

SUPPLEMENTARY MATERIAL

UPLC-QTOF-MS-guided isolation of anti-COPD ginsenosides from Wild Ginseng

Hailin Zhu^a, Junli Liu^a, Hongqiang Lin^a, Ying Zhang^{a,c}, Na Yang^a, Baisong Zhou^a,

Zhongyao Wang^a, Alan Chen-Yu Hsu^d, Jinping Liu^{a,b*}, Pingya Li^{a*}

^aSchool of Pharmaceutical Sciences, Jilin University, Changchun 130021, Jilin, China

^bResearch Center of Natural Drug, Jilin University, Changchun 130021, Jilin, China

^cThe First Hospital of Jilin University, Changchun 130021, Jilin, China

^dPriority Research Centre for Healthy Lungs, Faculty of Health and Medicine, The University of Newcastle, Newcastle, NSW, Australia

*Co-Correspondence authors: Prof. Jinping Liu and Prof. Pingya Li, School of Pharmaceutical Sciences, Jilin University, Fujin Road 1266, Changchun 130021, China, E-mail: liujp@jlu.edu.cn (J.L.); lipy@jlu.edu.cn (P.L.) Tel.: +86-431-85619803 (J.L.)

Contents of Supplementary Information

Figure S1-1. ¹H NMR spectrum of Rm1 in DMSO-D₆ (600 MHz).

Figure S1-2. ¹³C-DEPT NMR spectrum of Rm1 in DMSO-D₆ (150 MHz).

Figure S1-3. Heteronuclear multiple quantum correlation (HMQC) spectrum of Rm1.

Figure S1-4. Heteronuclear multiple bond coherence (HMBC) spectrum of Rm1.

Figure S1-5. High resolution electrospray ionization mass spectroscopy (HRESIMS) spectrum of MS^E fragment ions of Rm1.

Figure S2-1. ¹H NMR spectrum of Rm2 in DMSO-D₆ (600 MHz).

Figure S2-2. ¹³C-DEPT NMR spectrum of Rm2 in DMSO-D₆ (150 MHz).

Figure S2-3. Heteronuclear multiple quantum correlation (HMQC) spectrum of Rm2.

Figure S2-4. Heteronuclear multiple bond coherence (HMBC) spectrum of Rm2.

Figure S2-5. High resolution electrospray ionization mass spectroscopy (HRESIMS) spectrum of MS^E fragment ions of Rm2.

Figure S3-1. ¹H NMR spectrum of Rm3 in DMSO-D₆ (600 MHz).

Figure S3-2. ¹³C-DEPT NMR spectrum of Rm3 in DMSO-D₆ (150 MHz).

Figure S3-3. Heteronuclear multiple quantum correlation (HMQC) spectrum of Rm3.

Figure S3-4. Heteronuclear multiple bond coherence (HMBC) spectrum of Rm3.

Figure S3-5. High resolution electrospray ionization mass spectroscopy (HRESIMS) spectrum of MS^E fragment ions of Rm3.

Figure S4-1. ¹H NMR spectrum of Rm4 in DMSO-D₆ (600 MHz).

Figure S4-2. ¹³C-DEPT NMR spectrum of Rm4 in DMSO-D₆ (150 MHz).

Figure S4-3. Heteronuclear multiple quantum correlation (HMQC) spectrum of Rm4.

Figure S4-4. Heteronuclear multiple bond coherence (HMBC) spectrum of Rm4.

Figure S4-5. High resolution electrospray ionization mass spectroscopy (HRESIMS) spectrum of MS^E fragment ions of Rm4.

Figure S5-1. ¹H NMR spectrum of Rb2 in Pyridine-D₅(600 MHz).

Figure S5-2. ¹³C-DEPT NMR spectrum of Rb2 in Pyridine-D₅ (150 MHz).

Figure S6-1. ¹H NMR spectrum of Rd in Pyridine-D₅(600 MHz).

Figure S6-2. ¹³C-DEPT NMR spectrum of Rd in Pyridine-D₅ (150 MHz).

Figure S7-1. ¹H NMR spectrum of Rg3 in DMSO-D₆ (600 MHz).

Figure S7-2. ¹³C-DEPT NMR spectrum of Rg3 in DMSO-D₆ (150 MHz).

Figure S8-1. ¹H NMR spectrum of Rg1 in DMSO-D₆ (600 MHz).

Figure S8-2. ¹³C-DEPT NMR spectrum of Rg1 in Pyridine-D₆ (150 MHz).

Figure S9-1. ¹H NMR spectrum of Rh2 in DMSO-D₆ (600 MHz).

Figure S9-2. ¹³C-DEPT NMR spectrum of Rh2 in DMSO-D₆ (150 MHz).

Figure S10. Anti-inflammatory effect of petroleum ether soluble extracts (PEe), ethyl acetate soluble extracts(ASE) , n-butanol soluble extracts(BSe) (200 μM)(A), FrA-J(B) and G1-6(C) on the inflammatory cytokine tumor necrosis factor- α (TNF- α), interleukin-1 β (IL-1 β) and interleukin-6 (IL-6) in CSE-exposed A549 cells.

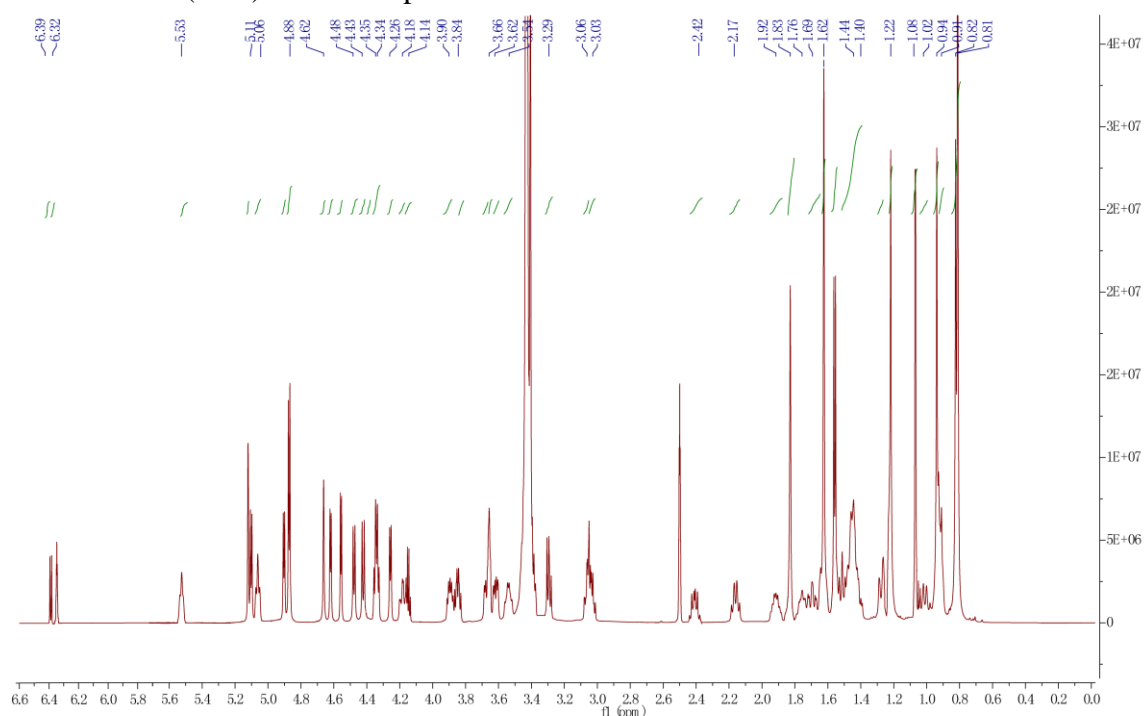


Figure S1-1. ¹H NMR spectrum of Rm1 in DMSO-D₆ (600 MHz).

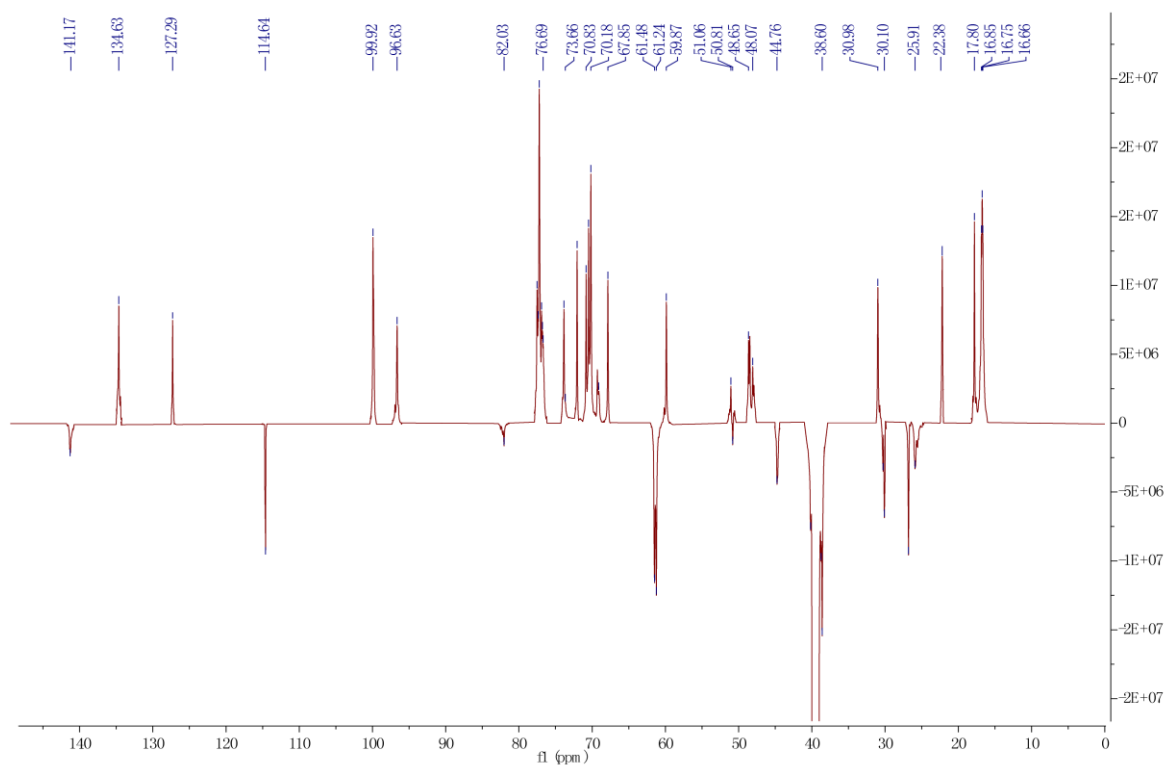


Figure S1-2. ^{13}C -DEPT NMR spectrum of Rm1 in DMSO-D_6 (150 MHz).

