Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Supporting Information

Concise construction of the tetracyclic core of lycorine-type alkaloids and the formal synthesis of α-lycorane based on asymmetric bifunctional thiourea-catalyzed cascade reaction

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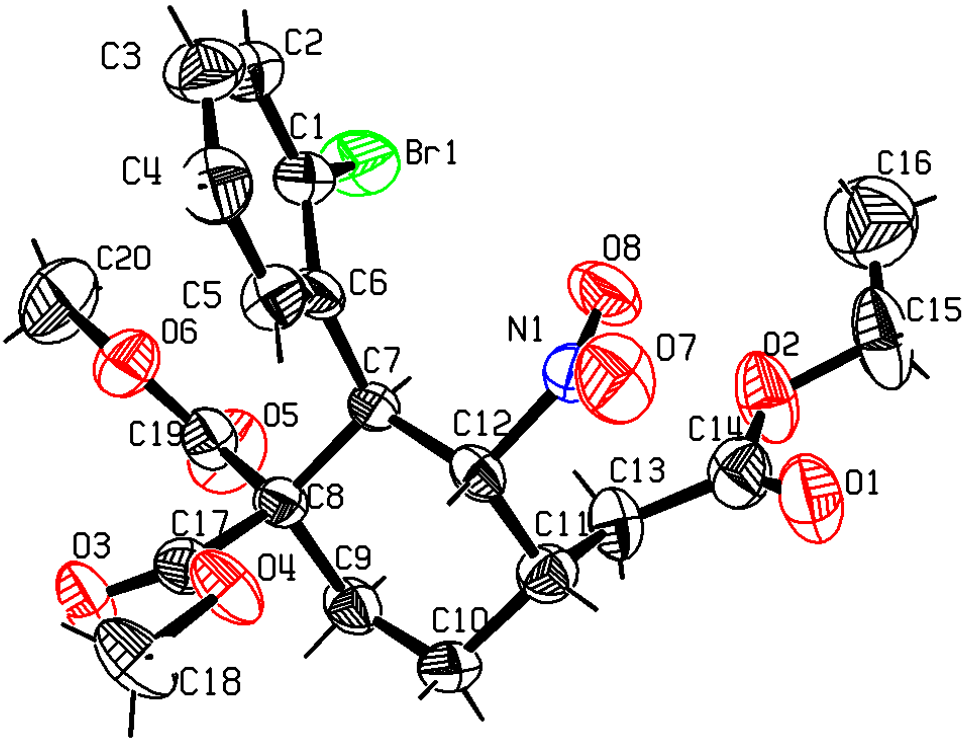
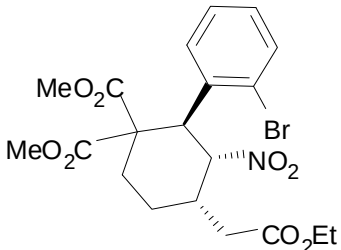
1. The X-ray crystal structure of the compound **3a**



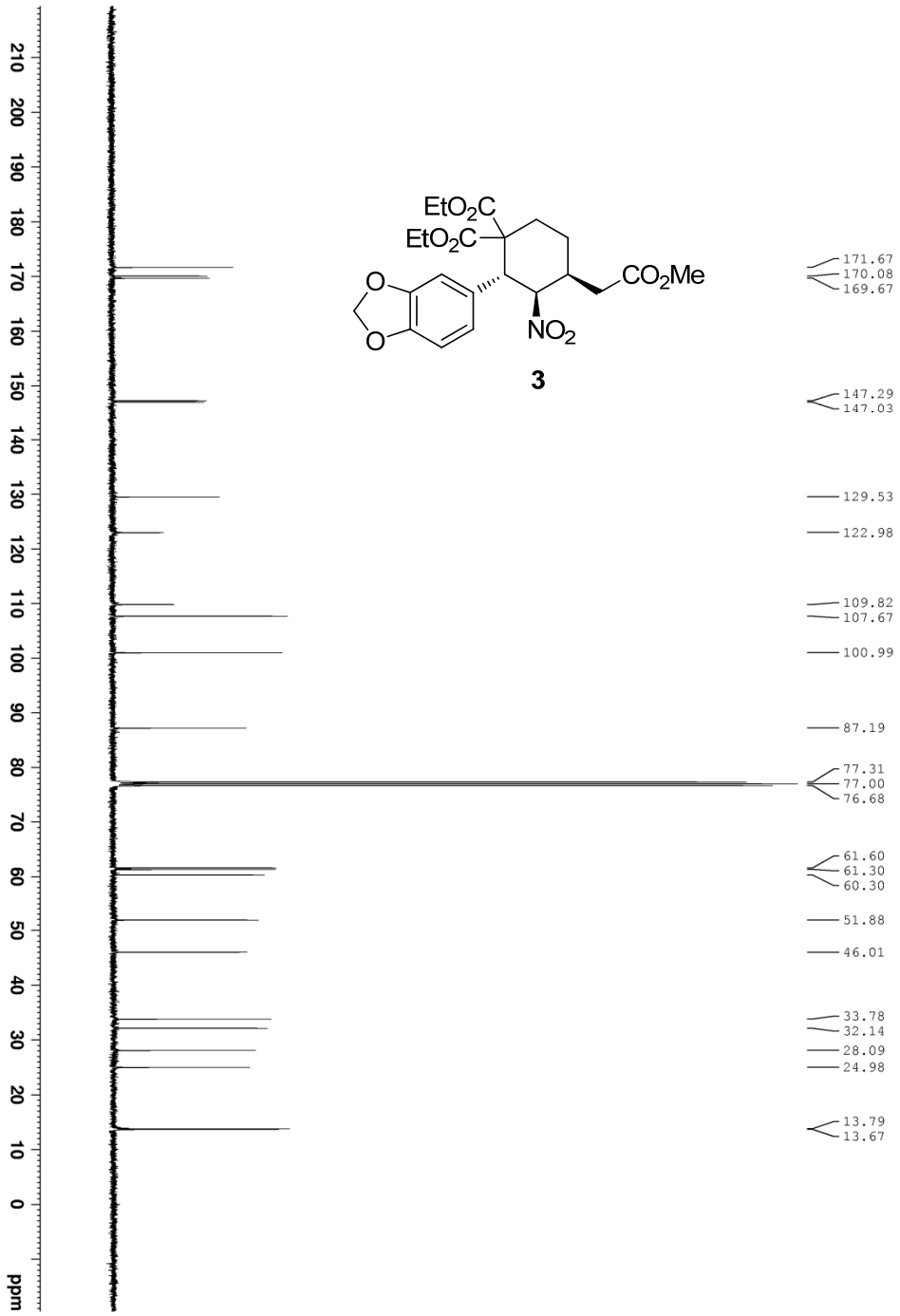


3a (99% ee)

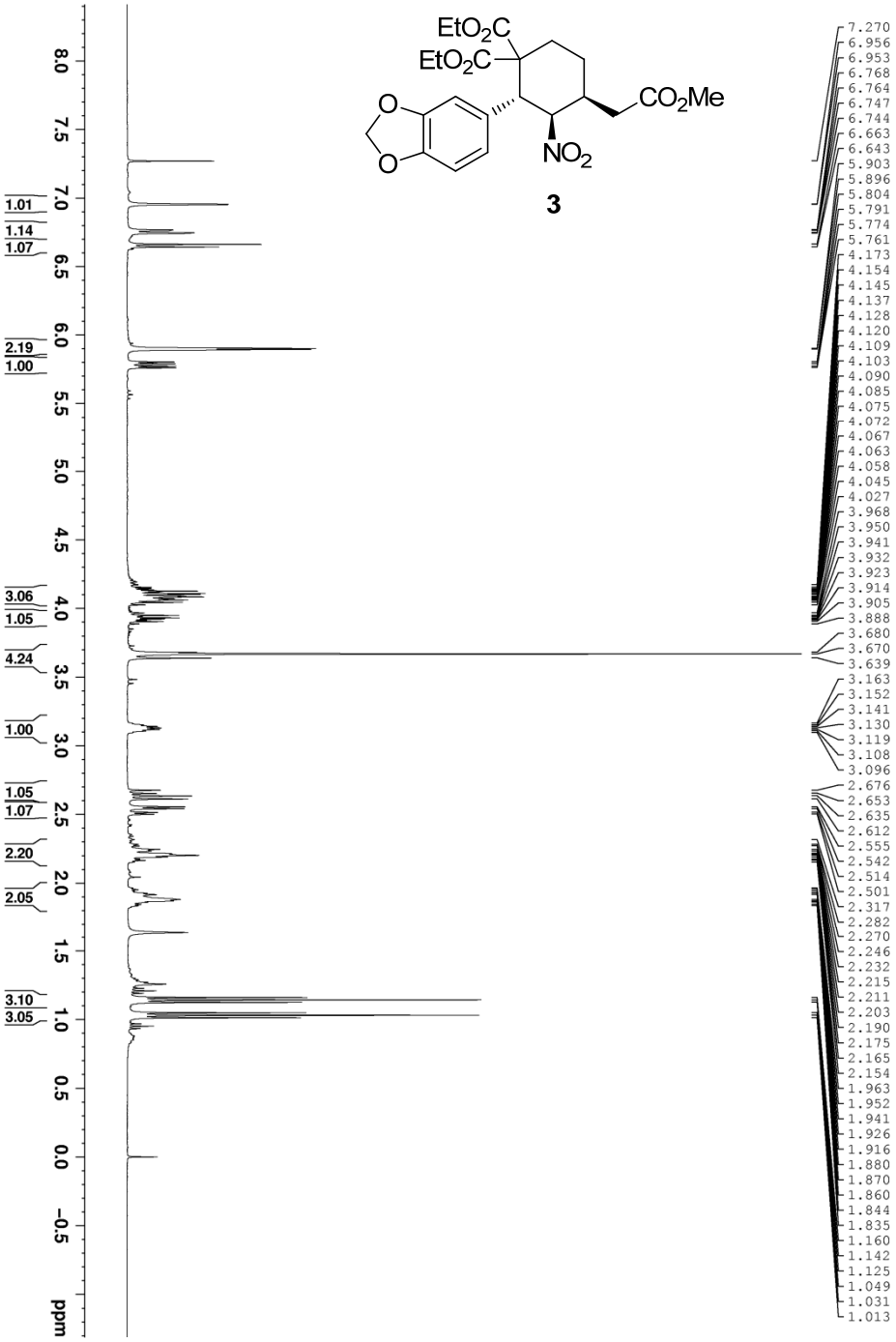
CCDC 724268 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

