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Tracking the reactivity of arylideneimidazol-4-ones in the Diels–Alder cycloaddition

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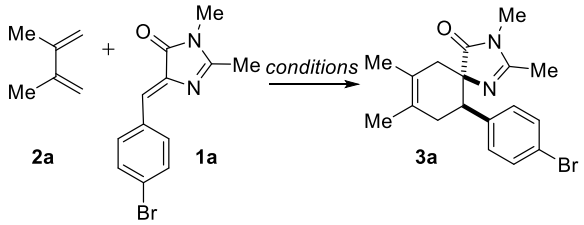
Optimization of LA-catalyzed cycloaddition



Scheme S1.

Table S1. Optimization of the reaction conditions. Lewis-acid catalysis

Entry	LA	Conditions	**3a**, yield
1	none	C_6H_5Cl, 130 °C, 16 h	<5%
2	$AlCl_3$ (1.0 equiv.)	CH_2Cl_2, −25 °C, 24 h	30%
3	$AlCl_3$ (1.0 equiv.)	CH_2Cl_2, 0 °C, 24 h	23%
4	$AlCl_3$ (1.0 equiv.)	CH_2Cl_2, 25°C, 24 h	18%
5	$AlCl_3$ (1.0 equiv.)	CH_3NO_2, −25°C, 24 h	15%
6	$AlCl_3$ (1.0 equiv.)	$C_6H_5CF_3$, −25°C, 24 h	<5%
7	$AlCl_3$ (1.5 equiv.)	CH_2Cl_2, −25°C, 24 h	37%
8	$AlCl_3$ (2.0 equiv.)	CH_2Cl_2, −25°C, 24 h	53%
9	$TiCl_4$ (2.0 equiv.)	CH_2Cl_2, −25°C, 24 h	50%

Experimental Part

^{1}H and ^{13}C NMR spectra were acquired on Bruker Avance II (800 and 201 MHz, respectively) and Bruker Fourier 300 (300 and 75 MHz, respectively) spectrometers in $CDCl_3$ and were calibrated to the residual solvent signal (^{1}H: $CHCl_3$ – 7.26 ppm; ^{13}C: $CDCl_3$ – 77.16 ppm). High-resolution mass spectra were recorded on an AB Sciex TripleTOF 5600+ instrument, electrospray ionization. Melting points were determined on a Kofler bench.

Typical procedure for $AlCl_3$-catalyzed Diels–Alder reaction:

A solution of arylidene imidazolone **1** (0.5 mmol, 1 equiv.) in CH_2Cl_2 (2 mL) in a screw-cap vial equipped with magnetic stirrer bar was cooled to −25 °C. Aluminium trichloride (134 mg, 1.0 mmol, 2.0 equiv.) and diene **2** (410 mg, 5.0 mmol, 10 equiv. for **2a**; 165 mg, 2.5 mmol, 5.0 equiv. for **2b**) were added successively. The reaction mixture was stirred for 30 min at −25 °C, then the vial was transferred into a freezer (−28 °C). After 24 h, the mixture was diluted with ethyl acetate and quenched with a saturated $NaHCO_3$ solution. This was transferred into separatory funnel with EtOAc (20 mL) and saturated aqueous $NaHCO_3$ solution (10 mL). The organic layer was separated, the aqueous layer was extracted with EtOAc (10 mL). The combined organic phase was washed with brine (20 mL), dried over anhydrous Na_2SO_4, filtered and evaporated to dryness on a rotary evaporator. Column chromatography of the residue on silica gel (eluent hexane/EtOAc 3/1 → 1/1) provided pure cycloaddition derivative **3** (10-89%).

(5*S,10*S**)-10-(4-Bromophenyl)-2,3,7,8-tetramethyl-1,3-diazaspiro[4.5]deca-1,7-dien-4-one 3a**

Yield 99 mg (0.27 mmol, 55%). White crystals, mp = 110−112 °C, R_f = 0.14 (SiO_2, hexane/EtOAc 1/1).

^{1}H NMR (300 MHz, $CDCl_3$, 300K): δ = 7.29 (d, *J* = 8.5 Hz, 2H), 7.02 (d, *J* = 8.5 Hz, 2H), 3.17 (dd, *J* = 12.4, 5.2 Hz, 1H), 2.81–2.64 (m, 2H), 2.60 (s, 3H), 2.08–1.98 (m, 1H), 2.03 (s, 3H), 1.77 (d, *J* = 17.0 Hz, 1H), 1.70 (s, 3H), 1.66 (s, 3H).

^{13}C{^{1}H} NMR (75 MHz, $CDCl_3$, 300K): δ = 184.4, 160.8, 138.6, 130.7 (×2), 130.4 (×2), 125.2, 122.0, 120.9, 73.5, 45.7, 40.0, 35.0, 26.2, 19.2, 18.7, 15.4.

HRMS (ESI+) m/z: calctd for $C_{18}H_{22}{}^{79}BrN_2O$ [M+H$^+$] 361.0910, found 361.0908.

(5*S,10*S**)-2,3,7,8-Tetramethyl-10-(4-nitrophenyl)-1,3-diazaspiro[4.5]deca-1,7-dien-4-one 3b**

Yield 106 mg (0.32 mmol, 65%). White crystals, mp = 149−151 °C, R_f = 0.14 (SiO_2, hexane/EtOAc 1/2).

^{1}H NMR (300 MHz, $CDCl_3$, 300K): δ = 8.04 (d, *J* = 8.8 Hz, 2H), 7.33 (d, *J* = 8.8 Hz, 2H), 3.35 (dd, *J* = 12.4, 5.2 Hz, 1H), 2.91–2.68 (m, 2H), 2.62 (s, 3H), 2.15–1.99 (m, 1H), 2.07 (s, 3H), 1.81 (d, *J* = 17.5 Hz, 1H), 1.73 (s, 3H), 1.69 (s, 3H).

^{13}C{^{1}H} NMR (75 MHz, $CDCl_3$, 300K): δ = 184.1, 161.2, 147.5, 147.1, 129.6 (×2), 124.8, 122.8 (×2), 122.2, 73.3, 46.0, 40.3, 34.8, 26.3, 19.2, 18.7, 15.4.

HRMS (ESI+) m/z: calctd for $C_{18}H_{22}N_3O_3$ [M+H$^+$] 328.1656, found 328.1655

(5*S,10*S**)-2,3,7,8-Tetramethyl-10-phenyl-1,3-diazaspiro[4.5]deca-1,7-dien-4-one 3c**

Yield 14 mg (0.05 mmol, 10%). Yellowish oil, R_f = 0.10 (SiO_2, hexane/EtOAc 1/1).

^{1}H NMR (300 MHz, $CDCl_3$, 300K): δ = 7.22–7.05 (m, 5H), 3.20 (ddd, *J* = 12.3, 5.3, 1.8 Hz, 1H), 2.88–2.65 (m, 2H), 2.53 (s, 3H), 2.15–2.01 (m, 1H), 2.01 (s, 3H), 1.79 (d, *J* = 17.3 Hz, 1H), 1.71 (s, 3H), 1.67 (s, 3H).

^{13}C{^{1}H} NMR (75 MHz, $CDCl_3$, 300K): δ = 184.6, 160.5, 139.4, 128.6 (×2), 127.5 (×2), 127.0, 125.4, 121.9, 73.7, 46.5, 39.8, 35.0, 26.0, 19.2, 18.7, 15.3.

HRMS (ESI+) m/z: calctd for $C_{18}H_{23}N_2O$ [M+H$^+$] 283.1805, found 283.1805.

(5*S,10*S**)-10-(4-Methoxyphenyl)-2,3,7,8-tetramethyl-1,3-diazaspiro[4.5]deca-1,7-dien-4-one 3d**

Yield 42 mg (0.13 mmol, 27%). Yellowish amorphous solid, R_f = 0.17 (SiO_2, hexane/EtOAc 1/2).

^{1}H NMR (800 MHz, $CDCl_3$, 298K): δ = 7.10 (d, *J* = 8.6 Hz, 2H), 6.75 (d, *J* = 8.6 Hz, 2H), 3.77 (s, 3H), 3.20 (dd, *J* = 12.2, 5.3 Hz, 1H), 2.83–2.74 (m, 2H), 2.61 (s, 3H), 2.08 (dd, *J* = 16.5, 6.4 Hz, 1H), 2.06 (s, 3H), 1.82 (d, *J* = 17.1 Hz, 1H), 1.75 (s, 3H), 1.71 (s, 3H).

$^{13}C\{^{1}H\}$ NMR (201 MHz, $CDCl_3$, 298K): δ = 184.9, 160.4, 158.6, 131. 7, 129.6 (×2), 125.5, 121.9, 113.0 (×2), 74.0, 55.3, 45.6, 39.9, 35.4, 26.2, 19.2, 18.7, 15.4.

HRMS (ESI+) m/z: calctd for $C_{19}H_{25}N_2O_2$ [M+H$^+$] 313.1911, found 313.1915.

(5*S,10*S**)-10-(3-Chlorophenyl)-2,3,7,8-tetramethyl-1,3-diazaspiro[4.5]deca-1,7-dien-4-one 3e**

Yield 79 mg (0.25 mmol, 50%). Yellowish crystals, mp = 123−125 °C, R_f = 0.17 (SiO_2, hexane/EtOAc 1/1).

^{1}H NMR (300 MHz, $CDCl_3$, 300K): δ = 7.16 (d, *J* = 2.1 Hz, 1H), 7.11–6.92 (m, 3H), 3.16 (ddd, *J* = 12.4, 5.1, 2.1 Hz, 1H), 2.79–2.63 (m, 2H), 2.57 (s, 3H), 2.15–1.93 (m, 4H), 1.77 (d, *J* = 17.6 Hz, 1H), 1.69 (s, 3H), 1.65 (s, 3H).

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$, 300K): δ = 184.3, 160.9, 141.6, 133.2, 128.8, 128.3, 127.2, 127.1, 125.0, 121.9, 73.4, 46.0, 39.8, 35.0, 26.1, 19.1, 18.6, 15.2.

HRMS (ESI+) m/z: calctd for $C_{18}H_{22}{}^{35}ClN_2O$ [M+H$^+$] 317.1415, found 317.1415.

(5*S,10*R**)-2,3,7,8-Tetramethyl-10-(thiophen-2-yl)-1,3-diazaspiro[4.5]deca-1,7-dien-4-one 3f**

Yield 24 mg (0.08 mmol, 17%). Yellowish oil, R_f = 0.15 (SiO_2, hexane/EtOAc 1/1).

^{1}H NMR (300 MHz, $CDCl_3$, 300K): δ = 7.05 (dd, *J* = 5.1, 1.5 Hz, 1H), 6.85–6.77 (m, 2H), 3.56 (dd, *J* = 12.1, 5.3 Hz, 1H), 2.78–2.58 (m, 5H), 2.21 (dd, *J* = 15.7, 5.4 Hz, 1H), 2.11 (s, 3H), 1.80 (d, *J* = 17.0 Hz, 1H), 1.70 (s, 3H), 1.66 (s, 3H).

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$, 300K): δ = 184.6, 161.8, 142.6, 126.0, 125.4, 125.2, 123.8, 122.2, 73.5, 42.0, 39.7, 37.8, 26.3, 19.0, 18.7, 15.6.

HRMS (ESI+) m/z: calctd for $C_{16}H_{21}N_2OS$ [M+H$^+$] 289.1369, found 289.1370.

(1*S,2*R**,3*R**,4*R**)-3-(4-Bromophenyl)-1',2'-dimethylspiro[bicyclo[2.2.1]heptane-2,4'-imidazol]-5-en-5'(1'*H*)-one *endo*-3g**

Yield 78 mg (0.23 mmol, 45%). White gum, R_f = 0.23 (SiO_2, hexane/EtOAc 2/1).

^{1}H NMR (300 MHz, $CDCl_3$, 300K): δ = 7.27 (d, *J* = 8.5 Hz, 2H), 6.93 (d, *J* = 8.5 Hz, 2H), 6.53 (dd, *J* = 5.7, 2.9 Hz, 1H), 6.42 (dd, *J* = 5.7, 3.1 Hz, 1H), 3.68 (d, *J* = 2.9 Hz, 1H), 3.22 (s, 1H), 3.07 (s, 3H), 2.87–2.82 (m, 1H), 2.42 (d, *J* = 8.8 Hz, 1H), 2.04 (s, 3H), 1.55 (d, *J* = 8.8 Hz, 1H).

^{13}C NMR (75 MHz, $CDCl_3$, 300K): δ = 186.2, 159.7, 138.7, 137.9, 136.4, 131.1 (×2), 130.7 (×2), 120.3, 79.1, 56.4, 54.3, 48.8, 47.0, 26.9, 15.5.

HRMS (ESI+) m/z: calctd for $C_{17}H_{18}{}^{79}BrN_2O$ [M+H$^+$] 345.0597, found 345.0620.

(1*R,2*R**,3*R**,4*S**)-3-(4-bromophenyl)-1',2'-dimethylspiro[bicyclo[2.2.1]heptane-2,4'-imidazol]-5-en-5'(1'*H*)-one *exo*-3g**

Yield 76 mg (0.22 mmol, 44%). White gum, R_f = 0.15 (SiO_2, hexane/EtOAc 2/1).

^{1}H NMR (300 MHz, $CDCl_3$, 300K): δ = 7.32 (d, *J* = 8.4 Hz, 2H), 6.90 (d, *J* = 8.4 Hz, 2H), 6.62 (dd, *J* = 5.6, 3.2 Hz, 1H), 6.30 (dd, *J* = 5.6, 3.0 Hz, 1H), 3.19 (s, 1H), 3.03 (s, 3H), 2.97 (d, *J* = 2.1 Hz, 1H), 2.68 (s, 1H), 2.59 (d, *J* = 8.7 Hz, 1H), 1.97 (s, 3H), 1.78 (dd, *J* = 8.7, 1.9 Hz, 1H).

^{13}C NMR (75 MHz, $CDCl_3$, 300K): δ = 184.6, 158.8, 141.3, 139.7, 133.5, 131.1 (×2), 129.6 (×2), 120.0, 77.0, 56.6, 54.0, 48.9, 48.6, 26.9, 15.4.

HRMS (ESI+) m/z: calctd for $C_{17}H_{18}{}^{79}BrN_2O$ [M+H$^+$] 345.0597, found 345.0595.

Typical procedure for thermal Diels–Alder reaction:

A solution of arylideneimidazol-4-one **1** (0.50 mmol, 1 equiv.) and diene **2** (92 mg, 0.75 mmol, 1.5 equiv. for **2c**; 130 mg, 0.75 mmol, 1.5 equiv. for **2d**; 410 mg, 5.0 mmol, 10.0 equiv. for **2a**) in toluene (1 mL) was heated to reflux for 8-24 h. The reaction progress was monitored by TLC. Then the volatile substances were removed on a rotary evaporator. Column chromatography of the residue on silica gel (eluent hexane/EtOAc 10/1→3/1→1/1) provided pure cycloaddition derivative **3** (65-97%).

(5S*,6*S,10R*)-10-(4-Bromophenyl)-2,3-dimethyl-6-phenyl-1,3-diazaspiro[4.5]deca-1,7-dien-4-one *endo*-3h**

Yield 65 mg (0.16 mmol, 32%). White crystals, mp = 133−135 °C, R_f = 0.34 (SiO_2, hexane/EtOAc 1/1).

^{1}H NMR (800 MHz, $CDCl_3$, 298K): δ = 7.29 (d, *J* = 8.5 Hz, 2H), 7.18–7.04 (m, 7H), 6.09 (ddt, *J* = 10.0, 4.9, 2.4 Hz, 1H), 5.83 (dq, *J* = 10.0, 2.0 Hz, 1H), 4.11 (hept, *J* = 2.2 Hz, 1H), 3.50 (dd, *J* = 12.0, 5.4 Hz, 1H), 2.89–2.71 (m, 1H), 2.47–2.29 (m, 1H), 2.30 (s, 3H), 1.68 (s, 3H).

^{13}C{^{1}H} NMR (75 MHz, $CDCl_3$, 300K): δ = 183.5, 161.0, 139.0, 138.6, 130.9 (×2), 130.4 (×2), 129.3 (×2), 127.8, 127.5, 127.4 (×2), 127.0, 121.1, 76.2, 49.8, 45.4, 29.4, 25.8, 14.6.

HRMS (ESI+) m/z: calctd for $C_{22}H_{22}{}^{79}BrN_2O$ [M+H$^+$] 409.0910, found 409.0903.

(5*S,6*S**,10*R**)-10-(4-Bromophenyl)-2,3-dimethyl-6-phenyl-1,3-diazaspiro[4.5]deca-1,7-dien-4-one *exo*-3h**

Yield 133 mg (0.32 mmol, 65%). Yellowish crystals, mp = 191−193 °C, Rf = 0.18 (SiO_2, hexane/EtOAc 1/1).

^{1}H NMR (300 MHz, $CDCl_3$, 300K): δ = 7.36–7.21 (m, 5H), 7.19–7.10 (m, 2H), 7.06 (d, *J* = 8.5 Hz, 2H), 6.31–6.09 (m, 1H), 5.92–5.79 (m, 1H), 3.39 (dd, *J* = 4.1, 2.2 Hz, 1H), 3.21 (dd, *J* = 8.2, 6.3 Hz, 1H), 2.81–2.58 (m, 2H), 2.53 (s, 3H), 2.06 (s, 3H).

^{13}C{^{1}H} NMR (75 MHz, CDCl3, 300K): δ = 181.0, 160.6, 139.4, 138.0, 130.9 (×2), 130.7 (×2), 130.2 (×2), 127.7 (×2), 127.3, 127.2, 126.7, 120.9, 75.1, 48.1, 41.2, 29.2, 26.0, 15.4.

HRMS (ESI+) m/z: calctd for $C_{22}H_{22}{}^{79}BrN_2O$ [M+H$^+$] 409.0910, found 409.0903.

(5*S,6*R**,10*R**)-10-(4-Methoxyphenyl)-2,3-dimethyl-6-phenyl-1,3-diazaspiro[4.5]deca-1,7-dien-4-one *endo*-3i**

Yield 58 mg (0.16 mmol, 32%). Yellowish gum, R_f = 0.16 (SiO_2, hexane/EtOAc 1/1).

^{1}H NMR (800 MHz, $CDCl_3$, 298K): δ = 7.21–7.00 (m, 7H), 6.72 (d, *J* = 8.6 Hz, 2H), 6.11 (ddt, *J* = 10.1, 5.3, 2.6 Hz, 1H), 5.84 (dq, *J* = 10.1, 2.1 Hz, 1H), 4.14 (p, *J* = 2.3 Hz, 1H), 3.74 (s, 3H), 3.49 (dd, *J* = 12.0, 5.4 Hz, 1H), 2.83 (dddd, *J* = 18.6, 9.4, 4.6, 2.4 Hz, 1H), 2.38 (dtt, *J* = 18.6, 5.2, 2.0 Hz, 1H), 2.29 (s, 3H), 1.69 (s, 3H).

^{13}C{^{1}H} NMR (201 MHz, $CDCl_3$, 298K): δ = 183.9, 160.7, 158.7, 139.4, 131.7, 129.8 (×2), 129.4 (×2), 127.9, 127.7, 127.4, 126.9 (×2), 113.1 (×2), 76.7, 55.3, 49.7, 45.4, 29.7, 25.8, 14.7.

HRMS (ESI+) m/z: calctd for $C_{23}H_{25}N_2O_2$ [M+H$^+$] 361.1911, found 361.1916.

(5*S,6*S**,10*R**)-10-(4-Methoxyphenyl)-2,3-dimethyl-6-phenyl-1,3-diazaspiro[4.5]deca-1,7-dien-4-one *endo*-3i**

Yield 104 mg (0.29 mmol, 58%). Yellowish crystals, mp =129−131 °C, R_f = 0.10 (SiO_2, hexane/EtOAc 1/1).

^{1}H NMR (300 MHz, $CDCl_3$, 300K): δ = 7.39–7.20 (m, 3H), 7.20–7.13 (m, 2H), 7.09 (d, *J* = 8.8 Hz, 2H), 6.71 (d, *J* = 8.8 Hz, 2H), 6.21 (dtd, *J* = 10.0, 3.6, 1.8 Hz, 1H), 5.85 (ddt, *J* = 10.0, 4.2, 2.1 Hz, 1H), 3.74 (s, 3H), 3.41 (dq, *J* = 4.0, 1.9 Hz, 1H), 3.22 (t, *J* = 7.5 Hz, 1H), 2.76–2.58 (m, 2H), 2.50 (s, 3H), 2.06 (s, 3H).

^{13}C{^{1}H} NMR (75 MHz, $CDCl_3$, 300K): δ = 181.3, 160.4, 158.6, 138.4, 132.3, 130.3 (×2), 130.2 (×2), 127.7 (×3), 127.2, 126.7, 113.0 (×2), 75.6, 55.3, 48.4, 41.0, 29.7, 26.0, 15.4.

HRMS (ESI+) m/z: calctd for $C_{23}H_{25}N_2O_2$ [M+H$^+$] 361.1911, found 361.1907.

(5*S,6*S**,10*R**)-10-(4-Bromophenyl)-8-((*tert*-butyldimethylsilyl)oxy)-2,3-dimethyl-6-phenyl-1,3-diazaspiro[4.5]deca-1,7-dien-4-one *endo*-3j**

Yield 77 mg (0.14 mmol, 29%). Yellowish gum, R_f = 0.60 (SiO_2, hexane/EtOAc 1/1).

^{1}H NMR (300 MHz, $CDCl_3$, 300K): δ = 7.30 (d, *J* = 8.5 Hz, 2H), 7.20–6.83 (m, 7H), 4.92 (t, *J* = 1.9 Hz, 1H), 4.18 (dt, *J* = 3.8, 1.7 Hz, 1H), 3.57 (dd, *J* = 12.3, 5.4 Hz, 1H), 2.93 (dddd, *J* = 17.4, 12.3, 3.7, 2.8 Hz, 1H), 2.31 (s, 3H), 2.24 (dd, J = 17.4, 5.4 Hz, 1H), 1.67 (s, 3H), 0.93 (s, 9H), 0.22 (s, 3H), 0.19 (s, 3H).

^{13}C{^{1}H} NMR (75 MHz, $CDCl_3$, 300K): δ = 183.7, 160.7, 150.5, 139.9 (×2), 138.1 (×2), 130.9 (×2), 130.5 (×2), 129.4 (×2), 127.3, 127.0, 121.2, 104.2, 76.2, 49.6, 45.9, 33.7, 25.9, 25.8 (×3), 18.1, 14.6, −4.1, −4.3.

HRMS (ESI+) m/z: calctd for $C_{28}H_{36}{}^{79}BrN_2O2Si$ [M+H$^+$] 539.1724, found 539.1724.

(5*S,6*R**,10*R**)-10-(4-Bromophenyl)-8-((*tert*-butyldimethylsilyl)oxy)-2,3-dimethyl-6-phenyl-1,3-diazaspiro[4.5]deca-1,7-dien-4-one *exo*-3j**

Yield 178 mg (0.33 mmol, 66%). Yellowish gum, R_f = 0.40 (SiO_2, hexane/EtOAc 1/1).

^{1}H NMR (300 MHz, $CDCl_3$, 300K): δ = 7.38 (d, *J* = 8.4 Hz, 2H), 7.34–7.20 (m, 3H), 7.21–7.12 (m, 4H), 5.03 (dd, *J* = 3.8, 1.6 Hz, 1H), 3.57 (dt, *J* = 3.8, 1.8 Hz, 1H), 3.23 (t, *J* = 6.6 Hz, 1H), 2.85 (ddt, *J* = 17.6, 6.6, 1.8 Hz, 1H), 2.59 (ddt, *J* = 17.6, 6.8, 1.6 Hz, 1H), 2.56 (s, 3H), 2.03 (s, 3H), 1.00 (s, 9H), 0.31 (s, 3H), 0.29 (s, 3H).

^{13}C{^{1}H} NMR (75 MHz, $CDCl_3$, 300K): δ = 181.4, 160.4, 151.1, 139.6, 139.3, 131.0 (×4), 129.9 (×2), 127.6 (×2), 127.2, 121.1, 104.0, 74.8, 47.0, 42.7, 33.3, 26.0, 25.8 (×3), 18.1, 15.4, −3.8, −4.2.

HRMS (ESI+) m/z: calctd for $C_{28}H_{36}{}^{79}BrN_2O_2Si$ [M+H$^+$] 539.1724, found 539.1721.

(5*S,10*S**)-10-(4-Bromophenyl)-3,7,8-trimethyl-2-(trifluoromethyl)-1,3-diazaspiro[4.5]deca-1,7-dien-4-one 3′a**

Yield 151 mg (0.36 mmol, 73%). Yellowish oil, R_f = 0.46 (SiO_2, hexane/EtOAc 5/1).

1H NMR (300 MHz, $CDCl_3$, 300K): δ = 7.33 (d, *J* = 8.4 Hz, 2H), 6.98 (d, *J* = 8.4 Hz, 2H), 3.29 (dd, *J* = 12.3, 5.1 Hz, 1H), 2.85–2.67 (m, 5H), 2.09 (dd, *J* = 16.6, 4.3 Hz, 1H), 1.88 (d, *J* = 16.9 Hz, 1H), 1.73 (s, 3H), 1.69 (s, 3H).

^{13}C{^{1}H} NMR (75 MHz, $CDCl_3$, 300K): δ = 183.4, 152.3 (q, *J* = 38.6 Hz), 137.2, 131.1 (×2), 130.2 (×2), 125.5, 121.6, 121.1, 117.0 (q, *J* = 274.8 Hz), 75.1, 46.1, 39.5, 34.6, 26.8 (q, *J* = 2.2 Hz), 19.2, 18.5.

HRMS (ESI+) m/z: calctd for $C_{18}H_{19}{}^{79}BrF_3N_2O$ [M+H$^+$] 415.0627, found 415.0631.

(5*S,10*S**)-3,7,8-Trimethyl-10-phenyl-2-(trifluoromethyl)-1,3-diazaspiro[4.5]deca-1,7-dien-4-one 3′b**

Yield 109 mg (0.33 mmol, 65%). Yellowish oil, R_f = 0.43 (SiO_2, hexane/EtOAc, 5/1).

^{1}H NMR (800 MHz, $CDCl_3$, 298K): δ = 7.23–7.16 (m, 3H), 7.10 (d, *J* = 7.9 Hz, 2H), 3.32 (dd, *J* = 12.3, 5.2 Hz, 1H), 2.87–2.78 (m, 2H), 2.68 (q, *J* = 1.2 Hz, 3H), 2.13 (dd, *J* = 16.5, 4.9 Hz, 1H), 1.90 (d, *J* = 17.3 Hz, 1H), 1.75 (s, 3H), 1.70 (s, 3H).

^{13}C{^{1}H} NMR (201 MHz, $CDCl_3$, 298K): δ = 183.5, 151.9 (q, *J* = 38.2 Hz), 137.9, 128.4 (×2), 127.8 (×2), 127.5, 125.6, 120.9, 116.9 (q, *J* = 274.6 Hz), 75.3, 46.8, 39.1, 34.4, 26.5 (q, *J* = 2.1 Hz), 19.1, 18.1.

HRMS (ESI+) m/z: calctd for $C_{18}H_{20}F_3N_2O$ [M+H+] 337.1522, found 337.1527.