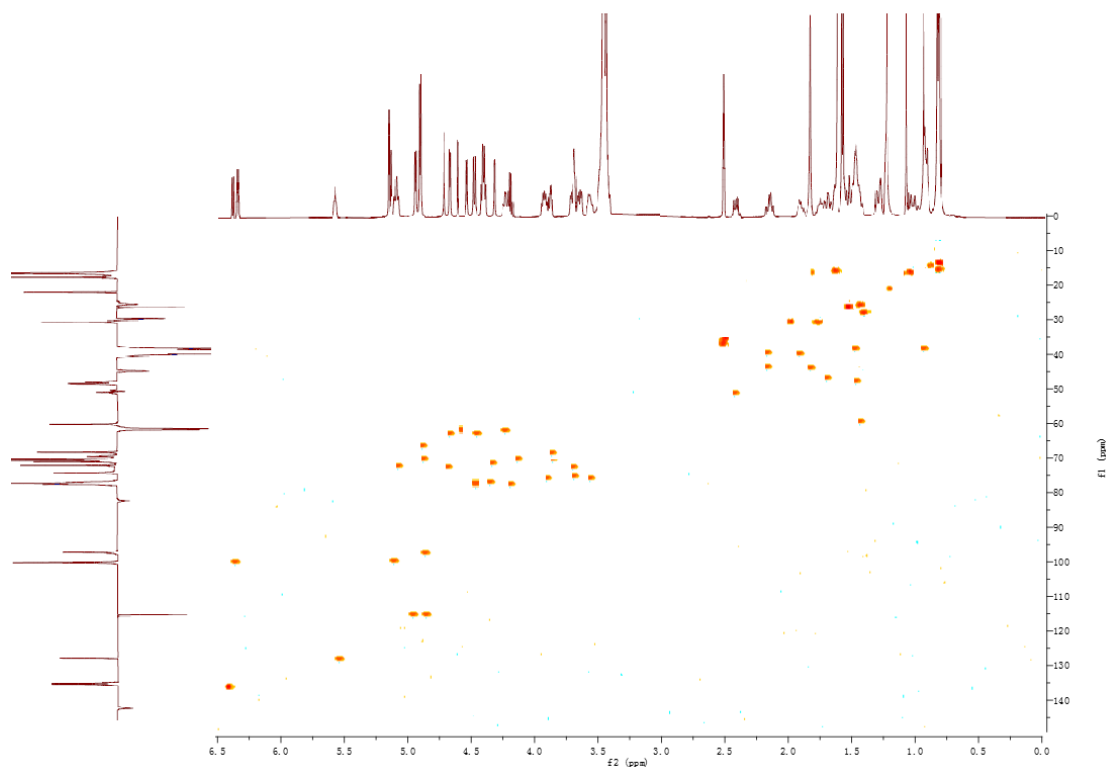


Figure S1-3. Heteronuclear multiple quantum correlation (HMQC) spectrum of Rm1.

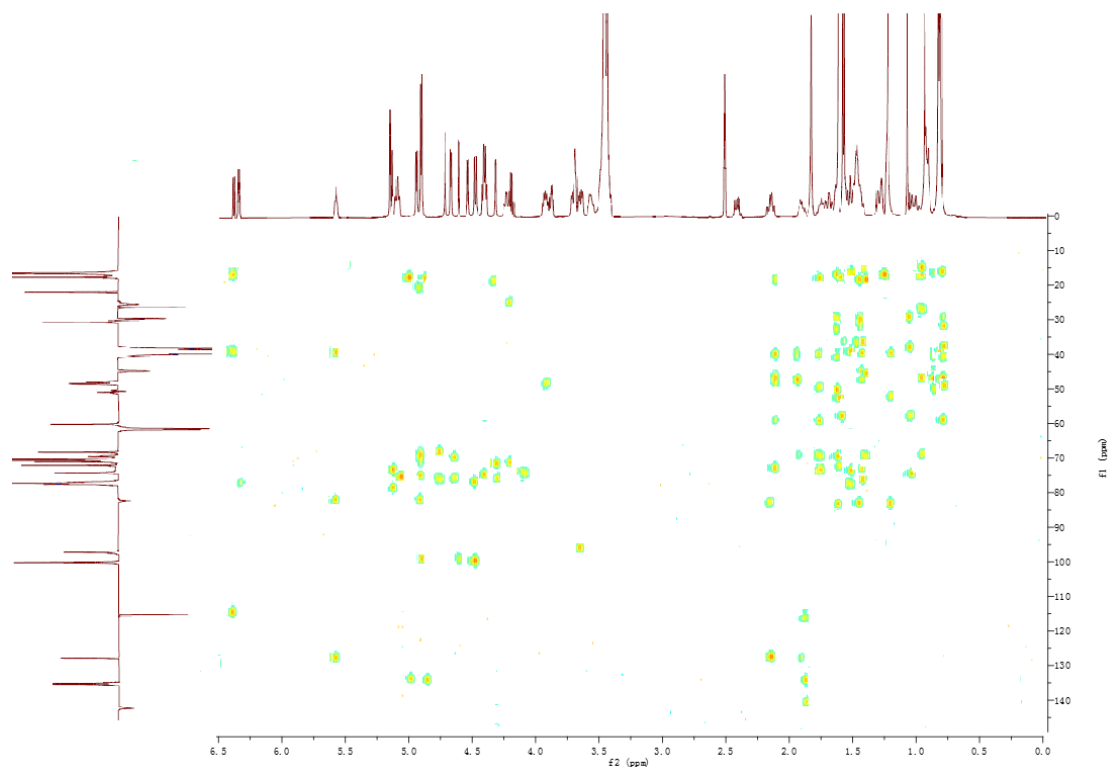


Figure S1-4. Heteronuclear multiple bond coherence (HMBC) spectrum of Rm1.

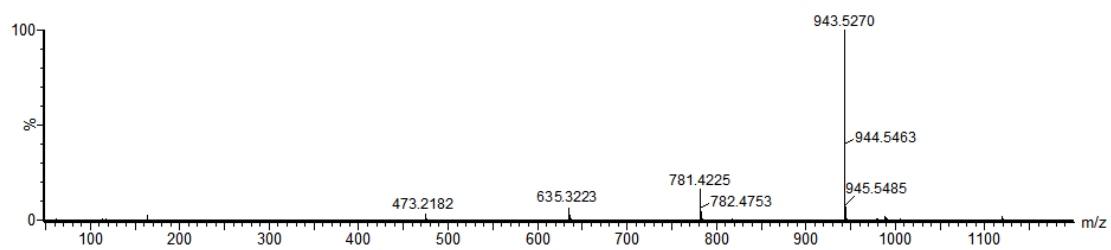


Figure S1-5. High resolution electrospray ionization mass spectrometry (HRESIMS) spectrum of MS^E fragment ions of Rm1.

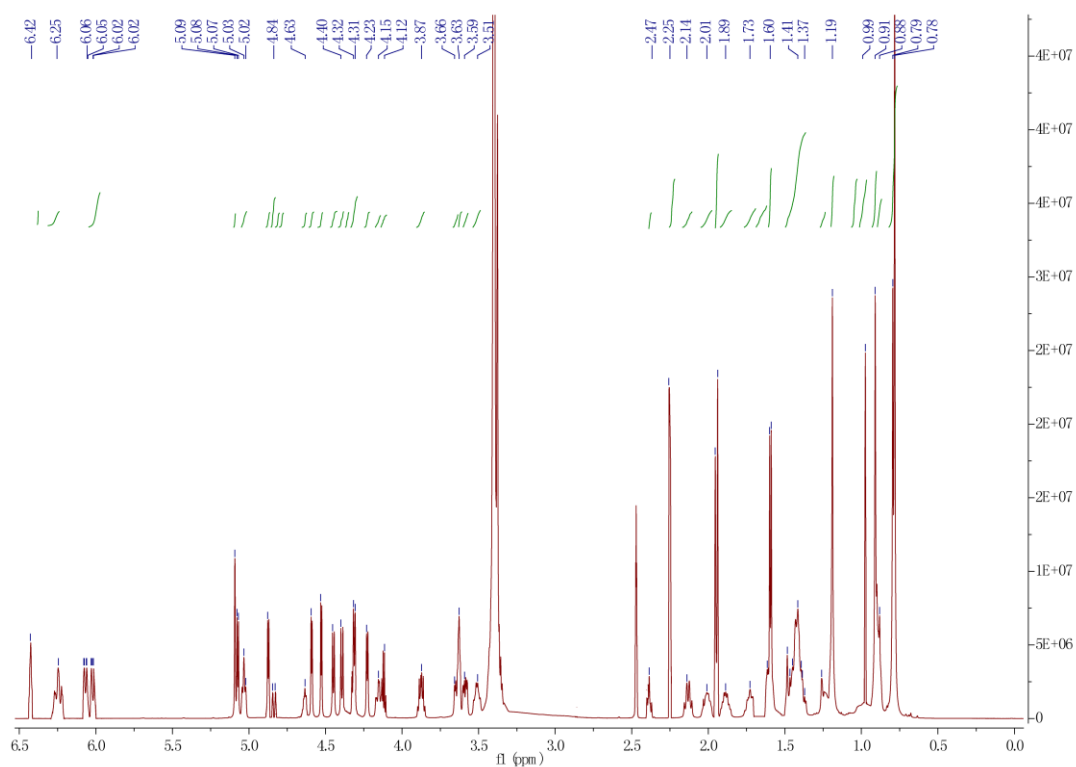


Figure S2-1. ^1H NMR spectrum of Rm2 in DMSO-D_6 (600 MHz).

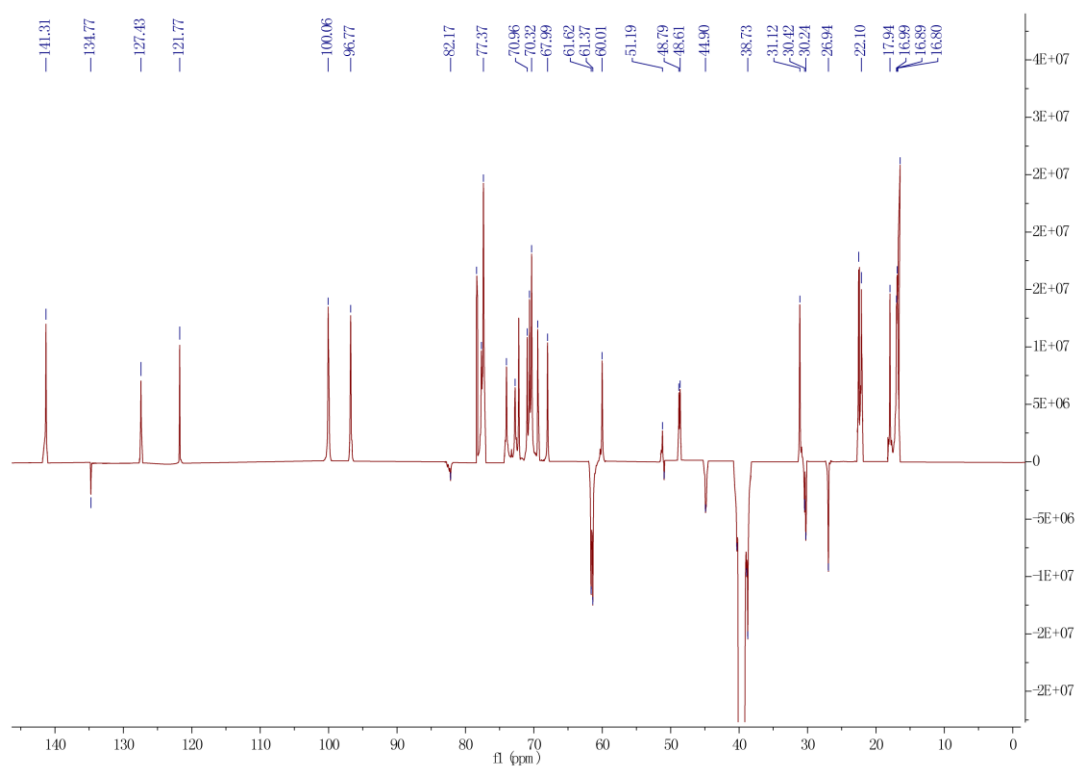


Figure S2-2. ^{13}C -DEPT NMR spectrum of Rm2 in DMSO-D_6 (150 MHz).

