

Supporting Information

A Highly Efficient Asymmetric Michael Addition of α , α -Disubstituted Aldehydes to Maleimides Catalyzed by Primary Amine Thiourea Salt

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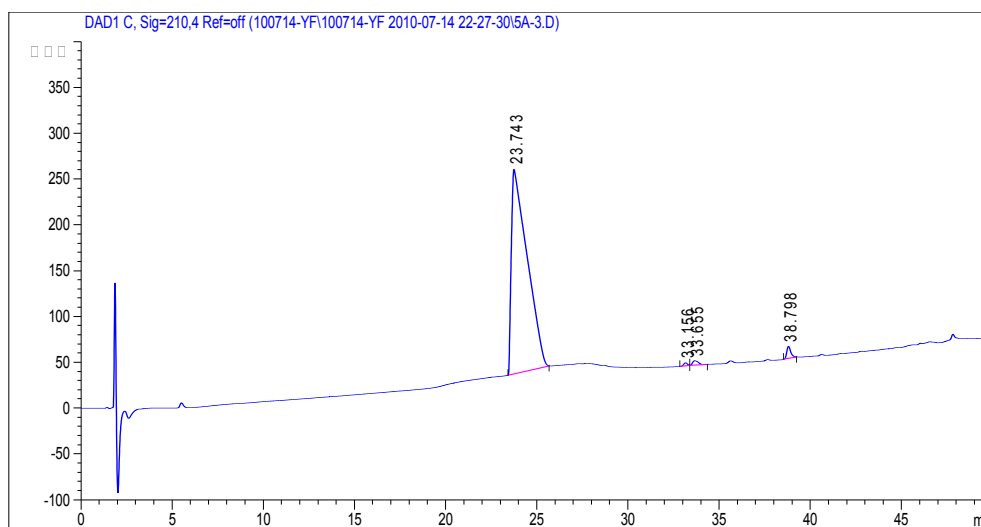
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A: HPLC and NMR Analysis of Catalysts 5 and 6.

1) HPLC Analysis of the Purity of Catalysts 5 and 6.

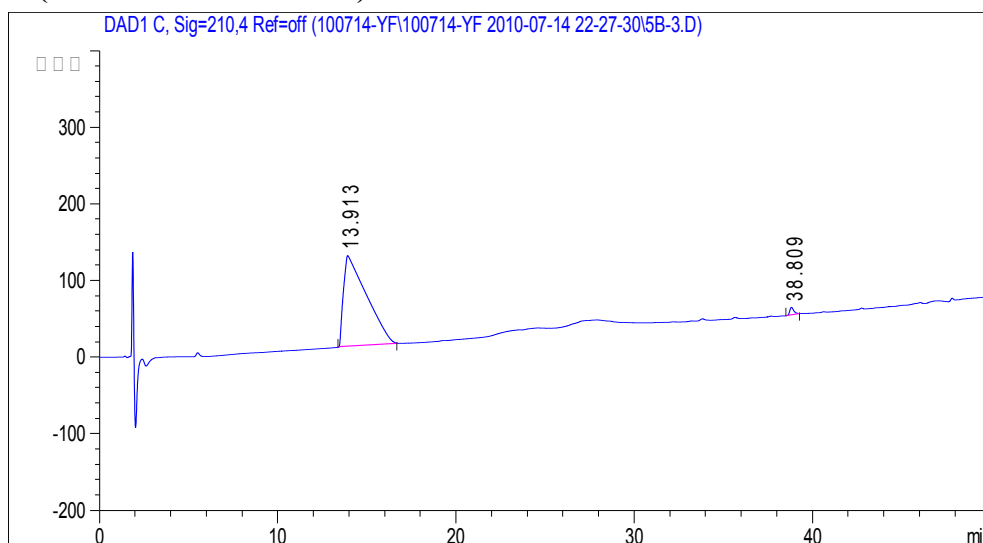
The purity of the catalysts was performed on an Agilent 1200 HPLC system, which comprised a binary pump, a degasser, an autosampler an automatic thermostatic column compartment and a diode array detector (DAD). XTerra MS C18 column (5 μ m, 150mm \times 2.1mm i.d., Waters) was employed, the column temperatures were maintained at 30°C and the flow rate was set at 0.2mL/min during all of the analysis. Mobile phase A: 0.1% formic acid solution, mobile phase B: 0.1% formic acid in methanol. Linear gradient eluent from 40% B to 100% B in 45min and kept for 5 min.

N-((1*R*,2*R*)-2-amino-1,2-diphenylethyl)-3,5-bis (trifluoromethyl) benzenesulfonamide (**5a**) (UV detection: 210 nm)



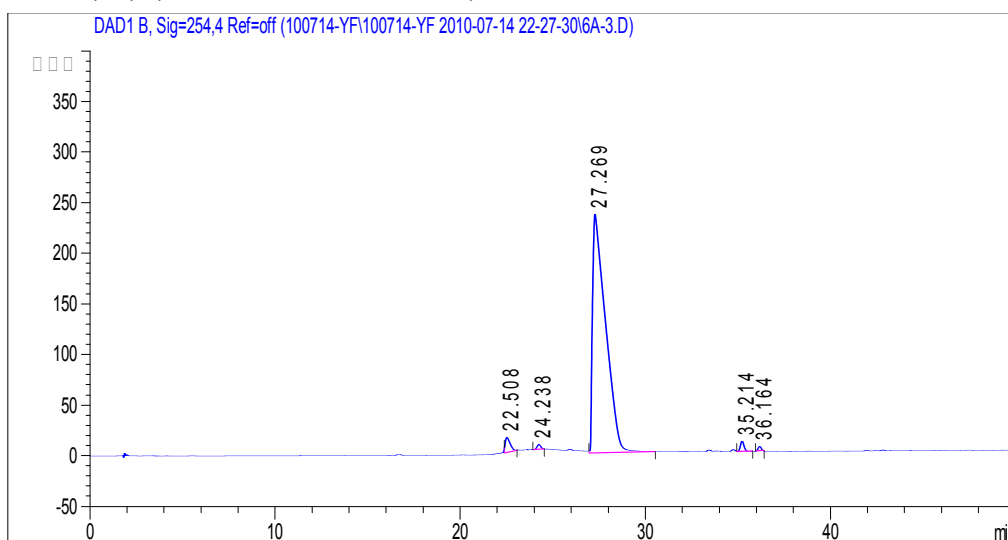
#	Time	Area	Height	Width	Symmetry	Area %
1	23.743	13484.3	223.4	0.8101	0.195	97.209
2	33.156	54.5	3.6	0.2366	1.011	0.393
3	33.655	116.7	5.1	0.3735	0.613	0.841
4	38.798	216	13.6	0.2394	0.673	1.557

***N*-((1*R*,2*R*)-2-aminocyclohexyl)-3,5-bis (trifluoromethyl)benzenesulfonamide
 (5b) (UV detection: 210 nm)**



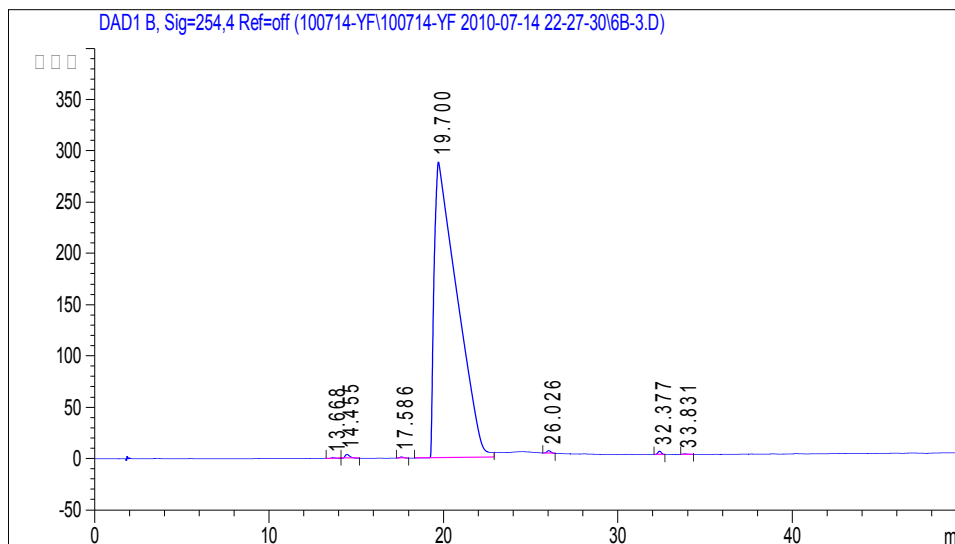
#	Time	Area	Height	Width	Symmetry	Area %
1	13.913	10646.2	119.1	1.1908	0.213	98.456
2	38.809	167	10.1	0.2474	0.66	1.544

**1-(3,5-bis(trifluoromethyl)phenyl)-3-((1*R*,2*R*)-2-amino-1,2-diphenylethyl)
 thiourea (6a) (UV detection: 254 nm)**



#	Time	Area	Height	Width	Symmetry	Area %
1	22.508	282.2	15	0.3142	0.44	2.331
2	24.238	72	4.8	0.2485	1.006	0.595
3	27.269	11560.3	235.7	0.7003	0.224	95.468
4	35.214	144.5	10.3	0.2191	0.826	1.193
5	36.164	50	4.3	0.1923	1.192	0.413

**1-(3,5-bis(trifluoromethyl)phenyl)-3-((1*R*,2*R*)-2-aminocyclohexyl)thiourea
(6b) (UV detection: 254 nm)**

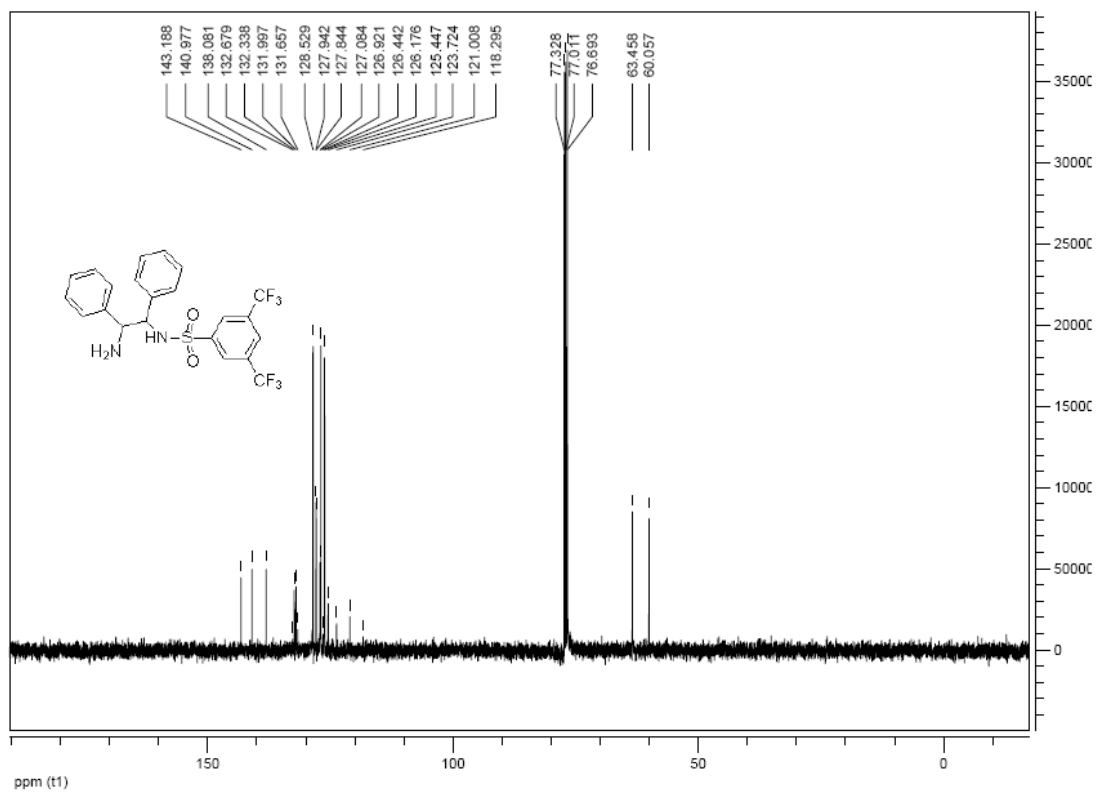
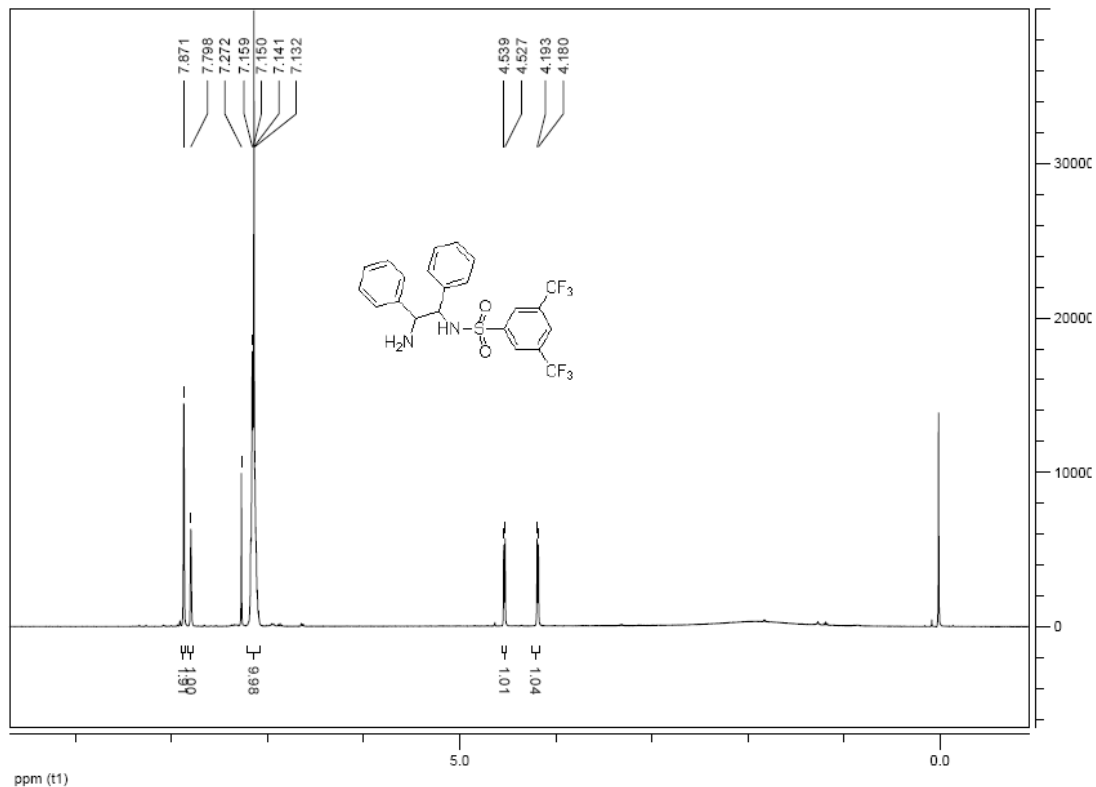


#	Time	Area	Height	Width	Symmetry	Area %
1	13.668	9.4	5.3E-1	0.2867	0.95	0.036
2	14.455	76.2	3.9	0.3027	0.559	0.288
3	17.586	18.9	1.2	0.2335	0.806	0.072
4	19.7	26225.5	288.5	1.2165	0.213	99.203
5	26.026	43.3	2.6	0.2758	0.856	0.164
6	32.377	45.9	3.4	0.2272	0.931	0.174
7	33.831	16.9	6.5E-1	0.4368	0.518	0.064

2) ^1H NMR and ^{13}C NMR Analysis of the Organocatalysts 5 and 6.

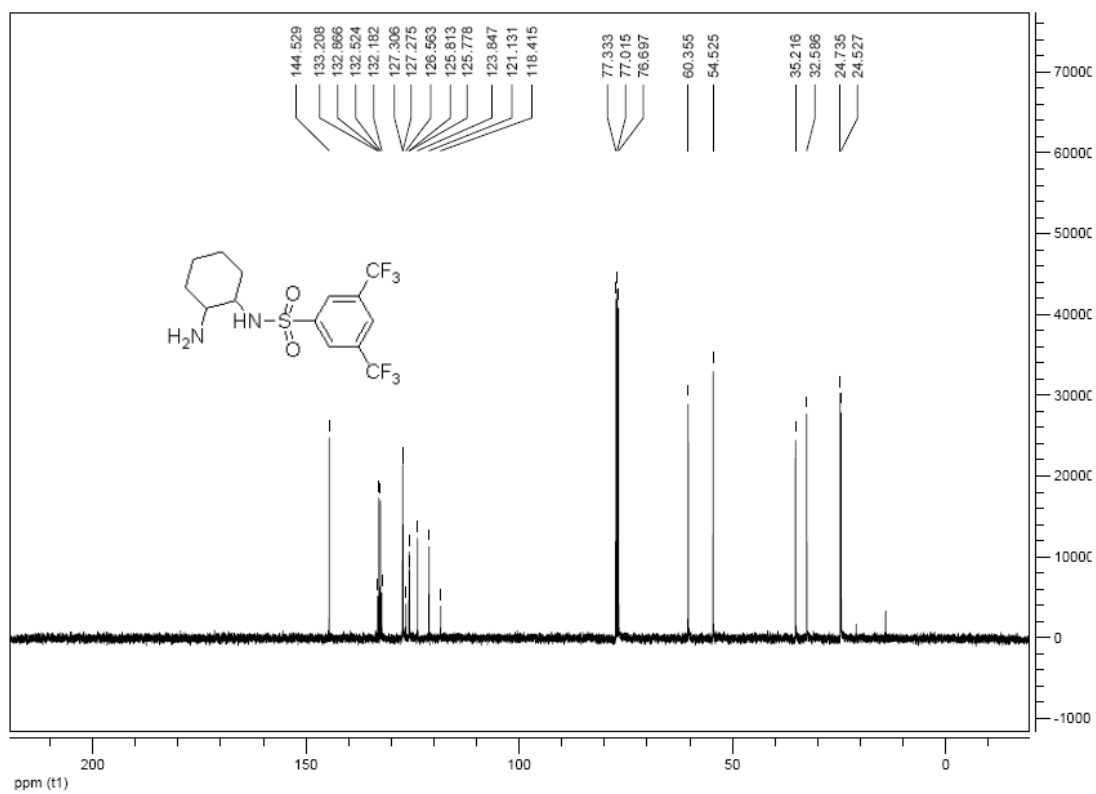
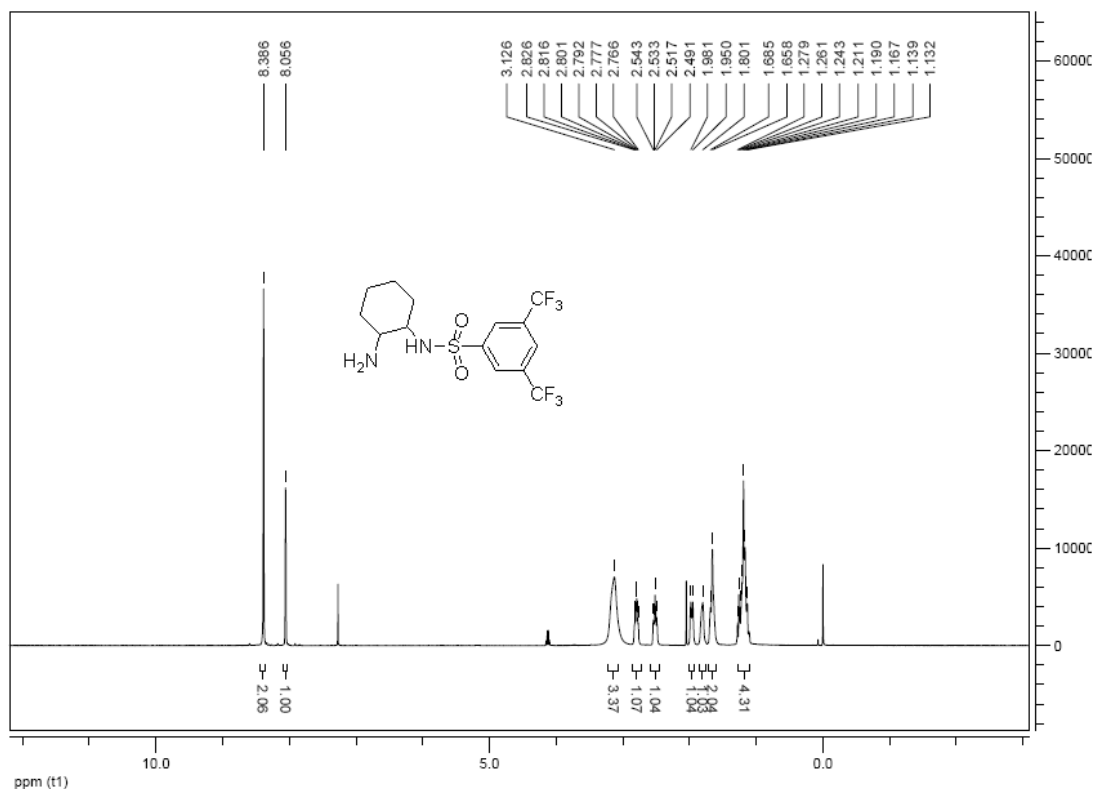
N-((1*R*,2*R*)-2-amino-1,2-diphenylethyl)-3,5-bis (trifluoromethyl)

benzenesulfonamide (5a)

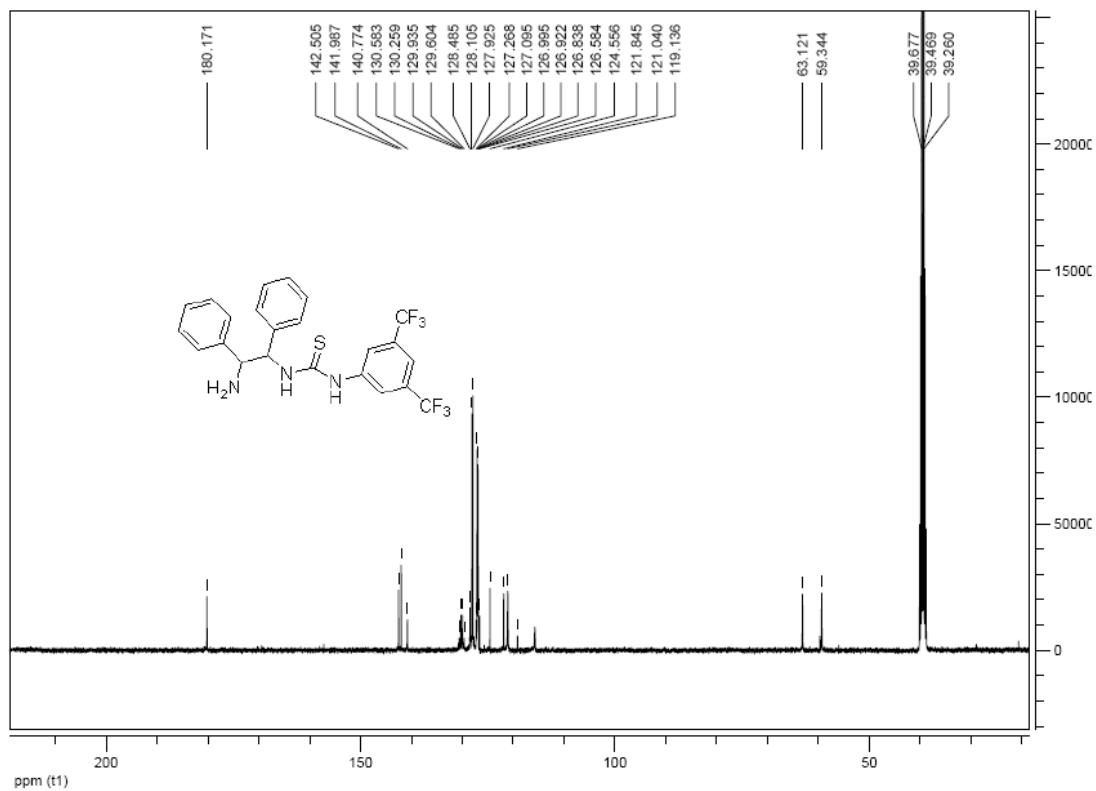
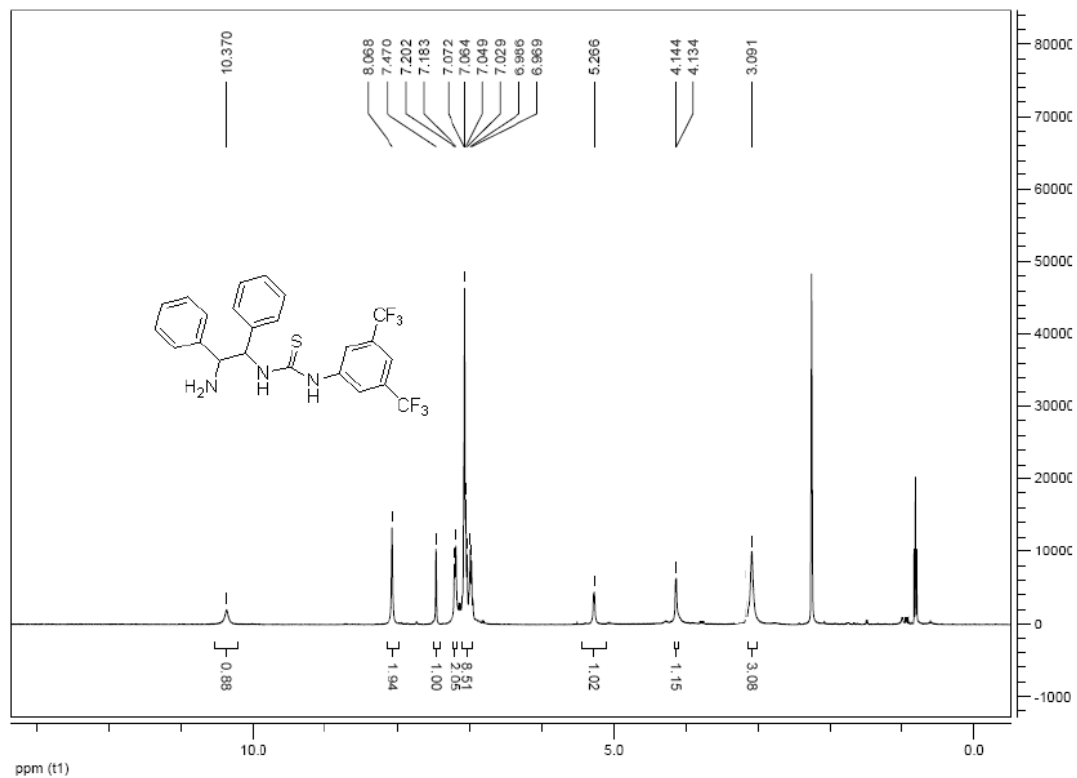


***N*-((1*R*,2*R*)-2-aminocyclohexyl)-3,5-bis (trifluoromethyl)benzenesulfonamide**

(5b)

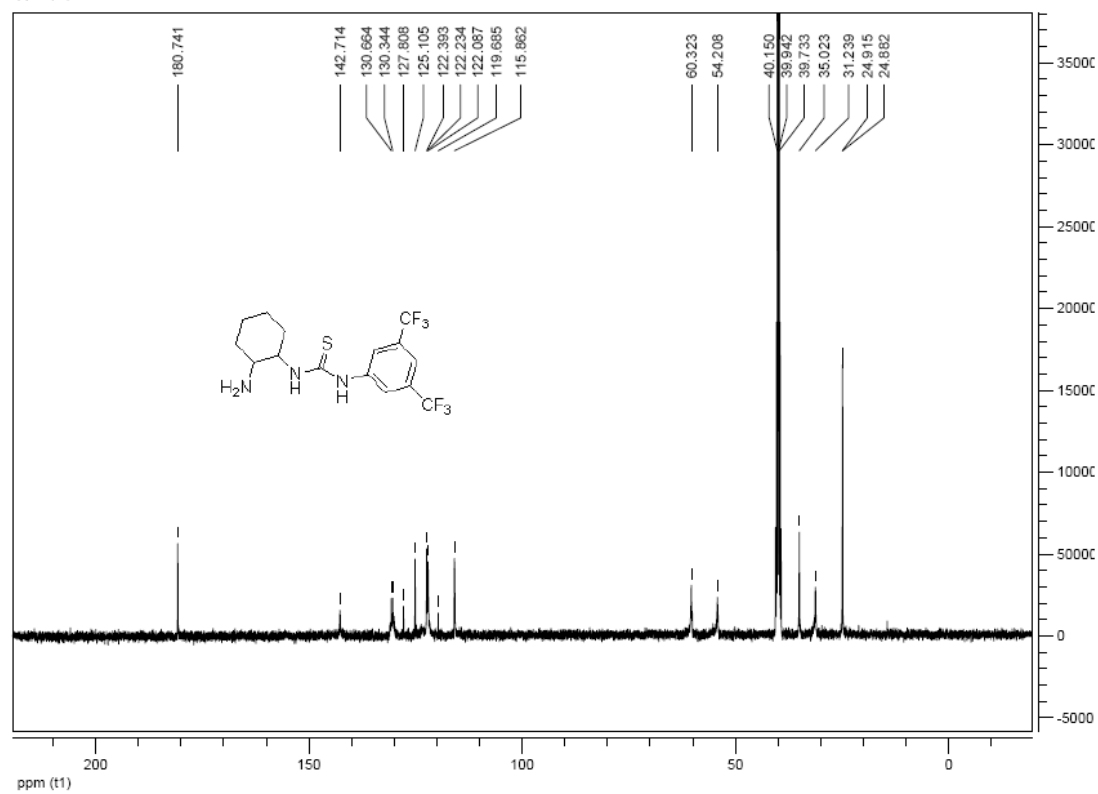
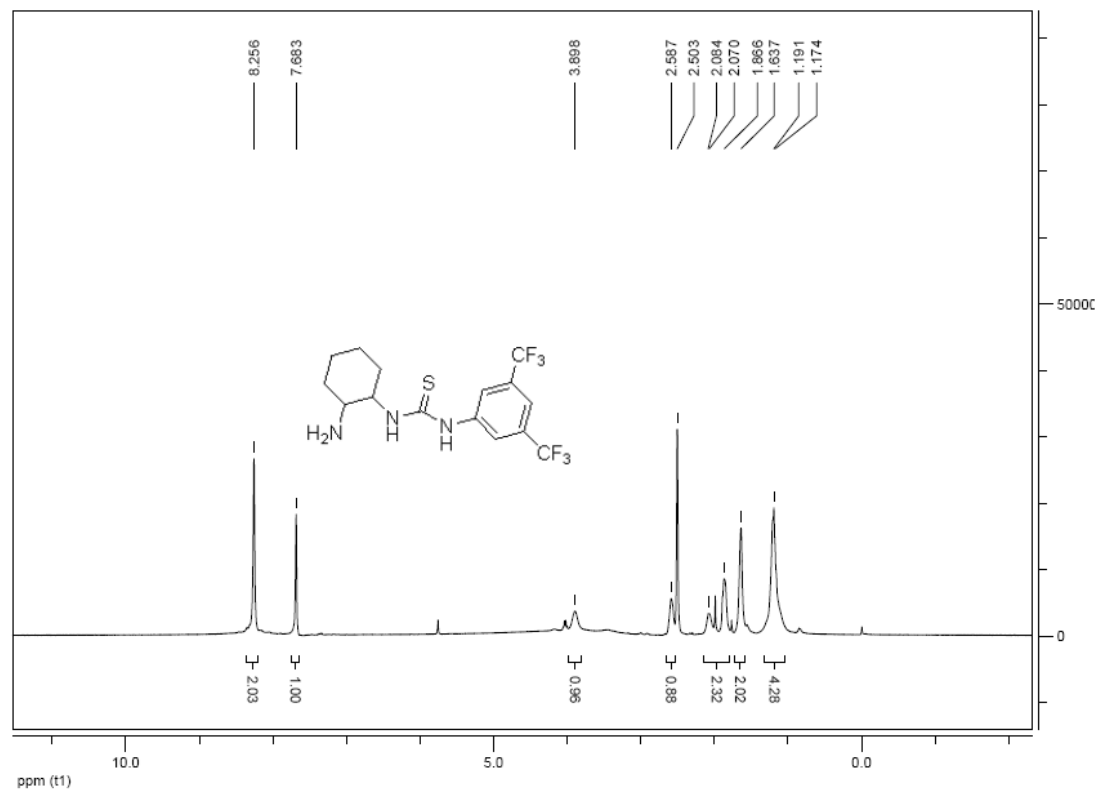


**1-(3,5-bis(trifluoromethyl)phenyl)-3-((1*R*,2*R*)-2-amino-1,2-diphenylethyl)
thiourea (6a)**



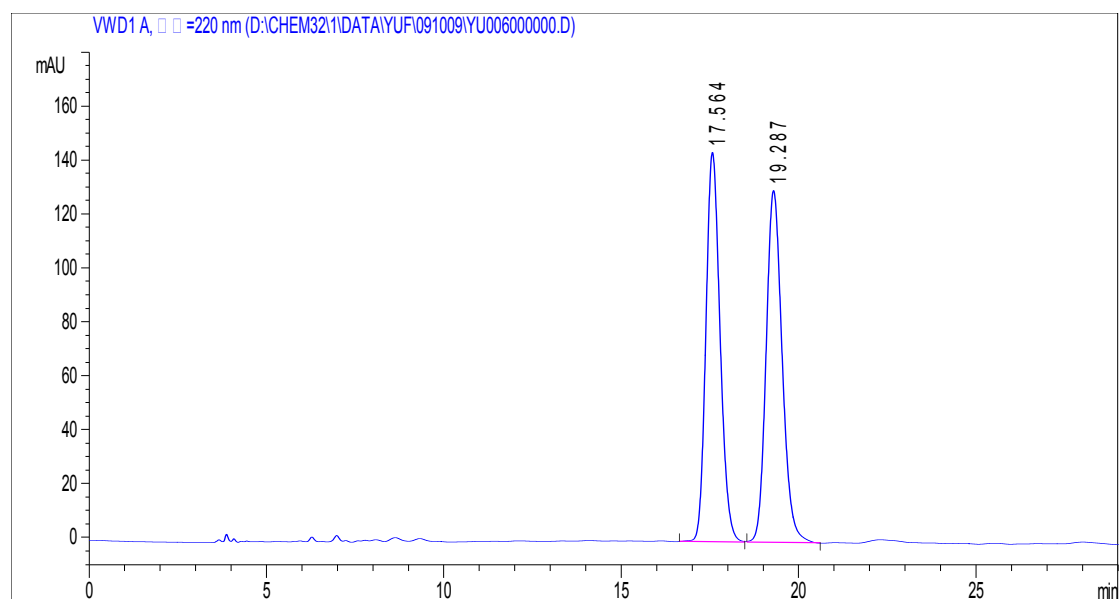
1-(3,5-bis(trifluoromethyl)phenyl)-3-((1R,2R)-2-aminocyclohexyl)thiourea

(6b)

