A Preparation of (–)-Nutlin-3 Using Enantioselective Organocatalysis at Decagram Scale

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SI-I-X

Figure 1. ^{1}H NMR (400 MHz, $CDCl_3$) of **19.** 2
Figure 2. ^{13}C NMR (150 MHz, $CDCl_3$) of **19** 3
Figure 5. ^{1}H NMR (500 MHz, $CDCl_3$) of **21** 4
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Figure 1. ^{1}H NMR (400 MHz, $CDCl_3$) of **19.**

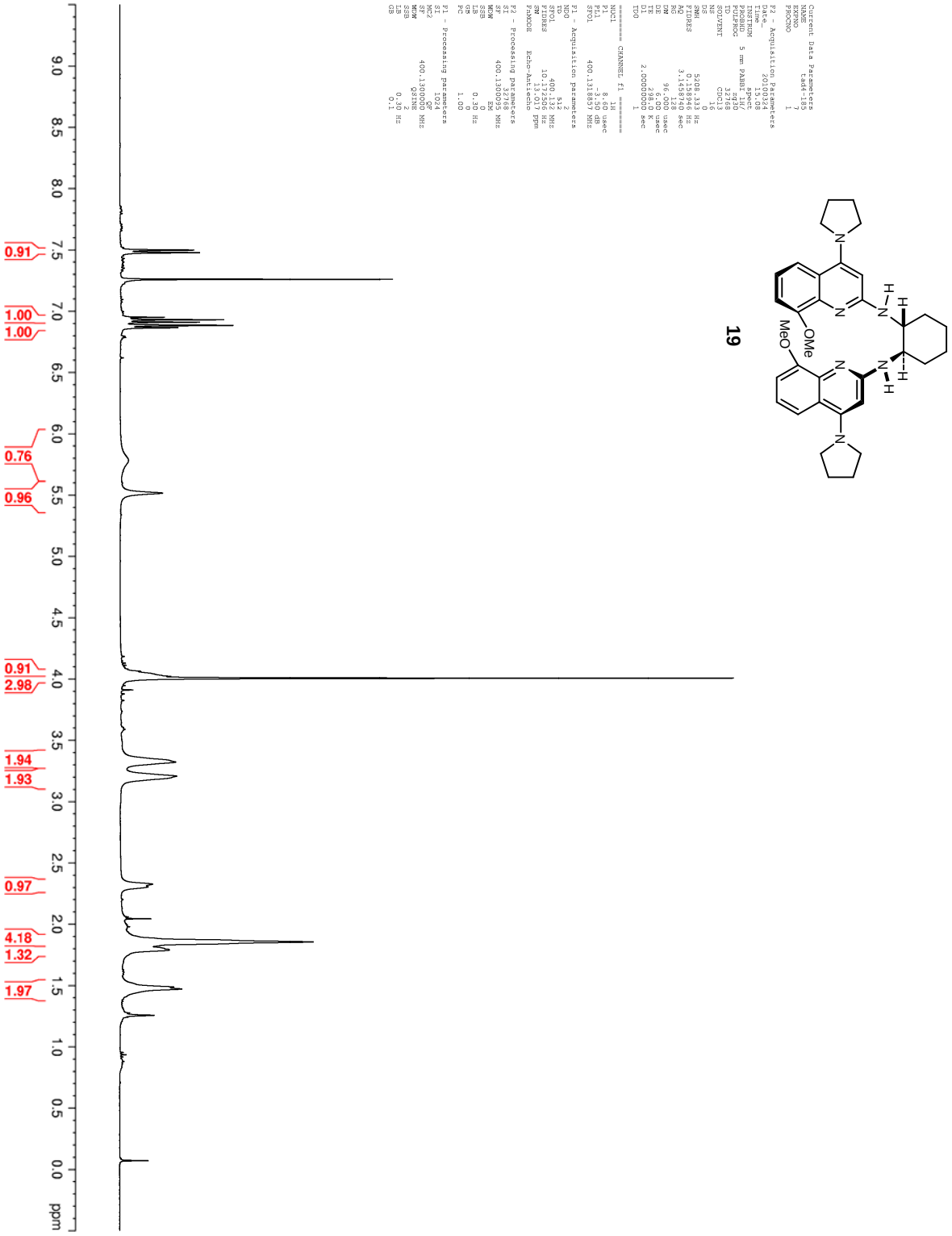