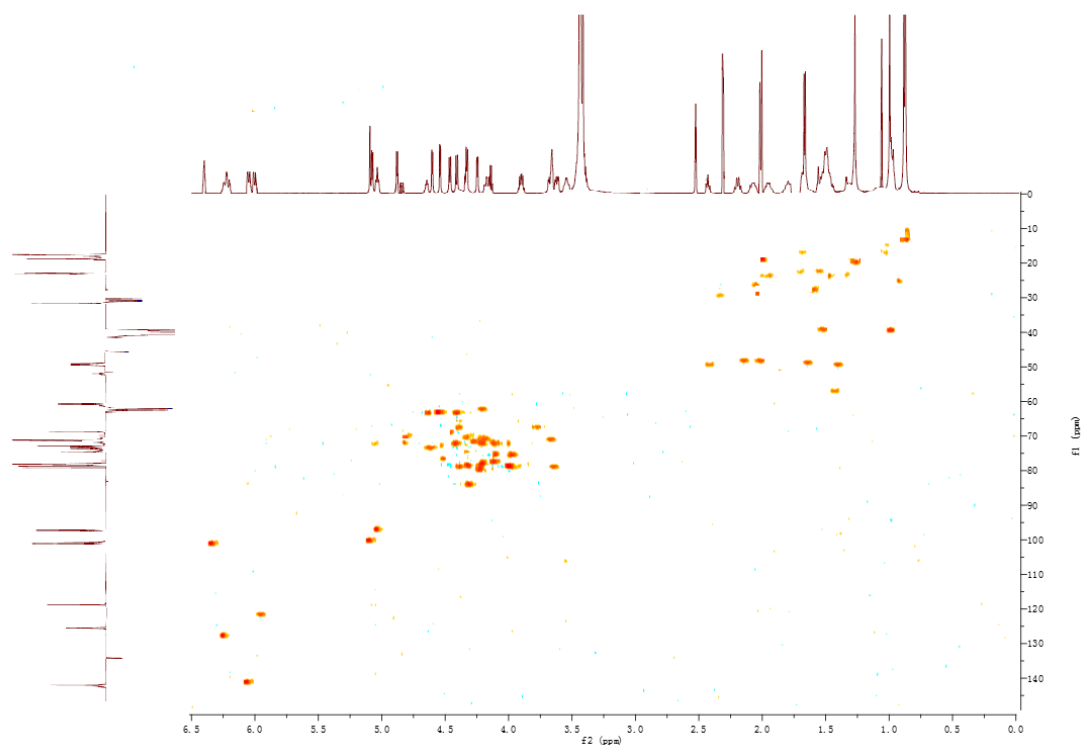


Figure S2-3. Heteronuclear multiple quantum correlation (HMQC) spectrum of Rm2.

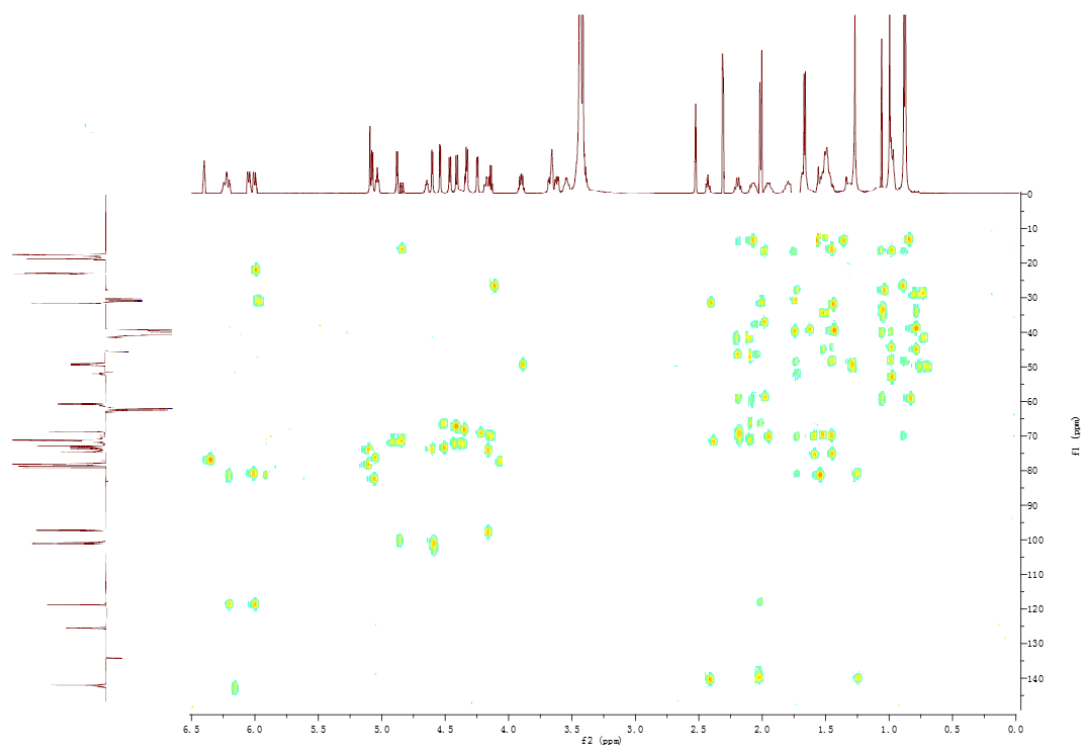


Figure S2-4. Heteronuclear multiple bond coherence (HMBC) spectrum of Rm2.

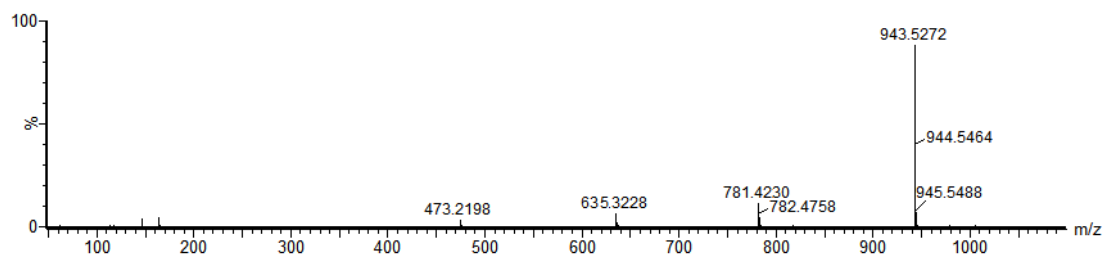


Figure S2-5. High resolution electrospray ionization mass spectroscopy (HRESIMS) spectrum of MS_E fragment ions of Rm2.

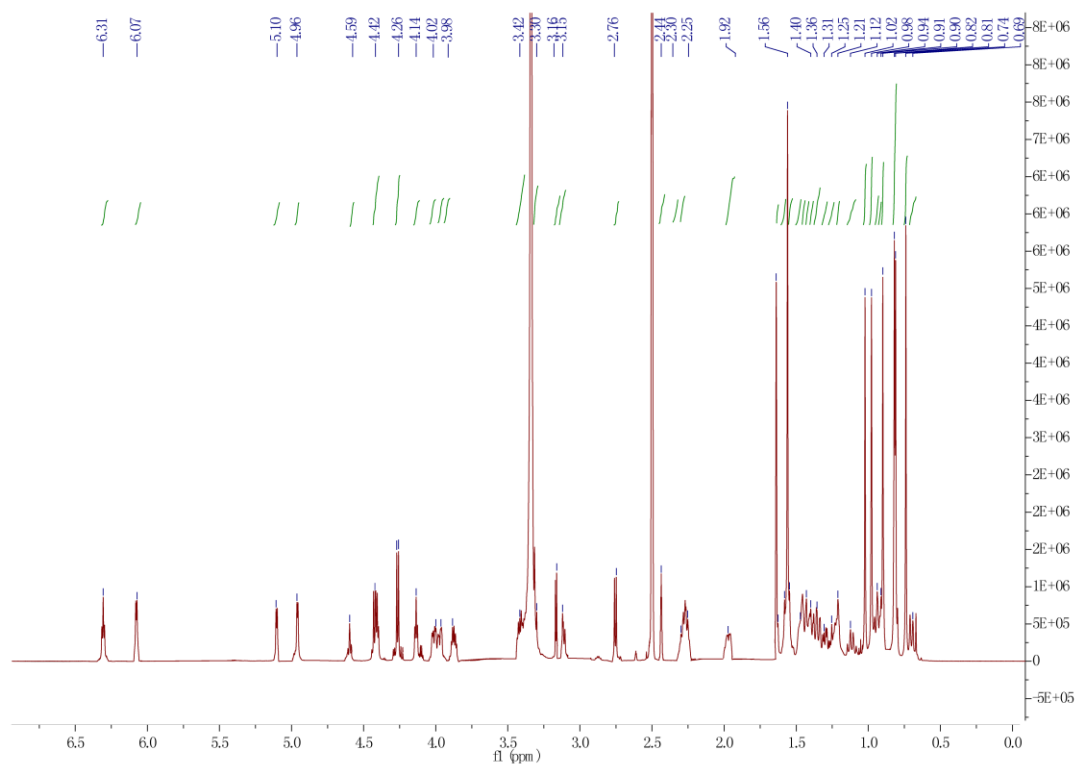


Figure S3-1. ¹H NMR spectrum of Rm3 in DMSO-D₆ (600 MHz).

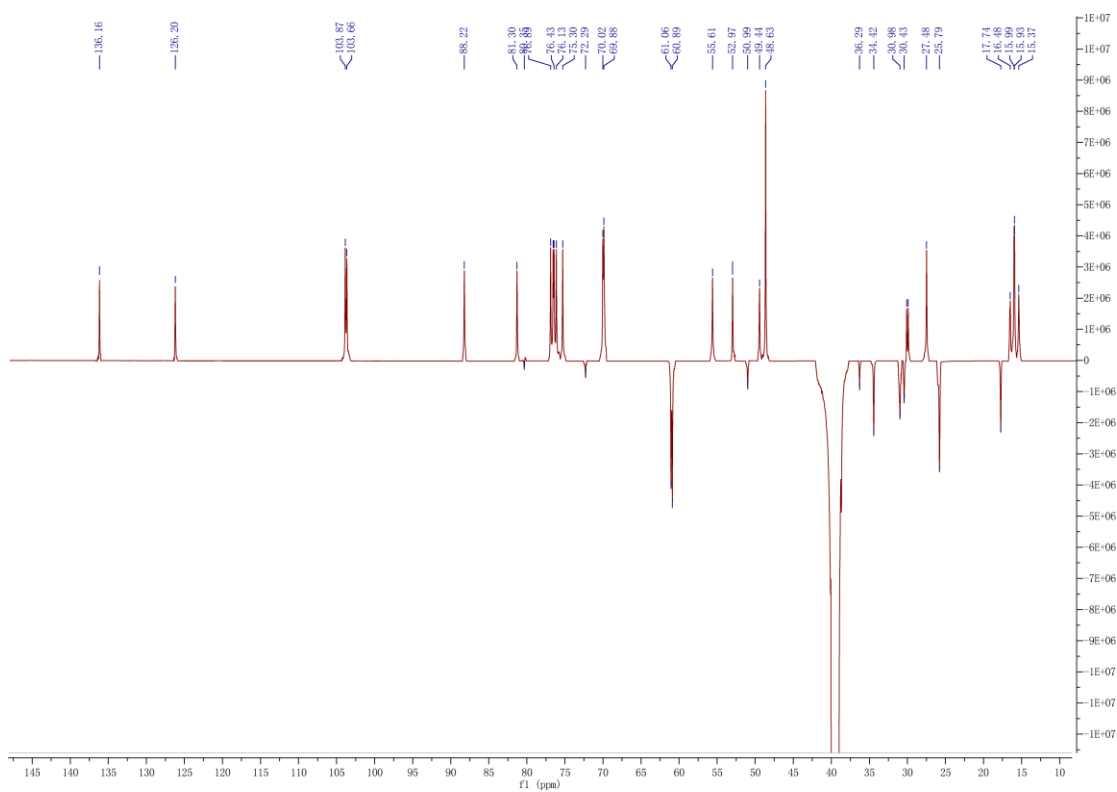


Figure S3-2. ^{13}C -DEPT NMR spectrum of Rm3 in DMSO-D_6 (150 MHz).

