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#### **Supporting Information**

# Peptide Synthesis Beyond DMF: THF and ACN as Excellent and Friendlier Alternatives

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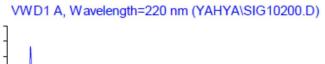
<sup>&</sup>lt;sup>f</sup> Department of Chemistry, College of Science, King Saud University P.O. Box 2455,Riyadh11451, Saudi Arabia

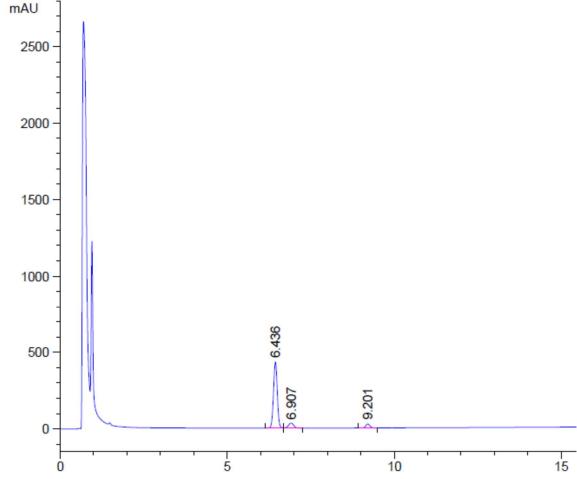
<sup>&</sup>lt;sup>g</sup> School of Chemistry and Physics, University ofKwazulu-Natal, Durban 4001, South Africa

h Department of Organic Chemistry, University of Barcelona, 08028-Barcelona, Spain

# 1- Z-Phg-Pro-NH<sub>2</sub> Racemization test:

#### 1.1- HOBt in DMF



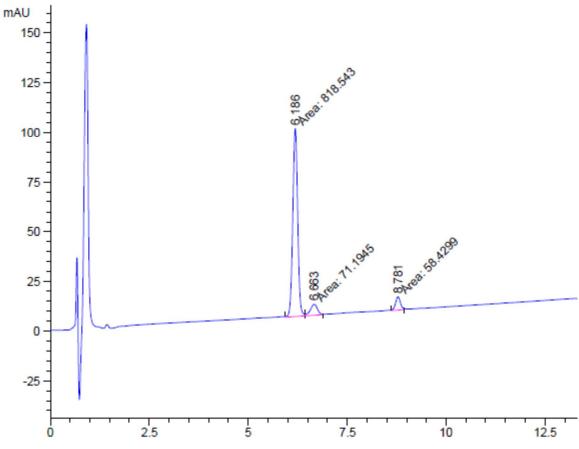


Peak	RetTime	Туре	Width	A:	rea	Hei	.ght	Area
#	[min]		[min]	mAU	*S	[mAU	]	8
1	6.436	PV	0.1242	3414	.27417	432.	31744	85.0454
2	6.907	VB	0.1751	375	.11981	33.	31265	9.3438
3	9.201	BB	0.1312	225	.25343	26.	50491	5.6108

Totals: 4014.64742 492.13500

#### 1.2- HOBt in THF



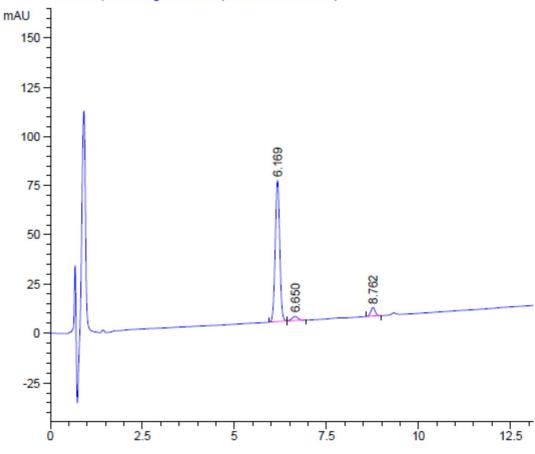


Peak	RetTime	Type	Width	A:	rea	Hei	ght	Area
#	[min]		[min]	mAU	*s	[mAU	]	8
1	6.186	MF	0.1435	818	.54346	95.	06696	86.3290
2	6.663	FM	0.2107	71	.19449	5.	63223	7.5086
3	8.781	MM	0.1449	58	.42990	6.	71888	6.1624

Totals: 948.16784 107.41807

#### 1.2- HOBt in ACN

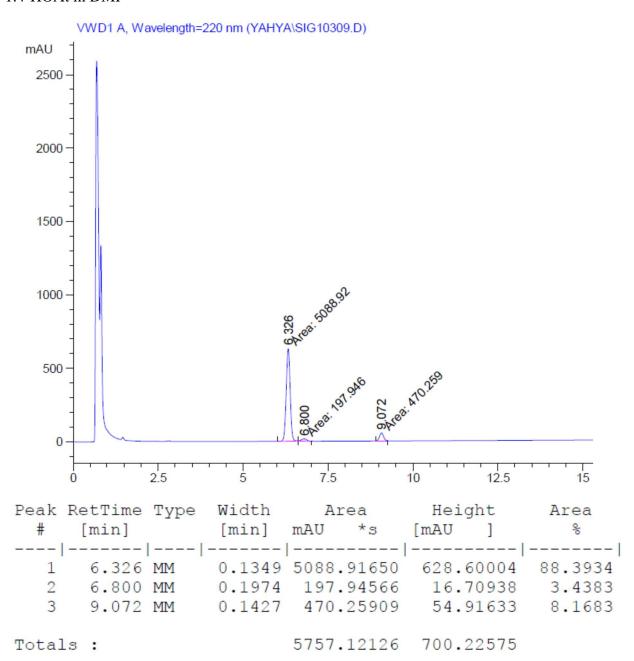




Peak	RetTime	Type	Width	Aı	rea	Heig	ght	Area
#	[min]		[min]	mAU	*s	[mAU	]	8
1	6.169	BB	0.1328	612.	57001	71.9	94768	90.6859
2	6.650	BP	0.1825	26.	65774	2.2	21760	3.9465
3	8.762	BB	0.1287	36.	25735	4.3	37463	5.3676

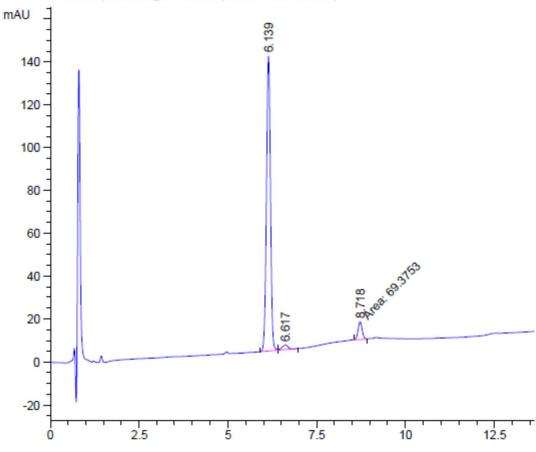
Totals: 675.48510 78.53990

#### 1.4-HOAt in DMF



#### 1.5-HOAt in THF



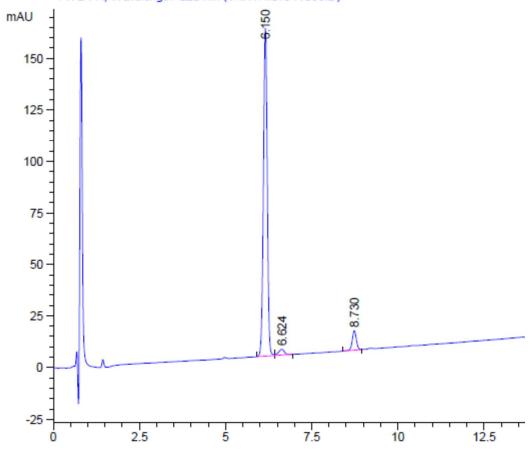


Peak	RetTime	RetTime Type Width		A	rea	Height		Area
#	[min]		[min]	mAU	* s	[mAU	]	8
1	6.139	BB	0.1216	1074	.28430	137.	72717	91.9310
2	6.617	BP	0.1844	24	.91660	2.	06585	2.1322
3	8.718	MM	0.1395	69	.37532	8.	28620	5.9367

Totals: 1168.57622 148.07922

#### 1.6-HOAt in ACN



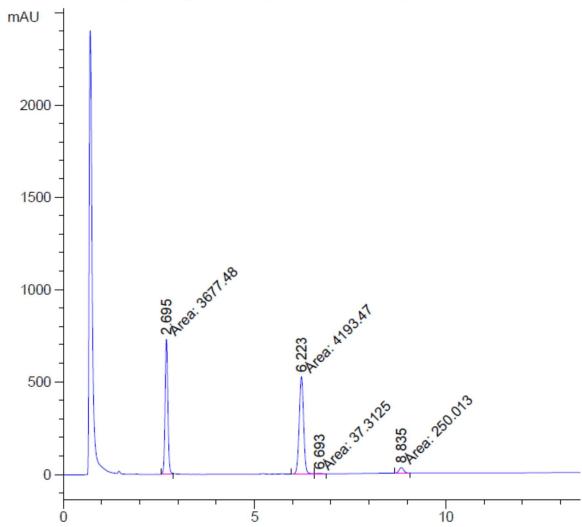


Peak	RetTime	Type	Width	A.	rea	Heig	ght	Area
#	[min]		[min]	mAU	*s	[mAU	]	8
1	6.150	BB	0.1208	1229	.13416	159.0	02092	91.6198
2	6.624	BP	0.1798	33	.10896	2.8	30707	2.4679
3	8.730	BB	0.1306	79	.31651	9.3	38959	5.9123

Totals: 1341.55962 171.21757

## 1.7-OxymaPure in DMF



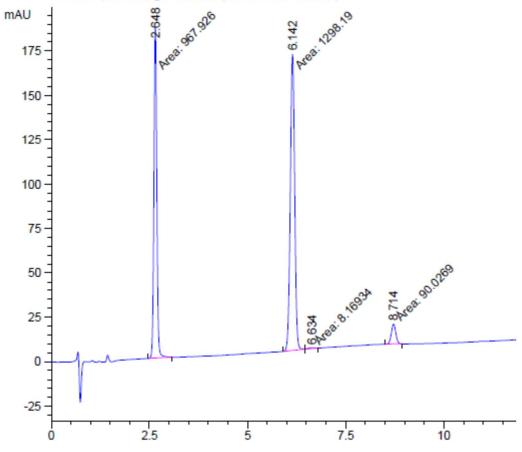


RetTime	Type	Width	Area		Height		Area
[min]		[min]	mAU	* S	[mAU	]	ક
2.695	MM	0.0834	3677	.48315	735.	30951	45.0767
6.223	MF	0.1329	4193	.46582	525.	72107	51.4014
6.693	FM	0.2116	37	.31252	2.	93868	0.4574
8.835	MM	0.1394	250	.01295	29.	89925	3.0645
	[min]  2.695 6.223 6.693	[min]   - 2.695 MM 6.223 MF 6.693 FM	[min] [min]	[min] [min] mAU    2.695 MM	[min] [min] mAU *s	[min] [min] mAU *s [mAU	

Totals: 8158.27445 1293.86851

## 1.8-OxymaPure in THF



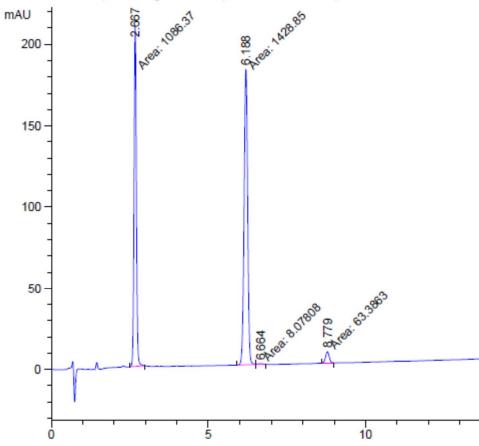


Peak	RetTime	Type	Width	A:	rea	Hei	ght	Area	
#	[min]		[min]	mAU	*s	[mAU	]	8	
1	2.648	MM	0.0860	967	.92639	187.	50903	40.9390	
2	6.142	MF	0.1294	1298	.19031	167.	14500	54.9077	
3	6.634	FM	0.2187	8	.16934	6.224	77e-1	0.3455	
4	8.714	MM	0.1339	90	.02687	11.	20521	3.8077	

