

**Supporting Info*

**Nine New Dihydro- β -agarofuran Sesquiterpene Polyesters
from the Leaves of *Tripterygium wilfordii***

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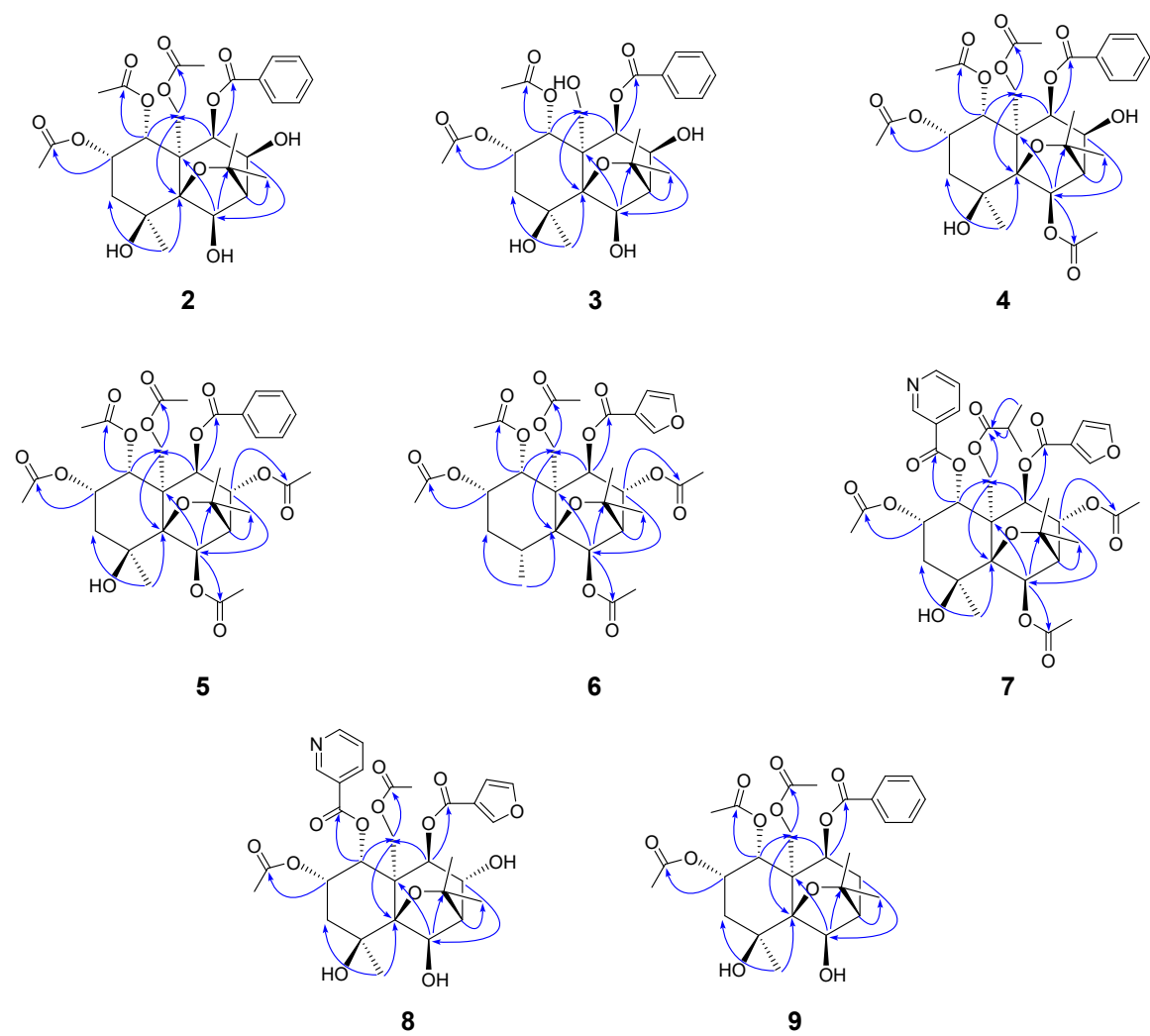


Figure S1 Key HMBC correlations of compounds **2-9**.

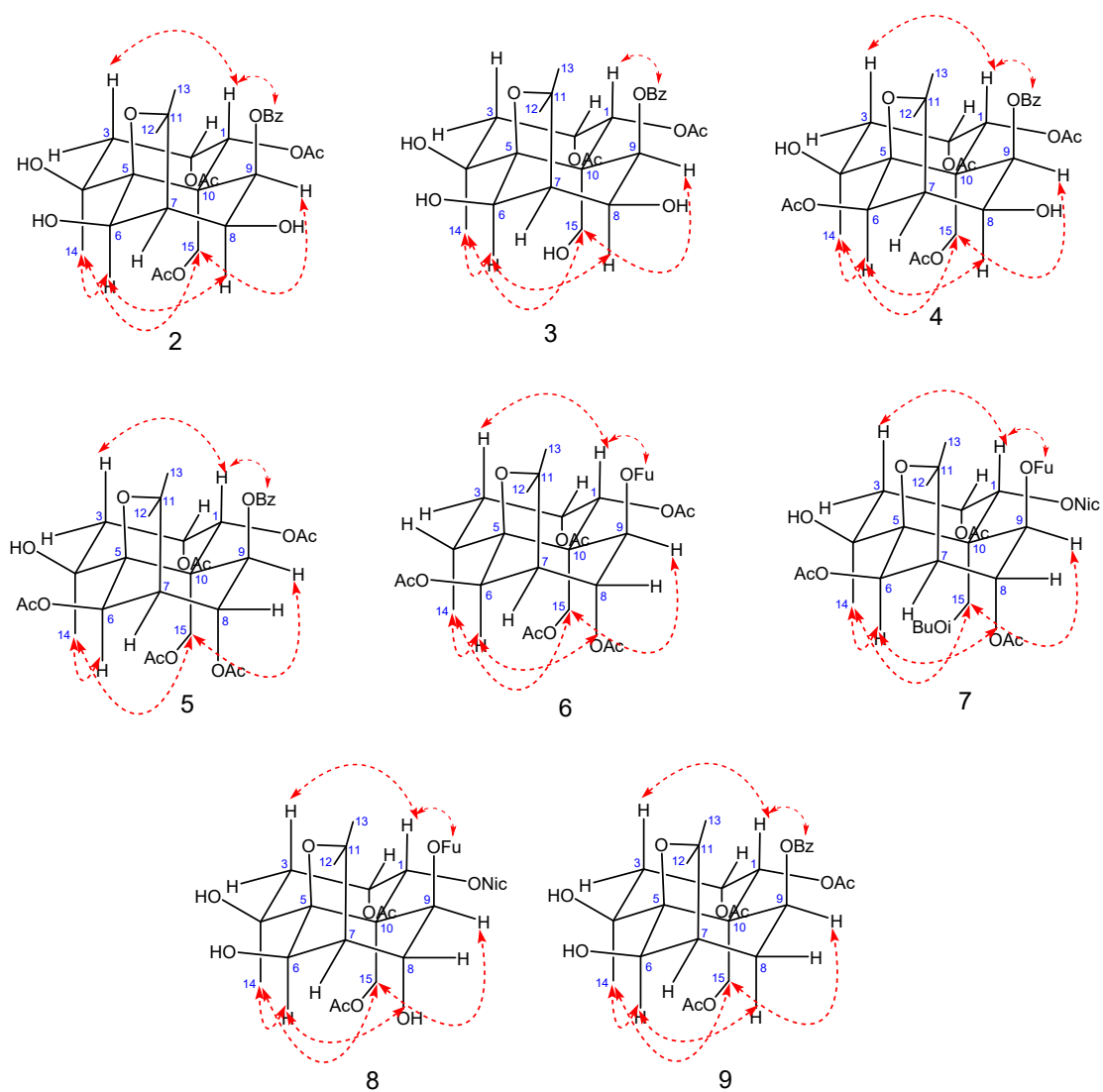


Figure S2 Key NOESY correlations of compounds **2-9**.