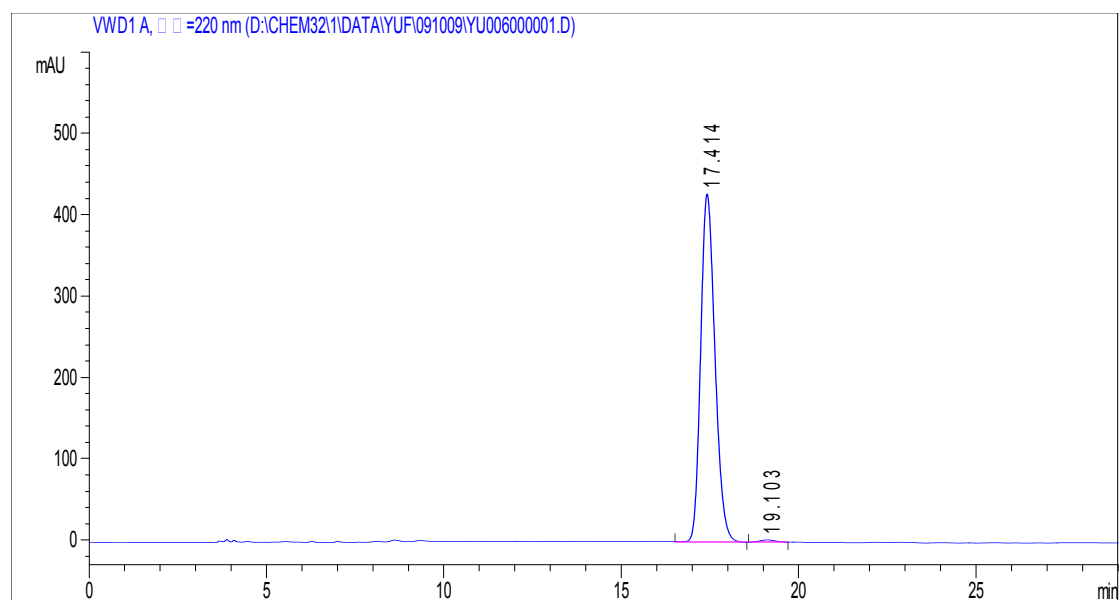


B: HPLC Analysis of Michael Addition Products

2-(2,5-dioxo-1-phenylpyrrolidin-3-yl)-2-methylpropanal (3a)

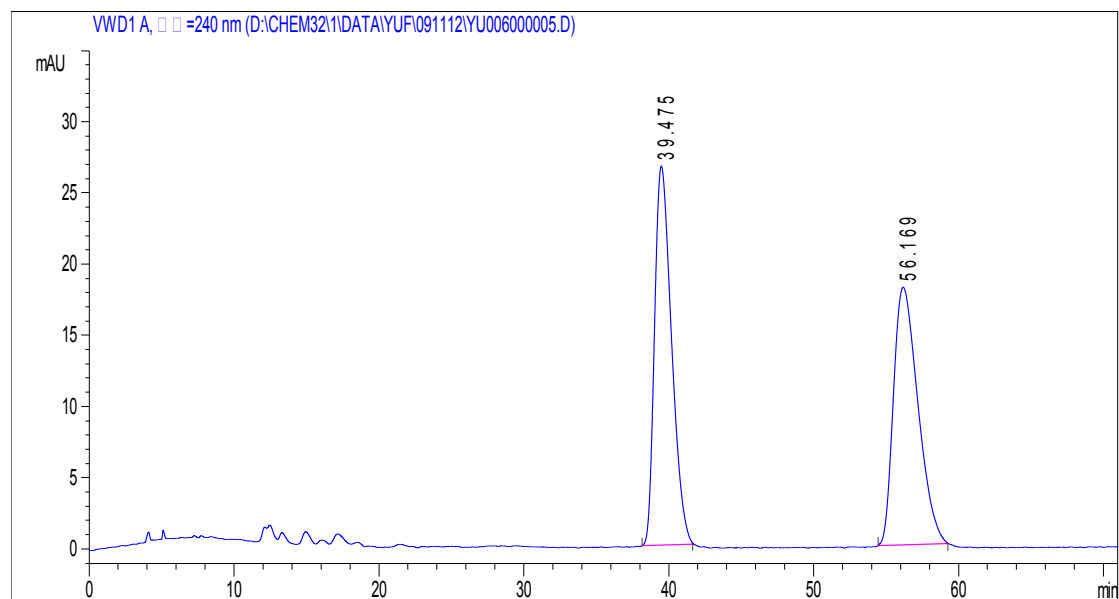


#	Time	Area	Height	Width	Symmetry	Area %
1	17.564	4013	144.5	0.4304	0.818	49.419
2	19.287	4107.4	130.6	0.4849	0.816	50.581

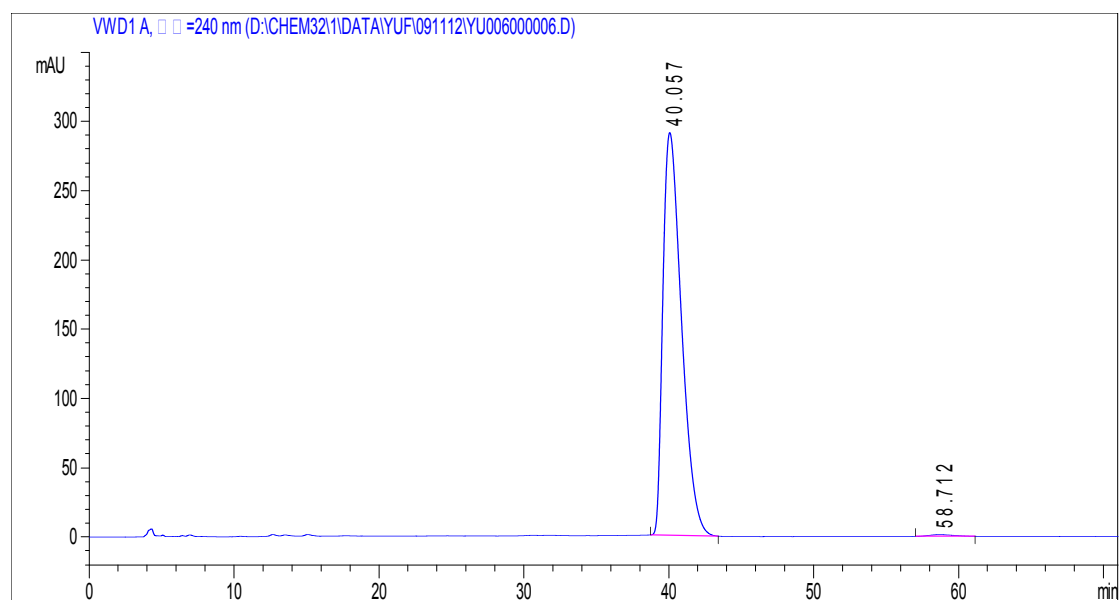


#	Time	Area	Height	Width	Symmetry	Area %
1	17.414	12043.6	428.2	0.4688	0.79	99.243
2	19.103	91.9	2.9	0.5274	0.916	0.757

2-(2, 5-dioxo-1-p-tolylpyrrolidin-3-yl)-2-methylpropanal (3b)

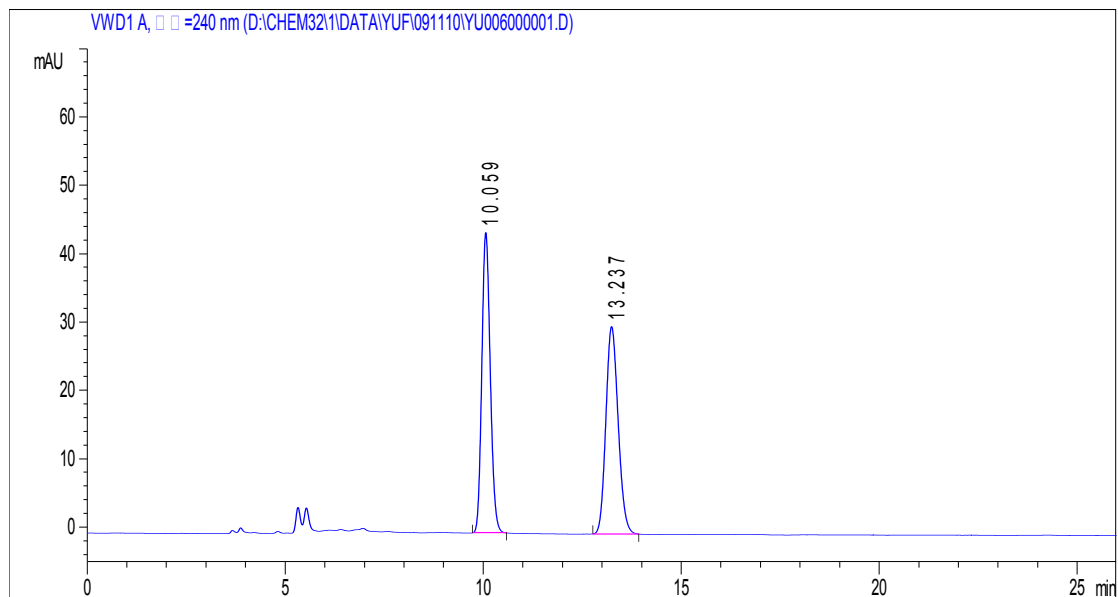


#	Time	Area	Height	Width	Symmetry	Area %
1	39.475	2168.6	26.6	1.2117	0.63	49.884
2	56.169	2178.7	18.3	1.9891	0.628	50.116

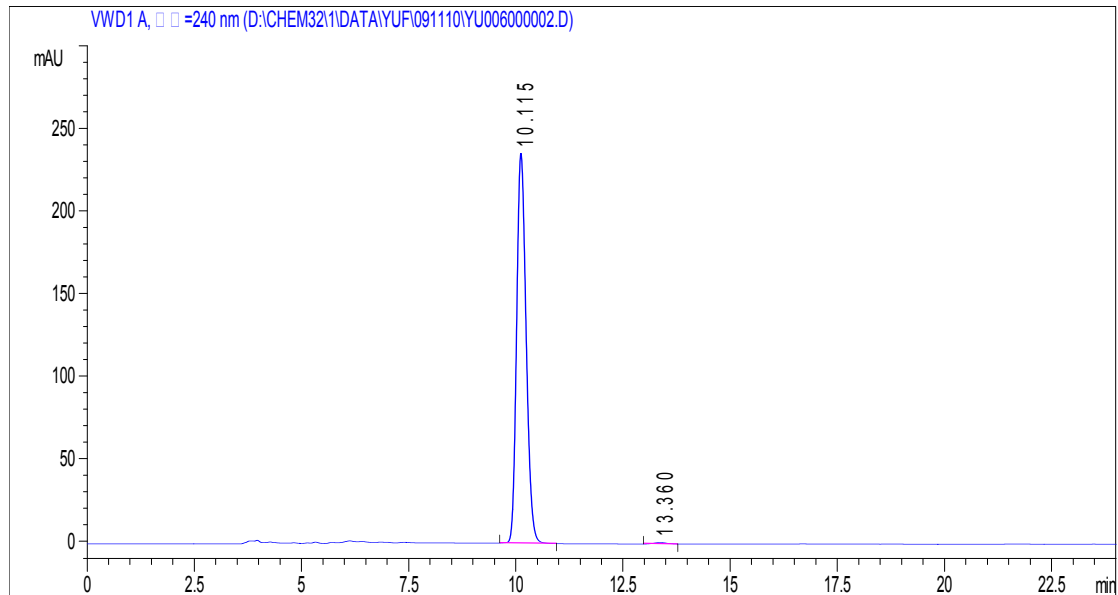


#	Time	Area	Height	Width	Symmetry	Area %
1	40.057	25956.6	290.7	1.3649	0.546	99.423
2	58.712	150.6	1.3	1.974	0.807	0.577

2-(2,5-dioxo-1-(3-(trifluoromethyl)phenyl)pyrrolidin-3-yl)-2-methylpropanal (3c)

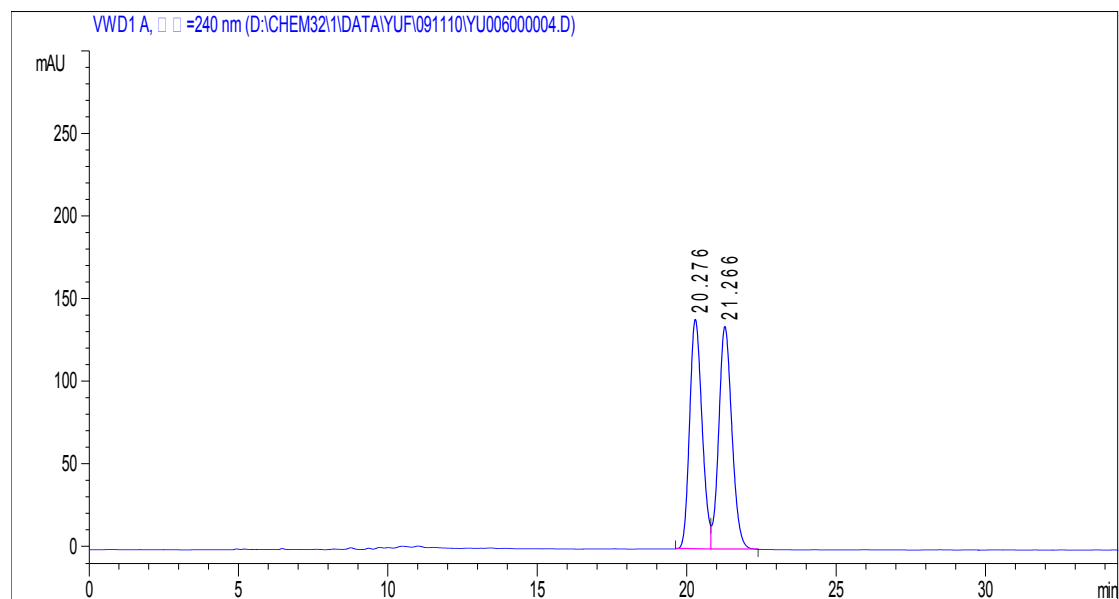


#	Time	Area	Height	Width	Symmetry	Area %
1	10.059	654	44	0.2303	0.836	49.932
2	13.237	655.8	30.4	0.3348	0.836	50.068

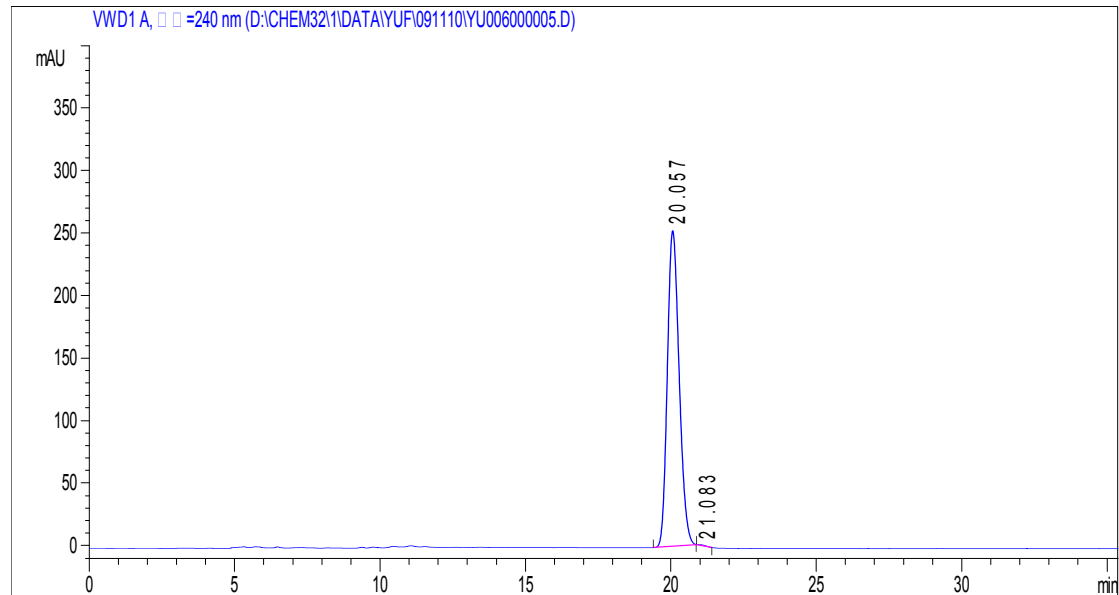


#	Time	Area	Height	Width	Symmetry	Area %
1	10.115	3642.5	235.8	0.2414	0.784	99.525
2	13.36	17.4	8.6E-1	0.3364	0.858	0.475

2-(1-(4-chlorophenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (3d)

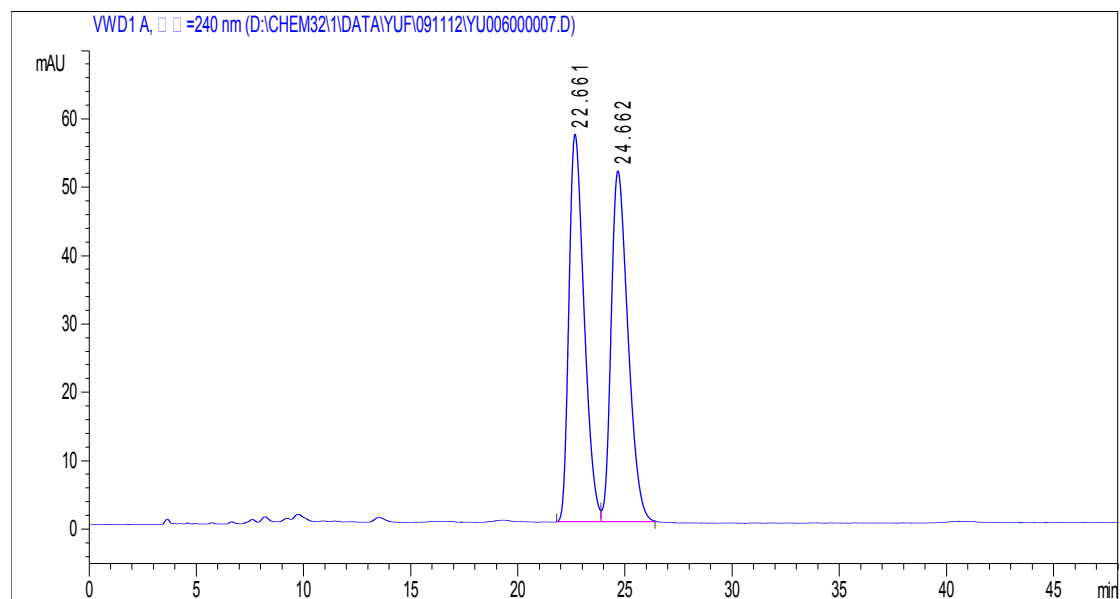


#	Time	Area	Height	Width	Symmetry	Area %
1	20.276	4027.8	139	0.4502	0.808	49.474
2	21.266	4113.5	134.8	0.4697	0.828	50.526

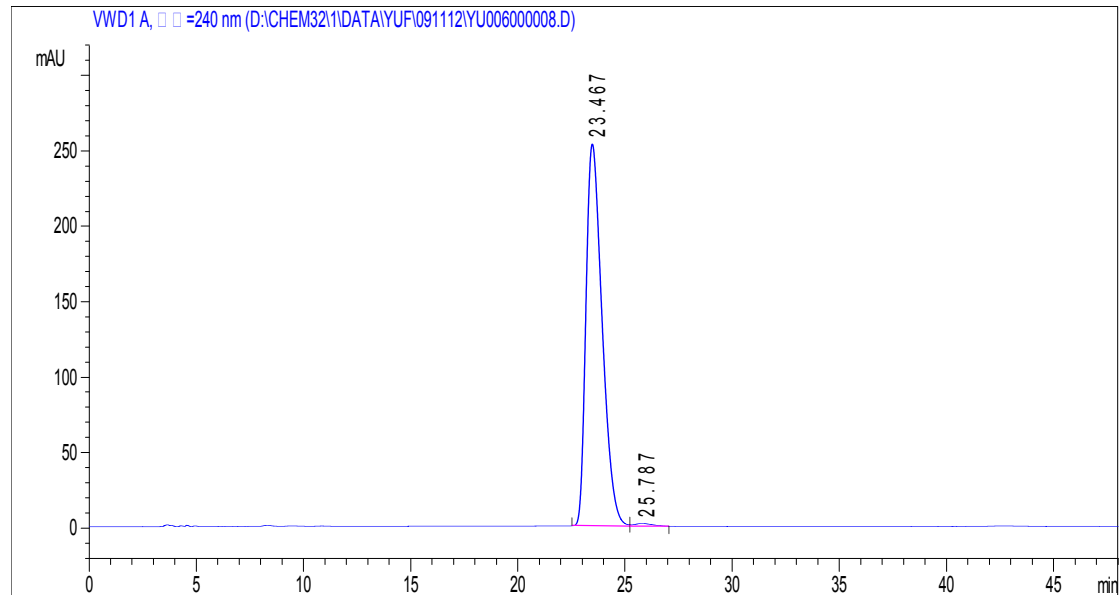


#	Time	Area	Height	Width	Symmetry	Area %
1	20.057	7129.7	252.6	0.4703	0.799	99.750
2	21.083	17.8	8.7E-1	0.3407	0.116	0.250

2-(1-(4-bromophenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (3e)

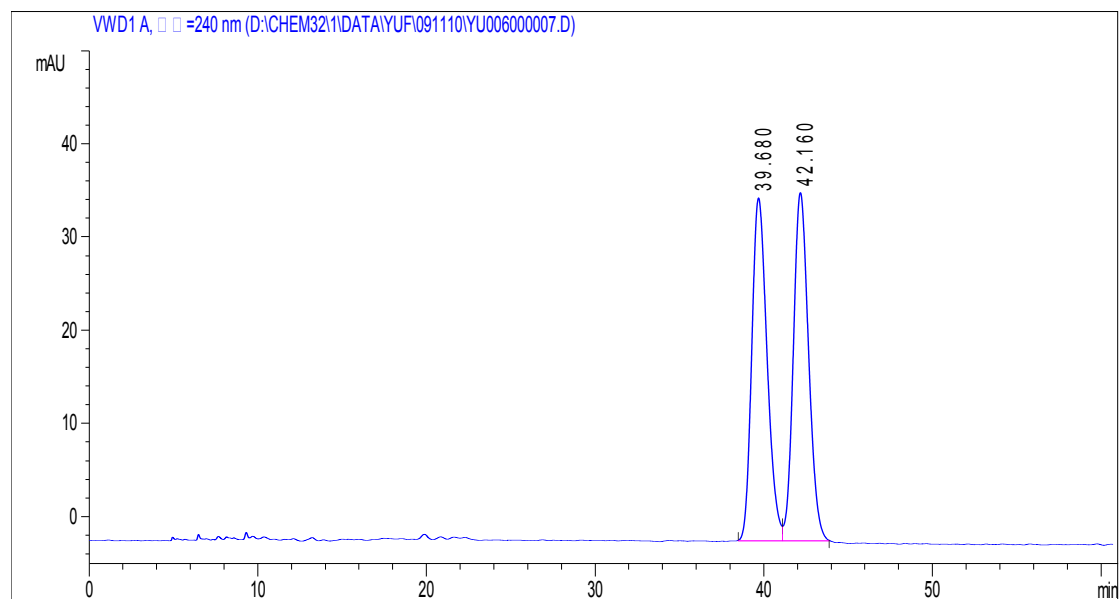


#	Time	Area	Height	Width	Symmetry	Area %
1	22.661	2793.5	56.7	0.7668	0.63	49.914
2	24.662	2803.2	51.3	0.8437	0.632	50.086

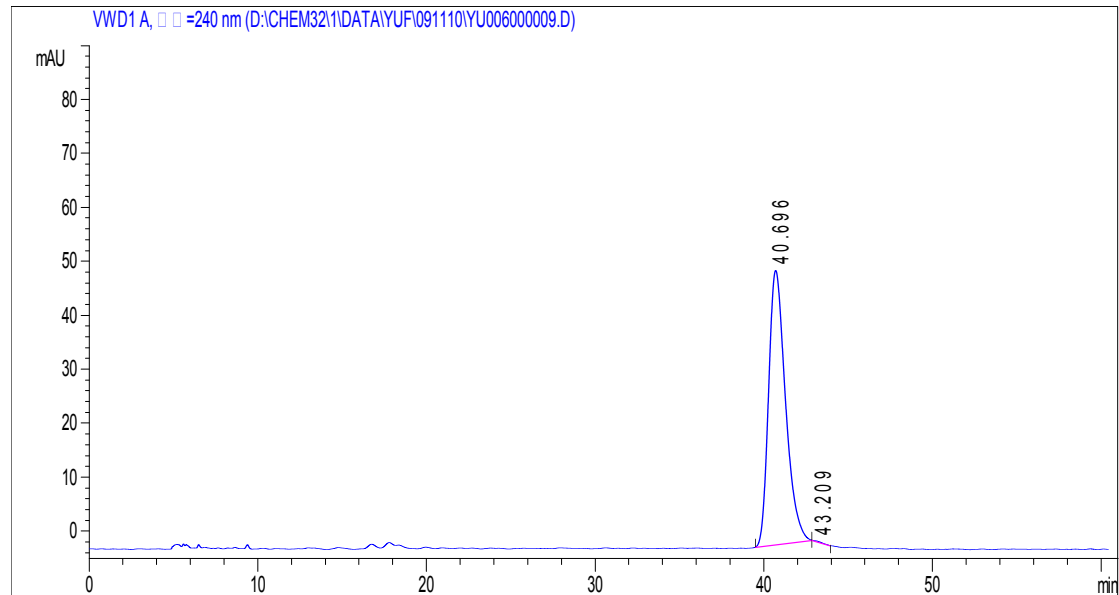


#	Time	Area	Height	Width	Symmetry	Area %
1	23.467	13202.1	253.1	0.8694	0.626	99.550
2	25.839	59.7	1.2	0.809	0.541	0.450

2-(1-(4-methoxyphenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (3f)

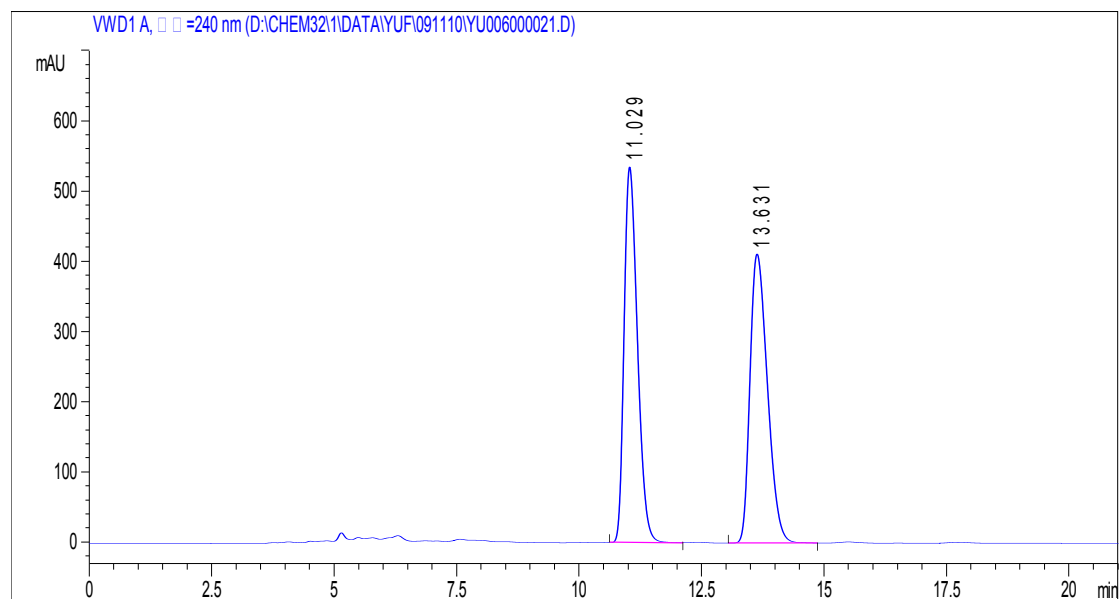


#	Time	Area	Height	Width	Symmetry	Area %
1	39.68	2324.8	36.8	0.973	0.755	49.734
2	42.16	2349.7	37.4	0.969	0.822	50.266

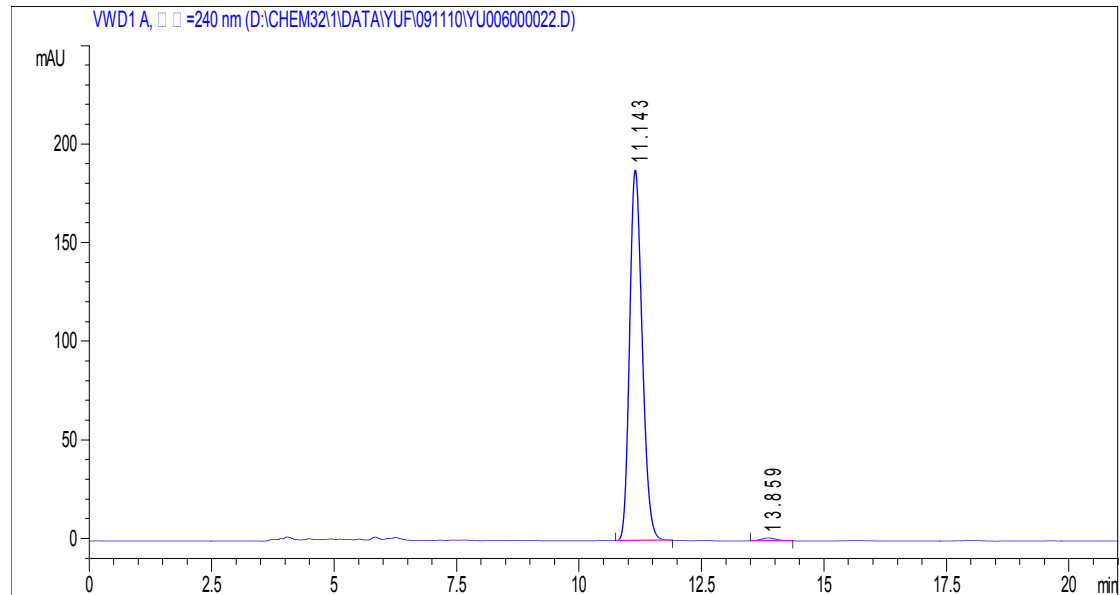


#	Time	Area	Height	Width	Symmetry	Area %
1	40.696	3556.1	50.9	1.164	0.695	99.657
2	43.209	12.2	2.8E-1	0.7413	0	0.343

2-(2,5-dioxo-1-phenylpyrrolidin-3-yl)-2-ethylbutanal (3g)

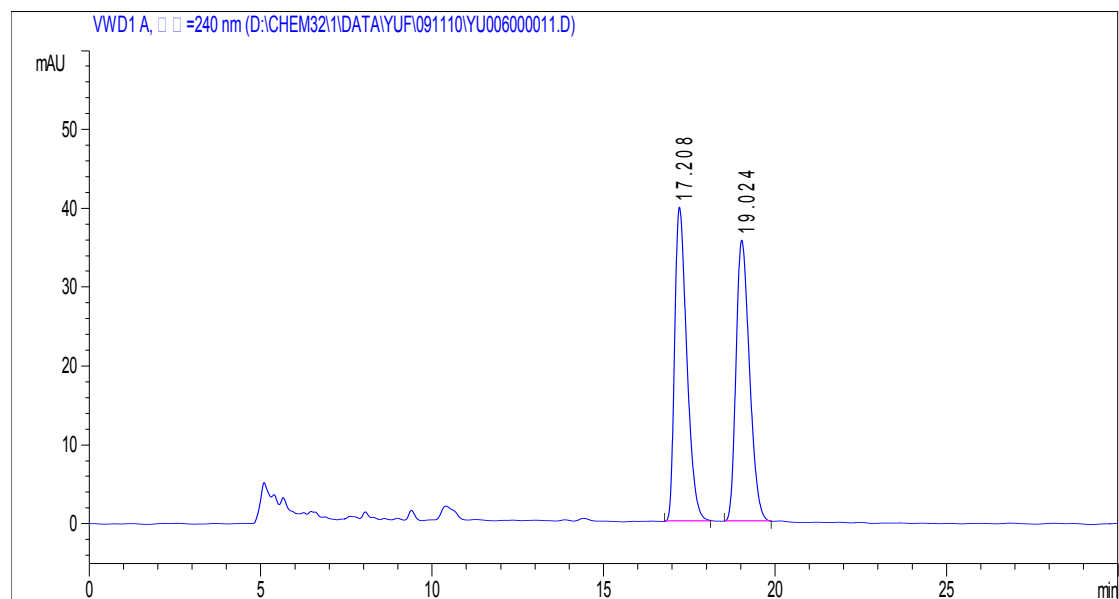


#	Time	Area	Height	Width	Symmetry	Area %
1	11.029	10152.1	535.3	0.2955	0.668	49.919
2	13.631	10185	411.6	0.3877	0.65	50.081

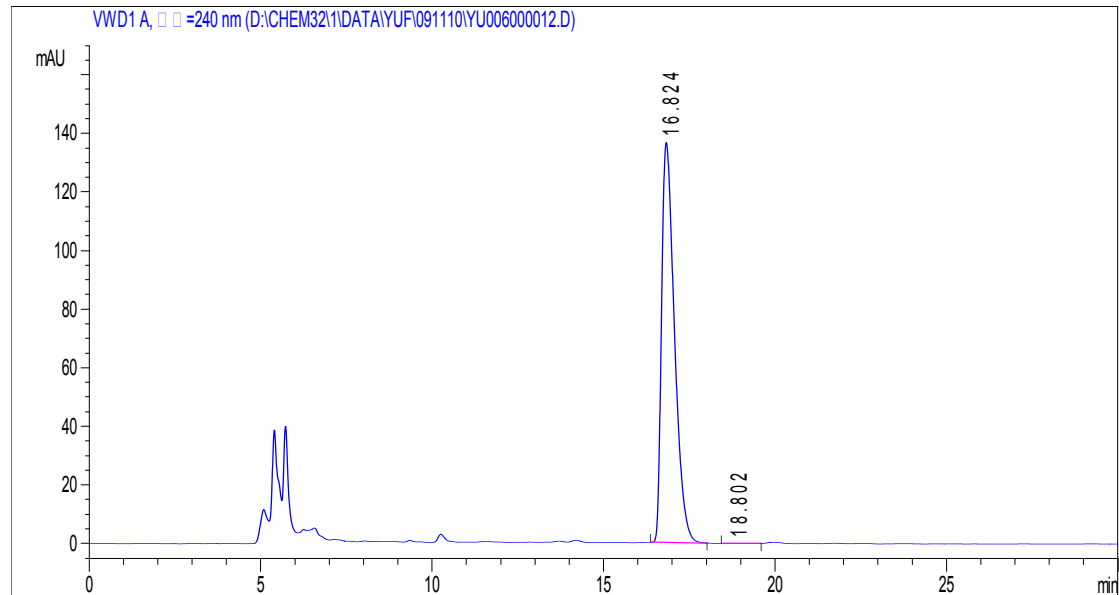


#	Time	Area	Height	Width	Symmetry	Area %
1	11.143	3361	187.9	0.2785	0.771	99.043
2	13.859	32.5	1.5	0.3636	0.876	0.957

2-(1-benzyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (3h)