Totals: 2364.31291 366.48172

## 1.9-OxymaPure in ACN



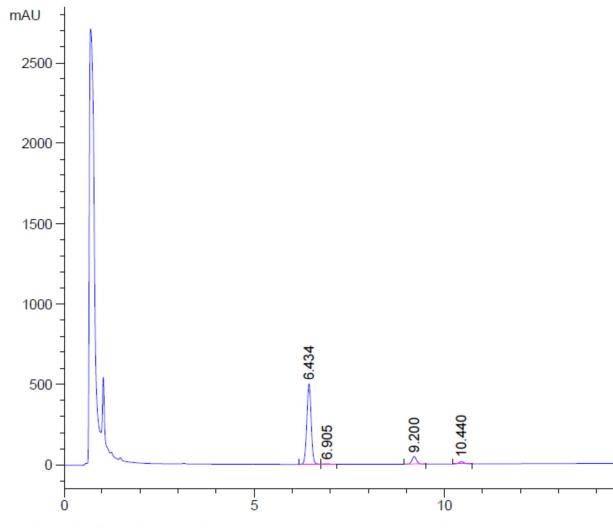


Peak :			Width Area			Heig	ght	Area
#	[min]		[min]	mAU	* s	[mAU	]	8
1	2.667	MM	0.0860	1086.	37109	210.4	44673	41.9986
2	6.188	MF	0.1305	1428.	84729	182.4	46661	55.2386
3	6.664	FM	0.2152	8.	07808	6.257	71e-1	0.3123
4	8.779	MM	0.1456	63.	38634	7.2	25517	2.4505

Totals: 2586.68281 400.79428

## 1.10-Oxyma-B in DMF

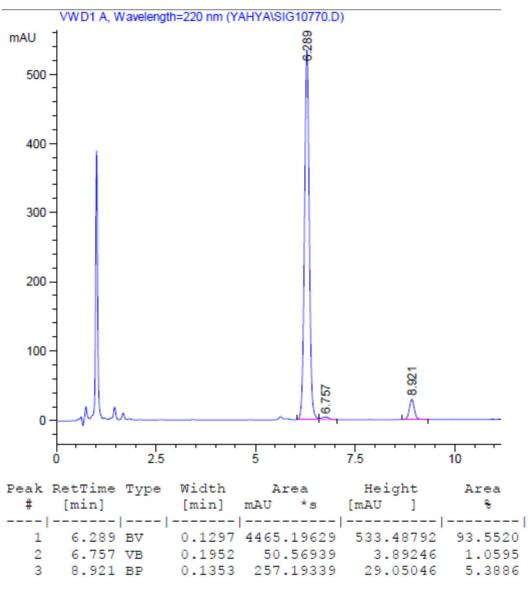




Peak	RetTime	Type	Width	Aı	rea	Heig	ght	Area
#	[min]		[min]	mAU	* S	[mAU	]	ક
		-						
1	6.434	PV	0.1222	3953.	95679	495.5	6058	87.3388
2	6.905	VV	0.1890	40.	04154	3.1	L5152	0.8845
3	9.200	BB	0.1298	394.	05420	47.0	2825	8.7042
4	10.440	BB	0.1418	139.	09808	15.1	L8406	3.0725

Totals: 4527.15061 560.92440

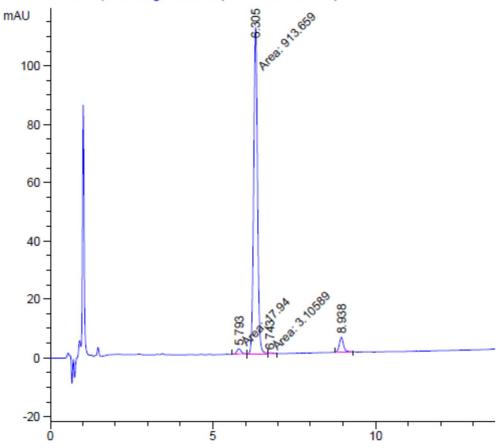
#### 1.11-Oxyma-B in THF



Totals: 4772.95906 566.43084

## 1.12-Oxyma-B in ACN





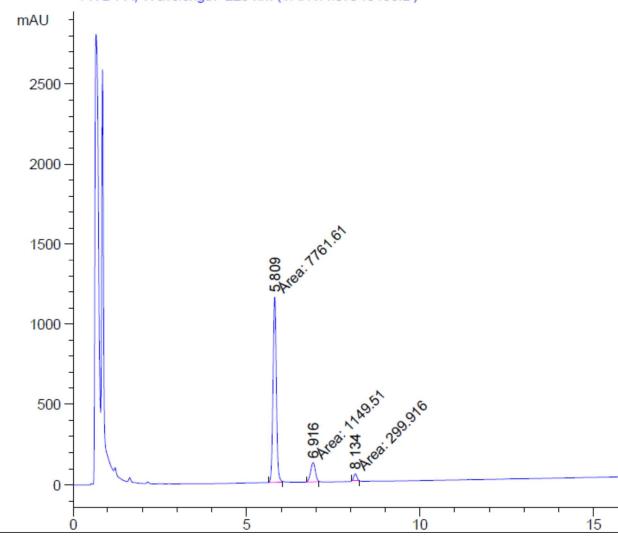
Peak 1	RetTime	Type	Width	Aı	rea	Hei	ght	Area
#	[min]		[min]	mAU	*s	[mAU	]	8
1	5.793	MF	0.1604	17.	.93997	1.	86427	1.8260
2	6.305	MF	0.1359	913.	.65930	112.	06988	92.9941
3	6.743	FM	0.1699	3.	.10589	3.047	17e-1	0.3161
4	8.938	BB	0.1448	47.	.78630	5.	07285	4.8638

Totals: 982.49146 119.31171

## 2-Z-Phe-Val-Pro-NH<sub>2</sub> Racemization test:

#### 2.1- HOBt in DMF



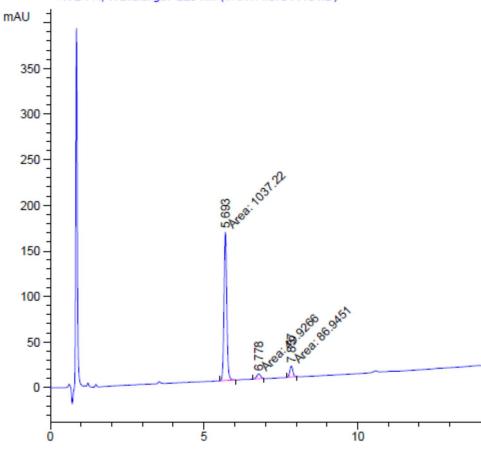


Peak RetTime	Type	Width	Ar	ea	Hei	ght	Area
# [min]		[min]	mAU	* S	[mAU	]	8
1 5.809	MM	0.1118	7761.	60791	1156.	86853	84.2642
2 6.916	MM	0.1590	1149.	51404	120.	45636	12.4797
3 8.134	MM	0.1096	299.	91574	45.	59239	3.2560

Totals: 9211.03769 1322.91728

#### 2.2- HOBt in THF



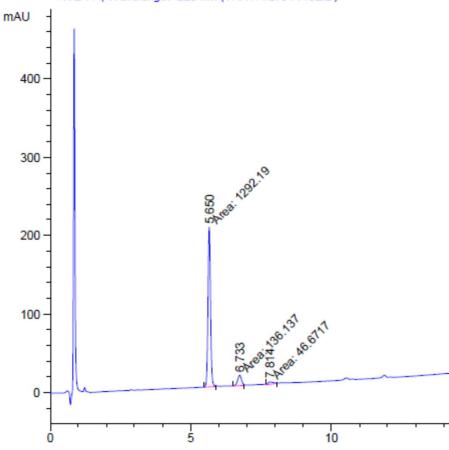


Peak RetTime T	ype Width	Width Area		Area
# [min]	[min]	mAU *s	[mAU ]	8
-				
1 5.693 M	M 0.1055	1037.21655	163.83356	88.3423
2 6.778 N	M 0.1548	49.92662	5.37366	4.2524
3 7.837 M	M 0.1173	86.94510	12.35470	7.4053

Totals: 1174.08827 181.56192

#### 2.3- HOBt in ACN

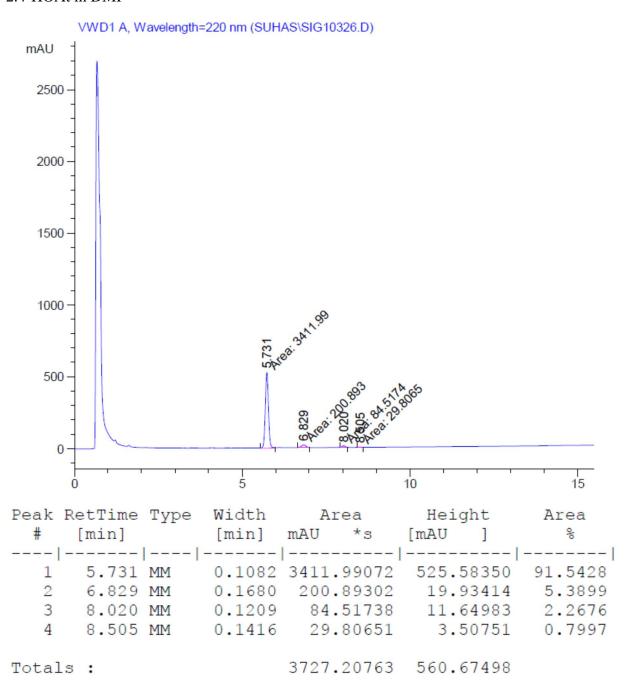




Peak RetTime # [min]					-		Area %
1 5.650 2 6.733 3 7.814	MM MM	0.1057	1292 136	.18628 .13747	203.7	77806 52609	

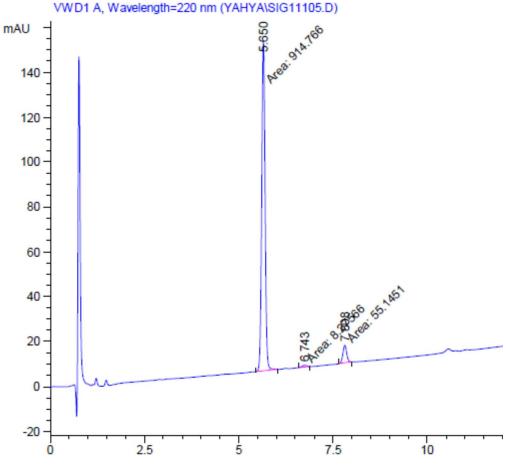
Totals: 1474.99547 220.49031

#### 2.4-HOAt in DMF



#### 2.5-HOAt in THF



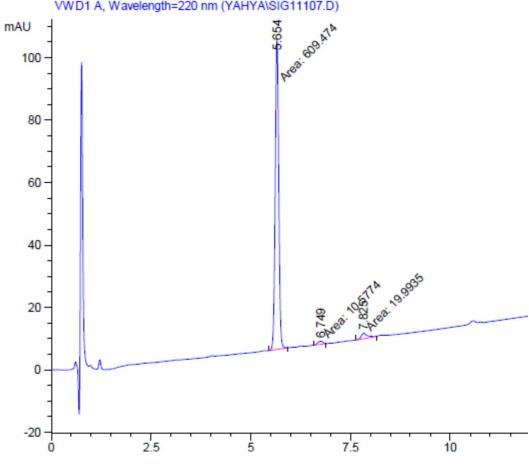


Peak	RetTime	Type	Width	Are	a	Heig	ht	Area	
#	[min]		[min]	mAU	*s	[mAU	]	8	
1	5.650	MM	0.1035	914.7	6642	147.3	2126	93.5165	
2	6.743	MM	0.1553	8.2	7566	8.8791	4e-1	0.8460	
3	7.808	MM	0.1172	55.1	4510	7.8	3931	5.6375	

