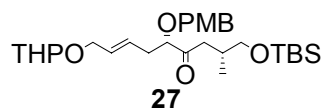


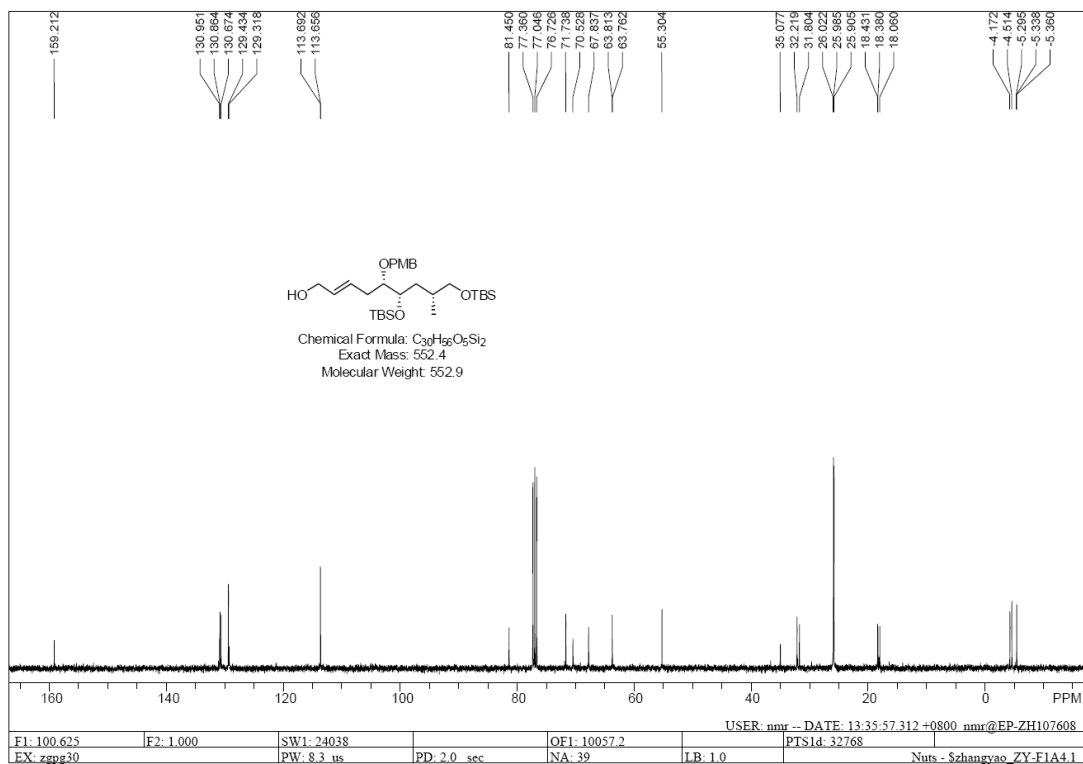
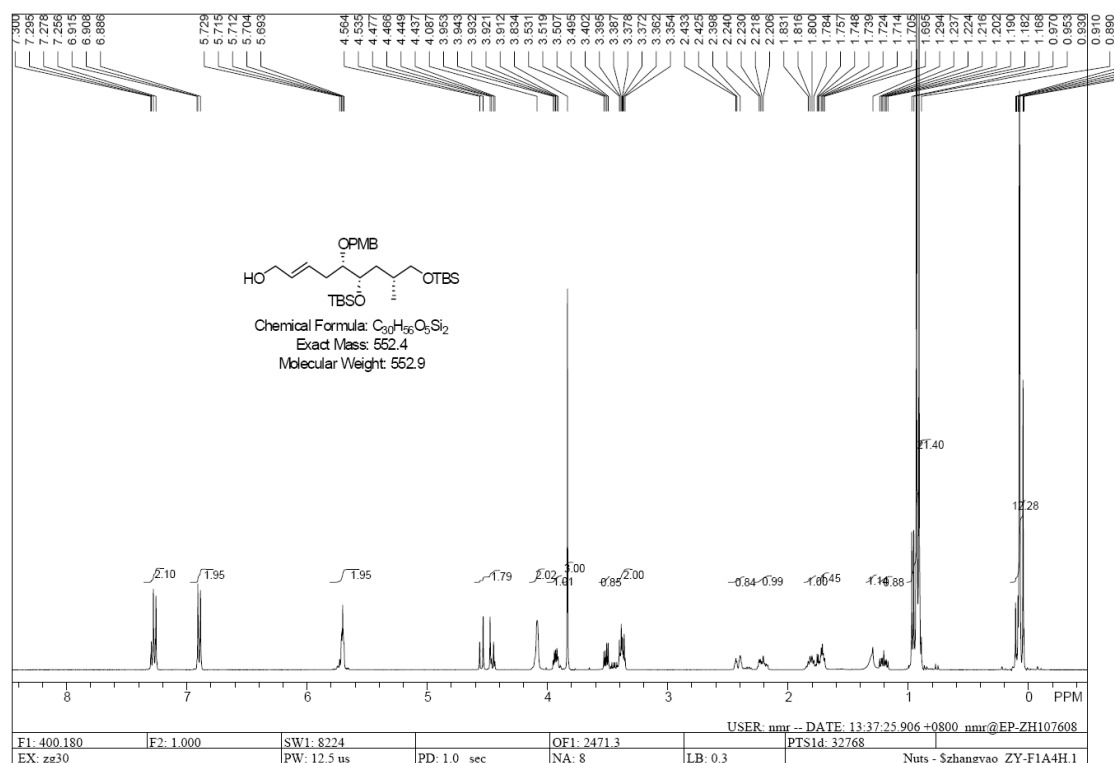
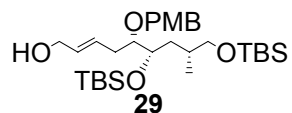