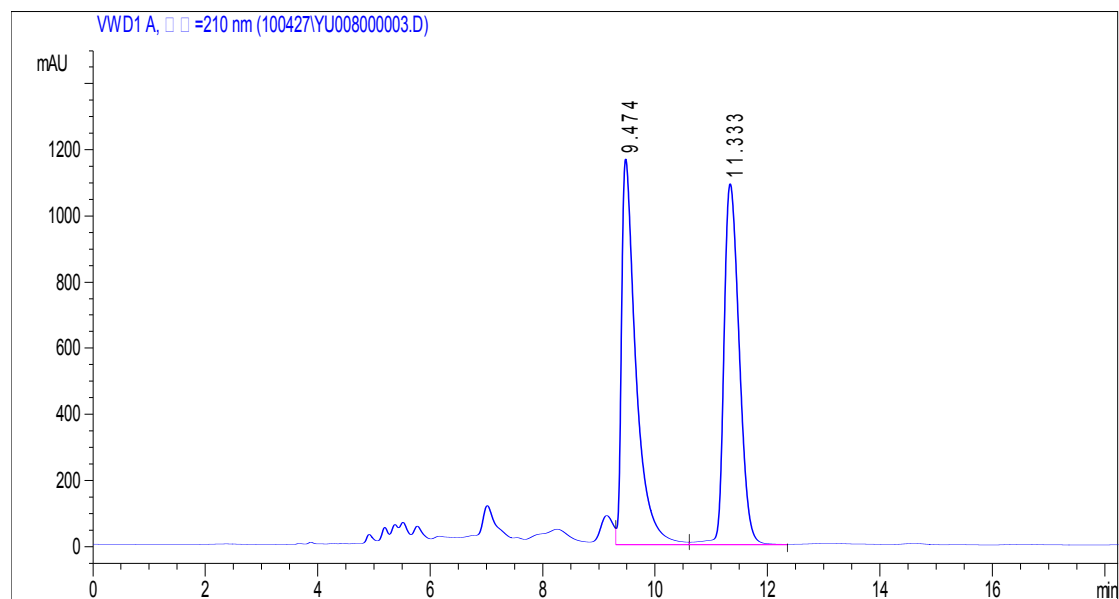


#	Time	Area	Height	Width	Symmetry	Area %
1	17.208	983.7	39.8	0.3769	0.608	50.230
2	19.024	974.7	35.6	0.4254	0.693	49.770

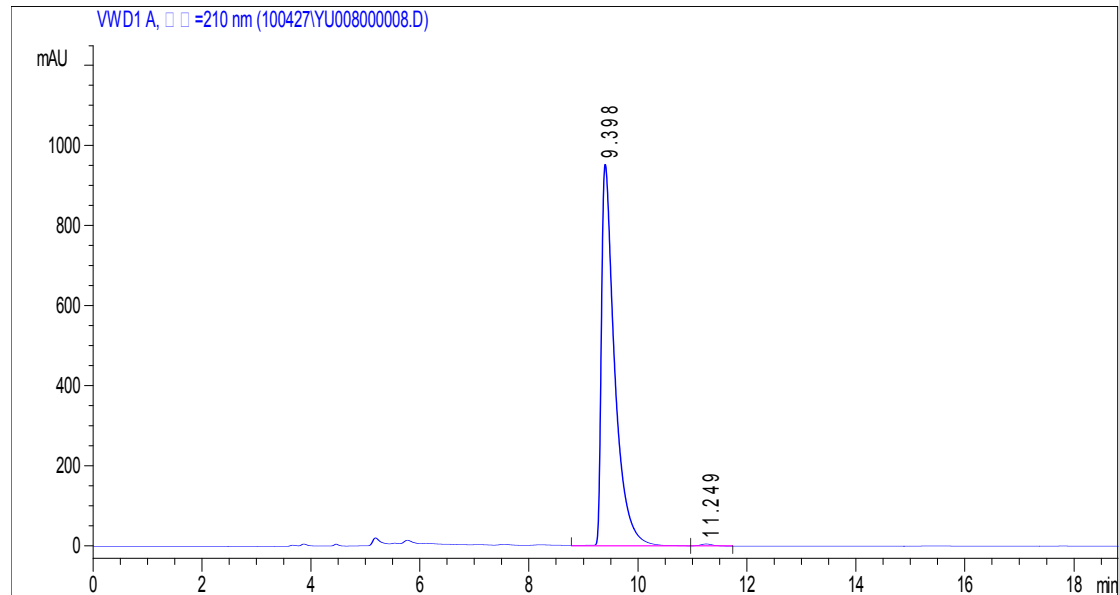


#	Time	Area	Height	Width	Symmetry	Area %
1	16.824	3575.3	136.6	0.4028	0.536	99.784
2	18.802	7.7	1.9E-1	0.6765	0.388	0.216

2-(2,5-dioxo-1-propylpyrrolidin-3-yl)-2-methylpropanal (3i)

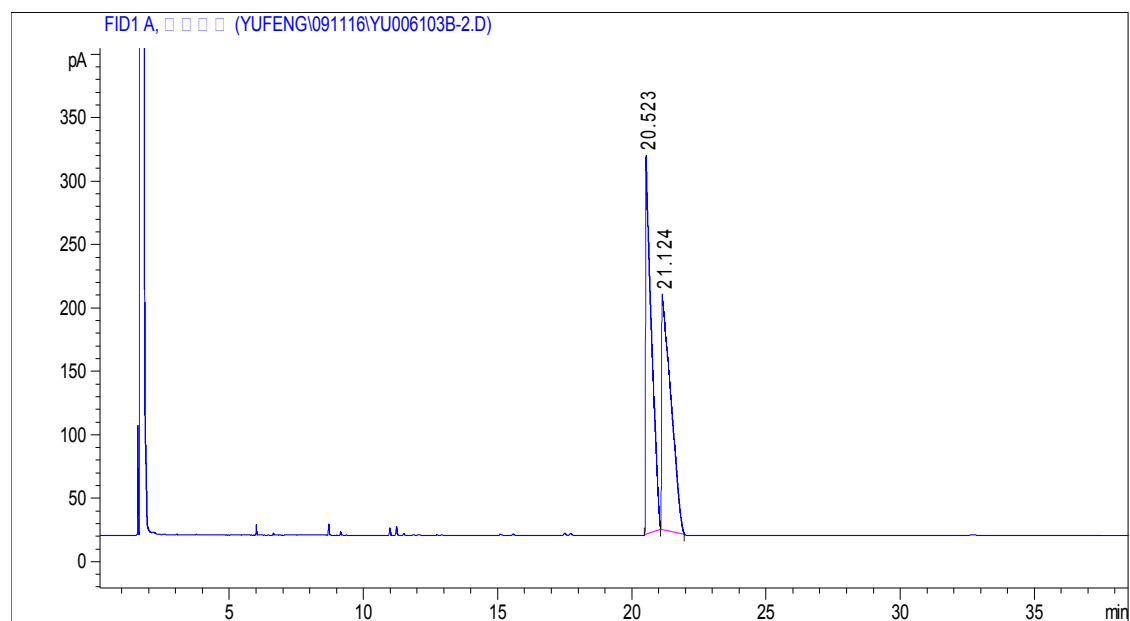


#	Time	Area	Height	Width	Symmetry	Area %
1	9.474	20770.9	1167.4	0.2614	0.368	50.135
2	11.333	20658.9	1092.6	0.2992	0.662	49.865

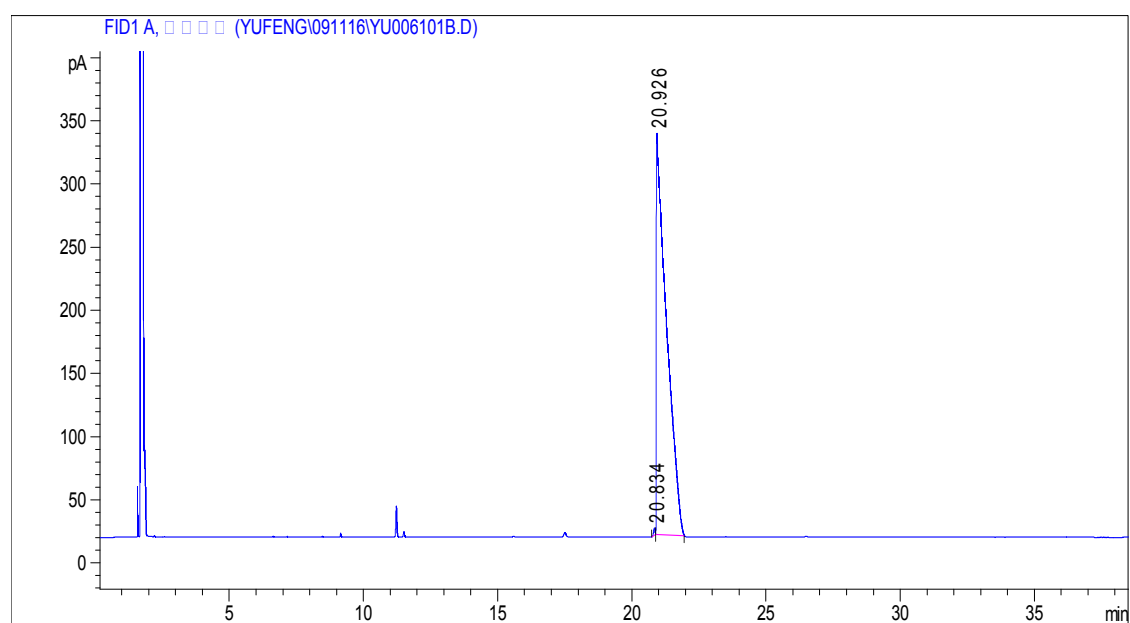


#	Time	Area	Height	Width	Symmetry	Area %
1	9.398	16177.7	954.2	0.2473	0.373	99.450
2	11.249	89.5	5.5	0.247	0.903	0.550

2-(1-isopropyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (3j)

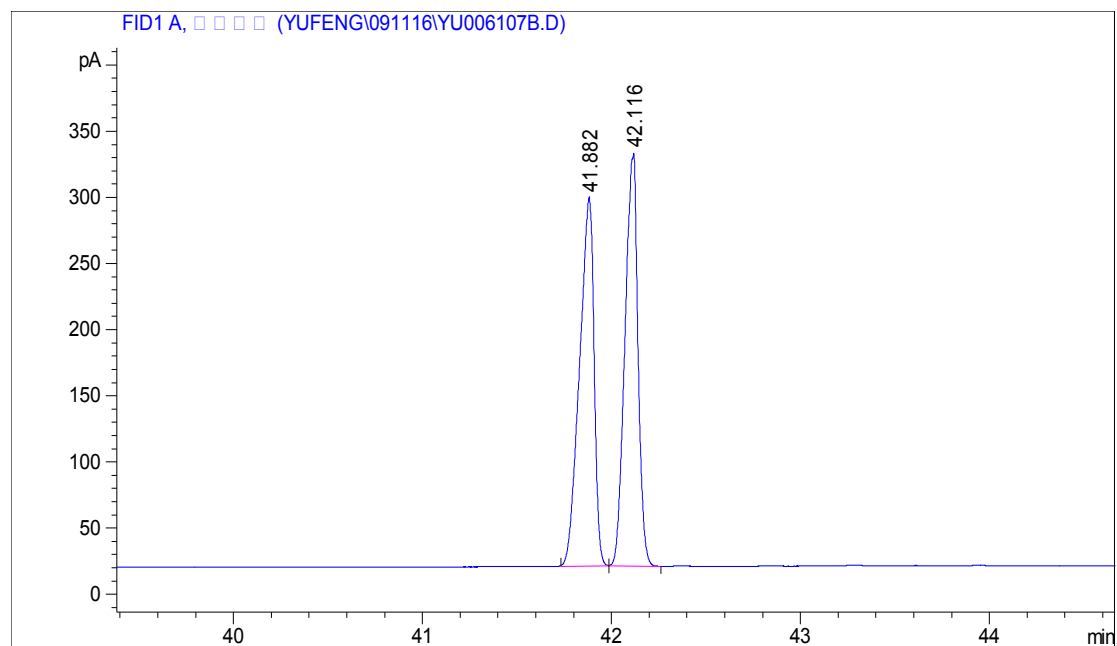


#	Time	Area	Height	Width	Symmetry	Area %
1	20.523	4496.2	298.6	0.1837	8.68E-2	50.289
2	21.124	4444.5	185.6	0.2894	6.81E-2	49.711

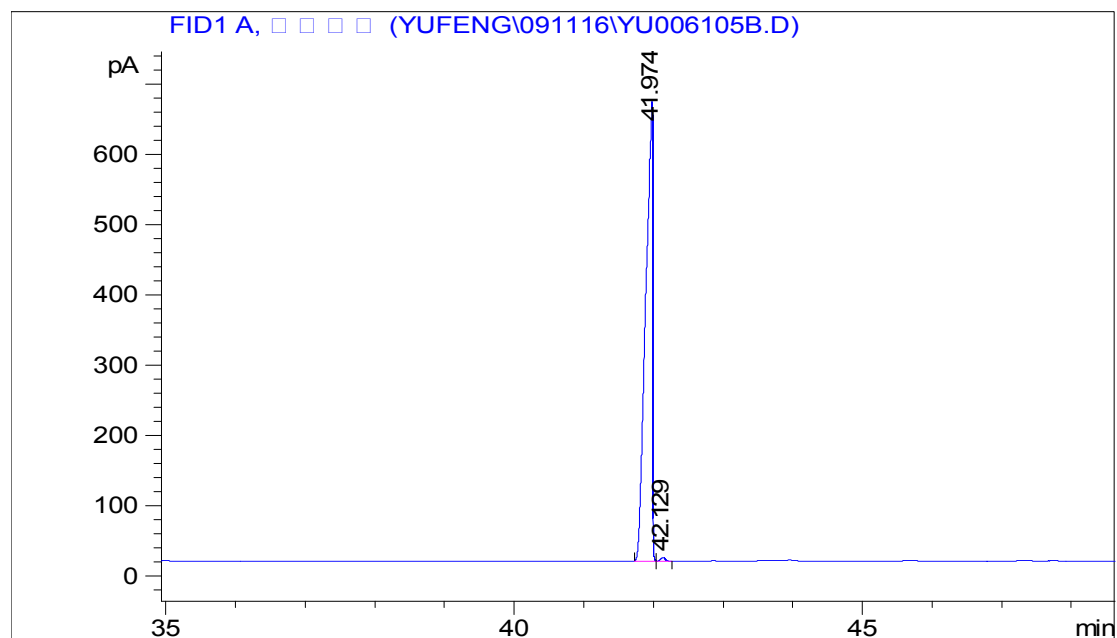


#	Time	Area	Height	Width	Symmetry	Area %
1	20.834	20.7	5.7	0.0612	1.398	0.248
2	20.926	8320.8	316.9	0.3194	5.69E-2	99.752

2-(2,5-dioxo-1-propylpyrrolidin-3-yl)-2-ethylbutanal (3k)

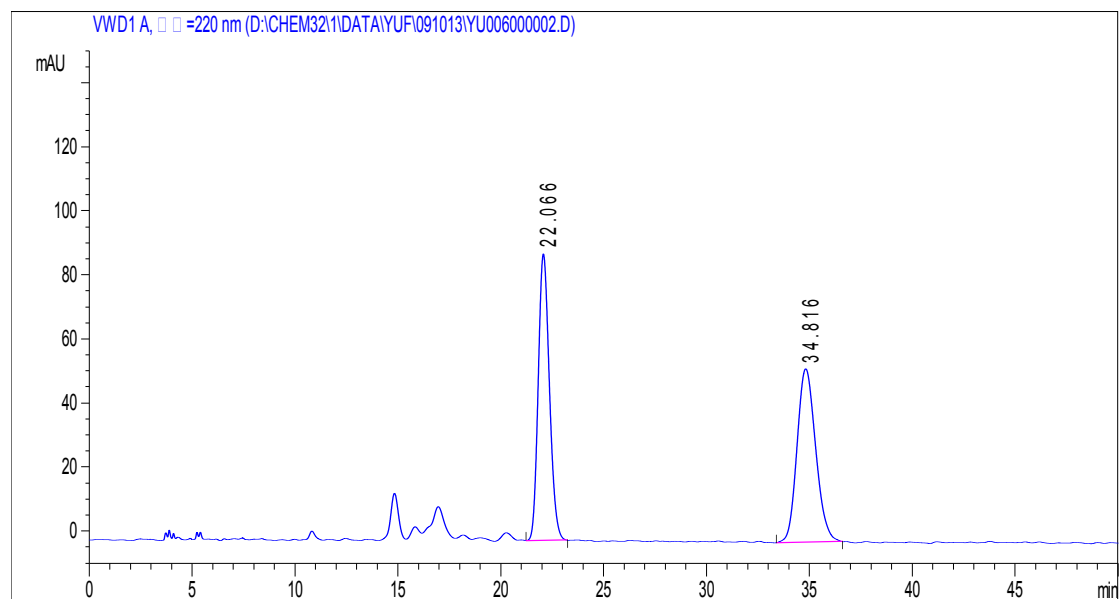


#	Time	Area	Height	Width	Symmetry	Area %
1	41.882	1487.8	278.3	0.0784	1.861	49.979
2	42.116	1489.1	312.1	0.0657	1.629	50.021

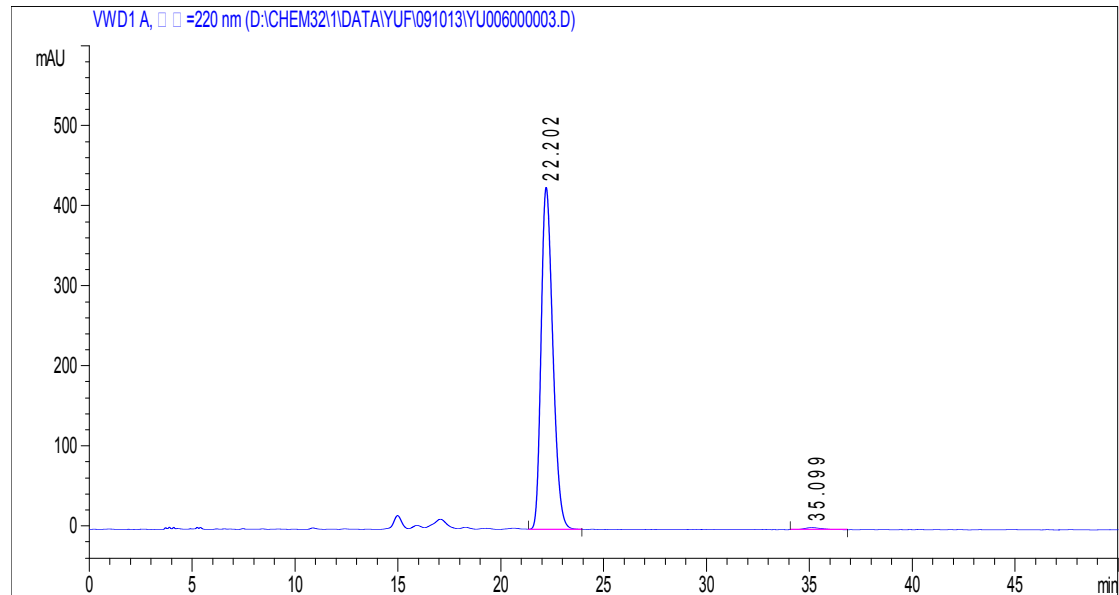


#	Time	Area	Height	Width	Symmetry	Area %
1	41.974	4461.4	649.5	0.0851	8.804	99.489
2	42.129	22.9	5.3	0.0675	0.879	0.511

1-(2,5-dioxo-1-phenylpyrrolidin-3-yl)cyclohexanecarbaldehyde(3I)

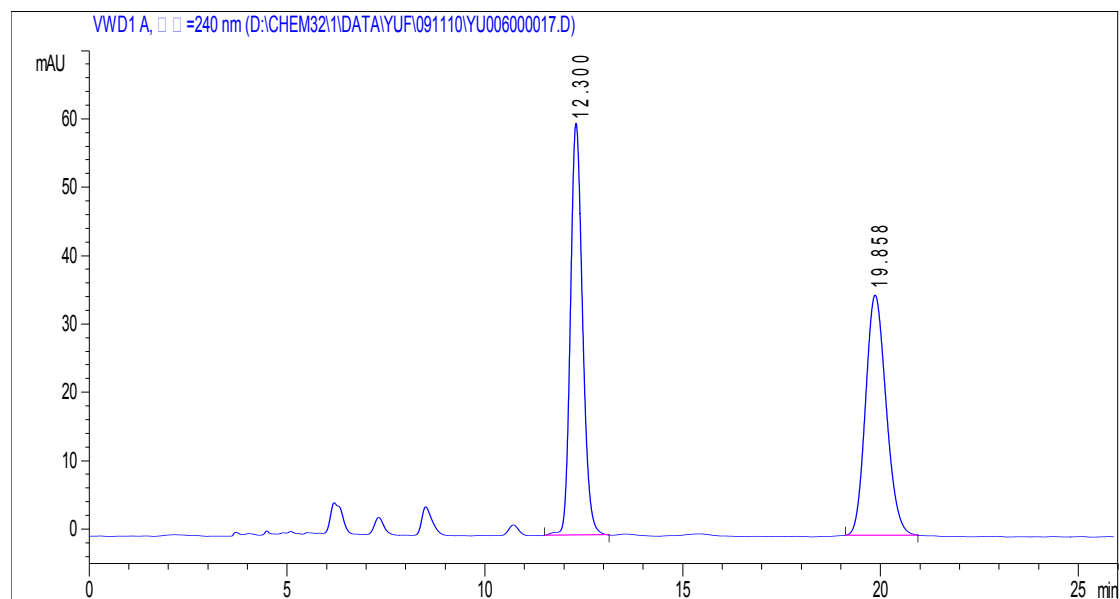


#	Time	Area	Height	Width	Symmetry	Area %
1	22.066	3376.3	89.5	0.5856	0.815	49.707
2	34.816	3416.1	54.2	0.9886	0.831	50.293

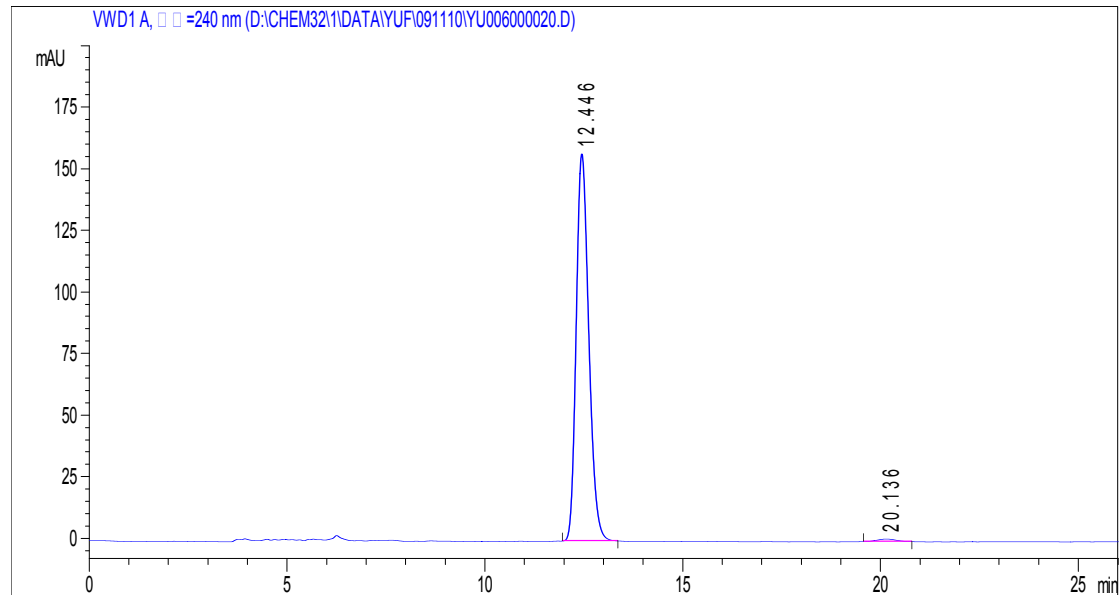


#	Time	Area	Height	Width	Symmetry	Area %
1	22.202	16954.2	427.4	0.6171	0.715	99.028
2	35.099	166.5	2.7	0.8889	0.667	0.972

1-(1-(3-chlorophenyl)-2,5-dioxopyrrolidin-3-yl)cyclohexanecarbaldehyde (3m)

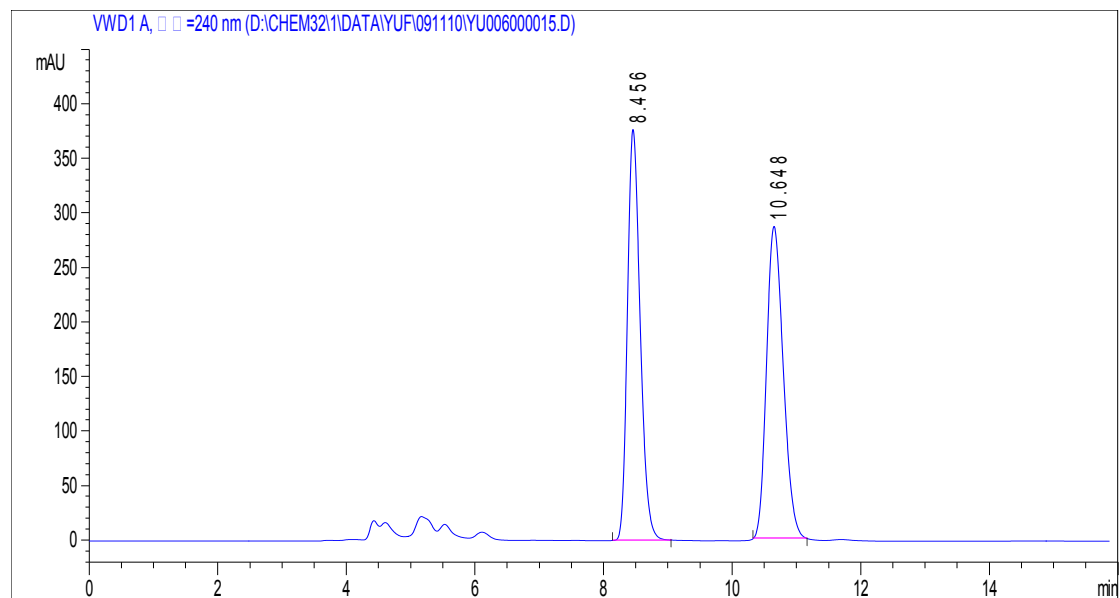


#	Time	Area	Height	Width	Symmetry	Area %
1	12.3	1286.3	60.3	0.3304	0.821	50.208
2	19.858	1275.7	35.2	0.5669	0.823	49.792

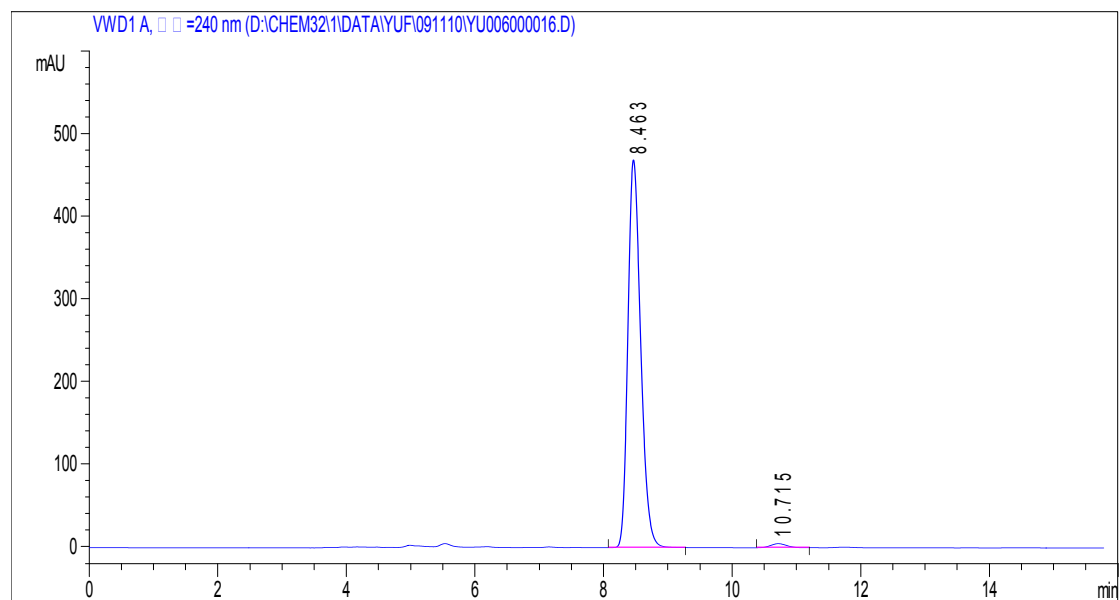


#	Time	Area	Height	Width	Symmetry	Area %
1	12.446	3480.8	157.2	0.3428	0.761	98.947
2	20.136	37	1.1	0.5636	0.883	1.053

1-(2,5-dioxo-1-(4-(trifluoromethyl)phenyl)pyrrolidin-3-yl)cyclohexanecarbaldehyde (3n)

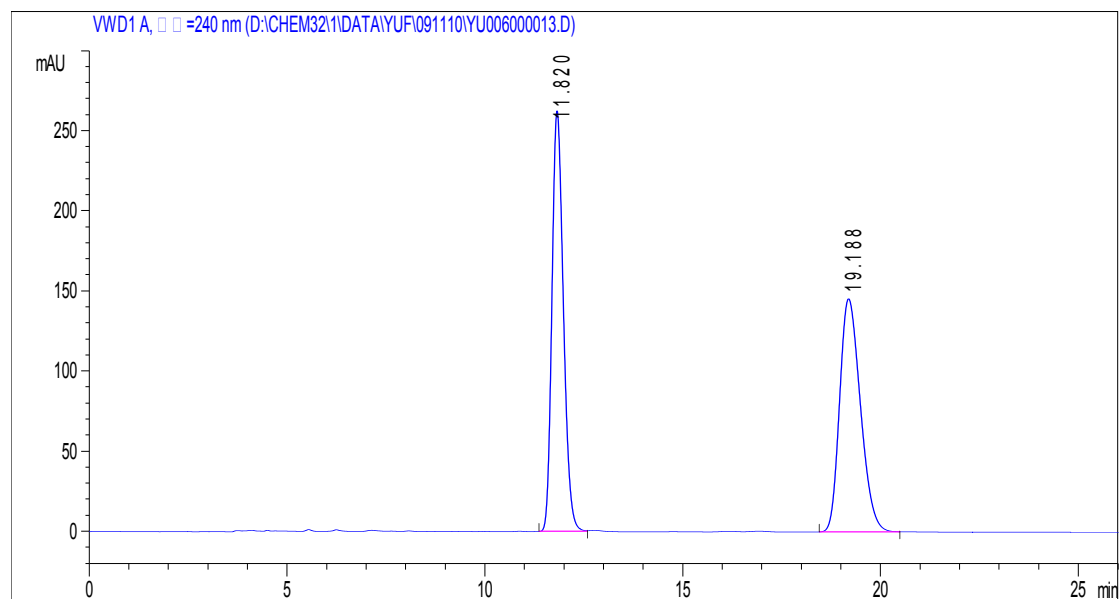


#	Time	Area	Height	Width	Symmetry	Area %
1	8.456	5188.4	376.9	0.2294	0.759	50.096
2	10.648	5168.6	286.2	0.3009	0.78	49.904

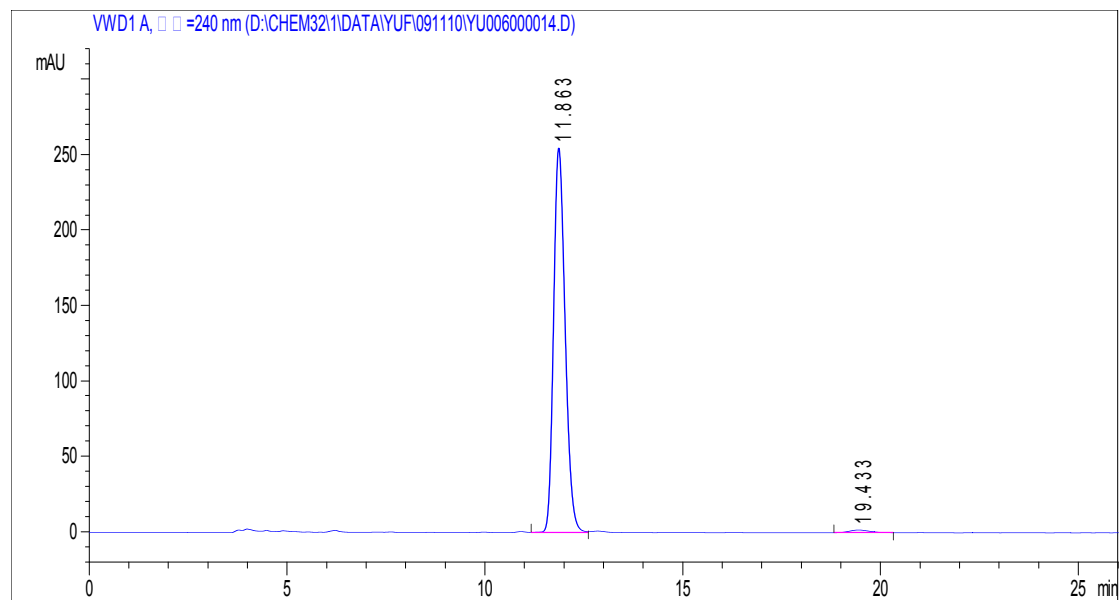


#	Time	Area	Height	Width	Symmetry	Area %
1	8.463	6470.9	469.1	0.2133	0.734	98.669
2	10.715	87.3	5	0.2754	0.877	1.331

1-(2,5-dioxo-1-m-tolylpyrrolidin-3-yl)cyclohexanecarbaldehyde (3o)

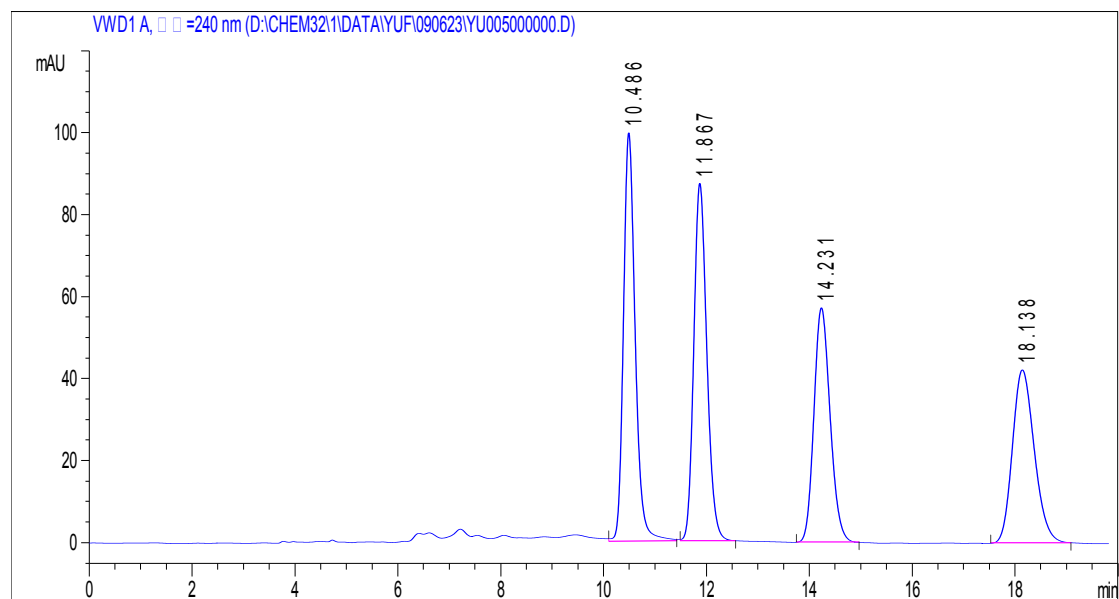


#	Time	Area	Height	Width	Symmetry	Area %
1	11.82	5288.7	262.7	0.3121	0.764	49.878
2	19.188	5314.6	145.6	0.57	0.721	50.122

