Supporting information

L-MALIC ACID AS CHIRAL AUXILIARY AND BUILDING BLOCK IN THE ASYMMETRIC SYNTHESIS OF (*S*)-(−)-CRISPINE A

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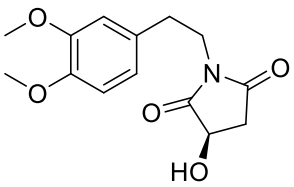
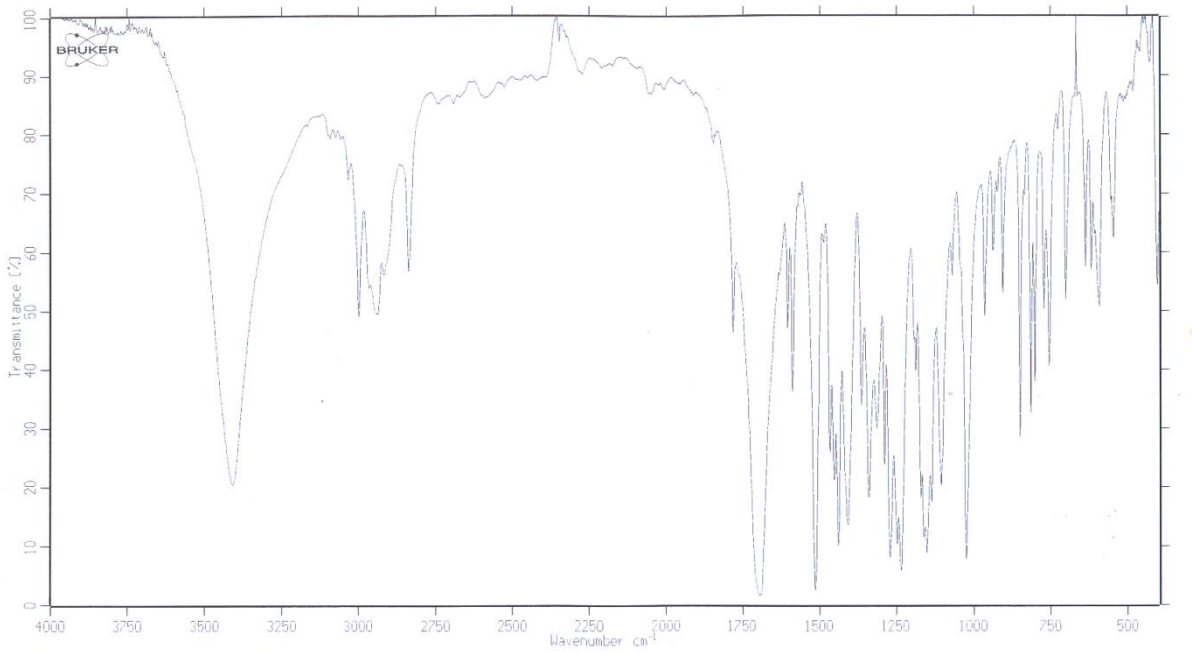
Contents

(3*R*)-(+)-1-(3,4-dimethoxyphenethyl)-3-hydroxypyrrolidine-2,5-dione 8

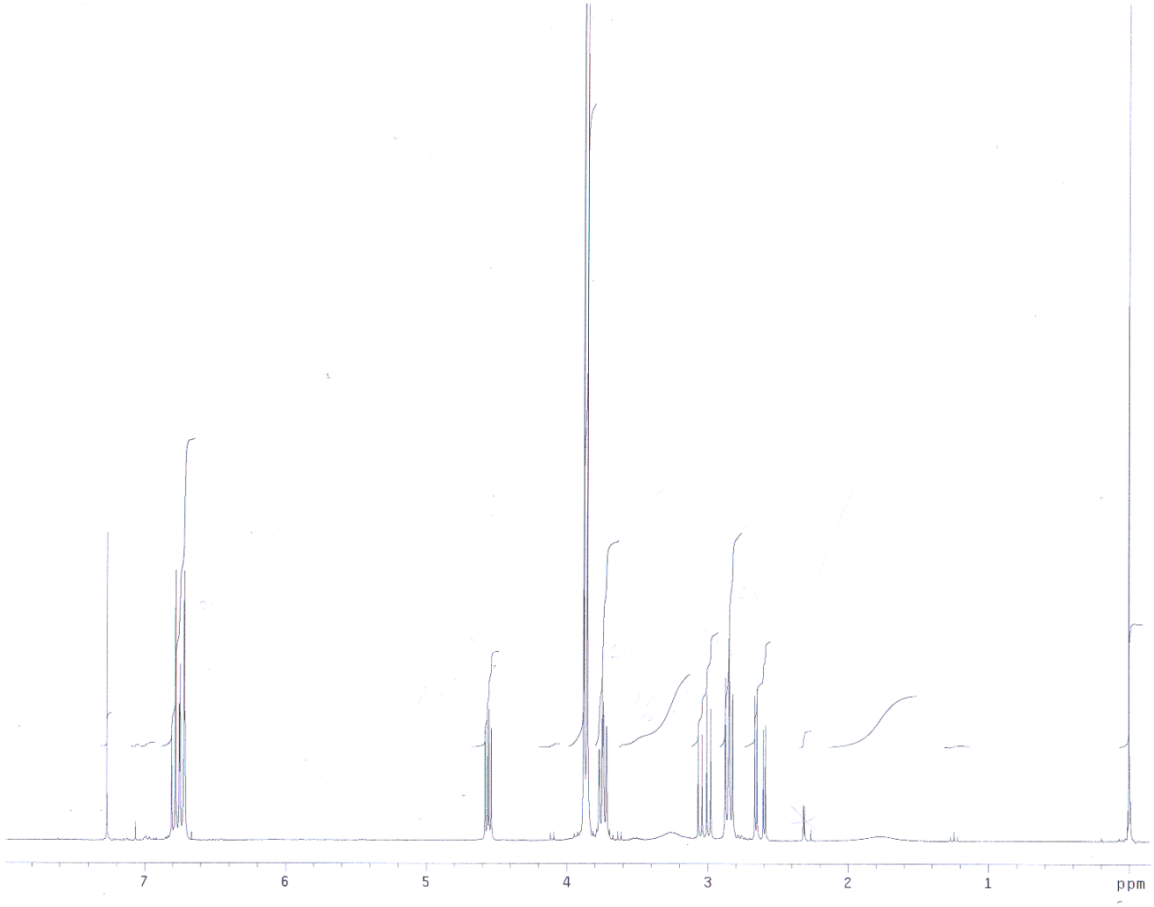


(+)-8

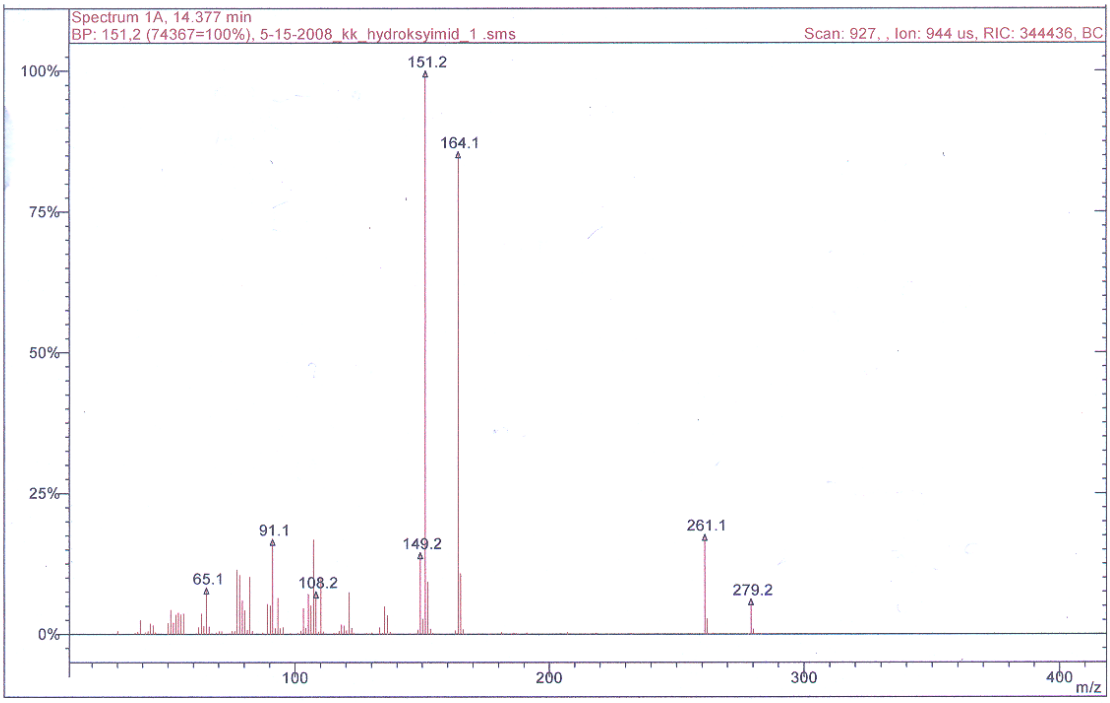
IR (KBr)



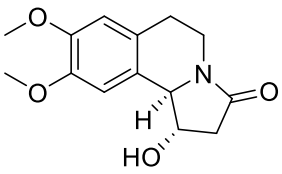
^{1}H NMR ($CDCl_3$)



