

Electronic supplementary information for

Ricinodols A–G: New Tetracyclic Triterpenoids as 11 β -HSD1

Inhibitors from *Ricinodendron heudelotii*

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Table S2. Preliminary assay results of tested compounds against mouse 11 β -HSD1 at 10.0 μ M.

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Figure S64. ESI(+)MS spectrum of ricinodol G (7)

Figure S65. ESI(-)MS spectrum of ricinodol G (7)

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Figure S67. IR spectrum of ricinodol G (7)

Table S1. Preliminary assay results of tested compounds against human 11 β -HSD1 at 10.0 μ M.

Compds no.	Exp. 1	Exp. 2	Exp. 3	Average	SD
1	63.0%	51.3%	61.6%	58.6%	6.4%
2	21.0%	17.4%	15.8%	18.1%	2.6%
3	64.3%	58.5%	59.8%	60.8%	3.0%
4	62.2%	58.5%	54.3%	58.4%	4.0%
5	98.8%	94.3%	95.4%	96.2%	2.4%
6	56.9%	53.0%	53.4%	54.4%	2.1%
7	55.2%	56.1%	56.5%	56.0%	0.6%
8	28.5%	28.8%	37.0%	31.4%	4.8%
9	12.1%	14.6%	19.6%	15.4%	3.8%
10	35.6%	27.8%	34.6%	32.7%	4.2%
Glycyrrhetic acid 1 nM	27.6%	21.0%	19.1%	22.6%	4.4%
Glycyrrhetic acid 10 nM	61.1%	67.6%	69.7%	66.1%	4.5%
Glycyrrhetic acid 100 nM	95.8%	98.1%	94.7%	96.2%	1.8%

Table S2. Preliminary assay results of tested compounds against mouse 11 β -HSD1 at 10.0 μ M.

Compds no.	Exp. 1	Exp. 2	Exp. 3	Average	SD
1	39.3%	40.2%	35.6%	38.4%	2.5%
2	41.6%	40.8%	33.4%	38.6%	4.5%
3	44.9%	47.9%	40.2%	44.3%	3.9%
4	35.8%	39.0%	32.1%	35.6%	3.5%
5	95.6%	96.4%	91.5%	94.5%	2.6%
6	74.2%	78.5%	73.9%	75.5%	2.6%
7	67.8%	64.1%	59.9%	63.9%	3.9%
8	28.2%	25.9%	25.6%	26.6%	1.4%
9	38.7%	44.0%	36.7%	39.8%	3.8%
10	38.8%	42.0%	35.9%	38.9%	3.1%
Glycyrrhetic acid 1 nM	21.8%	17.4%	15.3%	18.2%	3.3%
Glycyrrhetic acid 10 nM	55.9%	52.7%	51.5%	53.4%	2.3%
Glycyrrhetic acid 100 nM	93.3%	95.5%	95.9%	94.9%	1.4%

Figure S1. Induced CD spectrum of the Mo_2^{4+} complex for **2** in DMSO

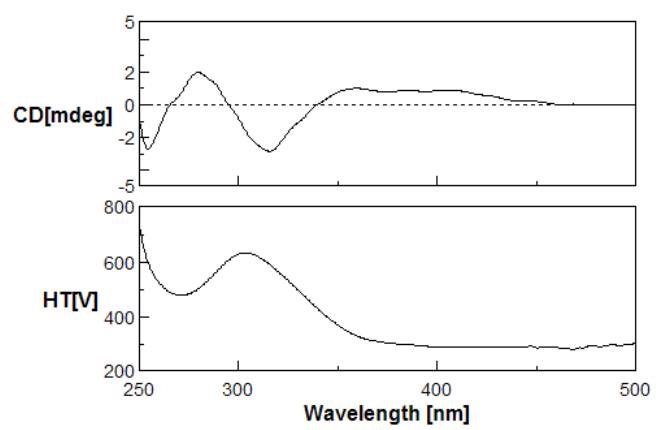


Figure S2. Induced CD spectrum of the Mo_2^{4+} complex for **5** in DMSO

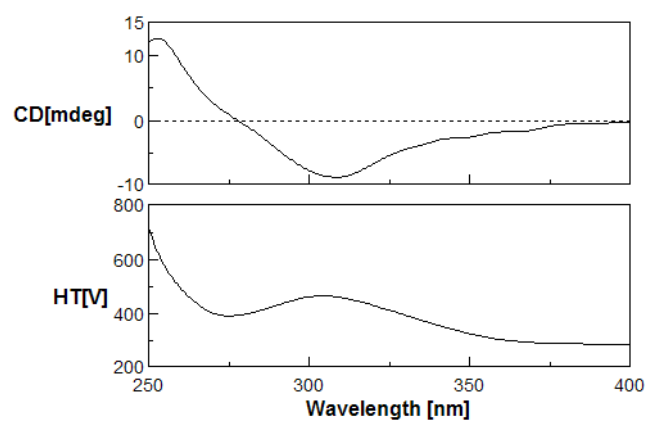


Figure S3. ^1H NMR spectrum of ricinodol A (**1**) in CDCl_3

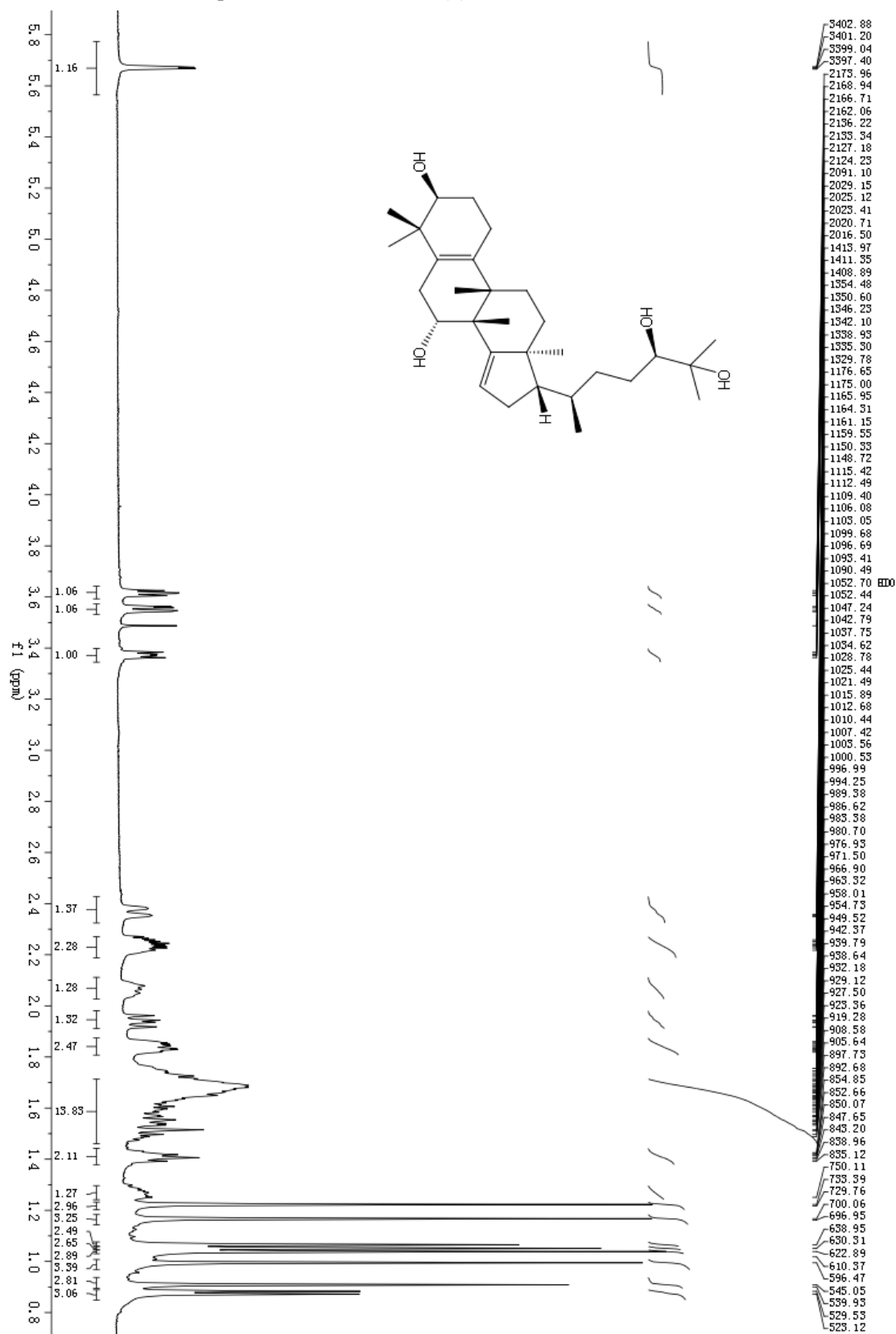


Figure S4. ^{13}C NMR spectrum of ricinodol A (**1**) in CDCl_3

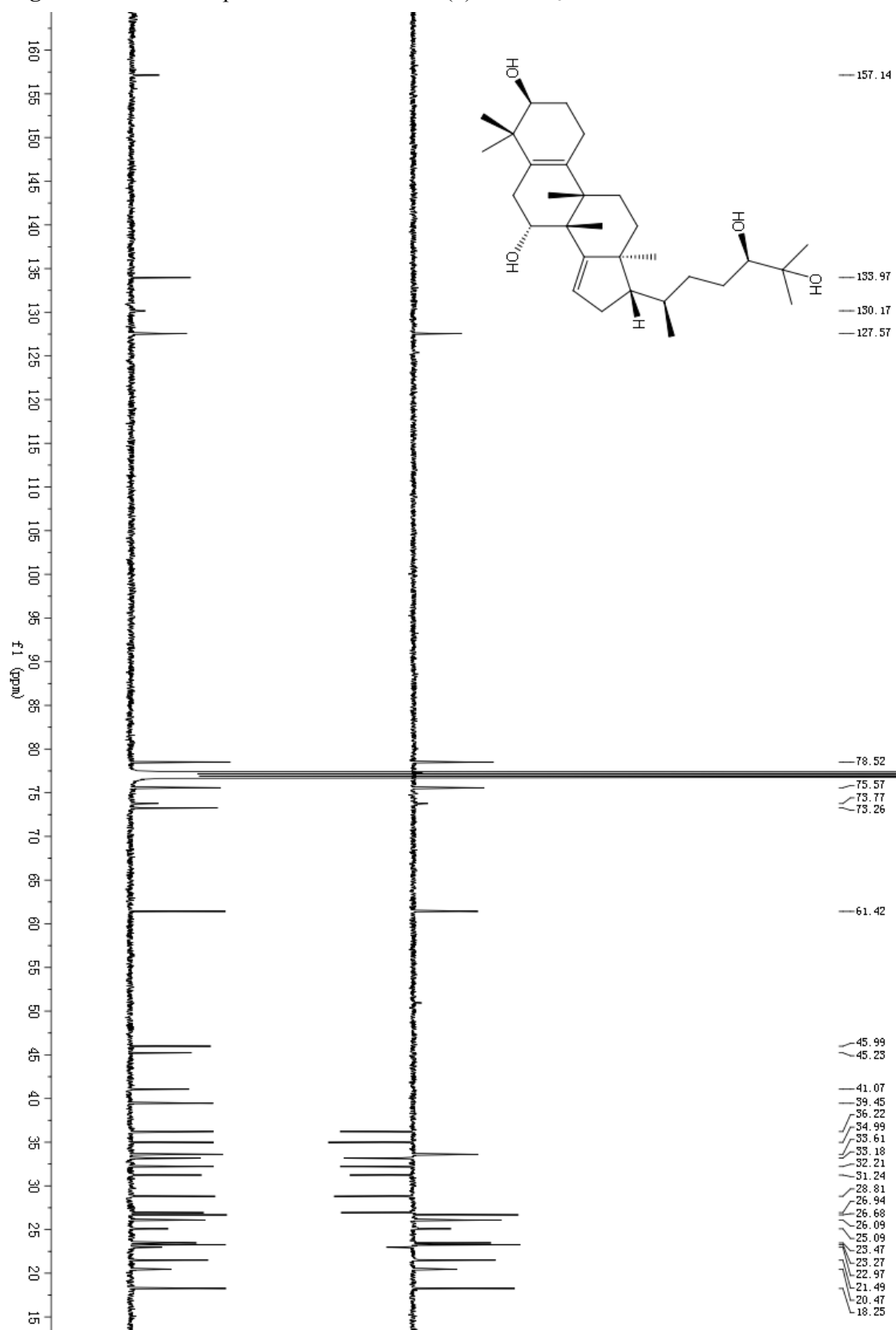


Figure S5. HSQC spectrum of ricinodol A (**1**) in CDCl₃

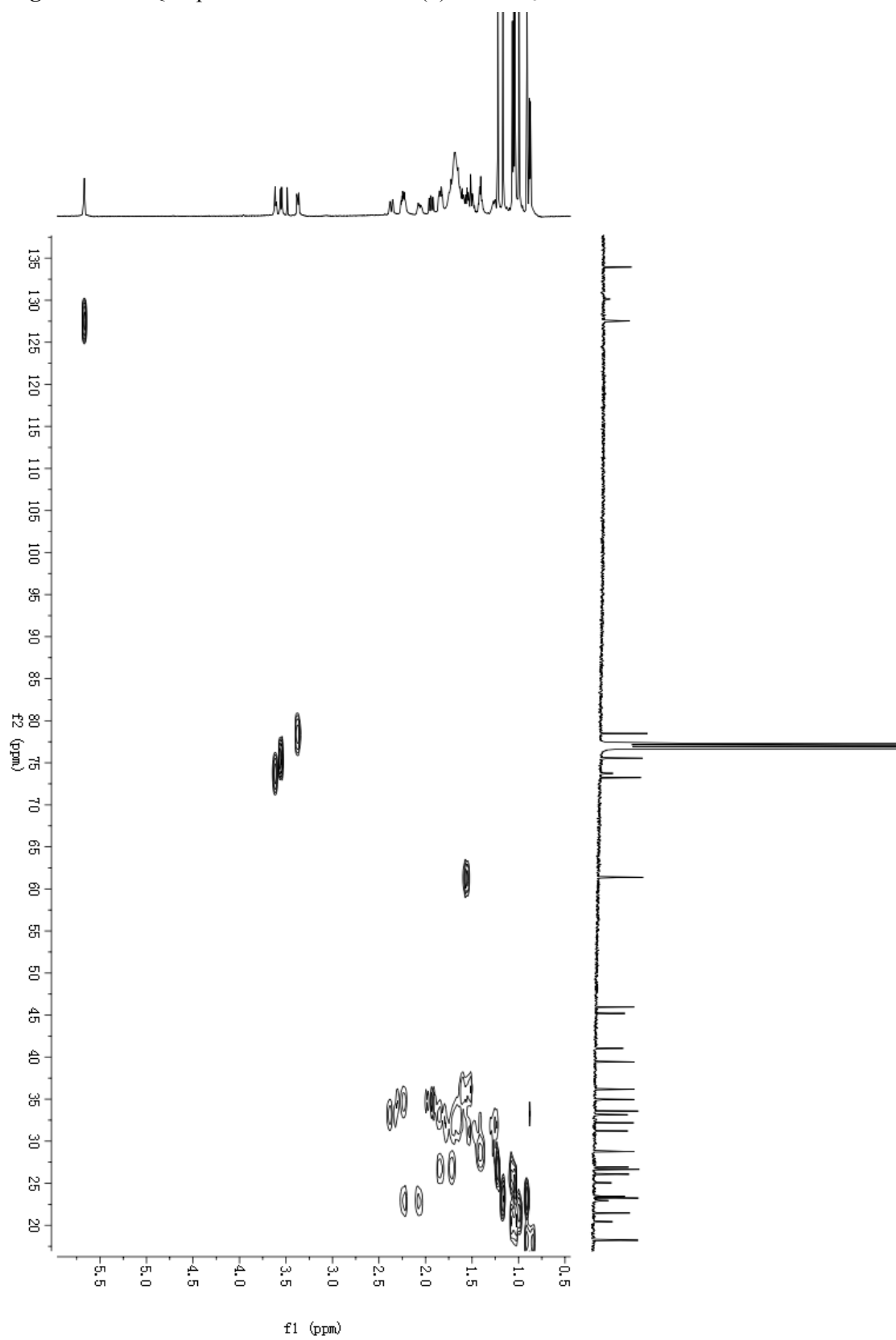


Figure S6. ^1H - ^1H COSY spectrum of ricinodol A (**1**) in CDCl_3

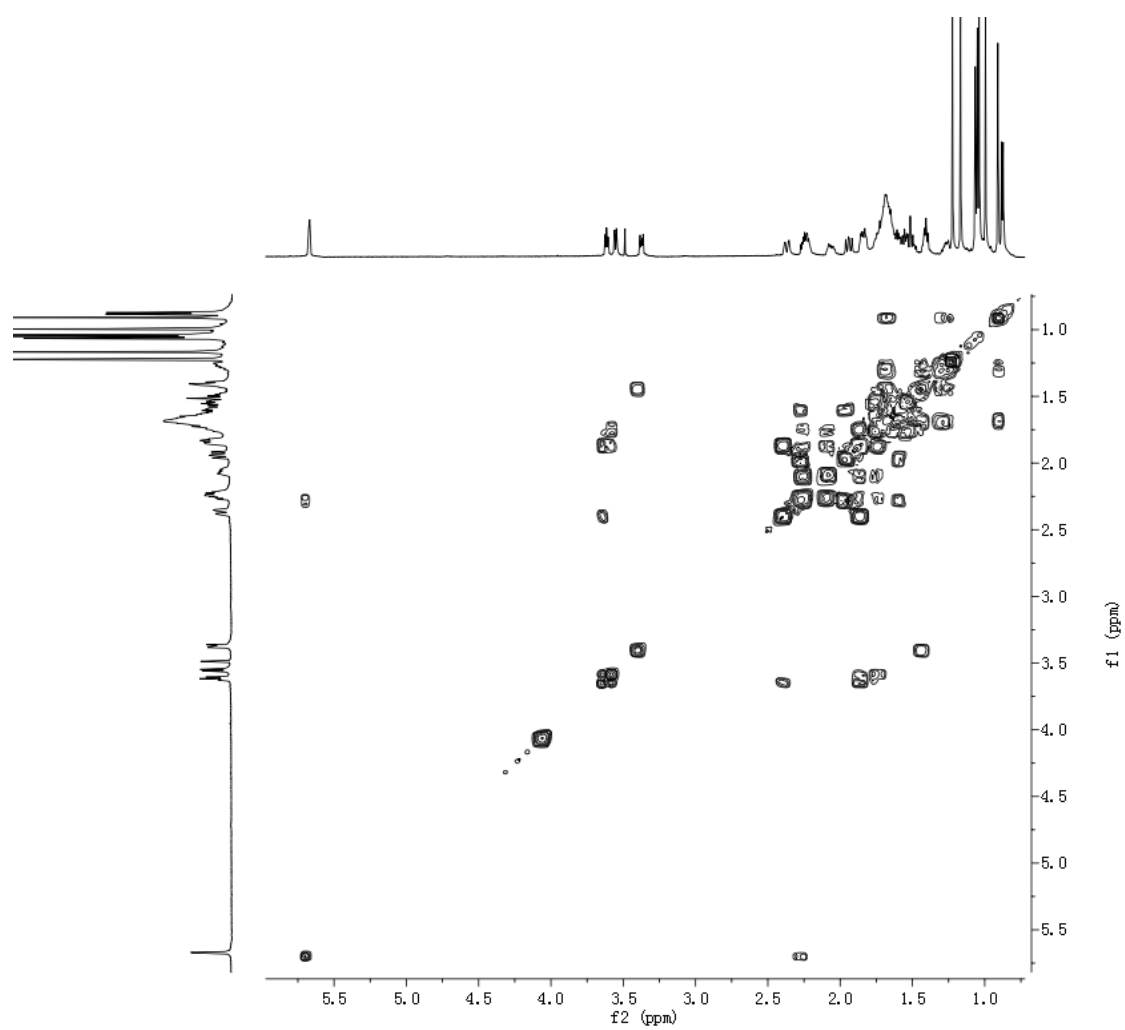


Figure S7. HMBC spectrum of ricinodol A (**1**) in CDCl₃

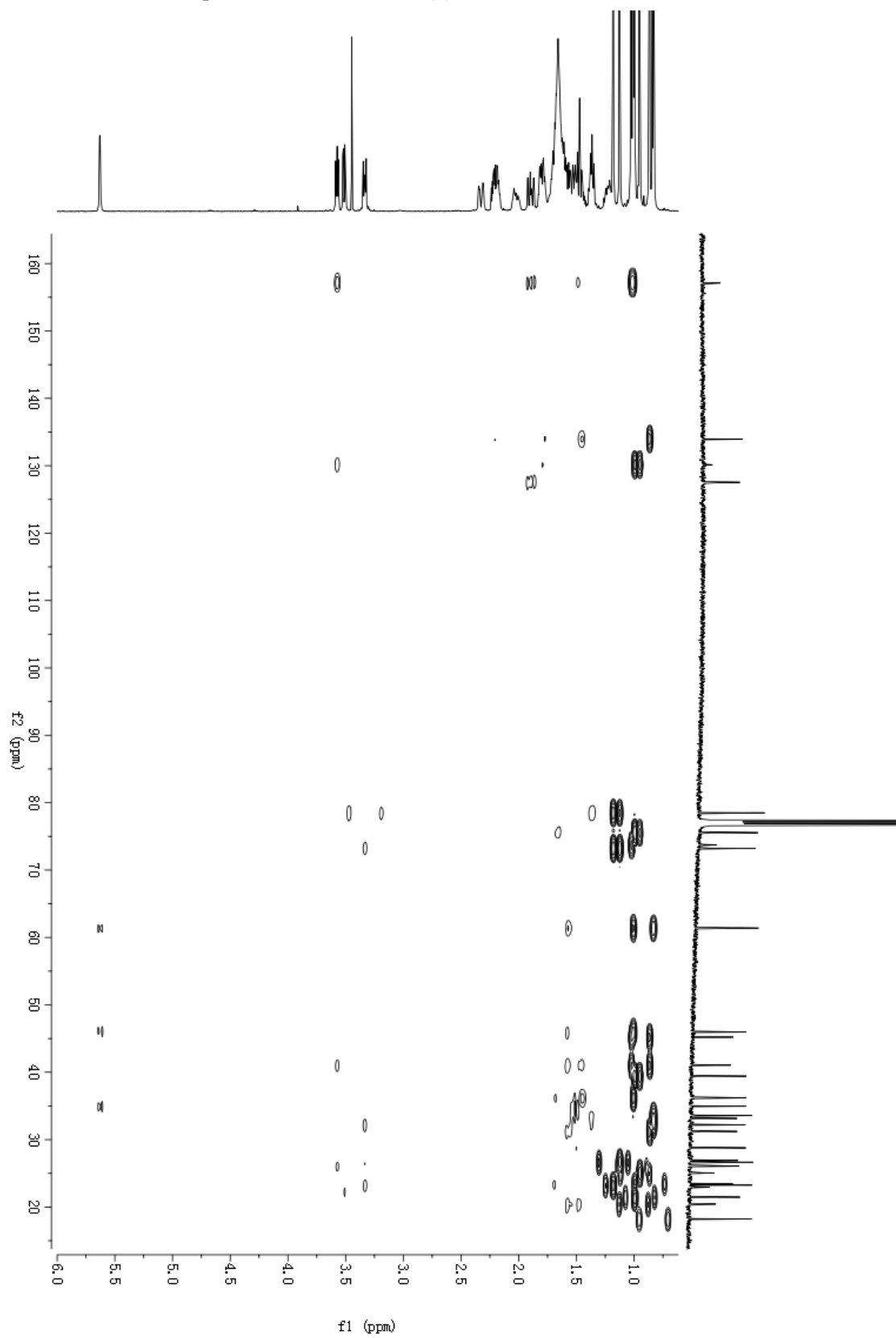


Figure S8. ROESY spectrum of ricinodol A (1) in CDCl₃

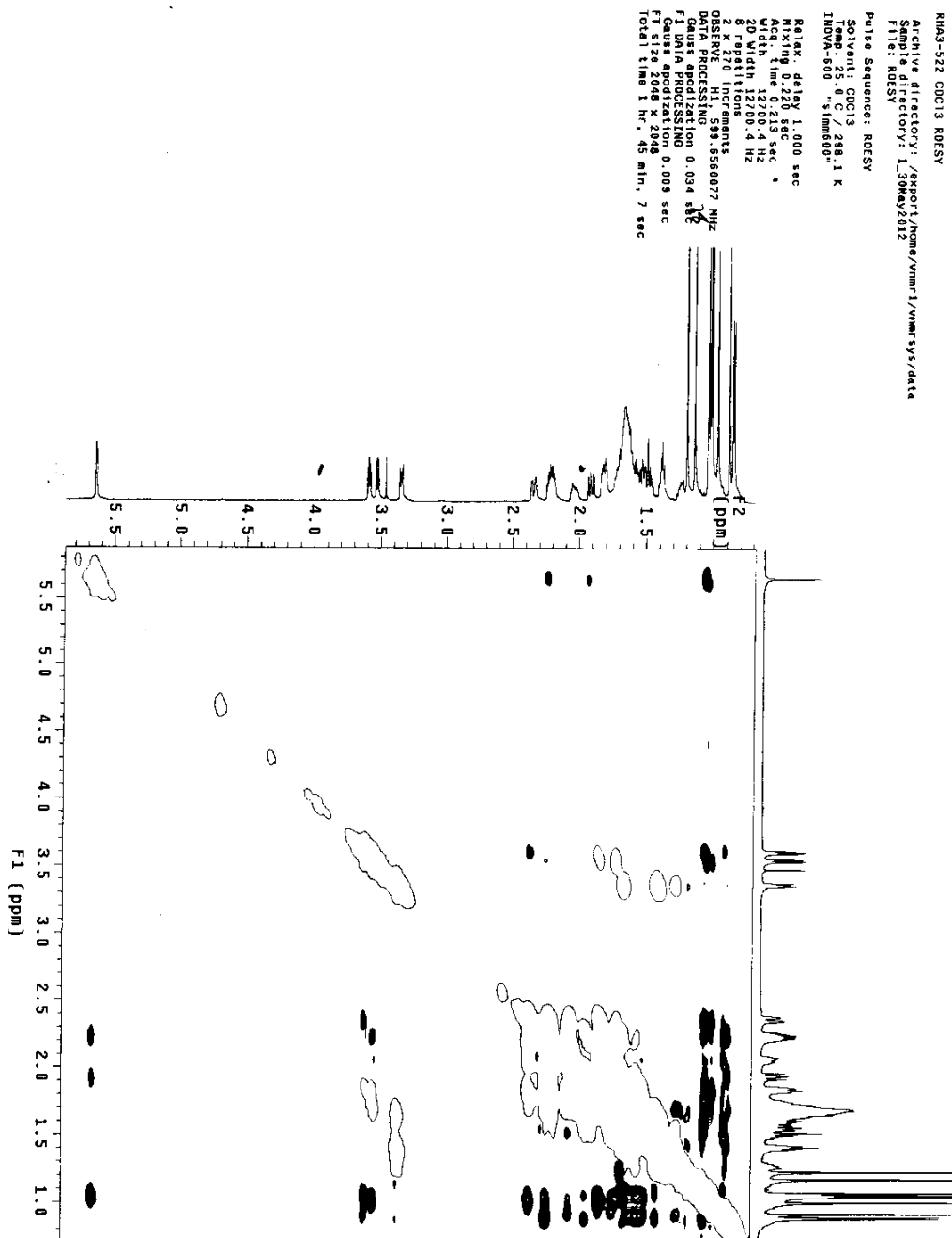


Figure S9. ESI(+)-MS spectrum of ricinodol A (1)

Display Report

Analysis Info

Analysis Name	002-0501.D	Acquisition Date	05/30/12 09:59:26
Method	Copy of DSOPMS2P.M	Operator	Administrator
Sample Name	yjm-RHA3-5212	Instrument	esquire3000plus
Comment	*)□		

Acquisition Parameter

Ion Source Type	ESI	Ion Polarity	Positive	Alternating Ion Polarity	off
Mass Range Mode	Std/Normal	Scan Begin	100 m/z	Scan End	1750 m/z
Capillary Exit	158.5 Volt	Skim 1	40.0 Volt	Trap Drive	85.4
Accumulation Time	15000	Averages	3 Spectra	Auto MS/MS	on