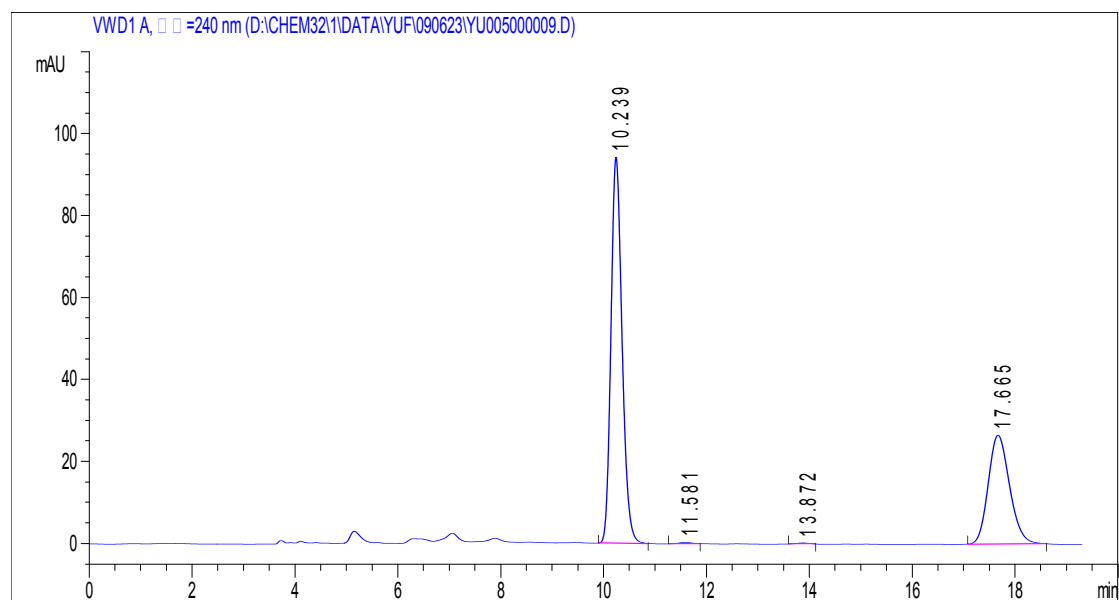


#	Time	Area	Height	Width	Symmetry	Area %
1	11.863	5178.2	255.1	0.3154	0.771	98.724
2	19.433	66.9	1.8	0.5101	0.76	1.276

2-(2,5-dioxo-1-phenylpyrrolidin-3-yl)-2-methylpentanal (7a)

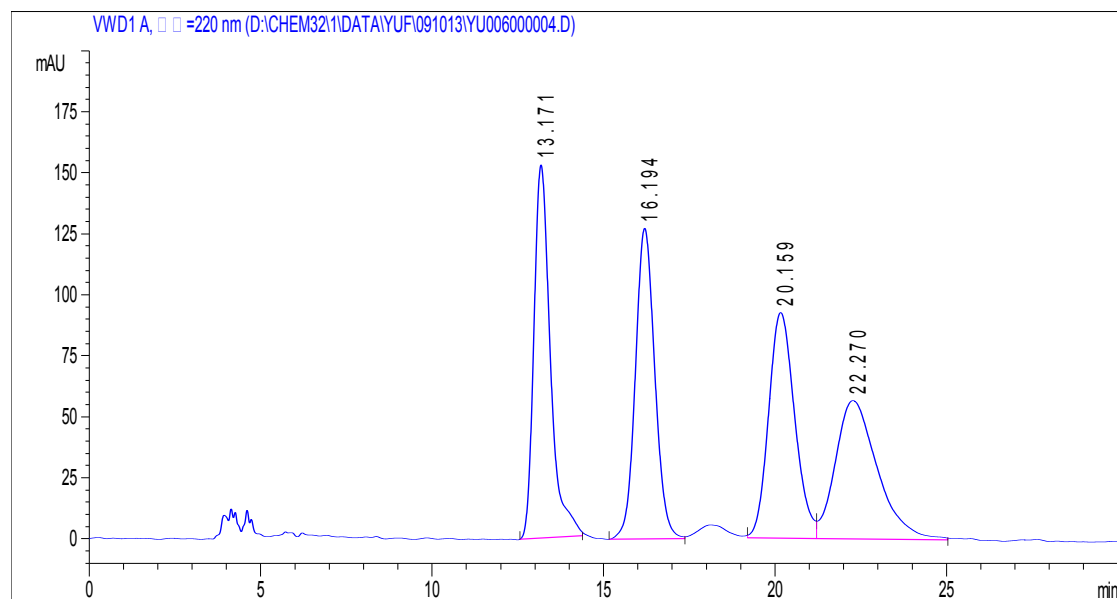


#	Time	Area	Height	Width	Symmetry	Area %
1	10.486	1585.8	99.7	0.2438	0.764	28.176
2	11.867	1536.1	87.3	0.2708	0.81	27.294
3	14.231	1254.3	57.3	0.3386	0.813	22.287
4	18.138	1251.8	42.3	0.4603	0.79	22.242

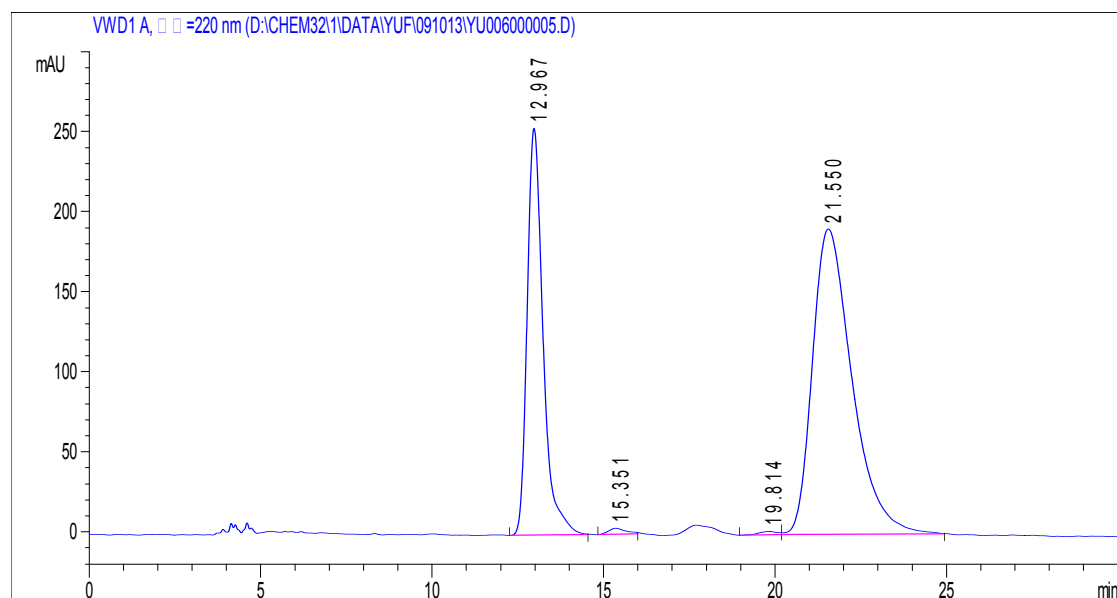


#	Time	Area	Height	Width	Symmetry	Area %
1	10.239	1411.1	94.2	0.2301	0.795	64.359
2	11.581	7.1	4E-1	0.2975	0.92	0.322
3	13.872	5.3	2.9E-1	0.3017	0.996	0.240
4	17.665	769.1	26.6	0.4451	0.804	35.079

3-(4-tert-butylphenyl)-2-(2,5-dioxo-1-phenylpyrrolidin-3-yl)-2-methylpropanal (7b)

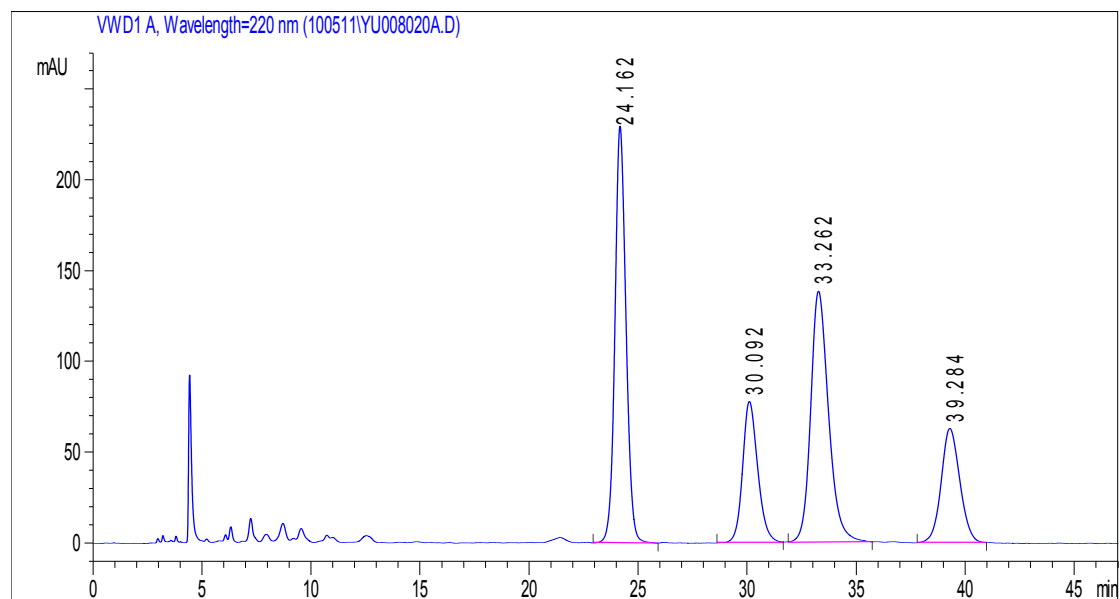


#	Time	Area	Height	Width	Symmetry	Area %
1	13.171	5115.7	153	0.5572	0.723	25.699
2	16.194	5030	127.5	0.6088	0.881	25.269
3	20.159	4915	92.7	0.8841	0.832	24.691
4	22.27	4845.2	56.8	1.4208	0.699	24.341

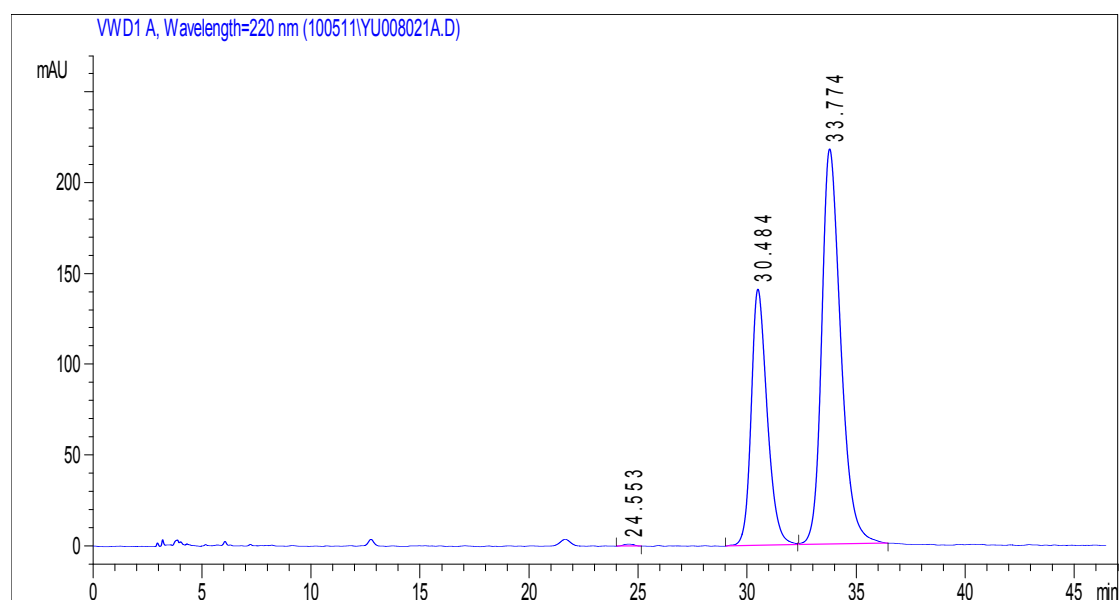


#	Time	Area	Height	Width	Symmetry	Area %
1	12.967	8189	254	0.494	0.7	34.424
2	15.351	146.7	4	0.6172	0.724	0.617
3	19.814	88.8	2.3	0.5375	1.184	0.373
4	21.55	15364.1	191	1.2223	0.626	64.586

3-(4-tert-butylphenyl)-2-(1-(4-chlorophenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (7c)

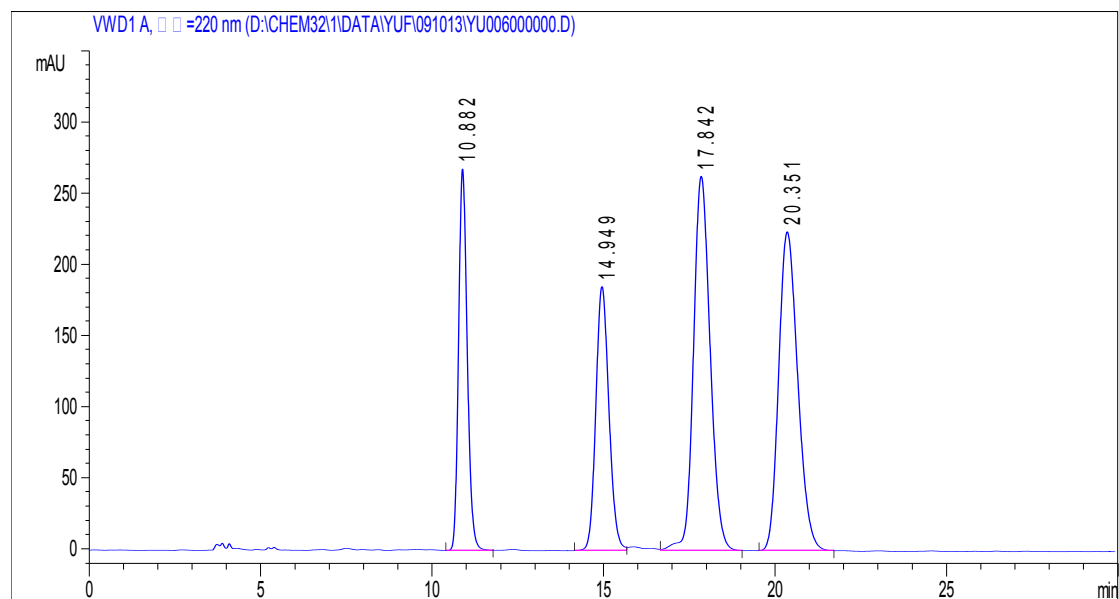


#	Time	Area	Height	Width	Symmetry	Area %
1	24.162	8094	229.8	0.5391	0.833	34.424
2	30.092	3765.7	77.9	0.7359	0.777	16.016
3	33.262	7892.3	138.4	0.8633	0.74	33.566
4	39.284	3760.6	63	0.9231	0.846	15.994

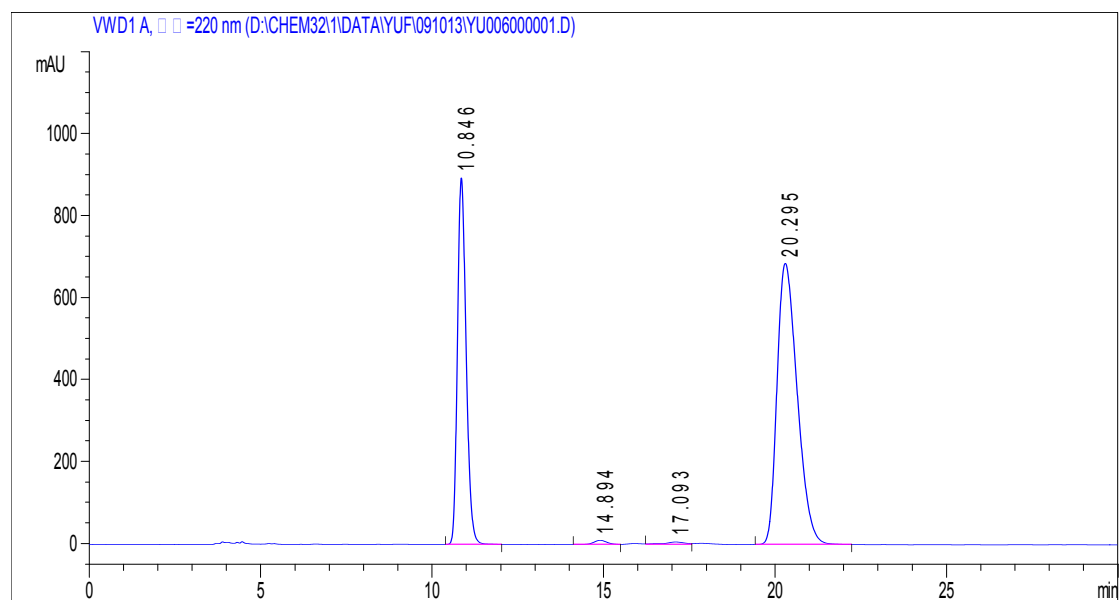


#	Time	Area	Height	Width	Symmetry	Area %
1	24.553	38.2	1.1	0.566	0.968	0.186
2	30.484	7215.5	141.3	0.7719	0.695	35.170
3	33.774	13262.5	217.9	0.93	0.681	64.644

2-(2,5-dioxo-1-phenylpyrrolidin-3-yl)-2,6-dimethylhept-5-enal (7d)

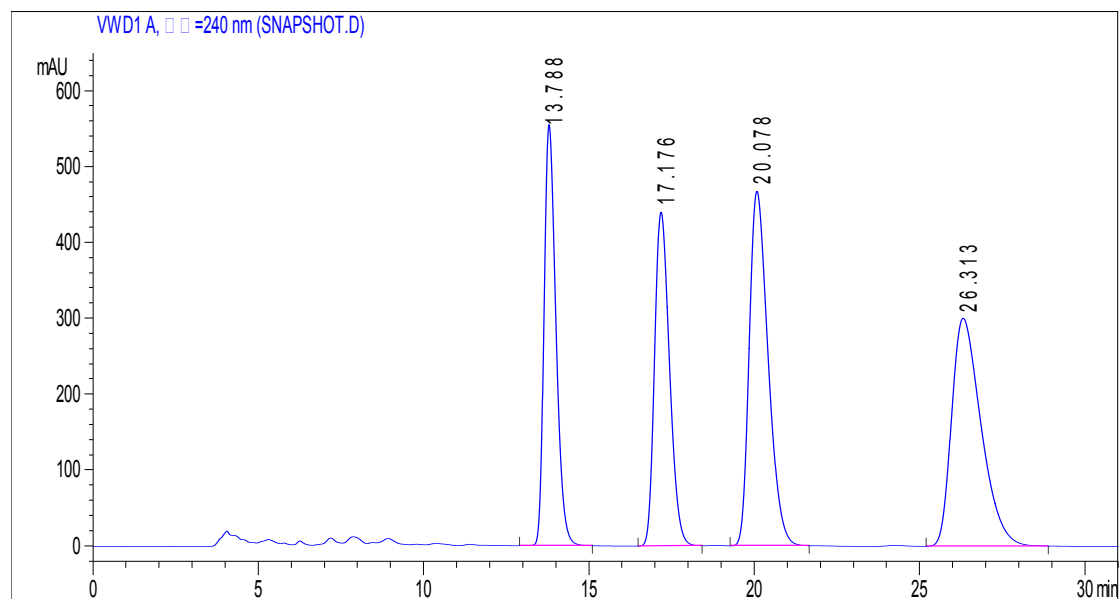


#	Time	Area	Height	Width	Symmetry	Area %
1	10.882	4701.5	268	0.2702	0.801	17.302
2	14.949	4917.6	185.5	0.4112	0.844	18.097
3	17.842	8899.1	262.8	0.5246	0.839	32.749
4	20.351	8655.6	224	0.6007	0.773	31.853

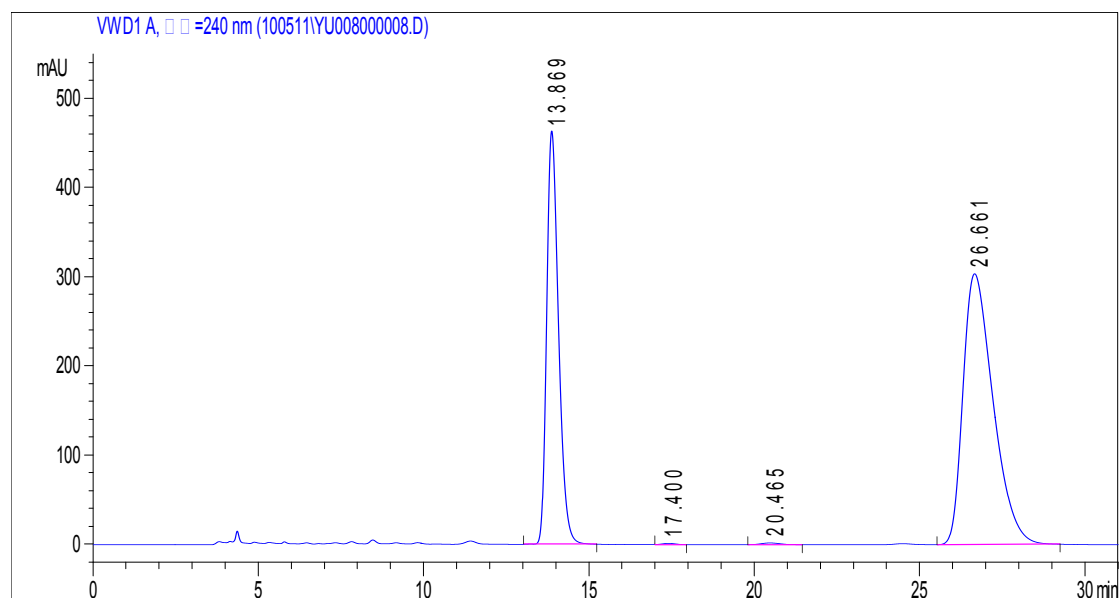


#	Time	Area	Height	Width	Symmetry	Area %
1	10.846	15741.4	894.2	0.2723	0.754	35.541
2	14.894	278.8	10.2	0.4212	0.921	0.629
3	17.093	270.9	6.1	0.6406	1.376	0.612
4	20.295	27999.5	685.8	0.6365	0.654	63.218

2-(1-(4-methoxyphenyl)-2,5-dioxopyrrolidin-3-yl)-2,6-dimethylhept-5-enal (7e)

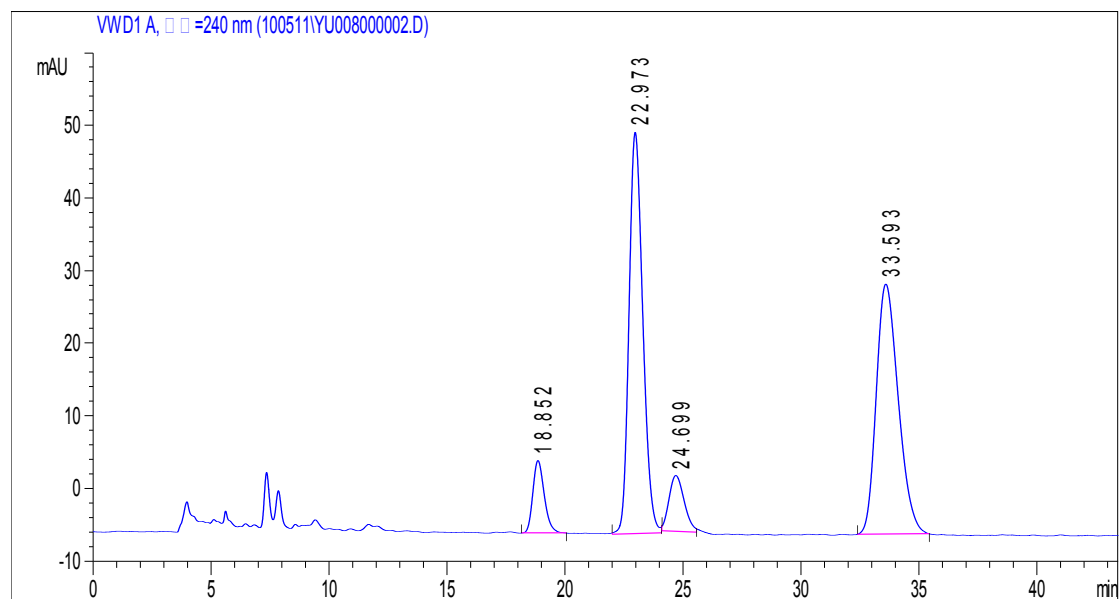


#	Time	Area	Height	Width	Symmetry	Area %
1	13.788	13821	555.1	0.3837	0.679	21.350
2	17.176	13764.7	440.2	0.4871	0.718	21.263
3	20.078	18537.2	467.5	0.614	0.652	28.635
4	26.313	18614	300.7	0.9537	0.602	28.753

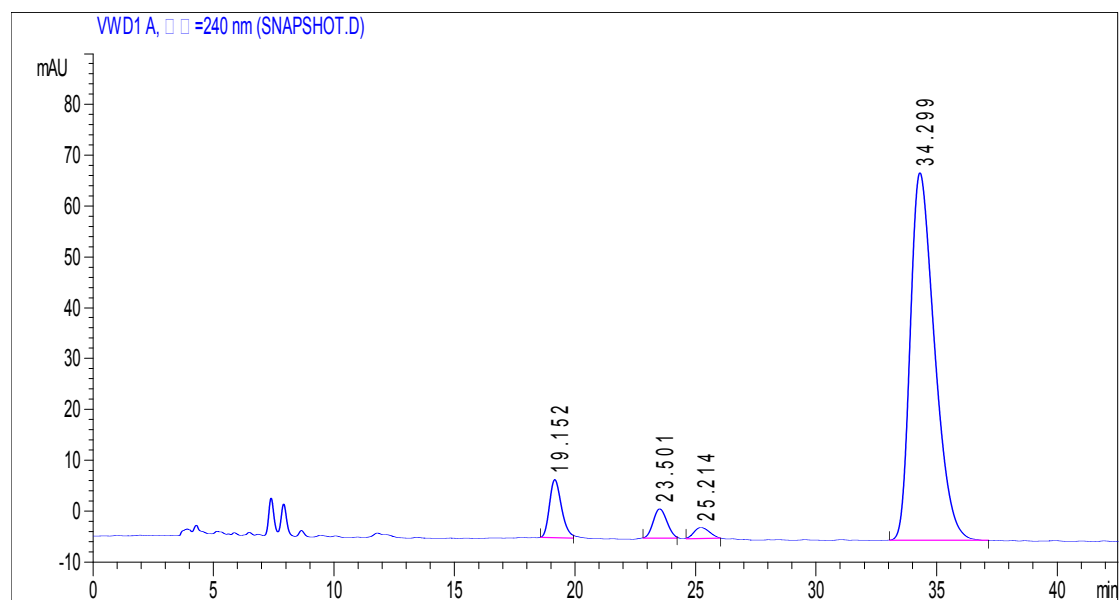


#	Time	Area	Height	Width	Symmetry	Area %
1	13.869	11551.2	463.8	0.3837	0.688	37.350
2	17.4	43.9	1.5	0.4869	0.891	0.142
3	20.465	80.6	2	0.6068	0.783	0.261
4	26.661	19251.3	303.9	0.9765	0.591	62.248

2-(2,5-dioxo-1-phenylpyrrolidin-3-yl)-2-phenylpropanal (7f)

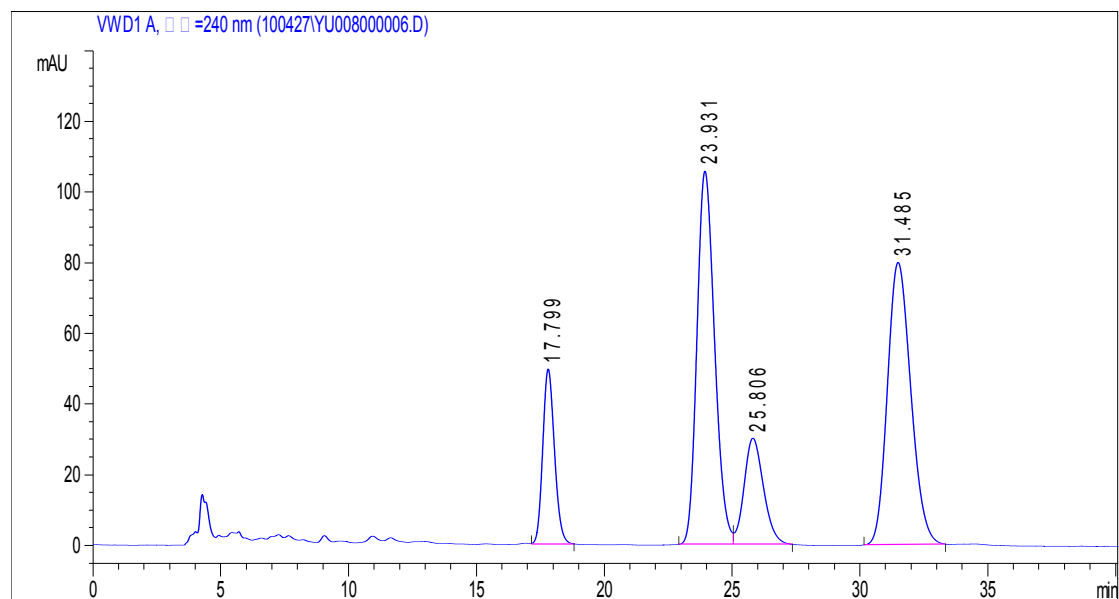


#	Time	Area	Height	Width	Symmetry	Area %
1	18.852	341	10	0.5667	0.777	6.584
2	22.973	2250.9	55.3	0.678	0	43.456
3	24.699	347.4	7.7	0.7476	0.784	6.708
4	33.593	2240.4	34.5	0.9906	0.727	43.253

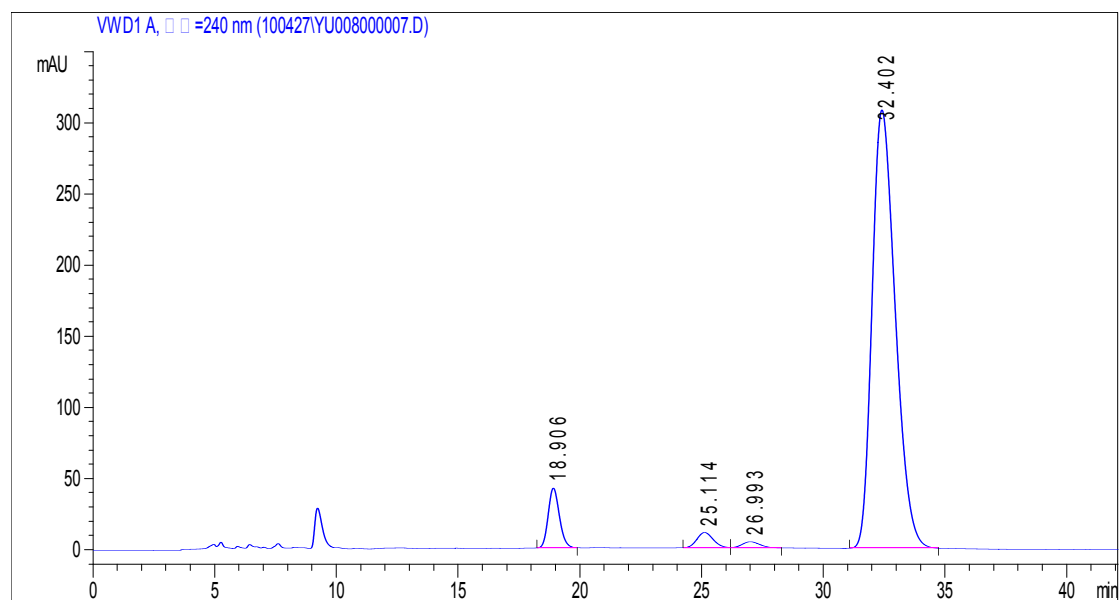


#	Time	Area	Height	Width	Symmetry	Area %
1	19.152	402.3	11.5	0.5835	0.761	6.943
2	23.501	231.4	5.8	0.6644	0.862	3.994
3	25.214	105.7	2.3	0.7765	0.786	1.824
4	34.299	5055	72.4	1.164	0.635	87.240

2-(1-(4-bromophenyl)-2,5-dioxopyrrolidin-3-yl)-2-phenylpropanal (7g)

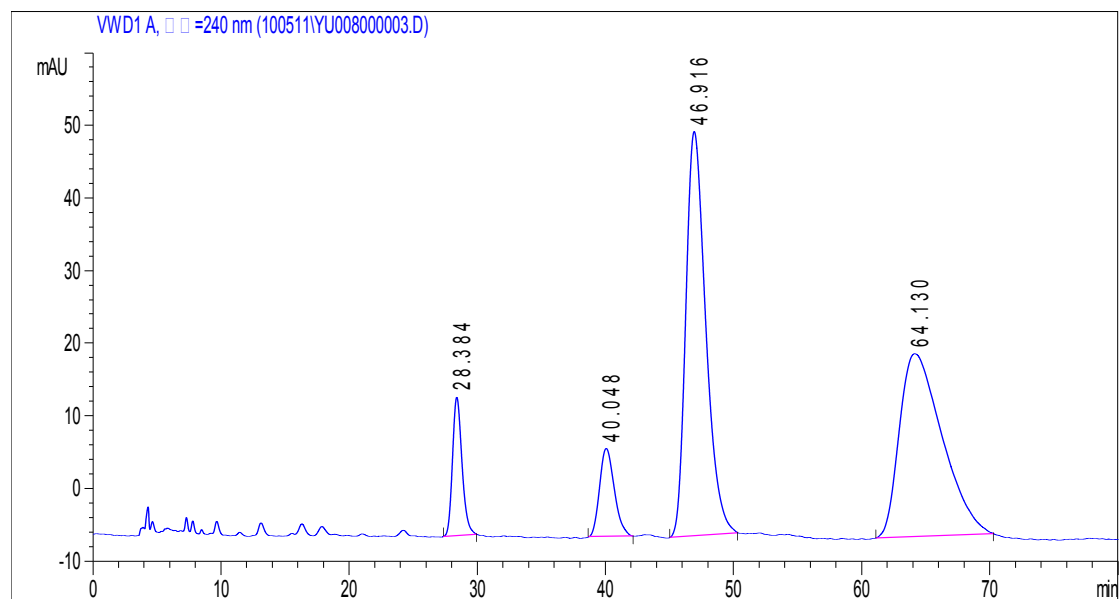


#	Time	Area	Height	Width	Symmetry	Area %
1	17.799	1581.6	49.6	0.4944	0.823	11.853
2	23.931	5073.7	105.7	0.7464	0.786	38.025
3	25.806	1597.2	30.1	0.8121	0.798	11.970
4	31.485	5090.6	79.9	0.9935	0.799	38.152