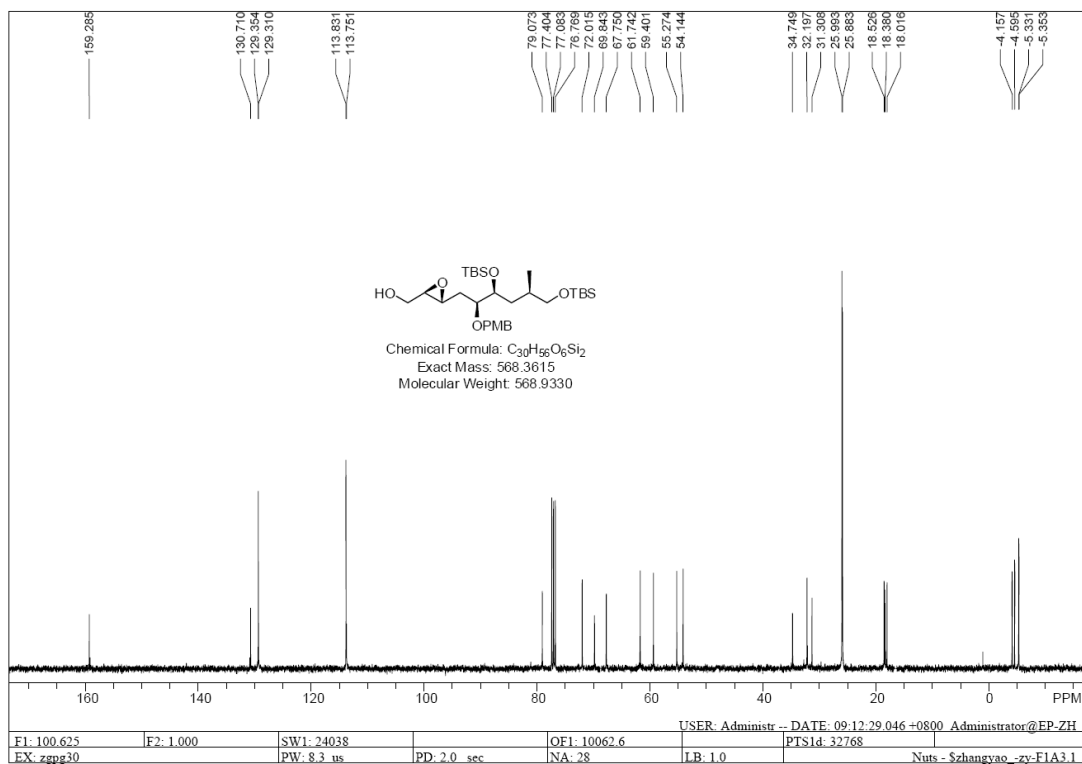
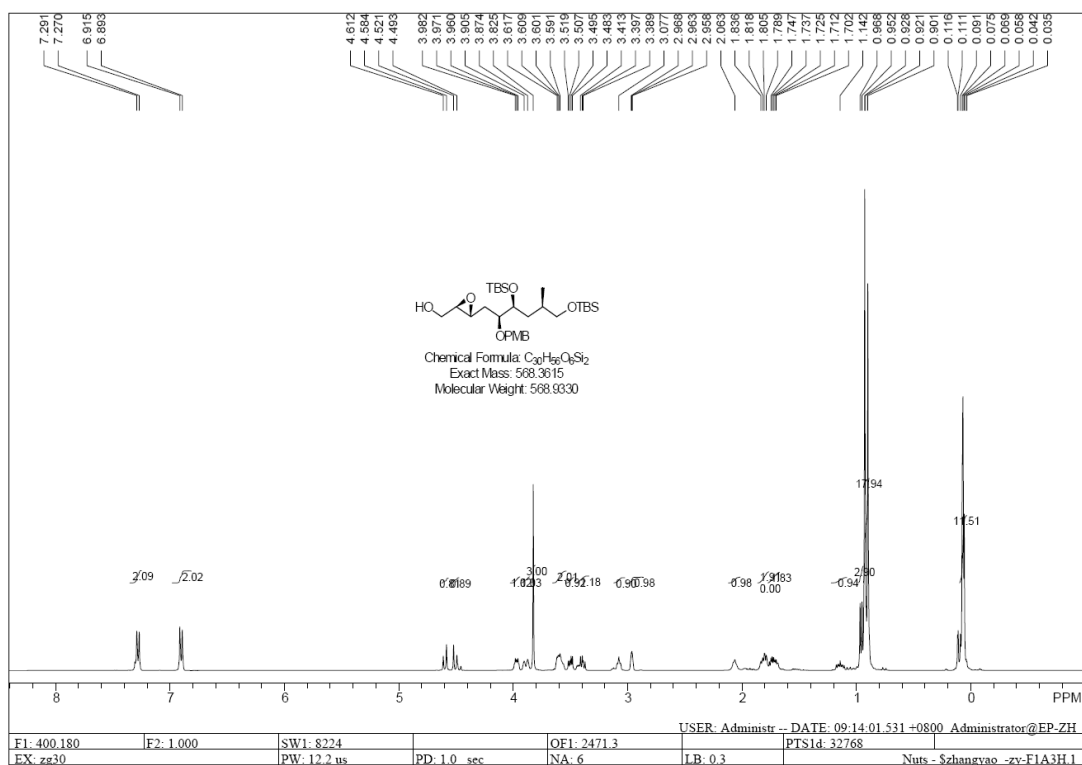
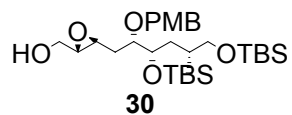


