Electronic Supporting Information

Preparation of Reusable Copper-Based Biomass-Carbon Aerogel Catalysts and Application in Highly Selective Reduction of Maleimides to Succinimides with Hydrosilane as Hydrogen Source

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Total 45 pages including 38 Figures.

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I. *General remarks*

All reagents were obtained from commercial sources and used as received. Technical grade petroleum ether (40-60°C bp.) and ethyl acetate were used for chromatography column. Chitin, potassium hydroxide, urea, tert-butanol, and copper trifluoromethane sulfonate ($Cu(OTf)_2$) were purchased from Aladdin Biochemical Technology Co., Ltd. (Shanghai, China).

SEM images of $Cu(OTf)_2$@CAC were taken with an FE-SEM (Sigma 500, Zeiss, Germany) operating at an acceleration voltage of 5 kV. Optical microscope picture was recorded on a polarizing microscope (DM2700P, Leica, Germany). TEM images of the super-thin sliced $Cu(OTf)_2$@CAC were taken with a transmission electron microscopy (JEM-2100, JEOL, Japan) operating at an accelerating voltage of 200 kV. The partial carbonization process on the chitin/Cu^{2+} composite aerogels was carried out on a thermogravimetric analysis system (Q50, TA, America) at a heating rate of 10 °C min^{-1} from 25 to 250 °C, followed by a constant temperature at 250 °C for 4 h under the nitrogen atmosphere. The Brunauer-Emmett-Teller (BET) surface areas were measured and calculated by the nitrogen sorption-desorption isotherms using an adsorption analyzer (ASAP 2020, Micromeritics, USA). 2D Raman imaging was performed using a laser confocal Raman imaging microscope (inVia, Renishaw, UK). The wavelength of the excitation laser was 532 nm. Raman maps (scan range, 50 μm × 50 μm) were collected using a spatial resolution of 200 nm. The direct classical least squares (DCLS) method developed by accessory software was applied for imaging the chitin and the amorphous carbon domains. X-ray photoelectron spectra was conducted on an X-ray photoelectron spectrometer (ESCALAB250Xi, Thermo Fisher Scientific, America) with monochromatic Al target test, and full spectrum passing energy 100 eV.

^1H NMR spectra were recorded in $CDCl_3$ at ambient temperature on Bruker AVANCE I 500 spectrometers at 500.1 MHz, using the solvent as internal standard (7.26 ppm). ^{13}C NMR spectra were obtained at 125 MHz and referenced to the internal solvent signals (central peak is 77.2 ppm). Chemical shift (δ) and coupling constants (*J*) are given in ppm and in Hz, respectively. The peak patterns are indicated as follows: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet, and br. for broad.

GC analyses were performed with GC-7890A (Agilent) equipped with a 30-m capillary column (HP-5ms, fused silica capillary column, 30 M*0.25 mm*0.25 mm film thickness), was used with N_2/air as vector gas. GCMS were measured by GCMS-7890A-5975C (Agilent) with GC-7890A equipped with a 30-m capillary column (HP-5ms, fused silica capillary column, 30 M*0.25 mm*0.25 mm film thickness), was used with helium as vector gas. HRMS were measured by MAT 95XP (Termol) (LCMS-IT-TOF).

II. Supplementary information of $Cu(OTf)_2$@CAC

2.1 Chitin powder purification

First, 400 g crab shells were crushed, dispersed in the 4 L, 4 wt.% NaOH solution, and stirred for 8h to remove proteins. Then, the substance was successively dispersed in 4 L, 0.3 wt.% Na_2ClO (8 h, stirring, 80 ^oC) and the pH level of the mixture was constantly maintained at by adding 7 wt.% HCl to remove pigments and minerals. After repeating the above steps twice, the chitin powders were obtained by drying at 50 ^oC (Fig S1).

Figure S1. The crab shells after purification are pale yellow chitin powder.

2.2 Chitin hydrogel and chitin aerogel preparation

First, 5.26 g chitin powders were added slowly to the 100 g 20 wt.% KOH/4 wt.% Urea solution while stirring in the trap at -40 ^oC for 2h. the turbid mixture turned into yellow viscous chitin solution after centrifugation at 10000 rpm, 0 ^oC (Fig S2). Then, the chitin solution was poured and cast into the homemade mold. The chitin gel was formed by immersing the mold in 1 L absolute alcohol at -25 ^oC for 24h (Fig S3). Finally, the transparent chitin hydrogel was cut into 2×2 cm chitin gel pieces.

The chitin aerogels were fabricated by freezing-drying. Specifically, the chitin hydrogel was replaced solvent with 50 mL tert-butanol twice. Then, the gels were immersed in a box filled with liquid nitrogen for 15min and displace to freeze-drying (Christ Alpha 2-4 LDplus, -80 ^oC，0.045 atm) for 12h to obtain the white and lightweight chitin aerogel.

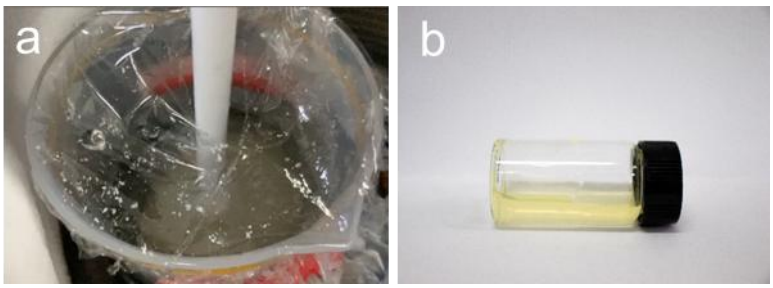


Figure S2. a) the process of the crab shells dissolution. **b)** the chitin solution with excellent fluidity.

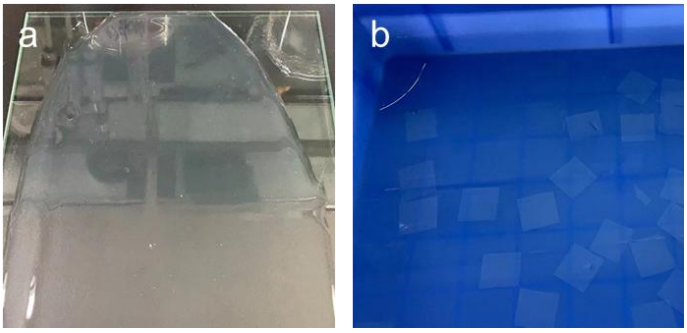


Figure S3. a) the chitin solution was poured into the mold through casting method. **b)** the chitin hydrogel with 3D nanofibrous network.

2.3 $Cu(OTf)_2$@CAC with different pyrolysis times preparation

First, 16 pieces of the chitin gel were replaced solvent with 50 mL acetone to obtain chitin organogels and immersed into 50 mL, 5 wt.% $Cu(OTf)_2$ acetone solution and stirred for 5 h (Fig S4). Subsequently, the chitin/Cu gels were washed and replaced with 50 mL acetone and 50 mL tert-butanol for 24 h, respectively. Then the chitin/Cu gels were immersed in a box filled with liquid nitrogen and the chitin/Cu aerogels were obtained by freeze-drying (Christ Alpha 2-4 LDplus, -80 ^oC, 0.045 atm). Finally, 120 mg of the chitin/Cu aerogels were carbonized under a nitrogen atmosphere at 250 ^oC (heating rate is 5 ^oC/min and cooling rate is 10 ^oC/min) for 1h to obtain $Cu(OTf)_2$@CAC-1h. Whatmore, 120 mg of the chitin/Cu aerogels were carbonized under a nitrogen atmosphere at 250 ^oC (heating rate is 5 ^oC/min and cooling rate is 10 ^oC/min) for 3h to obtain $Cu(OTf)_2$@CAC-3h, and 120 mg of the chitin/Cu aerogels were carbonized for 4h as the same procedure to obtain $Cu(OTf)_2$@CAC-4h. All $Cu(OTf)_2$@CAC-1h, $Cu(OTf)_2$@CAC-3h and $Cu(OTf)_2$@CAC-4h were washed by 10 mL acetone for 12h and dryed at 50 ^oC to remove impurities.

Figure S4. the process of the chitin organogel chelated with Cu^{2+} in the $Cu(OTf)_2$-containing acetone solution

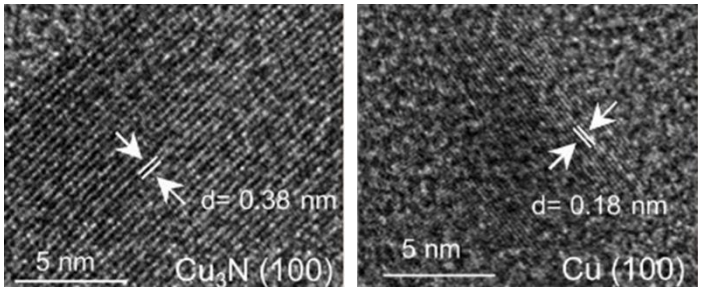


Figure S5. HRTEM image of $Cu(OTf)_2$@CAC with obvious lattice spacing.

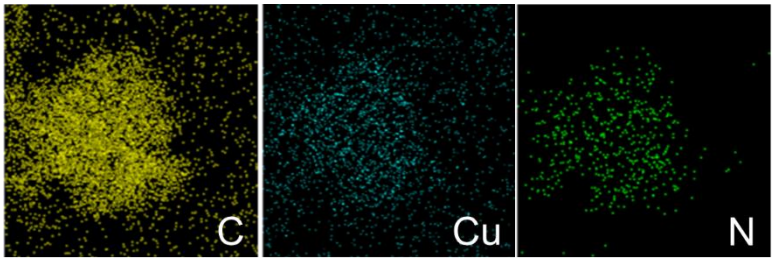


Figure S6. EDS test of the C, N, and Cu elements inside the $Cu(OTf)_2$@CAC.

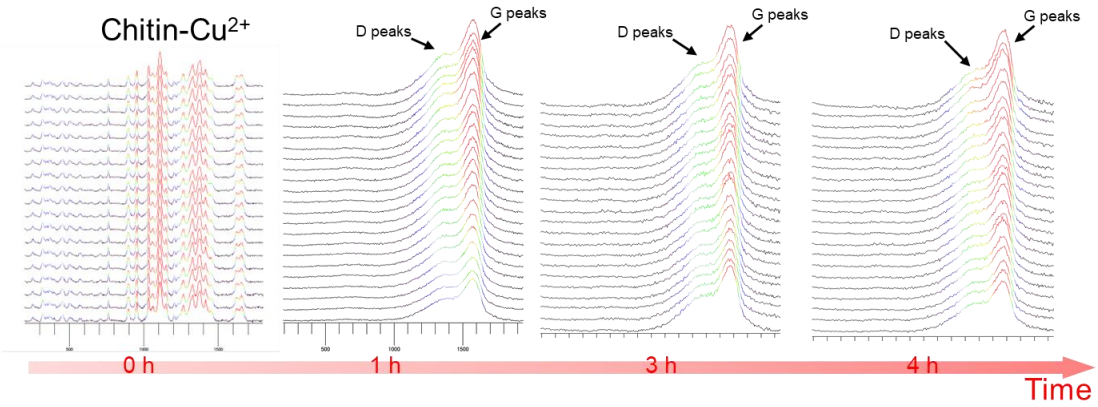


Figure S7. Representative Raman spectra of the composite aerogel treated with different pyrolysis times.

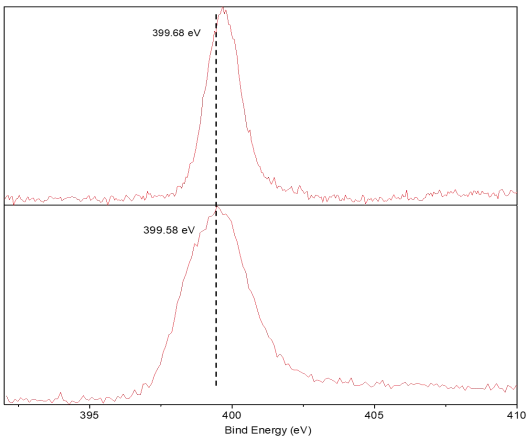


Figure S8. N 1s spectra of composite carbon aerogels, chitin/Cu aerogels

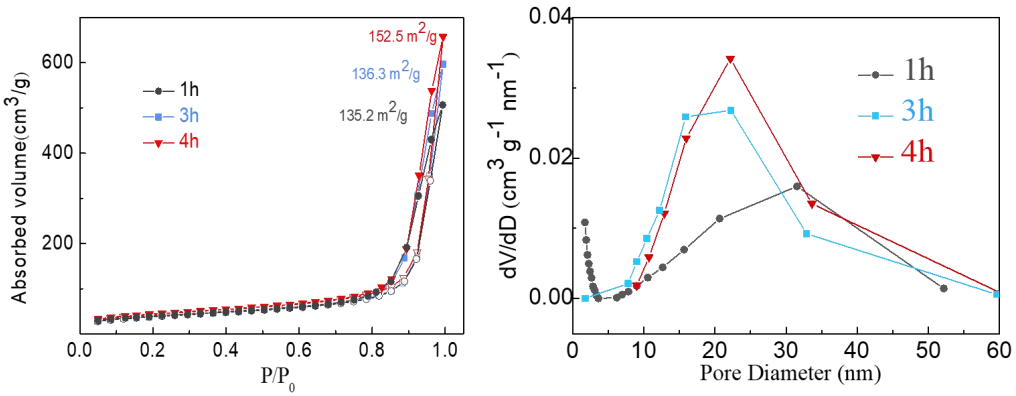


Figure S9. Nitrogen adsorption-desorption isotherms and pore size distributions of aerogel with different carbonization treatment times

2.4 $Cu^{(0)}$@CAC and $Cu^{(I)}$(OTf)@CAC with different pyrolysis times preparation

First, 1 g chitin aerogels were carbonized under a nitrogen atmosphere at 250 °C (heating rate is 5 °C/min and cooling rate is 10 °C/min) for 1h to obtain chitin carbon aerogel (CAC)-1h, and 1 g chitin aerogels were carbonized under a nitrogen atmosphere at 250 °C (heating rate is 5 °C/min and cooling rate is 10 °C/min) for 3h to obtain CAC-3h, then, at the same procedure, chitin aerogels were carbonized for 4h to obtain CAC-4h.

Subsequently, 500 mg CAC-1h was mixed with 5 mg $Cu^{(0)}$ powder and $Cu^{(I)}$(OTf) powder by grinding thoroughly to fabricate $Cu^{(0)}$@chitin carbon aerogel catalyst ($Cu^{(0)}$@CAC-1h) and $Cu^{(I)}$@CAC-1h. Whatmore, CAC-3h and CAC-4h were treated as the same procedure to obtain $Cu^{(0)}$@CAC-3h, $Cu^{(I)}$(OTf)@CAC-3h, and $Cu^{(0)}$@CAC-4h, $Cu^{(I)}$(OTf)@CAC-4h, respectively.

2.5 $RuCl_3$@CAC preparation

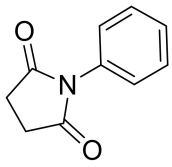
First, 500 mg of the chitin gel were replaced solvent with 50 mL acetone to obtain chitin organogels which immersed into 50 mL acetone solution with 8.93 mg of the $RuCl_3$ powder, while stirring for 5h. Subsequently, the chitin/Ru gels were washed and replaced with 50 mL acetone and 50 mL tert-butanol for 24h, respectively. Then the chitin/Ru gels were immersed in a box filled with liquid nitrogen and the chitin/Ru aerogels were obtained by freeze-drying (Christ Alpha 2-4 LDplus, -80 °C, 0.045 atm). Finally, 120 mg of the chitin/Cu aerogels were carbonized under a nitrogen atmosphere at 250 °C (heating rate is 5 °C/min and cooling rate is 10 °C/min) for 4h to obtain $RuCl_3$@chitin carbon aerogel catalyst ($RuCl_3$@CAC).

III. General procedures for $Cu(OTf)_2$@CAC-catalyzed reduction of maleimides

$Cu(OTf)_2$@CAC (1.2 mol%, 20 mg), maleimide (0.25 mmol), $PhSiH_3$ (0.5 mmol, 62 μL), and THF (1 mL) were introduced in tube under air, equipped with magnetic stirring bar and was stirred at 60 °C. After 12 h, the conversion of the reaction was analyzed by gas chromatography. The solvent was then evaporated under vacuum and the desired product was purified by using a silica gel column chromatography and a mixture of petrol ether/ethyl acetate as eluent.

IV. Characterization data for all compounds

1-phenylpyrrolidine-2,5-dione[1] (3a)

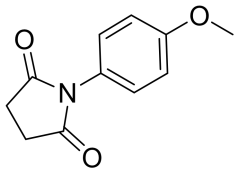


White solid, yield = 82%, 36 mg

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.521-7.516 (m, 2H), 7.44-7.41 (m, 1H), 7.32-7.29 (m, 2H), 2.91 (s, 4H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 176.3, 131.9, 129.3, 128.7, 126.5, 28.5.

1-(4-methoxyphenyl)pyrrolidine-2,5-dione[1] (3b)

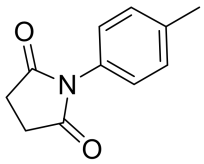


Light yellow solid, yield = 75%, 38 mg

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.20 (d, 2H, *J* = 9.0 Hz), 7.00 (d, 2H, *J* = 9.0 Hz), 3.83 (s, 3H), 2.88 (s, 4H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 176.6, 159.5, 127.7, 124.5, 114.6, 55.5, 28.4.

1-(p-tolyl)pyrrolidine-2,5-dione[1] (3c)

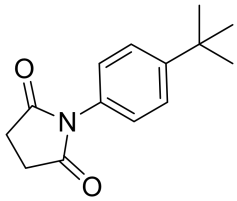


White solid, yield = 80%, 38 mg.

^{1}H NMR (500 MHz, DMSO): δ = 7.29 (d, 2H, *J* = 8.0 Hz), 7.14 (d, 2H, *J* = 8.0 Hz), 2.77 (s, 4H), 2.35 (s, 3H).

^{13}C NMR (125 MHz, DMSO): δ = 177.5, 138.1, 130.6, 129.7, 127.4, 28.9, 21.2.

1-(4-(tert-butyl)phenyl)pyrrolidine-2,5-dione[2] (3d)

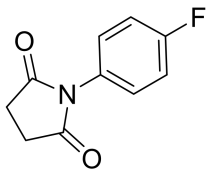


White solid, yield = 86%, 50 mg.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.51 (d, 2H, *J* = 8.5 Hz), 7.22 (d, 2H, *J* = 8.5 Hz), 2.89 (s, 4H), 1.35 (s, 9H).

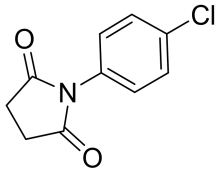
^{13}C NMR (125 MHz, $CDCl_3$): δ = 176.5, 151.7, 129.1, 126.3, 125.9, 34.8, 31.3, 28.4.

1-(4-fluorophenyl)pyrrolidine-2,5-dione[3] (3e)



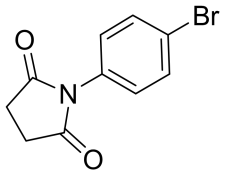
White solid, yield = 92%, 44 mg.
^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.32-7.29 (m, 2H), 7.21-7.17 (m, 2H), 2.93 (s, 4H).
^{13}C NMR (125 MHz, $CDCl_3$): δ = 176.1, 162.2 (d, J_{CF} = 247.50 Hz), 128.3 (d, J_{CF} = 8.75 Hz), 127.8 (d, J_{CF} = 3.38 Hz), 116.3 (d, J_{CF} = 22.75 Hz), 28.4.
^{19}F NMR (470 MHz, $CDCl_3$): δ = -112.2.

1-(4-chlorophenyl)pyrrolidine-2,5-dione[1] (3f)



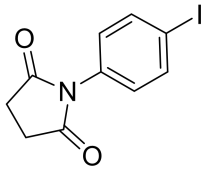
White solid, yield = 75%, 39 mg.
^{1}H NMR (500 MHz, DMSO): δ = 7.57 (d, 2H, J = 8.5 Hz), 7.31 (d, 2H, J = 9.0 Hz), 2.92 (s, 4H).
^{13}C NMR (125 MHz, DMSO): δ = 177.2, 133.0, 132.0, 129.35, 129.33, 29.0.