Figure 2. ^{13}C NMR (150 MHz, $CDCl_3$) of **19.**

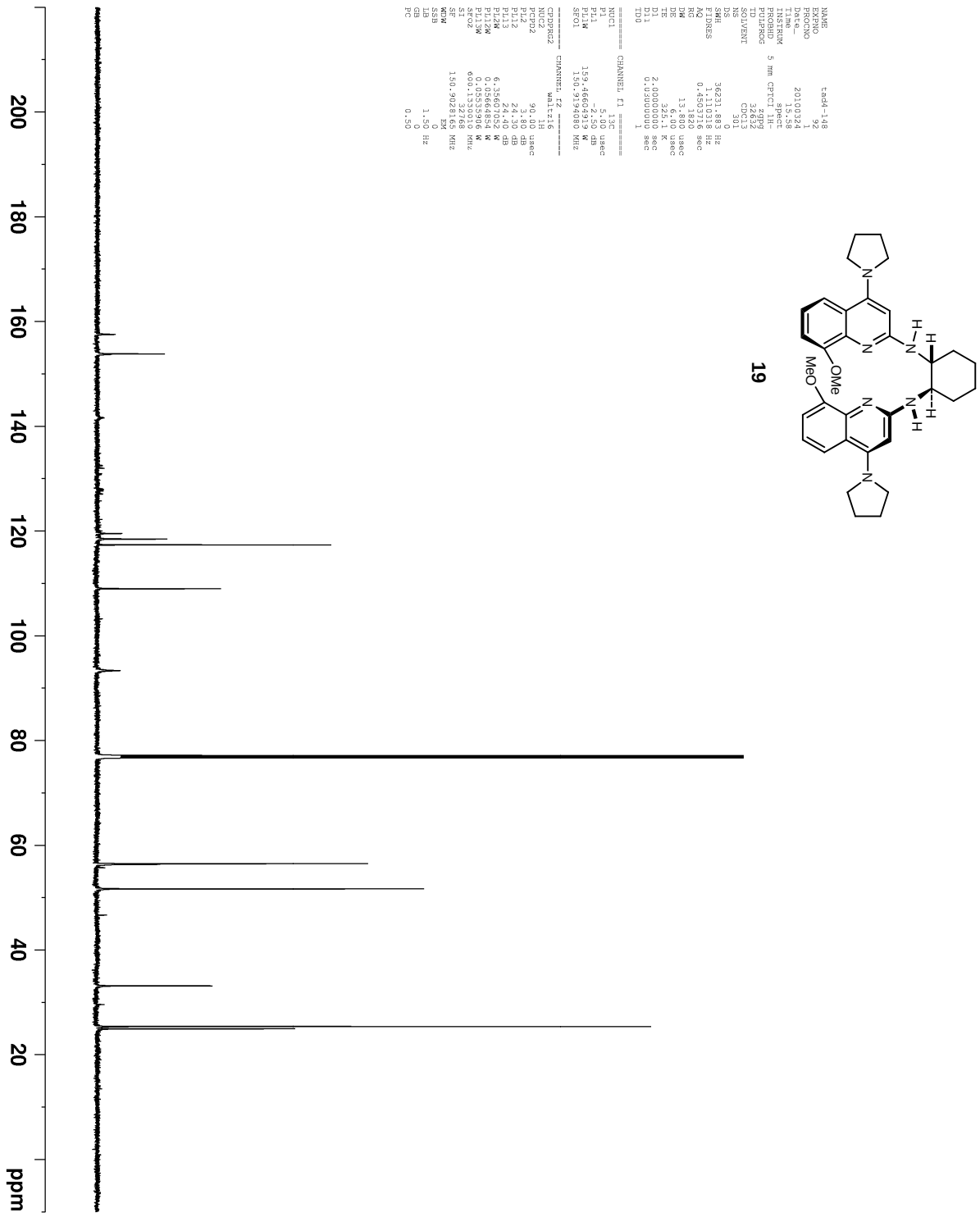


Figure 3. ^{1}H NMR (500 MHz, $CDCl_3$) of **21**

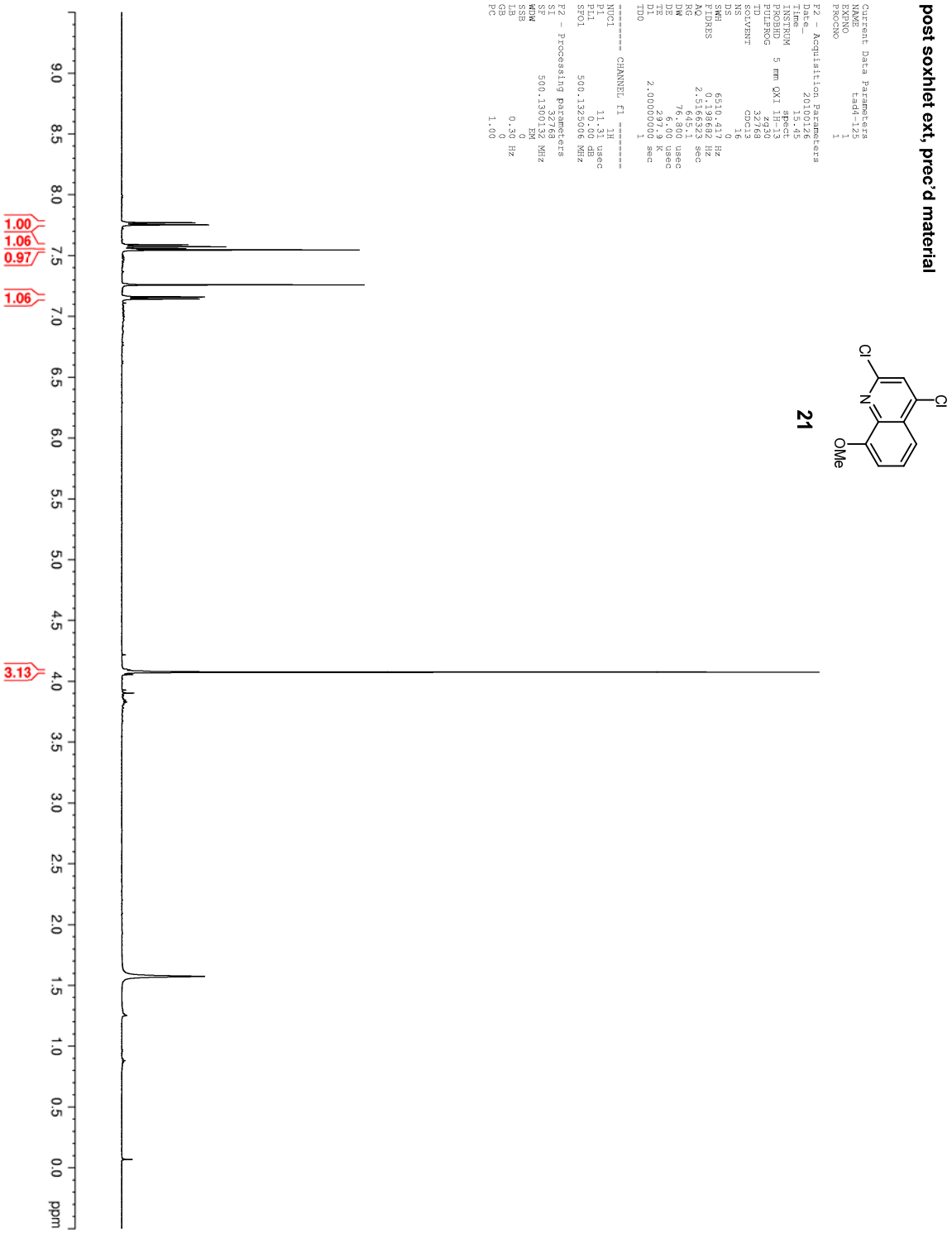