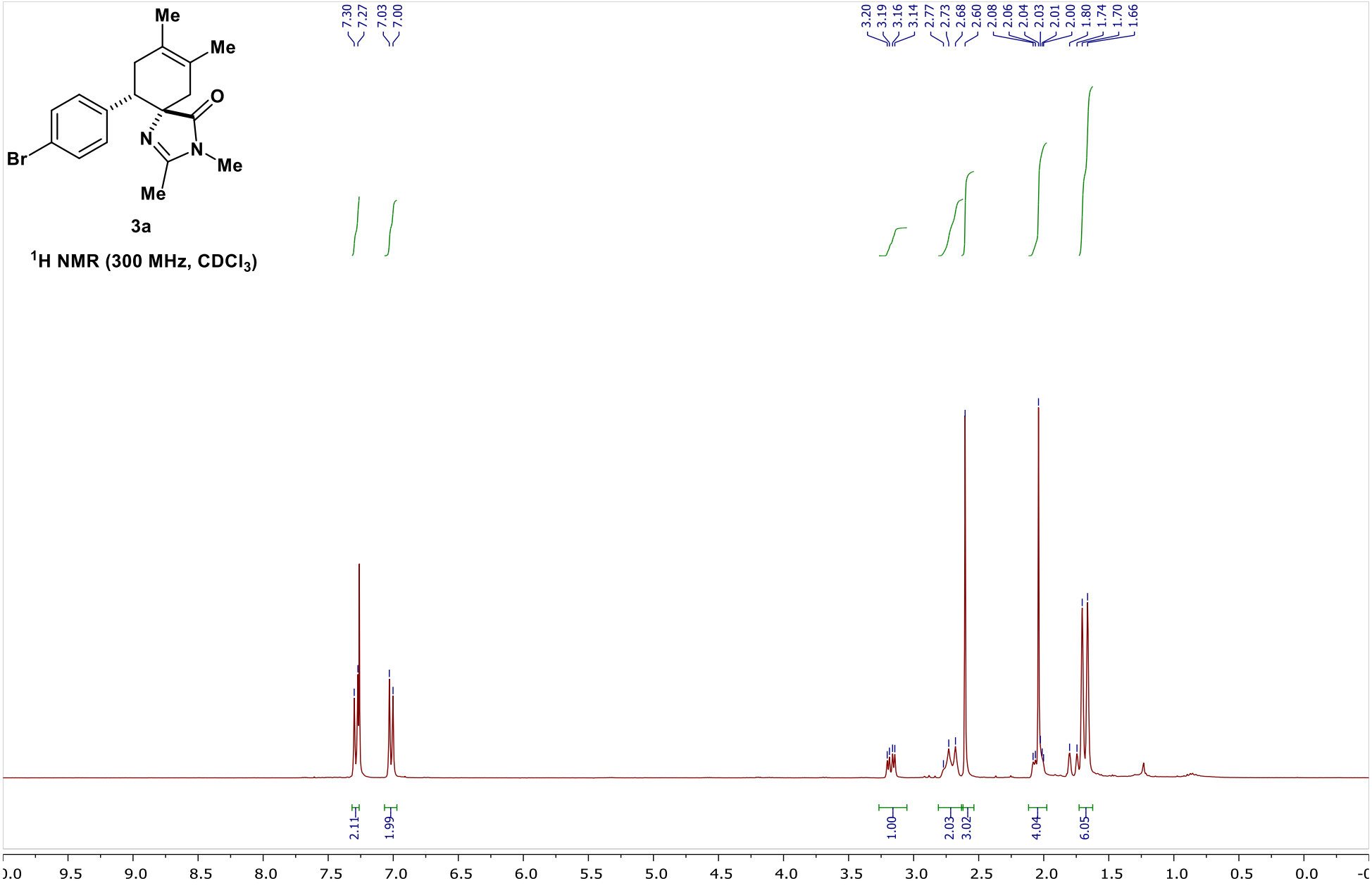
(5*S,10*S**)-10-(4-Methoxyphenyl)-3,7,8-trimethyl-2-(trifluoromethyl)-1,3-diazaspiro[4.5]deca-1,7-dien-4-one 3′c**

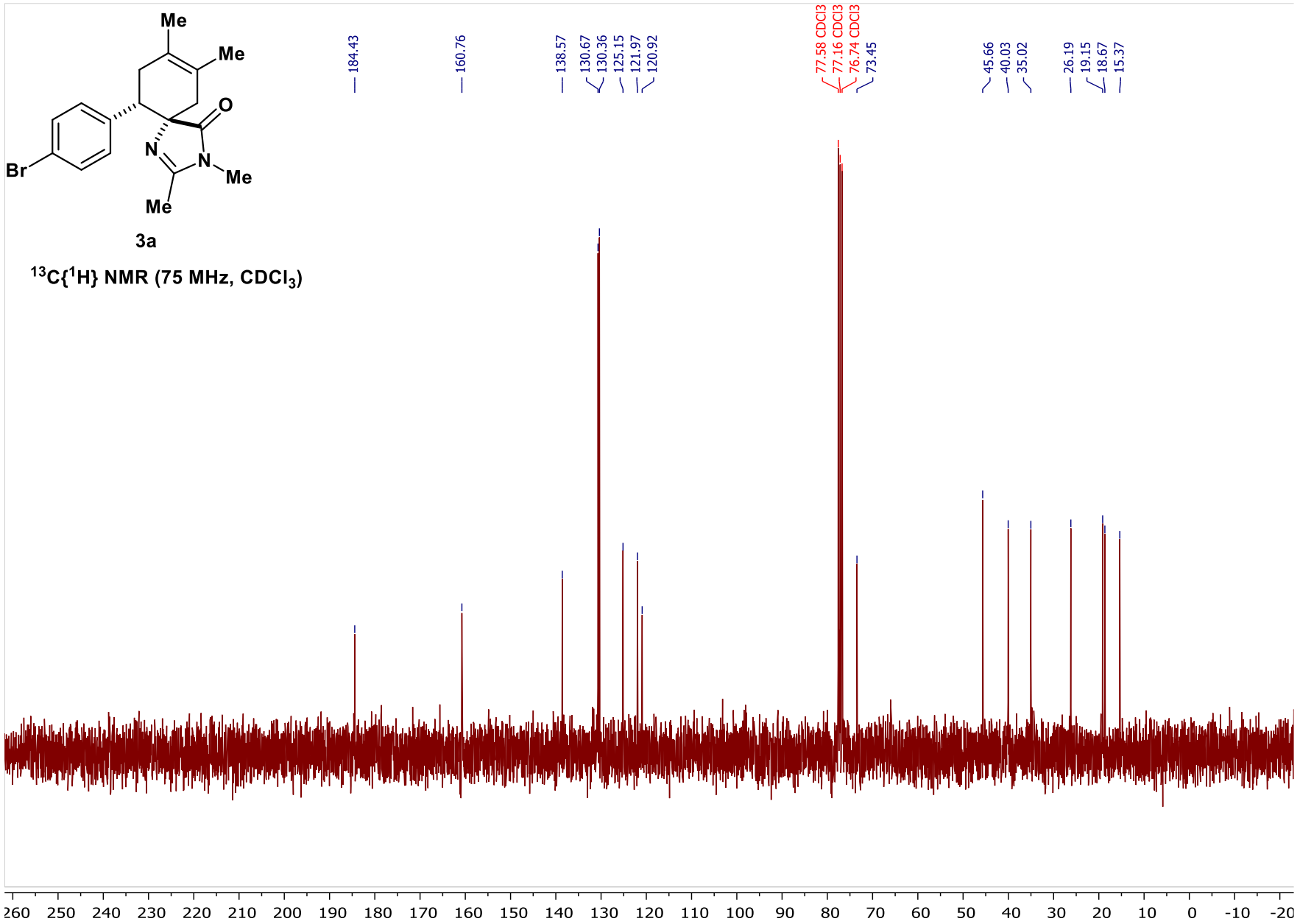
Yield 183 mg (0.35 mmol, 71%). Yellowish oil, R_f =0.37 (SiO_2, hexane/EtOAc 5/1).

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.02 (d, *J* = 8.7 Hz, 2H), 6.74 (d, *J* = 8.7 Hz, 2H), 3.75 (s, 3H), 3.27 (dd, *J* = 12.3, 5.1 Hz, 1H), 2.91–2.67 (m, 5H), 2.09 (dd, *J* = 15.6, 5.0 Hz, 1H), 1.88 (d, *J* = 16.9 Hz, 1H), 1.73 (s, 3H), 1.69 (s, 3H).

^{13}C{^{1}H} NMR (75 MHz, $CDCl_3$, 300K): δ = 183.8, 159.0, 152.0 (q, *J* = 38.5 Hz), 130.1, 129.5 (×2), 125.9, 121.0, 117.1 (q, *J* = 274.8 Hz), 113.3 (×2), 75.6, 55.3, 46.0, 39.3, 34.9, 26.7 (q, *J* = 2.1 Hz), 19.2, 18.5.

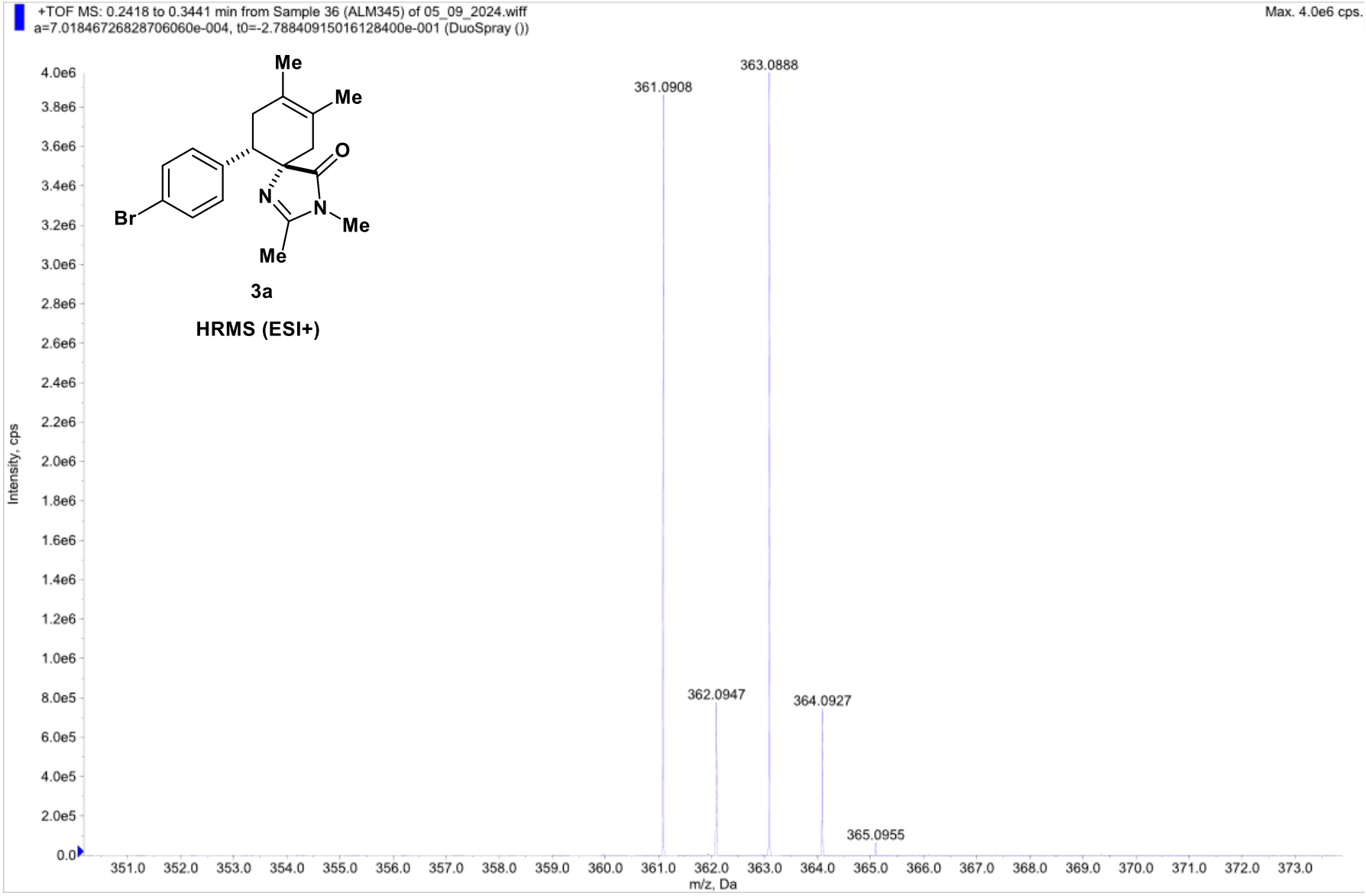
HRMS (ESI+) m/z: calctd for $C_{19}H_{22}F_3N_2O_2$ [M+H$^+$] 367.1628, found 367.1631.

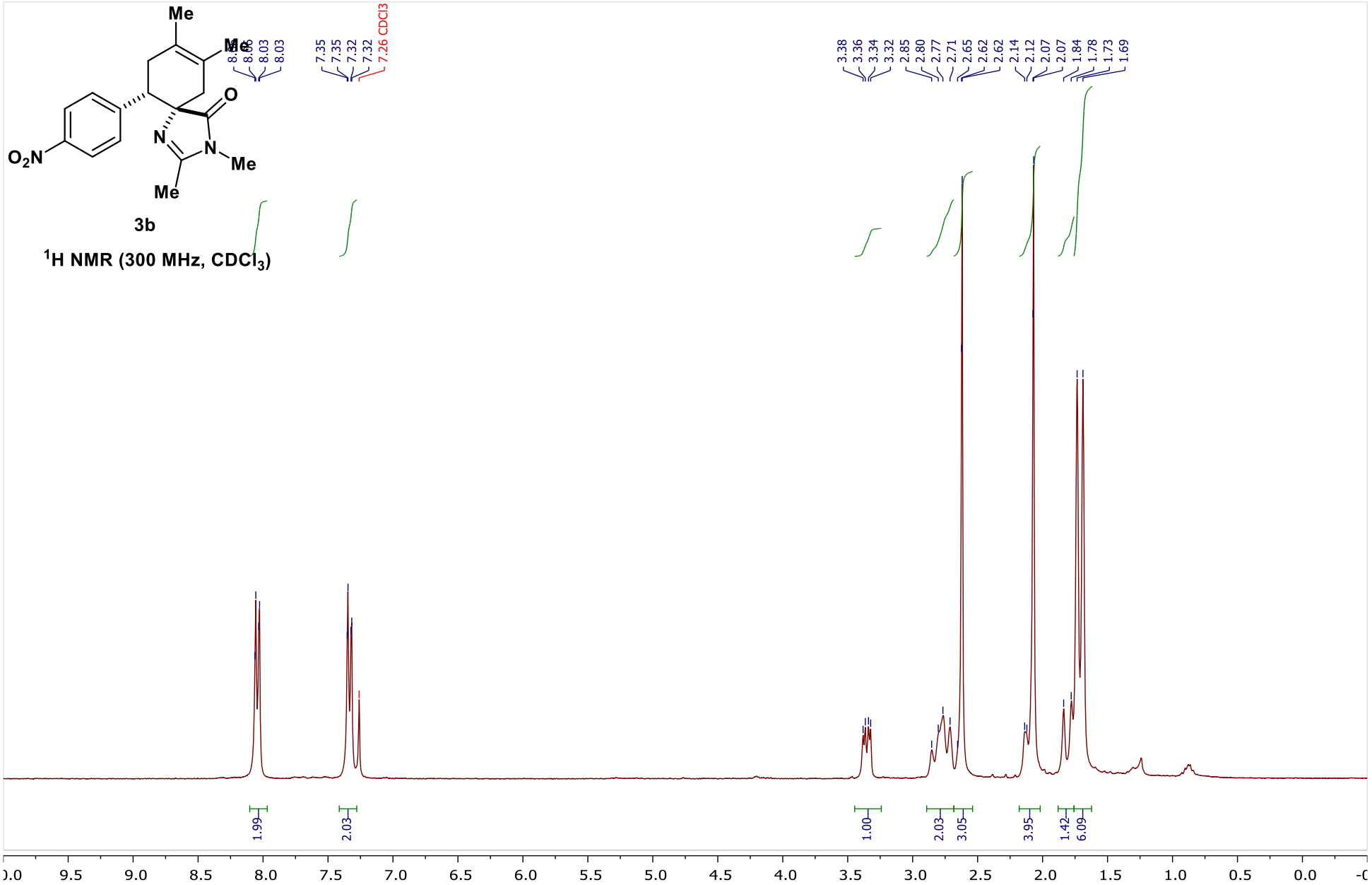

Me
Me
O
N
N
Me
Me
Br
3a
^{1}H NMR (300 MHz, CDCl$_3$)
7.30
7.27
7.03
7.00
3.20
3.19
3.16
3.14
2.77
2.73
2.68
2.60
2.08
2.06
2.04
2.03
2.01
2.00
1.80
1.74
1.70
1.66
2.11
1.99
1.00
2.03
3.02
4.04
6.05
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

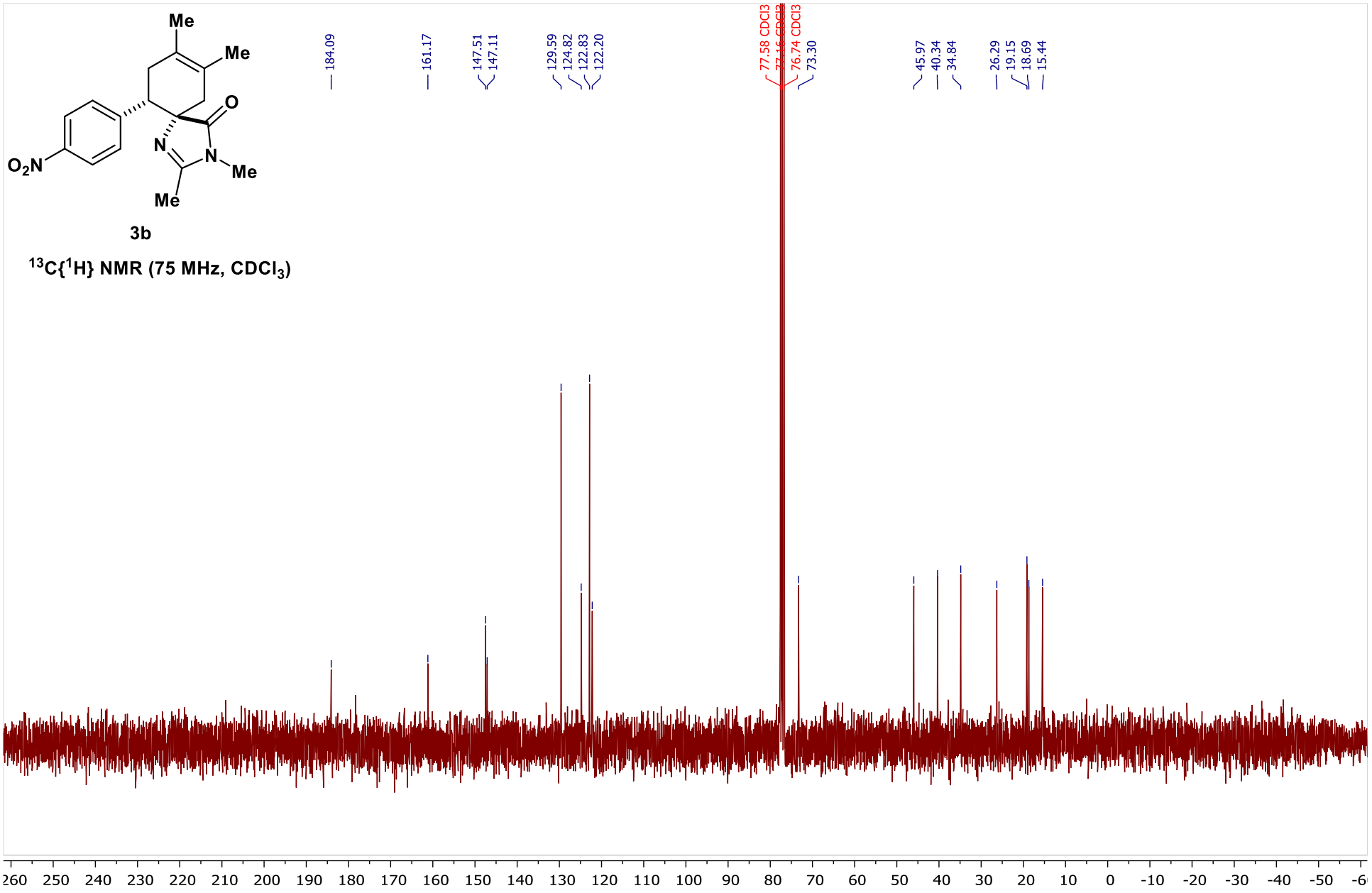

Me
Me
O
N
N
Me
Me
Br
3a
$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$)
184.43
160.76
138.57
130.67
130.36
125.15
121.97
120.92
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
73.45
45.66
40.03
35.02
26.19
19.15
18.67
15.37
260 250 240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20

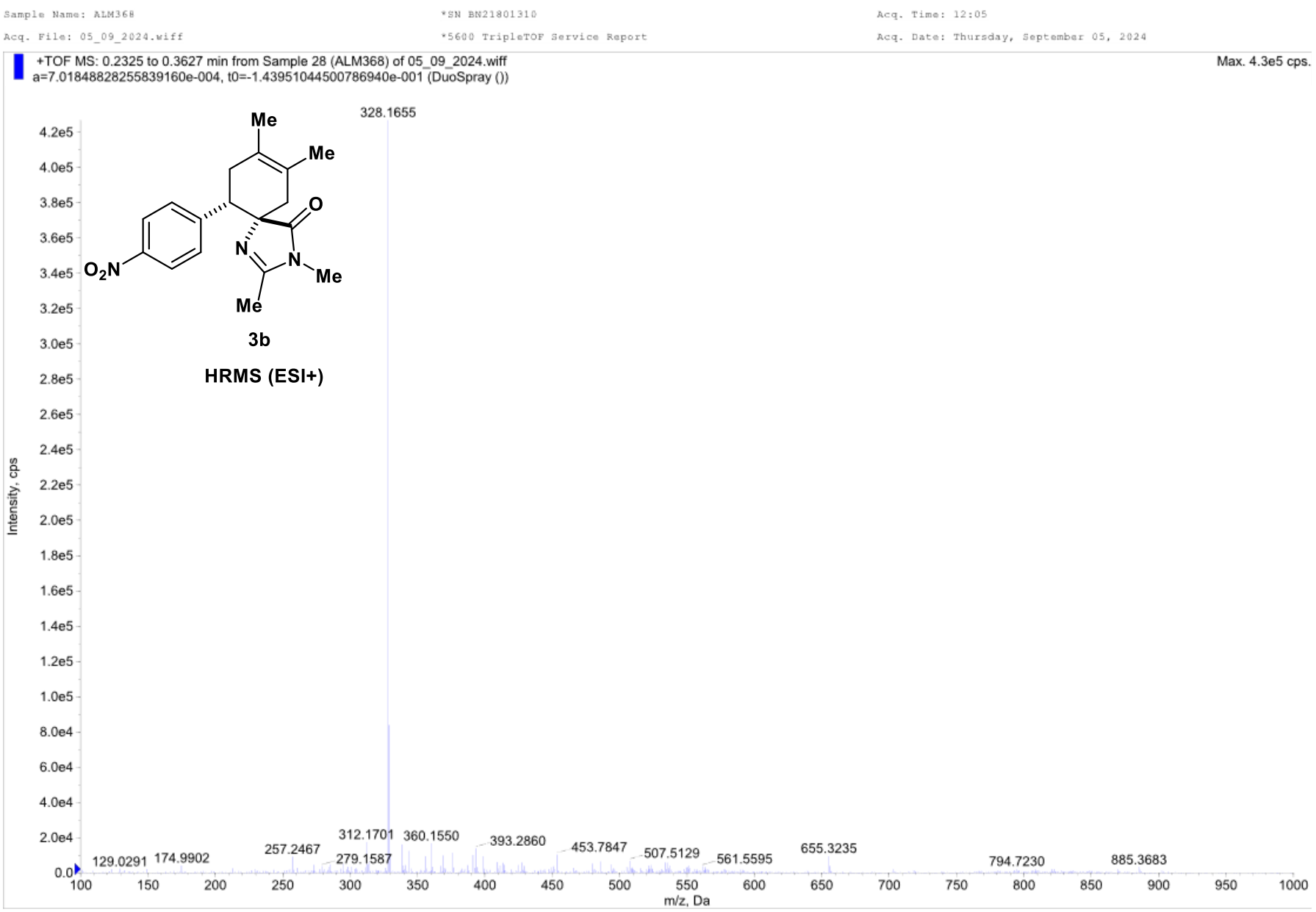
Sample Name: ALM345 *SN BN21801310 Acq. Time: 12:20

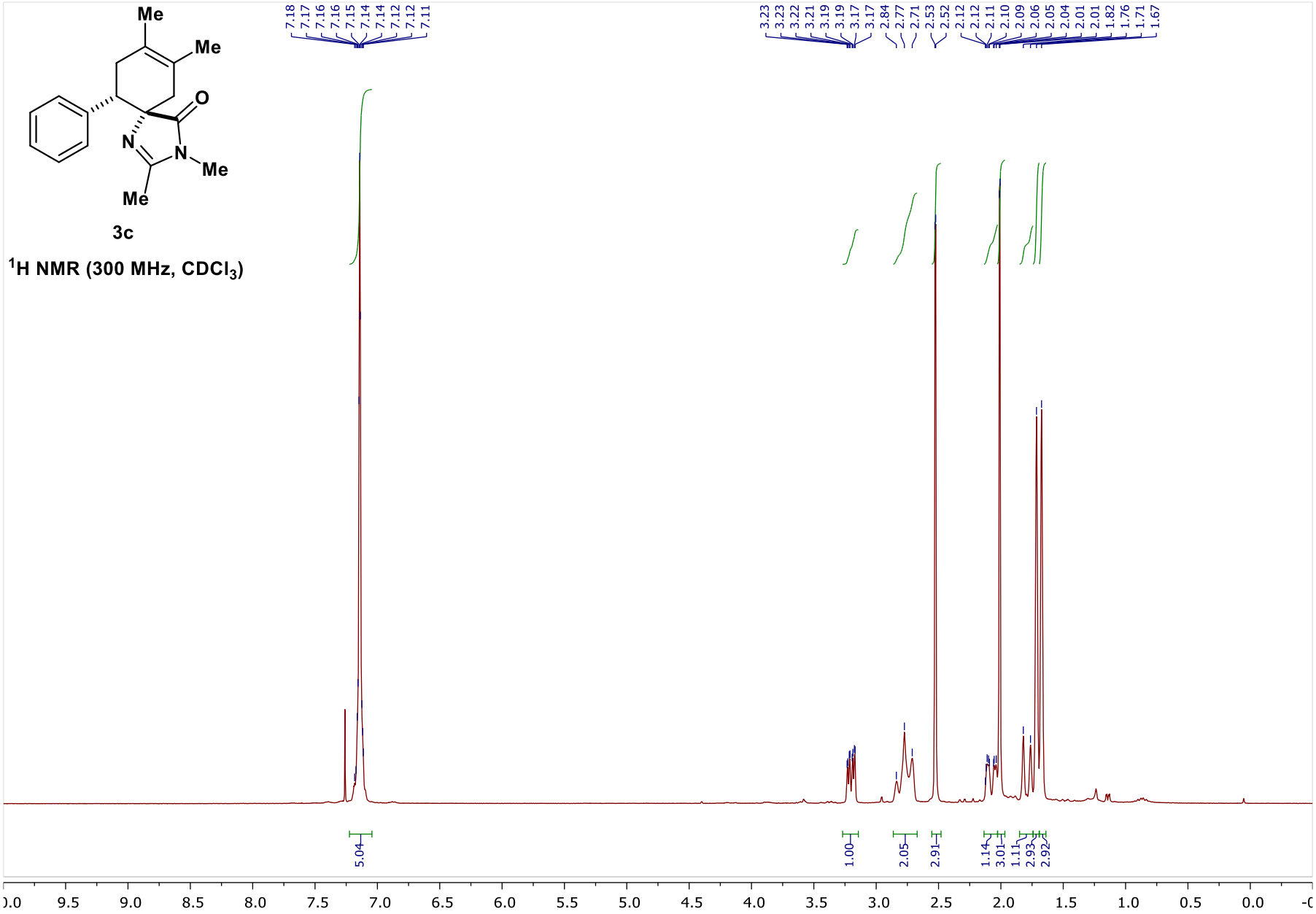
Acq. File: 05_09_2024.wiff *5600 TripleTOF Service Report Acq. Date: Thursday, September 05, 2024

