Supplementary Information for:

Remote Activating Strategy Enabled (RASE) π-Bond Migratory Dealkylative C-N coupling Utilising *N*-Fluorobenzenesulfonimide (NFSI) as A Bifunctional Domino Reagent

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Table of Contents

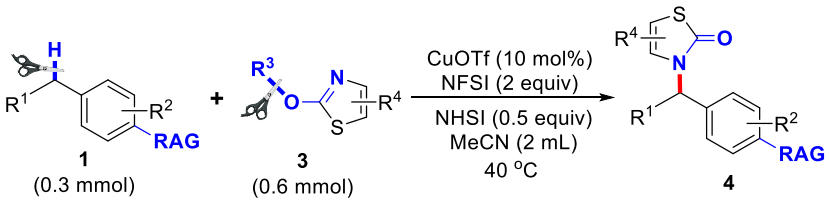
1. General Remarks

All commercially available compounds were purchased from Sigma-Aldrich, Alfa-Aesar, Acros, J&K Chemicals, Adamas-beta, Accela ChemBio and Aladdin Chemicals. Unless otherwise noted, materials obtained from commercial suppliers were used without further purification. Alkyl benzenes **1** and anilines **2** with remote activating groups were prepared according to the methods reported in our recently published remote activating strategy enabled (RASE) reactions.[1] Products were purified by flash chromatography on silica gel using petroleum ether, ethyl acetate and dichloromethane as the eluents. ^{1}H-NMR spectra were recorded on JNM-ECZ400S/L1 spectrometers. Chemical shifts (in ppm) were referenced with TMS in $CDCl_3$ or DMSO-d_6 (0 ppm). ^{13}C-NMR spectra were obtained by using the same NMR spectrometers and were calibrated with $CDCl_3$ (δ = 77.00 ppm) or DMSO-d_6 (δ = 39.50 ppm). High resolution mass spectra were obtained from an Agilent 6520B Q-TOF mass spectrometer with electron spray ionization (ESI) as the ion source.

2. General Procedure and Characterization Data

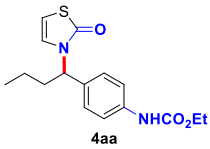
2.1 General Procedure A

RASE *π*-bond migratory dealkylative C(sp^3)-N coupling with 2-alkoxylthiazoles offering *N*-benzyl thiazol-2(3*H*)-ones 4 (results shown in Table 3)



To a reaction tube charged with CuOTf (6.4 mg, 0.03 mmol, 10 mol%), NFSI (189.2 mg, 0.6 mmol, 2 equiv) and NHSI (44.6 mg, 0.15 mmol, 0.5 equiv) was added a solution of alkyl benzene (**1**, 0.3 mmol, 1 equiv) and 2-alkoxythiazole (**3**, 0.6 mmol, 2 equiv) in anhydrous MeCN (2 mL, 0.15 M of **1**) via a syringe under argon (1 atm) at 25 °C, and the reaction mixture was stirred for 8 hours at 40 °C (oil bath temperature). After quenched with Na_2CO_3 (aq.), the mixture was extracted with ethyl acetate. The combined organic phase was concentrated *in vacuo* to give dark residue, which was then purified by flash chromatography using petroleum ether and ethyl acetate as the eluent on silica gel to afford corresponding *N*-benzyl thiazol-2(3*H*)-ones **4**.

Ethyl (4-(1-(2-oxothiazol-3(2*H*)-yl)butyl)phenyl)carbamate (4aa) [1a]:



The RASE π-bond migratory dealkylative C(sp^3)-N coupling between 0.3 mmol of ethyl (4-butylphenyl)carbamate (**1a**, 66.4 mg) and 0.6 mmol of 2-methoxythiazole (**3a**, 69.0 mg) afforded 74.5 mg of **4aa** (78%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (4:1 to 3:1, *v*/*v*) as the eluent.

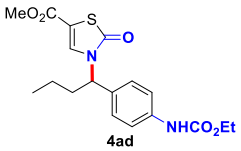
The RASE π-bond migratory dealkylative C(sp^3)-N coupling between 0.3 mmol of ethyl (4-butylphenyl)carbamate (**1a**, 66.4 mg) and 0.6 mmol of 2-ethoxythiazole (**3b**, 77.4 mg) afforded 63.6 mg of **4aa** (66%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 3:1, *v*/*v*) as the eluent.

The RASE π-bond migratory dealkylative C(sp^3)-N coupling between 0.3 mmol of ethyl (4-butylphenyl)carbamate (**1a**, 66.4 mg) and 0.6 mmol of 2-butoxythiazole (**3c**, 94.2 mg) afforded 59.8 mg of **4aa** (62%) after flash chromatography on silica gel using petroleum ether and ethyl

acetate (4:1 to 3:1, *v*/*v*) as the eluent.

White solid, m.p. 78-80 °C. **^{1}H NMR ($CDCl_3$, 400 MHz):** δ = 7.38 (d, *J* = 8.4 Hz, 2H), 7.24 (d, *J* = 8.4 Hz, 2H), 7.01 (s, 1H), 6.53 (d, *J* = 5.4 Hz, 1H), 6.07 (d, *J* = 5.4 Hz, 1H), 5.43 (dd, *J* = 6.7 Hz, 2.5 Hz, 1H), 4.21 (q, *J* = 7.1 Hz, 2H), 2.11-1.95 (m, 2H), 1.37-1.34 (m, 2H), 1.29 (t, *J* = 7.1 Hz, 3H), 0.97 (t, *J* = 7.4 Hz, 3H). **^{13}C NMR ($CDCl_3$, 100 MHz):** δ = 172.1, 153.6, 137.9, 134.1, 127.8, 121.7, 118.8, 101.4, 61.2, 56.6, 35.4, 19.4, 14.5, 13.6 ppm.

Methyl 3-(1-(4-((ethoxycarbonyl)amino)phenyl)butyl)-2-oxo-2,3-dihydrothiazole-5-carboxylate (4ad) [1a]:

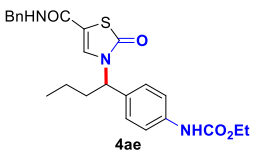


4ad

The RASE π-bond migratory dealkylative C(sp^3)-N coupling between 0.3 mmol of ethyl (4-butylphenyl)carbamate (**1a**, 66.4 mg) and 0.6 mmol of methyl 2-methoxythiazole-5-carboxylate (**3d**, 103.8 mg) afforded 91.0 mg of **4ad** (80%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 3:1, *v*/*v*) as the eluent.

Colorless oil. **^{1}H NMR ($CDCl_3$, 400 MHz):** δ = 7.44 (s, 1H), 7.41 (d, *J* = 8.3 Hz, 2H), 7.25 (d, *J* = 8.6 Hz, 2H), 7.09 (s, 1H), 5.44 (dd, *J* = 7.0 Hz, 1.7 Hz, 1H), 4.21 (q, *J* = 7.1 Hz, 2H), 3.80 (s, 3H), 2.14-1.99 (m, 2H), 1.38-1.3326 (m, 5H), 0.97 (t, *J* = 7.3 Hz, 3H) ppm. **^{13}C NMR ($CDCl_3$, 100 MHz):** δ = 171.3, 161.2, 153.5, 138.3, 133.0, 130.3, 127.9, 118.9, 108.4, 61.2, 57.3, 52.2, 35.4, 19.4, 14.4, 13.5 ppm.

Ethyl (4-(1-(5-(benzylcarbamoyl)-2-oxothiazol-3(2*H*)-yl)butyl)phenyl)carbamate (4ae) [1a]:



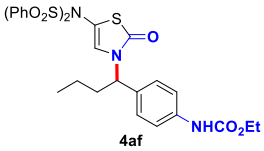
4ae

The RASE π-bond migratory dealkylative C(sp^3)-N coupling between 0.3 mmol of ethyl (4-butylphenyl)carbamate (**1a**, 66.4 mg) and 0.6 mmol of *N*-benzyl-2-methoxythiazole-5-carboxamide (**3e**, 148.8 mg) afforded 111.2 mg of **4ae** (82%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 4:1, *v*/*v*) as the eluent.

Colorless oil. **^{1}H NMR ($CDCl_3$, 400 MHz):** δ = 7.44 (s, 1H), 7.39 (s, 1H), 7.30 (d, *J* = 8.3 Hz, 2H), 7.25-7.18 (m, 5H), 7.12 (d, *J* = 8.4 Hz, 2H), 7.06 (t, *J* = 5.7 Hz, 1H), 5.36 (dd, *J* = 7.0 Hz, 1.8 Hz, 1H), 4.42-4.32 (m, 2H), 4.11 (q, *J* = 7.1 Hz, 2H), 1.99-1.88 (m, 2H), 1.29-1.21 (m, 5H), 0.91

(t, *J* = 7.3 Hz, 3H) ppm. **^{13}C NMR ($CDCl_3$, 100 MHz):** δ = 170.9, 160.3, 153.8, 138.2, 137.5, 133.1, 128.4, 127.7, 127.4, 126.1, 119.1, 112.8, 61.1, 57.2, 43.7, 35.3, 19.3, 14.3, 13.4 ppm.

Ethyl (4-(1-(2-oxo-5-(*N*-(phenylsulfonyl)phenylsulfonamido)thiazol-3(2*H*)-yl)butyl)phenyl) carbamate (4af) [1a]:

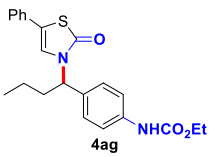


4af

The RASE π-bond migratory dealkylative C(sp^3)-N coupling between 0.3 mmol of ethyl (4-butylphenyl)carbamate (**1a**, 66.4 mg) and 0.6 mmol of *N*-(2-methoxythiazol-5-yl)-*N*-(phenylsulfonyl)benzenesulfonamide (**3f**, 246.0 mg) afforded 142.3 mg of **4af** (77%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 3:1, *v*/*v*) as the eluent.

White solid, m.p. 68-69 °C. **^{1}H NMR ($CDCl_3$, 400 MHz):** δ = 7.91 (d, *J* = 7.1 Hz, 4H), 7.67 (t, *J* = 7.5 Hz, 2H), 7.51 (t, *J* = 7.9 Hz, 4H), 7.40 (d, *J* = 8.4 Hz, 2H), 7.11 (d, *J* = 8.6 Hz, 2H), 7.02 (s, 1H), 6.25 (s, 1H), 5.43 (dd, *J* = 6.7 Hz, 2.5 Hz, 1H), 4.22 (q, *J* = 7.1 Hz, 2H), 1.99-1.80 (m, 2H), 1.34-1.28 (m, 5H), 0.95 (t, *J* = 7.4 Hz, 3H) ppm. **^{13}C NMR ($CDCl_3$, 100 MHz):** δ = 169.8, 153.5, 138.1, 138.1, 134.5, 133.3, 129.2, 128.4, 127.6, 127.3, 118.9, 109.9, 61.3, 56.7, 35.4, 19.3, 14.4, 13.5 ppm.

Ethyl (4-(1-(2-oxo-5-phenylthiazol-3(2*H*)-yl)butyl)phenyl)carbamate (4ag) [1a]:

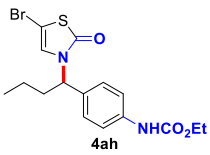


4ag

The RASE π-bond migratory dealkylative C(sp^3)-N coupling between 0.3 mmol of ethyl (4-butylphenyl)carbamate (**1a**, 66.4 mg) and 0.6 mmol of 2-methoxy-5-phenylthiazole (**3g**, 114.6 mg) afforded 50.1 mg of **4ag** (42%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 3:1, *v*/*v*) as the eluent.

Colorless oil. **^{1}H NMR ($CDCl_3$, 400 MHz):** δ = 7.43 (d, *J* = 8.3 Hz, 2H), 7.33-7.22 (m, 8H), 6.79 (s, 1H), 5.51 (dd, *J* = 6.8 Hz, 2.1 Hz, 1H), 4.20 (q, *J* = 7.1 Hz, 2H), 2.14-2.03 (m, 2H), 1.43-1.34 (m, 2H), 1.27 (t, *J* = 7.1 Hz, 3H), 0.98 (t, *J* = 7.4 Hz, 3H) ppm. **^{13}C NMR ($CDCl_3$, 100 MHz):** δ = 171.0, 153.6, 138.0, 133.7, 131.3, 128.8, 127.8, 127.6, 124.7, 118.8, 116.4, 61.1, 56.6, 35.2, 19.4, 14.4, 13.6 ppm.

Ethyl (4-(1-(5-bromo-2-oxothiazol-3(2*H*)-yl)butyl)phenyl)carbamate (4ah) [1a]:

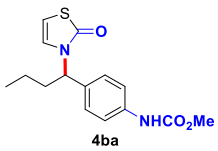


4ah

The RASE π-bond migratory dealkylative C(sp^3)-N coupling between 0.3 mmol of ethyl (4-butylphenyl)carbamate (**1a**, 66.4 mg) and 0.6 mmol of 5-bromo-2-methoxythiazole (**3h**, 116.4 mg) afforded 57.6 mg of **4ah** (48%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 3:1, *v*/*v*) as the eluent.

Colorless oil. **^{1}H NMR ($CDCl_3$, 400 MHz):** δ = 7.42 (d, *J* = 8.2 Hz, 2H), 7.24 (d, *J* = 8.6 Hz, 2H), 7.12 (s, 1H), 6.55 (s, 1H), 5.43 (dd, *J* = 6.8 Hz, 2.3 Hz, 1H), 4.21 (q, *J* = 7.1 Hz, 2H), 2.10-1.90 (m, 2H), 1.40-1.32 (m, 2H), 1.29 (t, *J* = 7.1 Hz, 3H), 0.97 (t, *J* = 7.4 Hz, 3H) ppm. **^{13}C NMR ($CDCl_3$, 100 MHz):** δ = 170.9, 153.6, 138.1, 133.3, 127.9, 122.2, 118.8, 88.9, 61.2, 56.9, 35.1, 19.4, 14.4, 13.5 ppm.

Methyl (4-(1-(2-oxothiazol-3(2*H*)-yl)butyl)phenyl)carbamate (4ba) [1a]:

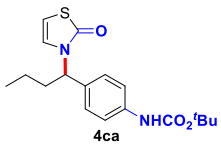


4ba

The RASE π-bond migratory dealkylative C(sp^3)-N coupling between 0.3 mmol of methyl (4-butylphenyl)carbamate (**1b**, 62.1 mg) and 0.6 mmol of 2-methoxythiazole (**3a**, 69.0 mg) afforded 74.8 mg of **4ba** (81%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 3:1, *v*/*v*) as the eluent.

White solid, m.p. 165-166 °C. **^{1}H NMR (DMSO-d_6, 400 MHz):** δ = 9.65 (s, 1H), 7.40 (d, *J* = 8.6 Hz, 2H), 7.23 (d, *J* = 8.7 Hz, 2H), 7.11 (d, *J* = 5.5 Hz, 1H), 6.40 (d, *J* = 5.5 Hz, 1H), 5.19 (dd, *J* = 6.3 Hz, 3.4 Hz, 1H), 3.61 (s, 3H), 2.11-1.91 (m, 2H), 1.21-1.13 (m, 2H), 0.86 (t, *J* = 7.4 Hz, 3H) ppm. **^{13}C NMR (DMSO-d_6, 100 MHz):** δ = 171.0, 154.0, 138.7, 134.0, 127.5, 123.4, 118.3, 101.1, 56.5, 51.6, 34.6, 19.1, 13.3 ppm.

***Tert*-Butyl (4-(1-(2-oxothiazol-3(2*H*)-yl)butyl)phenyl)carbamate (4ca) [1a]:**



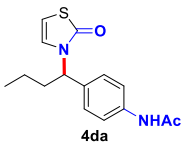
4ca

The RASE π-bond migratory dealkylative C(sp^3)-N coupling between 0.3 mmol of *tert*-butyl (4-butylphenyl)carbamate (**1c**, 74.8 mg) and 0.6 mmol of 2-methoxythiazole (**3a**, 69.0 mg) afforded 65.0 mg of **4ca** (62%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 3:1, *v*/*v*) as the eluent.

Colorless oil. **^{1}H NMR ($CDCl_3$, 400 MHz):** δ = 7.36 (d, *J* = 8.3 Hz, 2H), 7.22 (d, *J* = 8.6 Hz, 2H), 6.81 (s, 1H), 6.51 (d, *J* = 5.5 Hz, 1H), 6.05 (d, *J* = 5.5 Hz, 1H), 5.42 (dd, *J* = 6.8 Hz, 2.4 Hz, 1H), 2.10-1.94 (m, 2H), 1.50 (s, 9H), 1.37-1.28 (m, 2H), 0.96 (t, *J* = 7.4 Hz, 3H) ppm. **^{13}C NMR**

($CDCl_3$, 100 MHz): δ = 172.0, 152.7, 138.2, 133.7, 127.8, 121.7, 118.7, 101.3, 80.5, 56.5, 35.3, 28.2, 19.4, 13.6 ppm.

***N*-(4-(1-(2-oxothiazol-3(2*H*)-yl)butyl)phenyl)acetamide (4da) [1a]:**

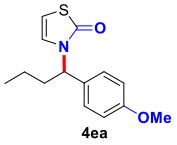


4da

The RASE π-bond migratory dealkylative C(sp^3)-N coupling between 0.3 mmol of *N*-(4-butylphenyl)acetamide (**1d**, 57.3 mg) and 0.6 mmol of 2-methoxythiazole (**3a**, 69.0 mg) afforded 59.6 mg of **4da** (68%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 2.5:1, *v*/*v*) as the eluent.

Colorless oil. **^{1}H NMR (DMSO-d_6, 400 MHz):** δ = 9.98 (s, 1H), 7.56 (d, *J* = 8.6 Hz, 2H), 7.28 (d, *J* = 8.6 Hz, 2H), 7.16 (d, *J* = 5.4 Hz, 1H), 6.46 (d, *J* = 5.4 Hz, 1H), 5.24 (dd, *J* = 6.3 Hz, 3.4 Hz, 1H), 2.16-1.96 (m, 5H), 1.26-1.18 (m, 2H), 0.90 (t, *J* = 7.4 Hz, 3H) ppm. **^{13}C NMR (DMSO-d_6, 100 MHz):** δ = 171.0, 168.3, 138.9, 134.5, 127.4, 123.4, 119.1, 101.2, 56.5, 34.6, 24.0, 19.1, 13.3 ppm.

3-(1-(4-methoxyphenyl)butyl)thiazol-2(3H)-one (4ea) [1a]:

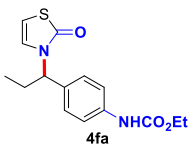


4ea

The RASE π-bond migratory dealkylative C(sp^3)-N coupling between 0.3 mmol of 1-butyl-4-methoxybenzene (**1e**, 49.2 mg) and 0.6 mmol of 2-methoxythiazole (**3a**, 69.0 mg) afforded 55.6 mg of **4ea** (70%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 4:1, *v*/*v*) as the eluent.

Light yellow oil. **^{1}H NMR ($CDCl_3$, 400 MHz):** δ = 7.25 (d, *J* = 8.6 Hz, 2H), 6.87 (d, *J* = 8.8 Hz, 2H), 6.52 (d, *J* = 5.5 Hz, 1H), 6.05 (d, *J* = 5.5 Hz, 1H), 5.43 (dd, *J* = 6.8 Hz, 2.2 Hz, 1H), 3.79 (s, 3H), 2.10-1.95 (m, 2H), 1.42-1.30 (m, 2H), 0.97 (t, *J* = 7.4 Hz, 3H) ppm. **^{13}C NMR ($CDCl_3$, 100 MHz):** δ = 171.9, 159.2, 131.4, 128.4, 121.7, 114.1, 101.2, 56.5, 55.2, 35.5, 19.5, 13.6 ppm.

Ethyl (4-(1-(2-oxothiazol-3(2*H*)-yl)propyl)phenyl)carbamate (4fa) [1a]:

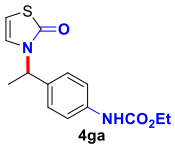


4fa

The RASE π-bond migratory dealkylative C(sp^3)-N coupling between 0.3 mmol of ethyl (4-propylphenyl)carbamate (**1f**, 62.1 mg) and 0.6 mmol of 2-methoxythiazole (**3a**, 69.0 mg) afforded 76.0 mg of **4fa** (83%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 3:1, *v*/*v*) as the eluent.

White solid, m.p. 146-147 °C. **^{1}H NMR ($CDCl_3$, 400 MHz):** δ = 7.39 (d, *J* = 8.4 Hz, 2H), 7.23 (d, *J* = 8.6 Hz, 2H), 7.00 (s, 1H), 6.51 (d, *J* = 5.5 Hz, 1H), 6.08 (d, *J* = 5.5 Hz, 1H), 5.33 (dd, *J* = 6.7 Hz, 2.5 Hz, 1H), 4.21 (q, *J* = 7.1 Hz, 2H), 2.20-1.96 (m, 2H), 1.29 (t, *J* = 7.1 Hz, 3H), 0.96 (t, *J* = 7.3 Hz, 3H) ppm. **^{13}C NMR ($CDCl_3$, 100 MHz):** δ = 172.2, 153.6, 137.9, 133.9, 127.9, 121.7, 118.8, 101.4, 61.2, 58.5, 26.5, 14.5, 10.8 ppm.

Ethyl (4-(1-(2-oxothiazol-3(2*H*)-yl)ethyl)phenyl)carbamate (4ga) [1a]:

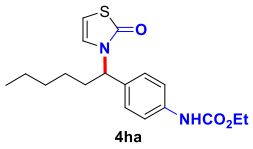


4ga

The RASE π-bond migratory dealkylative C(sp^3)-N coupling between 0.3 mmol of ethyl (4-ethylphenyl)carbamate (**1g**, 57.9 mg) and 0.6 mmol of 2-methoxythiazole (**3a**, 69.0 mg) afforded 62.5 mg of **4ga** (71%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 4:1, *v*/*v*) as the eluent.

Light yellow oil. **^{1}H NMR ($CDCl_3$, 400 MHz):** δ = 7.40 (d, *J* = 8.2 Hz, 2H), 7.22 (d, *J* = 8.5 Hz, 2H), 7.16 (s, 1H), 6.45 (d, *J* = 5.5 Hz, 1H), 6.07 (d, *J* = 5.4 Hz, 1H), 5.61 (q, *J* = 7.1 Hz, 1H), 4.21 (q, *J* = 7.1 Hz, 2H), 1.68 (d, *J* = 7.1 Hz, 3H), 1.29 (t, *J* = 7.1 Hz, 3H) ppm. **^{13}C NMR ($CDCl_3$, 100 MHz):** δ = 171.8, 153.6, 137.9, 134.5, 127.5, 121.8, 118.8, 101.4, 61.2, 52.2, 19.2, 14.5 ppm.

Ethyl (4-(1-(2-oxothiazol-3(2*H*)-yl)hexyl)phenyl)carbamate (4ha) [1a]:

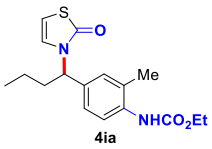


4ha

The RASE π-bond migratory dealkylative C(sp^3)-N coupling between 0.3 mmol of ethyl (4-hexylphenyl)carbamate (**1h**, 72.9 mg) and 0.6 mmol of 2-methoxythiazole (**3a**, 69.0 mg) afforded 65.1 mg of **4ha** (70%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 3:1, *v*/*v*) as the eluent.

Colorless oil. **^{1}H NMR ($CDCl_3$, 400 MHz):** δ = 7.39 (d, *J* = 8.3 Hz, 2H), 7.23 (d, *J* = 8.6 Hz, 2H), 7.07 (s, 1H), 6.53 (d, *J* = 5.5 Hz, 1H), 6.07 (d, *J* = 5.5 Hz, 1H), 5.41 (dd, *J* = 6.8 Hz, 2.4 Hz, 1H), 4.21 (q, *J* = 7.1 Hz, 2H), 2.12-1.94 (m, 2H), 1.34-1.26 (m, 9H), 0.86 (t, *J* = 7.0 Hz, 3H) ppm. **^{13}C NMR ($CDCl_3$, 100 MHz):** δ = 172.1, 153.6, 137.9, 134.0, 127.8, 121.7, 118.8, 101.4, 61.2, 56.9, 33.3, 31.3, 25.8, 22.3, 14.4, 13.9 ppm.

Ethyl (2-methyl-4-(1-(2-oxothiazol-3(2*H*)-yl)butyl)phenyl)carbamate (4ia):

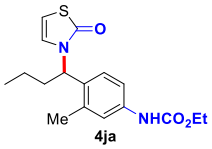


4ia

The RASE π-bond migratory dealkylative C(sp^3)-N coupling between 0.3 mmol of ethyl (4-butyl-2-methylphenyl)carbamate (**1i**, 70.5 mg) and 0.6 mmol of 2-methoxythiazole (**3a**, 69.0 mg) afforded 56.8 mg of **4ia** (57%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 3:1, *v*/*v*) as the eluent.

Light yellow oil. **^{1}H NMR ($CDCl_3$, 400 MHz):** δ = 7.79 (d, *J* = 8.4 Hz, 1H), 7.16 (dd, *J* = 8.5 Hz, 2.2 Hz, 1H), 7.10 (d, *J* = 2.3 Hz, 1H), 6.53 (d, *J* = 5.5 Hz, 1H), 6.46 (s, 1H), 6.06 (d, *J* = 5.5 Hz, 1H), 5.41 (dd, *J* = 6.8 Hz, 2.4 Hz, 1H), 4.22 (q, *J* = 7.1 Hz, 2H), 2.23 (s, 3H), 2.10-1.94 (m, 2H), 1.37-1.26 (m, 5H), 0.97 (t, *J* = 7.3 Hz, 3H) ppm. **^{13}C NMR ($CDCl_3$, 100 MHz):** δ = 172.0, 153.8, 135.7, 134.7, 129.7, 125.1, 121.7, 101.3, 61.3, 56.6, 35.4, 19.4, 17.7, 14.4, 13.6 ppm. **HRMS (ESI)** *m/z* calcd for $C_{17}H_{23}N_2O_3S$ $[M+H]^+$: 335.1424, found 335.1421.

Ethyl (3-methyl-4-(1-(2-oxothiazol-3(2*H*)-yl)butyl)phenyl)carbamate (4ja) [1a]:

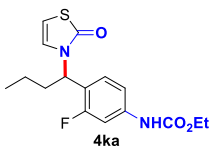


4ja

The RASE π-bond migratory dealkylative C(sp^3)-N coupling between 0.3 mmol of ethyl (4-butyl-3-methylphenyl)carbamate (**1j**, 70.5 mg) and 0.6 mmol of 2-methoxythiazole (**3a**, 69.0 mg) afforded 82.5 mg of **4ja** (82%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 3:1, *v*/*v*) as the eluent.

Light yellow oil. **^{1}H NMR ($CDCl_3$, 400 MHz):** δ = 7.38 (dd, *J* = 8.6 Hz, 2.3 Hz, 1H), 7.31 (d, *J* = 8.4 Hz, 1H), 7.19 (d, *J* = 2.3 Hz, 1H), 7.09 (s, 1H), 6.37 (d, *J* = 5.4 Hz, 1H), 6.02 (d, *J* = 5.4 Hz, 1H), 5.54 (t, *J* = 7.7 Hz, 1H), 4.21 (q, *J* = 7.1 Hz, 2H), 2.25 (s, 3H), 2.05-1.98 (m, 2H), 1.44-1.34 (m, 2H), 1.29 (t, *J* = 7.1 Hz, 3H), 0.97 (t, *J* = 7.3 Hz, 3H) ppm. **^{13}C NMR ($CDCl_3$, 100 MHz):** δ = 171.7, 153.6, 138.7, 137.9, 131.6, 126.6, 122.1, 120.9, 116.0, 101.2, 61.1, 53.5, 35.8, 19.5, 19.3, 14.4, 13.7 ppm.

Ethyl (3-fluoro-4-(1-(2-oxothiazol-3(2*H*)-yl)butyl)phenyl)carbamate (4ka) :



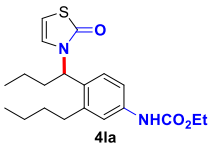
4ka

The RASE π-bond migratory dealkylative C(sp^3)-N coupling between 0.3 mmol of ethyl (4-butyl-3-fluorophenyl)carbamate (**1k**, 71.7 mg) and 0.6 mmol of 2-methoxythiazole (**3a**, 69.0 mg) afforded 53.0 mg of **4ka** (52%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 3:1, *v*/*v*) as

the eluent.

Light yellow solid, m.p. 98-99 °C. **^{1}H NMR ($CDCl_3$, 400 MHz):** δ = 7.35 (dd, *J* = 13.2 Hz, 2.1 Hz, 1H), 7.24 (t, *J* = 8.4 Hz, 1H), 7.14 (s, 1H), 7.03 (dd, *J* = 8.4 Hz, 2.2 Hz, 1H), 6.67 (dd, *J* = 5.5 Hz, 0.7 Hz, 1H), 6.09 (d, *J* = 5.5 Hz, 1H), 5.51 (t, *J* = 8.0 Hz, 1H), 4.21 (q, *J* = 7.1 Hz, 2H), 2.11-2.04 (m, 2H), 1.40-1.32 (m, 2H), 1.30 (t, *J* = 7.1 Hz, 3H), 0.96 (t, *J* = 7.4 Hz, 3H) ppm. **^{13}C NMR ($CDCl_3$, 100 MHz):** δ = 171.9, 161.0 (d, *J* = 245.5 Hz), 153.3, 139.8 (d, *J* = 11.5 Hz), 129.1 (d, *J* = 5.7 Hz), 122.4 (d, *J* = 2.7 Hz), 120.6 (d, *J* = 14.2 Hz), 113.9, 106.4 (d, *J* = 27.5 Hz), 101.2, 61.4, 53.0, 34.8, 19.6, 14.4, 13.5 ppm. **^{19}F NMR ($CDCl_3$, 376 MHz):** δ = -113.8 ppm. **HRMS (ESI)** *m/z* calcd for $C_{16}H_{20}FN_2O_3S$ $[M+H]^+$: 339.1173, found 339.1169.

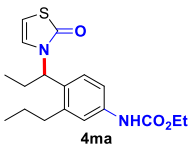
Ethyl (3-butyl-4-(1-(2-oxothiazol-3(2*H*)-yl)butyl)phenyl)carbamate (4la) [1a]:



4la

The RASE π-bond migratory dealkylative C(sp^3)-N coupling between 0.3 mmol of ethyl (3,4-dibutylphenyl)carbamate (**1l**, 83.2 mg) and 0.6 mmol of 2-methoxythiazole (**3a**, 69.0 mg) afforded 81.5 mg of **4la** (72%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 5:1, *v*/*v*) as the eluent. Colorless oil. **^{1}H NMR ($CDCl_3$, 400 MHz):** δ = 7.37-7.32 (m, 2H), 7.22 (d, *J* = 2.0 Hz, 1H), 6.92 (s, 1H), 6.38 (d, *J* = 5.5 Hz, 1H), 6.00 (d, *J* = 5.5 Hz, 1H), 5.61 (dd, *J* = 7.0 Hz, 1.3 Hz, 1H), 4.22 (q, *J* = 7.1 Hz, 2H), 2.69-2.51 (m, 2H), 2.09-1.93 (m, 2H), 1.49-1.28 (m, 9H), 0.97 (t, *J* = 7.4 Hz, 3H), 0.89 (t, *J* = 7.1 Hz, 3H) ppm. **^{13}C NMR ($CDCl_3$, 100 MHz):** δ = 171.5, 153.6, 143.8, 138.0, 130.9, 126.9, 122.1, 120.1, 116.0, 101.1, 61.2, 53.0, 36.1, 33.5, 32.0, 22.7, 19.6, 14.5, 14.0, 13.7 ppm.

Ethyl (4-(1-(2-oxothiazol-3(2*H*)-yl)propyl)-3-propylphenyl)carbamate (4ma) [1a]:

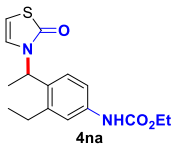


4ma

The RASE π-bond migratory dealkylative C(sp^3)-N coupling between 0.3 mmol of ethyl (3,4-dipropylphenyl)carbamate (**1m**, 74.8 mg) and 0.6 mmol of 2-methoxythiazole (**3a**, 69.0 mg) afforded 87.3 mg of **4ma** (84%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 5:1, *v*/*v*) as the eluent. Colorless oil. **^{1}H NMR ($CDCl_3$, 400 MHz):** δ = 7.37 (dd, *J* = 8.5 Hz, 2.3 Hz, 1H), 7.33 (d, *J* = 8.5 Hz, 1H), 7.23 (d, *J* = 2.3 Hz, 1H), 7.03 (s, 1H), 6.38 (d, *J* = 5.5 Hz, 1H), 6.00 (d, *J* = 5.5 Hz, 1H), 5.52 (t, *J* = 7.7 Hz, 1H), 4.21 (q, *J* = 7.1 Hz, 2H), 2.67-2.47 (m, 2H), 2.11-2.03 (m, 2H), 1.56-1.34

(m, 2H), 1.30 (t, *J* = 7.1 Hz, 3H), 0.97 (t, *J* = 7.3 Hz, 3H), 0.94 (t, *J* = 7.3 Hz, 3H) ppm. **^{13}C NMR (CDCl$_3$, 100 MHz):** δ = 171.6, 153.6, 143.5, 138.0, 130.7, 126.9, 122.1, 120.1, 116.0, 101.1, 61.1, 54.7, 34.1, 26.9, 24.4, 14.5, 13.9, 11.0 ppm.

Ethyl (3-ethyl-4-(1-(2-oxothiazol-3(2*H*)-yl)ethyl)phenyl)carbamate (4na) [1a]:

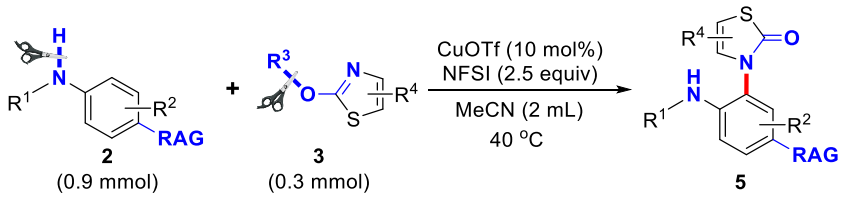


4na

The RASE π-bond migratory dealkylative C(sp^3)-N coupling between 0.3 mmol of ethyl (3,4-diethylphenyl)carbamate (**1n**, 66.3 mg) and 0.6 mmol of 2-methoxythiazole (**3a**, 69.0 mg) afforded 67.6 mg of **4na** (70%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 5:1, *v*/*v*) as the eluent. Light yellow solid, m.p. 144-145 °C. **^{1}H NMR (CDCl$_3$, 400 MHz):** δ = 7.39-7.35 (m, 2H), 7.24 (d, *J* = 2.0 Hz, 1H), 6.95 (s, 1H), 6.29 (d, *J* = 5.5 Hz, 1H), 6.00 (d, *J* = 5.5 Hz, 1H), 5.73 (q, *J* = 7.0 Hz, 1H), 4.22 (q, *J* = 7.1 Hz, 2H), 2.67-2.44 (m, 2H), 1.66 (d, *J* = 7.0 Hz, 3H), 1.30 (t, *J* = 7.1 Hz, 3H), 1.09 (t, *J* = 7.5 Hz, 3H) ppm. **^{13}C NMR (CDCl$_3$, 100 MHz):** δ = 171.2, 153.6, 144.3, 138.3, 131.4, 126.6, 122.1, 119.2, 116.0, 101.2, 61.2, 49.2, 24.7, 19.4, 14.6, 14.5 ppm.

2.2 General Procedure B

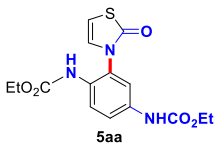
RASE *π*-bond migratory dealkylative C(sp^2)-N coupling with 2-alkoxylthiazoles offering *N*-phenyl thiazol-2(3*H*)-ones 5 (results shown in Table 4)



To a reaction tube charged with CuOTf (6.4 mg, 0.03 mmol, 10 mol%) and NFSI (236.5 mg, 0.75 mmol, 2.5 equiv) was added a solution of aniline (**2**, 0.9 mmol, 3 equiv) and 2-alkoxythiazole (**3**, 0.3 mmol, 1 equiv) in anhydrous MeCN (2 mL, 0.15 M of **3**) via a syringe under argon (1 atm) at 25 °C, and the reaction mixture was stirred for 4 hours at 40 °C (oil bath temperature). After quenched with Na_2CO_3 (aq.), the mixture was extracted with ethyl acetate. The combined organic phase was concentrated *in vacuo* to give dark residue, which was then purified by flash chromatography using petroleum ether and ethyl acetate as the eluent on silica gel to afford

corresponding *N*-phenyl thiazol-2(3*H*)-ones **5**.

Diethyl (2-(2-oxothiazol-3(2*H*)-yl)-1,4-phenylene)dicarbamate (5aa):



5aa

The RASE π-bond migratory dealkylative C(sp^2)-N coupling between 0.9 mmol of diethyl 1,4-phenylenedicarbamate (**2a**, 226.9 mg) and 0.3 mmol of 2-methoxythiazole (**3a**, 34.5 mg) afforded 69.8 mg of **5aa** (66%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (4:1 to 3:1, *v*/*v*) as the eluent.

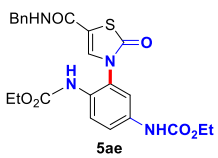
The RASE π-bond migratory dealkylative C(sp^2)-N coupling between 0.9 mmol of diethyl 1,4-phenylenedicarbamate (**2a**, 226.9 mg) and 0.3 mmol of 2-ethoxythiazole (**3b**, 38.7 mg) afforded 60.5 mg of **5aa** (57%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (4:1 to 3:1, *v*/*v*) as the eluent.

The RASE π-bond migratory dealkylative C(sp^2)-N coupling between 0.9 mmol of diethyl 1,4-phenylenedicarbamate (**2a**, 226.9 mg) and 0.3 mmol of 2-butoxythiazole (**3c**, 47.1 mg) afforded 51.2 mg of **5aa** (49%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (4:1 to 3:1, *v*/*v*) as the eluent.

The RASE π-bond migratory dealkylative C(sp^2)-N coupling between 0.9 mmol of diethyl 1,4-phenylenedicarbamate (**2a**, 226.9 mg) and 0.3 mmol of 2-(benzyloxy)thiazole (**3i**, 57.3 mg) afforded 67.5 mg of **5aa** (64%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (4:1 to 3:1, *v*/*v*) as the eluent.

Off-white solid, m.p. 207-213 °C. **^{1}H NMR (DMSO-d_6, 400 MHz):** δ = 9.82 (s, 1H), 8.84 (s, 1H), 7.51-7.42 (m, 3H), 6.90 (d, *J* = 5.4 Hz, 1H), 6.57 (d, *J* = 5.4 Hz, 1H), 4.13 (q, *J* = 7.1 Hz, 2H), 4.02 (q, *J* = 7.1 Hz, 2H), 1.24 (t, *J* = 7.1 Hz, 3H), 1.17 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (DMSO-d_6, 100 MHz):** δ = 170.3, 154.2, 153.6, 136.9, 130.9, 128.4, 126.7, 126.3, 118.6, 117.1, 101.8, 60.4, 14.5 ppm. **HRMS (ESI)** *m/z* calcd for $C_{15}H_{18}N_3O_5S$ $[M+H]^+$: 352.0962, found 352.0960.

Diethyl (2-(5-(benzylcarbamoyl)-2-oxothiazol-3(2*H*)-yl)-1,4-phenylene)dicarbamate (5ae):



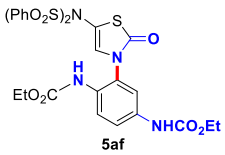
5ae

The RASE π-bond migratory dealkylative C(sp^2)-N coupling between 0.9 mmol of diethyl 1,4-phenylenedicarbamate (**2a**, 226.9 mg) and 0.3 mmol of *N*-benzyl-2-methoxythiazole-5-carboxamide (**3e**, 74.4 mg) afforded 85.1 mg of **5ae** (59%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (4:1

to 3:1, *v*/*v*) as the eluent.

White solid, m.p. 170-175 °C. **^{1}H NMR (DMSO-d_6, 400 MHz):** δ = 9.83 (s, 1H), 9.15 (s, 1H), 8.82 (t, *J* = 5.8 Hz, 1H), 7.85 (s, 1H), 7.56 (d, *J* = 2.3 Hz, 1H), 7.51-7.44 (m, 2H), 7.35-7.24 (m, 5H), 4.42 (d, *J* = 5.8 Hz, 2H), 4.15 (q, *J* = 7.1 Hz, 2H), 4.04 (q, *J* = 7.1 Hz, 2H), 1.24 (t, *J* = 7.2 Hz, 3H), 1.16 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (DMSO-d_6, 100 MHz):** δ = 169.2, 159.7, 154.1, 153.5, 139.0, 136.5, 129.4, 128.6, 128.3, 127.4, 126.9, 126.0, 119.1, 117.0, 113.6, 60.4, 42.4, 14.4, 14.4 ppm. **HRMS (ESI)** *m/z* calcd for $C_{23}H_{25}N_4O_6S$ $[M+H]^+$: 485.1489, found 485.1479.

Diethyl (2-(2-oxo-5-(*N*-(phenylsulfonyl)phenylsulfonamido)thiazol-3(2*H*)-yl)-1,4-phenylene)-dicarbamate (5af):

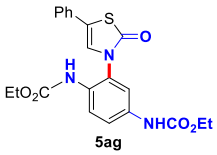


5af

The RASE π-bond migratory dealkylative C(sp^2)-N coupling between 0.9 mmol of diethyl 1,4-phenylenedicarbamate (**2a**, 226.9 mg) and 0.3 mmol of *N*-(2-methoxythiazol-5-yl)-*N*-(phenylsulfonyl)benzenesulfonamide (**3f**, 123.0 mg) afforded 63.5 mg of **5af** (33%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (4:1 to 2.5:1, *v*/*v*) as the eluent.

White solid, m.p. 191-196 °C. **^{1}H NMR (DMSO-d_6, 400 MHz):** δ = 9.85 (s, 1H), 9.07 (s, 1H), 7.98-7.96 (m, 4H), 7.87-7.84 (m, 2H), 7.71 (t, *J* = 7.9 Hz, 4H), 7.52 (d, *J* = 2.4 Hz, 1H), 7.48 (dd, *J* = 8.8 Hz, 2.4 Hz, 1H), 7.36 (d, *J* = 8.8 Hz, 1H), 7.11 (s, 1H), 4.15 (d, *J* = 7.1 Hz, 2H), 4.07 (q, *J* = 7.1 Hz, 2H), 1.25 (t, *J* = 7.1 Hz, 3H), 1.17 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (DMSO-d_6, 100 MHz):** δ = 167.6, 154.2, 153.5, 137.7, 137.1, 135.2, 132.1, 130.2, 129.7, 128.5, 128.2, 126.9, 119.1, 117.4, 108.4, 60.5, 60.4, 14.4, 14.3 ppm. **HRMS (ESI)** *m/z* calcd for $C_{27}H_{27}N_4O_9S_3$ $[M+H]^+$: 647.0935, found 647.0933.

Diethyl (2-(2-oxo-5-phenylthiazol-3(2*H*)-yl)-1,4-phenylene)dicarbamate (5ag):

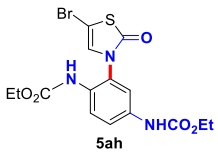


5ag

The RASE π-bond migratory dealkylative C(sp^2)-N coupling between 0.9 mmol of diethyl 1,4-phenylenedicarbamate (**2a**, 226.9 mg) and 0.3 mmol of 2-methoxy-5-phenylthiazole (**3g**, 57.3 mg) afforded 79.6 mg of **5ag** (62%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (4:1 to 3:1, *v*/*v*) as the eluent.

White solid, m.p. 163-168 °C. **^{1}H NMR (DMSO-d_6, 400 MHz):** δ = 9.82 (s, 1H), 8.99 (s, 1H), 7.57-7.55 (m, 2H), 7.49-7.47 (m, 4H), 7.41 (t, *J* = 7.7 Hz, 2H), 7.30 (t, *J* = 7.3 Hz, 1H), 4.14 (q, *J* = 7.1 Hz, 2H), 4.01 (q, *J* = 7.1 Hz, 2H), 1.25 (t, *J* = 7.1 Hz, 3H), 1.11 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (DMSO-d_6, 100 MHz):** δ = 168.7, 154.2, 153.5, 136.7, 131.2, 129.0, 128.6, 127.6, 124.4, 122.4, 118.8, 117.3, 116.7, 60.3, 14.4, 14.3 ppm. **HRMS (ESI)** *m/z* calcd for $C_{21}H_{22}N_3O_5S$ $[M+H]^+$: 428.1275, found 428.1268.

Diethyl (2-(5-bromo-2-oxothiazol-3(2*H*)-yl)-1,4-phenylene)dicarbamate (5ah):

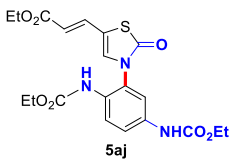


5ah

The RASE π-bond migratory dealkylative C(sp^2)-N coupling between 0.9 mmol of diethyl 1,4-phenylenedicarbamate (**2a**, 226.9 mg) and 0.3 mmol of 5-bromo-2-methoxythiazole (**3h**, 58.2 mg) afforded 50.6 mg of **5ah** (39%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (5:1, *v*/*v*) as the eluent.

Light yellow solid, m.p. 152-158 °C. **^{1}H NMR (DMSO-d_6, 400 MHz):** δ = 9.81 (s, 1H), 9.06 (s, 1H), 7.49-7.45 (m, 3H), 7.27 (s, 1H), 4.13 (q, *J* = 7.1 Hz, 2H), 4.05 (q, *J* = 7.1 Hz, 2H), 1.24 (t, *J* = 7.1 Hz, 3H), 1.19 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (DMSO-d_6, 100 MHz):** δ = 168.8, 154.1, 153.5, 136.5, 129.4, 128.7, 127.4, 125.9, 118.9, 117.2, 87.4, 60.4, 14.4 ppm. **HRMS (ESI)** *m/z* calcd for $C_{15}H_{17}BrN_3O_5S$ $[M+H]^+$: 430.0066, found 430.0057.

Diethyl (*E*)-3-(3-(2,5-bis((ethoxycarbonyl)amino)phenyl)-2-oxo-2,3-dihydrothiazol-5-yl)-acrylate (5aj):



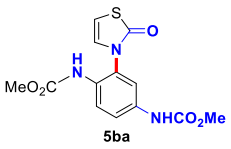
5aj

The RASE π-bond migratory dealkylative C(sp^2)-N coupling between 0.9 mmol of diethyl 1,4-phenylenedicarbamate (**2a**, 226.9 mg) and 0.3 mmol of ethyl (*E*)-3-(2-methoxythiazol-5-yl)acrylate (**3j**, 63.9 mg) afforded 58.9 mg of **5aj** (44%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (4:1 to 3:1, *v*/*v*) as the eluent.

White solid, m.p. 182-187 °C. **^{1}H NMR (DMSO-d_6, 400 MHz):** δ = 9.82 (s, 1H), 9.12 (s, 1H), 7.67-7.63 (m, 2H), 7.52-7.47 (m, 3H), 5.83 (d, *J* = 15.5 Hz, 1H), 4.19-4.10 (m, 4H), 4.03 (q, *J* = 7.1 Hz, 2H), 1.26-1.22 (m, 6H), 1.16 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (DMSO-d_6, 100 MHz):** δ =

168.3, 165.6, 154.1, 153.5, 136.6, 135.7, 132.6, 128.6, 119.1, 117.1, 115.5, 114.2, 60.4, 60.0, 14.4, 14.3, 14.1 ppm. **HRMS (ESI)** *m/z* calcd for $C_{20}H_{24}N_3O_7S$ $[M+H]^+$: 450.1130, found 450.1133.

Dimethyl (2-(2-oxothiazol-3(2*H*)-yl)-1,4-phenylene)dicarbamate (5ba):

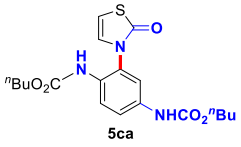


5ba

The RASE π-bond migratory dealkylative C(sp^2)-N coupling between 0.9 mmol of dimethyl 1,4-phenylenedicarbamate (**2b**, 201.7 mg) and 0.3 mmol of 2-methoxythiazole (**3a**, 34.5 mg) afforded 71.1 mg of **5ba** (73%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (3:1 to 2:1, *v*/*v*) as the eluent.

White solid, m.p. 177-182 °C. **^{1}H NMR (DMSO-d_6, 400 MHz):** δ = 9.79 (s, 1H), 8.83 (s, 1H), 7.44-7.41 (m, 3H), 6.87 (d, *J* = 5.4 Hz, 1H), 6.54 (d, *J* = 5.4 Hz, 1H), 3.63 (s, 3H), 3.53 (s, 3H). **^{13}C NMR (DMSO-d_6, 100 MHz):** δ = 170.3, 154.6, 154.0, 136.8, 130.8, 128.4, 126.6, 126.2, 118.6, 117.1, 101.8, 51.8, 51.8 ppm. **HRMS (ESI)** *m/z* calcd for $C_{13}H_{14}N_3O_5S$ $[M+H]^+$: 324.0649, found 324.0642.

Dibutyl (2-(2-oxothiazol-3(2*H*)-yl)-1,4-phenylene)dicarbamate (5ca):

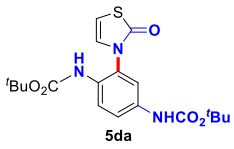


5ca

The RASE π-bond migratory dealkylative C(sp^2)-N coupling between 0.9 mmol of dibutyl 1,4-phenylenedicarbamate (**2c**, 277.4 mg) and 0.3 mmol of 2-methoxythiazole (**3a**, 34.5 mg) afforded 84.3 mg of **5ca** (69%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 4:1, *v*/*v*) as the eluent.

White solid, m.p. 280-287 °C. **^{1}H NMR (DMSO-d_6, 400 MHz):** δ = 9.75 (s, 1H), 8.75 (s, 1H), 7.47 (d, *J* = 2.4 Hz, 1H), 7.42 (dd, *J* = 8.8 Hz, 2.4 Hz, 1H), 7.37 (d, *J* = 8.8 Hz, 1H), 6.85 (d, *J* = 5.5 Hz, 1H), 6.52 (d, *J* = 5.4 Hz, 1H), 4.05 (t, *J* = 6.6 Hz, 2H), 3.95 (t, *J* = 6.6 Hz, 2H), 1.61-1.46 (m, 4H), 1.37-1.25 (m, 4H), 0.88 (t, *J* = 7.3 Hz, 3H), 0.85 (t, *J* = 7.4 Hz, 3H). **^{13}C NMR (DMSO-d_6, 100 MHz):** δ = 170.2, 154.3, 153.6, 136.9, 131.0, 128.3, 126.8, 126.2, 118.5, 117.1, 101.6, 64.0, 30.5, 18.6, 18.5, 13.5 ppm. **HRMS (ESI)** *m/z* calcd for $C_{19}H_{26}N_3O_5S$ $[M+H]^+$: 408.1587, found 408.1578.

Di-*tert*-butyl (2-(2-oxothiazol-3(2*H*)-yl)-1,4-phenylene)dicarbamate (5da):

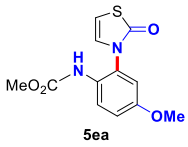


5da

The RASE π-bond migratory dealkylative C(sp^2)-N coupling between 0.9 mmol of di-*tert*-butyl 1,4-phenylenedicarbamate (**2d**, 277.4 mg) and 0.3 mmol of 2-methoxythiazole (**3a**, 34.5 mg) afforded 83.9 mg of **5da** (69%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 4:1, *v*/*v*) as the eluent.

White solid, m.p. 232-239 °C. **^{1}H NMR (DMSO-d_6, 400 MHz):** δ = 9.48 (s, 1H), 8.44 (s, 1H), 7.50 (d, *J* = 2.5 Hz, 1H), 7.35 (dd, *J* = 8.8 Hz, 2.5 Hz, 1H), 7.26 (d, *J* = 8.8 Hz, 1H), 6.83 (d, *J* = 5.4 Hz, 1H), 6.52 (d, *J* = 5.4 Hz, 1H), 1.44 (s, 9H), 1.34 (s, 9H). **^{13}C NMR (DMSO-d_6, 100 MHz):** δ = 170.1, 153.2, 152.7, 137.2, 131.4, 128.0, 126.9, 126.2, 118.4, 117.0, 101.4, 79.3, 78.8, 28.0, 27.9 ppm. **HRMS (ESI)** *m/z* calcd for $C_{19}H_{26}N_3O_5S$ $[M+H]^+$: 408.1587, found 408.1581.

Methyl (4-methoxy-2-(2-oxothiazol-3(2*H*)-yl)phenyl)carbamate (5ea):

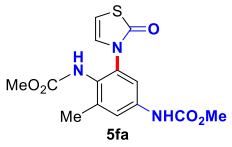


5ea

The RASE π-bond migratory dealkylative C(sp^2)-N coupling between 0.9 mmol of methyl (4-methoxyphenyl)carbamate (**2e**, 163.0 mg) and 0.3 mmol of 2-methoxythiazole (**3a**, 34.5 mg) afforded 43.6 mg of **5ea** (51%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (6:1 to 4:1, *v*/*v*) as the eluent.

White solid, m.p. 107-114 °C. **^{1}H NMR (DMSO-d_6, 400 MHz):** δ = 8.74 (s, 1H), 7.39 (d, *J* = 8.8 Hz, 1H), 7.01 (dd, *J* = 8.8 Hz, 3.0 Hz, 1H), 6.91 (d, *J* = 3.0 Hz, 1H), 6.57 (d, *J* = 5.4 Hz, 1H), 3.77 (s, 3H), 3.55 (s, 3H). **^{13}C NMR (DMSO-d_6, 100 MHz):** δ = 170.2, 156.9, 154.8, 131.9, 127.7, 126.7, 126.3, 114.8, 112.9, 101.5, 55.6, 51.7 ppm. **HRMS (ESI)** *m/z* calcd for $C_{12}H_{13}N_2O_4S$ $[M+H]^+$: 281.0591, found 281.0584.

Dimethyl (2-methyl-6-(2-oxothiazol-3(2*H*)-yl)-1,4-phenylene)dicarbamate (5fa):



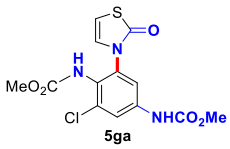
5fa

The RASE π-bond migratory dealkylative C(sp^2)-N coupling between 0.9 mmol of dimethyl (2-methyl-1,4-phenylene)dicarbamate (**2f**, 214.3 mg) and 0.3 mmol of 2-methoxythiazole (**3a**, 34.5 mg) afforded 76.1 mg of **5fa** (75%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (4:1 to 3:1, *v*/*v*) as the eluent.