Figure 4. ^{13}C NMR (100 MHz, $CDCl_3$) of **21**

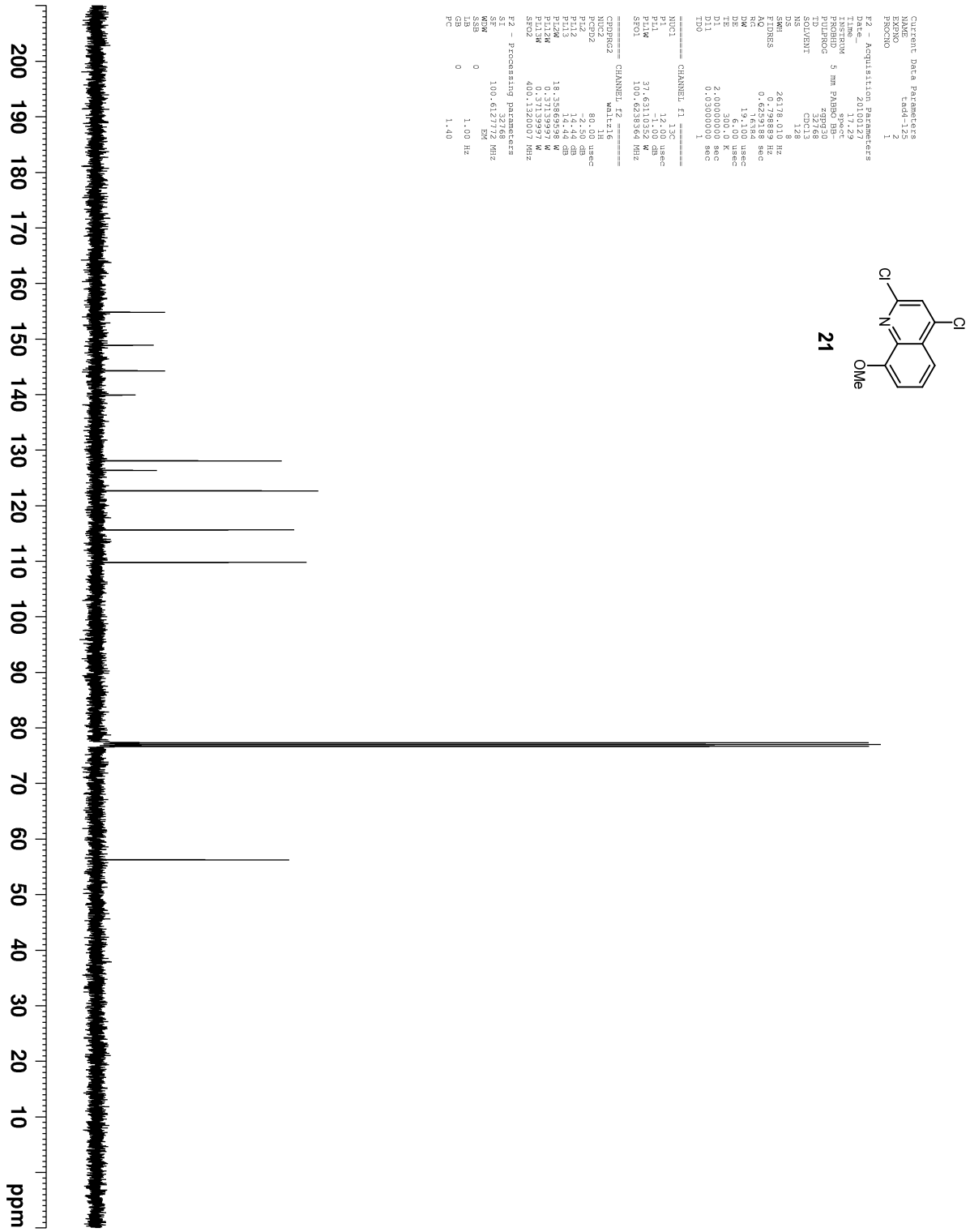


Figure 5. ^{1}H NMR (400 MHz, $CDCl_3$) of **22**

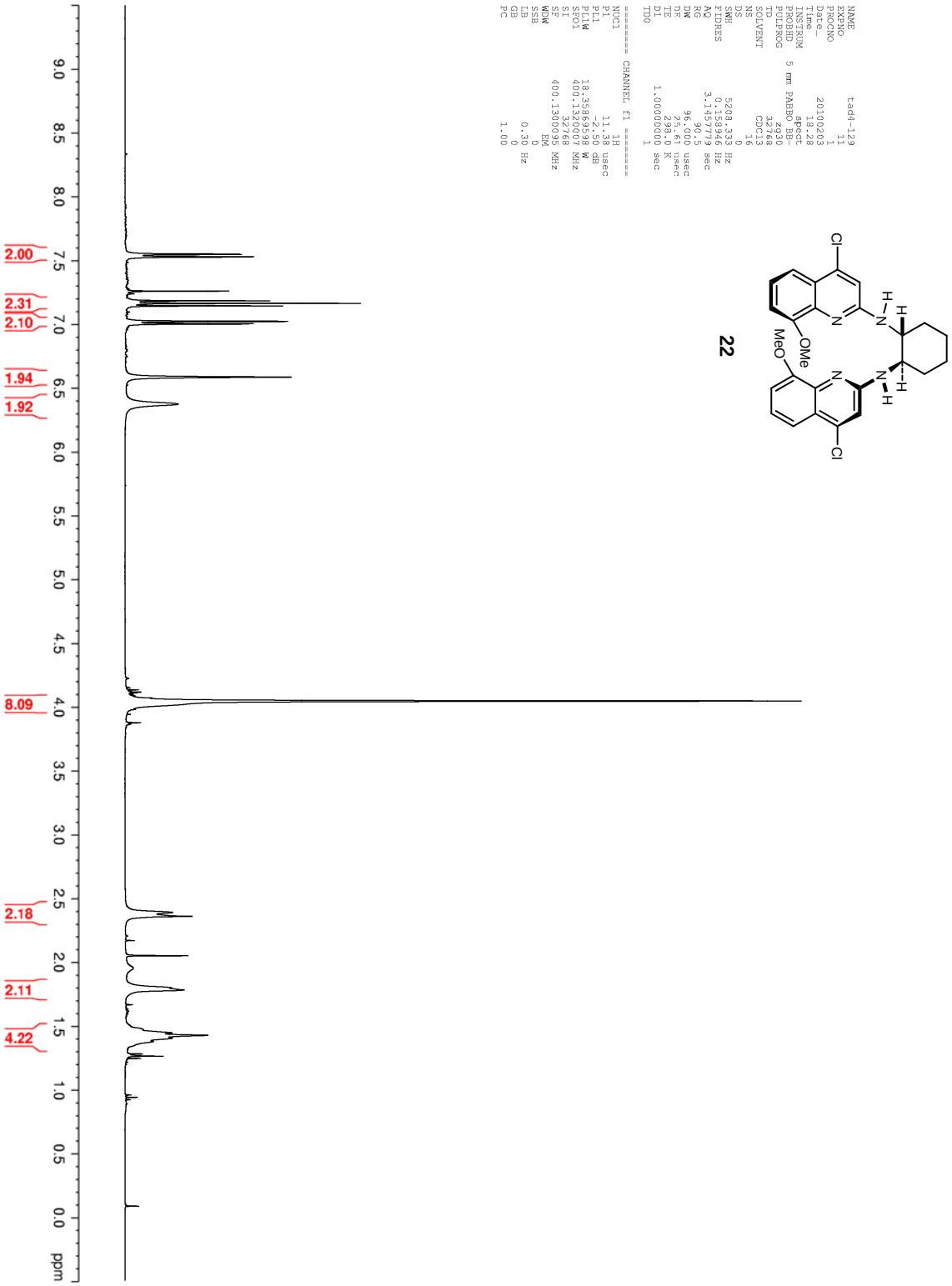


Figure 6. ^{13}C NMR (150 MHz, $CDCl_3$) of **22**

