Supporting Information II (LC-MS and NMR characterization)

"Click" synthesis of vinyl sulfone-containing small molecule inhibitors targeting caspases

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1. Summary of Characterizations of "Click" Inhibitors

#	ID of "Click" Product	Azide Used	LCMS			^{1}H NMR (Scaled up and purified)
			Est % Purity*	Cal. MW	Obs. MW	
001	VSB-A1	A1	>95	515.13	516.1330	-
002	VSB-A2	A2	>95	515.13	516.1190	-
003	VSB-A3	A3	-	533.12	-	-
004	VSB-A4	A4	>95	533.12	534.1190	-
005	VSB-A5	A5	>95	529.14	530.1530	-
006	VSB-A6	A6	>95	529.14	530.1470	-
007	VSB-A7	A7	-	565.06	N.D.	-
008	VSB-A8	A8	>95	531.10	532.0890	-
009	VSB-A9	A9	>95	545.11	546.1020	-
010	VSB-A10	A10	-	565.06	-	-
011	VSB-A11	A11	>95	561.11	562.1000	-
012	VSB-A12	A12	>95	541.16	542.1530	-
013	VSB-B1	B1	>95	589.16	590.1540	-
014	VSB-B2	B2	>95	557.16	558.1460	-
015	VSB-B3	B3	>95	541.16	542.1540	-
016	VSB-B4	B4	>95	527.15	528.1420	-
017	VSB-B5	B5	>95	587.17	588.1550	-
018	VSB-B6	B6	>95	555.18	556.1840	-
019	VSB-B7	B7	>95	543.12	544.1300	-
020	VSB-B8	B8	>98	581.12	582.1320	-
021	VSB-B9	B9	>95	527.15	528.1520	-
022	VSB-B10	B10	>95	557.16	558.1480	-
023	VSB-B11	B11	>95	589.16	590.1490	-
024	VSB-B12	B12	>95	527.15	528.1400	-
025	VSB-C1	C1	>95	583.21	584.2160	-

026	VSB-C2	C2	-	623.12	-	-
027	VSB-C3	C3	>95	581.12	582.1260	-
028	VSB-C4	C4	>95	525.17	526.1580	-
029	VSB-C5	C5	>95	525.17	526.1760	-
030	VSB-C6	C6	>95	553.20	554.2070	-
031	VSB-C7	C7	>95	525.17	526.1600	-
032	VSB-C8	C8	>95	567.22	568.2050	-
033	VSB-C9	C9	>95	569.16	570.1630	-
034	VSB-C10	C10	>95	569.16	570.1450	-
035	VSB-C11	C11	>90	595.25	596.2520	-
036	VSB-C12	C12	>95	547.15	548.1560	-
037	VSB-D1	D1	>90	597.17	598.1790	-
038	VSB-D2	D2	>95	551.18	552.1940	-
039	VSB-D3	D3	>95	599.17	623.1900	-
040	VSB-D4	D4	>95	561.17	562.1780	-
041	VSB-SA1	SA1	>95	589.13	590.1400	-
042	VSB-SA2	SA2	-	623.15	-	-
043	VSB-SA3	SA3	>95	581.08	582.0890	-
044	VSB-SA4	SA4	>95	587.04	588.0430	-
045	VSB-SA5	SA5	-	561.14	-	-
046	VSB-SA6	SA6	>95	583.10	584.1130	-
047	VSB-SA7	SA7	>95	583.10	584.1080	-
048	VSB-SA8	SA8	>95	575.15	576.1610	-
049	VSB-SA9	SA9	>95	565.11	566.1060	-
050	VSB-SA10	SA10	>95	565.11	566.1100	-
051	VSB-SA11	SA11	>95	565.11	566.1150	-
052	VSB-SA12	SA12	>95	579.13	580.1300	-
053	VSB-SB1	SB1	-	579.13	-	-
054	VSB-SB2	SB2	>95	589.17	590.1760	-
055	VSB-SB3	SB3	-	577.13	-	-
056	VSB-SB5	SB5	-	597.14	-	-
057	VSB-SB6	SB6	>95	597.14	598.1310	-
058	VSB-SB7	SB7	>95	592.10	598.136	-
059	VSB-SB8	SB8	>95	592.10	593.1040	-
060	VSB-SB9	SB9	>95	603.18	N.D.	-
061	VSB-SB10	SB10	>95	553.08	N.D.	-
062	VSB-SB11	SB11	>95	561.14	N.D.	-
063	VSB-SB12	SB12	>95	545.11	542.163	-
064	VSB-SC1	SC1	>95	541.16	546.117	-
065	VSB-SC2	SC2	>95	607.10	608.0910	-

066	VSB-SC3	SC3	>95	591.11	592.1140	-
067	VSP-A1	A1	>95	495.16	496.1660	-
068	VSP-A2	A2	>95	495.16	496.1660	-
069	VSP-A3	A3	-	513.15	-	-
070	VSP-A4	A4	>95	513.15	514.1590	-
071	VSP-A5	A5	>95	509.17	510.1790	-
072	VSP-A6	A6	>95	509.17	510.1850	-
073	VSP-A7	A7	-	545.09	N.D.	-
074	VSP-A8	A8	>95	511.13	512.1380	-
075	VSP-A9	A9	>95	525.14	526.1510	-
076	VSP-A10	A10	-	545.09	-	-
077	VSP-A11	A11	>95	541.14	542.1490	-
078	VSP-A12	A12	>95	521.19	522.2030	-
079	VSP-B1	B1	>95	569.19	570.2080	-
080	VSP-B2	B2	>95	537.19	538.1980	-
081	VSP-B3	B3	>95	521.19	522.2030	-
082	VSP-B4	B4	>95	507.18	508.1840	-
083	VSP-B5	B5	>95	567.20	568.2090	-
084	VSP-B6	B6	>95	535.21	536.2190	-
085	VSP-B7	B7	>95	523.16	524.1650	-
086	VSP-B8	B8	>95	561.15	562.1610	-
087	VSP-B9	B9	>95	507.18	508.1820	-
088	VSP-B10	B10	>95	537.19	538.1970	-
089	VSP-B11	B11	>95	569.19	570.1990	-
090	VSP-B12	B12	>95	507.18	508.1870	-
091	VSP-C1	C1	>95	563.24	564.2460	-
092	VSP-C2	C2	-	603.16	-	-
093	VSP-C3	C3	>95	561.15	562.1590	-
094	VSP-C4	C4	>95	505.20	506.2050	-
095	VSP-C5	C5	>95	505.20	506.2090	-
096	VSP-C6	C6	>95	533.23	534.2340	-
097	VSP-C7	C7	>95	505.20	506.2100	-
098	VSP-C8	C8	>95	547.25	548.2530	-
099	VSP-C9	C9	>95	549.19	550.1900	-
100	VSP-C10	C10	-	569.16	N.D.	-
101	VSP-C11	C11	>95	575.28	576.2840	-
102	VSP-C12	C12	>95	527.18	528.1890	-
103	VSP-D1	D1	>95	577.20	578.1940	-
104	VSP-D2	D2	>90	531.22	532.2240	-
105	VSP-D3	D3	-	579.20	N.D.	-

106	VSP-D4	D4	>95	541.20	542.2100	-
107	VSP-SA1	SA1	>95	569.16	570.1690	-
108	VSP-SA2	SA2	>95	603.18	604.1910	-
109	VSP-SA3	SA3	>95	561.11	562.1210	-
110	VSP-SA4	SA4	>95	567.07	568.0750	-
111	VSP-SA5	SA5	>95	541.17	542.1760	-
112	VSP-SA6	SA6	>95	563.13	563.1300	-
113	VSP-SA7	SA7	>95	563.13	564.1370	-
114	VSP-SA8	SA8	>95	555.18	556.1870	-
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117	VSP-SA11	SA11	>95	545.14	546.1440	-
118	VSP-SA12	SA12	>95	559.16	560.1570	-
119	VSP-SB1	SB1	>95	559.16	560.1680	-
120	VSP-SB2	SB2	-	569.20	N.D.	√
121	VSP-SB3	SB3	>95	557.16	558.1640	-
122	VSP-SB5	SB5	-	577.17	-	-
123	VSP-SB6	SB6	>95	597.14	N.D.	-
124	VSP-SB7	SB7	>95	572.14	578.1670	-
125	VSP-SB8	SB8	>95	572.14	573.1420	-
126	VSP-SB9	SB9	>95	583.21	N.D.	-
127	VSP-SB10	SB10	>95	533.11	N.D.	-
128	VSP-SB11	SB11	>95	541.17	N.D.	-
129	VSP-SB12	SB12	>95	525.14	N.D.	-
130	VSP-SC1	SC1	-	521.19	-	-
131	VSP-SC2	SC2	>98	587.14	588.1330	-
132	VSP-SC3	SC3	>90	571.14	572.1340	-
133	Ald-A1	A1	-	363.10	364.1013	-
134	Ald-A2	A2	-	363.10	364.1067	-
135	Ald-A3	A3	-	381.09	N.D.	-
136	Ald-A4	A4	-	381.09	N.D.	-
137	Ald-A5	A5	-	377.11	378.1137	-
138	Ald-A6	A6	-	377.11	378.1246	-
139	Ald-A7	A7	-	379.07	-	-
140	Ald-A8	A8	-	379.07	380.0730	-
141	Ald-A9	A9	-	393.08	394.1000	-
142	Ald-A10	A10	-	413.03	-	-
143	Ald-A11	A11	-	409.08	410.0900	-
144	Ald-A12	A12	-	389.13	390.1450	-
145	Ald-B1	B1	-	437.13	438.1478	-

146	Ald-B2	B2	-	405.13	406.1385	-
147	Ald-B3	B3	-	389.13	390.1411	-
148	Ald-B4	B4	-	375.12	375.1200	-
149	Ald-B5	B5	-	435.14	436.1390	-
150	Ald-B6	B6	-	403.15	404.1530	-
151	Ald-B7	B7	-	391.10	392.0980	-
152	Ald-B8	B8	-	429.09	429.0900	-
153	Ald-B9	B9	-	375.12	376.1200	-
154	Ald-B10	B10	-	405.13	406.1330	-
155	Ald-B11	B11	-	437.13	438.1350	-
156	Ald-B12	B12	-	375.12	376.1250	-
157	Ald-C1	C1	-	431.18	432.1850	-
158	Ald-C2	C2	-	471.06	-	-
159	Ald-C3	C3	-	429.05	-	-
160	Ald-C4	C4	-	373.14	374.1450	-
161	Ald-C5	C5	-	373.14	374.1380	-
162	Ald-C6	C6	-	401.17	402.1750	-
163	Ald-C7	C7	-	373.14	374.1450	-
164	Ald-C8	C8	-	415.19	416.1840	-
165	Ald-C9	C9	-	417.15	-	-
166	Ald-C10	C10	-	417.13	418.1320	-
167	Ald-C11	C11	-	443.22	444.2150	-
168	Ald-C12	C12	-	415.15	-	-
169	Ald-D1	D1	-	445.14	446.1320	-
170	Ald-D2	D2	-	399.17	-	-
171	Ald-D3	D3	-	447.16	-	-
172	Ald-D4	D4	-	409.16	410.1510	-
173	Ald-SA1	SA1	-	437.10	438.1056	-
174	Ald-SA2	SA2	-	471.12	472.1228	-
175	Ald-SA3	SA3	-	429.05	430.0572	-
176	Ald-SA4	SA4	-	435.01	436.0099	-
177	Ald-SA5	SA5	-	409.11	410.1061	-
178	Ald-SA6	SA6	-	431.07	432.0739	-
179	Ald-SA7	SA7	-	431.07	432.0750	-
180	Ald-SA8	SA8	-	423.12	424.1260	-
181	Ald-SA9	SA9	-	413.08	414.0850	-
182	Ald-SA10	SA10	-	413.08	414.0860	-
183	Ald-SA11	SA11	-	413.08	414.0830	-
184	Ald-SA12	SA12	-	427.10	428.0990	-
185	Ald-SB1	SB1	-	427.10	428.0955	-

186	Ald-SB2	SB2	-	487.14	438.1403	-
187	Ald-SB3	SB3	-	425.10	425.1000	-
188	Ald-SB5	SB5	-	445.11	446.1185	-
189	Ald-SB6	SB6	-	445.11	446.1162	-
190	Ald-SB7	SB7	-	440.08	441.0760	-
191	Ald-SB8	SB8	-	440.08	441.0700	-
192	Ald-SB9	SB9	-	451.18	-	-
193	Ald-SB10	SB10	-	401.07	-	-
194	Ald-SB11	SB11	-	409.11	410.1100	-
195	Ald-SB12	SB12	-	393.11	-	-
196	Ald-SC1	SC1	-	389.13	390.1410	-
197	Ald-SC2	SC2	-	455.07	456.0900	-
198	Ald-SC3	SC3	-	439.08	N.D.	-

* Excluding the peak generated from excessive azide used

"-" = No characterizations (LCMS/NMR) were done

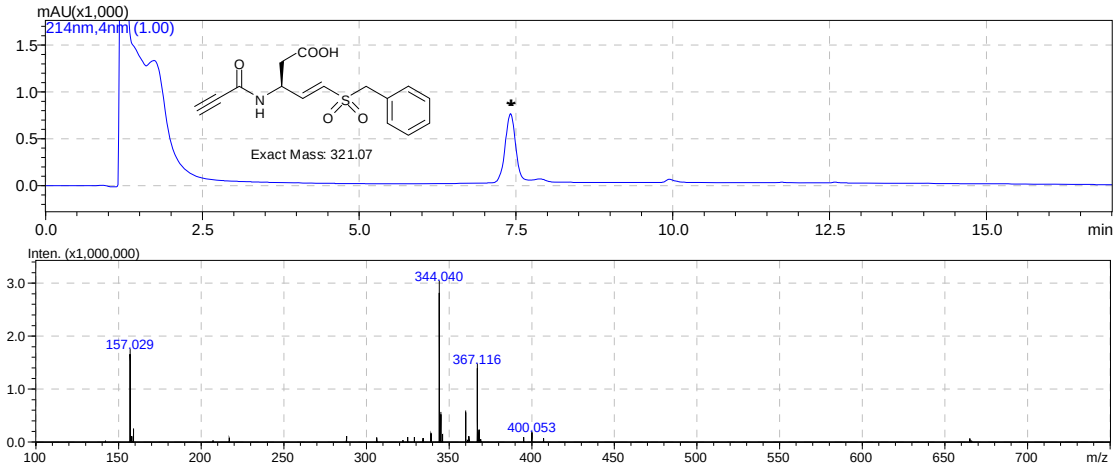
"N.D." = Although the product is correct, the molecular mass did not show up in the mass spectrometry (due to low ionization)

2. LC-MS profiles of “Click” inhibitors

2.1 LC-MS profiles of benzyl vinyl sulfone inhibitors (17)

All conditions are based on:
0 – 10 min: 20% B (1% TFA in ACN) → 80% B
10 – 15 min: 80% B (1% TFA in ACN) → 100% B
15 – 17 min: 100% B
(*) Starting materials; (Arrowed) desired product.

VSB-warhead:



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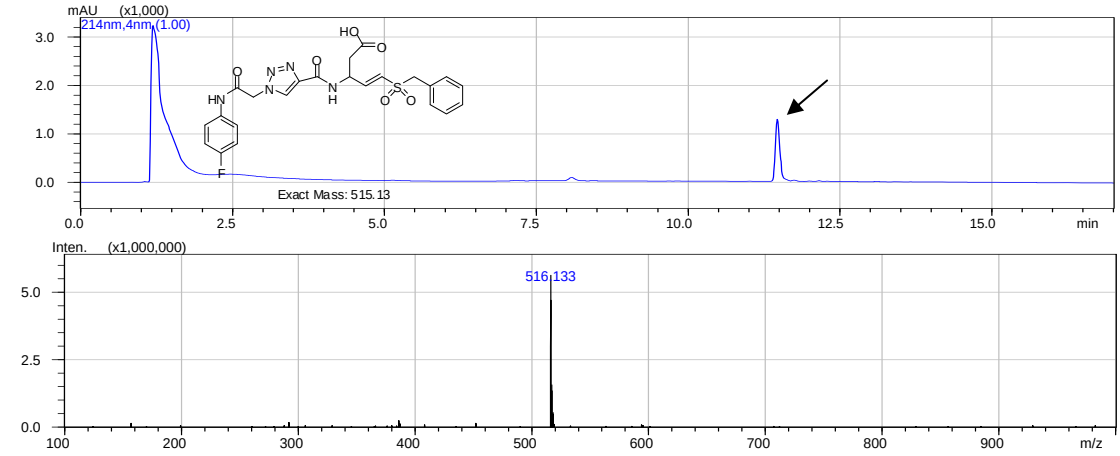

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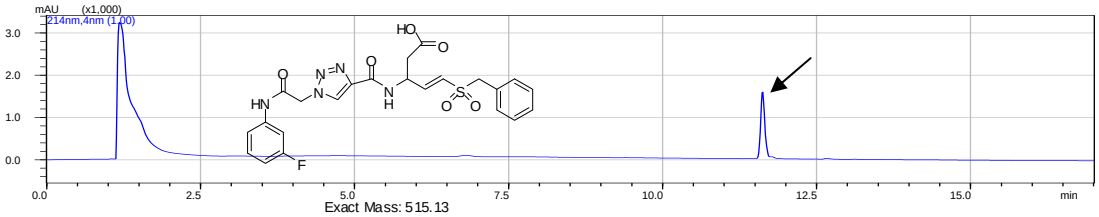

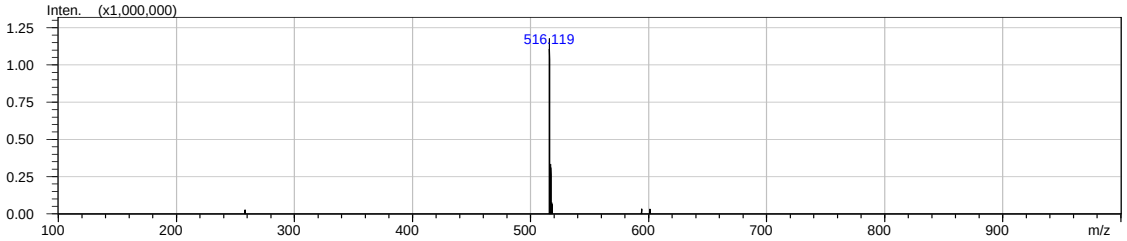

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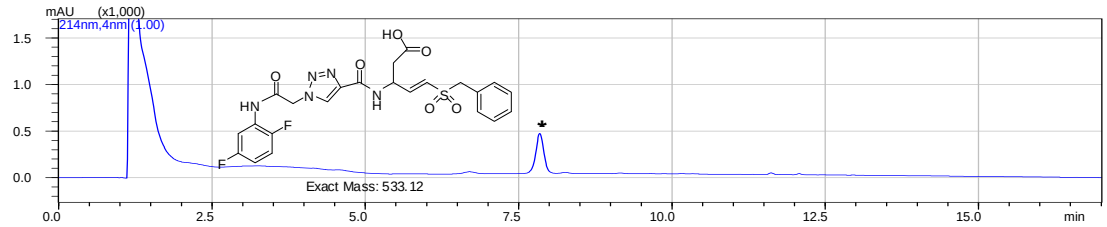

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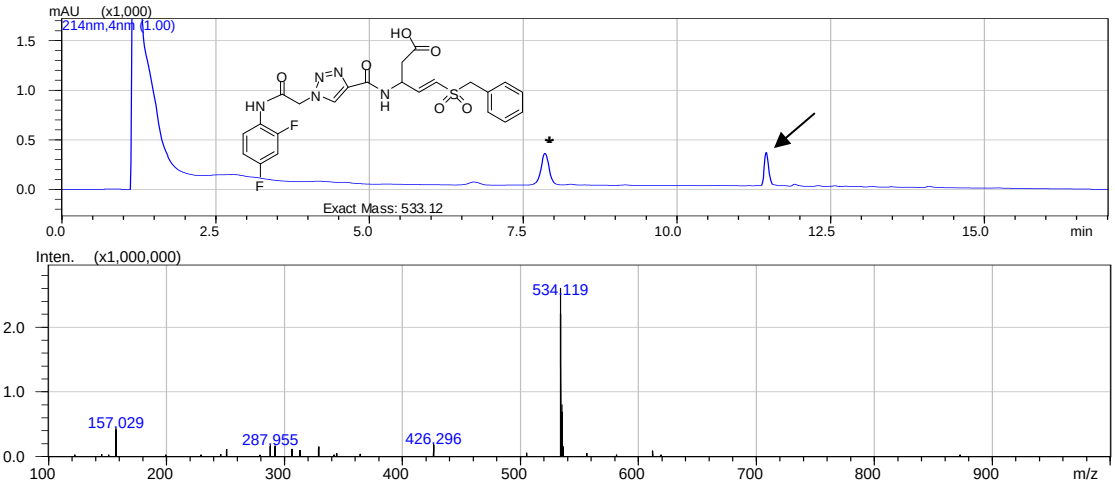

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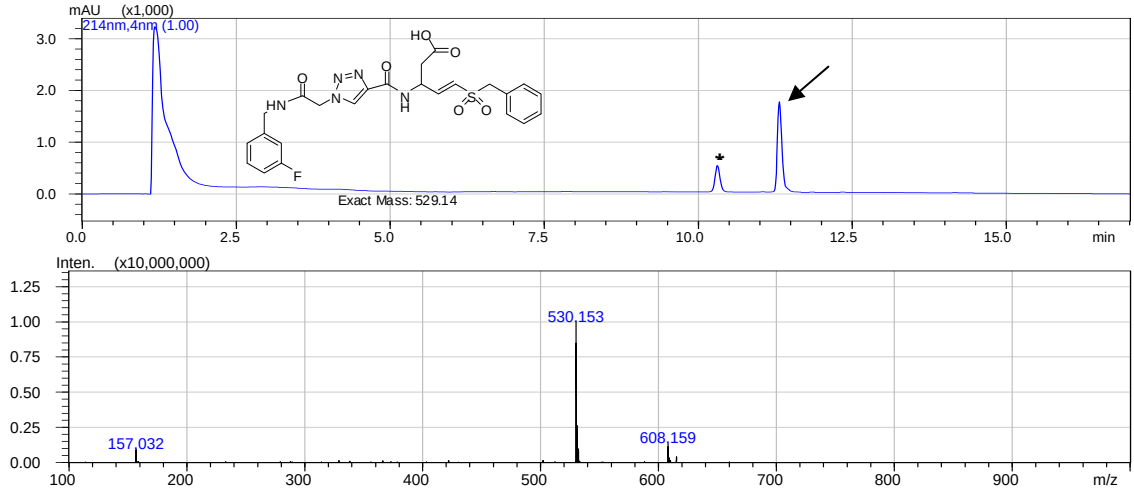

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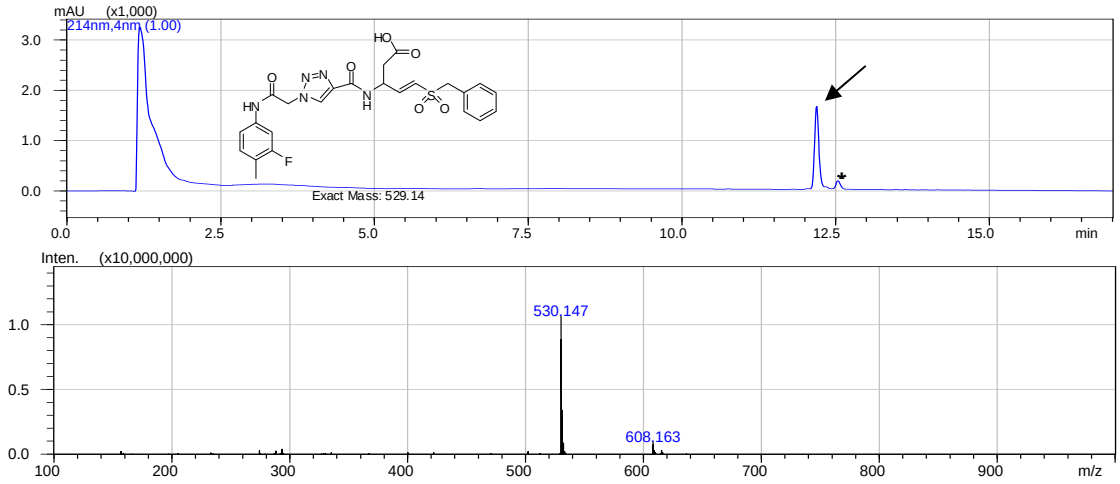

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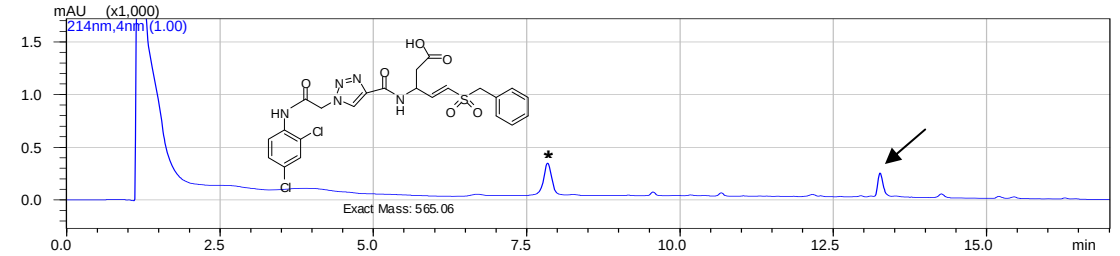

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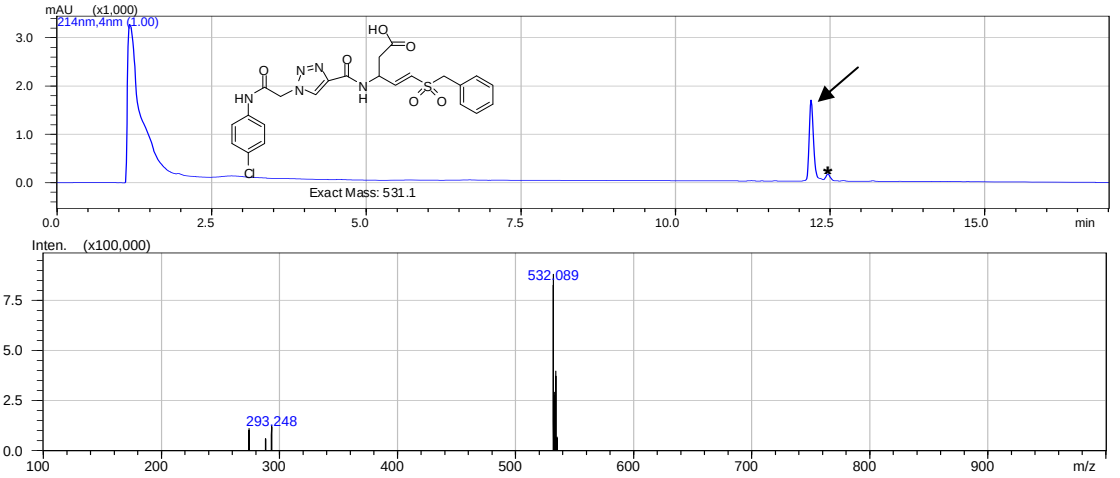

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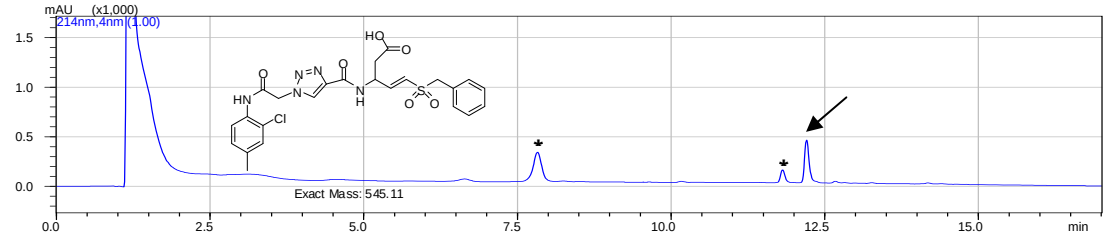

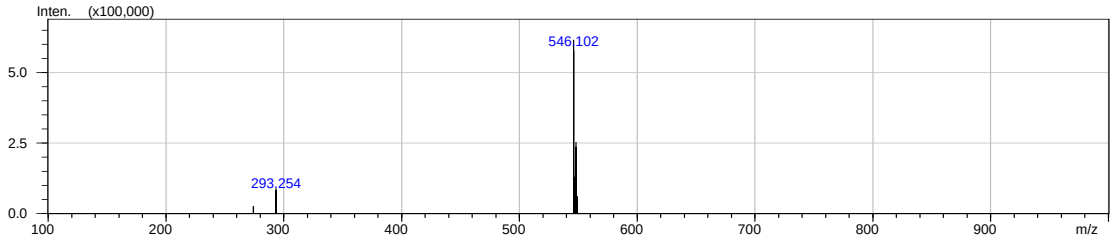

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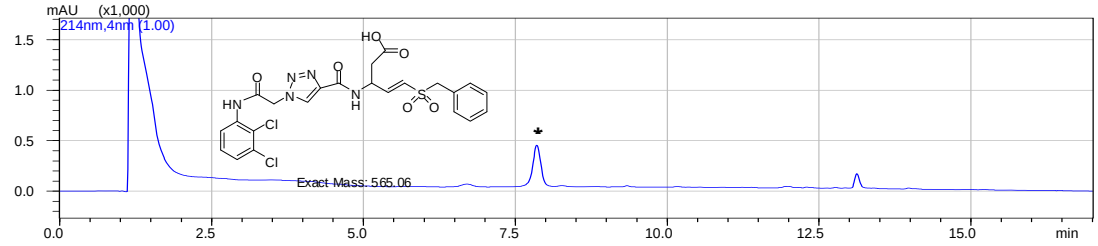

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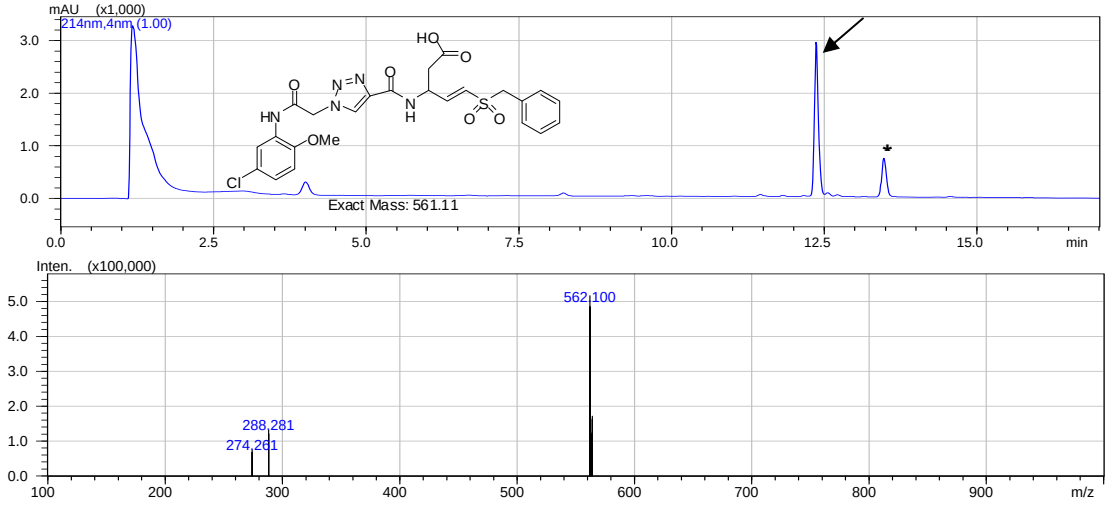

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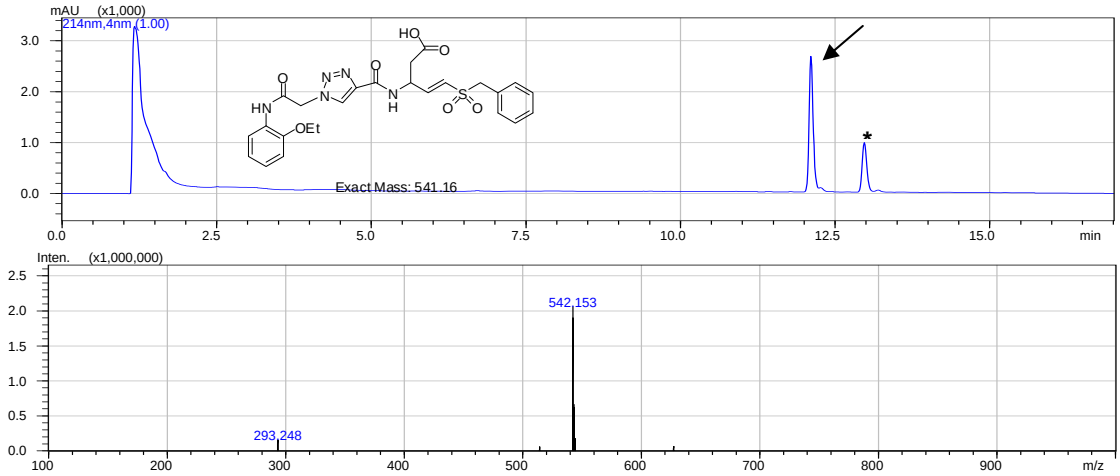

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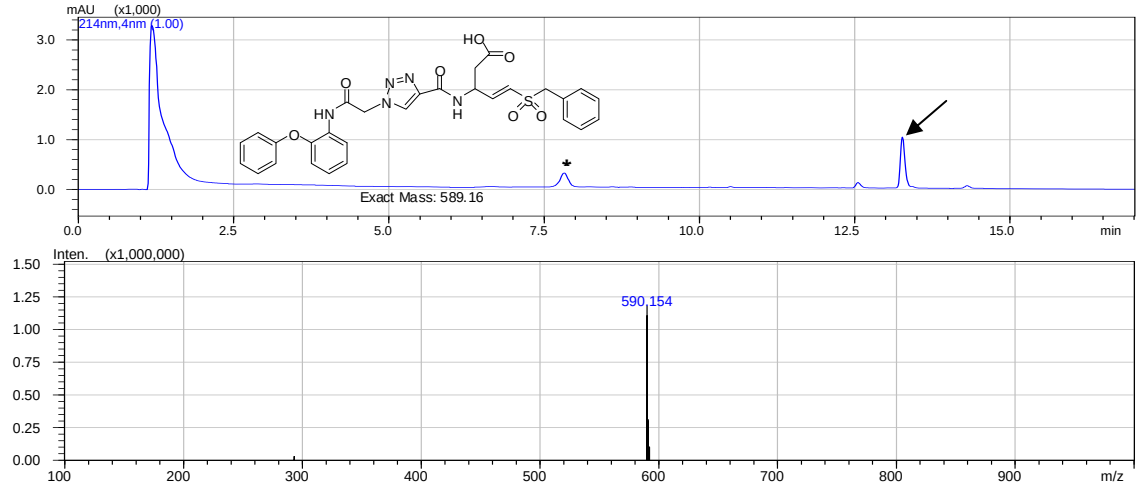

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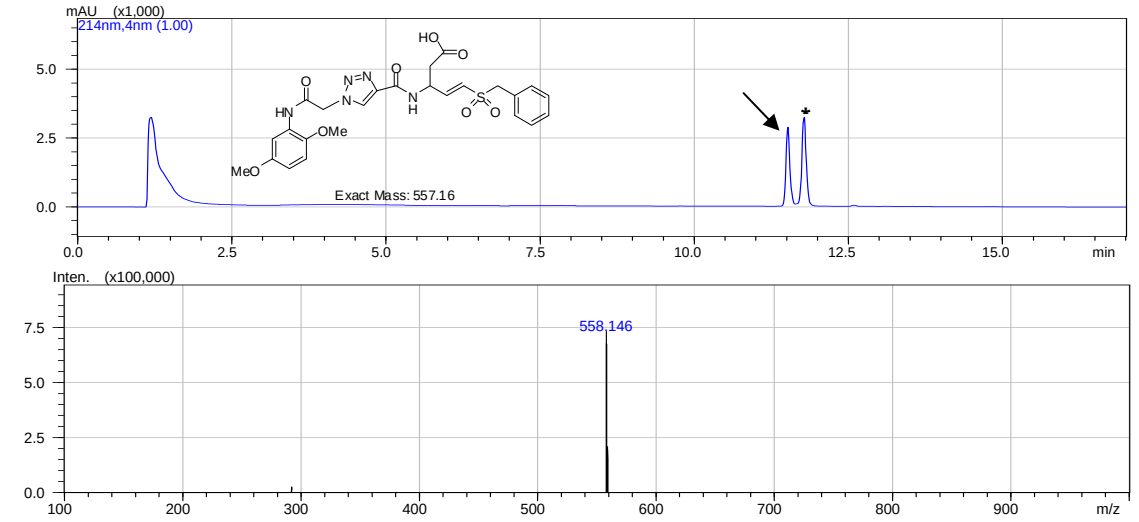

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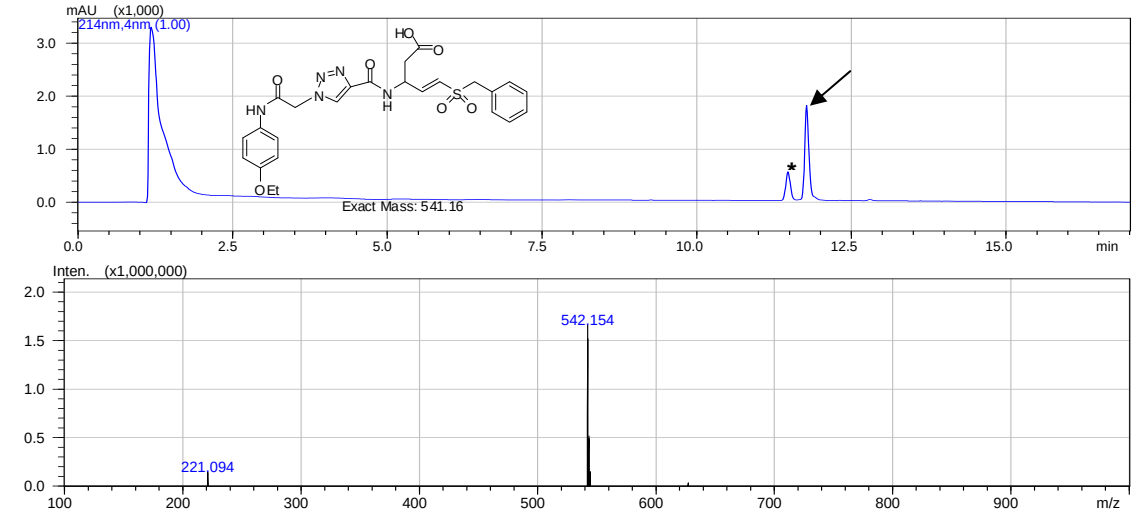

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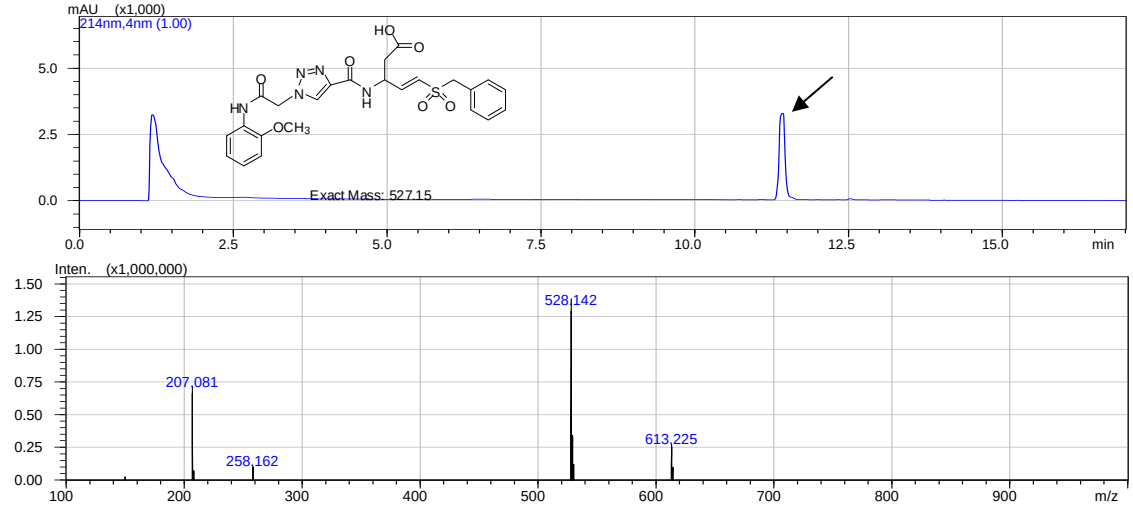

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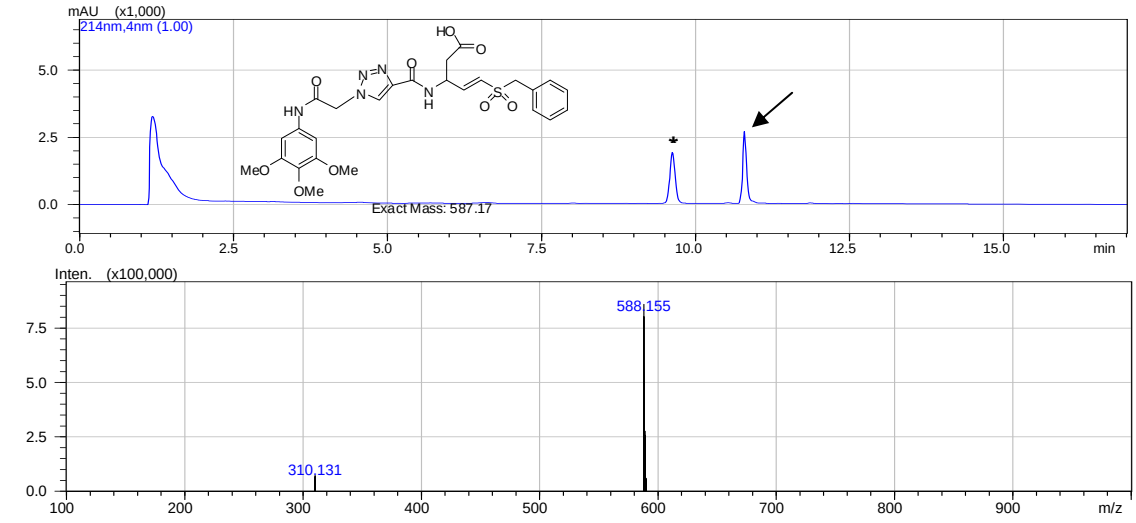

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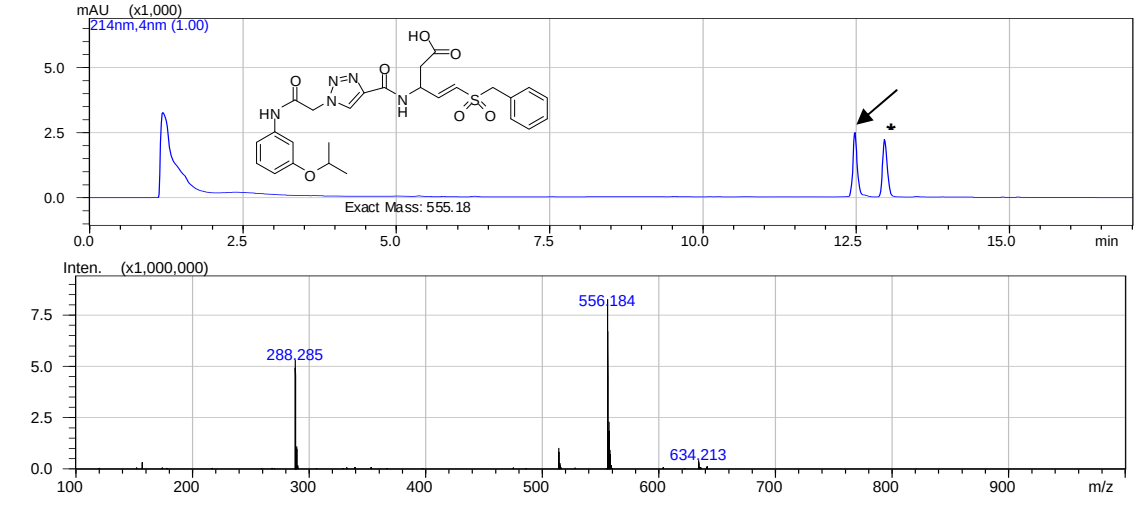

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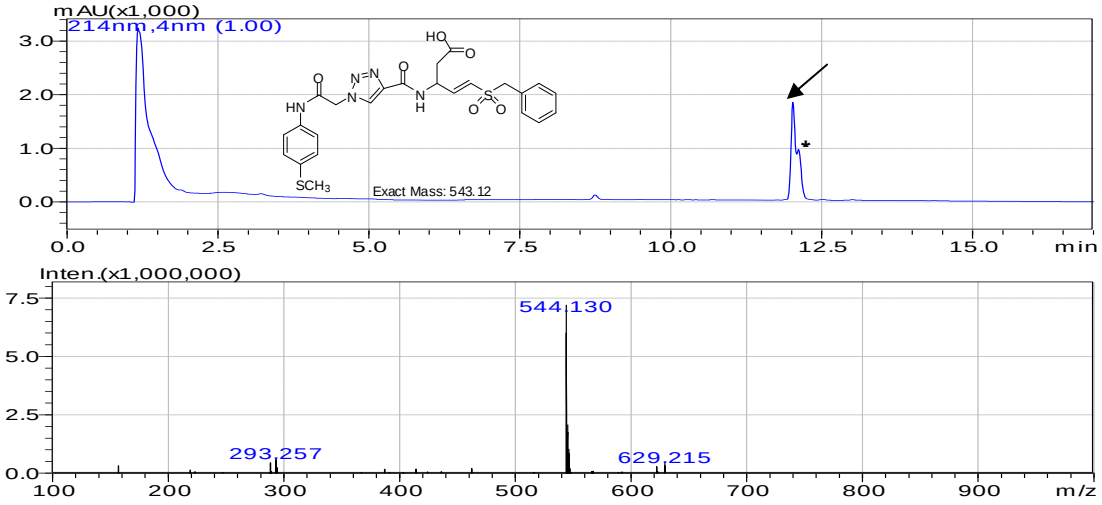

