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Microbial Transformation of *Calamintha glandulosa* Essential Oil by *Aspergillus niger*

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Supplementary data

Text S1. General experimental procedures:

Silica gel 60 (SiO2; under 0.063 mm, Merck) was used for the column chromatography. Analytical and preparative TLC were carried out on silica gel 60 GF254 20 × 20 cm plates, layer thickness 0.25 mm (Merck). NMR spectra (1H, 13C, HSQC, HMBC) were recorded on a Varian 500-PS spectrometer at 500 MHz for 1H and 125 MHz for 13C, with CDCl3 as solvent and TMS as reference. GC/MS was conducted on an Agilent Technologies 6890N gas chromatograph coupled with a mass detector Agilent Technologies 5973, provided with a DB 5 (30 m × 0.25 mm ID × 0.25 μm df) capillary column. The analyses were performed in EI mode (70eV) using He at 1 mL/min. The injection temperature was set at 250 ºC. The analyses were carried out using a temperature program starting from 50 ºC with an initial 5 min hold to 250 ºC, with a 10 ºC/min heating increase and keeping the final temperature stable for 20 min. The mass range was set at m/z 40-500 with 3 scans. Transfer line was set at 280 ºC. Co-injection of the extracts with C9-C25 hydrocarbons was performed under the same conditions.

Table S1. Elution system for the silica gel column chromatography separation of biotransformed products

V (ml)	100	700	200	200	200	300	300	100	100	300	100	300	300
n-hexane (%)	100	95	94	93	92	91	90	89	88	87	86	85	84
EtOAc (%)	0	5	6	7	8	9	10	11	12	13	14	15	16
Fraction number	-	0-21	22-30	31-39	40-48	49-64	65-82	83-89	90-96	97-114	115-137	138-156	157-173

Compounds **1** and **2** were isolated from the fractions 24-26 and 21-23, respectively; compounds **3** and **4** from the fractions 42-48, and 76-80, respectively; compound **5**, **6**, and **7** from the fractions 65-70 and 59-64, respectively. Further separation and purification was done using preparative TLC plates of silica gel. The system used was n-hexane/ethyl acetate 85:15 for compounds **1**, **2**, **3**, and **4**, and 80:20 for compounds **5**, **6**, and **7**.