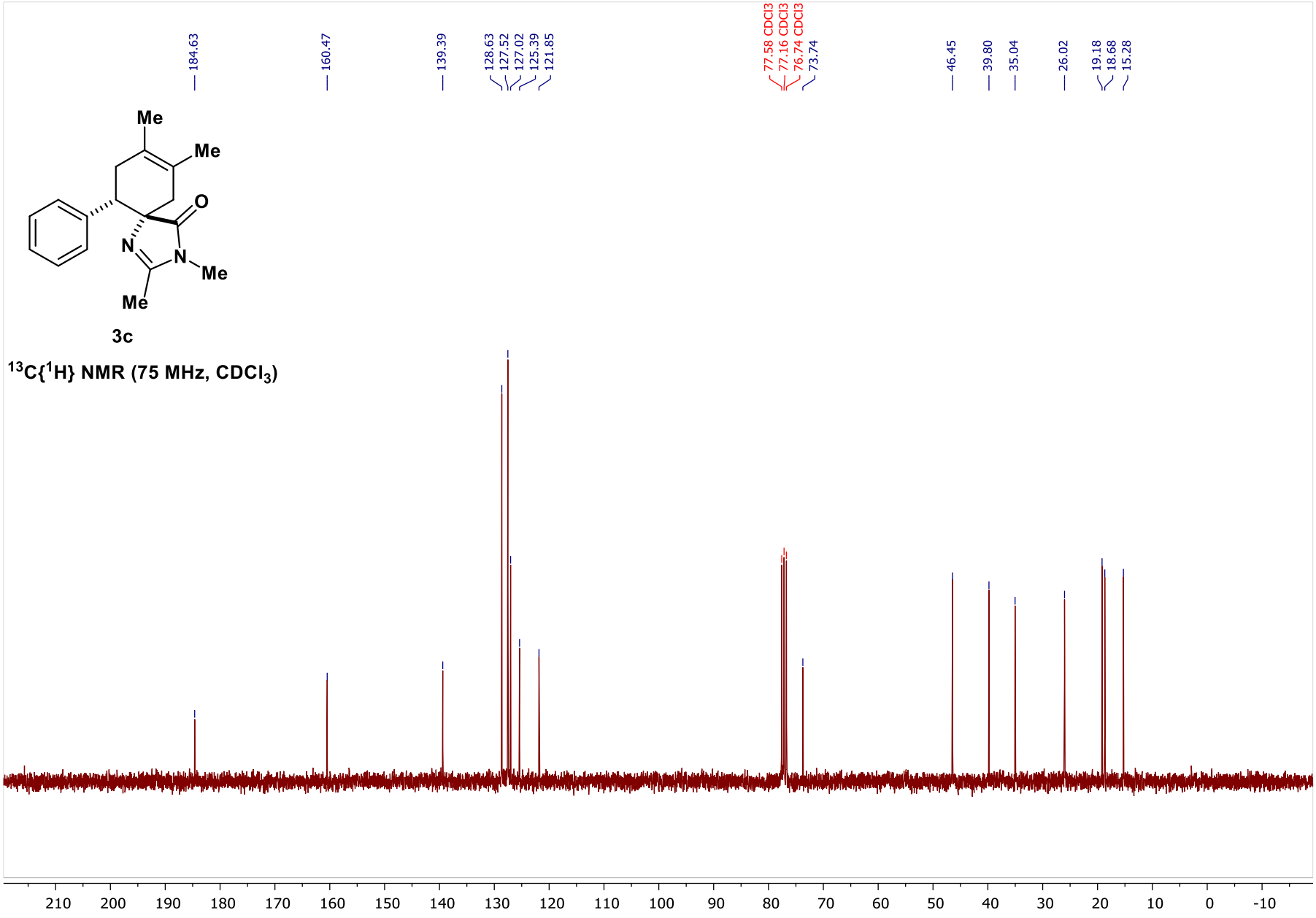


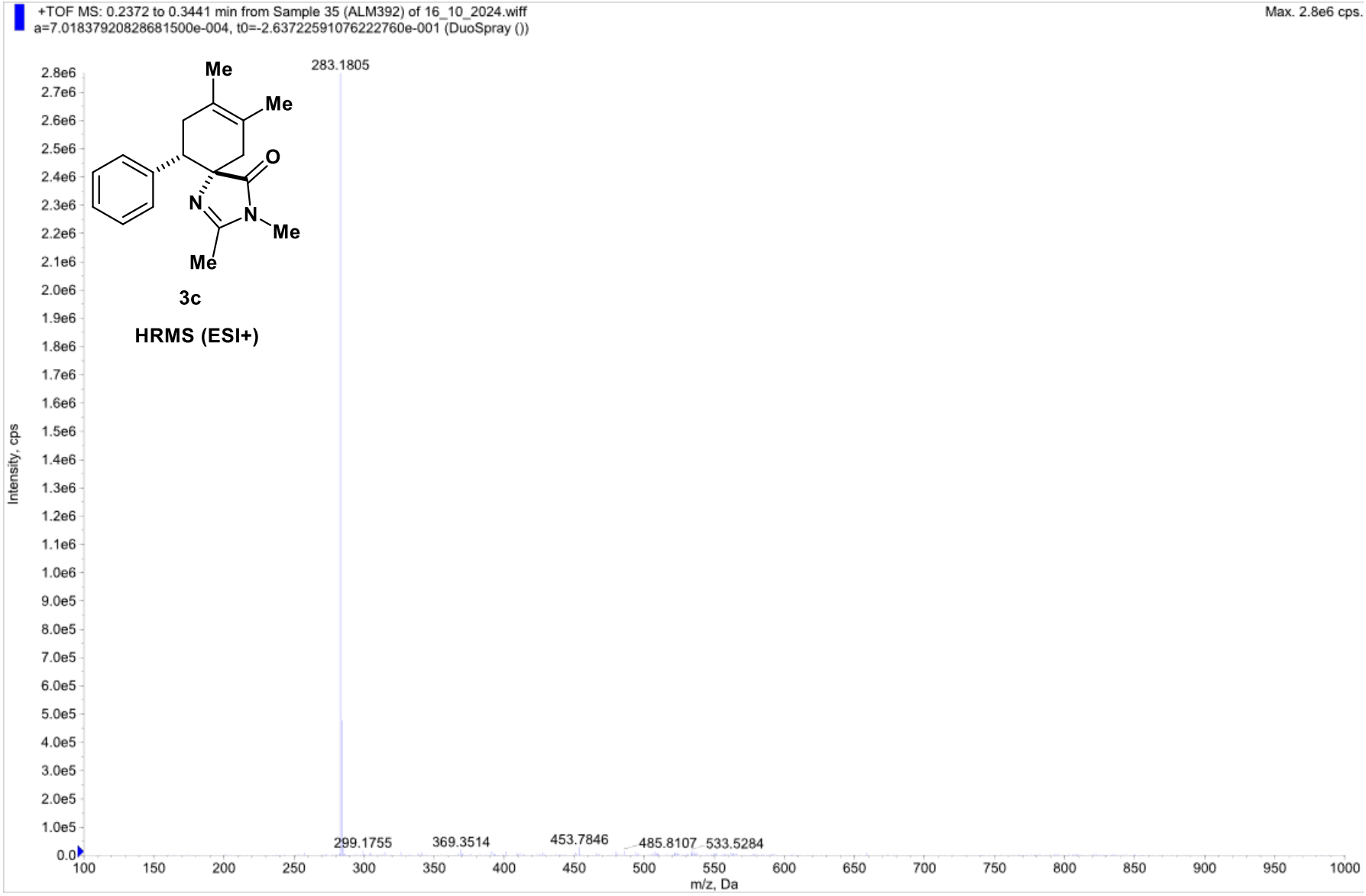

Me
Me
O
N
N
Me
Me
O2N
3b
1H NMR (300 MHz, CDCl3)
8.06
8.03
8.03
7.35
7.35
7.32
7.32
7.26 CDCl3
3.38
3.36
3.34
3.32
2.85
2.80
2.77
2.71
2.65
2.62
2.62
2.14
2.12
2.07
2.07
1.84
1.78
1.73
1.69
1.99
2.03
1.00
2.03
3.05
3.95
1.42
6.09
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0

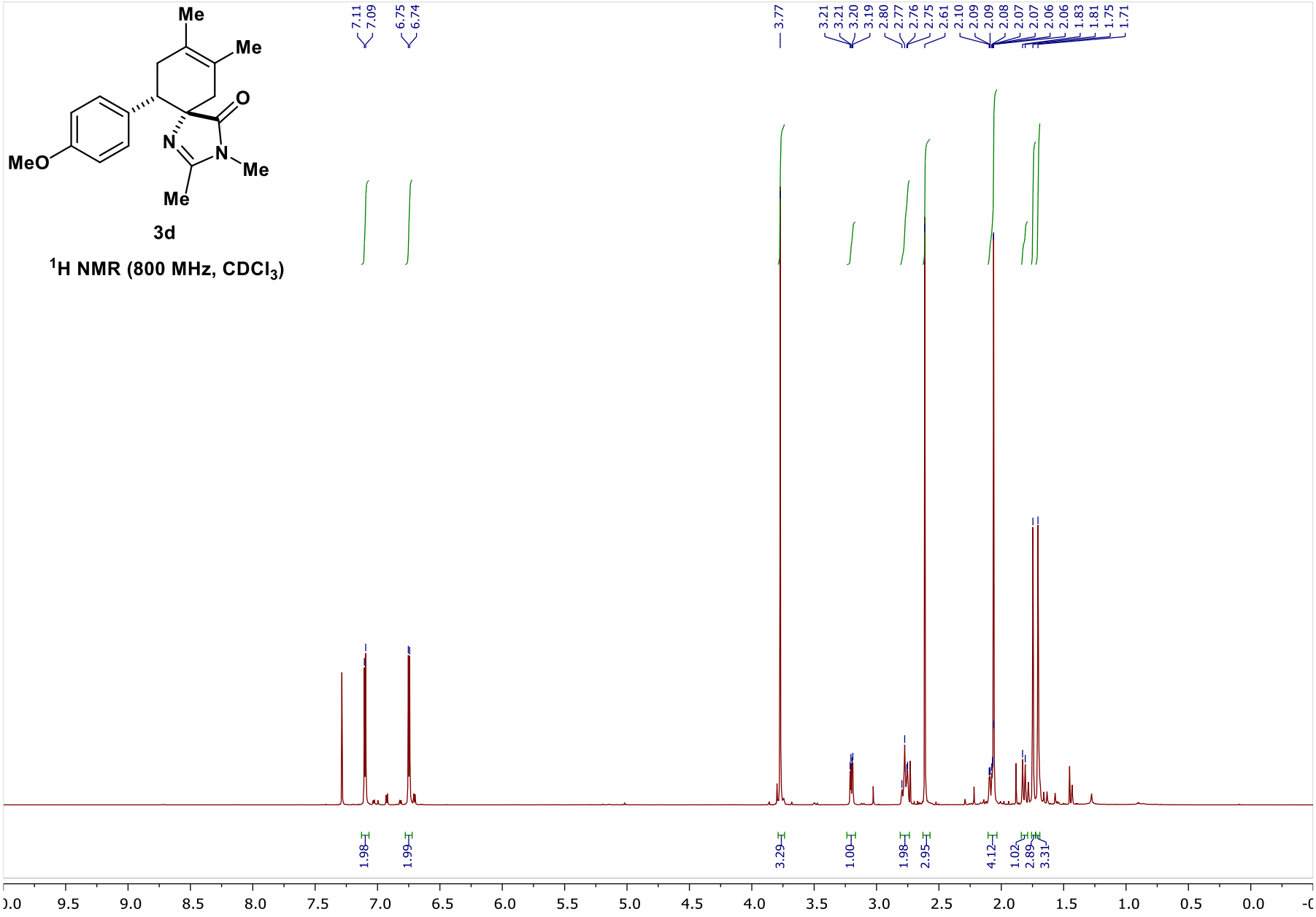

Me
Me
O
N
N
Me
Me
O2N
3b
13C{1H} NMR (75 MHz, CDCl3)
184.09
161.17
147.51
147.11
129.59
124.82
122.83
122.20
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
73.30
45.97
40.34
34.84
26.29
19.15
18.69
15.44
260 250 240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -6

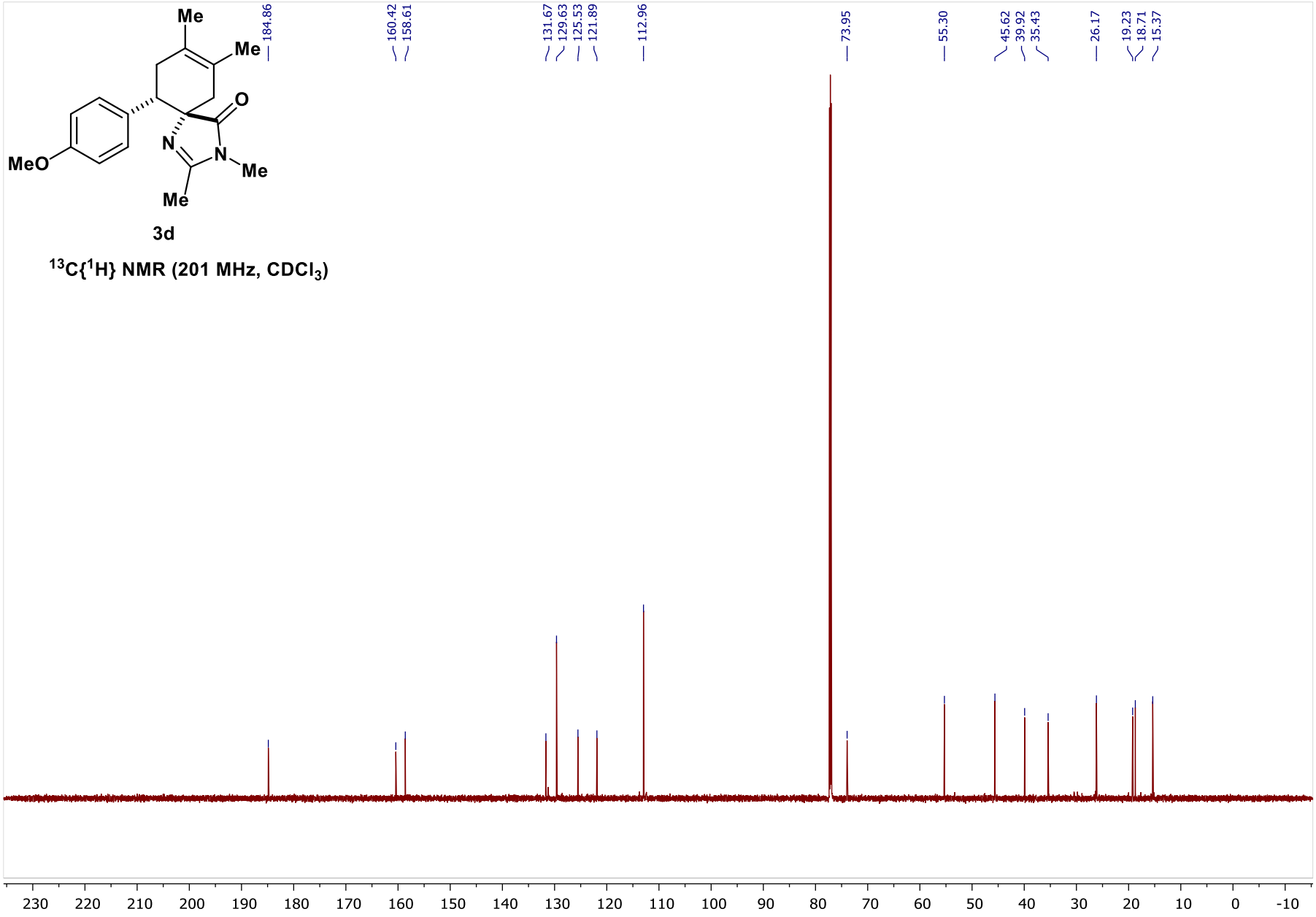

Sample Name: ALM368
*SN BN21801310
Acq. Time: 12:05
Acq. File: 05_09_2024.wiff
*5600 TripleTOF Service Report
Acq. Date: Thursday, September 05, 2024
+TOF MS: 0.2325 to 0.3627 min from Sample 28 (ALM368) of 05_09_2024.wiff
a=7.01848828255839160e-004, t0=-1.43951044500786940e-001 (DuoSpray ())
Max. 4.3e5 cps.
328.1655
Me
Me
O
N
N
Me
Me
O_2N
3b
HRMS (ESI+)
Intensity, cps
4.2e5
4.0e5
3.8e5
3.6e5
3.4e5
3.2e5
3.0e5
2.8e5
2.6e5
2.4e5
2.2e5
2.0e5
1.8e5
1.6e5
1.4e5
1.2e5
1.0e5
8.0e4
6.0e4
4.0e4
2.0e4
0.0
129.0291
174.9902
257.2467
279.1587
312.1701
360.1550
393.2860
453.7847
507.5129
561.5595
655.3235
794.7230
885.3683
100
150
200
250
300
350
400
450
500
550
600
650
700
750
800
850
900
950
1000
m/z, Da


Me
Me
O
N
N
Me
Me
3c
1H NMR (300 MHz, CDCl3)
7.18 7.17 7.16 7.16 7.15 7.14 7.14 7.12 7.12 7.11
3.23 3.23 3.22 3.21 3.19 3.19 3.17 3.17 2.84 2.77 2.71 2.53 2.52 2.12 2.12 2.11 2.10 2.09 2.06 2.05 2.04 2.01 2.01 1.82 1.76 1.71 1.67
5.04
1.00
2.05
2.91
1.14
3.01
1.11
2.93
2.92
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


184.63
160.47
139.39
128.63
127.52
127.02
125.39
121.85
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
73.74
46.45
39.80
35.04
26.02
19.18
18.68
15.28
Me
Me
O
N
N
Me
Me
3c
13C{1H} NMR (75 MHz, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10


Sample Name: ALM392
*SN BN21801310
Acq. Time: 11:51
Acq. File: 16_10_2024.wiff
*5600 TripleTOF Service Report
Acq. Date: Wednesday, October 16, 2024
+TOF MS: 0.2372 to 0.3441 min from Sample 35 (ALM392) of 16_10_2024.wiff
a=7.01837920828681500e-004, t0=-2.63722591076222760e-001 (DuoSpray ())
Max. 2.8e6 cps.
Me
Me
O
N
N
Me
Me
3c
HRMS (ESI+)
283.1805
299.1755
369.3514
453.7846
485.8107
533.5284
Intensity, cps
m/z, Da


Me
Me
O
MeO
N
N
Me
Me
3d
^{1}H NMR (800 MHz, $CDCl_3$)
7.11
7.09
6.75
6.74
3.77
3.21
3.21
3.20
3.19
2.80
2.77
2.76
2.75
2.61
2.10
2.09
2.09
2.08
2.07
2.07
2.06
2.06
1.83
1.81
1.75
1.71
1.98
1.99
3.29
1.00
1.98
2.95
4.12
1.02
2.89
3.31
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



3d

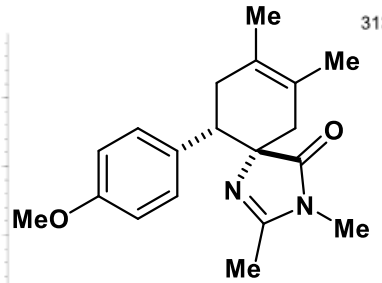
$^{13}C\{^{1}H\}$ NMR (201 MHz, $CDCl_3$)

Sample Name: ALM376 *SN BN21801310 Acq. Time: 11:58

Acq. File: 16_10_2024.wiff *5600 TripleTOF Service Report Acq. Date: Wednesday, October 16, 2024

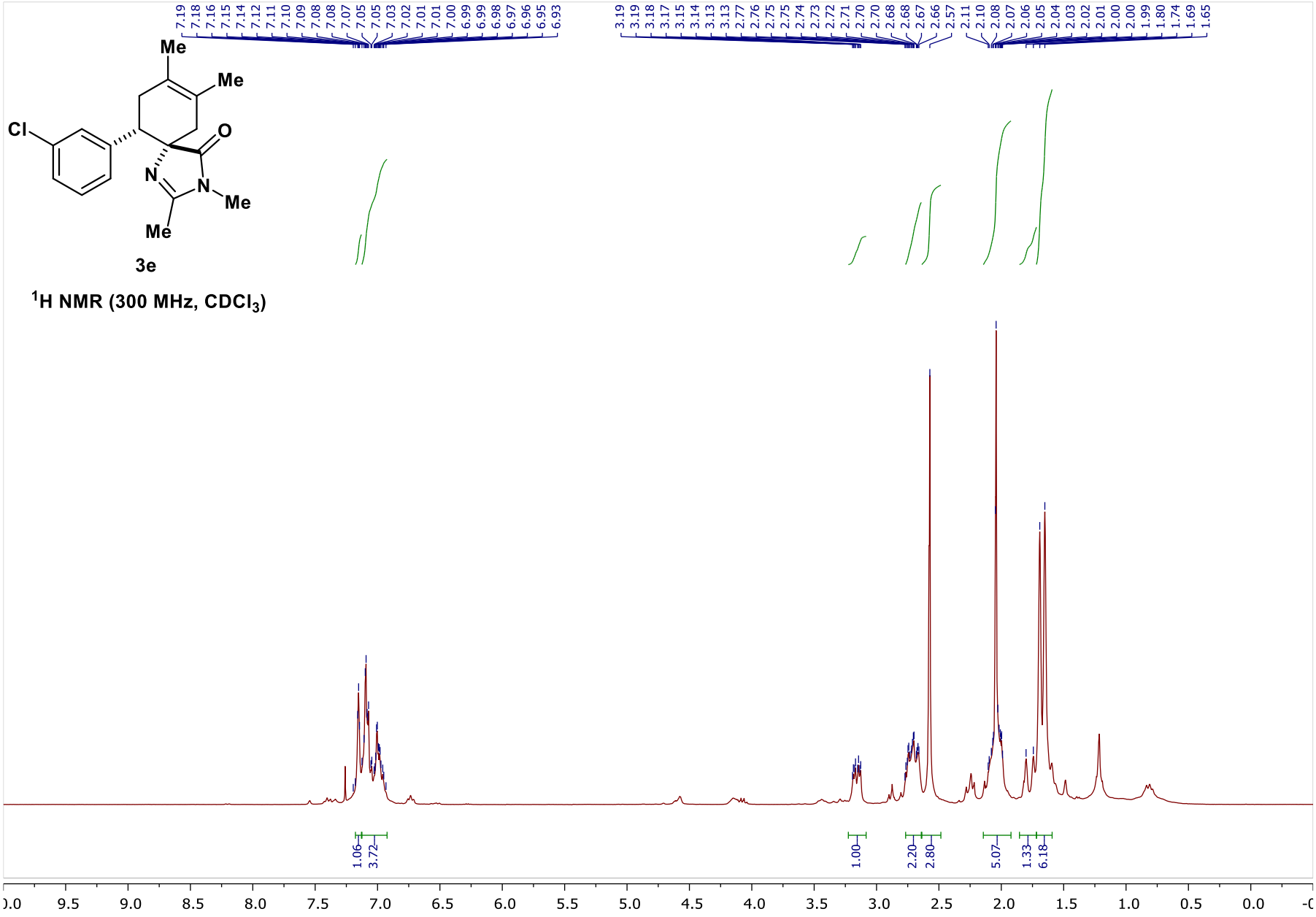
+TOF MS: 0.2371 to 0.3441 min from Sample 38 (ALM376) of 16_10_2024.wiff
a=7.01837605705241140e-004, t0=-3.04897474481943710e-001 (DuoSpray ())

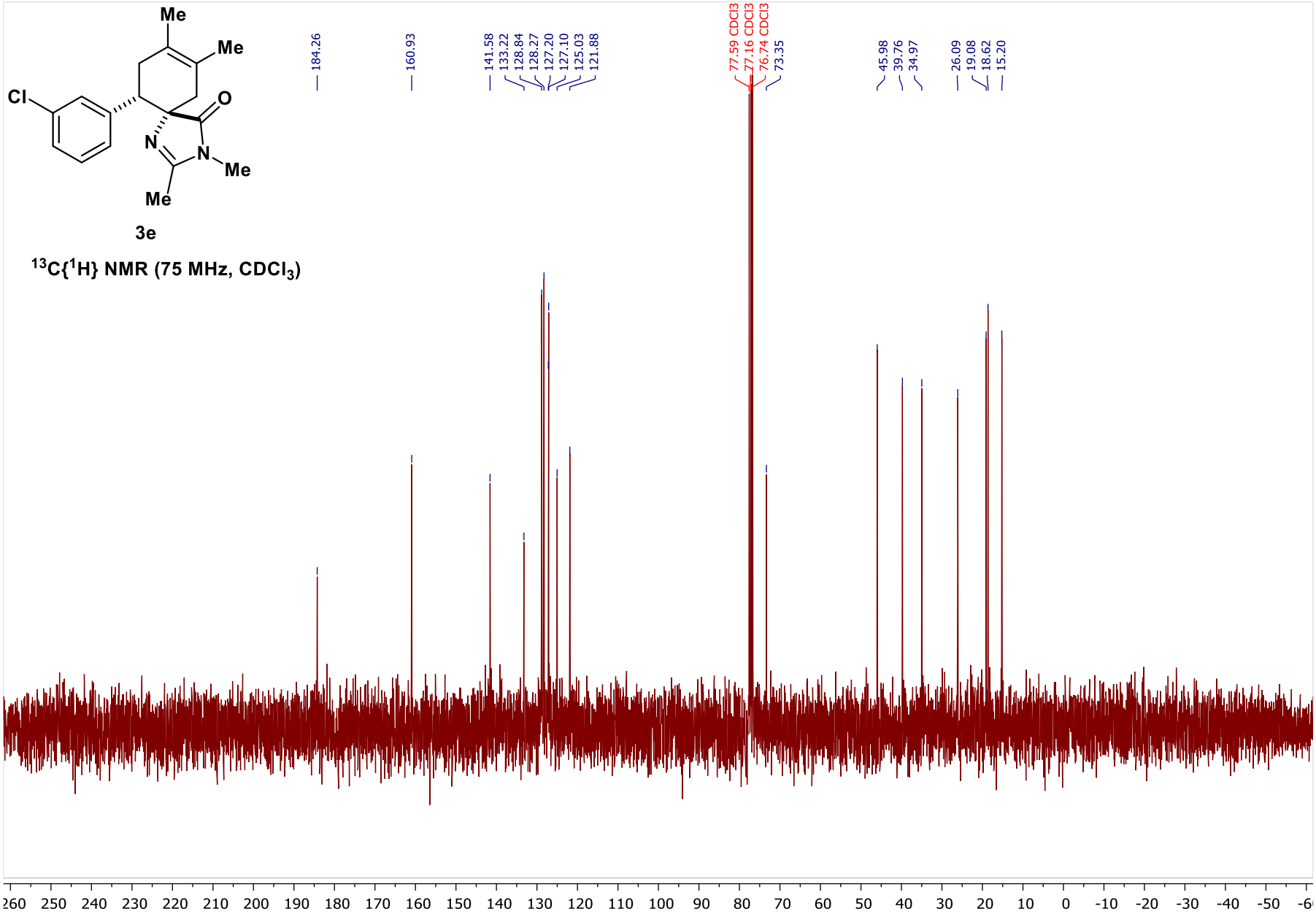
Max. 7.5e5 cps.