1-(4-bromophenyl)pyrrolidine-2,5-dione[1] (3g)



White solid, yield = 74%, 47 mg.
^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.62 (d, 2H, J = 8.5 Hz), 7.22 (d, 2H, J = 8.5 Hz), 2.92-2.90 (m, 4H).
^{13}C NMR (125 MHz, $CDCl_3$): δ = 169.1, 134.3, 132.3, 130.3, 127.4, 121,6.

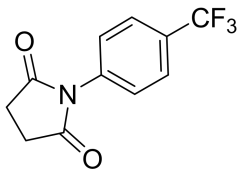
1-(4-iodophenyl)pyrrolidine-2,5-dione[4] (3h)



White solid, yield = 55%, 42 mg.
^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.83 (d, 2H, J = 8.5 Hz), 7.09 (d, 2H, J = 8.5 Hz), 2.92 (s, 4H).

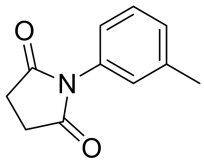
^{13}C NMR (125 MHz, $CDCl_3$): δ = 175.8, 138.4, 131.6, 128.1, 94.1, 28.4.

1-(4-(trifluoromethyl)phenyl)pyrrolidine-2,5-dione[4] (3i)



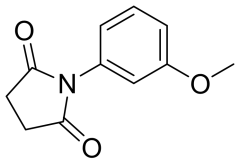
White solid, yield = 40%, 25 mg.
^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.75 (d, 2H, *J* = 8.5 Hz), 7.48 (d, 2H, *J* = 8.0 Hz), 2.94 (s, 4H).
^{13}C NMR (125 MHz, $CDCl_3$): δ = 175.8, 126.8, 126.5, 126.4, 28.6.

1-(*m*-tolyl)pyrrolidine-2,5-dione[5] (3j)



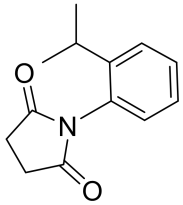
White solid, yield = 90%, 43 mg.
^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.39-7.36 (m, 1H), 7.23 (d, 1H, *J* = 7.5 Hz), 7.10-7.07 (m, 2H), 2.88 (s, 4H) , 2.41 (s, 3H).
^{13}C NMR (125 MHz, $CDCl_3$): δ = 176.4, 139.3, 129.6, 129.1, 127.2, 123.6, 28.5, 21.4.

1-(3-methoxyphenyl)pyrrolidine-2,5-dione[6] (3k)



White solid, yield = 79%, 41 mg.
^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.40 (t, 1H, *J* = 8.0 Hz), 6.97-6.83 (m, 3H), 3.83 (s, 3H), 2.90 (s, 4H).
^{13}C NMR (125 MHz, $CDCl_3$): δ = 176.2, 160.1, 132.9, 130.0, 118.8, 114.6, 112.4, 55.4, 28.5.

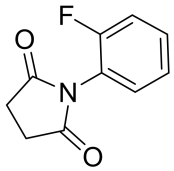
1-(2-isopropylphenyl)pyrrolidine-2,5-dione[7] (3l)



White solid, yield = 72%, 39 mg.
^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.47-7.45 (m, 2H), 7.33-7.30 (m, 1H), 7.04-7.02 (m, 1H), 2.95-2.94 (m, 4H), 2.71-2.65 (m, 1H), 1.22 (s, 3H) , 1.21 (s, 3H).

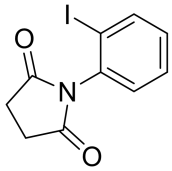
^{13}C NMR (125 MHz, $CDCl_3$): δ = 176.8, 146.2, 130.1, 129.7, 128.2, 127.0, 126.8, 28.7, 28.6, 23.7.

1-(2-fluorophenyl)pyrrolidine-2,5-dione (3m)



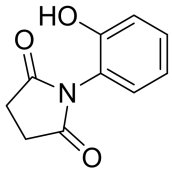
White solid, yield = 85%, 41 mg.
^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.46-7.43 (m, 1H), 7.29-7.23 (m, 3H), 2.95 (d, 4H, J = 9.0 Hz).
^{13}C NMR (125 MHz, $CDCl_3$): δ = 175.4, 157.4 (d, J_{CF} = 250.88 Hz), 131.1 (d, J_{CF} = 8.13 Hz), 129.3, 124.7 (d, J_{CF} = 4.00 Hz), 119.7 (d, J_{CF} = 13.00 Hz), 116.8 (d, J_{CF} = 19.63 Hz), 28.6.
^{19}F NMR (470 MHz, $CDCl_3$): δ = -119.4.

1-(2-iodophenyl)pyrrolidine-2,5-dione[3] (3n)



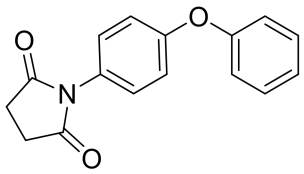
White solid, yield = 66%, 50 mg.
^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.97 (dd, 1H, J = 8.0,1.5 Hz), 7.51-7.48 (m, 1H), 7.22-7.18 (m, 2H), 3.03-2.91 (m, 4H).
^{13}C NMR (125 MHz, $CDCl_3$): δ = 175.4, 140.0, 135.4, 131.1, 129.6, 129.4, 97.9, 28.9.

1-(2-hydroxyphenyl)pyrrolidine-2,5-dione[8] (3o)



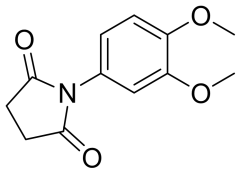
White solid, yield = 87%, 42 mg.
^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.31-7.29 (m, 1H), 7.15 (dd, 1H, J = 8.0,1.5 Hz), 7.05-7.02 (m, 1H), 6.99 (dd, 1H, J = 8.0,1.5 Hz), 2.88 (s, 4H) , 1.28 (s, 1H).
^{13}C NMR (125 MHz, $CDCl_3$): δ = 177.2, 151.2, 130.7, 128.3, 121.2, 119.8, 118.6, 28.6.

1-(4-phenoxyphenyl)pyrrolidine-2,5-dione (3p)



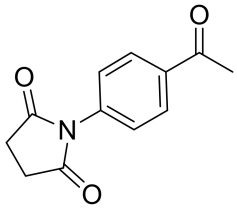
White solid, yield = 67%, 45 mg.
^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.41-7.36 (m, 2H), 7.25 (d, 2H, *J* = 9.0 Hz), 7.17 (t, 1H, *J* = 7.5 Hz), 7.11-7.07 (m, 4H), 2.78 (s, 4H).
^{13}C NMR (125 MHz, $CDCl_3$): δ = 176.4, 157.7, 156.3, 129.9, 127.9, 126.4, 124.0, 119.7, 118.8, 28.4.

1-(3,4-dimethoxyphenyl)pyrrolidine-2,5-dione (3q)



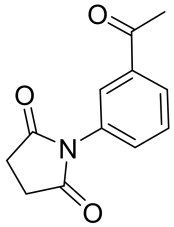
White solid, yield = 85%, 50 mg.
^{1}H NMR (500 MHz, $CDCl_3$): δ = 6.95 (d, 1H, *J* = 8.5 Hz), 6.84 (dd, 1H, *J* = 8.5, 2.5 Hz), 6.78 (d, 1H, *J* = 2.5Hz), 3.91 (s, 3H), 3.88 (s, 3H), 2.90 (s, 4H).
^{13}C NMR (125 MHz, $CDCl_3$): δ = 176.5, 149.3, 149.2, 124.6, 119.1, 111.2, 109.9, 56.1, 56.0, 28.4.

1-(4-acetylphenyl)pyrrolidine-2,5-dione[9] (3r)



White solid, yield = 83%, 45 mg.
^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.08 (d, 2H, *J* = 9.0 Hz), 7.47 (d, 2H, *J* = 8.5 Hz), 2.95 (s, 4H), 2.64 (s, 3H).
^{13}C NMR (125 MHz, $CDCl_3$): δ = 197.1, 175.7, 136.7, 135.9, 129.2, 126.4, 28.5, 26.7.

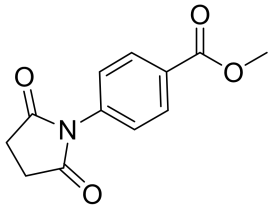
1-(3-acetylphenyl)pyrrolidine-2,5-dione (3s)



White solid, yield = 80%, 43 mg.

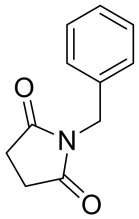
^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.01 (d, 1H, *J* = 7.5 Hz), 7.92 (t, 1H, *J* = 2.0 Hz), 7.62 (t, 1H, *J* = 8.0 Hz), 7.54-7.52 (m, 1H), 2.96 (s, 4H), 2.64 (s, 3H).
^{13}C NMR (125 MHz, $CDCl_3$): δ = 196.9, 175.9, 138.1, 132.4, 131.0, 129.6, 128.4, 126.5, 28.5, 26.7.

methyl 4-(2,5-dioxopyrrolidin-1-yl)benzoate[10] (3t)



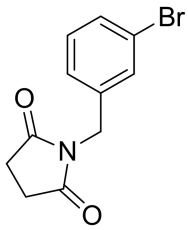
White solid, yield = 69%, 40 mg.
^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.17 (d, 2H, *J* = 8.5 Hz), 7.44 (d, 2H, *J* = 8.5 Hz), 3.95 (s, 3H), 2.94 (s, 4H).
^{13}C NMR (125 MHz, $CDCl_3$): δ = 175.7, 166.2, 135.9, 130.49, 130.04, 126.2, 52.4, 28.4.

1-benzylpyrrolidine-2,5-dione[3] (3u)



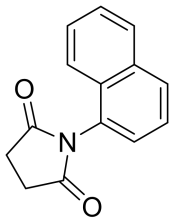
White solid, yield = 74%, 35 mg.
^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.42-7.40 (m, 2H), 7.35-7.30 (m, 3H), 4.68 (s, 2H), 2.72 (s, 4H).
^{13}C NMR (125 MHz, $CDCl_3$): δ = 176.9, 135.8, 128.9, 128.7, 128.0, 42.4, 28.2.

1-(3-bromobenzyl)pyrrolidine-2,5-dione[11] (3v)



White solid, yield = 83%, 56 mg.
^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.55 (s, 1H), 7.43 (d, 1H, *J* = 8.5 Hz), 7.35 (d, 1H, *J* = 7.5 Hz), 7.22-7.19 (m, 1H), 4.64 (s, 2H), 2.75 (s, 4H).
^{13}C NMR (125 MHz, $CDCl_3$): δ = 176.7, 137.8, 131.8, 131.2, 130.2, 127.6, 122.7, 41.8, 28.2.

1-(naphthalen-1-yl)pyrrolidine-2,5-dione (3w)

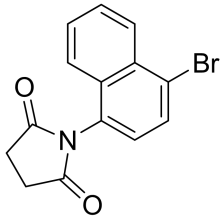


White solid, yield = 71%, 40 mg.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.00-7.95 (m, 2H), 7.61-7.53 (m, 4H), 7.36 (dd, 1H, *J* = 7.0,1.0 Hz), 3.12-2.99 (m, 4H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 176.6, 134.5, 130.1, 129.3, 128.8, 128.6, 127.3, 126.7, 126.3, 125.4, 121.8, 28.8.

1-(4-bromonaphthalen-1-yl)pyrrolidine-2,5-dione (3x)

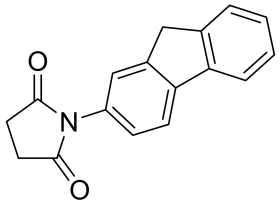


White solid, yield = 51%, 39 mg.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.35 (d, 1H, *J* = 8.5 Hz), 7.91 (d, 1H, *J* = 8.0 Hz), 7.68 (t, 1H, *J* = 7.5 Hz), 7.60 (t, 1H, *J* = 7.0 Hz), 7.52 (d, 1H, *J* = 8.0Hz), 7.44 (d, 1H, *J* = 8.0 Hz), 3.14-3.01 (m, 4H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 176.3, 132.9, 130.4, 129.5, 128.5, 128.2, 128.1, 128.0, 126.7, 125.0, 122.4, 28.9.

1-(9H-fluoren-2-yl)pyrrolidine-2,5-dione (3y)

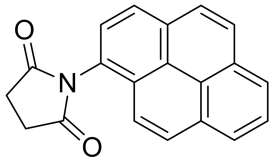


White solid, yield = 79%, 52 mg.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.89 (d, 1H, *J* = 8.0 Hz), 7.82 (d, 1H, *J* = 7.5 Hz), 7.58 (d, 1H, *J* = 7.5 Hz),7.48 (s, 1H), 7.43-7.30 (m, 3H), 3.97(s, 2H), 2.96(s, 4H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 176.5, 144.1, 143.6, 142.3, 140.7, 130.2, 127.3, 126.9, 125.3, 125.1, 123.3, 120.4, 120.3, 37.0, 28.5.

1-(pyren-1-yl)pyrrolidine-2,5-dione (3z)

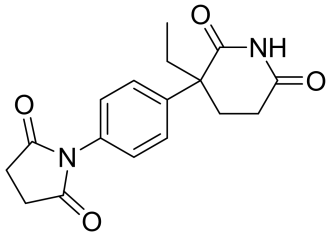


White solid, yield = 61%, 46 mg.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.30-8.24 (m, 3H), 8.18-8.06 (m, 4H), 7.84 (d, 1H, *J* = 8.0 Hz), 7.74 (d, 1H, *J* = 9.5 Hz), 3.22-3.10 (m, 4H).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 176.7, 132.2, 131.0, 130.7, 129.1, 128.6, 127.4, 127.1, 126.5, 126.2, 126.0, 125.6, 125.4, 125.2, 124.4, 121.1, 28.9.

3-(4-(2,5-dioxopyrrolidin-1-yl)phenyl)-3-ethylpiperidine-2,6-dione (3aa)

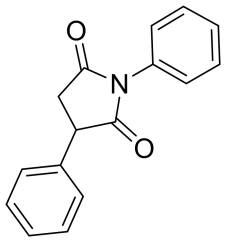


White solid, yield = 54%, 42 mg.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.42 (d, 2H, *J* = 8.5 Hz), 7.56 (d, 2H, *J* = 8.5 Hz), 2.93 (s, 4H), 2.66-2.61 (m, 1H), 2.49-2.39 (m, 2H), 2.31-2.25 (m, 1H), 2.14-2.06 (m, 1H), 1.97-1.90 (m, 1H), 0.91 (t, 3H, *J* = 7.5 Hz).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 176.1, 174.1, 171.2, 139.2, 131.3, 127.1, 126.8, 51.0, 33.0, 29.3, 28.4, 26.9, 9.1.

1,3-diphenylpyrrolidine-2,5-dione (3ab)

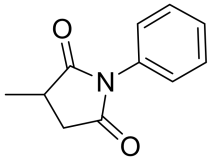


White solid, yield = 61%, 38 mg.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.53-7.49 (m, 2H), 7.45-7.42 (m, 3H), 7.39-7.33 (m, 5H), 4.23 (dd, 1H, *J* = 9.5,5.0 Hz), 3.44-3,38 (m, 1H), 3.04 (dd, 1H, *J* = 18.5,5.0 Hz).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 176.7, 175.2, 137.2, 131.9, 129.3, 129.2, 128.8, 128.1, 127.4, 126.5, 46.0, 37.3.

3-methyl-1-phenylpyrrolidine-2,5-dione (3ac)



White solid, yield = 93%, 44 mg.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.51-7.48 (m, 2H), 7.43-7.40 (m, 1H), 7.35-7.30 (m, 2H), 3.14-3.02 (m, 2H), 2.55-2.51 (m, 1H), 1.475 (d, 3H, *J* = 7.0 Hz).

^{13}C NMR (125 MHz, $CDCl_3$): δ = 179.6, 175.5, 132.0, 129.2, 128.6, 126.5, 36.7, 34.9, 16.9.

V. Reference

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VI. ^{1}H and ^{13}C NMR Spectra for all compounds

1-phenylpyrrolidine-2,5-dione (3a)

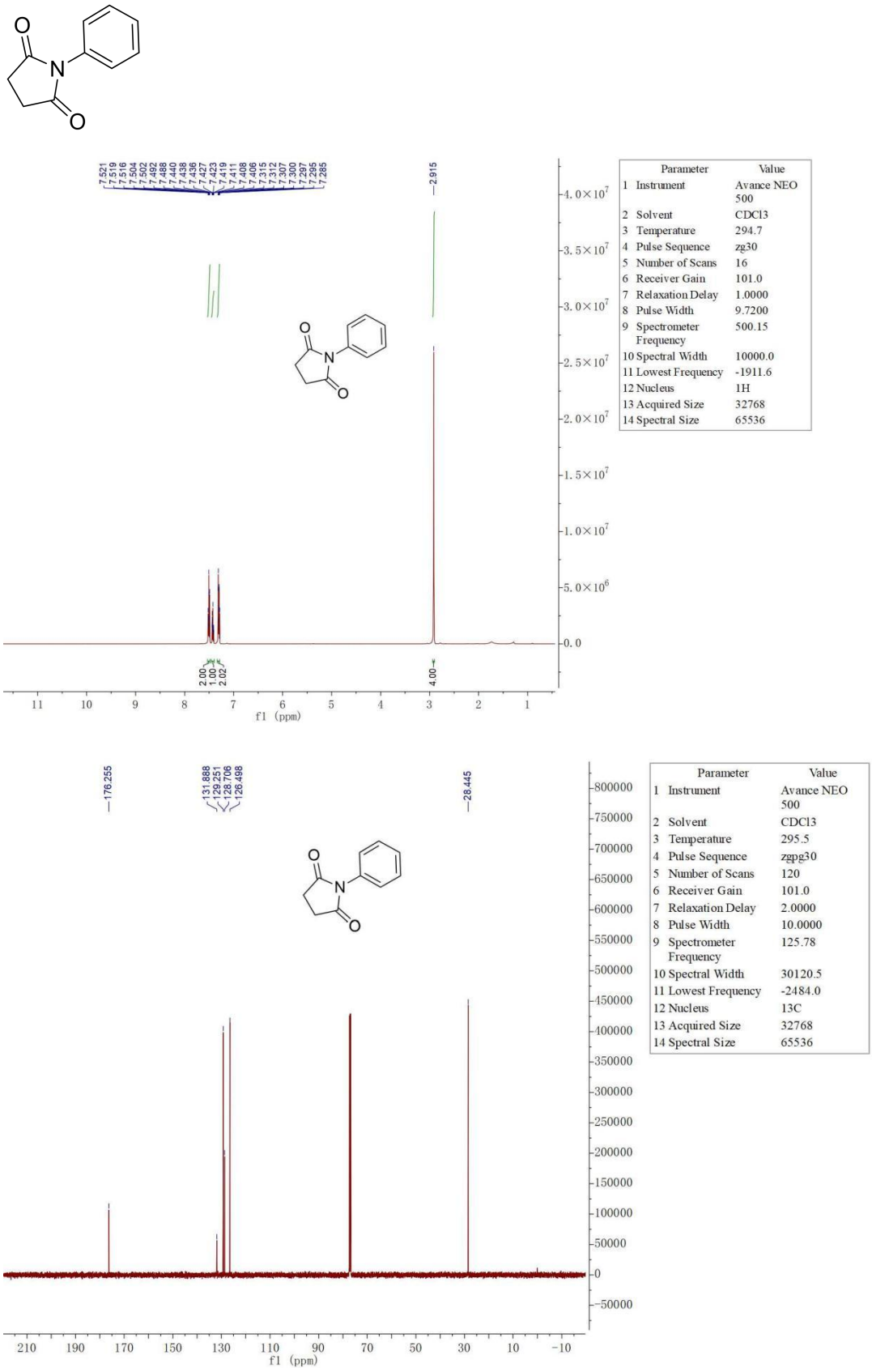
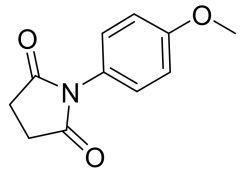


Figure S10. ^{1}H and ^{13}C spectrum of compound **3a.**

1-(4-methoxyphenyl)pyrrolidine-2,5-dione (3b)



