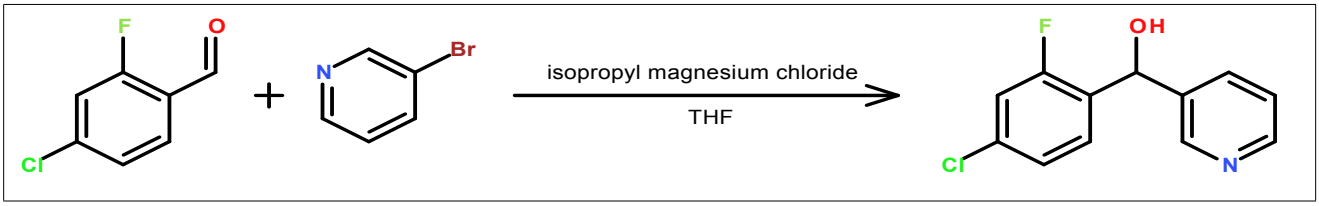
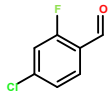
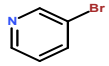
Reaction Details:

Temperature	Time	Pressure	Main Yield	Succeed	Recommendation	Lab Room
4-15 °C	5 hr		67.04%	Succeeded	★★★	16-415
Compound ID	WuXi ID	Compound Novelty	Compound Color	Compound state	Stability	Final Step
N/A	N/A	Unknown Compound	White	Solid		X

Scheme:



Reactants:

Sample ID	Structure	Salt	MW	Eq	Amount	Moles	Source	[Stock]	Vol	Purity	%ee
EW618-1841-R1			158.56	1	80 g	504.55 mmol	Domestic				
EW618-1841-R2			158.00	1.13	90 g	569.64 mmol	Domestic		54.88 mL		

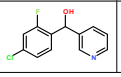
Reagents:

Reagent	MW	Eq	Amount	Moles	Source	[Stock]	Vol	Cat.	Purity
isopropyl magnesium chloride	102.85	1.19	61.51 g	598.12 mmol		2 M	299.06 mL		

Solvents:

Solvent	Vol	Source	Notes
THF	800 mL		
THF	300 mL		

Products:

Sample ID	Structure	MW	Amount	Moles	Theory Amount	Purity	%ee	Yld	Batches in parallel	Products state	Vol
EW618-1841-P1		237.66	81.2 g	338.25 mmol	119.91 g	99%		67.04%			

Reference:

Journal of Abbr.	Year	Volume	Issue	Page Range	Notes
N/A					

Procedure:

[Reaction Setup]

To a solution of 3-bromopyridine (90 g, 569.64 mmol, 54.88 mL, 1.13 *eq*) in THF (800 mL) was added isopropyl magnesium chloride (2 M, 299.06 mL, 1.19 *eq*) dropwise at 4 °C. After stirred 2 h at 15°C(room temperature). A solution of 4-chloro-2-fluoro-benzaldehyde (80 g, 504.55 mmol, 1 *eq*) in THF (300 mL) was added at 4°C. The mixture was stirred at 15°C(room temperature) for 3 h.

[Monitoring]

TLC(PE/EA=1/1) indicated no Reactant 1 was remained, and one major new spot was detected.

[Work-up]

The reaction mixture was poured into sat. NH4Cl(1 L) and extracted with EA(800 mL*2). The combined organic layers were washed with brine(1 L), dried over Na2SO4, filtered and concentrated under reduced pressure to give a residue.

[Purification]

The residue was triturated with the mixed solvent(PE/EA=10/1, 200 mL) at room temperature for 30 min, filtered and the cake

was dried under reduced pressure.

[Result]

Compound (4-chloro-2-fluoro-phenyl)-(3-pyridyl)methanol (81.2 g, 338.25 mmol, 67.04% yield, 99% purity) (NMR: EW618-1841-P1A, HPLC:EW618-1841-P1H, LCMS:EW618-1841-P1L)was obtained as a white solid.