Totals : 978.18717 156.04848

#### 2.6-HOAt in ACN

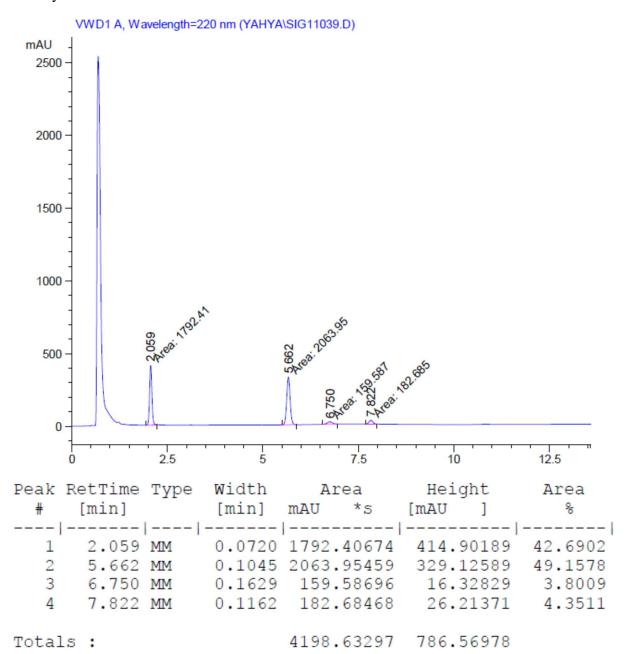




	RetTime [min]					_		Area %
	-		-			-	-	
1	5.654	MM	0.1023	609.	47351	99.3	1754	95.2236
2	6.749	MM	0.1609	10.	57745	1.0	9575	1.6526
3	7.825	MM	0.1868	19.	99349	1.7	8424	3.1238

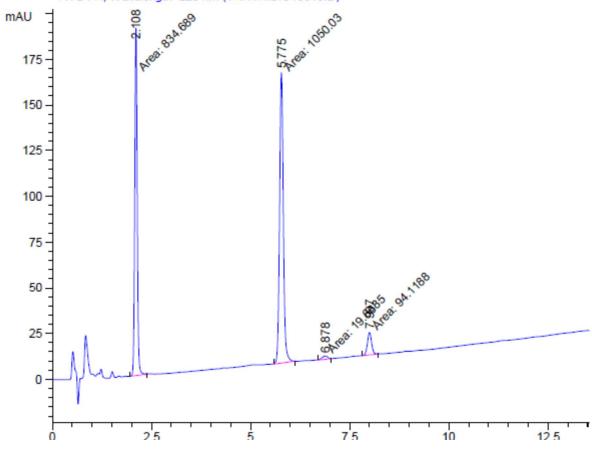
640.04445 102.19752 Totals :

#### 2.7-OxymaPure in DMF



## 2.8-OxymaPure in THF



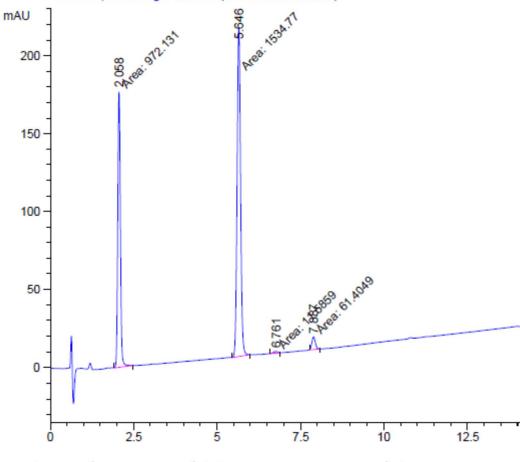


Peak	RetTime	Type	Width	A:	rea	Heig	ght	Area
#	[min]		[min]	mAU	* s	[mAU	]	8
1	2.108	MM	0.0727	834	.68903	191.4	41022	41.7656
2	5.775	MM	0.1097	1050	.03101	159.	54111	52.5408
3	6.878	MM	0.1721	19	.66851	1.9	90459	0.9842
4	7.997	MM	0.1266	94	.11881	12.3	38939	4.7095

Totals: 1998.50736 365.24531

## 2.9-OxymaPure in ACN



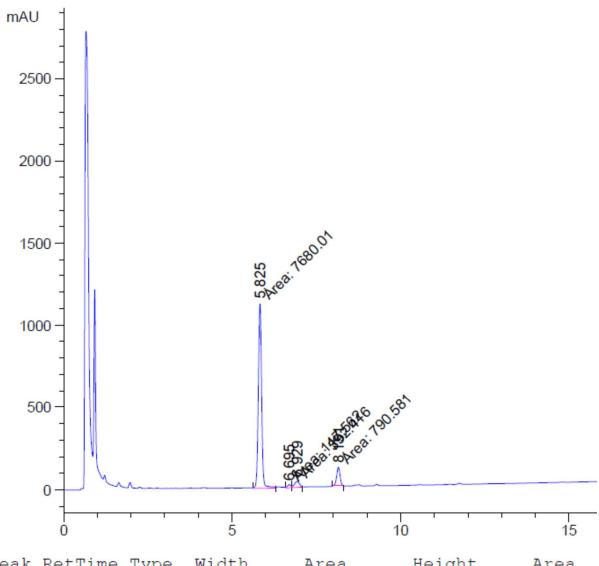


			Width [min]	mAU	*s	[mAU	]	Area %
1	2.058	MM	0.0914	972.1	3092	177.	35133	37.6811
2	5.646	MM	0.1209	1534.7	6538	211.5	51978	59.4896
3	6.761	MM	0.1674	11.5	8589	1.1	15338	0.4491
4	7.887	MM	0.1249	61.4	0490	8.1	19594	2.3801

Totals: 2579.88708 398.22043

## 2.10-Oxyma-B in DMF

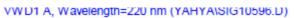
VWD1 A, Wavelength=220 nm (YAHYA\SIG10109.D)

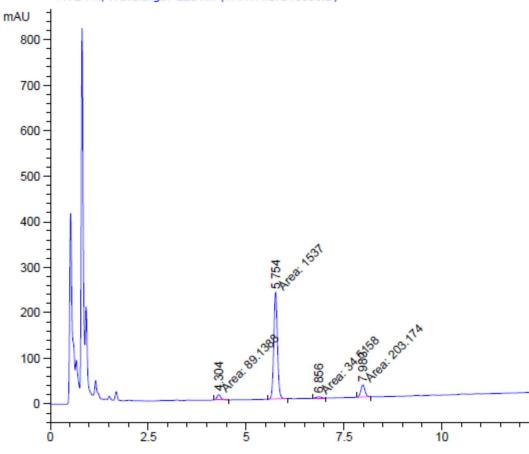


Peak 1	RetTime	Type	Width	n Area		Height		Area
#	[min]		[min]	mAU	* S	[mAU	]	용
1	5.825	MM	0.1138	7680.	01074	1124.	86694	85.2334
2	6.695	MF	0.1221	147.	56154	20.	13989	1.6376
3	6.929	FM	0.1768	392.	41583	36.	99476	4.3551
4	8.151	MM	0.1176	790.	58063	112.	03448	8.7739

Totals: 9010.56874 1294.03608

## 2.11- Oxyma-B in THF



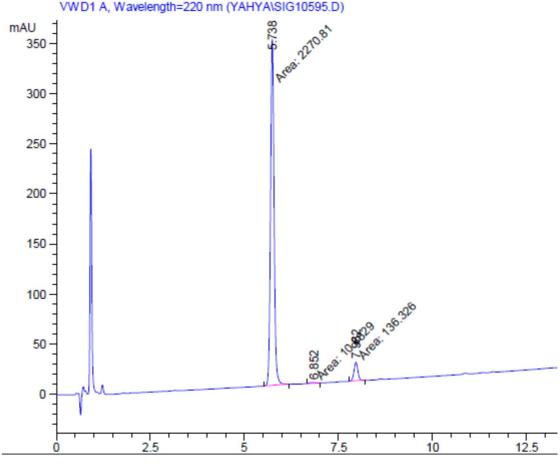


Peak I	RetTime	Type	Width	A:	rea	Hei	ght	Area
#	[min]		[min]	mAU	*s	[mAU	]	8
-								
1	4.304	MM	0.1249	89	.13880	11.	89540	4.7826
2	5.754	MM	0.1094	1536	.99585	234.	20184	82.4646
3	6.856	MM	0.1735	34	.51583	3.	31647	1.8519
4	7.983	MM	0.1261	203	.17403	26.	85485	10.9009

Totals: 1863.82451 276.26856

## 2.12- Oxyma-B in ACN





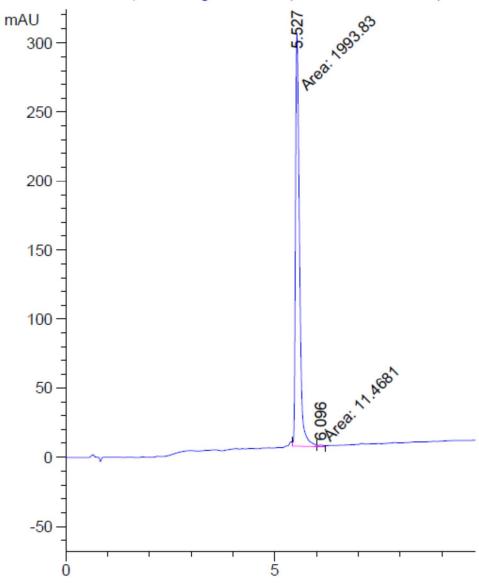
Peak	RetTime	Type	Width	A	rea	Heig	ght	Area	
#	[min]		[min]	mAU	* 8	[mAU	]	8	
1	5.738	MM	0.1092	2270	.80737	346.	43256	93.9120	
2	6.852	MM	0.2469	10	.88295	7.345	94e-1	0.4501	
3	7.962	MM	0.1218	136	.32571	18.	65207	5.6379	

Totals : 2418.01604 365.81922

# 3- H-Gly-Ser-Phe-NH<sub>2</sub> racemization Experiments

#### 3.1-HOBt in DMF



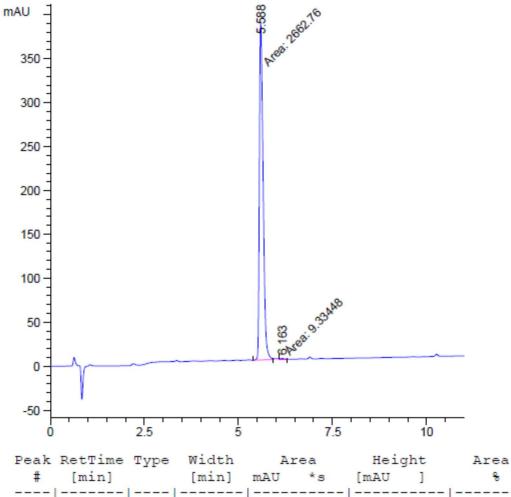


Peak	RetTime	Type	Width	Are	a	Hei	ght	Area
	[min]		-			-	_	
1	5.527	FM	0.1110	1993.8	2751	299.	39377	99.4281
2	6.096	FM	0.1540	11.4	6806	1.	24112	0.5719