Light brown solid, m.p. 147-151 °C. **^{1}H NMR (DMSO-d_6, 400 MHz):** δ = 9.83 (s, 1H), 8.62 (s, 1H), 7.40 (d, *J* = 2.5 Hz, 1H), 7.38 (d, *J* = 2.5 Hz, 1H), 6.79 (d, *J* = 5.1 Hz, 1H), 6.55 (d, *J* = 5.4

Hz, 1H), 3.68 (s, 3H), 3.55 (s, 3H), 2.19 (s, 3H). **^{13}C NMR (DMSO-d_6, 100 MHz):** δ = 170.0, 155.0, 154.0, 138.0, 134.5, 126.8, 126.3, 124.4, 119.7, 115.0, 101.2, 54.9, 51.8, 18.1 ppm. **HRMS (ESI)** *m/z* calcd for $C_{14}H_{16}N_3O_5S$ $[M+H]^+$: 338.0805, found 338.0798.

Dimethyl (2-chloro-6-(2-oxothiazol-3(2*H*)-yl)-1,4-phenylene)dicarbamate (5ga):

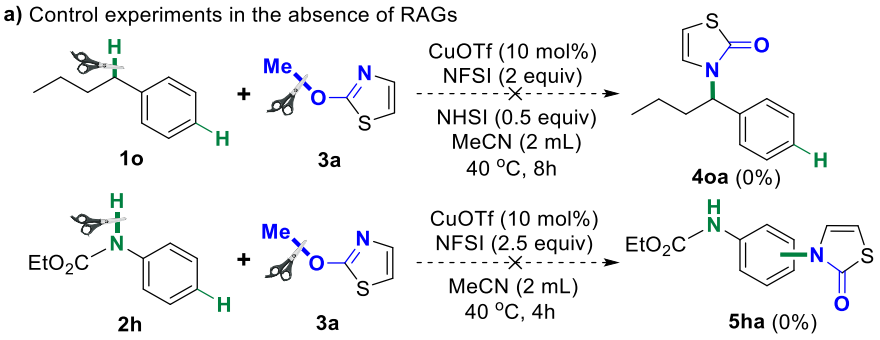


5ga

The RASE π-bond migratory dealkylative C(sp^2)-N coupling between 0.9 mmol of dimethyl (2-chloro-1,4-phenylene)dicarbamate (**2g**, 232.8 mg) and 0.3 mmol of 2-methoxythiazole (**3a**, 34.5 mg) afforded 69.0 mg of **5ga** (64%) after flash chromatography on silica gel using petroleum ether and ethyl acetate (4:1 to 3:1, *v*/*v*) as the eluent.

Light brown solid, m.p. 155-165 °C. **^{1}H NMR (DMSO-d_6, 400 MHz):** δ = 10.11 (s, 1H), 8.98 (s, 1H), 7.75 (d, *J* = 2.5 Hz, 1H), 7.48 (d, *J* = 2.5 Hz, 1H), 6.83 (t, *J* = 3.0 Hz, 1H), 6.60 (d, *J* = 5.4 Hz, 1H), 3.70 (s, 3H), 3.56 (s, 3H). **^{13}C NMR (DMSO-d_6, 100 MHz):** δ = 170.0, 153.9, 139.0, 136.1, 133.7, 125.7, 118.4, 116.3, 101.7, 54.9, 52.1 ppm. **HRMS (ESI)** *m/z* calcd for $C_{13}H_{13}ClN_3O_5S$ $[M+H]^+$: 358.0259, found 358.0249.

3. Control Experiments Demonstrating the Significance of the RAG (Results Shown in Figure 2)



To a reaction tube charged with CuOTf (6.4 mg, 0.03 mmol, 10 mol%), NFSI (189.2 mg, 0.6 mmol, 2 equiv) and NHSI (44.6 mg, 0.15 mmol, 0.5 equiv) was added a solution of butylbenzene (**1o**, 40.2 mg, 0.3 mmol, 1 equiv) and 2-methoxythiazole (**3a**, 69.0 mg, 0.6 mmol, 2 equiv) in anhydrous MeCN (2 mL, 0.15 M of **1o**) via a syringe under argon (1 atm) at 25 °C, and the reaction mixture was stirred for 8 hours at 40 °C (oil bath temperature). TLC and LC-MS suggested that no *N*-benzyl thiazol-2(3*H*)-one **4oa** was generated. After quenched with Na_2CO_3 (aq.), the mixture was extracted with ethyl acetate. The combined organic phase was concentrated *in vacuo* to give dark residue, which was then purified by flash chromatography using petroleum ether and ethyl acetate as the eluent on silica gel to recover 37.7 mg of **1o** (94%).

Butylbenzene (1o) [2a]

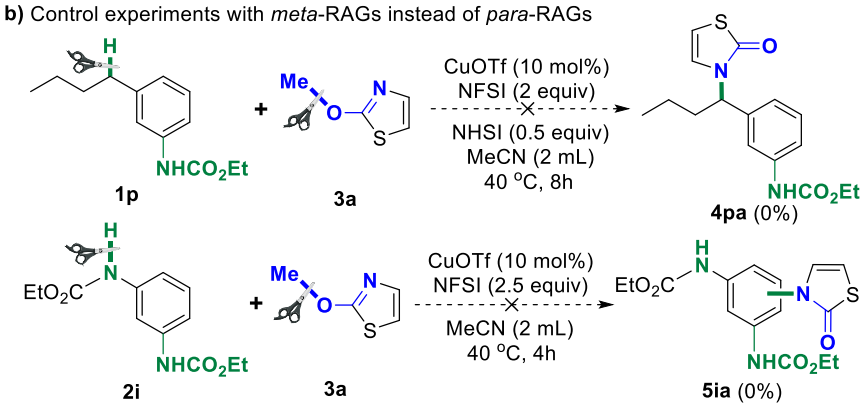
Colorless oil. **^{1}H NMR (DMSO-d_6, 400 MHz):** δ = 7.28-7.24 (m, 2H), 7.18-7.13 (m, 3H), 2.56 (t, *J* = 7.7 Hz, 2H), 1.53 (p, *J* = 7.6 Hz, 2H), 1.29 (sext, *J* = 7.4 Hz, 2H), 0.88 (t, *J* = 7.4 Hz, 3H) ppm. **^{13}C NMR (DMSO-d_6, 100 MHz):** δ = 142.3, 128.2, 128.2, 125.6, 34.9, 33.2, 21.8, 13.8 ppm.

To a reaction tube charged with CuOTf (6.4 mg, 0.03 mmol, 10 mol%) and NFSI (236.5 mg, 0.75 mmol, 2.5 equiv) was added a solution of ethyl phenylcarbamate (**2h**, 148.6 mg, 0.9 mmol, 3 equiv) and 2-methoxythiazole (**3a**, 34.5 mg, 0.3 mmol, 1 equiv) in anhydrous MeCN (2 mL, 0.15 M of **3a**) via a syringe under argon (1 atm) at 25 °C, and the reaction mixture was stirred for 4 hours at 40 °C (oil bath temperature). TLC and LC-MS suggested that no *N*-phenyl thiazol-2(3*H*)-one **5ha** was generated. After quenched with Na_2CO_3 (aq.), the mixture was

extracted with ethyl acetate. The combined organic phase was concentrated *in vacuo* to give dark residue, which was then purified by flash chromatography using petroleum ether and ethyl acetate as the eluent on silica gel to recover 129.7 mg of **2h** (87%).

Ethyl phenylcarbamate (2h) [2b]

Off-white solid, m.p. 50-52 °C. **^{1}H NMR (DMSO-d_6, 400 MHz):** δ = 9.61 (s, 1H), 7.47 (d, *J* = 8.0 Hz, 2H), 7.26 (t, *J* = 7.9 Hz, 2H), 6.97 (d, *J* = 7.5 Hz, 2H), 4.12 (p, *J* = 7.1 Hz, 2H), 1.23 (t, *J* = 7.1 Hz, 3H) ppm. **^{13}C NMR (DMSO-d_6, 100 MHz):** δ = 153.6, 139.3, 128.7, 122.3, 118.1, 60.1, 14.5 ppm.



To a reaction tube charged with CuOTf (6.4 mg, 0.03 mmol, 10 mol%), NFSI (189.2 mg, 0.6 mmol, 2 equiv) and NHSI (44.6 mg, 0.15 mmol, 0.5 equiv) was added a solution of ethyl (3-butylphenyl)carbamate (**1p**, 66.4 mg, 0.3 mmol, 1 equiv) and 2-methoxythiazole (**3a**, 69.0 mg, 0.6 mmol, 2 equiv) in anhydrous MeCN (2 mL, 0.15 M of **1p**) via a syringe under argon (1 atm) at 25 °C, and the reaction mixture was stirred for 8 hours at 40 °C (oil bath temperature). TLC and LC-MS suggested that no *N*-benzyl thiazol-2(3*H*)-one **4pa** was generated. After quenched with Na_2CO_3 (aq.), the mixture was extracted with ethyl acetate. The combined organic phase was concentrated *in vacuo* to give dark residue, which was then purified by flash chromatography using petroleum ether and ethyl acetate as the eluent on silica gel to recover 64.2 mg of **1p** (97%).

Ethyl (3-butylphenyl)carbamate (1p) [1a]

Colorless oil. **^{1}H NMR (DMSO-d_6, 400 MHz):** δ = 9.49 (s, 1H), 7.29 (s, 1H), 7.23 (d, *J* = 7.9 Hz, 1H), 7.10 (t, *J* = 7.8 Hz, 1H), 6.75 (d, *J* = 7.6 Hz, 1H), 4.07 (q, *J* = 7.1 Hz, 2H), 2.46 (t, *J* = 7.7 Hz, 2H), 1.47 (p, *J* = 7.6 Hz, 2H), 1.29-1.18 (m, 5H), 0.84 (t, *J* = 7.3 Hz, 3H) ppm. **^{13}C NMR (DMSO-d_6, 100 MHz):** δ = 153.6, 142.8, 139.2, 128.5, 122.3, 118.0, 115.6, 60.0, 35.0, 33.2, 21.8, 14.5, 13.8 ppm.

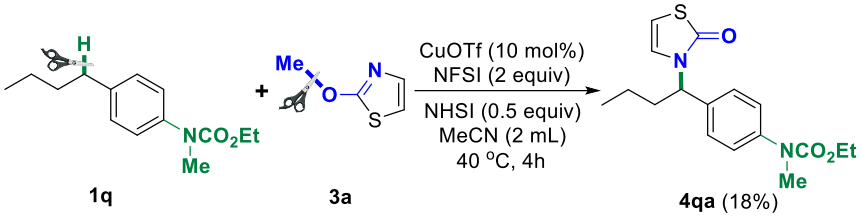
To a reaction tube charged with CuOTf (6.4 mg, 0.03 mmol, 10 mol%) and NFSI (236.5 mg, 0.75 mmol, 2.5 equiv) was added a solution of diethyl 1,3-phenylenedicarbamate (**2i**, 226.9 mg, 0.9 mmol, 3 equiv) and 2-methoxythiazole (**3a**, 34.5 mg, 0.3 mmol, 1 equiv) in anhydrous MeCN (2 mL, 0.15 M of **3a**) via a syringe under argon (1 atm) at 25 °C, and the reaction mixture was stirred for 4 hours at 40 °C (oil bath temperature). TLC and LC-MS suggested that no *N*-phenyl thiazol-2(3*H*)-one **5ia** was generated. After quenched with Na_2CO_3 (aq.), the mixture was

extracted with ethyl acetate. The combined organic phase was concentrated *in vacuo* to give dark residue, which was then purified by flash chromatography using petroleum ether and ethyl acetate as the eluent on silica gel to recover 206.1 mg of **2i** (91%).

Diethyl 1,3-phenylenedicarbamate (2i) [2c]

White solid, m.p. 112-114 ºC. **^{1}H NMR (DMSO-d_6, 400 MHz):** δ = 9.58 (s, 2H), 7.71 (s, 1H), 7.16-7.07 (m, 3H), 4.10 (q, *J* = 7.1 Hz, 2H), 1.23 (t, *J* = 7.1 Hz, 3H) ppm. **^{13}C NMR (DMSO-d_6, 100 MHz):** δ = 153.6, 139.7, 128.8, 112.7, 108.7, 60.1, 14.6 ppm.

c) Control experiment with a carbamate in the absence of free N-H bond as the *para*-RAG

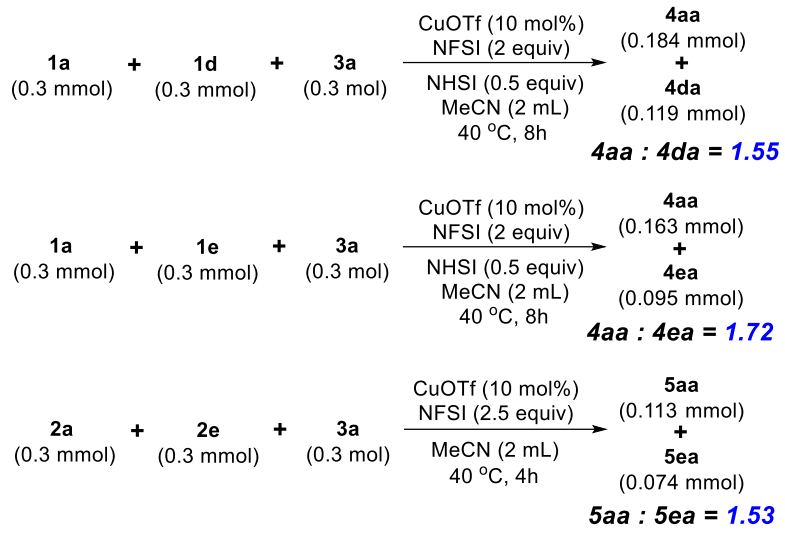


To a reaction tube charged with CuOTf (6.4 mg, 0.03 mmol, 10 mol%), NFSI (189.2 mg, 0.6 mmol, 2 equiv) and NHSI (44.6 mg, 0.15 mmol, 0.5 equiv) was added a solution of ethyl (4-butylphenyl)(methyl)carbamate (**1q**, 70.6 mg, 0.3 mmol, 1 equiv) and 2-methoxythiazole (**3a**, 69.0 mg, 0.6 mmol, 2 equiv) in anhydrous MeCN (2 mL, 0.15 M of **1p**) via a syringe under argon (1 atm) at 25 °C, and the reaction mixture was stirred for 8 hours at 40 °C (oil bath temperature). After quenched with Na_2CO_3 (aq.), the mixture was extracted with ethyl acetate. The combined organic phase was concentrated *in vacuo* to give dark residue, which was then purified by flash chromatography using petroleum ether and ethyl acetate (4:1 to 3:1, *v*/*v*) as the eluent on silica gel to afforded 18.2 mg of **4qa** (18%).

Ethyl methyl(4-(1-(2-oxothiazol-3(2*H*)-yl)butyl)phenyl)carbamate (4qa) [1a]

Light yellow oil. **^{1}H NMR ($CDCl_3$, 400 MHz):** δ = 7.30-7.22 (m, 4H), 6.57 (d, *J* = 5.2 Hz, 1H), 6.10 (d, *J* = 5.2 Hz, 1H), 5.46 (t, *J* = 8.1 Hz, 1H), 4.17 (q, *J* = 7.0 Hz, 2H), 3.29 (s, 3H), 2.13-1.96 (m, 2H), 1.42-1.33 (m, 2H), 1.25 (t, *J* = 7.3 Hz, 3H), 0.98 (t, *J* = 7.3 Hz, 3H) ppm. **^{13}C NMR ($CDCl_3$, 100 MHz):** δ = 172.1, 155.5, 143.0, 136.8, 127.5, 125.6, 121.7, 101.5, 61.8, 56.5, 37.3, 35.5, 19.5, 14.6, 13.6 ppm.

d) Comparison between the carbamte RAG and other RAGs



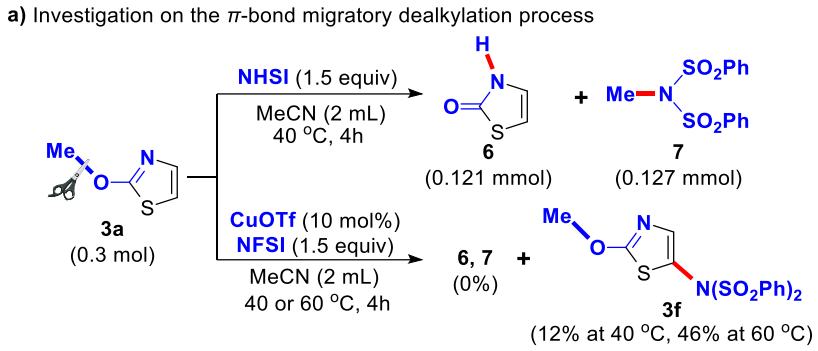
To a reaction tube charged with CuOTf (6.4 mg, 0.03 mmol, 10 mol%), NFSI (189.2 mg, 0.6 mmol, 2 equiv) and NHSI (44.6 mg, 0.15 mmol, 0.5 equiv) was added a solution of ethyl (4-butylphenyl)carbamate (**1a**, 66.4 mg, 0.3 mmol, 1 equiv), *N*-(4-butylphenyl)acetamide (**1d**, 57.3 mg, 0.3 mmol, 1 equiv) and 2-methoxythiazole (**3a**, 34.5 mg, 0.3 mmol, 1 equiv) in anhydrous MeCN (2 mL) via a syringe under argon (1 atm) at 25 °C, and the reaction mixture was stirred for 8 hours at 40 °C (oil bath temperature). After quenched with Na_2CO_3 (aq.), the mixture was extracted with ethyl acetate. The combined organic phase was concentrated *in vacuo* to give dark residue, which was then purified by flash chromatography using petroleum ether and ethyl acetate (4:1 to 3:1 to 3:2, *v*/*v*) as the eluent on silica gel to afforded 58.9 mg of **4aa** (0.184 mmol, 61%) and 34.5 mg of **4da** (0.119 mmol, 40%).

To a reaction tube charged with CuOTf (6.4 mg, 0.03 mmol, 10 mol%), NFSI (189.2 mg, 0.6 mmol, 2 equiv) and NHSI (44.6 mg, 0.15 mmol, 0.5 equiv) was added a solution of ethyl (4-butylphenyl)carbamate (**1a**, 66.4 mg, 0.3 mmol, 1 equiv), 1-butyl-4-methoxybenzene (**1e**, 49.2 mg, 0.3 mmol, 1 equiv) and 2-methoxythiazole (**3a**, 34.5 mg, 0.3 mmol, 1 equiv) in anhydrous MeCN (2 mL) via a syringe under argon (1 atm) at 25 °C, and the reaction mixture was stirred for 8 hours at 40 °C (oil bath temperature). After quenched with Na_2CO_3 (aq.), the mixture was extracted with ethyl acetate. The combined organic phase was concentrated *in vacuo* to give dark residue, which was then purified by flash chromatography using petroleum ether and ethyl acetate (4:1 to 3:1 to 3:2, *v*/*v*) as the eluent on silica gel to afforded 52.2 mg of **4aa** (0.163 mmol, 54%)

and 25.0 mg of **4ea** (0.095 mmol, 32%).

To a reaction tube charged with CuOTf (6.4 mg, 0.03 mmol, 10 mol%) and NFSI (236.5 mg, 0.75 mmol, 2.5 equiv) was added a solution of diethyl 1,4-phenylenedicarbamate (**2a**, 75.6 mg, 0.3 mmol, 1 equiv), methyl (4-methoxyphenyl)carbamate (**2e**, 54.3 mg, 0.3 mmol, 1 equiv) and 2-methoxythiazole (**3a**, 34.5 mg, 0.3 mmol, 1 equiv) in anhydrous MeCN (2 mL) via a syringe under argon (1 atm) at 25 °C, and the reaction mixture was stirred for 4 hours at 40 °C (oil bath temperature). After quenched with Na_2CO_3 (aq.), the mixture was extracted with ethyl acetate. The combined organic phase was concentrated *in vacuo* to give dark residue, which was then purified by flash chromatography using petroleum ether and ethyl acetate (6:1 to 4:1 to 3:1, *v*/*v*) as the eluent on silica gel to afforded 39.7 mg of **5aa** (0.113 mmol, 38%) and 20.7 mg of **5ea** (0.074 mmol, 25%).

4. Control Experiments Demonstrating the Significance of the Radical Relay Process to the π-Bond Migratory Dealkylation (Results Shown in Figure 3)



To a reaction tube charged with NHSI (133.7 mg, 0.45 mmol, 1.5 equiv) was added a solution of 2-methoxythiazole (**3a**, 34.5 mg, 0.3 mmol, 1 equiv) in anhydrous MeCN (2 mL, 0.15 M of **3a**) via a syringe under argon (1 atm) at 25 °C, and the reaction mixture was stirred for 4 hours at 40 °C (oil bath temperatu. After quenched with Na_2CO_3 (aq.), the mixture was extracted with ethyl acetate. The combined organic phase was concentrated *in vacuo* to give dark residue, which was then purified by flash chromatography using petroleum ether and ethyl acetate as the eluent on silica gel to afford 12.2 mg of **6** (0.121 mmol, 40%) and 39.5 mg of **7** (0.127 mmol, 42%).

Thiazol-2(3*H*)-one (6) [1a, 3]**:**

Yellow solid. **^{1}H NMR (DMSO-d_6, 400 MHz):** δ = 11.16 (s, 1H), 6.81 (dd, *J* = 5.4 Hz, 2.7 Hz, 1H), 6.34 (dd, *J* = 5.4 Hz, 1.6 Hz, 1H) ppm. **^{13}C NMR ($CDCl_3$, 100 MHz):** δ = 176.6, 121.0, 103.7 ppm.

***N*-methylbenzenesulfonimide (NMSI, 7)** [4]**:**

Colorless oil. **^{1}H NMR ($CDCl_3$, 400 MHz):** δ = 8.03-8.00 (m, 4H), 7.68-7.65 (m, 2H), 7.58-7.54 (m, 4H), 3.29 (s, 3H) ppm. **^{13}C NMR ($CDCl_3$, 100 MHz):** δ = 139.0, 133.9, 129.1, 127.9, 34.8 ppm.

To a reaction tube charged with CuOTf (6.4 mg, 0.03 mmol, 10 mol%) and NFSI (141.8 mg, 0.45 mmol, 1.5 equiv) was added a solution of 2-methoxythiazole (**3a**, 34.5 mg, 0.3 mmol, 1 equiv) in anhydrous MeCN (2 mL, 0.15 M of **3a**) via a syringe under argon (1 atm) at 25 °C, and the

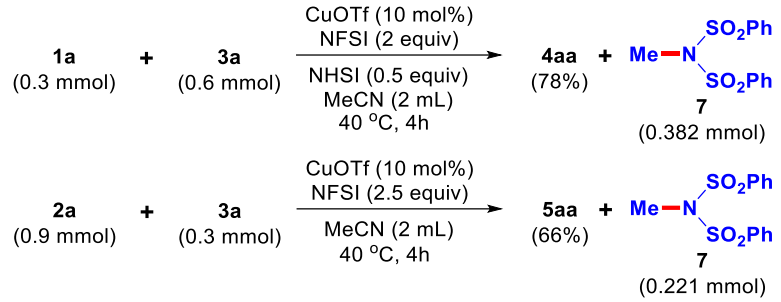
reaction mixture was stirred for 4 hours at 40 °C (oil bath temperature). TLC and LC-MS suggested that neither **6** nor **7** was generated. After quenched with Na_2CO_3 (aq.), the mixture was extracted with ethyl acetate. The combined organic phase was concentrated *in vacuo* to give dark residue, which was then purified by flash chromatography using petroleum ether and ethyl acetate as the eluent on silica gel to afford 14.9 mg of *C*5-imidated thiazole **3f** (12%) and recover 25.2 mg of **3a** (73%).

To another reaction tube charged with CuOTf (6.4 mg, 0.03 mmol, 10 mol%) and NFSI (141.8 mg, 0.45 mmol, 1.5 equiv) was added a solution of 2-methoxythiazole (**3a**, 34.5 mg, 0.3 mmol, 1 equiv) in anhydrous MeCN (2 mL, 0.15 M of **3a**) via a syringe under argon (1 atm) at 25 °C, and the reaction mixture was stirred for 4 hours at 60 °C (oil bath temperature). TLC and LC-MS suggested that neither **6** nor **7** was generated. After quenched with Na_2CO_3 (aq.), the mixture was extracted with ethyl acetate. The combined organic phase was concentrated *in vacuo* to give dark residue, which was then purified by flash chromatography using petroleum ether and ethyl acetate as the eluent on silica gel to afford 57.0 mg of *C*5-imidated thiazole **3f** (46%) and recover 14.6 mg of **3a** (42%).

***N*-(2-Methoxythiazol-5-yl)-*N*-(phenylsulfonyl)benzenesulfonamide (3f) [1a]:**

Colorless oil. **^{1}H NMR ($CDCl_3$, 400 MHz):** δ = 7.99-7.97 (m, 4H), 7.71-7.67 (m, 2H), 7.58-7.54 (m, 4H), 6.76 (s, 1H), 4.06 (s, 3H) ppm. **^{13}C NMR ($CDCl_3$, 100 MHz):** δ = 175.4, 141.1, 138.4, 134.5, 129.2, 128.6, 121.6, 57.9 ppm.

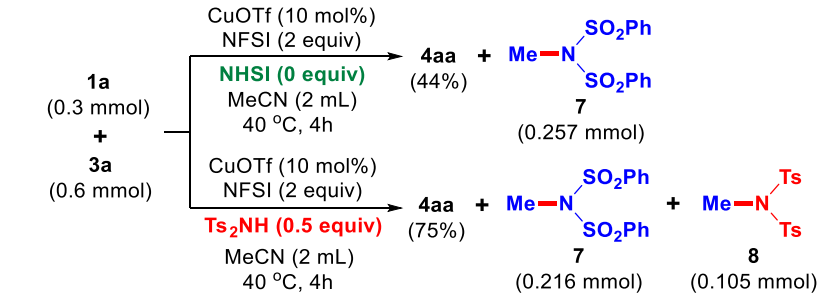
b) Exploration on the capture of the methyl carbocation under standard conditions



To a reaction tube charged with CuOTf (6.4 mg, 0.03 mmol, 10 mol%), NFSI (189.2 mg, 0.6 mmol, 2 equiv) and NHSI (44.6 mg, 0.15 mmol, 0.5 equiv) was added a solution of ethyl (4-butylphenyl)carbamate (**1a**, 66.4 mg, 0.3 mmol, 1 equiv) and 2-methoxythiazole (**3a**, 69.0 mg, 0.6 mmol, 2 equiv) in anhydrous MeCN (2 mL, 0.15 M of **1a**) via a syringe under argon (1 atm) at 25 °C, and the reaction mixture was stirred for 8 hours at 40 °C (oil bath temperature). After quenched with Na_2CO_3 (aq.), the mixture was extracted with ethyl acetate. The combined organic phase was concentrated *in vacuo* to give dark residue, which was then purified by flash chromatography using petroleum ether and ethyl acetate (8:1 to 6:1 to 4:1 to 3:1, *v*/*v*) as the eluent on silica gel to afford 74.5 mg of **4aa** (78%) and 118.8 mg of **7** (0.382 mmol).

To a reaction tube charged with CuOTf (6.4 mg, 0.03 mmol, 10 mol%) and NFSI (236.5 mg, 0.75 mmol, 2.5 equiv) was added a solution of diethyl 1,4-phenylenedicarbamate (**2a**, 226.9 mg, 0.9 mmol, 3 equiv) and 2-methoxythiazole (**3a**, 34.5 mg, 0.3 mmol, 1 equiv) in anhydrous MeCN (2 mL, 0.15 M of **3a**) via a syringe under22 argon (1 atm) at 25 °C, and the reaction mixture was stirred for 4 hours at 40 °C (oil bath temperature). After quenched with Na_2CO_3 (aq.), the mixture was extracted with ethyl acetate. The combined organic phase was concentrated *in vacuo* to give dark residue, which was then purified by flash chromatography using petroleum ether and ethyl acetate (8:1 to 6:1 to 4:1 to 3:1, *v*/*v*) as the eluent on silica gel to afford 69.8 mg of **5aa** (66%) and 68.7 mg of **7** (0.221 mmol).

c) Control experiments for the capture of the methyl carbocation



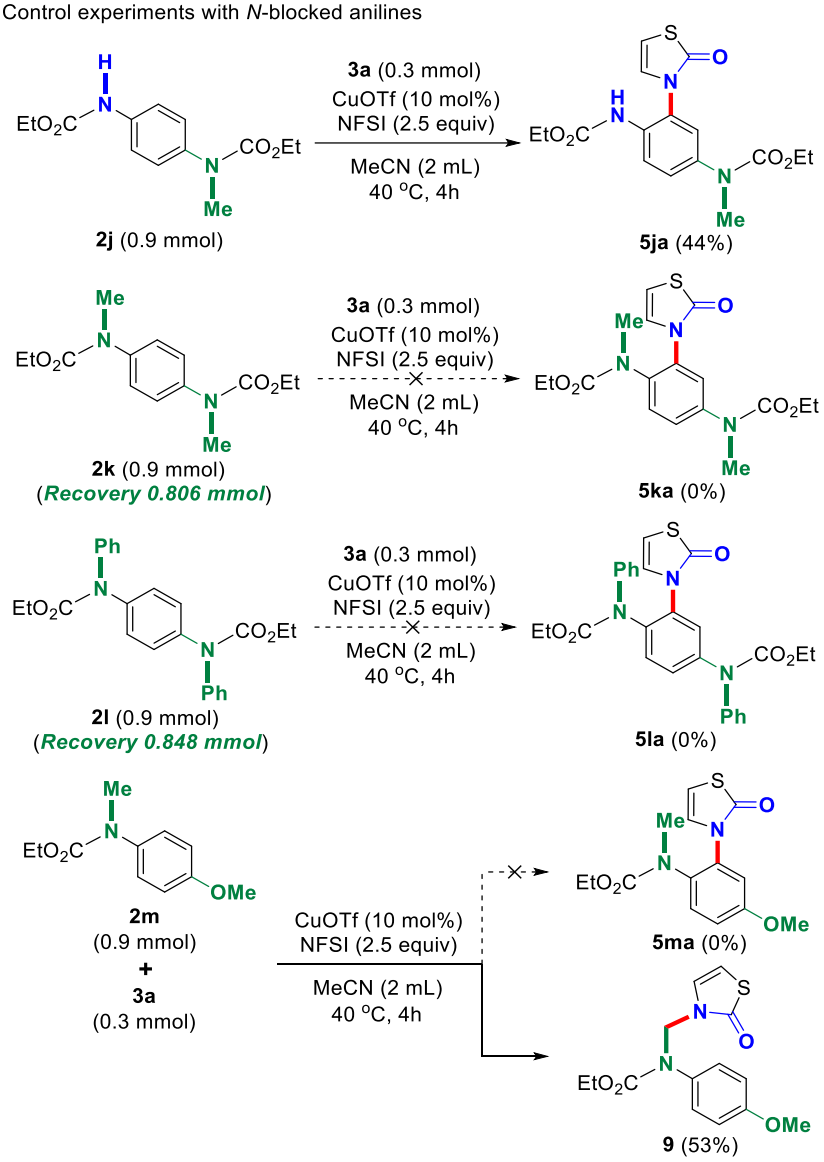
To a reaction tube charged with CuOTf (6.4 mg, 0.03 mmol, 10 mol%) and NFSI (189.2 mg, 0.6 mmol, 2 equiv) was added a solution of ethyl (4-butylphenyl)carbamate (**1a**, 66.4 mg, 0.3 mmol, 1 equiv) and 2-methoxythiazole (**3a**, 69.0 mg, 0.6 mmol, 2 equiv) in anhydrous MeCN (2 mL, 0.15 M of **1a**) via a syringe under argon (1 atm) at 25 °C, and the reaction mixture was stirred for 8 hours at 40 °C (oil bath temperature). After quenched with Na_2CO_3 (aq.), the mixture was extracted with ethyl acetate. The combined organic phase was concentrated *in vacuo* to give dark residue, which was then purified by flash chromatography using petroleum ether and ethyl acetate (8:1 to 6:1 to 4:1 to 3:1, *v*/*v*) as the eluent on silica gel to afford 42.4 mg of **4aa** (44%) and 79.9 mg of **7** (0.257 mmol).

To a reaction tube charged with CuOTf (6.4 mg, 0.03 mmol, 10 mol%), NFSI (189.2 mg, 0.6 mmol, 2 equiv) and Ts_2NH (48.8 mg, 0.15 mmol, 0.5 equiv) was added a solution of ethyl (4-butylphenyl)carbamate (**1a**, 66.4 mg, 0.3 mmol, 1 equiv) and 2-methoxythiazole (**3a**, 69.0 mg, 0.6 mmol, 2 equiv) in anhydrous MeCN (2 mL, 0.15 M of **1a**) via a syringe under argon (1 atm) at 25 °C, and the reaction mixture was stirred for 8 hours at 40 °C (oil bath temperature). After quenched with Na_2CO_3 (aq.), the mixture was extracted with ethyl acetate. The combined organic phase was concentrated *in vacuo* to give dark residue, which was then purified by flash chromatography using petroleum ether and ethyl acetate (8:1 to 6:1 to 4:1 to 3:1, *v*/*v*) as the eluent on silica gel to afford 72.1 mg of **4aa** (75%), along with 67.2 mg of **7** (0.216 mmol) and 35.6 mg of **8** (0.105 mmol).

***N*,4-dimethyl-*N*-tosylbenzenesulfonamide (Ts_2NMe, 8)** [4]**:**

Colorless oil. **^{1}H NMR ($CDCl_3$, 400 MHz):** δ = 7.89 (d, *J* = 8.2 Hz, 4H), 7.35 (d, *J* = 8.2 Hz, 4H), 3.26 (s, 3H), 2.46 (s, 6H) ppm. **^{13}C NMR ($CDCl_3$, 100 MHz):** δ = 145.0, 136.2, 129.7, 128.0, 34.7, 21.7 ppm.

d) Control experiments with *N*-blocked anilines



To a reaction tube charged with CuOTf (6.4 mg, 0.03 mmol, 10 mol%) and NFSI (236.5 mg, 0.75 mmol, 2.5 equiv) was added a solution of ethyl (4-((ethoxycarbonyl)amino)phenyl)(methyl)-carbamate (**2j**, 239.5, 0.9 mmol, 3 equiv) and 2-methoxythiazole (**3a**, 34.5 mg, 0.3 mmol, 1 equiv) in anhydrous MeCN (2 mL, 0.15 M of **3a**) via a syringe under argon (1 atm) at 25 °C, and the reaction mixture was stirred for 4 hours at 40 °C (oil bath temperature). After quenched with Na_2CO_3 (aq.), the mixture was extracted with ethyl acetate. The combined organic phase was concentrated *in vacuo* to give dark residue, which was then purified by flash chromatography using petroleum ether and ethyl acetate as the eluent on silica gel to afford 48.5 mg of **5ja** (44%).

Ethyl (4-((ethoxycarbonyl)amino)-3-(2-oxothiazol-3(2*H*)-yl)phenyl)(methyl)carbamate (5ja): Yellow solid, m.p. 120-125 °C. **^{1}H NMR ($CDCl_3$, 400 MHz):** δ = 7.85 (d, *J* = 8.8 Hz, 1H), 7.29

(dd, *J* = 8.9 Hz, 2.3 Hz, 1H), 7.20 (brs, 2H), 6.73 (d, *J* = 5.4 Hz, 1H), 6.35 (d, *J* = 5.4 Hz, 1H), 4.22-4.15 (m, 4H), 3.29 (s, 3H), 1.31-1.24 (m, 6H). **^{13}C NMR ($CDCl_3$, 100 MHz):** δ = 171.6, 155.5, 154.3, 140.2, 131.1, 129.0, 128.6, 125.9, 125.1, 123.6, 103.7, 62.1, 61.7, 37.4, 14.7, 14.6 ppm. **HRMS (ESI)** *m/z* calcd for $C_{16}H_{20}N_3O_5S$ $[M+H]^+$: 366.1118, found 366.1114.

To a reaction tube charged with CuOTf (6.4 mg, 0.03 mmol, 10 mol%) and NFSI (236.5 mg, 0.75 mmol, 2.5 equiv) was added a solution of diethyl 1,4-phenylenebis(methylcarbamate) (**2k**, 252.1 mg, 0.9 mmol, 3 equiv) and 2-methoxythiazole (**3a**, 34.5 mg, 0.3 mmol, 1 equiv) in anhydrous MeCN (2 mL, 0.15 M of **3a**) via a syringe under argon (1 atm) at 25 °C, and the reaction mixture was stirred for 4 hours at 40 °C (oil bath temperature). TLC indicated poor chemo-selectivity as a variety of trace products could be observed. After quenched with Na_2CO_3 (aq.), the mixture was extracted with ethyl acetate. The combined organic phase was concentrated *in vacuo* to give dark residue, which was then purified by flash chromatography using petroleum ether and ethyl acetate as the eluent on silica gel to recover 225.8 mg of **2k** (0.806 mmol).

Diethyl 1,4-phenylenebis(methylcarbamate) (2k):

Off-white solid, m.p. 75-77 °C. **^{1}H NMR (DMSO-d_6, 400 MHz):** δ = 7.28 (s, 4H), 4.08 (q, *J* = 7.1 Hz, 4H), 2.21 (s, 6H), 1.17 (t, *J* = 7.1 Hz, 6H). **^{13}C NMR (DMSO-d_6, 100 MHz):** δ = 154.7, 140.4, 125.6, 61.1, 37.2, 14.4 ppm. **HRMS (ESI)** *m/z* calcd for $C_{14}H_{21}N_2O_4$ $[M+H]^+$: 281.1496, found 281.1493.

To a reaction tube charged with CuOTf (6.4 mg, 0.03 mmol, 10 mol%) and NFSI (236.5 mg, 0.75 mmol, 2.5 equiv) was added a solution of diethyl 1,4-phenylenebis(phenylcarbamate) (**2l**, 363.8 mg, 0.9 mmol, 3 equiv) and 2-methoxythiazole (**3a**, 34.5 mg, 0.3 mmol, 1 equiv) in anhydrous MeCN (2 mL, 0.15 M of **3a**) via a syringe under argon (1 atm) at 25 °C, and the reaction mixture was stirred for 4 hours at 40 °C (oil bath temperature). TLC and LC-MS suggested that no *N*-phenyl thiazol-2(3*H*)-one **5la** was generated. After quenched with Na_2CO_3 (aq.), the mixture was extracted with ethyl acetate. The combined organic phase was concentrated *in vacuo* to give dark residue, which was then purified by flash chromatography using petroleum ether and ethyl acetate as the eluent on silica gel to recover 342.7 mg of **2l** (0.848 mmol).

Diethyl 1,4-phenylenebis(phenylcarbamate) (2l):

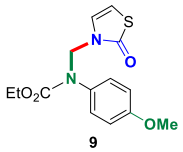
Light yellow solid, m.p. 130-132 °C. **^{1}H NMR ($CDCl_3$, 400 MHz):** δ = 7.35-7.31 (m, 4H), 7.23-7.16 (m, 10H), 4.21 (q, *J* = 7.1 Hz, 4H), 1.22 (t, *J* = 7.1 Hz, 6H). **^{13}C NMR ($CDCl_3$, 100 MHz):** δ = 154.7, 142.2, 140.0, 128.9, 127.2, 126.7, 126.2, 62.1, 14.4 ppm. **HRMS (ESI)** *m/z* calcd for $C_{24}H_{24}KN_2O_4$ $[M+K]^+$: 443.1368, found 443.1363.

To a reaction tube charged with CuOTf (6.4 mg, 0.03 mmol, 10 mol%) and NFSI (236.5 mg, 0.75 mmol, 2.5 equiv) was added a solution of ethyl (4-methoxyphenyl)(methyl)carbamate (**2m**, 188.2, 0.9 mmol, 3 equiv) and 2-methoxythiazole (**3a**, 34.5 mg, 0.3 mmol, 1 equiv) in anhydrous MeCN (2 mL, 0.15 M of **3a**) via a syringe under argon (1 atm) at 25 °C, and the reaction mixture was stirred for 4 hours at 40 °C (oil bath temperature). After quenched with Na_2CO_3 (aq.), the mixture was extracted with ethyl acetate. The combined organic phase was concentrated *in vacuo* to give dark residue, which was then purified by flash chromatography using petroleum ether and ethyl acetate as the eluent on silica gel to afford 49.2 mg of **9** (53%) and recovery 97.9 mg of **2m** (0.468 mmol) while no **5ma** was generated.

Ethyl (4-methoxyphenyl)(methyl)carbamate (2m):

Light brown oil. **^{1}H NMR ($CDCl_3$, 400 MHz):** δ = 7.18 (d, *J* = 8.9 Hz, 2H), 6.91 (d, *J* = 8.9 Hz, 2H), 4.03 (q, *J* = 7.1 Hz, 2H), 3.75 (s, 3H), 3.16 (s, 3H), 1.13 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR ($CDCl_3$, 100 MHz):** δ = 157.1, 154.9, 136.1, 127.0, 113.9, 60.8, 55.2, 37.6, 14.4 ppm. **HRMS (ESI)** *m/z* calcd for $C_{11}H_{16}NO_3$ $[M+K]^+$: 210.1125, found 210.1125.

Ethyl (4-methoxyphenyl)((2-oxothiazol-3(2*H*)-yl)methyl)carbamate (9):



9

Light brown oil. **^{1}H NMR (DMSO-d_6, 400 MHz):** δ = 7.06 (d, *J* = 8.9 Hz, 2H), 6.90 (d, *J* = 8.9 Hz, 2H), 6.89 (d, *J* = 5.8 Hz, 1H), 6.44 (d, *J* = 5.5 Hz, 1H), 5.41 (s, 2H), 4.09 (q, *J* = 7.2 Hz, 2H), 3.75 (s, 3H), 1.12 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR (DMSO-d_6, 100 MHz):** δ = 171.0, 158.1, 132.5, 128.6, 128.3, 124.8, 114.2, 113.9, 101.5, 61.7, 58.0, 55.2, 14.2 ppm. **HRMS (ESI)** *m/z* calcd for $C_{14}H_{17}N_2O_4S$ $[M+H]^+$: 309.0904, found 309.0899.

5. References

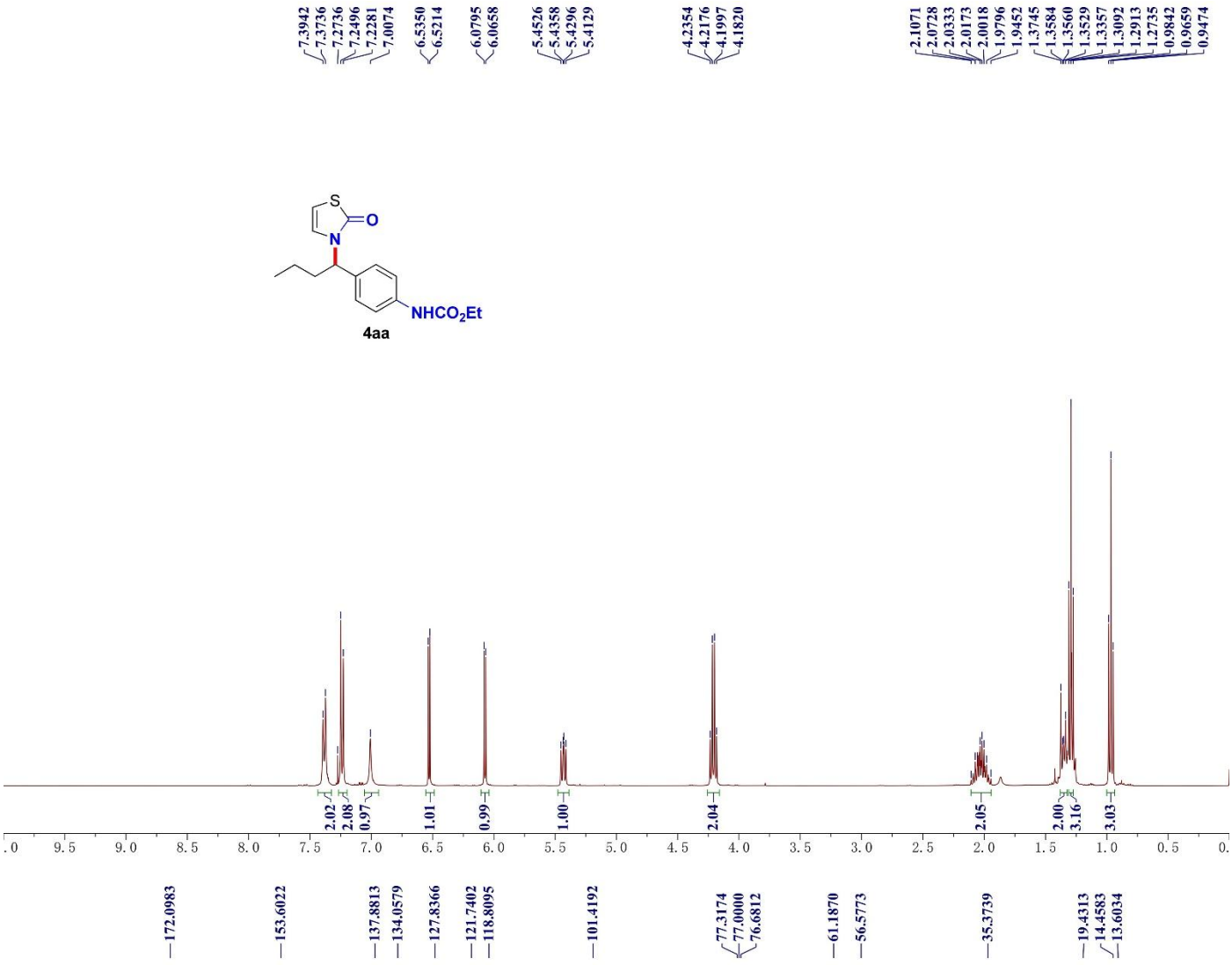
[1] a) D. Zhuang, L. Cheng, Y. Yan, Y. Tang, Z. Wan, J. Gu, Y. Lu, X. Li, Z. Li, *Org. Chem. Front.* **2024**, *11*, 4138–4148 ; b) F. Wang, J. Chen, X. Jia, D. Zhuang, Z. Wan, L. Ma, Z. Li, *J. Org. Chem.* **2022**, *87*, 10698−10709; c) J. Chen, F. Wang, Y. Huang, X. Jia, D. Zhuang, Z. Wan, Z. Li, *Org. Chem. Front.* **2022**, *9*, 3169−3178.

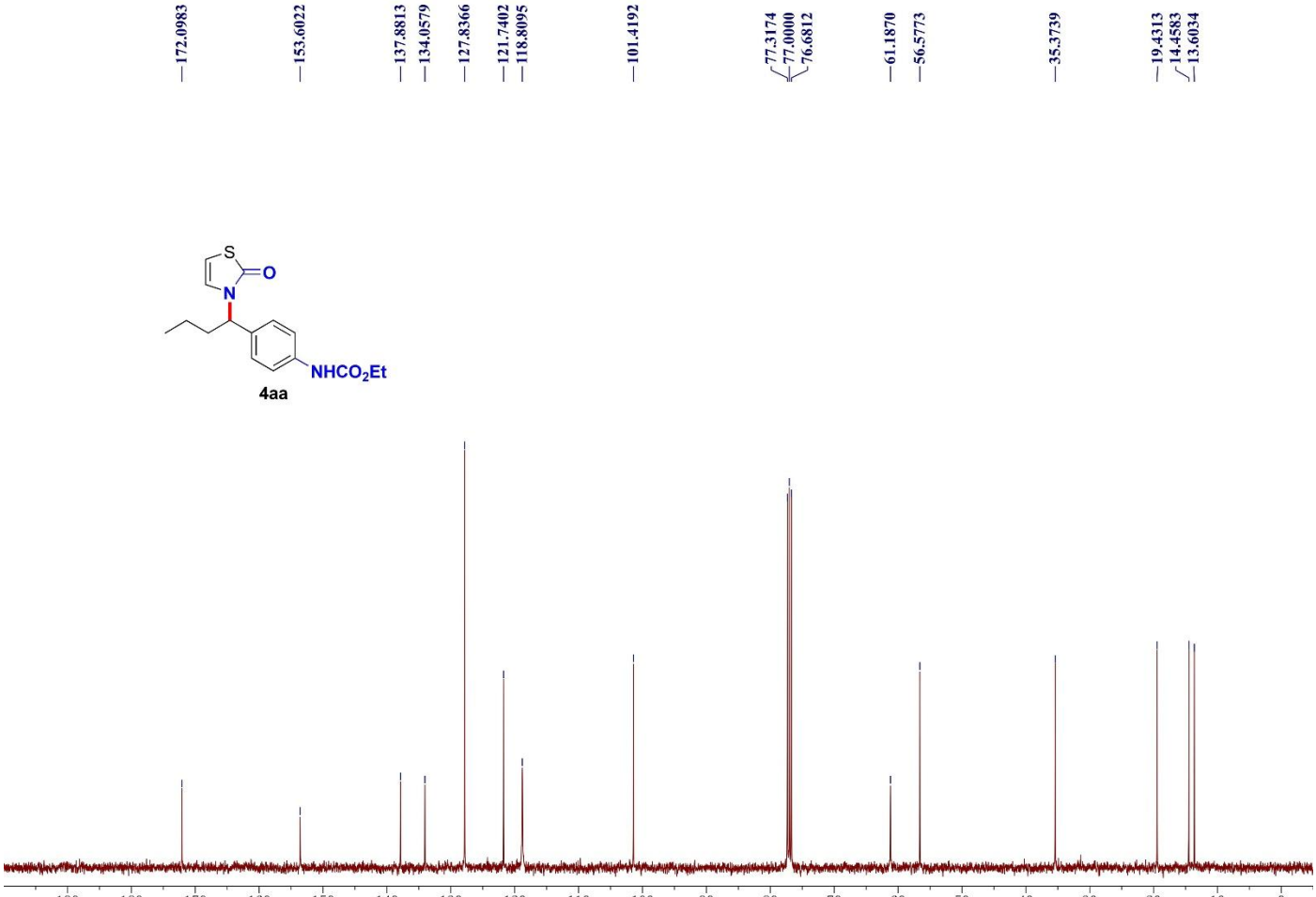
[2] a) B. A. F. Le Bailly, M. D. Greenhalgh, S. P. Thomas, *Chem. Commun.* **2012**, *48*, 1580–1582; b) J. Zhu, Y. Wang, J. Yao, H. Li, *Chem. Sci.* **2024**, *15*, 20534–20544; c) A. Chilin, G. Marzaro, S. Zanatta, V. Barbieri, G. Pastorini, P. Manzini, A. Guiotto, *Tetrahedron* **2006**, *62*, 12351–12356.

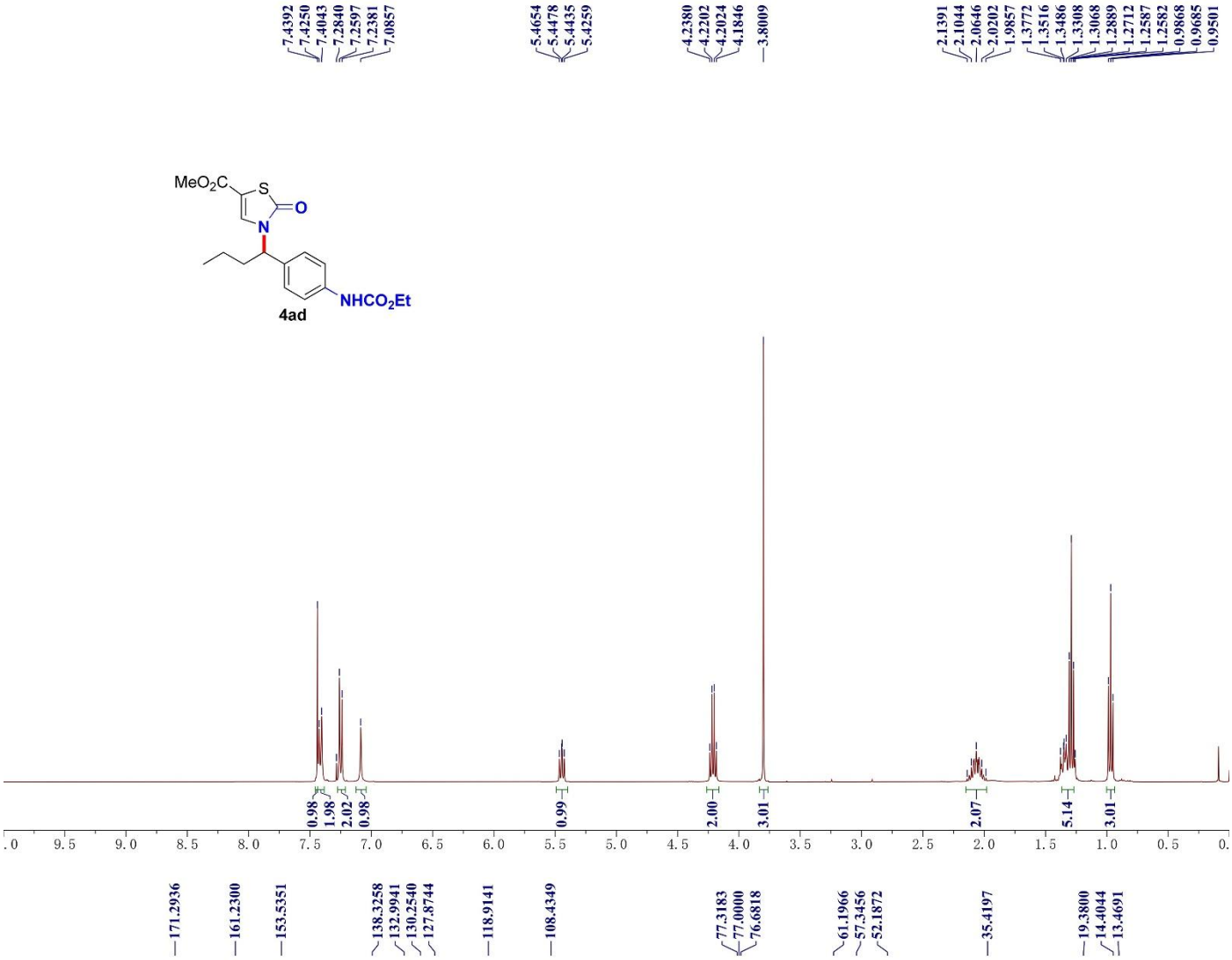
[3] (a) S. R. Dandepally, R. Elgoummadi and A. L. Williams, *Tetrahedron Lett.,* 2013, **54**, 925–928; (b) G. S. Kumar, S. P. Ragini, A. S. Kumar and H. M. Meshram, *RSC Adv.,* 2015, **5**, 51576–51580.

[4] L. Chen, H. Lang, L. Fang, J. Yu and L. Wang, *Eur. J. Org. Chem.*, **2014**, 6385-6389.