MS

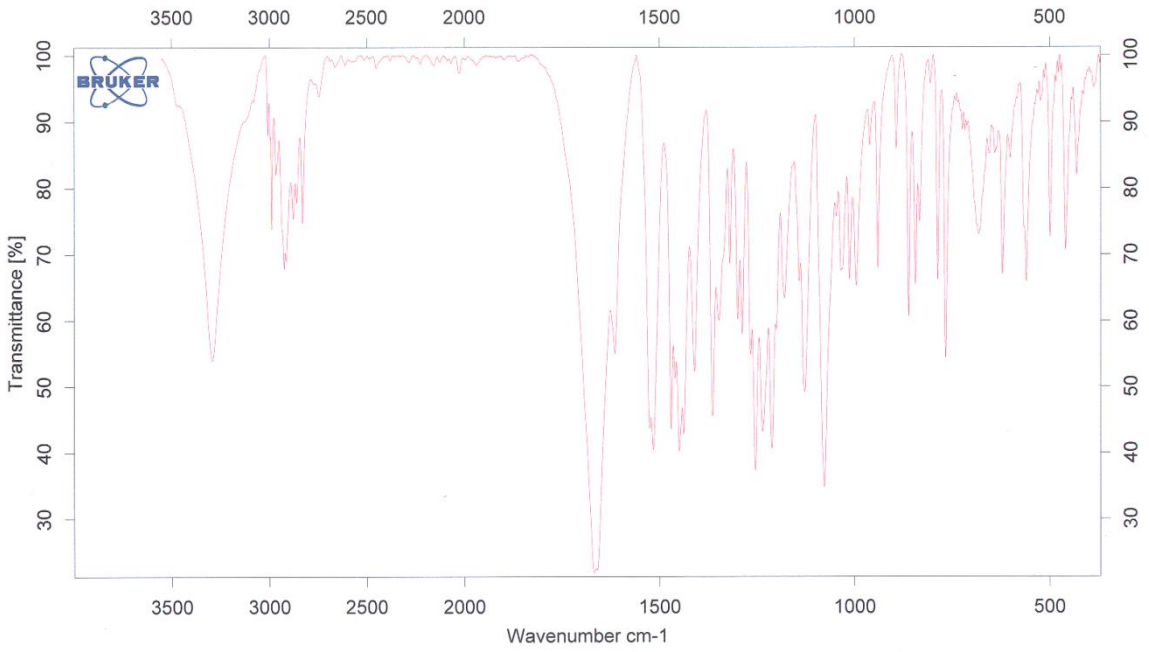


(1*S*,10b*R*)-(–)1-Hydroxy-8,9-dimethoxy-1,5,6,10b-tetrahydro-2H-pyrrolo[2,1-α]-isoquinolin-3-one 9

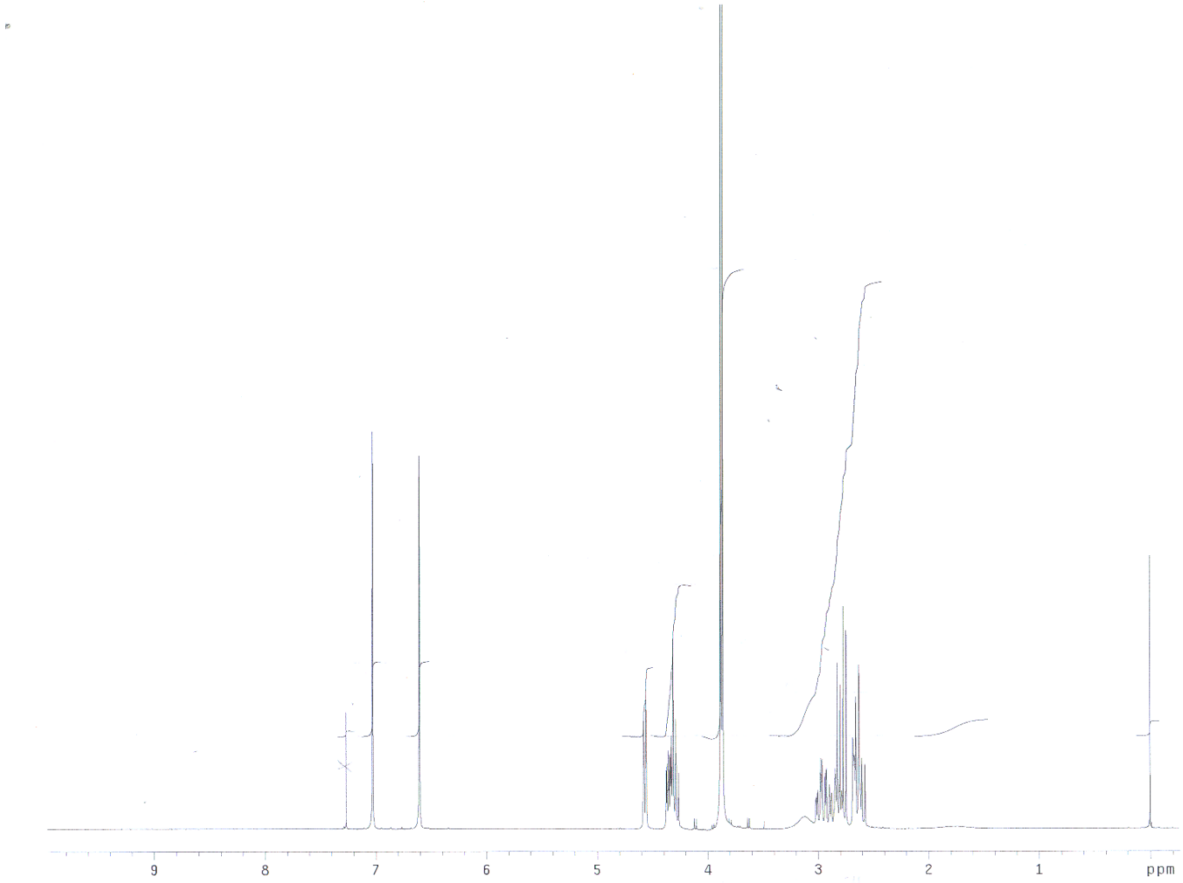


9

IR (KBr)



^{1}H NMR ($CDCl_3$)



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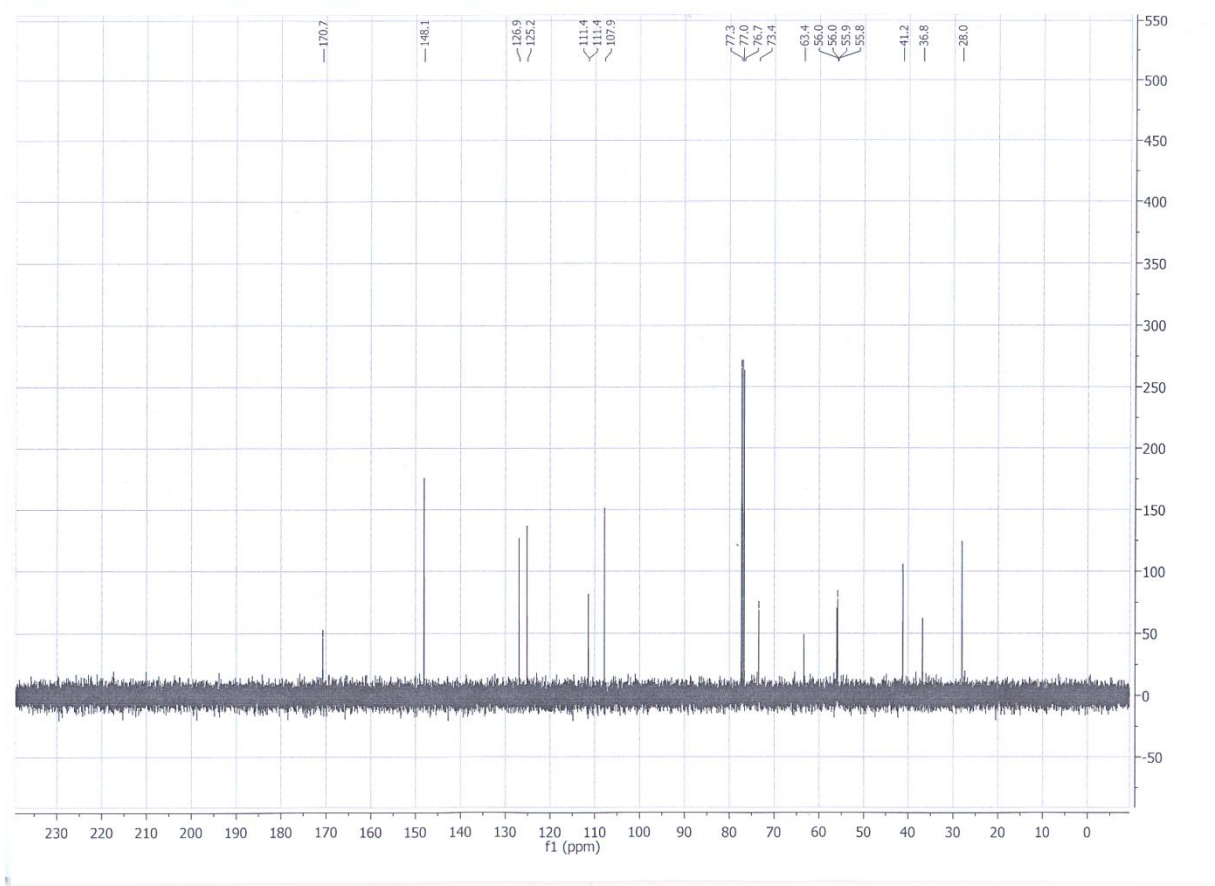