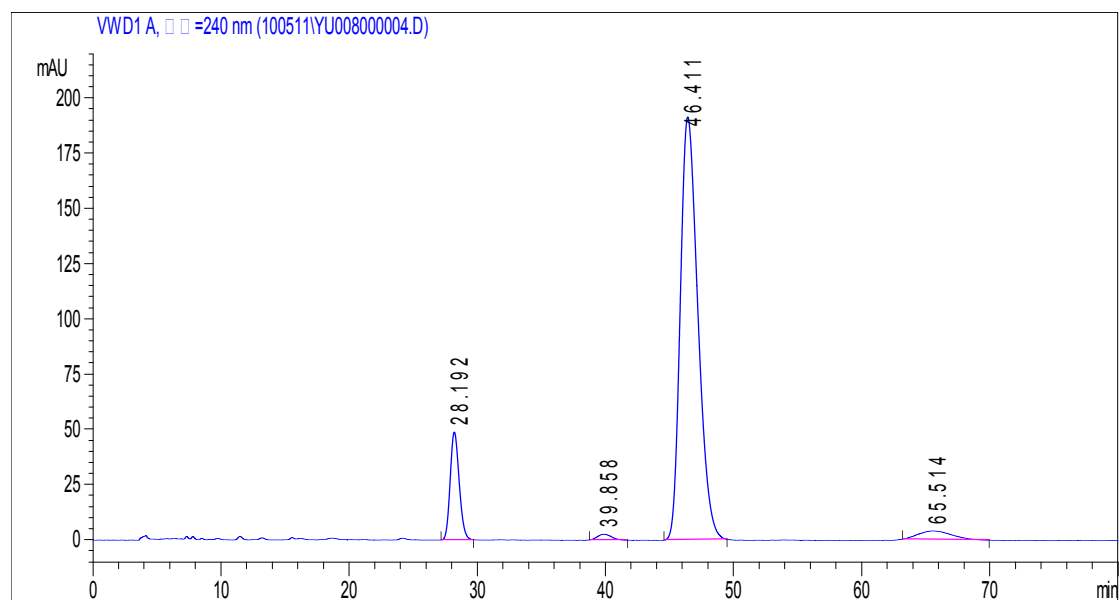


#	Time	Area	Height	Width	Symmetry	Area %
1	18.906	1345	42.1	0.4954	0.821	6.003
2	25.114	501.5	10.7	0.7142	0.842	2.238
3	26.993	216.3	4.3	0.7629	0.843	0.966
4	32.402	20340.6	308	1.0296	0.698	90.793

2-(1-(4-methoxyphenyl)-2,5-dioxopyrrolidin-3-yl)-2-phenylpropanal (7h)

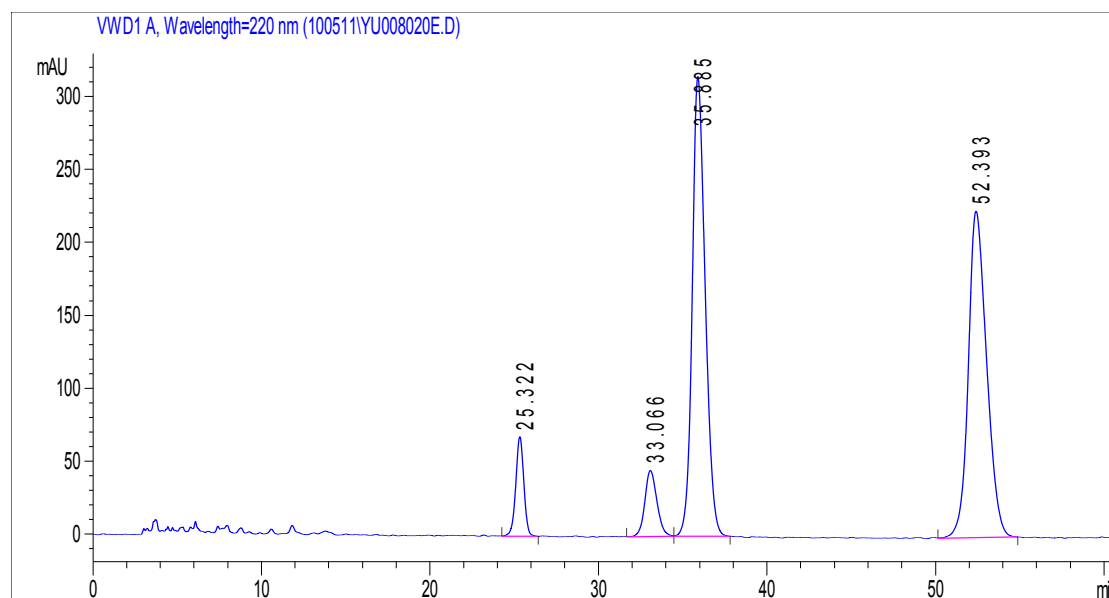


#	Time	Area	Height	Width	Symmetry	Area %
1	28.384	991.6	19.1	0.7944	0.775	7.161
2	40.048	977.2	12.2	1.1742	0.784	7.057
3	46.916	6026	55.8	1.6104	0.686	43.519
4	64.13	5852.1	25.2	2.7383	0.544	42.263

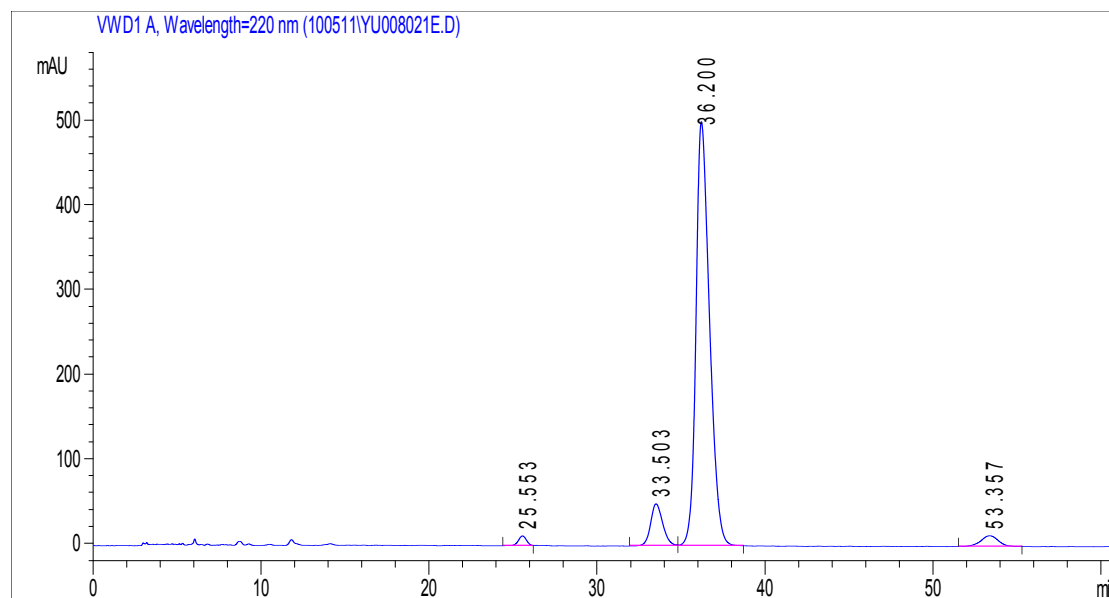


#	Time	Area	Height	Width	Symmetry	Area %
1	28.192	2389.2	48.9	0.7641	0.815	11.096
2	39.858	199.7	2.6	0.9016	0.792	0.928
3	46.411	18237.2	191.5	1.4723	0.697	84.697
4	65.514	706.1	3.9	2.1452	0.702	3.279

2-(1-(3-chlorophenyl)-2,5-dioxopyrrolidin-3-yl)-2-phenylpropanal (7i)



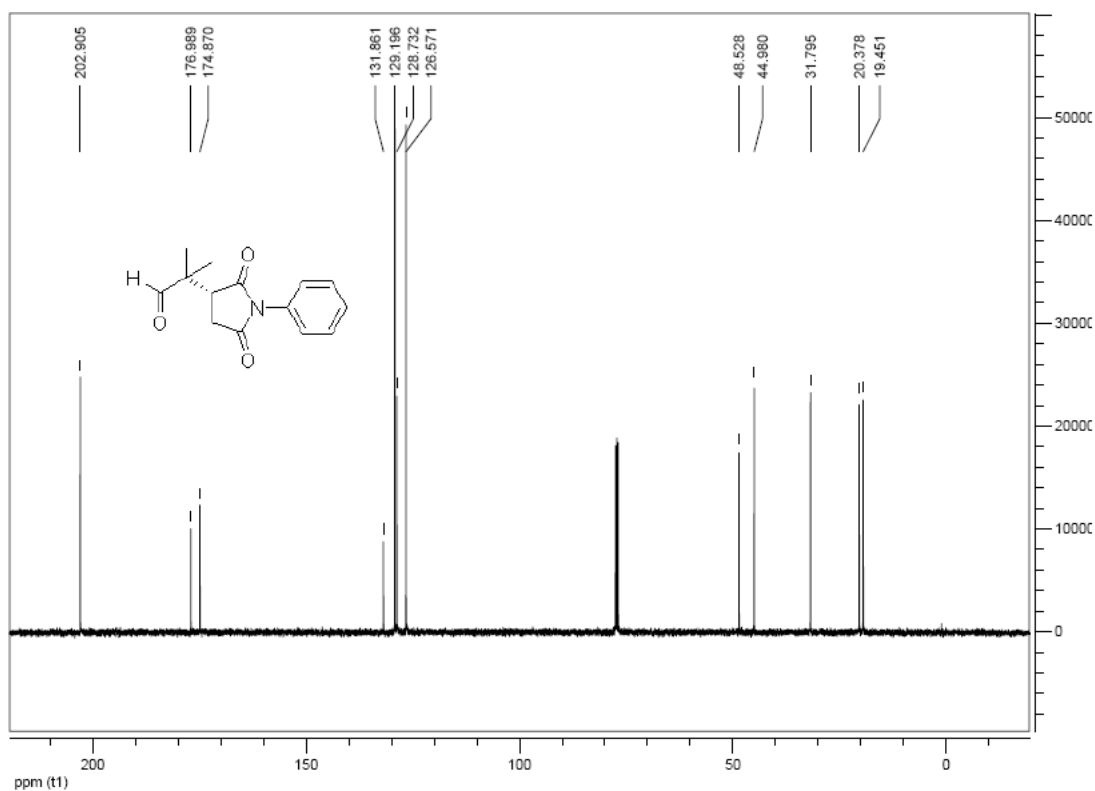
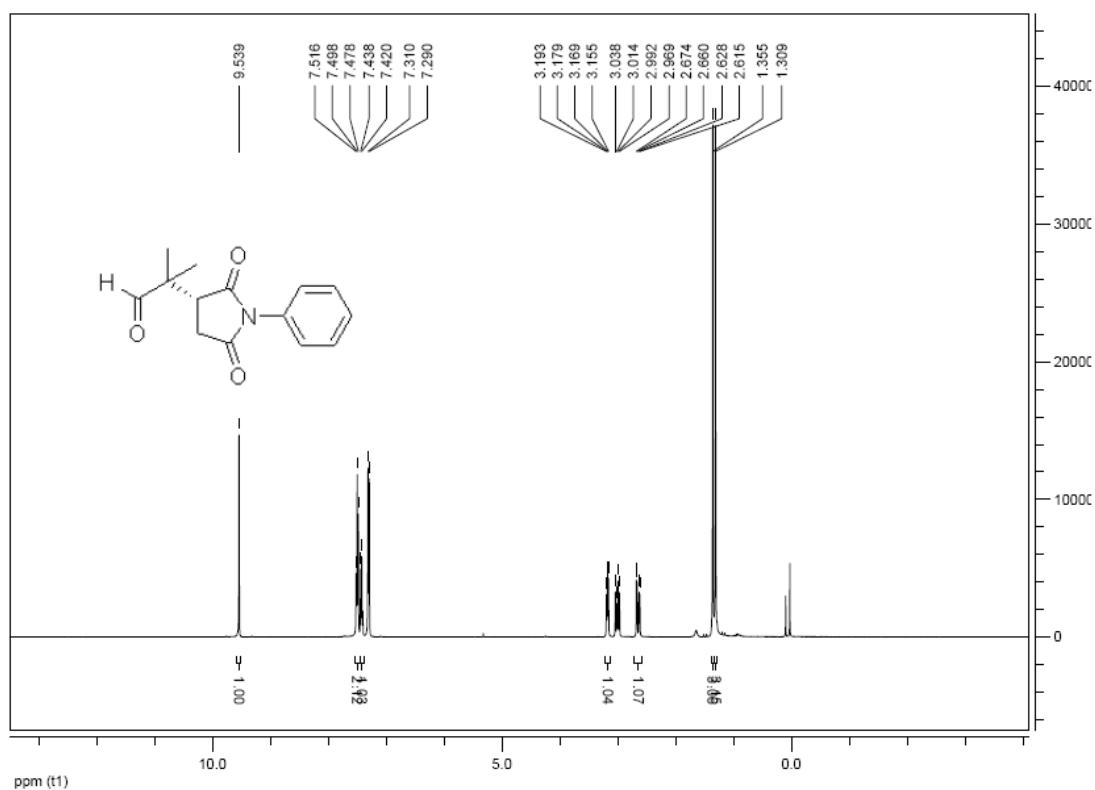
#	Time	Area	Height	Width	Symmetry	Area %
1	25.322	2311.8	68.6	0.5167	1.004	6.002
2	33.066	2316.9	45.6	0.779	0.813	6.015
3	35.885	16917.7	315.8	0.8142	0.743	43.922
4	52.393	16970.8	224.3	1.13	0.717	44.060



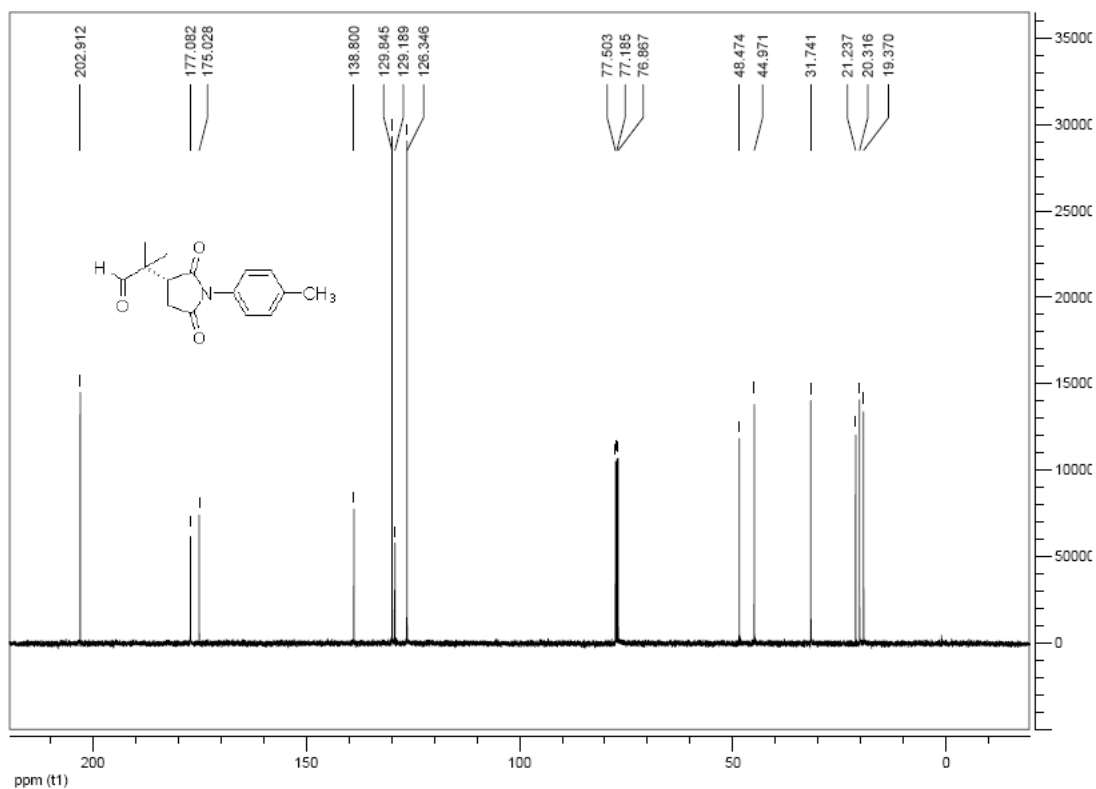
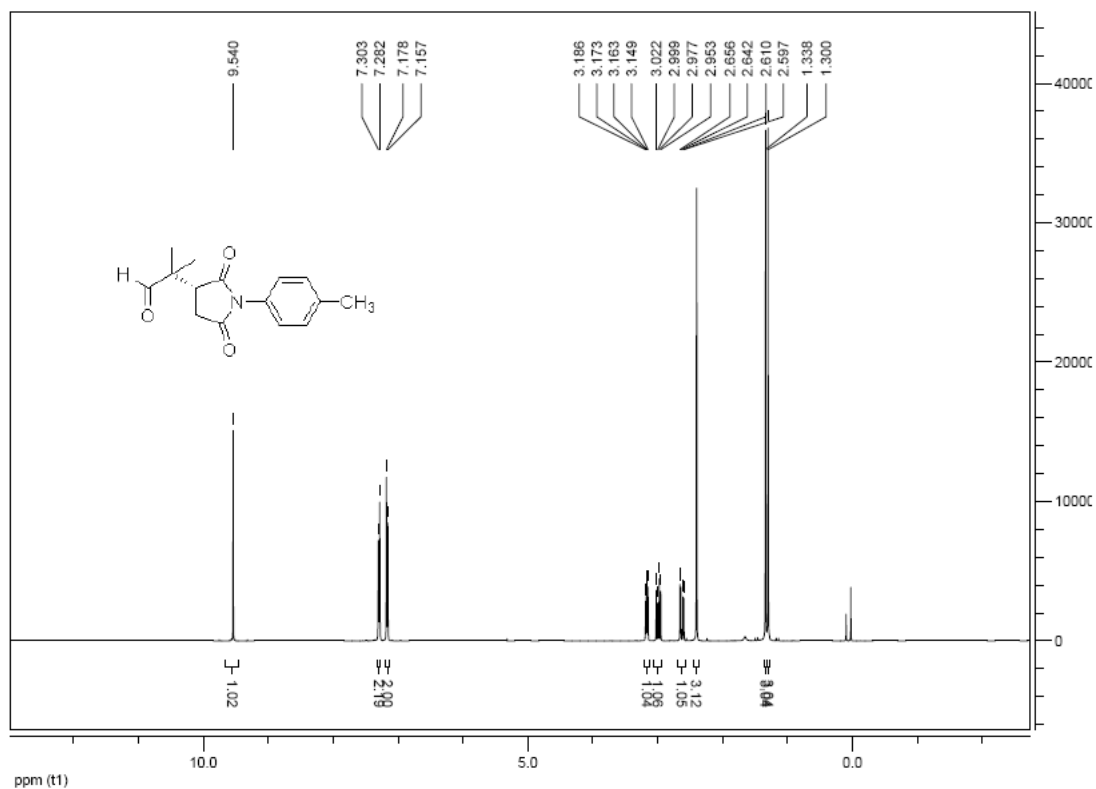
#	Time	Area	Height	Width	Symmetry	Area %
1	25.553	388.8	11.6	0.5198	1.03	1.216
2	33.503	2522.1	49.8	0.777	0.789	7.887
3	36.2	28132	501.1	0.853	0.64	87.975
4	53.357	934.4	12.7	1.1149	1.037	2.922

C: NMR Analysis of Michael Addition Products

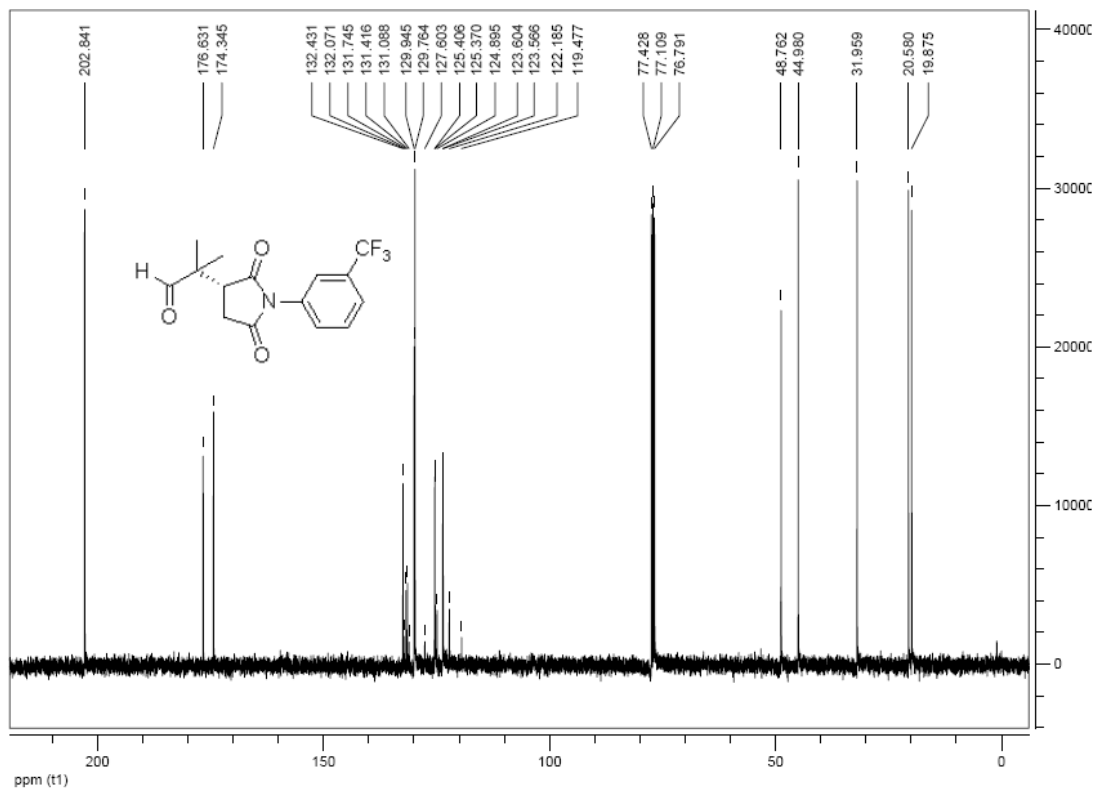
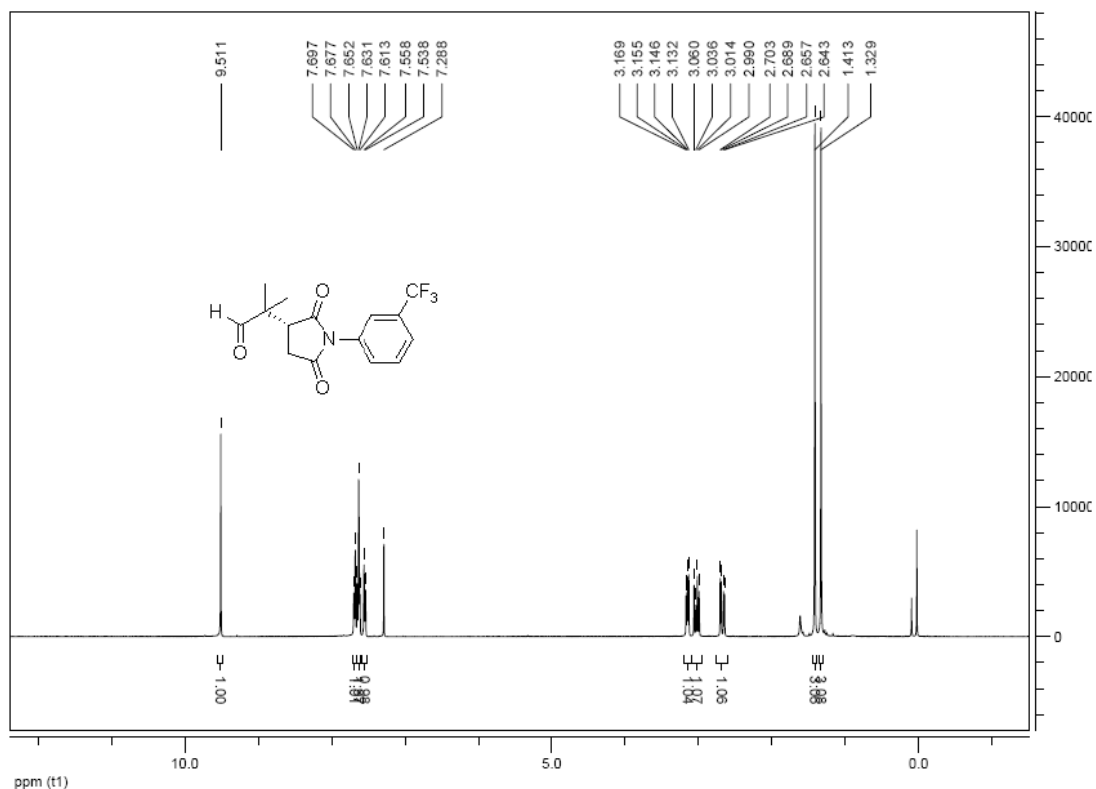
2-(2,5-dioxo-1-phenylpyrrolidin-3-yl)-2-methylpropanal (3a)



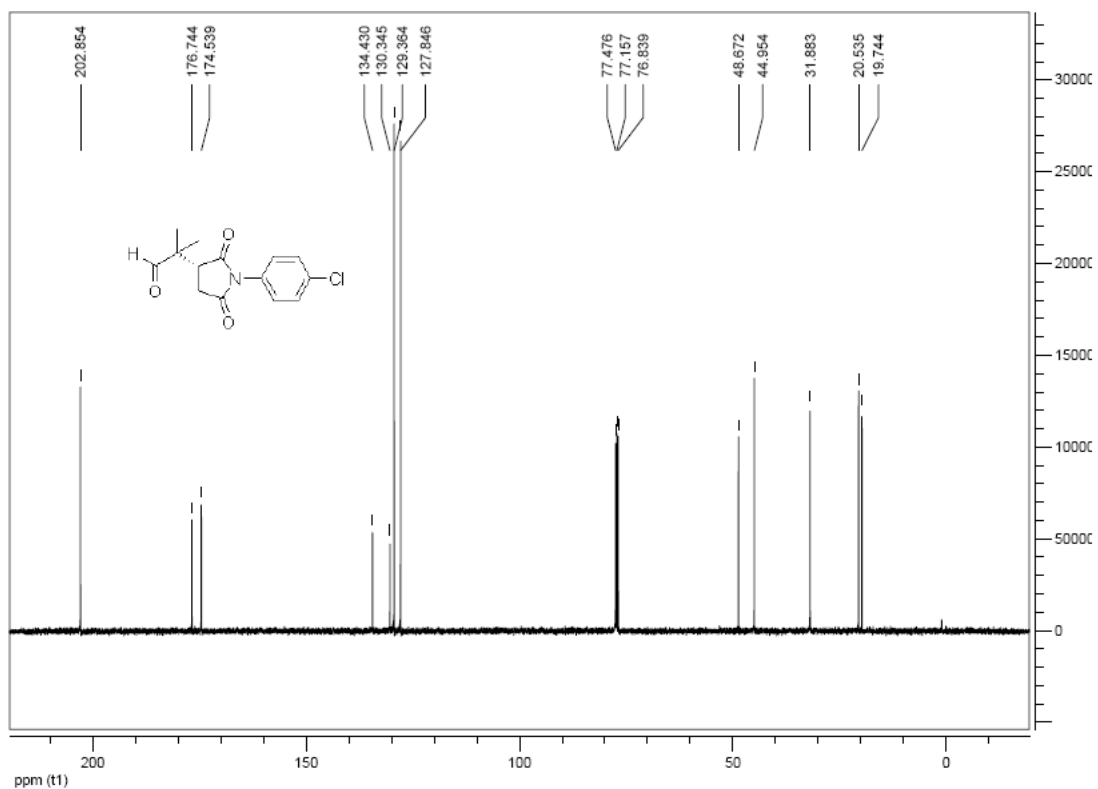
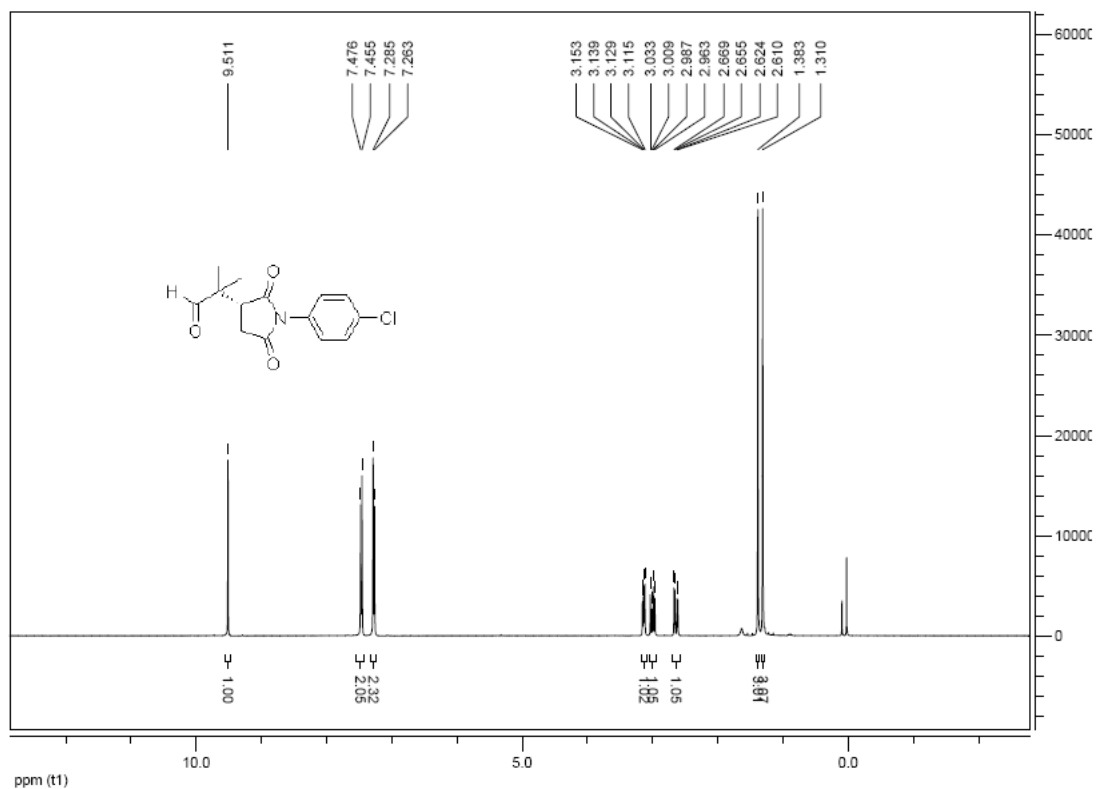
2-(2, 5-dioxo-1-p-tolylpyrrolidin-3-yl)-2-methylpropanal (3b)



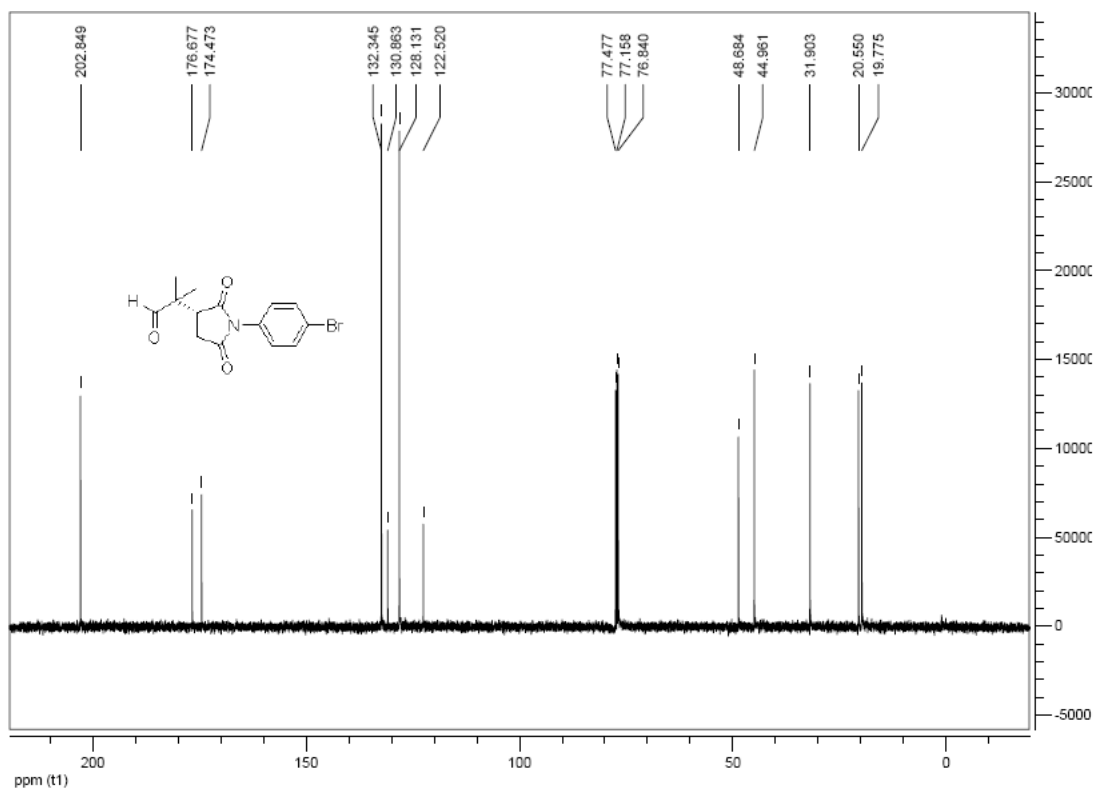
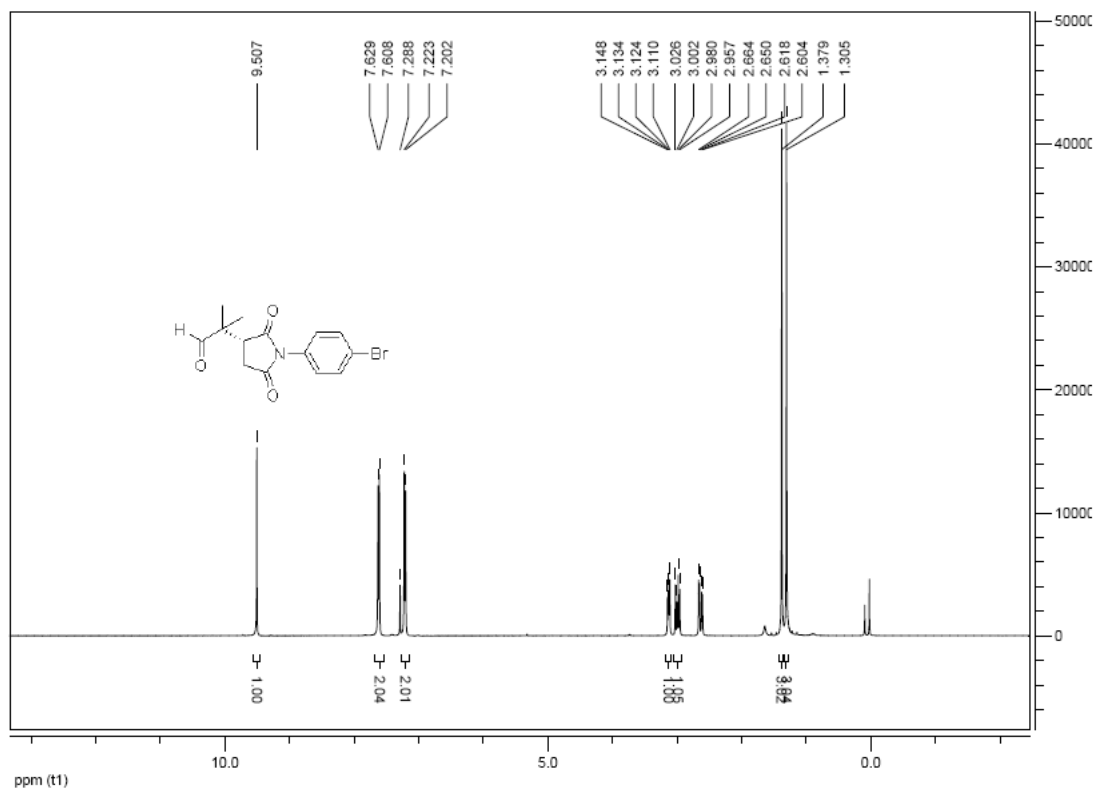
2-(2,5-dioxo-1-(3-(trifluoromethyl)phenyl)pyrrolidin-3-yl)-2-methylpropanal (3c)