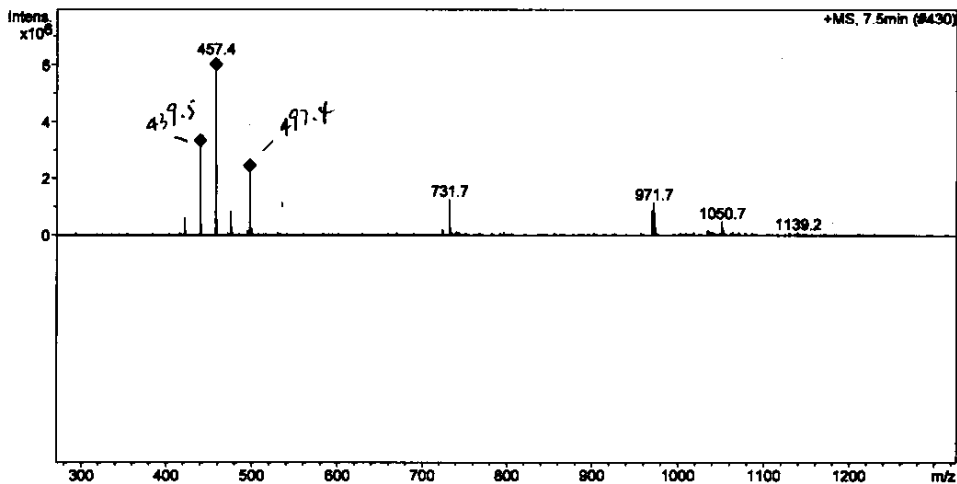
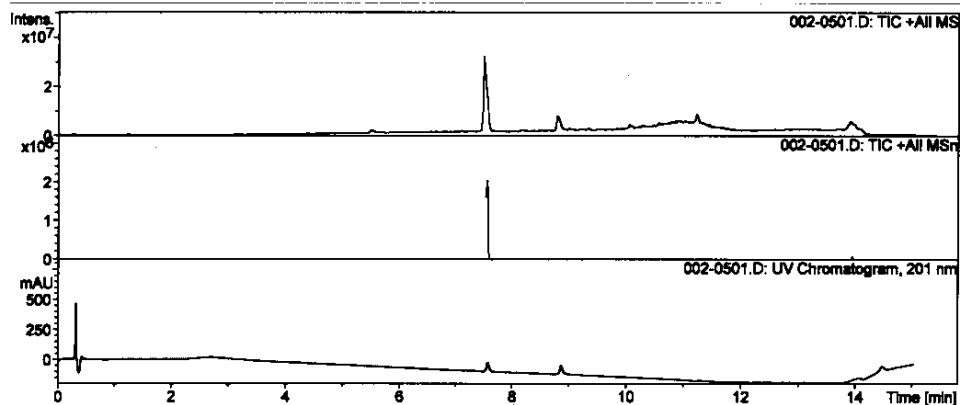


Figure S10. ESI(-)MS spectrum of ricinodol A (1)

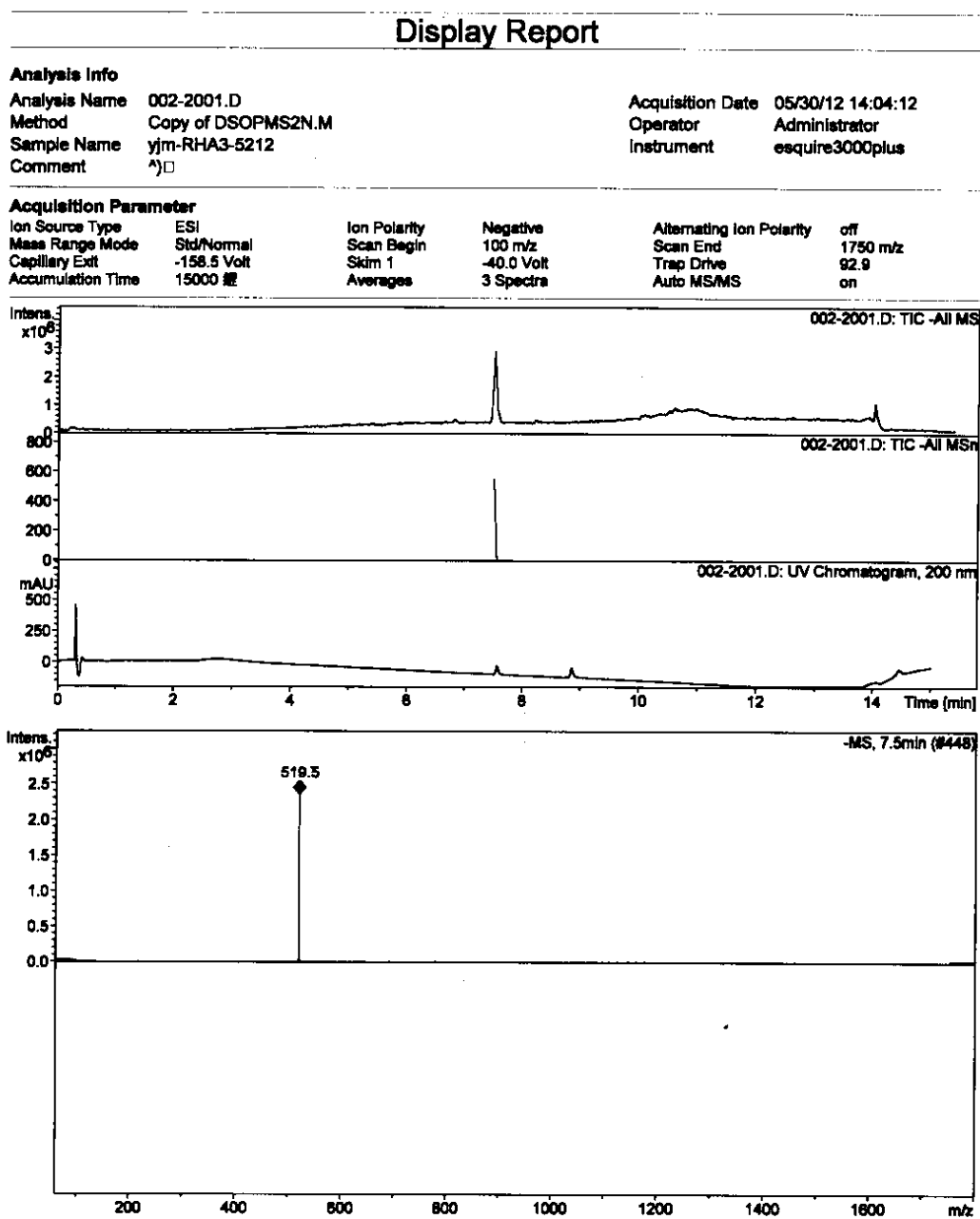


Figure S11. HRESI(-)MS spectrum of ricinodol A (1)

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 3.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

121 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 10-60 H: 1-110 O: 0-30

YJH

LCT PXE KE324

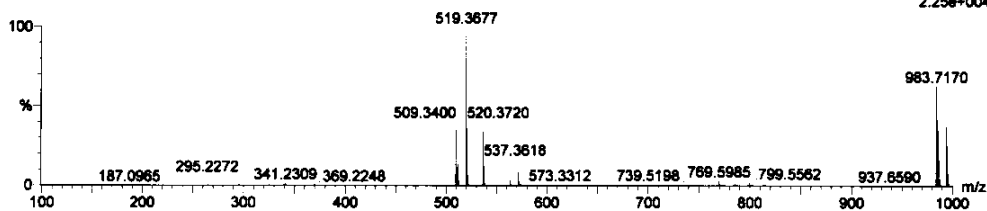
01-Jun-2012

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2.25e+004



Minimum:

Maximum: 3.0 3.0 -1.5

50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
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Figure S12. IR spectrum of ricinodol A (1)

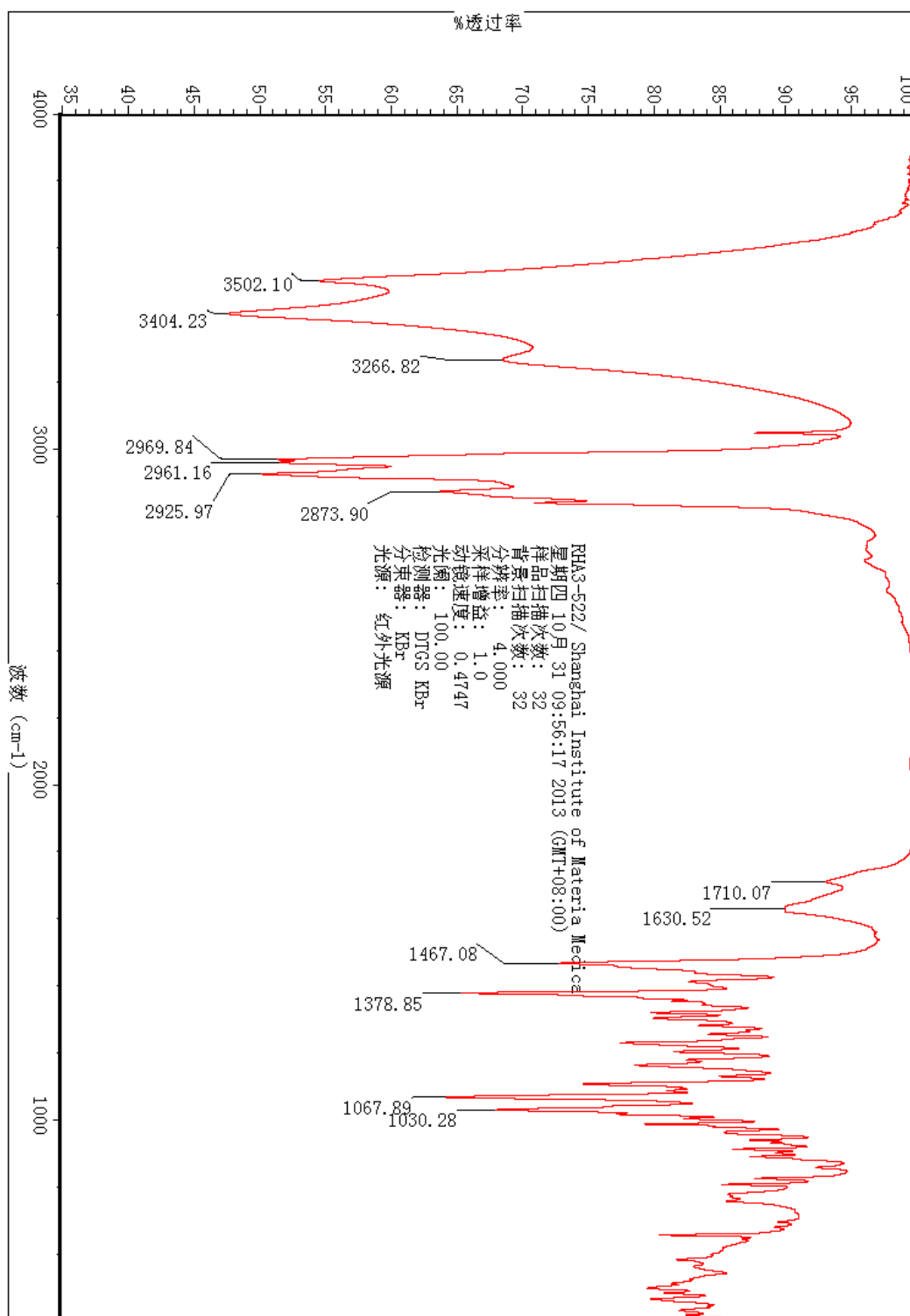


Figure S13. ^1H NMR spectrum of ricinodol B (2) in CDCl_3

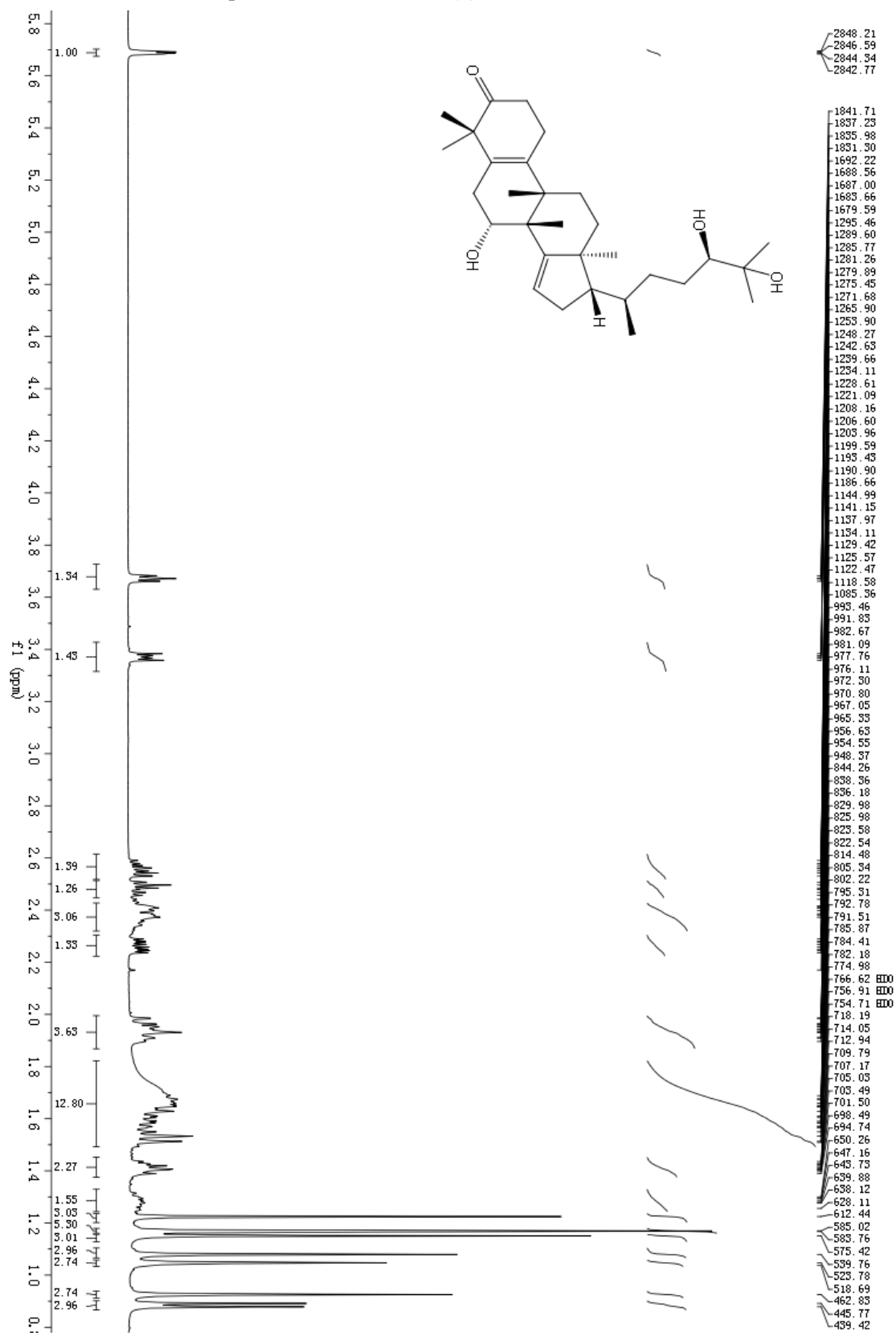


Figure S14. ^{13}C NMR spectrum of ricinodol B (**2**) in CDCl_3

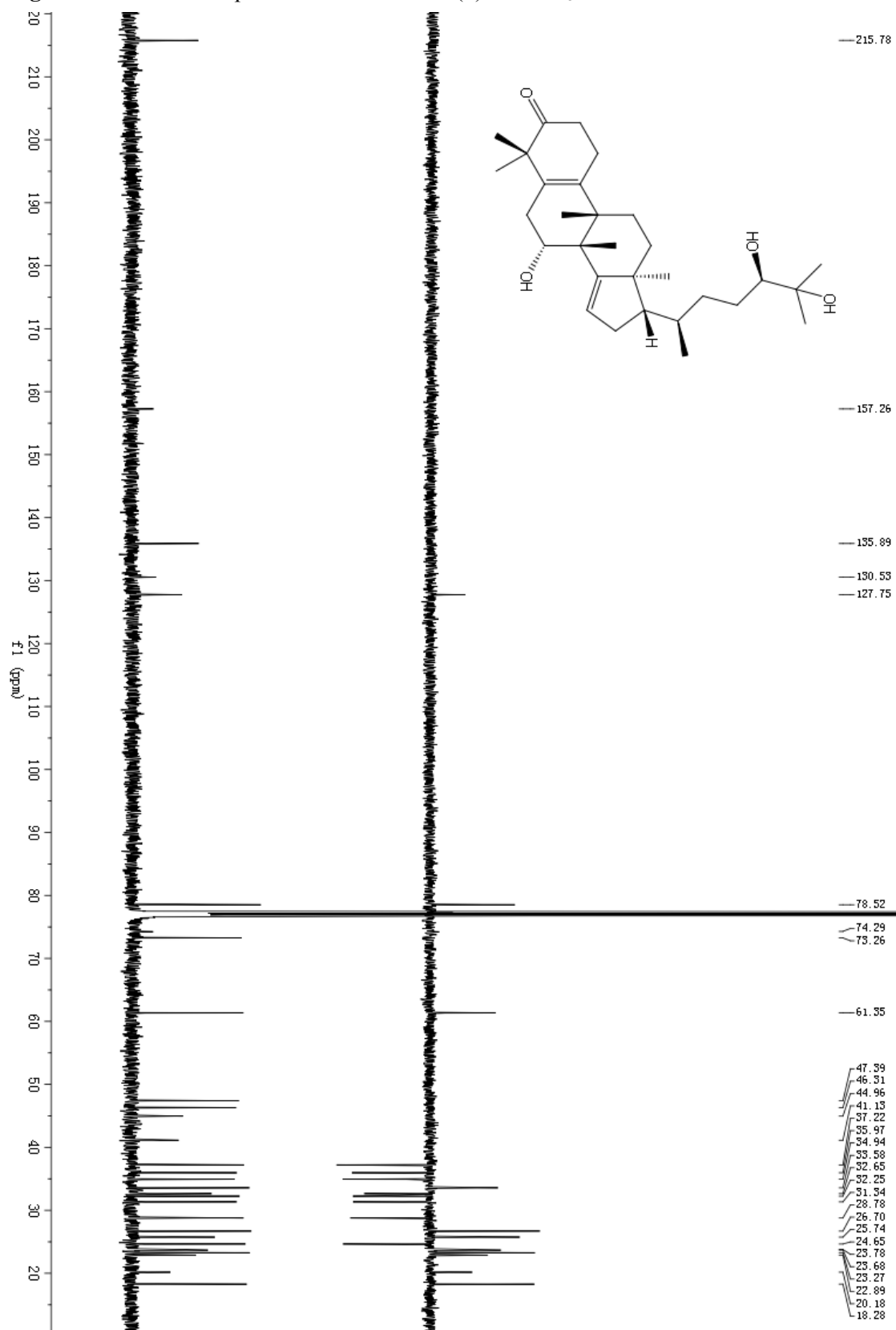


Figure S15. HSQC spectrum of ricinodol B (**2**) in CDCl₃

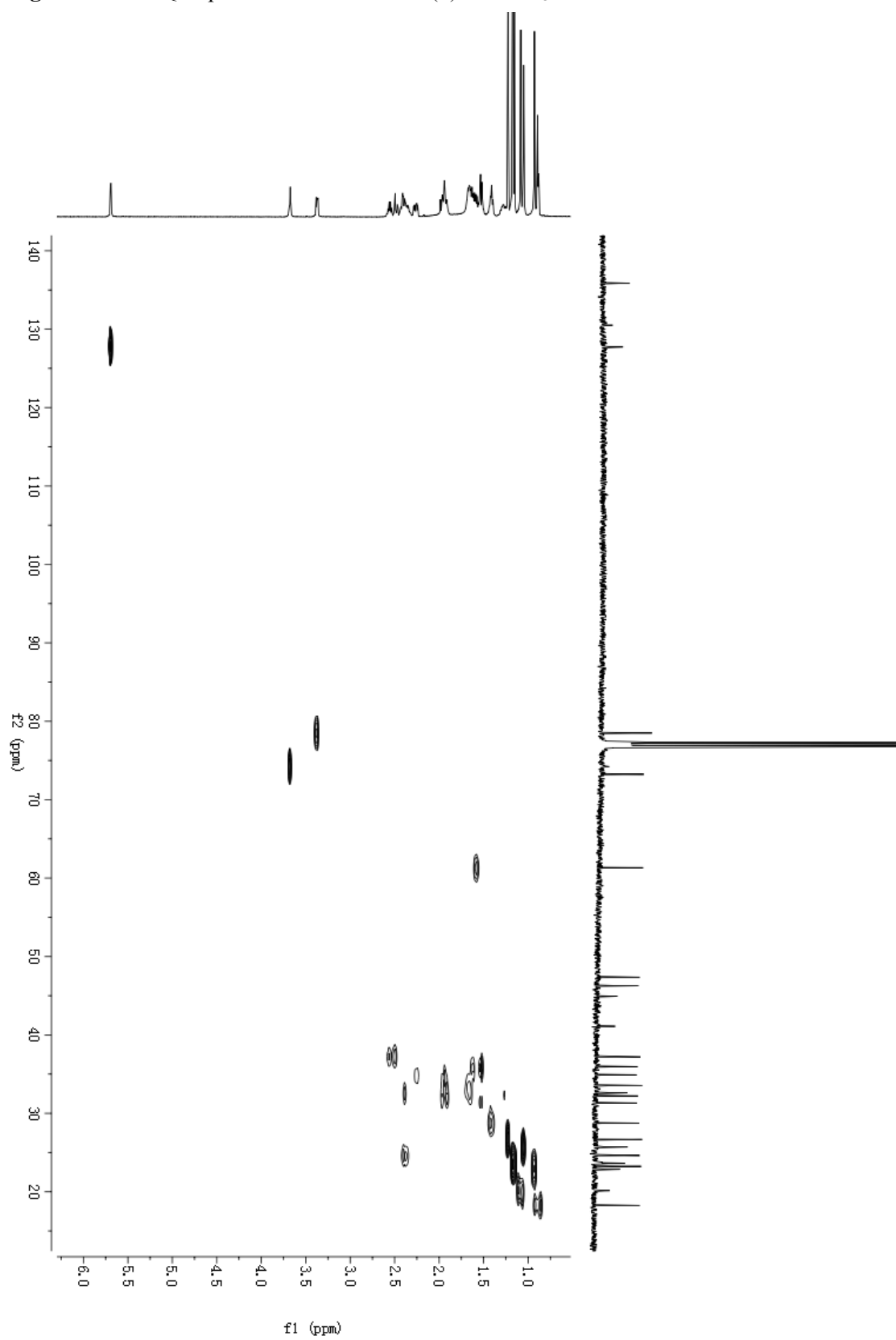


Figure S16. HMBC spectrum of ricinodol B (**2**) in CDCl₃

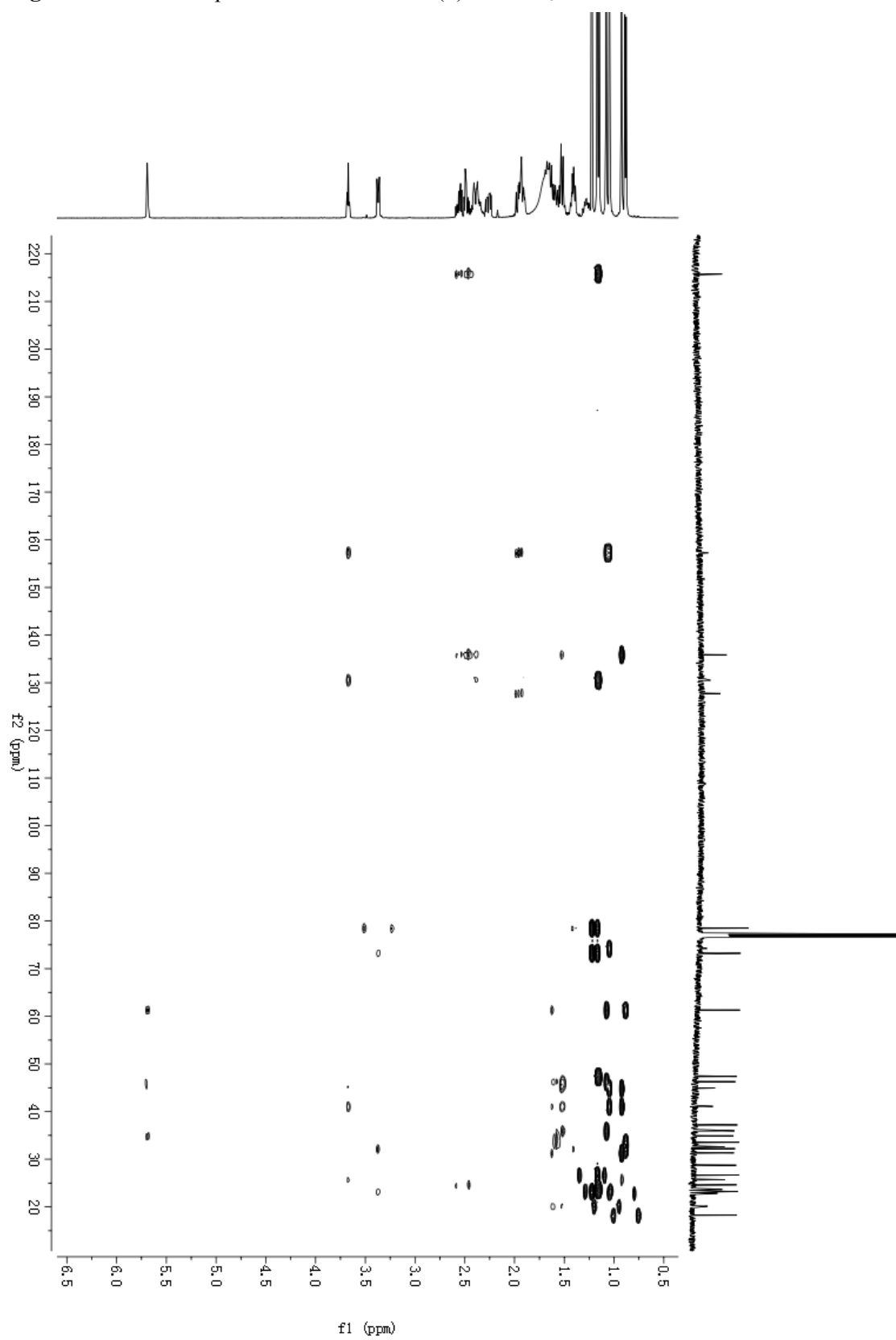


Figure S17. ROESY spectrum of ricinodol B (2) in CDCl₃

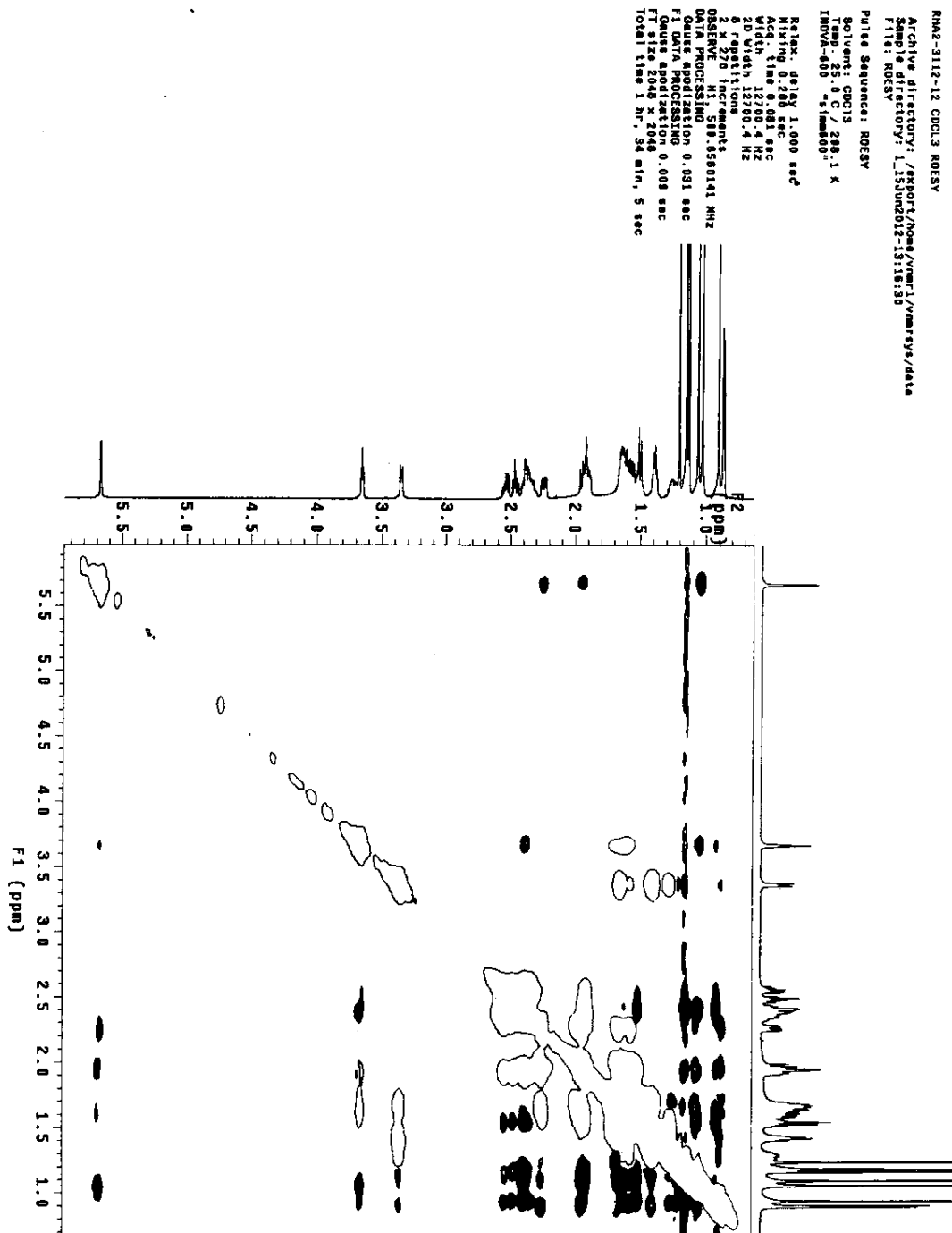


Figure S18. ESI(+)MS spectrum of ricinodol B (2)