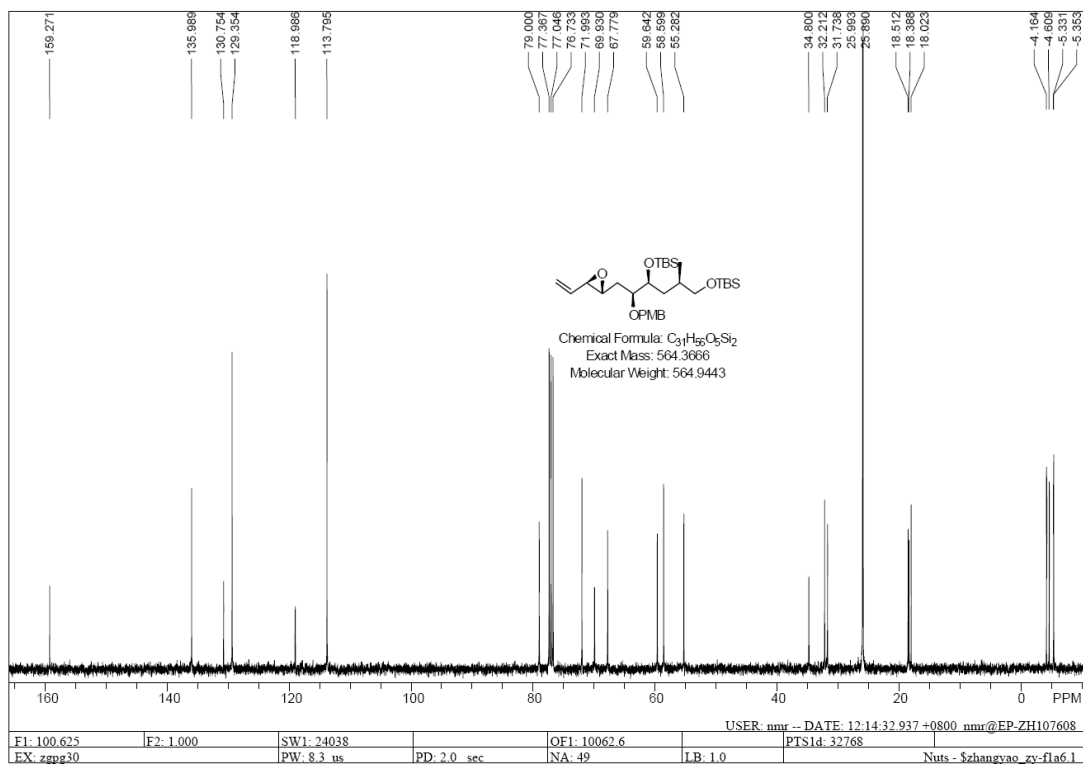
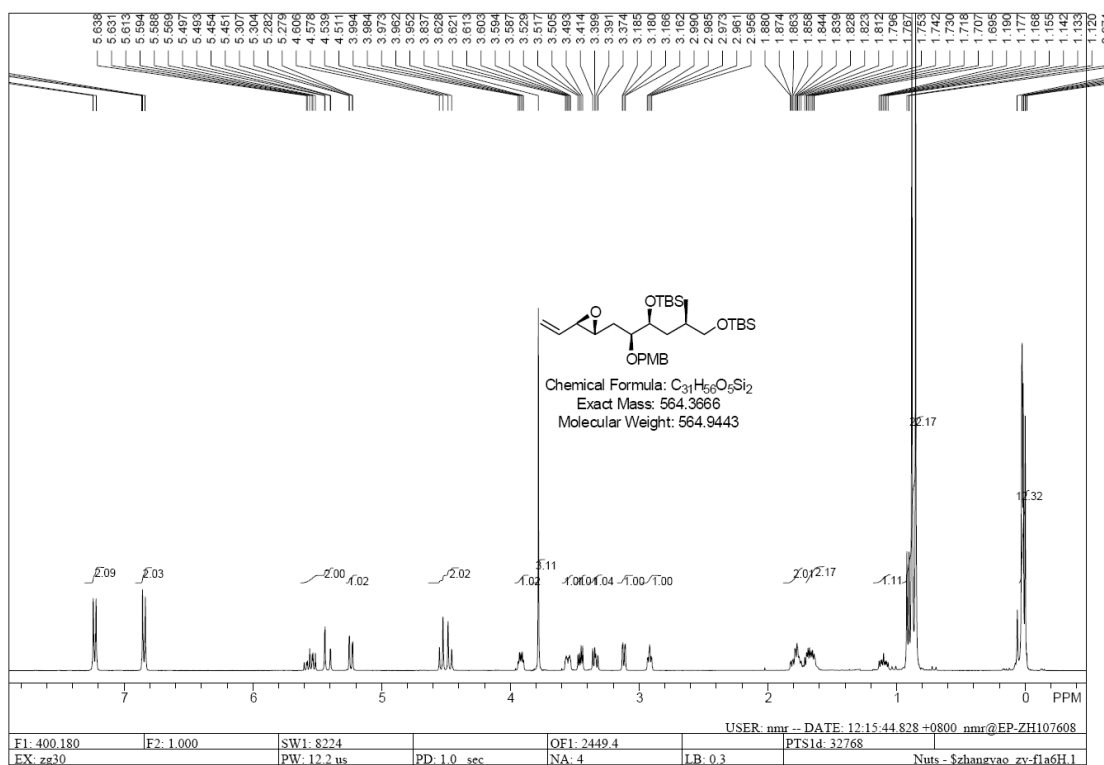
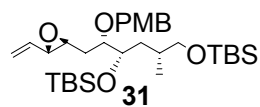


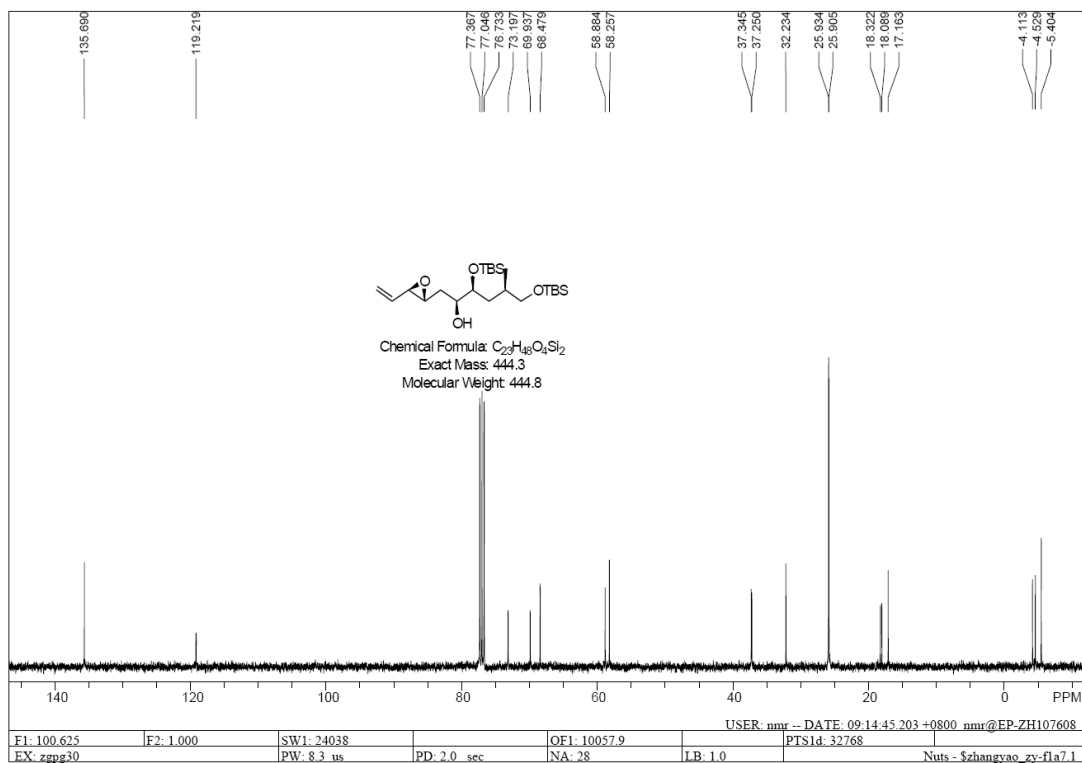
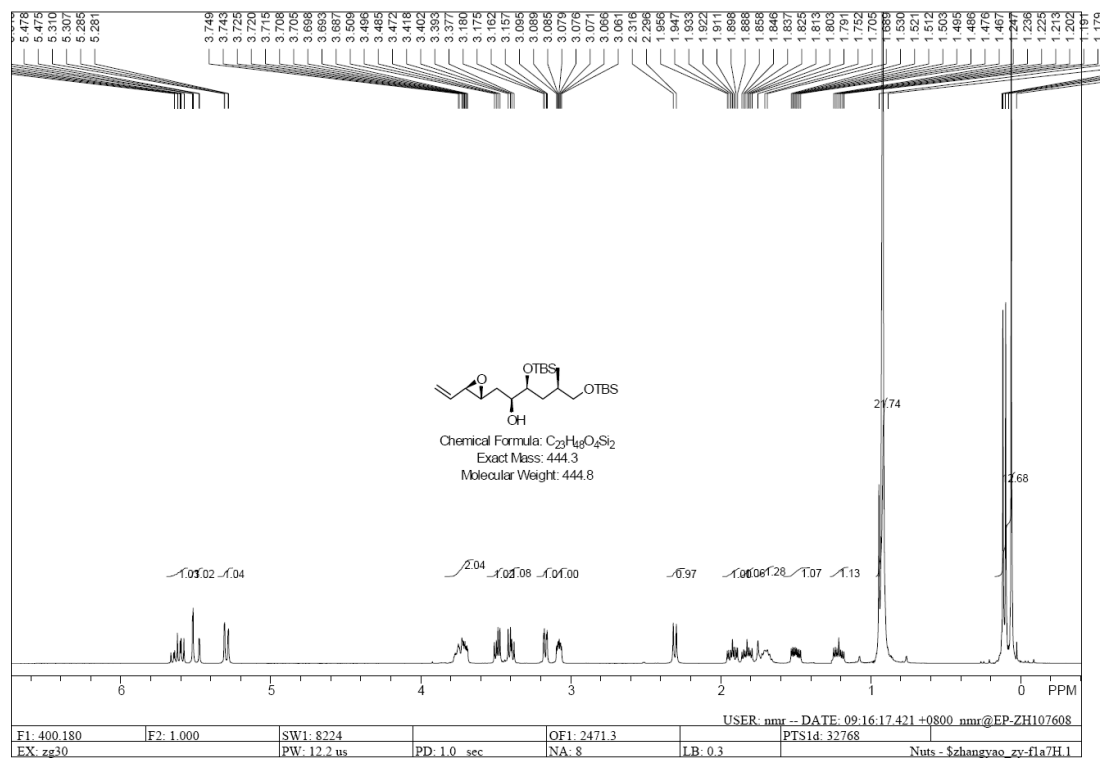
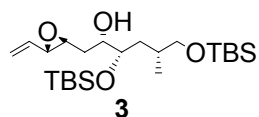


