

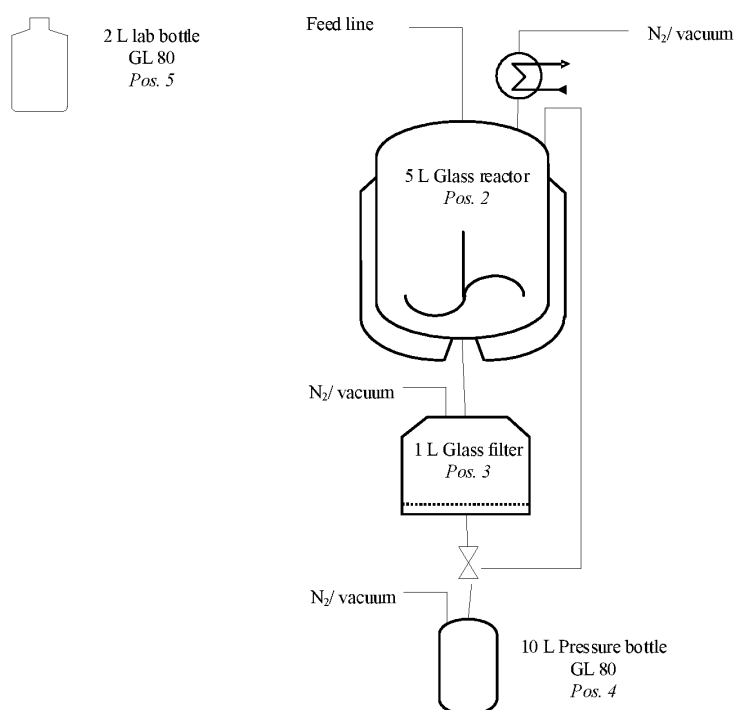
Step 1 - Acetoxyindole glyoxylic acid diethylamideAcid chloride formation

Figure 1 part 1.

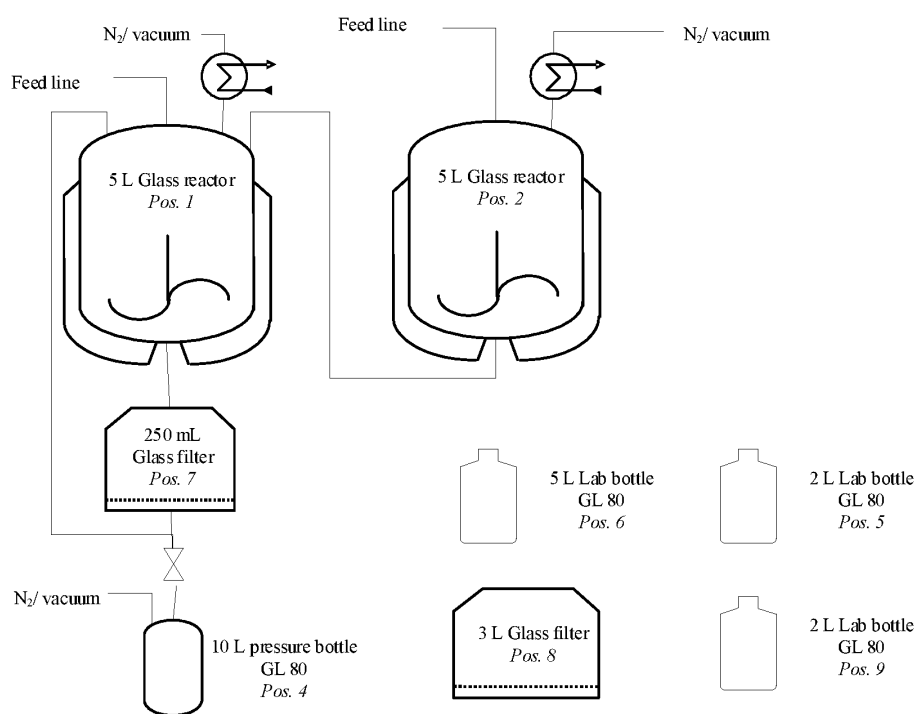


Figure 1 part 2.

Amount [g]	Input	Reaction and equipment	Amount [mL]	Output
125 540	4-Acetoxyindole CPME	Dissolution of 4-Acetoxyindole Flask Pos. 5 5 vol		
381 136	CPME (COCl) ₂	Synthesis of acid chloride		
50.7	CPME (via Pos. 1)			
540	CPME	Distillation	625	HCl (g) Distillate 1
323	CPME		625	Distillate 2
428	n-heptane	Reactor Pos. 2 Vmax = 15 vol	320	Distillate 3
		ca 10 vol		
214	n-heptane	Filtration of acid chloride	1200	Mother liquor
214	n-heptane		313	Washing liquor 1
1800	2-MeTHF (via Pos. 2)	Glass filter Pos. 3	313	Washing liquor 2
		ca. 17 vol		
		Solution of Acid chloride Reactor Pos. 2		

Figure 1 part 3.

Amount [g]	Input	Reaction and equipment	Amount [mL]	Output
211	2-MeTHF	Synthesis of Diethylamide		
110	DEA			
150	2-MeTHF (via Pos. 2 and Pos. 3)			
150	2-MeTHF (via Pos. 2 and Pos. 3)			
		Workup		
568	0.5N HCl		570	Aqueous phase 1 (from Pos. 2)
568	0.5N HCl		570	Aqueous phase 2 (from Pos. 2)
480	2-MeTHF (via Pos. 2)			
480	2-MeTHF (via Pos. 2)	Reactor Pos. 1 Vmax = 33.5 vol		
281	deion. water		280	Aqueous phase 3
12.5 g	Activated charcoal	Activated charcoal treatment Lab. flask Pos. 6		
100 g	Celite	Filtration Glass filter Pos. 8 Flask Pos. 4		
150	2-MeTHF (via Pos. 6)			
150	2-MeTHF (via Pos. 4)			
513	n-heptane	Destillation Fällung Reaktor Pos. 2 Vmax = 34 vol	3500	Distillate 3

Figure 1 part 4.

Amount [g]	Input	Reaction and equipment		Amount [mL]	Output
197	2-MeTHF (via Pos. 2)	↓ Product-isolation Glass filter Pos. 7		1500	Mother liquor 2
99.3	2-MeTHF (via Pos. 2)			230	Washing liquor 3
79.5	n-heptane (via Pos. 2)			230l	Washing liquor 4
513	n-heptane (via Pos. 2)			750	Washing liquor 5
			Product		waste
		163 g	Acetoxyindole glyoxylic acid diethylamide C-029855		Aqueous waste org. waste

Figure 1 part 5.

Step 2 – 4-HO-DET

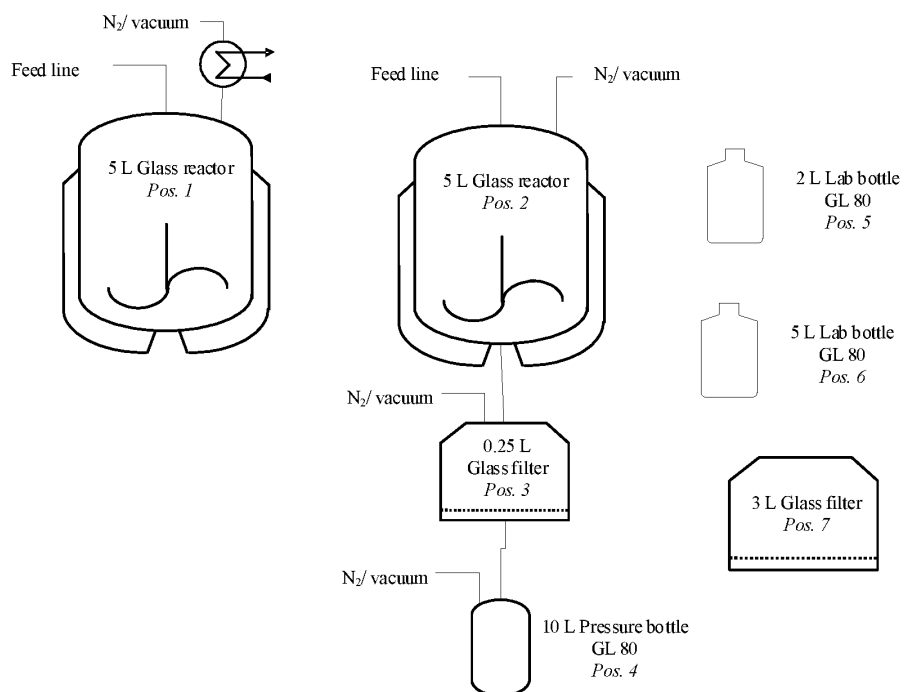


Figure 2 part 1.

Amount [g]	Input	Reaction and equipment	Amount [L]	Output
100 15 vol	Acetoxyindole glyoxylic acid diethylamide Toluene	Reaction and workup Reactor Pos. 1 ca. 35 vol	3 vol	Distillate 1 (Toluene/Water)
370 mL 0.5 vol 3 vol	LAH 2.4M in THF Toluene Toluene			
5 vol 20 vol 4 vol 4 vol 4 vol	K-Na-Tartrate K-Na-Tartrate Toluene Toluene Toluene		4 vol	Distillate 2 (THF/Toluene)
2.5 vol 2.5 vol 3.5 vol 3.5 vol	K-Na-Tartrate deion. water Toluene Toluene			
			25 vol	Acqueous Phase 1
			5 vol	Acqueous Phase 2
5 g	activated charcoal	Activated charcoal treatment Flask Pos. 6 ca. 35 vol		
ca 125 g	Celite Celpure	Filtration Glass filter Pos. 7 ca. 35 vol		
250 1.5 vol	Sodium sulfate Toluene (via Pos. 6+7)	Drying Flask Pos. 4 ca. 36 vol		

Figure 2 part 2.

Amount [g]	Input	Reaction and equipment		Amount [L]	Output
2.0 vol 0.5 vol	Isopropylacetate n-heptane	↓ Distillation Precipitation Reactor Pos. 2 ca. 4 vol Product isolation Glass filter Pos. 3		35 vol	Distillate 3 (Toluene)
1.5 vol 1.5 vol	Isopropylacetate n-heptane	↓ Product isolation Glass filter Pos. 3		4 vol	Mother liquor
				1.5 vol	Washing liquor 1
				1.5 vol	Washing liquor 2
			Product		Waste
		46 g	4-HO-DET C-029847	3.5 4.5	Acqueous waste org. waste

Figure 2 part 3.

Step 3 – Ethocybin crude

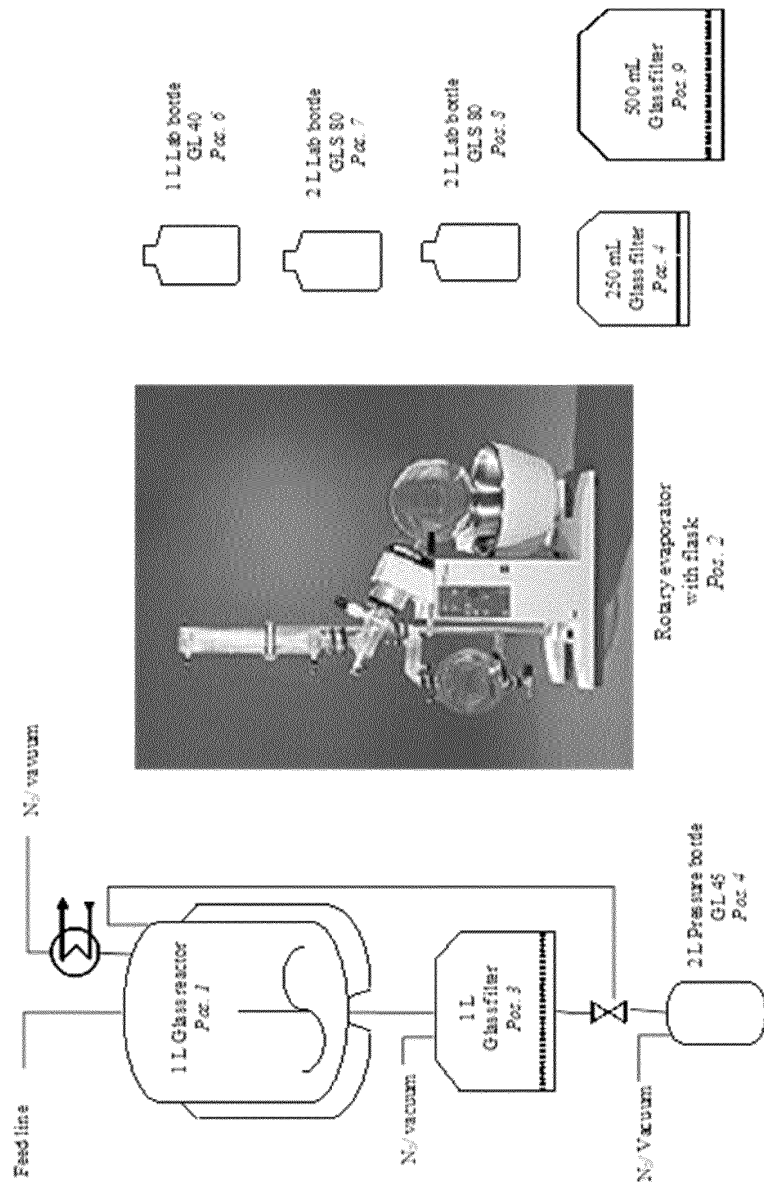


Figure 3 part 1.

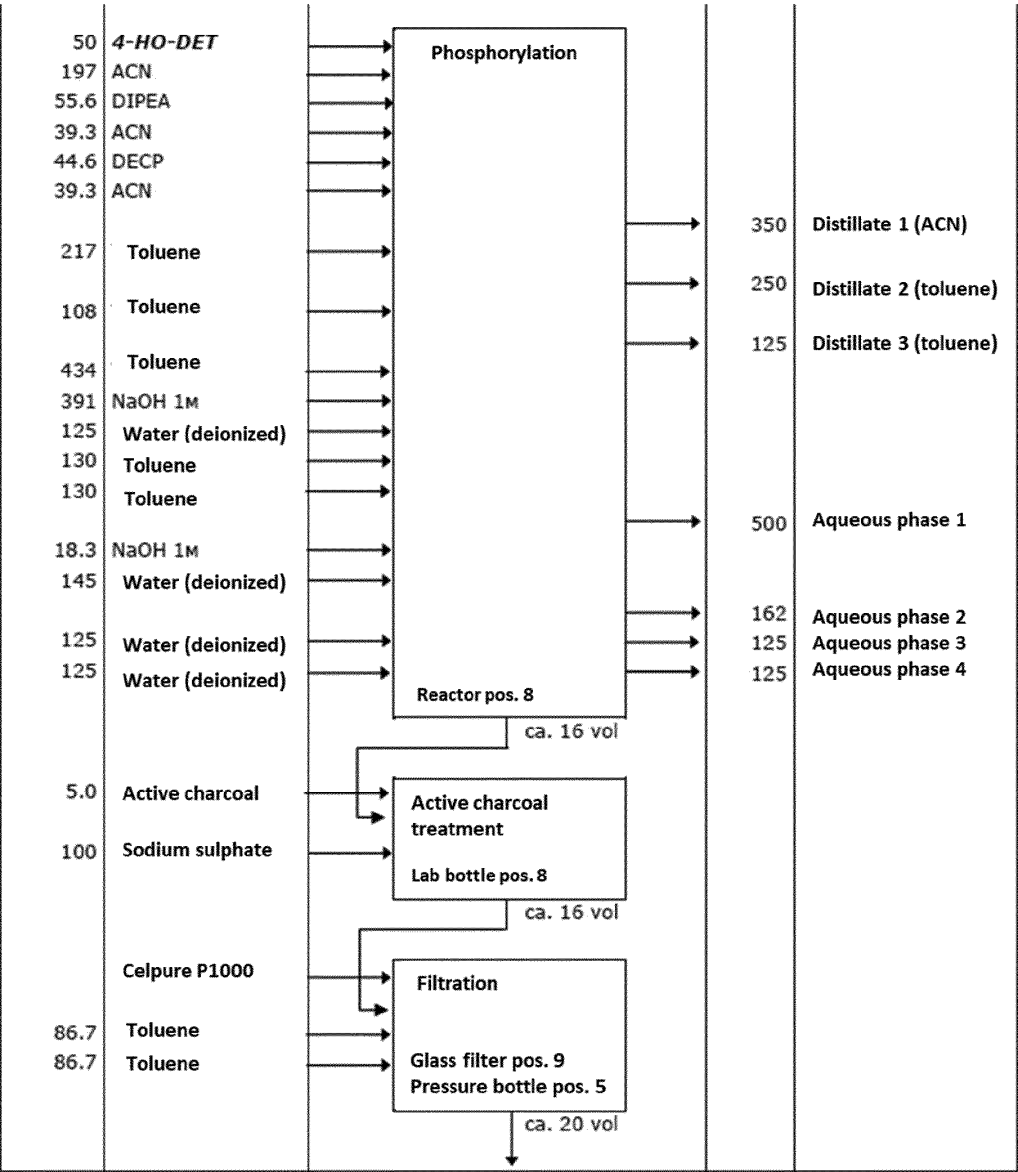


Figure 3 part 2.