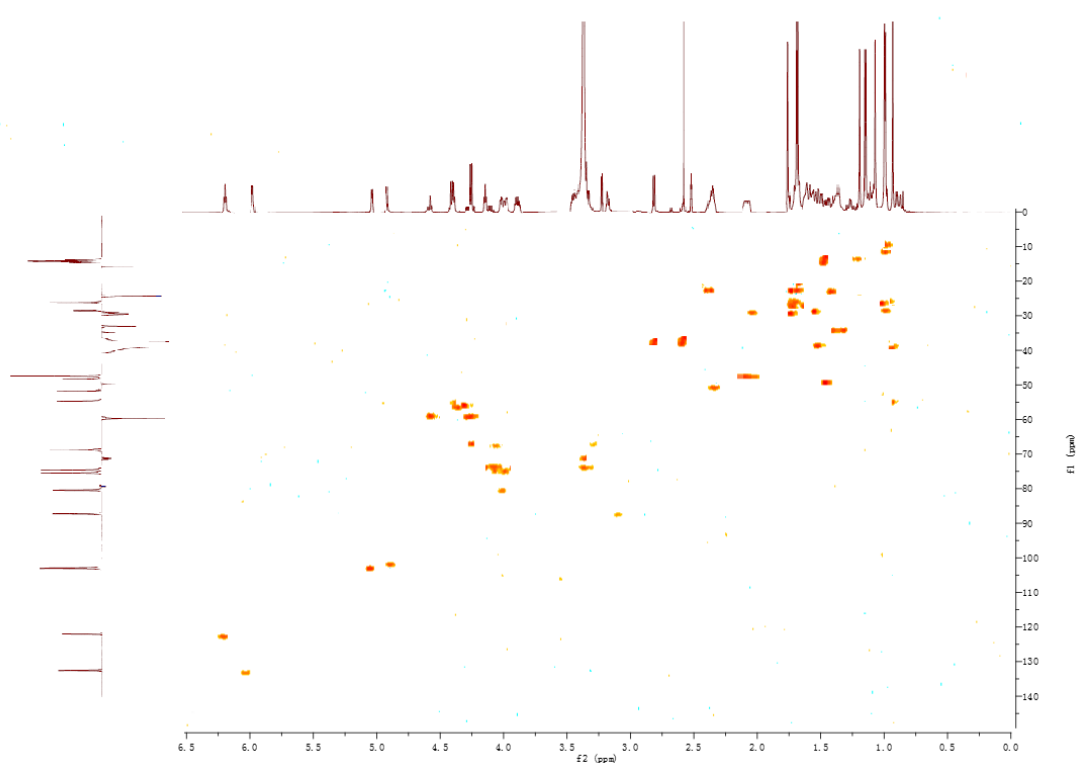


Figure S3-3. Heteronuclear multiple quantum correlation (HMQC) spectrum of Rm3.

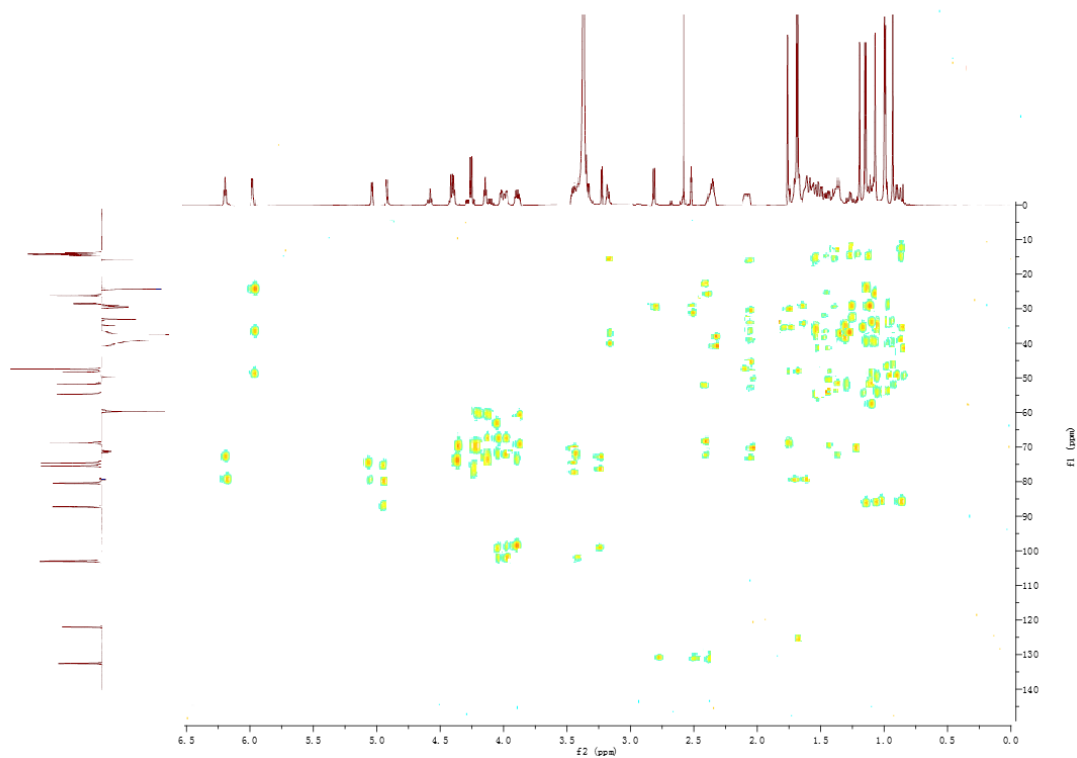


Figure S3-4. Heteronuclear multiple bond coherence (HMBC) spectrum of Rm3.

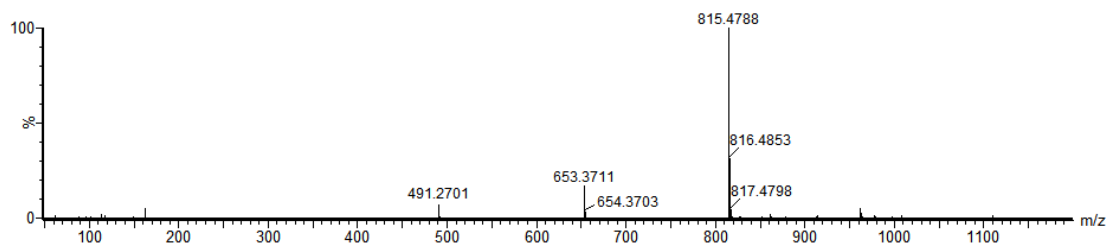


Figure S3-5. High resolution electrospray ionization mass spectrometry (HRESIMS) spectrum of MS_E fragment ions of Rm3.

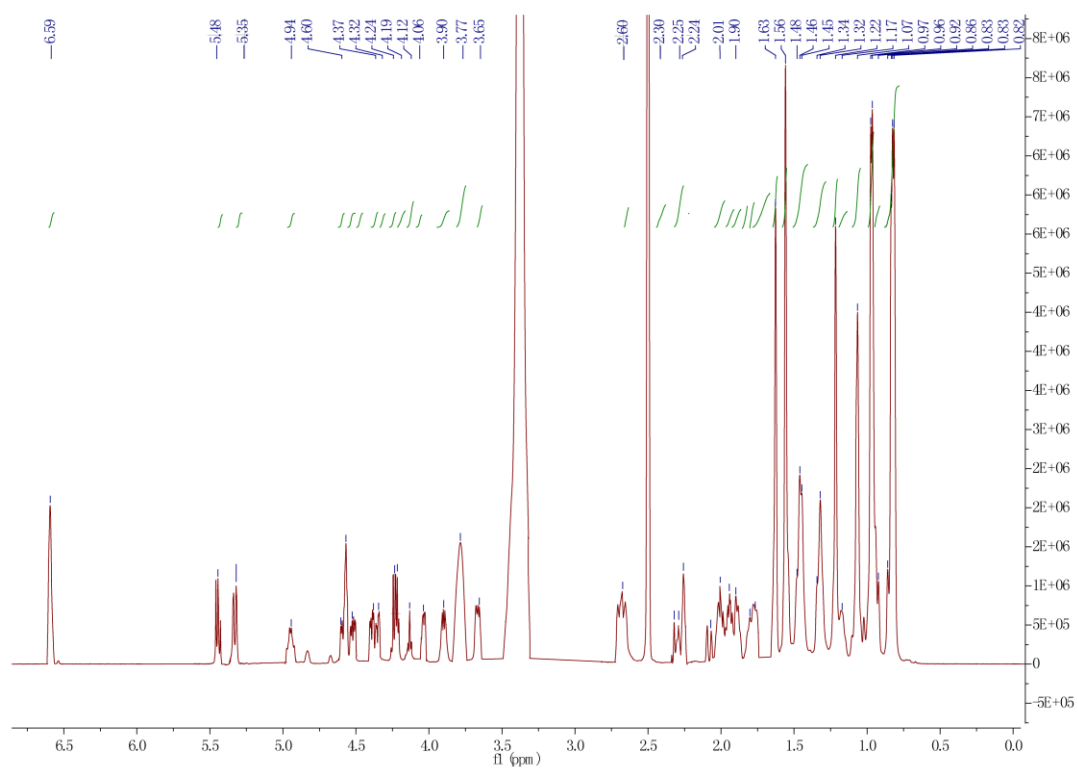


Figure S4-1. ^1H NMR spectrum of Rm4 in DMSO-D_6 (600 MHz).