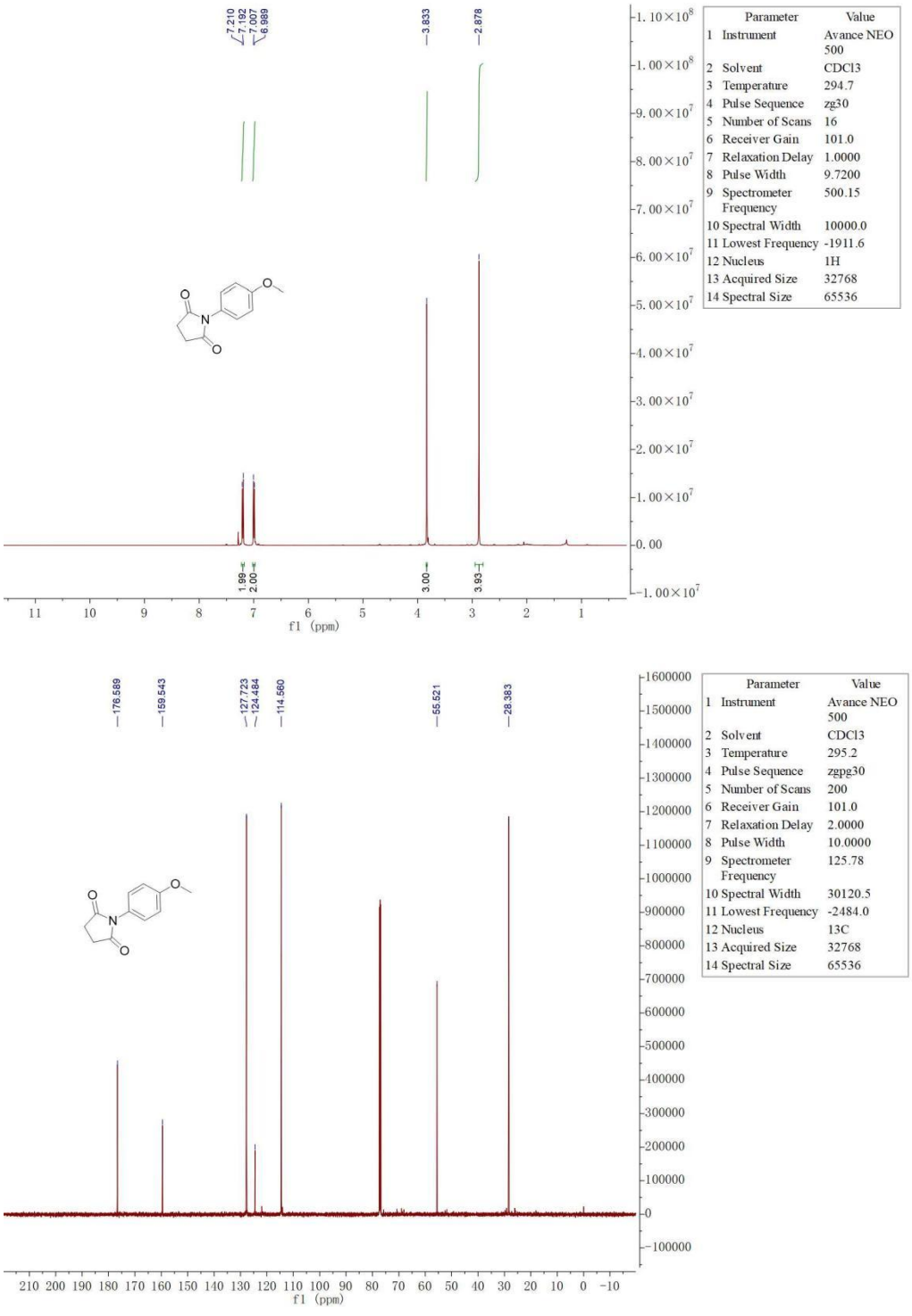


Figure S11. ^{1}H and ^{13}C spectrum of compound **3b.**

1-(p-tolyl)pyrrolidine-2,5-dione (3c)

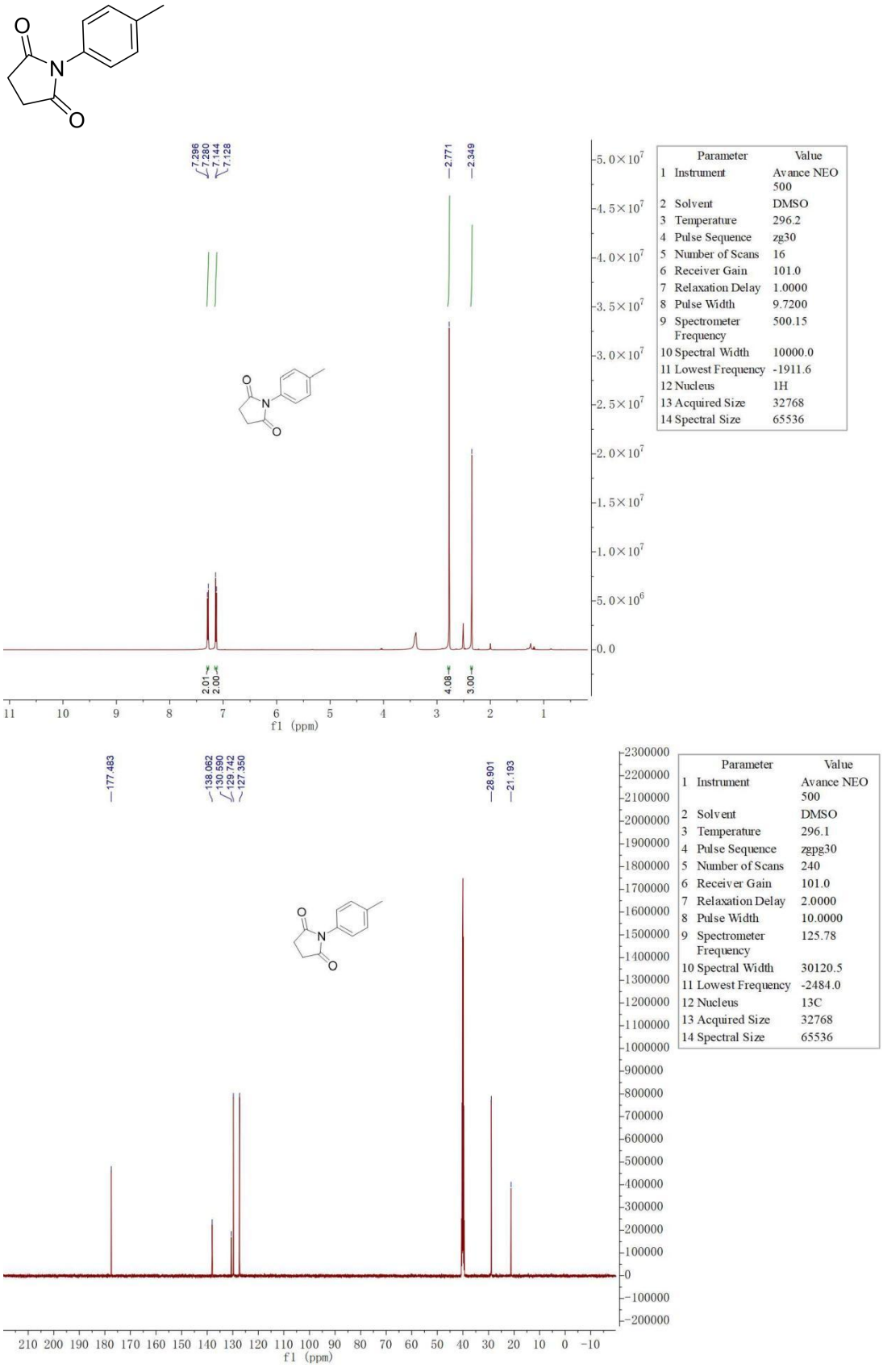
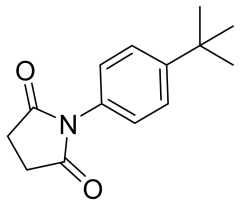
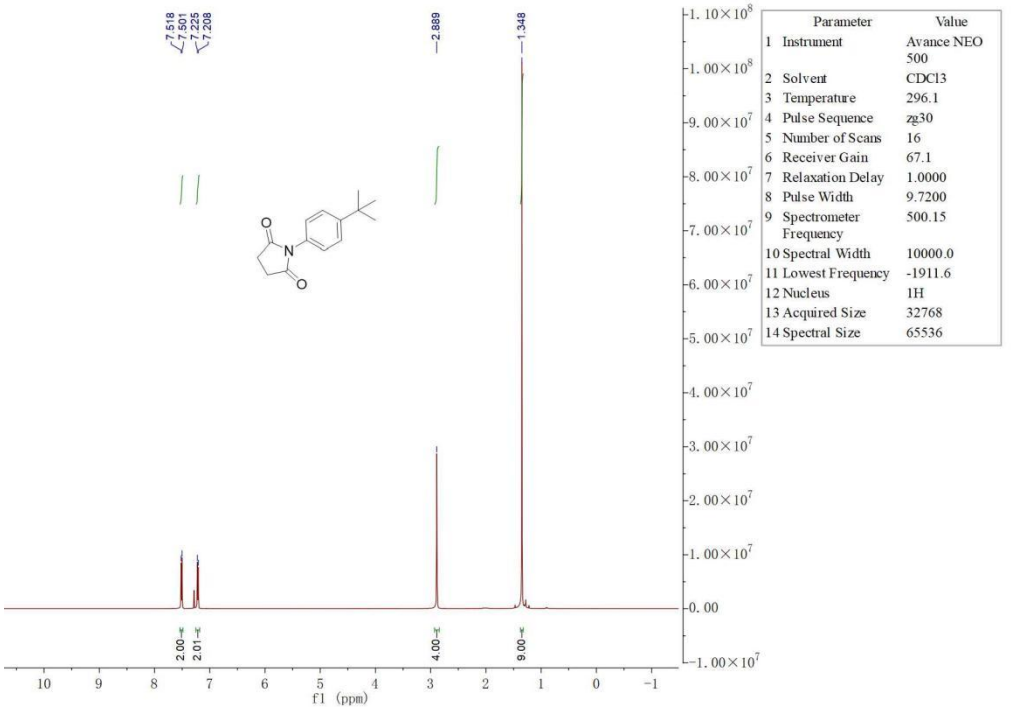


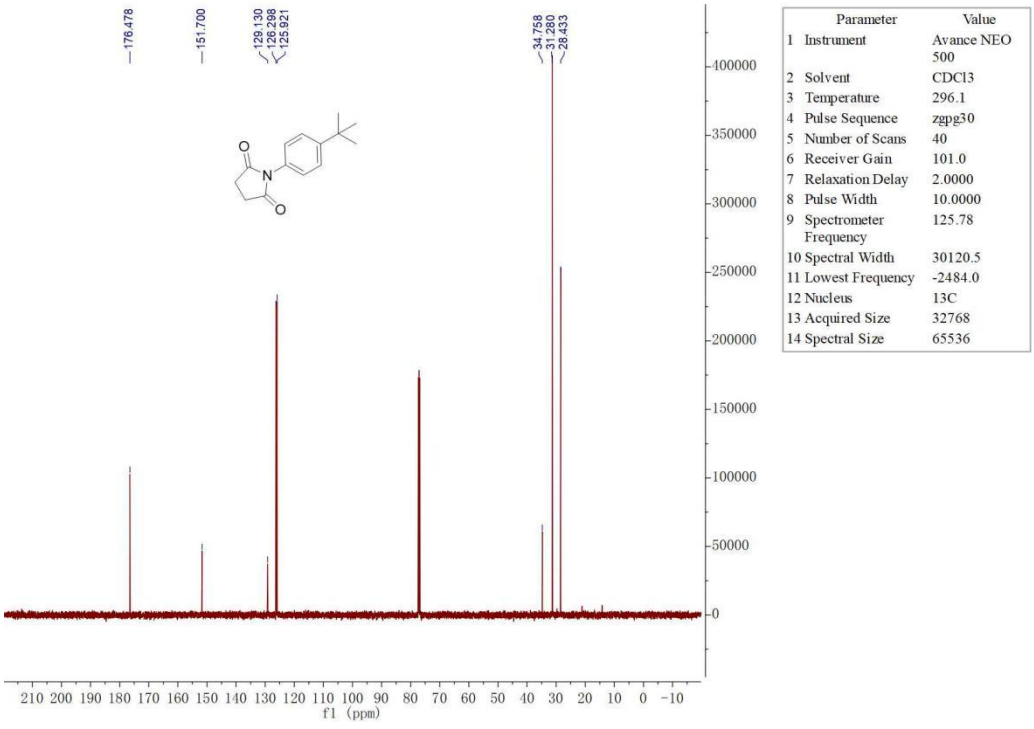
Figure S12. ^{1}H and ^{13}C spectrum of compound **3c.**

1-(4-(tert-butyl)phenyl)pyrrolidine-2,5-dione (3d)





	Parameter	Value
1	Instrument	Avance NEO 500
2	Solvent	CDCl3
3	Temperature	296.1
4	Pulse Sequence	zg30
5	Number of Scans	16
6	Receiver Gain	67.1
7	Relaxation Delay	1.0000
8	Pulse Width	9.7200
9	Spectrometer Frequency	500.15
10	Spectral Width	10000.0
11	Lowest Frequency	-1911.6
12	Nucleus	1H
13	Acquired Size	32768
14	Spectral Size	65536



	Parameter	Value
1	Instrument	Avance NEO 500
2	Solvent	CDCl3
3	Temperature	296.1
4	Pulse Sequence	zgpg30
5	Number of Scans	40
6	Receiver Gain	101.0
7	Relaxation Delay	2.0000
8	Pulse Width	10.0000
9	Spectrometer Frequency	125.78
10	Spectral Width	30120.5
11	Lowest Frequency	-2484.0
12	Nucleus	13C
13	Acquired Size	32768
14	Spectral Size	65536

Figure S13. ^{1}H and ^{13}C spectrum of compound **3d.**

1-(4-fluorophenyl)pyrrolidine-2,5-dione (3e)

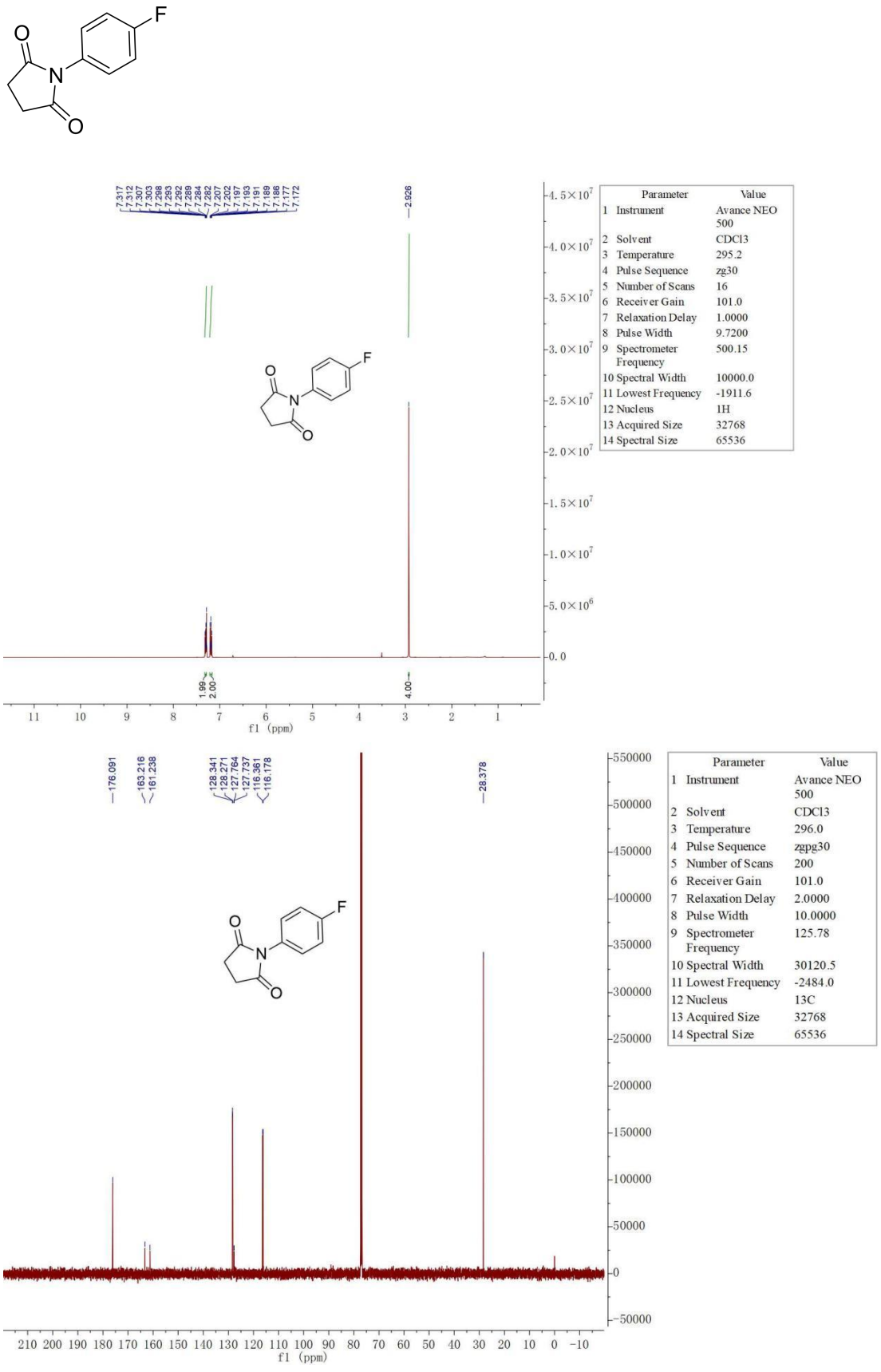
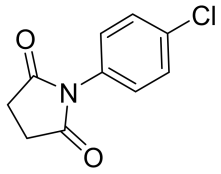
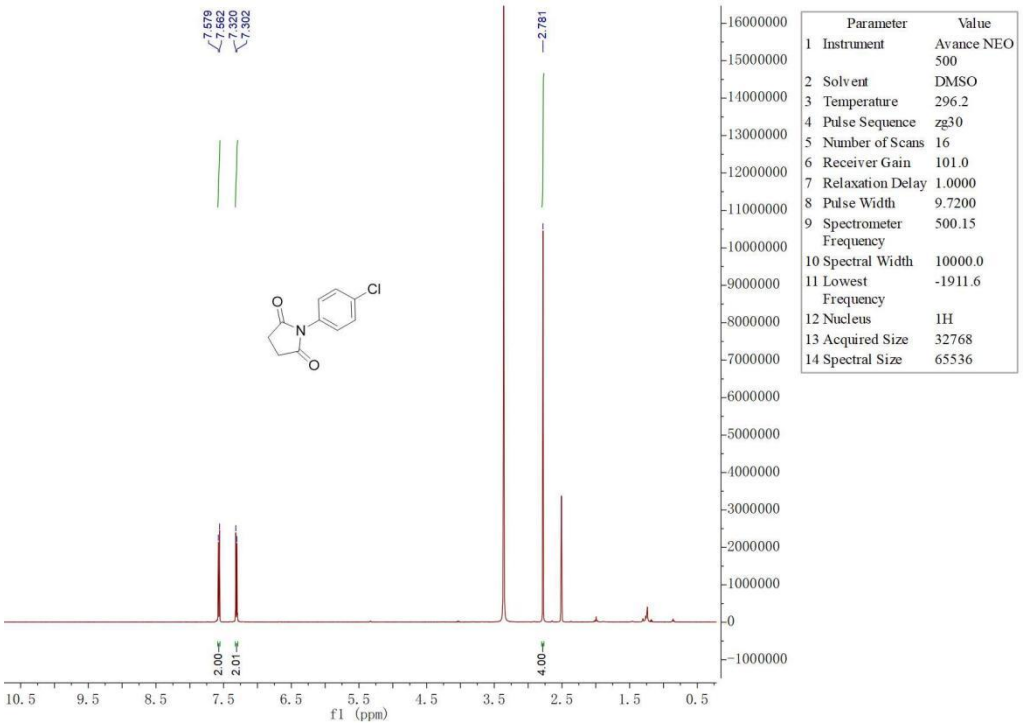


Figure S14. ^{1}H and ^{13}C spectrum of compound **3e.**

1-(4-chlorophenyl)pyrrolidine-2,5-dione (3f)