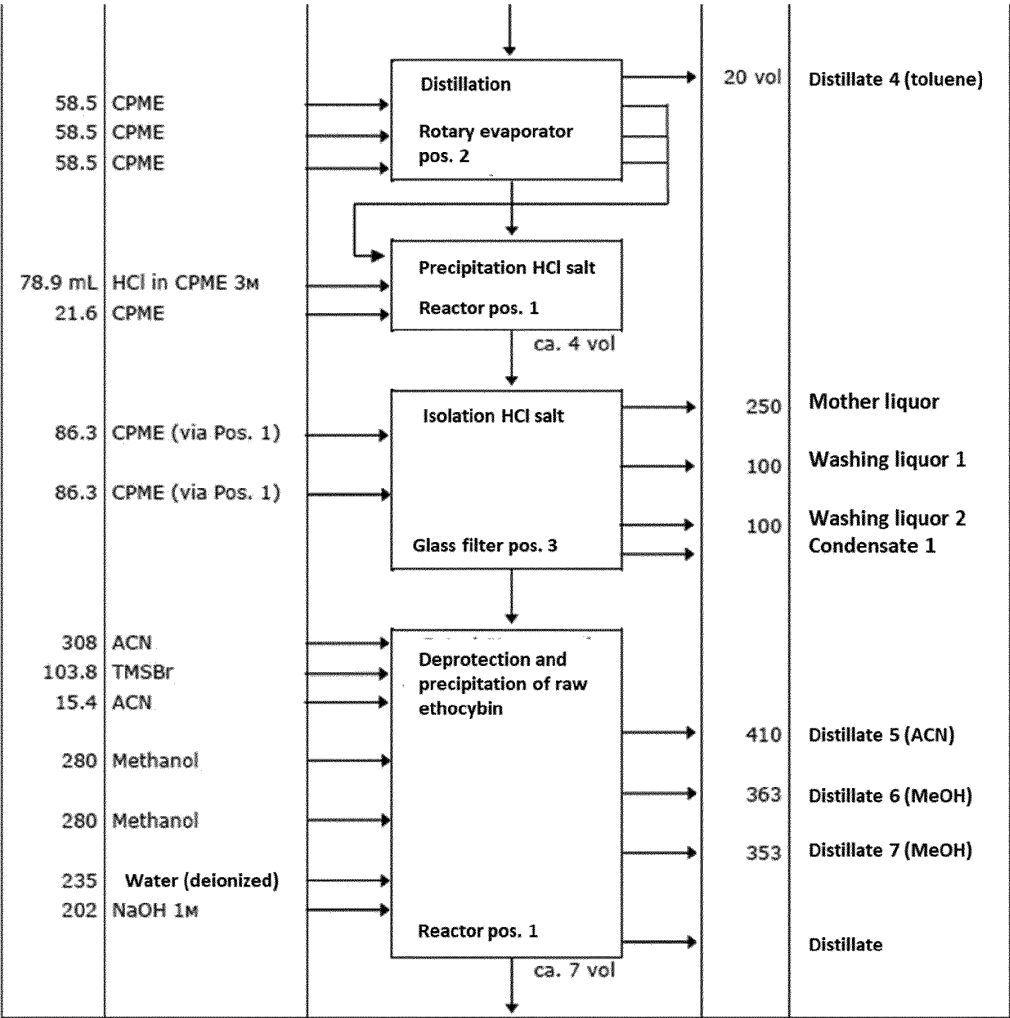


Figure 3 part 3.

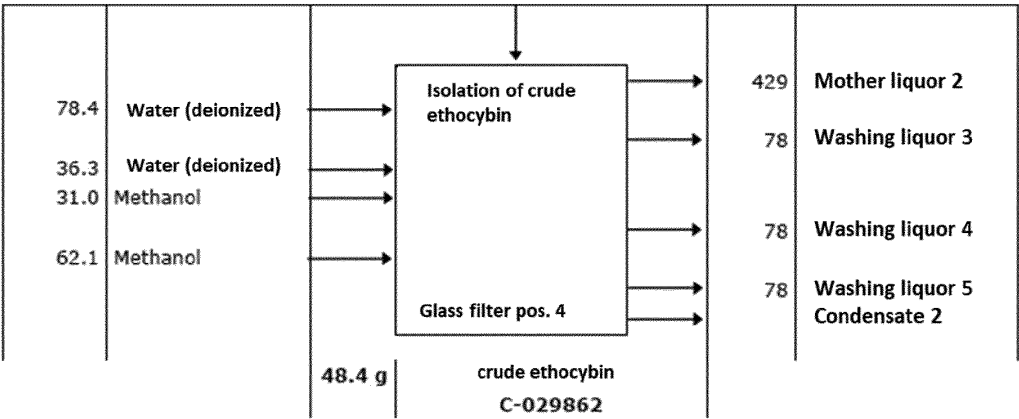


Figure 3 part 4.

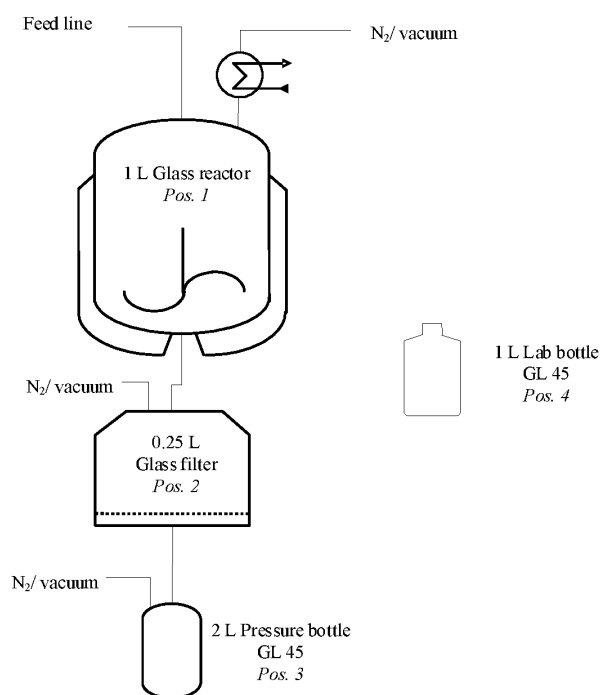
Step 4 – Ethocybin

Figure 4 part 1.

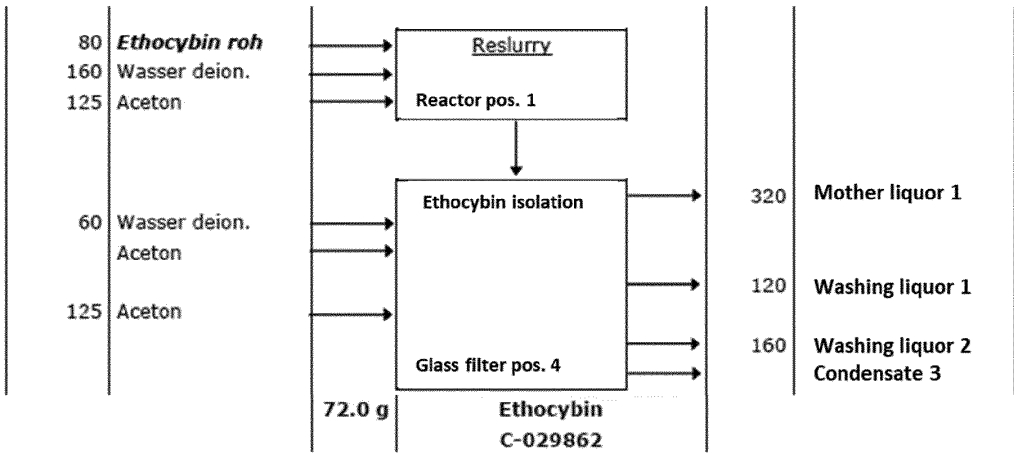


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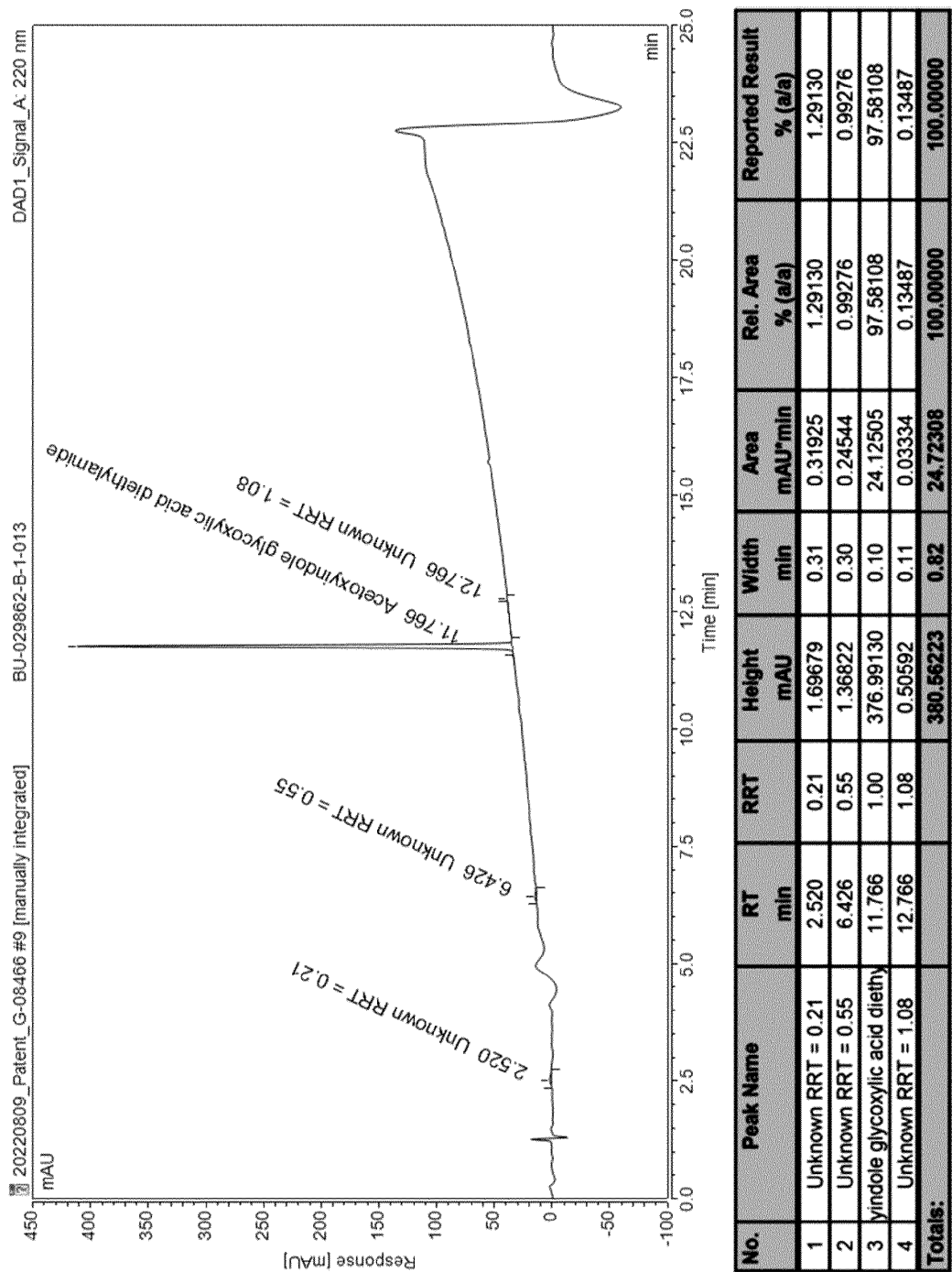


Figure 5 part 1.

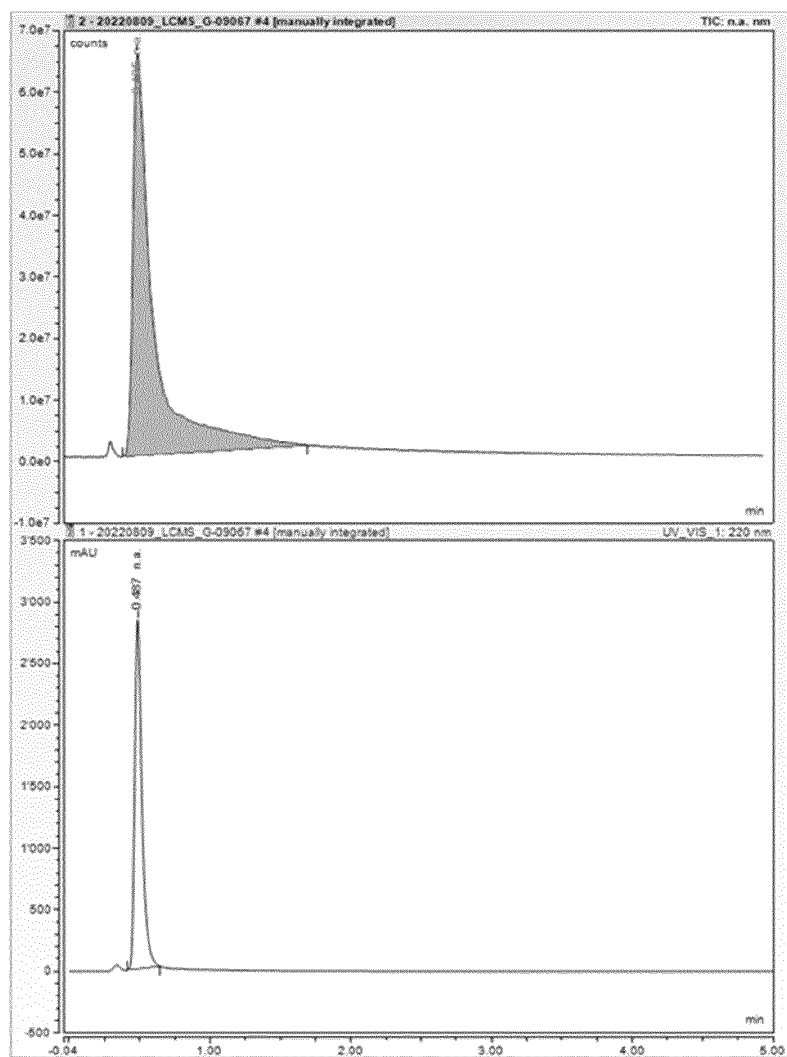


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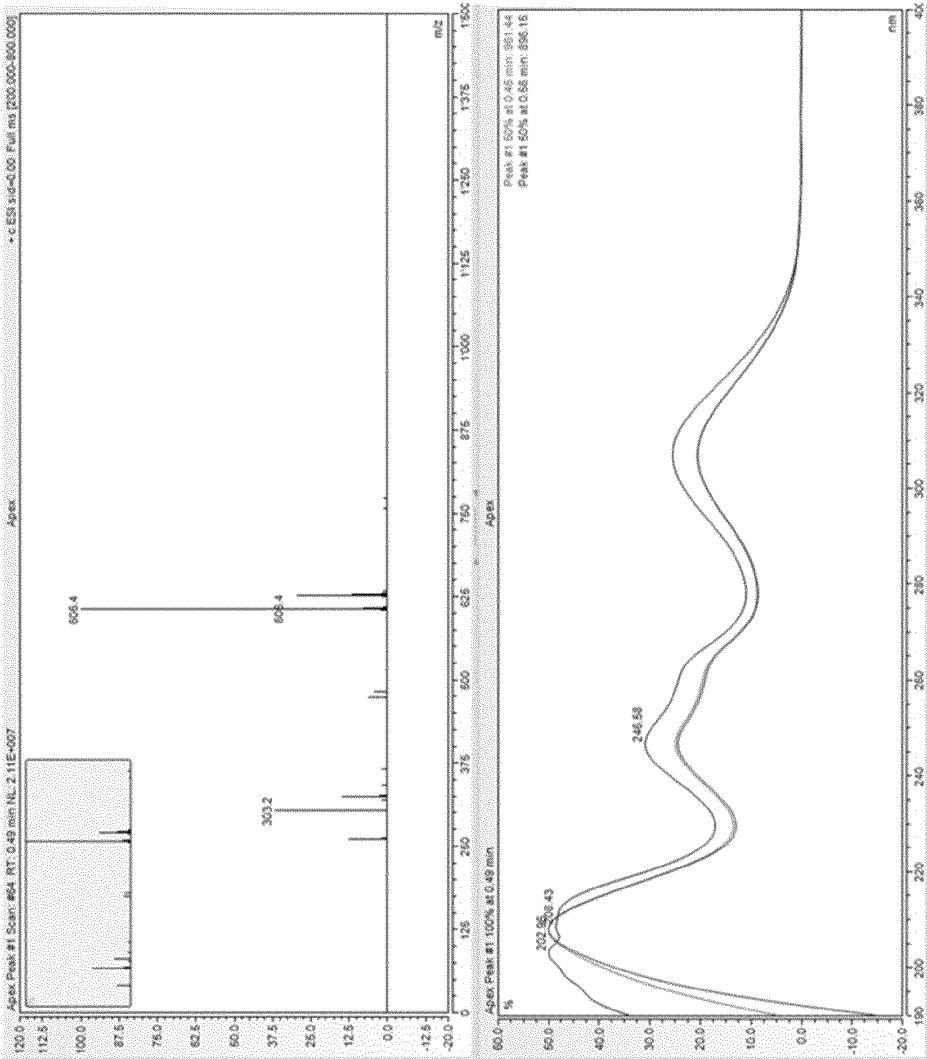


