Supporting Information

Catalytic Asymmetric (4+1) Annulation of Nitroalkenes with Allylic Acetates: Stereoselective Synthesis of Isoxazoline *N*-Oxides

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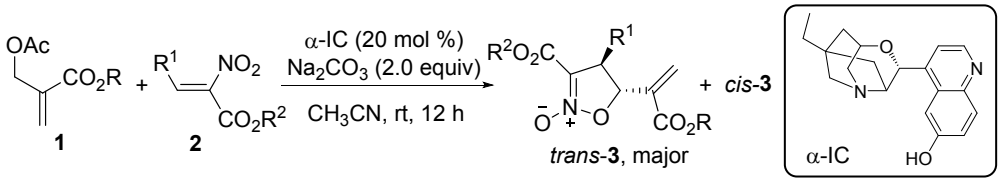
I. General Remarks

Unless otherwise noted, all reactions were run under anhydrous conditions. Solvents were purified or dried by conventional procedures. NMR spectra were recorded in $CDCl_3$ with tetramethylsilane (TMS) used as the internal standard. HRMS spectra were acquired in the ESI mode by using the mass analyzer of TOF. X-ray crystal diffraction data were collected with Mo Kα radiation (λ = 0.7107 Å). Column chromatography isolation was performed by using silica gel (200–300 mesh) stationary phase and petroleum ether (60–90 °C)/ethyl acetate eluent. The values of ee were determined through chiral HPLC using chiral columns with hexane/*i*-PrOH as the eluent. Catalysts **C1**, **C3**, and **C5** were commercially available. Catalysts **C2**,[1a] **C4**,[1b] **C6**,[1c] **C7**,[1c] and **C8**,[1c] were prepared according to the corresponding reported procedures. MBH acetates were prepared by a known method.[2] Nitroalkenes were prepared by a literature procedure.[3]

References:

1. (a) A. E. Taggi, A. M. Hafez, H. Wack, B. Young, D. Ferraris and T. Lectka, *J. Am. Chem. Soc*., 2002, **124**, 6626; (b) P. Hammar, T. Marcelli, H. Hiemstra and F. Himoa, *Adv. Synth. Catal*., 2007, **349**, 2537; (c) Y. Nakamoto, F. Urabe, K. Takahashi, J. Ishihara and S. Hatakeyama, *Chem. Eur. J*., 2013, **19**, 12653.
2. (a) G. Sudhakar, K. Satish and J. Raghavaiah, *J. Org. Chem*., 2012, **77**, 10010; (b) P. V. Ramachandran, T. E. Burghardt and L. Bland-Berry, *J. Org. Chem*., 2005, **70**, 7911.
3. R. S. Fornicola, E. Oblinger and J. Montgomery, *J. Org. Chem*., 1998, **63**, 3528.

II. General Procedure for the Catalytic Asymmetric (4+1) Annulation Reactions of MBH Acetates 1 and Nitroalkenes 2



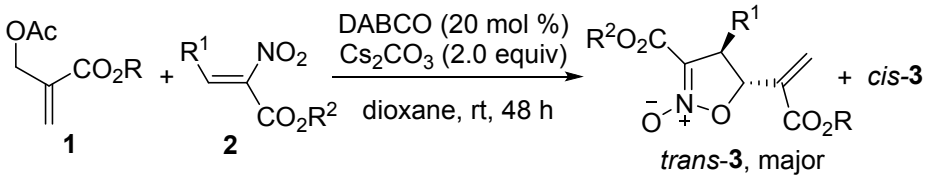
A reaction mixture of MBH acetate **1** (0.30 mmol), nitroalkene **2** (0.15 mmol), Na_2CO_3 (32 mg, 0.30 mmol), and the amine catalyst **C7** (9.3 mg, 0.03 mmol) in acetonitrile (1.0 mL) was stirred at rt for 12 h. The reaction mixture was then concentrated on a rotary evaporator under reduced pressure, the residue was mixed with CH_2Cl_2 (20 mL) and then washed twice with water (5 mL × 2). The organic layer was separated and dried over anhydrous sodium sulfate. After filtration and removal of solvent, the crude product was purified by column chromatography on silica gel (gradient eluent: petroleum ether (60–90 °C)/ethyl acetate 30:1–

10:1) to afford products **3a**–**3q**.

III. Procedure for the DABCO-catalyzed (4+1) Annulation Reactions

A reaction mixture of MBH acetate **1** (0.30 mmol), nitroalkene **2** (0.15 mmol), Cs_2CO_3 (0.30 mmol), and DABCO (0.03 mmol) in dioxane (1.0 mL) was stirred at rt for 48 h. After evaporation of all volatile components, the residue was purified by column chromatography on silica gel (gradient eluant: petroleum ether/ethyl acetate 30:1–10:1) to afford **3a-3i**, **3k**, **3p**, **3q**.

Table S1. DABCO-catalyzed (4+1) annulation reactions of MBH acetates **1** and nitroalkenes **2**



R = *t*-Bu (**1a**), Et (**1b**)

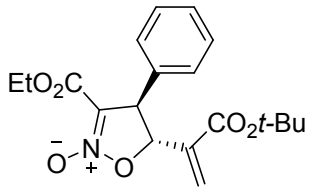
Entry	**1**	R^1, R^2 in **2**	Yield (%)[a]	Dr[b]
1	**1a**	Ph, Et (**2a**)	**3a**, 93	6:1
2	**1a**	4-CF_3-C_6H_4, Et (**2b**)	**3b**, 94	6:1
3	**1a**	3-CF_3-C_6H_4, Et (**2c**)	**3c**, 82	4:1
4	**1a**	4-Cl-C_6H_4, Et(**2d**)	**3d**, 97	4:1
5	**1a**	3-Cl-C_6H_4, Et (**2e**)	**3e**, 91	10:1
6	**1a**	4-Br-C_6H_4, Et (**2f**)	**3f**, 92	5:1
7	**1a**	3-Br-C_6H_4, Et (**2g**)	**3g**, 95	4:1
8	**1a**	2-Br-C_6H_4, Et (**2h**)	**3h**,80	9:1
9	**1a**	4-F-C_6H_4, Et (**2i**)	**3i**, 97	5:1
10	**1a**	4-CH_3-C_6H_4, Et (**2k**)	**3k**, 88	4:1
11	**1b**	Ph, Et (**2a**)	**3p**, 67	7:1
12	**1b**	4-Br-C_6H_4, Et (**2f**)	**3q**, 61	6:1

[a] Isolated total yield of product **3**. [b] Ratio of *trans*-/*cis*-**3** determined by ^{1}H NMR assay of the crude products.

IV. Procedure for Product 3r in Scheme 2

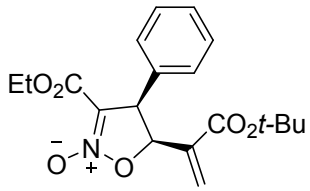
A stirred solution of **1c** (0.45 mmol) and **2q** (0.30 mmol) in DMF (2.0 mL) was added DABCO (0.06 mmol), and the resulting mixture was continuously stirred at rt for 12 h (monitored by TLC). Water (10 mL) was added into it and the mixture was extracted twice with CH_2Cl_2 (20 mL × 2). The combined organic layer was dried over anhydrous sodium sulfate. After filtration and concentration on a rotary evaporator under reduced pressure, the residue was subjected to column chromatography on silica gel to give the product **3r**.

V. NMR Analytical Data for New Compounds 3 and 4



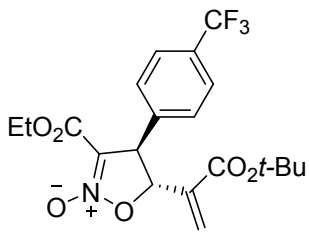
trans-**3a**

*5-(3-(tert-Butoxy)-3-oxoprop-1-en-2-yl)-3-(ethoxycarbonyl)-4-phenyl-4,5-dihydroisoxazole 2-oxide (trans-**3a**)*. Colorless oil; 28 mg, yield 51%; ee > 99%, $[\alpha]_D^{25}$ (CH_2Cl_2, c = 0.5) = +59.6; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.32 (m, 5H), 6.38 (s, 1H), 6.00 (d, *J* = 0.8 Hz, 1H), 5.33 – 5.24 (m, 1H), 4.48 (d, *J* = 2.8 Hz, 1H), 4.18 (q, *J* = 7.1 Hz, 2H), 1.46 (s, 9H), 1.17 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.5, 157.6, 137.9, 137.2, 128.1, 127.3, 126.3, 124.7, 109.4, 81.4, 79.8, 60.8, 54.3, 27.0, 12.9; HRMS-ESI calcd for $C_{19}H_{24}NO_6$ $[M + H]^+$ 362.1598, found 362.1596. The enantiomeric excess of *trans*-**3a** was measured by HPLC analysis with a Chiralcel AS-H column (hexane/*i*-PrOH: 95/5, flow rate: 1.0 mL/min, λ = 254 nm, t_{major} = 13.8 min, t_{minor} = 11.2 min).



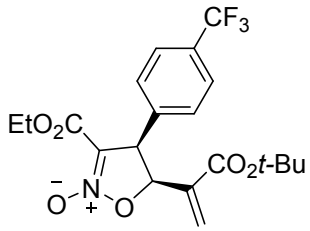
cis-**3a**

*5-(3-(tert-Butoxy)-3-oxoprop-1-en-2-yl)-3-(ethoxycarbonyl)-4-phenyl-4,5-dihydroisoxazole 2-oxide (cis-**3a**)*. Colorless oil; 9 mg, yield 16%; ee > 99%, $[\alpha]_D^{25}$ (CH_2Cl_2, c = 0.5) = -44.8; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26–7.23 (m, 3H), 7.08–7.06 (m, 2H), 6.03 (s, 1H), 5.77 (d, *J* = 8.3 Hz, 1H), 5.73 (s, 1H), 4.95 (d, *J* = 8.3 Hz, 1H), 4.17 (q, *J* = 7.1 Hz, 2H), 1.40 (s, 9H), 1.13 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.3, 158.5, 134.9, 134.1, 128.7, 128.5, 128.3, 126.3, 112.9, 81.9, 78.2, 61.8, 52.3, 27.9, 13.9; HRMS-ESI calcd for $C_{19}H_{24}NO_6$ $[M + H]^+$ 362.1598, found 362.1595. The enantiomeric excess of *cis*-**3a** was assessed by HPLC analysis with a Chiralcel AS-H column (hexane/*i*-PrOH: 95/5, flow rate: 1.0 mL/min, λ = 254 nm, t_{major} = 23.4 min, t_{minor} = 19.5 min). HPLC analysis showed that the sample of *cis*-**3a** contained a small amount of single *trans*-**3a** diastereomer.



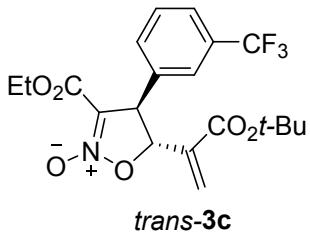
trans-**3b**

*5-(3-(tert-Butoxy)-3-oxoprop-1-en-2-yl)-3-(ethoxycarbonyl)-4-(4-(trifluoromethyl)phenyl)-4,5-dihydroisoxazole 2-oxide (trans-**3b**)*. Yellow oil; 39 mg, yield 61%; ee > 99%, $[\alpha]_D^{25}$ (CH_2Cl_2, c = 0.5) = +32.4; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.67 (d, *J* = 8.1 Hz, 2H), 7.52 (d, *J* = 8.1 Hz, 2H), 6.40 (s, 1H), 6.04 (s, 1H), 5.27 (s, 1H), 4.56 (d, *J* = 2.5 Hz, 1H), 4.23–4.18 (m, 2H), 1.48 (s, 9H), 1.19 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.5, 158.5, 142.9, 137.8, 130.6 (q, *J* = 32.6 Hz), 127.8, 126.1 (q, *J* = 7.5 Hz), 125.8, 123.8 (q, *J* = 271.9 Hz), 109.8, 82.7, 80.3, 62.1, 55.0, 28.0, 13.9; HRMS-ESI calcd for $C_{20}H_{23}F_3NO_6$ $[M + H]^+$ 430.1472, found 430.1479. The enantiomeric excess of *trans*-**3b** was assessed by HPLC analysis with a Chiralcel AS-H column (hexane/*i*-PrOH: 95/5, flow rate: 1.0 mL/min, λ = 254 nm, t_{major} = 8.4 min, t_{minor} = 6.0 min).



cis-**3b**

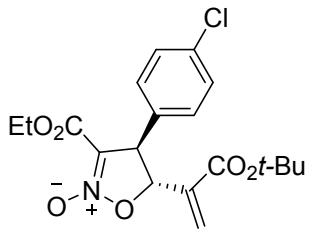
*5-(3-(tert-Butoxy)-3-oxoprop-1-en-2-yl)-3-(ethoxycarbonyl)-4-(4-(trifluoromethyl)phenyl)-4,5-dihydroisoxazole 2-oxide (cis-**3b**)*. Yellow oil; 10 mg, yield 15%; ee > 99%, $[\alpha]_D^{25}$ (CH_2Cl_2, c = 0.5) = -26.4; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (d, *J* = 8.1 Hz, 2H), 7.22 (d, *J* = 8.1 Hz, 2H), 6.07 (d, *J* = 0.7 Hz, 1H), 5.79 (d, *J* = 8.2 Hz, 1H), 5.78 (s, 1H), 5.04 (d, *J* = 8.2 Hz, 1H), 4.18 (q, *J* = 7.1 Hz, 2H), 1.38 (s, 9H), 1.15 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.2, 158.3, 139.2, 133.9, 130.5 (q, *J* = 32.4 Hz), 129.2, 126.7, 125.4 (q, *J* = 3.8 Hz), 123.8 (q, *J* = 272.2 Hz), 112.3, 82.1, 77.8, 62.0, 52.0, 27.9, 13.9; HRMS-ESI calcd for $C_{20}H_{23}F_3NO_6$ $[M + H]^+$ 430.1472, found 430.1479. The enantiomeric excess of *cis*-**3b** was measured by HPLC analysis with a Chiralcel AS-H column (hexane/*i*-PrOH: 95/5, flow rate: 1.0 mL/min, λ = 254 nm, t_{major} = 13.5 min, t_{minor} = 9.6 min).



trans-**3c**

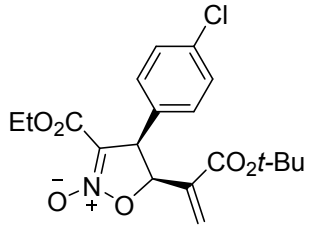
*5-(3-(tert-Butoxy)-3-oxoprop-1-en-2-yl)-3-(ethoxycarbonyl)-4-(3-(trifluoromethyl)phenyl)-4,5-dihydroisoxazole 2-oxide (trans-**3c**)*. Yellow oil; 37 mg, yield 58%; ee 98%, $[\alpha]_D^{25}$ (CH_2Cl_2, c = 0.5) = +6; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 – 7.58 (m, 3H), 7.57–7.52 (m, 1H), 6.40 (s, 1H), 6.03 (d, *J* = 1.2 Hz, 1H), 5.33–5.30 (m, 1H), 4.55 (d, *J* = 2.7 Hz, 1H), 4.27 – 4.11 (m, 2H), 1.46 (s, 9H), 1.17 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.4, 158.4, 140.0, 137.8,

131.4 (q, J = 26.0 Hz), 130.6, 129.8, 125.9, 125.3 (q, J = 7.4 Hz), 124.4 (q, J = 3.9 Hz), 124.1 (q, J = 271.9 Hz), 109.8, 82.7, 80.3, 62.0, 55.1, 28.0, 13.8; HRMS-ESI calcd for $C_{20}H_{23}F_3NO_6$ $[M + H]^+$ 430.1472, found 430.1475. The enantiomeric excess of *trans*-**3c** was determined by HPLC analysis with a Chiralcel AD-H column (hexane/*i*-PrOH: 95/5, flow rate: 1.0 mL/min, λ = 254 nm, t_{major} = 8.2 min, t_{minor} = 6.4 min).



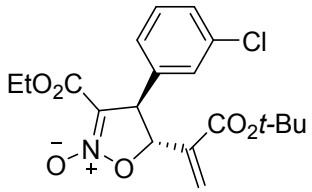
trans-**3d**

*5-(3-(tert-Butoxy)-3-oxoprop-1-en-2-yl)-4-(4-chlorophenyl)-3-(ethoxycarbonyl)-4,5-dihydroisoxazole 2-oxide (trans-**3d**)*. Colorless oil; 35 mg, yield 59%; ee > 99%, $[\alpha]_D^{25}$ (CH_2Cl_2, c = 0.5) = +42.8; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (d, J = 8.5 Hz, 2H), 7.32 (d, J = 8.5 Hz, 2H), 6.38 (s, 1H), 6.01 (d, J = 1.0 Hz, 1H), 5.28 – 5.22 (m, 1H), 4.47 (d, J = 2.7 Hz, 1H), 4.22 – 4.16 (m, 2H), 1.48 (s, 9H), 1.19 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.4, 158.5, 137.9, 137.4, 134.3, 129.3, 128.6, 125.6, 109.9, 82.5, 80.5, 61.9, 54.7, 28.0, 13.9; HRMS-ESI calcd for $C_{19}H_{23}ClNO_6$ $[M + H]^+$ 396.1208, found 396.1210. The enantiomeric excess of *trans*-**3d** was assessed by HPLC analysis with a Chiralcel AD-H column (hexane/*i*-PrOH: 95/5, flow rate: 1.0 mL/min, λ = 254 nm, t_{major} = 12.4 min, t_{minor} = 8.8 min).



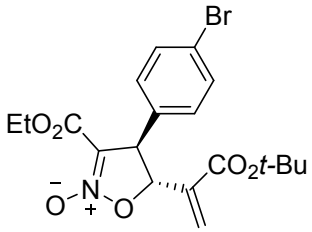
cis-**3d**

*5-(3-(tert-Butoxy)-3-oxoprop-1-en-2-yl)-4-(4-chlorophenyl)-3-(ethoxycarbonyl)-4,5-dihydroisoxazole 2-oxide (cis-**3d**)*. Colorless oil; 7 mg, yield 12%; ee > 99%, $[\alpha]_D^{25}$ (CH_2Cl_2, c = 0.5) = -19.2; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 (d, J = 8.4 Hz, 2H), 7.02 (d, J = 8.4 Hz, 2H), 6.07 (s, 1H), 5.77 – 5.71 (m, 2H), 4.95 (d, J = 8.6 Hz, 1H), 4.18 (q, J = 7.1 Hz, 2H), 1.41 (s, 9H), 1.16 (t, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.2, 158.3, 134.2, 133.9, 133.5, 130.0, 128.7, 126.5, 112.5, 82.0, 78.0, 61.9, 51.7, 27.9, 13.9; HRMS-ESI calcd for $C_{19}H_{23}ClNO_6$ $[M + H]^+$ 396.1208, found 396.1210. The enantiomeric excess of *cis*-**3d** was measured by HPLC analysis with a Chiralcel AS-H column (hexane/*i*-PrOH: 95/5, flow rate: 1.0 mL/min, λ = 254 nm, t_{major} = 21.0 min, t_{minor} = 13.9 min).



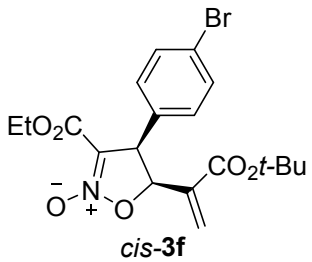
trans-**3e**

*5-(3-(tert-Butoxy)-3-oxoprop-1-en-2-yl)-4-(3-chlorophenyl)-3-(ethoxycarbonyl)-4,5-dihydroisoxazole 2-oxide (trans-**3e**)*. Yellow oil; 30 mg, yield 51%; ee 97%, $[\alpha]_D^{25}$ (CH_2Cl_2, c = 0.5) = +7.6; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (s, 1H), 7.35–7.32 (m, 2H), 7.29–7.25 (m, 1H), 6.39 (s, 1H), 6.01 (d, *J* = 1.2 Hz, 1H), 5.30 – 5.26 (m, 1H), 4.46 (d, *J* = 2.7 Hz, 1H), 4.26 – 4.14 (m, 2H), 1.48 (s, 9H), 1.20 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.4, 158.5, 140.9, 137.9, 134.9, 130.4, 128.6, 127.6, 125.9, 125.4, 109.9, 82.6, 80.4, 62.0, 54.9, 28.0, 13.9; HRMS-ESI calcd for $C_{38}H_{48}Cl_2N_3O_{12}$ $[2M + NH_4]^+$ 808.2610, found 808.2614. The enantiomeric excess of *trans*-**3e** was measured by HPLC analysis with a Chiralcel AS-H column (hexane/*i*-PrOH: 95/5, flow rate: 1.0 mL/min, λ = 254 nm, t_{major} = 10.2 min, t_{minor} = 7.9 min).



trans-**3f**

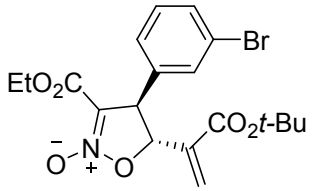
*4-(4-Bromophenyl)-5-(3-(tert-butoxy)-3-oxoprop-1-en-2-yl)-3-(ethoxycarbonyl)-4,5-dihydroisoxazole 2-oxide (trans-**3f**)*. Yellow oil; 32 mg, yield 48%; ee > 99%, $[\alpha]_D^{25}$ (CH_2Cl_2, c = 0.5) = +28; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (d, *J* = 8.4 Hz, 2H), 7.26 (d, *J* = 8.4 Hz, 2H), 6.37 (s, 1H), 6.01 (d, *J* = 1.1 Hz, 1H), 5.27 – 5.21 (m, 1H), 4.46 (d, *J* = 2.7 Hz, 1H), 4.24 – 4.14 (m, 2H), 1.48 (s, 9H), 1.19 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.4, 158.5, 138.0, 137.8, 132.2, 129.0, 125.7, 122.4, 109.9, 82.5, 80.4, 61.9, 54.7, 28.0, 13.9; HRMS-ESI calcd for $C_{19}H_{23}BrNO_6$ $[M + H]^+$ 440.0703, found 440.0698. The enantiomeric excess of *trans*-**3f** was determined by HPLC analysis with a Chiralcel AS-H column (hexane/*i*-PrOH: 95/5, flow rate: 1.0 mL/min, λ = 254 nm, t_{major} = 13.8 min, t_{minor} = 9.7 min).



cis-**3f**

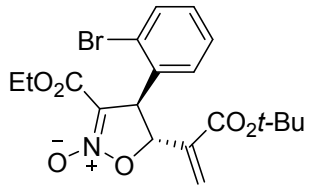
*4-(4-Bromophenyl)-5-(3-(tert-butoxy)-3-oxoprop-1-en-2-yl)-3-(ethoxycarbonyl)-4,5-dihydroisoxazole 2-oxide (cis-**3f**)*. Yellow oil; 10 mg, yield 16%; ee > 99%, $[\alpha]_D^{25}$ (CH_2Cl_2, c =

0.5) = -14; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 (d, *J* = 8.4 Hz, 2H), 6.96 (d, *J* = 8.4 Hz, 2H), 6.07 (s, 1H), 5.77–5.73 (m, 2H), 4.93 (d, *J* = 8.7 Hz, 1H), 4.23 – 4.14 (m, 2H), 1.41 (s, 9H), 1.16 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.2, 158.3, 134.1, 133.9, 131.7, 130.3, 126.5, 122.4, 112.4, 82.0, 77.9, 61.9, 51.7, 27.9, 13.9; HRMS-ESI calcd for $C_{19}H_{23}BrNO_6$ $[M + H]^+$ 440.0703, found 440.0698. The enantiomeric excess of *cis*-**3f** was determined by HPLC analysis with a Chiralcel AS-H column (hexane/*i*-PrOH: 95/5, flow rate: 1.0 mL/min, λ = 254 nm, t_{major} = 24.3 min, t_{minor} = 16.8 min).



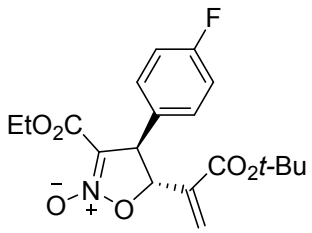
trans-**3g**

*4-(3-Bromophenyl)-5-(3-(tert-butoxy)-3-oxoprop-1-en-2-yl)-3-(ethoxycarbonyl)-4,5-dihydroisoxazole 2-oxide (trans-**3g**)*. Colorless oil; 36 mg, yield 55%; ee 97%, $[\alpha]_D^{25}$ (CH_2Cl_2, c = 0.5) = +31.2; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (s, 1H), 7.48 (d, *J* = 7.6 Hz, 1H), 7.33 – 7.25 (m, 2H), 6.39 (s, 1H), 6.01 (s, 1H), 5.32 – 5.23 (m, 1H), 4.45 (d, *J* = 2.7 Hz, 1H), 4.25 – 4.15 (m, 2H), 1.48 (s, 9H), 1.20 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.4, 158.4, 141.1, 137.8, 131.5, 130.7, 130.4, 125.9, 125.8, 123.0, 109.8, 82.6, 80.4, 61.9, 54.9, 28.0, 13.9; HRMS-ESI calcd for $C_{19}H_{23}BrNO_6$ $[M + H]^+$ 440.0703, found 440.0709. The enantiomeric excess of *trans*-**3g** was measured by HPLC analysis with a Chiralcel AS-H column (hexane/*i*-PrOH: 95/5, flow rate: 1.0 mL/min, λ = 254 nm, t_{major} = 11.2 min, t_{minor} = 8.9 min).



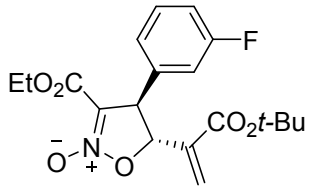
trans-**3h**

*4-(2-Bromophenyl)-5-(3-(tert-butoxy)-3-oxoprop-1-en-2-yl)-3-(ethoxycarbonyl)-4,5-dihydroisoxazole 2-oxide (trans-**3h**)*. Yellow oil; 41 mg, yield 62%; ee 88%, $[\alpha]_D^{25}$ (CH_2Cl_2, c = 0.5) = +72.4; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 (dd, *J* = 8.0, 1.0 Hz, 1H), 7.43–7.34 (m, 2H), 7.22–7.17 (m, 1H), 6.40 (s, 1H), 5.97 (s, 1H), 5.28 (d, *J* = 4.2 Hz, 1H), 5.19 (d, *J* = 4.3 Hz, 1H), 4.25 – 4.15 (m, 2H), 1.46 (s, 9H), 1.13 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.2, 158.3, 138.6, 138.2, 133.4, 129.82, 129.84, 128.6, 126.7, 123.6, 110.6, 82.6, 80.7, 61.8, 54.2, 27.9, 13.9; HRMS-ESI calcd for $C_{19}H_{23}BrNO_6$ $[M + H]^+$ 440.0703, found 440.0696. The enantiomeric excess of *trans*-**3h** was determined by HPLC analysis with a Chiralcel AS-H column (hexane/*i*-PrOH: 95/5, flow rate: 1.0 mL/min, λ = 254 nm, t_{major} = 13.7 min, t_{minor} = 12.1 min).



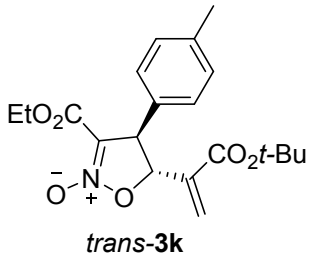
trans-**3i**

*5-(3-(tert-Butoxy)-3-oxoprop-1-en-2-yl)-3-(ethoxycarbonyl)-4-(4-fluorophenyl)-4,5-dihydroisoxazole 2-oxide (trans-**3i**)*. Yellow oil; 30 mg, yield 52%; ee > 99%, $[\alpha]_D^{25}$ (CH_2Cl_2, c = 0.5) = +59.2; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.31 (m, 2H), 7.12 – 7.05 (m, 2H), 6.38 (s, 1H), 6.01 (d, *J* = 0.8 Hz, 1H), 5.31 – 5.20 (m, 1H), 4.48 (d, *J* = 2.6 Hz, 1H), 4.25 – 4.15 (m, 2H), 1.47 (s, 9H), 1.19 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.5, 162.6 (d, *J* = 247.3 Hz), 158.5, 138.0, 134.8 (d, *J* = 3.4 Hz), 129.0, (d, *J* = 8.2 Hz), 125.6, 116.1 (d, *J* = 21.7 Hz), 110.1, 82.5, 80.7, 61.9, 54.6, 28.0, 13.9; HRMS-ESI calcd for $C_{38}H_{48}F_2N_3O_{12}$ $[2M + NH_4]^+$ 776.3201, found 776.3200. The enantiomeric excess of *trans*-**3i** was assessed by HPLC analysis with a Chiralcel AS-H column (hexane/*i*-PrOH: 95/5, flow rate: 1.0 mL/min, λ = 254 nm, t_{major} = 12.1 min, t_{minor} = 9.2 min).



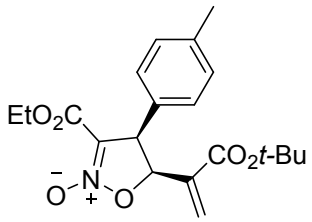
trans-**3j**

*5-(3-(tert-Butoxy)-3-oxoprop-1-en-2-yl)-3-(ethoxycarbonyl)-4-(3-fluorophenyl)-4,5-dihydroisoxazole 2-oxide (trans-**3j**)*. Yellow oil; 31 mg, yield 54%; ee 99%, $[\alpha]_D^{25}$ (CH_2Cl_2, c = 0.5) = +15.6; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40–7.34 (m, 1H), 7.17 (d, *J* = 7.8 Hz, 1H), 7.13 – 7.01 (m, 2H), 6.39 (s, 1H), 6.01 (d, *J* = 1.1 Hz, 1H), 5.31 – 5.28 (m, 1H), 4.49 (d, *J* = 2.7 Hz, 1H), 4.26 – 4.14 (m, 2H), 1.48 (s, 9H), 1.19 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.5, 163.0 (d, *J* = 247.4 Hz), 158.5, 141.3 (d, *J* = 7.1 Hz), 137.9, 130.7 (d, *J* = 8.2 Hz), 125.8, 122.9 (d, *J* = 2.8 Hz), 115.4 (d, *J* = 21.0 Hz), 114.6 (d, *J* = 22.4 Hz), 109.9, 82.6, 80.5, 62.0, 55.0, 28.0, 14.0; HRMS-ESI calcd for $C_{38}H_{48}F_2N_3O_{12}$ $[2M + NH_4]^+$ 776.3200, found 776.3201. The enantiomeric excess of *trans*-**3j** was measured by HPLC analysis with a Chiralcel AS-H column (hexane/*i*-PrOH: 95/5, flow rate: 1.0 mL/min, λ = 254 nm, t_{major} = 10.1 min, t_{minor} = 8.1 min).



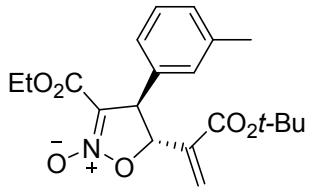
trans-**3k**

*5-(3-(tert-Butoxy)-3-oxoprop-1-en-2-yl)-3-(ethoxycarbonyl)-4-(p-tolyl)-4,5-dihydroisoxazole 2-oxide (trans-**3k**)*. Yellow oil; 24 mg, yield 42%; ee > 99%, $[\alpha]_D^{25}$ (CH_2Cl_2, c = 0.5) = +69.6; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 (d, *J* = 8.1 Hz, 2H), 7.18 (d, *J* = 8.0 Hz, 2H), 6.36 (s, 1H), 5.98 (d, *J* = 0.7 Hz, 1H), 5.29 – 5.24 (m, 1H), 4.45 (d, *J* = 2.8 Hz, 1H), 4.25 – 4.15 (m, 2H), 2.35 (s, 3H), 1.47 (s, 9H), 1.18 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.5, 158.7, 138.3, 138.1, 136.0, 129.8, 127.2, 125.6, 110.6, 82.4, 81.0, 61.8, 55.0, 28.0, 21.2, 14.0; HRMS-ESI calcd for $C_{40}H_{54}N_3O_{12}$ $[2M + NH_4]^+$ 768.3702, found 768.3707. The enantiomeric excess of *trans*-**3k** was determined by HPLC analysis with a Chiralcel AS-H column (hexane/*i*-PrOH: 95/5, flow rate: 1.0 mL/min, λ = 254 nm, t_{major} = 6.9 min, t_{minor} = 6.4 min).



cis-**3k**

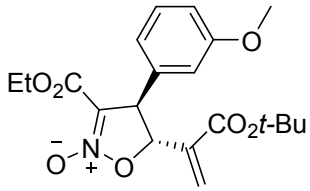
*5-(3-(tert-Butoxy)-3-oxoprop-1-en-2-yl)-3-(ethoxycarbonyl)-4-(p-tolyl)-4,5-dihydroisoxazole 2-oxide (cis-**3k**)*. Yellow oil; 12 mg, yield 21%; ee > 99%, $[\alpha]_D^{25}$ (CH_2Cl_2, c = 0.5) = -66.8; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.05 (d, *J* = 7.9 Hz, 2H), 6.94 (d, *J* = 8.1 Hz, 2H), 6.04 (s, 1H), 5.74 (d, *J* = 8.1 Hz, 1H), 5.72 (s, 1H), 4.90 (d, *J* = 8.1 Hz, 1H), 4.25 – 4.15 (m, 2H), 2.30 (s, 3H), 1.42 (s, 9H), 1.15 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.3, 158.6, 138.0, 134.0, 131.7, 129.2, 128.5, 126.3, 113.0, 81.8, 78.3, 61.8, 51.9, 27.9, 21.2, 13.9. HRMS-ESI calcd for $C_{40}H_{54}N_3O_{12}$ $[2M + NH_4]^+$ 768.3702, found 768.3707. The enantiomeric excess of *cis*-**3k** was determined by HPLC analysis with a Chiralcel AD-H column (hexane/*i*-PrOH: 95/5, flow rate: 1.0 mL/min, λ = 254 nm, t_{major} = 7.6 min, t_{minor} = 11.3 min).



trans-**3l**

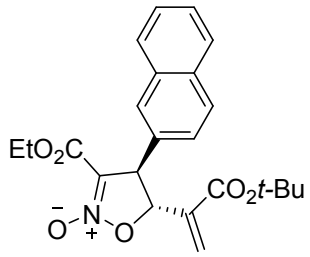
*5-(3-(tert-Butoxy)-3-oxoprop-1-en-2-yl)-3-(ethoxycarbonyl)-4-(m-tolyl)-4,5-dihydroisoxazole 2-oxide (trans-**3l**)*. Yellow oil; 35 mg, yield 62%; ee > 99%, $[\alpha]_D^{25}$ (CH_2Cl_2, c = 0.5) = +76; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 – 7.24 (m, 1H), 7.17 – 7.12 (m, 3H), 6.37 (s, 1H), 5.98 (d, *J* = 1.0 Hz, 1H), 5.30 – 5.26 (m, 1H), 4.44 (d, *J* = 2.9 Hz, 1H), 4.24 – 4.13 (m, 2H), 2.36 (s, 3H), 1.46 (s, 9H), 1.18 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.5, 158.7, 138.9, 138.8, 138.3, 129.1, 128.9, 127.8, 125.7, 124.5, 110.6, 82.4, 80.9, 61.8, 55.3, 28.0, 21.4,

13.9; HRMS-ESI calcd for $C_{40}H_{54}N_3O_{12}$ [2M + NH_4]$^+$ 768.3702, found 768.3714. The enantiomeric excess of *trans*-**3l** was measured by HPLC analysis with a Chiralcel OD-H column (hexane/*i*-PrOH: 95/5, flow rate: 1.0 mL/min, λ = 254 nm, t_{major} = 5.5 min, t_{minor} = 6.0 min).



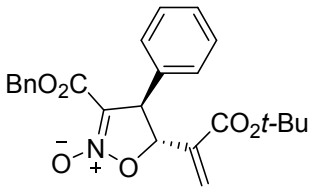
trans-**3m**

*5-(3-(tert-Butoxy)-3-oxoprop-1-en-2-yl)-3-(ethoxycarbonyl)-4-(3-methoxyphenyl)-4,5-dihydroisoxazole 2-oxide (trans-**3m**)*. Yellow oil; 40 mg, yield 68%; ee > 99%, $[\alpha]_D^{25}$ (CH_2Cl_2, c = 0.5) = +76.8; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 (t, *J* = 7.9 Hz, 1H), 6.94 (d, *J* = 7.7 Hz, 1H), 6.91 – 6.85 (m, 2H), 6.37 (s, 1H), 5.98 (s, 1H), 5.33 – 5.25 (m, 1H), 4.46 (d, *J* = 2.8 Hz, 1H), 4.25 – 4.14 (m, 2H), 3.82 (s, 3H), 1.47 (s, 9H), 1.19 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.5, 160.1, 158.7, 140.4, 138.2, 130.1, 125.8, 119.6, 113.5, 113.2, 110.4, 82.5, 80.8, 77.4, 61.8, 55.3, 28.0, 14.0; HRMS-ESI calcd for $C_{20}H_{26}NO_7$ [M + H]$^+$ 392.1704, found 392.1698. The enantiomeric excess of *trans*-**3m** was determined by HPLC analysis with a Chiralcel AS-H column (hexane/*i*-PrOH: 95/5, flow rate: 1.0 mL/min, λ = 254 nm, t_{major} = 22.6 min, t_{minor} = 16.5 min).



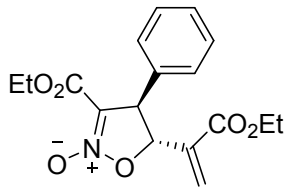
trans-**3n**

*5-(3-(tert-Butoxy)-3-oxoprop-1-en-2-yl)-3-(ethoxycarbonyl)-4-(naphthalen-2-yl)-4,5-dihydroisoxazole 2-oxide (trans-**3n**)*. Yellow oil; 33 mg, yield 53%; ee 91%, $[\alpha]_D^{25}$ (CH_2Cl_2, c = 0.5) = +61.6; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, *J* = 8.2 Hz, 1H), 7.93 – 7.80 (m, 2H), 7.59 – 7.46 (m, 4H), 6.44 (s, 1H), 5.98 (s, 1H), 5.43 – 5.34 (m, 2H), 4.04 (q, *J* = 7.1 Hz, 2H), 1.30 (s, 9H), 0.93 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.4, 158.6, 138.8, 135.3, 134.0, 130.8, 129.10, 129.06, 126.9, 126.6, 126.1, 125.7, 124.4, 123.0, 111.2, 82.6, 81.4, 61.7, 46.3, 27.7, 13.7; HRMS-ESI calcd for $C_{23}H_{26}NO_6$ [M + H]$^+$ 412.1755, found 412.1749. The enantiomeric excess of *trans*-**3n** was measured by HPLC analysis with a Chiralcel AS-H column (hexane/*i*-PrOH: 95/5, flow rate: 1.0 mL/min, λ = 254 nm, t_{major} = 21.5 min, t_{minor} = 17.8 min). HPLC analysis showed that the sample of *trans*-**3n** contained a small amount of single *cis*-**3n** diastereomer.



