Supporting Information

Cytotoxic isovaleryl sucrose esters from Ainsliaea yunnanensis: reduction of

MMP and increment of ROS levels in A549 cells

Xin Fang^{a, b}, Zhi-Guo Zhuo^a, Xi-Ke Xu^a, Ji Ye^a, Hui-Liang Li^a, Yun-Heng Shen^{a, *}, and Wei-Dong Zhang^{a, b, c, *}

^a Department of Phytochemistry, Second Military Medical University, Shanghai 200433, P. R. China

^b Shanghai University of Traditional Chinese Medicine, Shanghai 201203, P. R. China

^c Shanghai Institute of Pharmaceutical Industry, Shanghai 201203, P. R. China

*E-mail: shenyunheng@hotmail.com (Y. H. Shen); wdzhangy@hotmail.com (W. D. Zhang), Tel: +86-21-81871244.

Contents

- Figure S1. HRESIMS spectrum of compound 1
- Figure S2. IR spectrum of compound 1
- Figure S3. ¹H NMR spectrum of compound 1 (500 MHz, CDCl₃)
- Figure S4. ¹³C NMR spectrum of compound 1 (125 MHz, CDCl₃)
- Figure S5. DEPT NMR spectrum of compound 1 (125 MHz, CDCl₃)
- Figure S6. ¹H-¹H COSY NMR spectrum of compound 1 (500 MHz, CDCl₃)
- Figure S7. HSQC NMR spectrum of compound 1 (500 MHz, CDCl₃)
- Figure S8. HMBC NMR spectrum of compound 1 (500 MHz, CDCl₃)
- Figure S9. The magnified HMBC NMR spectrum of compound 1 (500 MHz, CDCl₃)
- Figure S10. NOESY NMR spectrum of compound 1 (500 MHz, CDCl₃)
- Figure S11. HRESIMS spectrum of compound 2
- Figure S12. IR spectrum of compound 2
- **Figure S13.** ¹H NMR spectrum of compound **2** (500 MHz, CDCl₃)
- Figure S14. ¹³C NMR spectrum of compound 2 (125 MHz, CDCl₃)
- Figure S15. DEPT NMR spectrum of compound 2 (125 MHz, CDCl₃)
- Figure S16. ¹H-¹H COSY NMR spectrum of compound 2 (500 MHz, CDCl₃)
- Figure S17. HSQC NMR spectrum of compound 2 (500 MHz, CDCl₃)
- Figure S18. HMBC NMR spectrum of compound 2 (500 MHz, CDCl₃)
- Figure S19. The magnified HMBC NMR spectrum of compound 2 (500 MHz, CDCl₃)
- Figure S20. NOESY NMR spectrum of compound 2 (500 MHz, CDCl₃)
- Figure S21. HRESIMS spectrum of compound 3
- Figure S22. IR spectrum of compound 3
- **Figure S23.** ¹H NMR spectrum of compound **3** (500 MHz, CDCl₃)
- Figure S24. ¹³C NMR spectrum of compound 3 (125 MHz, CDCl₃)
- Figure S25. DEPT NMR spectrum of compound 3 (125 MHz, CDCl₃)
- Figure S26. ¹H-¹H COSY NMR spectrum of compound 3 (500 MHz, CDCl₃)
- Figure S27. HSQC NMR spectrum of compound 3 (500 MHz, CDCl₃)
- Figure S28. HMBC NMR spectrum of compound 3 (500 MHz, CDCl₃)
- Figure S29. The magnified HMBC NMR spectrum of compound 3 (500 MHz, CDCl₃)
- Figure S30. NOESY NMR spectrum of compound 3 (500 MHz, CDCl₃)

Figure S31. HRESIMS spectrum of compound 4

Figure S32. IR spectrum of compound 4

Figure S33. ¹H NMR spectrum of compound 4 (500 MHz, CDCl₃)

Figure S34. ¹³C NMR spectrum of compound 4 (125 MHz, CDCl₃)

Figure S35. DEPT NMR spectrum of compound 4 (125 MHz, CDCl₃)

