

Rapid construction of imidazo[4,5-*b*]pyridine skeleton from 2-chloro-3-nitropyridine via tandem reaction in H₂O-IPA medium

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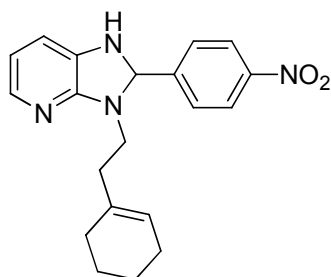
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X

3-(2-(Cyclohex-1-en-1-yl)ethyl)-2-(4-nitrophenyl)-2,3-dihydro-1H-imidazo[4,5-b]pyridine (X).

^1H NMR (300 MHz, CDCl_3) δ 8.62 (s, 1H), 8.32 (d, $J = 8.6$ Hz, 2H), 8.09 (dd, $J = 4.9, 1.5$ Hz, 1H), 8.04 (d, $J = 8.6$ Hz, 2H), 7.25 (d, $J = 1.5$ Hz, 1H), 6.55 (dd, $J = 7.7, 4.9$ Hz, 1H), 5.67 (s, 1H), 5.60 (s, 1H), 3.55 (t, $J = 6.5$ Hz, 2H), 2.32 (t, $J = 6.5$ Hz, 2H), 2.03-2.01 (m, 4H), 1.66-1.53 (m, 4H); ^{13}C NMR (75MHz, CDCl_3) δ 155.3, 155.1, 149.6, 148.4, 141.9, 135.7, 130.9, 129.5, 124.5, 124.1, 122.5, 112.2, 38.9, 38.2, 28.1, 25.8, 23.3, 22.8; IR (KBr, cm^{-1}): 3415, 2929, 2830, 1539, 1513, 1336 cm^{-1} ; MS (ESI) m/z : 351 (MH^+);

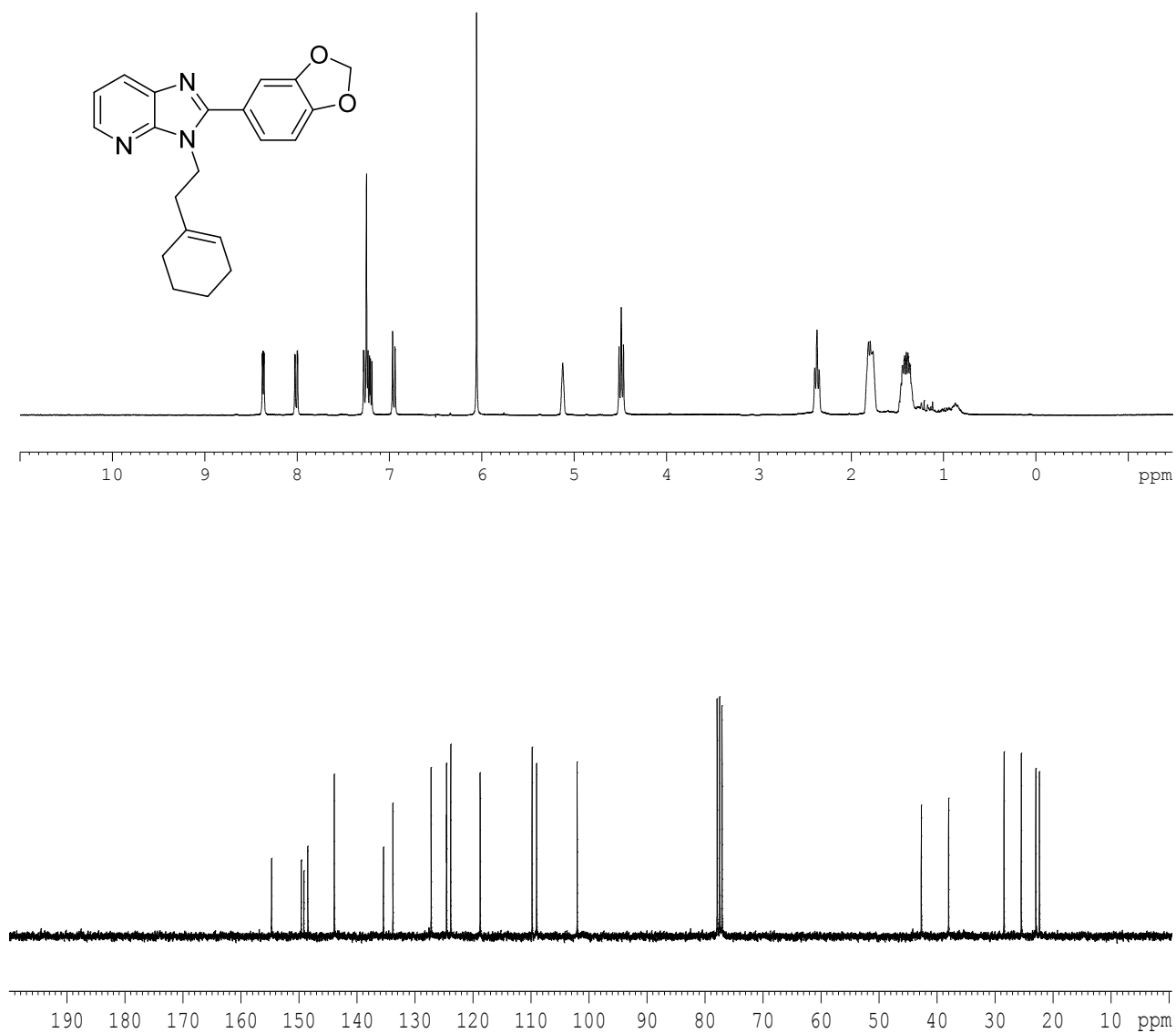


Figure S1: ^1H and ^{13}C NMR spectrum of compound 4a in CDCl_3

6eihr-72-c1 #5 RT: 0,21 AV: 1 NL: 4,18E6
T: + c EI Full ms [227,50-370,50]

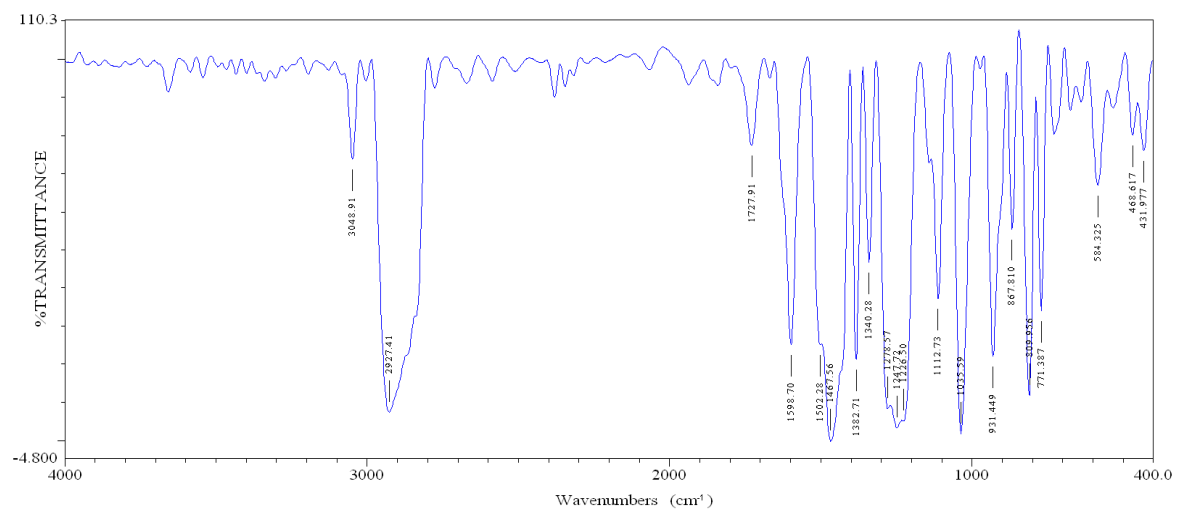
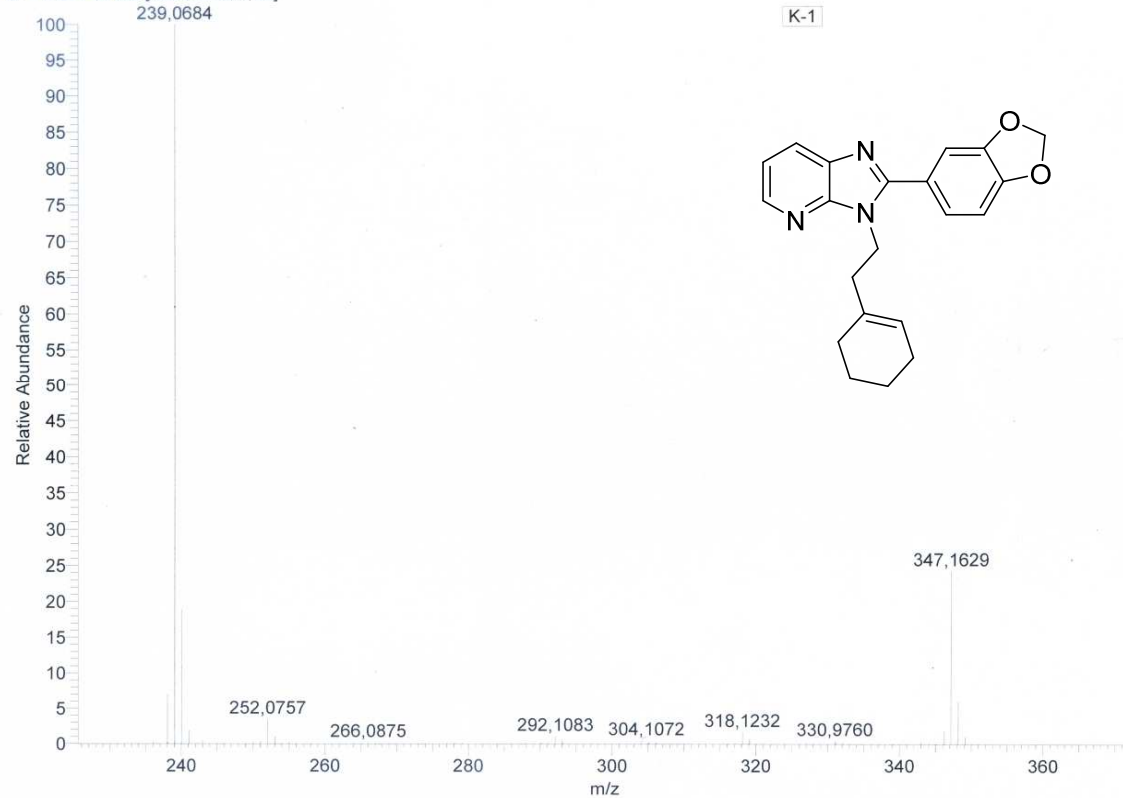