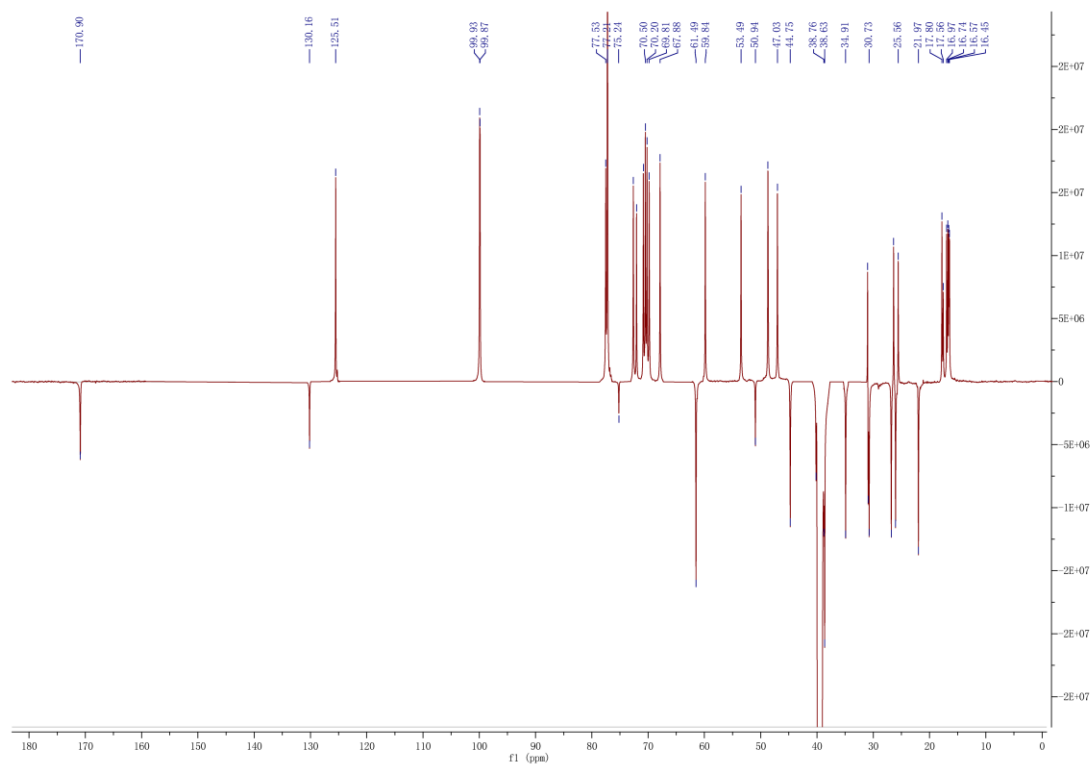


Figure S4-2. ^{13}C -DEPT NMR spectrum of Rm4 in DMSO-D_6 (150 MHz).

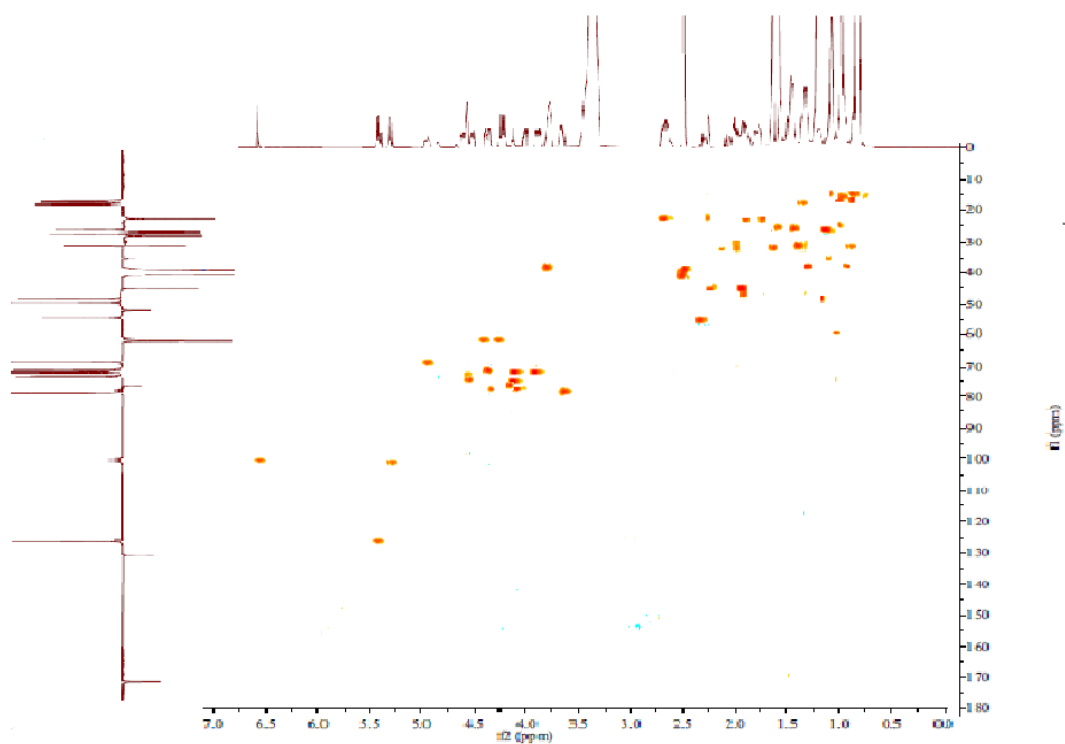


Figure S4-3. Heteronuclear multiple quantum correlation (HMQC) spectrum of Rm4.

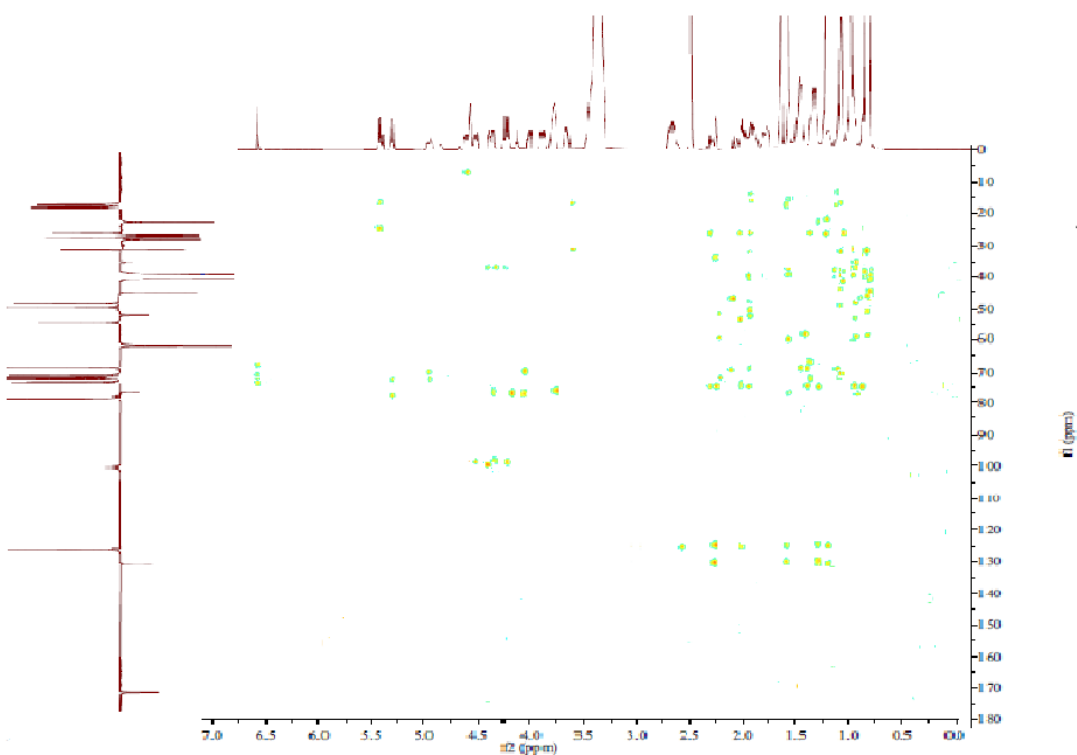


Figure S4-4. Heteronuclear multiple bond coherence (HMBC) spectrum of Rm4.

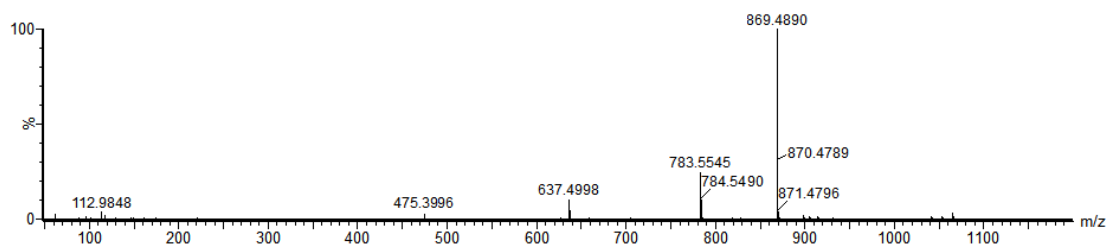


Figure S4-5. High resolution electrospray ionization mass spectroscopy (HRESIMS) spectrum of MS_E fragment ions of Rm4.

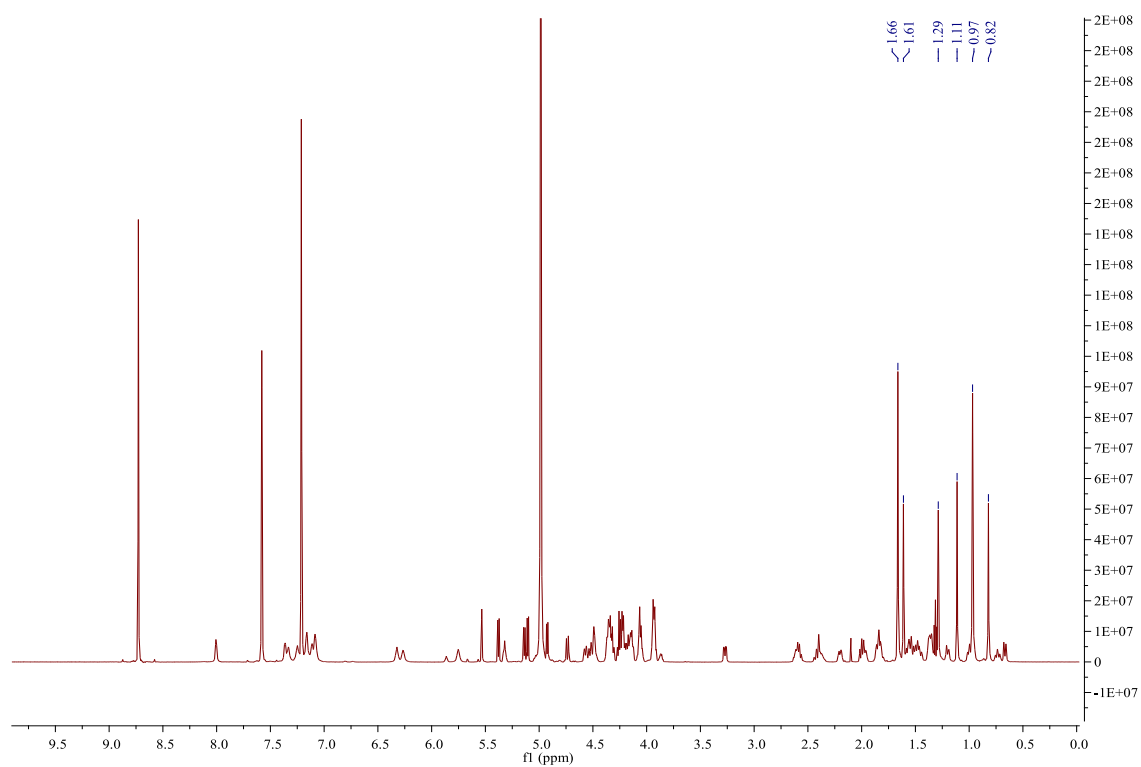


Figure S5-1. 1H NMR spectrum of Rb2 in Pyridine- D_5 (600 MHz).