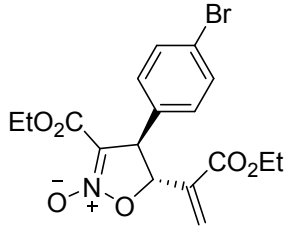
trans-**3o**

*3-((Benzyloxy)carbonyl)-5-(3-(tert-butoxy)-3-oxoprop-1-en-2-yl)-4-phenyl-4,5-dihydroisoxazole 2-oxide (trans-**3o**)*. Yellow semi-solid, m.p. 57–59 °C; ee 99%, $[\alpha]_D^{25}$ (CH_2Cl_2, c = 0.5) = +23.6; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 – 7.25 (m, 8H), 7.10–7.06 (m, 2H), 6.36 (s, 1H), 5.98 (d, *J* = 0.9 Hz, 1H), 5.29 (d, *J* = 2.8 Hz, 1H), 5.21 (d, *J* = 12.5 Hz, 1H), 5.07 (d, *J* = 12.5 Hz, 1H), 4.50 (d, *J* = 3.0 Hz, 1H), 1.44 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.5, 158.5, 138.9, 138.1, 134.8, 129.2, 128.5, 128.3, 128.0, 127.7, 127.4, 125.9, 110.3, 82.5, 81.0, 67.2, 55.2, 28.0; HRMS-ESI calcd for $C_{24}H_{26}NO_6$ $[M + H]^+$ 424.1755, found 424.1756. The enantiomeric excess of *trans*-**3o** was determined by HPLC analysis with a Chiralcel AS-H column (hexane/*i*-PrOH: 95/5, flow rate: 1.0 mL/min, λ = 254 nm, t_{major} = 18.8 min, t_{minor} = 14.3 min). HPLC analysis showed that the sample of *trans*-**3o** contained a small amount of single *cis*-**3o** diastereomer.



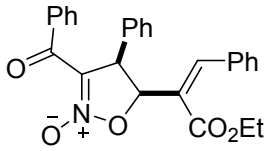
trans-**3p**

*5-(3-Ethoxy-3-oxoprop-1-en-2-yl)-3-(ethoxycarbonyl)-4-phenyl-4,5-dihydroisoxazole 2-oxide (trans-**3p**)*. Colorless oil; 26 mg, yield 52%; ee > 99%, $[\alpha]_D^{25}$ (CH_2Cl_2, c = 0.5) = +52; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 – 7.33 (m, 5H), 6.48 (s, 1H), 6.11 (d, *J* = 1.1 Hz, 1H), 5.30 (s, 1H), 4.53 (d, *J* = 2.5 Hz, 1H), 4.27 (q, *J* = 7.1 Hz, 2H), 4.20 (q, *J* = 7.1 Hz, 2H), 1.29 (t, *J* = 7.1 Hz, 3H), 1.16 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.4, 158.6, 138.8, 136.8, 129.1, 128.3, 127.1, 126.4, 110.2, 80.6, 61.8, 61.4, 55.1, 14.0, 13.9; HRMS-ESI calcd for $C_{17}H_{20}NO_6$ $[M + H]^+$ 334.1285, found 334.1287. The enantiomeric excess of *trans*-**3p** was determined by HPLC analysis with a Chiralcel AS-H column (hexane/*i*-PrOH: 95/5, flow rate: 1.0 mL/min, λ = 254 nm, t_{major} = 19.3 min, t_{minor} = 14.3 min).



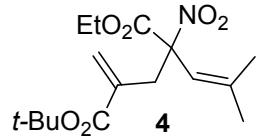
trans-**3q**

*4-(4-Bromophenyl)-5-(3-ethoxy-3-oxoprop-1-en-2-yl)-3-(ethoxycarbonyl)-4,5-dihydroisoxazole 2-oxide (trans-**3q**)*. Yellow oil; 28 mg, yield 45%; ee 98%, $[\alpha]_D^{25}$ (CH_2Cl_2, c = 0.5) = +14.8; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (d, *J* = 8.4 Hz, 2H), 7.26 (d, *J* = 8.7 Hz, 2H), 6.48 (s, 1H), 6.12 (d, *J* = 1.4 Hz, 1H), 5.26 – 5.22 (m, 1H), 4.50 (d, *J* = 2.4 Hz, 1H), 4.31 – 4.24 (m, 2H), 4.23 – 4.15 (m, 2H), 1.30 (t, *J* = 7.1 Hz, 3H), 1.19 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.3, 158.4, 137.9, 136.5, 132.3, 128.9, 126.4, 122.4, 109.7, 80.2, 62.0, 61.5, 54.6, 14.1, 13.9; HRMS-ESI calcd for $C_{17}H_{19}BrNO_6$ $[M + H]^+$ 412.0390, found 412.0383. The enantiomeric excess of *trans*-**3q** was determined by HPLC analysis with a Chiralcel AS-H column (hexane/*i*-PrOH: 95/5, flow rate: 1.0 mL/min, λ = 254 nm, t_{major} = 19.1 min, t_{minor} = 14.7 min).



***cis*-3r**

*3-Benzoyl-5-(3-ethoxy-3-oxo-1-phenylprop-1-en-2-yl)-4-phenyl-4,5-dihydroisoxazole 2-oxide (cis-**3r**)*. White solid, m.p. 149–151 °C; 25 mg, yield 19%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (s, 1H), 7.79 (d, *J* = 7.7 Hz, 2H), 7.55 (t, *J* = 7.4 Hz, 1H), 7.44 (t, *J* = 7.7 Hz, 2H), 7.41 – 7.32 (m, 5H), 7.24 (d, *J* = 7.7 Hz, 1H), 7.09 (t, *J* = 7.7 Hz, 2H), 6.79 (d, *J* = 7.7 Hz, 2H), 5.66 (d, *J* = 6.9 Hz, 1H), 5.37 (d, *J* = 6.9 Hz, 1H), 4.38 (q, *J* = 7.1 Hz, 2H), 1.34 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 185.2, 165.3, 146.1, 138.4, 135.5, 133.4, 132.9, 129.5, 129.0, 128.9, 128.5, 128.4 (2C), 128.0, 118.6, 79.3, 61.7, 55.8, 14.1; HRMS-ESI calcd for $C_{27}H_{24}NO_5$ $[M + H]^+$ 442.1649, found 442.1652.



4

*5-(tert-Butyl)-1-ethyl-4-methylene-2-(2-methylprop-1-en-1-yl)-2-nitropentanedioate (**4**)*. Colorless oil; 40 mg, yield 82%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.18 (s, 1H), 5.80 (s, 1H), 5.49 (s, 1H), 4.32 – 4.21 (m, 2H), 3.48 (d, *J* = 14.2 Hz, 1H), 3.34 (d, *J* = 14.2 Hz, 1H), 1.80 (s, 3H), 1.62 (s, 3H), 1.47 (s, 9H), 1.28 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.3, 165.5, 141.0, 135.2, 128.8, 117.8, 94.9, 81.1, 62.9, 37.6, 27.8, 27.4, 18.8, 13.7; HRMS-ESI calcd for $C_{16}H_{29}N_2O_6$ $[M + NH_4]^+$ 345.2020, found 345.2022.

VI. ORTEP Drawing for *trans*-3o

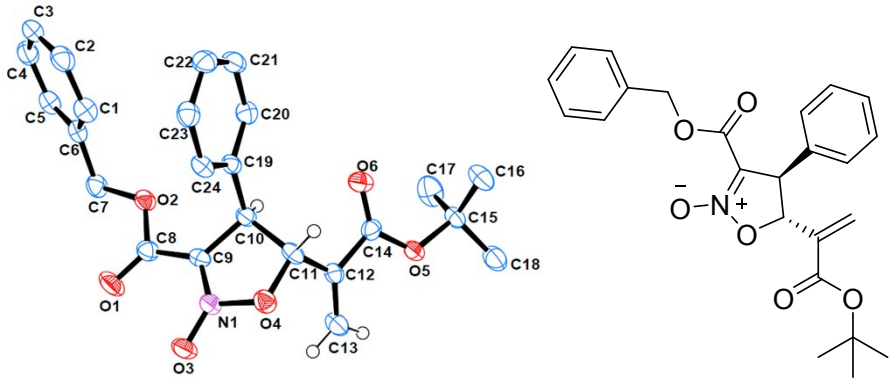
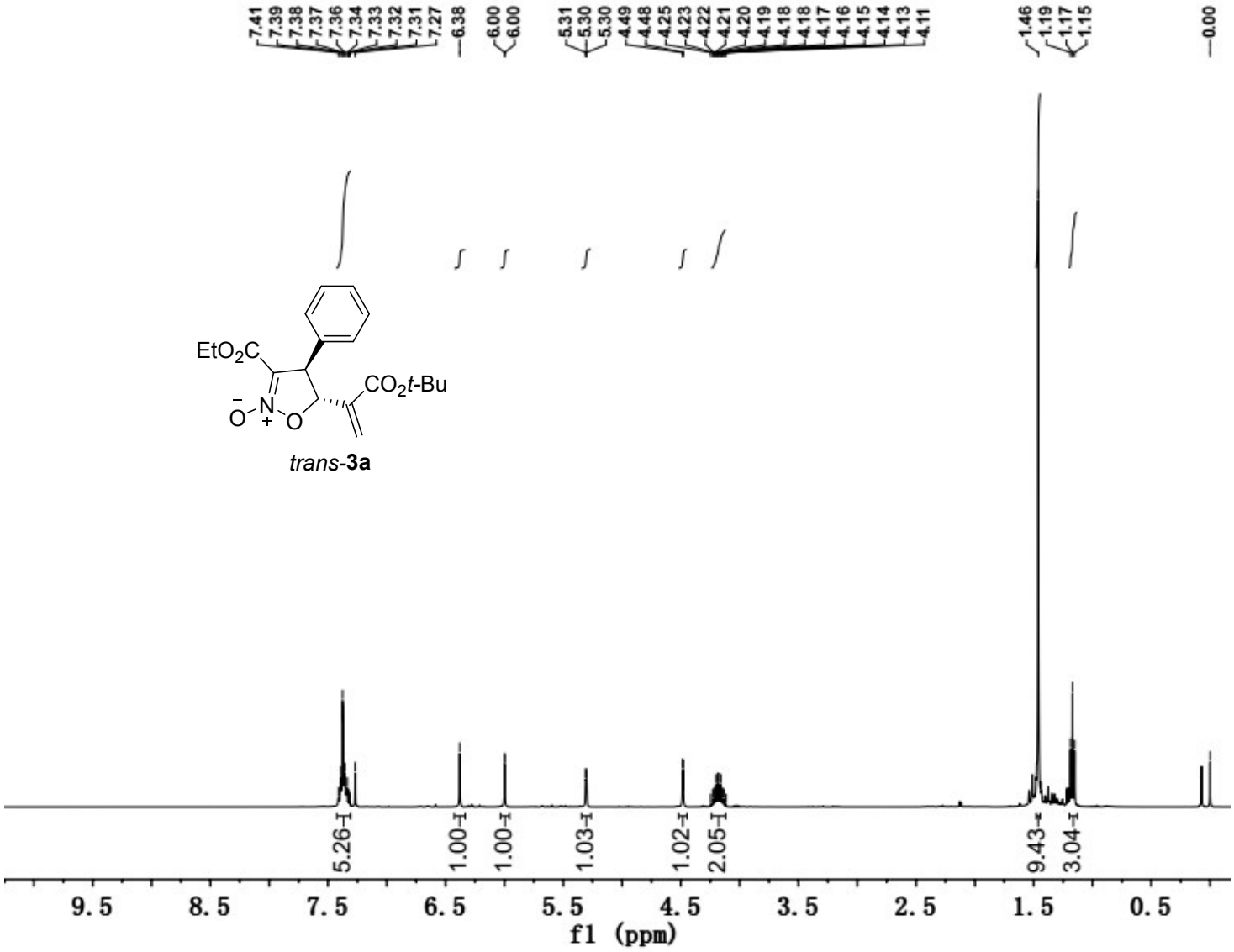
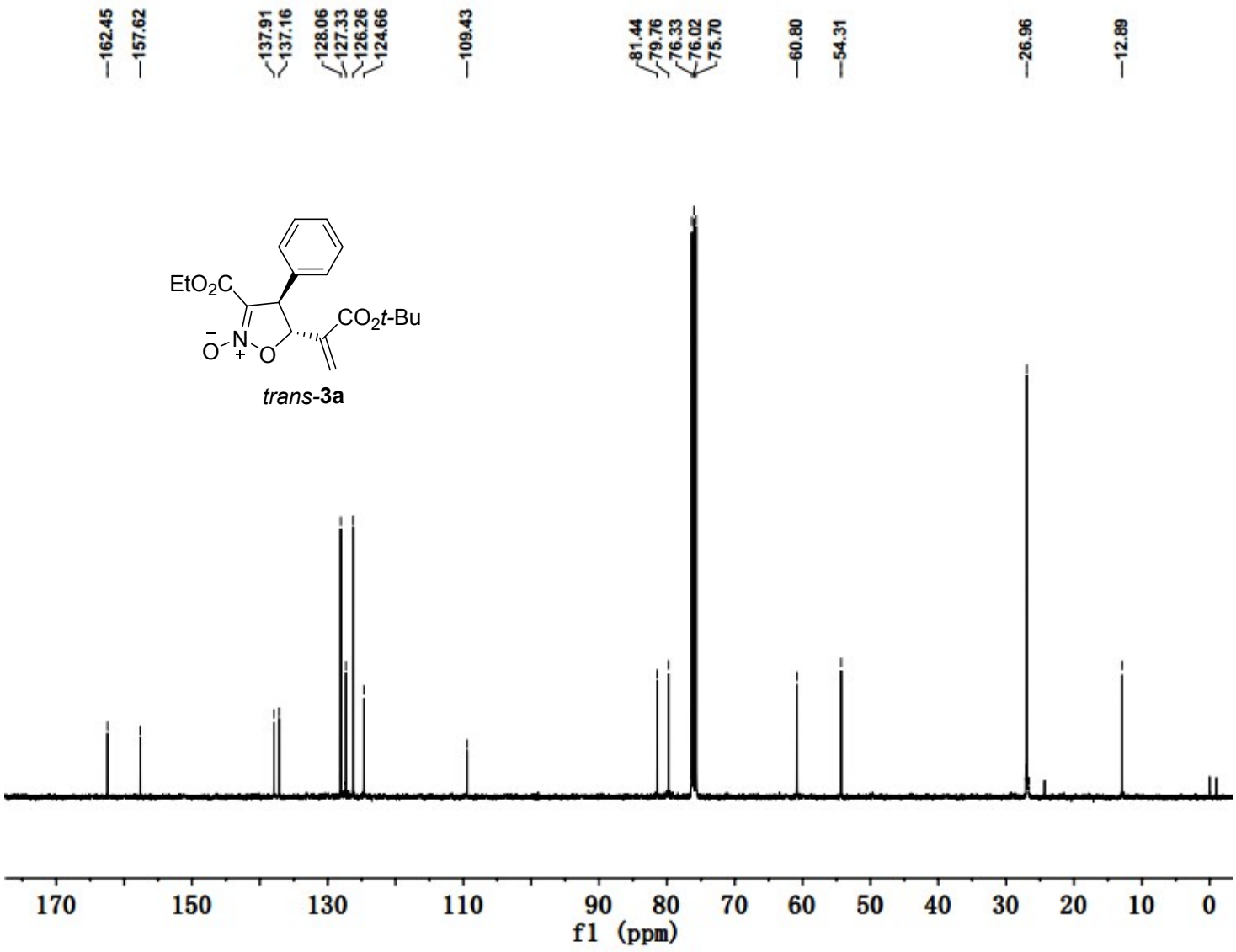


Figure S1. ORTEP Drawing for *trans*-**3o** with 50% ellipsoid probability

Identification code	*trans*-**3o**
Empirical formula	$C_{24}H_{25}NO_6$
Formula weight	423.45
Temperature	113(2) K
Wavelength	0.71073 Å
Crystal system, space group	Orthorhombic, P2(1)2(1)2(1)
Unit cell dimensions	a = 5.530(2)Å, α= 90°
	b = 12.216(4)Å, β= 90°
	c = 32.227(13)Å, γ= 90°
Volume	2177.1(14) $Å^3$
Z, Calculated density	4, 1.292 Mg/m^3
Absorption coefficient	0.093 mm^{-1}
F(000)	896
Crystal size	0.20 x 0.18 x 0.12 mm^3
Theta range for data collection	3.03 to 25.69°
Limiting indices	-6<=h<=6,-14<=k<=14,-28<=l<=39
Reflections collected / unique	19718 / 4121 [R(int) = 0.0960]
Completeness to the θ = 27.50°	99.6 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9889 and 0.9816
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4121 / 0 / 283
Goodness-of-fit on F^2	1.164
Final R indices [I>2σ(I)]	R_1 = 0.0585, wR_2 = 0.1060
R indices (all data)	R_1 = 0.0916, wR_2 = 0.1182
Largest diff. peak and hole	0.241 and -0.210 e. $Å^{-3}$

VII. NMR Spectra for 3a-3r and 4

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126.26
124.66
109.43
81.44
79.76
76.33
76.02
75.70
60.80
54.31
26.96
12.89
EtO2C
CO2t-Bu
trans-3a
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150
130
110
90
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50
40
30
20
10
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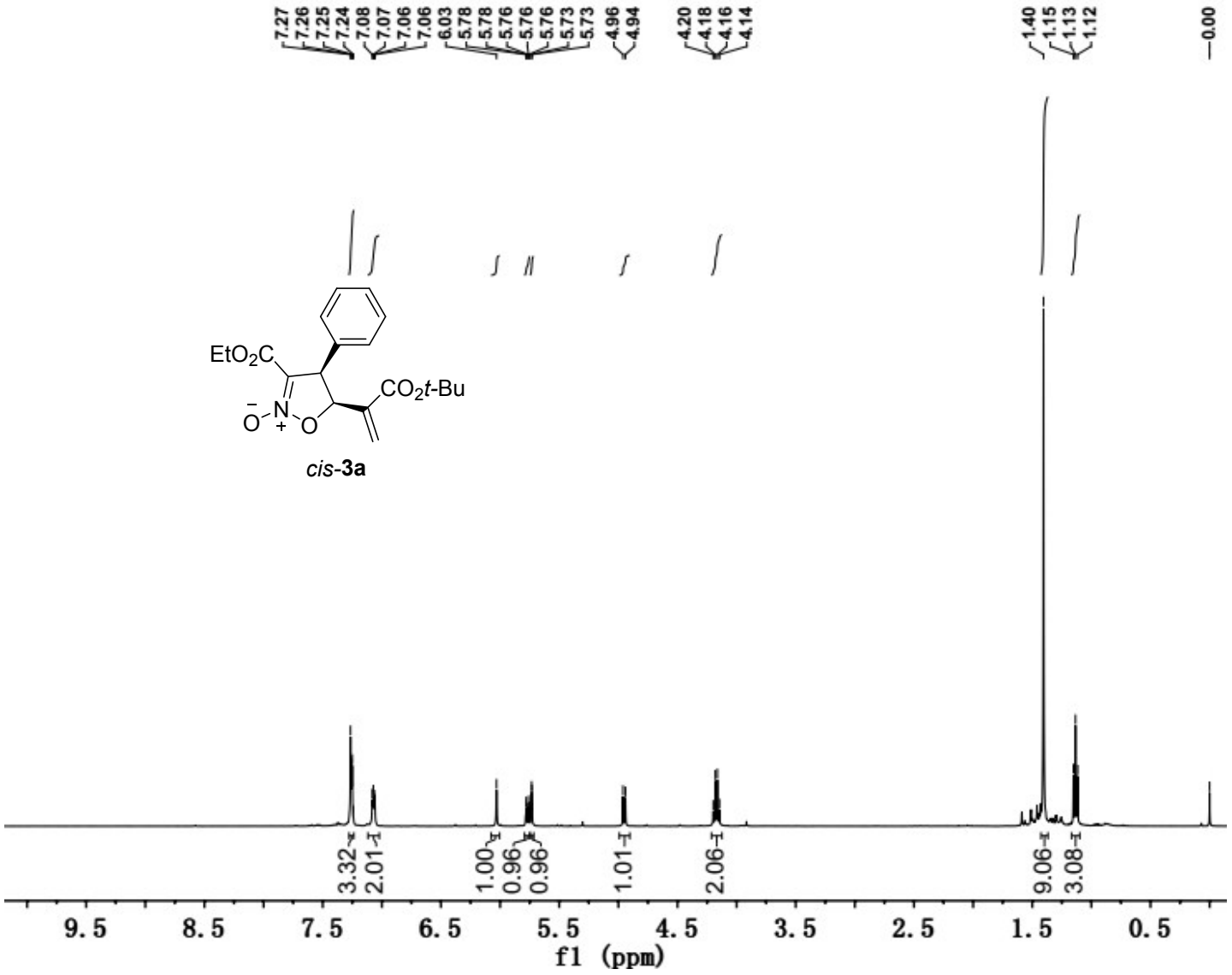
f1 (ppm)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7.06
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5.76
5.76
5.73
5.73
4.96
4.94
4.20
4.18
4.16
4.14
1.40
1.15
1.13
1.12
0.00
EtO2C
CO2t-Bu
cis-3a
3.32
2.01
1.00
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0.96
1.01
2.06
9.06
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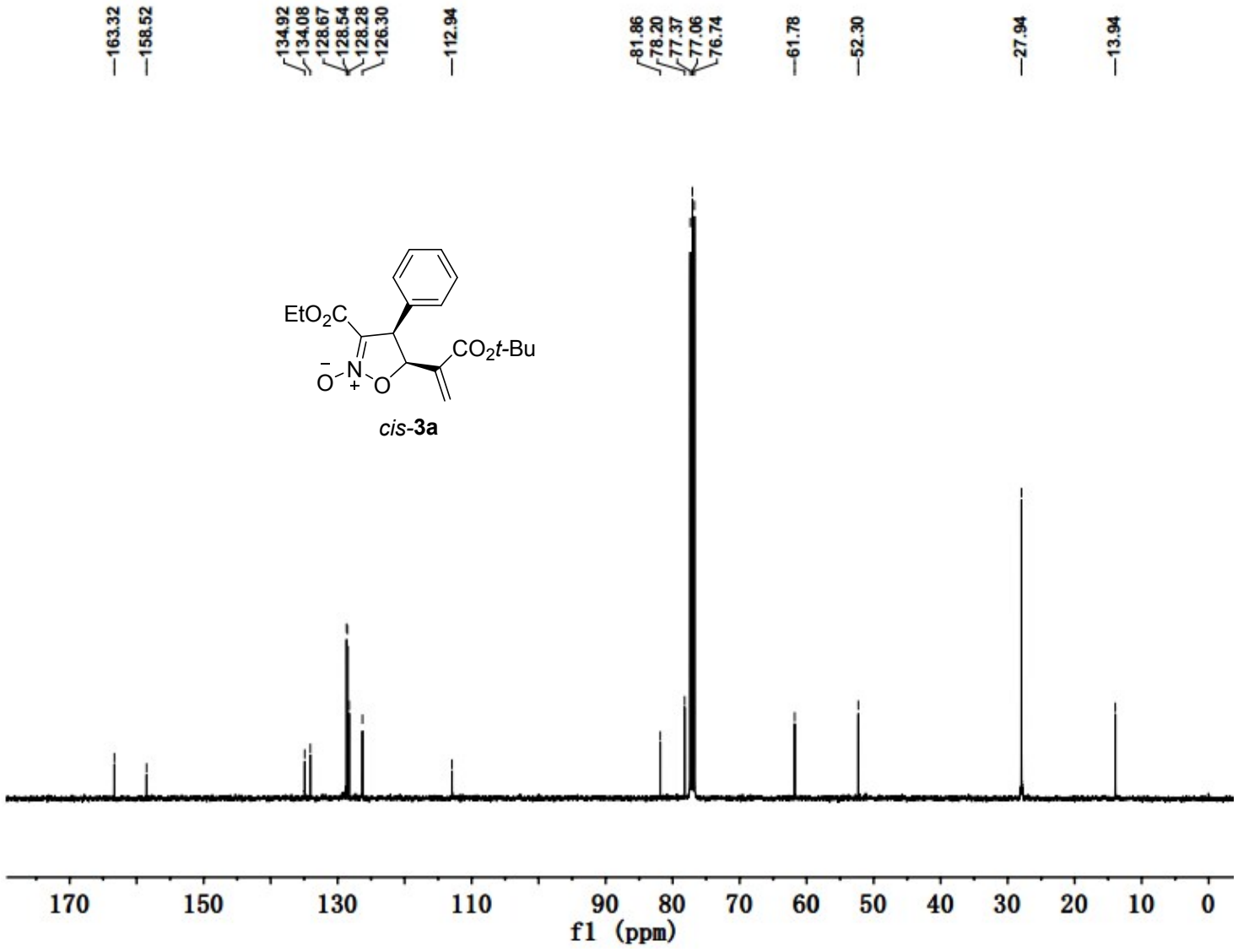
f1 (ppm)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77.06
76.74
61.78
52.30
27.94
13.94
EtO2C
CO2t-Bu
O
N
O
cis-3a
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130
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90
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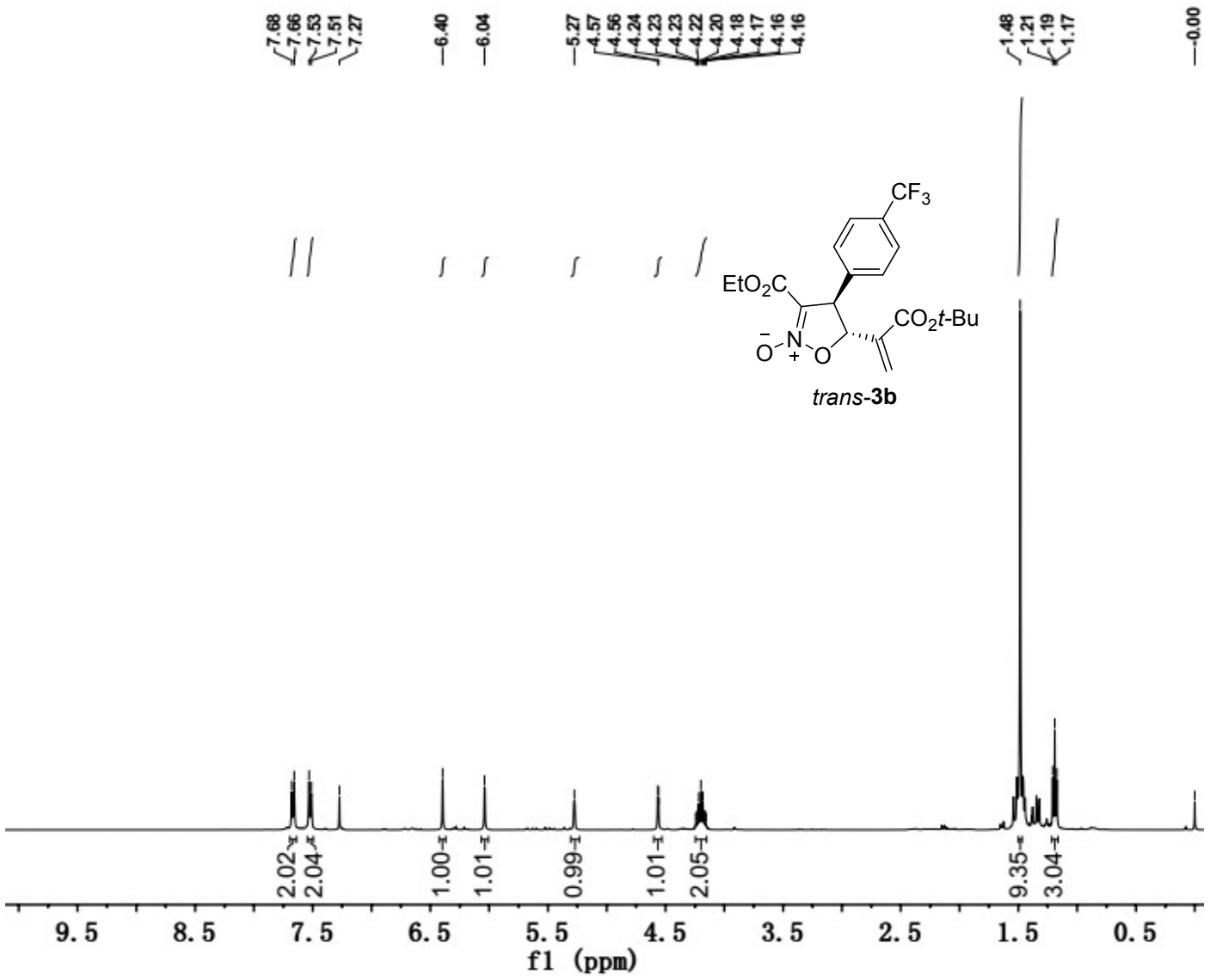
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4.23
4.22
4.20
4.18
4.17
4.16
4.16
1.48
1.21
1.19
1.17
0.00
CF3
EtO2C
CO2t-Bu
trans-3b
2.02
2.04
1.00
1.01
0.99
1.01
2.05
9.35
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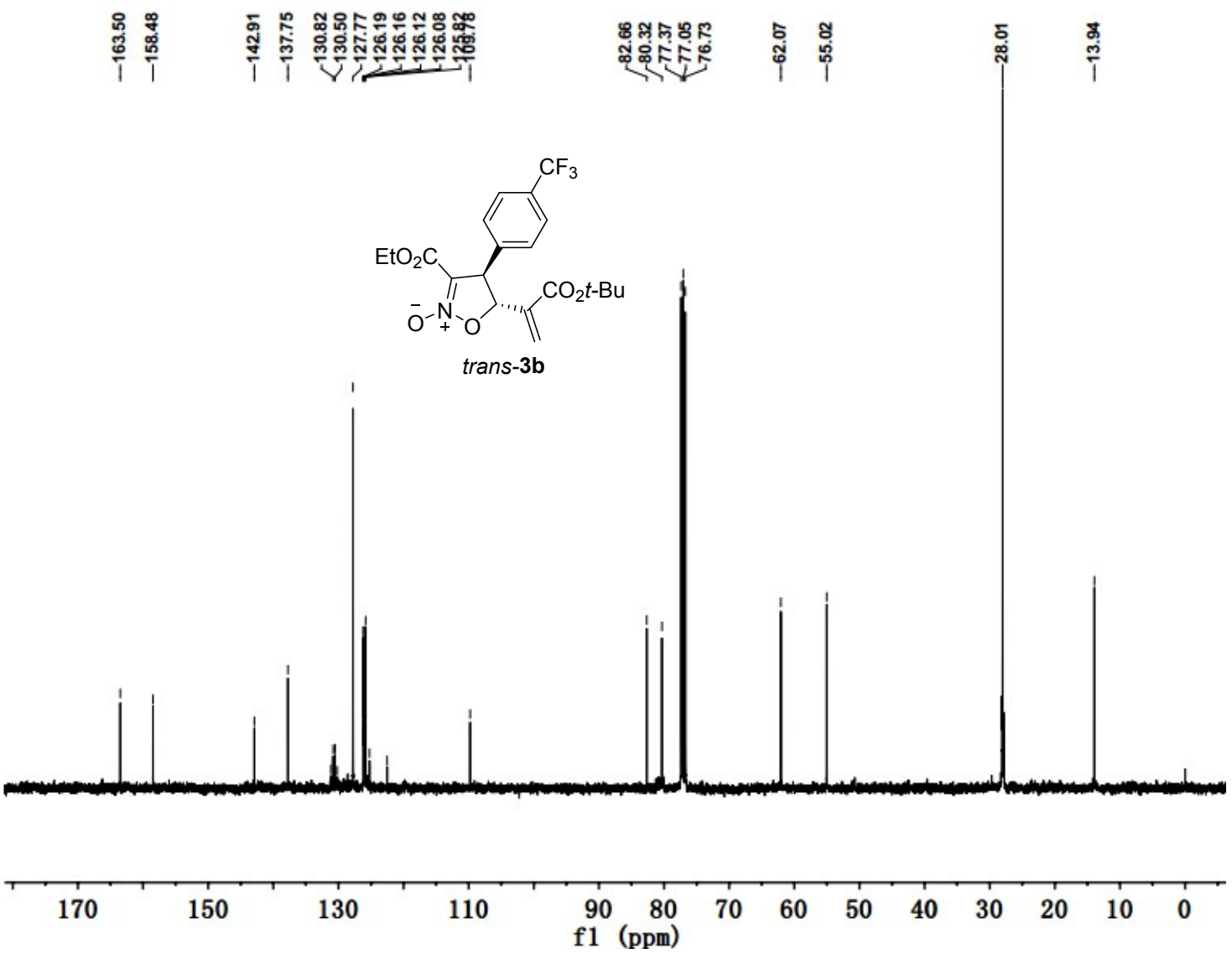
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77.37
77.05
76.73
62.07
55.02
28.01
13.94
CF3
EtO2C
CO2t-Bu
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130
110
90
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50
40
30
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10
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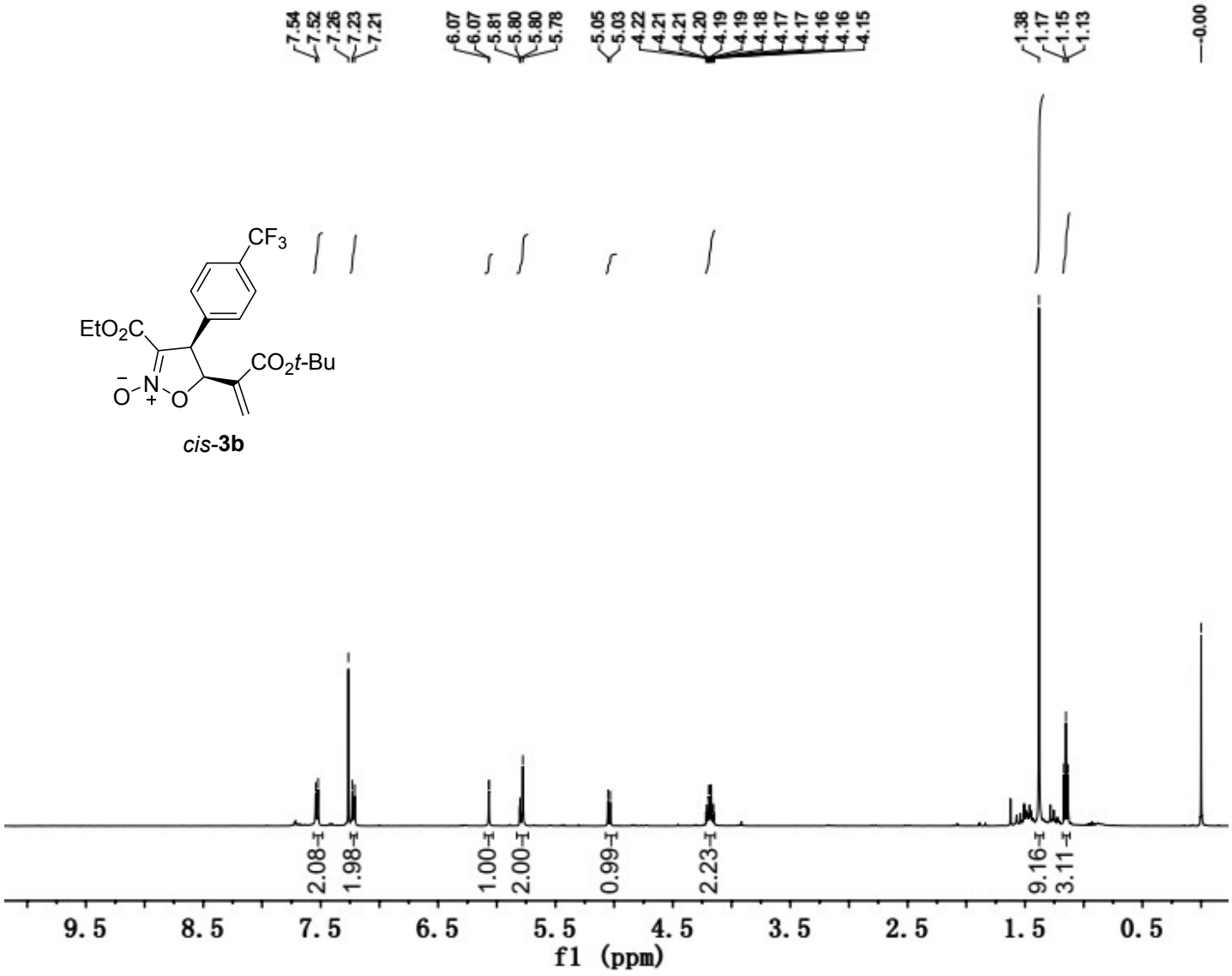
f1 (ppm)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5.80
5.78
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4.21
4.21
4.20
4.19
4.19
4.18
4.17
4.17
4.16
4.16
4.15
1.38
1.17
1.15
1.13
0.00
CF3
EtO2C
CO2t-Bu
cis-3b
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2.00
0.99
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2.5
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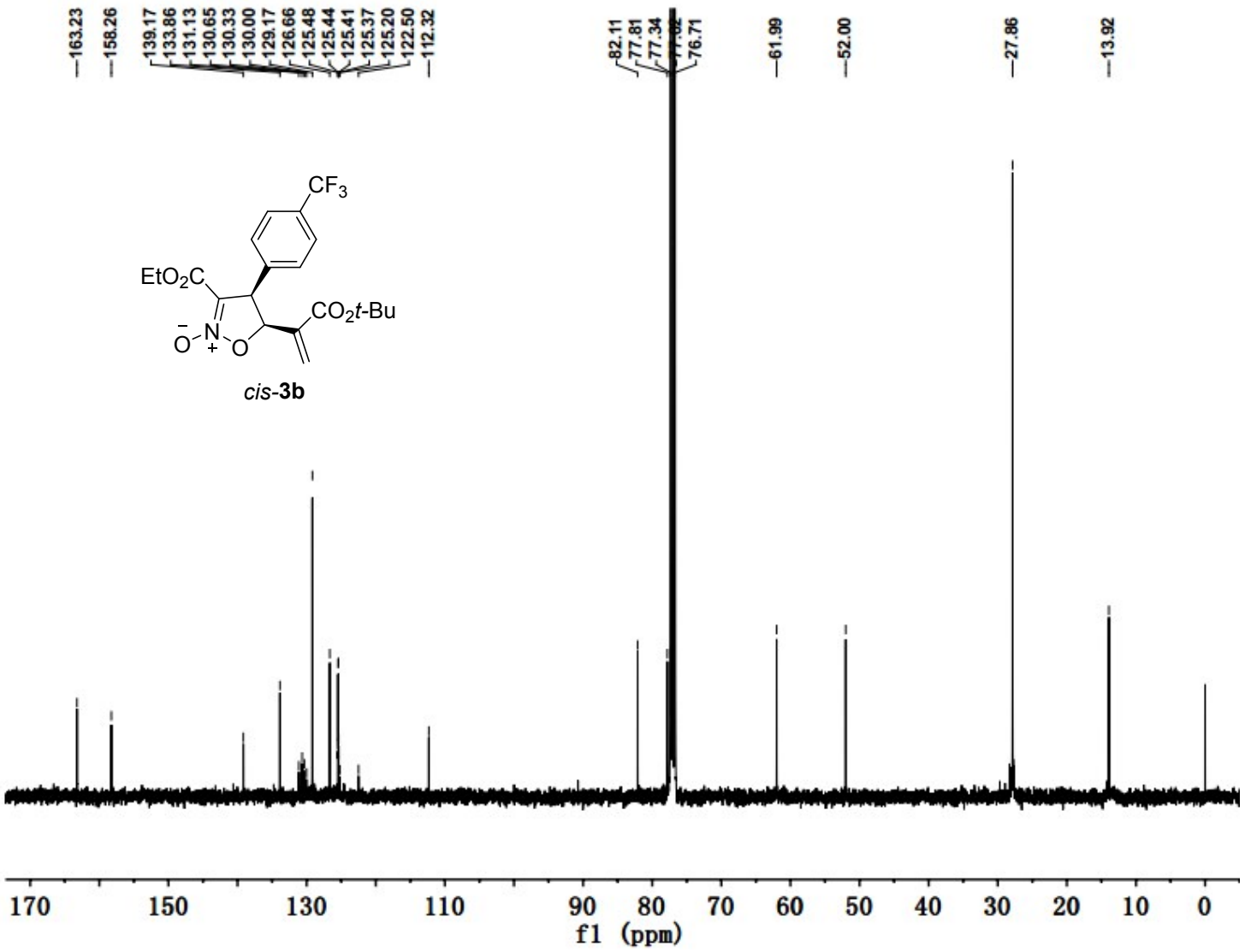
f1 (ppm)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125.41
125.37
125.20
122.50
112.32
82.11
77.81
77.34
76.71
61.99
52.00
27.86
13.92
CF3
EtO2C
CO2t-Bu
cis-3b
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150
130
110
90
80
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50
40
30
20
10
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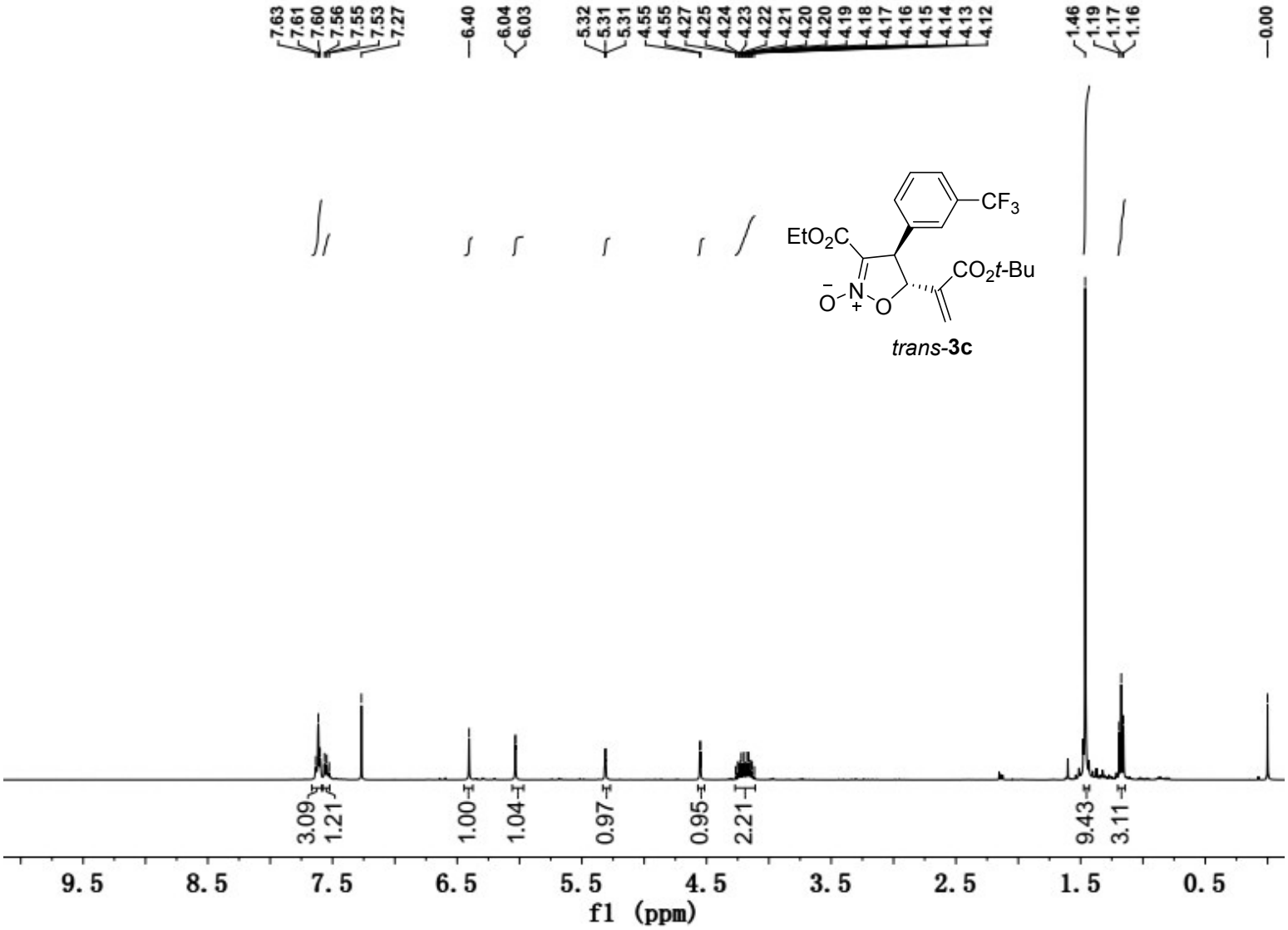
f1 (ppm)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4.19
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4.16
4.15
4.14
4.13
4.12
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1.19
1.17
1.16
0.00
CF3
EtO2C
CO2t-Bu
trans-3c
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1.00
1.04
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2.5
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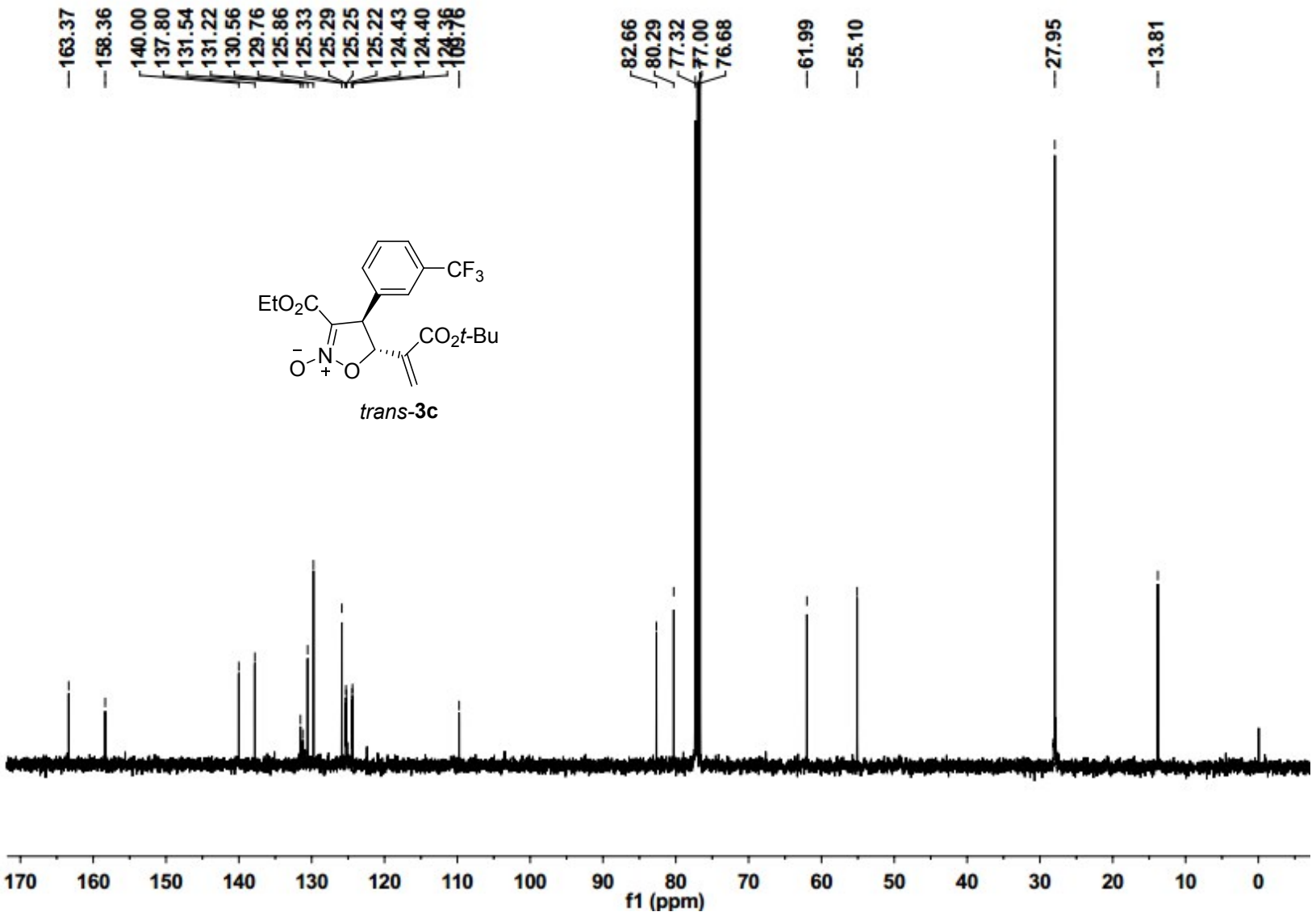
f1 (ppm)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80.29
77.32
77.00
76.68
61.99
55.10
27.95
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CF3
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CO2t-Bu
trans-3c
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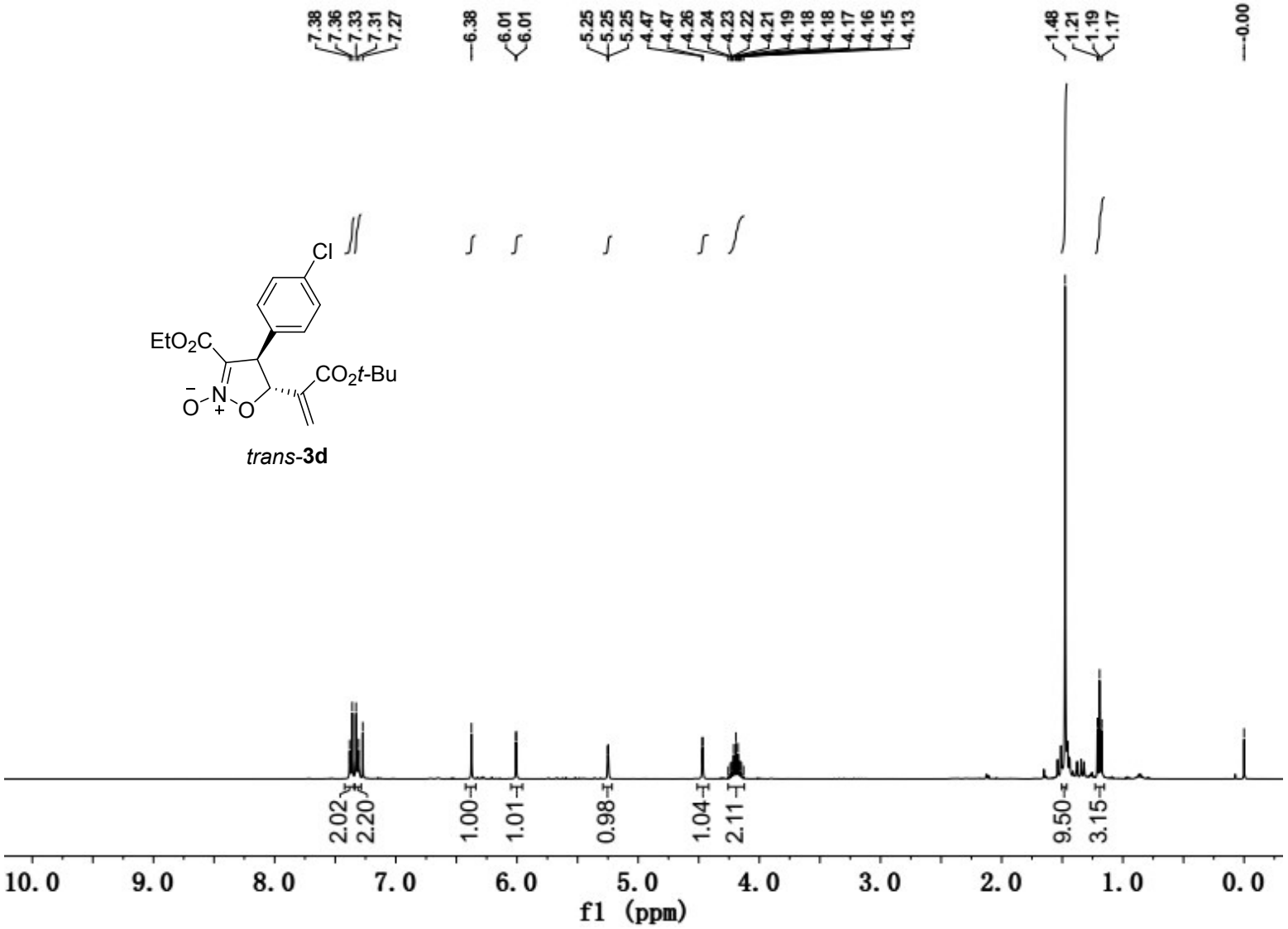
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4.26
4.24
4.23
4.22
4.21
4.19
4.18
4.18
4.17
4.16
4.15
4.13
1.48
1.21
1.19
1.17
0.00
Cl
EtO2C
CO2t-Bu
O
N
O
2.02
2.20
1.00
1.01
0.98
1.04
2.11
9.50
3.15
10.0
9.0
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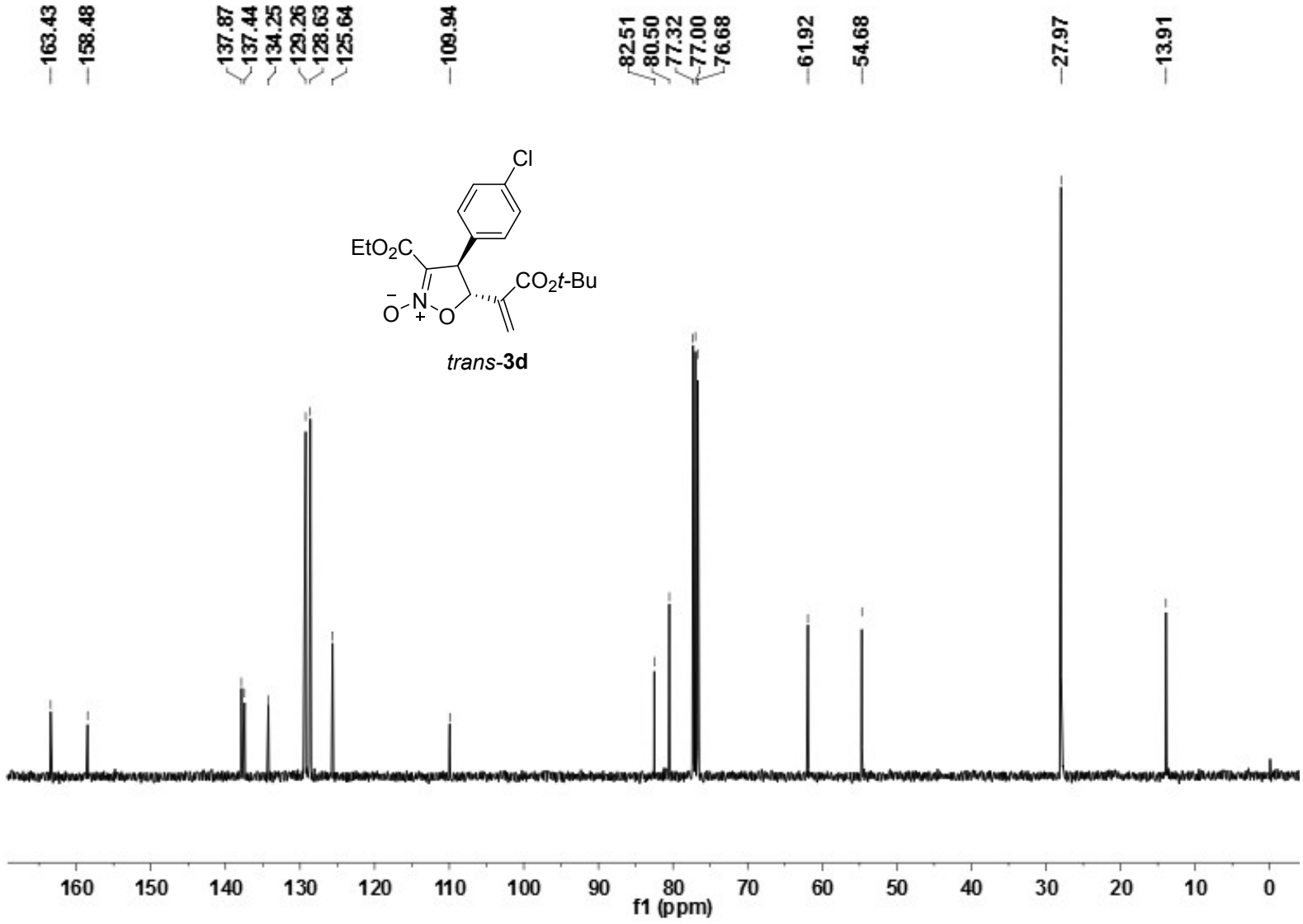
f1 (ppm)