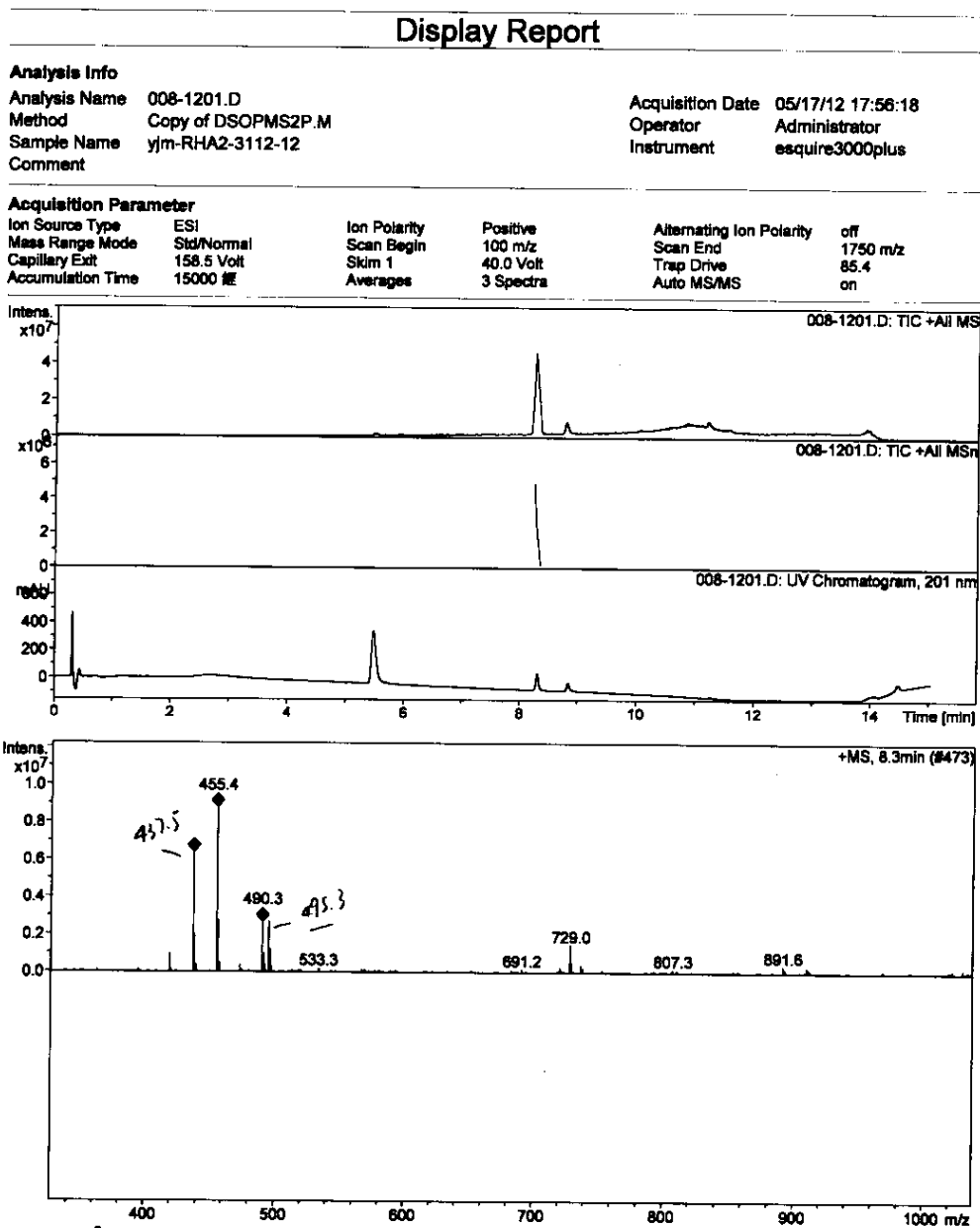


Figure S19. ESI(-)MS spectrum of ricinodol B (2)

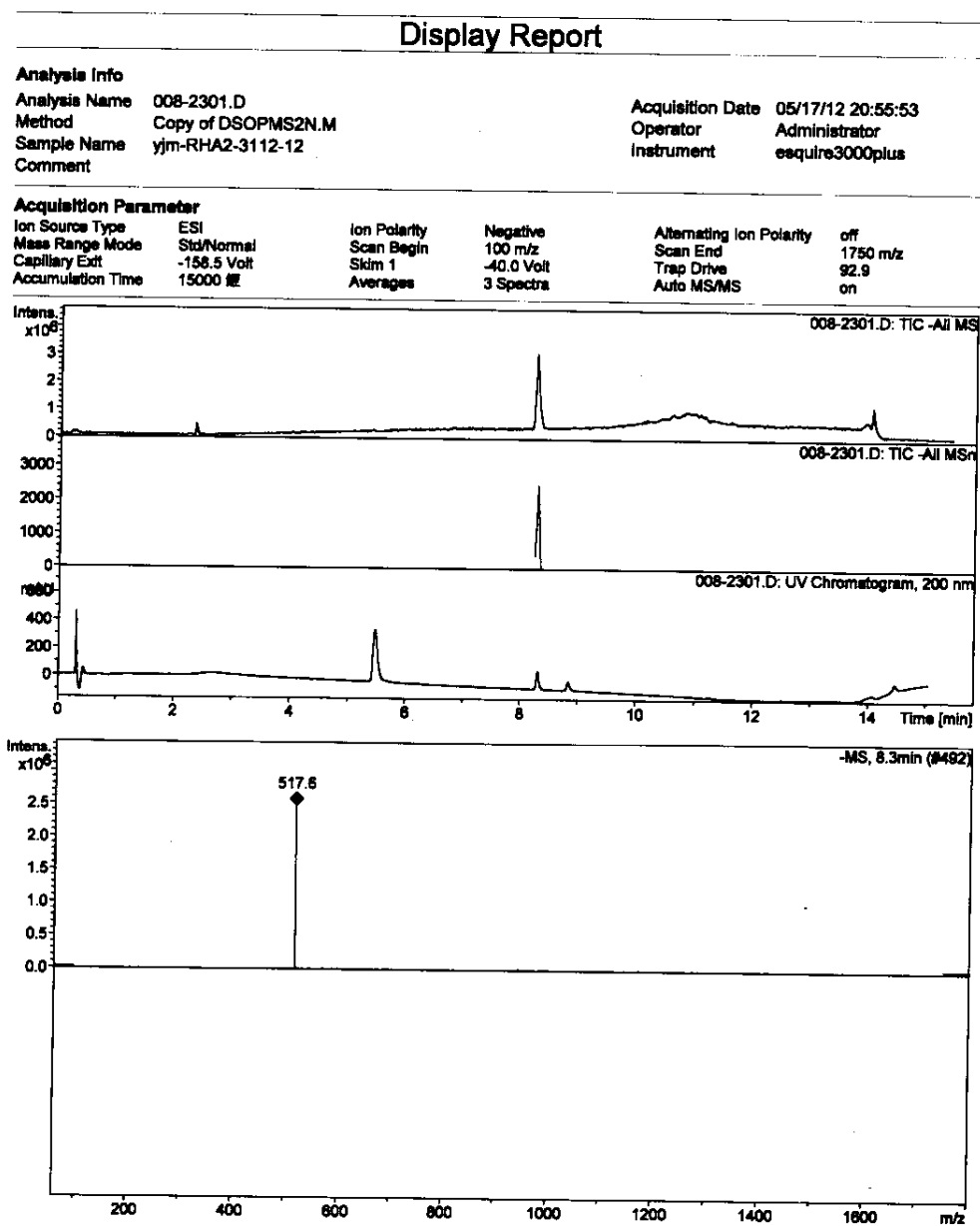


Figure S20. HRESI(+)-MS spectrum of ricinodol B (2)

Elemental Composition Report

Single Mass Analysis

Tolerance = 3.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

213 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:

C: 10-60 H: 1-110 O: 0-30 Na: 0-1

YJH

LCT PXE KE324

18-May-2012

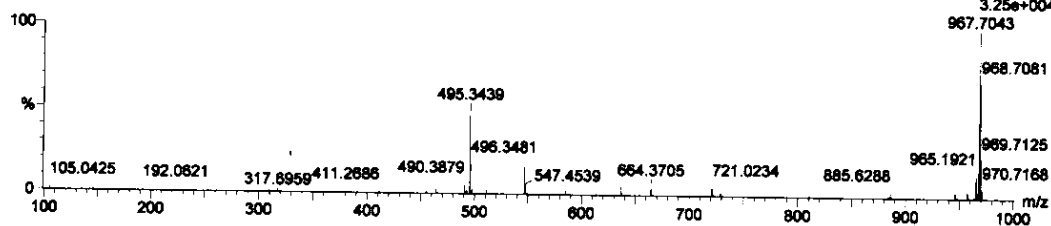
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1: TOF MS ES+

3.25e+004

967.7043

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Minimum:

Maximum: 3.0 3.0 -1.5

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
495.3439	495.3450	-1.1	-2.2	6.5	70.0	0.0	C30 H48 O4 Na

Figure S21. IR spectrum of ricinodol B (2)