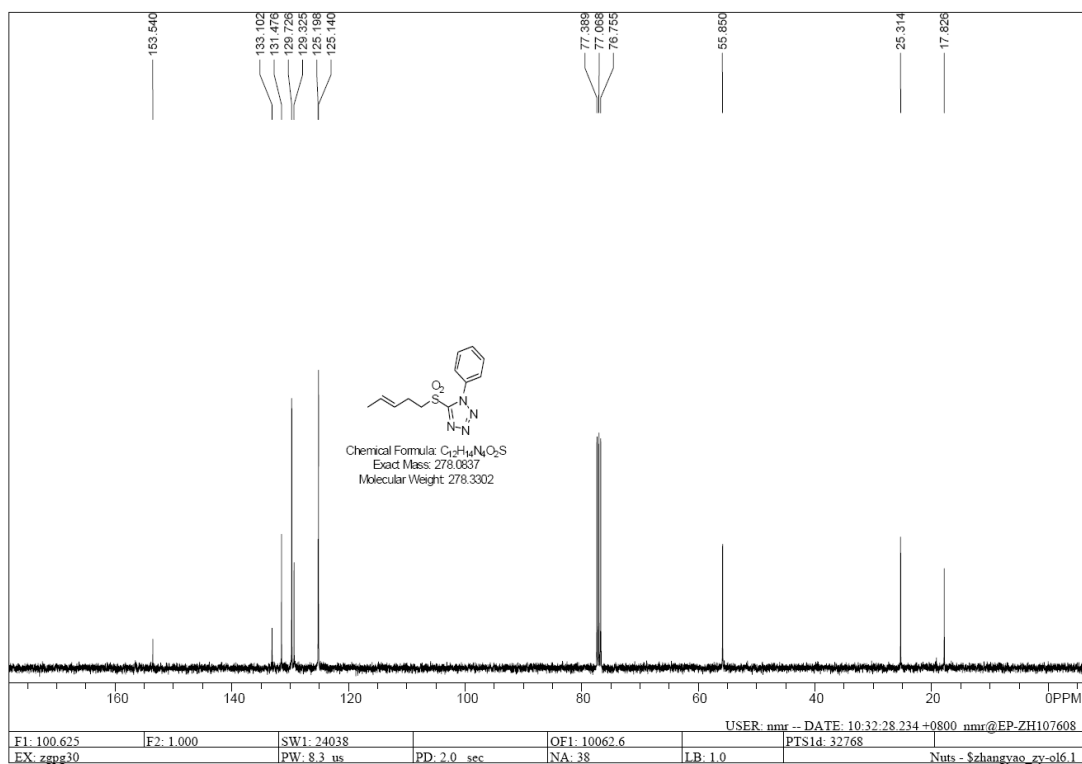
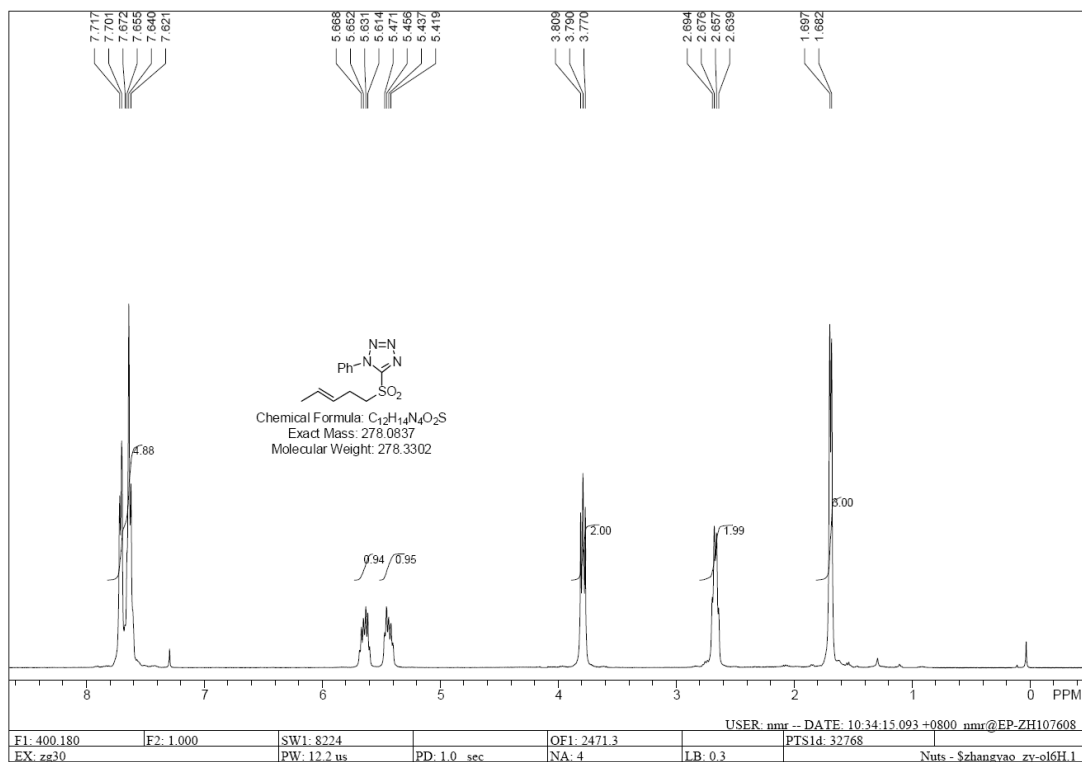
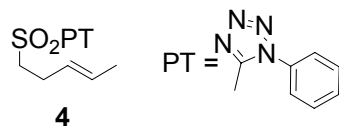