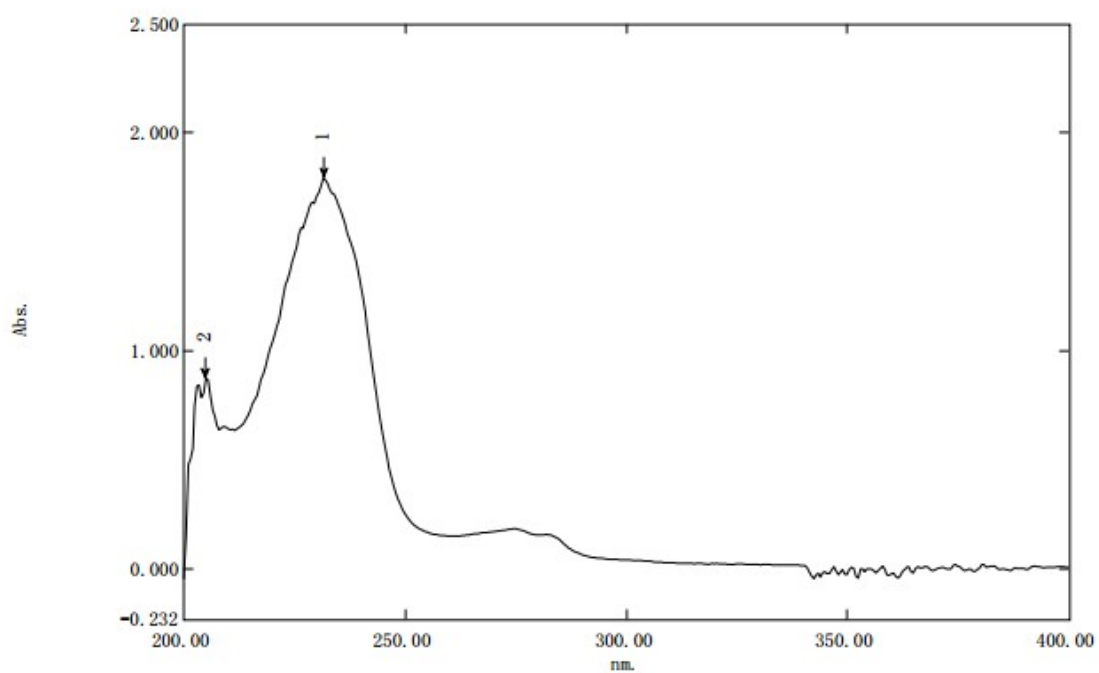


Figure S3 UV spectrum of compound **1**

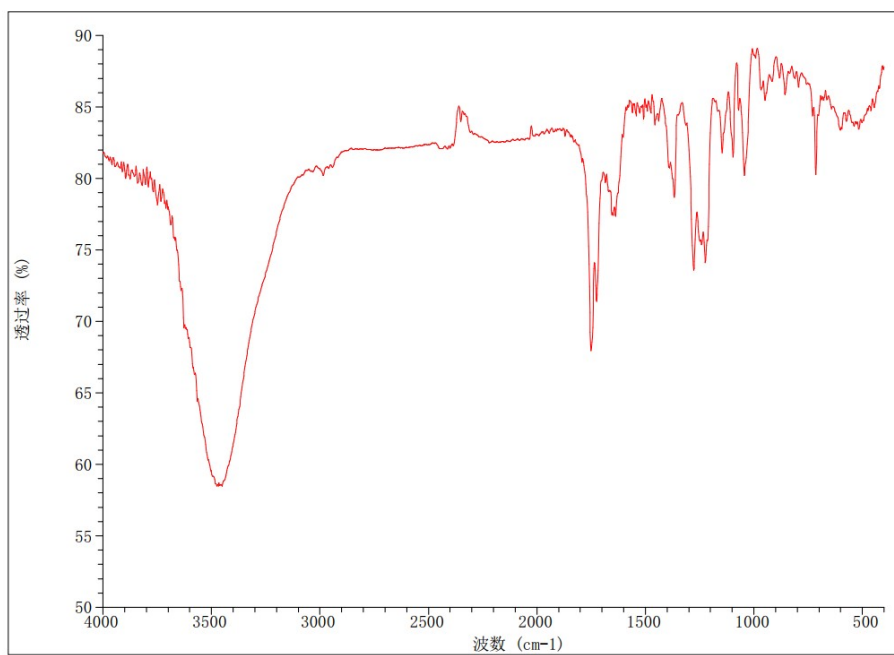


Figure S4 IR spectrum of compound **1**

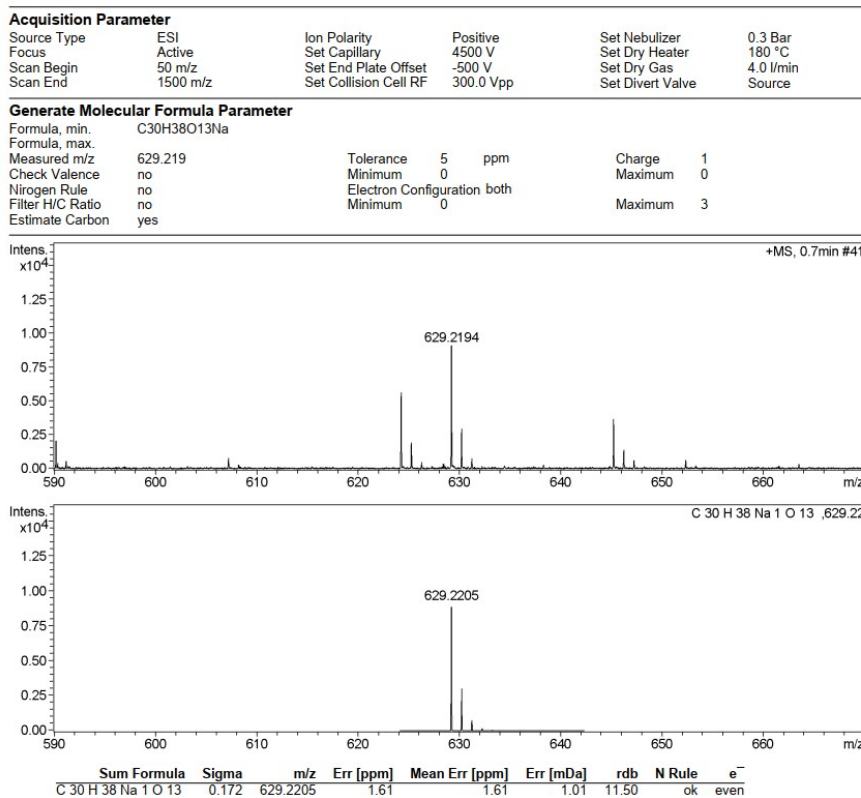


Figure S5 HRESIMS spectrum of compound 1

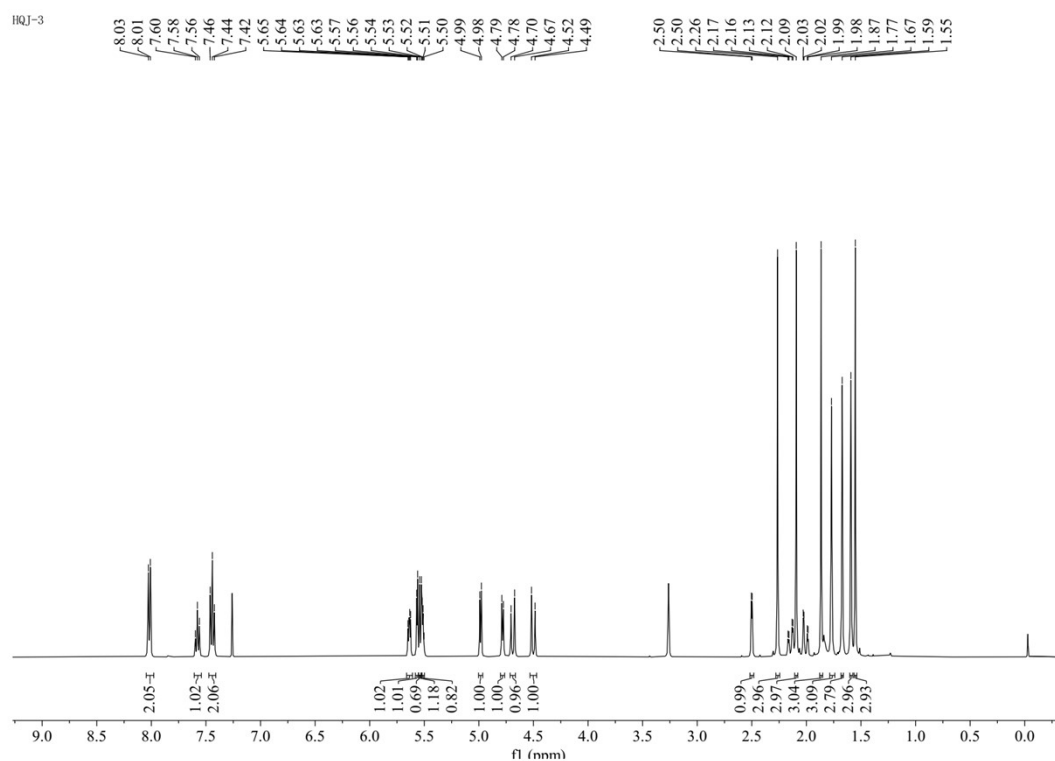


Figure S6 ¹H NMR spectrum (400 MHz, CDCl₃) of compound 1

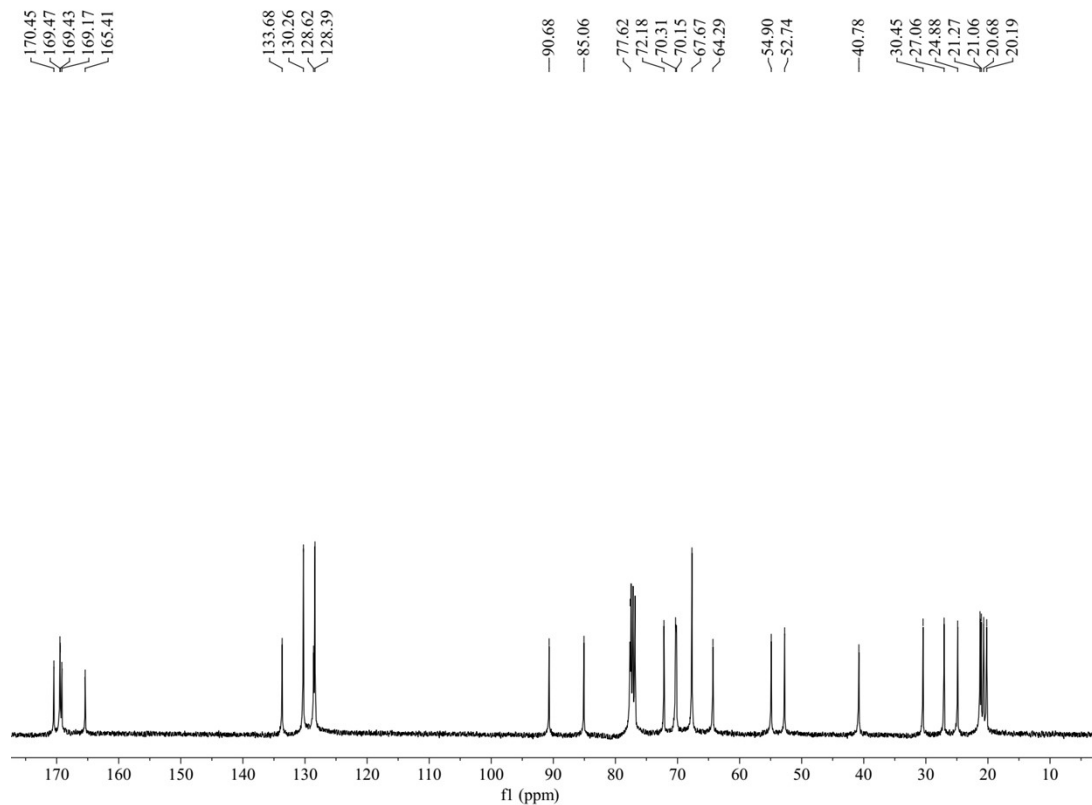


Figure S7 ^{13}C NMR spectrum (100 MHz, CDCl_3) of compound **1**

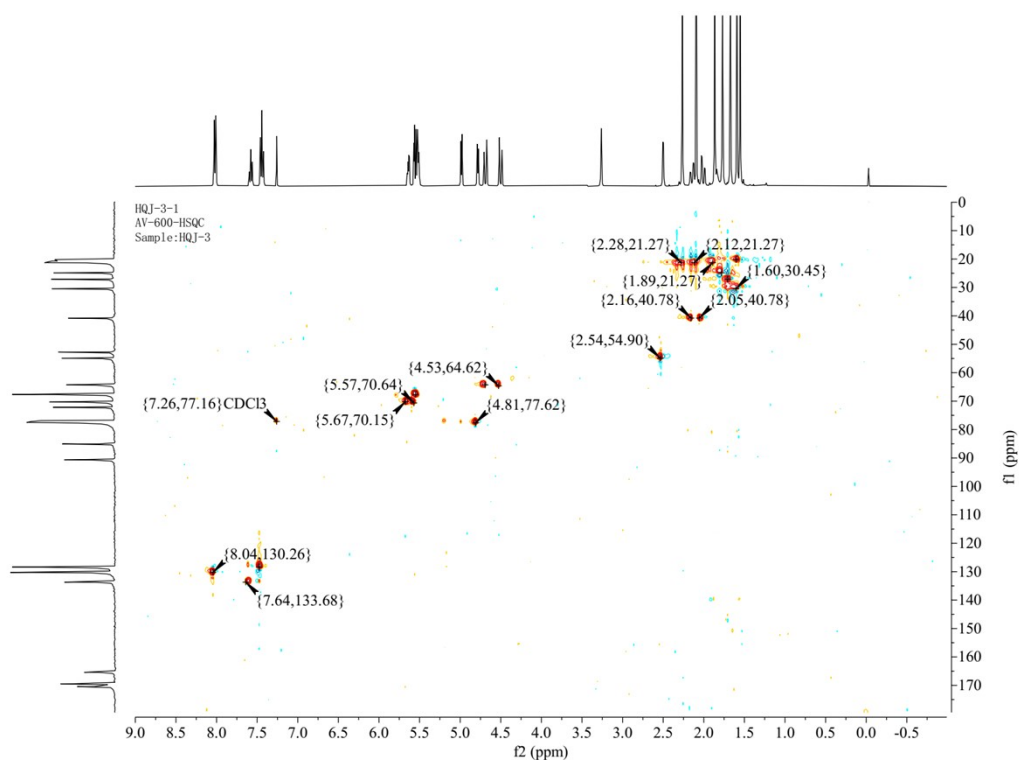


Figure S8 HSQC spectrum (600 MHz, CDCl_3) of compound **1**

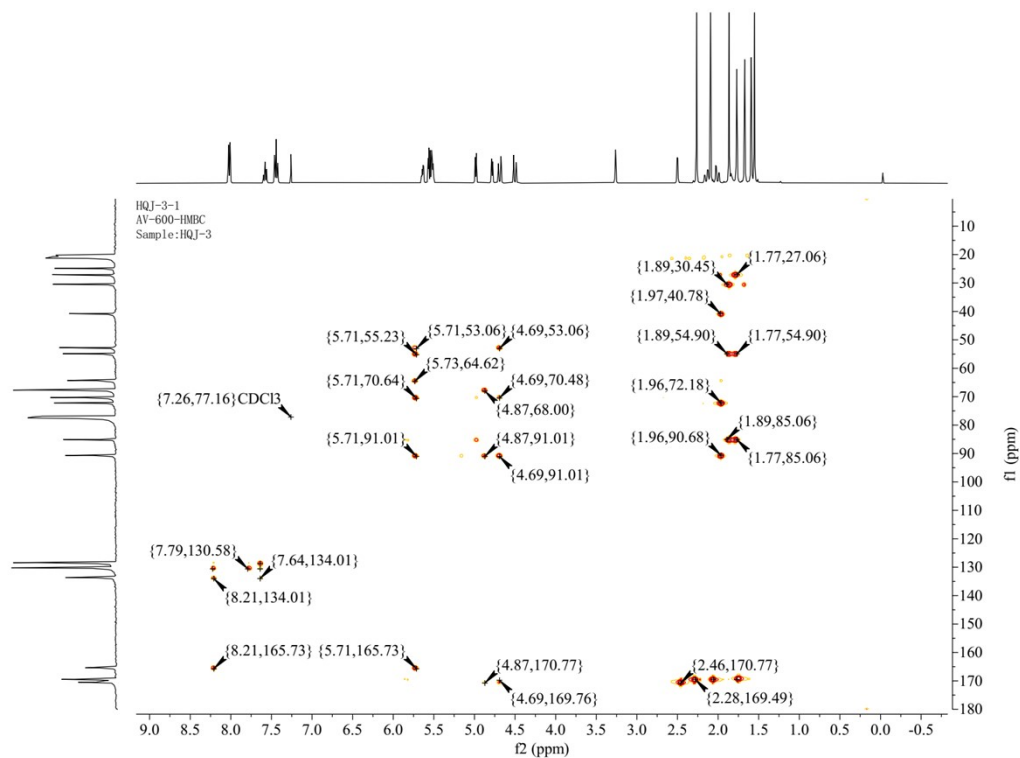


Figure S9 HMBC spectrum (600 MHz, CDCl₃) of compound 1

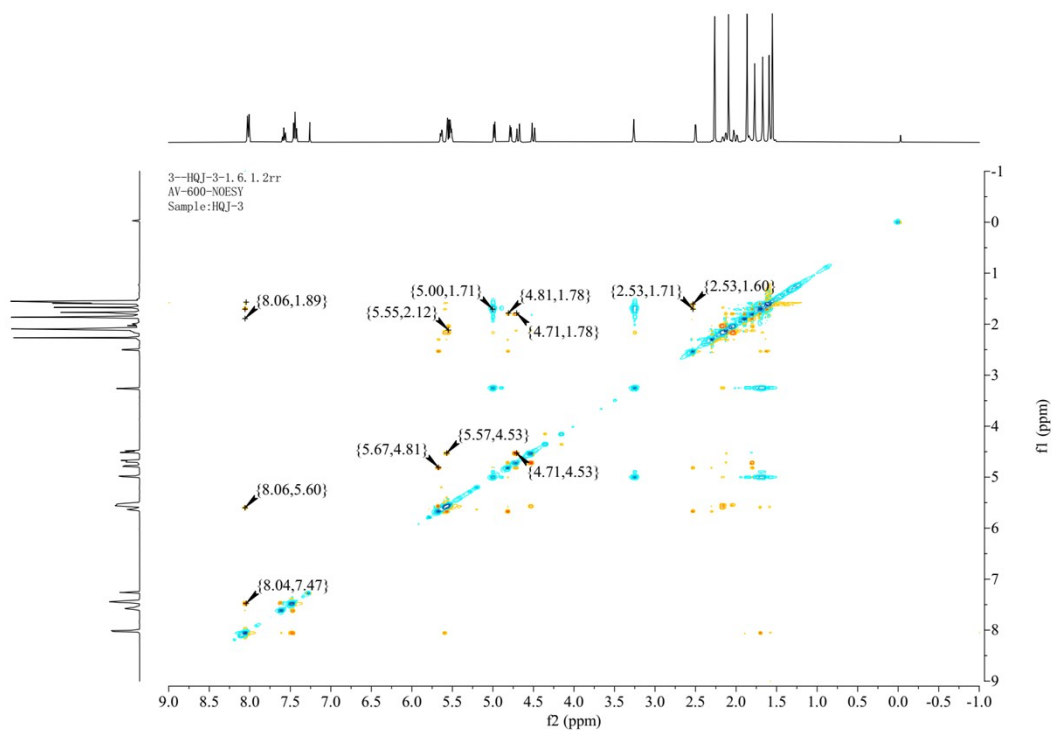


Figure S10 NOESY spectrum (600 MHz, CDCl₃) of compound 1

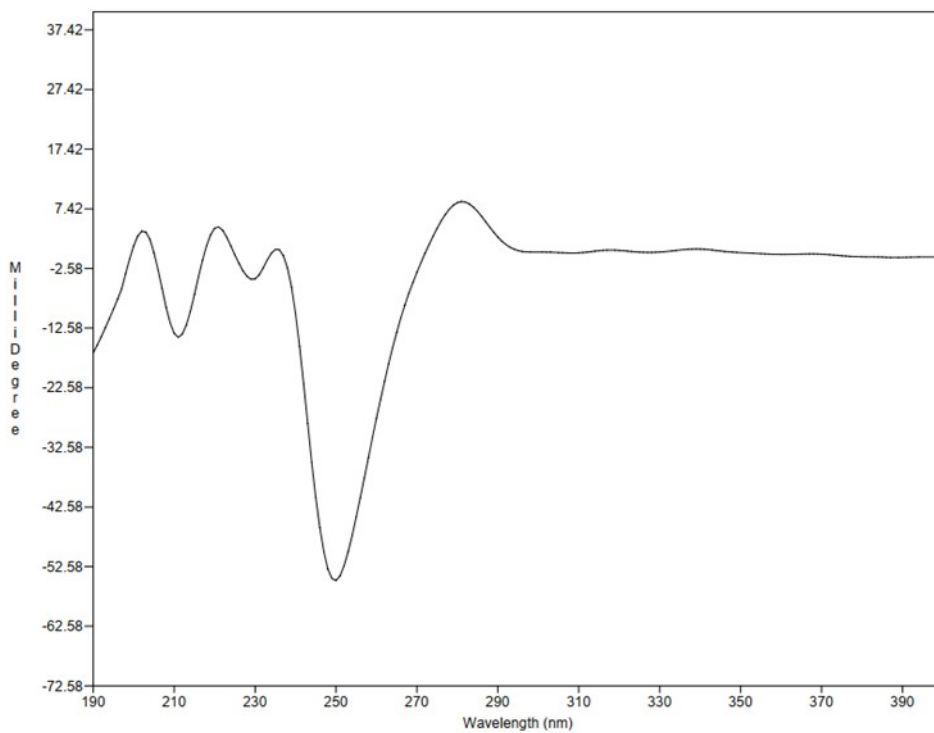


Figure S11 Experimental ECD spectrum of compound **1**

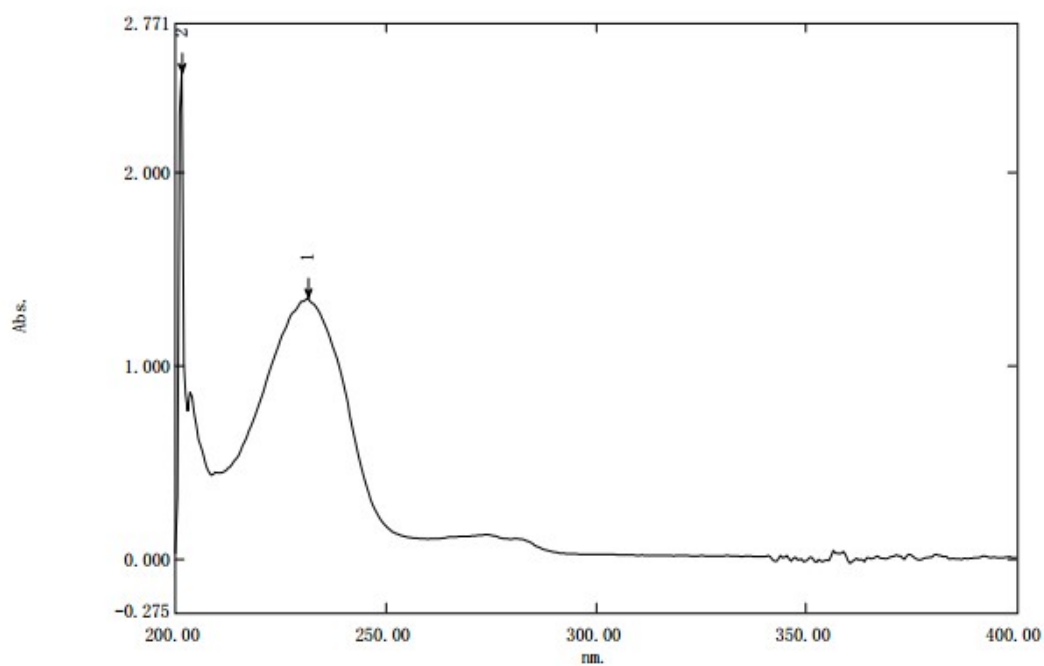


Figure S12 UV spectrum of compound **2**

| Acquisition Parameter | | | | | |
|-----------------------|----------|-----------------------|-----------|------------------|-----------|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 1.2 Bar |
| Focus | Active | Set Capillary | 4500 V | Set Dry Heater | 180 °C |
| Scan Begin | 50 m/z | Set End Plate Offset | -500 V | Set Dry Gas | 8.0 l/min |
| Scan End | 1500 m/z | Set Collision Cell RF | 400.0 Vpp | Set Divert Valve | Source |

| Generate Molecular Formula Parameter | | |
|--------------------------------------|------------------------|---------|
| Formula, min. | | |
| Formula, max. | | |
| Measured m/z | Tolerance | Charge |
| Check Valence | Minimum | Maximum |
| Nitrogen Rule | Electron Configuration | |
| Filter H/C Ratio | Minimum | Maximum |
| Estimate Carbon | | |