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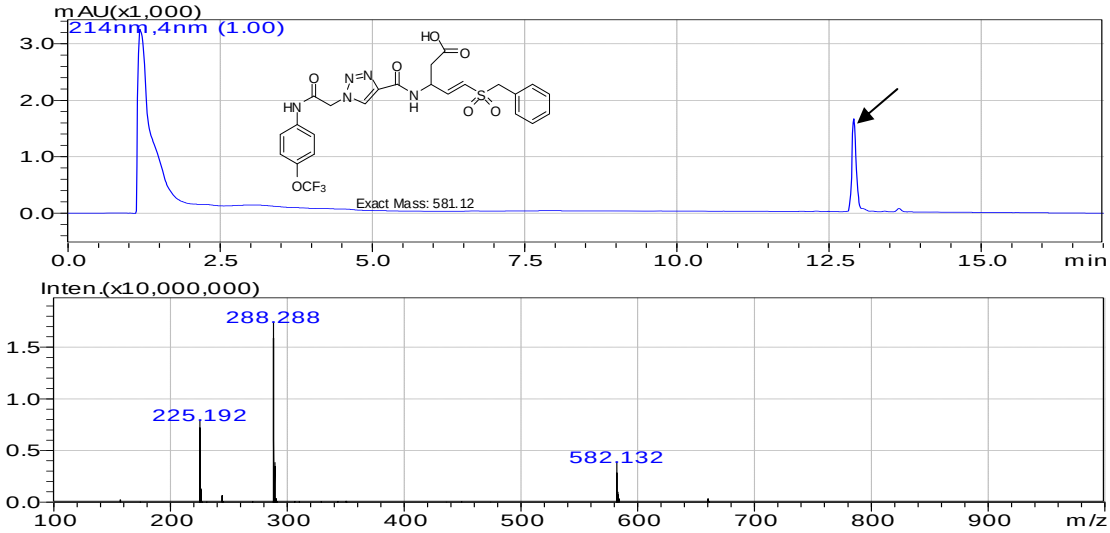

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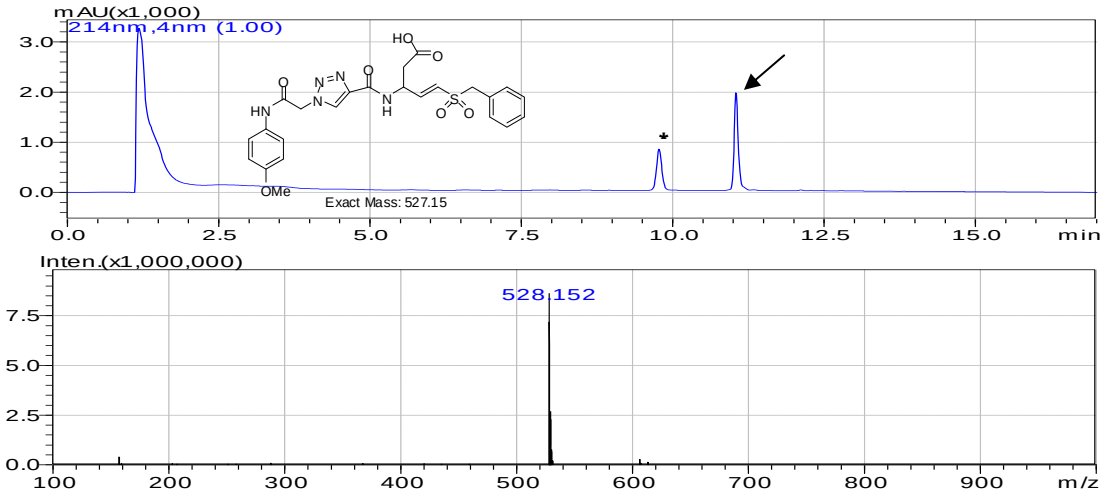

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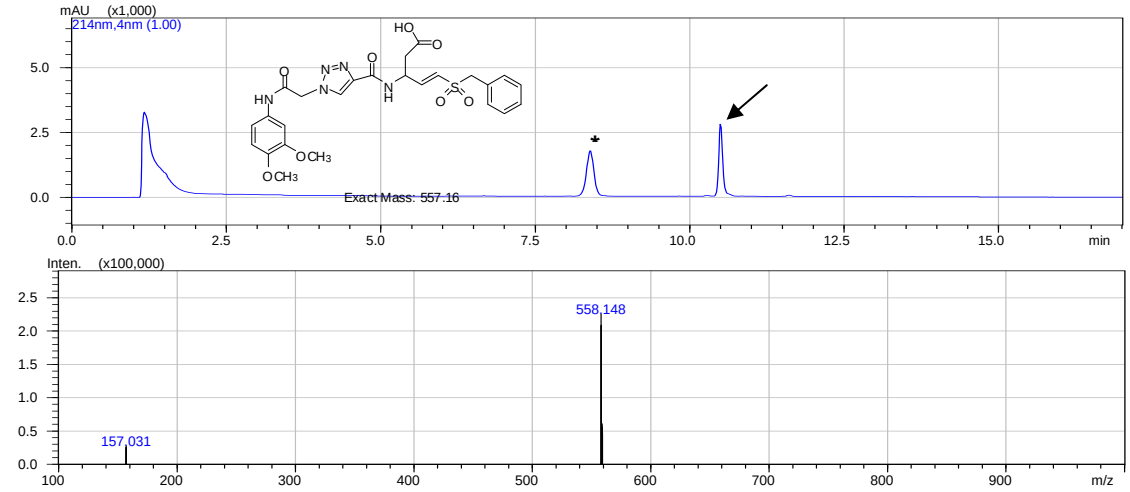

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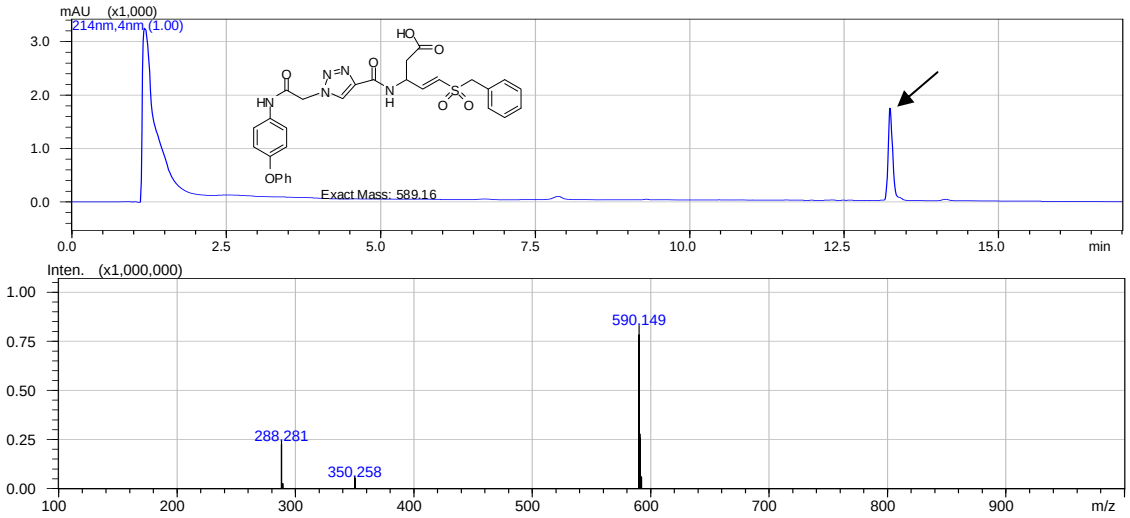

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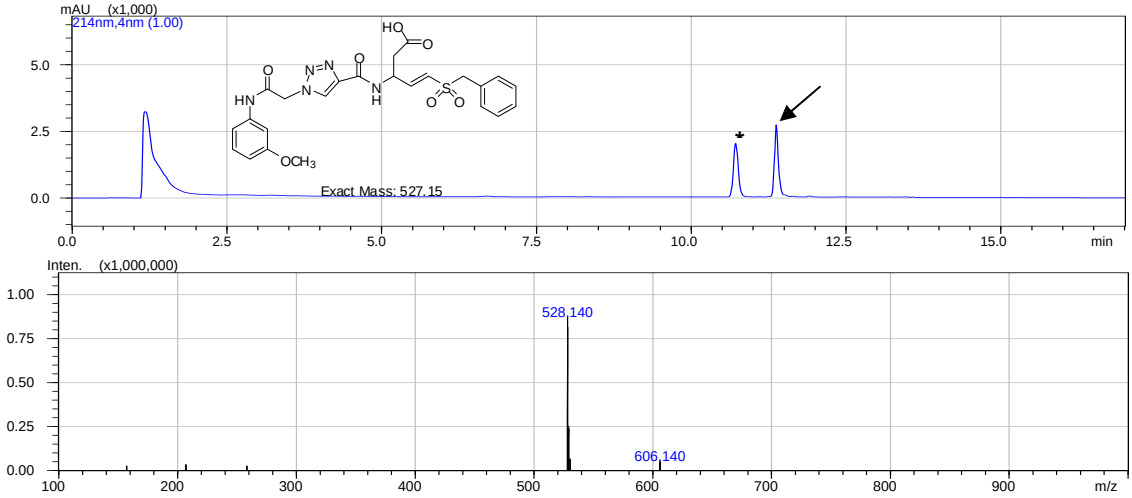

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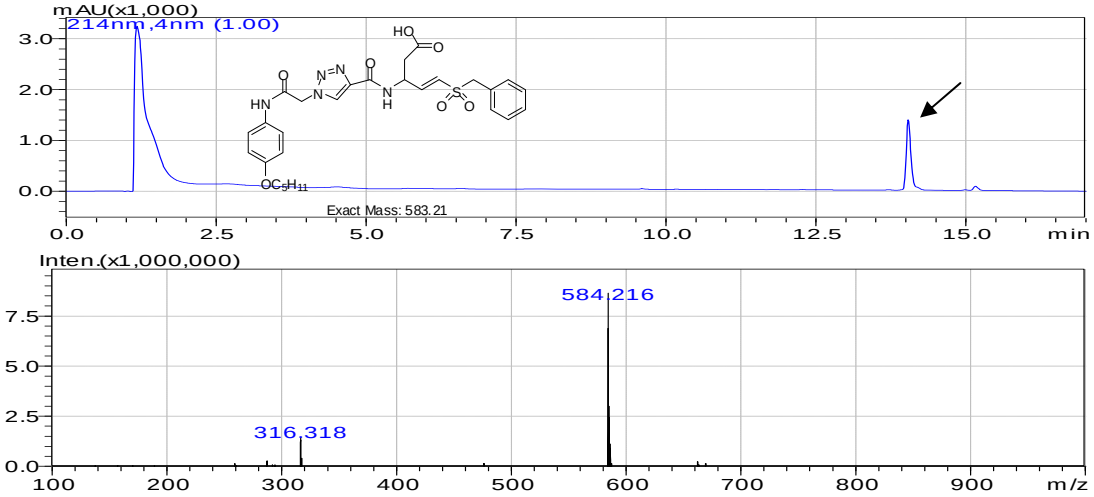

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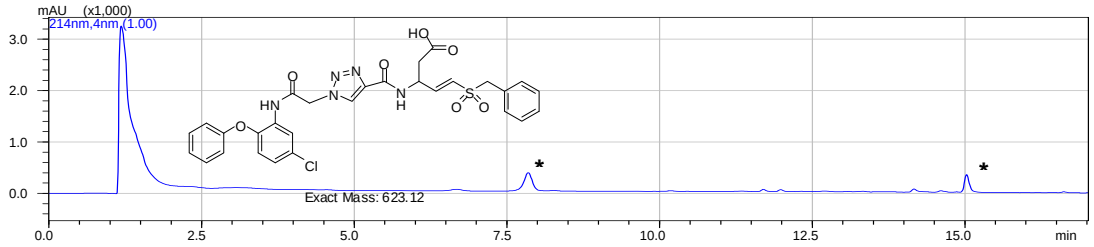

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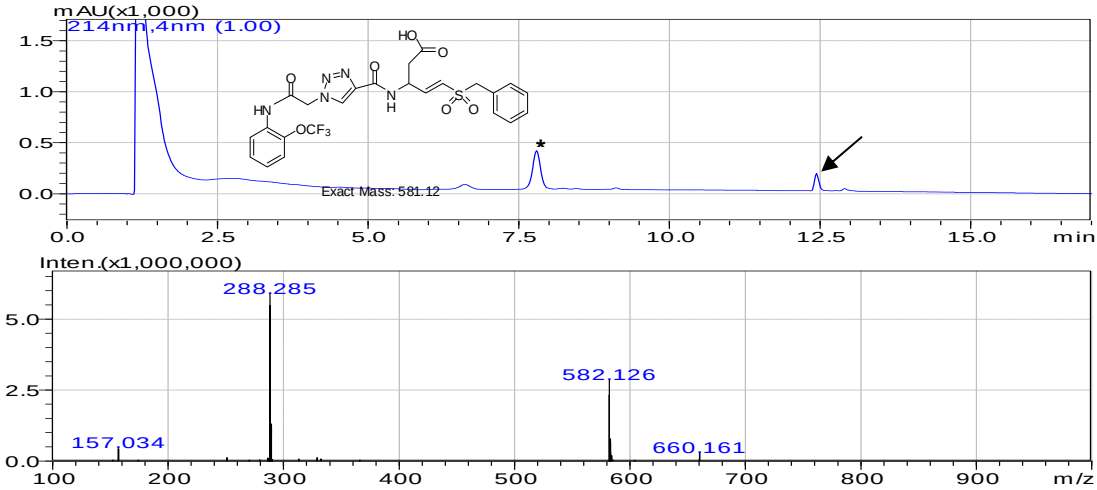

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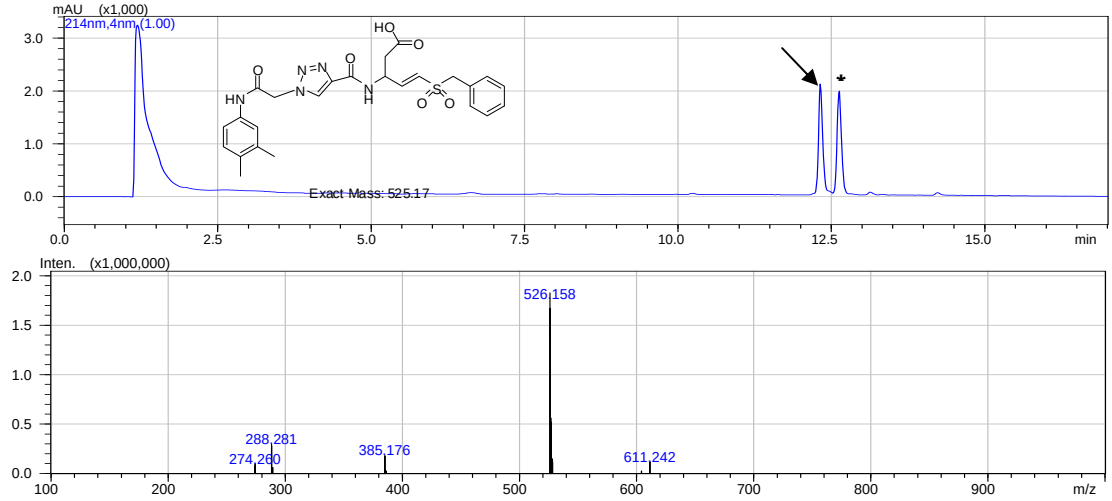

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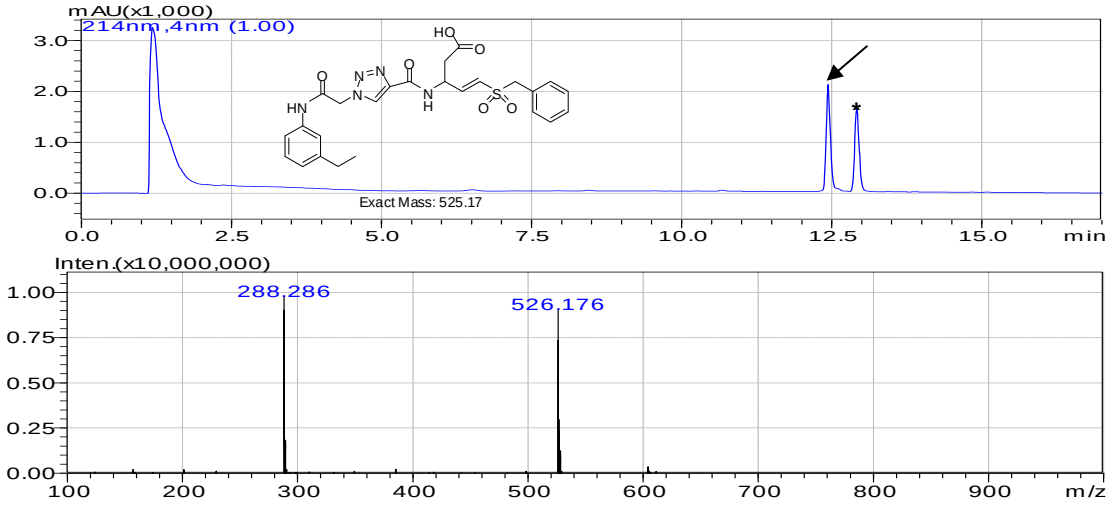

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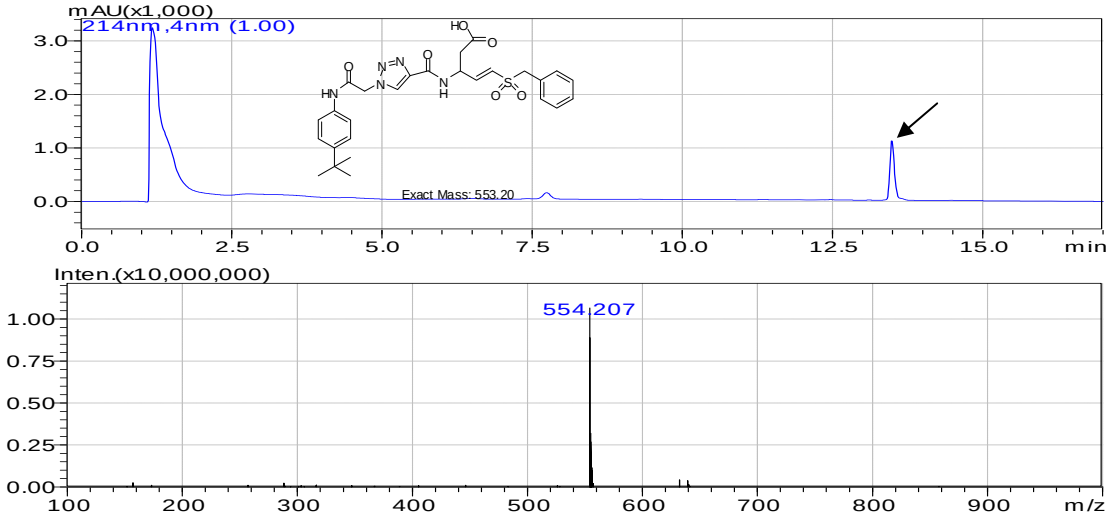

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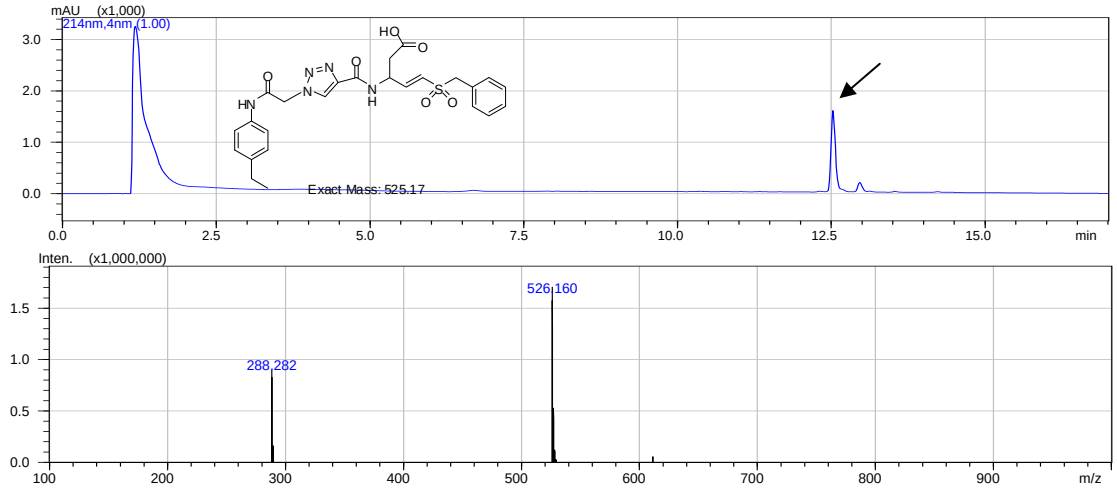

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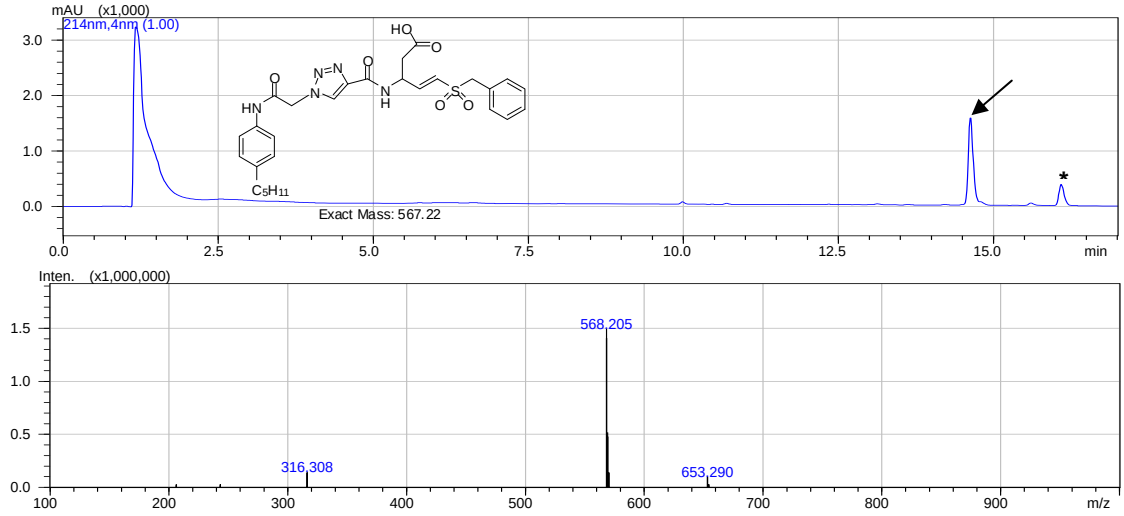

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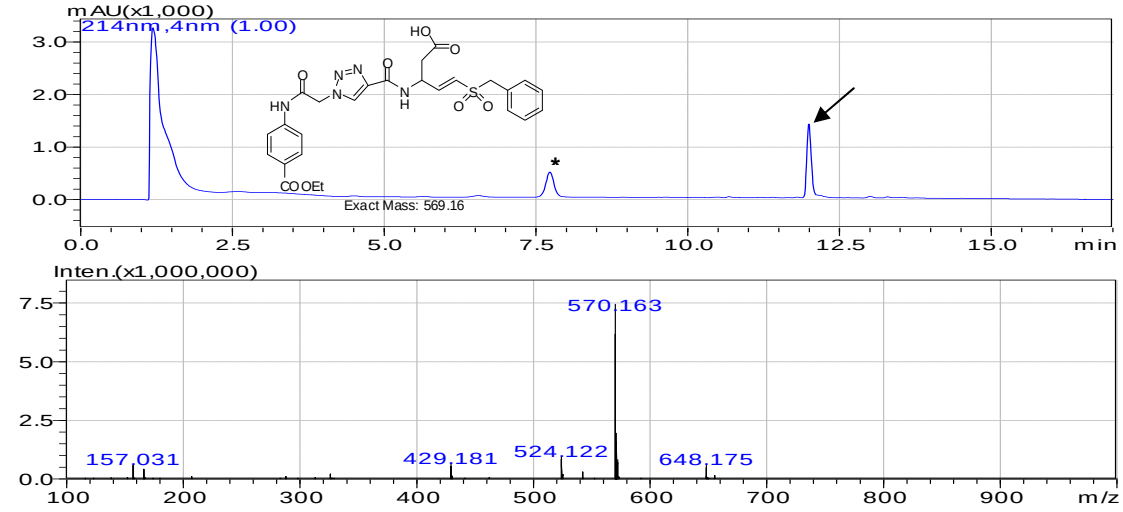

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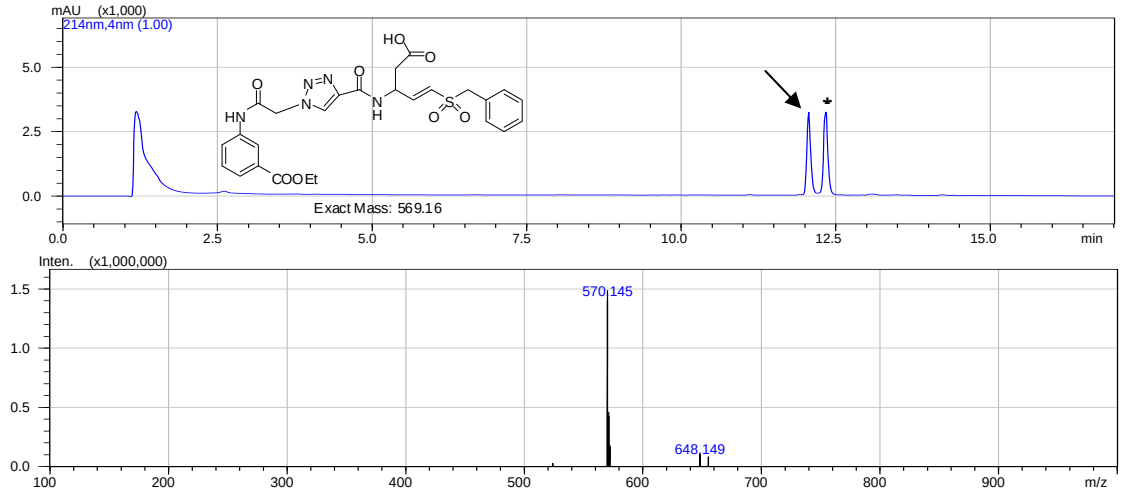

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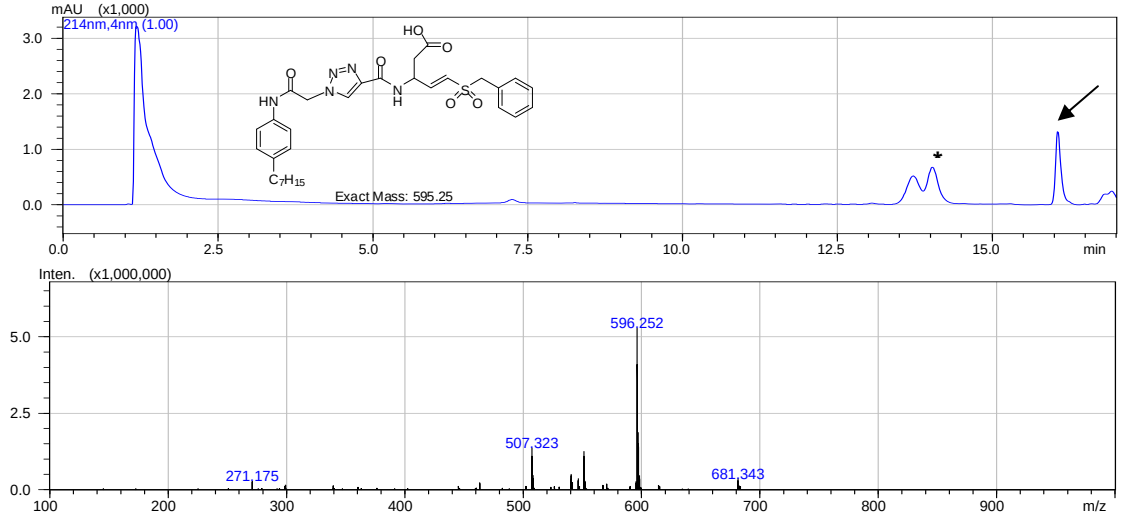

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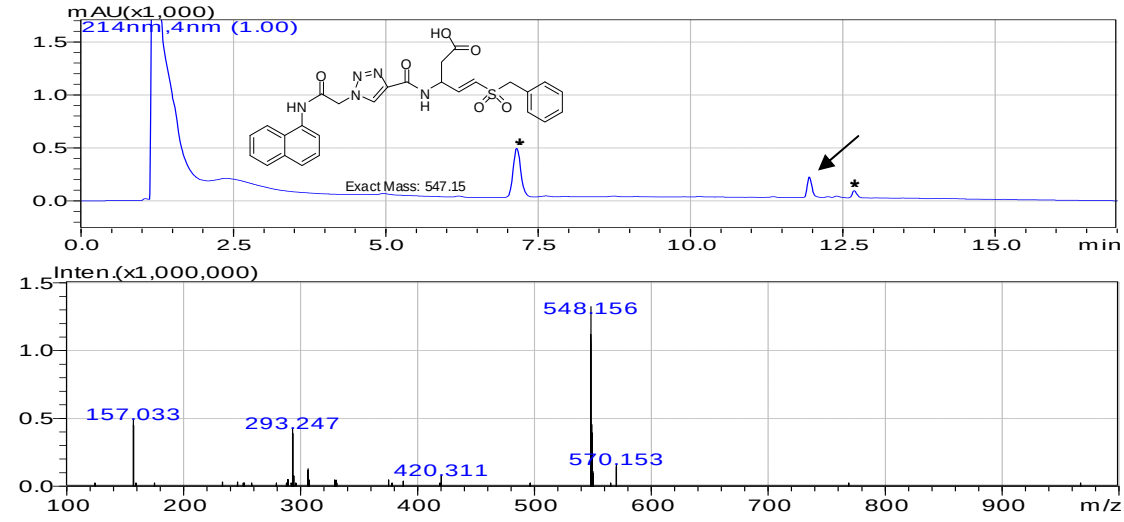

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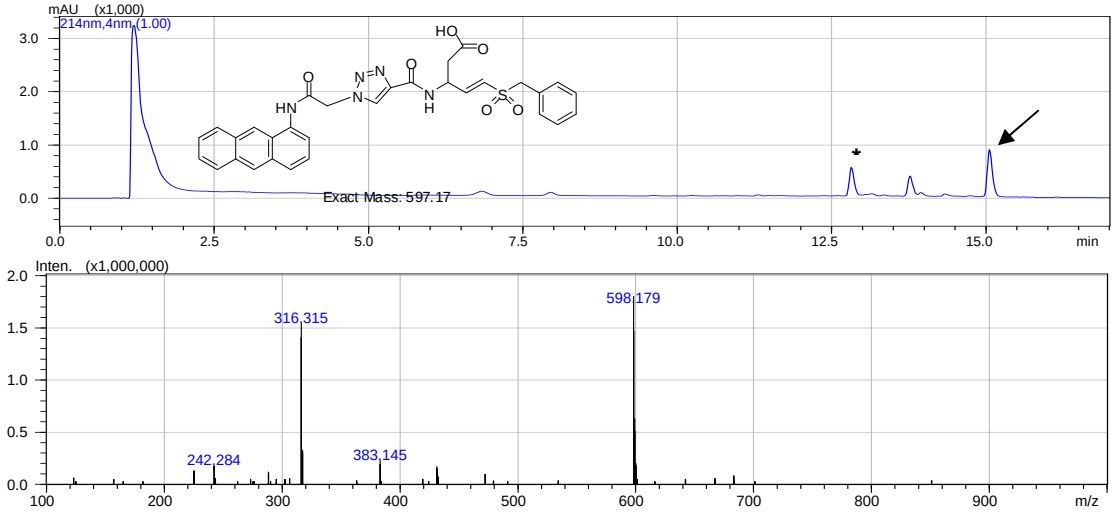

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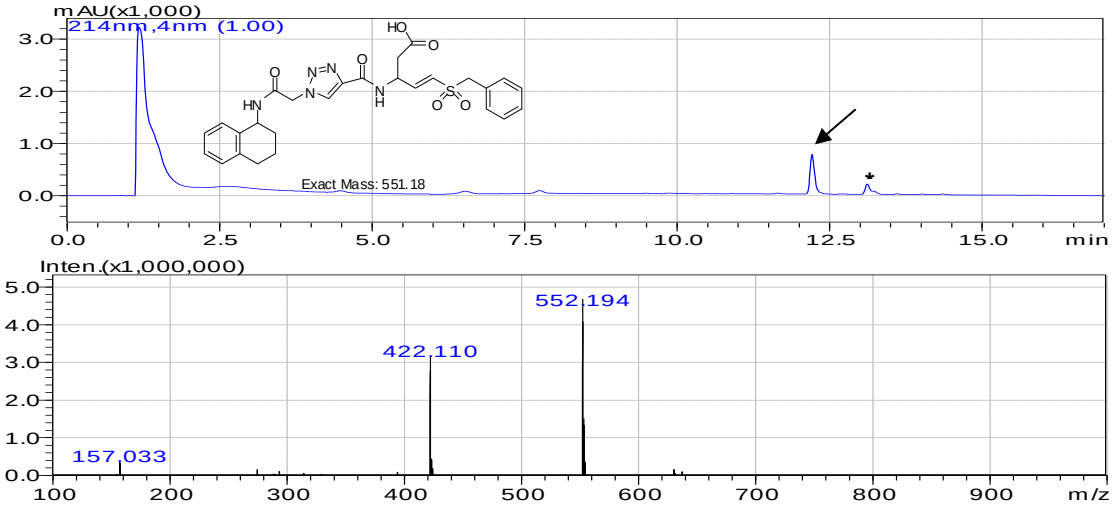

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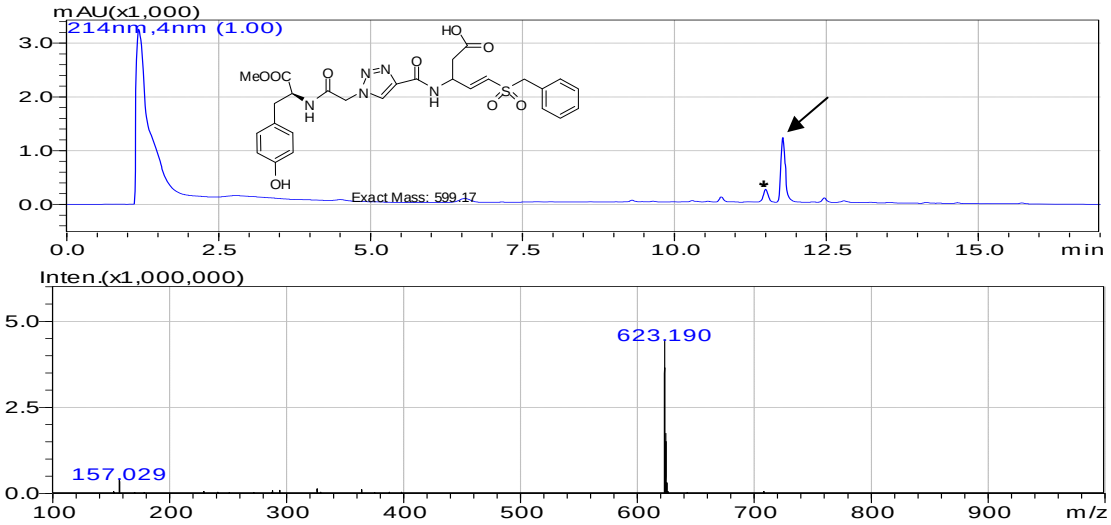

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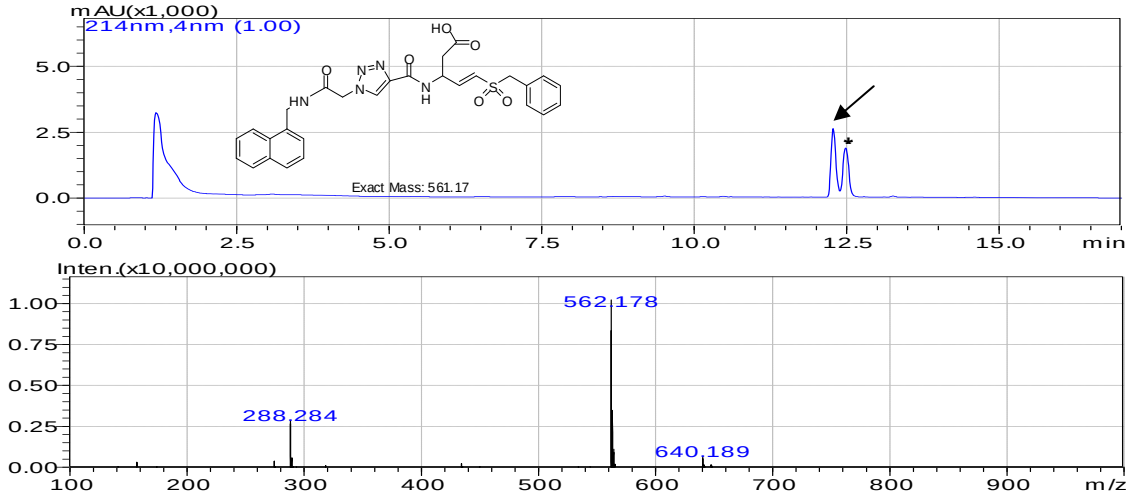

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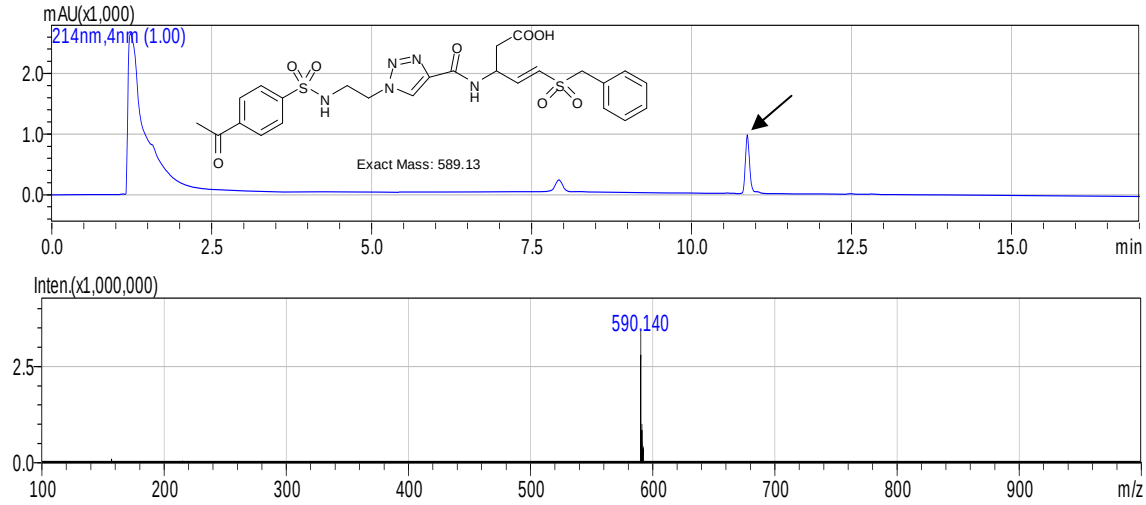

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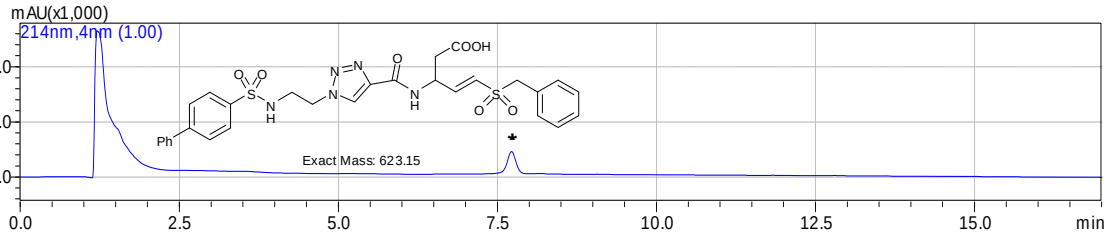

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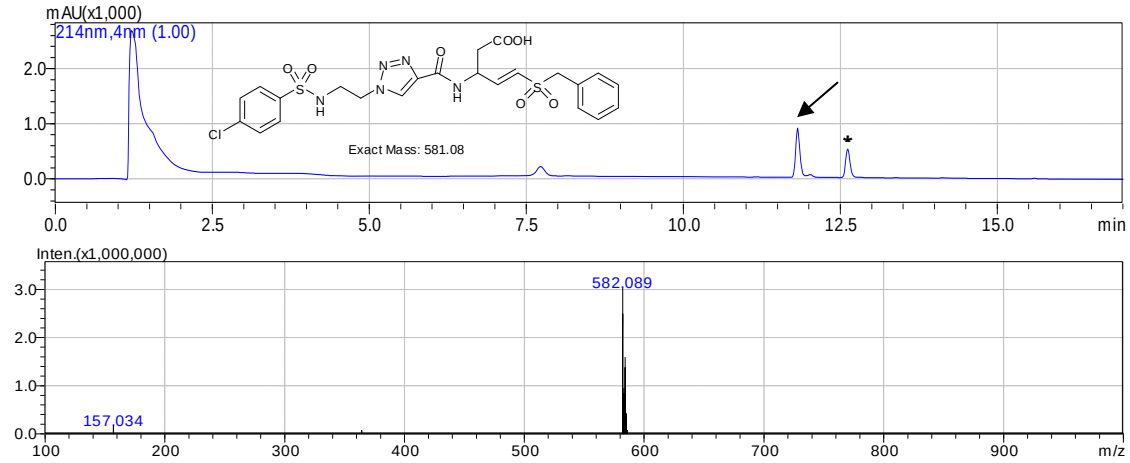

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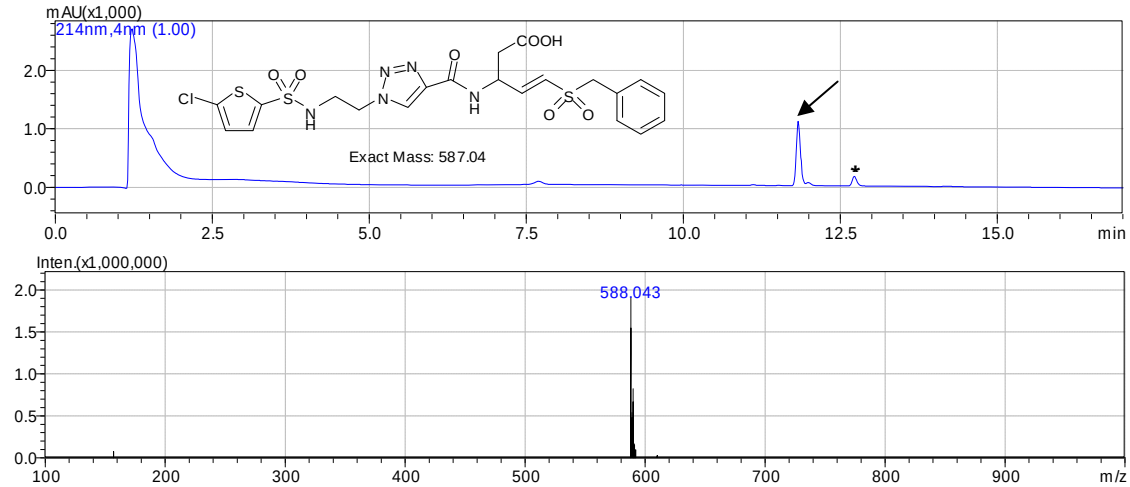

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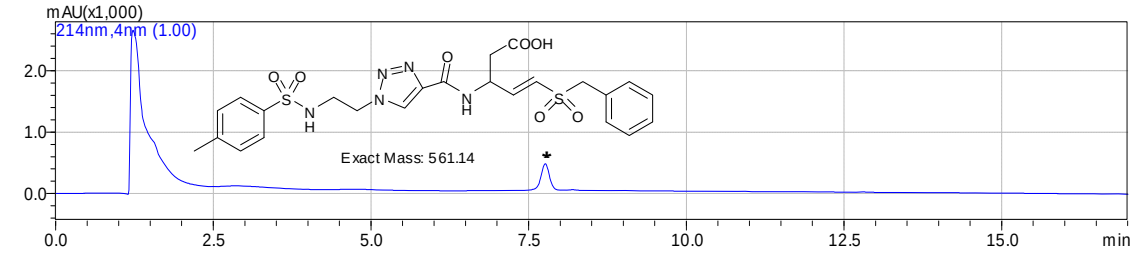

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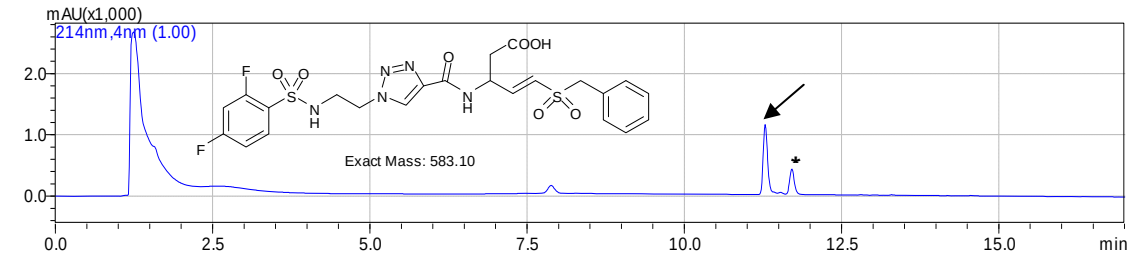

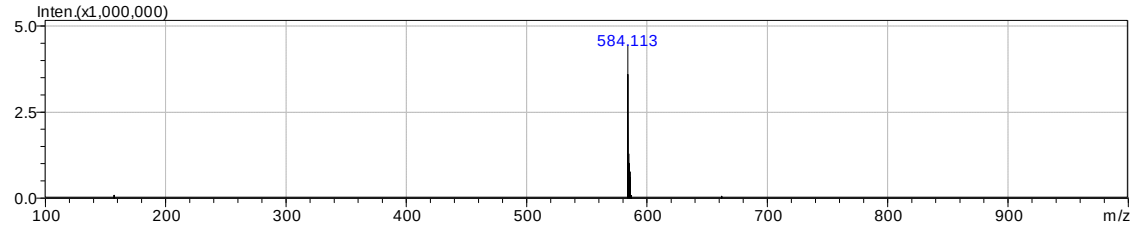

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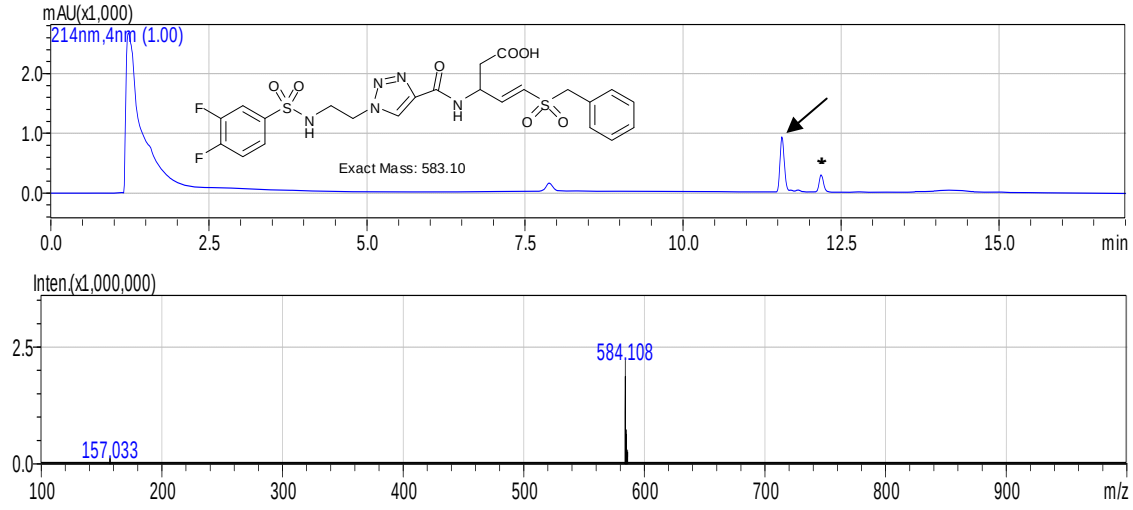