NMR Spectra

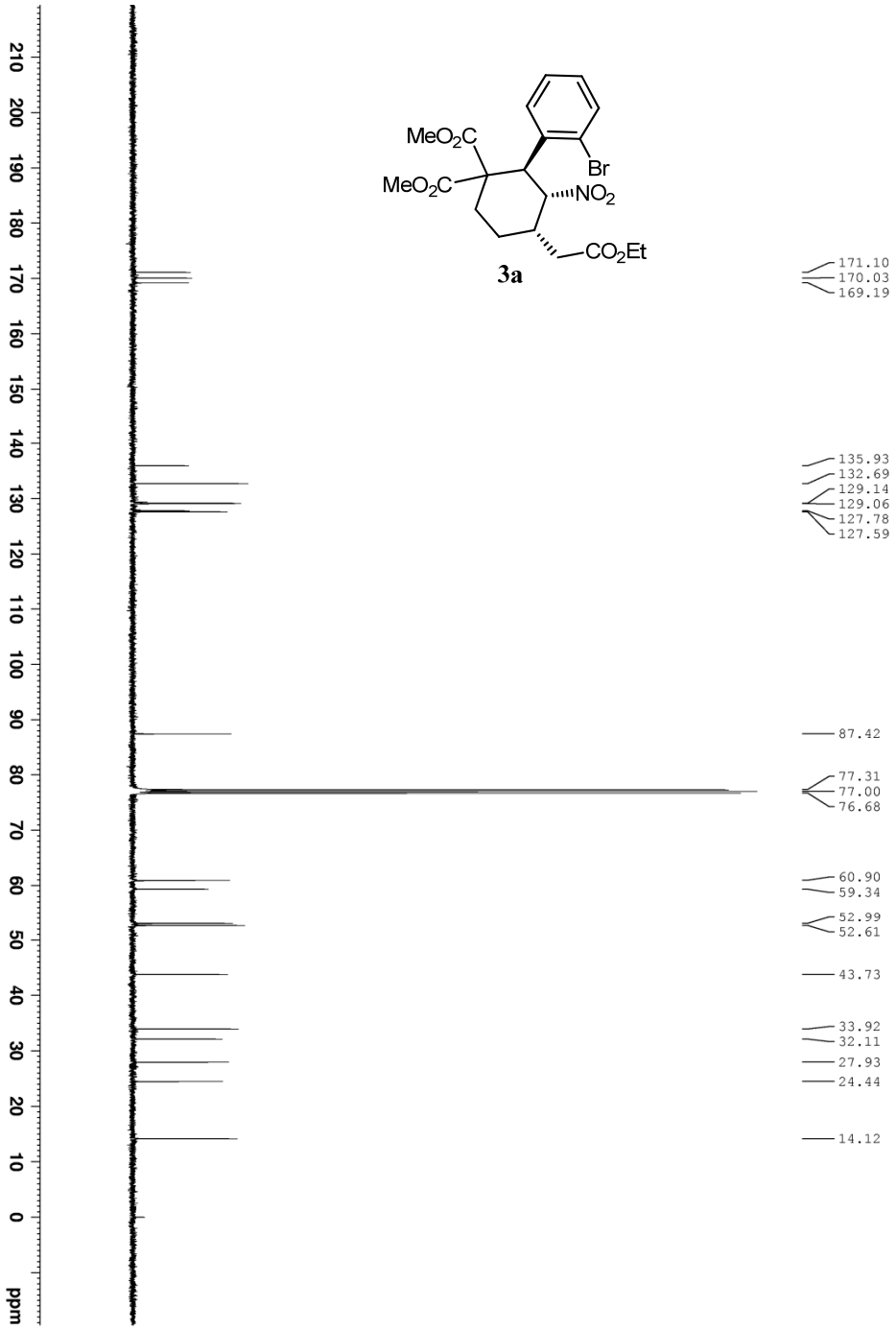
^{13}C NMR spectrum of compound **3** ($CDCl_3$, 100MHz)



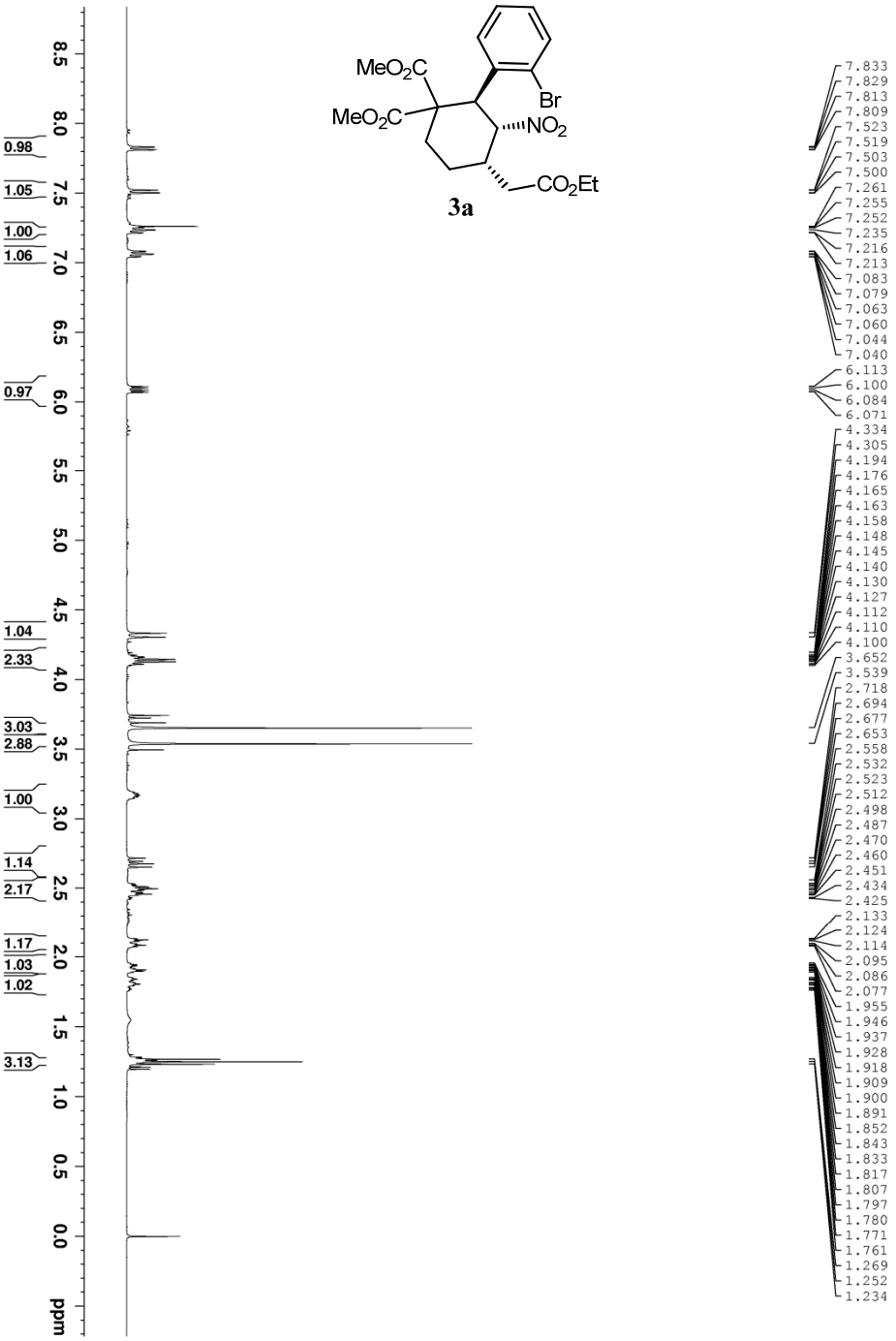
^{1}H NMR spectrum of compound **3** ($CDCl_3$, 400MHz)