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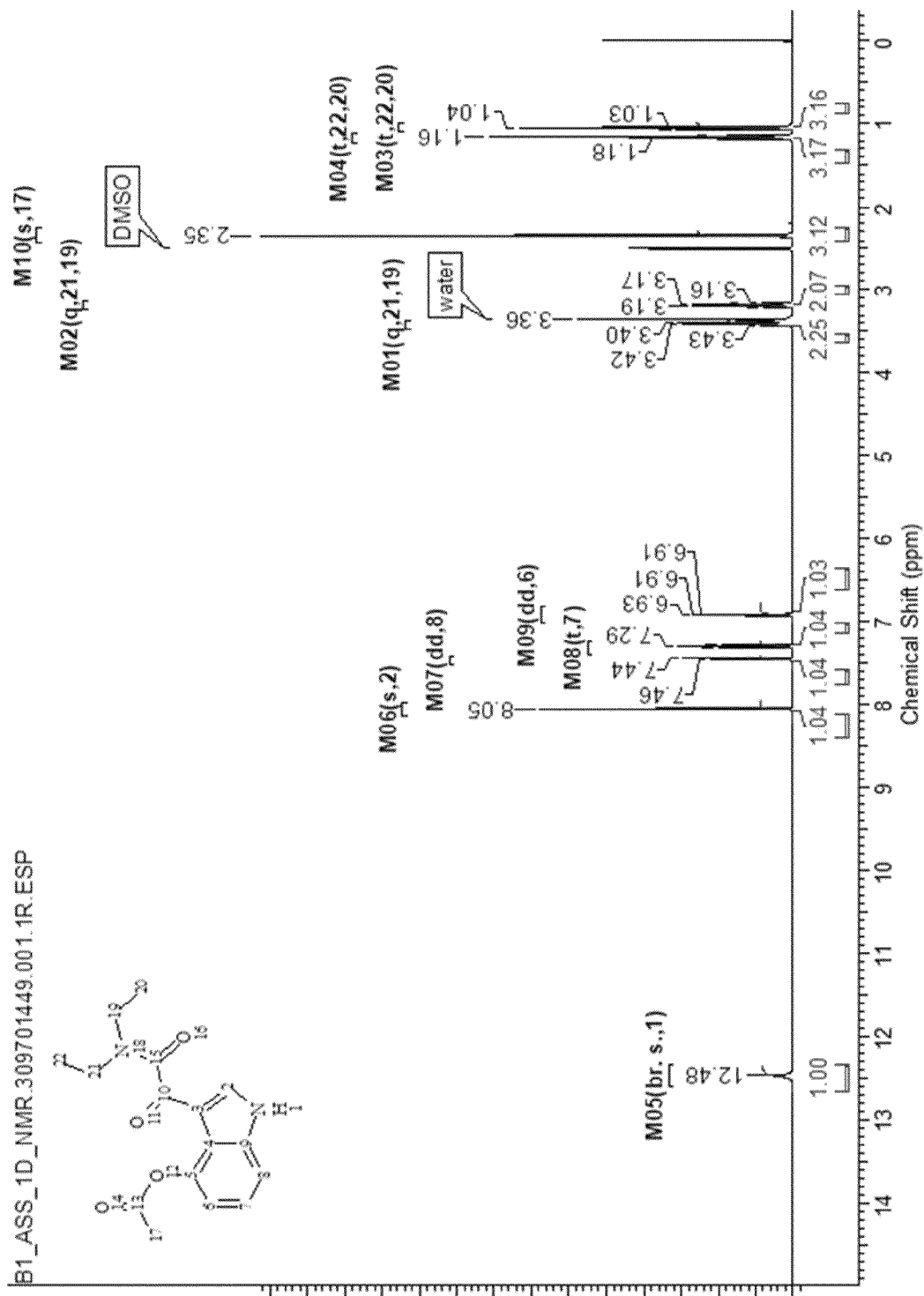


Figure 5 part 4.

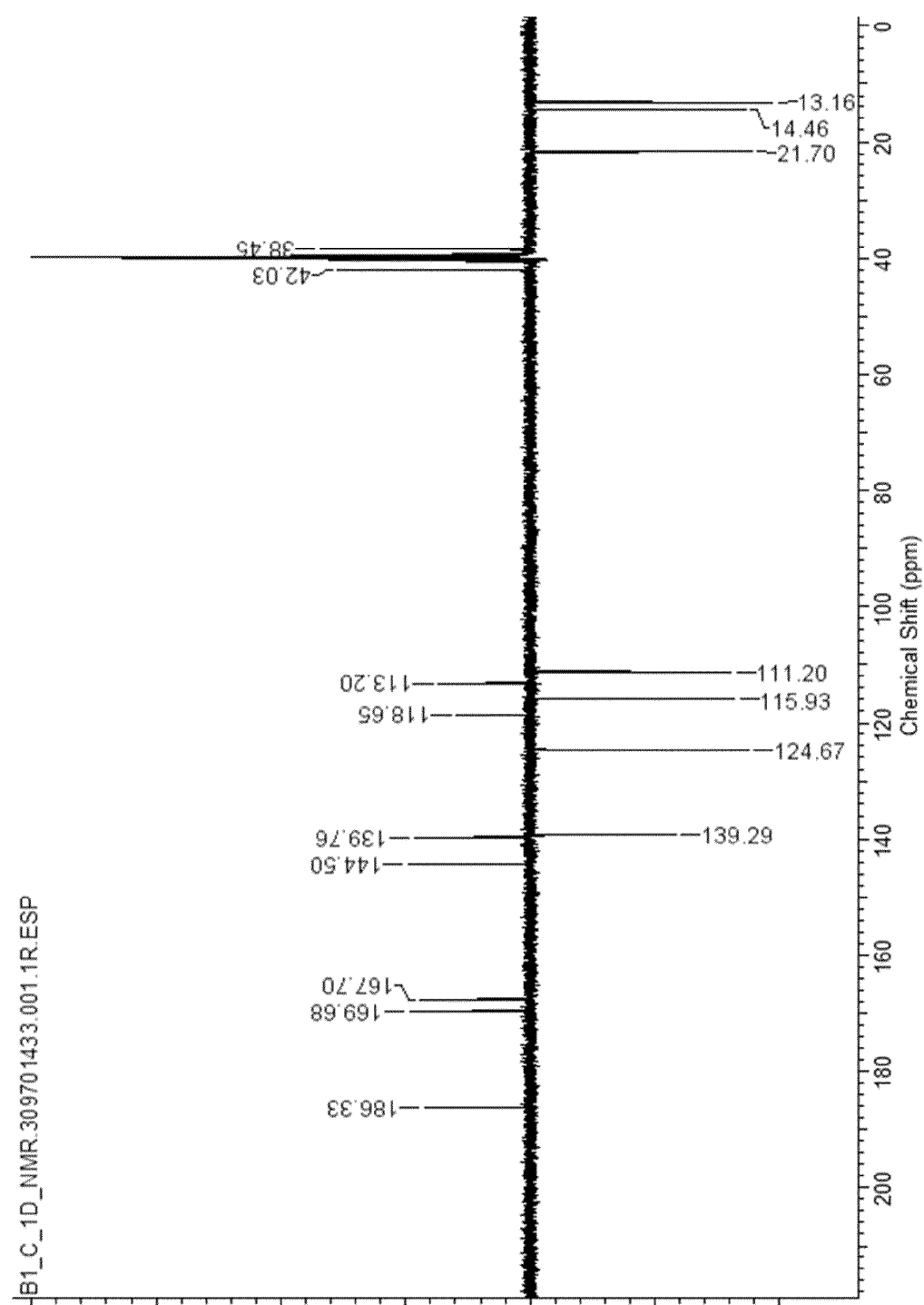


Figure 5 part 5.

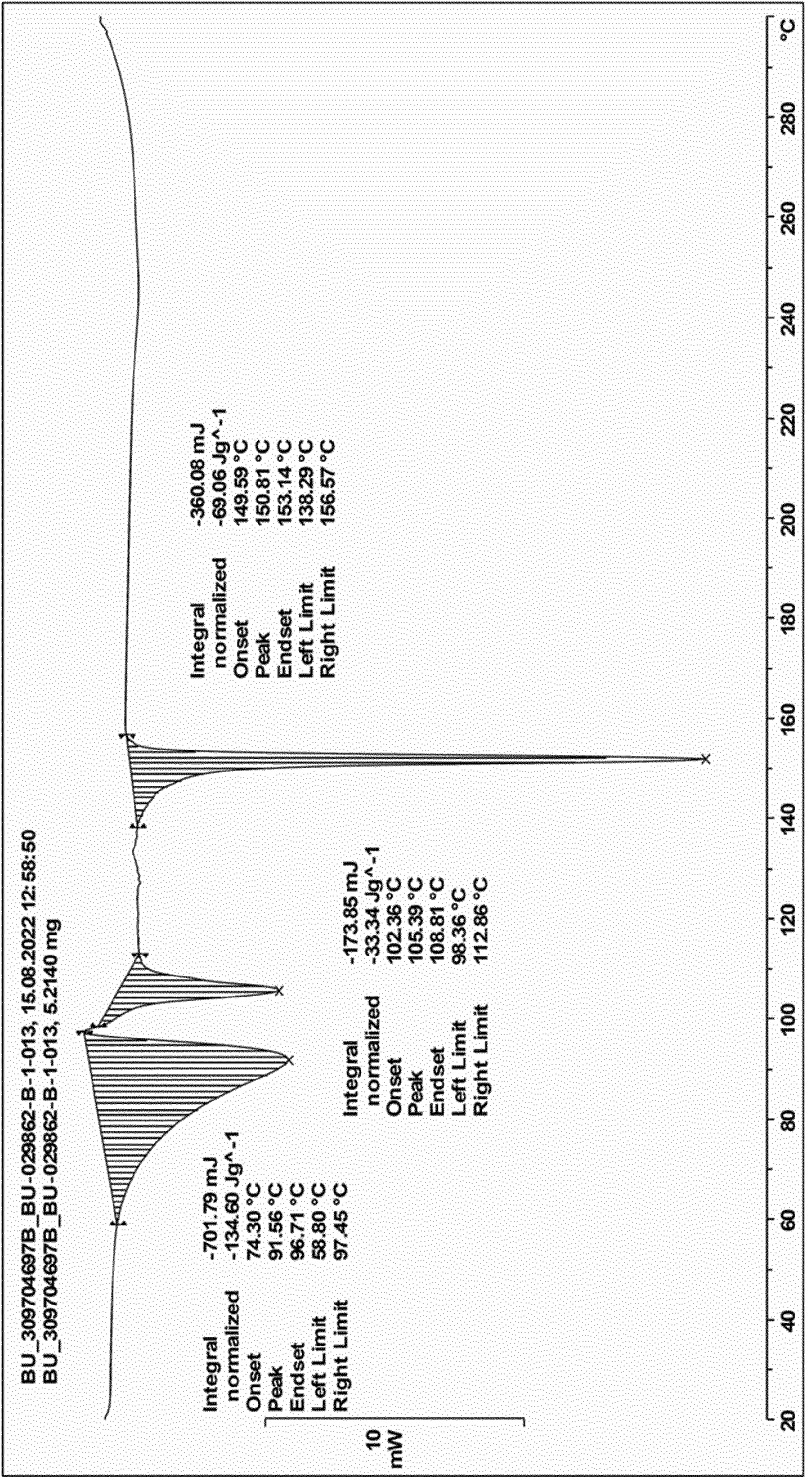
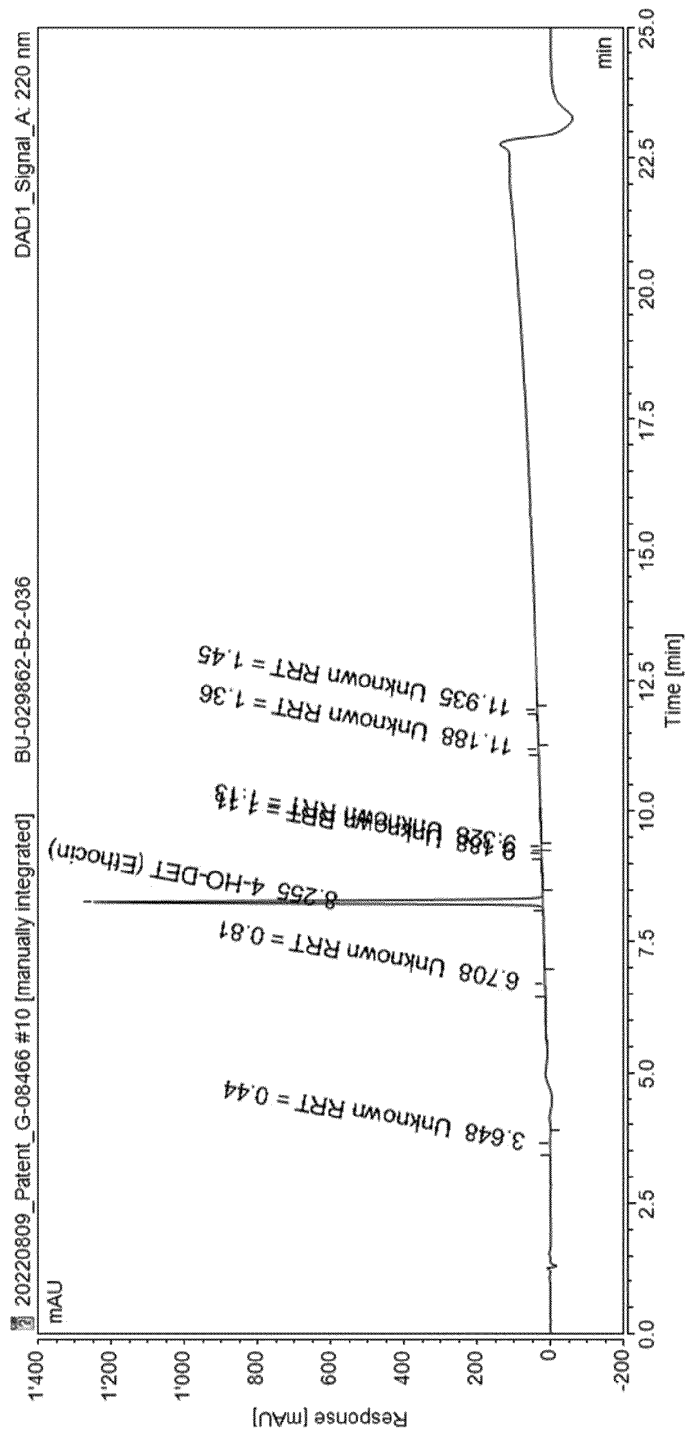


Figure 5 part 6.



No.	Peak Name	RT min	RRT	Height mAU	Width min	Area mAU*min	Rel. Area % (a/a)	Reported Result % (a/a)
1	Unknown RRT = 0.44	3.648	0.44	1.99754	0.35	0.43628	0.57492	0.57492
2	Unknown RRT = 0.81	6.708	0.81	1.11692	0.44	0.33583	0.44256	0.44256
3	4-HO-DET (Ethocin)	8.255	1.00	1228.74335	0.10	74.68731	98.42231	98.42231
4	Unknown RRT = 1.11	9.188	1.11	2.47063	0.13	0.19516	0.25717	0.25717
5	Unknown RRT = 1.13	9.328	1.13	1.42923	0.09	0.08542	0.11256	0.11256
6	Unknown RRT = 1.36	11.188	1.36	1.00523	0.10	0.06279	0.08274	0.08274
7	Unknown RRT = 1.45	11.935	1.45	1.13048	0.11	0.08175	0.10773	0.10773
Totals:				1237.89338	1.31	75.88453	100.00000	100.00000

Figure 6 part 1.