trans-**3d**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76.68
61.92
54.68
27.97
13.91
Cl
EtO2C
CO2t-Bu
O
N
O
trans-3d
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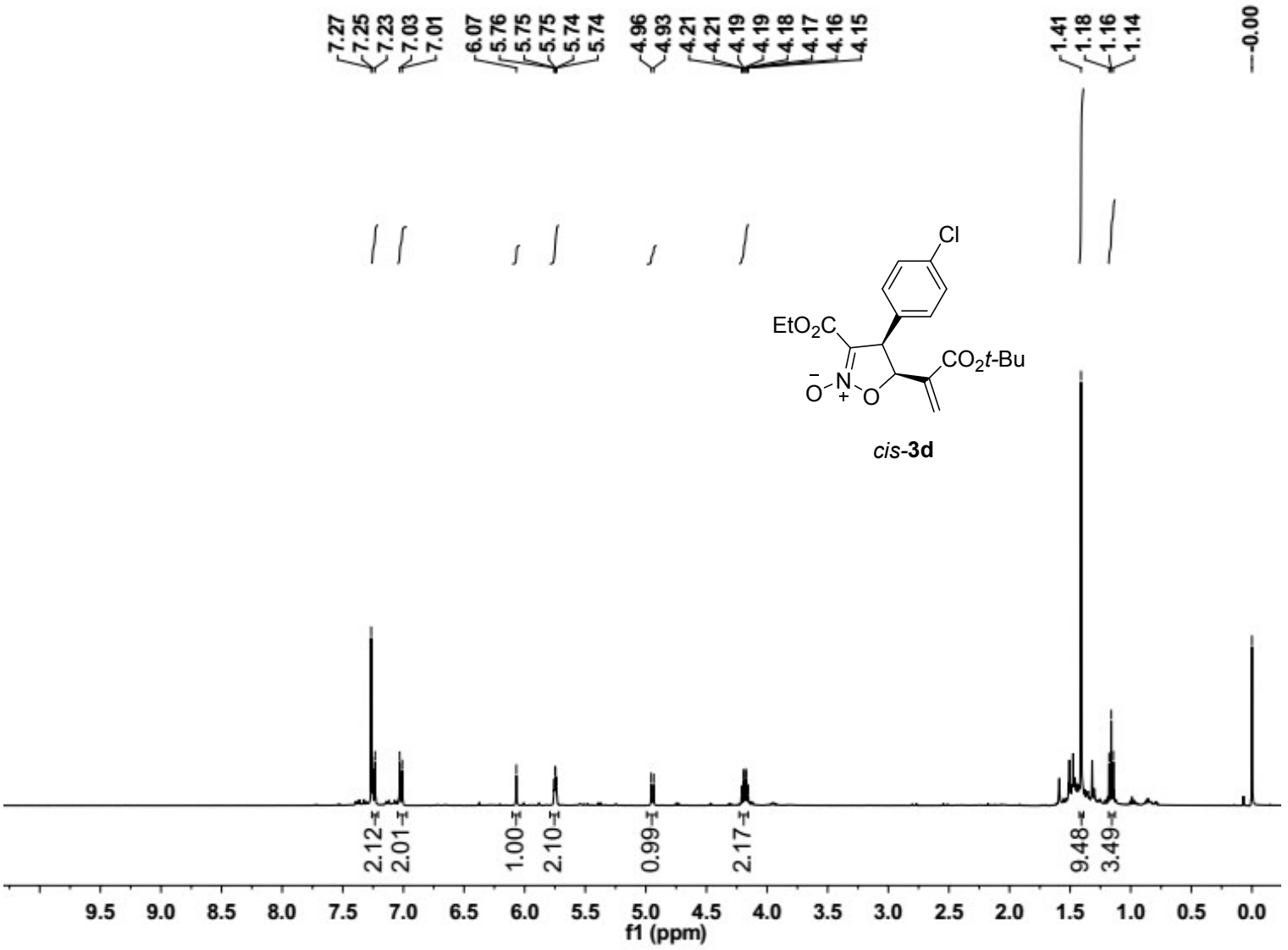
f1 (ppm)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4.21
4.19
4.19
4.18
4.17
4.16
4.15
1.41
1.18
1.16
1.14
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EtO2C
CO2t-Bu
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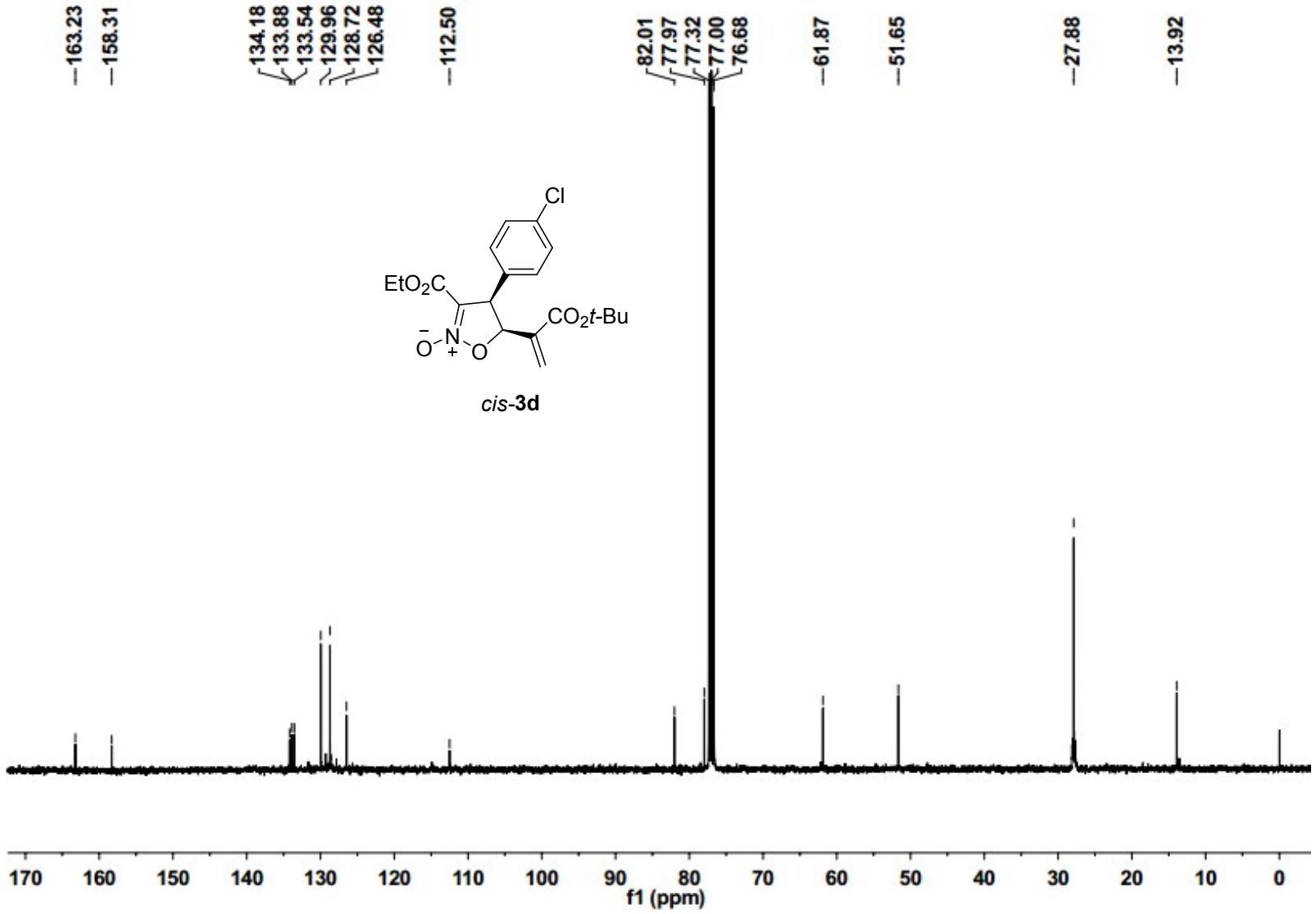
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61.87
51.65
27.88
13.92
Cl
EtO2C
CO2t-Bu
cis-3d
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50
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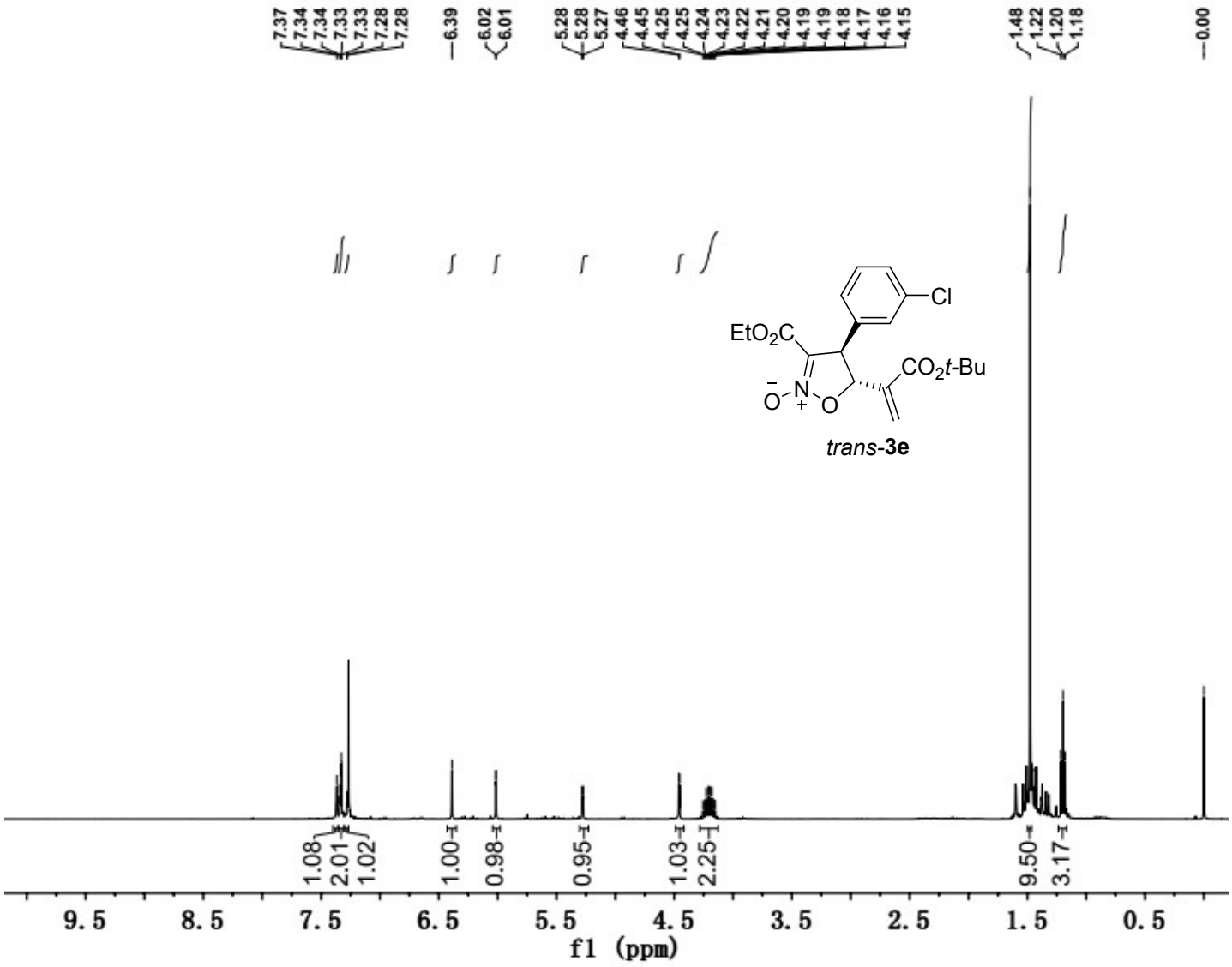
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4.24
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4.21
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4.19
4.19
4.18
4.17
4.16
4.15
1.48
1.22
1.20
1.18
0.00
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EtO2C
CO2t-Bu
trans-3e
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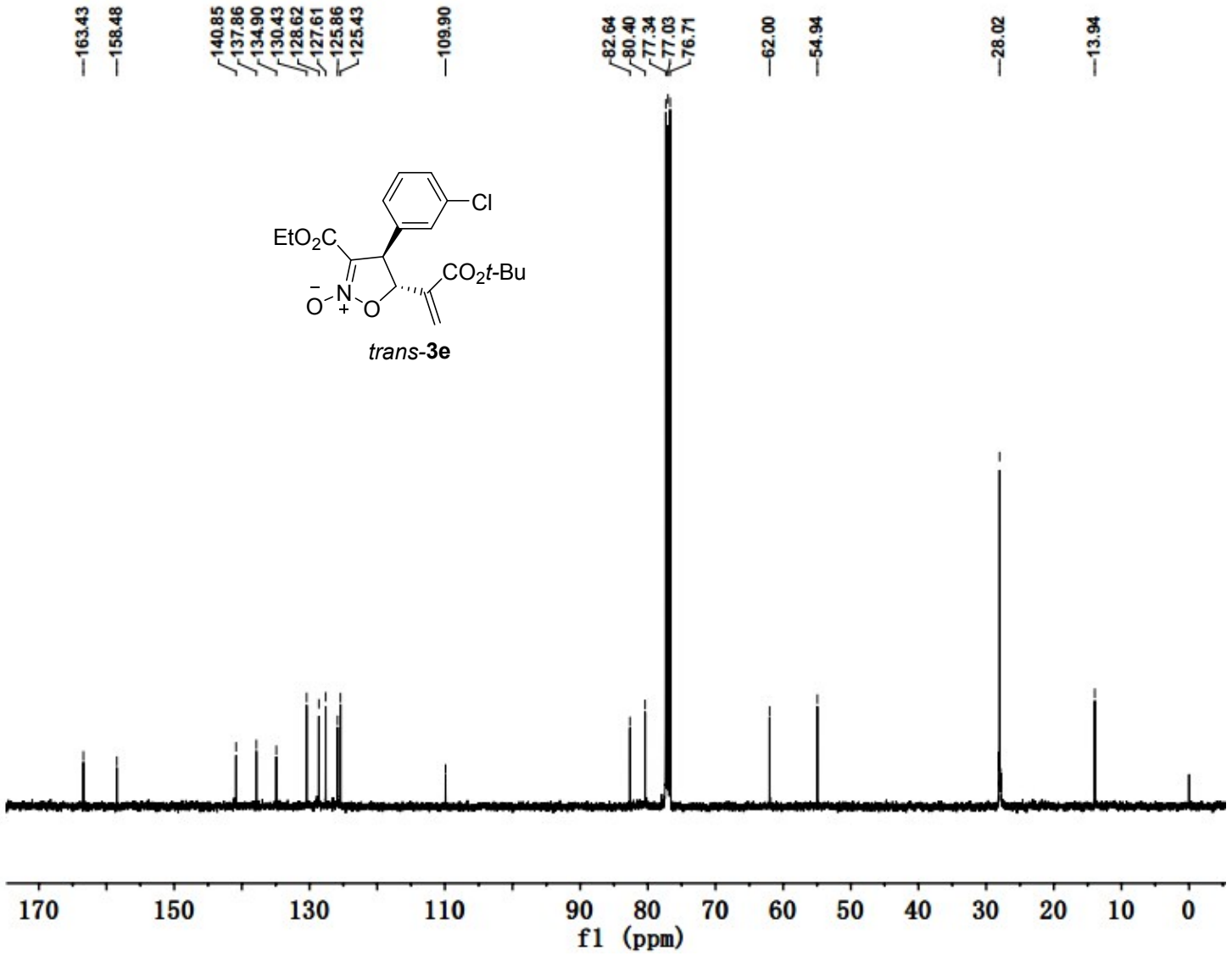
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54.94
28.02
13.94
Cl
EtO2C
CO2t-Bu
N
O
O
trans-3e
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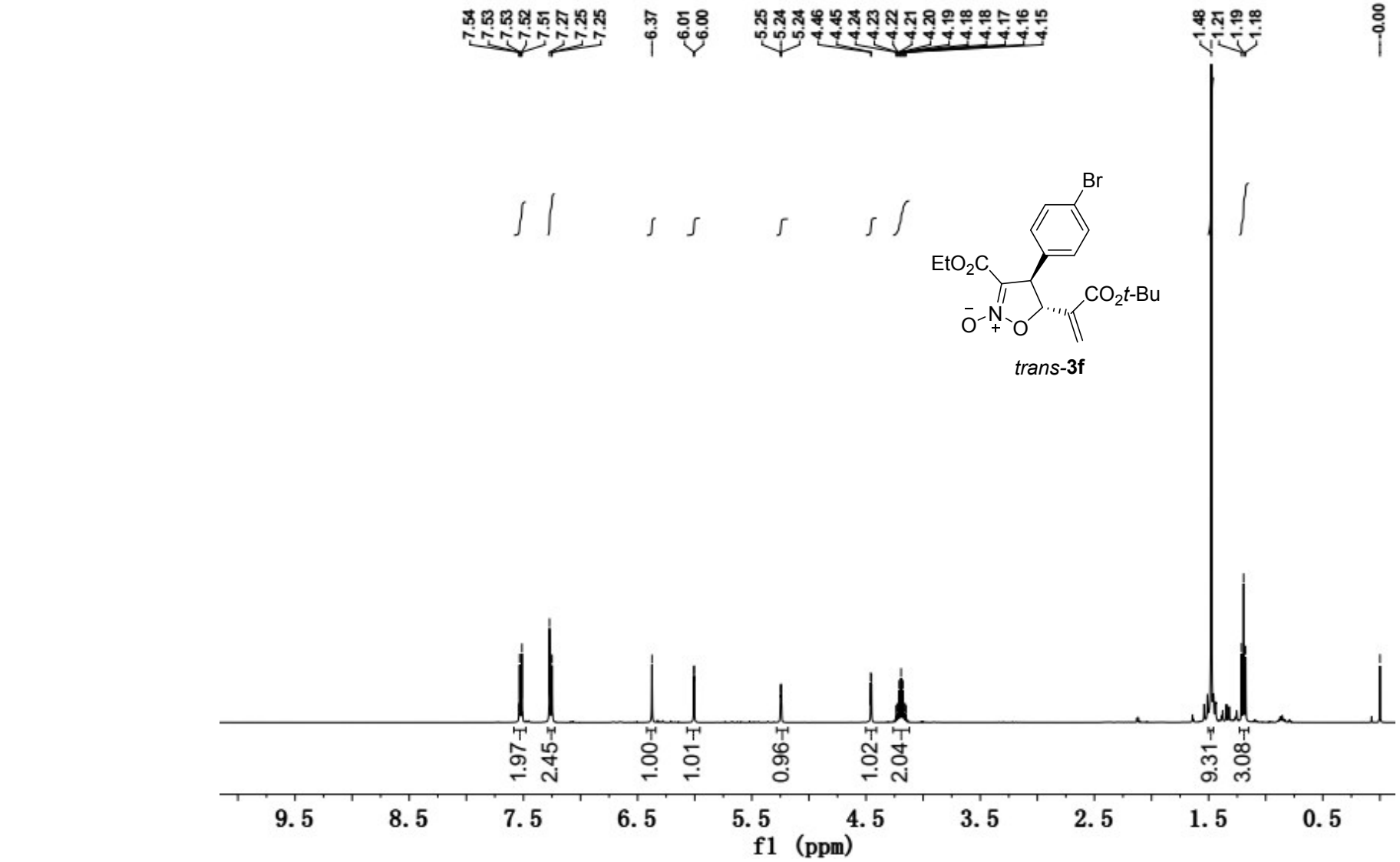
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4.20
4.19
4.18
4.18
4.17
4.16
4.15
1.48
1.21
1.19
1.18
0.00
Br
EtO2C
CO2t-Bu
trans-3f
1.97
2.45
1.00
1.01
0.96
1.02
2.04
9.31
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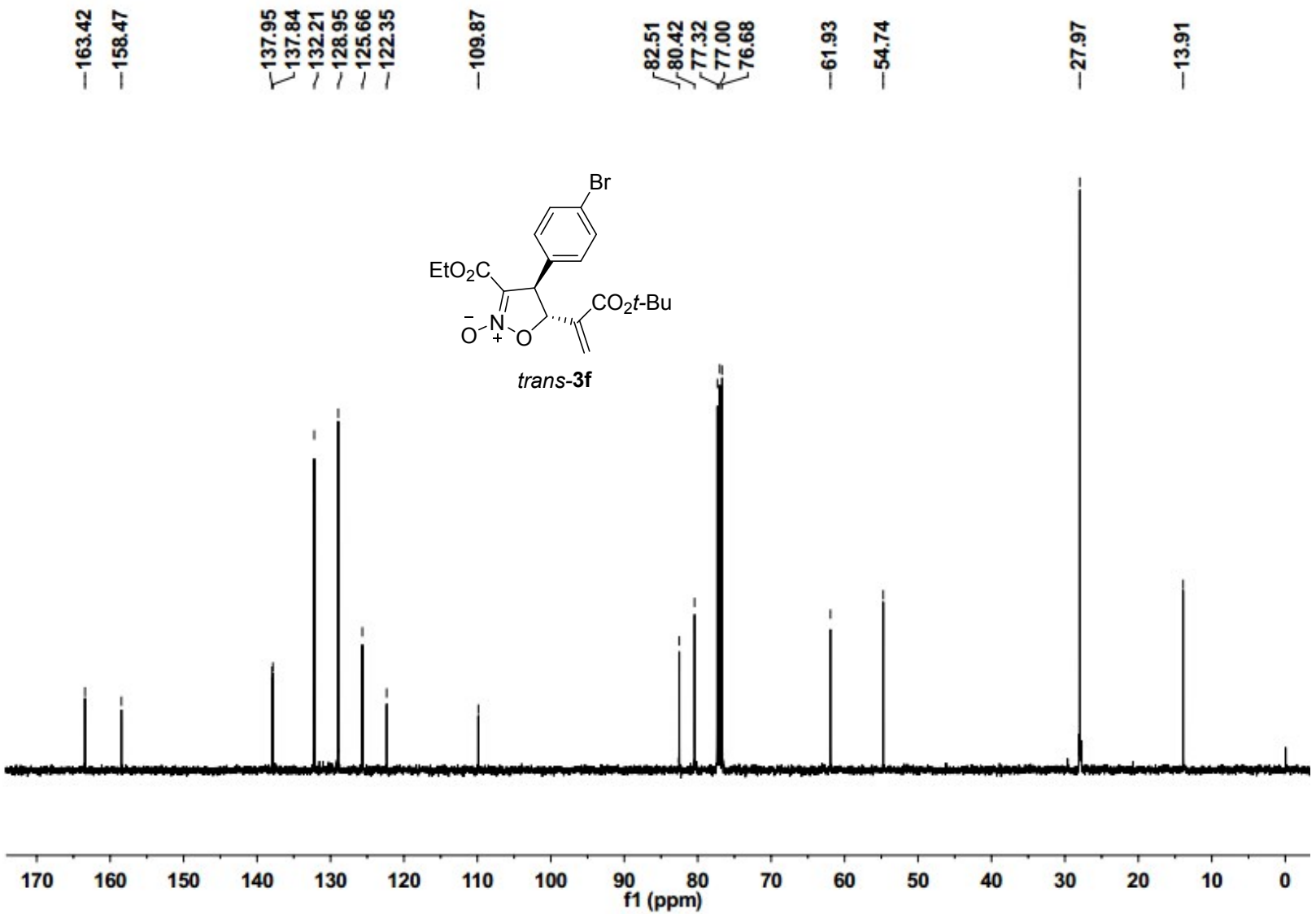
f1 (ppm)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13.91
Br
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CO2t-Bu
trans-3f
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110
100
90
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50
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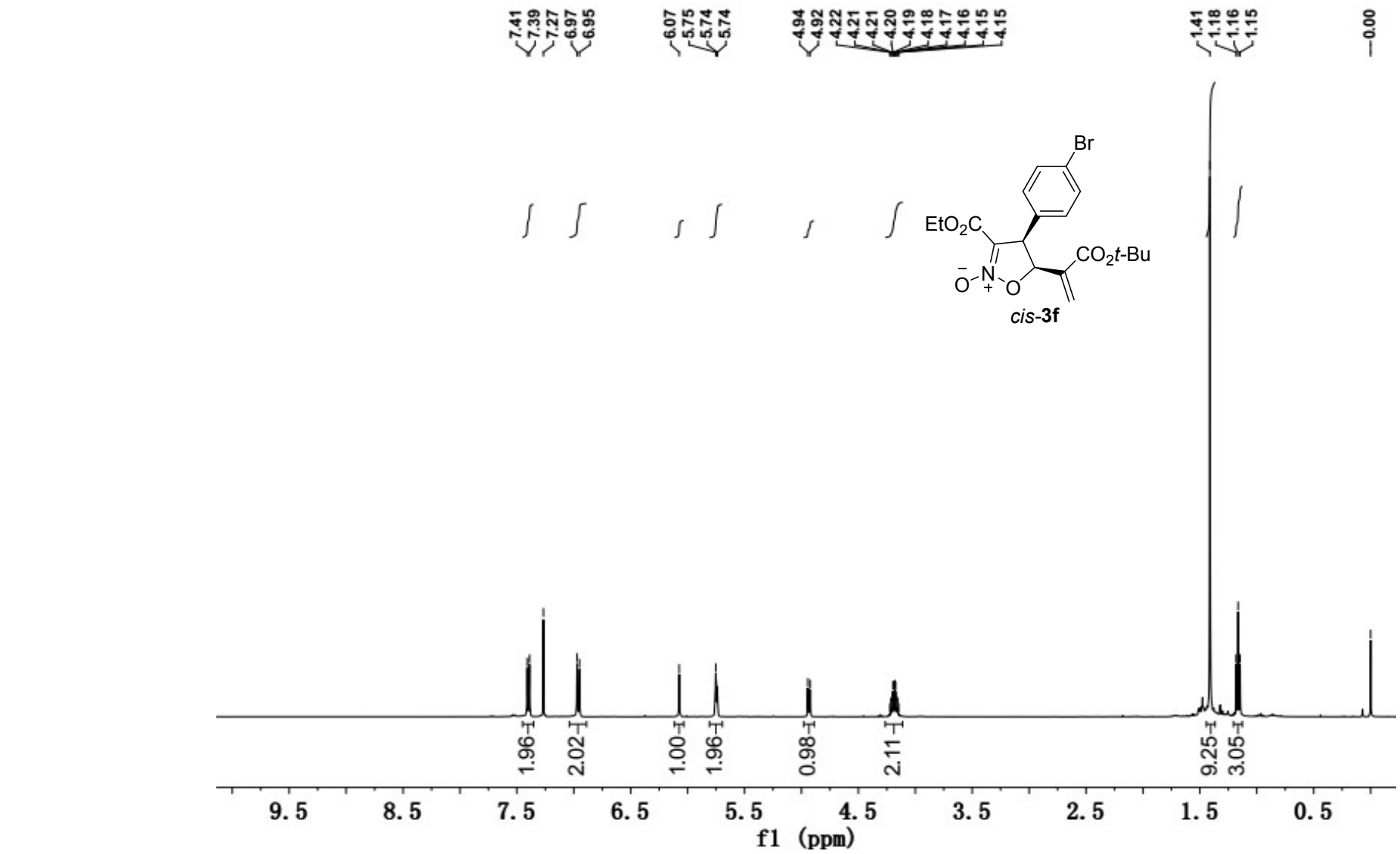
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4.18
4.17
4.16
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4.15
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1.18
1.16
1.15
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Br
EtO2C
CO2t-Bu
cis-3f
1.96
2.02
1.00
1.96
0.98
2.11
9.25
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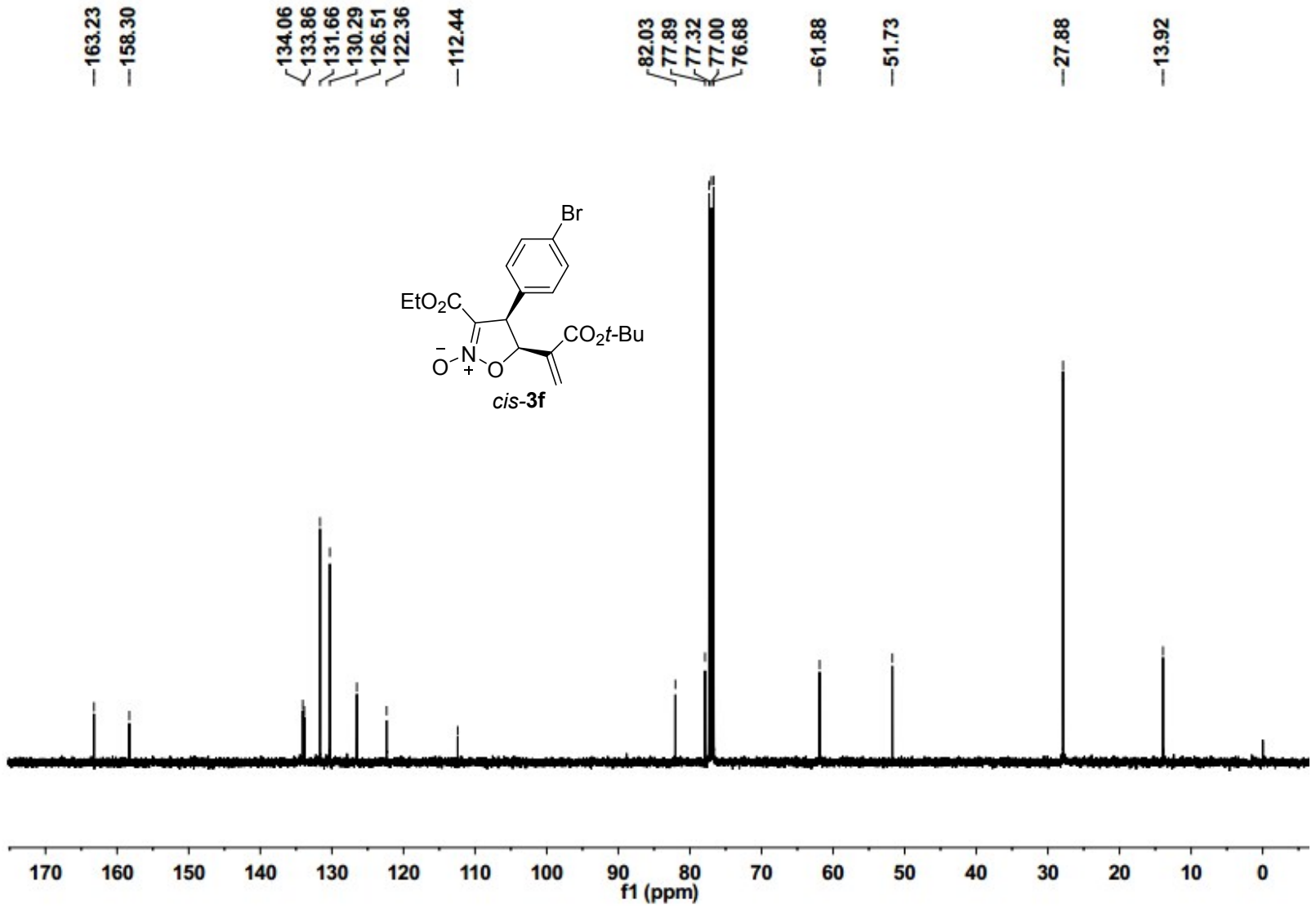
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51.73
27.88
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Br
EtO2C
CO2t-Bu
cis-3f
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50
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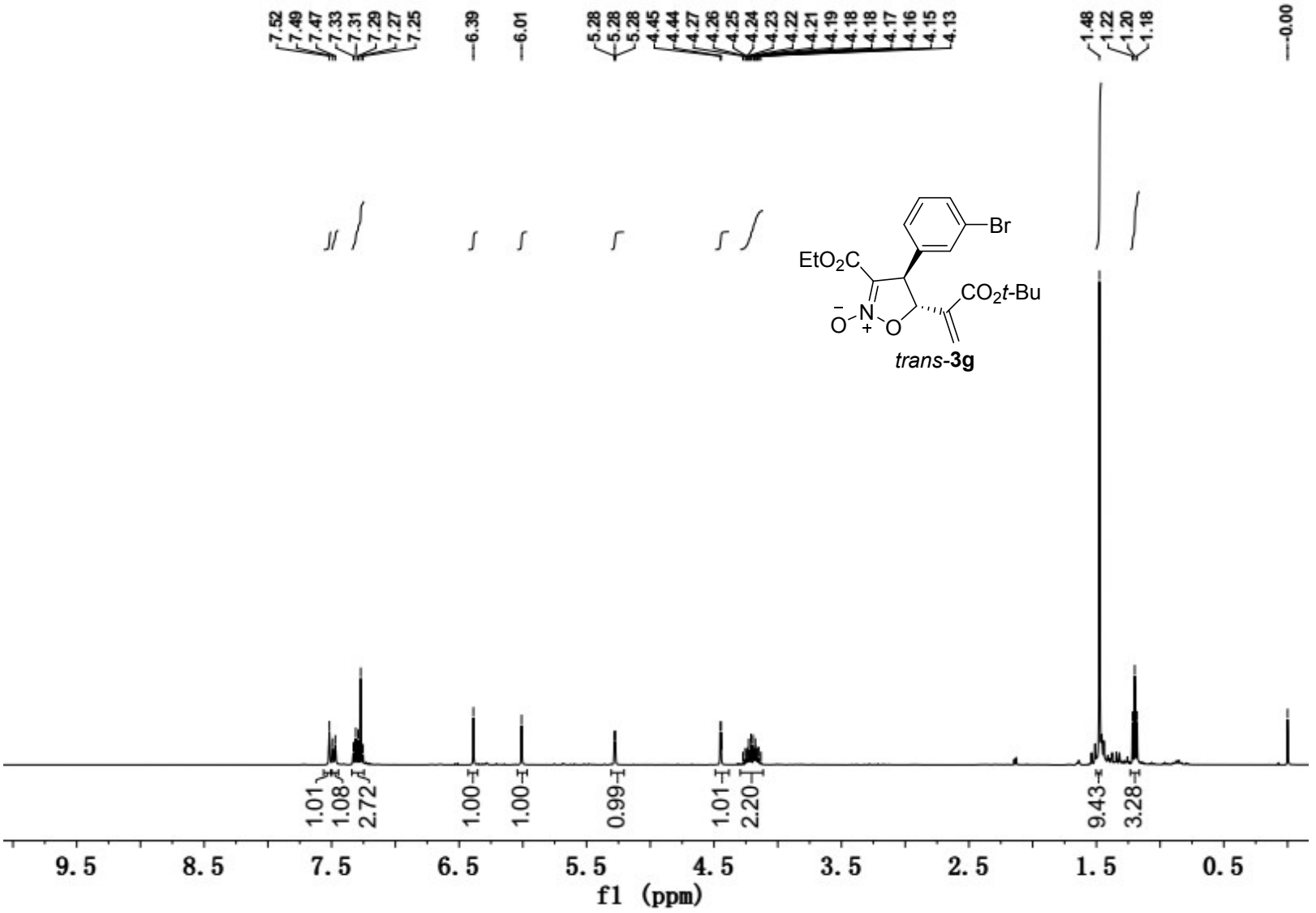
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4.27
4.26
4.25
4.24
4.23
4.22
4.21
4.19
4.18
4.18
4.17
4.16
4.15
4.13
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1.22
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EtO2C
CO2t-Bu
trans-3g
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1.08
2.72
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1.00
0.99
1.01
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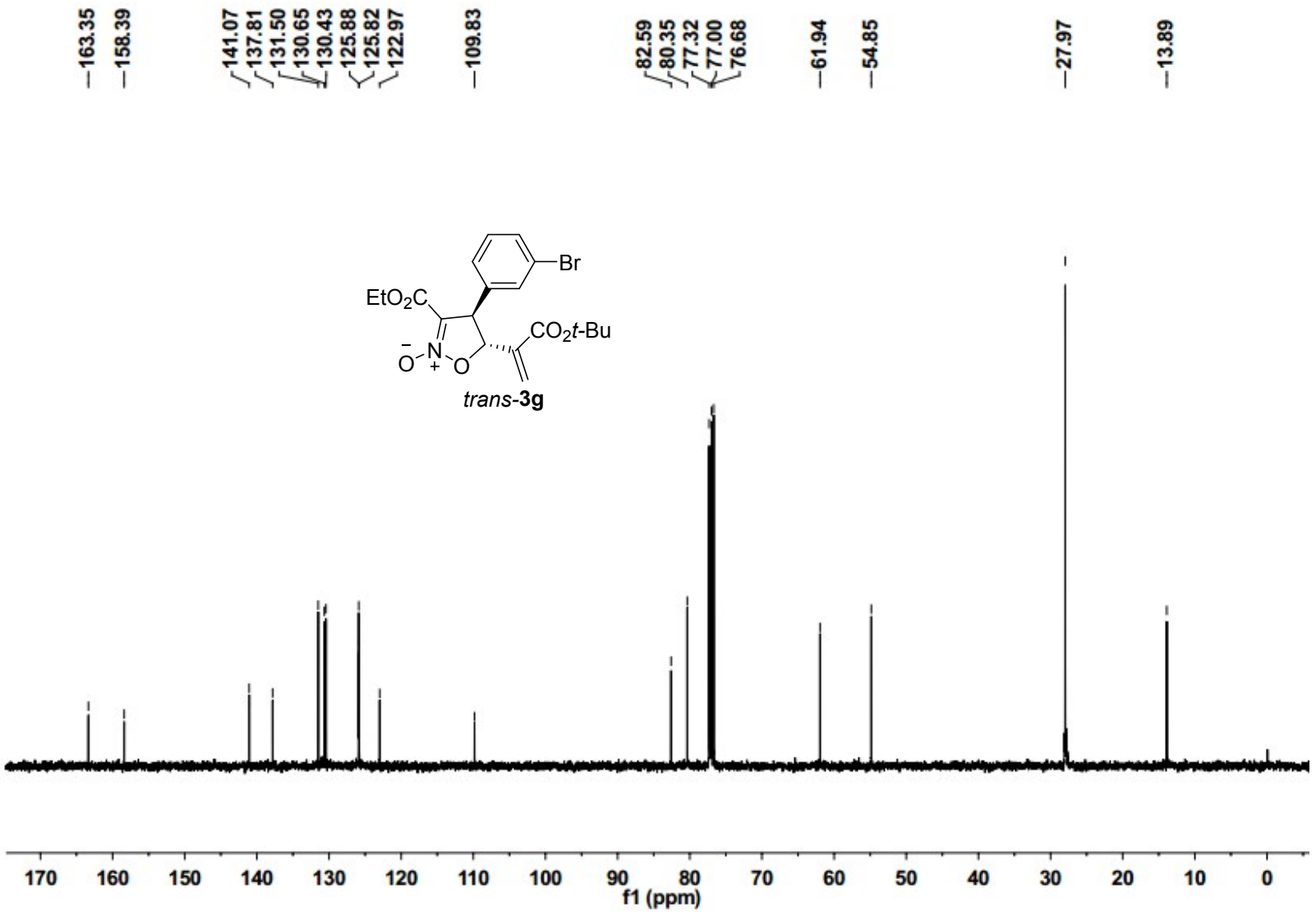
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77.00
76.68
61.94
54.85
27.97
13.89
Br
EtO2C
CO2t-Bu
trans-3g
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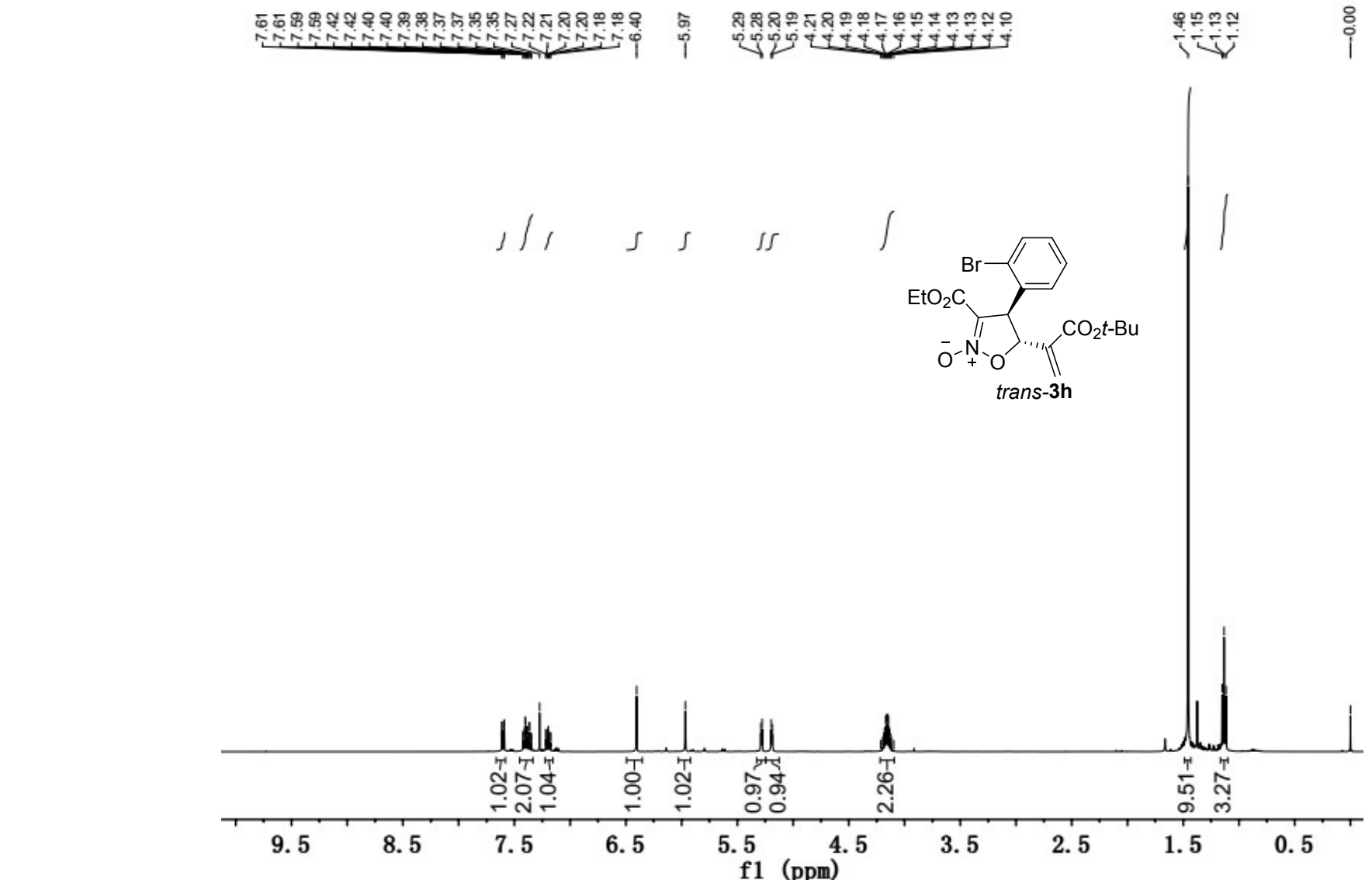
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4.13
4.12
4.10