6. NMR Spectra of Title Compounds



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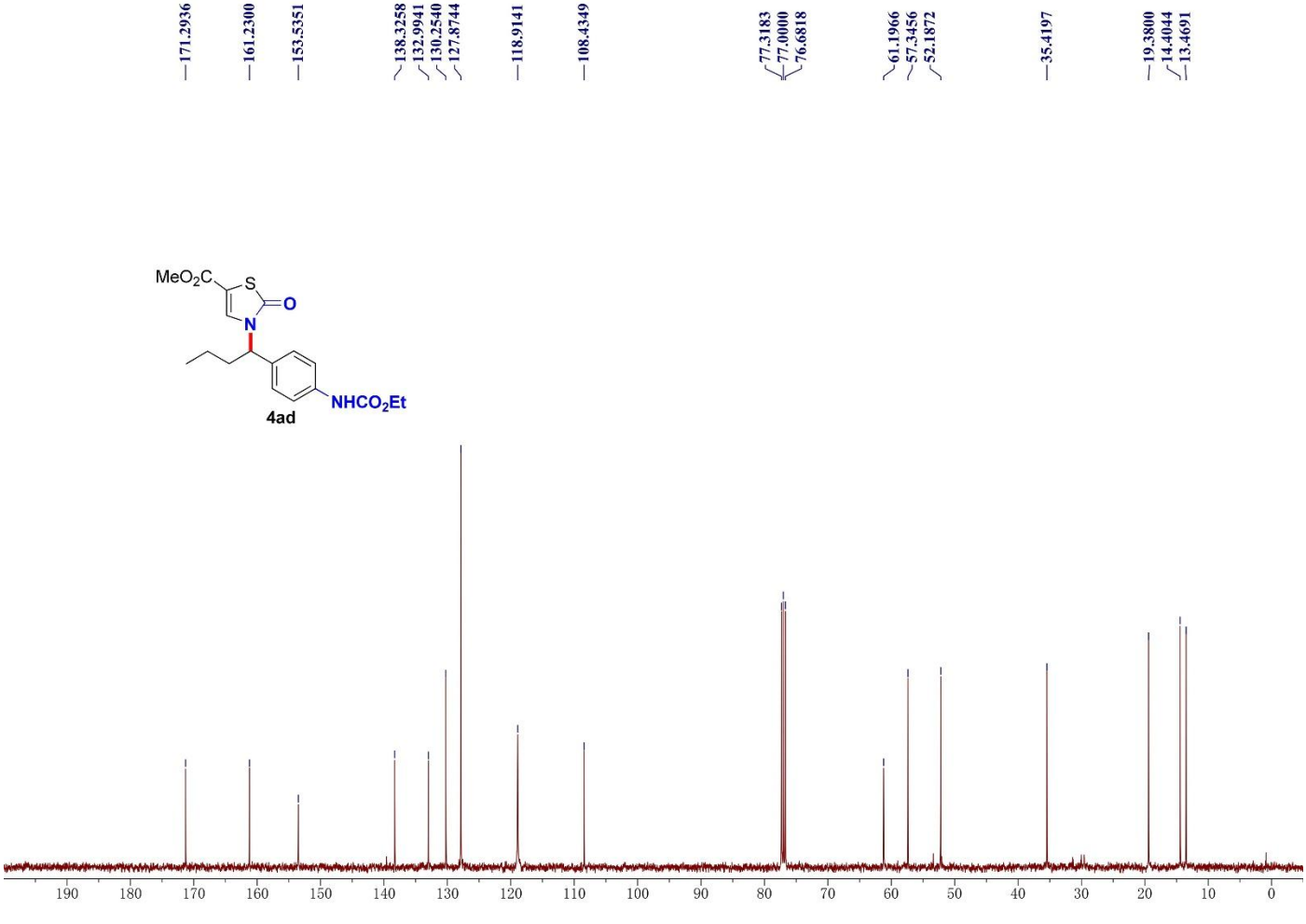
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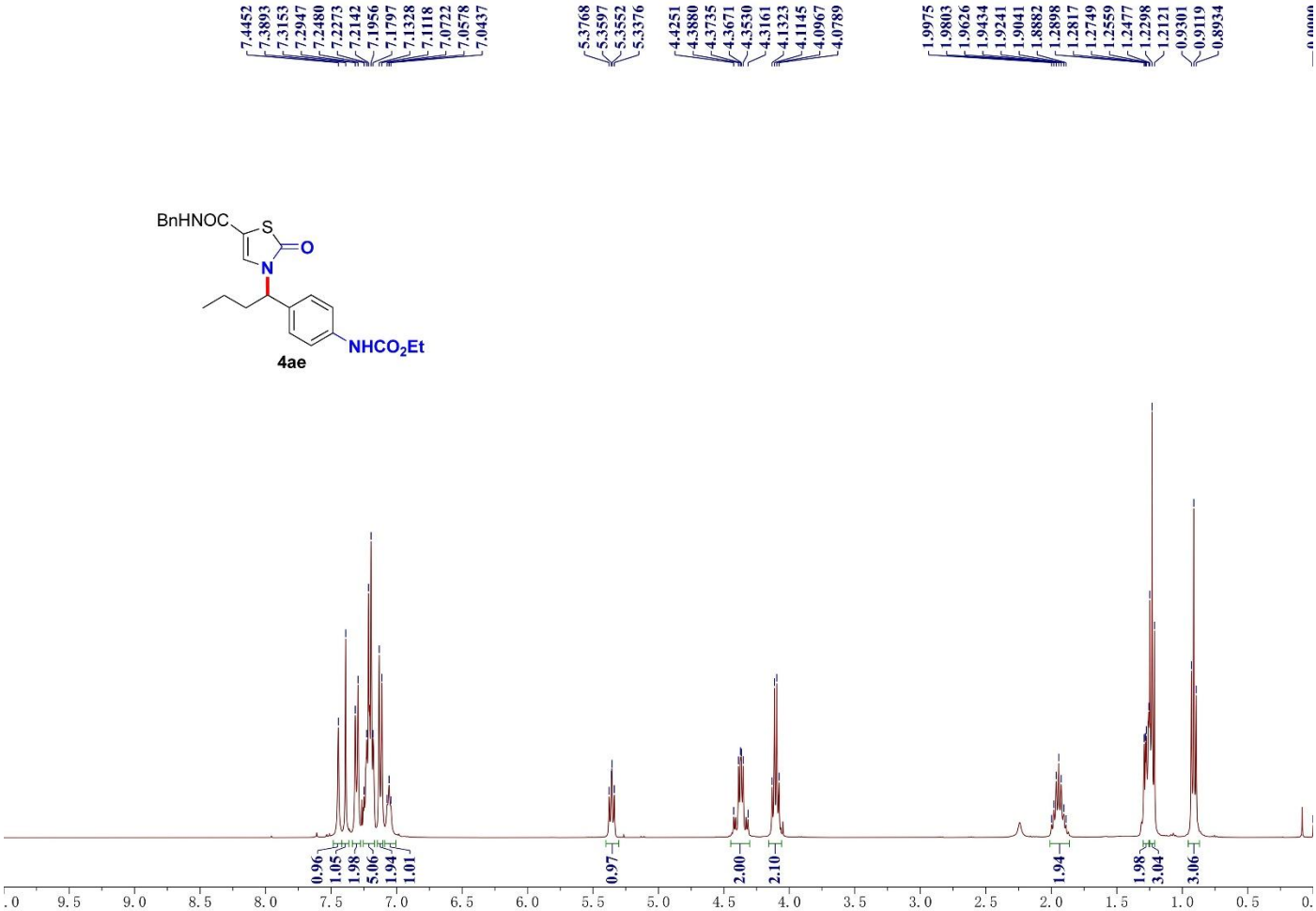
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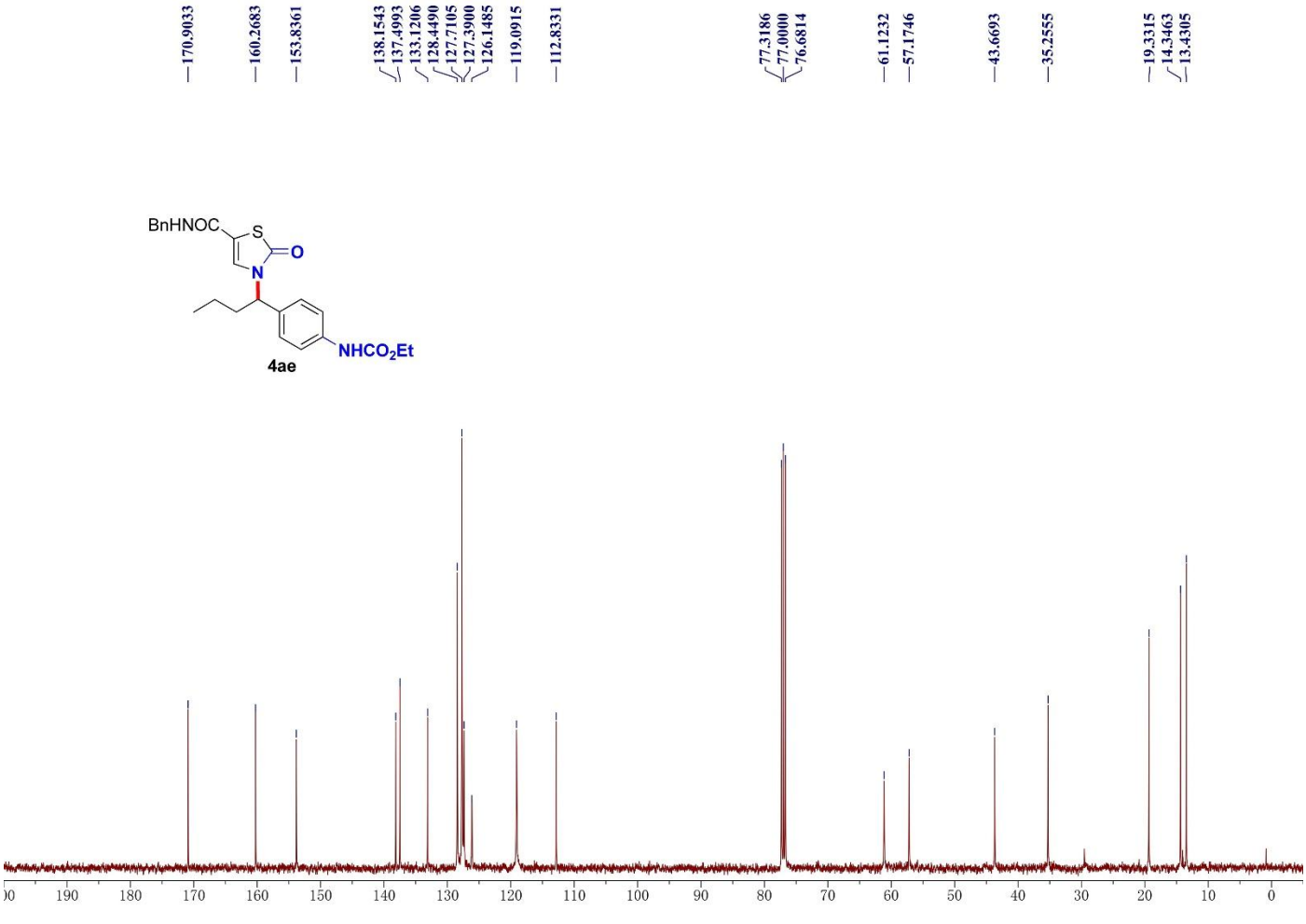
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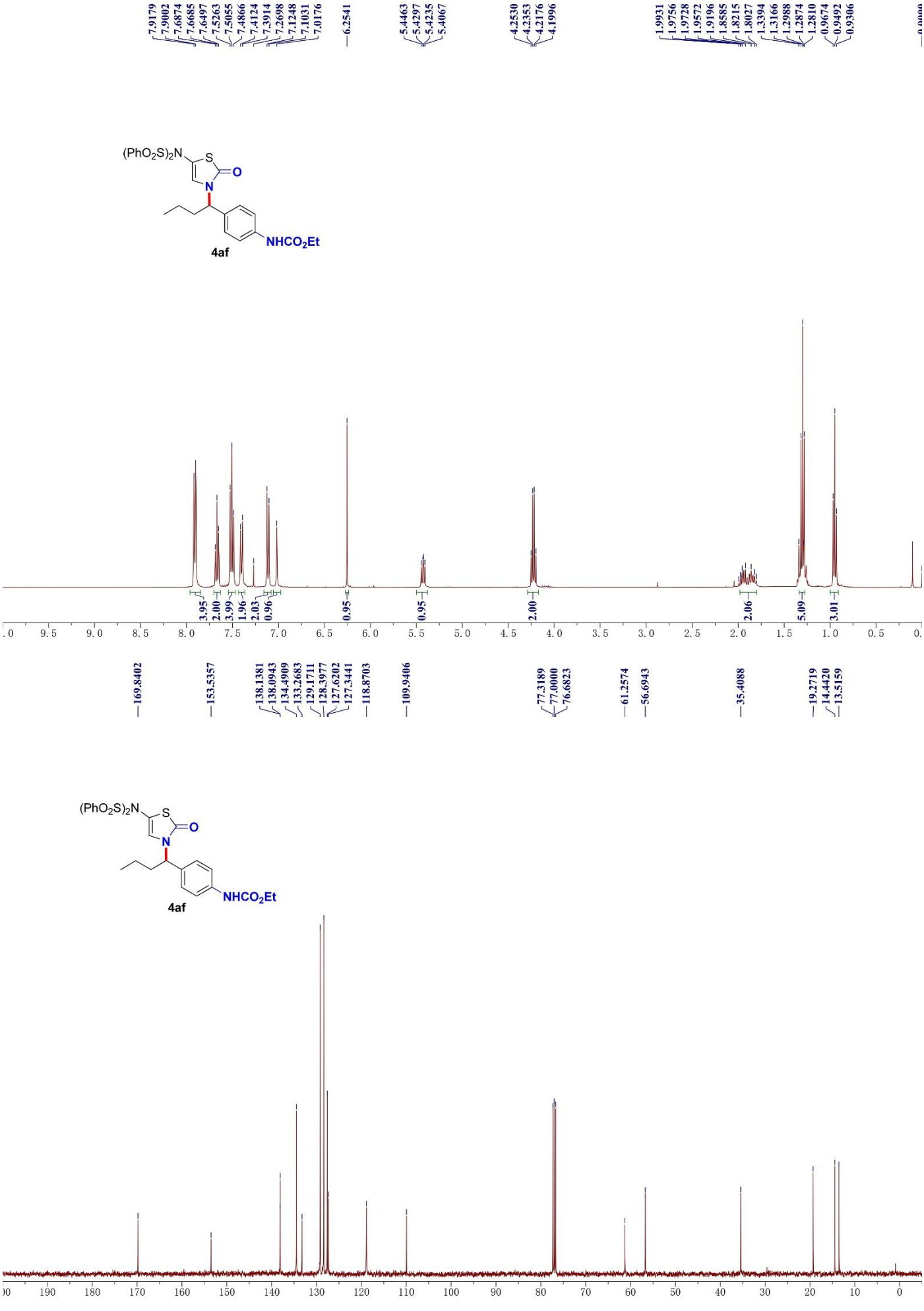
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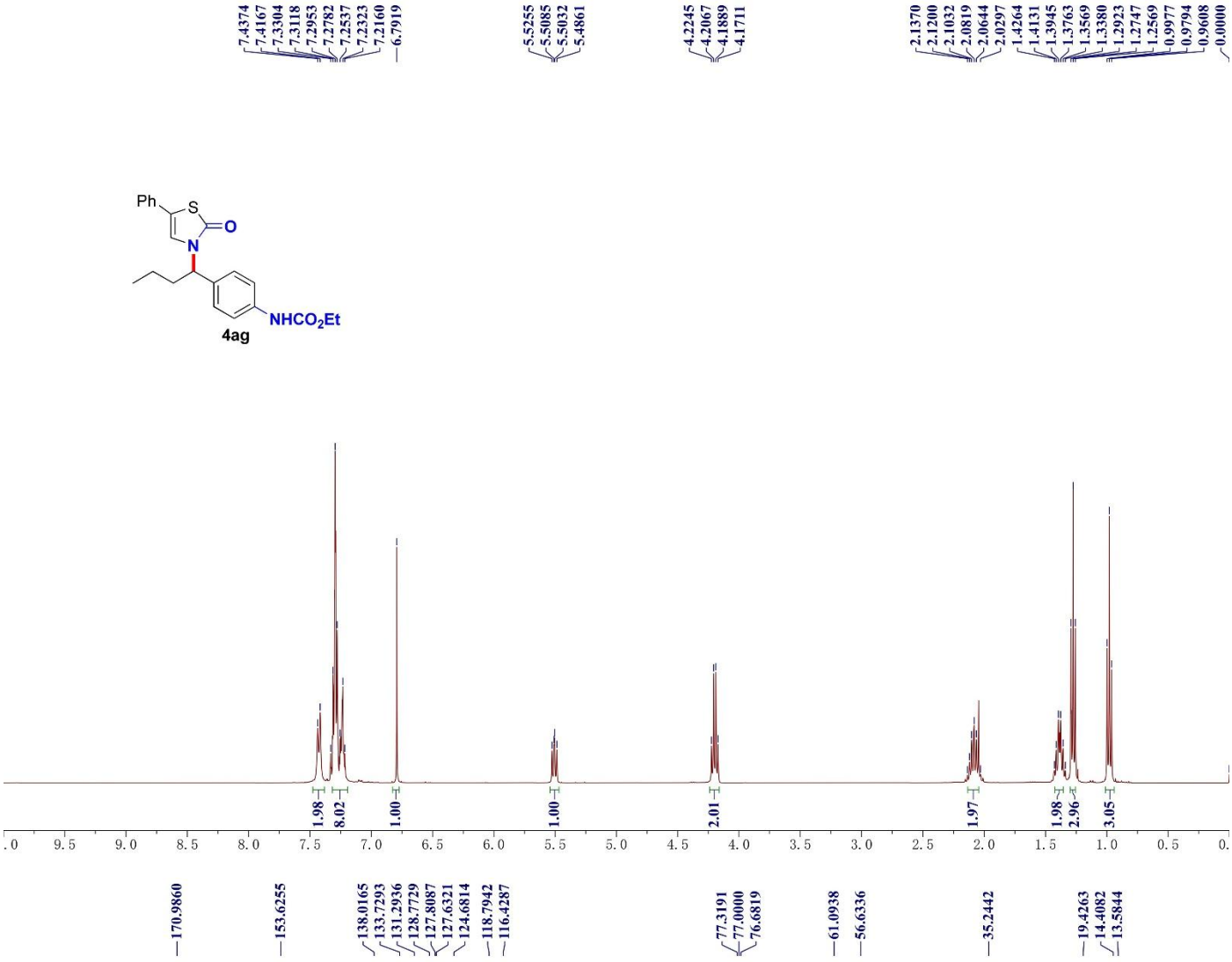
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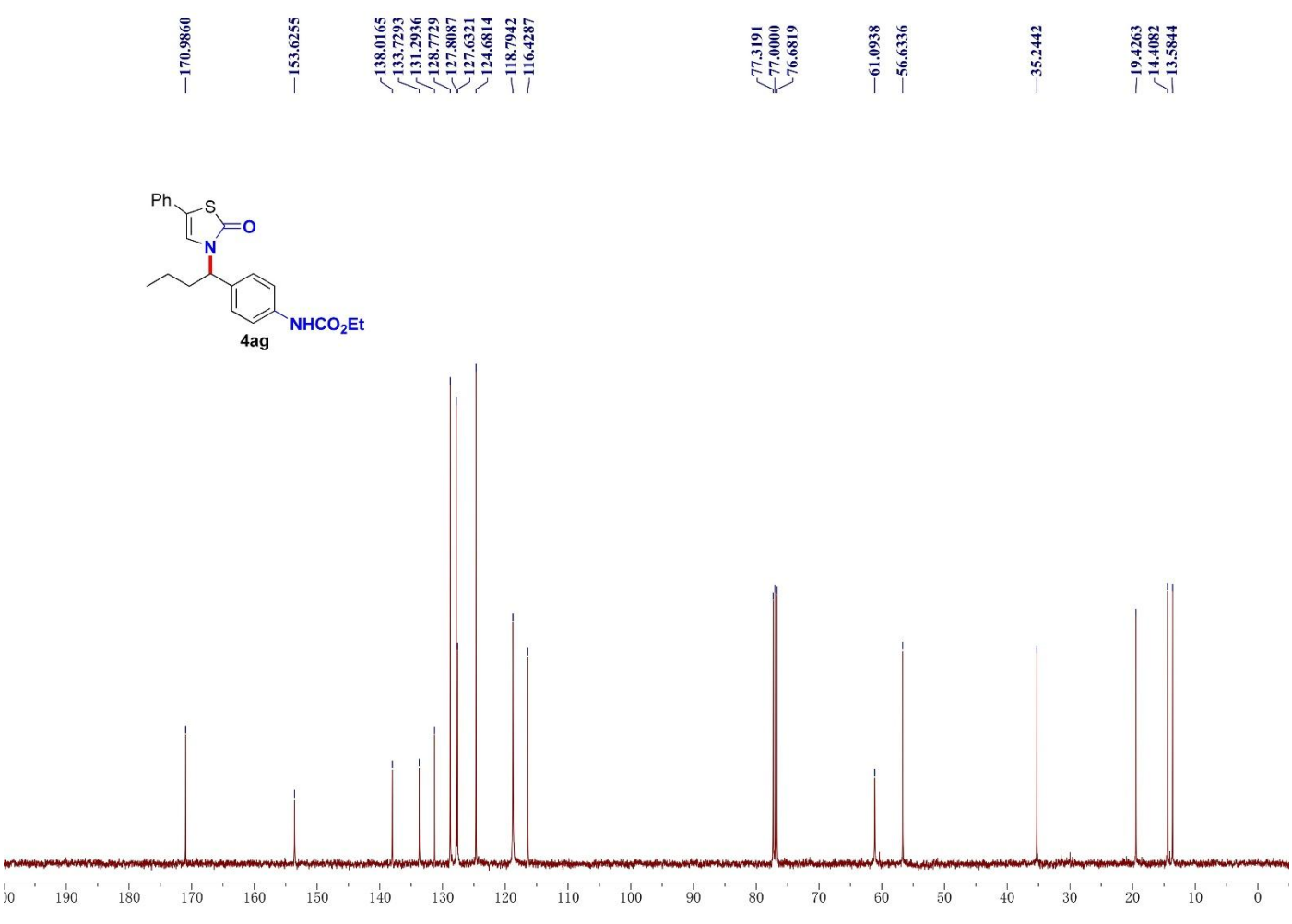
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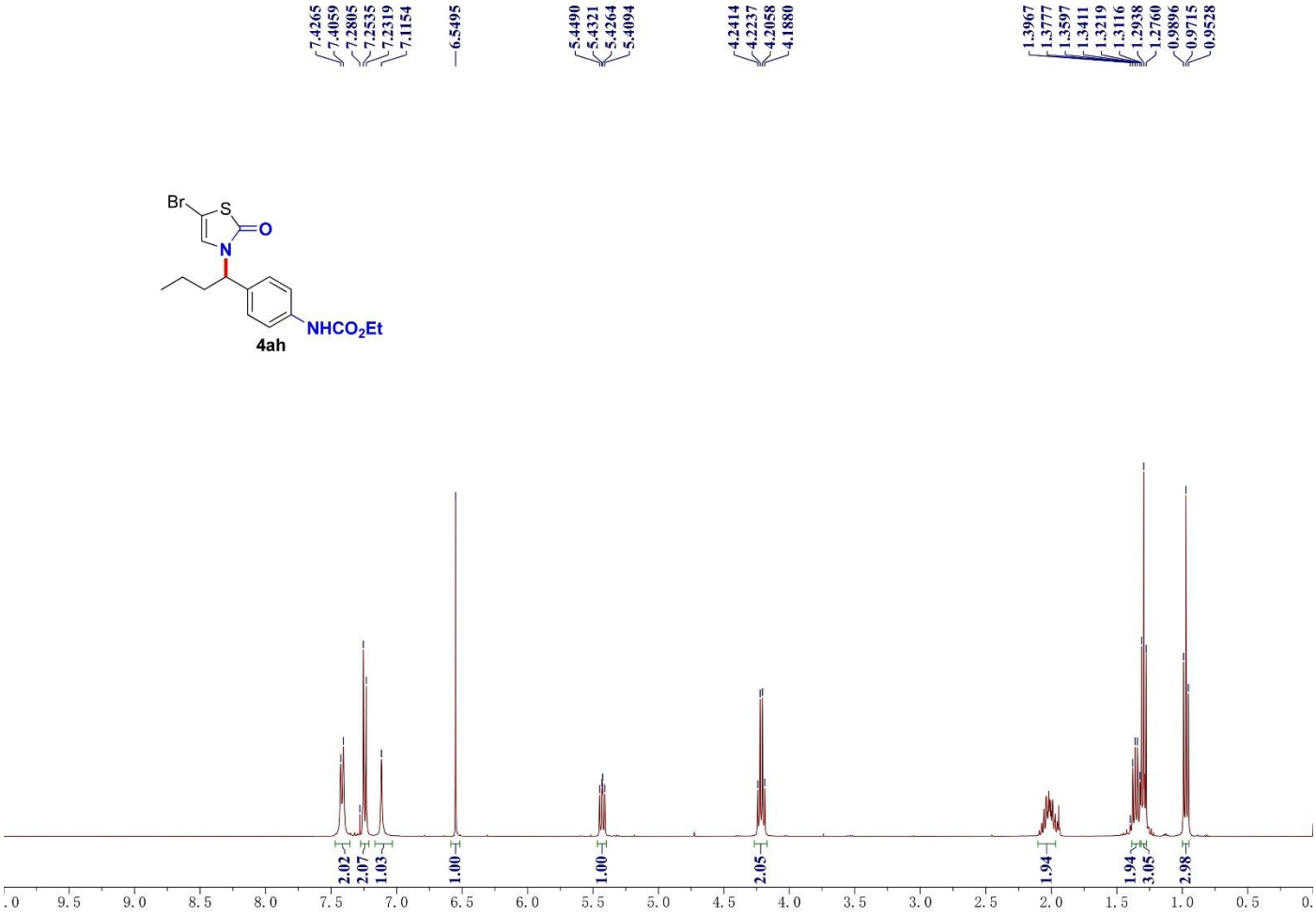
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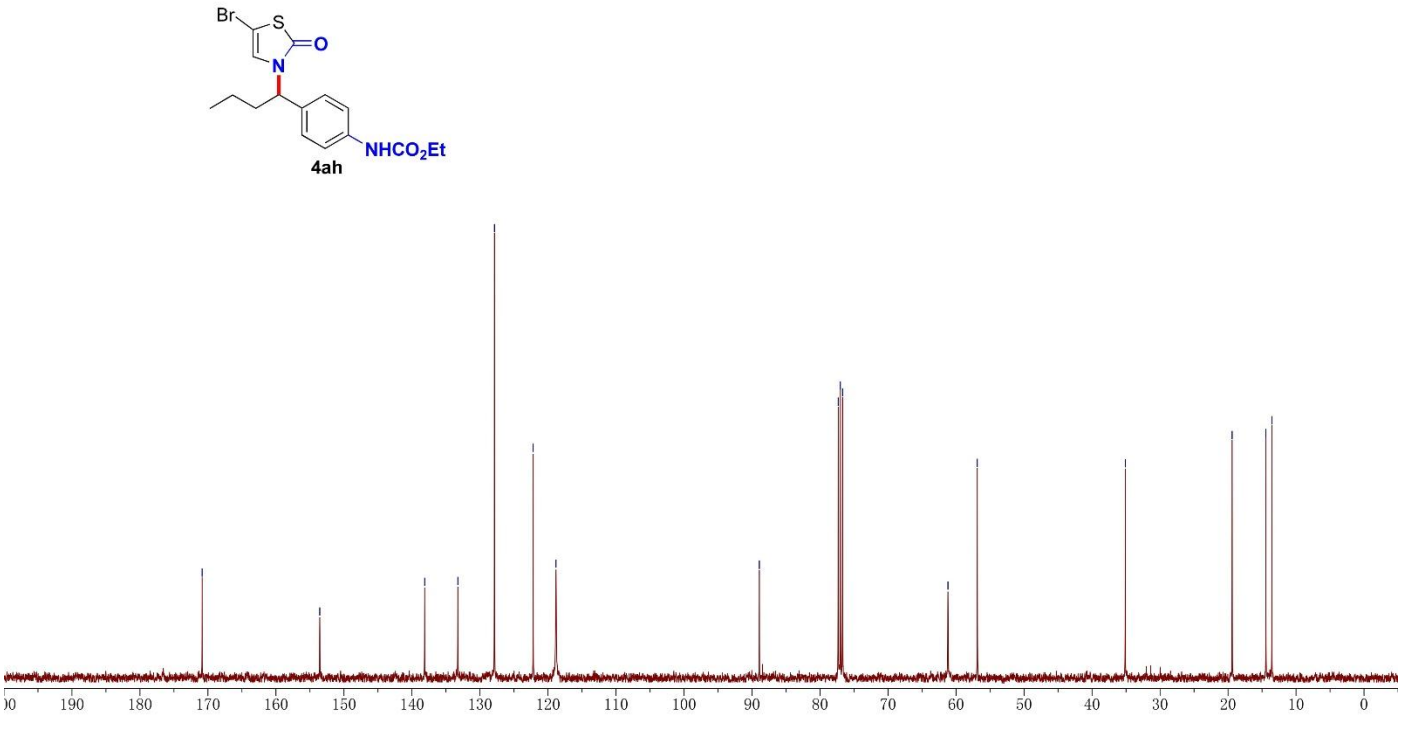
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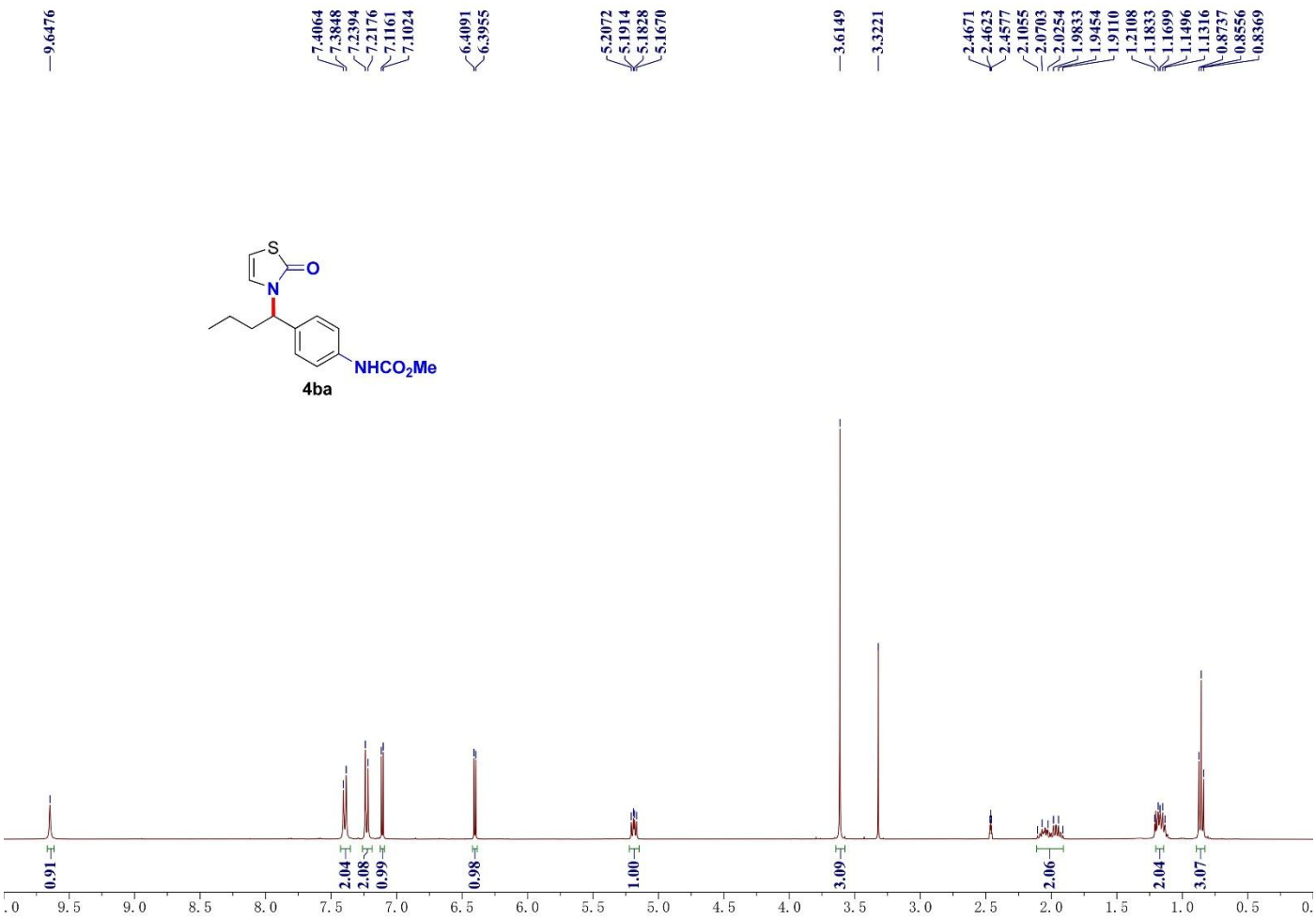
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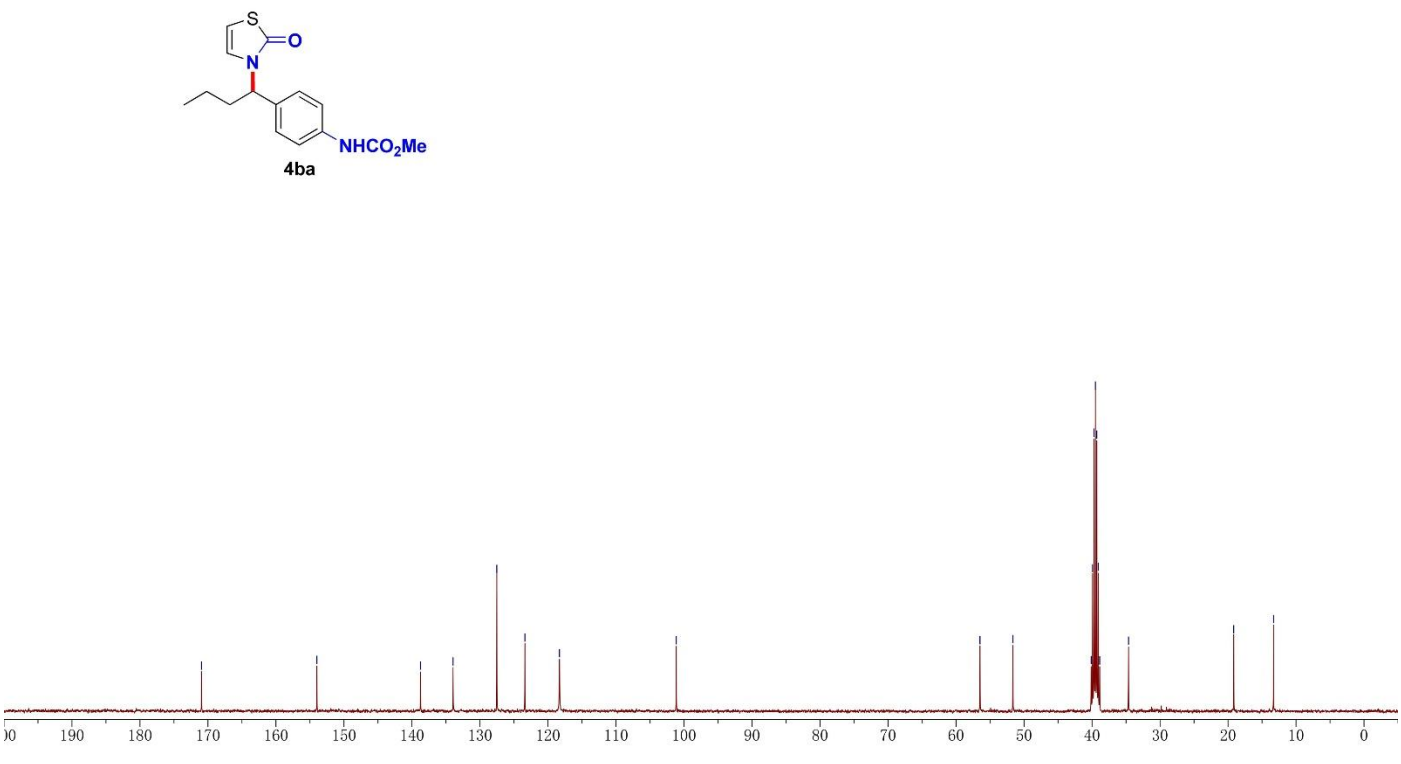
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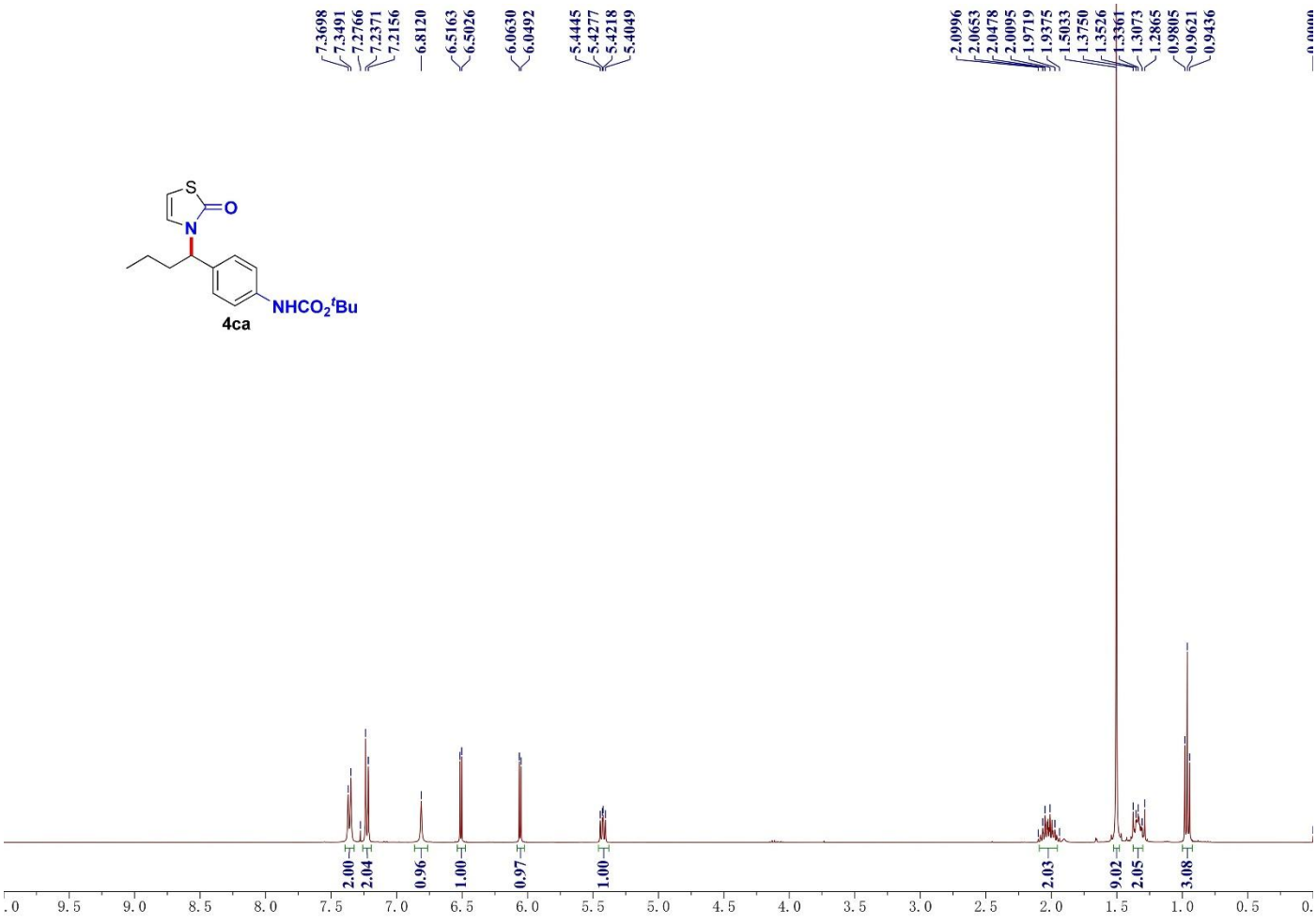
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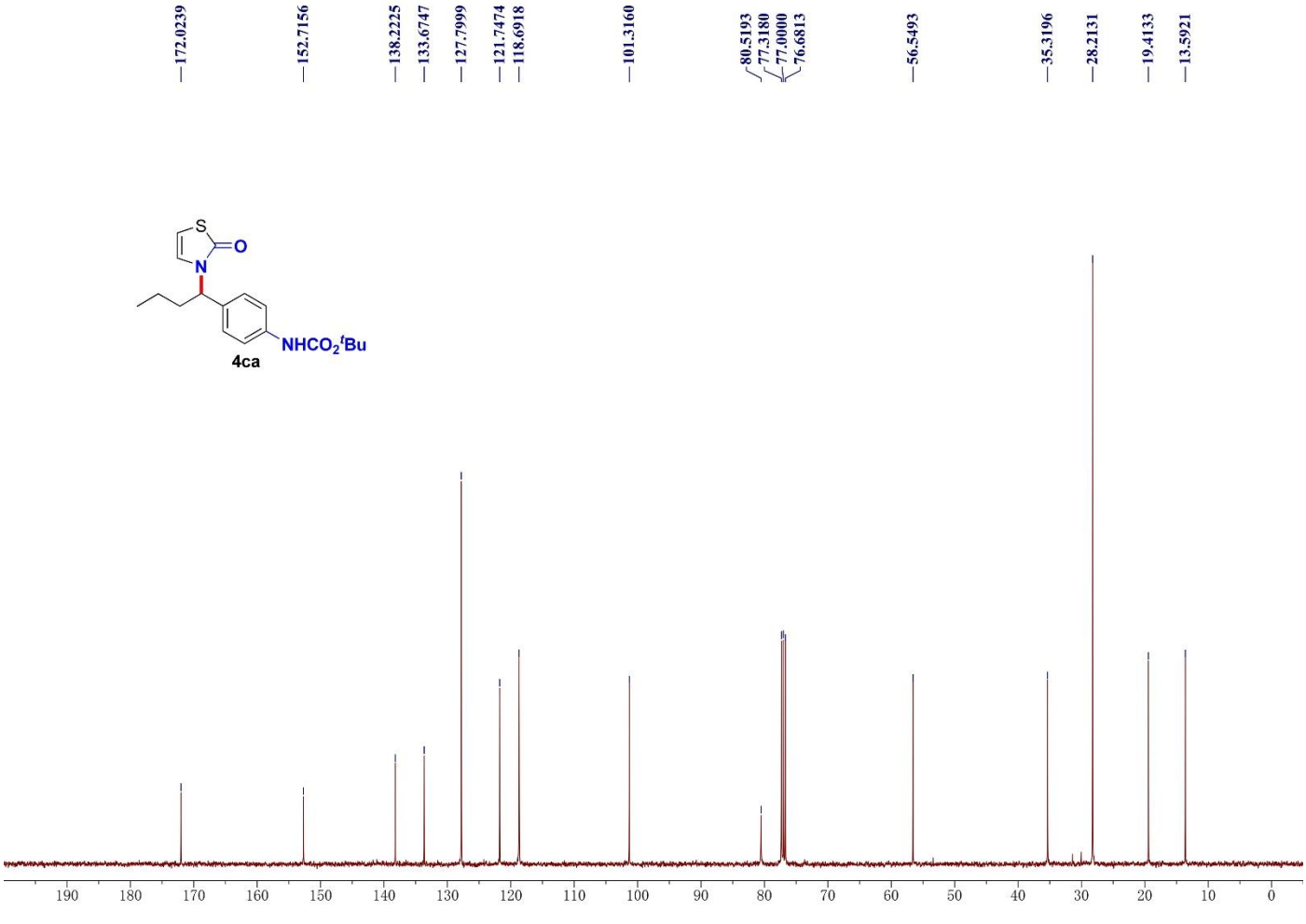
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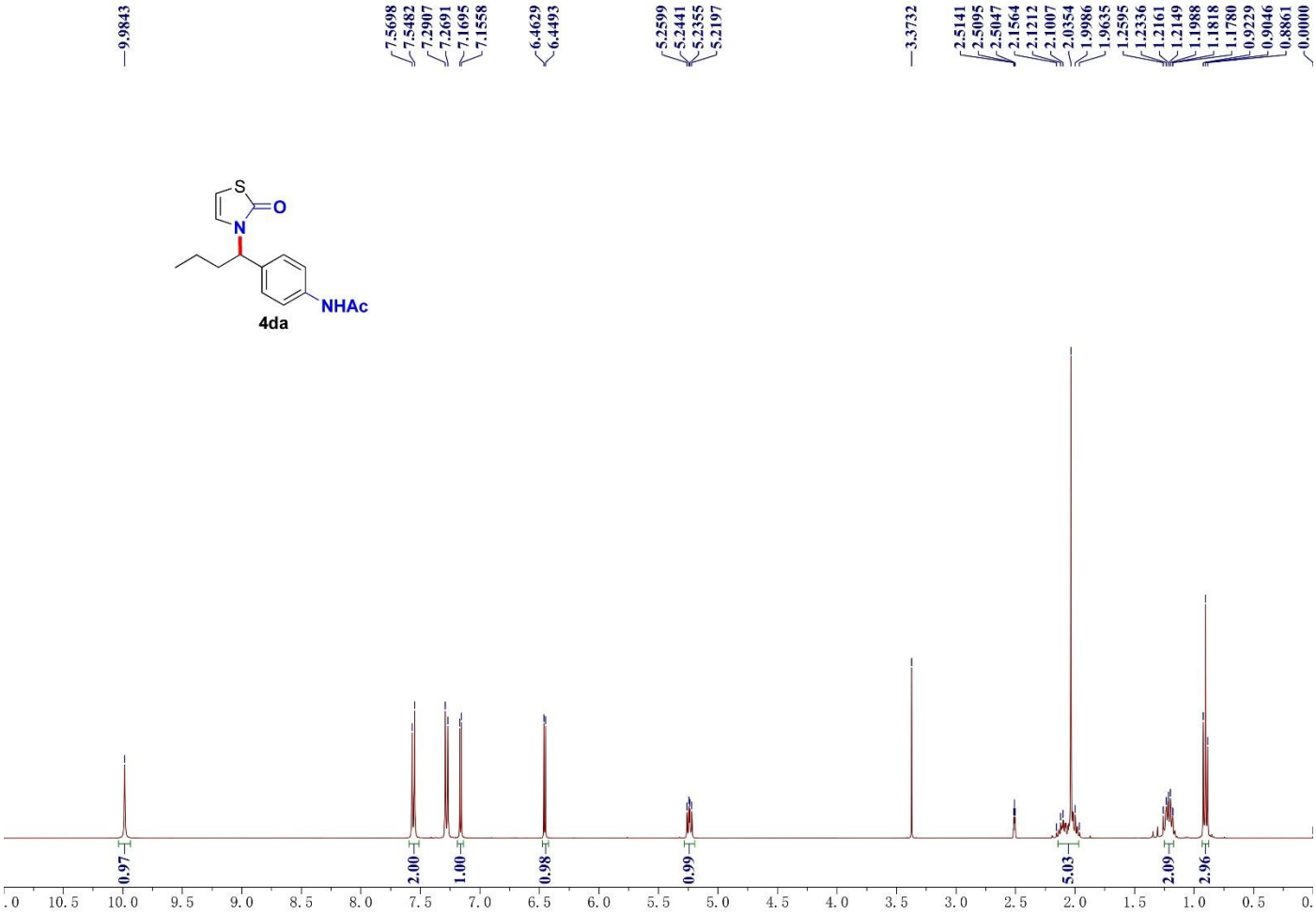
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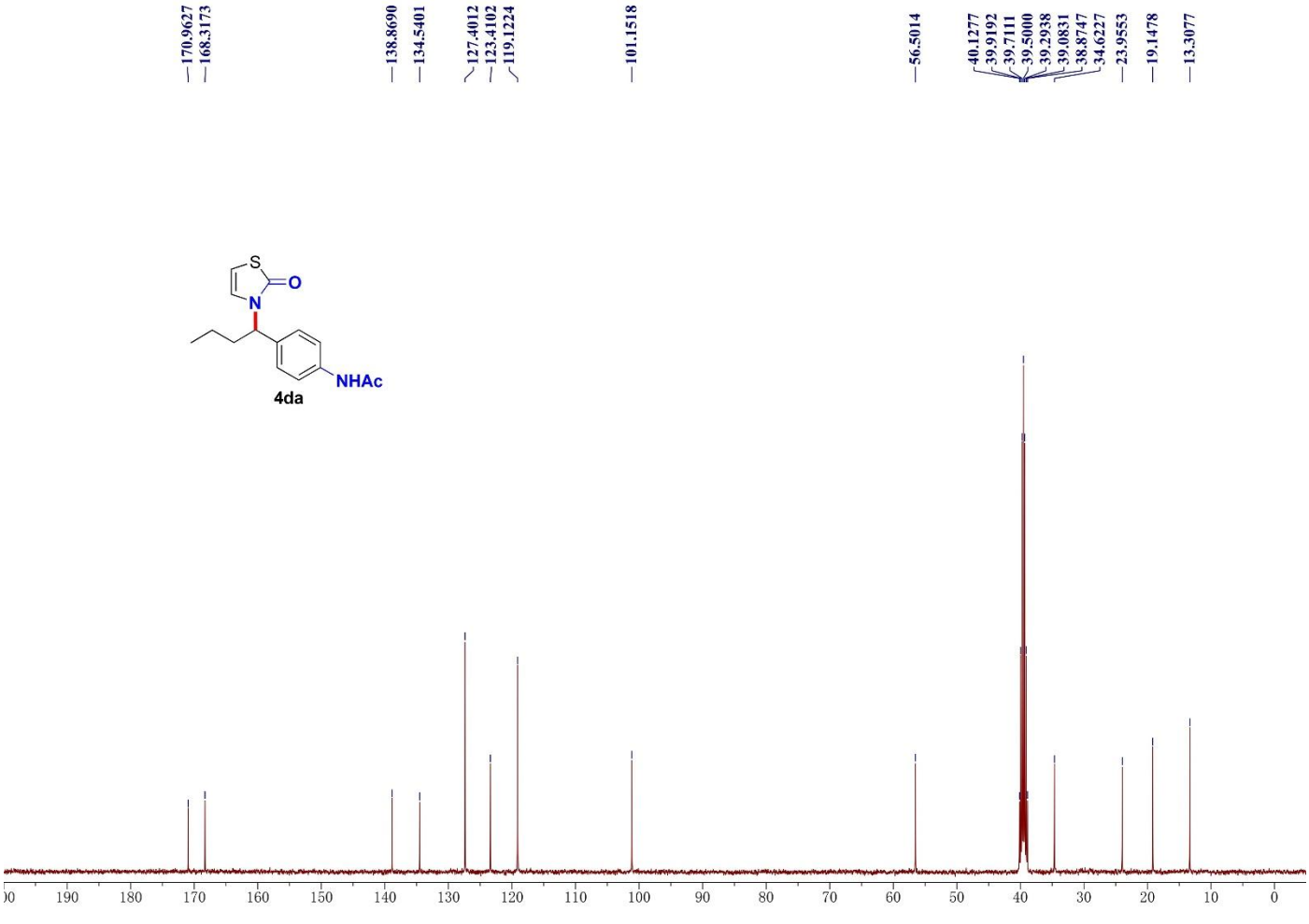
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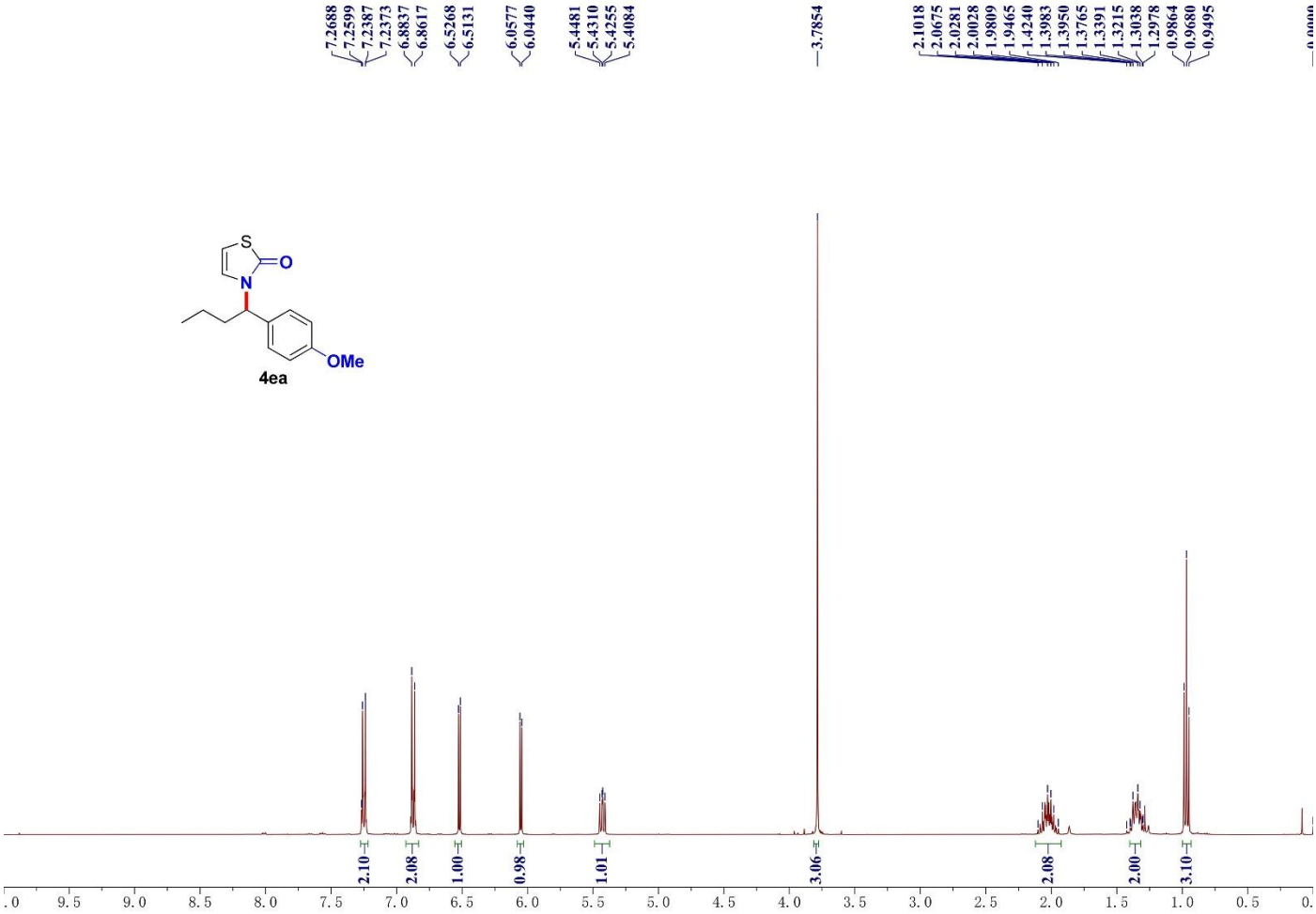
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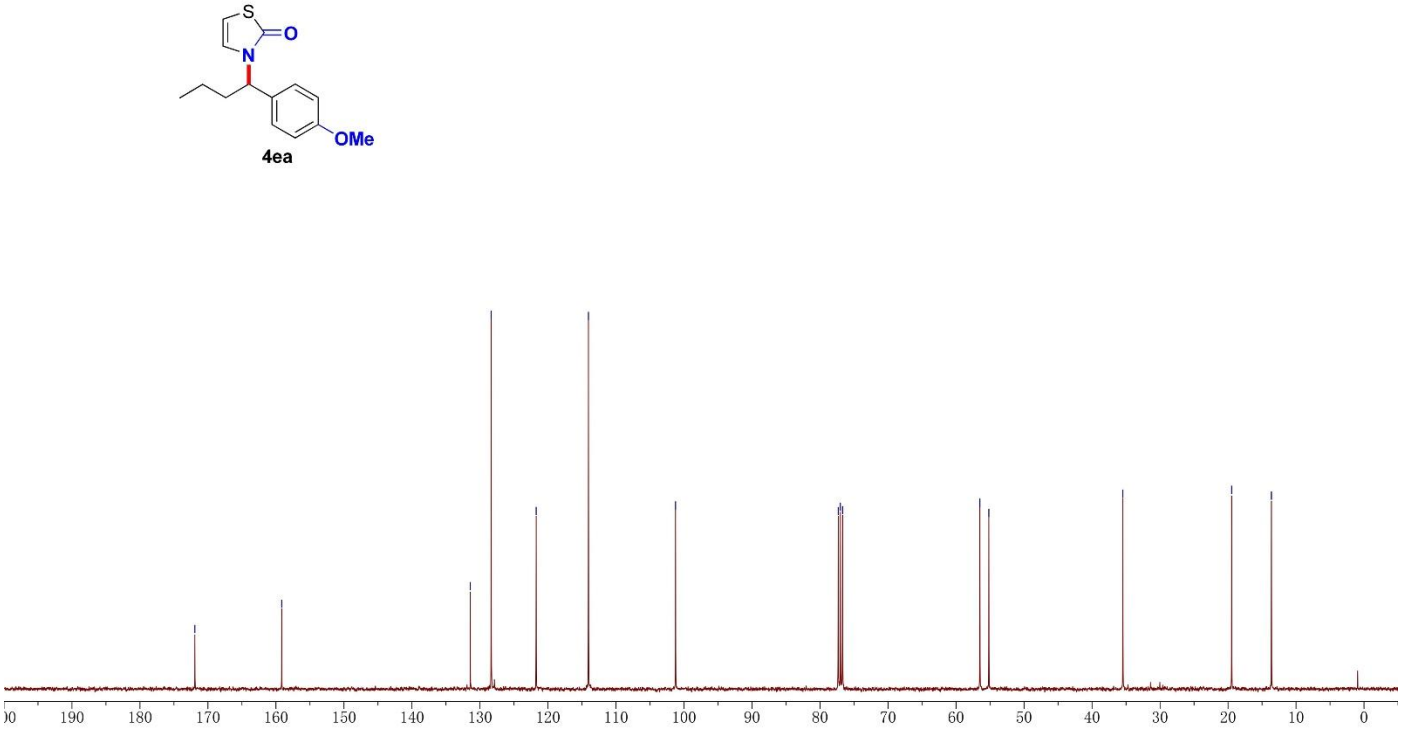
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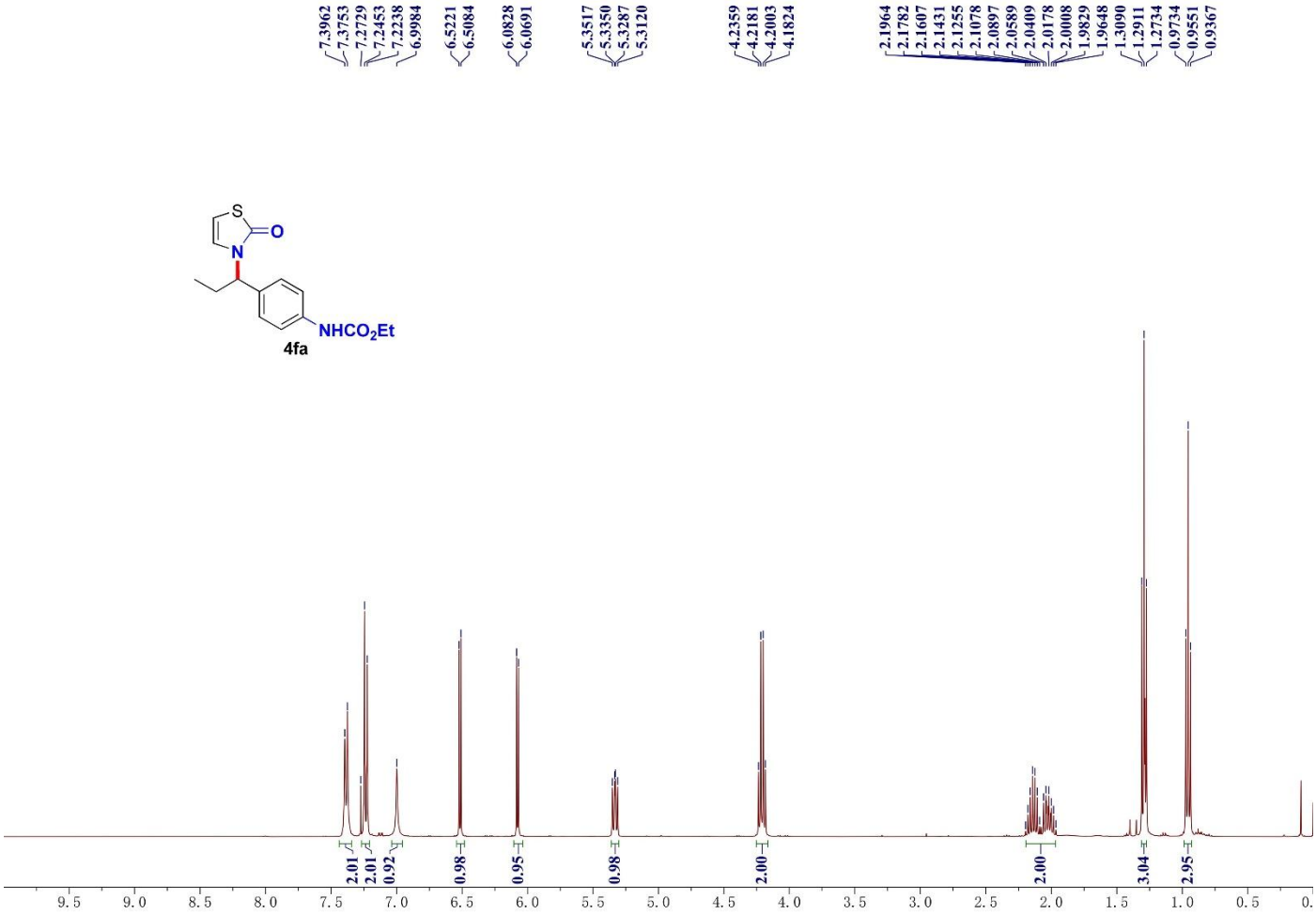
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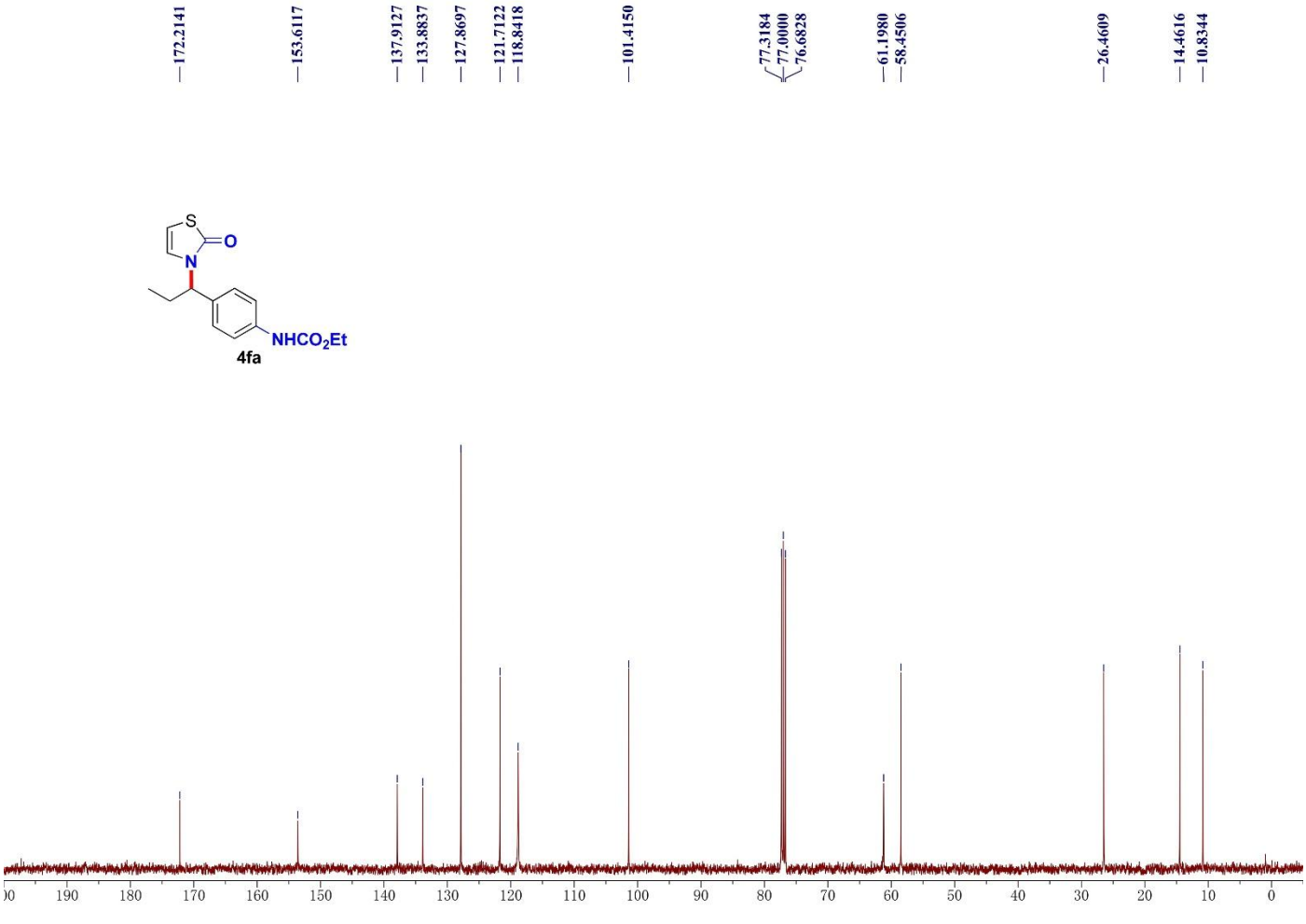
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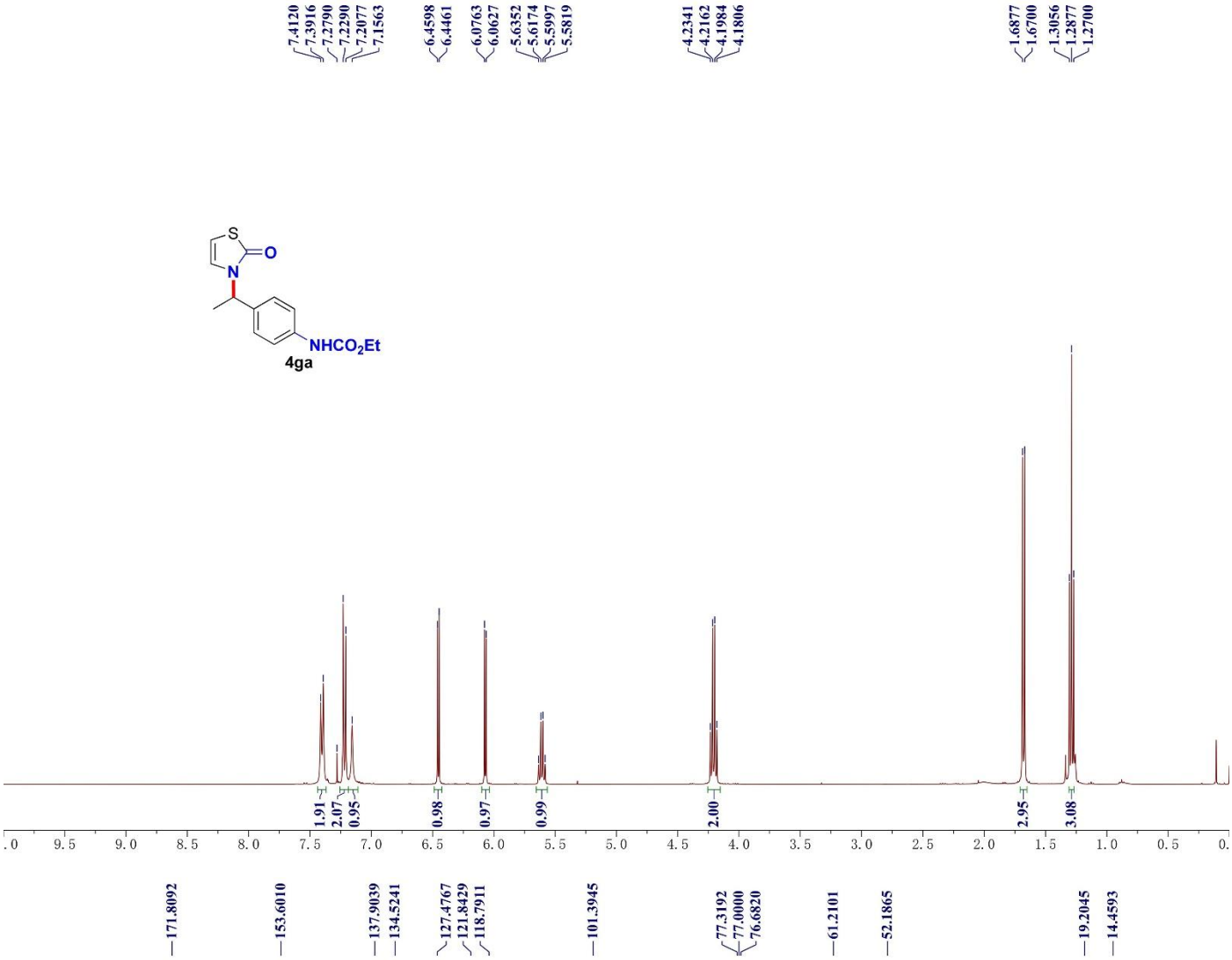
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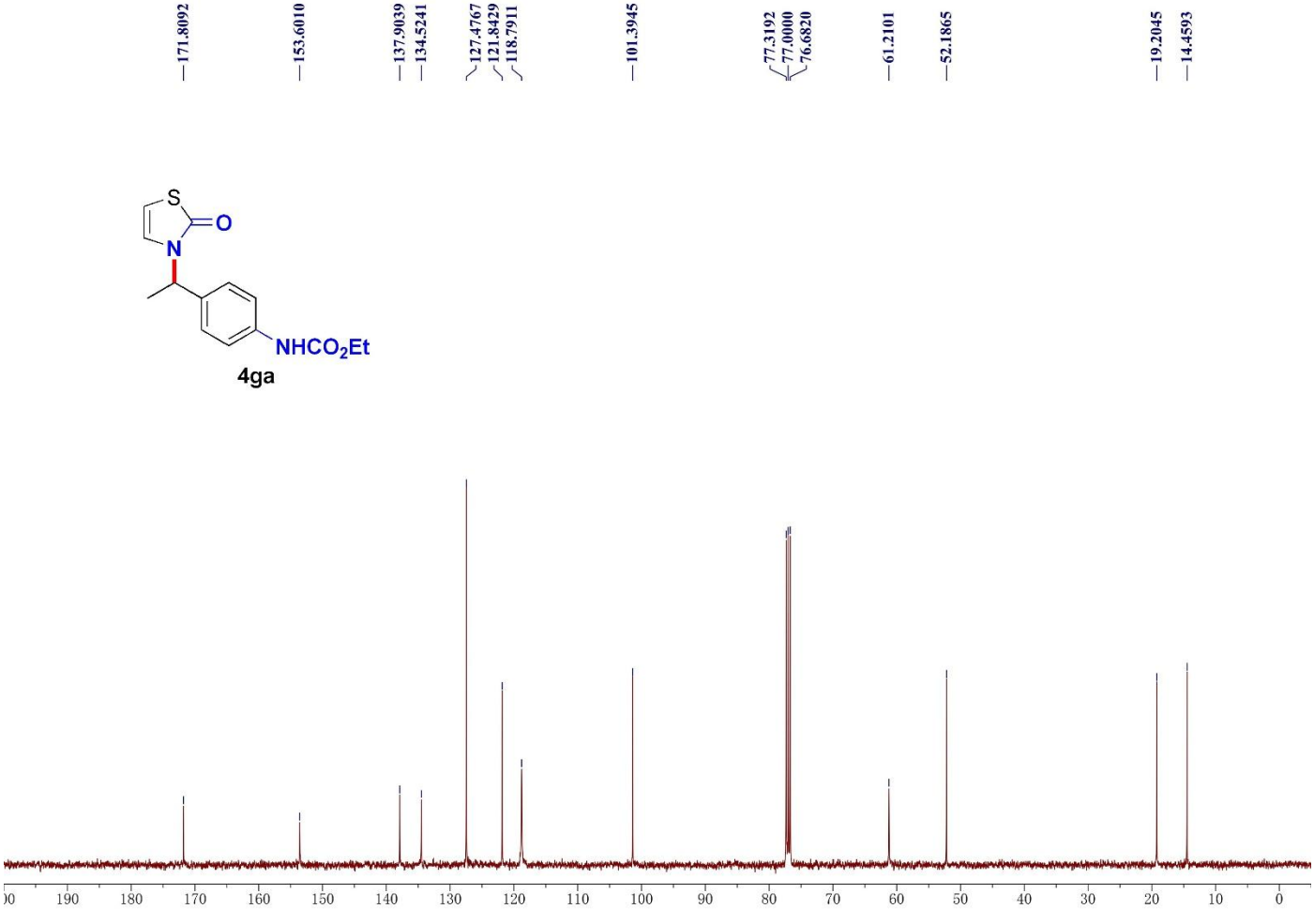
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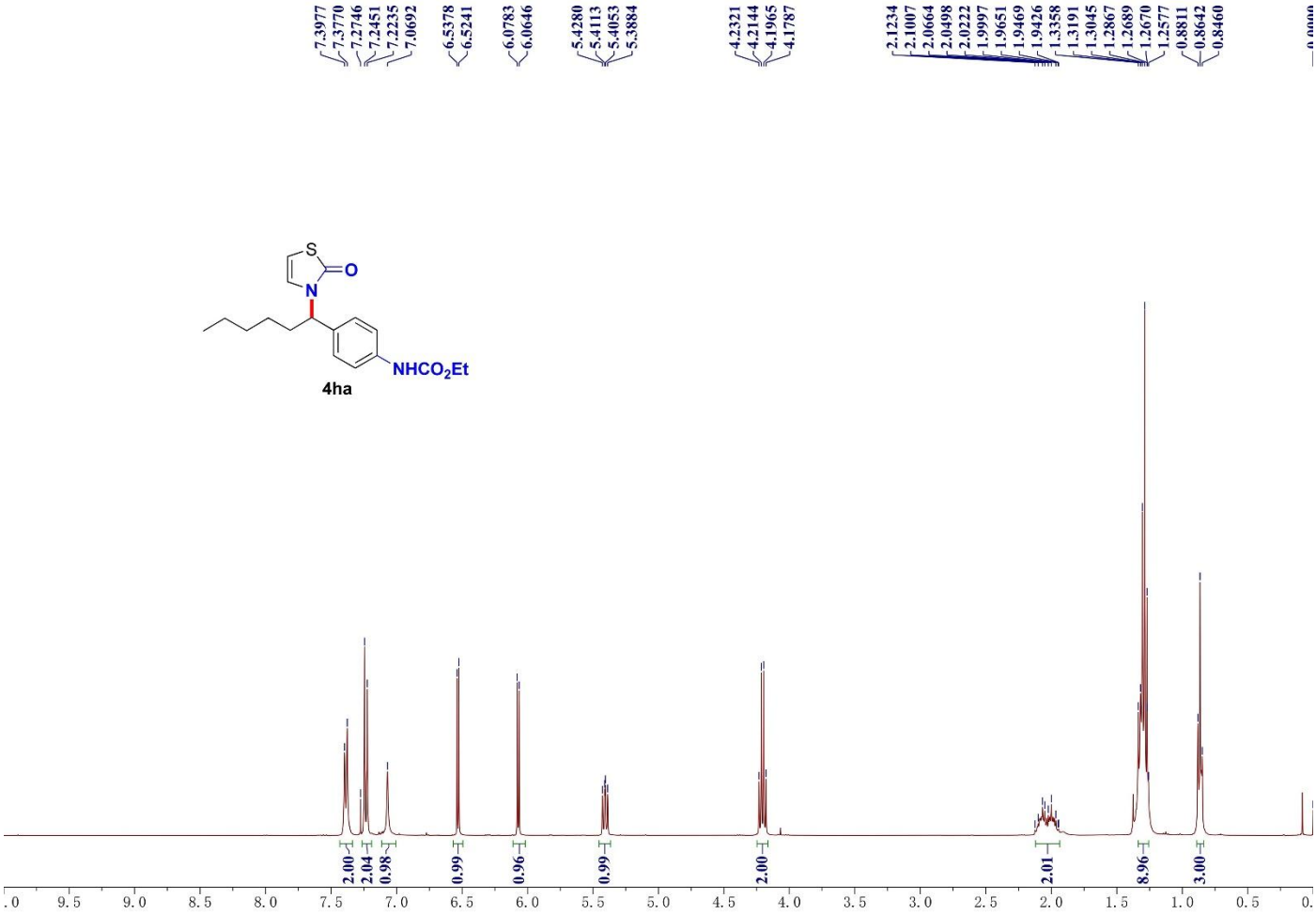
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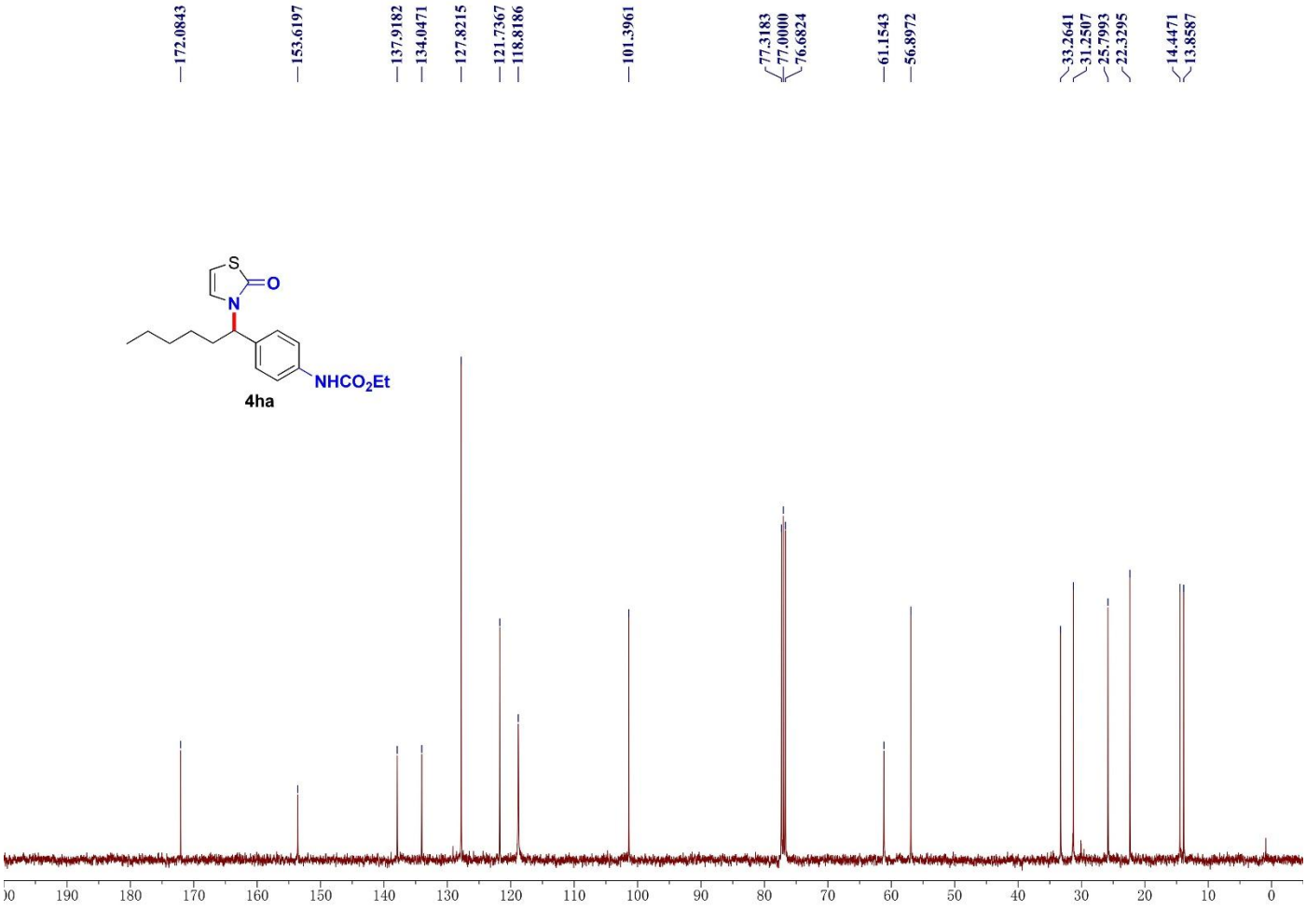