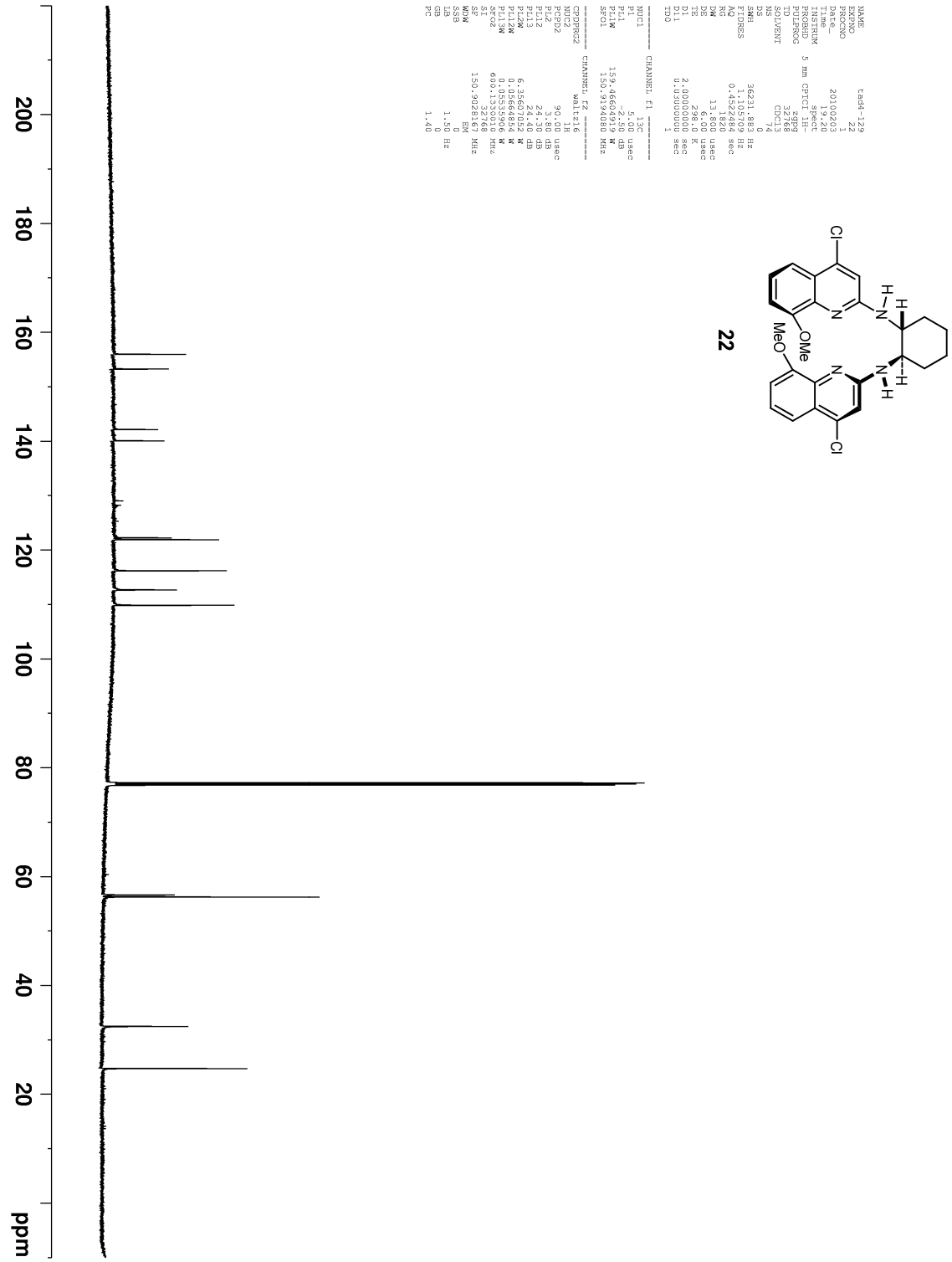


Figure 7. ^{1}H NMR (400 MHz, $CDCl_3$) of **23**

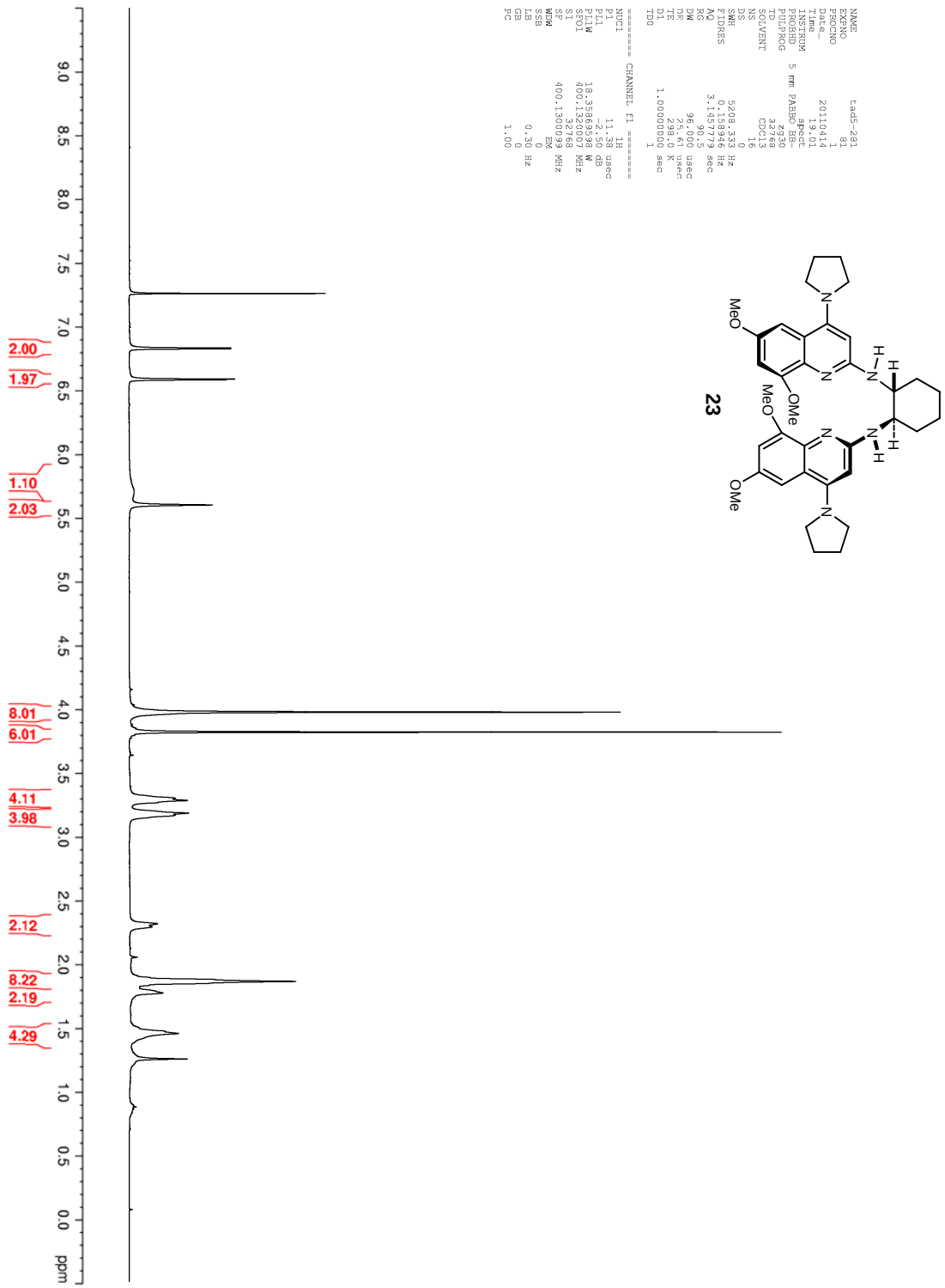


Figure 8. ^{13}C NMR (100 MHz, $CDCl_3$) of **23**

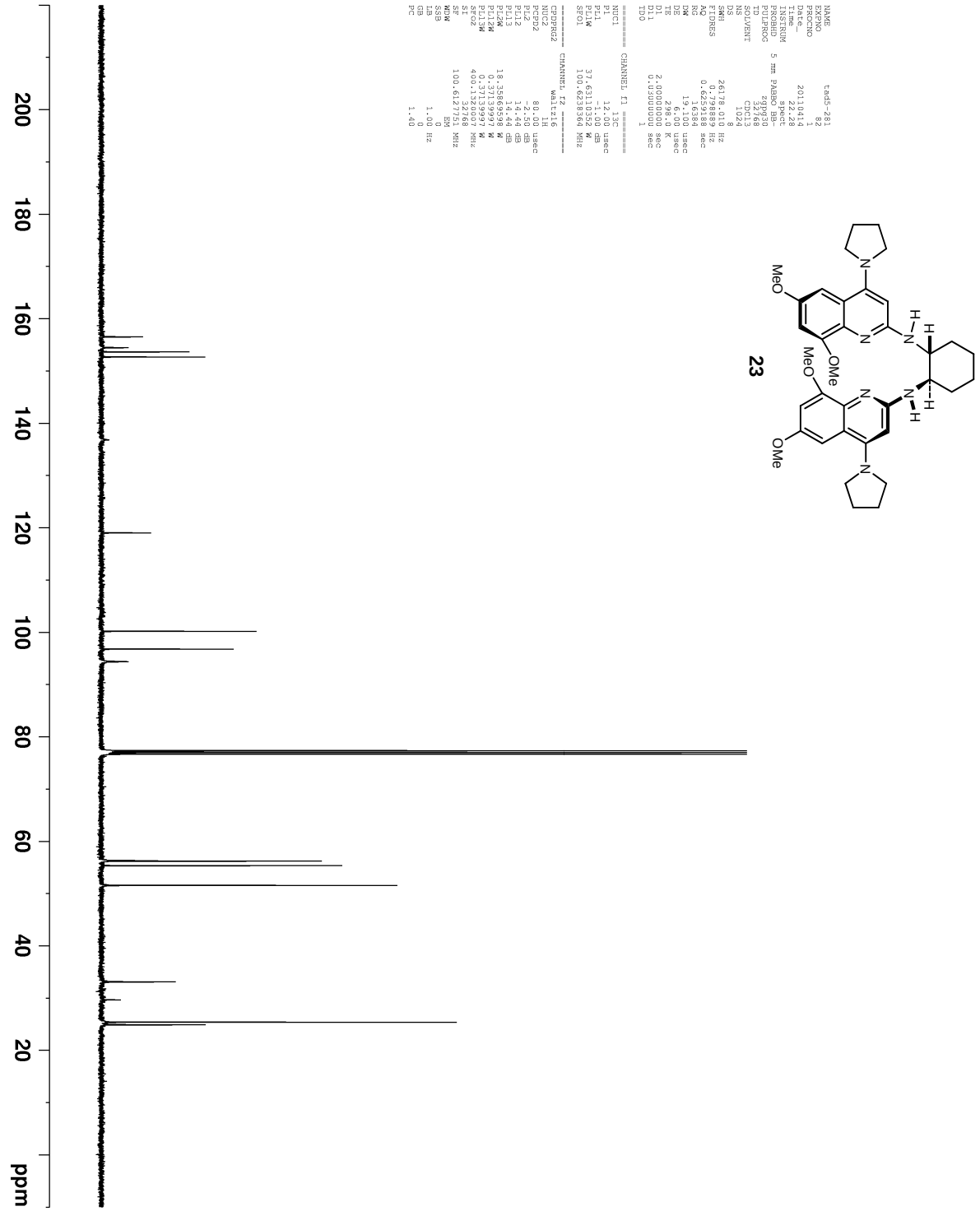