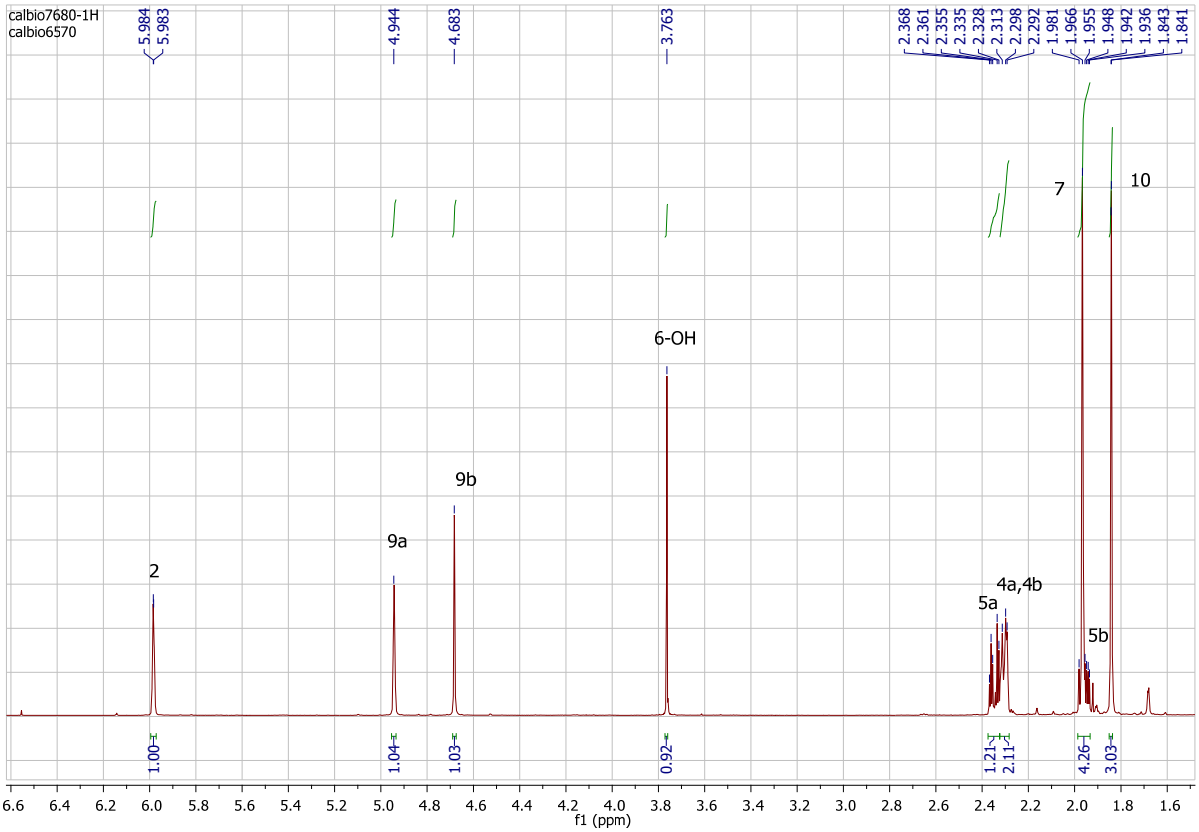

Figure 1S. ^{1}H NMR spectrum of compound **5**

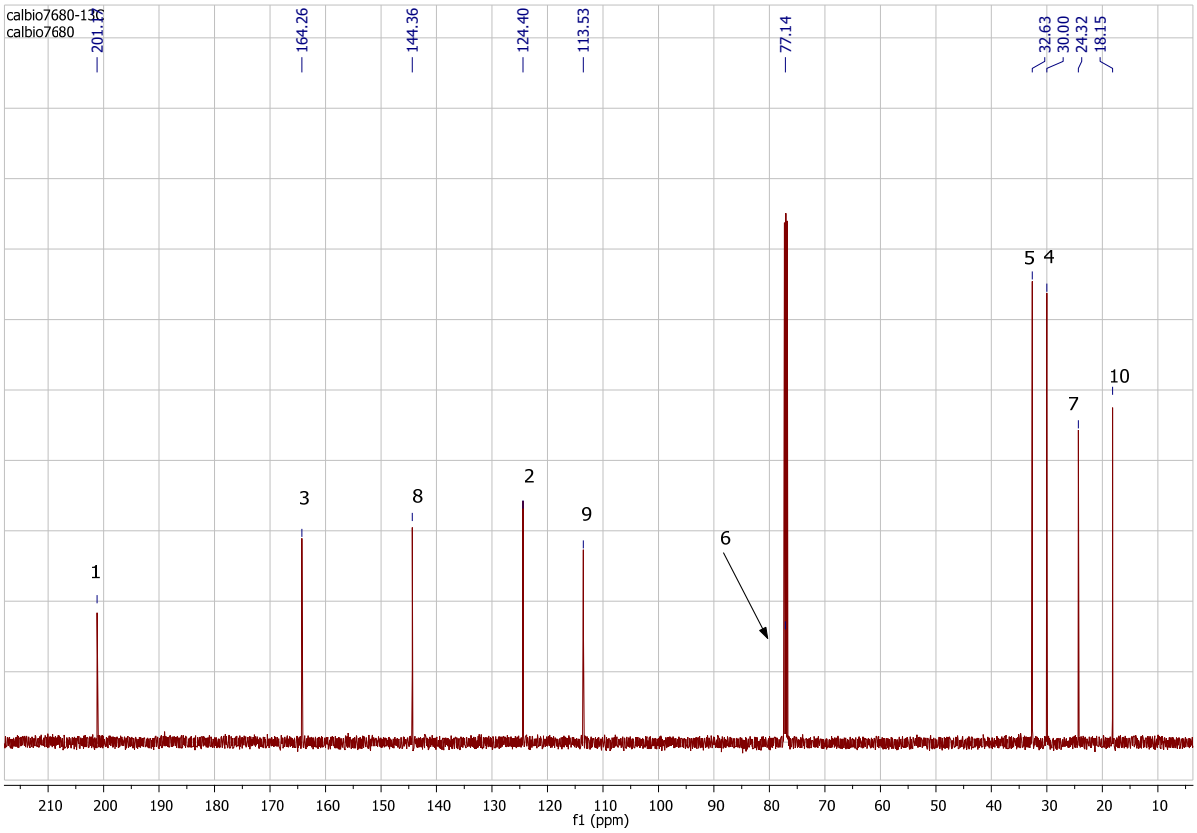