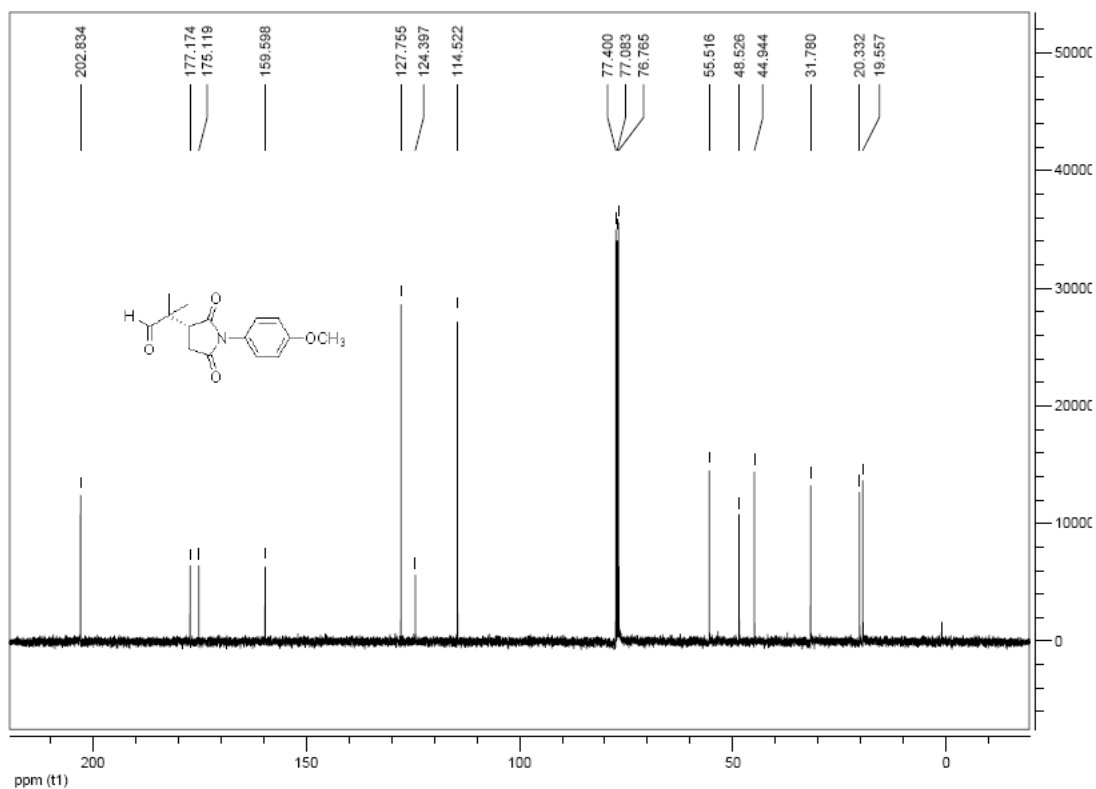
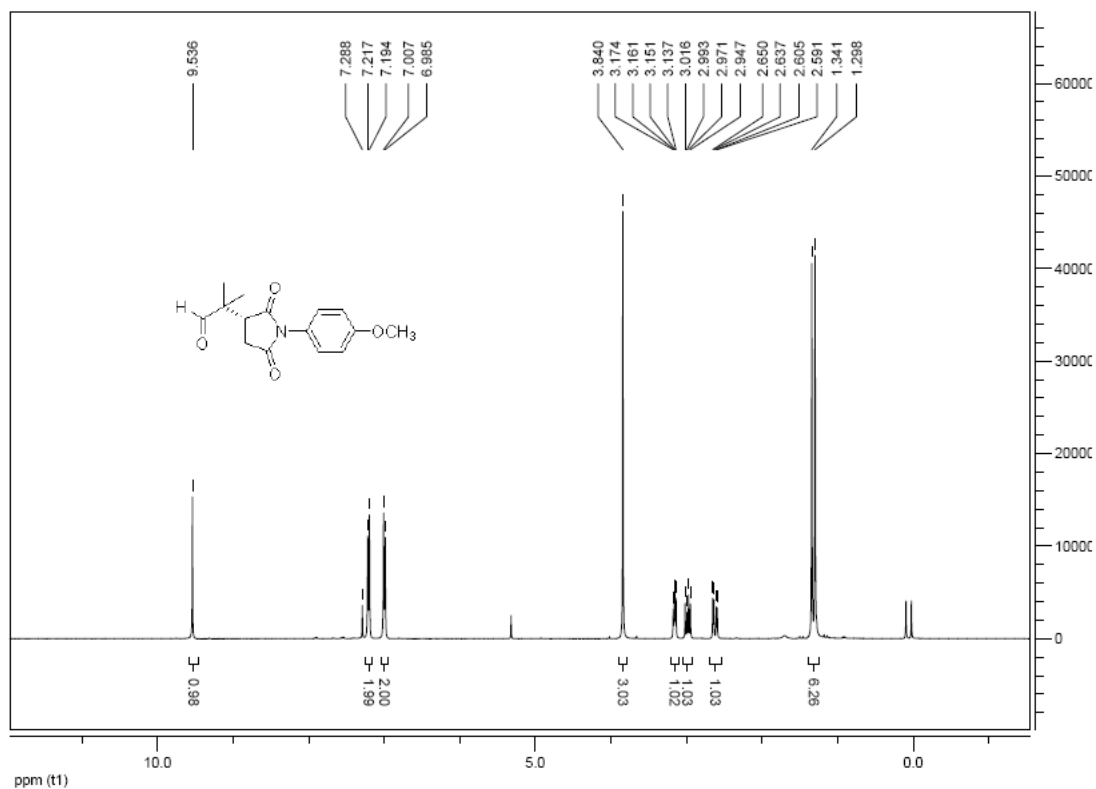
2-(1-(4-chlorophenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (3d)



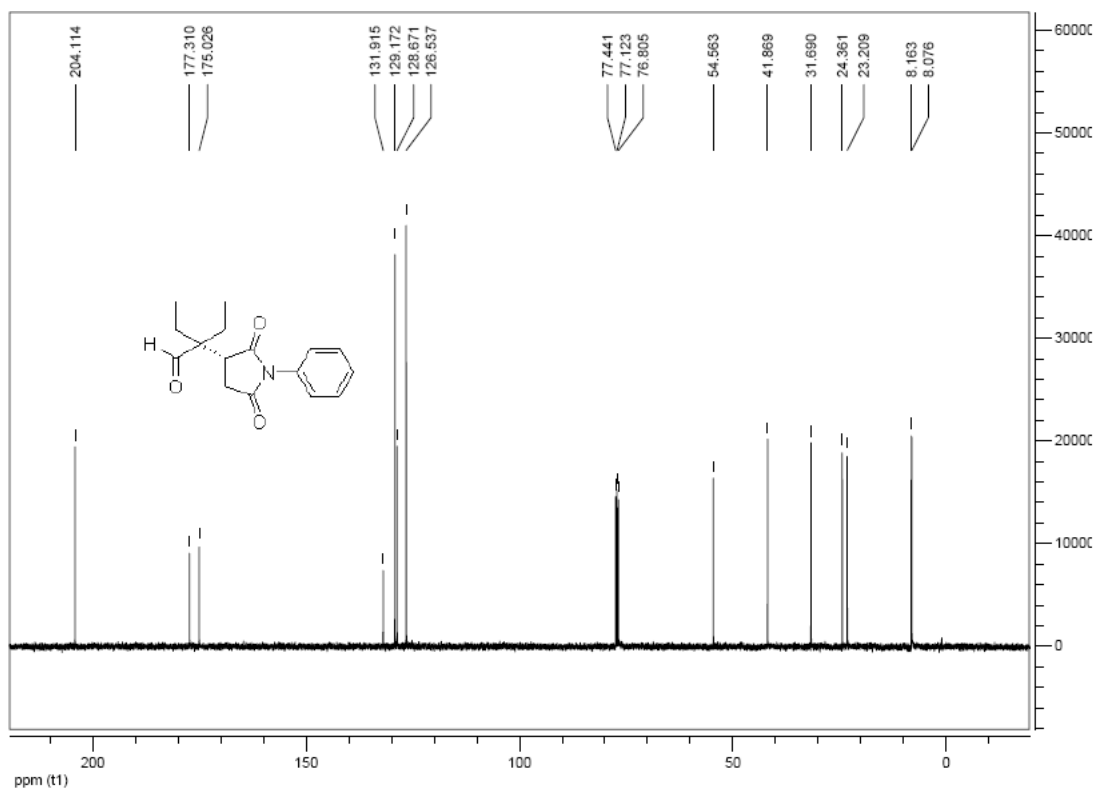
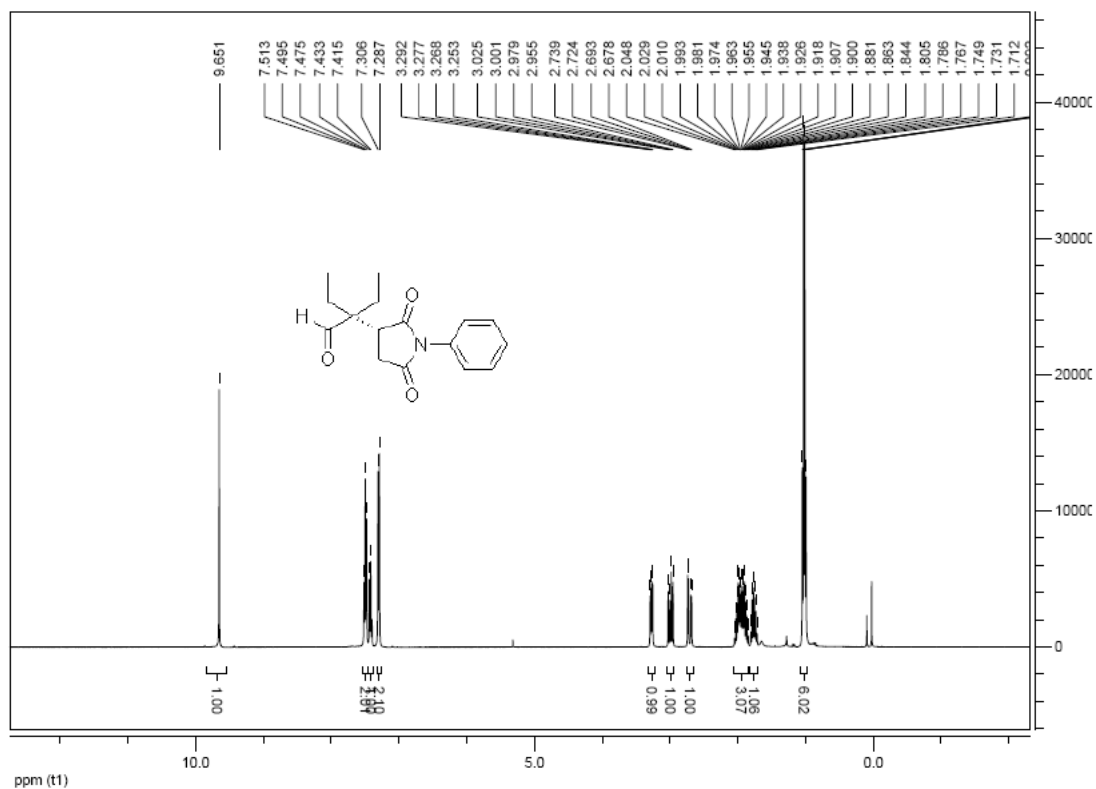
2-(1-(4-bromophenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (3e)



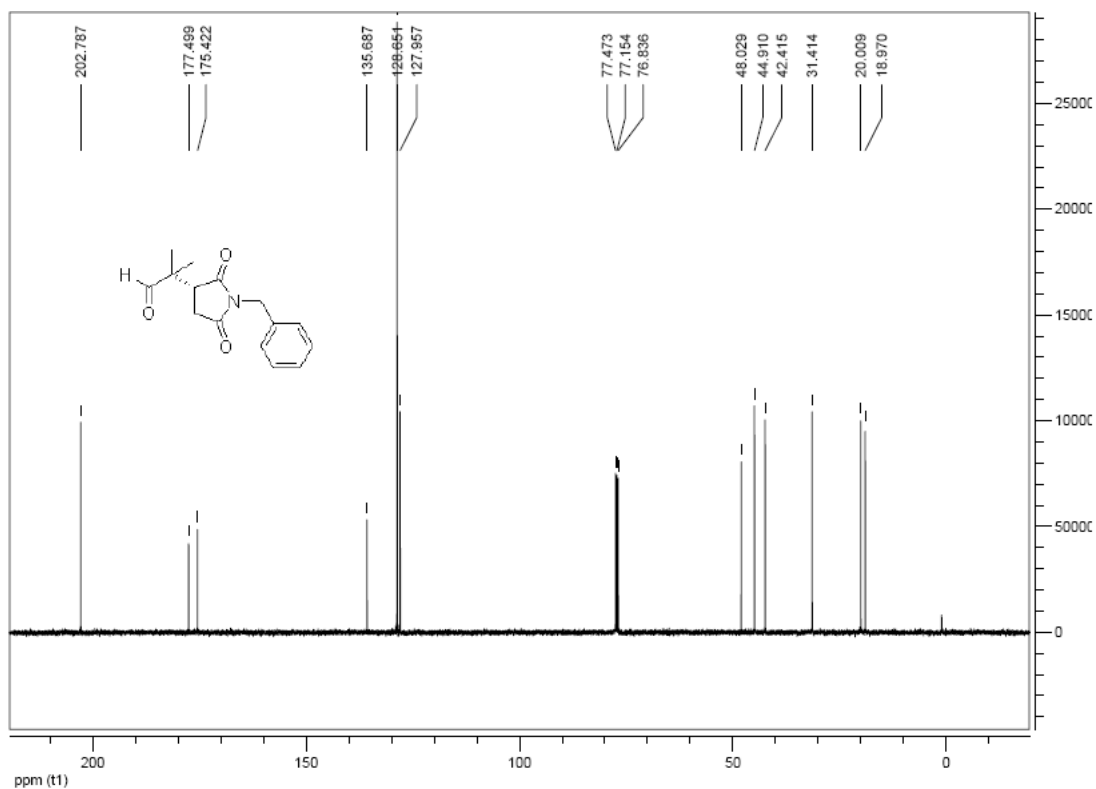
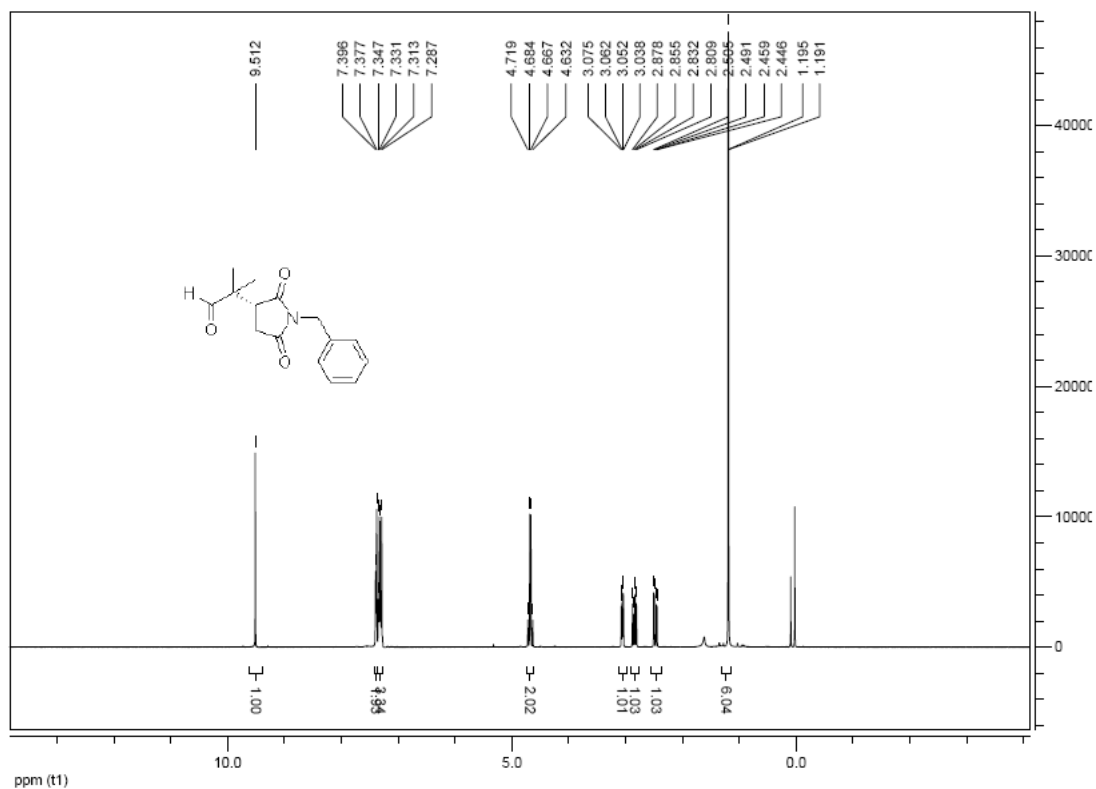
2-(1-(4-methoxyphenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (3f)



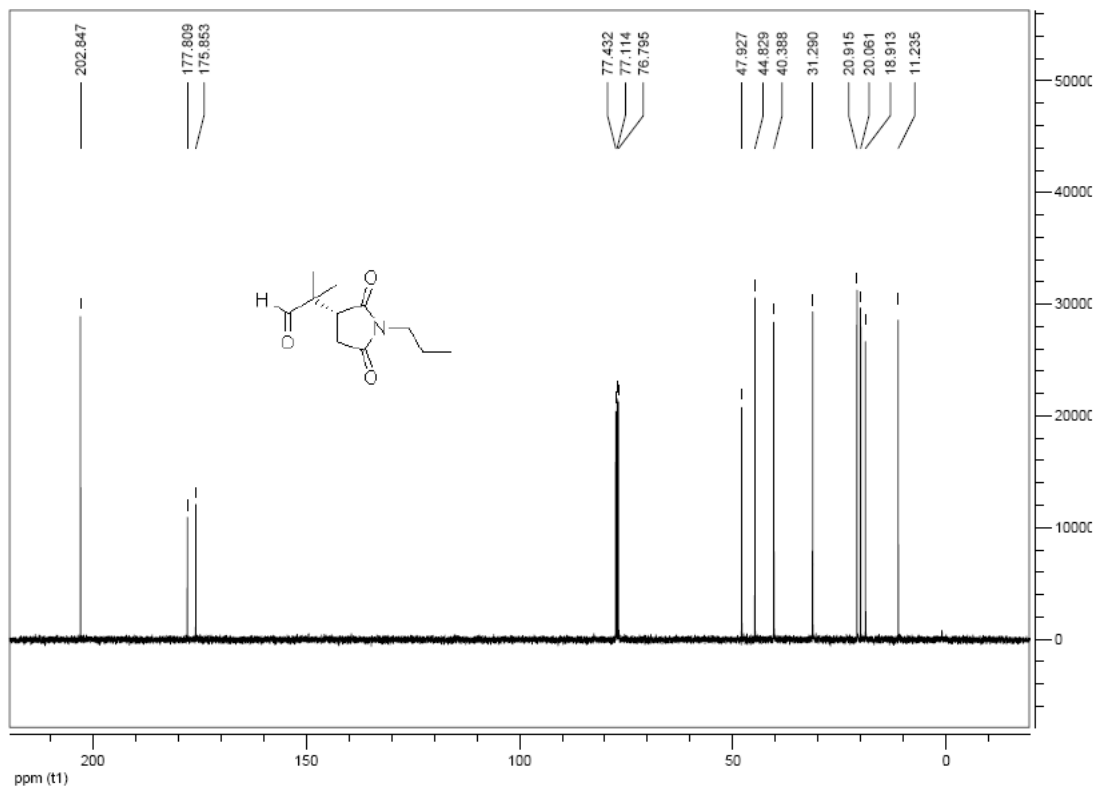
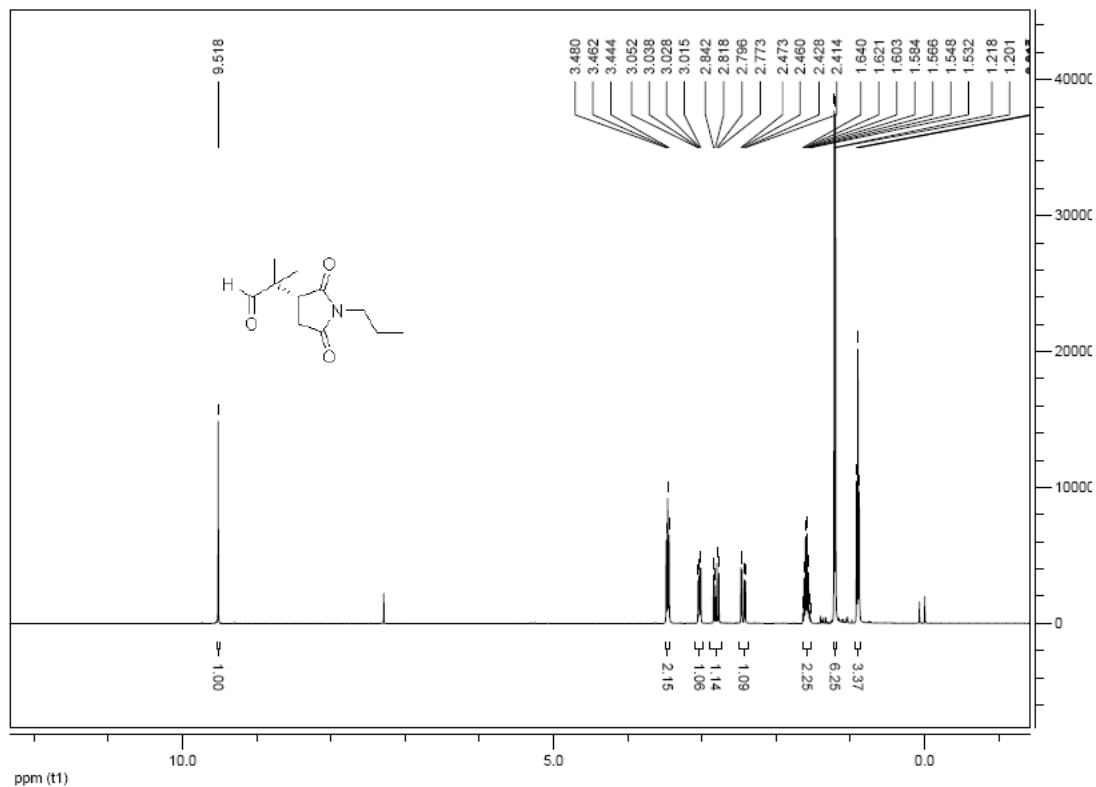
2-(2,5-dioxo-1-phenylpyrrolidin-3-yl)-2-ethylbutanal (3g)



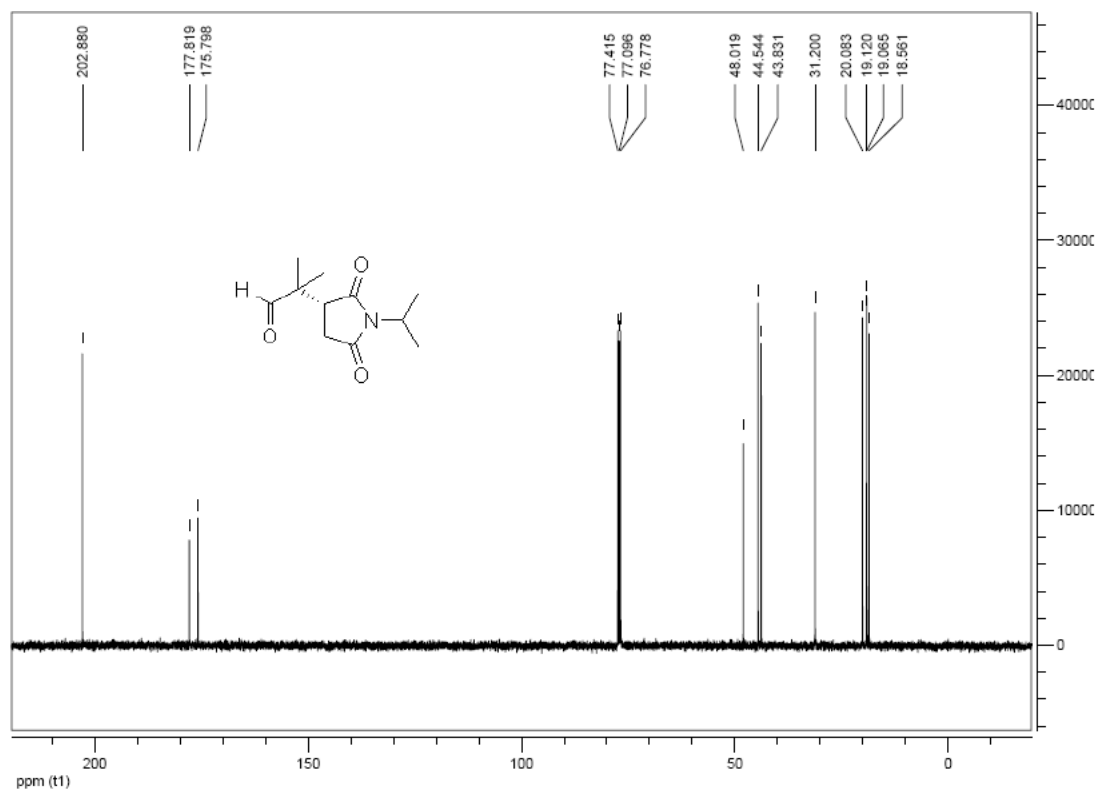
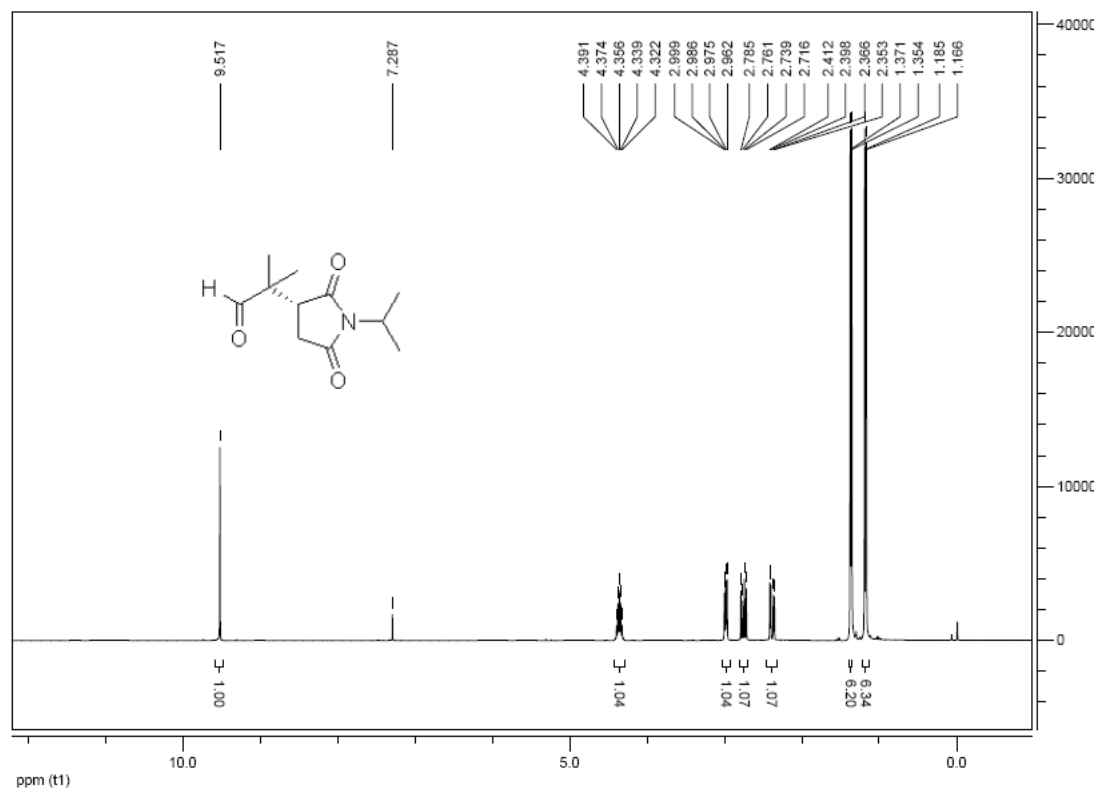
2-(1-benzyl-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (3h)



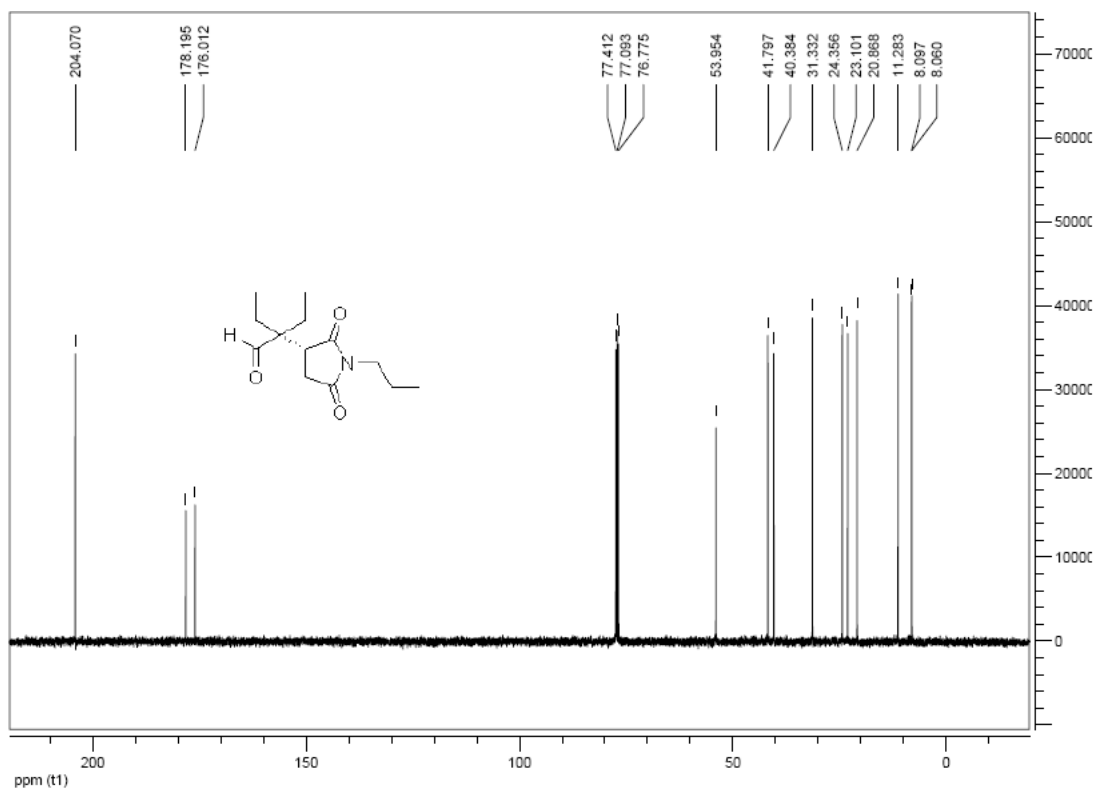
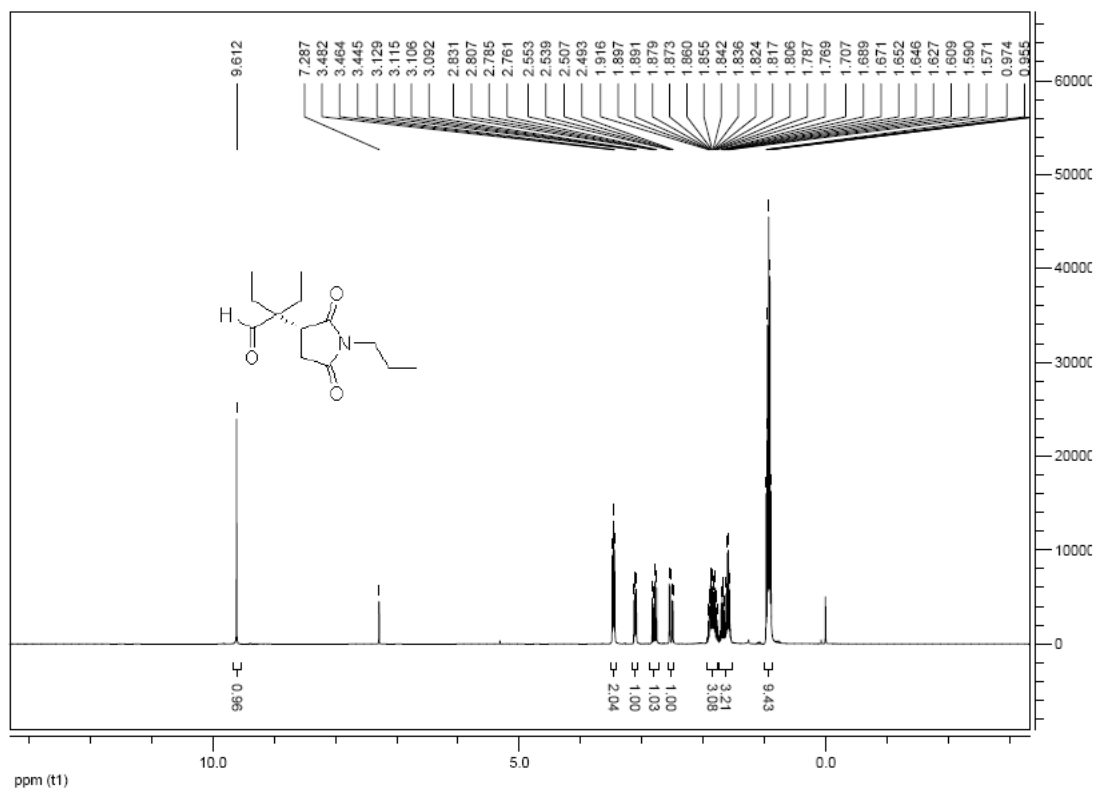
2-(2,5-dioxo-1-propylpyrrolidin-3-yl)-2-methylpropanal (3i)