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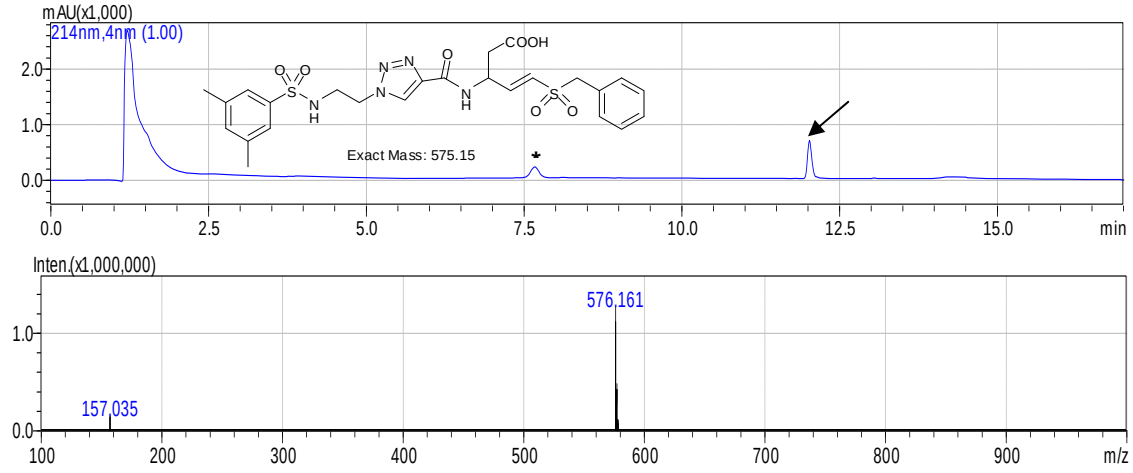

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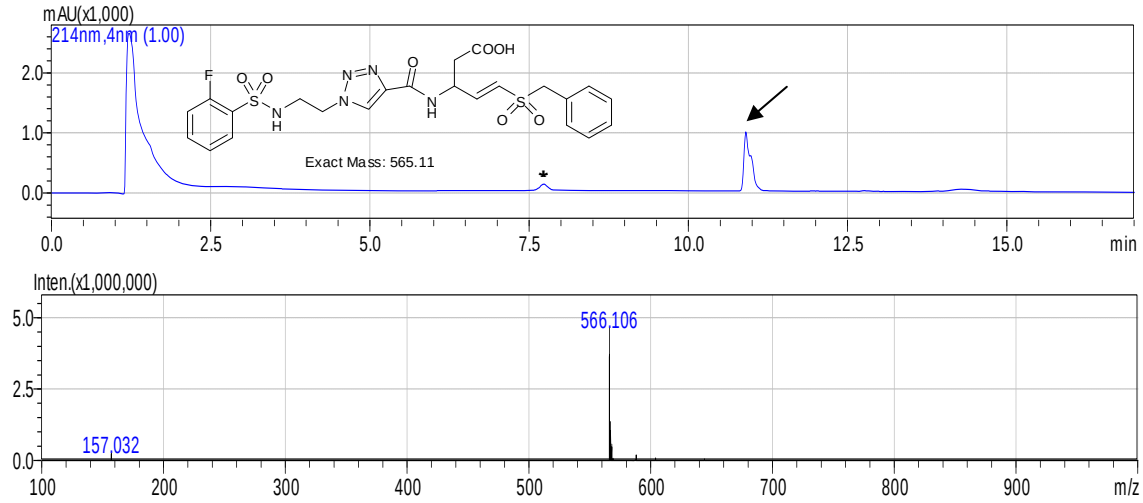

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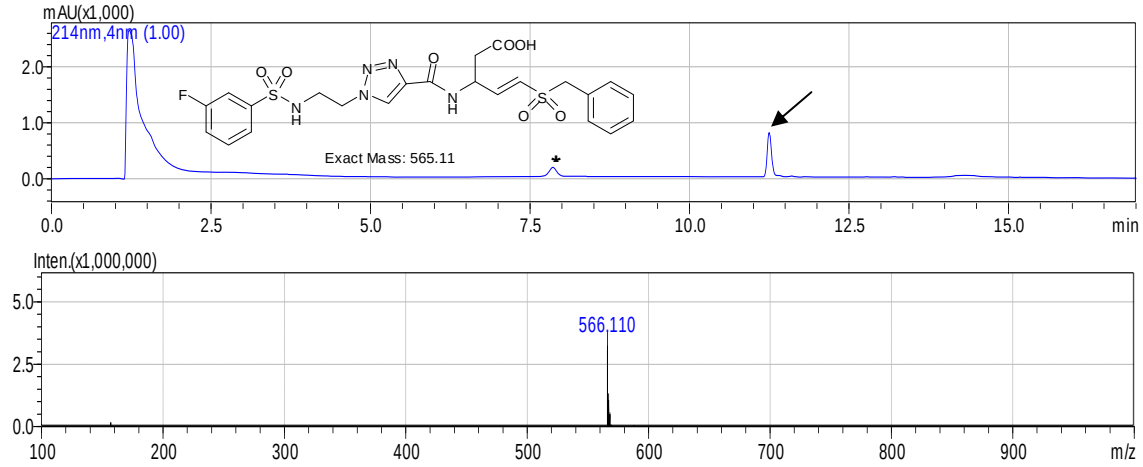

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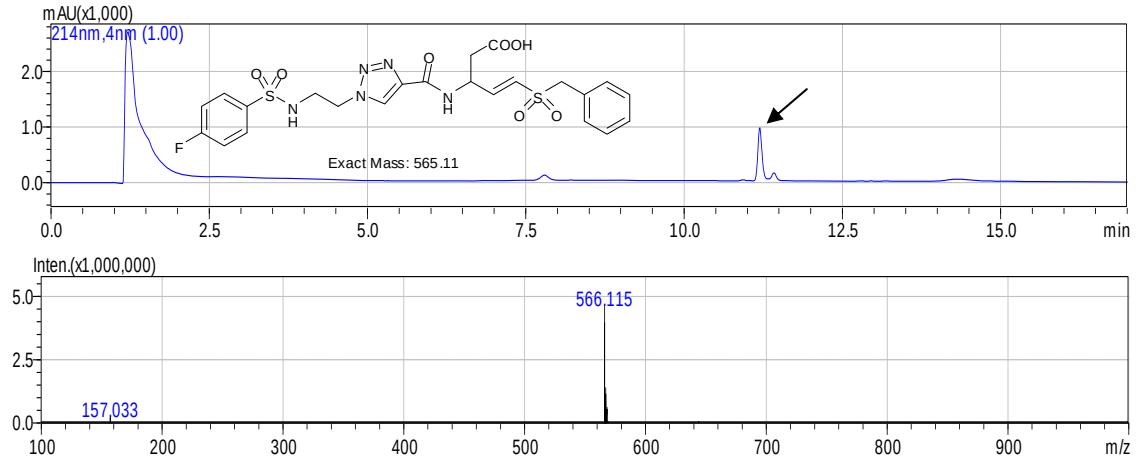

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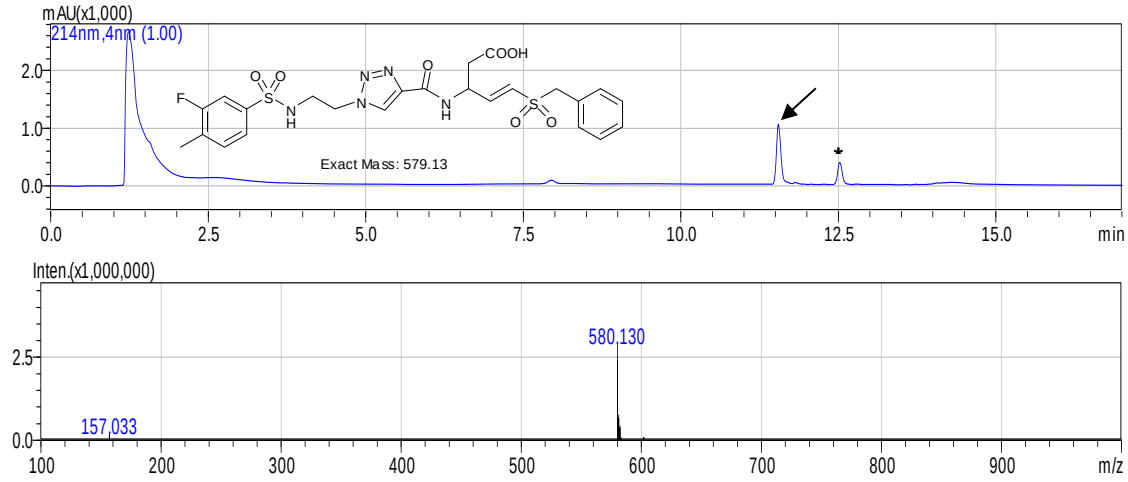

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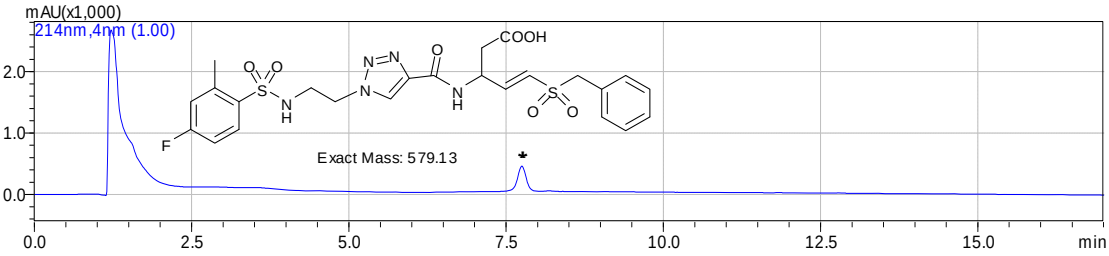

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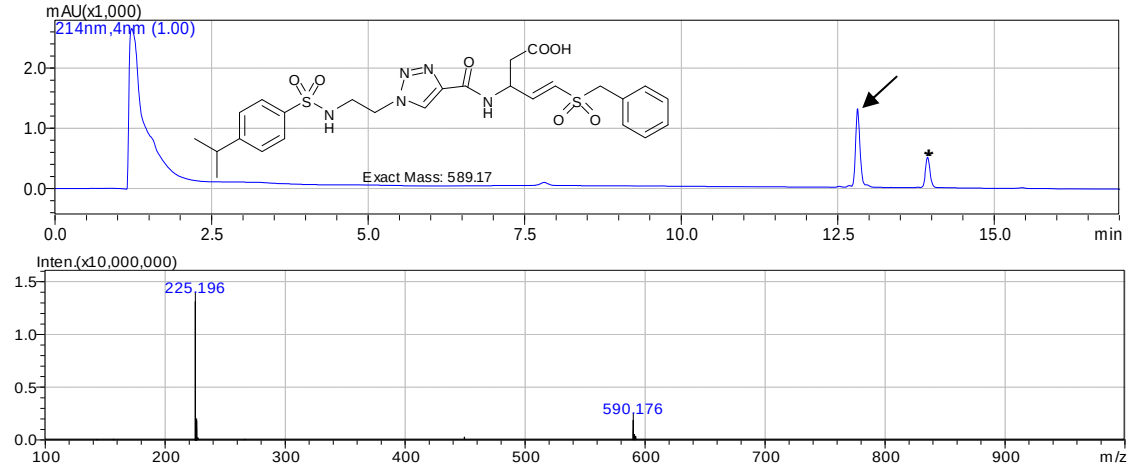

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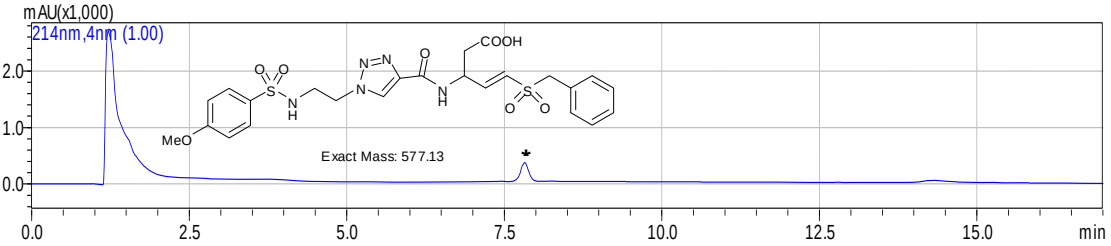

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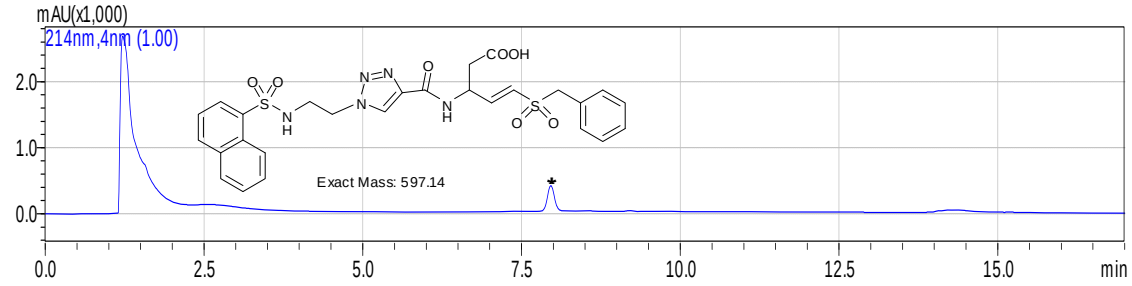

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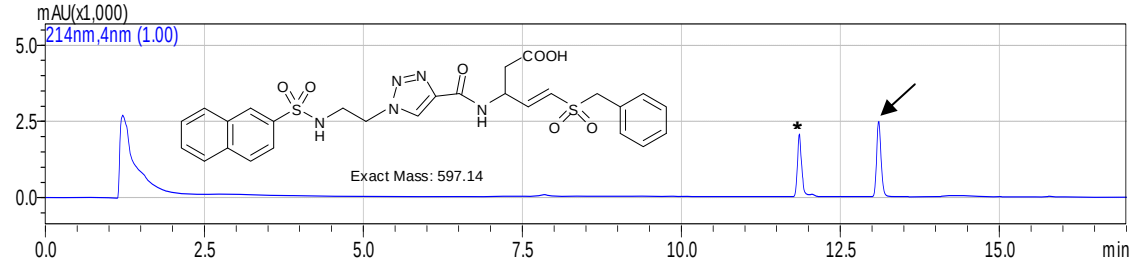

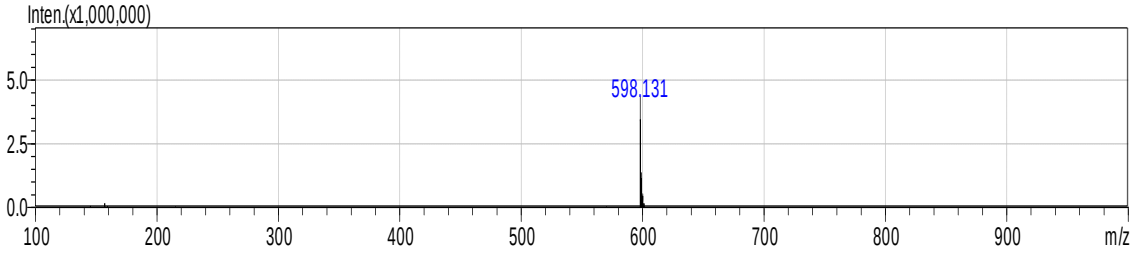

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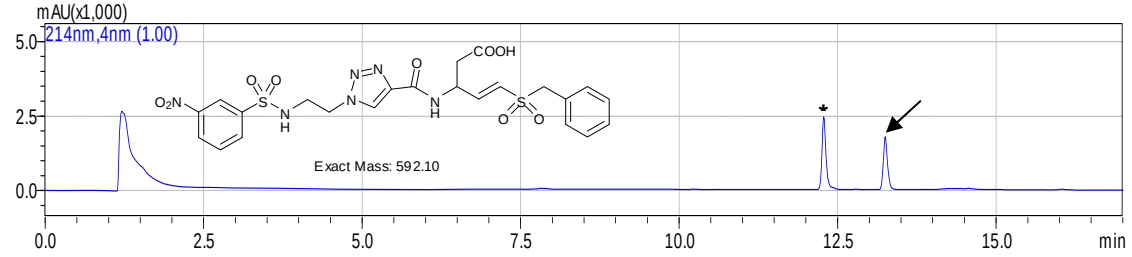

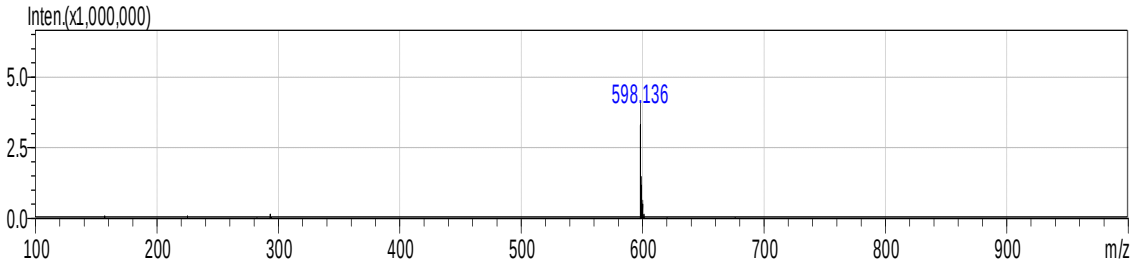

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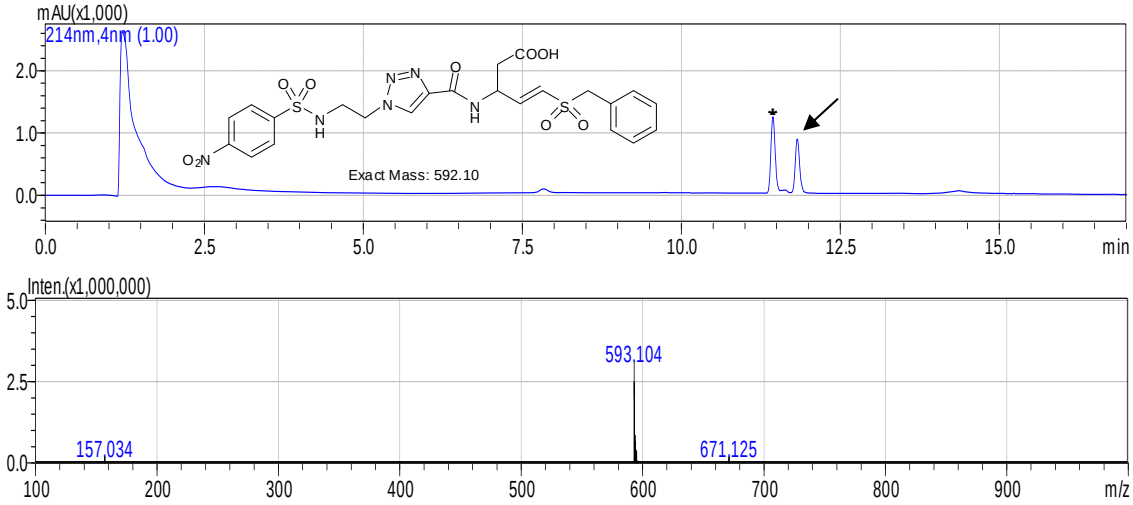

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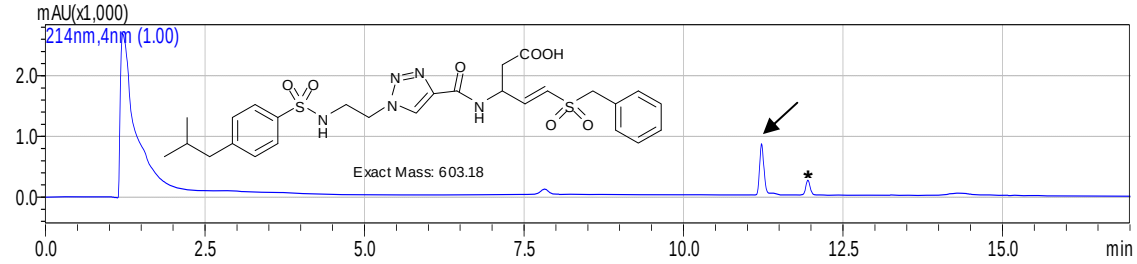

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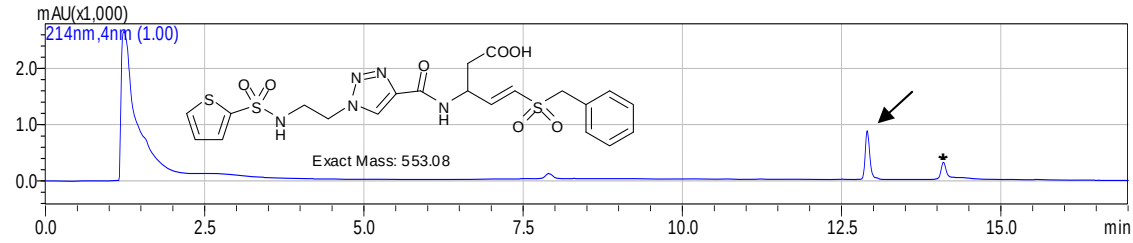

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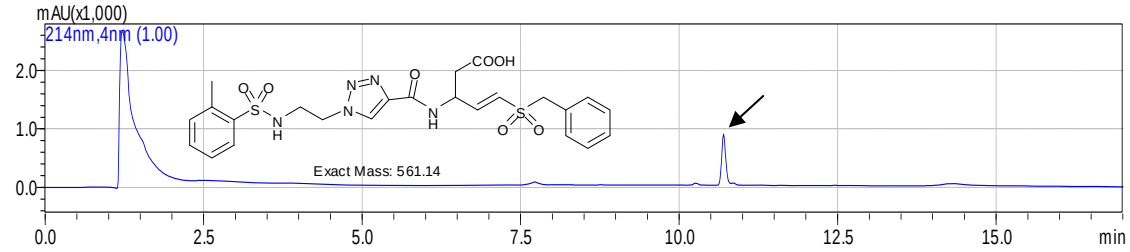

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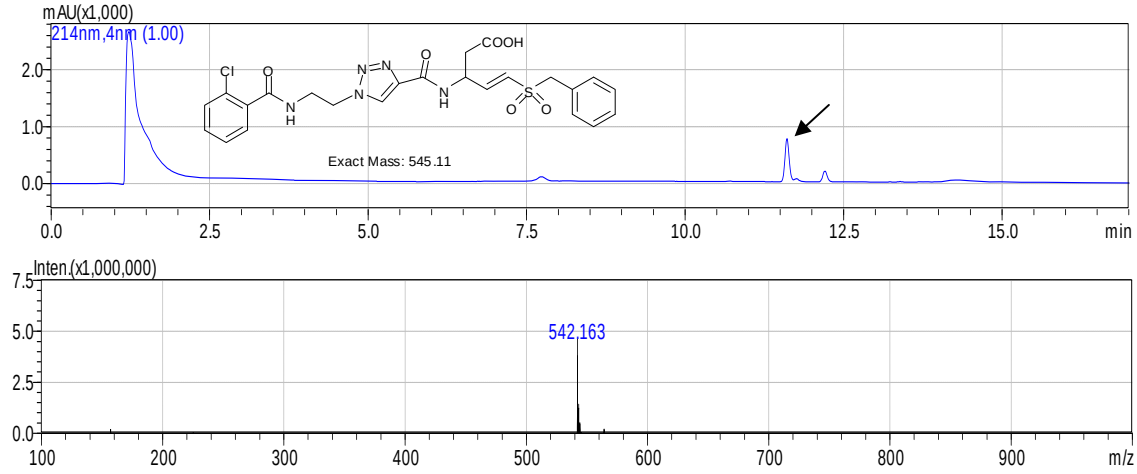

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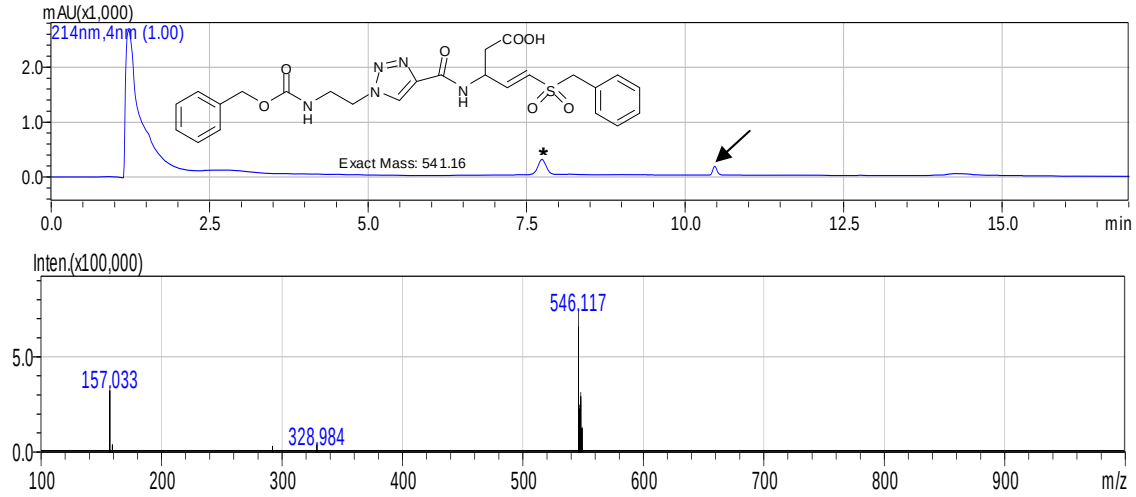

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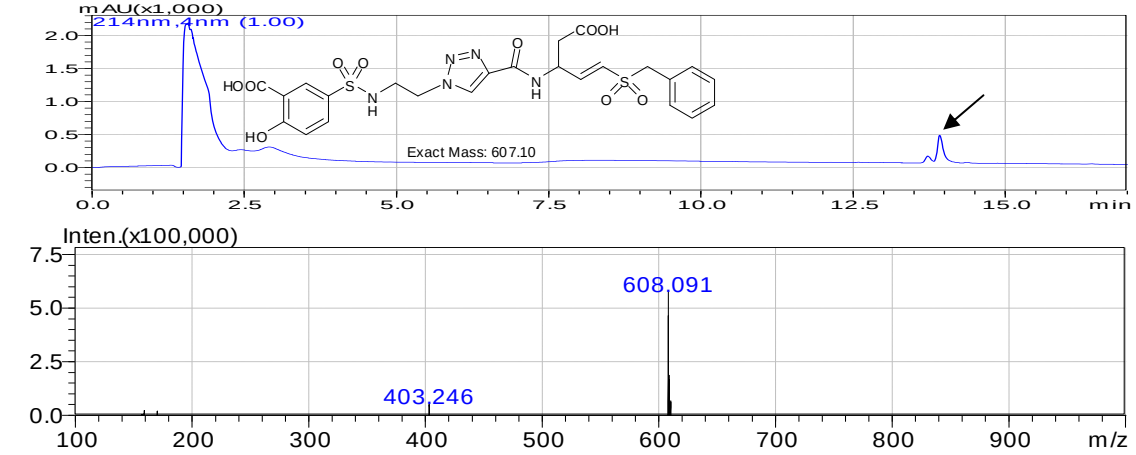

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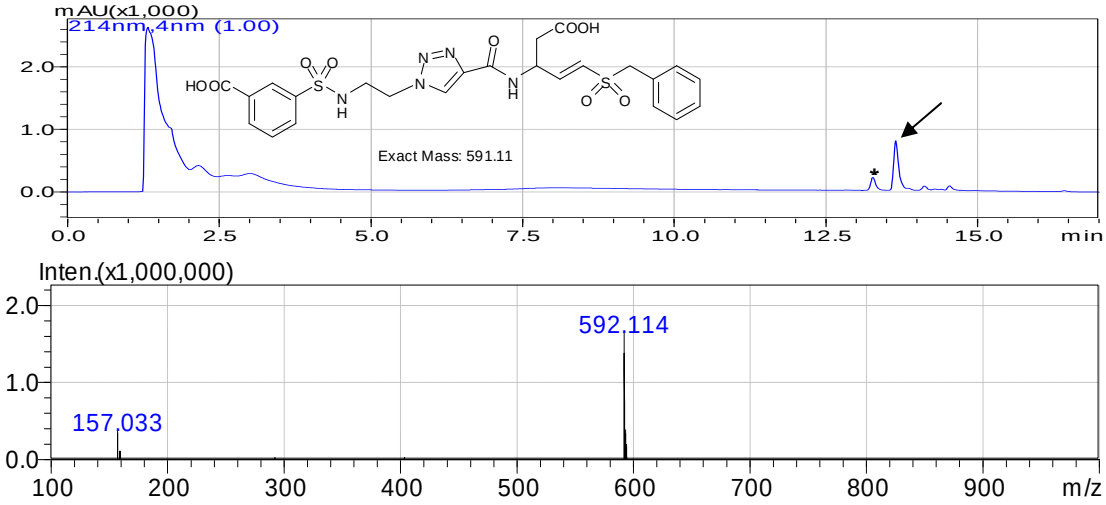
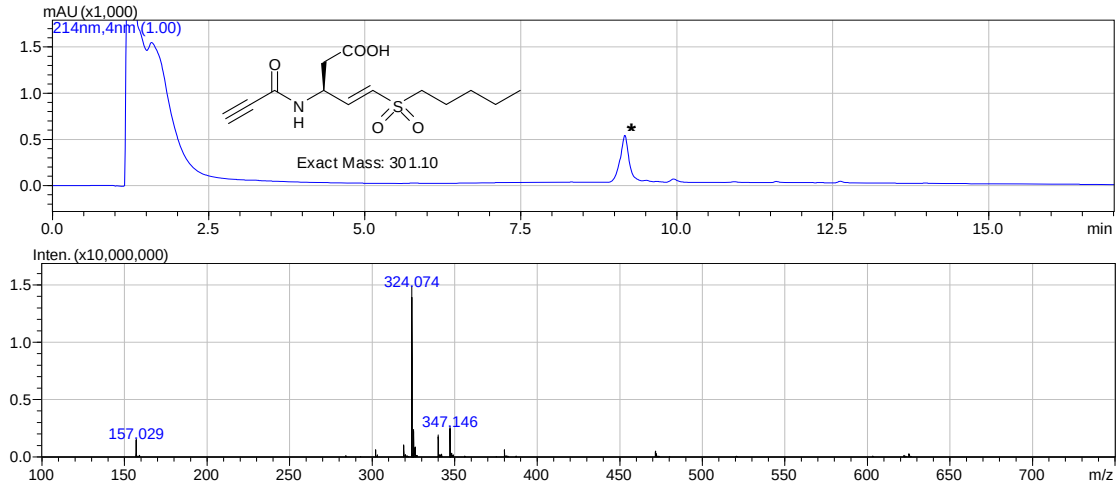


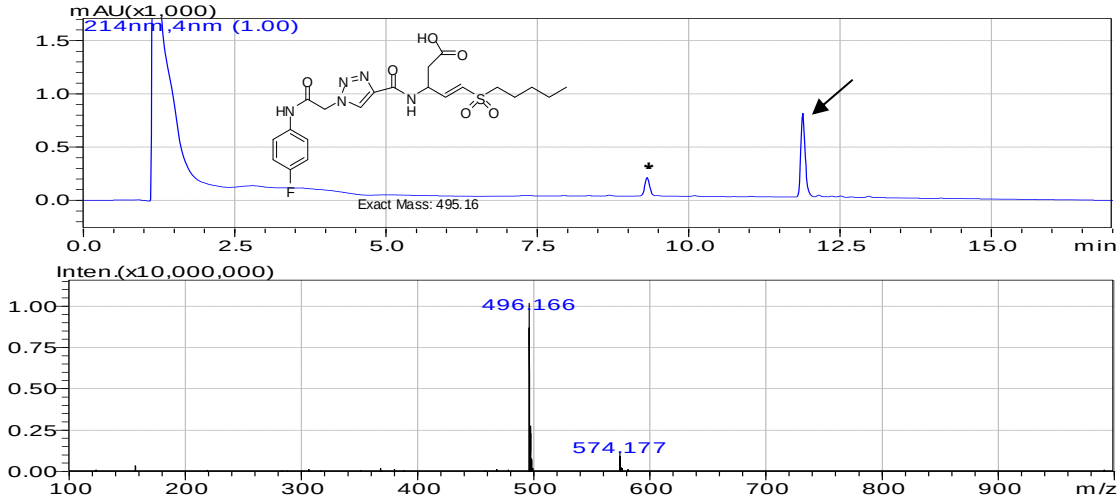
2.2 LC-MS profiles of pentyl vinyl sulfone inhibitors (18)

All conditions are based on:
0 – 10 min: 20% B (1% TFA in ACN) → 80% B
10 – 15 min: 80% B (1% TFA in ACN) → 100% B
15 – 17 min: 100% B
* Starting material

VSP-warhead:



VSP-A1:



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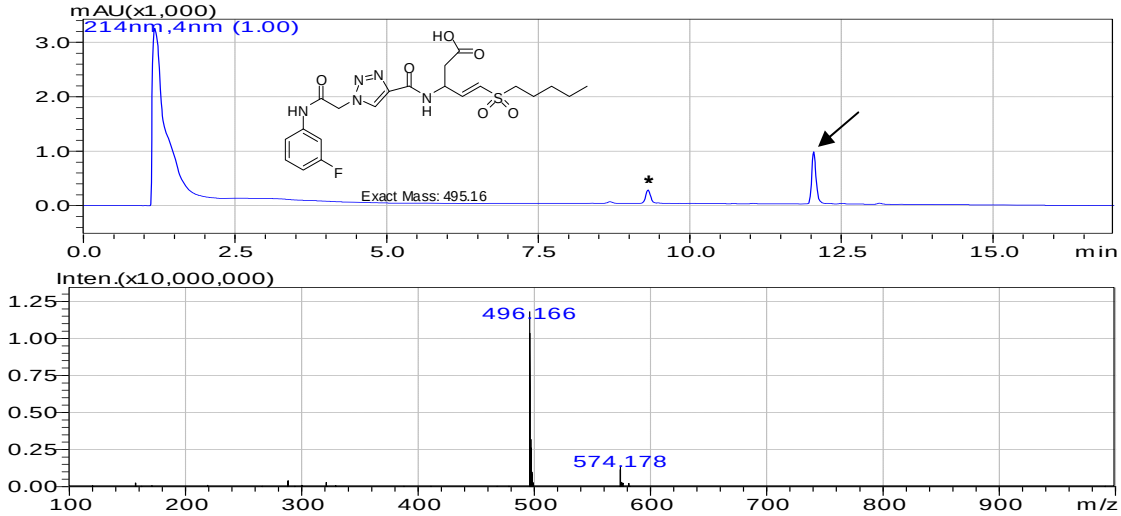

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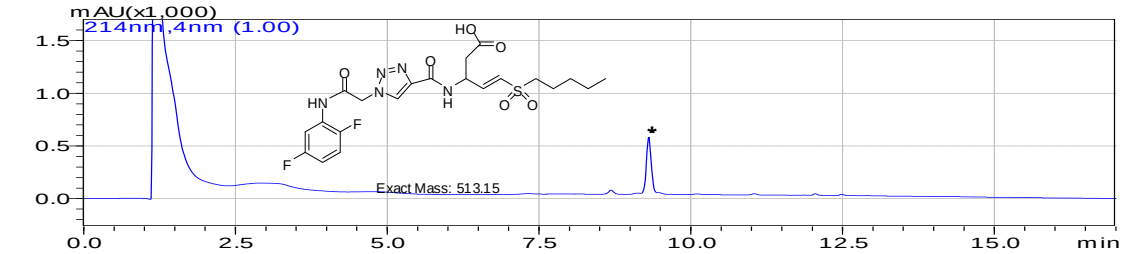

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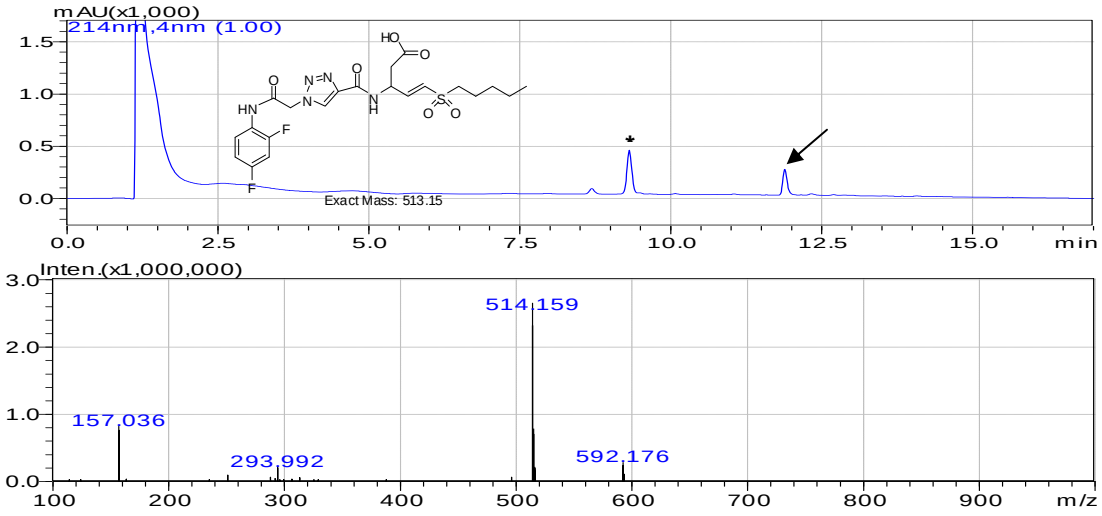

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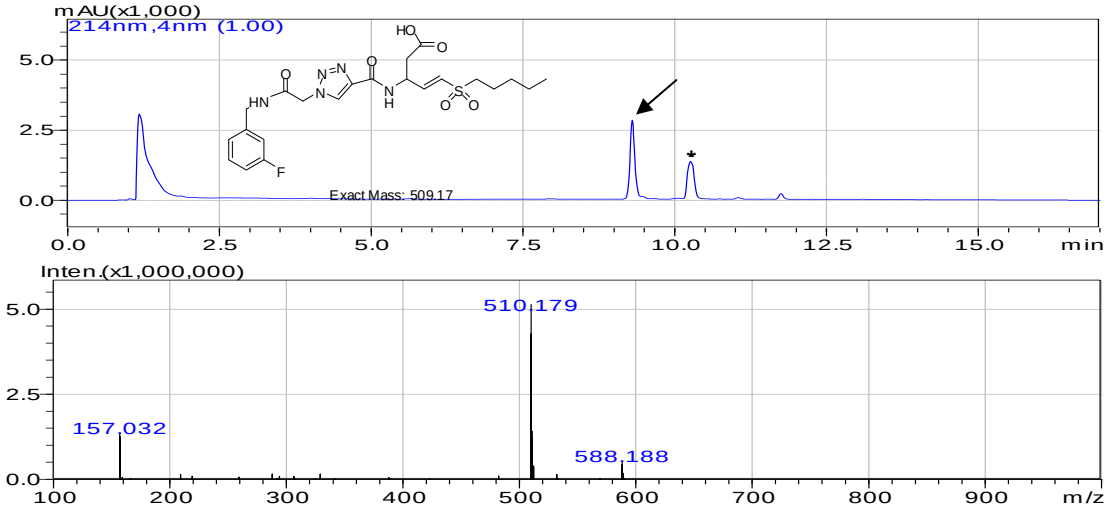

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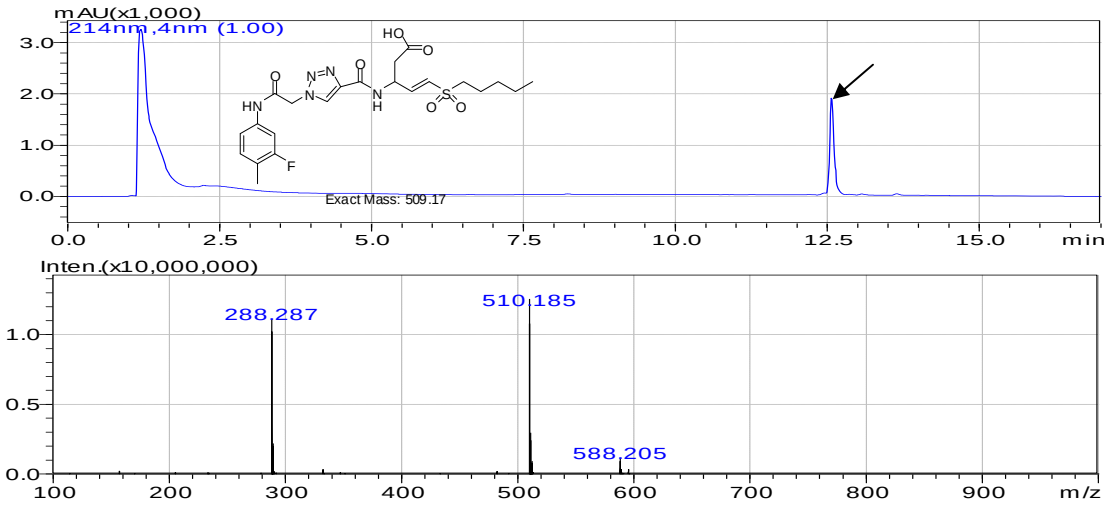

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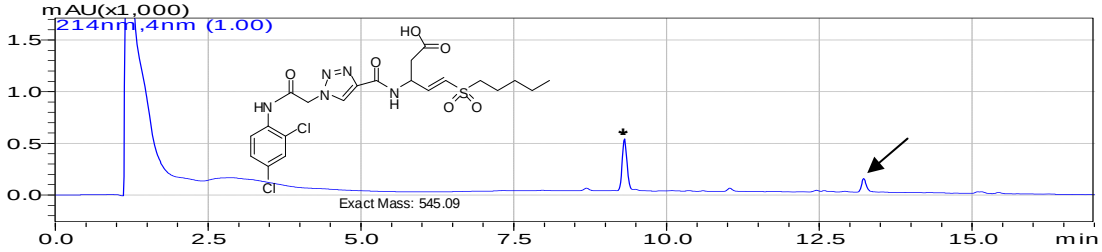

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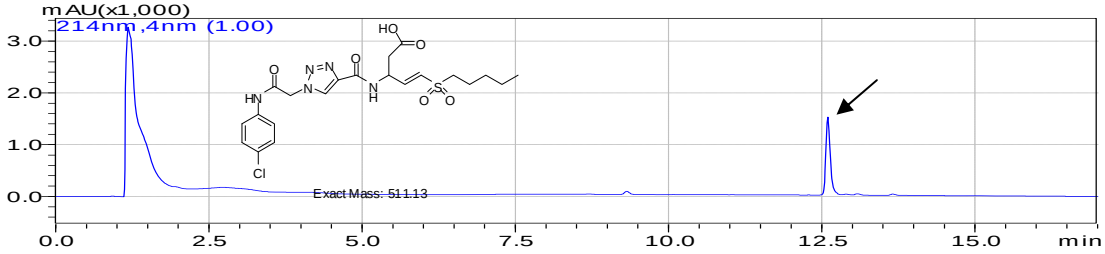

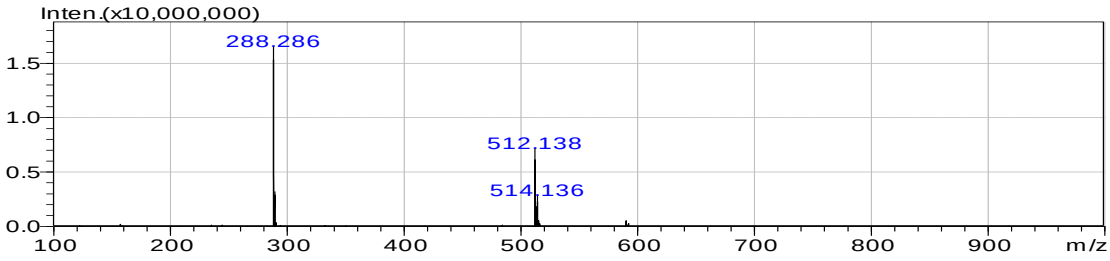

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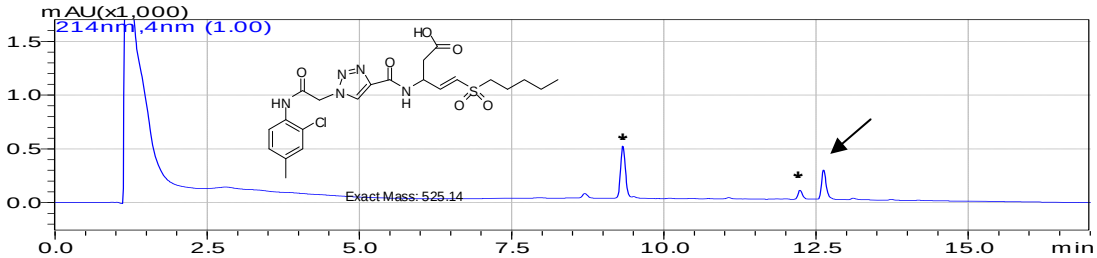

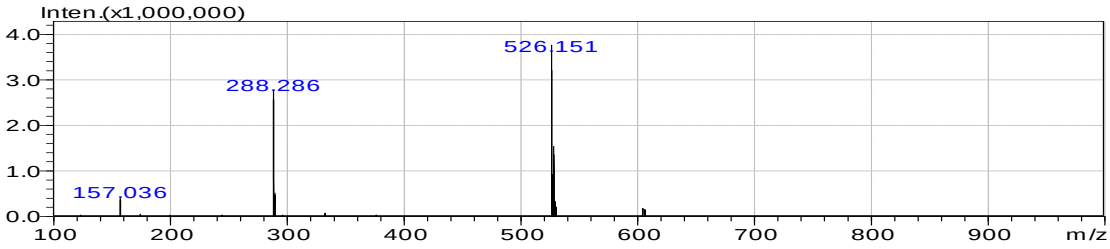

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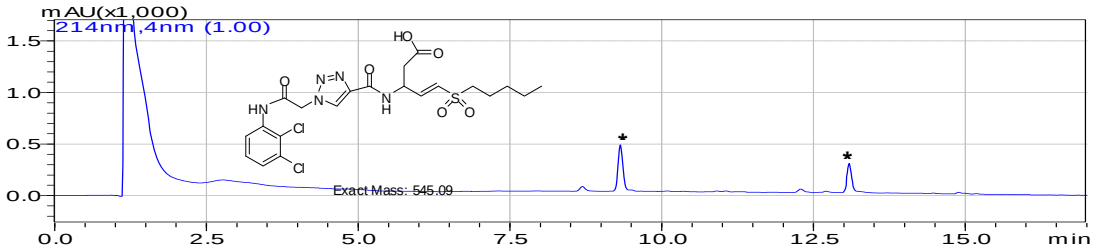