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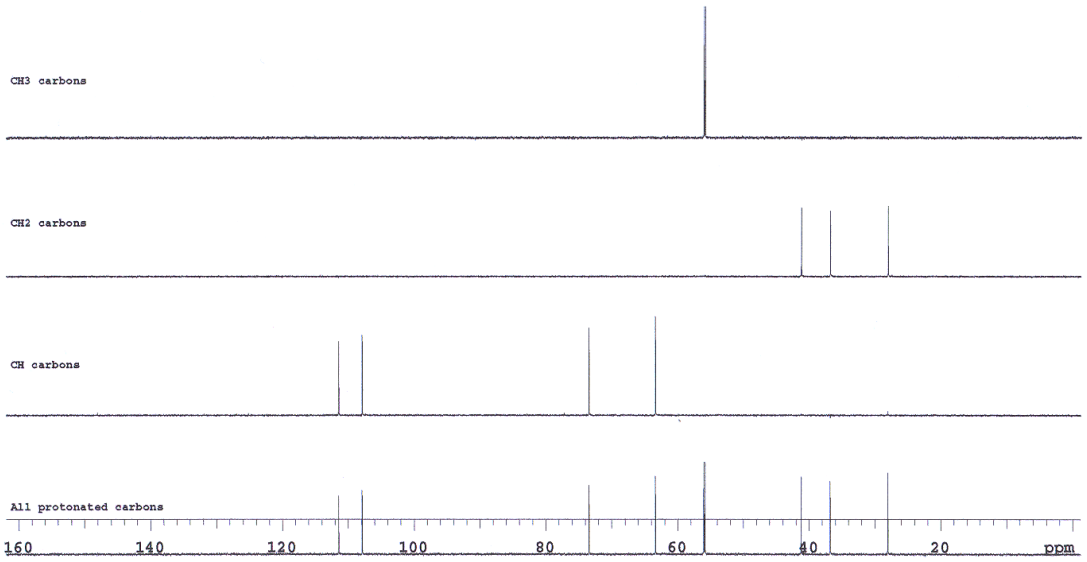
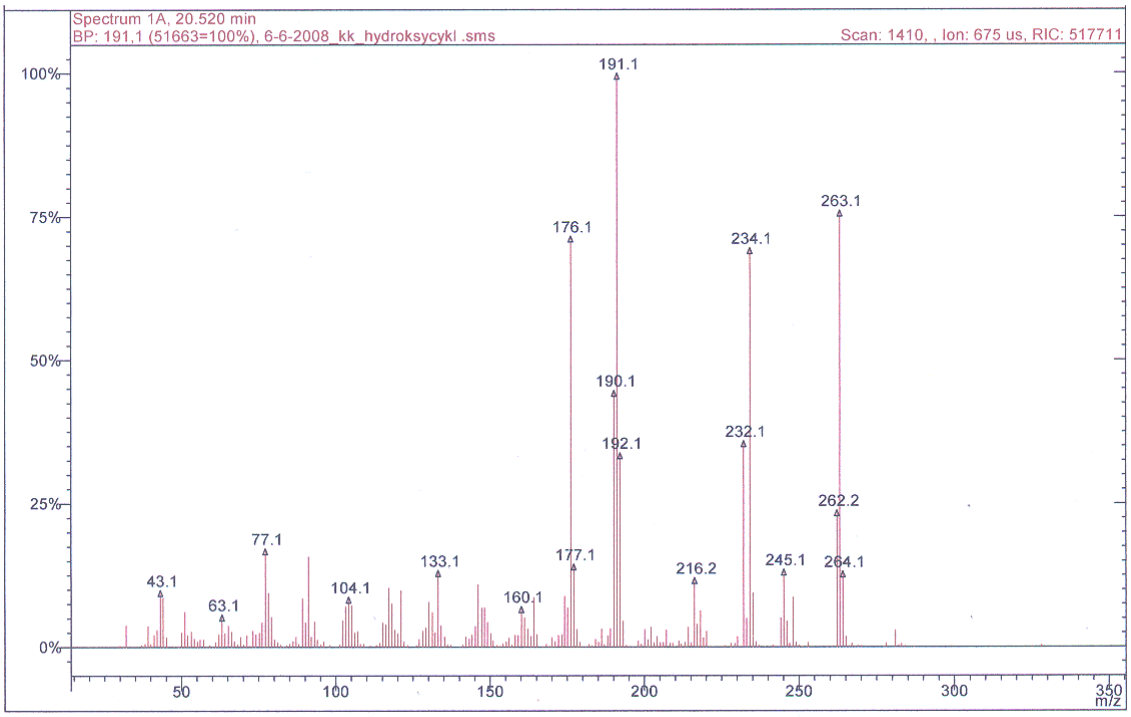
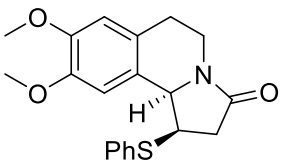


MS



(1*R*,10b*R*)-(–)-8,9-Dimethoxy-1-(phenylthio)-1,5,6,10b-tetrahydropyrrolo[2,1-α]-isoquinolin-3(2H)-one 10



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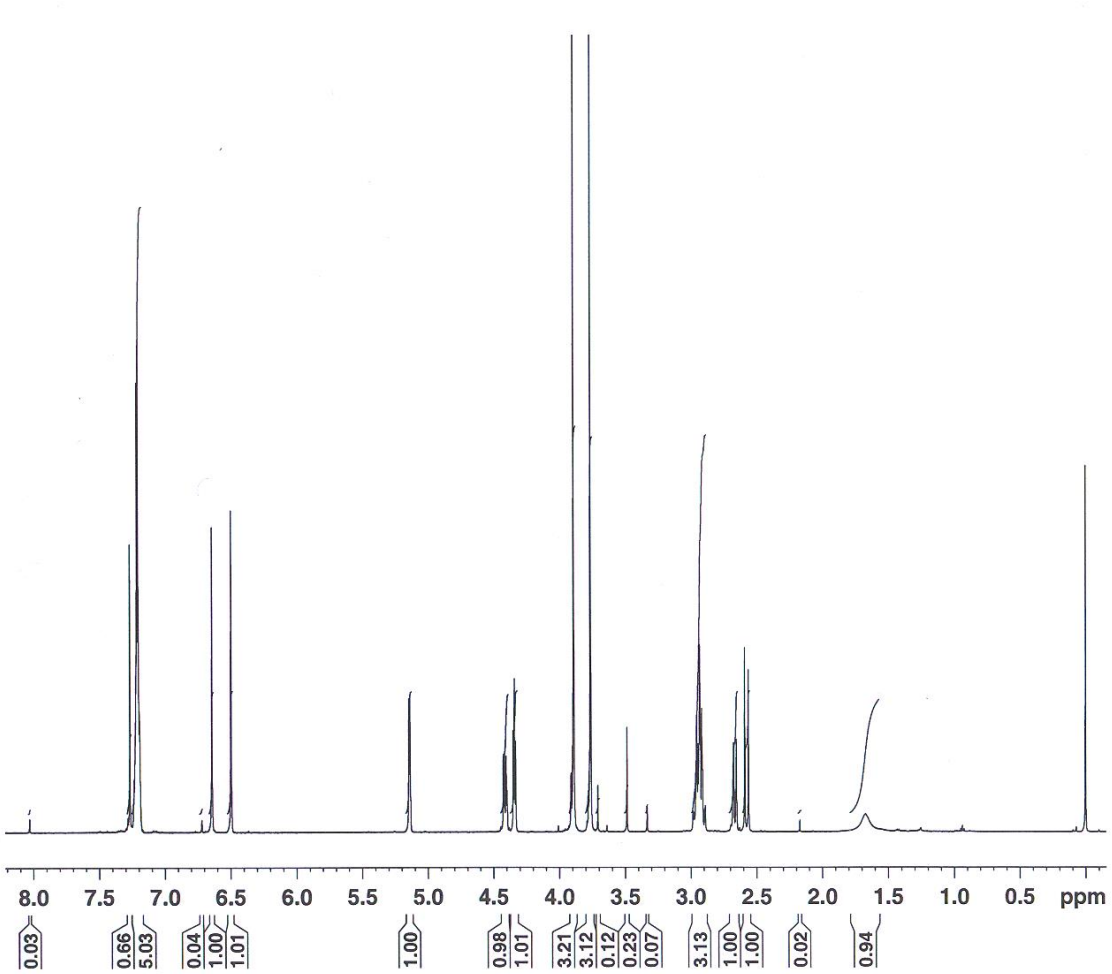
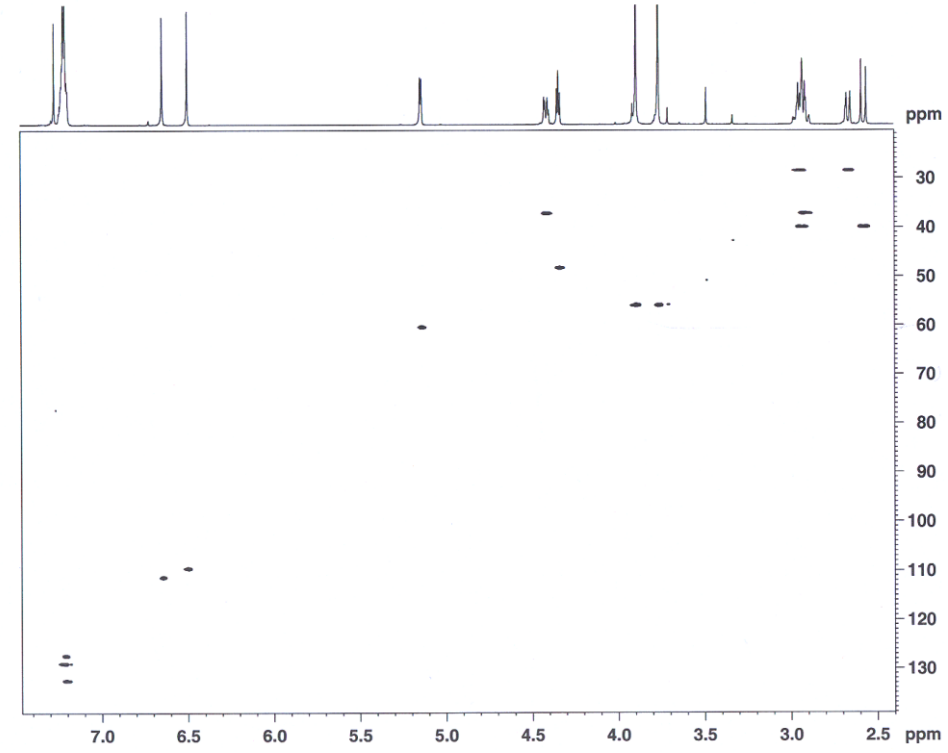
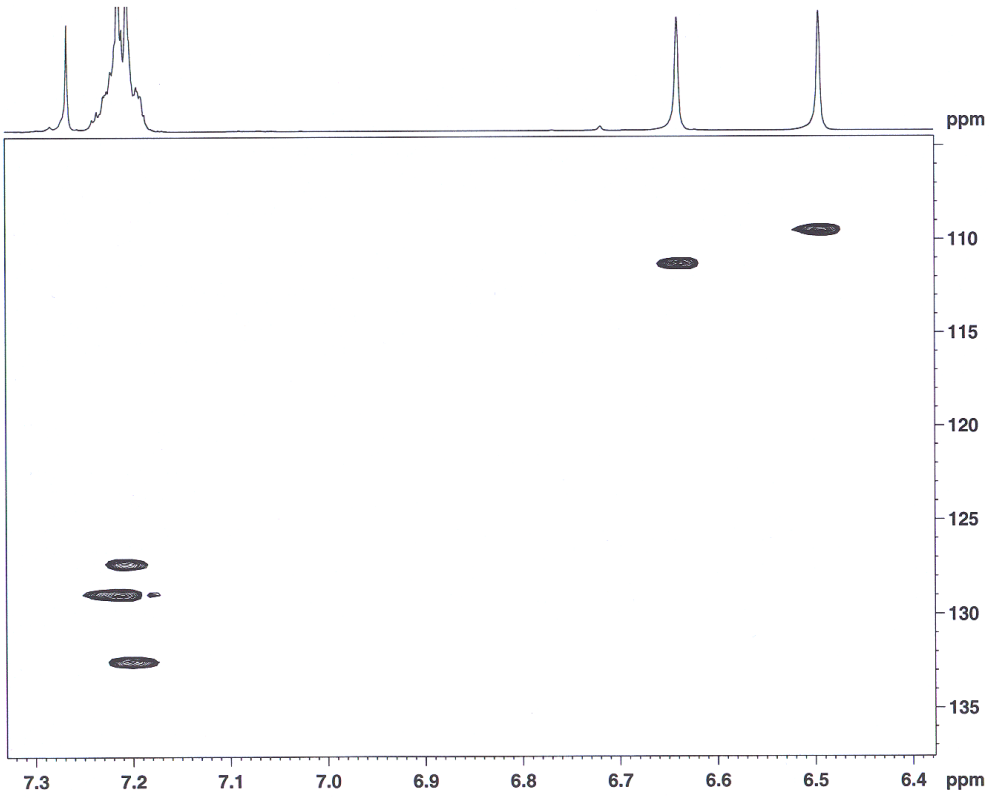
^{1}H NMR ($CDCl_3$)