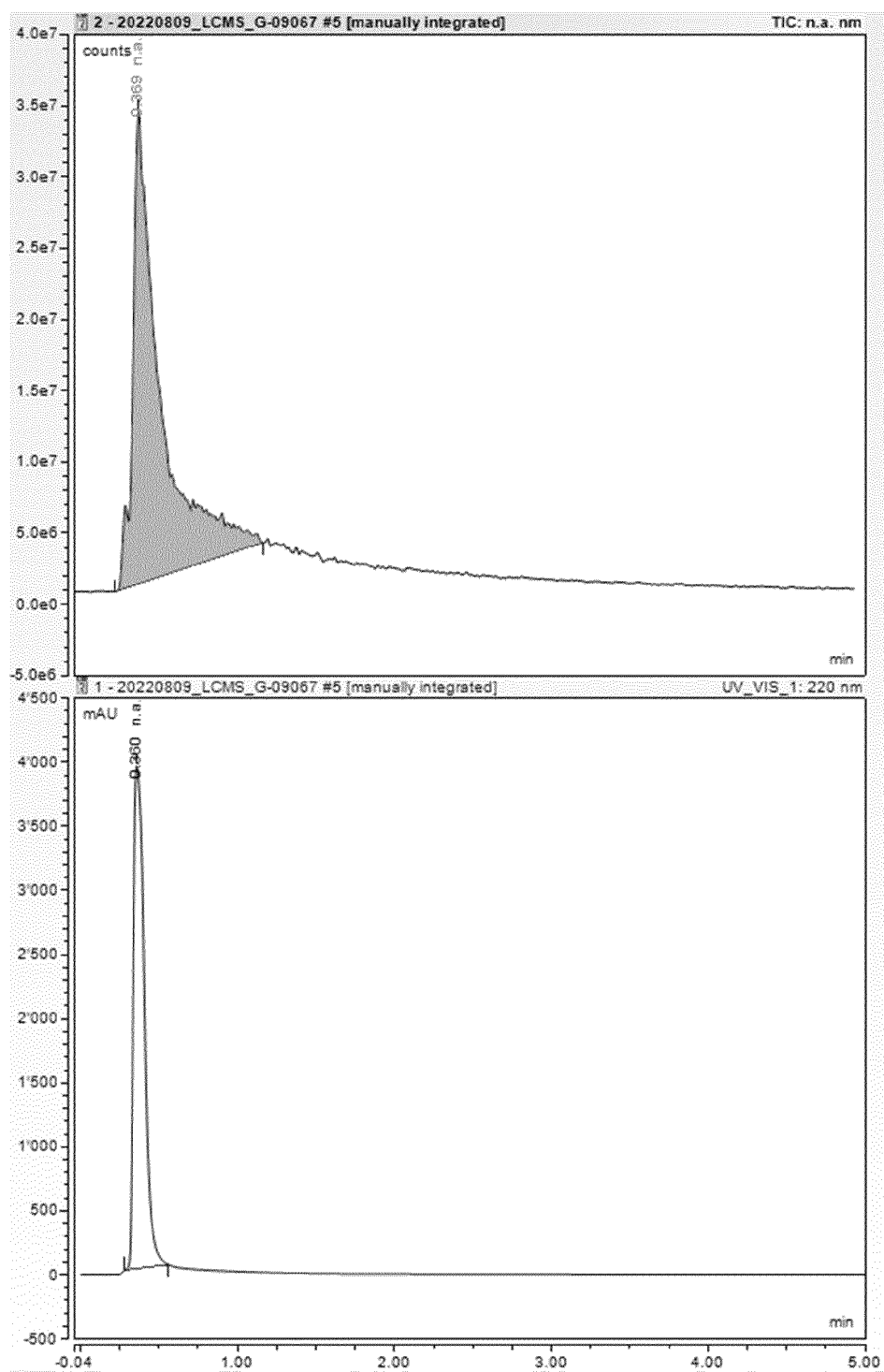


Figure 6 part 2.

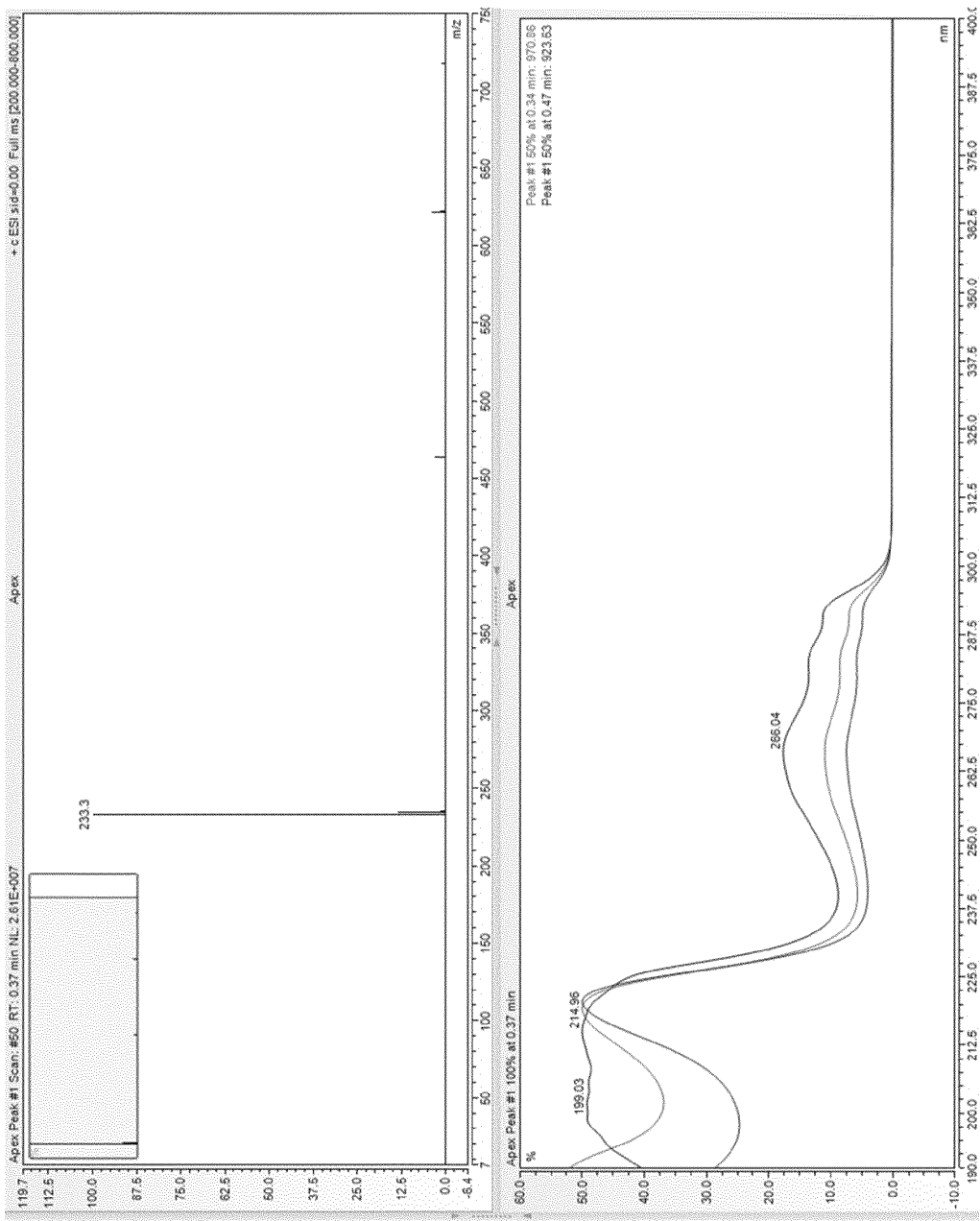


Figure 6 part 3.

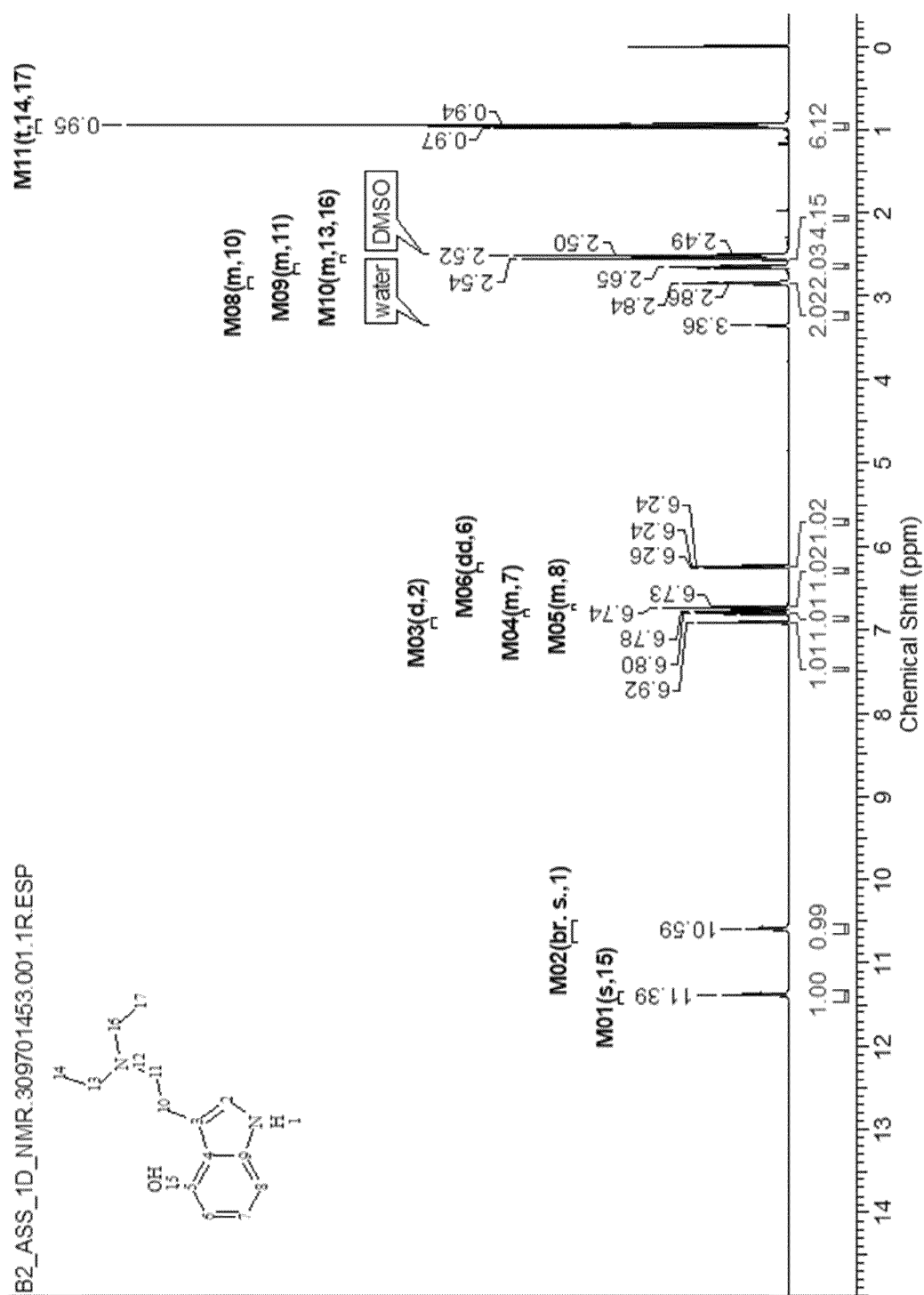


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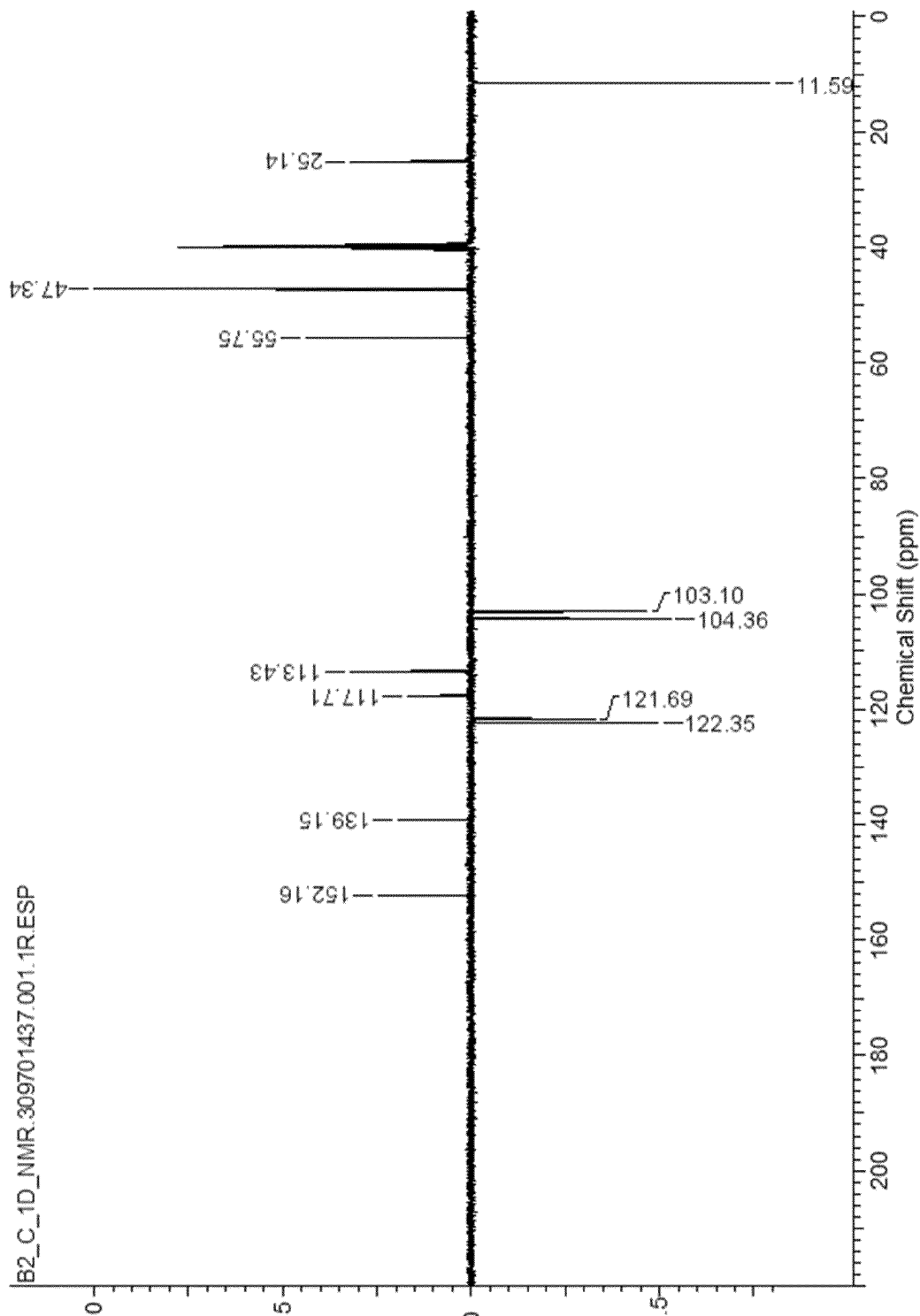


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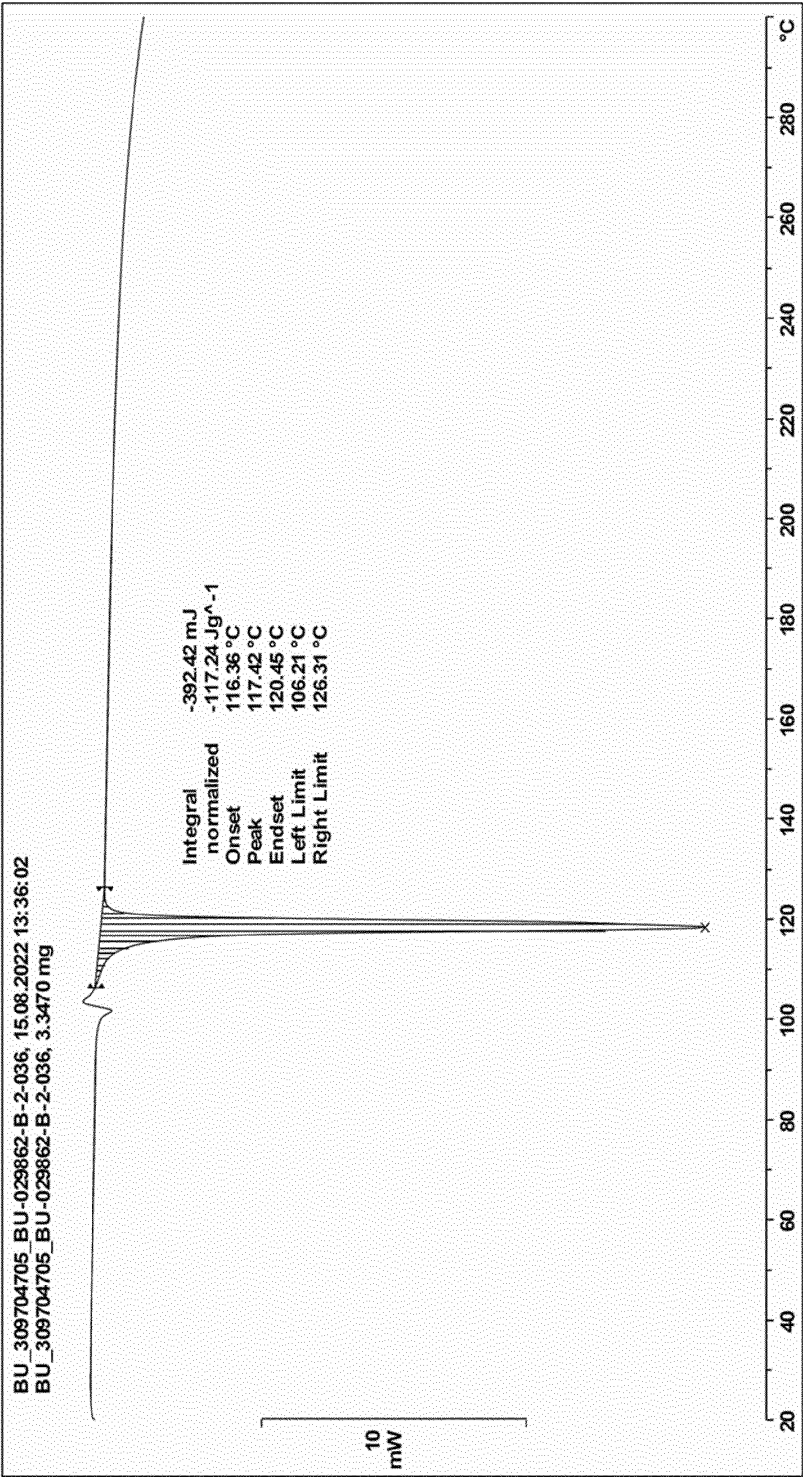


Figure 6 part 6.