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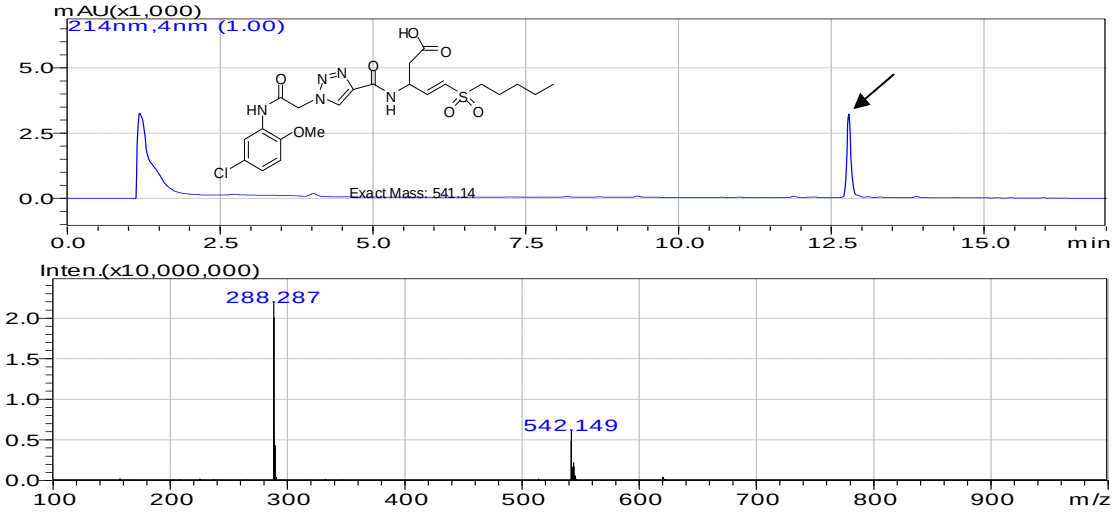

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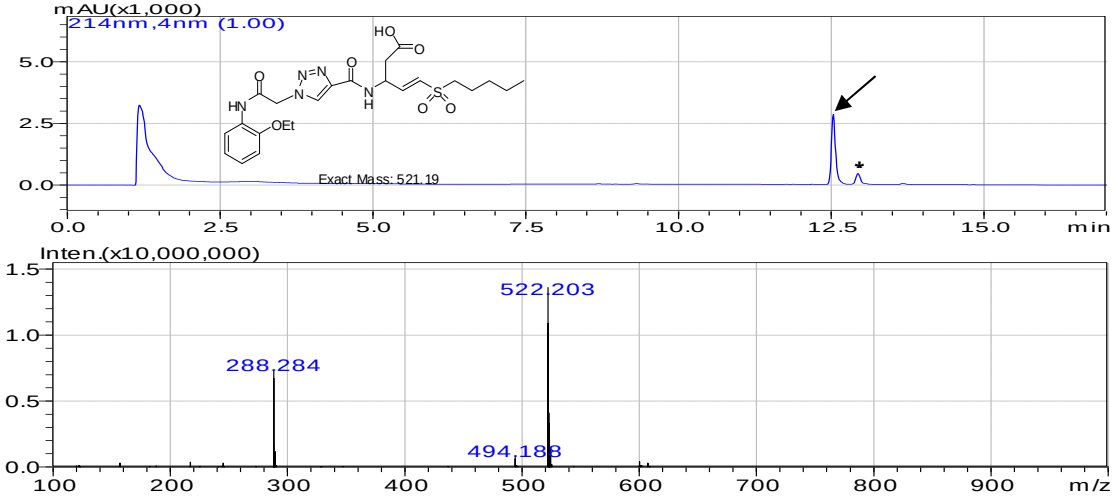

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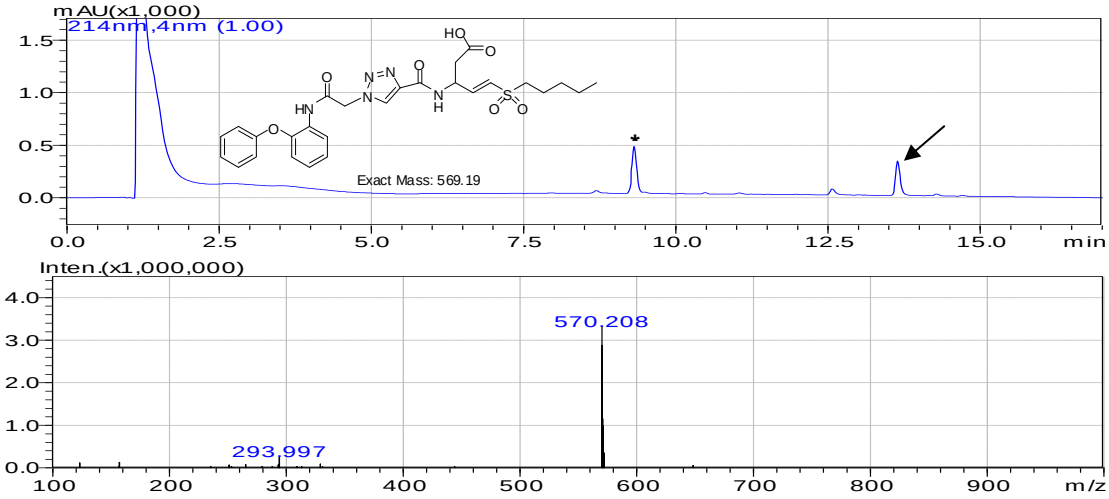

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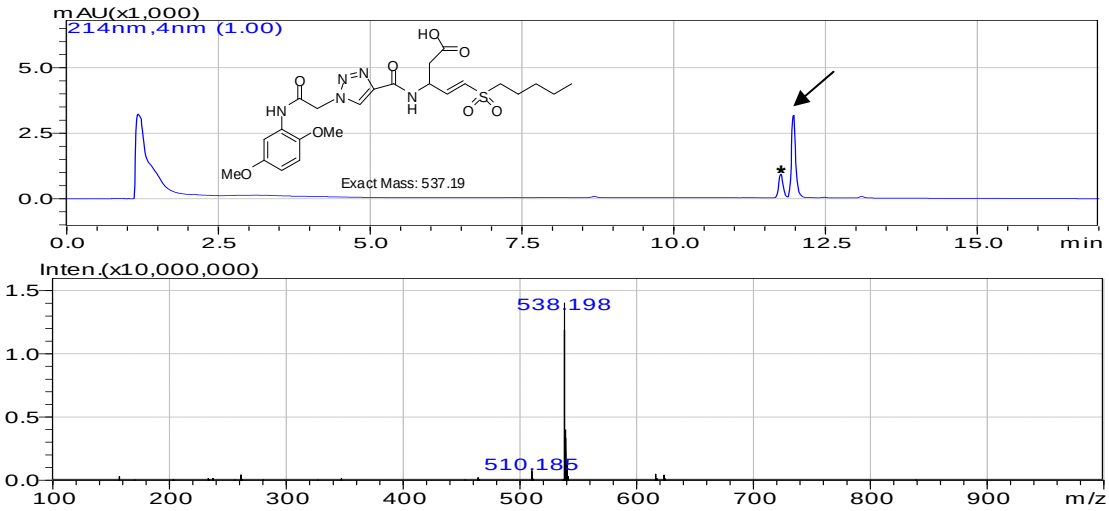

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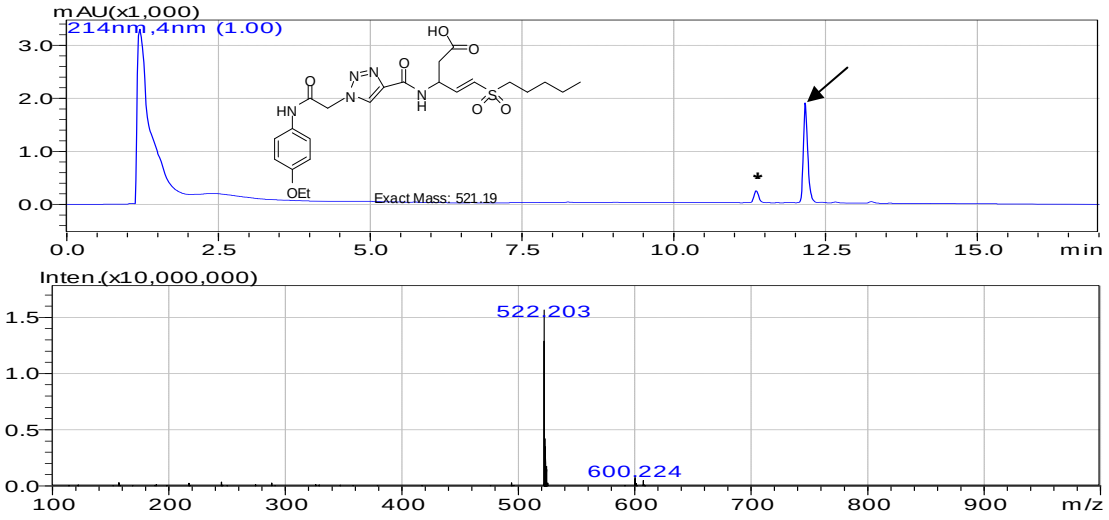

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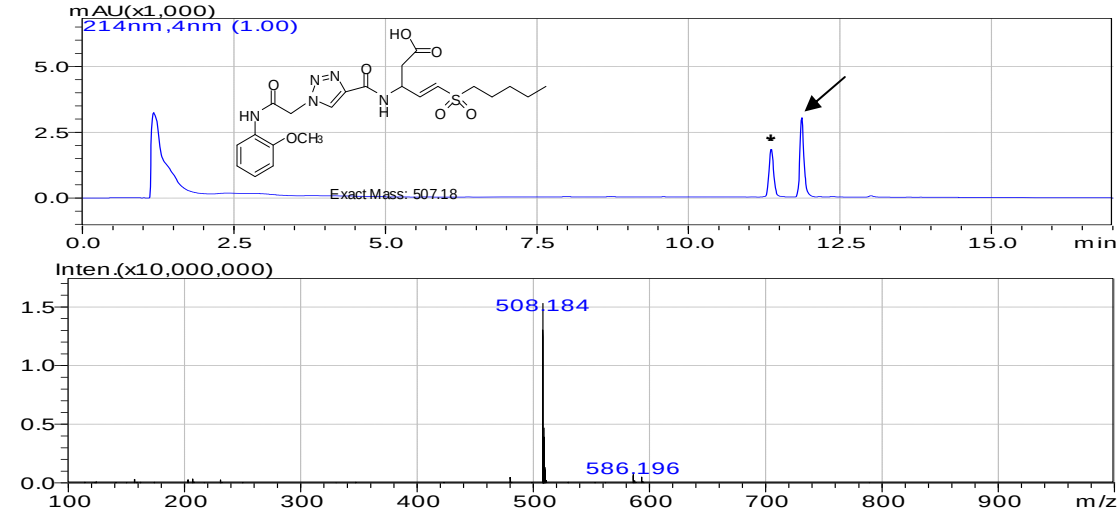

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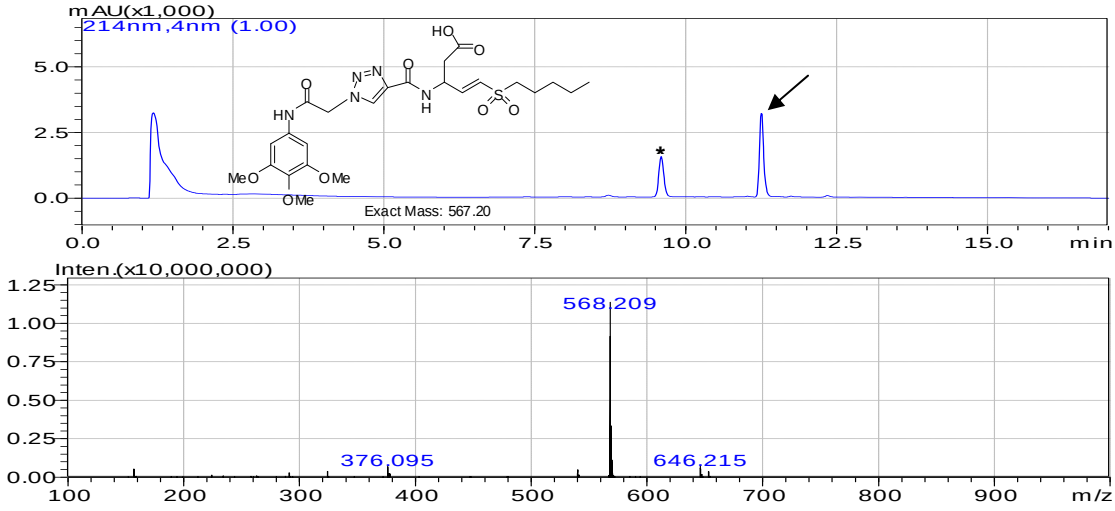

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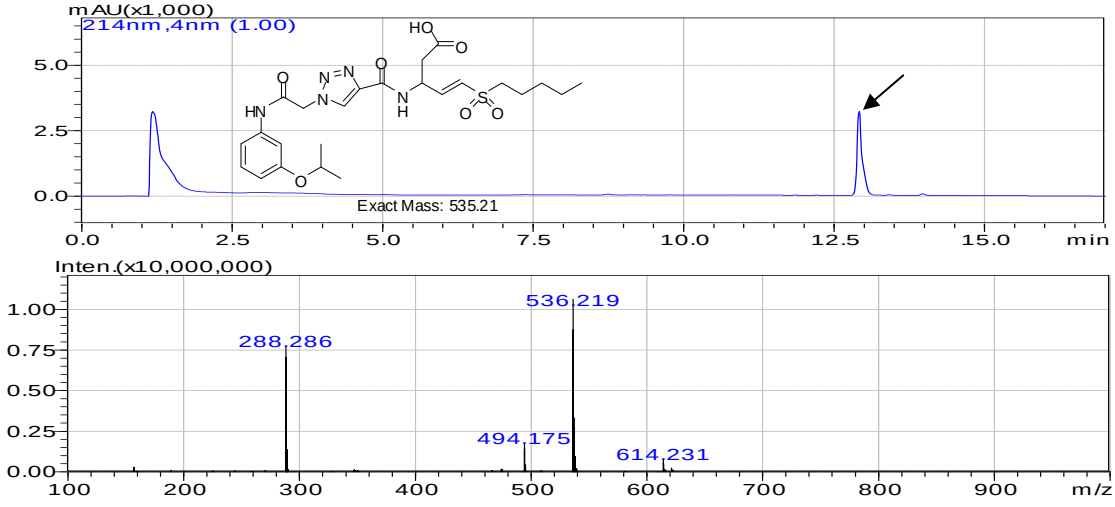

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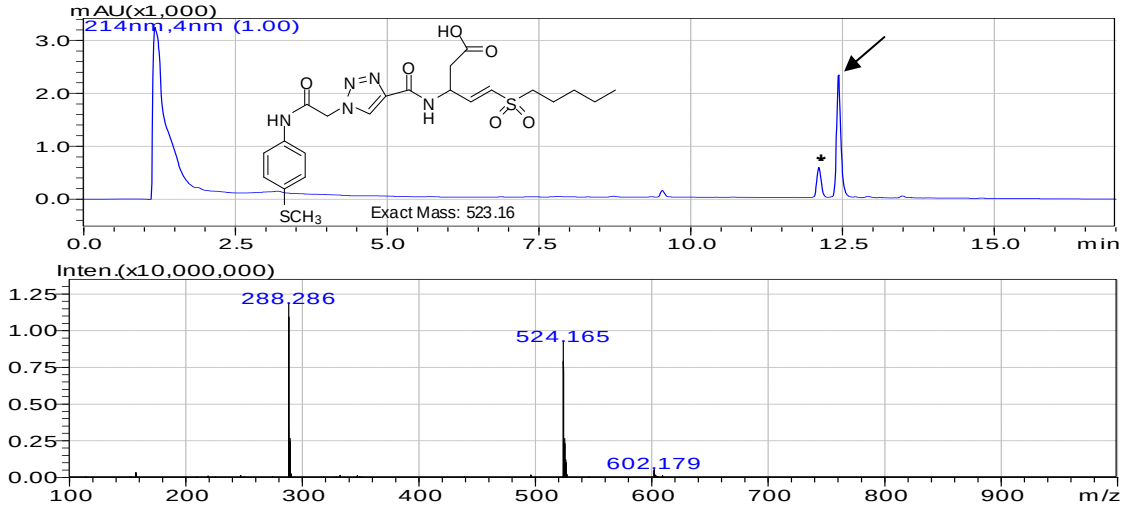

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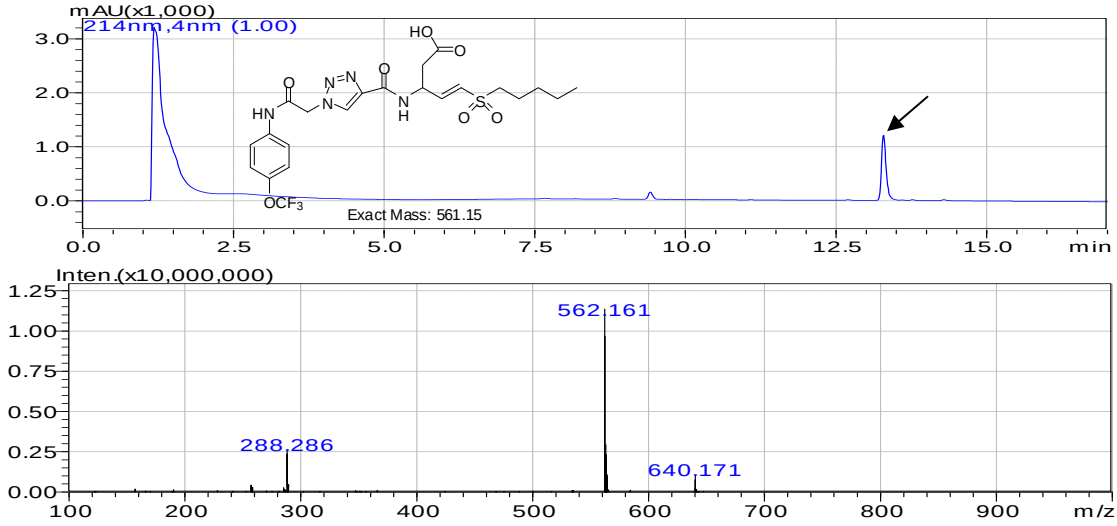

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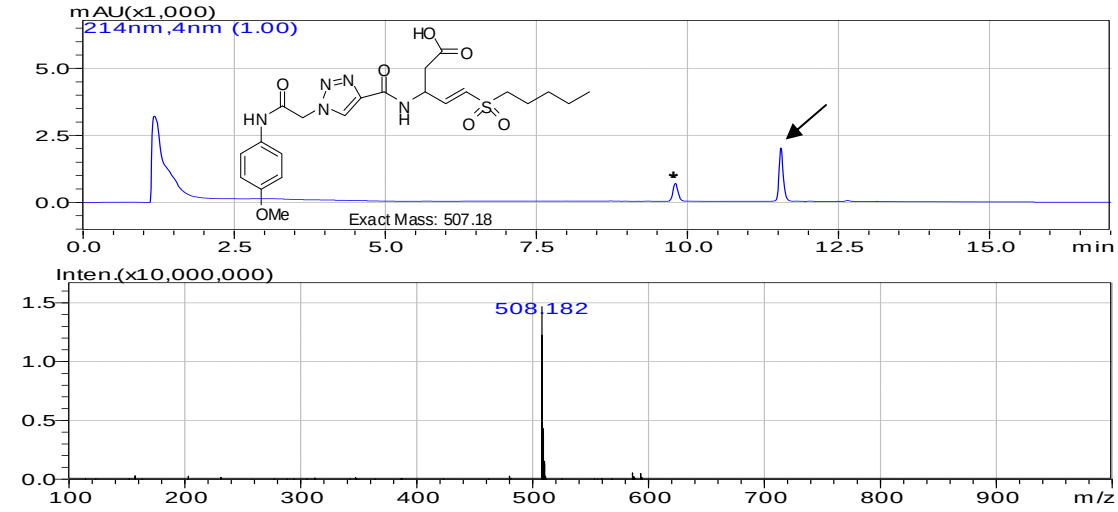

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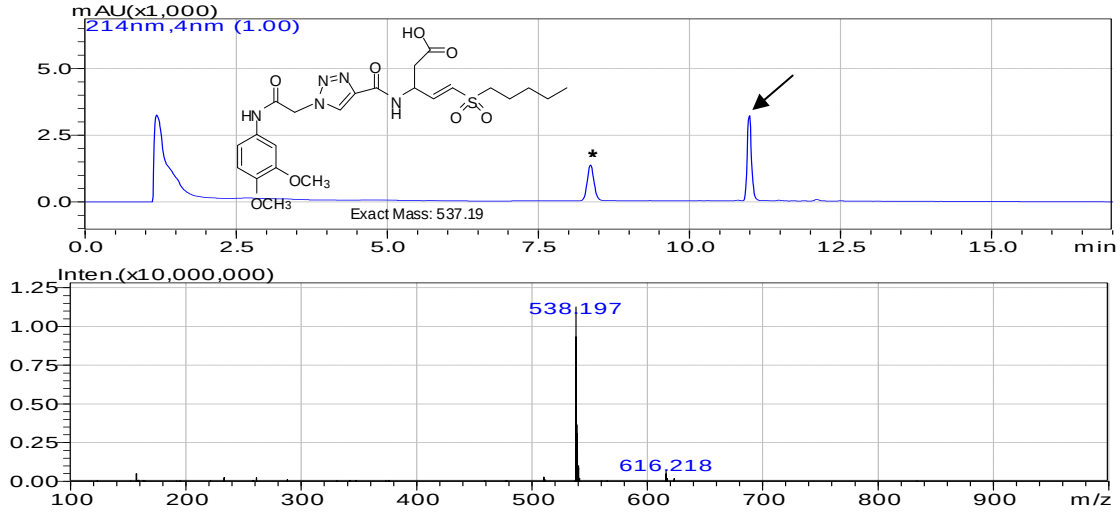

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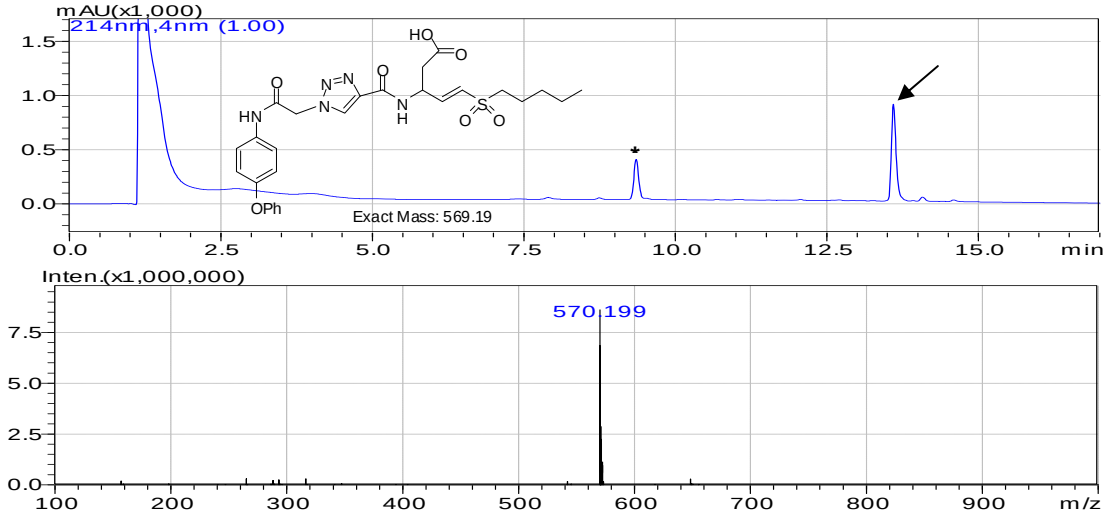

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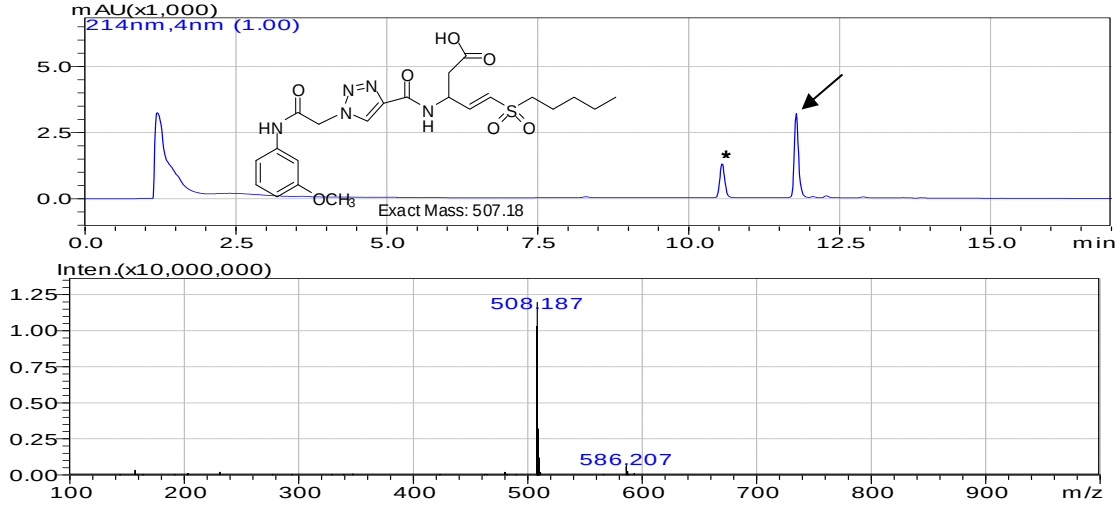

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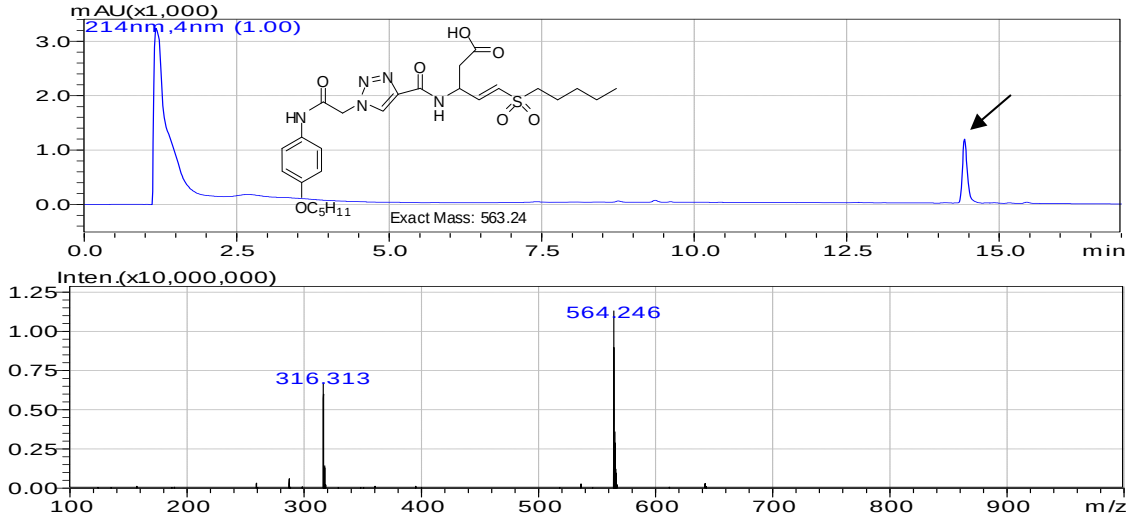

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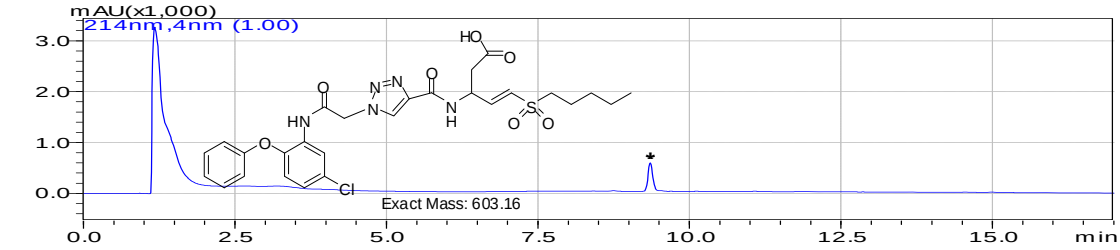

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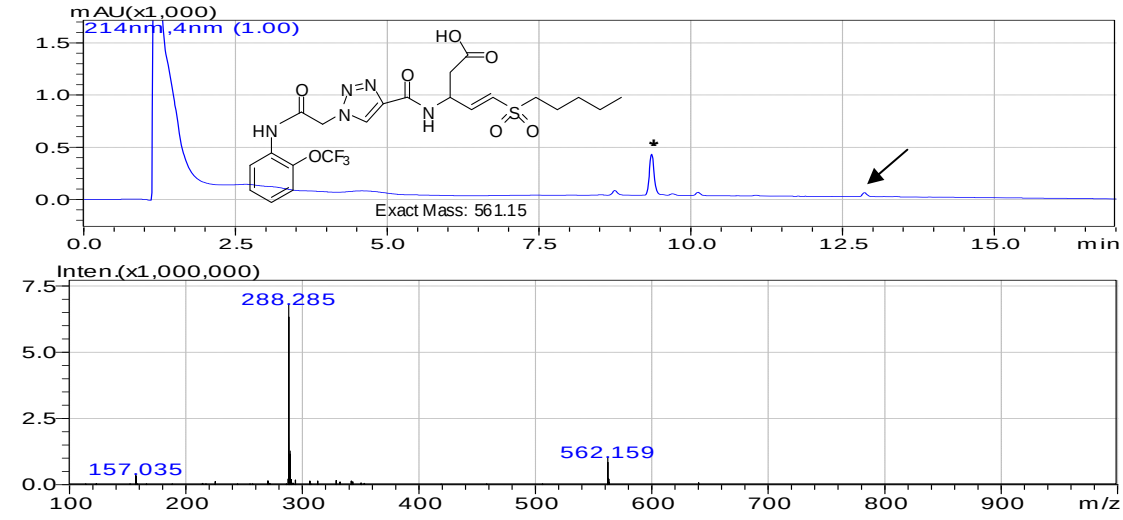

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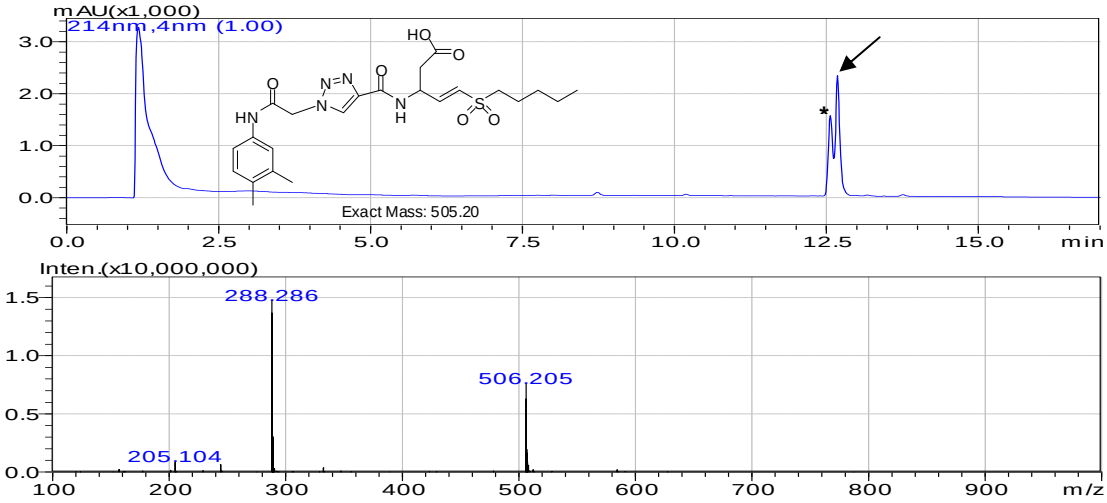

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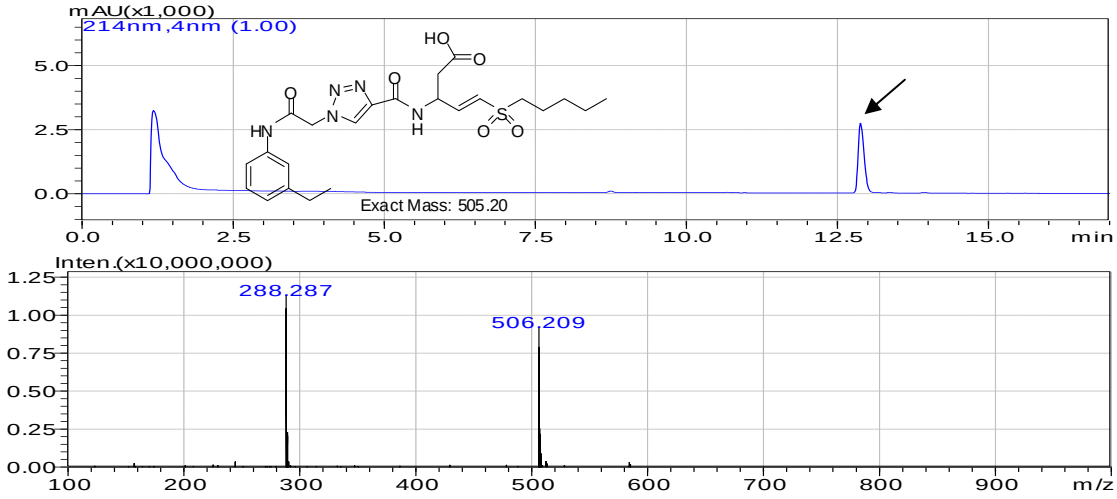

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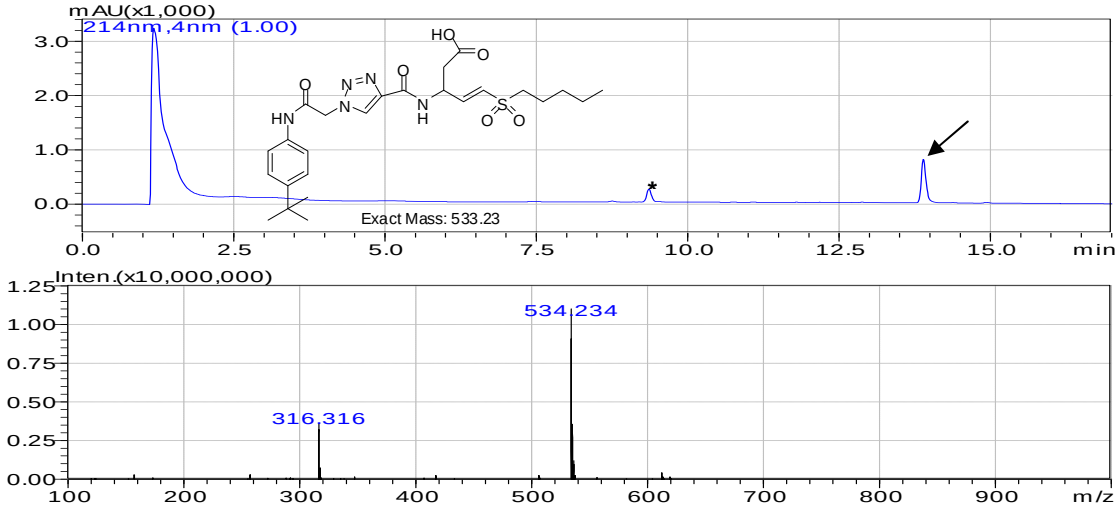

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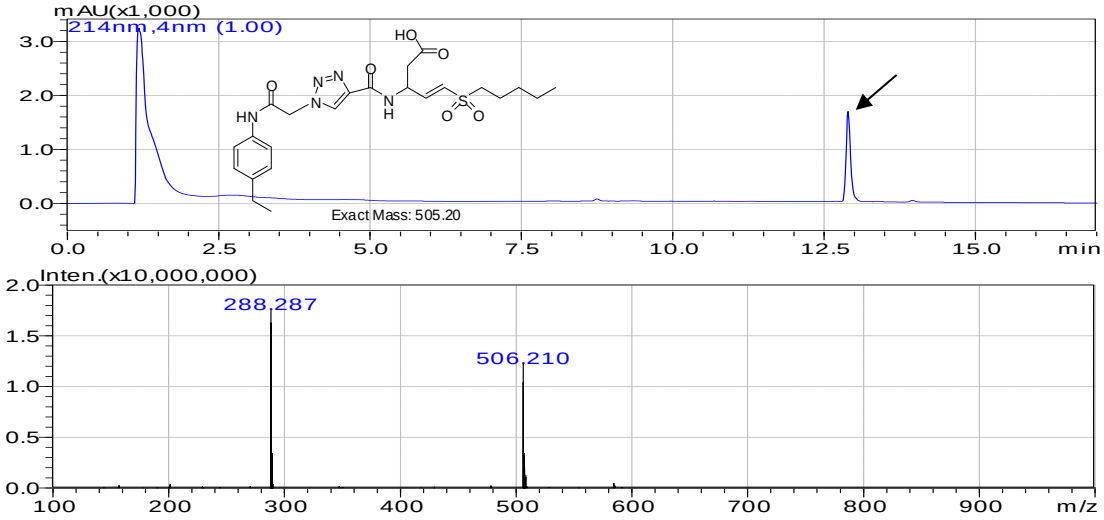

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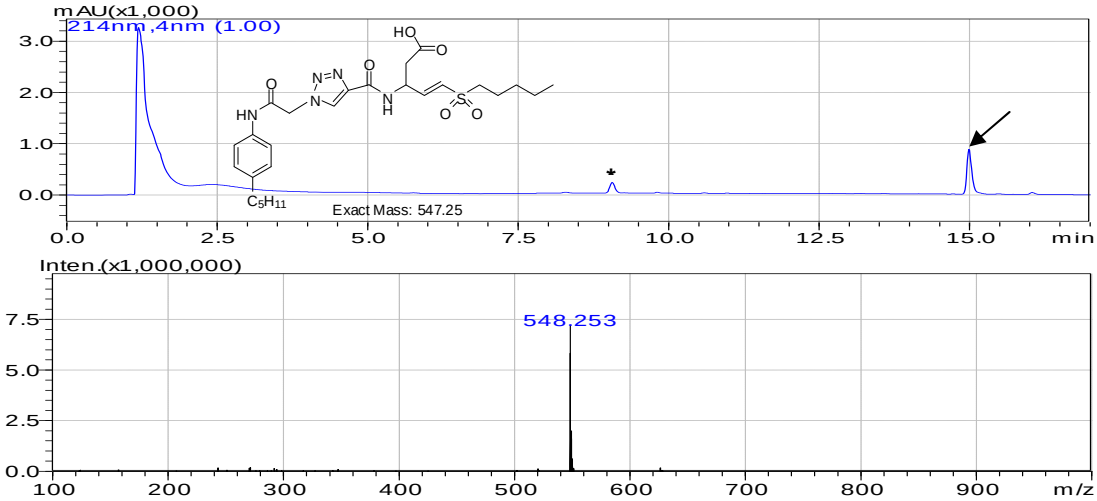

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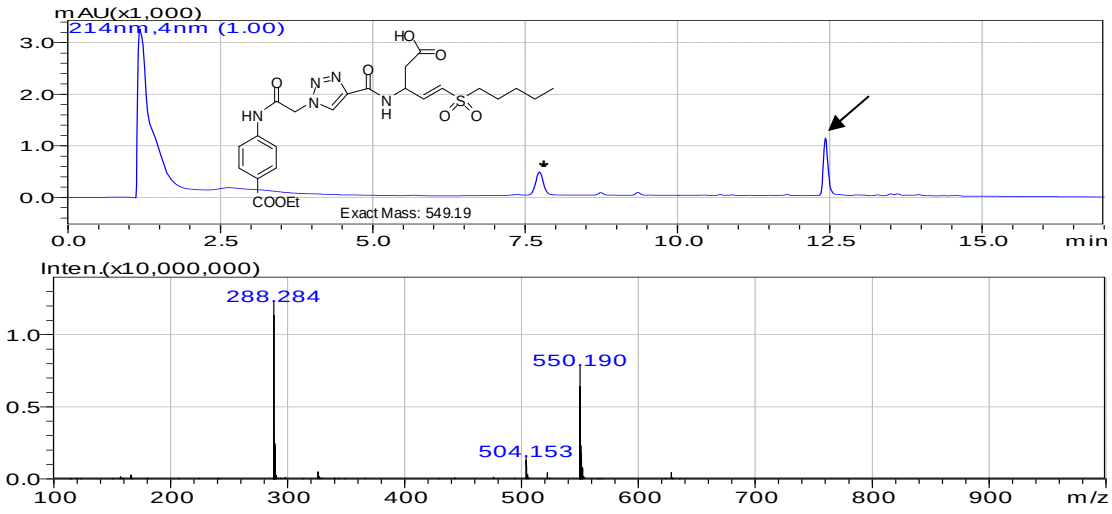

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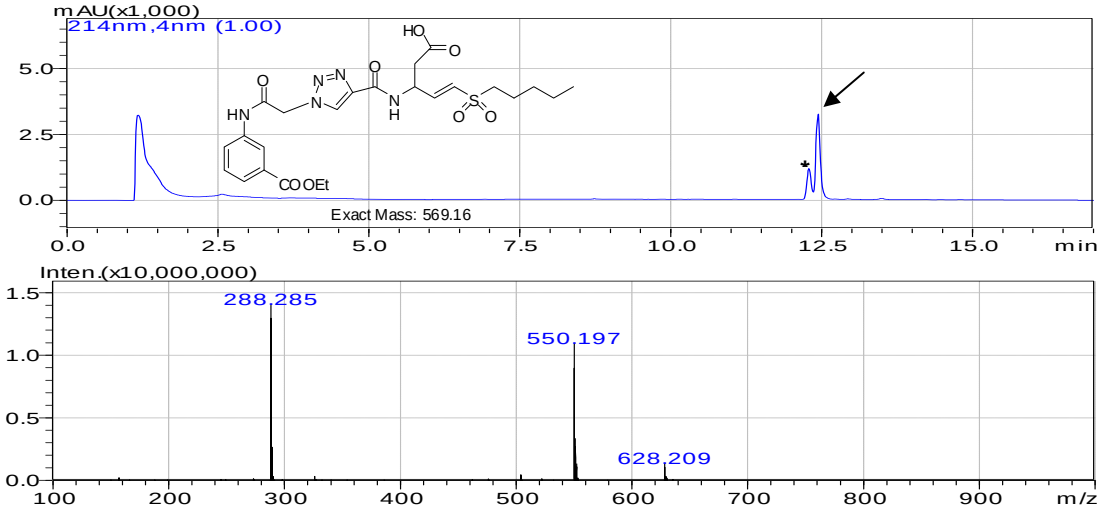

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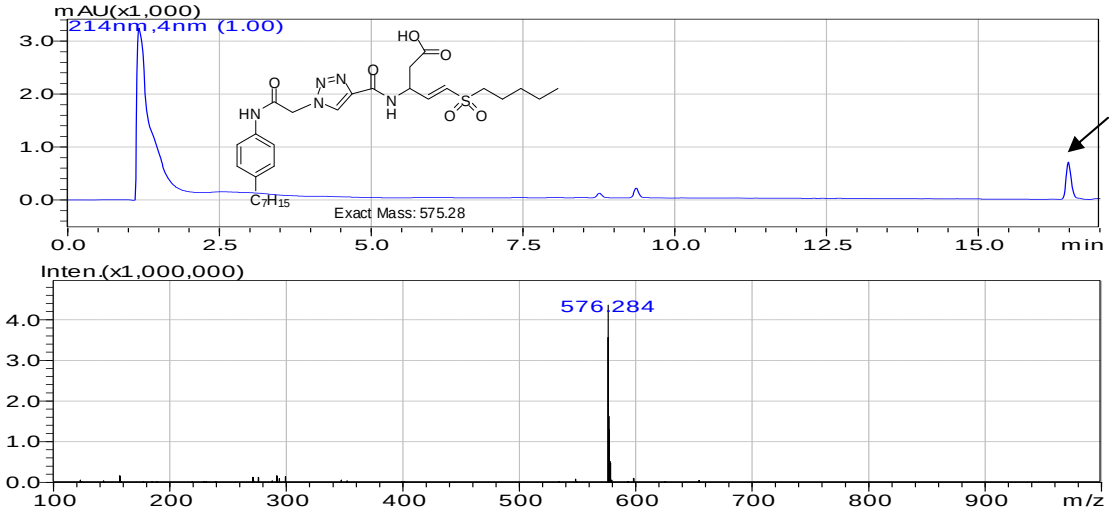

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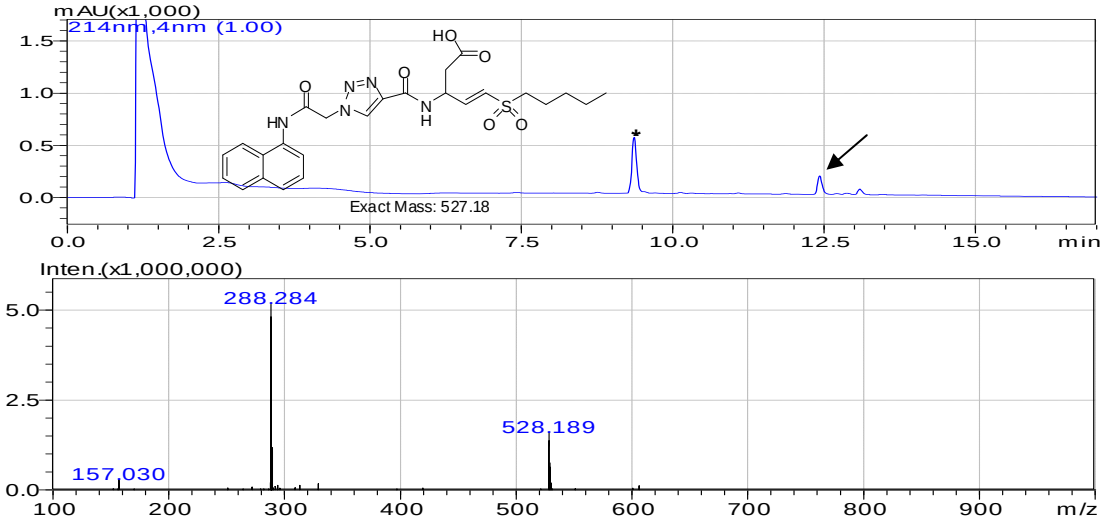

