

## LCMS Analysis Report

Acquired by : System Administrator  
 Sample Name : LCMS29-PH-FMA-PJ00194-17-0-1(59340-083F1)1T003  
 Injection Volume : 1  
 Data File : LCMS29-PH-FMA-PJ00194-17-0-1-C.lcd  
 Method File : ACN-Water-0.05%TFA-5%B-1.5-3.0MIN(90-900).lcm  
 Report Format File : LCMS2020-ELSD-PDA-TIC-MS.lsr  
 Date Acquired : 2016-11-10 11:10:03  
 Comment : Mobile phase A:Water/0.05%TFA  
           : Mobile phase B:Acetonitrile/0.05%TFA

Instrument Name:Shimadzu LCMS-2020

<<Pump>>

Mode : Binary gradient  
 Pump A : LC-30AD  
 Pump B : LC-30AD  
 Total Flow : 1.5000 mL/min  
 B Conc. : 5.0 %

<<Interface>>

Interface :ESI  
 DL Temperature :300 C  
 Nebulizing Gas Flow :1.50 L/min  
 Heat Block :300 C  
 Drying Gas :On  
                   :15.00 L/min

<<Oven>>

Oven Temperature : 45 C

<<MS Parameter>>

--Segment 1 Event 1--  
 Start Time :0.00 min  
 End Time :3.00 min  
 Acquisition Mode :Scan  
 Polarity :Positive  
 Event Time :0.50 sec  
 Detector Voltage :+1.10 kV  
 Threshold :20  
 Start m/z :90.00  
 End m/z :900.00  
 Scan Speed :1667 u/sec  
 Interface Volt. :Use the Data in the Tuning File  
 DL Volt. :Use the Data in the Tuning File  
 Qarray DC Voltage :Use the Data in the Tuning File  
 Qarray DC Voltage :Use the Data in the Tuning File

System Configuration

<<Column>>

Column Name : Kinetex 2.6u XB-C18  
 Length : 50 mm  
 Internal Diameter : 3.0 mm  
 Description : 2.6 µm