Figure S2: HRMS and IR spectra of compound 4a

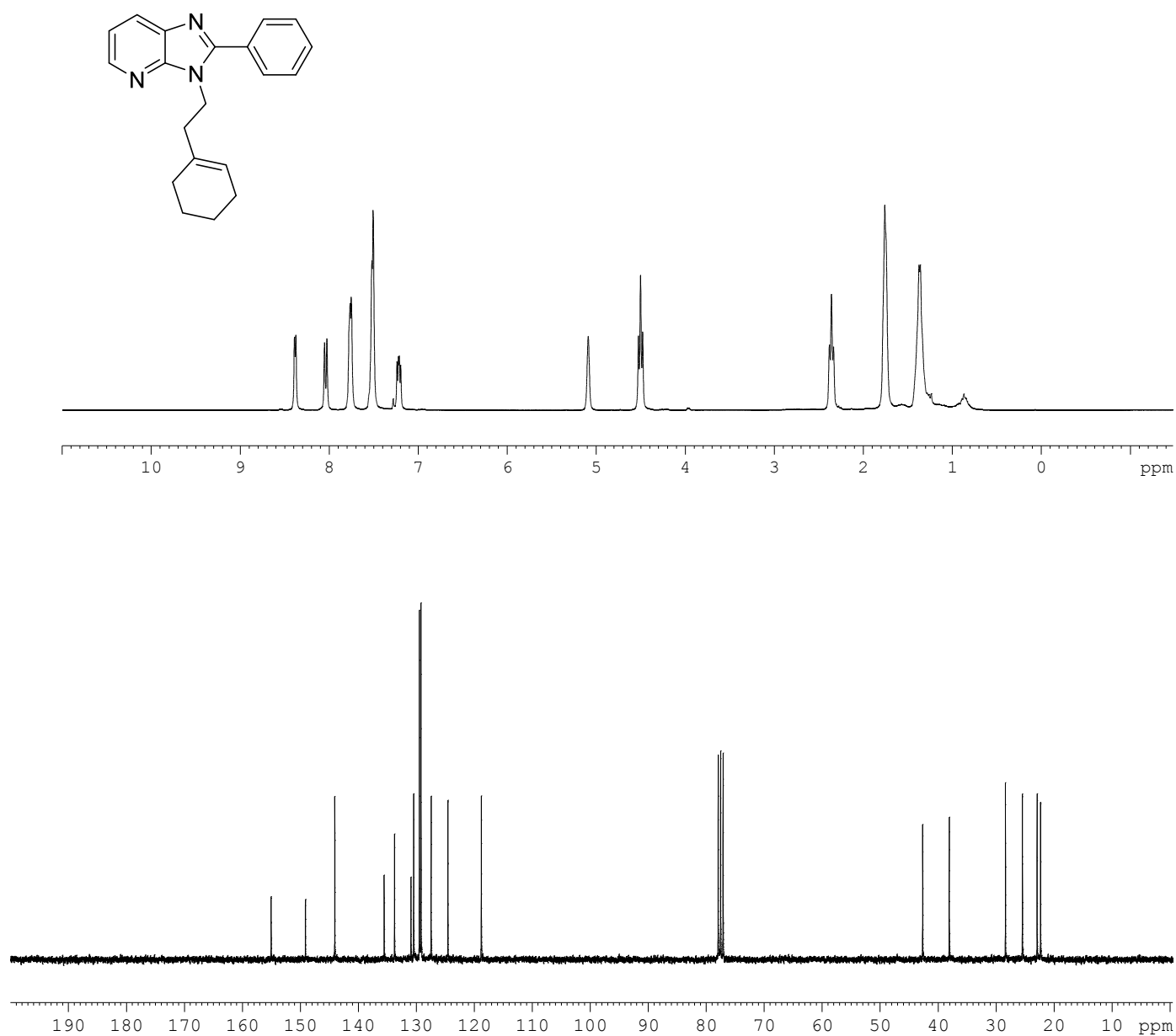


Figure S3: ^1H and ^{13}C NMR spectrum of compound 4b in CDCl_3

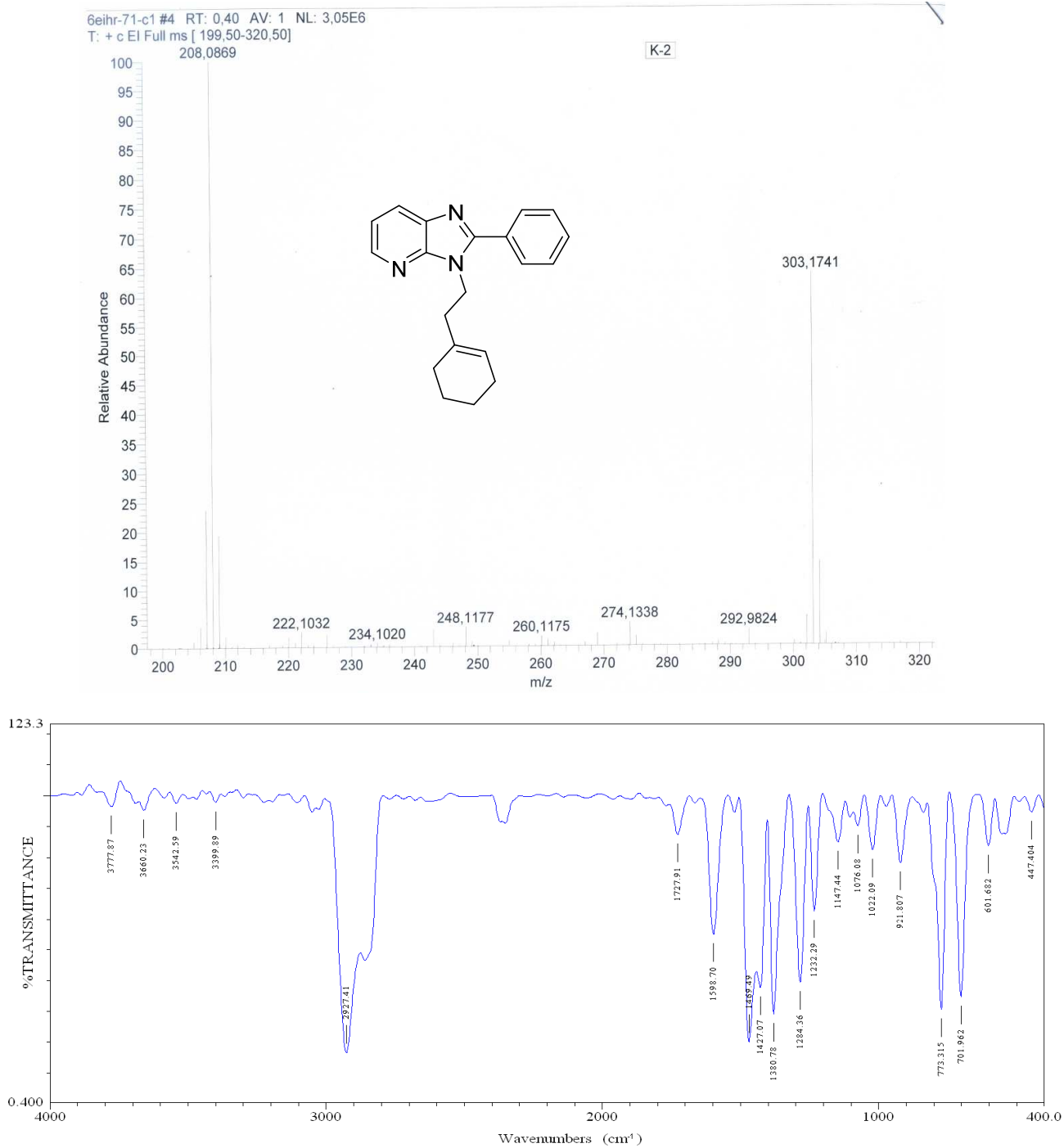


Figure S4: HRMS and IR spectra of compound 4b

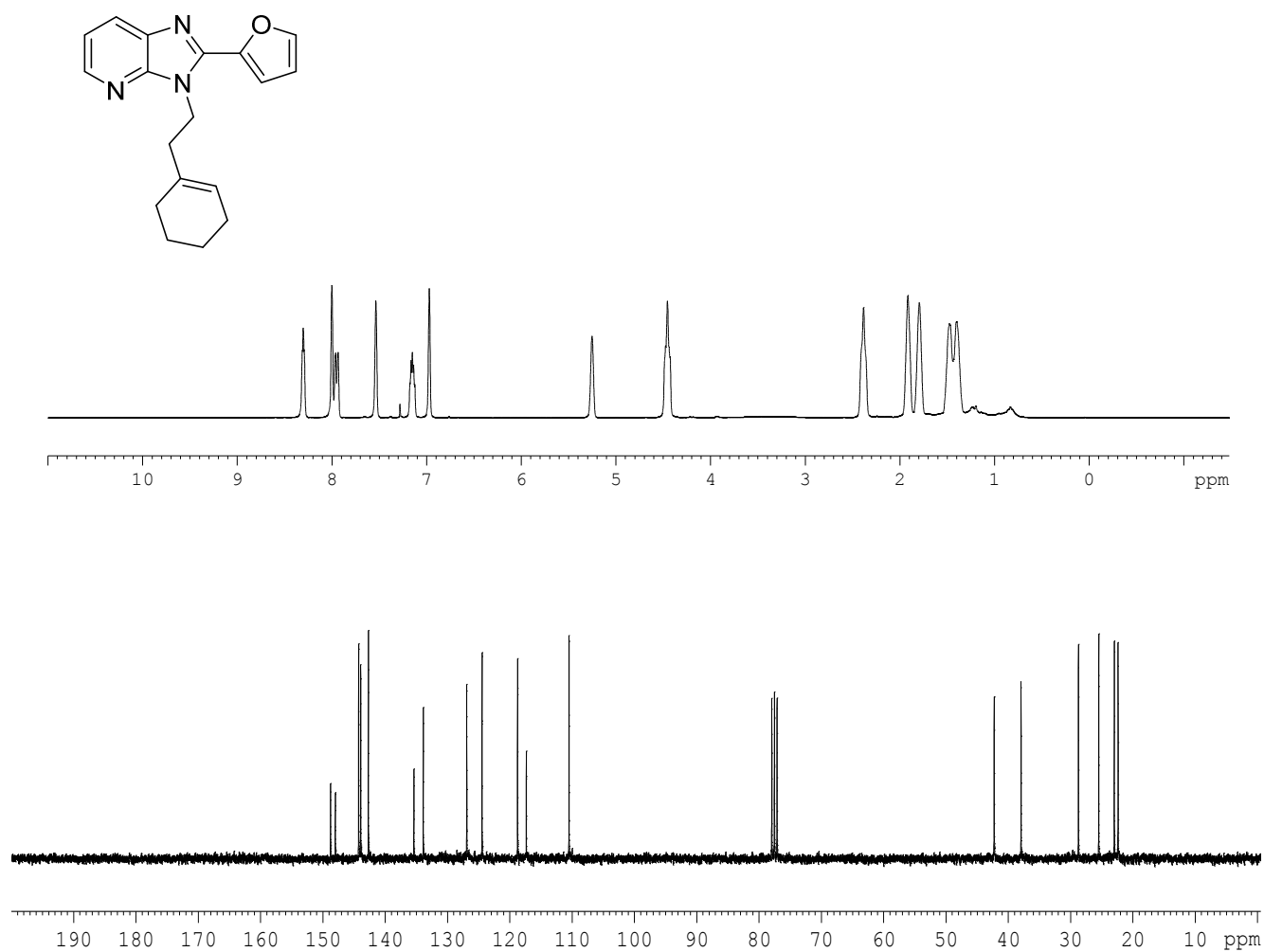


Figure S5: ^1H and ^{13}C NMR spectrum of compound 4c in CDCl_3

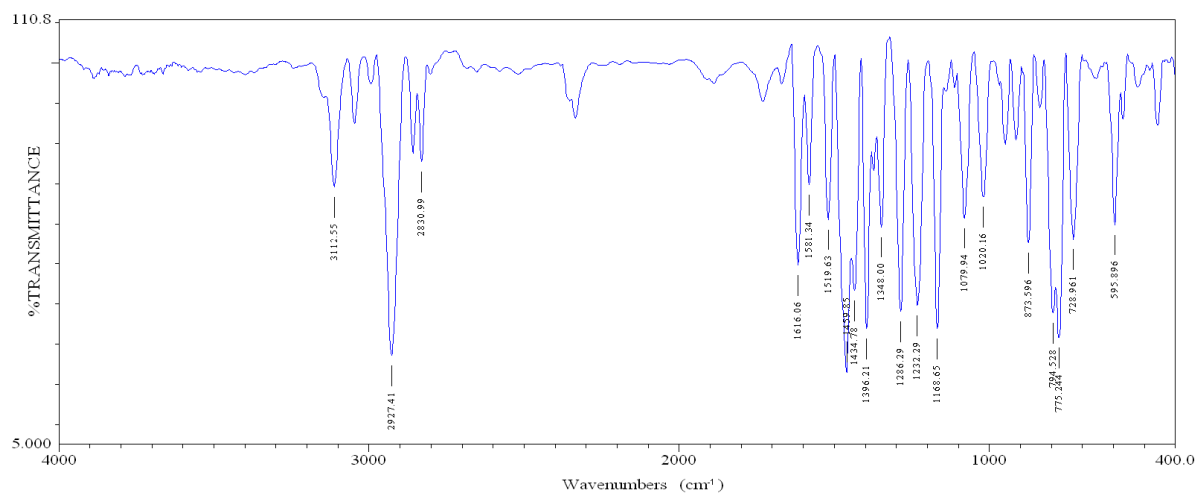
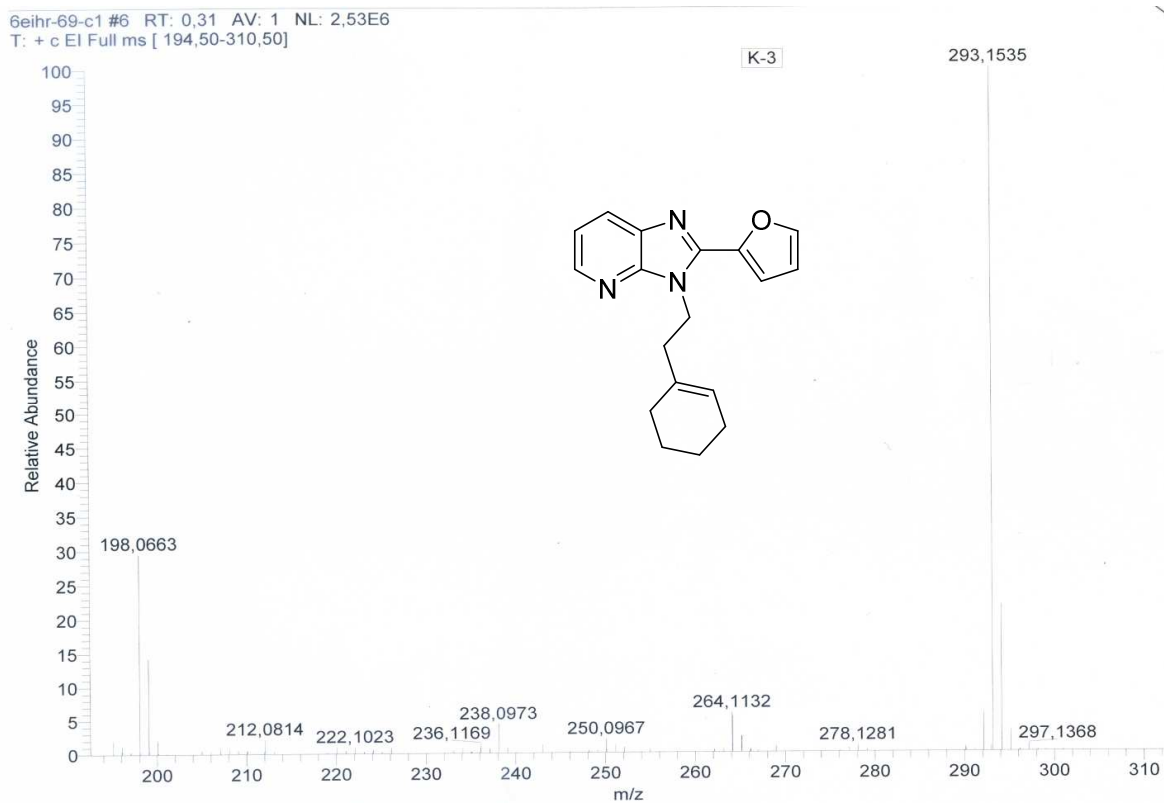


Figure S6: HR Mass and IR spectra of compound 4c

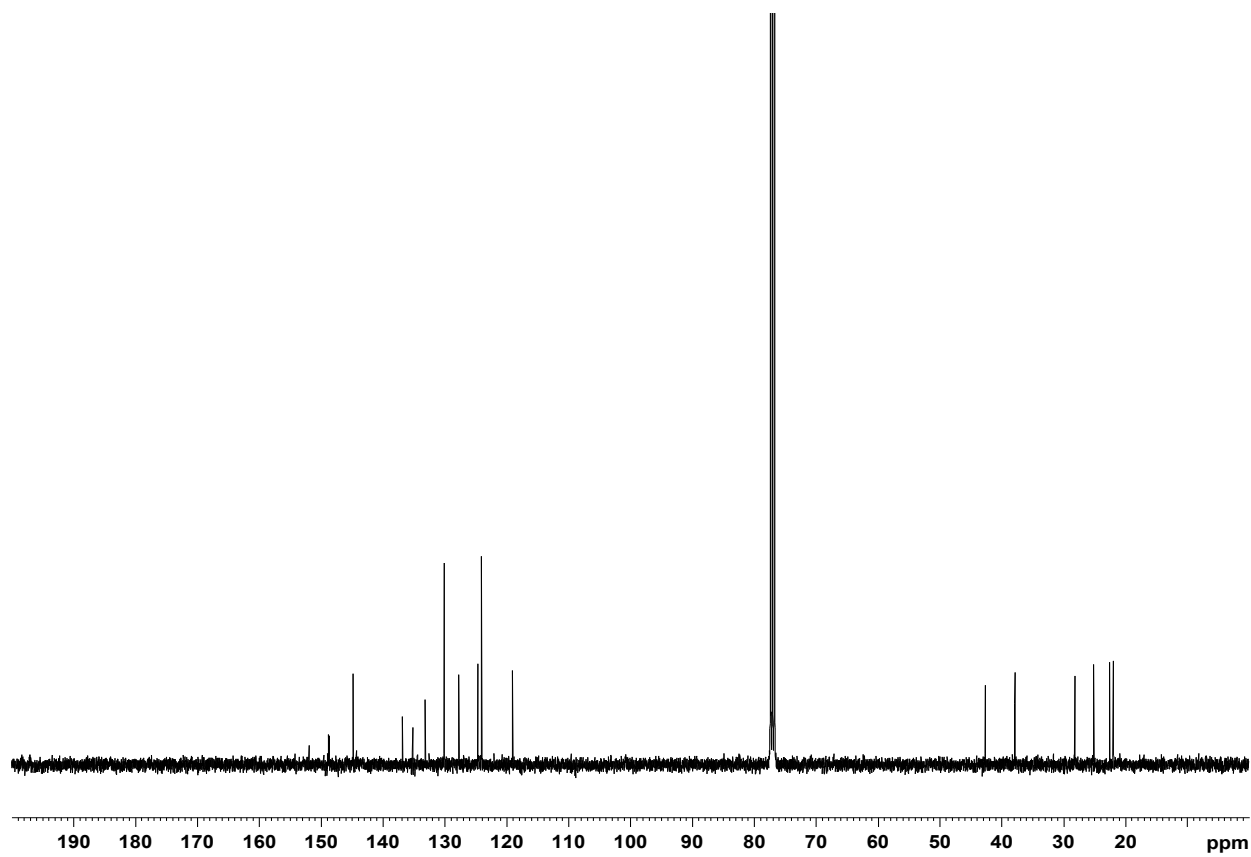
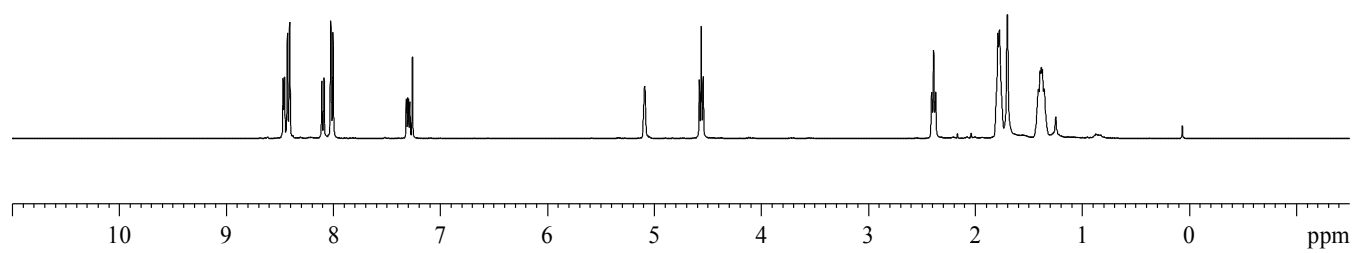
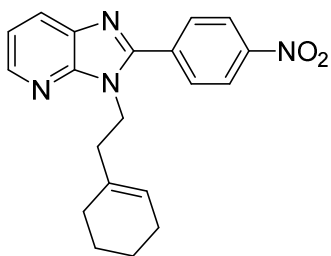


Figure S7: ^1H and ^{13}C NMR spectrum of compound 4d in CDCl_3

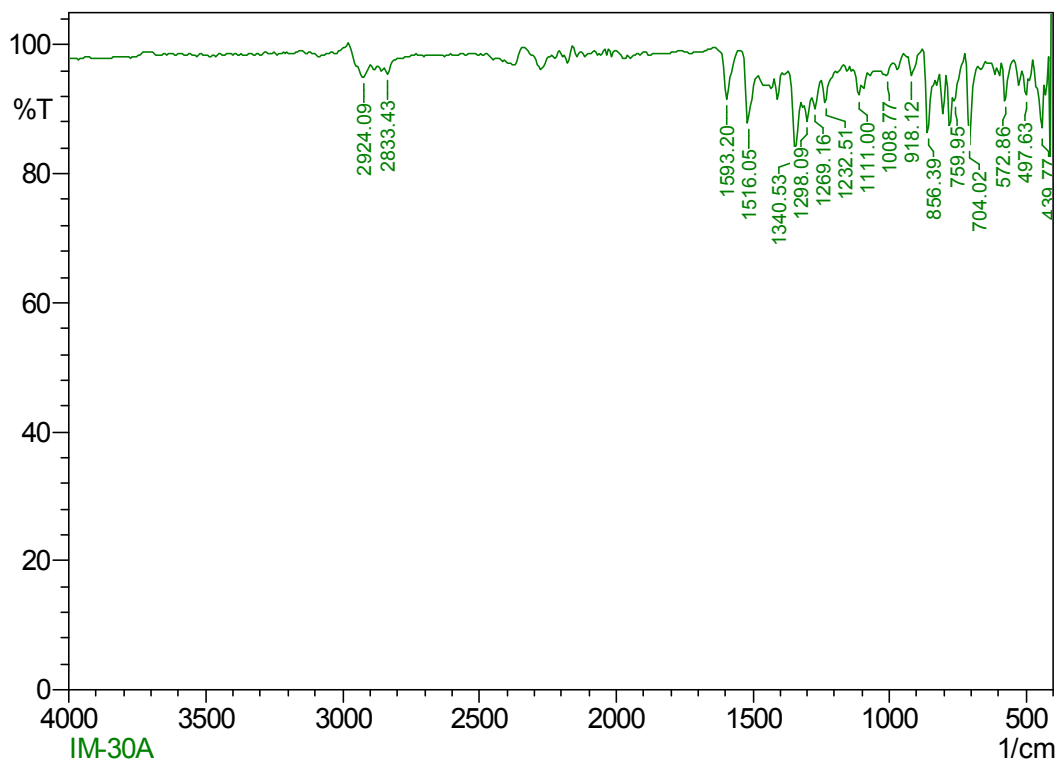
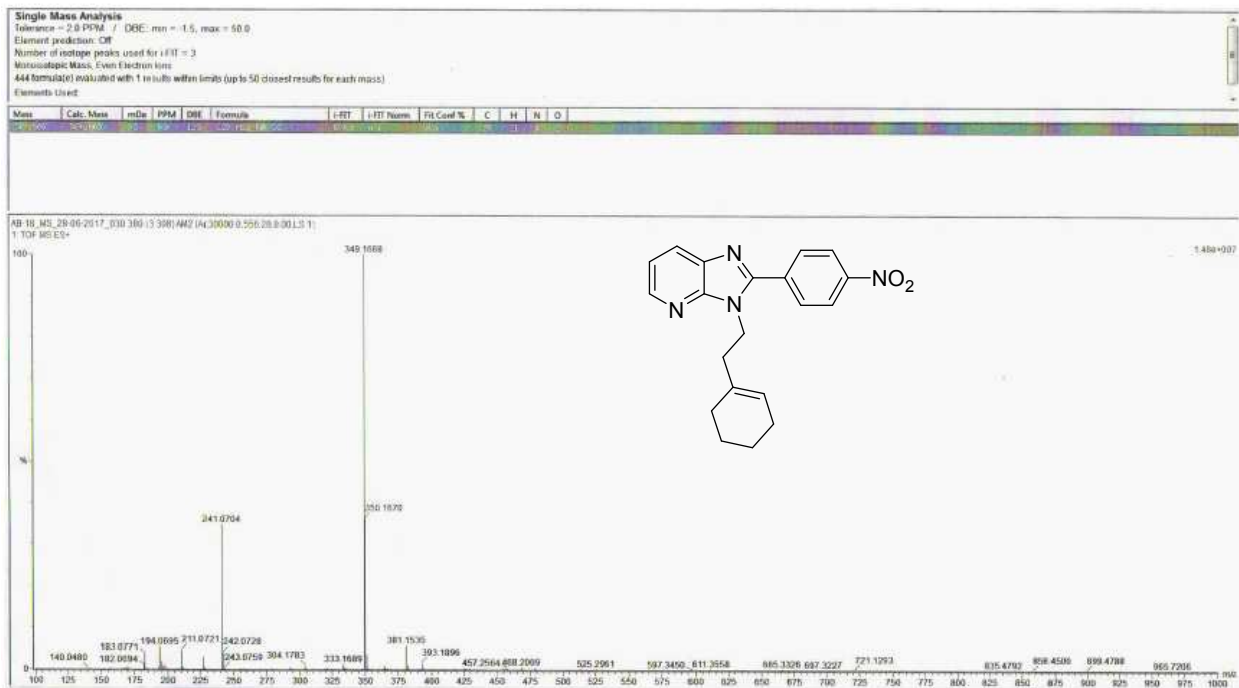