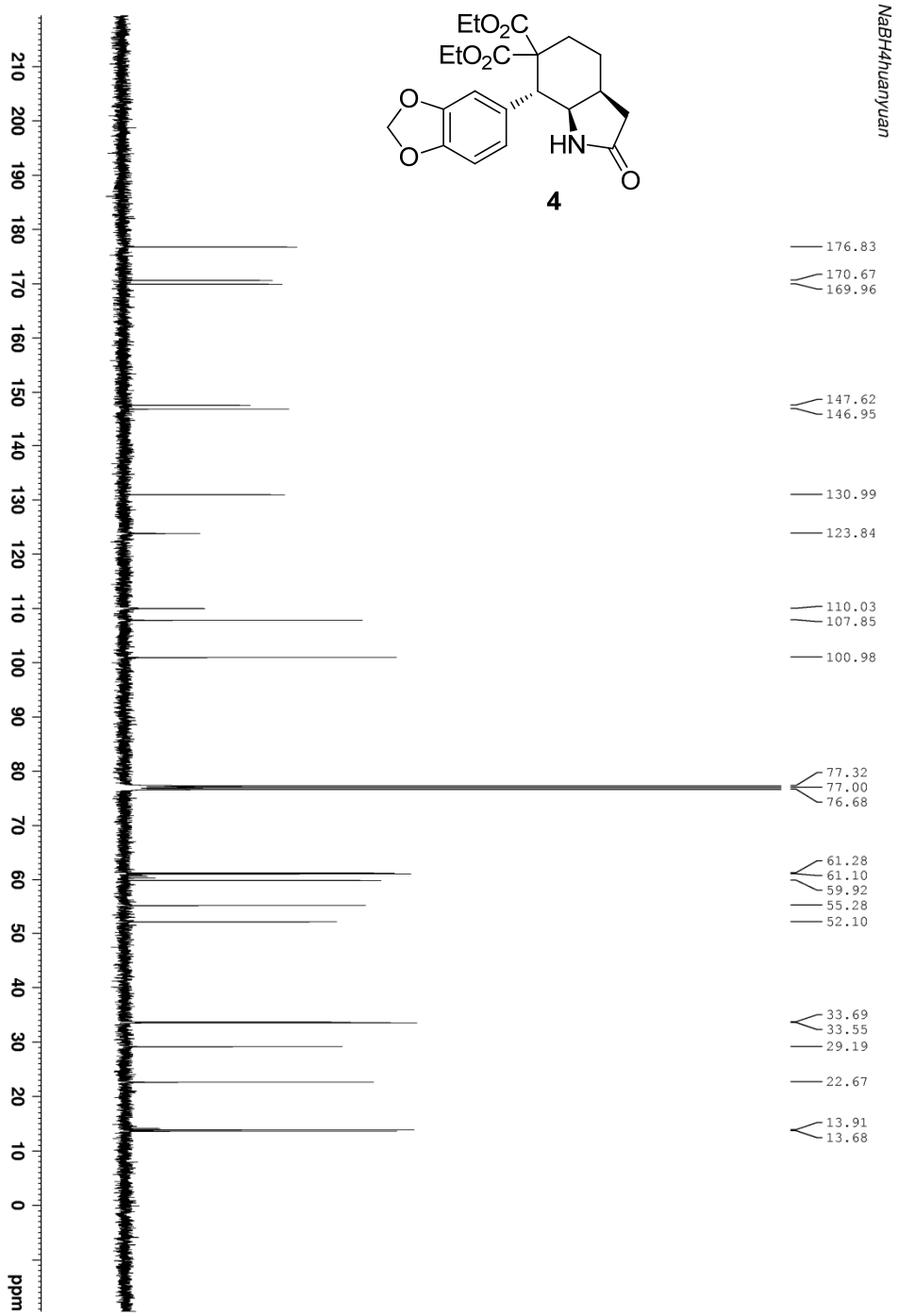
^{13}C NMR spectrum of compound **3a** ($CDCl_3$, 100MHz)



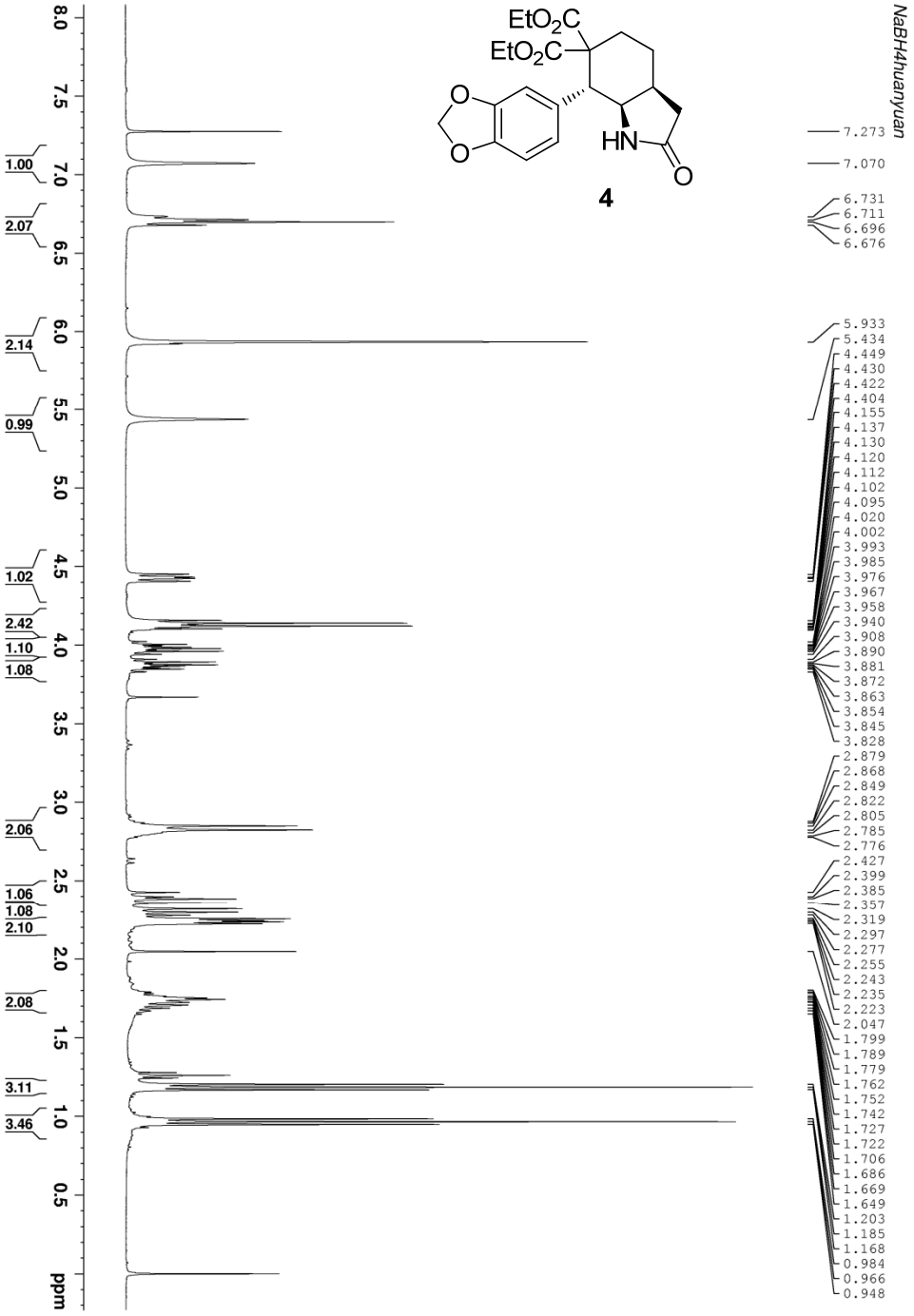
^{1}H NMR spectrum of compound **3a** ($CDCl_3$, 400MHz)



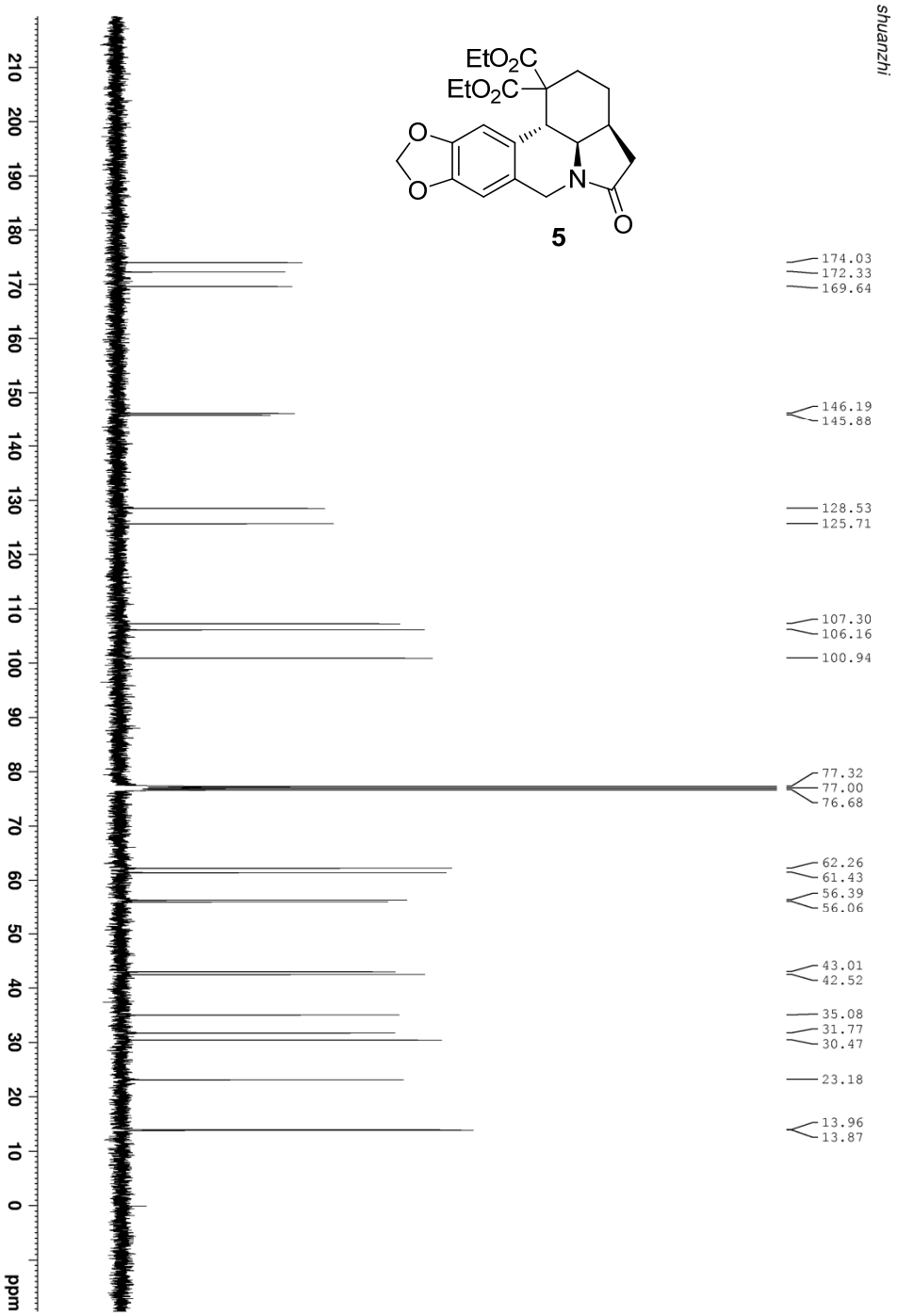
^{13}C NMR spectrum of compound **4** ($CDCl_3$, 400MHz)