TLC:

Plate NO	Eluent	Ratio	Color Developing Reagent	Others
Plate 1	Petroleum ether : Ethyl acetate	1:1	紫外(UV 254 nm)	

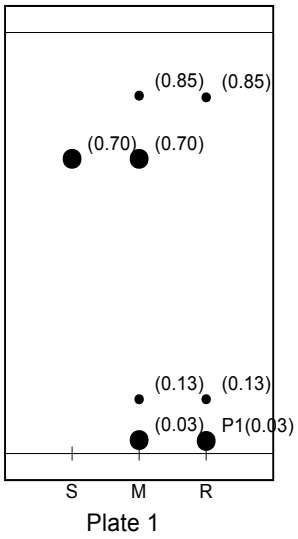


Plate 1

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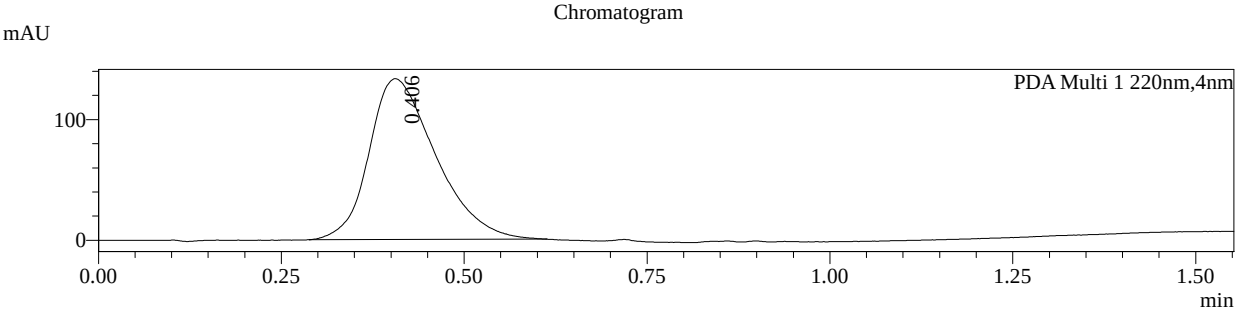
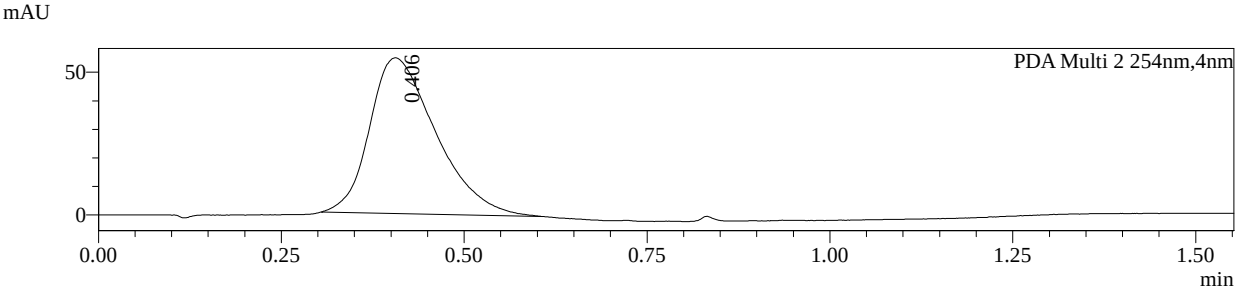
LCMS REPORT

Compound ID : EW618-1841-P1A
Sample ID : EW618-1841-P1A
Injection Vol : 1ul
Location : vial42
Acq Method : d:\method\5-95AB_R_220&254.lcm
Org DataFile : D:\DATA\1903\190315\EW618-1841-P1A.lcd
Injection Date : 2019-03-15 11:11:42
Instrument : LCMS-Q 17-102

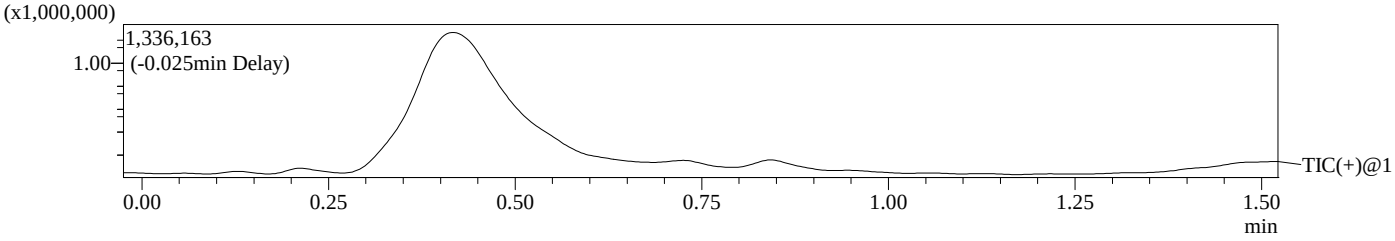
Chromatogram





1 PDA Multi 1 / 220nm,4nm
2 PDA Multi 2 / 254nm,4nm

MS Chromatogram



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Integration Result

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Peak Table

PDA Ch1 220nm

Peak#	Ret. Time	Height	Height%	USP Width	Area	Area%
1	0.406	133294	100.000	0.164	844153	100.000