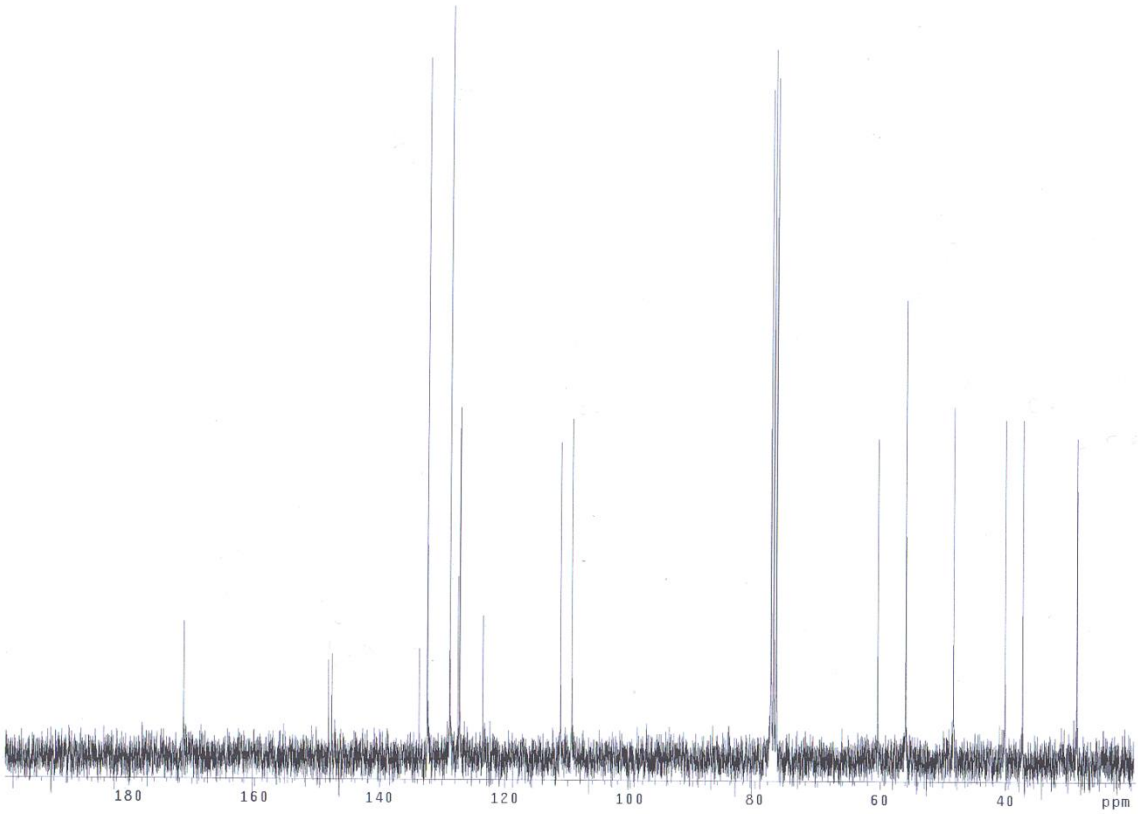


2D ^{1}H-^{13}C NMR ($CDCl_3$)

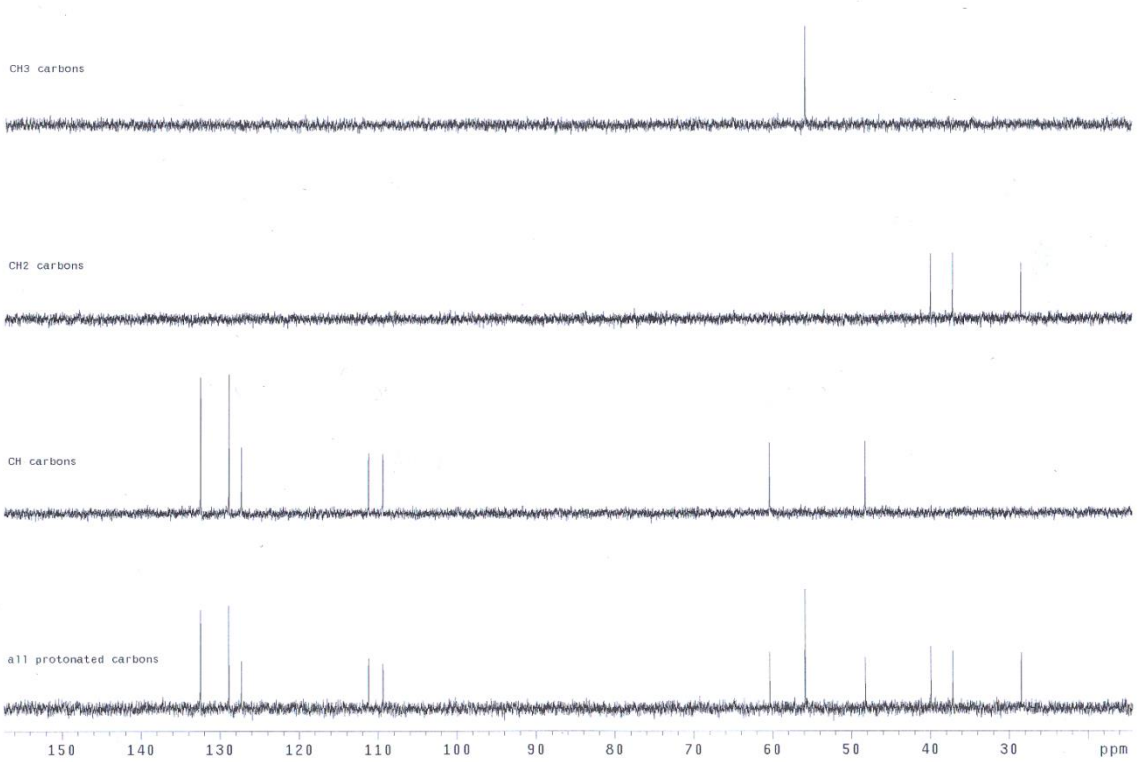




^{13}C NMR ($CDCl_3$)