Figure S8: HR Mass and IR spectra of compound 4d

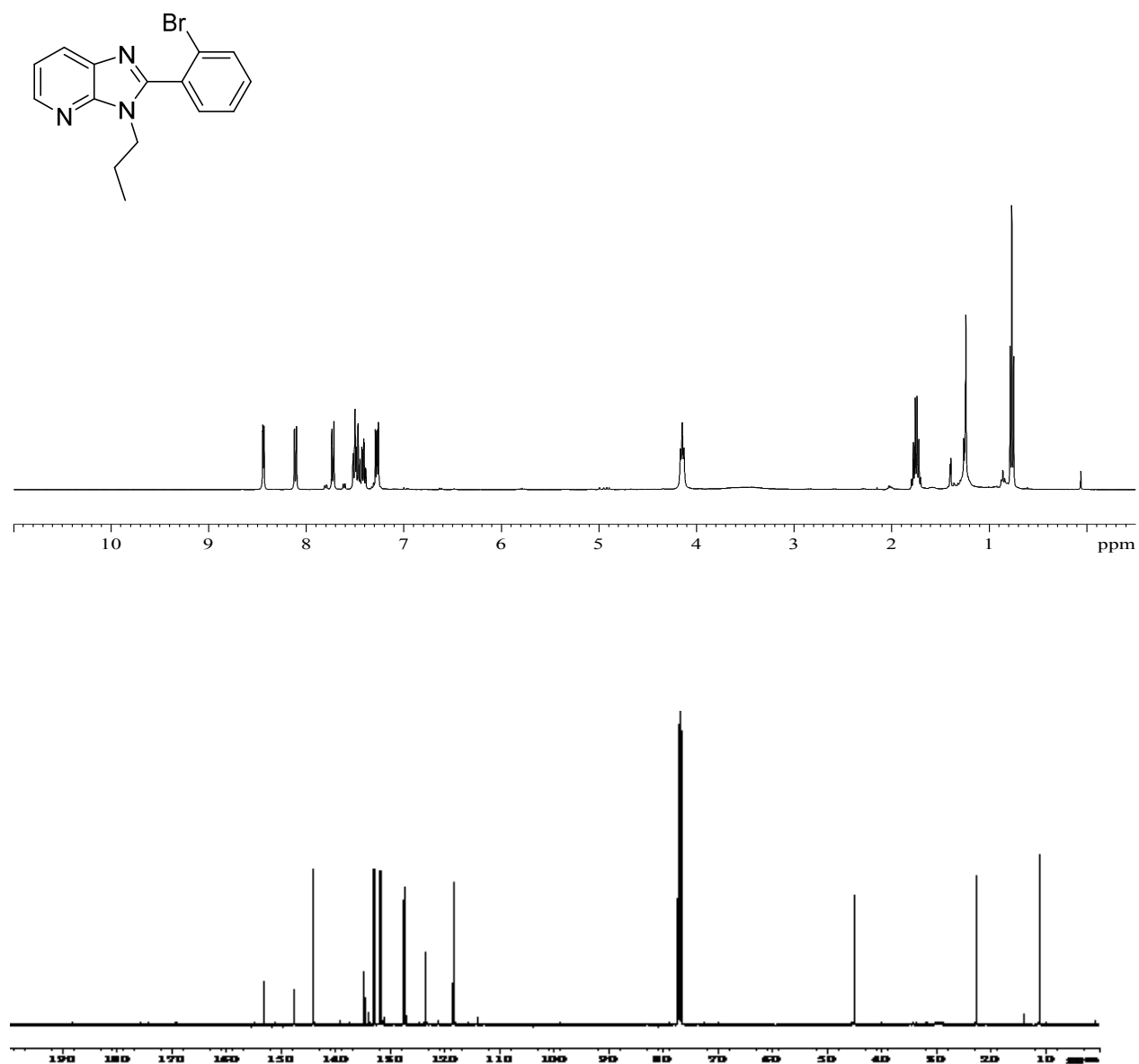


Figure S9: ^1H and ^{13}C NMR spectrum of compound 4e in CDCl_3

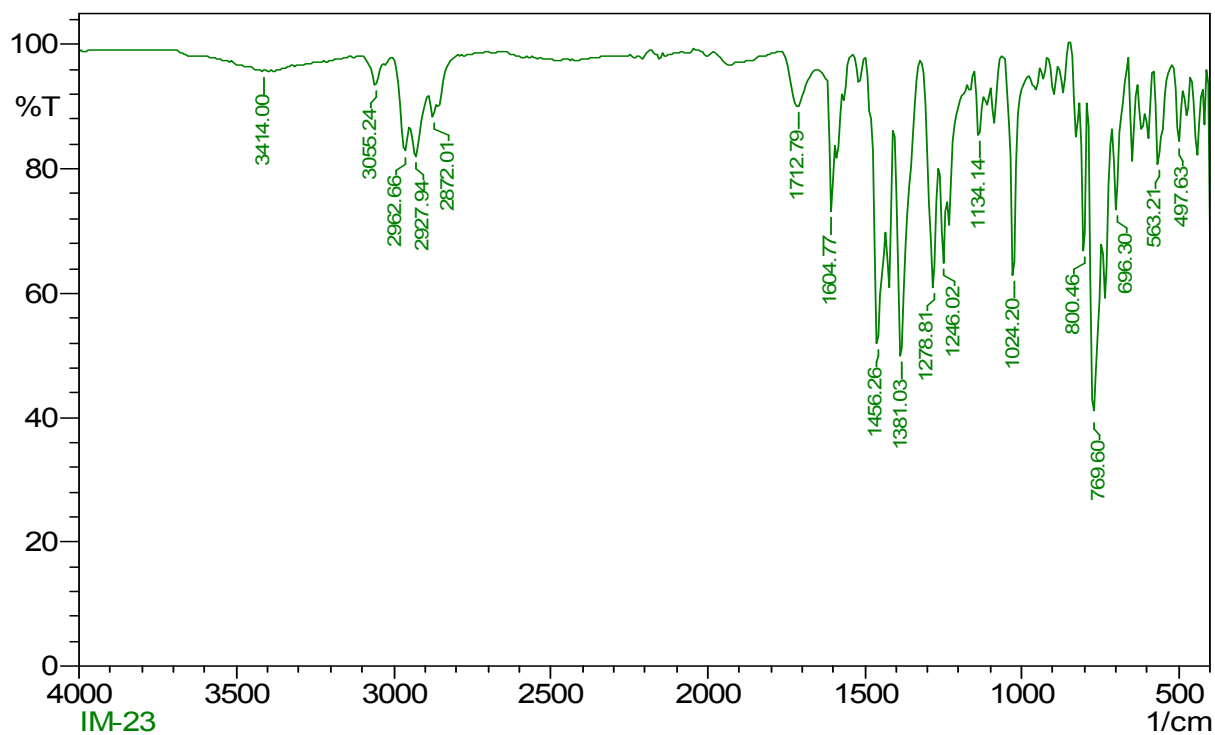
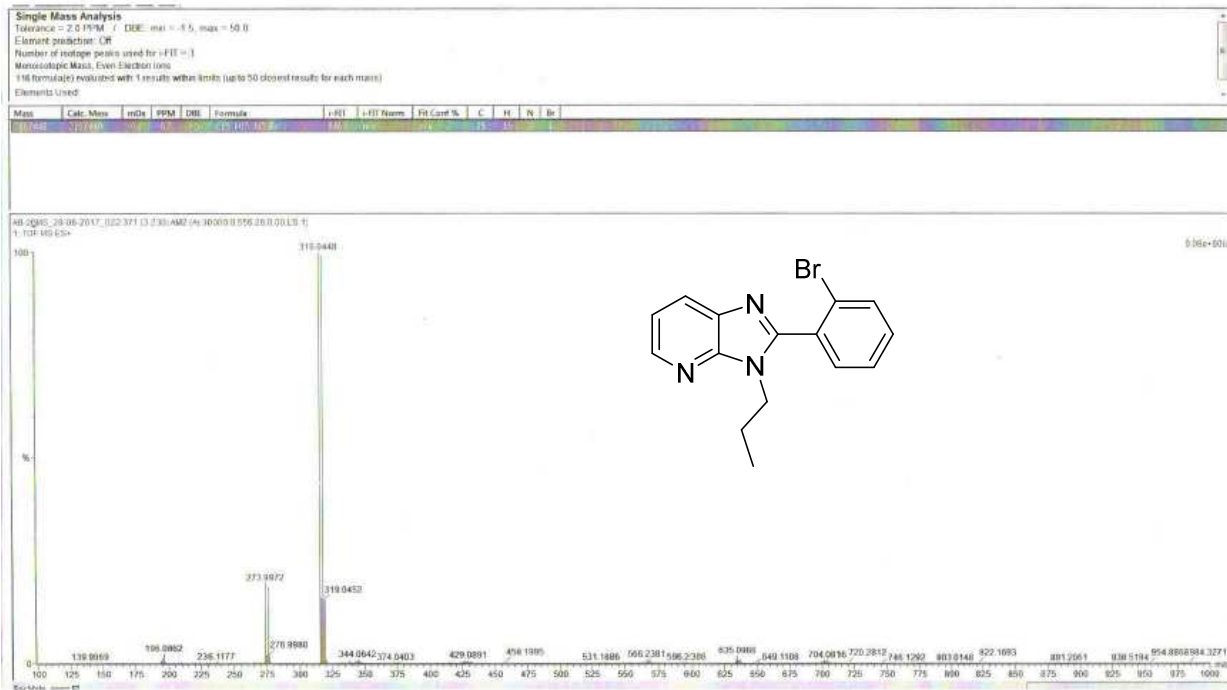


Figure S10:HR mass and IR spectra of Compound 4e

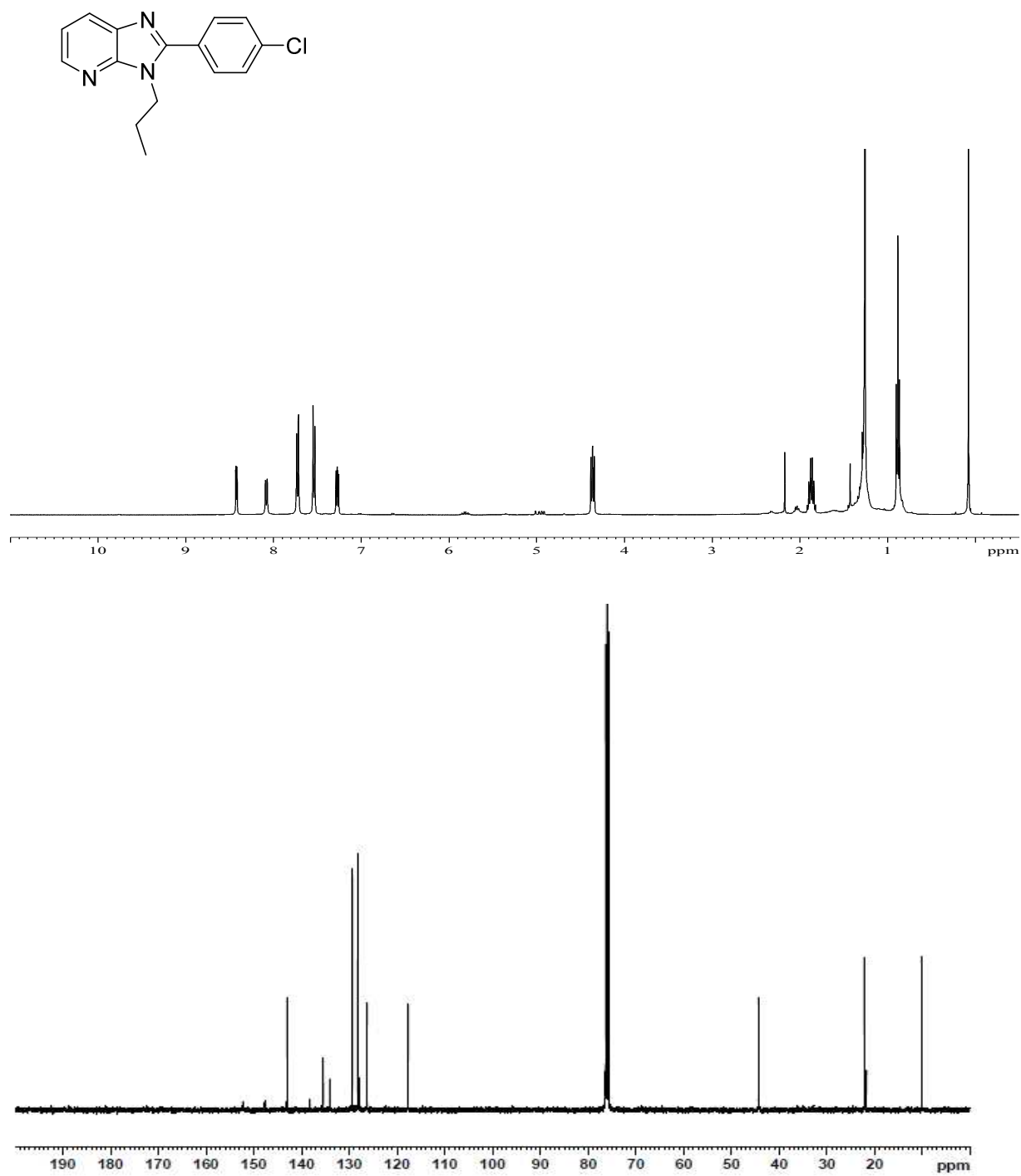


Figure S11: ^1H and ^{13}C NMR spectrum of compound 4f in CDCl_3

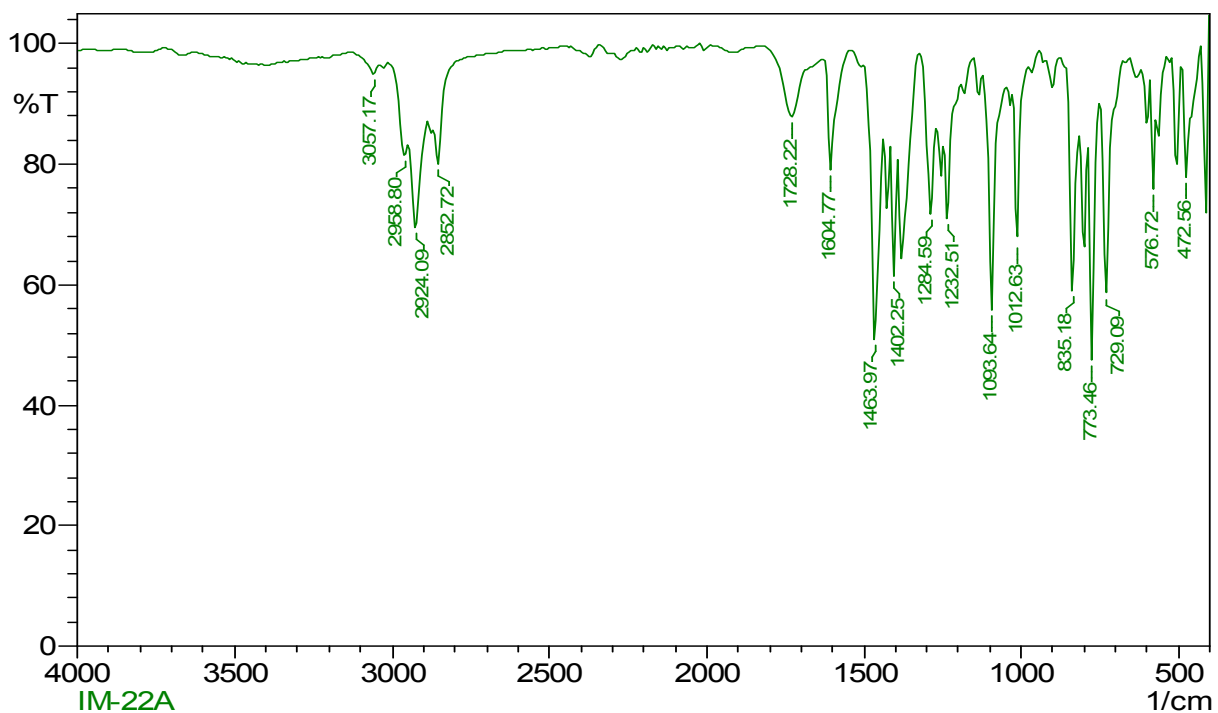
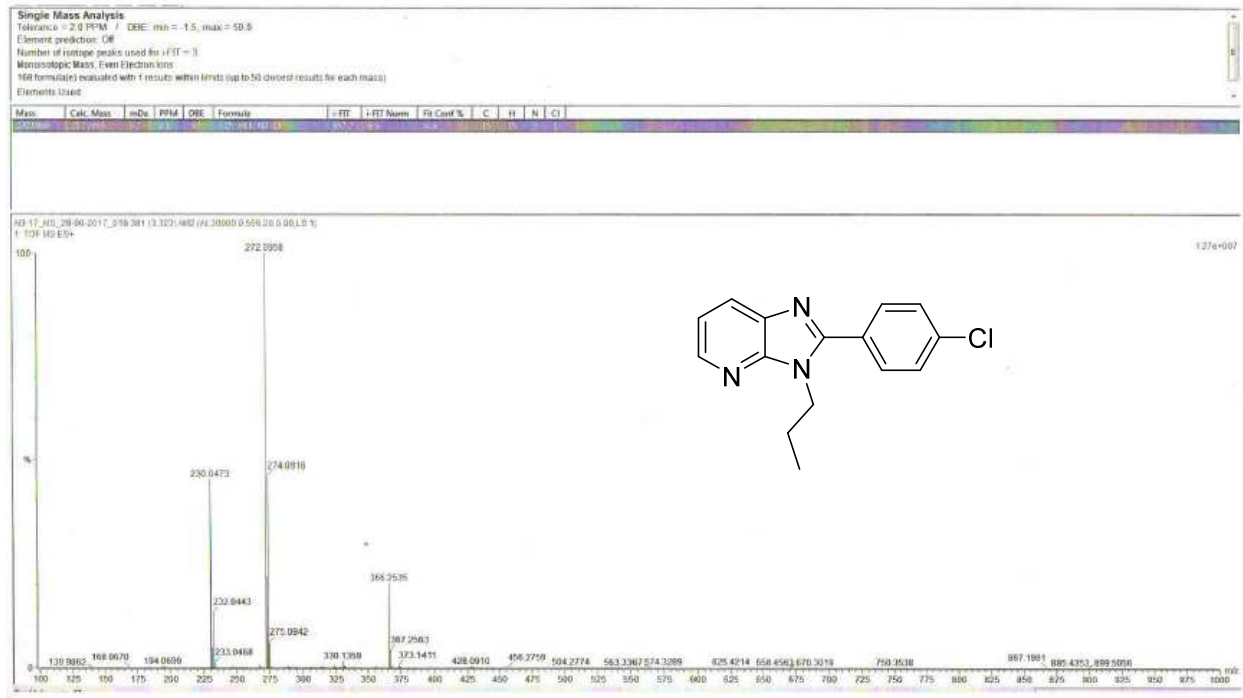