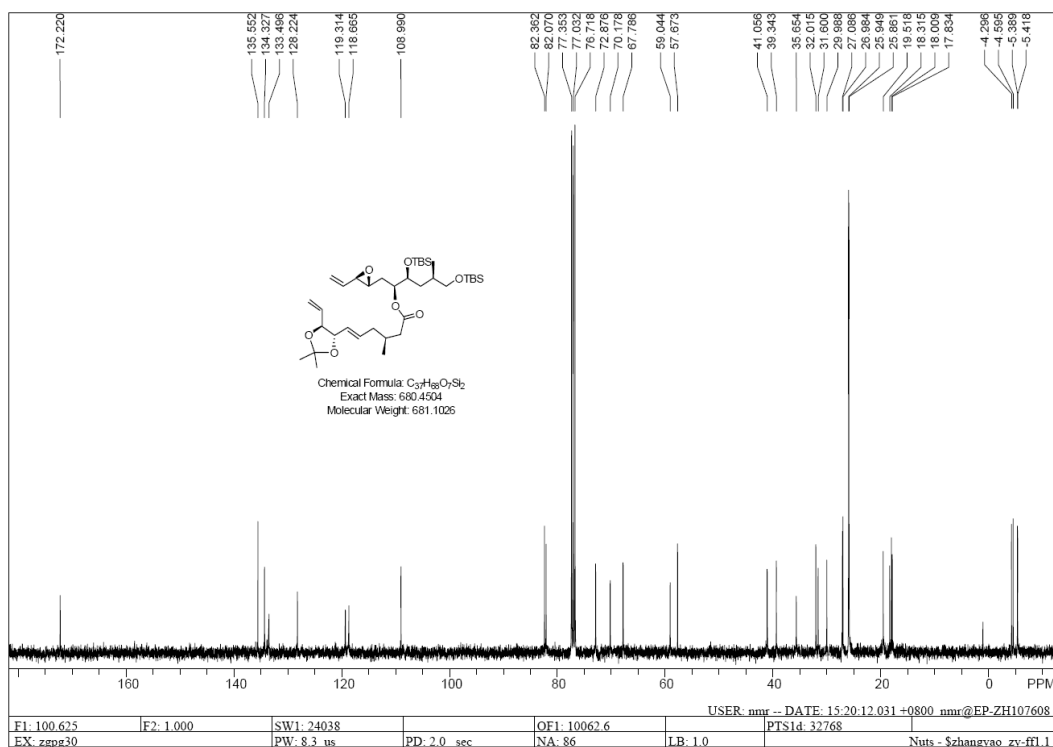
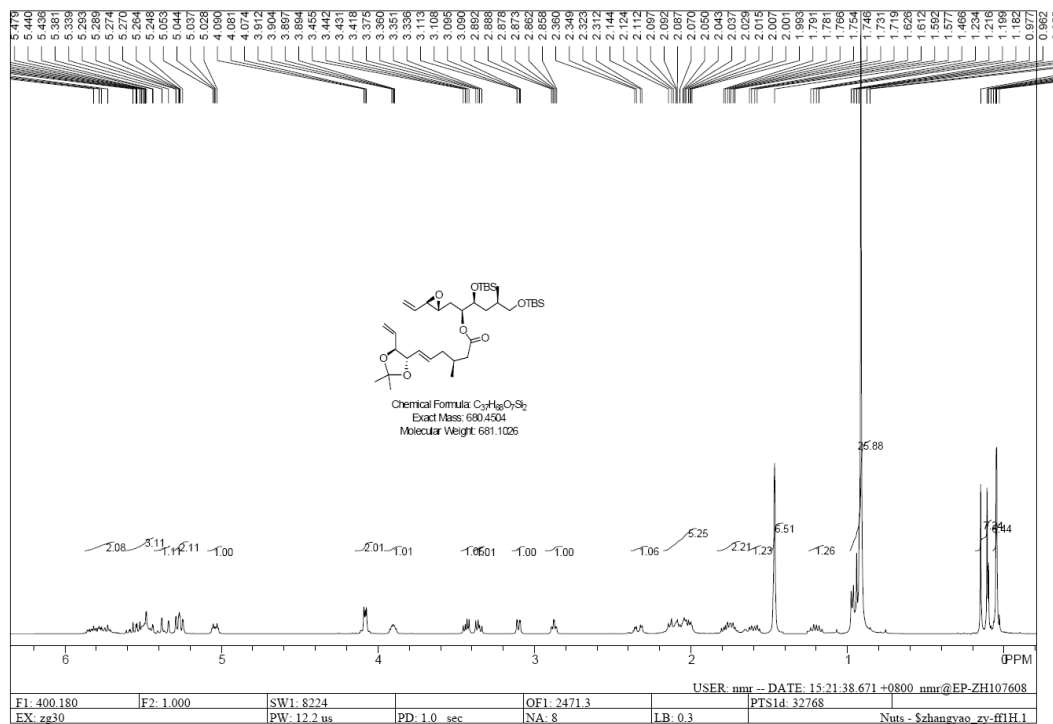
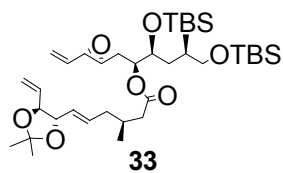