Figure 2S. ^{13}C NMR spectrum of compound **5**

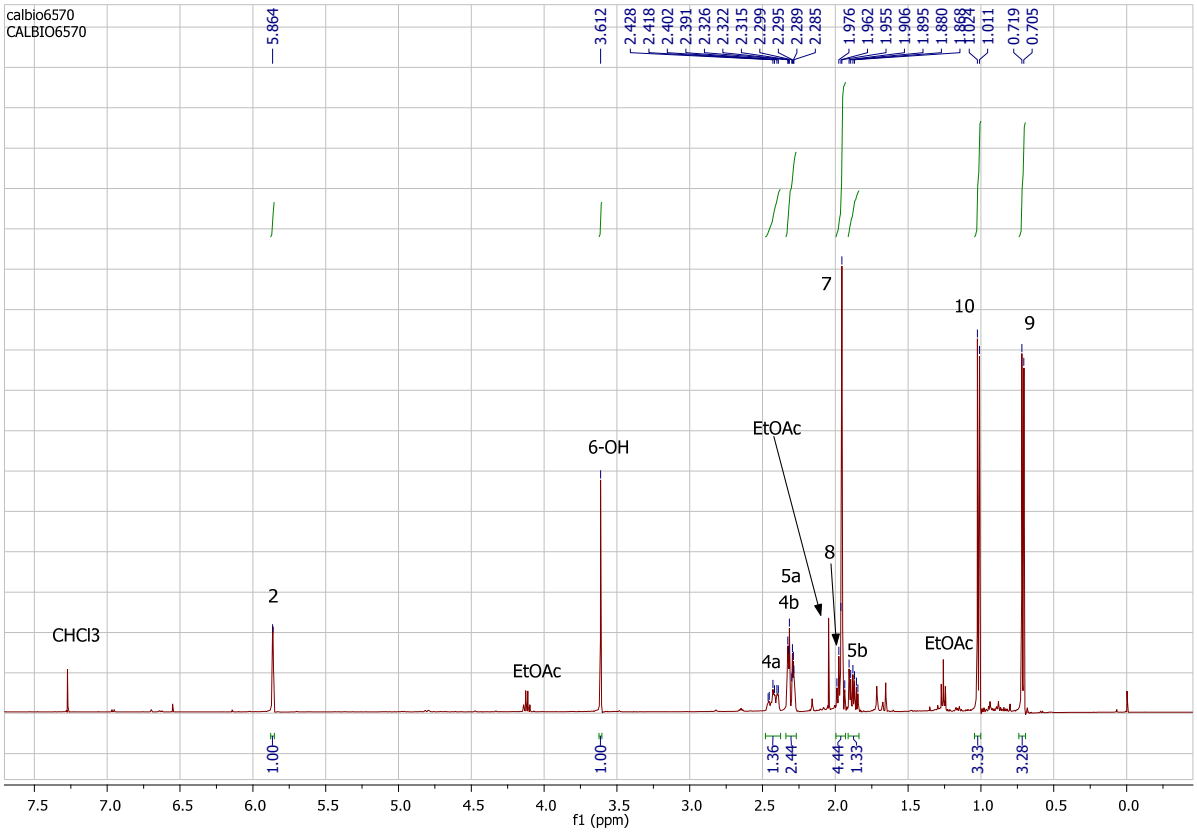

Figure 3S. ^{1}H NMR spectrum of compound **6**