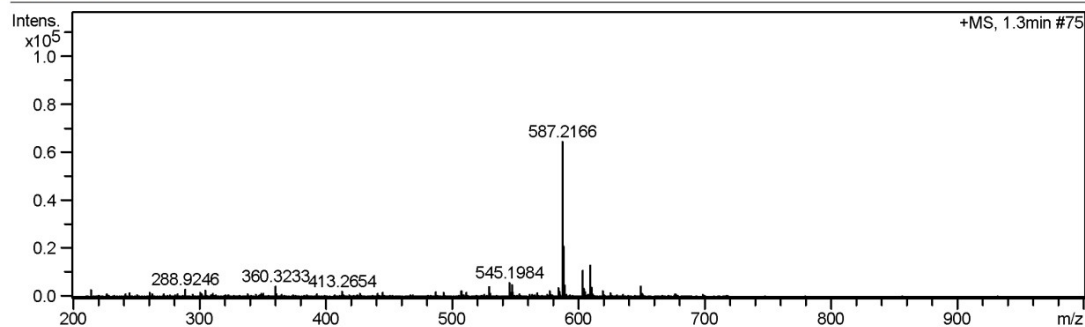


Figure S13 HRESIMS spectrum of compound **2**

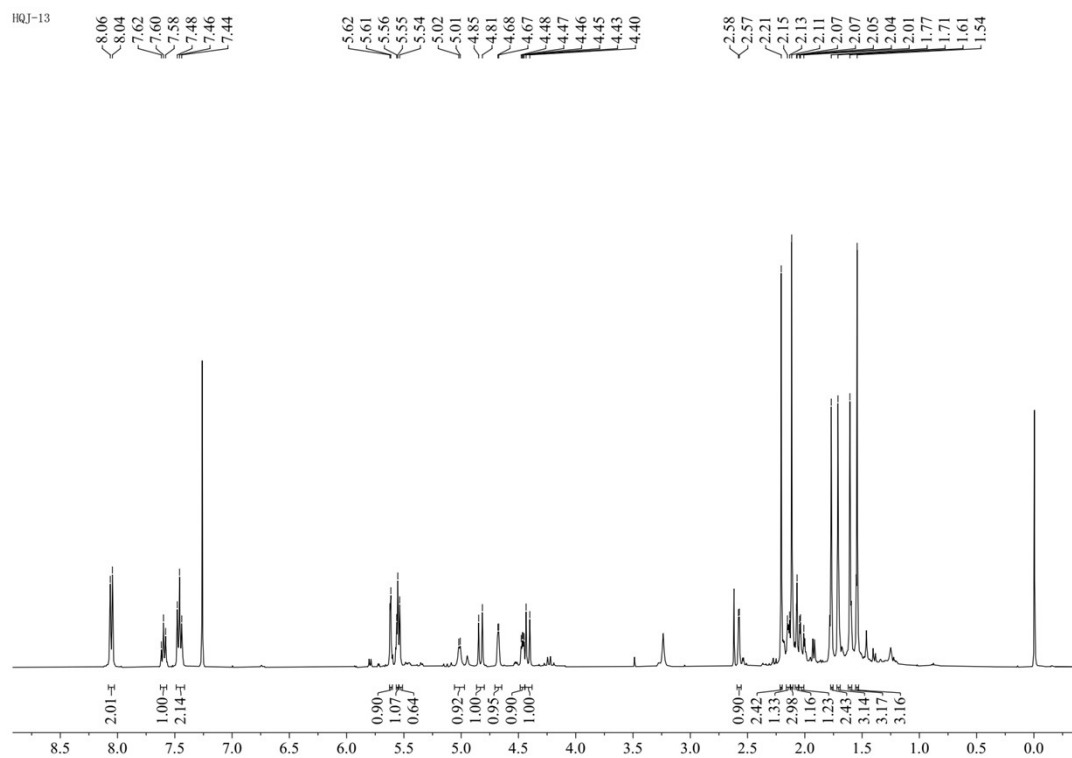


Figure S14 ¹H NMR spectrum (400 MHz, CDCl₃) of compound **2**

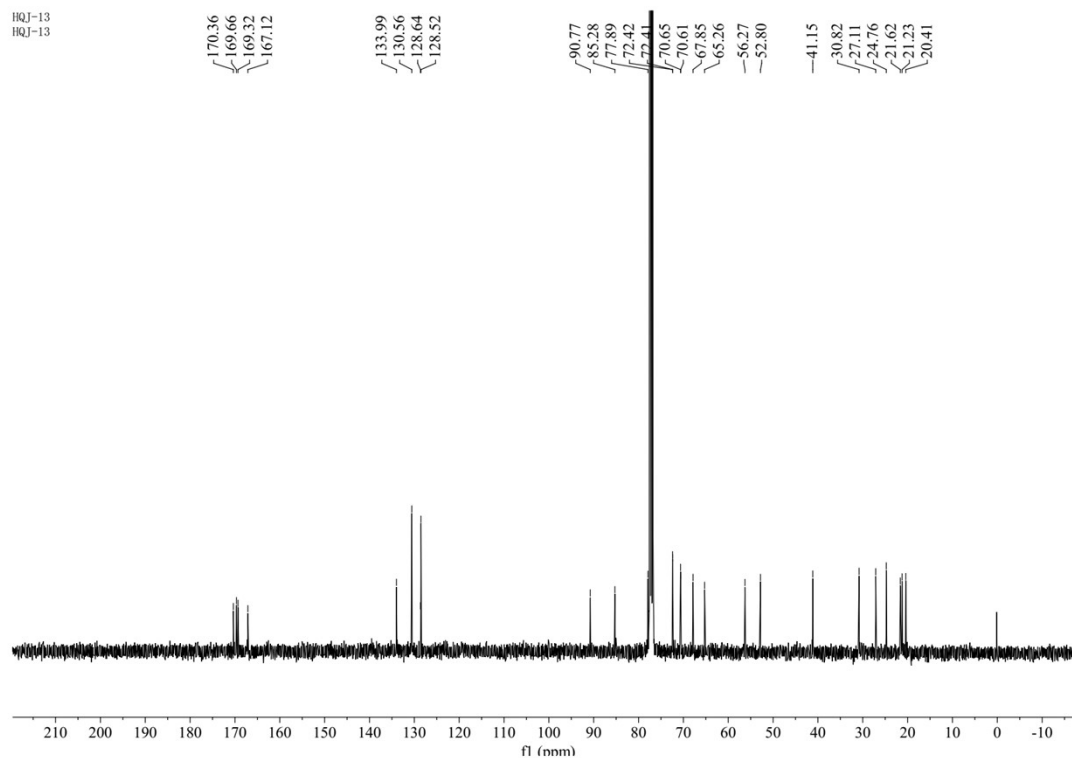


Figure S15 ^{13}C NMR spectrum (100 MHz, CDCl_3) of compound **2**

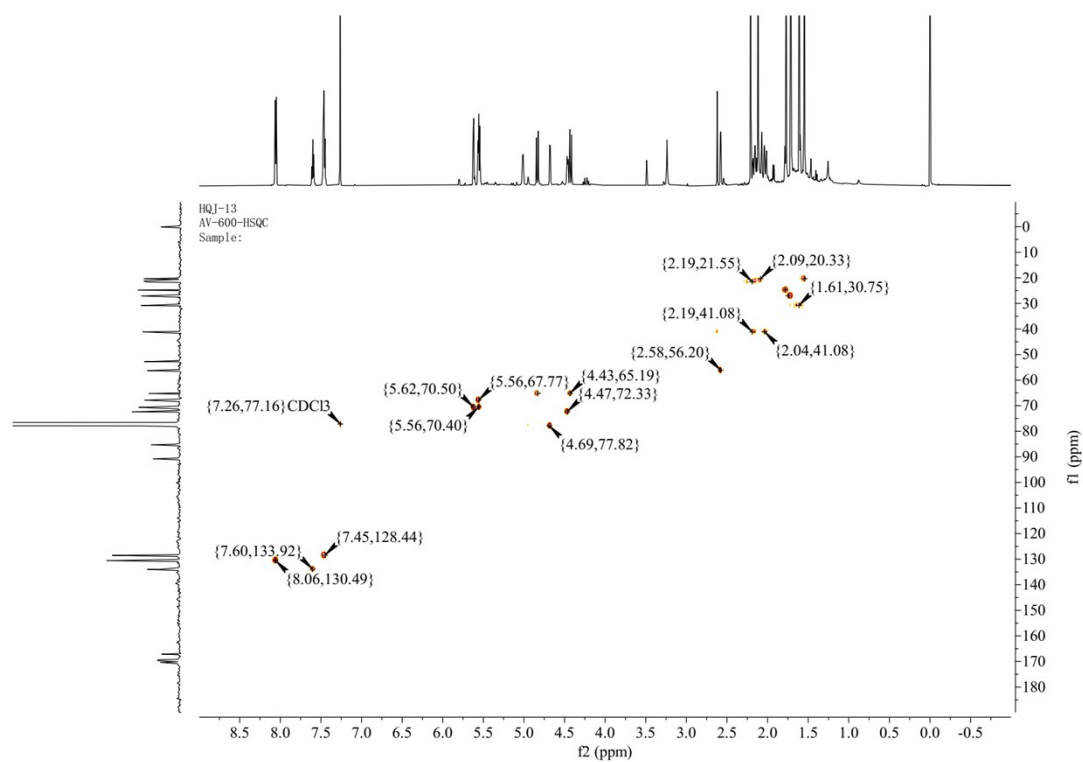


Figure S16 HSQC spectrum (600 MHz, CDCl_3) of compound **2**

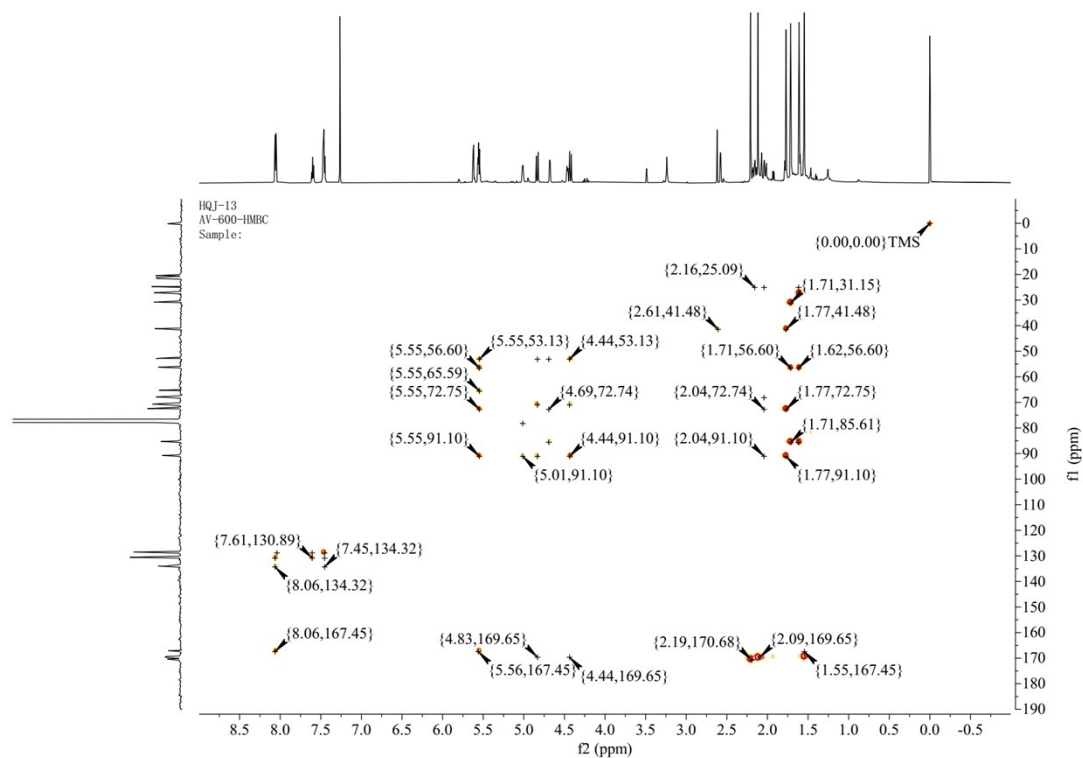


Figure S17 HMBC spectrum (600 MHz, CDCl₃) of compound **2**

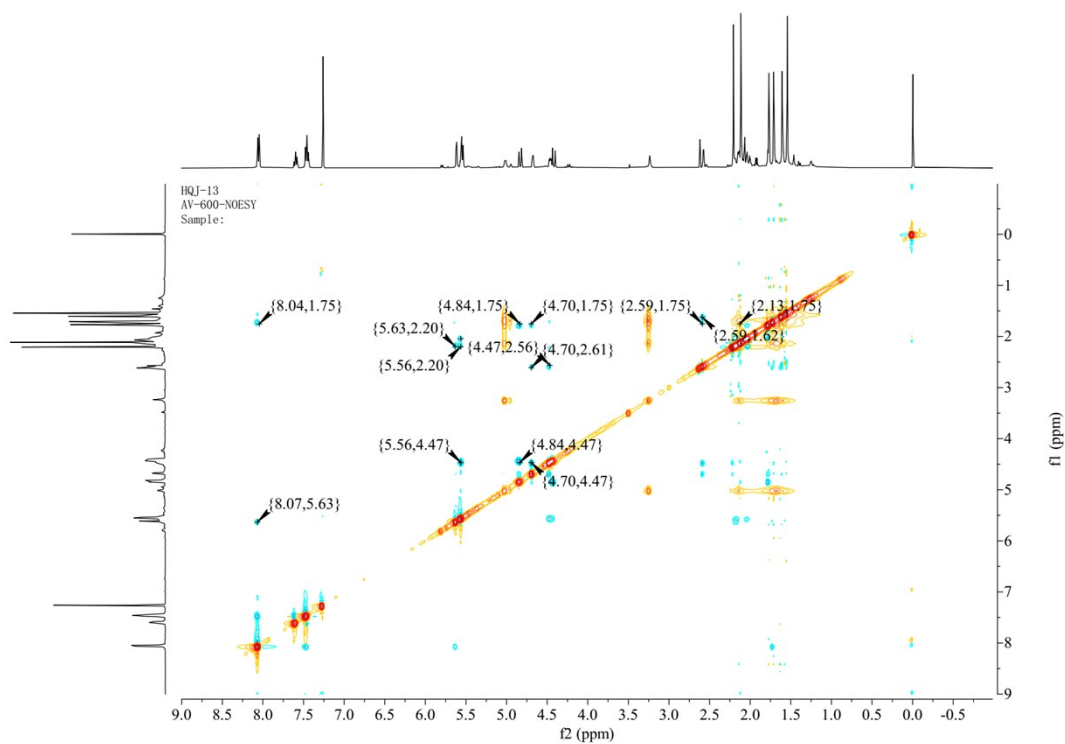


Figure S18 NOESY spectrum (600 MHz, CDCl₃) of compound **2**

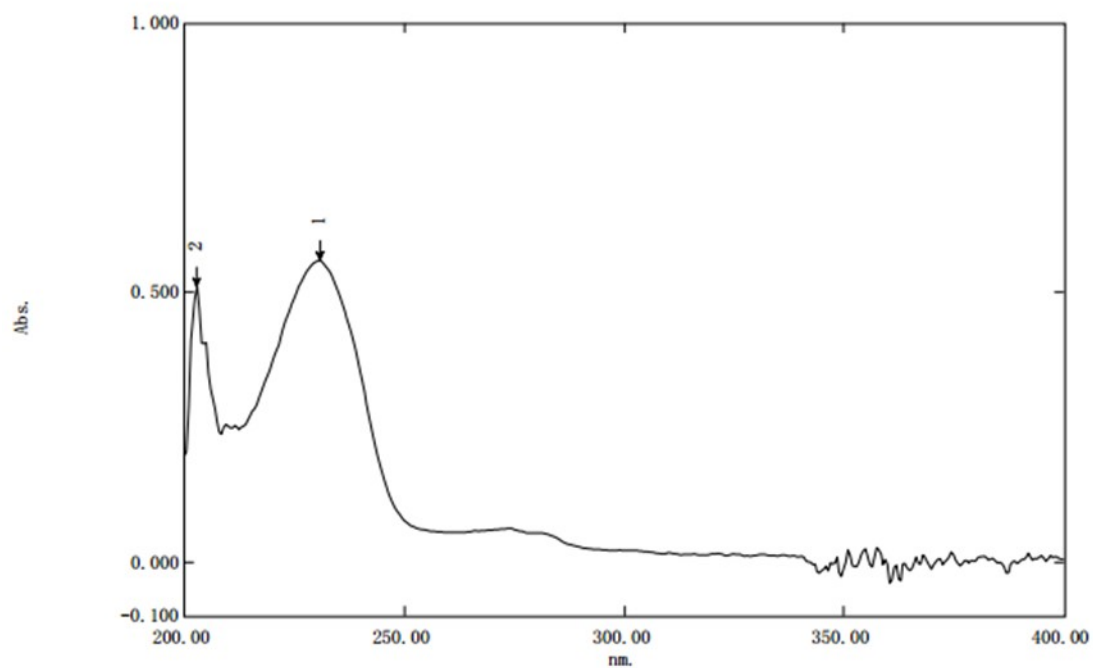


Figure S19 Experimental ECD spectrum of compound 2

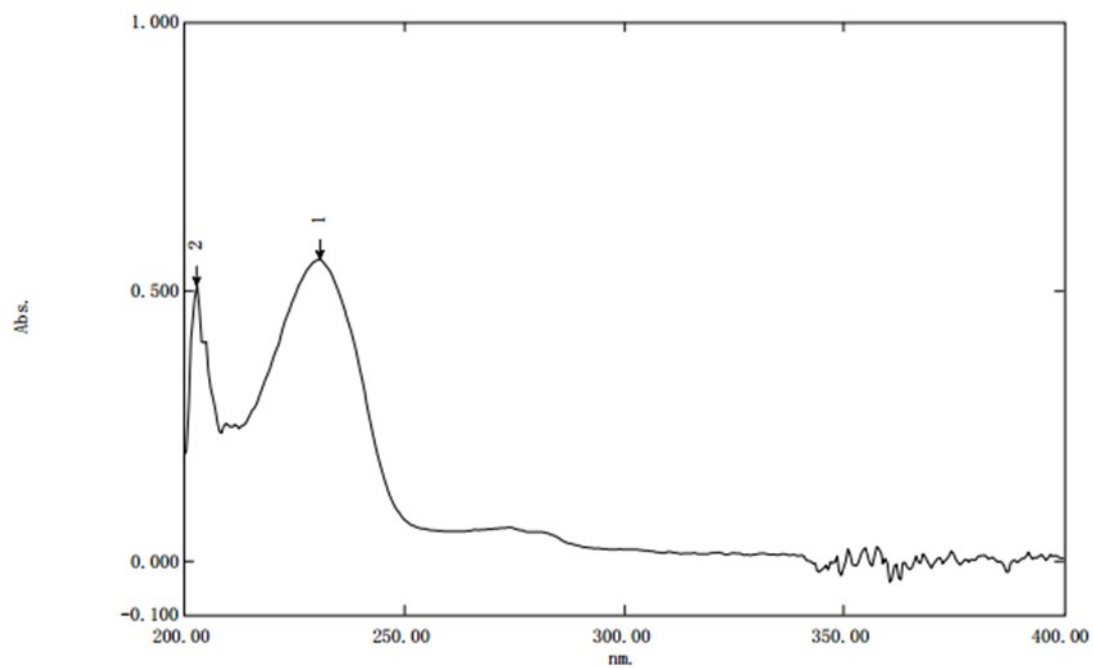


Figure S20 UV spectrum of compound 3

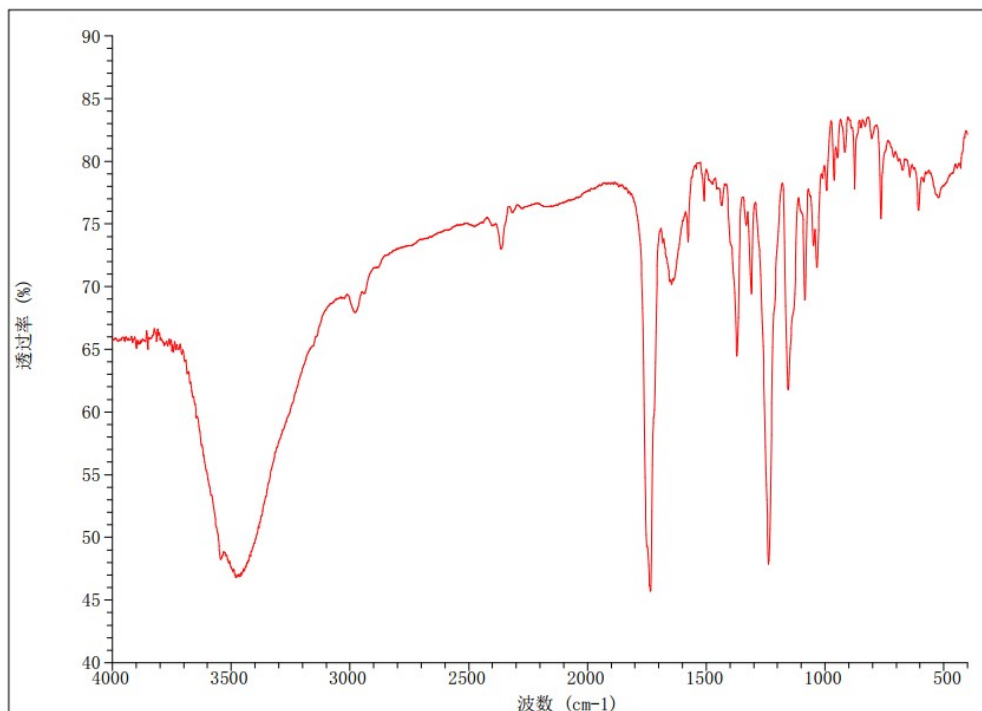


Figure S21 IR spectrum of compound **3**

Acquisition Parameter

| | | | | | |
|-------------|----------|-----------------------|-----------|------------------|-----------|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 1.2 Bar |
| Focus | Active | Set Capillary | 4500 V | Set Dry Heater | 180 °C |
| Scan Begin | 50 m/z | Set End Plate Offset | -500 V | Set Dry Gas | 8.0 l/min |
| Scan End | 1500 m/z | Set Collision Cell RF | 400.0 Vpp | Set Divert Valve | Source |

Generate Molecular Formula Parameter

| | | |
|------------------|------------------------|---------|
| Formula, min. | | |
| Formula, max. | | |
| Measured m/z | Tolerance | Charge |
| Check Valence | Minimum | Maximum |
| Nitrogen Rule | Electron Configuration | |
| Filter H/C Ratio | Minimum | Maximum |
| Estimate Carbon | | |