Figure S36. ¹H-¹H COSY NMR spectrum of compound 4 (500 MHz, CDCl₃)

Figure S37. HSQC NMR spectrum of compound 4 (500 MHz, CDCl₃)

Figure S38. HMBC NMR spectrum of compound 4 (500 MHz, CDCl₃)

Figure S39. The magnified HMBC NMR spectrum of compound 4 (500 MHz, CDCl₃)

Figure S40. NOESY NMR spectrum of compound 4 (500 MHz, CDCl₃)

Figure S41. HRESIMS spectrum of compound 5

Figure S42. IR spectrum of compound 5

Figure S43. ¹H NMR spectrum of compound **5** (500 MHz, CDCl₃)

Figure S44. ¹³C NMR spectrum of compound 5 (125 MHz, CDCl₃)

Figure S45. DEPT NMR spectrum of compound 5 (125 MHz, CDCl₃)

Figure S46. ¹H-¹H COSY NMR spectrum of compound 5 (500 MHz, CDCl₃)

Figure S47. HSQC NMR spectrum of compound 5 (500 MHz, CDCl₃)

Figure S48. HMBC NMR spectrum of compound 5 (500 MHz, CDCl₃)

Figure S49. The magnified HMBC NMR spectrum of compound 5 (500 MHz, CDCl₃)

Figure S50. NOESY NMR spectrum of compound 5 (500 MHz, CDCl₃)

Figure S51. HRESIMS spectrum of compound 6

Figure S52. IR spectrum of compound 6

Figure S53. ¹H NMR spectrum of compound 6 (500 MHz, CDCl₃)

Figure S54. ¹³C NMR spectrum of compound 6 (125 MHz, CDCl₃)

Figure S55. DEPT NMR spectrum of compound 6 (125 MHz, CDCl₃)

Figure S56. ¹H-¹H COSY NMR spectrum of compound 6 (500 MHz, CDCl₃)

Figure S57. HSQC NMR spectrum of compound 6 (500 MHz, CDCl₃)

Figure S58. HMBC NMR spectrum of compound 6 (500 MHz, CDCl₃)

Figure S59. The magnified HMBC NMR spectrum of compound 6 (500 MHz, CDCl₃)

Figure S60. NOESY NMR spectrum of compound 6 (500 MHz, CDCl₃)

Figure S61. HRESIMS spectrum of compound 7

Figure S62. IR spectrum of compound 7

Figure S63. ¹H NMR spectrum of compound **7** (500 MHz, CDCl₃)

Figure S64. ¹³C NMR spectrum of compound 7 (125 MHz, CDCl₃)

Figure S65. DEPT NMR spectrum of compound 7 (125 MHz, CDCl₃)

Figure S66. ¹H-¹H COSY NMR spectrum of compound 7 (500 MHz, CDCl₃)

Figure S67. HSQC NMR spectrum of compound 7 (500 MHz, CDCl₃)

Figure S68. HMBC NMR spectrum of compound 7 (500 MHz, CDCl₃)

Figure S69. The magnified HMBC NMR spectrum of compound 7 (500 MHz, CDCl₃)

Figure S70. NOESY NMR spectrum of compound 7 (500 MHz, CDCl₃)

Figure S71. HRESIMS spectrum of compound 8

Figure S72. IR spectrum of compound 8

Figure S73. ¹H NMR spectrum of compound **8** (500 MHz, CDCl₃)

Figure S74. ¹³C NMR spectrum of compound 8 (125 MHz, CDCl₃)

Figure S75. DEPT NMR spectrum of compound 8 (125 MHz, CDCl₃)

Figure S76. ¹H-¹H COSY NMR spectrum of compound 8 (500 MHz, CDCl₃)

Figure S77. HSQC NMR spectrum of compound 8 (500 MHz, CDCl₃)

Figure S78. HMBC NMR spectrum of compound 8 (500 MHz, CDCl₃)

Figure S79. The magnified HMBC NMR spectrum of compound 8 (500 MHz, CDCl₃)

Figure S80. NOESY NMR spectrum of compound 8 (500 MHz, CDCl₃)

Figure S81. ¹H NMR spectrum of Isovaleric acid (500 MHz, CDCl₃)

Figure S82. ¹³C NMR spectrum of Isovaleric acid (125 MHz, CDCl₃)

Figure S83. DEPT NMR spectrum of Isovaleric acid (125 MHz, CDCl₃)

Figure S84. Viability of A549 cells after exposure to 0.1% DMSO or various concentrations of compound 2 for 24, 48, and 72 h.