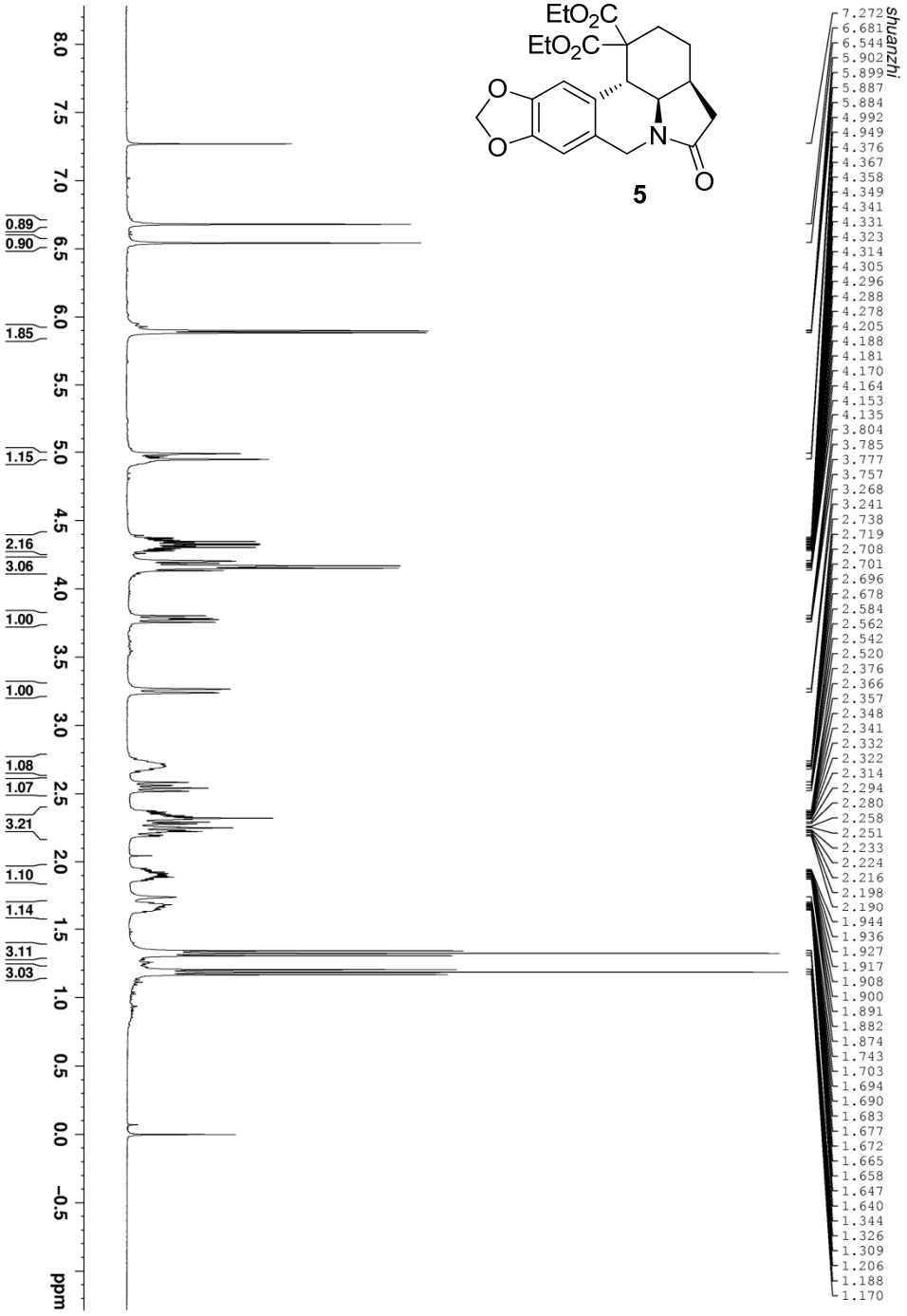
^{1}H NMR spectrum of compound **4** ($CDCl_3$, 400MHz)



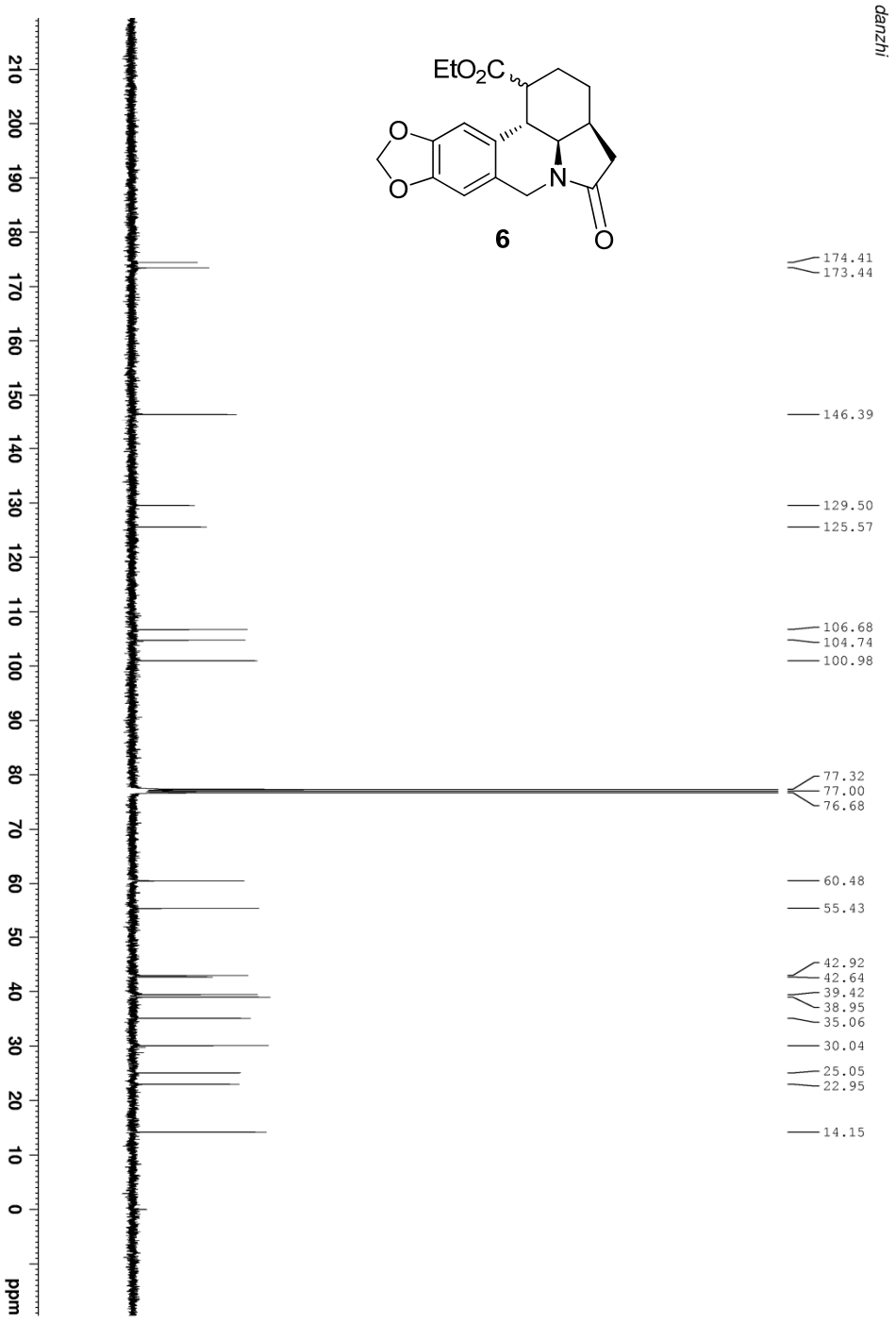
^{13}C NMR spectrum of compound **5** ($CDCl_3$, 400MHz)