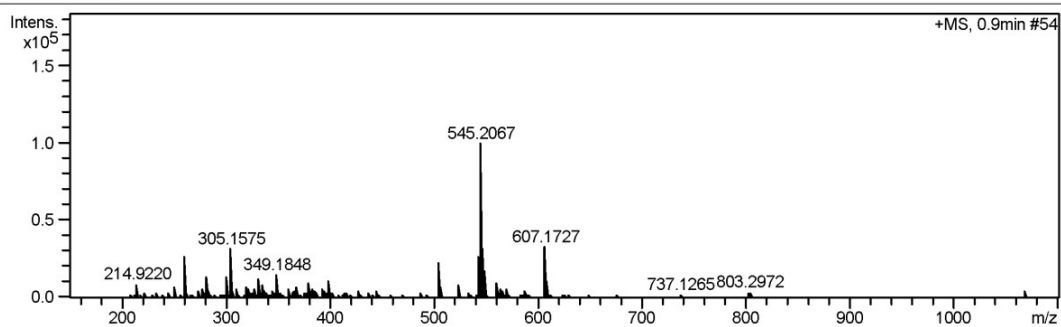


Figure S22 HRESIMS spectrum of compound **3**

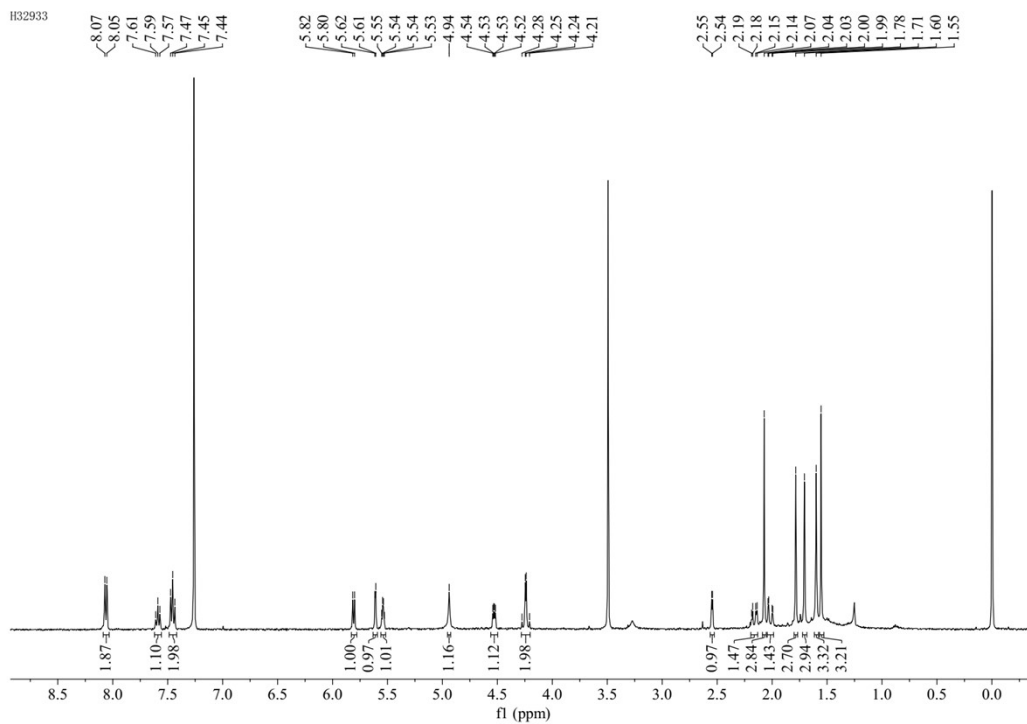


Figure S23 ^1H NMR spectrum (400 MHz, CDCl_3) of compound **3**

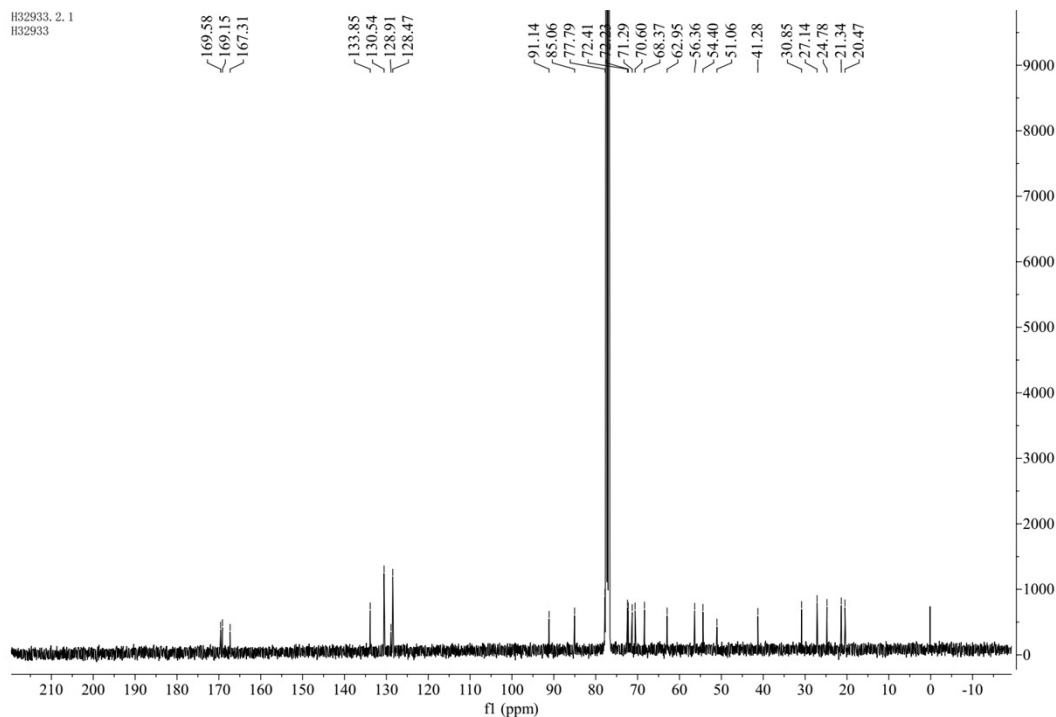


Figure S24 ^{13}C NMR spectrum (100 MHz, CDCl_3) of compound **3**

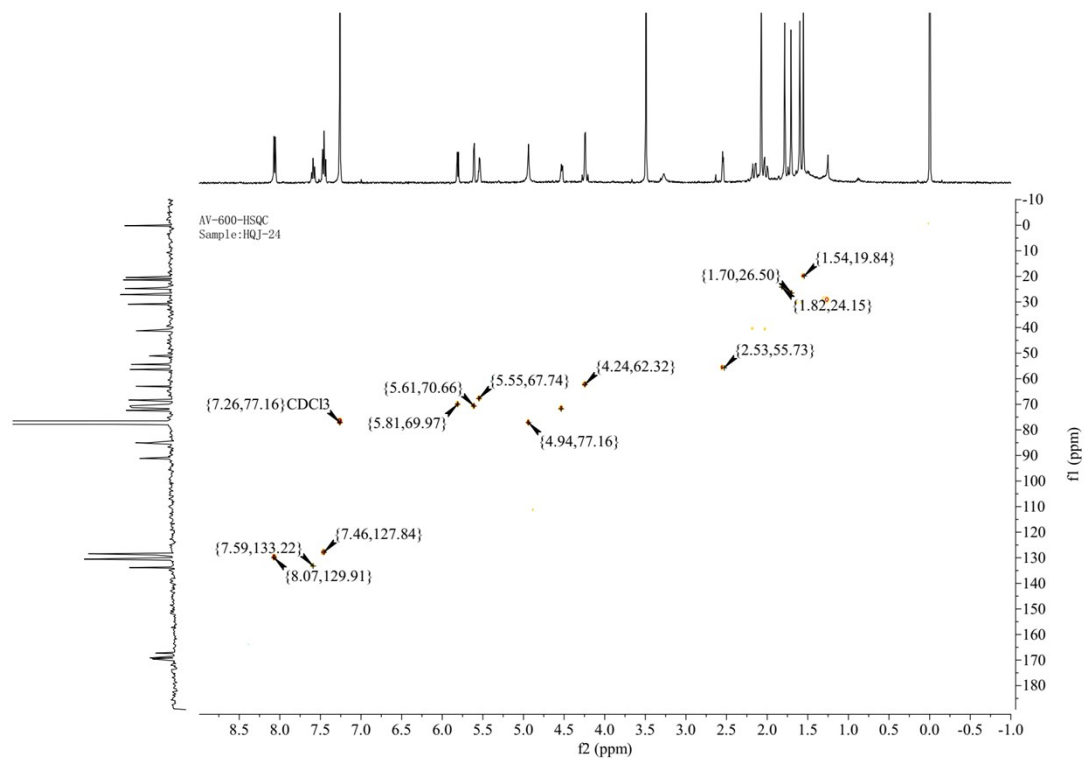


Figure S25 HSQC spectrum (600 MHz, CDCl₃) of compound **3**

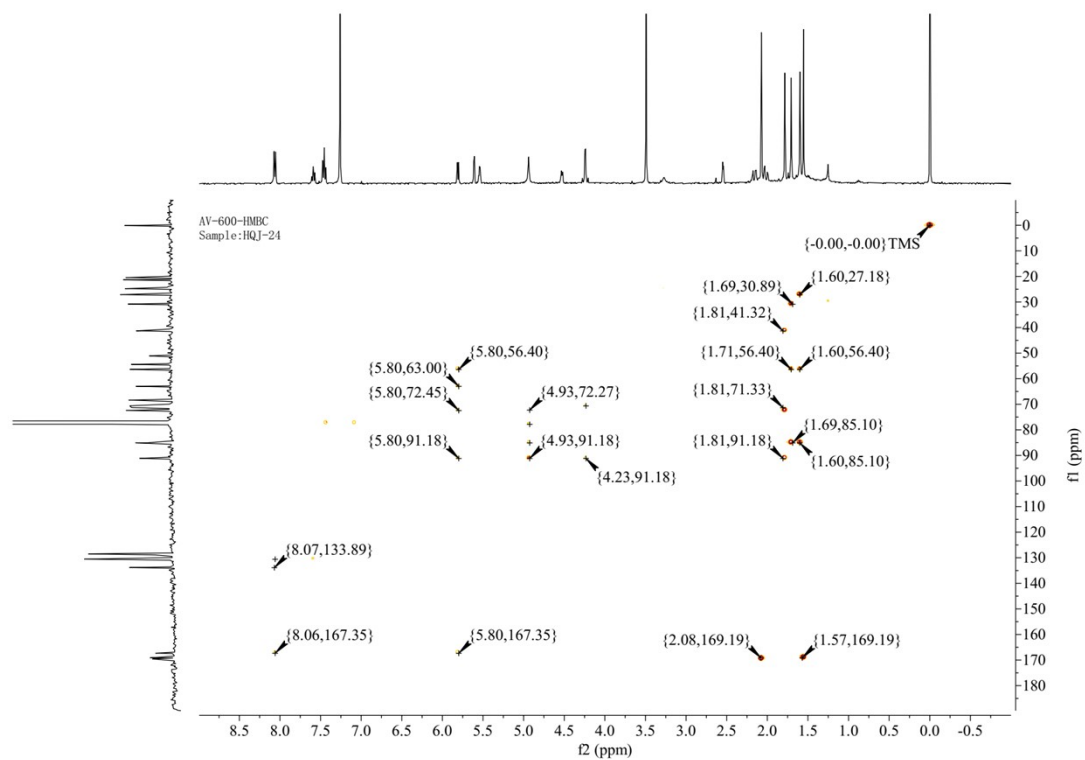


Figure S26 HMBC spectrum (600 MHz, CDCl₃) of compound **3**

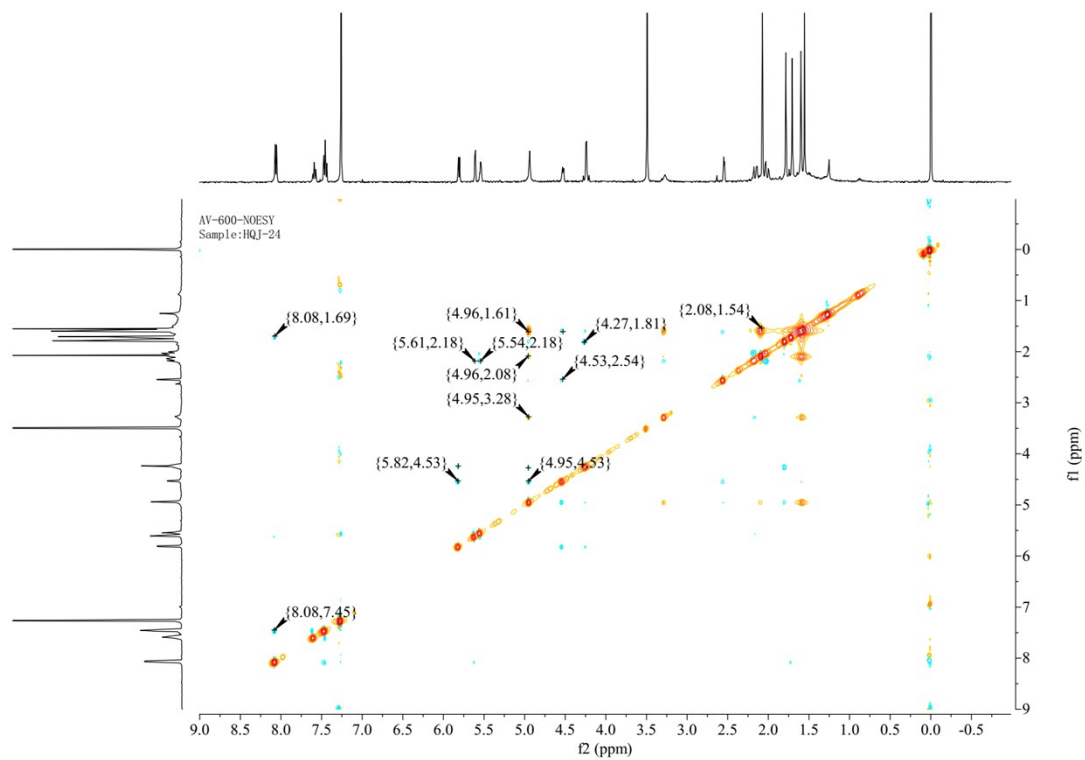


Figure S27 NOESY spectrum (600 MHz, CDCl_3) of compound **3**

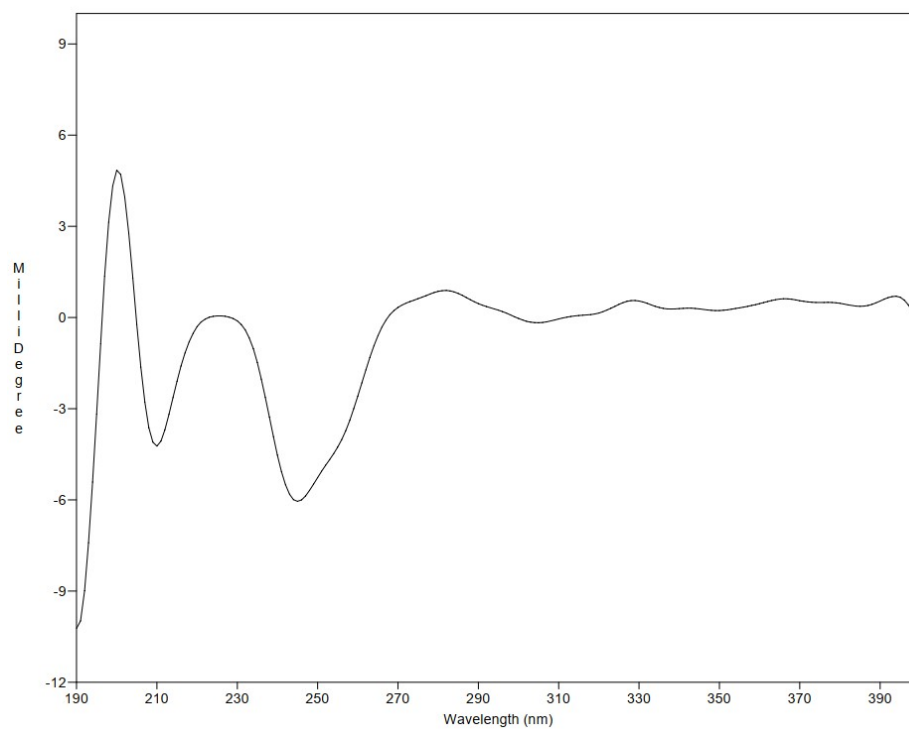


Figure S28 Experimental ECD spectrum of compound **3**

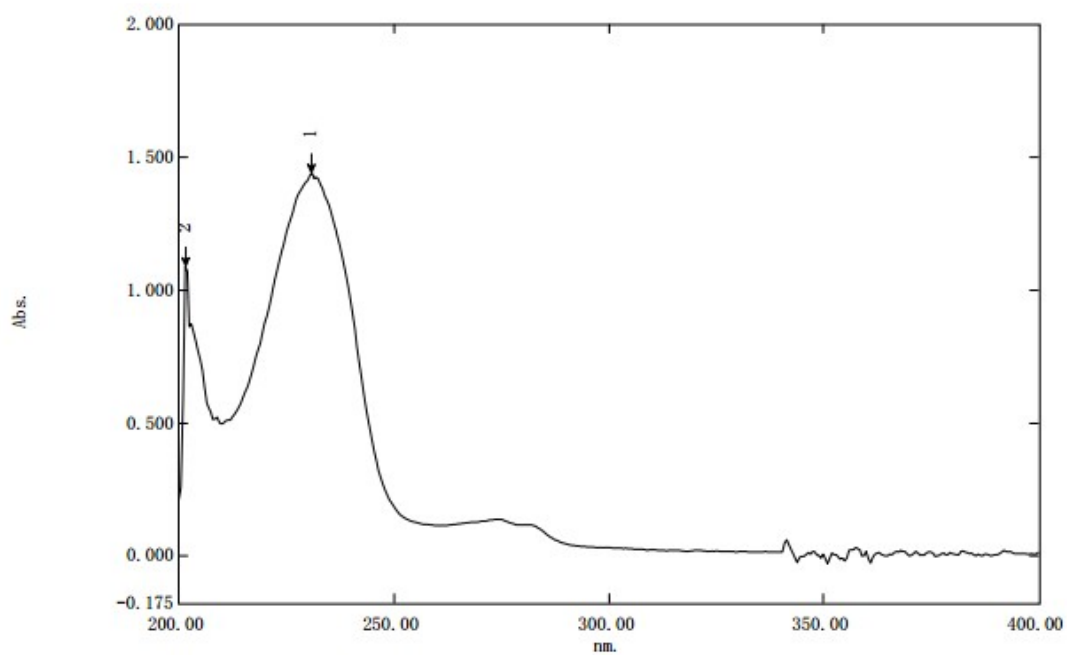


Figure S29 UV spectrum of compound 4

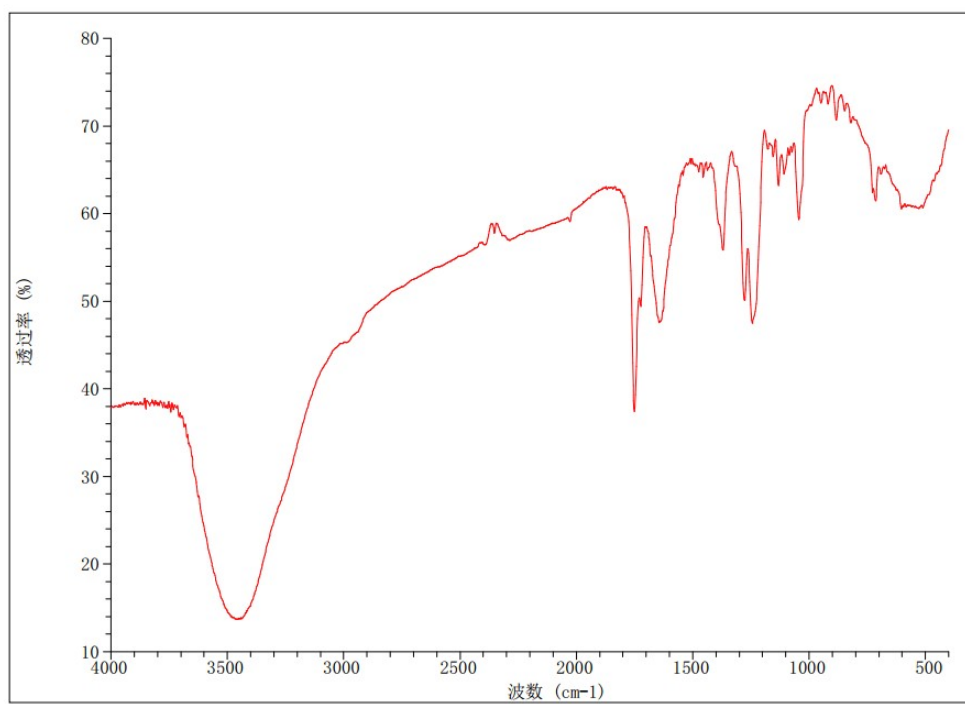


Figure S30 IR spectrum of compound 4

| Acquisition Parameter | | | | | |
|--------------------------------------|----------|------------------------|-----------|------------------|-----------|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 1.2 Bar |
| Focus | Active | Set Capillary | 4500 V | Set Dry Heater | 180 °C |
| Scan Begin | 50 m/z | Set End Plate Offset | -500 V | Set Dry Gas | 8.0 l/min |
| Scan End | 1500 m/z | Set Collision Cell RF | 300.0 Vpp | Set Divert Valve | Source |
| Generate Molecular Formula Parameter | | | | | |
| Formula, min. | | Tolerance | | Charge | |
| Formula, max. | | Minimum | | Maximum | |
| Measured m/z | | Electron Configuration | | | |
| Check Valence | | Minimum | | Maximum | |
| Nitrogen Rule | | | | | |
| Filter H/C Ratio | | | | | |
| Estimate Carbon | | | | | |