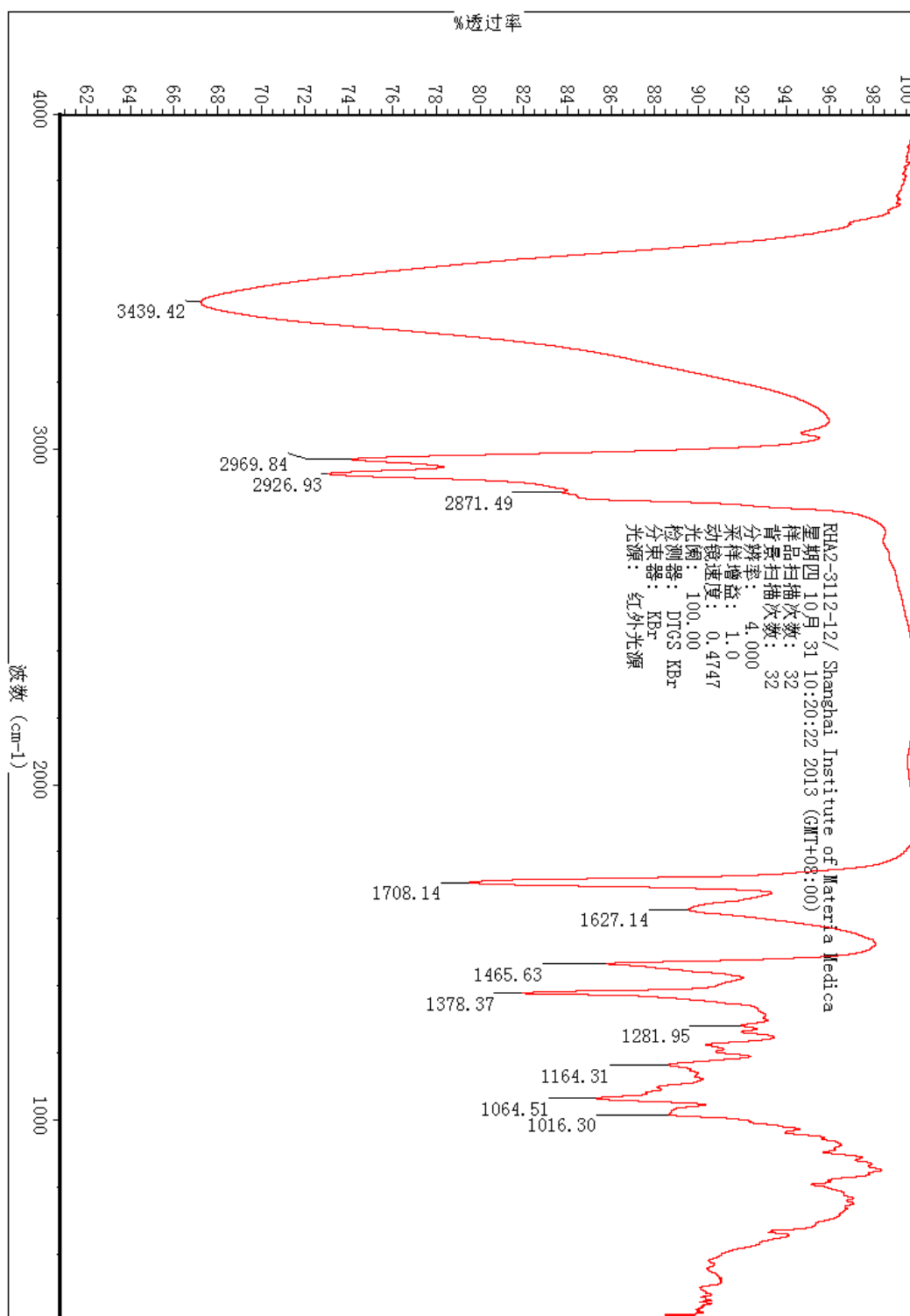


Figure S22. ^1H NMR spectrum of ricinodol C (**3**) in CDCl_3

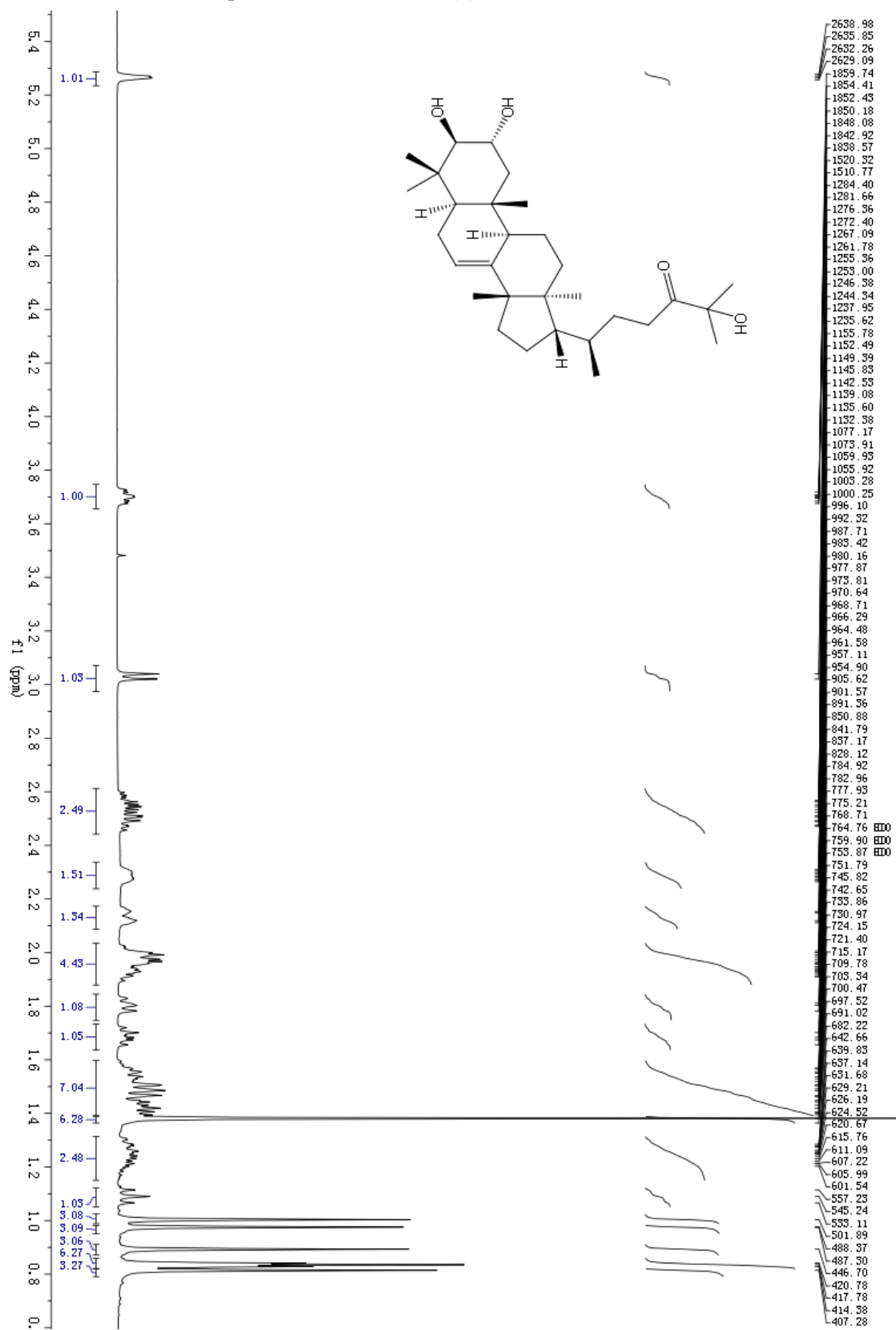


Figure S23. ^{13}C NMR spectrum of ricinodol C (**3**) in CDCl_3

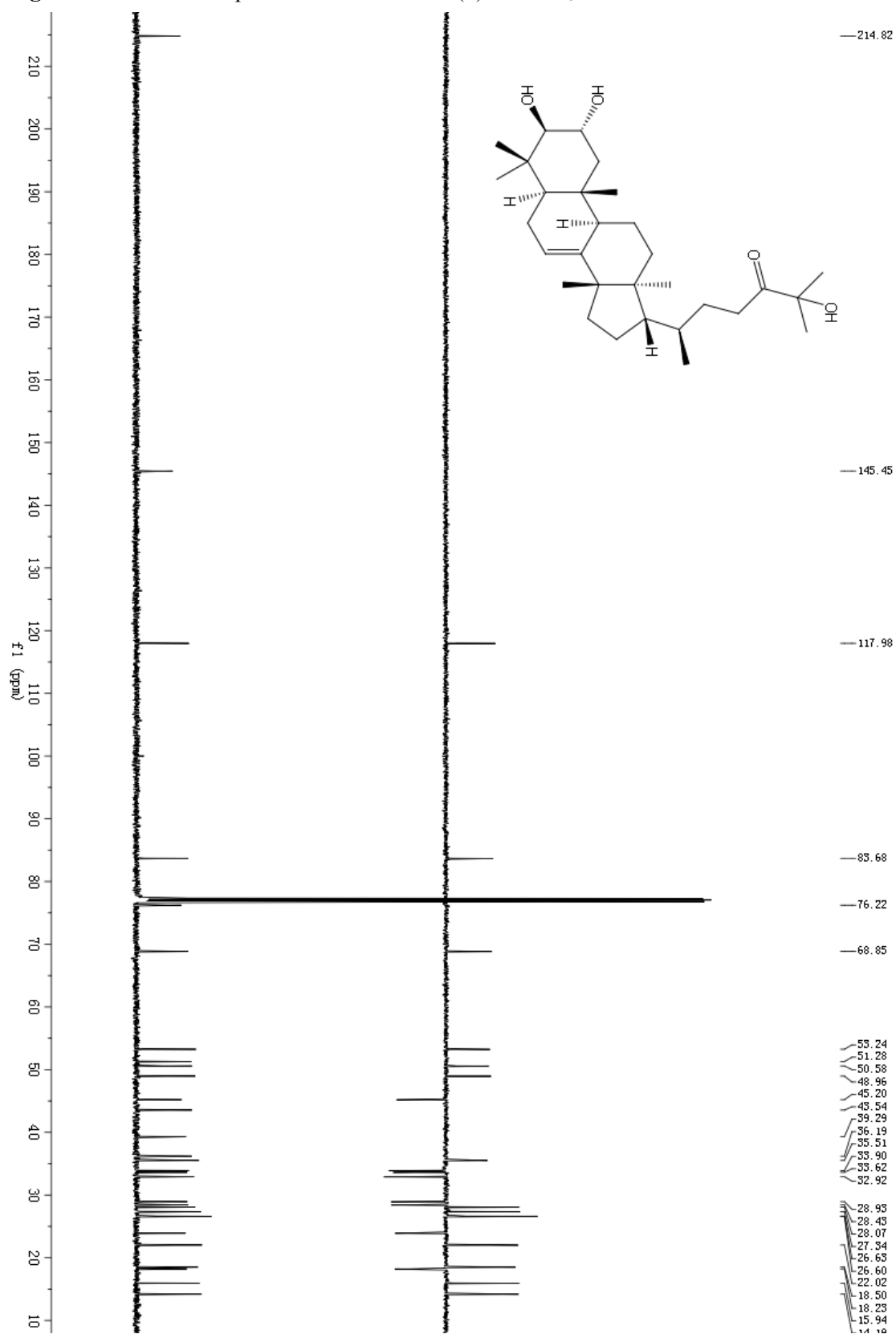


Figure S24. HSQC spectrum of ricinodol C (**3**) in CDCl₃

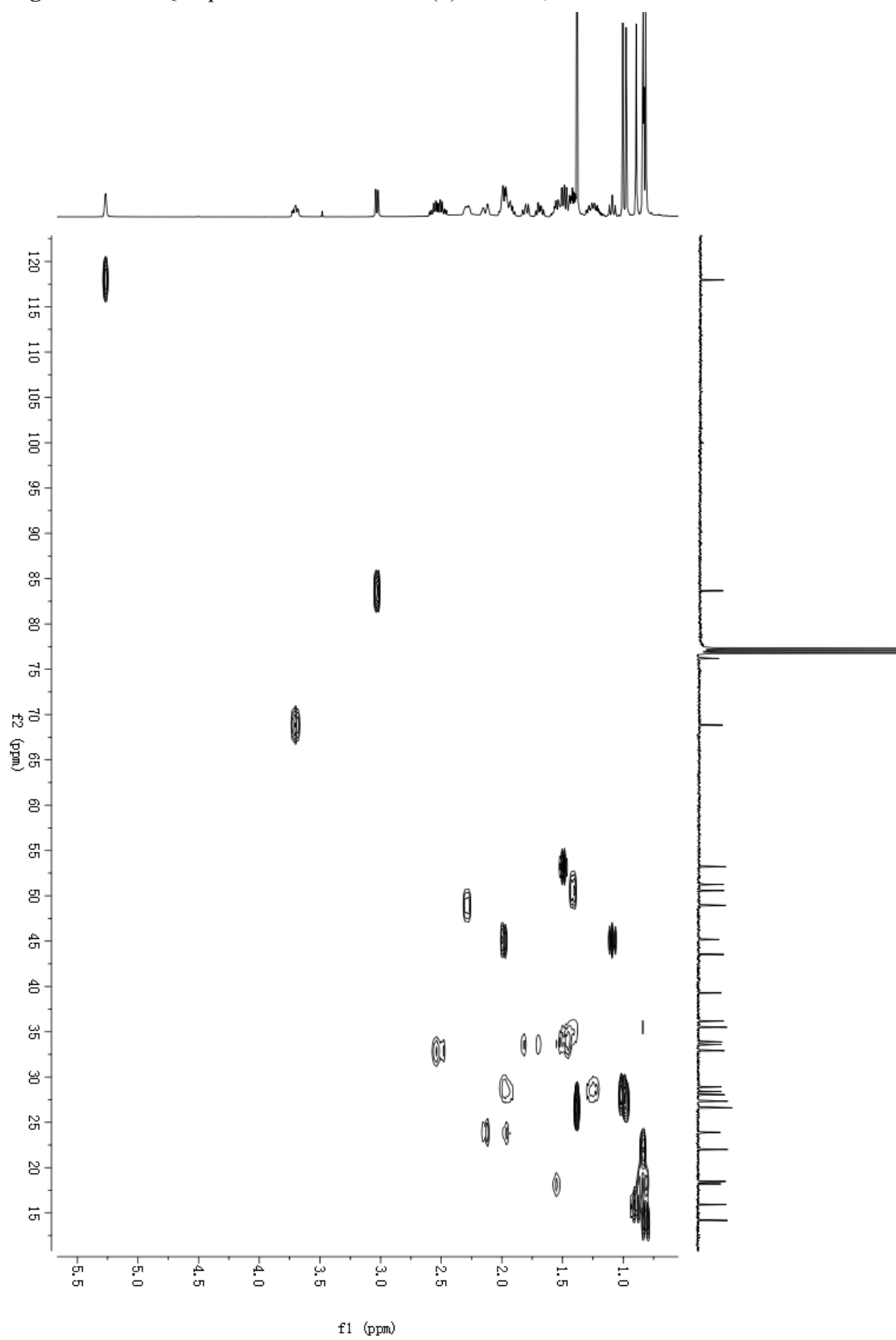


Figure S26. HMBC spectrum of ricinodol C (**3**) in CDCl₃

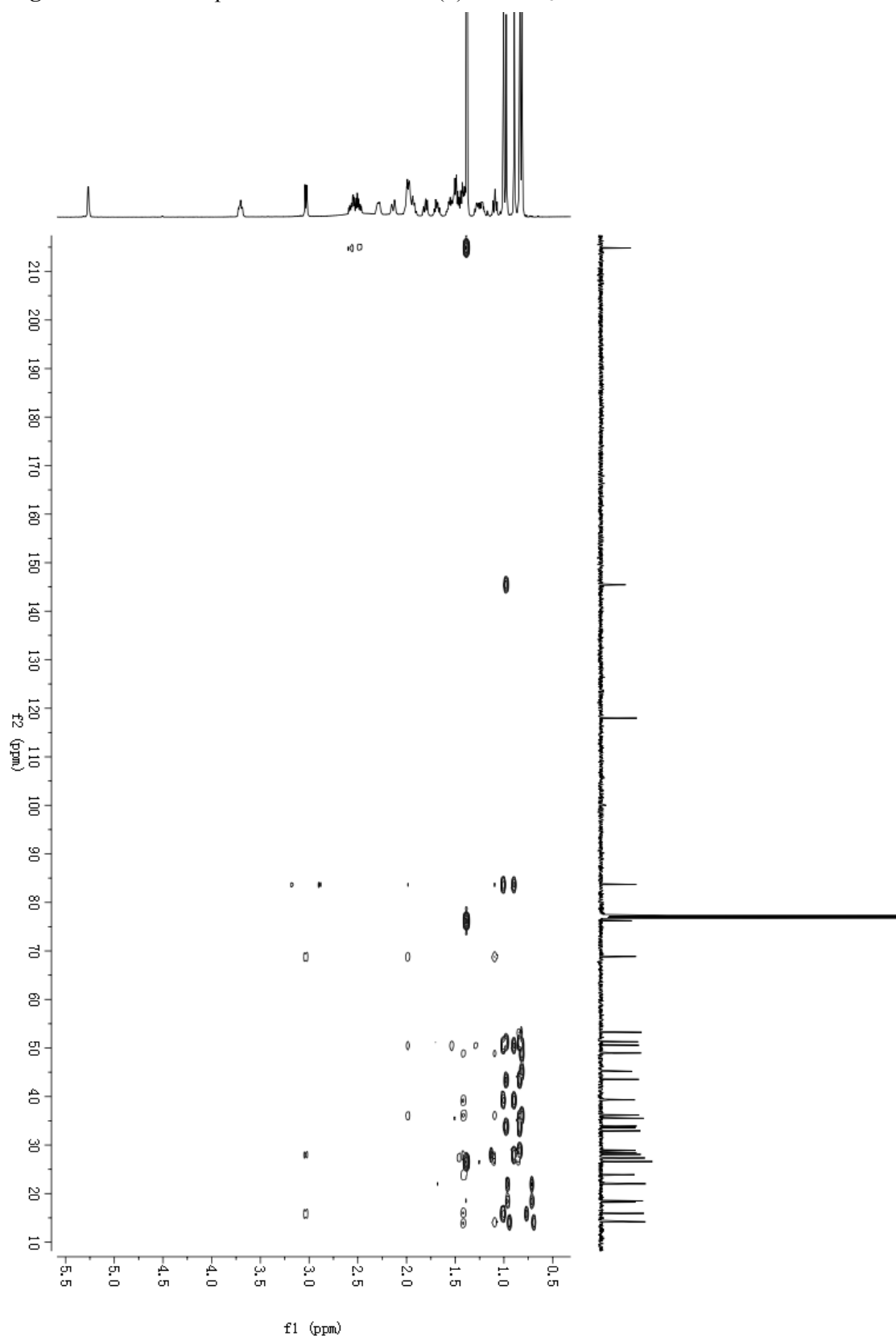


Figure S27. ROESY spectrum of ricinodol C (3) in CDCl₃

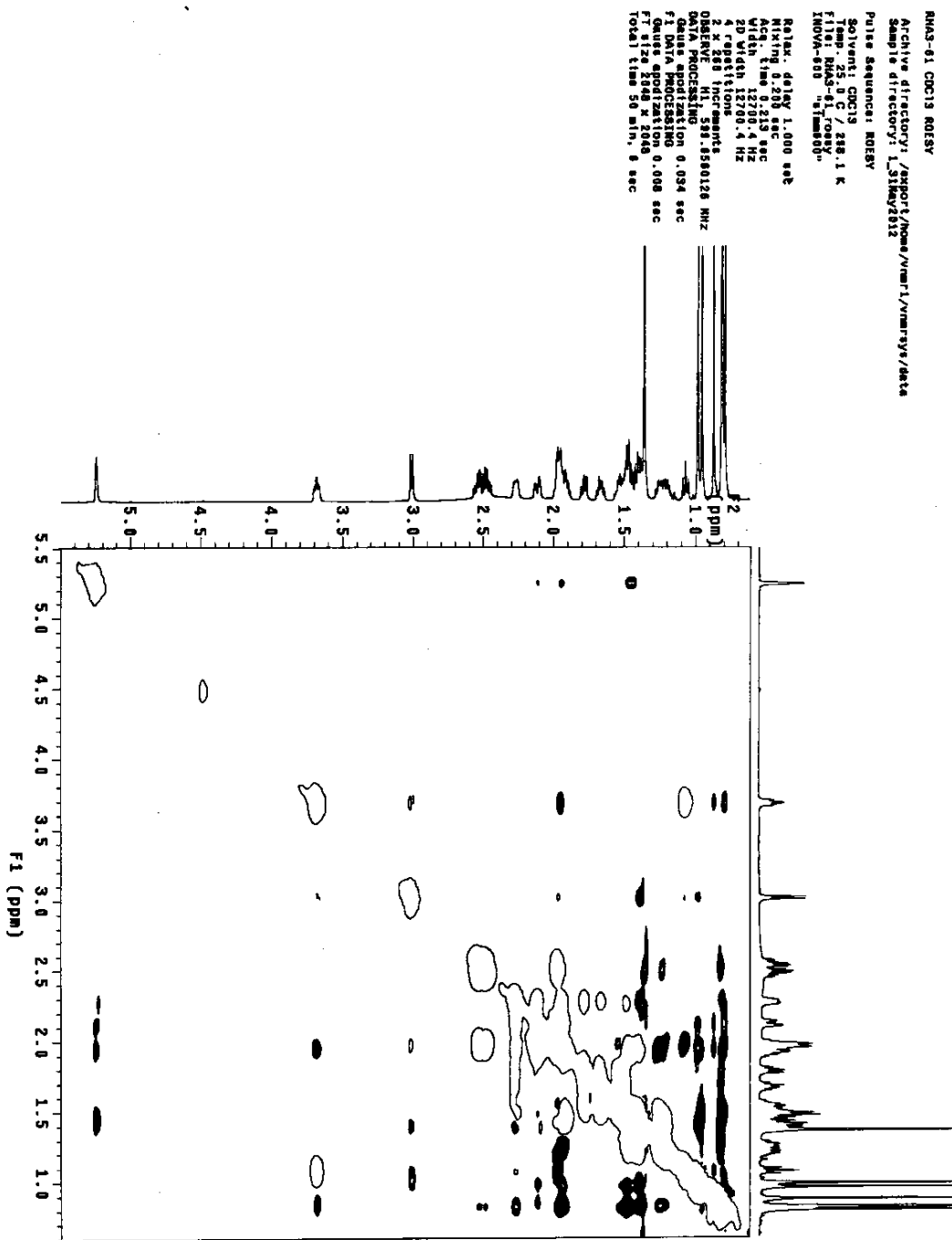


Figure S28. ESI(+)MS spectrum of ricinodol C (3)

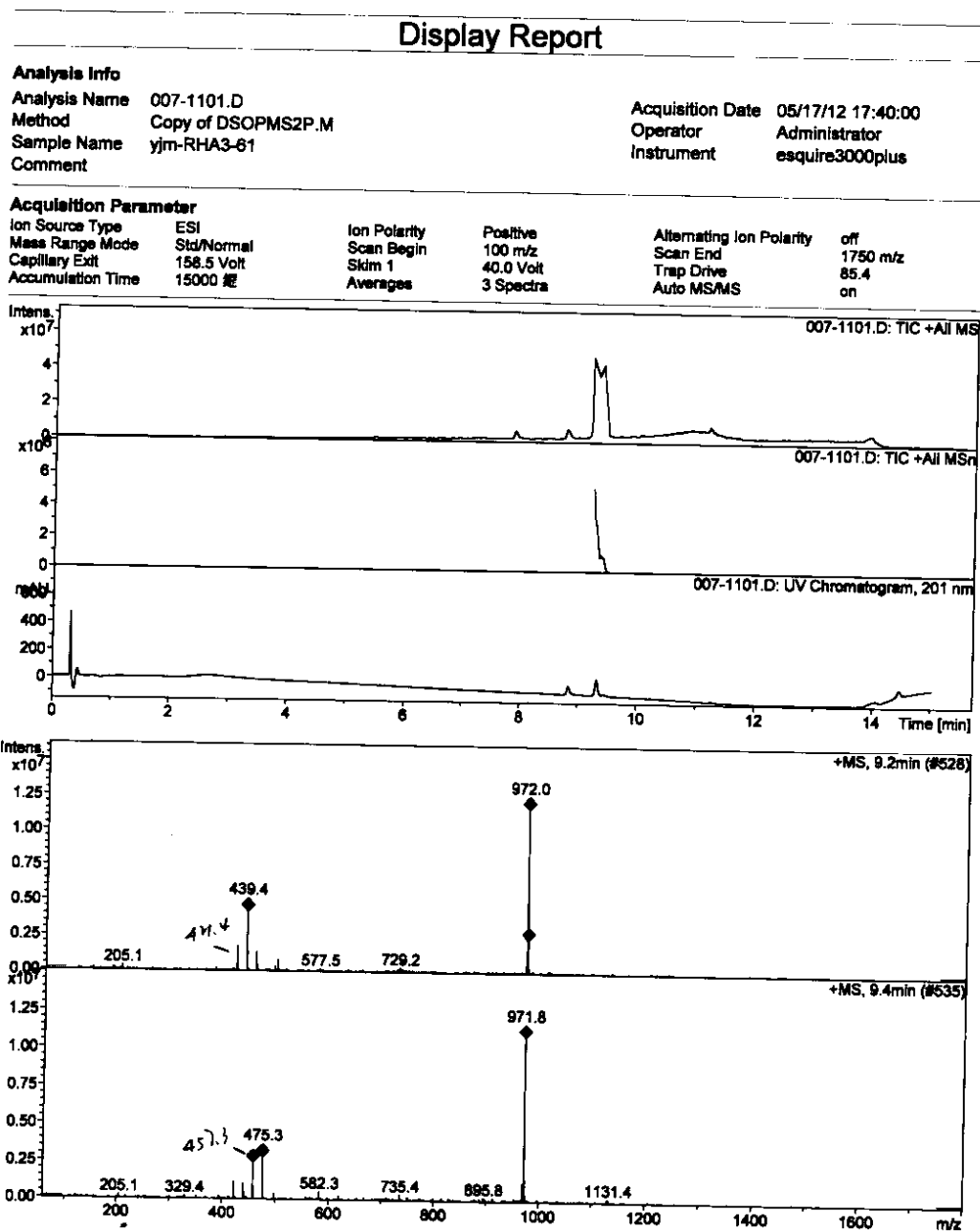


Figure S29. ESI(-)MS spectrum of ricinodol C (3)

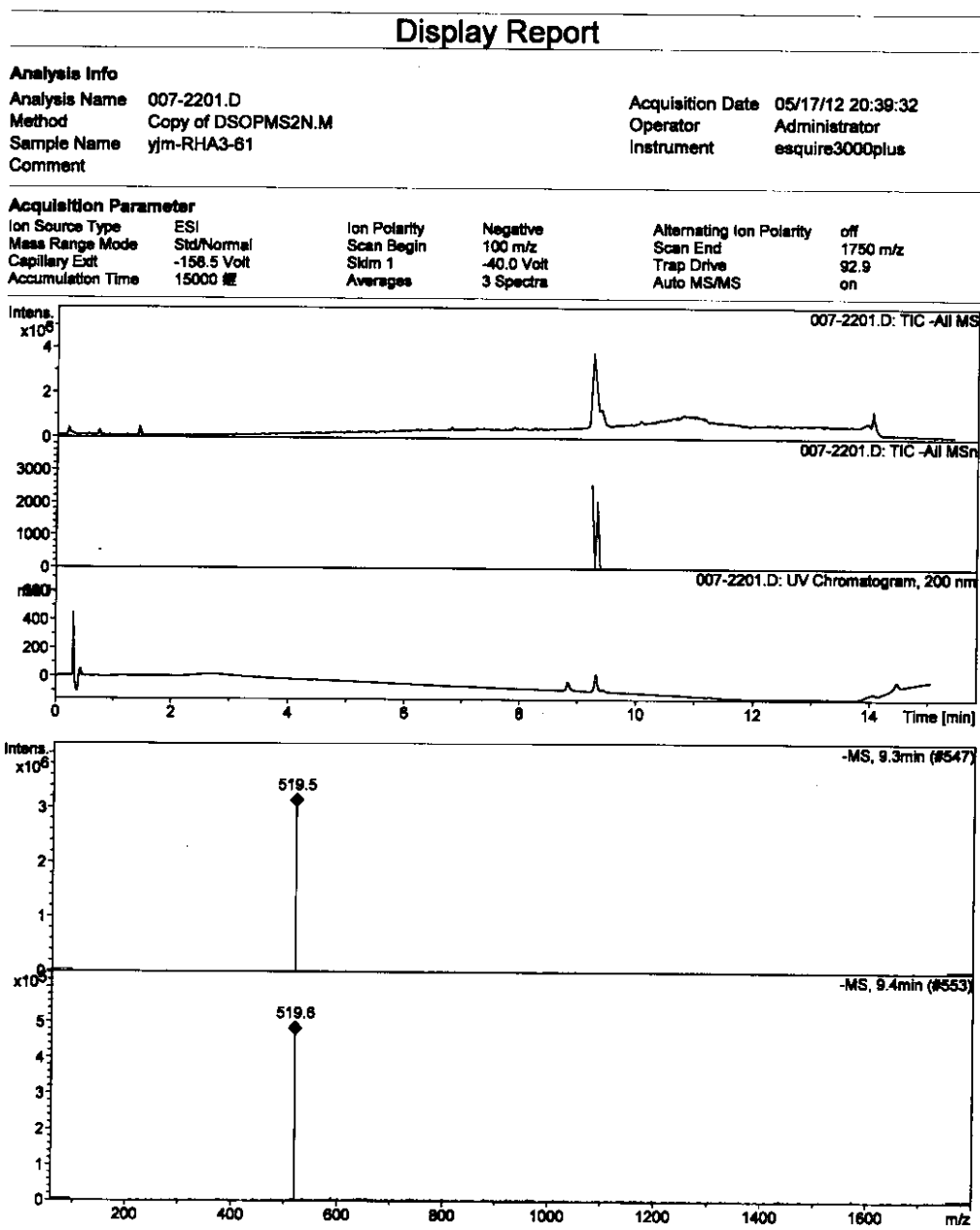


Figure S30. HRESI(-)MS spectrum of ricinodol C (3)

Elemental Composition Report

Single Mass Analysis

Tolerance = 3.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

121 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:

C: 10-60 H: 1-110 O: 0-30

YJH

LCT PXE KE324

18-May-2012

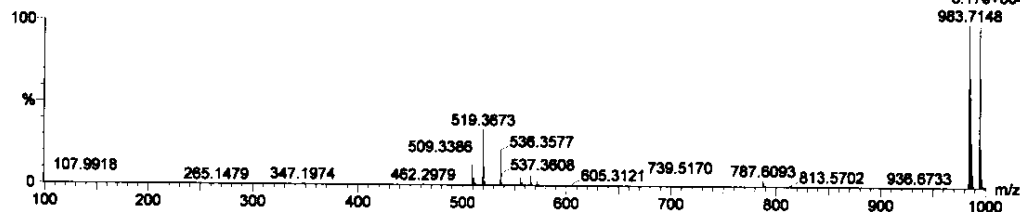
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8.17e+004

983.7148

RHA3-61 16 (0.351) AM2 (Ar,10000.0,0.00,1.00); ABS; Cm (5:30)



Minimum:

Maximum: 3.0 3.0 -1.5

Maximum: 3.0 3.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
519.3673	519.3686	-1.3	-2.5	6.5	123.9	0.0	C31 H51 O6

Figure S31. IR spectrum of ricinodol C (3)