3d

HRMS (ESI+)


Me
Me
Cl
O
N
N
Me
Me
3e
1H NMR (300 MHz, CDCl3)
7.19 7.18 7.16 7.15 7.14 7.12 7.11 7.10 7.09 7.08 7.08 7.07 7.05 7.05 7.03 7.02 7.01 7.01 7.00 6.99 6.99 6.98 6.97 6.96 6.95 6.93
3.19 3.19 3.18 3.17 3.15 3.14 3.13 3.13 2.77 2.76 2.75 2.75 2.74 2.73 2.72 2.71 2.70 2.70 2.68 2.68 2.67 2.66 2.57 2.11 2.10 2.08 2.07 2.06 2.05 2.04 2.03 2.02 2.01 2.00 2.00 1.99 1.80 1.74 1.69 1.65
1.06 3.72 1.00 2.20 2.80 5.07 1.33 6.18
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

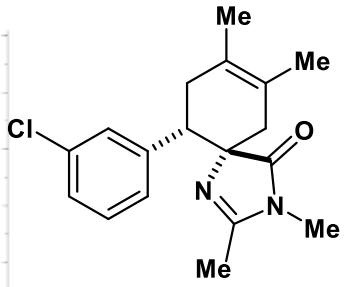

Me
Me
Cl
O
N
N
Me
Me
3e
$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$)
184.26
160.93
141.58
133.22
128.84
128.27
127.20
127.10
125.03
121.88
77.59 CDCl3
77.16 CDCl3
76.74 CDCl3
73.35
45.98
39.76
34.97
26.09
19.08
18.62
15.20
260 250 240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50

Sample Name: ALM362 *SN BN21801310 Acq. Time: 12:02

Acq. File: 05_09_2024.wiff *5600 TripleTOF Service Report Acq. Date: Thursday, September 05, 2024

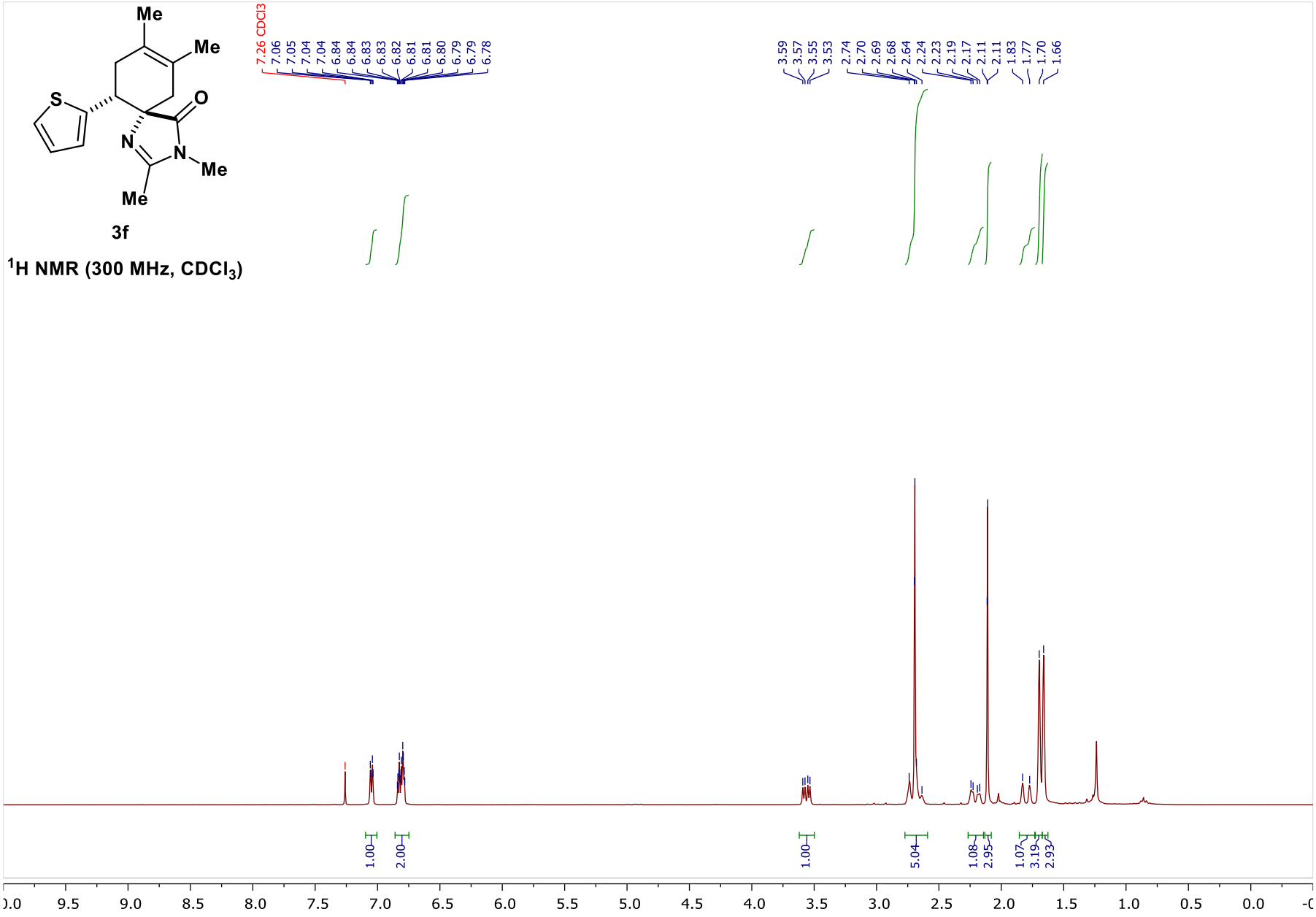
+TOF MS: 0.2650 to 0.3720 min from Sample 26 (ALM362) of 05_09_2024.wiff
a=7.01848266980272670e-004, t0=-1.58028826286703120e-001 (DuoSpray ())

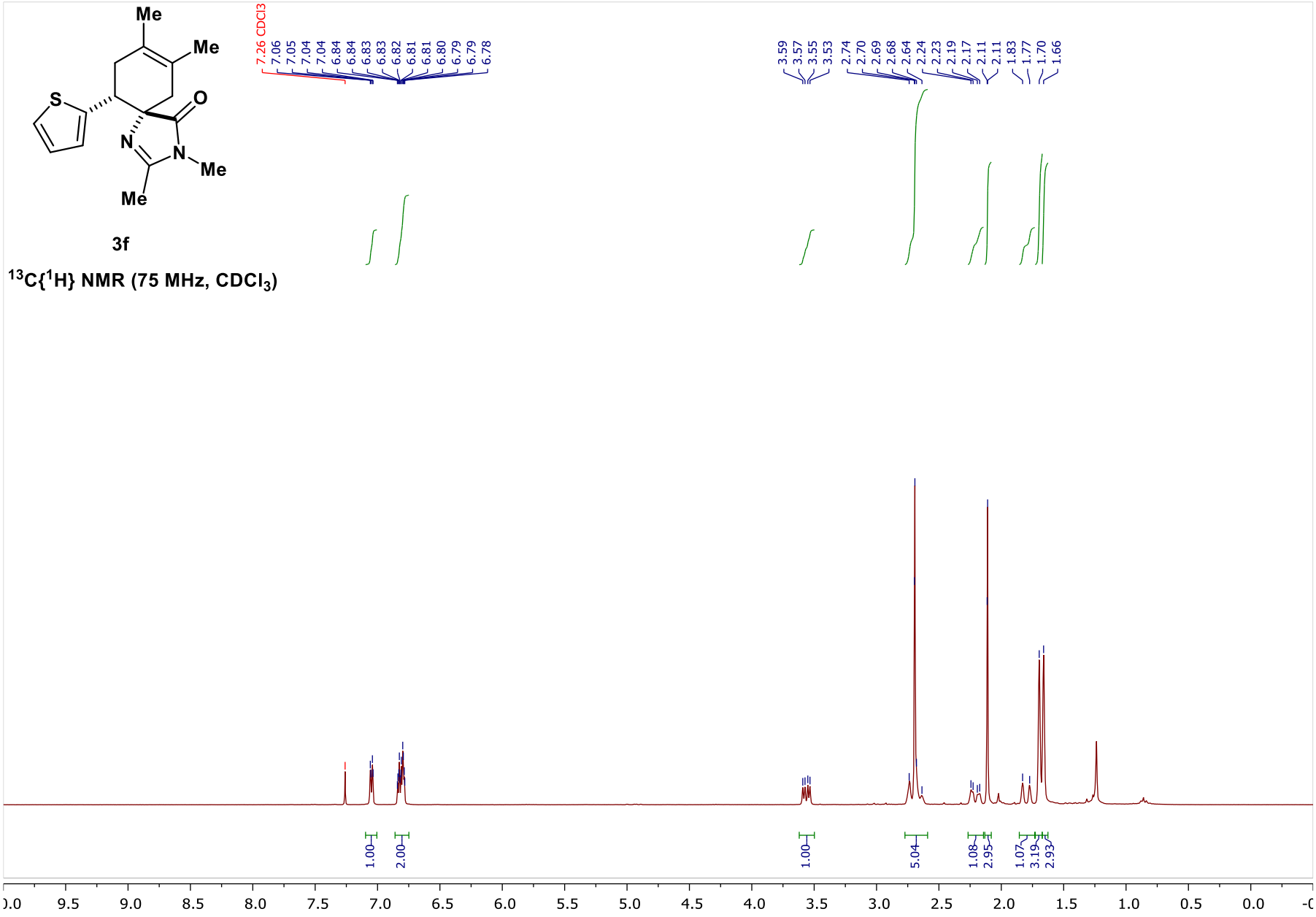
Max. 1.8e5 cps.



3e

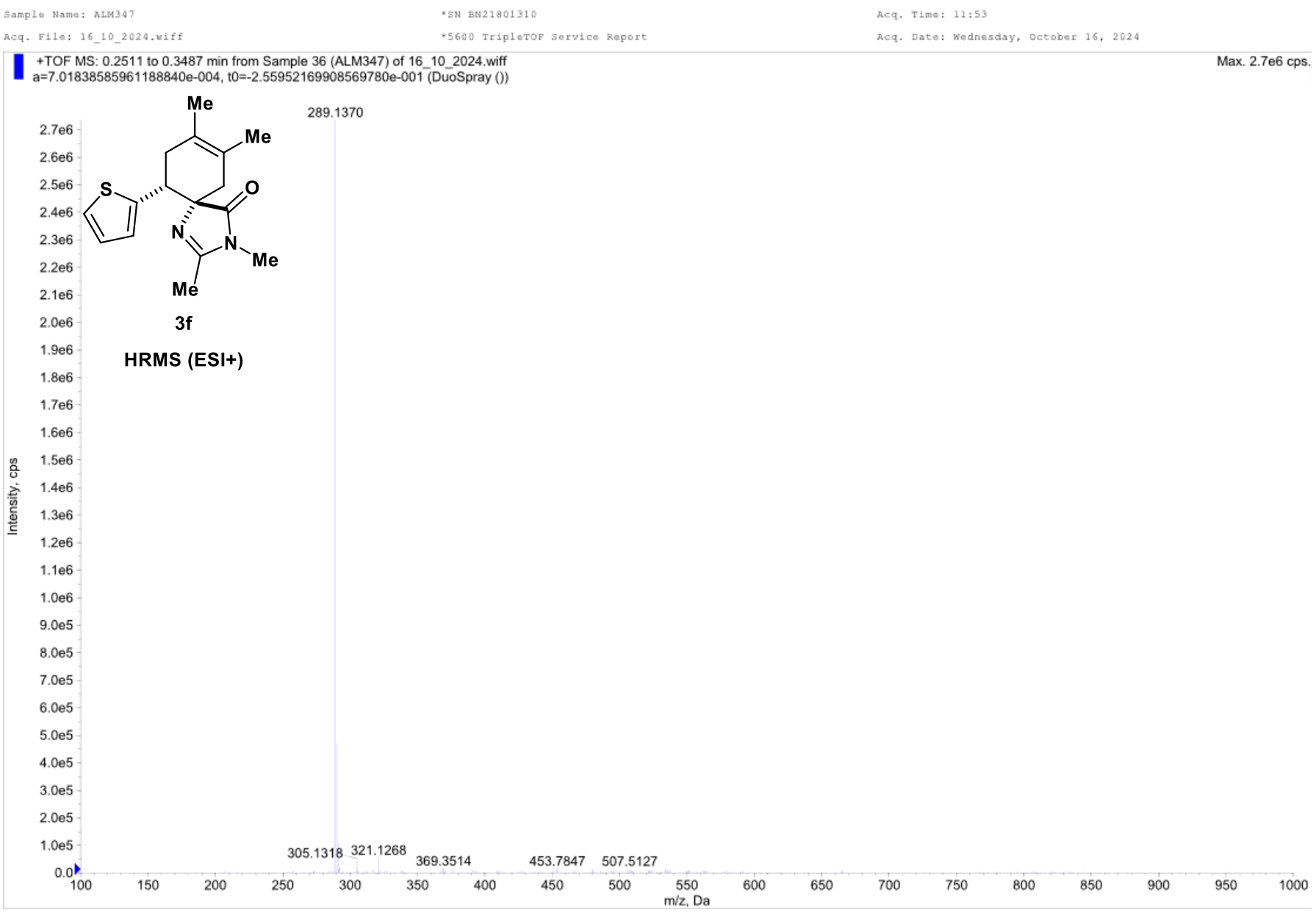
HRMS (ESI+)

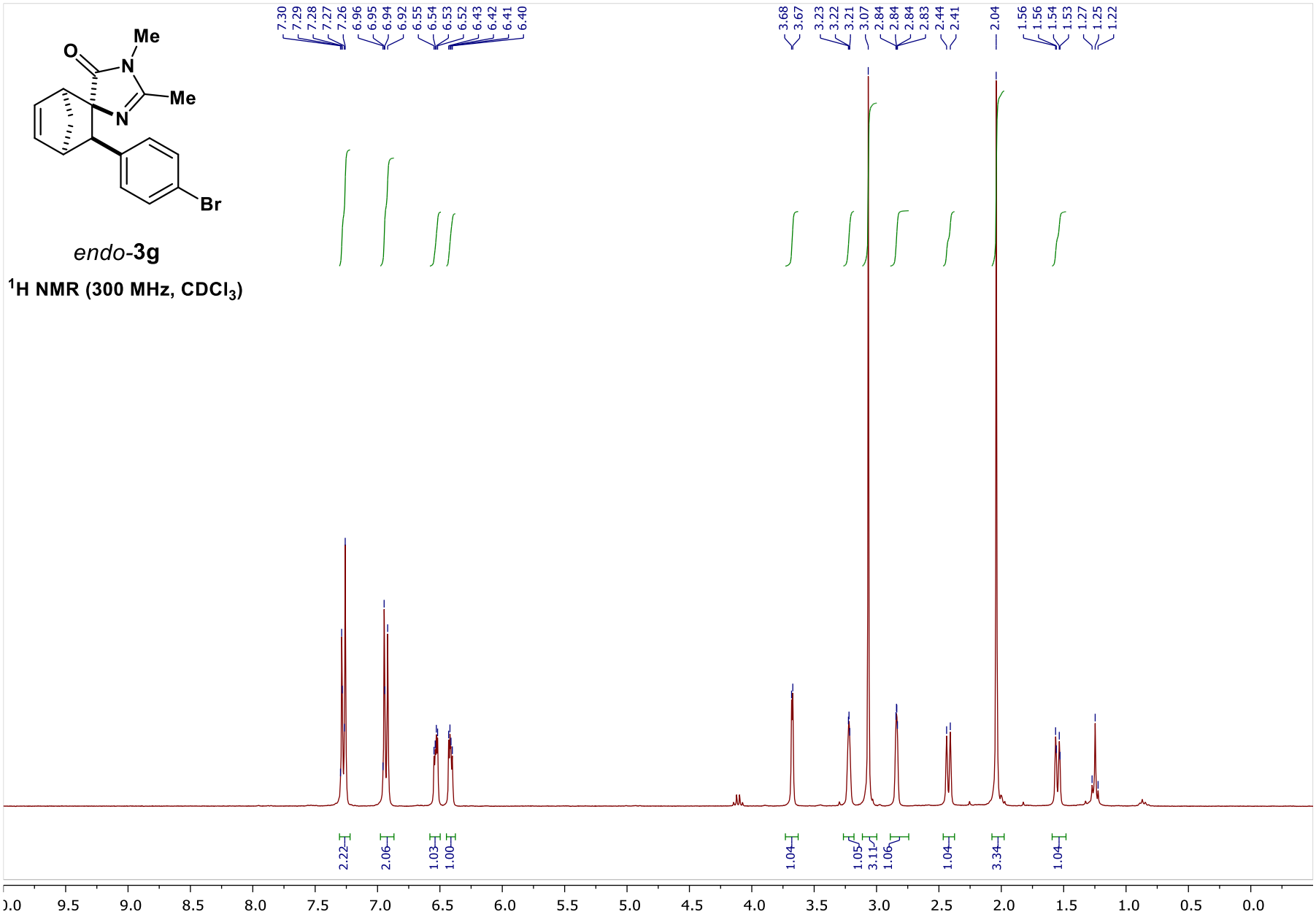

Me
Me
S
O
N
N
Me
Me
3f
^{1}H NMR (300 MHz, CDCl$_3$)
7.26 CDCl3
7.06
7.05
7.04
7.04
6.84
6.84
6.83
6.83
6.82
6.81
6.81
6.80
6.79
6.79
6.78
3.59
3.57
3.55
3.53
2.74
2.70
2.69
2.68
2.64
2.24
2.23
2.19
2.17
2.11
2.11
1.83
1.77
1.70
1.66
1.00
2.00
1.00
5.04
1.08
2.95
1.07
3.19
2.93
9.5
9.0
8.5
8.0
7.5
7.0
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



3f

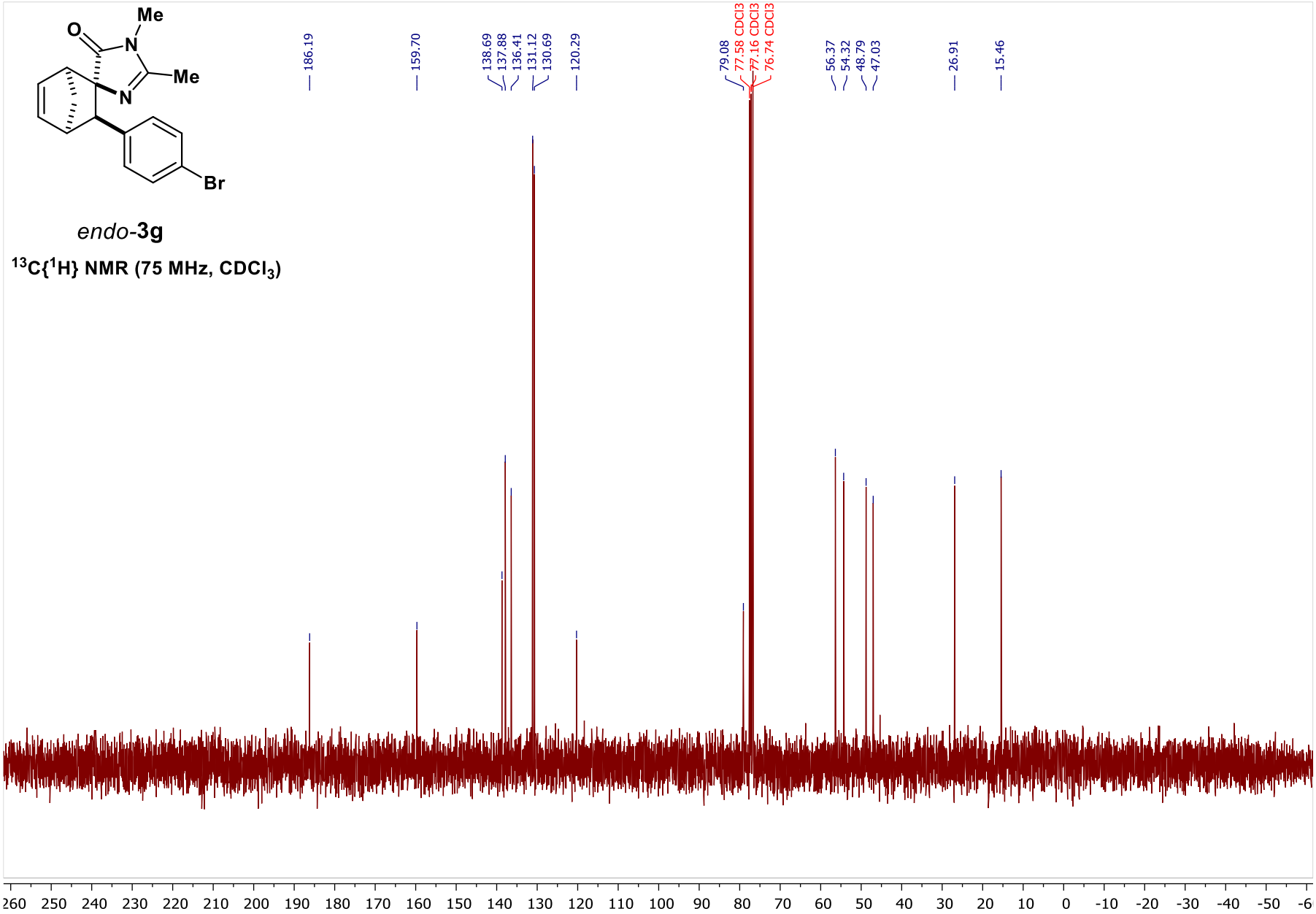
$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$)


Sample Name: ALM347
*SN BN21801310
Acq. Time: 11:53
Acq. File: 16_10_2024.wiff
*5600 TripleTOF Service Report
Acq. Date: Wednesday, October 16, 2024
+TOF MS: 0.2511 to 0.3487 min from Sample 36 (ALM347) of 16_10_2024.wiff
a=7.01838585961188840e-004, t0=-2.55952169908569780e-001 (DuoSpray ())
Max. 2.7e6 cps.
Me
Me
S
O
N
N
Me
Me
3f
HRMS (ESI+)
289.1370
Intensity, cps
m/z, Da
305.1318
321.1268
369.3514
453.7847
507.5127



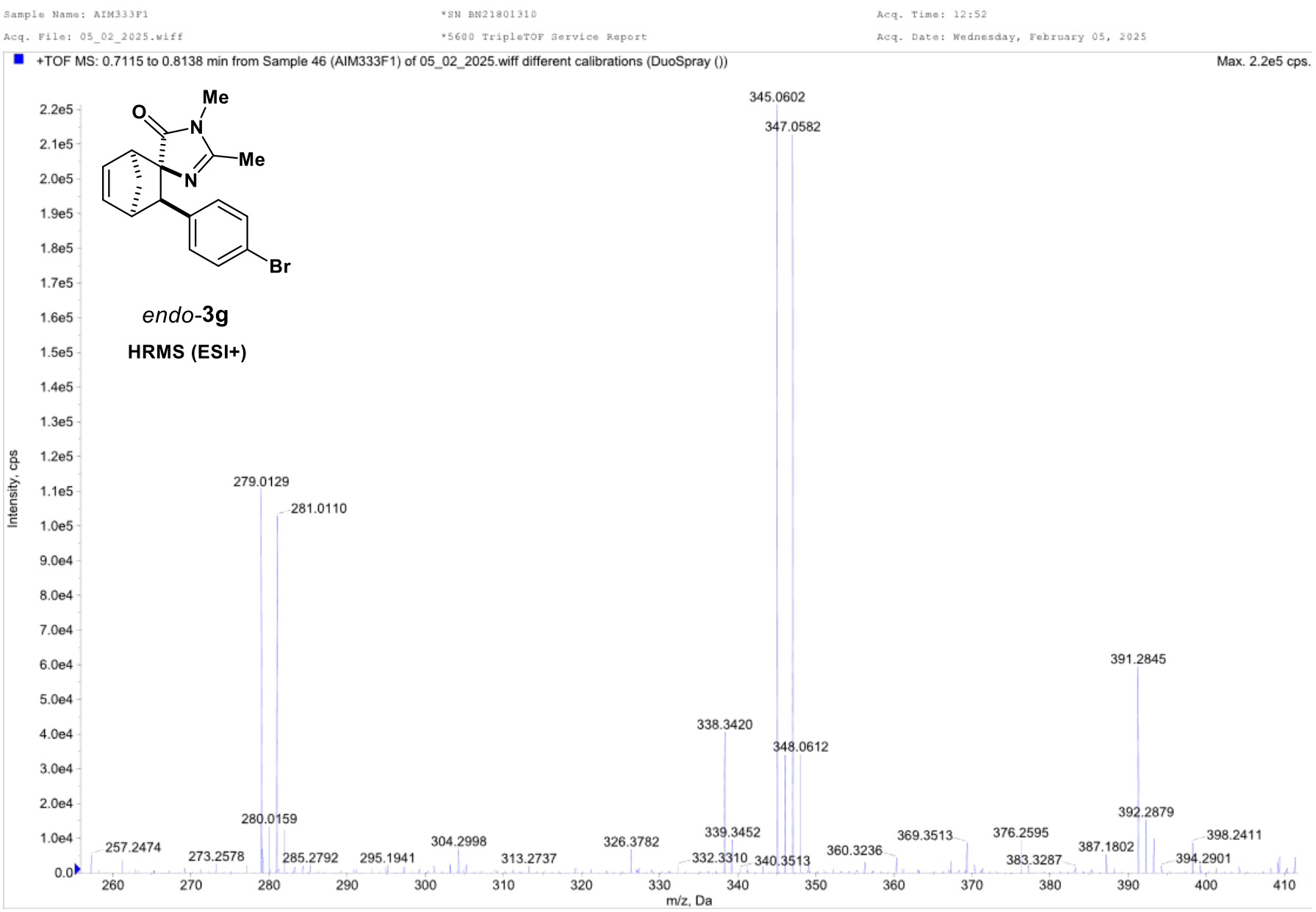
endo-**3g**

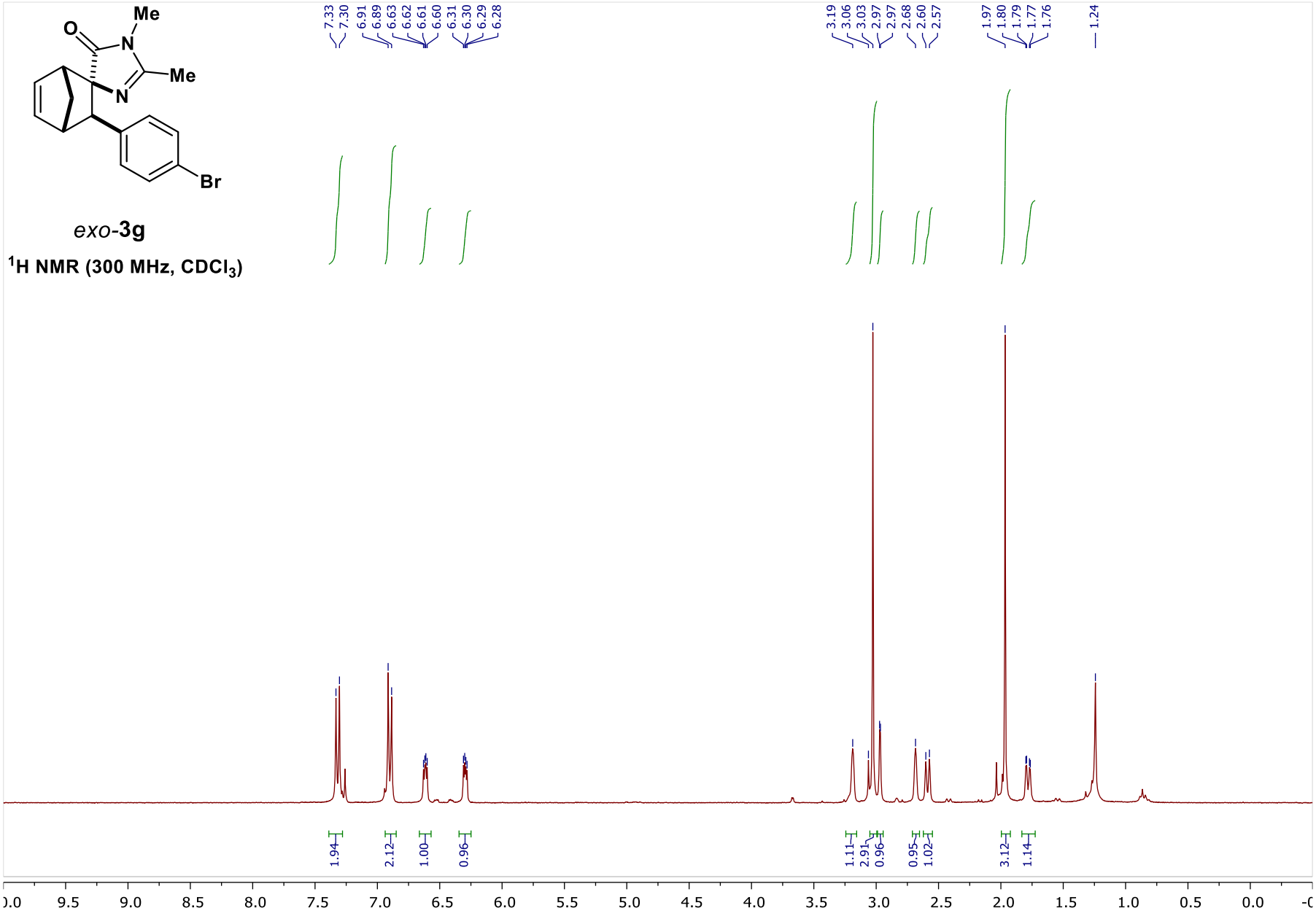
^{1}H NMR (300 MHz, $CDCl_3$)



endo-**3g**

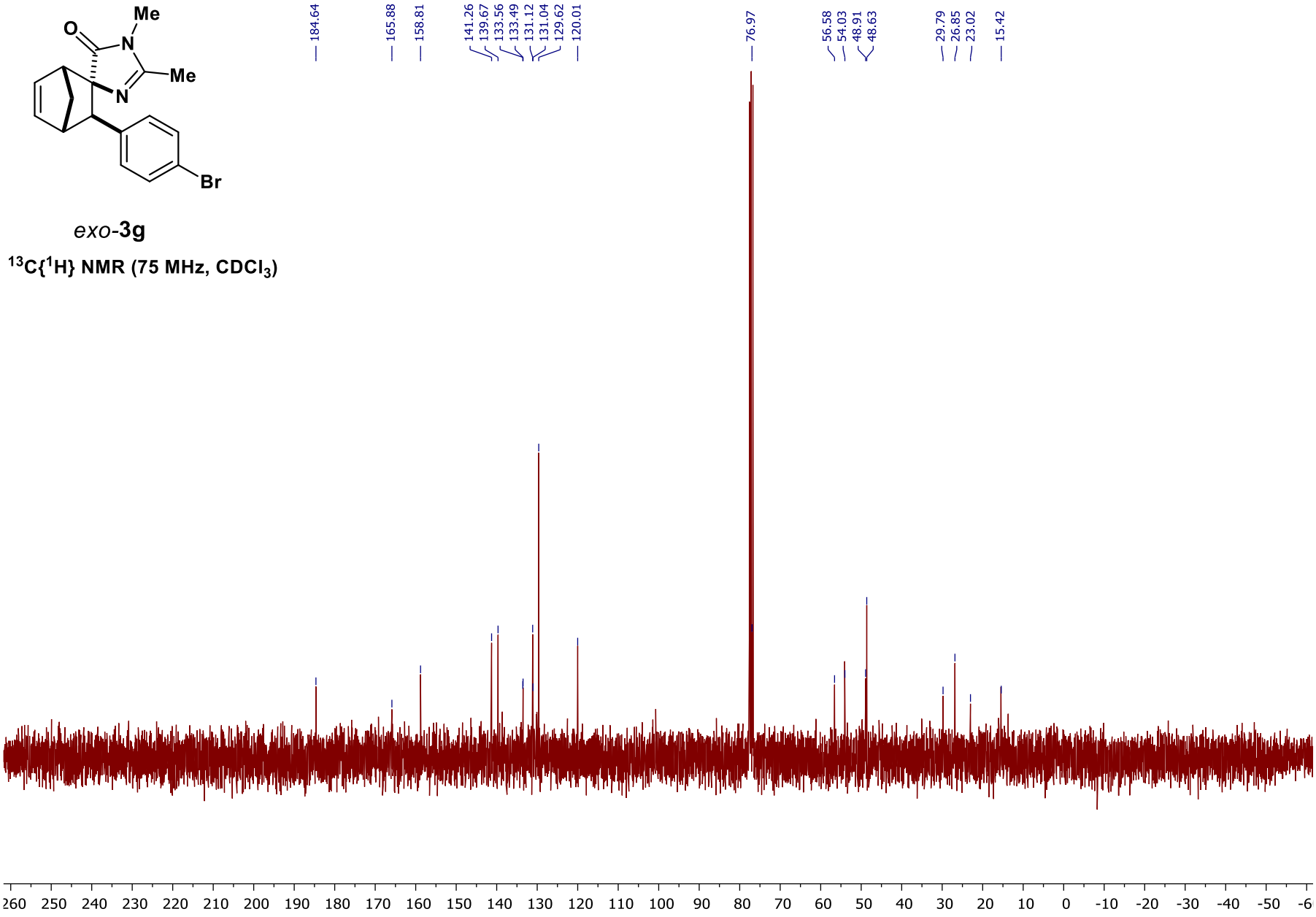
$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$)