^{13}C NMR DEPT



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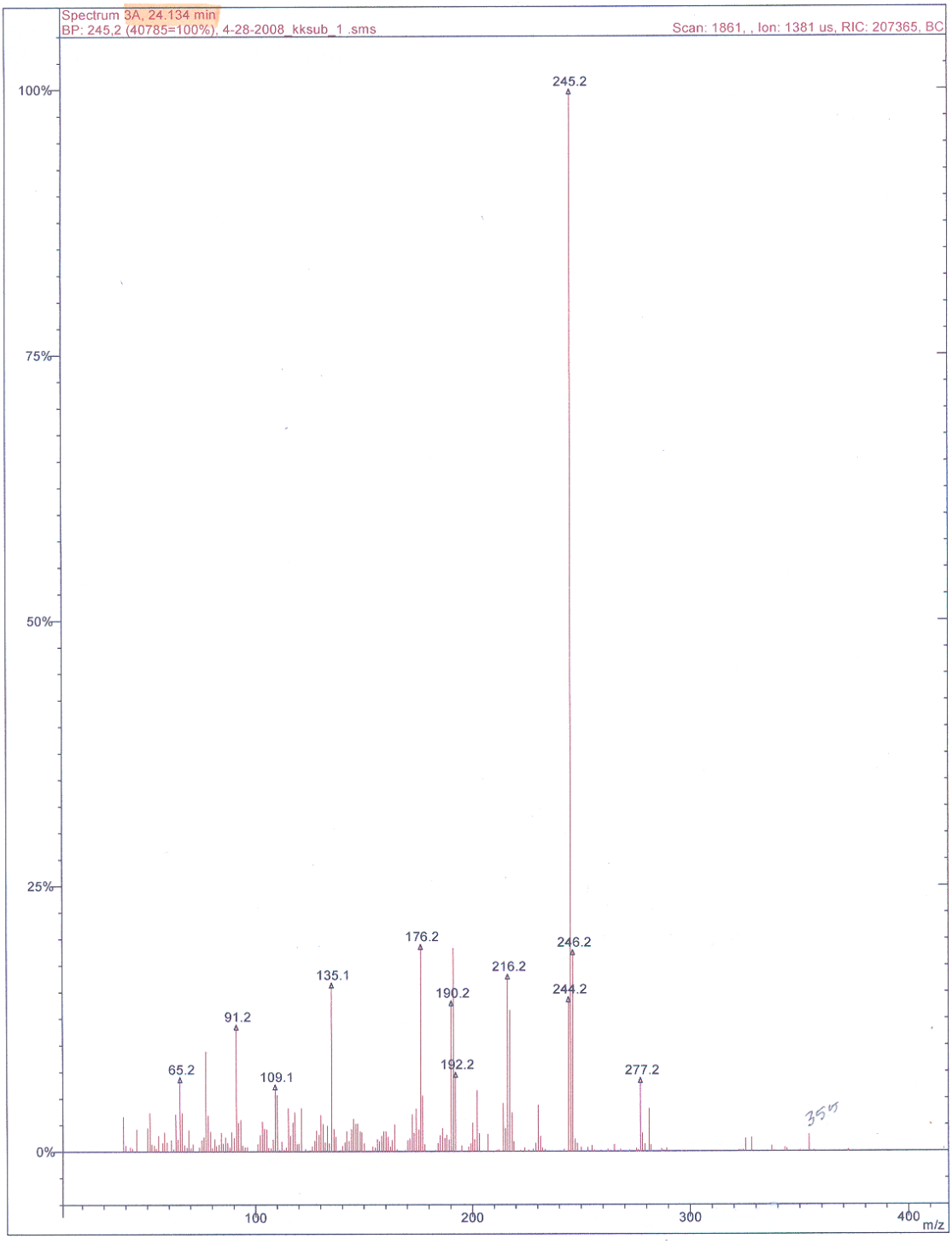
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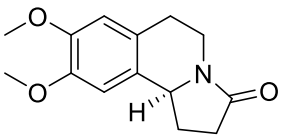
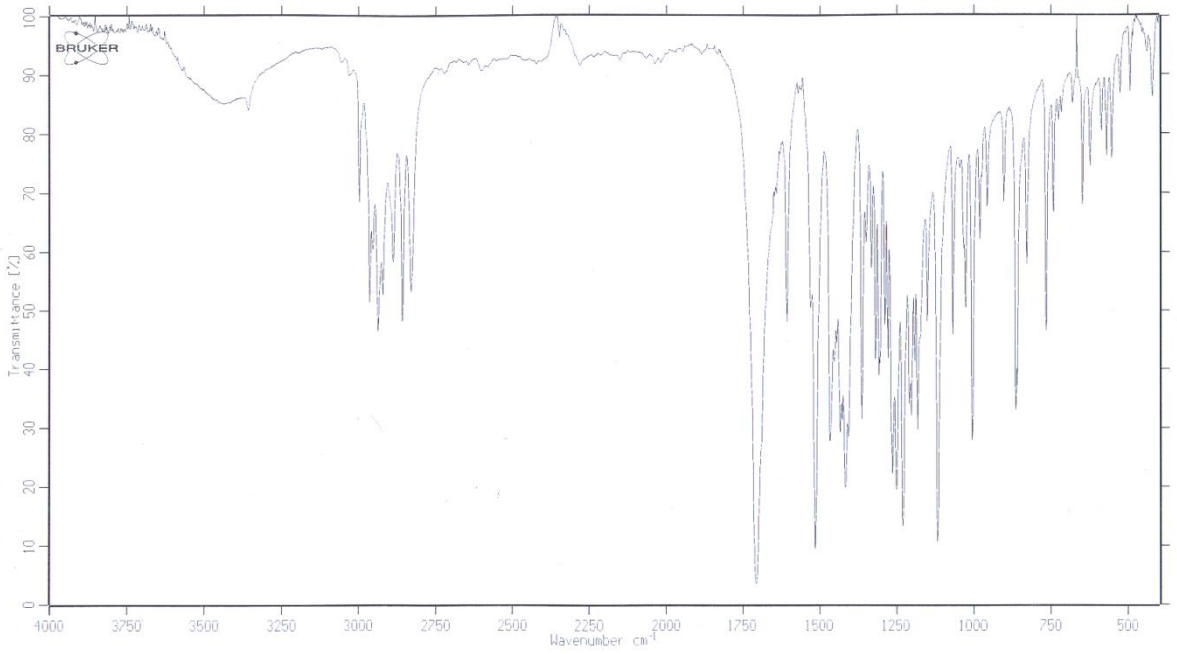
Scan: 1861, , Ion: 1381 us, RIC: 207365, BC
100%
75%
50%
25%
0%
245.2
246.2
244.2
176.2
216.2
135.1
190.2
91.2
65.2
109.1
192.2
277.2
100
200
300
400
m/z

(10b*S*)-(–)-8,9-Dimethoxy-1,5,6,10b-tetrahydropyrrolo[2,1-α]isoquinolin-3(*2H)*-one 7