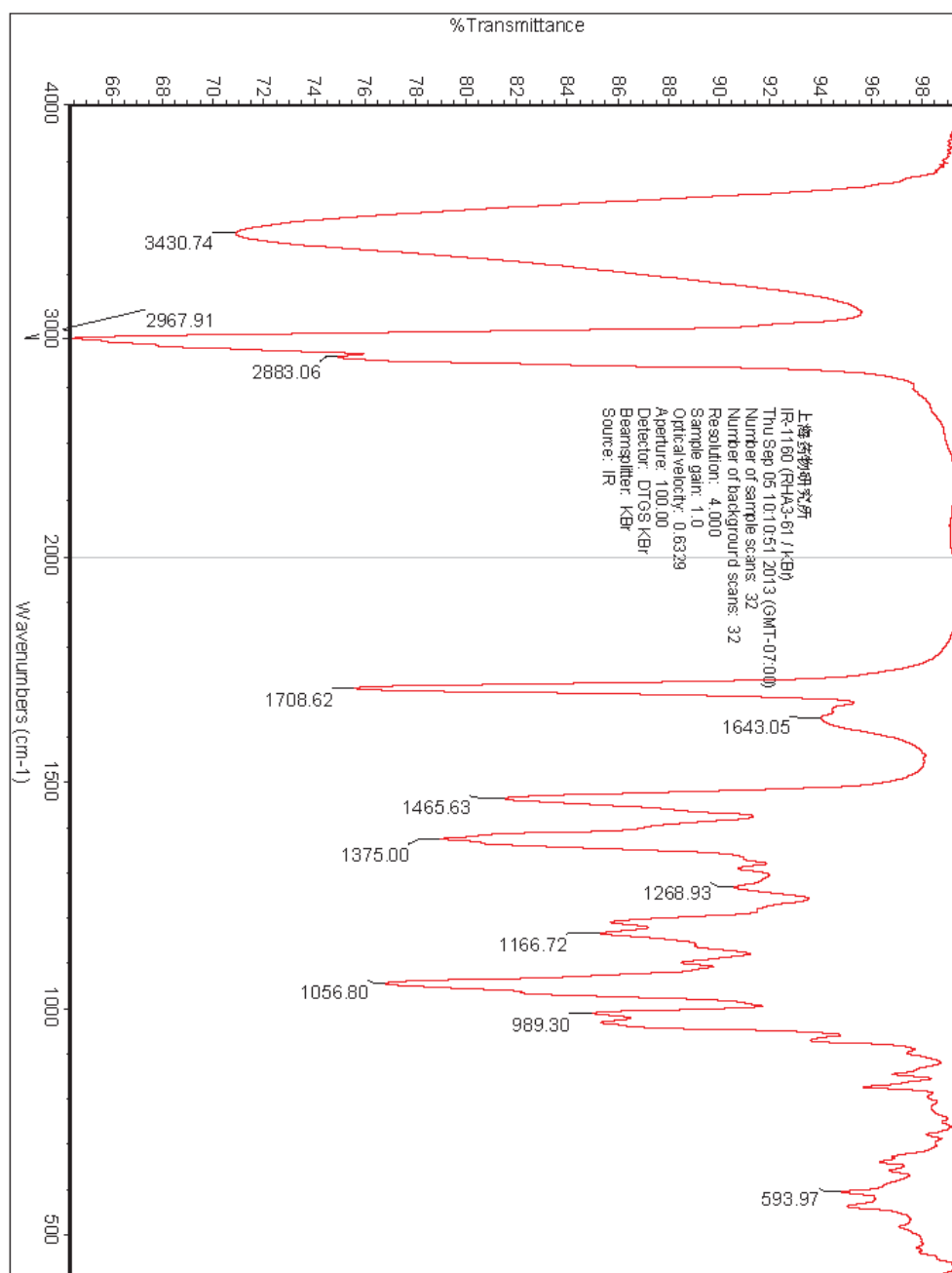


Figure S32. ^1H NMR spectrum of ricinodol D (**4**) in CDCl_3

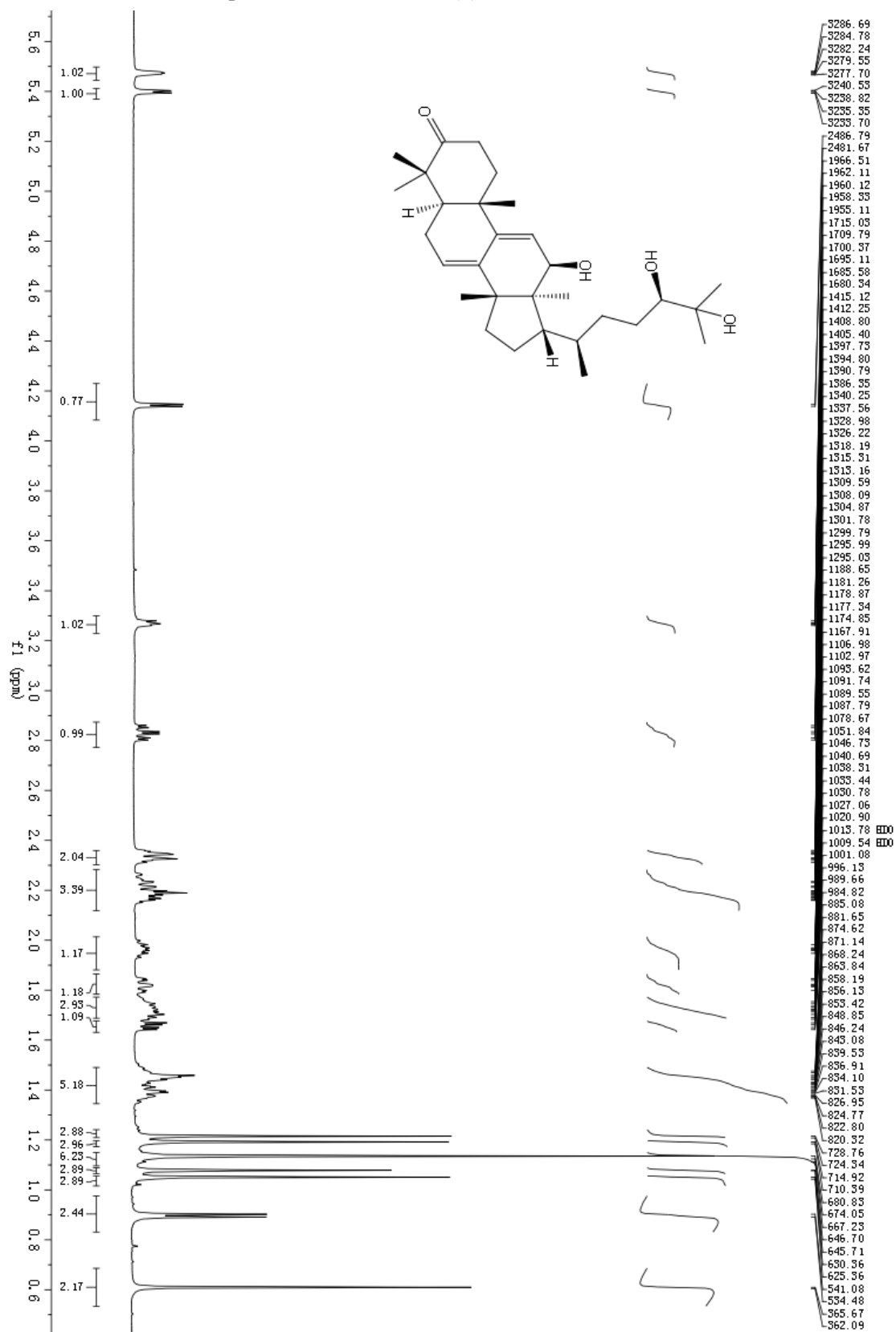


Figure S33. ^{13}C NMR spectrum of ricinodol D (**4**) in CDCl_3

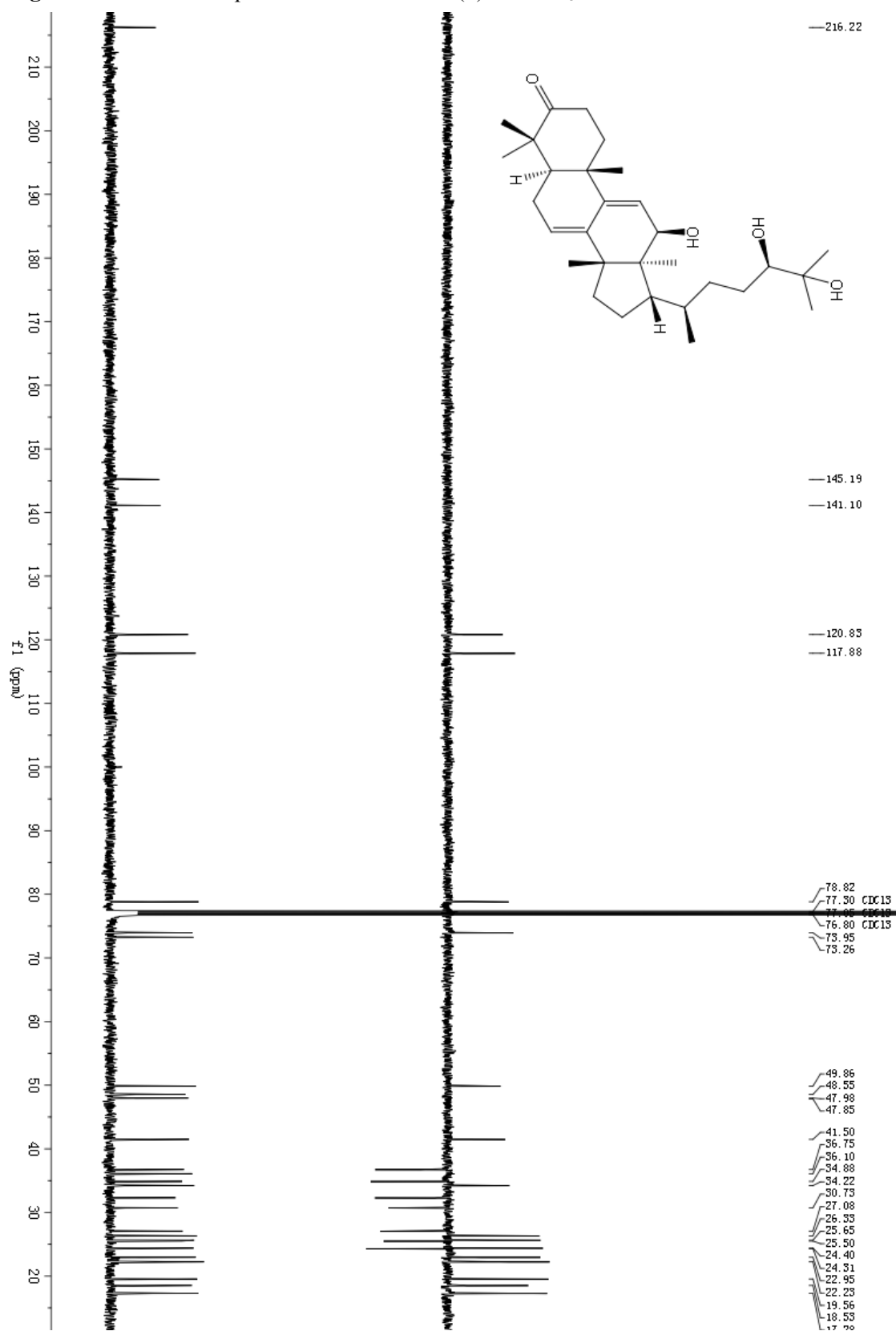


Figure S34. HSQC spectrum of ricinodol D (**4**) in CDCl₃

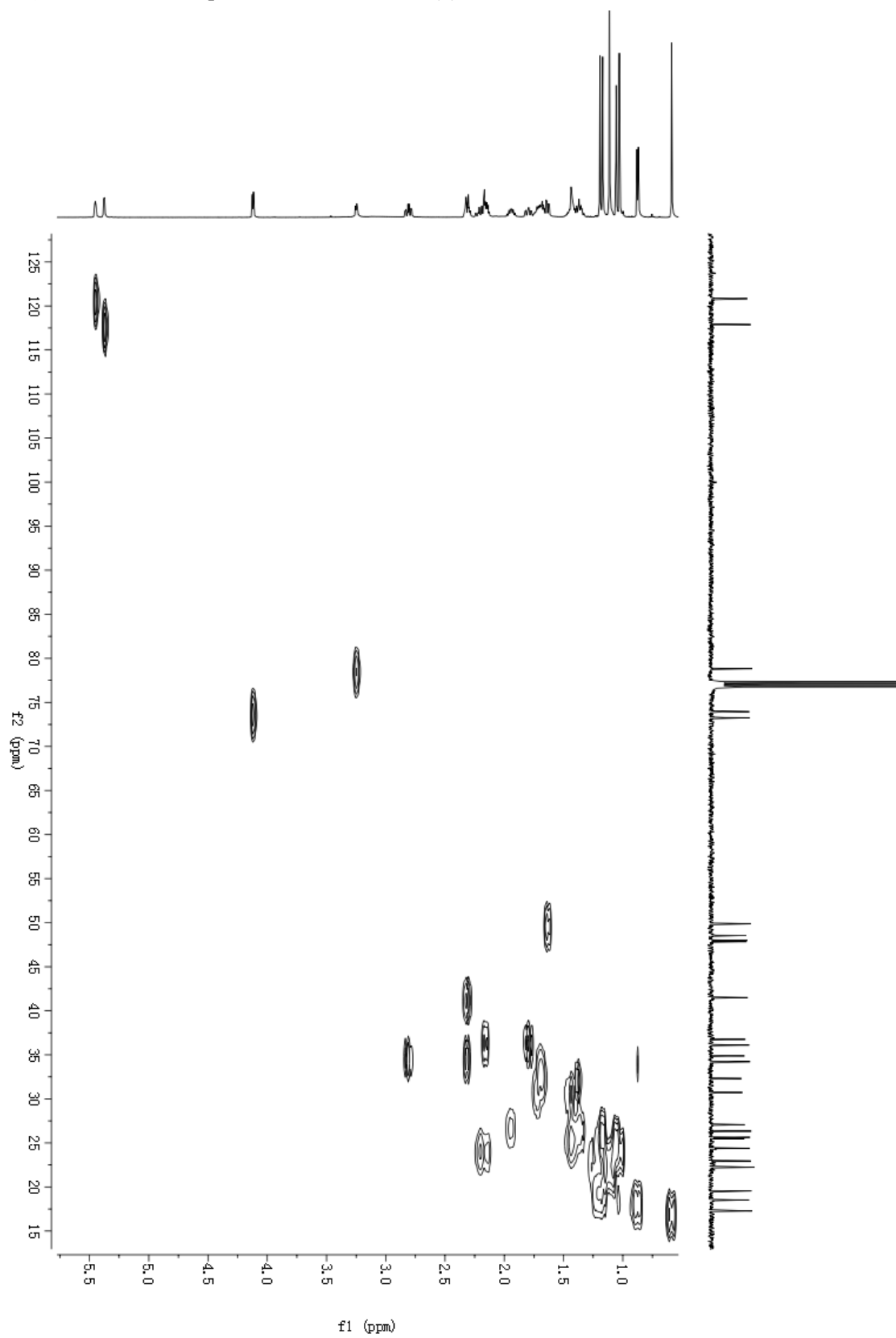


Figure S35. HMBC spectrum of ricinodol D (**4**) in CDCl₃

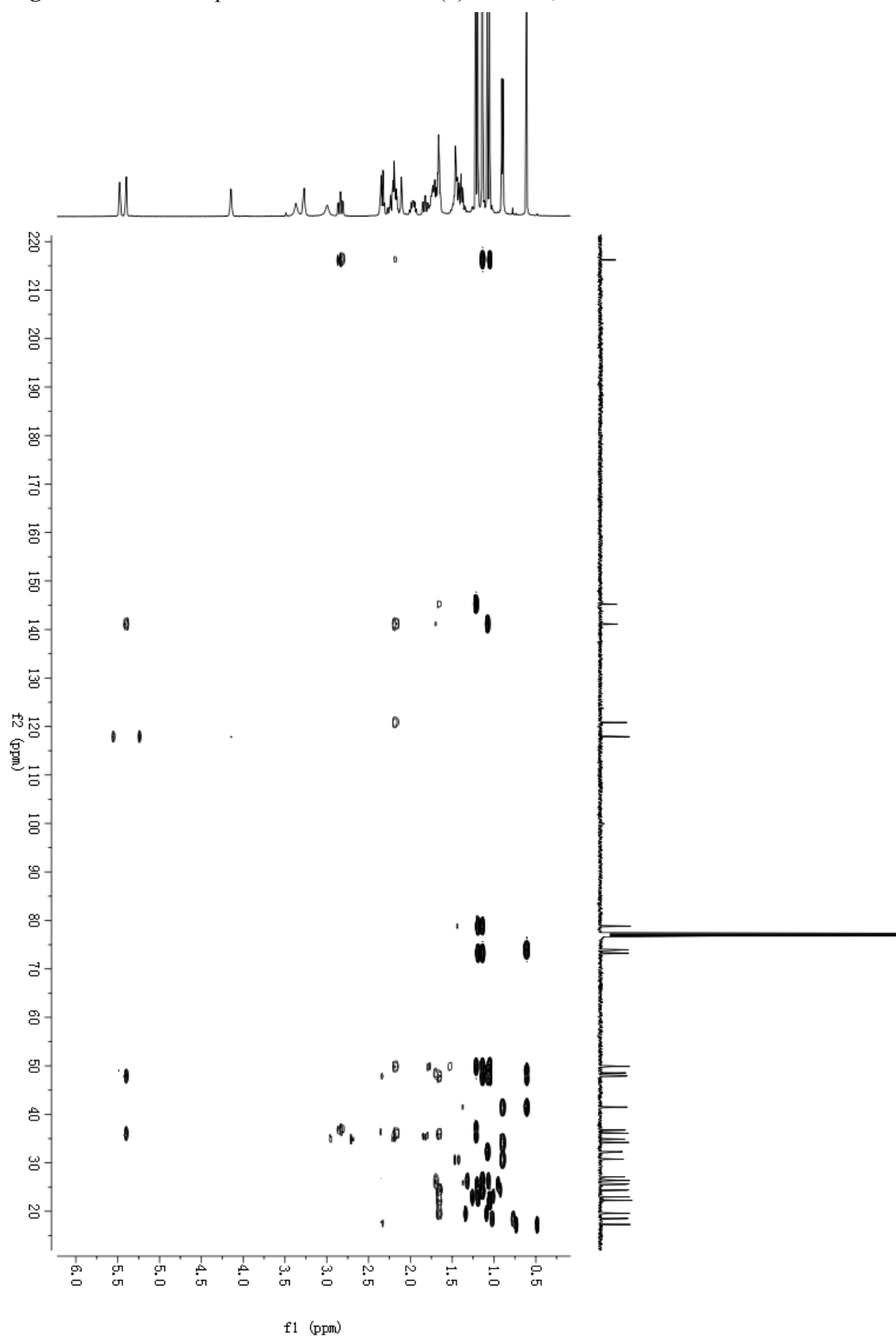


Figure S36. ROESY spectrum of ricinodol C (4) in CDCl₃

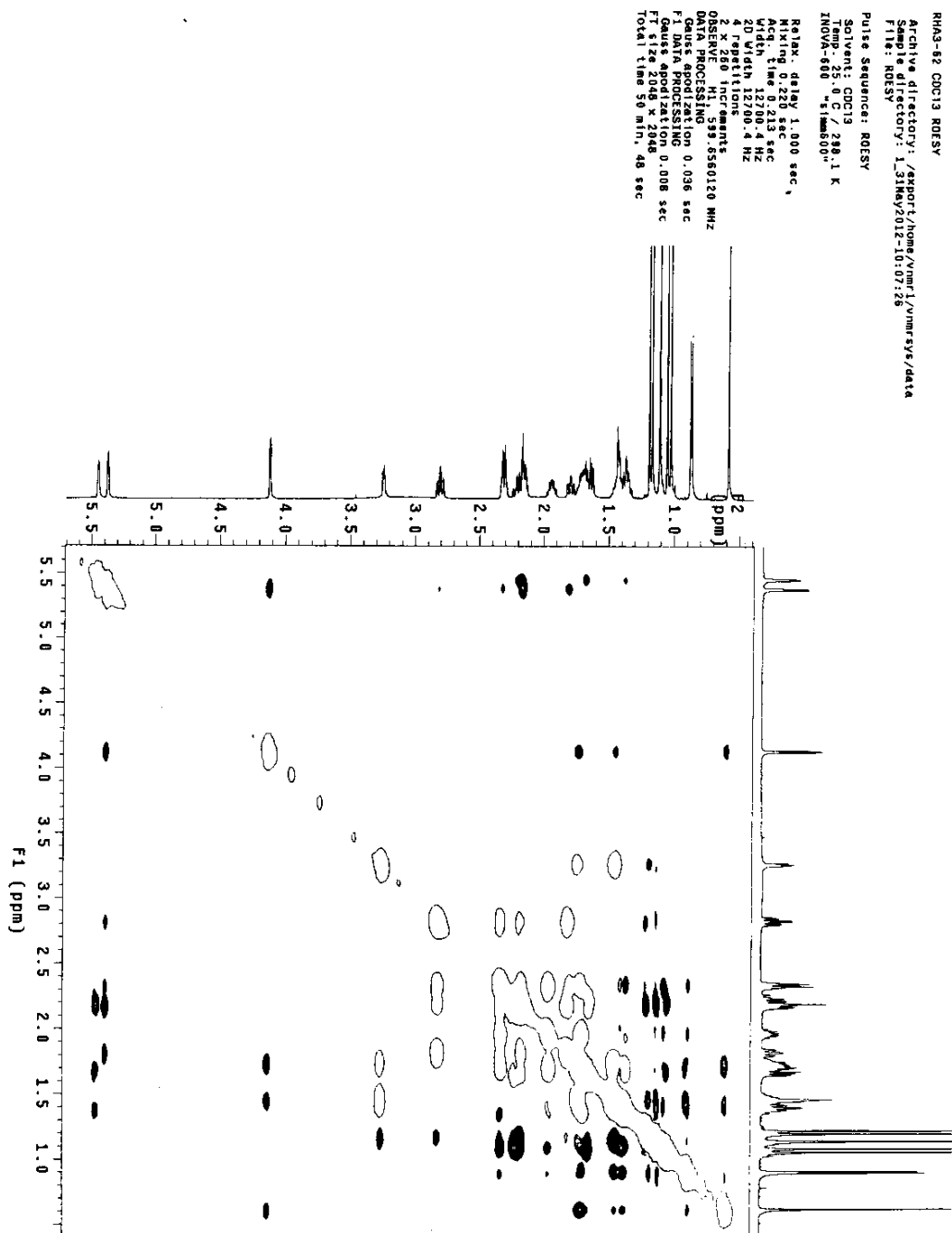


Figure S37. ESI(+)MS spectrum of ricinodol D (4)

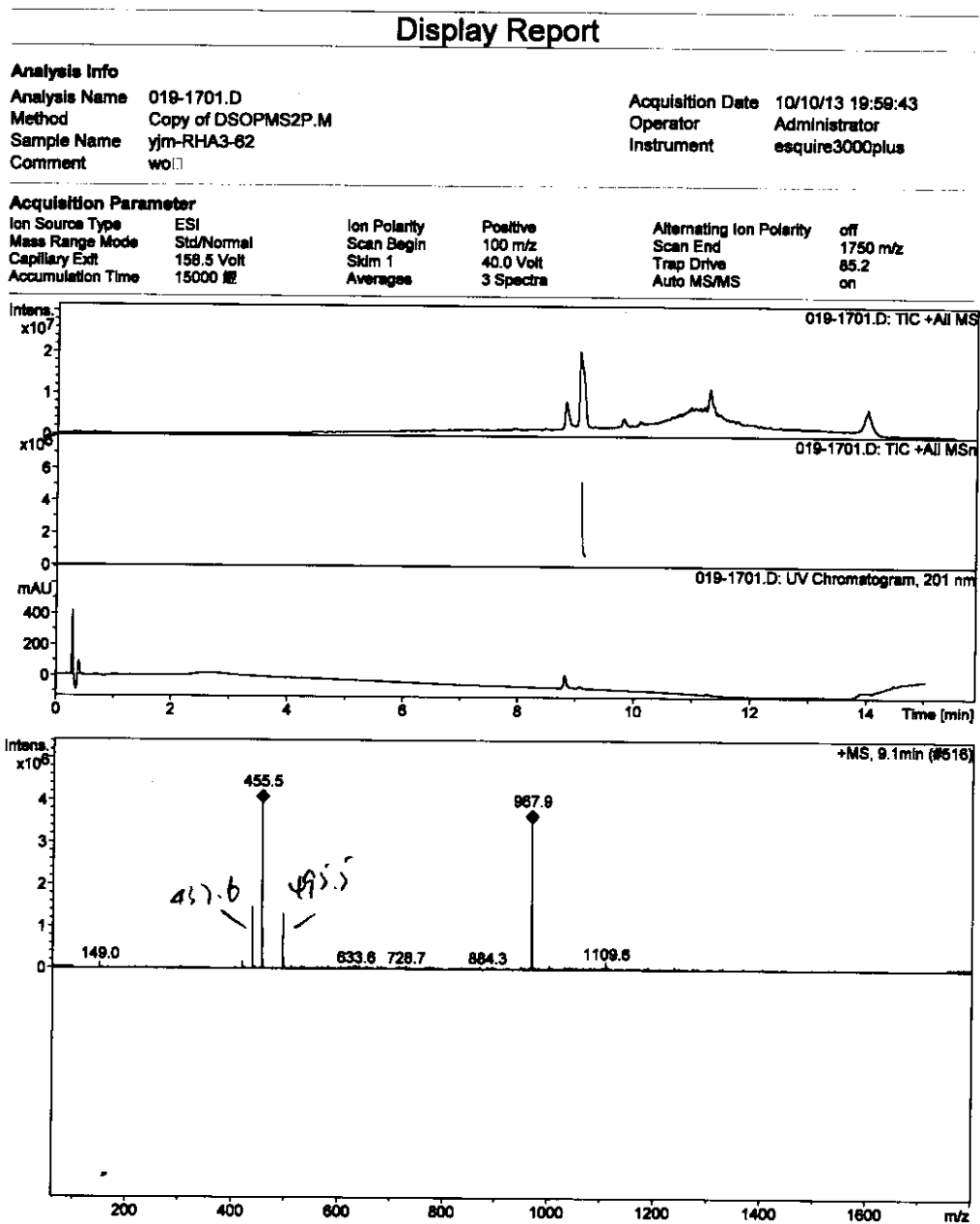


Figure S38. ESI(-)MS spectrum of ricinodol D (4)

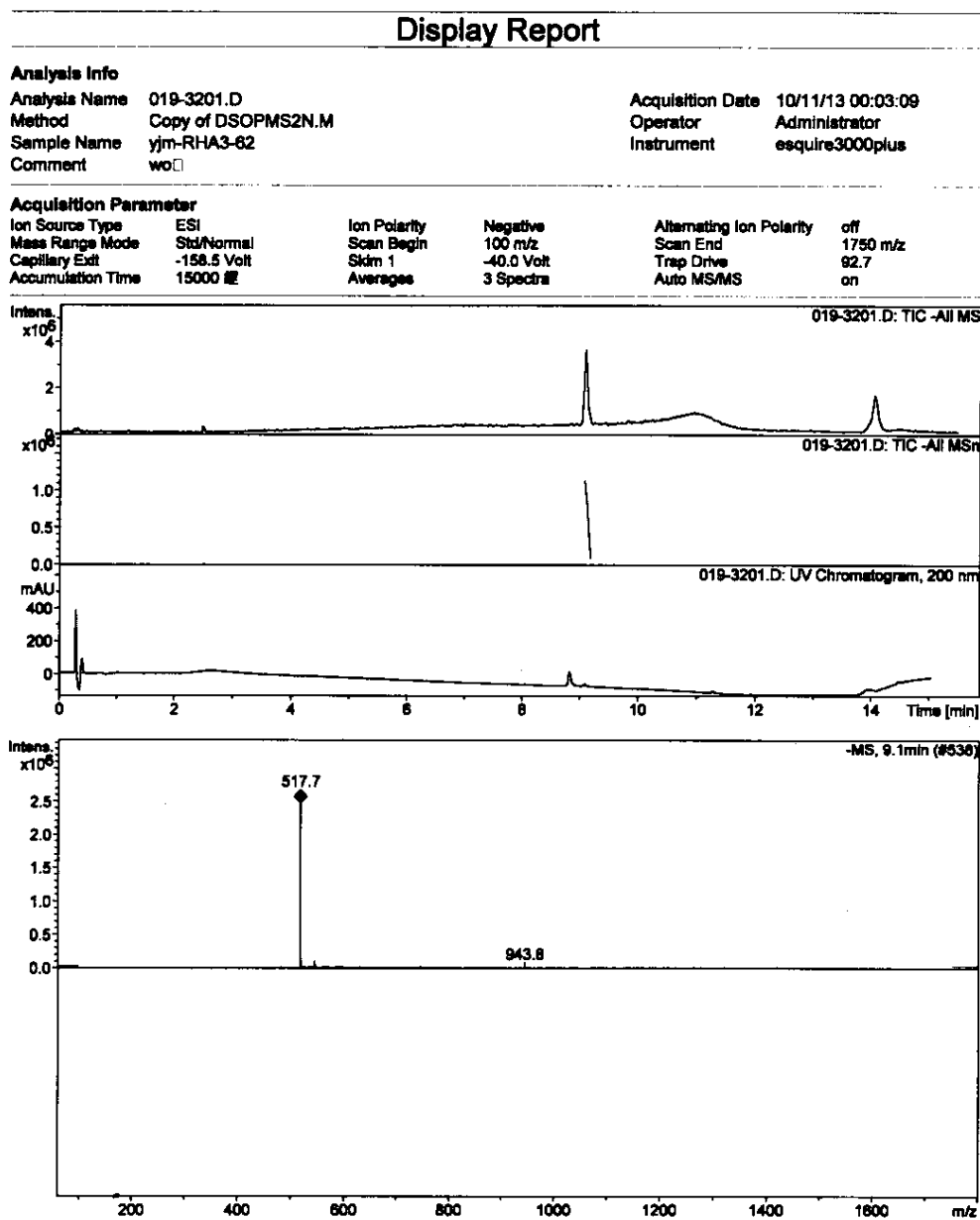


Figure S39. HRESI(+)MS spectrum of ricinodol D (4)

Elemental Composition Report

Single Mass Analysis

Tolerance = 4.0 PPM / DBE: min = -1.5, max = 50.0
 Element prediction: Off
 Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

191 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 5-80 H: 2-120 O: 0-20 Na: 0-1

RHA3-62

LCT PXE KE324

03-Sep-2013

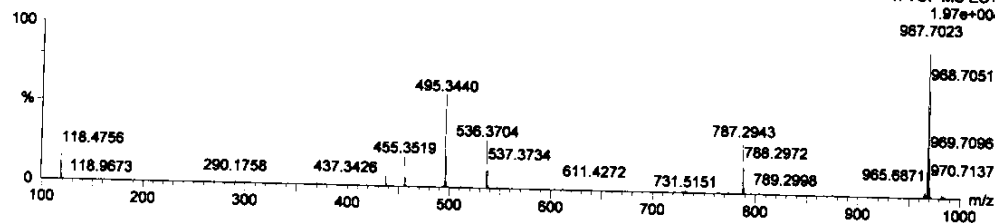
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1.97e+004

987.7023

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Minimum:

Maximum:

3.0 4.0 -1.5

50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
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495.3440	495.3450	-1.0	-2.0	6.5	60.8	0.0	C30 H48 O4 Na
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Figure S40. IR spectrum of ricinodol D (4)