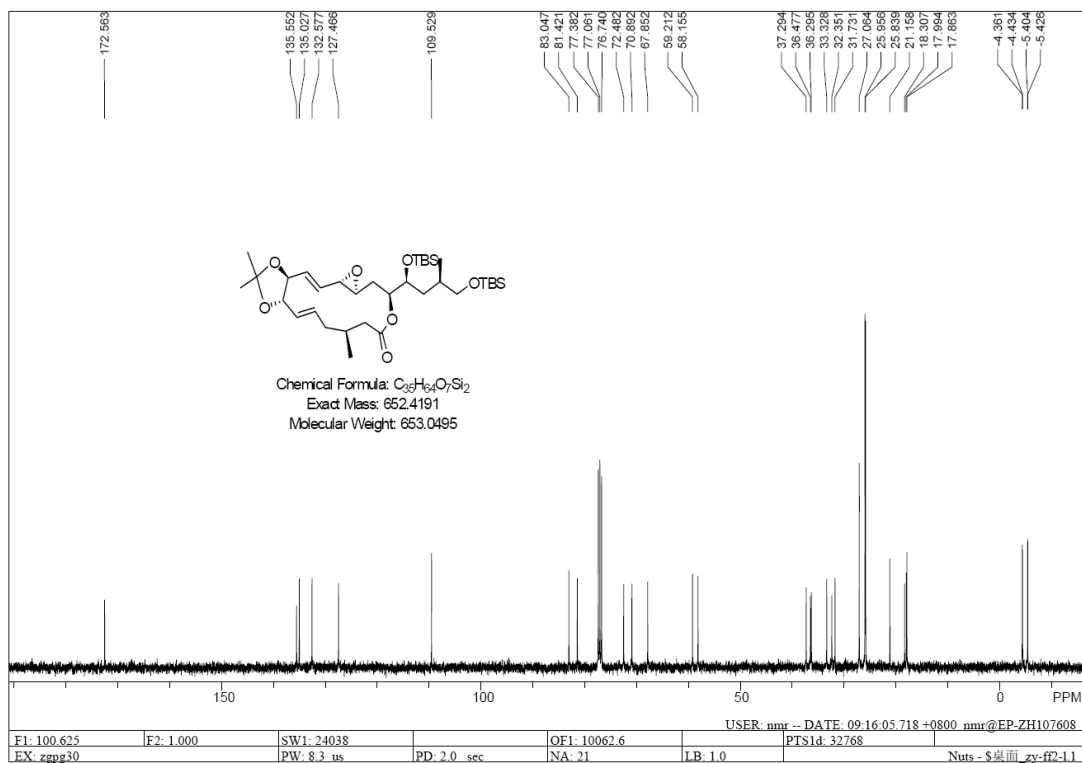
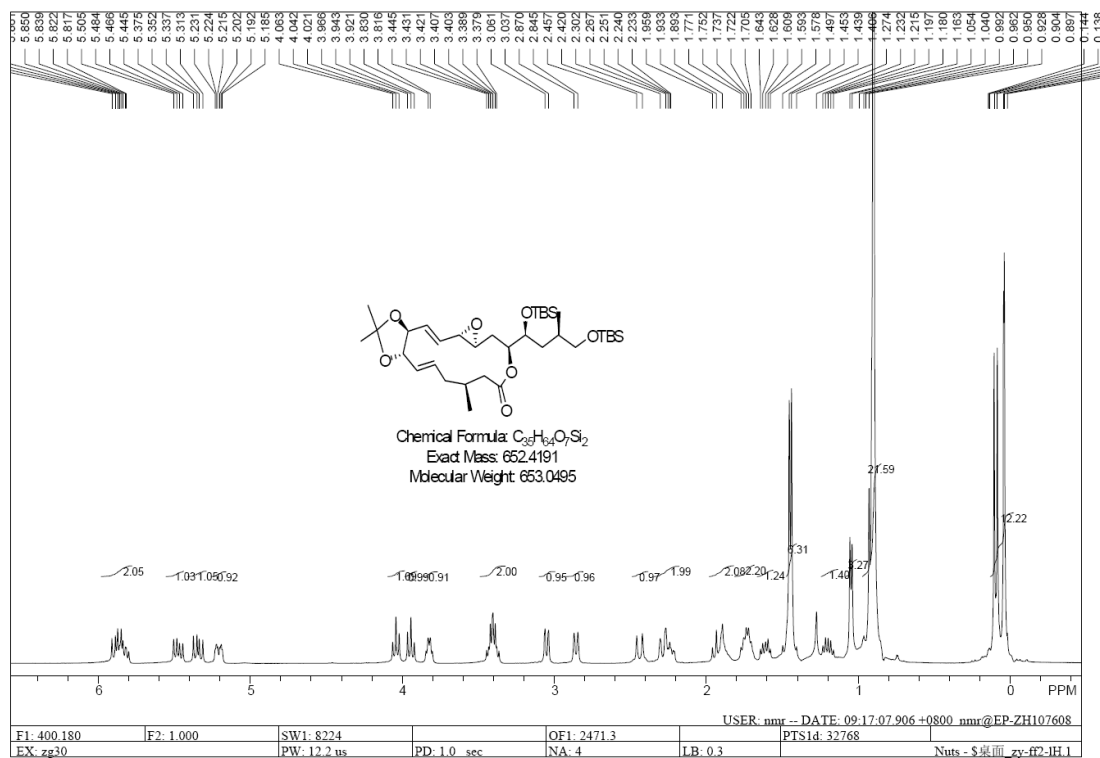
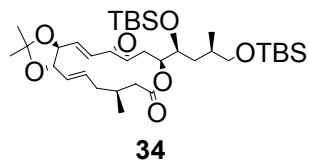