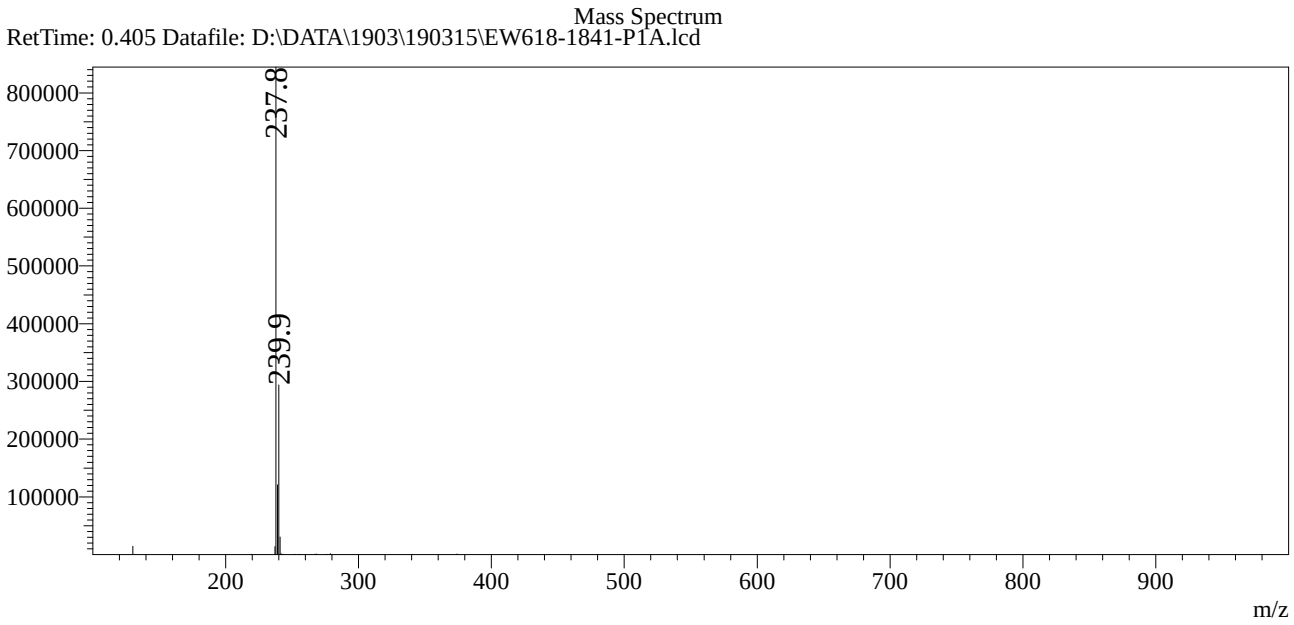
Peak Table

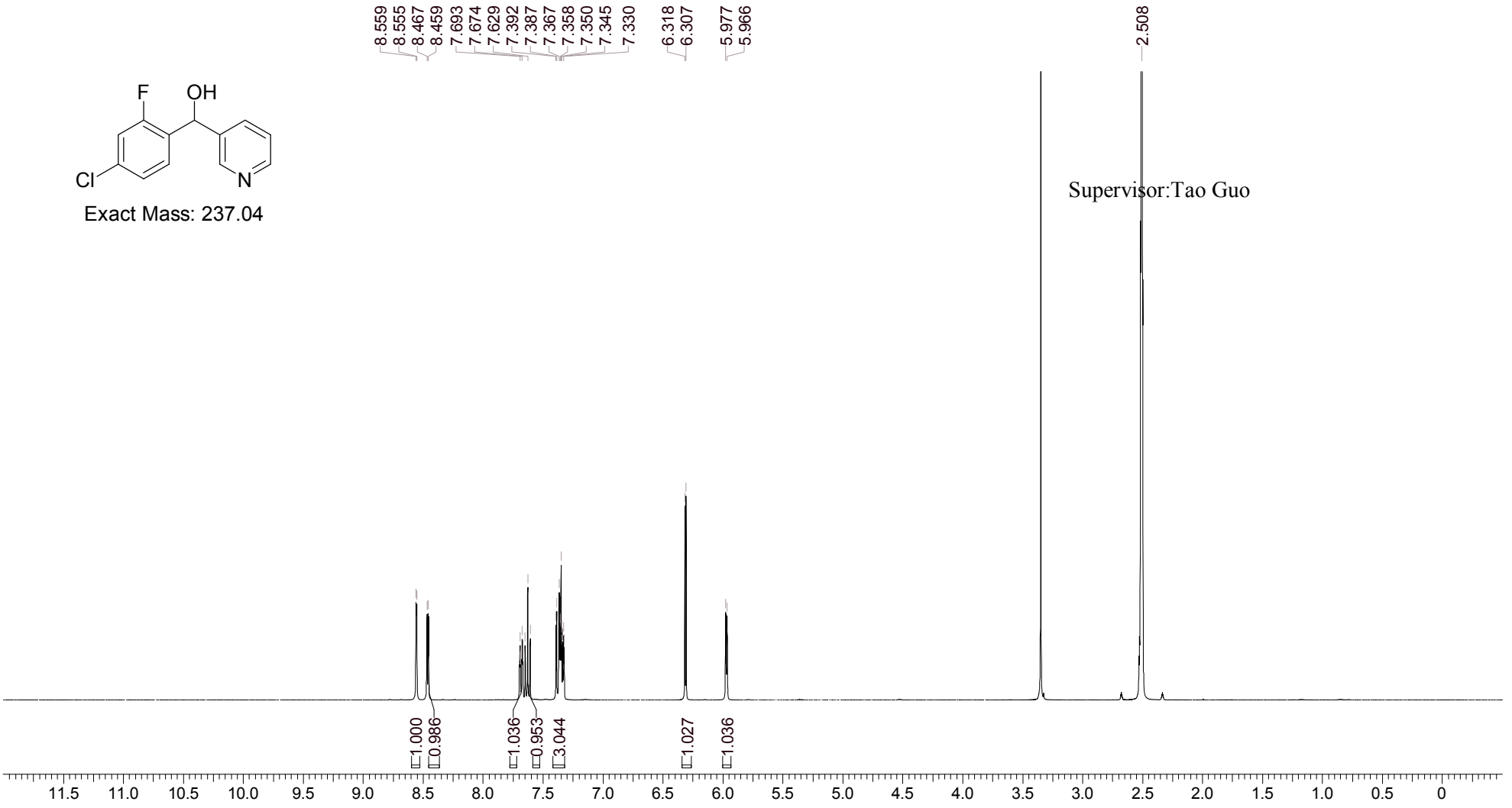
PDA Ch2 254nm

Peak#	Ret. Time	Height	Height%	USP Width	Area	Area%
1	0.406	54507	100.000	0.164	343138	100.000

Operator:_____

Date:_____


Mass Spectrum
RetTime: 0.405 Datafile: D:\DATA\1903\190315\EW618-1841-P1A.lcd
237.8
239.9
800000
700000
600000
500000
400000
300000
200000
100000
200
300
400
500
600
700
800
900
m/z



Operator: Date:

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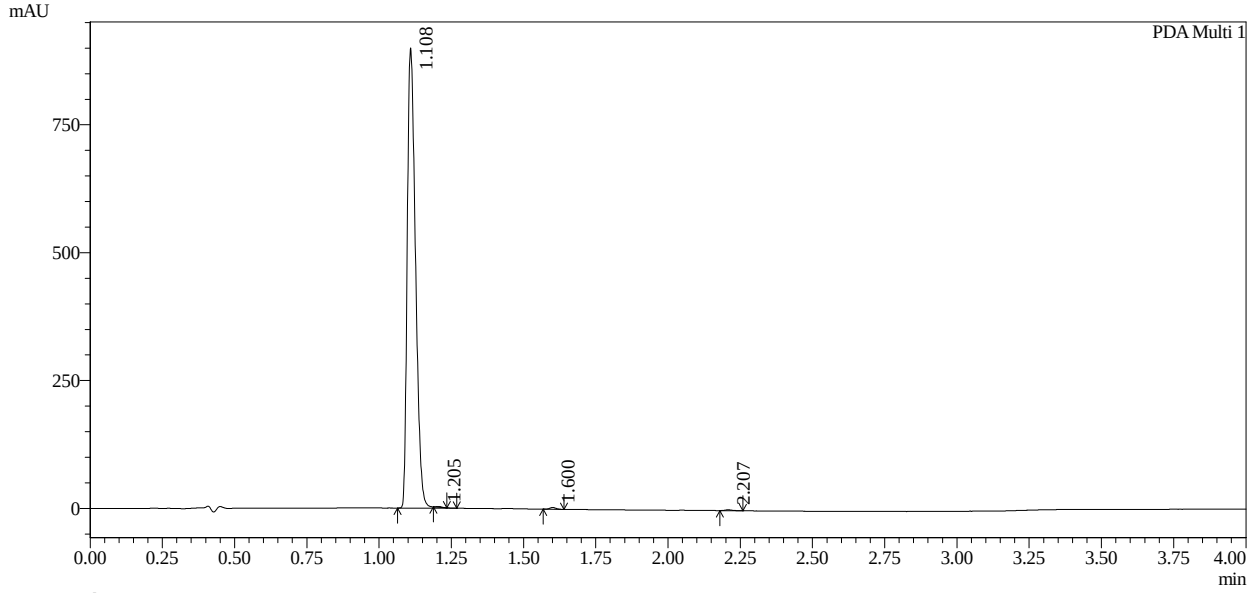
HPLC REPORT

Compound ID	: EW618-1841-P1A
Sample ID	: EW618-1841-P1H
Vial#	: 64
Injection Volumn	: 1
Filename	: D:\DATA\2019\1903\190315\EW618-1841-P1H.lcd
Method Name	: D:\Method\10-80AB_4min.lcm
Instrument	: HPLC-R
Run time	: 2019-03-15 10:44:17



1 PDA Multi 1 / 220nm,4nm

Integration result

PeakTable

PDA Ch1 220nm

Peak#	Ret. Time	USP Width	Resolution	Height	Area	Area %
1	1.108	0.054	0.000	899786	1796014	99.440
2	1.205	0.038	2.119	1508	1987	0.110
3	1.600	0.041	9.980	3493	5282	0.292
4	2.207	0.046	13.922	1715	2845	0.157
Total				906501	1806128	100.000

Operater : _____

Date : _____

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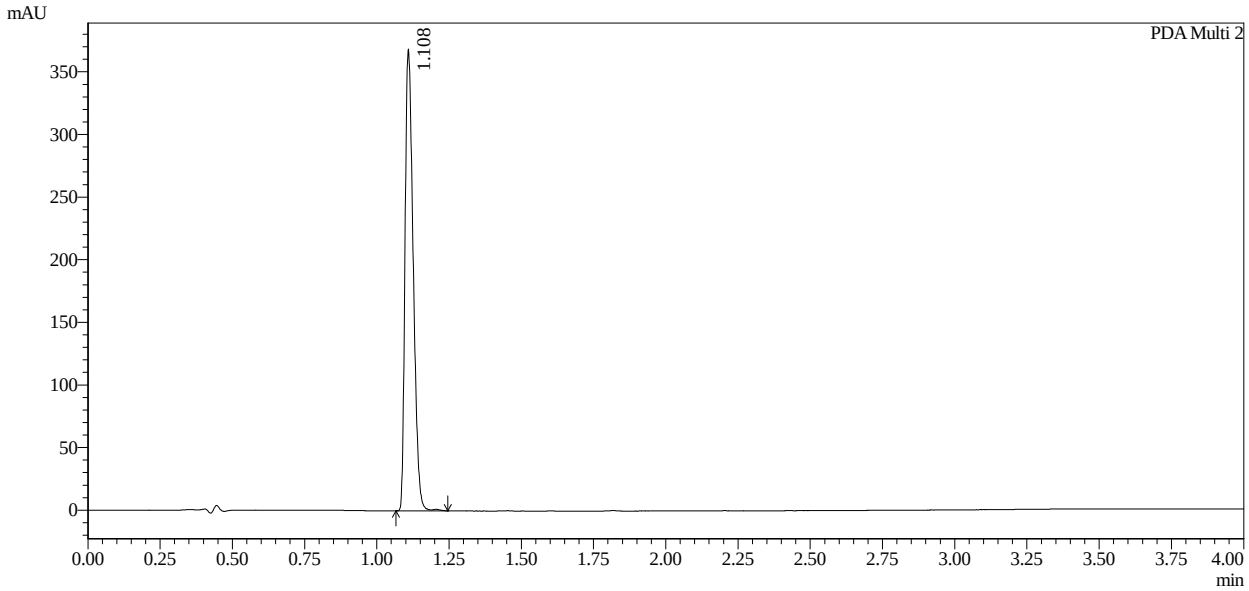
HPLC REPORT