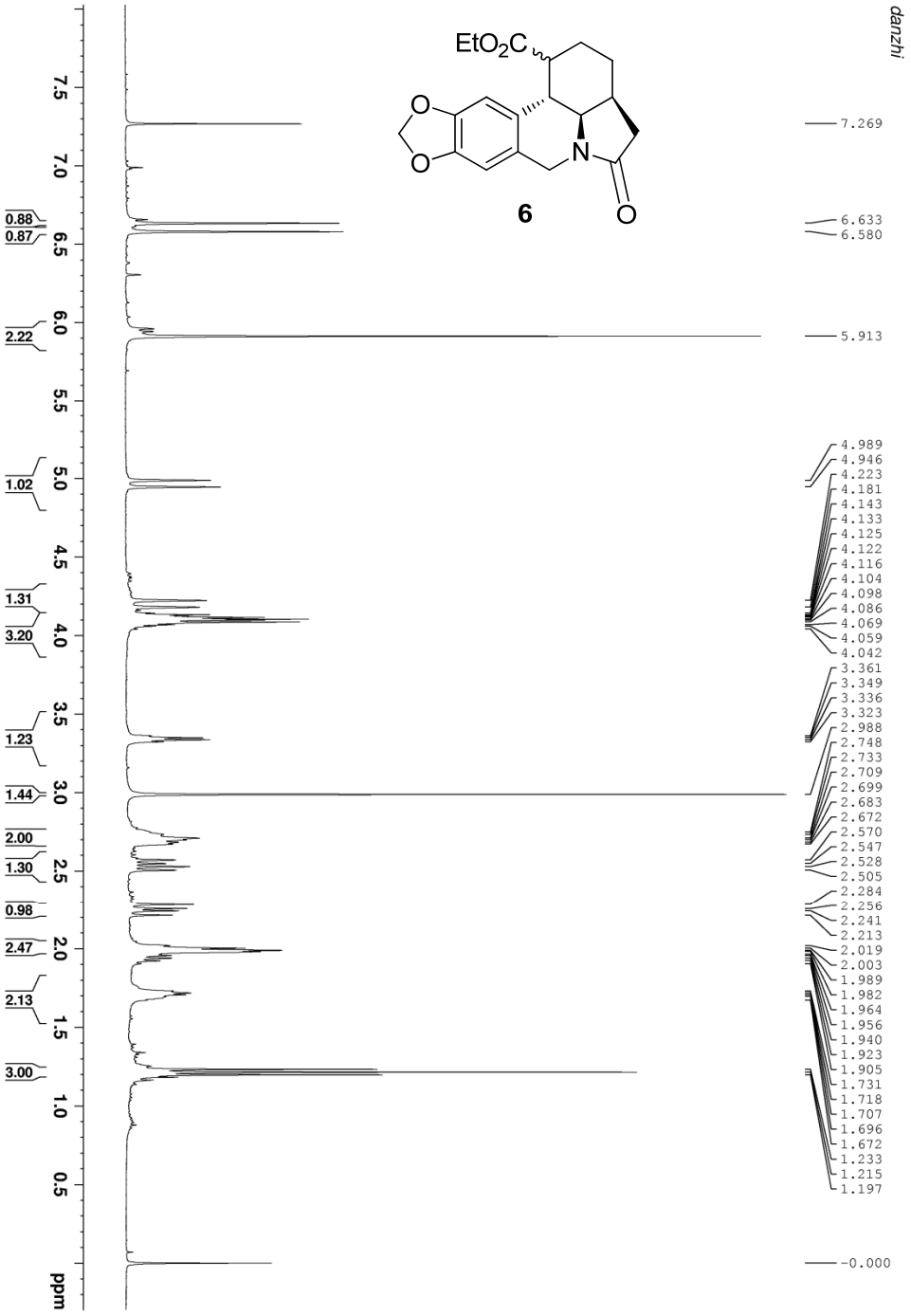
^{1}H NMR spectrum of compound **5** ($CDCl_3$, 400MHz)



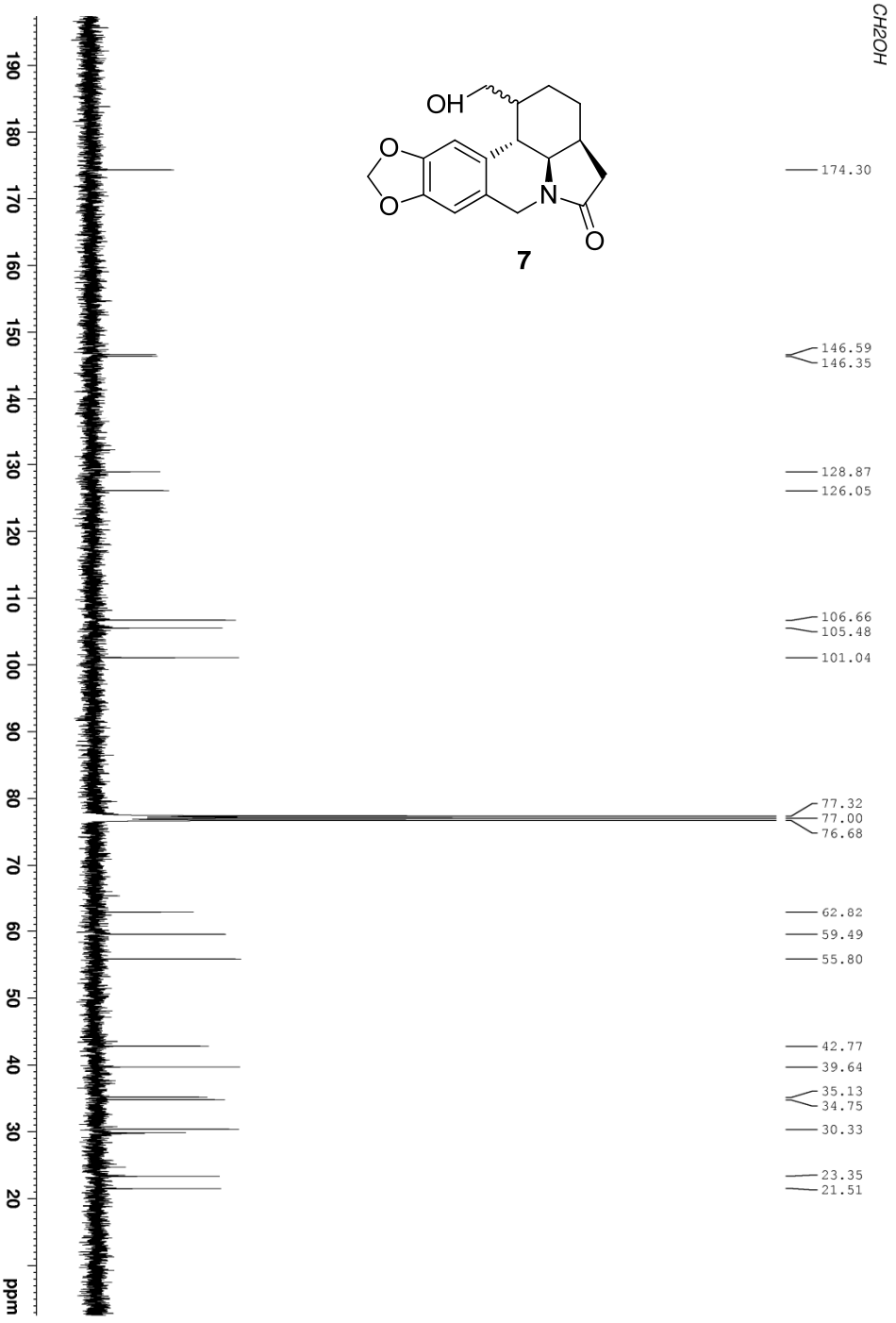
^{13}C NMR spectrum of compound **6** ($CDCl_3$, 400MHz)



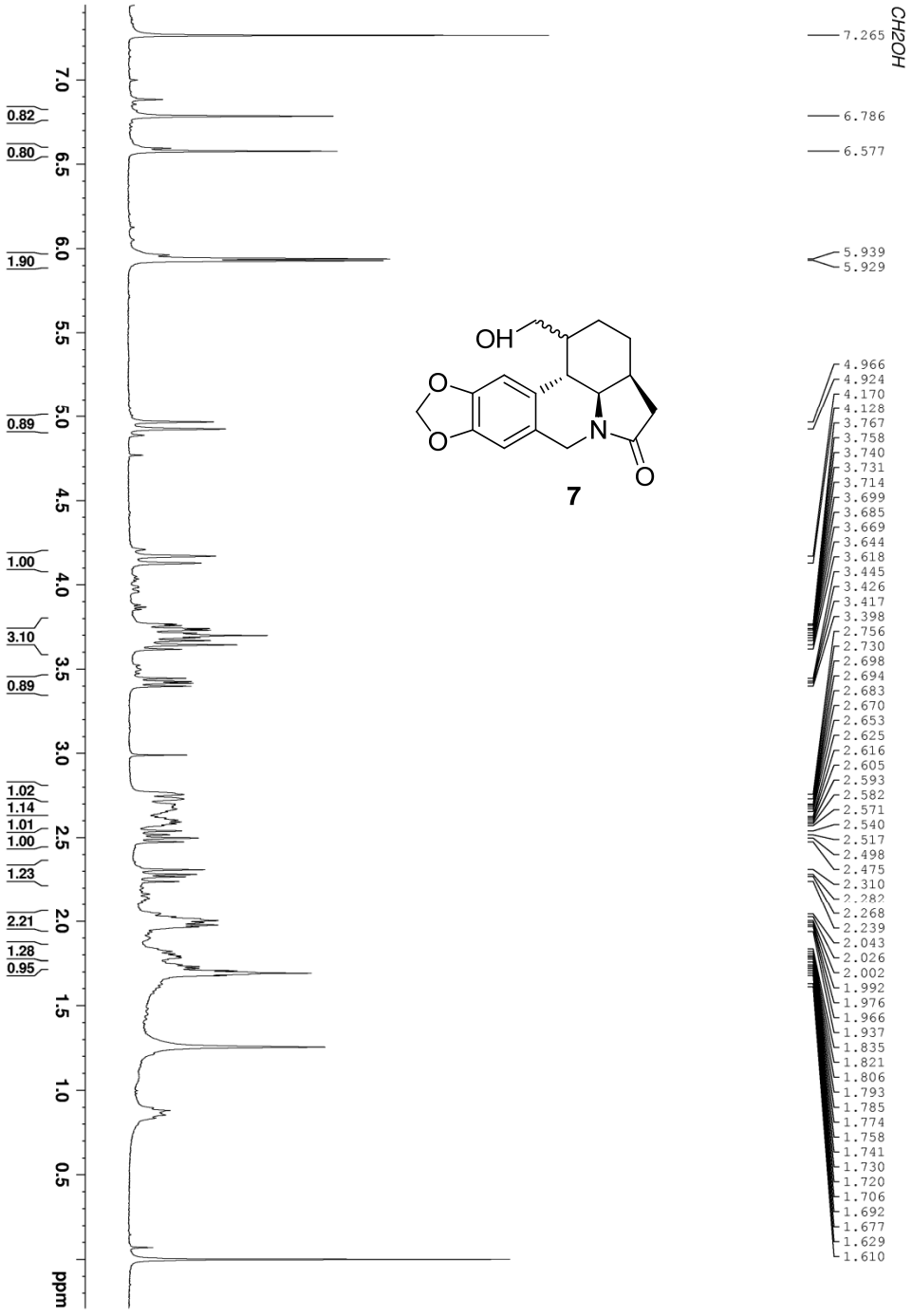
^{1}H NMR spectrum of compound **6** ($CDCl_3$, 400MHz)