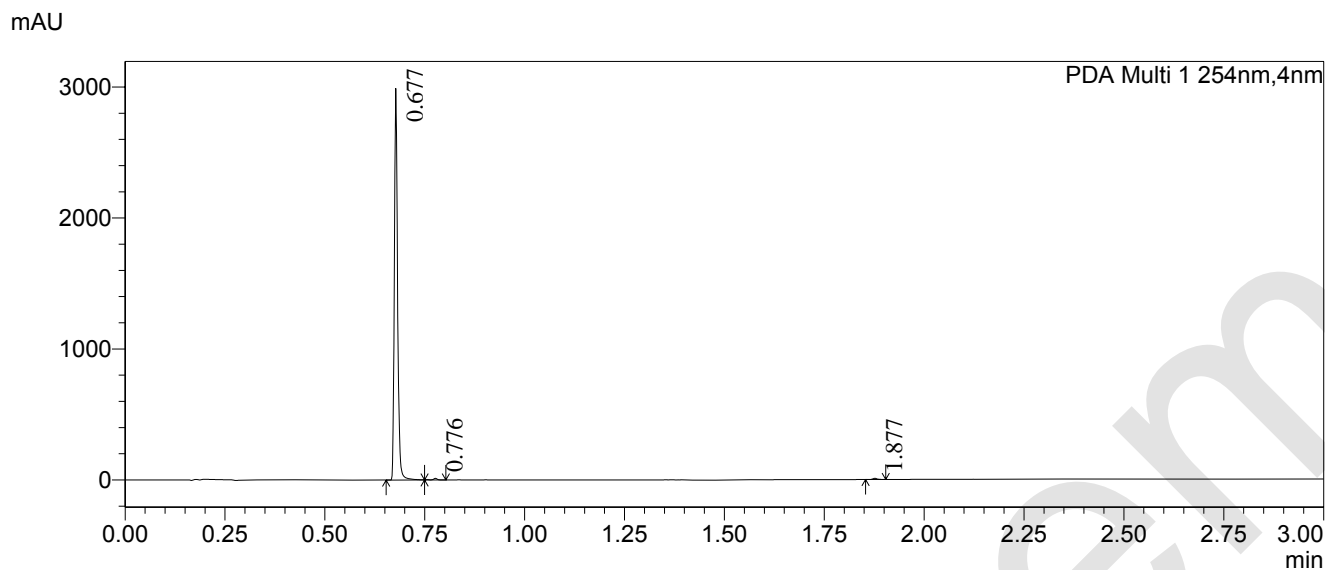
<<LC Time Program>>

Time	Module	Command	Value
0.01	Pumps	Pump B Conc.	5
2.00	Pumps	Pump B Conc.	100
2.80	Pumps	Pump B Conc.	100
2.90	Pumps	Pump B Conc.	5
3.00	Controller	Stop	

# HPLC Analysis for Raltitrexed



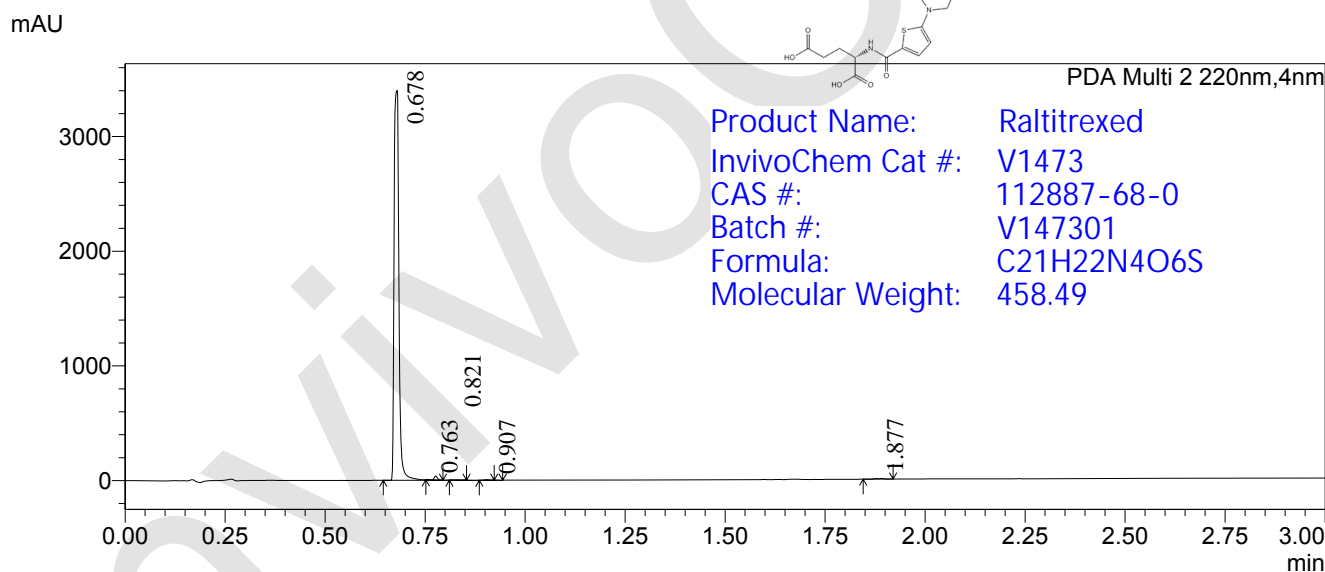
## <Chromatogram>



Peak Table

PDA Ch1 254nm

Peak#	Ret. Time	Height	Height%	Area	Area%
1	0.677	2814925	99.246	1681520	99.034
2	0.776	12434	0.438	9908	0.584
3	1.877	8951	0.316	6501	0.383
Total		2836310	100.000	1697929	100.000