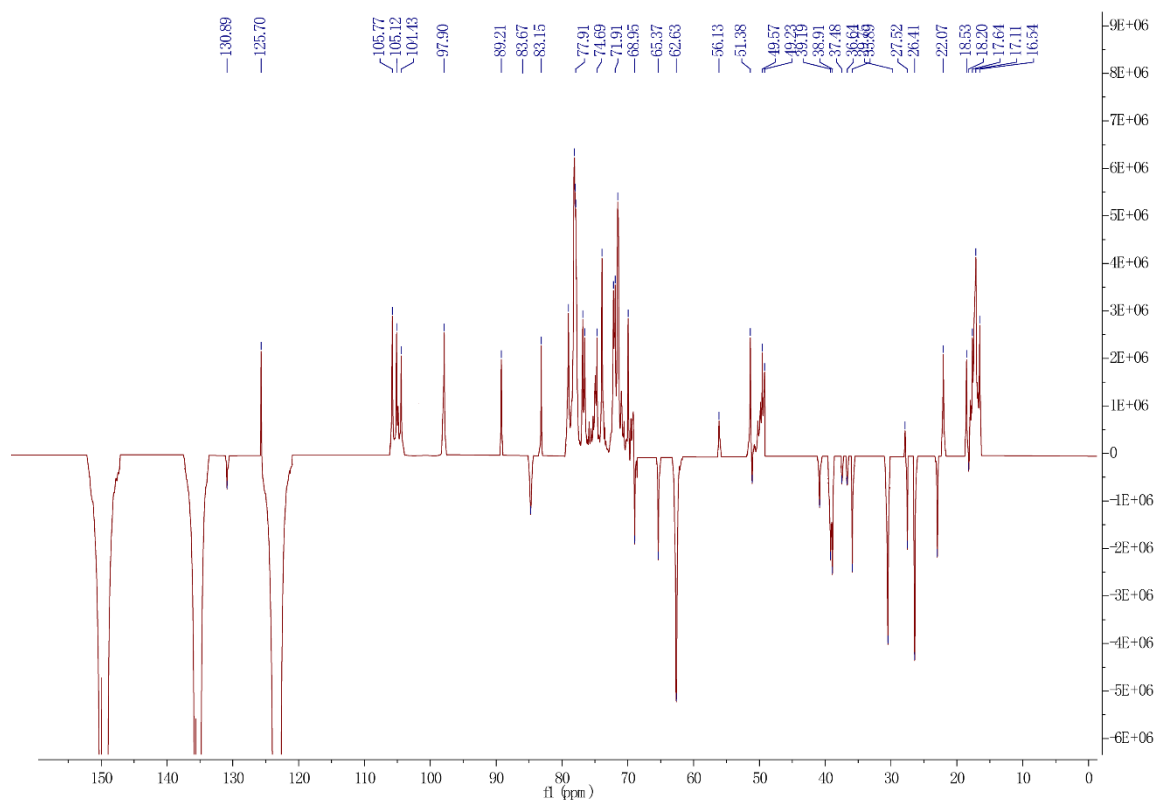


Figure S5-2. ^{13}C -DEPT NMR spectrum of Rb2 in Pyridine- D_5 (150 MHz).

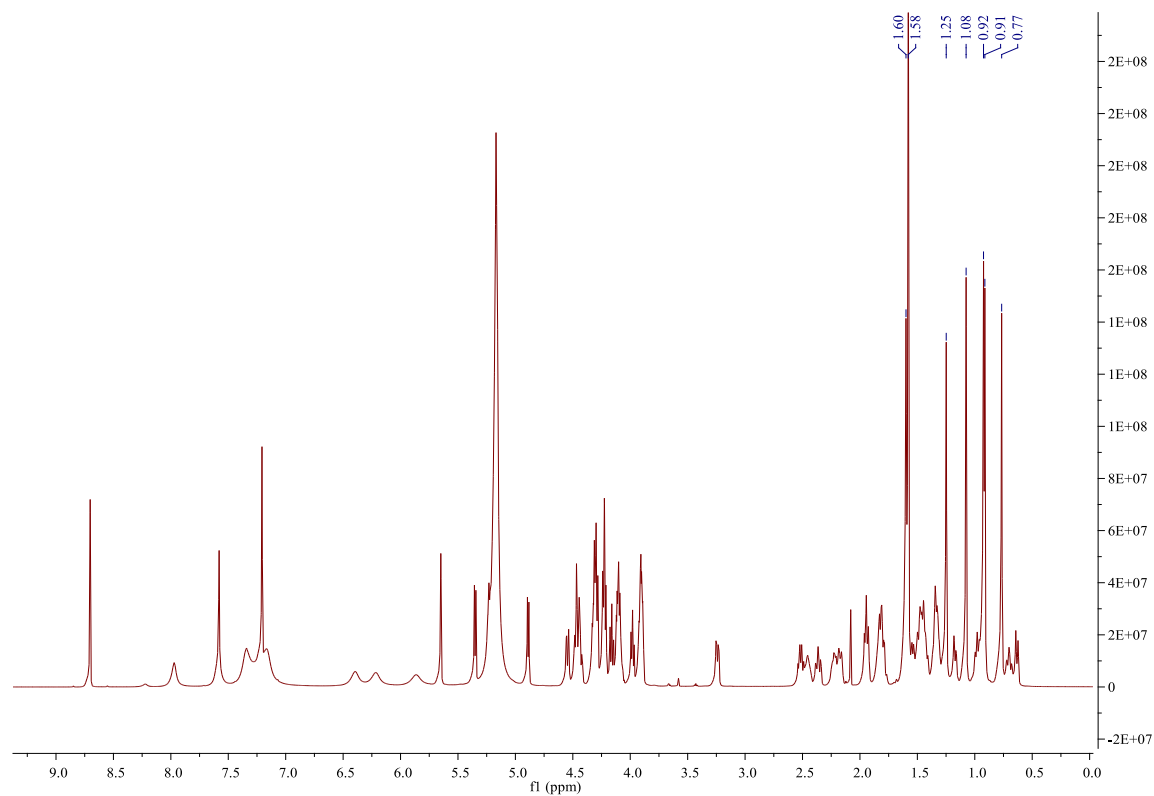


Figure S6-1. ^1H NMR spectrum of Rd in Pyridine- D_5 (600 MHz).

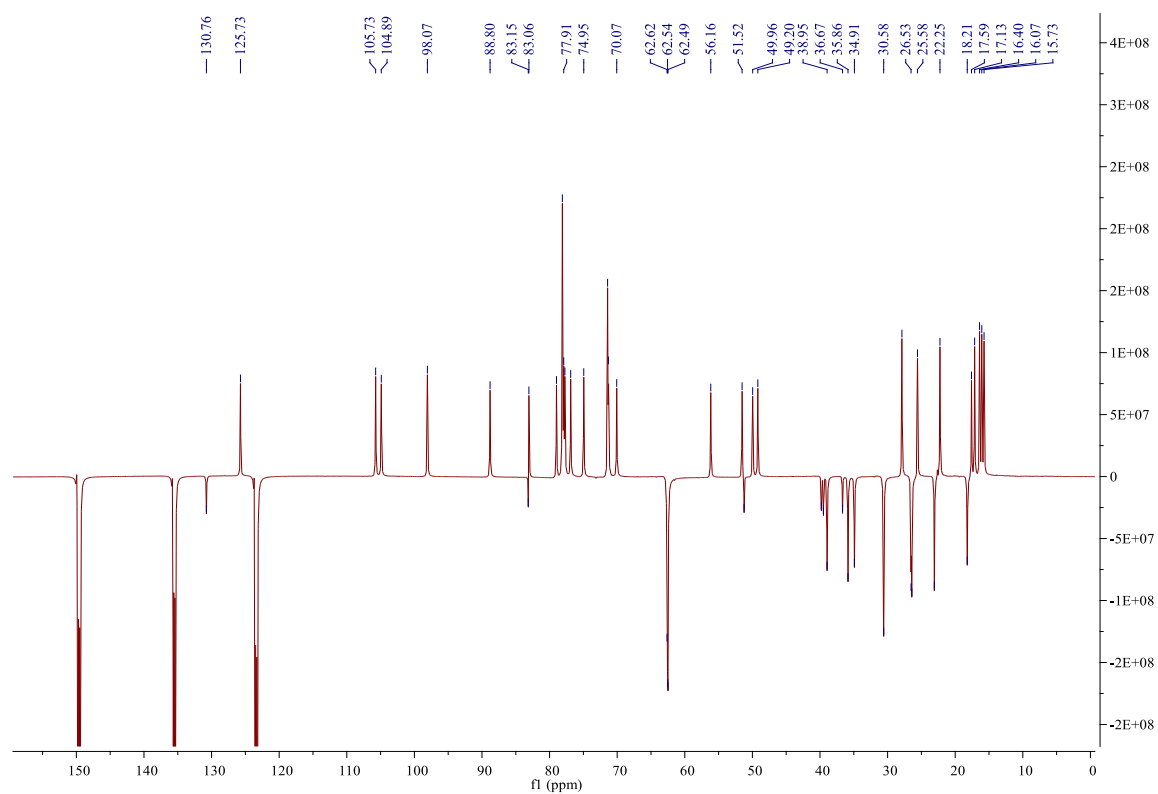


Figure S6-2. ^{13}C -DEPT NMR spectrum of Rd in Pyridine- D_5 (150 MHz).

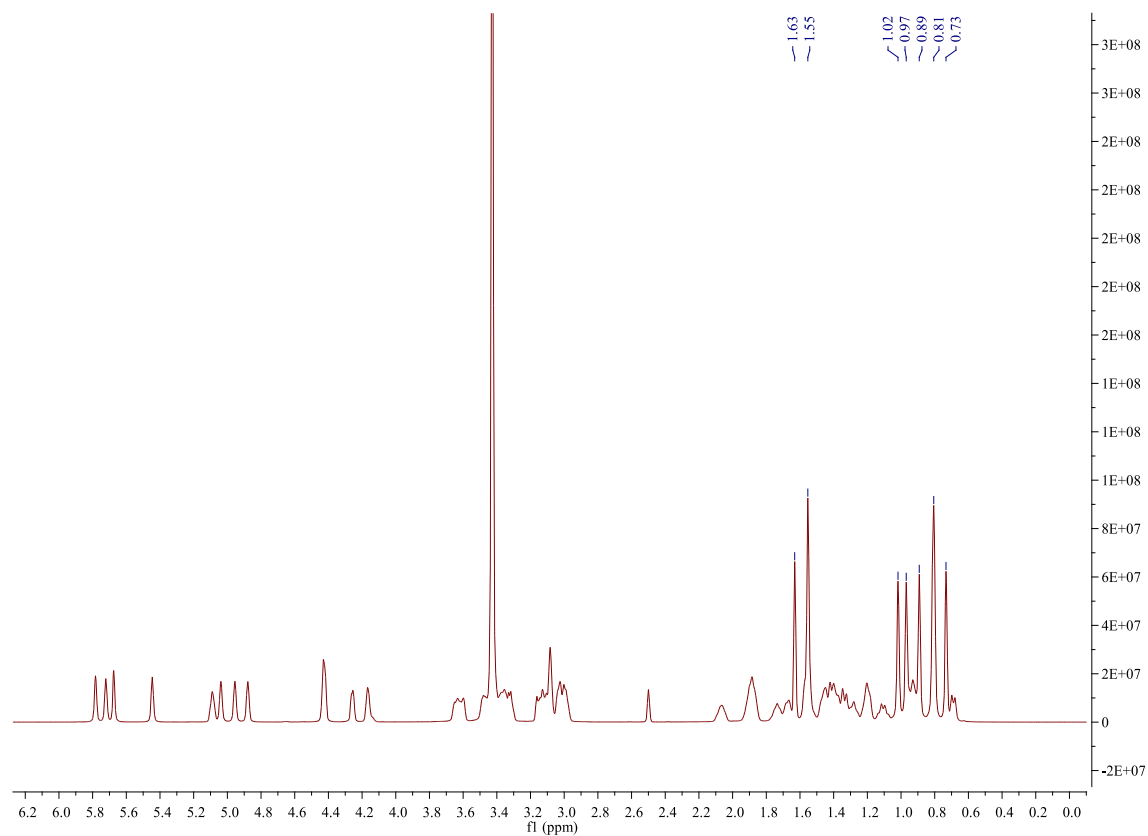


Figure S7-1. ^1H NMR spectrum of Rg3 in DMSO- D_6 (600 MHz).