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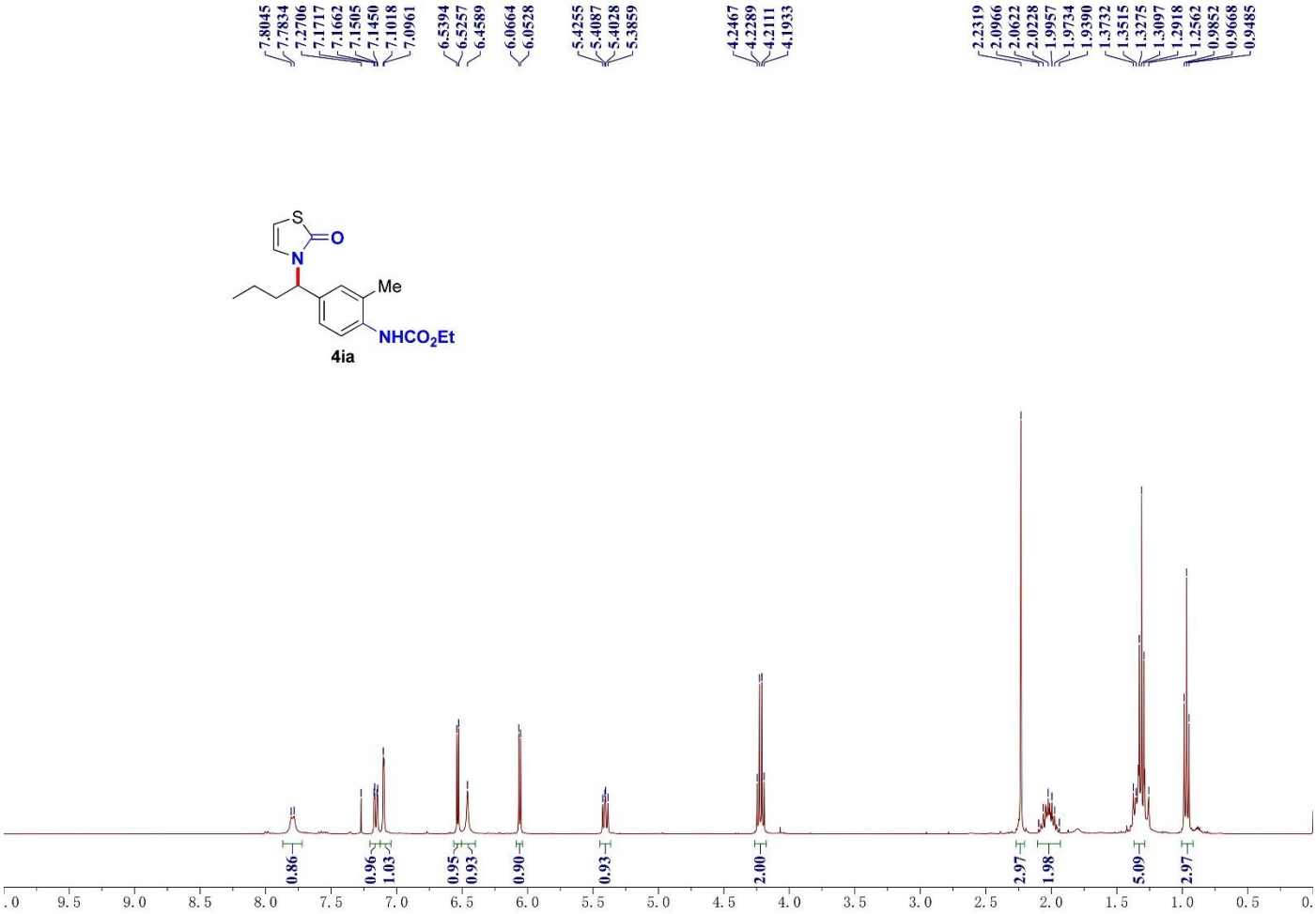
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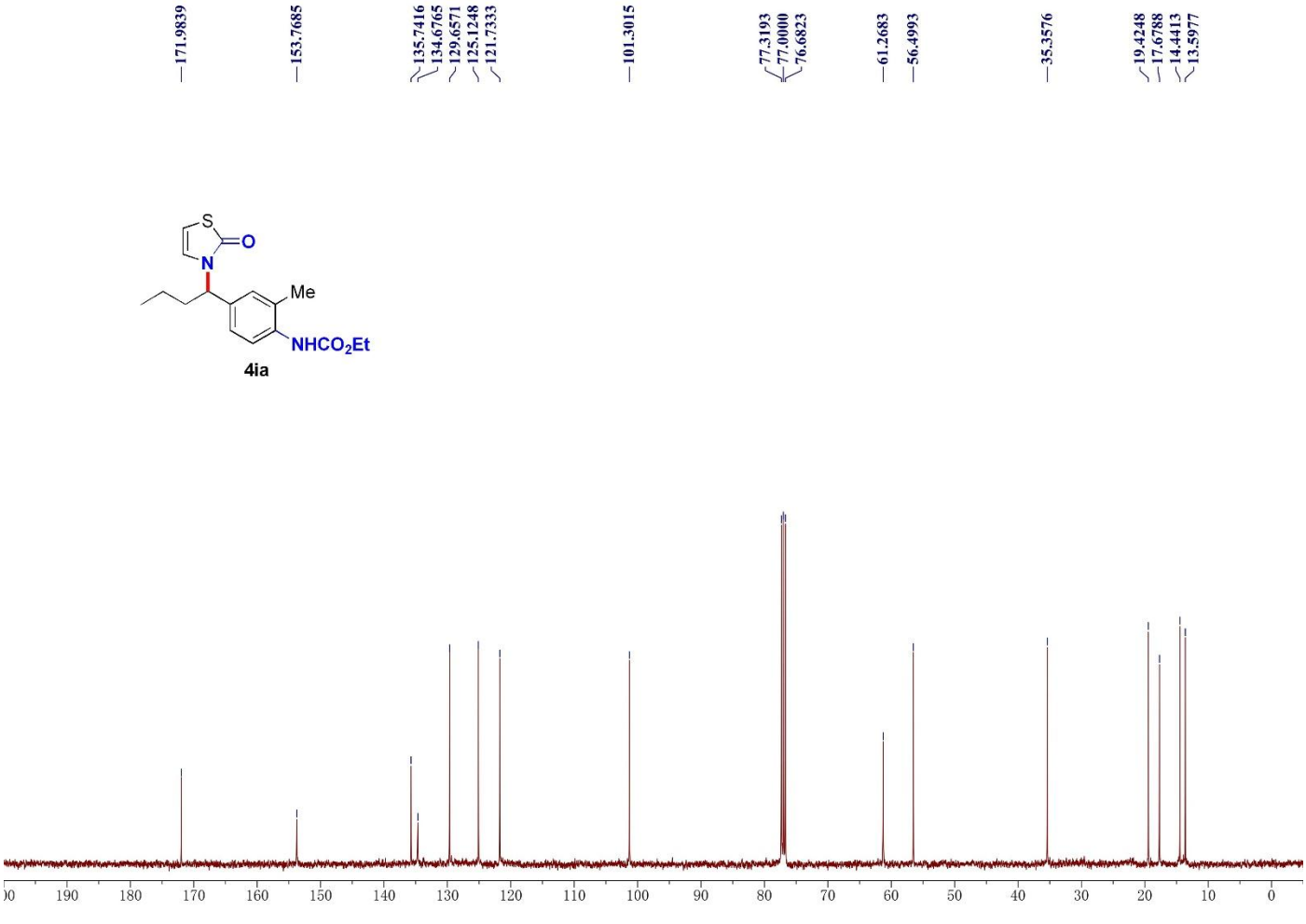
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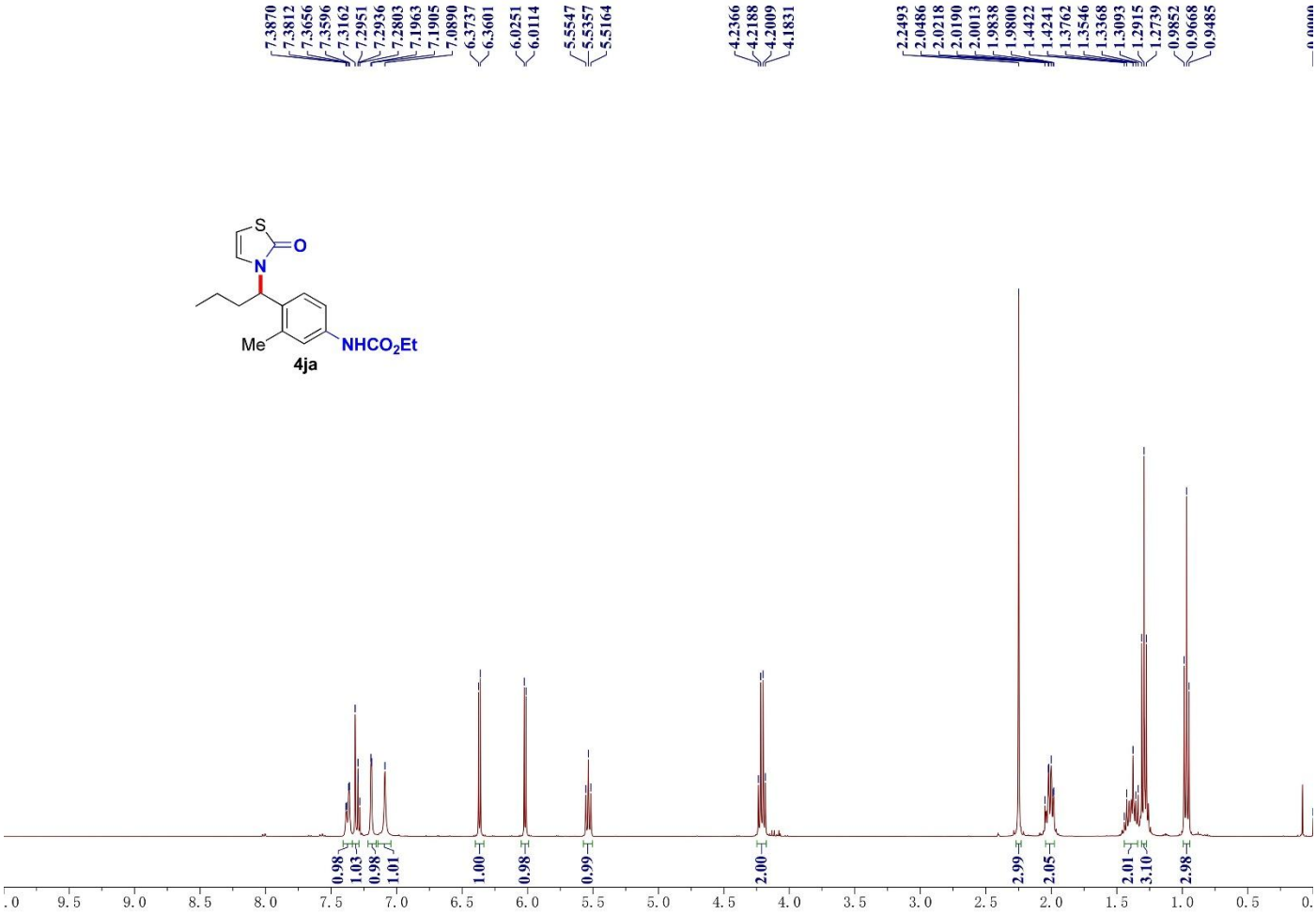
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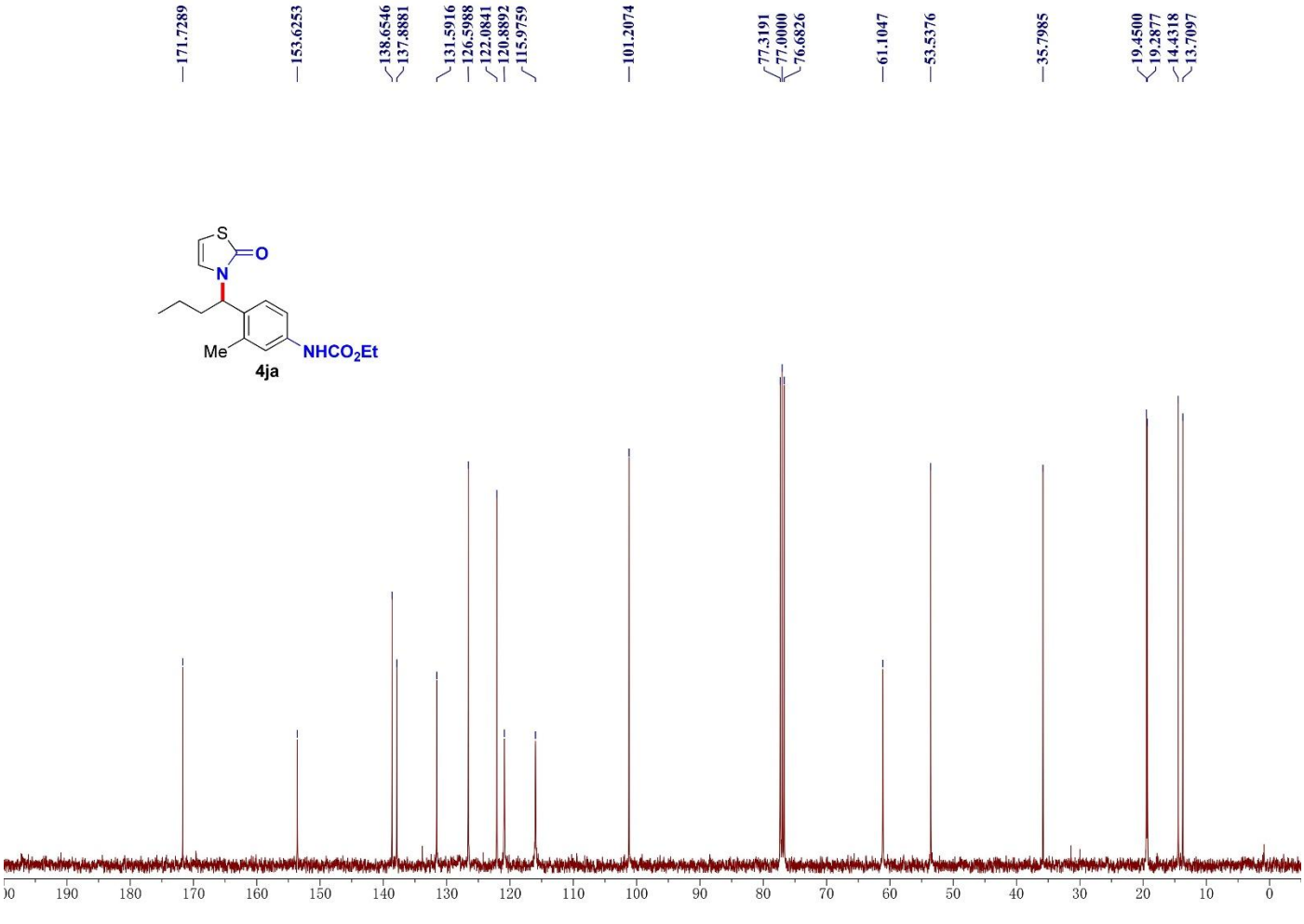
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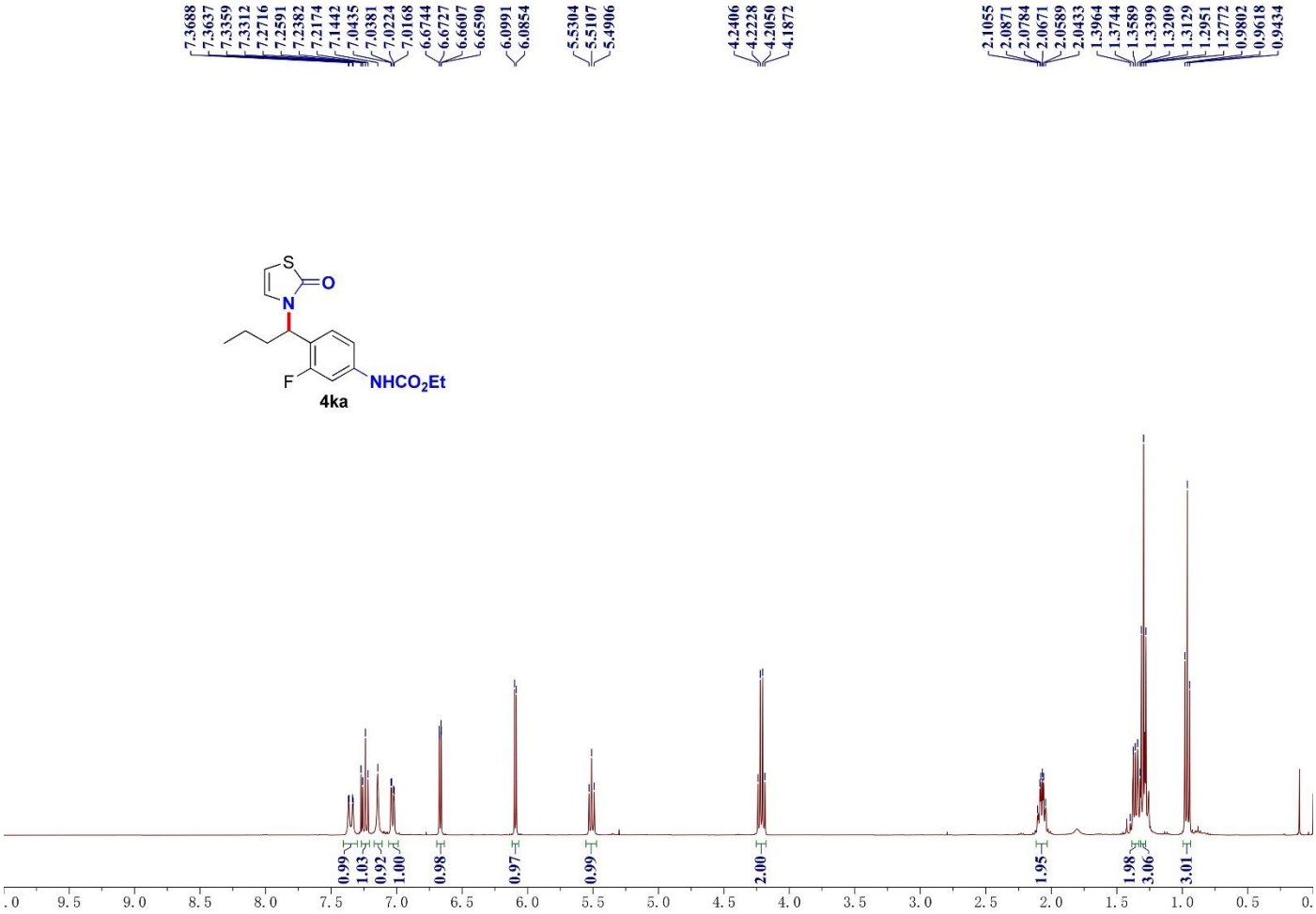
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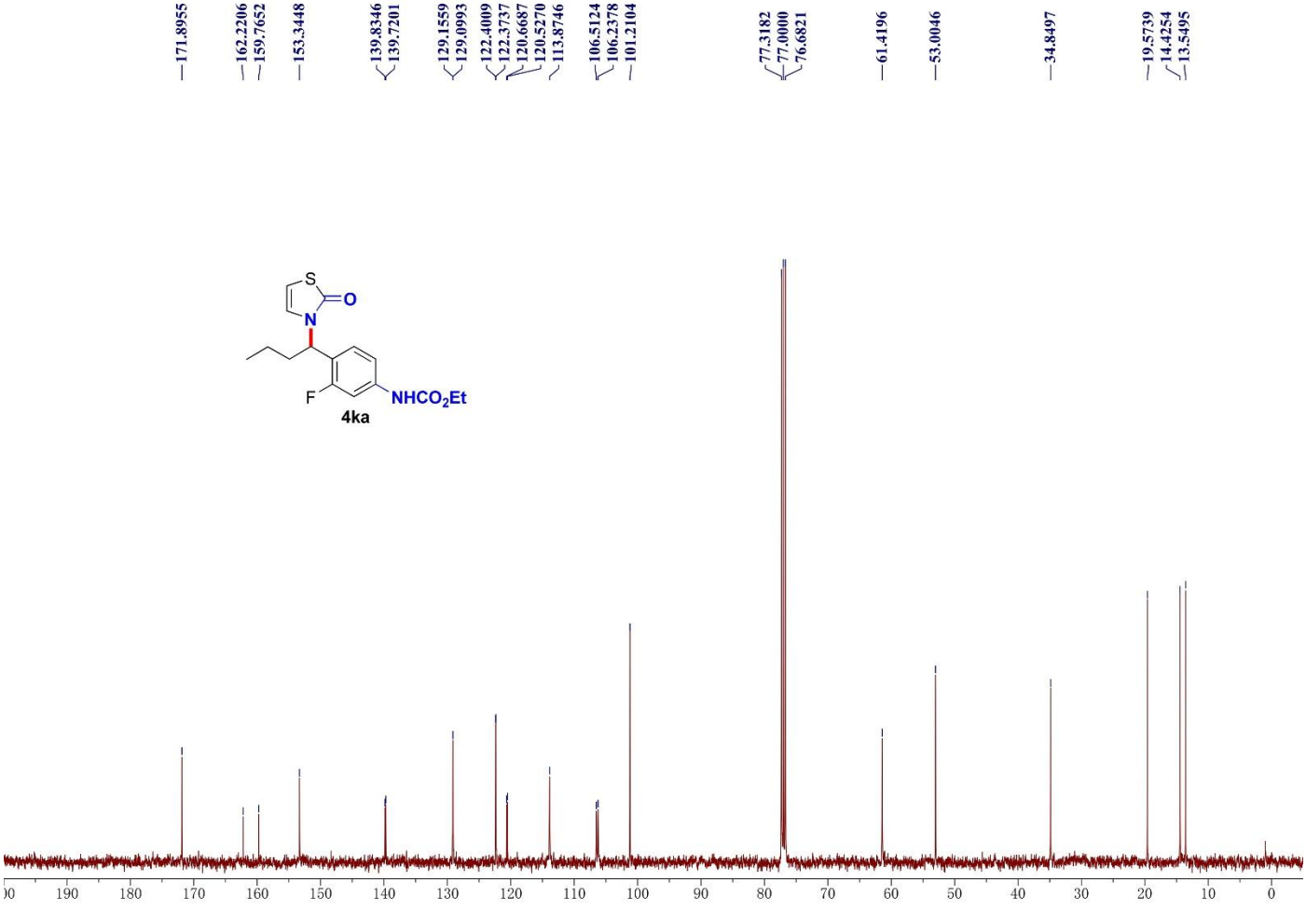
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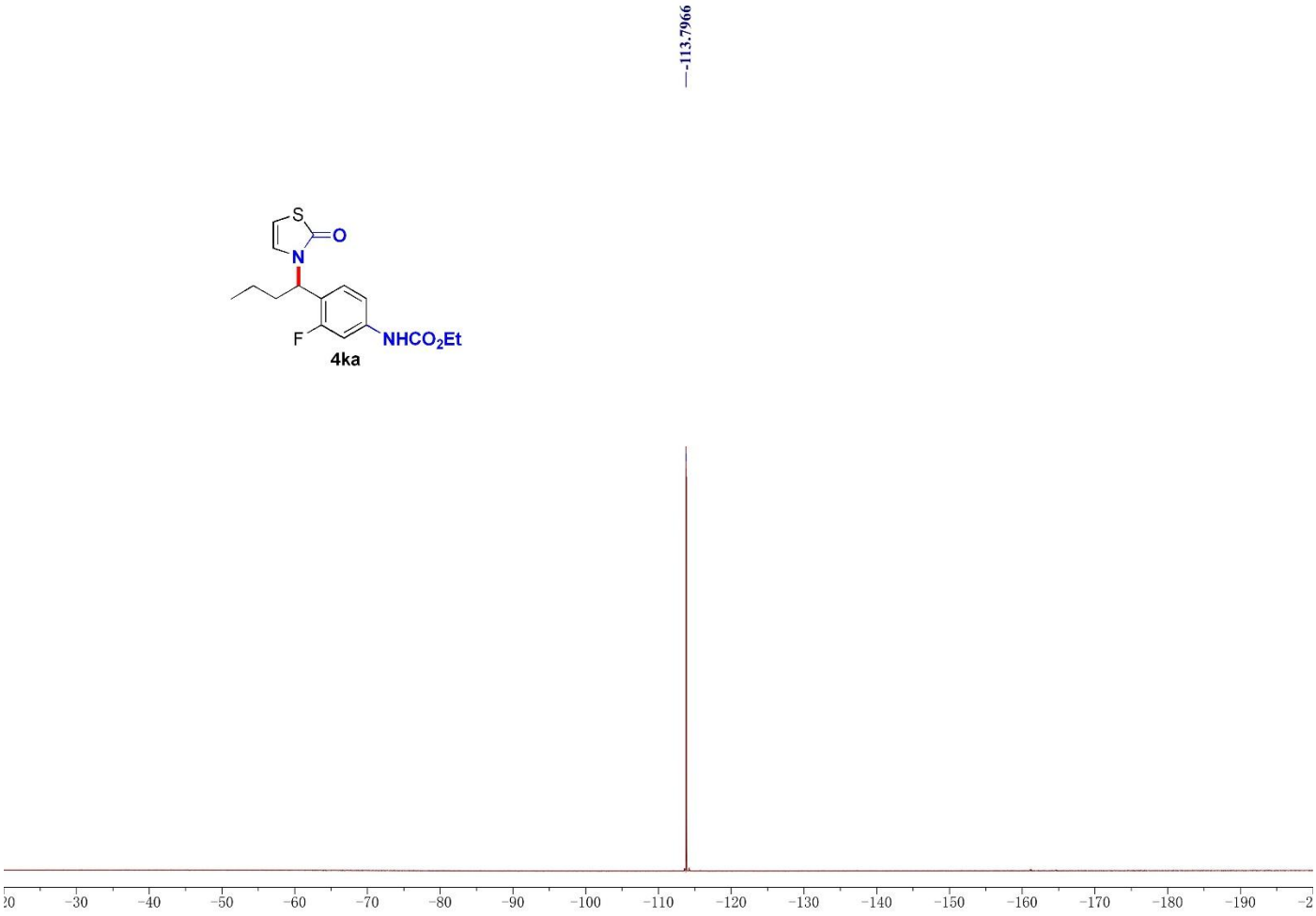
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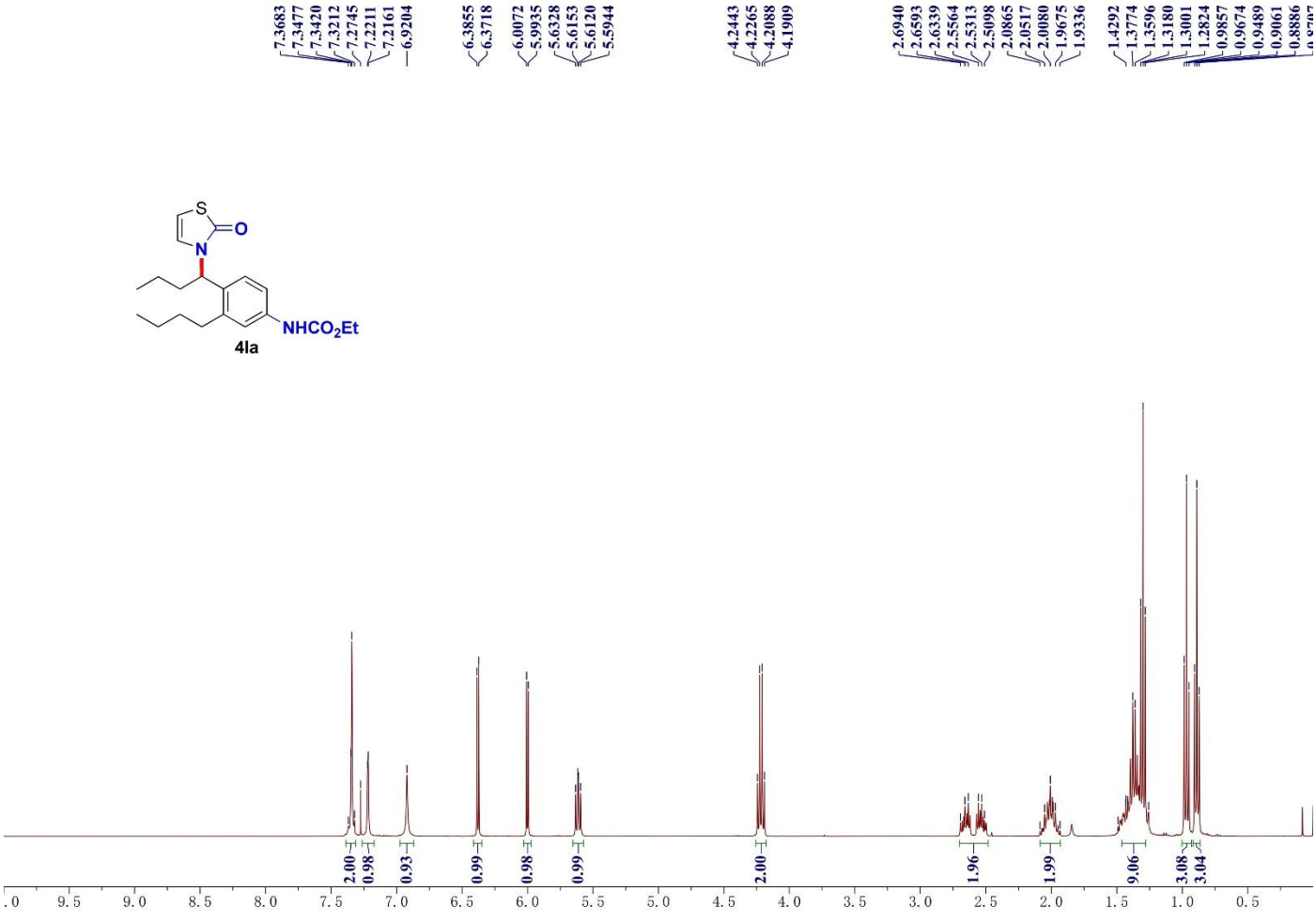
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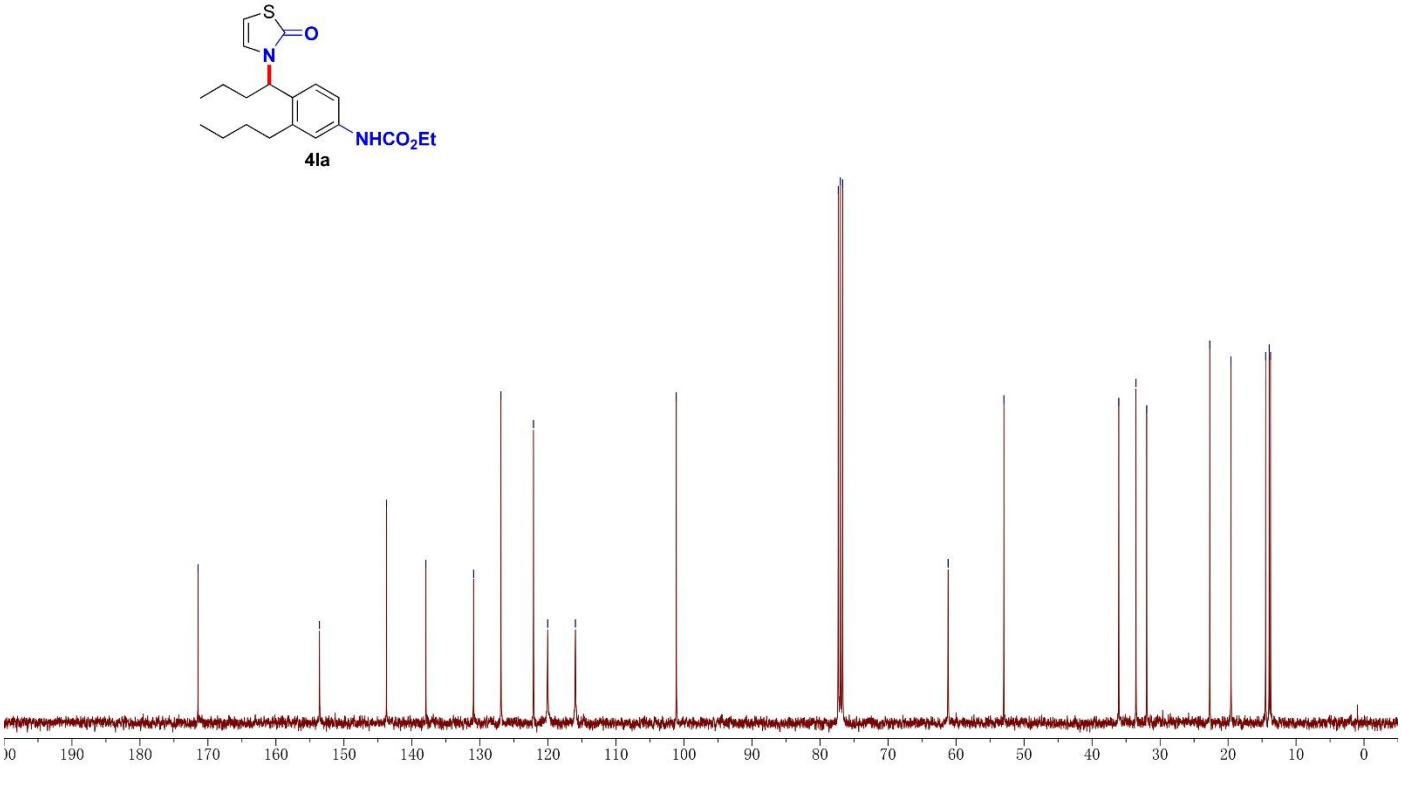
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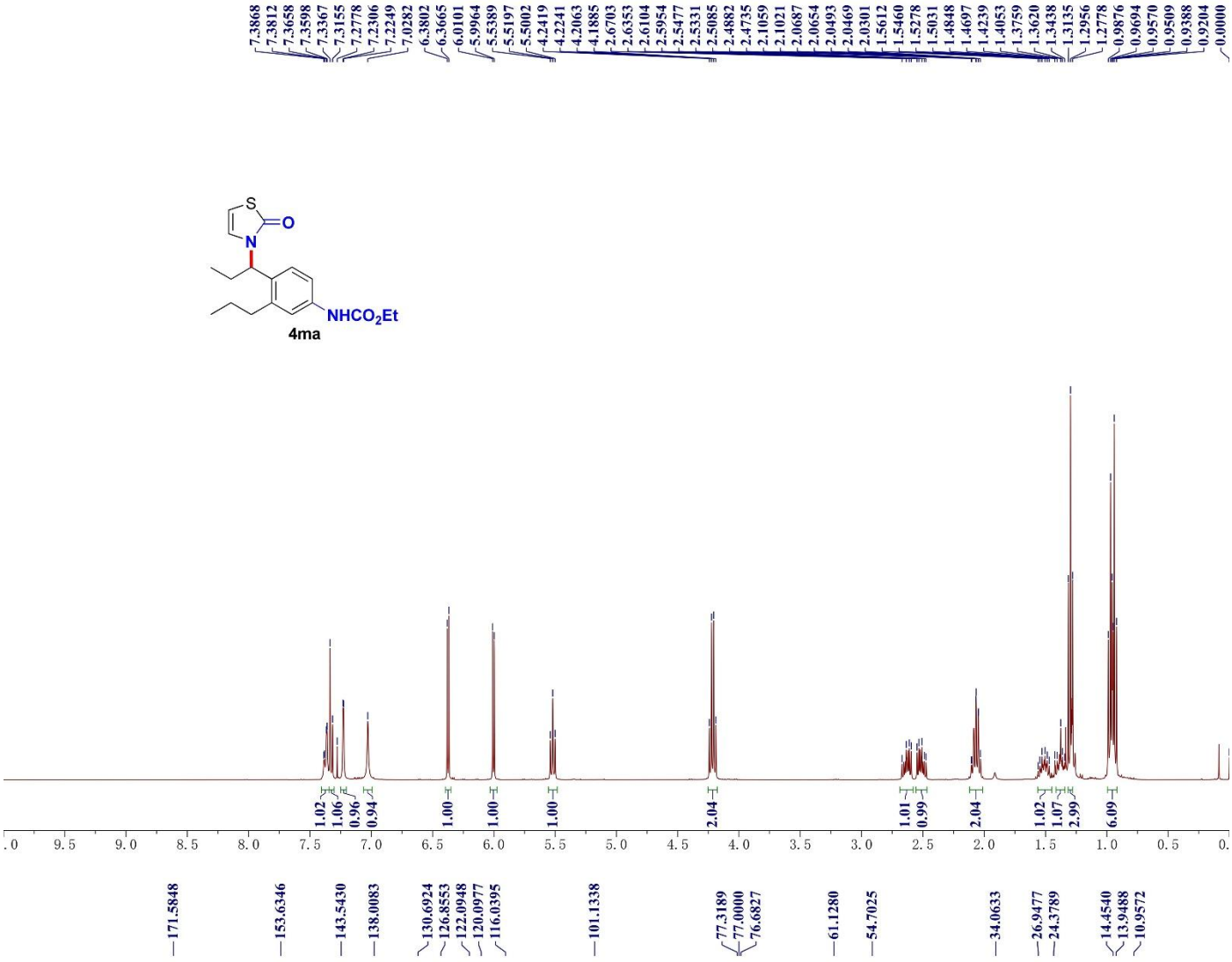
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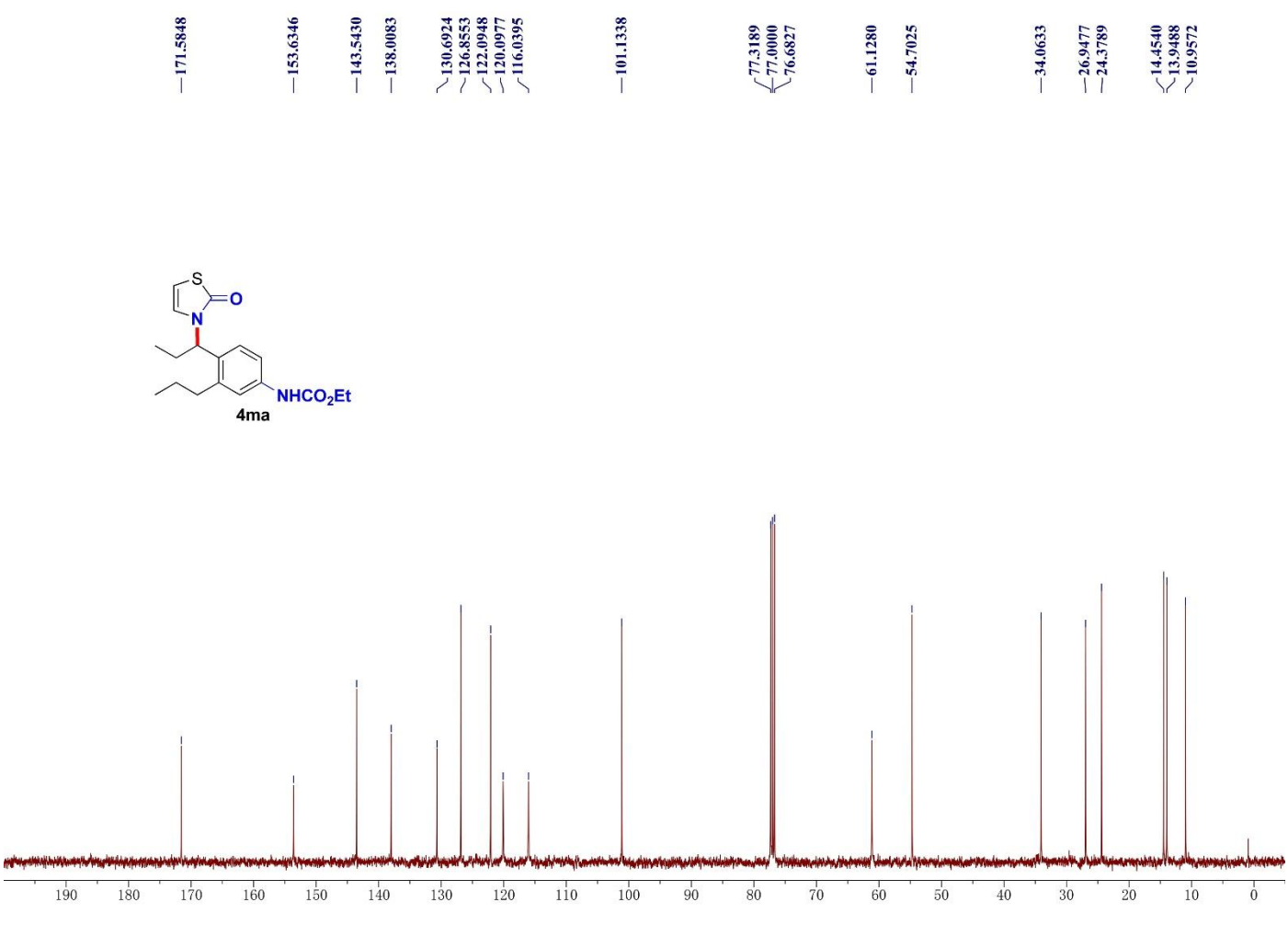
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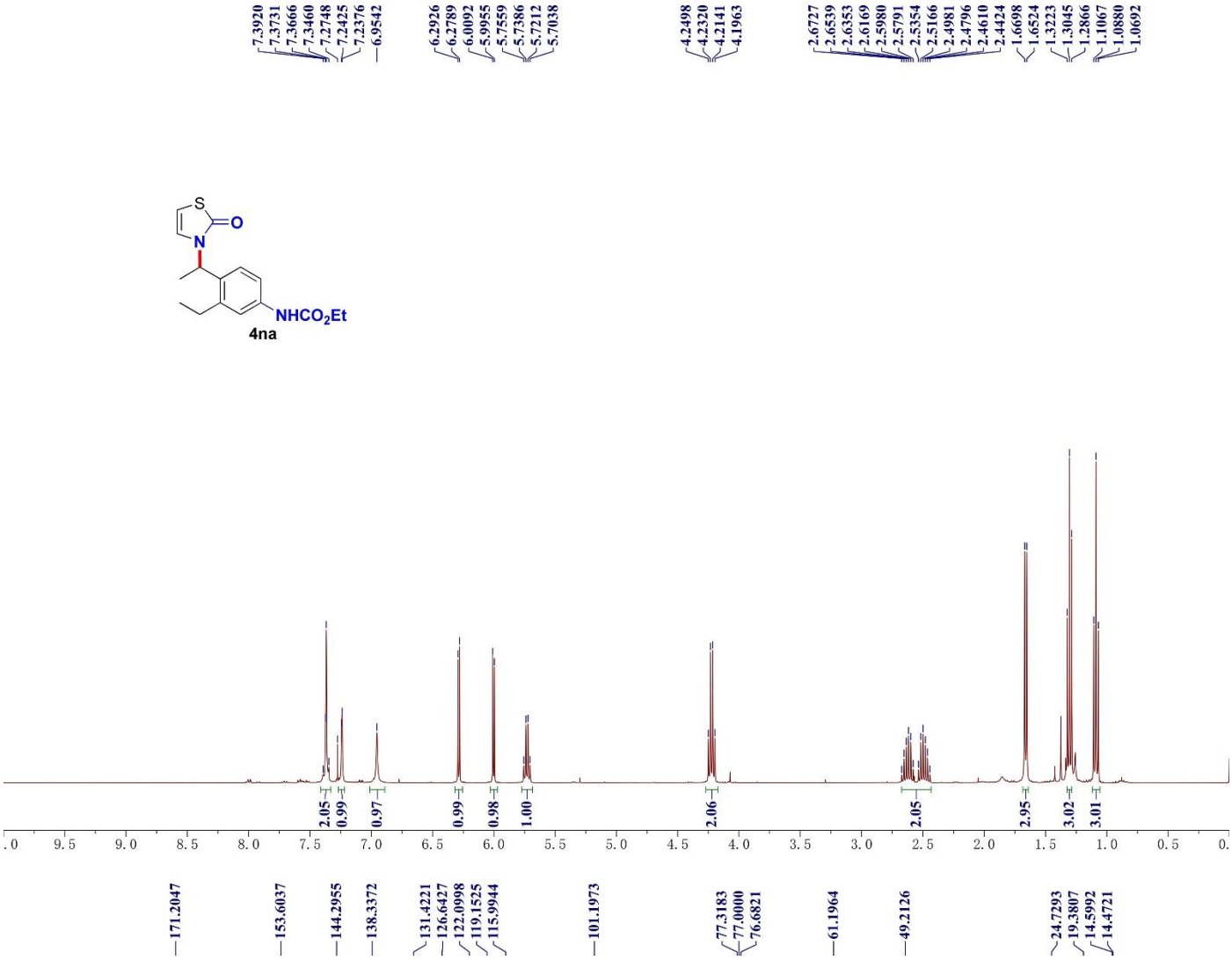
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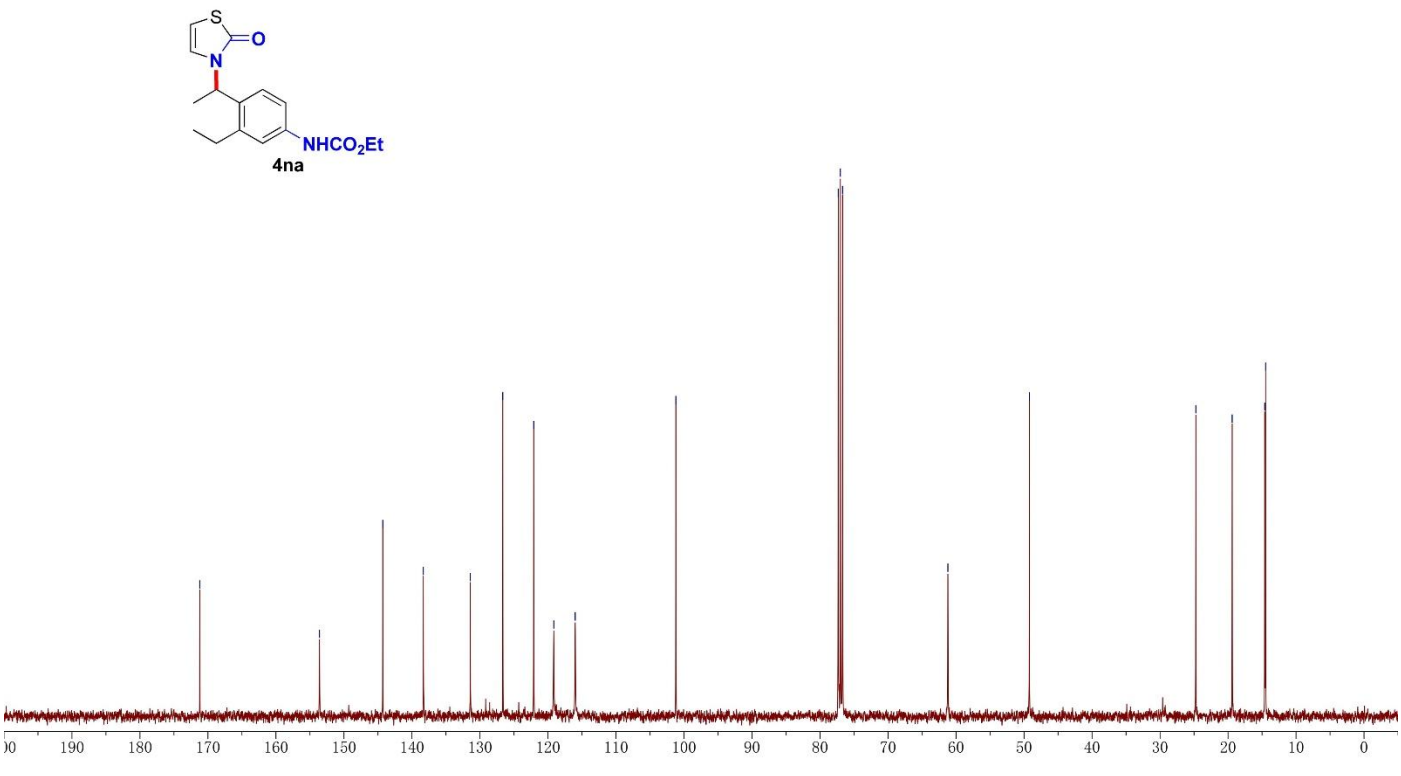
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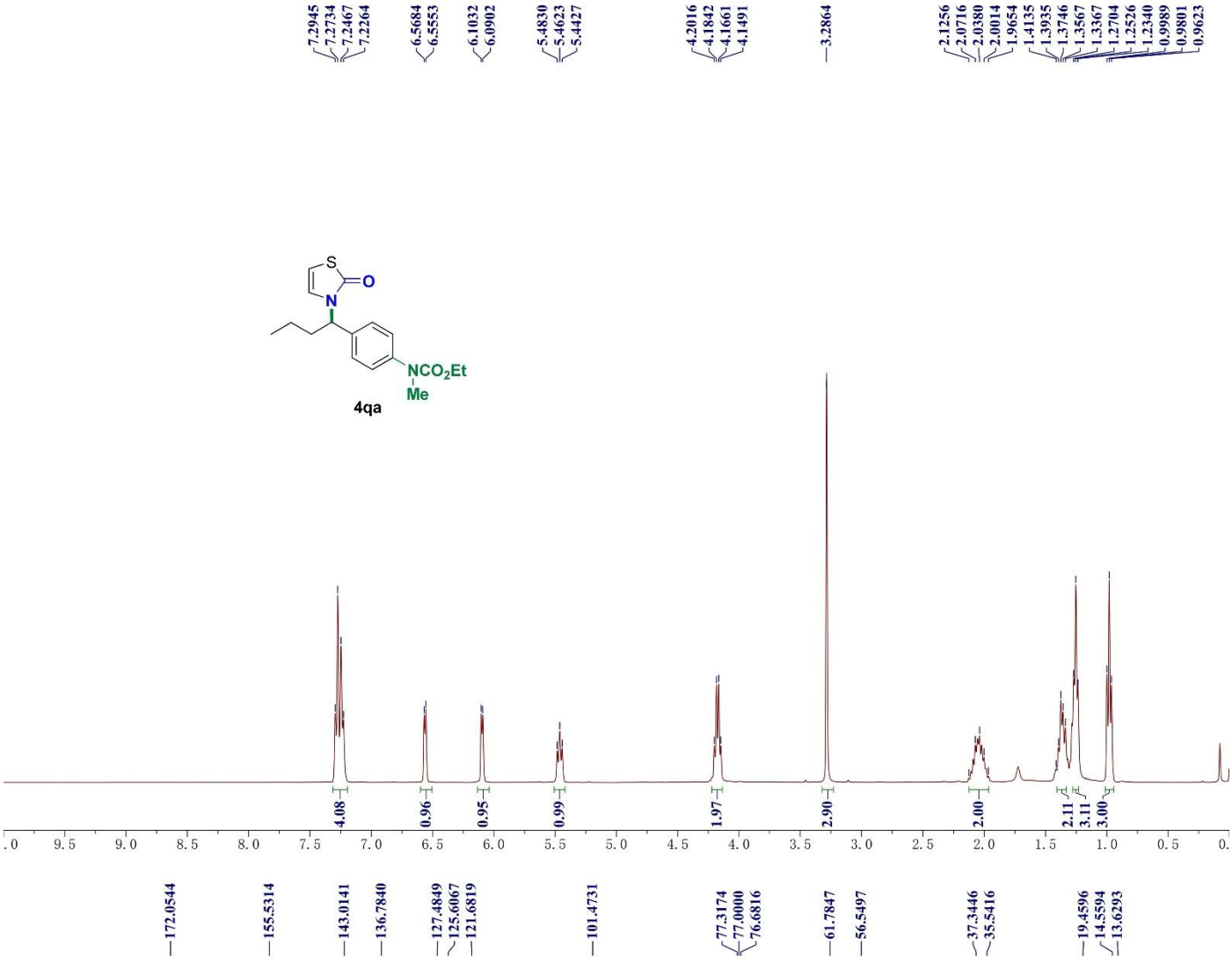
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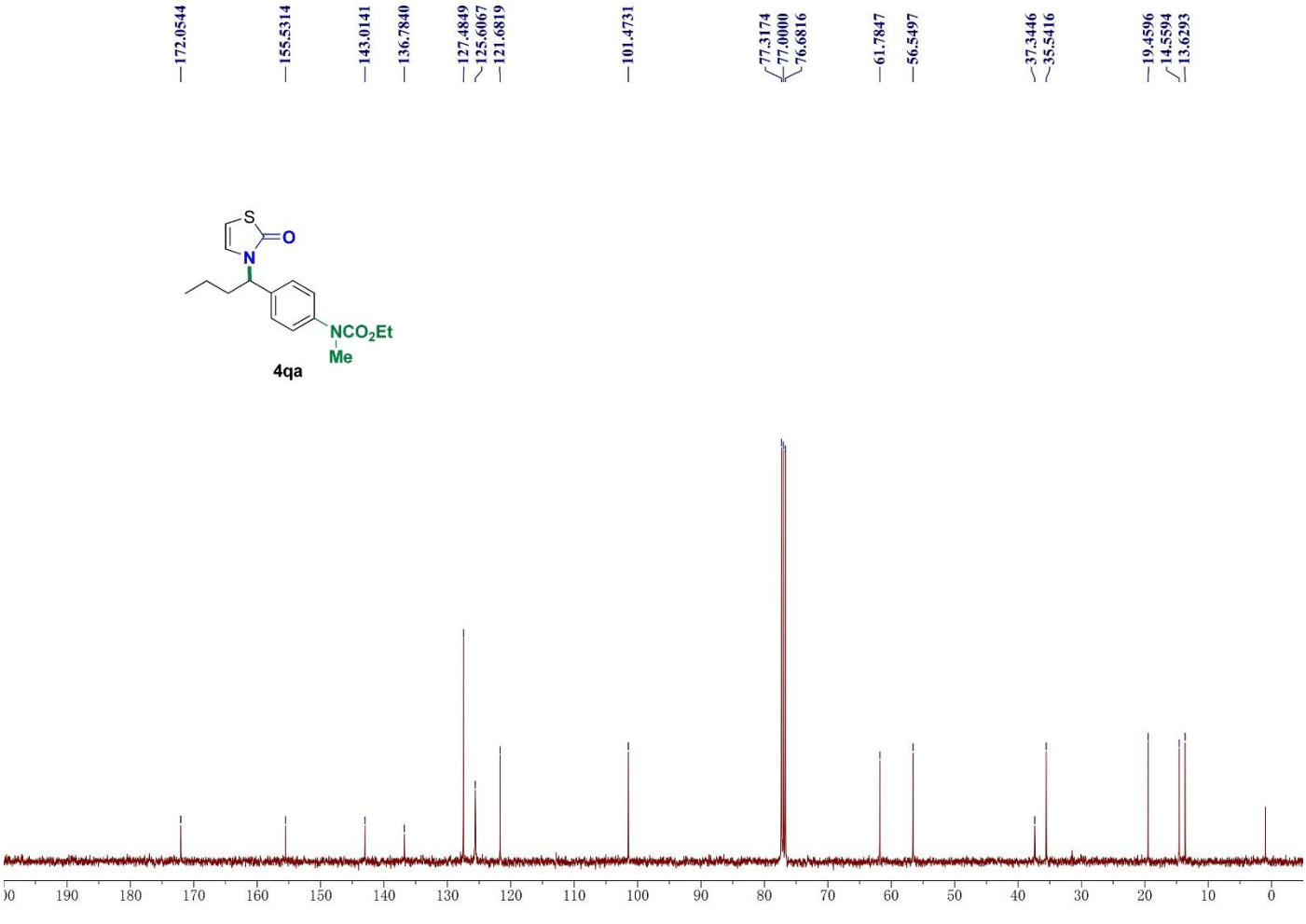
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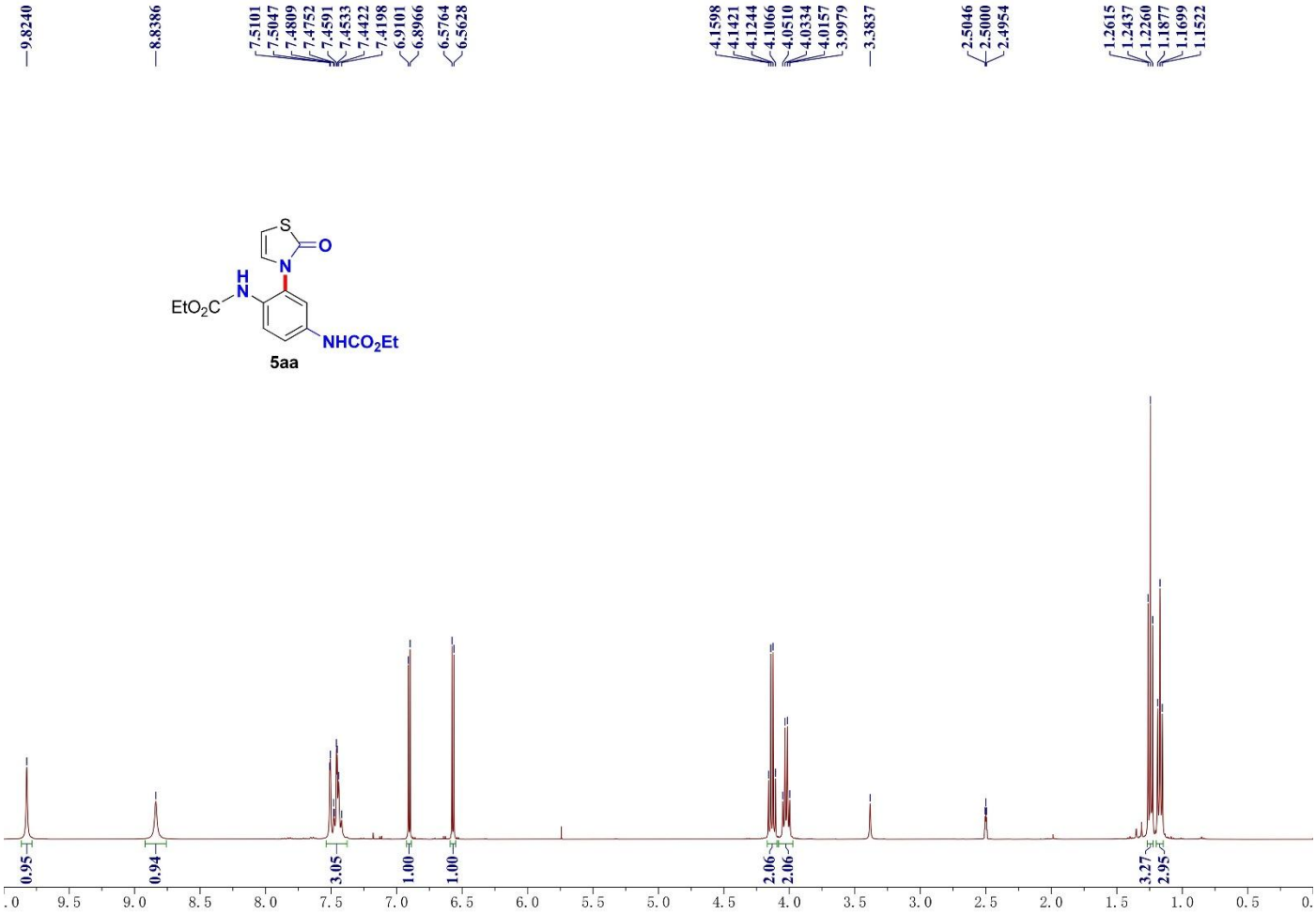
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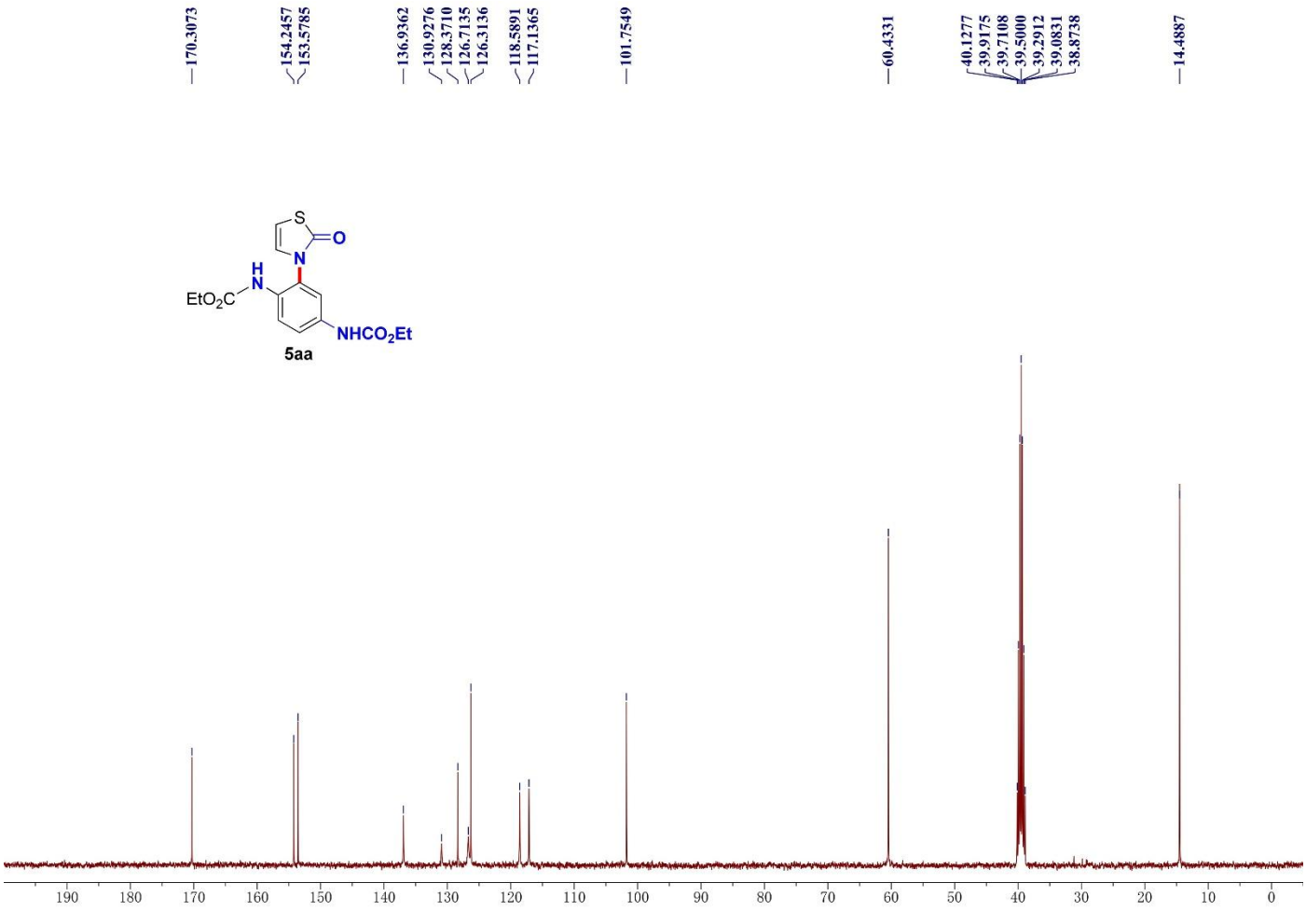
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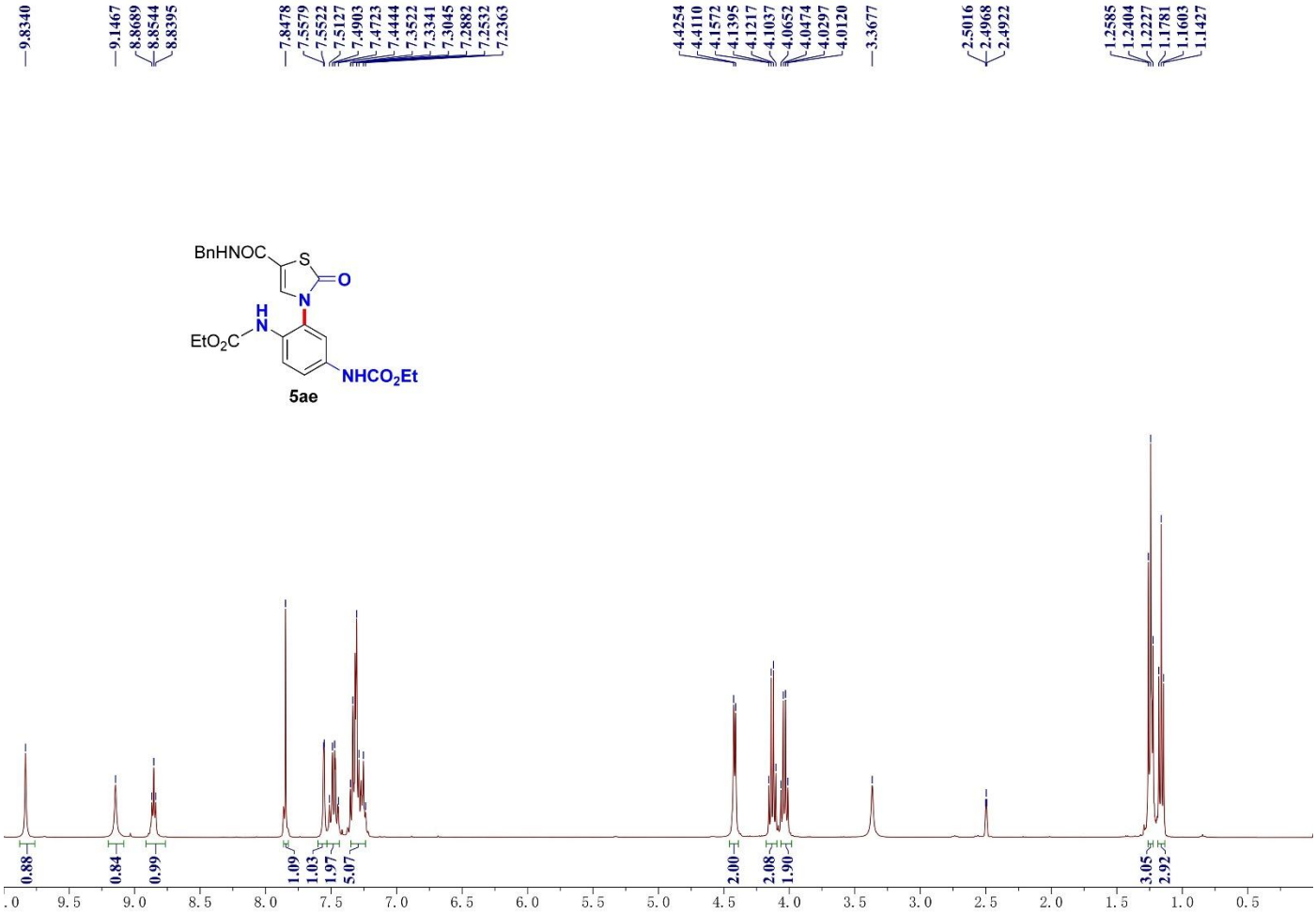
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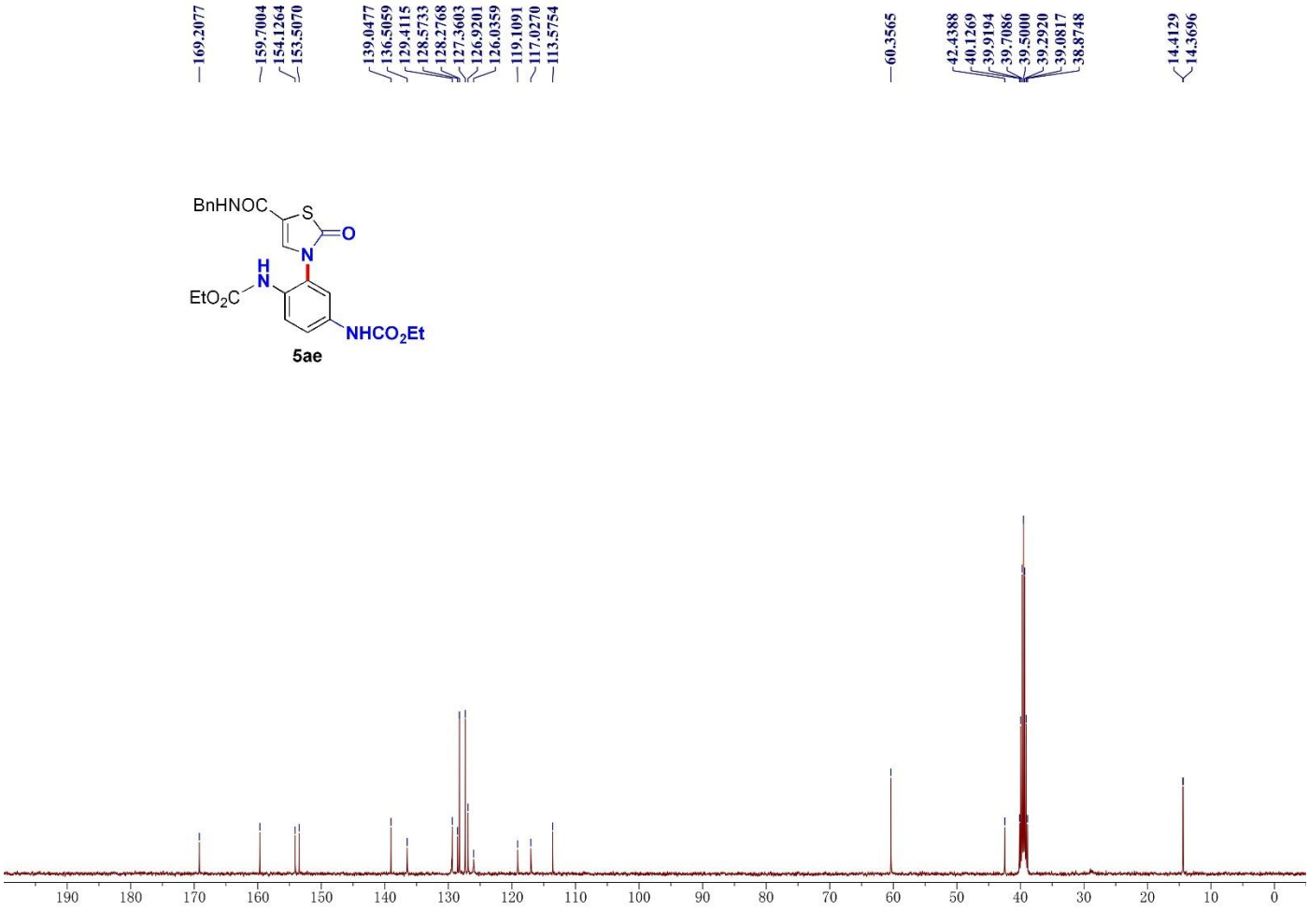
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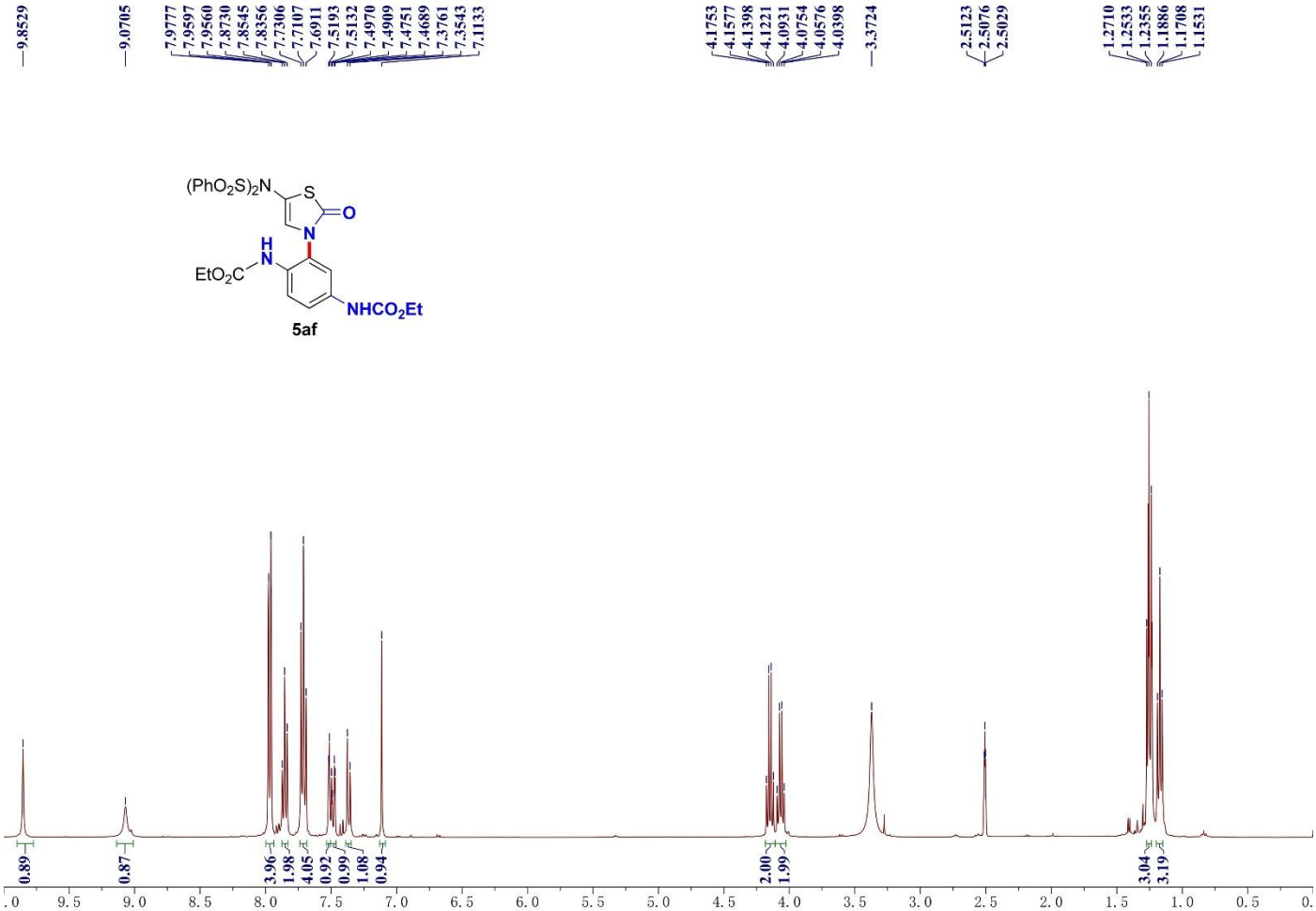
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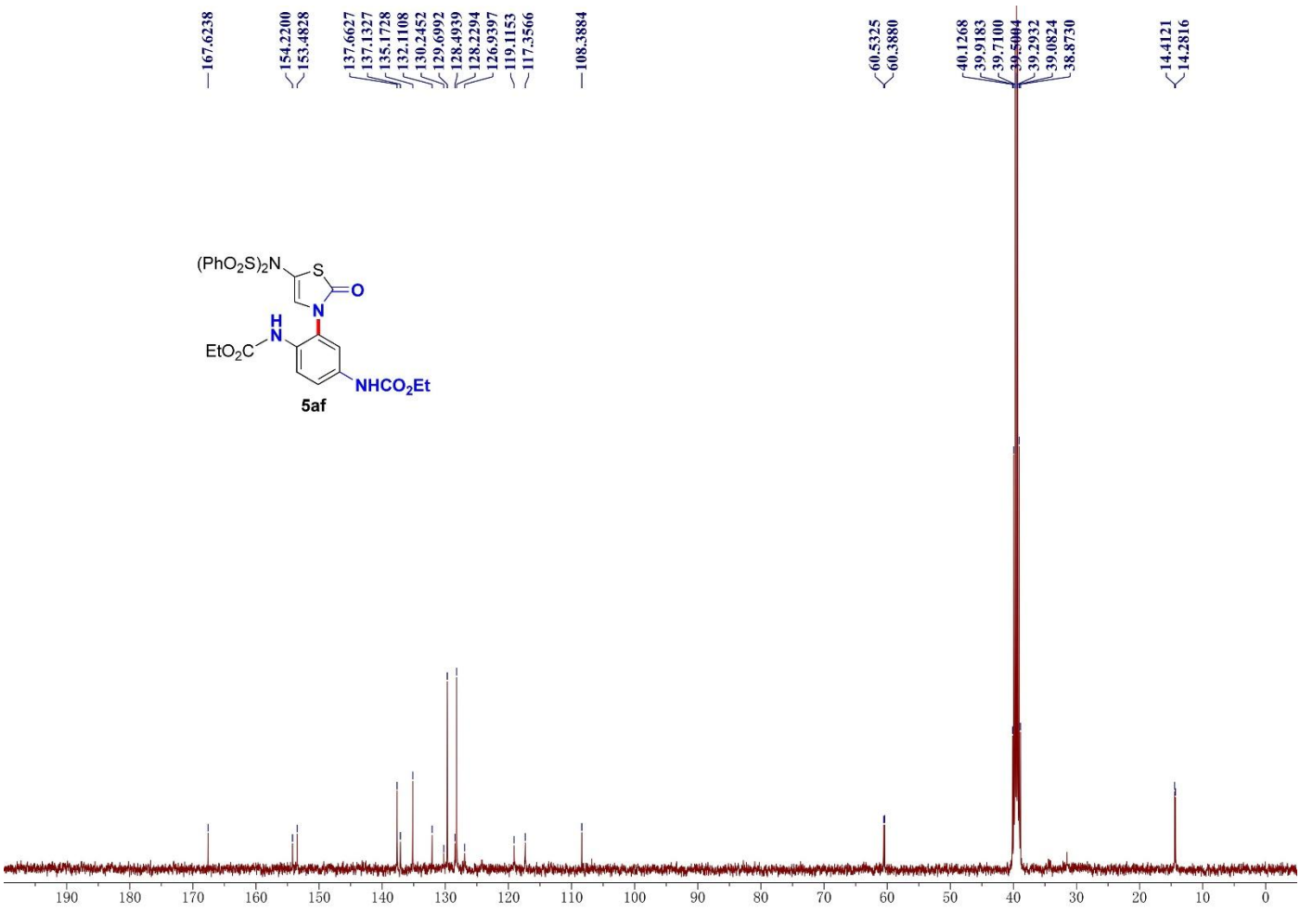
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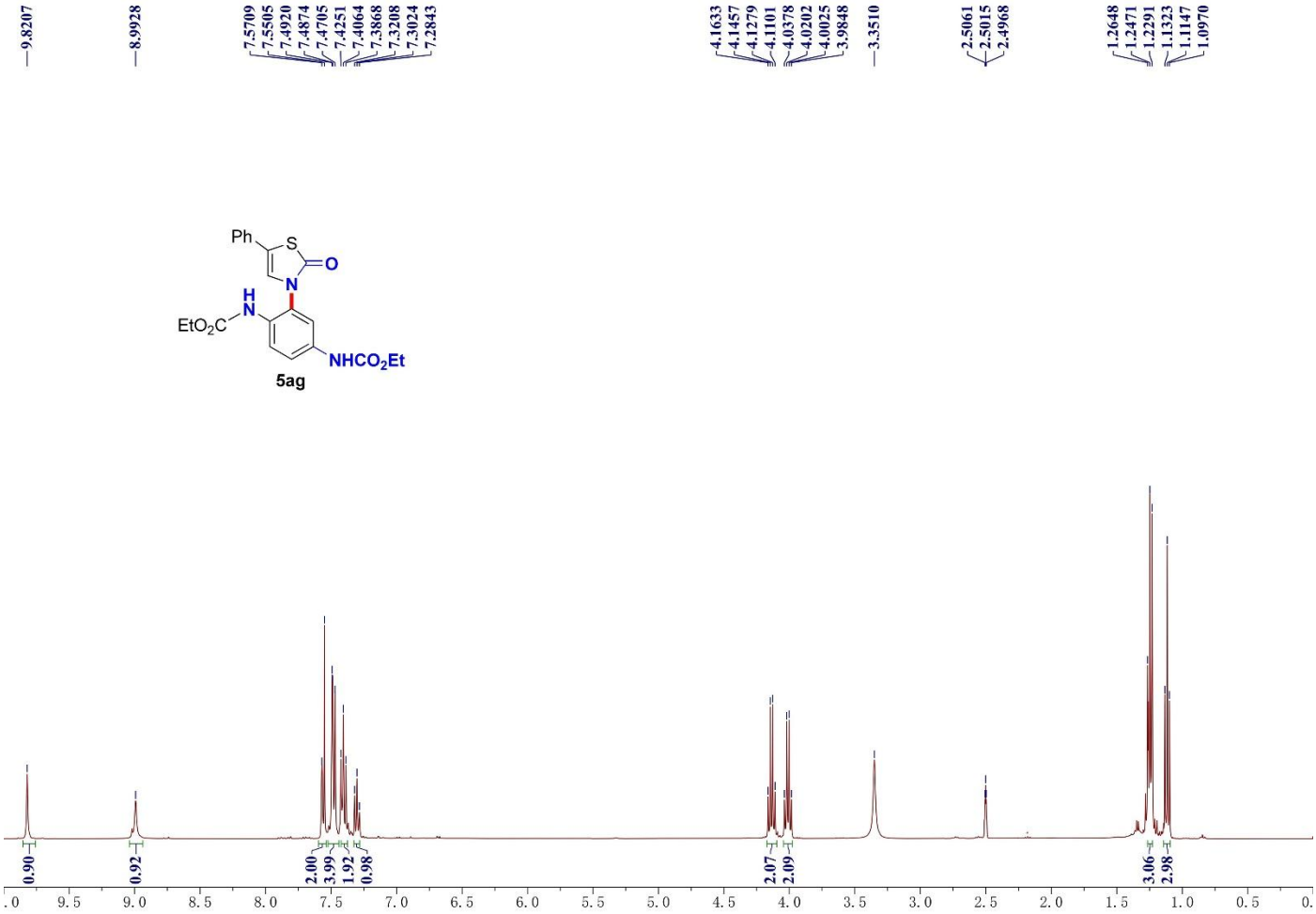