Figure S1. HRESIMS spectrum of compound 1



Figure S2. IR spectrum of compound 1



Figure S3. ¹H NMR spectrum of compound 1 (500 MHz, CDCl₃)



Figure S4. ¹³C NMR spectrum of compound 1 (125 MHz, CDCl₃)



Figure S5. DEPT NMR spectrum of compound 1 (125 MHz, CDCl₃)



Figure S6. ¹H-¹H COSY NMR spectrum of compound 1 (500 MHz, CDCl₃)



Figure S7. HSQC NMR spectrum of compound 1 (500 MHz, CDCl₃)



Figure S8. HMBC NMR spectrum of compound 1 (500 MHz, CDCl₃)



Figure S9. The magnified HMBC NMR spectrum of compound 1 (500 MHz, CDCl₃)



Figure S10. NOESY NMR spectrum of compound 1 (500 MHz, CDCl₃)



Figure S11. HRESIMS spectrum of compound 2



Figure S12. IR spectrum of compound 2



Figure S13. ¹H NMR spectrum of compound 2 (500 MHz, CDCl₃)



Figure S14. ¹³C NMR spectrum of compound 2 (125 MHz, CDCl₃)



Figure S15. DEPT NMR spectrum of compound 2 (125 MHz, CDCl₃)



Figure S16. ¹H-¹H COSY NMR spectrum of compound 2 (500 MHz, CDCl₃)



Figure S17. HSQC NMR spectrum of compound 2 (500 MHz, CDCl₃)

AFC-463/5 - AFC-463 HSQC



Figure S18. HMBC NMR spectrum of compound 2 (500 MHz, CDCl₃)



Figure S19. The magnified HMBC NMR spectrum of compound 2 (500 MHz, CDCl₃)



Figure S20. NOESY NMR spectrum of compound 2 (500 MHz, CDCl₃)



Figure S21. HRESIMS spectrum of compound 3



Figure S22. IR spectrum of compound 3



Figure S23. ¹H NMR spectrum of compound 3 (500 MHz, CDCl₃)



Figure S24. ¹³C NMR spectrum of compound 3 (125 MHz, CDCl₃)



Figure S25. DEPT NMR spectrum of compound 3 (125 MHz, CDCl₃)



Figure S26. ¹H-¹H COSY NMR spectrum of compound 3 (500 MHz, CDCl₃)



Figure S27. HSQC NMR spectrum of compound 3 (500 MHz, CDCl₃)



Figure S28. HMBC NMR spectrum of compound 3 (500 MHz, CDCl₃)



Figure S29. The magnified HMBC NMR spectrum of compound 3 (500 MHz, CDCl₃)



Figure S30. NOESY NMR spectrum of compound 3 (500 MHz, CDCl₃)



Figure S31. HRESIMS spectrum of compound 4



Figure S32. IR spectrum of compound 4



Figure S33. ¹H NMR spectrum of compound 4 (500 MHz, CDCl₃)



Figure S34. ¹³C NMR spectrum of compound 4 (125 MHz, CDCl₃)



Figure S35. DEPT NMR spectrum of compound 4 (125 MHz, CDCl₃)



Figure S36. ¹H-¹H COSY NMR spectrum of compound 4 (500 MHz, CDCl₃)



Figure S37. HSQC NMR spectrum of compound 4 (500 MHz, CDCl₃)



Figure S38. HMBC NMR spectrum of compound 4 (500 MHz, CDCl₃)



Figure S39. The magnified HMBC NMR spectrum of compound 4 (500 MHz, $CDCl_3$)



Figure S40. NOESY NMR spectrum of compound 4 (500 MHz, CDCl₃)