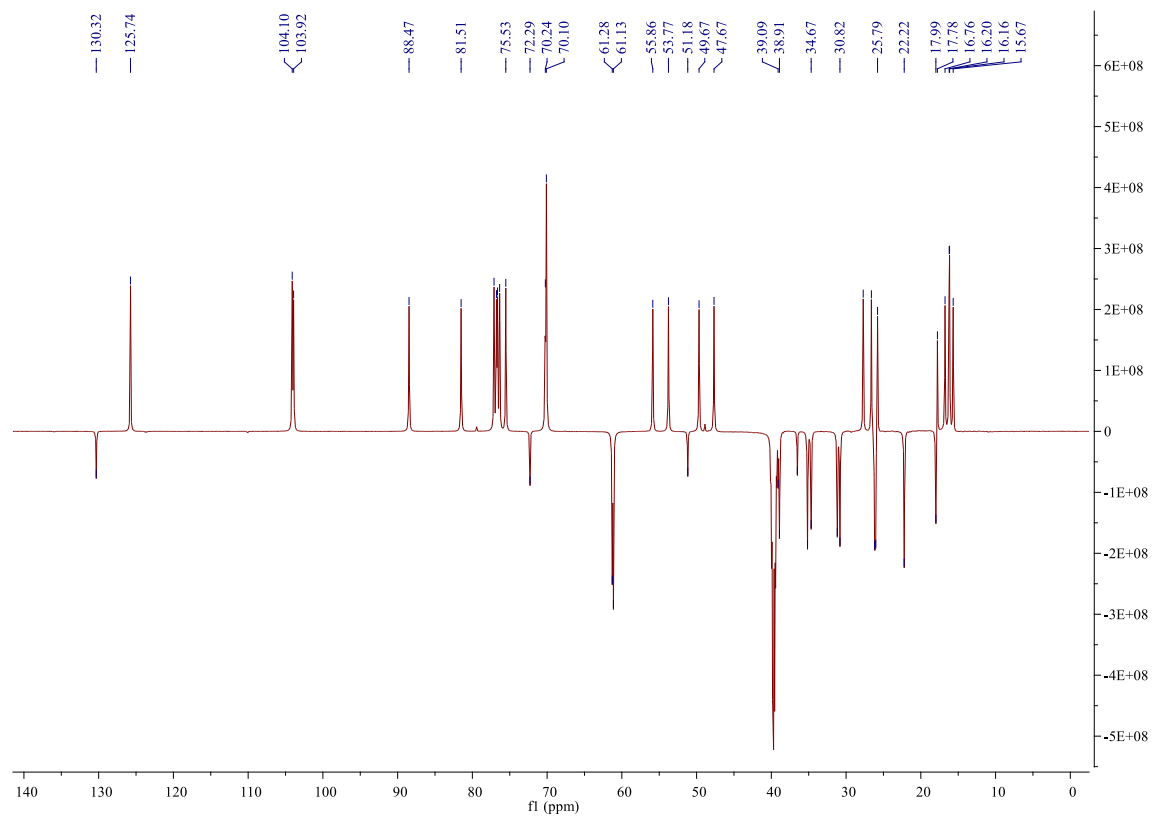


Figure S7-2. ^{13}C -DEPT NMR spectrum of Rd in DMSO- D_6 (150 MHz).

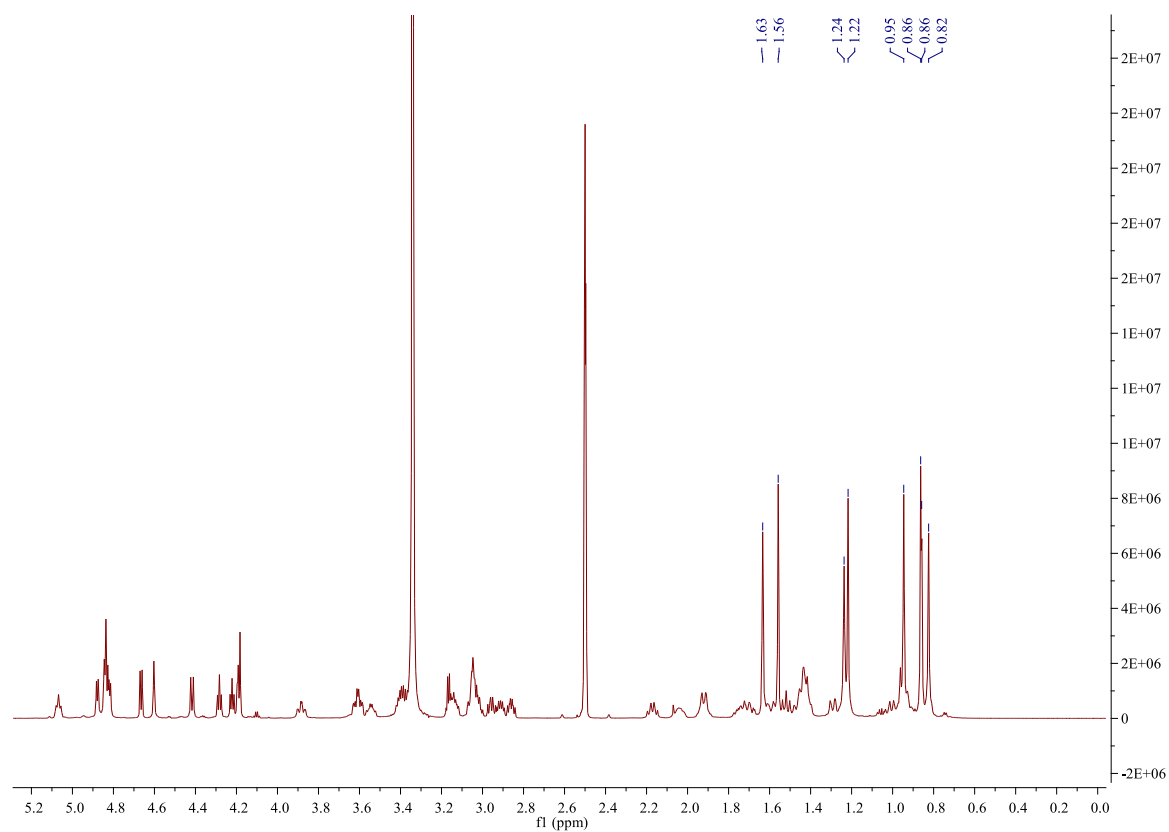


Figure S8-1. ^1H NMR spectrum of Rg1 in DMSO- D_6 (600 MHz).

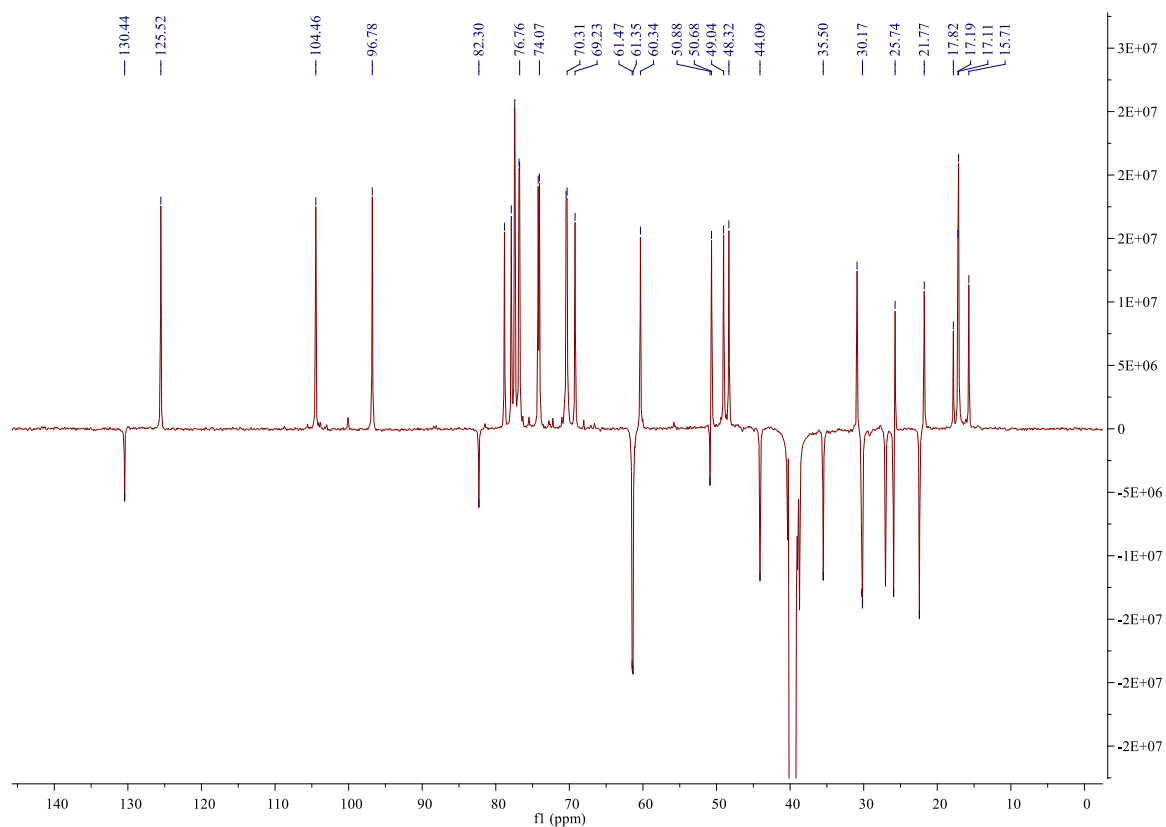


Figure S8-2. ^{13}C -DEPT NMR spectrum of Rg1 in Pyridine- D_6 (150 MHz).

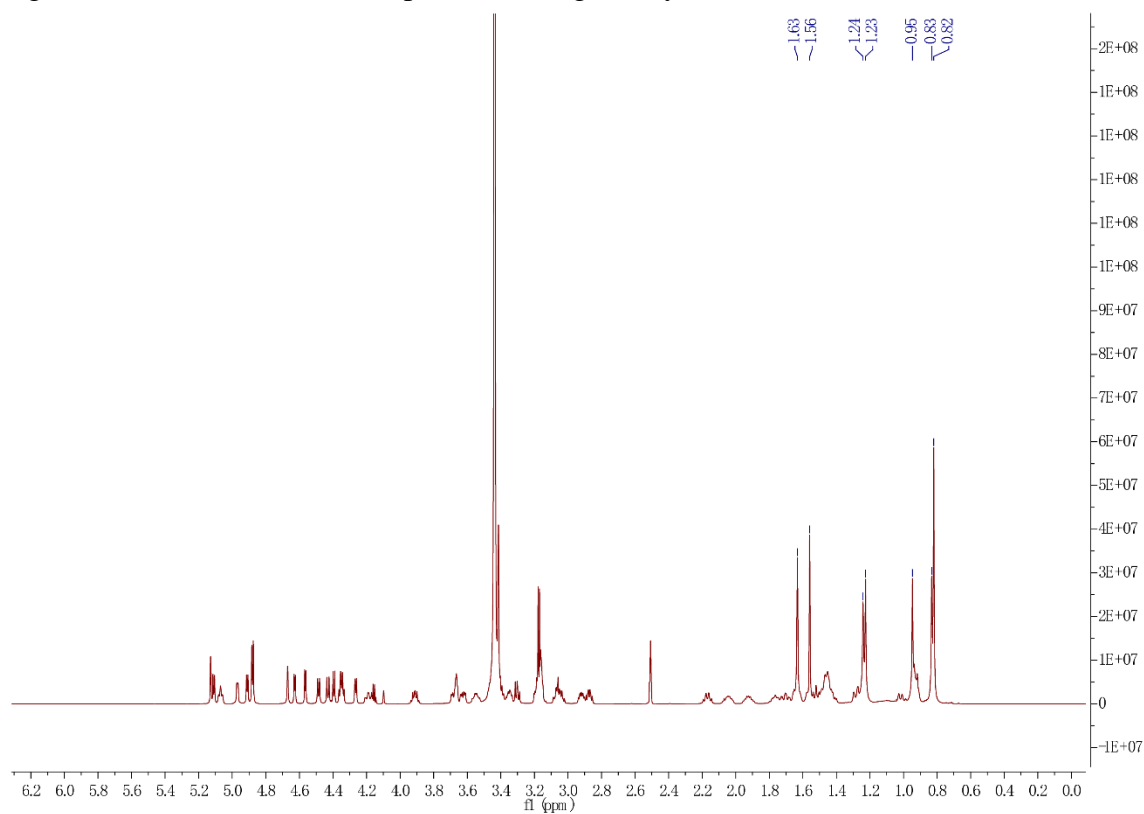


Figure S9-1. ^1H NMR spectrum of Rh2 in DMSO- D_6 (600 MHz).