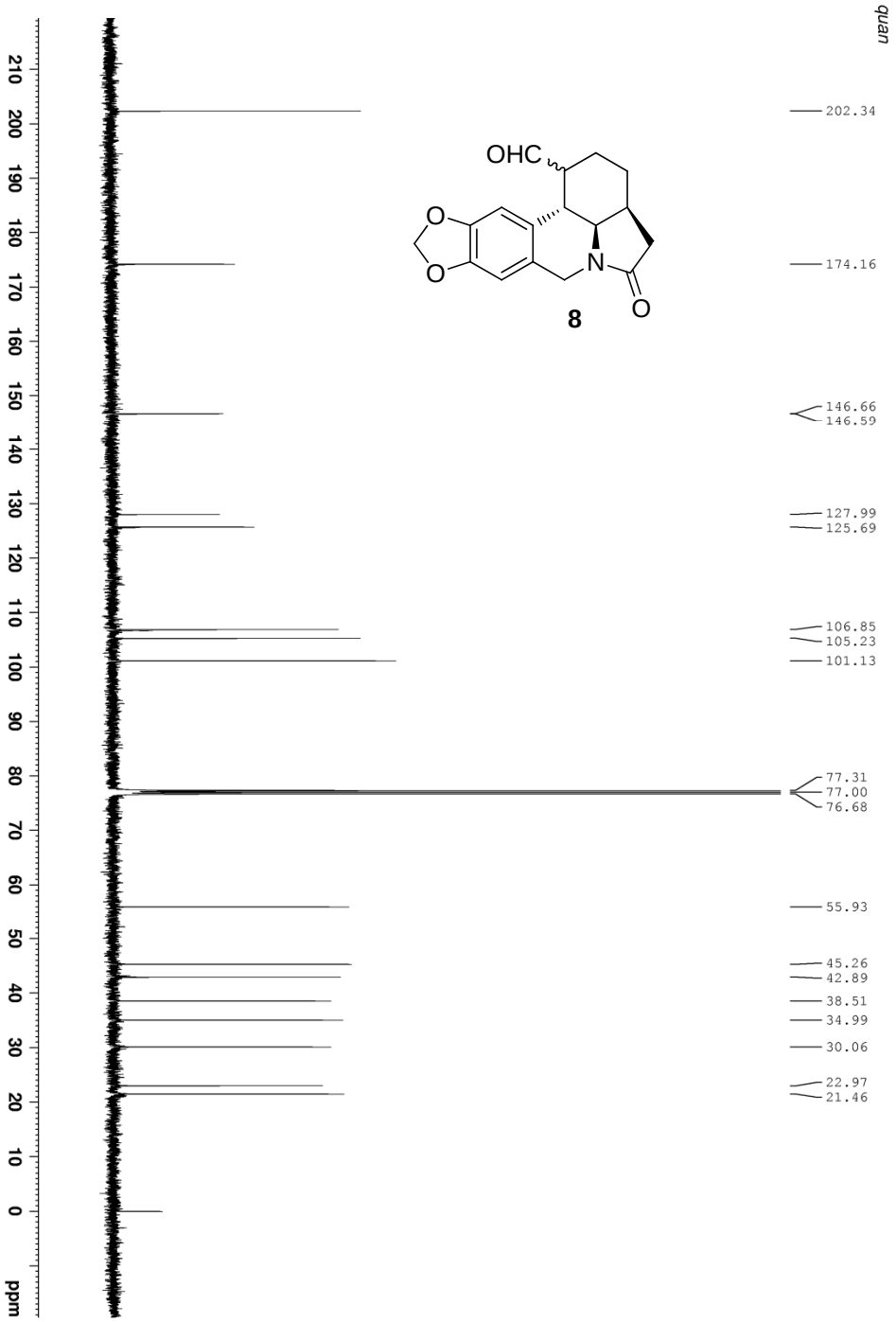
^{13}C NMR spectrum of compound **7** ($CDCl_3$, 400MHz)



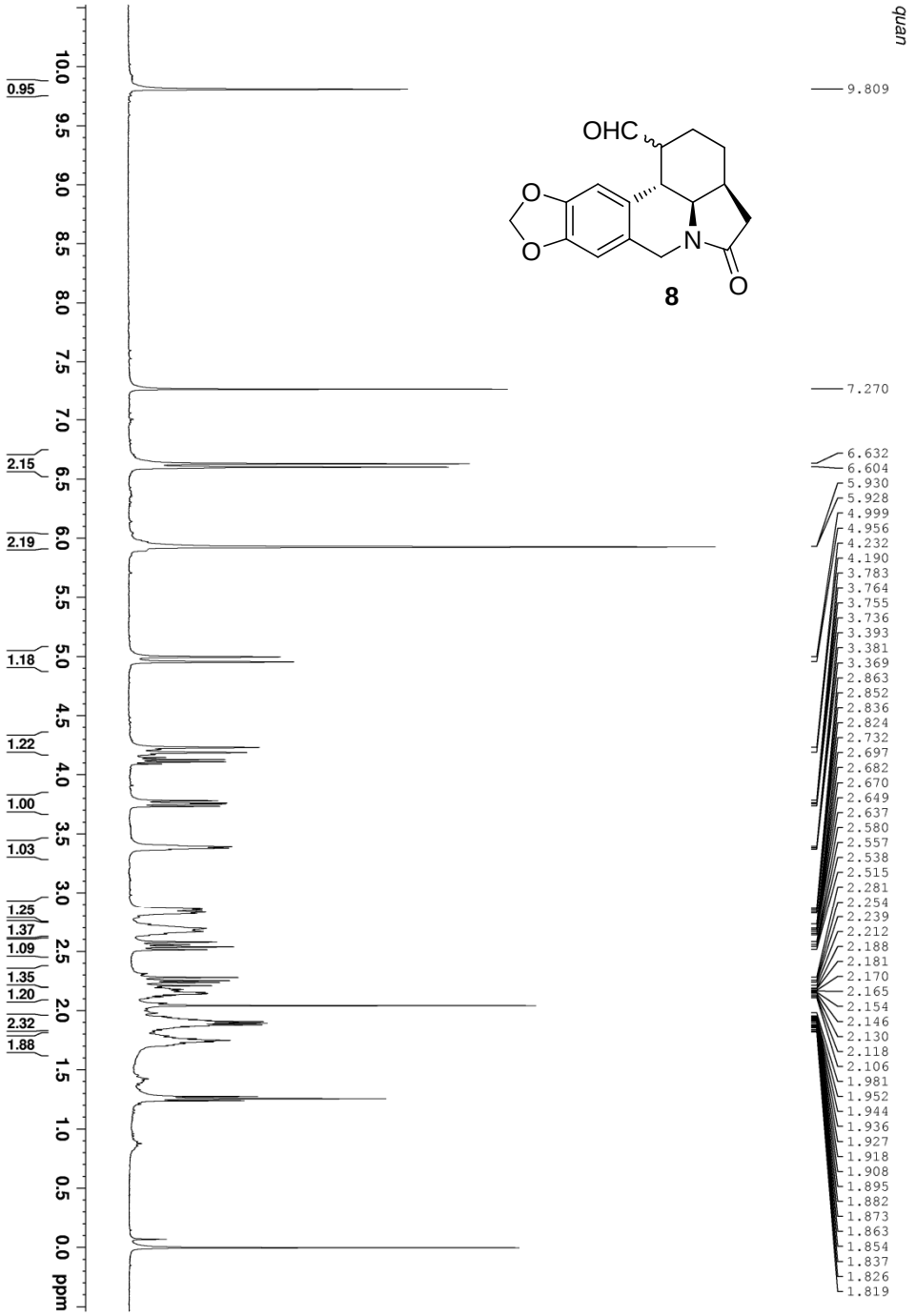
^{1}H NMR spectrum of compound **7** ($CDCl_3$, 400MHz)