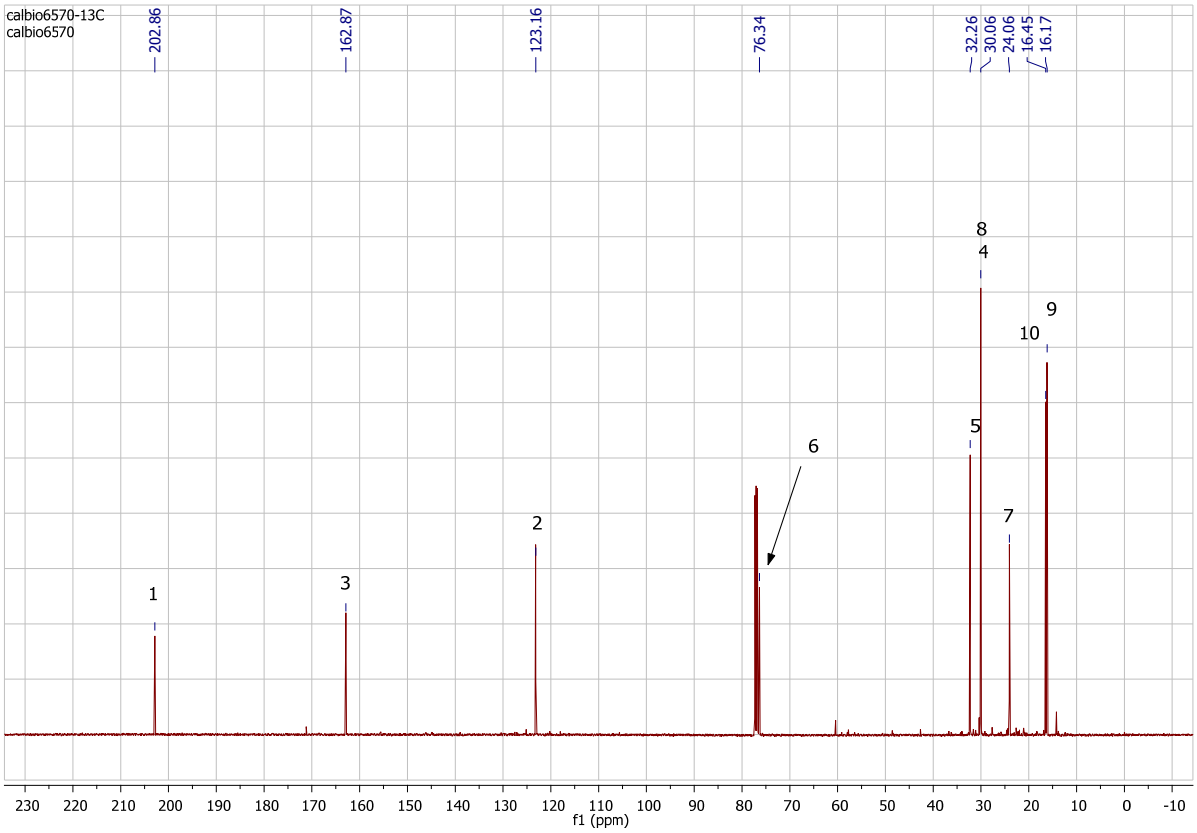

Figure 4S. ^{13}C NMR spectrum of compound **6**

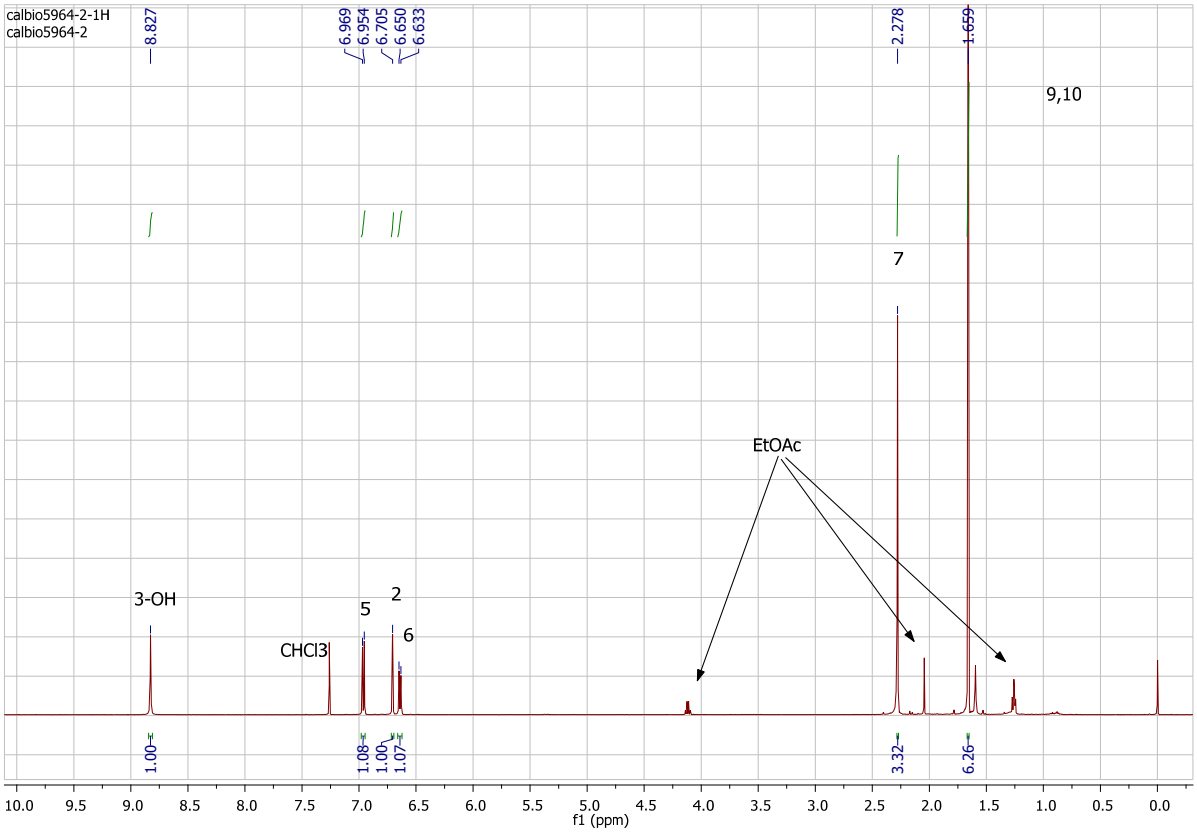