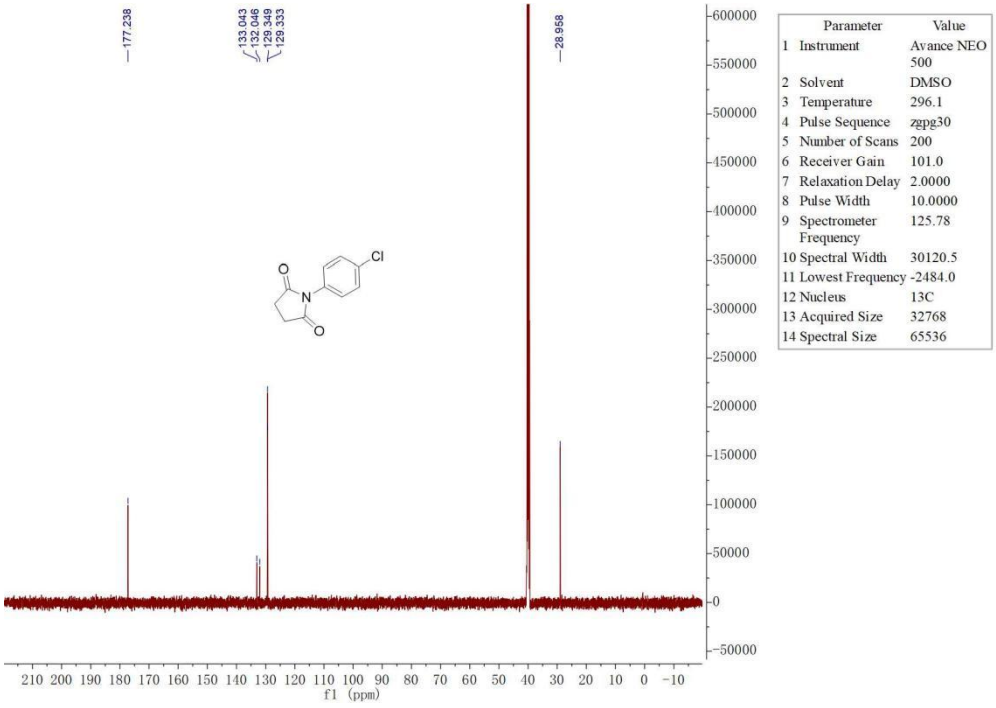


Figure S15. ^{1}H and ^{13}C spectrum of compound **3f.**

1-(4-bromophenyl)pyrrolidine-2,5-dione (3g)

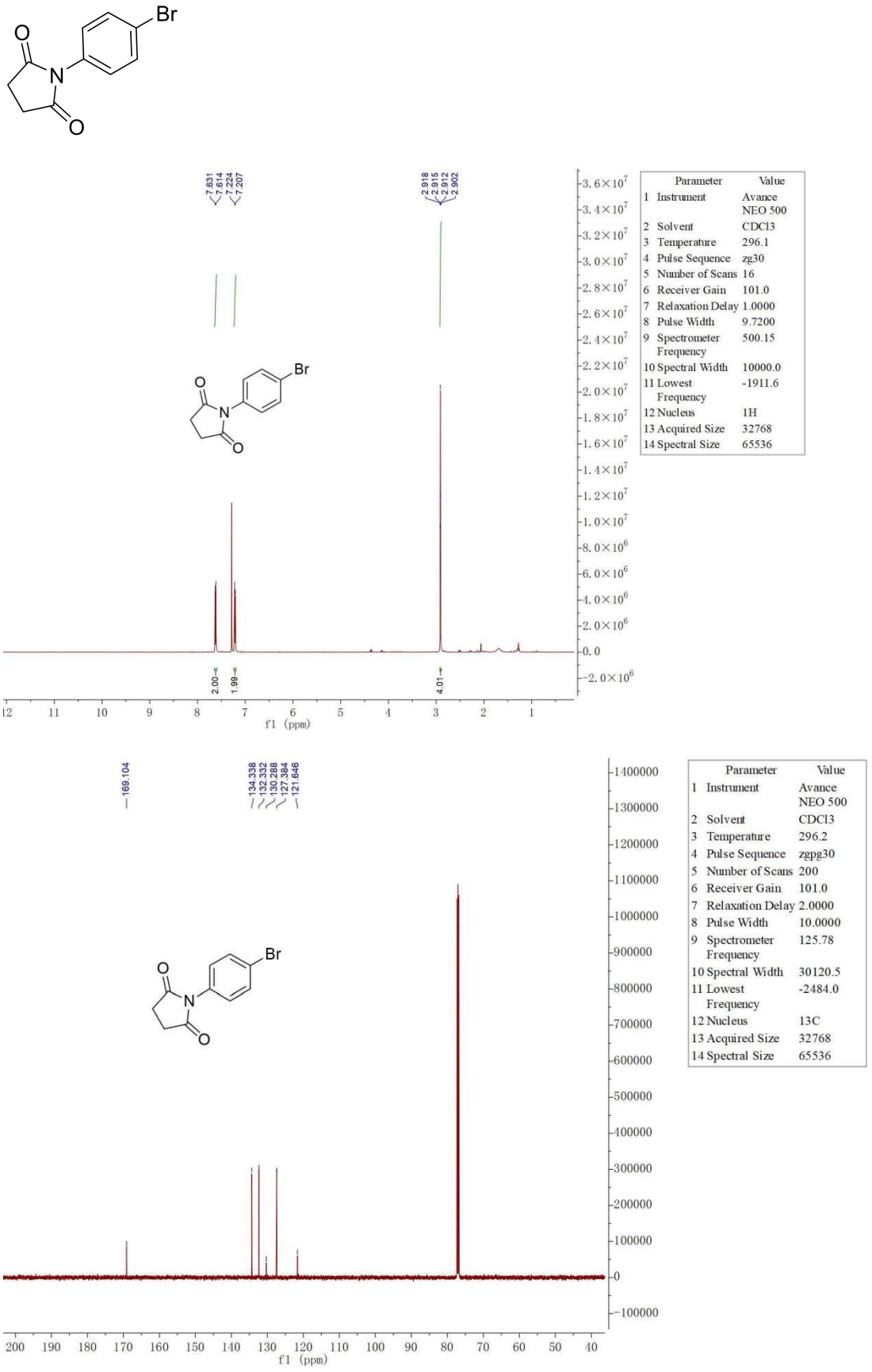


Figure S16. ^{1}H and ^{13}C spectrum of compound **3g.**

1-(4-iodophenyl)pyrrolidine-2,5-dione (3h)

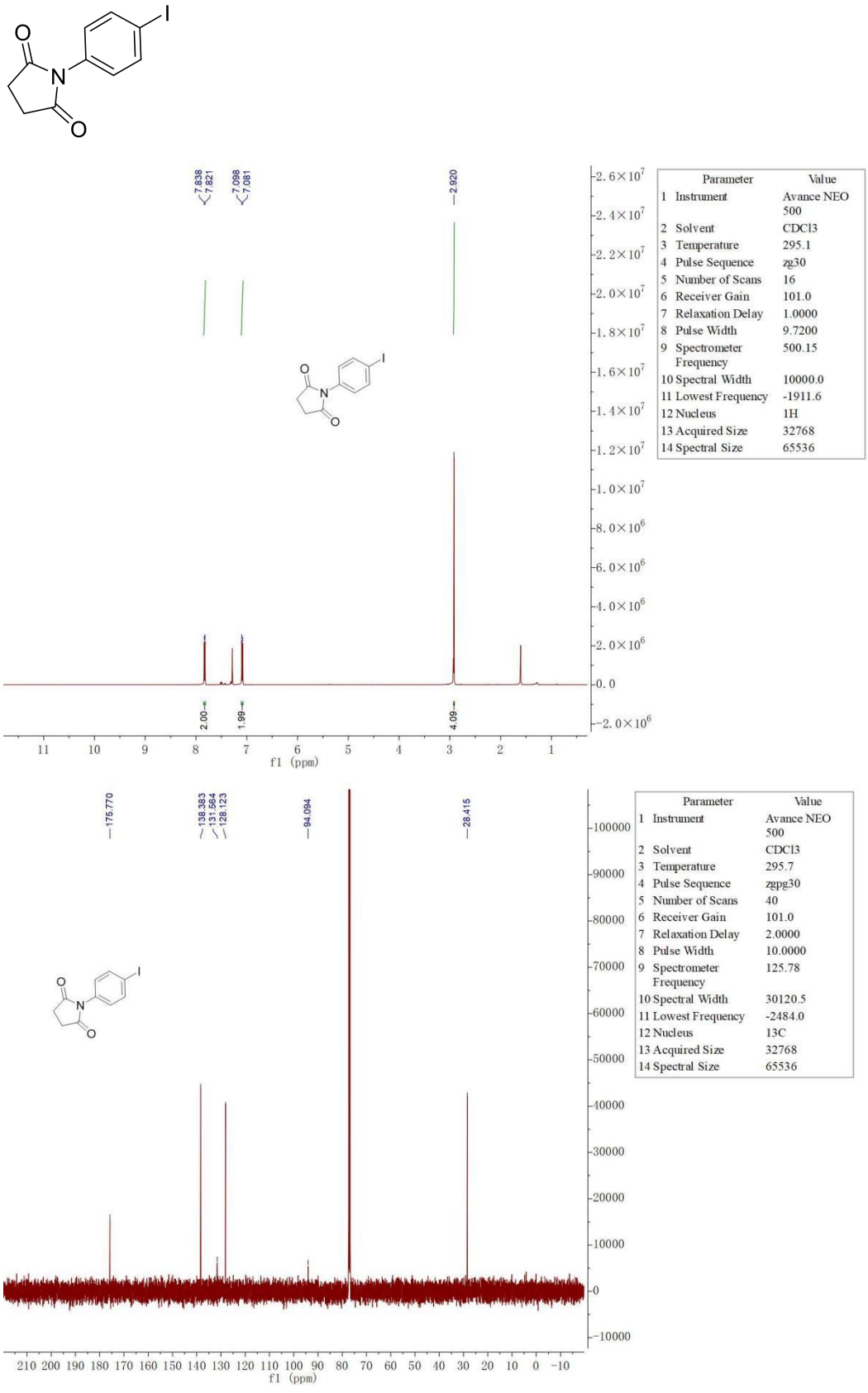
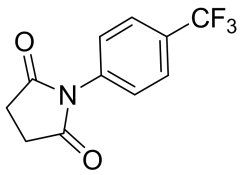


Figure S17. ^{1}H and ^{13}C spectrum of compound **3h.**

1-(4-(trifluoromethyl)phenyl)pyrrolidine-2,5-dione (3i)



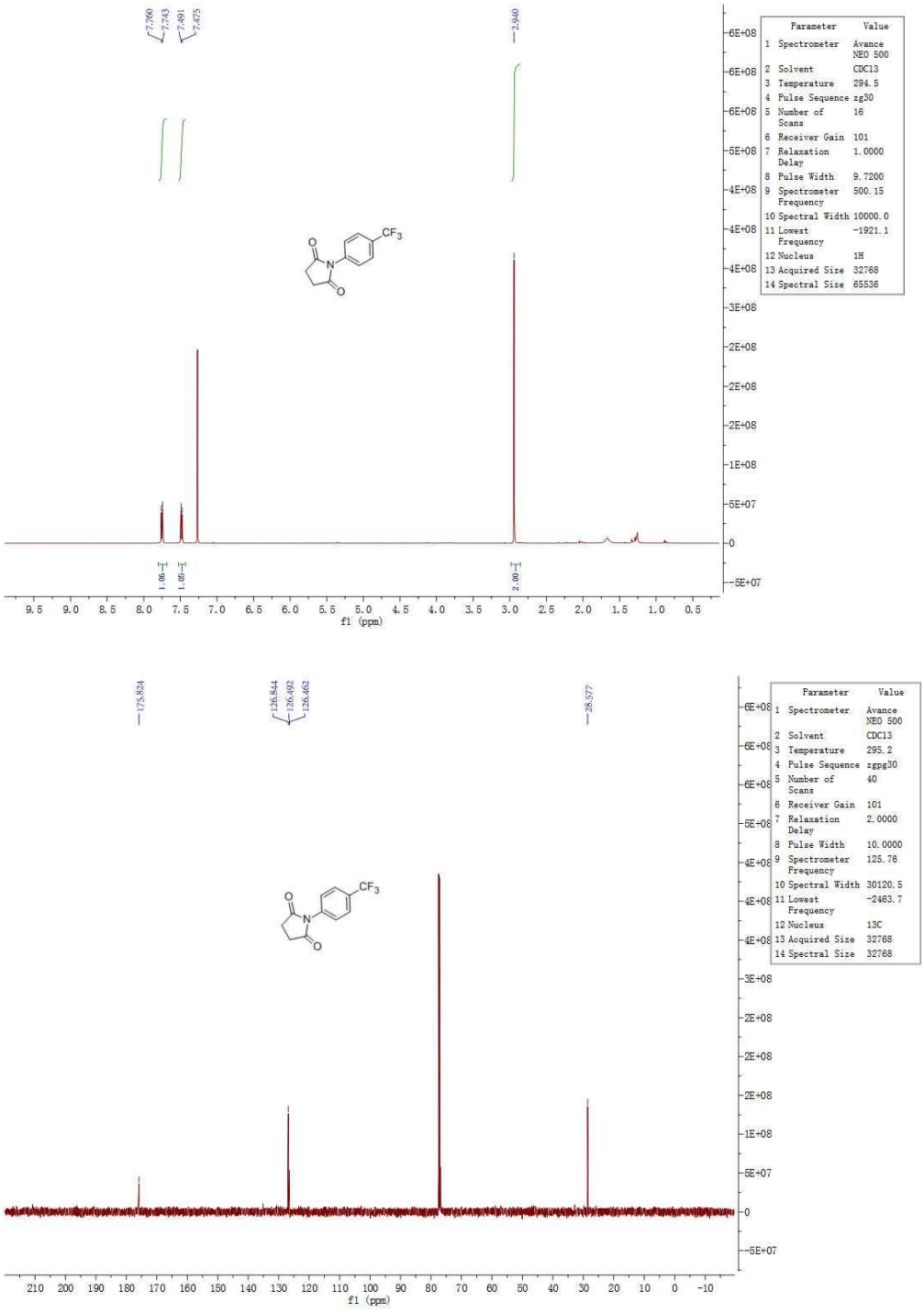


Figure S18. ^{1}H and ^{13}C spectrum of compound **3i.**

1-(*m*-tolyl)pyrrolidine-2,5-dione (3j)

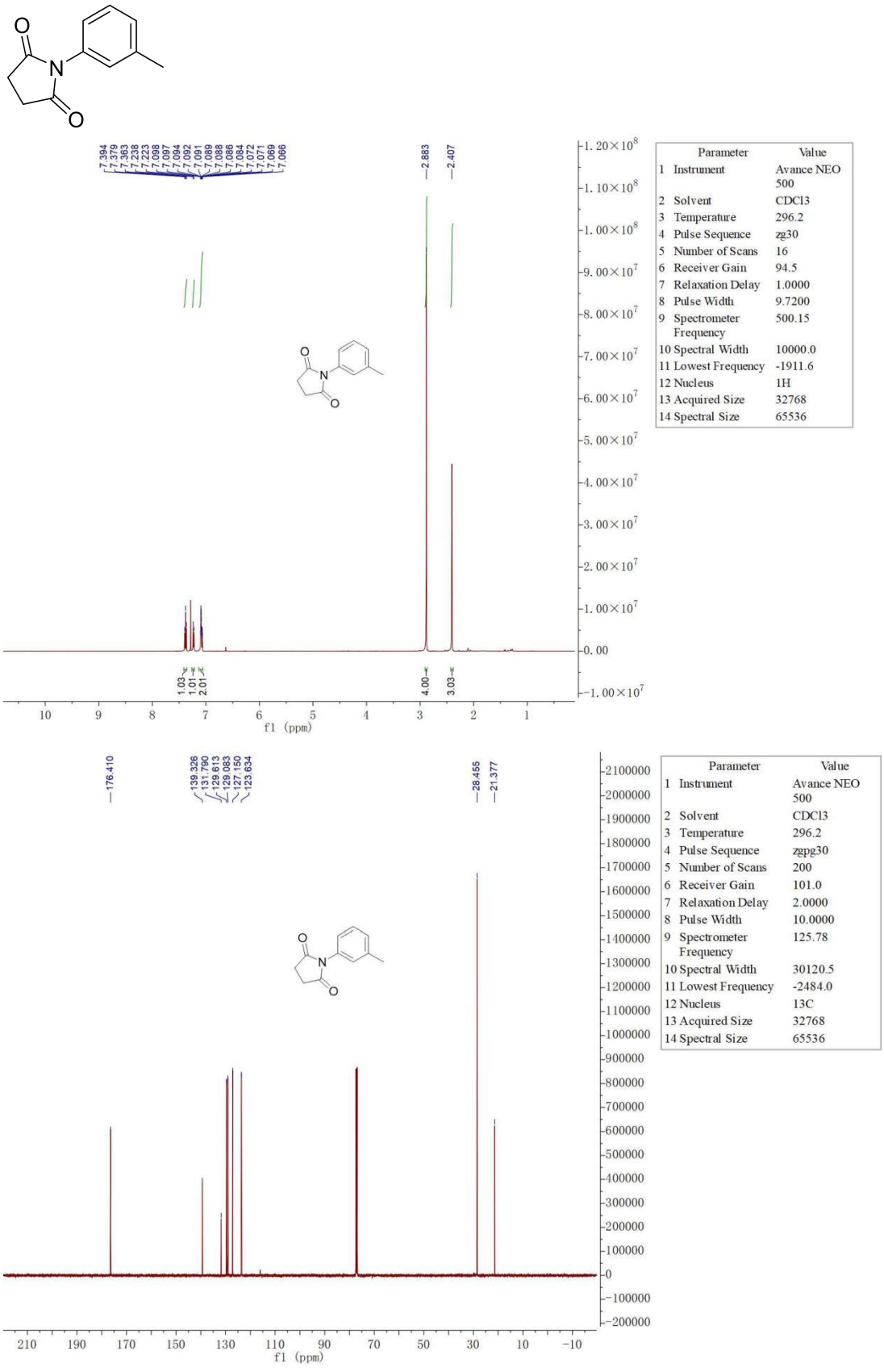
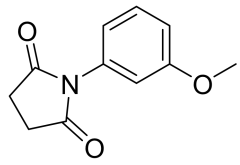
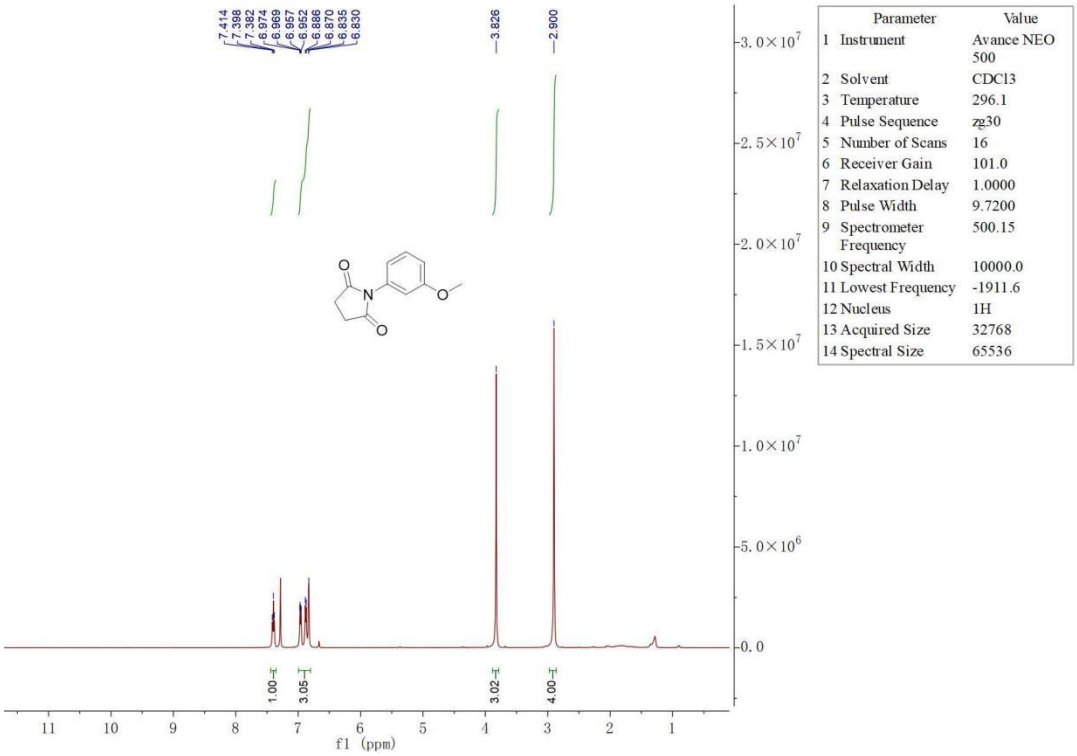