Sample Name: AIM333F1
*SN BN21801310
Acq. Time: 12:52
Acq. File: 05_02_2025.wiff
*5600 TripleTOF Service Report
Acq. Date: Wednesday, February 05, 2025
+TOF MS: 0.7115 to 0.8138 min from Sample 46 (AIM333F1) of 05_02_2025.wiff different calibrations (DuoSpray ())
Max. 2.2e5 cps.
Me
O
N
Me
N
Br
endo-3g
HRMS (ESI+)
Intensity, cps
m/z, Da
345.0602
347.0582
279.0129
281.0110
391.2845
338.3420
348.0612
392.2879
280.0159
339.3452
398.2411
257.2474
304.2998
326.3782
369.3513
376.2595
387.1802
273.2578
285.2792
295.1941
313.2737
332.3310
340.3513
360.3236
383.3287
394.2901



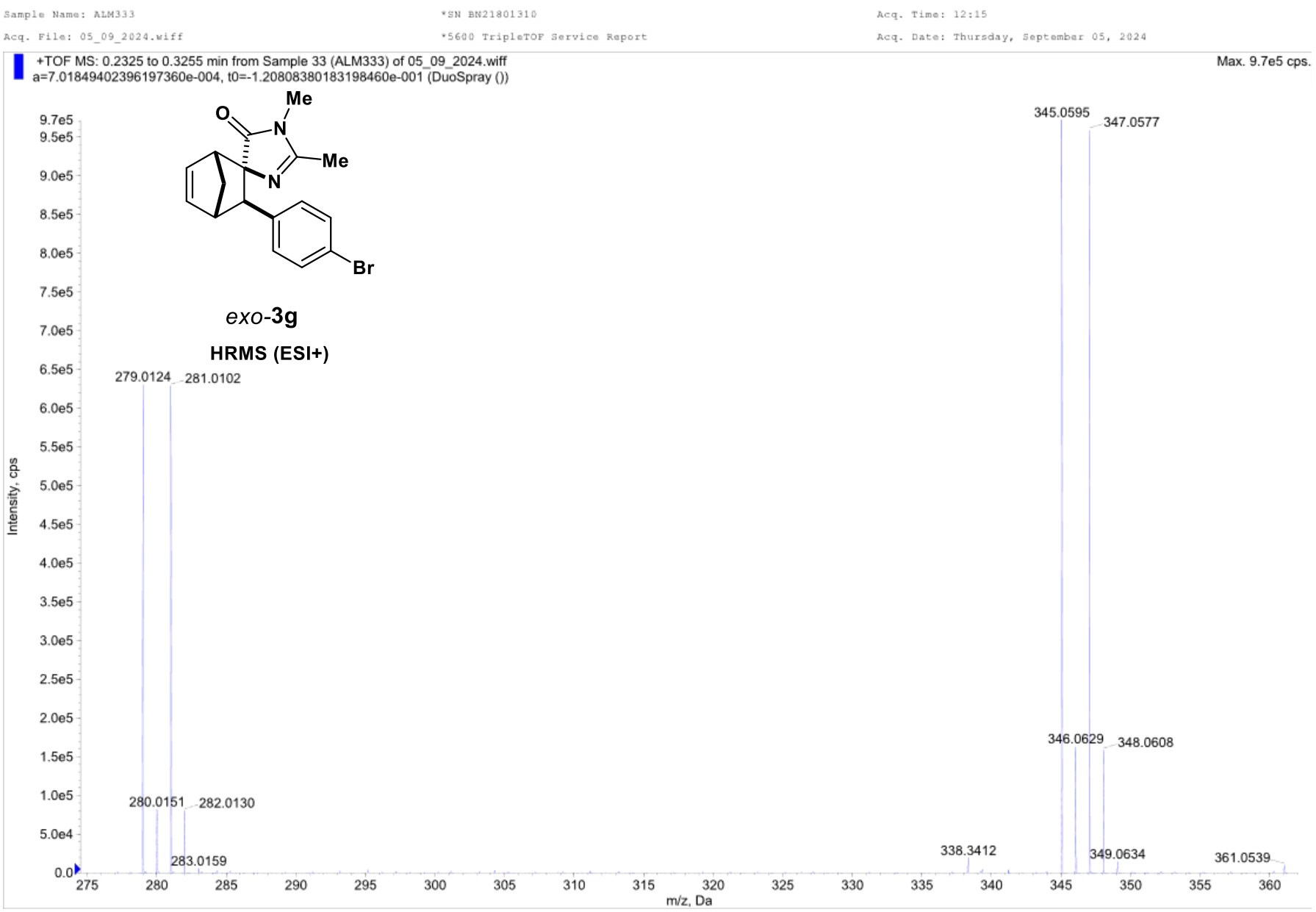
exo-**3g**

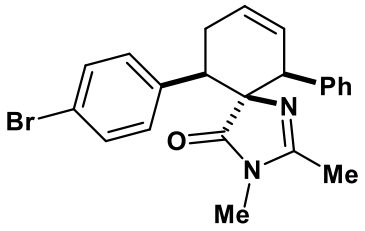
^{1}H NMR (300 MHz, $CDCl_3$)



exo-**3g**

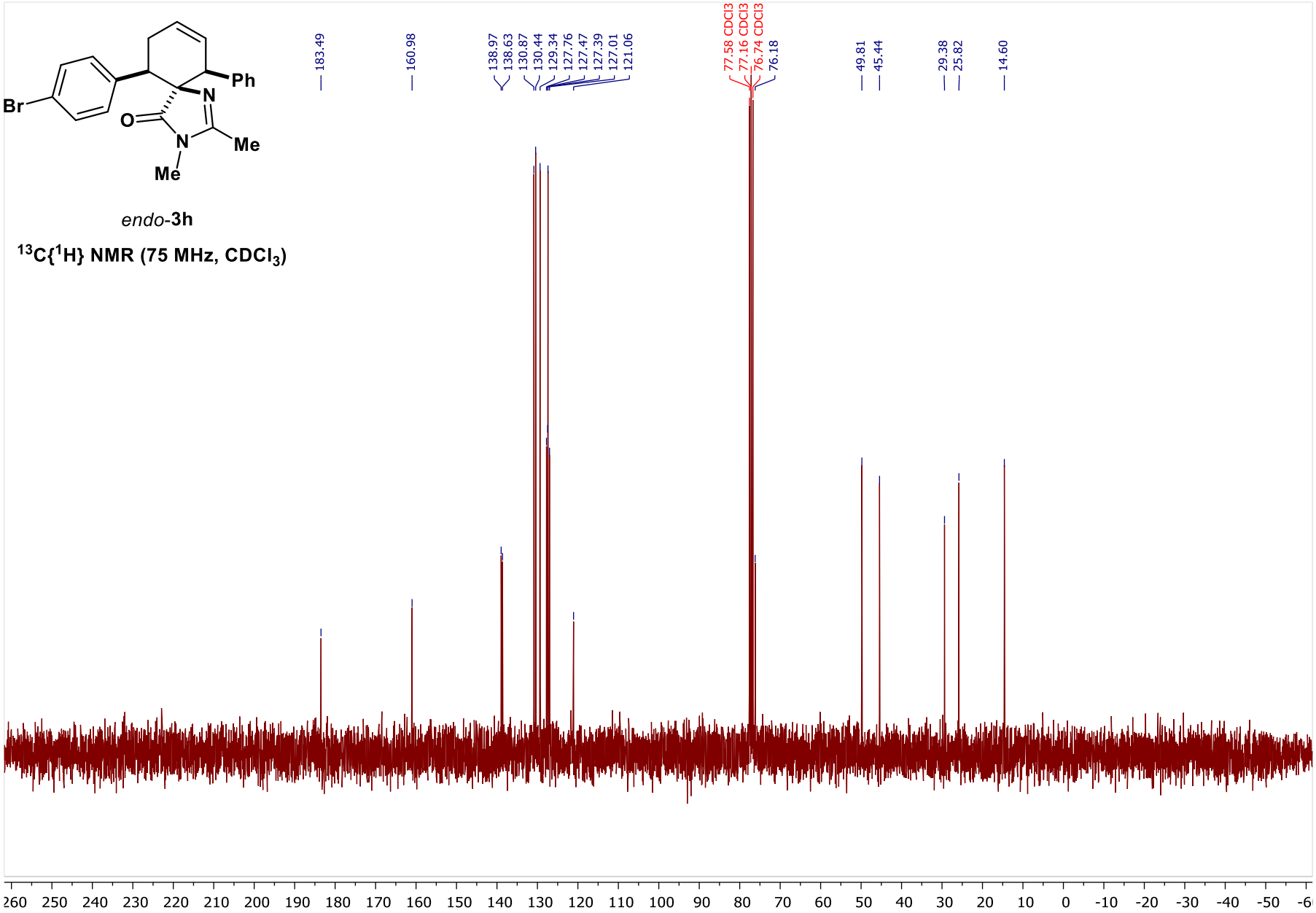
$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$)


Sample Name: ALM333
*SN BN21801310
Acq. Time: 12:15
Acq. File: 05_09_2024.wiff
*5600 TripleTOF Service Report
Acq. Date: Thursday, September 05, 2024
+TOF MS: 0.2325 to 0.3255 min from Sample 33 (ALM333) of 05_09_2024.wiff
a=7.01849402396197360e-004, t0=-1.20808380183198460e-001 (DuoSpray ())
Max. 9.7e5 cps.
Me
O
N
Me
N
Br
exo-3g
HRMS (ESI+)
Intensity, cps
m/z, Da
345.0595
347.0577
279.0124
281.0102
346.0629
348.0608
280.0151
282.0130
283.0159
338.3412
349.0634
361.0539



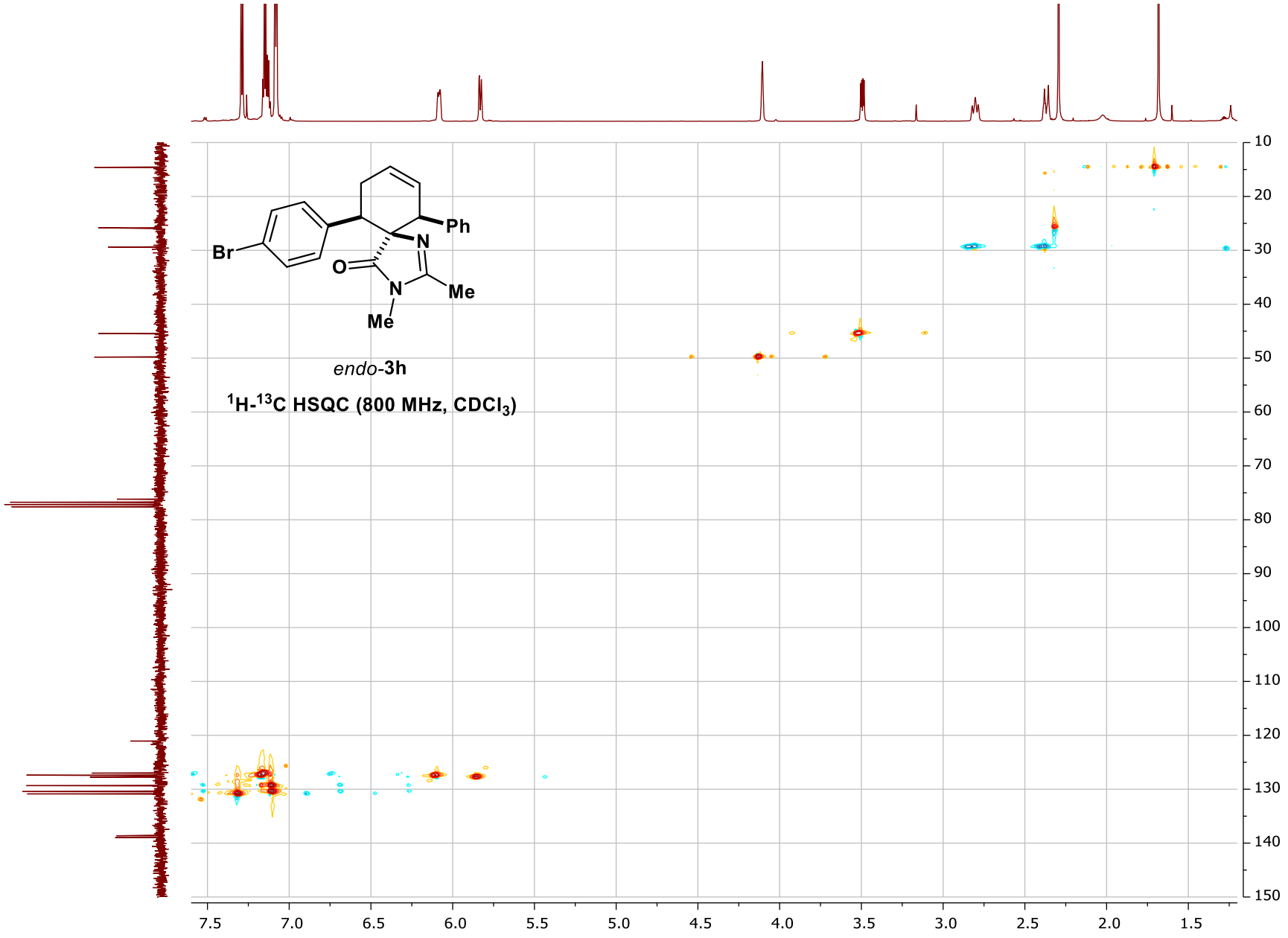
endo-**3h**

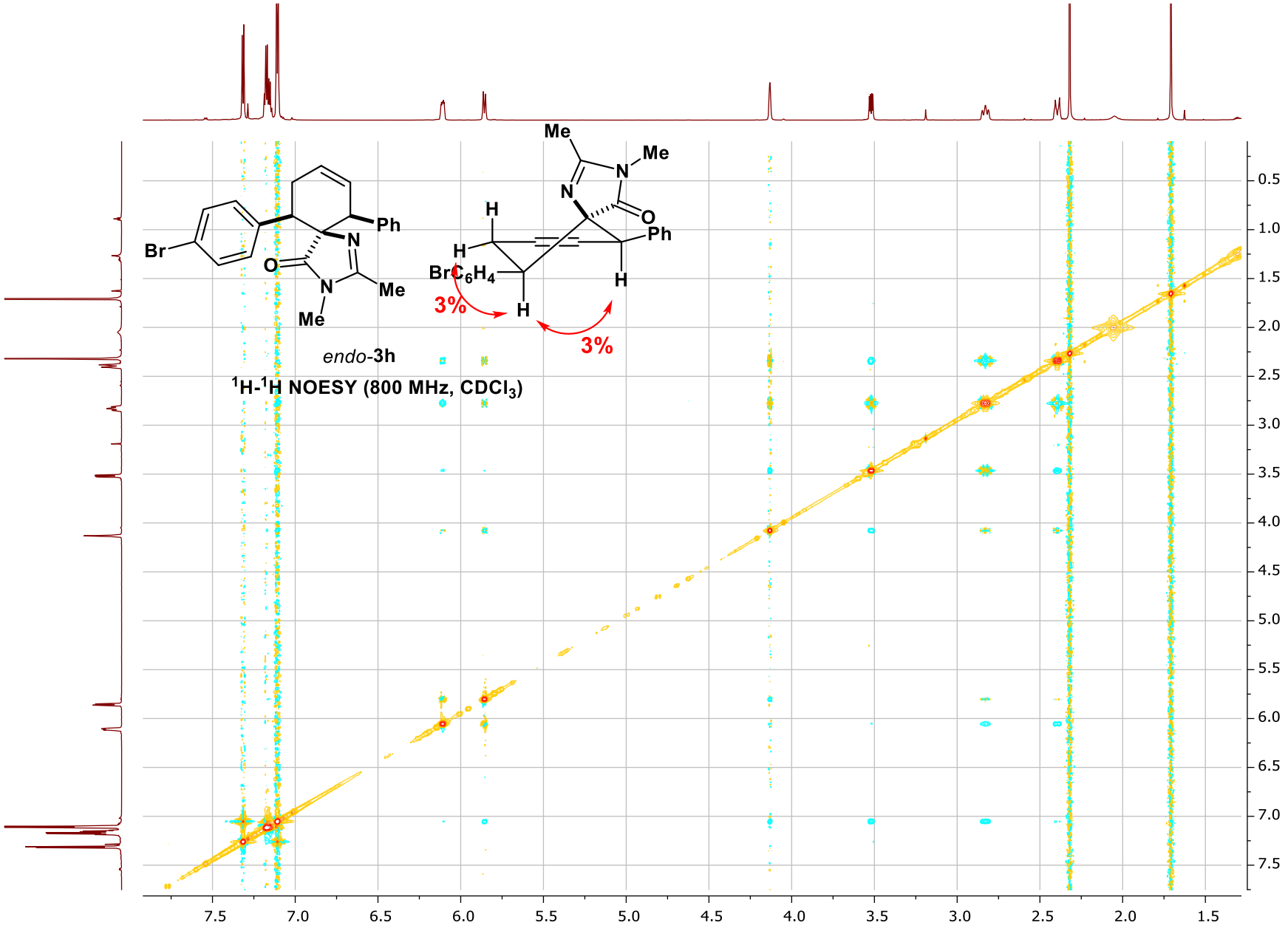
^{1}H NMR (800 MHz, $CDCl_3$)

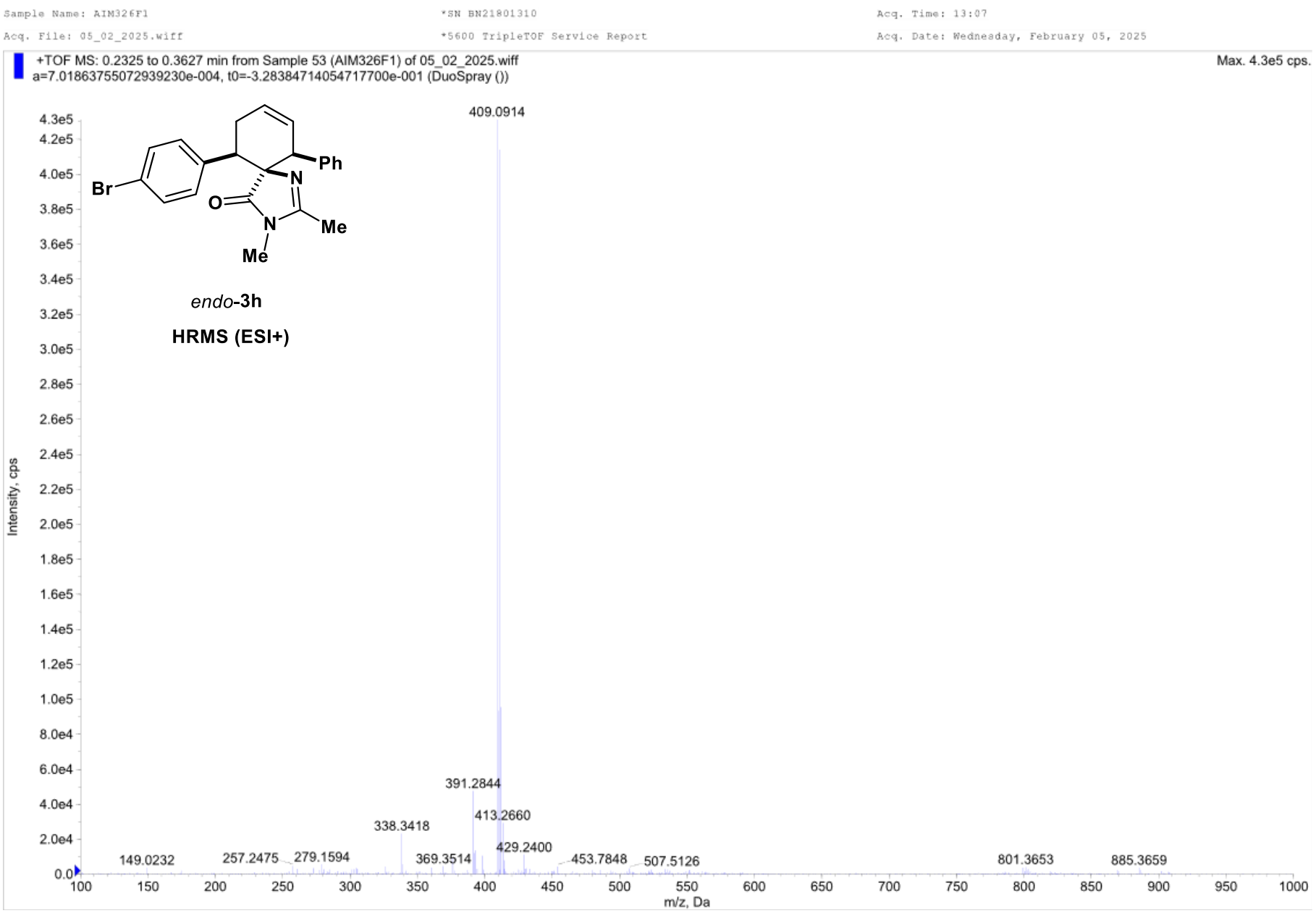


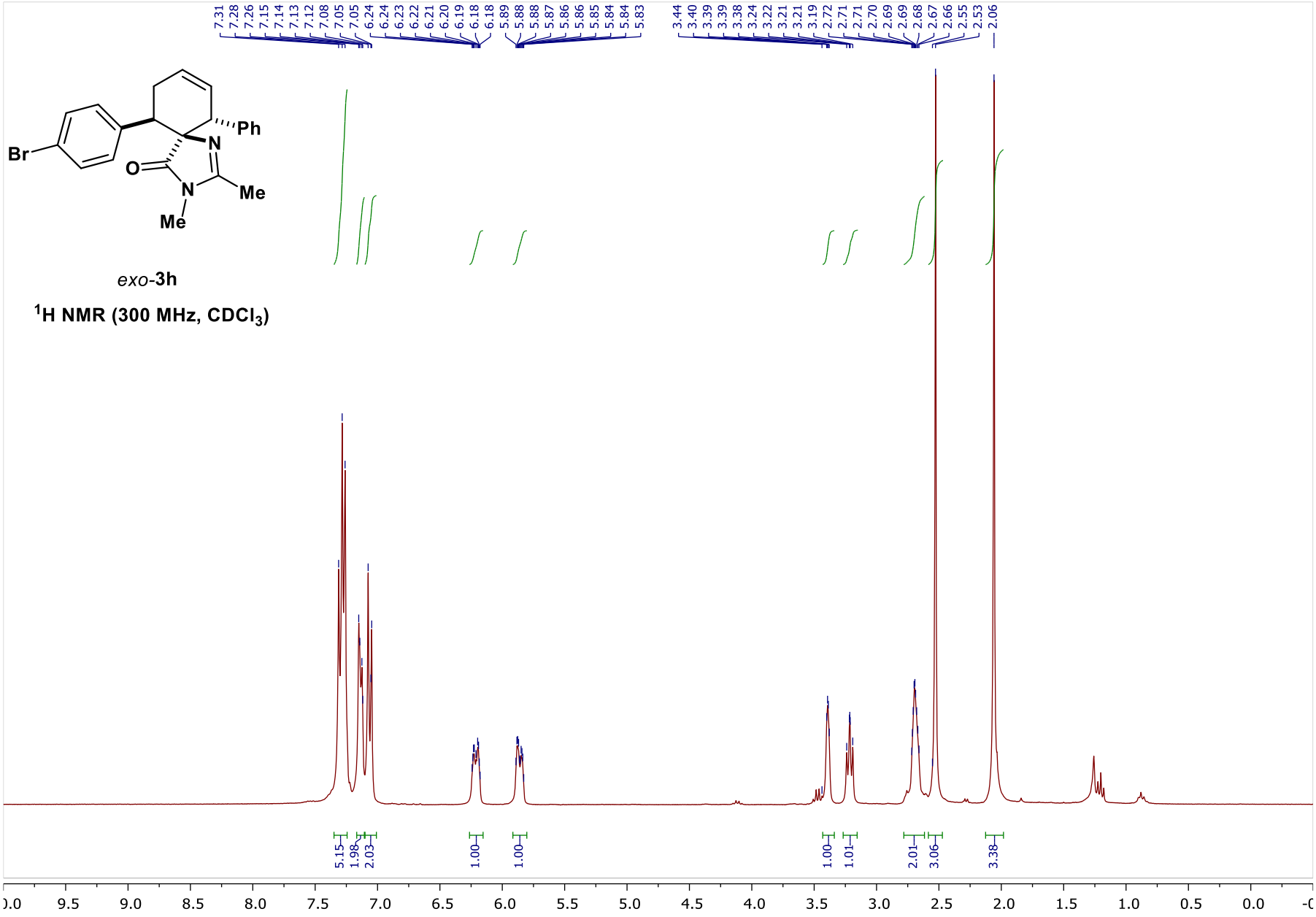
endo-**3h**

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$)


Br
Ph
N
O
N
Me
Me
endo-3h
1H-13C HSQC (800 MHz, CDCl3)
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
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