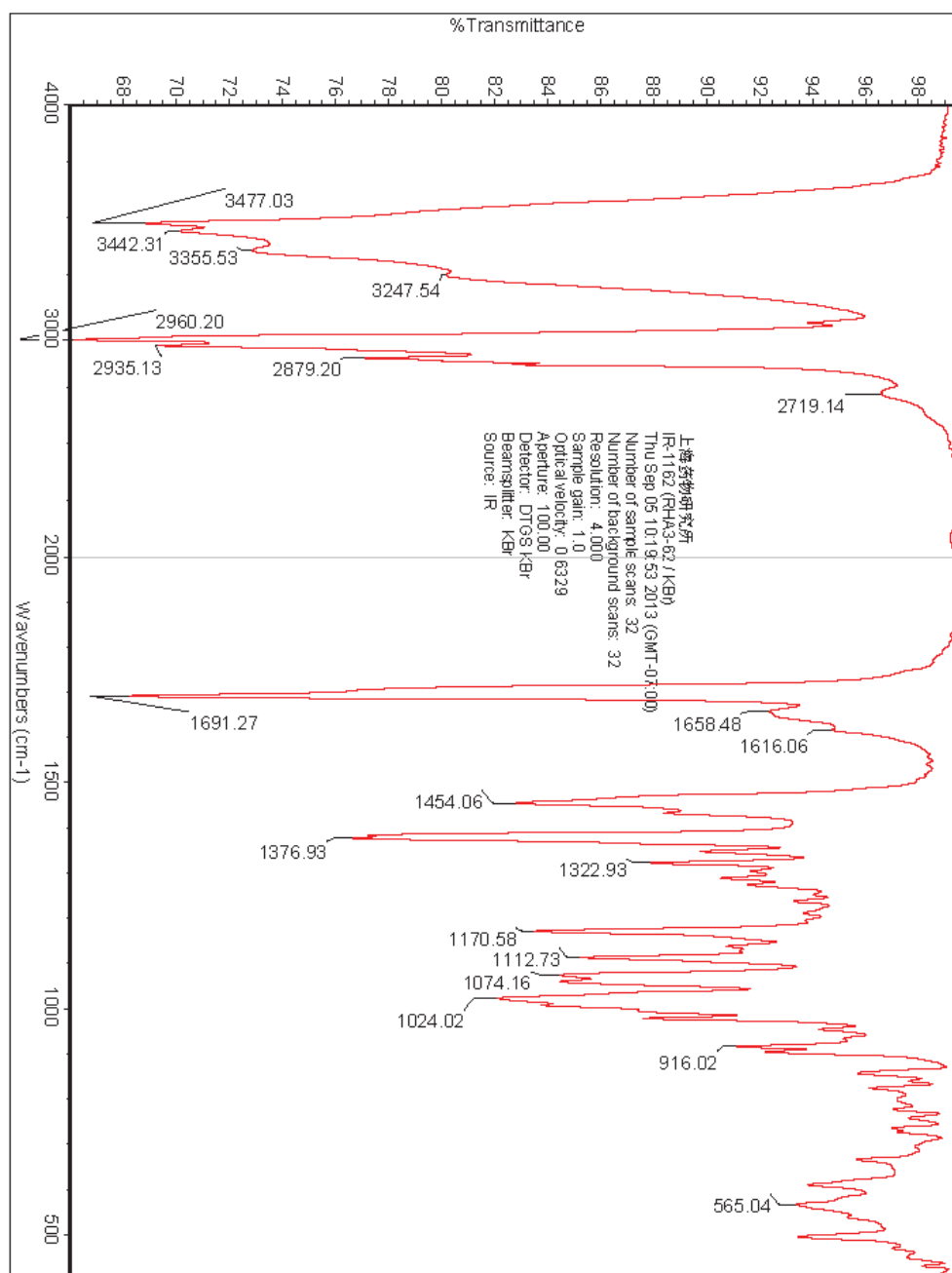


Figure S41. ^1H NMR spectrum of ricinodol E (**5**) in CDCl_3

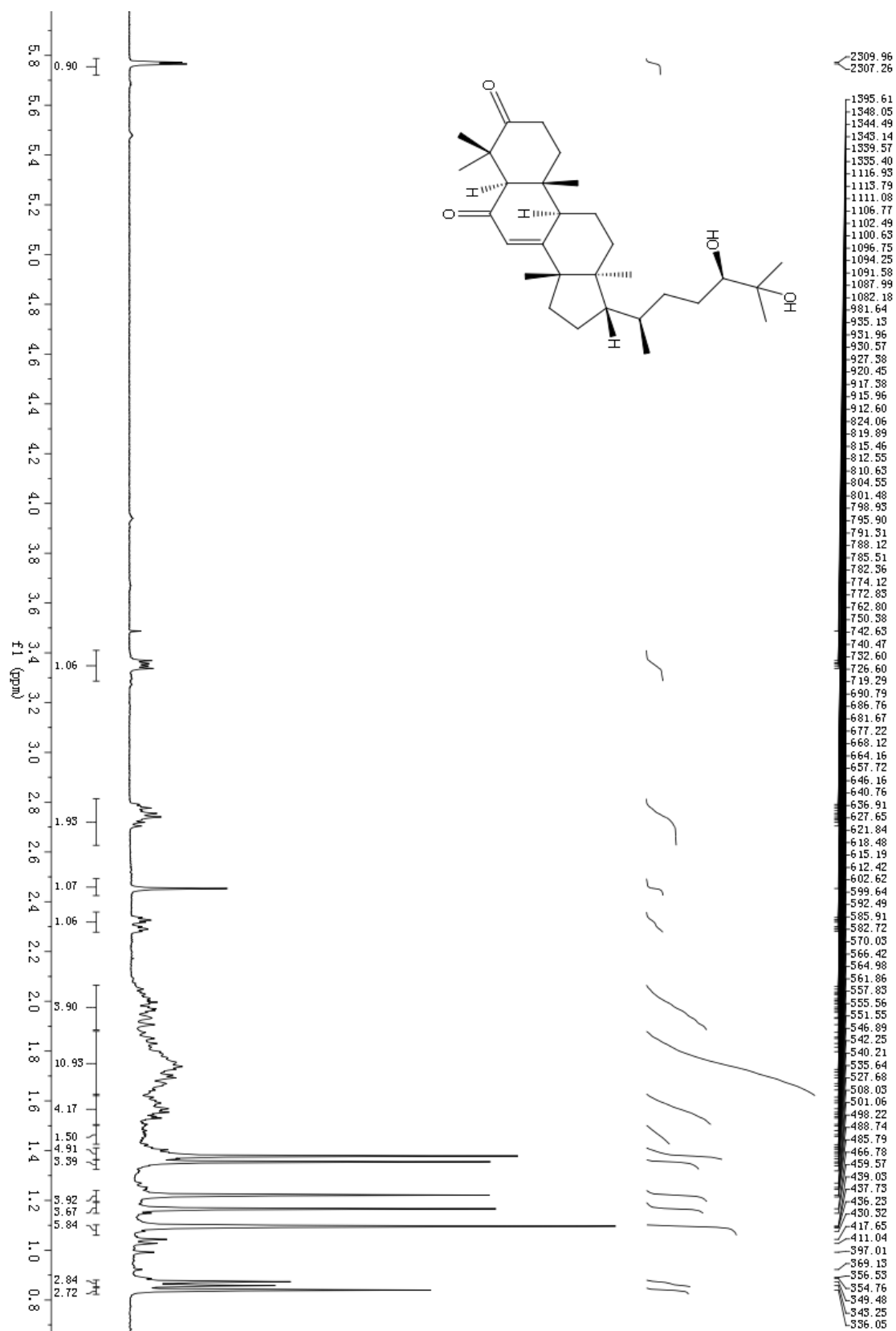


Figure S42. ^{13}C NMR spectrum of ricinodol E (5) in CDCl_3

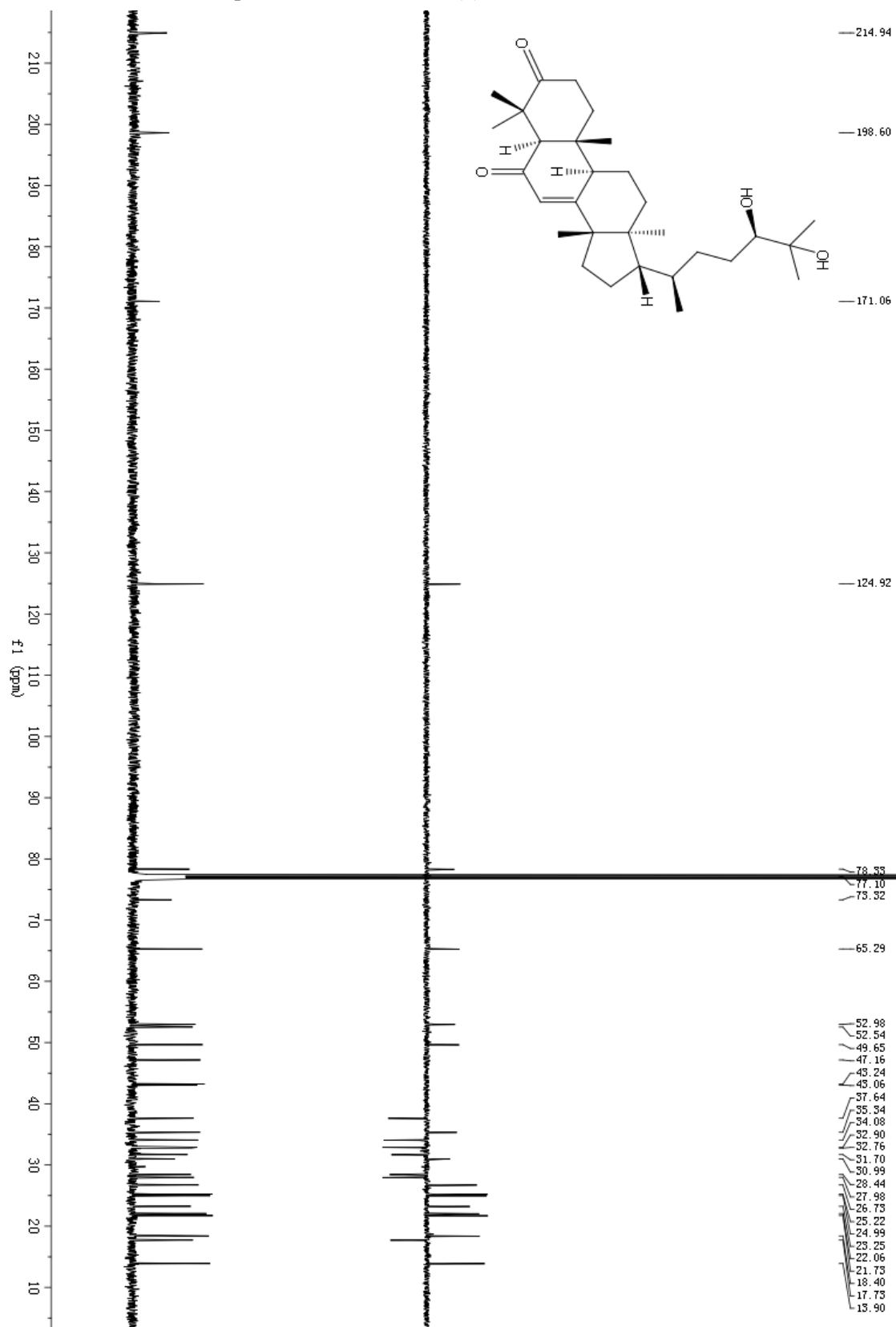


Figure S43. HSQC spectrum of ricinodol E (**5**) in CDCl₃

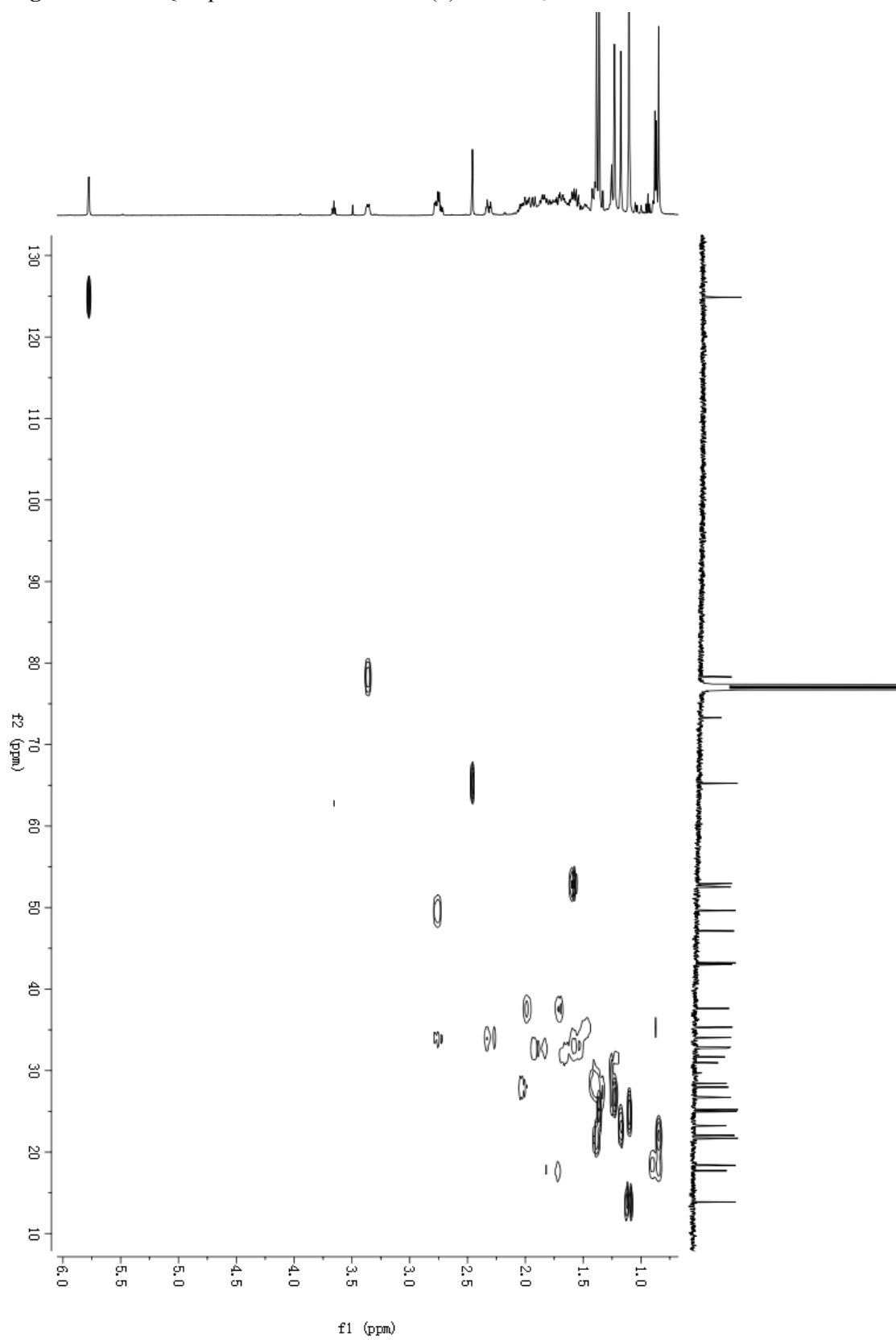


Figure S44. HMBC spectrum of ricinodol E (**5**) in CDCl₃

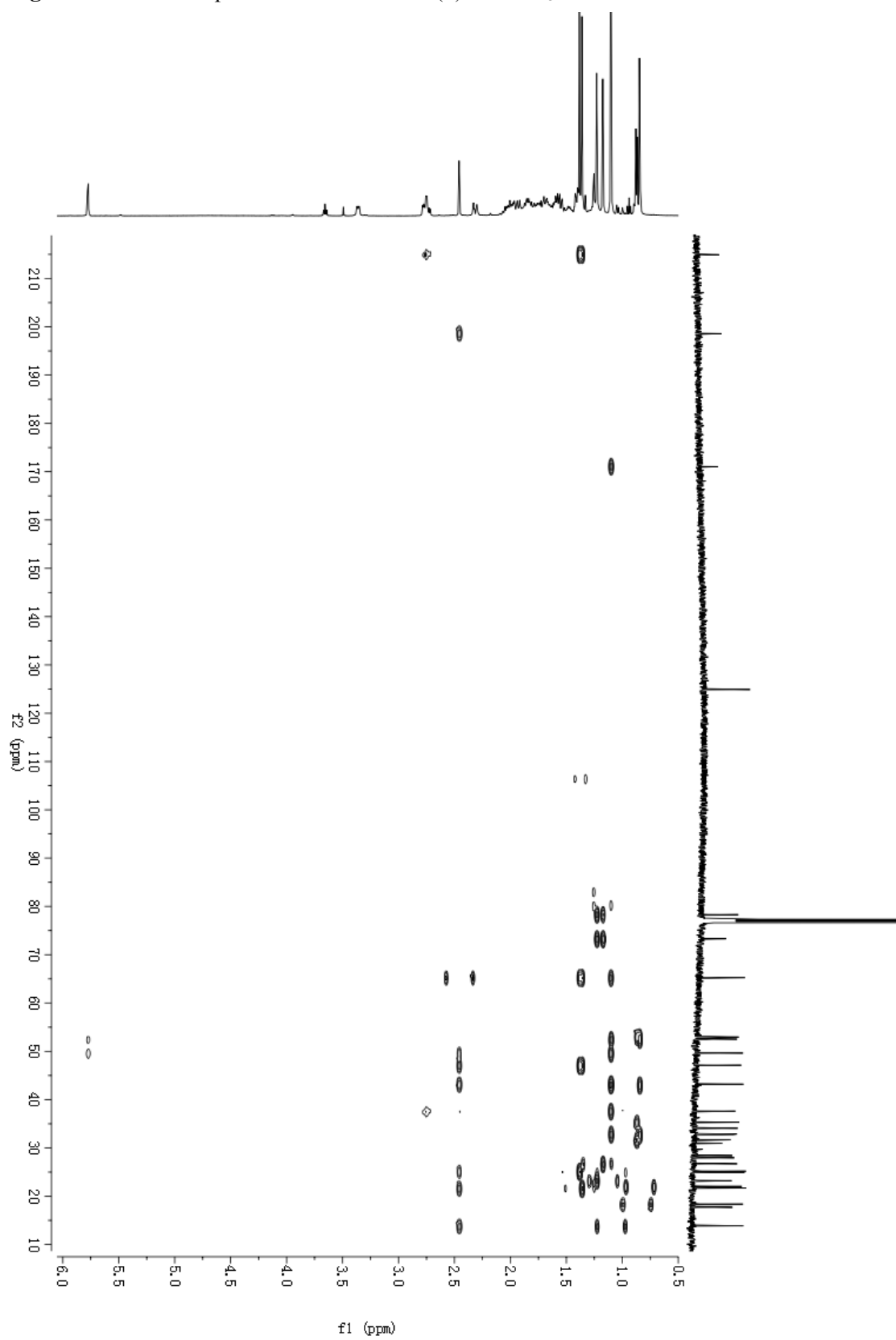


Figure S45. ROESY spectrum of ricinodol E (5) in CDCl₃

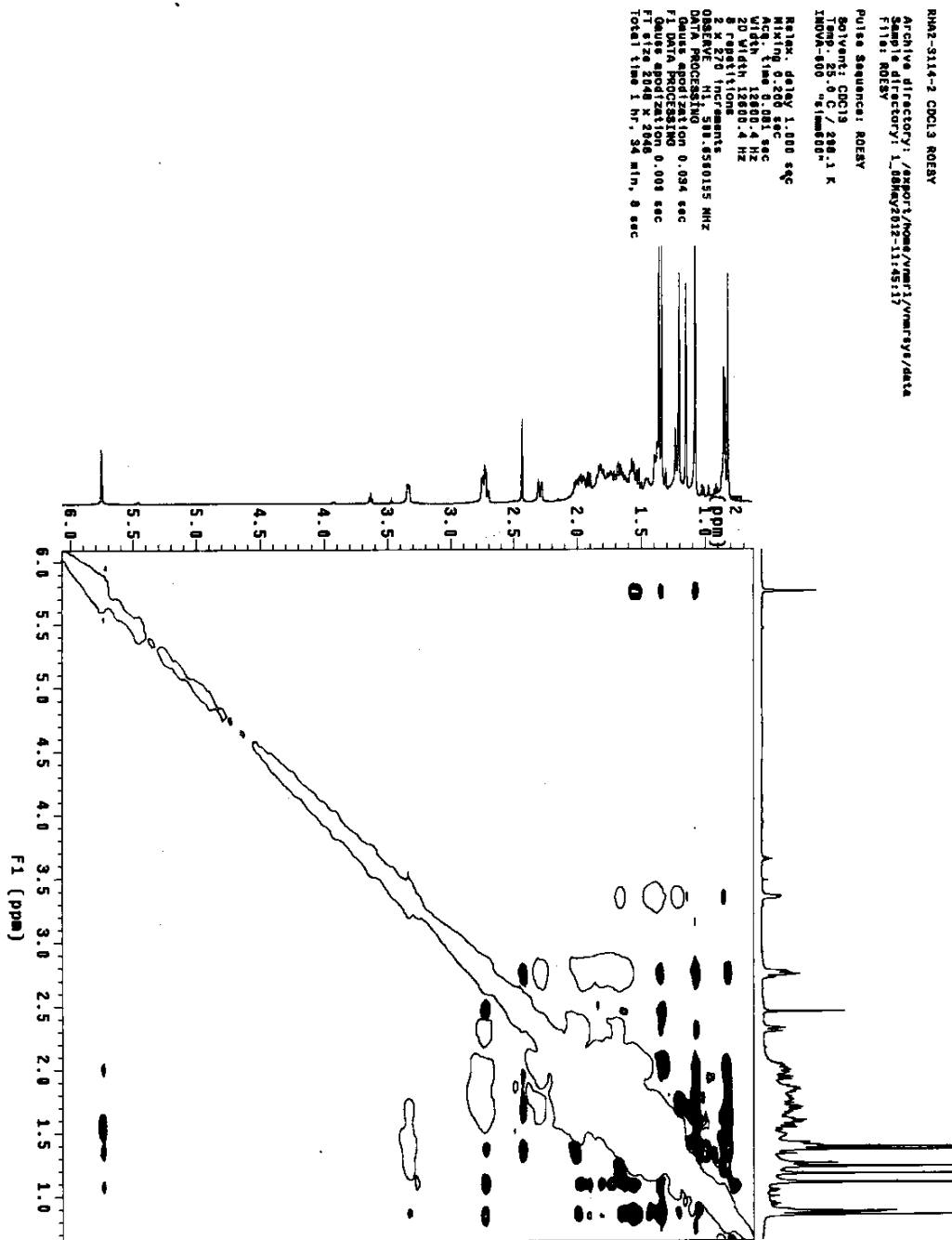


Figure S46. ESI(+)MS spectrum of ricinodol E (5)

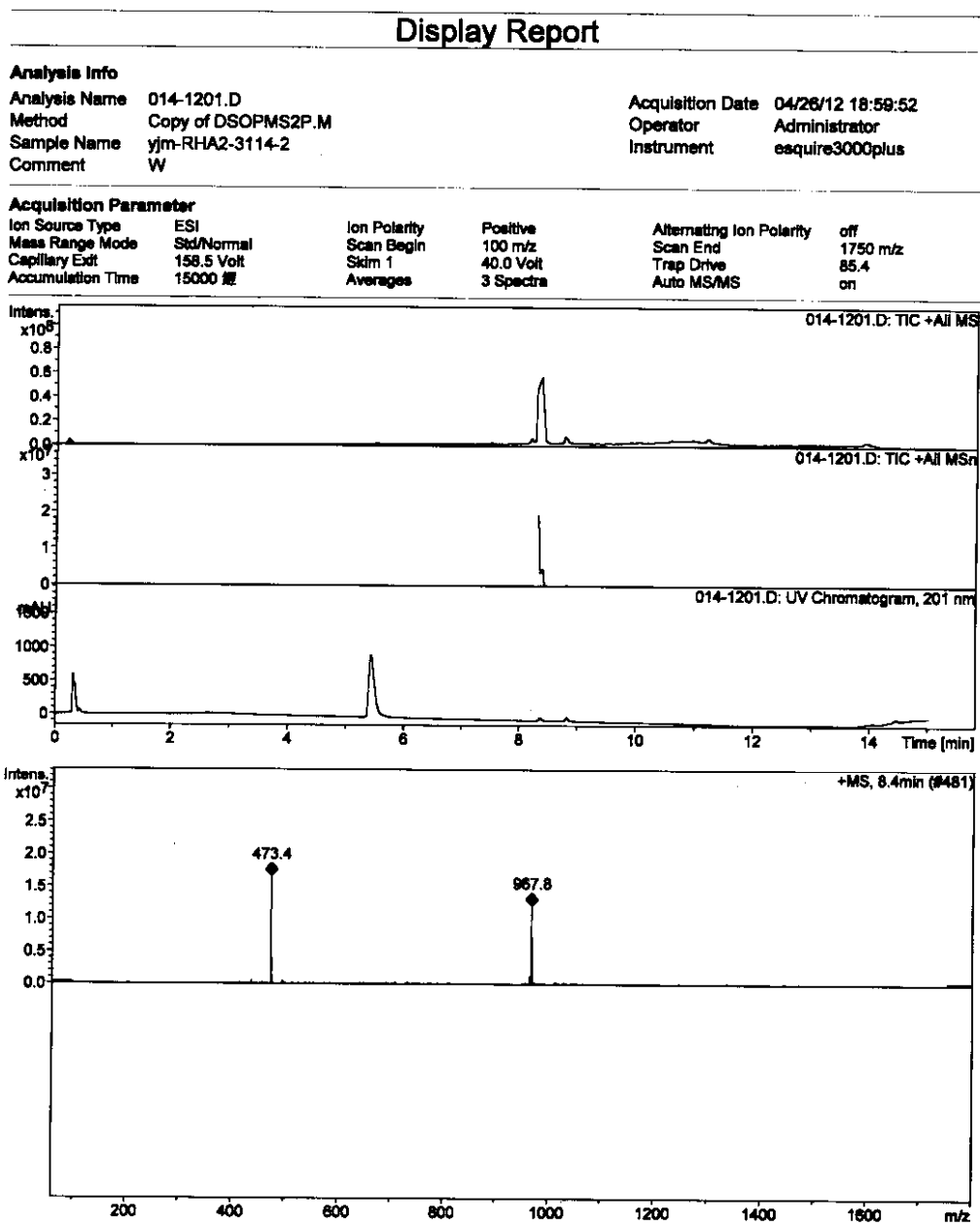


Figure S47. ESI(-)MS spectrum of ricinodol E (5)

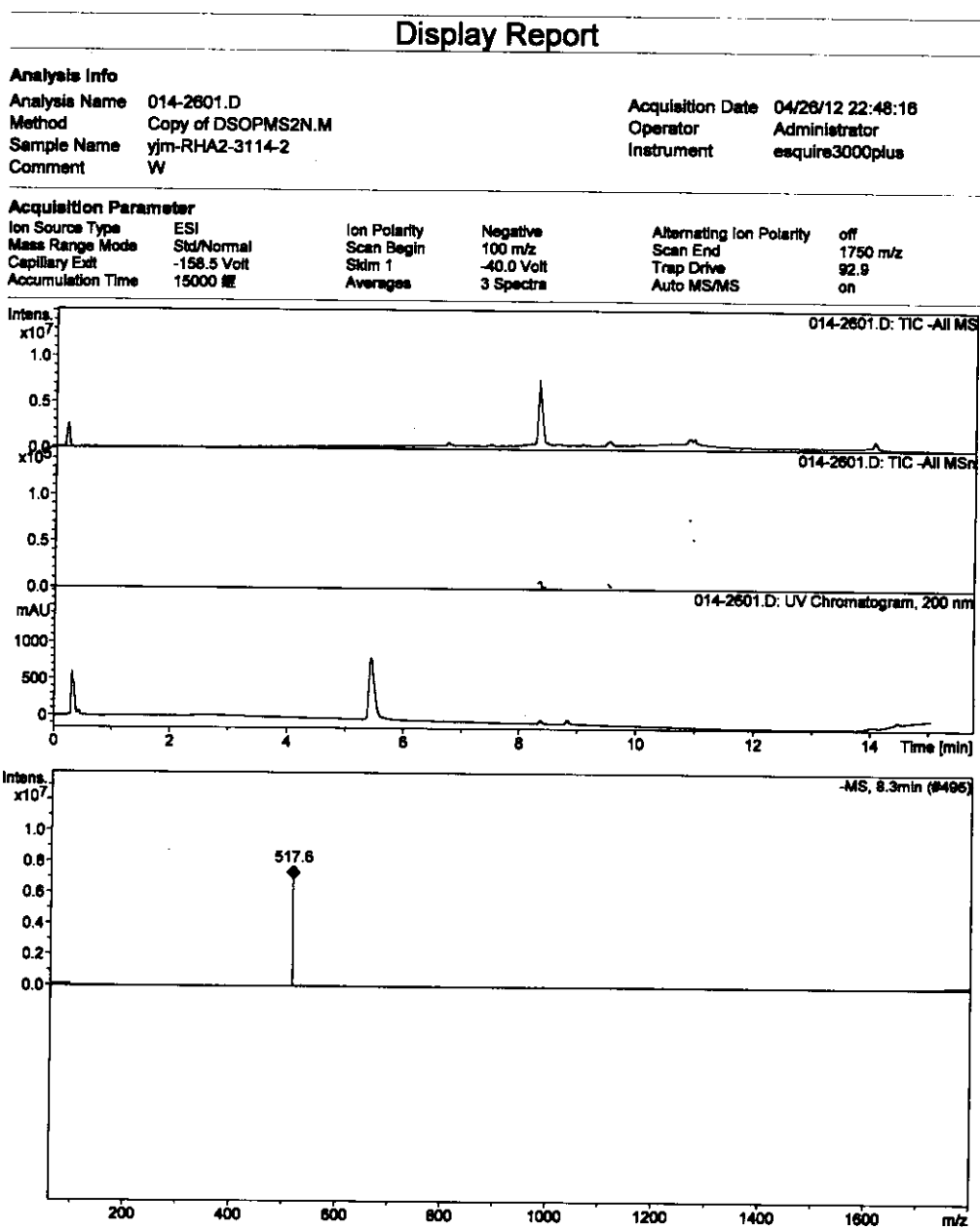


Figure S48. HRESI(-)MS spectrum of ricinodol E (5)

Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 3.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

179 formula(e) evaluated with 1 results within limits (up to 50 closest results for each mass)

Elements Used:

C: 5-80 H: 2-120 O: 0-20 Na: 0-1

RHA3-3114-2

LCT PXE KE324

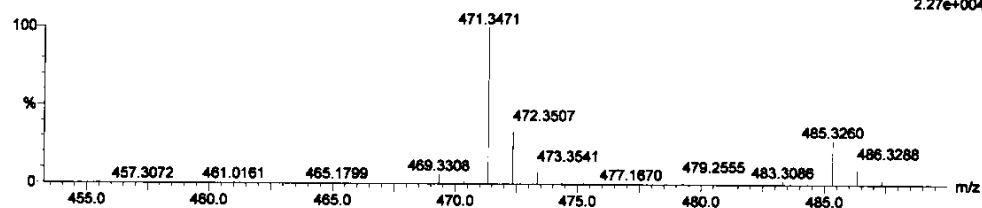
27-Aug-2014

09:41:12

1: TOF MS ES-

2.27e+004

RHA3-3114-2_0827 42 (0.934) AM2 (Ar,10000.0,0.00,1.00); ABS; Cm (42:56)



Minimum:

Maximum: 5.0 3.0 -1.5

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
471.3471	471.3474	-0.3	-0.6	7.5	161.7	0.0	C30 H47 O4

Figure S49. IR spectrum of ricinodol E (5)