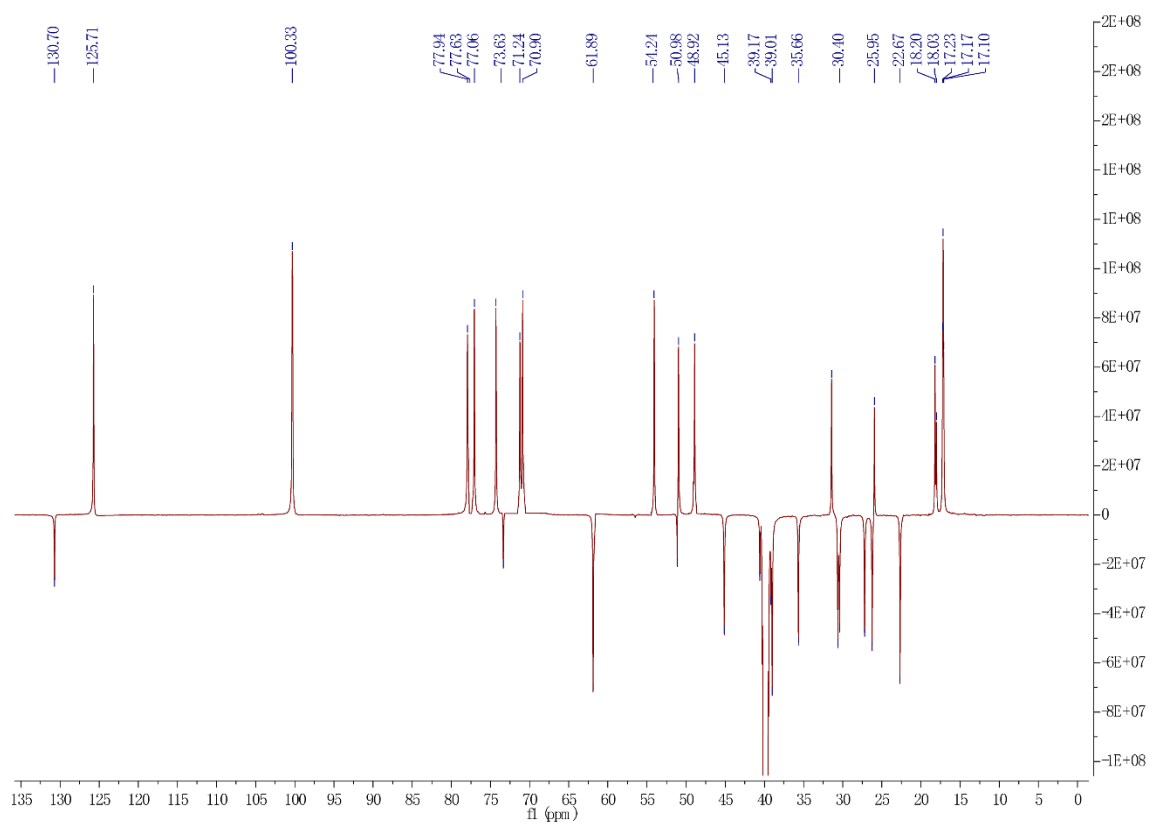
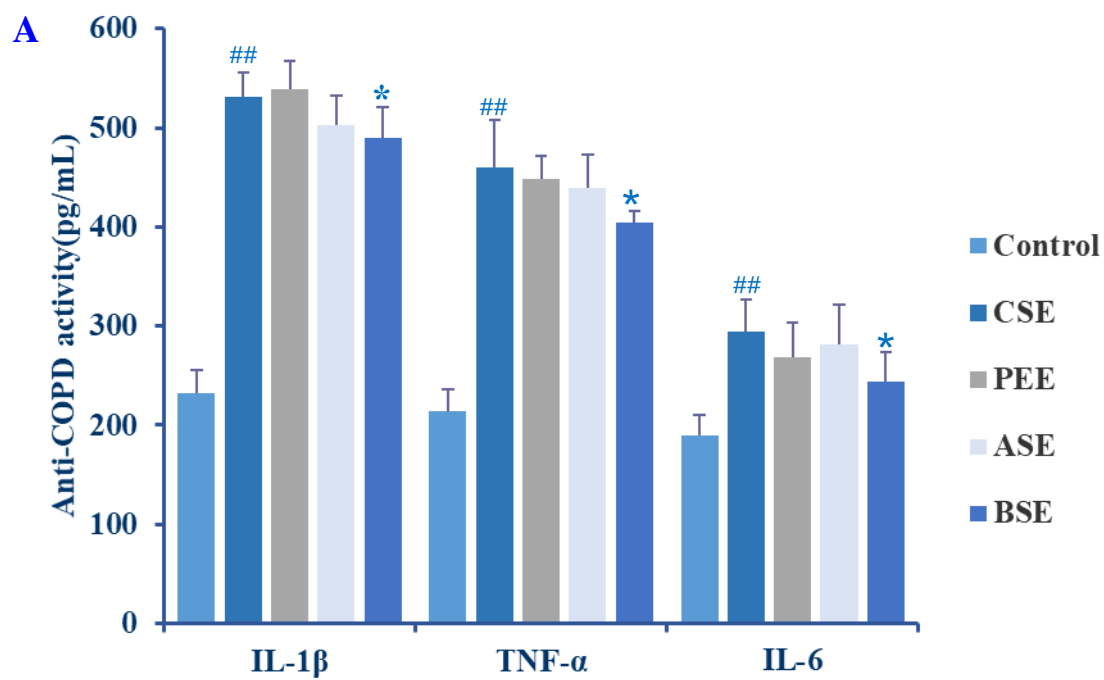


Figure S9-2. ^{13}C -DEPT NMR spectrum of Rh₂ in DMSO-D₆ (150 MHz).



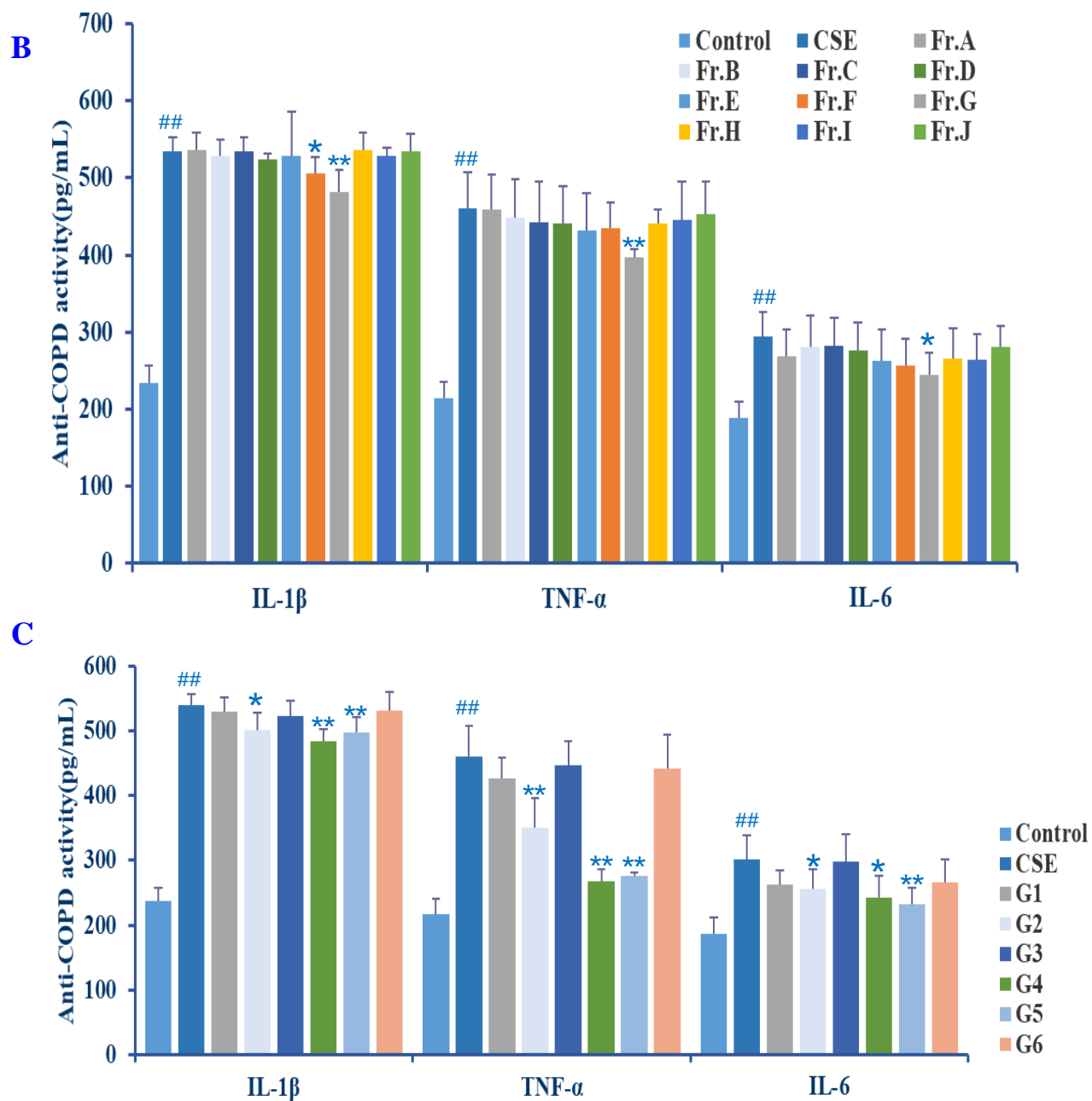


Figure S10. Anti-inflammatory effect of petroleum ether soluble extracts (PEe), ethyl acetate soluble extracts (ASe), n-butanol soluble extracts (BSe) (200 μ M) (A), FrA-J (B) and G1-6 (C) on the inflammatory cytokine tumor necrosis factor- α (TNF- α), interleukin-1 β (IL-1 β) and interleukin-6 (IL-6) in CSE-exposed A549 cells. The results are expressed as mean \pm S.D., n=6. ## p < 0.01, compared with control group; ** p < 0.01, compared with CSE group; * p < 0.05, compared with CSE group.