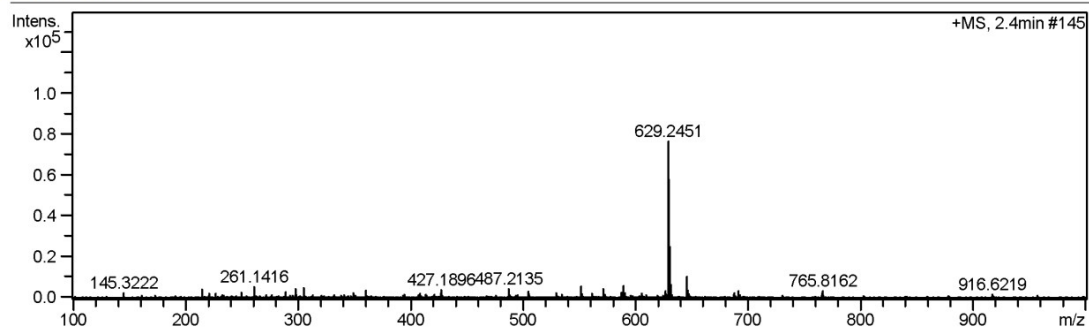


Figure S31 HRESIMS spectrum of compound 4

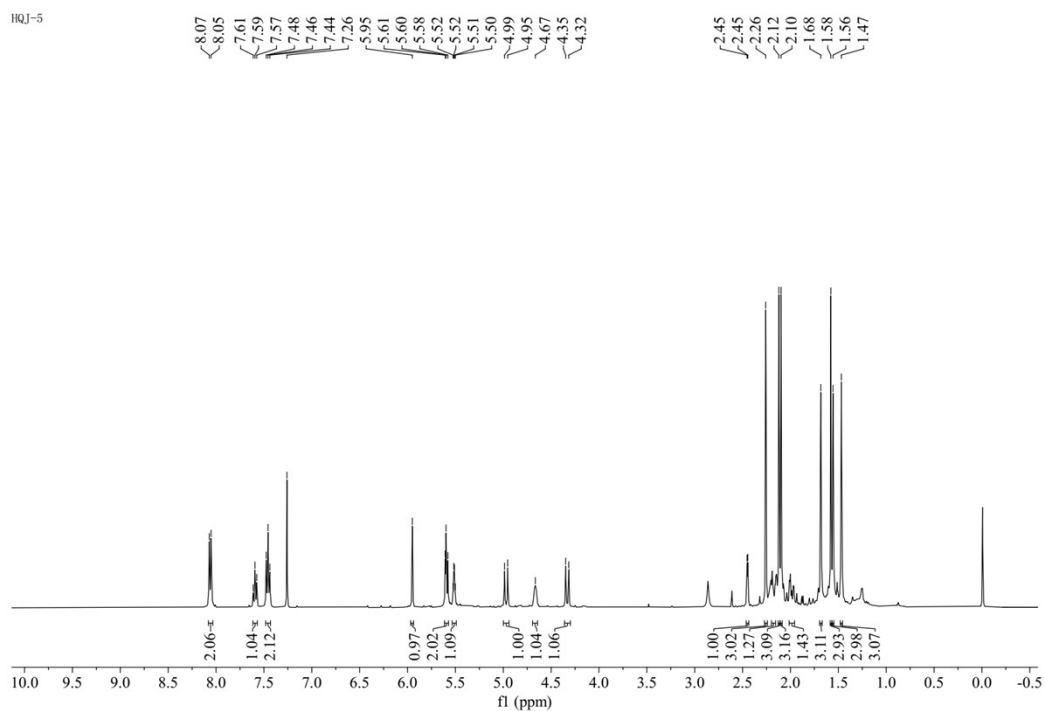


Figure S32 ¹H NMR spectrum (400 MHz, CDCl₃) of compound 4

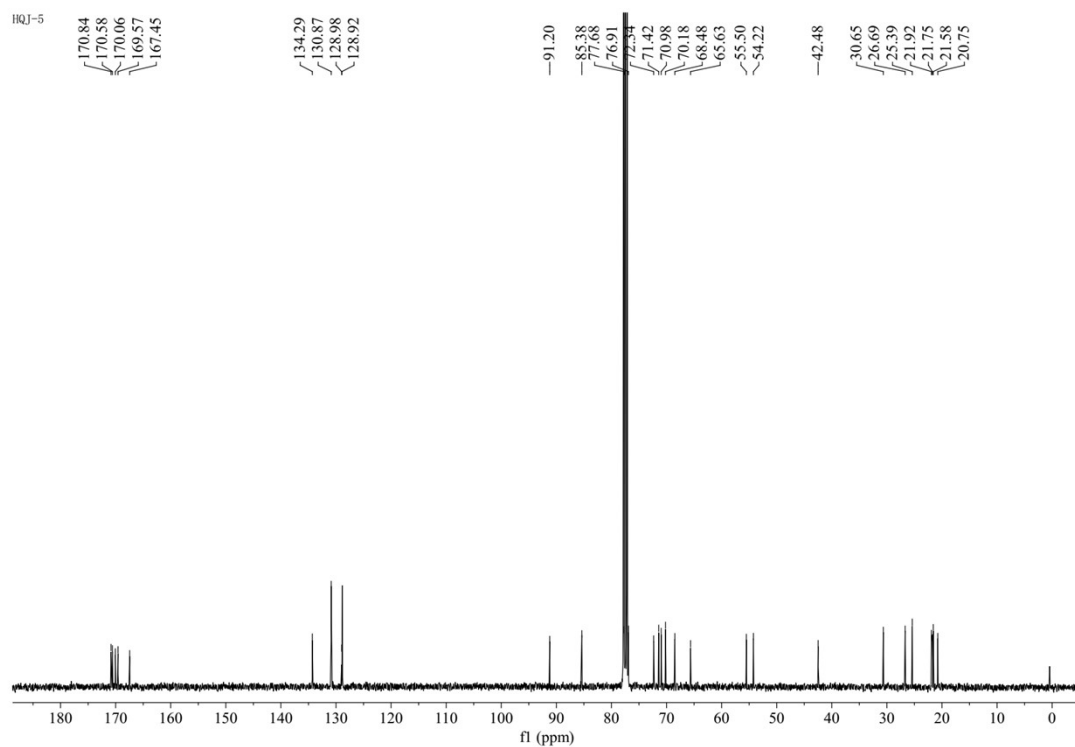


Figure S33 ^{13}C NMR spectrum (100 MHz, CDCl_3) of compound 4

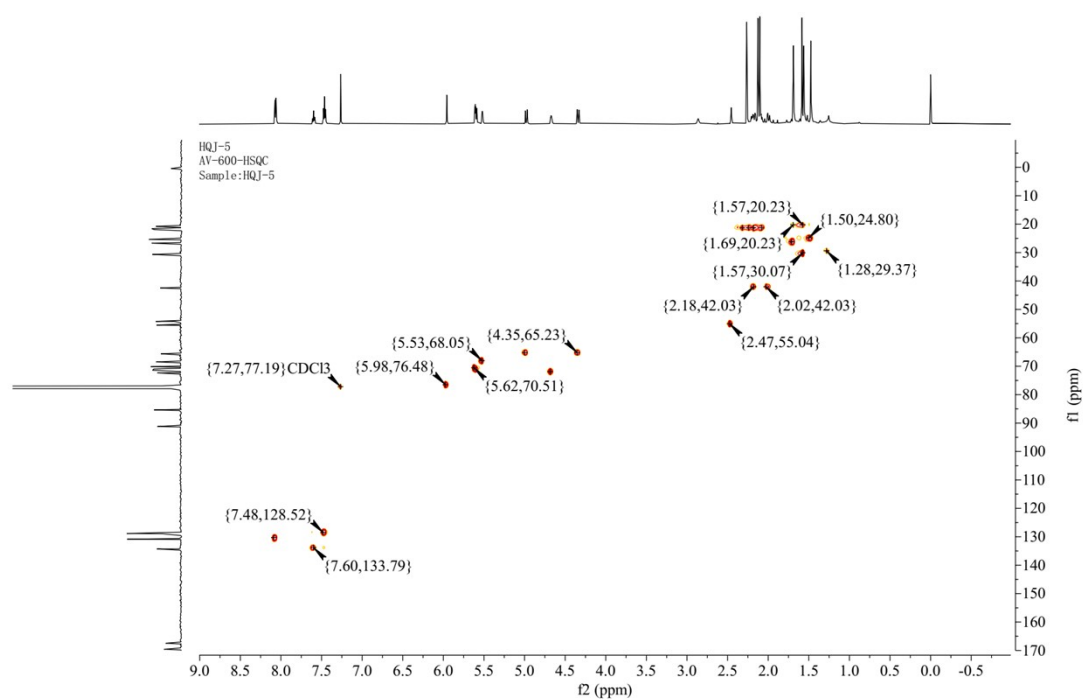


Figure S34 HSQC spectrum (600 MHz, CDCl_3) of compound 4

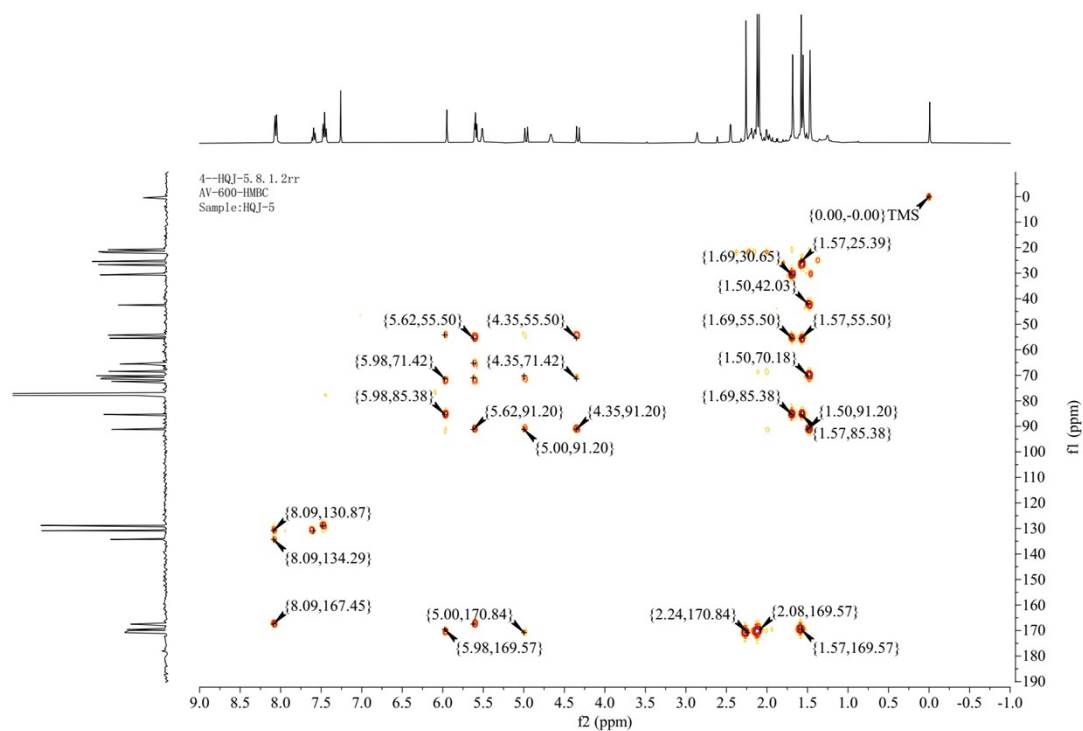


Figure S35 HMBC spectrum (600 MHz, CDCl_3) of compound 4

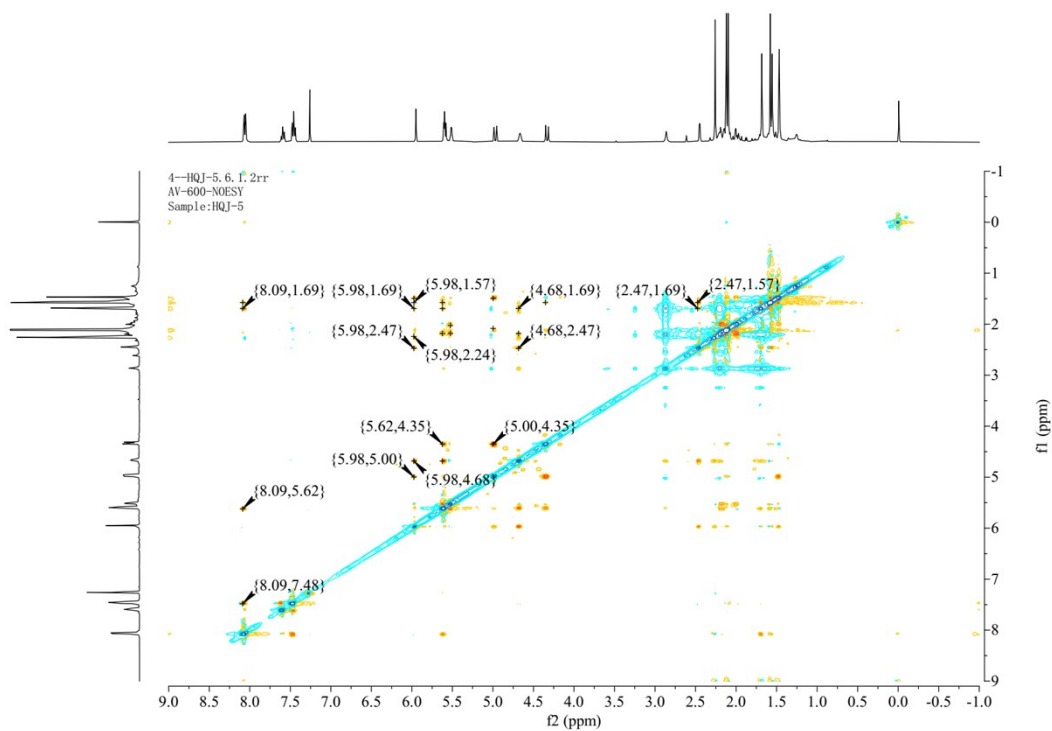


Figure S36 NOESY spectrum (600 MHz, CDCl_3) of compound 4

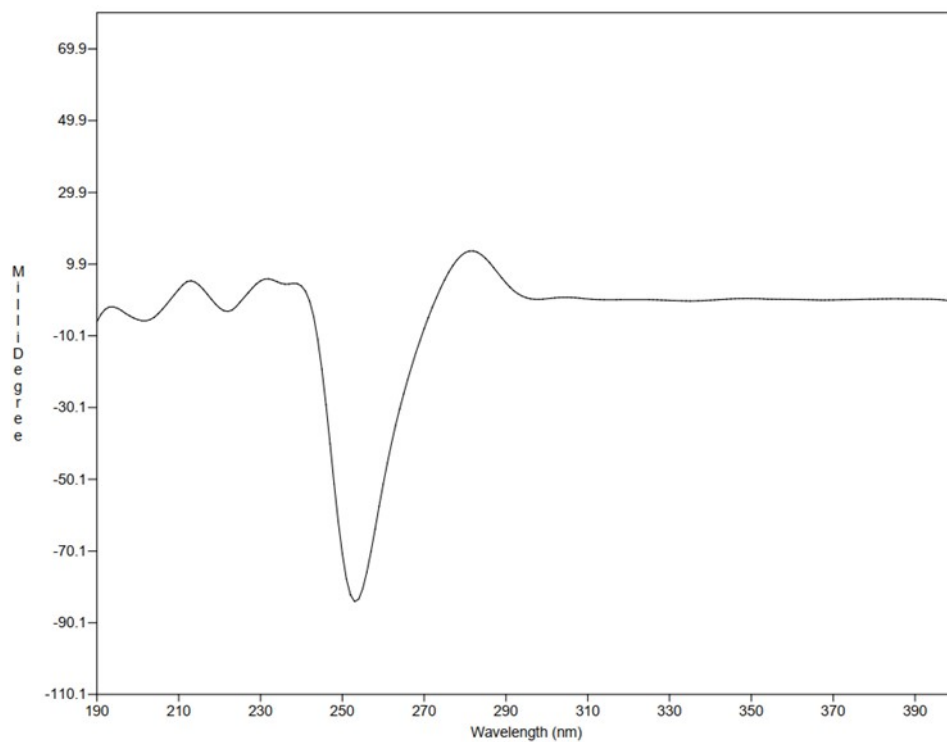


Figure S37 Experimental ECD spectrum of compound 4

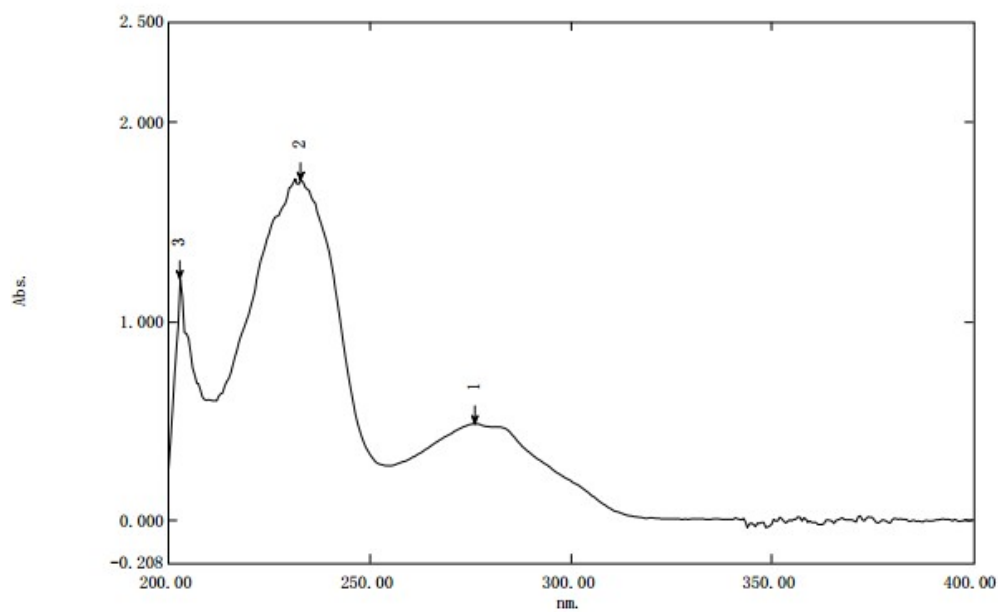


Figure S38 UV spectrum of compound 5

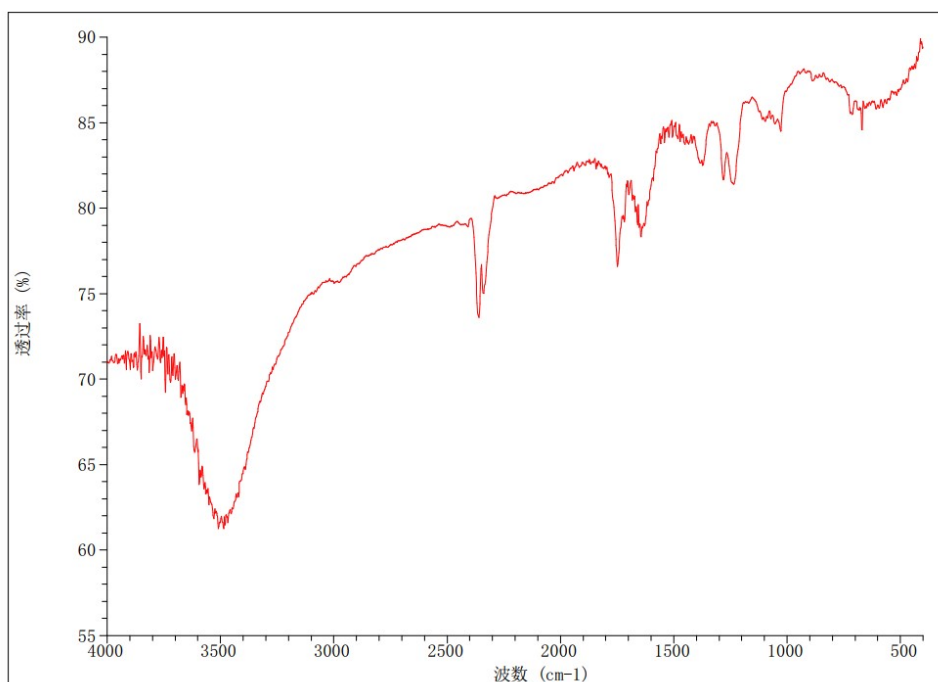


Figure S39 IR spectrum of compound 5

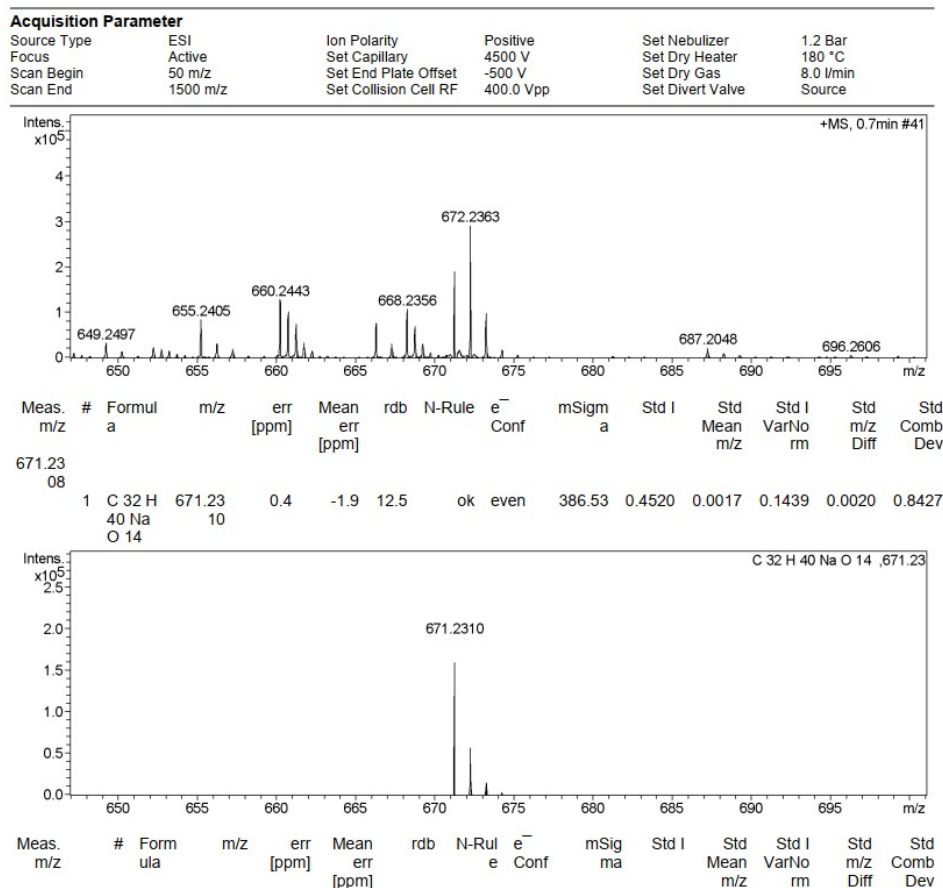


Figure S40 HRESIMS spectrum of compound 5

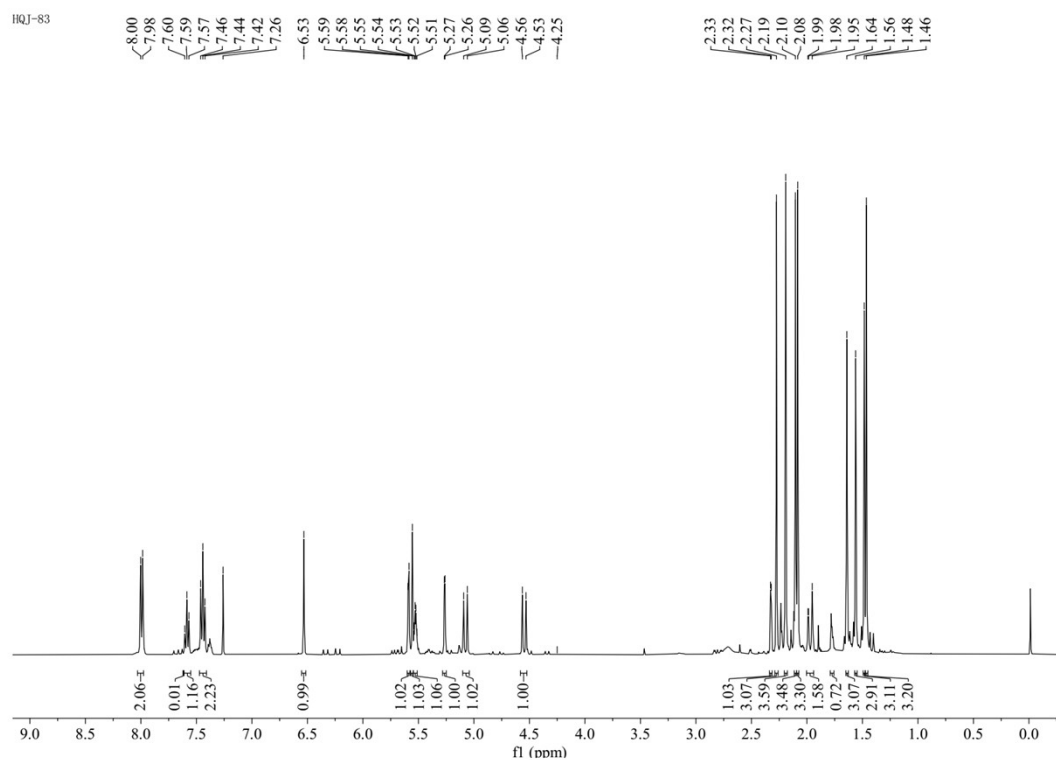


Figure S41 ^1H NMR spectrum (400 MHz, CDCl_3) of compound **5**

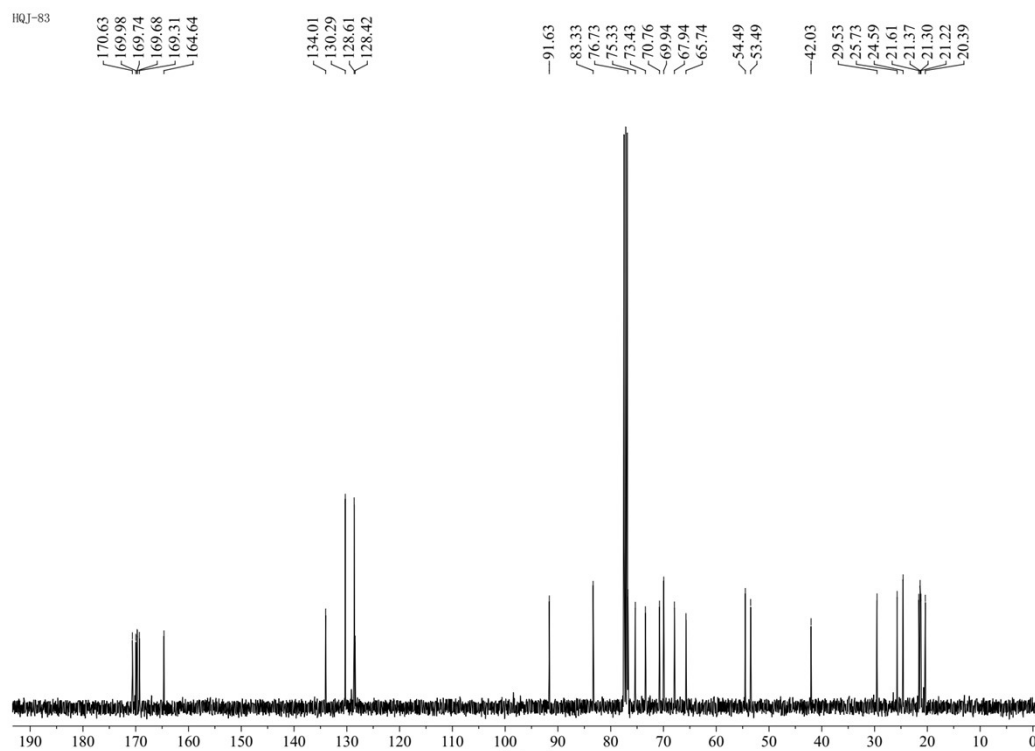


Figure S42 ^{13}C NMR spectrum (100 MHz, CDCl_3) of compound **5**

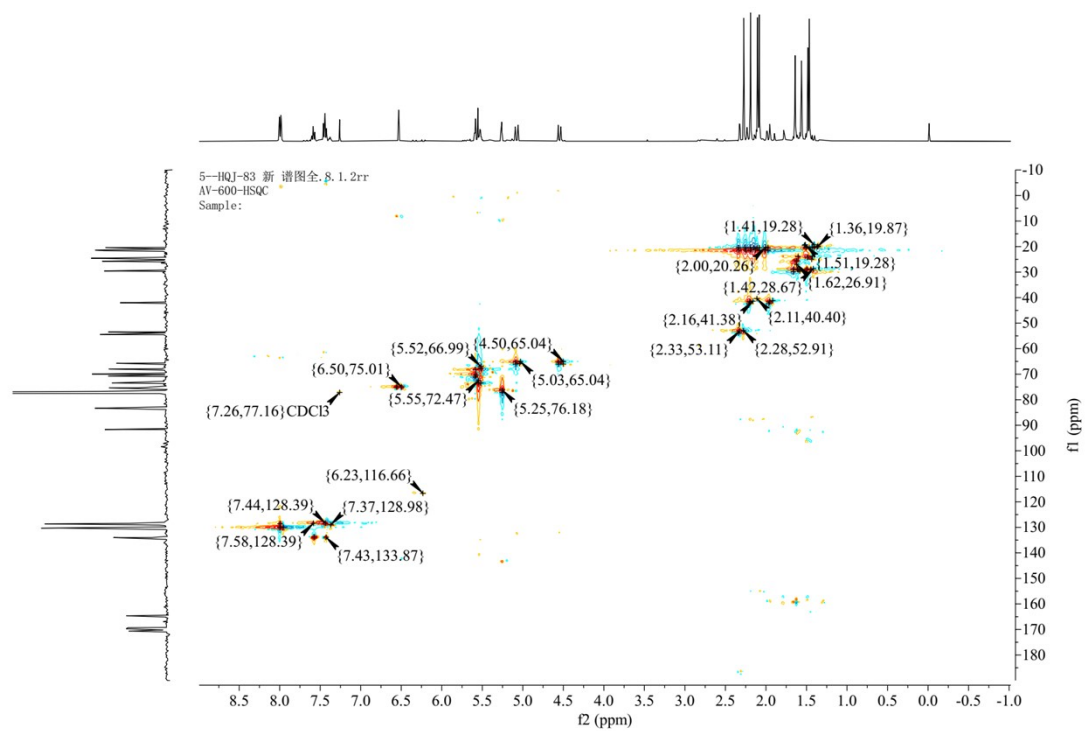


Figure S43 HSQC spectrum (600 MHz, CDCl₃) of compound **5**

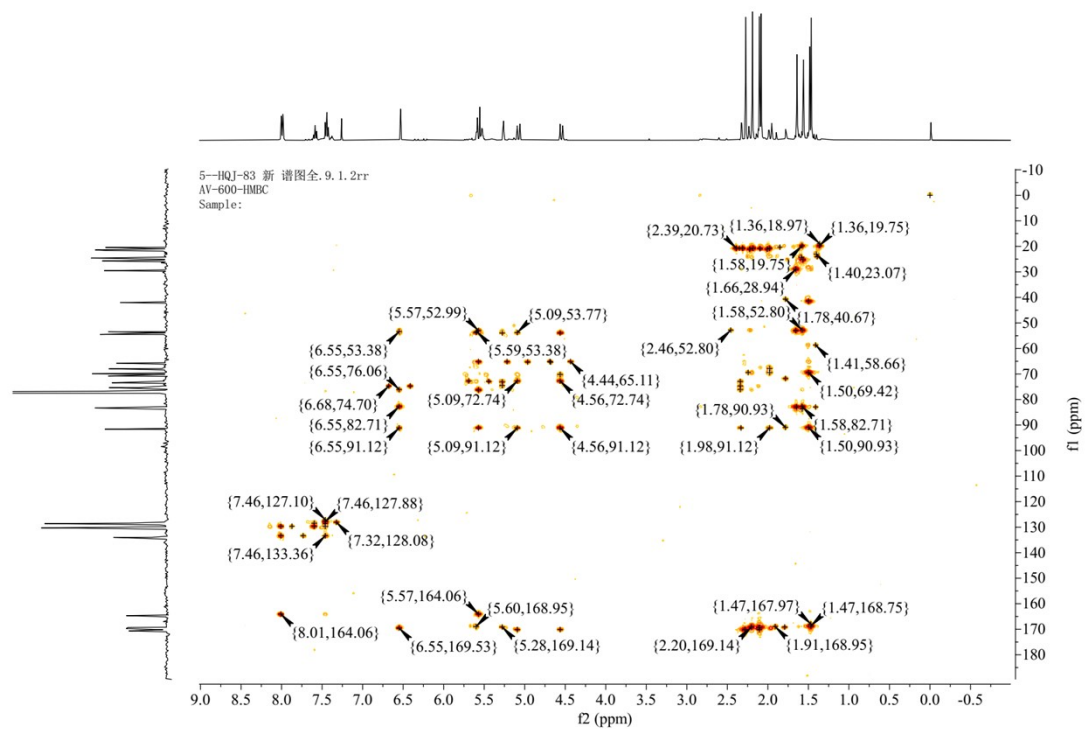


Figure S44 HMBC spectrum (600 MHz, CDCl₃) of compound **5**

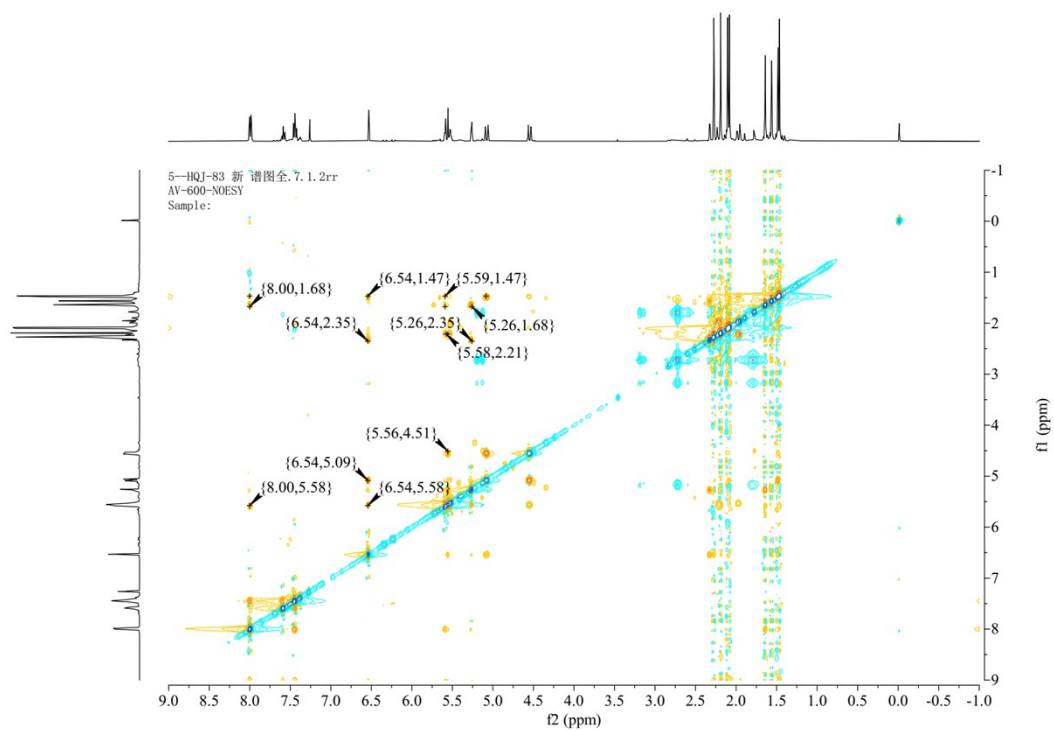


Figure S45 NOESY spectrum (600 MHz, CDCl_3) of compound **5**

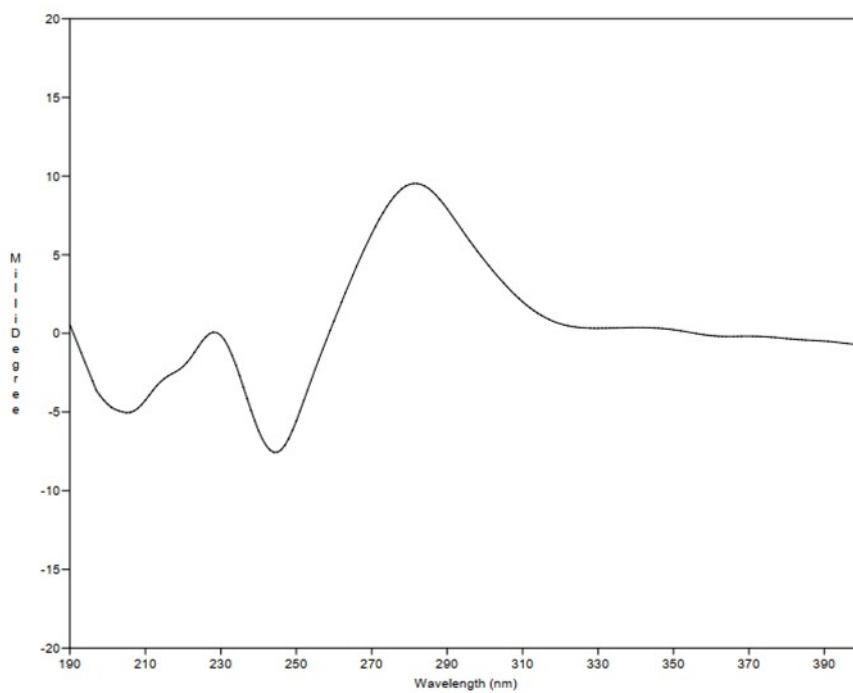


Figure S46 Experimental ECD spectrum of compound **5**

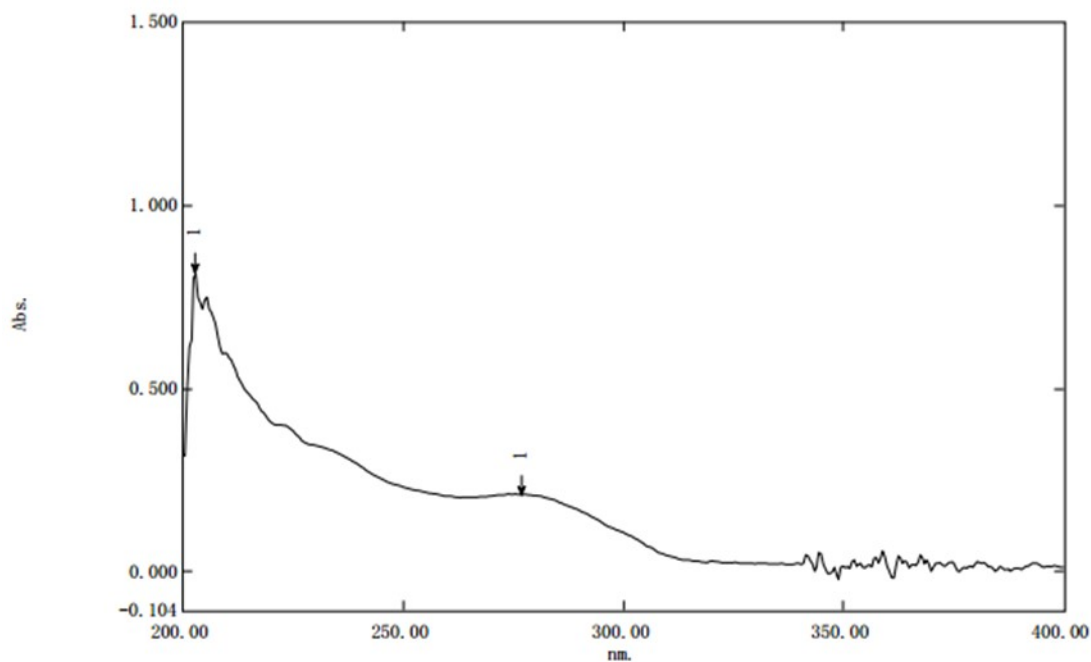
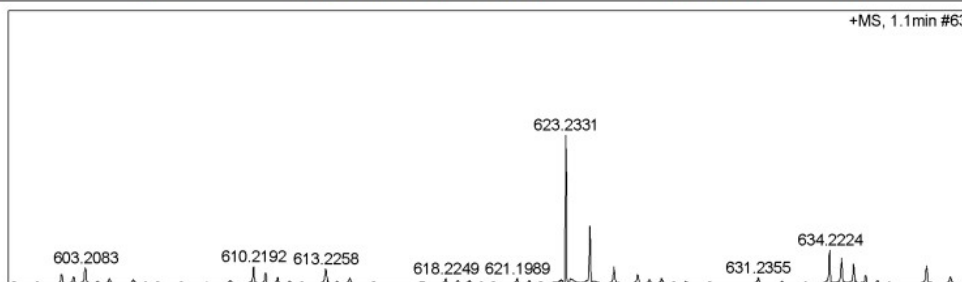


Figure S47 UV spectrum of compound **6**

Acquisition Parameter

| | | | | | |
|-------------|----------|-----------------------|-----------|------------------|-----------|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 1.2 Bar |
| Focus | Active | Set Capillary | 4500 V | Set Dry Heater | 180 °C |
| Scan Begin | 50 m/z | Set End Plate Offset | -500 V | Set Dry Gas | 8.0 l/min |
| Scan End | 1500 m/z | Set Collision Cell RF | 400.0 Vpp | Set Divert Valve | Source |



| Meas. # m/z | Formula | m/z | err [ppm] | Me an err [ppm] | rdb | N- R ul e | e ⁻ Conf | mSig ma | Std I | Std Mean m/z | Std I VarNo rm | Std m/z Diff | Std Comb Dev |
|----------------|---|----------|--------------|--------------------------|------|--------------------|------------------------|------------|--------|--------------------|----------------------|--------------------|--------------------|
| 623.2331 | C ₃₀ H ₃₉ O ₁₄ | 623.2334 | 0.5 | 1.3 | 11.5 | ok | even | 37.10 | 0.0651 | 0.0039 | 0.0262 | 0.0032 | 0.8427 |