Figure S19. ^{1}H and ^{13}C spectrum of compound **3j.**

1-(3-methoxyphenyl)pyrrolidine-2,5-dione (3k)





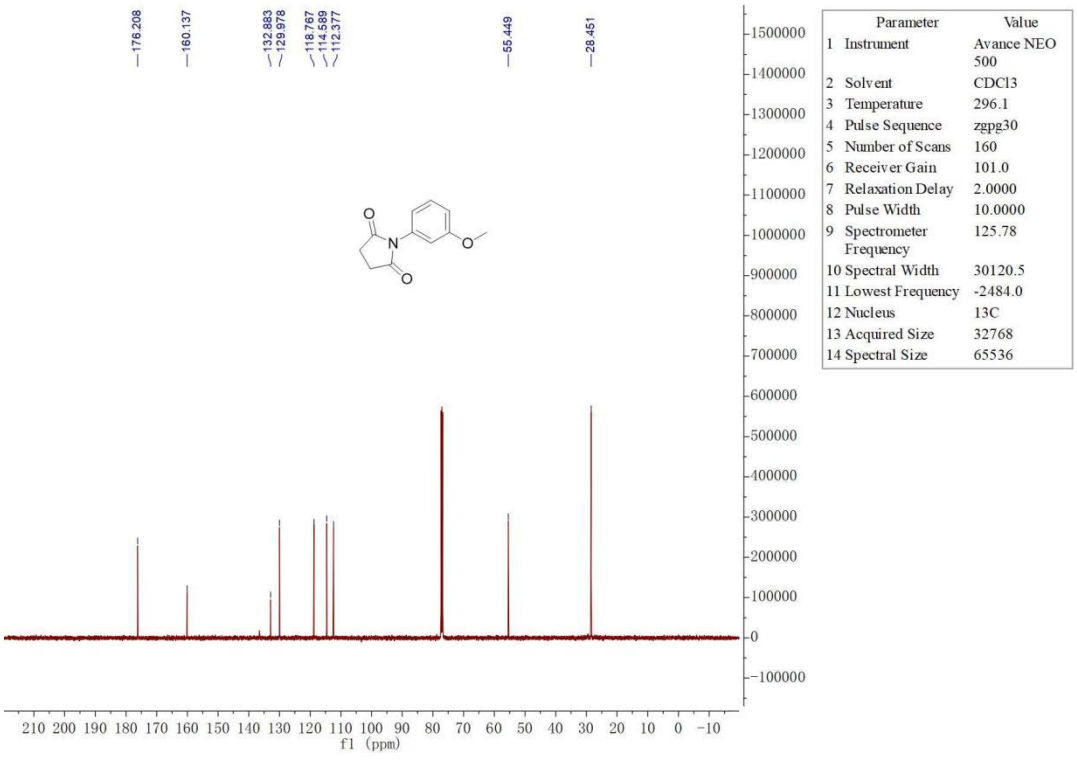
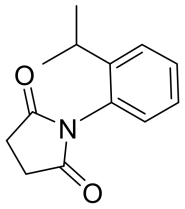
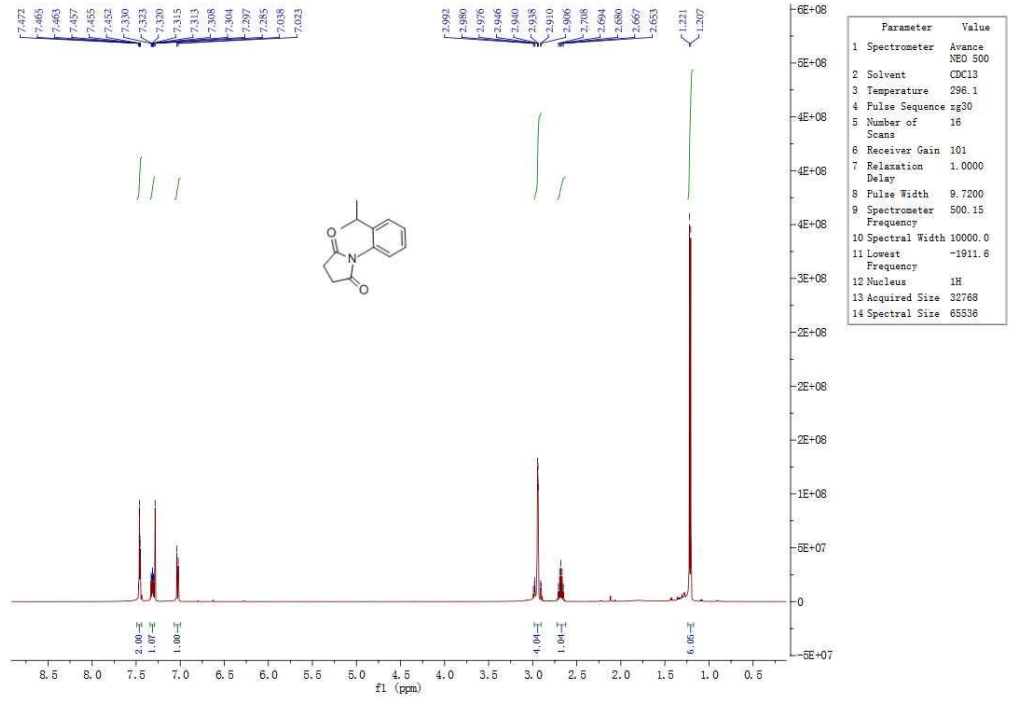


Figure S20. ^{1}H, ^{13}C and ^{19}F spectrum of compound **3k.**

1-(2-isopropylphenyl)pyrrolidine-2,5-dione (3l)





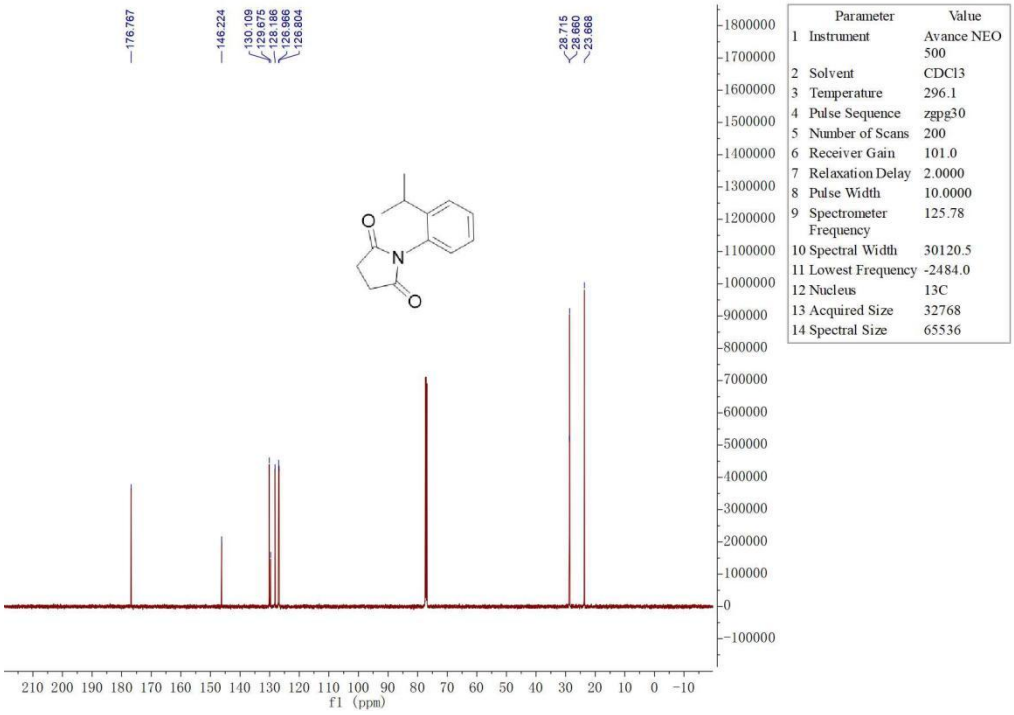
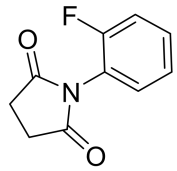
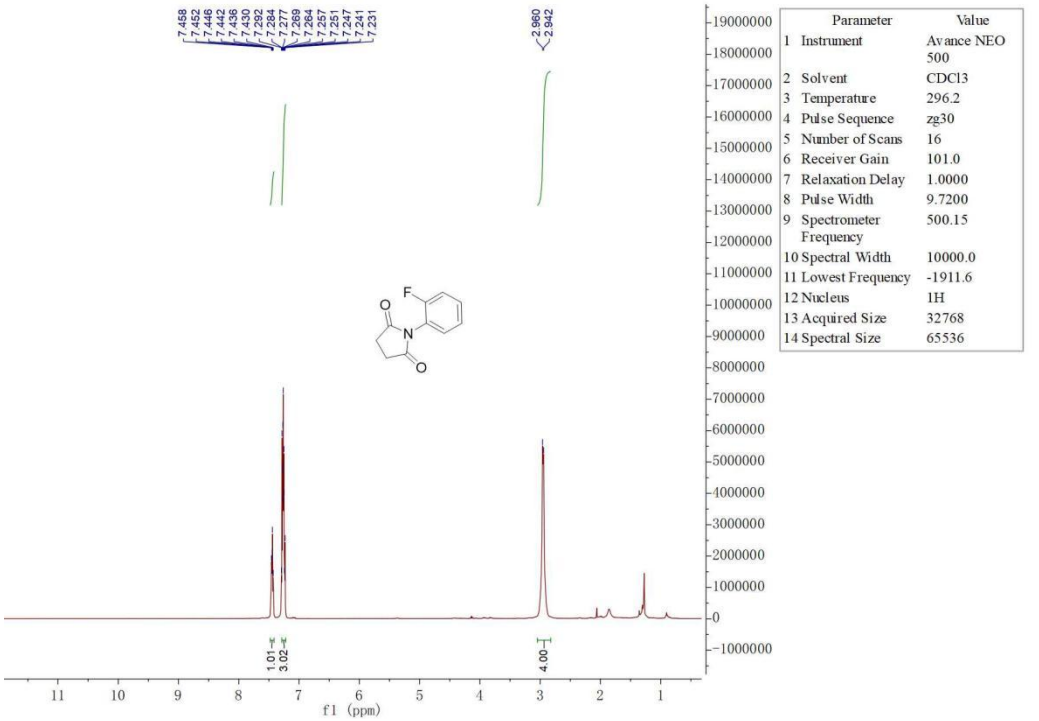


Figure S21. ^{1}H, ^{13}C and ^{19}F spectrum of compound **3l.**

1-(2-fluorophenyl)pyrrolidine-2,5-dione (3m)





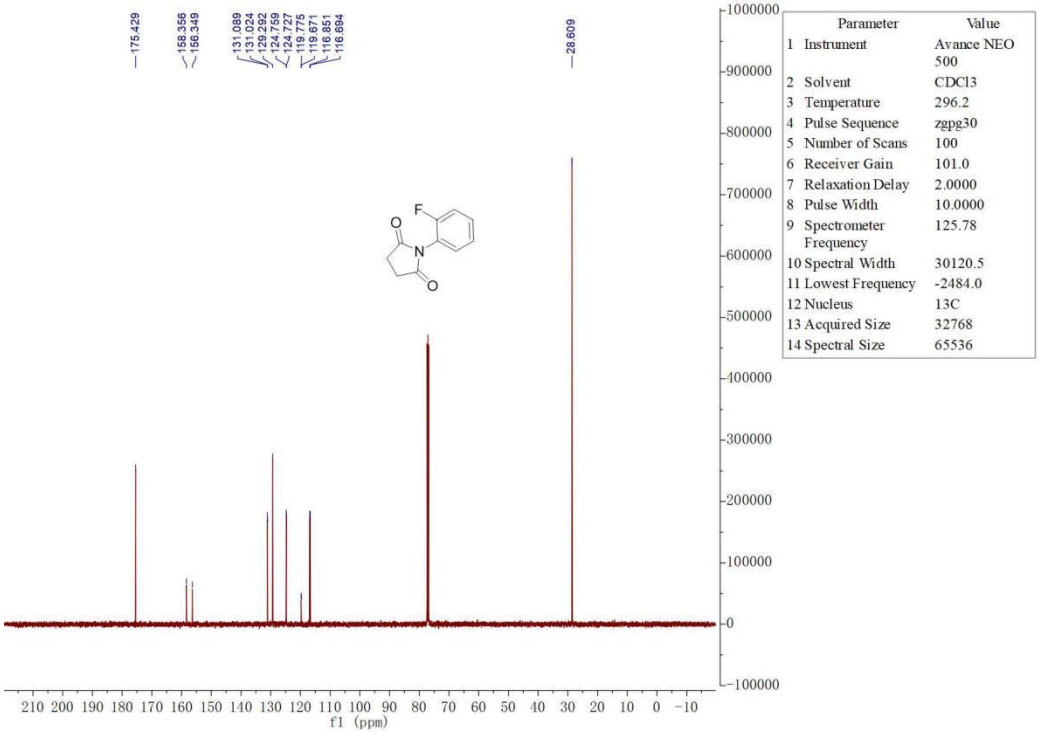


Figure S22. ^{1}H and ^{13}C spectrum of compound **3m.**

1-(2-iodophenyl)pyrrolidine-2,5-dione (3n)

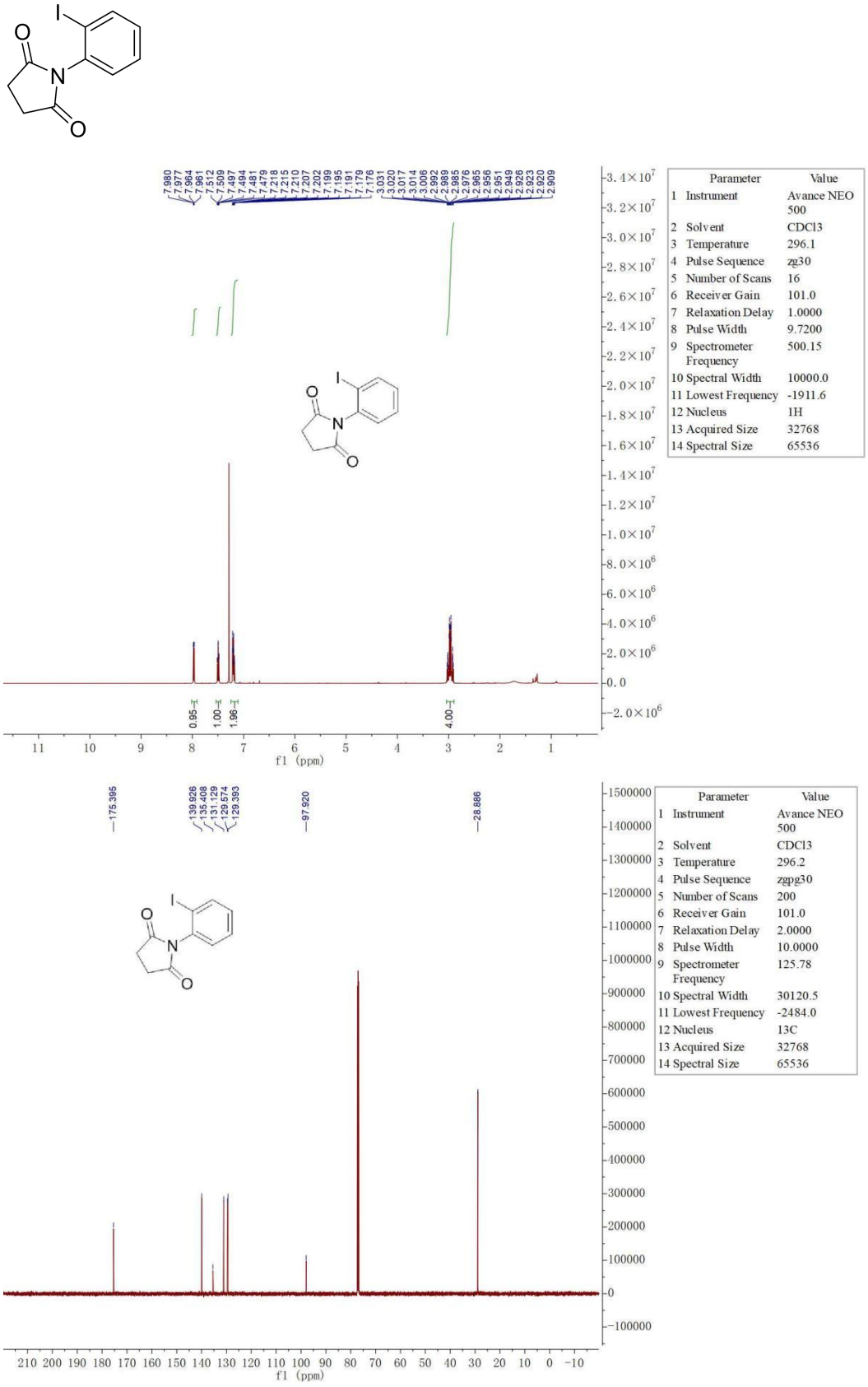
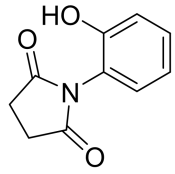
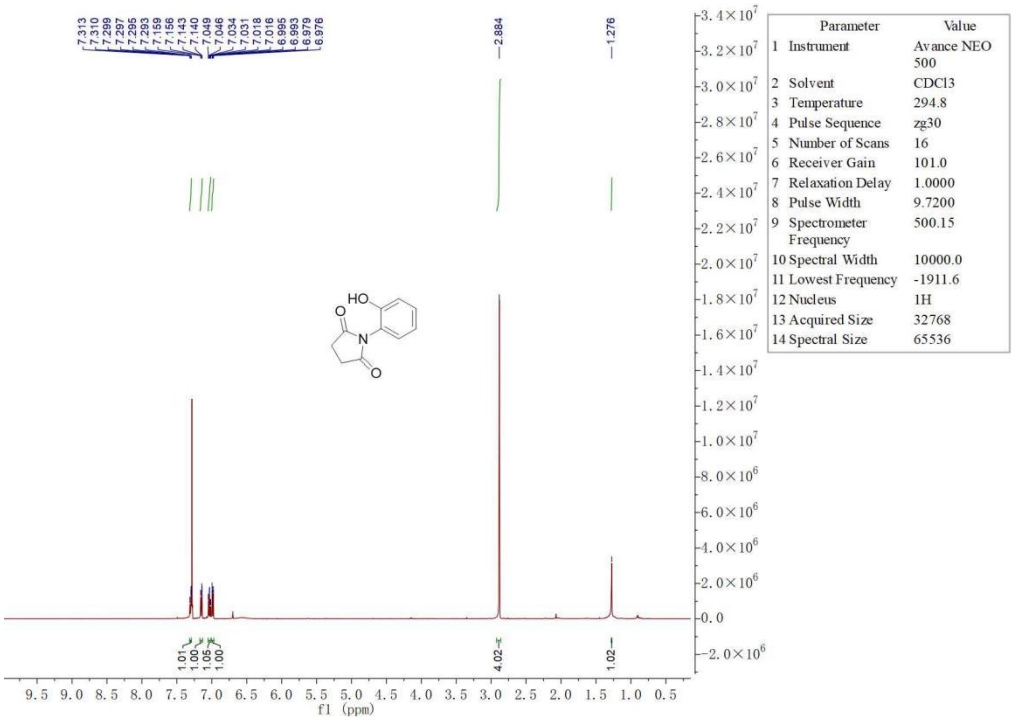


Figure S23. ^{1}H and ^{13}C spectrum of compound **3n.**

1-(2-hydroxyphenyl)pyrrolidine-2,5-dione (3o)