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1.15
1.13
1.12
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CO2t-Bu
trans-3h
1.02
2.07
1.04
1.00
1.02
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0.94
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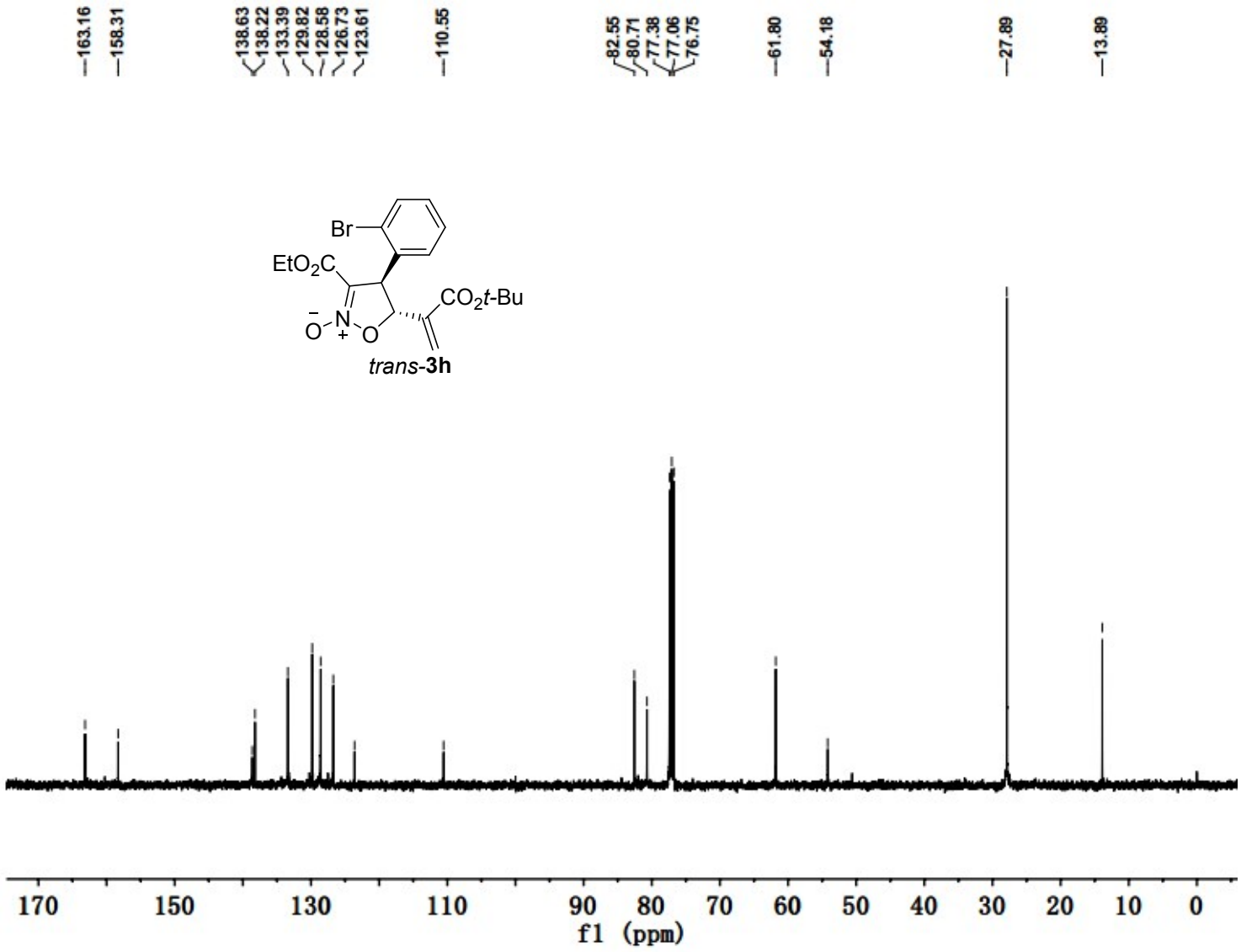
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13.89
Br
EtO2C
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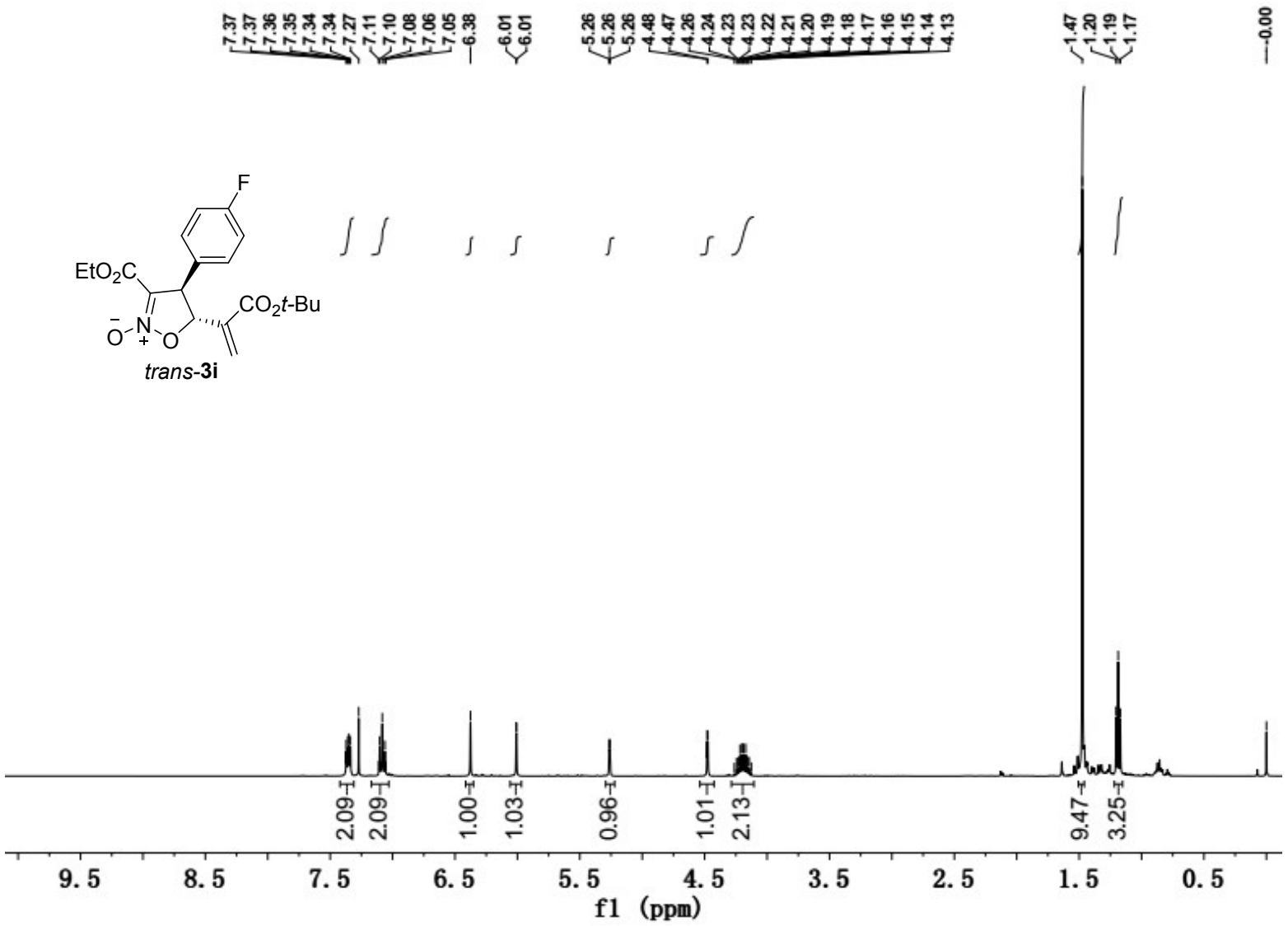
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4.14
4.13
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1.20
1.19
1.17
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F
EtO2C
CO2t-Bu
trans-3i
2.09
2.09
1.00
1.03
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1.01
2.13
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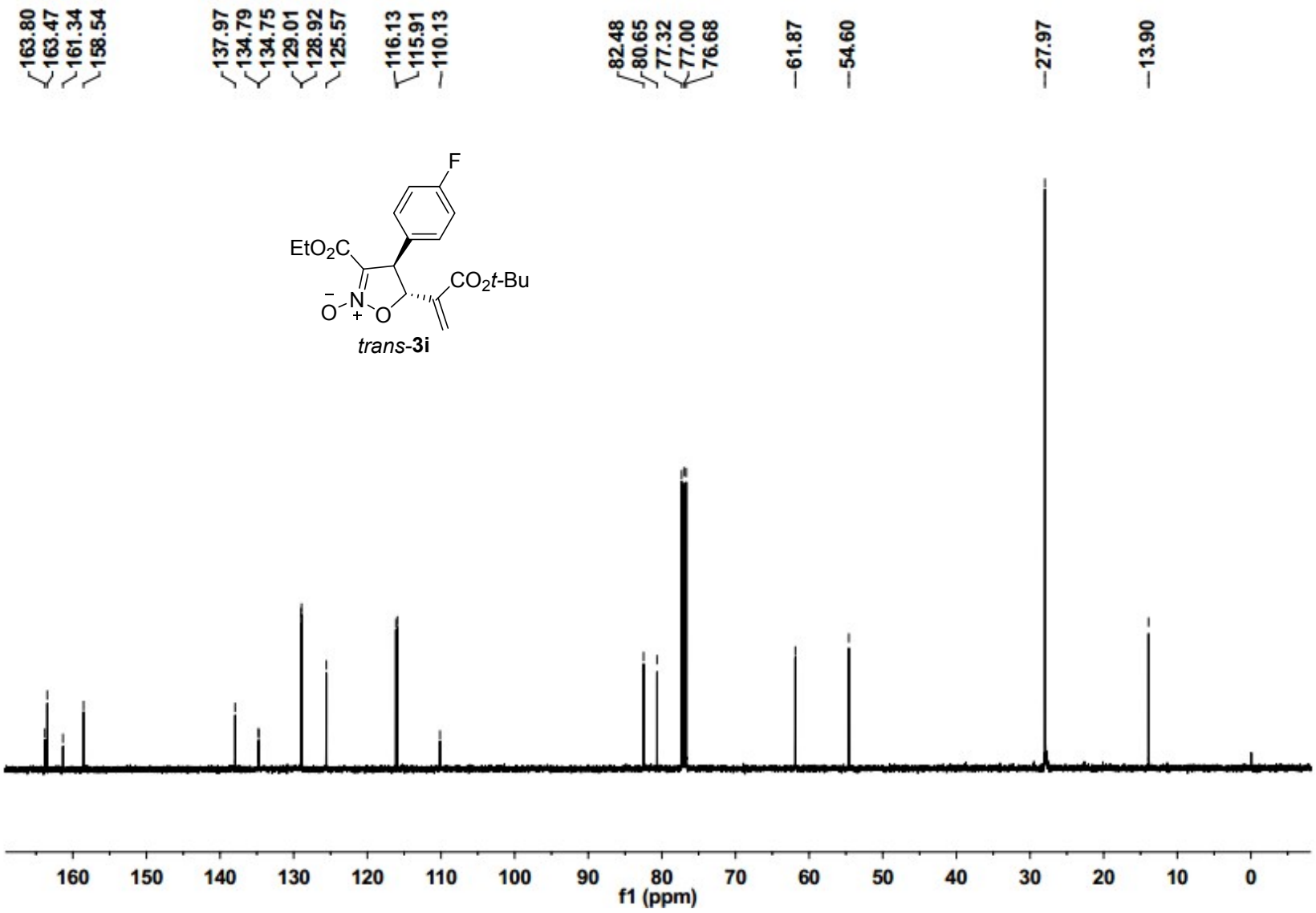
f1 (ppm)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77.32
77.00
76.68
61.87
54.60
27.97
13.90
F
EtO2C
CO2t-Bu
trans-3i
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130
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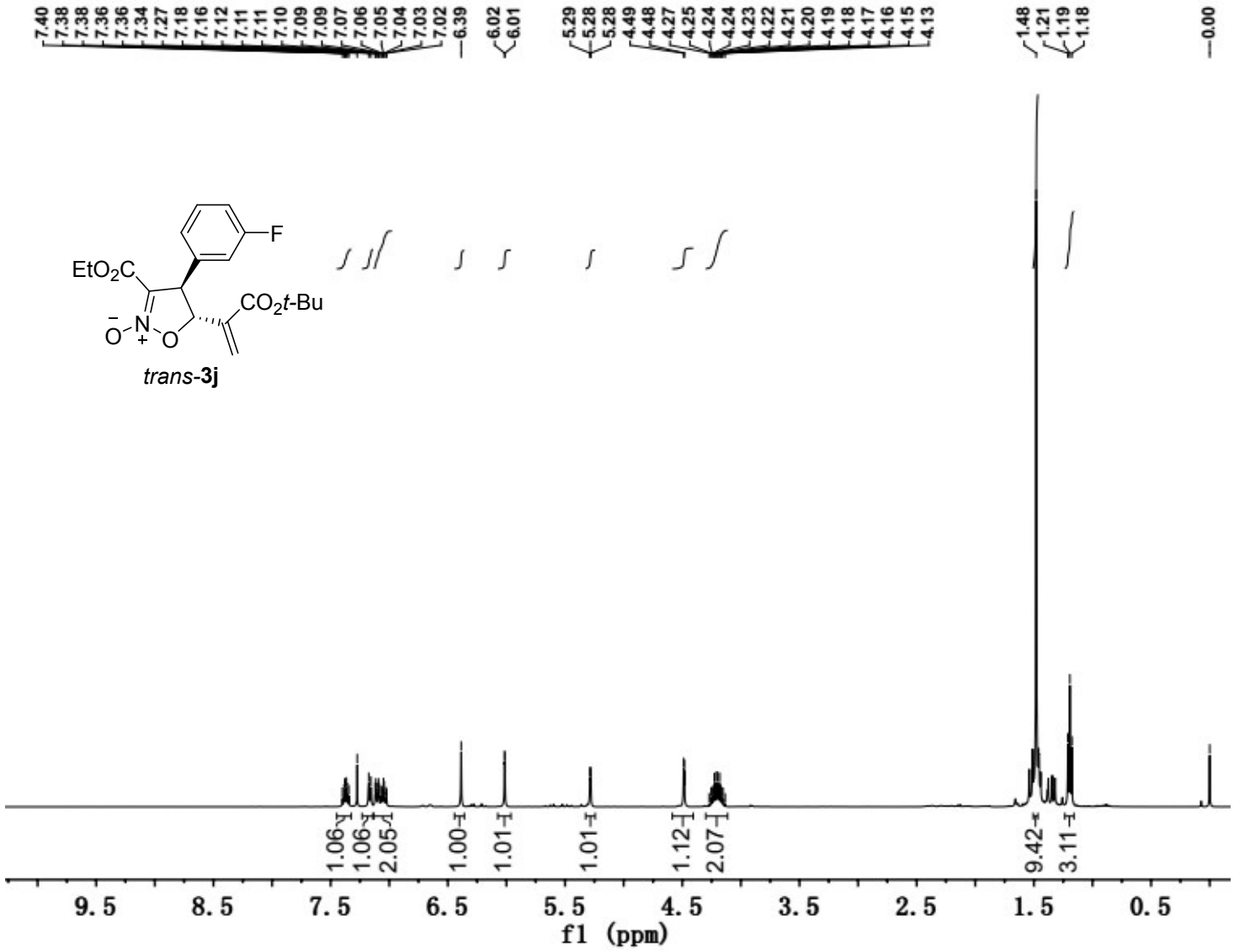
f1 (ppm)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4.21
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4.19
4.18
4.17
4.16
4.15
4.13
1.48
1.21
1.19
1.18
0.00
F
EtO2C
CO2t-Bu
O
N
O
trans-3j
1.06
1.06
2.05
1.00
1.01
1.01
1.12
2.07
9.42
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3.5
2.5
1.5
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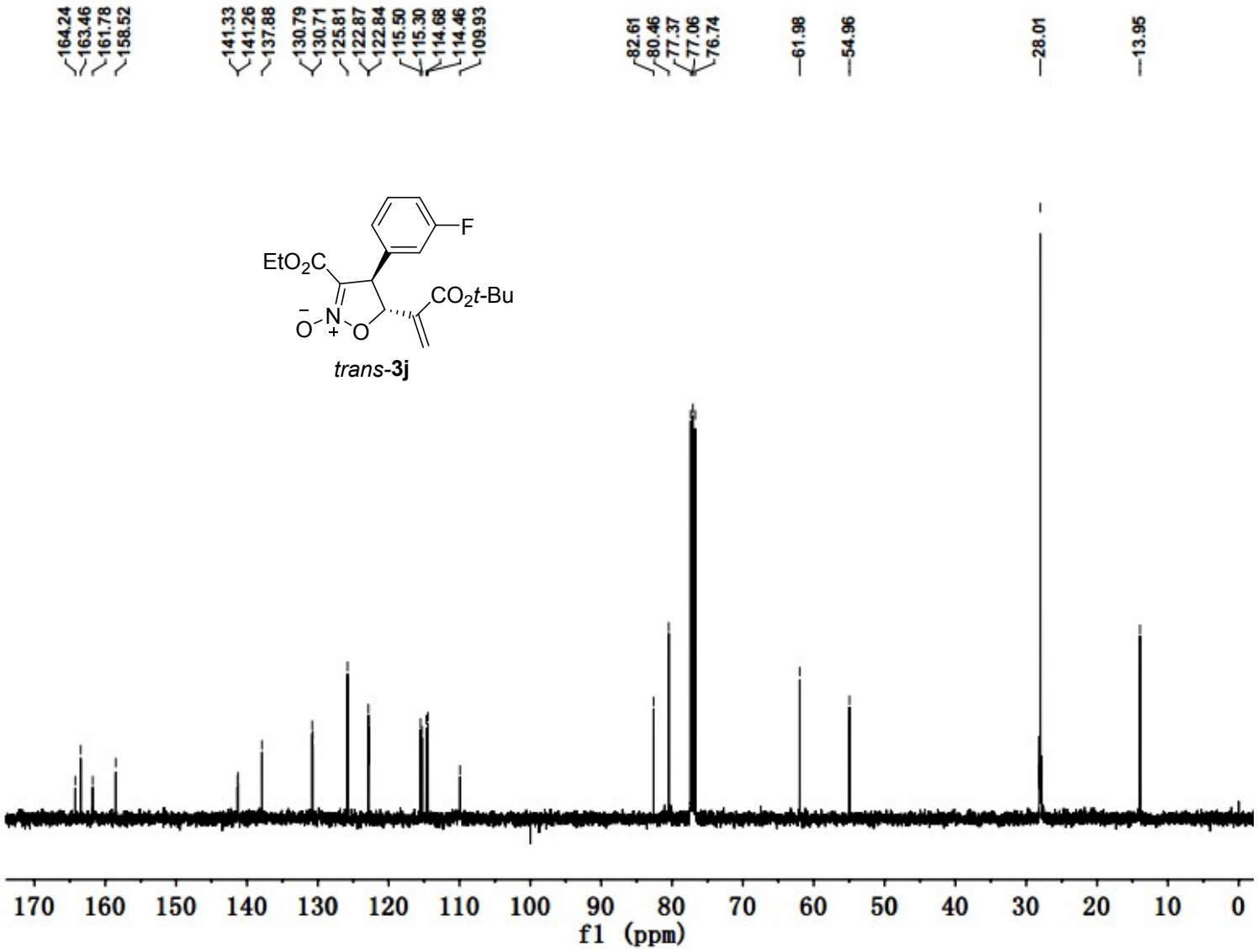
f1 (ppm)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115.30
114.68
114.46
109.93
82.61
80.46
77.37
77.06
76.74
61.98
54.96
28.01
13.95
F
EtO2C
CO2t-Bu
N
O
O
trans-3j
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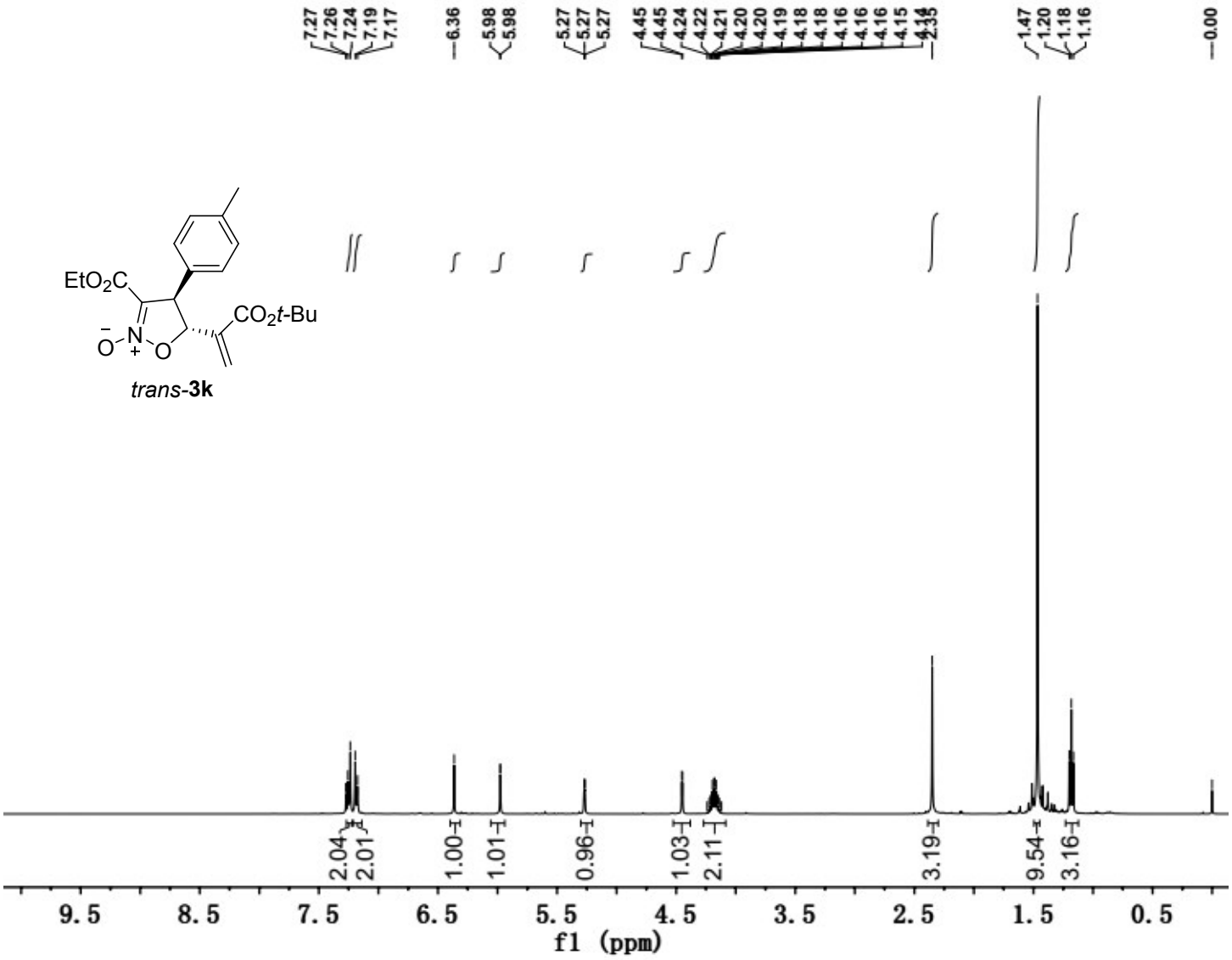
f1 (ppm)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4.24
4.22
4.21
4.20
4.20
4.19
4.18
4.18
4.16
4.16
4.16
4.15
2.35
1.47
1.20
1.18
1.16
0.00
EtO2C
CO2t-Bu
trans-3k
2.04
2.01
1.00
1.01
0.96
1.03
2.11
3.19
9.54
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2.5
1.5
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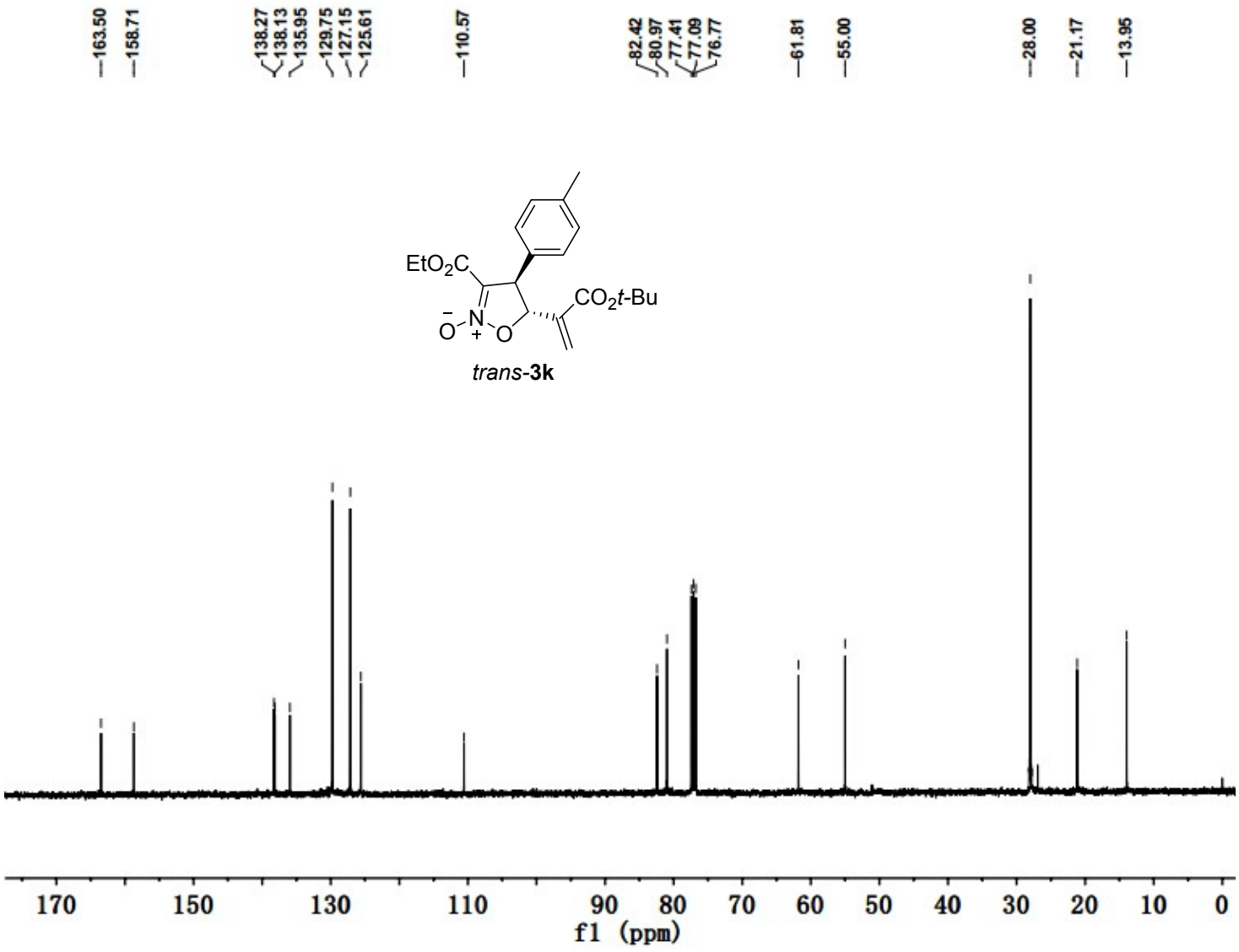
f1 (ppm)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77.41
77.09
76.77
61.81
55.00
28.00
21.17
13.95
EtO2C
CO2t-Bu
trans-3k
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150
130
110
90
80
70
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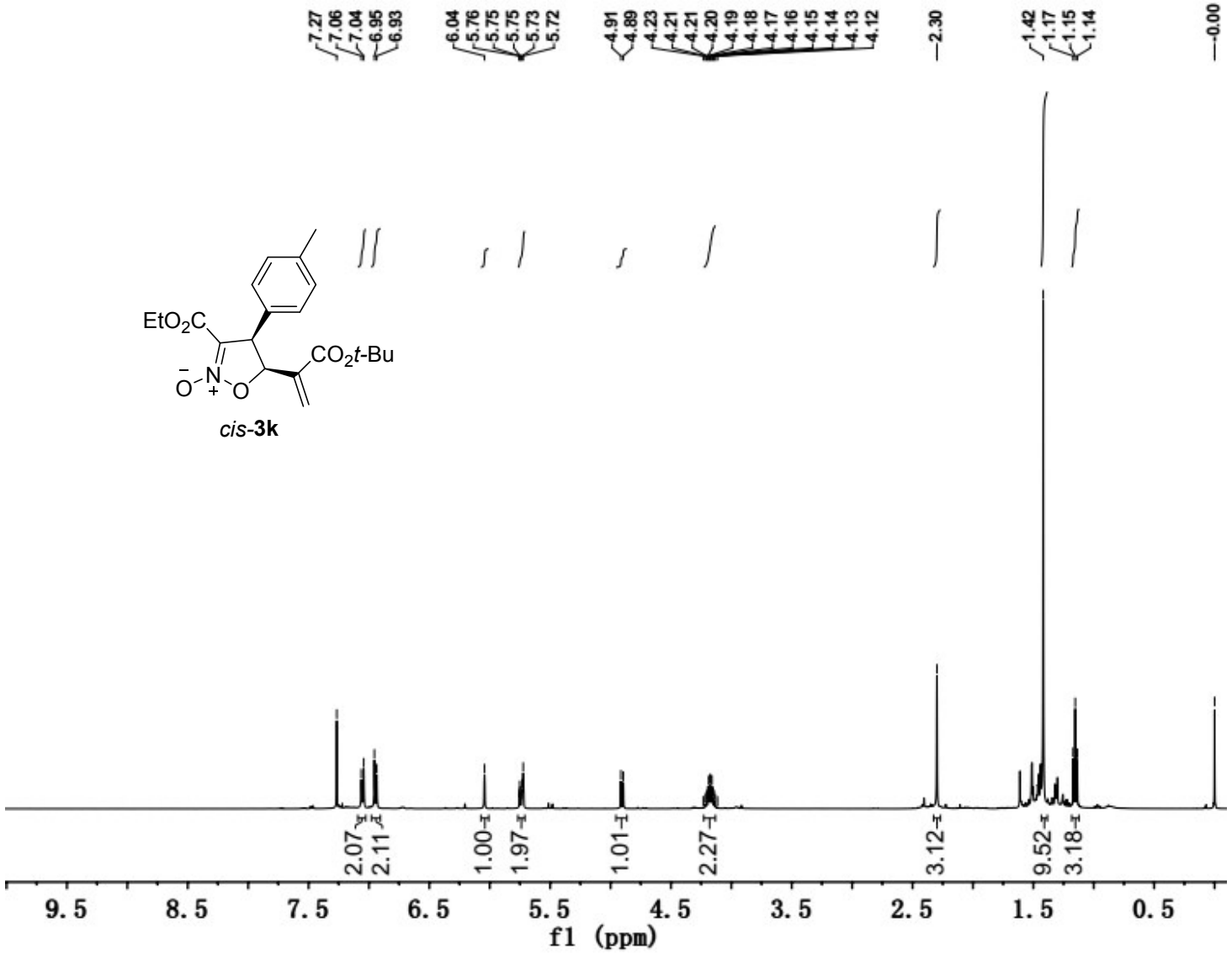
50
40
30
20
10
0
f1 (ppm)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4.21
4.20
4.19
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4.17
4.16
4.15
4.14
4.13
4.12
2.30
1.42
1.17
1.15
1.14
0.00
EtO2C
CO2t-Bu
cis-3k
2.07
2.11
1.00
1.97
1.01
2.27
3.12
9.52
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4.5
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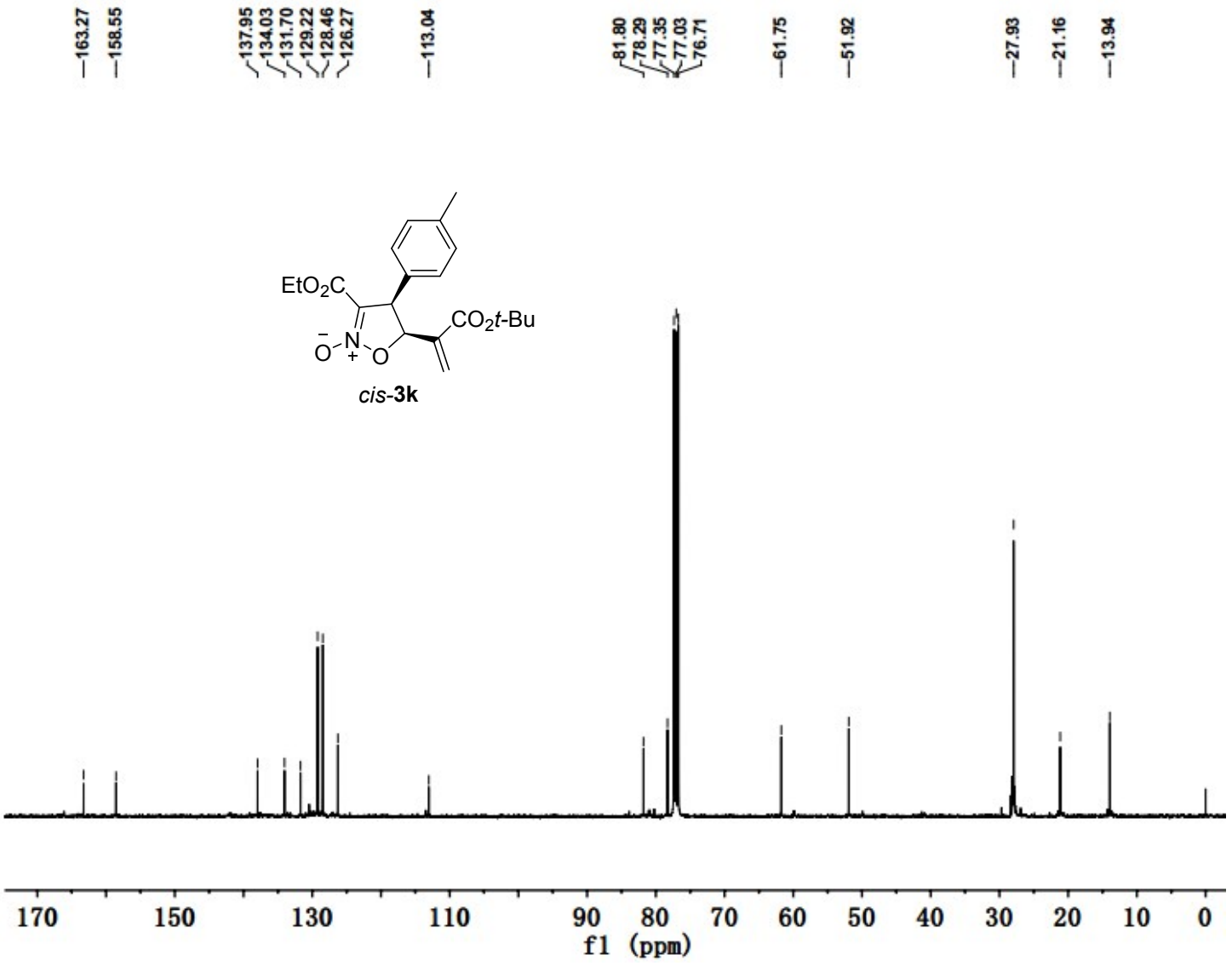
f1 (ppm)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77.03
76.71
61.75
51.92
27.93
21.16
13.94
EtO2C
CO2t-Bu
cis-3k
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150
130
110
90
80
70
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50
40
30
20
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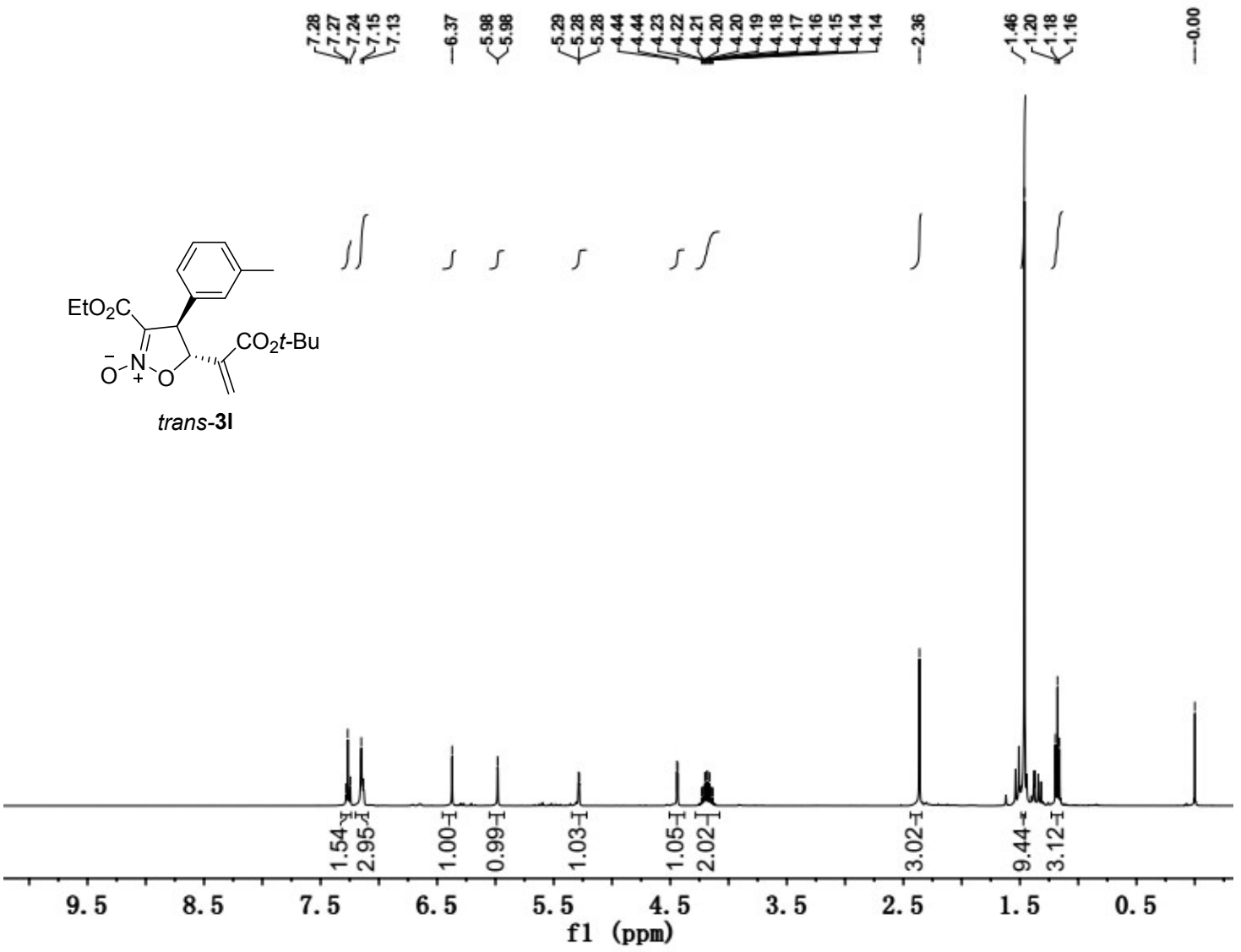
f1 (ppm)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4.18
4.17
4.16
4.15
4.14
4.14
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1.46
1.20
1.18