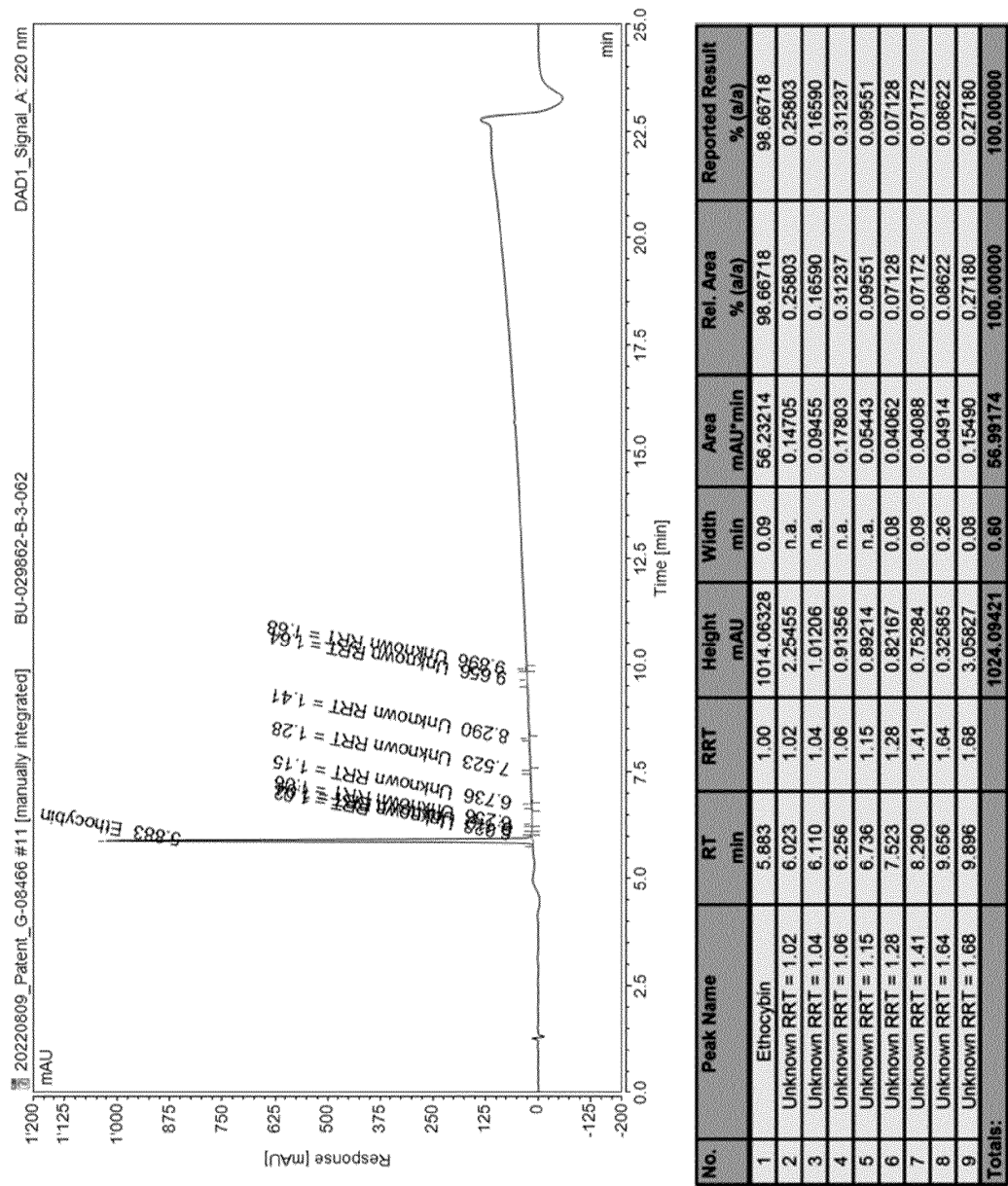


Figure 7 part 1.

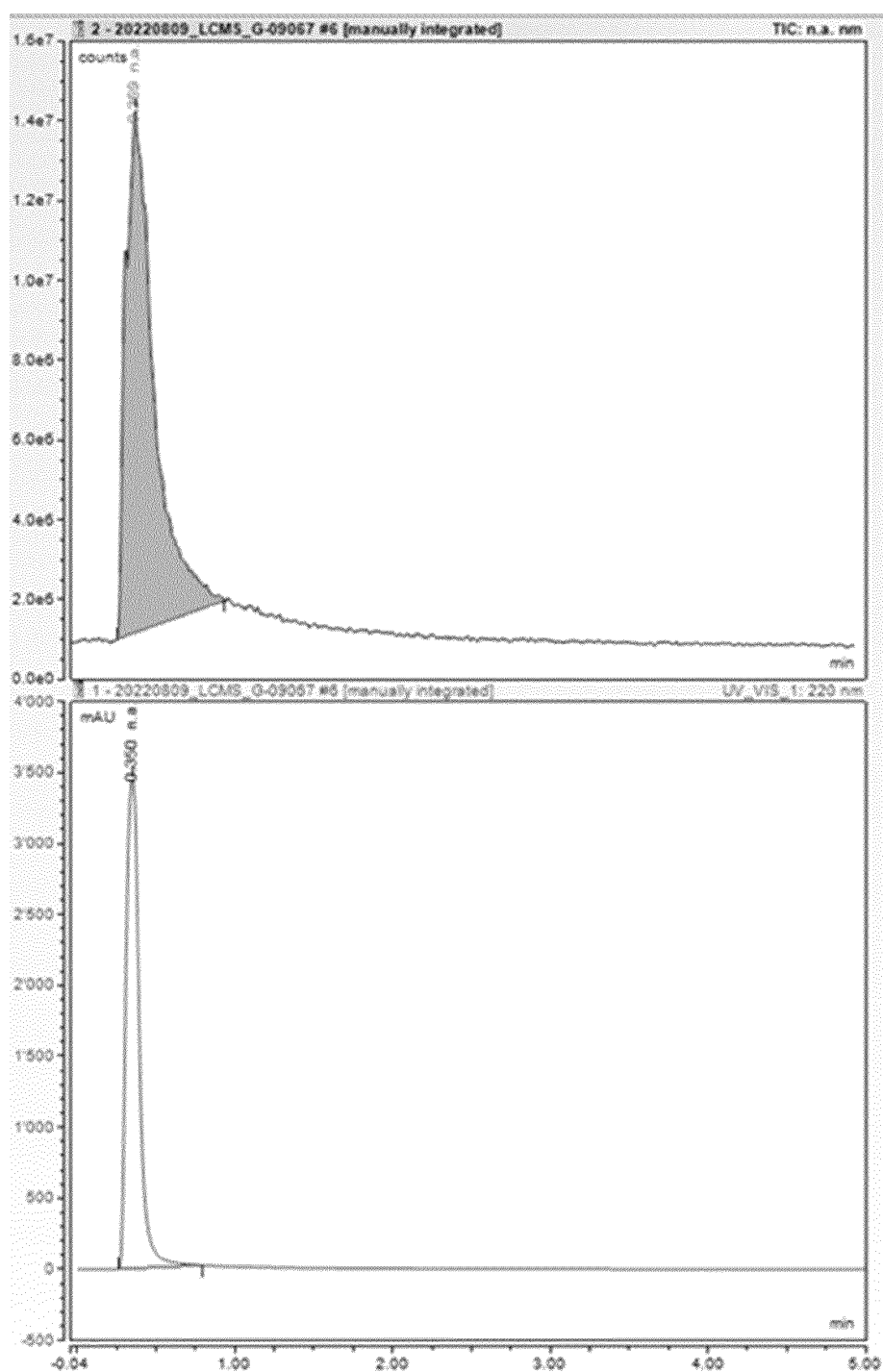


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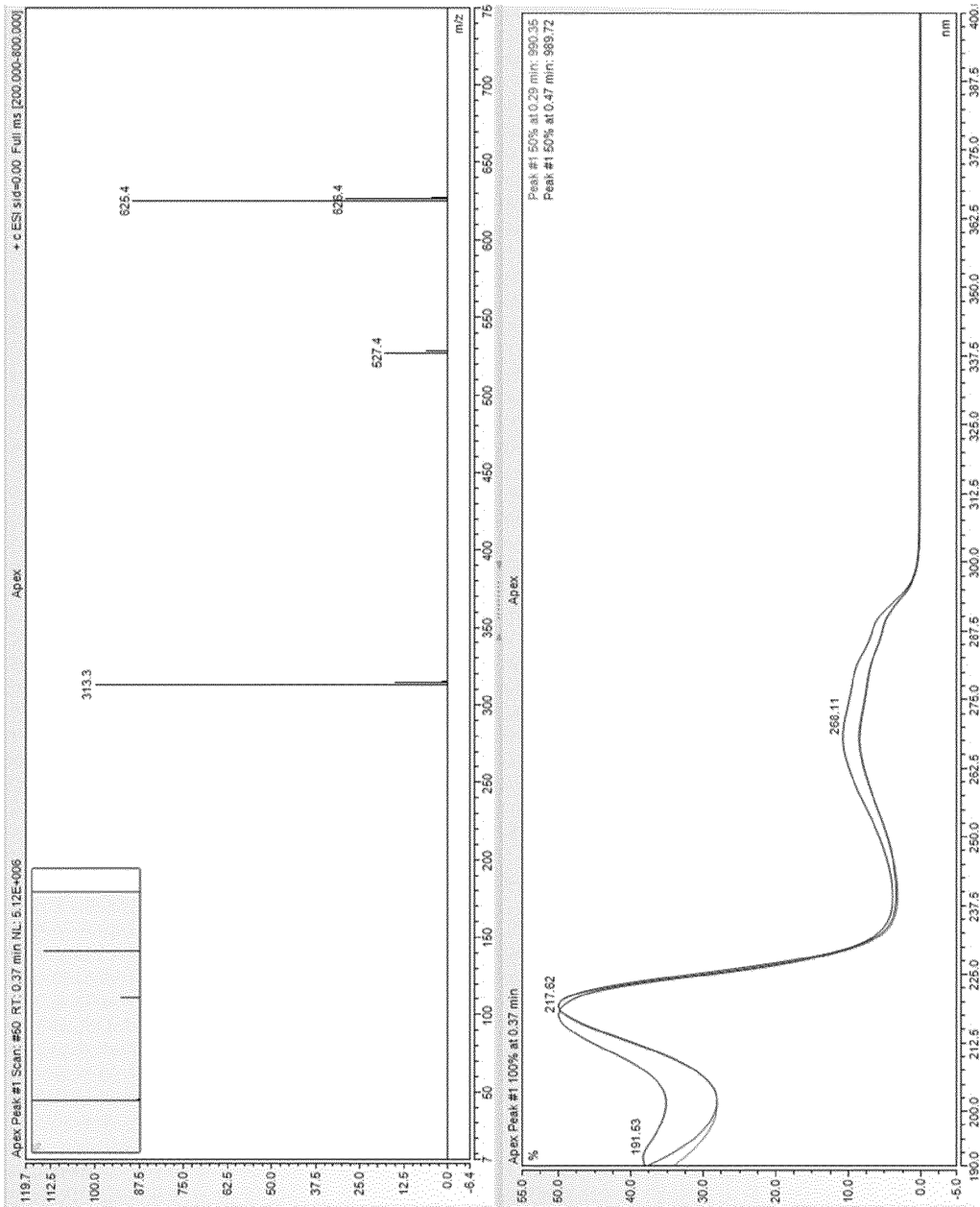


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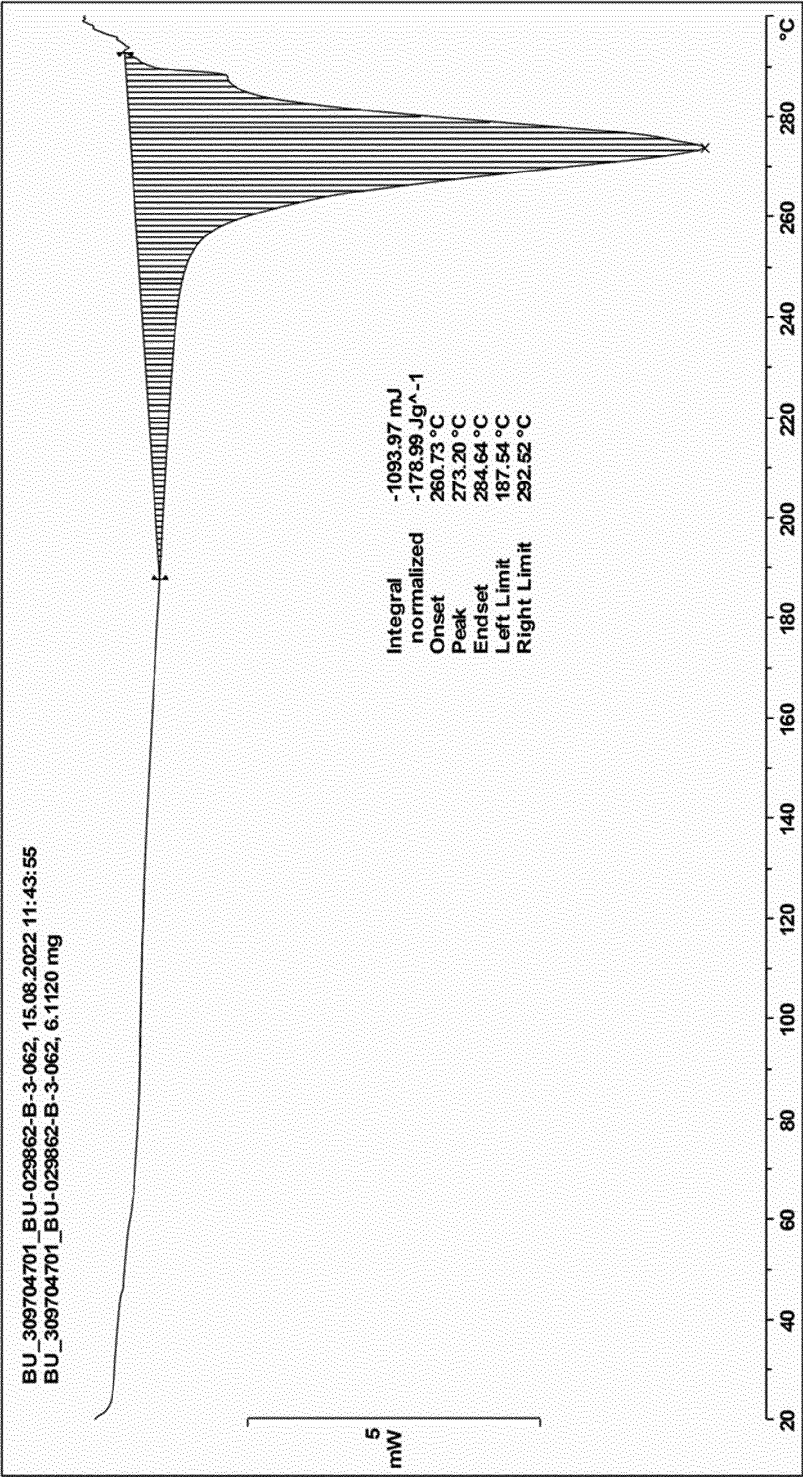


