

Liquid chromatography and mass spectrometric studies of gilteritinib fumarate and characterization of its major degradation product by NMR

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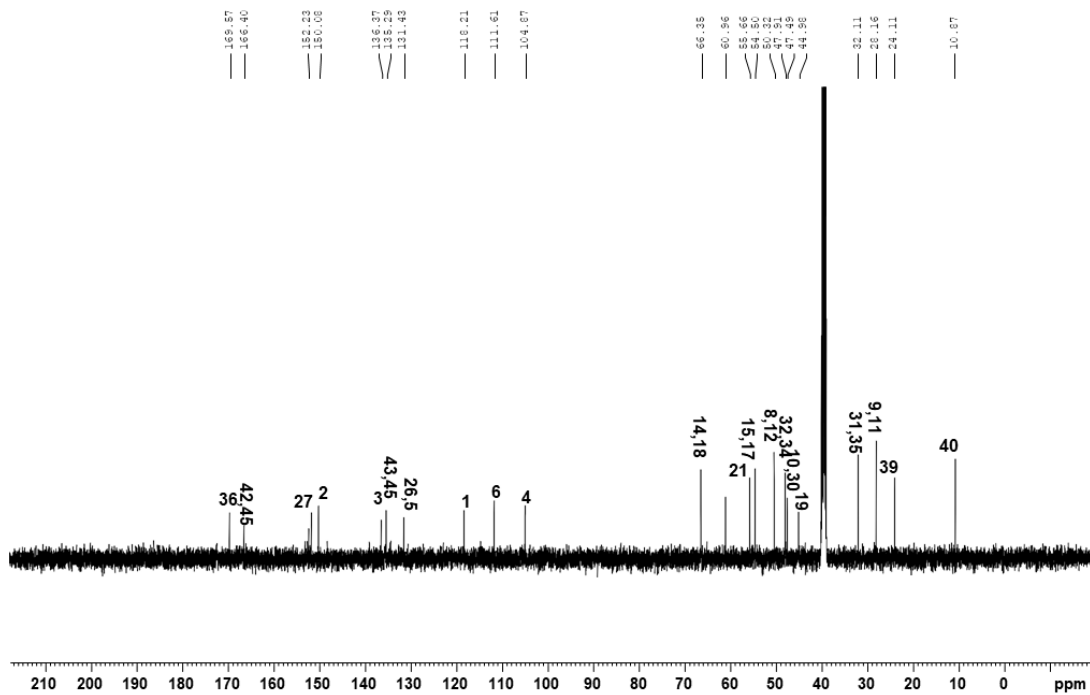