Figure S12:HR Mass and IR spectra of compound 4f

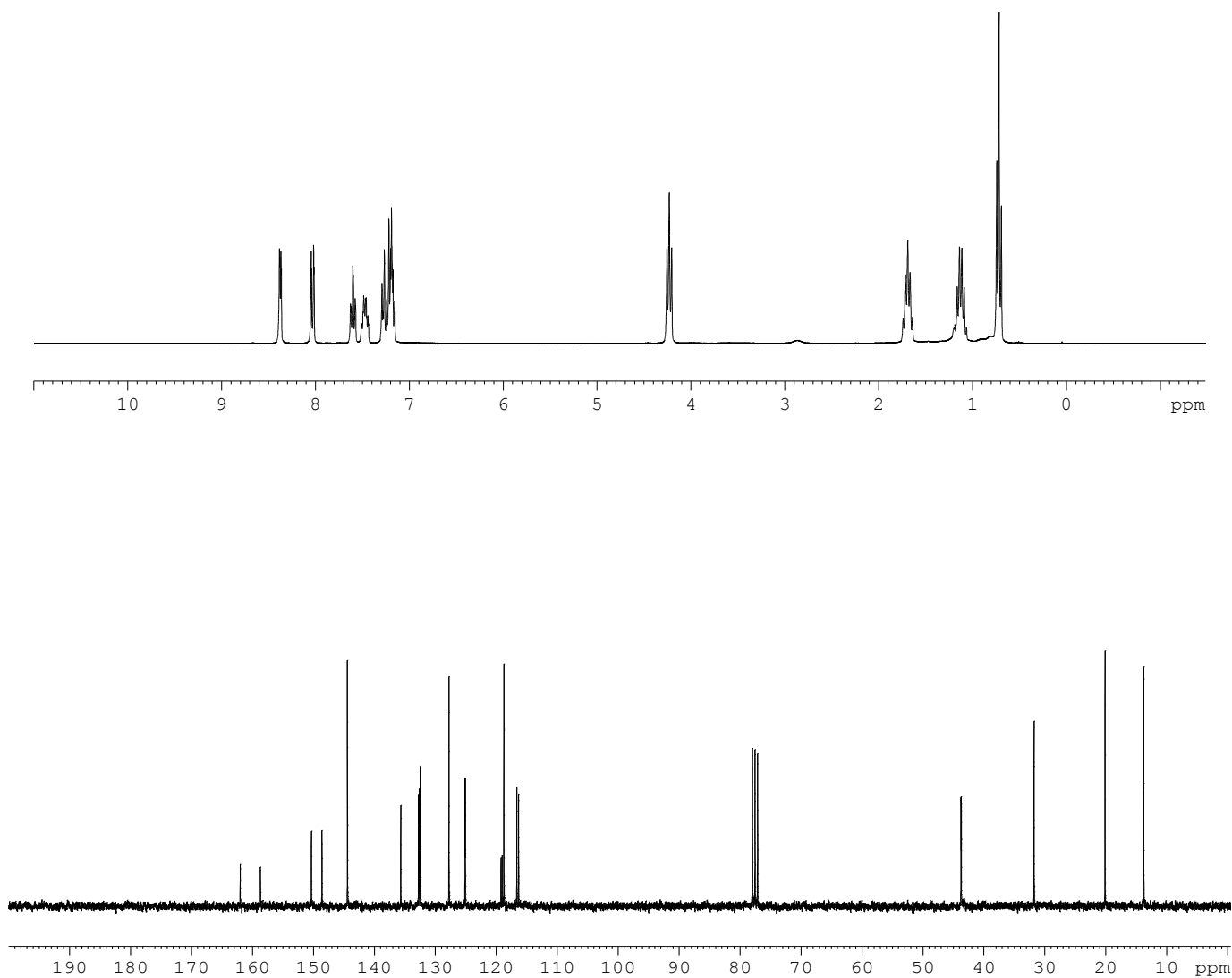
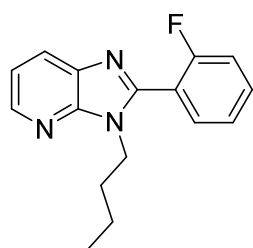