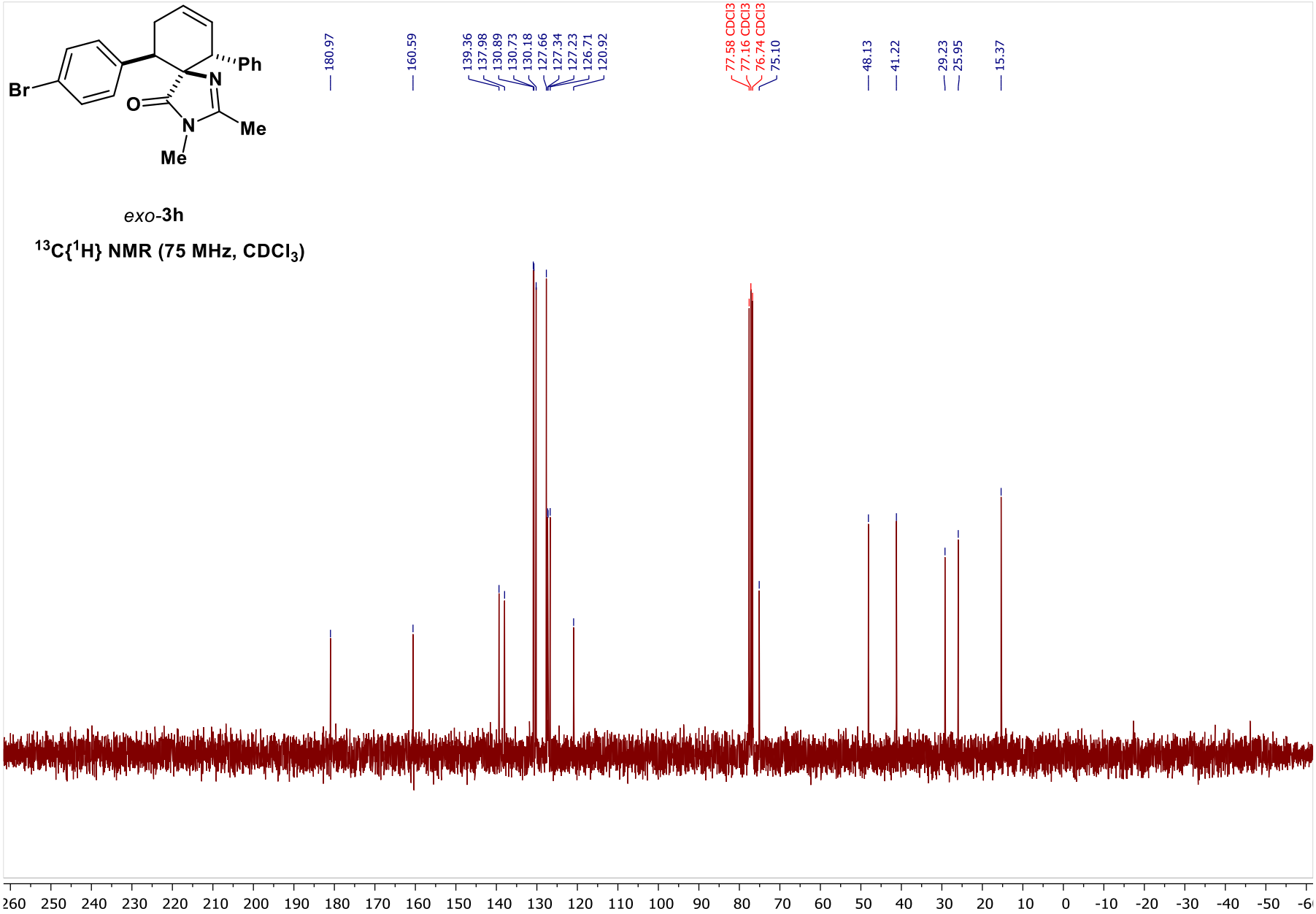

Br
Ph
N
O
N
Me
Me
Me
N
N
O
Ph
H
H
BrC6H4
H
3%
3%
endo-3h
1H-1H NOESY (800 MHz, CDCl3)
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5


Sample Name: AIM326F1
*SN BN21801310
Acq. Time: 13:07
Acq. File: 05_02_2025.wiff
*5600 TripleTOF Service Report
Acq. Date: Wednesday, February 05, 2025
+TOF MS: 0.2325 to 0.3627 min from Sample 53 (AIM326F1) of 05_02_2025.wiff
a=7.0186375072939230e-004, t0=-3.2838471405471700e-001 (DuoSpray ())
Max. 4.3e5 cps.
Br
O
N
Ph
N
Me
Me
endo-3h
HRMS (ESI+)
409.0914
391.2844
413.2660
338.3418
429.2400
149.0232
257.2475
279.1594
369.3514
453.7848
507.5126
801.3653
885.3659
Intensity, cps
m/z, Da



exo-**3h**

^{1}H NMR (300 MHz, $CDCl_3$)



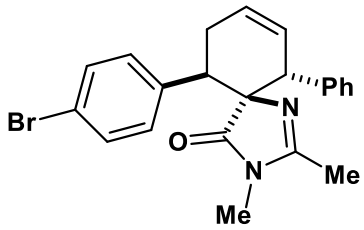
exo-**3h**

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$)

Sample Name: ALM326 *SN BN21801310 Acq. Time: 12:34

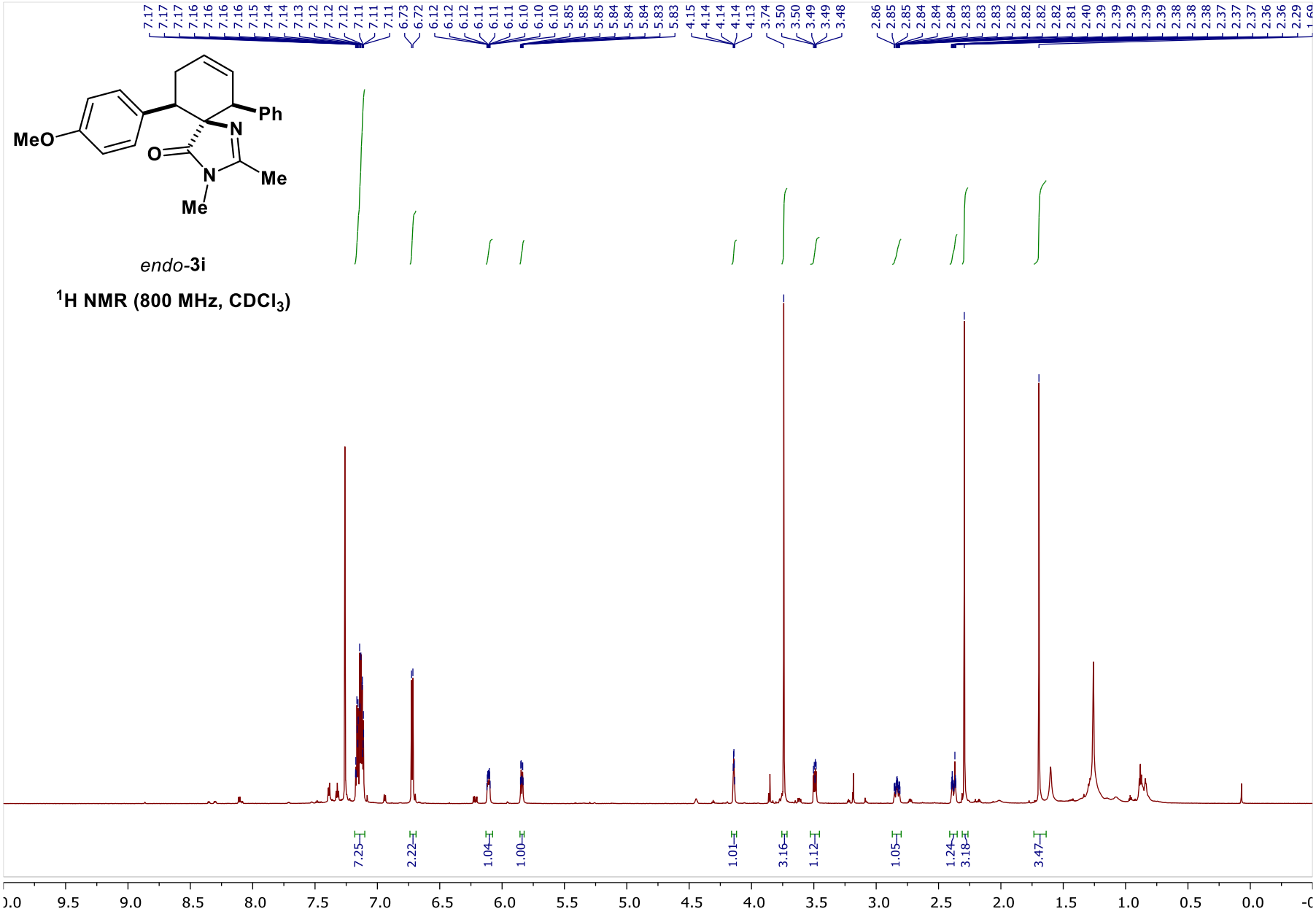
Acq. File: 05_09_2024.wiff *5600 TripleTOF Service Report Acq. Date: Thursday, September 05, 2024

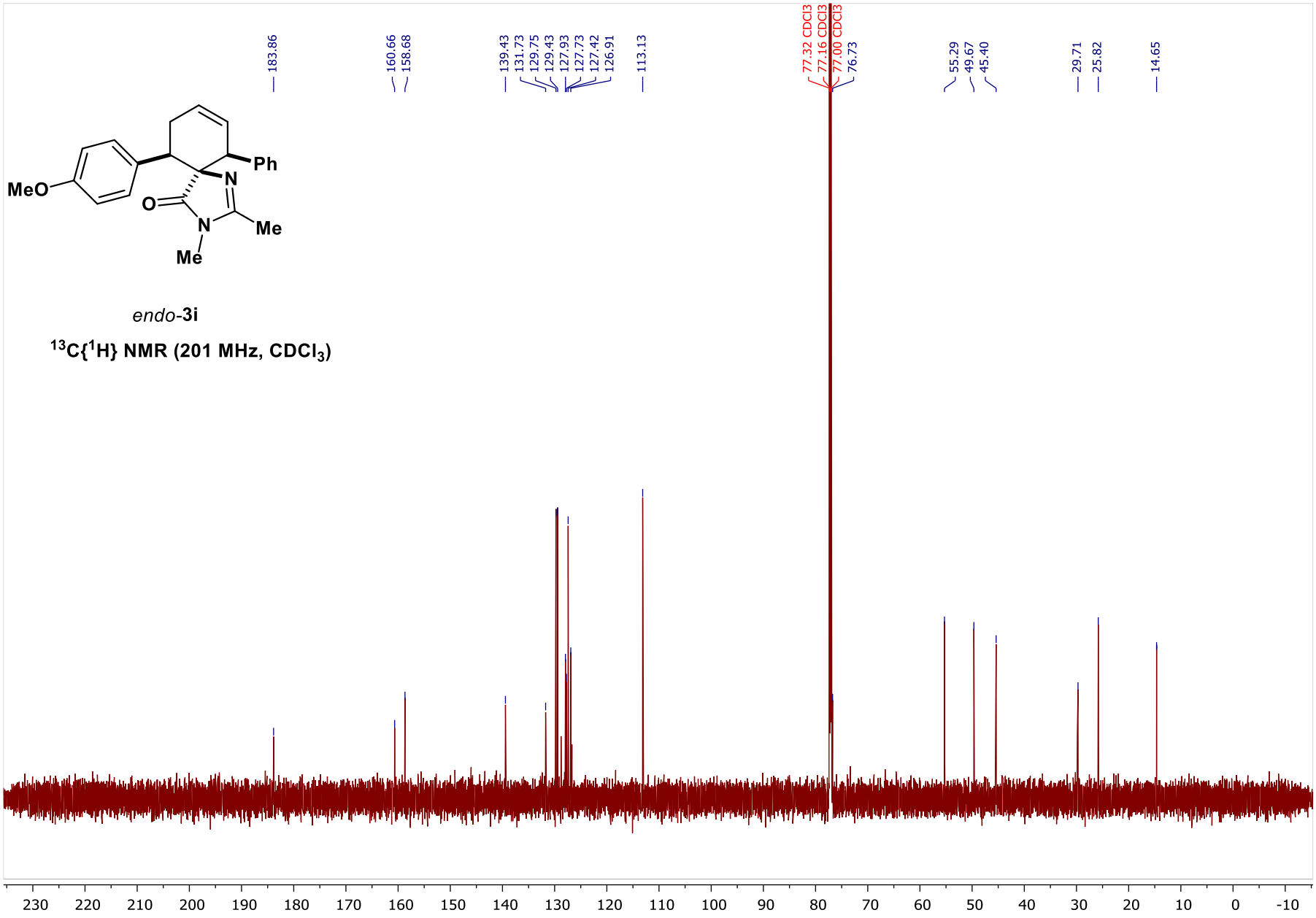
+TOF MS: 0.6836 to 0.8138 min from Sample 41 (ALM326) of 05_09_2024.wiff different calibrations (DuoSpray ()) Max. 2.0e6 cps.



exo-**3h**

HRMS (ESI+)


MeO
Ph
N
O
N
Me
Me
endo-3i
1H NMR (800 MHz, CDCl3)



endo-**3i**

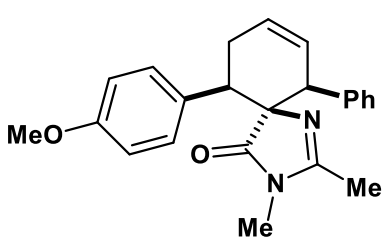
$^{13}C\{^{1}H\}$ NMR (201 MHz, $CDCl_3$)

Sample Name: AIM377F1 *SN BN21801310 Acq. Time: 12:56

Acq. File: 05_02_2025.wiff *5600 TripleTOF Service Report Acq. Date: Wednesday, February 05, 2025

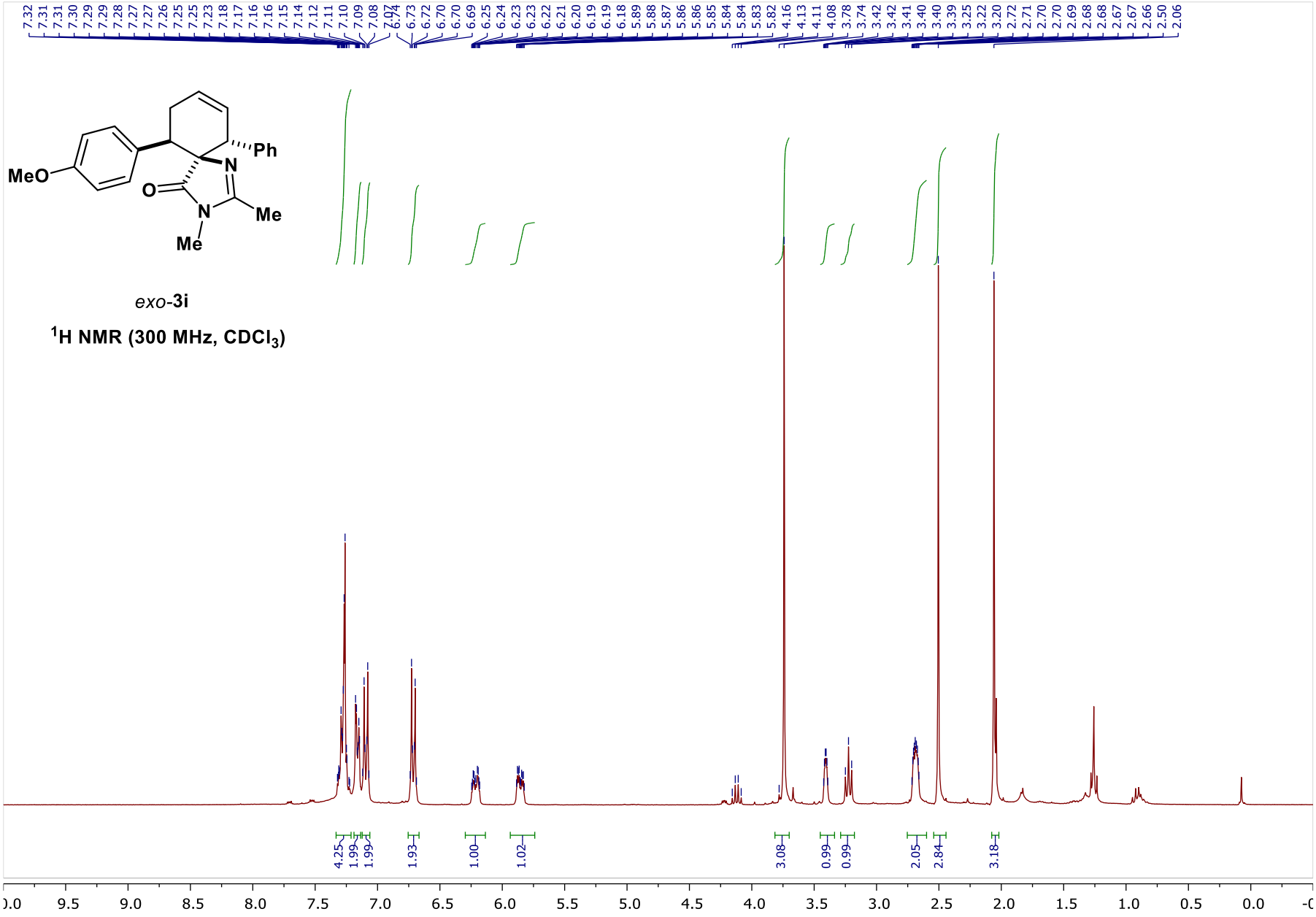
+TOF MS: 0.2325 to 0.3395 min from Sample 48 (AIM377F1) of 05_02_2025.wiff
a=7.01863721688047920e-004, t0=-3.30382014527007940e-001 (DuoSpray ())

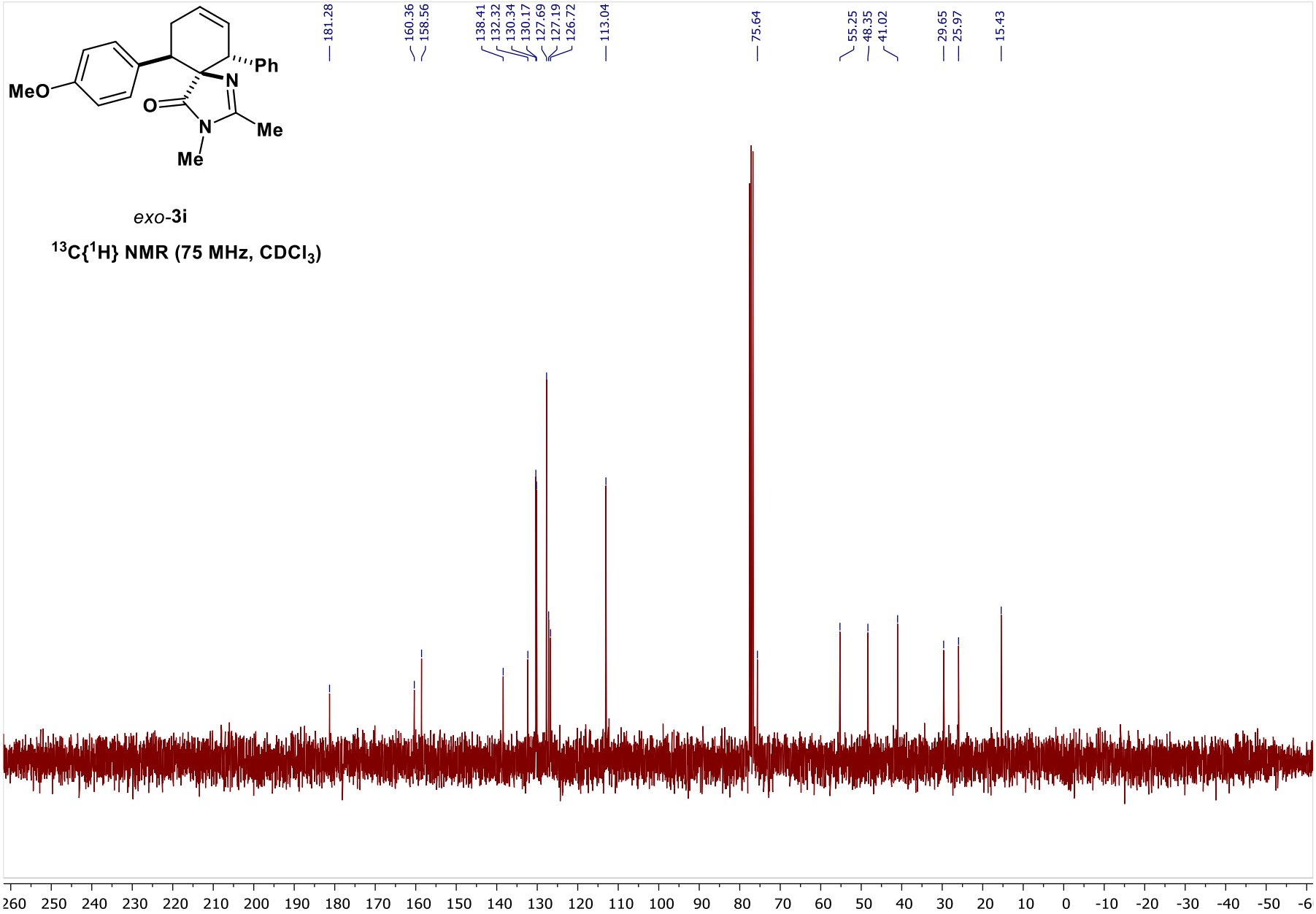
Max. 2.6e5 cps.



endo-**3i**

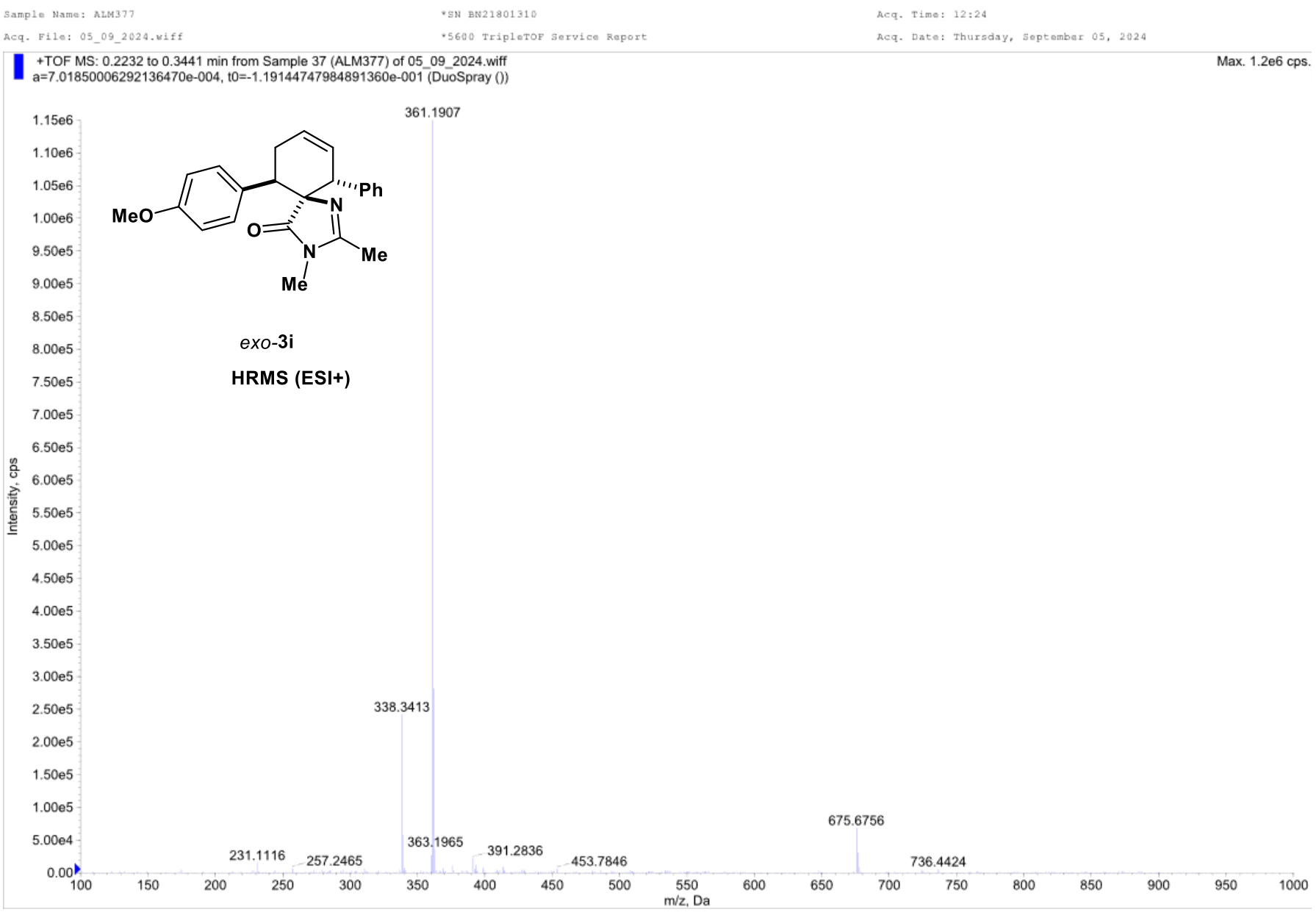
HRMS (ESI+)

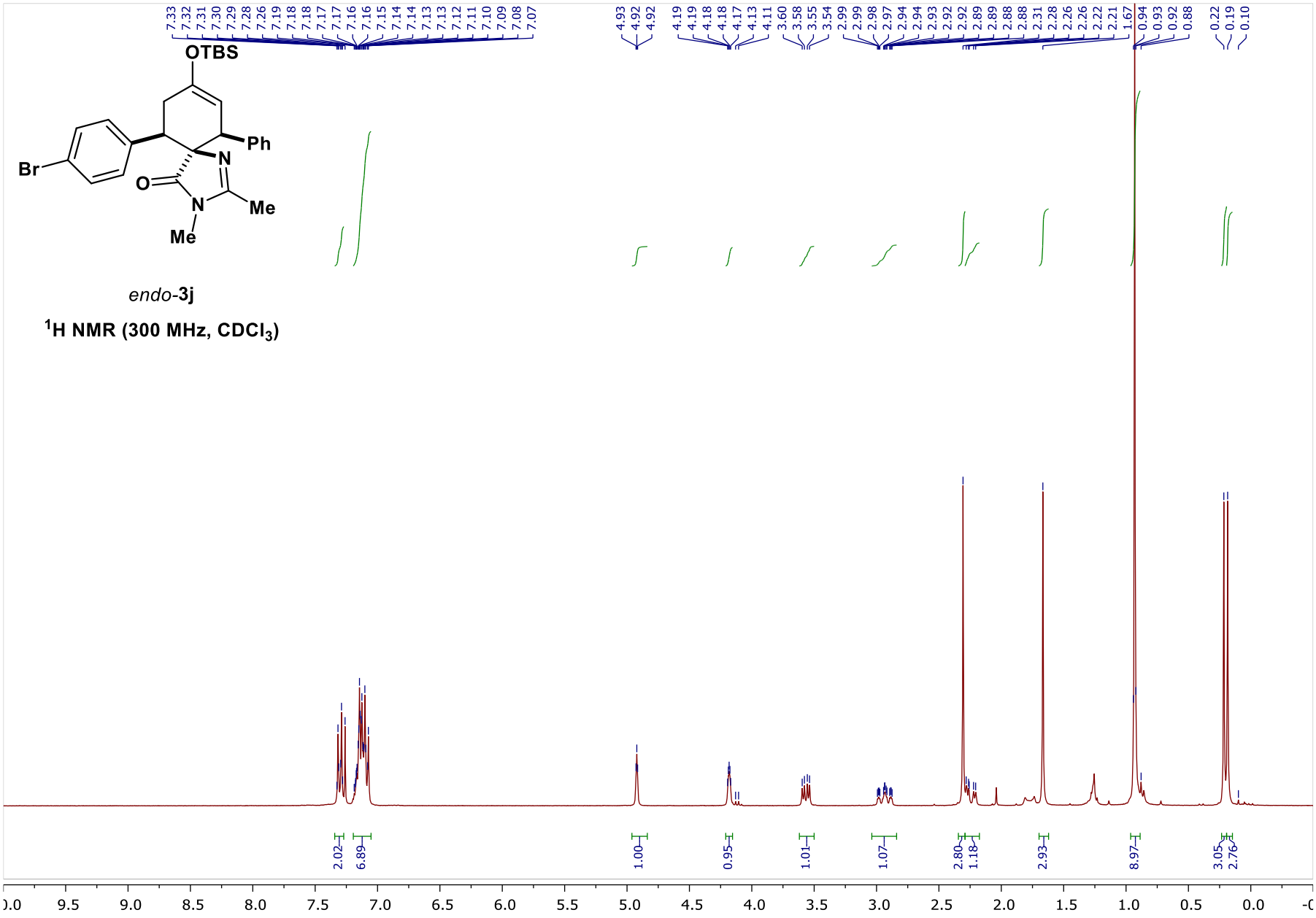

MeO
Ph
N
O
N
Me
Me
exo-3i
1H NMR (300 MHz, CDCl3)



exo-**3i**

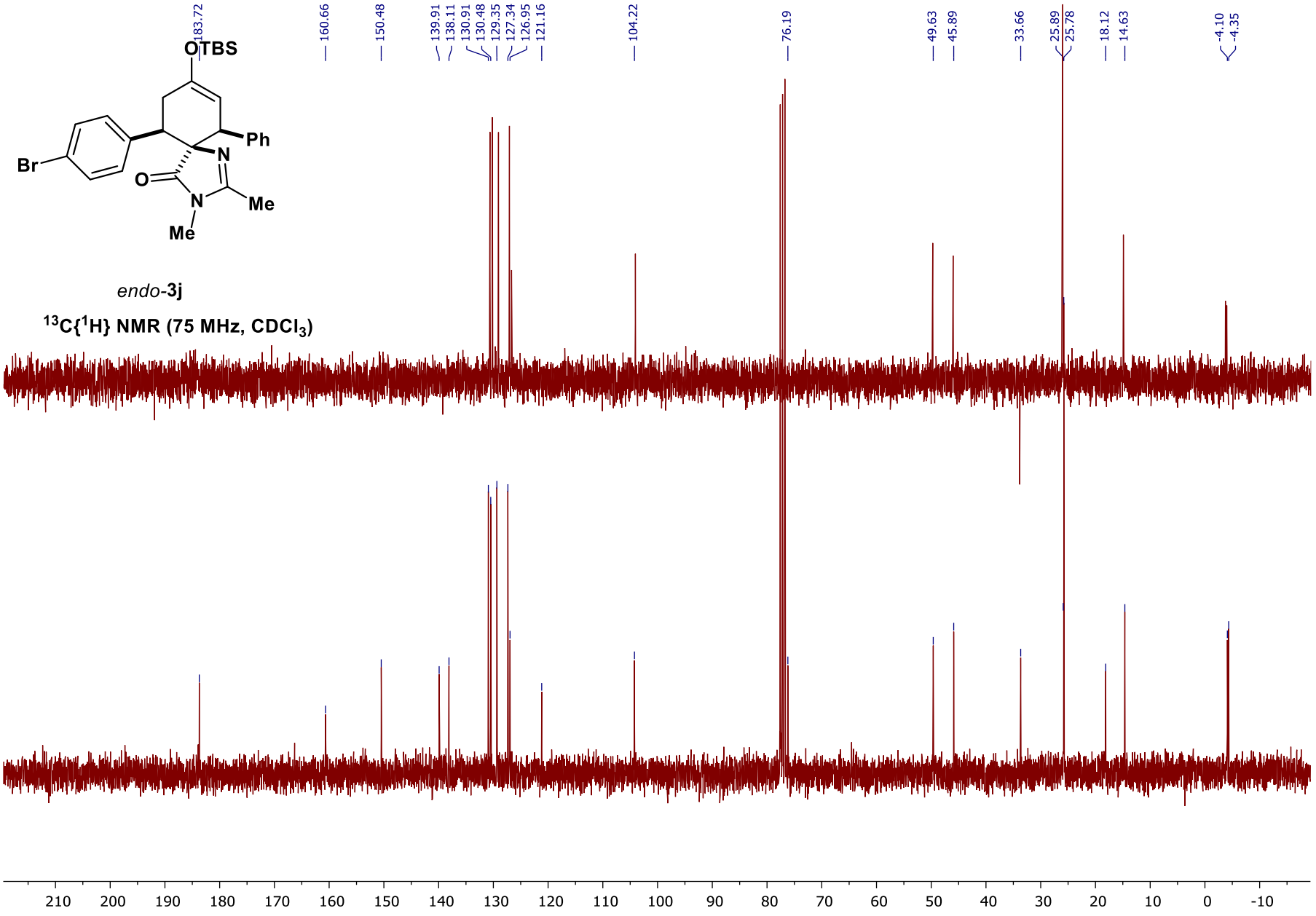
$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$)