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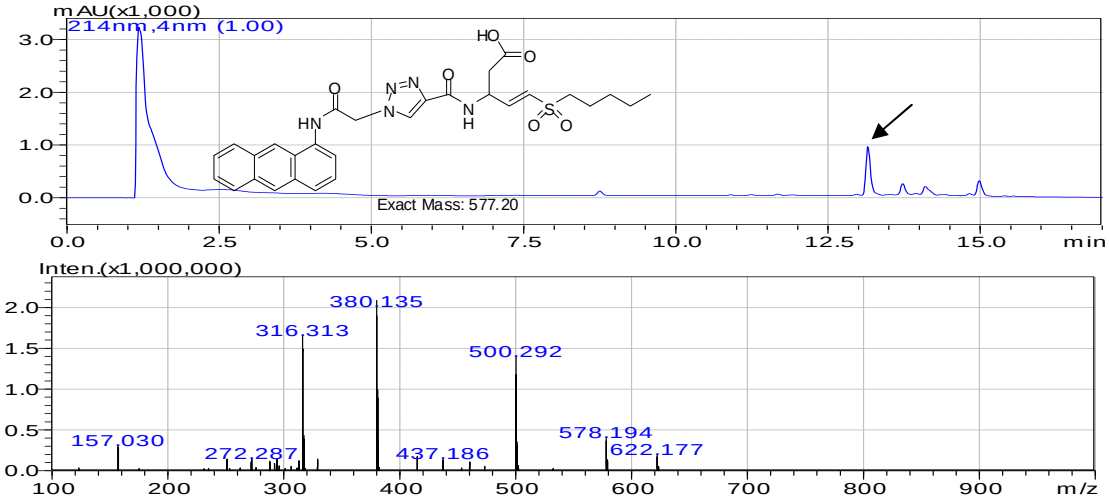

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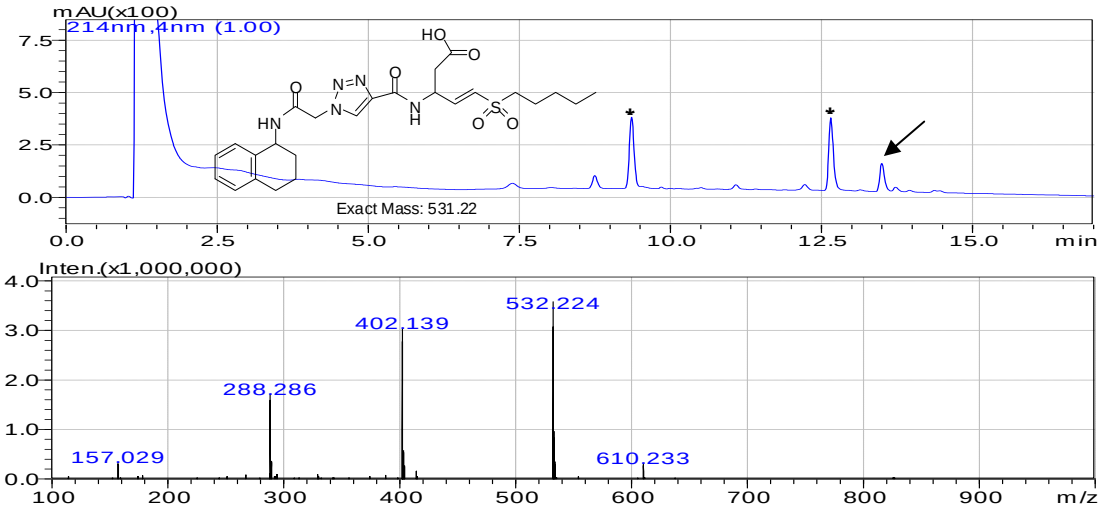

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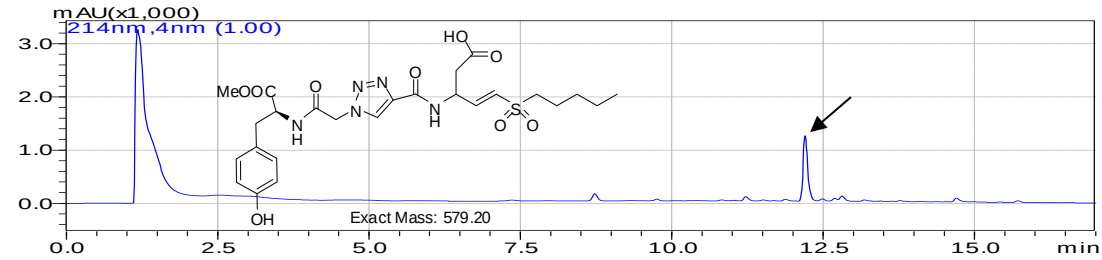

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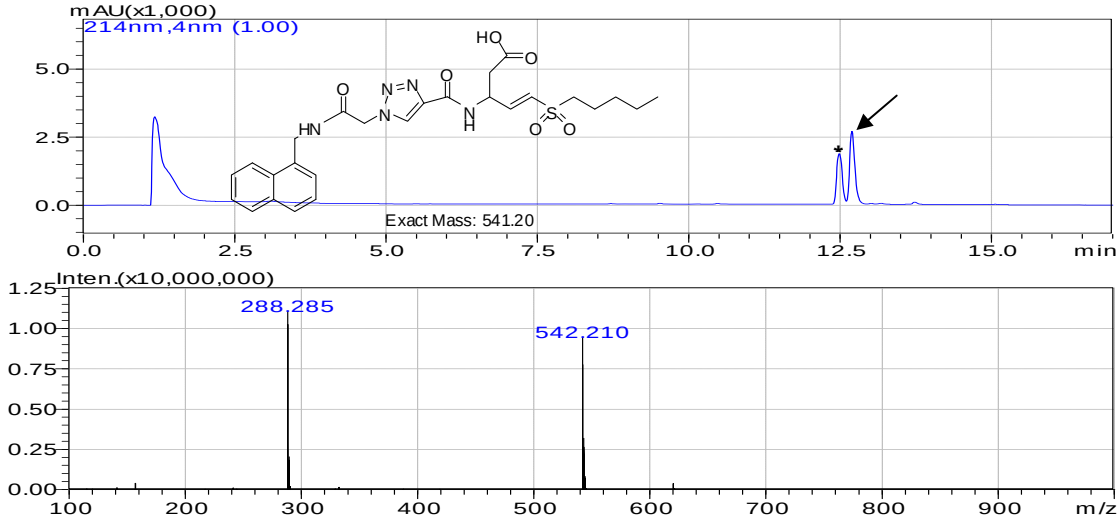

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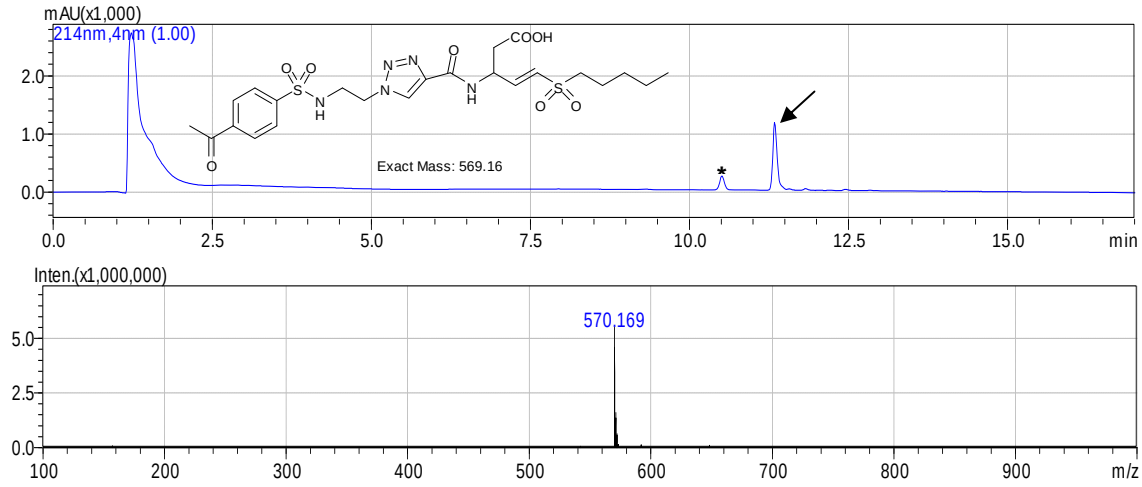

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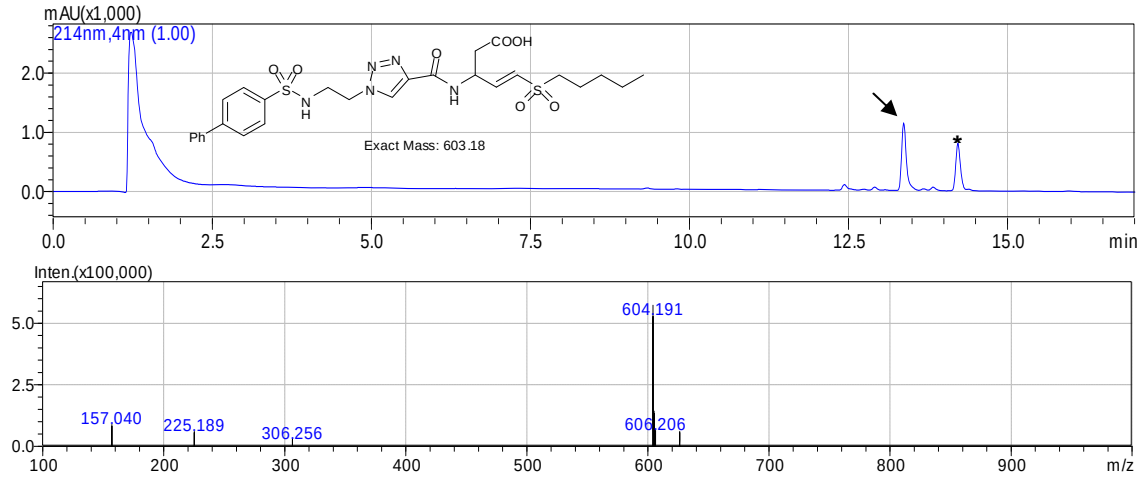

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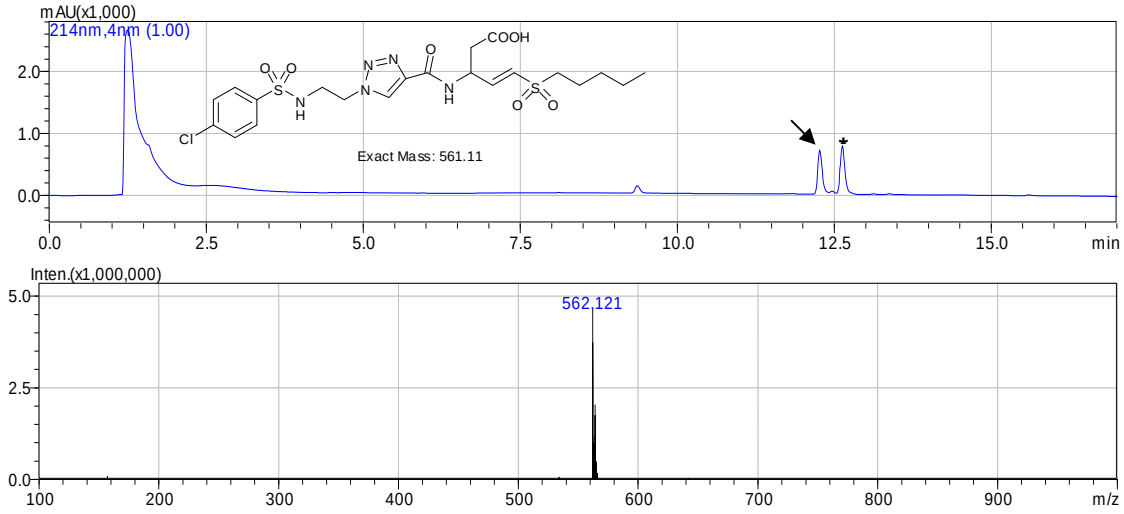

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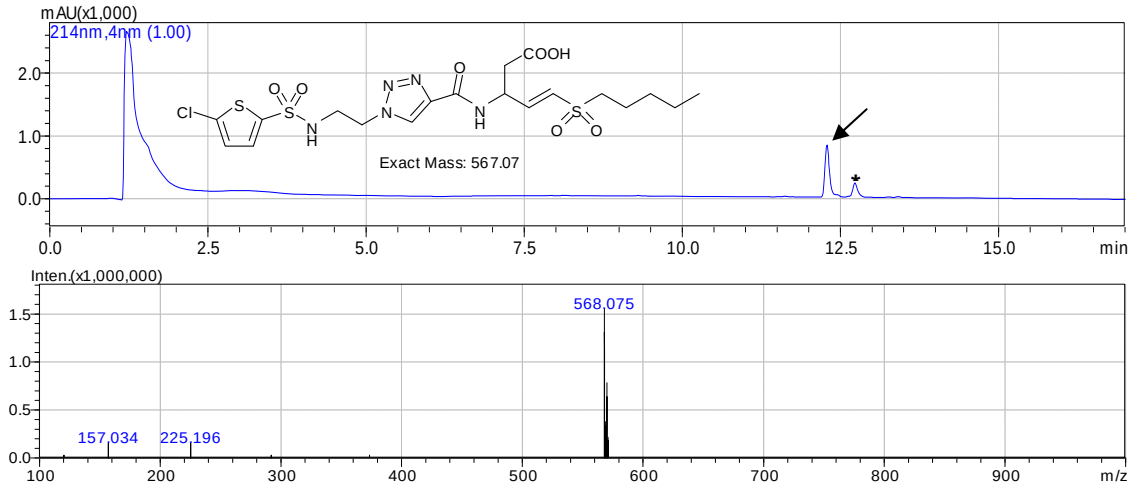

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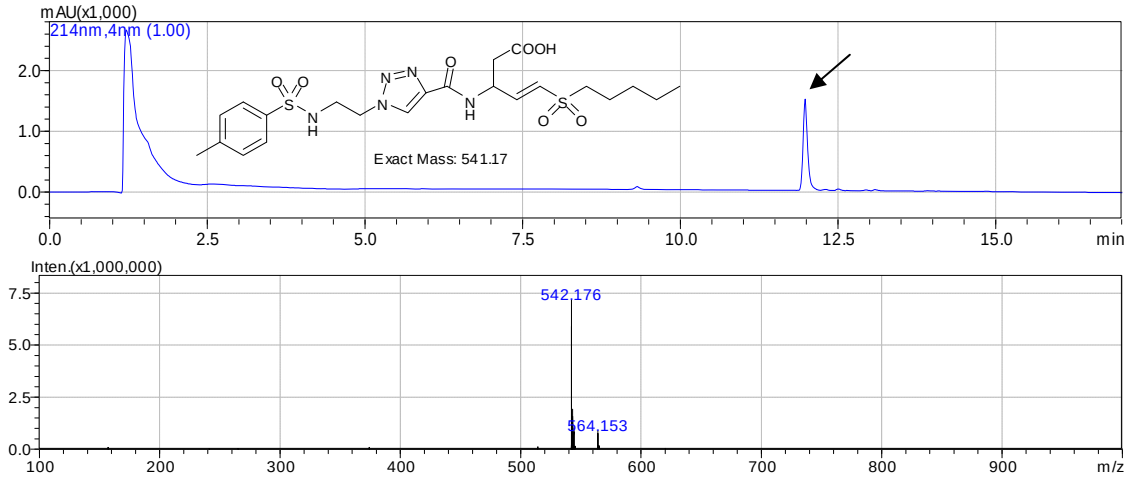

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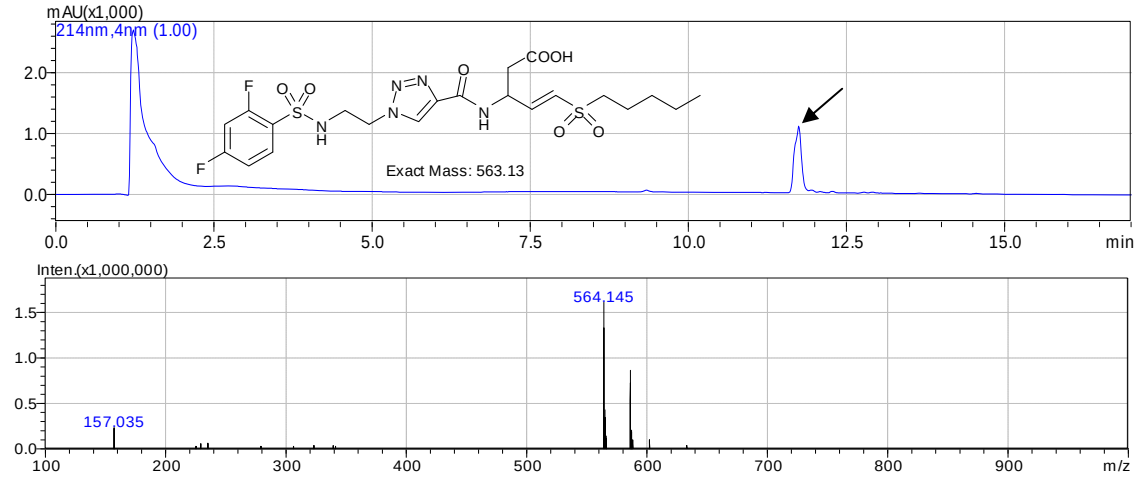

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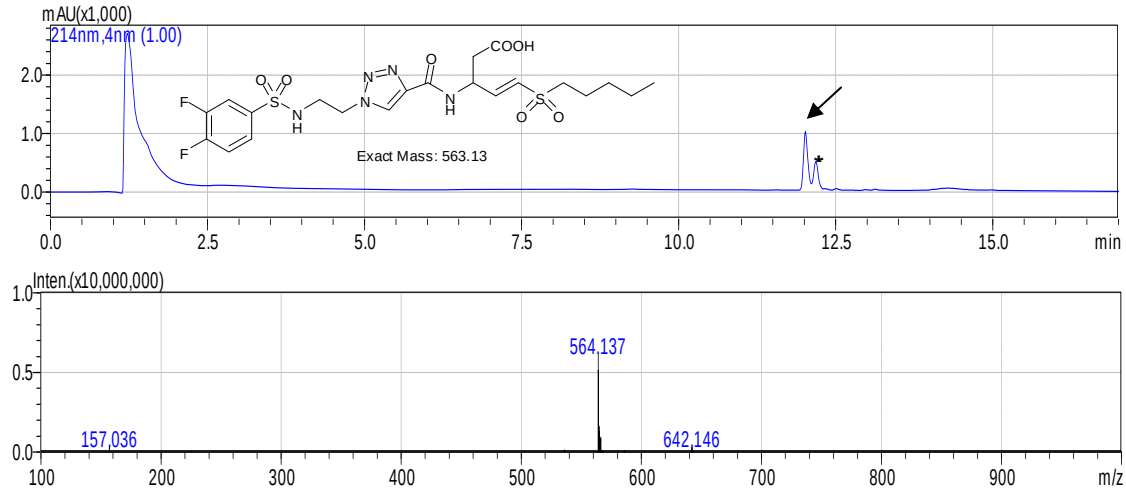

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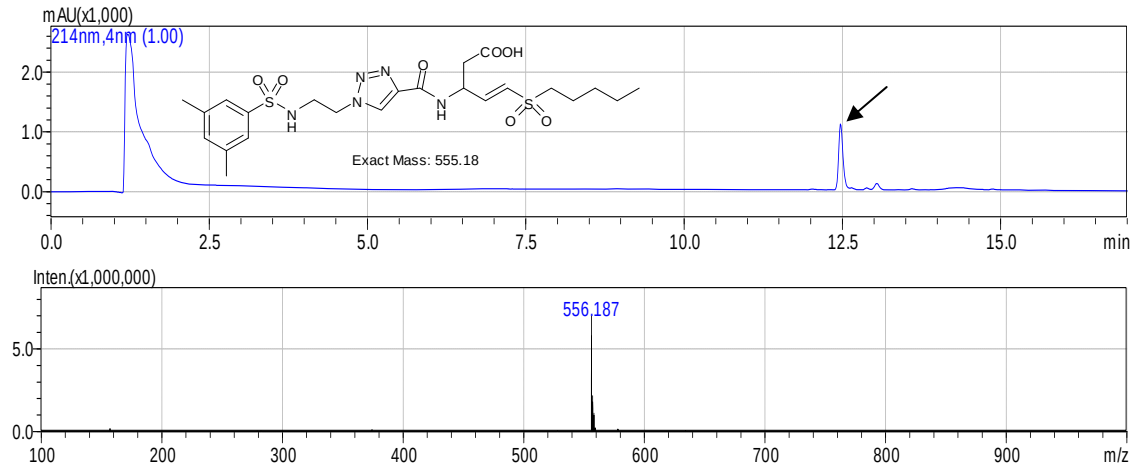

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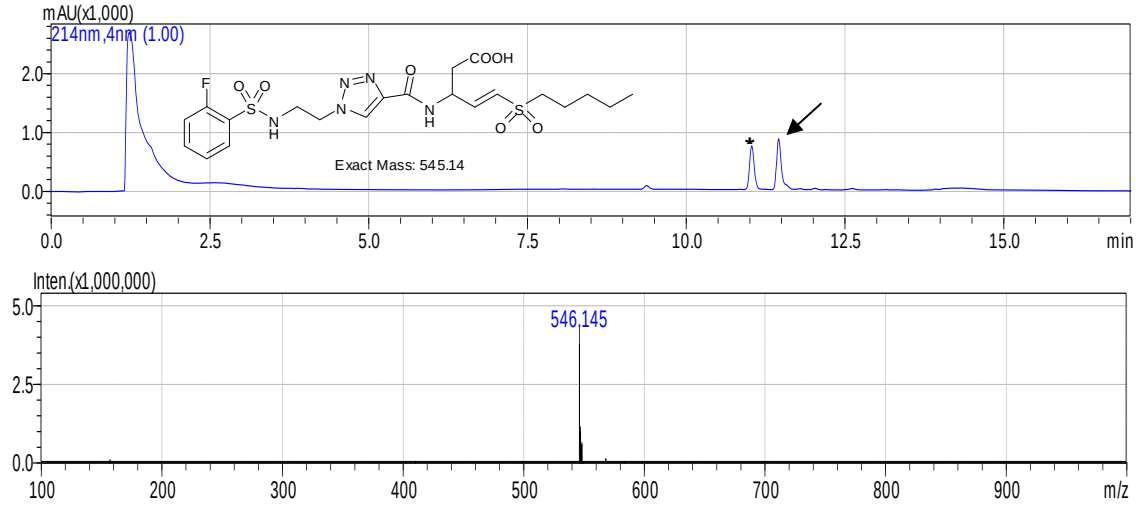

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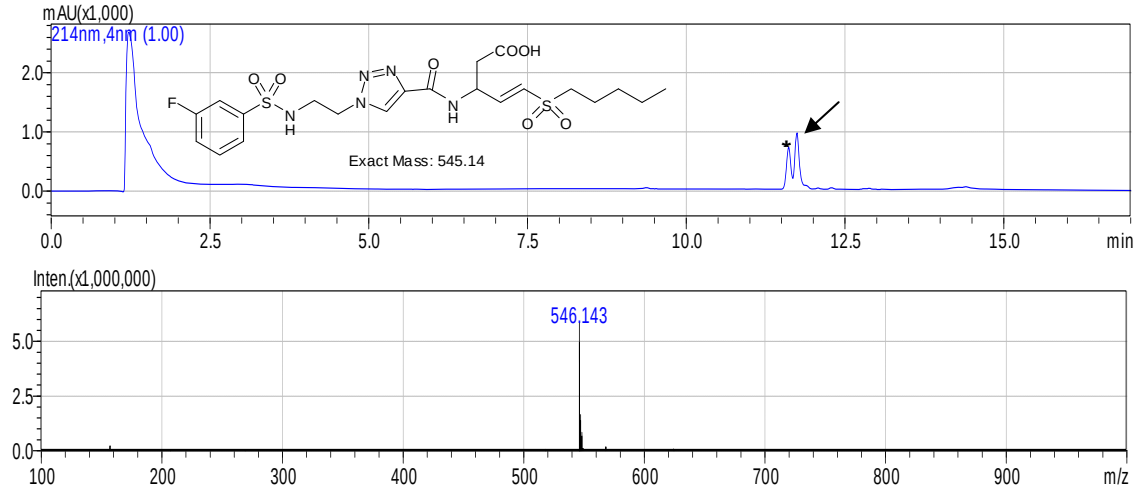

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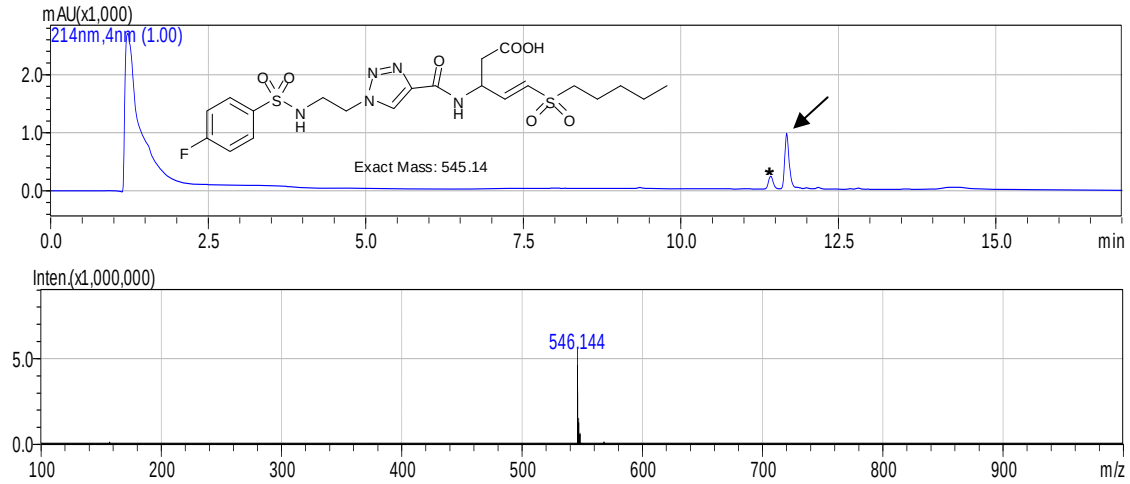

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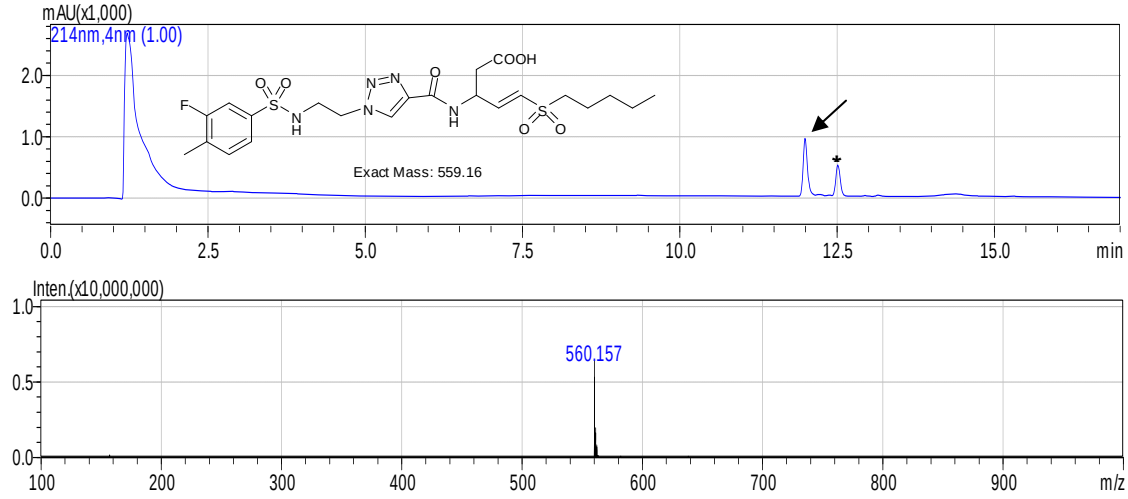

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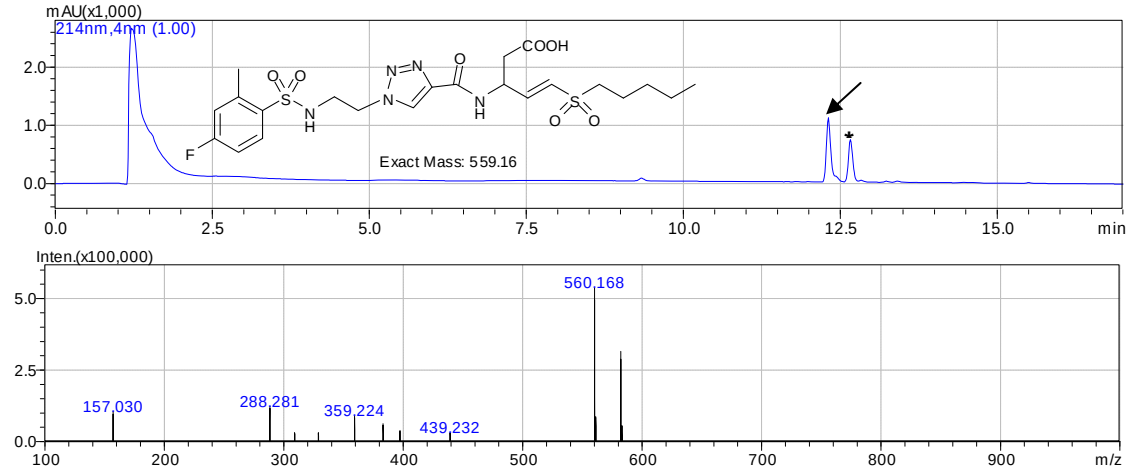

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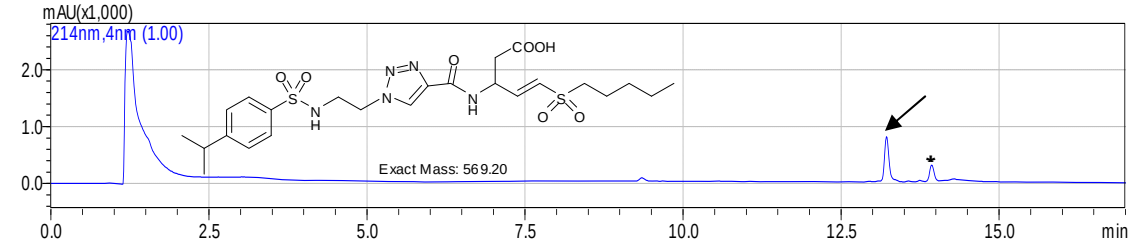

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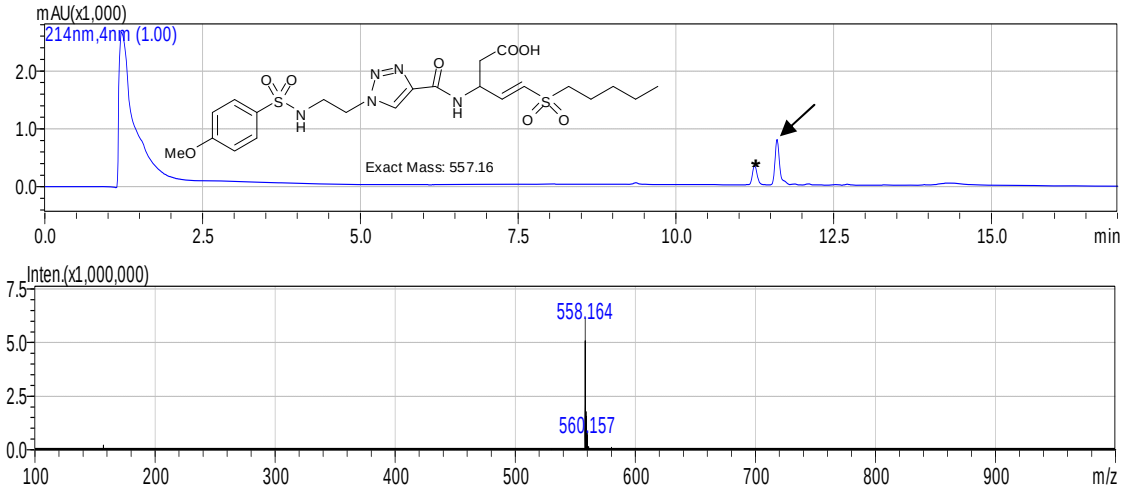

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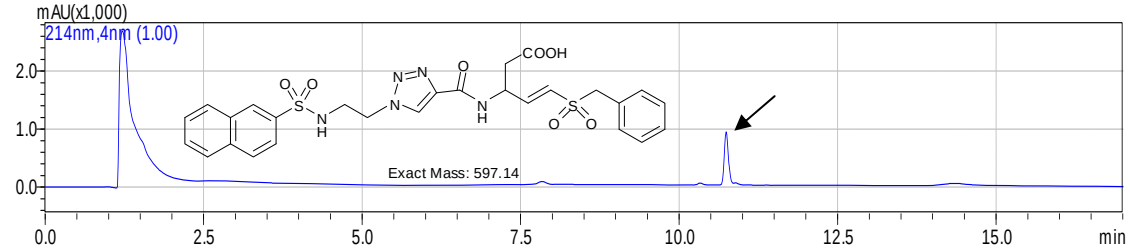

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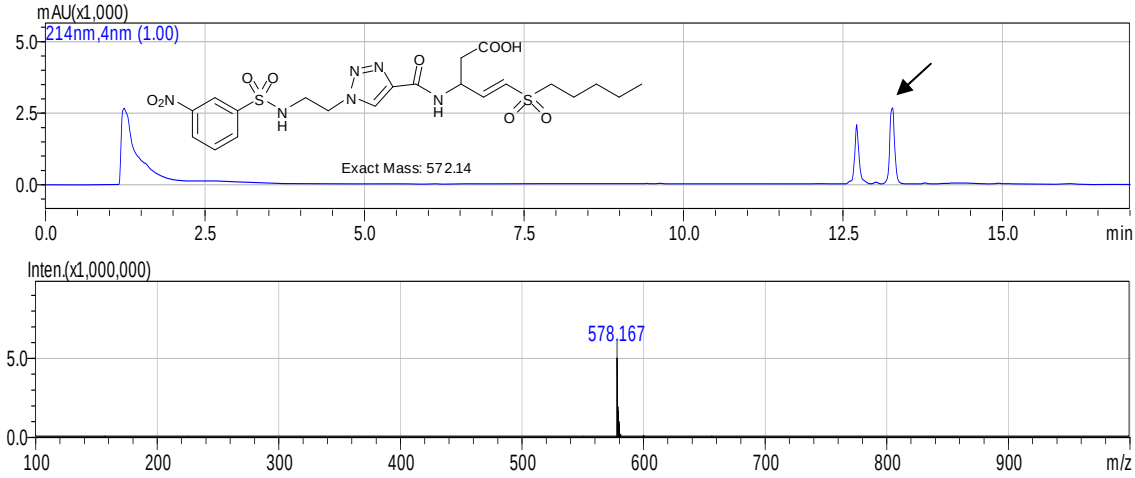

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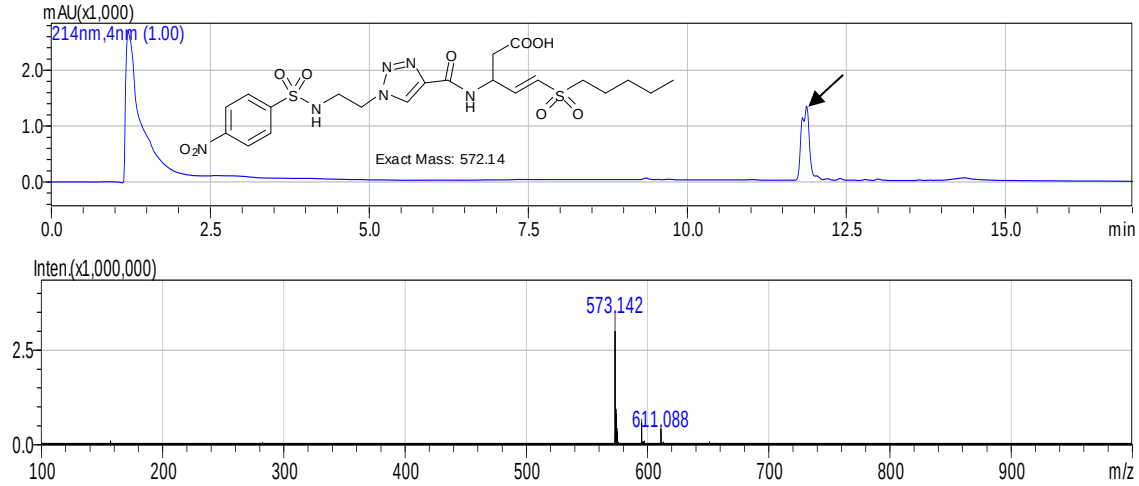

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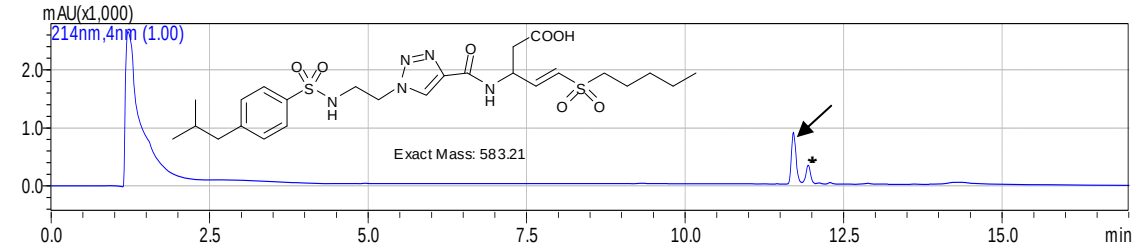
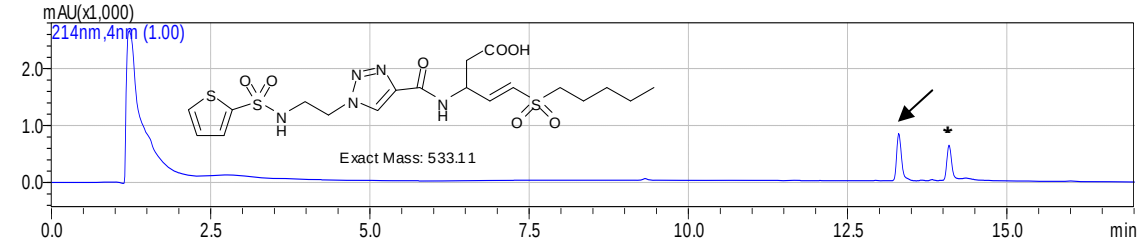


VSP-SB10:



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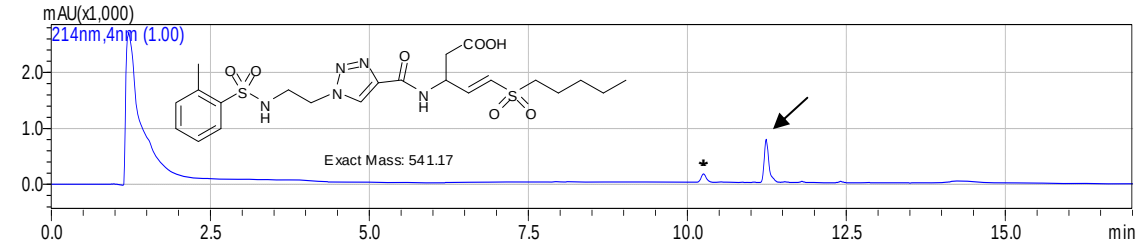

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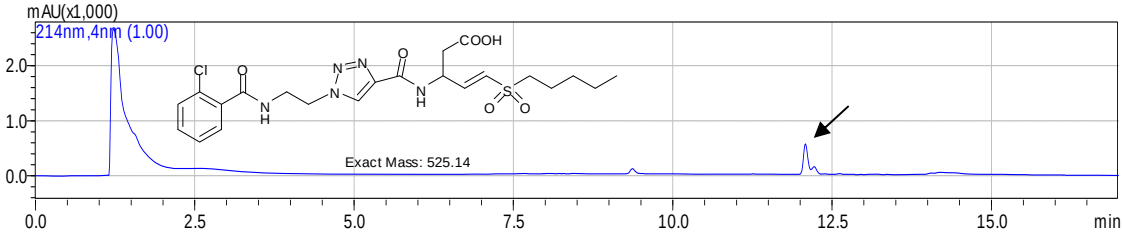
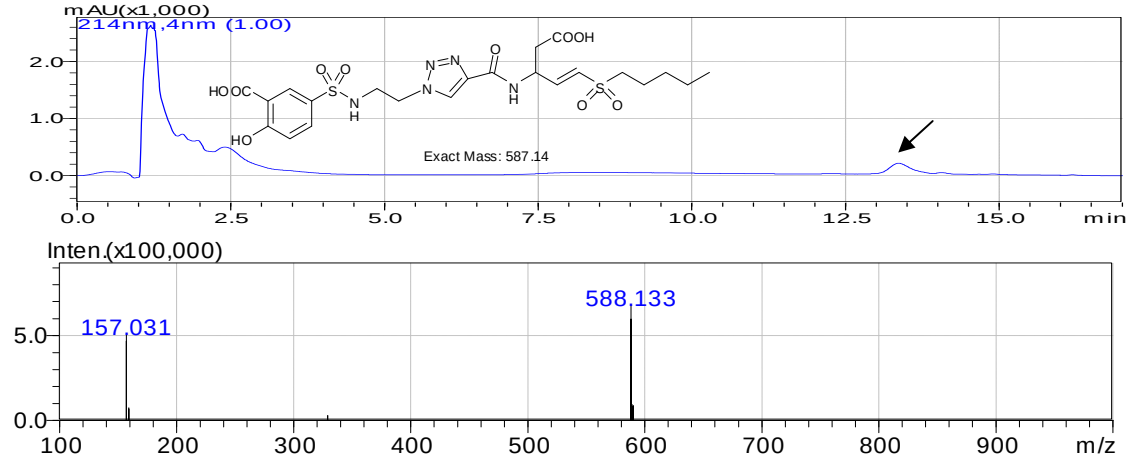
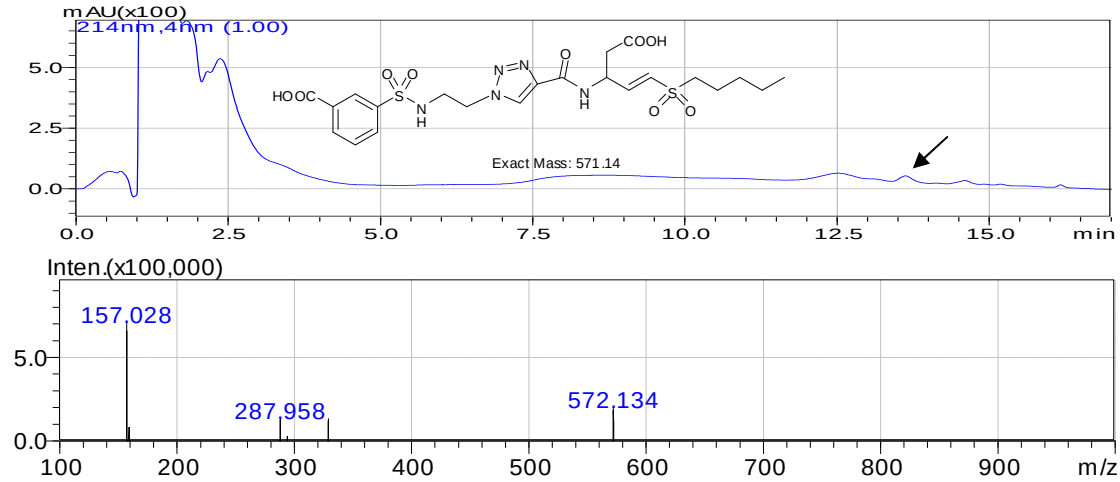


VSP-SC2:



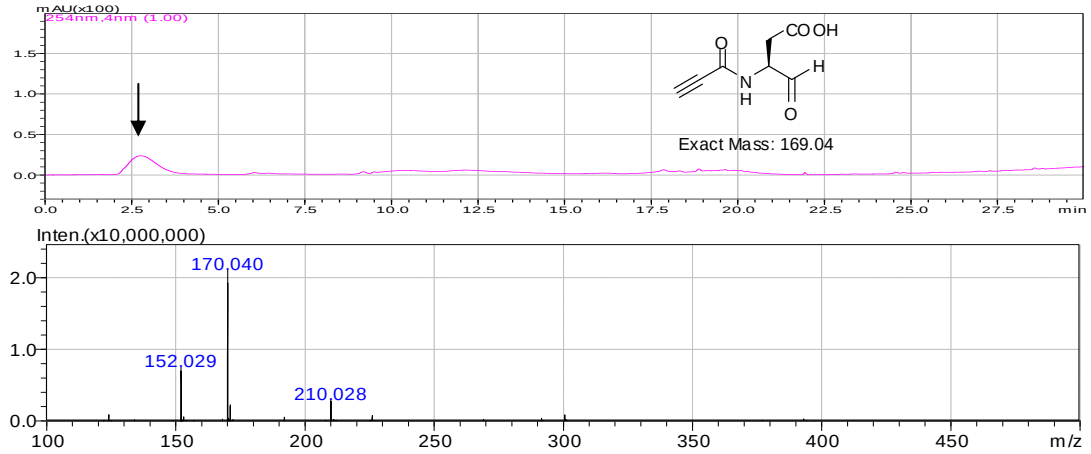
VSP-SC3:



2.3 LC-MS profiles of aldehyde inhibitors (19)

All conditions are based on:
0 – 10 min: 5% B (1% TFA in ACN) → 20% B
10 – 15 min: 20% B (1% TFA in ACN) → 80% B
15 – 20 min: 80% B (1% TFA in ACN) → 100% B
20 – 25 min: 100% B
* Starting materials

Ald-warhead:



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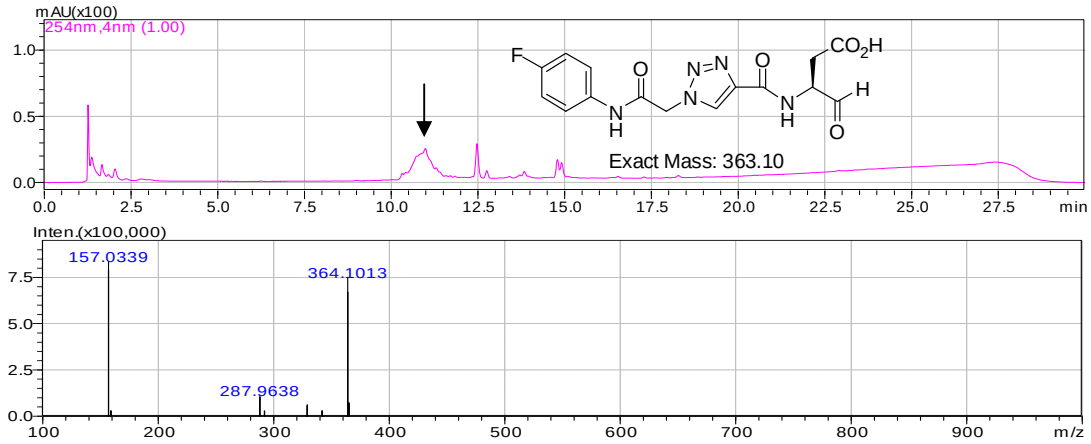