Figure S13: ¹H and ¹³C NMR spectrum of compound 4g in CDCl₃

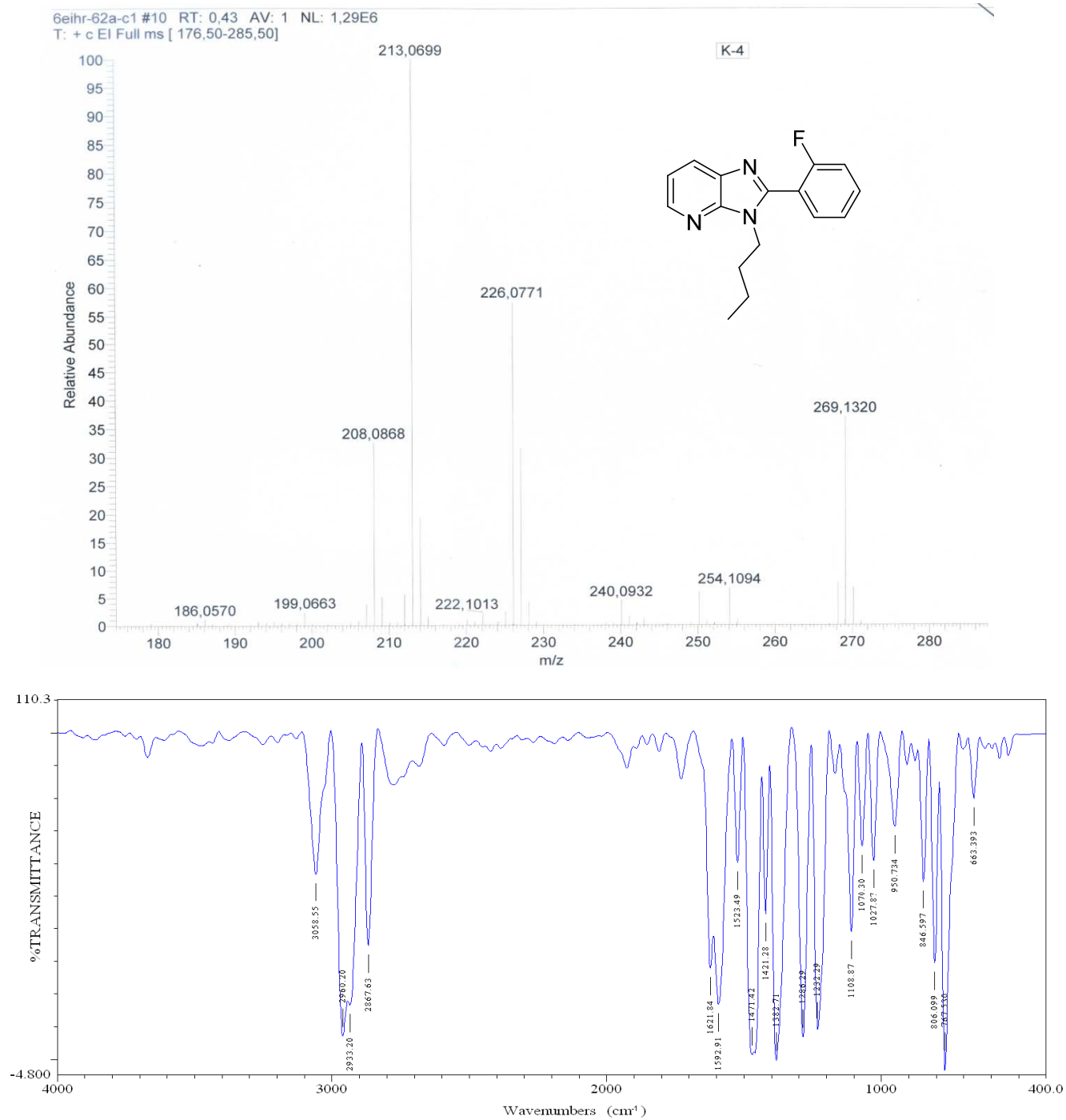


Figure S14: HR Mass and IR spectra of compound 4g

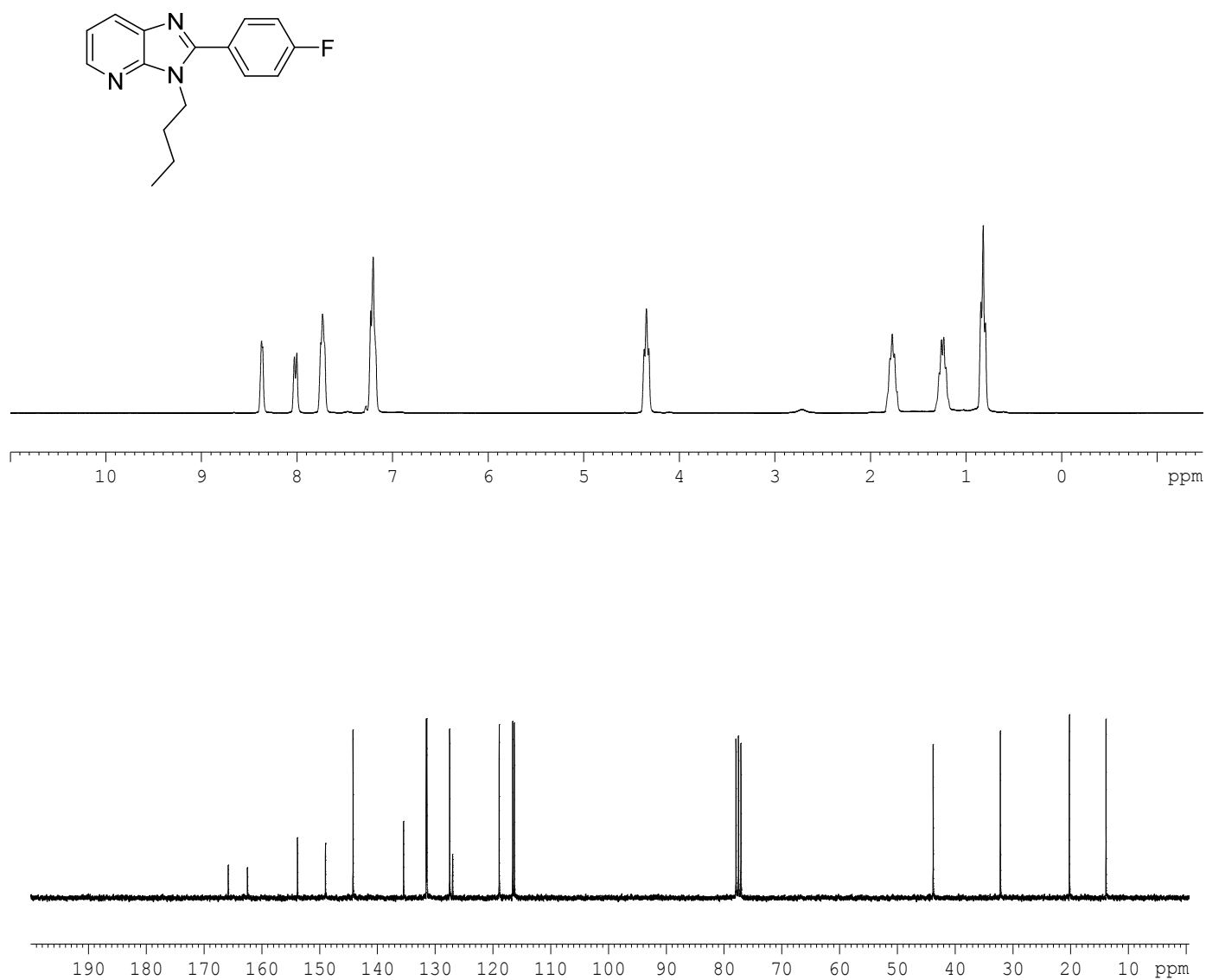


Figure S15: ^1H and ^{13}C NMR spectrum of compound 4h in CDCl_3

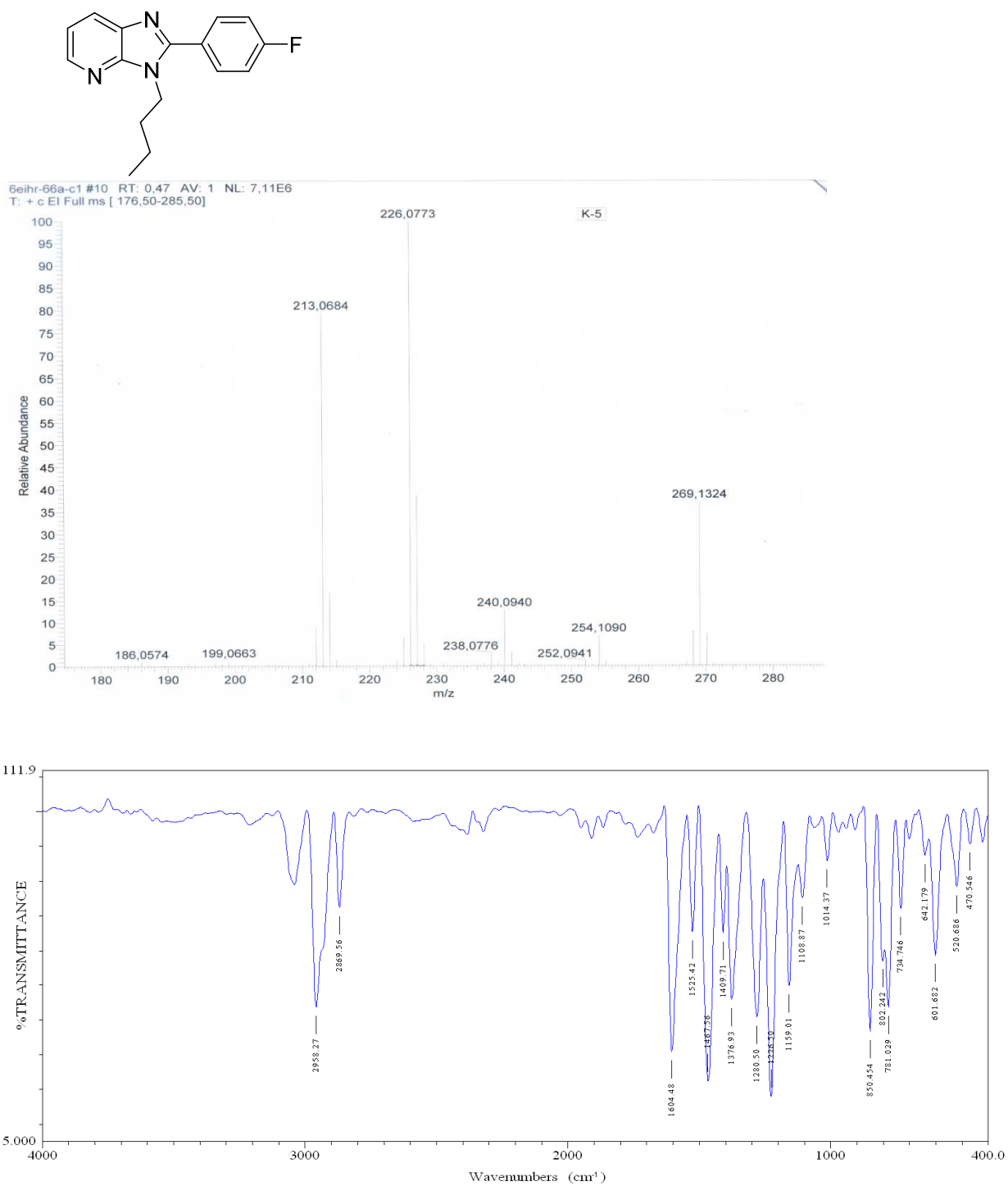


Figure S16: HR Mass and IR spectra of compound 4h

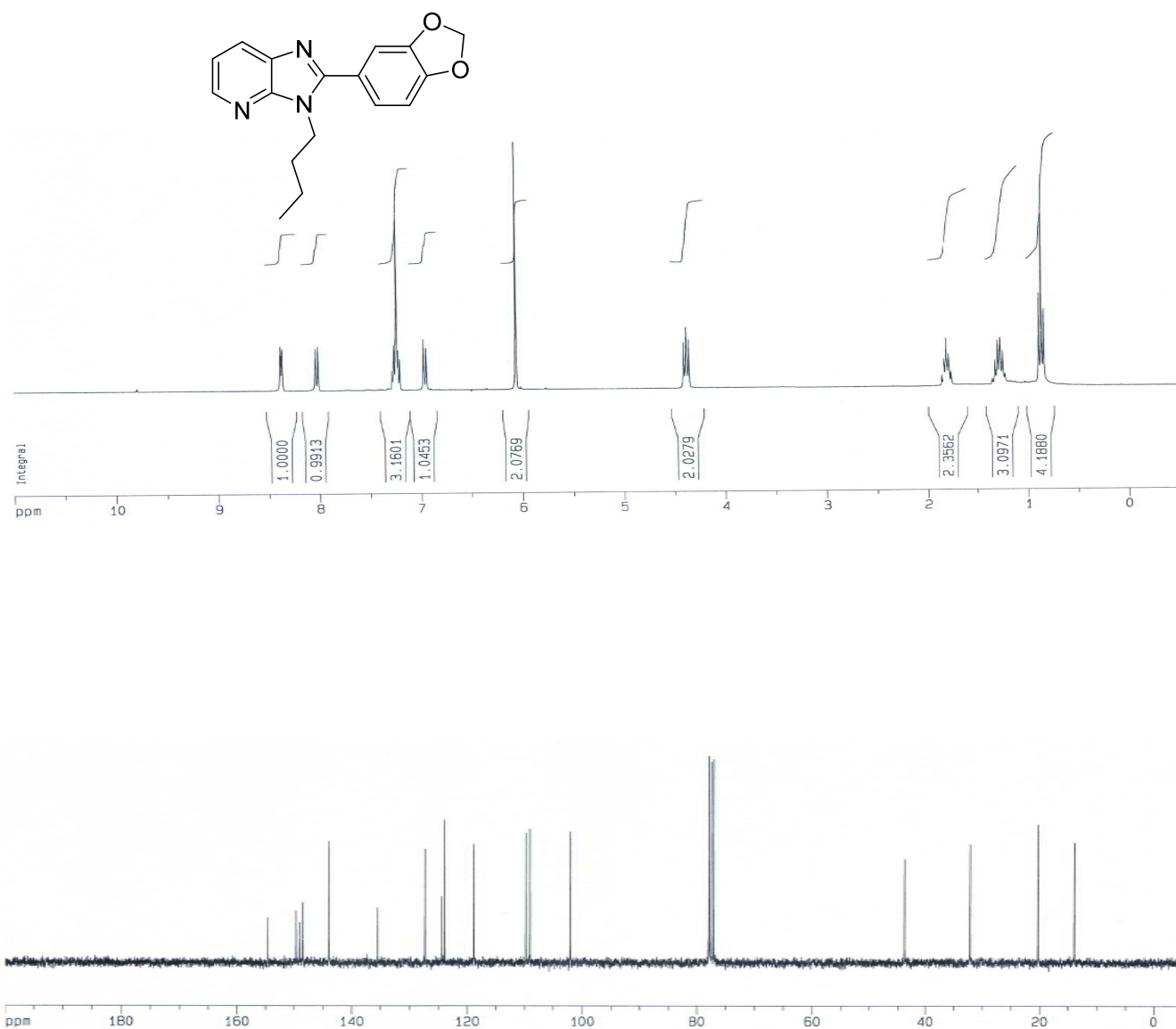


Figure S17: ¹H and ¹³C NMR spectrum of compound 4i in CDCl₃

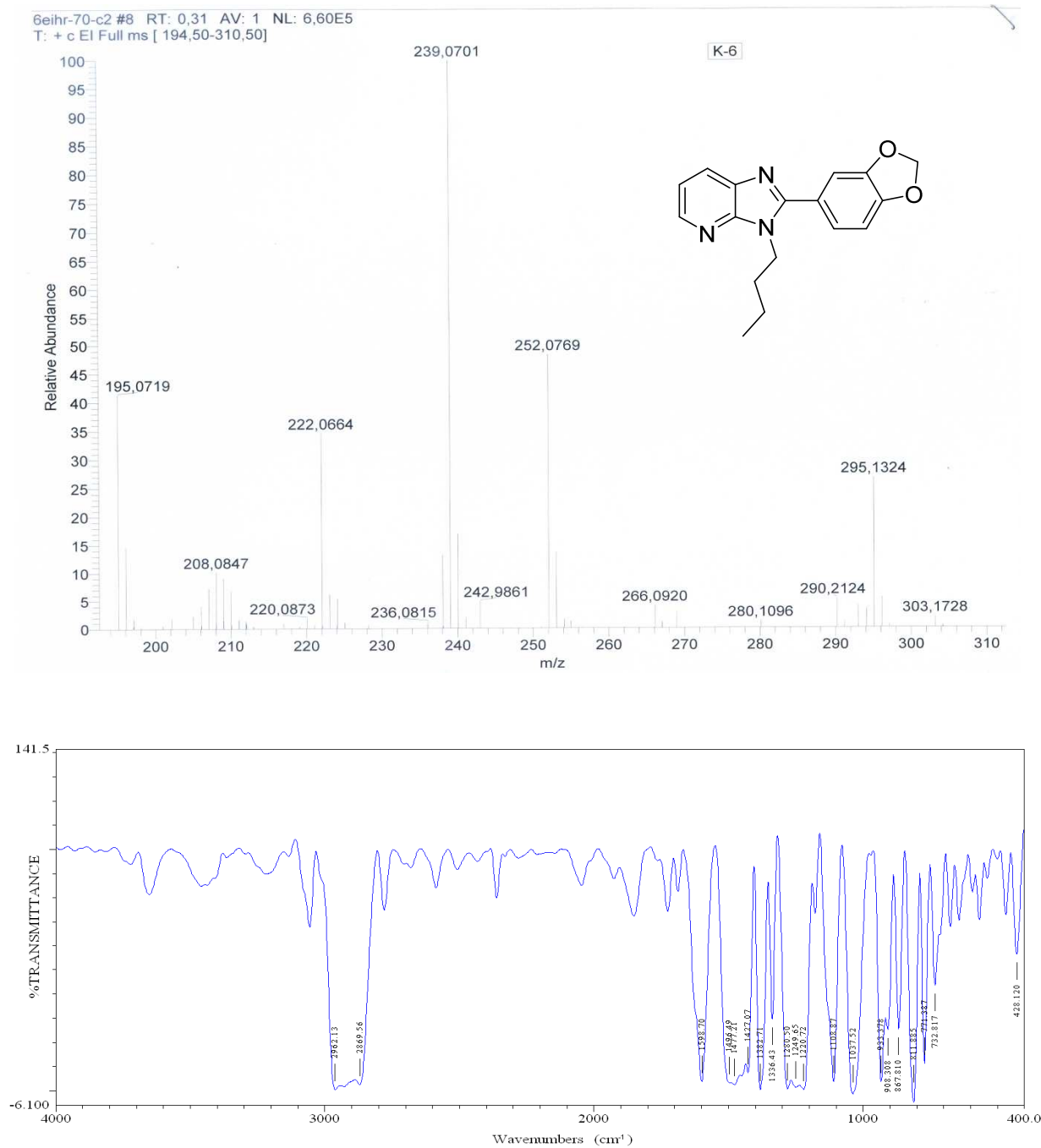


Figure S18: HR Mass and IR spectra of compound 4i

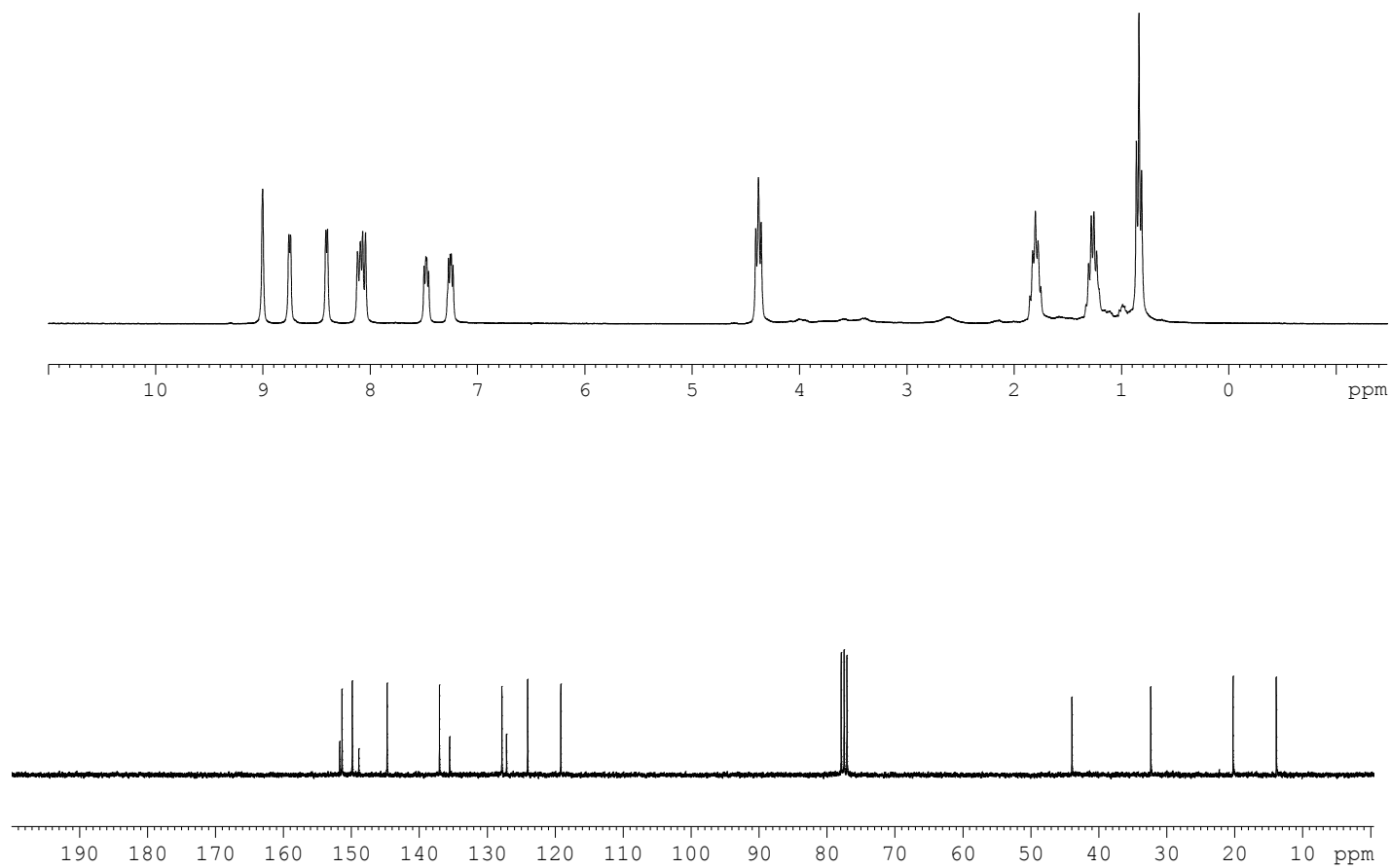
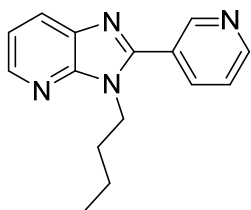


Figure S19: ^1H and ^{13}C NMR spectrum of compound 4j in CDCl_3

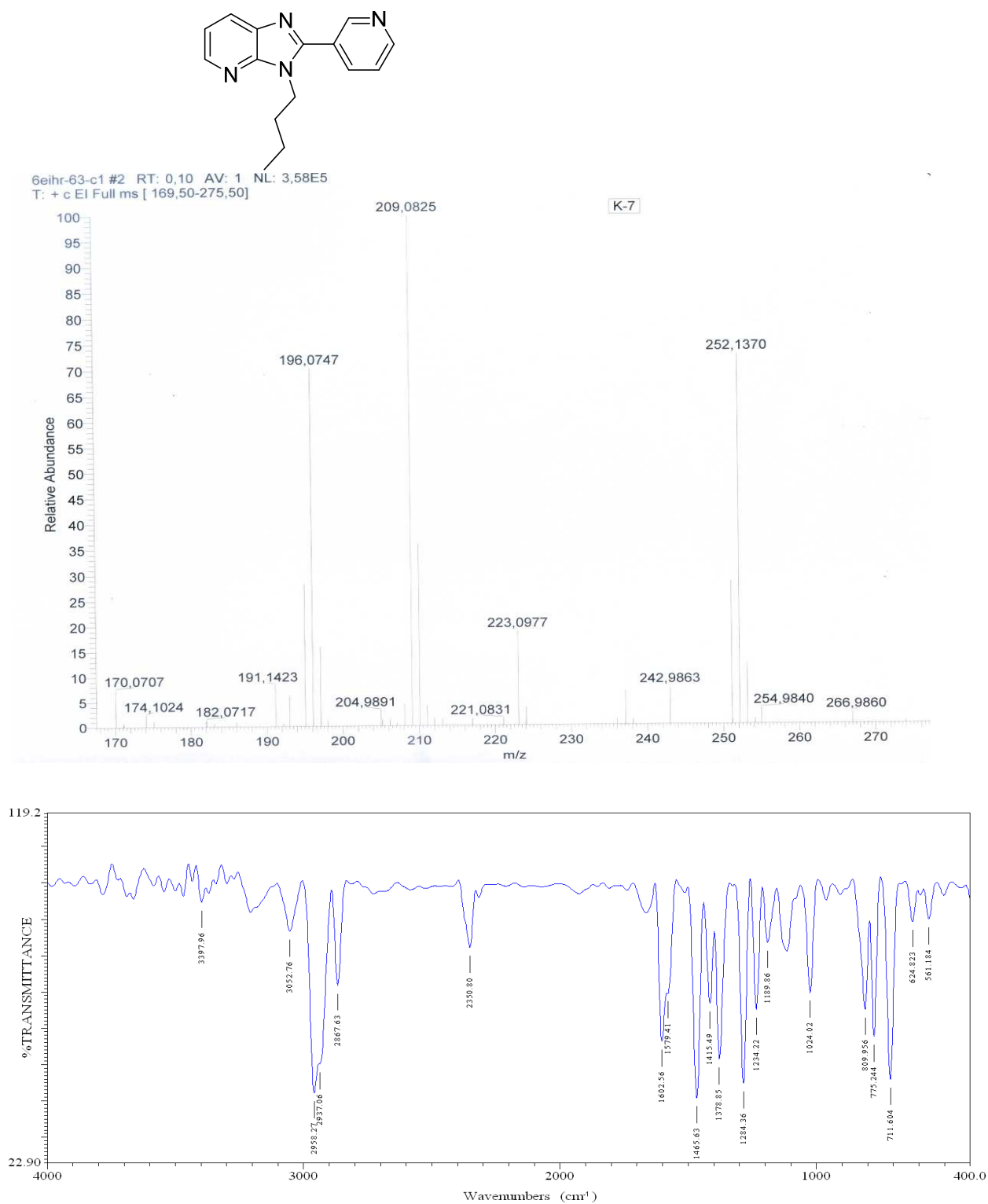


Figure S20: HR Mass and IR spectra of compound 4j

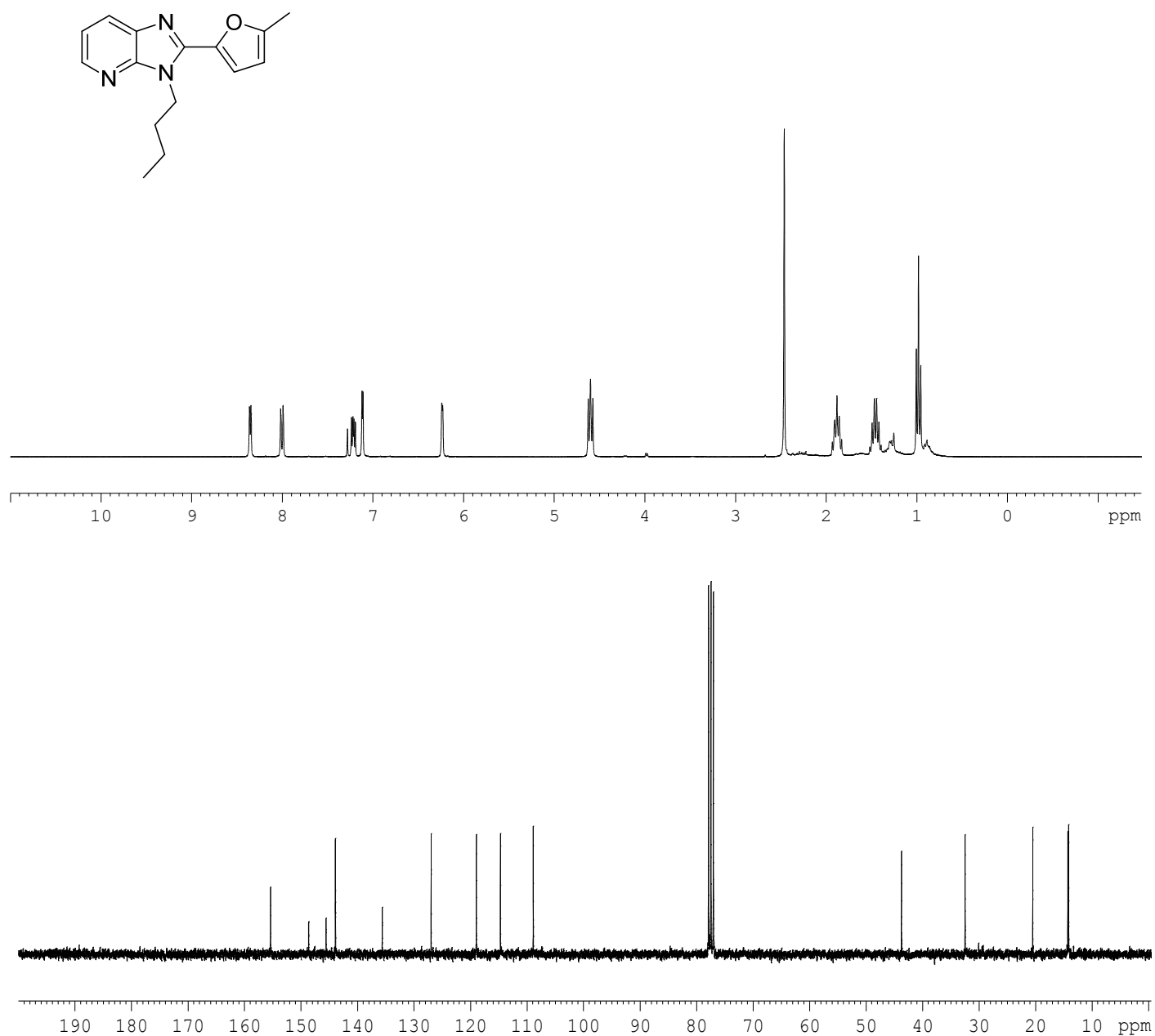


Figure S21: ^1H and ^{13}C NMR spectrum of compound 4k in CDCl_3

6e1hr-64-c1 #2 RT: 0.10 AV: 1 NL: 6,21E5
T: + c EI Full ms [169,50-275,50]