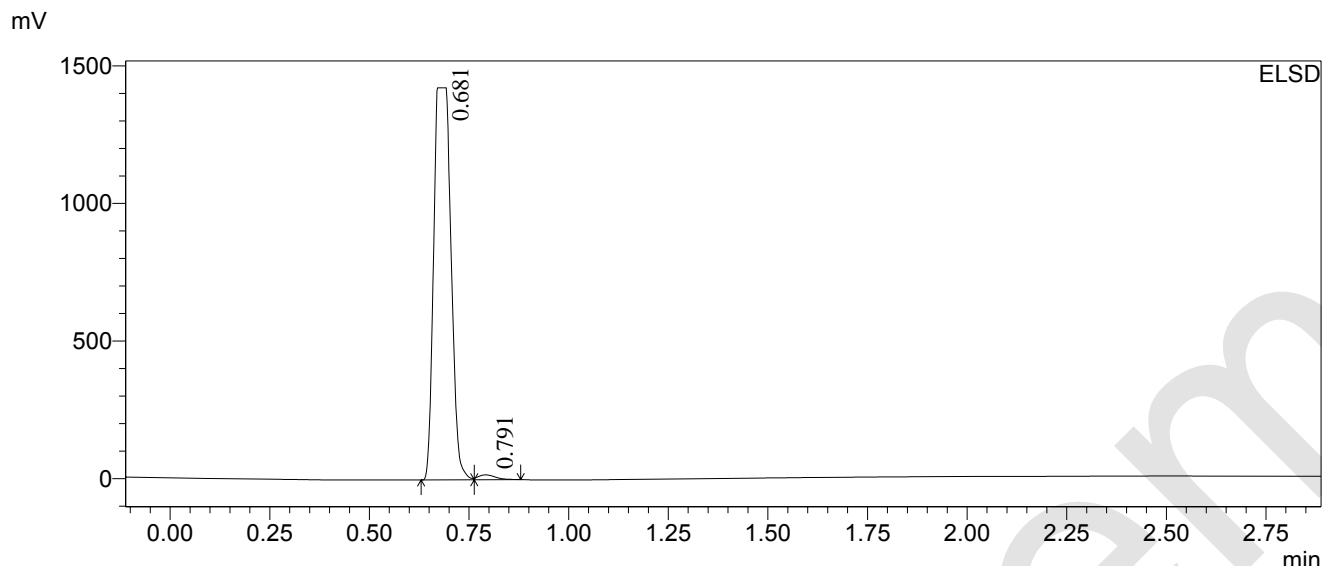
Product Name: Raltitrexed  
 InvivoChem Cat #: V1473  
 CAS #: 112887-68-0  
 Batch #: V147301  
 Formula: C<sub>21</sub>H<sub>22</sub>N<sub>4</sub>O<sub>6</sub>S  
 Molecular Weight: 458.49

Peak Table

PDA Ch2 220nm

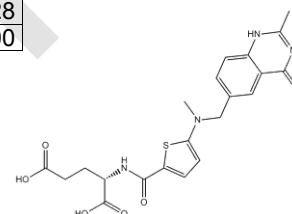
Peak#	Ret. Time	Height	Height%	Area	Area%
1	0.678	3212844	99.573	2854191	98.798
2	0.763	1859	0.058	20258	0.701
3	0.821	3180	0.099	3608	0.125
4	0.907	4384	0.136	3193	0.111
5	1.877	4367	0.135	7663	0.265
Total		3226633	100.000	2888913	100.000

# LC-MS Analysis for Raltitrexed



Peak Table

Peak#	Ret. Time	Height	Height%	Area	Area%
1	0.681	1425137	98.782	4288860	98.772
2	0.791	17570	1.218	53341	1.228
Total		1442707	100.000	4342201	100.000



Mass Spectrum

Line#:1 R.Time:0.681(Scan#:87)  
 MassPeaks:391  
 Spectrum Mode:Averaged 0.672-0.689(86-88) BasePeak:459.2(1951190)  
 BG Mode:Calc Segment 1 - Event 1

Product Name: Raltitrexed  
 InvivoChem Cat #: V1473  
 CAS #: 112887-68-0  
 Batch #: V147301  
 Formula: C<sub>21</sub>H<sub>22</sub>N<sub>4</sub>O<sub>6</sub>S  
 Molecular Weight: 458.49  
 Exact Mass: 458.1260