1.16
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EtO2C
CO2t-Bu
N
O
O
trans-3l
1.54
2.95
1.00
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1.05
2.02
3.02
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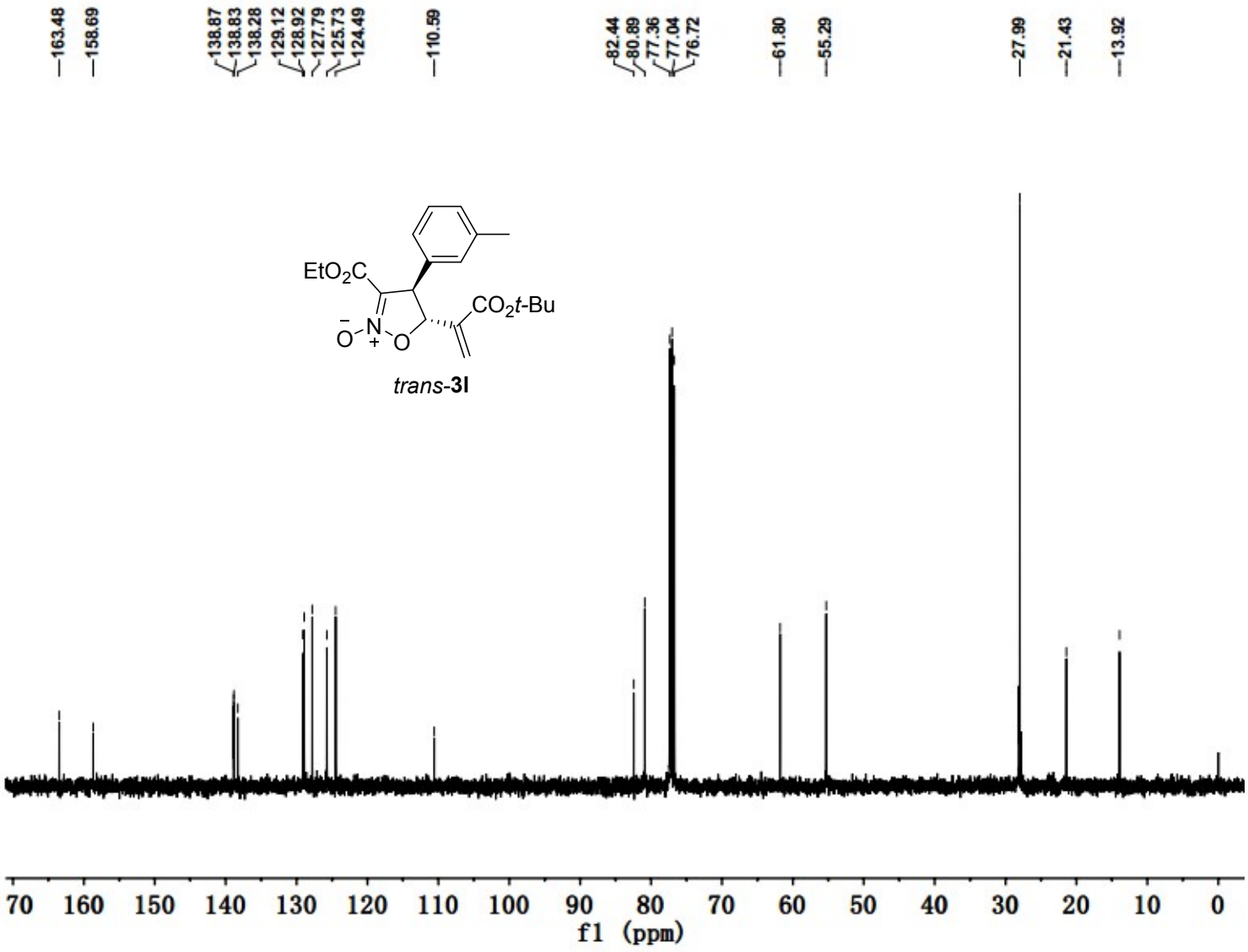
f1 (ppm)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77.04
76.72
61.80
55.29
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EtO2C
CO2t-Bu
trans-3l
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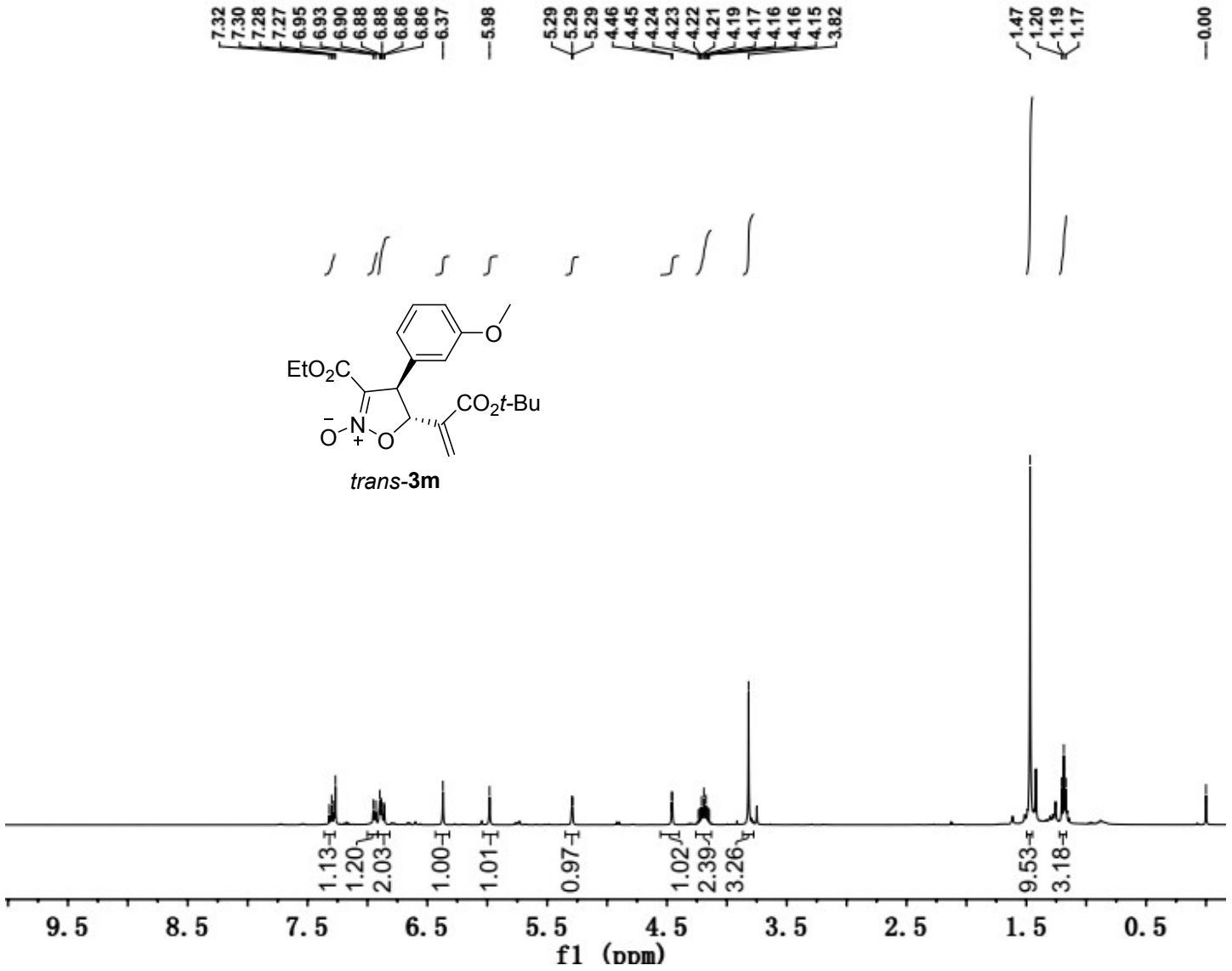
f1 (ppm)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6.86
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5.29
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4.19
4.17
4.16
4.16
4.15
3.82
1.47
1.20
1.19
1.17
0.00
EtO2C
CO2t-Bu
O
N
O
O
trans-3m
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1.20
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1.00
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1.02
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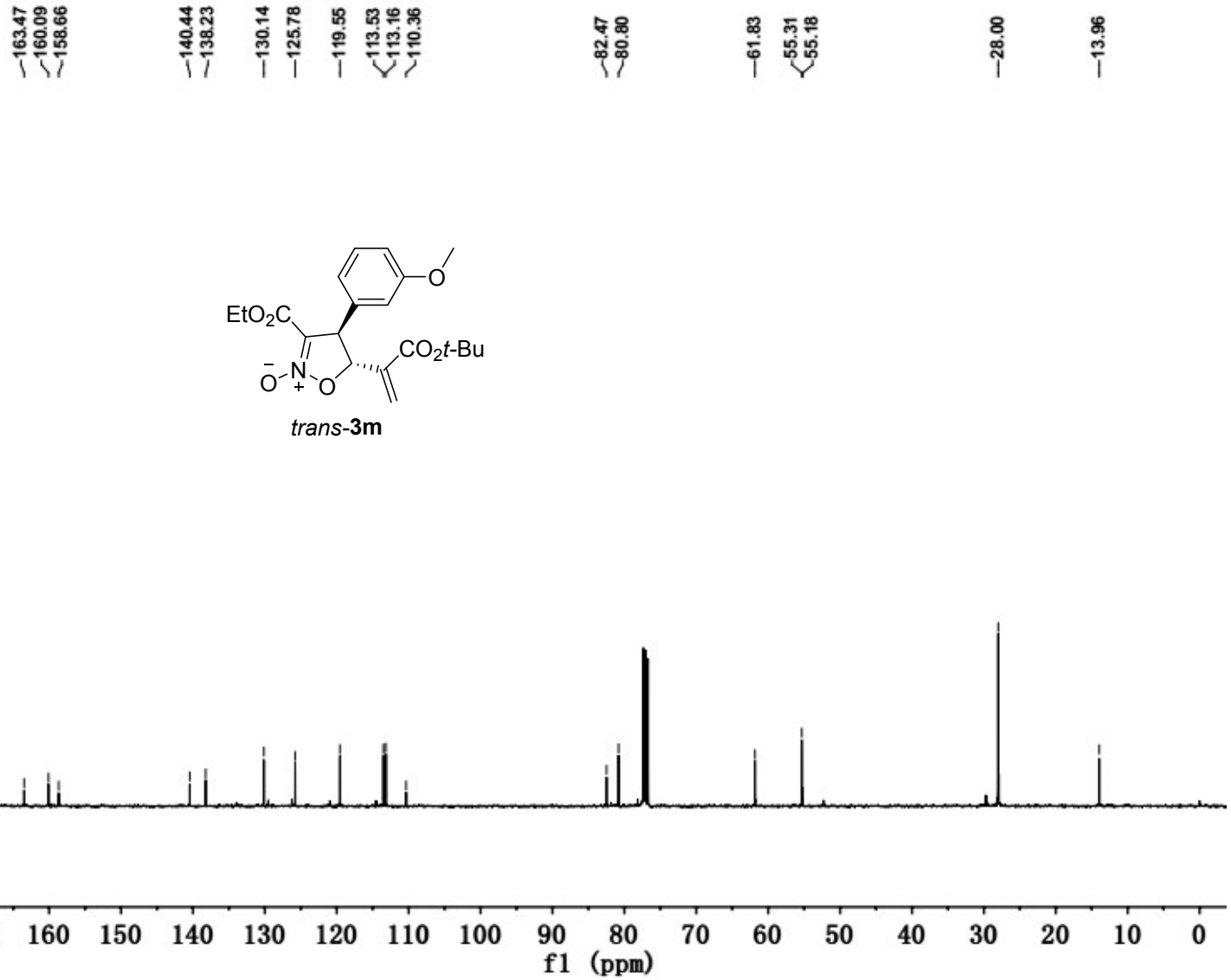
f1 (ppm)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61.83
55.31
55.18
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EtO2C
CO2t-Bu
trans-3m
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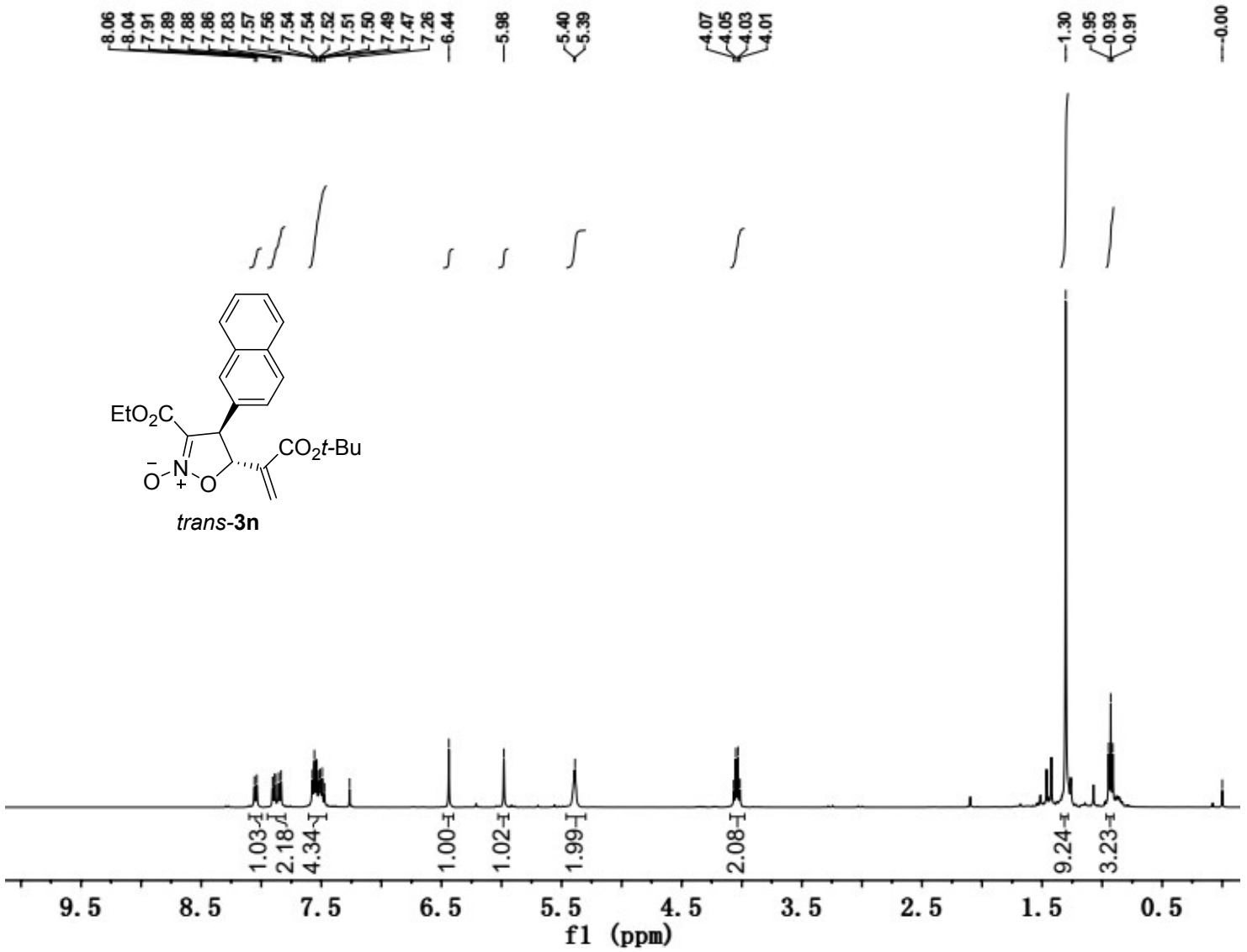
f1 (ppm)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7.49
7.47
7.26
6.44
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5.39
4.07
4.05
4.03
4.01
1.30
0.95
0.93
0.91
0.00
EtO2C
CO2t-Bu
trans-3n
1.03
2.18
4.34
1.00
1.02
1.99
2.08
9.24
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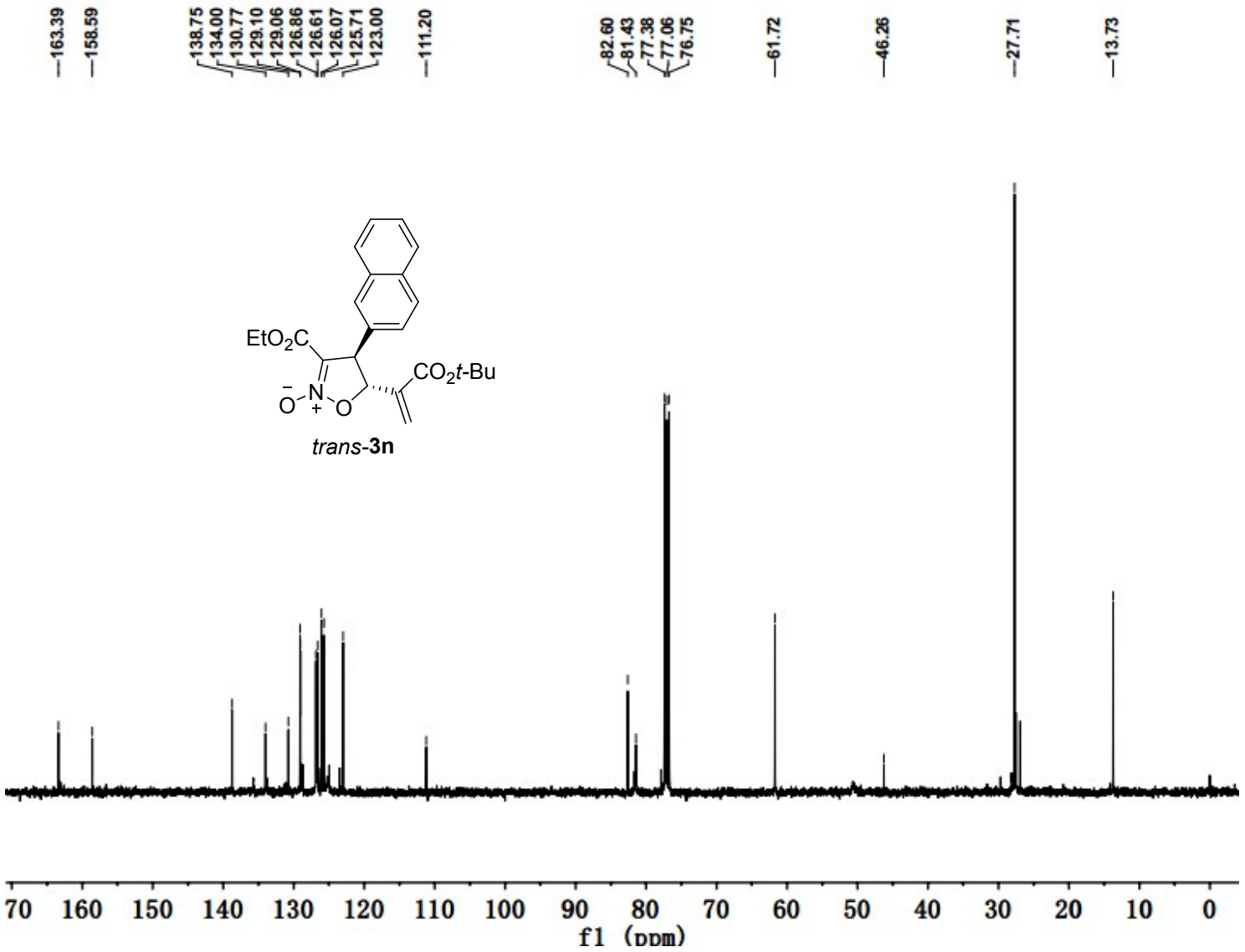
4.5
3.5
2.5
1.5
0.5
f1 (ppm)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81.43
77.38
77.06
76.75
61.72
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EtO2C
CO2t-Bu
trans-3n
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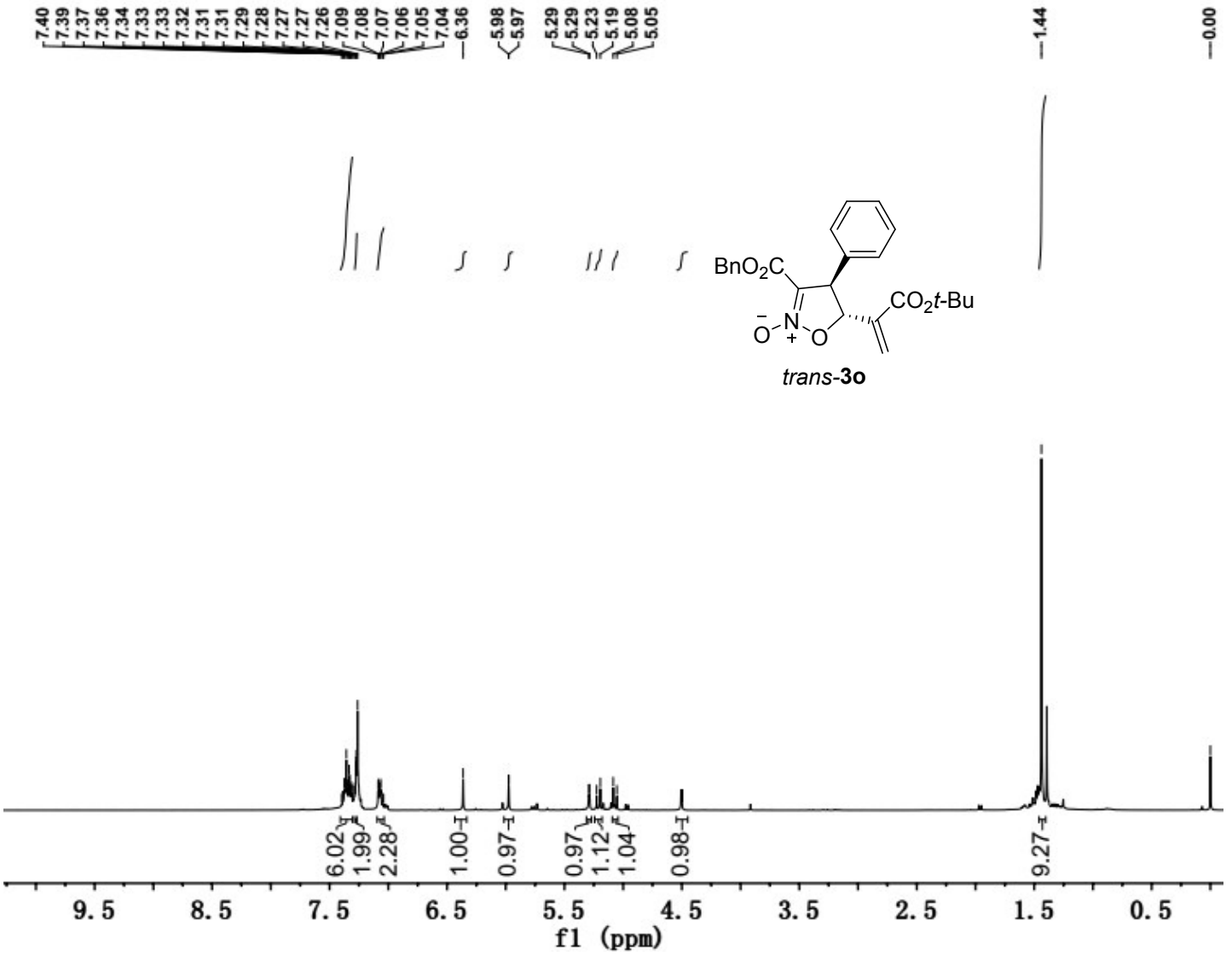
f1 (ppm)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BnO2C
CO2t-Bu
trans-3o
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1.04
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2.5
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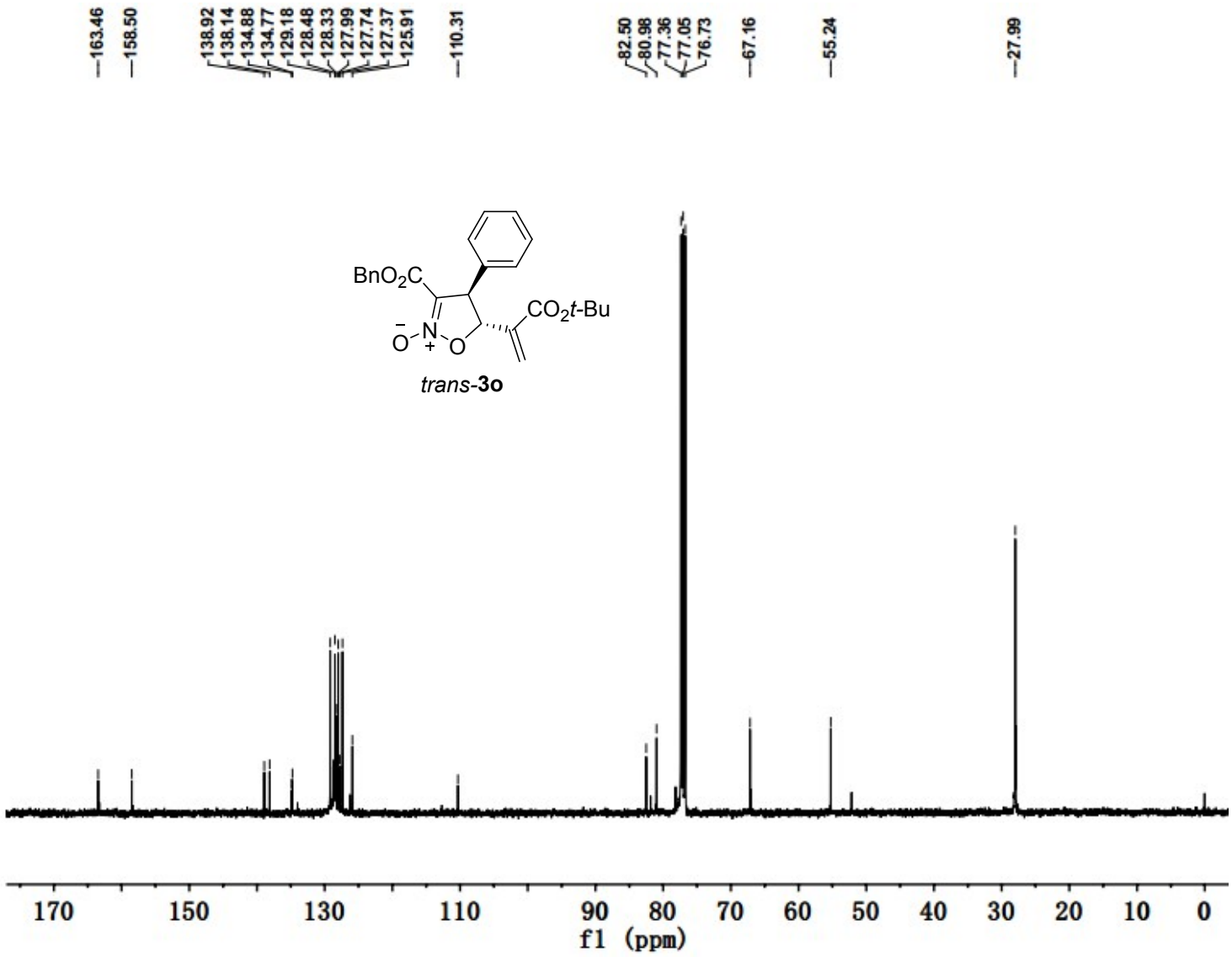
f1 (ppm)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80.98
77.36
77.05
76.73
67.16
55.24
27.99
BnO2C
CO2t-Bu
trans-3o
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90
80
70
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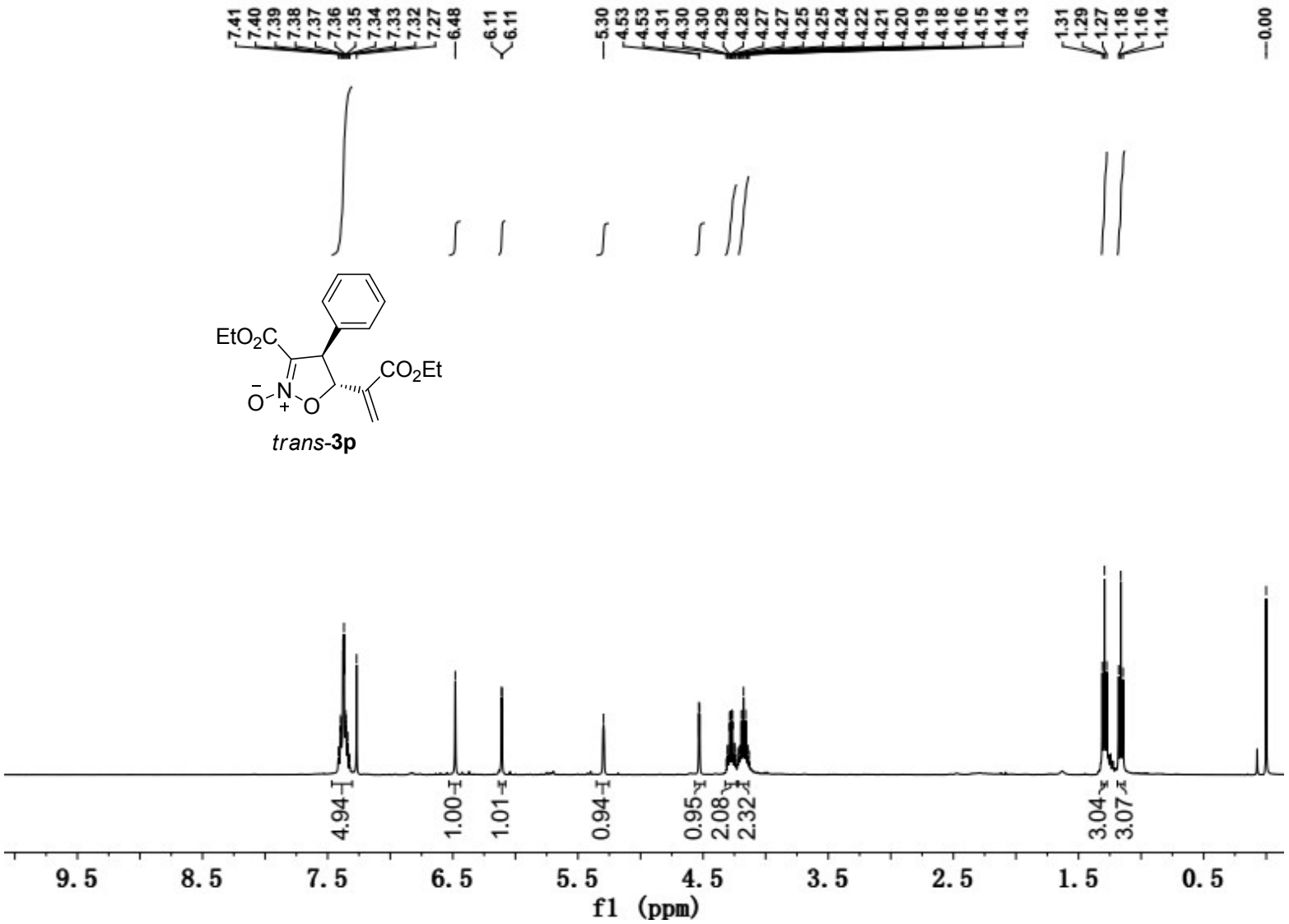
50
40
30
20
10
0
f1 (ppm)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4.14
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1.16
1.14
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CO2Et
trans-3p
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2.32
3.04
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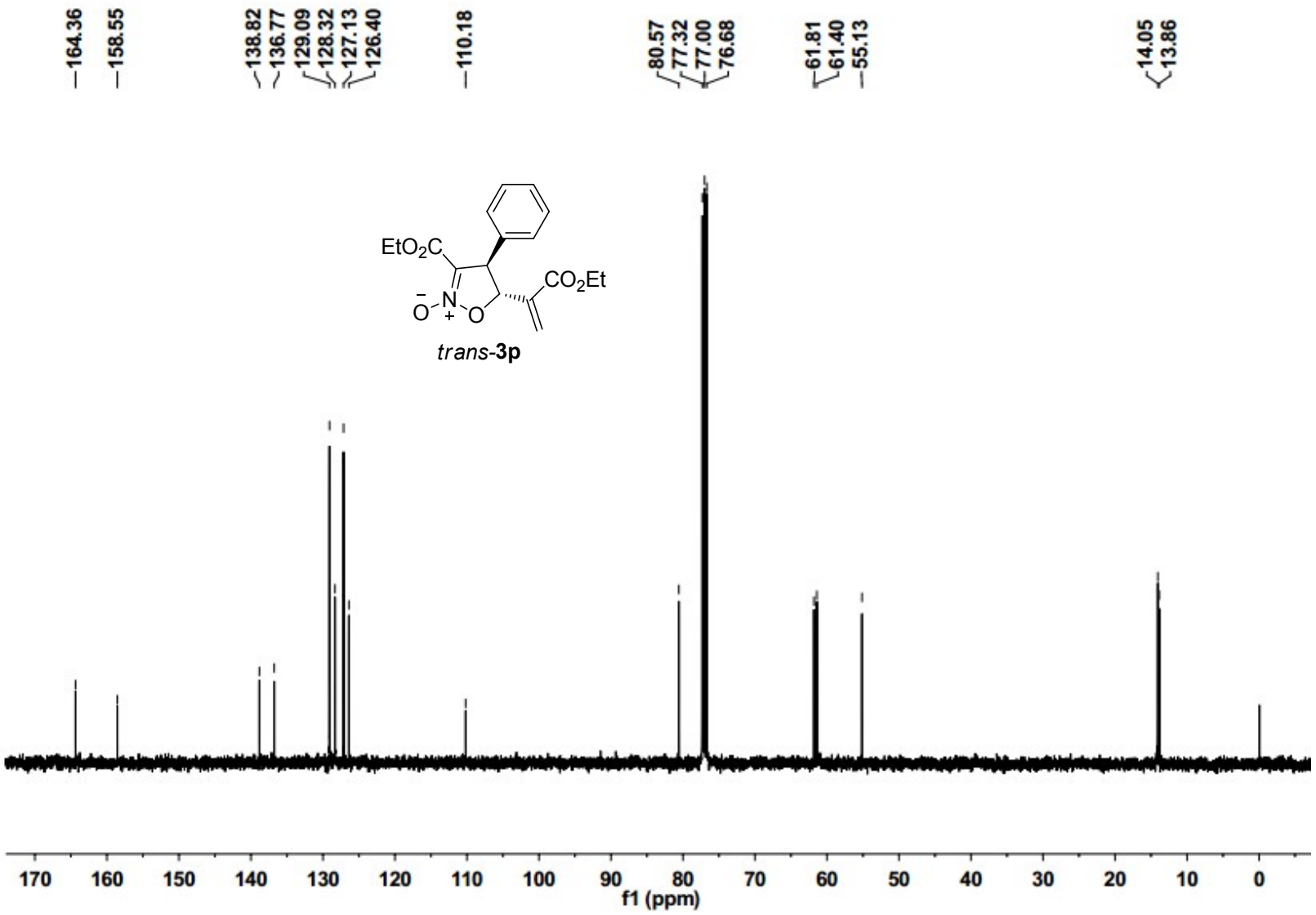
f1 (ppm)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61.40
55.13
14.05
13.86
EtO2C
CO2Et
trans-3p
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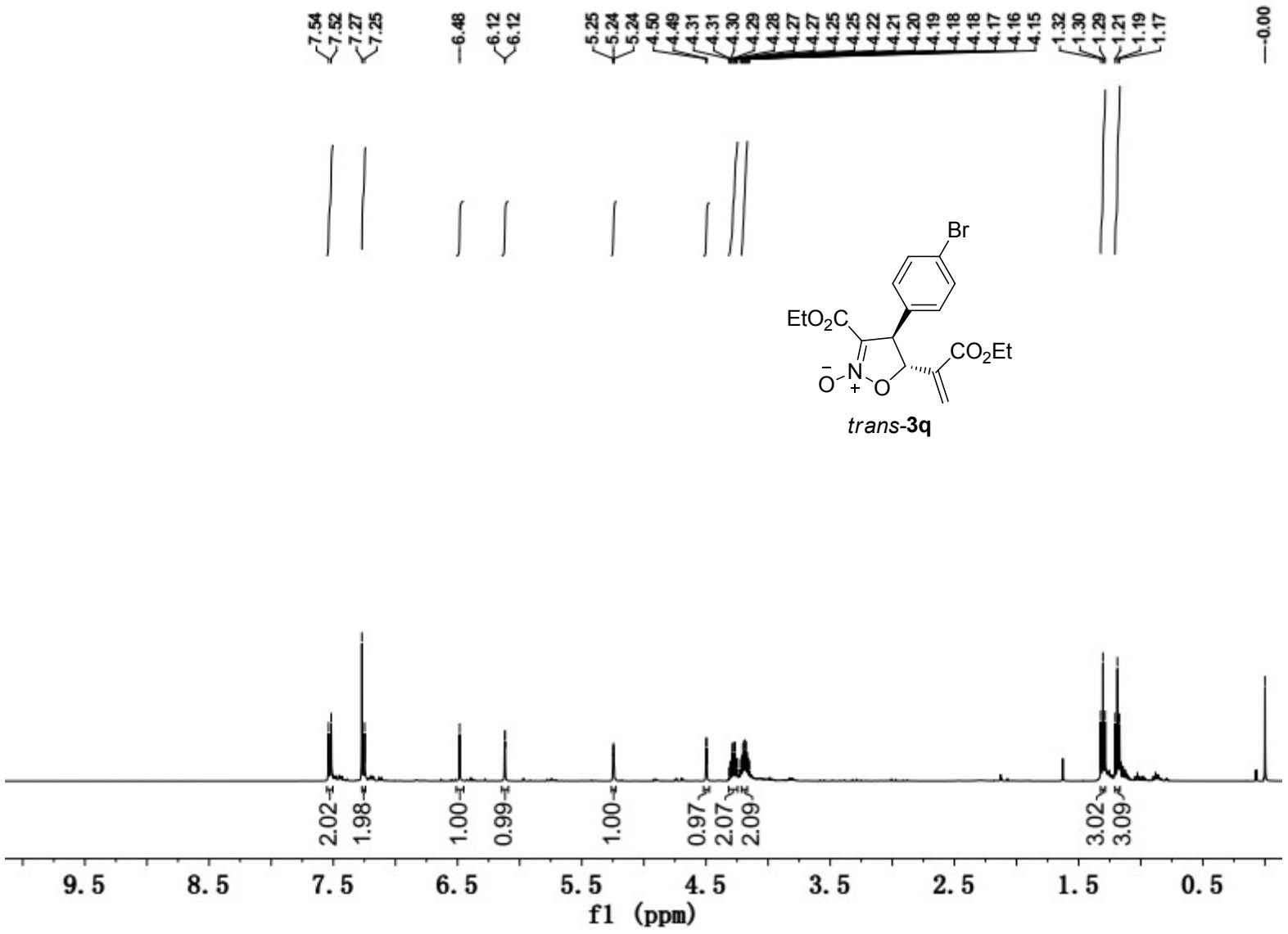
f1 (ppm)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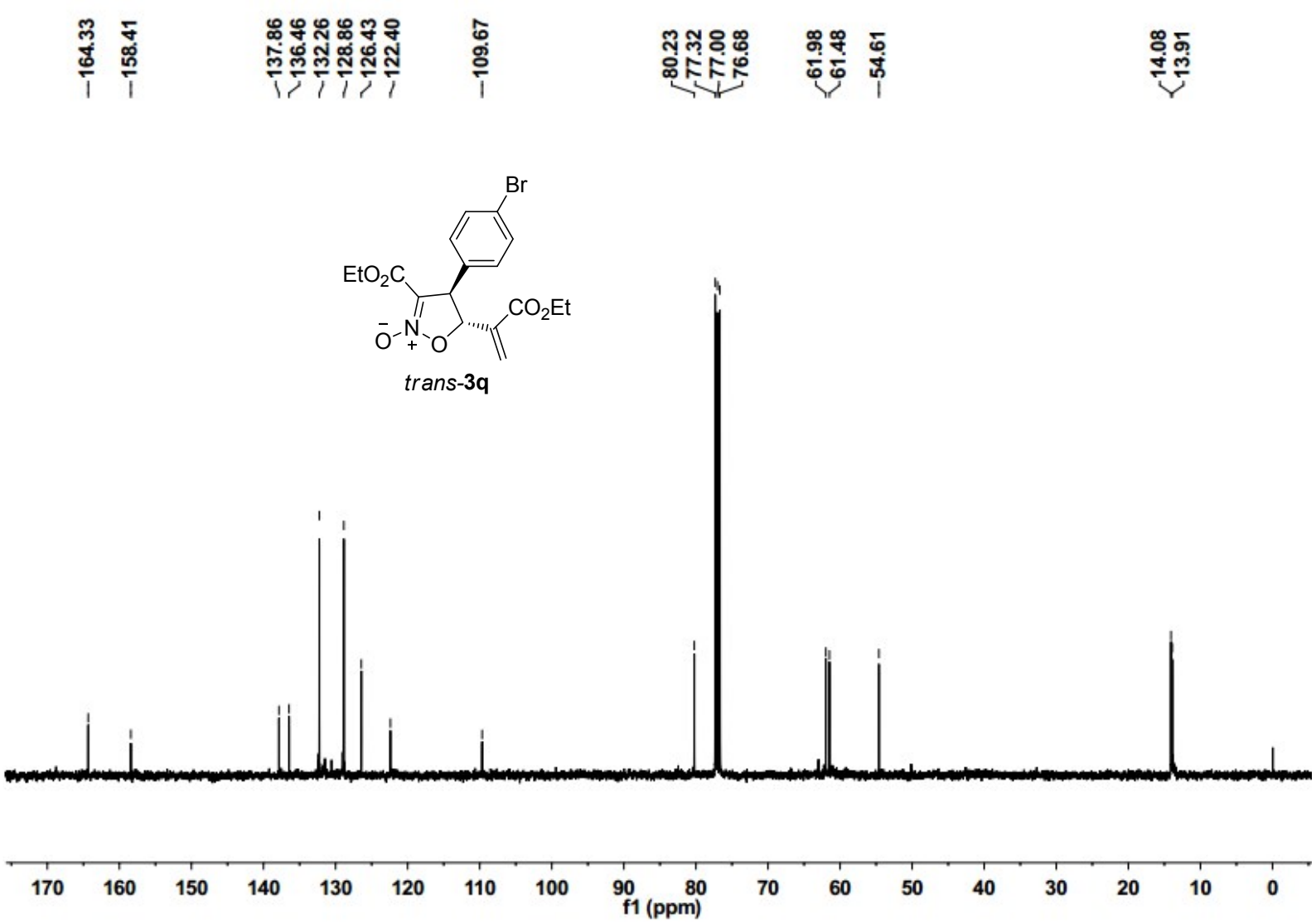
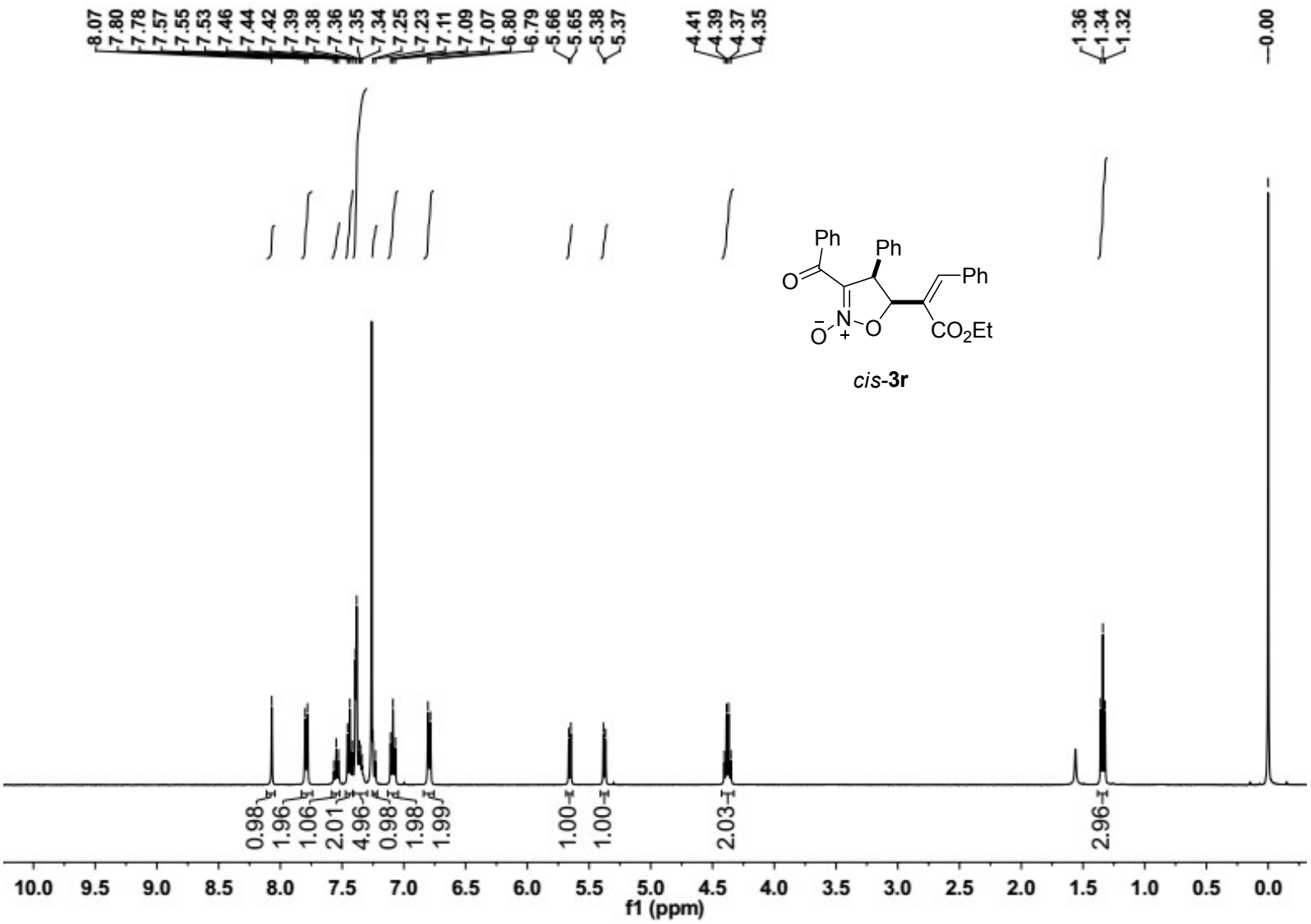
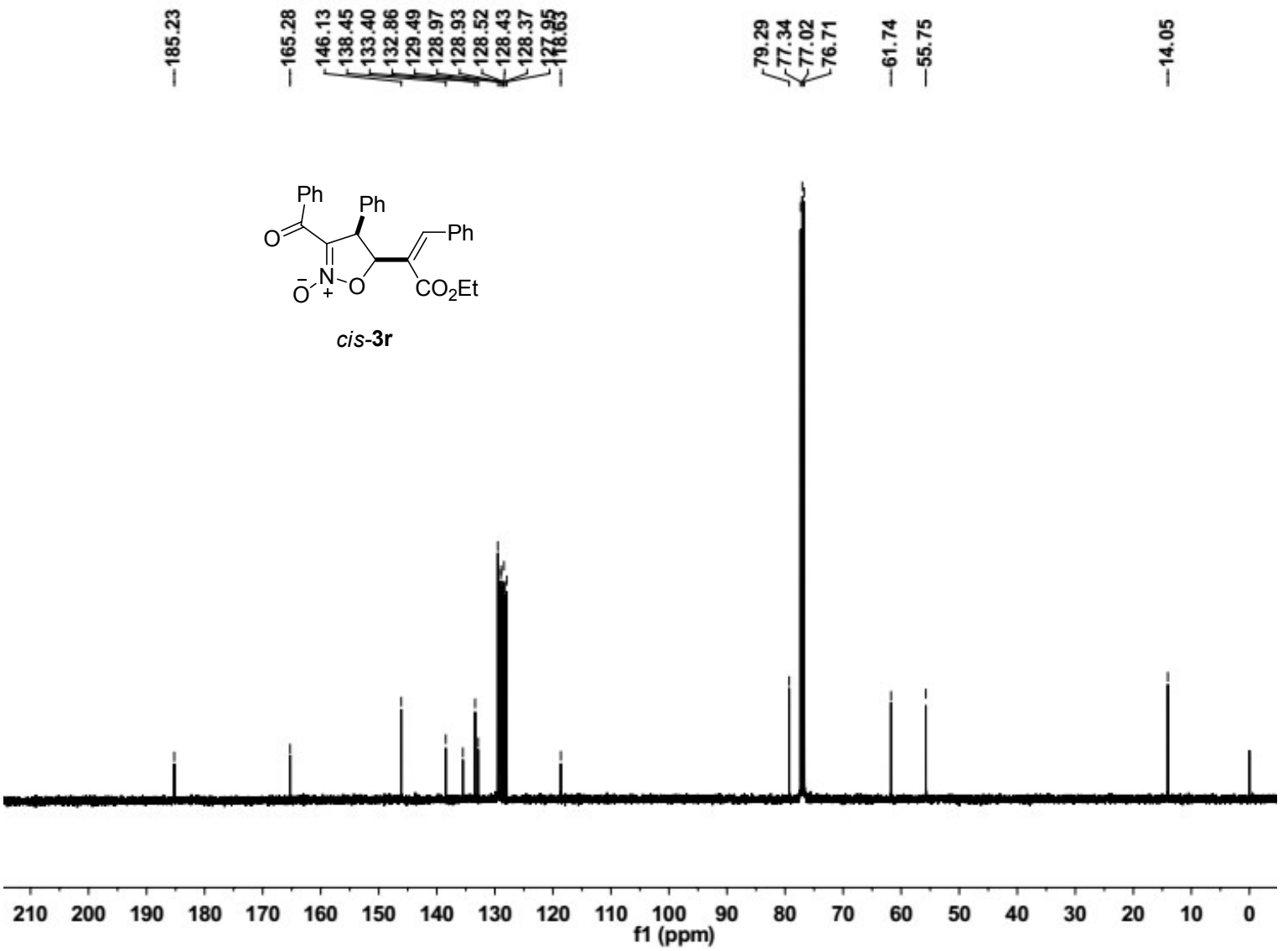
4.16
4.15
1.32
1.30
1.29
1.21
1.19
1.17
0.00
Br
EtO2C
CO2Et
N
O
O
2.02
1.98
1.00
0.99
1.00
0.97
2.07
2.09
3.02
3.09
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