Figure 5S. ^{1}H NMR spectrum of compound **7**

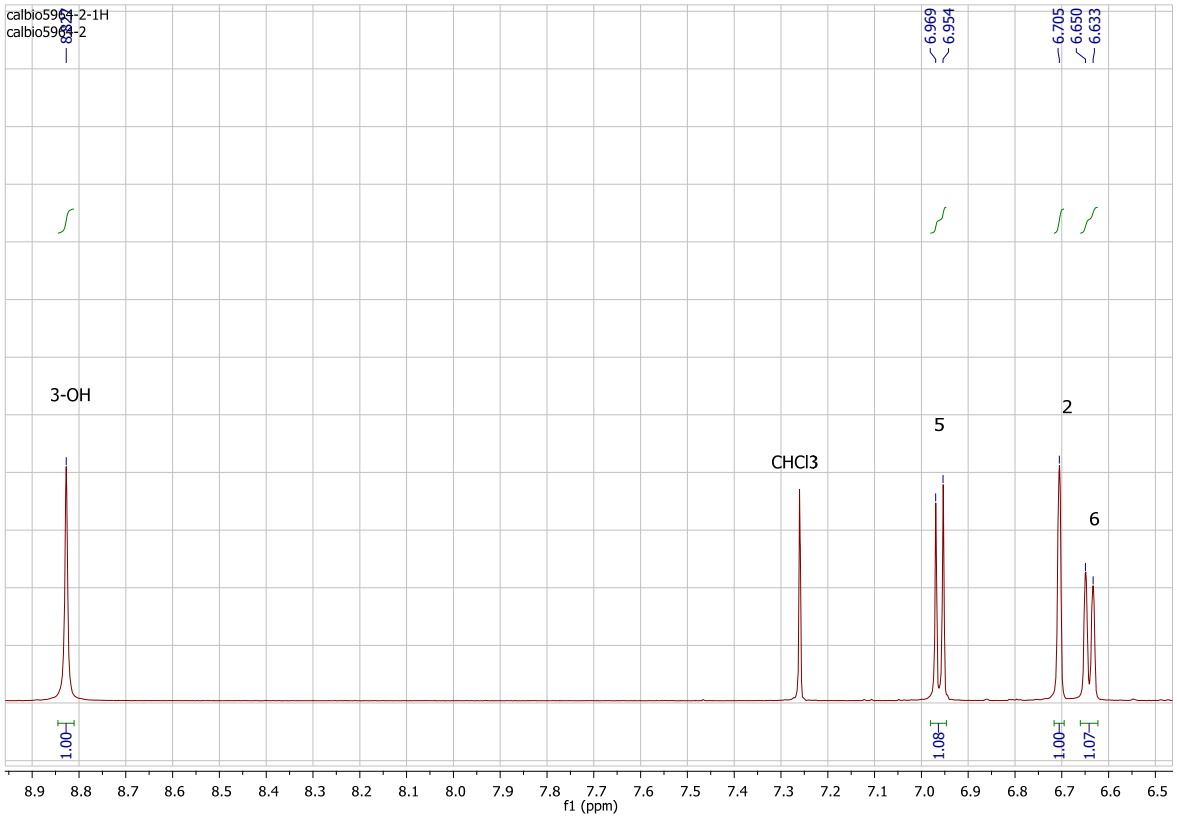

Figure 6S. Aromatic part of