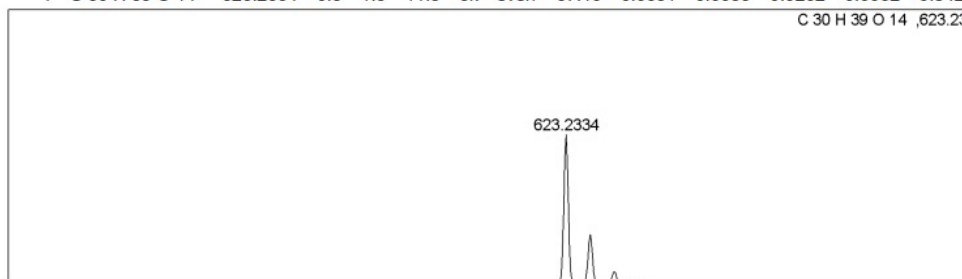


Figure S48 HRESIMS spectrum of compound **6**

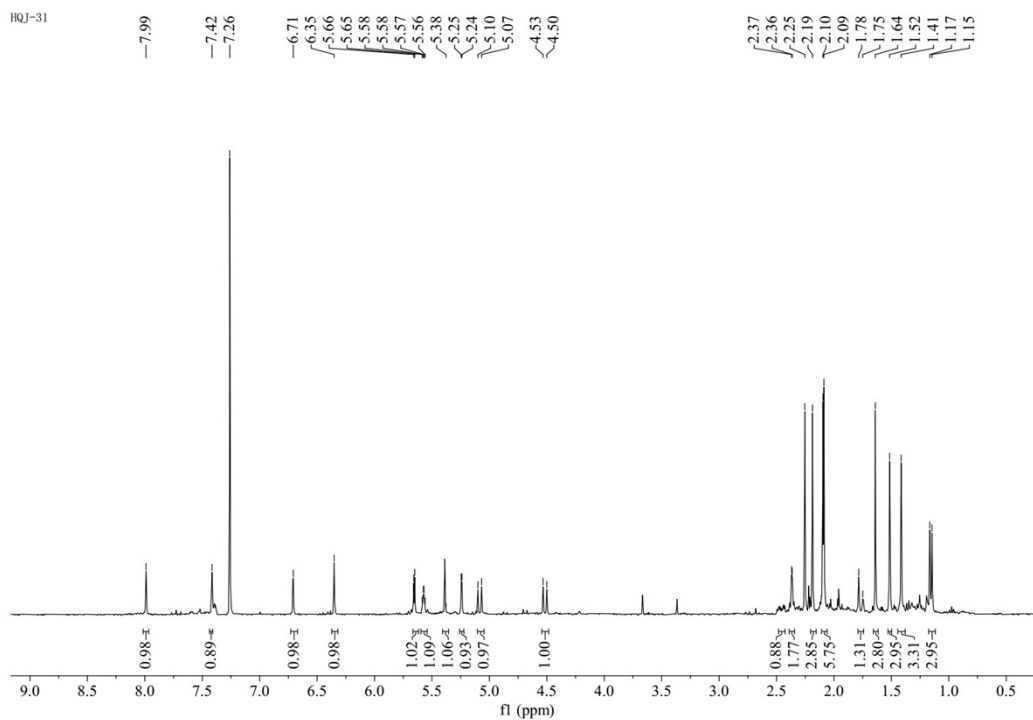


Figure S49 ^1H NMR spectrum (400 MHz, CDCl_3) of compound **6**

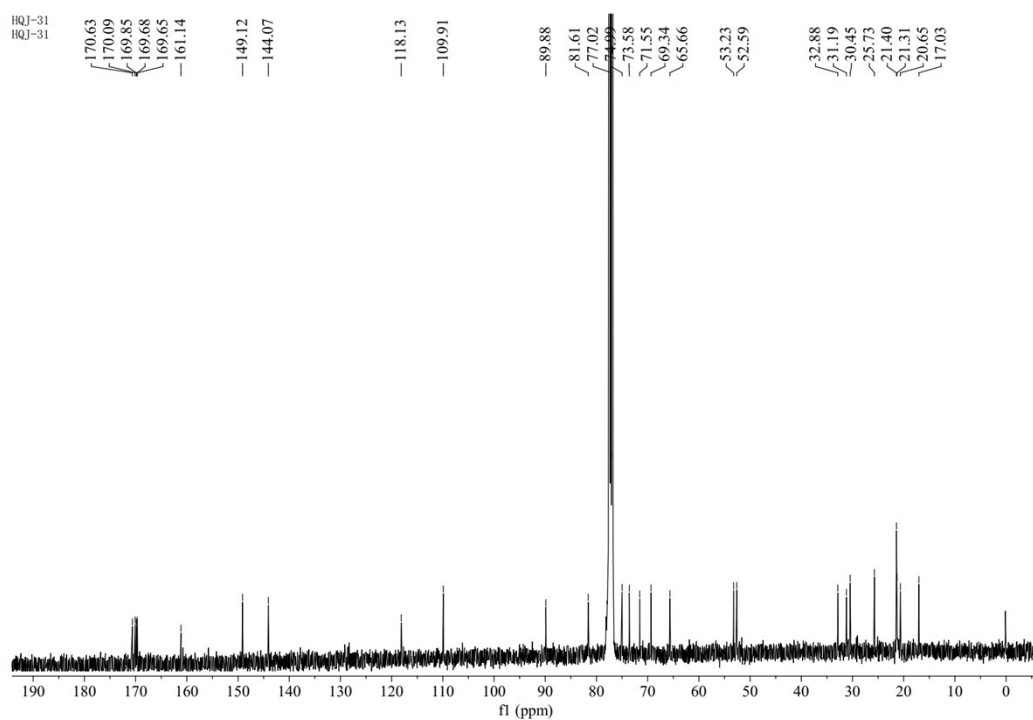


Figure S50 ^{13}C NMR spectrum (100 MHz, CDCl_3) of compound **6**

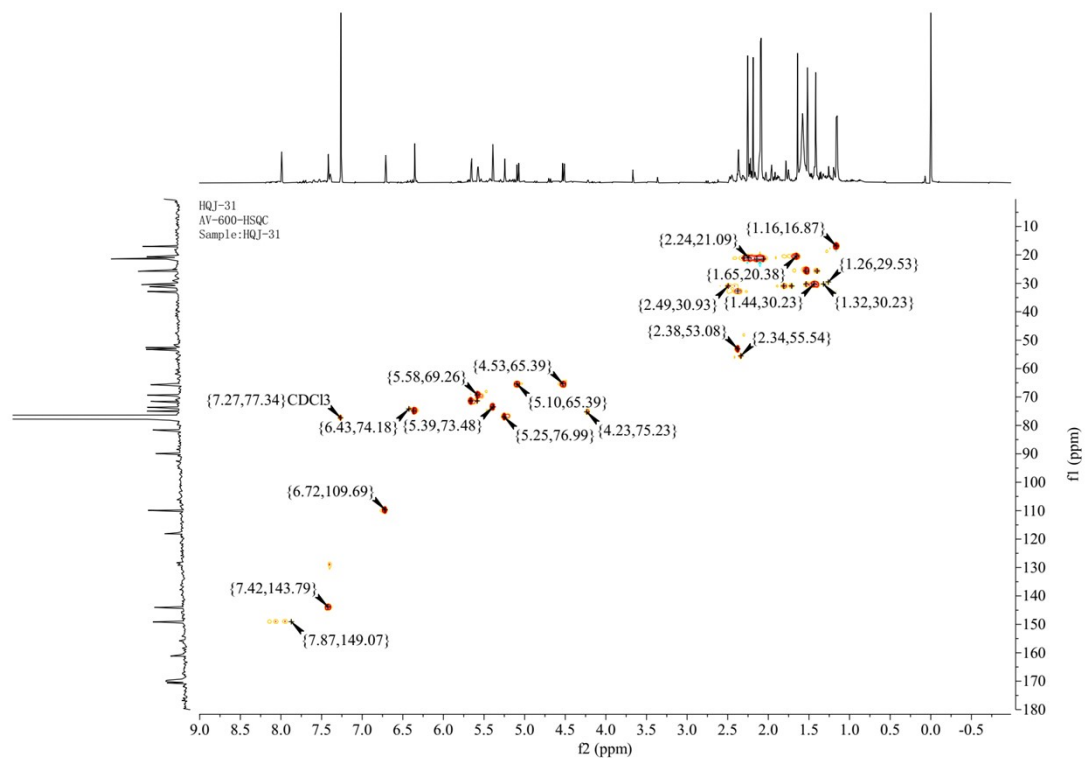


Figure S51 HSQC spectrum (600 MHz, CDCl₃) of compound 6

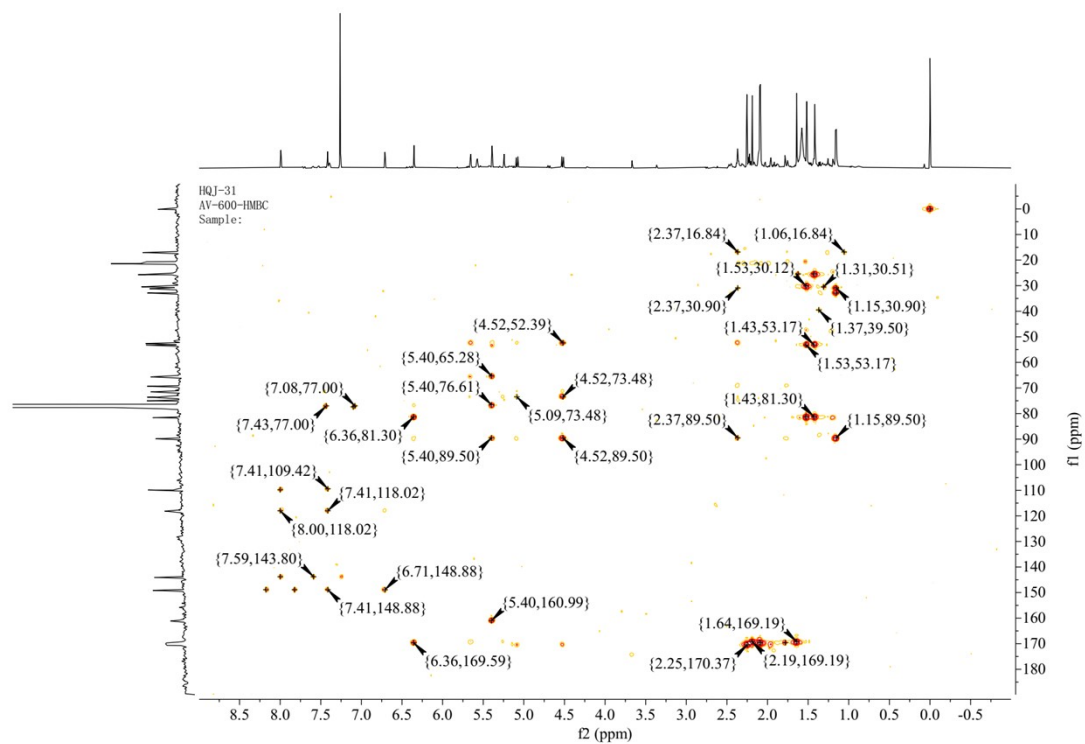


Figure S52 HMBC spectrum (600 MHz, CDCl₃) of compound 6

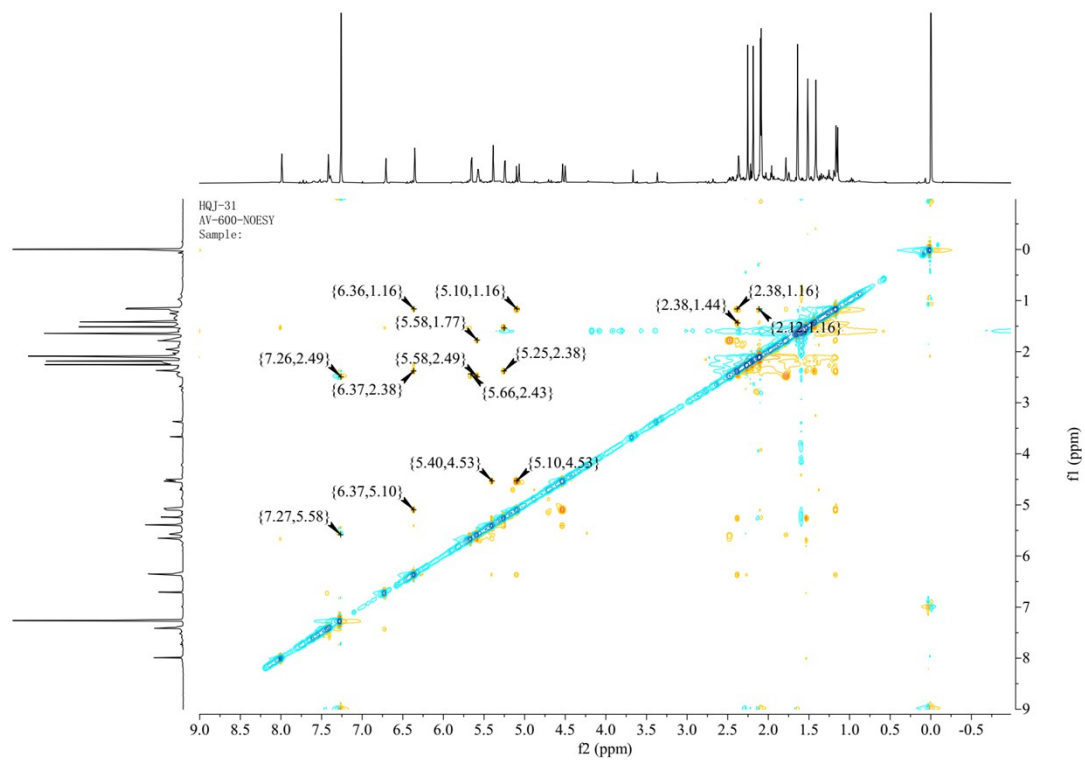


Figure S53 NOESY spectrum (600 MHz, CDCl_3) of compound **6**

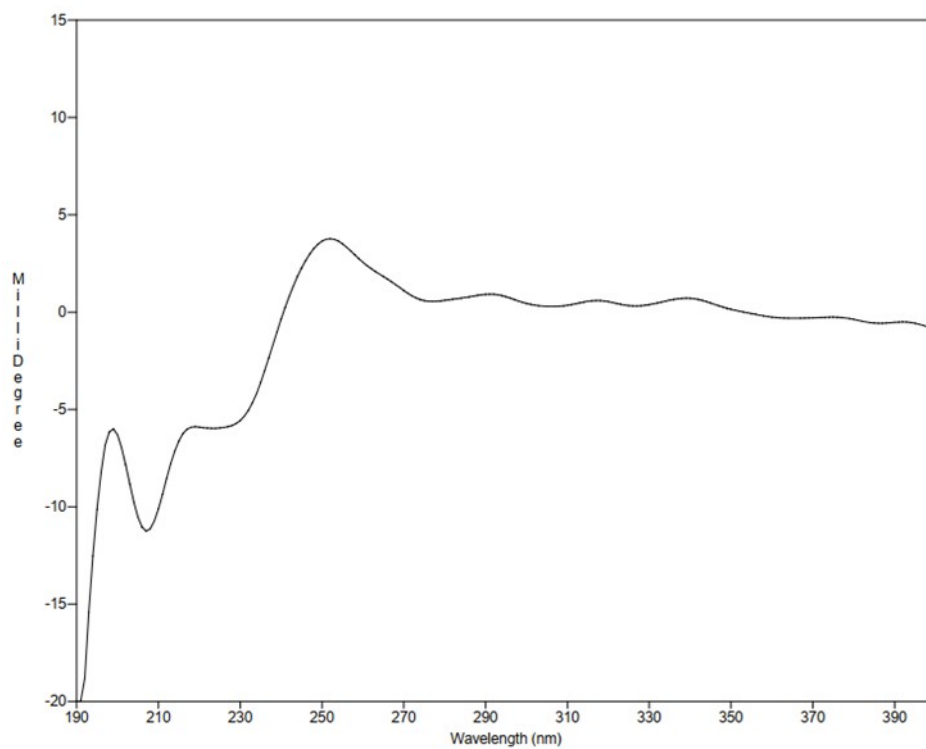


Figure S54 Experimental ECD spectrum of compound **6**

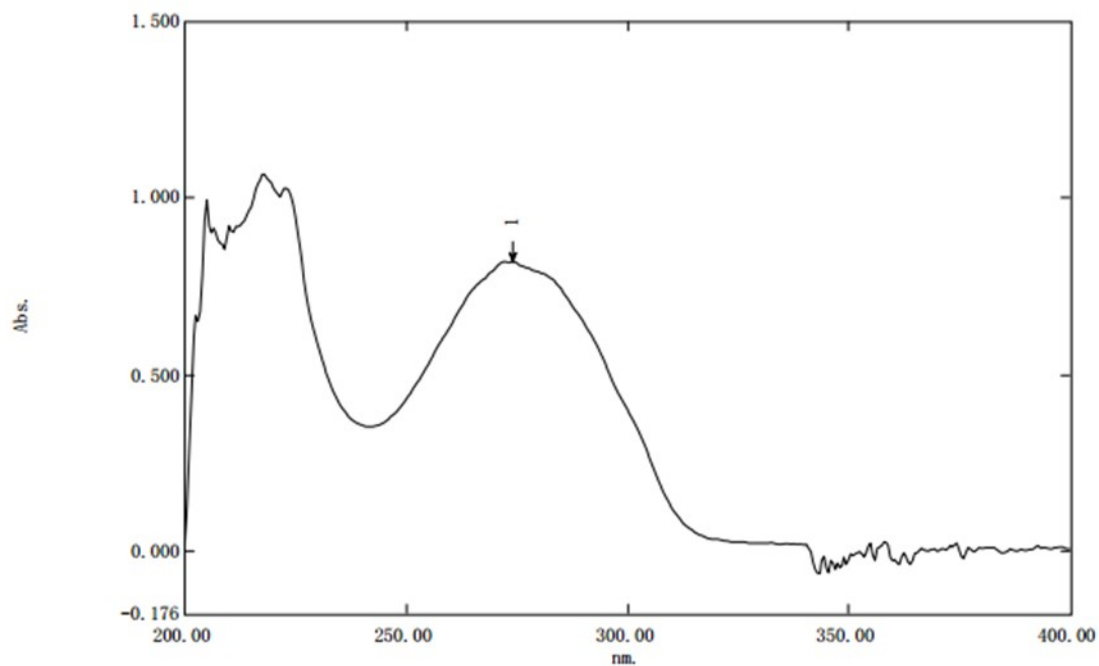


Figure S55 UV spectrum of compound 7

Acquisition Parameter

| | | | | | |
|-------------|----------|-----------------------|-----------|------------------|-----------|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 1.2 Bar |
| Focus | Active | Set Capillary | 4500 V | Set Dry Heater | 180 °C |
| Scan Begin | 50 m/z | Set End Plate Offset | -500 V | Set Dry Gas | 8.0 l/min |
| Scan End | 1500 m/z | Set Collision Cell RF | 400.0 Vpp | Set Divert Valve | Source |

Generate Molecular Formula Parameter

| | | |
|------------------|------------------------|---------|
| Formula, min. | | |
| Formula, max. | | |
| Measured m/z | Tolerance | Charge |
| Check Valence | Minimum | Maximum |
| Nitrogen Rule | Electron Configuration | |
| Filter H/C Ratio | Minimum | Maximum |
| Estimate Carbon | | |