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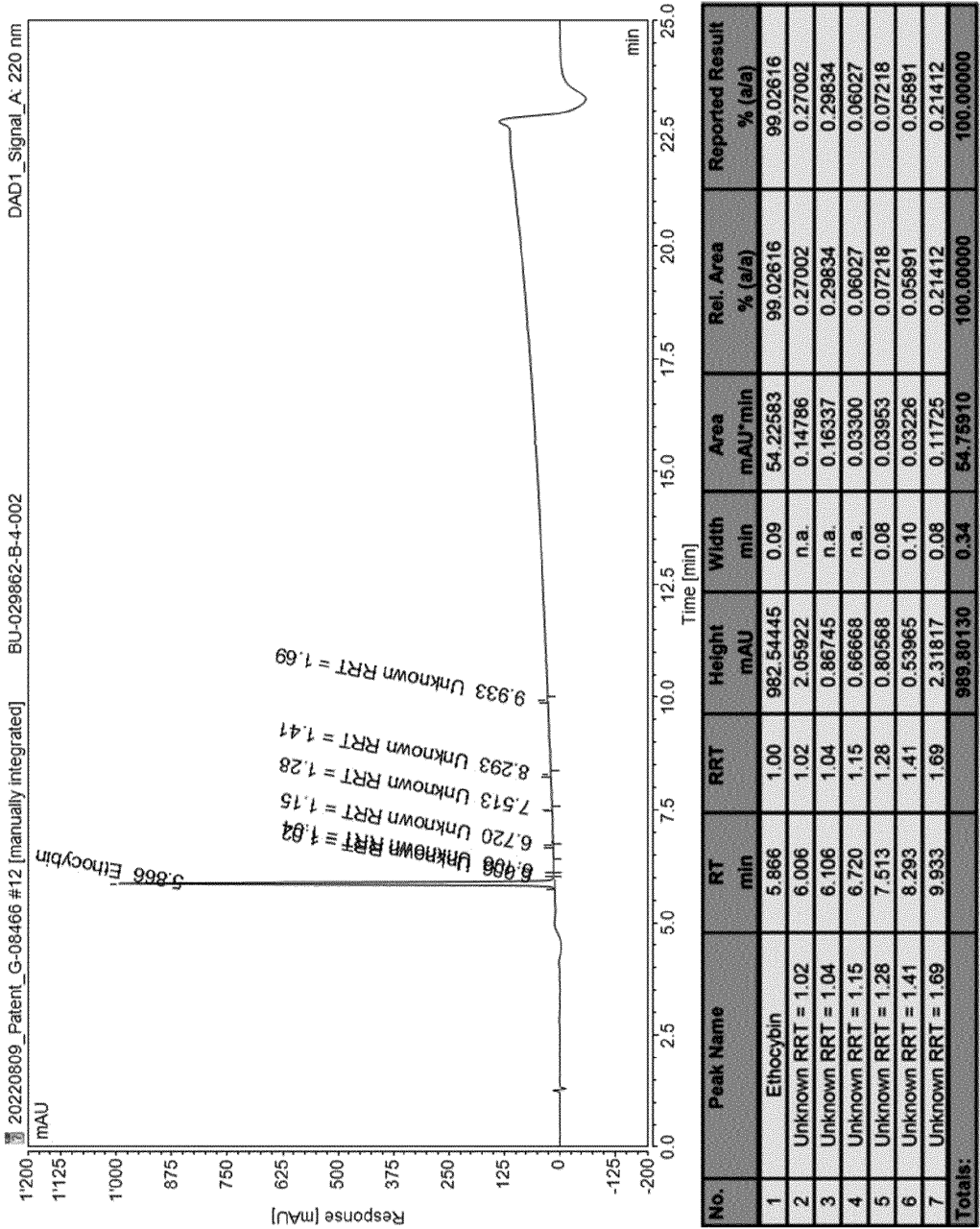


Figure 8 part 1.

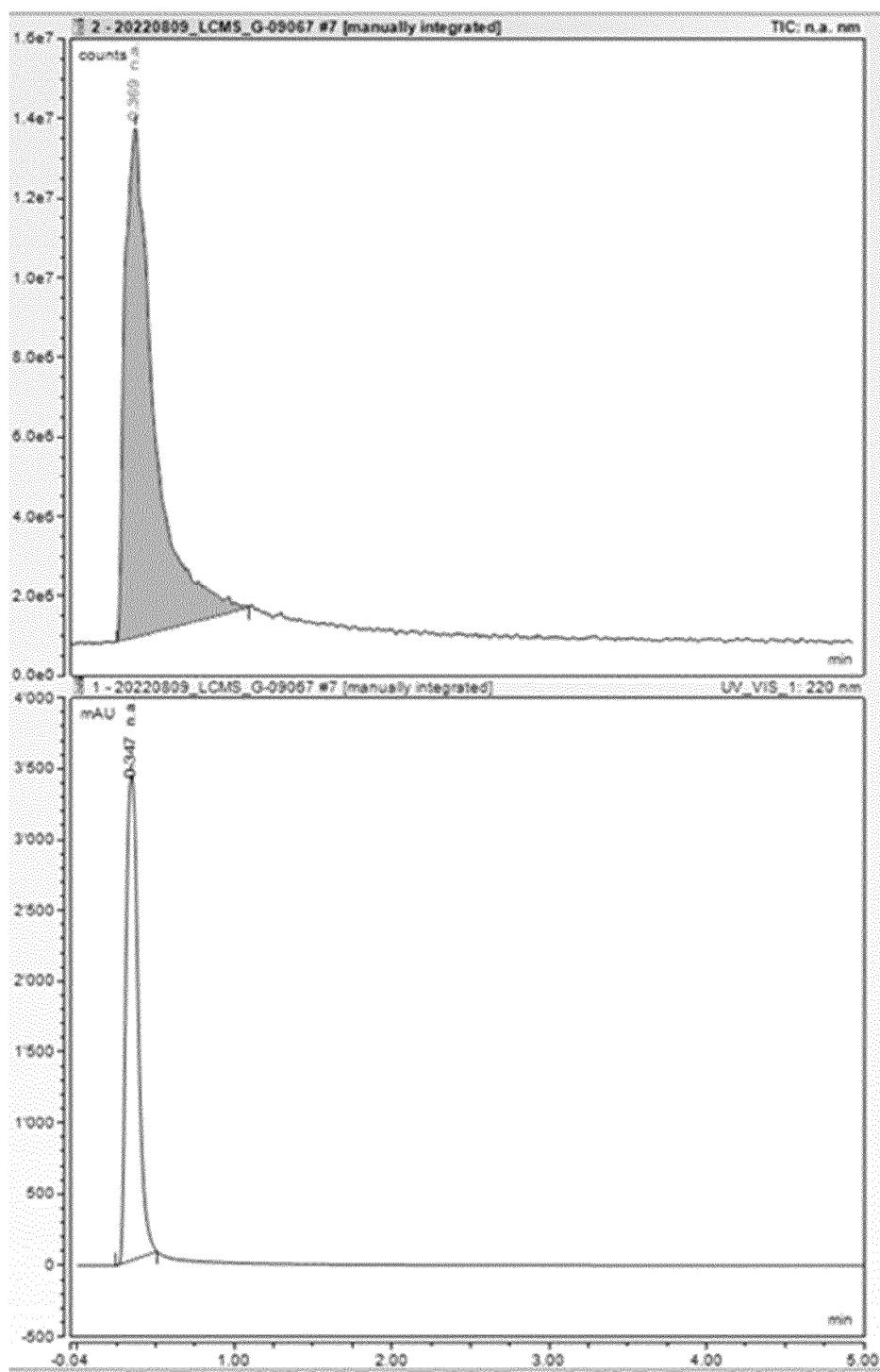


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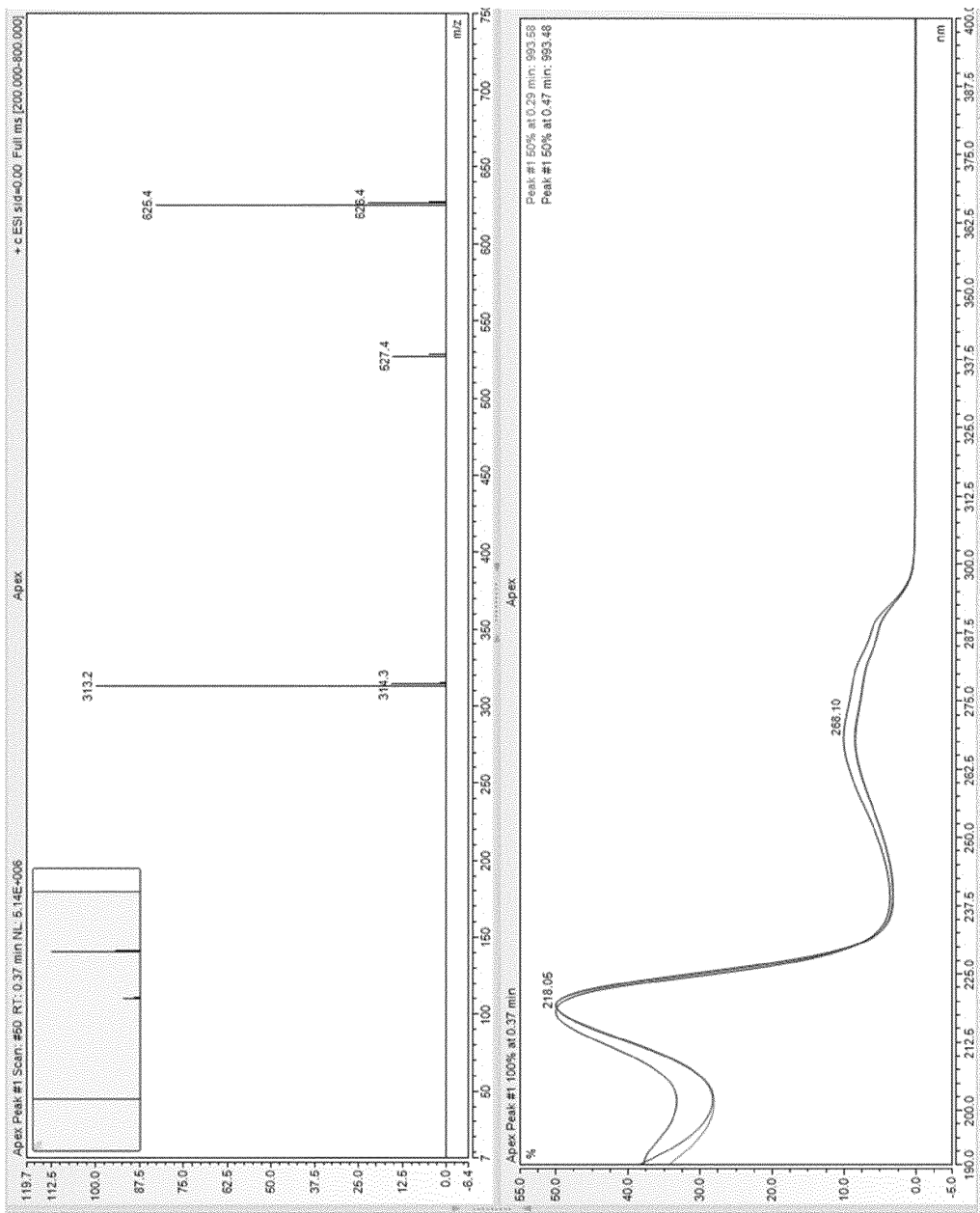


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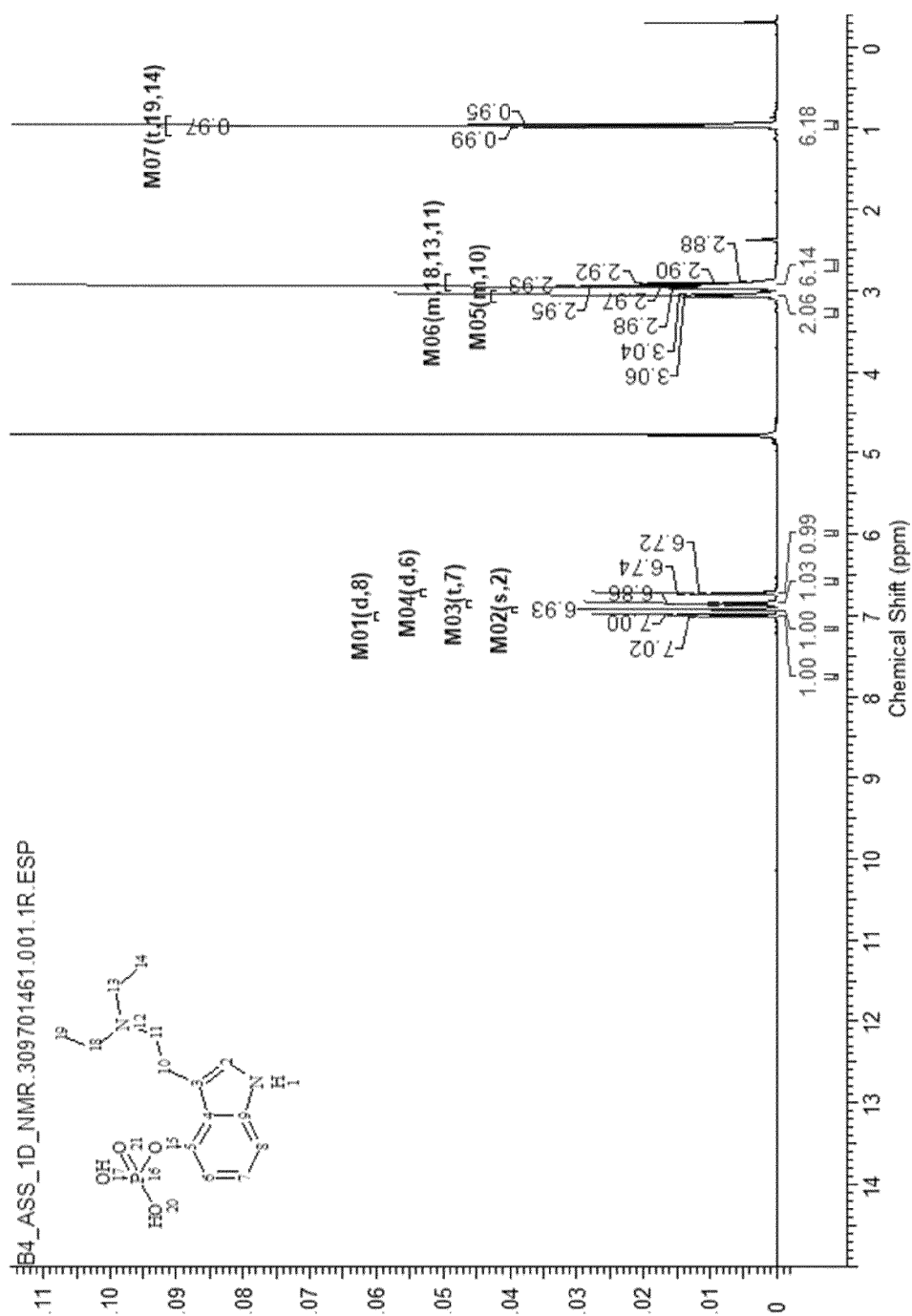