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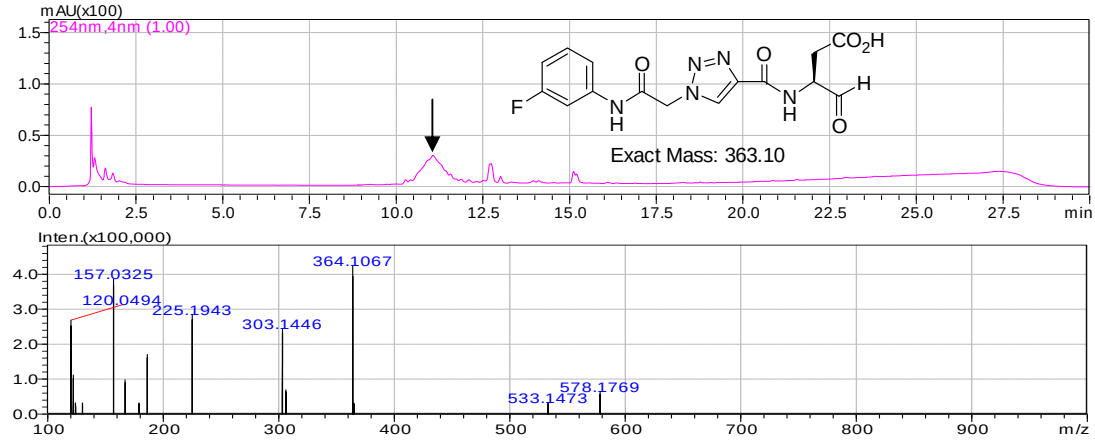
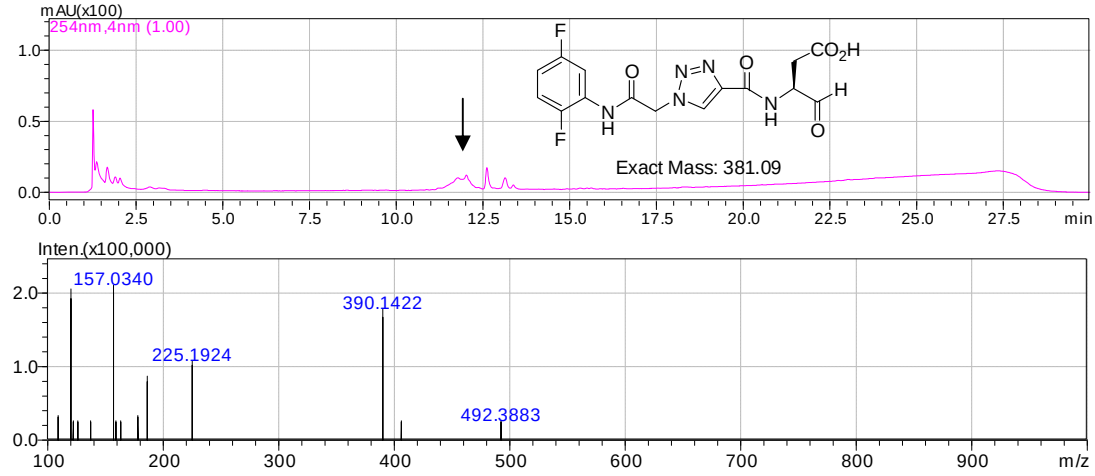


Ald-A3:



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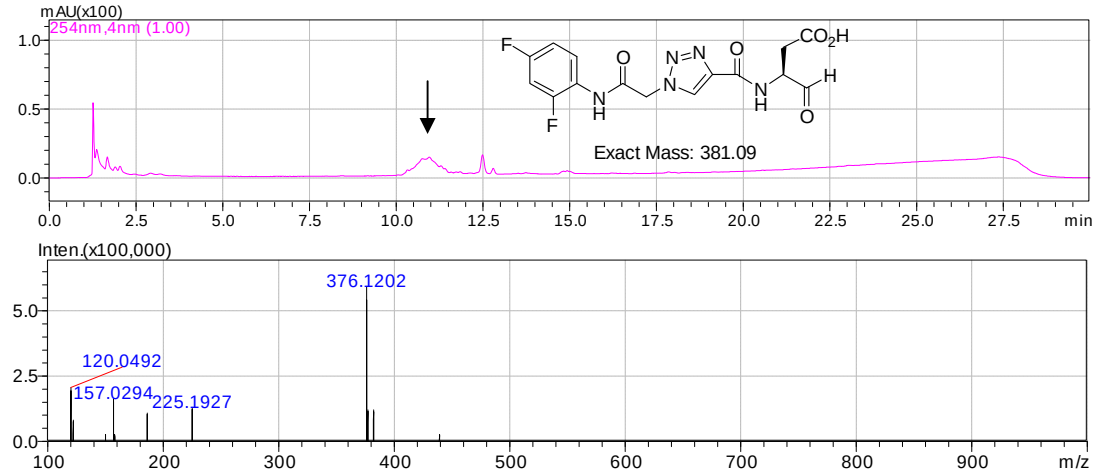
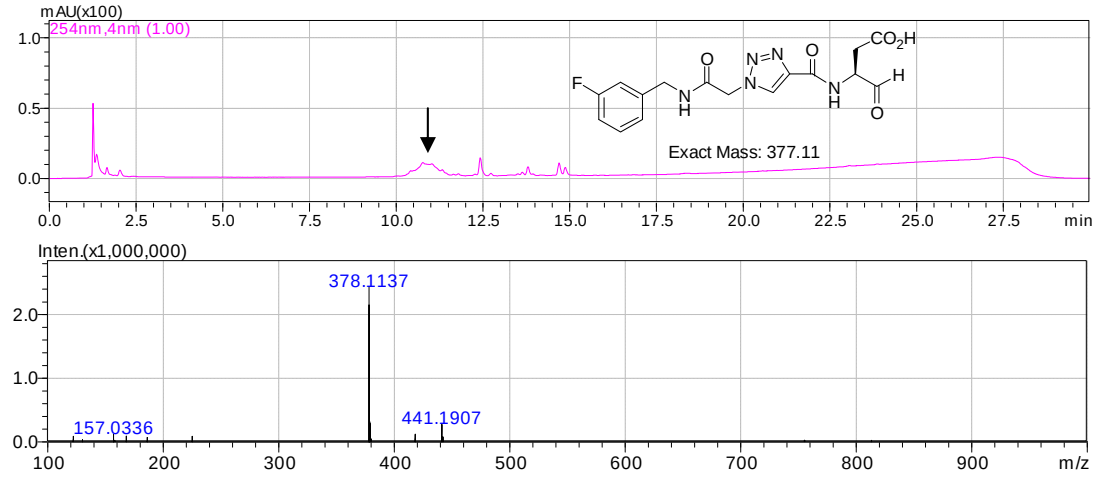
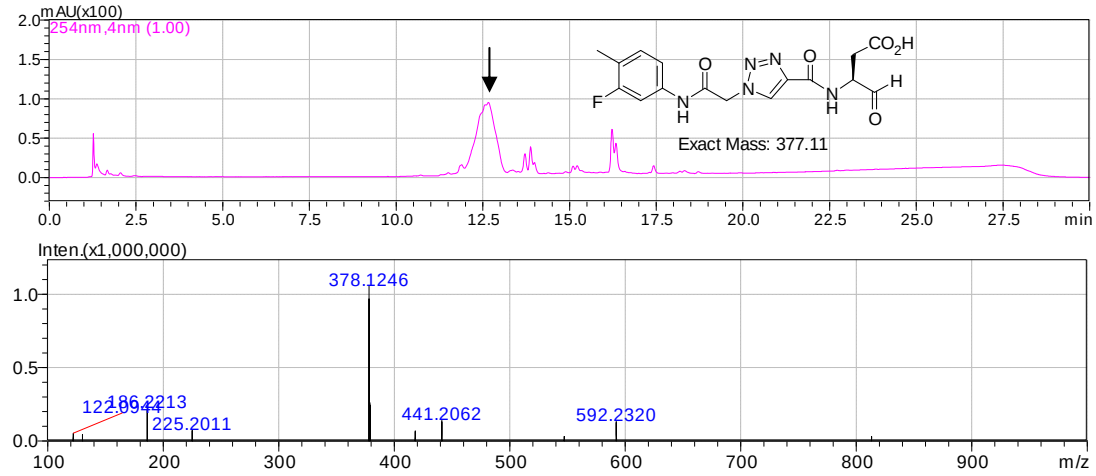


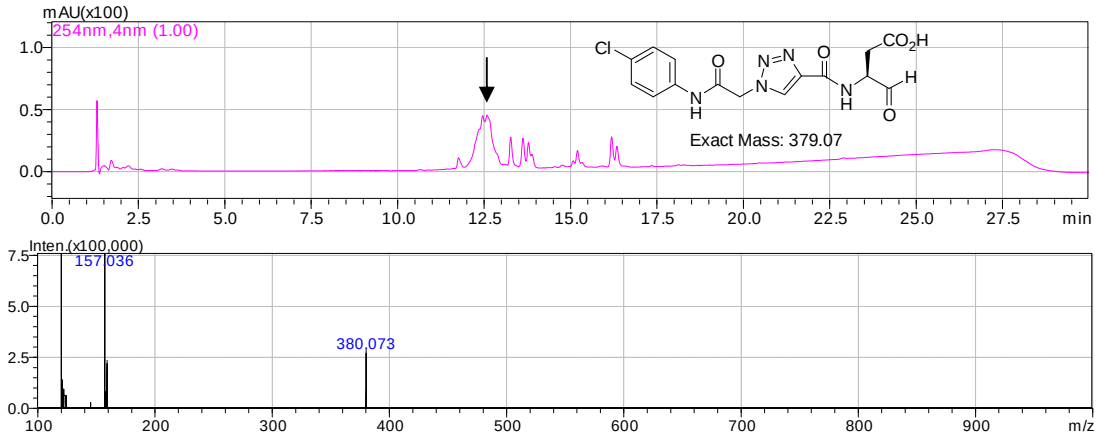
Ald-A5:



Ald-A6:



Ald-A8:



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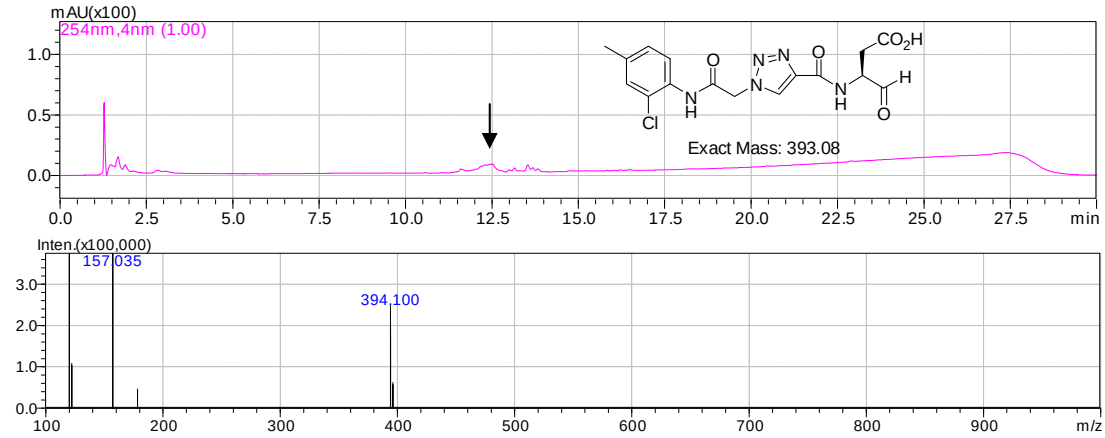
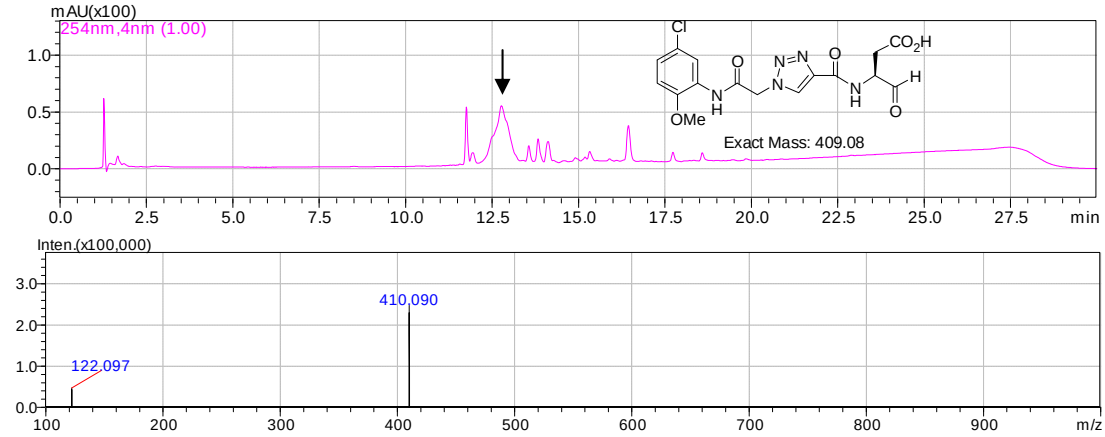


Ald-A11:



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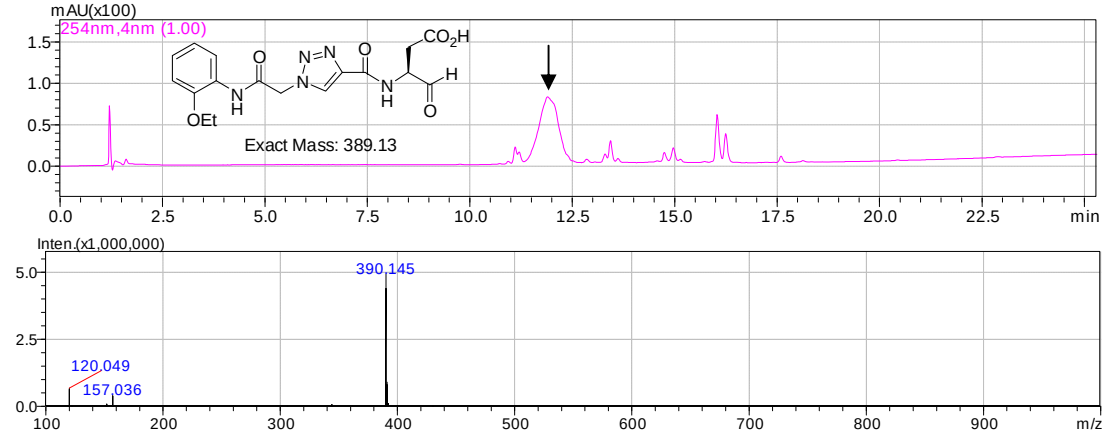
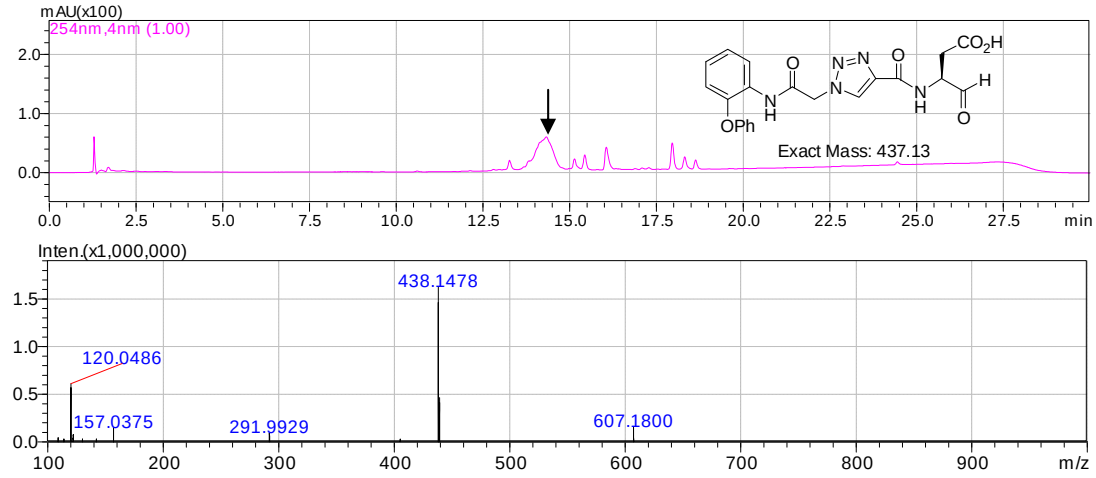


Ald-B1:



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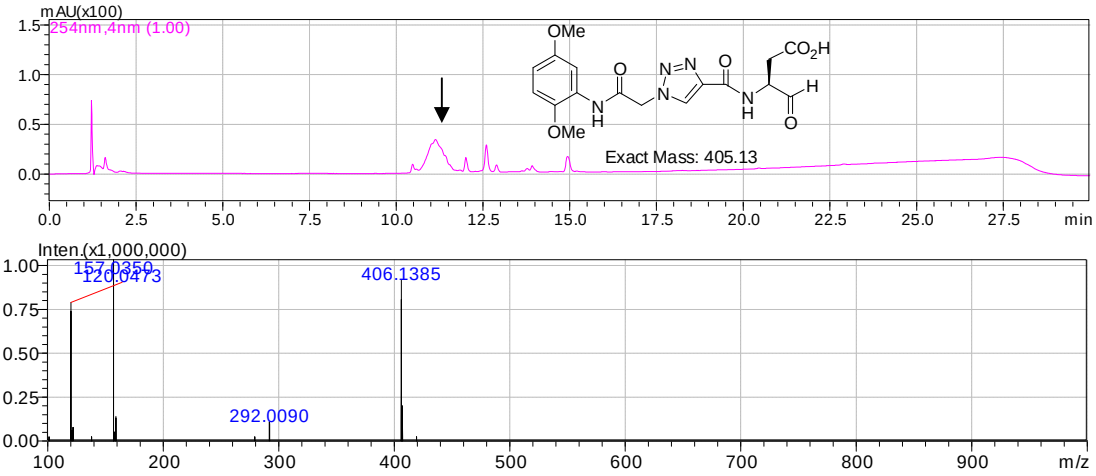

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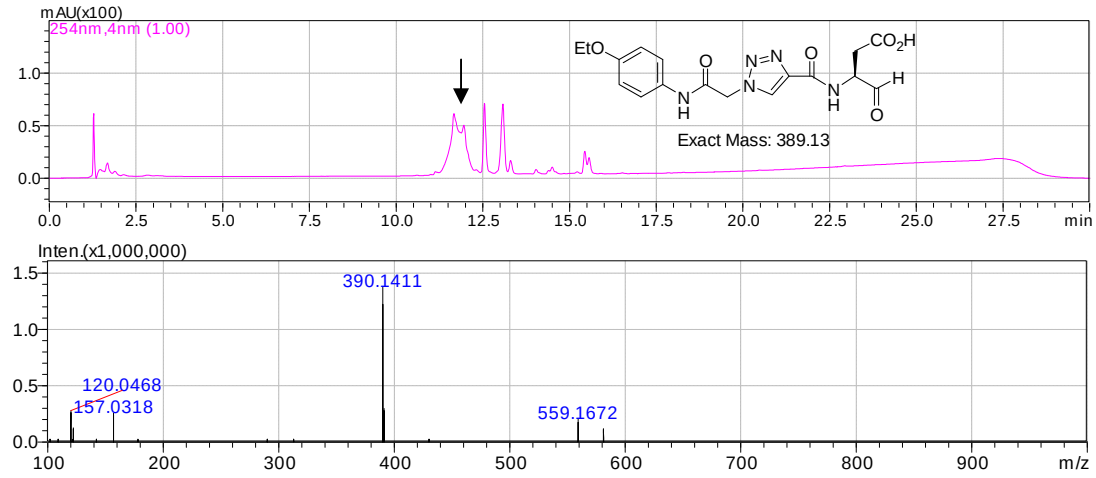
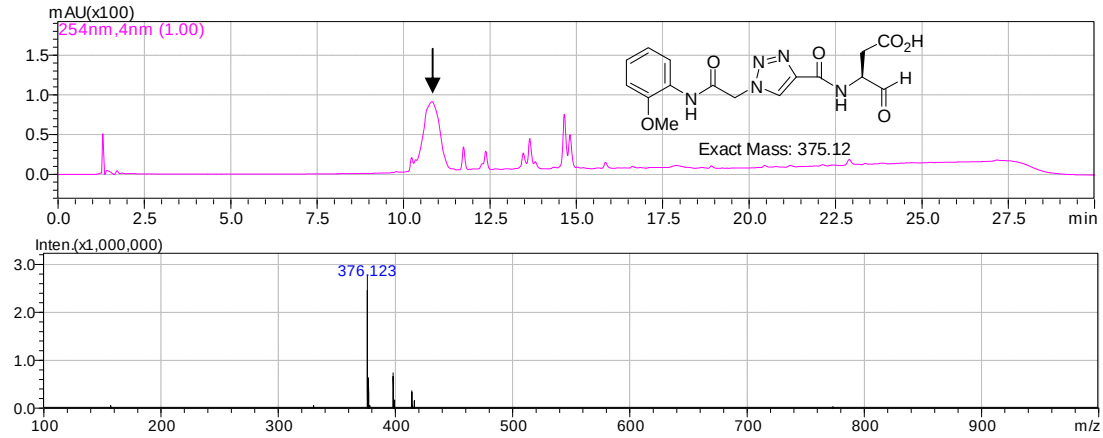
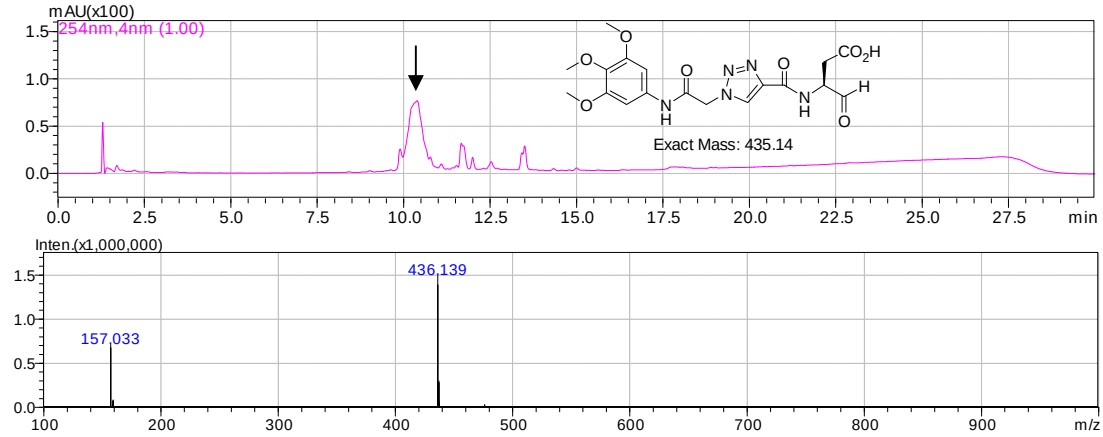


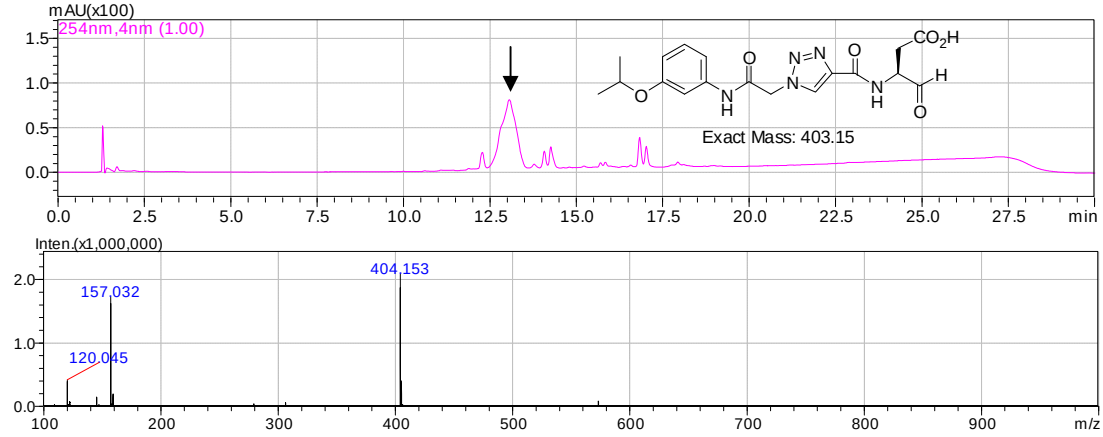
Ald-B4:



Ald-B5:



Ald-B6:



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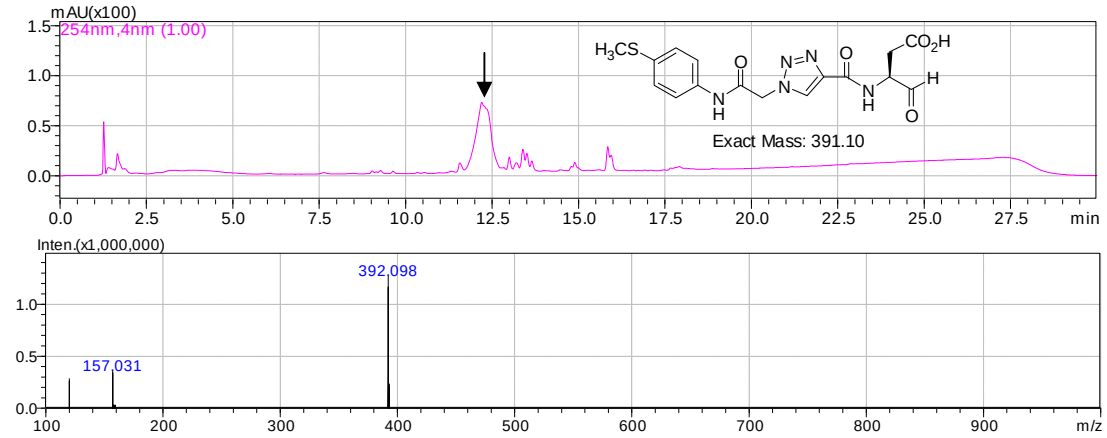

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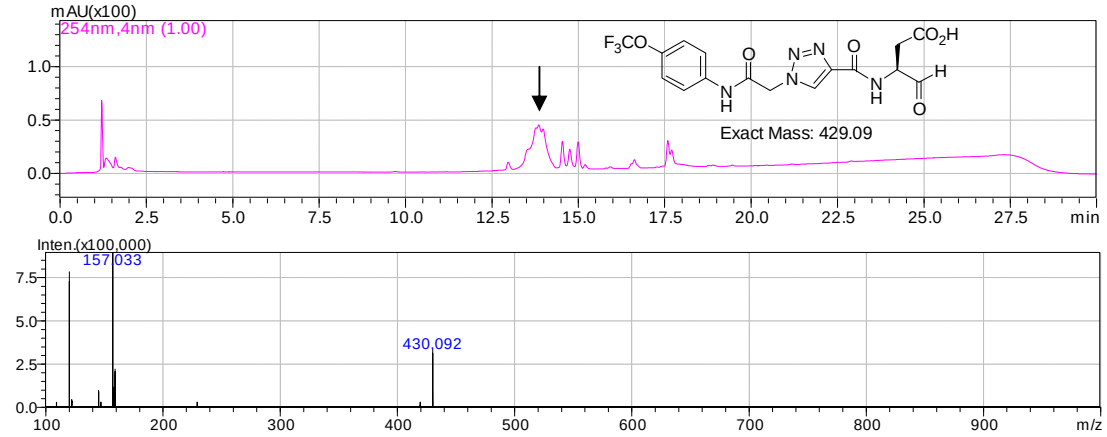

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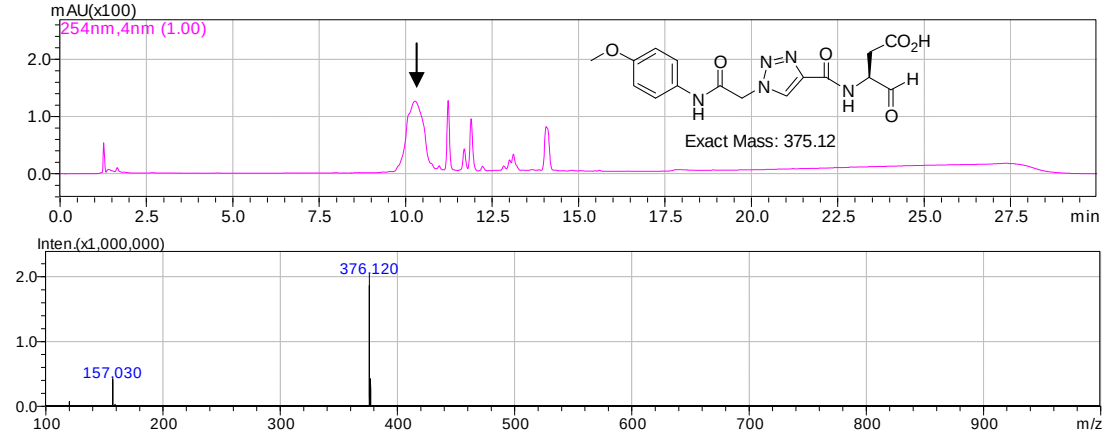

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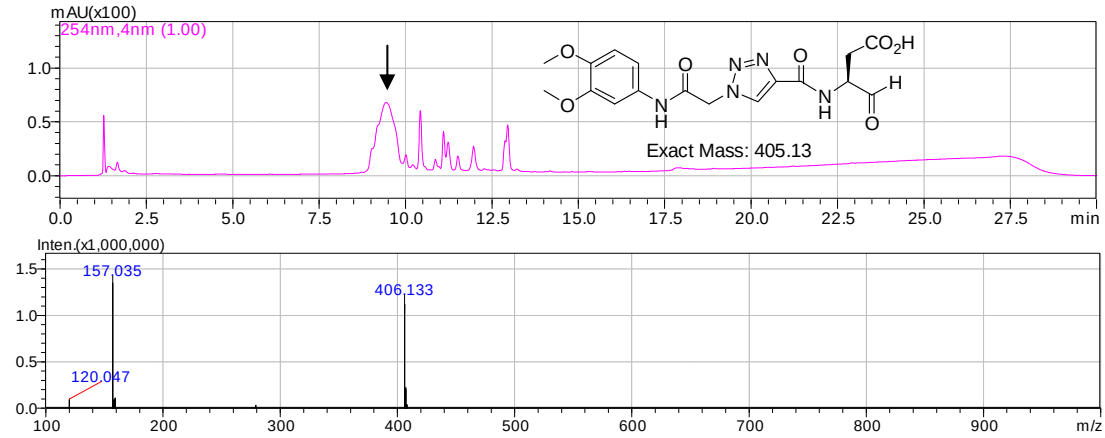

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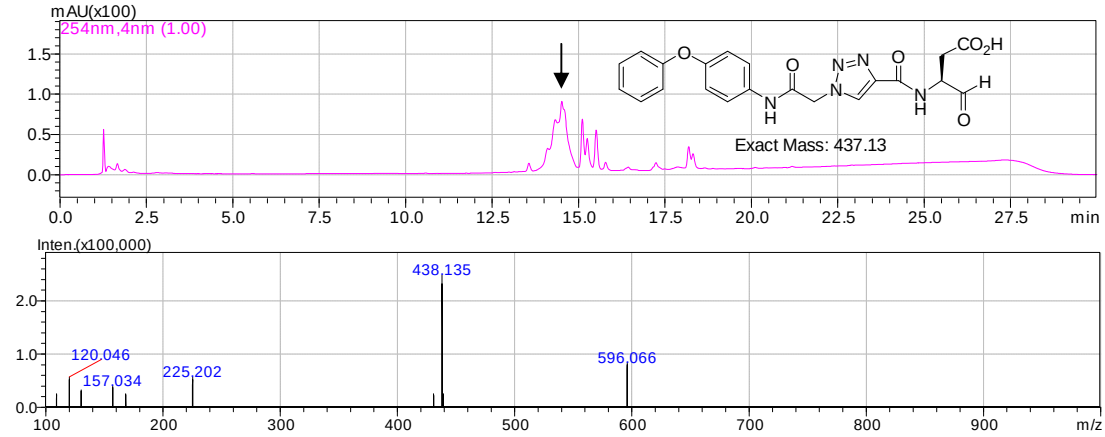

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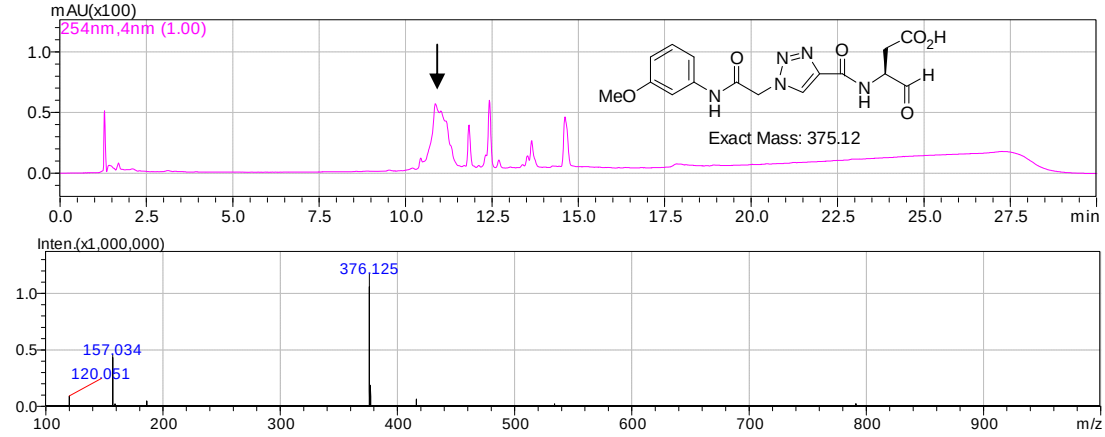

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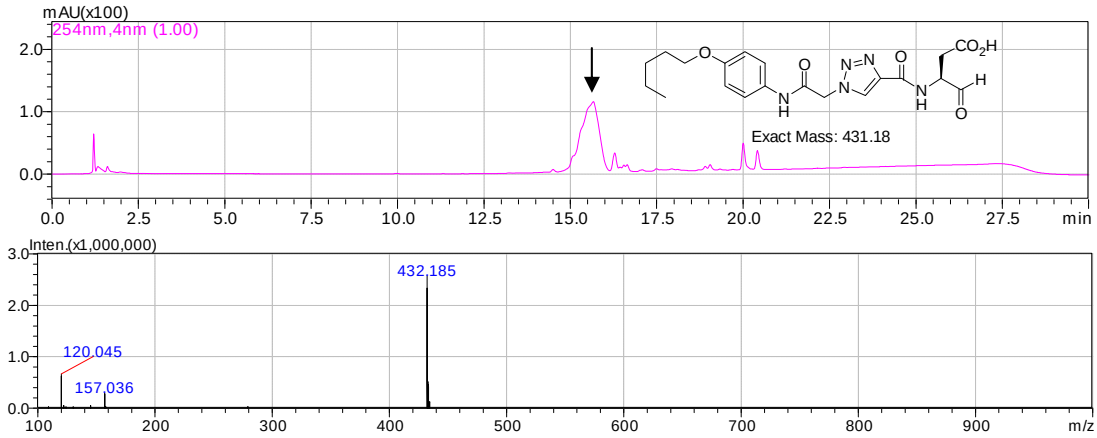

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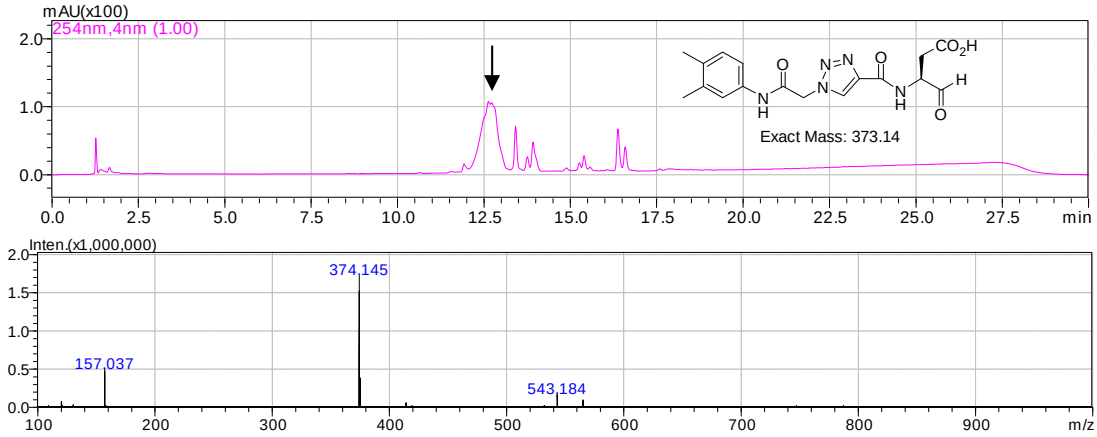

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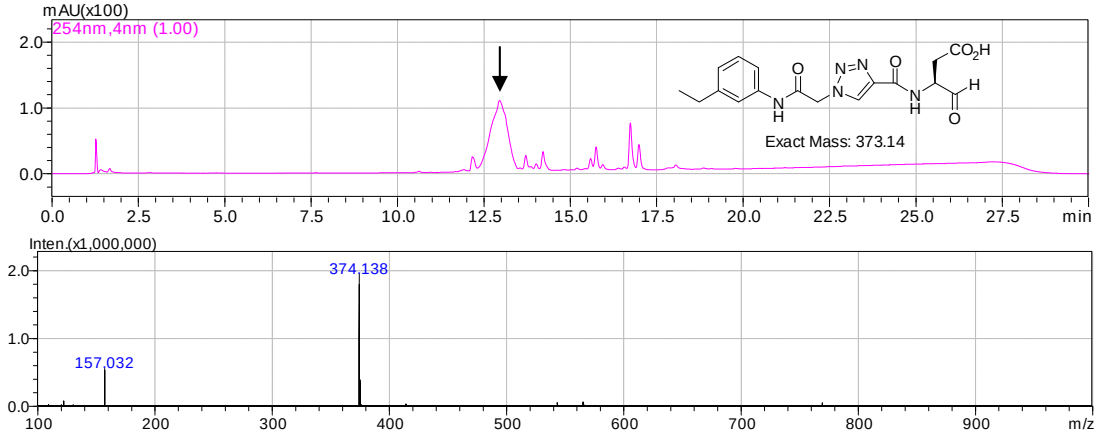

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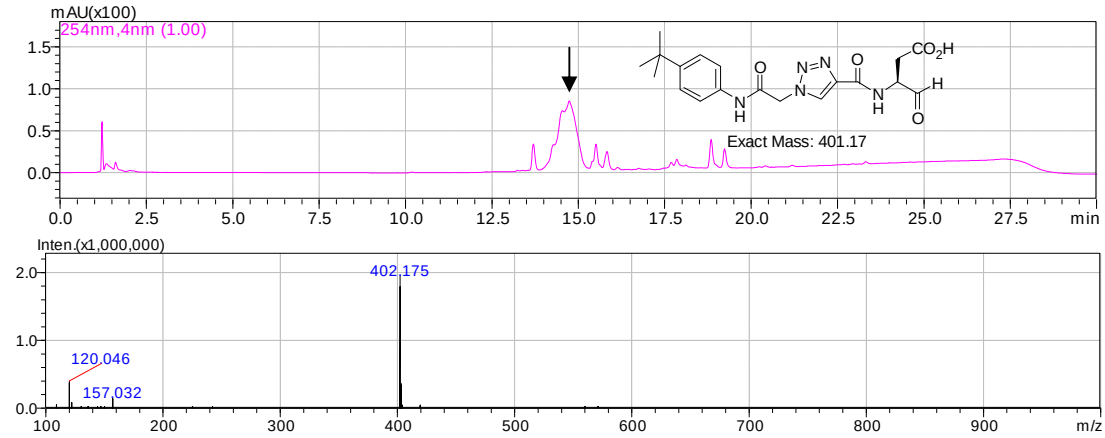
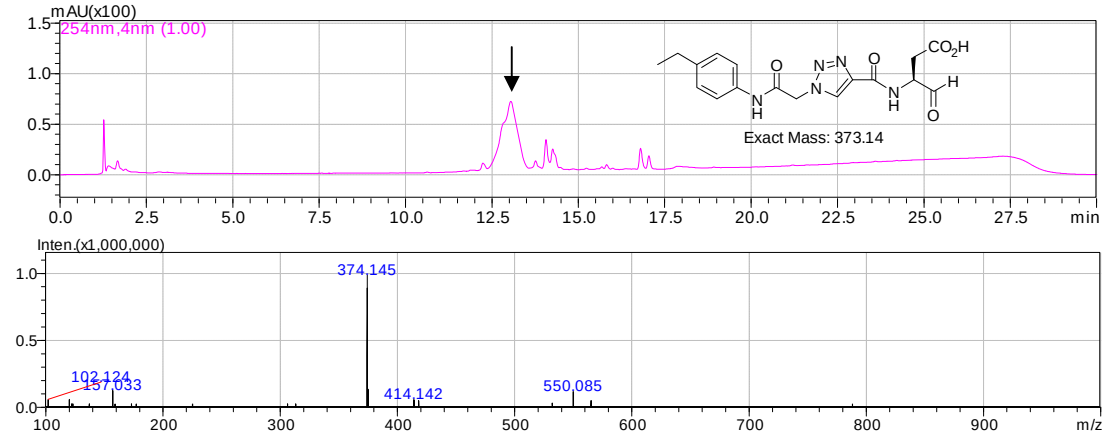


Ald-C7:



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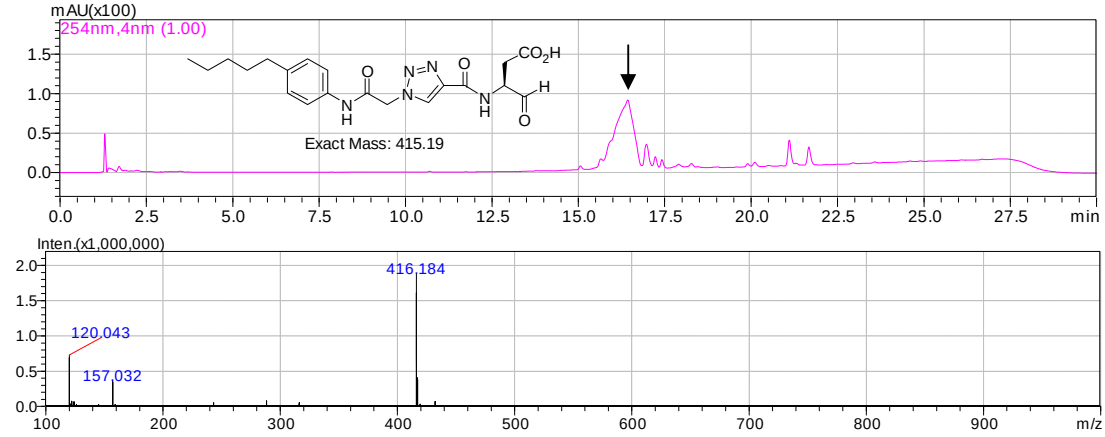

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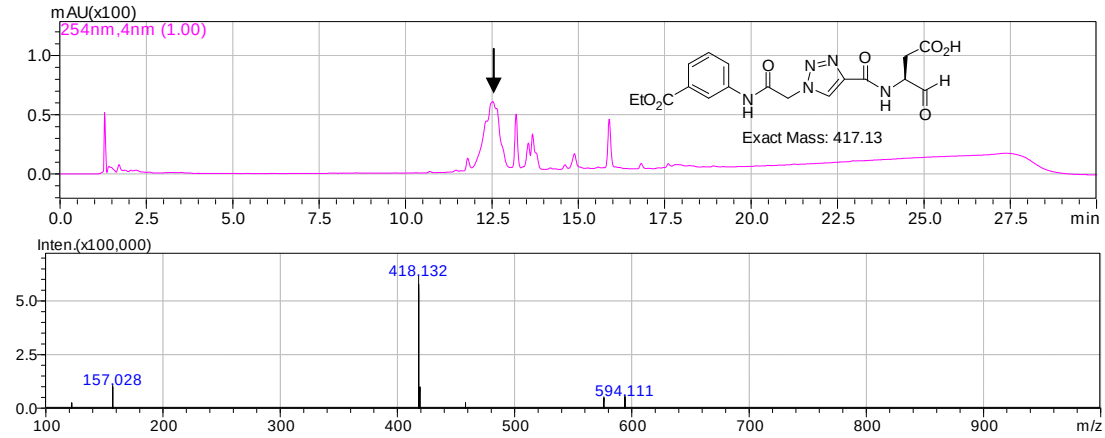

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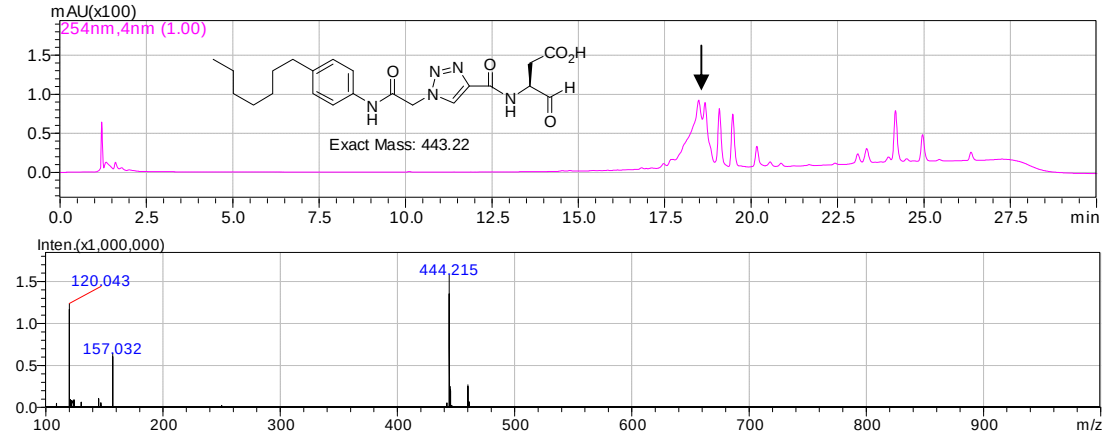