Figure 9. ^{1}H NMR (400 MHz, $CDCl_3$) of **S1**

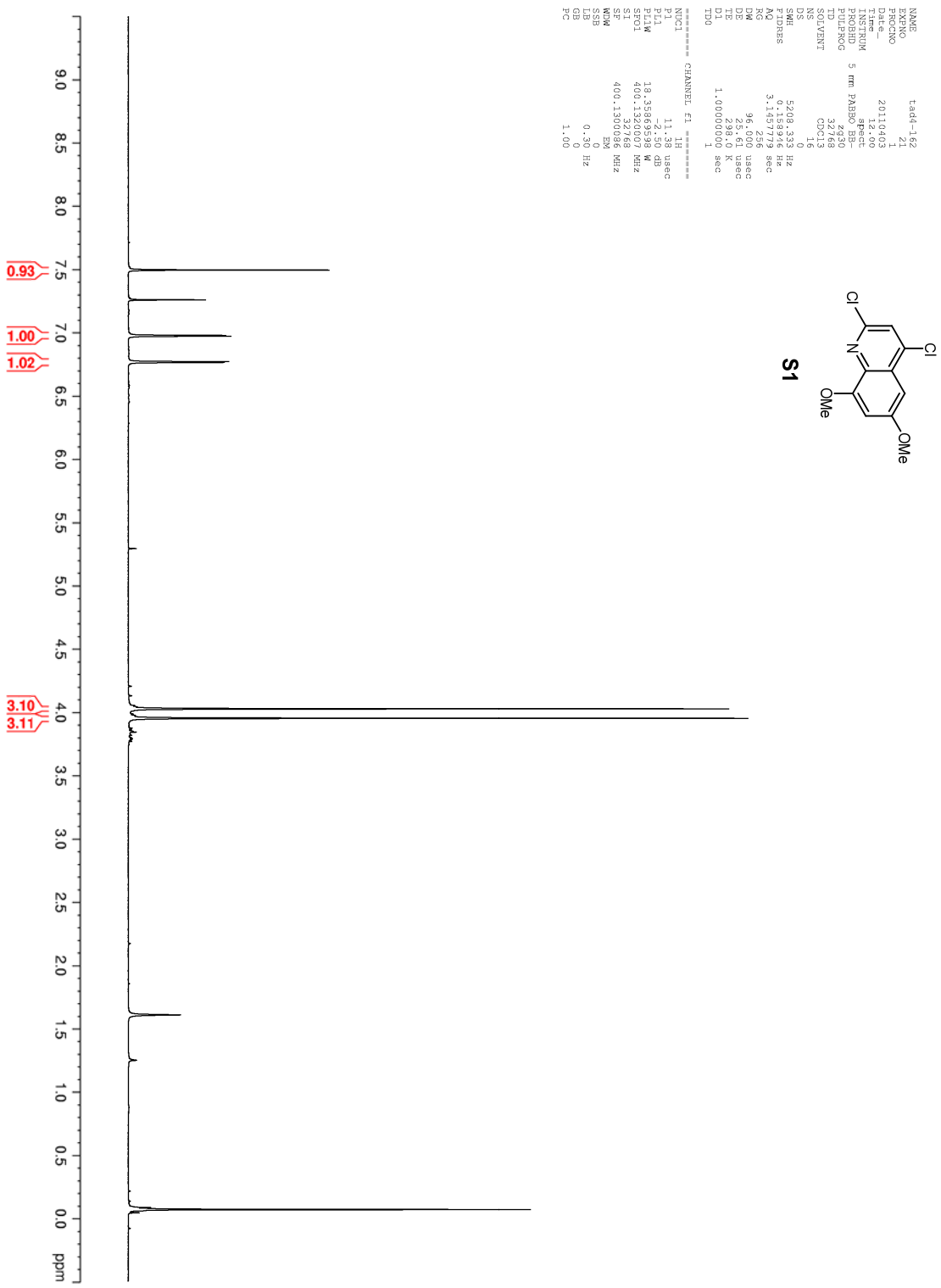


Figure 10. ^{13}C NMR (100 MHz, $CDCl_3$) of **S1**

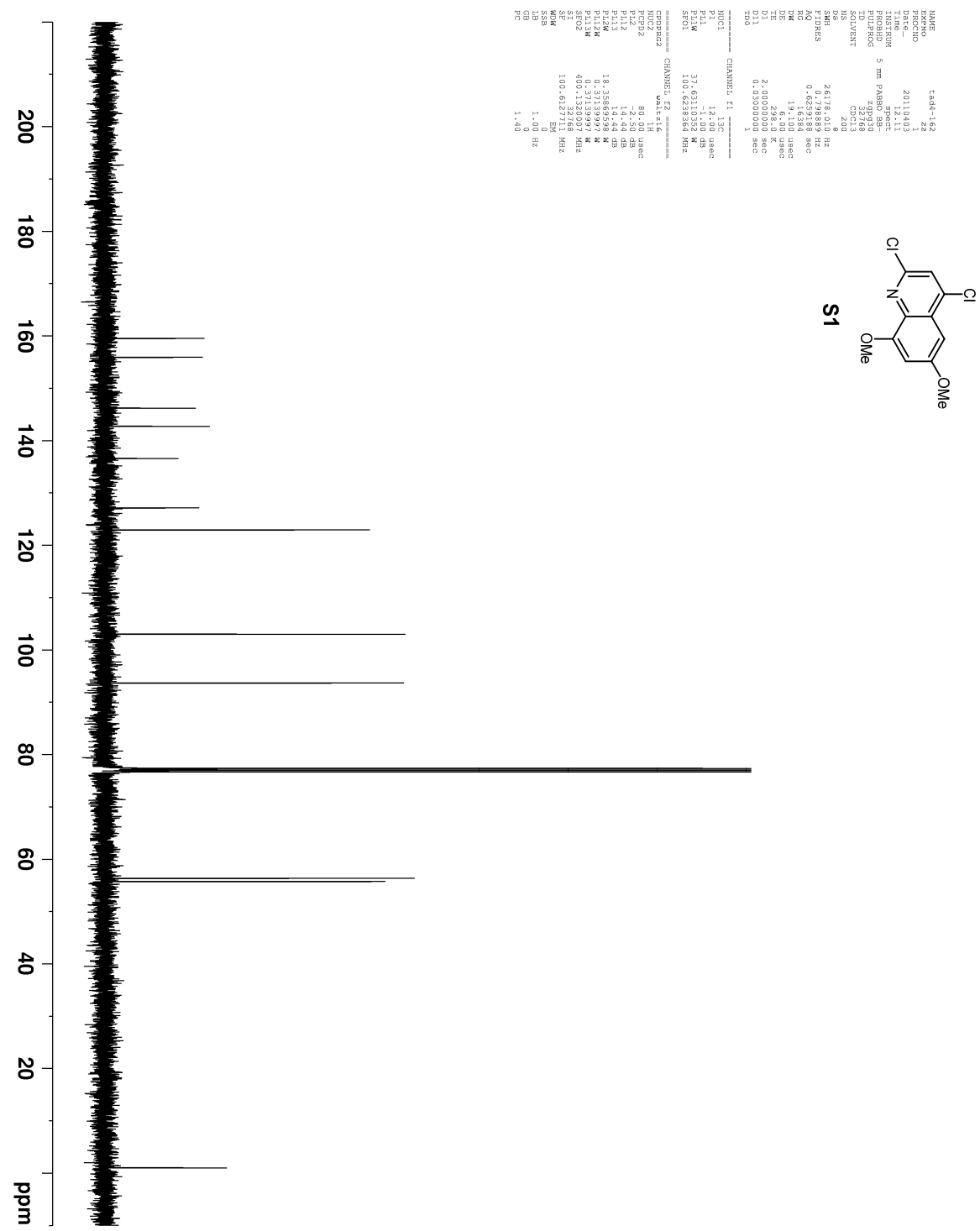