the ^{1}H NMR spectrum of compound **7**

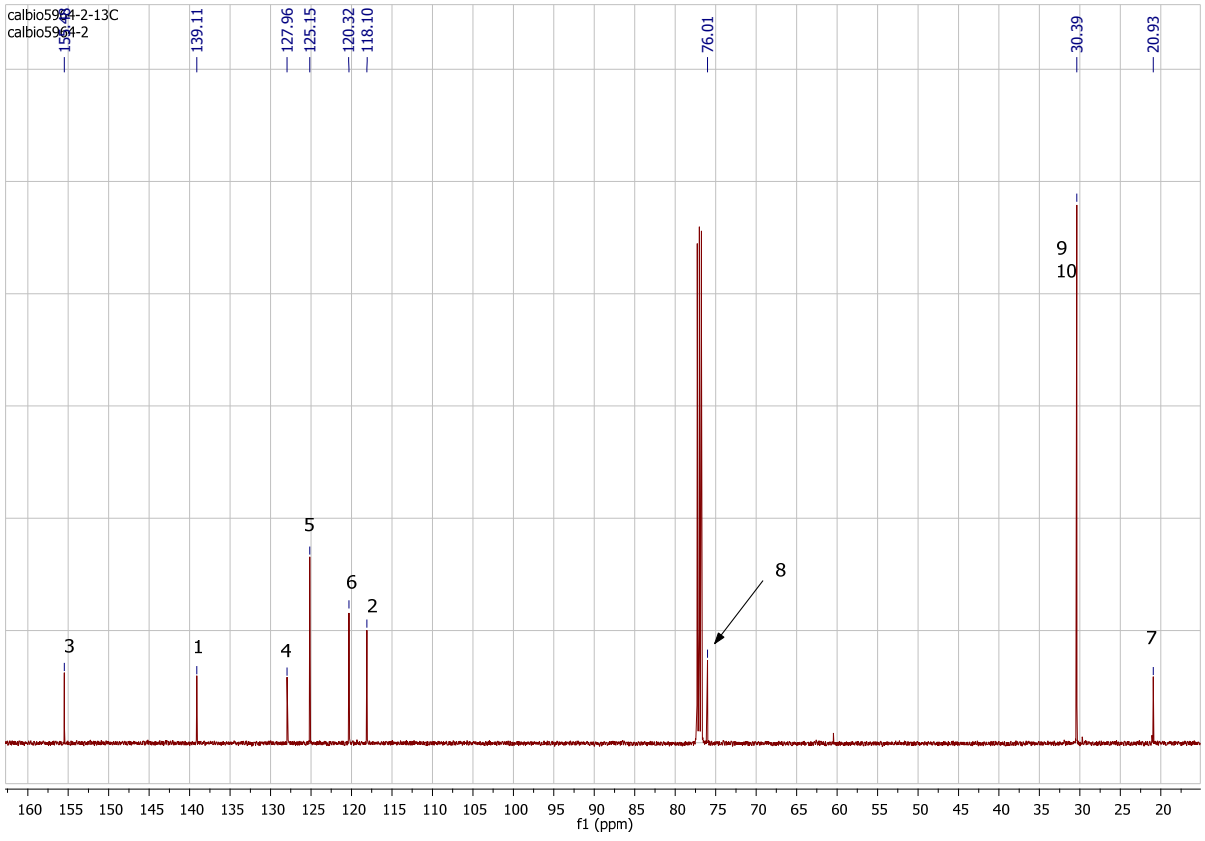

Figure 7S. ^{13}C NMR spectrum of compound **7**