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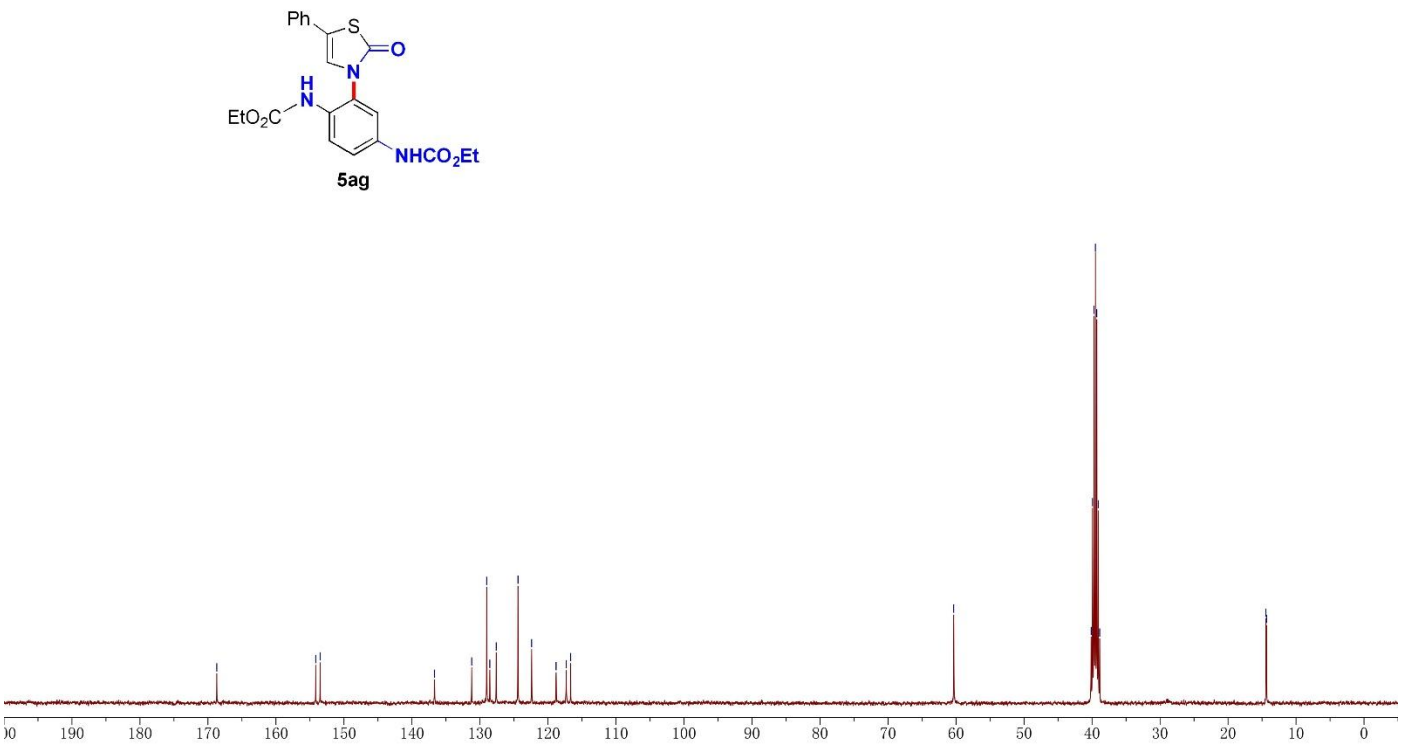
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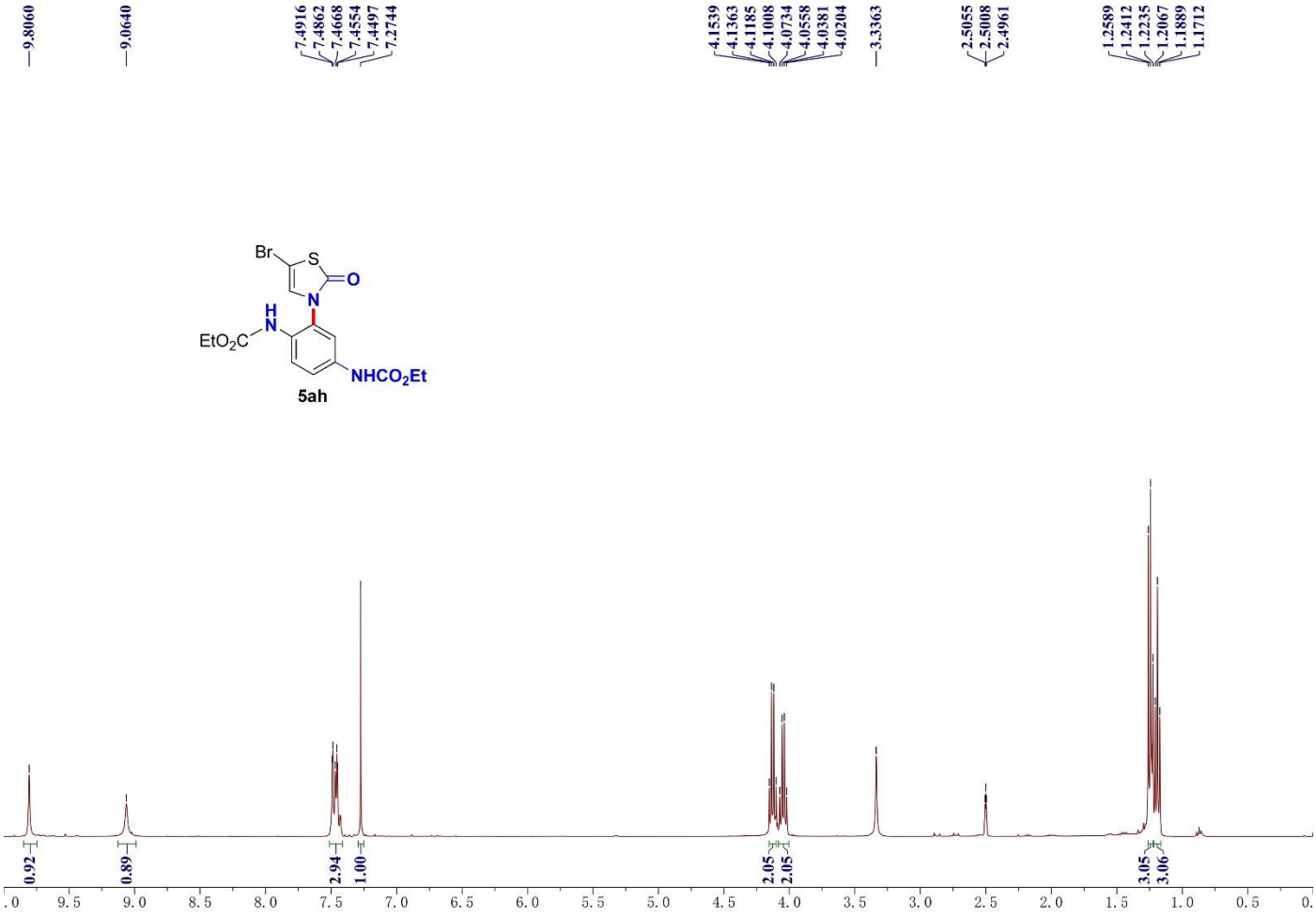
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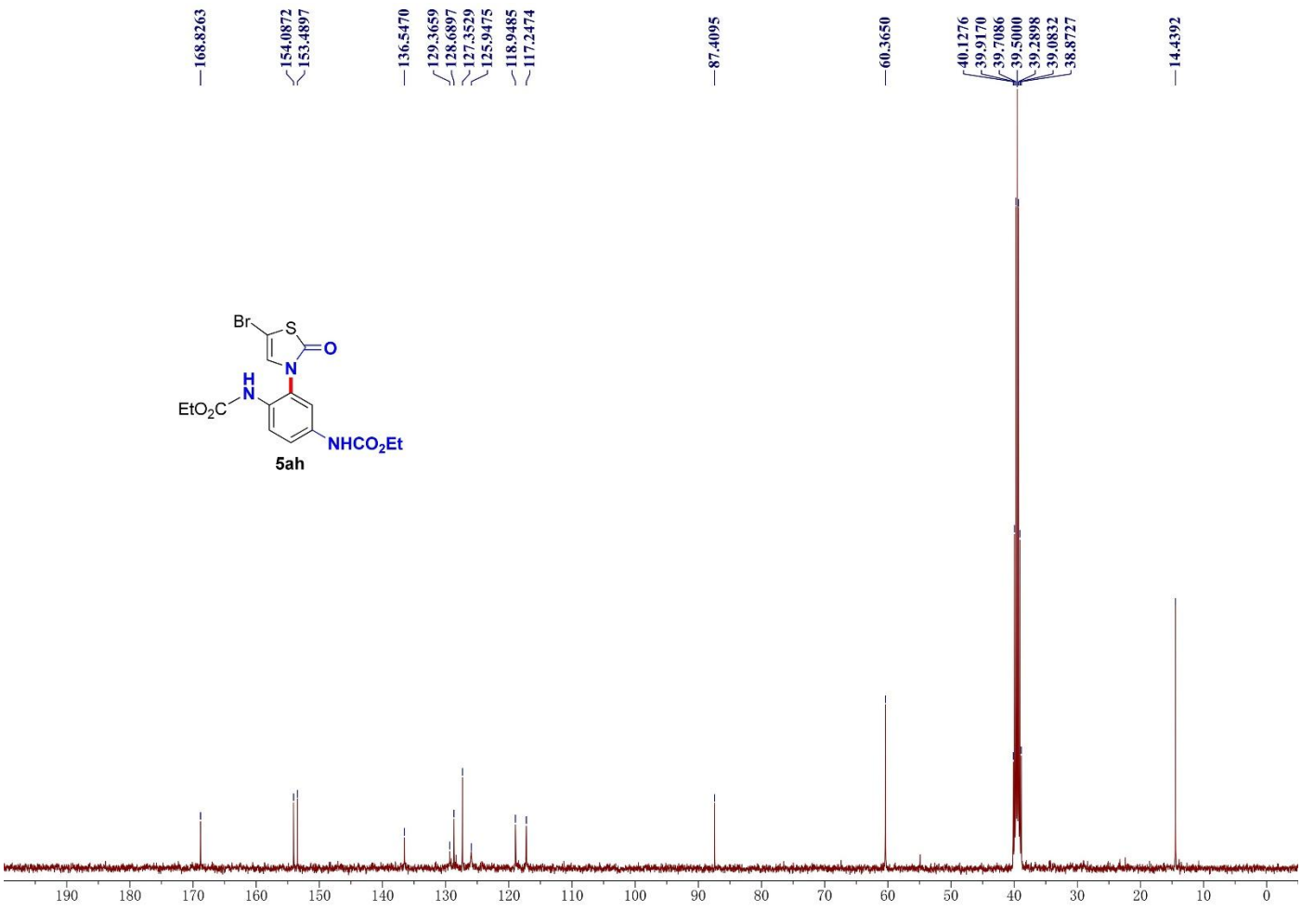
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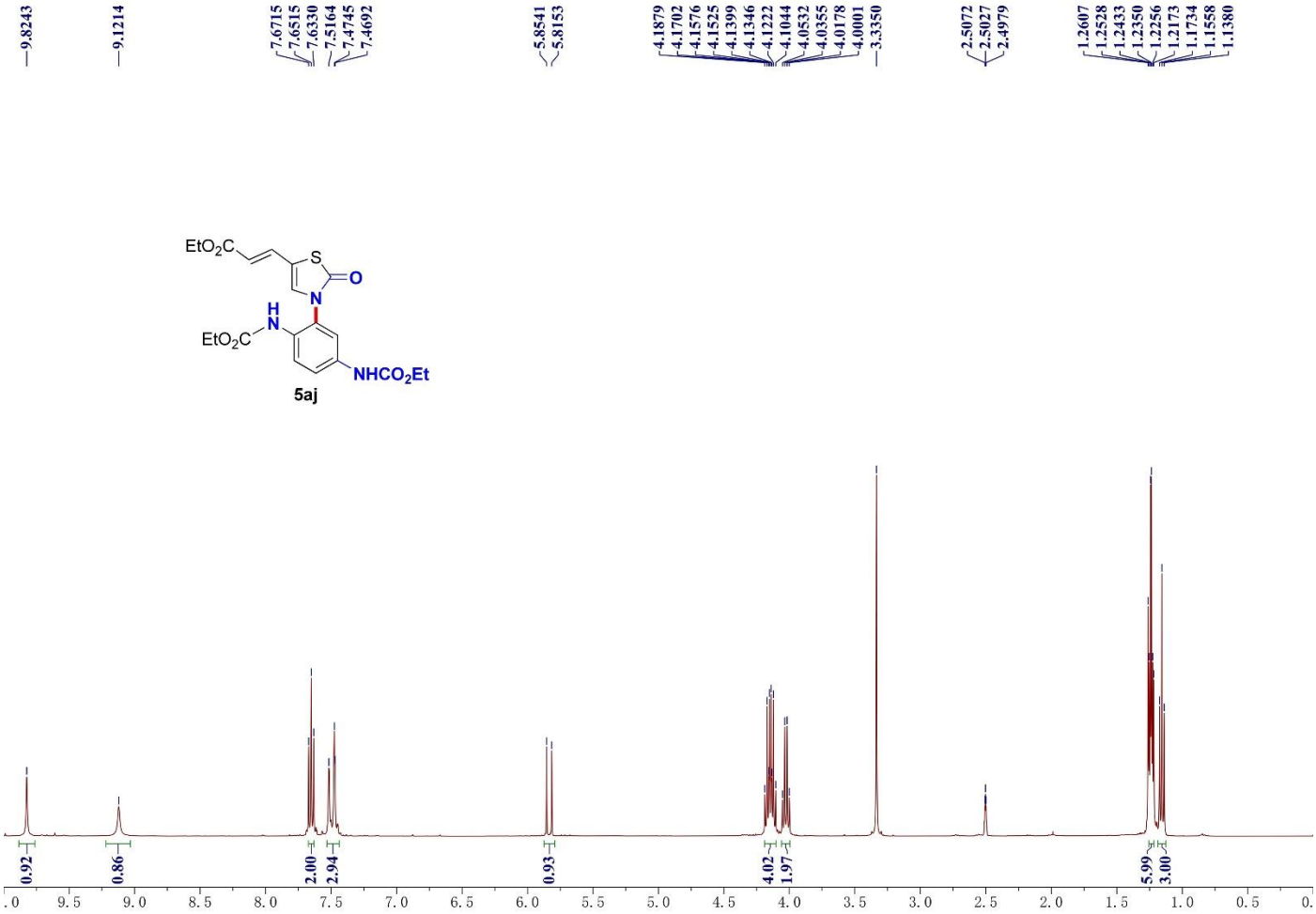
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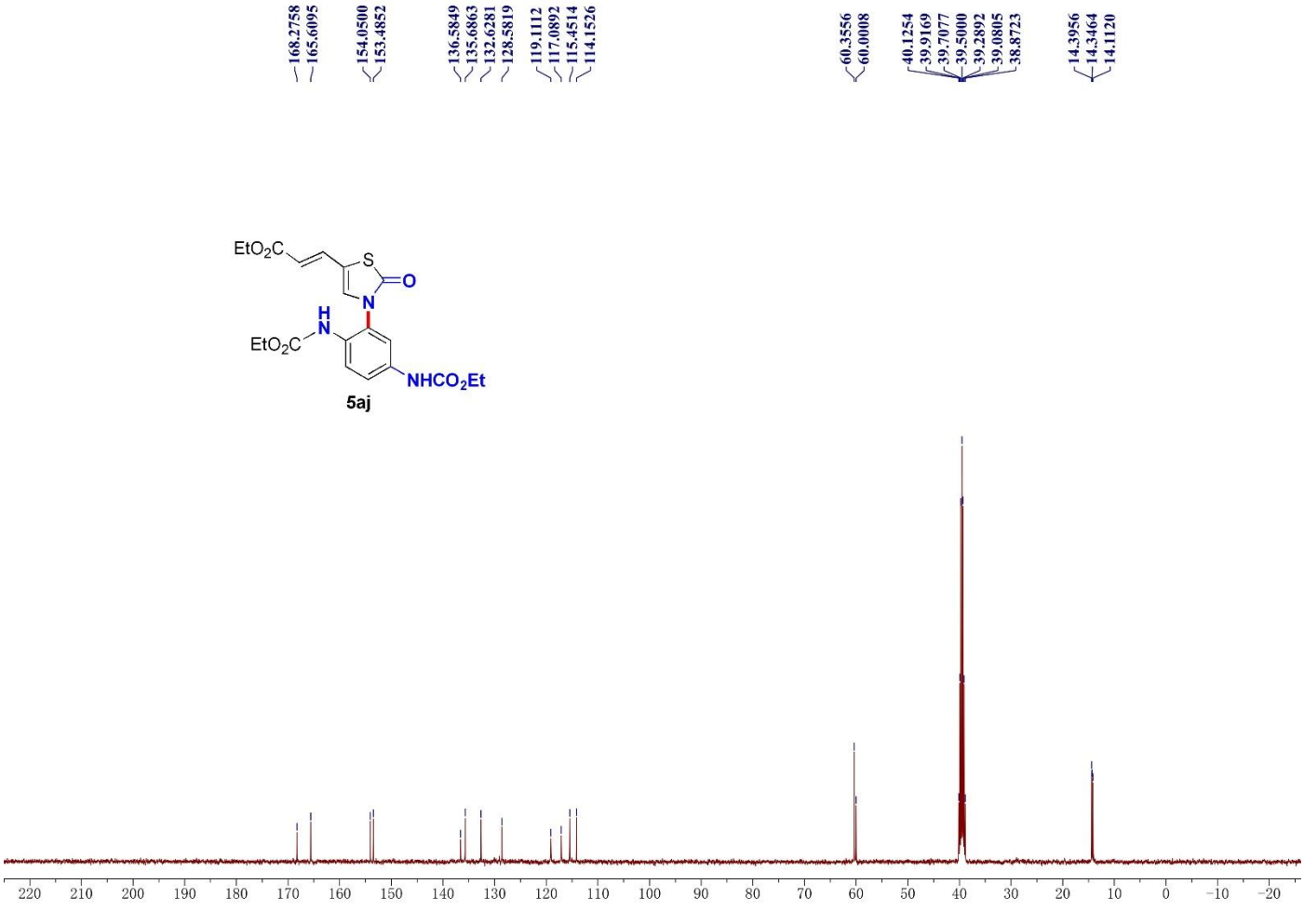
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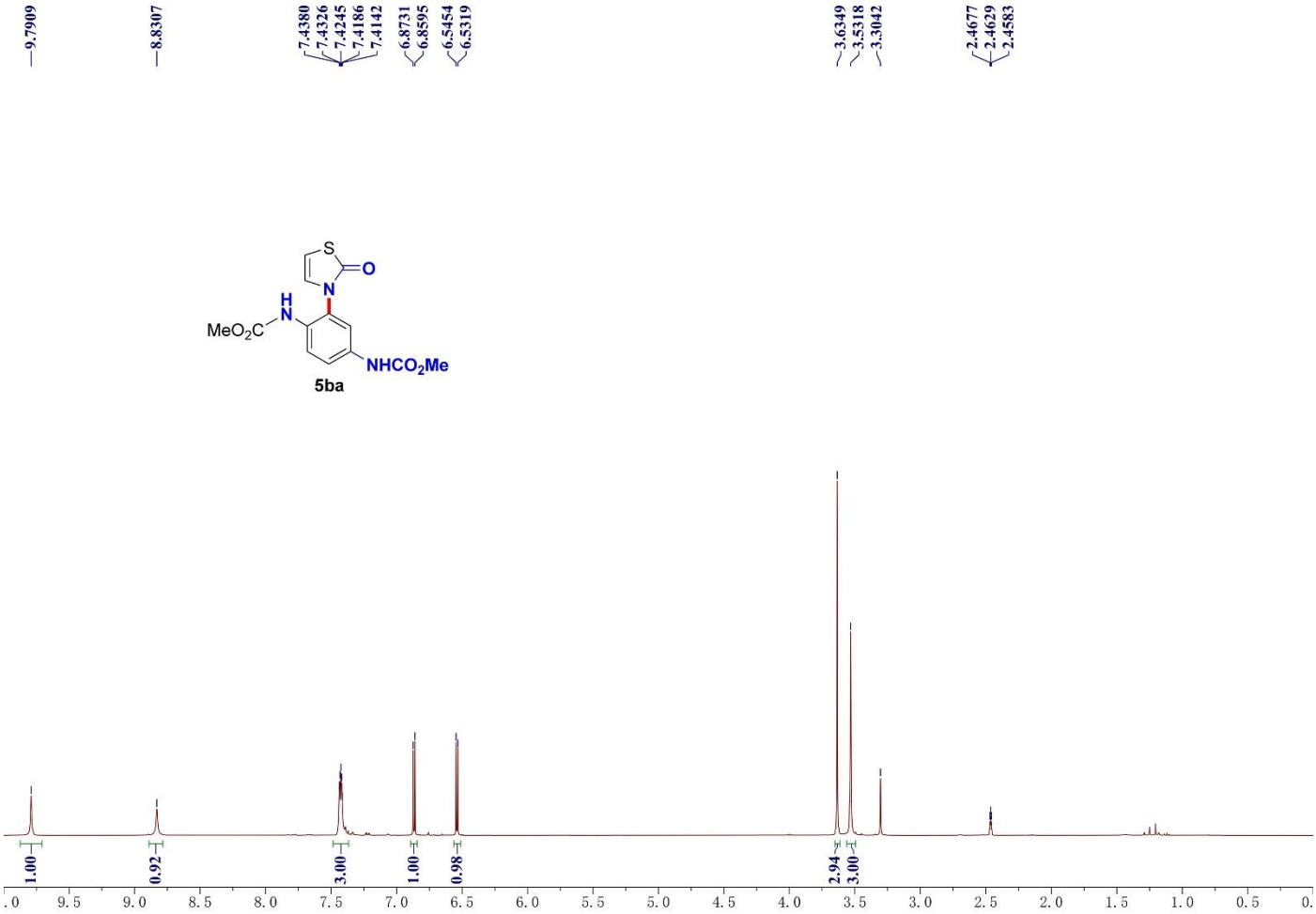
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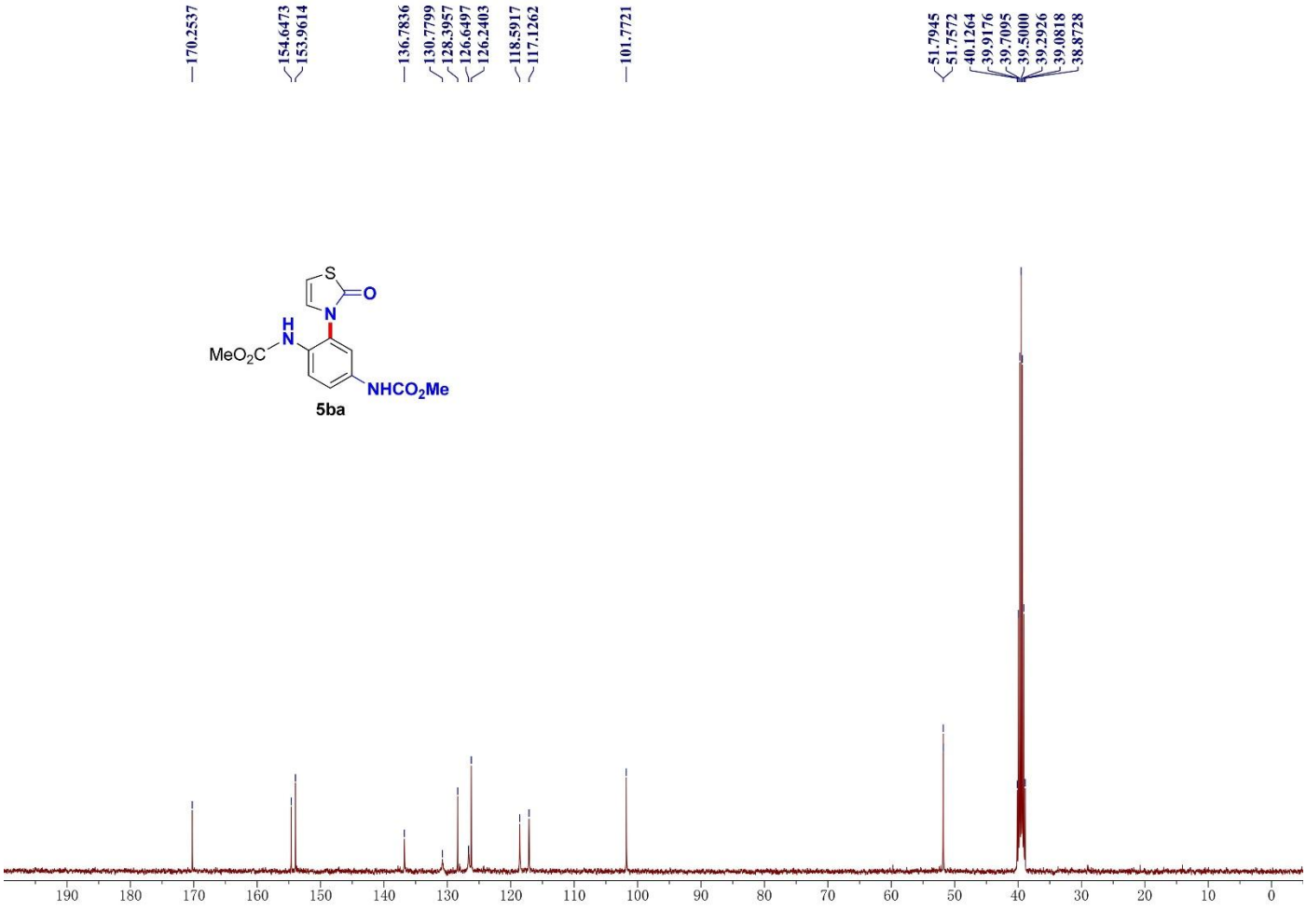
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40.1264
39.9176
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N
H
N
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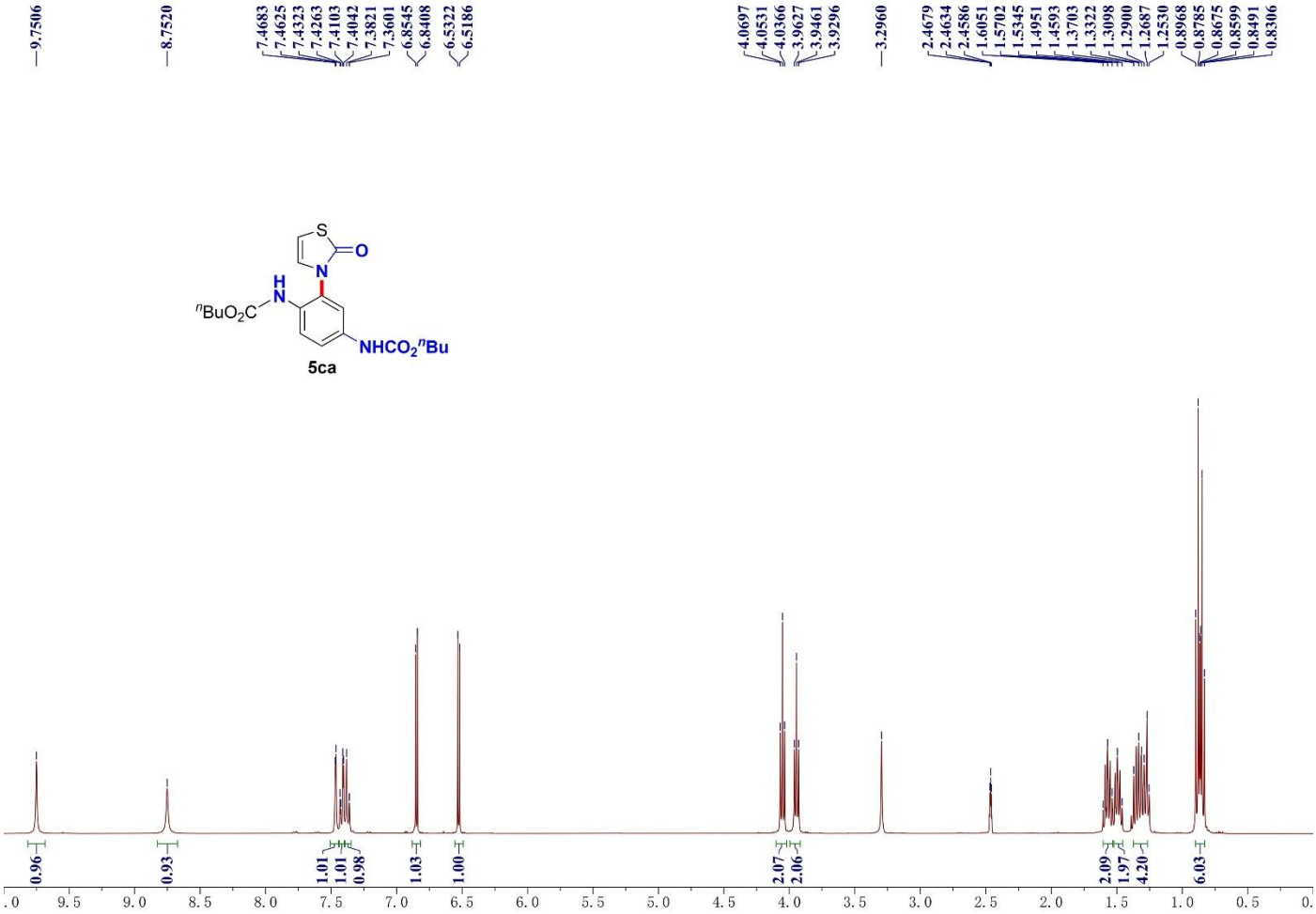
5ba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3.2960
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2.4634
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1.5345
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1.4593
1.3703
1.3322
1.3098
1.2900
1.2687
1.2530
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0.8785
0.8675
0.8599
0.8491
0.8306
S
O
N
H
N
nBuO2C
NHCO2nBu
5ca
0.96
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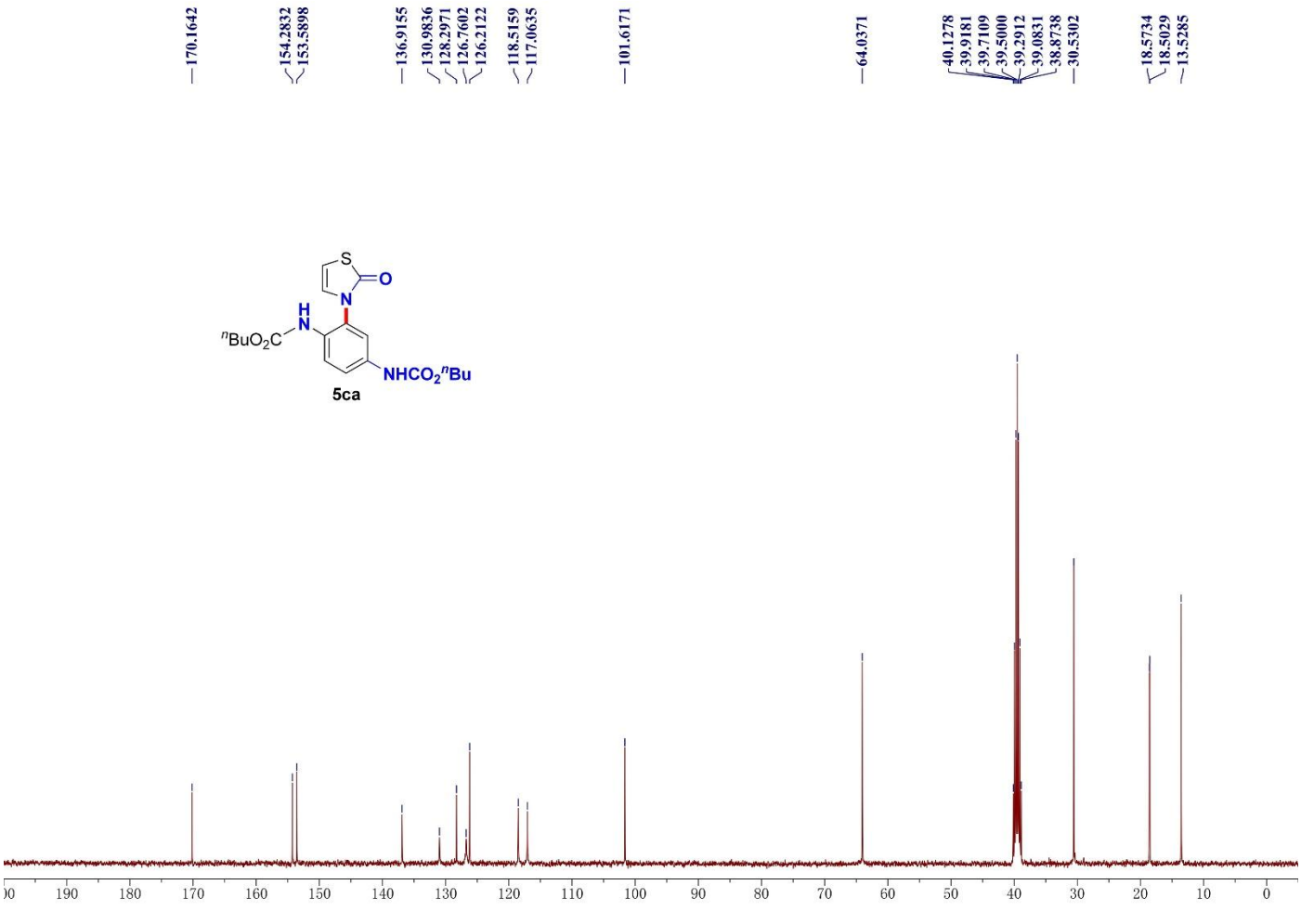
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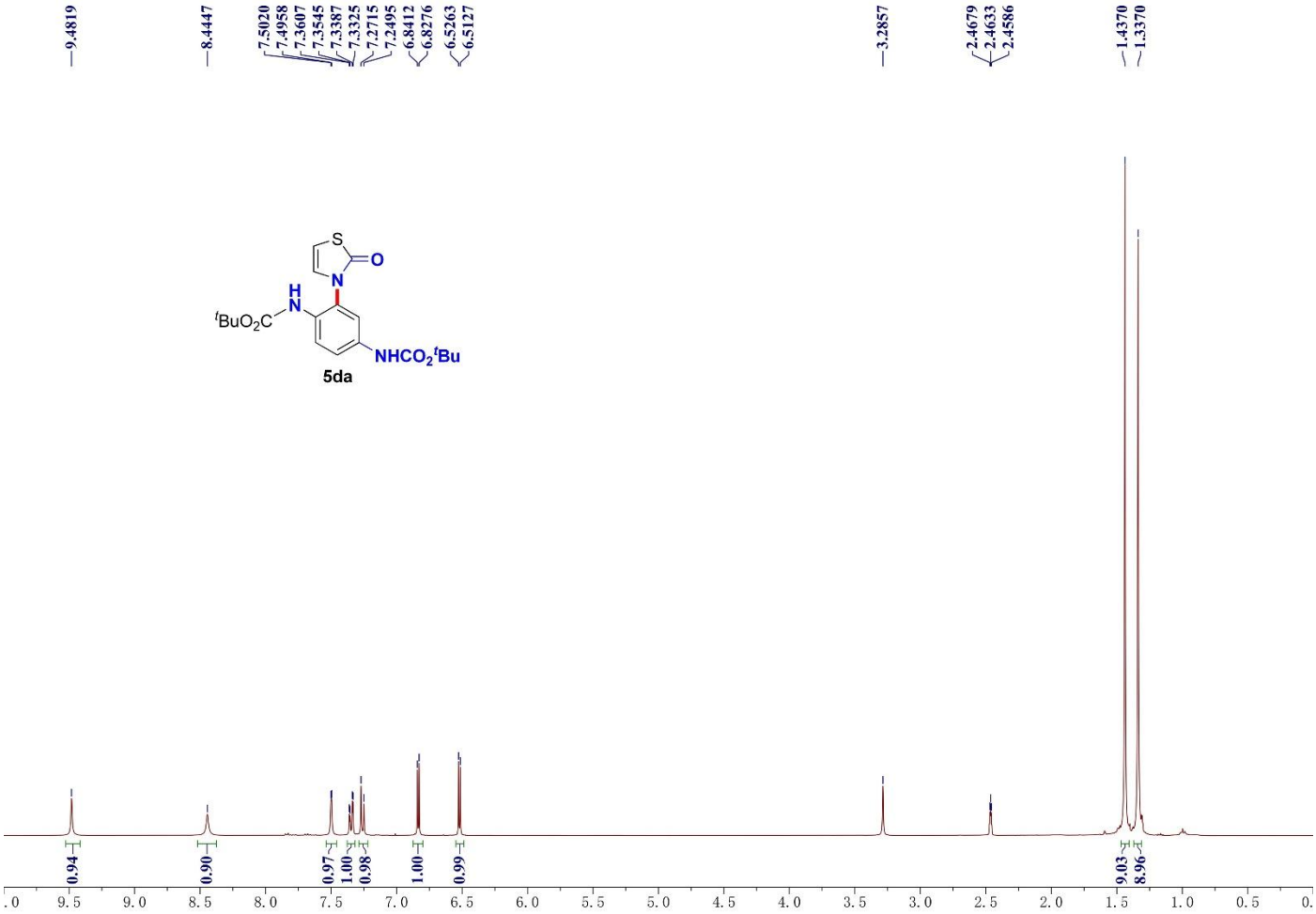
N
H
N
nBuO2C
NHCO2nBu
5ca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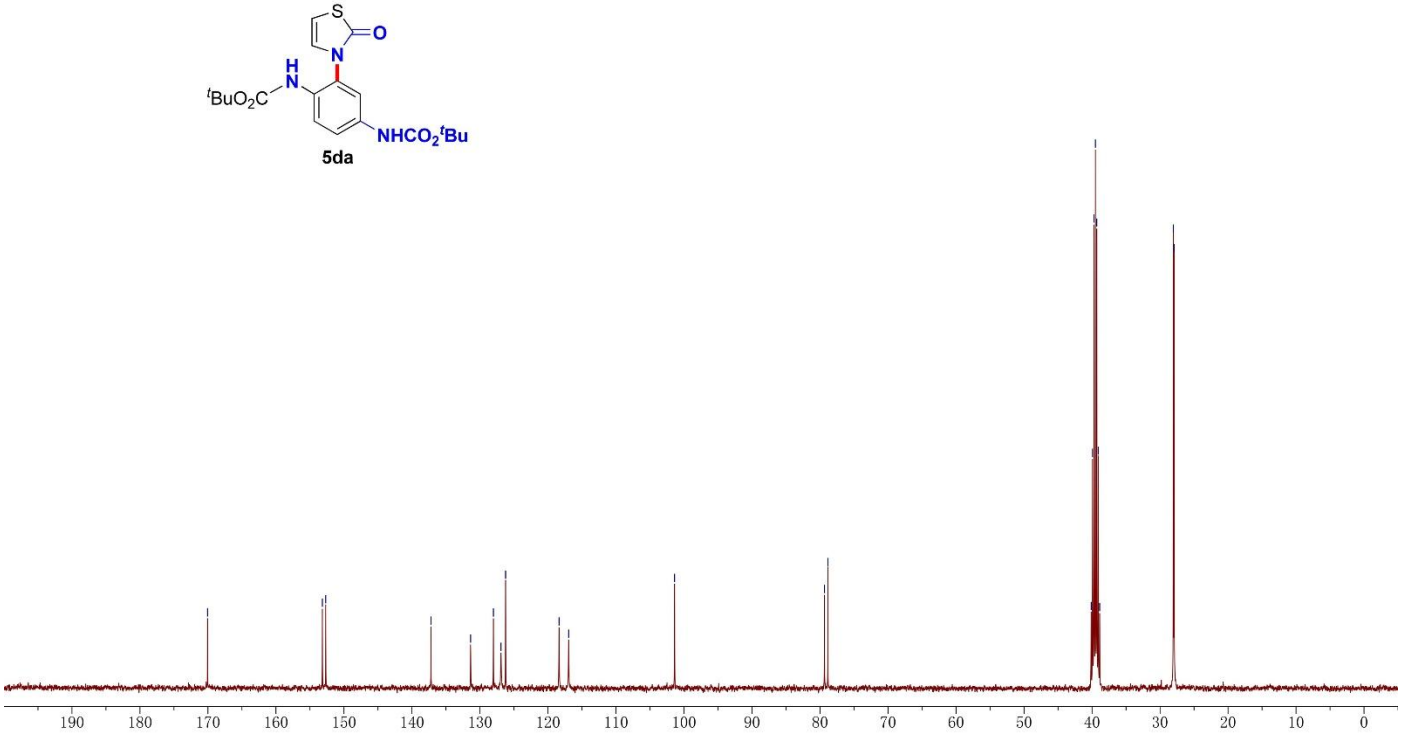
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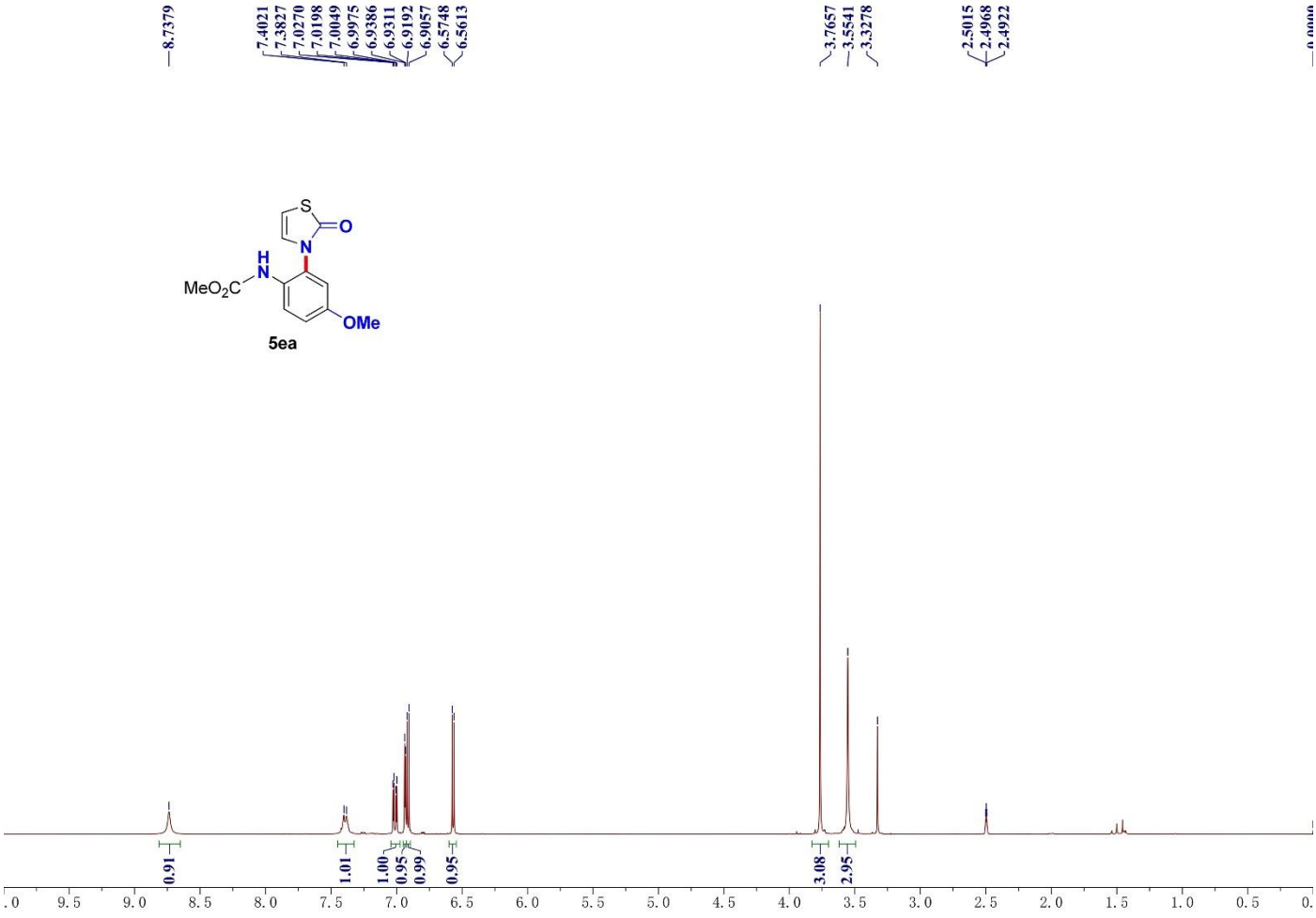
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6.5748
6.5613
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3.5541
3.3278
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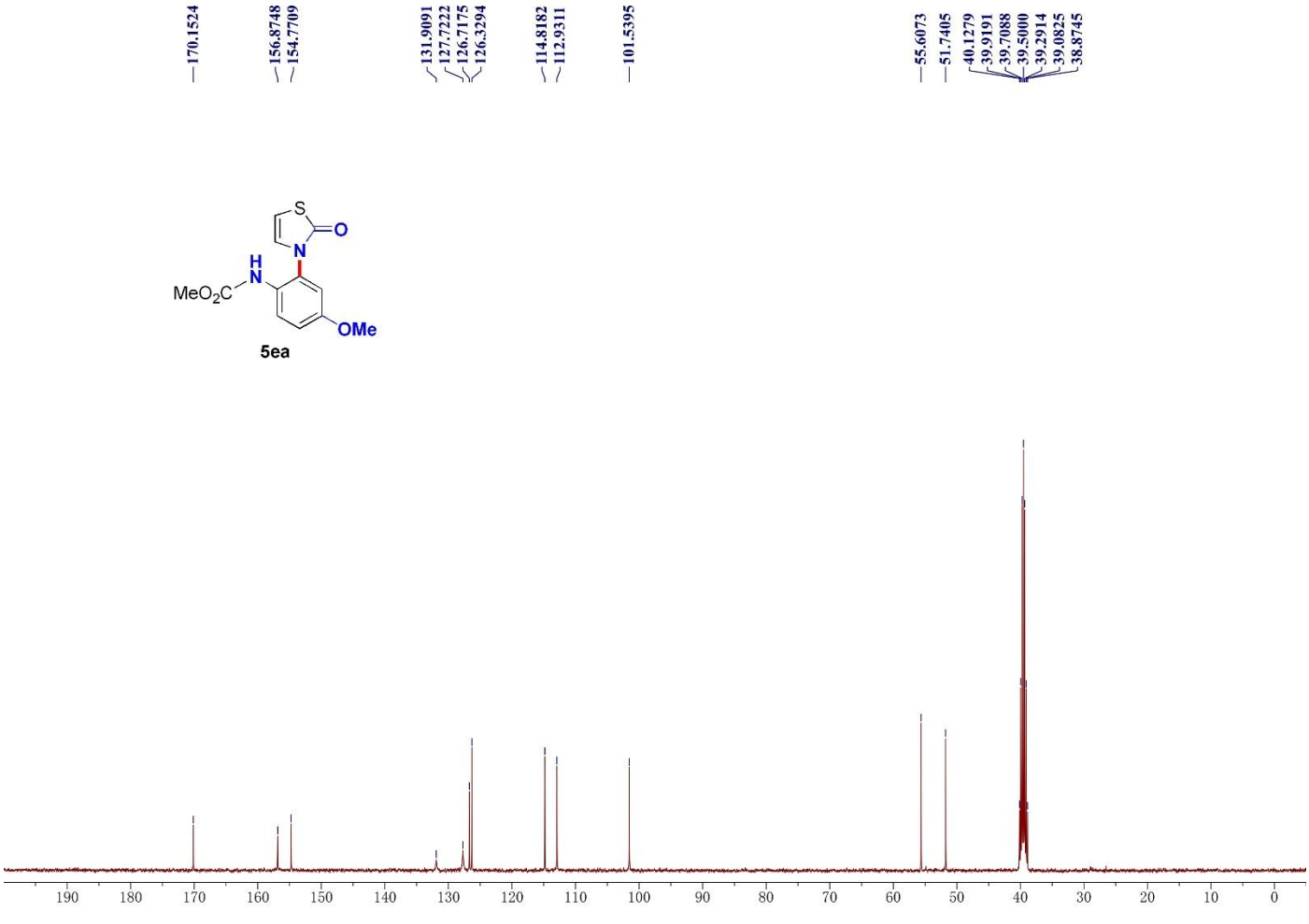
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39.0825
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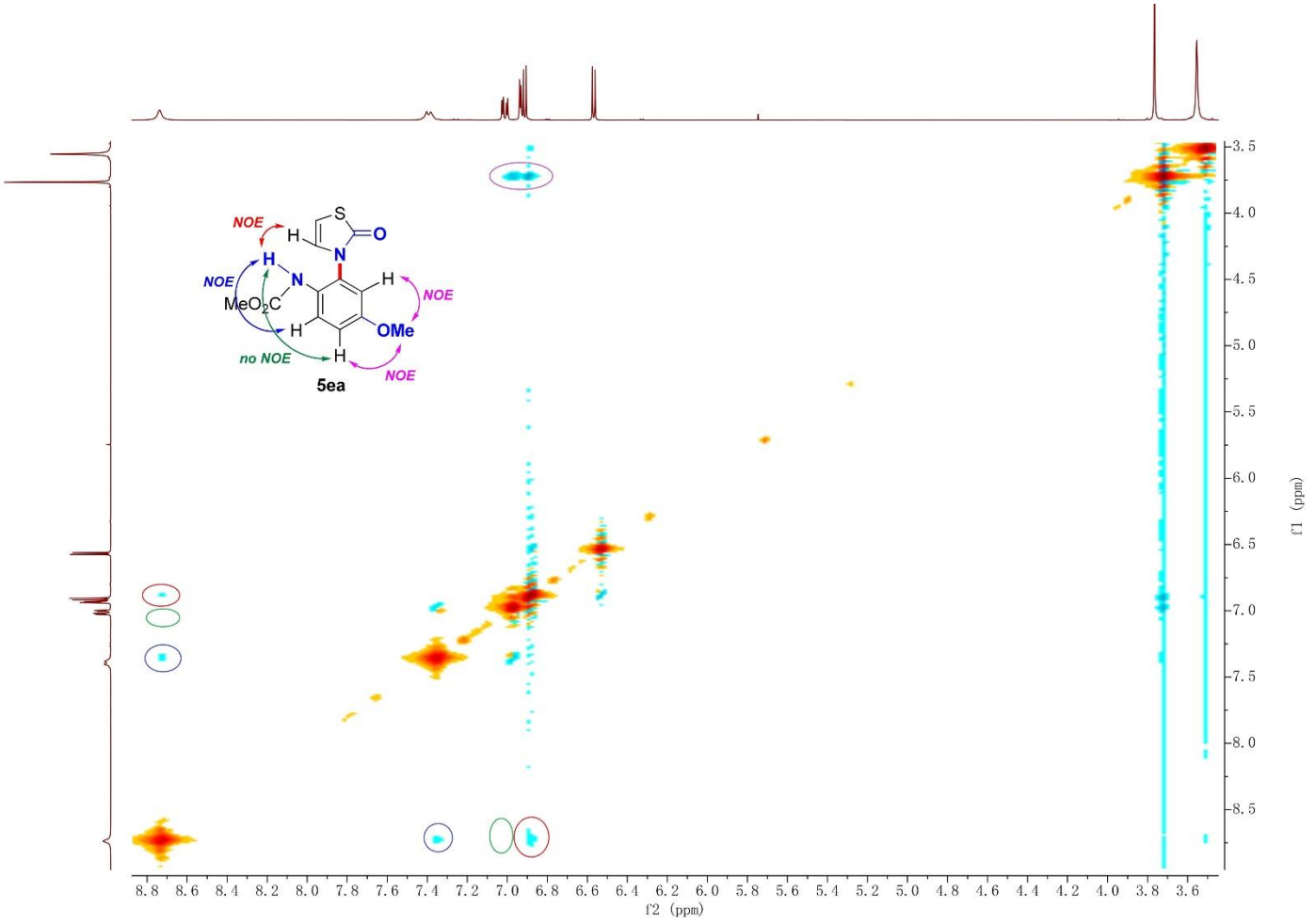
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NOE
no NOE