Figure S1: ^{13}C spectrum of GTB

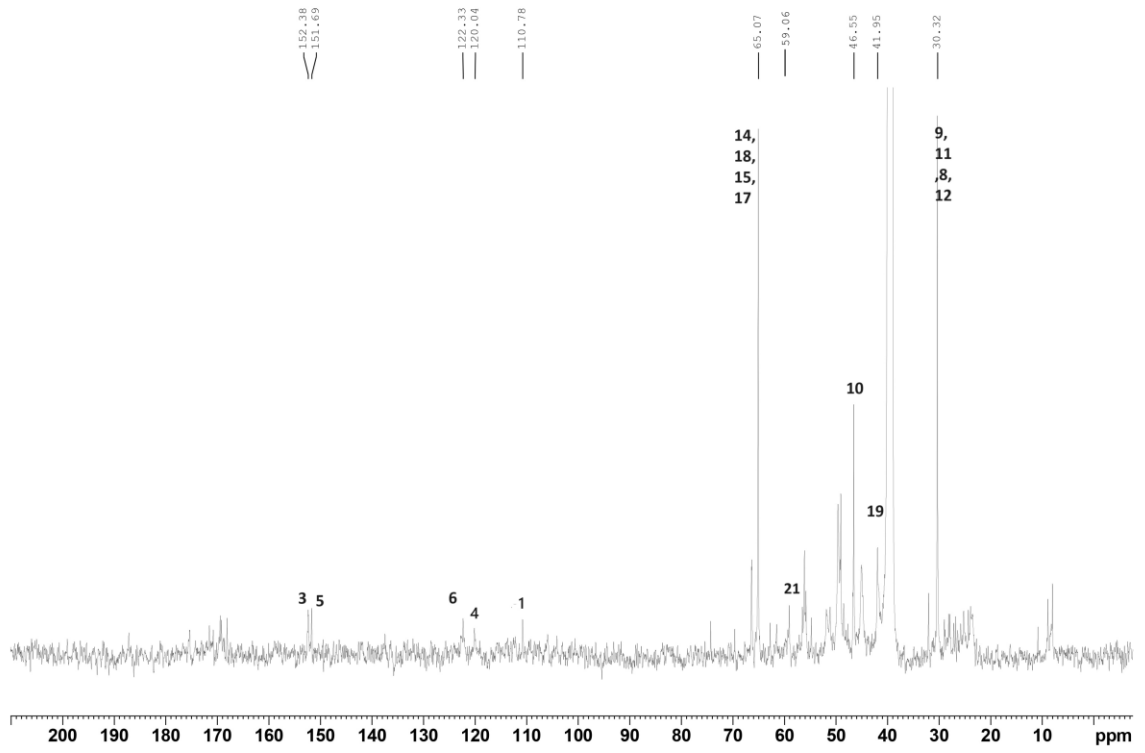


Figure S2: ^{13}C spectrum of DP-1