Totals: 2005.29557 300.63489

#### 3.2-HOBt in THF



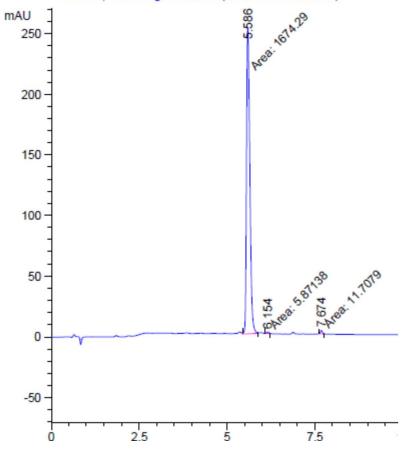


Peak	RetTime	Type	Width	Area	Height	Area
#	[min]		[min]	mAU *s	[mAU ]	8
					-	
1	5.588	MF	0.1154	2662.7575	7 384.60492	99.6507
2	6.163	FM	0.1340	9.3344	3 1.16087	0.3493

Totals: 2672.09205 385.76579

#### 3.2-HOBt in ACN

VWD1 A, Wavelength=220 nm (YAHYA\SIG10813.D)

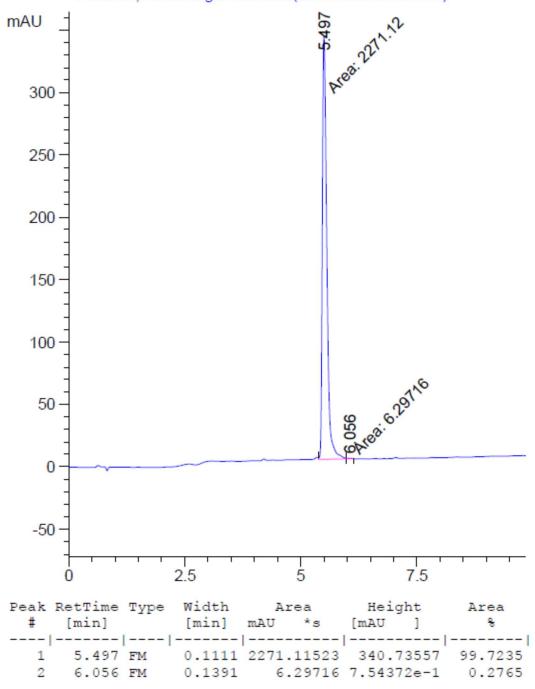


#	RetTime [min]		[min]	mAU	*s	[mAU	]	
1	5.586	MM	0.1101	1674.	28699	253.	38156	98.9610
2	6.154	MM	0.0771	5.	87138	1.	26933	0.3470
3	7.674	MM	0.0681	11.	70785	2.	86335	0.6920

Totals: 1691.86622 257.51424

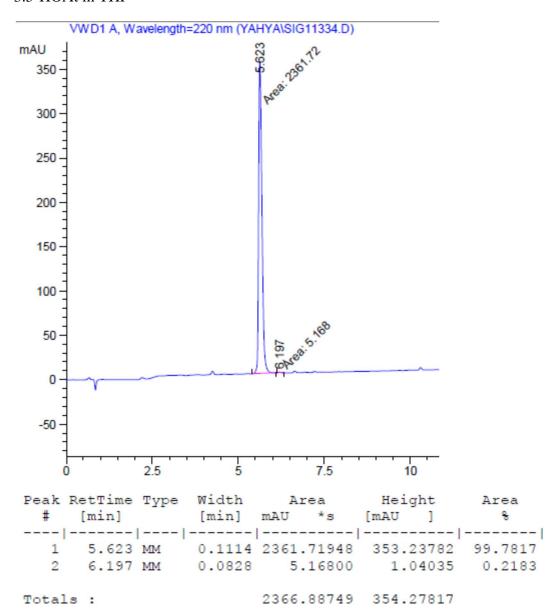
#### 3.4-HOAt in DMF

VWD1 A, Wavelength=220 nm (YAHYA\SIG11932.D)



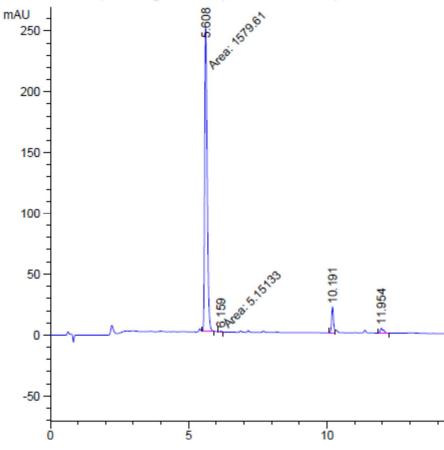
Totals: 2277.41239 341.48994

#### 3.5-HOAt in THF



#### 3.6-HOAt in ACN

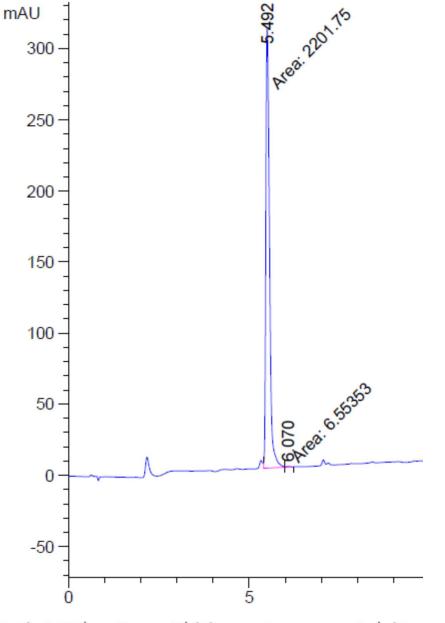




Peak	RetTime	Type	Width	dth Area		Height		Area
#	[min]		[min]	mAU	*s	[mAU	]	8
1	5.608	MM	0.1045	1579.	61108	251.	91493	91.9447
2	6.159	MM	0.1048	5.	15133	8.188	93e-1	0.2998
3	10.191	BV	0.0733	101.	99277	21.	32008	5.9367
4	11.954	BB	0.1097	31.	24550	3.	95163	1.8187

Totals: 1718.00069 278.00554

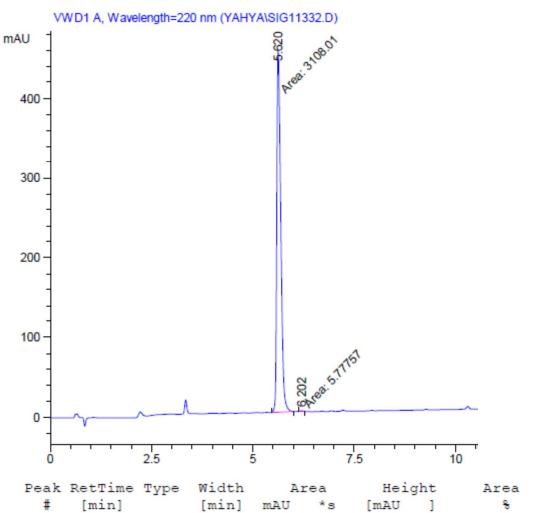
# 3.7-OxymaPure in DMF



				Area		Area	
#	[min]		[min]	mAU *s	[mAU ]	8	
1	5.492	FM	0.1184	2201.74780	310.03635	99.7032	
2	6.070	FM	0.1490	6.55353	7.33042e-1	0.2968	

Totals: 2208.30133 310.76939

## 3.8-OxymaPure in THF

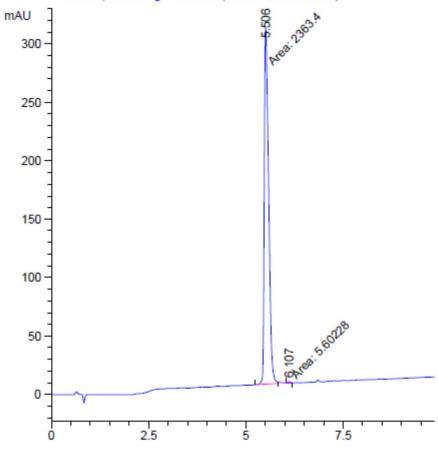


Peak RetTim	e Type	Width	Area		Height		Area	
# [min]		[min]	mAU	*s	[mAU	]	8	
	-							
1 5.62	0 MM	0.1138	3108.	01392	455.	16977	99.8145	
2 6.20	2 MM	0.0798	5.	77757	1.	20715	0.1855	

Totals: 3113.79149 456.37692

## 3.9-OxymaPure in ACN



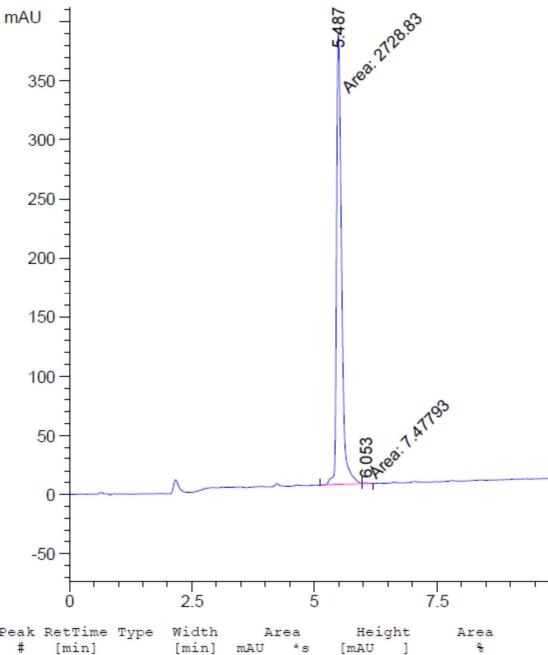


Peak	RetTime	Type	Width	Are	ea	Hei	ght	Area
	_		[min]			[mAU	-	
1	5.506	MF	0.1285	2363.3	39551	306.	42755	99.7635
2	6.107	FM	0.0795	5.6	50228	1.	17441	0.2365

Totals: 2368.99779 307.60196

## 3.10- Oxyma-B in DMF

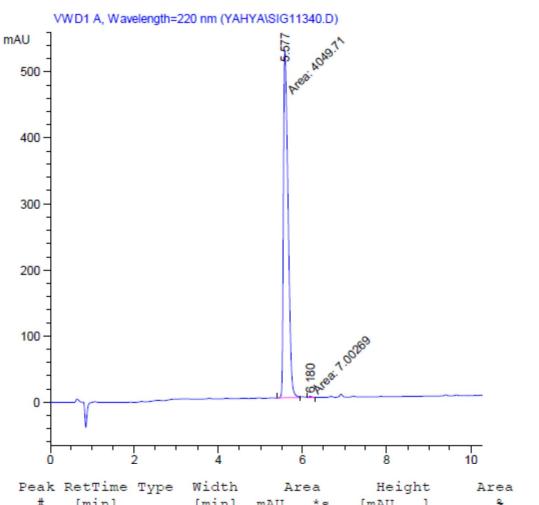
VWD1 A, Wavelength=220 nm (YAHYA\SIG11933.D)



Peak	RetTime	Type	Width	Ar	ea	Hei	ght	Area
#	[min]		[min]	mAU	*s	[mAU	]	8
1	5.487	MF	0.1186	2728.	83398	383.	47549	99.7267
2	6.053	FM	0.1387	7.	47793	8.983	71e-1	0.2733

Totals: 2736.31191 384.37387

## 3.11- Oxyma-B in THF

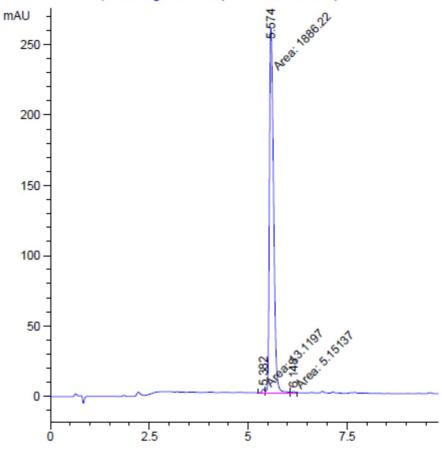


Peak RetTime	tTime Type Width Area		He	ight	Area	
# [min]		[min]	mAU *s	[mAU	]	8
1 5.577	MF	0.1283	4049.709	72 526.	.22577	99.8274
2 6.180	MM	0.0848	7.002	69 1.	.37558	0.1726