NOE
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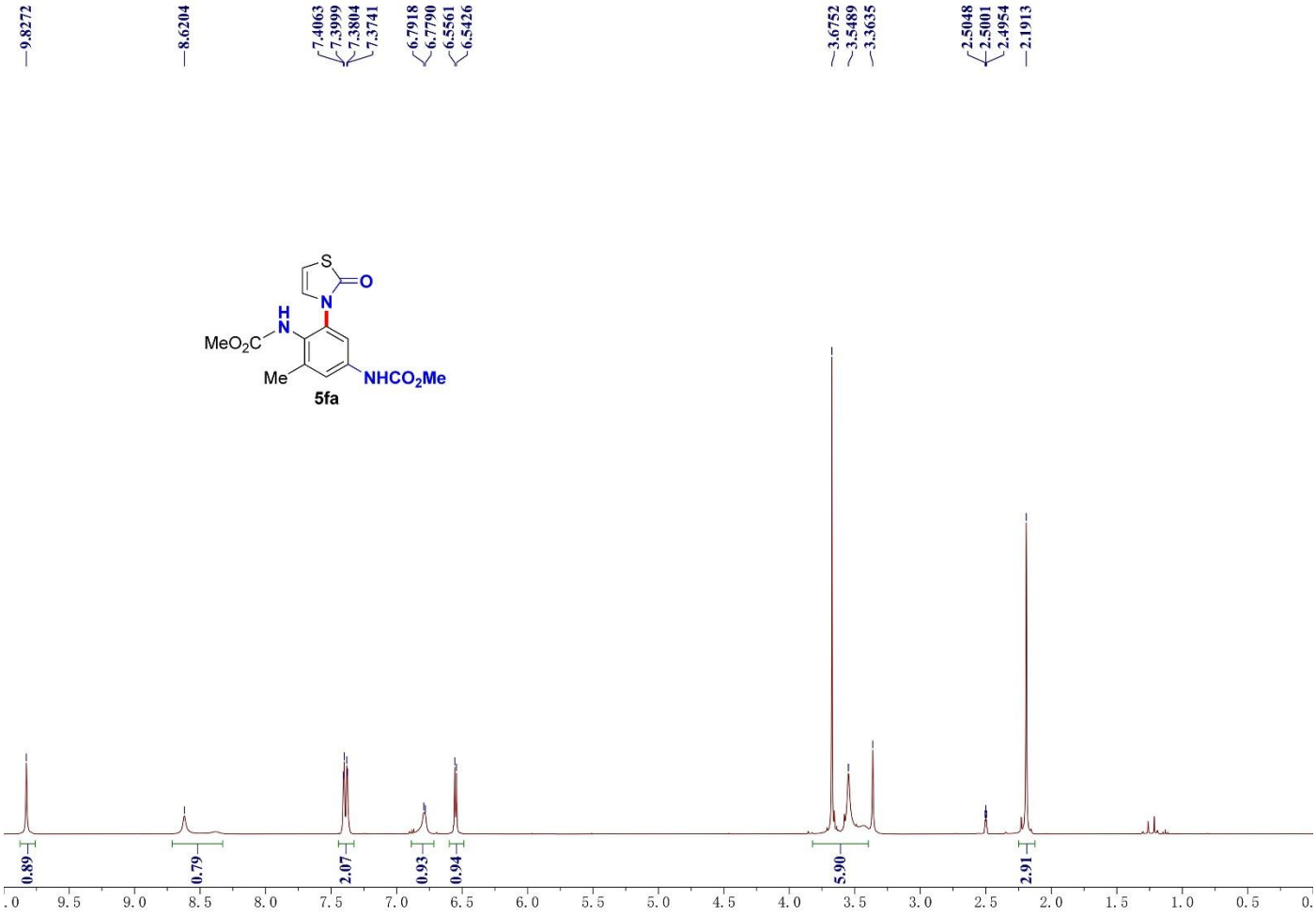
f2 (ppm)
f1 (ppm)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3.6752
3.5489
3.3635
2.5048
2.5001
2.4954
2.1913
0.89
0.79
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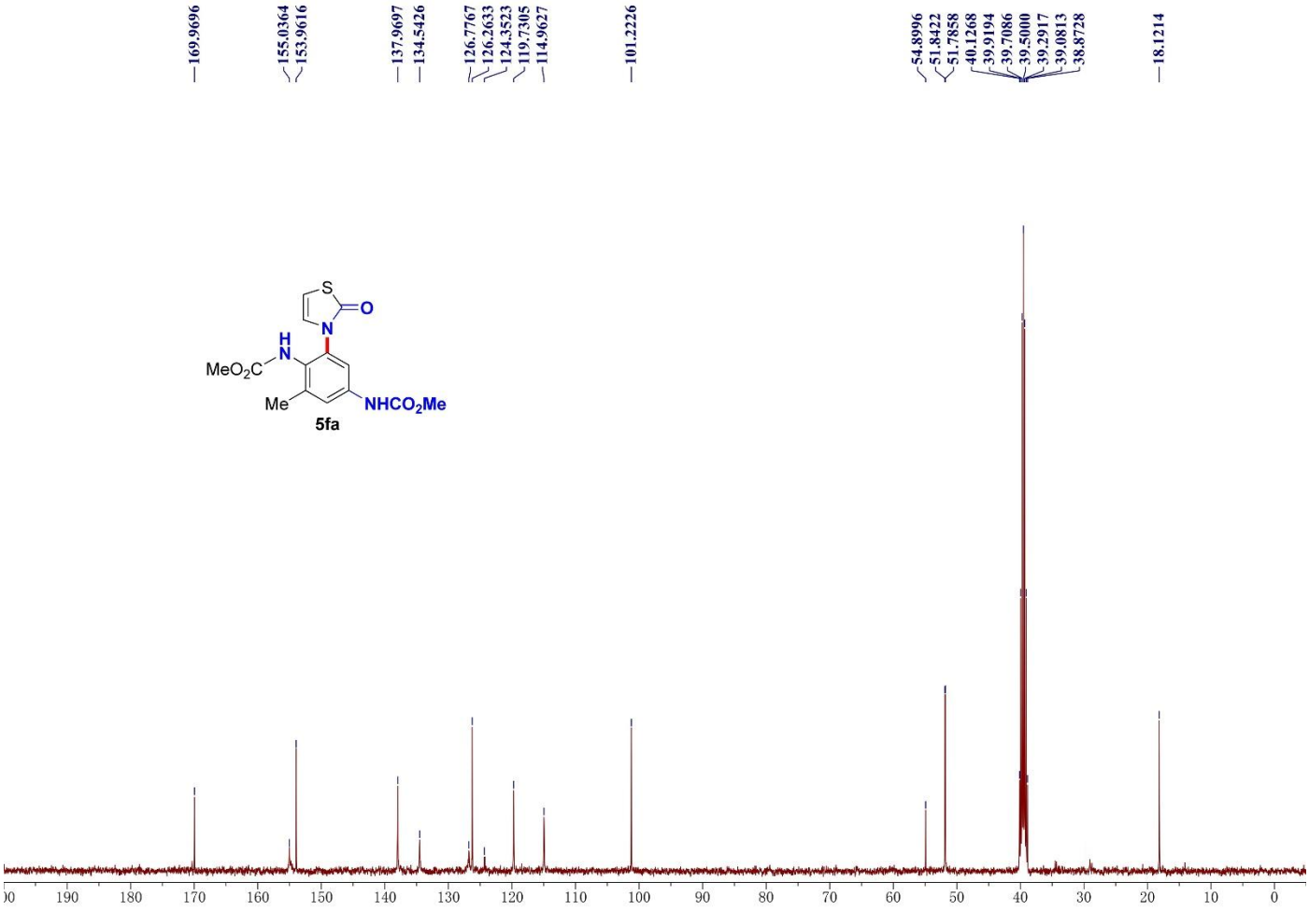
0.94
5.90
2.91
MeO2C
H
N
S
O
N
Me
NHCO2Me
5fa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MeO2C
H
N
S
O
N
Me
NHCO2Me
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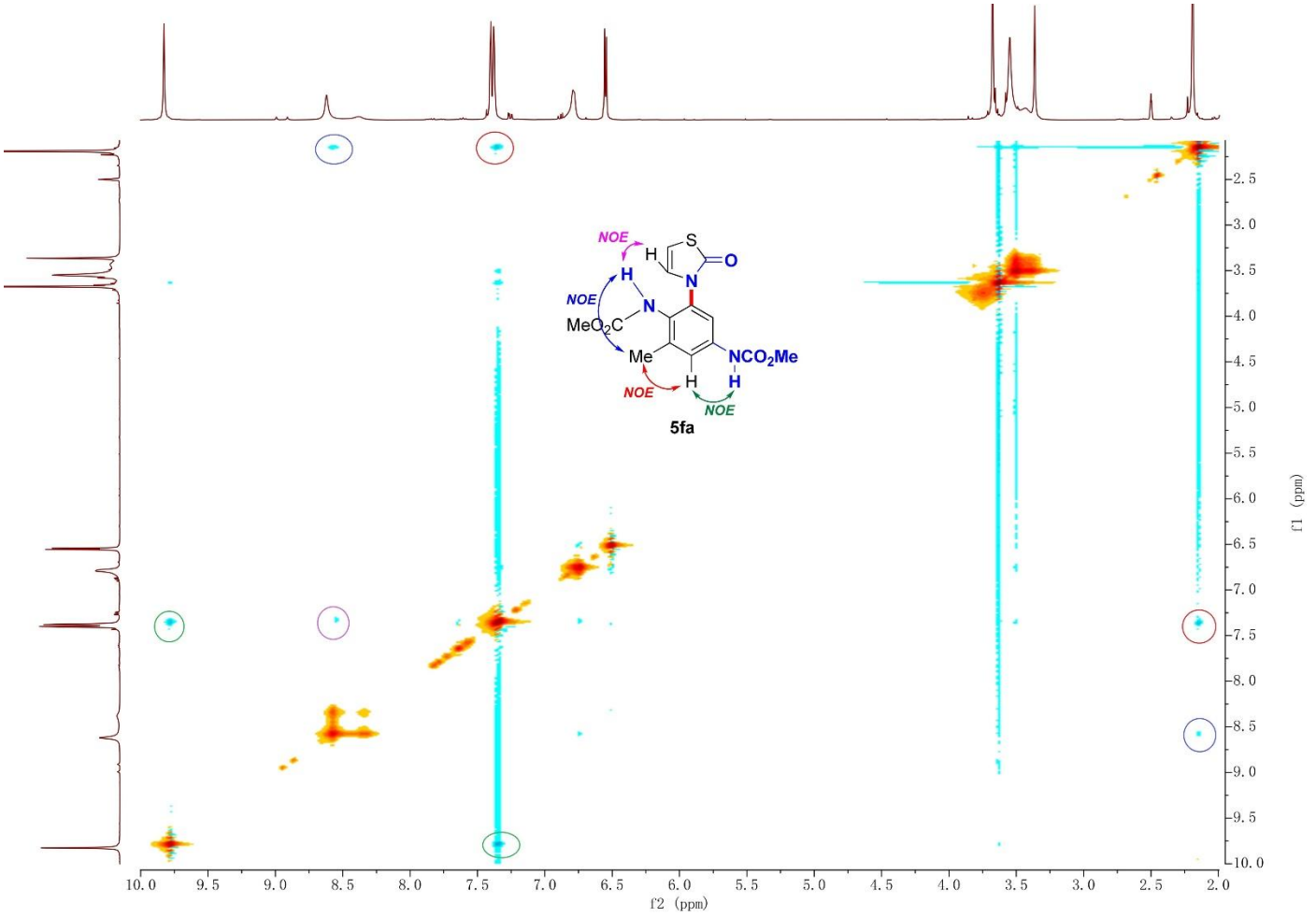
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NOE
NOE
NOE
NOE
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O
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H
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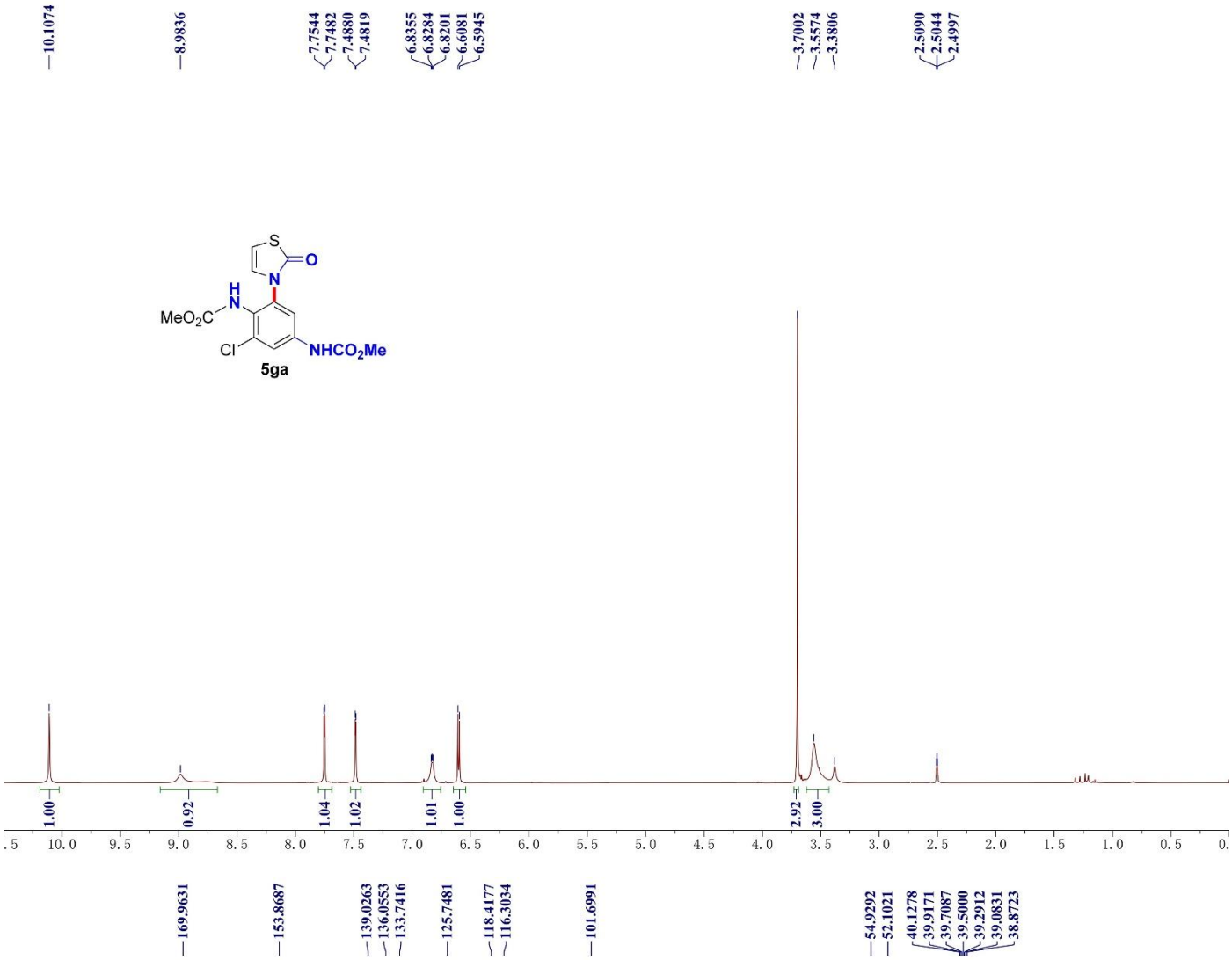
N
MeO2C
Me
H
NCO2Me
H
5fa
f2 (ppm)
f1 (ppm)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3.3806
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1.01
1.00
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3.00
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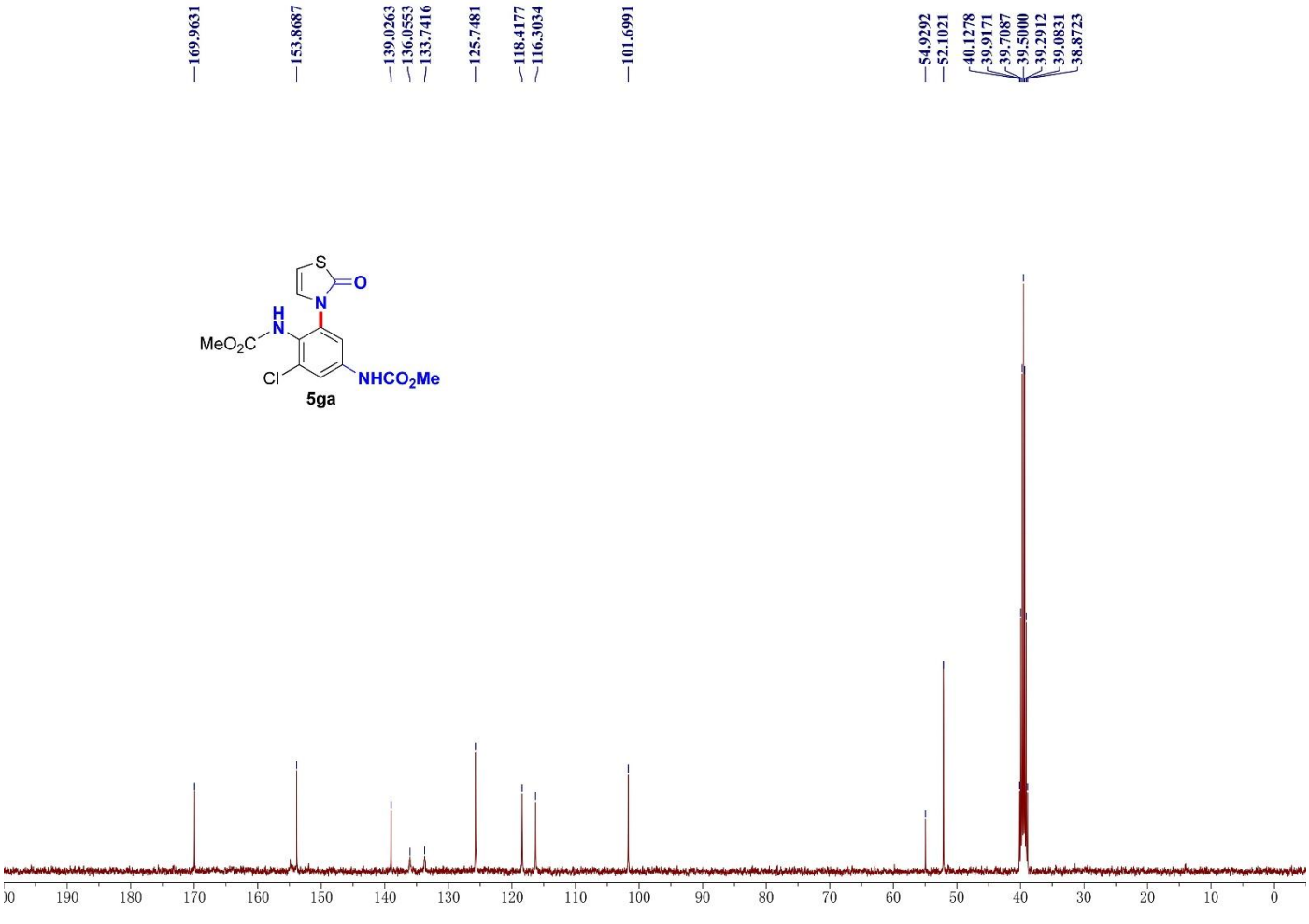
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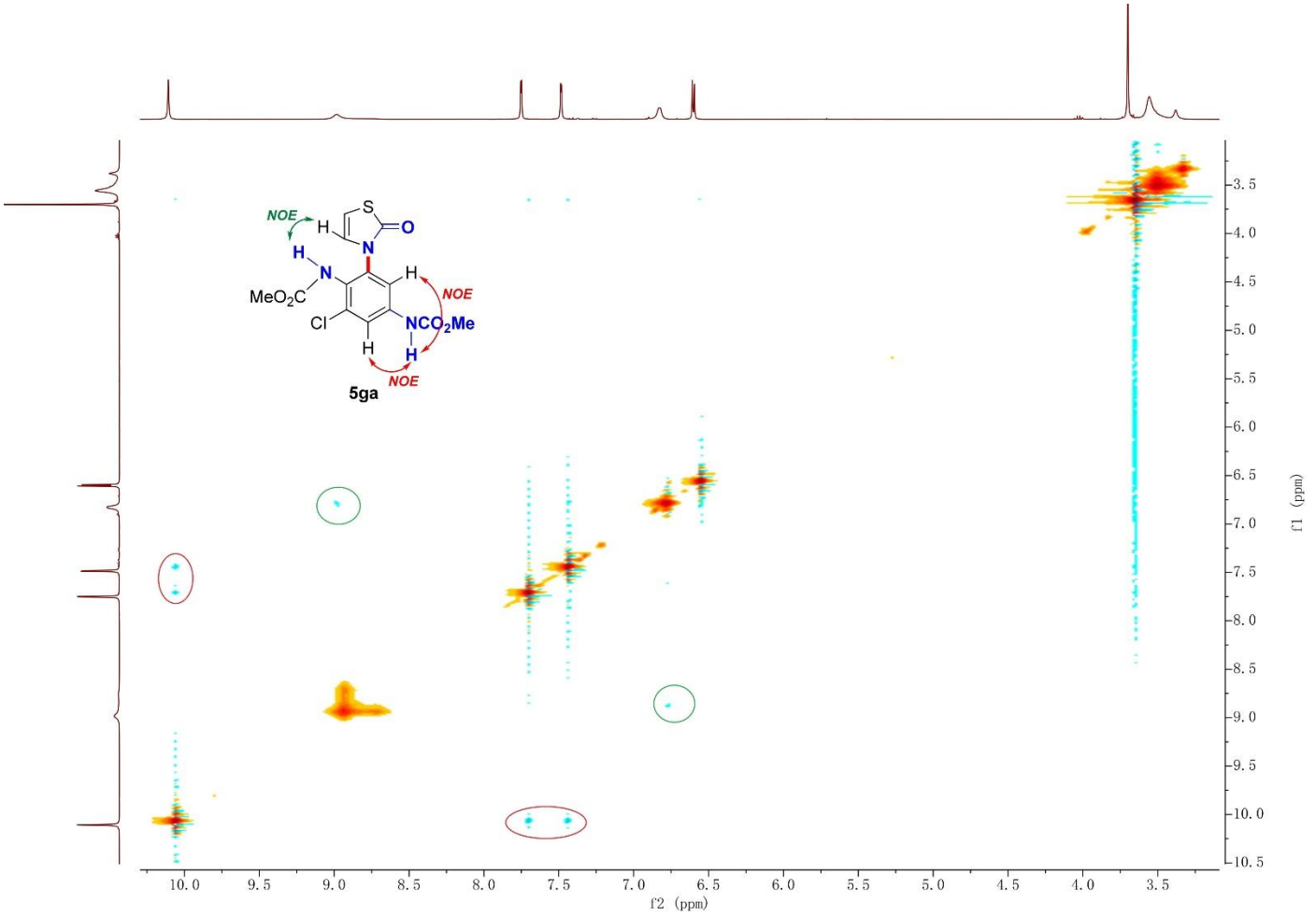
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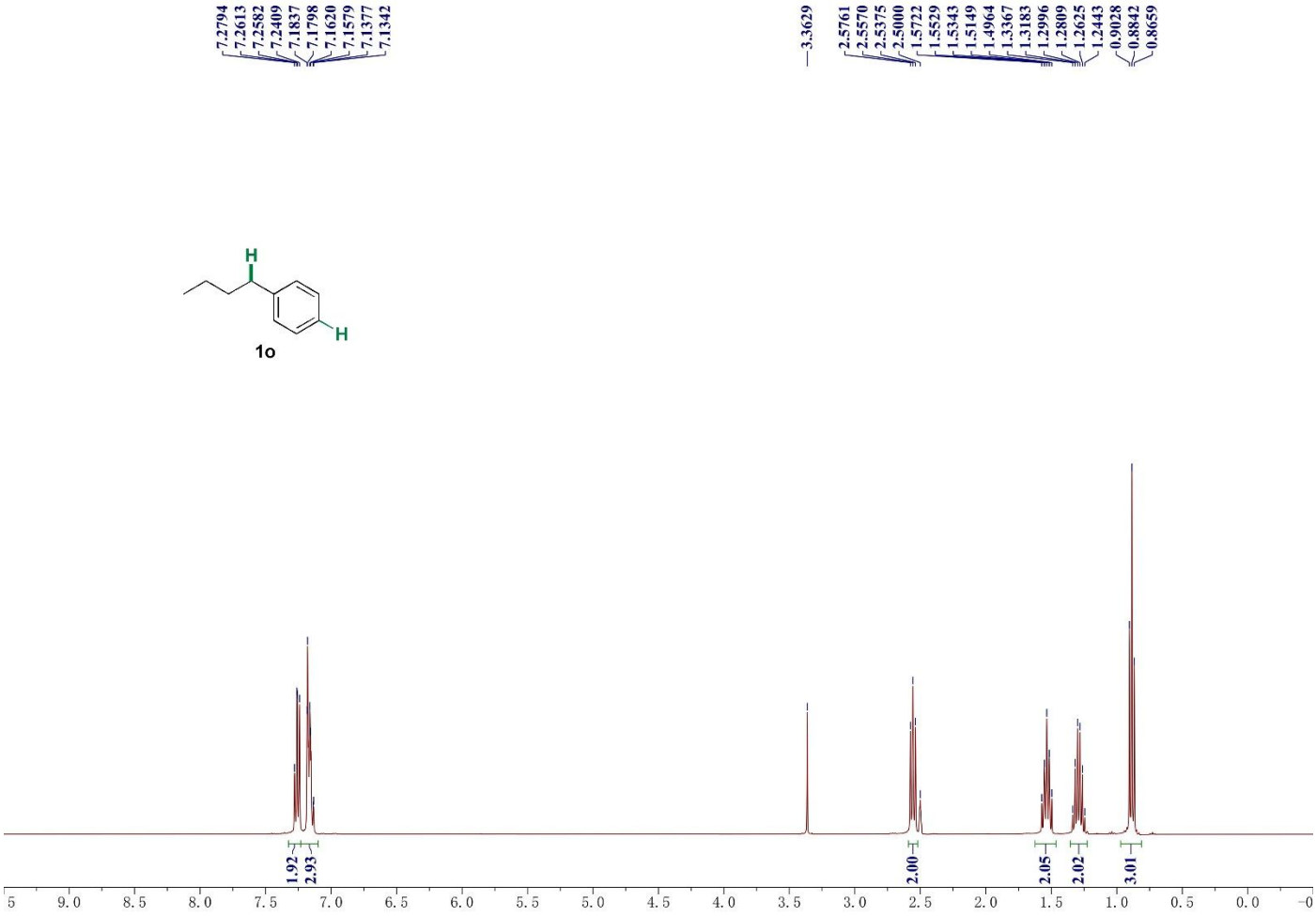
f1 (ppm)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2.5000
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1.5529
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1.5149
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1.2809
1.2625
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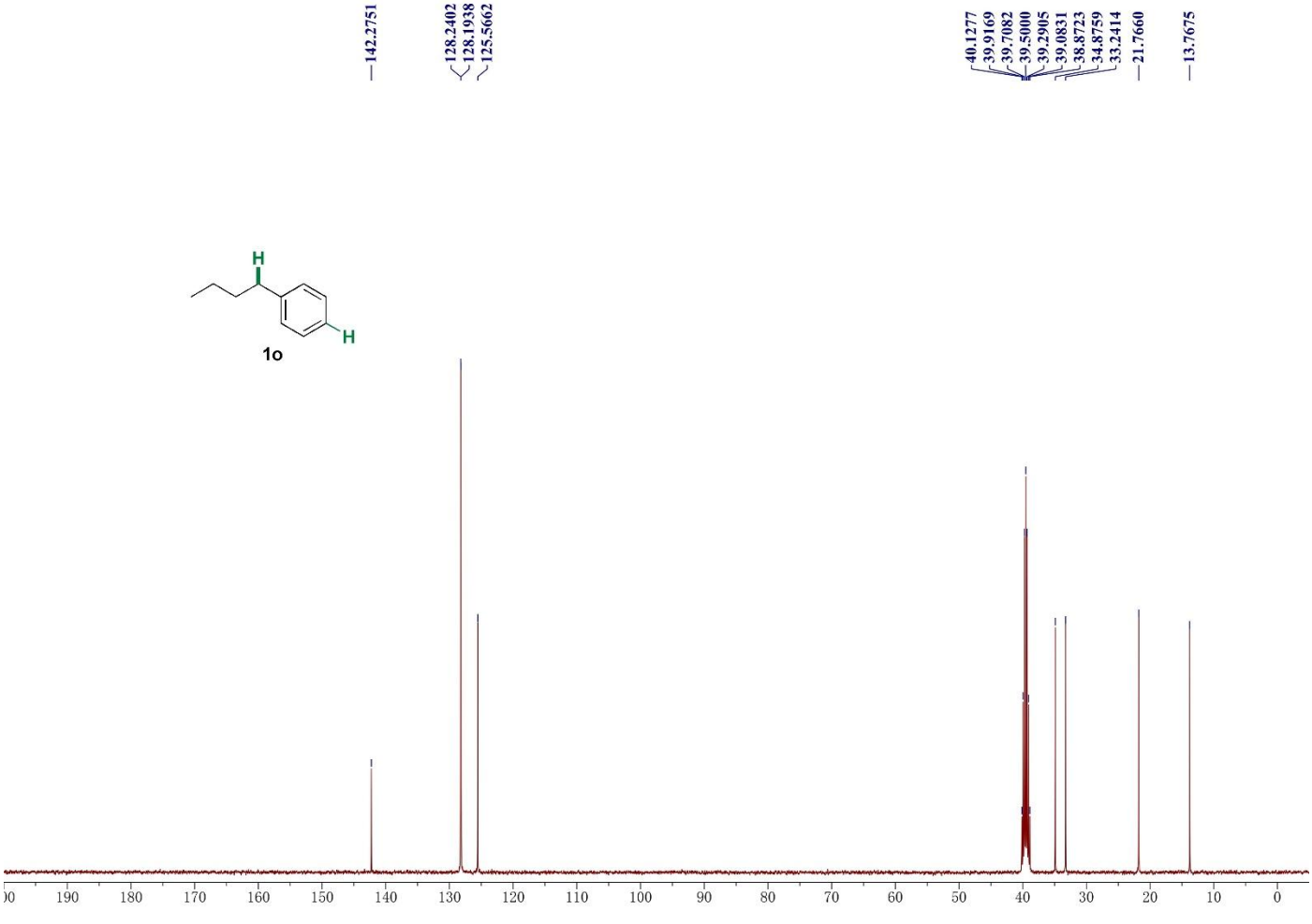
2.02
3.01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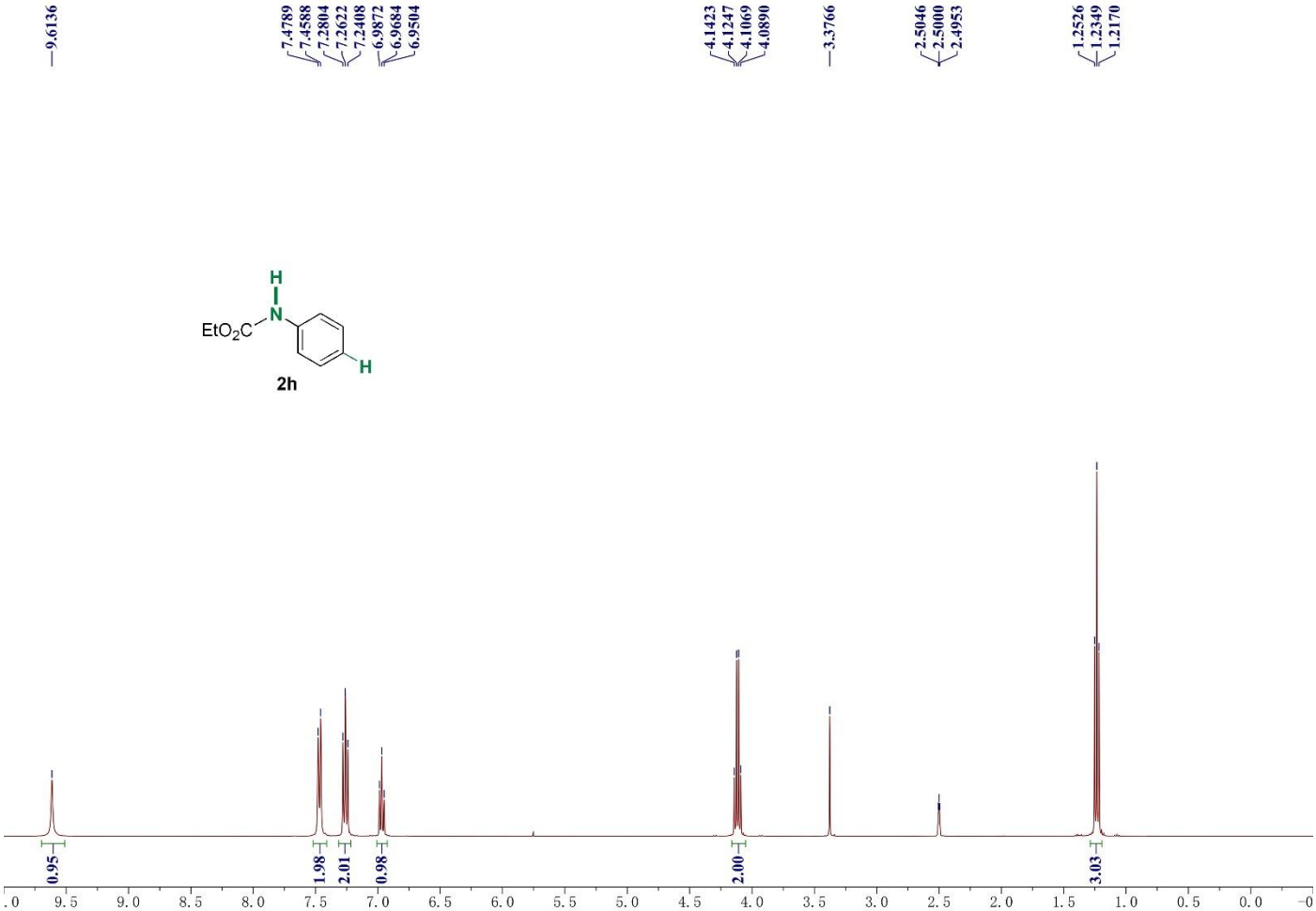
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EtO2C
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H
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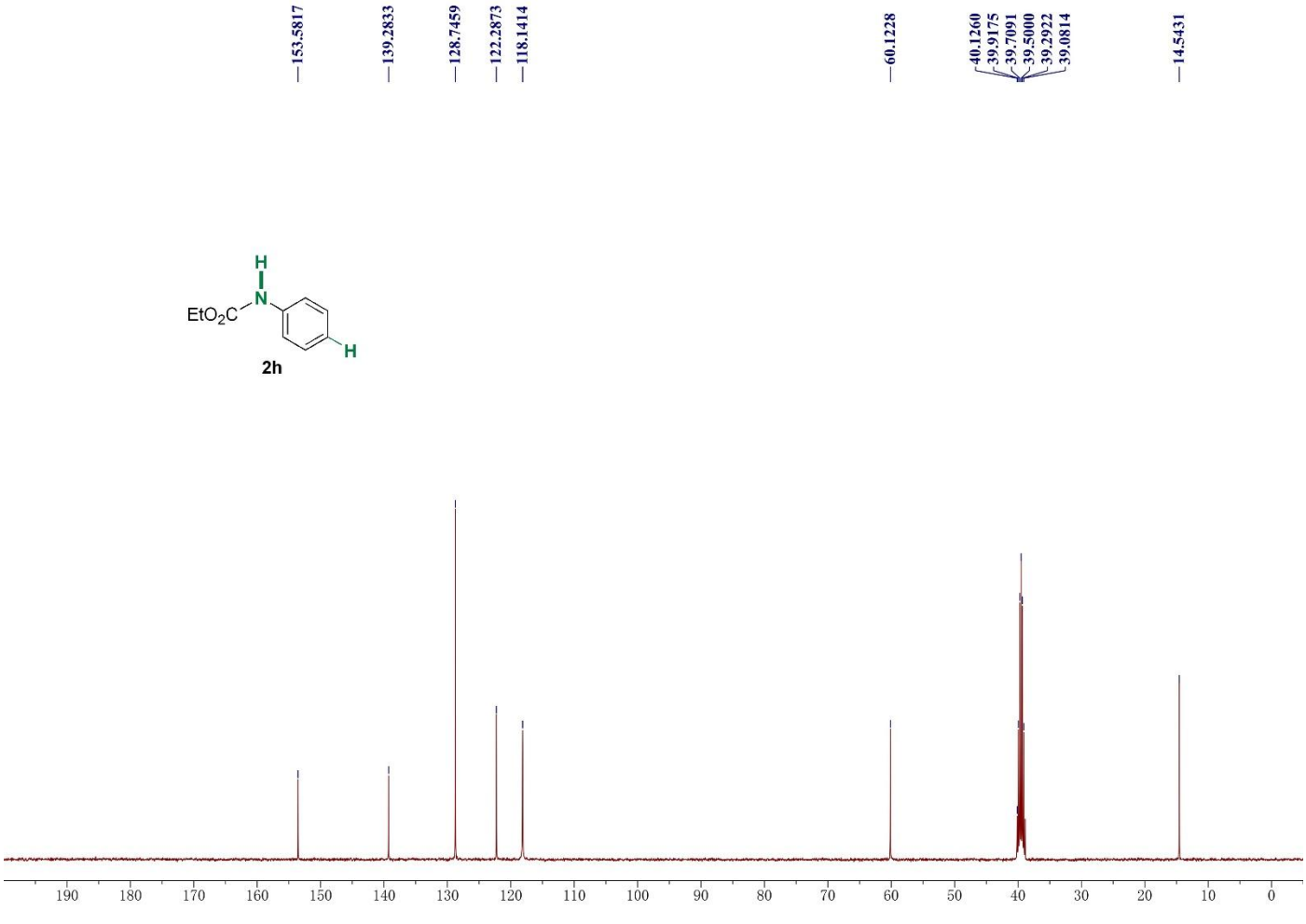
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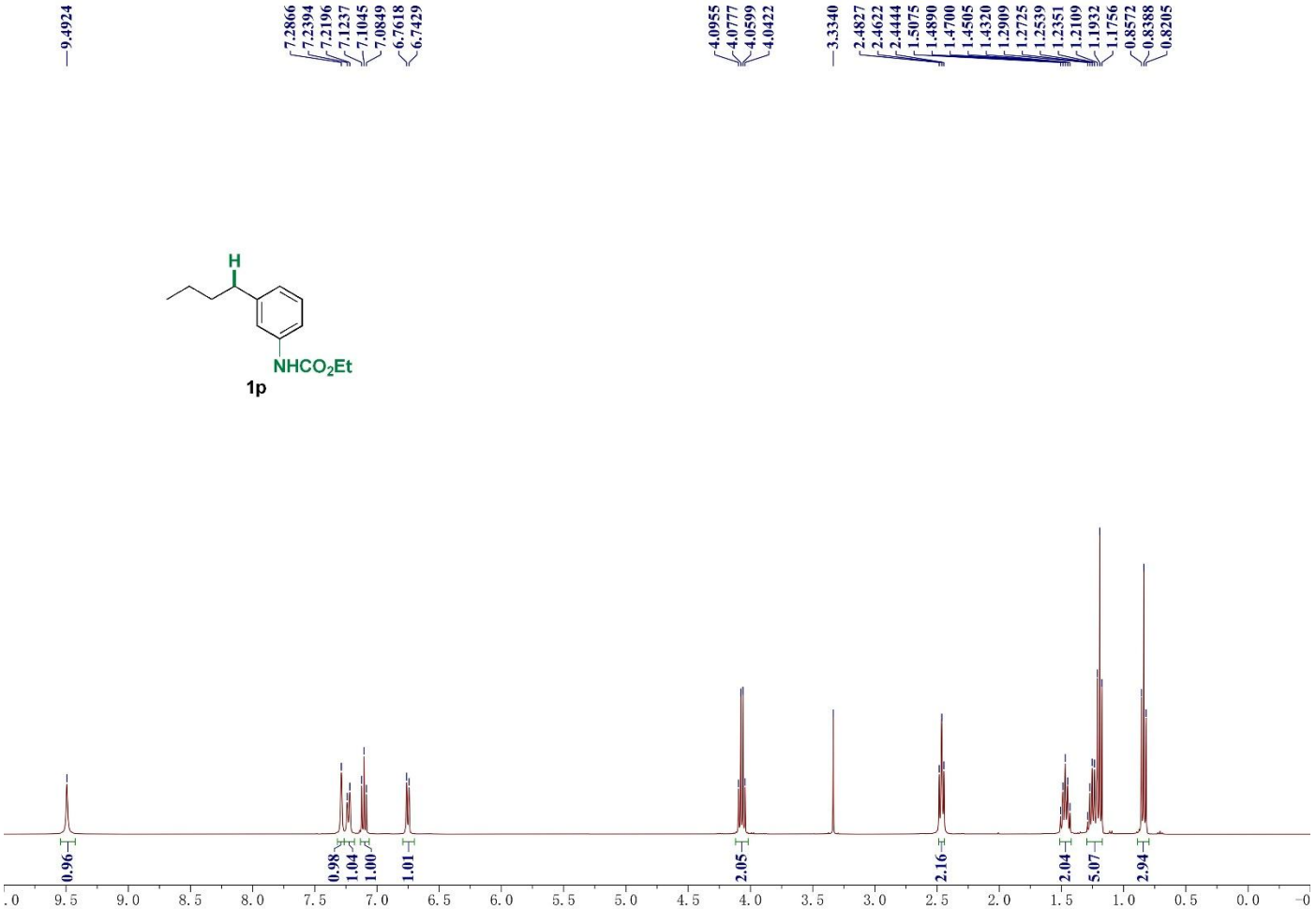
H