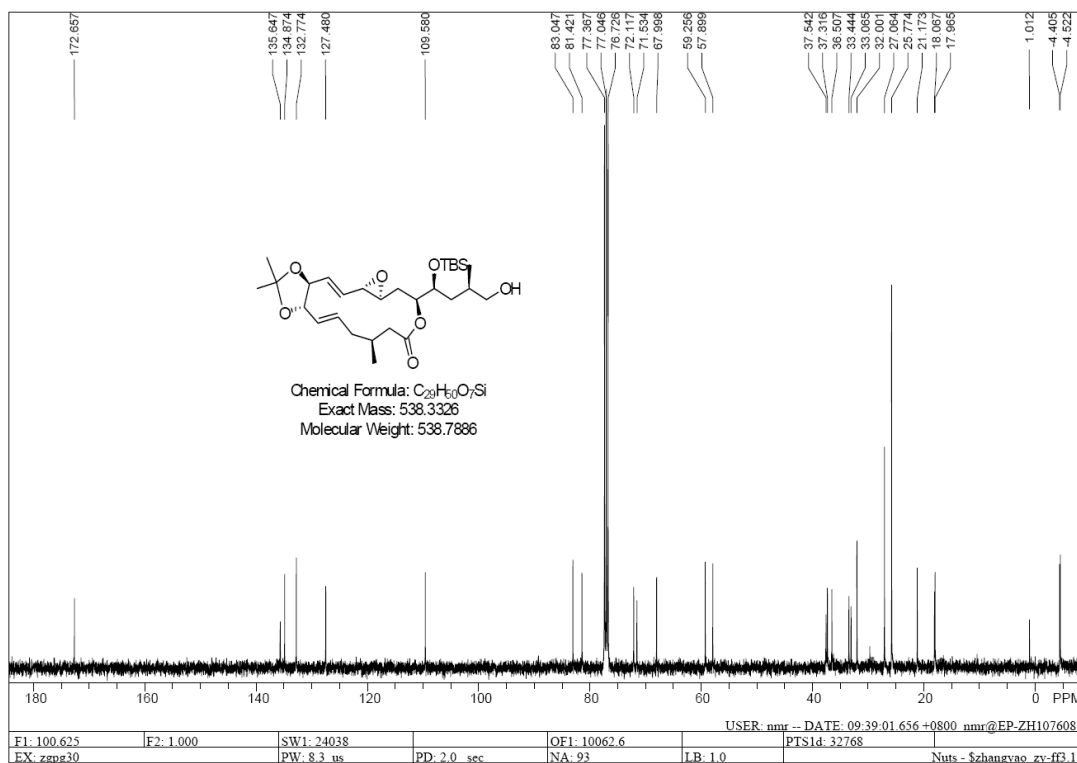
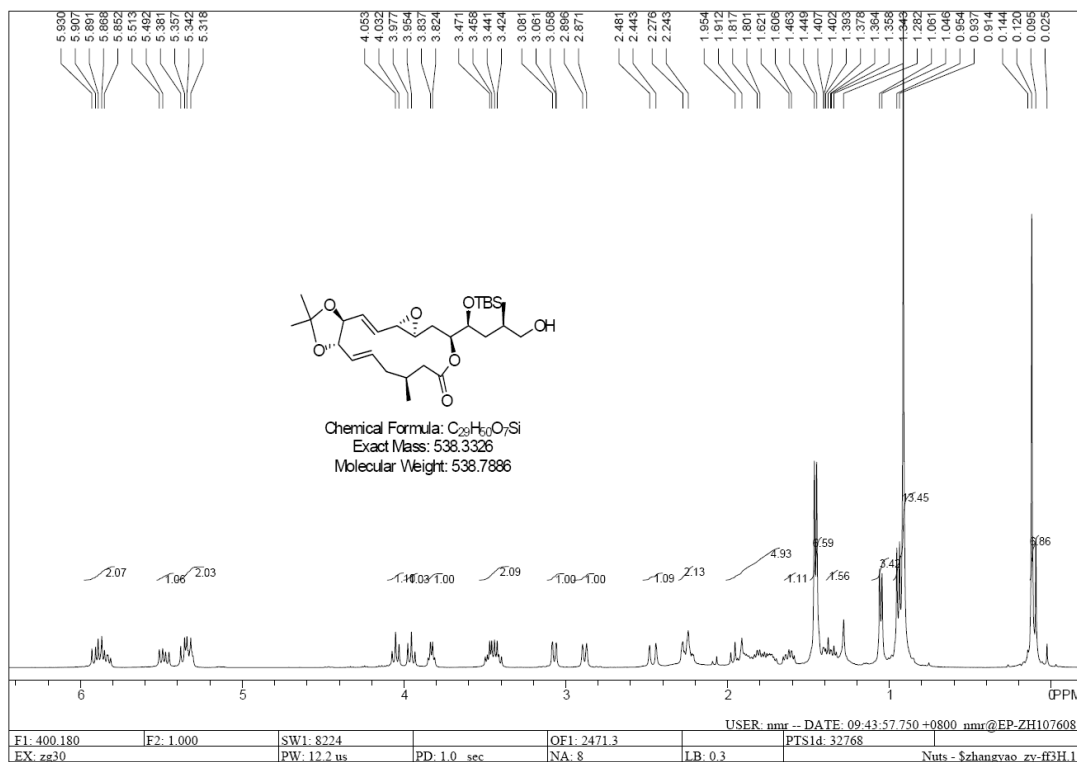
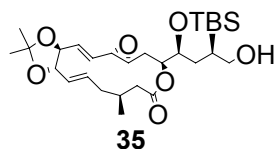