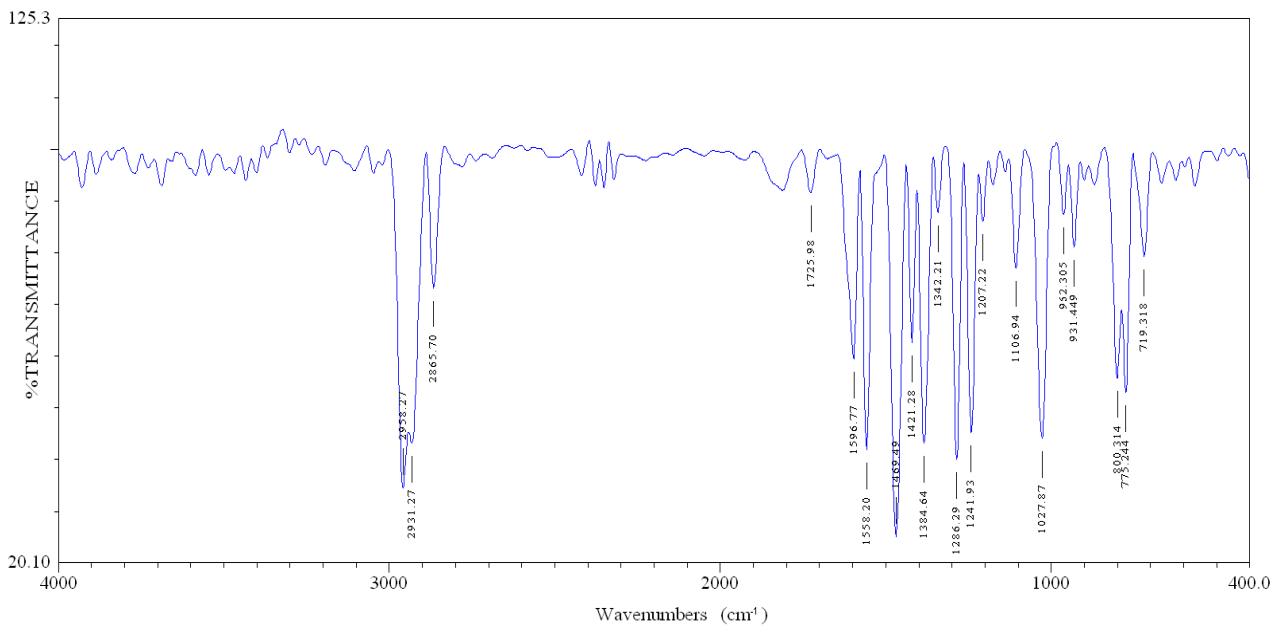
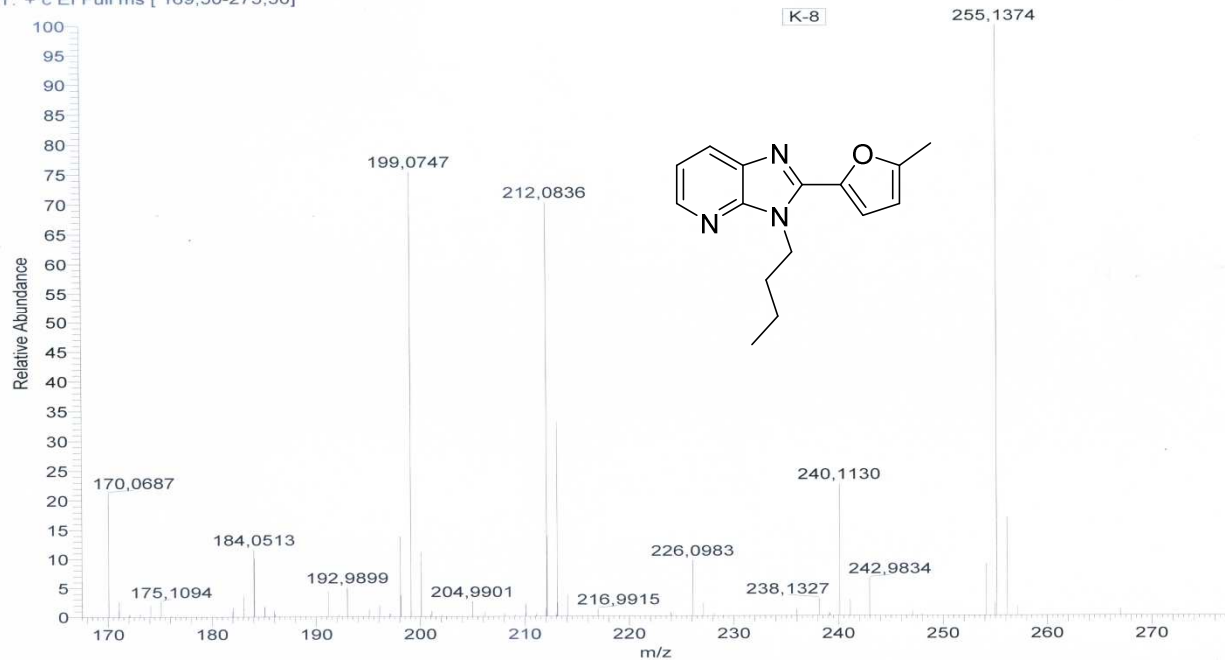


Figure S22: HR Mass and IR spectra of compound 4k

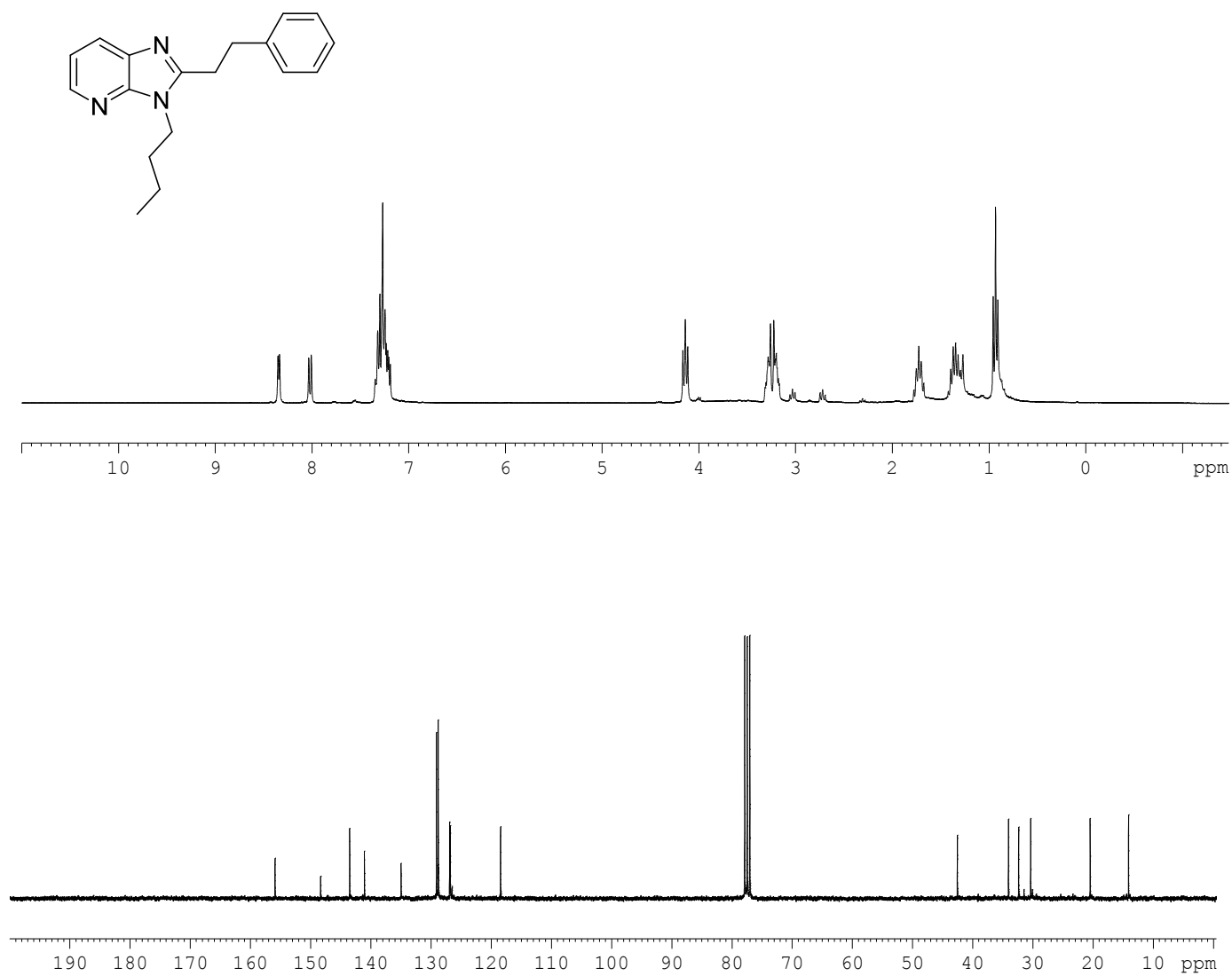


Figure S23: ^1H and ^{13}C NMR spectrum of compound 4l in CDCl_3

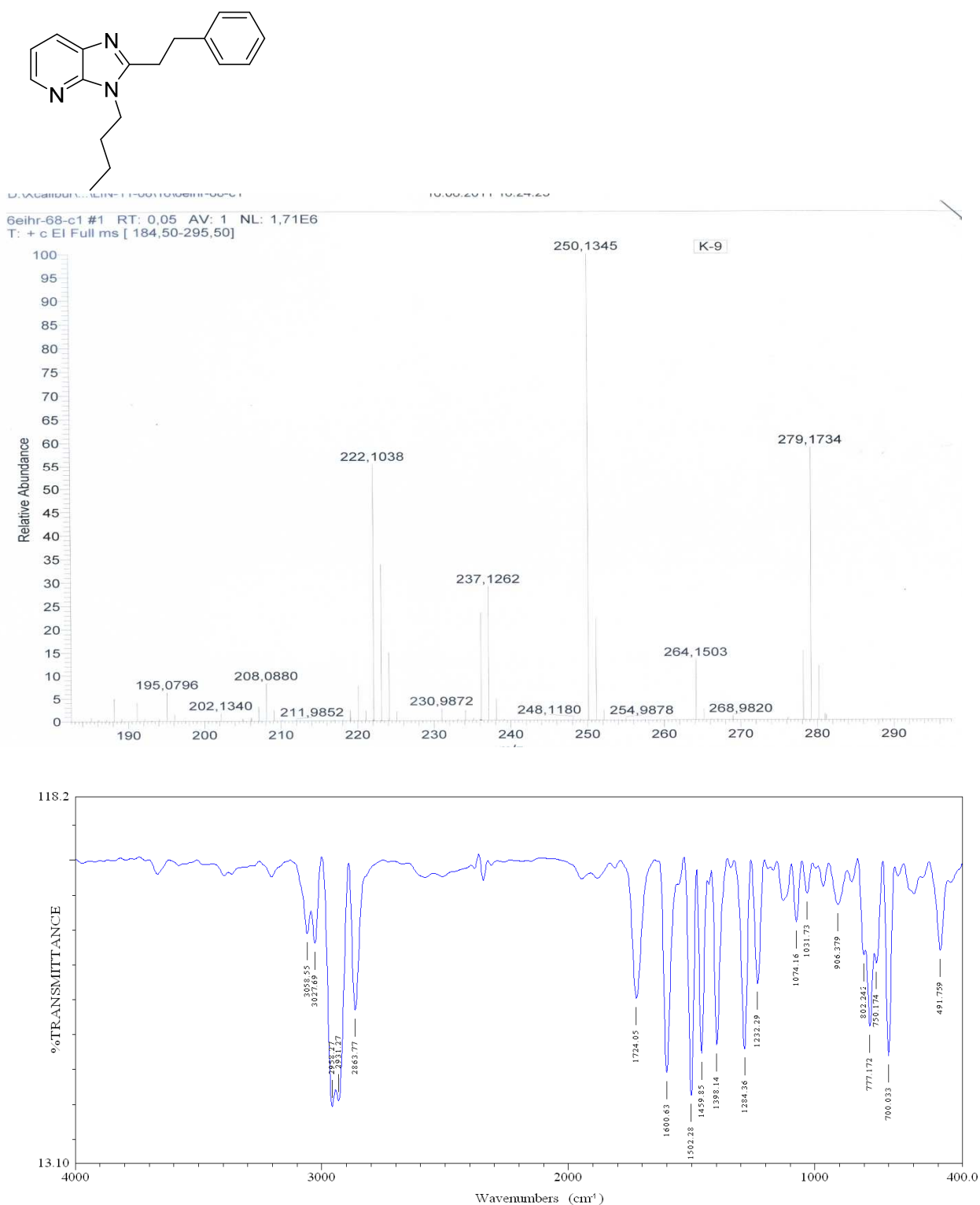


Figure S24: HR Mass and IR spectra of compound 41

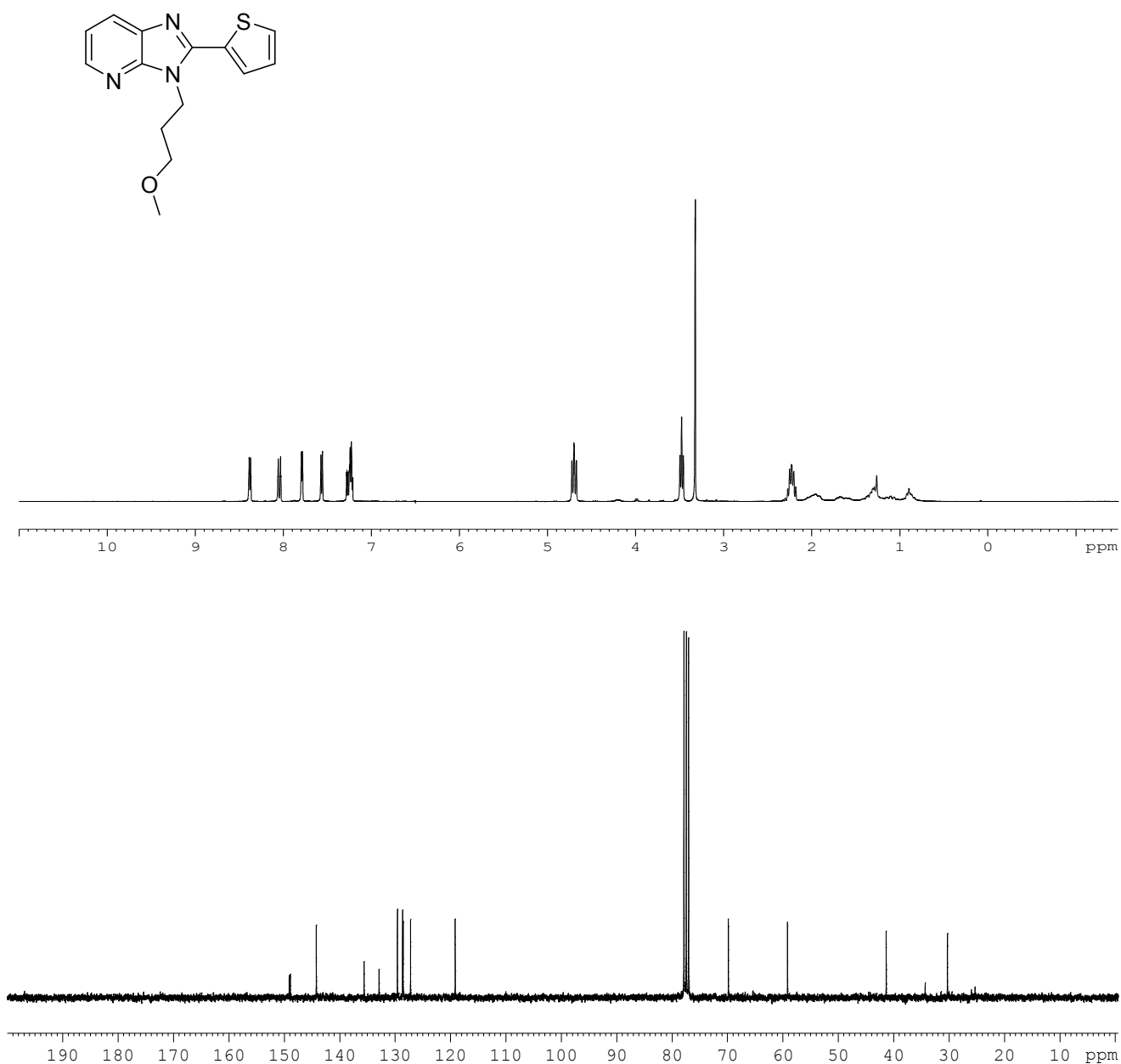


Figure S25: ^1H and ^{13}C NMR spectrum of compound 4m in CDCl_3

6e1hr-67-c1 #1 RT: 0,06 AV: 1 NL: 1,97E6
T: + c EI Full ms [176,50-285,50]