Sample Name: ALM377
*SN BN21801310
Acq. Time: 12:24
Acq. File: 05_09_2024.wiff
*5600 TripleTOF Service Report
Acq. Date: Thursday, September 05, 2024
+TOF MS: 0.2232 to 0.3441 min from Sample 37 (ALM377) of 05_09_2024.wiff
a=7.01850006292136470e-004, t0=-1.19144747984891360e-001 (DuoSpray ())
Max. 1.2e6 cps.
MeO
Ph
N
O
N
Me
Me
exo-3i
HRMS (ESI+)
Intensity, cps
m/z, Da
361.1907
338.3413
675.6756
363.1965
391.2836
231.1116
257.2465
453.7846
736.4424



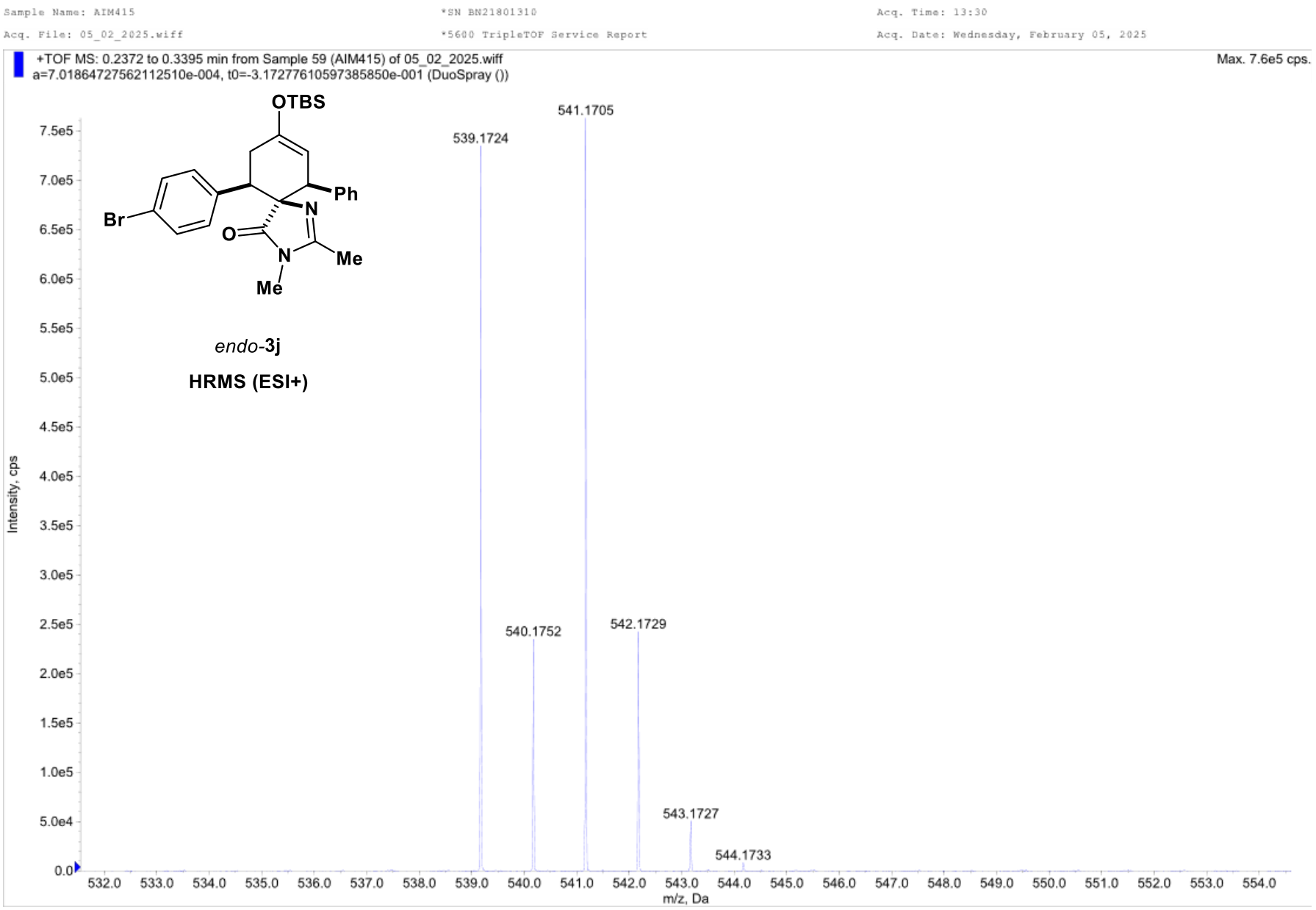
endo-**3j**

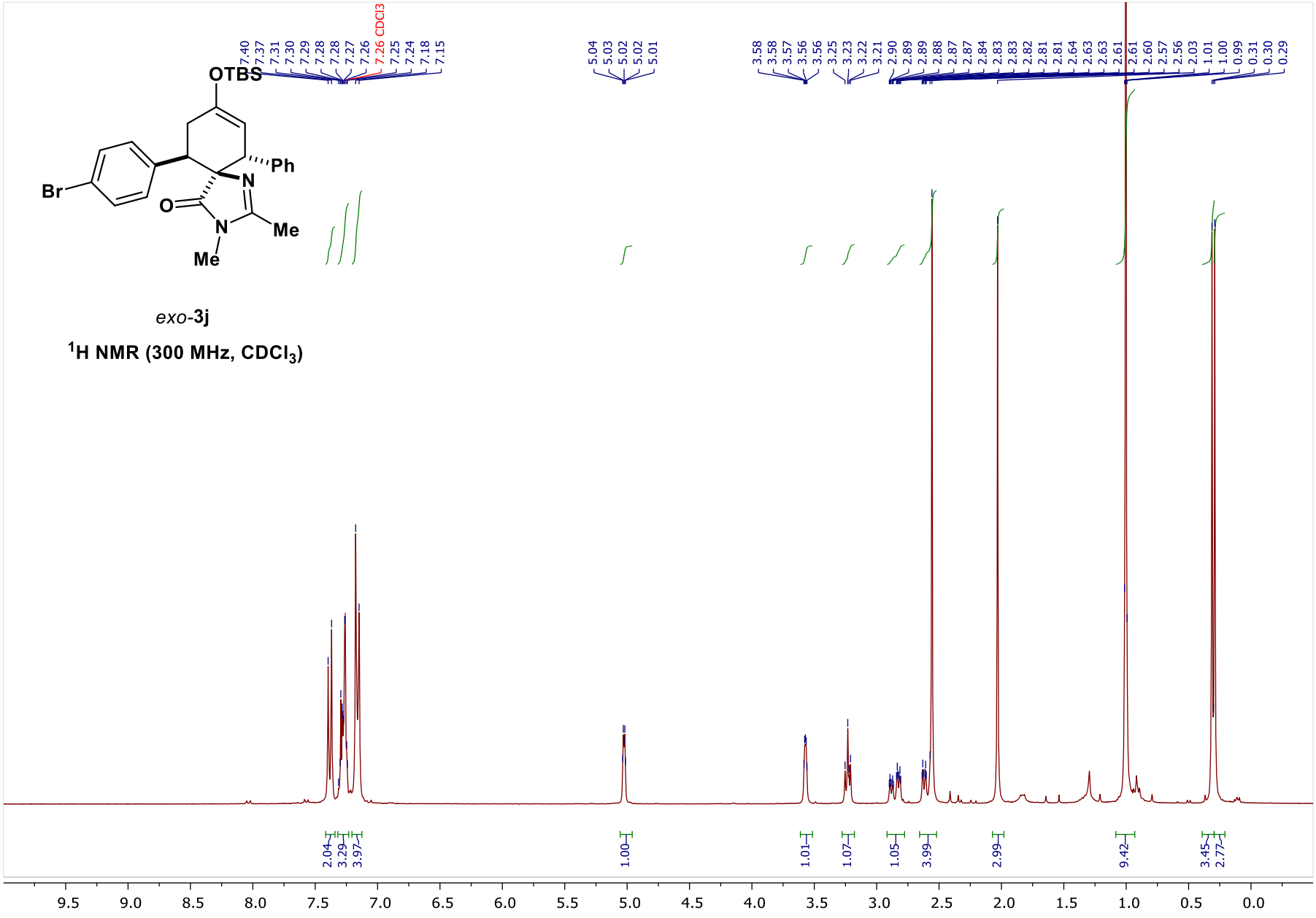
^{1}H NMR (300 MHz, $CDCl_3$)



endo-**3j**

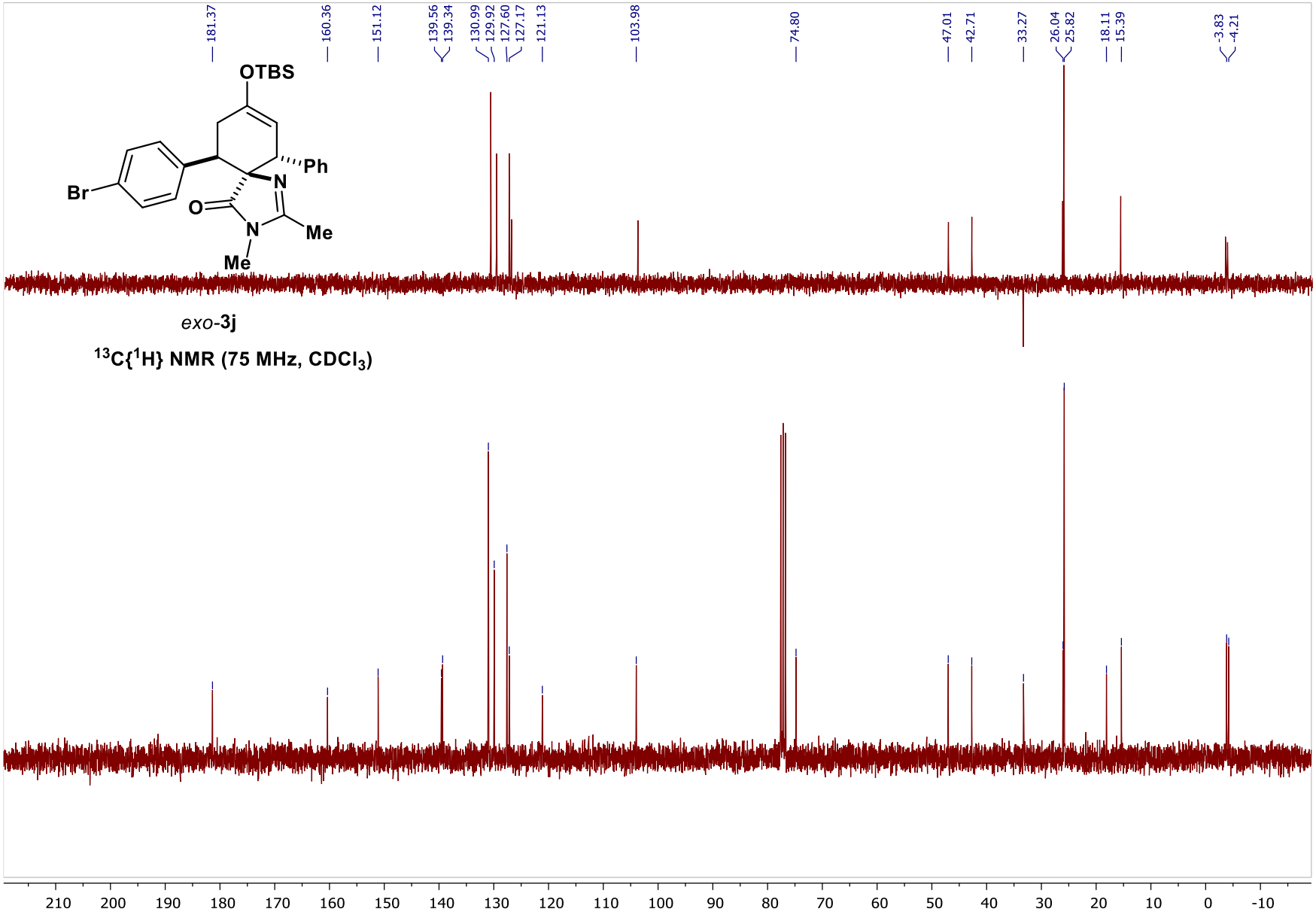
$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$)


Sample Name: AIM415
*SN BN21801310
Acq. Time: 13:30
Acq. File: 05_02_2025.wiff
*5600 TripleTOF Service Report
Acq. Date: Wednesday, February 05, 2025
+TOF MS: 0.2372 to 0.3395 min from Sample 59 (AIM415) of 05_02_2025.wiff
a=7.01864727562112510e-004, t0=-3.17277610597385850e-001 (DuoSpray ())
Max. 7.6e5 cps.
OTBS
Ph
Br
N
O
N
Me
Me
endo-3j
HRMS (ESI+)
539.1724
541.1705
540.1752
542.1729
543.1727
544.1733
Intensity, cps
m/z, Da



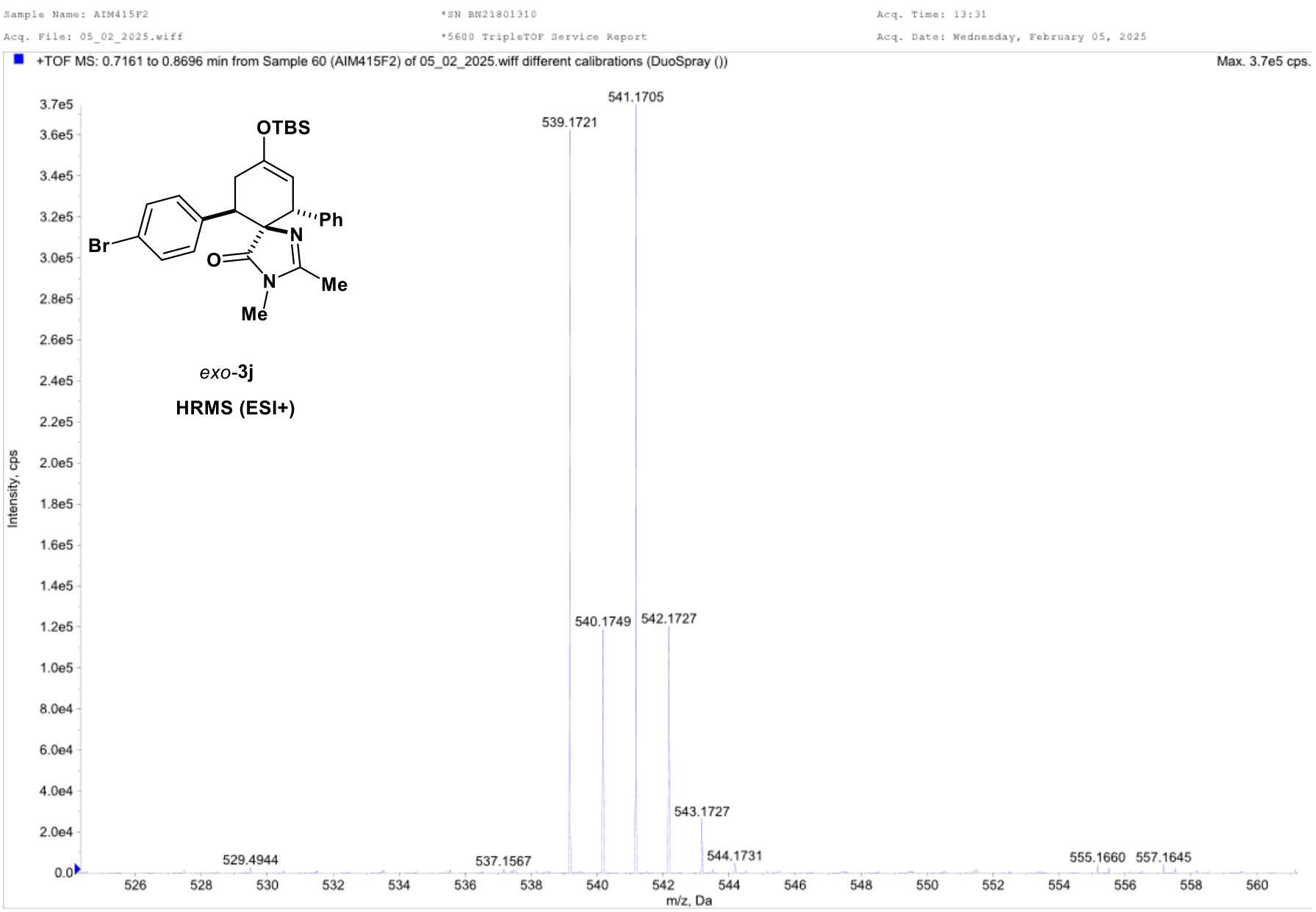
exo-**3j**

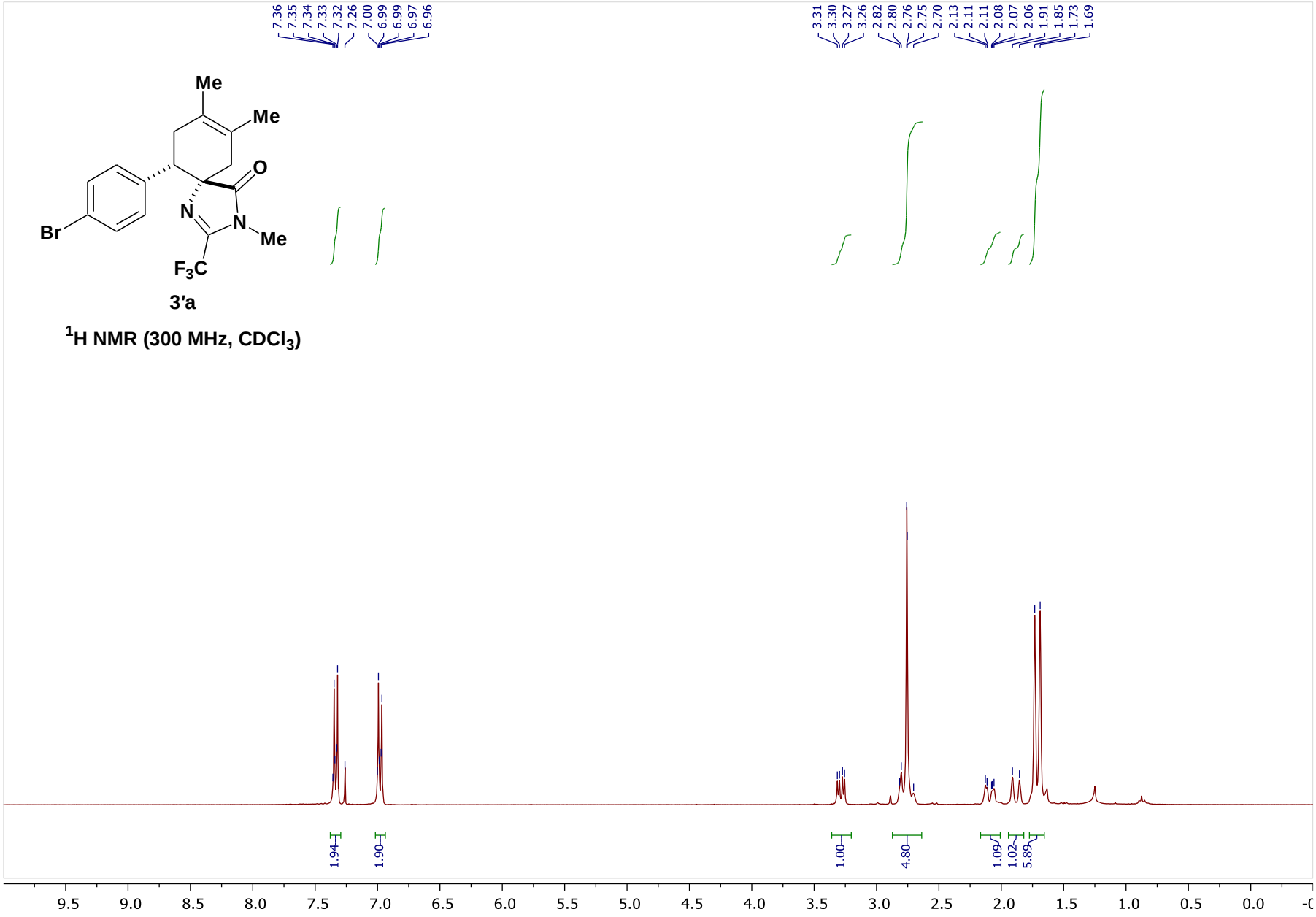
^{1}H NMR (300 MHz, $CDCl_3$)



exo-**3j**

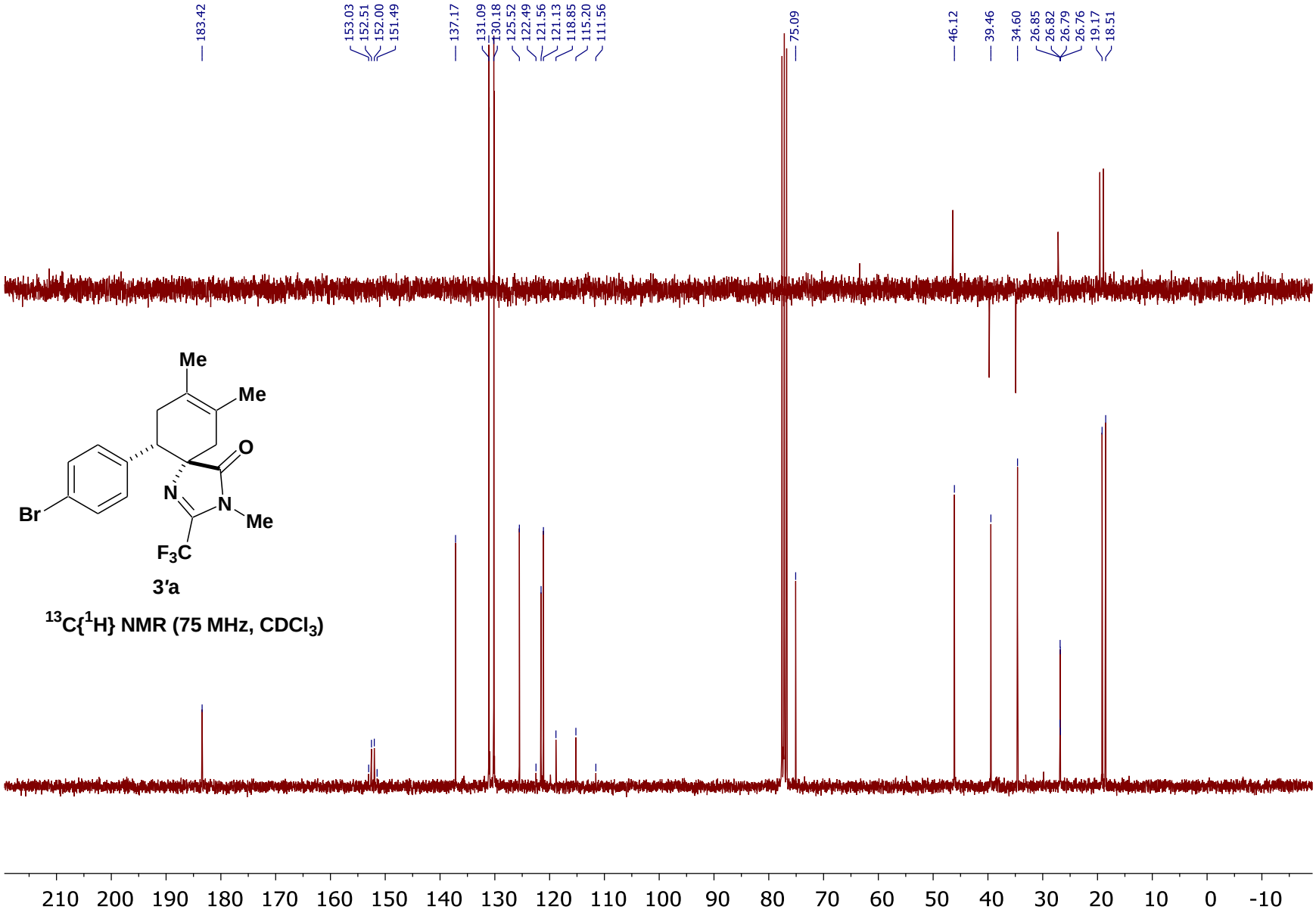
$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$)


Sample Name: AIM415F2
*SN BN21801310
Acq. Time: 13:31
Acq. File: 05_02_2025.wiff
*5600 TripleTOF Service Report
Acq. Date: Wednesday, February 05, 2025
+TOF MS: 0.7161 to 0.8696 min from Sample 60 (AIM415F2) of 05_02_2025.wiff different calibrations (DuoSpray ())
Max. 3.7e5 cps.
OTBS
Br
Ph
N
O
N
Me
Me
exo-3j
HRMS (ESI+)
Intensity, cps
m/z, Da
541.1705
539.1721
540.1749
542.1727
543.1727
544.1731
529.4944
537.1567
555.1660
557.1645