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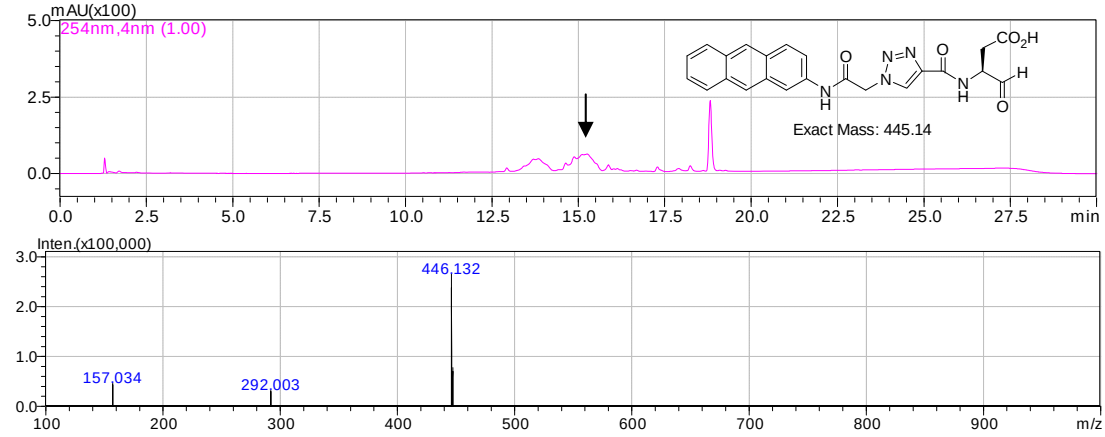
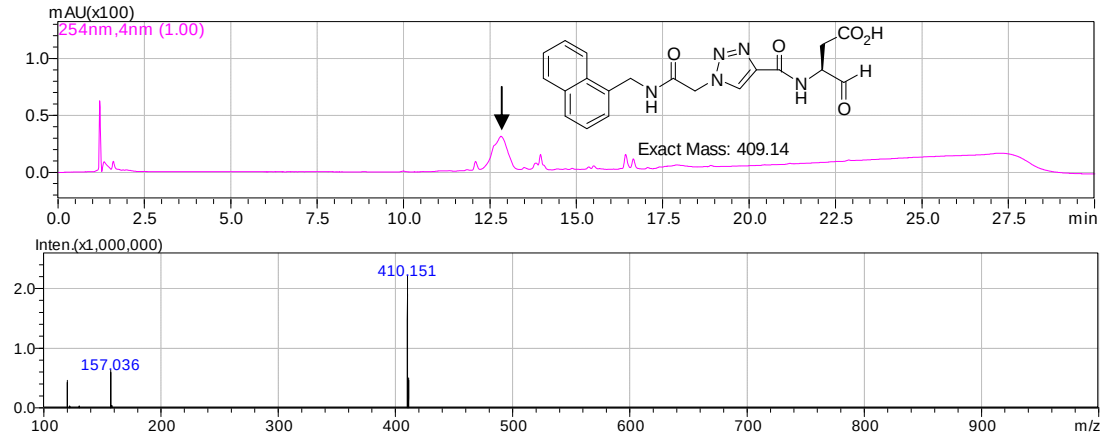


Ald-D4:



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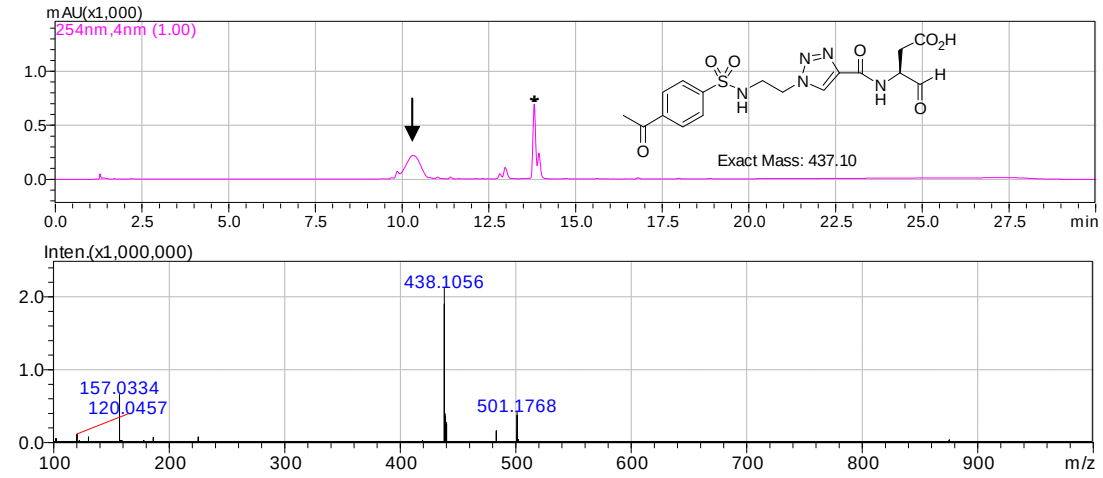

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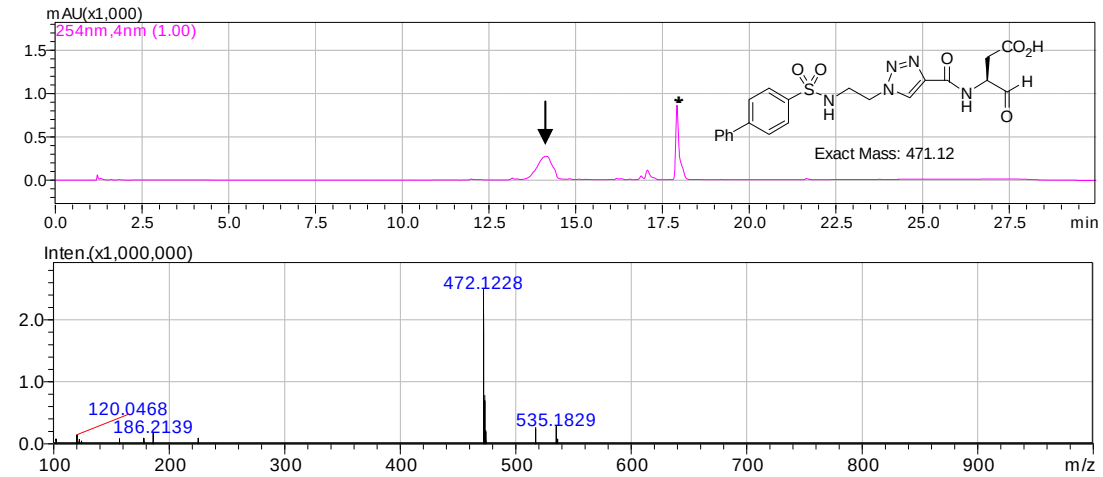

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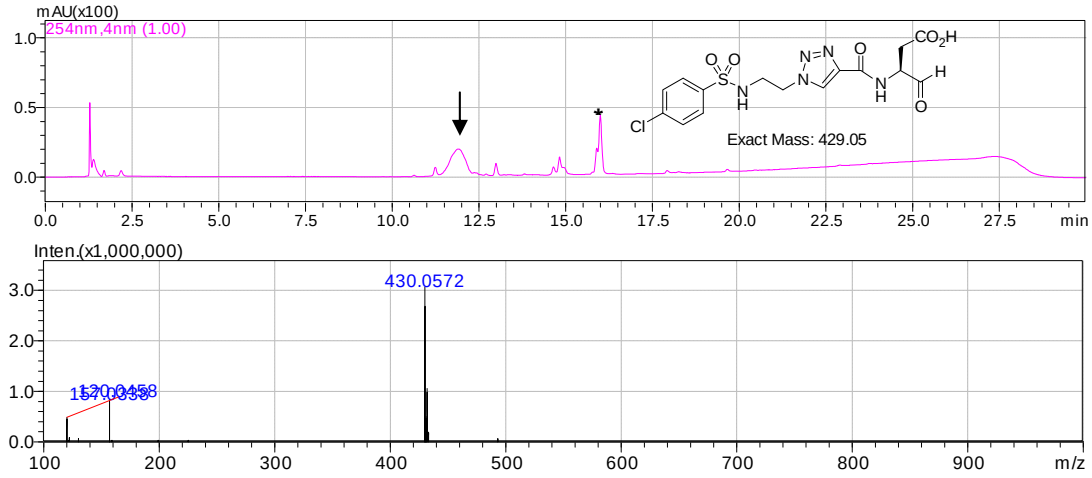

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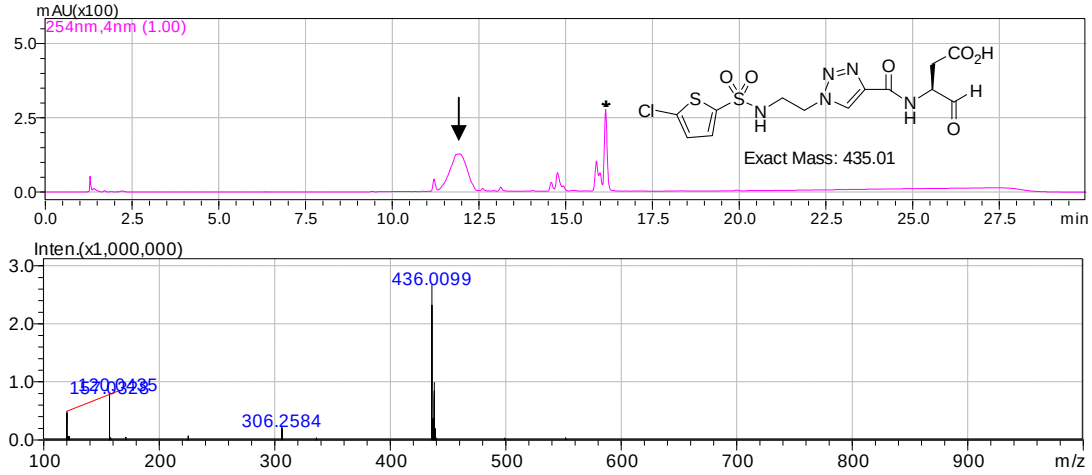

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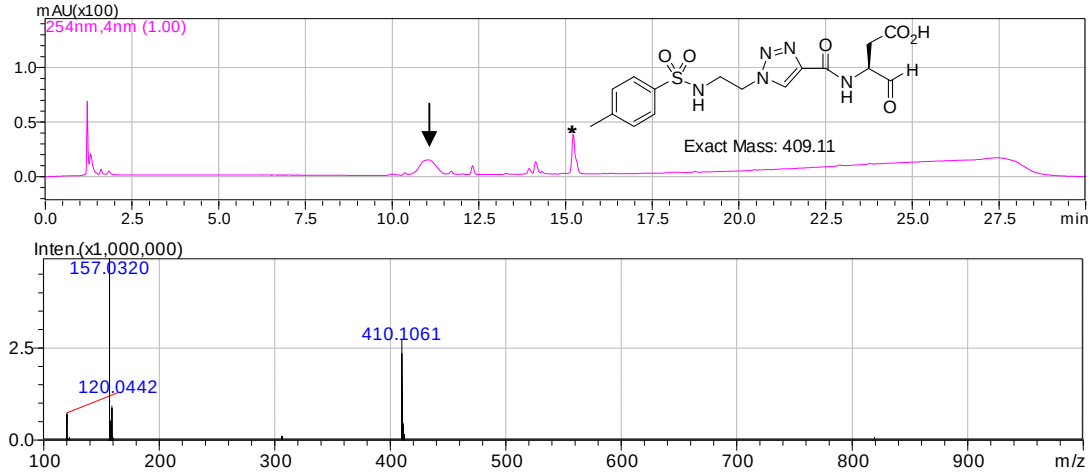

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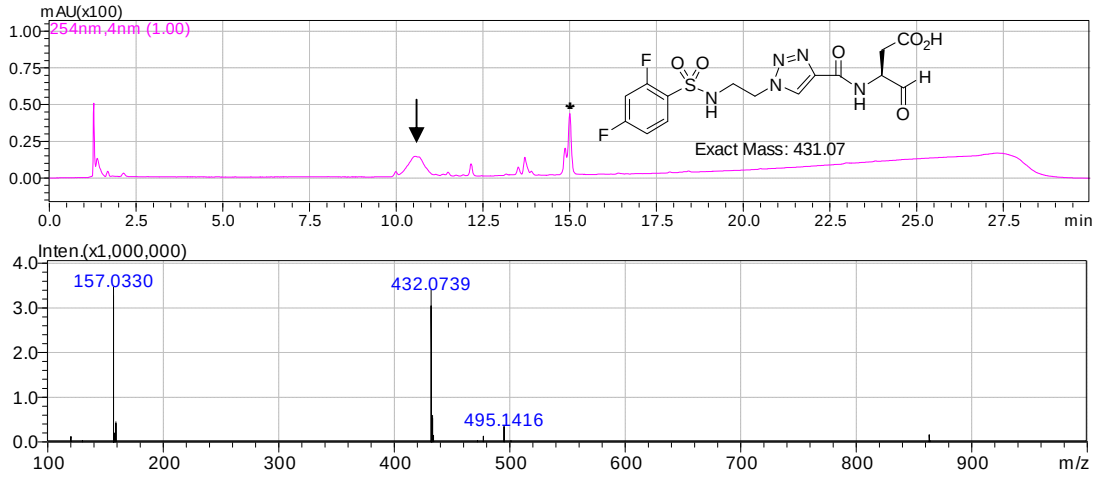

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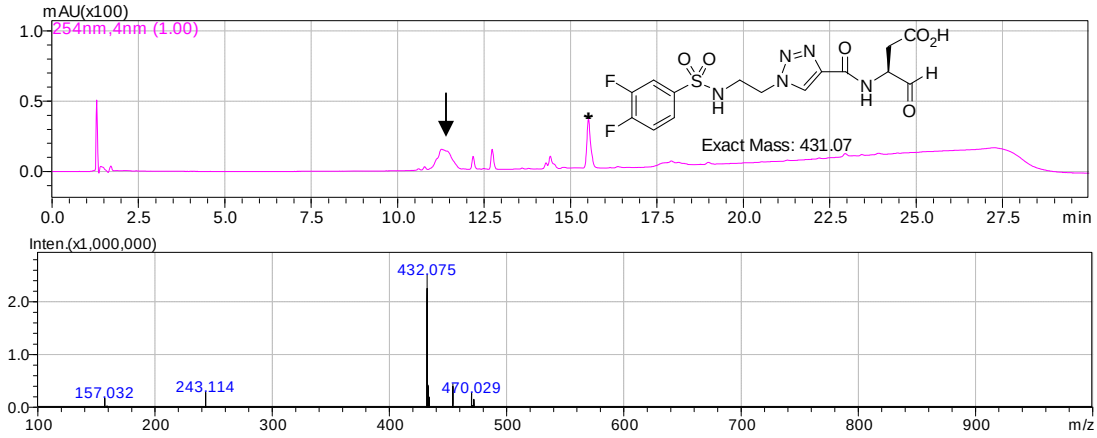

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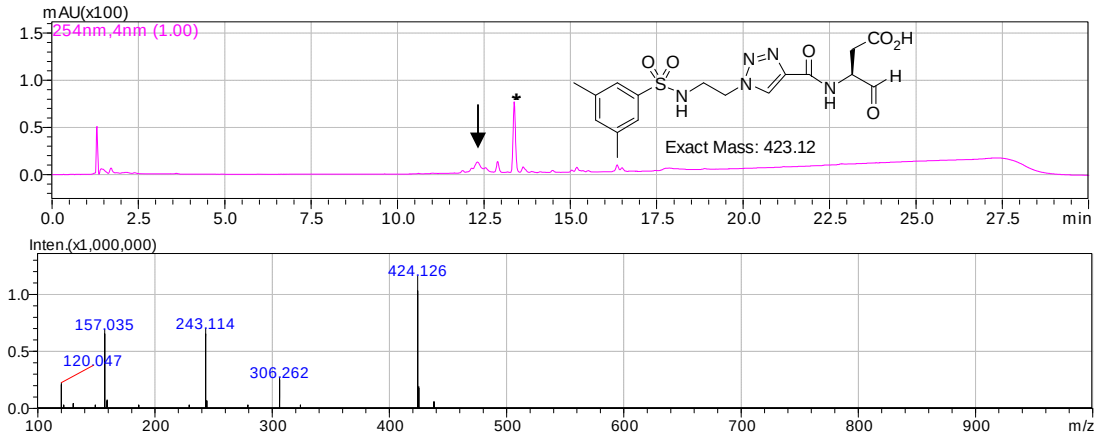

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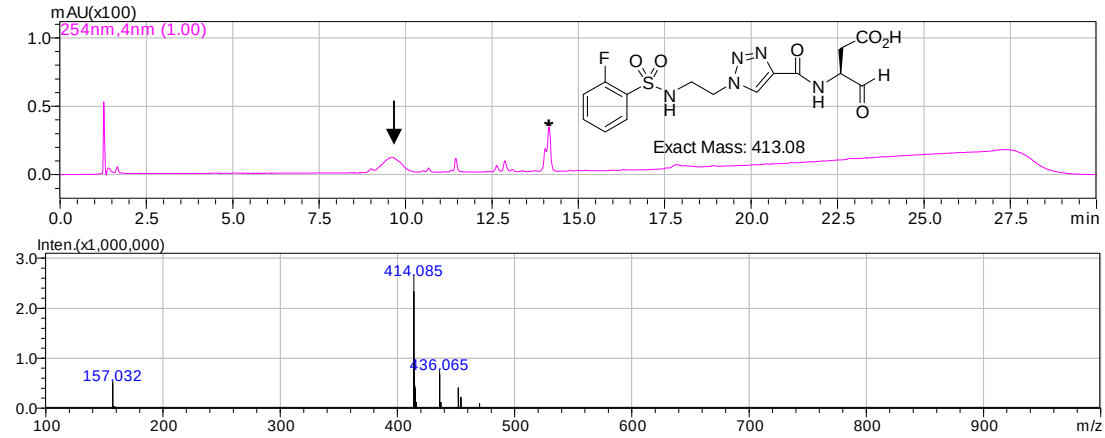

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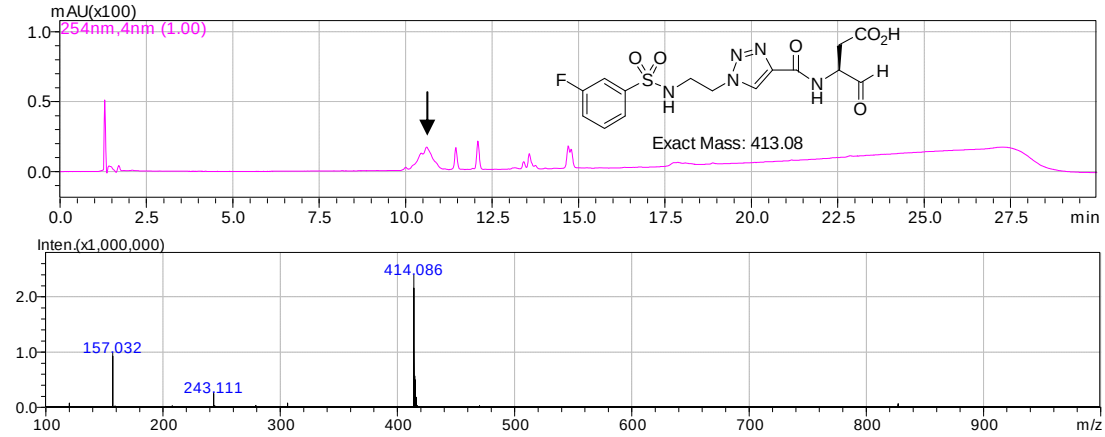
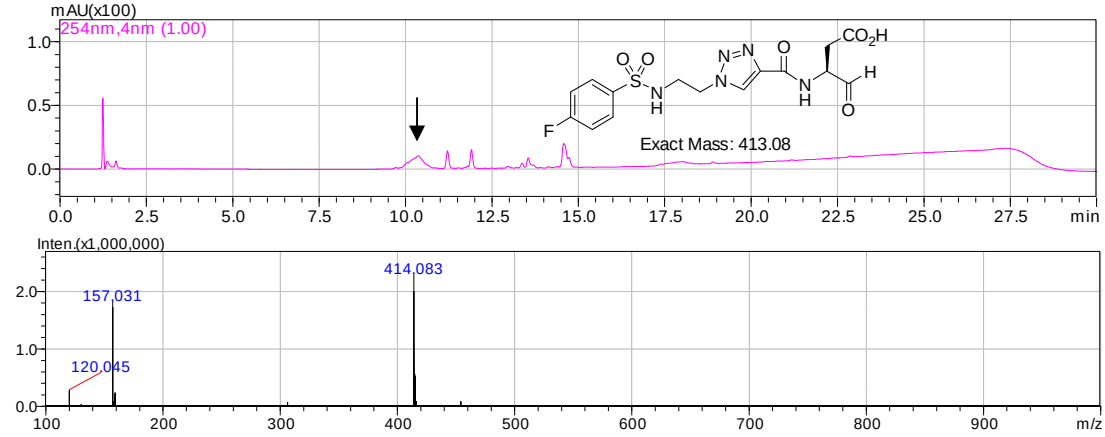


Ald-SA11:



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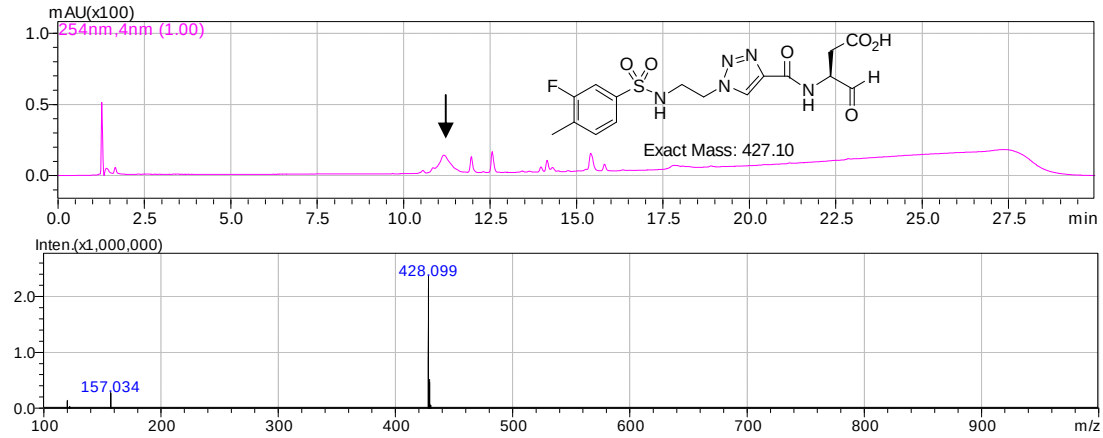

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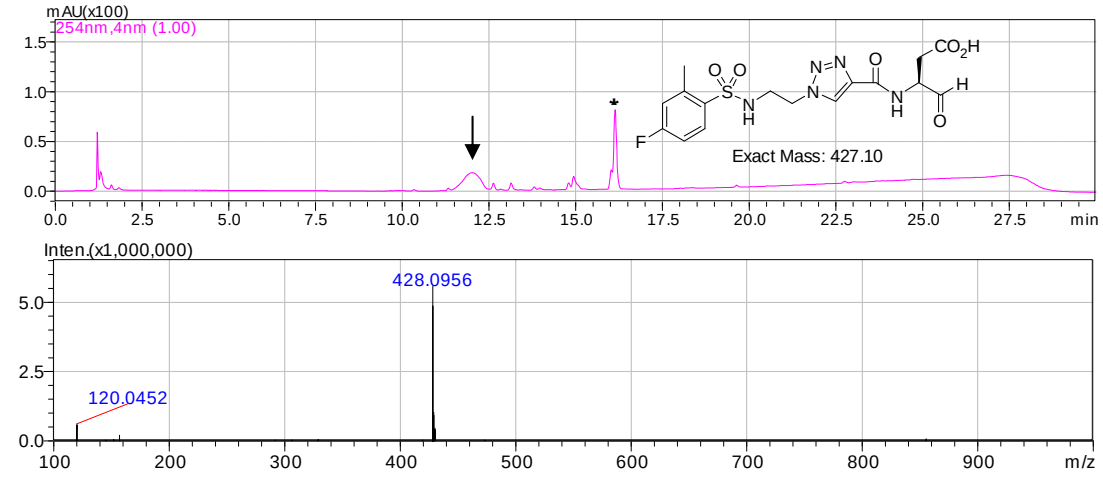

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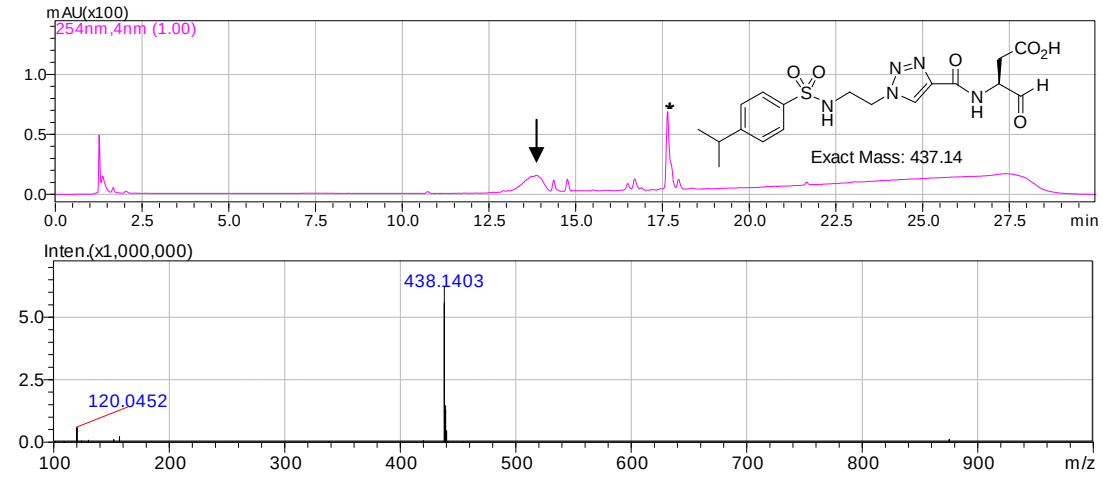
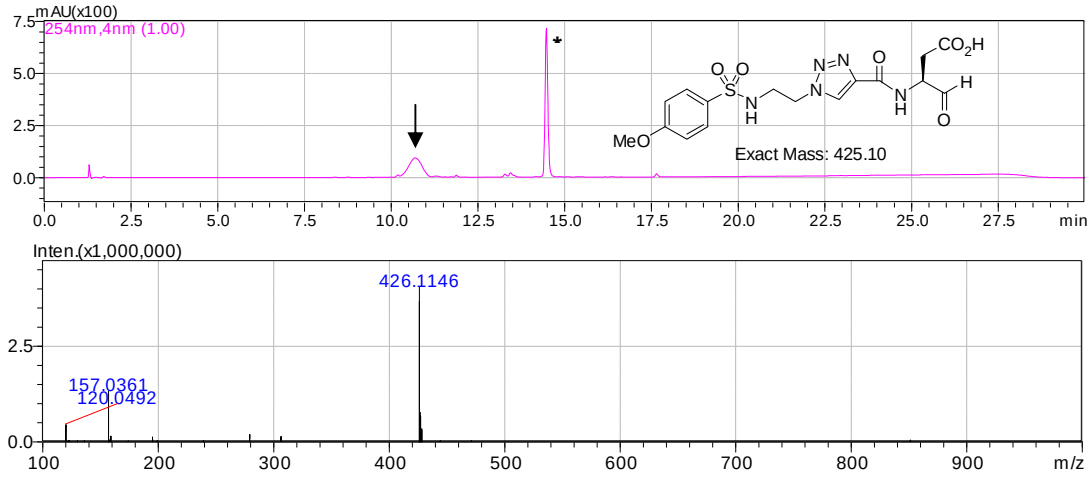


Ald-SB3:



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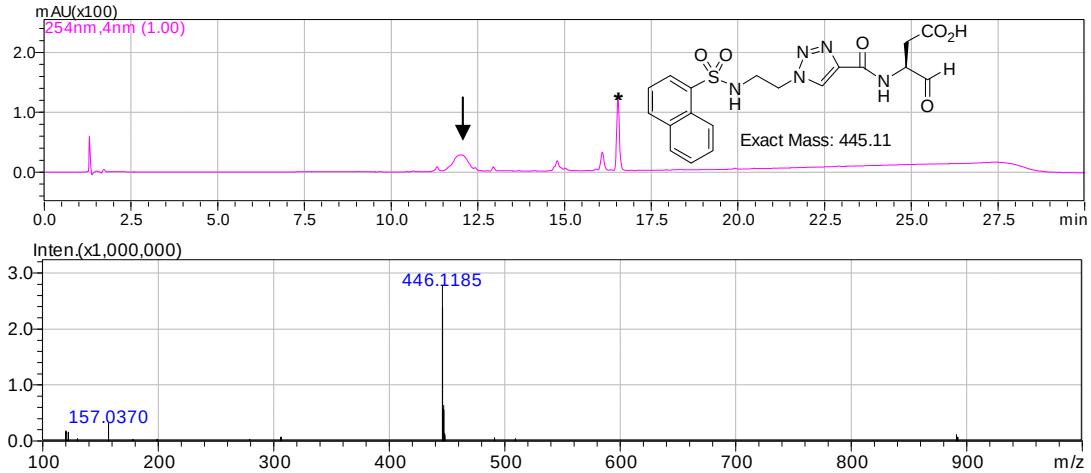
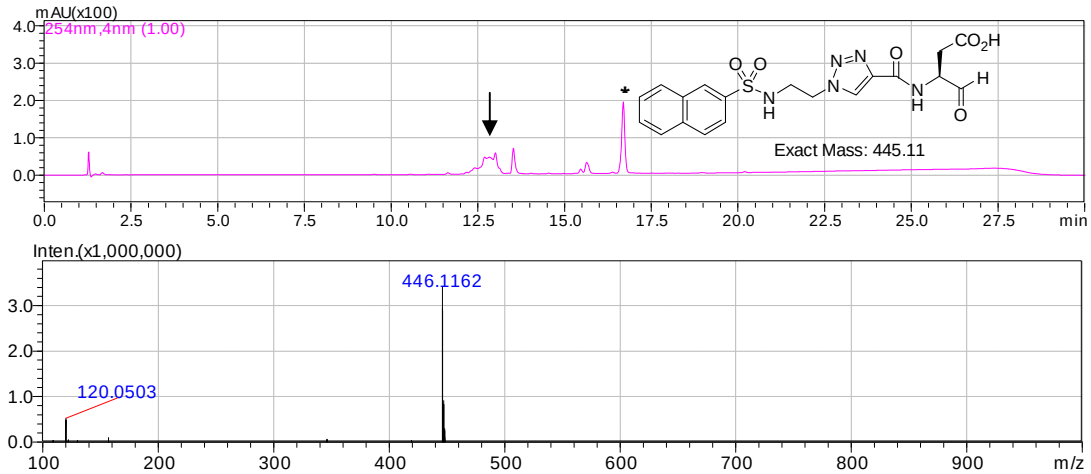


Ald-SB6:



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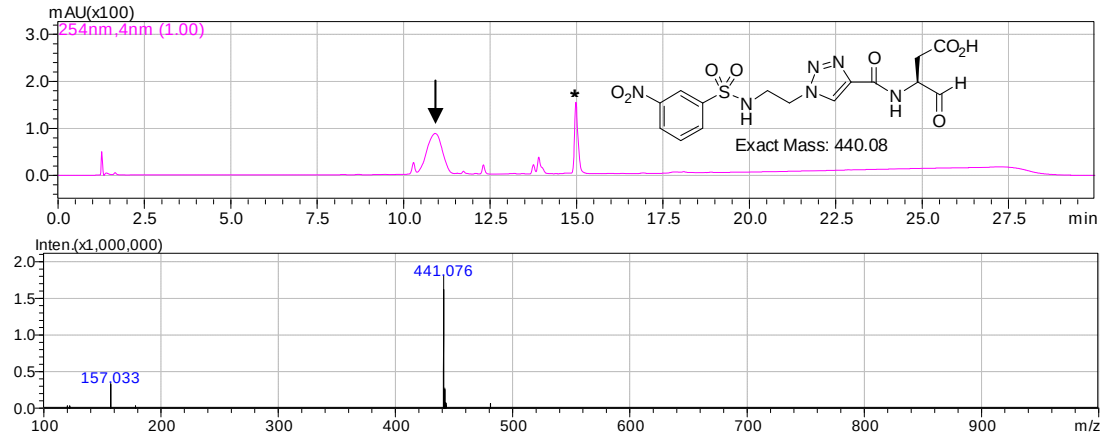

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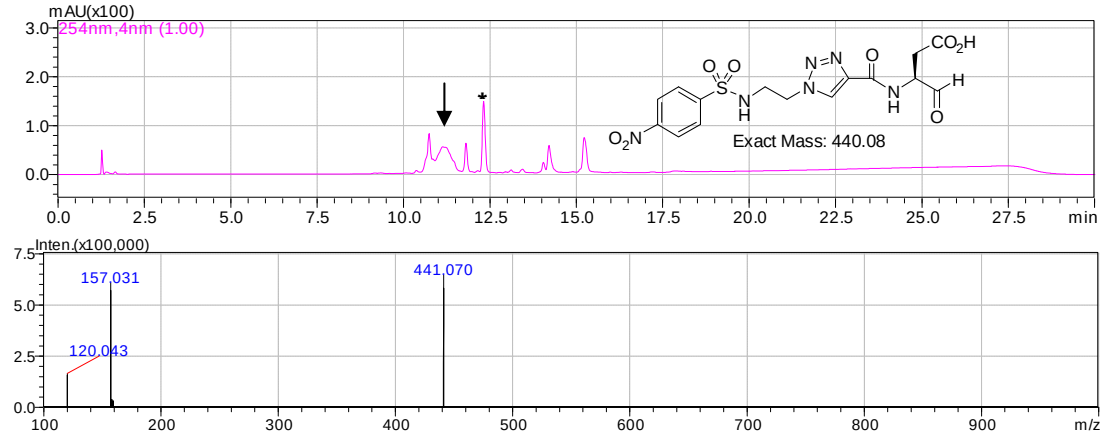
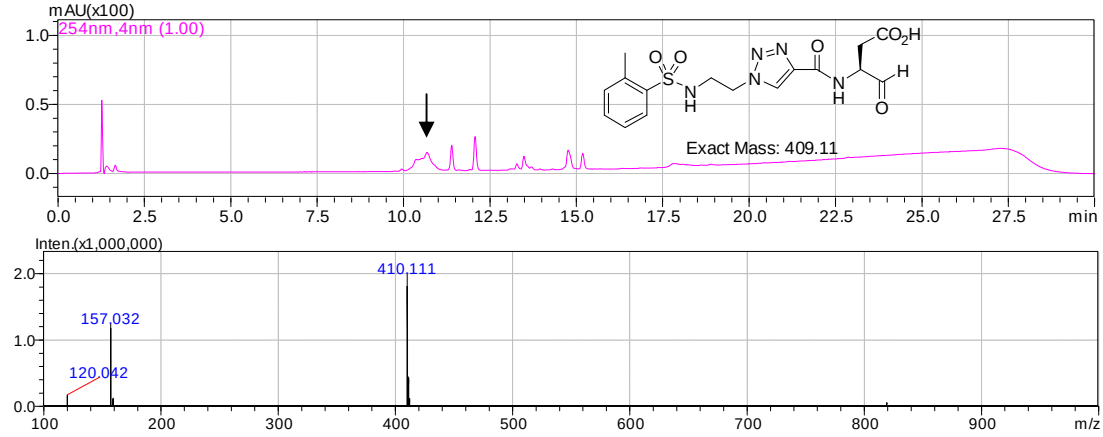
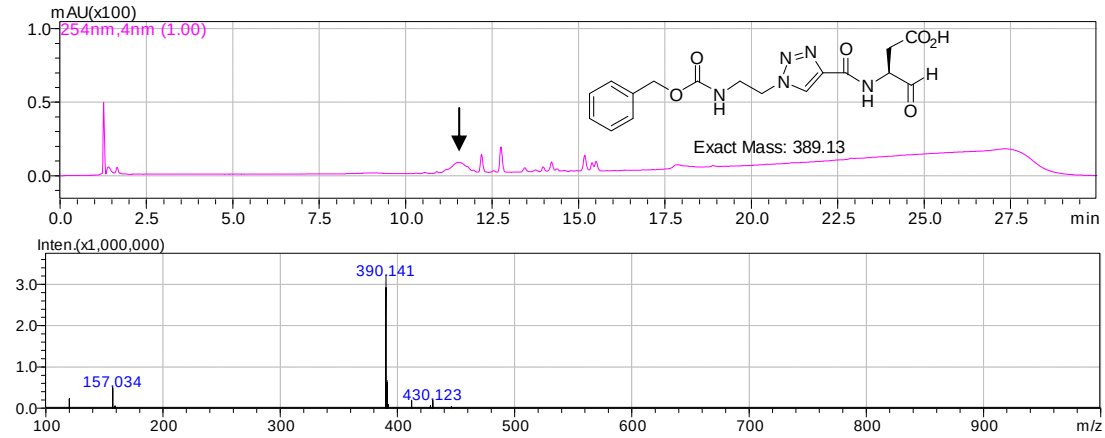


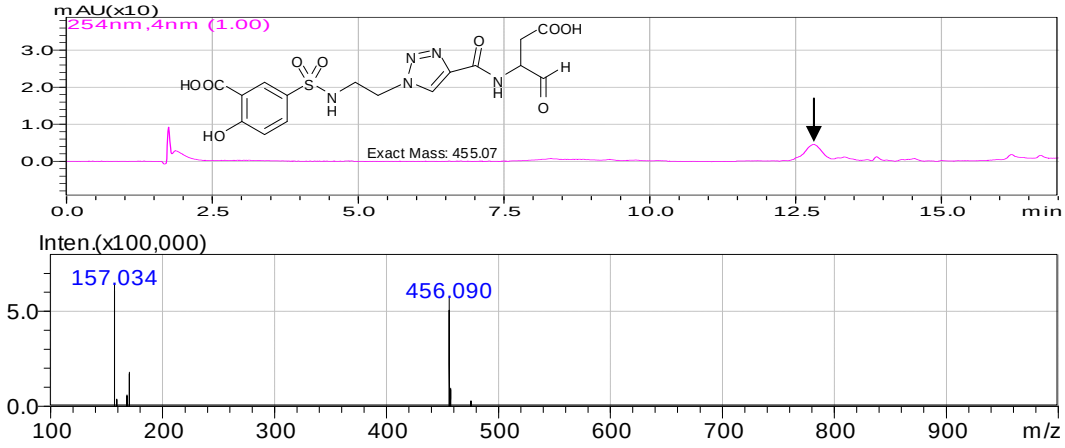
Ald-SB11:



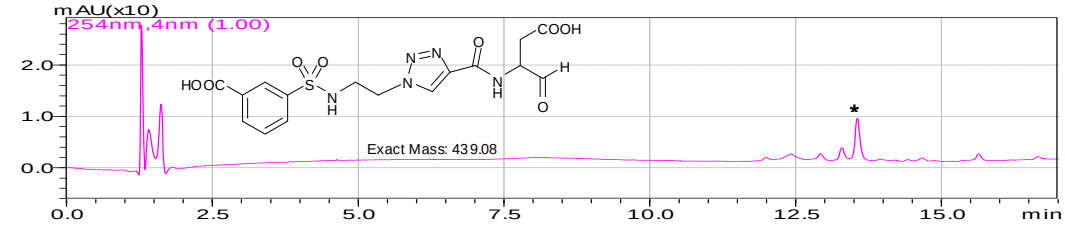
Ald-SC1:



Ald-SC2:

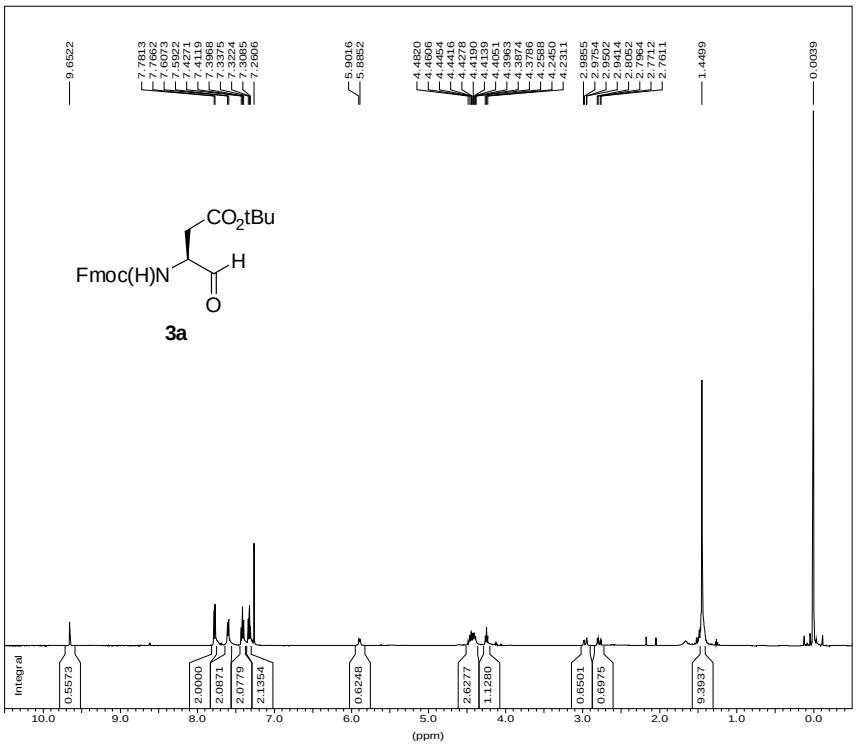


Ald-SC3:



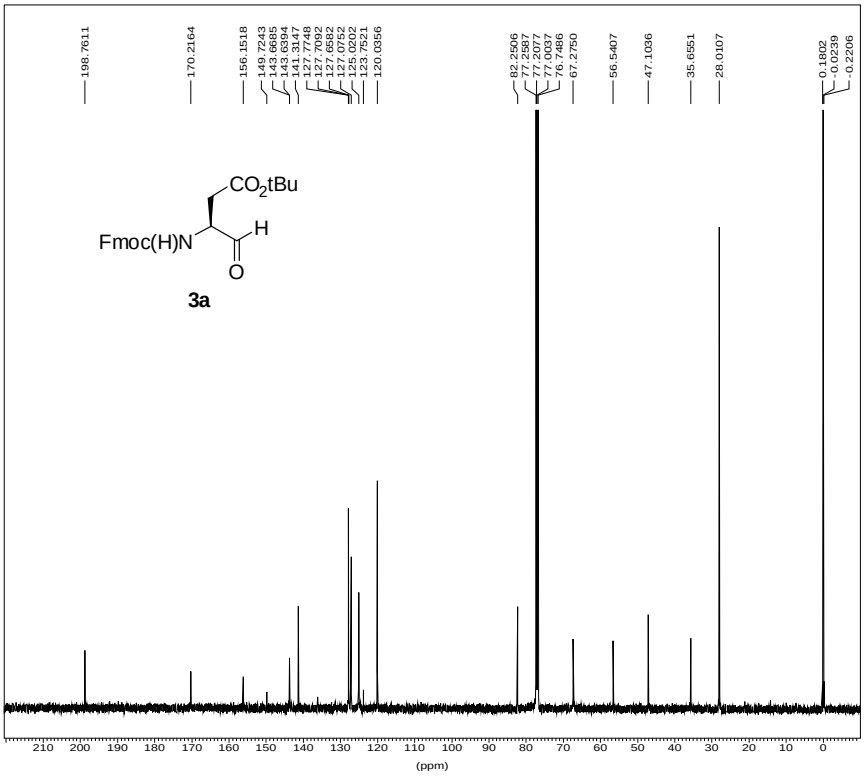
3. NMR Characterization of key intermediates

1H AMX500 Fmoc-Asp(OtBu)-CHO in CDCl3



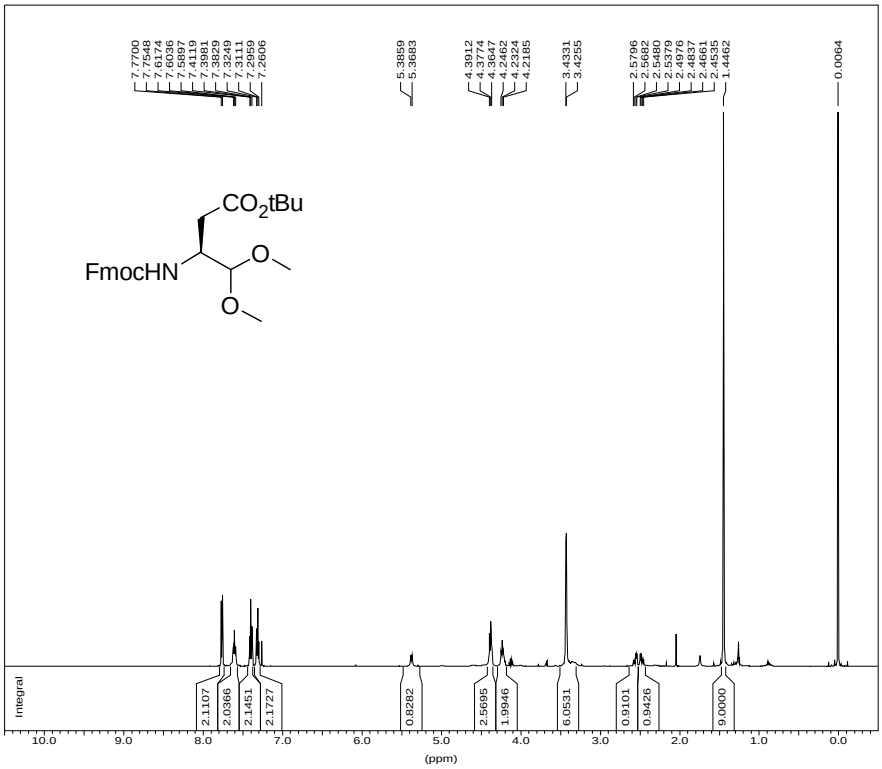
*** Current Data Parameters ***
NAME : py0718
EXPNO : 1
PROCNO : 1
*** Acquisition Parameters ***
NS : 32
PULPROG : zg30
RG : 143.6999969
SFO1 : 500.1330885 MHz
SOLVENT : CDCl3
SW : 20.6557 ppm
TD : 32768
TE : 295.1 K
*** Processing Parameters ***
LB : 0.30 Hz
WDW : EM

13C AMX500 Fmoc-Asp(OtBu)-CHO in CDCl3



*** Current Data Parameters ***
NAME : py0718
EXPNO : 2
PROCNO : 1
*** Acquisition Parameters ***
NS : 1606
PULPROG : zgpg30
RG : 16384.0000000
SFO1 : 125.7709936 MHz
SOLVENT : CDCl3
SW : 238.7675 ppm
TD : 65536
TE : 295.2 K
*** Processing Parameters ***
LB : 1.00 Hz
WDW : EM

1H AMX500 Fmoc-Asp(OtBu)-CH(OMe)2 in CDCl3

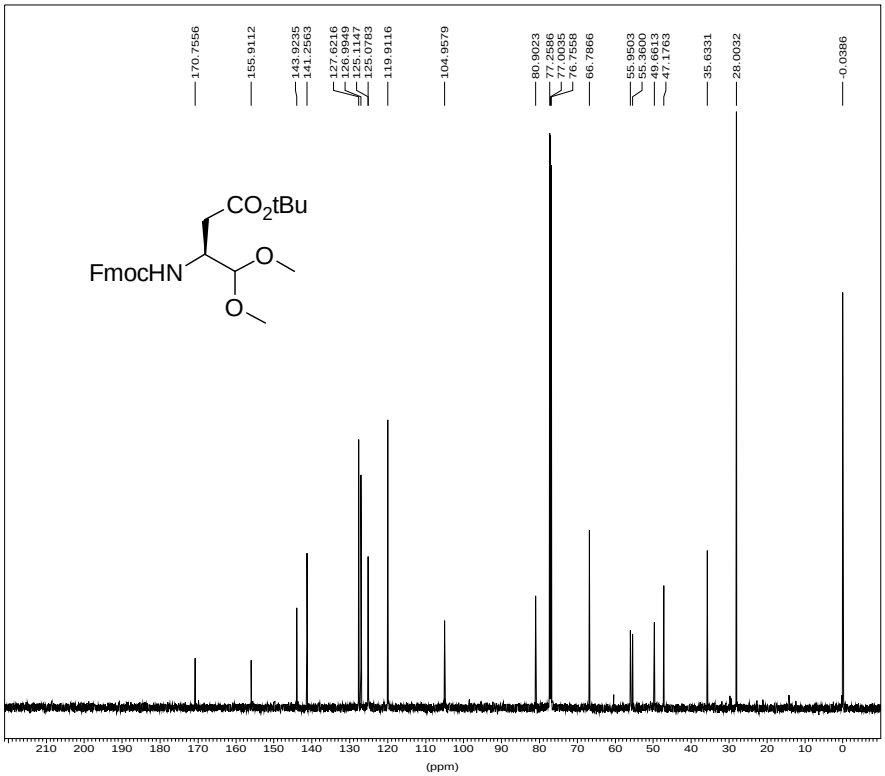


Bruker

*** Current Data Parameters ***

NAME	:	py0718
EXPNO	:	3
PROCNO	:	1
*** Acquisition Parameters ***		
NS	:	8
PULPROG	:	zg30
RG	:	64.0000000
SFO1	:	500.1330885 MHz
SOLVENT	:	CDCl3
SW	:	20.6557 ppm
TD	:	32768
TE	:	295.2 K
*** Processing Parameters ***		
LB	:	0.30 Hz
WDW	:	EM