Totals: 4056.71241 527.60135

# 3.12- Oxyma-B in ACN





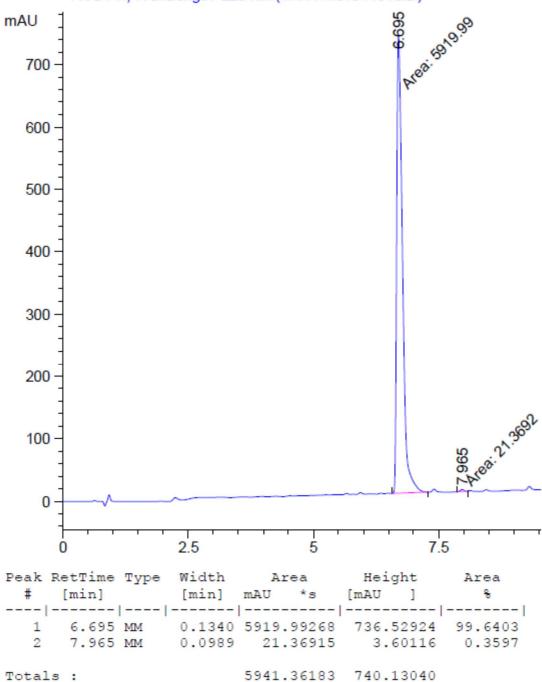
				Area		Height [mAU ]			
			-			-	-	<del>5</del>	i
				•		•		0.6889	•
2	5.574	FM	0.1209	1886	.22241	259.	94125	99.0406	
3	6.148	FM	0.1006	5	.15137	8.530	31e-1	0.2705	

Totals: 1904.49346 263.42276

# 4- H-Gly-Cys-Phe-NH $_2$ racemization experiments

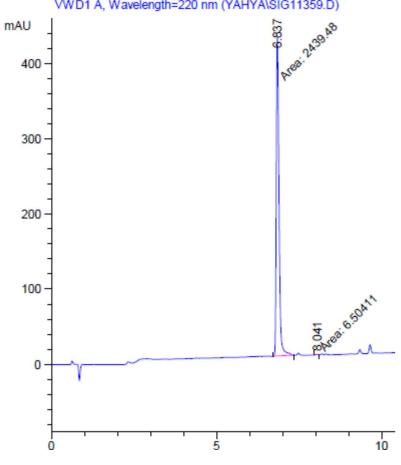
#### 4.1-HOBt in DMF





# 4.2-HOBt in THF



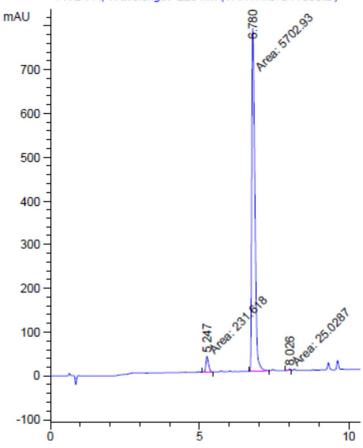


Peak I	RetTime	Type	Width	Area		Height		Area	
#	[min]		[min]	mAU	* 8	[mAU	]	8	
-									
1	6.837	MM	0.0955	2439.	.47510	425.	72949	99.7341	
2	8.041	MM	0.1043	6.	.50411	1.	03965	0.2659	

Totals : 2445.97921 426.76914

## 4.3-HOBt in ACN



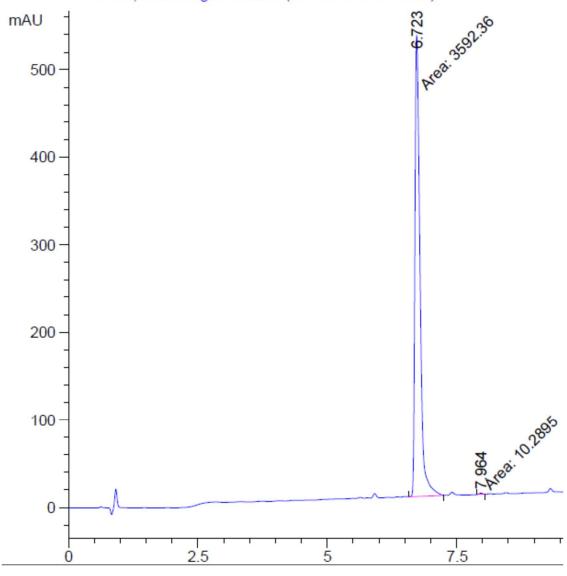


- 1
5
5
0

Totals: 5959.58000 826.48721

## 4.4-HOAt in DMF

VWD1 A, Wavelength=220 nm (YAHYA\SIG11945.D)

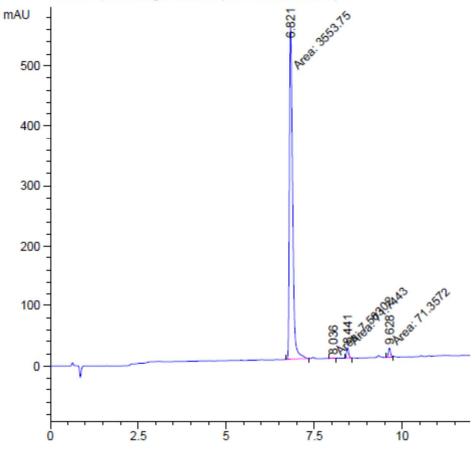


Peak	RetTime	Type	Width	Are	a	Heig	jht	Area
	-		[min]			-	-	8
1	6.723	MM	0.1132	3592.3	6328	528.8	32007	99.7144
2	7.964	MM	0.0931	10.2	8946	1.8	34258	0.2856

Totals: 3602.65274 530.66265

## 4.5-HOAt in THF



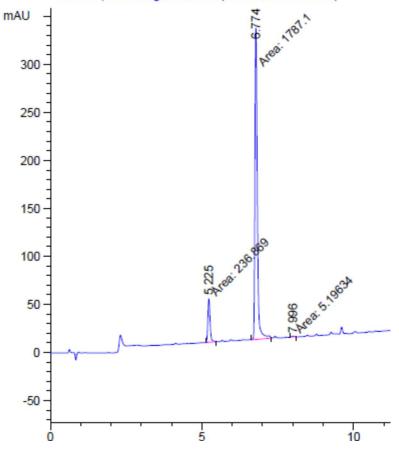


Peak	RetTime	Type	Width	Area		_		Area
#	[min]		[min]	mAU	*s	[mAU	]	8
1	6.821	MM	0.1065	3553.	75195	556.2	20020	95.9328
2	8.036	MM	0.1072	7.	56302	1.1	17606	0.2042
3	8.441	MM	0.0678	71.	74433	17.6	3185	1.9367
4	9.628	MM	0.0751	71.	35717	15.8	33867	1.9263

Totals: 3704.41647 590.84677

## 4.6-HOAt in ACN



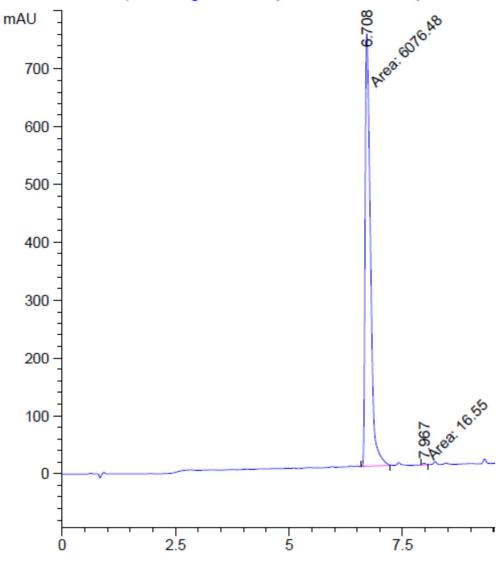


RetTime	Type	Width	Area		Height		Area	
[min]		[min]	mAU	*s	[mAU	]	8	
5.225	MM	0.0868	236	.86908	45.	48138	11.6732	
6.774	MM	0.0913	1787	.09766	326.	34229	88.0707	
7.996	MM	0.1049	5	.19634	8.255	80e-1	0.2561	
	[min]   5.225 6.774	[min]    5.225 MM 6.774 MM	[min] [min] 	[min] [min] mAU 	[min] [min] mAU *s 	[min] [min] mAU *s [mAU	[min] [min] mAU *s [mAU ]	 5.225 MM

Totals: 2029.16307 372.64924

## 4.7-OxymaPure in DMF

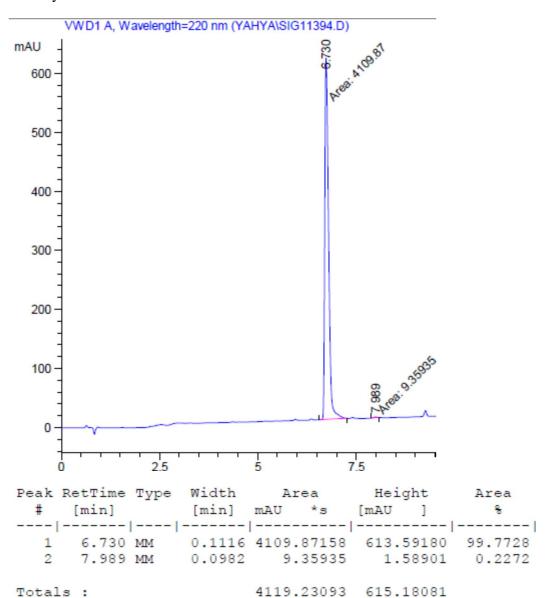
VWD1 A, Wavelength=220 nm (YAHYA\SIG11947.D)



Peak R	RetTime	Type	Width	Area		Height		Area	
#	[min]		[min]	mAU	*s	[mAU	]	8	
-									
1	6.708	MM	0.1353	6076.	47998	748.	66882	99.7284	
2	7.967	MM	0.0886	16.	55003	3.	11349	0.2716	

Totals: 6093.03001 751.78232

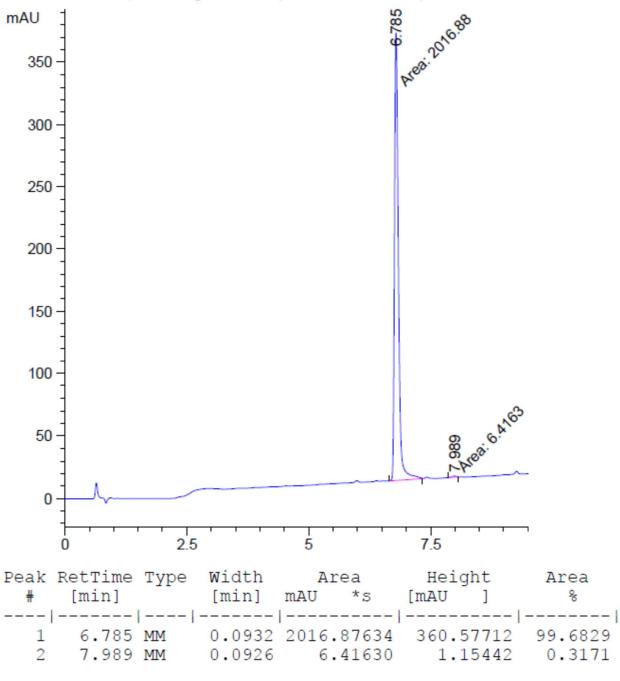
## 4.8-OxymaPure in THF



## 4.9-OxymaPure in ACN

Totals:

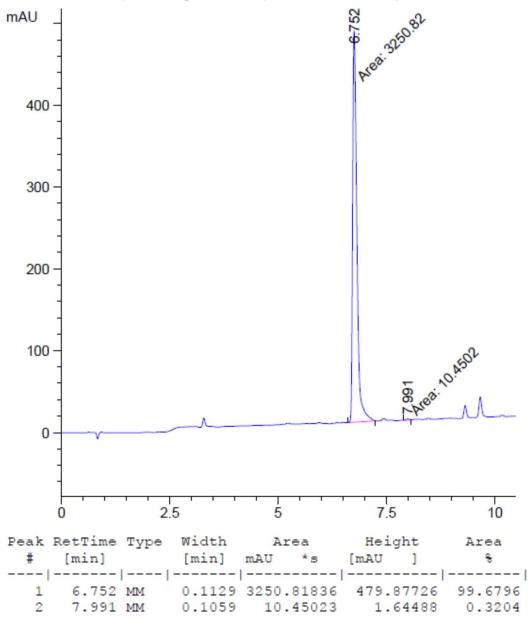




2023.29264 361.73154

## 4.10- Oxyma-B in DMF

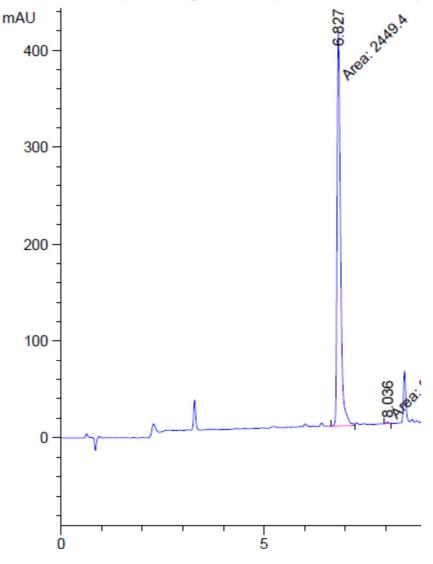




Totals: 3261.26859 481.52214

# 4.11- Oxyma-B in THF

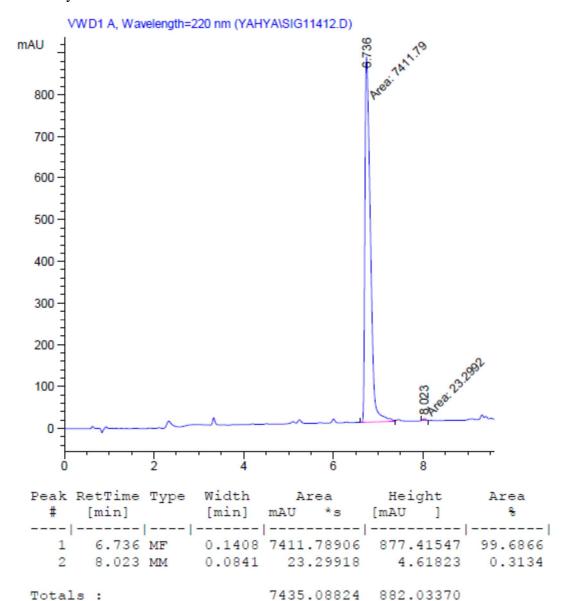
VWD1 A, Wavelength=220 nm (YAHYA\SIG11433.D)



Peak	RetTime	Туре	Width	Area		Height		Area
#	[min]		[min]	mAU	* S	[mAU	]	8
1	6.827	MM	0.0998	2449.	40479	408.	85620	99.6601
2	8.036	MM	0.0882	8.	35345	1.	57886	0.3399

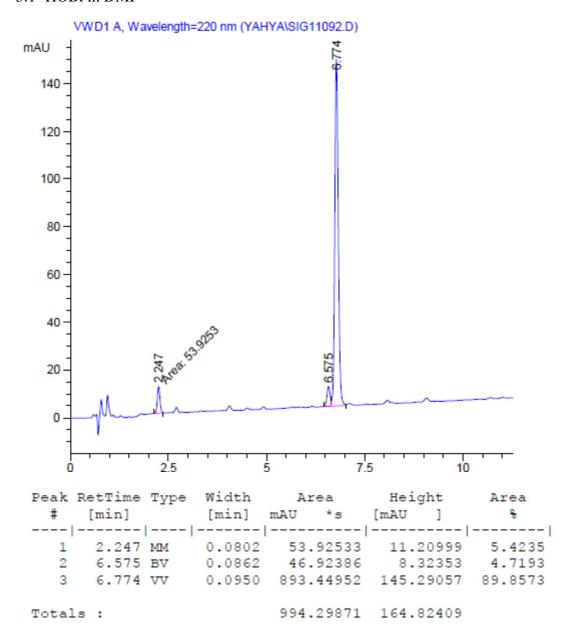
Totals: 2457.75824 410.43507

## 4.12- Oxyma-B in ACN



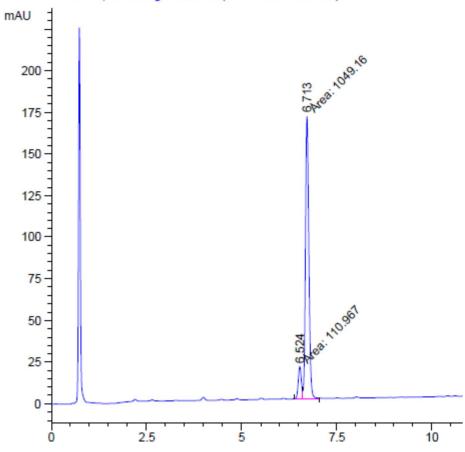
# 5- Peptide synthesis experiments (Synthesis of H-Tyr-Aib-Aib-Phe-Leu-NH<sub>2</sub>)

#### 5.1- HOBt in DMF



## 5.2- HOBt in THF



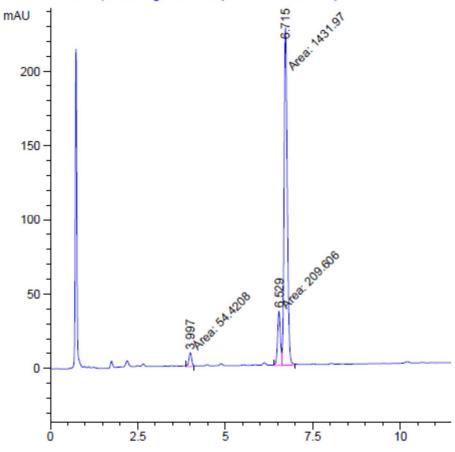


Peak	RetTime	Type	Width	Ar	ea	Hei	ght	Area
	[min]					_	_	
1	6.524	MF	0.0949	110.	96663	19.	48471	9.5651
2	6.713	FM	0.1028	1049.	15552	170.	06830	90.4349

Totals: 1160.12215 189.55301

## 5.3- HOBt in ACN

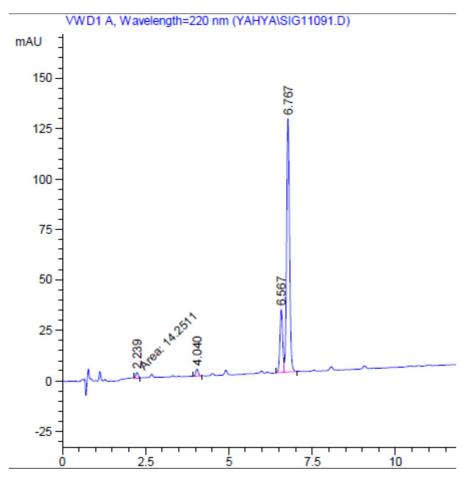




			Width [min]			Heig [mAU		Area %
1	3.997	MM	0.0959	54.	42081	9.4	6010	3.2088
2	6.529	MF	0.0959	209.	60553	36.4	2484	12.3588
3	6.715	FM	0.1048	1431.	97473	227.8	30069	84.4324

Totals: 1696.00107 273.68564

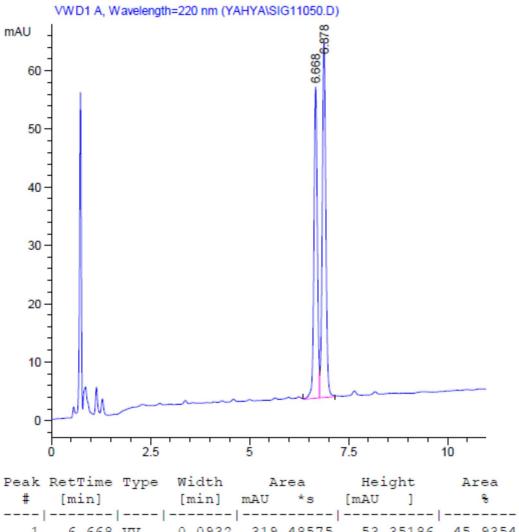
## 5.4- HOAt in DMF



Peak	RetTime	Туре	Width	A	rea	Hei	ght	Area
#	[min]		[min]	mAU	*s	[mAU	]	8
1	2.239	MM	0.0844	14	.25107	2.	81496	1.4482
2	4.040	BB	0.0833	18	.71312	3.	47215	1.9017
3	6.567	BV	0.0887	179	.05470	31.	25850	18.1961
4	6.767	VB	0.0949	772	.00934	125.	85519	78.4540

Totals: 984.02823 163.40080

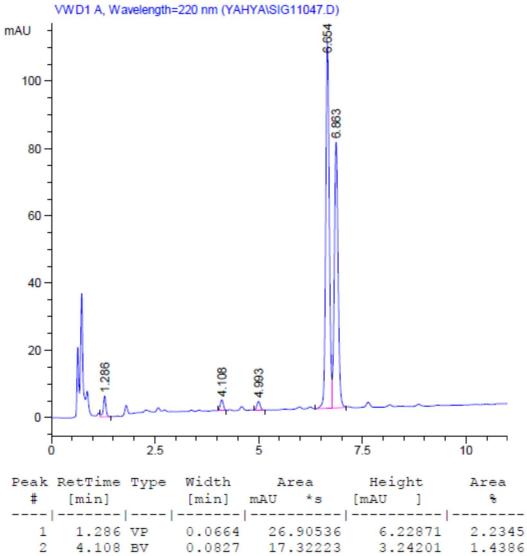
## 5.5- HOAt in THF



Peak	RetTime	Type	Width	Area		Height		Area	
#	[min]		[min]	mAU	*s	[mAU	]	8	
1	6.668	VV	0.0932	319.	48575	53.3	35186	45.9354	
2	6.878	VB	0.0956	376.	02457	60.	66456	54.0646	

Totals : 695.51031 114.01642

## 5.6- HOAt in ACN

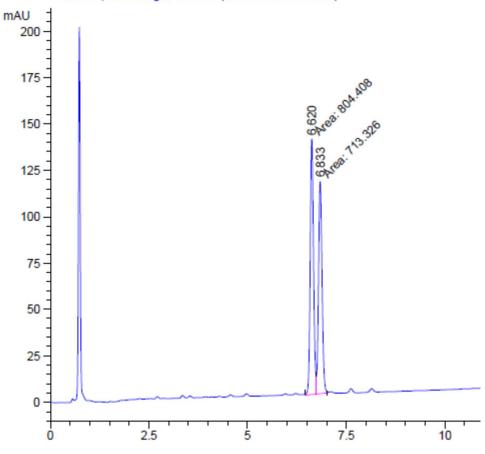


1	1.286	VP	0.0664	26.90536	6.22871	2.2345
2	4.108	BV	0.0827	17.32223	3.24201	1.4386
3	4.993	BP	0.0861	14.46956	2.62754	1.2017
4	6.654	VV	0.0911	645.20673	108.71227	53.5851
5	6.863	VB	0.0970	500.17480	79.16366	41.5400