2h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0.8388
0.8205
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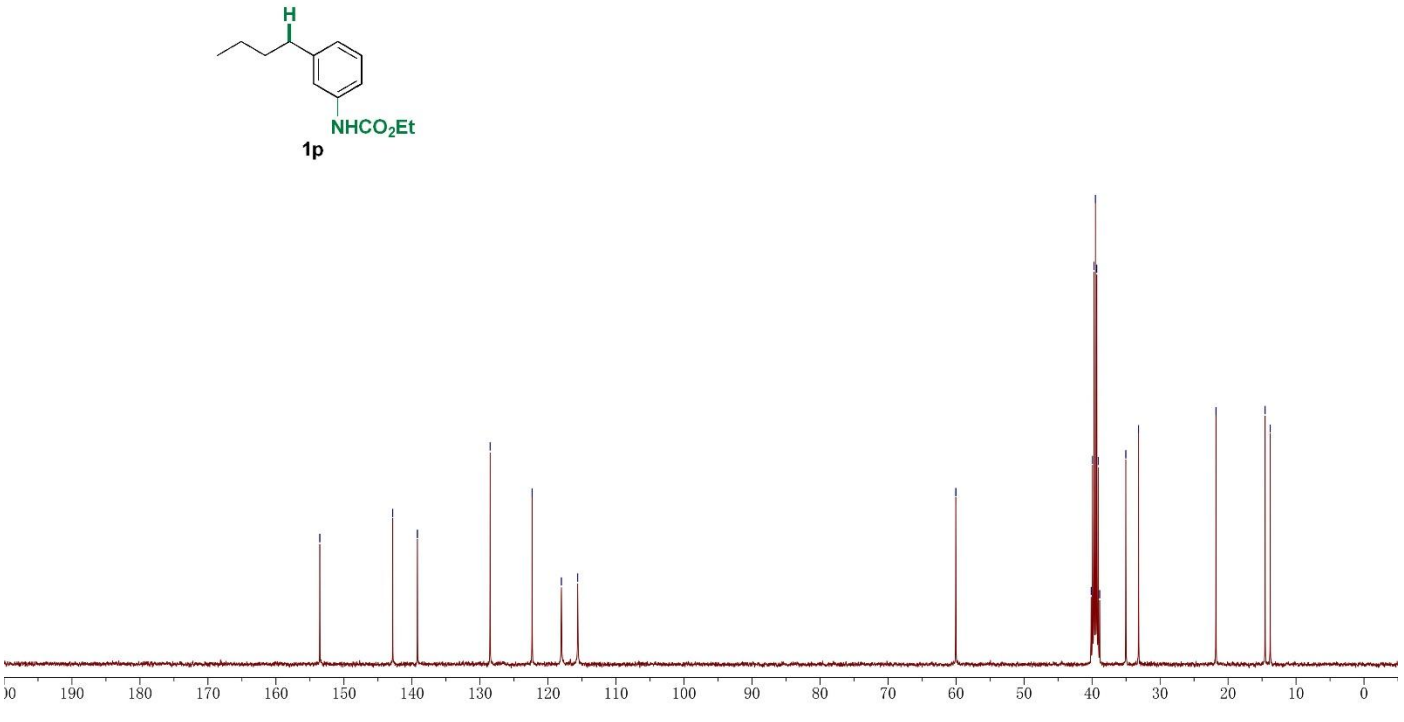
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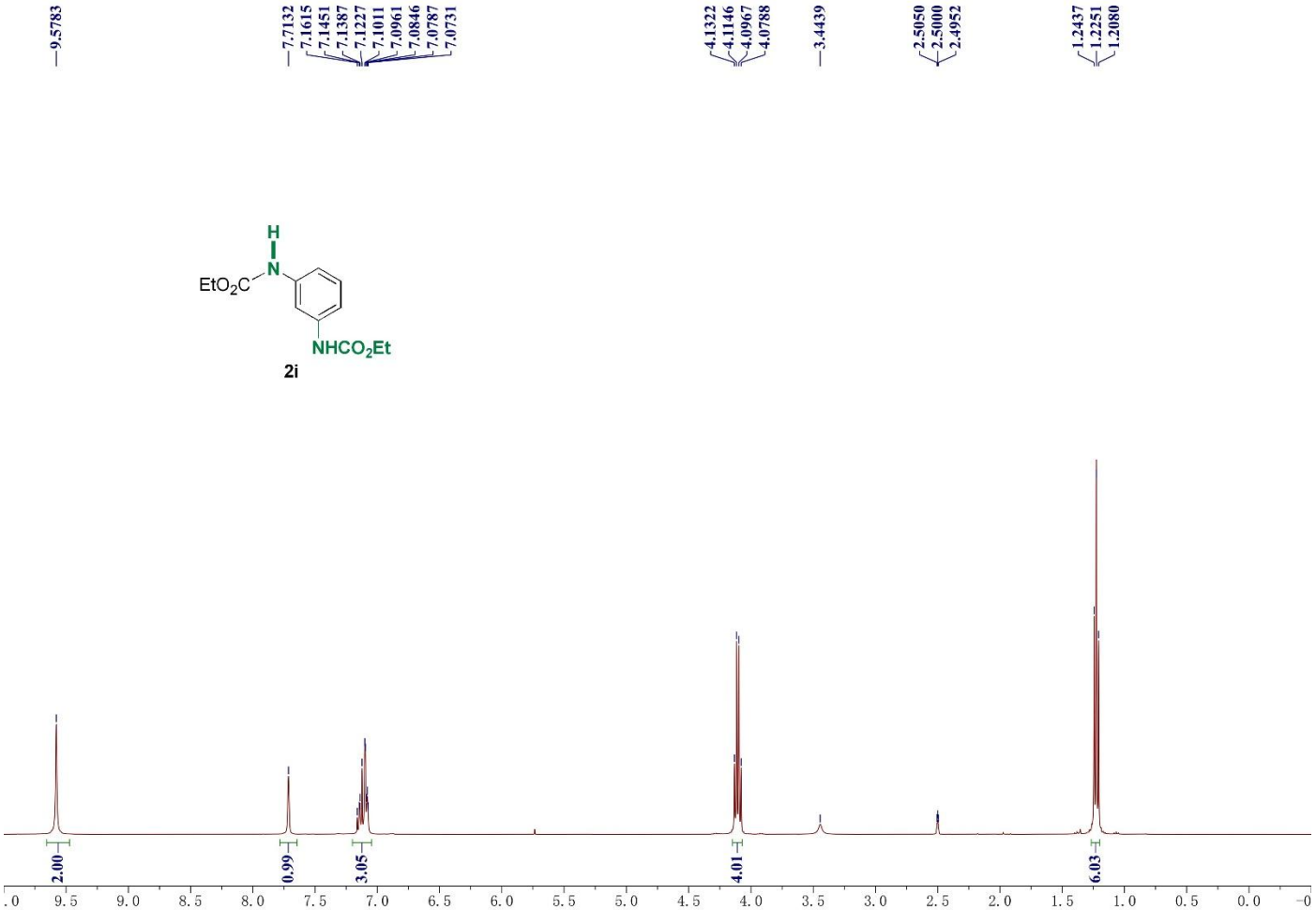
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NHCO2Et
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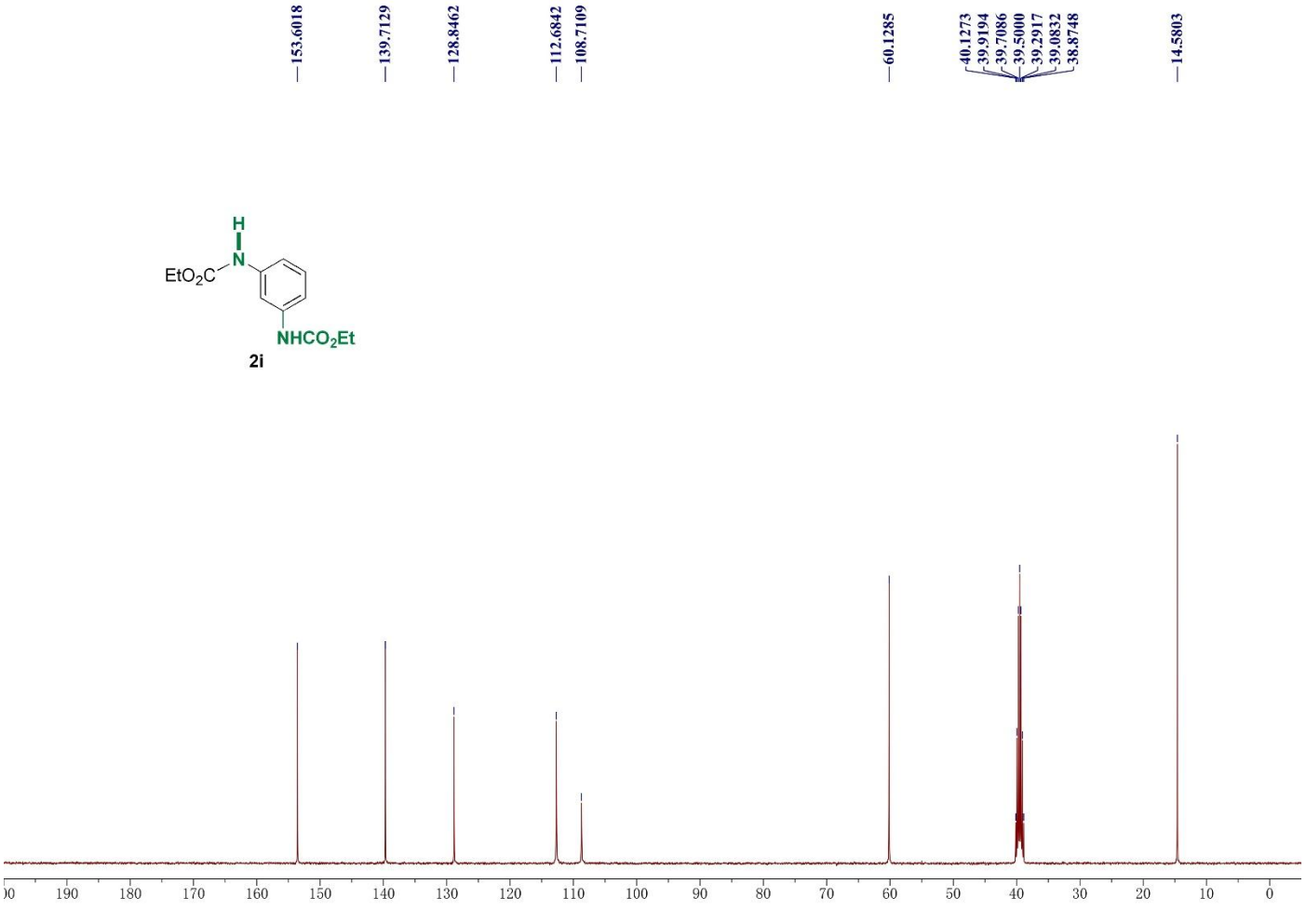
6.03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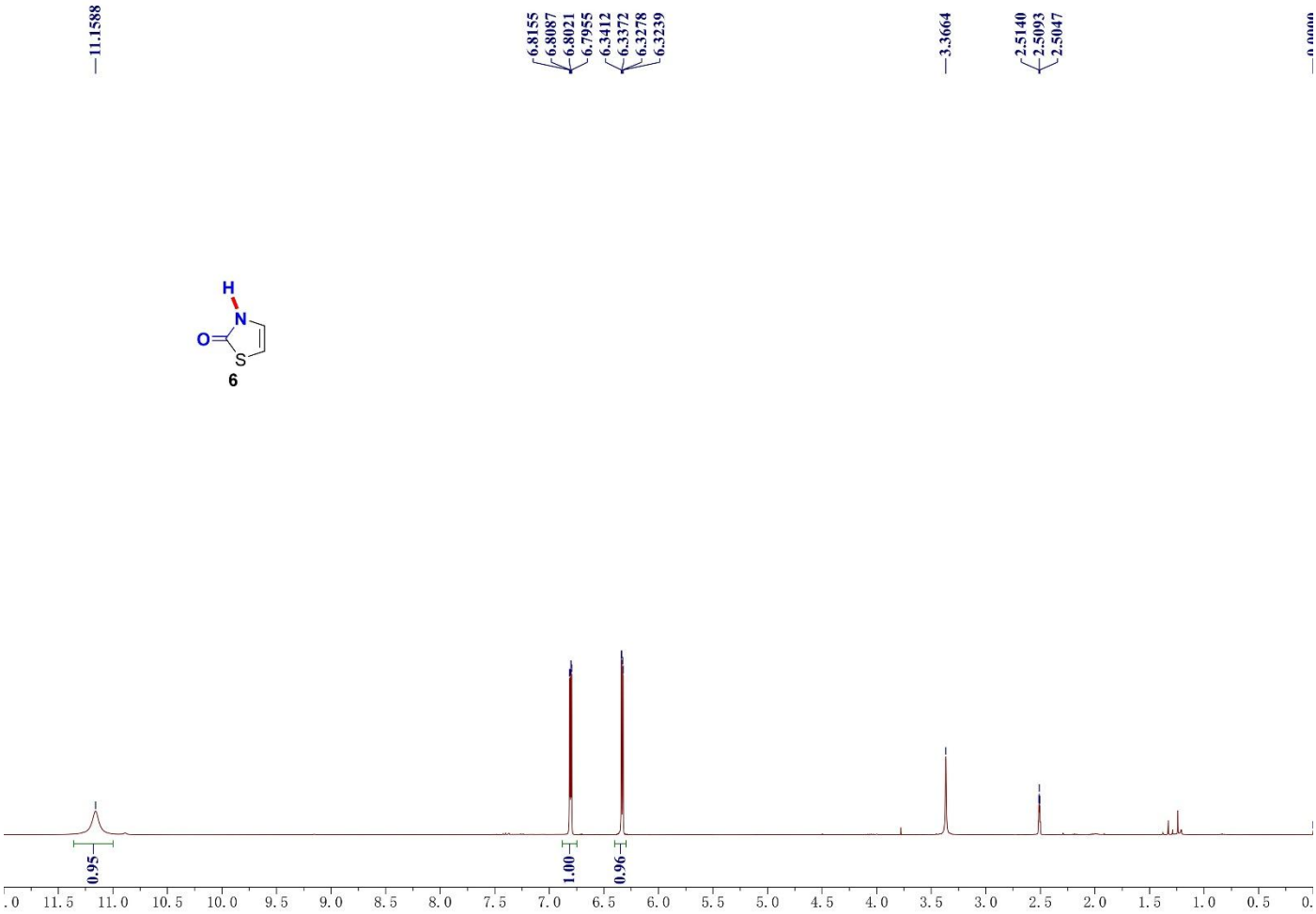
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O
S
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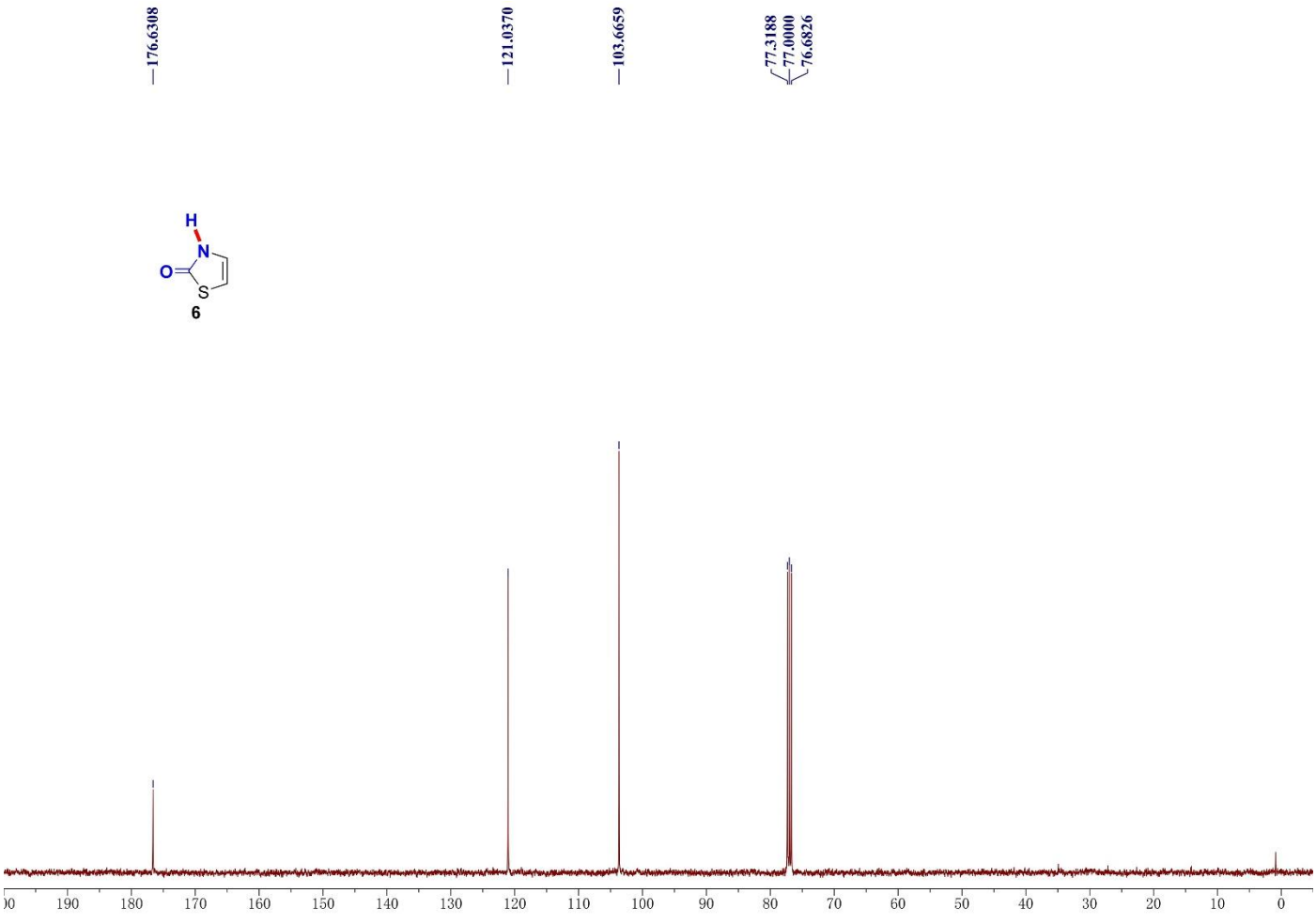
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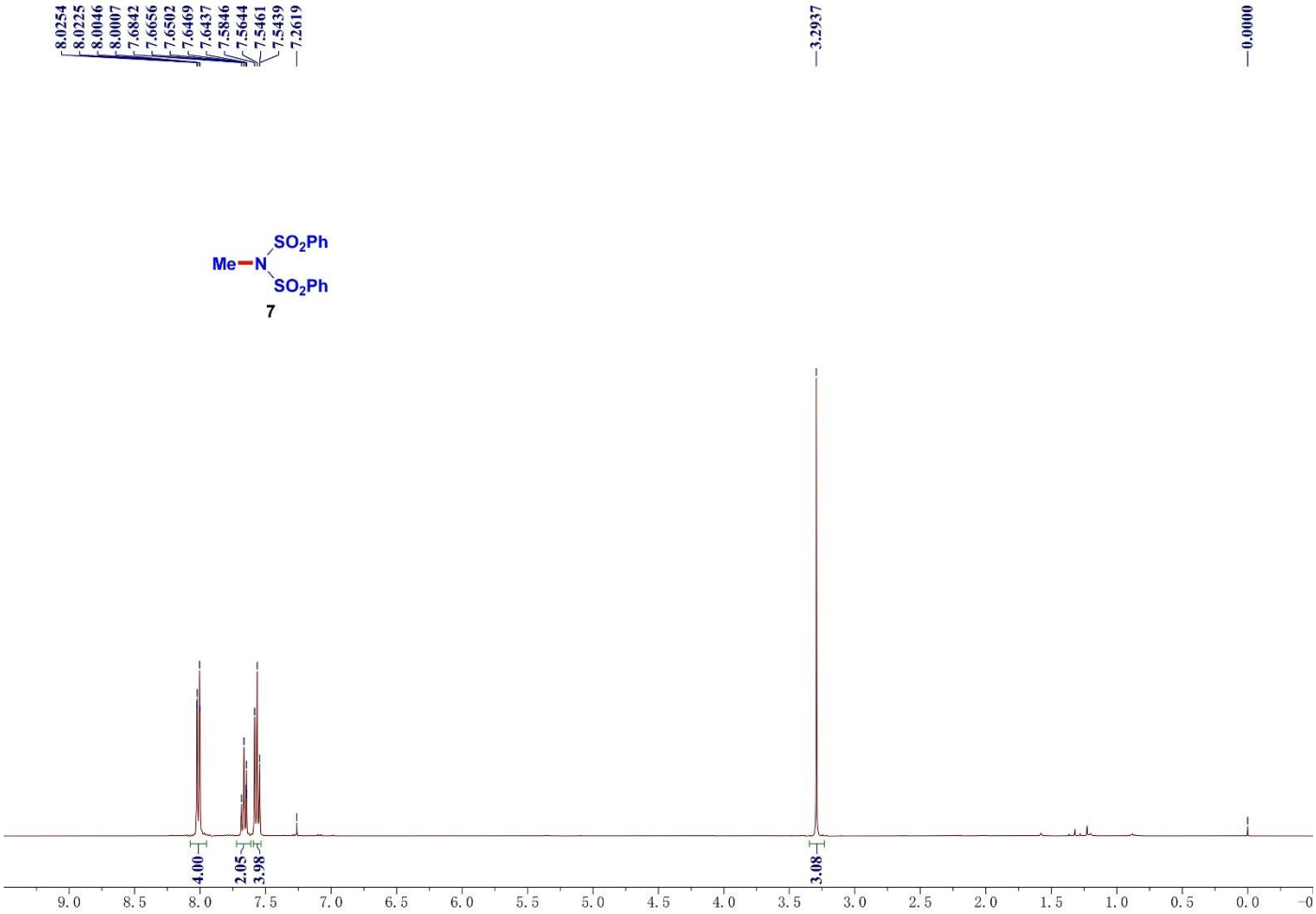
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SO2Ph
SO2Ph
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3.08
9.0
8.5
8.0
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7.0
6.5
6.0
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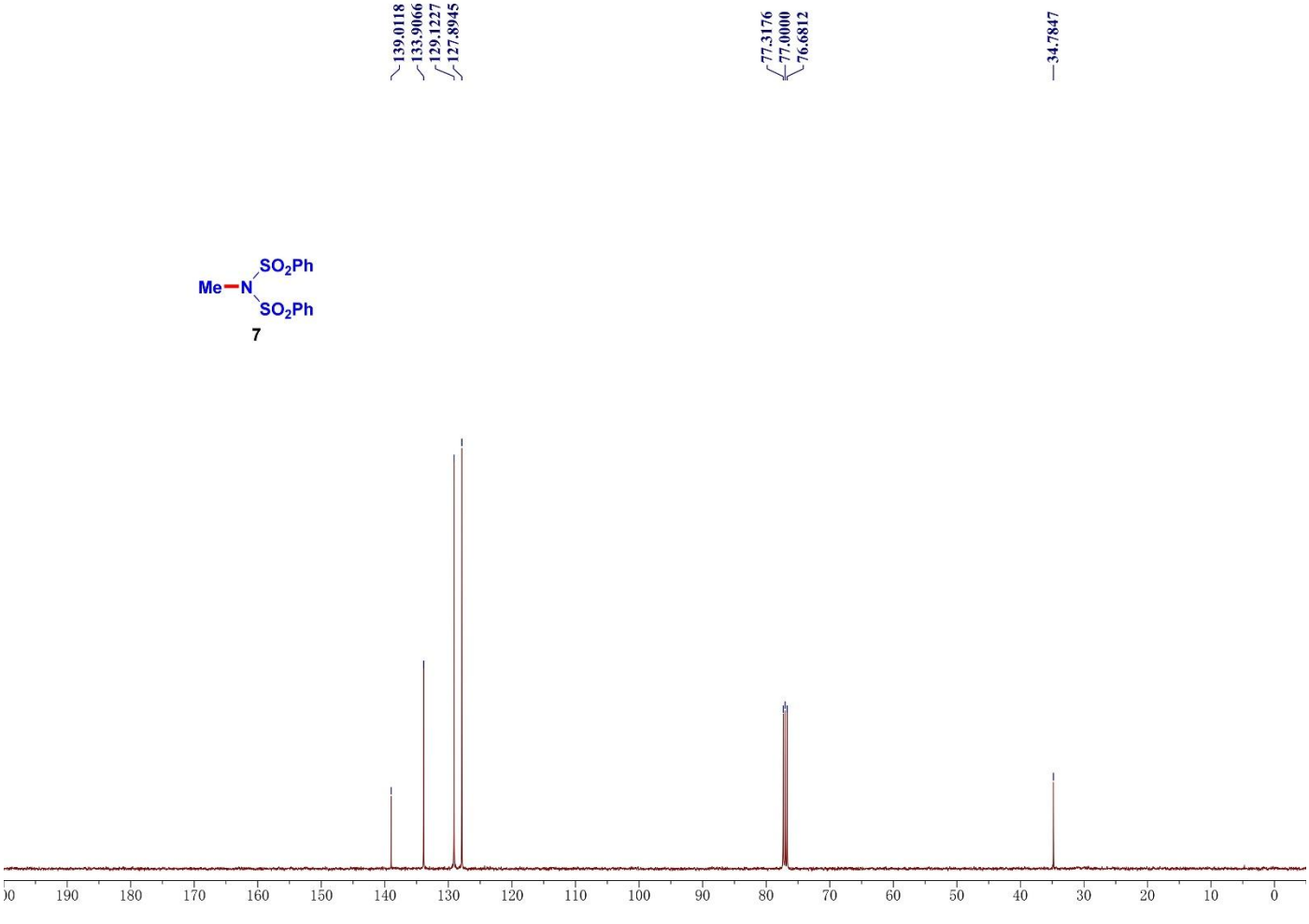
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SO2Ph
SO2Ph
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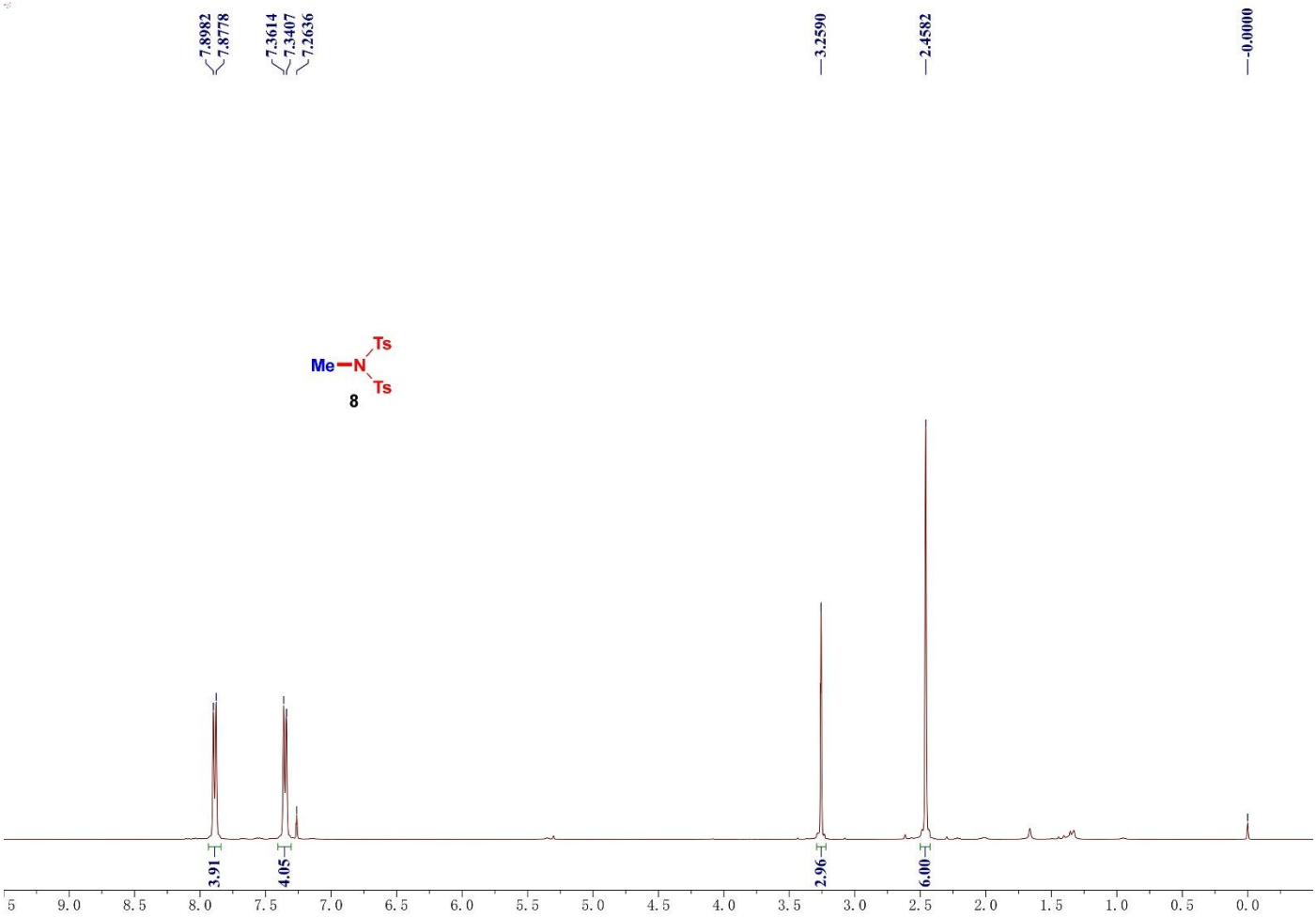
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Ts
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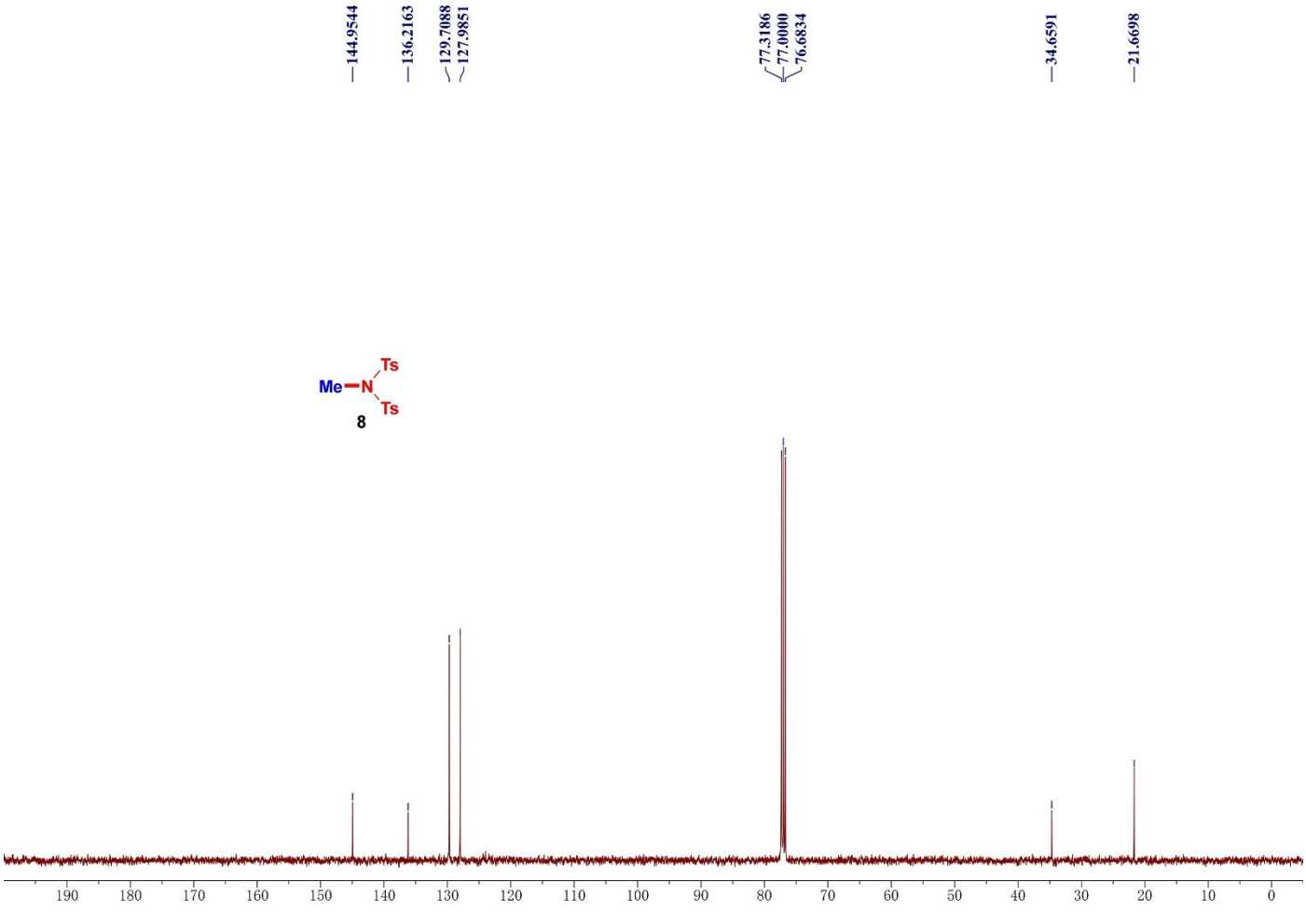
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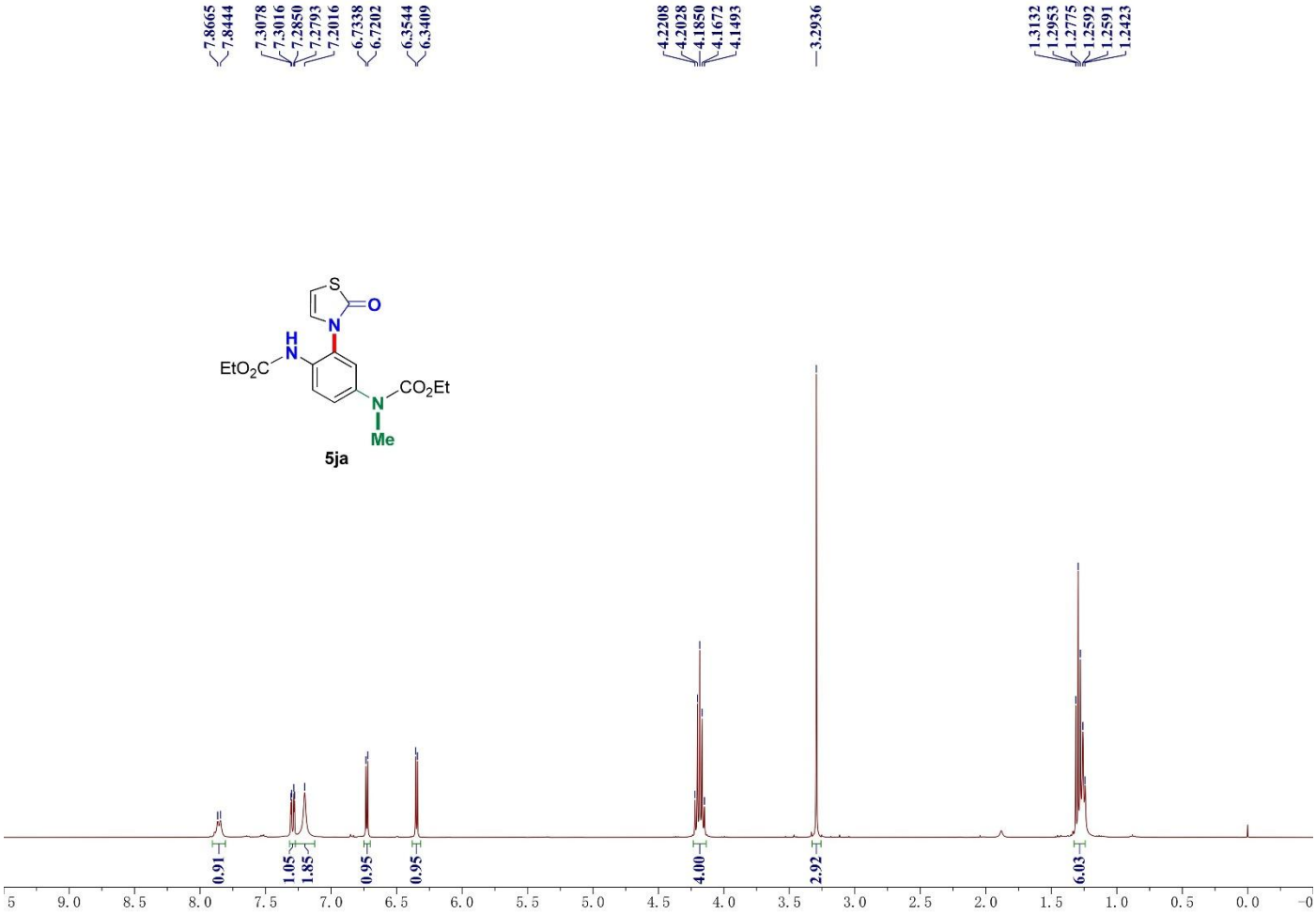
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2.92
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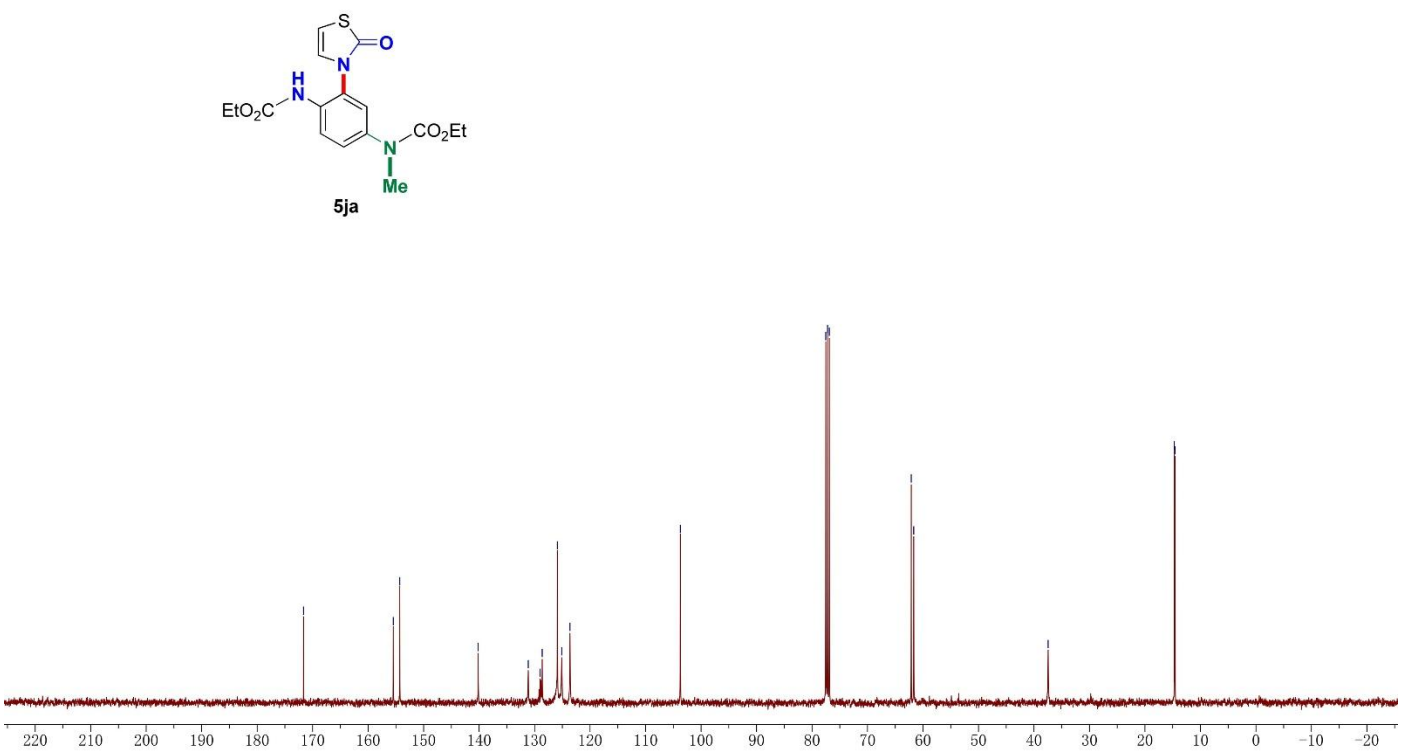
5ja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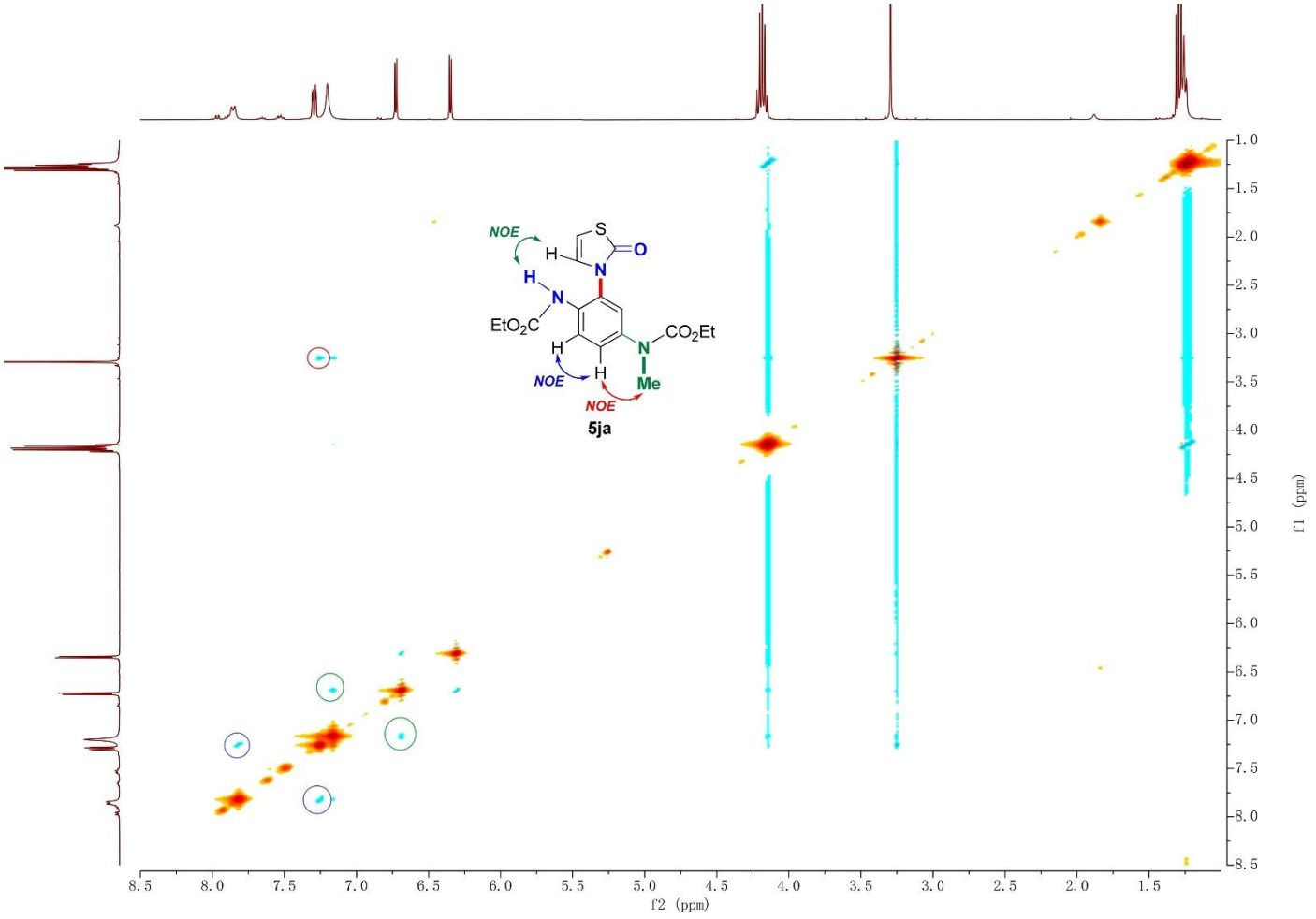
5ja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NOE
NOE
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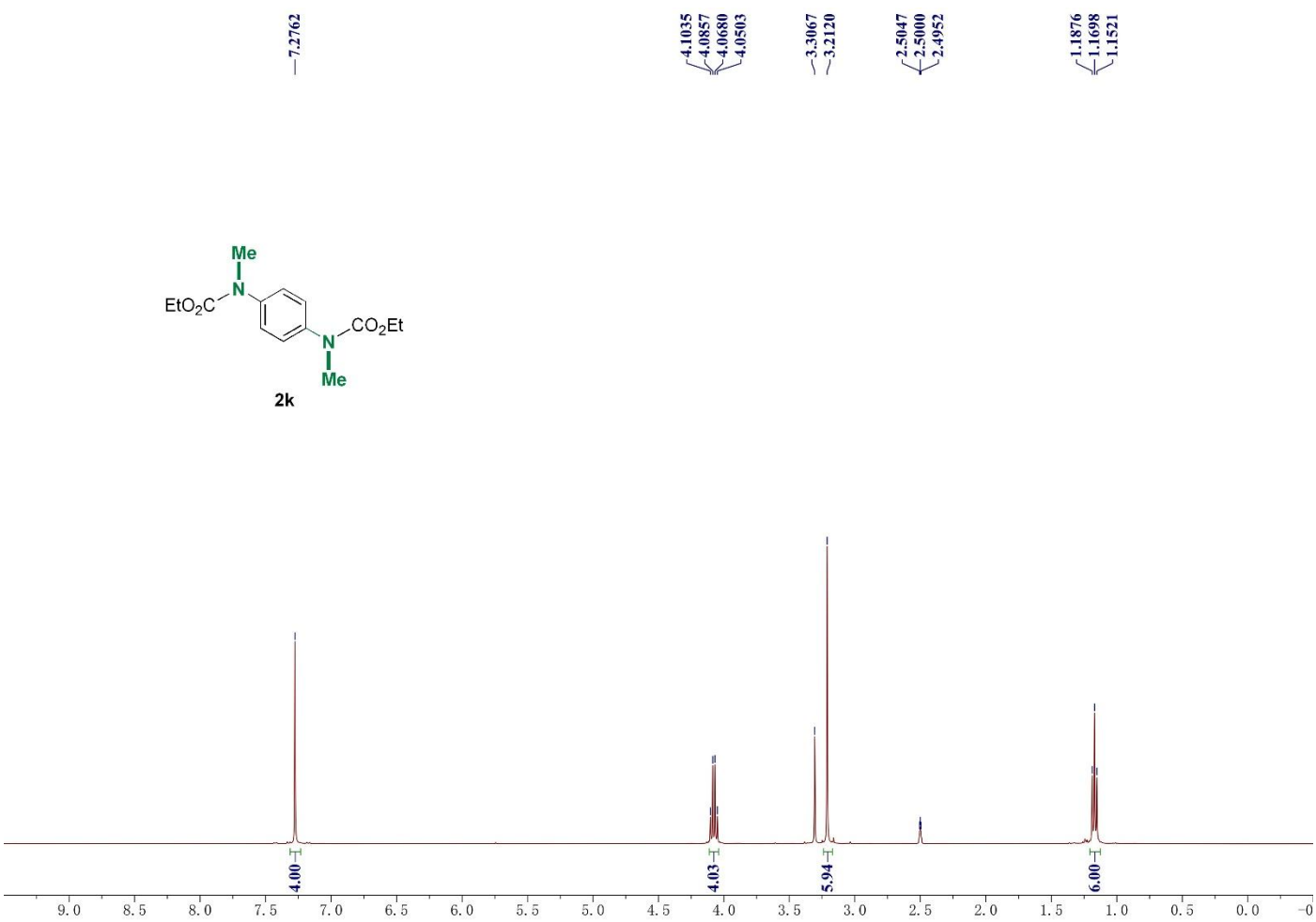
f1 (ppm)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1.1876
1.1698
1.1521
Me
N
EtO2C
CO2Et
N
Me
4.00
4.03
5.94
6.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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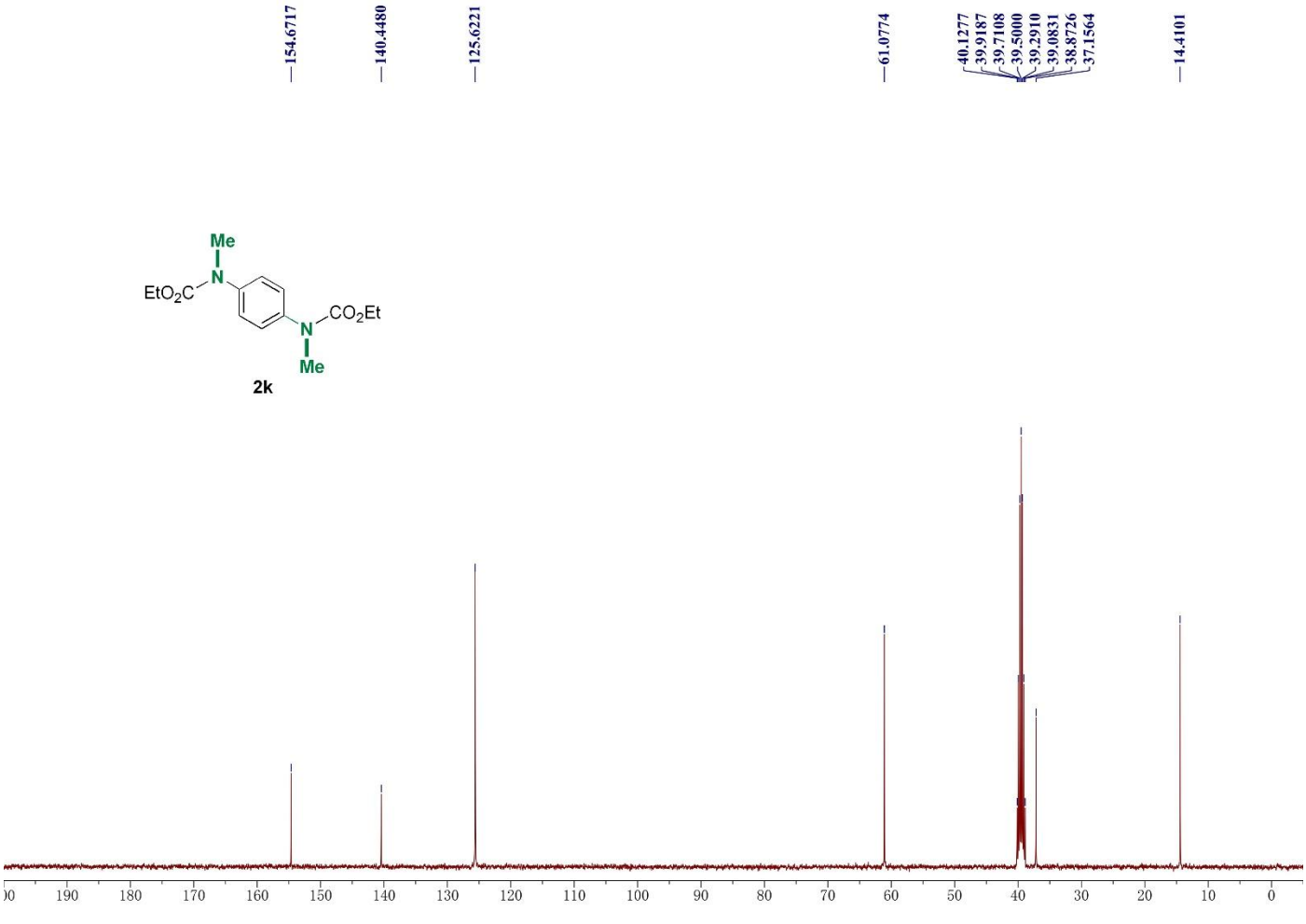
0.0