13C AMX500 Fmoc-Asp(OtBu)-CH(OMe)2 in CDCl3

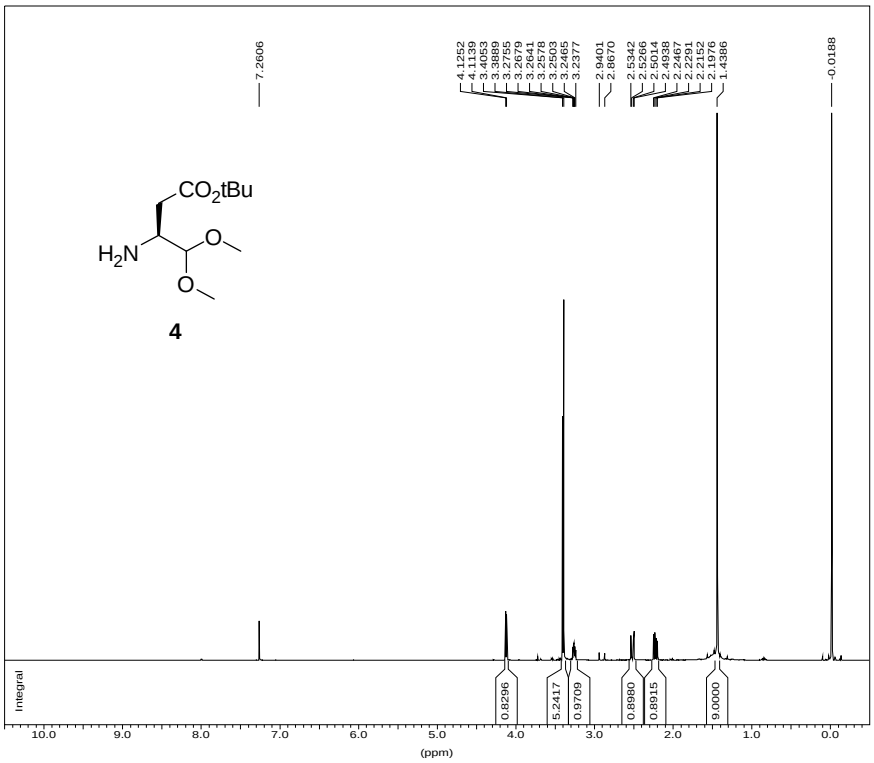


Bruker

*** Current Data Parameters ***

NAME	:	py0718
EXPNO	:	14
PROCNO	:	1
*** Acquisition Parameters ***		
NS	:	351
PULPROG	:	zgpg30
RG	:	16384.0000000
SFO1	:	125.7709936 MHz
SOLVENT	:	CDCl3
SW	:	238.7675 ppm
TD	:	65536
TE	:	295.3 K
*** Processing Parameters ***		
LB	:	1.00 Hz
WDW	:	EM

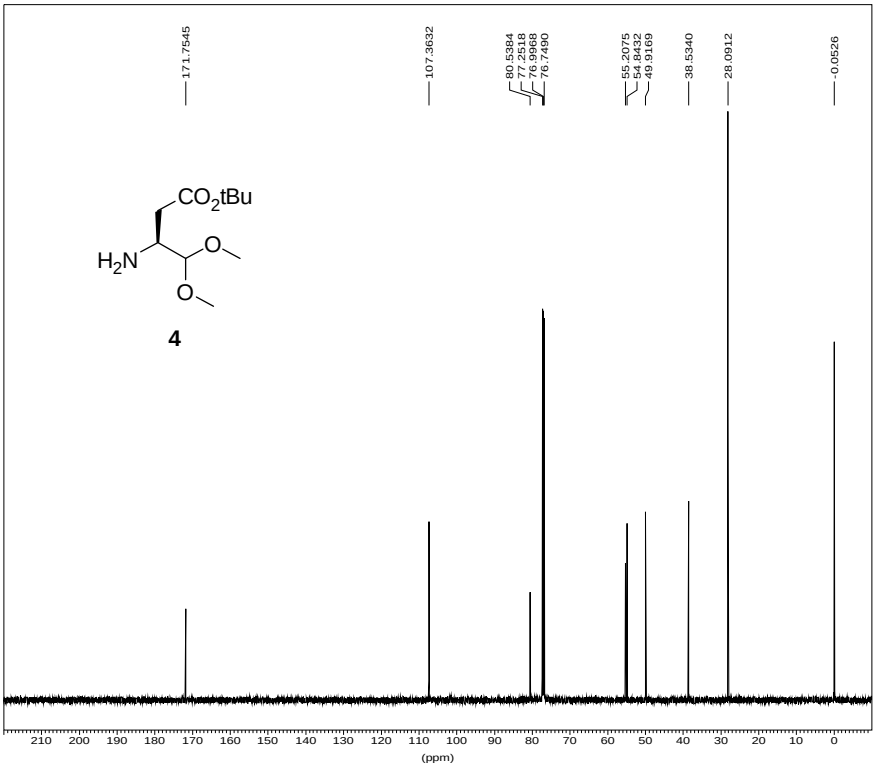
1H AMX500 warhead1 in CDCl3



*** Current Data Parameters ***

NAME	:	py0717
EXPNO	:	1
PROCNO	:	1
*** Acquisition Parameters ***		
NS	:	8
PULPROG	:	zg30
RG	:	80.5999985
SFO1	:	500.1330885 MHz
SOLVENT	:	CDCl3
SW	:	20.6557 ppm
TD	:	32768
TE	:	295.6 K
*** Processing Parameters ***		
LB	:	0.30 Hz
WDW	:	EM

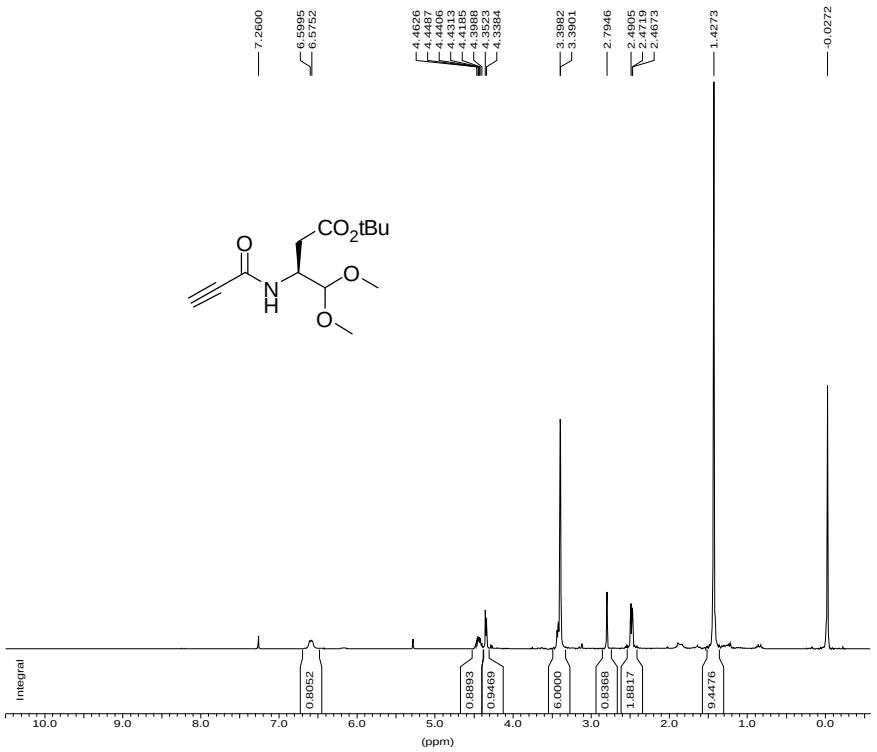
13C AMX500 warhead2 in CDCl3



*** Current Data Parameters ***

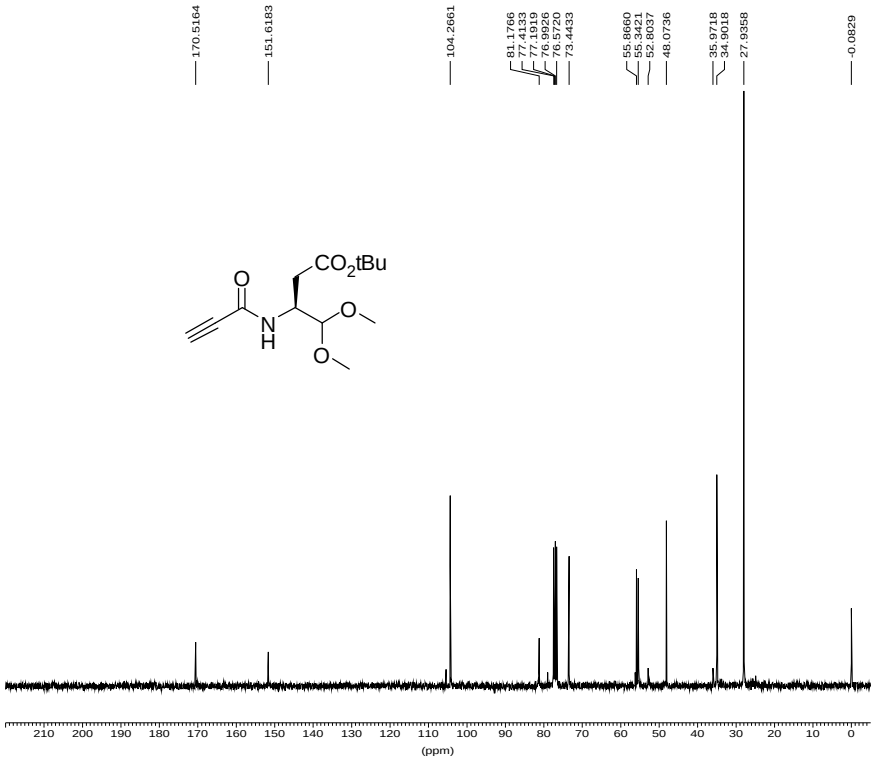
NAME	:	py0717
EXPNO	:	2
PROCNO	:	1
*** Acquisition Parameters ***		
NS	:	301
PULPROG	:	zgpg30
RG	:	16384.0000000
SFO1	:	125.7709936 MHz
SOLVENT	:	CDCl3
SW	:	238.7675 ppm
TD	:	65536
TE	:	295.8 K
*** Processing Parameters ***		
LB	:	1.00 Hz
WDW	:	EM

PY_02_158 AldWarhClicPrecusor in CDCl3

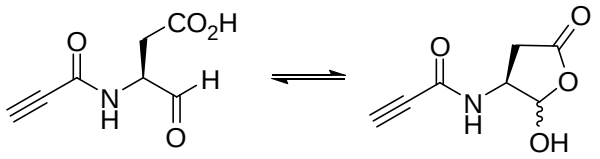


*** Current Data Parameters ***
NAME : ag23py
EXPNO : 1
PROCNO : 1
*** Acquisition Parameters ***
NS : 8
PULPROG : zg30
RG : 101.5999985
SFO1 : 300.1315000 MHz
SOLVENT : CDCl3
SW : 19.0175 ppm
TD : 16384
TE : 0.0 K
*** Processing Parameters ***
LB : 0.20 Hz
WDW : EM

PY_02_158 AldWarhClicPrecusor in CDCl3

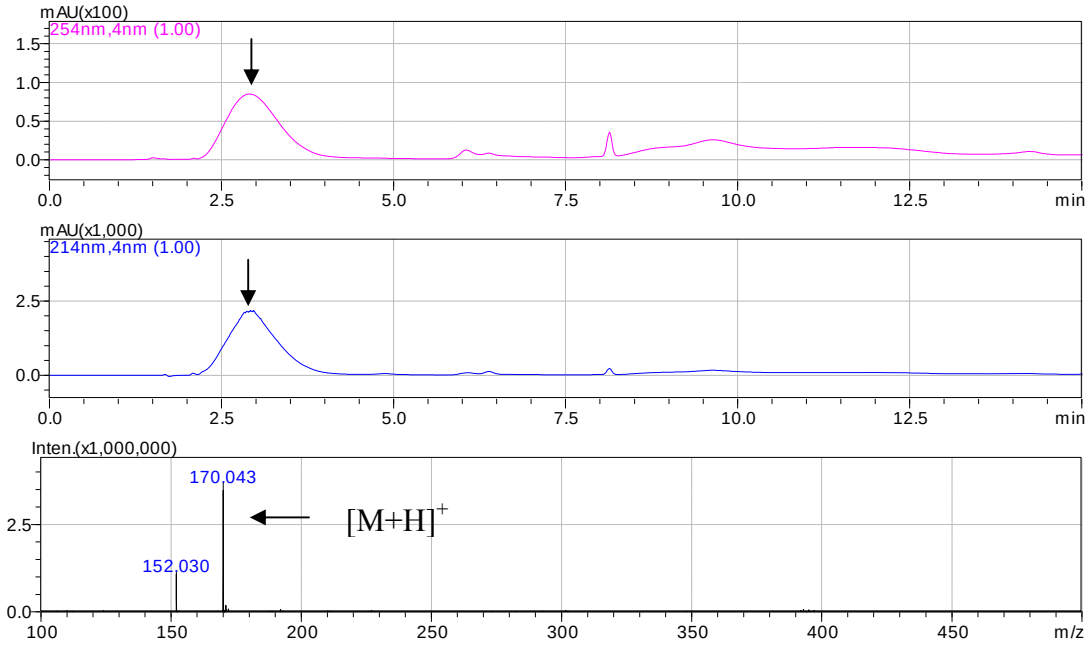


*** Current Data Parameters ***
NAME : ag23py
EXPNO : 2
PROCNO : 1
*** Acquisition Parameters ***
NS : 206
PULPROG : zgdc
RG : 16384.0000000
SFO1 : 75.4760840 MHz
SOLVENT : CDCl3
SW : 241.7743 ppm
TD : 32768
TE : 0.0 K
*** Processing Parameters ***
LB : 2.00 Hz
WDW : EM

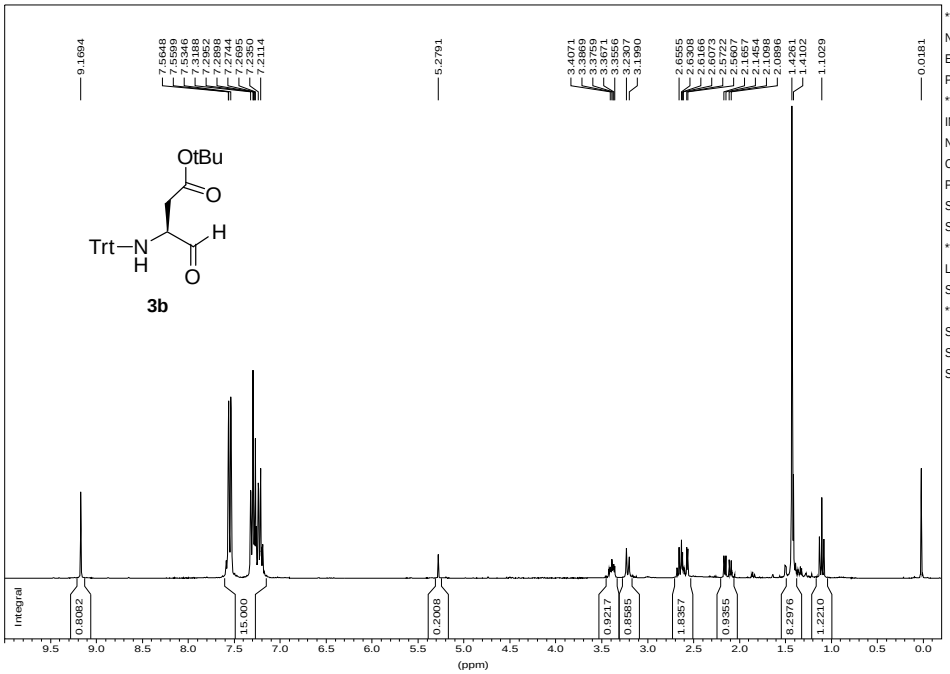


5

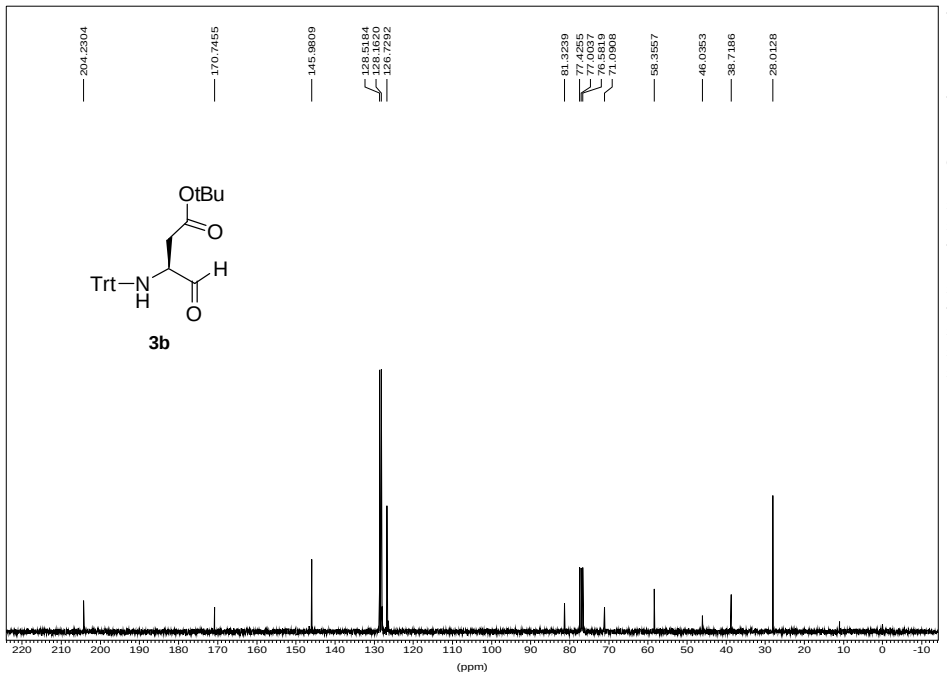
Exact Mass: 169.04



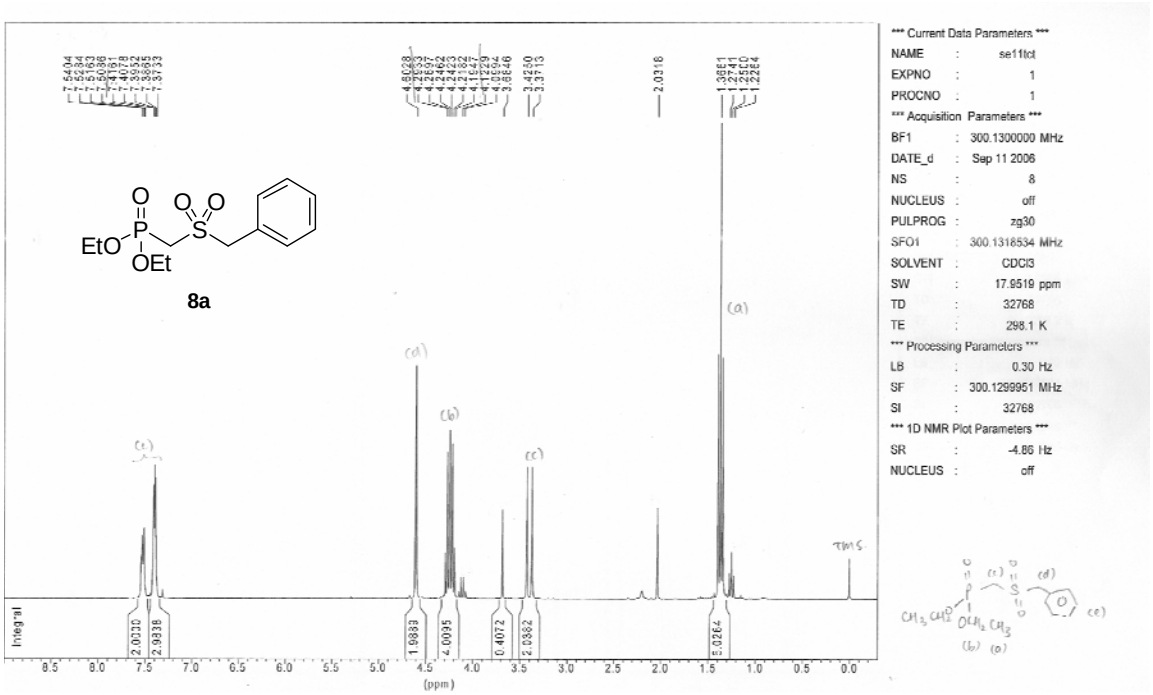
Compound **5** exists as an isomeric mixture (3 isomers). ^{1}H NMR cannot be properly interpreted. LCMS shows a broad peak corresponding to the correct molecular mass, TOF-MS: *m/z* 170.039 $[M + H]^{+}$.

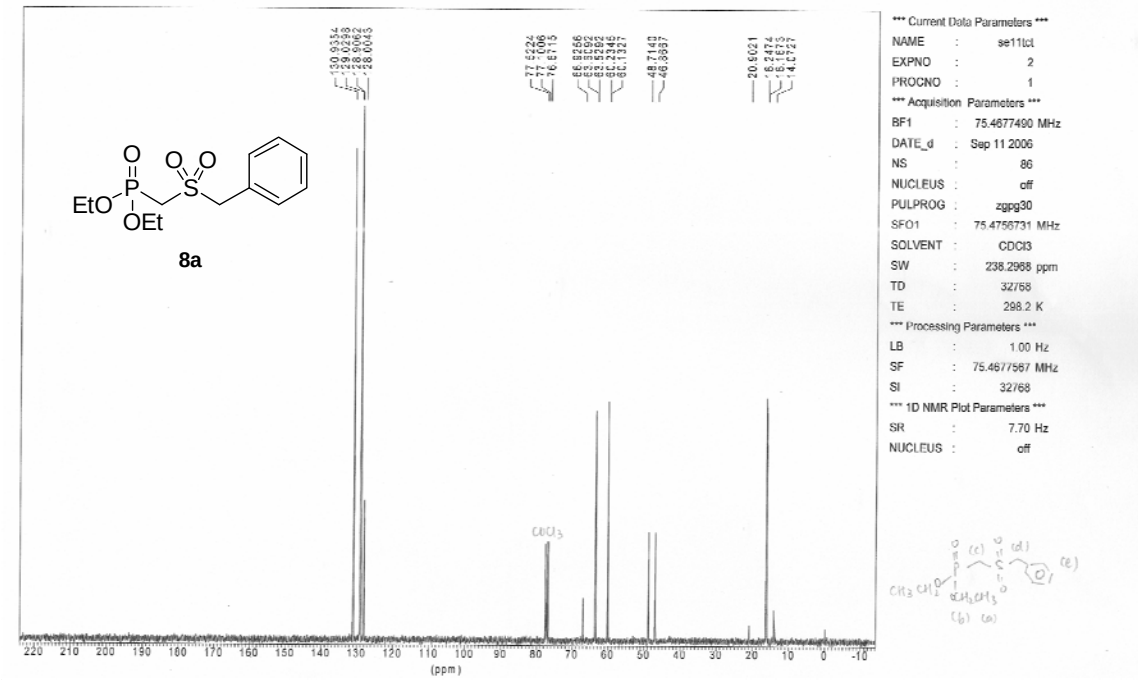


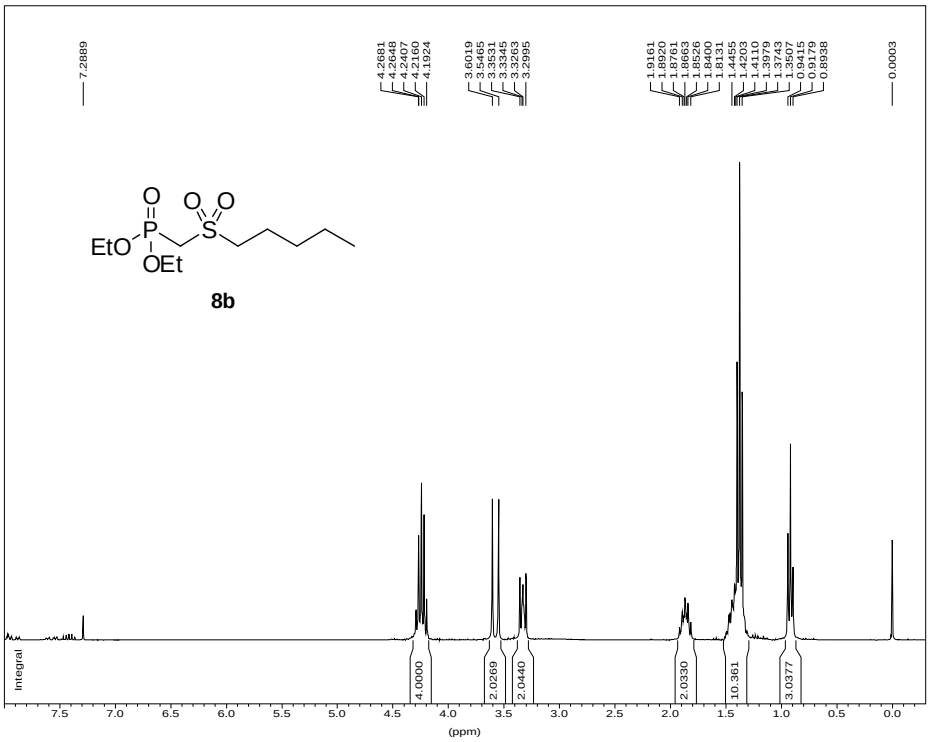
*** Current Data Parameters ***
NAME : de22my
EXPNO : 1
PROCNO : 1
*** Acquisition Parameters ***
INSTRUM : spect
NS : 8
O1 : 1853.43 Hz
PULPROG : zg30
SFO1 : 300.1318534 MHz
SW : 17.9519 ppm
*** Processing Parameters ***
LB : 0.30 Hz
SI : 32768
*** 1D NMR Plot Parameters ***
Start : 10.00 ppm
Stop : -0.20 ppm
SR : 11.83 Hz

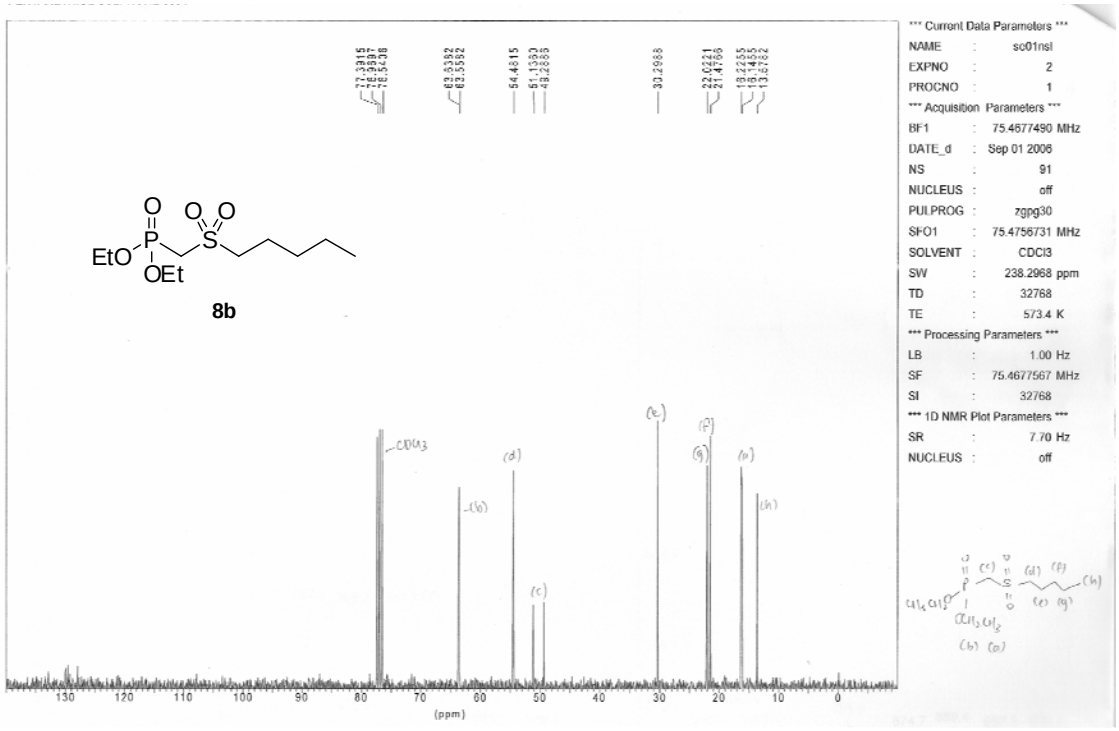


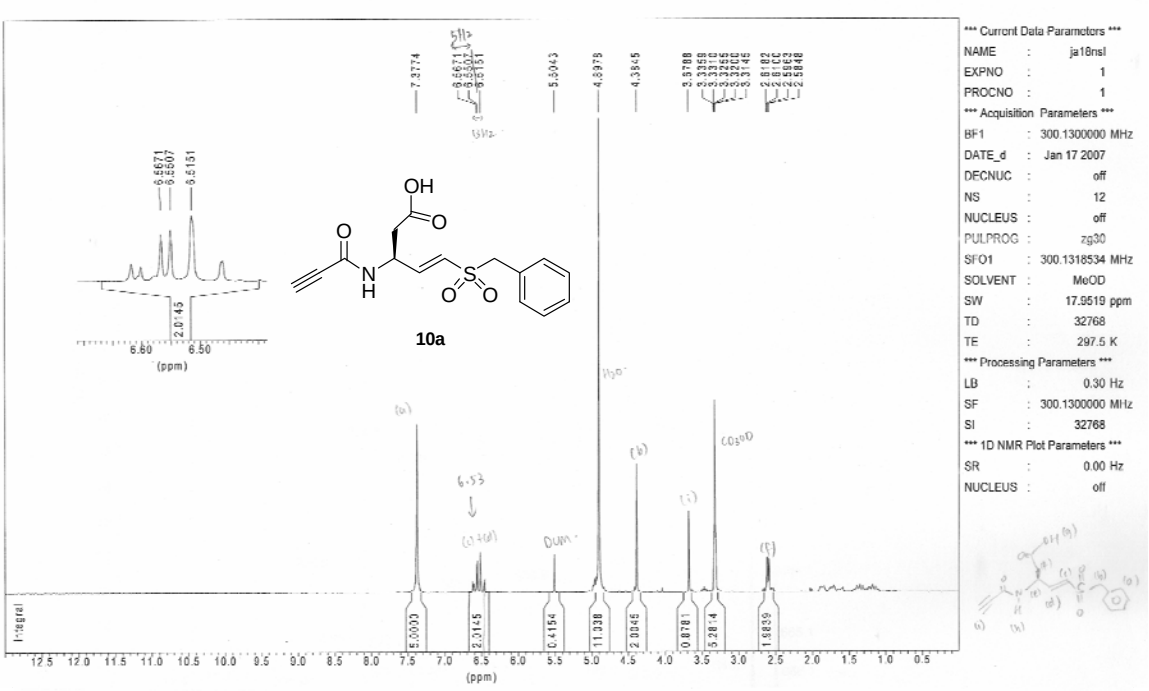
*** Current Data Parameters ***
NAME : de22my
EXPNO : 2
PROCNO : 1
*** Acquisition Parameters ***
INSTRUM : spect
NS : 67
O1 : 7924.11 Hz
PULPROG : zgpg30
SFO1 : 75.4756731 MHz
SW : 238.2968 ppm
*** Processing Parameters ***
LB : 1.00 Hz
SI : 32768
*** 1D NMR Plot Parameters ***
Start : 224.08 ppm
Stop : -14.24 ppm
SR : 6.23 Hz

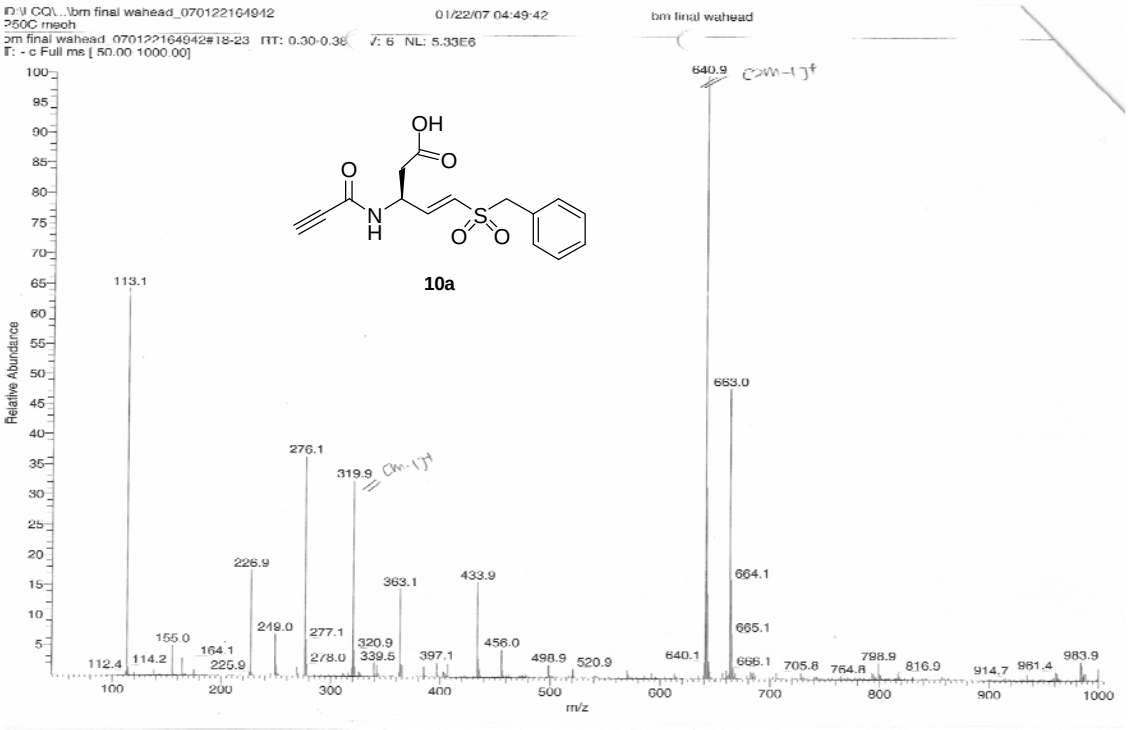

8a
*** Current Data Parameters ***
NAME : se11tct
EXPNO : 1
PROCNO : 1
*** Acquisition Parameters ***
BF1 : 300.1300000 MHz
DATE_d : Sep 11 2006
NS : 8
NUCLEUS : off
PULPROG : zg30
SFO1 : 300.1318534 MHz
SOLVENT : CDCl3
SW : 17.9519 ppm
TD : 32768
TE : 298.1 K
*** Processing Parameters ***
LB : 0.30 Hz
SF : 300.1299951 MHz
SI : 32768
*** 1D NMR Plot Parameters ***
SR : -4.86 Hz
NUCLEUS : off
Integral
(ppm)

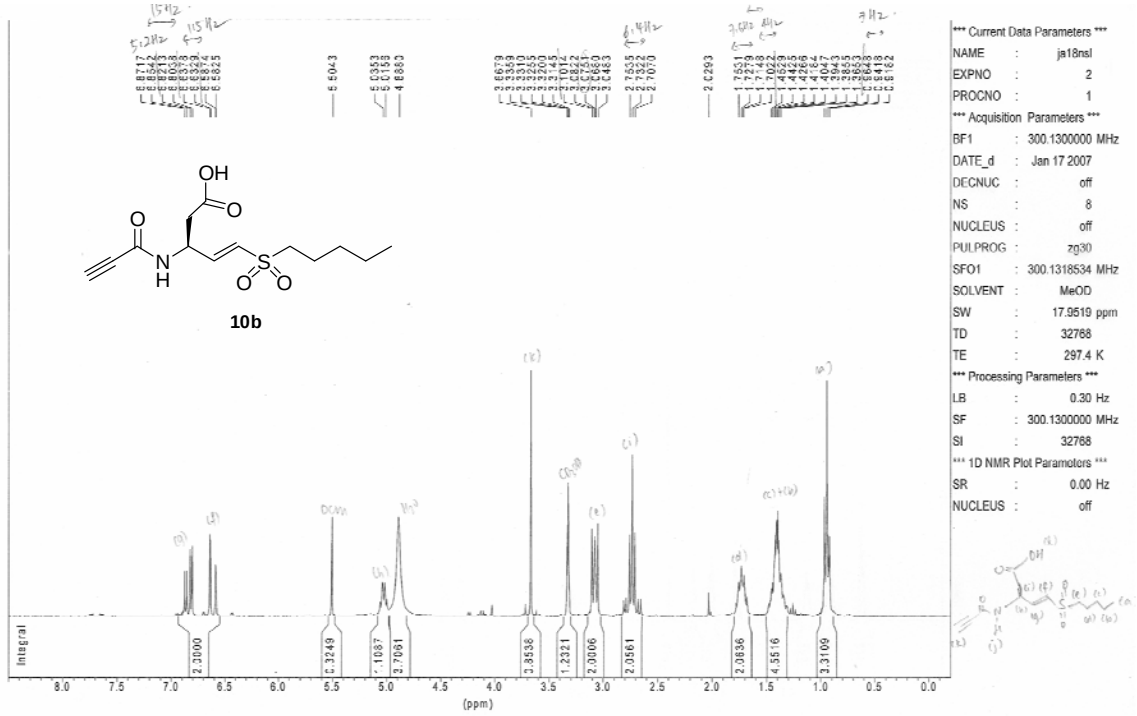

8a
*** Current Data Parameters ***
NAME : se11tct
EXPNO : 2
PROCNO : 1
*** Acquisition Parameters ***
BF1 : 75.4677490 MHz
DATE_d : Sep 11 2006
NS : 86
NUCLEUS : off
PULPROG : zgpg30
SFO1 : 75.4756731 MHz
SOLVENT : CDCl3
SW : 238.2968 ppm
TD : 32768
TE : 298.2 K
*** Processing Parameters ***
LB : 1.00 Hz
SF : 75.4677567 MHz
SI : 32768
*** 1D NMR Plot Parameters ***
SR : 7.70 Hz
NUCLEUS : off
CDCl3
(ppm)


7.2889
4.2681
4.2648
4.2407
4.2160
4.1924
3.6019
3.5465
3.3531
3.3345
3.3063
3.2995
1.9161
1.8920
1.8761
1.8663
1.8525
1.8400
1.8131
1.4455
1.4203
1.4110
1.3979
1.3743
1.3507
0.9415
0.9179
0.8938
0.0003
EtO
OEt
8b
Integral
4.0000
2.0269
2.0440
2.0330
10.361
3.0377
(ppm)
*** Current Data Parameters ***
NAME : se01nsl
EXPNO : 1
PROCNO : 1
*** Acquisition Parameters ***
BF1 : 300.1300000 MHz
DATE_d : Sep 01 2006
NS : 5
NUCLEUS : off
PULPROG : zg30
SFO1 : 300.1318534 MHz
SOLVENT : CDCl3
SW : 17.9519 ppm
TD : 32768
TE : 299.7 K
*** Processing Parameters ***
LB : 0.30 Hz
SF : 300.1300032 MHz
SI : 32768
*** 1D NMR Plot Parameters ***
SR : 3.21 Hz
NUCLEUS : off

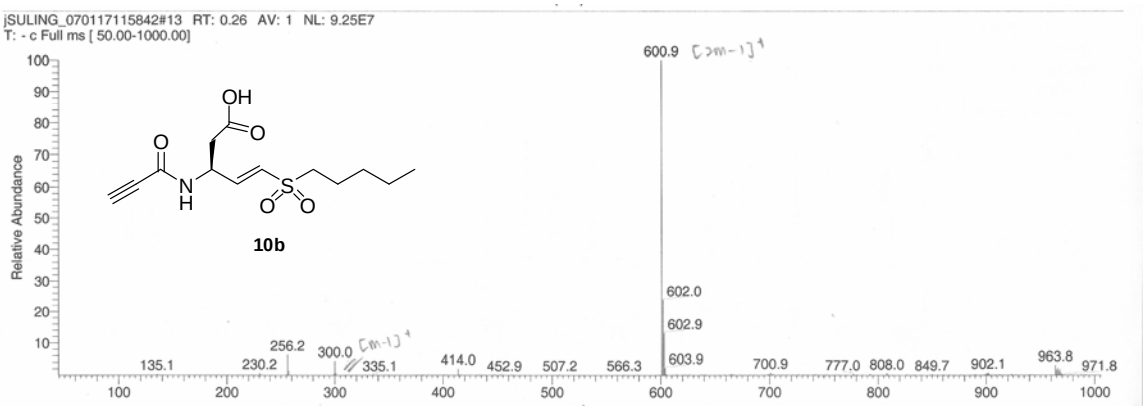

77.3815
77.0633
76.7451
76.5308
63.5392
63.5552
54.4615
51.1352
49.2883
30.2988
22.0221
21.4760
13.2255
13.1455
13.0722
EtO
OEt
8b
CDCl3
(b)
(d)
(c)
(e)
(f)
(g)
(a)
(h)
(ppm)
*** Current Data Parameters ***
NAME : sc01nsl
EXPNO : 2
PROCNO : 1
*** Acquisition Parameters ***
BF1 : 75.4677490 MHz
DATE_d : Sep 01 2006
NS : 91
NUCLEUS : off
PULPROG : zgpg30
SFO1 : 75.4756731 MHz
SOLVENT : CDCl3
SW : 238.2968 ppm
TD : 32768
TE : 573.4 K
*** Processing Parameters ***
LB : 1.00 Hz
SF : 75.4677567 MHz
SI : 32768
*** 1D NMR Plot Parameters ***
SR : 7.70 Hz
NUCLEUS : off


*** Current Data Parameters ***
NAME : ja18nsl
EXPNO : 1
PROCNO : 1
*** Acquisition Parameters ***
BF1 : 300.1300000 MHz
DATE_d : Jan 17 2007
DECNUC : off
NS : 12
NUCLEUS : off
PULPROG : zg30
SFO1 : 300.1318534 MHz
SOLVENT : MeOD
SW : 17.9519 ppm
TD : 32768
TE : 297.5 K
*** Processing Parameters ***
LB : 0.30 Hz
SF : 300.1300000 MHz
SI : 32768
*** 1D NMR Plot Parameters ***
SR : 0.00 Hz
NUCLEUS : off
10a
(ppm)
Integral


bm final wahead
10a
Relative Abundance
m/z

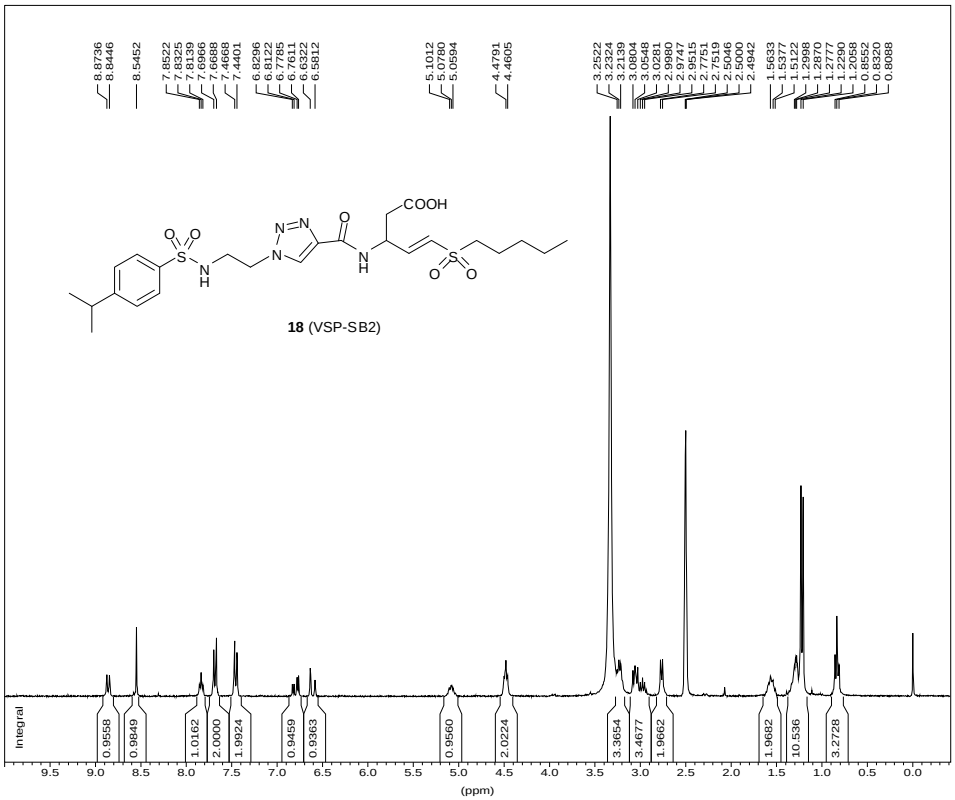

*** Current Data Parameters ***
NAME : ja18nsl
EXPNO : 2
PROCNO : 1
*** Acquisition Parameters ***
BF1 : 300.1300000 MHz
DATE_d : Jan 17 2007
DECNUC : off
NS : 8
NUCLEUS : off
PULPROG : zg30
SFO1 : 300.1318534 MHz
SOLVENT : MeOD
SW : 17.9519 ppm
TD : 32768
TE : 297.4 K
*** Processing Parameters ***
LB : 0.30 Hz
SF : 300.1300000 MHz
SI : 32768
*** 1D NMR Plot Parameters ***
SR : 0.00 Hz
NUCLEUS : off
Integral
(ppm)

10b


jSULING_070117115842#13 RT: 0.26 AV: 1 NL: 9.25E7
T: - c Full ms [50.00-1000.00]
Relative Abundance
600.9
602.0
602.9
603.9

10b

14-1



*** Current Data Parameters ***

NAME	:	ag20shb
EXPNO	:	1
PROCNO	:	1
*** Acquisition Parameters ***		
DATE_t	:	21:48:43
DATE_d	:	Aug 19 2007
NS	:	8
SFO1	:	300.1315000 MHz
SOLVENT	:	DMSO
*** Processing Parameters ***		
AQORDER	:	0
*** 1D NMR Plot Parameters ***		
Start	:	10.00 ppm
Stop	:	-0.42 ppm