Totals: 1204.07869 199.97419

# 5.7- OxymaPure in DMF



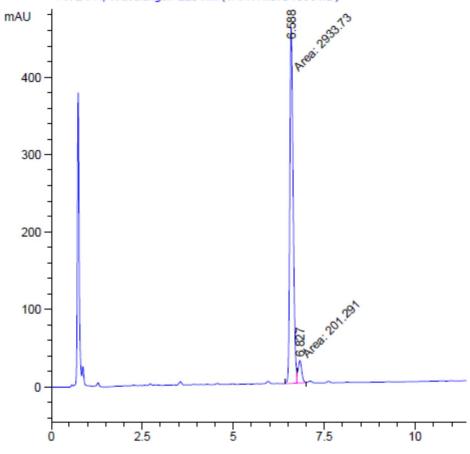


Peak RetT	ime Type	Width	Ar	ea	Hei	ght	Area	
# [mi:	n]	[min]	mAU	*s	[mAU	]	8	
1 6.	620 MF	0.0969	804.	40753	138.	30249	53.0006	
2 6.	833 FM	0.1036	713.	32593	114.	76675	46.9994	

Totals: 1517.73346 253.06924

# 5.8- OxymaPure in THF



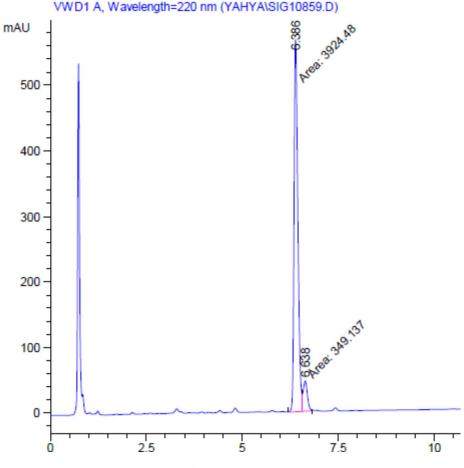


Peak	RetTime	Type	Width	Aı	cea	Heig	ght	Area	
#	[min]		[min]	mAU	*s	[mAU	]	8	
1	6.588	MF	0.1064	2933.	72656	459.7	72269	93.5793	
2	6.827	FM	0.1146	201.	29103	29.2	27492	6.4207	

Totals: 3135.01759 488.99760

# 5.9- OxymaPure in ACN



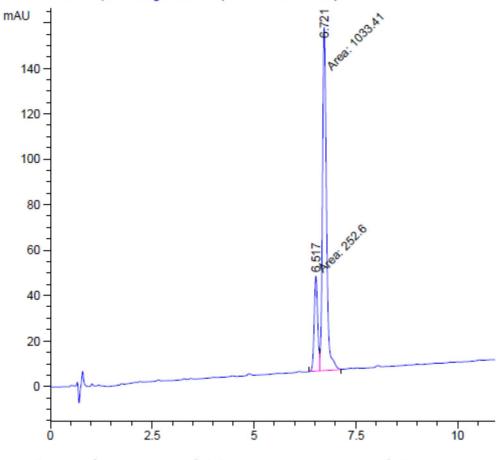


Peak	RetTime	Type	Width	Ar	ea	Hei	ght	Area
	[min]							
1	6.386	MF	0.1149	3924.	47827	569.	50177	91.8304
2	6.638	FM	0.1257	349.	13699	46.	29751	8.1696

Totals : 4273.61526 615.79928

# 5.10- Oxyma-B in DMF



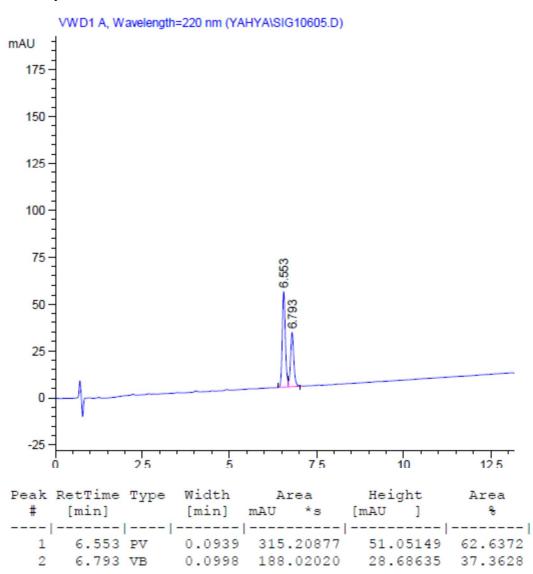


Peak	RetTime	Type	Width	Area		Height		Area	
#	[min]		[min]	mAU	*s	[mAU	]	8	
1	6.517	MF	0.0997	252.	59979	42.2	22205	19.6421	
2	6.721	FM	0.1136	1033.	41101	151.	64464	80.3579	

Totals: 1286.01080 193.86669

## 5.11- Oxyma-B in THF

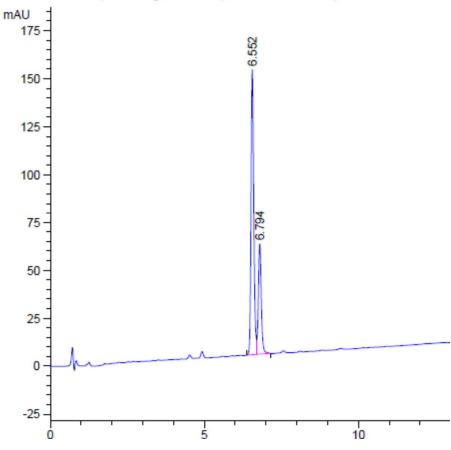
Totals :



503.22897 79.73784

# 5.12- Oxyma-B in ACN



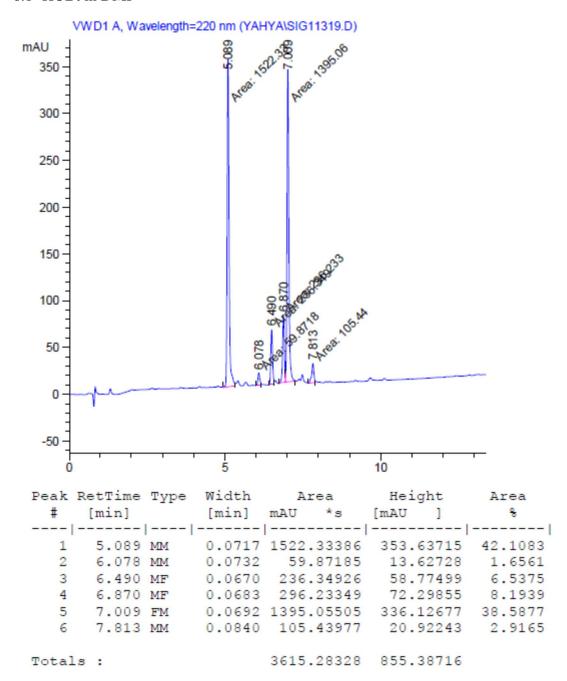


Peak	RetTime	Type	Width	Aı	cea	Height		Area	
#	[min]		[min]	mAU	*s	[mAU	]	8	
1	6.552	PV	0.0935	914.	51971	148.	85092	70.3082	
2	6.794	VB	0.1029	386.	21091	57.	68308	29.6918	
_	0.751	V 2	0.1023	500.	22001	0,.	00000	23.0310	

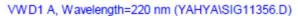
Totals: 1300.73062 206.53400

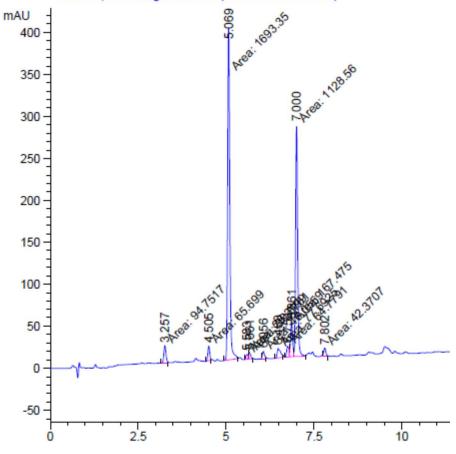
# 6- Peptide synthesis experiments (Synthesis of Aib-modified ACP decapeptide)

#### 6.1- HOBt in DMF



## 6.2- HOBt in THF



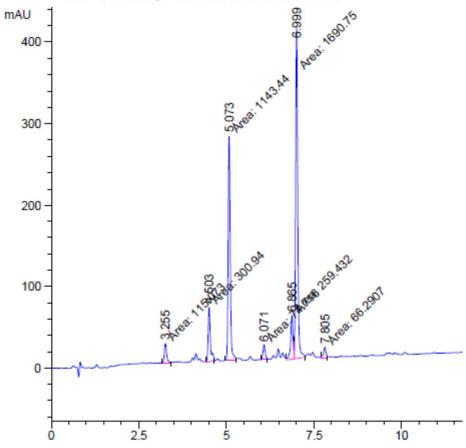


Peak RetTime	Type	Width	Area	Height	Area
# [min]		[min]	mAU *s	[mAU ]	8
1 3.257	MM	0.0748	94.75173	21.12108	2.7569
2 4.505	MM	0.0623	65.69898	17.57346	1.9116
3 5.069	MM	0.0707	1693.34741	399.26398	49.2693
4 5.583	MF	0.0740	25.31987	5.70302	0.7367
5 5.661	FM	0.0660	30.87609	7.79360	0.8984
6 6.056	MM	0.0789	46.05588	9.72683	1.3400
7 6.480	MM	0.1135	77.69253	11.40971	2.2605
8 6.750	MF	0.0827	64.77909	13.05362	1.8848
9 6.861	FM	0.0680	167.47545	41.03422	4.8728
10 7.000	FM	0.0681	1128.55688	276.34662	32.8362
11 7.802	MM	0.0710	42.37067	9.94491	1.2328

Totals: 3436.92459 812.97104

## 6.3- HOBt in ACN

VWD1 A, Wavelength=220 nm (YAHYA\SIG11357.D)

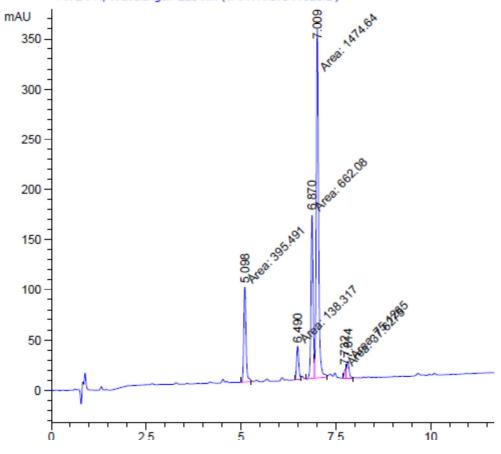


Peak RetTime 7		Type	Width	Area	Height	Area
#	[min]		[min]	mAU *s	[mAU ]	8
1	3.255	MM	0.0786	115.0729	24.40655	3.1521
2	4.503	MM	0.0743	300.9397	67.48935	8.2435
3	5.073	MM	0.0690	1143.4367	77 276.18784	31.3215
4	6.071	MM	0.0697	74.7159	17.86805	2.0467
5	6.865	MF	0.0799	259.4321	9 54.14082	7.1065
6	6.999	FM	0.0688	1690.7547	76 409.87616	46.3139
7	7.805	MM	0.0835	66.2906	13.23226	1.8159