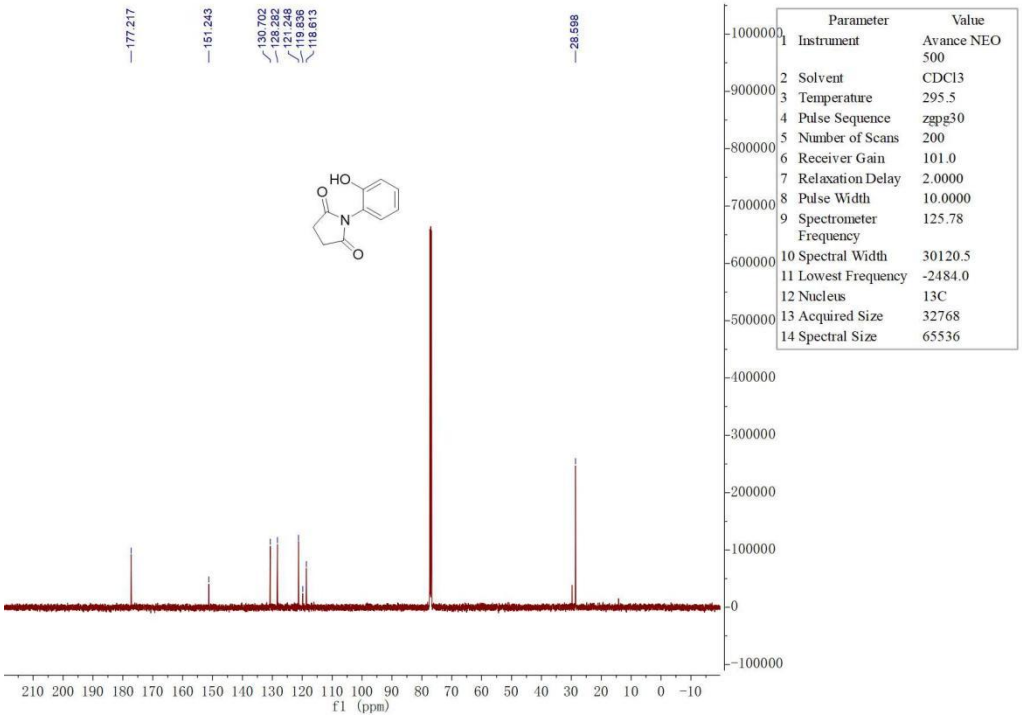
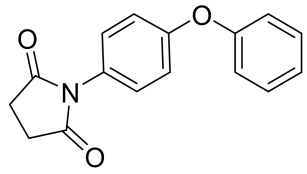


Figure S24. ^{1}H and ^{13}C spectrum of compound **3o.**

1-(4-phenoxyphenyl)pyrrolidine-2,5-dione (3p)



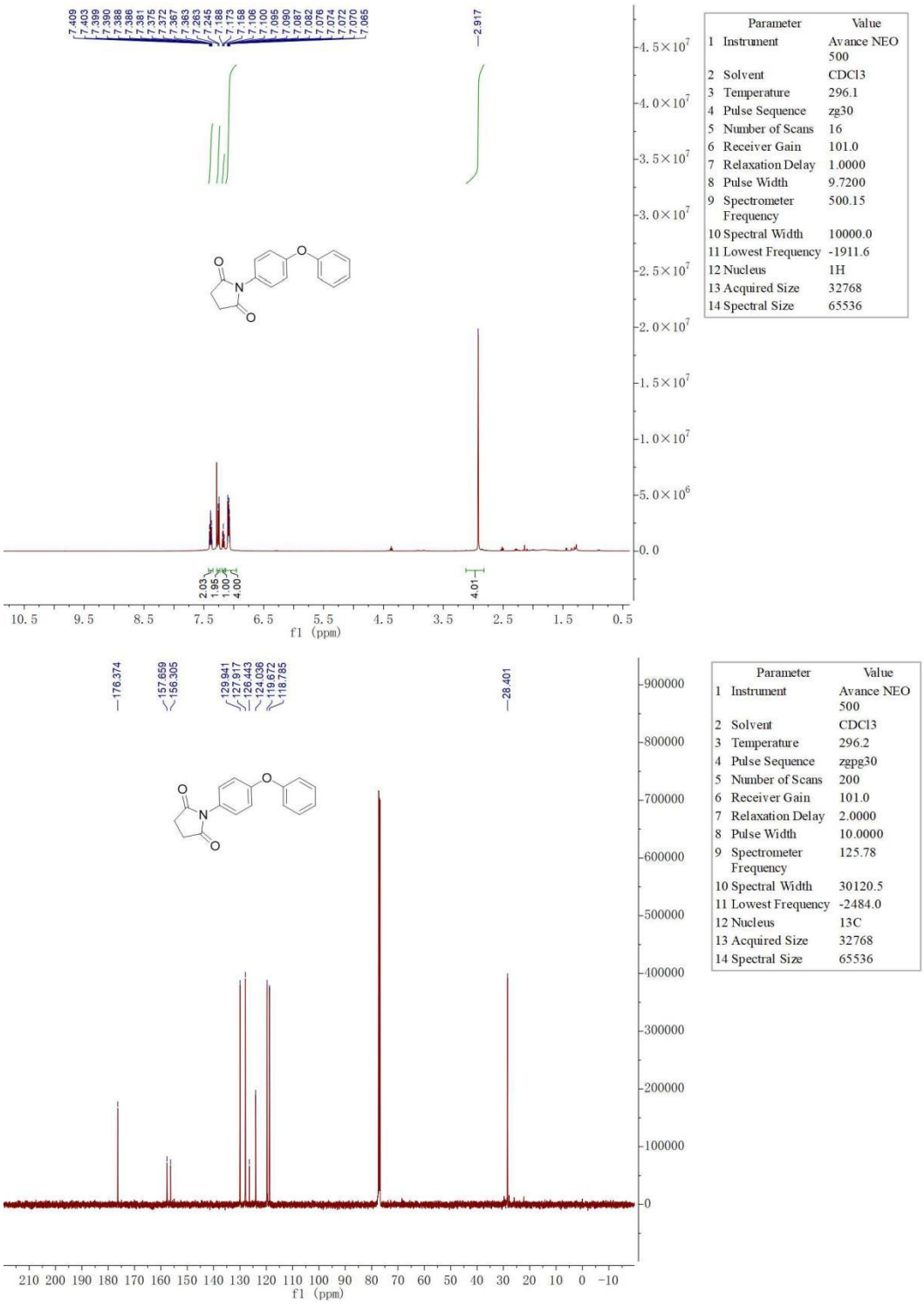


Figure S25. ^{1}H and ^{13}C spectrum of compound **3p.**

1-(3,4-dimethoxyphenyl)pyrrolidine-2,5-dione (3q)

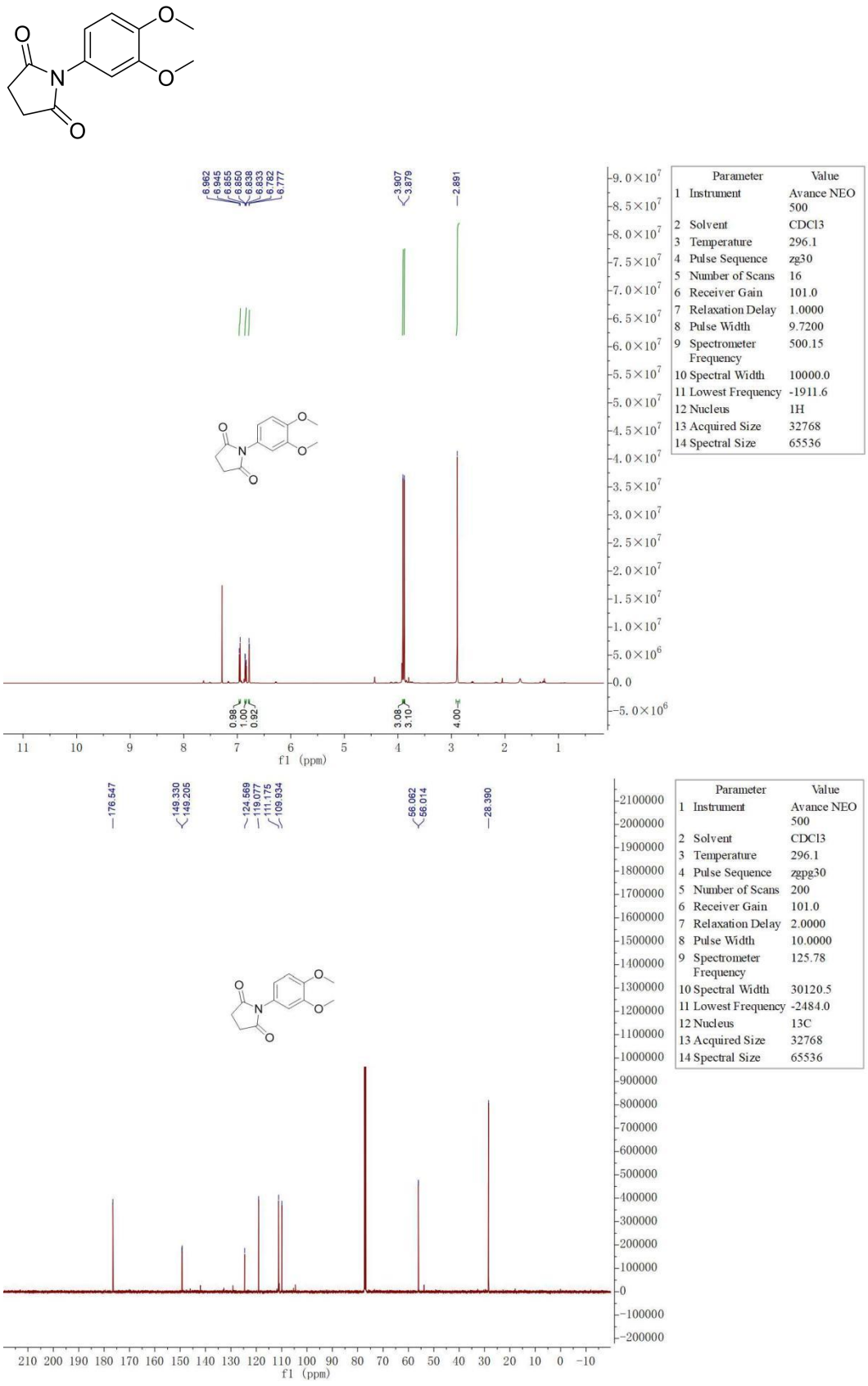
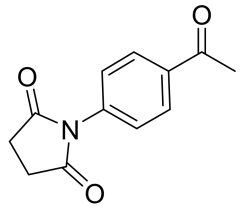
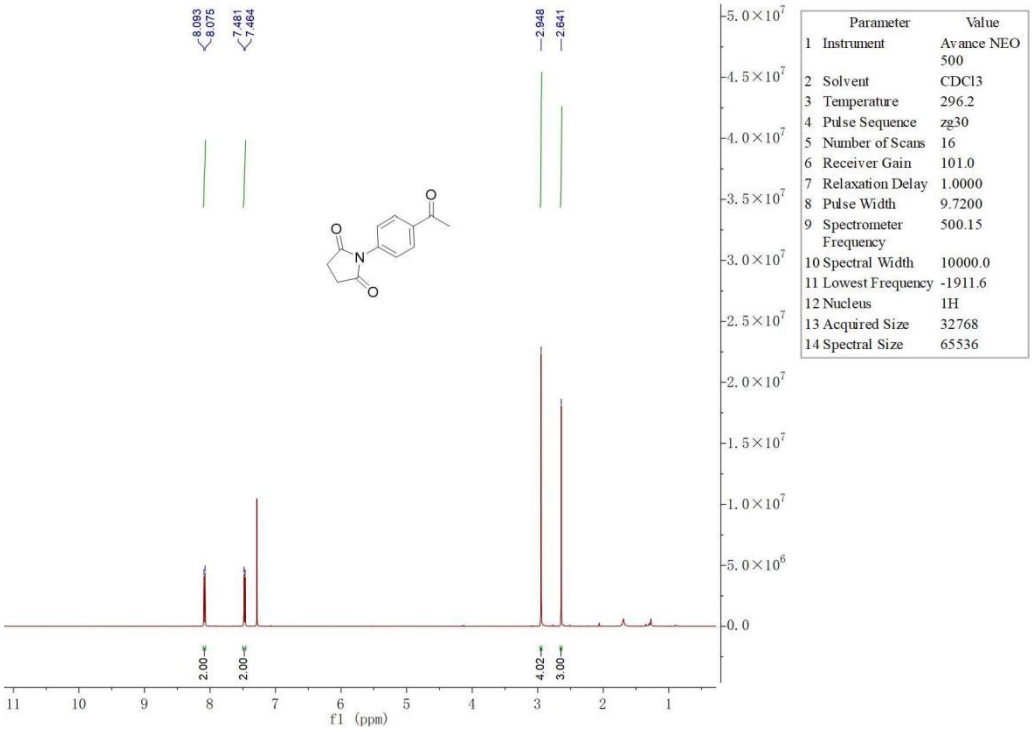


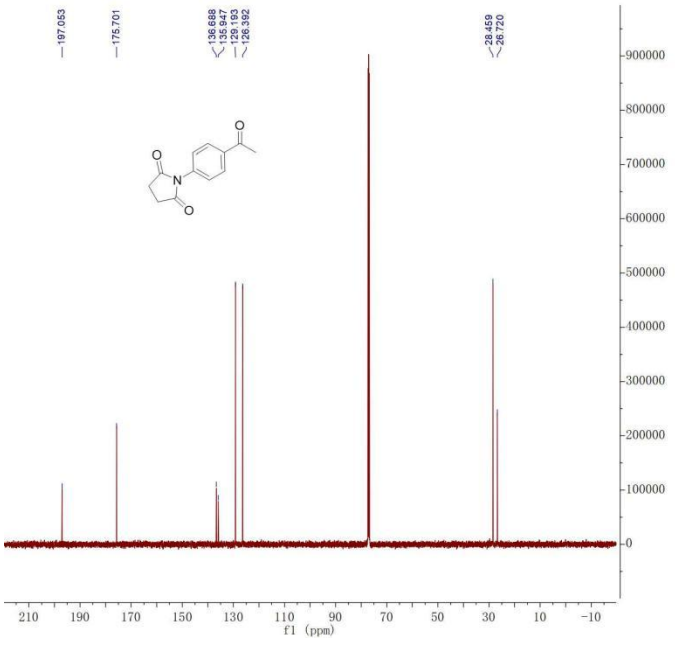
Figure S26. ^{1}H and ^{13}C spectrum of compound **3q.**

1-(4-acetylphenyl)pyrrolidine-2,5-dione (3r)





Parameter	Value
1 Instrument	Avance NEO 500
2 Solvent	CDCl3
3 Temperature	296.2
4 Pulse Sequence	zg30
5 Number of Scans	16
6 Receiver Gain	101.0
7 Relaxation Delay	1.0000
8 Pulse Width	9.7200
9 Spectrometer Frequency	500.15
10 Spectral Width	10000.0
11 Lowest Frequency	-1911.6
12 Nucleus	1H
13 Acquired Size	32768
14 Spectral Size	65536



Parameter	Value
1 Instrument	Avance NEO 500
2 Solvent	CDCl3
3 Temperature	296.2
4 Pulse Sequence	zgpg30
5 Number of Scans	200
6 Receiver Gain	101.0
7 Relaxation Delay	2.0000
8 Pulse Width	10.0000
9 Spectrometer Frequency	125.78
10 Spectral Width	30120.5
11 Lowest Frequency	-2484.0
12 Nucleus	13C
13 Acquired Size	32768
14 Spectral Size	65536

Figure S27. ^{1}H and ^{13}C spectrum of compound **3r.**

1-(3-acetylphenyl)pyrrolidine-2,5-dione (3s)

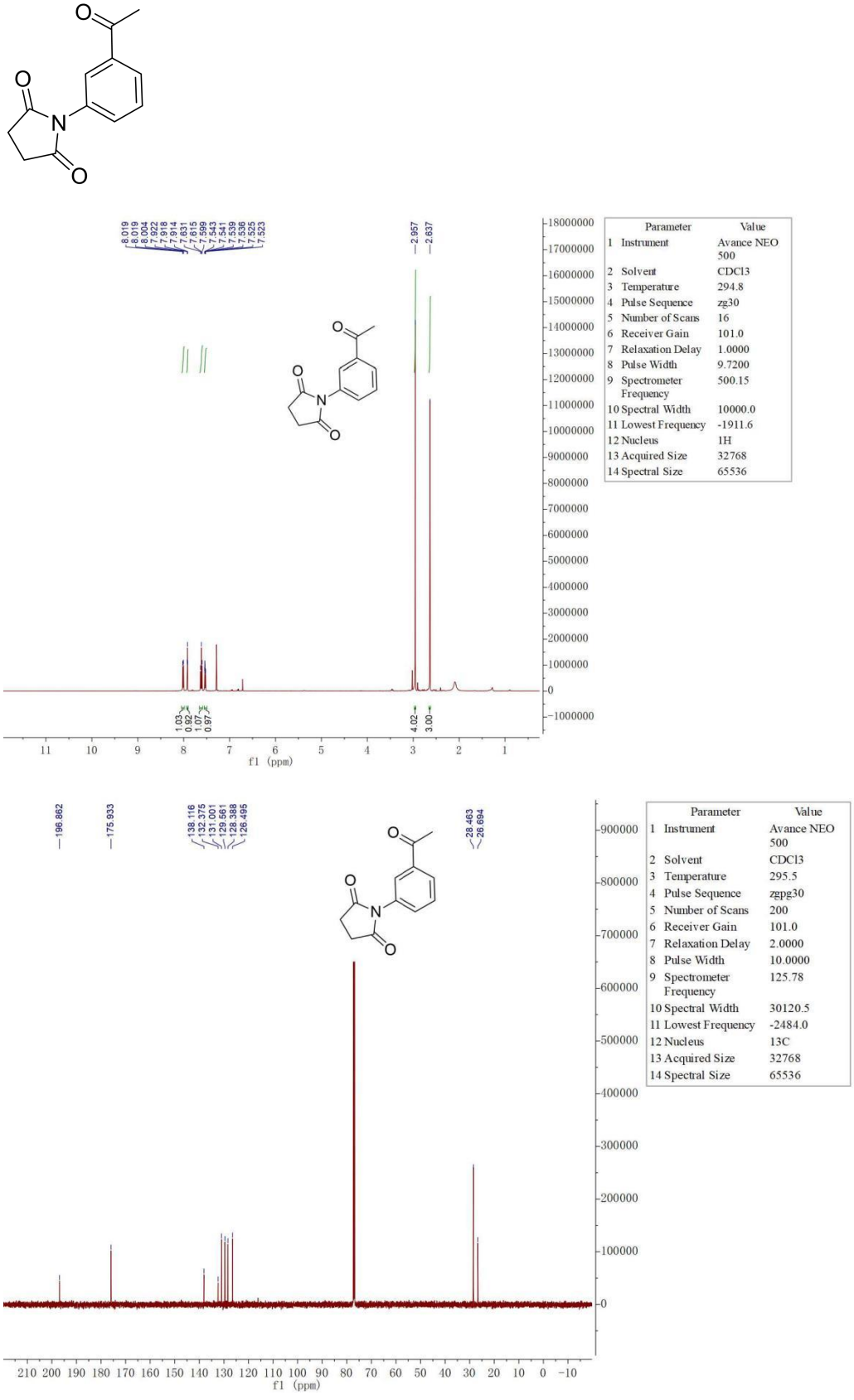
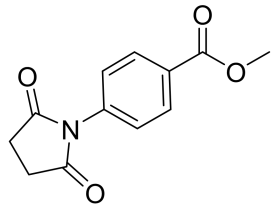
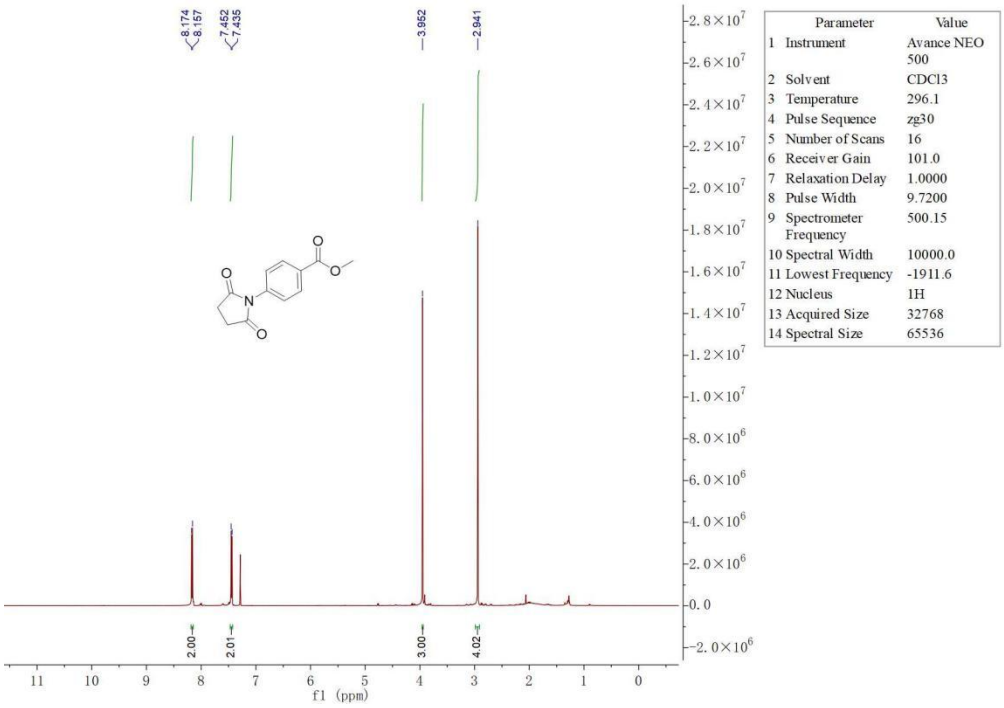