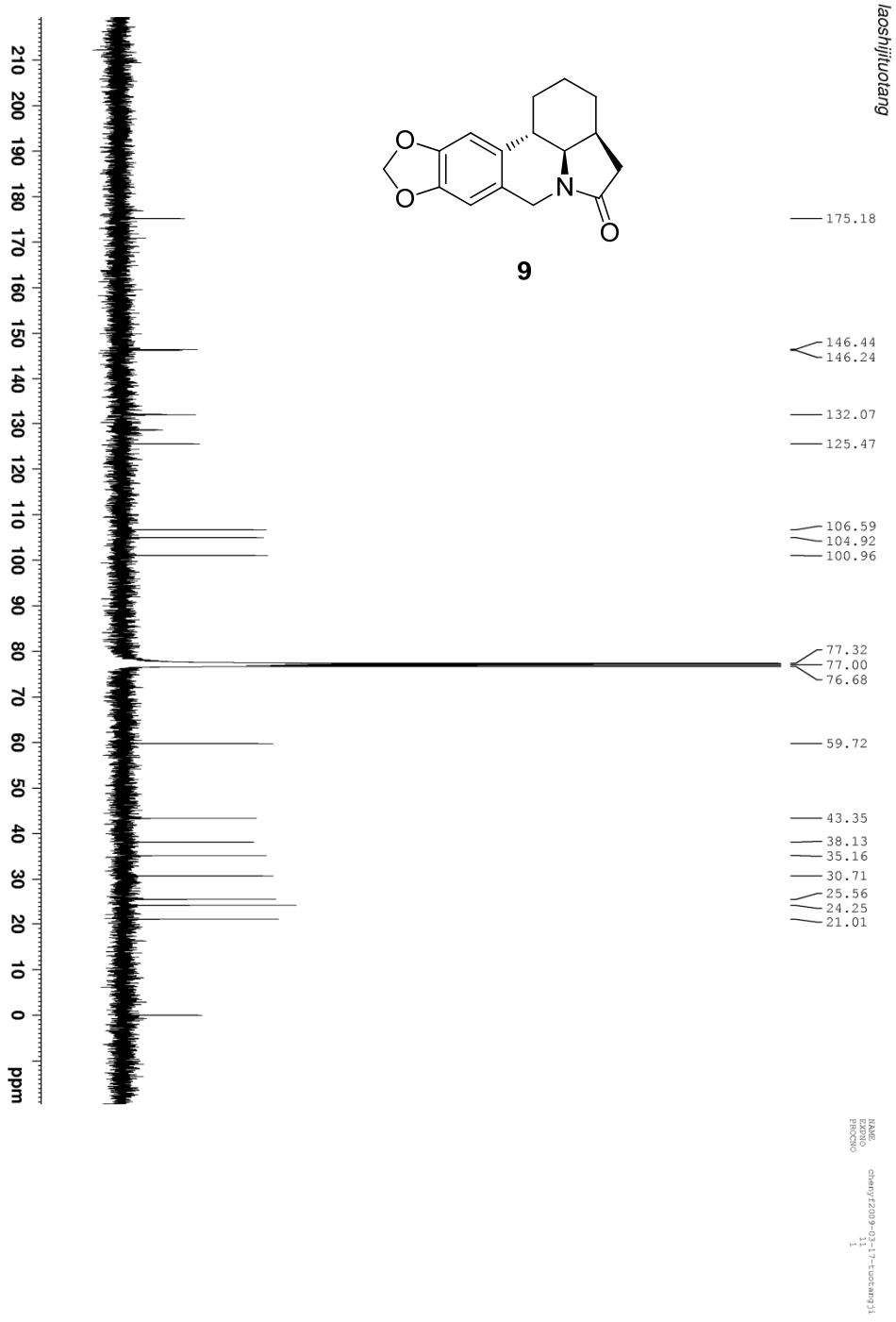
^{13}C NMR spectrum of compound **8** ($CDCl_3$, 400MHz)



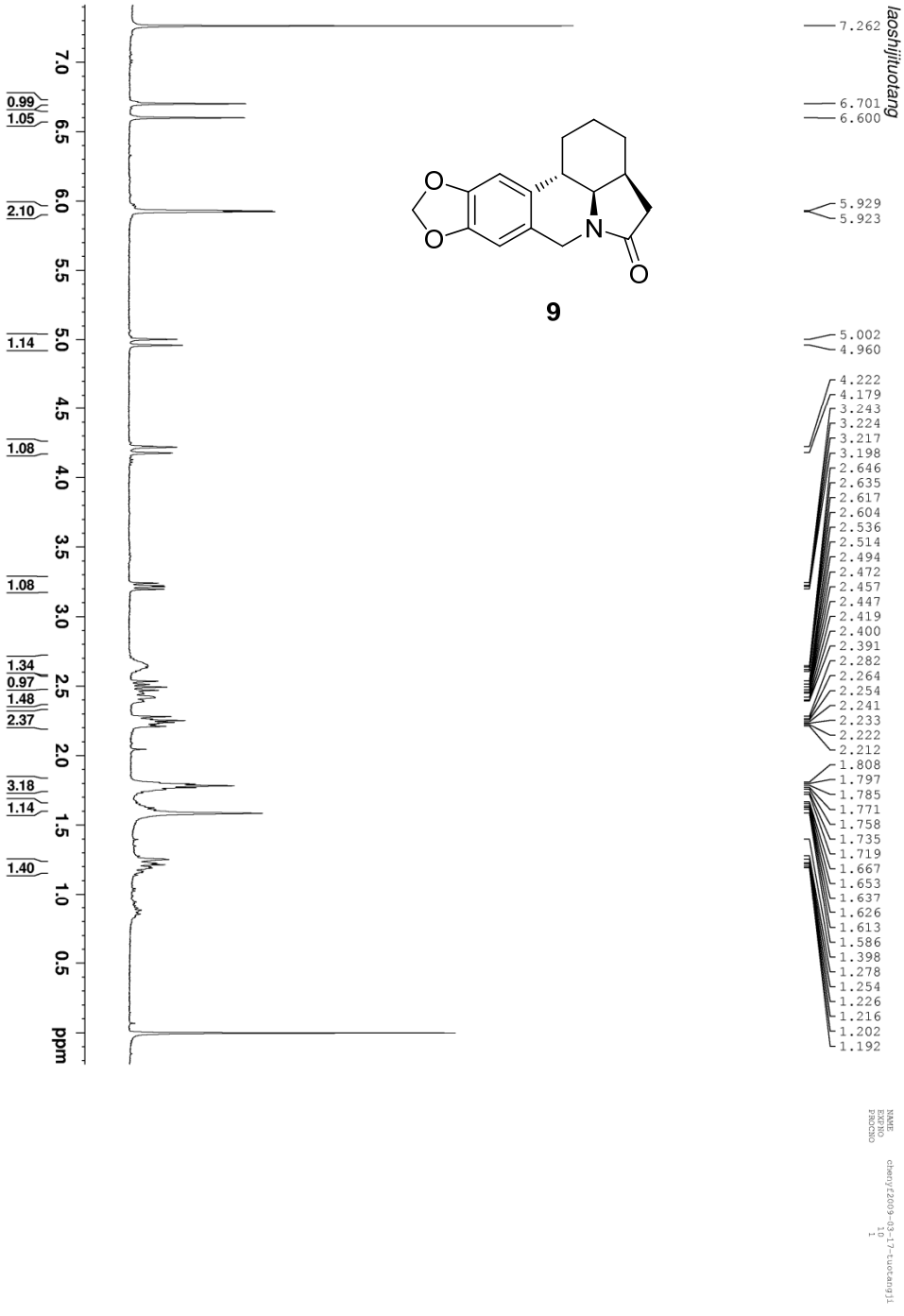
^{1}H NMR spectrum of compound **8** ($CDCl_3$, 400MHz)



^{13}C NMR spectrum of compound **9** ($CDCl_3$, 400MHz)

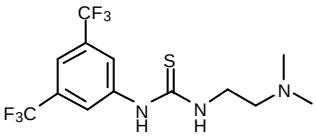


^{1}H NMR spectrum of compound **9** ($CDCl_3$, 400MHz)



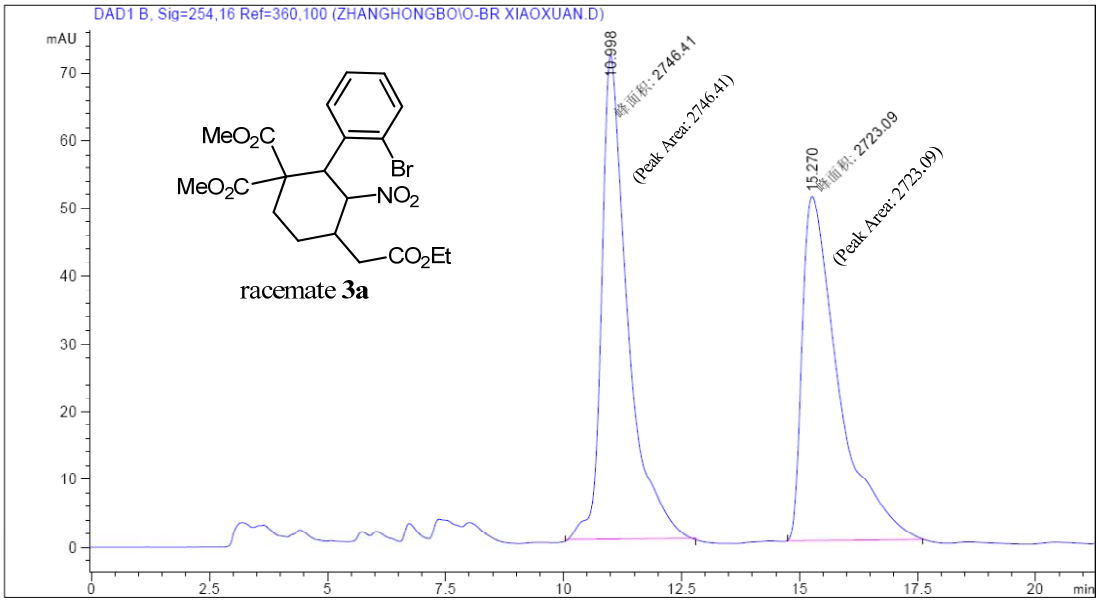
HPLC Spectra

The racemic sample of **3** were prepared by the following genreal method**:** To a mixture of unsaturated ester **1** (0.5 mmol) and nitroolefin **2** (0.2 mmol) in toluene (0.4 mL) was added achiral catalyst **10** (20 mol %). The reaction mixture was then stirred at room temperature until the reaction was complete. Then the diastereomers were purified by column chromatography (eluent: petroleum ether/ethyl acetate = 15:1) to afford racemic sample **3**. Racemic sample **3a** was prepared by the similar method.