Figure S41. HRESIMS spectrum of compound 5



Figure S42. IR spectrum of compound 5



Figure S43. ¹H NMR spectrum of compound 5 (500 MHz, CDCl₃)



Figure S44. ¹³C NMR spectrum of compound 5 (125 MHz, CDCl₃)



Figure S45. DEPT NMR spectrum of compound 5 (125 MHz, CDCl₃)



Figure S46. ¹H-¹H COSY NMR spectrum of compound 5 (500 MHz, CDCl₃)



Figure S47. HSQC NMR spectrum of compound 5 (500 MHz, CDCl₃)



Figure S48. HMBC NMR spectrum of compound 5 (500 MHz, CDCl₃)



Figure S49. The magnified HMBC NMR spectrum of compound 5 (500 MHz, CDCl₃)



Figure S50. NOESY NMR spectrum of compound 5 (500 MHz, CDCl₃)



Figure S51. HRESIMS spectrum of compound 6



Figure S52. IR spectrum of compound 6



Figure S53. ¹H NMR spectrum of compound 6 (500 MHz, CDCl₃)



Figure S54. ¹³C NMR spectrum of compound 6 (125 MHz, CDCl₃)



Figure S55. DEPT NMR spectrum of compound 6 (125 MHz, CDCl₃)



Figure S56. ¹H-¹H COSY NMR spectrum of compound 6 (500 MHz, CDCl₃)



Figure S57. HSQC NMR spectrum of compound 6 (500 MHz, CDCl₃)

AFC-563b/5 - AFC-563b HSQC



Figure S58. HMBC NMR spectrum of compound 6 (500 MHz, CDCl₃)



Figure S59. The magnified HMBC NMR spectrum of compound 6 (500 MHz, CDCl₃)



Figure S60. NOESY NMR spectrum of compound 6 (500 MHz, CDCl₃)



Figure S61. HRESIMS spectrum of compound 7



Figure S62. IR spectrum of compound 7



Figure S63. ¹H NMR spectrum of compound 7 (500 MHz, CDCl₃)



Figure S64. ¹³C NMR spectrum of compound 7 (125 MHz, CDCl₃)



Figure S65. DEPT NMR spectrum of compound 7 (125 MHz, CDCl₃)



Figure S66. ¹H-¹H COSY NMR spectrum of compound 7 (500 MHz, CDCl₃)



Figure S67. HSQC NMR spectrum of compound 7 (500 MHz, CDCl₃)



Figure S68. HMBC NMR spectrum of compound 7 (500 MHz, CDCl₃)



Figure S69. The magnified HMBC NMR spectrum of compound 7 (500 MHz, CDCl₃)



Figure S70. NOESY NMR spectrum of compound 7 (500 MHz, CDCl₃)



Figure S71. HRESIMS spectrum of compound 8



Figure S72. IR spectrum of compound 8



Figure S73. ¹H NMR spectrum of compound 8 (500 MHz, CDCl₃)



Figure S74. ¹³C NMR spectrum of compound 8 (125 MHz, CDCl₃)



Figure S75. DEPT NMR spectrum of compound 8 (125 MHz, CDCl₃)



Figure S76. ¹H-¹H COSY NMR spectrum of compound 8 (500 MHz, CDCl₃)



Figure S77. HSQC NMR spectrum of compound 8 (500 MHz, CDCl₃)



Figure S78. HMBC NMR spectrum of compound 8 (500 MHz, CDCl₃)



Figure S79. The magnified HMBC NMR spectrum of compound 8 (500 MHz, CDCl₃)



Figure S80. NOESY NMR spectrum of compound 8 (500 MHz, CDCl₃)



Figure S81. ¹H NMR spectrum of Isovaleric acid (500 MHz, CDCl₃)



Figure S82. ¹³C NMR spectrum of Isovaleric acid (125 MHz, CDCl₃)



Figure S83. DEPT NMR spectrum of Isovaleric acid (125 MHz, CDCl₃)



Figure S84. Viability of A549 cells after exposure to 0.1% DMSO or various concentrations of compound 2 for 24, 48, and 72 h.



The data are expressed as the means \pm SEM of three independent experiments.