3′a

^{1}H NMR (300 MHz, $CDCl_3$)



3′a

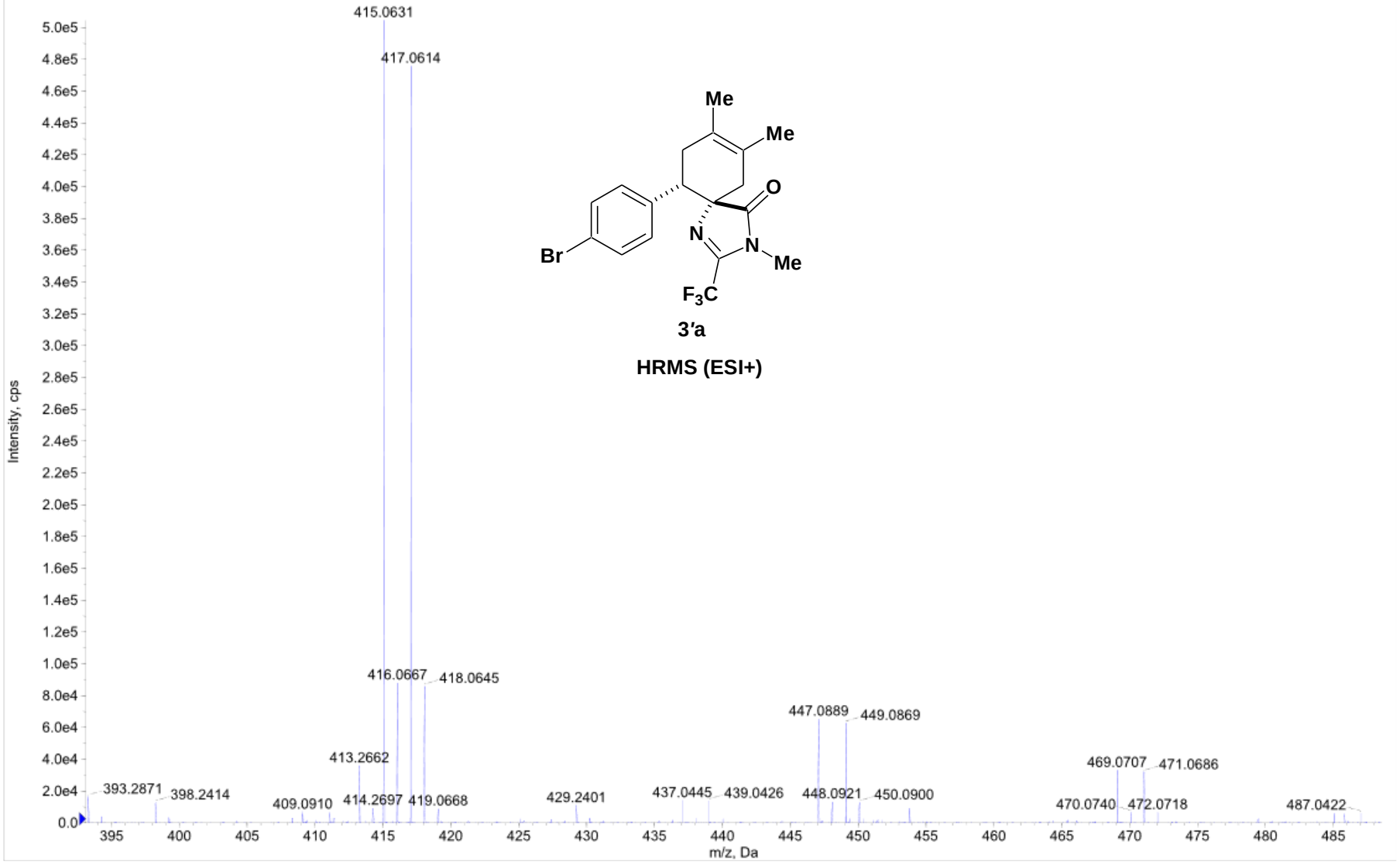
$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$)

Sample Name: AIM409 *SN BN21801310 Acq. Time: 13:09

Acq. File: 05_02_2025.wiff *5600 TripleTOF Service Report Acq. Date: Wednesday, February 05, 2025

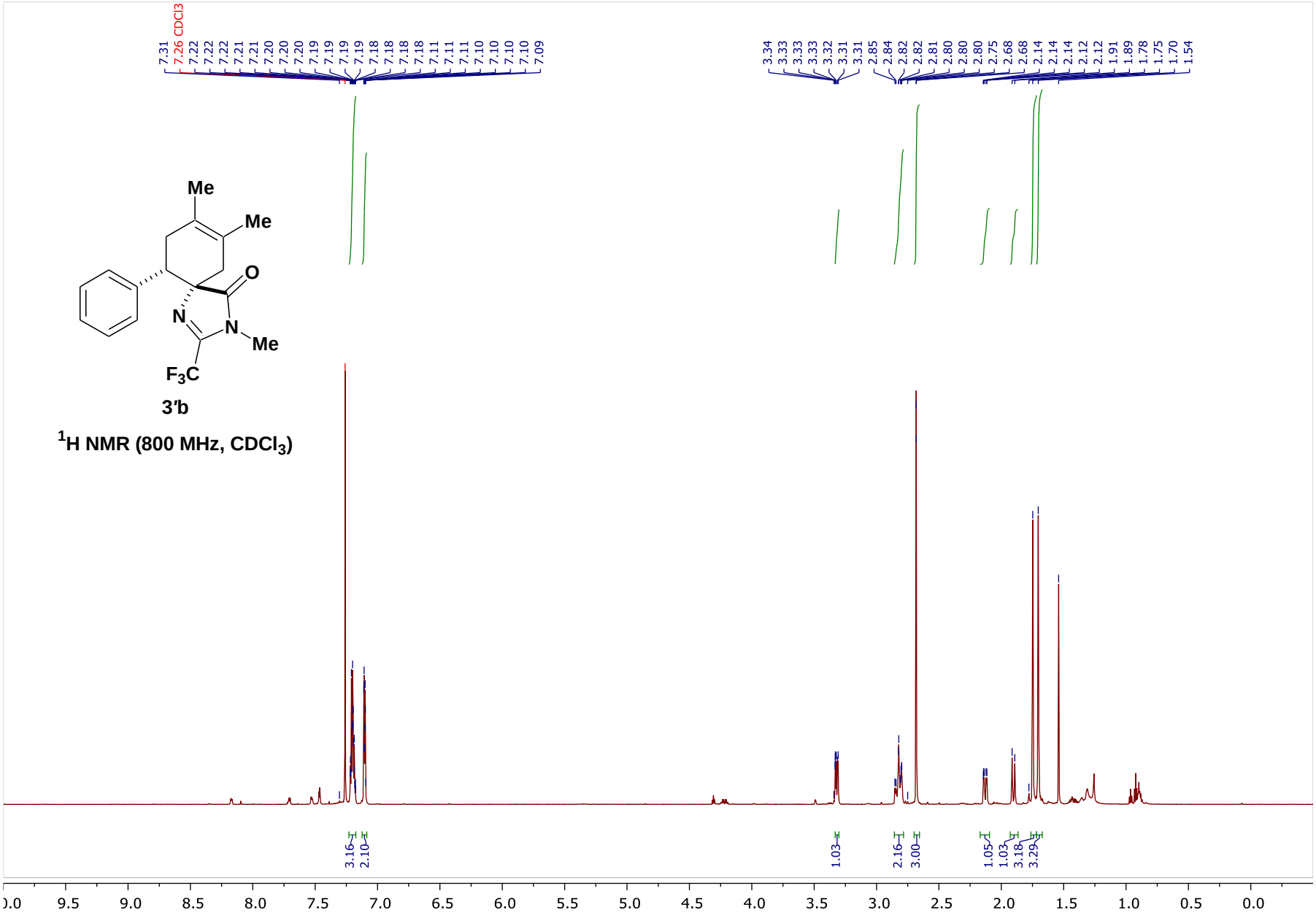
+TOF MS: 0.2325 to 0.3487 min from Sample 54 (AIM409) of 05_02_2025.wiff
a=7.01863049039572590e-004, t0=-3.37753263103003910e-001 (DuoSpray ())

Max. 5.0e5 cps.



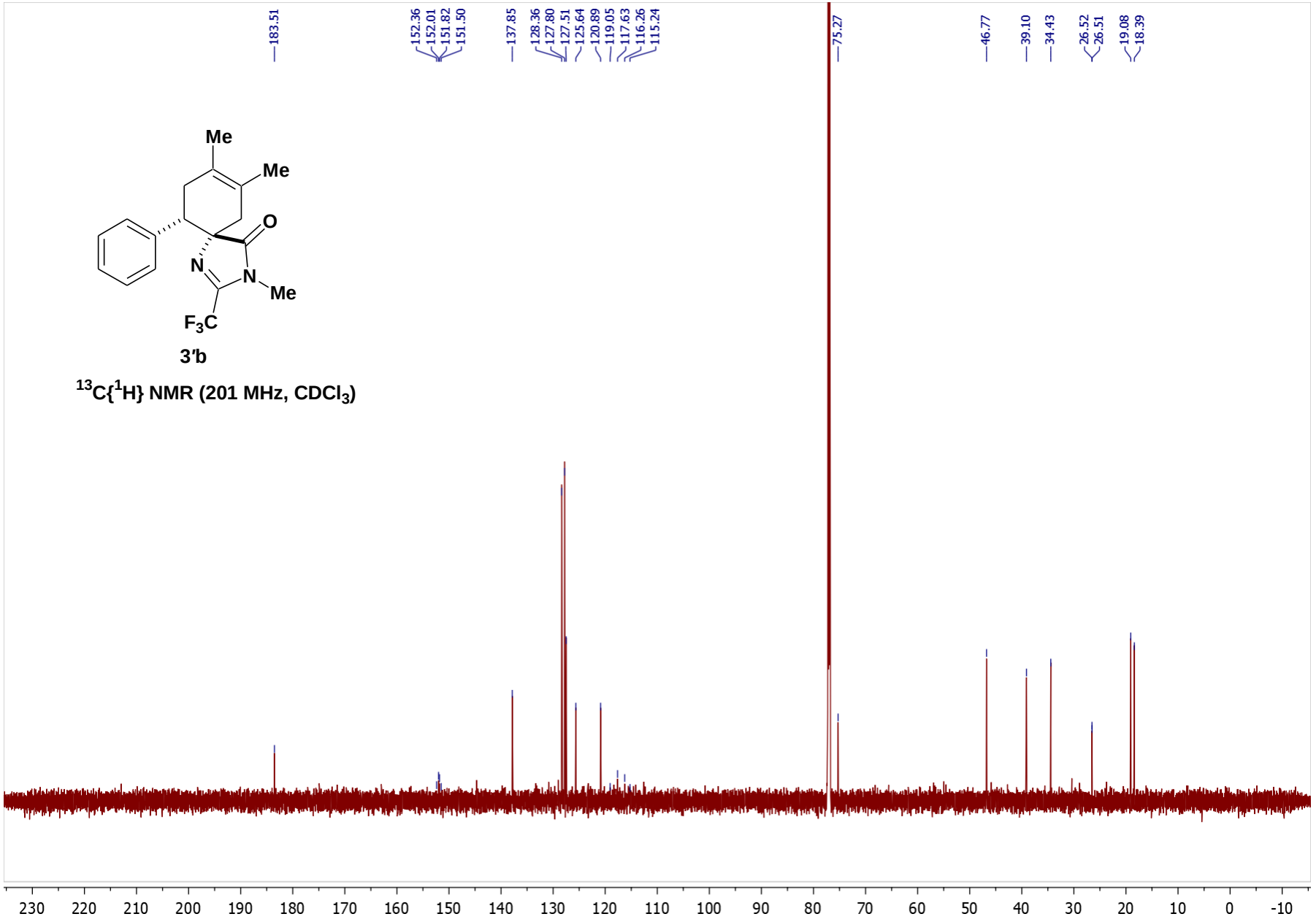
3′a

HRMS (ESI+)



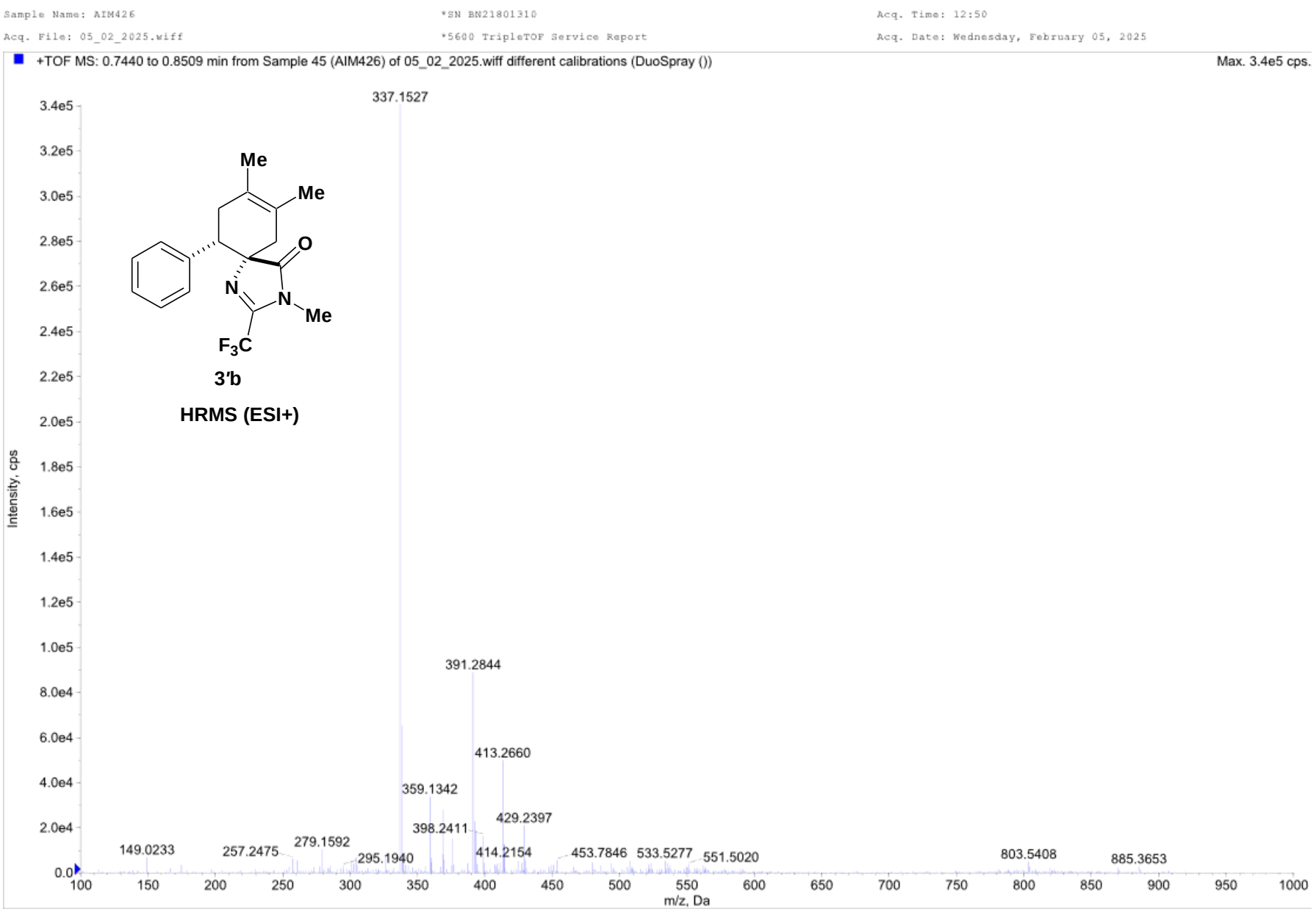
3′b

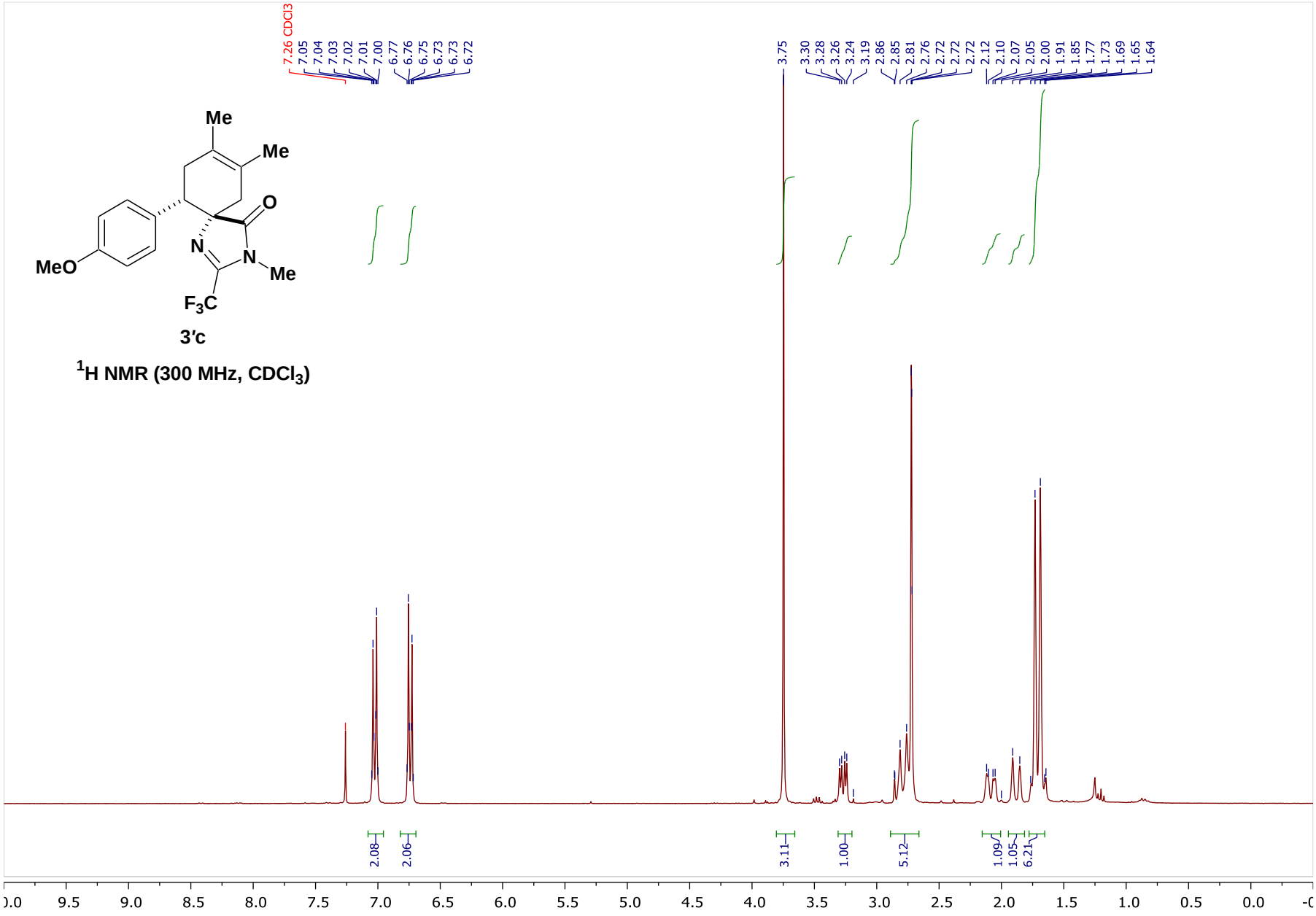
^{1}H NMR (800 MHz, $CDCl_3$)



3′b

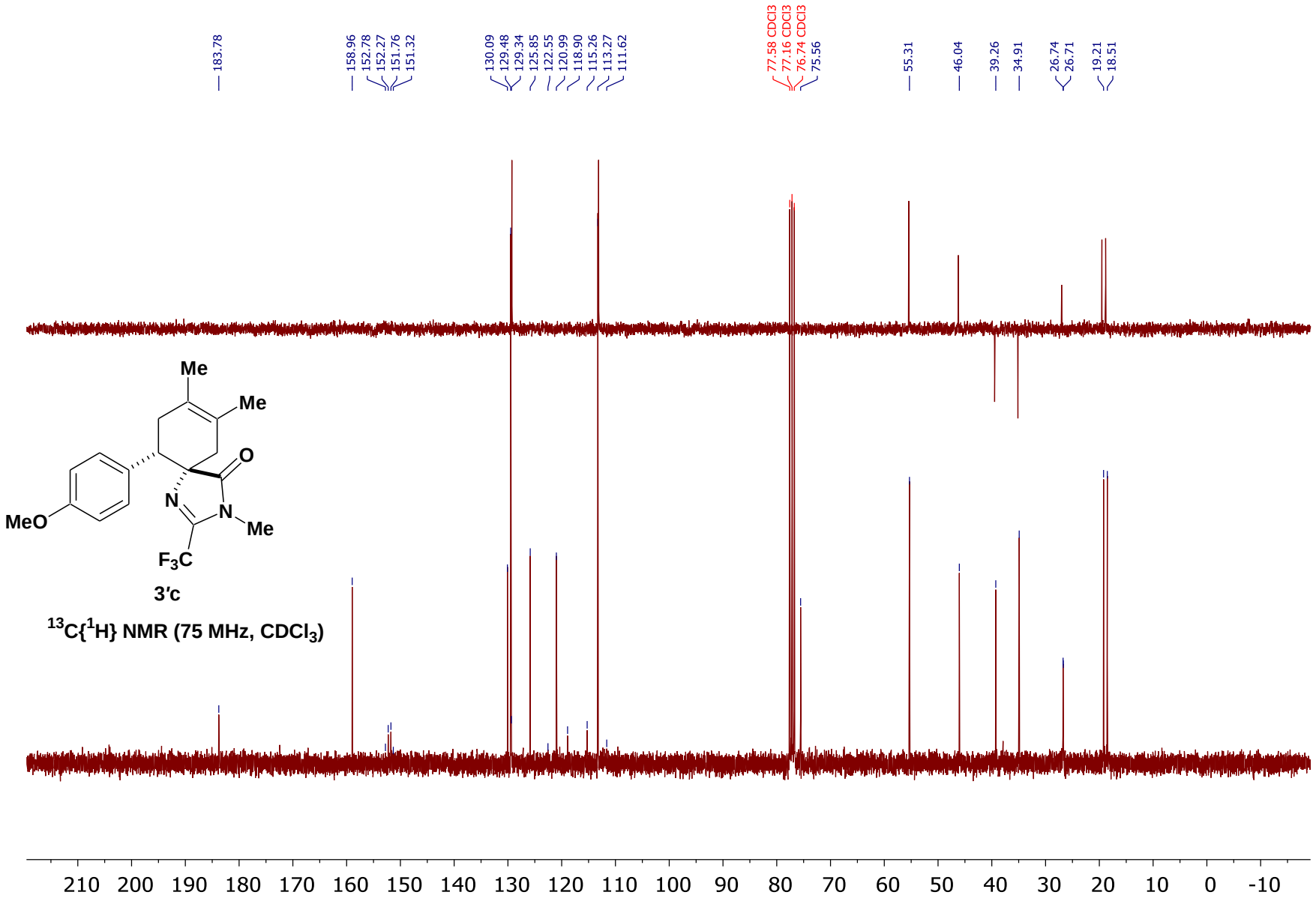
$^{13}C\{^{1}H\}$ NMR (201 MHz, $CDCl_3$)


Sample Name: AIM426
*SN BN21801310
Acq. Time: 12:50
Acq. File: 05_02_2025.wiff
*5600 TripleTOF Service Report
Acq. Date: Wednesday, February 05, 2025
+TOF MS: 0.7440 to 0.8509 min from Sample 45 (AIM426) of 05_02_2025.wiff different calibrations (DuoSpray ())
Max. 3.4e5 cps.
Me
Me
O
N
N
Me
F3C
3′b
HRMS (ESI+)
Intensity, cps
m/z, Da
337.1527
391.2844
413.2660
359.1342
429.2397
398.2411
149.0233
257.2475
279.1592
295.1940
414.2154
453.7846
533.5277
551.5020
803.5408
885.3653



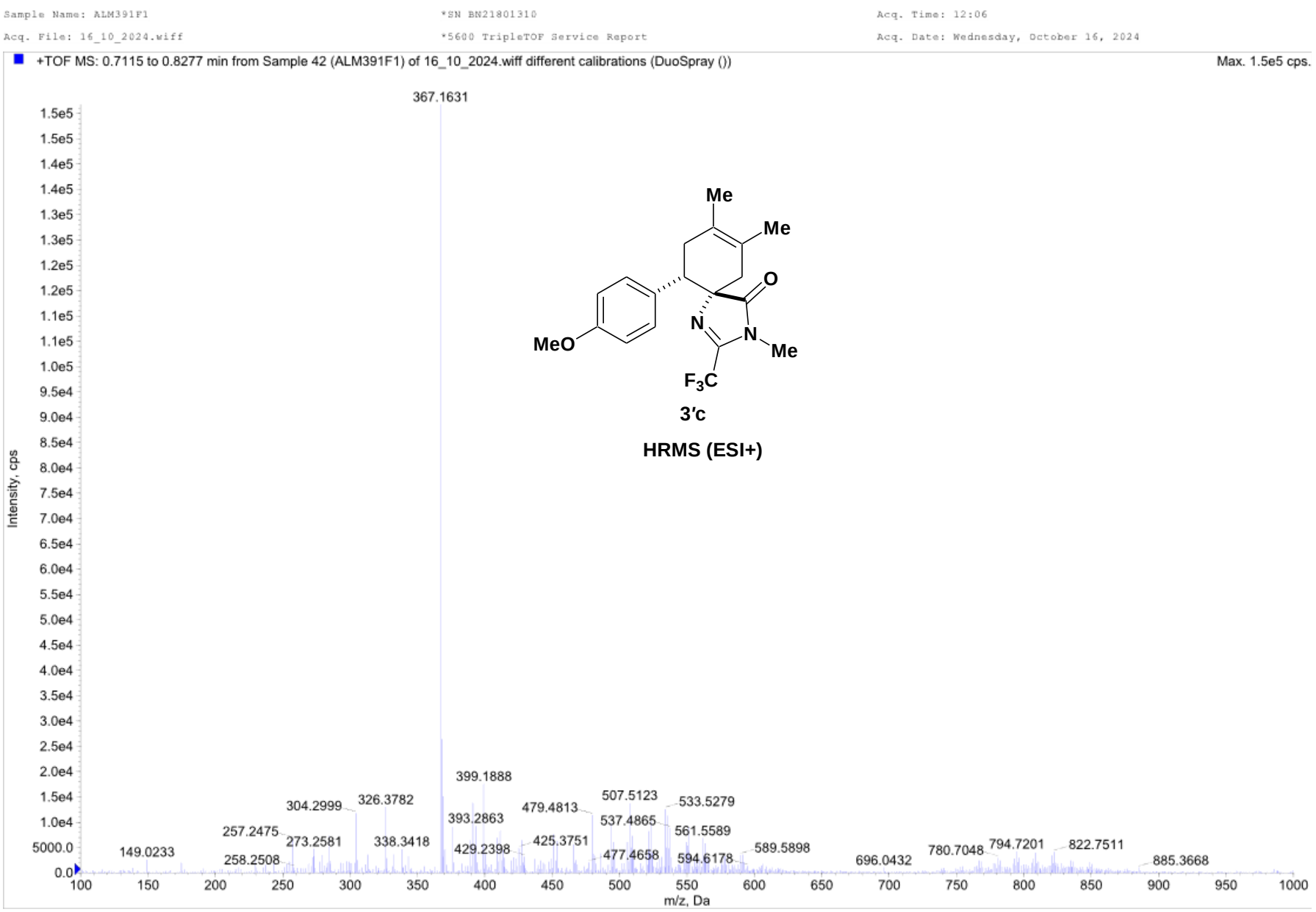
3′c

^{1}H NMR (300 MHz, $CDCl_3$)



3′c

$^{13}C\{^{1}H\}$ NMR (75 MHz, $CDCl_3$)


Sample Name: ALM391F1
*SN BN21801310
Acq. Time: 12:06
Acq. File: 16_10_2024.wiff
*5600 TripleTOF Service Report
Acq. Date: Wednesday, October 16, 2024
+TOF MS: 0.7115 to 0.8277 min from Sample 42 (ALM391F1) of 16_10_2024.wiff different calibrations (DuoSpray ())
Max. 1.5e5 cps.
367.1631
Intensity, cps
m/z, Da
Me
Me
O
N
N
Me
MeO
F3C
3′c
HRMS (ESI+)