10

HPLC spectrum of racemic compound **3a**



信号 1: DAD1 B, Sig=254,16 Ref=360,100

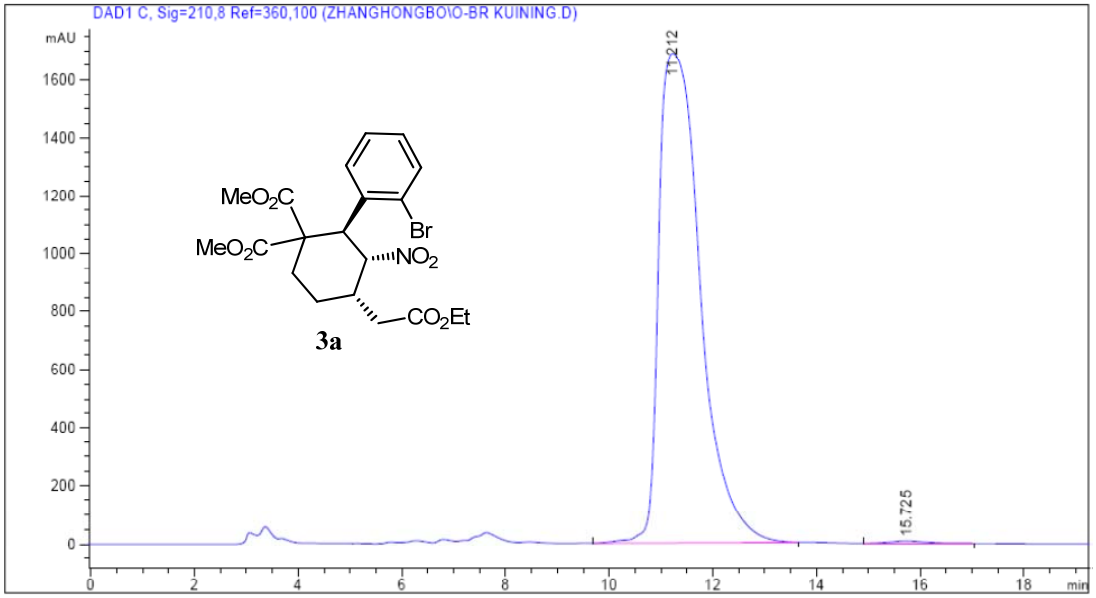
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	10.998	MM	0.6406	2746.41138	71.45315	50.2132
2	15.270	MM	0.8941	2723.08887	50.76307	49.7868

Replacement of Chinese characters in the HPLC spectrum (above) with corresponding English:

Signal 1: DAD1 B, Sig=254, 16 Ref=360,100

Peak #	Retention Time [min]	Type	Peak Width [min]	Peak Area [mAU*s]	Peak Height [mAU]	Peak Area %
1	10.998	MM	0.6406	2746.41138	71.45315	50.2132
2	15.270	MM	0.8941	2723.08887	50.76307	49.7868

HPLC spectrum of chiral compound **3a**



信号 1: DAD1 C, Sig=210,8 Ref=360,100

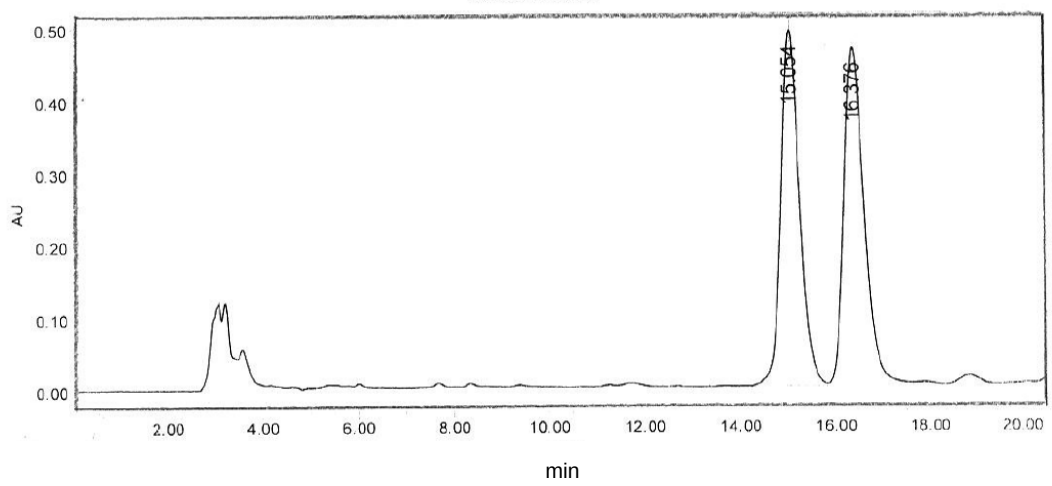
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	11.212	VB	0.6735	9.37856e4	1684.70276	99.5817
2	15.725	BB	0.5704	393.98724	8.17241	0.4183

Replacement of Chinese characters in the HPLC spectrum (above) with corresponding English:

Signal 1: DAD1 C, Sig=210, 8 Ref=360,100

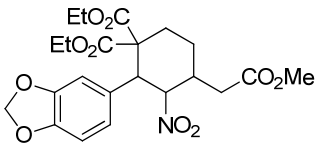
Peak #	Retention Time [min]	Type	Peak Width [min]	Peak Area [mAU*s]	Peak Height [mAU]	Peak Area %
1	11.212	VB	0.6735	9.37856e4	1684.70276	99.5817
2	15.725	BB	0.5704	393.98724	8.17241	0.4183

HPLC spectrum of racemic compound **3**



处理通道: **PDA 206.1** 纳米

	处理通道	保留时间 (min)	面积	% 面积	高度
1	PDA 206.1 纳米	15.054	13296842	49.01	489829
2	PDA 206.1 纳米	16.376	13832993	50.99	466310



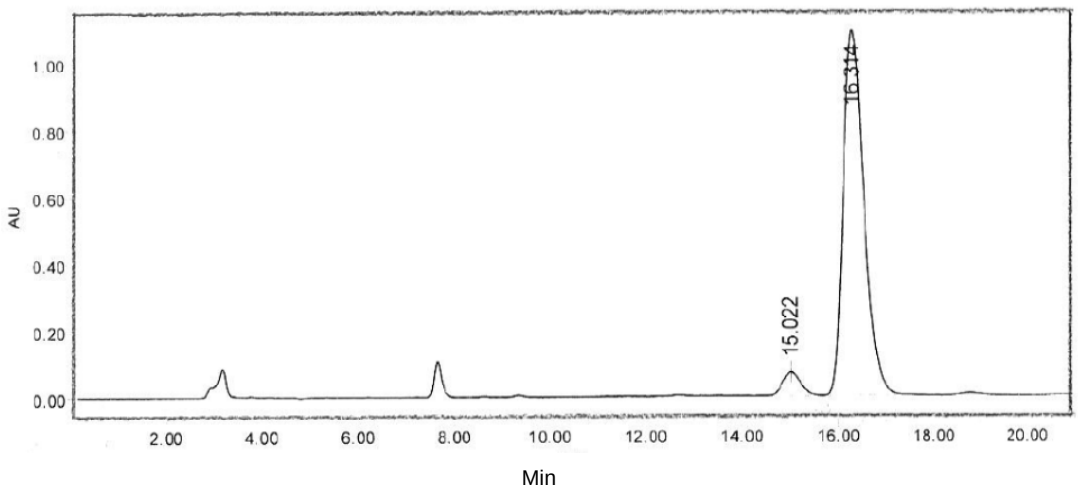
racemate **3**

Replacement of Chinese characters in the HPLC spectrum (above) with corresponding English:

Processing Channel: PDA 206.1 nm

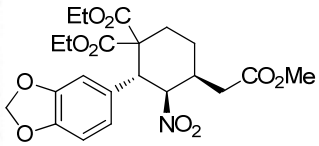
	Processing Channel	Retention Time (min)	Area	% Area	Height
1	PDA 206.1 nm	15.054	13296842	49.01	489829
2	PDA 206.1 nm	16.376	13832993	50.99	466310

HPLC spectrum of chiral compound **3**



处理通道: **PDA 206.1** 纳米

	处理通道	保留时间 (min)	面积	% 面积	高度
1	PDA 206.1 纳米	15.022	1816298	5.24	71122
2	PDA 206.1 纳米	16.314	32870813	94.76	1097015



3

Replacement of Chinese characters in the HPLC spectrum (above) with corresponding English:

Processing Channel: PDA 206.1 nm

	Processing Channel	Retention Time (min)	Area	% Area	Height
1	PDA 206.1 nm	15.022	1816298	5.24	71122
2	PDA 206.1 nm	16.314	32870813	94.76	1097015