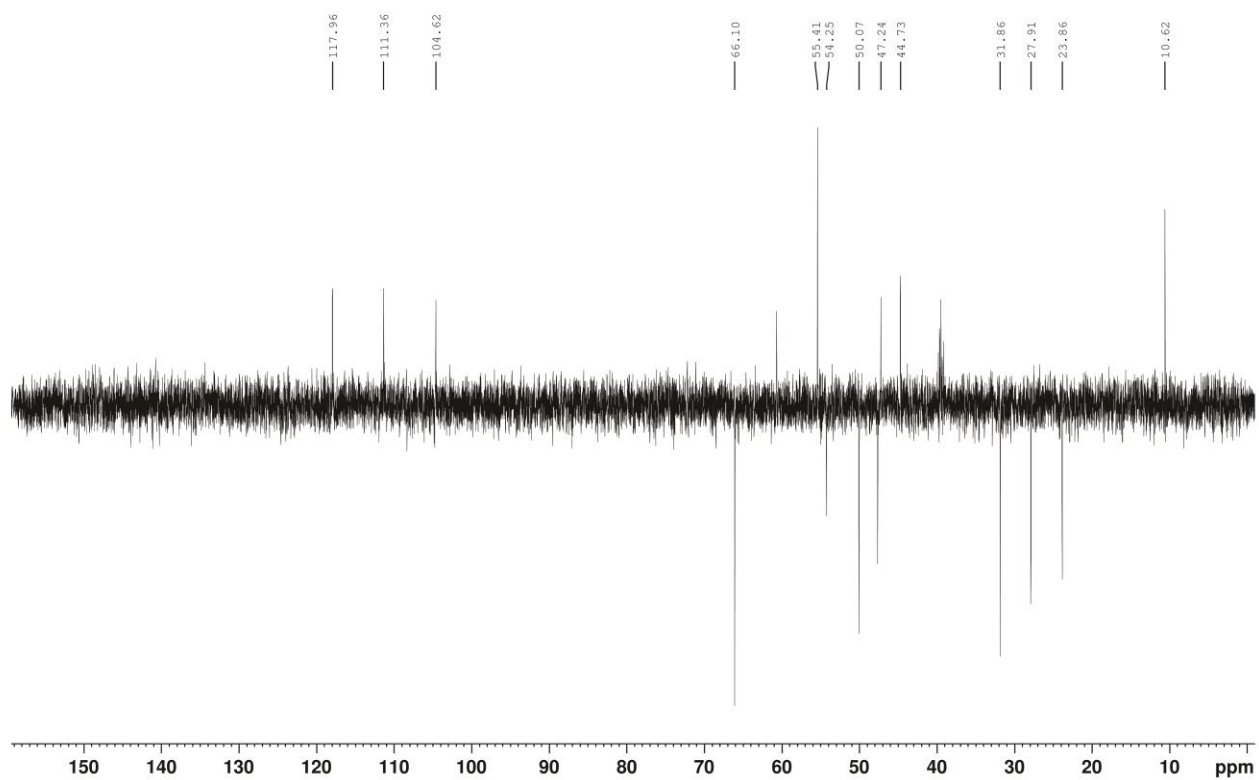


Figure S3: DEPT-135 spectrum of GTB

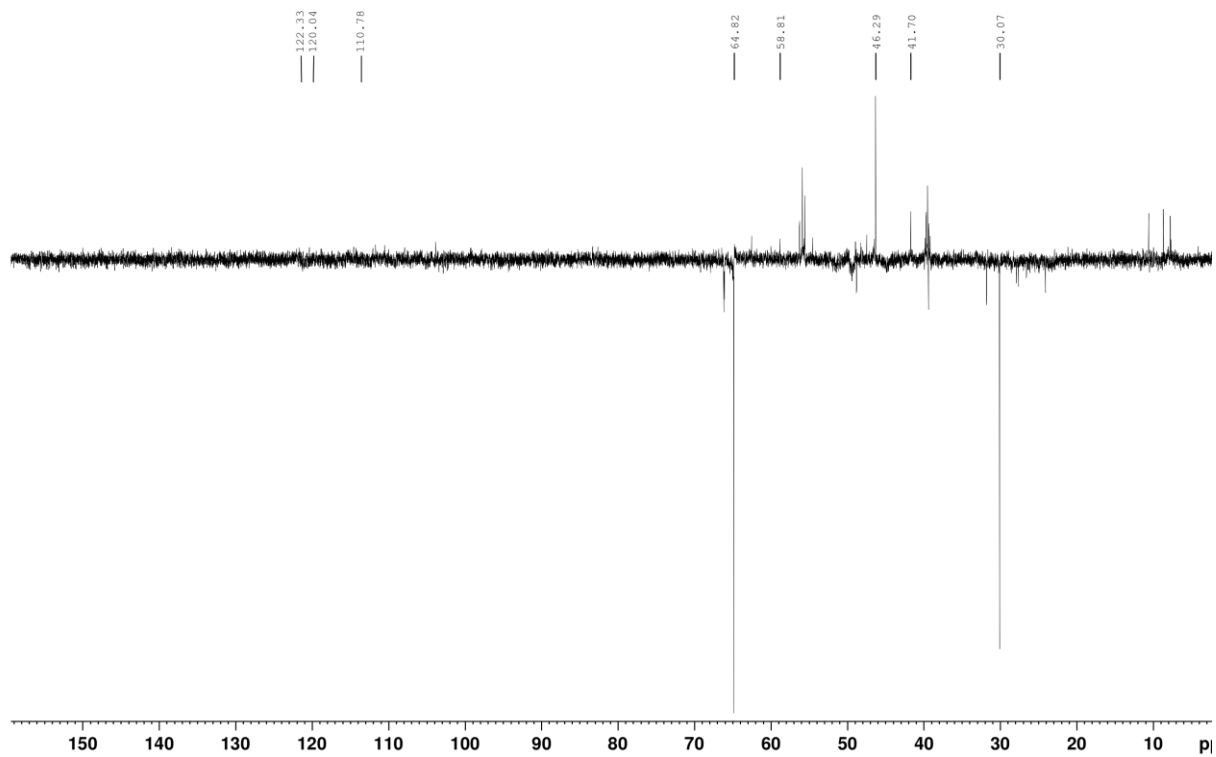


Figure S4: DEPT-135 spectrum of DP-1