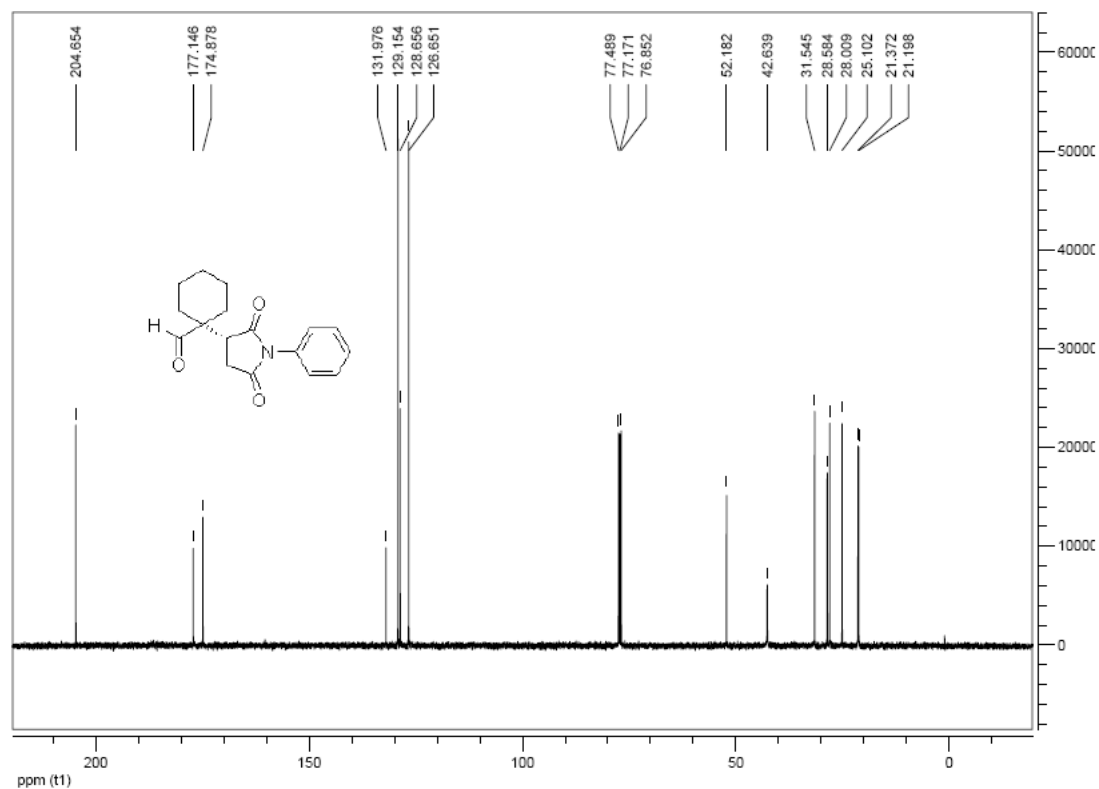
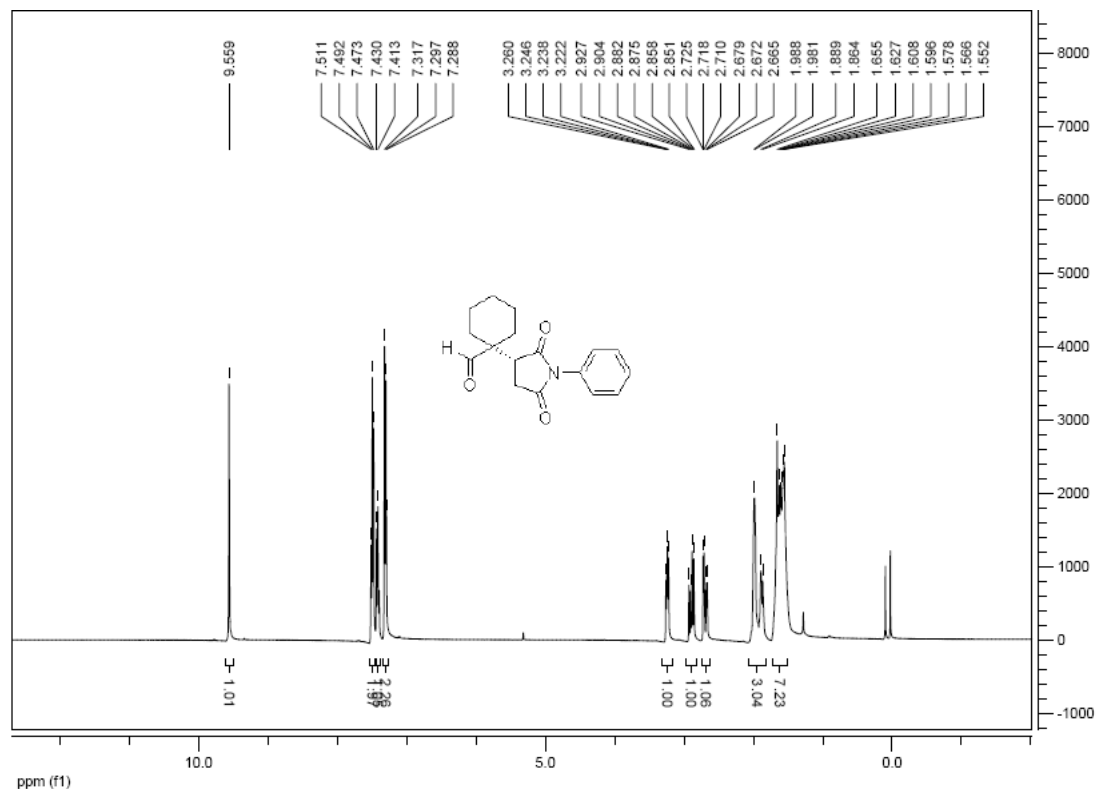
2-(1-isopropyl-2,5-dioxypyrrolidin-3-yl)-2-methylpropanal (3j)



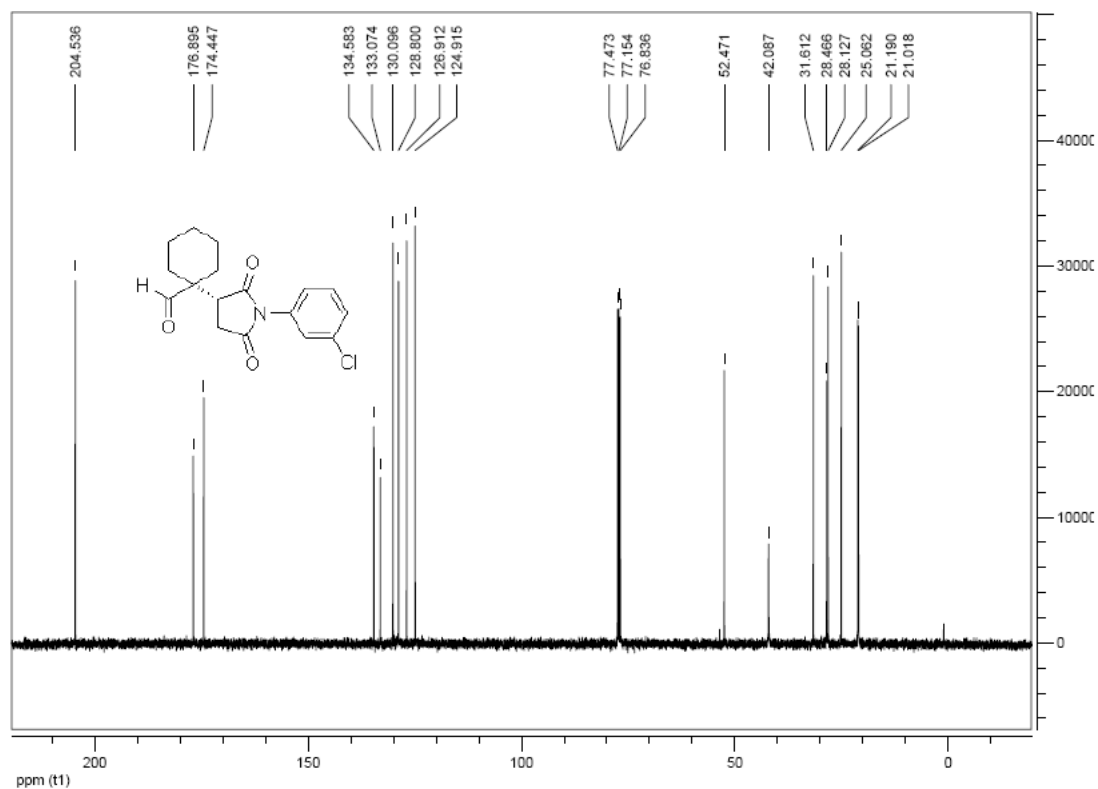
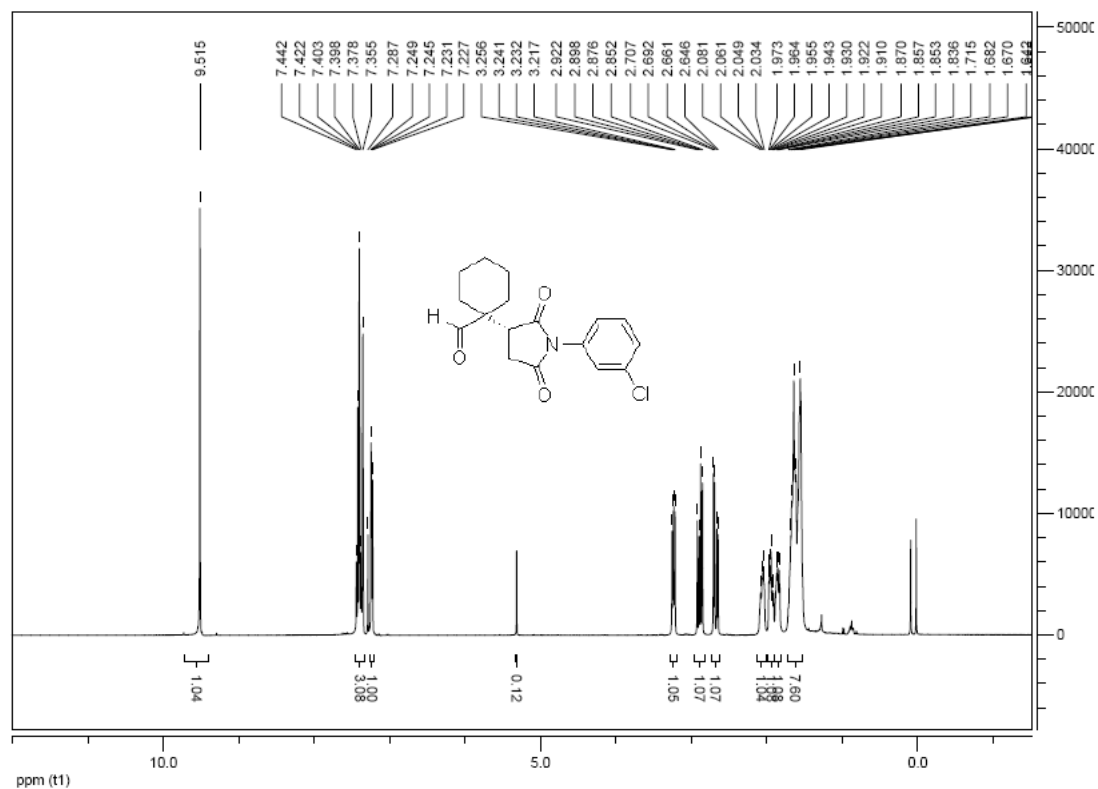
2-(2,5-dioxo-1-propylpyrrolidin-3-yl)-2-ethylbutanal (3k)



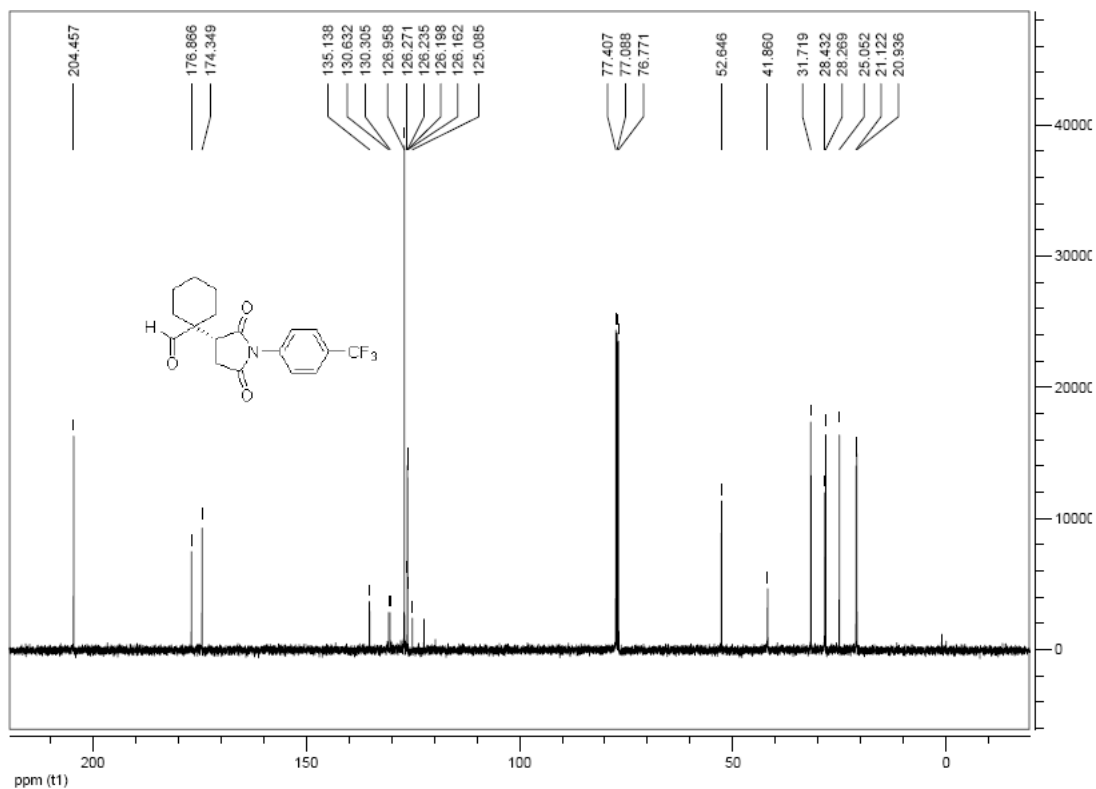
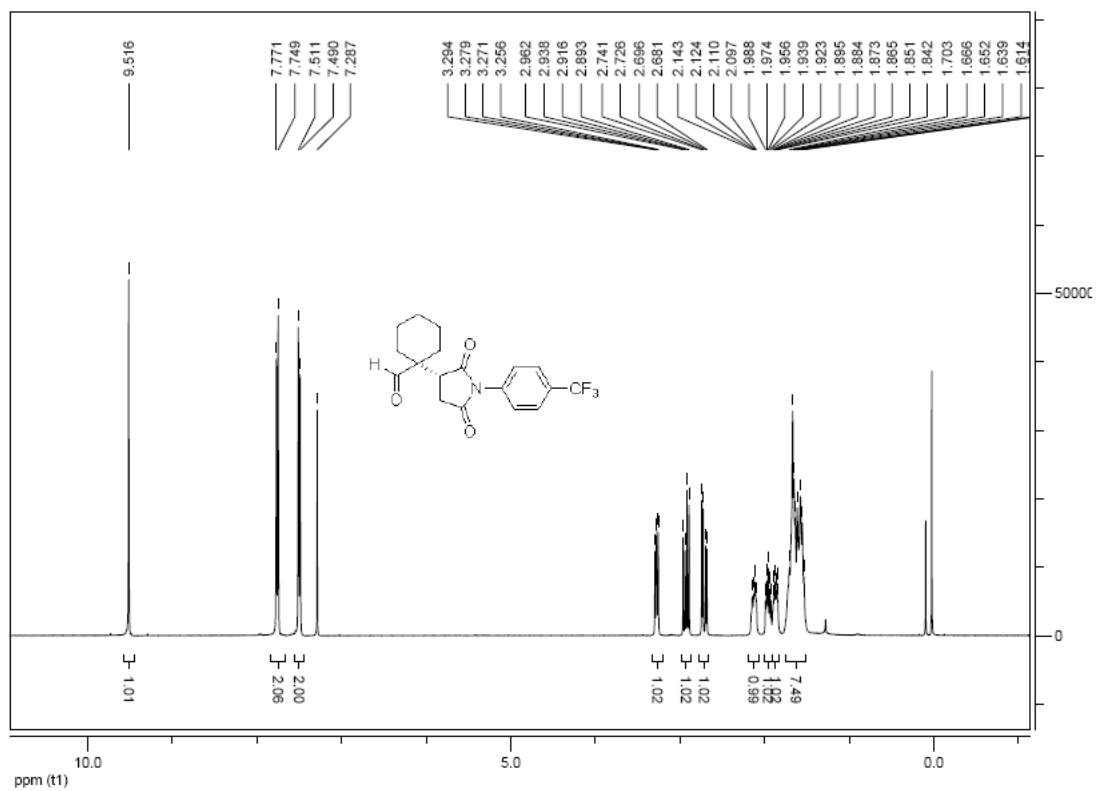
1-(2,5-dioxo-1-phenylpyrrolidin-3-yl)cyclohexanecarbaldehyde (3l)



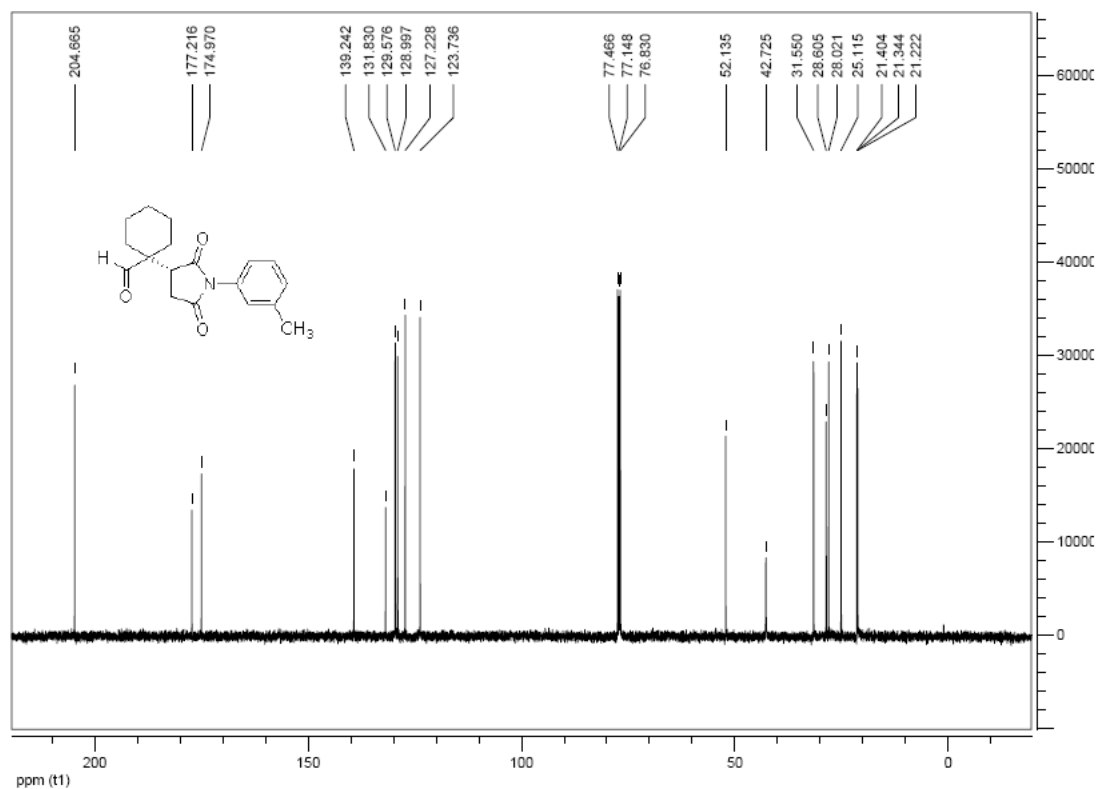
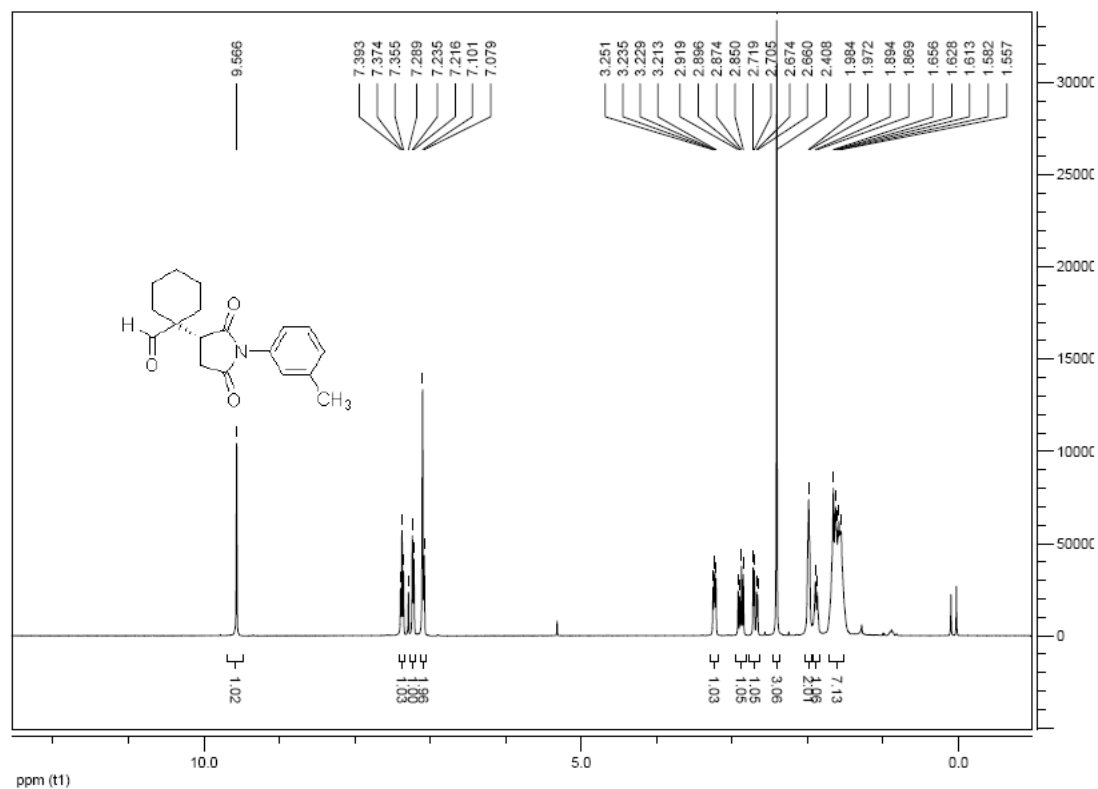
1-(1-(3-chlorophenyl)-2,5-dioxopyrrolidin-3-yl)cyclohexanecarbaldehyde (3m)



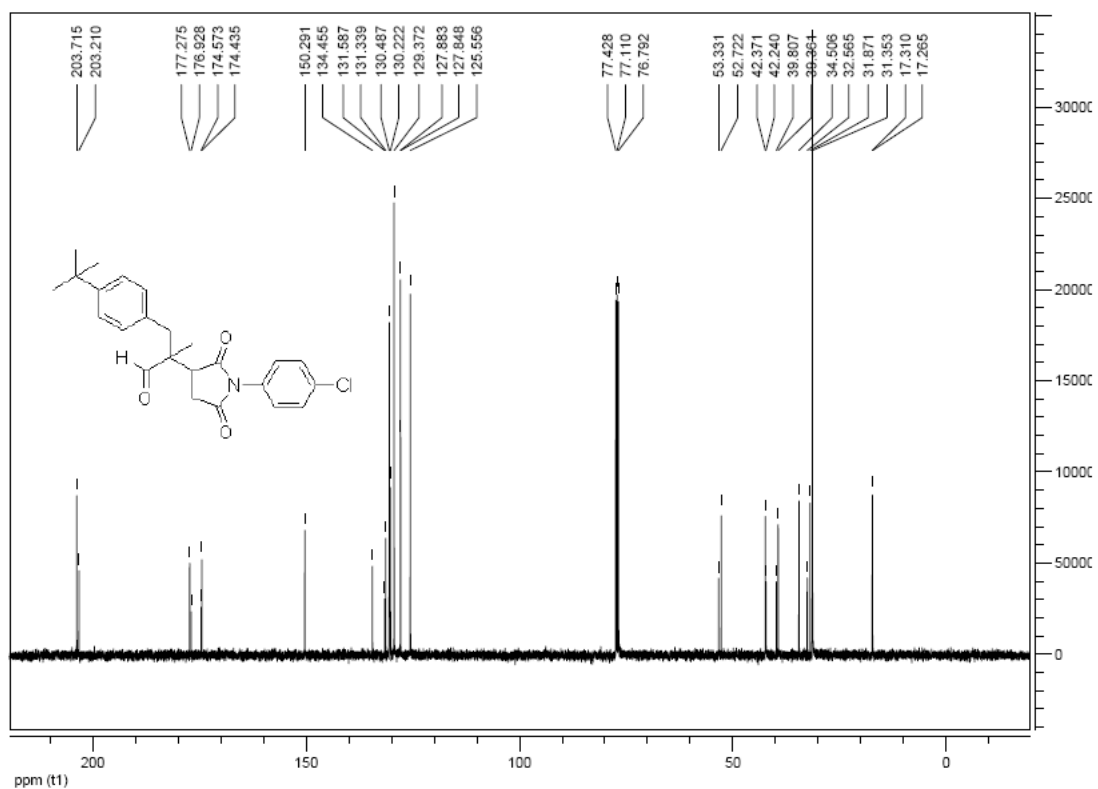
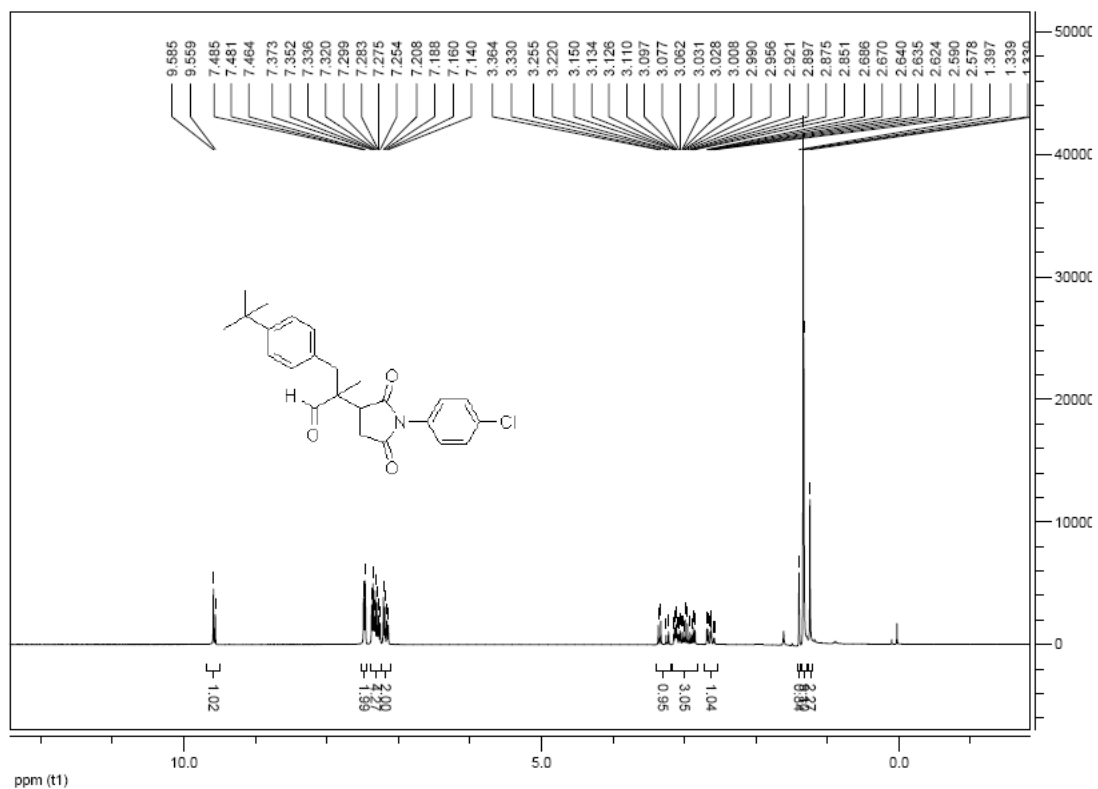
1-(2,5-dioxo-1-(4-(trifluoromethyl)phenyl)pyrrolidin-3-yl)cyclohexanecarbaldehyde (3n)



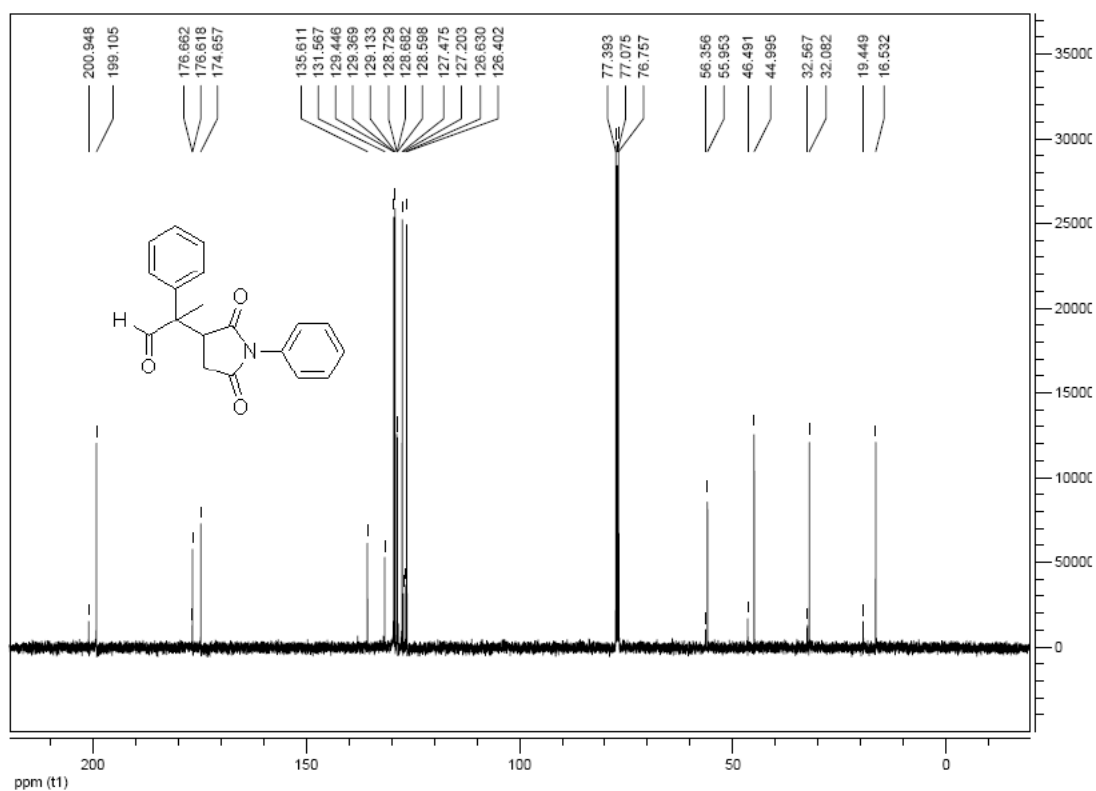
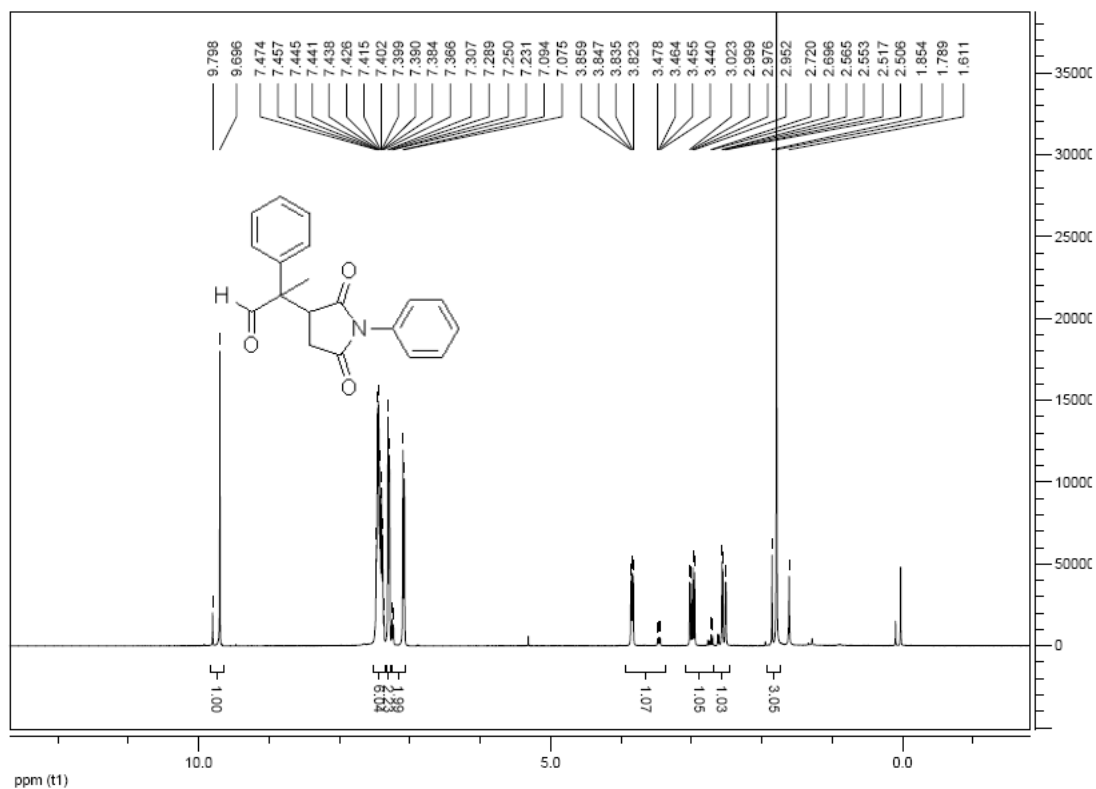
1-(2,5-dioxo-1-m-tolylpyrrolidin-3-yl)cyclohexanecarbaldehyde (3o)