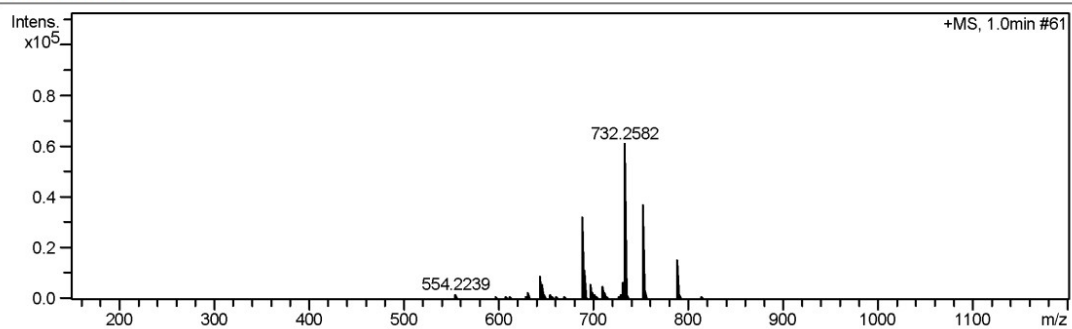


Figure S56 HRESIMS spectrum of compound 7

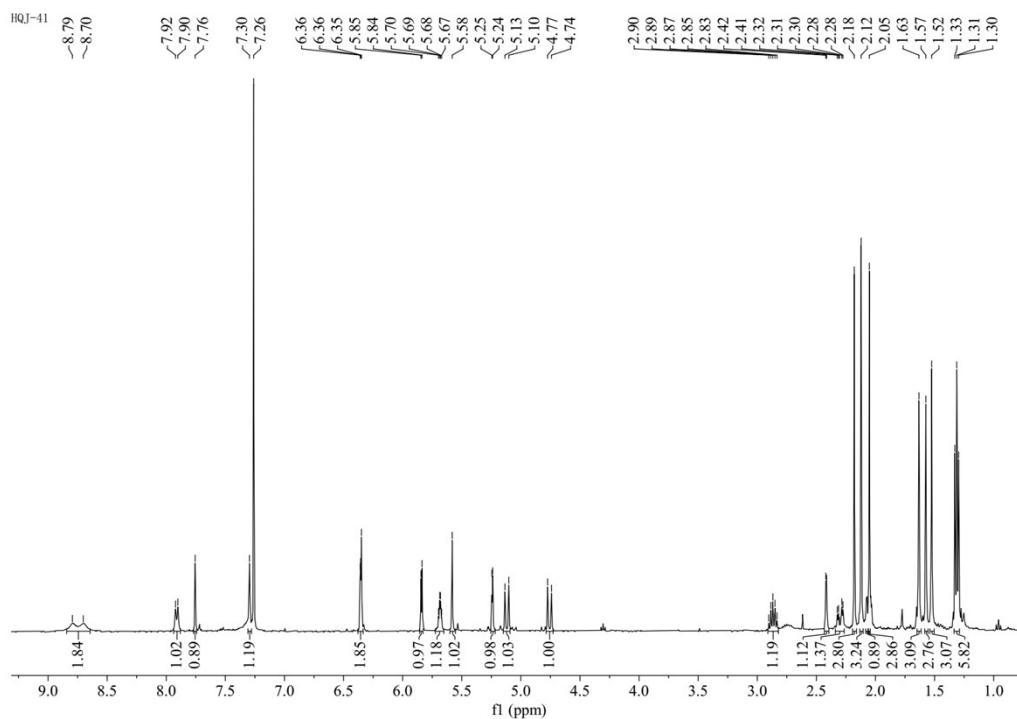


Figure S57 ^1H NMR spectrum (400 MHz, CDCl_3) of compound 7

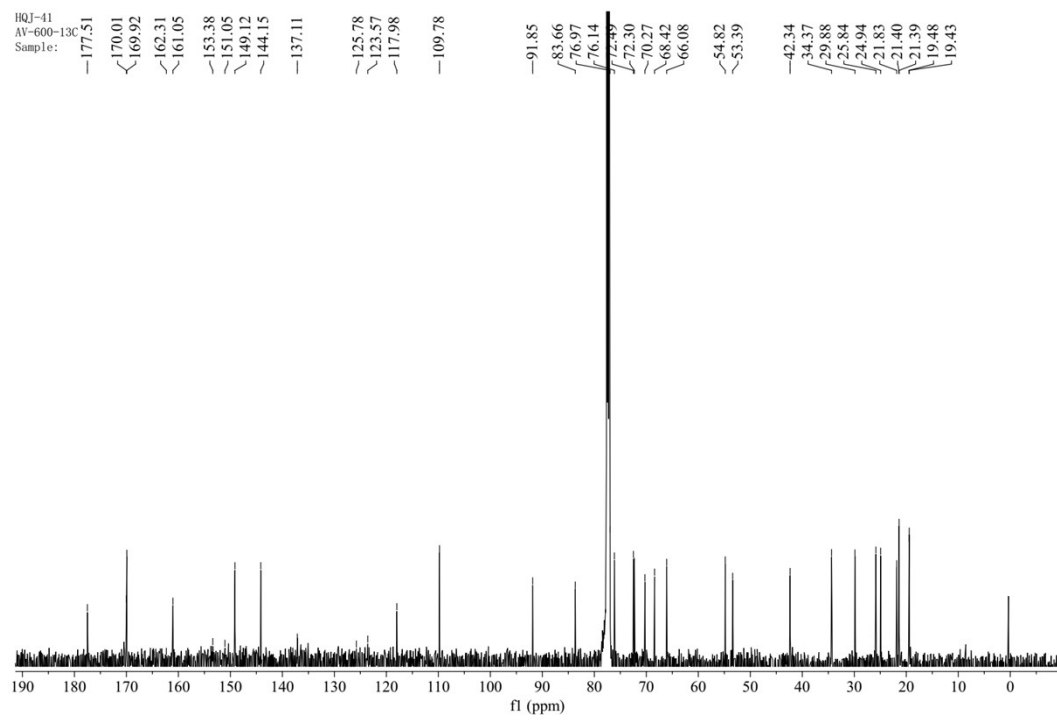


Figure S58 ^{13}C NMR spectrum (100 MHz, CDCl_3) of compound 7

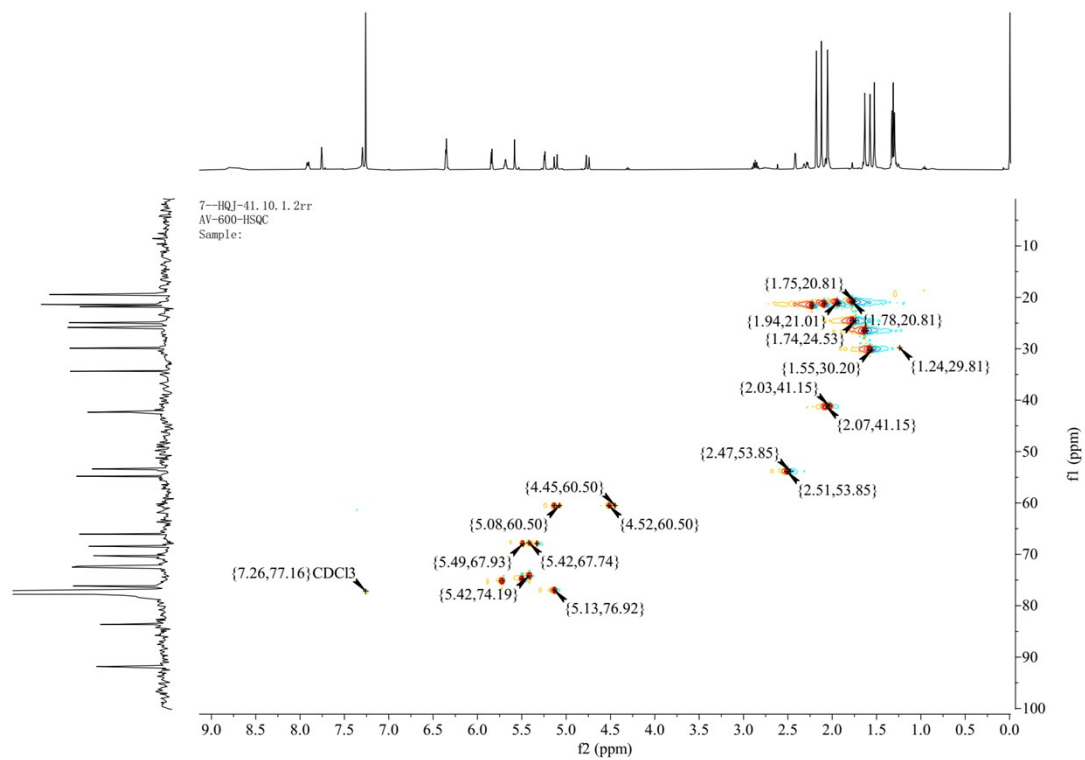


Figure S59 HSQC spectrum (600 MHz, CDCl_3) of compound **7**

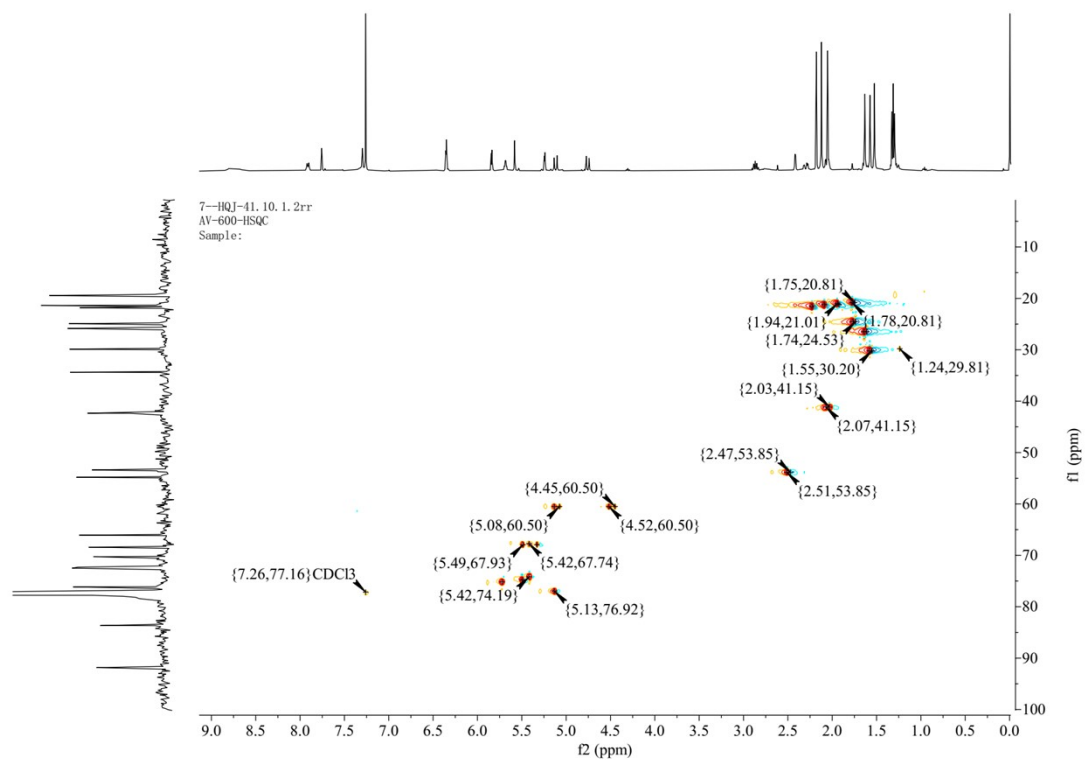


Figure S60 HMBC spectrum (600 MHz, CDCl_3) of compound **7**

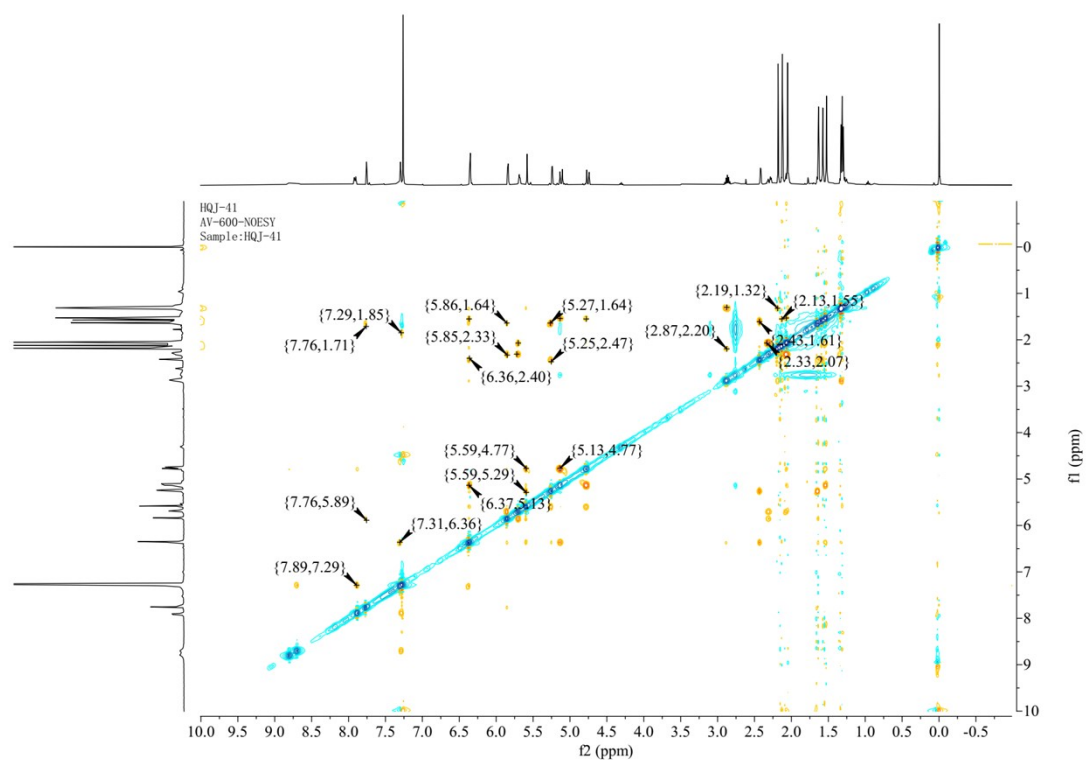


Figure S61 NOESY spectrum (600 MHz, CDCl_3) of compound **7**

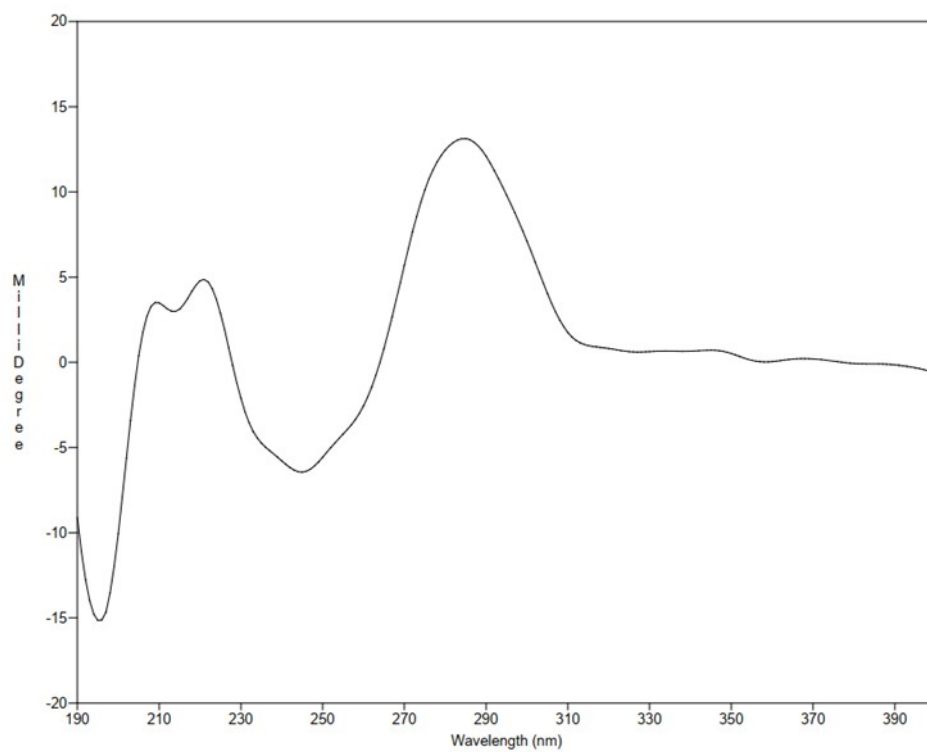


Figure S62 Experimental ECD spectrum of compound **7**

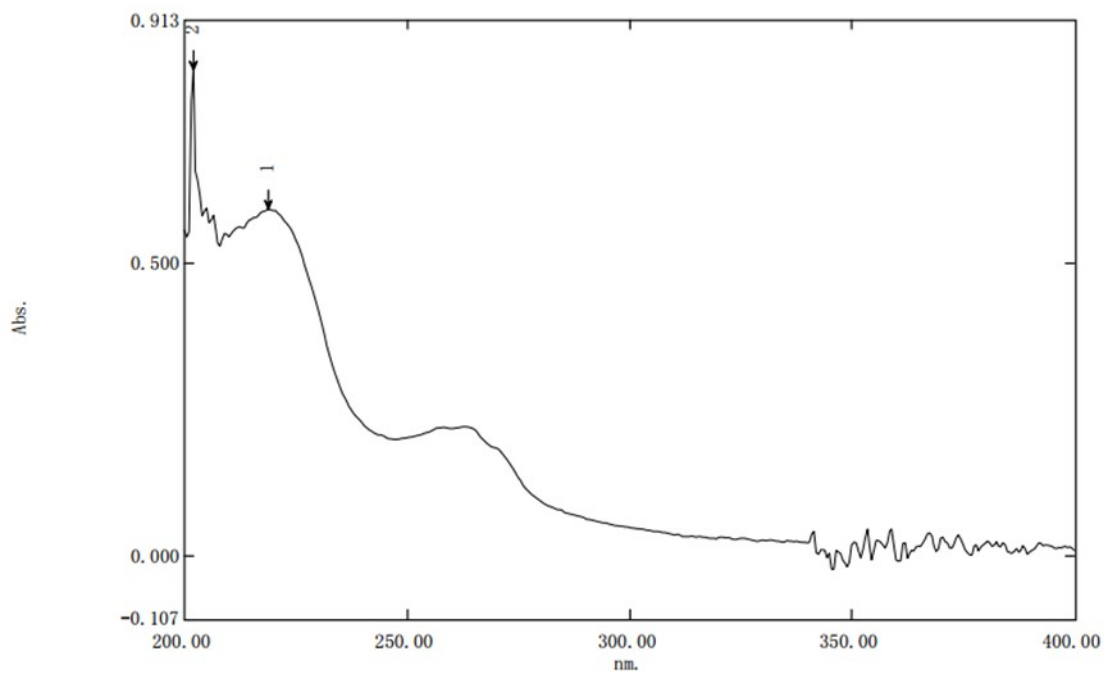


Figure S63 UV spectrum of compound **8**

Acquisition Parameter

| | | | | | |
|-------------|----------|-----------------------|-----------|------------------|-----------|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 1.2 Bar |
| Focus | Active | Set Capillary | 4500 V | Set Dry Heater | 180 °C |
| Scan Begin | 50 m/z | Set End Plate Offset | -500 V | Set Dry Gas | 8.0 l/min |
| Scan End | 1500 m/z | Set Collision Cell RF | 400.0 Vpp | Set Divert Valve | Source |

Generate Molecular Formula Parameter

| | | |
|------------------|------------------------|---------|
| Formula, min. | | |
| Formula, max. | | |
| Measured m/z | Tolerance | Charge |
| Check Valence | Minimum | Maximum |
| Nitrogen Rule | Electron Configuration | |
| Filter H/C Ratio | Minimum | Maximum |
| Estimate Carbon | | |