Figure 11. ^{1}H NMR (400 MHz, $CDCl_3$) of **S2**

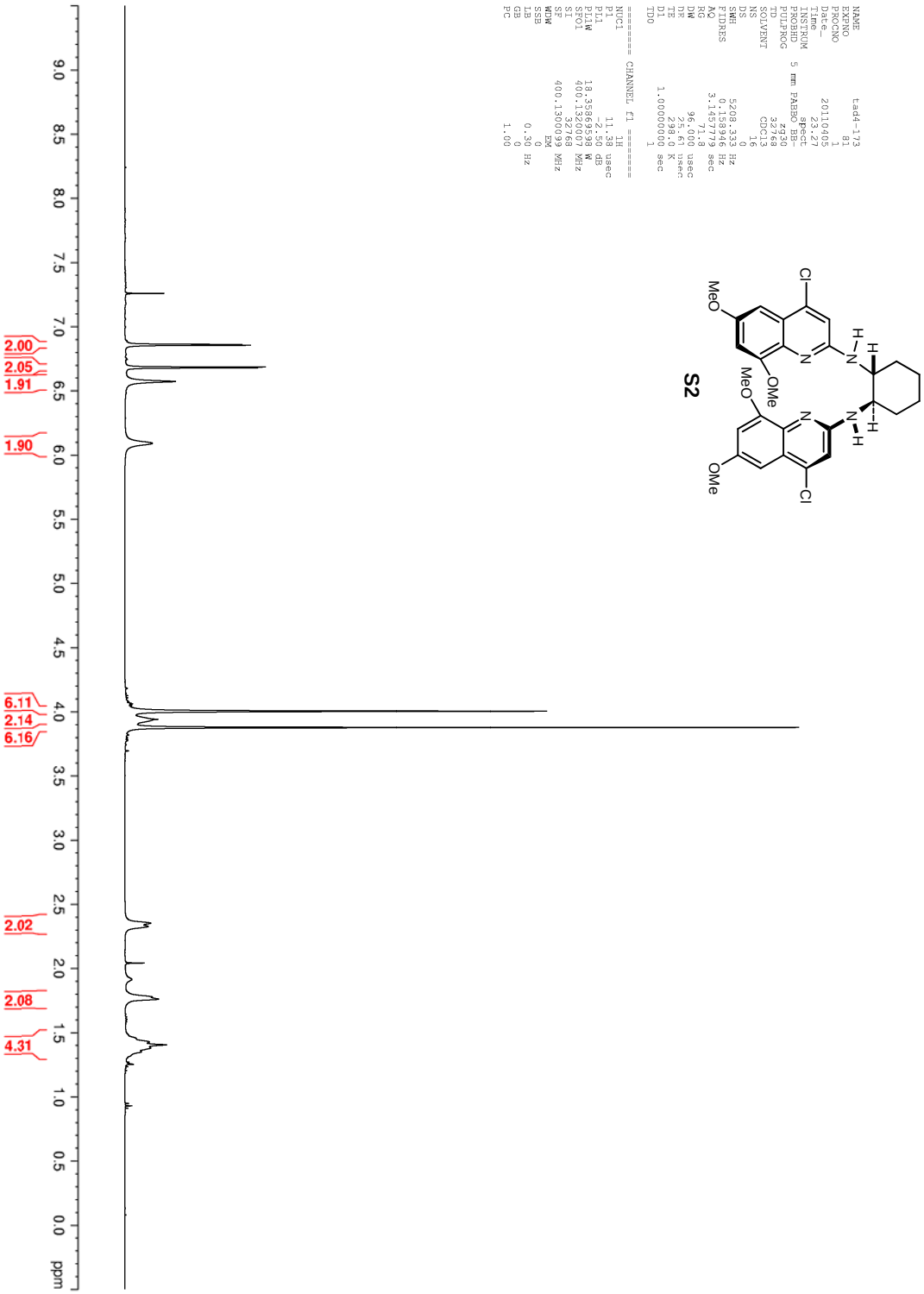


Figure 12. ^{13}C NMR (100 MHz, $CDCl_3$) of **S2**

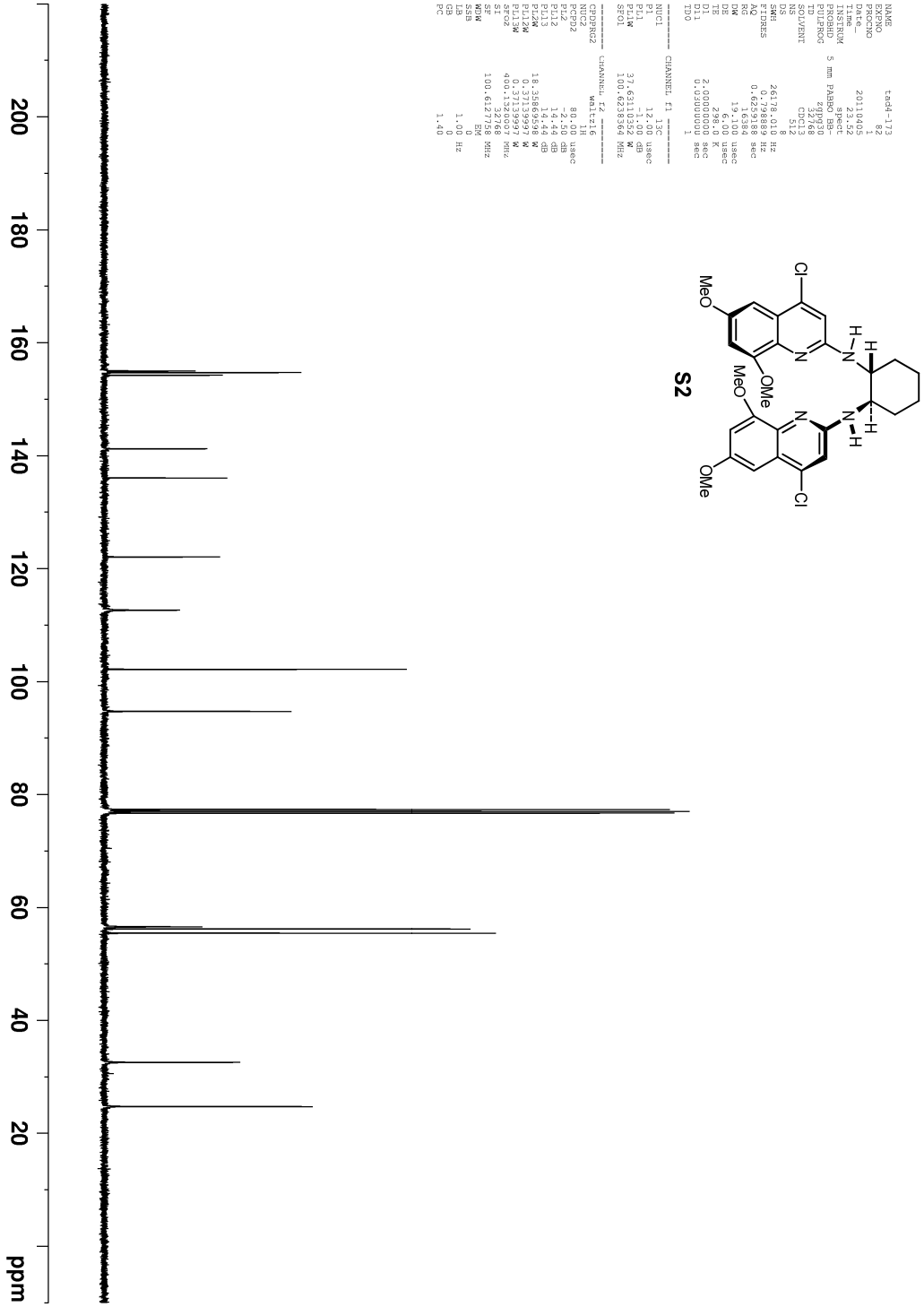
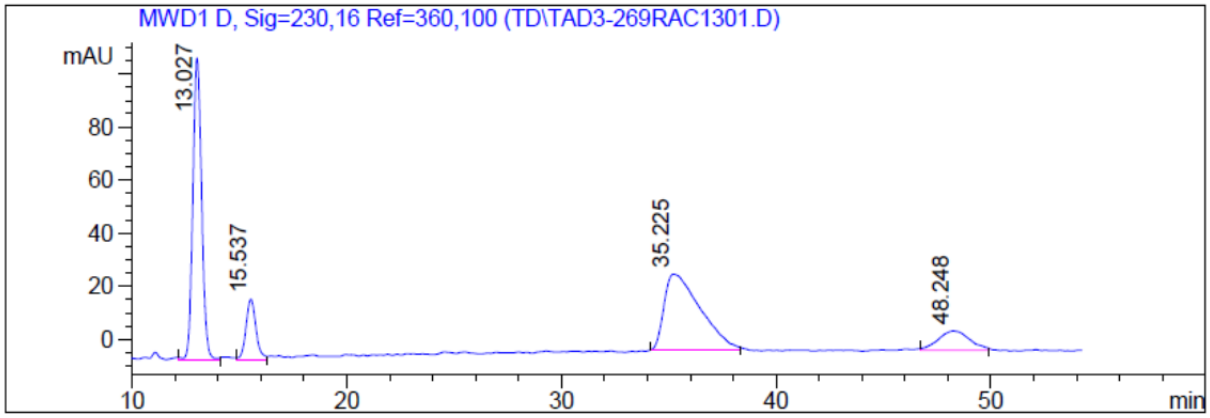