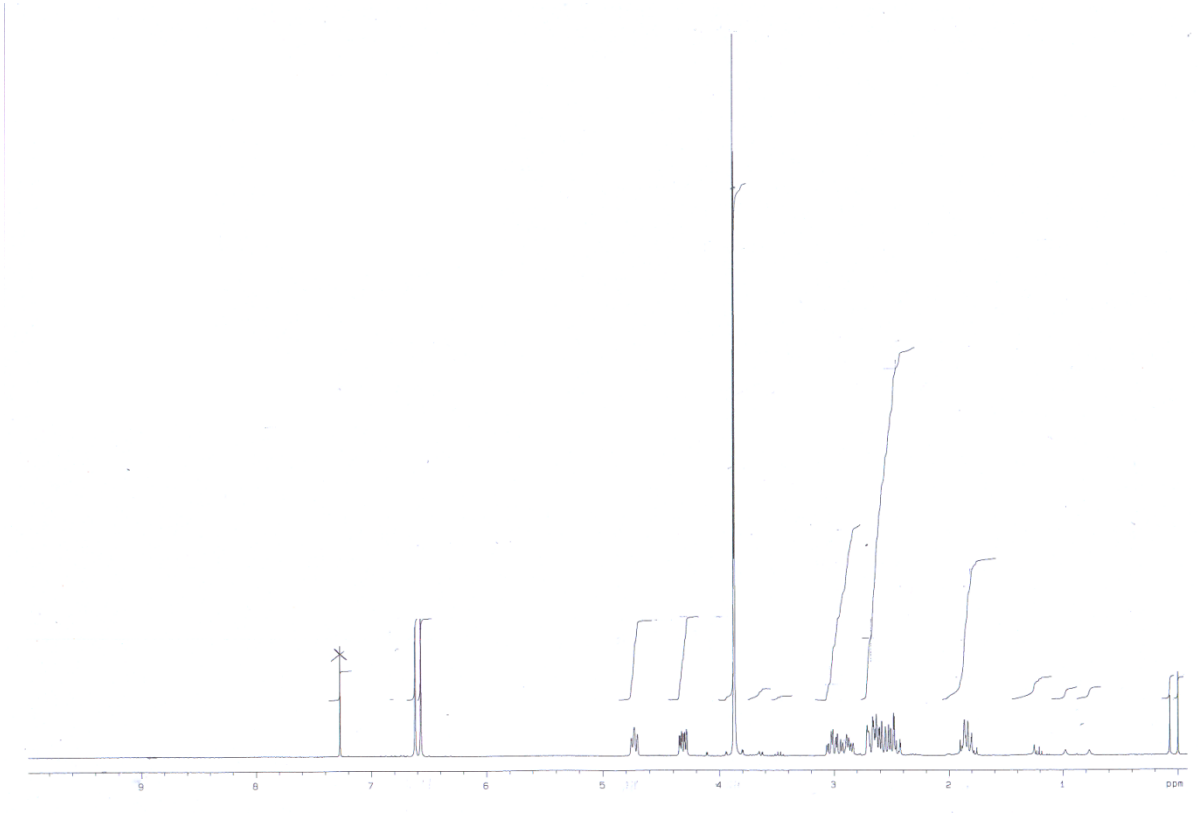


(–)-7

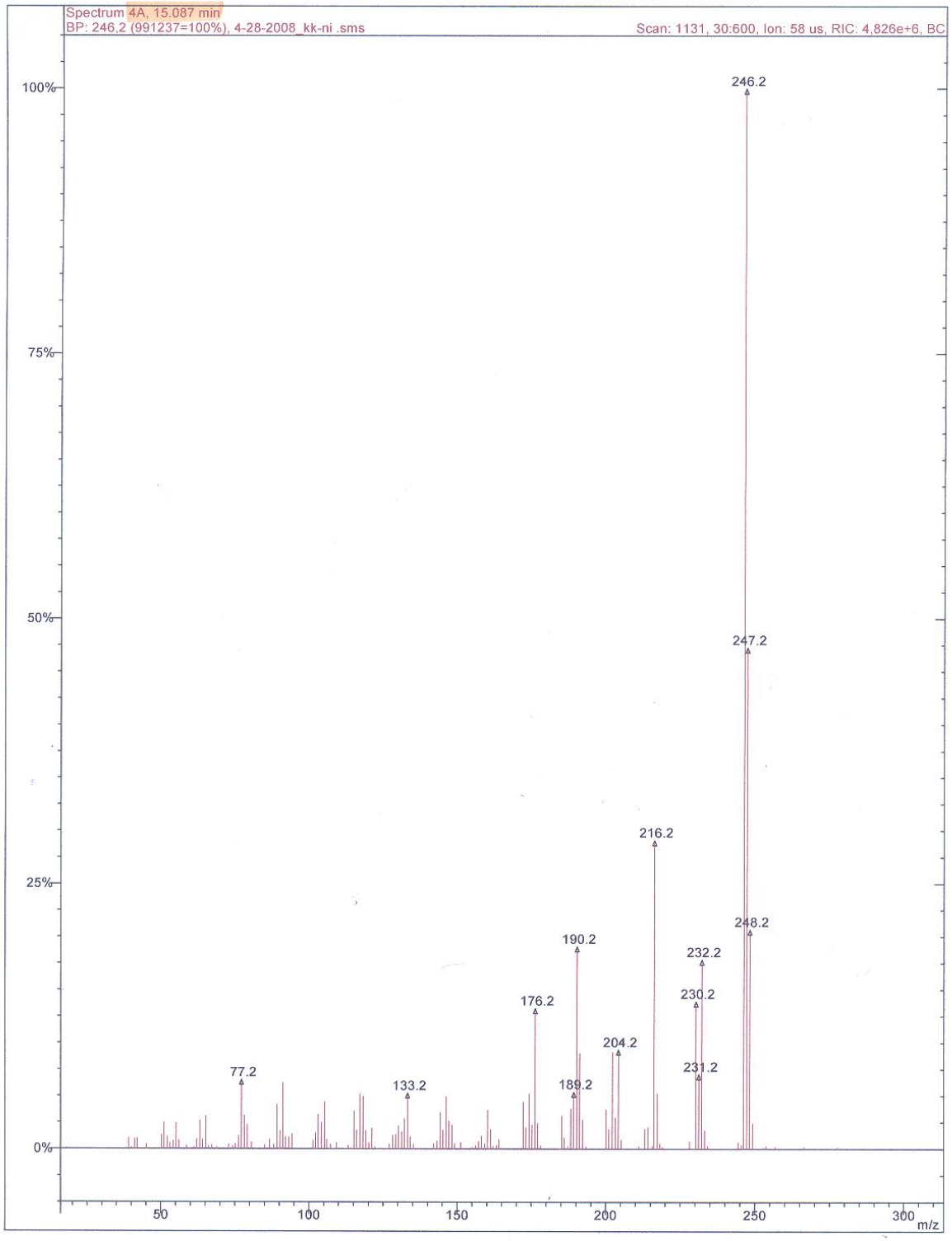
IR (film)



^{1}H NMR ($CDCl_3$)



MS


Spectrum 4A, 15.087 min
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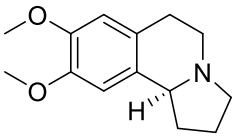
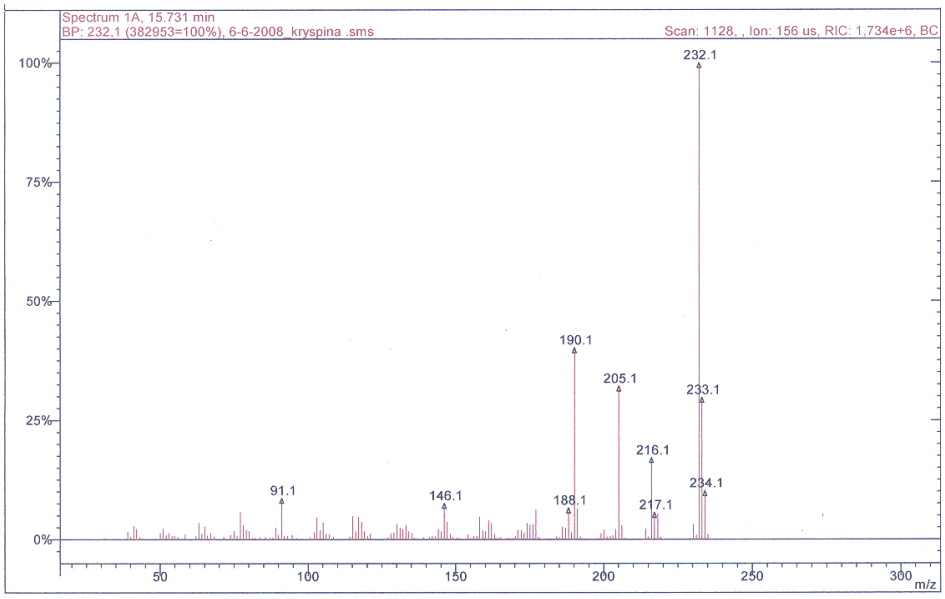
Scan: 1131, 30:600, Ion: 58 us, RIC: 4,826e+6, BC
100%
75%
50%
25%
0%
246.2
247.2
216.2
248.2
190.2
232.2
230.2
176.2
204.2
231.2
77.2
133.2
189.2
50
100
150
200
250
300
m/z

(10b*S*)-(–)-8,9-Dimethoxy-1,2,3,5,6,10b-hexahydropyrrolo[2,1-α]isoquinoline Crispine A 1