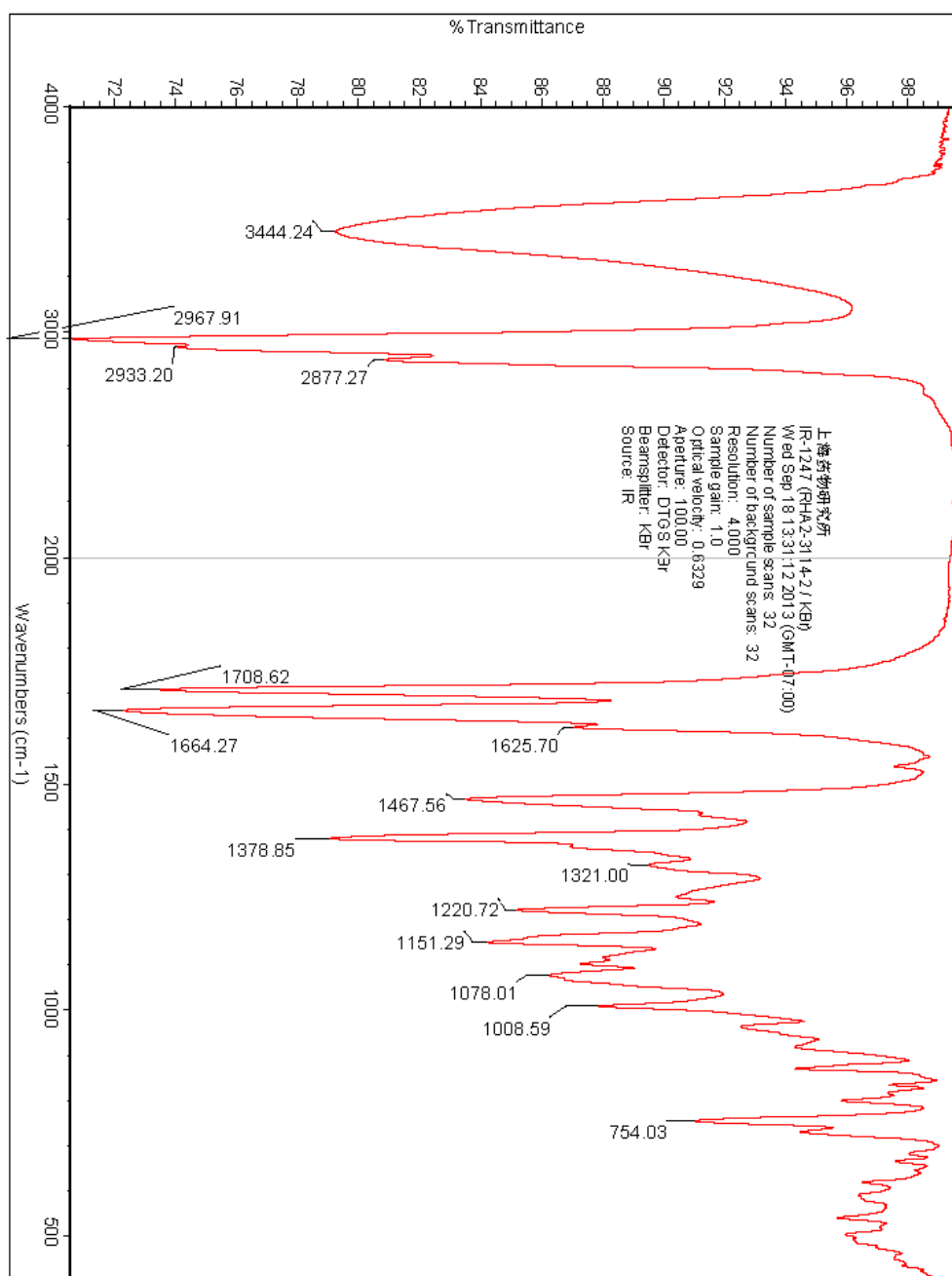


Figure S50. ^1H NMR spectrum of ricinodol F (**6**) in CDCl_3

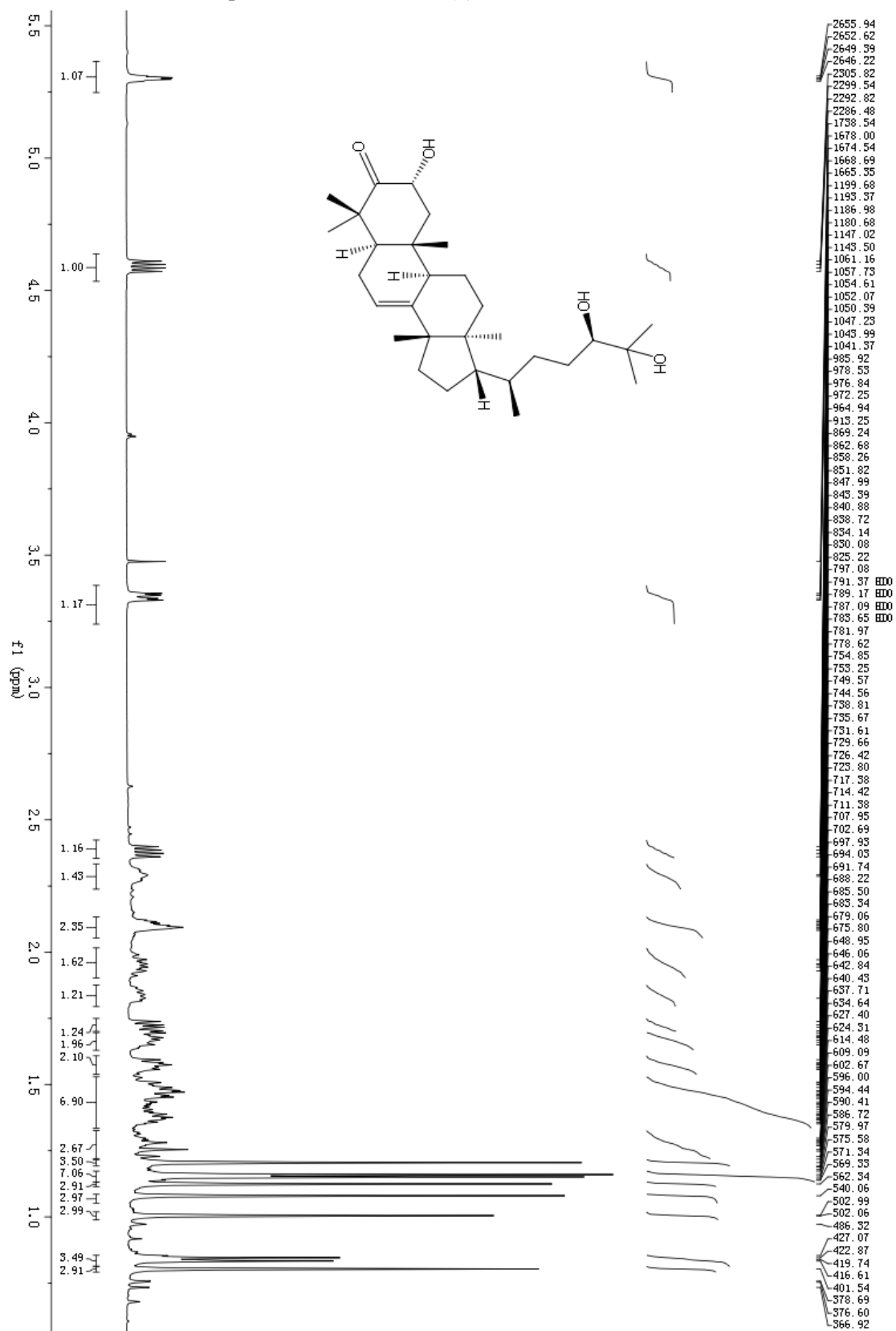


Figure S51. ^{13}C NMR spectrum of ricinodol F (**6**) in CDCl_3

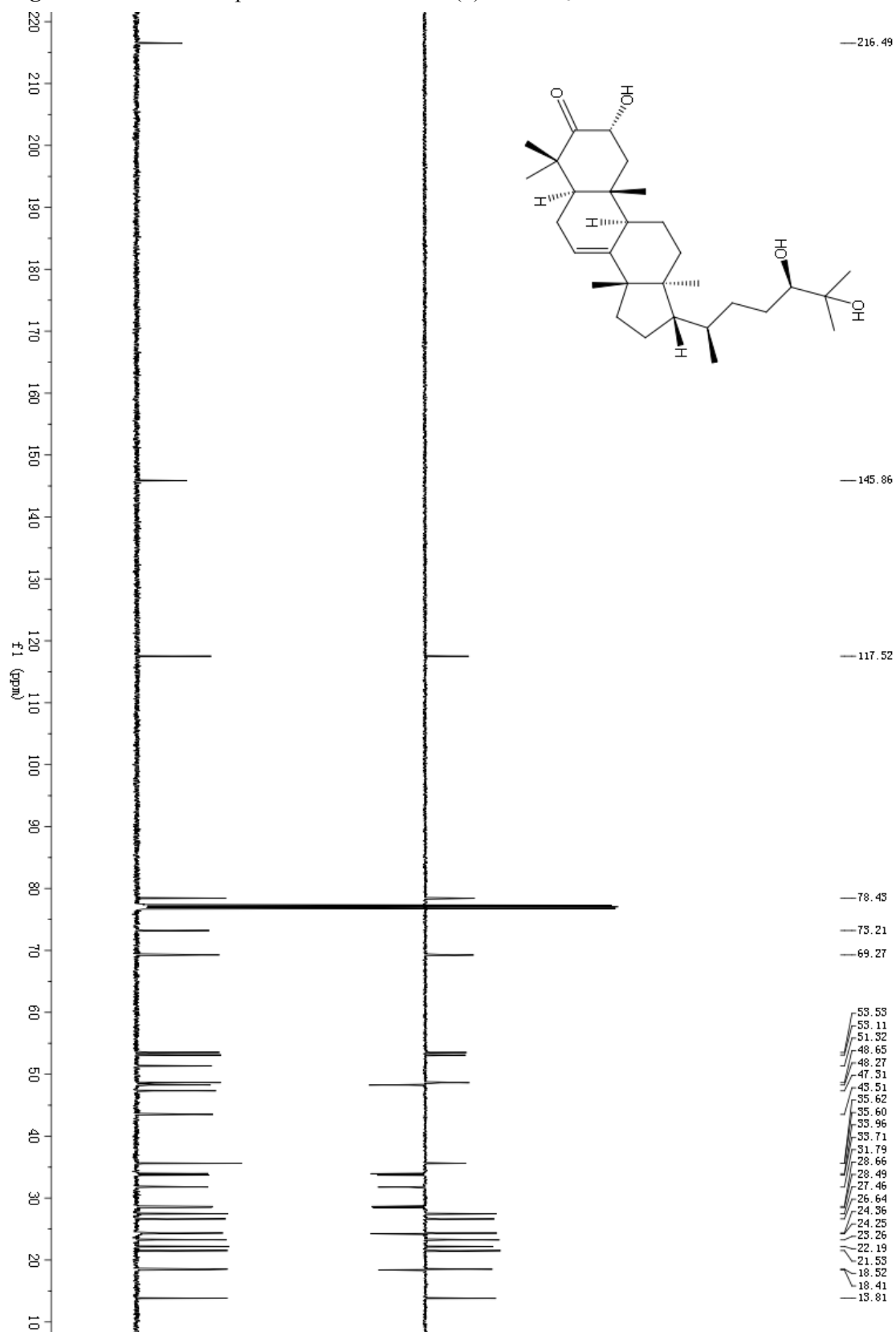


Figure S52. HSQC spectrum of ricinodol F (**6**) in CDCl₃

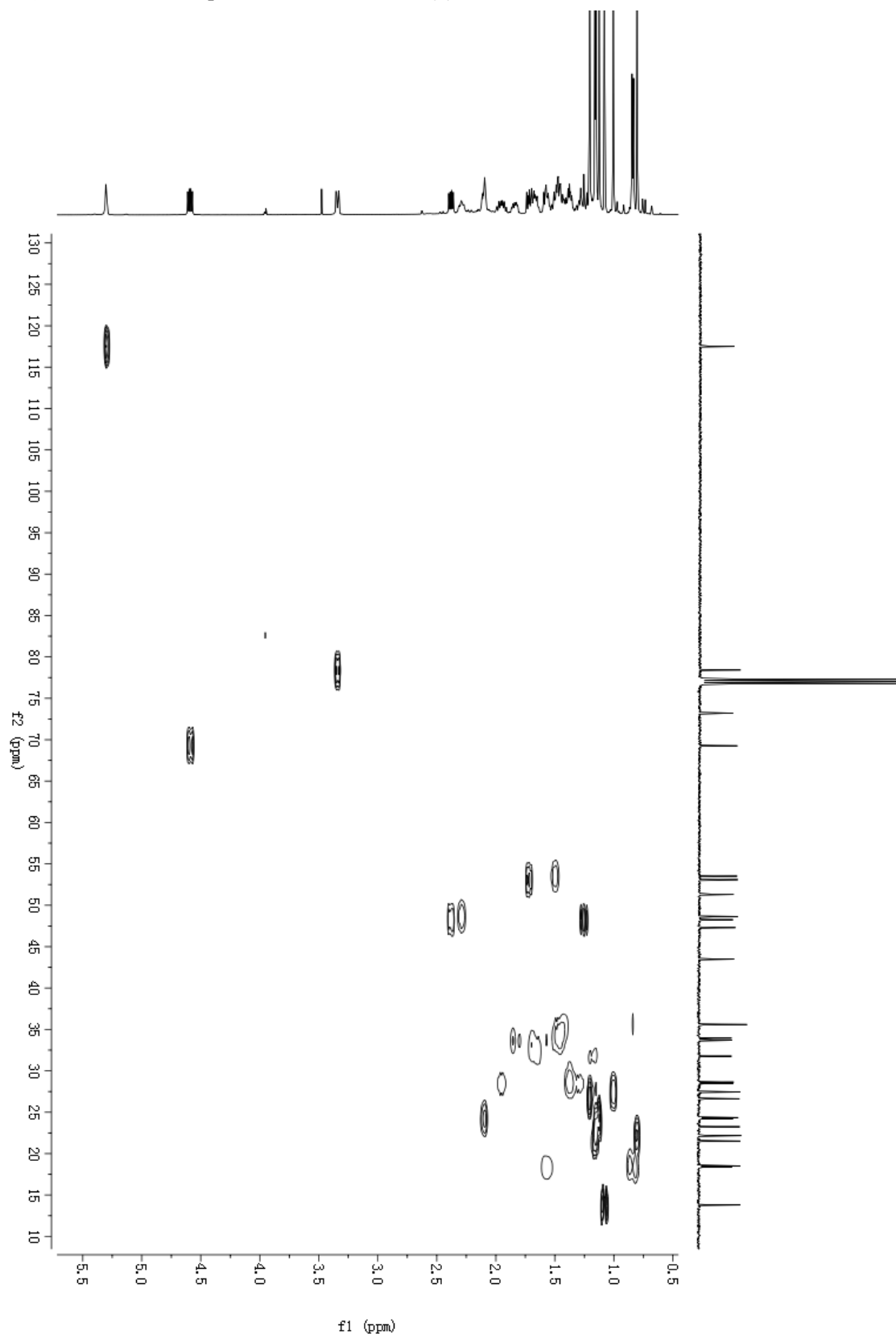


Figure S53. HMBC spectrum of ricinodol F (**6**) in CDCl₃

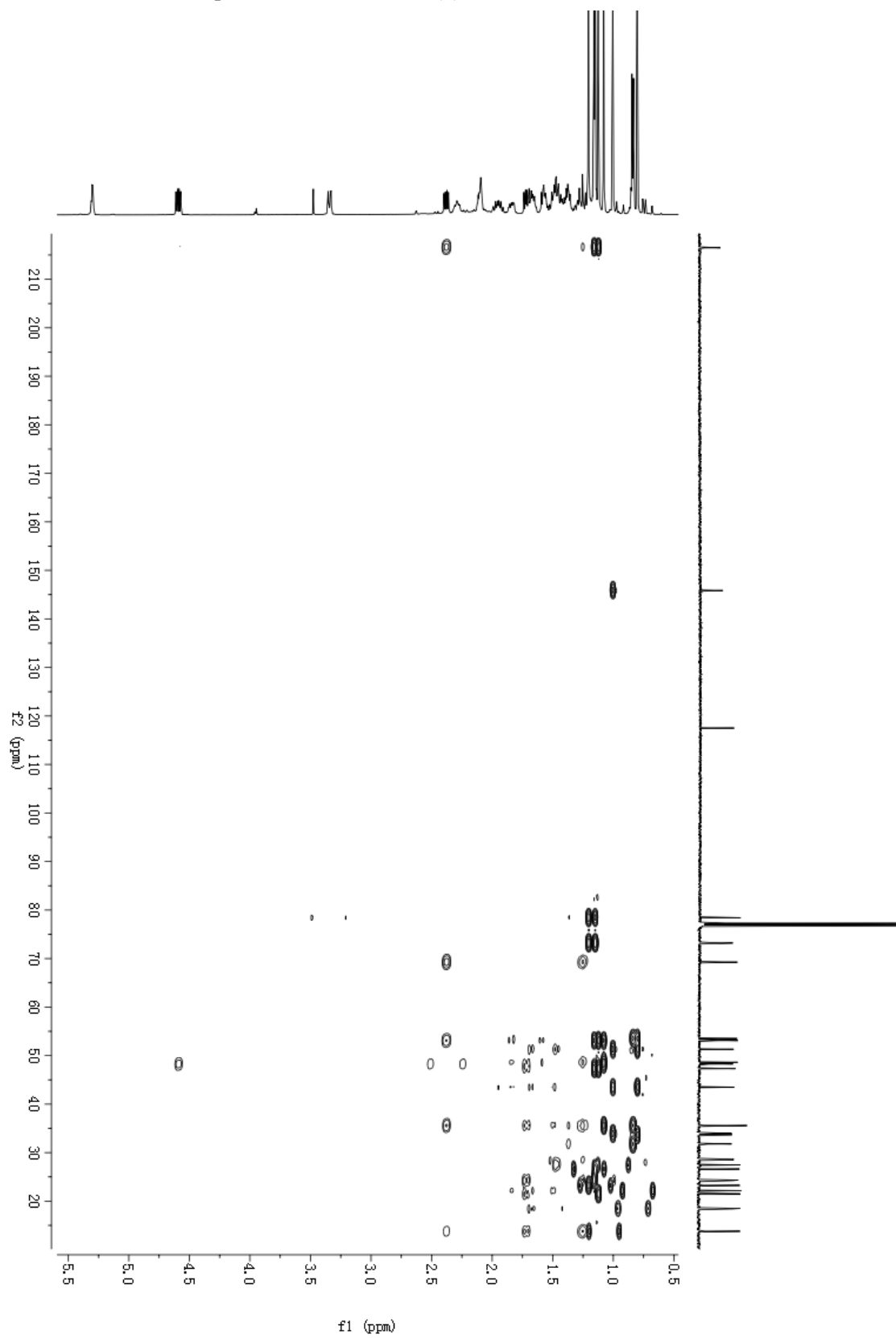


Figure S54. ROESY spectrum of ricinodol F (6) in CDCl₃

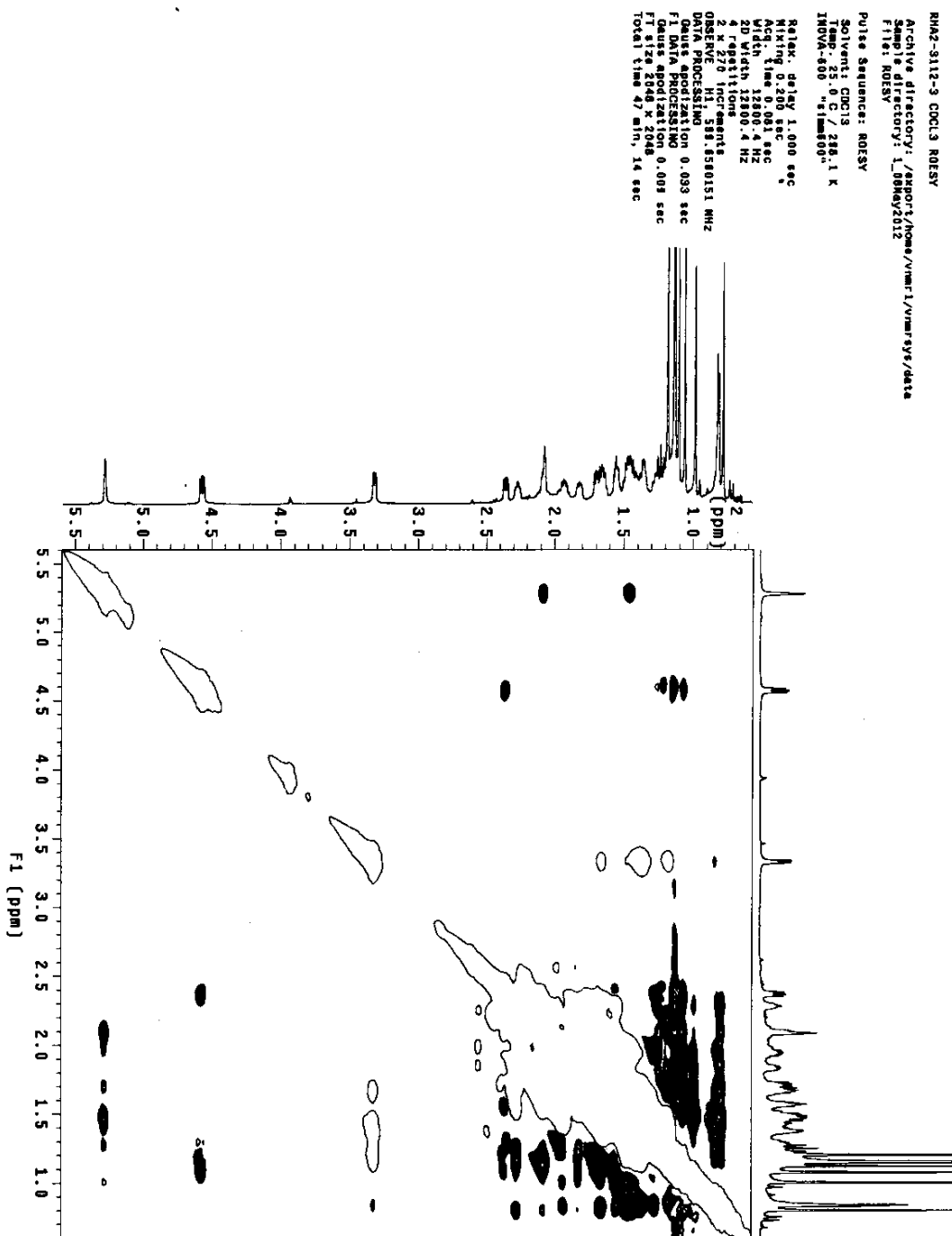


Figure S55. ESI(+)MS spectrum of ricinodol F (6)

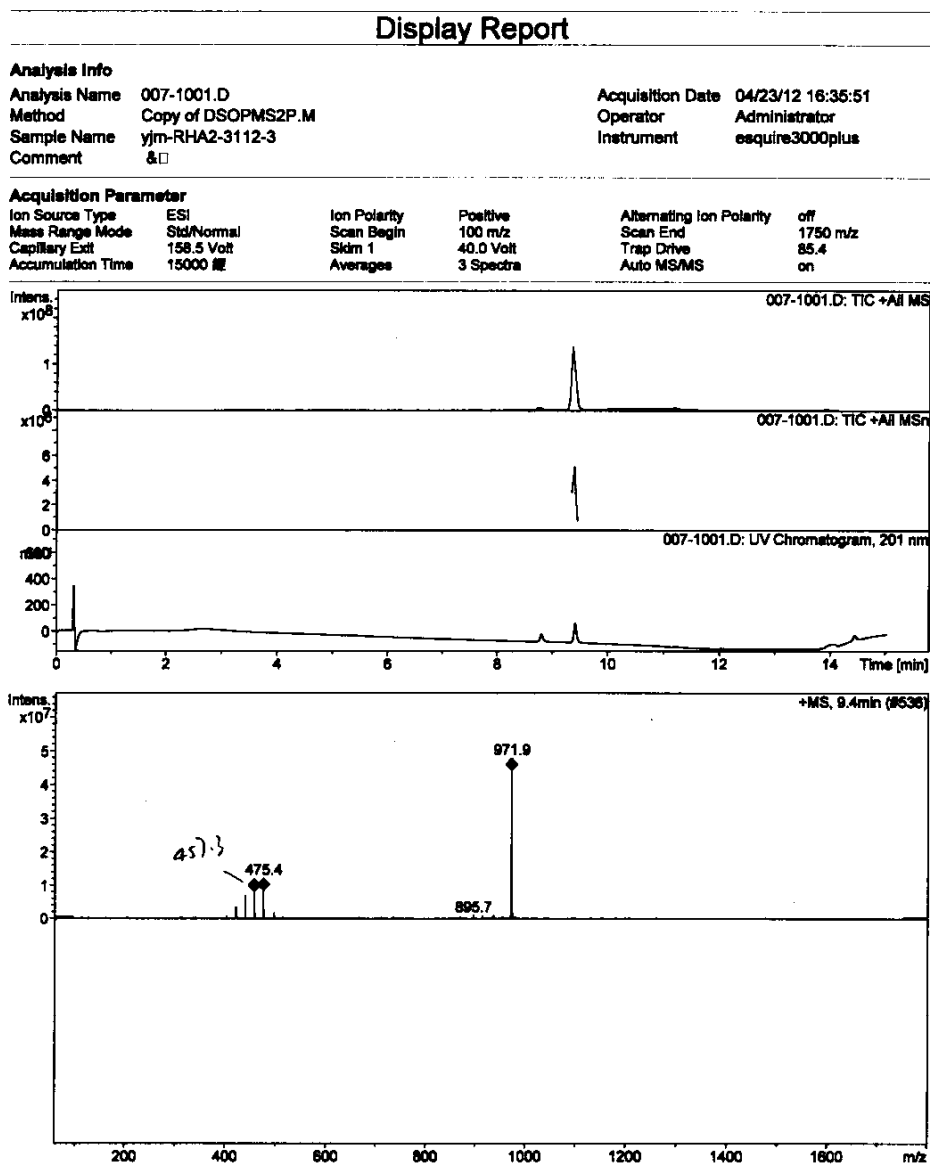


Figure S56. ESI(-)MS spectrum of ricinodol F (6)

Display Report

Analysis Info

Analysis Name 007-2101.D
Method Copy of DSOPMS2N.M
Sample Name yjm-RHA2-3112-3
Comment &□

Acquisition Date 04/23/12 19:35:17
Operator Administrator
Instrument esquire3000plus

Acquisition Parameter

Ion Source Type ESI
Mass Range Mode Std/Normal
Capillary Exit -156.5 Volt
Accumulation Time 15000 程

Ion Polarity Negative
Scan Begin 100 m/z
Skim 1 -40.0 Volt
Averages 3 Spectra

Alternating Ion Polarity off
Scan End 1750 m/z
Trap Drive 92.9
Auto MS/MS on

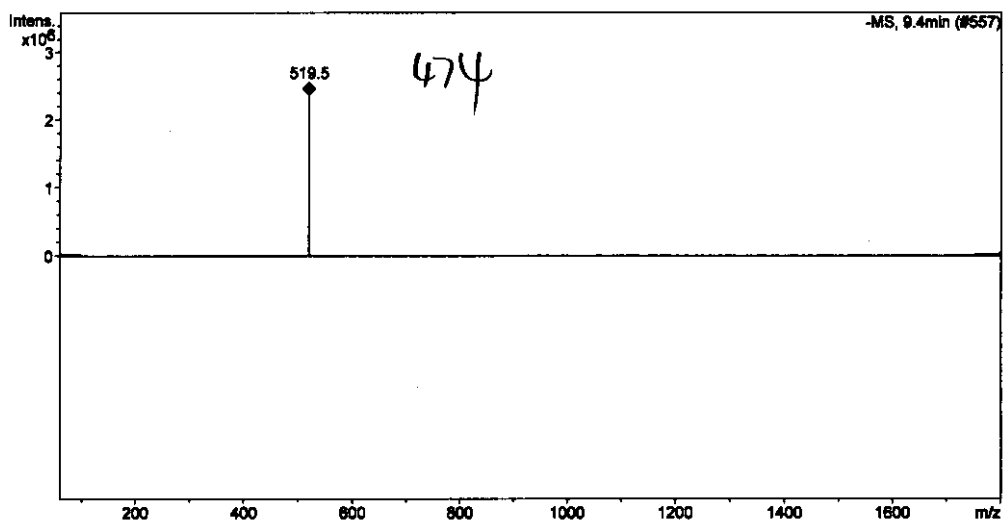
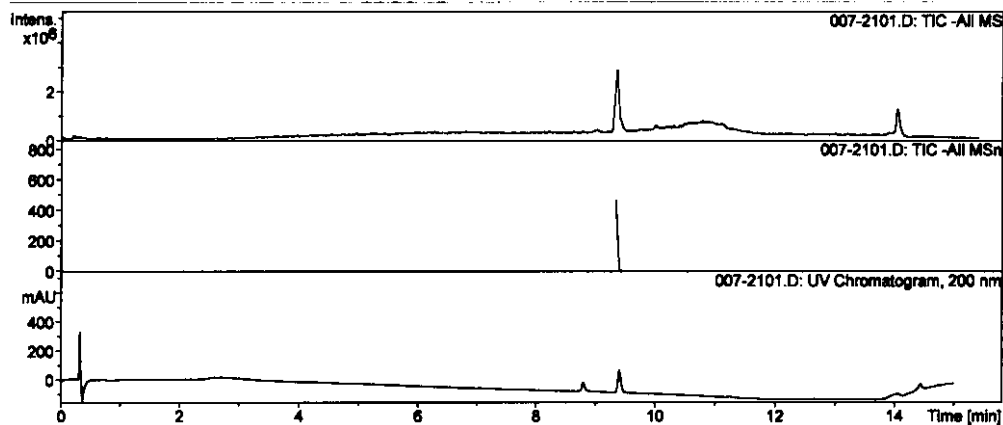


Figure S57. HRESI(-)MS spectrum of ricinodol F (6)

Elemental Composition Report

Single Mass Analysis

Tolerance = 3.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

121 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:

C: 10-80 H: 1-110 O: 0-30

yjh

LCT PXE KE324

04-May-2012

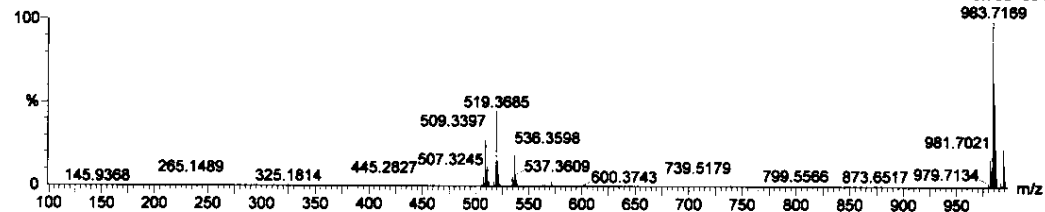
16:19:21

1: TOF MS ES-

3.70e+004

963.7169

RHA2-3112-3 17 (0.335) AM2 (Ar,10000.0,0.00,1.00); ABS; Cm (4:21)



Minimum:

5.0 3.0 -1.5

Maximum:

50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
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519.3685	519.3686	-0.1	-0.2	6.5	114.6	0.0	C31 H51 O6
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Figure S58. IR spectrum of ricinodol F (6)