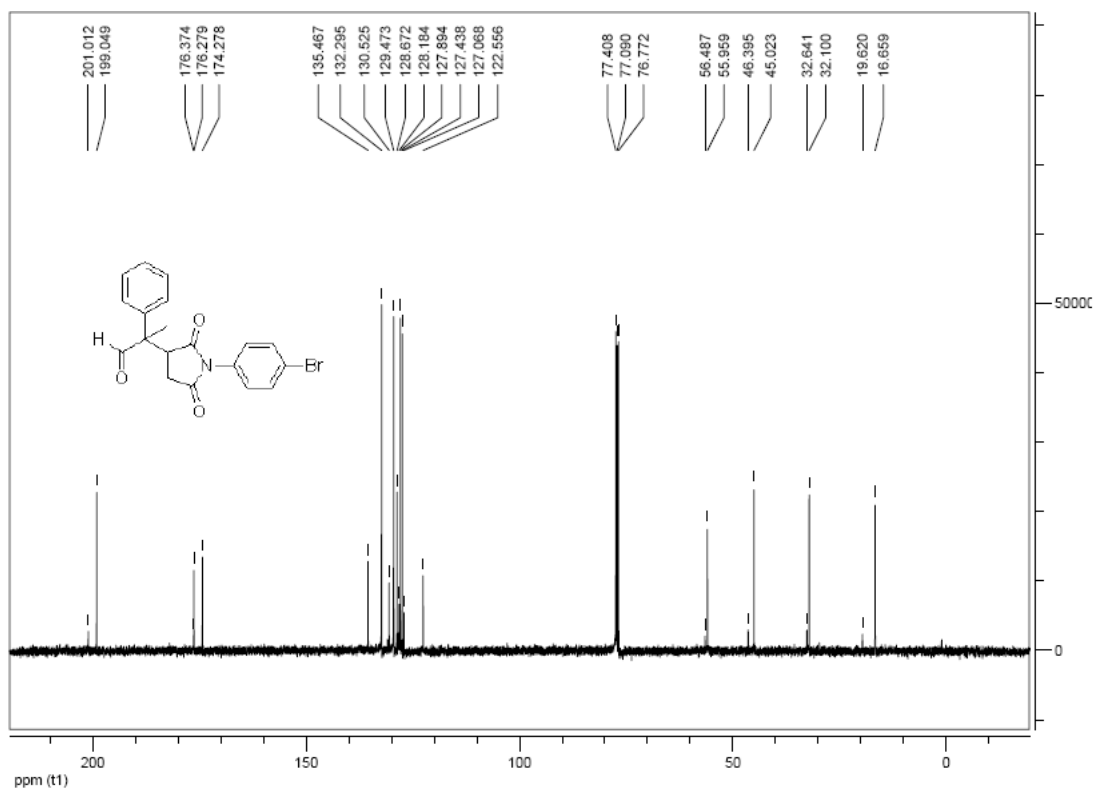
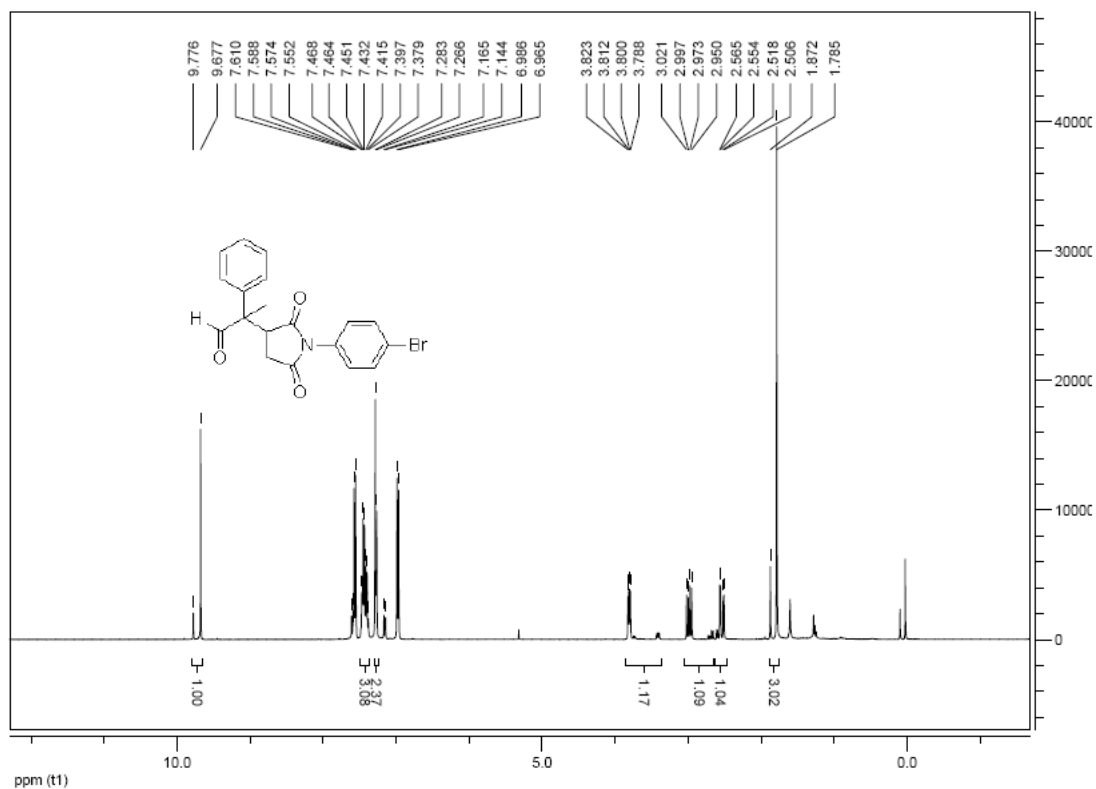
3-(4-tert-butylphenyl)-2-(1-(4-chlorophenyl)-2,5-dioxopyrrolidin-3-yl)-2-methylpropanal (7c)



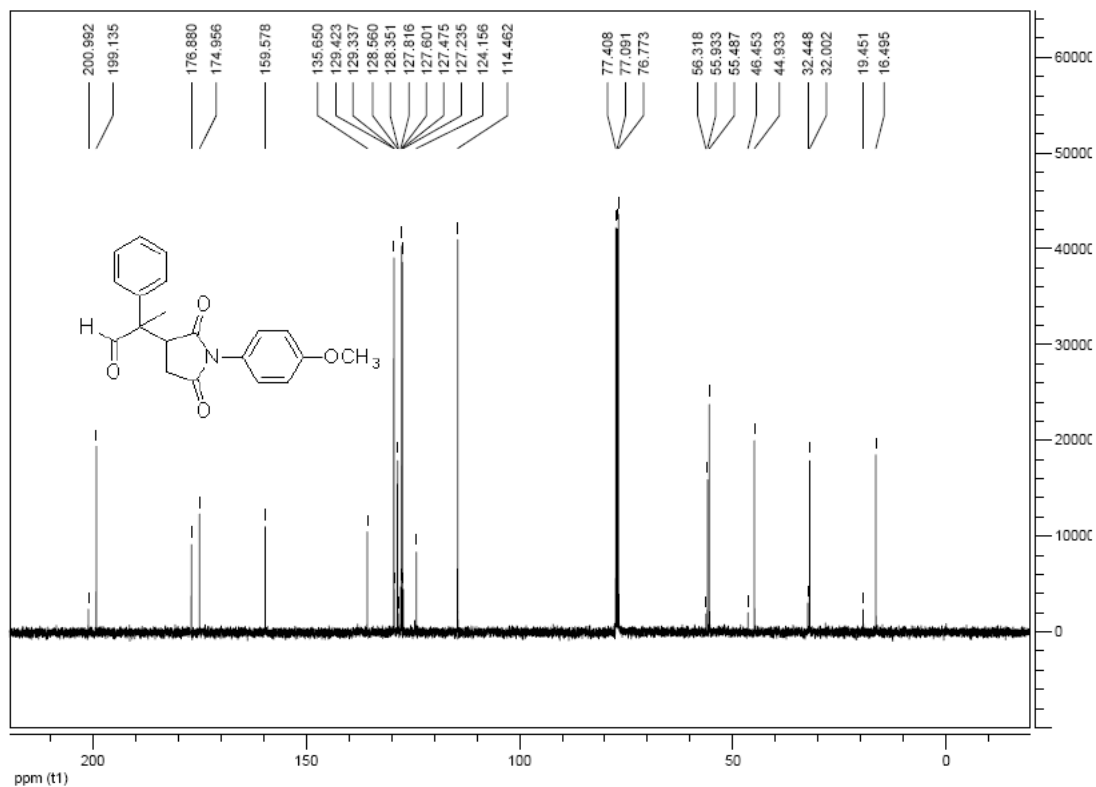
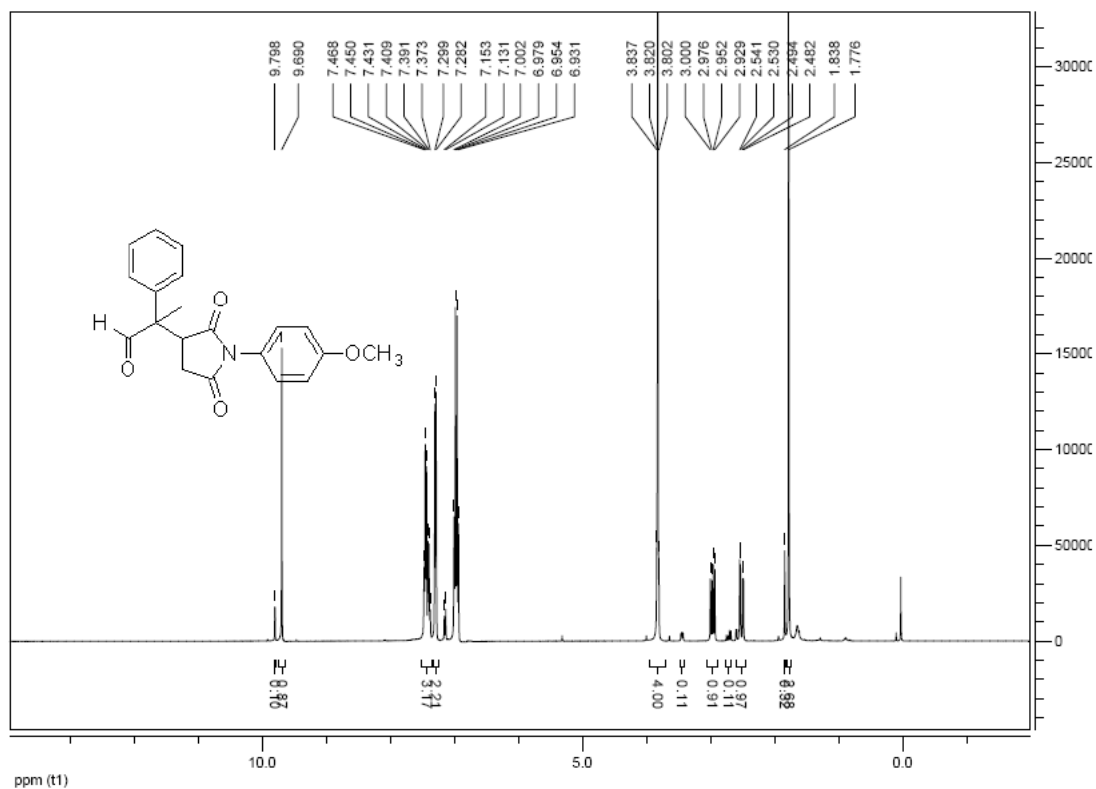
2-(2,5-dioxo-1-phenylpyrrolidin-3-yl)-2-phenylpropanal (7f)



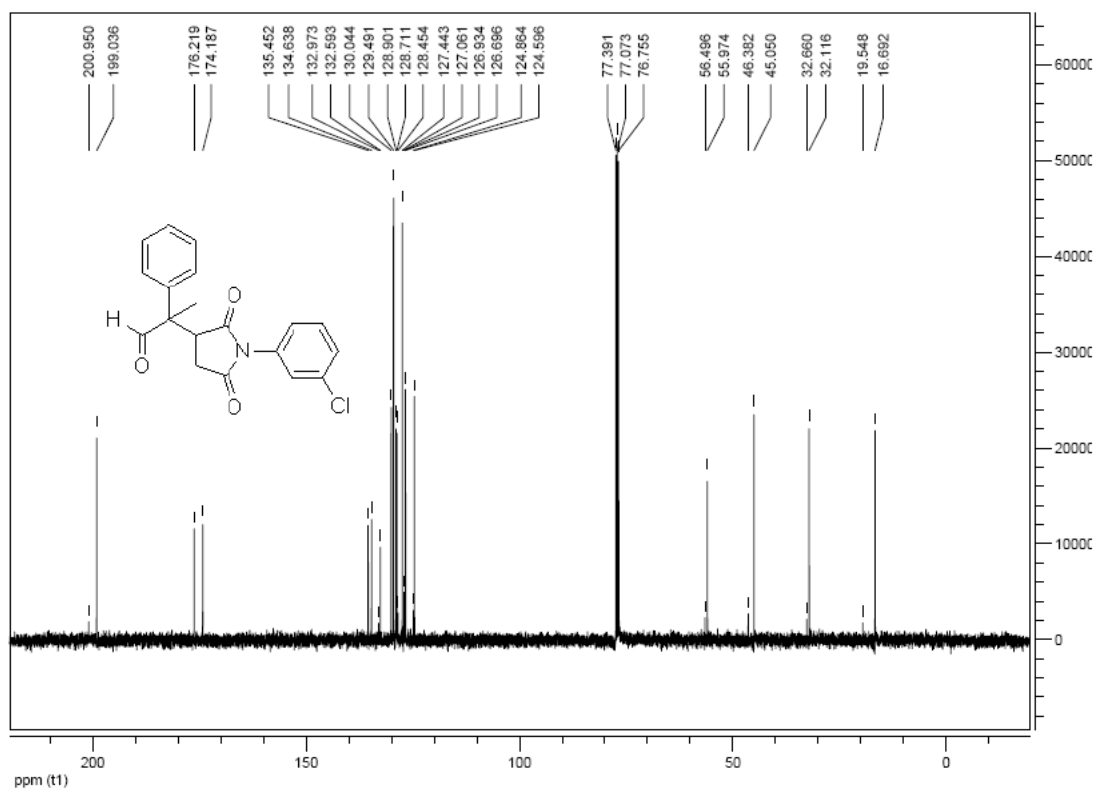
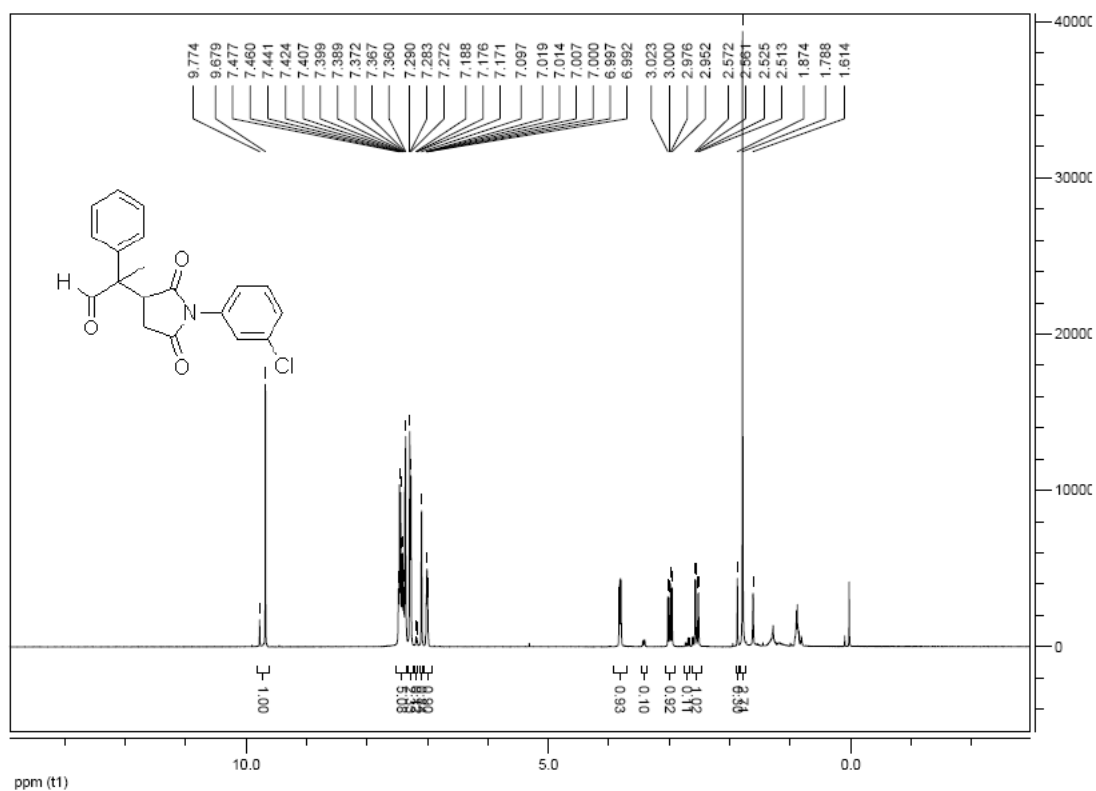
2-(1-(4-bromophenyl)-2,5-dioxopyrrolidin-3-yl)-2-phenylpropanal (7g)



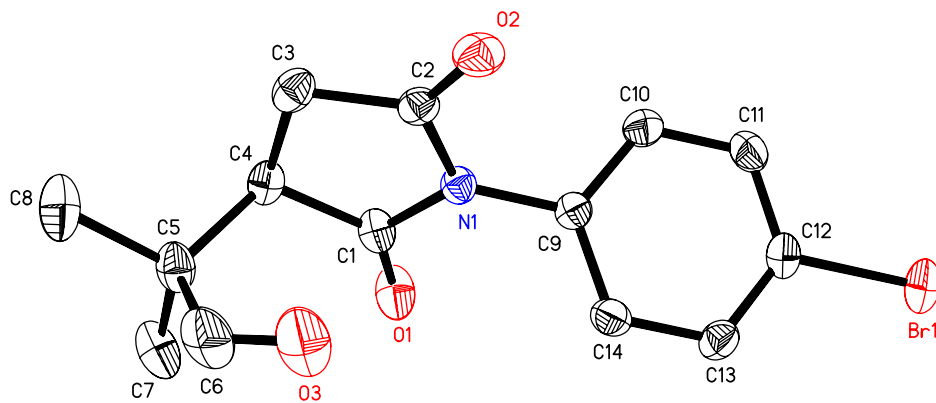
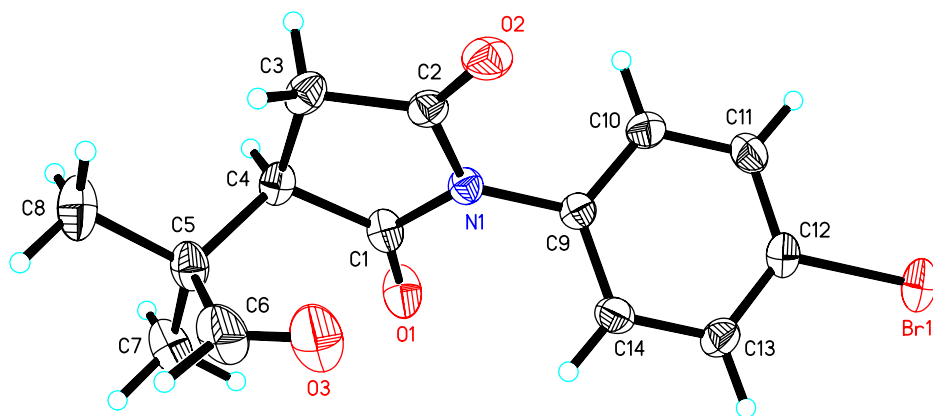
2-(1-(4-methoxyphenyl)-2,5-dioxopyrrolidin-3-yl)-2-phenylpropanal (7h)



2-(1-(3-chlorophenyl)-2,5-dioxopyrrolidin-3-yl)-2-phenylpropanal (7i)



D: Determination of Absolute Configuration



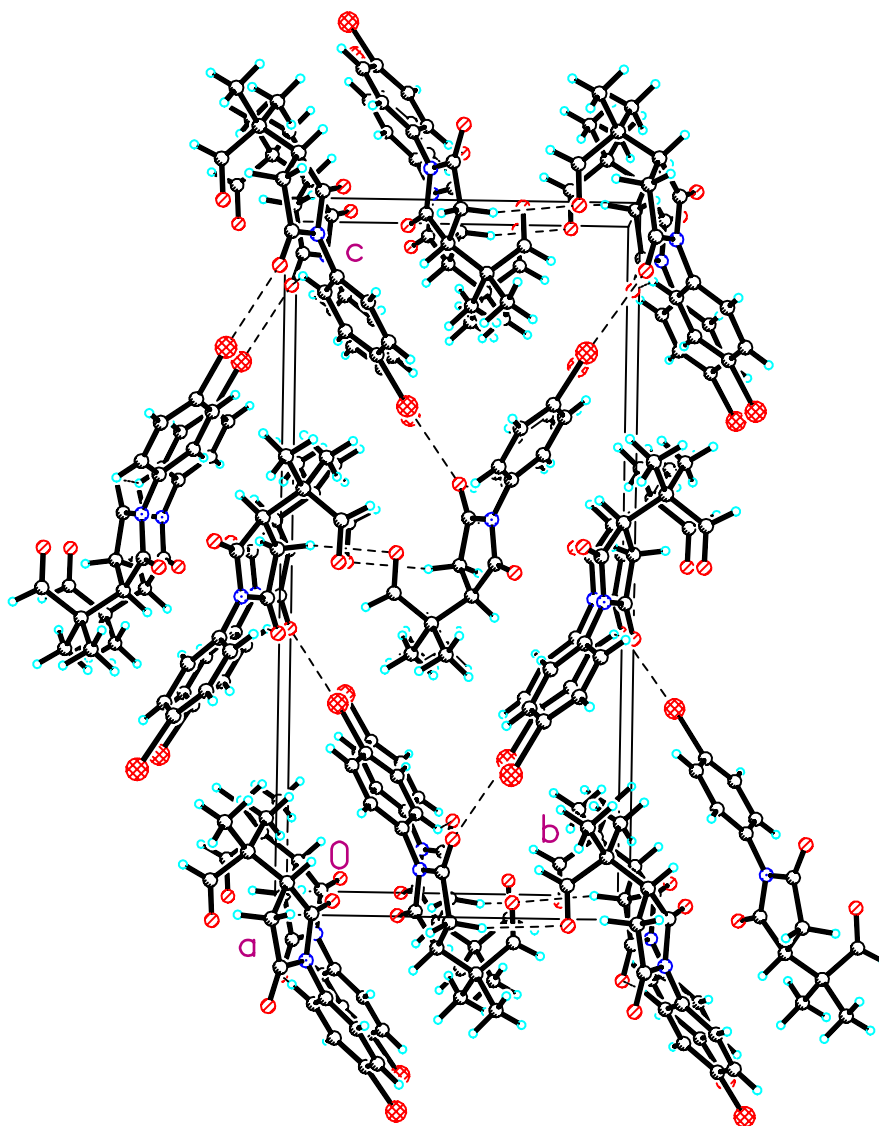


Table 1. Crystal data and structure refinement for cd201259.

Identification code	cd201259
Empirical formula	C ₁₄ H ₁₄ Br N O ₃
Formula weight	324.17
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	Orthorhombic, P2(1)2(1)2(1)
Unit cell dimensions	a = 6.607(3) Å alpha = 90 deg. b = 10.212(4) Å beta = 90 deg. c = 20.641(8) Å gamma = 90 deg.
Volume	1392.7(9) Å ³
Z, Calculated density	4, 1.546 Mg/m ³

Absorption coefficient	2.954 mm ⁻¹
F(000)	656
Crystal size	0.367 x 0.351 x 0.237 mm
Theta range for data collection	1.97 to 26.00 deg.
Limiting indices	-8<=h<=8, -12<=k<=12, -21<=l<=25
Reflections collected / unique	7653 / 2719 [R(int) = 0.0766]
Completeness to theta = 26.00	99.6 %
Absorption correction	Empirical
Max. and min. transmission	1.00000 and 0.50343
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	2719 / 0 / 175

Goodness-of-fit on F^2	0.913
Final R indices [$I > 2\sigma(I)$]	R1 = 0.0398, wR2 = 0.0822
R indices (all data)	R1 = 0.0511, wR2 = 0.0850
Absolute structure parameter	0.022(13)
Extinction coefficient	0.0075(11)
Largest diff. peak and hole	0.446 and -0.389 e.Å ⁻³

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for cd201259.

U(eq) is defined as one third of the trace of the orthogonalized

Uij tensor.

	x	y	z	U(eq)
Br(1)	12370(1)	8484(1)	7849(1)	60(1)
N(1)	8159(4)	5950(3)	5561(1)	34(1)
O(1)	10502(4)	6604(3)	4817(1)	58(1)
O(2)	5445(4)	5017(3)	6075(1)	50(1)
O(3)	9324(6)	3257(3)	5069(2)	87(1)
C(1)	8934(6)	6066(3)	4938(2)	40(1)
C(2)	6328(5)	5303(4)	5585(2)	36(1)
C(3)	5663(6)	5065(3)	4904(2)	44(1)
C(4)	7459(6)	5418(3)	4474(1)	38(1)
C(5)	8322(6)	4282(4)	4079(2)	47(1)
C(6)	9084(8)	3209(4)	4505(2)	70(1)

C(7)	10151(8)	4711(4)	3659(2)	71(1)
C(8)	6705(7)	3700(4)	3634(2)	70(1)
C(9)	9144(5)	6490(3)	6120(2)	34(1)
C(10)	8133(6)	7399(4)	6488(2)	44(1)
C(11)	9083(6)	7970(4)	7012(2)	50(1)
C(12)	11014(6)	7614(3)	7153(2)	42(1)
C(13)	12027(5)	6685(3)	6805(2)	43(1)
C(14)	11059(5)	6106(3)	6283(2)	41(1)

Table 3. Bond lengths [Å] and angles [deg] for cd201259.

Br(1)-C(12)	1.912(4)
N(1)-C(2)	1.380(4)
N(1)-C(1)	1.388(4)
N(1)-C(9)	1.435(4)
O(1)-C(1)	1.199(4)
O(2)-C(2)	1.204(4)
O(3)-C(6)	1.176(5)
C(1)-C(4)	1.518(5)
C(2)-C(3)	1.492(5)
C(3)-C(4)	1.525(5)
C(3)-H(3A)	0.9700
C(3)-H(3B)	0.9700
C(4)-C(5)	1.528(5)
C(4)-H(4)	0.9800
C(5)-C(6)	1.492(6)
C(5)-C(8)	1.530(5)
C(5)-C(7)	1.550(6)
C(6)-H(6)	0.9300

C(7)-H(7A)	0.9600
C(7)-H(7B)	0.9600
C(7)-H(7C)	0.9600
C(8)-H(8A)	0.9600
C(8)-H(8B)	0.9600
C(8)-H(8C)	0.9600
C(9)-C(14)	1.366(5)
C(9)-C(10)	1.373(5)
C(10)-C(11)	1.378(5)
C(10)-H(10)	0.9300
C(11)-C(12)	1.359(5)
C(11)-H(11)	0.9300
C(12)-C(13)	1.365(5)
C(13)-C(14)	1.387(5)
C(13)-H(13)	0.9300
C(14)-H(14)	0.9300
C(2)-N(1)-C(1)	113.4(3)
C(2)-N(1)-C(9)	123.5(3)
C(1)-N(1)-C(9)	123.0(3)
O(1)-C(1)-N(1)	123.4(3)
O(1)-C(1)-C(4)	128.5(3)

N(1)-C(1)-C(4)	108.1(3)
O(2)-C(2)-N(1)	124.8(3)
O(2)-C(2)-C(3)	127.6(3)
N(1)-C(2)-C(3)	107.6(3)
C(2)-C(3)-C(4)	106.3(3)
C(2)-C(3)-H(3A)	110.5
C(4)-C(3)-H(3A)	110.5
C(2)-C(3)-H(3B)	110.5
C(4)-C(3)-H(3B)	110.5
H(3A)-C(3)-H(3B)	108.7
C(1)-C(4)-C(3)	103.6(3)
C(1)-C(4)-C(5)	115.3(3)
C(3)-C(4)-C(5)	114.9(3)
C(1)-C(4)-H(4)	107.5
C(3)-C(4)-H(4)	107.5
C(5)-C(4)-H(4)	107.5
C(6)-C(5)-C(4)	111.7(3)
C(6)-C(5)-C(8)	107.7(3)
C(4)-C(5)-C(8)	110.8(3)
C(6)-C(5)-C(7)	105.9(4)
C(4)-C(5)-C(7)	112.0(3)
C(8)-C(5)-C(7)	108.5(3)

O(3)-C(6)-C(5)	126.7(4)
O(3)-C(6)-H(6)	116.7
C(5)-C(6)-H(6)	116.7
C(5)-C(7)-H(7A)	109.5
C(5)-C(7)-H(7B)	109.5
H(7A)-C(7)-H(7B)	109.5
C(5)-C(7)-H(7C)	109.5
H(7A)-C(7)-H(7C)	109.5
H(7B)-C(7)-H(7C)	109.5
C(5)-C(8)-H(8A)	109.5
C(5)-C(8)-H(8B)	109.5
H(8A)-C(8)-H(8B)	109.5
C(5)-C(8)-H(8C)	109.5
H(8A)-C(8)-H(8C)	109.5
H(8B)-C(8)-H(8C)	109.5
C(14)-C(9)-C(10)	120.5(3)
C(14)-C(9)-N(1)	120.5(3)
C(10)-C(9)-N(1)	119.0(3)
C(9)-C(10)-C(11)	119.9(3)
C(9)-C(10)-H(10)	120.1
C(11)-C(10)-H(10)	120.1
C(12)-C(11)-C(10)	118.9(4)

C(12)-C(11)-H(11)	120.5
C(10)-C(11)-H(11)	120.5
C(11)-C(12)-C(13)	122.2(3)
C(11)-C(12)-Br(1)	118.6(3)
C(13)-C(12)-Br(1)	119.2(3)
C(12)-C(13)-C(14)	118.6(3)
C(12)-C(13)-H(13)	120.7
C(14)-C(13)-H(13)	120.7
C(9)-C(14)-C(13)	119.7(3)
C(9)-C(14)-H(14)	120.1
C(13)-C(14)-H(14)	120.1

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{Å}^2 \times 10^3$) for cd201259.

The anisotropic displacement factor exponent takes the form:

$$-2 \pi^2 [h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12}]$$

	U11	U22	U33	U23	U13	U12
Br(1)	78(1)	61(1)	41(1)	-5(1)	-14(1)	-8(1)
N(1)	35(2)	32(2)	35(2)	-2(1)	0(1)	-2(1)
O(1)	66(2)	62(2)	45(2)	-6(1)	12(1)	-32(2)
O(2)	39(2)	59(2)	52(2)	8(1)	7(1)	-5(1)
O(3)	147(3)	51(2)	62(2)	3(2)	-21(2)	40(2)
C(1)	51(2)	31(2)	37(2)	1(2)	4(2)	-7(2)
C(2)	36(2)	32(2)	41(2)	3(2)	-3(2)	5(2)
C(3)	44(2)	36(2)	51(2)	2(2)	-11(2)	-3(2)
C(4)	51(2)	28(2)	36(2)	5(1)	-9(2)	-1(2)
C(5)	67(3)	31(2)	42(2)	-2(2)	-5(2)	-2(2)
C(6)	109(4)	30(2)	71(3)	-3(2)	-3(3)	15(2)
C(7)	93(4)	63(3)	56(3)	-15(2)	16(3)	-5(3)

C(8)	96(4)	61(3)	51(3)	-10(2)	-6(2)	-24(3)
C(9)	35(2)	29(2)	38(2)	-1(2)	-1(1)	-2(2)
C(10)	42(2)	46(2)	45(2)	-3(2)	-6(2)	10(2)
C(11)	56(3)	54(2)	41(2)	-11(2)	1(2)	15(2)
C(12)	56(2)	39(2)	32(2)	-1(2)	-5(2)	-3(2)
C(13)	37(2)	44(2)	46(2)	-2(2)	-8(2)	-3(2)
C(14)	41(2)	41(2)	42(2)	-2(2)	4(2)	6(2)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{Å}^2 \times 10^3$) for cd201259.

	x	y	z	U(eq)
H(3A)	4504	5608	4799	52
H(3B)	5292	4154	4845	52
H(4)	7002	6088	4168	46
H(6)	9403	2420	4304	84
H(7A)	11181	5076	3933	106
H(7B)	9719	5358	3351	106
H(7C)	10684	3966	3433	106
H(8A)	7254	2958	3409	104
H(8B)	6284	4349	3325	104
H(8C)	5562	3429	3887	104
H(10)	6810	7629	6385	53
H(11)	8413	8588	7263	60
H(13)	13340	6446	6917	51

H(14)	11710	5459	6044	50
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Table 6. Torsion angles [deg] for cd201259.

C(2)-N(1)-C(1)-O(1)	179.5(4)
C(9)-N(1)-C(1)-O(1)	1.4(5)
C(2)-N(1)-C(1)-C(4)	-0.1(4)
C(9)-N(1)-C(1)-C(4)	-178.2(3)
C(1)-N(1)-C(2)-O(2)	175.0(4)
C(9)-N(1)-C(2)-O(2)	-6.9(5)
C(1)-N(1)-C(2)-C(3)	-6.3(4)
C(9)-N(1)-C(2)-C(3)	171.9(3)
O(2)-C(2)-C(3)-C(4)	-171.4(4)
N(1)-C(2)-C(3)-C(4)	9.9(4)
O(1)-C(1)-C(4)-C(3)	-173.5(4)
N(1)-C(1)-C(4)-C(3)	6.1(4)
O(1)-C(1)-C(4)-C(5)	60.1(5)
N(1)-C(1)-C(4)-C(5)	-120.3(3)
C(2)-C(3)-C(4)-C(1)	-9.5(4)
C(2)-C(3)-C(4)-C(5)	117.2(3)
C(1)-C(4)-C(5)-C(6)	59.5(4)
C(3)-C(4)-C(5)-C(6)	-60.9(5)
C(1)-C(4)-C(5)-C(8)	179.6(3)

C(3)-C(4)-C(5)-C(8)	59.1(4)
C(1)-C(4)-C(5)-C(7)	-59.0(4)
C(3)-C(4)-C(5)-C(7)	-179.5(3)
C(4)-C(5)-C(6)-O(3)	-11.4(8)
C(8)-C(5)-C(6)-O(3)	-133.3(6)
C(7)-C(5)-C(6)-O(3)	110.7(6)
C(2)-N(1)-C(9)-C(14)	122.5(3)
C(1)-N(1)-C(9)-C(14)	-59.6(5)
C(2)-N(1)-C(9)-C(10)	-57.6(5)
C(1)-N(1)-C(9)-C(10)	120.3(4)
C(14)-C(9)-C(10)-C(11)	2.6(6)
N(1)-C(9)-C(10)-C(11)	-177.3(3)
C(9)-C(10)-C(11)-C(12)	-0.2(6)
C(10)-C(11)-C(12)-C(13)	-1.8(6)
C(10)-C(11)-C(12)-Br(1)	176.5(3)
C(11)-C(12)-C(13)-C(14)	1.3(6)
Br(1)-C(12)-C(13)-C(14)	-177.0(2)
C(10)-C(9)-C(14)-C(13)	-3.2(5)
N(1)-C(9)-C(14)-C(13)	176.7(3)
C(12)-C(13)-C(14)-C(9)	1.2(5)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for cd201259 [Å and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
C(3)-H(3B)...O(3)#1	0.97	2.55	3.506(5)	168.7
C(6)-H(6)...O(2)#2	0.93	2.70	3.619(5)	170.2
C(13)-H(13)...O(2)#3	0.93	2.66	3.205(5)	118.1
C(14)-H(14)...O(2)#3	0.93	2.51	3.134(5)	124.7

Symmetry transformations used to generate equivalent atoms:

#1 $x-1/2, -y+1/2, -z+1$ #2 $x+1/2, -y+1/2, -z+1$ #3 $x+1, y, z$