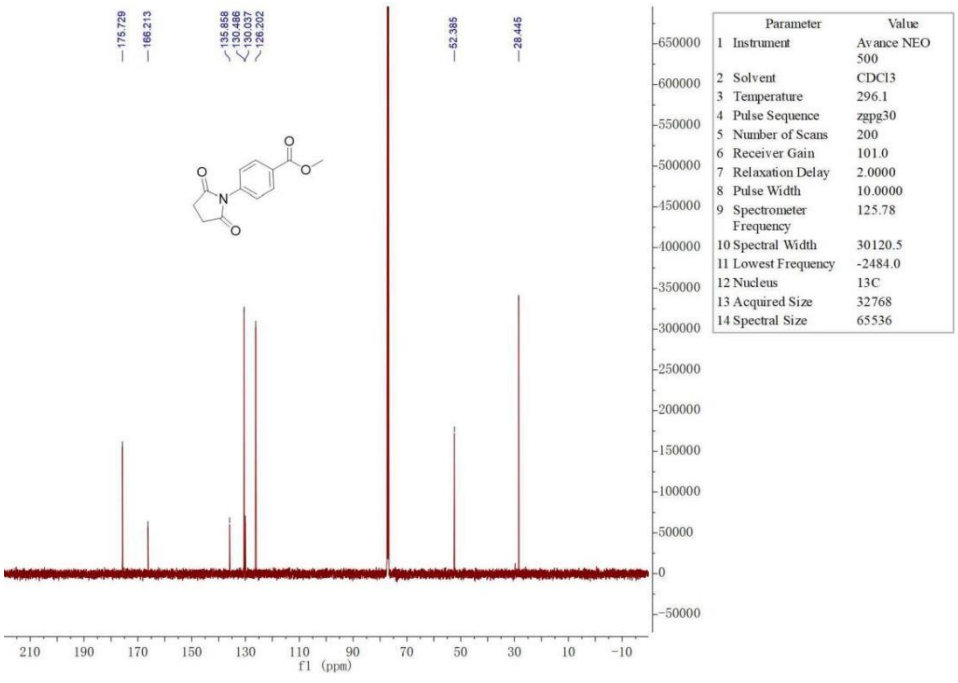
Figure S28. ^{1}H and ^{13}C spectrum of compound **3s.**

methyl 4-(2,5-dioxopyrrolidin-1-yl)benzoate (3t)





Parameter	Value
1 Instrument	Avance NEO 500
2 Solvent	CDCl3
3 Temperature	296.1
4 Pulse Sequence	zg30
5 Number of Scans	16
6 Receiver Gain	101.0
7 Relaxation Delay	1.0000
8 Pulse Width	9.7200
9 Spectrometer Frequency	500.15
10 Spectral Width	10000.0
11 Lowest Frequency	-1911.6
12 Nucleus	1H
13 Acquired Size	32768
14 Spectral Size	65536



Parameter	Value
1 Instrument	Avance NEO 500
2 Solvent	CDCl3
3 Temperature	296.1
4 Pulse Sequence	zgpg30
5 Number of Scans	200
6 Receiver Gain	101.0
7 Relaxation Delay	2.0000
8 Pulse Width	10.0000
9 Spectrometer Frequency	125.78
10 Spectral Width	30120.5
11 Lowest Frequency	-2484.0
12 Nucleus	13C
13 Acquired Size	32768
14 Spectral Size	65536

Figure S29. ^{1}H and ^{13}C spectrum of compound **3t.**

1-benzylpyrrolidine-2,5-dione (3u)

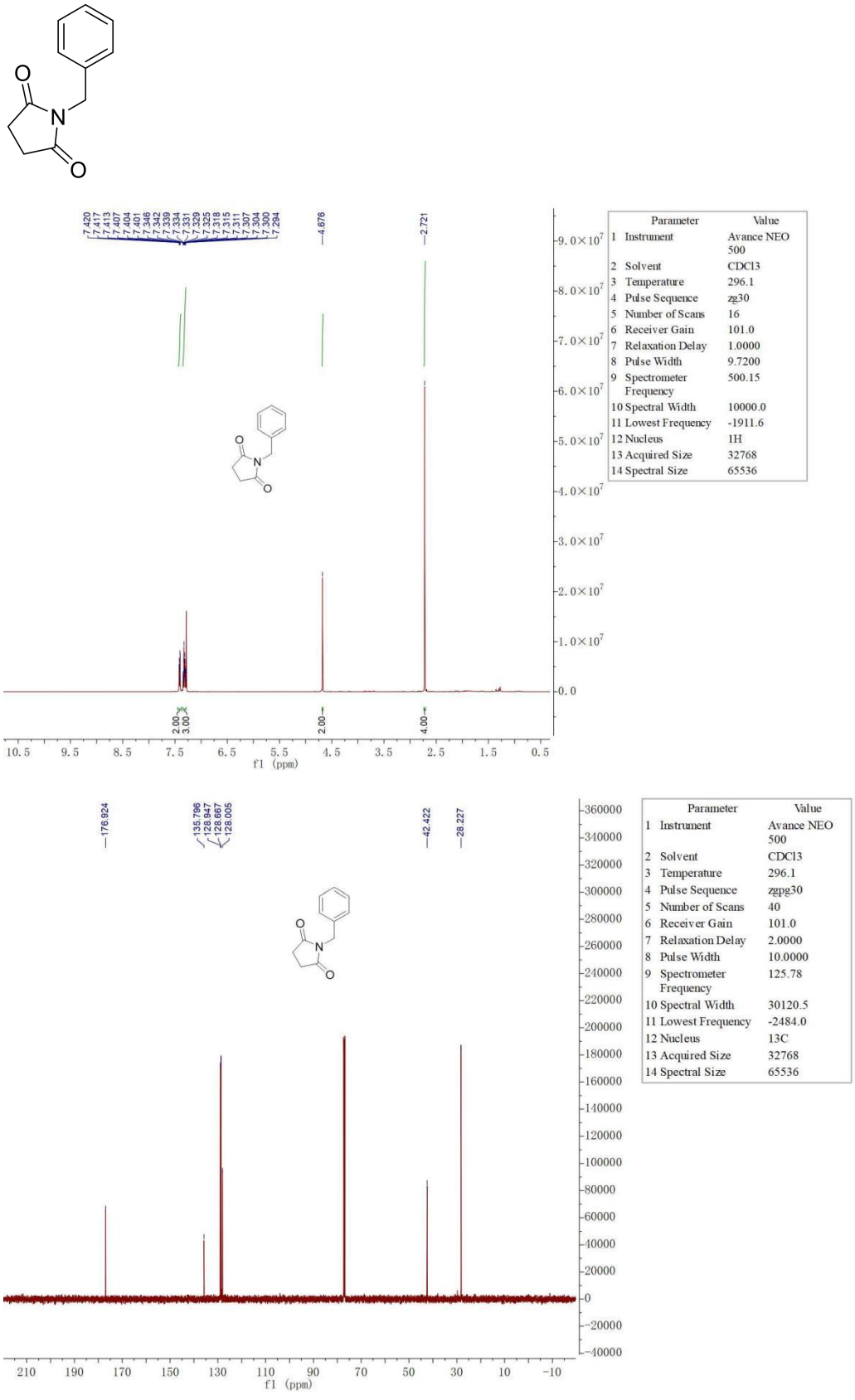


Figure S30. ^{1}H and ^{13}C spectrum of compound **3u.**

1-(3-bromobenzyl)pyrrolidine-2,5-dione (3v)

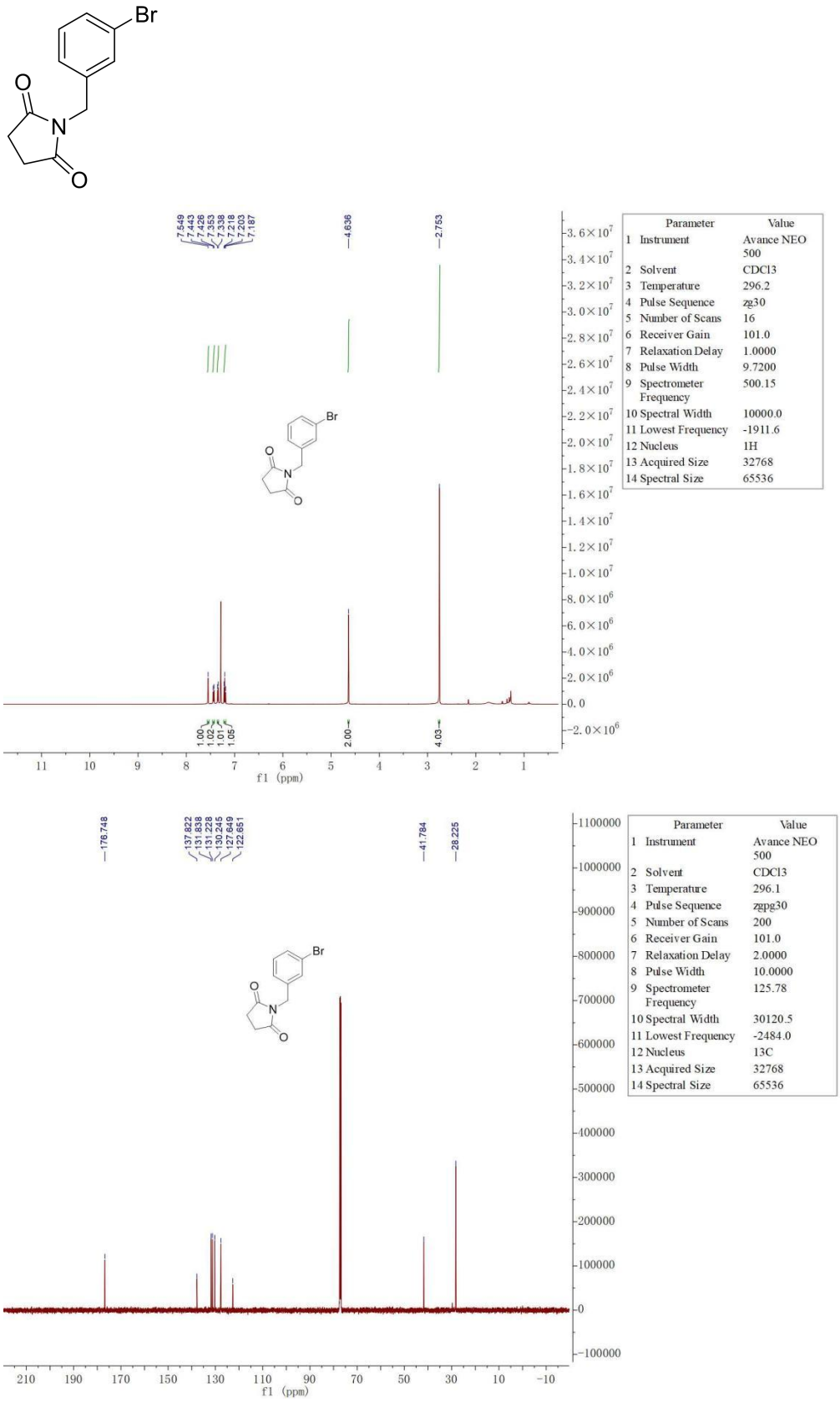


Figure S31. ^{1}H and ^{13}C spectrum of compound **3v.**

1-(naphthalen-1-yl)pyrrolidine-2,5-dione (3w)

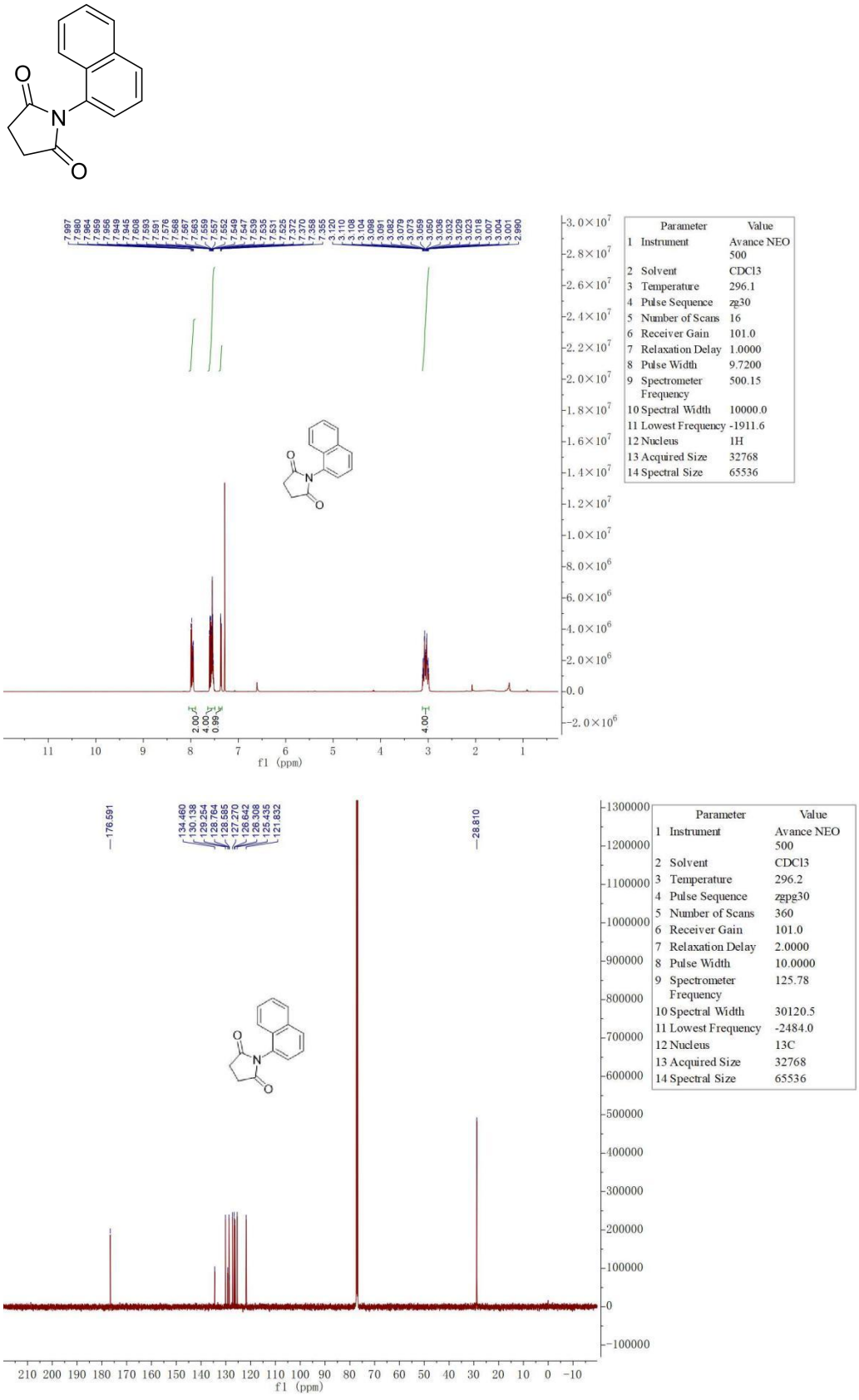
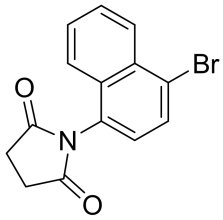
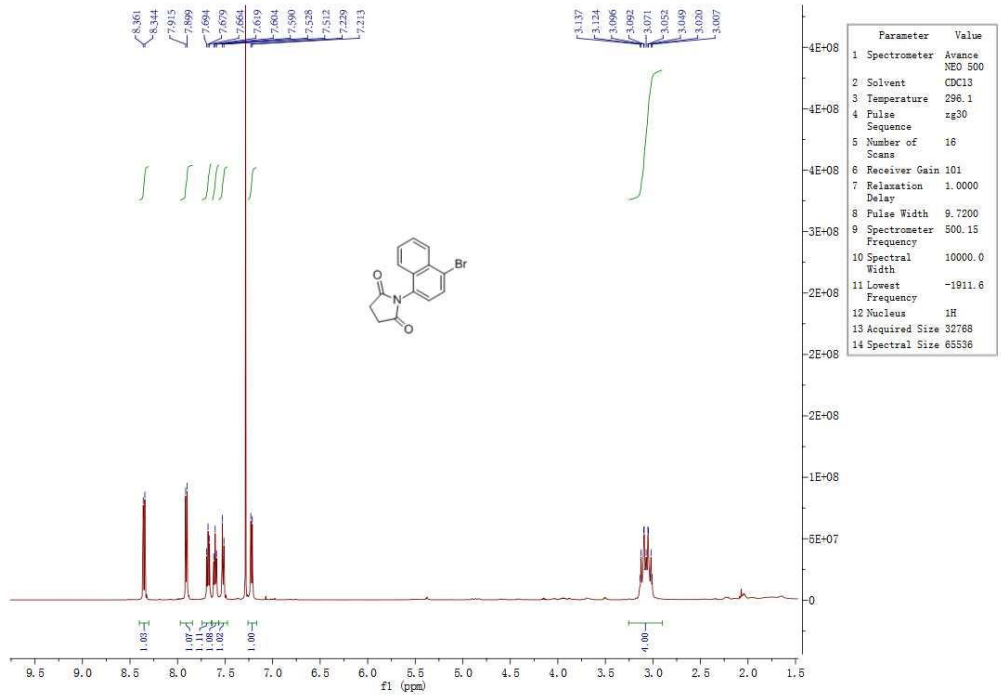


Figure S32. ^{1}H and ^{13}C spectrum of compound **3w.**

1-(4-bromonaphthalen-1-yl)pyrrolidine-2,5-dione (3x)





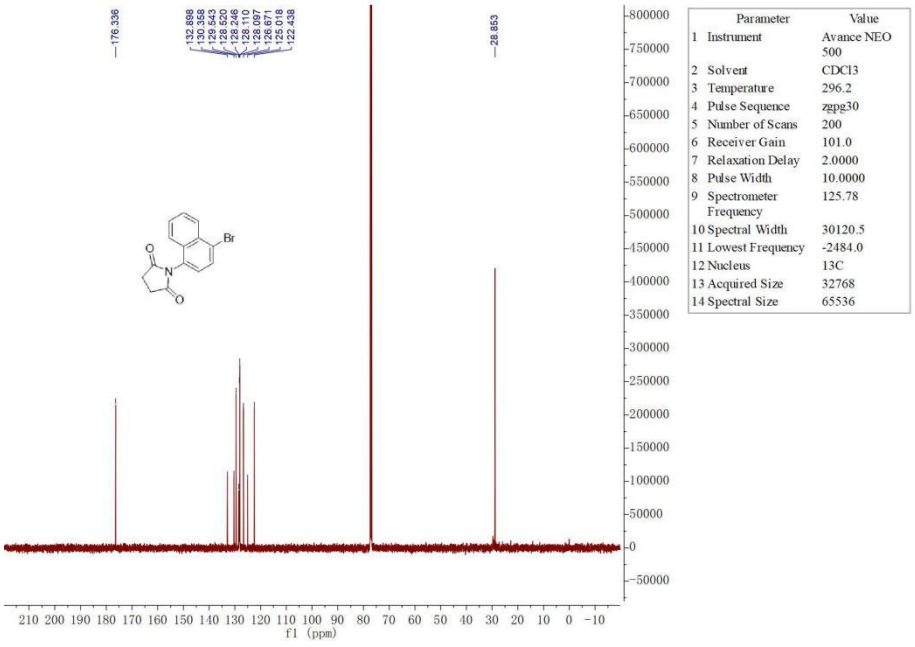
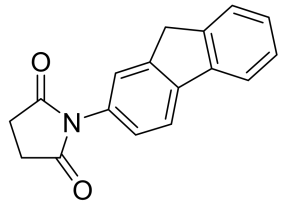