2k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N
EtO2C
CO2Et
N
Me
190
180
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130
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50
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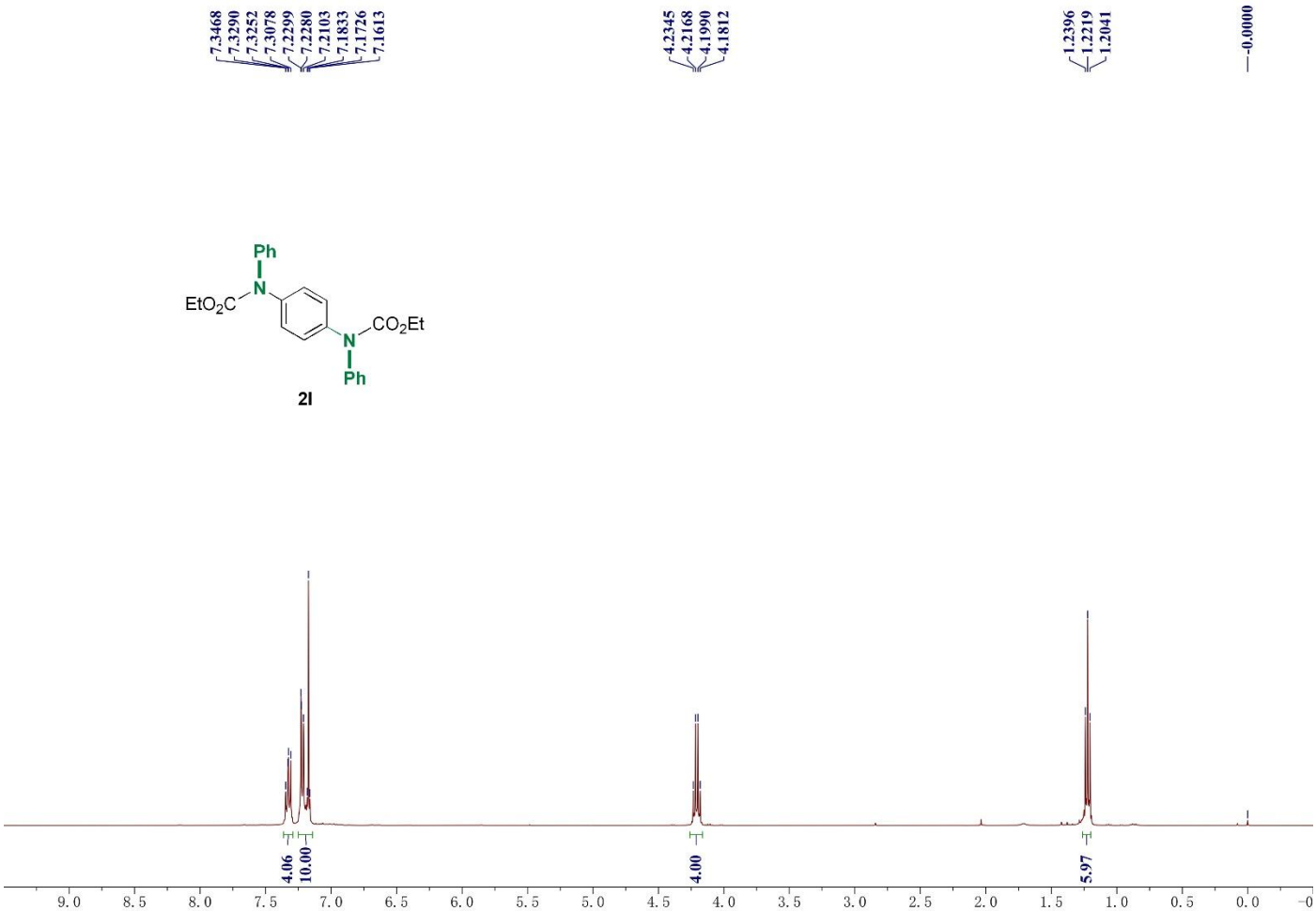
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4.2168
4.1990
4.1812
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1.2219
1.2041
-0.0000
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N
EtO2C
CO2Et
N
Ph
2l
4.06
10.00
4.00
5.97
9.0
8.5
8.0
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7.0
6.5
6.0
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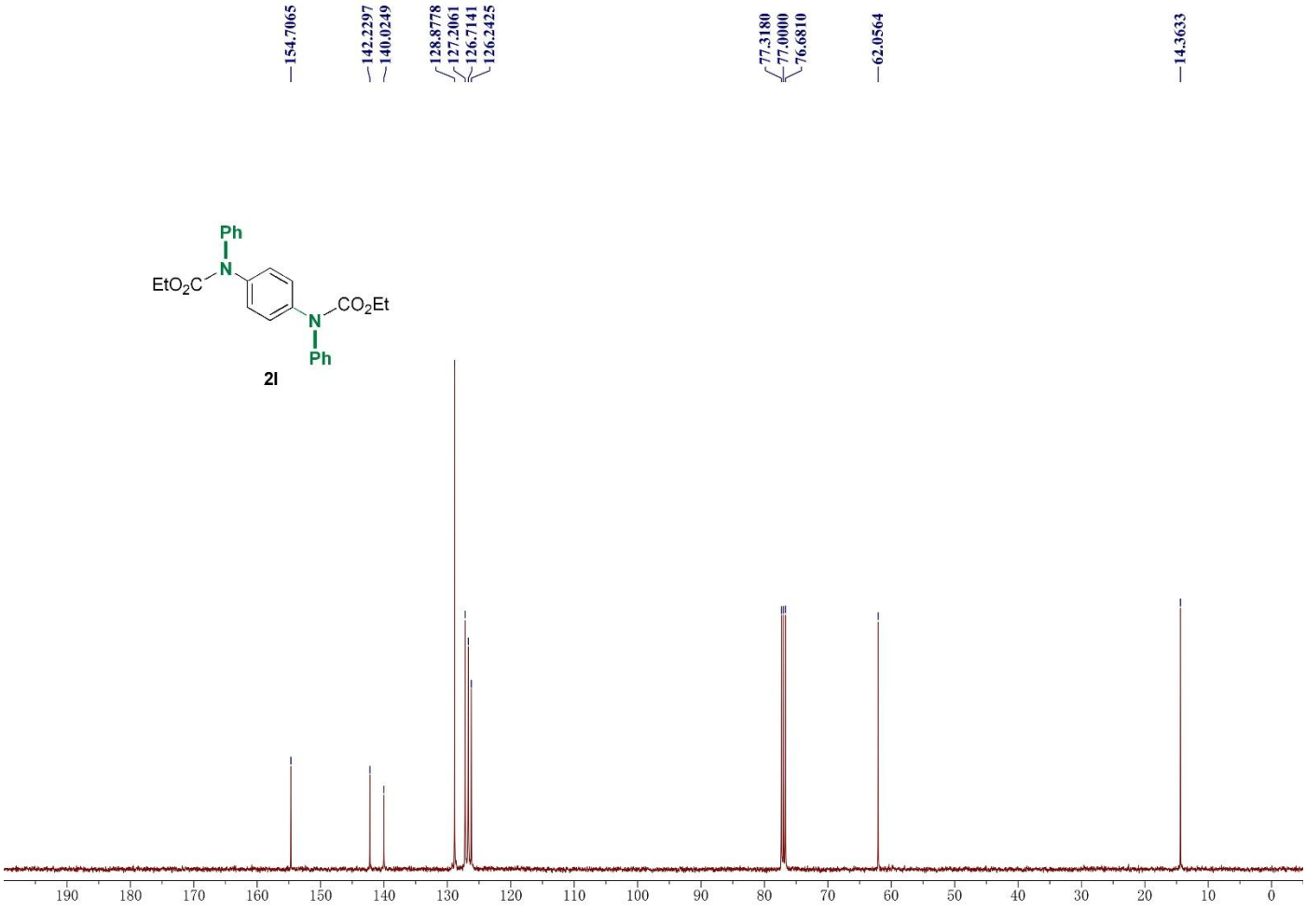
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CO2Et
N
Ph
2l
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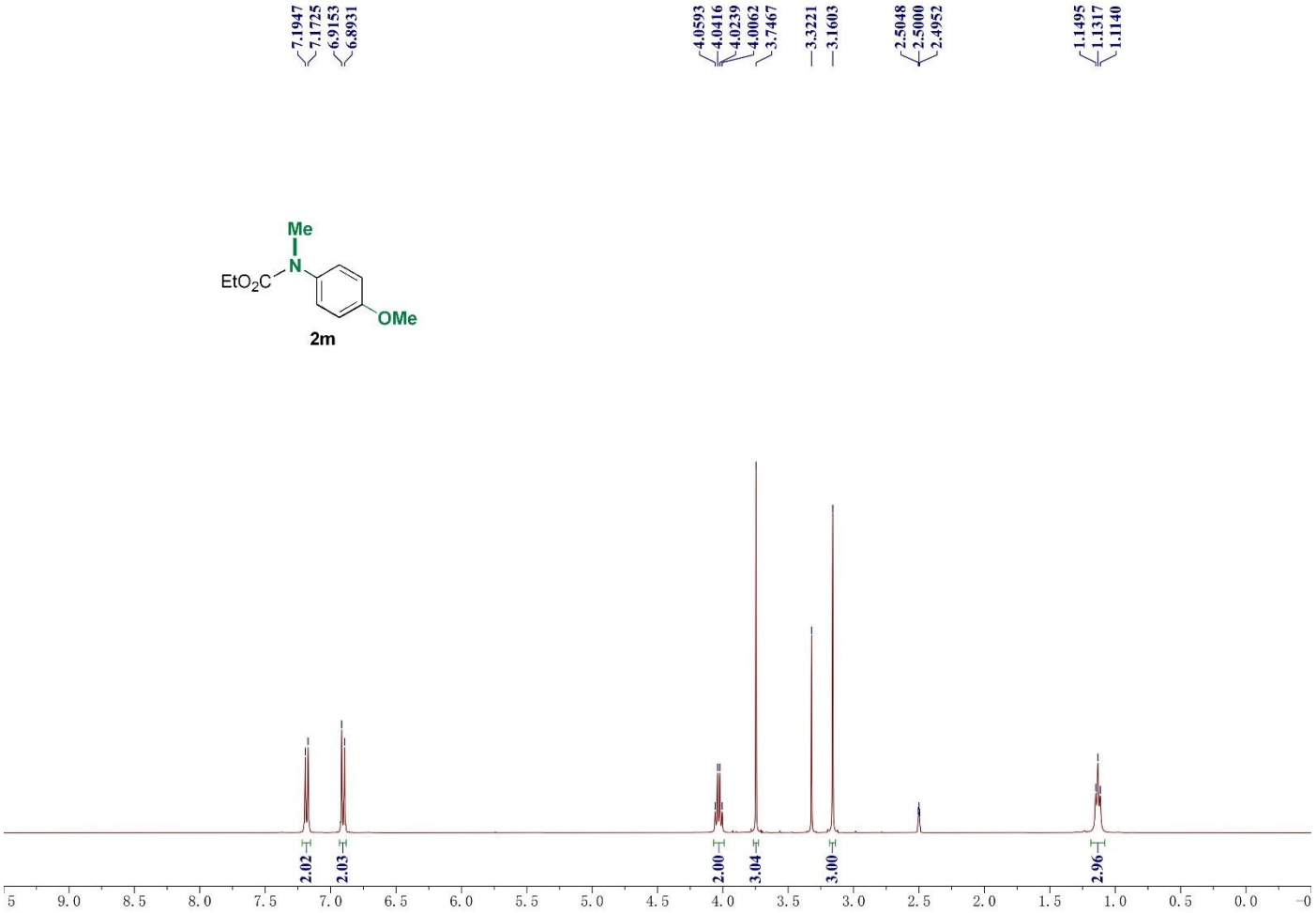
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2.4952
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EtO2C
OMe
2m
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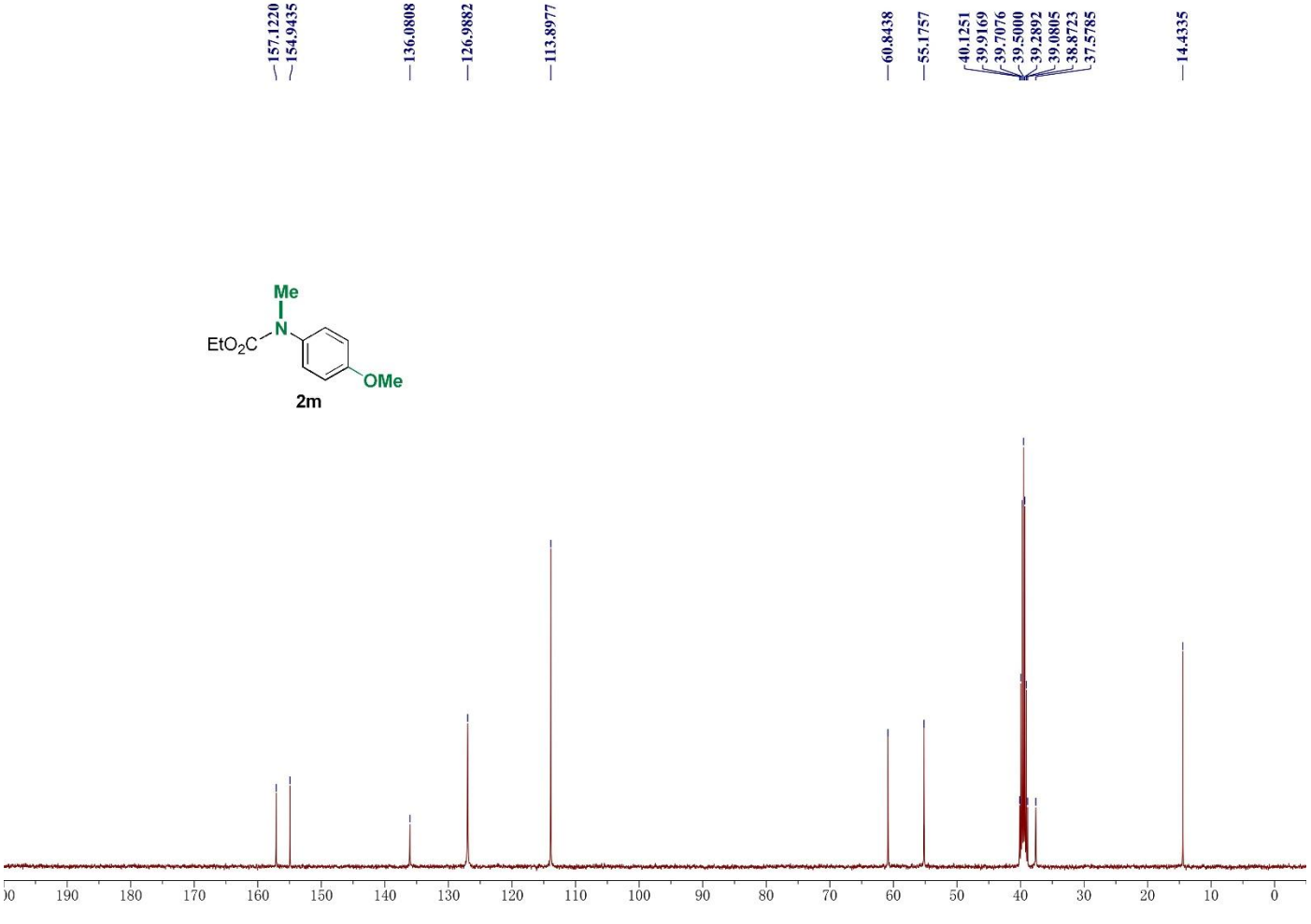
2.03
2.00
3.04
3.00
2.96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39.0805
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37.5785
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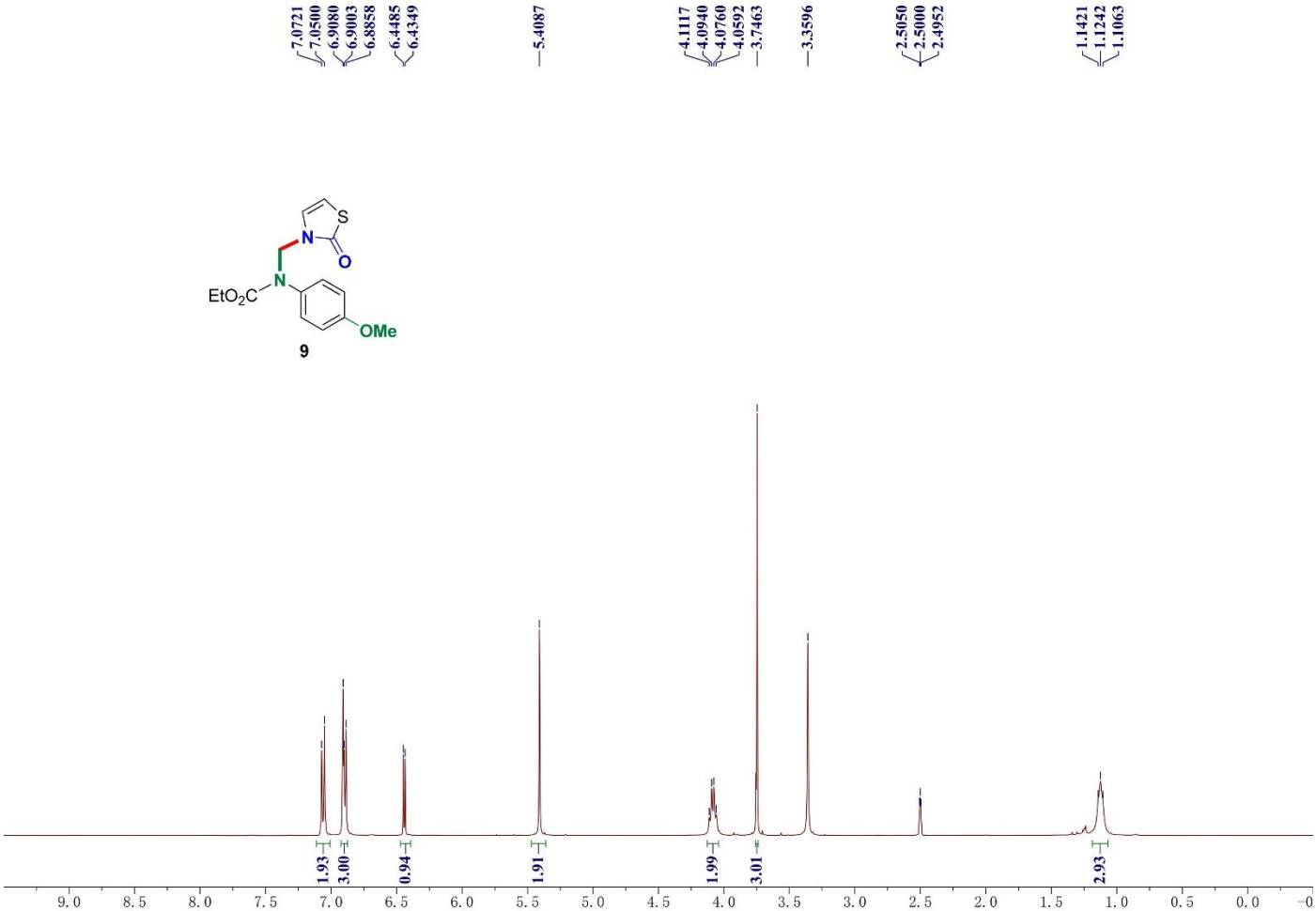
Me
N
EtO2C
OMe
2m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4.0592
3.7463
3.3596
2.5050
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2.4952
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EtO2C
OMe
9
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0.94
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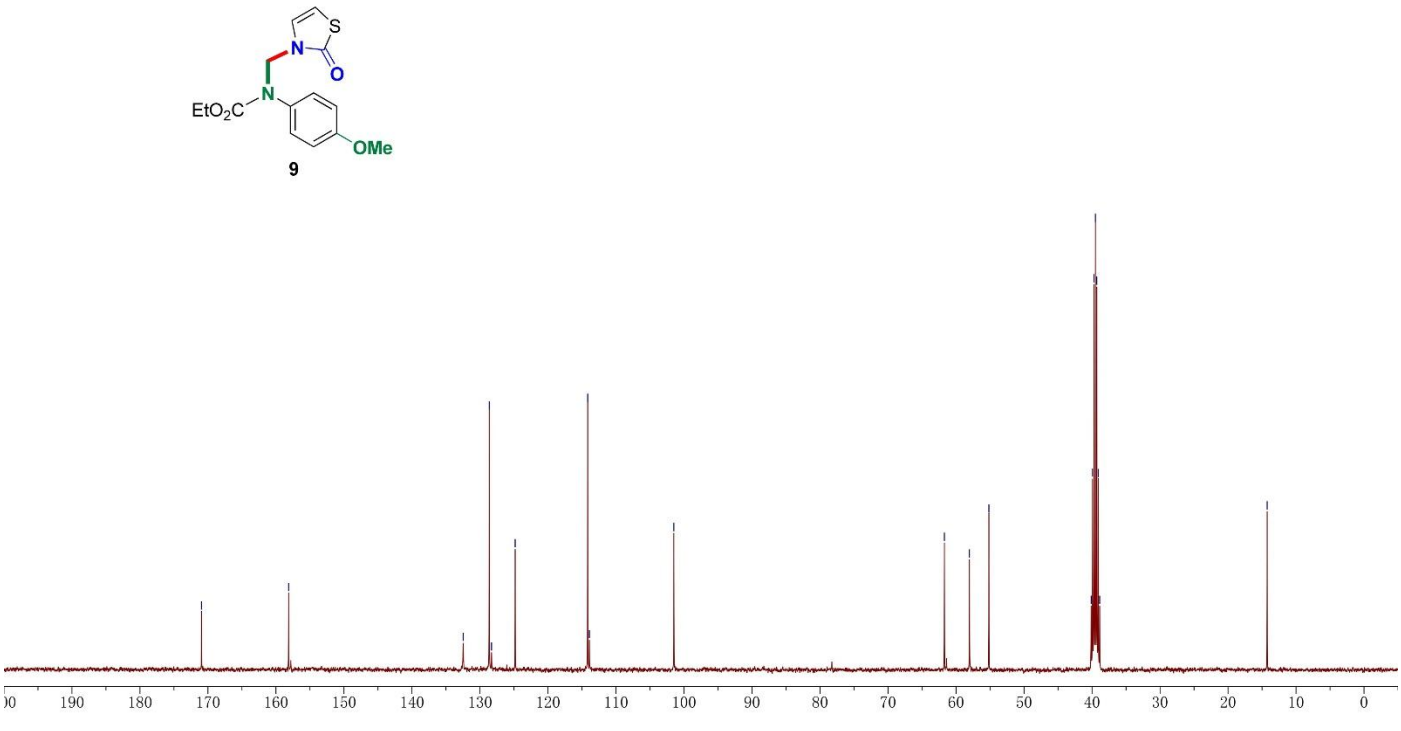
1.99
3.01
2.93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39.0822
38.8751
14.2403
EtO2C
OMe
9