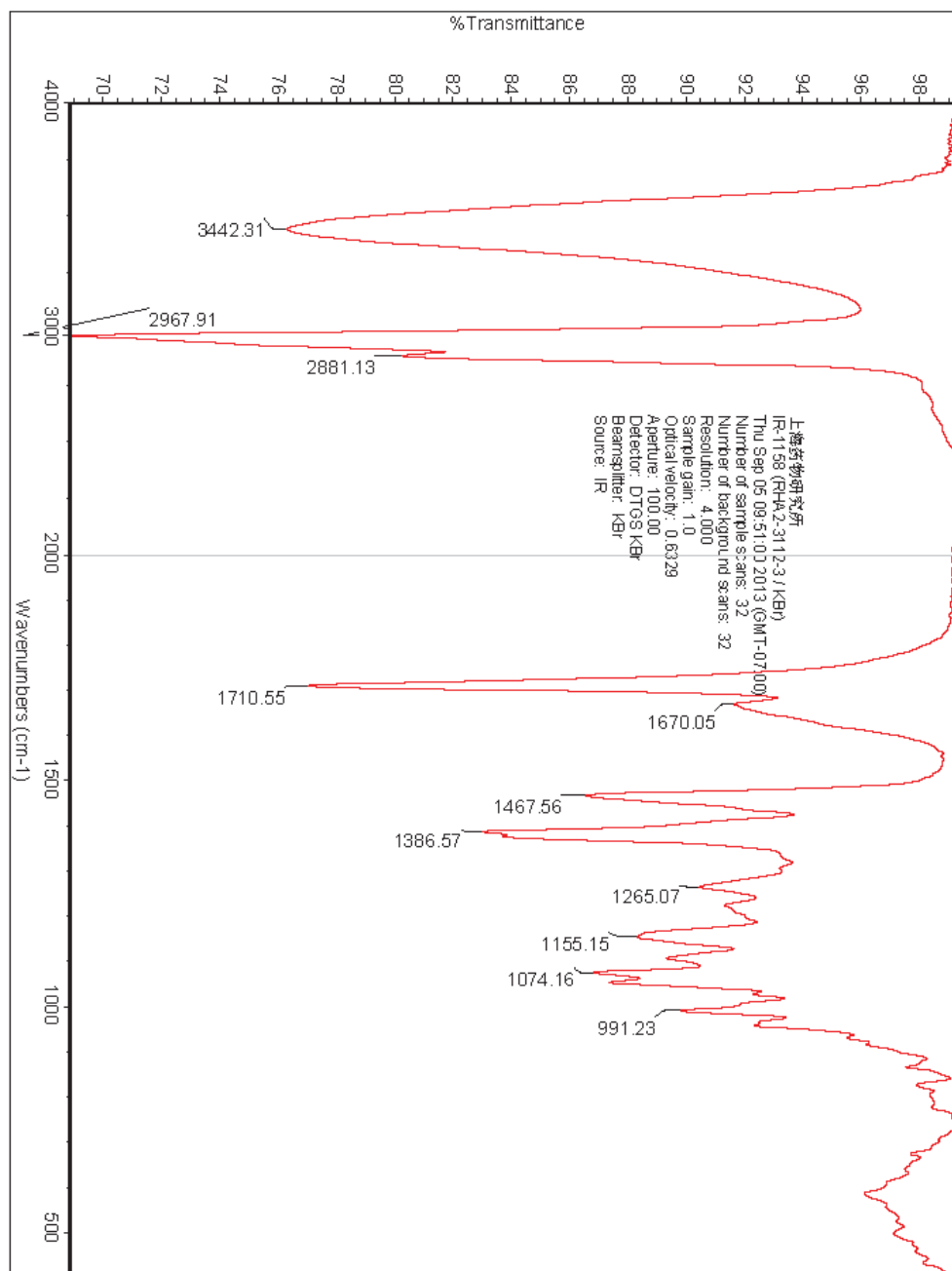


Figure S59. ^1H NMR spectrum of ricinodol G (**7**) in CDCl_3

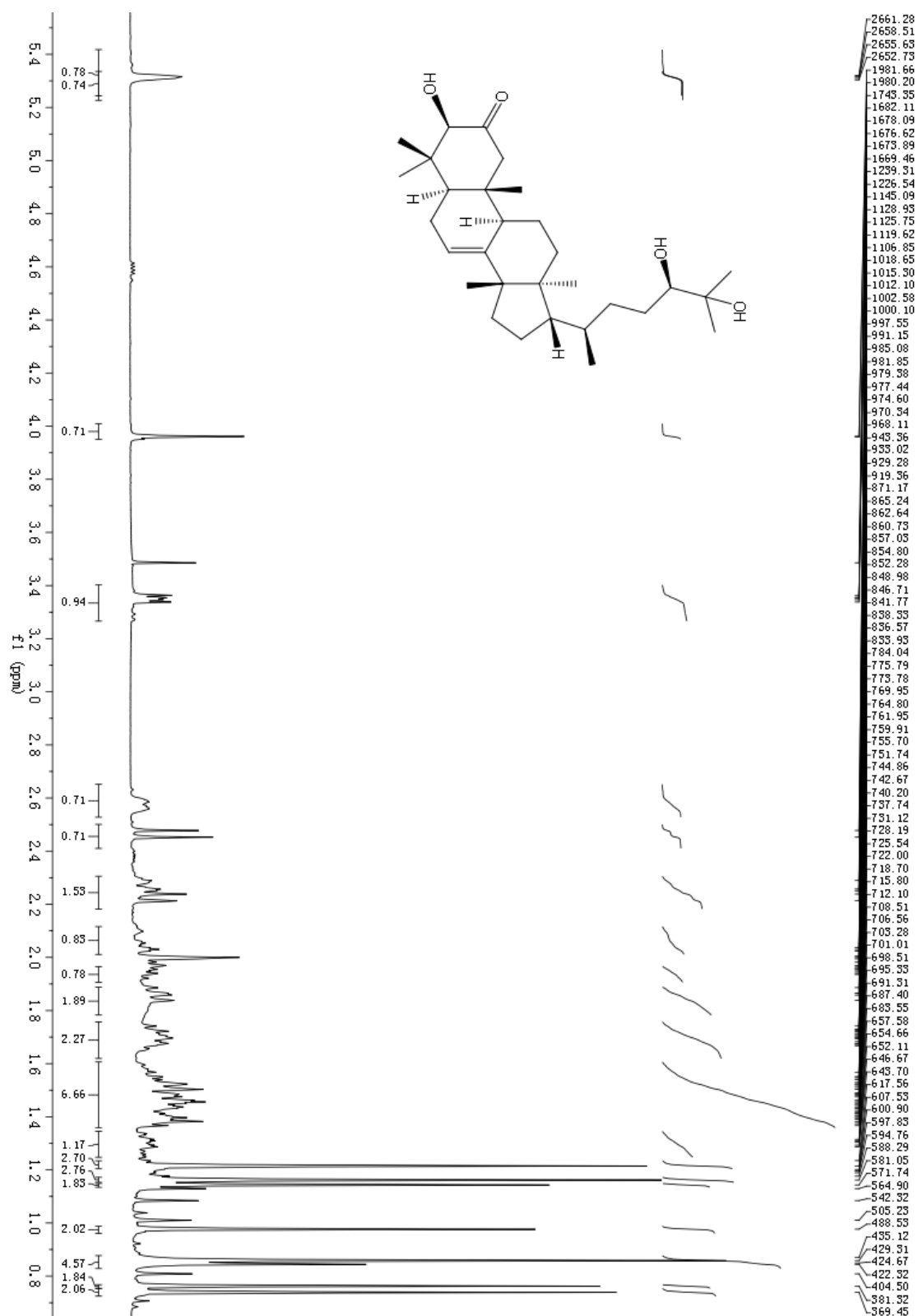


Figure S60. ^{13}C NMR spectrum of ricinodol G (7) in CDCl_3

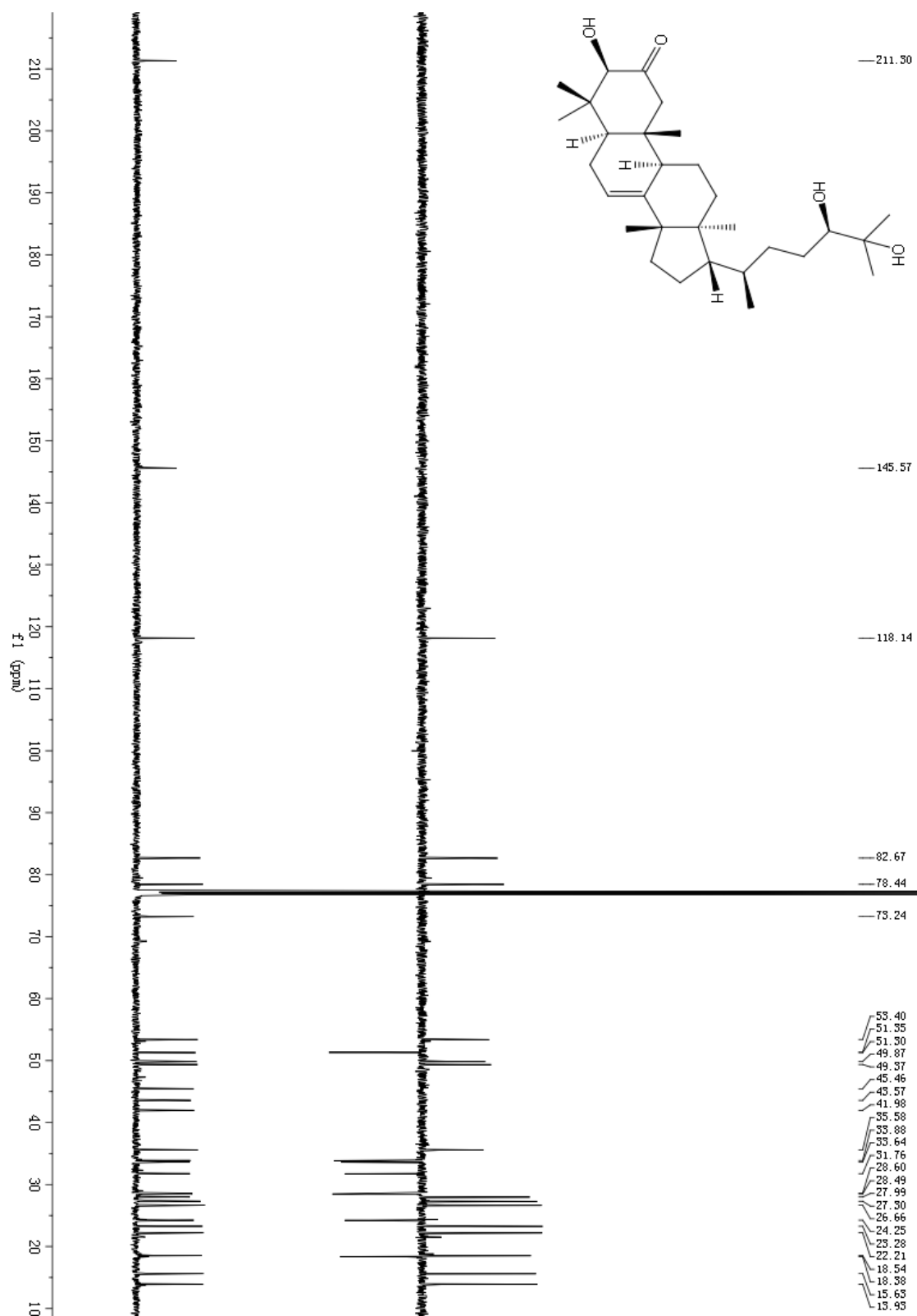


Figure S61. HSQC spectrum of ricinodol G (7) in CDCl₃

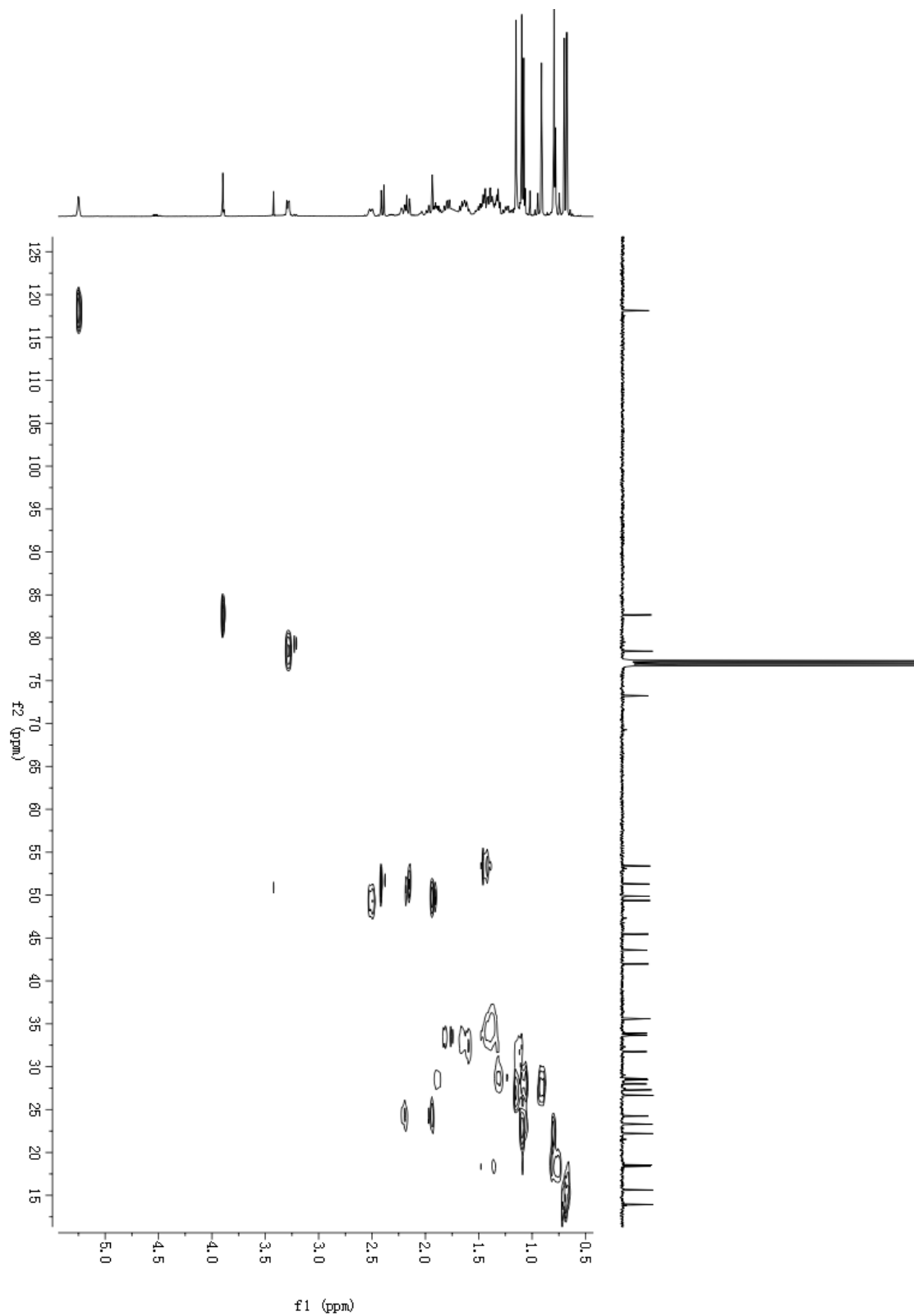


Figure S62. HMBC spectrum of ricinodol G (**7**) in CDCl₃

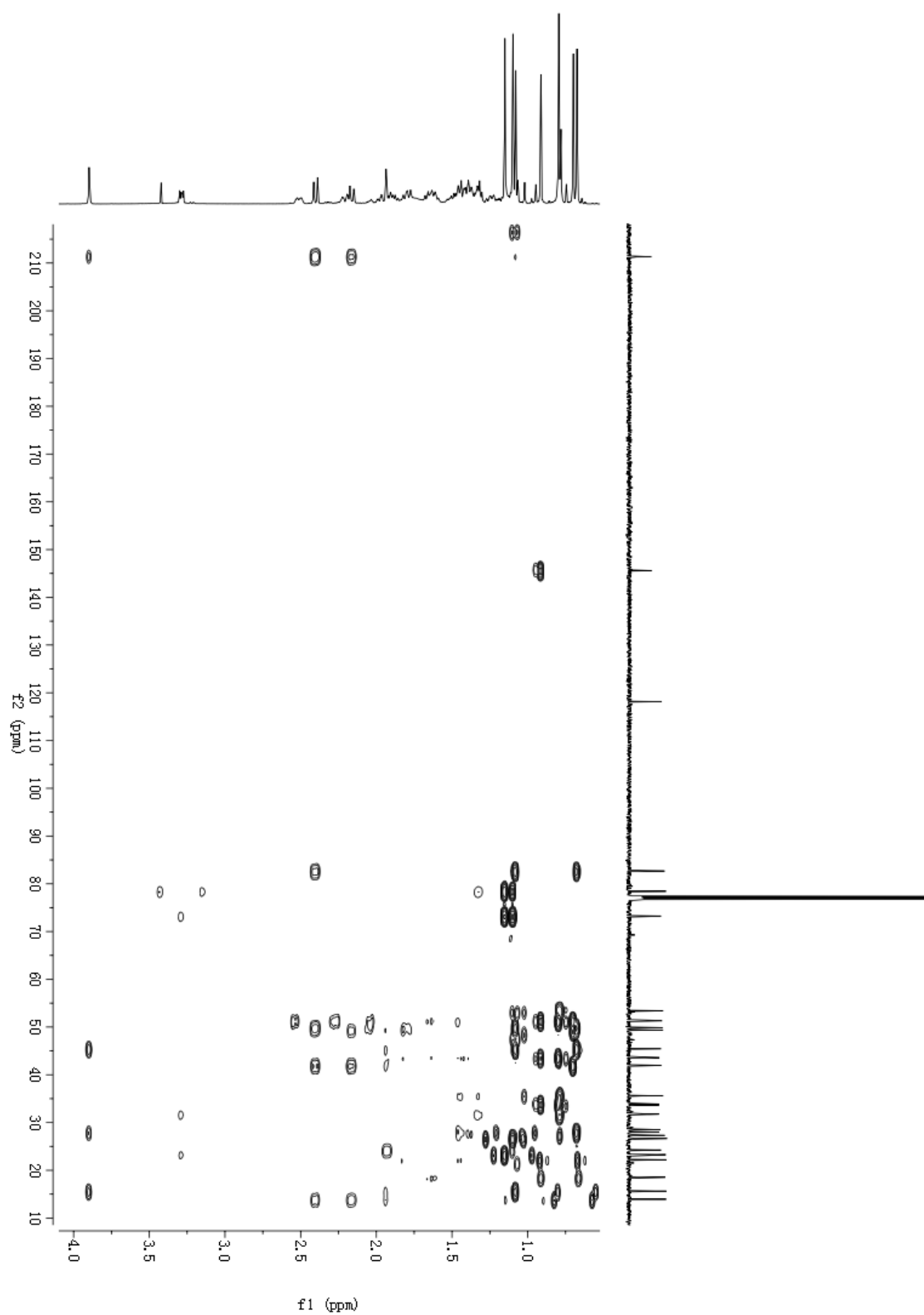


Figure S63. ROESY spectrum of ricinodol G (7) in CDCl₃

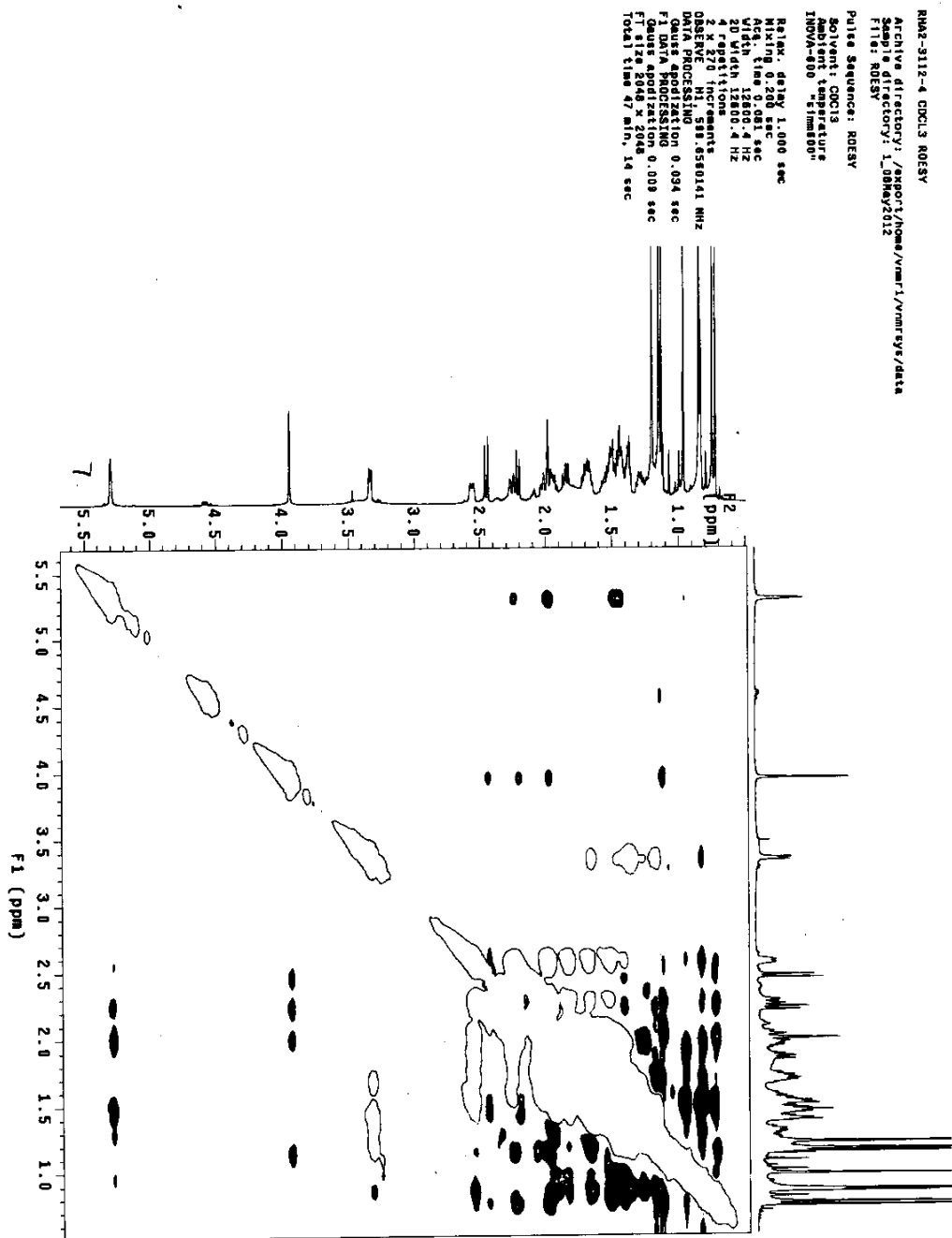


Figure S64. ESI(+)MS spectrum of ricinodol G (7)

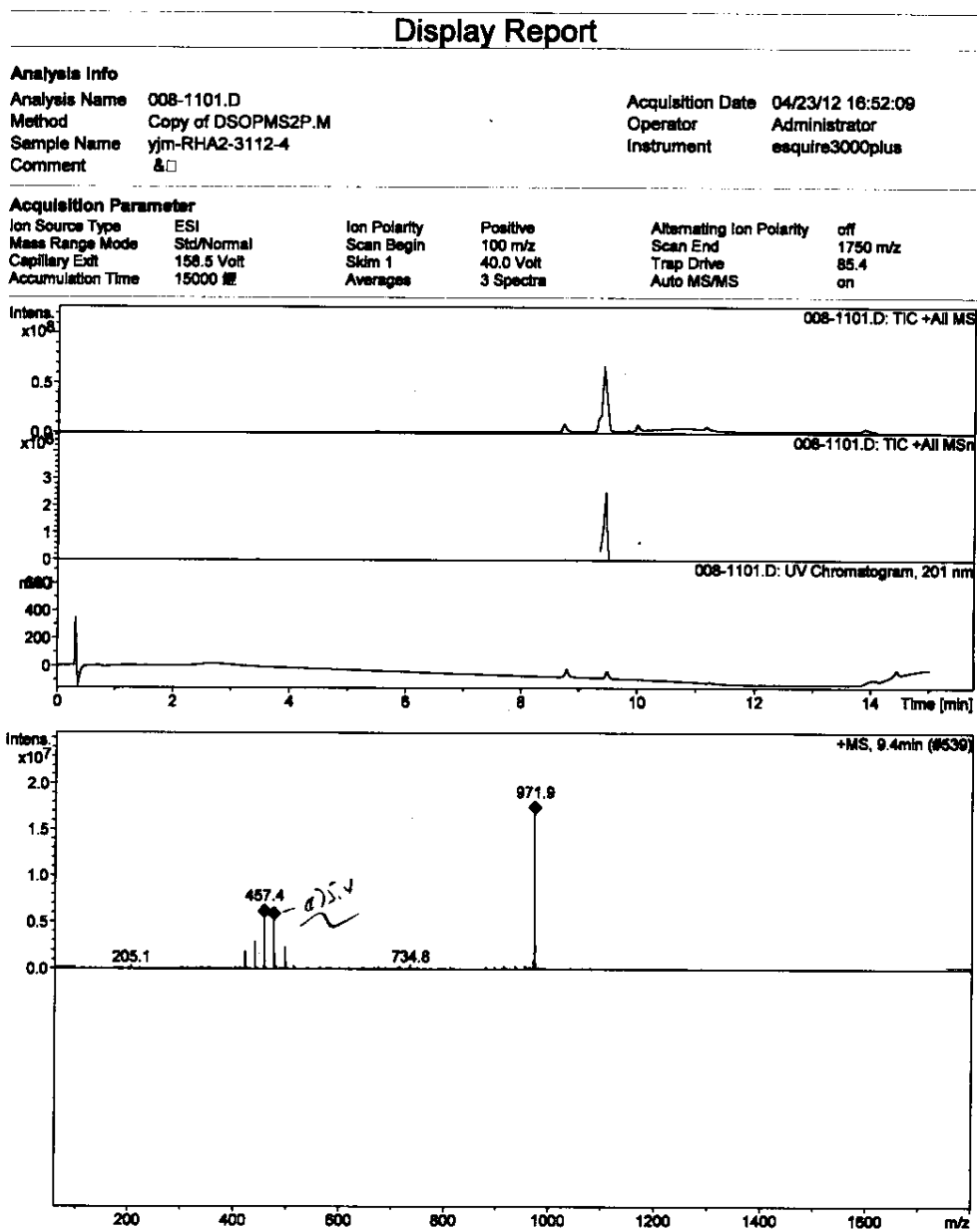


Figure S65. ESI(-)MS spectrum of ricinodol G (7)

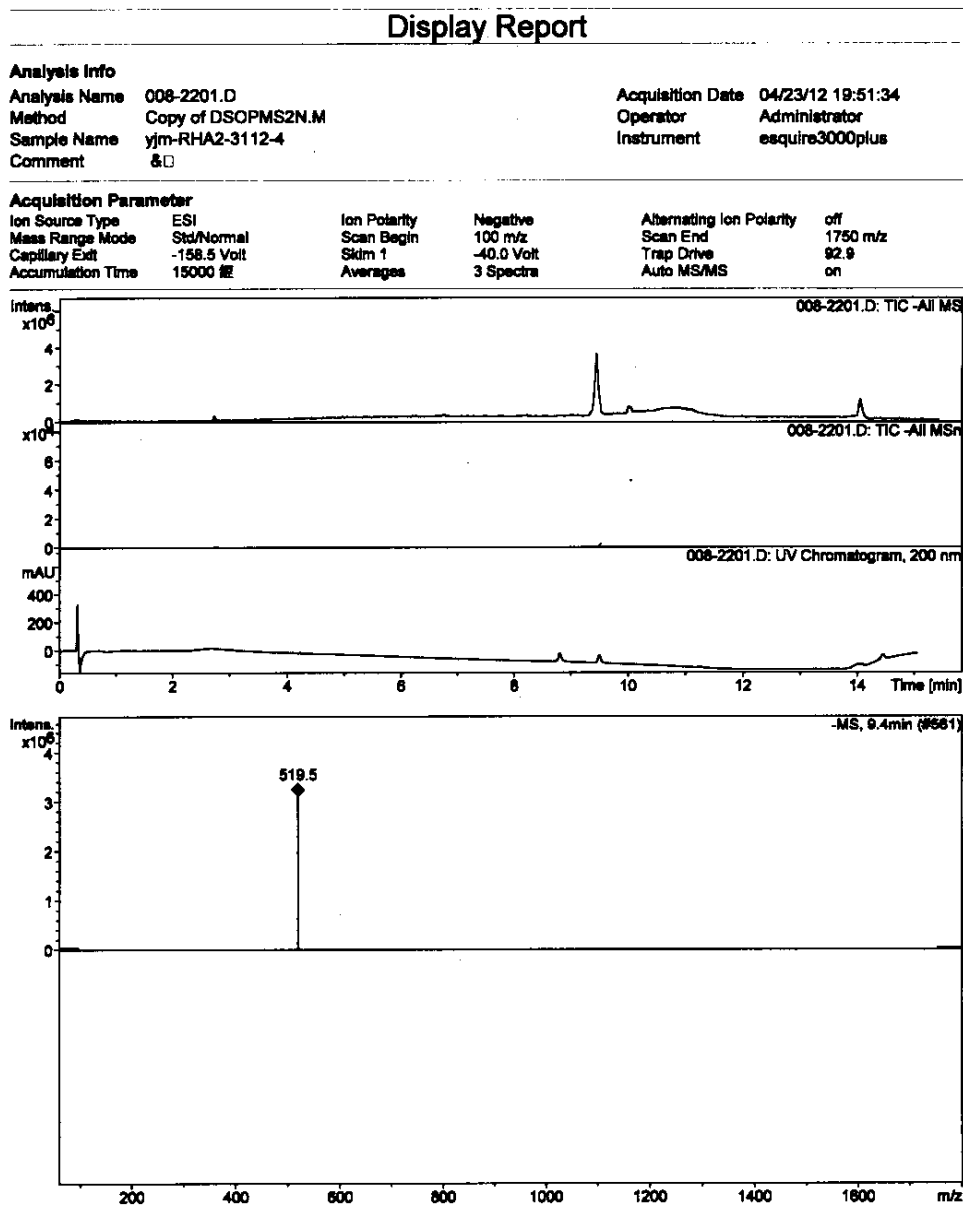


Figure S66. HRESI(-)MS spectrum of ricinodol G (7)

Elemental Composition Report

Single Mass Analysis

Tolerance = 3.0 PPM / DBE: min = -1.5, max = 50.0
 Element prediction: Off
 Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

121 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:

C: 10-80 H: 1-110 O: 0-30

yjh

LCT PXE KE324

04-May-2012

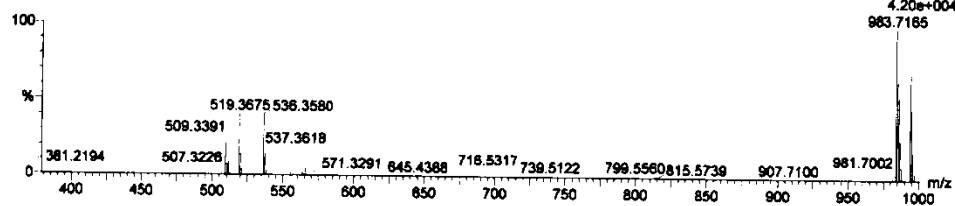
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1: TOF MS ES-

4.20e+004

983.7165

RHA2-3112-4 36 (0.759) AM2 (Ar, 10000.0, 0.00, 1.00); ABS: Cm (23.42)



Minimum: 5.0 3.0 -1.5
 Maximum: 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
519.3675	519.3686	-1.1	-2.1	6.5	106.5	0.0	C31 H51 O6

Figure S67. IR spectrum of ricinodol G (7)