trans-**3q**

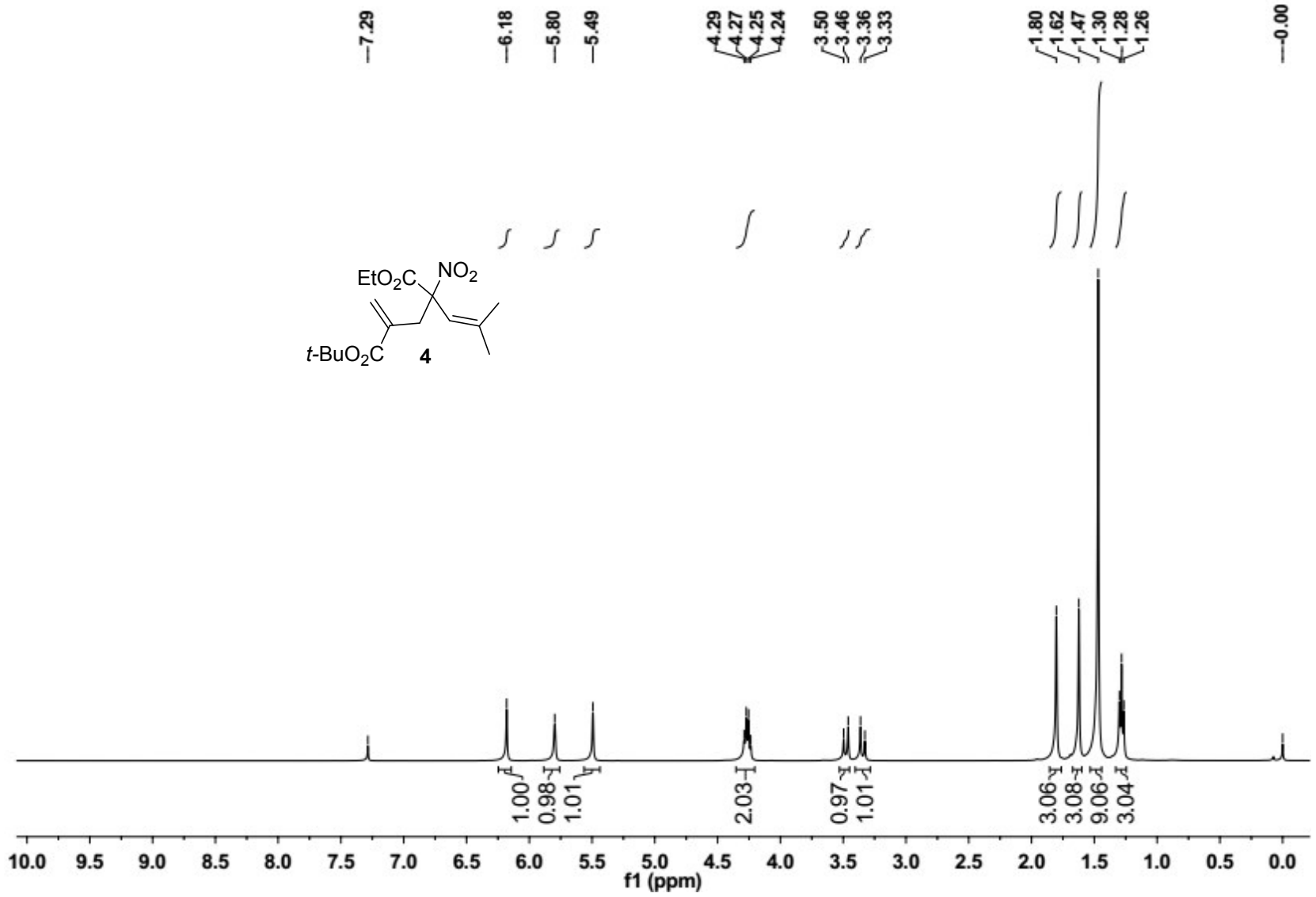

164.33
158.41
137.86
136.46
132.26
128.86
126.43
122.40
109.67
80.23
77.32
77.00
76.68
61.98
61.48
54.61
14.08
13.91
Br
EtO2C
CO2Et
trans-3q
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