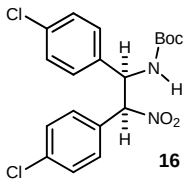
Figure 13. Representative HPLC traces for **16**

Racemate:



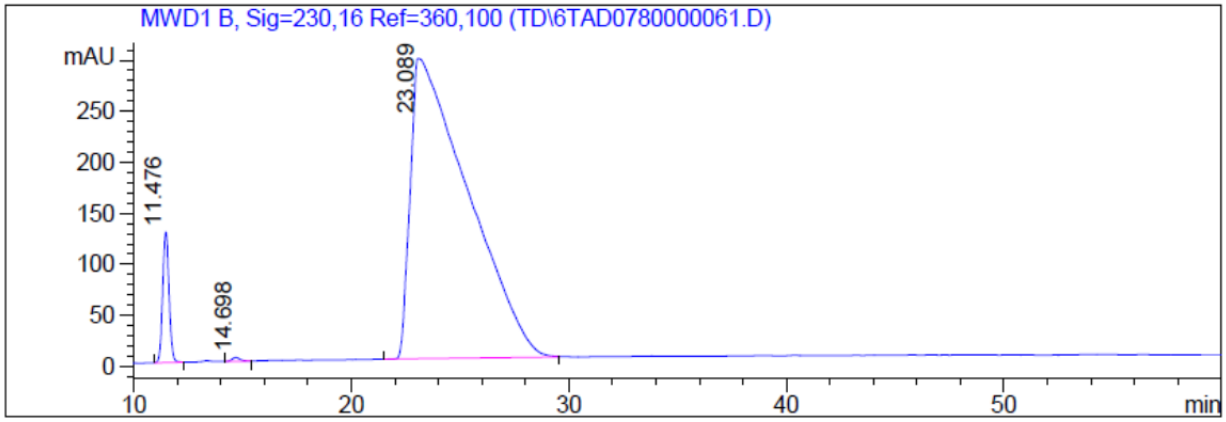
Signal 1: MWD1 D, Sig=230,16 Ref=360,100

Peak #	RT [min]	Width [min]	Area	Area %
1	13.027	0.474	3225.581	40.13
2	15.537	0.535	725.792	9.03
3	35.225	1.952	3345.620	41.63
4	48.248	1.698	739.845	9.21



16

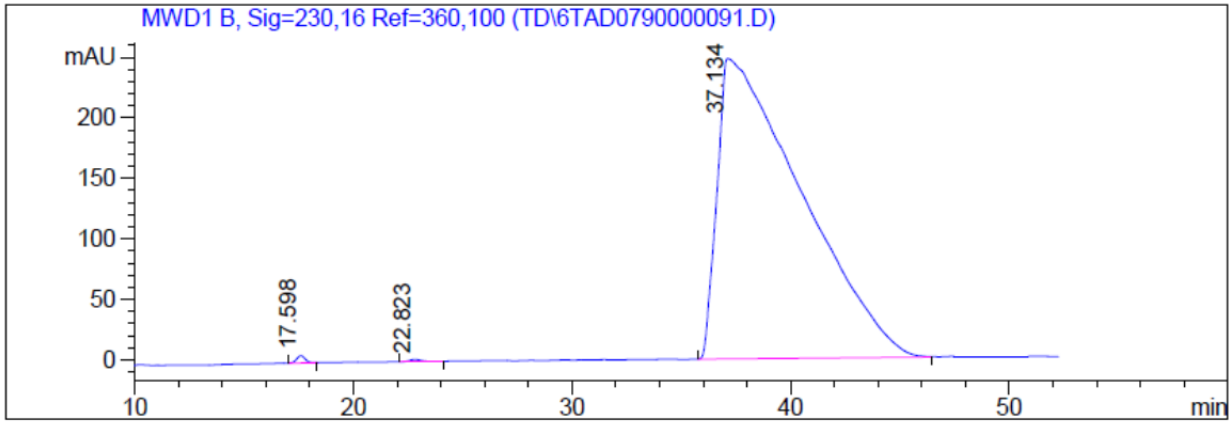
Enantioselective:



Signal 1: MWD1 B, Sig=230,16 Ref=360,100

Peak #	RT [min]	Width [min]	Area	Area %
1	11.476	0.314	2590.033	4.55
2	14.698	0.406	103.082	0.18
3	23.089	2.342	54242.629	95.27

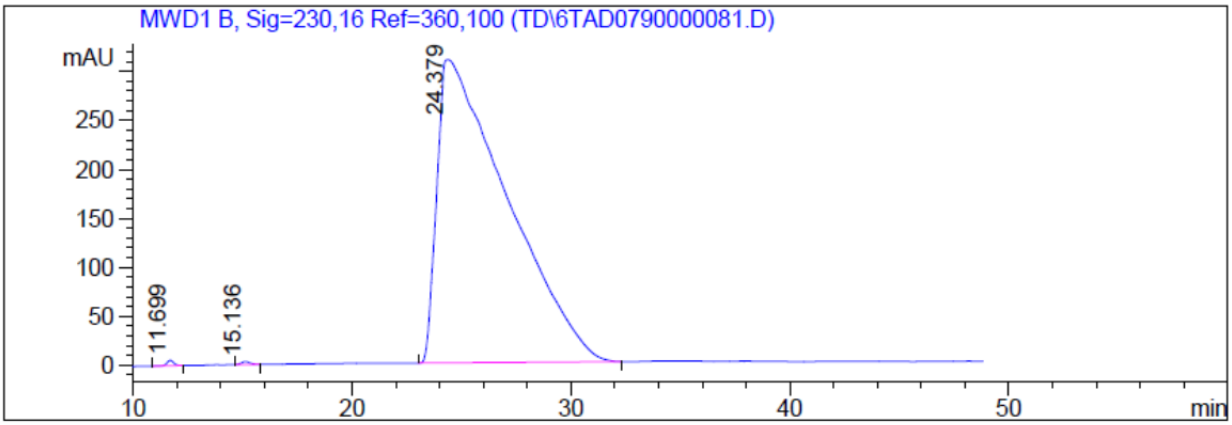
Recrystallized: Batch 1



Signal 1: MWD1 B, Sig=230,16 Ref=360,100

Peak #	RT [min]	Width [min]	Area	Area %
1	17.598	0.431	170.323	0.26
2	22.823	0.518	60.978	0.09
3	37.134	3.289	66417.211	99.65

Recrystallized: Batch 2



Signal 1: MWD1 B, Sig=230,16 Ref=360,100

Peak #	RT [min]	Width [min]	Area	Area %
1	11.699	0.323	115.710	0.17
2	15.136	0.417	88.944	0.13
3	24.379	2.782	69196.984	99.71