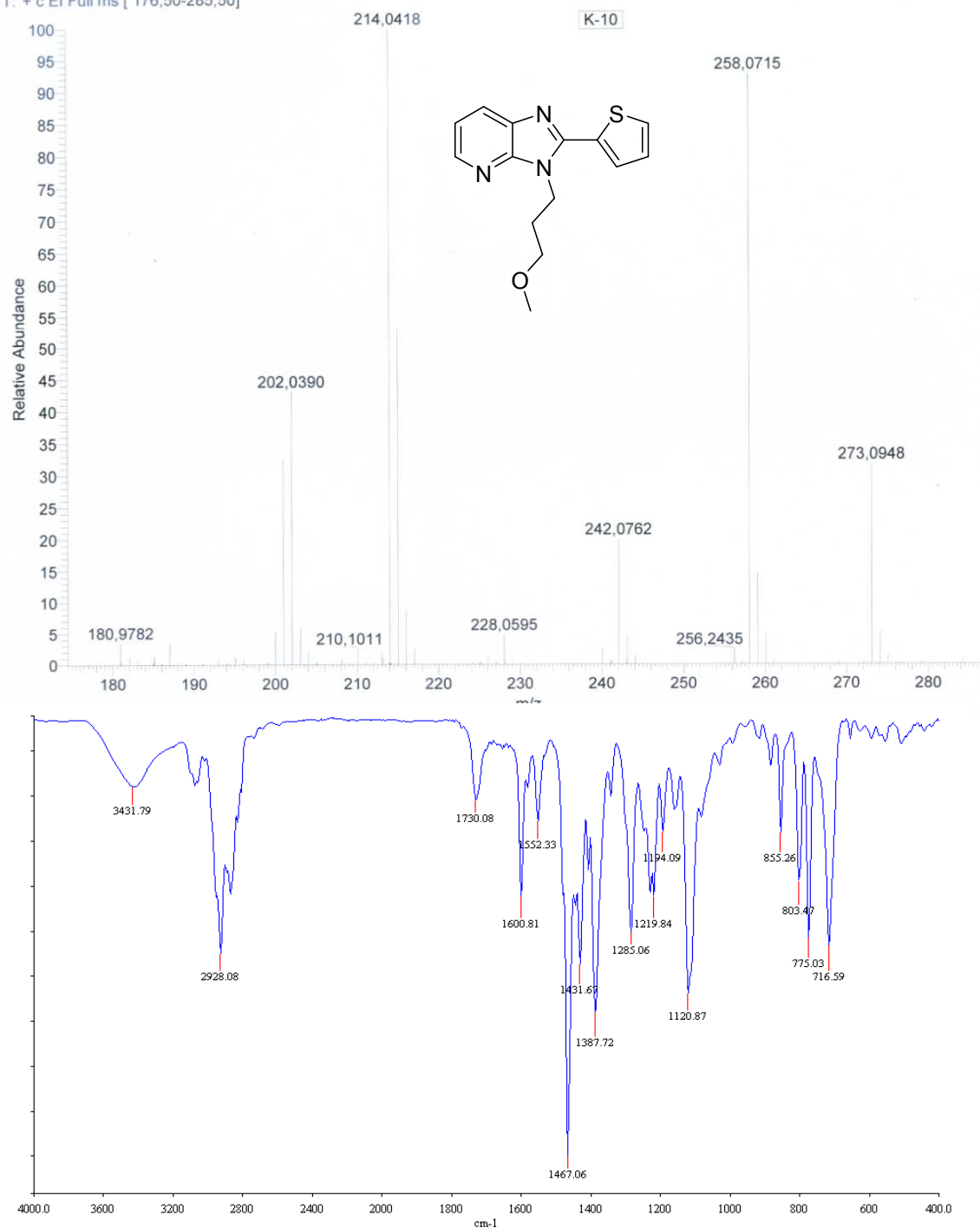


Figure S26: HR Mass and IR spectra of compound 4m

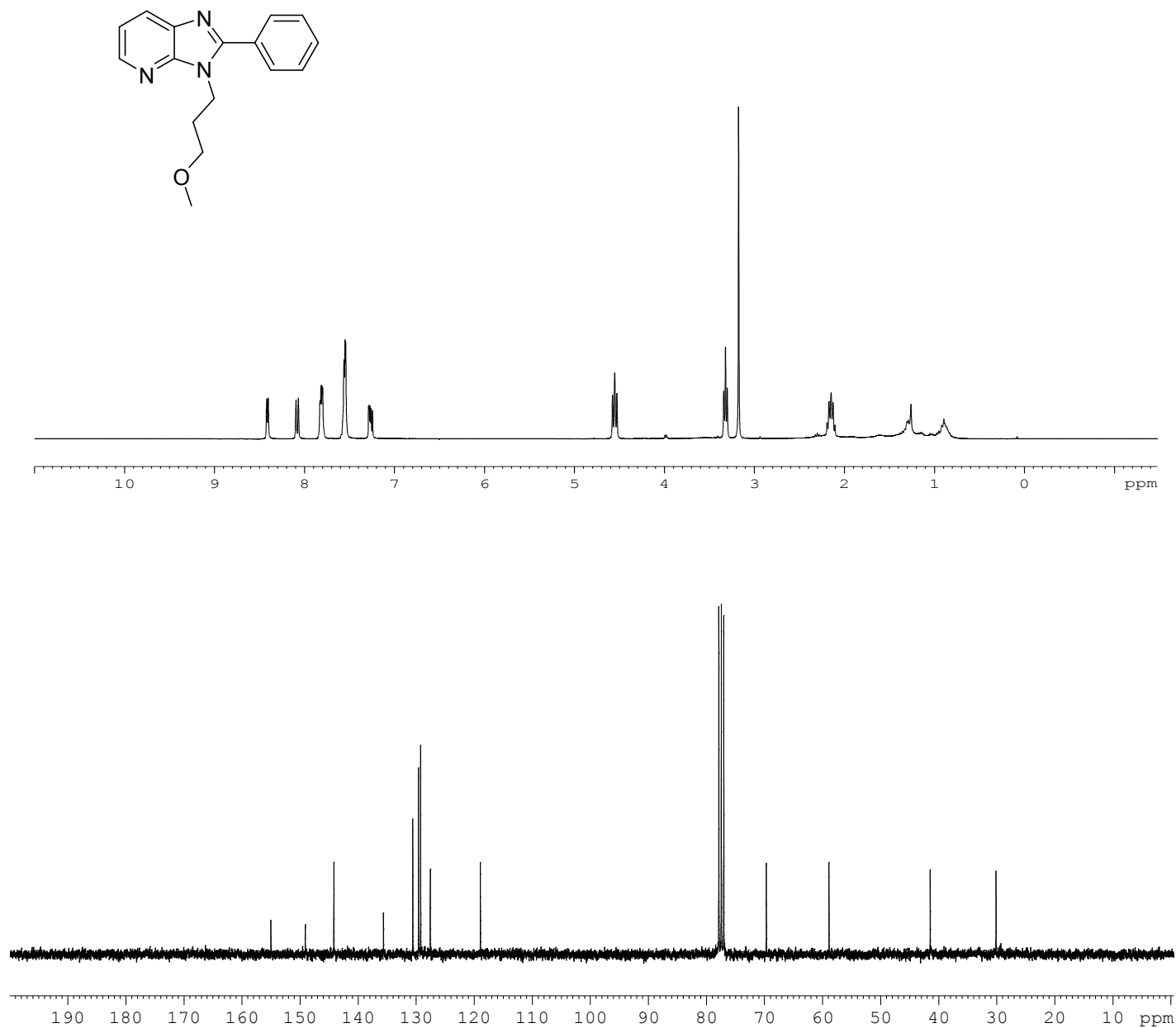


Figure S27: ^1H and ^{13}C NMR spectrum of compound 4n in CDCl_3

6ehr-74-c1 #1 RT: 0,05 AV: 1 NL: 1,66E6
T: + c EI Full ms [176,50-285,50]

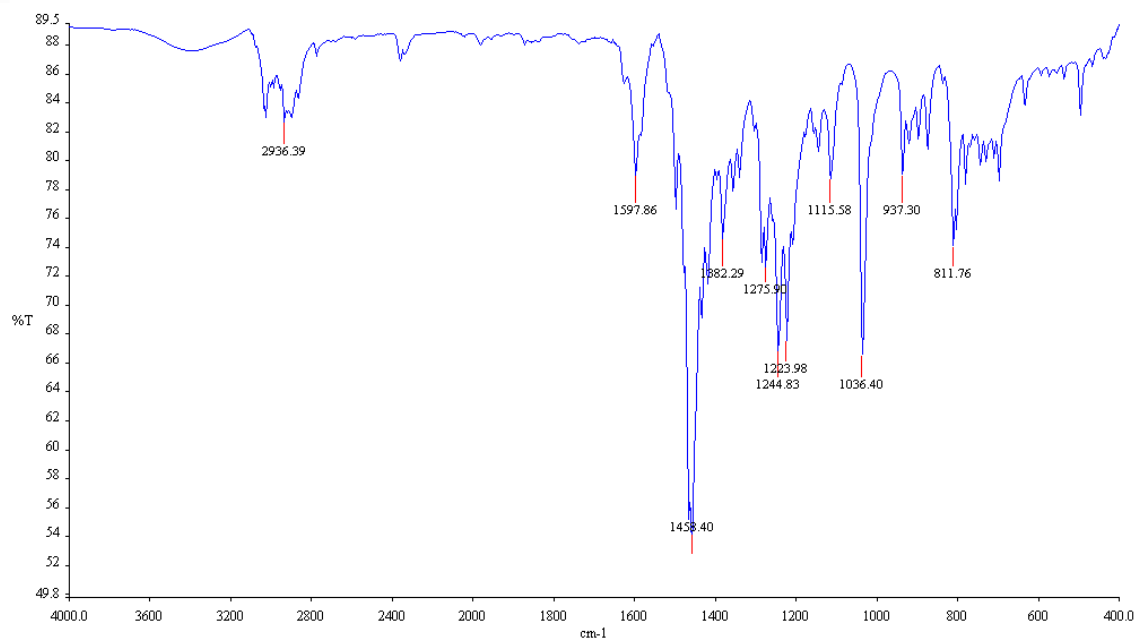
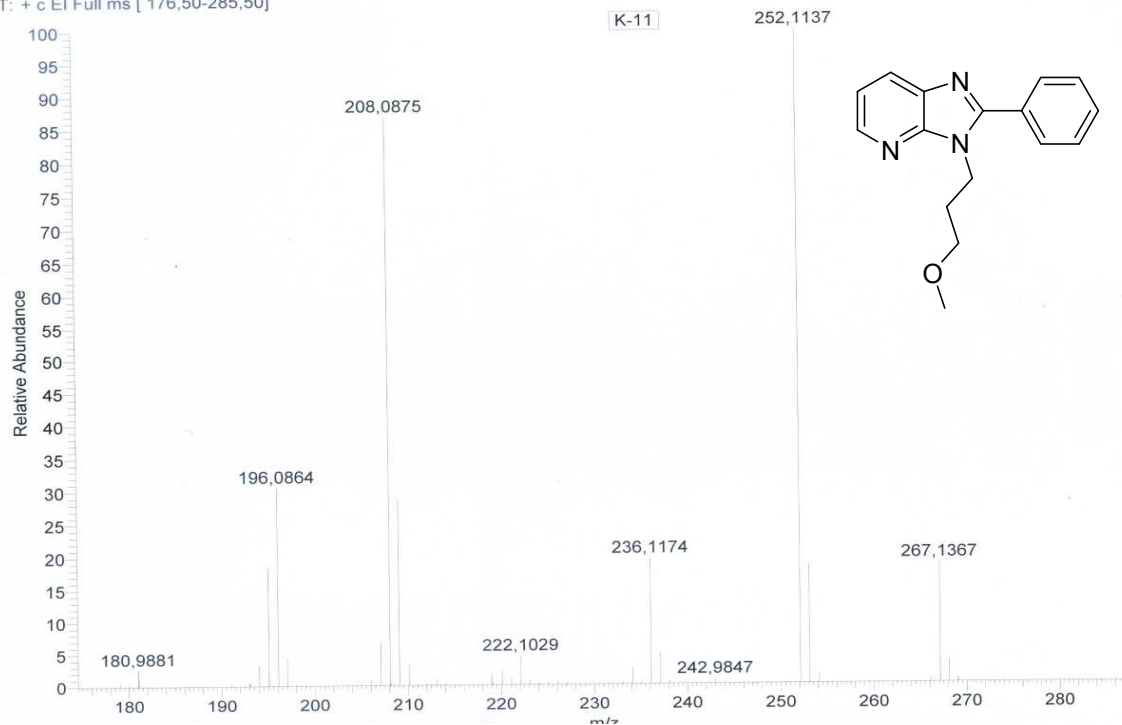