Totals: 3650.64300 863.20103

## 6.4- HOAt in DMF



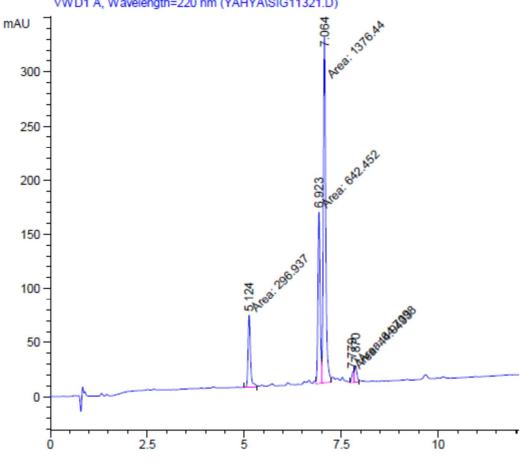


Peak	RetTime	Type	Width	A	rea	Hei	ght	Area
#	[min]		[min]	mAU	*s	[mAU	]	8
1	5.098	MM	0.0690	395	.49081	95.	52810	14.2095
2	6.490	MM	0.0681	138	.31744	33.	82759	4.9696
3	6.870	MF	0.0671	662	.08026	164.	33789	23.7877
4	7.009	FM	0.0701	1474	.64368	350.	36484	52.9821
5	7.732	MF	0.0647	37	.62754	9.	69558	1.3519
6	7.814	FM	0.0726	75.	.12651	17.	25028	2.6992

Totals: 2783.28625 671.00428

## 6.5- HOAt in THF



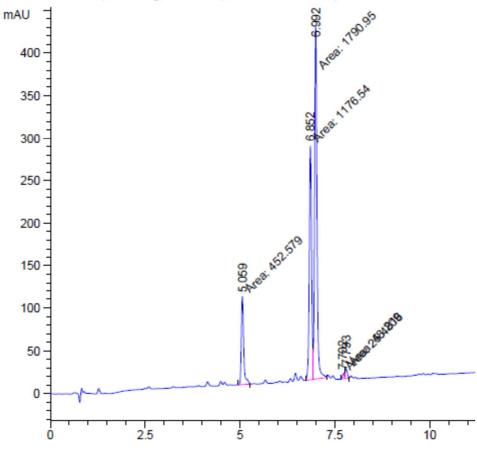


Peak	eak RetTime Type		Width	Area		Height			
#	[min]		[min]	mAU	*s	[mAU	]	8	
									- 1
1	5.124	MM	0.0738	296	.93680	67.	06278	12.242	2
2	6.923	FM	0.0671	642	.45160	159.	64384	26.487	1
3	7.064	MF	0.0709	1376	.44446	323.	46533	56.748	4
4	7.779	MF	0.0720	44	.94930	10.	39900	1.853	2
5	7.870	FM	0.0710	64	.73981	15.	20530	2.6693	1

Totals : 2425.52197 575.77626

## 6.6- HOAt in ACN



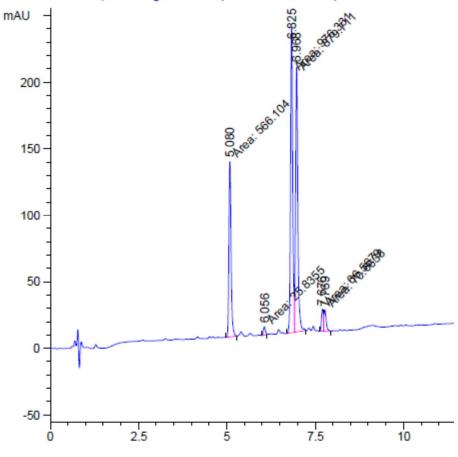


Peak	RetTime	Type	Width	Area	Height	Area
#	[min]		-	mAU *s	-	8
1	5.059	MM	0.0727	452.57864	103.81384	
2	6.852	MF	0.0710	1176.54395	276.24612	33.6755
3	6.992	FM	0.0714	1790.95044	417.95383	51.2614
4	7.702	MF	0.0608	25.48162	6.99078	0.7293
5	7.793	FM	0.0693	48.20901	11.59882	1.3799

Totals: 3493.76367 816.60339

# 6.7- OxymaPure in DMF



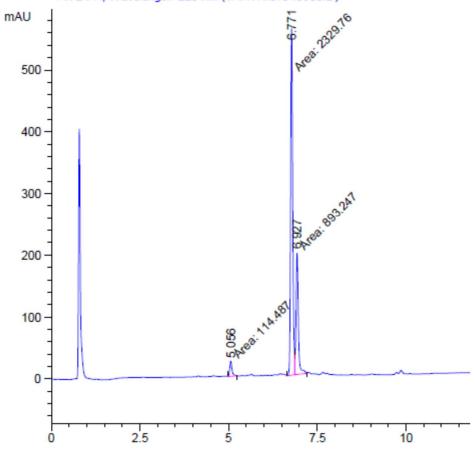


Peak	RetTime	Type	Width	Area	1	Hei	ght	Area	
#	[min]		[min]	mAU *	S	[mAU	]	8	
									- [
1	5.080	MM	0.0709	566.10	370	133.	08109	21.8978	3
2	6.056	MM	0.0675	25.83	3551	6.	38302	0.9994	1
3	6.825	MF	0.0706	976.33	3081	230.	47884	37.7660	)
4	6.968	FM	0.0731	879.71	143	200.	51784	34.028	5
5	7.676	MF	0.0660	66.56	786	16.	82010	2.5749	3
6	7.769	FM	0.0727	70.66	376	16.	19696	2.733	4

Totals: 2585.21306 603.47785

# 6.8- OxymaPure in THF



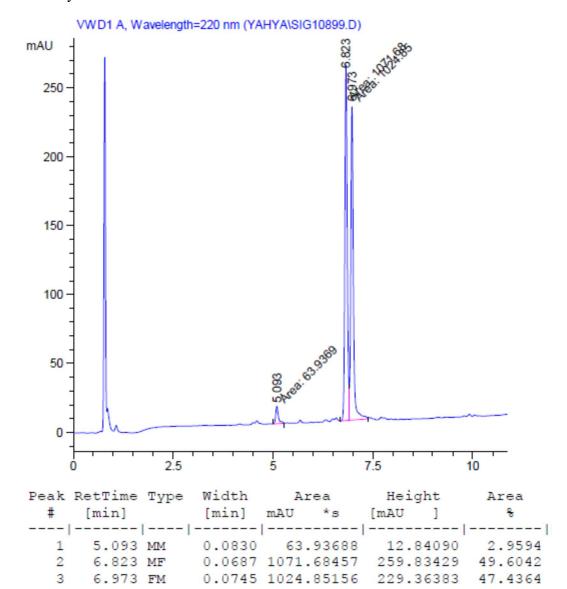


Peak	eak RetTime Type		Width	Area		Height		Area
#	[min]		[min]	mAU	*s	[mAU	]	8
1	5.056	MM	0.0771	114	.48705	24.7	74089	3.4303
2	6.771	MF	0.0689	2329	.76318	563.1	16357	69.8057
3	6.927	FM	0.0755	893	.24652	197.2	29671	26.7640

Totals: 3337.49675 785.20117

## 6.9- OxymaPure in ACN

Totals :



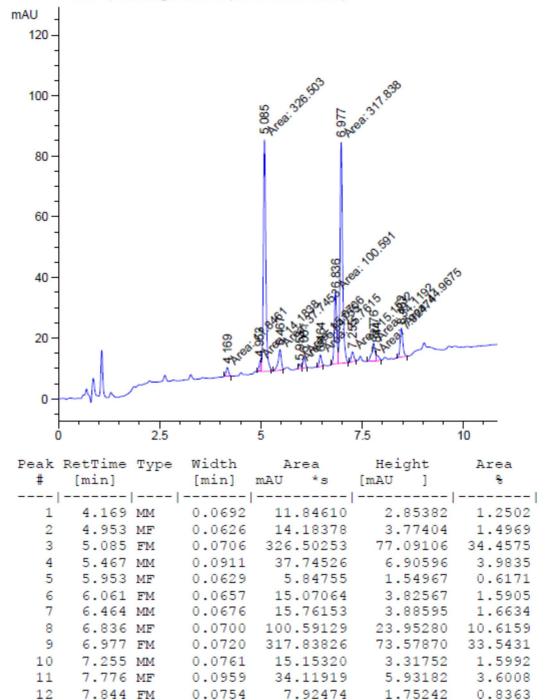
2160.47301 502.03902

#### 6.10- Oxyma-B in DMF

13

8.462 MM

VWD1 A, Wavelength=220 nm (YAHYA\SIG11182.D)



Totals: 947.55158 218.03075

44.96750

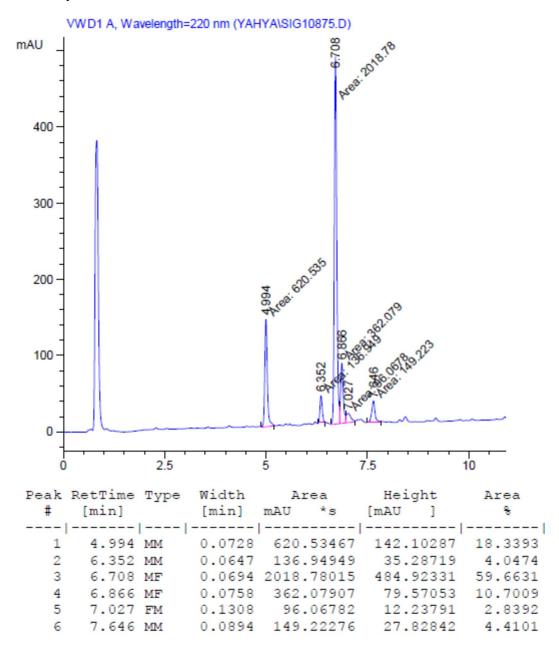
9.61133

4.7457

0.0780

#### 6.11- Oxyma-B in THF

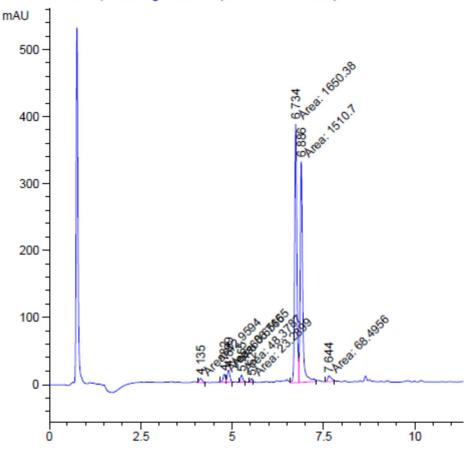
Totals :



3383.63396 781.95024

# 6.12- Oxyma-B in ACN

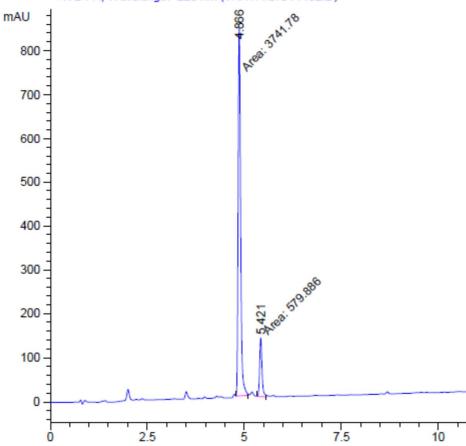
VWD1 A, Wavelength=220 nm (YAHYA\SIG10839.D)



Peak	RetTime	Type	Width	Area		Height		Area
#	[min]		[min]	mAU	*s	[mAU	]	8
1	4.135	MM	0.1092	42	.95938	6.	55540	1.2304
2	4.782	MF	0.0814	60	.65647	12.	41274	1.7372
3	4.899	FM	0.0792	86	.71651	18.	24301	2.4836
4	5.255	MM	0.0737	48	.37871	10.	94215	1.3856
5	5.528	MM	0.0626	23	.28985	6.	19931	0.6670
6	6.734	MF	0.0706	1650	.38013	389.	50598	47.2675
7	6.886	FM	0.0758	1510	.69861	332.	12930	43.2670
8	7.644	MM	0.1268	68	.49562	9.	00487	1.9617

Totals: 3491.57527 784.99276





6.14- des-Aib-Ile $^{72}$ -Asn mixed with Aib-modified ACP decapeptide obtained from HOBt and THF