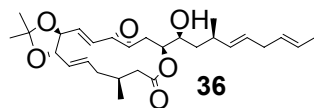




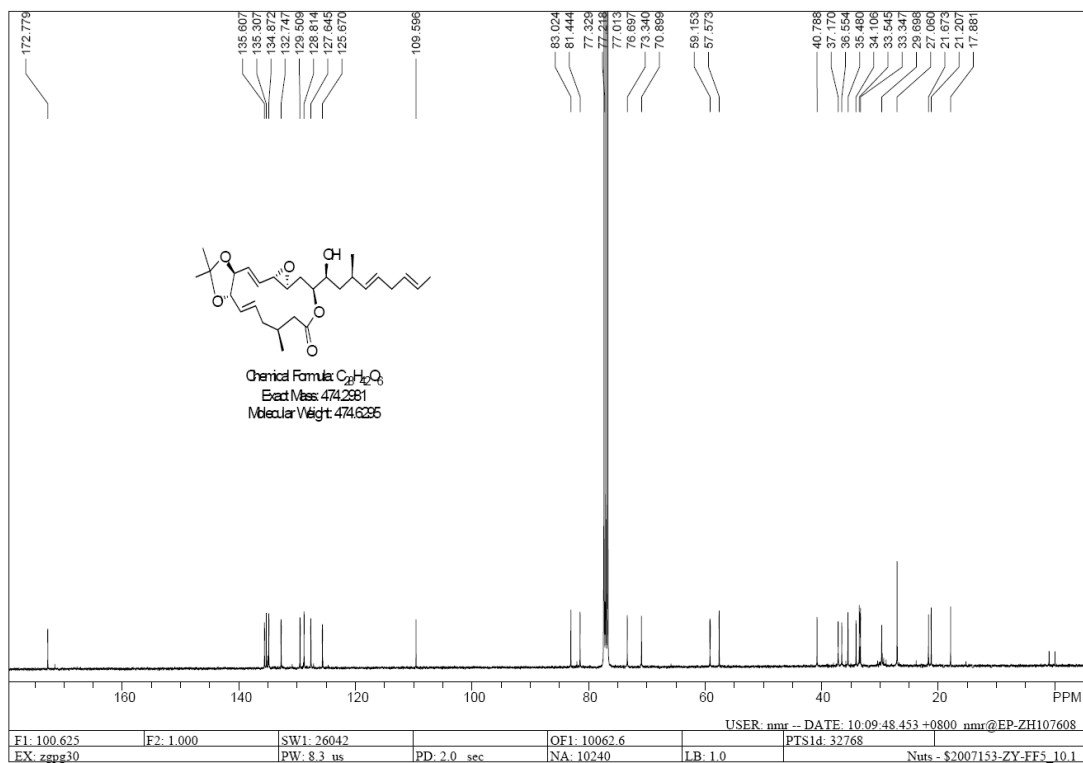
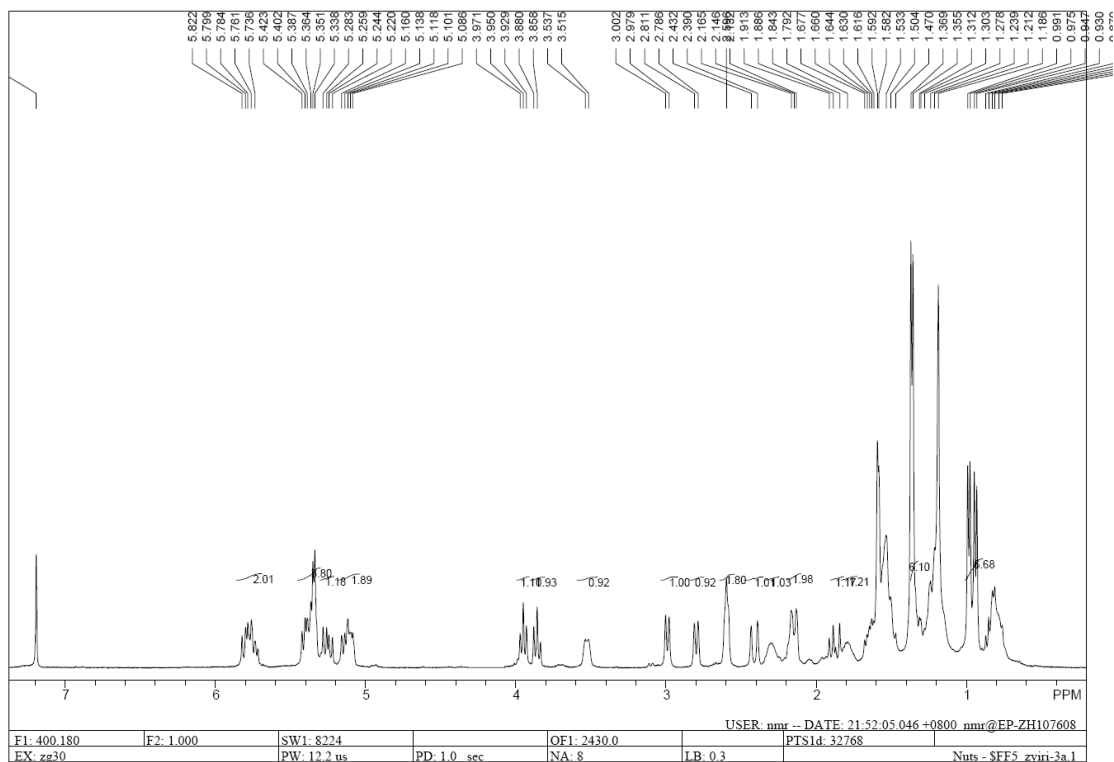




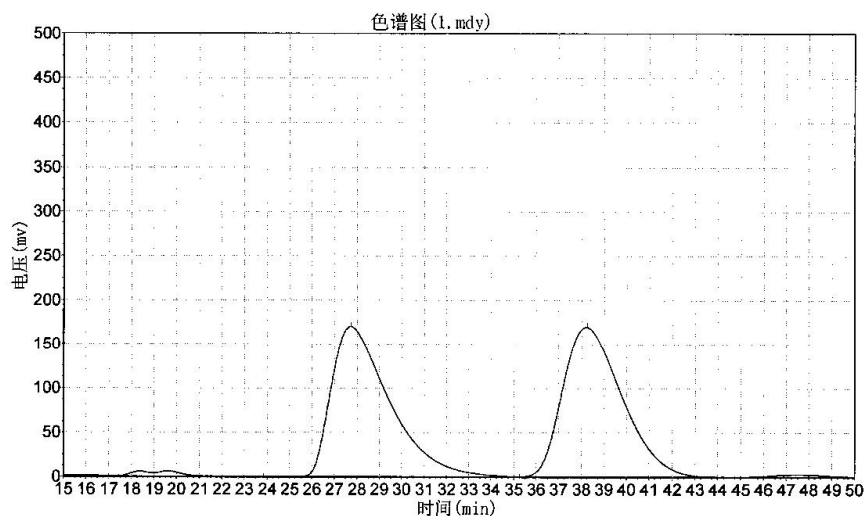




7,8-O-isopropylidene Iriomoteolide-3a (1)

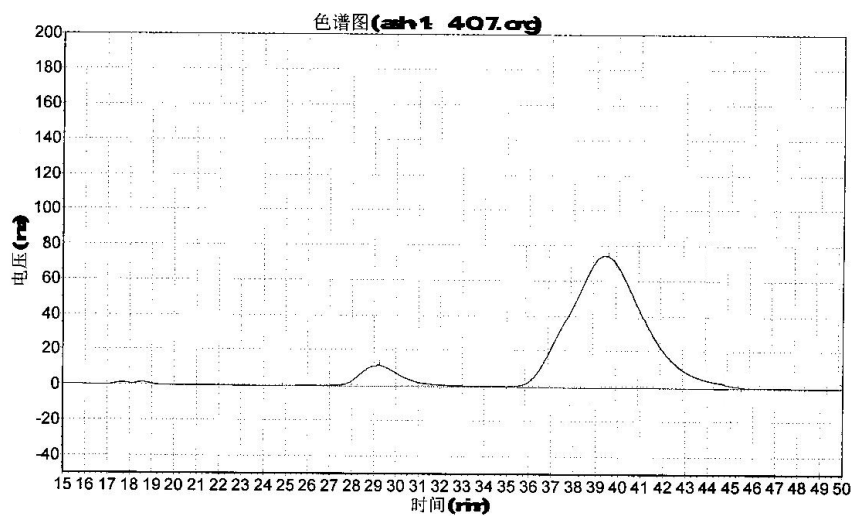


HPLC spectrum for **23**



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		27.723	169905.625	30794914.000	49.6635
2		38.257	170205.500	31212188.000	50.3365
总计			340111.125	62007102.000	100.0000



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		29.28	11938.211	189101.625	8.3444
2		41.68	74865.089	1746550.000	91.6556
总计			86803.300	1935651.625	100.0000