Figure S28:HR Mass and IR spectra of compound 4n

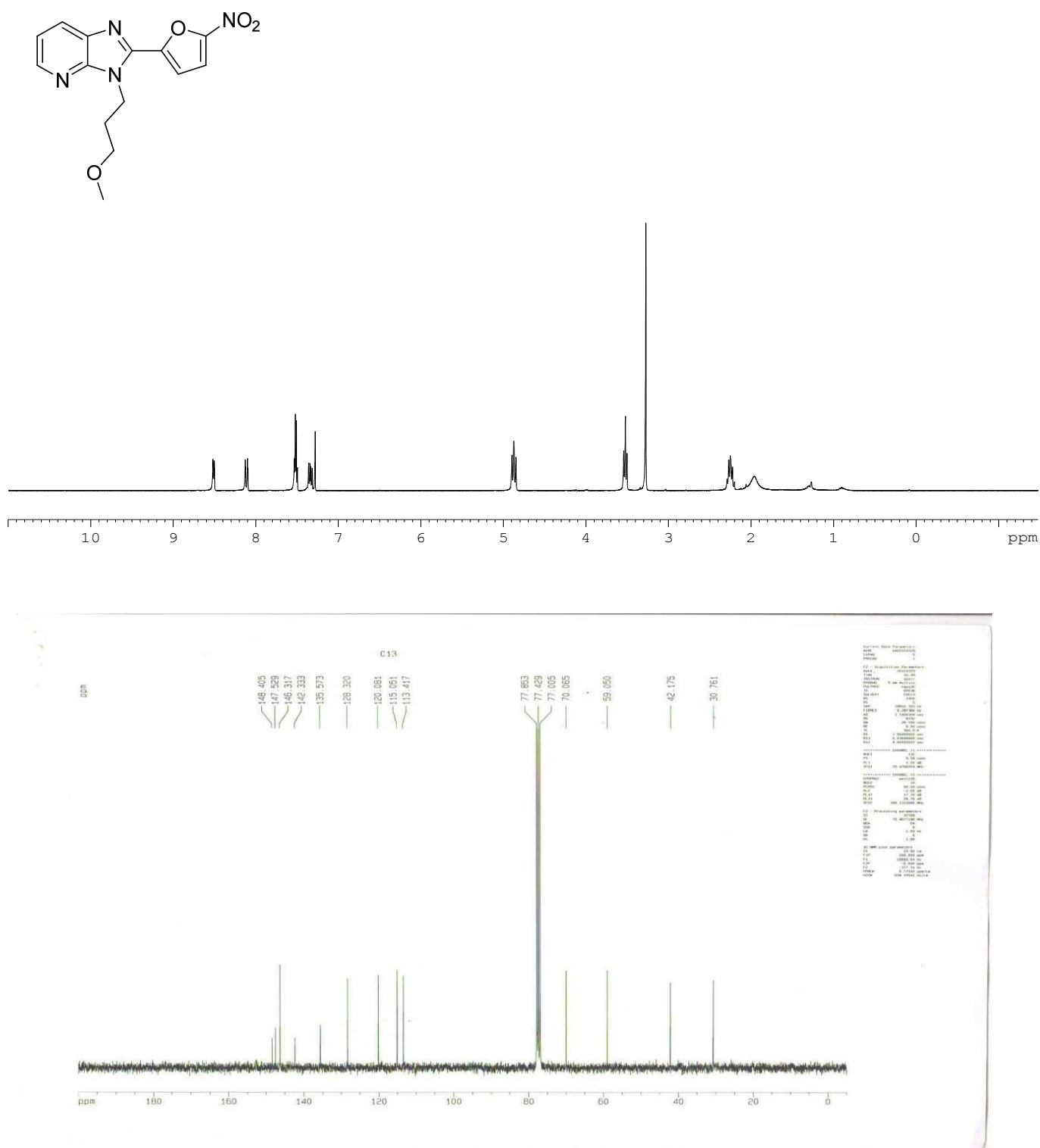


Figure S29: ¹H and ¹³C NMR spectrum of compound 40 in CDCl₃

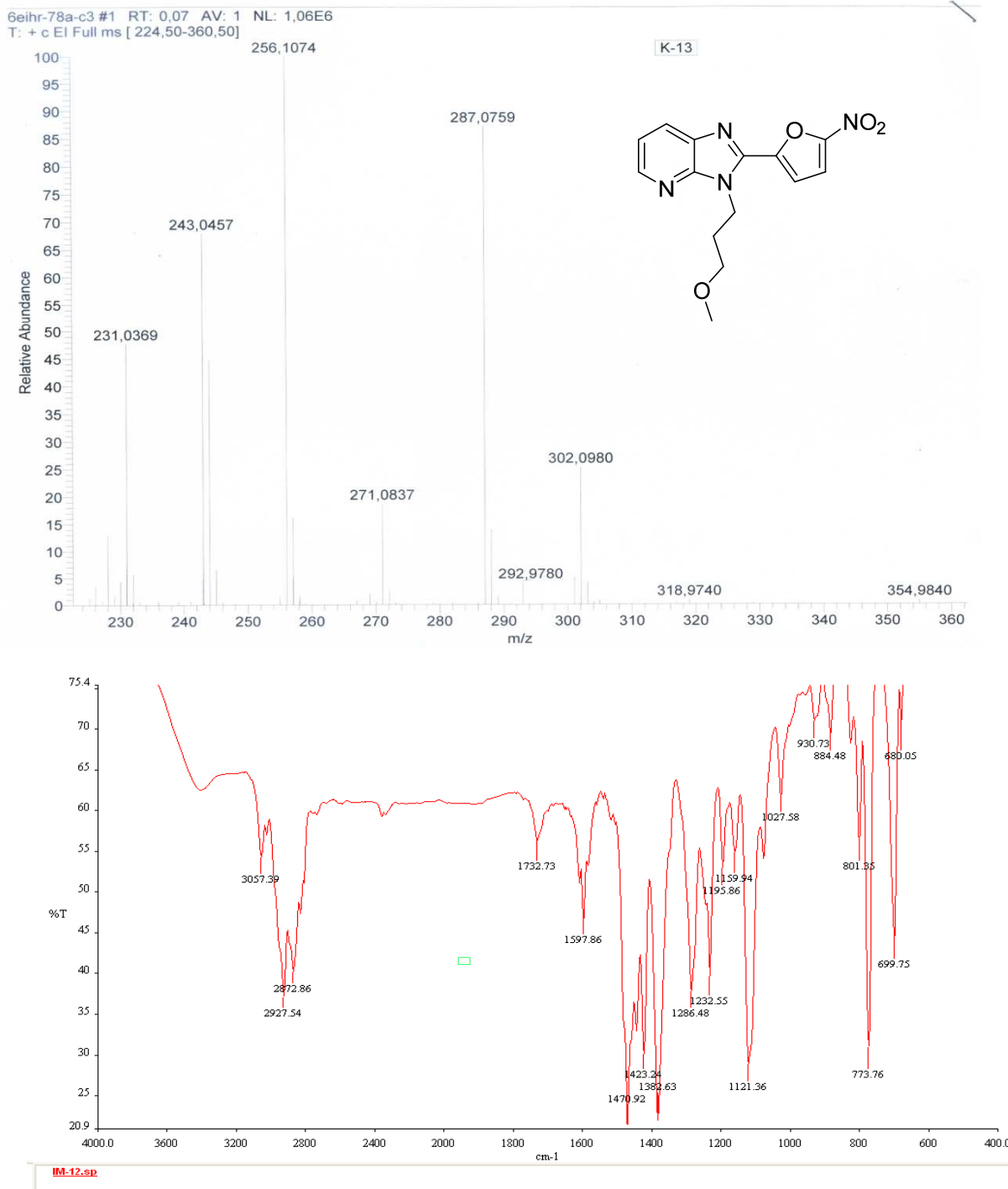


Figure S30:HR Mass and IR spectra of compound 4o

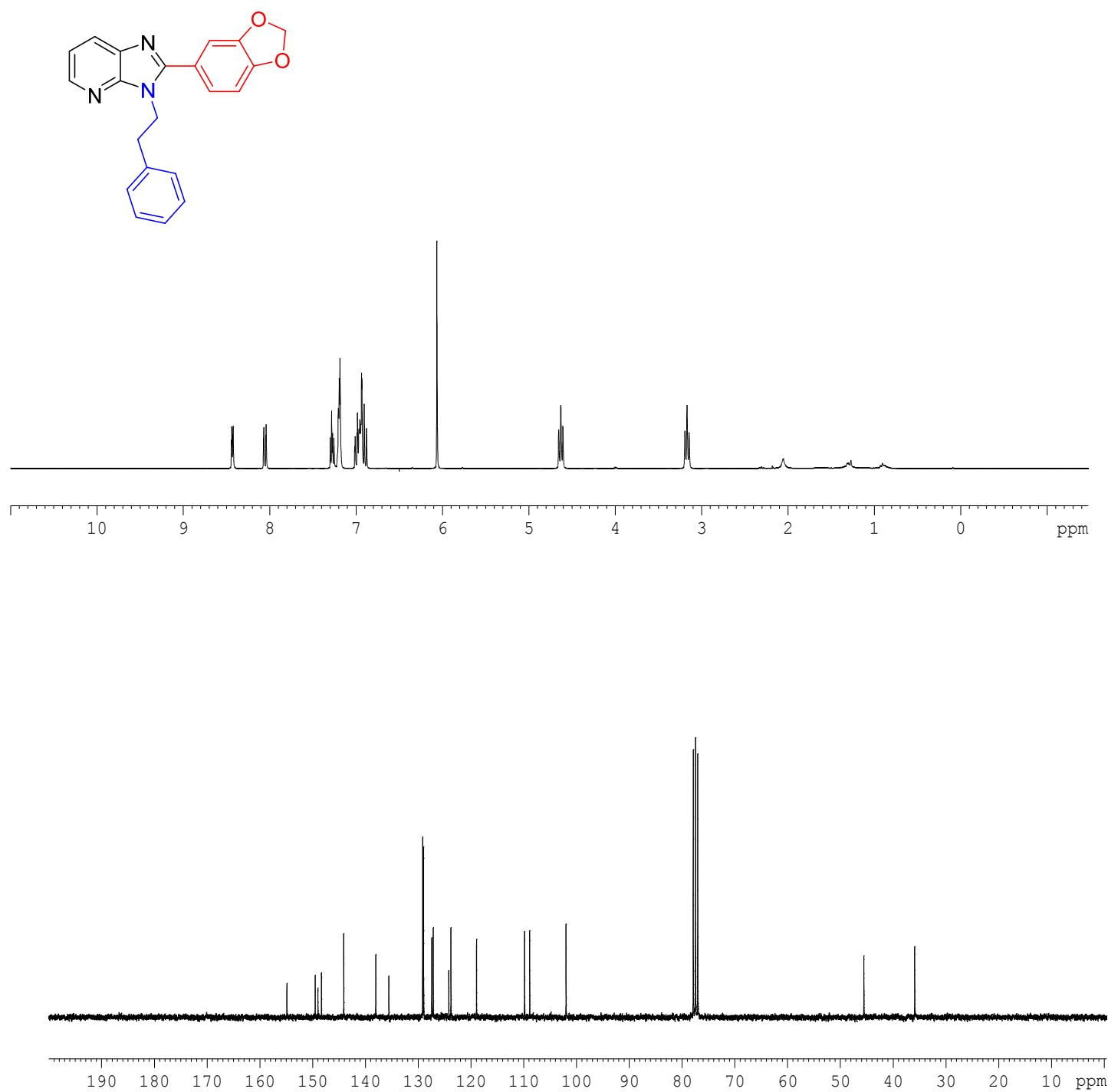


Figure S31: ^1H and ^{13}C NMR spectrum of compound 4p in CDCl_3

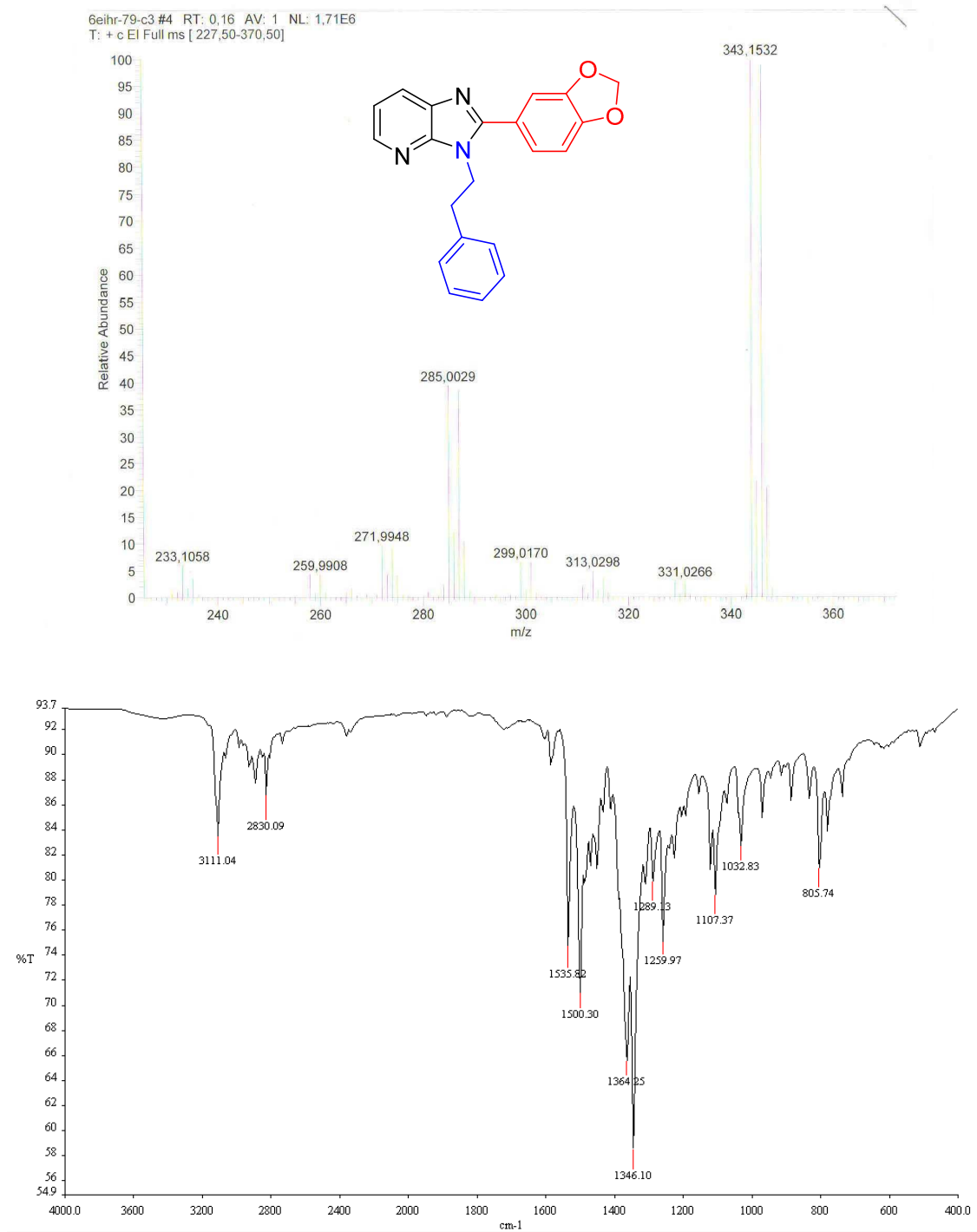


Figure S32: HR Mass and IR spectra of compound 4p

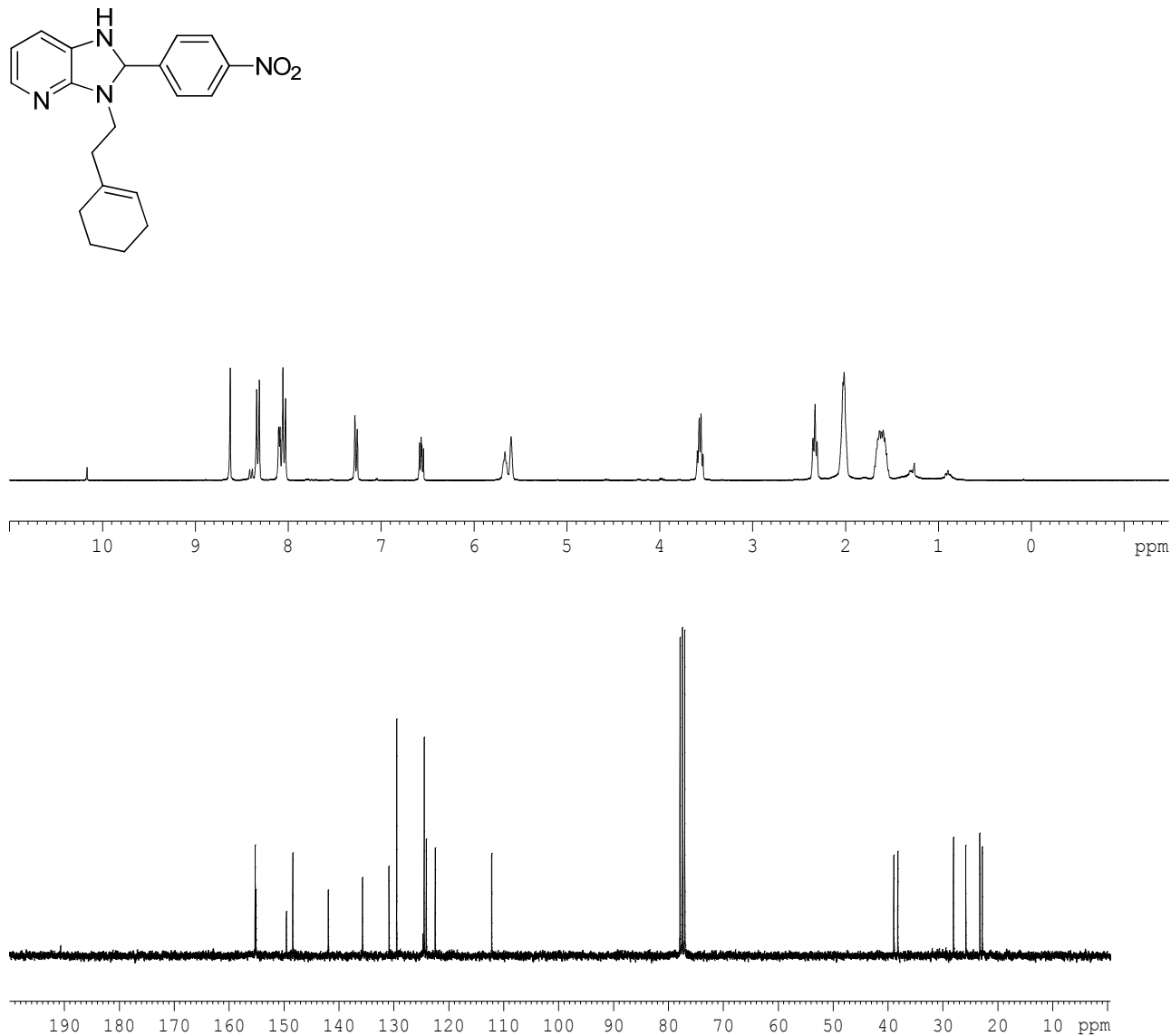


Figure S33: ^1H and ^{13}C NMR spectrum of intermediate X in CDCl_3

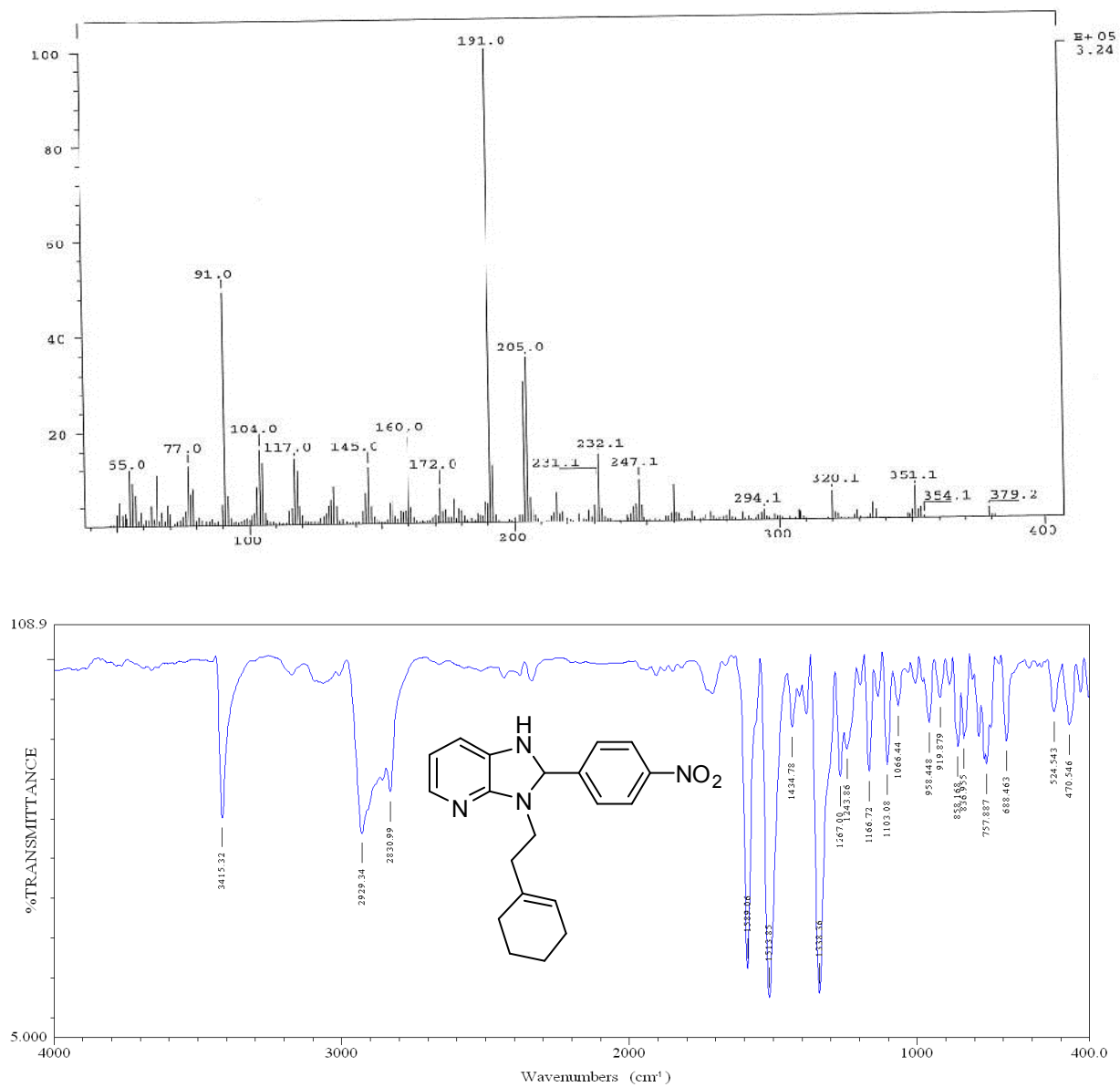
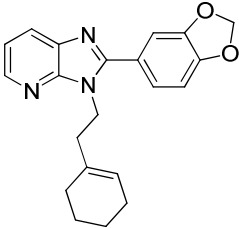
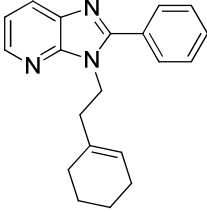
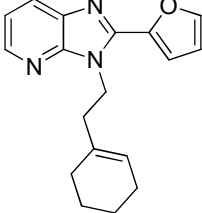
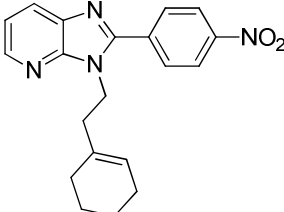
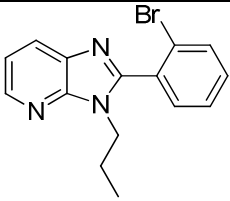
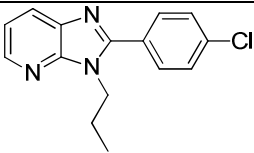
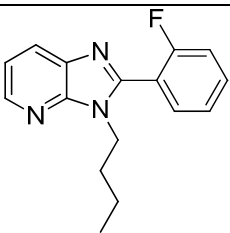
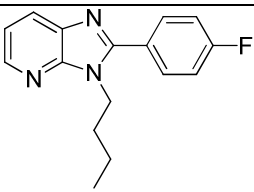
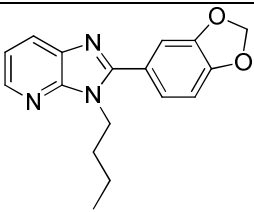
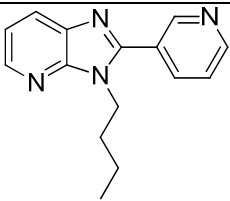
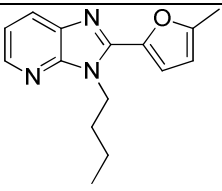


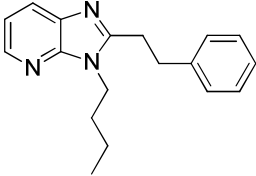
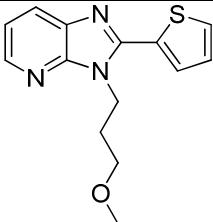
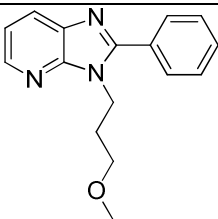
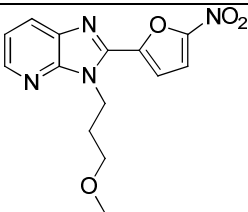
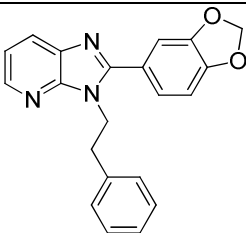
Figure S34: Mass and IR spectrum of intermediate X

Anti-bacterial activity – EC₅₀ values: Overnight culture of 3 different bacteria (E.coli, Staphylococcus and Klebsiella) were used at a concentration of 0.05 OD600 for the microbial inhibition reaction. Various concentration of samples dissolved in water were used and the cells were incubated at 37 °C in a shaker incubator for about 8 hours and the inhibition was monitored by the decrease in the cell density at OD600.

Table 1 for antibacterial assay

Entry	Compound	MIC ^a (μM)		
		<i>Staphylococcus</i>	<i>E.coli</i>	<i>Klebsiella</i>
4a		118	84	80
4b		65	82	62
4c		67	76	51
4d		66	89	54

4e		122	99	85
4f		>200	133	>200
4g		120	95	71
4h		125	89	99
4i		122	90	83
4j		98	244	>200
4k		>200	>200	>200

4l		127	128	106
4m		91	105	46
4n		132	>200	101
4o		>200	>200	>200
4p		>200	>200	>200

^aMIC; Minimum inhibitory concentration (μ M)