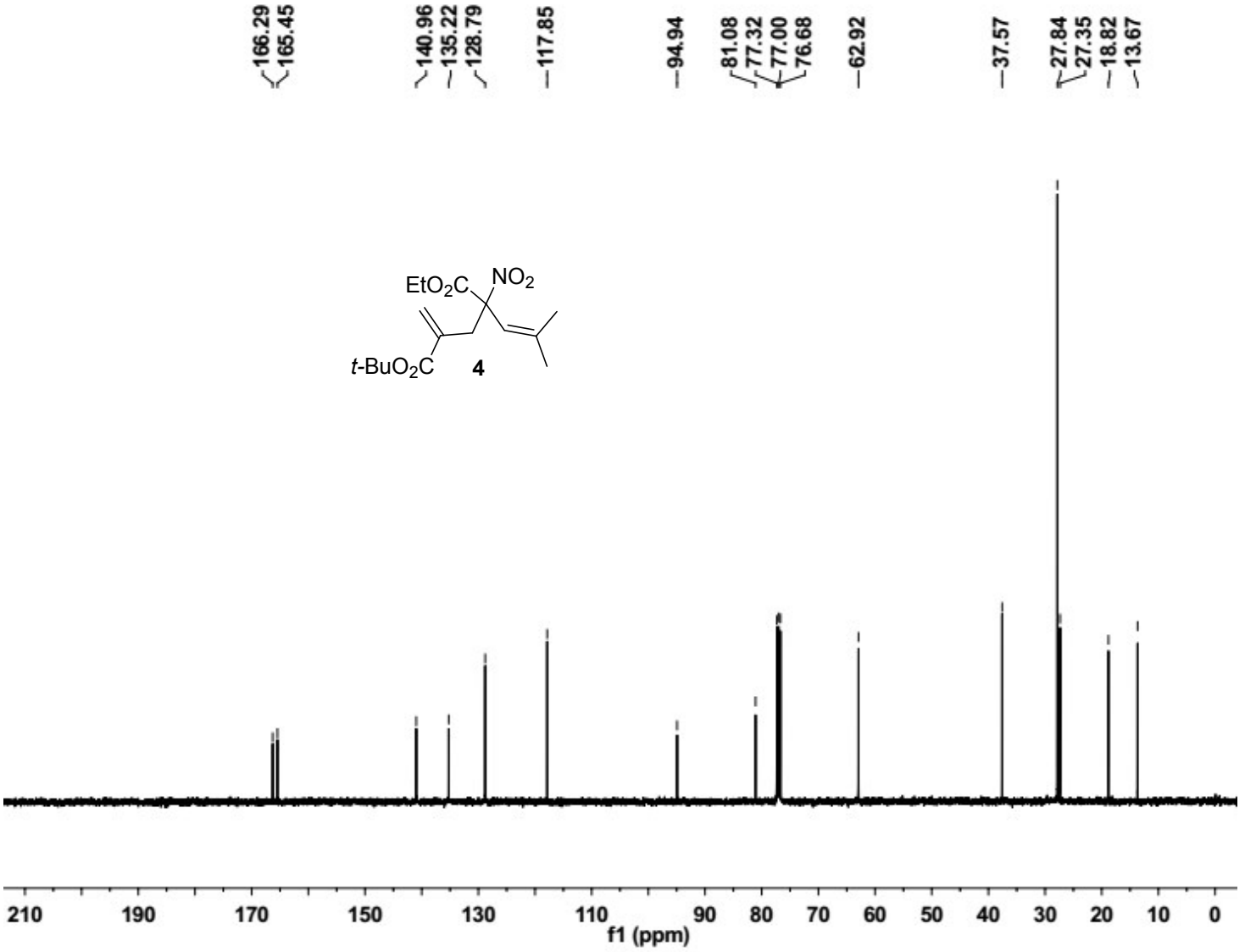

8.07
7.80
7.78
7.57
7.55
7.53
7.46
7.44
7.42
7.39
7.38
7.36
7.35
7.34
7.25
7.23
7.11
7.09
7.07
6.80
6.79
5.66
5.65
5.38
5.37
4.41
4.39
4.37
4.35
1.36
1.34
1.32
0.00
Ph
Ph
Ph
O
N
O
O
CO2Et
0.98
1.96
1.06
2.01
4.96
0.98
1.98
1.99
1.00
1.00
2.03
2.96
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

cis-**3r**


185.23
165.28
146.13
138.45
133.40
132.86
129.49
128.97
128.93
128.52
128.43
128.37
79.29
77.34
77.02
76.71
61.74
55.75
14.05
Ph
Ph
Ph
O
N
O
O
CO2Et
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

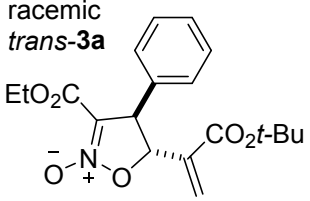
cis-**3r**

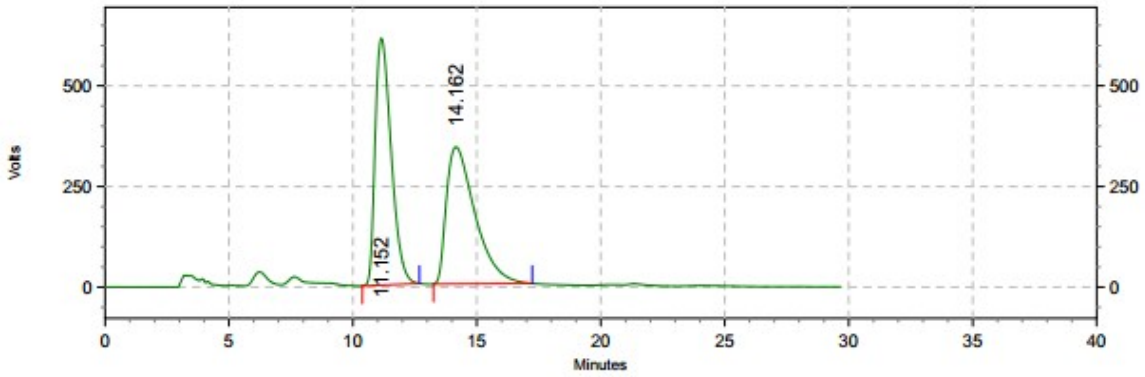

7.29
6.18
5.80
5.49
4.29
4.27
4.25
4.24
3.50
3.46
3.36
3.33
1.80
1.62
1.47
1.30
1.28
1.26
0.00
EtO2C
NO2
t-BuO2C
4
1.00
0.98
1.01
2.03
0.97
1.01
3.06
3.08
9.06
3.04
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


166.29
165.45
140.96
135.22
128.79
117.85
94.94
81.08
77.32
77.00
76.68
62.92
37.57
27.84
27.35
18.82
13.67
EtO2C
NO2
t-BuO2C
4
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

VIII. Chiral HPLC Graphs for 3a-3q

racemic
trans-**3a**

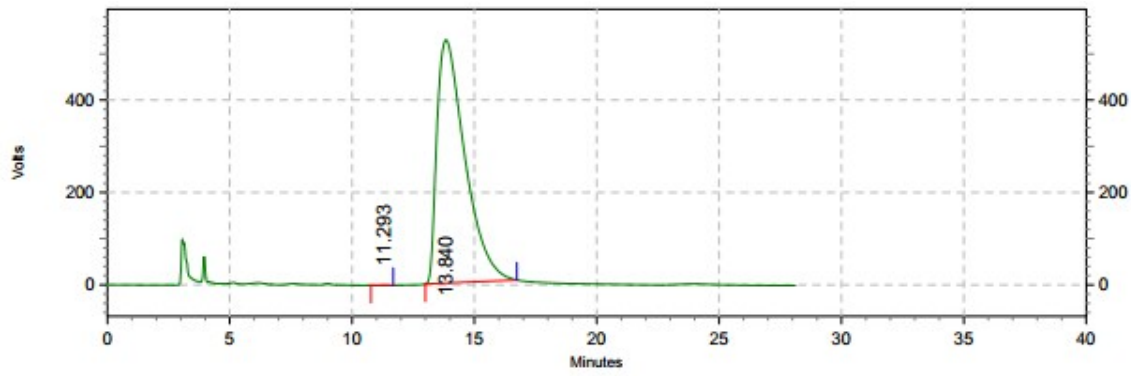




UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
11.152	27533020	50.187	612664	64.289
14.162	27327559	49.813	340325	35.711
Totals	54860579	100.000	952989	100.000

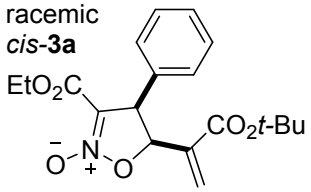
trans-**3a**

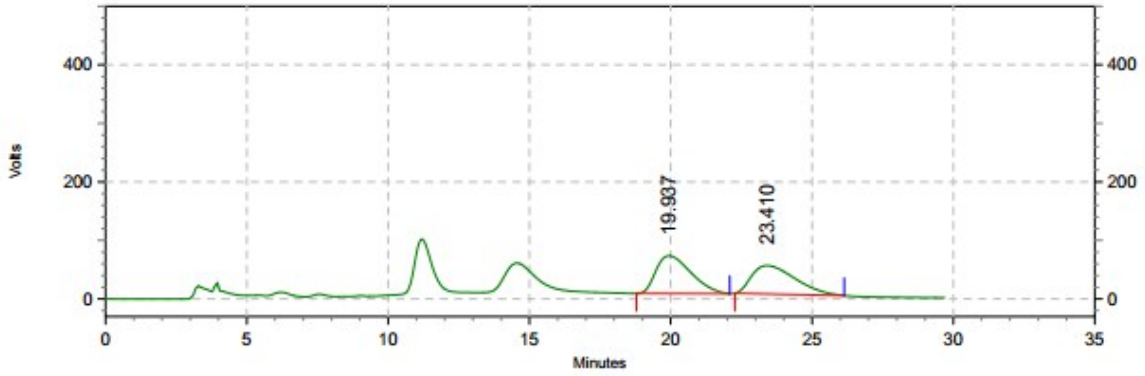


UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
11.293	3108	0.007	122	0.023
13.840	41883442	99.993	526976	99.977
Totals	41886550	100.000	527098	100.000

racemic
cis-**3a**

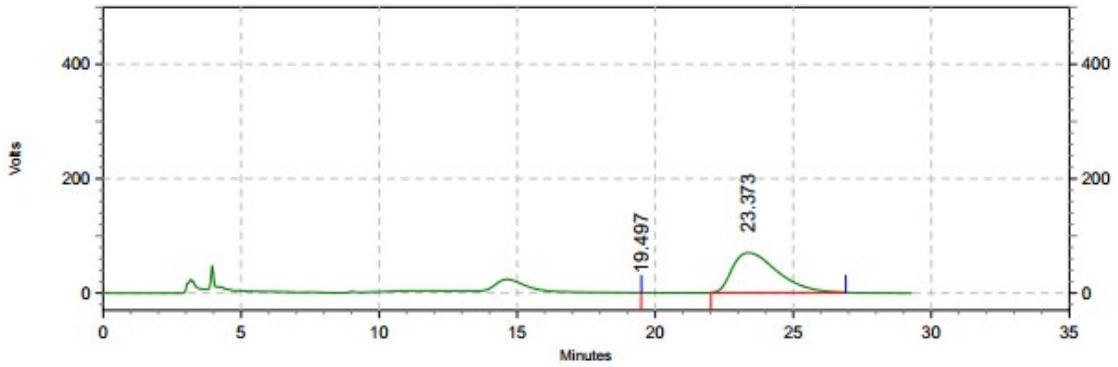




UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
19.937	5545649	51.634	64471	57.123
23.410	5194716	48.366	48392	42.877
Totals	10740365	100.000	112863	100.000

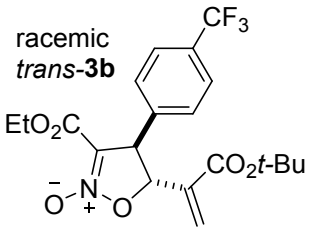
cis-**3a**

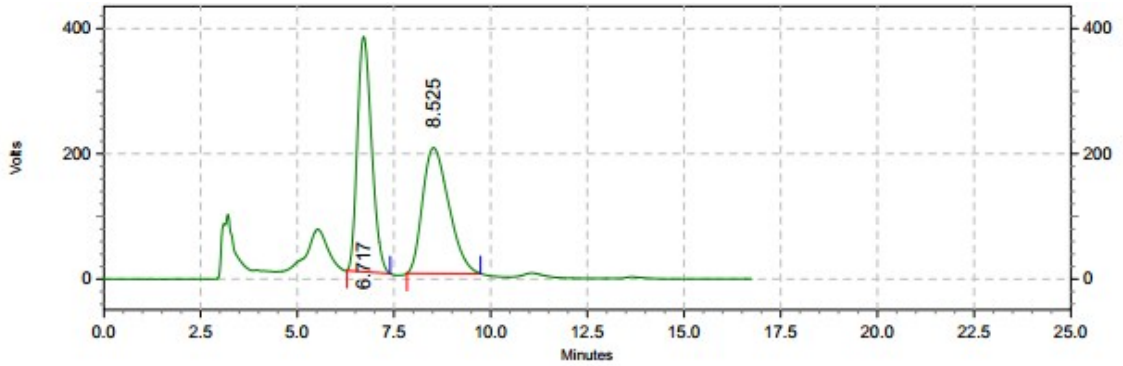


UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
19.497	0	0.000	0	0.000
23.373	7918252	100.000	69542	100.000
Totals	7918252	100.000	69542	100.000

racemic
trans-**3b**

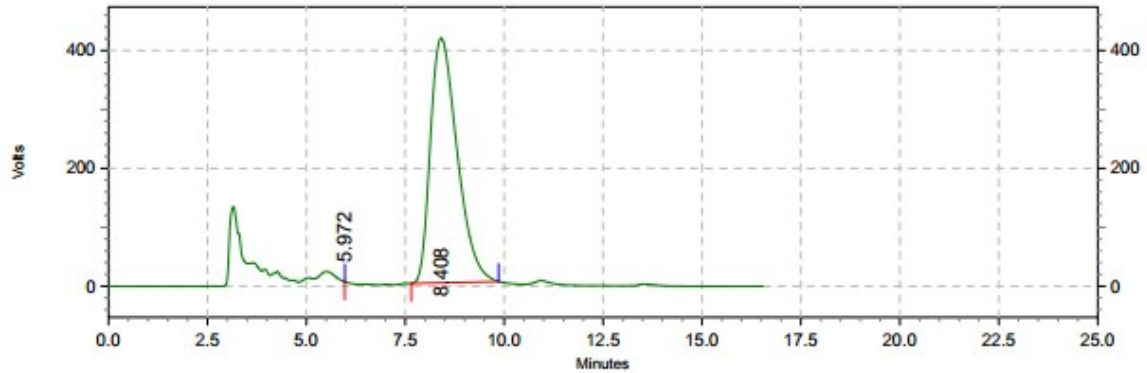




UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
6.717	9643831	50.464	375384	65.172
8.525	9466430	49.536	200604	34.828
Totals	19110261	100.000	575988	100.000

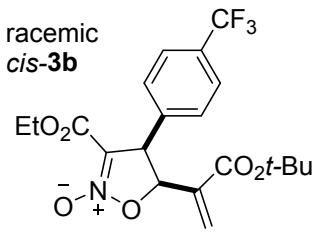
trans-**3b**

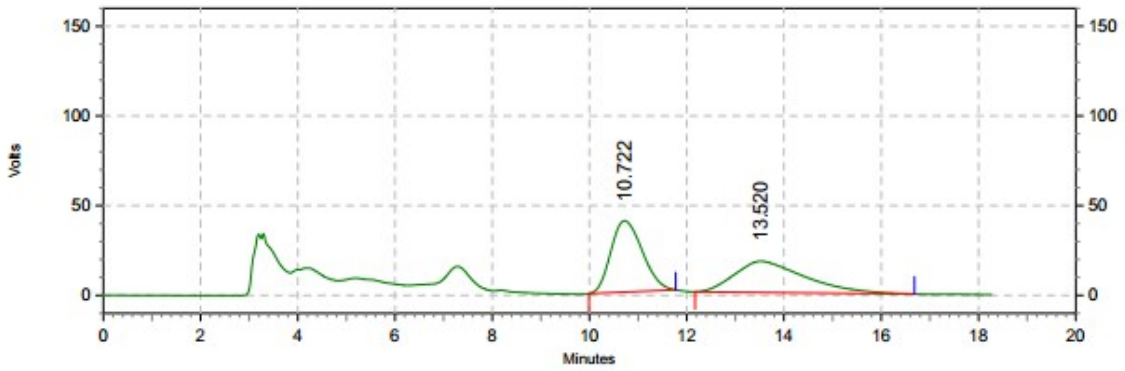


UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
5.972	0	0.000	0	0.000
8.408	19572421	100.000	415532	100.000
Totals	19572421	100.000	415532	100.000

racemic
cis-**3b**

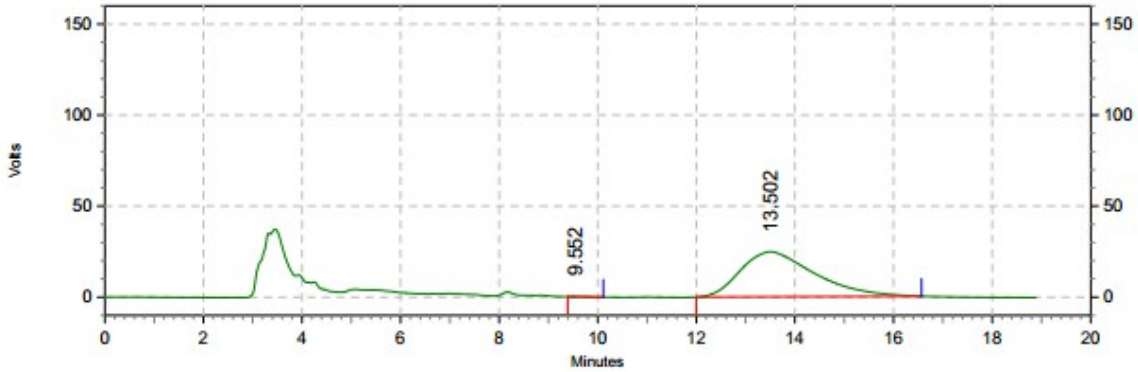




UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
10.722	1790949	50.535	39744	69.474
13.520	1753046	49.465	17463	30.526
Totals	3543995	100.000	57207	100.000

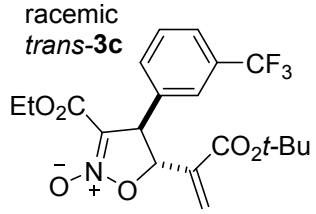
cis-**3b**

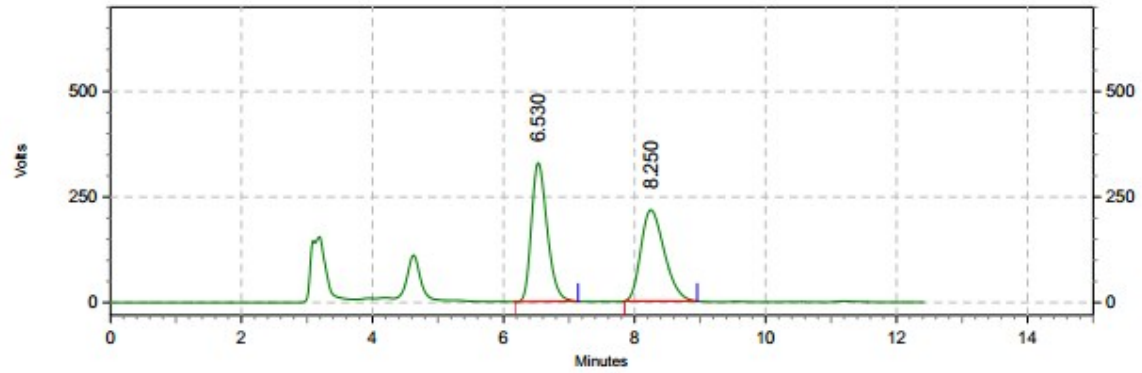


UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
9.552	2917	0.113	133	0.534
13.502	2581949	99.887	24795	99.466
Totals	2584866	100.000	24928	100.000

racemic
trans-**3c**

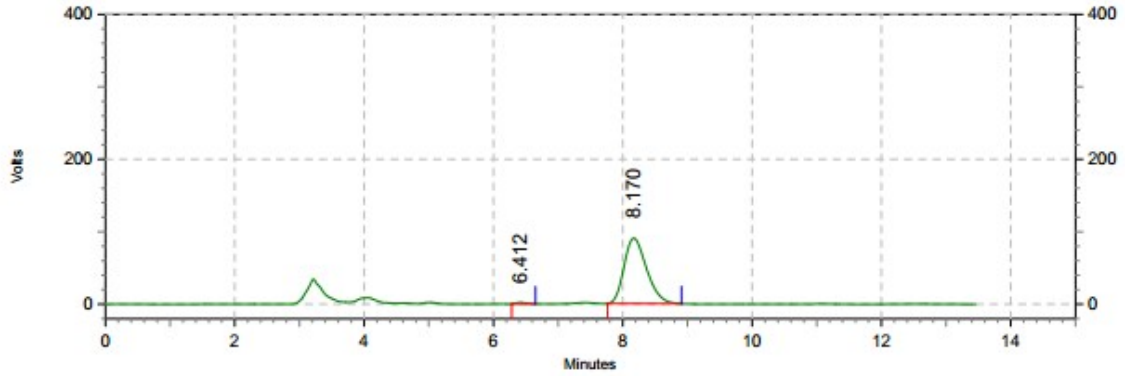




UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
6.530	5506758	50.530	328545	60.327
8.250	5391138	49.470	216065	39.673
Totals	10897896	100.000	544610	100.000

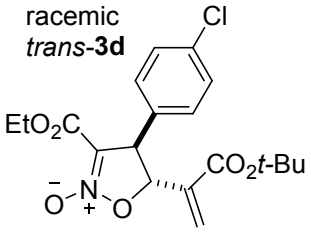
trans-**3c**

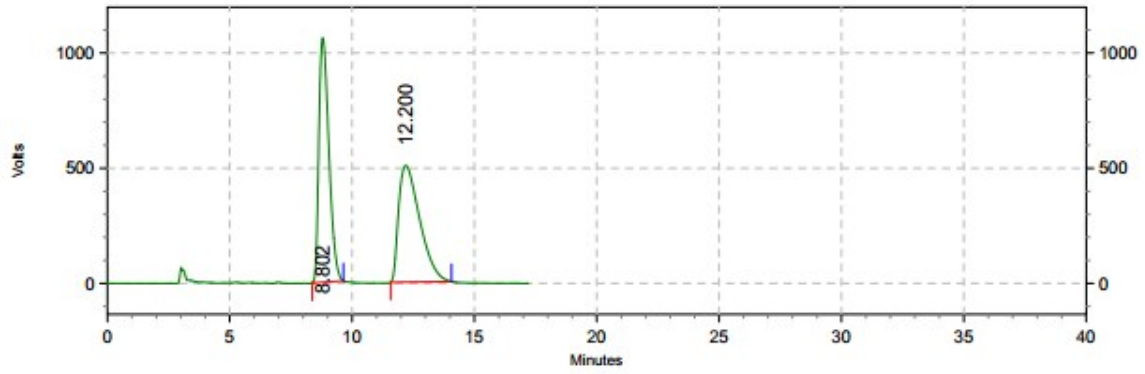


UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
6.412	20526	0.933	1916	2.081
8.170	2180271	99.067	90159	97.919
Totals	2200797	100.000	92075	100.000

racemic
trans-**3d**

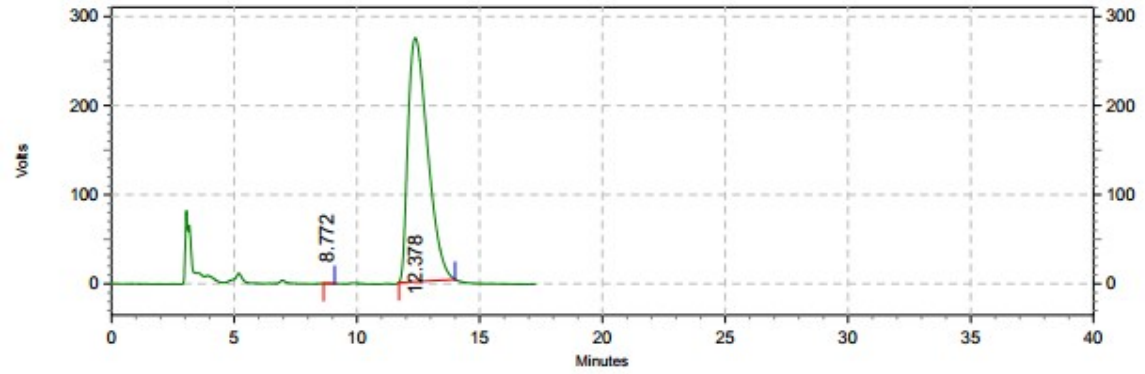




UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
8.802	30129387	49.834	1061604	67.695
12.200	30330667	50.166	506619	32.305
Totals	60460054	100.000	1568223	100.000

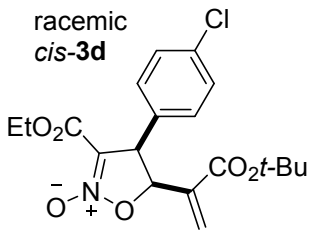
trans-**3d**

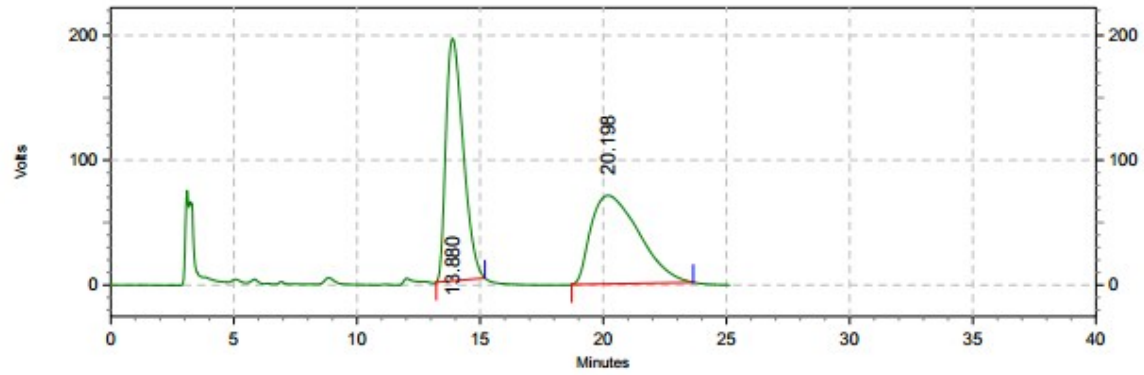


UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
8.772	4036	0.027	214	0.078
12.378	15221601	99.973	273517	99.922
Totals	15225637	100.000	273731	100.000

racemic
cis-**3d**

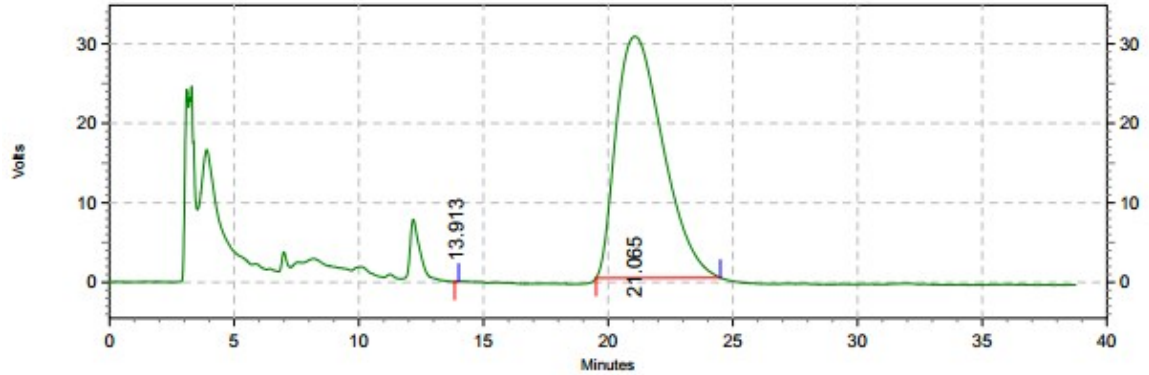




UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
13.880	9504854	49.672	194141	73.248
20.198	9630406	50.328	70906	26.752
Totals	19135260	100.000	265047	100.000

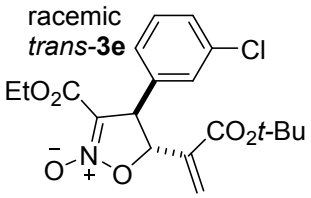
cis-**3d**

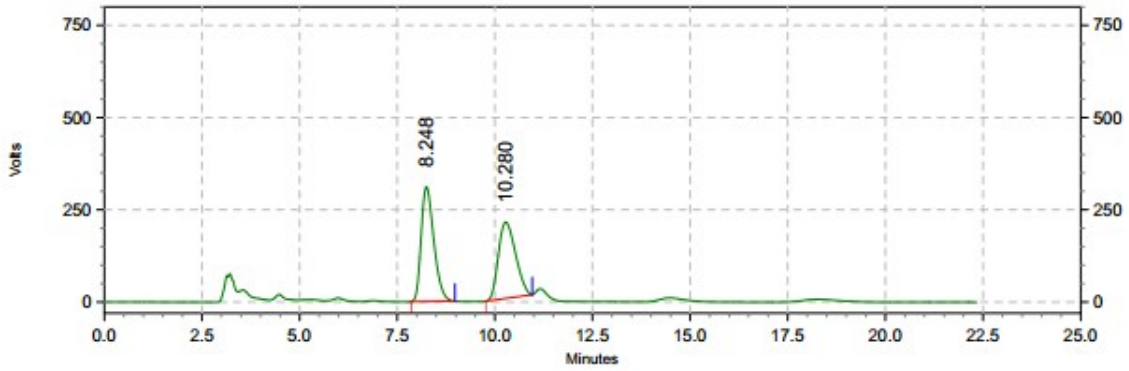


UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
13.913	118	0.003	30	0.099
21.065	3968224	99.997	30358	99.901
Totals	3968342	100.000	30388	100.000

racemic
trans-**3e**

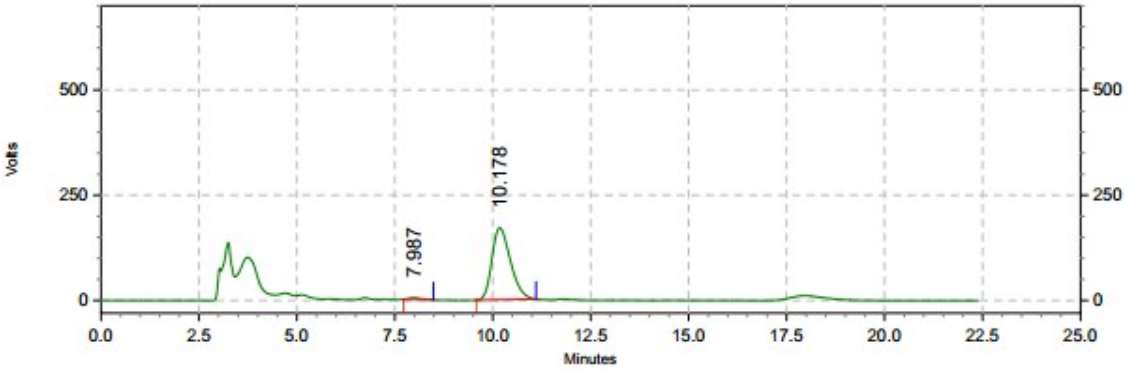




UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
8.248	7012547	52.562	310587	60.002
10.280	6328900	47.438	207044	39.998
Totals	13341447	100.000	517631	100.000

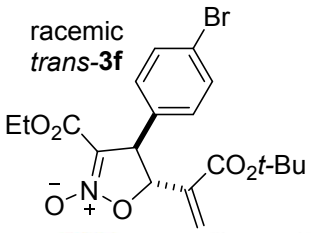
trans-**3e**

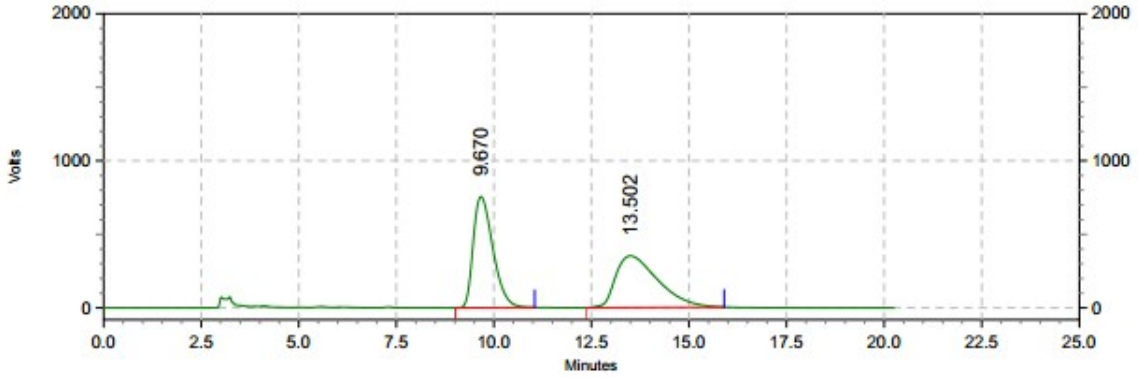


UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
7.987	102571	1.795	4468	2.552
10.178	5612354	98.205	170641	97.448
Totals	5714925	100.000	175109	100.000

racemic
trans-**3f**

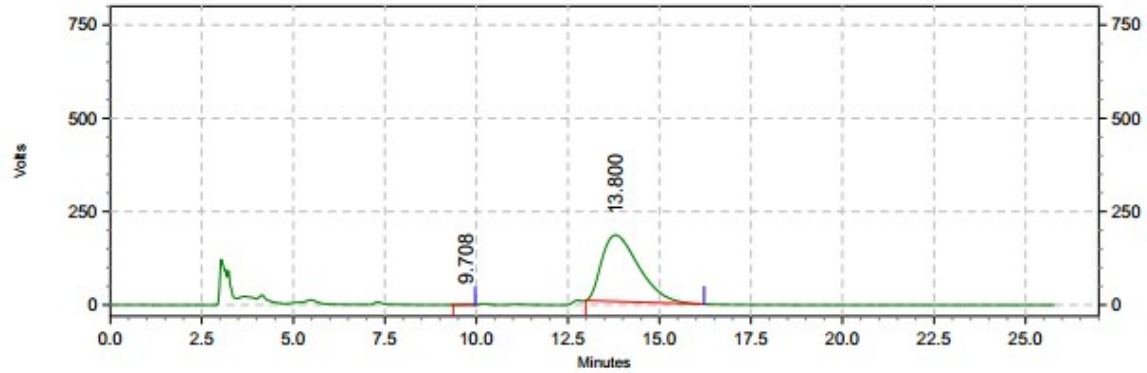




UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
9.670	26480873	50.017	755228	68.237
13.502	26463116	49.983	351546	31.763
Totals	52943989	100.000	1106774	100.000

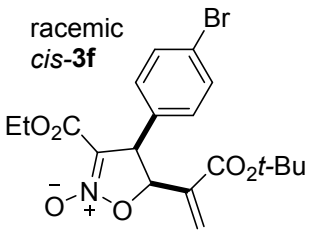
trans-**3f**

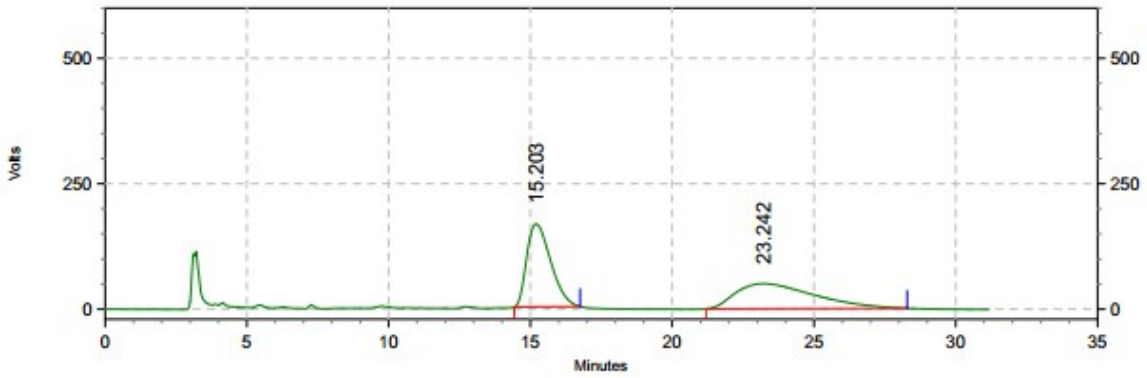


UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
9.708	9510	0.076	458	0.257
13.800	12552692	99.924	177771	99.743
Totals	12562202	100.000	178229	100.000

racemic
cis-**3f**

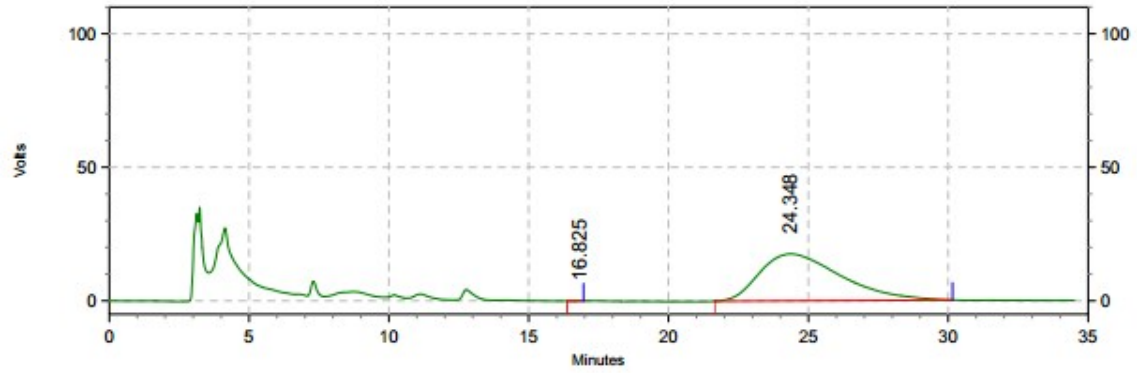




UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
15.203	9716514	50.353	165223	76.813
23.242	9580142	49.647	49875	23.187
Totals	19296656	100.000	215098	100.000