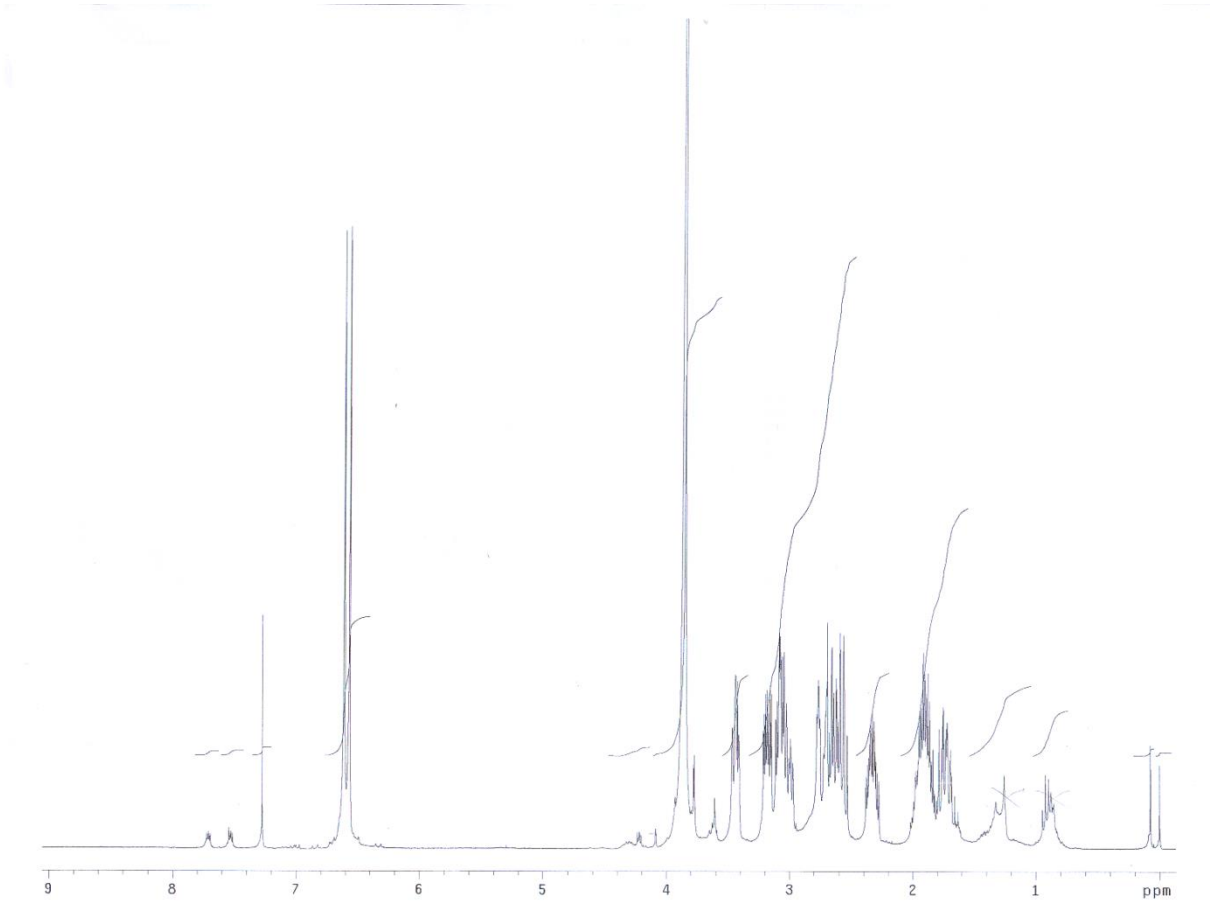


(–)-1

MS



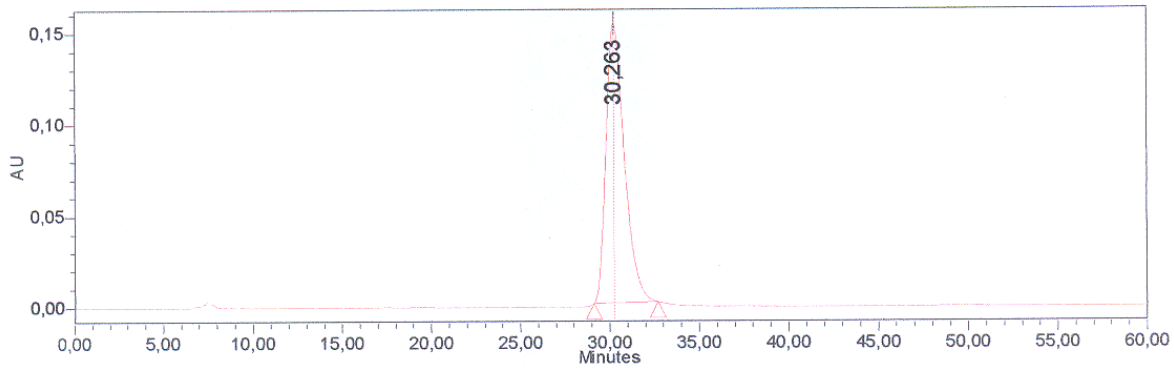
^{1}H NMR ($CDCl_3$)



HPLC data

(3*S*)-(−)-1-(3,4-dimethoxyphenethyl)-3-hydroxypyrrolidine-2,5-dione 8

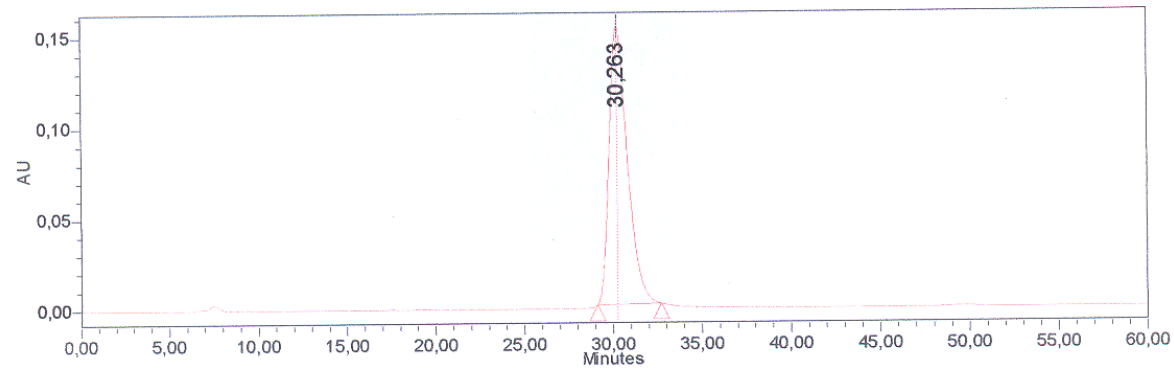
Daicel Chiralcel OD-H column (250 x 4.6 mm), hexane/propan-2-ol, flow rate 0.5 mL/min, 254 nm



	Processed Channel	Retention Time (min)	Area	% Area	Height
1	PDA 229,0 nm	30,263	10928154	100,00	152663

(3*R*)-(+)-1-(3,4-dimethoxyphenethyl)-3-hydroxypyrrolidine-2,5-dione 8

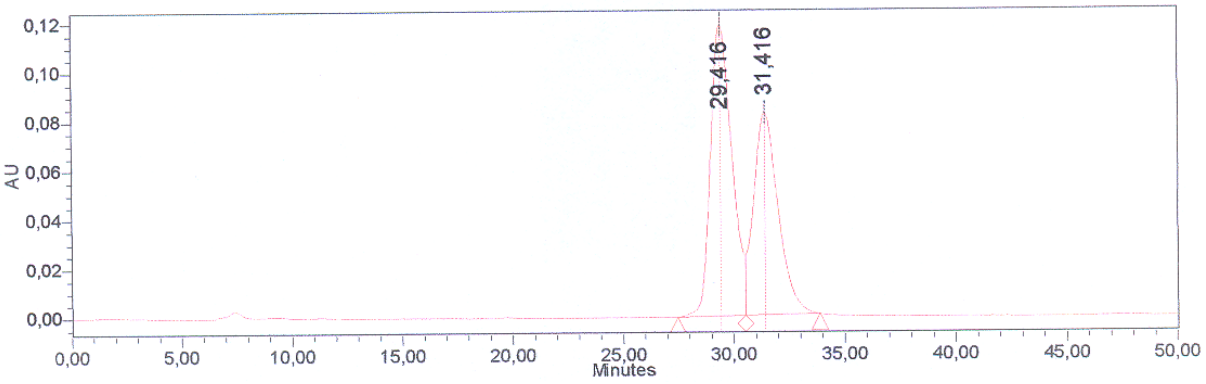
Daicel Chiralcel OD-H column (250 x 4.6 mm), hexane/propan-2-ol, flow rate 0.5 mL/min, 254 nm



	Processed Channel	Retention Time (min)	Area	% Area	Height
1	PDA 229,0 nm	30,263	10928154	100,00	152663

Racemic sample of 1-(3,4-dimethoxyphenethyl)-3-hydroxypyrrolidine-2,5-dione 8

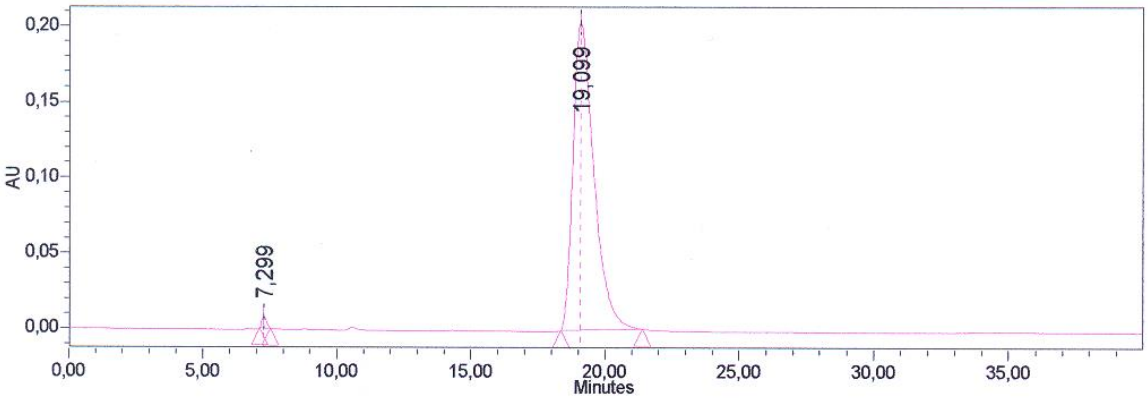
Daicel Chiralcel OD-H column (250 x 4.6 mm), hexane/propan-2-ol, flow rate 0.5 mL/min, 254 nm



	Processed Channel	Retention Time (min)	Area	% Area	Height
1	PDA 229,0 nm	29,416	8008077	55,64	118589
2	PDA 229,0 nm	31,416	6385324	44,36	82477

(1*S*,10b*R*)-(–)-1-Hydroxy-8,9-dimethoxy-1,5,6,10b-tetrahydro-2H-pyrrolo[2,1-α]isoquinolin-3-one 9

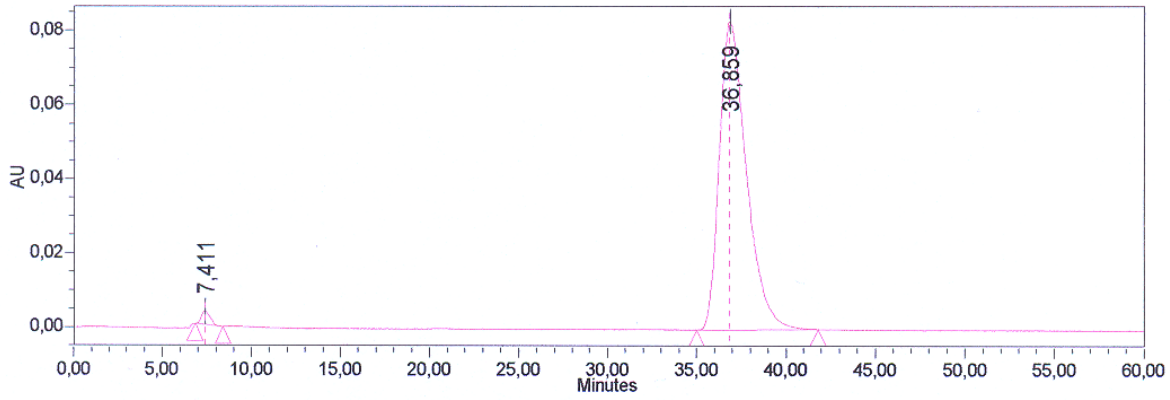
Daicel Chiralcel OD-H column (250 x 4.6 mm), hexane/propan-2-ol, flow rate 0.5 mL/min, 254 nm