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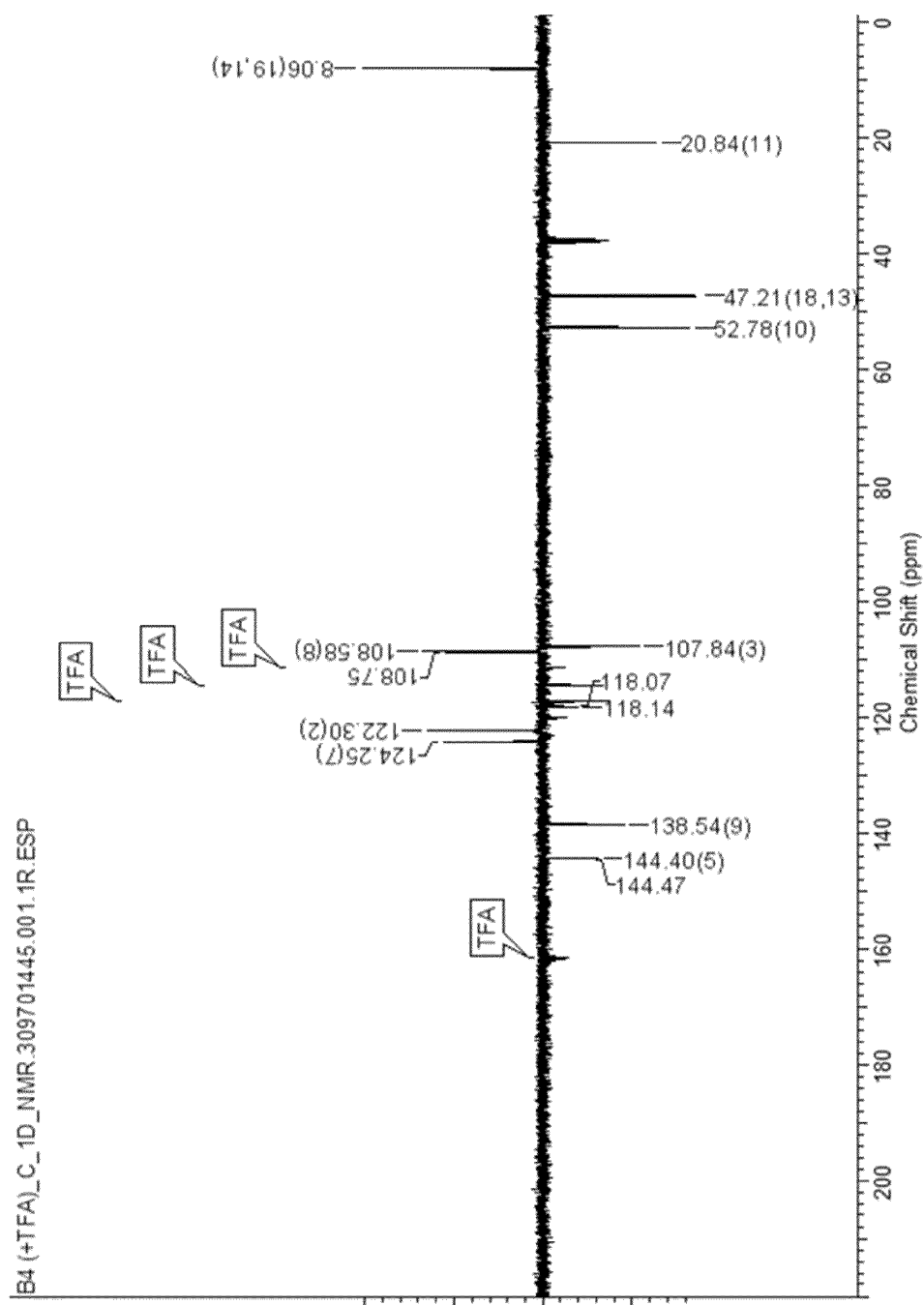


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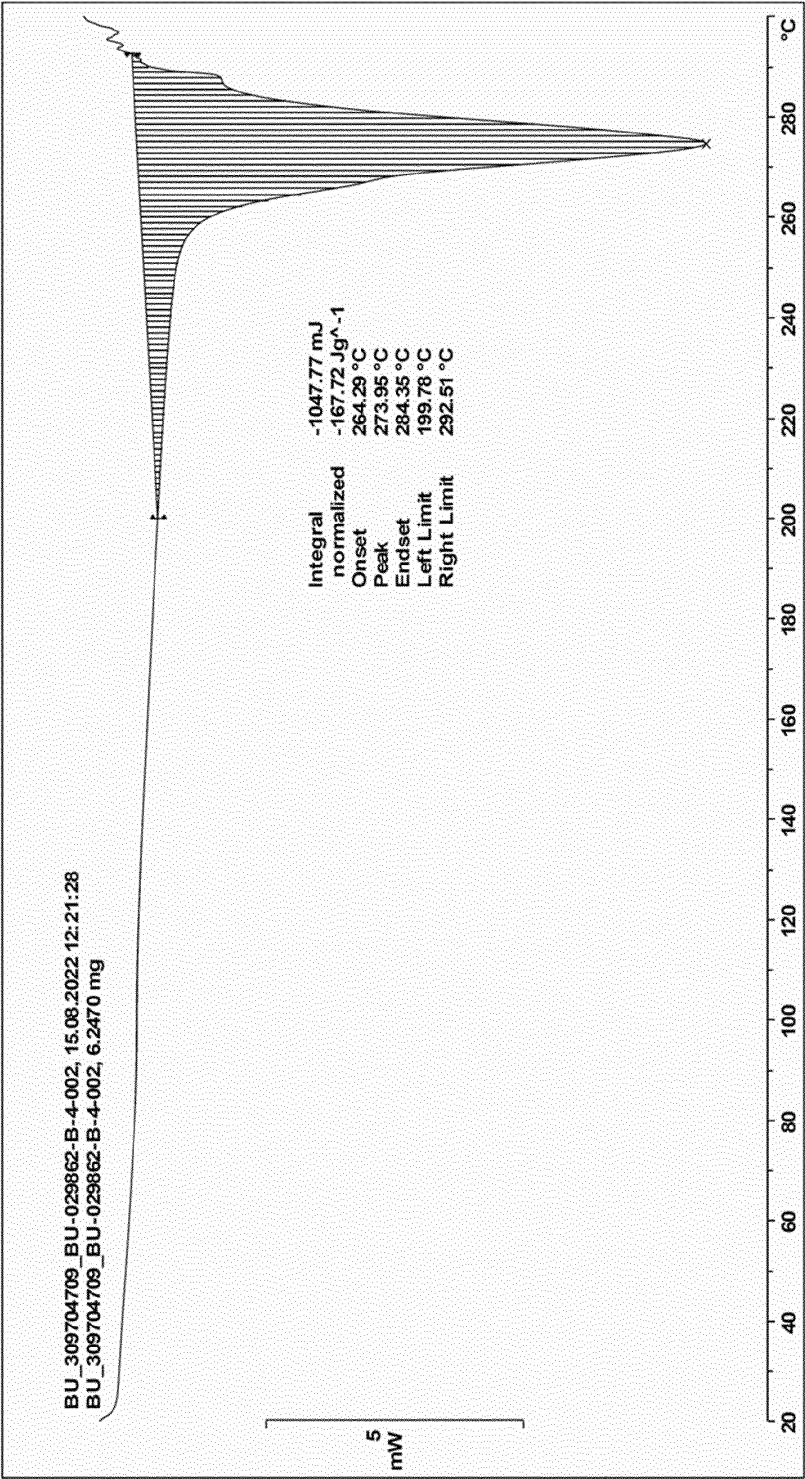


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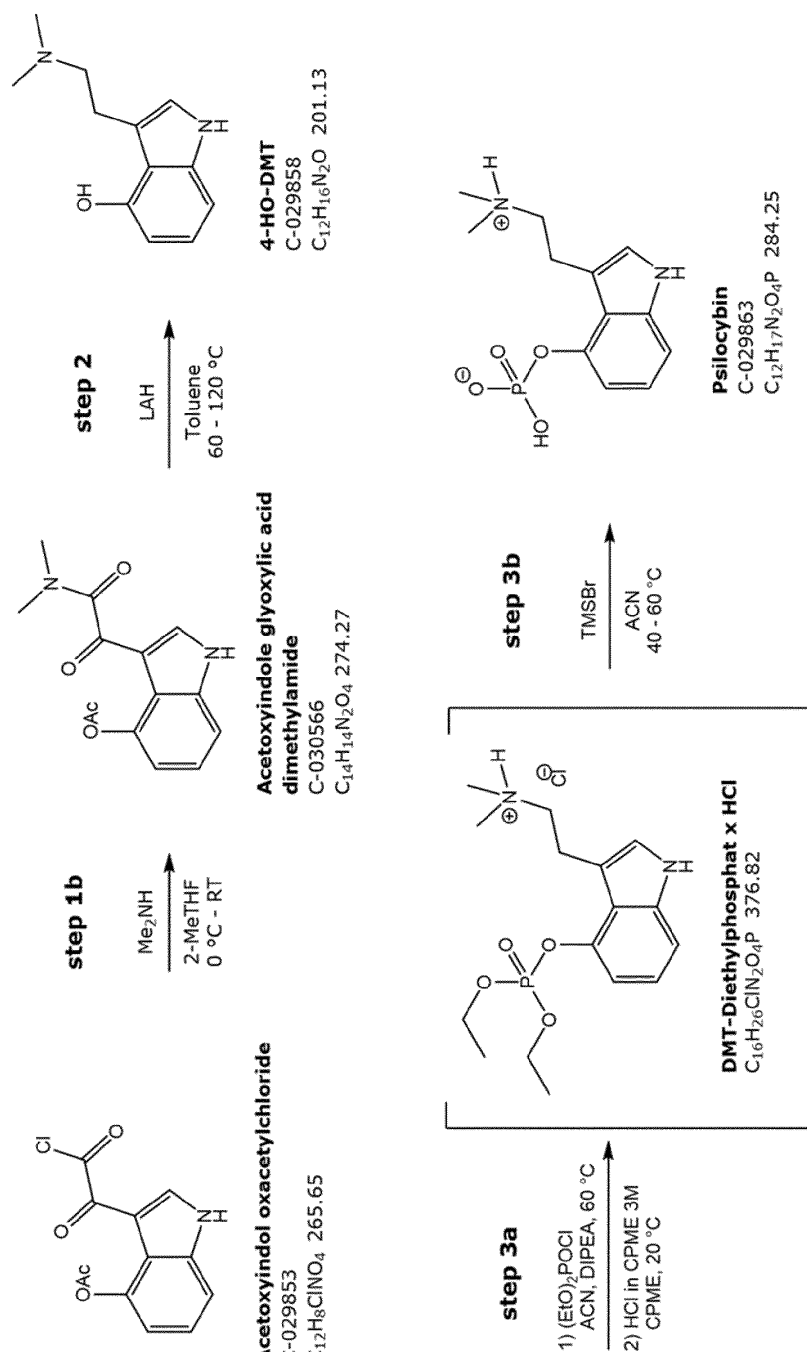


Figure 9.

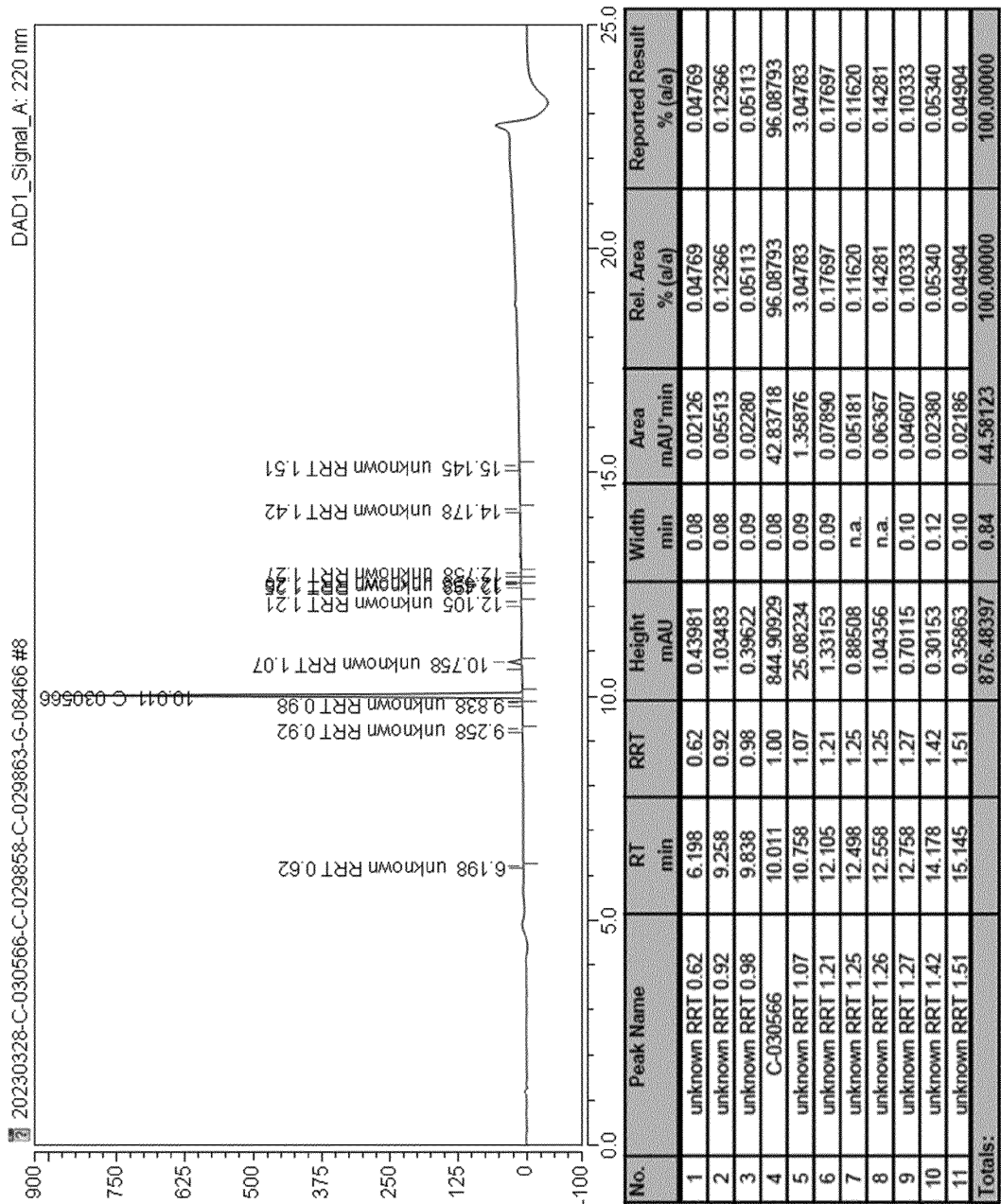


Figure 10 part 1.

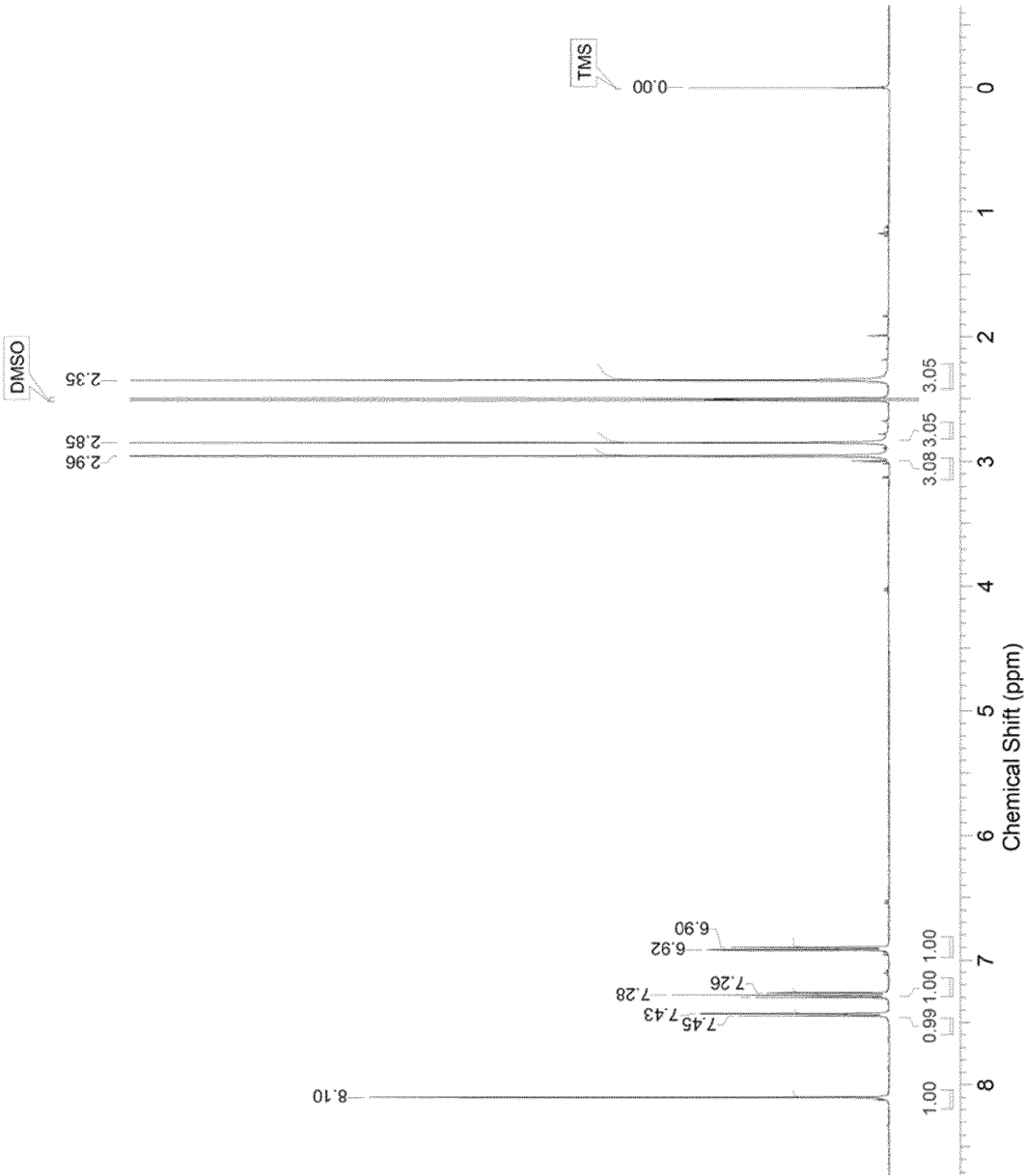


Figure 10 part 1.