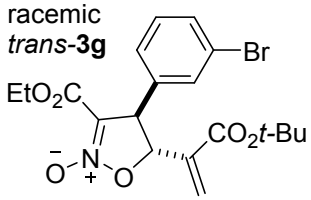
cis-**3f**

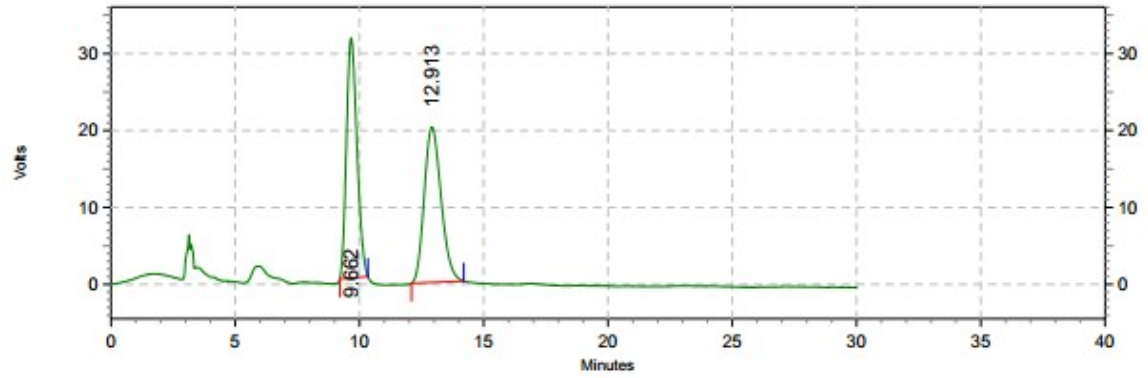


UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
16.825	707	0.020	42	0.239
24.348	3544917	99.980	17552	99.761
Totals	3545624	100.000	17594	100.000

racemic
trans-**3g**

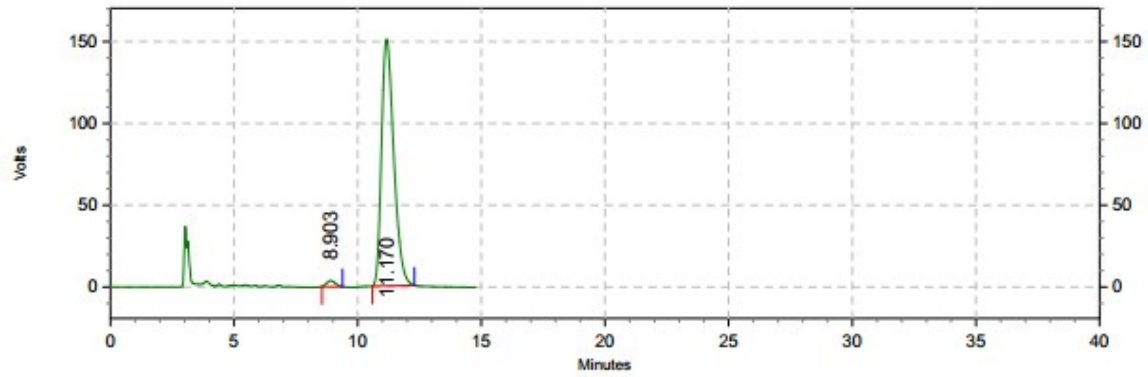




UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
9.662	914859	49.017	31189	60.686
12.913	951551	50.983	20205	39.314
Totals	1866410	100.000	51394	100.000

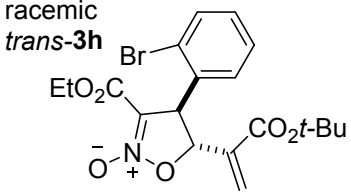
trans-**3g**

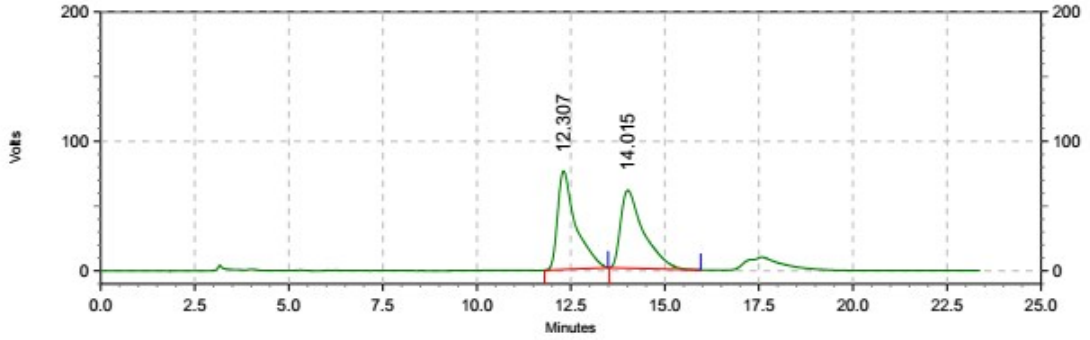


UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
8.903	89500	1.697	3662	2.371
11.170	5183183	98.303	150789	97.629
Totals	5272683	100.000	154451	100.000

racemic
trans-**3h**

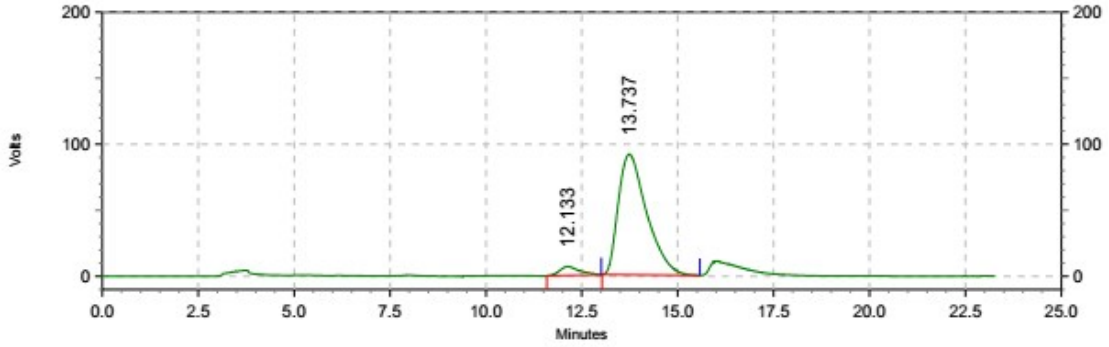




UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
12.307	2551788	50.002	76002	55.775
14.015	2551595	49.998	60263	44.225
Totals	5103383	100.000	136265	100.000

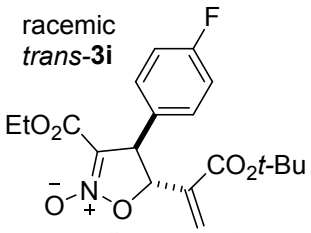
trans-**3h**

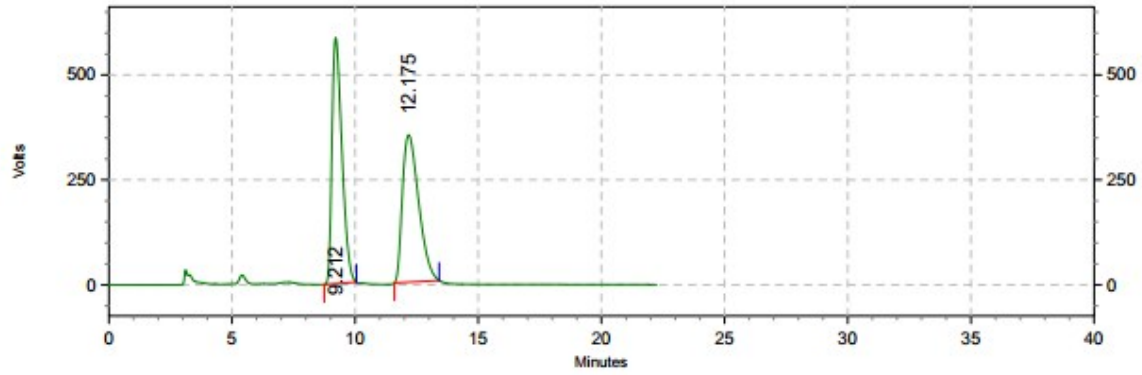


UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
12.133	238090	4.818	6687	6.830
13.737	4703986	95.182	91221	93.170
Totals	4942076	100.000	97908	100.000

racemic
trans-**3i**

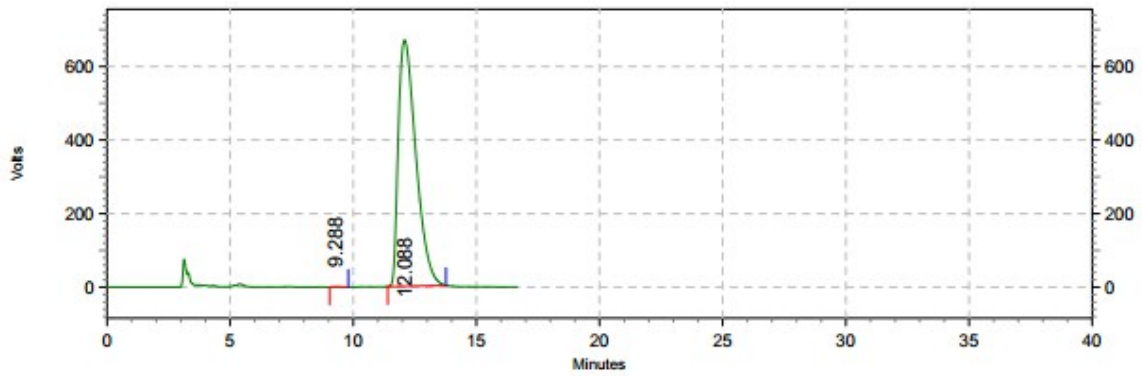




UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
9.212	16441379	50.564	585947	62.557
12.175	16074332	49.436	350721	37.443
Totals	32515711	100.000	936668	100.000

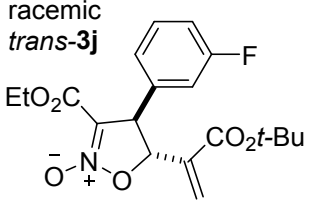
trans-**3i**

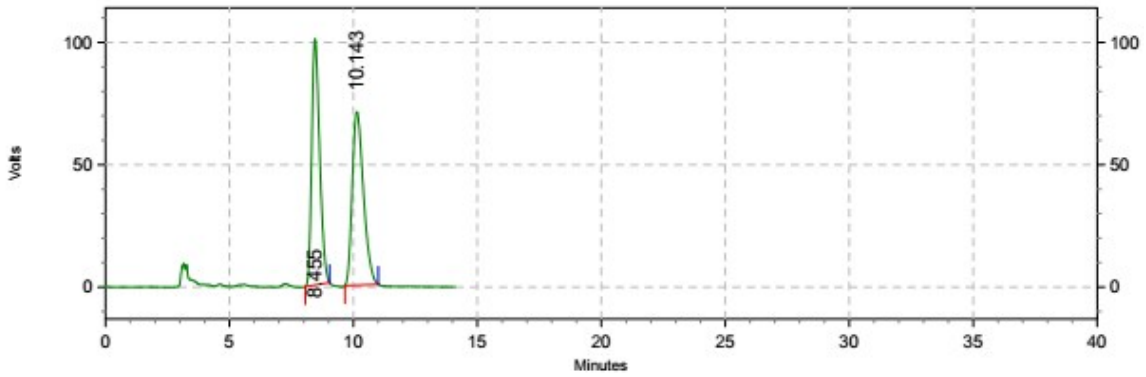


UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
9.288	18640	0.056	920	0.137
12.088	33201671	99.944	669329	99.863
Totals	33220311	100.000	670249	100.000

racemic
trans-**3j**

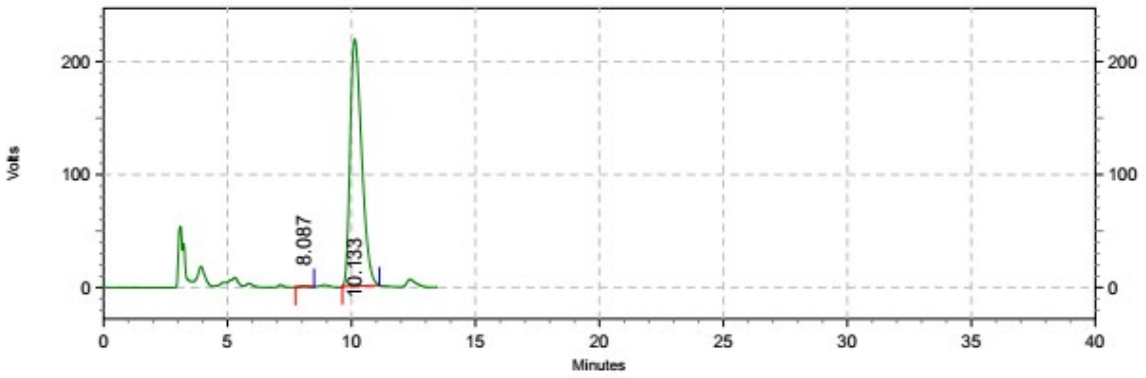




UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
8.455	2223527	49.918	100600	58.684
10.143	2230850	50.082	70827	41.316
Totals	4454377	100.000	171427	100.000

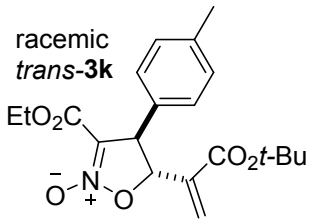
trans-**3j**

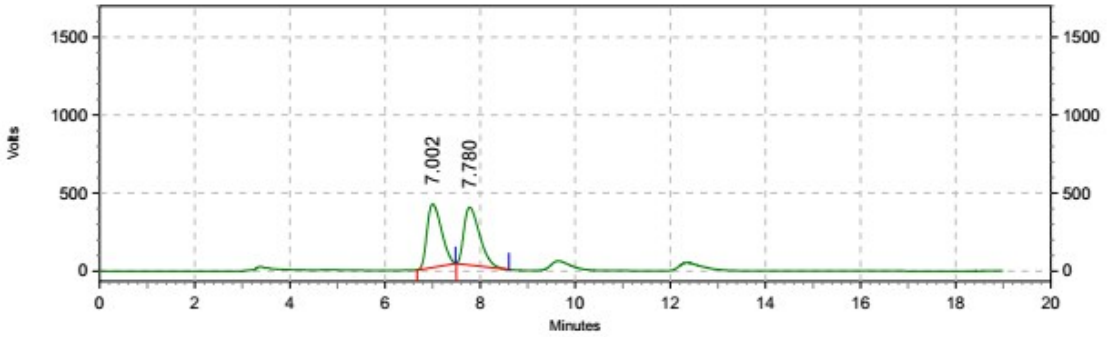


UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
8.087	29579	0.418	1233	0.561
10.133	7050706	99.582	218517	99.439
Totals	7080285	100.000	219750	100.000

racemic
trans-**3k**

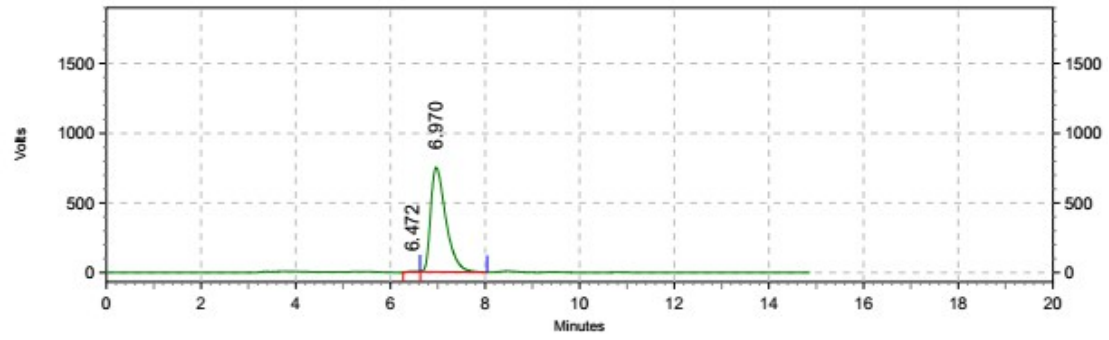




UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
7.002	8557536	50.053	405830	52.260
7.780	8539302	49.947	370735	47.740
Totals	17096838	100.000	776565	100.000

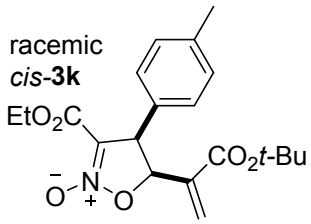
trans-**3k**

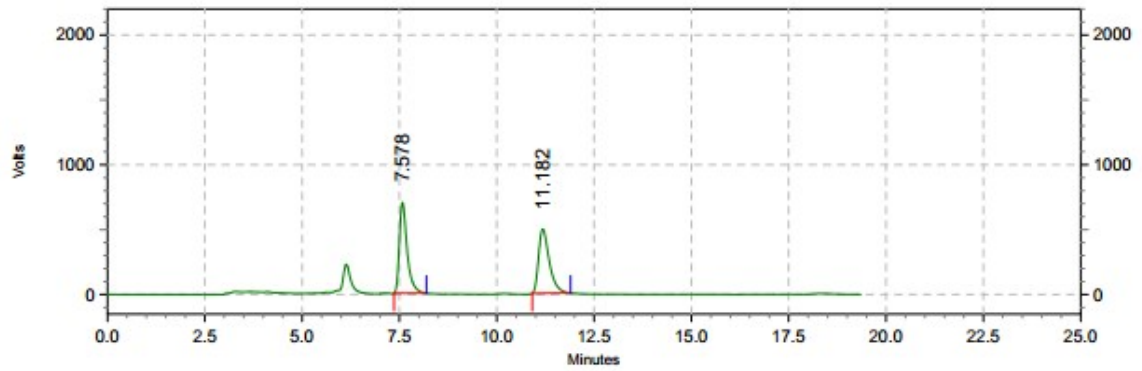


UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
6.472	49016	0.294	3869	0.517
6.970	16628275	99.706	744643	99.483
Totals	16677291	100.000	748512	100.000

racemic
cis-**3k**

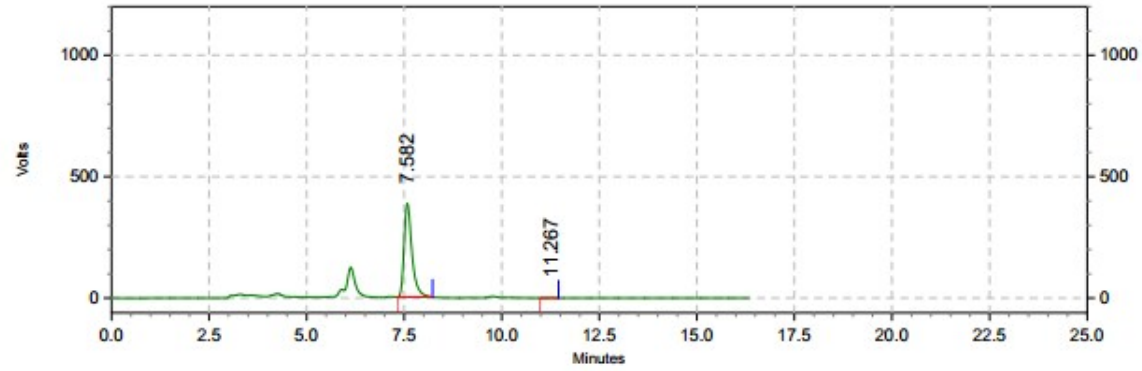




UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
7.578	9719782	50.595	698754	58.543
11.182	9491062	49.405	494822	41.457
Totals	19210844	100.000	1193576	100.000

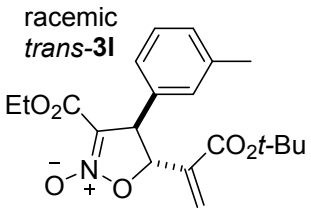
cis-**3k**

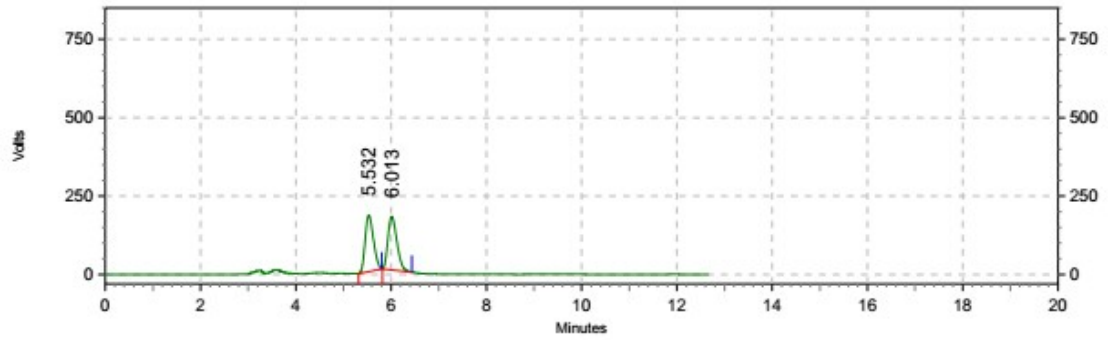


UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
7.582	5451412	99.924	384762	99.929
11.267	4151	0.076	273	0.071
Totals	5455563	100.000	385035	100.000

racemic
trans-**3l**

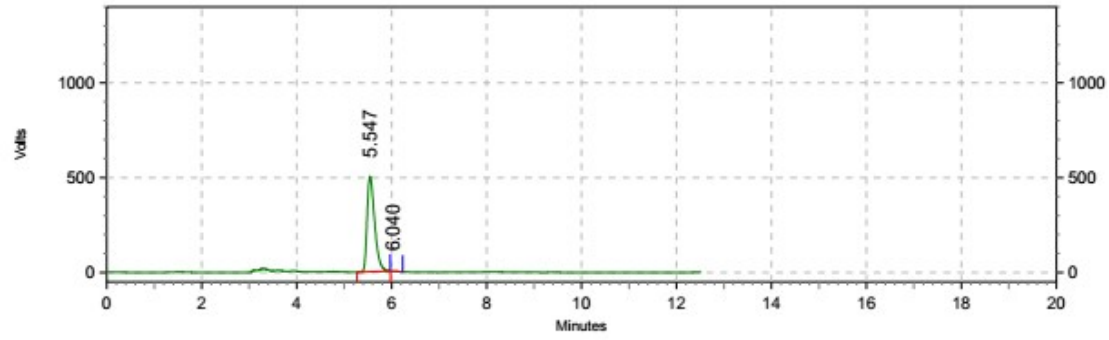




UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
5.532	2171395	49.469	179482	51.408
6.013	2218014	50.531	169653	48.592
Totals	4389409	100.000	349135	100.000

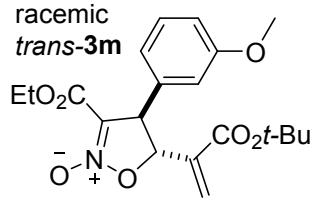
trans-**3l**

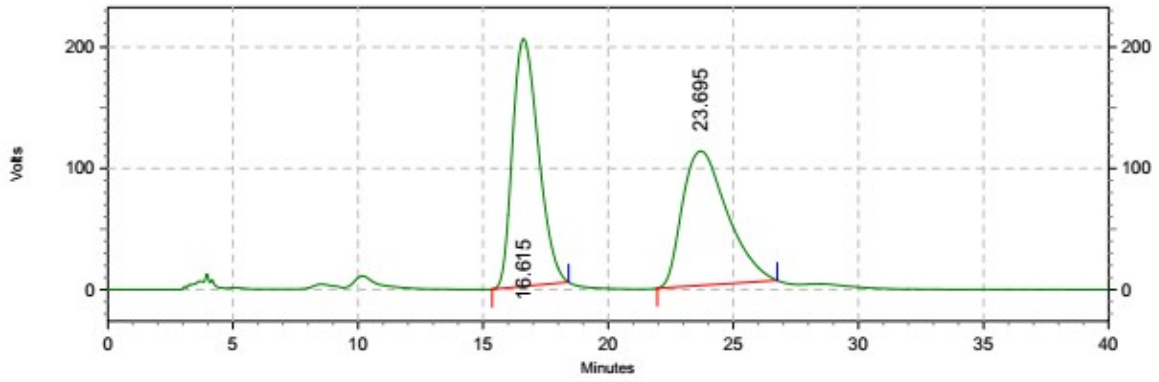


UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
5.547	5547481	99.790	502580	99.756
6.040	11656	0.210	1230	0.244
Totals	5559137	100.000	503810	100.000

racemic
trans-**3m**

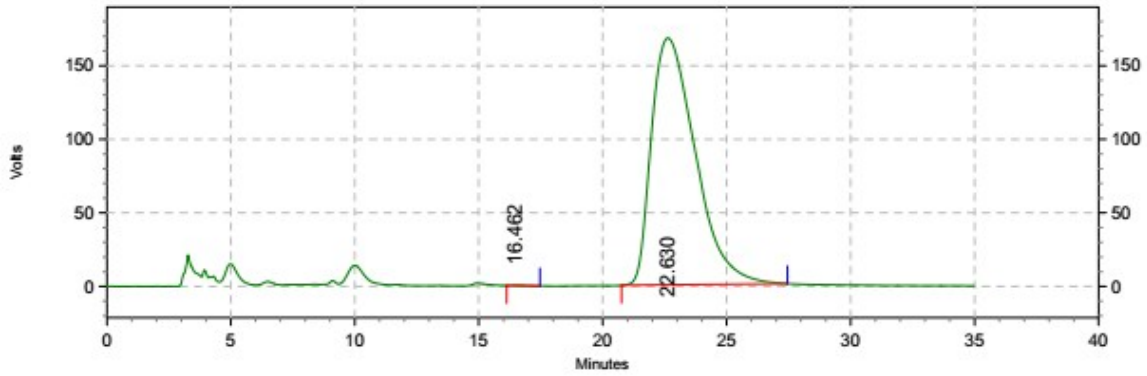




UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
16.615	14705526	51.529	204155	64.828
23.695	13832996	48.471	110761	35.172
Totals	28538522	100.000	314916	100.000

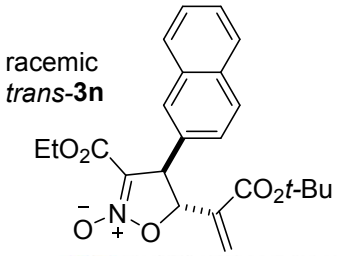
trans-**3m**

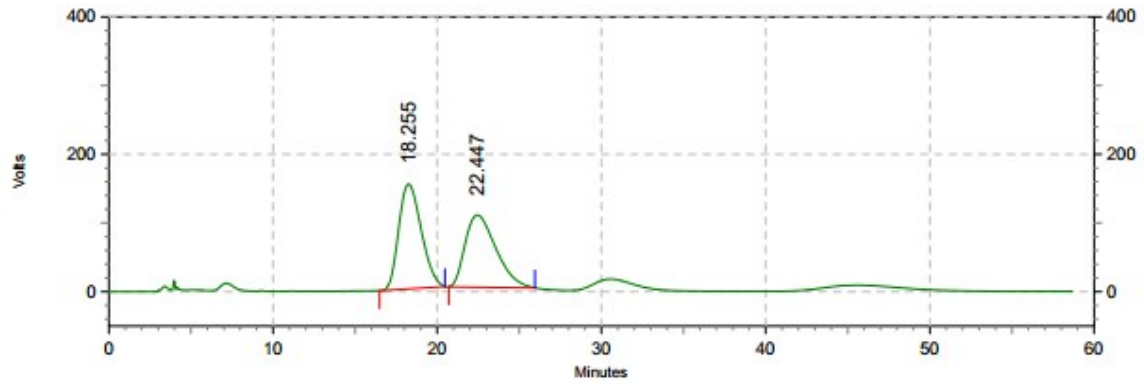


UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
16.462	6289	0.030	230	0.137
22.630	20613591	99.969	167688	99.863
Totals	20619880	100.000	167918	100.000

racemic
trans-**3n**

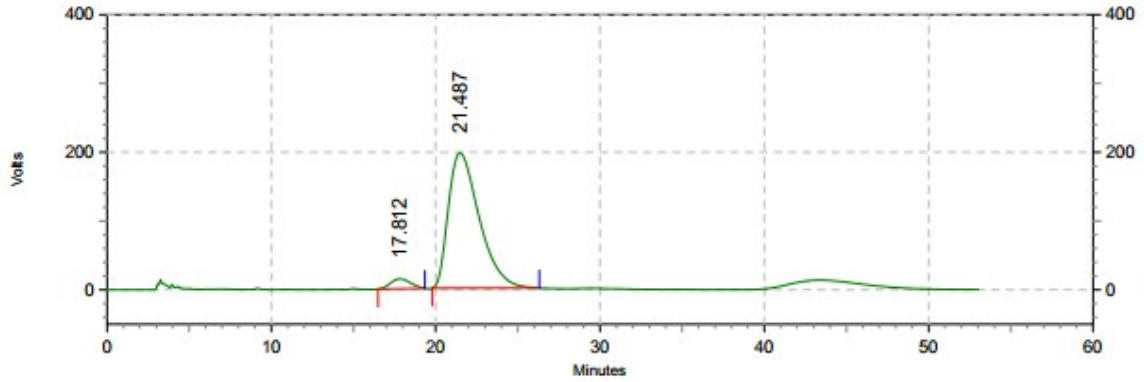




UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
18.255	14551198	51.504	152654	59.227
22.447	13701180	48.496	105088	40.773
Totals	28252378	100.000	257742	100.000

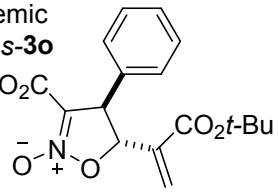
trans-**3n**

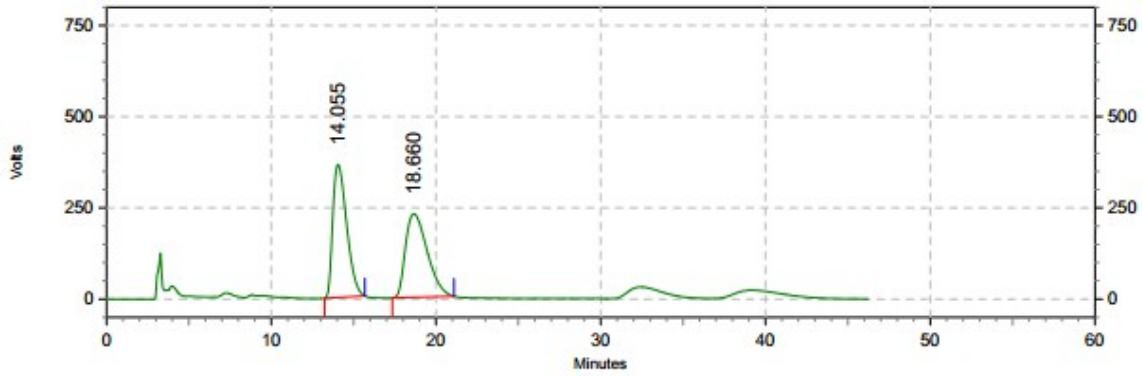


UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
17.812	1195341	4.456	14294	6.783
21.487	25631982	95.544	196433	93.217
Totals	26827323	100.000	210727	100.000

racemic
trans-**3o**

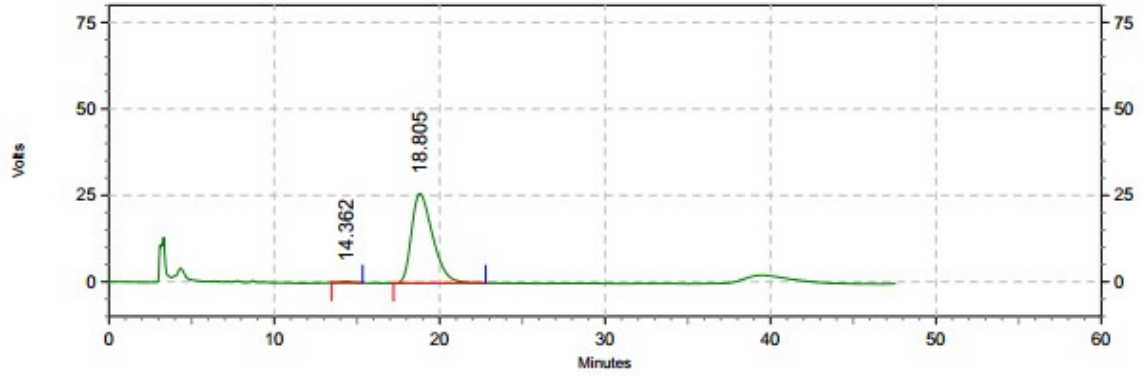




UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
14.055	21315070	50.302	364042	61.387
18.660	21058759	49.698	228985	38.613
Totals	42373829	100.000	593027	100.000

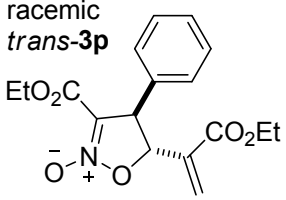
trans-**3o**

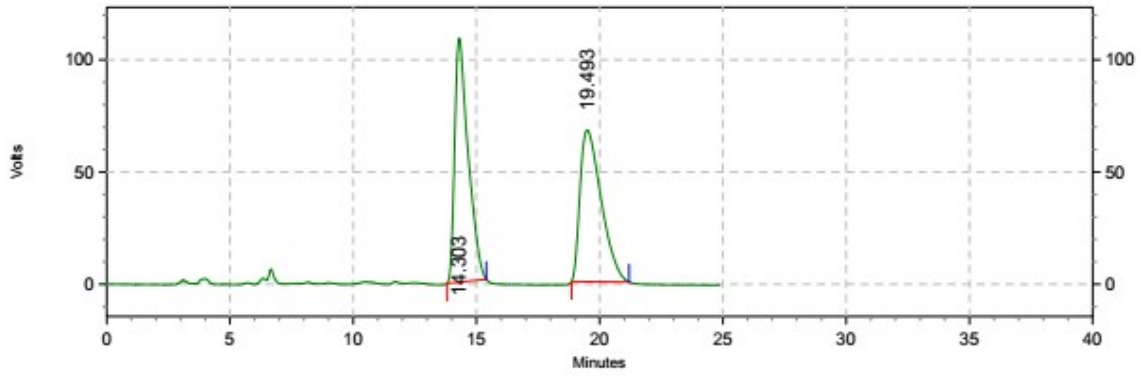


UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
14.362	19216	0.828	369	1.406
18.805	2302191	99.172	25879	98.594
Totals	2321407	100.000	26248	100.000

racemic
trans-**3p**

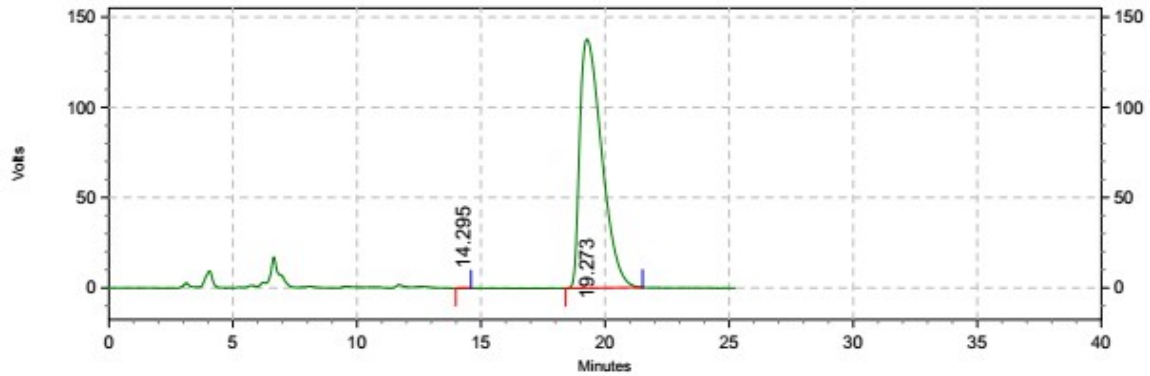




UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
14.303	4120353	50.586	108682	61.664
19.493	4024858	49.414	67566	38.336
Totals	8145211	100.000	176248	100.000

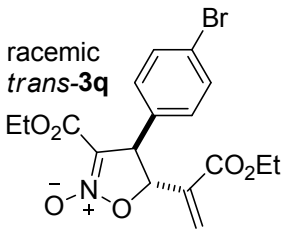
trans-**3p**

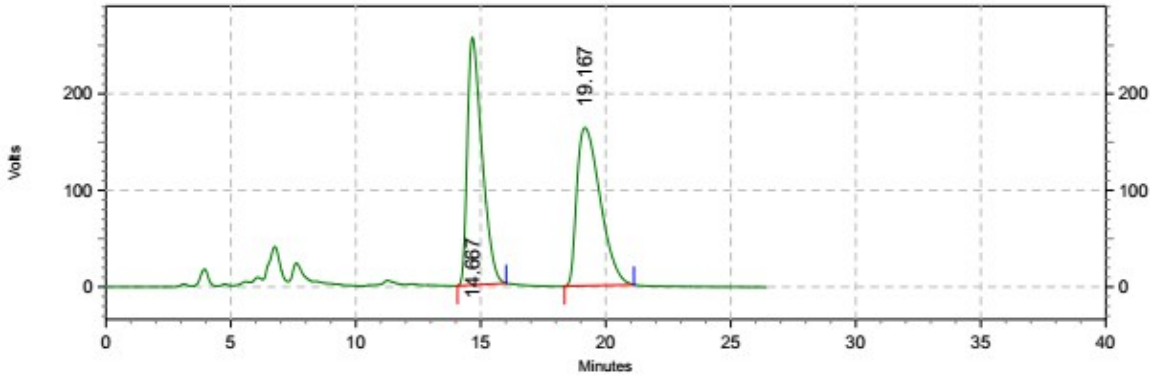


UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
14.295	1383	0.016	68	0.049
19.273	8470213	99.984	137874	99.951
Totals	8471596	100.000	137942	100.000

racemic
trans-**3q**

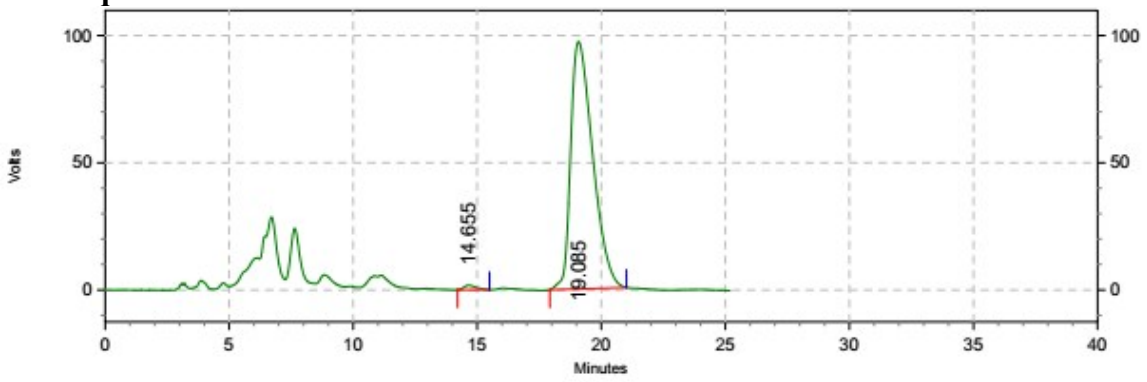




UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
14.667	10461764	49.559	256389	61.019
19.167	10647896	50.441	163787	38.981
Totals	21109660	100.000	420176	100.000

trans-**3q**



UV1000-254nm
Results

Retention Time	Area	Area%	Height	Height%
14.655	60024	0.990	1731	1.748
19.085	6004628	99.010	97283	98.252
Totals	6064652	100.000	99014	100.000