	Processed Channel	Retention Time (min)	Area	% Area	Height
1	PDA 224,0 nm	7,299	89776	0,81	7739
2	PDA 224,0 nm	19,099	10945722	99,19	202835

(1*R*,10b*R*)-(–)-8,9-Dimethoxy-1-(phenylthio)-1,5,6,10b-tetrahydropyrrolo[2,1-α]isoquinolin-3(2H)-one 10

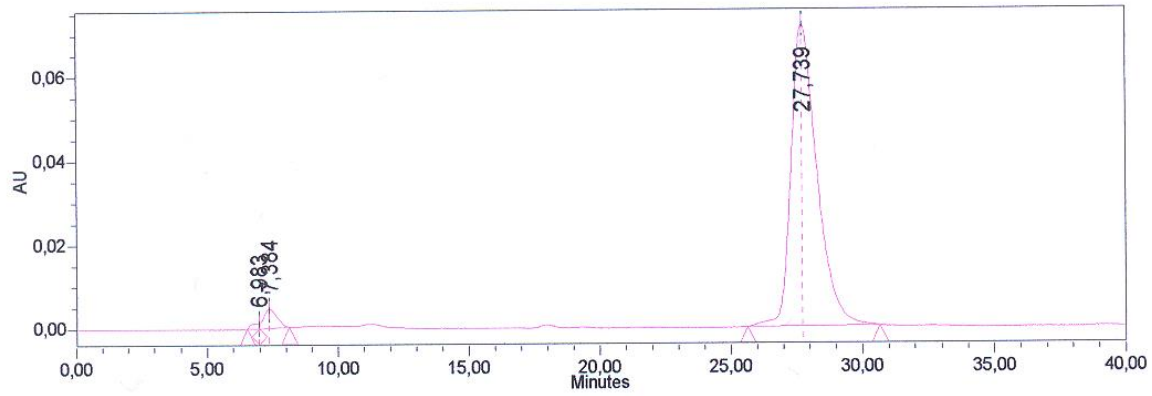
Daicel Chiralcel OD-H column (250 x 4.6 mm), hexane/propan-2-ol, flow rate 0.5 mL/min, 254 nm



	Processed Channel	Retention Time (min)	Area	% Area	Height
1	PDA 224,0 nm	7,411	119685	1,35	3593
2	PDA 224,0 nm	36,859	8739603	98,65	82812

(10b*S*)-(–)-8,9-Dimethoxy-1,5,6,10b-tetrahydropyrrolo[2,1-α]isoquinolin-3(*2H*)-one 7

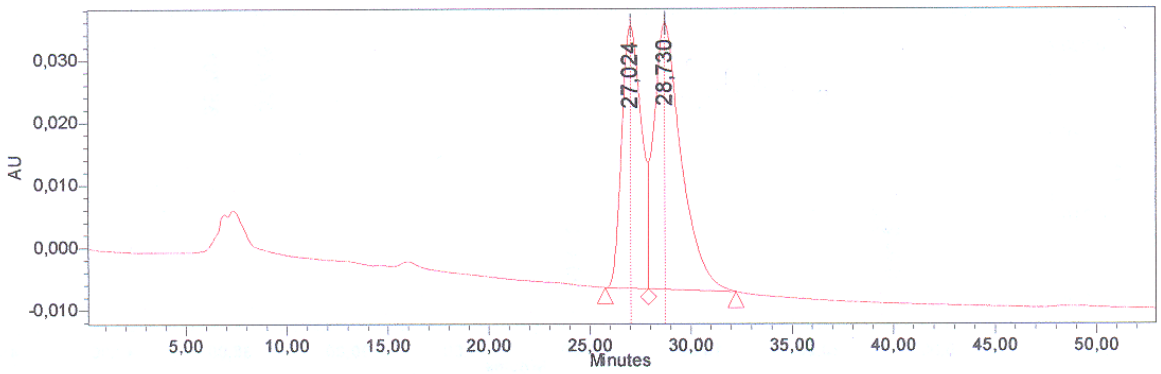
Daicel Chiralcel OD-H column (250 x 4.6 mm), hexane/propan-2-ol, flow rate 0.5 mL/min, 254 nm



	Processed Channel	Retention Time (min)	Area	% Area	Height
1	PDA 224,0 nm	6,983	24719	0,49	1139
2	PDA 224,0 nm	7,384	155906	3,06	4382
3	PDA 224,0 nm	27,739	4913636	96,45	71779

(±)-8,9-Dimethoxy-1,5,6,10b-tetrahydropyrrolo[2,1-α]isoquinolin-3(*2H*)-one 7

Daicel Chiralcel OD-H column (250 x 4.6 mm), hexane/propan-2-ol, flow rate 0.5 mL/min, 254 nm

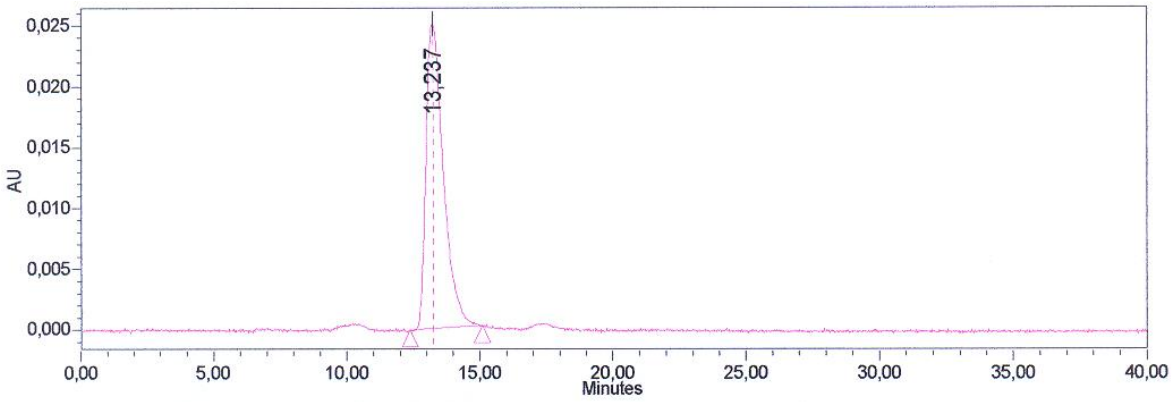


Peak Results

	Name	RT	Area	% Area	Height	Amount	Units
1		27,024	2850179	41,37	42040		
2		28,730	4039062	58,63	42655		

(10b*S*)-(–)-8,9-Dimethoxy-1,2,3,5,6,10b-hexahydropyrrolo[2,1-α]isoquinoline Crispine A 1

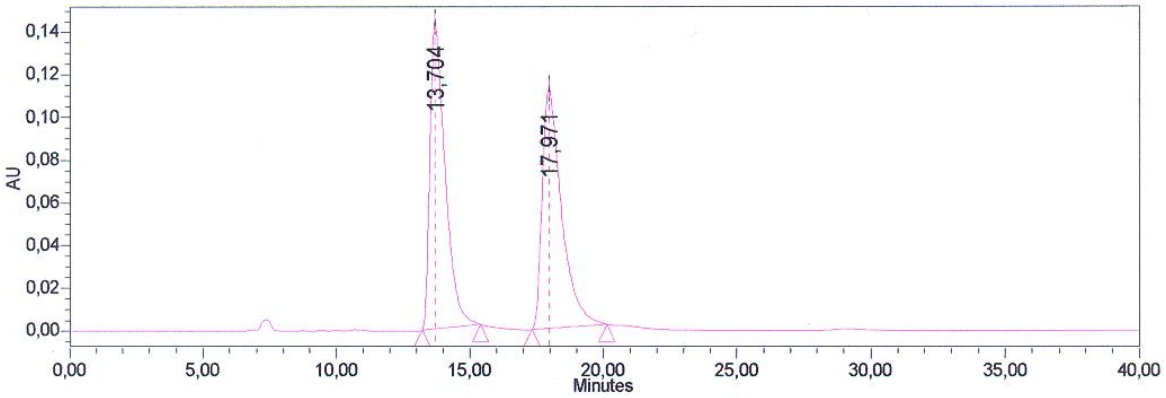
Daicel Chiralcel OD-H column (250 x 4.6 mm), hexane/propan-2-ol, flow rate 0.5 mL/min, 254 nm



	Processed Channel	Retention Time (min)	Area	% Area	Height
1	PDA 282,0 nm	13,237	1141884	100,00	25012

(±)-8,9-Dimethoxy-1,2,3,5,6,10b-hexahydropyrrolo[2,1-α]isoquinoline Crispine A 1

Daicel Chiralcel OD-H column (250 x 4.6 mm), hexane/propan-2-ol, flow rate 0.5 mL/min, 254 nm



	Processed Channel	Retention Time (min)	Area	% Area	Height
1	PDA 224,0 nm	13,704	5662019	50,17	143617
2	PDA 224,0 nm	17,971	5623810	49,83	112131