Compound ID : EW618-1841-P1A
Sample ID : EW618-1841-P1H
Filename : D:\DATA\2019\1903\190315\EW618-1841-P1H.lcd
Method Name : D:\Method\10-80AB_4min.lcm
Instrument &Column : HPLC-R
Run time : 2019-03-15 10:44:17



1 PDA Multi 2 / 254nm,4nm

Integration result

PeakTable

PDA Ch2 254nm

Peak#	Ret. Time	USP Width	Resolution	Height	Area	Area %
1	1.108	0.053	0.000	368799	728870	100.000
Total				368799	728870	100.000

Operater : _____

Date : _____

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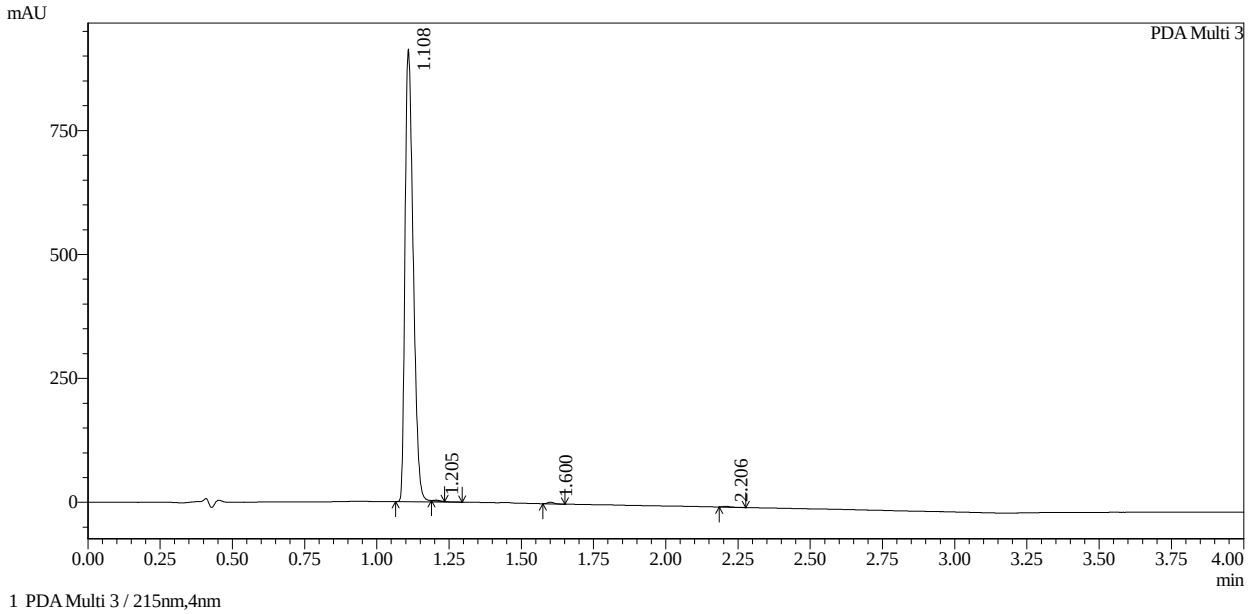
HPLC REPORT

Compound ID : EW618-1841-P1A
Sample ID : EW618-1841-P1H
Filename : D:\DATA\2019\1903\190315\EW618-1841-P1H.lcd
Method Name : D:\Method\10-80AB_4min.lcm
Instrument &Column : HPLC-R
Run time : 2019-03-15 10:44:17



1 PDA Multi 3 / 215nm,4nm

Integration result

PeakTable

PDA Ch3 215nm

Peak#	Ret. Time	USP Width	Resolution	Height	Area	Area %
1	1.108	0.054	0.000	913066	1811882	99.473
2	1.205	0.037	2.134	1466	1909	0.105
3	1.600	0.042	10.119	3360	4992	0.274
4	2.206	0.044	14.167	1556	2698	0.148
Total				919448	1821481	100.000

Operater : _____

Date : _____