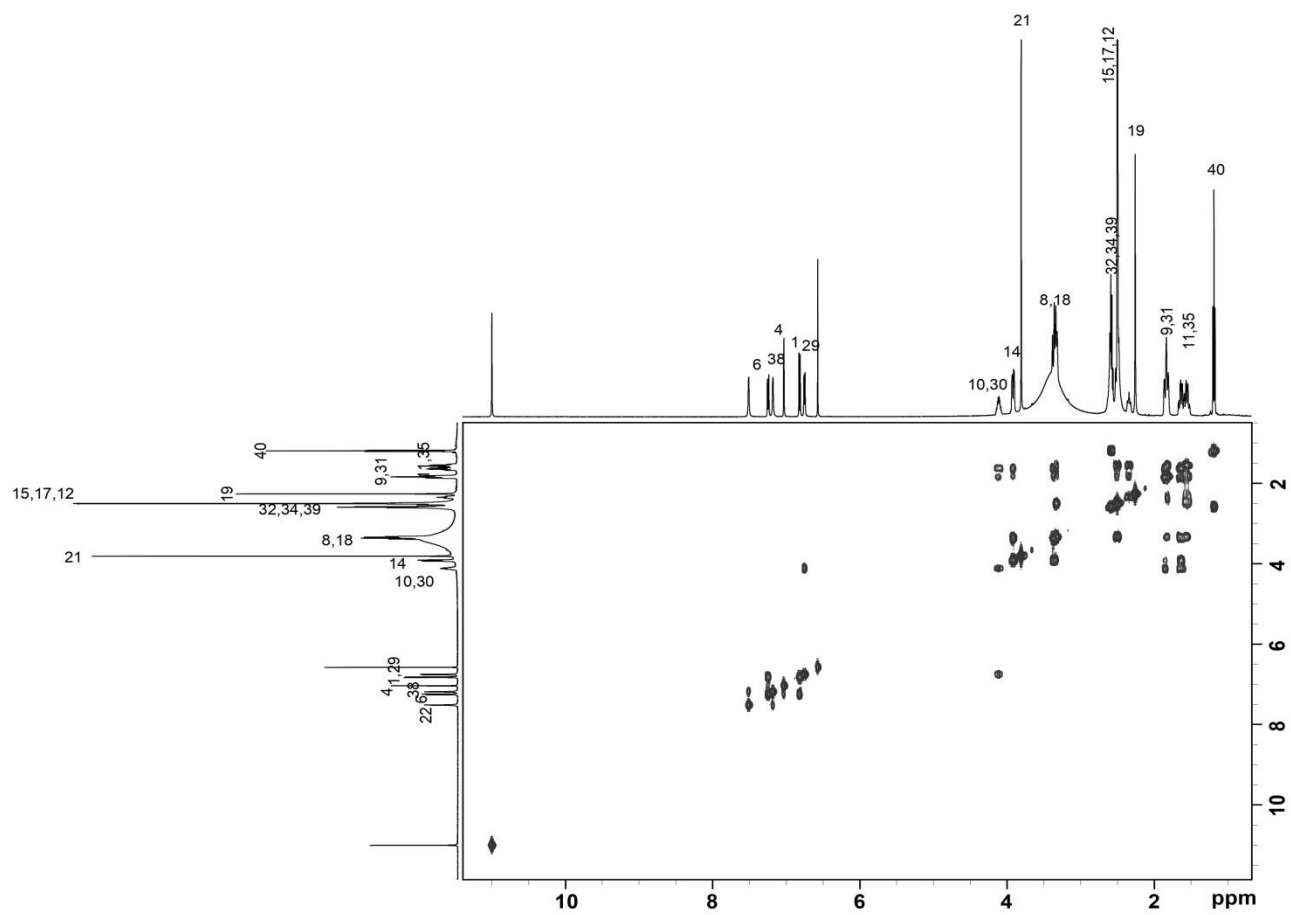


Figure S5: ^1H - ^1H COSY spectrum of GTB

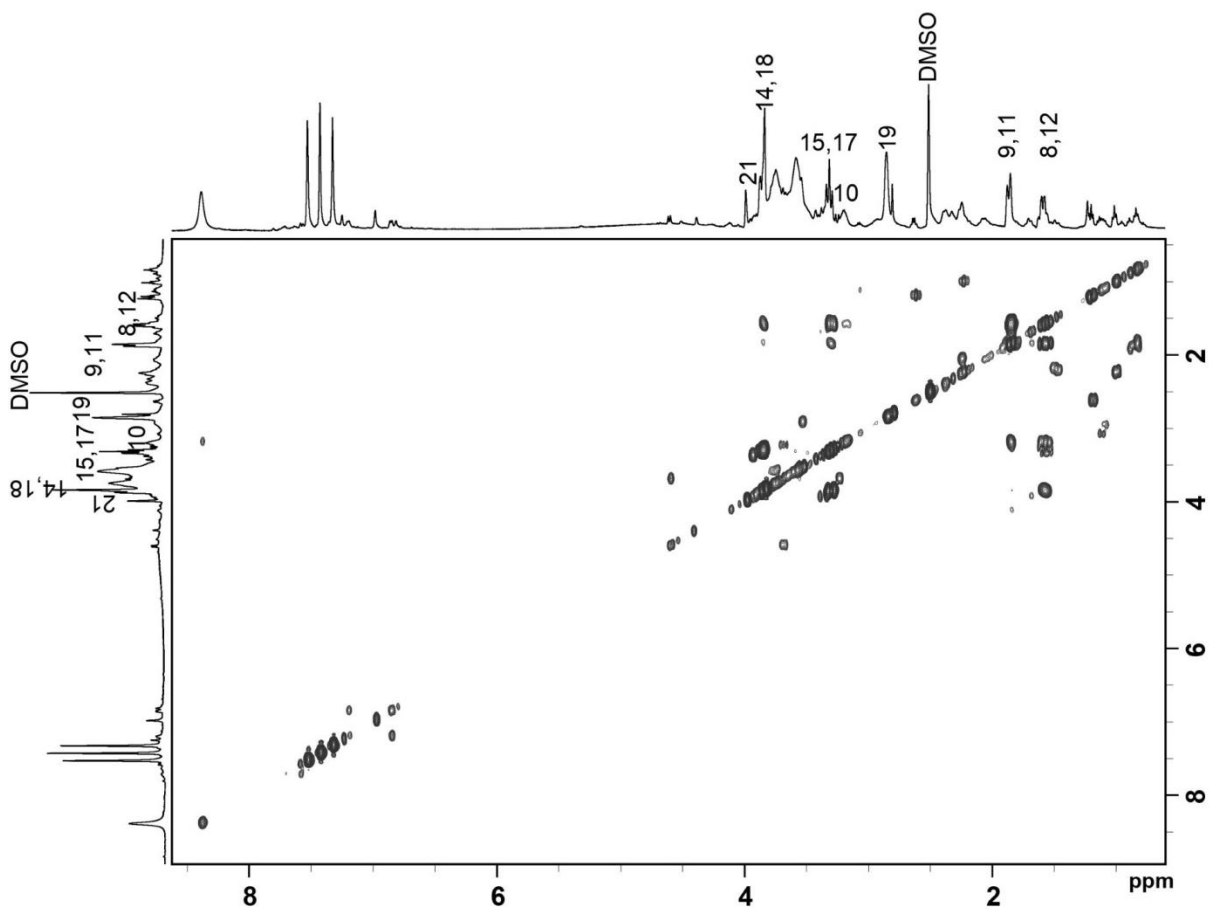


Figure S6: ^1H - ^1H COSY spectrum of DP-1

Table S1: Accurate mass data of GTB, its degradation products and their daughter ions