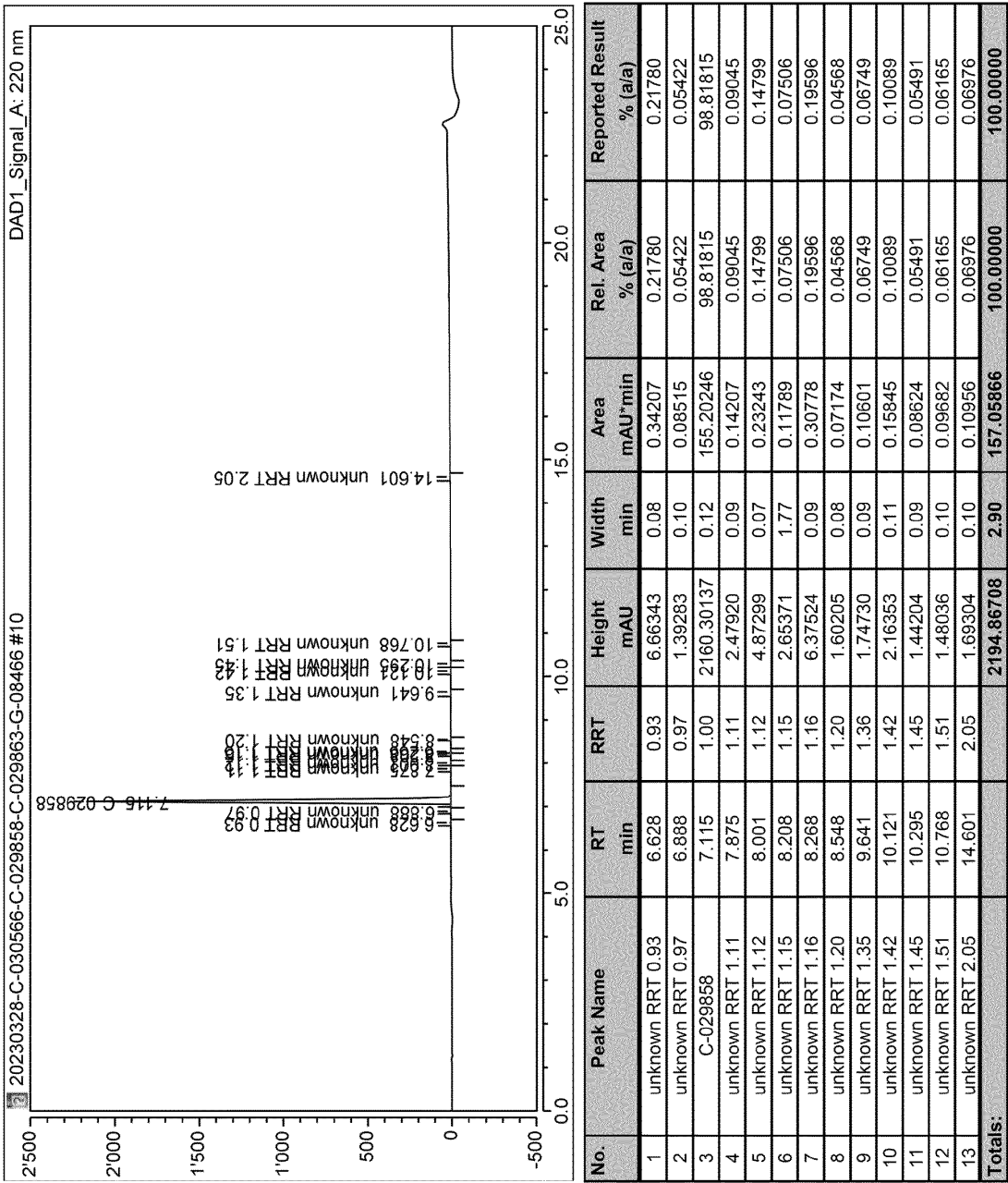


Figure 11 part 1.

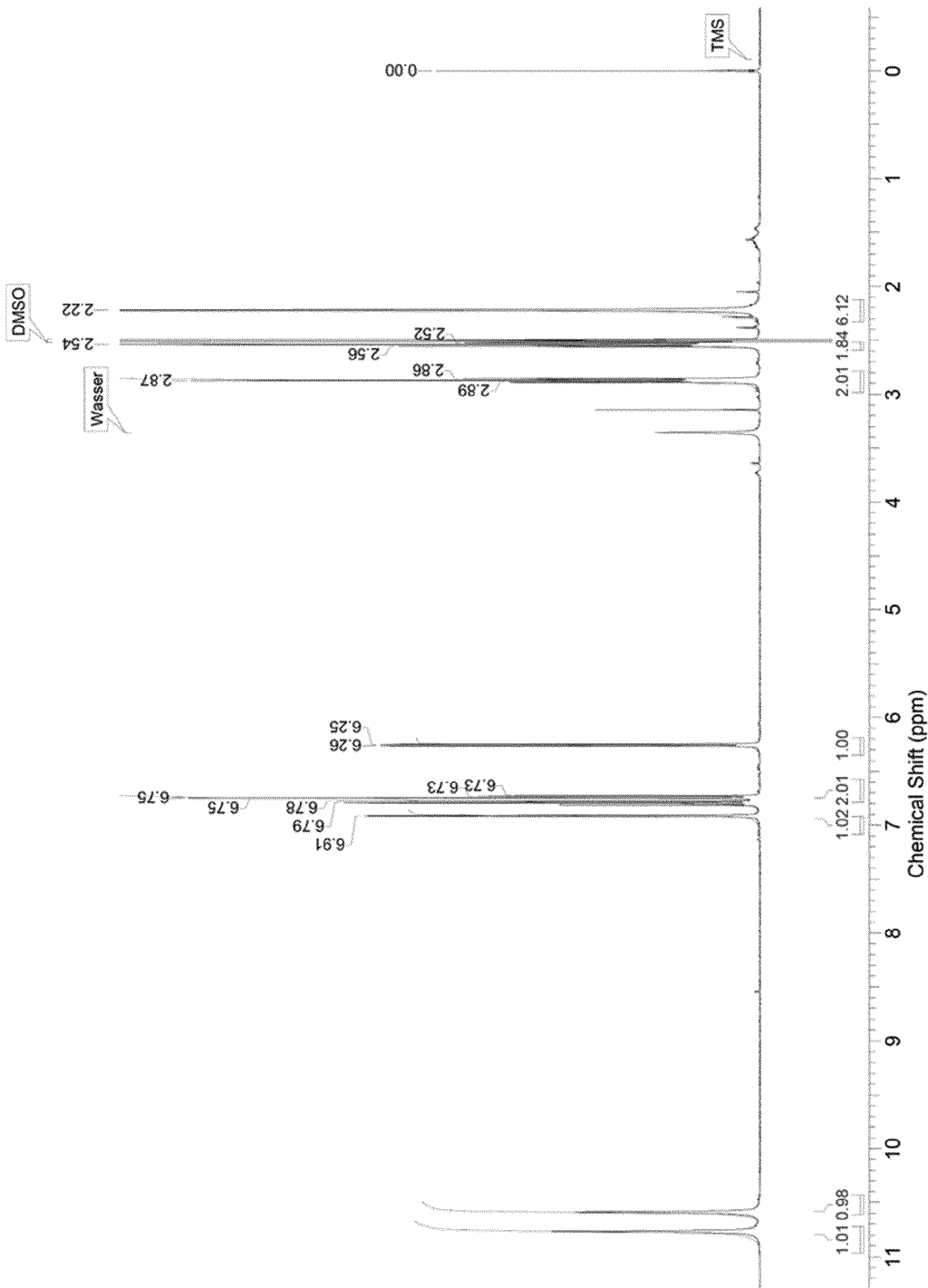


Figure 11 part 2.

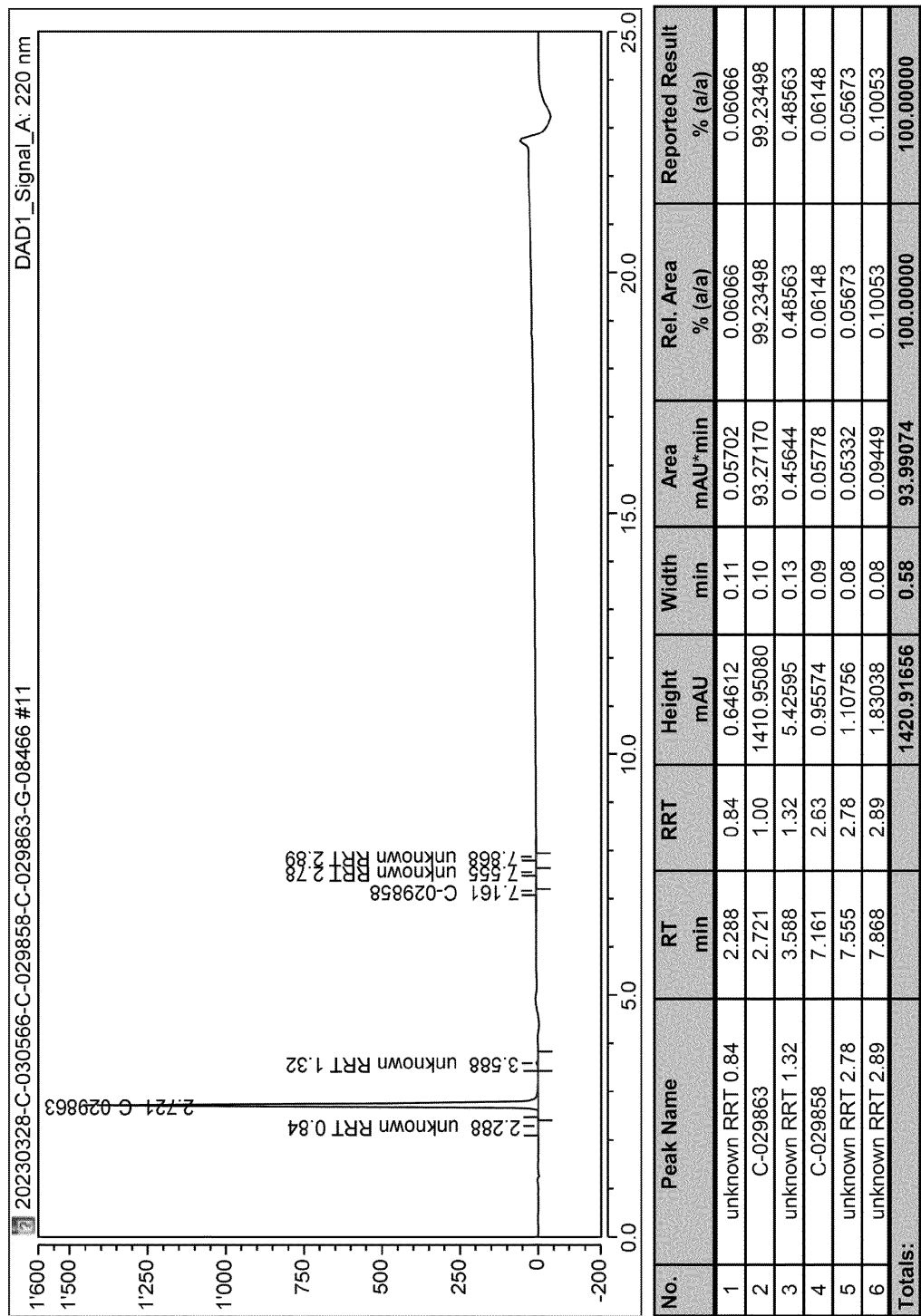


Figure 12 part 1.

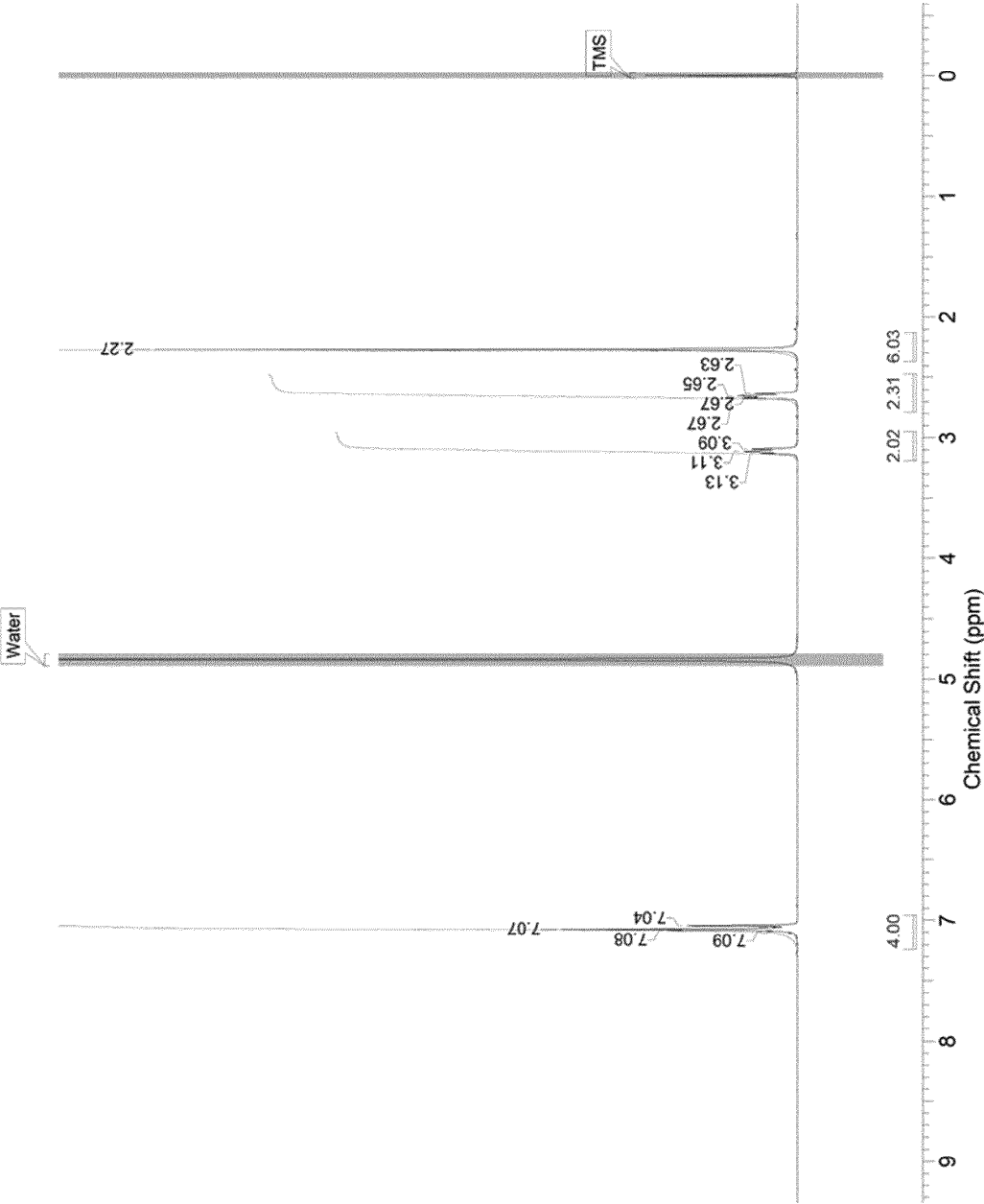


Figure 12 part 2.