Figure S33. ^{1}H and ^{13}C spectrum of compound **3x.**

1-(9H-fluoren-2-yl)pyrrolidine-2,5-dione (3y)



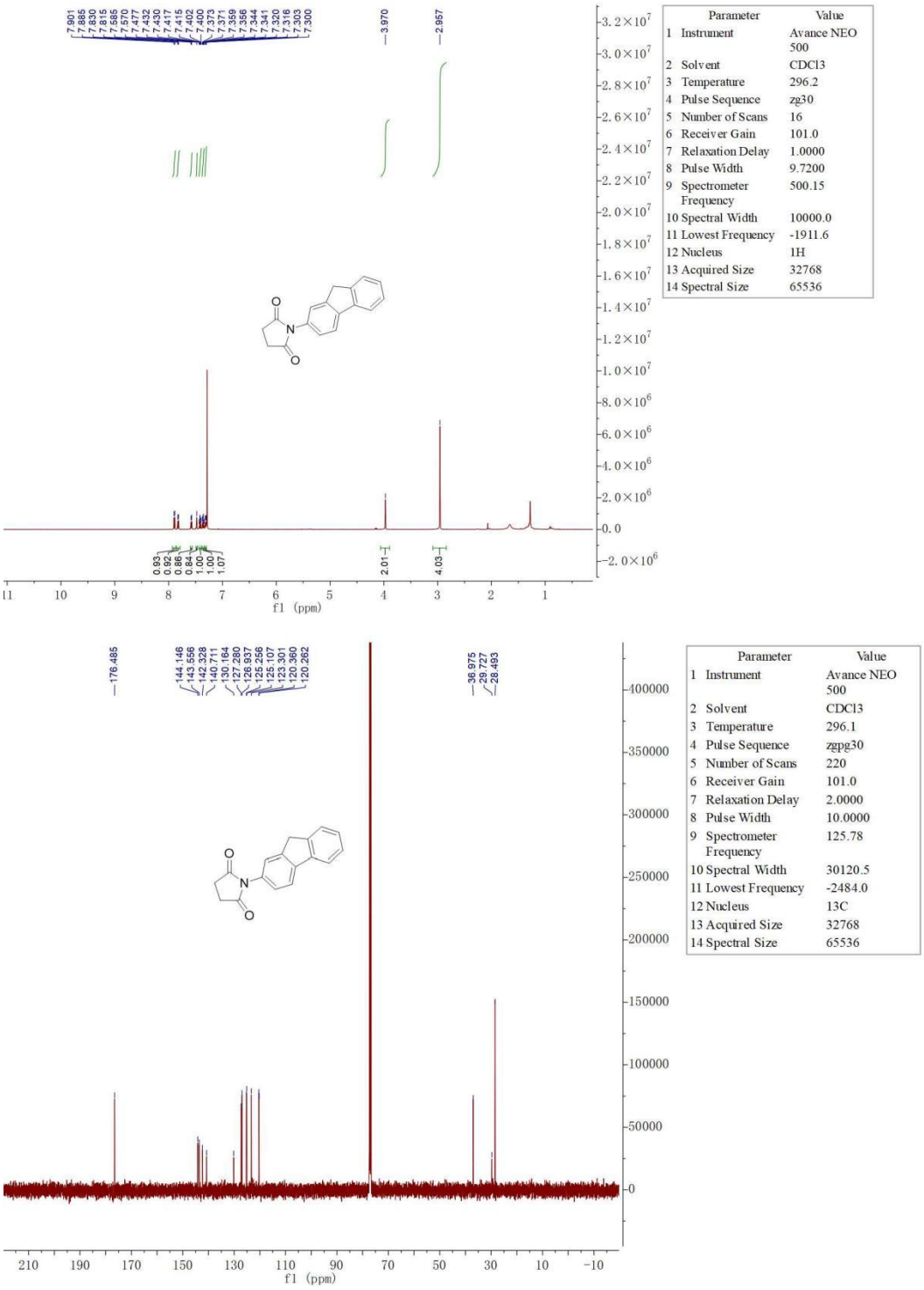


Figure S34. ^{1}H and ^{13}C spectrum of compound **3y.**

1-**(pyren-1-yl)pyrrolidine-2,5-dione (3z)**

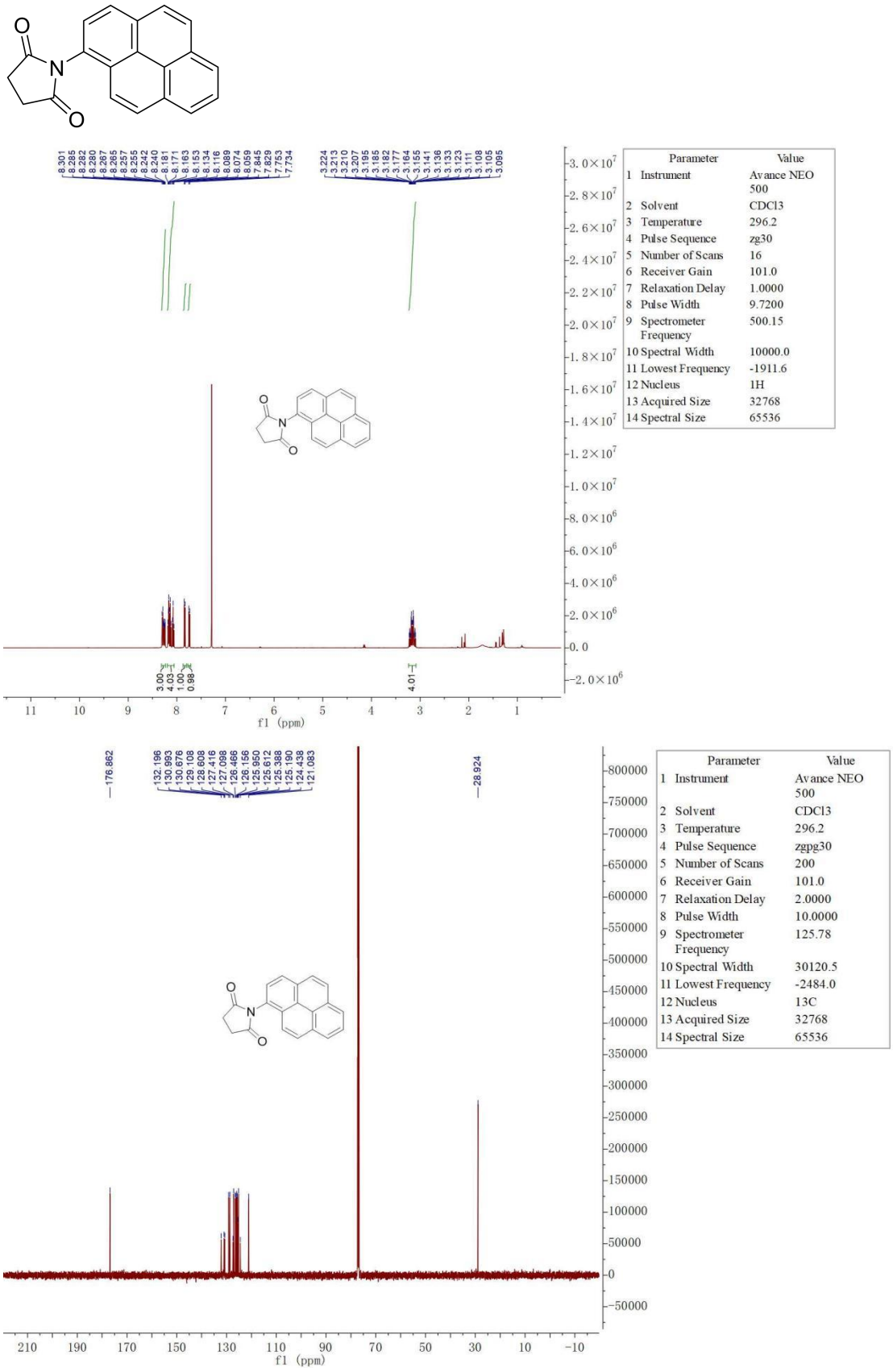
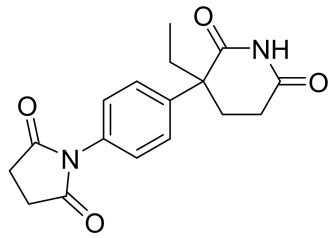


Figure S35. ^{1}H and ^{13}C spectrum of compound **3z.**

3-(4-(2,5-dioxopyrrolidin-1-yl)phenyl)-3-ethylpiperidine-2,6-dione (3aa)



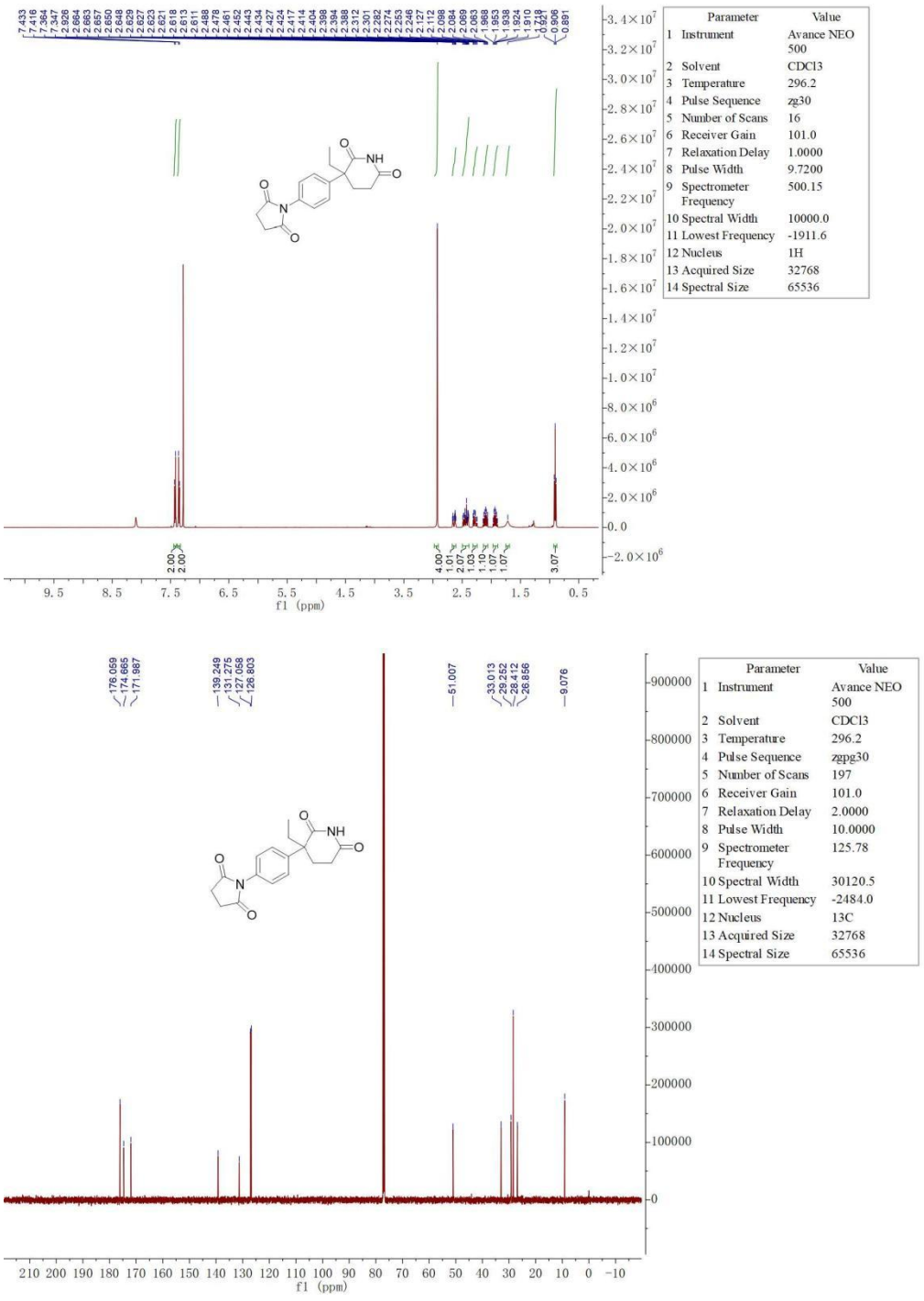


Figure S36. ^{1}H and ^{13}C spectrum of compound **3aa.**

1,3-diphenylpyrrolidine-2,5-dione (3ab)

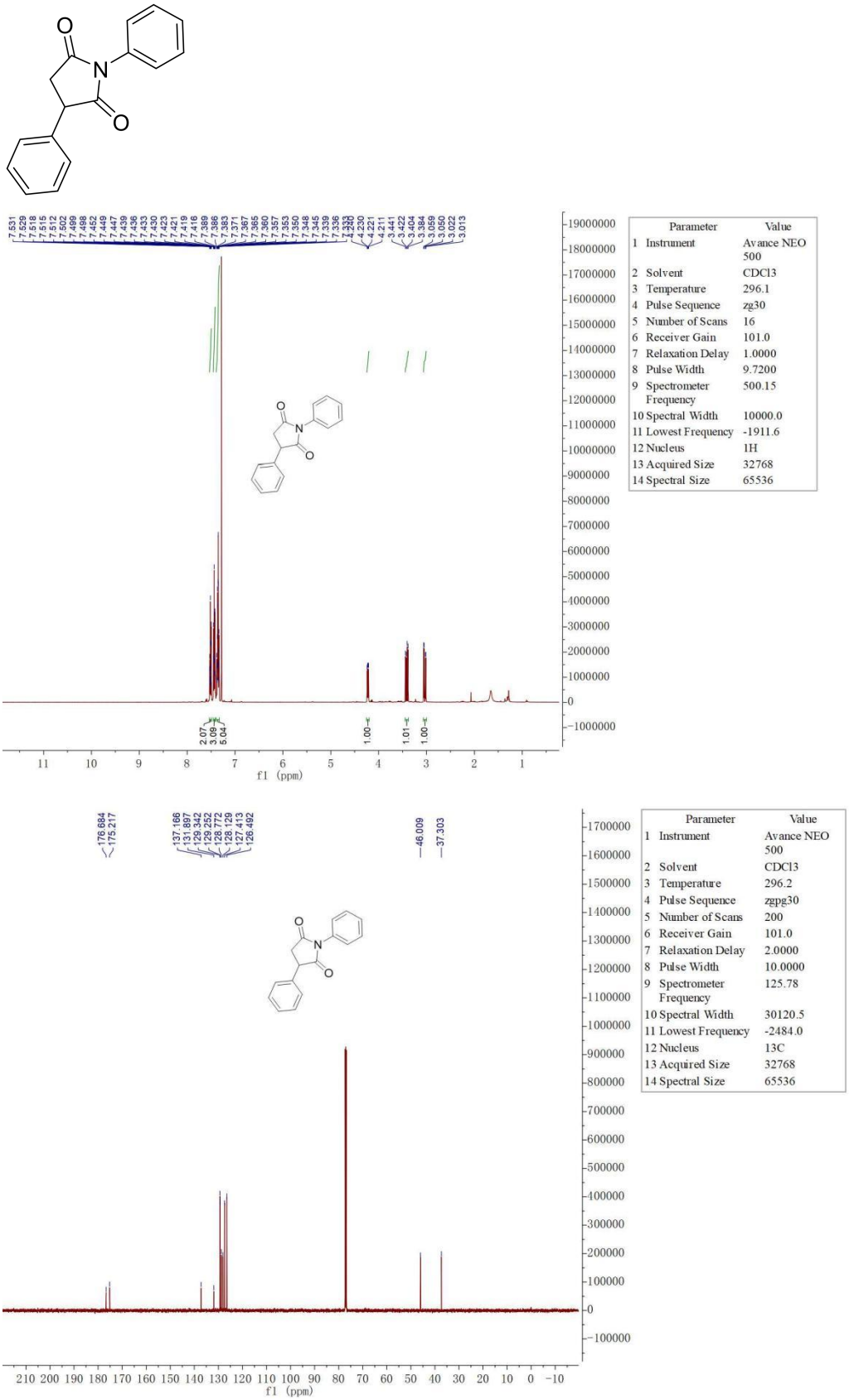
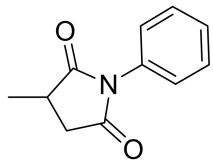


Figure S37. ^{1}H and ^{13}C spectrum of compound **3ab.**

3-methyl-1-phenylpyrrolidine-2,5-dione (3ac)



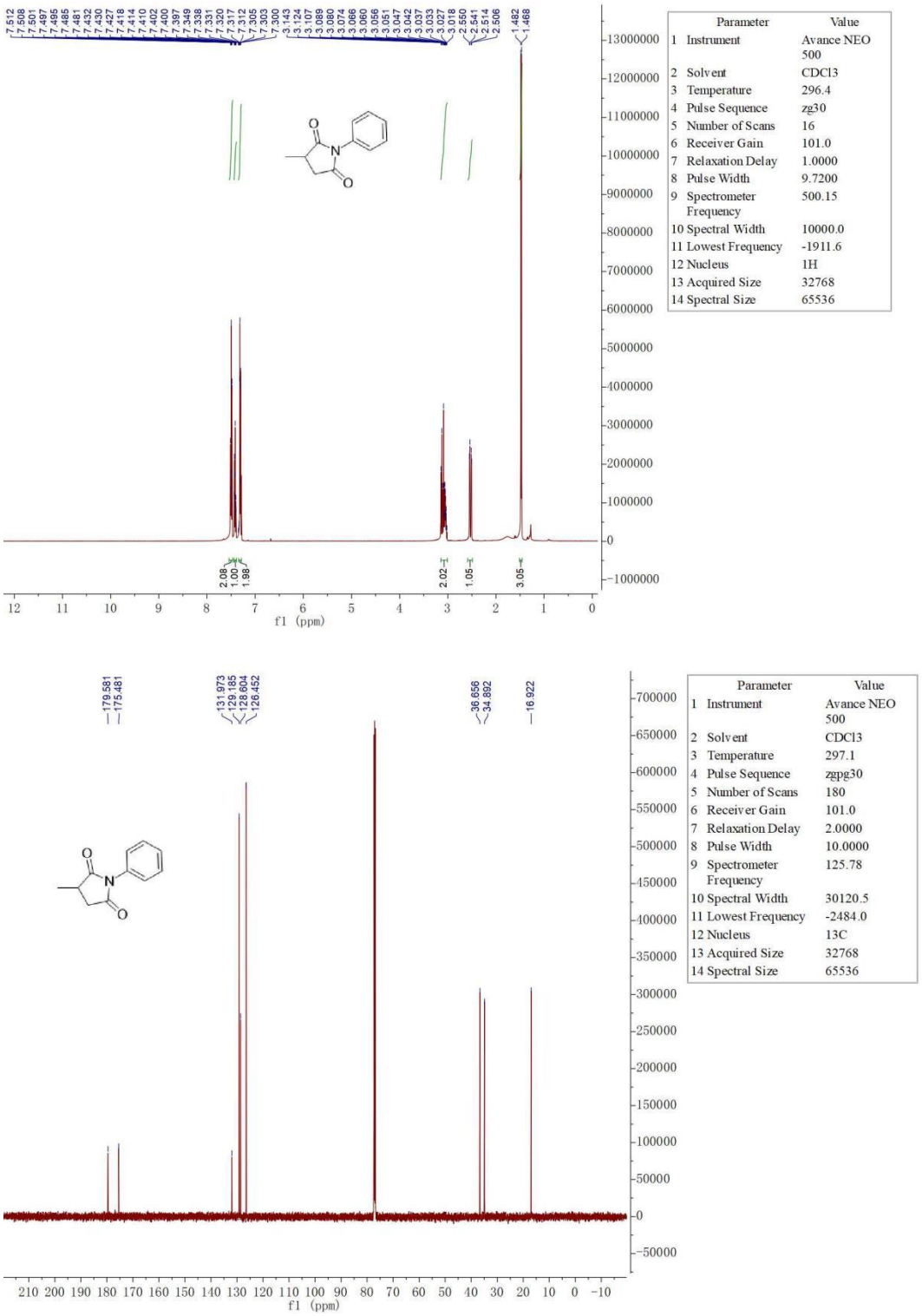


Figure S38. ^{1}H and ^{13}C spectrum of compound **3ac.**