Name	Theoretical m/z	Experimental m/z	Error (in ppm)
GTB	553.3609	553.3618	-1.6
	536.3344	536.3367	-4.3
	453.2609	453.2602	1.5
	436.2343	436.2343	0.0
	382.1874	382.187	1.0
	367.1765	367.1737	7.6
	325.1659	325.1629	9.2
DP 1	305.2336	305.2326	3.3
	205.1336	205.1325	5.4
	151.0866	151.0867	-0.7
	190.1101	190.1085	8.4
	177.1023	177.1018	2.8
DP2	427.2816	427.2818	-0.5
	327.1816	327.181	1.8
	312.1581	312.154	13.1
	273.1347	273.1355	-2.9
	299.1503	299.1522	-6.4
	258.1117	258.1172	-21.3
DP 3	470.2874	470.2885	-2.3
	370.1874	370.1878	-1.1
	353.1609	353.1609	0.0
	299.1139	299.1133	2.0
	341.1973	341.1951	6.4
	284.0904	284.1007	-36.3
DP 4	569.3558	569.3569	-1.9
	552.3531	552.3513	3.3
	452.2531	452.2499	7.1
	437.2296	437.2308	-2.7
	508.3031	508.2988	8.5
	425.2296	425.2251	10.6
	369.167	369.1529	38.2
	204.1132	204.099	69.6
	585.3507	585.3502	0.9
DP 5	550.3375	550.3329	8.4
	451.2452	451.2451	0.2
	436.2344	436.2343	0.2
	425.2296	425.2293	0.7
	408.2031	408.2015	3.9
	382.1874	382.187	1.0

Table S2: ^1H NMR Data of GTB

Chemical shifts(ppm)	Group	Multiplicity
11.01	OH(FA)	s
7.46	NH(22)	s
7.19	CH(4)	s
7.14	NH ₂	d
6.96	CH(6)	d
6.75	CH(1)	d
6.7	NH	s
6.50	H(FA)	s
4.05	CH(10,30)	m
3.86	CH ₂ (14)	d
3.70	CH ₃ (21)	s
3.32	CH ₂ (8,18)	d
2.51	CH ₂ (32,3439)	m
2.27	CH ₂ (12,15,17)	m
2.19	CH ₃ (19)	s
1.78	CH ₂ (9,31)	m
1.52	CH ₂ (11,35)	m
1.12	CH ₃ -40	t

Table S3: ^1H NMR Data of DP-1

Chemical shift	Group	Multiplicity
8.38	NH2(22)	s
7.25	CH(4)	s
7.20	CH(6)	d
7.01	CH(1)	s
4.01	CH3(21)	s
3.87	CH2(14,18)	m
3.32	CH2(15,17)	m
3.25	CH(10)	m
2.86	CH3(19)	s
1.92	CH2(9,12)	m
1.66	CH2(8,12)	m

Table S4: ^{13}C NMR and DEPT-135 of GTB

Carbon number	^{13}C(ppm)	DEPT-135
C-1	118.21	CH(positive)
C-6	111.61	CH(positive)
C-4	104.87	CH(positive)
C-14,C-18	66.35	CH ₂ (negative)
C-21	56.66	CH ₃ (positive)
C-15,C-17	54.60	CH ₂ (negative)
C-8,C-12	47.91	CH ₂ (negative)
C-32,C-34	47.49	CH ₂ (negative)
C-10,C-30	44.98	CH(positive)
C-19	40.02	CH ₃ (positive)
C-31,C-35	32.11	CH ₂ (negative)
C-9,C-11	26.16	CH ₂ (negative)
C-39	24.11	CH ₂ (negative)
C-40	10.87	CH ₃ (positive)

Table S5: ^{13}C NMR and DEPT-135 of DP-1

Carbon number	^{13}C	DEPT-135
C-1	110.78	CH (positive)
C-4	120.04	CH (positive)
C-6	122.33	CH (positive)
C-14,C-18,C-15,C-17	65.07	CH ₂ (negative)
C-21	59.06	CH ₃ (positive)
C-10	46.55	CH (positive)
C-19	41.95	CH ₃ (positive)
C-9,C-11,C-8,C-12	30.32	CH ₂ (negative)