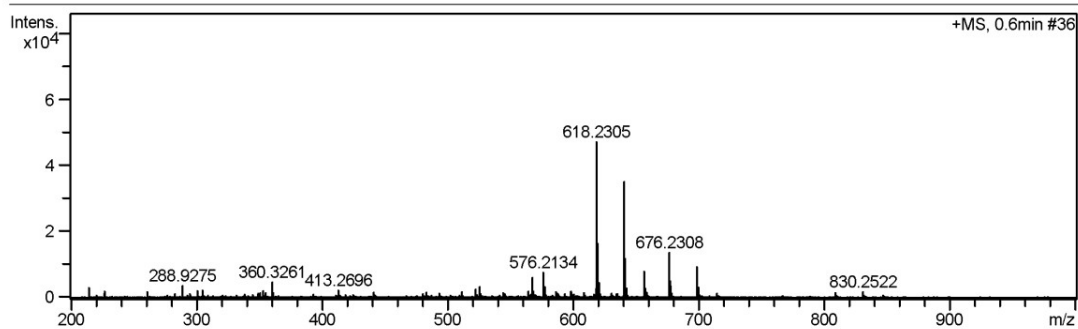


Figure S64 HRESIMS spectrum of compound **8**

HQJ-12

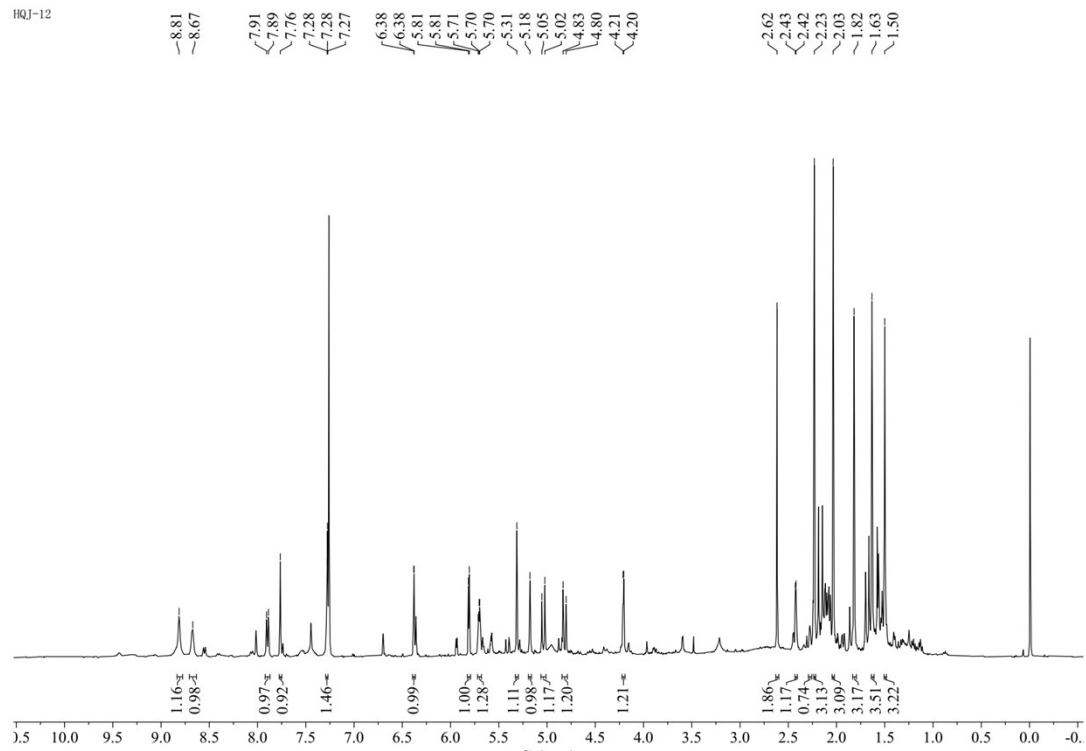


Figure S65 ¹H NMR spectrum (400 MHz, CDCl₃) of compound 8

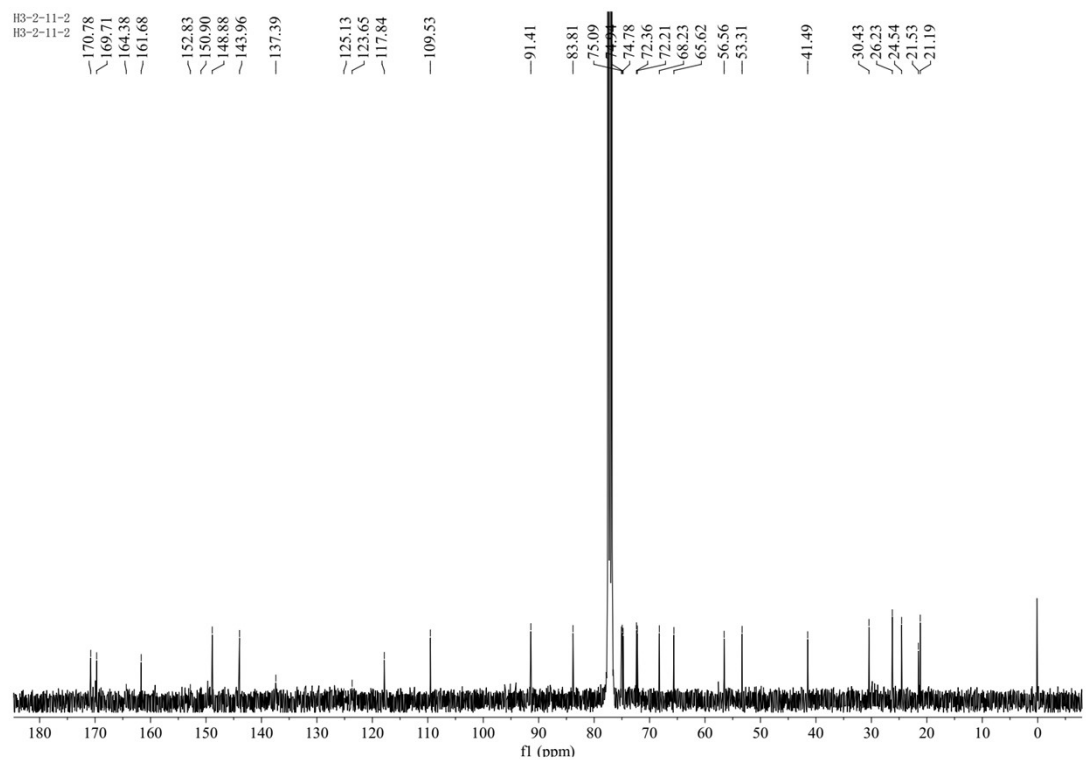


Figure S66 ¹³C NMR spectrum (100 MHz, CDCl₃) of compound 8

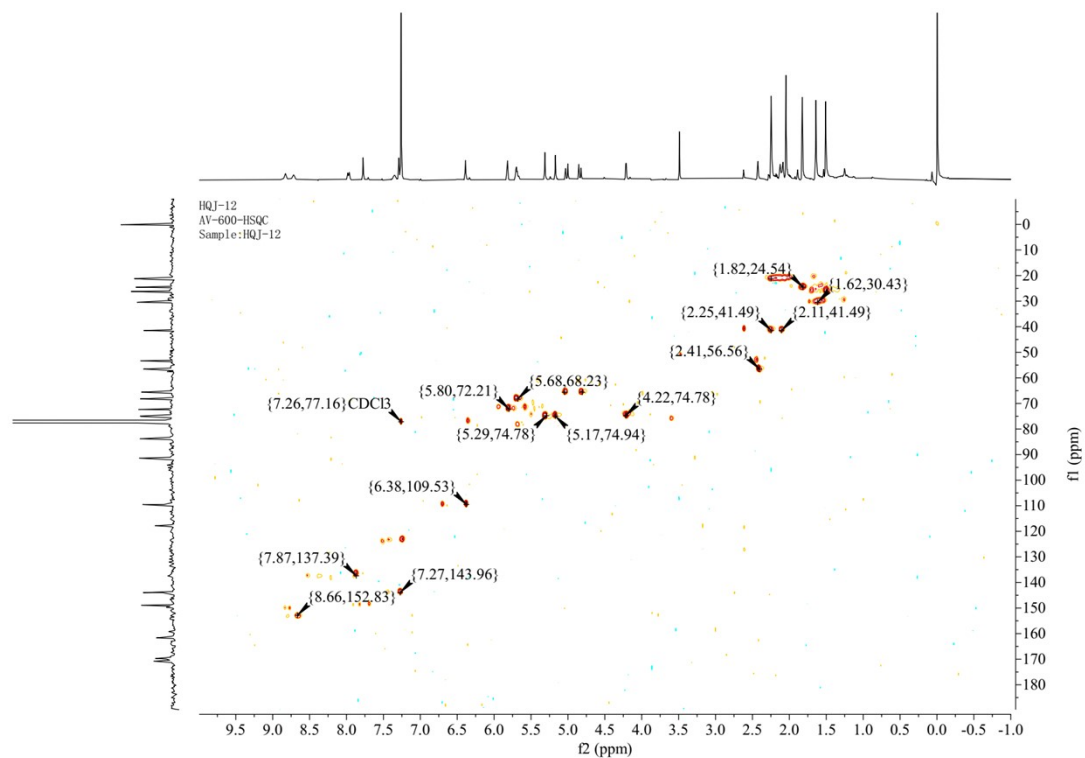


Figure S67 HSQC spectrum (600 MHz, CDCl_3) of compound **8**

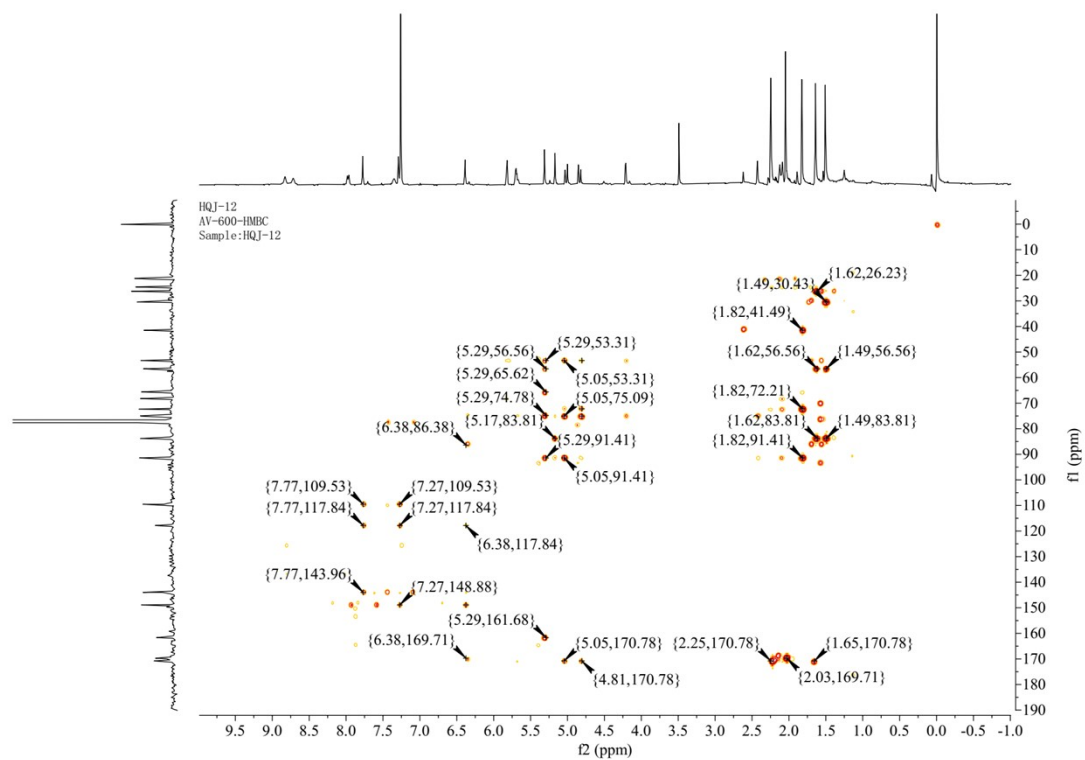


Figure S68 HMBC spectrum (600 MHz, CDCl_3) of compound **8**

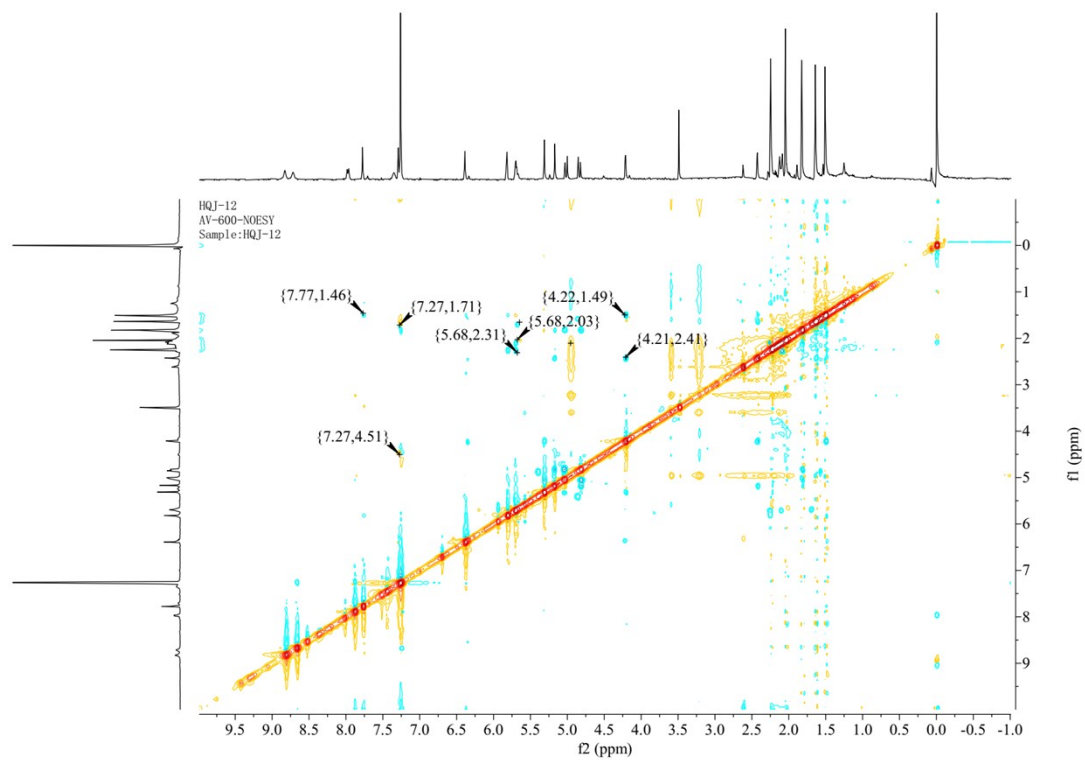


Figure S69 NOESY spectrum (600 MHz, CDCl_3) of compound **8**

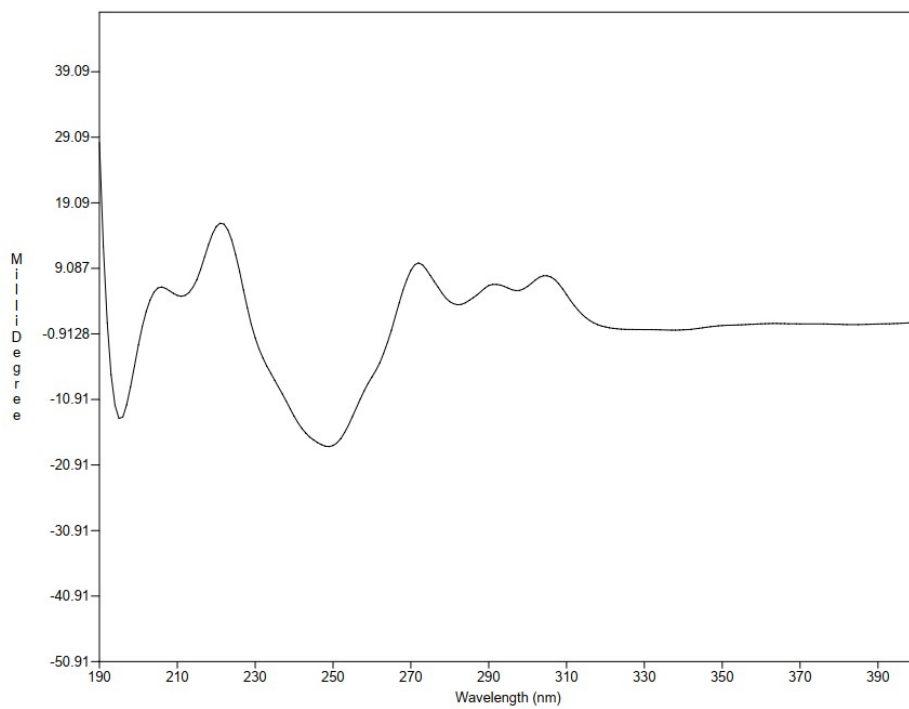


Figure S70 Experimental ECD spectrum of compound **8**

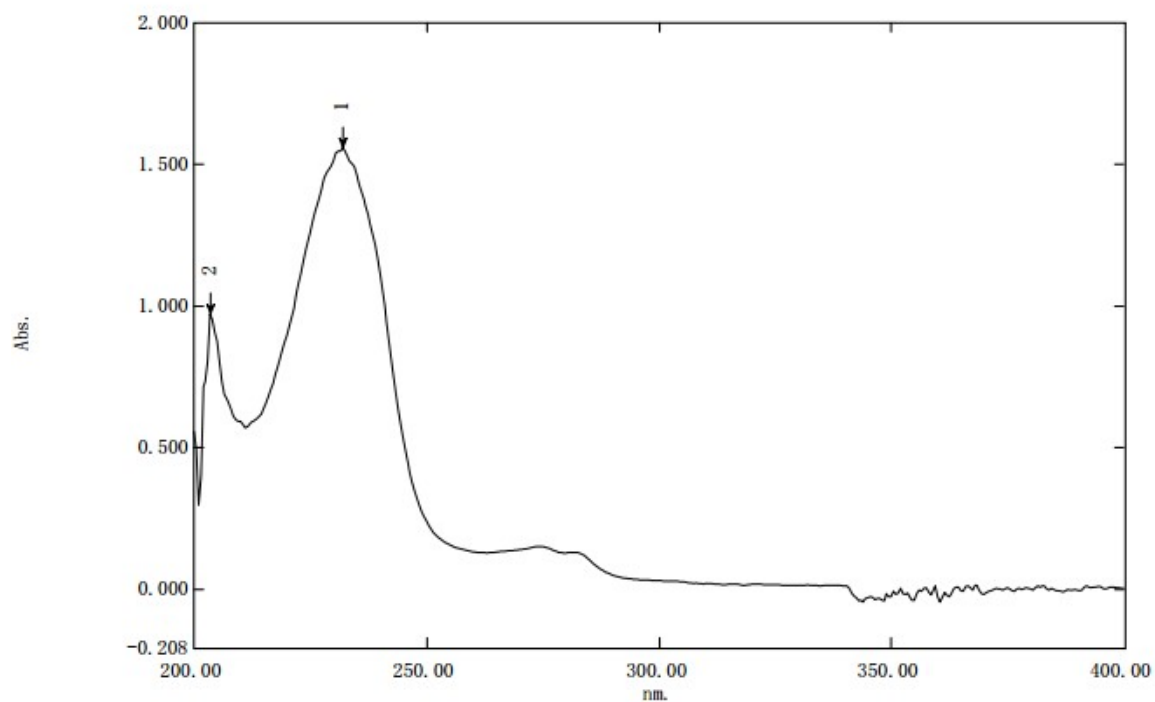
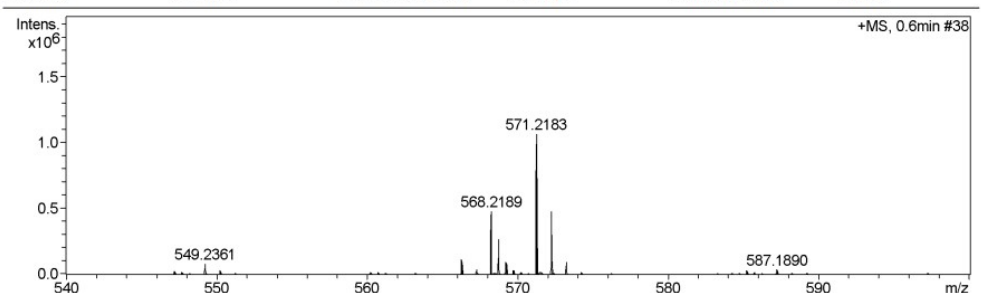


Figure S71 UV spectrum of compound **9**

| Acquisition Parameter | | | | | |
|-----------------------|----------|-----------------------|-----------|------------------|-----------|
| Source Type | ESI | Ion Polarity | Positive | Set Nebulizer | 1.2 Bar |
| Focus | Active | Set Capillary | 4500 V | Set Dry Heater | 180 °C |
| Scan Begin | 50 m/z | Set End Plate Offset | -500 V | Set Dry Gas | 8.0 l/min |
| Scan End | 1500 m/z | Set Collision Cell RF | 400.0 Vpp | Set Divert Valve | Source |



| Meas. # | m/z | Formula | m/z | err [ppm] | Mean err [ppm] | rdb | N-Rule | e ⁻ Conf | mSigma | Std I | Std Mean m/z | Std Var | Std I No | Std m/z Diff | Std Comb Dev |
|---------|----------|---|----------|-----------|----------------|------|--------|---------------------|--------|--------|--------------|---------|----------|--------------|--------------|
| 1 | 571.2183 | C ₂₈ H ₃₆ NaO ₁₁ | 571.2150 | -5.9 | -8.5 | 10.5 | ok | even | 67.14 | 0.0956 | 0.0056 | 0.0303 | 0.0054 | 0.8427 | |

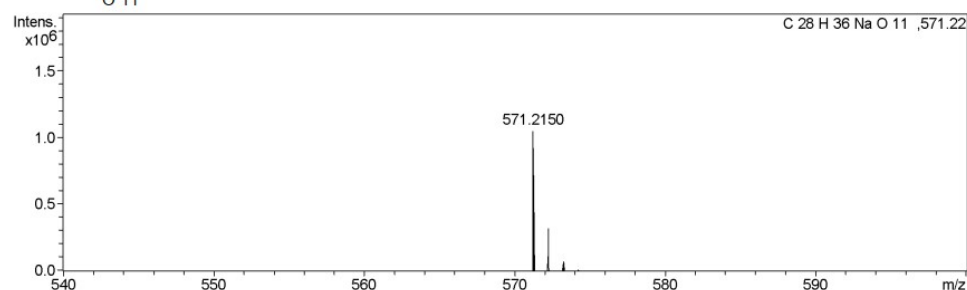


Figure S72 HRESIMS spectrum of compound **9**

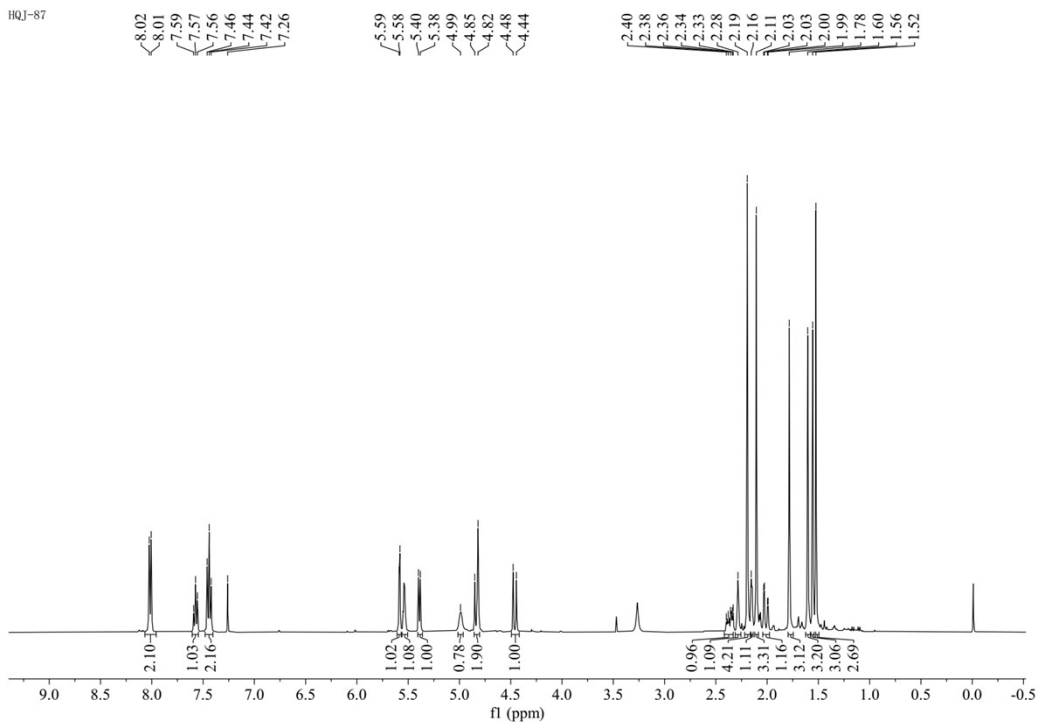


Figure S73 ^1H NMR spectrum (400 MHz, CDCl_3) of compound **9**

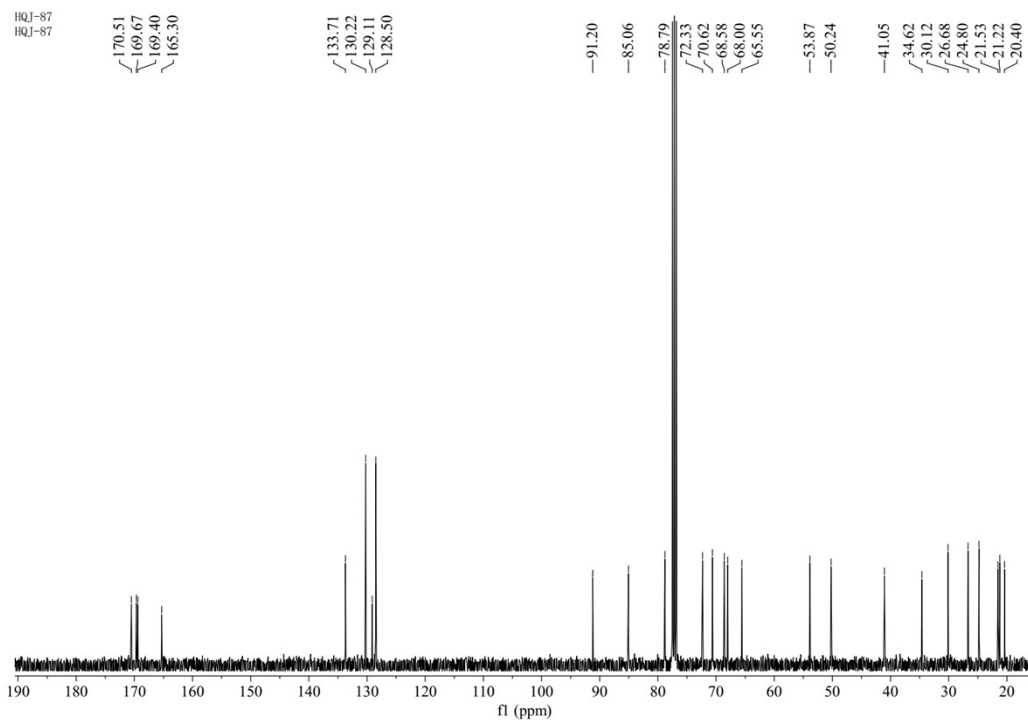


Figure S74 ^{13}C NMR spectrum (100 MHz, CDCl_3) of compound **9**

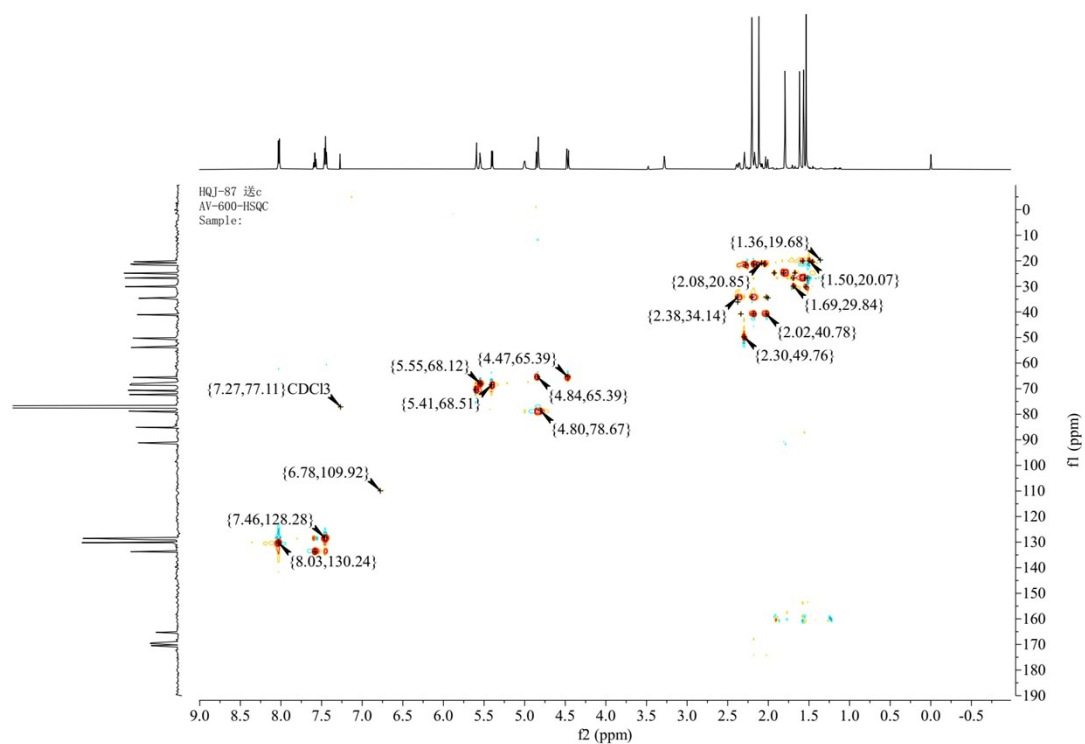


Figure S75 HSQC spectrum (600 MHz, CDCl_3) of compound **9**

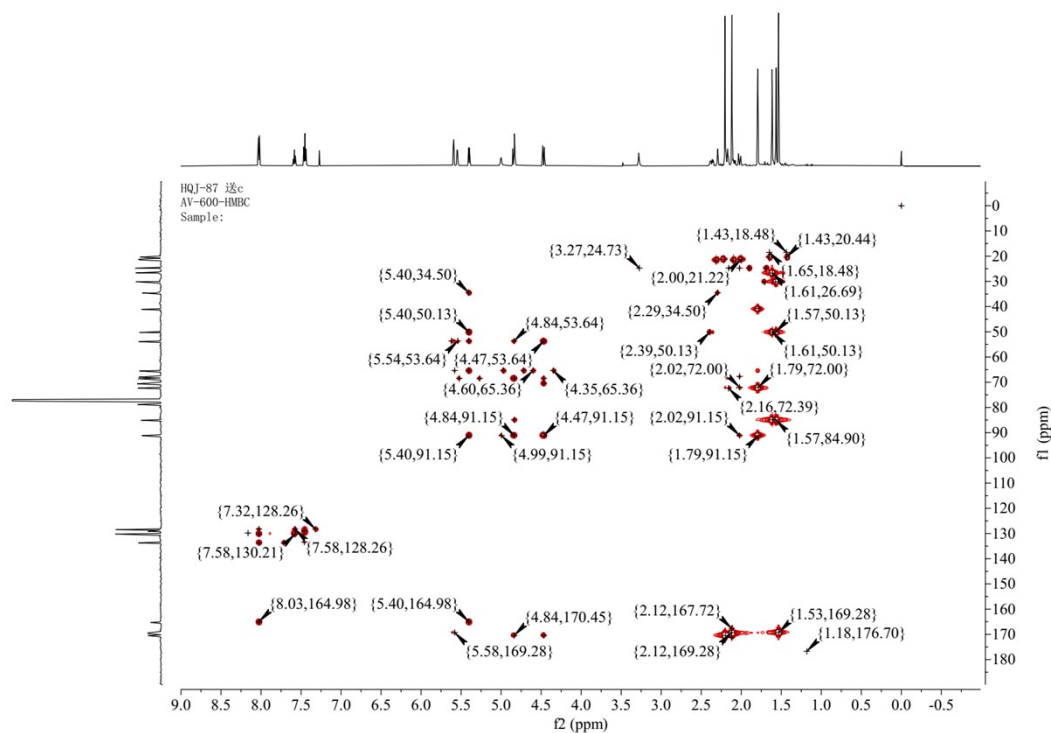


Figure S76 HMBC spectrum (600 MHz, CDCl_3) of compound **9**

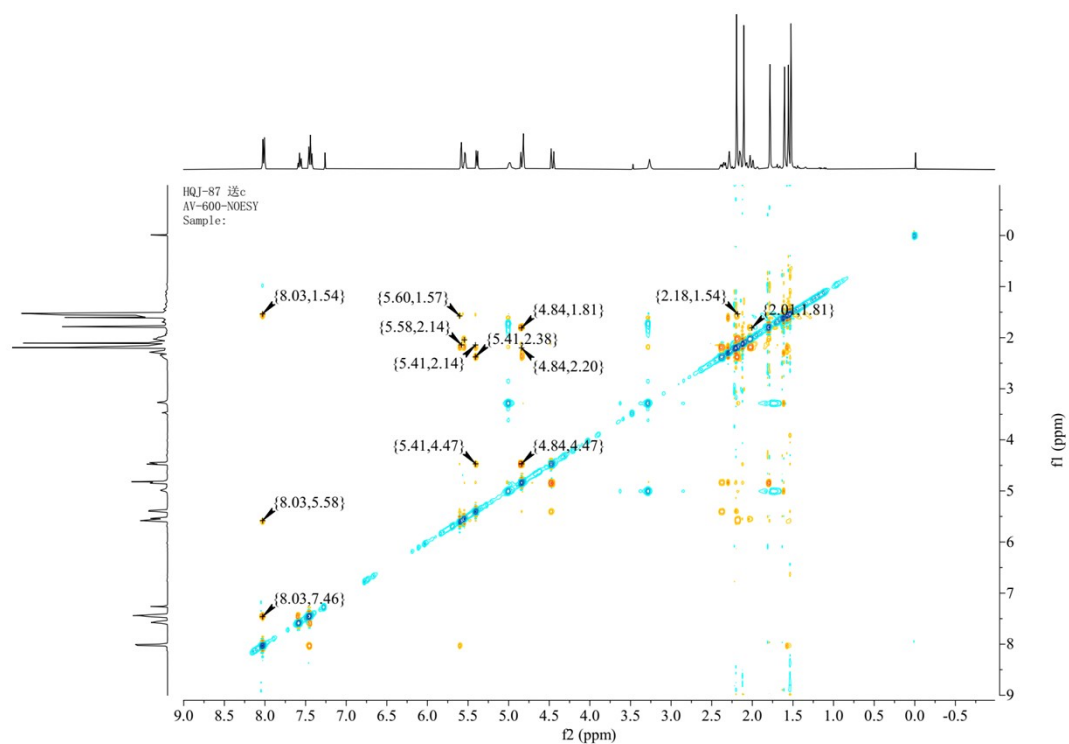


Figure S77 NOESY spectrum (600 MHz, CDCl_3) of compound **9**

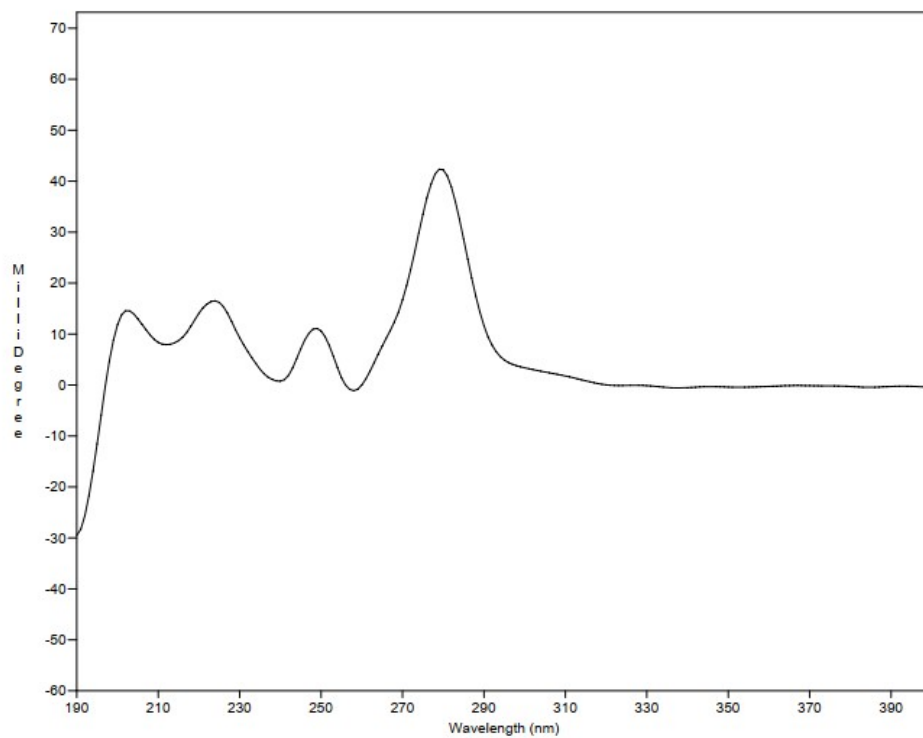


Figure S78 Experimental ECD spectrum of compound **9**